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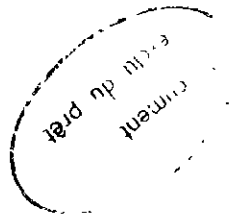

APPLICATION OF PHOSPHORIC ESTERS TO FLOTATION OF FINELY DIVIDED OXIDIZED ORES

Final Report

C.E.C. Research Contract MA1M-0059-C

G. Baudet (BRGM)
G. Morteani (TUM)
M.-P. Strub (CECA)

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BRGM
DIRECTION DES ACTIVITÉS MINIÈRES
Département Minéralurgie
B.P. 6009 - 45060 ORLÉANS CEDEX 2 - France - Tél. : (33) 38.64.34.24

ABSTRACT

A study dealing with flotation of rare earth (R.E.) minerals with phosphoric esters as collectors was conducted by the Mineral Technology Department of the BRGM, the Technical University of Munich and the laboratory for Research and Applications of CECA, within the C.E.C. Research Contract MA1M-0059-C.

The objectives were: to define RE bearing minerals occurring in typical ores, to develop new flotation processes and new phosphoric ester (P.E.) collectors with optimum performance for concentration of finely divided rare earth containing ores.

P.E. collectors were evaluated for their flotation performance on three typical RE ore samples with compositions as follows:

	RE ore sample		
	n° 1	n° 2	n° 3
R.E.E + Y %	0.746	4.483	7.512
(Eu+Y)/(REE + Y)	0.1329	0.0033	0.0063

- *RE ore sample 1 consists of Y and REE containing apatites as the major REE bearing minerals, REE bearing silicates: britholites and allanite as minor REE bearing minerals, clinopyroxene, magnetite, biotite, albite, amphilbola and clays as main gangue minerals. Compositions of RE and gangue minerals were determined by microprobe analysis. RE containing apatites were floated from the ore ground down to 160 µm, deslimed at 10 µm, and treated by low intensity magnetic separation to remove magnetite.*

- *RE ore sample 2 consists of low Th monazite as the major RE bearing mineral, Mn-Fe containing dolomite, strontianite, siderite, calcite, barite, manganese oxides, magnetite and quartz, as the major gangue minerals. Compositions of RE and gangue minerals were determined by microprobe analysis. Flotation of monazite by P.E. collectors was applied to - 160 + 10 µm, - 50 + 10 µm fractions, - 80 + 10 µm and - 40 + 10µm gravity preconcentrates, separated from the ore ground down to 160 µm and deslimed at 10 µm. Gravity preconcentration to remove part of the dolomite and calcite was found to be necessary to allow efficient concentration of monazite by flotation.*

- *RE ore sample 3 consists of bastnaesite as the major RE bearing mineral, fluorite and barite as potentially valuable minerals, silicates, carbonates, Fe and Mn oxides as major gangue minerals. Attempts to concentrate bastnaesite by flotation from the - 125 + 10 µm and - 10 µm(1) fractions, separated from the ore ground down to 125 µm, were unsuccessful due to poor liberation or the extremely fine size distribution of the slimes. However, preliminary flotation tests showed that P.E. could be used as fluorite collectors.*

Ten different types of P.E. collectors were characterized and evaluated on RE ore samples 1 and 2.

The best concentration performance on RE ore sample 1 was achieved using phosphoric ester B110 88094 as a RE containing apatite collector, with a low addition of 225 g/t, in an alkaline pulp of pH of about 9.5, a solution of Na silicate and Na citrate, in combination with corn starch, as gangue depressants.

The best concentration performance on RE ore sample 2 was achieved using phosphoric ester PE 89112 EA-89 as a monazite collector, with an extremely low addition of 70 g/t, in a slightly acidic pulp of pH of about 5.3., CO_2 as a depressant for strontianite, siderite, residual Fe-Mn containing dolomite and barite.

	Composite flotation concentrate	Feed to flotation
RE ore Sample 1		
P_2O_5 recovery %	81.93	100
P_2O_5 content %	31.82	3.42
apatite content %	88.9	9.55
CaO/P_2O_5	1.437	2.263
$(R_2O_3+MgO)/P_2O_5$	0.0716	4.7515
total REE + Y content %	4.3866	0.7642
Eu content %	0.0150	0.0020
Y content %	0.8015	0.1006
total REE + Y recovery %	50.40	100
Eu recovery %	65.15	100
Y recovery %	69.94	100
RE ore sample 2		
Fe_2O_3 content %	1	4,39
SrO content %	1,52	9.85
BaO content %	0.72	5.25
total REO content %	65.70	42.77
monazite content %	95.22	61.98
total REO recovery %	79.94	100

Flotation in a continuous circuit, involving recirculation of middling products could lead to increased RE recoveries in concentrates. Differences between RE, Y and P_2O_5 recoveries in concentrate separated from the RE ore sample 1 account for occurrence of RE bearing silicates which are not floated with PE collectors, this concentrate is of acceptable grade for production of wet process phosphoric acid besides extractions of Y, Eu and other valuable RE.

Statistical analysis of flotation data:

· showed that characteristics of the PE collectors provide variables to satisfactorily explain variations in selectivity and concentrate grade. Recovery of valuable minerals and concentration efficiency are not so well explained by characteristics of the collectors.

· *led to the following conclusions:*

- *the most efficient PE collectors for RE and Y containing apatite should have a high content in mono-orthophosphoric ester (MEO), a low concentration in residual H_3PO_4, and an optimized number n of ethylene oxide groups in the molecule, giving satisfactory apatite recovery with moderate detrimental effect on the apatite content of the concentrates*
- *the most effective PE collectors for monazite are characterized by high n values, within the experimental range explored: 2 to 14, high concentrations in MEO, low concentrations in residual non phosphated products and H_3PO_4.*

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1. INTRODUCTION

Within a C.E.C. Research Contract MA1M-0059-C, a study related to flotation of rare earth elements bearing minerals using phosphoric esters as collectors, was conducted by:

- the Mineral Technology Department of the BRGM (Bureau de Recherches Géologiques et Minières), Orléans - France
- the Lehrstuhl für Angewandte Mineralogie und Geochemie of the Technische Unversität of Munchen (T.U.M.) - Germany
- the laboratory for research and applications of the C.E.C.A. Company, at Corbehem - France.

The objectives of the Research program were:

- to develop new phosphoric ester (PE) derived flotation collectors with optimum performance for concentration of finely divided rare earth elements (REE) bearing ores
- to define REE bearing minerals occurring in typical ores
- to develop flotation processes using PE derivatives, alone or in conjunction with other promoters, as collectors applicable to difficult to beneficate REE ores. This could promote the development of new deposits which could constitute new potential REE sources for European producers of refined rare earth chemicals and metals.

Preliminary laboratory studies have shown that particular types of phosphoric esters, jointly developed by GERLAND-CHIMIE (acquired by S.H.P.C., at present CECA) and BRGM, for separating carbonates from phosphate by flotation of sedimentary high carbonate phosphate rock, exhibit good selectivity and strong collecting properties for certain finely oxidized divided minerals. This specially applies to rare earth oxide (REO) bearing minerals, as an example a high REO concentration ratio value of 11 was achieved, using a phosphoric ester as a collector, on a - 20 μm stabilized slimes sample containing less than 1% total REO, as monazite, associated with clays, silica, feldspar and iron minerals.

Despite a REO world production well below the mining capacity, it appears that REO supply to Europe and production of refined derivatives are sensitive to the following factors:

- increased semi-refining and refining capacities in REO concentrates producing countries
- fluctuating demand in certain rare earth derivatives depending upon end uses and new developing applications. This is related to the relative amounts of rare earth elements contained in bastnaesite, monazite and xenotime concentrates

- occurrence of radio active Th in high amount in most of the monazite concentrates, which are widely used in Europe, along with a significantly lower total REO content with respect to bastnaesite concentrates dominantly used by the refining plants in the USA

- insufficiently diversified sources of concentrates and low reserves of acceptable grade ores

- emergence of China as a major producer of both REO concentrates, with high Eu content, and refined oxides.

With a view to increase diversification and quality of European sources of REO concentrates it would be desirable to evaluate new potentially interesting deposits the development of which closely depends upon performance of beneficiation. This applies to flotation which appears as the most flexible and promising process for recovery of REO values form difficult to beneficiate ores containing finely divided REO.

The collaborative research project between BRGM, the Technical University of Munich, CECA-SHPC laboratories, was developed to investigate the response to flotation of REE Minerals with P.E. as collectors.

The research program covers the following:

BRGM and the Technical University of MUNICH	▪ selection and characterization of REE ore samples, with special attention given to determinations of REE contents in R.E. bearing minerals
BRGM	▪ preparation of REE ores to liberate minerals to be separated ▪ laboratory flotation tests to correlate compositions of phosphoric esters to their performance for REE concentration
CECA-SHPC and BRGM	▪ synthesis and characterization of new phosphoric ester collectors ▪ analysis of flotation data in order to select the more selective P.E. and optimize their composition.

2. CHARACTERIZATION AND MECHANICAL PREPARATION OF RARE EARTH ORE SAMPLES

Three REO ore samples were characterized and subjected to a mechanical preparation so as to liberate REO bearing minerals:

- REO ore sample n° 1 was taken from finely divided old tailing piles produced through processing operations applied to an iron ore at Mineville (U.S.A.). The main potentially valuable mineral is a REE and Y rich apatite

- REO ore sample n° 2 is a typical sample of raw carbonatite rock taken from the Kangankunde hill deposit in Malawy. Low Th containing monazite (ThO_2 content of monazite: 0,08 %) and strontianite represent the potentially valuable minerals

- REO ore sample n° 3 (TU sample) is a composite of bulk ore samples taken from the bastnaesite deposit of Kizicaören-Eskisehir in Central Turkey. Fluorite, barite and bastnaesite account for potentially valuable minerals.

2.1. CHEMICAL COMPOSITIONS

Major elements were determined by X ray Fluorescence on glass beads made with lithium tetraborate as a flux, REE and other trace elements were analysed by Induced Coupled Plasma associated to Mass Spectrometry (ICP/MS), these techniques were used by BRGM and TUM laboratories.

XRF and other spectrometric methods: FADCP, DCP, DCPA, FAA and GFAA, were also used by the TUM laboratories for determinations of REE, trace and major elements on the REO ore sample n° 3.

ICP spectrometry was used by BRGM laboratories for determinations of trace elements occurring in the REO ore sample n° 1. Wet chemical methods including gravimetry, potentiometry and ion selective electrodes, volumetry and colorimetry were also used by BRGM laboratories for analyzing Ba, F, S, P, FeO, Ti ; Sr was determined by S.A.A.

REE and major elements determinations are shown in table 1.

Trace elements other than REE, determined by ICP, are shown in paragraph 2.3., for size fractions separated from the REO ore sample 1, ground down to 0.16 mm.

More complete determinations of major elements, including alkaline oxides, were made on the - 0.16 + 0.01 mm fraction separated from the REO ore sample 1 ground down to 0.16 mm, compositions are given in the material balance relevant to flotation test n° 22 in paragraph 3.1.

The samples exhibit large differences in REE + Y contents, however, the REO ore sample with the lowest content in REE+Y has the most favourable composition in terms of Eu and Y contents:

	REO ore sample		
	n° 1	n° 2	n° 3
(REE + Y) % (Eu + Y) / (REE+Y)	0.746 0.1329	4.483 0.0033	7.512 0.0063

		REO ORE SAMPLE		REO ORE SAMPLE	
		n° 1*	n° 2	n° 3	
P_2O_5	%	3.13	3.56	SiO_2	% 1.85
CaO	%	7.32	16.77	P_2O_5	% 0.55
SiO_2	%	56.51	4.61	Fe_2O_3	% 2.53
Fe_2O_3	%	14.01	7.31	Al_2O_3	% 0.78
Al_2O_3	%	7.88	0.96	SO_4	% 8.10
MgO	%	1.67	12.94	Ca	% 28.05
TiO_2	%		0.06	F	% 30.30
LOI	%	0.93	30.71	Ba	% 11.36
Na_2O	%		< 0.20		
K_2O	%		< 0.05		
MnO	%		1.89		
CO_2	%		30.48		
H_2O-	%		0.23		
H_2O+	%		1.29		
SrO	%		9.98		
BaO	%		1.28		
SO_3	%		0.95		
La	ppm	1 532	14 045	La	ppm 32 500
Ce	ppm	2 988	23 044	Ce	ppm 34 500
Nd	ppm	1 142	6 466	Nd	ppm 4 822
Sm	ppm	217	715	Sm	ppm 316
Eu	ppm	20	88	Eu	ppm 74
Gd	ppm	207	261	Gd	ppm 143
Dy	ppm	196	103	Dy	ppm 66
Er	ppm	100	30	Er	ppm 39
Yb	ppm	86	11	Yb	ppm 28
Y	ppm	971	61	Y	ppm 398
				Ho	ppm 13
				Lu	ppm 4
				Pr	ppm 2 194
				Tb	ppm 18
				Tm	ppm 5
Total REE+Y	ppm	7 459	44 824	Total REE+Y	ppm 75 120

** fine grained facies*

Table 1 : Chemical compositions of REO ore samples

High SiO_2, Al_2O_3 and Fe_2O_3 contents in REO ore sample n° 1, high CO_2, CaO, MgO and SrO contents in REO ore sample n° 2, high F, Ba, SO_4 contents in REO ore sample n° 3, reflect the nature of the main gangue minerals and potentially valuable minerals associated with the REE bearing minerals:

- silicates, alumino silicates, iron oxides in REO ore sample 1
- carbonates: dolomite, strontianite, siderite and calcite in REO ore sample 2
- fluorite and barite in REO ore sample 3.

2.2. MINERALOGIC COMPOSITIONS

Ore mineralogy involved observations of typical ore fragments or grains by:

- optical microscopy on polished sections and thin sections
- Scanning Electron Microscopy (S.E.M.)

to assess identification of minerals, primarily based on their optical properties in both transmitted and reflected light, by XRF microanalysis. An EDAX analysis system coupled to the S.E.M. was used by the T.U.M. on the REO ore sample n° 3.

Additional determinations were made on fine sized fractions containing clay minerals by X ray diffractometry.

Quantitative chemical determinations on minerals occurring in REO ore samples n° 1 and 2 were effected by BRGM laboratories, using a CAMECA electron microprobe and data processing specially designed for REE analysis. P, Ca, Si, F, Fe, Ce, Pr, Nd, La, Sm, Gd, Y (or Dy, Yb, and Er, substituted for Ce, Pr and La) were determined on REE bearing minerals ; S, Ba, Sr, Ca, Ti, Mn, Mg, La, Fe, Ce, Si, P, Al, K, Na, on gangue minerals.

2.2.1. Mineralogic characterization of the REO ore sample n° 1

Most of the REE mineralization occurs as apatite with small contents in heavy REO.

Other REO bearing minerals are:

- a Cerium - britholite, a REO containing silico-phosphate with the following theoretical formula: $(Ce, Ca)_5 (SiO_4, PO_4)_3 (OH, F)$
- A Yttrium - britholite with the following theoretical formula: $Ca_2 Y_3 Si_3 O_{12} (OH)$, however, the mineral also contains a small amount of heavy REO as Dy_2O_3 and Yb_2O_3
- allanite a REO bearing silicate with the theoretical formula: $6SiO_2$, $3M_2O_3$, $4RO$, H_2O where R could be Ca, Fe^{2+} and M: Al, Fe^{3+} and REE
- a Cerium apatite
- a mixture of REO
- a secondary apatite with a low content in REO.

Most of REO bering minerals other than apatites are very finely disseminated (1 to 50 μm) within the apatites, except a part of the Y - britholite. Most of the apatite is liberated at about 500 μm, a grinding size of about 160 μm has been selected for the purpose of separating REO minerals from the gangue by flotation, and reach liberation of Mg, Al, Fe containing silicates sometimes associated to the apatite particles.

Gangue minerals consist of clinopyroxene, magnetite, biotite, albite, amphibola, ilmenite, zircon, iron carbonate, pyrite and clay minerals: kaolinite, smectite, chlorite, sepiolite and talc.

The average chemical compositions of the major minerals, as determined by microprobe analysis, are shown in tables 2 to 4.

Average REO contents in REE bearing minerals, expressed in weight percentages, are shown in the following table:

	Y_2O_3	La_2O_3	Ce_2O_3	Pr_2O_3	Nd_2O_3	Sm_2O_3	Gd_2O_3	Dy_2O_3	Er_2O_3	Yb_2O_3
Apatite (A)	1.74	1.76	3.74	0.63	2.02	0.33	0.29	0.15	0.02	0.10
Ce Apatite (TR)	0.49	14.19	18.08	3.01	5.54	0.68	0.21	ND	ND	ND
Ce Britholite (W)	4.55	9.58	21.61	3.67	11.79	2.02	1.72	ND	ND	ND
Y Britholite (R)	27.12	ND	ND	ND	0.73	0.79	1.85	2.69	2.47	2.60
Allanite (R)	0.00	4.46	10.97	1.92	4.89	0.59	0.10	ND	ND	ND
Oxides (R)	1.18	14.17	26.56	4.64	12.90	1.98	1.19	ND	ND	ND

where proportions of REO bearing minerals are estimated as A: Abundant, W: Weak, R: Rare, TR: Traces.

The report of the mineralogic studies conducted by O. LEGENDRE (BRGM) is shown in the appendix 1.

1	2	3	4	5	6
Fe_2O_3	0.12	0.85	23.05	2.22	0.00
SiO_2	3.87	6.32	31.87	23.26	0.51
Ce_2O_3	3.74	18.08	10.97	21.61	26.56
Pr_2O_3	0.63	3.01	1.92	3.67	4.64
P_2O_5	37.07	33.61	0.66	4.23	7.49
CaO	45.53	19.05	8.67	9.09	9.88
Nd_2O_3	2.02	5.54	4.89	11.79	12.90
Y_2O_3	1.74	0.49	-	6.02	1.18
La_2O_3	1.76	14.19	4.46	9.58	14.17
Sm_2O_3	0.33	0.68	0.59	2.02	1.98
Gd_2O_3	0.29	0.21	0.10	1.72	1.19
total	97.11	102.02	87.19	95.20	80.51

Column 1: Elements analyzed
Column 2: REE bearing apatite
Column 3: Ce bearing apatite
Column 4: Allanite
Column 5: Ce britholite
Column 6: Other REO bearing minerals (bastnaesite, parisite)

Table 2: Average compositions of minerals occurring in the REO ore sample n° 1

1	2	3
Fe_2O_3	0.17	0.70
SiO_2	1.83	36.56
CaO^2	50.95	16.61
Na_2O_3	1.00	0.73
P_2O_5	40.58	2.20
Sm2O3	0.06	0.79
Y2O3	1.04	27.12
Gd2O3	0.42	1.85
Dy2O3	0.14	2.69
Yb2O3	0.10	2.60
Er2O3	0.02	2.47
total	96.31	94.31

Column 1: Elements analyzed
Column 2: Heavy REO in apatite
Column 3: Y britholite

Table 3: Average compositions of minerals occurring in the REO ore sample 1

	1	2	3	4
SiO_2	46.87	36.74	67.32	37.84
TiO_2	0.80	3.21	-	1.02
Al_2O_3	3.65	15.90	19.24	10.77
FeO	18.86	30.22	-	24.76
MnO	-	-	-	-
MgO	10.24	0.14	-	9.75
CaO	17.52	-	0.02	9.77
Na_2O	0.64	0.10	6.42	2.10
K_2O	0.70	9.01	7.12	1.92
total	99.28	95.32	100.12	97.93

REO ORE SAMPLE 1

Column 1 : Pyroxene
Column 2 : Biotite
Column 3 : Feldspar
Column 4 : Amphibola

Table n° 4: Average compositions of minerals occurring in the REO ore sample 1.

Chemical analysis of apatite enriched fractions produced by flotation or by combinations of heavy liquid and high intensity magnetic separations have indicated that the maximum P_2O_5 content obtainable in apatite particles separated from the ore ground at 160 μm is 35.8 % P_2O_5. This reference value was used in estimating separation performance achieved in flotation tests.

2.2.2. Mineralogic characterization of the REO ore sample n° 2

REO bearing minerals occur as:

- monazite, apatite and traces of bastnaesite CO_3 (Ce, La, Dy) F or parisite $(CO_3)_3F_2Ca(Ce, La)_2$.

It should be pointed out that monazite has been estimated to account for more than 95 % of the REO occurring in the ore.

Gangue minerals or potentially valuable minerals are:

- carbonates : . Mn and Fe containing dolomite
  . strontianite
  . siderite containing Mn, Ca, Mg
  . calcite
  . dialogite ($MnCO_3$) was also found to occur in polished sections observed by optical microscopy
- sulfate: . barite
- oxides : . as manganese oxides (psilomelane) containing varying amounts of Fe, Ba, Ce, Sr and magnetite
- quartz.

Average chemical compositions of the major minerals, determined by electron microprobe analysis, are shown in table 5.

The liberation size of monazite was estimated to be less than 100 μm, however a grinding size of 160 μm was selected so as to prevent excessive monazite losses in slimes.

The report of the mineralogic studies conducted by O. LEGENDRE (BRGM) is shown in the appendix 2.

Average total REO content in monazite was estimated to lie somewhere between 69.4 and 70.6 % on the basis of the microprobe analysis. Chemical determinations of the total REO through acid leaching ($HCl+H_2SO_4$), separation of rare earth hydroxides refining with HF and $HClO_4$ followed by precipitation of rare earth oxalates, indicated an average REO content of 69 %. This latter value was used as a reference maximum REO content in pure monazite when estimating concentration performance for REO, in flotation tests. It was also shown that the following relationship holds:

total REO content= (La_2O_3 content + CeO_2 content) x 1.25

for the monazite occurring in the REO ore sample 2. This simplifies the analytical procedure since total REO contents or monazite contents could be derived from La and Ce assays.

	1		2		3	4	5
F	0.0043	F	0.0328	SO_3	0.0004	0.0004	0.3429
Pr_2O_3	0.0537	Pr_2O_3	0.0035	BaO	0.0011	0.0022	0.6538
P_2O_5	0.3054	P_2O_5	0.4046	SrO	0.0068	0.9685	0.0033
Ce_2O_3	0.3499	Ce_2O_3	0.0186	CaO	0.5116	0.0258	0.0000
Nd_2O_3	0.1187	Nd_2O_3	0.0112	TiO_2	0.0002	0.0004	0.0000
CaO	0.0033	CaO	0.4950	MnO	0.0154	0.0002	0.0003
Sm_2O_3	0.0111	Sm_2O_3	0.0015	MgO	0.3964	0.0001	0.0000
La_2O_3	0.1844	La_2O_3	0.0083	La_2O_3	0.0001	0.0001	0.0000
Gd_2O_3	0.0324	Gd_2O_3	0.0020	Fe_2O_3	0.0269	0.0001	0.0002
MnO	0.0000	MnO	0.0009	Ce_2O_3	0.0002	0.0005	0.0000
Fe_2O_3	0.0003	Fe_2O_3	0.0047				
O for F	-0.0018	H_2O	0.0028		------	------	------
	------	O for F	-0.0138	total	0.9591	0.9983	1.0006
total	1.0616	total	------				
			0.9721				

Column 1: Average composition of monazite
Column 2: Average composition of apatite
Column 3: Average composition of dolomite
Column 4: Average composition of strontianite
Column 5: Average composition of barite

Table 5 : Average compositions of minerals occurring in the REO ore sample 2

2.2.3. Mineralogic characterization of the REO ore sample n° 3

Ore 3 is a mixture of two types of ores:

- fluorite - barite - REE minerals ores
- carbonate ores.

The first type consists of:

fluorite: 15-60 %, barite: 10-50 %, bastnaesite: up to 20 %, Fe-Mn oxides: up to 10 %, withe mica and phlogopite: up to 15 %, calcite and dolomite: up to 20 %, chlorite, quartz, chalcedony and other REE-minerals: up to 3 %.

Most of the bastnaesite occurs in extremely fine grained felty aggregates which will generate slimes on grinding and will cause problems for its concentration and recovery by flotation.

The second type consists of:
calcite and dolomite: 40-50 %, fluorite: 15-25 %, barite: 15-20 %, bastnaesite: up to 15 %, white micas and phlogopite, Fe-Mn oxides and other REE-minerals (REO containing phosphates): up to 3 %.

Similarly to the first type, most of the bastenaesite grains exhibit a felty texture characterizing associations of fine sized crystals as aggregates.

Chemical compositions of the bulk composite sample TU and separation products clearly show a low content of carbonate minerals, this indicates that the content of ore type n° 2 is likely to be low in the composite. An estimate of the mineralogic composition of the TU sample was derived from the theoretical compositions of the minerals occurring in the material:

fluorite: 58.7 %, barite: 19.3 %, bastnaesite: 11.8 %, gangue minerals (silicates, carbonates, iron oxides, silica): 10.2 %. Bastnaesite was assumed to contain 63.8 % total REE.

Density fractionations through heavy liquid separations were effected on three size fractions separated from the attrition-scrubbed ore, results presented in chapter 2, clearly indicate that a fine grinding size of 125 μm should promote liberation of fluorite and barite.

Material balances of the attrition-scrubbing, sizing and grinding operations show that most of the REE report to the - 10 μm slimes generated by attrition and grinding, this is consistent with the occurence of bastnaesite particles as aggregates of tiny bastnaesite crystals.

The report of the characterization studies carried out by Dr. G. MORTEANI from the T.U.M. is shown in the appendix 3.

2.3. PREPARATION OF THE REO ORE SAMPLES FOR BENEFICIATION STUDIES

Materials were processed at laboratory scale by wet batch preparation on a few tens of kilogrammes, using basic flowsheets which include:

- attrition-scrubbing of a - 4 mm crushed material (REO ore samples n° 2 and 3) or the fine material as received (REO ore sample n° 1). Material with a particle size lower than the estimated liberation size is separated by wet sizing (160 μm for samples 1 and 2, 125 μm for sample 3) and then deslimed at 10 μm to produce primary - 10 μm (1) slimes and a deslimed primary fraction - 160 + 10 μm (1) or - 125 + 10 μm (1) for further treatment. This leads to lower losses of potentially valuable minerals in slimes with respect to conventional preparation involving direct grinding, sizing and desliming

- stage grinding in a rod mill of the primary oversize fraction separated from the attrition-scrubbed material so as to produce a final ground material with a minimum proportion (< 2 %) of oversize at the grinding size (160 μm for samples 1 and 2, 125 μm for sample 3). Stage grinding (including 4 to 5 stages) is expected to prevent excessive generation of slimes as achieved in a continuous closed grinding circuit. The final ground material is sized and deslimed to separate - 10 μm (2) secondary slimes from ground deslimed secondary fractions: - 160 + 10 μm (2) or - 125 + 10 μm (2), to be used in further processing tests.

2.3.1. Estimation of the liberation size for the REO ore sample n° 3

Investigations were made to estimate the liberation size for the REO ore sample n° 3, size fractions:

+ 0.5 mm (1)
- 0.5 + 0.125 mm (1)
- 0.125 + 0.04 mm (1)

separated from the attrition-scrubbed ore were subjected to heavy liquid separations at specific gravities of 3 and 3.3 g/cm^3, density fractions were analyzed for F, Ba, Y and REE (14 elements).

Size - assay analysis as well as material balances for primary preparation: attrition-scrubbing, sizing and desliming, are shown in tables 3H04 to 3H06.

Material balances for density fractionations are given in:

- tables 3H07 to 3H09 for size fraction + 0.5 mm (1)
- tables 3H10 to 3H12 for size fraction - 0.5 + 0.125 mm (1)
- tables 3H13 to 3H15 for size fraction - 0125 + 0.04 mm (1).

It can be seen that:

- liberation of fluorite, barite and REE bearing minerals improves with decreasing size, satisfactory liberation of both fluorite and barite occurs in the - 0.125 + 0.04 mm fraction. This led to the selection of 125 μm as a grinding size in stage grinding preparation

- distributions of Cerics (La to Sm) and Yttrics (Eu to Lu + Y) elements in both density and size fractions are not similar. This indicates that at least two types of REE bearing minerals occur in the REO ore sample n° 3, the first one would be low in heavy REE (Yttrics) while the second one would be higher in heavy REE and Y, it would also be more intricately associated to other minerals (barite, fluorite, silicates...) as shown by distributions of REE in density and size fractions

- more than 3/4 of the total REE + Y (77.3 %) report to the - 40 μm primary fraction, this clearly confirms the occurrence of most of the REE minerals as soft aggregates of extremely fine crystals as shown by microscopic observations. It should be noted that the recovery of cerics elements in the - 40 μm (1) fraction: 77.56 %, is much higher than that corresponding to yttrics elements: 56.02 %.

TABLE N° 3HD4 : RED ORE SAMPLE N° 3, REE OVERALL BALANCE RELEVANT TO THE PRIMARY ATTRITION-SCRUBBING STAGE.

PRODUCTS	WEIGHT	La		Ce		Pr		Nd	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADEppm	RECOVERY	GRADE %	RECOVERY
1 +0.5mm(1)	37.18	1.26	14.57	1.43	15.49	918.90	15.71	.20	15.88
2 -0.5+0.125mm(1)	21.45	.74	4.92	.84	5.26	538.10	5.31	.12	5.29
3 -.125+0.04mm(1)	8.93	.77	2.14	.85	2.22	557.30	2.29	.13	2.35
4 -0.040mm(1)	32.44	7.79	78.38	8.12	77.03	5140.50	76.69	1.13	76.48
CALCULATED HEAD	100.00	3.22	100.00	3.42	100.00	2174.41	100.00	.48	100.00

PRODUCTS	WEIGHT	Sm		Eu		Gd		Tb	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 +0.5mm(1)	37.18	147.10	17.20	35.30	18.07	70.10	18.42	8.75	18.76
2 -0.5+0.125mm(1)	21.45	85.90	5.79	25.30	7.47	46.00	6.97	5.90	7.30
3 -.125+0.04mm(1)	8.93	90.60	2.54	34.06	4.19	48.80	3.08	6.06	3.12
4 -0.040mm(1)	32.44	730.00	74.46	157.35	70.27	311.90	71.52	37.86	70.82
CALCULATED HEAD	100.00	318.02	100.00	72.64	100.00	141.47	100.00	17.34	100.00

TABLE N° 3HD4 : RED ORE SAMPLE N°3,REE BALANCE FOR ATTRITION-SCRUBBING.

PRODUCTS	WEIGHT	La		Ce		Pr		Nd	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADEppm	RECOVERY	GRADE %	RECOVERY
1 +0.040mm(1) 1 2 3 0 0 0	67.56	1.03	21.62	1.16	22.97	750.20	23.31	.17	23.52
2 -0.040mm(1) 4 0 0 0 0 0	32.44	7.79	78.38	8.12	77.03	5140.50	76.69	1.13	76.48

PRODUCTS	WEIGHT	Sm		Eu		Gd		Tb	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 +0.040mm(1) 1 2 3 0 0 0	67.56	120.20	25.54	31.96	29.73	59.63	28.48	7.49	29.18
2 -0.040mm(1) 4 0 0 0 0 0	32.44	730.00	74.46	157.35	70.27	311.90	71.52	37.86	70.82

TABLE N° 3H05 : REO ORE SAMPLE N° 3, REE OVERALL BALANCE RELEVANT TO THE PRIMARY ATTRITION-SCRUBBING STAGE.

PRODUCTS	WEIGHT	Dy		Ho		Er		Tm	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 +0.5mm(1)	37.18	35.80	21.25	7.15	22.20	21.22	22.11	2.70	24.30
2 -0.5+0.125mm(1)	21.45	24.90	8.53	5.80	10.39	17.00	10.22	2.10	10.90
3 -.125+0.04mm(1)	8.93	26.15	3.73	5.87	4.38	20.60	5.15	2.81	6.07
4 -0.040mm(1)	32.44	128.40	66.50	23.26	63.03	68.78	62.52	7.48	58.73
CALCULATED HEAD	100.00	62.64	100.00	11.97	100.00	35.69	100.00	4.13	100.00

PRODUCTS	WEIGHT	Yb		Lu		Y	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 +0.5mm(1)	37.18	20.77	27.05	2.48	27.08	333.20	30.85
2 -0.5+0.125mm(1)	21.45	15.70	11.80	2.20	13.86	313.00	16.72
3 -.125+0.04mm(1)	8.93	20.26	6.34	2.50	6.56	333.65	7.42
4 -0.040mm(1)	32.44	48.23	54.81	5.51	52.50	557.30	45.02
CALCULATED HEAD	100.00	28.54	100.00	3.40	100.00	401.61	100.00

TABLE N° 3H05 : REO ORE SAMPLE N°3,REE BALANCE FOR THE ATTRITION STAGE.

PRODUCTS	WEIGHT	Dy		Ho		Er		Tm	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 +0.040mm(1) 1 2 3 0 0 0	67.56	31.06	33.50	6.55	36.97	19.80	37.48	2.52	41.27
2 -0.040mm(1) 4 0 0 0 0 0	32.44	128.40	66.50	23.26	63.03	68.78	62.52	7.48	58.73

PRODUCTS	WEIGHT	Yb		Lu		Y	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 +0.040mm(1) 1 2 3 0 0 0	67.56	19.09	45.19	2.39	47.50	326.85	54.98
2 -0.040mm(1) 4 0 0 0 0 0	32.44	48.23	54.81	5.51	52.50	557.30	45.02

TABLE N° 3H06 : REO ORE SAMPLE N° 3, OVERALL MATERIAL BALANCE RELEVANT TO THE PRIMARY ATTRITION-SCRUBBING STAGE.

PRODUCTS	WEIGHT	F		Ba		Cerics		Yttrics	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 +0.5mm(1)	37.18	36.860	45.773	7.450	24.018	3.000	15.126	.054	25.637
2 -0.5+0.125mm(1)	21.45	38.120	27.310	9.920	18.450	1.759	5.117	.046	12.602
3 -.125+0.04mm(1)	8.93	18.500	5.518	31.690	24.538	1.814	2.197	.050	5.737
4 -0.040mm(1)	32.44	19.750	21.399	11.730	32.994	17.630	77.560	.135	56.024
ANALYSED HEAD	100.00	29.940	100.000	11.533	100.000	7.374	100.000	.078	100.000

PRODUCTS	WEIGHT	REE+Y	
	%	GRADE %	RECOVERY
1 +0.5mm(1)	37.18	3.054	15.236
2 -0.5+0.125mm(1)	21.45	1.805	5.195
3 -.125+0.04mm(1)	8.93	1.864	2.234
4 -0.040mm(1)	32.44	17.764	77.335
ANALYSED HEAD	100.00	7.452	100.000

PRODUCTS	WEIGHT %	Yttrics GRADE / REE+Y GRADE
1 +0.5mm(1)	37.18	.0176
2 -0.5+0.125mm(1)	21.45	.0254
3 -.125+0.04mm(1)	8.93	.0269
4 -0.040mm(1)	32.44	.0076
CALCULATED HEAD	100.00	.0105

TABLE N° 3H06 : RED ORE SAMPLE N°3,ATTRITION-SCRUBBING STAGE.

PRODUCTS	WEIGHT	F		Ba		Cerics		Yttrics	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 +0.040mm(t) 1 2 3 0 0 0	67.56	34.833	78.601	11.438	67.006	2.449	22.440	.051	43.976
2 -0.040mm(1) 4 0 0 0 0 0	32.44	19.750	21.399	11.730	32.994	17.630	77.560	.135	56.024

PRODUCTS	WEIGHT	REE+Y	
	%	GRADE %	RECOVERY
1 +0.040mm(t) 1 2 3 0 0 0	67.56	2.500	22.665
2 -0.040mm(1) 4 0 0 0 0 0	32.44	17.764	77.335

TABLE N° 3H07 : REO ORE SAMPLE N° 3, HEAVY LIQUID SEPARATIONS ON THE +0.5mm(1) PRIMARY SIZE FRACTION.

PRODUCTS	WEIGHT	La		Ce		Pr		Nd	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
d>3.3	23.32	3.58	66.09	4.15	67.90	2562.00	65.02	5626.00	64.05
3<d<3.3	71.54	.57	32.28	.61	30.62	427.70	33.30	979.30	34.20
d<3	5.14	.40	1.63	.41	1.48	300.20	1.68	695.10	1.74
ALCULATED HEAD	100.00	1.26	100.00	1.43	100.00	918.87	100.00	2048.30	100.00

PRODUCTS	WEIGHT	Sm		Eu		Gd		Tb	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
d>3.3	23.32	390.20	61.84	91.50	60.46	181.30	60.26	23.60	62.93
3<d<3.3	71.54	74.40	36.17	18.30	37.09	36.10	36.81	4.10	33.54
d<3	5.14	56.70	1.98	16.80	2.45	39.90	2.92	6.00	3.53
ALCULATED HEAD	100.00	147.13	100.00	35.29	100.00	70.16	100.00	8.75	100.00

TABLE N° 3H08 : RED ORE SAMPLE N° 3, HEAVY LIQUID SEPAPRATIONS ON THE +0.5mm(1) PRIMARY SIZE FRACTION.

PRODUCTS	WEIGHT	Dy		Ho		Er		Tm	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 d>3.3	23.32	72.30	47.11	12.10	39.48	34.60	38.01	3.40	29.31
2 3<d<3.3	71.54	24.40	48.78	5.50	55.05	16.30	54.94	2.40	63.47
3 d<3	5.14	28.60	4.11	7.60	5.47	29.10	7.05	3.80	7.22
CALCULATED HEAD	100.00	35.79	100.00	7.15	100.00	21.23	100.00	2.71	100.00

PRODUCTS	WEIGHT	Yb		Lu		Y	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 d>3.3	23.32	30.00	33.68	3.80	35.71	382.30	26.76
2 3<d<3.3	71.54	17.00	58.55	1.90	54.77	305.40	65.57
3 d<3	5.14	31.40	7.77	4.60	9.53	497.20	7.67
CALCULATED HEAD	100.00	20.77	100.00	2.48	100.00	333.19	100.00

TABLE N° 3H09 : RED ORE SAMPLE N° 3,HEAVY LIQUID SEPARATIONS ON THE +0.5mm(1) PRIMARY SIZE FRACTION.

PRODUCTS	WEIGHT	F		Ba		Cerics		Yttrics	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
d>3.3	23.32	19.700	12.465	19.700	61.670	8.588	66.759	.083	36.223
3<d<3.3	71.54	42.100	81.719	3.780	36.301	1.328	31.673	.043	57.418
d<3	5.14	41.700	5.816	2.940	2.029	.915	1.568	.067	6.359
ALYSED HEAD	100.00	36.856	100.000	7.449	100.000	3.000	100.000	.054	100.000

PRODUCTS	WEIGHT	REE+Y	
	%	GRADE %	RECOVERY
d>3.3	23.32	8.671	66.221
3<d<3.3	71.54	1.371	32.126
d<3	5.14	.982	1.652
ALYSED HEAD	100.00	3.054	100.000

PRODUCTS	WEIGHT %	Yttrics GRADE / REE+Y GRADE
d>3.3	23.32	.0096
3<d<3.3	71.54	.0315
d<3	5.14	.0677
LCULATED HEAD	100.00	.0176

TABLE N° 3H10 : RED ORE SAMPLE N° 3, HEAVY LIQUID SEPARATIONS ON THE -0.5+0.125mm(1) PRIMARY SIZE FRACTION.

PRODUCTS	WEIGHT	La		Ce		Pr		Nd	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 d>3.3	25.79	2.15	75.04	2.51	77.13	1540.00	73.81	3391.00	73.87
2 3<d<3.3	72.59	.25	24.56	.26	22.49	191.10	25.78	419.20	25.70
3 d<3	1.62	.19	.41	.20	.38	135.00	.41	310.00	.42
CALCULATED HEAD	100.00	.74	100.00	.84	100.00	538.07	100.00	1183.86	100.00

PRODUCTS	WEIGHT	Sm		Eu		Gd		Tb	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 d>3.3	25.79	249.90	74.74	72.60	73.94	103.40	57.98	13.00	56.89
2 3<d<3.3	72.59	29.50	24.83	8.90	25.51	26.00	41.03	3.40	41.88
3 d<3	1.62	23.00	.43	8.50	.54	28.00	.99	4.50	1.24
CALCULATED HEAD	100.00	86.24	100.00	25.32	100.00	45.99	100.00	5.89	100.00

TABLE N° 3H11 : REO ORE SAMPLE N° 3, HEAVY LIQUID SEPAPRATIONS ON THE -0.5+0.125mm(1) PRIMARY SIZE FRACTION.

PRODUCTS	WEIGHT	Dy		Ho		Er		Tm	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
d>3.3	25.79	42.80	44.37	9.90	44.17	21.70	32.89	2.90	35.56
3<d<3.3	72.59	18.60	54.27	4.30	54.00	15.30	65.28	1.80	62.13
d<3	1.62	20.80	1.35	6.50	1.82	19.20	1.83	3.00	2.31
LCULATED HEAD	100.00	24.88	100.00	5.78	100.00	17.01	100.00	2.10	100.00

PRODUCTS	WEIGHT	Yb		Lu		Y	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
d>3.3	25.79	23.20	38.16	2.80	32.37	294.60	24.27
3<d<3.3	72.59	12.80	59.25	2.00	65.09	317.30	73.58
d<3	1.62	25.10	2.59	3.50	2.54	415.00	2.15
LCULATED HEAD	100.00	15.68	100.00	2.23	100.00	313.03	100.00

TABLE N° 3H12 : REO ORE SAMPLE N° 3,HEAVY LIQUID SEPARATIONS ON THE -0.5+0.125mm(1) PRIMARY SIZE FRACTION.

PRODUCTS	WEIGHT	F		Ba		Cerics		Yttrics	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 d>3.3	25.79	14.100	9.539	35.120	91.345	5.178	75.920	.059	33.054
2 3<d<3.3	72.59	46.600	88.739	1.160	8.492	.574	23.687	.041	65.057
3 d<3	1.62	40.500	1.721	1.000	.163	.427	.393	.053	1.889
ANALYSED HEAD	100.00	38.119	100.000	9.916	100.000	1.759	100.000	.046	100.000

PRODUCTS	WEIGHT	REE+Y	
	%	GRADE %	RECOVERY
1 d>3.3	25.79	5.237	74.832
2 3<d<3.3	72.59	.615	24.737
3 d<3	1.62	.480	.431
ANALYSED HEAD	100.00	1.805	100.000

PRODUCTS	WEIGHT %	Yttrics GRADE / REE+Y GRADE
1 d>3.3	25.79	.0112
2 3<d<3.3	72.59	.0667
3 d<3	1.62	.1112
CALCULATED HEAD	100.00	.0254

TABLE N° 3H13 : RED ORE SAMPLE N° 3, HEAVY LIQUID SEPARATIONS ON THE -0.125+0.04mm(1) PRIMARY SIZE FRACTION.

PRODUCTS	WEIGHT	La		Ce		Pr		Nd	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
d>3.3	64.50	1.02	85.31	1.14	86.29	734.80	85.04	1663.00	84.97
3<d<3.3	34.50	.32	14.32	.33	13.36	236.10	14.62	537.50	14.69
d<3	1.00	.28	.37	.30	.35	189.00	.34	431.00	.34
CALCULATED HEAD	100.00	.77	100.00	.85	100.00	557.29	100.00	1262.38	100.00

PRODUCTS	WEIGHT	Sm		Eu		Gd		Tb	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
d>3.3	64.50	117.90	83.93	46.80	88.62	59.60	78.81	7.30	77.68
3<d<3.3	34.50	41.10	15.65	10.90	11.04	29.00	20.51	3.80	21.63
d<3	1.00	38.00	.42	11.50	.34	33.00	.68	4.20	.69
CALCULATED HEAD	100.00	90.61	100.00	34.06	100.00	48.78	100.00	6.06	100.00

TABLE N° 3H14 : RED ORE SAMPLE N° 3, HEAVY LIQUID SEPAPRATIONS ON THE -0.125+0.04mm(1) PRIMARY SIZE FRACTION.

PRODUCTS	WEIGHT	Dy		Ho		Er		Tm	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 d>3.3	64.50	29.00	71.52	6.30	69.21	18.80	58.86	2.60	59.76
2 3<d<3.3	34.50	21.00	27.70	5.10	29.97	23.90	40.02	3.20	39.34
3 d<3	1.00	20.50	.78	4.80	.82	23.00	1.12	2.50	.89
CALCULATED HEAD	100.00	26.16	100.00	5.87	100.00	20.60	100.00	2.81	100.00

PRODUCTS	WEIGHT	Yb		Lu		Y	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 d>3.3	64.50	19.40	61.75	2.50	64.45	300.40	58.07
2 3<d<3.3	34.50	21.80	37.12	2.50	34.47	396.80	41.03
3 d<3	1.00	23.00	1.14	2.70	1.08	300.50	.90
CALCULATED HEAD	100.00	20.26	100.00	2.50	100.00	333.66	100.00

TABLE N° 3H15 : REO ORE SAMPLE N° 3,HEAVY LIQUID SEPARATIONS ON THE -0.125+0.04mm(1) PRIMARY SIZE FRACTION.

PRODUCTS	WEIGHT	F		Ba		Cerics		Yttrics	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
d>3.3	64.50	4.600	16.036	48.050	97.789	2.412	85.727	.049	63.462
3<d<3.3	34.50	43.800	81.672	2.000	2.177	.731	13.908	.052	35.688
d<3	1.00	42.400	2.292	1.070	.034	.661	.364	.043	.850
NALYSED HEAD	100.00	18.502	100.000	31.693	100.000	1.814	100.000	.050	100.000

PRODUCTS	WEIGHT	REE+Y	
	%	GRADE %	RECOVERY
d>3.3	64.50	2.461	85.129
3<d<3.3	34.50	.783	14.493
d<3	1.00	.703	.377
NALYSED HEAD	100.00	1.865	100.000

PRODUCTS	WEIGHT %	Yttrics GRADE	REE+Y / GRADE
d>3.3	64.50		.0200
3<d<3.3	34.50		.0661
d<3	1.00		.0605
ALCULATED HEAD	100.00		.0269

2.3.2. Flowsheets and size distributions

Flowsheets for preparation of REO ore samples n° 1, 2 and 3 are presented in figures 1HP01, 2HP01 and 3H01.

Ore samples 2 and 3, as received, were crushed down to about 12 mm in a jaw crusher, then down to about 4 mm in a roll crusher.

- 4 mm materials (samples 2 and 3) and the ore sample n° 1, as received, were attrition-scrubbed in a stainless steel WEMCO laboratory attrition cell of 7 l capacity under the following operating conditions:

  - solids concentration 65 % by weight
  - peripheral speed: 5.1 m/s at the tip of the impellers
  - time: 3 minutes (samples 1 and 2) or 5 minutes (samples 3).

High density pulps discharged from the attrition cell were wet sized on a multi deck vibrating screen of 400 mm diameter (TONINDUSTRIE) or 600 mm diameter (CHAUVIN) equipped with stainless steel cloths of 0.5, 0.16 and 0.05 mm mesh size (samples 1 and 2) or 0.4 and 0.125 mm (sample 3), oversize materials were cleaned by high pressure water sprays.

The fine fractions - 0.05 or - 0.125 mm (1) primary fractions were deslimed in two or three stages at about 10 μm, using 25 mm inner diameter porcelain hydrocyclones DORR OLIVER T54B, an inlet pressure of 2 bars and a solids concentration in feed pulp of 15 %.

+ 0.16 mm (samples 1 and 2) or + 0.125 mm (sample 3) (1) primary fractions were wet ground down to about 0.16 mm (samples 1 and 2) or 0.125 mm (sample 3), in 5 stages (ore sample 1) or 4 stages (ore samples 2 and 3), using:

- a 72 l capacity batch rod mill (SPMB) under the following operating conditions:

  - solids concentration: 65 %
  - weight ratio: rods/dry material: 14
  - diameter of rods: 20 and 30 mm (made of Mn steel)
  - speed of rotation: 68.6 % of the critical speed
  - time: 5 minutes per stage

  in stages 1 and 2. The ground materials discharged from the mill were wet sized at 0.5 mm, 0.16 and 0.05 mm (samples 1 and 2) or 0.5 and 0.125 mm (sample 3) on a vibrating screen (TONINDUSTRIE or CHAUVIN) + 0.16 mm or + 0.125 mm oversize fractions were fed to the mill to be reground in the next stage.

- a 10 l capacity batch rod mill (PASCALL ENGINEERING) in stages 3 and 4 (samples 2 and 3) or 3 to 5 (sample 1). The oversize materials (+ 0.16 or + 0.125 mm) separated after completion of stage 2 in the 72 l mill, were stage ground down to 0.16 or 0.125 mm, under the following operating conditions:

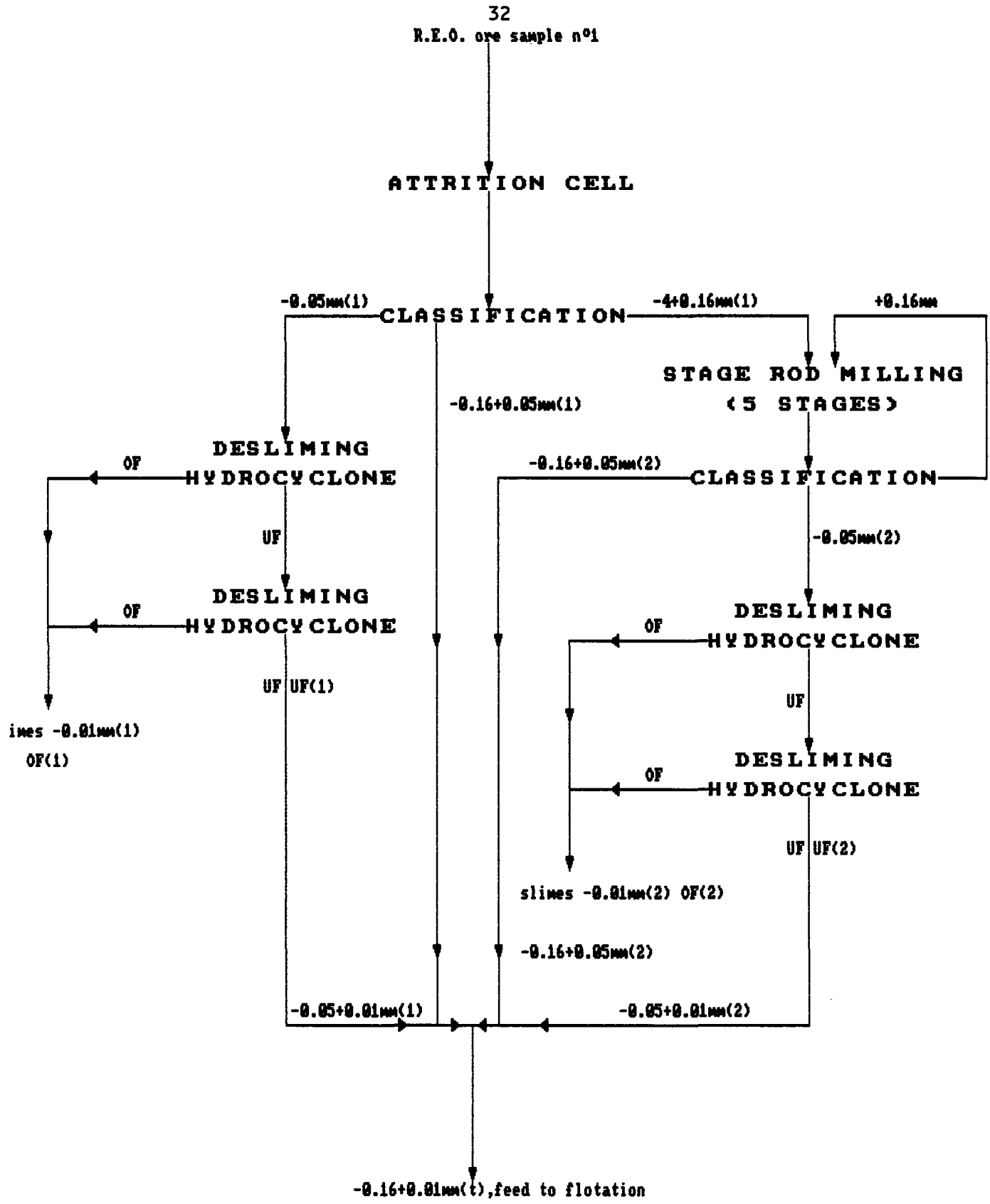


figure n° 1HP01 : Basic flowsheet for ore preparation.R.E.O. ore sample n°1.

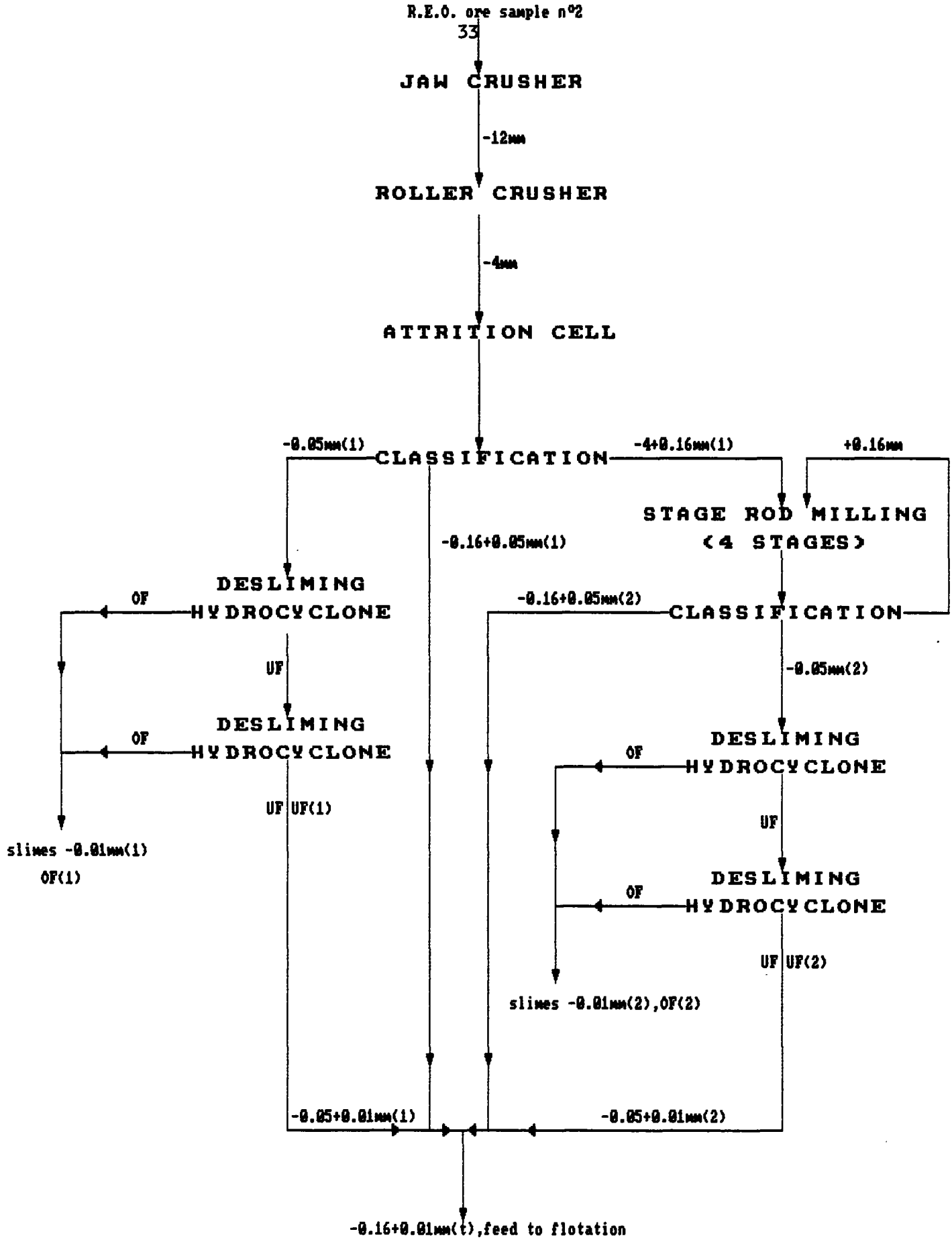


figure n° 2HP01 : Basic flowsheet for ore preparation.R.E.O. ore sample n°2.

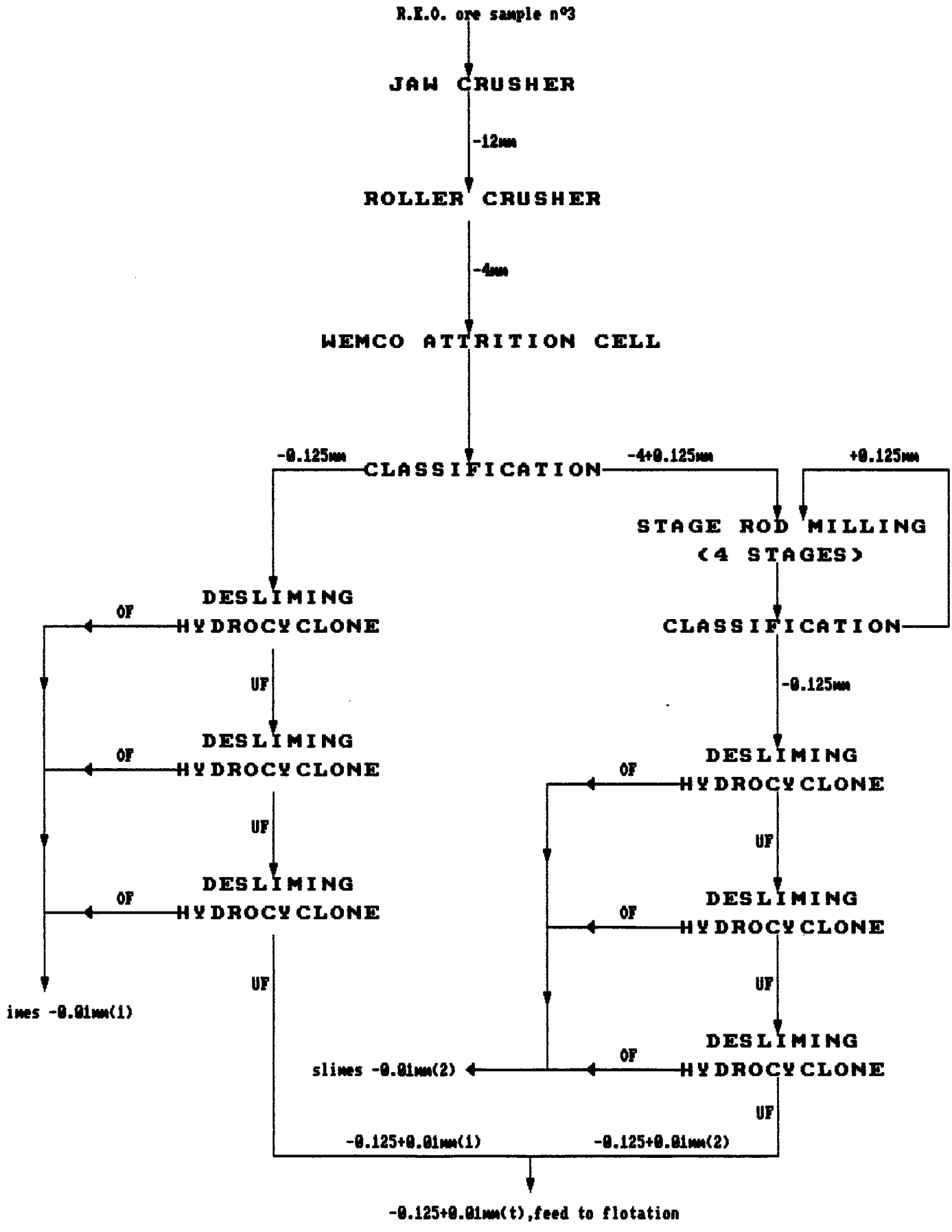


figure n° 3H01 : Basic flowsheet for ore preparation.R.E.O. ore sample n°3.

solids concentration: 65 %
weight ratio: rods/dry material: 14
diameter of rods: 20 mm (made of stainless steel)
speed of rotation: 68.6 % of the critical speed
time: 5 minutes per stage.

Composite secondary fine fractions - 0.05 or - 0.125 mm (2), separated from the ground materials, were deslimed at about 10 μm using operating conditions already used for desliming primary fractions.

Primary and secondary slimes, - 0.010 mm (1) separated from the attrition-scrubbed materials, - 0,010 mm (2) separated from the stage rod-milled materials, were sampled on line using a continuous cutter sampler. Other products: cyclone underflows and screen oversizes were sampled using a chute riffle splitter, then blended and homogeneized to constitute composite fractions:

- 0.16 + 0.01 mm (t) (samples 1 and 2)
- 0.125 + 0.01 mm (t) (sample 3)

to be used in further treatments. Final screen oversizes: 0.16 mm or + 0.125 mm, representing minor portions of the initial materials were incorporated with the major undersize deslimed fractions - 0.16 + 0.01 mm (t) or - 0.125 + 0.01 mm (t).

Mass balances relevant to wet primary and secondary preparations are shown in the table as follows:

	REO ore sample			REO ore sample
	n° 1 weight %	n° 2 weight %		n° 3 weight %
stage rod milling			stage rod milling	
+ 0.16 mm(2)	1.44	1.90	+ 0.125 mm(2)	0.58
- 0.16 + 0.05 mm(2)	17.53	43.10	- 0.125 + 0.01 mm(2)	48.97
- 0.05 + 0.01 mm(2)	2.58	21.66	- 0.01 mm(2)	9.08
- 0.01 mm(2)	0.44	5.99		
attrition-scrubbing			attrition-scrubbing	
- 0.16 + 0.05 mm(1)	45.49	10.25	- 0.125 + 0.01 mm(1)	25.02
- 0.05 + 0.01 mm(1)	28.32	11.90	- 0.01 mm(1)	16.35
- 0.01 mm(1)	4.20	5.20		
head	100.00	100.00		100.00

Size distribution of the attrition-scrubbed REO ore sample 3 was determined by wet sieving, results were as follows:

Screen mesh µm	Weight %	Cumulative undersize weight %
1 000	20.15	79.85
500	17.03	62.82
250	12.98	49.84
160	5.74	44.10
125	2.73	41.37
80	4.02	37.35
63	1.70	35.65
40	3.21	32.44
20	5.84	26.60
0	26.60	0

Size distributions of fractions separated by wet screening and by desliming hydrocyclones, determined by laser diffractometry (CILAS 715), are shown as cumulative undersize distributions and tables at the end of paragraph 2.3.2. Main characteristics of size distributions are given in the following table:

	d20 µm	d50 µm	d80* µm
REO ore sample 1			
primary slimes OF(1)	2.3	5.6	11.3
cyclone underflow UF(1)	15.5	27.7	42.5
secondary slimes OF(2)	2.8	6.4	14.5
cyclone underflow UF(2)	17.0	33.0	57.1
- 0.16 + 0.01 mm (t)	33.2	83.2	114.8
REO ore sample 2			
OF(1)	1.4	3.5	6.4
UF(1)	11.1	20.8	36.6
OF(2)	1.6	4.1	7.8
UF(2)	13.1	23.4	39.7
UF(1) + UF(2)	13.1	24.8	40.9
REO ore sample 3			
OF(1)	1.5	2.7	4.8
UF(1)	8.8	22.2	63.4
OF(2)	1.9	3.5	6.1
UF(2)	14.3	34.7	79.6
- 0.125 + 0.01 mm (t)	13.3	33.8	81.9

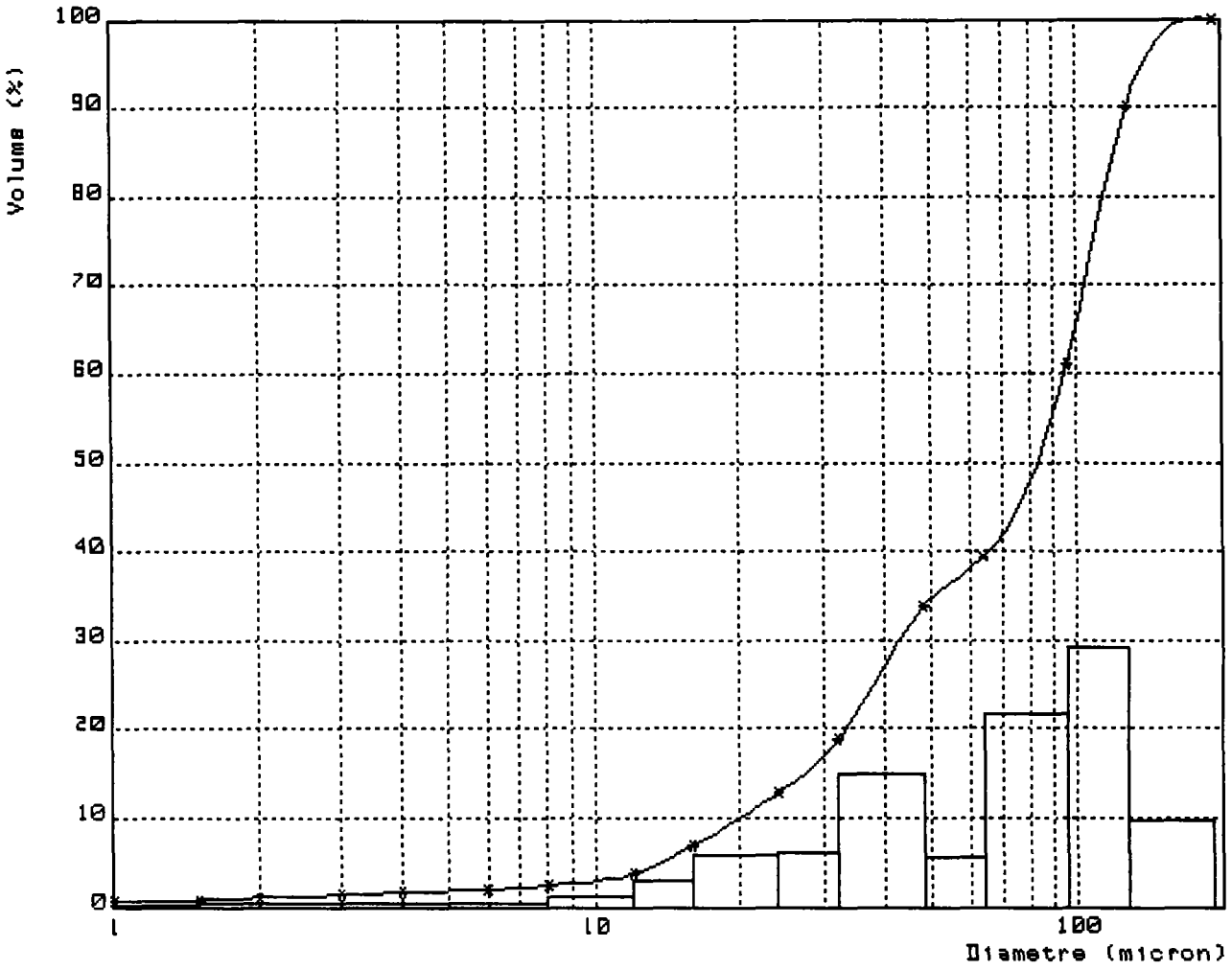
* dx indicates diameter corresponding to x % undersize by weight

It should be noted that the - 0.16 + 0.01 mm (t) fraction separated from the attrition-scrubbed and rod milled REO ore sample 1 was subjected to low intensity magnetic separation (L.I.M.S.) using a SALA drum separator, equipped with permanent magnets, at a feed flow rate of 120 l/h and solids concentration of 20 %, so as to extract magnetite particles. The mass balance for L.I.M.S. was as follows:

	% by weight
- 0.16 + 0.01 mm M, magnetic tails	9.02
- 0.16 + 0.01 mm NM, non magnetics used as feed to flotation	90.98
feed to L.I.M.S.	100.00

Granulomètre à laser CILAS 715

REO ORE SAMPLE N°1
Echantillon : REO1 FF
Durée d'ultrasons : 20 s
Commentaire : FEED TO FLOTATION -0.16+0.010mm(t)



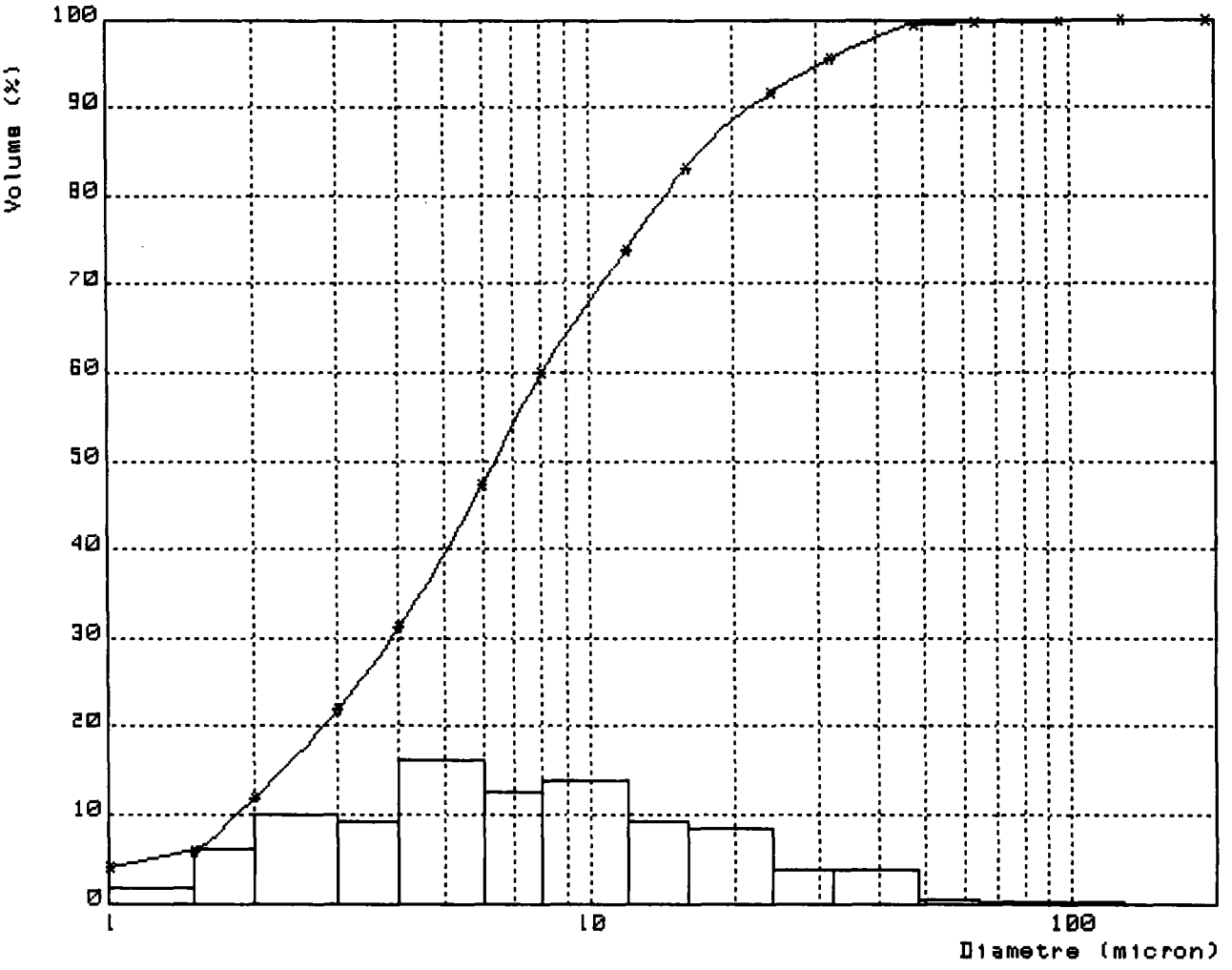
d20 = 33.2 d50 = 83.2 d80 = 114.8

HISTOGRAMME	
Classe	Volume %
<1	.7
1-1.5	.1
1.5-2	.3
2-3	.3
3-4	.3
4-6	.3
6-8	.5
8-12	1.3
12-16	3.1
16-24	5.9
24-32	6.0
32-48	15.0
48-64	5.6
64-96	21.7
96-128	29.1
128-192	9.8

PASSANTS CUMULES	
Diamètre	Volume %
1	.7
1.5	.8
2	1.1
3	1.4
4	1.7
6	2.0
8	2.5
12	3.8
16	6.9
24	12.8
32	18.8
48	33.8
64	39.4
96	61.1
128	90.2
192	100.0

Sous-fichier REO1 FF__1 2619
Fichier ALFL:F *MAGALI* BRGM/DAM/MIN 415

REO ORE SAMPLE N°1
Echantillon : OF(2)
Liquide Porteur : EAU
Dispersant : HMP+DISPEX 3kg/t
Durée d'ultrasons : 40 s
Commentaire : SLIMES -0.010mm(2)



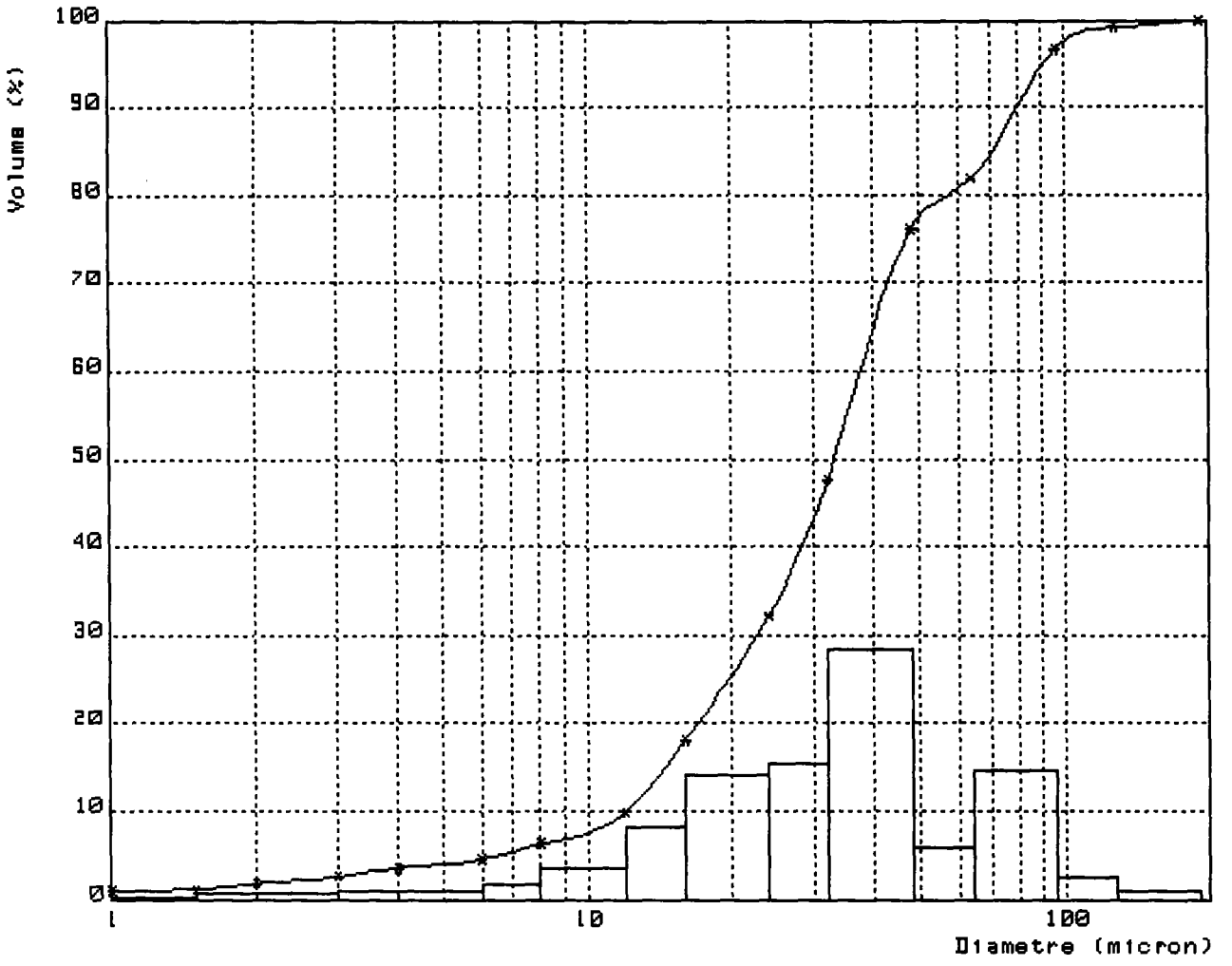
d20 = 2.8 d50 = 6.4 d80 = 14.5

HISTOGRAMME	
Classe	Volume %
<1	4.0
1-1.5	1.8
1.5-2	6.1
2-3	10.0
3-4	9.3
4-6	16.1
6-8	12.7
8-12	13.8
12-16	9.3
16-24	8.5
24-32	3.9
32-48	3.8
48-64	.3
64-96	.2
96-128	.2
128-192	0.0

PASSANTS CUMULES	
Diamètre	Volume %
1	4.0
1.5	5.8
2	11.9
3	21.9
4	31.2
6	47.3
8	60.0
12	73.8
16	83.1
24	91.6
32	95.5
48	99.3
64	99.6
96	99.8
128	100.0
192	100.0

Granulomètre à laser CILAS 715

REO ORE SAMPLE N°1
Echantillon : UF(2)
Dispersant : HMP:3Kg/T
Durée d'ultrasons : 20 s
Commentaire : -0.05+.010mm(2)



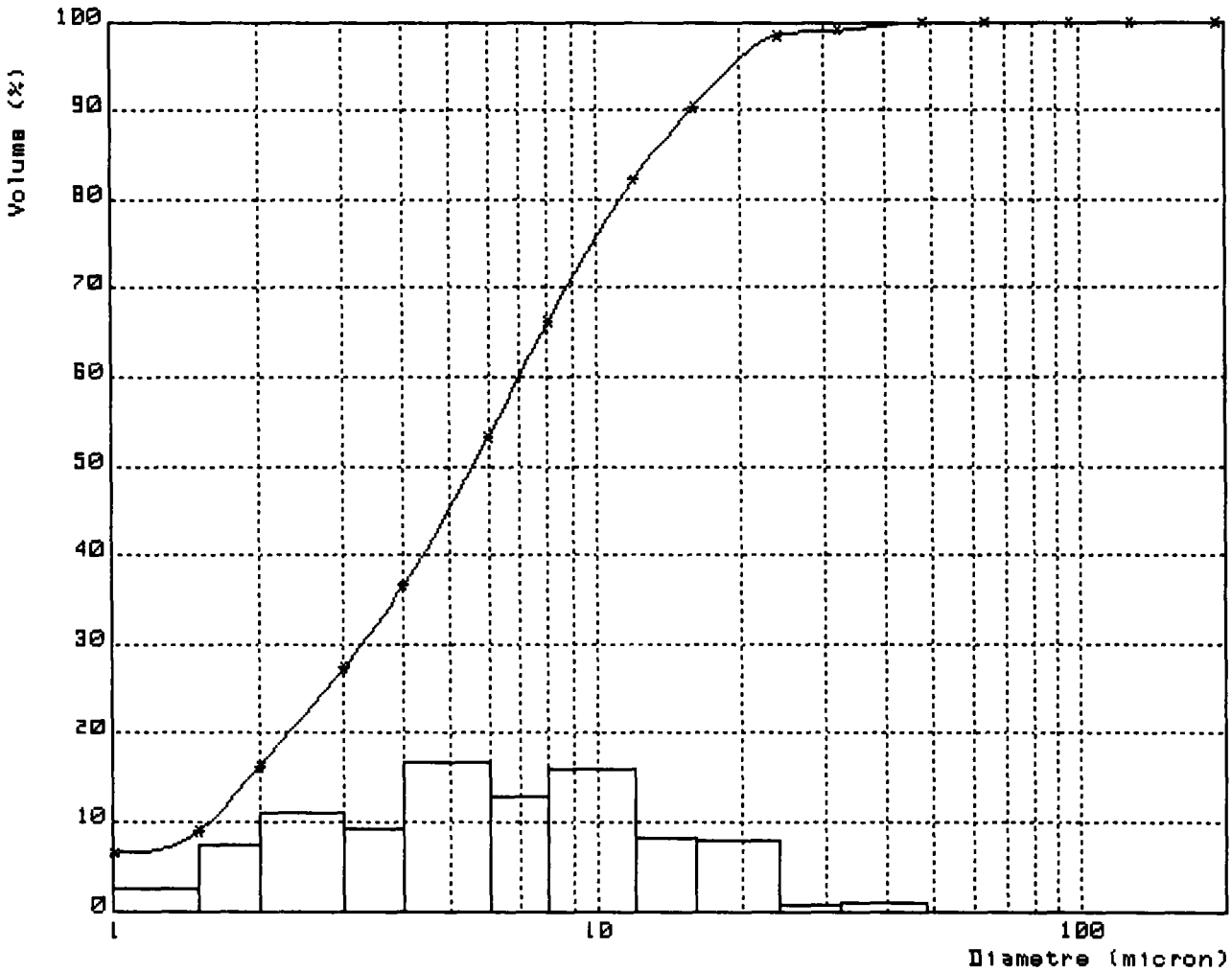
d20 = 17.0 d50 = 33.0 d80 = 57.1

HISTOGRAMME	
Classe	Volume %
<1	.9
1-1.5	.2
1.5-2	.7
2-3	.8
3-4	1.0
4-6	.9
6-8	1.8
8-12	3.6
12-16	8.2
16-24	14.1
24-32	15.5
32-48	28.4
48-64	5.8
64-96	14.7
96-128	2.5
128-192	.9

PASSANTS CUMULES	
Diamètre	Volume %
1	.9
1.5	1.1
2	1.8
3	2.6
4	3.6
6	4.5
8	6.3
12	9.9
16	18.1
24	32.2
32	47.7
48	76.1
64	81.9
96	96.6
128	99.1
192	100.0

Granulomètre à laser CILAS 715

REO ORE SAMPLE N°1
Echantillon : OF(1)
Liquide Porteur : EAU
Dispersant : HMP + DISPEX 3kg/t
Durée d'ultrasons : 40s
Commentaire : SLIMES -0.010mm(1)



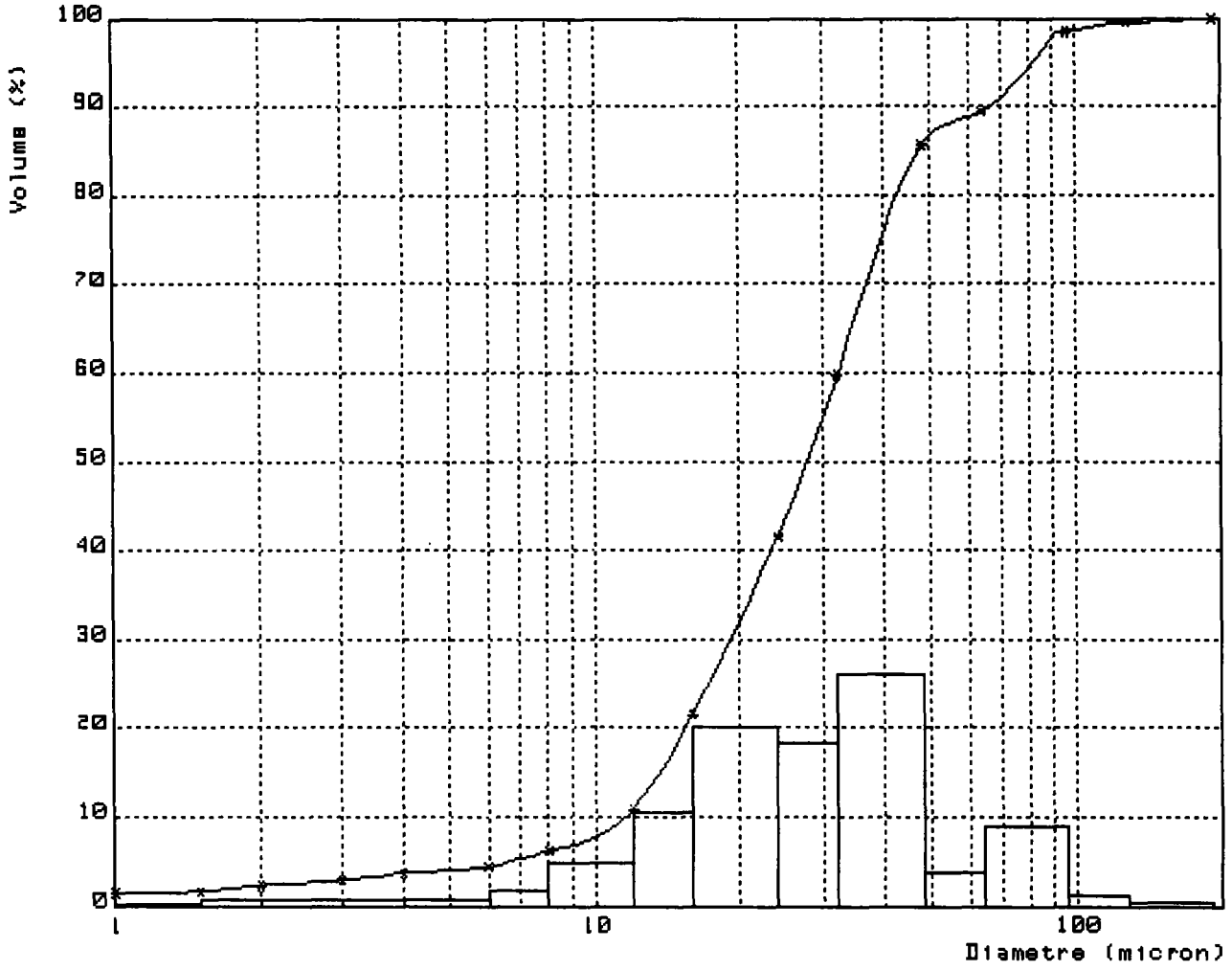
d20 = 2.3 d50 = 5.6 d80 = 11.3

HISTOGRAMME	
Classe	Volume %
<1	6.5
1-1.5	2.5
1.5-2	7.3
2-3	11.0
3-4	9.3
4-6	16.7
6-8	12.9
8-12	16.0
12-16	8.2
16-24	8.0
24-32	.6
32-48	1.0
48-64	0.0
64-96	0.0
96-128	0.0
128-192	0.0

PASSANTS CUMULES	
Diamètre	Volume %
1	6.5
1.5	9.0
2	16.3
3	27.3
4	36.6
6	53.3
8	66.2
12	82.2
16	90.4
24	98.4
32	99.0
48	100.0
64	100.0
96	100.0
128	100.0
192	100.0

Sous-fichier OF(1)__3

REO ORE SAMPLE N°1 42
Echantillon : UF(1)
Dispersant : HMP 3Kg/t
Durée d'ultrasons : 20 s
Commentaire : -0.05+0.01mm(1)



d20 = 15.5 d50 = 27.7 d80 = 42.5

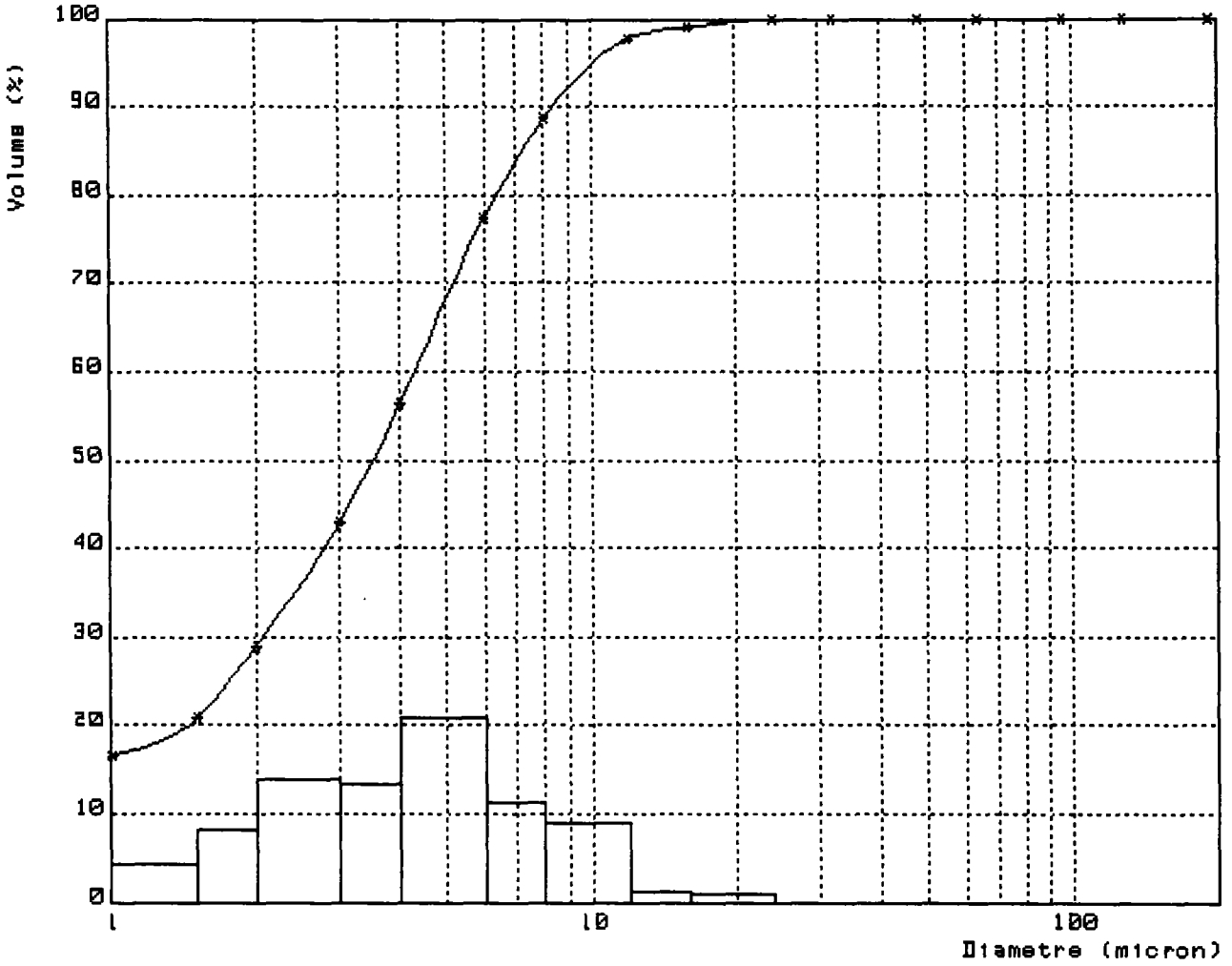
HISTOGRAMME

Classe	Volume %
<1	1.4
1-1.5	.2
1.5-2	.7
2-3	.6
3-4	.8
4-6	.7
6-8	1.8
8-12	4.7
12-16	10.6
16-24	20.0
24-32	18.2
32-48	26.0
48-64	3.7
64-96	8.9
96-128	1.2
128-192	.5

PASSANTS CUMULES

Diamètre	Volume %
1	1.4
1.5	1.6
2	2.3
3	2.9
4	3.7
6	4.4
8	6.2
12	10.9
16	21.5
24	41.5
32	59.7
48	85.7
64	89.4
96	98.3
128	99.5
192	100.0

REO ORE SAMPLE N°2
Echantillon : REO2,OF(1)
Dispersant : HMP:3Kg/T
Durée d'ultrasons : 20 s
Commentaire : SLIMES -0.010mm(1)

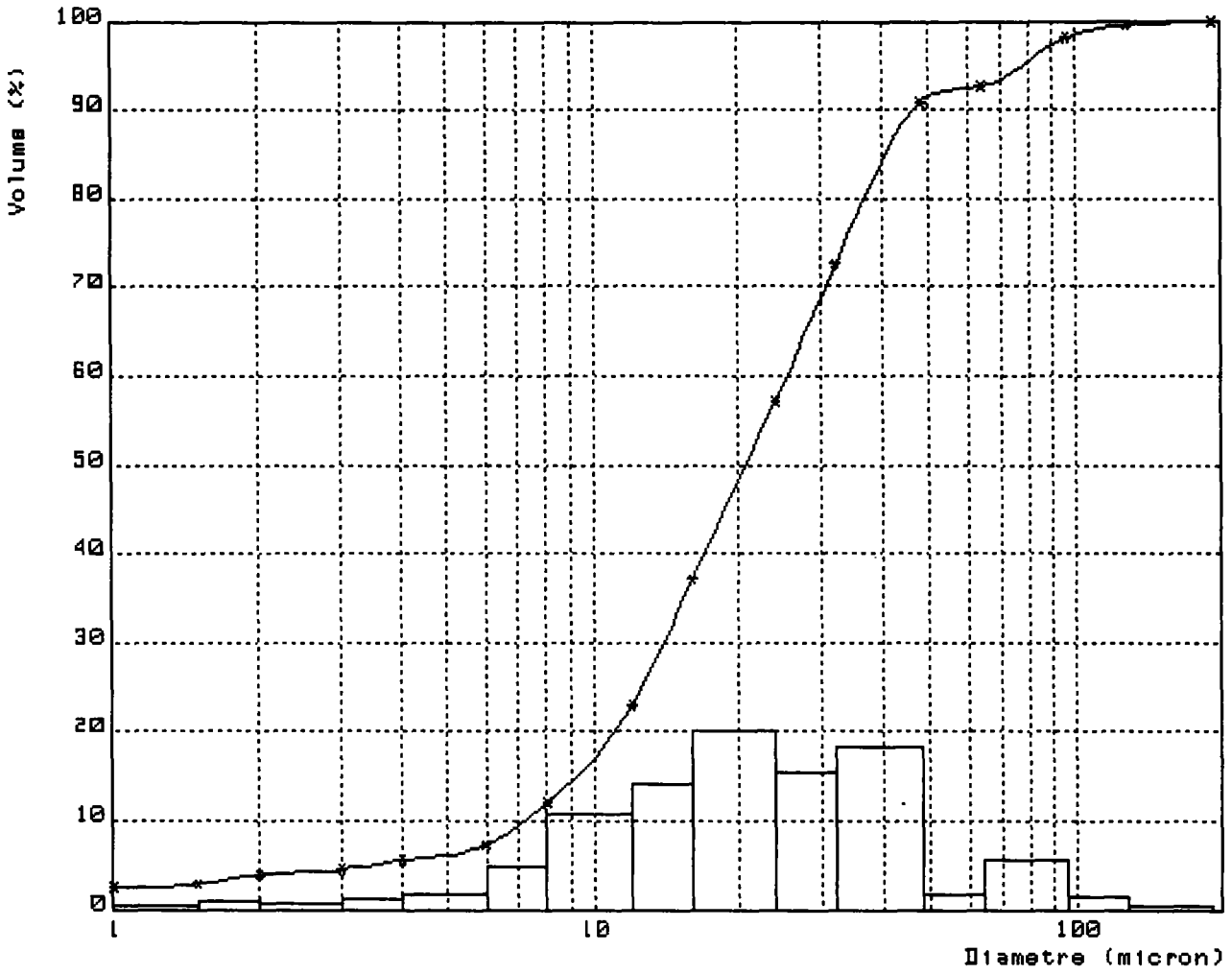


d20 = 1.4 d50 = 3.5 d80 = 6.4

HISTOGRAMME	
Classe	Volume %
<1	16.6
1-1.5	4.3
1.5-2	8.1
2-3	14.0
3-4	13.4
4-6	21.0
6-8	11.3
8-12	9.0
12-16	1.3
16-24	1.0
24-32	0.0
32-48	0.0
48-64	0.0
64-96	0.0
96-128	0.0
128-192	0.0

PASSANTS CUMULES	
Diamètre	Volume %
1	16.6
1.5	20.9
2	29.0
3	43.0
4	56.4
6	77.4
8	88.7
12	97.7
16	99.0
24	100.0
32	100.0
48	100.0
64	100.0
96	100.0
128	100.0
192	100.0

REO ORE SAMPLE N°2
Echantillon : REO2,UF(1)
Dispersant : HMP:3Kg/T
Durée d'ultrasons : 20 s
Commentaire : -0.05+0.010mm(1)

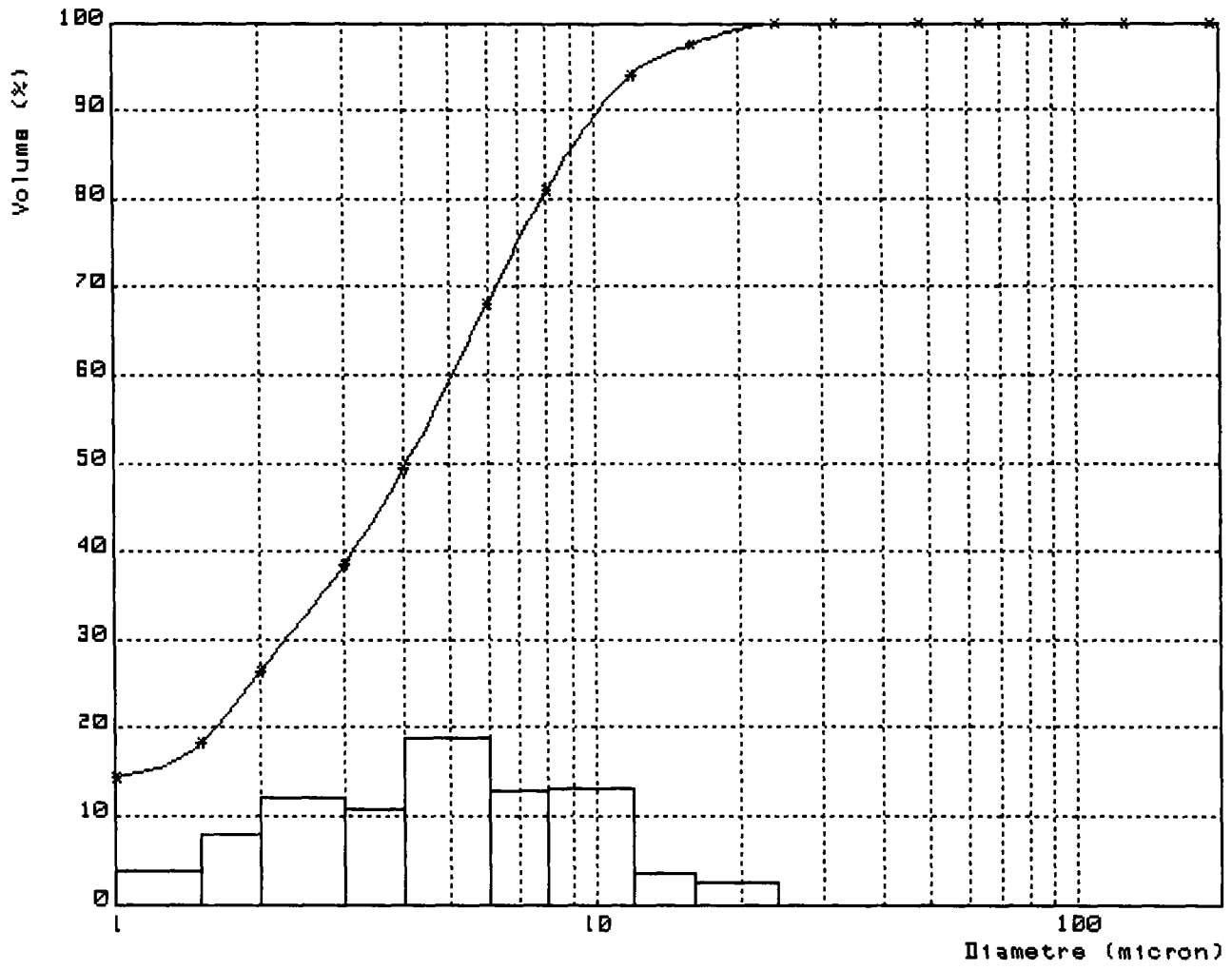


d20 = 11.1 d50 = 20.8 d80 = 36.6

HISTOGRAMME	
Classe	Volume %
<1	2.5
1-1.5	.4
1.5-2	1.0
2-3	.6
3-4	1.1
4-6	1.7
6-8	4.8
8-12	10.9
12-16	14.1
16-24	20.1
24-32	15.4
32-48	18.3
48-64	1.7
64-96	5.5
96-128	1.4
128-192	.5

PASSANTS CUMULES	
Diamètre	Volume %
1	2.5
1.5	2.9
2	3.9
3	4.5
4	5.6
6	7.3
8	12.1
12	23.0
16	37.1
24	57.2
32	72.6
48	90.9
64	92.6
96	98.1
128	99.5
192	100.0

REO ORE SAMPLE N°2
Echantillon : REO2,OF(2).
Dispersant : HMP:3kg/t
Durée d'ultrasons : 20 s
Commentaire : SLIMES -0.010mm(2)

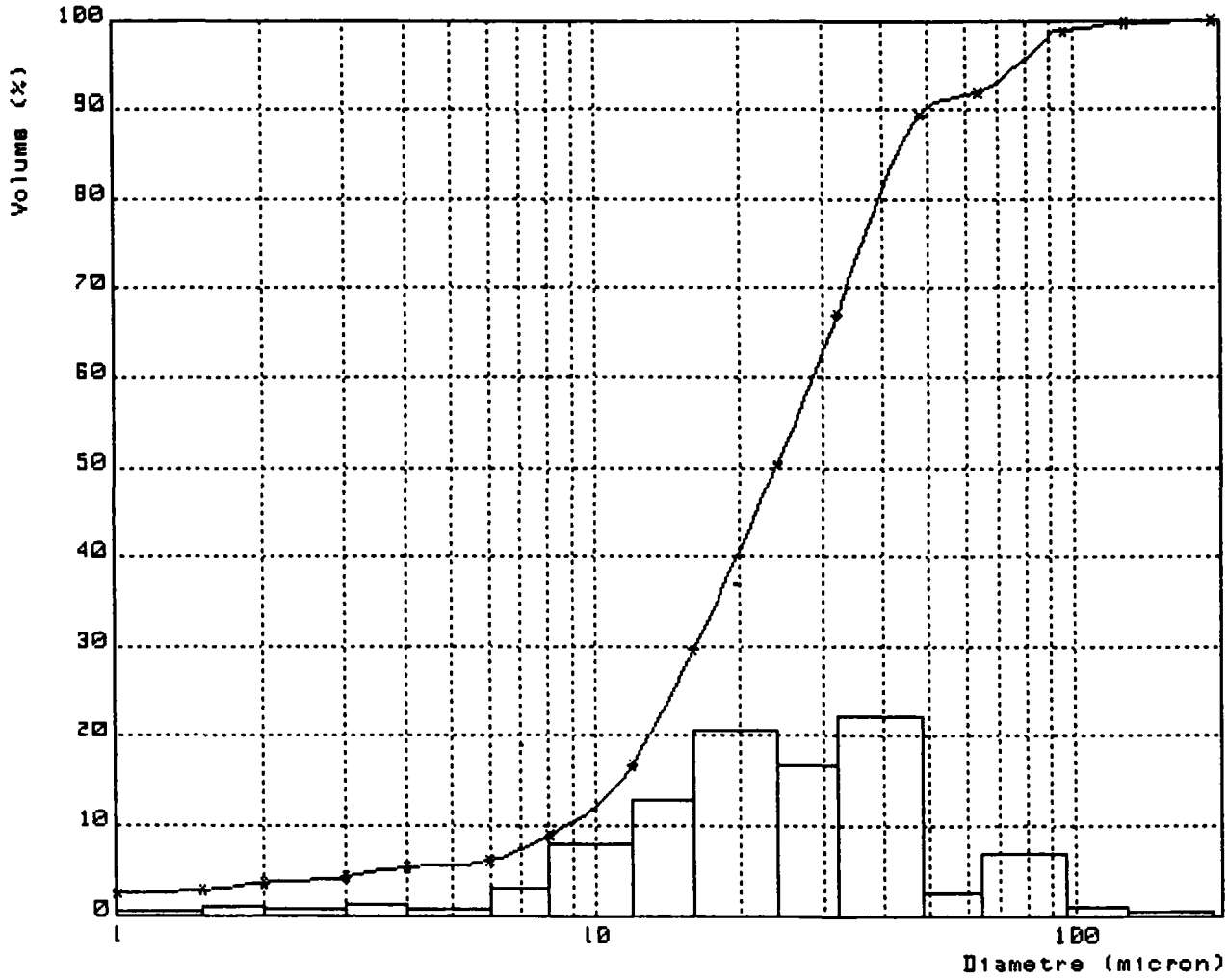


d20 = 1.6 d50 = 4.1 d80 = 7.8

HISTOGRAMME	
Classe	Volume %
<1	14.4
1-1.5	3.9
1.5-2	8.0
2-3	12.2
3-4	10.9
4-6	18.7
6-8	12.9
8-12	13.0
12-16	3.5
16-24	2.5
24-32	0.0
32-48	0.0
48-64	0.0
64-96	0.0
96-128	0.0
128-192	0.0

PASSANTS CUMULES	
Diamètre	Volume %
1	14.4
1.5	18.3
2	26.3
3	38.5
4	49.4
6	68.1
8	81.0
12	94.0
16	97.5
24	100.0
32	100.0
48	100.0
64	100.0
96	100.0
128	100.0
192	100.0

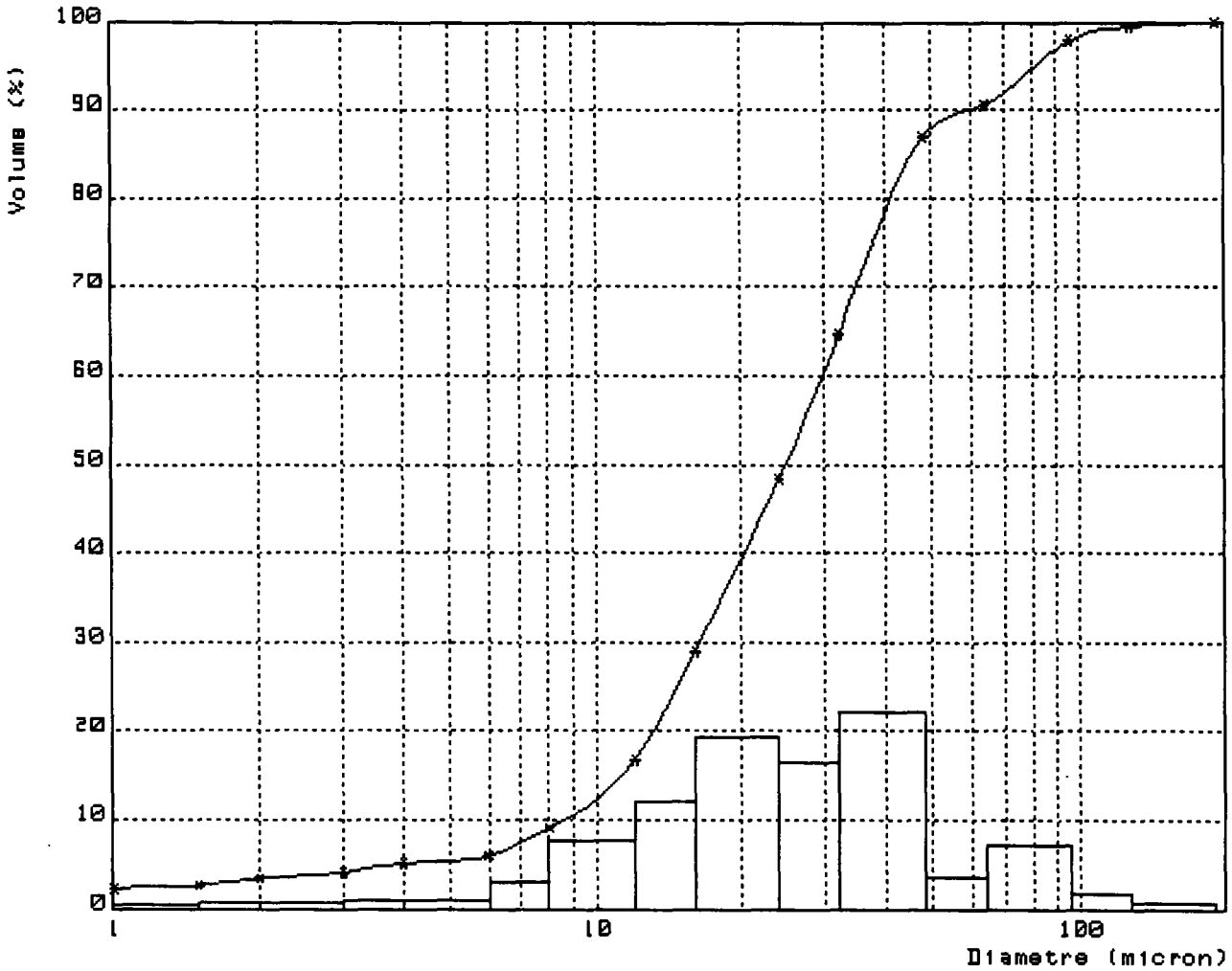
Echantillon : REO2, UF(2)
Densité moyenne :
Concentration g/l :
Dispersant : 3 kg/t
Durée d'ultrasons : 20 s
Commentaire : - 0.05 + 0.010 mm (2)



d20 = 13.1 d50 = 23.4 d80 = 39.7

HISTOGRAMME		PASSANTS CUMULES	
Classe	Volume %	Diamètre	Volume %
<1	2.4	1	2.4
[1-1.5]	.3	1.5	2.7
[1.5-2]	.9	2	3.6
[2-3]	.7	3	4.3
[3-4]	1.1	4	5.4
[4-6]	.7	6	6.1
[6-8]	2.9	8	9.0
[8-12]	7.8	12	16.8
[12-16]	12.8	16	29.6
[16-24]	20.7	24	50.3
[24-32]	16.8	32	67.1
[32-48]	22.3	48	89.4
[48-64]	2.5	64	91.9
[64-96]	6.9	96	98.8
[96-128]	.9	128	99.7
[128-192]	.3	192	100.0

REO ORE SAMPLE N°2 47
Echantillon : REO2,UF(1)+UF(2)
Dispersant : HMP:3kg/t
Durée d'ultrasons : 20 s
Commentaire : -0.05+0.010mm(1) & -0.05+.0.010mm(2)



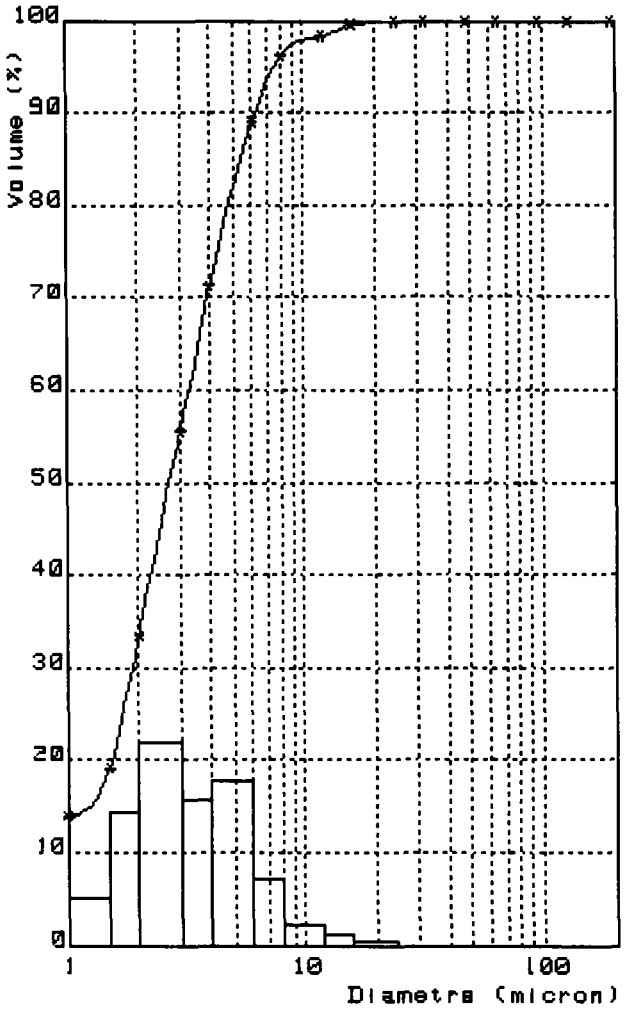
d20 = 13.1 d50 = 24.8 d80 = 40.9

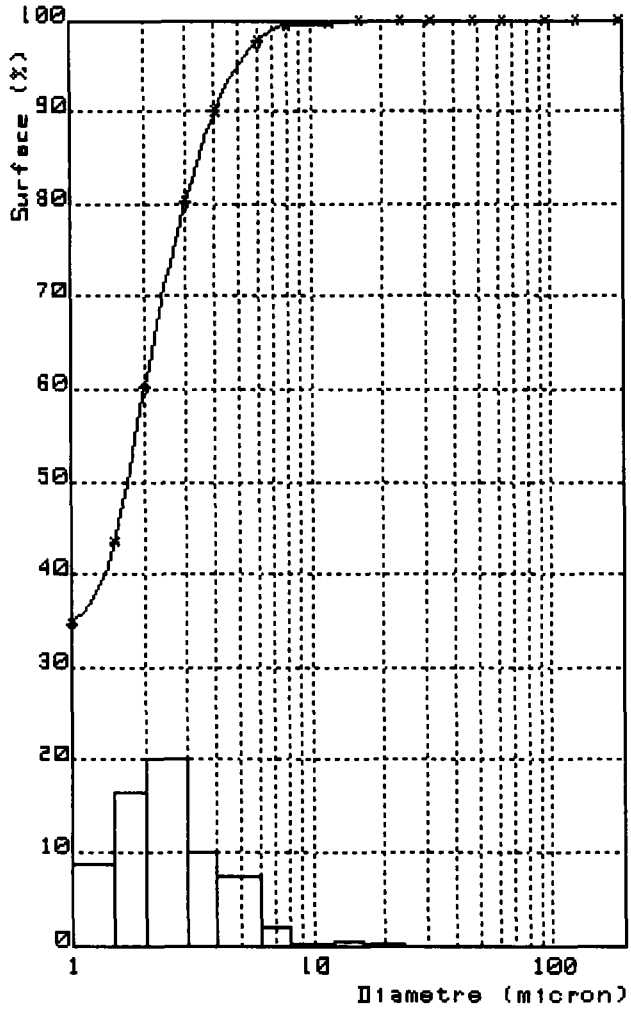
HISTOGRAMME	
Classe	Volume %
<1	2.3
1-1.5	.3
1.5-2	.8
2-3	.7
3-4	1.0
4-6	.9
6-8	3.1
8-12	7.7
12-16	12.2
16-24	19.3
24-32	16.4
32-48	22.3
48-64	3.6
64-96	7.2
96-128	1.6
128-192	.6

PASSANTS CUMULES	
Diamètre	Volume %
1	2.3
1.5	2.6
2	3.4
3	4.1
4	5.1
6	6.0
8	9.1
12	16.8
16	29.0
24	48.3
32	64.7
48	87.0
64	90.6
96	97.8
128	99.4
192	100.0

Granulomètre à laser CILAS 715

REO ORE SAMPLE N°3
Echantillon : OF(1)
Dispersant : HMP+DISPEX:3kg/t
Durée d'ultrasons : 30 s
Commentaire : SLIMES -0.010mm(1)





d20 = 1.5 d50 = 2.7 d80 = 4.8

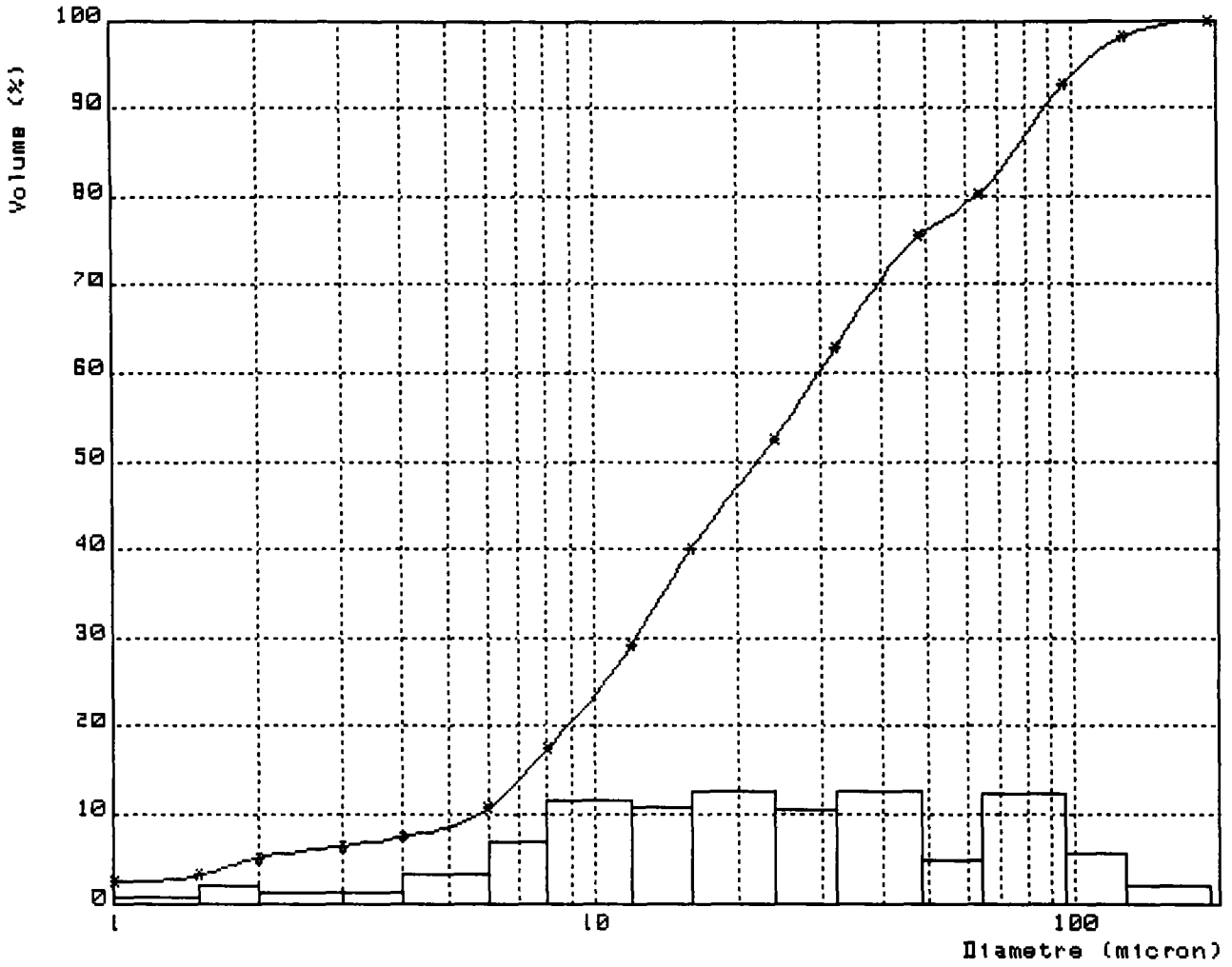
HISTOGRAMMES

Classe	Volume %	Surface %
<1	14.0	34.7
1-1.5	5.2	8.8
1.5-2	14.3	16.6
2-3	22.0	20.1
3-4	15.8	9.9
4-6	17.8	7.4
6-8	7.1	2.0
8-12	2.1	.0
12-16	1.3	.4
16-24	.4	.0
24-32	0.0	.0
32-48	0.0	.0
48-64	0.0	.0
64-96	0.0	.0
96-128	0.0	0.0
128-192	0.0	0.0

PASSANTS CUMULES

Diamètre	Volume %	Surface %
1	14.0	34.7
1.5	19.2	43.5
2	33.5	60.1
3	55.5	80.2
4	71.3	90.1
6	89.1	97.5
8	96.2	99.5
12	98.3	99.5
16	99.6	99.9
24	100.0	100.0
32	100.0	100.0
48	100.0	100.0
64	100.0	100.0
96	100.0	100.0
128	100.0	100.0
192	100.0	100.0

REO ORE SAMPLE N°3
Echantillon : REO3,(UF 1)
Dispersant : HMP:3kg/t 49
Durée d'ultrasons : 30 s
Commentaire : -0.125+0.010mm(1),UF(1)



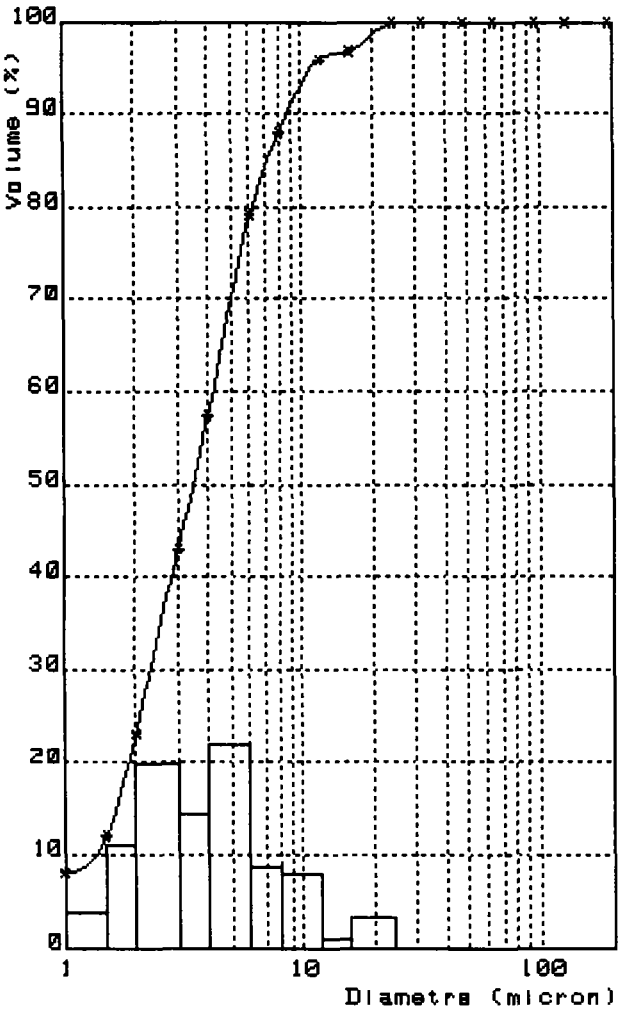
d20 = 8.8 d50 = 22.2 d80 = 63.4

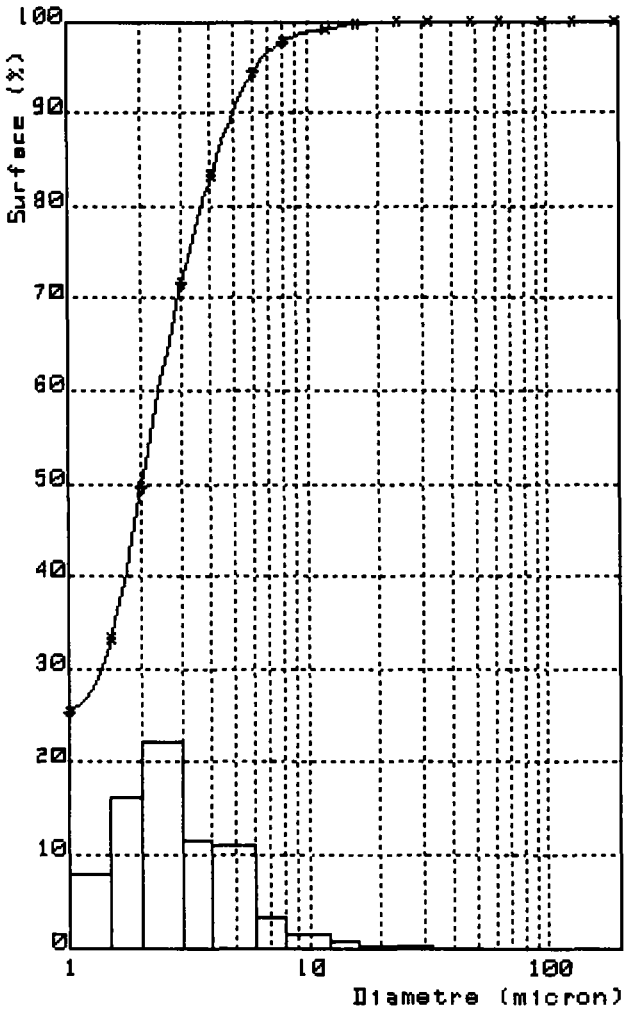
HISTOGRAMME	
Classe	Volume %
<1	2.5
1-1.5	.7
1.5-2	1.9
2-3	1.3
3-4	1.1
4-6	3.2
6-8	6.8
8-12	11.6
12-16	10.9
16-24	12.5
24-32	10.4
32-48	12.6
48-64	4.7
64-96	12.4
96-128	5.5
128-192	1.9

PASSANTS CUMULES	
Diamètre	Volume %
1	2.5
1.5	3.2
2	5.1
3	6.4
4	7.5
6	10.7
8	17.5
12	29.1
16	40.0
24	52.5
32	62.9
48	75.5
64	80.2
96	92.6
128	98.1
192	100.0

REO ORE SAMPLE N°3
Echantillon : REO3,OF(2)
Dispersant : HMP+DISPEX:3kg/t
Durée d'ultrasons : 30 s
Commentaire : SLIMES -0.010mm(2),OF(2)

50





d20 = 1.9 d50 = 3.5 d80 = 6.1

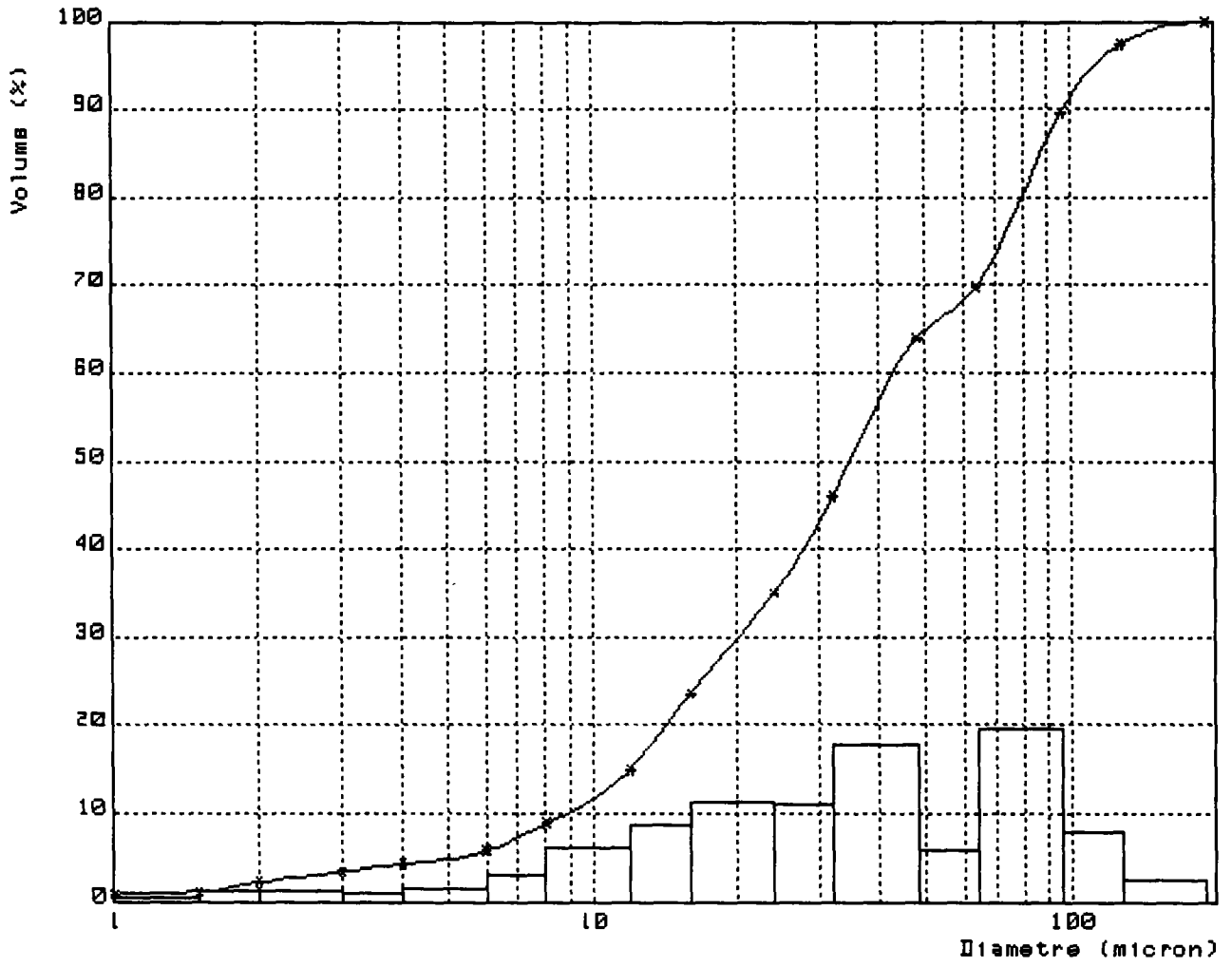
HISTOGRAMMES

Classe	Volume %	Surface %
<1	8.1	25.4
1-1.5	3.9	7.8
1.5-2	11.1	16.3
2-3	19.9	22.1
3-4	14.4	11.6
4-6	21.8	11.1
6-8	8.8	3.3
8-12	7.9	1.3
12-16	.9	.6
16-24	3.2	.2
24-32	0.0	.1
32-48	0.0	.0
48-64	0.0	.0
64-96	0.0	.0
96-128	0.0	0.0
128-192	0.0	0.0

PASSANTS CUMULES

Diamètre	Volume %	Surface %
1	8.1	25.4
1.5	12.0	33.3
2	23.1	49.6
3	43.0	71.7
4	57.4	83.3
6	79.2	94.4
8	88.0	97.7
12	95.9	99.0
16	96.8	99.6
24	100.0	99.9
32	100.0	99.9
48	100.0	100.0
64	100.0	100.0
96	100.0	100.0
128	100.0	100.0
192	100.0	100.0

REO ORE SAMPLE N°3
Echantillon : REO3,(UF 2)
Dispersant : HMP:3kg/t
Durée d'ultrasons : 30 s
Commentaire :-0.125+0.010mm(2),UF(2).



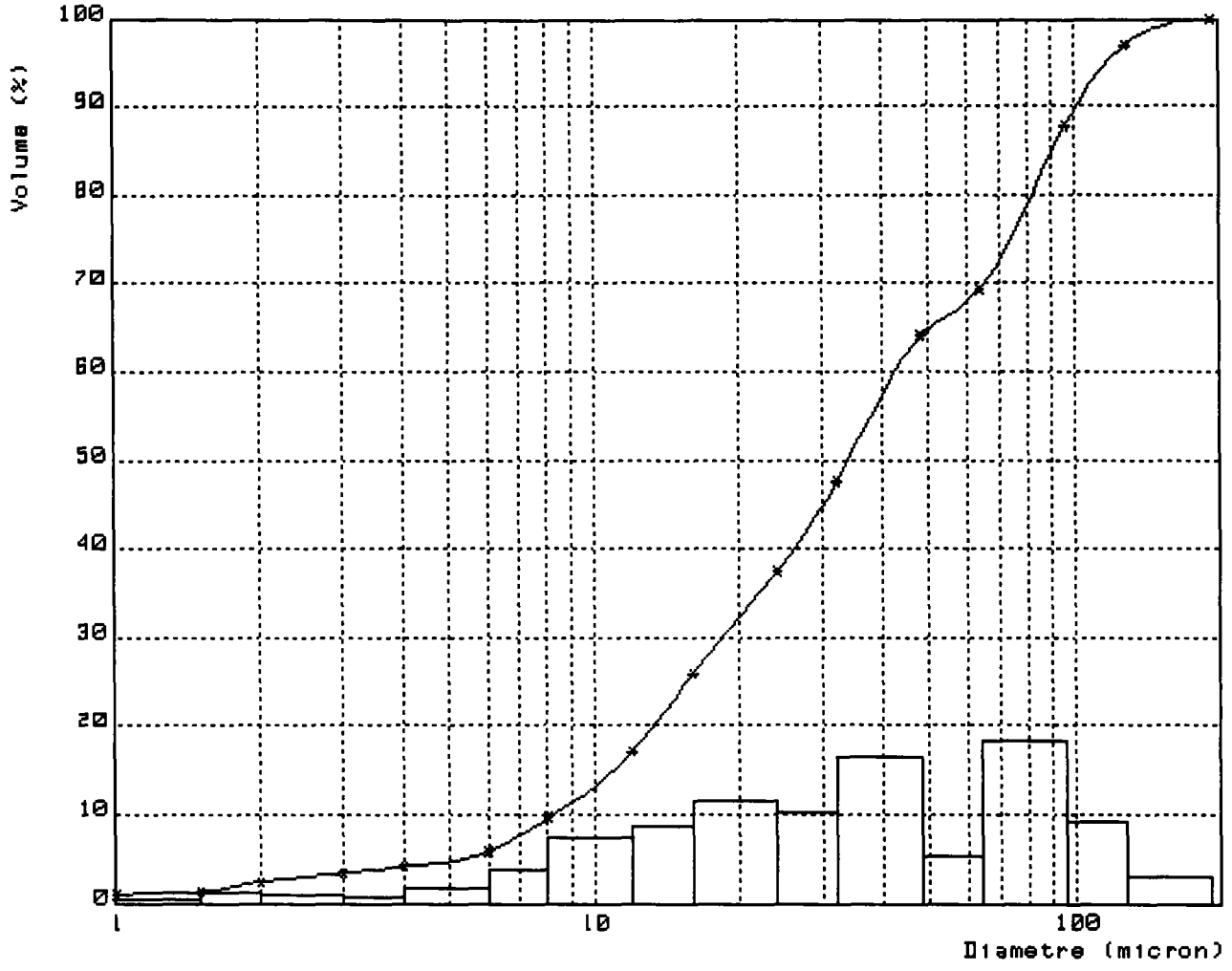
d20 = 14.3 d50 = 34.7 d80 = 79.6

HISTOGRAMME	
Classe	Volume %
<1	.8
1-1.5	.3
1.5-2	1.1
2-3	1.2
3-4	.9
4-6	1.5
6-8	3.1
8-12	6.1
12-16	8.6
16-24	11.4
24-32	11.1
32-48	17.9
48-64	5.9
64-96	19.7
96-128	7.8
128-192	2.6

PASSANTS CUMULES	
Diamètre	Volume %
1	.8
1.5	1.1
2	2.2
3	3.4
4	4.3
6	5.8
8	8.9
12	15.0
16	23.6
24	35.0
32	46.1
48	64.0
64	69.9
96	89.6
128	97.4
192	100.0

Granulomètre à laser CILAS 715

REO ORE SAMPLE N°3
Echantillon : REO3,FF
Dispersant : HMP:3kg/t
Durée d'ultrasons : 30 s
Commentaire : -0.125+0.010mm(t),FEED TO FLOTATION.



d20 = 13.3 d50 = 33.8 d80 = 81.9

HISTOGRAMME	
Classe	Volume %
<1	1.0
1-1.5	.3
1.5-2	1.1
2-3	1.0
3-4	.8
4-6	1.7
6-8	3.8
8-12	7.4
12-16	8.8
16-24	11.5
24-32	10.2
32-48	16.5
48-64	5.3
64-96	18.4
96-128	9.1
128-192	3.1

PASSANTS CUMULES	
Diamètre	Volume %
1	1.0
1.5	1.3
2	2.4
3	3.4
4	4.2
6	5.9
8	9.7
12	17.1
16	25.9
24	37.4
32	47.6
48	64.1
64	69.4
96	87.8
128	96.9
192	100.0

2.3.3. Material balances relevant to ore preparation

End products from the wet processing operations were analyzed:

- for major elements by XRF on glass beads and or by wet chemical procedures (F, Ba, SO_3, CO_2, H_2O-, H_2O+, Sr)
- for rare earth elements and Y (10 or 15 elements) by ICP/MS.

Material balances including REE balances are shown in tables:

- 1HO1 to 1HO4 for the REO ore sample n° 1
- 2HP1 to 2HP4 for the REO ore sample n° 2
- 3HO1 to 3HO3 for the REO ore sample n° 3.

Preparation of REO ore samples 1 and 2 led to low REO and Y losses in - 0.010 mm slimes as shown in the following summary table.

	- 160 + 10 µm fractions	
	Ore n° 1	Ore n° 2
Weight recovery %	95.36	88.81
P_2O_5 recovery %	97.17	90.19
Total R.E.O. recovery %	95.94	93.10
Y Recovery %	95.30	90.34
P_2O_5 assay %	3.19	3.62
CaO assay %	7.41	16.95
SiO_2 assay %	57.30	4.51
Fe_2O_3 assay %	13.22	6.40
Al_2O_3 assay %	7.91	0.76
MgO assay %	1.49	13.29
SrO assay %		10.39
BaO assay %		1.27
CO_2 assay %		31.29
Total R.E.O. assay ppm	6 528	46 929
Y assay ppm	970	62

Preparation of ore 3 gave rise to production of a high proportion of - 0.010 mm slimes, highly enriched in REE.

TABLE N° 1H01 : REO ORE SAMPLE N°1,OVERALL MATERIAL BALANCE RELEVANT TO ORE PREPARATION.

PRODUCTS	WEIGHT	P205		CaO		SiO2		Fe203	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 -0.16+0.05mm(1)	45.49	3.55	51.60	8.30	51.55	59.70	48.06	10.90	35.39
2 -0.16+0.05mm(2)	18.97	1.35	8.18	4.05	10.49	63.80	21.42	4.65	6.30
3 -0.05+0.01mm(1)	28.32	3.95	35.74	8.45	32.67	47.90	24.01	23.20	46.89
4 -0.05+0.01mm(2)	2.58	2.00	1.65	4.95	1.74	70.20	3.21	7.65	1.41
5 -0.01mm(1)	4.20	1.95	2.62	5.70	3.27	38.50	2.86	32.10	9.62
6 -0.01mm(2)	.44	1.50	.21	4.55	.27	58.20	.45	12.60	.40
CALCULATED HEAD	100.00	3.13	100.00	7.32	100.00	56.51	100.00	14.01	100.00

PRODUCTS	WEIGHT	A1203		MgO		L.O.I.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 -0.16+0.05mm(1)	45.49	8.30	47.91	1.60	43.59	.75	36.86
2 -0.16+0.05mm(2)	18.97	7.60	18.29	.73	8.29	.55	11.27
3 -0.05+0.01mm(1)	28.32	7.40	26.59	1.85	31.38	.84	25.70
4 -0.05+0.01mm(2)	2.58	8.80	2.88	1.15	1.78	.67	1.87
5 -0.01mm(1)	4.20	7.10	3.78	5.50	13.84	4.95	22.46
6 -0.01mm(2)	.44	9.65	.54	4.25	1.12	3.85	1.83
CALCULATED HEAD	100.00	7.88	100.00	1.67	100.00	.93	100.00

PRODUCTS	WEIGHT %	CaO GRADE / P205 GRADE
1 -0.16+0.05mm(1)	45.49	2.3380
2 -0.16+0.05mm(2)	18.97	3.0000
3 -0.05+0.01mm(1)	28.32	2.1392
4 -0.05+0.01mm(2)	2.58	2.4750
5 -0.01mm(1)	4.20	2.9231
6 -0.01mm(2)	.44	3.0333
CALCULATED HEAD	100.00	2.3402

TABLE N° 1H01 : RED ORE SAMPLE N°1, ORE PREPARATION.

PRODUCTS	WEIGHT	P205		CaO		SiO2		Fe203	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 -0.16+0.05mm(t) 1 2 0 0 0 0	64.46	2.90	59.78	7.05	62.04	60.91	69.48	9.06	41.68
2 -0.05+0.01mm(t) 3 4 0 0 0 0	30.90	3.79	37.39	8.16	34.42	49.76	27.21	21.90	48.30
3 -0.01mm(t) 5 6 0 0 0 0	4.64	1.91	2.83	5.59	3.54	40.37	3.31	30.25	10.02
4 -0.16+0.01mm(t) 1 2 3 4 0 0	95.36	3.19	97.17	7.41	96.46	57.30	96.69	13.22	89.98

PRODUCTS	WEIGHT	A1203		MgO		L.O.I.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 -0.16+0.05mm(t) 1 2 0 0 0 0	64.46	8.09	66.20	1.34	51.89	.69	48.14
2 -0.05+0.01mm(t) 3 4 0 0 0 0	30.90	7.52	29.47	1.79	33.16	.83	27.57
3 -0.01mm(t) 5 6 0 0 0 0	4.64	7.34	4.32	5.38	14.96	4.85	24.29
4 -0.16+0.01mm(t) 1 2 3 4 0 0	95.36	7.91	95.68	1.49	85.04	.73	75.71

TABLE N° 1HD2 : RED ORE SAMPLE N°1,OVERALL MATERIAL BALANCE
RELEVANT TO ORE PREPARATION AND LOW INTENSITY MAGNETIC SEPARATION.

PRODUCTS	WEIGHT	P205		CaO		SiO2		Fe203	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 -0.01mm(1)	4.20	1.95	2.71	5.70	3.38	38.50	2.85	32.10	9.65
2 -0.01mm(2)	.44	1.50	.22	4.55	.28	58.20	.45	12.60	.40
3 -0.16+0.01mmNM	86.76	3.35	96.22	7.75	95.00	62.40	95.37	5.80	36.02
4 -0.16+0.01mm M	8.60	.30	.85	1.10	1.34	8.75	1.33	87.60	53.93
CALCULATED HEAD	100.00	3.02	100.00	7.08	100.00	56.76	100.00	13.97	100.00

PRODUCTS	WEIGHT	A1203		MgO		TiO2	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 -0.01mm(1)	4.20	7.10	3.71	5.50	13.56	.69	3.54
2 -0.01mm(2)	.44	9.65	.53	4.25	1.10	.52	.28
3 -0.16+0.01mmNM	86.76	8.75	94.48	1.65	84.03	.84	89.13
4 -0.16+0.01mm M	8.60	1.20	1.28	.26	1.31	.67	7.05
CALCULATED HEAD	100.00	8.04	100.00	1.70	100.00	.82	100.00

PRODUCTS	WEIGHT %	CaO GRADE / P205 GRADE
1 -0.01mm(1)	4.20	2.9231
2 -0.01mm(2)	.44	3.0333
3 -0.16+0.01mmNM	86.76	2.3134
4 -0.16+0.01mm M	8.60	3.6667
CALCULATED HEAD	100.00	2.3431

TABLE N° 1H02 : RED ORE SAMPLE N° 1,PREPARATION AND L.I.M.S.

PRODUCTS	WEIGHT	P205		CaO		Si02		Fe203	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 -0.01mm(t) 1 2 0 0 0 0	4.64	1.91	2.93	5.59	3.67	40.37	3.30	30.25	10.05
2 -0.16+0.01mm(t) 3 4 0 0 0 0	95.36	3.07	97.07	7.15	96.33	57.56	96.70	13.18	89.95

PRODUCTS	WEIGHT	A1203		MgO		Ti02	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 -0.01mm(t) 1 2 0 0 0 0	4.64	7.34	4.24	5.38	14.66	.67	3.82
2 -0.16+0.01mm(t) 3 4 0 0 0 0	95.36	8.07	95.76	1.52	85.34	.82	96.18

TABLE N° 1HO3 : REO ORE SAMPLE N° 1,OVERALL REE BALANCE RELEVANT TO ORE PREPARATION.

PRODUCTS	WEIGHT	La		Ce		Nd		Sm	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 -0.16+0.01mm(t)	95.36	1540.00	95.84	3010.00	96.06	1150.00	96.02	218.00	95.97
2 -0.01mm(1)	4.20	1440.00	3.95	2650.00	3.72	1020.00	3.75	195.00	3.78
3 -0.01mm(2)	.44	740.00	.21	1455.00	.21	598.00	.23	124.00	.25
CALCULATED HEAD	100.00	1532.28	100.00	2988.04	100.00	1142.11	100.00	216.62	100.00

PRODUCTS	WEIGHT	Eu		Gd		Dy		Er	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 -0.16+0.01mm(t)	95.36	19.90	95.88	208.00	95.66	196.00	95.44	100.00	95.12
2 -0.01mm(1)	4.20	18.30	3.88	202.00	4.09	201.00	4.31	110.00	4.61
3 -0.01mm(2)	.44	10.50	.23	116.80	.25	110.00	.25	61.00	.27
CALCULATED HEAD	100.00	19.79	100.00	207.35	100.00	195.83	100.00	100.25	100.00

TABLE N° 1HO3 : REO ORE SAMPLE N°1,REE BALANCE FOR ORE PREPARATION.

PRODUCTS	WEIGHT	La		Ce		Nd		Sm	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 -0.16+0.01mm(t) 1 0 0 0 0 0	95.36	1540.00	95.84	3010.00	96.06	1150.00	96.02	218.00	95.97
2 -0.01mm(t) 2 3 0 0 0 0	4.64	1373.62	4.16	2536.68	3.94	979.98	3.98	188.27	4.03

PRODUCTS	WEIGHT	Eu		Gd		Dy		Er	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 -0.16+0.01mm(t) 1 0 0 0 0 0	95.36	19.90	95.88	208.00	95.66	196.00	95.44	100.00	95.12
2 -0.01mm(t) 2 3 0 0 0 0	4.64	17.56	4.12	193.92	4.34	192.37	4.56	105.35	4.88

TABLE N° 1H04 : REO ORE SAMPLE N° 1,OVERALL REE BALANCE RELEVANT TO ORE PREPARATION.

PRODUCTS	WEIGHT	Yb		Y	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 -0.16+0.01mm(t)	95.36	85.80	95.39	970.00	95.30
2 -0.01mm(1)	4.20	89.00	4.36	1030.00	4.46
3 -0.01mm(2)	.44	49.50	.25	540.00	.24
CALCULATED HEAD	100.00	85.77	100.00	970.63	100.00

TABLE N° 1H04 : REO ORE SAMPLE N° 1,REE BALANCE FOR ORE PREPARATION.

PRODUCTS	WEIGHT	Yb		Y	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 -0.16+0.01mm(t) 1 0 0 0 0 0	95.36	85.80	95.39	970.00	95.30
2 -0.01mm(t) 2 3 0 0 0 0	4.64	85.25	4.61	983.53	4.70

TABLE N° 2HP1 : REO ORE SAMPLE N°2,OVERALL MATERIAL BALANCE RELEVANT TO ORE PREPARATION.

PRODUCTS	WEIGHT	P2O5		CaO		SiO2		Fe2O3	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 +0.16mm(2)	1.90	2.15	1.15	19.20	2.18	7.10	2.93	10.70	2.78
2 -0.16+0.01mm(t)	86.91	3.65	89.04	16.90	87.60	4.45	83.86	6.40	76.09
3 -0.010mm(1)	5.20	3.15	4.60	14.20	4.40	7.40	8.34	14.50	10.31
4 -0.010mm(2)	5.99	3.10	5.21	16.30	5.82	3.75	4.87	13.20	10.82
CALCULATED HEAD	100.00	3.56	100.00	16.77	100.00	4.61	100.00	7.31	100.00

PRODUCTS	WEIGHT	Al2O3		MgO		TiO2		L.O.I.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 +0.16mm(2)	1.90	.43	.85	12.80	1.88	.06	1.77	32.20	1.99
2 -0.16+0.01mm(t)	86.91	.77	69.85	13.30	89.33	.06	80.88	31.00	87.72
3 -0.010mm(1)	5.20	3.90	21.17	9.55	3.84	.10	8.07	26.80	4.54
4 -0.010mm(2)	5.99	1.30	8.13	10.70	4.95	.10	9.29	29.50	5.75
CALCULATED HEAD	100.00	.96	100.00	12.94	100.00	.06	100.00	30.71	100.00

PRODUCTS	WEIGHT %	CaO GRADE / P2O5 GRADE
1 +0.16mm(2)	1.90	8.9302
2 -0.16+0.01mm(t)	86.91	4.6301
3 -0.010mm(1)	5.20	4.5079
4 -0.010mm(2)	5.99	5.2581
CALCULATED HEAD	100.00	4.7066

TABLE N° 2HP1 : REO ORE SAMPLE N°2, PREPARATION.

PRODUCTS	WEIGHT	P2O5		CaO		SiO2		Fe2O3	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 +0.010mm(t) 1 2 0 0 0 0	88.81	3.62	90.19	16.95	89.77	4.51	86.79	6.49	78.87
2 -0.010mm(t) 3 4 0 0 0 0	11.19	3.12	9.81	15.32	10.23	5.45	13.21	13.80	21.13

PRODUCTS	WEIGHT	Al2O3		MgO		TiO2		L.O.I.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 +0.010mm(t) 1 2 0 0 0 0	88.81	.76	70.70	13.29	91.21	.06	82.64	31.03	89.71
2 -0.010mm(t) 3 4 0 0 0 0	11.19	2.51	29.30	10.17	8.79	.10	17.36	28.25	10.29

TABLE N° 2HP2 : RED ORE SAMPLE N°2,OVERALL MATERIAL BALANCE RELEVANT TO ORE PREPARATION.

PRODUCTS	WEIGHT	MnO		CO2		H2O-		H2O+	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 +0.16mm(2)	1.90	3.35	3.37	31.00	1.93	.23	1.91	1.74	2.56
2 -0.16+0.01mm(t)	86.91	1.70	78.15	31.30	89.24	.14	53.26	1.02	68.76
3 -0.010mm(1)	5.20	3.55	9.76	21.95	3.74	1.37	31.19	4.00	16.13
4 -0.010mm(2)	5.99	2.75	8.71	25.85	5.08	.52	13.64	2.70	12.54
CALCULATED HEAD	100.00	1.89	100.00	30.48	100.00	.23	100.00	1.29	100.00

PRODUCTS	WEIGHT	SrO		BaO		SO3	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 +0.16mm(2)	1.90	7.43	1.41	.67	1.00	.20	.40
2 -0.16+0.01mm(t)	86.91	10.45	91.02	1.28	87.22	1.02	92.95
3 -0.010mm(1)	5.20	5.62	2.93	1.60	6.52	.62	3.38
4 -0.010mm(2)	5.99	7.72	4.63	1.12	5.26	.52	3.27
CALCULATED HEAD	100.00	9.98	100.00	1.28	100.00	.95	100.00

TABLE N° 2HP2 : RED ORE SAMPLE N°2,PREPARATION.

PRODUCTS	WEIGHT	MnO		CO2		H2O-		H2O+	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 +0.010mm(t) 1 2 0 0 0 0	88.81	1.74	81.52	31.29	91.18	.14	55.18	1.04	71.32
2 -0.010mm(t) 3 4 0 0 0 0	11.19	3.12	18.48	24.04	8.82	.91	44.82	3.30	28.68

PRODUCTS	WEIGHT	SrO		BaO		SO3	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 +0.010mm(t) 1 2 0 0 0 0	88.81	10.39	92.44	1.27	88.22	1.00	93.35
2 -0.010mm(t) 3 4 0 0 0 0	11.19	6.74	7.56	1.34	11.78	.57	6.65

TABLE N° 2HP3 : REO ORE SAMPLE N°2,REE BALANCE RELEVANT TO ORE PREPARATION.

PRODUCTS	WEIGHT	La		Ce		Nd		Sm	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 +0.16mm(2)	1.90	2600.00	.35	3960.00	.33	1142.00	.34	131.00	.35
2 -0.16+0.01mm(t)	86.91	15020.00	92.94	24590.00	92.74	6896.00	92.68	758.00	92.16
3 -0.010mm(1)	5.20	9240.00	3.42	15040.00	3.39	4050.00	3.26	436.00	3.17
4 -0.010mm(2)	5.99	7700.00	3.28	13620.00	3.54	4020.00	3.72	516.00	4.32
CALCULATED HEAD	100.00	14044.99	100.00	23044.33	100.00	6466.41	100.00	714.85	100.00

PRODUCTS	WEIGHT	Eu		Gd		Dy		Er	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 +0.16mm(2)	1.90	18.00	.39	58.40	.42	23.90	.44	6.50	.41
2 -0.16+0.01mm(t)	86.91	93.00	91.50	274.00	91.20	107.00	90.67	31.50	91.28
3 -0.010mm(1)	5.20	57.80	3.40	160.00	3.19	65.00	3.30	18.00	3.12
4 -0.010mm(2)	5.99	69.40	4.71	226.00	5.18	95.80	5.59	26.00	5.19
CALCULATED HEAD	100.00	88.33	100.00	261.10	100.00	102.57	100.00	29.99	100.00

TABLE N° 2HP3 : REO ORE SAMPLE N°2,REE BALANCE FOR ORE PREPARATION.

PRODUCTS	WEIGHT	La		Ce		Nd		Sm	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 +0.010mm(t) 1 2 0 0 0 0	88.81	14754.29	93.30	24148.64	93.07	6772.90	93.02	744.59	92.50
2 -0.010mm(t) 3 4 0 0 0 0	11.19	8415.64	6.70	14279.88	6.93	4033.94	6.98	478.82	7.50

PRODUCTS	WEIGHT	Eu		Gd		Dy		Er	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 +0.010mm(t) 1 2 0 0 0 0	88.81	91.40	91.89	269.39	91.63	105.22	91.11	30.97	91.69
2 -0.010mm(t) 3 4 0 0 0 0	11.19	64.01	8.11	195.33	8.37	81.49	8.89	22.28	8.31

TABLE N° 2HP4 : RED ORE SAMPLE N° 2,REE BALANCE RELEVANT TO ORE PREPARATION.

PRODUCTS	WEIGHT	Yb		Y	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 +0.16mm(2)	1.90	2.20	.38	20.60	.64
2 -0.16+0.01mm(t)	86.91	11.70	91.84	63.00	89.70
3 -0.01mm(1)	5.20	6.20	2.91	48.00	4.09
4 -0.01mm(2)	5.99	9.00	4.87	56.80	5.57
CALCULATED HEAD	100.00	11.07	100.00	61.04	100.00

TABLE N° 2HP4 : RED ORE SAMPLE N°2,REE BALANCE FOR ORE PREPARATION.

PRODUCTS	WEIGHT	Yb		Y	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 +0.010mm(t) 1 2 0 0 0 0	88.81	11.50	92.22	62.09	90.34
2 -0.010mm(t) 3 4 0 0 0 0	11.19	7.70	7.78	52.71	9.66

TABLE N° 3HO1 : RED ORE SAMPLE N° 3, REE OVERALL BALANCE RELEVANT TO ORE PREPARATION.

PRODUCTS	WEIGHT	La		Ce		Pr		Nd	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADEppm	RECOVERY	GRADE %	RECOVERY
1 -.125+0.01mm(1)	25.02	3.42	26.33	3.59	26.05	2349.00	26.79	.52	26.94
2 -.125+0.01mm(2)	49.55	.67	10.22	.75	10.78	500.60	11.31	.11	11.19
3 -0.01mm(1)	16.35	10.64	53.53	11.09	52.59	6909.00	51.49	1.52	51.47
4 -0.01mm(2)	9.08	3.55	9.92	4.02	10.59	2518.00	10.42	.55	10.40
CALCULATED HEAD	100.00	3.25	100.00	3.45	100.00	2194.02	100.00	.48	100.00

PRODUCTS	WEIGHT	Sm		Eu		Gd		Tb	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 -.125+0.01mm(1)	25.02	342.90	27.13	91.30	30.76	150.00	26.28	21.20	29.69
2 -.125+0.01mm(2)	49.55	76.10	11.93	25.60	17.08	43.50	15.09	6.10	16.92
3 -0.01mm(1)	16.35	973.20	50.32	191.10	42.08	416.00	47.62	46.00	42.10
4 -0.01mm(2)	9.08	369.70	10.62	82.40	10.08	173.20	11.01	22.20	11.28
CALCULATED HEAD	100.00	316.19	100.00	74.25	100.00	142.83	100.00	17.86	100.00

TABLE N° 3HO1 : RED ORE SAMPLE N°3, REE BALANCE FOR ORE PREPARATION.

PRODUCTS	WEIGHT	La		Ce		Pr		Nd	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADEppm	RECOVERY	GRADE %	RECOVERY
1 -.125+0.01mm(t) 1 2 0 0 0 0	74.57	1.59	36.55	1.70	36.83	1120.78	38.09	.25	38.13
2 -0.01mm(t) 3 4 0 0 0 0	25.43	8.11	63.45	8.57	63.17	5341.16	61.91	1.17	61.87

PRODUCTS	WEIGHT	Sm		Eu		Gd		Tb	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 -.125+0.01mm(t) 1 2 0 0 0 0	74.57	165.62	39.06	47.64	47.85	79.23	41.37	11.17	46.61
2 -0.01mm(t) 3 4 0 0 0 0	25.43	757.72	60.94	152.29	52.15	329.31	58.63	37.50	53.39

TABLE N° 3HO2 : REO ORE SAMPLE N° 3, REE OVERALL BALANCE RELEVANT TO ORE PREPARATION.

PRODUCTS	WEIGHT	Dy		Ho		Er		Tm	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 -.125+0.01mm(1)	25.02	71.00	27.09	13.70	26.52	39.80	25.25	4.30	21.94
2 -.125+0.01mm(2)	49.55	29.70	22.44	6.70	25.68	23.30	29.28	3.50	35.37
3 -0.01mm(1)	16.35	160.40	39.99	28.40	35.92	86.80	35.99	9.80	32.68
4 -0.01mm(2)	9.08	75.70	10.48	16.90	11.87	41.20	9.49	5.40	10.00
CALCULATED HEAD	100.00	65.58	100.00	12.93	100.00	39.44	100.00	4.90	100.00

PRODUCTS	WEIGHT	Yb		Lu		Y	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 -.125+0.01mm(1)	25.02	29.70	26.39	3.40	21.95	364.60	22.90
2 -.125+0.01mm(2)	49.55	15.30	26.92	2.90	37.08	277.00	34.46
3 -0.01mm(1)	16.35	61.30	35.59	7.10	29.96	730.00	29.96
4 -0.01mm(2)	9.08	34.40	11.09	4.70	11.01	556.40	12.68
CALCULATED HEAD	100.00	28.16	100.00	3.88	100.00	398.35	100.00

TABLE N° 3HO2 : REO ORE SAMPLE N°3,REE BALANCE FOR ORE PREPARATION.

PRODUCTS	WEIGHT	Dy		Ho		Er		Tm	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 -.125+0.01mm(t) 1 2 0 0 0 0	74.57	43.56	49.53	9.05	52.20	28.84	54.53	3.77	57.32
2 -0.010mm(t) 3 4 0 0 0 0	25.43	130.16	50.47	24.29	47.80	70.52	45.47	8.23	42.68

PRODUCTS	WEIGHT	Yb		Lu		Y	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 -.125+0.01mm(t) 1 2 0 0 0 0	74.57	20.13	53.31	3.07	59.03	306.39	57.36
2 -0.010mm(t) 3 4 0 0 0 0	25.43	51.70	46.69	6.24	40.97	668.01	42.64

TABLE N° 3H03 : RED ORE SAMPLE N° 3,OVERALL MATERIAL BALANCE RELEVANT TO ORE PREPARATION.

PRODUCTS	WEIGHT	F		Ba		Cerics		Yttrics	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 -.125+0.01mm(1)	25.02	18.600	15.708	25.410	55.761	7.799	26.257	.079	25.038
2 -.125+0.01mm(2)	49.55	37.300	62.382	8.750	38.027	1.587	10.579	.043	27.275
3 -0.01mm(1)	16.35	20.820	11.490	1.960	2.811	24.036	52.885	.174	36.021
4 -0.01mm(2)	9.08	34.000	10.420	4.270	3.401	8.411	10.278	.101	11.666
ANALYSED HEAD	100.00	29.627	100.000	11.401	100.000	7.431	100.000	.079	100.000

PRODUCTS	WEIGHT	REE+Y	
	%	GRADE %	RECOVERY
1 -.125+0.01mm(1)	25.02	7.877	26.245
2 -.125+0.01mm(2)	49.55	1.630	10.754
3 -0.01mm(1)	16.35	24.210	52.709
4 -0.01mm(2)	9.08	8.512	10.292
ANALYSED HEAD	100.00	7.510	100.000

PRODUCTS	WEIGHT %	Yttrics GRADE / REE+Y GRADE
1 -.125+0.01mm(1)	25.02	.0100
2 -.125+0.01mm(2)	49.55	.0266
3 -0.01mm(1)	16.35	.0072
4 -0.01mm(2)	9.08	.0119
CALCULATED HEAD	100.00	.0105

TABLE N° 3H03 : RED ORE SAMPLE N° 3,ORE PREPARATION.

PRODUCTS	WEIGHT	F		Ba		Cerics		Yttrics	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 -.125+0.01mm(t) 1 2 0 0 0 0	74.57	31.026	78.090	14.340	93.789	3.671	36.837	.055	52.313
2 -0.010mm(t) 3 4 0 0 0 0	25.43	25.526	21.910	2.785	6.211	18.457	63.163	.148	47.687

PRODUCTS	WEIGHT	REE+Y	
	%	GRADE %	RECOVERY
1 -.125+0.01mm(t) 1 2 0 0 0 0	74.57	3.726	36.999
2 -0.010mm(t) 3 4 0 0 0 0	25.43	18.605	63.001

Refer. Ech.	No	PP	SIO2	AL2X	FE2X	CAO	MGO	K2O	MNO	TIO2	P2O5	LI	BE
Refer. Ech.	UNIT	%	%	%	%	%	%	%	%	%	PPM	PPM	PPM
	BINF	0.05	1.0	1.0	1.0	1.0	1.0	0.50	0.01	0.01	100.	10.	2.
	BSUP	100.00	100.0	100.0	100.0	100.0	50.0	20.00	20.00	35.00	80000.	40000.	3500.
07 -10 µm(1)	0001										11160.	44.	17.
09 -10 µm(2)	0002										8842.	39.	9.
10 -160+10µm(t)	0003										15880.	10.	9.

Refer. Ech.	No	B	V	CR	CO	NI	CU	ZN	AS	SR	Y	NB	MO
Refer. Ech.	UNIT	PPM	PPM	PPM	PPM	PPM	PPM	PPM	PPM	PPM	PPM	PPM	PPM
	BINF	10.	10.	10.	5.	10.	5.	5.	20.	5.	20.	20.	5.
	BSUP	18000.	40000.	13000.	25000.	18000.	8000.	20000.	50000.	10000.	5000.	15000.	7500.
07 -10 µm(1)	0001	98.	62.	49.	82.	99.	98.	283.	319.	147.	894.	90.	6.
09 -10 µm(2)	0002	53.	49.	3174.	102.	644.	231.	782.	251.	249.	531.	98.	33.
10 -160+10µm(t)	0003	68.	41.	70.	14.	35.	17.	59.	244.	122.	867.	44.	-5.

Refer. Ech.	No	AG	CD	SN	SB	BA	LA	CE	W	PB	BI	ZR
Refer. Ech.	UNIT	PPM	PPM	PPM	PPM	PPM	PPM	PPM	PPM	PPM	PPM	PPM
	BINF	0.2	2.	10.	10.	10.	20.	10.	10.	10.	10.	20.
	BSUP	300.0	5000.	20000.	25000.	3500.	15000.	5500.	15000.	6000.	10000.	13000.
07 -10 µm(1)	0001	-0.2	-2.	61.	25.	197.	1518.	2089.	15.	71.	-10.	244.
09 -10 µm(2)	0002	0.7	2.	716.	-10.	330.	868.	1335.	34.	108.	-10.	59.
10 -160+10µm(t)	0003	-0.2	-2.	40.	14.	60.	1709.	2509.	-10.	11.	-10.	-20.

TABLE 1H05: Determinations of trace elements by ICP spectrometry. REO ore sample 1.

	Total - 125 + 10 µm	Total - 10 µm slimes
Weight recovery %	74.57	25.43
F recovery %	78.09	21.91
Ba recovery %	93.79	6.21
Total REE recovery %	36.85	63.15
Y recovery %	57.36	42.64
F assay %	31.03	25.53
Ba assay %	14.34	2.79
Total REE assay ppm	36 899	185 442
Y assay ppm	306	668

Trace elements other than REE, were determined by ICP spectrometry on - 0.01 mm slimes and the - 0.16 + 0.01 mm (t) fraction separated from the REO ore sample 1 ground down to 0.16 mm, results are shown in table 1HO5.

2.4. COMPOSITIONS OF GRAVITRY PRECONCENTRATES SEPARATED FROM THE REO ORE SAMPLE N° 2

Flotation tests conducted on - 160 + 10 μm (t) or - 50 + 10 μm (t) fractions separated from the attrition-scrubbed and rod milled REO ore sample n° 2, led to poor concentration performance and low monazite concentrates. Poor amenability to flotation was attributed to an extremely high content of carbonate minerals in the feed.

Gravity separation, using shaking tables or a centrifugal separator (Multi Gravity Separator: MGS manufactured by MOZLEY), was applied to closely sized fractions: - 160 + 80 μm, - 80 + 40 μm, - 40 + 10 μm, with the objective to separate monazite and strontianite preconcentrates and gravity tails high in dolomite, since there are sufficient differences in speficic gravities of the minerals:

	specific gravity g/cm^3
. monazite	5 to 5.3
. strontianite	3.7
. barite	4.45
. psilomelane	4.0
. siderite	3.85
. dolomite	2.85 to 2.92
. calcite	2.71

Gravity preconcentrates: - 80 + 10 μm composite preconcentrates and - 40 + 10 μm, with compositions as follows, were beneficiated by flotation:

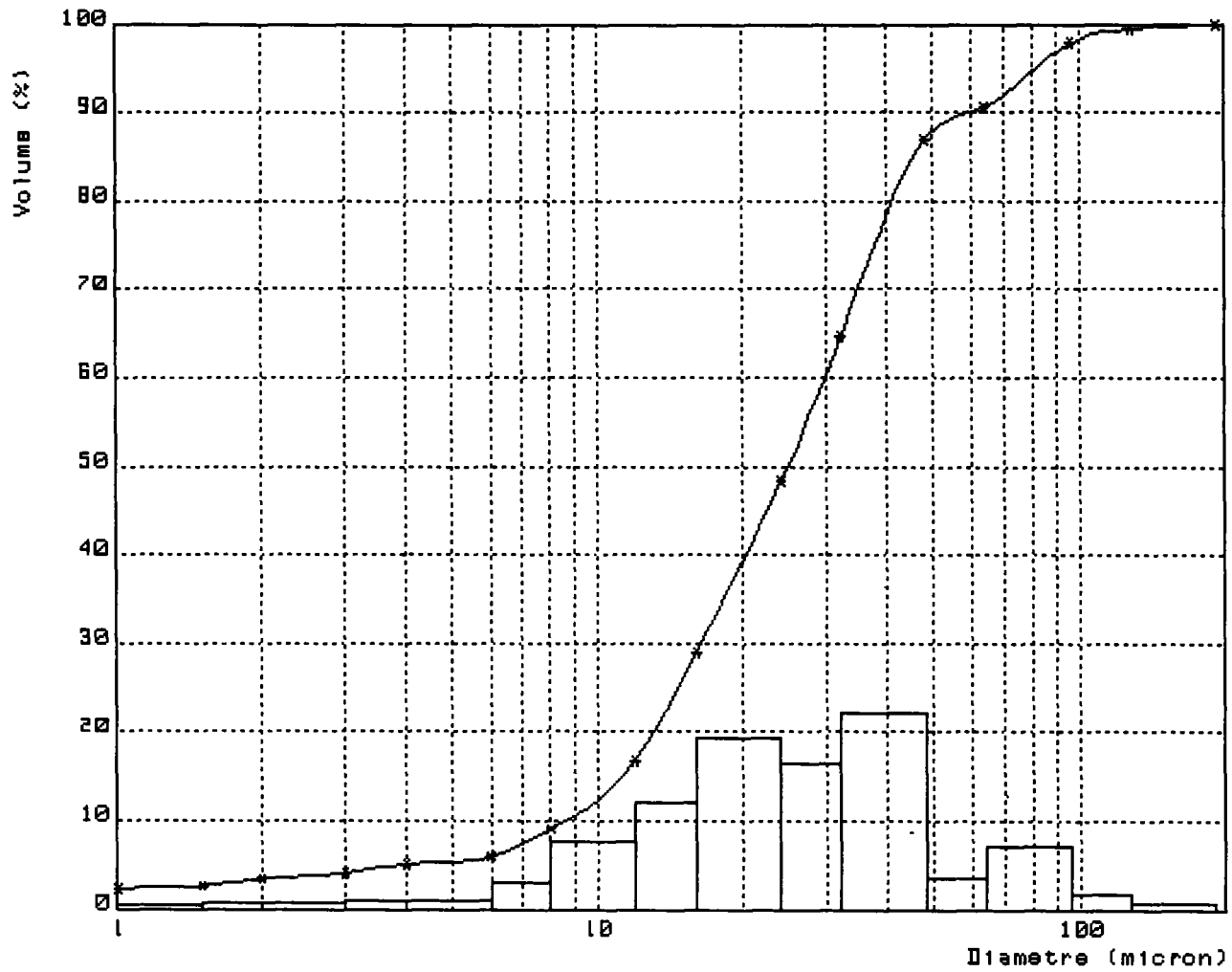
	Gravity preconcentrates		
	-0.08+0.01 mm n° 1	- 0.08+0.01 mm n° 2	-0.04+0.01 mm
Fe_2O_3 assay %	2.06	4.59	6.99
SrO assay %	12.02	9.73	11.53
BaO assay %	5.63	3.64	4.63
REO total, assay %	46.13	41.74	31.99

Fe_2O_3 contents in gravity preconcentrates account for residual Fe-Mn containing dolomite and siderite which should be removed by flotation prior to magnetic separation since this latter method is applicable to separation of monazite from strontianite in a final refining stage.

Size distribution of the - 0.08 + 0.01 mm gravity preconcentrate n° 2, determined by laser diffractometry is shown in the following diagram and table.

Granulomètre à laser CILAS 715

REO ORE SAMPLE N°2
Echantillon : -0.08+0.010mm(t) G.P.
Dispersant : HMP:3kg/t
Durée d'ultrasons : 20 s
Commentaire : -0.08+0.010mm(t) GRAVITY PRECONCENTRATE.



d20 = 13.1 d50 = 24.8 d80 = 40.9

HISTOGRAMME	
Classe	Volume %
<1	2.3
1-1.5	.3
1.5-2	.8
2-3	.7
3-4	1.0
4-6	.9
6-8	3.1
8-12	7.7
12-16	12.2
16-24	19.3
24-32	16.4
32-48	22.3
48-64	3.6
64-96	7.2
96-128	1.6
128-192	.6

PASSANTS CUMULES	
Diamètre	Volume %
1	2.3
1.5	2.6
2	3.4
3	4.1
4	5.1
6	6.0
8	9.1
12	16.8
16	29.0
24	48.3
32	64.7
48	87.0
64	90.6
96	97.8
128	99.4
192	100.0

2.5. DATA PROCESSING RELEVANT TO REE DETERMINATIONS ON THE REO ORE SAMPLE 1

Fractionations either by size, by density or by flotation allow distributions of REE and major elements to be compared, this can assist in determining associations of REE to major minerals and in identifying major REE bearing minerals.

Almost similar distributions of Y and 10 REE in size fractions and flotation products separated from the REO ore sample 2, clearly confirm conclusion of the mineralogic studies: monazite is the only major REE bearing mineral in REO ore sample 2. Occurrence of minor rare earth minerals do not significantly affect REE distributions.

As mentioned in paragraph 2.3.1. significant differences between distributions of Y and 14 REE, in size and density fractions separated from the REO ore sample 3, indicate the occurrence of at least two major REE bearing minerals which differ by their relative contents in heavy REO and Y (Yttrics).

Mineralogic investigations on the REO ore sample 1 have shown that REE are associated to apatite and silicates: Ce britholite, Y britholite and allanite. Compositions of fractions separated by sizing and flotation as well as the corresponding distributions for major and rare earth elements were subjected to a statistical analysis in order to reveal correlations and relate REE distributions to major elements distributions. Data from fifteen fractions:

- three size fractions: - 160 + 10 μm (t), - 10 μm (1), - 10 μm (2)
- twelve products from flotation tests n° 2, n° 20 and n° 24

were processed, data are shown in table n° 6, where as examples, the P_2O_5 and RP205 vectors give the P_2O_5 contents and recoveries (distributions) in fractions 1 to 15.

Correlation matrices for contents and distributions are shown in the appendix 4.

The most interesting correlation coefficients between contents for the 15 fractions are as follows:

variables	r	
P_2O_5-CaO	0.9953	
SiO_2-Al_2O_3	0.9778	
Fe_2O_3-MgO	0.8850	
P_2O_5-La	0.9334	
P_2O_5-Ce	0.9572	
P_2O_5-Eu	0.9968	
P_2O_5-Dy	0.9977	(highest correlation coefficient)
P_2O_5-Y	0.9933	

as expected, all REE are highly correlated.

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row	P2O5	CaO	SiO2	Fe2O3	Al2O3	MgO	LOI	La	Ce	Nd	Sm	Eu
1	3.19	7.41	57.3	13.22	7.91	1.5	0.73	1540	3010	1150	218.0	19.9
2	1.95	5.70	38.5	32.10	7.10	5.5	4.95	1440	2650	1020	195.0	18.3
3	1.50	4.55	58.2	12.60	9.65	4.3	3.85	740	1455	598	124.0	10.5
4	0.78	3.60	68.5	5.35	9.15	1.5	0.64	654	1200	360	70.0	5.8
5	7.25	14.50	48.9	8.40	6.80	2.4	1.95	3550	6200	2340	448.0	40.9
6	21.40	33.70	19.7	6.10	2.30	1.5	3.45	7530	13750	5775	1151.0	104.0
7	1.15	4.45	68.6	6.05	9.00	1.7	0.76	1302	1960	640	112.0	9.2
8	33.30	46.60	5.4	0.92	0.38	0.5	2.25	7600	13680	6615	1520.0	152.8
9	7.60	16.20	49.8	6.90	6.55	2.4	3.85	3116	5960	2380	480.0	39.7
10	30.40	44.90	8.3	1.45	0.68	0.6	3.85	7640	15620	7200	1520.0	144.4
11	5.10	12.70	53.2	7.10	6.85	2.3	3.30	3200	4980	2000	440.0	33.6
12	0.18	3.25	69.7	6.45	10.70	2.0	0.74	712	1200	330	50.8	4.9
13	32.90	46.20	5.9	1.35	0.46	0.5	1.95	7114	15350	7576	1655.0	152.0
14	8.51	17.15	47.3	8.21	6.30	2.3	2.84	4300	7440	2760	515.0	45.7
15	16.80	29.30	24.9	8.15	3.25	2.1	4.10	7094	12860	5208	996.0	96.0

row	Gd	Dy	Er	Yb	Y	RP2O5	RCaO	RSiO2	RFe2O3	RAl2O3	RMgO	RLOI
1	208.0	196.0	100.0	85.8	970	97.17	96.46	96.69	89.98	95.68	85.04	75.71
2	202.0	201.0	110.0	89.0	1030	2.62	3.27	2.86	9.62	3.78	13.84	22.46
3	116.8	110.0	61.0	49.5	540	0.21	0.27	0.45	0.40	0.54	1.12	1.83
4	60.6	50.0	30.8	29.0	268	17.27	36.27	87.82	73.04	87.85	73.60	47.25
5	450.0	410.0	230.0	196.0	2120	23.05	20.97	9.00	16.47	9.37	17.13	20.67
6	1110.0	995.0	572.0	580.0	5395	59.68	42.76	3.18	10.49	2.78	9.27	32.08
7	97.0	72.0	42.9	41.5	452	29.33	51.03	95.04	92.31	95.34	90.20	64.37
8	1612.0	1432.0	850.0	784.0	8040	42.48	26.73	0.37	0.70	0.20	1.37	9.53
9	412.0	352.0	208.0	198.0	2080	6.18	5.92	2.20	3.36	2.21	4.09	10.39
10	1600.0	1360.0	848.0	772.0	7880	18.29	12.15	0.27	0.52	0.17	0.74	7.69
11	380.0	314.0	174.0	150.0	1756	3.73	4.18	2.11	3.11	2.08	3.61	8.02
12	45.6	39.7	25.4	25.8	260	4.48	34.61	91.31	83.63	92.58	84.94	56.22
13	1590.0	1460.0	625.0	460.0	7150	60.90	36.61	0.58	1.30	0.30	1.71	11.03
14	485.0	425.0	215.0	162.0	2016	25.23	21.76	7.38	12.69	6.30	2.32	2.84
15	1010.0	835.0	402.0	338.0	3610	9.40	7.02	0.73	2.38	0.63	1.62	7.01

row	RLa	RCe	RNd	RSm	REu	RGd	RDy	REr	RYb	RY
1	95.84	96.06	96.02	95.97	95.88	95.66	95.44	95.12	95.39	95.30
2	3.95	3.72	3.75	3.78	3.88	4.09	4.31	4.61	4.36	4.46
3	0.21	0.21	0.23	0.25	0.23	0.25	0.25	0.27	0.25	0.24
4	30.97	31.59	25.29	25.06	23.42	22.87	21.35	22.67	22.28	21.41
5	24.13	23.43	23.61	23.03	23.71	24.58	25.14	24.31	21.62	24.32
6	44.90	44.98	51.10	51.90	52.88	52.75	53.51	53.02	56.11	54.28
7	63.40	58.58	50.24	44.48	40.93	40.52	36.75	36.65	37.89	39.08
8	18.51	20.45	25.97	30.19	34.00	33.68	36.55	36.32	35.80	34.77
9	4.84	5.68	5.95	6.08	5.63	5.48	5.73	5.66	5.76	5.73
10	8.78	11.01	13.33	14.24	15.15	15.77	16.37	17.09	16.63	16.07
11	4.47	4.27	4.50	5.01	4.29	4.55	4.60	4.27	3.93	4.36
12	37.21	34.11	24.63	19.71	20.57	18.65	18.23	23.83	29.67	23.35
13	27.67	32.47	42.08	47.79	47.49	48.41	49.89	43.64	39.38	47.78
14	26.78	25.20	24.55	23.81	22.87	23.64	23.26	24.04	22.21	21.57
15	8.34	8.22	8.74	8.69	9.07	9.30	8.63	8.49	8.75	7.29

Table 6: REO ore sample n° 1. Overall data relevant to chemical compositions, distributions of major and rare earth elements.

As far as distributions are concerned the most interesting correlation coefficients are:

variables			r
$RSiO_2$	-	RAl_2O_3	0.9999
RFe_2O_3	-	RAl_2O_3	0.9946
RFe_2O_3	-	RMgO	0.9959
$RSiO_2$	-	RMgO	0.9929
RP_2O_5	-	RCaO	0.8538
RCaO	-	$RSiO_2$	0.7170
RLa	-	RPO_5	0.7825
RLa	-	$RSiO_2$	0.7532
REu	-	RP_2O_5	0.9659
REu	-	$RSiO_2$	0.4855
RY	-	RP_2O_5	0.9654
RY	-	$RSiO_2$	0.4755

Values of correlation coefficients indicate that:

- SiO_2, Al_2O_3, Fe_2O_3, MgO are closely associated as constituent of the silicate gangue
- some of the REE are not fully associated to apatite, as previously shown by mineralogic studies.

Simple and multiple linear regressions were evaluated in an attempt to explain La, Ce, Eu, Y, distributions as a function of P_2O_5 and SiO_2 distributions. This was effected to estimate the degrees of association of REE to apatite and REE bearing silicates.

Regression equations were as follows:

Regression equations	Degree of freedom d.f. (error)	R squared	R squared adjusted for d.f.	F ratio
<u>Simple linear regression</u>:				
RLa = 0.7385 RP_2O_5+6.9722	13	0.6123		20.53
RCe = 0.7614 RP_2O_5+6.3606	13	0.6811		27.77
REu = 0.8790 RP_2O_5+3.2252	13	0.9330		180.93
RY = 0.8776 RP_2O_5+3.2645	13	0.9319		177.92
<u>Multiple linear regression</u>:				
RLa = 0.5949 RP_2O_5+0.3725 $RSiO_2$+0.8687	12	0.9383	0.9280	91.21
RCe = 0.6311 RP_2O_5+0.3380 $RSiO_2$+0.8232	12	0.9520	0.9556	151.76
REu = 0.8196 RP_2O_5+0.1542 $RSiO_2$+0.6996	12	0.9930	0.9918	851.68
RY = 0.8207 RP_2O_5+0.1476 $RSiO_2$+0.8468	12	0.9870	0.9849	457.28

It is interesting to note that constant terms in multiple linear regression equations were found to be not statistically significant at a 95 % confidence level, which confirms that REE are only associated to apatite, silico-phosphate, and silicates. Both F ratio and R^2 values clearly reveal significant improvements in the quality of the fit of predicted values to observed values for REE distributions when two independent variables: RP_2O_5 and $RSiO_2$ are used in the model instead of one (RP_2O_5). In the latter case, values of the regression coefficients: 0.7385 to 0.8790, clearly highlight the inadequacy of RP_2O_5 alone to explain variations of REE distributions, since values close to 1, should have been obtained if REE were fully associated to apatite.

It can be seen that quite satisfactory adjustments of predicted values to observed values are obtained with RP_2O_5 and $RSiO_2$ as the independent variables, since 93.8 to 99.3 % of the total variations in REE distributions, about the means, are explained by the regression equations.

Regressions coefficients indicate that about 61.5 % of La, 65.1 % of Ce, 84.2 % of Eu, 84.8 % of Y are associated to apatite and silicophosphate while silicates account for the remainder proportions.

Statistical analysis of the data clearly confirms the findings of the mineralogic studies, it can be concluded that most of the REE are associated to phosphate minerals, however, a significant portion of REE is also associated to silicates, this specially applies to light REE.

Detailed results from data processing are shown in the appendix 4.

3. PRELIMINARY FLOTATION TESTS

Exploratory laboratory test work was conducted on fractions separated from the rod milled and deslimed ores:

REO ore sample 1: - 160 + 10 μm (t) fraction after removal of magnetic particles extracted by low intensity magnetic separation

REO ore sample 2:
- - 160 + 10 μm (t) fraction (also identified as the - 160 + 8 μm fraction in flotation flowsheets)
- - 50 + 10 μm (t) fraction (also identified as the - 50 + 8 μm fraction in flotation flowsheets)
- - 80 + 10 μm gravity preconcentrate
- - 40 + 10 μm gravity preconcentrate

REO ore sample 3:
- - 125 + 10 μm (t) fraction
- - 10 μm (1) fraction

with the objectives:

- to assess their amenability to flotation using conventional flotation reagents and phosphoric ester (P.E.) collectors
- to define standard flotation flowsheet and procedure so as to compare performances of various types of phosphoric ester promoters elaborated by the laboratory for research and application of the C.E.C.A. Company at Corbehem.

Pups were conditioned either in flotation cells when low solid contents were used or in a variable speed turbine agitator when high solid concentrations (60 % and higher) were needed.

Flotation tests were made, using conventional laboratory mechanical flotation cells:

- Minemet cells: of the subaeration type and self aerating design that includes a control valve and an air flow meter (rotameter). The design also provides a metallic grid to separate the agitation zone containing the impeller from the upper quiescent froth zone.

A H1 model with a tank capacity of 2.5 l and a turbine agitator speed of 1940 r.p.m., a L3 model with a thank capacity of 0.8 l and a turbine agitator speed of 1 250 r.p.m., were used.

- Agitair cells of the subaeration type with external air supply, air rate is adjusted by a pressure control system, a control valve and a rotameter.

Transparent plastic tanks with 1.5 l and 5 l capacity were used, the agitator speed was 1 000 r.p.m. for the 1.5 l cell and 850 r.p.m. for the 5 l cell.

Aeration rates were adjusted to 3 Nl/min per litre of cell in rougher and scavenger flotation stages and 2 Nl/min per litre in cleaner stages.

Water used in tests had the following characteristics:

pH: 7.7, hardness: 12.07 T.H. degrees, resistivity at 20°C: 3 862 ohm/cm, Ca: 40.8 mg/l, Mg: 4.5 mg/l.

CO_2 saturated water was used in flotation tests carried out on the REO ore sample 2, CO_2 was also blown in the agitated pulp, when separating monazite from carbonates on the sample 2, as a gas mixture consisting of 10 % CO_2 and 90 % air.

3.1. PRELIMINARY TESTS ON THE REO ORE SAMPLE N° 1

Two series of tests were conducted:

- a first series using tall oil as a conventional apatite collector
- a second series using phosphoric esters as collectors for apatite.

3.1.1 Flotation with tall oil as a collector

Conventional flotation involved:

- conditioning at high solids concentration (60 % and 70 %) with sodium silicate as a depressant for silicate minerals and tall oil as an apatite collector
- depression of iron bearing silicates with a mixture of sodium silicate and sodium citrate, and/or with corn starch added at the first conditioning stage or at the cleaner stage.

Main characteristics of flotation reagents were as follows:

- NaOH, technical grade from Prolabo, used as a 100 g/l solution as a pH regulator, when necessary
- sodium silicate from Rhône-Poulenc, aqueous solution of density 1.33 with a SiO_2/Na_2O molar ratio of 3.4 to 3.5, with the average composition: 25.8 % SiO_2 - 7.7 % Na_2O - 66.3 % H_2O
- sodium citrate, technical grade neutral citrate from Prolabo, it was used as a binary solution consisting of sodium silicate and sodium citrate in a 5/1 weight ratio
- pregelatinized corn starch 12014 from Société des Produits du Maïs (french subsidiary of Corn Products)
- tall oil used as an aqueous emulsion of tall oil and diesel oil (10 % by volume of oil phase), with a weight ratio tall oil/diesel oil = 1, stabilized by NaOH (2.5 % by weight on the basis of the tall oil added). The emulsion was prepared by high speed agitation of a mixture of tall oil and diesel oil, incorporated progressively in alkalinized water, using a high speed disperser-homogeneizer Ultra-Turrax.

Low cost semi-refined tall oil used, was the HL grade from DRT (Derivés Résiniques et Terpéniques), with characteristics as follows:

  - . dark brown viscous liquid at about 20°C, viscosity: 15.25 Poises at 20°C, density: 0.98
  - . unsaponifiables: 28-32 %, acid number: 80-100
  - . iodine number: 120-150. Rosin acids: 15-20 %
  - . total fatty acids: about 30 % including: palmitic acid: 7-8 %, oleic acid: 9.6 %, linoleic acid: 6.6 %, other fatty acids: 6 %
- diesel oil was normal grade of fuel for diesel engines.

The Minemet Hl flotation cell was used for the conventional flotation tests n° 1 to 5.

Flowsheets and main operating conditions are shown in figures 1FT1 to 1FT5.

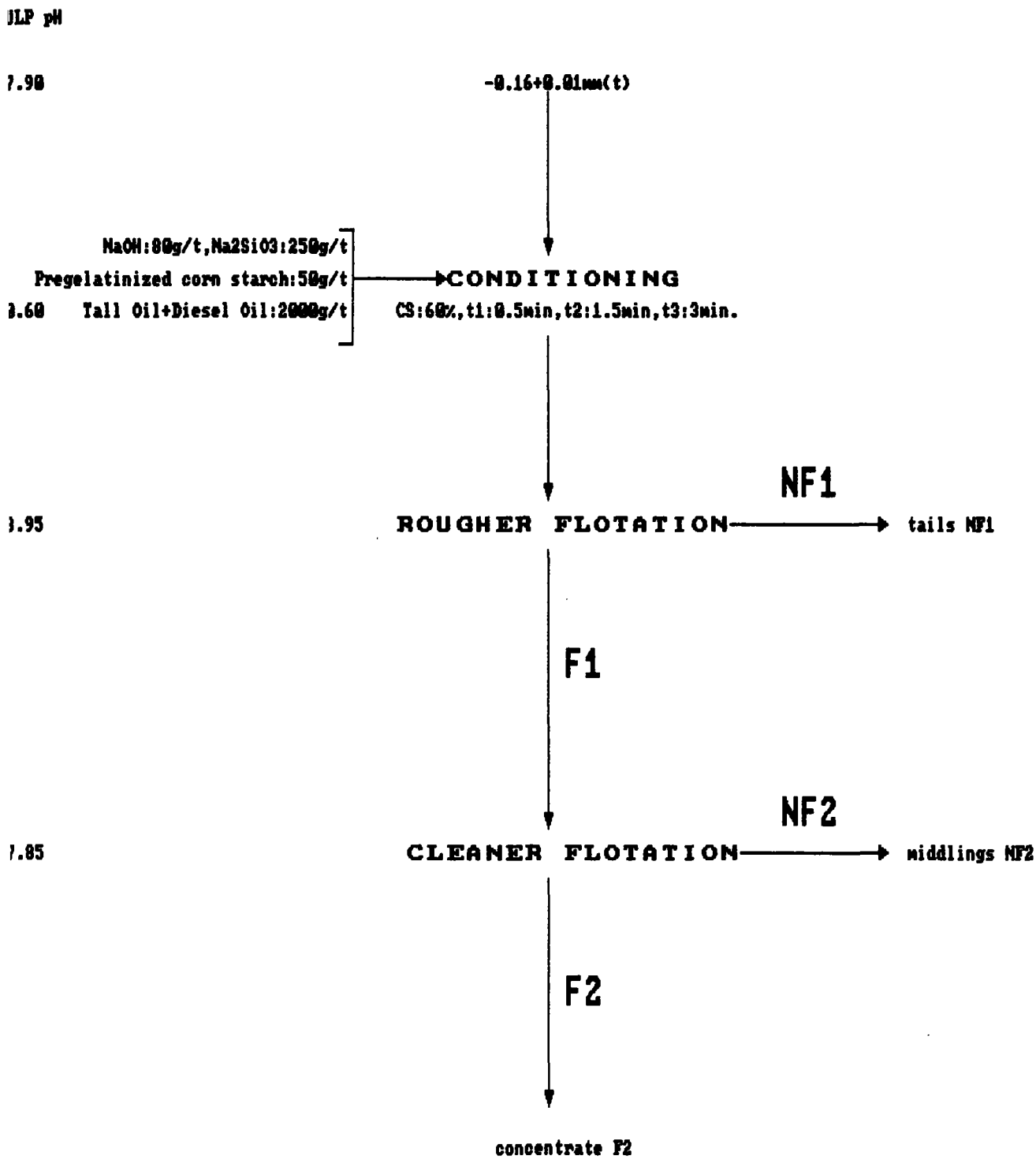


FIGURE N° 1FT1 : FLOWSHEET OF FLOTATION N° 1 ON REO ORE SAMPLE N°1

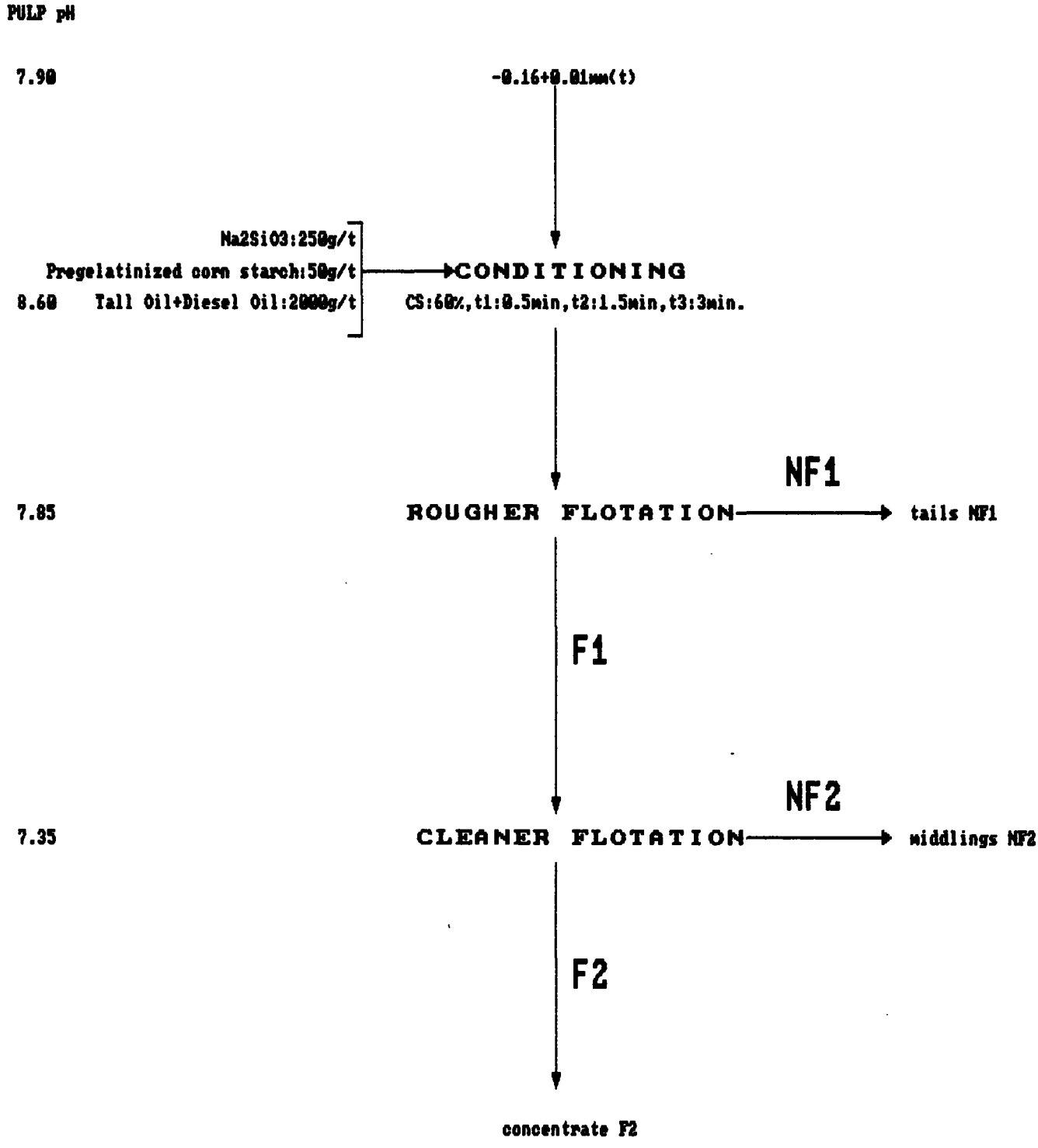


FIGURE N° 1FT2 : FLOWSHEET OF FLOTATION N° 2 ON REO ORE SAMPLE N°1

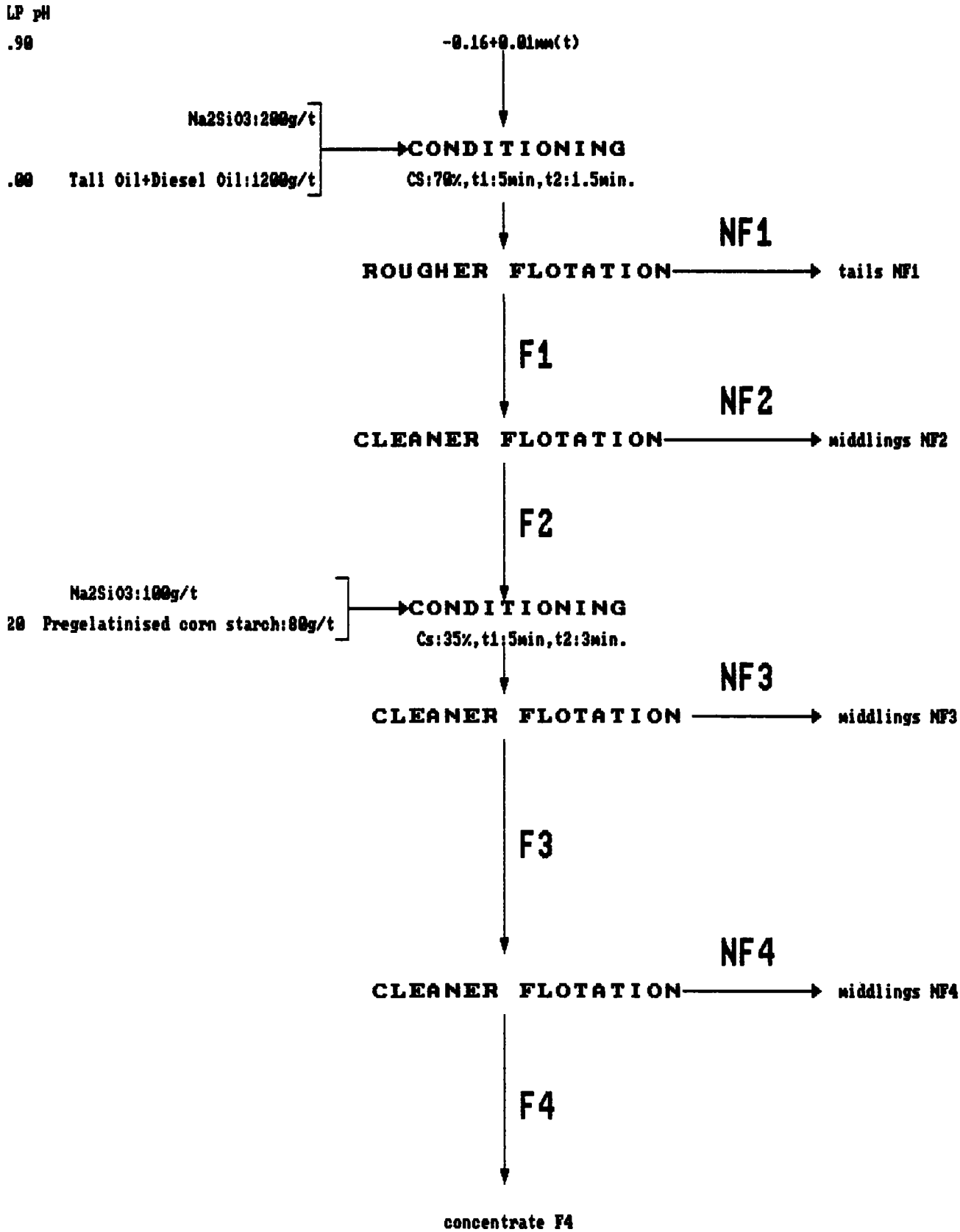


FIGURE N° 1FT3 : FLOWSHEET OF FLOTATION N° 3 ON REO ORE SAMPLE N°1

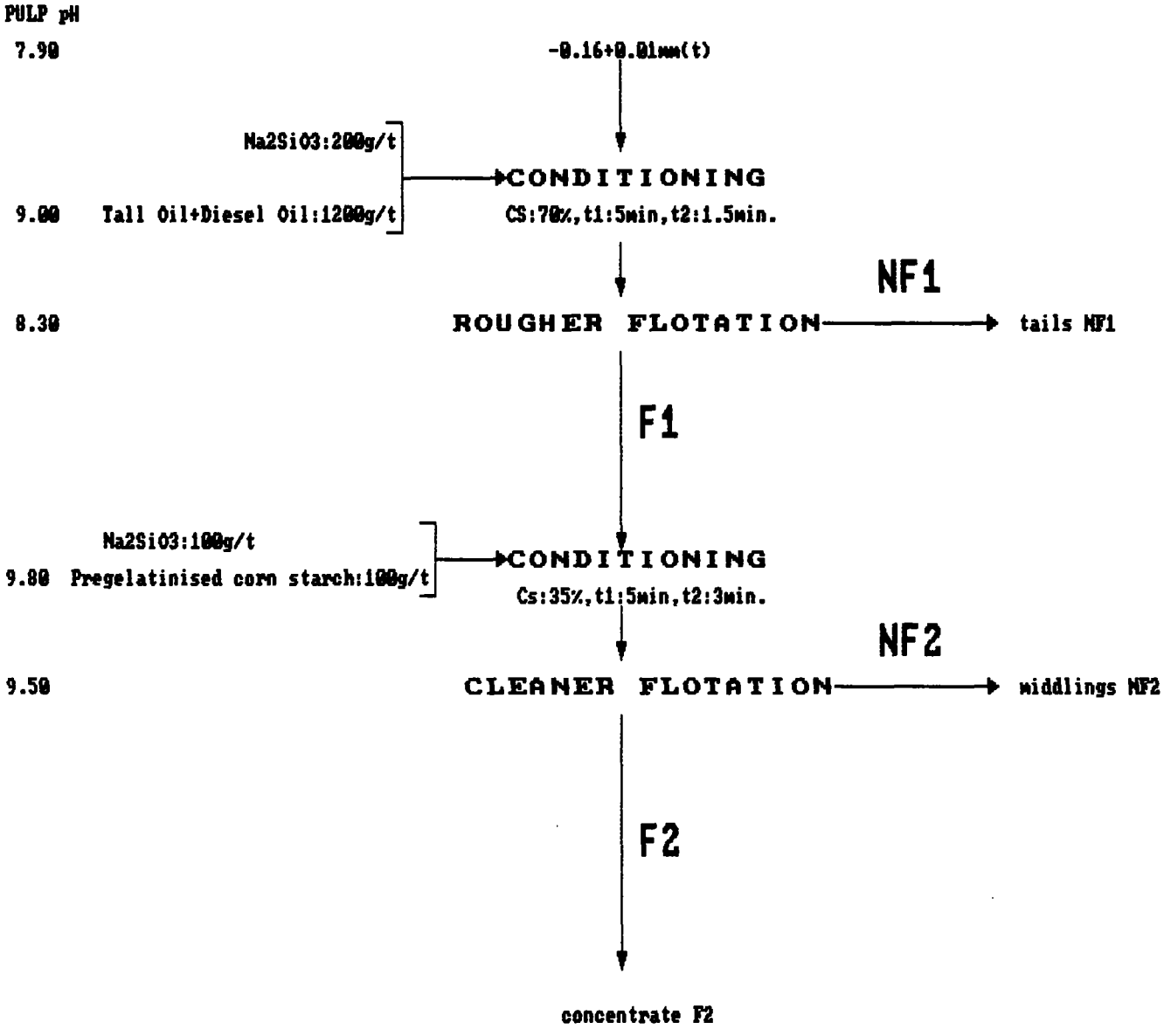


FIGURE N° 1FT4 : FLOWSHEET OF FLOTATION N° 4 ON REO ORE SAMPLE N°1

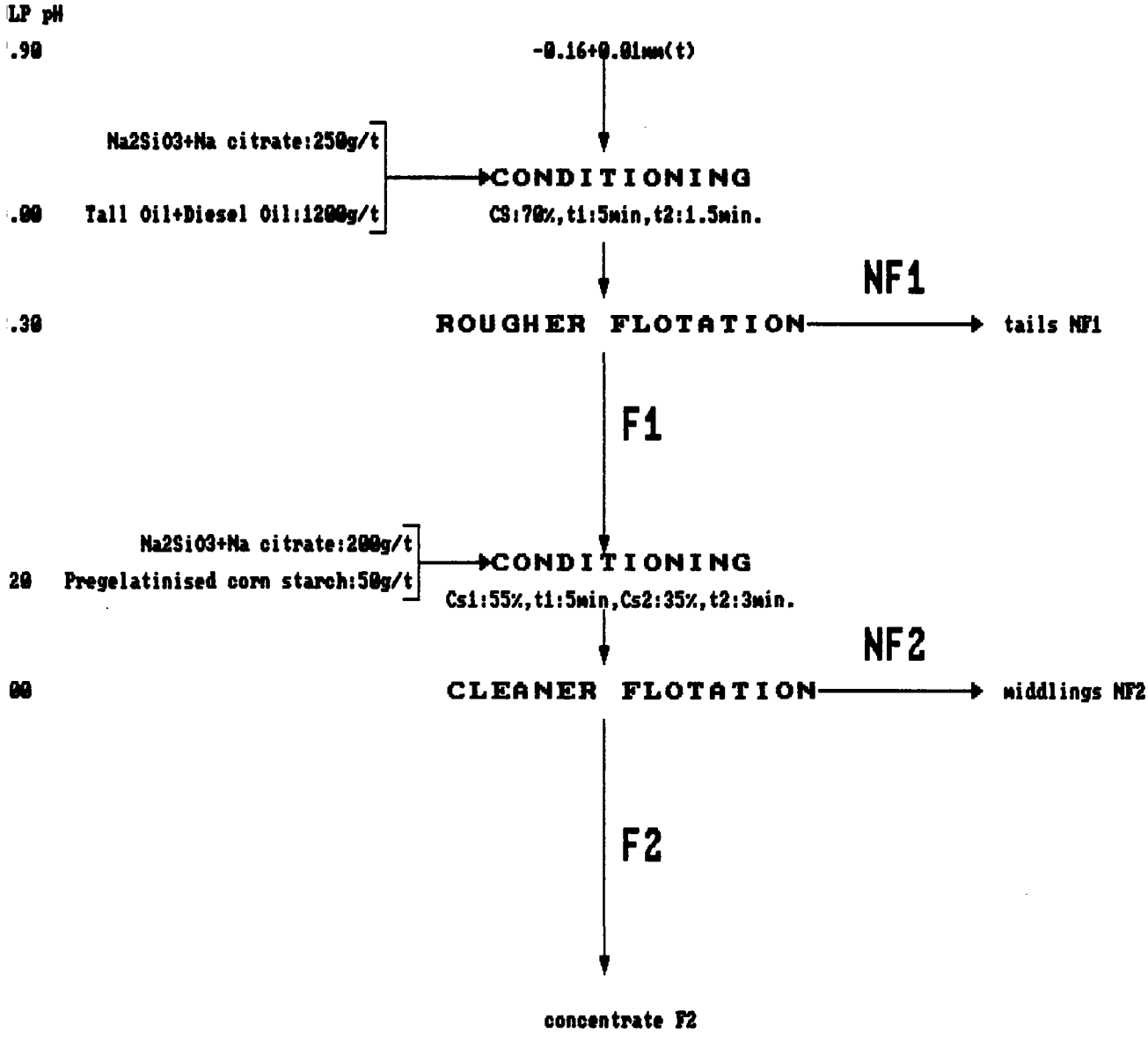


FIGURE N° 1FT5 : FLOWSHEET OF FLOTATION N° 5 ON REO ORE SAMPLE N°1

Multi elements determinations by XRF analysis on glass beads were made on products separated in tests 1 and 2, products from test n° 2 were also analyzed for REE. Material balances of tests 1 and 2 are shown in tables 1F01 and 1F02.

Products from test n° 3 were analyzed for P_2O_5, R_2O_3 and MgO, products from tests n° 4 and 5 were analyzed for P_2O_5 by XRF, material balances are shown in the following table:

	Weight %	P_2O_5		R_2O_3 content %	MgO content %
		Content %	Recovery %		
Test n° 3					
NF1	79.52	0.49	12.27	14.67	1.62
NF2	3.53	1.55	1.73	19.13	1.67
NF3	9.11	5.37	15.43	12.52	1.80
NF4	0.54	16.08	2.74	13.49	2.89
F4	7.30	29.45	67.83	6.20	0.86
calculated head	100.00	3.17	100.00	14.01	1.59
Test n° 4					
NF1	80.34	0.35	9.30		
NF2	12.39	5.05	20.70		
F2	7.27	29.10	70.00		
calculated head	100.00	3.02	100.00		
Test n° 5					
NF1	79.14	0.28	6.65		
NF2	12.15	4.44	16.20		
F2	8.71	29.50	77.15		
calculated head	100.00	3.33	100.00		

Concentration performance for P_2O_5 is shown as grade-recovery curves in figure n° 1.

Resultats clearly show that:

- a high level of solids concentration at the collector conditioning stage: Cs= 70 % instead of Cs= 60 %, results in a significant increase in selectivity and collecting power (tests 3 to 5)
- addition of corn starch is necessary to depress gangue minerals (test 3), addition in cleaner stages gives satisfactory selectivity
- the silicate-citrate mixture (test 5) substituted for silicate alone in primary and secondary conditioning stages provides maximum concentration performance

TABLE N° 1F01 : RED ORE SAMPLE N°1 GROUND DOWN TO 0.16mm,MATERIAL BALANCE OF FLOTATION TEST N°1 ON -0.16+0.010mm(t) FRACTION.

PRODUCTS	WEIGHT	P205		CaO		SiO2		Fe203	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1	72.67	.93	18.93	4.20	37.82	70.50	80.52	5.70	68.90
2 NF2	19.64	6.75	37.13	13.10	31.88	53.80	16.61	7.15	23.36
3 F2	7.69	20.40	43.94	31.80	30.30	23.80	2.88	6.05	7.74
CALCULATED HEAD	100.00	3.57	100.00	8.07	100.00	63.63	100.00	6.01	100.00

PRODUCTS	WEIGHT	A1203		MgO		L.O.I.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1	72.67	8.70	80.85	1.35	63.86	.70	48.99
2 NF2	19.64	6.55	16.45	2.20	28.13	1.60	30.27
3 F2	7.69	2.75	2.70	1.60	8.01	2.80	20.74
CALCULATED HEAD	100.00	7.82	100.00	1.54	100.00	1.04	100.00

PRODUCTS	WEIGHT %	CaO GRADE / P205 GRADE
1 NF1	72.67	4.5161
2 NF2	19.64	1.9407
3 F2	7.69	1.5588
CALCULATED HEAD	100.00	2.2604

TABLE N° 1F01 : FLOTATION TEST N°1.

PRODUCTS	WEIGHT	P205		CaO		Si02		Fe203	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1+NF2 1 2 0 0 0 0	92.31	2.17	56.06	6.09	69.70	66.95	97.12	6.01	92.26
2 F2 3 0 0 0 0 0	7.69	20.40	43.94	31.80	30.30	23.80	2.88	6.05	7.74
3 NF1 1 0 0 0 0 0	72.67	.93	18.93	4.20	37.82	70.50	80.52	5.70	68.90
4 F1 2 3 0 0 0 0	27.33	10.59	81.07	18.36	62.18	45.36	19.48	6.84	31.10

PRODUCTS	WEIGHT	A1203		MgO		L.O.I.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1+NF2 1 2 0 0 0 0	92.31	8.24	97.30	1.53	91.99	.89	79.26
2 F2 3 0 0 0 0 0	7.69	2.75	2.70	1.60	8.01	2.80	20.74
3 NF1 1 0 0 0 0 0	72.67	8.70	80.85	1.35	63.86	.70	48.99
4 F1 2 3 0 0 0 0	27.33	5.48	19.15	2.03	36.14	1.94	51.01

TABLE N° 1F02A : REO ORE SAMPLE N°1 GROUND DOWN TO 0.16mm,MATERIAL BALANCE OF FLOTATION TEST N°2 ON -0.16+0.010mm(t) FRACTION.

PRODUCTS	WEIGHT	P205		CaO		SiO2		Fe2O3	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1	78.77	.78	17.27	3.60	36.27	68.50	87.82	5.35	73.04
2 NF2	11.31	7.25	23.05	14.50	20.97	48.90	9.00	8.40	16.47
3 F2	9.92	21.40	59.68	33.70	42.76	19.70	3.18	6.10	10.49
CALCULATED HEAD	100.00	3.56	100.00	7.82	100.00	61.44	100.00	5.77	100.00

PRODUCTS	WEIGHT	Al2O3		MgO		L.O.I.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1	78.77	9.15	87.85	1.45	73.60	.64	47.25
2 NF2	11.31	6.80	9.37	2.35	17.13	1.95	20.67
3 F2	9.92	2.30	2.78	1.45	9.27	3.45	32.08
CALCULATED HEAD	100.00	8.20	100.00	1.55	100.00	1.07	100.00

PRODUCTS	WEIGHT %	CaO GRADE / P2O5 GRADE
1 NF1	78.77	4.6154
2 NF2	11.31	2.0000
3 F2	9.92	1.5748
CALCULATED HEAD	100.00	2.1980

TABLE N°1F02 A : FLOTATION TEST N°2.

PRODUCTS	WEIGHT	P205		CaO		SiO2		Fe203	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1+NF2 1 2 0 0 0 0	90.08	1.59	40.32	4.97	57.24	66.04	96.82	5.73	89.51
2 F2 3 0 0 0 0 0	9.92	21.40	59.68	33.70	42.76	19.70	3.18	6.10	10.49
3 NF1 1 0 0 0 0 0	78.77	.78	17.27	3.60	36.27	68.50	87.82	5.35	73.04
4 F1 2 3 0 0 0 0	21.23	13.86	82.73	23.47	63.73	35.26	12.18	7.33	26.96

PRODUCTS	WEIGHT	A1203		MgO		L.O.I.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1+NF2 1 2 0 0 0 0	90.08	8.85	97.22	1.56	90.73	.80	67.92
2 F2 3 0 0 0 0 0	9.92	2.30	2.78	1.45	9.27	3.45	32.08
3 NF1 1 0 0 0 0 0	78.77	9.15	87.85	1.45	73.60	.64	47.25
4 F1 2 3 0 0 0 0	21.23	4.70	12.15	1.93	26.40	2.65	52.75

TABLE N° 1F02B : RED ORE SAMPLE N°1 GROUND DOWN TO 0.16mm,MATERIAL BALANCE OF FLOTATION TEST N°2 ON -0.16+0.010mm(t) FRACTION.

PRODUCTS	WEIGHT	La		Ce		Nd		Sm	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 NF1	78.77	654.00	30.97	1200.00	31.59	360.00	25.29	70.00	25.06
2 NF2	11.31	3550.00	24.13	6200.00	23.43	2340.00	23.61	448.00	23.03
3 F2	9.92	7530.00	44.90	13570.00	44.98	5775.00	51.10	1151.00	51.90
CALCULATED HEAD	100.00	1663.64	100.00	2992.60	100.00	1121.11	100.00	219.99	100.00

PRODUCTS	WEIGHT	Eu		Gd		Dy		Er	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 NF1	78.77	5.80	23.42	60.60	22.87	50.00	21.35	30.80	22.67
2 NF2	11.31	40.90	23.71	450.00	24.38	410.00	25.14	230.00	24.31
3 F2	9.92	104.00	52.88	1110.00	52.75	995.00	53.51	572.00	53.02
CALCULATED HEAD	100.00	19.51	100.00	208.74	100.00	184.46	100.00	107.02	100.00

TABLE N°1F02 B : FLOTATION TEST N°2.

PRODUCTS	WEIGHT	La		Ce		Nd		Sm	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 NF1+NF2 1 2 0 0 0 0	90.08	1017.61	55.10	1827.78	55.02	608.60	48.90	117.46	48.10
2 F2 3 0 0 0 0 0	9.92	7530.00	44.90	13570.00	44.98	5775.00	51.10	1151.00	51.90
3 NF1 1 0 0 0 0 0	78.77	654.00	30.97	1200.00	31.59	360.00	25.29	70.00	25.06
4 F1 2 3 0 0 0 0	21.23	5409.71	69.03	9643.73	68.41	3945.05	74.71	776.49	74.94

PRODUCTS	WEIGHT	Eu		Gd		Dy		Er	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 NF1+NF2 1 2 0 0 0 0	90.08	10.21	47.12	109.49	47.25	95.20	46.49	55.81	46.98
2 F2 3 0 0 0 0 0	9.92	104.00	52.88	1110.00	52.75	995.00	53.51	572.00	53.02
3 NF1 1 0 0 0 0 0	78.77	5.80	23.42	60.60	22.87	50.00	21.35	30.80	22.67
4 F1 2 3 0 0 0 0	21.23	70.38	76.58	758.39	77.13	683.35	78.65	389.80	77.33

TABLE N° 1F02C : RED ORE SAMPLE N°1 GROUND DOWN TO 0.16mm,MATERIAL BALANCE OF FLOTATION TEST N°2 ON -0.16+0.010mm(t) FRACTION.

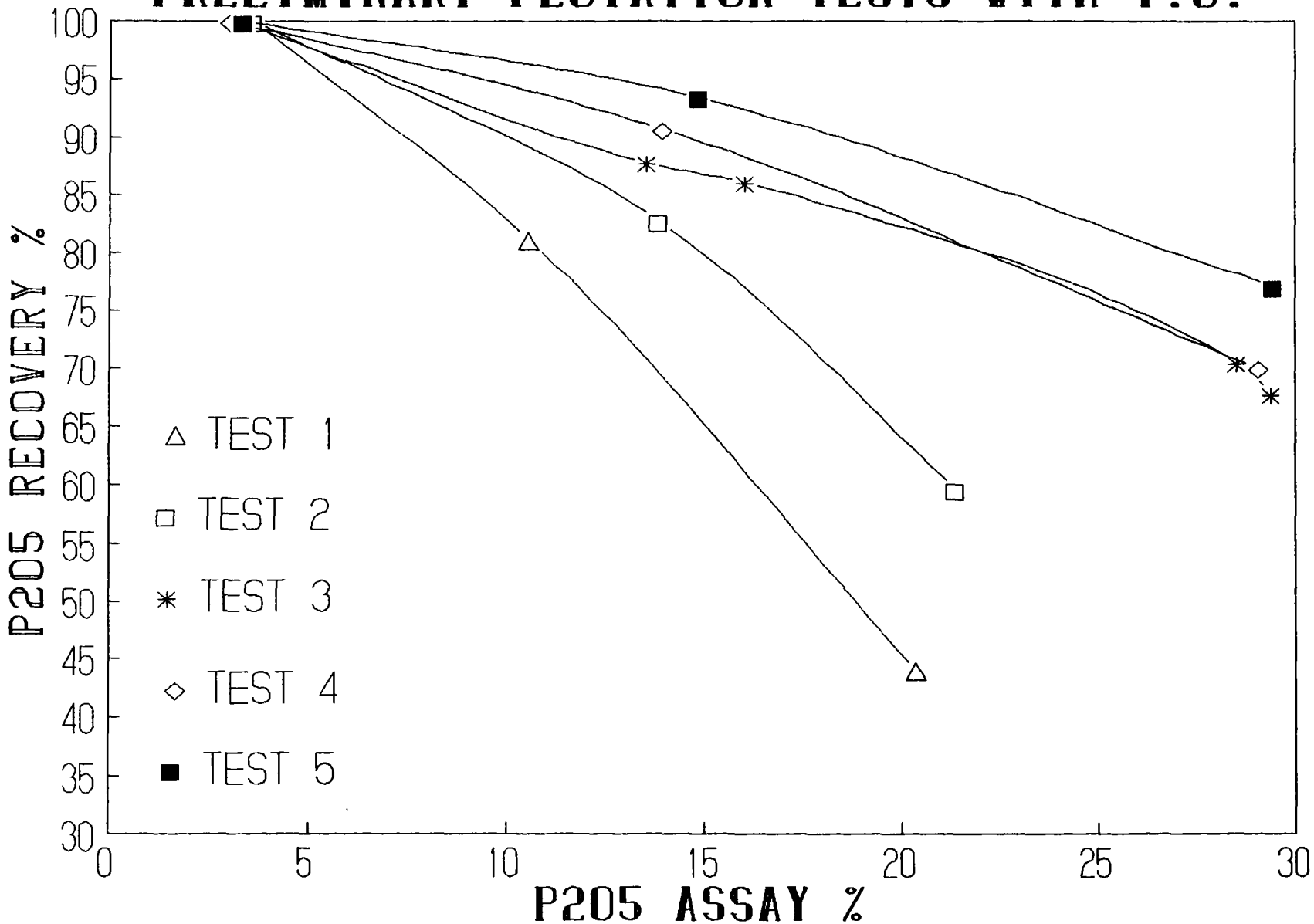
PRODUCTS	WEIGHT	Yb		Y	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 NF1	78.77	29.00	22.28	268.00	21.41
2 NF2	11.31	196.00	21.62	2120.00	24.32
3 F2	9.92	580.00	56.11	5395.00	54.28
CALCULATED HEAD	100.00	102.55	100.00	986.06	100.00

TABLE N° 1F02 C : FLOTATION TEST N°2.

PRODUCTS	WEIGHT	Yb		Y	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 NF1+NF2 1200	90.08	49.97	43.89	500.53	45.72
2 F2 3000	9.92	580.00	56.11	5395.00	54.28
3 NF1 1000	78.77	29.00	22.28	268.00	21.41
4 F1 2300	21.23	375.43	77.72	3650.29	78.59

FIGURE N I REU URE SAMPLE I

PRELIMINARY FLOTATION TESTS WITH T.O.



- flotation with cheap semi-refined tall oil leads to high contents in residual R_2O_3+MgO. Final concentrate from test 3 assays 29.45 % P_2O_5 with a high $(R_2O_3+MgO)/P_2O_5$ ratio value of 0.240 which by far exceeds the usual tolerable value of about 0.09 to 0.1, required for production of wet process phosphoric acid (W.P.A.)

- a high pH value (10.6) in conditioning with starch is detrimental to selectivity (comparison of tests 1 and 2 with pH values of 10.6 and 8.6 in primary conditioning).

3.1.2. Flotation with phosphoric esters as apatite collectors

Apatite was floated with a phosphoric ester in an alkaline pulp. Pregelatinized corn starch (12014), dextrin, sodium silicate, were evaluated as gangue depressants and dispersants using basic flowsheets shown in figures 1F01 to 1F08.

In flowsheets 1a to 4a, the - 0.16 + 0.01 mm (t) fraction (N.M.) is subjected to a rougher flotation stage, the rougher concentrate is cleaned to produce a final concentrate F3 and flotation middlings NF3. Rougher tailings NF1 are subjected to scavenger flotation to produce a concentrate F2 and final tailings NF2. Depressants and dispersants were added in the primary conditioning stage, as follows:

flowsheet 1a: Na silicate alone
flowsheet 2a: Na silicate + pregelatinized corn starch
flowsheet 3a: Na silicate + dextrin
flowsheet 4a: pregelatinized corn starch + Na silicate.

In flowsheets 1b to 4b, the - 0.16 + 0.01 mm (t) fraction (N.M.) is subjected to a rougher flotation stage, the rougher concentrate is cleaned to produce a final concentrate F3 and flotation middlings NF3. Rougher tailings are subjected to scavenger flotation to produce final tailings NF2. The scavenger concentrate F2 is cleaned to produce a final concentrate F4 and flotation middlings NF4. Depressants and dispersants were added in the primary conditioning stage, as follows:

flowsheet 1b: Na silicate alone
flowsheet 2b: Na silicate+pregelatinized corn starch
flowsheet 3b: Na silicate+dextrin
flowsheet 4b: pregelatinized corn starch + Na silicate.

Exploratory tests (flotation tests n° 10 and 17) were made to remove residual silicate minerals from scavenger apatite concentrates by cationic flotation, using amine acetate as a collector in a near neutral pH pulp (7.75), and a polymer as an apatite depressant. Characteristics of the flotation reagents used were as follows:

- cationic collector: Noramac C from CECA, primary amine acetate derived from coco fatty acids with the following distribution of hydrophobic chains:

Number of carbon atoms in hydrophobic radicals	%
C8	3
C10	6
C12	55
C14	18
C16	10
C18	2
C18 (with a double bond)	5

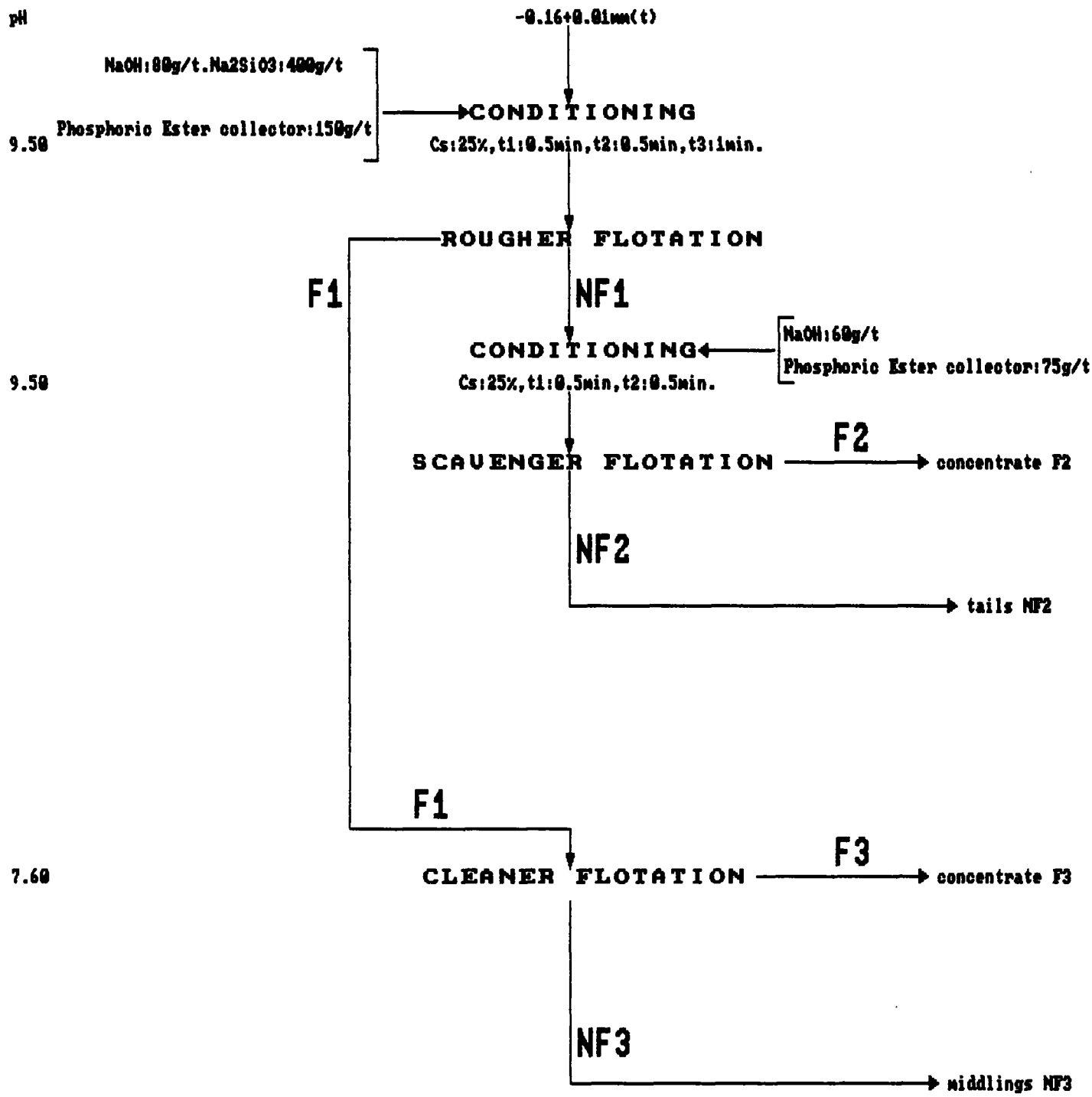


figure n° 1F01 : basic flowsheet n°1a for flotation of the REO ore sample n°1.

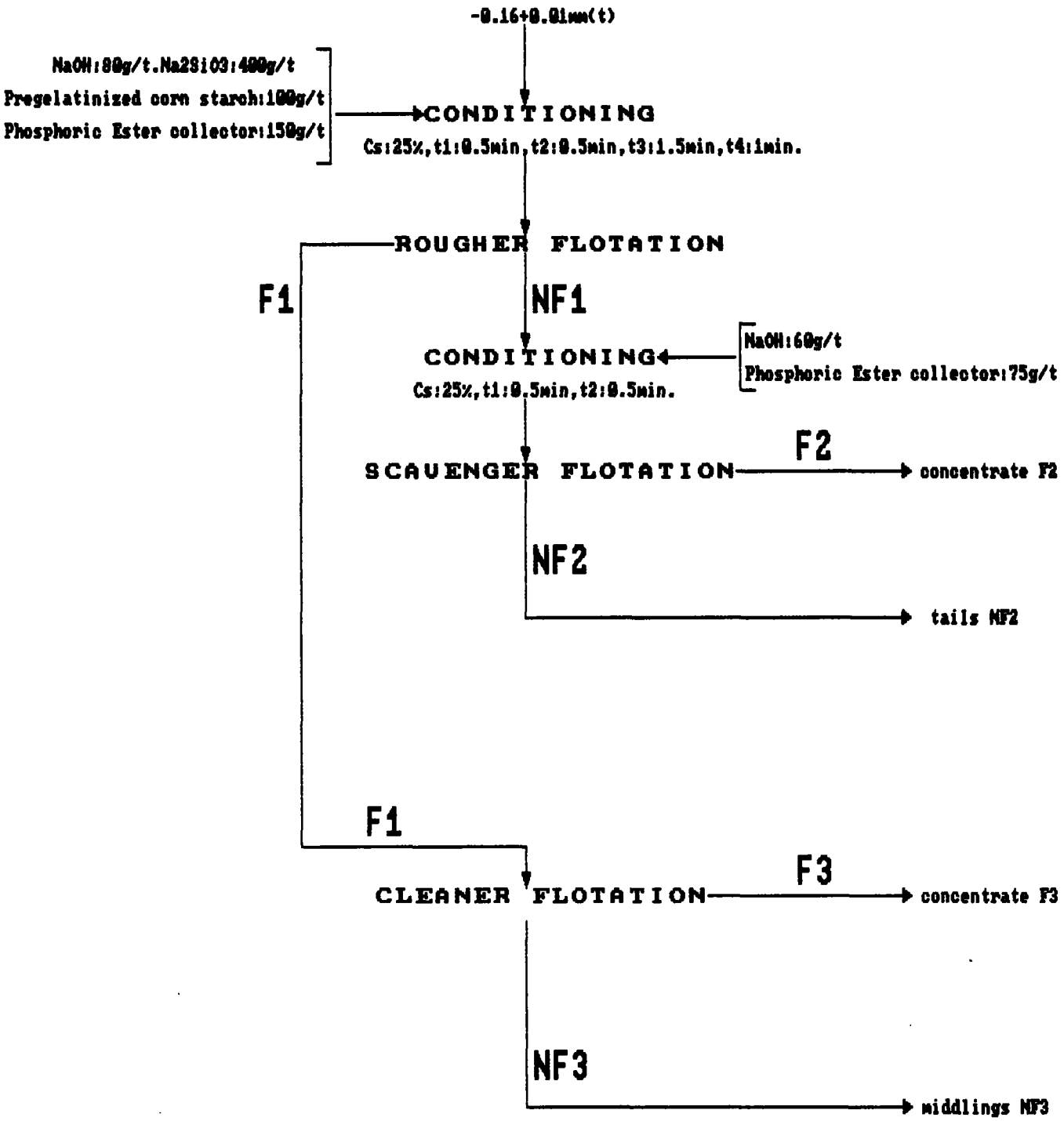


figure n° 1F02 : basic flowsheet n°2a for flotation of the REO ore sample n°1.

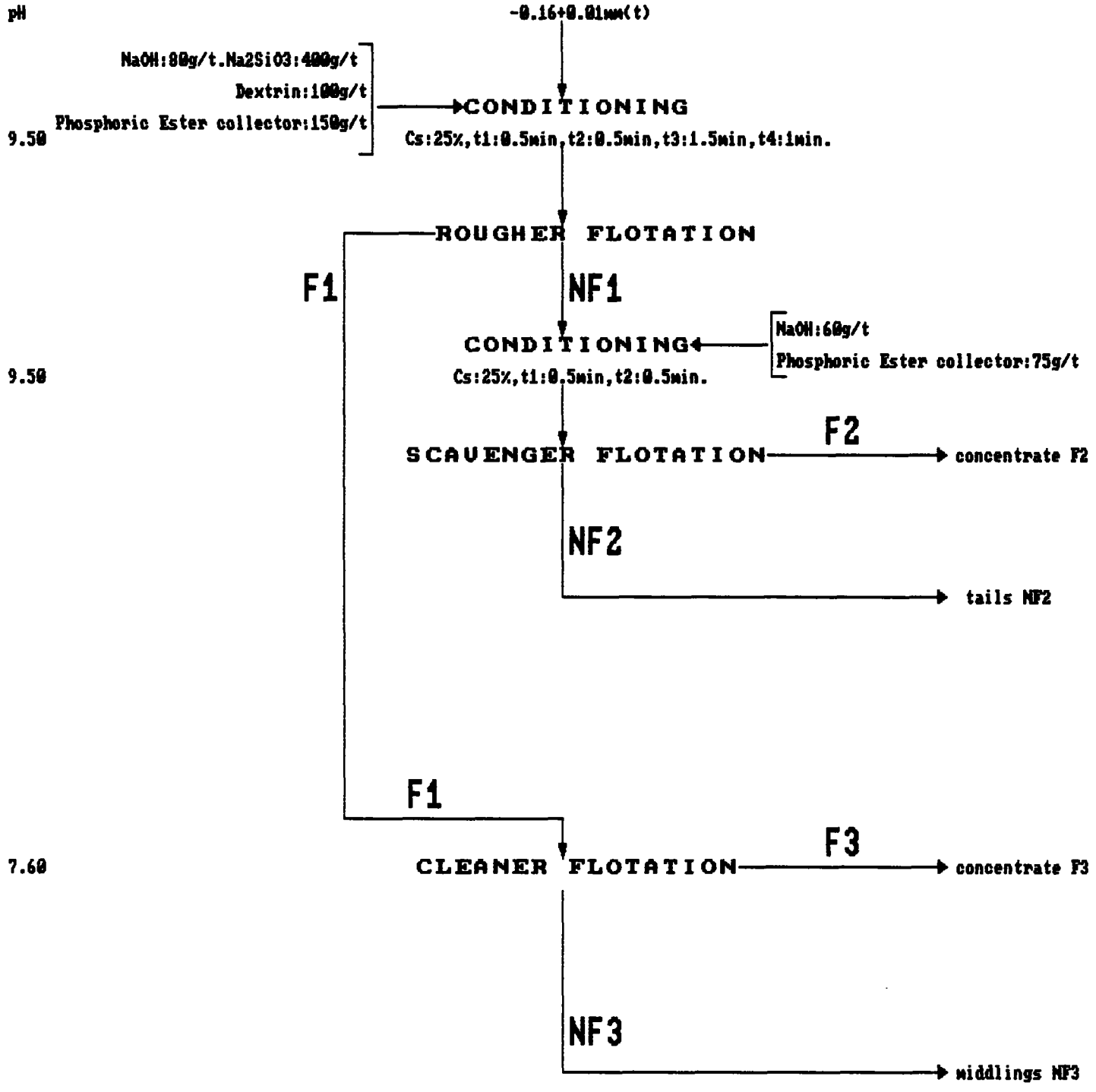


figure n° 1F03 : basic flowsheet n°3a for flotation of the REO ore sample n°1.

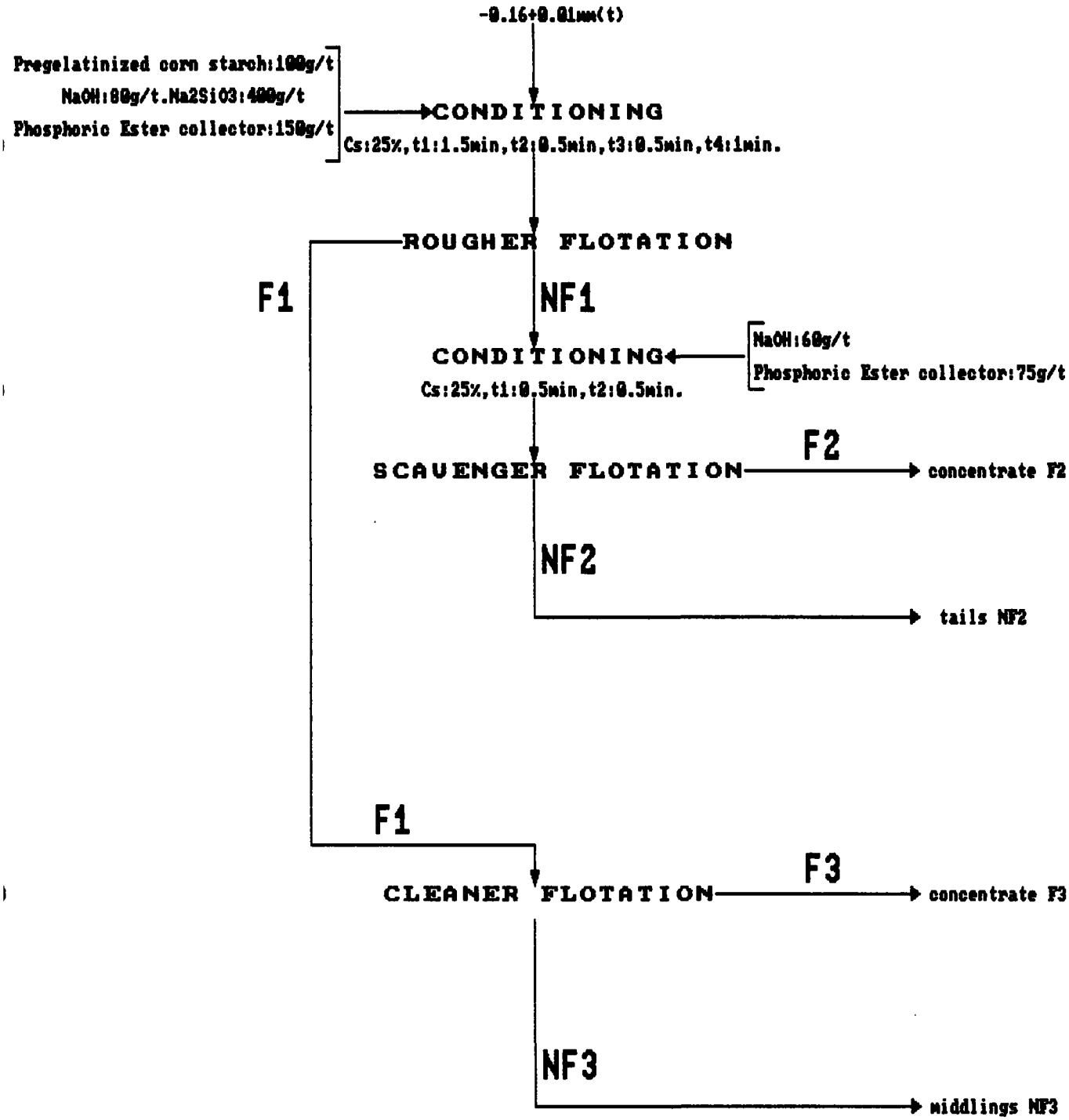


figure n° 1F04 : basic flowsheet n°4a for flotation of the REO ore sample n°1.

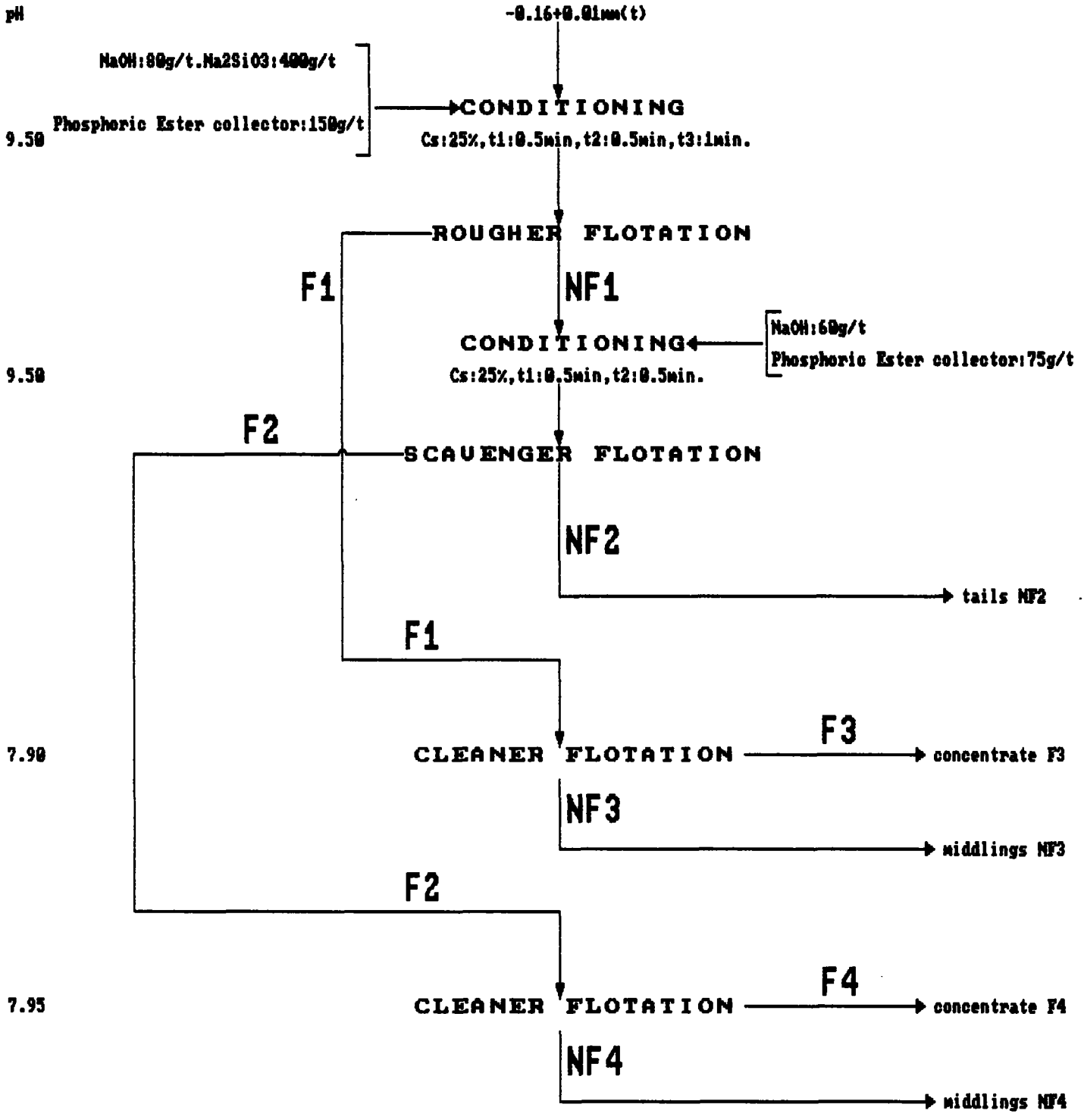


figure n° 1F05 : basic flowsheet n°1b for flotation of the REO ore sample n°1.

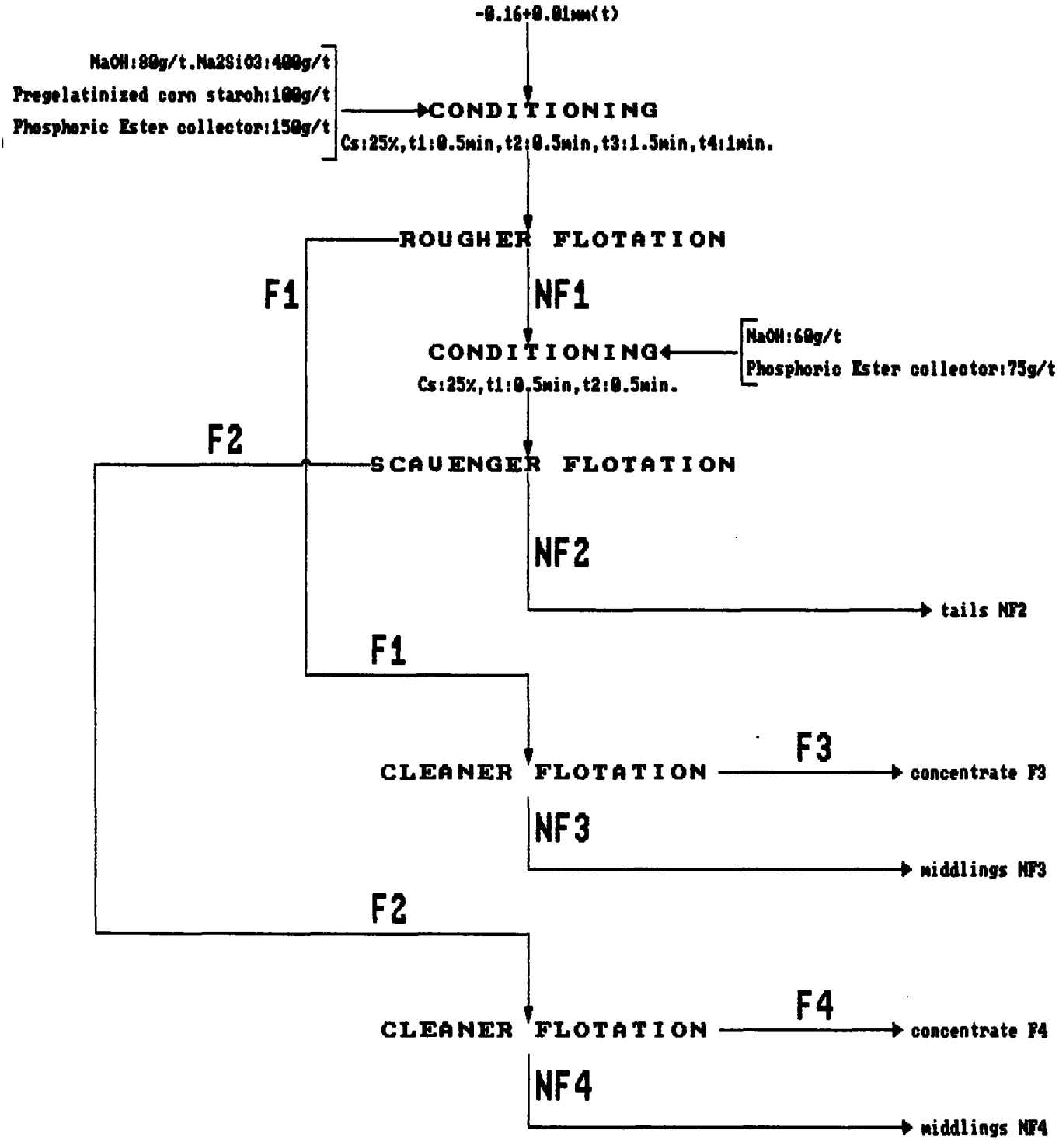


figure n° 1F06 : basic flowsheet n°2b for flotation of the REO ore sample n°1.

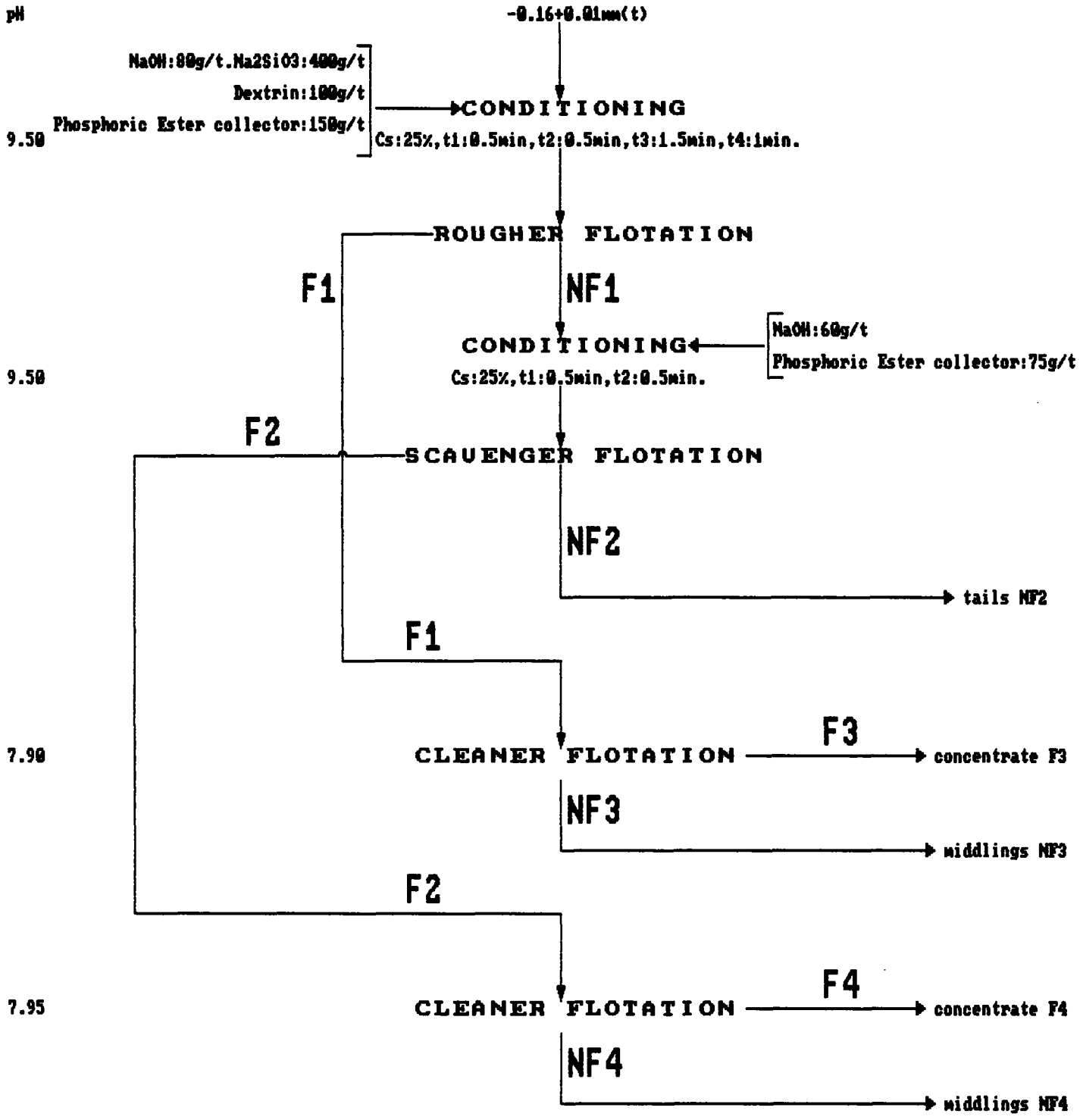


figure n° 1F07 : basic flowsheet n°3b for flotation of the REO ore sample n°1.

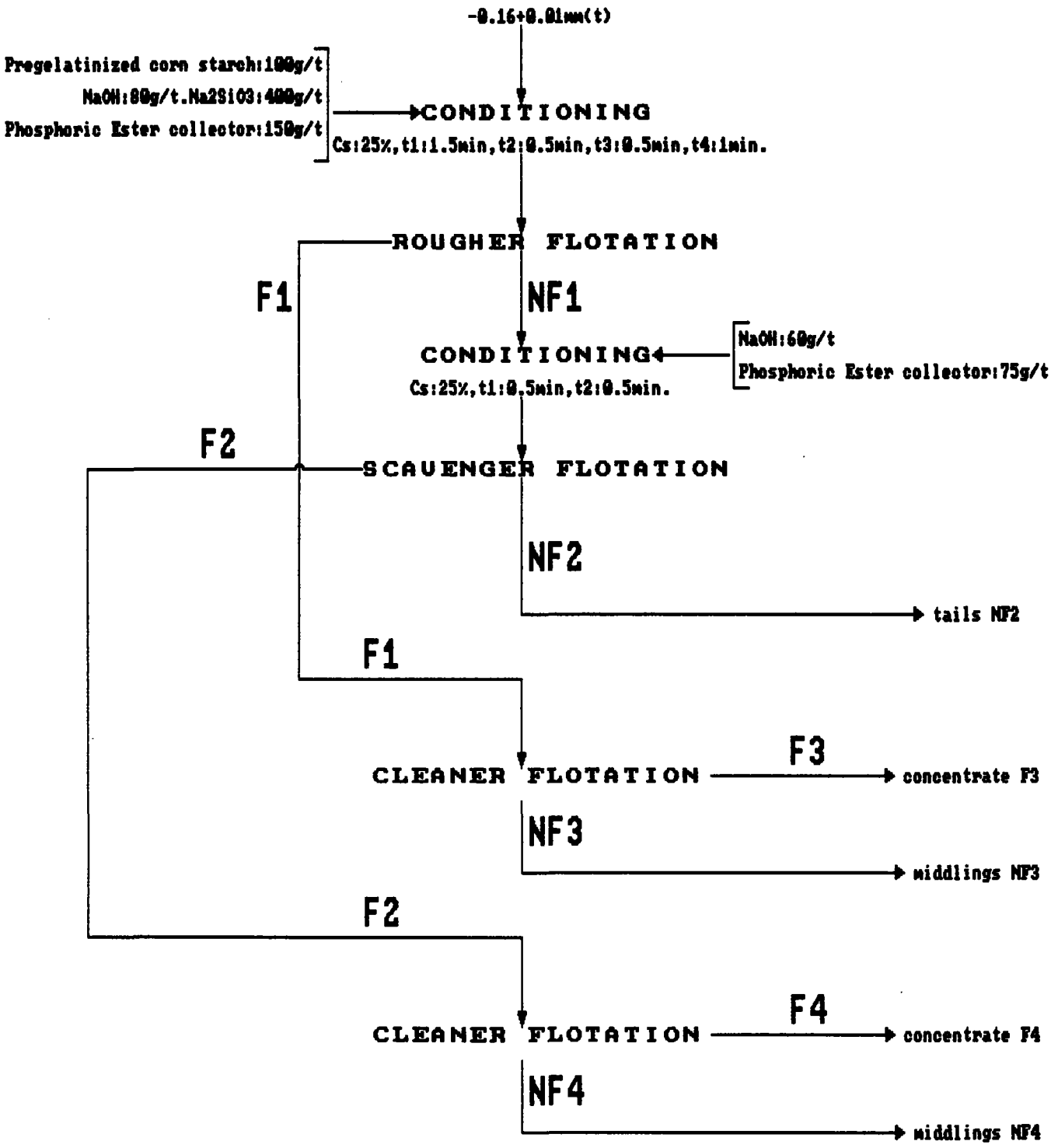


figure n° 1F08 : basic flowsheet n°4b for flotation of the REO ore sample n°1.

- polymer depressant: S4852 from American Cyanamid, probably of the low molecular weight polyacrylonitrile type
- frother: AF 65 from American Cyanamid.

Flowsheets for flotation tests n° 10 and 17 are shown in figures 1F10 and 1F17.

Reagents used in phosphoric ester flotation stages: rougher and scavenger, were as follows:

- gangue depressants and dispersants:
  - Na silicate, same grade as previously used in tall oil flotation, (paragraph 3.1.1.)
  - pregelatinized corn starch , 12014 from SPM
  - dextrin, standard technical grade from Prolabo
- phosphoric ester promoters
  main characteristics of P.E. promoters from GERLAND-CECA are given in chapter 4. Phosphoric ester grades used in preliminary tests conducted on the REO ore sample 1 were : P301, P312, B110 and 88094.

Pulp pH in low density pulp conditioning (solids concentration equal to 25 % or lower) was about 9.5.

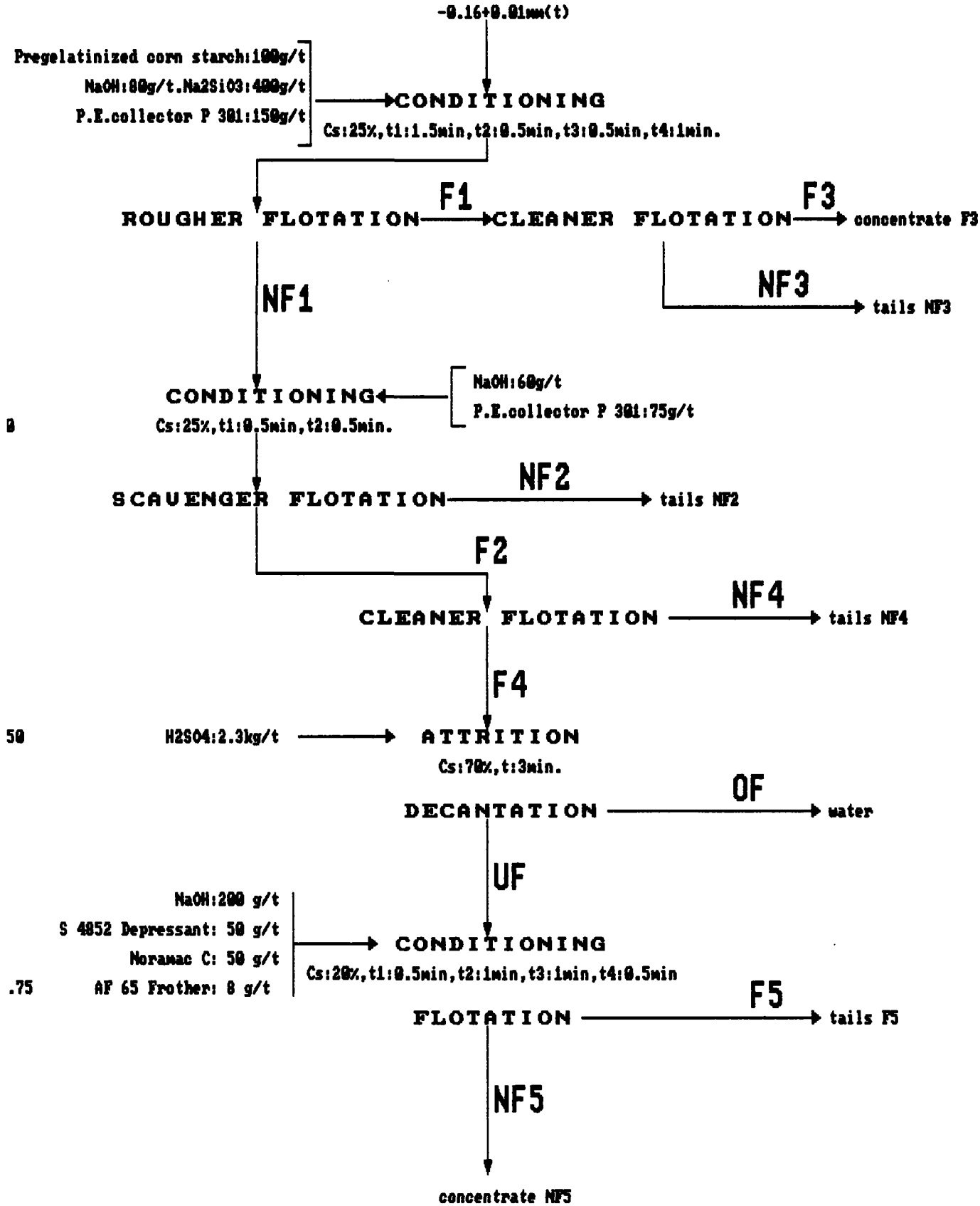


figure n° 1F10 : flowsheet for flotation n°10 on the REO ore sample n°1.

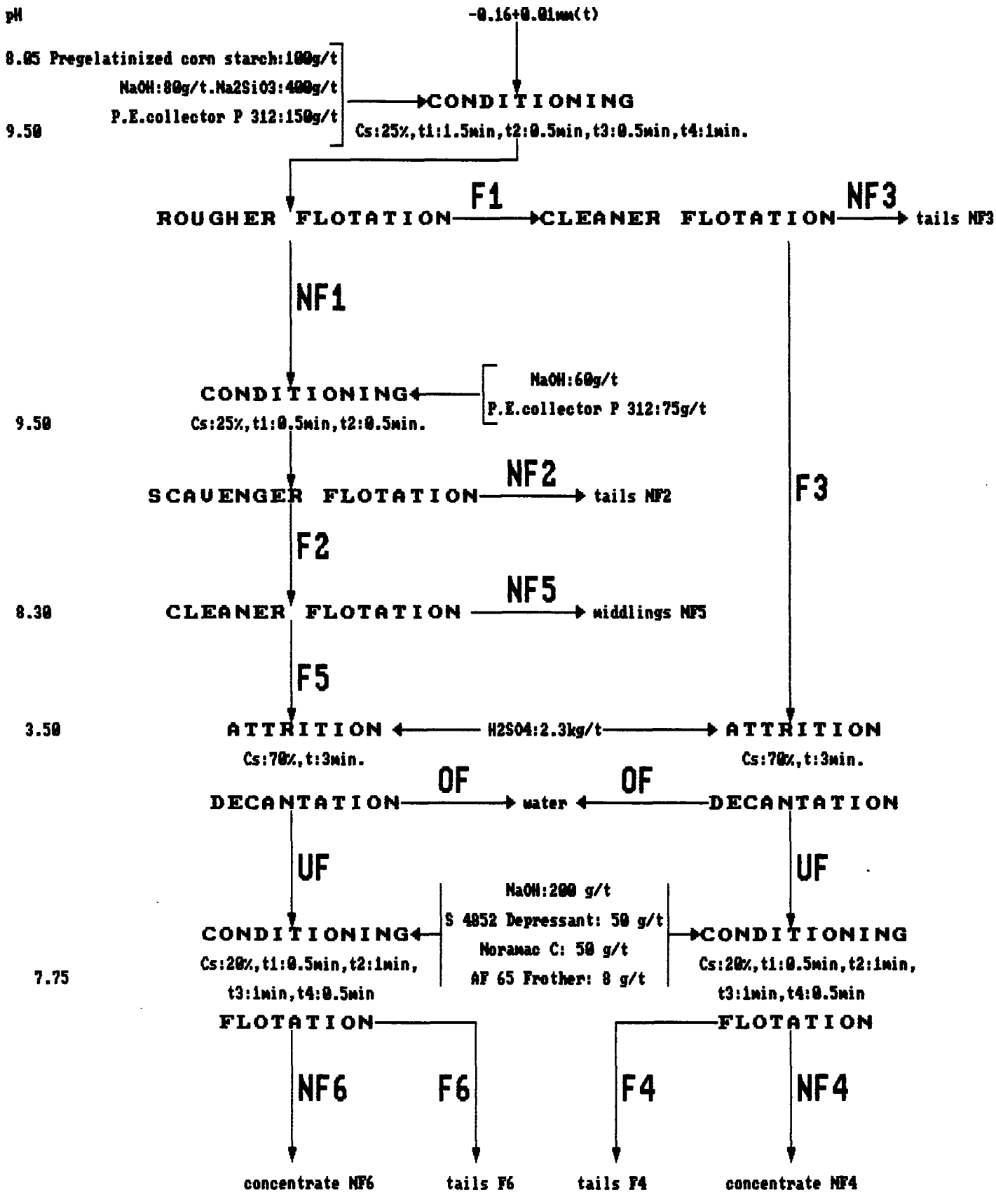


figure n° 1F17 : flowsheet for flotation n°17 on the REO ore sample n°1.

Products separated in flotation tests n° 10, 17, 19, 20, 21, 22, 24 were subjected to multi elements analysis by XRF on glass beads: P_2O_5, CaO, SiO_2, Fe_2O_3, Al_2O_3, MgO, LOI, optionally Na_2O, K_2O, TiO_2, MnO were determined.

Products separated in flotation tests 20 and 24 were also analyzed for REE by ICP/MS.

Detailed material balances are shown in tables:

1F10, 1F17, 1F19, 120A to 120C, 1F21, 122A, 122B, 124A to 124 C.

Products from flotation tests 7 to 10, 16 to 24 were analyzed for P_2O_5 by XRF, material balances are shown in the synthesis table n° 7.

Grade-recovery curves for P_2O_5 and basic flowsheets b, are shown in figures 2 and 3.

It can be seen that:

- addition of corn starch or dextrin slightly improves concentration performance for P_2O_5 with respect to Na silicate alone. However, addition of starch or dextrin appears to be more effective as an iron depressant, this is sustained by data shown in the following table:

Depressants	Flotation test n°	Concentrate	P_2O_5		Fe_2O_3	Al_2O_3	MgO
			assay %	recovery %	assay %	assay %	assay %
Na Silicate	19	F3	31.50	55.04	1.60	0.62	0.53
		F3 + F4	28.35	69.93	2.87	1.33	0.83
Na Silicate + Corn starch	20	F3	33.30	42.48	0.92	0.38	0.50
		F3 + F4	32.37	60.76	1.09	0.48	0.52
Na Silicate + dextrin	21	F3	32.00	57.06	1.15	0.98	0.42
		F3 + F4	29.92	70.89	1.84	1.17	0.84
Na Silicate	24	F3	32.90	60.90	1.35	0.46	0.54
		F3 + F4	29.16	70.30	2.93	1.11	0.81

Flotation test n°	Flowsheet	Depressant	P.E. collector	Weight %	P_2O_5	
					Content %	Recovery %
9	1a	Na Silicate	P301			
F2				8.15	3.00	7.03
NF2				75.80	0.28	6.10
F3				9.70	26.80	74.71
NF3				6.35	6.66	12.16
calculated head				100.00	3.48	100.00
7						
F2	2a	Na Silicate + corn starch	P301	9.38	7.15	18.80
NF2				78.10	0.33	7.22
F3				8.06	29.15	65.85
NF3				4.46	6.50	8.13
calculated hed				100.00	3.57	100.00
8	3a	Na Silicate + dextrin	P301			
F2				7.46	6.00	13.18
NF2				78.36	0.30	6.92
F3				8.21	28.80	69.62
NF3				5.97	5.85	10.28
calculated head				100.00	3.40	100.00
10	4a	Corn starch + Na Silicate	P301			
F2				8.22	9.65	22.37
NF2				81.95	0.52	12.02
F3				7.12	29.60	59.42
NF3				2.71	8.10	6.19
calculated head				100.00	3.55	100.00
16	1b	Na Silicate	P312			
NF2				54.29	0.25	3.79
F3				23.72	13.18	87.34
NF3				10.65	1.32	3.93
F4				7.67	2.10	4.50
NF4				3.67	0.43	0.44
calculated head				100.00	3.58	100.00

Table n° 7: Material balances of preliminary flotation tests on REO ore sample 1, using phosphoric esters as collectors

Flotation test n°	Flowsheet	Depressant	P.E. collector	Weight %	P_2O_5	
					Content %	Recovery %
17	4b	Corn starch	P312			
NF2		+		68.31	0.34	5.91
F3		Na Silicate		16.42	18.84	86.27
NF3				7.32	1.75	3.57
F4				4.45	2.91	3.62
NF4				3.50	0.65	0.63
calculated head				100.00	3.58	100.00
19	1b	Na Silicate	B110			
NF2				80.64	0.47	11.56
F3				5.73	31.50	55.05
NF3				5.97	7.95	14.47
F4				2.36	20.70	14.88
NF4				5.30	2.50	4.04
calculted head				100.00	3.28	100.00
20	2b	Na Silicate	B110			
NF2		+		88.20	1.15	29.33
F3		corn starch		4.40	33.30	42.37
NF3				2.80	7.60	6.15
F4				2.10	30.40	18.46
NF4				2.50	5.10	3.69
calculated head				100.00	3.46	100.00
21	3b	Na Silicate	B110			
NF2		+		85.48	0.74	17.87
F3		dextrin		6.30	32.00	56.94
NF3				3.88	8.95	9.86
F4				2.07	23.60	13.99
NF4				2.27	2.15	1.34
calculated head				100.00	3.54	100.00
18	4b	Corn starch	B110			
NF2		+		87.04	1.02	25.57
F3		Na silicate		3.28	33.50	31.60
NF3				4.12	13.15	15.58
F4				2.18	32.30	20.25
NF4				3.38	7.15	7.00
calculated head				100.00	3.47	100.00

Table n° 7: Material balances of preliminary flotation tests on REO ore sample 1, using phosphoric esters as collectors

Flotation test n°	Flowsheet	Depressant	P.E. collector	Weight %	P_2O_5	
					Content %	Recovery %
24	1b	Na Silicate	88094			
NF2				82.23	0.18	4.48
F3				6.12	32.90	60.90
NF3				5.08	13.40	20.57
F4				1.85	16.80	9.42
NF4				4.72	3.25	4.63
calculated head				100.00	3.31	100.00
22	2b	Na Silicate + Corn starch	88094			
NF2				87.05	0.44	12.23
F3				3.82	33.45	40.80
NF3				3.34	14.85	15.84
F4				2.33	32.20	23.95
NF4				3.46	6.50	7.18
calculated head				100.00	3.12	100.00
23	3b	Na Silicate + dextrin	88094			
NF2				82.91	0.27	6.95
F3				5.33	33.35	55.20
NF3				4.54	9.48	13.37
F4				2.07	28.85	18.55
NF4				5.15	3.71	5.93
calculated head				100.00	3.22	100.00

Table 7: Material balances of preliminary flotation tests on REO ore sample 1, using phosphoric esters as collectors

TABLE N° 1F10 : RED ORE SAMPLE N°1 GROUND DOWN TO 0.16mm,MATERIAL BALANCE OF FLOTATION TEST N°10 ON -0.16+0.010mm(t) FRACTION.

PRODUCTS	WEIGHT	P205		CaO		SiO2		Fe203	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF2	81.95	.52	13.89	3.60	37.71	69.00	90.79	5.95	82.98
2 F3	7.12	29.60	68.67	42.80	38.95	10.30	1.18	2.15	2.61
3 NF3	2.71	8.10	7.15	16.10	5.58	47.50	2.07	7.45	3.44
4 NF4	3.45	2.40	2.70	7.50	3.31	60.40	3.35	8.15	4.79
5 F5	1.39	1.20	.54	17.90	3.18	43.80	.98	7.20	1.70
6 NF5	3.38	6.40	7.05	26.10	11.28	30.30	1.64	7.80	4.49
CALCULATED HEAD	100.00	3.07	100.00	7.82	100.00	62.28	100.00	5.88	100.00

PRODUCTS	WEIGHT	A1203		MgO		L.O.I.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF2	81.95	9.50	91.29	1.65	82.71	.65	56.78
2 F3	7.12	1.05	.88	.68	2.96	2.80	21.25
3 NF3	2.71	6.55	2.08	2.35	3.90	2.70	7.80
4 NF4	3.45	8.30	3.36	2.40	5.06	1.55	5.70
5 F5	1.39	5.70	.93	1.70	1.45	.97	1.44
6 NF5	3.38	3.70	1.47	1.90	3.93	1.95	7.03
CALCULATED HEAD	100.00	8.53	100.00	1.63	100.00	.94	100.00

PRODUCTS	WEIGHT %	CaO GRADE / P205 GRADE
1 NF2	81.95	6.9231
2 F3	7.12	1.4459
3 NF3	2.71	1.9877
4 NF4	3.45	3.1250
5 F5	1.39	14.9167
6 NF5	3.38	4.0781
CALCULATED HEAD	100.00	2.5493

TABLE N° 1F10 : FLOTATION TEST N° 10.

PRODUCTS	WEIGHT	P2O5		CaO		SiO2		Fe2O3	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F4 5 6 0 0 0 0	4.77	4.88	7.59	23.71	14.46	34.23	2.62	7.63	6.19
2 NF4 4 0 0 0 0 0	3.45	2.40	2.70	7.50	3.31	60.40	3.35	8.15	4.79
3 F2 4 5 6 0 0 0	8.22	3.84	10.29	16.91	17.76	45.22	5.97	7.85	10.98
4 NF2 1 0 0 0 0 0	81.95	.52	13.89	3.60	37.71	69.00	90.79	5.95	82.98
5 F1 2 3 0 0 0 0	9.83	23.67	75.82	35.44	44.53	20.56	3.24	3.61	6.04
6 NF1 1 4 5 6 0 0	90.17	.82	24.18	4.81	55.47	66.83	96.76	6.12	93.96
7 F3 2 0 0 0 0 0	7.12	29.60	68.67	42.80	38.95	10.30	1.18	2.15	2.61
8 NF3+NF5 3 5 0 0 0 0	4.10	5.76	7.70	16.71	8.76	46.25	3.04	7.37	5.14
9 NF2+NF4+F5 1 4 5 0 0 0	86.79	.61	17.13	3.98	44.20	68.25	95.11	6.06	89.47

PRODUCTS	WEIGHT	Al2O3		MgO		L.O.I.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F4 5 6 0 0 0 0	4.77	4.28	2.40	1.84	5.37	1.66	8.46
2 NF4 4 0 0 0 0 0	3.45	8.30	3.36	2.40	5.06	1.55	5.70
3 F2 4 5 6 0 0 0	8.22	5.97	5.75	2.08	10.44	1.62	14.16
4 NF2 1 0 0 0 0 0	81.95	9.50	91.29	1.65	82.71	.65	56.78
5 F1 2 3 0 0 0 0	9.83	2.57	2.96	1.14	6.86	2.77	29.05
6 NF1 1 4 5 6 0 0	90.17	9.18	97.04	1.69	93.14	.74	70.95
7 F3 2 0 0 0 0 0	7.12	1.05	.88	.68	2.96	2.80	21.25
8 NF3+NF5 3 5 0 0 0 0	4.10	6.26	3.01	2.13	5.34	2.11	9.24
9 NF2+NF4+F5 1 4 5 0 0 0	86.79	9.39	95.58	1.68	89.22	.69	63.92

TABLE N° 1F17 : RED ORE SAMPLE N°1 GROUND DOWN TO 0.16mm,MATERIAL BALANCE OF FLOTATION TEST N°17 ON -0.16+0.010mm(t) FRACTION.

PRODUCTS	WEIGHT	P205		CaO		SiO2		Fe203	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF2	68.32	.34	6.44	2.95	25.62	72.20	77.93	5.55	63.15
2 NF3	7.32	1.75	3.55	5.95	5.54	63.00	7.29	7.45	9.08
3 F4	6.42	18.60	33.12	27.20	22.19	32.40	3.29	5.20	5.56
4 NF4	9.99	19.00	52.65	31.70	40.25	24.00	3.79	6.30	10.48
5 NF5	3.50	.65	.63	3.70	1.65	66.00	3.65	6.70	3.91
6 F6	2.30	1.80	1.15	5.90	1.72	63.10	2.29	8.15	3.12
7 NF6	2.15	4.10	2.45	11.10	3.03	52.00	1.77	13.10	4.69
CALCULATED HEAD	100.00	3.60	100.00	7.87	100.00	63.30	100.00	6.00	100.00

PRODUCTS	WEIGHT	A1203		MgO		L.O.I.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF2	68.32	9.85	78.39	1.60	64.16	.51	36.51
2 NF3	7.32	8.70	7.42	2.35	10.10	1.50	11.50
3 F4	6.42	4.05	3.03	1.30	4.90	.77	5.18
4 NF4	9.99	2.85	3.32	1.55	9.09	3.70	38.73
5 NF5	3.50	9.25	3.77	2.25	4.62	1.10	4.03
6 F6	2.30	8.85	2.37	2.15	2.90	.56	1.35
7 NF6	2.15	6.80	1.70	3.35	4.23	1.20	2.70
CALCULATED HEAD	100.00	8.58	100.00	1.70	100.00	.95	100.00

PRODUCTS	WEIGHT %	CaO GRADE / P205 GRADE
1 NF2	68.32	8.6765
2 NF3	7.32	3.4000
3 F4	6.42	1.4624
4 NF4	9.99	1.6684
5 NF5	3.50	5.6923
6 F6	2.30	3.2778
7 NF6	2.15	2.7073
CALCULATED HEAD	100.00	2.1826

TABLE N° 1F17 : FLOTATION TEST N° 17.

PRODUCTS	WEIGHT	P2O5		CaO		SiO2		Fe2O3	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F3 4 5 0 0 0 0	13.49	14.24	53.28	24.44	41.90	34.90	7.44	6.40	14.39
2 NF3 2 0 0 0 0 0	7.32	1.75	3.55	5.95	5.54	63.00	7.29	7.45	9.08
3 F2 5 6 7 0 0 0	7.95	1.92	4.22	6.34	6.40	61.37	7.71	8.85	11.72
4 NF2 1 0 0 0 0 0	68.32	.34	6.44	2.95	25.62	72.20	77.93	5.55	63.15
5 F1 2 3 4 0 0 0	23.73	13.57	89.33	22.54	67.98	38.30	14.36	6.36	25.13
6 NF1 1 5 6 7 0 0	76.27	.50	10.67	3.30	32.02	71.07	85.64	5.89	74.87
7 F5 6 7 0 0 0 0	4.45	2.91	3.59	8.41	4.76	57.74	4.06	10.54	7.81
8 NF5 5 0 0 0 0 0	3.50	.65	.63	3.70	1.65	66.00	3.65	6.70	3.91

PRODUCTS	WEIGHT	Al2O3		MgO		L.O.I.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F3 4 5 0 0 0 0	13.49	4.51	7.09	1.73	13.71	3.03	42.76
2 NF3 2 0 0 0 0 0	7.32	8.70	7.42	2.35	10.10	1.50	11.50
3 F2 5 6 7 0 0 0	7.95	8.47	7.85	2.52	11.75	.97	8.09
4 NF2 1 0 0 0 0 0	68.32	9.85	78.39	1.60	64.16	.51	36.51
5 F1 2 3 4 0 0 0	23.73	4.98	13.76	1.73	24.08	2.23	55.41
6 NF1 1 5 6 7 0 0	76.27	9.71	86.24	1.70	75.92	.56	44.59
7 F5 6 7 0 0 0 0	4.45	7.86	4.07	2.73	7.13	.87	4.05
8 NF5 5 0 0 0 0 0	3.50	9.25	3.77	2.25	4.62	1.10	4.03

TABLE N° 1F19 : REO ORE SAMPLE N°1 GROUND DOWN TO 0.16mm,MATERIAL BALANCE OF FLOTATION TEST N°19 ON -0.16+0.010mm(t) FRACTION.

PRODUCTS	WEIGHT	P205		CaO		SiO2		Fe203	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF2	80.64	.47	11.56	3.45	36.66	69.80	88.85	6.25	81.08
2 F3	5.73	31.50	55.04	45.80	34.58	7.35	.66	1.60	1.47
3 NF3	5.97	7.95	14.47	16.30	12.82	47.70	4.50	7.95	7.64
4 F4	2.36	20.70	14.90	33.30	10.35	22.90	.85	5.95	2.26
5 NF4	5.30	2.50	4.04	8.00	5.59	61.40	5.14	8.85	7.55
CALCULATED HEAD	100.00	3.28	100.00	7.59	100.00	63.35	100.00	6.22	100.00

PRODUCTS	WEIGHT	A1203		MgO		L.O.I.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF2	80.64	10.90	90.43	1.70	80.31	.73	54.69
2 F3	5.73	.62	.37	.53	1.78	3.15	16.77
3 NF3	5.97	6.55	4.02	2.20	7.69	2.55	14.14
4 F4	2.36	3.05	.74	1.55	2.14	3.20	7.02
5 NF4	5.30	8.15	4.44	2.60	8.07	1.50	7.39
CALCULATED HEAD	100.00	9.72	100.00	1.71	100.00	1.08	100.00

PRODUCTS	WEIGHT %	CaO GRADE / P205 GRADE
1 NF2	80.64	7.3404
2 F3	5.73	1.4540
3 NF3	5.97	2.0503
4 F4	2.36	1.6087
5 NF4	5.30	3.2000
CALCULATED HEAD	100.00	2.3141

TABLE N° 1F19 : FLOTATION TEST N° 19.

PRODUCTS	WEIGHT	P205		CaO		SiO2		Fe203	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F3 2 0 0 0 0 0	5.73	31.50	55.04	45.80	34.58	7.35	.66	1.60	1.47
2 TAILS 3 1 3 4 5 0 0	94.27	1.56	44.96	5.27	65.42	66.75	99.34	6.50	98.53
3 F3+F4 2 4 0 0 0 0	8.09	28.35	69.93	42.15	44.93	11.89	1.52	2.87	3.73
4 TAILS 2 1 3 5 0 0 0	91.91	1.07	30.07	4.55	55.07	67.88	98.48	6.51	96.27
5 F3+F4+NF3 4 2 3 0 0 0	14.06	19.69	84.40	31.18	57.76	27.09	6.01	5.03	11.37
6 TAILS 1 1 5 0 0 0 0	85.94	.60	15.60	3.73	42.24	69.28	93.99	6.41	88.63
7 NF3+NF4 3 5 0 0 0 0	11.27	5.39	18.51	12.40	18.41	54.14	9.63	8.37	15.18
8 F1 2 3 0 0 0 0	11.70	19.48	69.51	30.75	47.40	27.94	5.16	4.84	9.11
9 NF1 1 4 5 0 0 0	88.30	1.13	30.49	4.52	52.60	68.04	94.84	6.40	90.89
10 F2 4 5 0 0 0 0	7.66	8.11	18.94	15.79	15.94	49.54	5.99	7.96	9.81
11 NF2 1 0 0 0 0 0	80.64	.47	11.56	3.45	36.66	69.80	88.85	6.25	81.08
12 F1+F2 2 3 4 5 0 0	19.36	14.98	88.44	24.83	63.34	36.48	11.15	6.07	18.92

PRODUCTS	WEIGHT	A1203		MgO		L.O.I.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F3 2 0 0 0 0 0	5.73	.62	.37	.53	1.78	3.15	16.77
2 TAILS 3 1 3 4 5 0 0	94.27	10.27	99.63	1.78	98.22	.95	83.23
3 F3+F4 2 4 0 0 0 0	8.09	1.33	1.11	.83	3.92	3.16	23.78
4 TAILS 2 1 3 5 0 0 0	91.91	10.46	98.89	1.78	96.08	.89	76.22
5 F3+F4+NF3 4 2 3 0 0 0	14.06	3.55	5.13	1.41	11.62	2.90	37.93
6 TAILS 1 1 5 0 0 0 0	85.94	10.73	94.87	1.76	88.38	.78	62.07
7 NF3+NF4 3 5 0 0 0 0	11.27	7.30	8.47	2.39	15.77	2.06	21.53
8 F1 2 3 0 0 0 0	11.70	3.65	4.39	1.38	9.47	2.84	30.91
9 NF1 1 4 5 0 0 0	88.30	10.53	95.61	1.75	90.53	.84	69.09
10 F2 4 5 0 0 0 0	7.66	6.58	5.18	2.28	10.22	2.02	14.40
11 NF2 1 0 0 0 0 0	80.64	10.90	90.43	1.70	80.31	.73	54.69
12 F1+F2 2 3 4 5 0 0	19.36	4.81	9.57	1.74	19.69	2.52	45.31

TABLE N° 1F20 A: RED ORE SAMPLE N°1 GROUND DOWN TO 0.16mm,MATERIAL BALANCE OF FLOTATION TEST N°20 ON -0.16+0.010mm(t) FRACTION.

PRODUCTS	WEIGHT	P205		CaO		SiO2		Fe203	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF2	88.17	1.15	29.33	4.45	51.03	68.60	95.04	6.05	92.31
2 F3	4.41	33.30	42.48	46.60	26.73	5.40	.37	.92	.70
3 NF3	2.81	7.60	6.18	16.20	5.92	49.80	2.20	6.90	3.36
4 F4	2.08	30.40	18.29	44.90	12.15	8.25	.27	1.45	.52
5 NF4	2.53	5.10	3.73	12.70	4.18	53.20	2.11	7.10	3.11
CALCULATED HEAD	100.00	3.46	100.00	7.69	100.00	63.64	100.00	5.78	100.00

PRODUCTS	WEIGHT	A1203		MgO		L.O.I.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF2	88.17	9.00	95.34	1.65	90.20	.76	64.37
2 F3	4.41	.38	.20	.50	1.37	2.25	9.53
3 NF3	2.81	6.55	2.21	2.35	4.09	3.85	10.39
4 F4	2.08	.68	.17	.57	.74	3.85	7.69
5 NF4	2.53	6.85	2.08	2.30	3.61	3.30	8.02
CALCULATED HEAD	100.00	8.32	100.00	1.61	100.00	1.04	100.00

PRODUCTS	WEIGHT %	CaO GRADE / P205 GRADE
1 NF2	88.17	3.8696
2 F3	4.41	1.3994
3 NF3	2.81	2.1316
4 F4	2.08	1.4770
5 NF4	2.53	2.4902
CALCULATED HEAD	100.00	2.2240

TABLE N° 1F20 A : FLOTATION TEST N° 20.

PRODUCTS	WEIGHT	P205		CaO		SiO2		Fe203	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F3 2 0 0 0 0 0	4.41	33.30	42.48	46.60	26.73	5.40	.37	.92	.70
2 TAILS 3 1 3 4 5 0 0	95.59	2.08	57.52	5.89	73.27	66.33	99.63	6.00	99.30
3 F3+F4 2 4 0 0 0 0	6.49	32.37	60.76	46.06	38.87	6.31	.64	1.09	1.22
4 TAILS 2 1 3 5 0 0 0	93.51	1.45	39.24	5.03	61.13	67.62	99.36	6.10	98.78
5 F3+F4+NF3 2 4 3 0 0 0	9.30	24.89	66.94	37.03	44.79	19.45	2.84	2.85	4.58
6 TAILS 1 1 5 0 0 0 0	90.70	1.26	33.06	4.68	55.21	68.17	97.16	6.08	95.42
7 NF3+NF4 3 5 0 0 0 0	5.34	6.42	9.91	14.54	10.10	51.41	4.31	6.99	6.46
8 F1 2 3 0 0 0 0	7.22	23.30	48.65	34.77	32.65	22.68	2.57	3.25	4.06
9 NF1 1 4 5 0 0 0	92.78	1.91	51.35	5.58	67.35	66.83	97.43	5.98	95.94
10 F2 4 5 0 0 0 0	4.61	16.52	22.02	27.23	16.32	32.92	2.38	4.55	3.63
11 NF2 1 0 0 0 0 0	88.17	1.15	29.33	4.45	51.03	68.60	95.04	6.05	92.31
12 F1+F2 2 3 4 5 0 0	11.83	20.65	70.67	31.83	48.97	26.67	4.96	3.76	7.69

TABLE N°1F20A: FLOTATION TEST N° 20

PRODUCTS	WEIGHT	Al2O3		MgO		L.O.I.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F3 2 0 0 0 0 0	4.41	.38	.20	.50	1.37	2.25	9.53
2 TAILS 3 1 3 4 5 0 0	95.59	8.69	99.80	1.66	98.63	.99	90.47
3 F3+F4 2 4 0 0 0 0	6.49	.48	.37	.52	2.10	2.76	17.22
4 TAILS 2 1 3 5 0 0 0	93.51	8.87	99.63	1.69	97.90	.92	82.78
5 F3+F4+NF3 2 4 3 0 0 0	9.30	2.31	2.58	1.07	6.20	3.09	27.61
6 TAILS 1 1 5 0 0 0 0	90.70	8.94	97.42	1.67	93.80	.83	72.39
7 NF3+NF4 3 5 0 0 0 0	5.34	6.69	4.29	2.33	7.70	3.59	18.41
8 F1 2 3 0 0 0 0	7.22	2.78	2.41	1.22	5.46	2.87	19.92
9 NF1 1 4 5 0 0 0	92.78	8.75	97.59	1.64	94.54	.90	80.08
10 F2 4 5 0 0 0 0	4.61	4.07	2.25	1.52	4.34	3.55	15.71
11 NF2 1 0 0 0 0 0	88.17	9.00	95.34	1.65	90.20	.76	64.37
12 F1+F2 2 3 4 5 0 0	11.83	3.28	4.66	1.34	9.80	3.14	35.63

TABLE N° 1F20 B: RED ORE SAMPLE N°1 GROUND DOWN TO 0.16mm,MATERIAL BALANCE OF FLOTATION TEST N°20 ON -0.16+0.010mm(t) FRACTION.

PRODUCTS	WEIGHT	La		Ce		Nd		Sm	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 NF2	88.17	1302.00	63.40	1960.00	58.58	640.00	50.24	112.00	44.48
2 F3	4.41	7600.00	18.51	13680.00	20.45	6615.00	25.97	1520.00	30.19
3 NF3	2.81	3116.00	4.84	5960.00	5.68	2380.00	5.95	480.00	6.08
4 F4	2.08	7640.00	8.78	15620.00	11.01	7200.00	13.33	1520.00	14.24
5 NF4	2.53	3200.00	4.47	4980.00	4.27	2000.00	4.50	440.00	5.01
CALCULATED HEAD	100.00	1810.56	100.00	2949.79	100.00	1123.25	100.00	222.02	100.00

PRODUCTS	WEIGHT	Eu		Gd		Dy		Er	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 NF2	88.17	9.20	40.93	97.00	40.52	72.00	36.75	42.90	36.65
2 F3	4.41	152.80	34.00	1612.00	33.68	1432.00	36.55	850.00	36.32
3 NF3	2.81	39.70	5.63	412.00	5.48	352.00	5.73	208.00	5.66
4 F4	2.08	144.40	15.15	1600.00	15.77	1360.00	16.37	848.00	17.09
5 NF4	2.53	33.60	4.29	380.00	4.55	314.00	4.60	174.00	4.27
CALCULATED HEAD	100.00	19.82	100.00	211.09	100.00	172.76	100.00	103.20	100.00

TABLE N° 1F20 B : FLOTATION TEST N° 20.

PRODUCTS	WEIGHT	La		Ce		Nd		Sm	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 F3 2 0 0 0 0 0	4.41	7600.00	18.51	13680.00	20.45	6615.00	25.97	1520.00	30.19
2 TAILS 3 1 3 4 5 0 0	95.59	1543.47	81.49	2454.75	79.55	869.89	74.03	162.14	69.81
3 F3+F4 2 4 0 0 0 0	6.49	7612.82	27.29	14301.76	31.47	6802.49	39.30	1520.00	44.43
4 TAILS 2 1 3 5 0 0 0	93.51	1407.86	72.71	2161.91	68.53	729.08	60.70	131.93	55.57
5 F3+F4+NF3 2 4 3 0 0 0	9.30	6254.10	32.12	11781.29	37.14	5466.23	45.26	1205.76	50.51
6 TAILS 1 1 5 0 0 0 0	90.70	1354.94	67.88	2044.24	62.86	677.94	54.74	121.15	49.49
7 NF3+NF4 3 5 0 0 0 0	5.34	3155.80	9.31	5495.69	9.95	2199.96	10.46	461.05	11.09
8 F1 2 3 0 0 0 0	7.22	5854.84	23.35	10675.40	26.13	4966.75	31.93	1115.24	36.27
9 NF1 1 4 5 0 0 0	92.78	1495.85	76.65	2348.59	73.87	824.15	68.07	152.51	63.73
10 F2 4 5 0 0 0 0	4.61	5203.30	13.25	9780.70	15.29	4346.20	17.84	927.29	19.25
11 NF2 1 0 0 0 0 0	88.17	1302.00	63.40	1960.00	58.58	640.00	50.24	112.00	44.48
12 F1+F2 2 3 4 5 0 0	11.83	5600.94	36.60	10326.75	41.42	4724.93	49.76	1041.99	55.52

PRODUCTS	WEIGHT	Eu		Gd		Dy		Er	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 F3 2 0 0 0 0 0	4.41	152.80	34.00	1612.00	33.68	1432.00	36.55	850.00	36.32
2 TAILS 3 1 3 4 5 0 0	95.59	13.68	66.00	146.45	66.32	114.66	63.45	68.74	63.68
3 F3+F4 2 4 0 0 0 0	6.49	150.11	49.15	1608.15	49.44	1408.92	52.93	849.36	53.42
4 TAILS 2 1 3 5 0 0 0	93.51	10.78	50.85	114.12	50.56	86.96	47.07	51.41	46.58
5 F3+F4+NF3 2 4 3 0 0 0	9.30	116.75	54.78	1246.74	54.93	1089.57	58.65	655.57	59.08
6 TAILS 1 1 5 0 0 0 0	90.70	9.88	45.22	104.89	45.07	78.75	41.35	46.56	40.92
7 NF3+NF4 3 5 0 0 0 0	5.34	36.81	9.92	396.84	10.04	334.00	10.32	191.89	9.93
8 F1 2 3 0 0 0 0	7.22	108.78	39.63	1144.96	39.16	1011.67	42.28	600.14	41.99
9 NF1 1 4 5 0 0 0	92.78	12.90	60.37	138.41	60.84	107.47	57.72	64.52	58.01
10 F2 4 5 0 0 0 0	4.61	83.59	19.44	930.46	20.32	785.95	20.97	478.10	21.36
11 NF2 1 0 0 0 0 0	88.17	9.20	40.93	97.00	40.52	72.00	36.75	42.90	36.65
12 F1+F2 2 3 4 5 0 0	11.83	98.97	59.07	1061.37	59.48	923.71	63.25	552.58	63.35

TABLE N° 1F20 C: RED ORE SAMPLE N°1 GROUND DOWN TO 0.16mm,MATERIAL BALANCE OF FLOTATION TEST N°20 ON -0.16+0.010mm(t) FRACTION.

PRODUCTS	WEIGHT	Yb		Y	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 NF2	88.17	41.50	37.89	452.00	39.08
2 F3	4.41	784.00	35.80	8040.00	34.77
3 NF3	2.81	198.00	5.76	2080.00	5.73
4 F4	2.08	772.00	16.63	7880.00	16.07
5 NF4	2.53	150.00	3.93	1756.00	4.36
CALCULATED HEAD	100.00	96.58	100.00	1019.87	100.00

TABLE N° 1F20 C : FLOTATION TEST N° 20.

PRODUCTS	WEIGHT	Yb		Y	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 F3 2 0 0 0 0 0	4.41	784.00	35.80	8040.00	34.77
2 TAILS 3 1 3 4 5 0 0	95.59	64.87	64.20	696.00	65.23
3 F3+F4 2 4 0 0 0 0	6.49	780.15	52.42	7988.72	50.84
4 TAILS 2 1 3 5 0 0 0	93.51	49.14	47.58	536.20	49.16
5 F3+F4+NF3 2 4 3 0 0 0	9.30	604.26	58.18	6203.40	56.57
6 TAILS 1 1 5 0 0 0 0	90.70	44.53	41.82	488.37	43.43
7 NF3+NF4 3 5 0 0 0 0	5.34	175.26	9.69	1926.49	10.09
8 F1 2 3 0 0 0 0	7.22	555.93	41.56	5720.39	40.50
9 NF1 1 4 5 0 0 0	92.78	60.84	58.44	654.08	59.50
10 F2 4 5 0 0 0 0	4.61	430.64	20.56	4519.11	20.43
11 NF2 1 0 0 0 0 0	88.17	41.50	37.89	452.00	39.08
12 F1+F2 2 3 4 5 0 0	11.83	507.11	62.11	5252.26	60.92

TABLE N° 1F21 : RED ORE SAMPLE N°1 GROUND DOWN TO 0.16mm,MATERIAL BALANCE OF FLOTATION TEST N°21 ON -0.16+0.010mm(t) FRACTION.

PRODUCTS	WEIGHT	P205		CaO		Si02		Fe203	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF2	85.48	.74	17.90	4.00	42.92	73.20	94.31	6.30	89.31
2 F3	6.30	32.00	57.06	45.70	36.14	6.25	.59	1.15	1.20
3 NF3	3.88	8.95	9.83	18.30	8.91	44.60	2.61	7.25	4.67
4 F4	2.07	23.60	13.83	37.40	9.72	15.90	.50	3.95	1.36
5 NF4	2.27	2.15	1.38	8.10	2.31	58.10	1.99	9.20	3.46
CALCULATED HEAD	100.00	3.53	100.00	7.97	100.00	66.34	100.00	6.03	100.00

PRODUCTS	WEIGHT	A1203		MgO		L.O.I.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF2	85.48	10.50	94.65	1.95	88.05	.65	55.18
2 F3	6.30	.98	.65	.42	1.40	2.70	16.89
3 NF3	3.88	5.85	2.39	2.30	4.71	3.50	13.49
4 F4	2.07	1.75	.38	2.10	2.30	4.50	9.25
5 NF4	2.27	8.05	1.93	2.95	3.54	2.30	5.19
CALCULATED HEAD	100.00	9.48	100.00	1.89	100.00	1.01	100.00

PRODUCTS	WEIGHT %	CaO GRADE / P205 GRADE
1 NF2	85.48	5.4054
2 F3	6.30	1.4281
3 NF3	3.88	2.0447
4 F4	2.07	1.5847
5 NF4	2.27	3.7674
CALCULATED HEAD	100.00	2.2548

TABLE N° 1F21 : FLOTATION TEST N° 21.

PRODUCTS	WEIGHT	P205		CaO		SiO2		Fe203	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F3 2 0 0 0 0 0	6.30	32.00	57.06	45.70	36.14	6.25	.59	1.15	1.20
2 TAILS 3 1 3 4 5 0 0	93.70	1.62	42.94	5.43	63.86	70.38	99.41	6.36	98.80
3 F3+F4 2 4 0 0 0 0	8.37	29.92	70.89	43.65	45.86	8.64	1.09	1.84	2.56
4 TAILS 2 1 3 5 0 0 0	91.63	1.12	29.11	4.71	54.14	71.61	98.91	6.41	97.44
5 F3+F4+NF3 2 4 3 0 0 0	12.25	23.28	80.72	35.62	54.77	20.03	3.70	3.56	7.22
6 TAILS 1 1 5 0 0 0 0	87.75	.78	19.28	4.11	45.23	72.81	96.30	6.38	92.78
7 NF3+NF4 3 5 0 0 0 0	6.15	6.44	11.21	14.54	11.22	49.58	4.60	7.97	8.13
8 F1 2 3 0 0 0 0	10.18	23.21	66.89	35.26	45.05	20.87	3.20	3.47	5.87
9 NF1 1 4 5 0 0 0	89.82	1.30	33.11	4.87	54.95	71.50	96.80	6.32	94.13
10 F2 4 5 0 0 0 0	4.34	12.38	15.21	22.07	12.03	37.97	2.48	6.70	4.82
11 NF2 1 0 0 0 0 0	85.48	.74	17.90	4.00	42.92	73.20	94.31	6.30	89.31
12 F1+F2 2 3 4 5 0 0	14.52	19.98	82.10	31.32	57.08	25.98	5.69	4.44	10.69

PRODUCTS	WEIGHT	A1203		MgO		L.O.I.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F3 2 0 0 0 0 0	6.30	.98	.65	.42	1.40	2.70	16.89
2 TAILS 3 1 3 4 5 0 0	93.70	10.05	99.35	1.99	98.60	.89	83.11
3 F3+F4 2 4 0 0 0 0	8.37	1.17	1.03	.84	3.69	3.15	26.15
4 TAILS 2 1 3 5 0 0 0	91.63	10.24	98.97	1.99	96.31	.81	73.85
5 F3+F4+NF3 2 4 3 0 0 0	12.25	2.65	3.43	1.30	8.41	3.26	39.63
6 TAILS 1 1 5 0 0 0 0	87.75	10.44	96.57	1.98	91.59	.69	60.37
7 NF3+NF4 3 5 0 0 0 0	6.15	6.66	4.32	2.54	8.25	3.06	18.67
8 F1 2 3 0 0 0 0	10.18	2.84	3.04	1.14	6.11	3.00	30.38
9 NF1 1 4 5 0 0 0	89.82	10.24	96.96	1.98	93.89	.78	69.62
10 F2 4 5 0 0 0 0	4.34	5.05	2.31	2.54	5.83	3.35	14.44
11 NF2 1 0 0 0 0 0	85.48	10.50	94.65	1.95	88.05	.65	55.18
12 F1+F2 2 3 4 5 0 0	14.52	3.50	5.35	1.56	11.95	3.11	44.82

TABLE N° 1F22 A : RED ORE SAMPLE N°1 GROUND DOWN TO 0.16mm,MATERIAL BALANCE OF FLOTATION TEST N° 22 ON THE -0.16+0.010mm(t) FRACTION.

PRODUCTS	WEIGHT	P2O5		CaO		SiO2		Fe2O3	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF2	87.05	.44	12.23	3.80	44.26	67.30	94.98	6.70	92.92
2 F1+F4	9.49	26.60	80.59	38.65	49.08	14.95	2.30	2.20	3.33
3 NF4	3.46	6.50	7.18	14.40	6.67	48.55	2.72	6.80	3.75
CALCULATED HEAD	100.00	3.13	100.00	7.47	100.00	61.68	100.00	6.28	100.00

PRODUCTS	WEIGHT	Al2O3		MgO		Na2O		K2O	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF2	87.05	9.85	95.78	1.70	92.03	5.00	94.65	.76	93.64
2 F1+F4	9.49	1.65	1.75	.64	3.78	1.30	2.68	.20	2.69
3 NF4	3.46	6.40	2.47	1.95	4.20	3.55	2.67	.75	3.67
CALCULATED HEAD	100.00	8.95	100.00	1.61	100.00	4.60	100.00	.71	100.00

PRODUCTS	WEIGHT %	CaO GRADE / P2O5 GRADE
1 NF2	87.05	8.6364
2 F1+F4	9.49	1.4530
3 NF4	3.46	2.2154
CALCULATED HEAD	100.00	2.3861

TABLE N° 1F22 B : REO ORE SAMPLE N°1 GROUND DOWN TO 0.16mm,MATERIAL BALANCE OF FLOTATION TEST N° 22 ON THE -0.16+0.010mm(t) FRACTION.

PRODUCTS	WEIGHT	TiO2		MnO		L.O.I.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF2	87.05	.76	91.92	.08	86.58	.56	75.01
2 F1+F4	9.49	.23	3.03	.07	8.26	1.00	14.60
3 NF4	3.46	1.05	5.05	.12	5.16	1.95	10.38
CALCULATED HEAD	100.00	.72	100.00	.08	100.00	.65	100.00

TABLE N° 1F24 A : REO ORE SAMPLE N°1 GROUND DOWN TO 0.16mm,MATERIAL BALANCE OF FLOTATION TEST N°24 ON -0.16+0.010mm(t) FRACTION.

PRODUCTS	WEIGHT	P2O5		CaO		SiO2		Fe2O3	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF2	82.23	.18	4.48	3.25	34.61	69.70	91.31	6.45	83.63
2 F3	6.12	32.90	60.90	46.20	36.61	5.90	.58	1.35	1.30
3 NF3	5.08	13.40	20.59	23.60	15.52	37.90	3.07	6.55	5.25
4 F4	1.85	16.80	9.40	29.30	7.02	24.90	.73	8.15	2.38
5 NF4	4.72	3.25	4.64	10.20	6.23	57.40	4.32	10.00	7.44
CALCULATED HEAD	100.00	3.31	100.00	7.72	100.00	62.77	100.00	6.34	100.00

PRODUCTS	WEIGHT	Al2O3		MgO		L.O.I.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF2	82.23	10.70	92.58	2.00	84.94	.74	56.22
2 F3	6.12	.46	.30	.54	1.71	1.95	11.03
3 NF3	5.08	4.95	2.65	2.10	5.51	3.35	15.72
4 F4	1.85	3.25	.63	1.70	1.62	4.10	7.01
5 NF4	4.72	7.75	3.85	2.55	6.22	2.30	10.03
CALCULATED HEAD	100.00	9.50	100.00	1.94	100.00	1.08	100.00

PRODUCTS	WEIGHT %	CaO GRADE / P2O5 GRADE
1 NF2	82.23	18.0556
2 F3	6.12	1.4043
3 NF3	5.08	1.7612
4 F4	1.85	1.7440
5 NF4	4.72	3.1385
CALCULATED HEAD	100.00	2.3355

TABLE N° 1F24 A : FLOTATION TEST N°24.

PRODUCTS	WEIGHT	P205		CaO		SiO2		Fe203	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F3 2 0 0 0 0 0	6.12	32.90	60.90	46.20	36.61	5.90	.58	1.35	1.30
2 TAILS 3 1 3 4 5 0 0	93.88	1.38	39.10	5.21	63.39	66.48	99.42	6.67	98.70
3 F3+F4 2 4 0 0 0 0	7.97	29.16	70.30	42.28	43.63	10.31	1.31	2.93	3.68
4 TAILS 2 1 3 5 0 0 0	92.03	1.07	29.70	4.73	56.37	67.31	98.69	6.64	96.32
5 F3+F4+NF3 2 4 3 0 0 0	13.05	23.03	90.88	35.01	59.16	21.05	4.38	4.34	8.93
6 TAILS 1 1 5 0 0 0 0	86.95	.35	9.12	3.63	40.84	69.03	95.62	6.64	91.07
7 NF3+NF4 3 5 0 0 0 0	9.80	8.51	25.23	17.15	21.76	47.29	7.38	8.21	12.69
8 F1 2 3 0 0 0 0	11.20	24.06	81.48	35.95	52.14	20.41	3.64	3.71	6.55
9 NF1 1 4 5 0 0 0	88.80	.69	18.52	4.16	47.86	68.11	96.36	6.67	93.45
10 F2 4 5 0 0 0 0	6.57	7.07	14.04	15.58	13.25	48.25	5.05	9.48	9.82
11 NF2 1 0 0 0 0 0	82.23	.18	4.48	3.25	34.61	69.70	91.31	6.45	83.63
12 F1+F2 2 3 4 5 0 0	17.77	17.77	95.52	28.42	65.39	30.71	8.69	5.84	16.37

TABLE N° 1F24A: FLOTATION TEST N°24

PRODUCTS	WEIGHT	Al2O3		MgO		L.O.I.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F3 2 0 0 0 0 0	6.12	.46	.30	.54	1.71	1.95	11.03
2 TAILS 3 1 3 4 5 0 0	93.88	10.09	99.70	2.03	98.29	1.03	88.97
3 F3+F4 2 4 0 0 0 0	7.97	1.11	.93	.81	3.33	2.45	18.03
4 TAILS 2 1 3 5 0 0 0	92.03	10.23	99.07	2.03	96.67	.96	81.97
5 F3+F4+NF3 2 4 3 0 0 0	13.05	2.60	3.57	1.31	8.84	2.80	33.75
6 TAILS 1 1 5 0 0 0 0	86.95	10.54	96.43	2.03	91.16	.82	66.25
7 NF3+NF4 3 5 0 0 0 0	9.80	6.30	6.49	2.32	11.73	2.84	25.75
8 F1 2 3 0 0 0 0	11.20	2.50	2.94	1.25	7.22	2.58	26.75
9 NF1 1 4 5 0 0 0	88.80	10.39	97.06	2.02	92.78	.89	73.25
10 F2 4 5 0 0 0 0	6.57	6.48	4.48	2.31	7.84	2.81	17.04
11 NF2 1 0 0 0 0 0	82.23	10.70	92.58	2.00	84.94	.74	56.22
12 F1+F2 2 3 4 5 0 0	17.77	3.97	7.42	1.64	15.06	2.67	43.78

TABLE N° 1F24 B: REO ORE SAMPLE N°1 GROUND DOWN TO 0.16mm,MATERIAL BALANCE OF FLOTATION TEST N°24 ON -0.16+0.010mm(t) FRACTION.

PRODUCTS	WEIGHT	La		Ce		Nd		Sm	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 NF2	82.23	712.00	37.21	1200.00	34.11	330.00	24.63	50.80	19.71
2 F3	6.12	7114.00	27.67	15350.00	32.47	7576.00	42.08	1655.00	47.79
3 NF3+NF4	9.80	4300.00	26.78	7440.00	25.20	2760.00	24.55	515.00	23.81
4 F4	1.85	7094.00	8.34	12860.00	8.22	5208.00	8.74	996.00	8.69
CALCULATED HEAD	100.00	1573.49	100.00	2893.21	100.00	1101.84	100.00	211.95	100.00

PRODUCTS	WEIGHT	Eu		Gd		Dy		Er	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 NF2	82.23	4.90	20.57	45.60	18.65	39.70	18.23	25.40	23.83
2 F3	6.12	152.00	47.49	1590.00	48.41	1460.00	49.89	625.00	43.64
3 NF3+NF4	9.80	45.70	22.87	485.00	23.64	425.00	23.26	215.00	24.04
4 F4	1.85	96.00	9.07	1010.00	9.30	835.00	8.63	402.00	8.49
CALCULATED HEAD	100.00	19.59	100.00	201.02	100.00	179.09	100.00	87.64	100.00

TABLE N° 1F24 B : FLOTATION TEST N°24.

PRODUCTS	WEIGHT	La		Ce		Nd		Sm	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 F3 2 0 0 0 0 0	6.12	7114.00	27.67	15350.00	32.47	7576.00	42.08	1655.00	47.79
2 TAILS 3 1 3 4 0 0 0	93.88	1212.31	72.33	2081.16	67.53	679.79	57.92	117.88	52.21
3 F3+F4 2 4 0 0 0 0	7.97	7109.36	36.01	14772.02	40.69	7026.34	50.82	1502.03	56.48
4 TAILS 2 1 3 0 0 0 0	92.03	1094.08	63.99	1864.48	59.31	588.76	49.18	100.23	43.52

PRODUCTS	WEIGHT	Eu		Gd		Dy		Er	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 F3 2 0 0 0 0 0	6.12	152.00	47.49	1590.00	48.41	1460.00	49.89	625.00	43.64
2 TAILS 3 1 3 4 0 0 0	93.88	10.95	52.51	110.47	51.59	95.59	50.11	52.61	56.36
3 F3+F4 2 4 0 0 0 0	7.97	139.00	56.56	1455.37	57.70	1314.92	58.52	573.24	52.13
4 TAILS 2 1 3 0 0 0 0	92.03	9.24	43.44	92.39	42.30	80.73	41.48	45.59	47.87

TABLE N° 1F24 C: RED ORE SAMPLE N°1 GROUND DOWN TO 0.16mm,MATERIAL BALANCE OF FLOTATION TEST N°24 ON -0.16+0.010mm(t) FRACTION.

PRODUCTS	WEIGHT	Yb		Y	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 NF2	82.23	25.80	29.67	260.00	23.35
2 F3	6.12	460.00	39.38	7150.00	47.78
3 NF3+NF4	9.80	162.00	22.21	2016.00	21.57
4 F4	1.85	338.00	8.75	3610.00	7.29
CALCULATED HEAD	100.00	71.50	100.00	915.73	100.00

TABLE N° 1F24 C : FLOTATION TEST N° 24.

PRODUCTS	WEIGHT	Yb		Y	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 F3 2 0 0 0 0 0	6.12	460.00	39.38	7150.00	47.78
2 TAILS 3 1 3 4 0 0 0	93.88	46.17	60.62	509.32	52.22
3 F3+F4 2 4 0 0 0 0	7.97	431.68	48.12	6328.29	55.08
4 TAILS 2 1 3 0 0 0 0	92.03	40.30	51.88	446.99	44.92

FIGURE IV-2 ORE SAMPLE 1

PRELIMINARY FLOTATION TESTS WITH P.E.

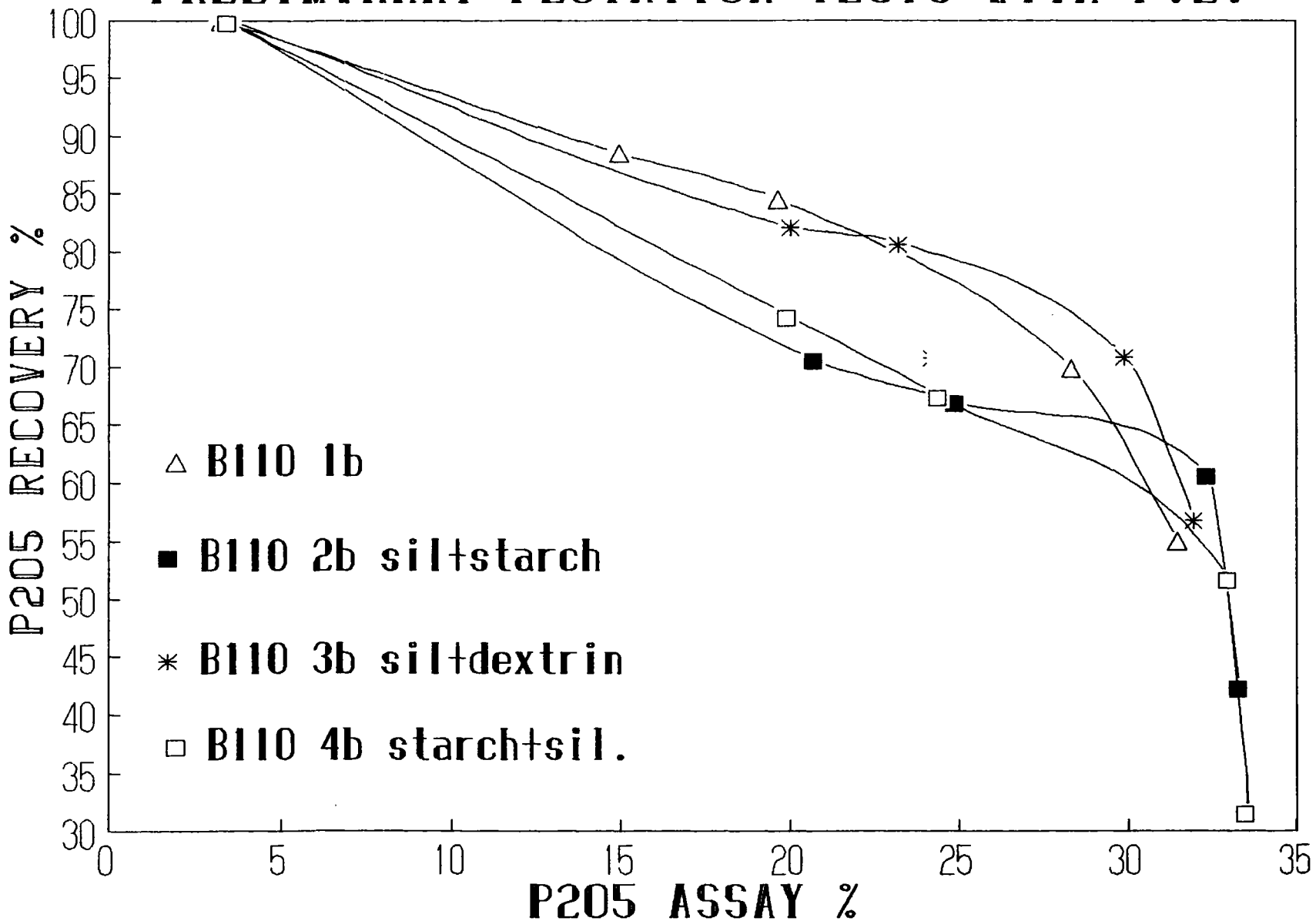
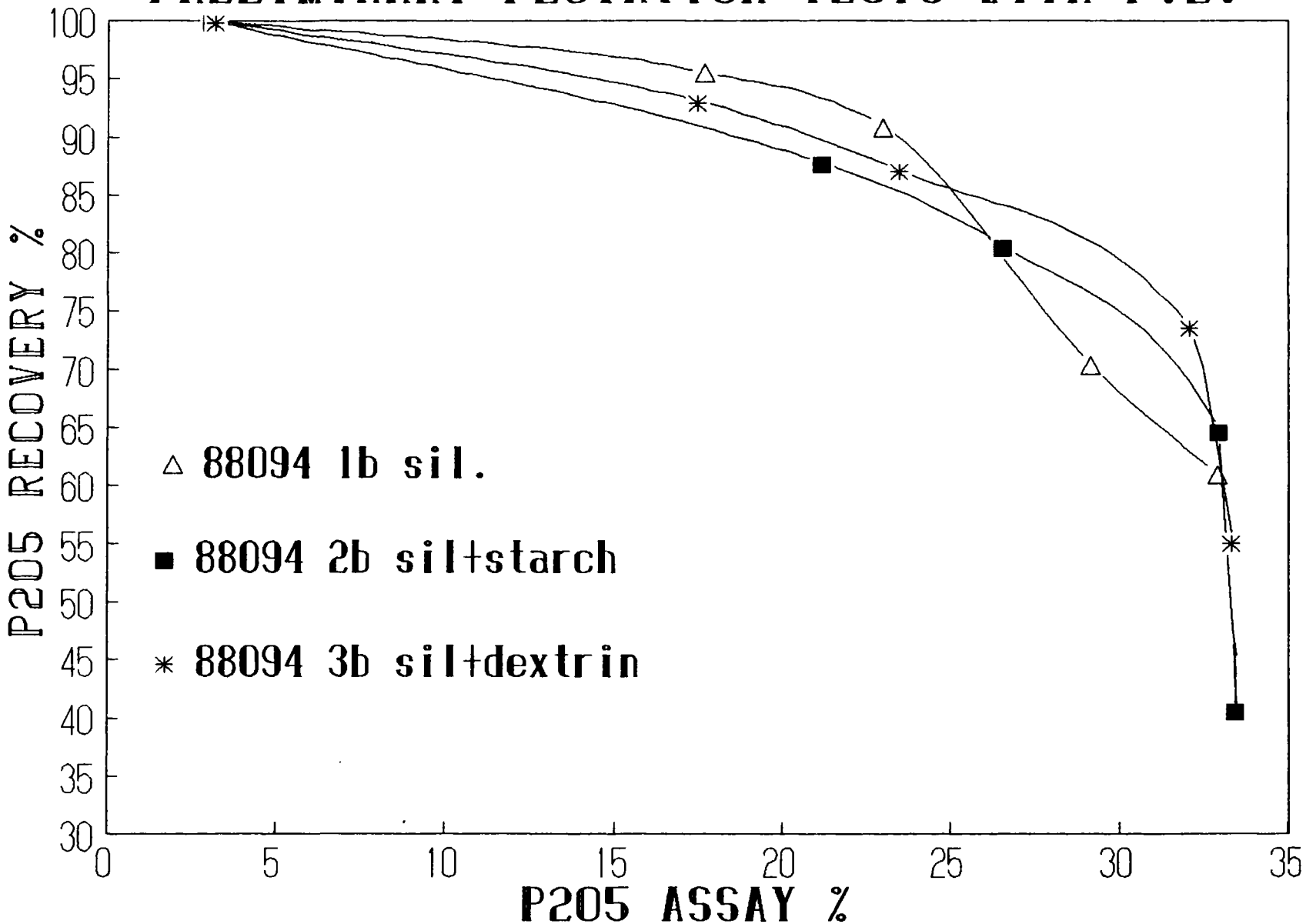


FIGURE N 3: REO ORE SAMPLE 1

PRELIMINARY FLOTATION TESTS WITH P.E.



At comparable P_2O_5 contents and recoveries in concentrates, Fe_2O_3 contents in concentrates separated from feeds conditioned with starch or dextrin are lower than those obtained in concentrates separated from feeds conditioned with Na silicate alone.

- addition of starch after addition of Na silicate appears to provide slightly better separation performance with respect to addition of starch prior to that of Na silicate
- lower R_2O_3 and slightly higher P_2O_5 contents in concentrates are obtained with corn starch as a depressant, instead of dextrin
- phosphoric ester collector 1E 88094 leads to better concentration performance with respect to other P.E. collectors tested: P301, B110 and P312.

Concentrates produced in flotation tests n° 20 and 24 have low R_2O_3 and MgO contents which meet usual specifications for concentrates used in production of wet process phosphoric acid:

	Flotation test	
	n° 20 F3 + F4	n° 24 F3
content:		
P_2O_5 %	32.37	32.90
CaO %	46.06	46.20
SiO_2 %	6.31	5.90
Fe_2O_3 %	1.09	1.35
Al_2O_3 %	0.48	0.46
MgO %	0.52	0.54
L.O.I. %	2.76	1.95
Eu ppm	150	152
Y ppm	7 989	7 150
Total REE + Y ppm	43 023	43 132
P_2O_5 recovery % (on flotation feed basis)	60.76	60.90
CaO/P_2O_5	1.423	1.404
R_2O_3/P_2O_5	0.0485	0.0550
TCP %	70.73	71.89
Y recovery %	50.84	47.78
Apatite content %	90.4	91.9
$(R_2O_3 + MgO)/P_2O_5$	0.0646	0.0714

3.2. PRELIMINARY TESTS ON THE REO ORE SAMPLE N° 2

Four series of tests were effected on fractions separated from the attrition-scrubbed and rod-milled material:

. flotation of the - 160 + 10 μm(t)
. flotation of the - 50 + 10 μm(t)
. flotation of the - 80 + 10 μm(t) gravity preconcentrate
. flotation of the - 40 + 10 μm(t) gravity preconcentrate.

Reagents used were the following:

■ pH regulators and gangue depressants:

- Na_2CO_3, technical grade from Prolabo
- waxi starch snow flake 04201 FROM CERESTAR France S.A., powdered starch was allowed to disperse and hydrate by heating in water at 85°C for 10 minutes, (starch concentration: 16.7 g/l), under agitation
- quebracho, ordinary grade, spray-dried
- Na silicate, technical grade from Prolabo, identifical to that used in flotation tests on REO ore sample 1
- citric acid, technical grade from Prolabo
- oxalic acid, technical grade from Prolabo
- Ca lignosulfonate from l'Avébène
- NaOH, H_2SO_4, H_3PO_4, $NH_4H_2PO_4$ H_2SiF_6, technical grades from Prolabo
- CO_2 was obtained from a liquefied - CO_2 - container (technical grade) from L'Air Liquide.

Waxi starch was used as an iron-carbonates depressant for Fe-Mn containing dolomite and siderite, citric and oxalic acids were added as iron depressants. Ca lignosulfonate, quebracho and CO_2 were evaluated as carbonates depressants. Na carbonate and silicate were used as pH regulators, slimes dispersants and carbonates depressants. H_2SO_4 was used as a pH regulator and a barite depressant

■ collectors:

- phosphoric ester P301, 88094 and EA 14-89 were used as monazite collectors, a detailed characterization is shown in chapter 4

- petroleum sulfonate AP 825, an oil soluble and water dispersible sulfonate from American Cyanamid, was used as a strontianite collector

- fatty acids AP 765 from American Cyanamid and Na Oleate, technical grade from Prolabo, were also evaluated as collectors for strontianite and barite.

3.2.1. Flotation of the - 160 + 10 µm fraction

Operating conditions and flowsheets of flotation tests are shown in figures 2H01 to 2H04.

Material balances are shown in tables 2H01 to 2H04.

In an alkaline pulp (pH: 10.5) results reveal that:

- strontianite as well as barite are floated with phosphoric ester P301
- iron, mostly associated to Fe-Mn containing dolomite and siderite, exhibits a low floatability
- addition of waxi starch and quebracho does not improve selectivity.

Too high a content in carbonates in the feed to flotation strongly impairs selectivity for monazite at high pulp pH value.

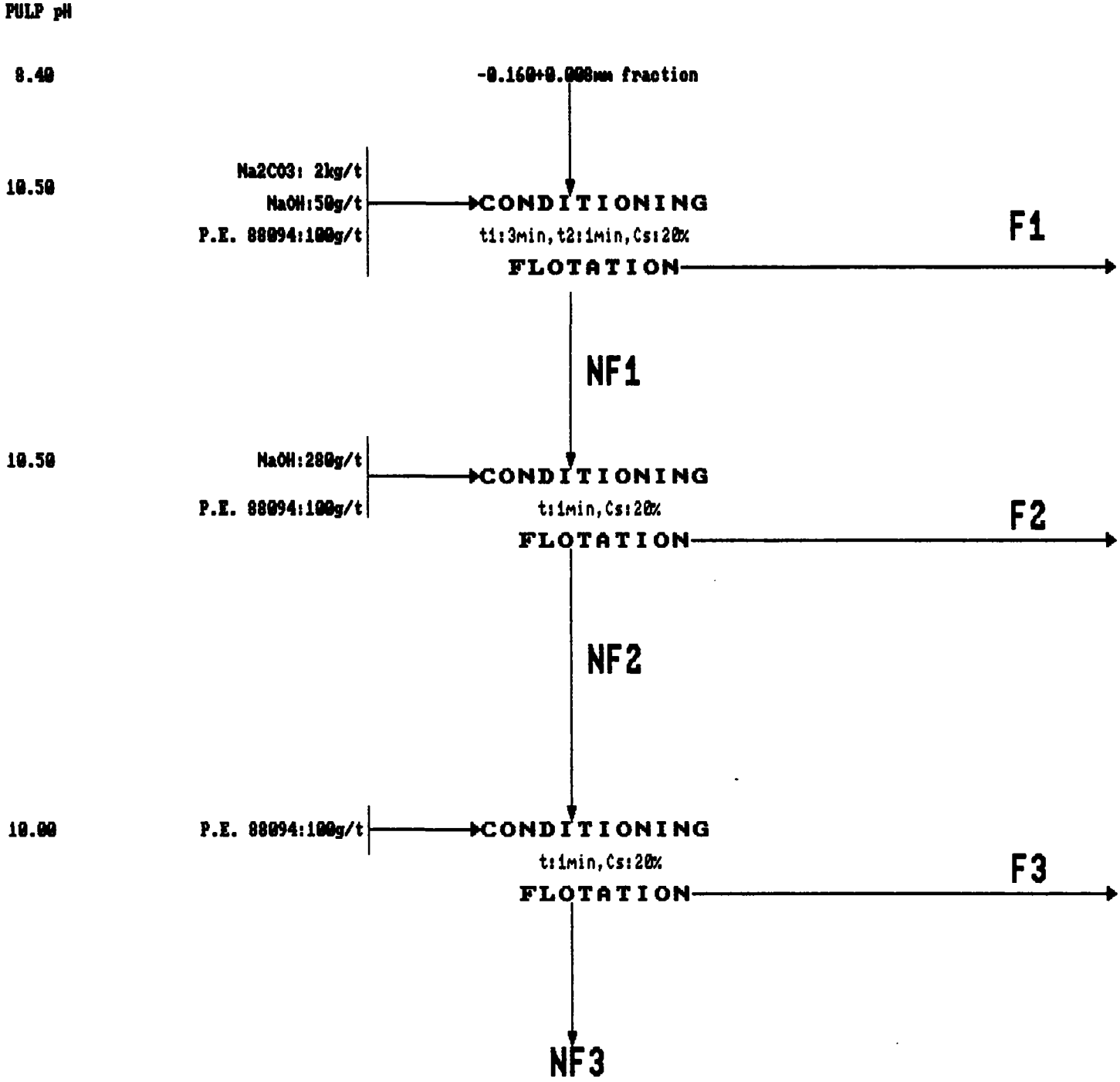


FIGURE N° 2HQ1 : FLOWSHEET OF FLOTATION N°1 ON THE -0.16+0.000 mm FRACTION SEPARATED FROM THE REO ORE SAMPLE N°2 GROUND DOWN TO 0.16mm.

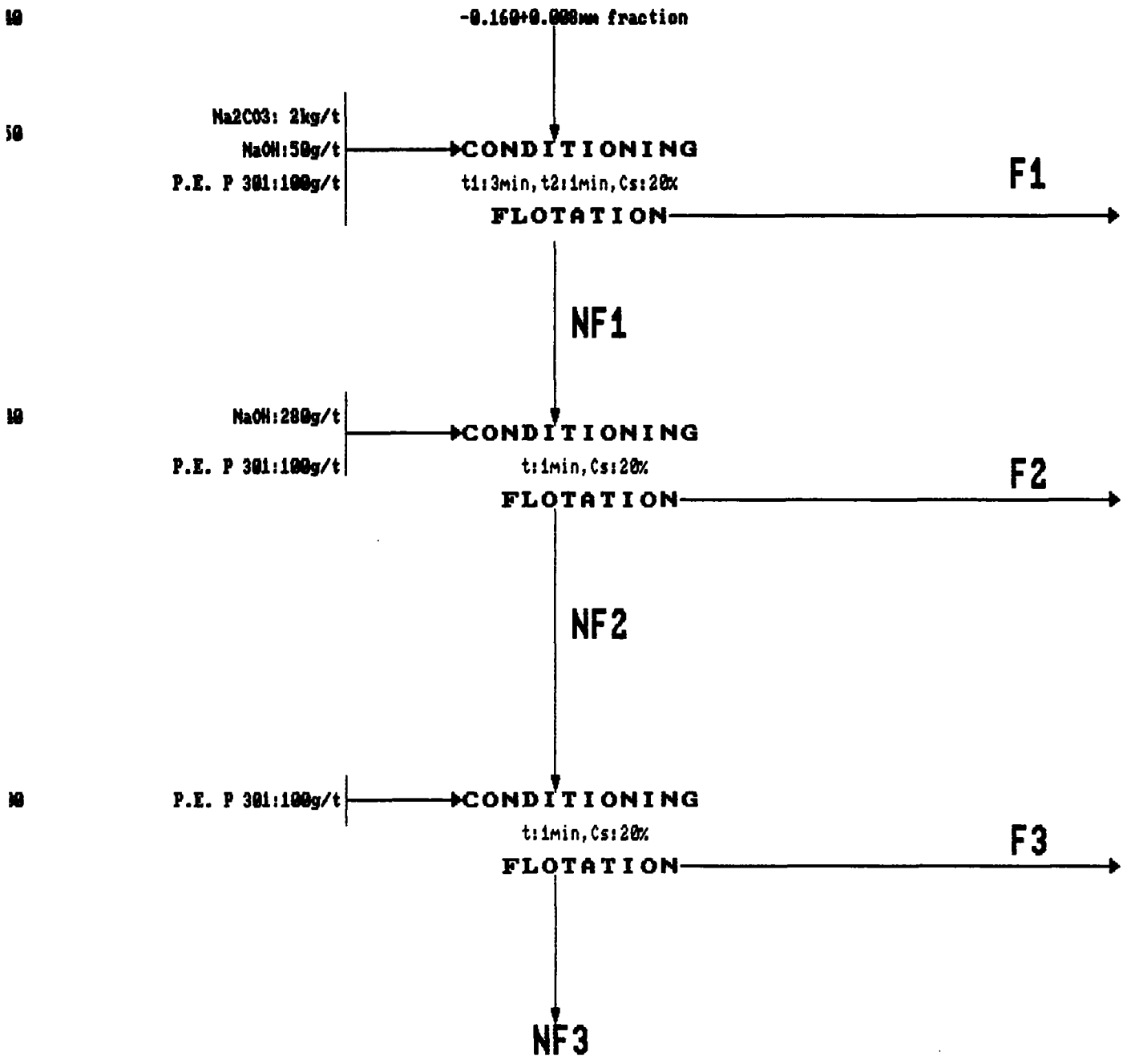


FIGURE N° 2H02 : FLOWSHEET OF FLOTATION N°2 ON THE -0.16+0.000 mm FRACTION SEPARATED FROM THE REO ORE SAMPLE N°2 GROUND DOWN TO 0.16mm.

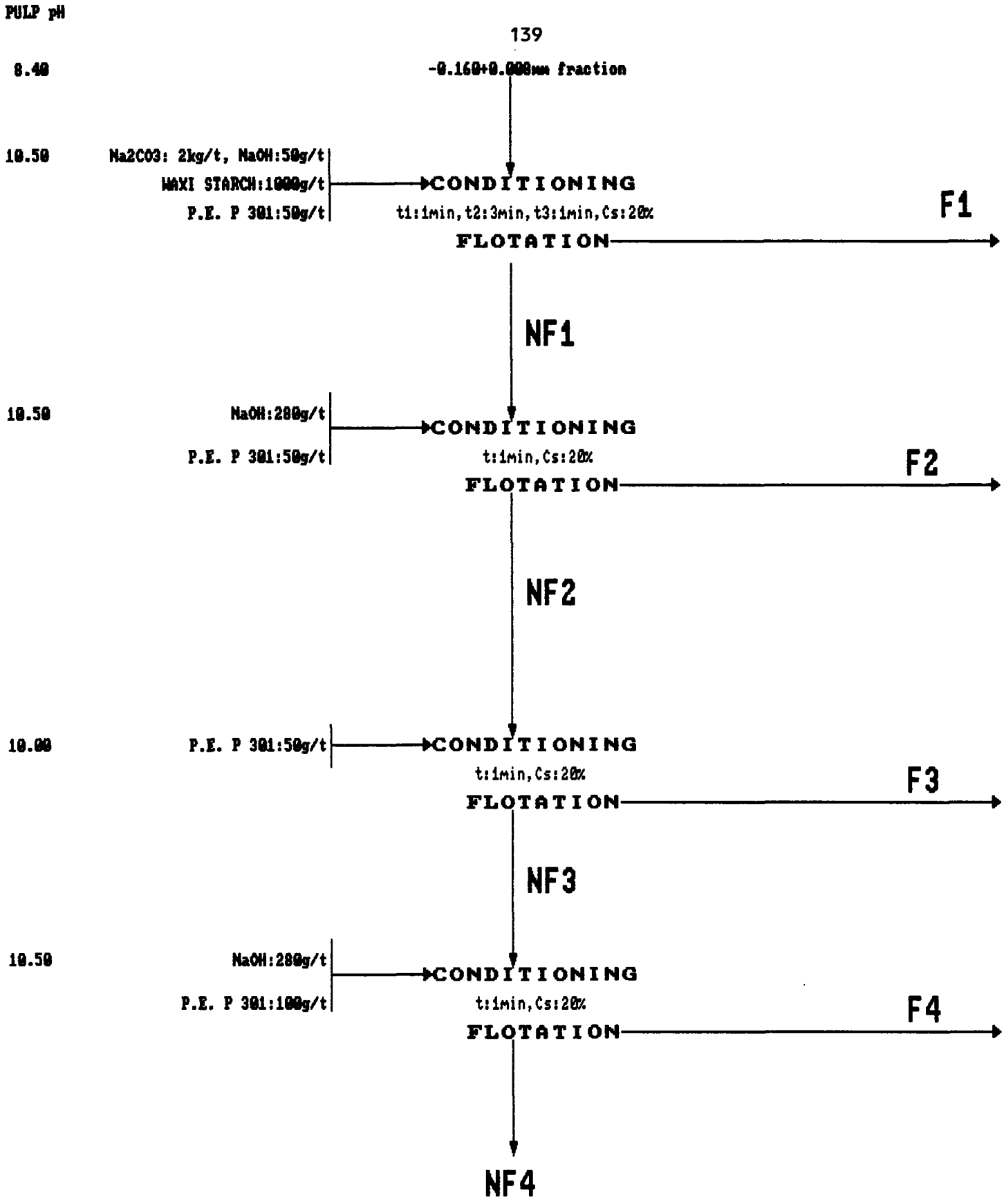


FIGURE N° 2H83 : FLOWSHEET OF FLOTATION N°3 ON THE -0.16+0.000 mm FRACTION SEPARATED FROM THE REO ORE SAMPLE N°2 GROUND DOWN TO 0.16mm.

pH

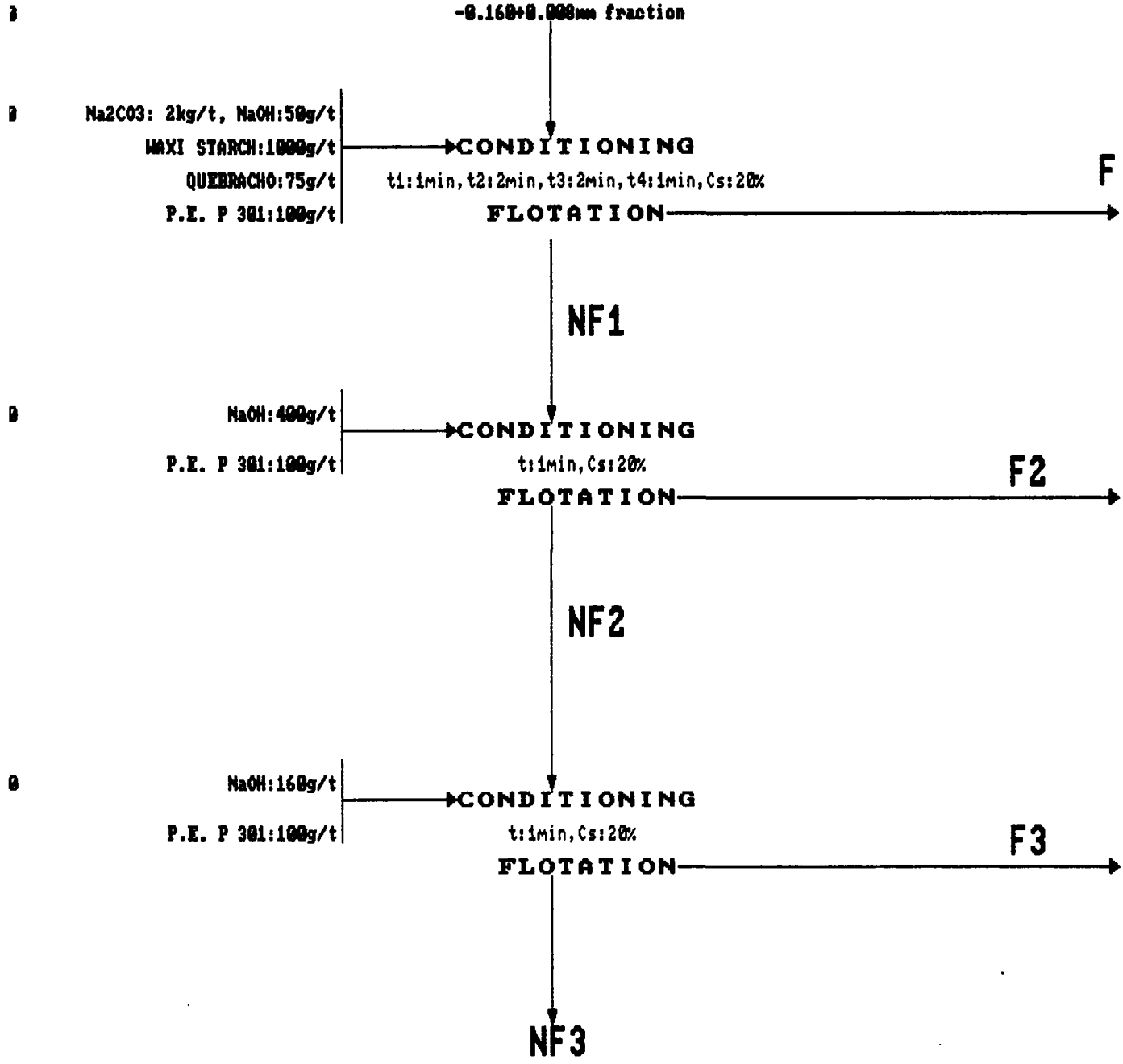


FIGURE N° 2H04 : FLOWSHEET OF FLOTATION N°4 ON THE -0.16+0.000 mm FRACTION SEPARATED FROM THE REO ORE SAMPLE N°2 GROUND DOWN TO 0.16mm.

TABLE N° 2H01 : RED ORE SAMPLE N°2 GROUND DOWN TO 0.16mm,-0.16+0.008mm FRACTION,MATERIAL BALANCE OF FLOTATION TEST N° 1.

PRODUCTS	WEIGHT	Fe2O3		SrO		BaO		La2O3	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1	63.98	8.02	52.39	13.93	77.38	1.75	84.39	2.68	83.42
2 F2	21.80	11.23	24.99	8.02	15.18	.54	8.87	1.17	12.41
3 F3	7.02	12.64	9.06	7.11	4.33	.37	1.96	.72	2.46
4 NF3	7.20	18.45	13.56	4.98	3.11	.88	4.78	.49	1.72
CALCULATED HEAD	100.00	9.80	100.00	11.52	100.00	1.33	100.00	2.06	100.00

PRODUCTS	WEIGHT	CeO2		RED TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1	63.98	4.37	81.83	8.81	82.43
2 F2	21.80	2.12	13.53	4.11	13.10
3 F3	7.02	1.35	2.77	2.59	2.66
4 NF3	7.20	.89	1.88	1.72	1.81
CALCULATED HEAD	100.00	3.42	100.00	6.84	100.00

PRODUCTS	WEIGHT %	RED TOT. GRADE / SrO GRADE
1 F1	63.98	.6324
2 F2	21.80	.5125
3 F3	7.02	.3643
4 NF3	7.20	.3454
CALCULATED HEAD	100.00	.5937

TABLE N° 2H01 : FLOTATION TEST N°1.

PRODUCTS	WEIGHT	Fe2O3		SrO		BaO		La2O3	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1 1 0 0 0 0 0	63.98	8.02	52.39	13.93	77.38	1.75	84.39	2.68	83.42
2 NF1 2 3 4 0 0 0	36.02	12.95	47.61	7.23	22.62	.57	15.61	.95	16.58
3 F2 2 0 0 0 0 0	21.80	11.23	24.99	8.02	15.18	.54	8.87	1.17	12.41
4 NF2 3 4 0 0 0 0	14.22	15.58	22.62	6.03	7.45	.63	6.73	.60	4.18
5 F3 3 0 0 0 0 0	7.02	12.64	9.06	7.11	4.33	.37	1.96	.72	2.46
6 NF3 4 0 0 0 0 0	7.20	18.45	13.56	4.98	3.11	.88	4.78	.49	1.72

PRODUCTS	WEIGHT	CeO2		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1 1 0 0 0 0 0	63.98	4.37	81.83	8.81	82.43
2 NF1 2 3 4 0 0 0	36.02	1.72	18.17	3.34	17.57
3 F2 2 0 0 0 0 0	21.80	2.12	13.53	4.11	13.10
4 NF2 3 4 0 0 0 0	14.22	1.12	4.65	2.15	4.47
5 F3 3 0 0 0 0 0	7.02	1.35	2.77	2.59	2.66
6 NF3 4 0 0 0 0 0	7.20	.89	1.88	1.72	1.81

TABLE N° 2H02 : RED ORE SAMPLE N°2 GROUND DOWN TO 0.16mm,-0.16+0.008mm FRACTION,MATERIAL BALANCE OF FLOTATION TEST N° 2.

PRODUCTS	WEIGHT	Fe203		SrO		BaO		La203	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1	53.76	8.30	46.13	12.86	57.72	1.63	65.79	2.21	58.99
2 F2	35.75	9.87	36.48	11.43	34.12	1.04	27.91	1.96	34.79
3 F3	8.11	14.05	11.78	10.27	6.95	.74	4.51	1.35	5.44
4 NF3	2.38	22.79	5.61	6.07	1.21	1.00	1.79	.66	.78
CALCULATED HEAD	100.00	9.67	100.00	11.98	100.00	1.33	100.00	2.01	100.00

PRODUCTS	WEIGHT	CeO2		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1	53.76	4.00	60.44	7.76	59.91
2 F2	35.75	3.32	33.36	6.60	33.88
3 F3	8.11	2.37	5.40	4.65	5.42
4 NF3	2.38	1.20	.80	2.32	.79
CALCULATED HEAD	100.00	3.56	100.00	6.96	100.00

PRODUCTS	WEIGHT %	REO TOT. GRADE / SrO GRADE
1 F1	53.76	.6034
2 F2	35.75	.5774
3 F3	8.11	.4528
4 NF3	2.38	.3822
CALCULATED HEAD	100.00	.5814

TABLE N° 2HD2 : FLOTATION TEST N° 2.

PRODUCTS	WEIGHT %	Fe2O3		SrO		BaO		La2O3	
		GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1 1 0 0 0 0 0	53.76	8.30	46.13	12.86	57.72	1.63	65.79	2.21	58.99
2 NF1 2 3 4 0 0 0	46.24	11.27	53.87	10.95	42.28	.99	34.21	1.79	41.01
3 F2 2 0 0 0 0 0	35.75	9.87	36.48	11.43	34.12	1.04	27.91	1.96	34.79
4 NF2 3 4 0 0 0 0	10.49	16.03	17.39	9.32	8.16	.80	6.29	1.19	6.22
5 F3 3 0 0 0 0 0	8.11	14.05	11.78	10.27	6.95	.74	4.51	1.35	5.44
6 NF3 4 0 0 0 0 0	2.38	22.79	5.61	6.07	1.21	1.00	1.79	.66	.78

PRODUCTS	WEIGHT %	CeO2		REO TOT.	
		GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1 1 0 0 0 0 0	53.76	4.00	60.44	7.76	59.91
2 NF1 2 3 4 0 0 0	46.24	3.04	39.56	6.04	40.09
3 F2 2 0 0 0 0 0	35.75	3.32	33.36	6.60	33.88
4 NF2 3 4 0 0 0 0	10.49	2.10	6.20	4.12	6.21
5 F3 3 0 0 0 0 0	8.11	2.37	5.40	4.65	5.42
6 NF3 4 0 0 0 0 0	2.38	1.20	.80	2.32	.79

TABLE N° 2H03 : REE ORE SAMPLE N°2 GROUND DOWN TO 0.16mm,-0.16+0.008mm FRACTION,MATERIAL BALANCE OF FLOTATION TEST N°3.

PRODUCTS	WEIGHT	Fe2O3		SrO		BaO		La2O3	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1	7.69	5.28	4.31	22.57	15.48	1.90	11.76	1.82	8.69
2 F2	8.19	6.63	5.77	18.41	13.44	2.81	18.52	1.80	9.15
3 F3	10.28	6.93	7.57	14.13	12.95	2.39	19.77	1.40	8.94
4 F4	26.47	8.37	23.53	12.78	30.16	1.29	27.47	1.52	24.98
5 NF4	47.37	11.69	58.82	6.62	27.96	.59	22.49	1.64	48.24
CALCULATED HEAD	100.00	9.41	100.00	11.21	100.00	1.24	100.00	1.61	100.00

PRODUCTS	WEIGHT	CeO2		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1	7.69	3.26	8.74	6.35	8.73
2 F2	8.19	3.26	9.31	6.33	9.26
3 F3	10.28	2.78	9.97	5.23	9.61
4 F4	26.47	2.75	25.39	5.34	25.26
5 NF4	47.37	2.82	46.59	5.57	47.15
CALCULATED HEAD	100.00	2.87	100.00	5.60	100.00

PRODUCTS	WEIGHT %	REO TOT. GRADE / SrO GRADE
1 F1	7.69	.2813
2 F2	8.19	.3438
3 F3	10.28	.3701
4 F4	26.47	.4178
5 NF4	47.37	.8414
CALCULATED HEAD	100.00	.4990

TABLE N° 2HD3 : FLOTATION TEST N°3.

PRODUCTS	WEIGHT	Fe203		SrO		BaO		La203	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1 1 0 0 0 0 0	7.69	5.28	4.31	22.57	15.48	1.90	11.76	1.82	8.69
2 NF1 2 3 4 5 0 0	92.31	9.76	95.69	10.27	84.52	1.19	88.24	1.59	91.31
3 F2 2 0 0 0 0 0	8.19	6.63	5.77	18.41	13.44	2.81	18.52	1.80	9.15
4 NF2 3 4 5 0 0 0	84.12	10.06	89.92	9.48	71.08	1.03	69.73	1.57	82.16
5 F3 3 0 0 0 0 0	10.28	6.93	7.57	14.13	12.95	2.39	19.77	1.40	8.94
6 NF3 4 5 0 0 0 0	73.84	10.50	82.35	8.83	58.13	.84	49.96	1.60	73.22
7 F4 4 0 0 0 0 0	26.47	8.37	23.53	12.78	30.16	1.29	27.47	1.52	24.98
8 NF4 5 0 0 0 0 0	47.37	11.69	58.82	6.62	27.96	.59	22.49	1.64	48.24

PRODUCTS	WEIGHT	CeO2		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1 1 0 0 0 0 0	7.69	3.26	8.74	6.35	8.73
2 NF1 2 3 4 5 0 0	92.31	2.83	91.26	5.53	91.27
3 F2 2 0 0 0 0 0	8.19	3.26	9.31	6.33	9.26
4 NF2 3 4 5 0 0 0	84.12	2.79	81.94	5.46	82.01
5 F3 3 0 0 0 0 0	10.28	2.78	9.97	5.23	9.61
6 NF3 4 5 0 0 0 0	73.84	2.79	71.98	5.49	72.40
7 F4 4 0 0 0 0 0	26.47	2.75	25.39	5.34	25.26
8 NF4 5 0 0 0 0 0	47.37	2.82	46.59	5.57	47.15

TABLE N° 2H04 : REO ORE SAMPLE N°2 GROUND DOWN TO 0.16mm,-0.16+0.008mm FRACTION,MATERIAL BALANCE OF FLOTATION TEST N° 4.

PRODUCTS	WEIGHT	Fe2O3		SrO		BaO		La2O3	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1	14.47	5.84	9.33	18.30	23.05	2.34	25.97	1.47	11.93
2 F2	20.12	6.77	15.03	15.22	26.66	1.84	28.40	1.43	16.14
3 F3	22.28	7.03	17.29	11.91	23.10	1.16	19.82	1.67	20.87
4 NF3	43.13	12.26	58.36	7.24	27.19	.78	25.81	2.11	51.05
CALCULATED HEAD	100.00	9.06	100.00	11.49	100.00	1.30	100.00	1.78	100.00

PRODUCTS	WEIGHT	CeO2		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1	14.47	2.93	13.06	5.50	12.66
2 F2	20.12	2.85	17.66	5.35	17.12
3 F3	22.28	3.05	20.93	5.90	20.91
4 NF3	43.13	3.64	48.35	7.19	49.32
CALCULATED HEAD	100.00	3.25	100.00	6.29	100.00

PRODUCTS	WEIGHT %	REO TOT. GRADE / SrO GRADE
1 F1	14.47	.3005
2 F2	20.12	.3515
3 F3	22.28	.4954
4 NF3	43.13	.9931
CALCULATED HEAD	100.00	.5474

TABLE N° 2HD4 : FLOTATION TEST N°4.

PRODUCTS	WEIGHT	Fe2O3		SrO		BaO		La2O3	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1 1 0 0 0 0 0	14.47	5.84	9.33	18.30	23.05	2.34	25.97	1.47	11.93
2 NF1 2 3 4 0 0 0	85.53	9.61	90.67	10.33	76.95	1.13	74.03	1.84	88.07
3 F2 2 0 0 0 0 0	20.12	6.77	15.03	15.22	26.66	1.84	28.40	1.43	16.14
4 NF2 3 4 0 0 0 0	65.41	10.48	75.64	8.83	50.29	.91	45.63	1.96	71.93
5 F3 3 0 0 0 0 0	22.28	7.03	17.29	11.91	23.10	1.16	19.82	1.67	20.87
6 NF3 4 0 0 0 0 0	43.13	12.26	58.36	7.24	27.19	.78	25.81	2.11	51.05

PRODUCTS	WEIGHT	CeO2		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1 1 0 0 0 0 0	14.47	2.93	13.06	5.50	12.66
2 NF1 2 3 4 0 0 0	85.53	3.30	86.94	6.42	87.34
3 F2 2 0 0 0 0 0	20.12	2.85	17.66	5.35	17.12
4 NF2 3 4 0 0 0 0	65.41	3.44	69.28	6.75	70.22
5 F3 3 0 0 0 0 0	22.28	3.05	20.93	5.90	20.91
6 NF3 4 0 0 0 0 0	43.13	3.64	48.35	7.19	49.32

3.2.2. Flotation of the - 50 + 10 µm fraction

Flotation tests were made on a fine sized fraction so as to suppress particle size effects which often occur when floating feeds with a broad size distribution, using phosphoric ester as a collector.

Operating conditions and flowsheets of flotation tests are shown in figures 2H05 to 2H12.

Material balances are shown in tables 2H05 to 2H12.

Results of flotation 5 confirm difficulties encountered in separating monazite from a high carbonate gangue. Similar distributions of various REE show that monazite is probably the major REE bearing mineral with an almost homogeneous composition.

Addition of CO_2 up to near water saturation to provide a slightly acidic pH: 5.3. to 5.8, significantly improves depression of carbonates. The best concentration performance was achieved in test 8 using, phosphoric ester P301 as a monazite collector, citric and oxalic acids as co-depressants for iron containing dolomite and siderite. The final concentrate assays 43.14 % REO, 0.17 % BaO, 2.32 % SrO and 1 % Fe_2O_3, however total REO losses in non float fractions are high with respect to REO losses in light fractions separated by gravity concentration. Addition of quebracho or Ca lignosulfonate appears to reduce selectivity for carbonates.

Results of test n° 9 show that strontianite responds well to flotation with petroleum sulfonate as a collector and H_2SO_4 as a barite depressant, it should be noted that selectivity for monazite is poor. Strontianite is also floated together with barite by fatty acids under alkaline pH conditions (test n° 10), however, flotation is not selective for monazite.

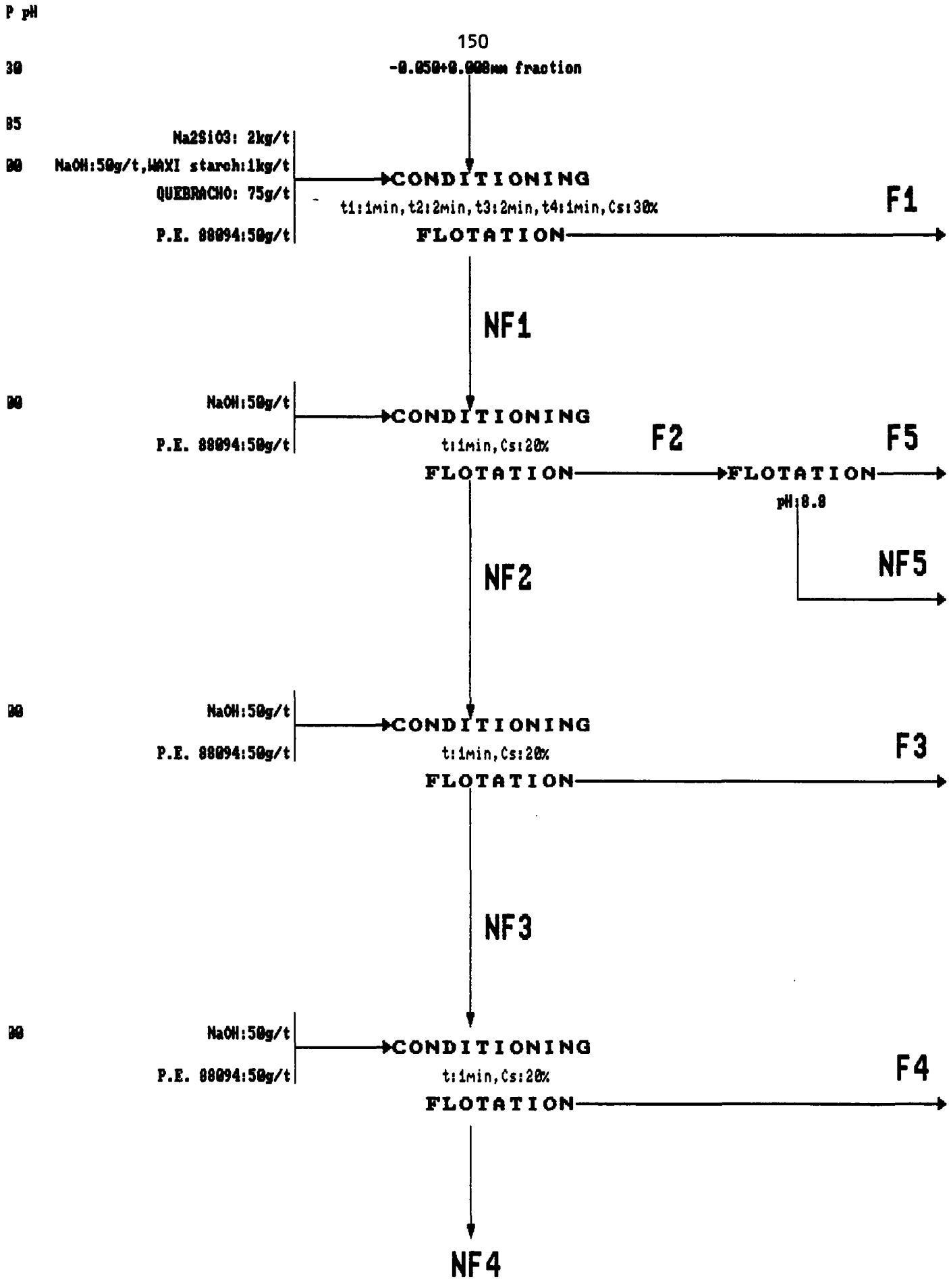


FIGURE N° 2H05 : FLOWSHEET OF FLOTATION N°5 ON THE -0.05+0.000 mm FRACTION

SEPARATED FROM THE REO ORE SAMPLE GROUND DOWN TO 0.16mm.

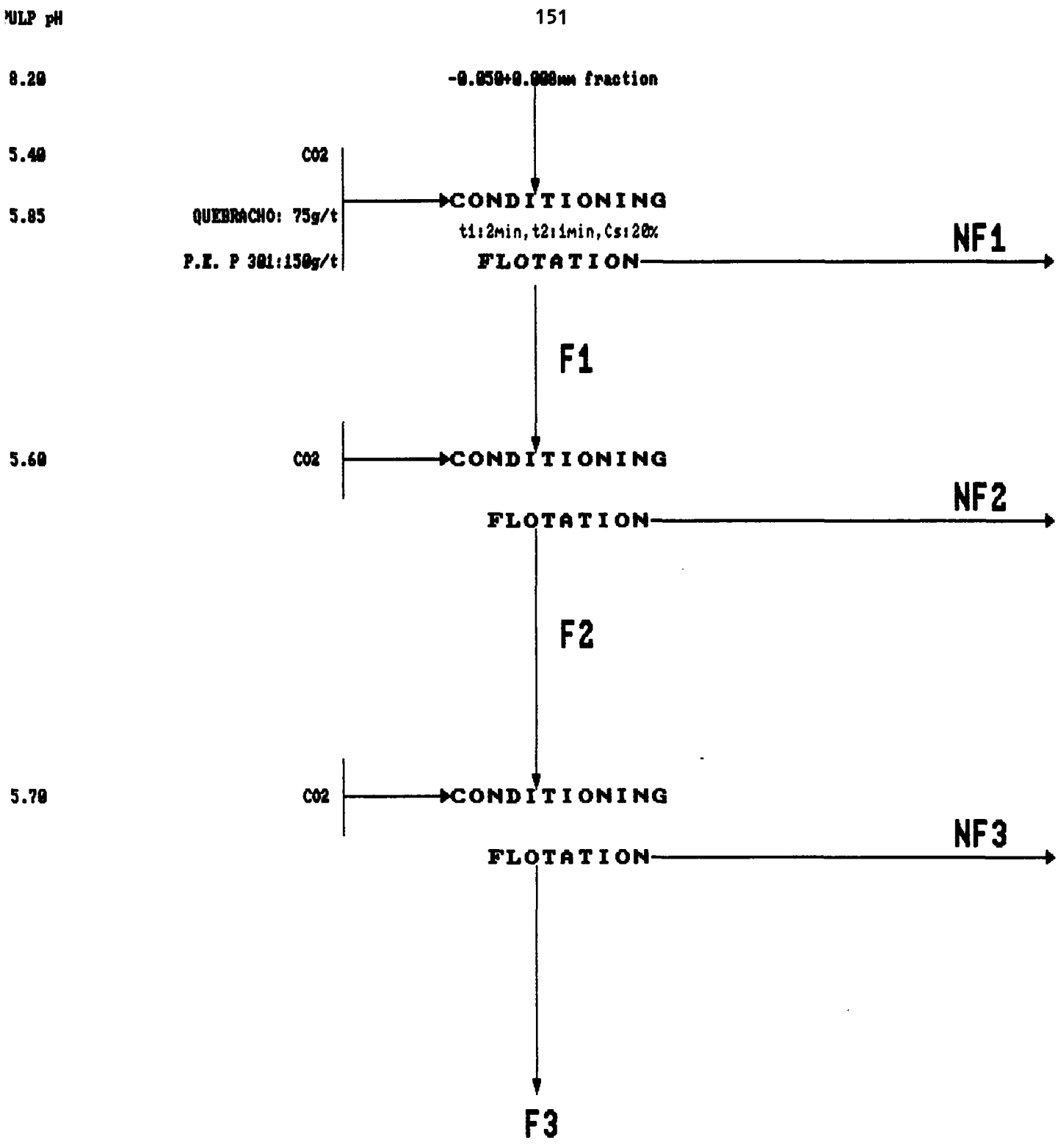


FIGURE N° 2H07 : FLOWSHEET OF FLOTATION N°7 ON THE -0.05+0.000 mm FRACTION SEPARATED FROM THE REO ORE SAMPLE GROUND DOWN TO 0.16mm.

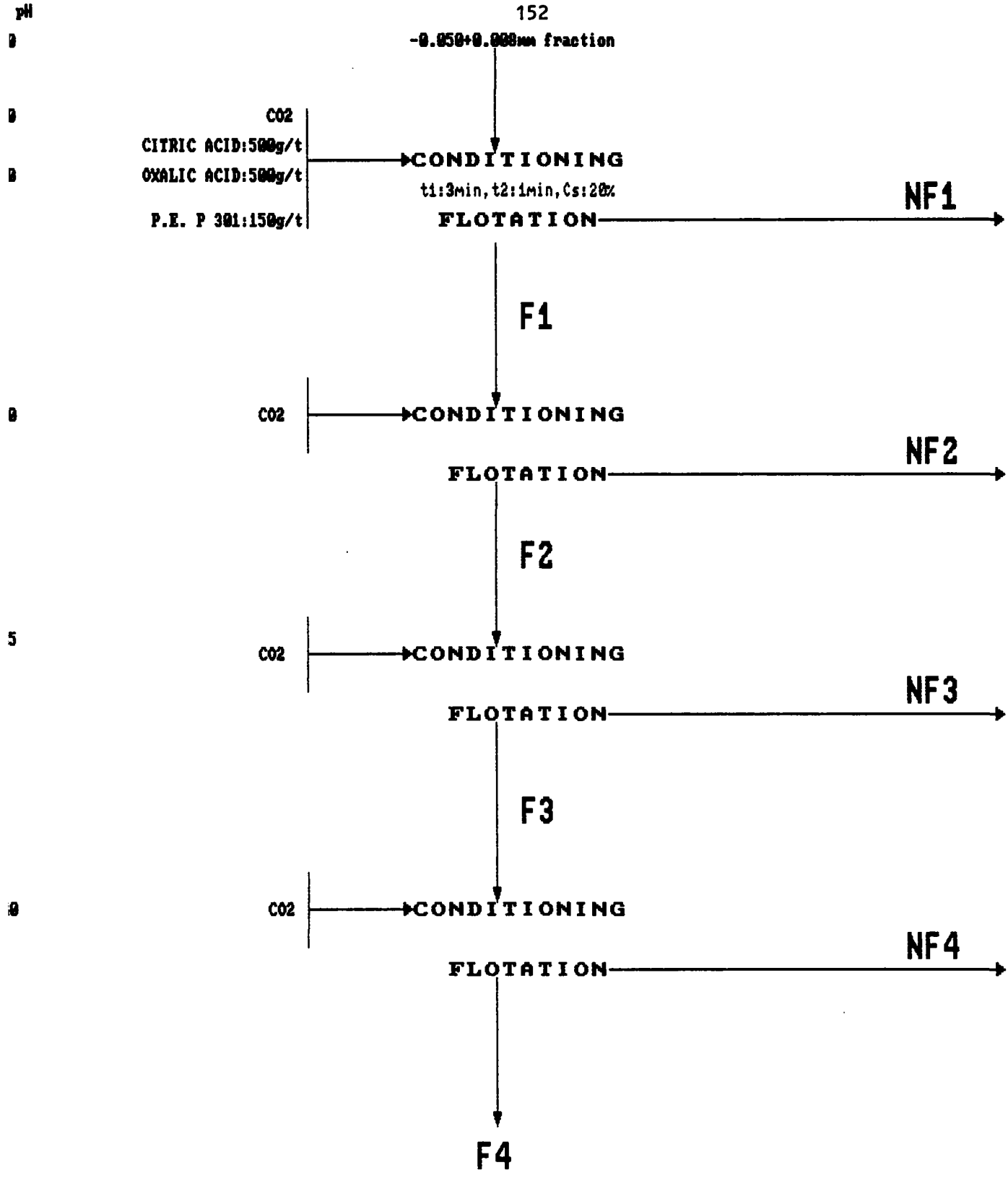


FIGURE N° 2H08 : FLOWSHEET OF FLOTATION N°8 ON THE -0.05+0.000 mm FRACTION SEPARATED FROM THE REO ORE SAMPLE GROUND DOWN TO 0.16mm.

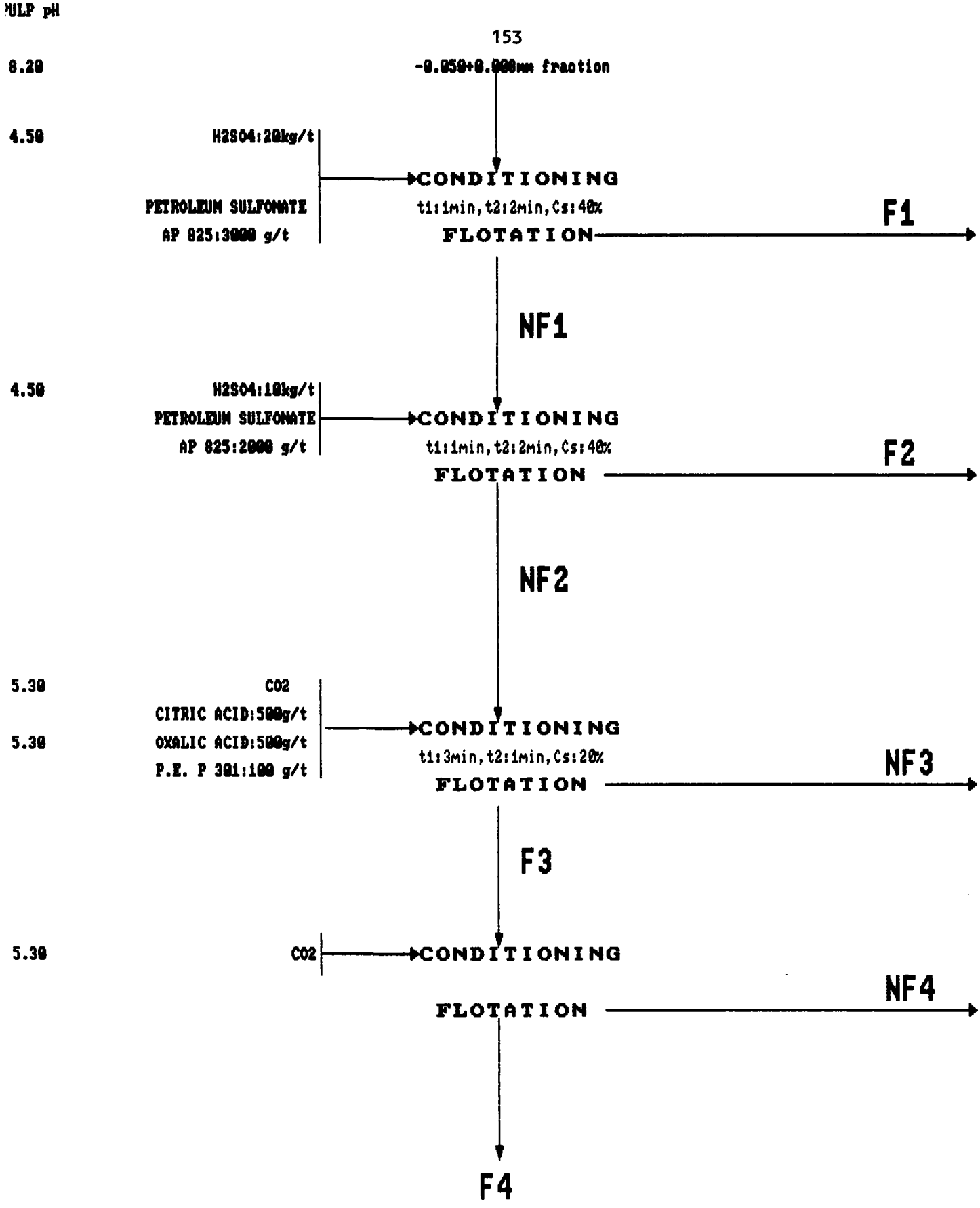


FIGURE N° 2H09 : FLOWSHEET OF FLOTATION N°9 ON THE -0.05+0.000 mm FRACTION SEPARATED FROM THE REO ORE SAMPLE N°2 GROUND DOWN TO 0.16mm.

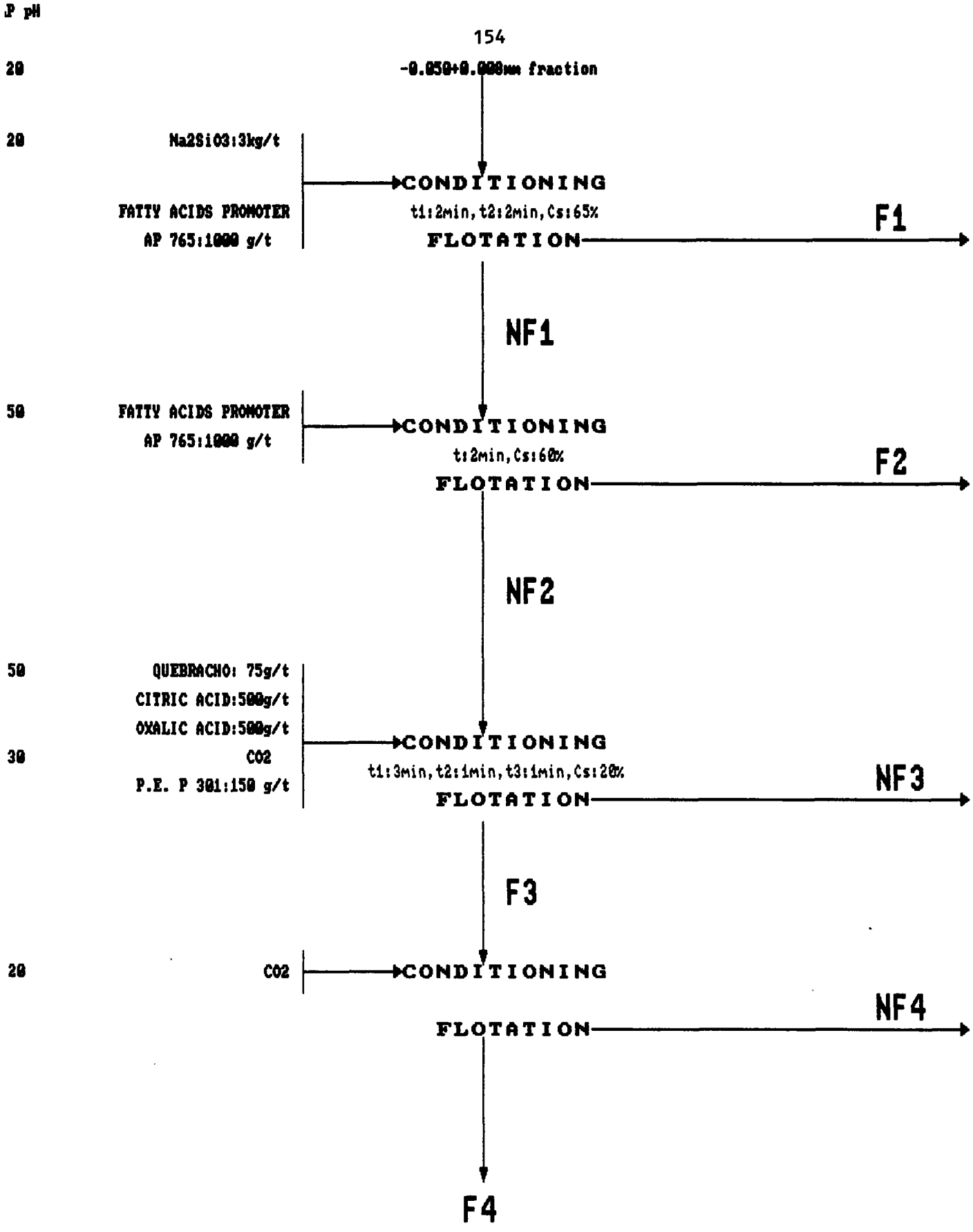


FIGURE N° 2H10 : FLOWSHEET OF FLOTATION N°10 ON THE -0.05+0.000 mm FRACTION SEPARATED FROM THE REO ORE SAMPLE N°2 GROUND DOWN TO 0.16mm.

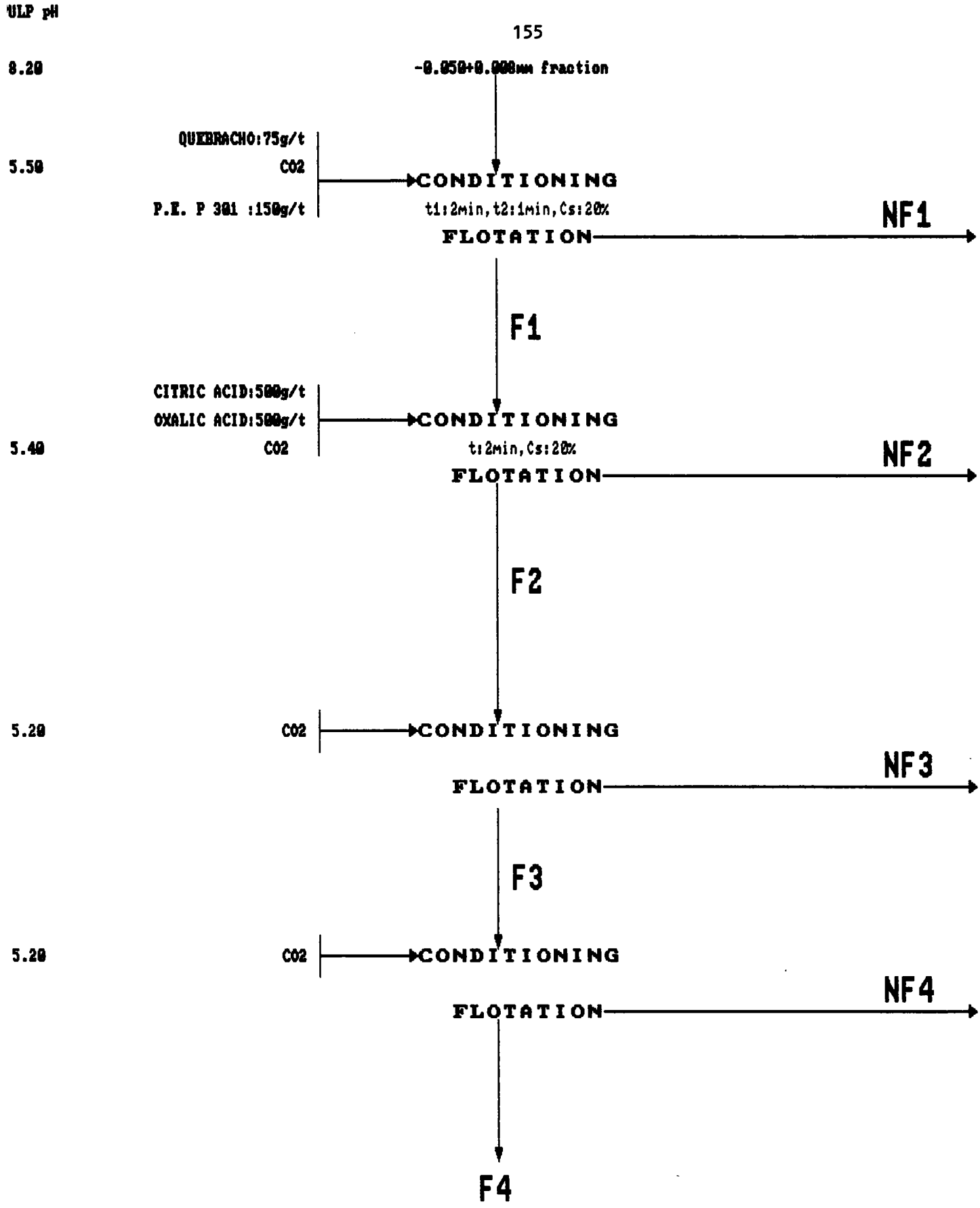


FIGURE N° 2H11 : FLOWSHEET OF FLOTATION N°11 ON THE -0.85+0.000 mm FRACTION SEPARATED FROM THE REO ORE SAMPLE N°2 GROUND DOWN TO 0.16mm.

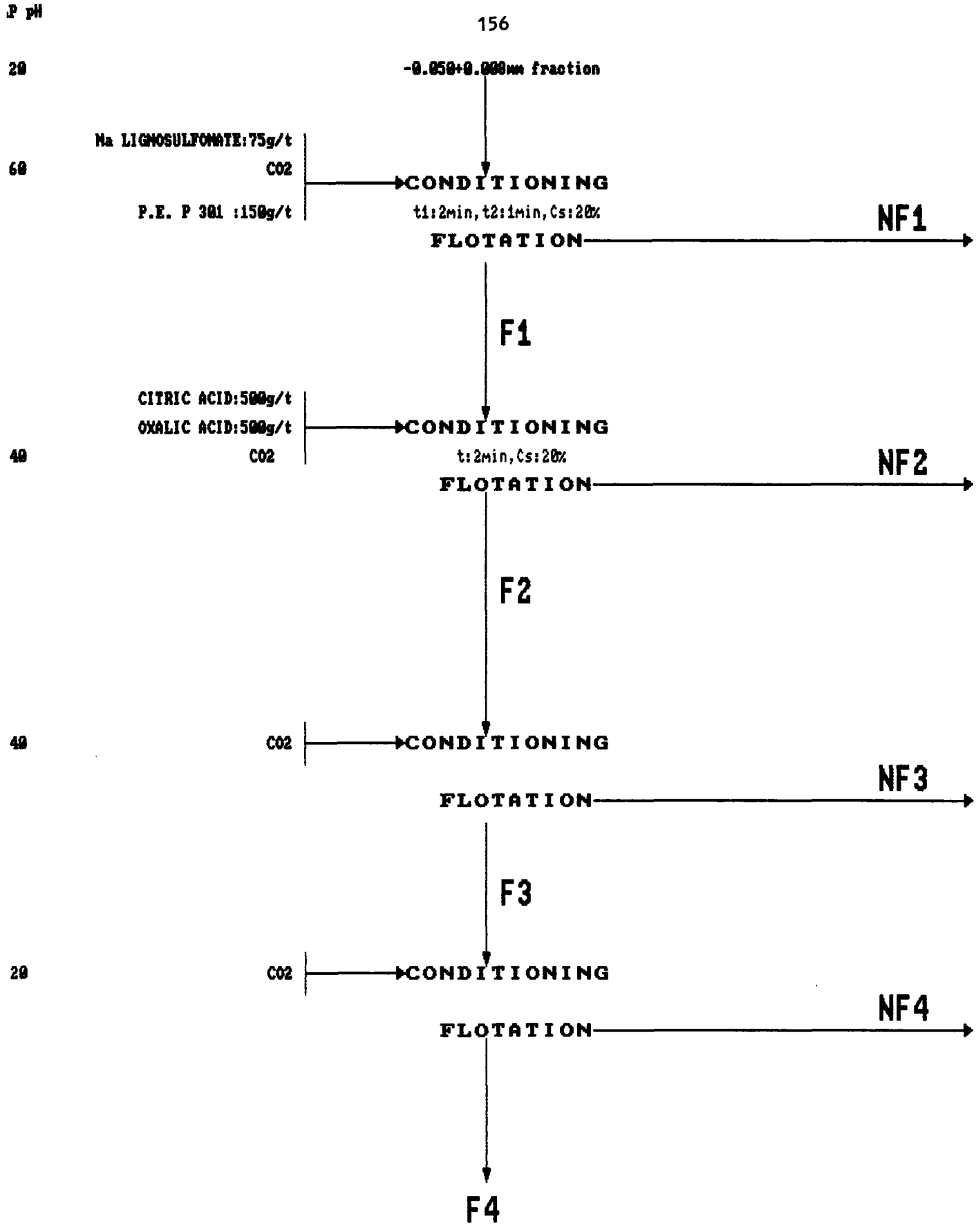


FIGURE N° 2H12 : FLOWSHEET OF FLOTATION N°12 ON THE -0.05+0.000 mm FRACTION SEPARATED FROM THE REO ORE SAMPLE N°2 GROUND DOWN TO 0.16mm.

TABLE N° 2H05 A : REO ORE SAMPLE N° 2,GROUND DOWN TO 0.16mm, FRACTION -0.05+0.008mm,MATERIAL BALANCE OF FLOTATION TEST N° 5.

PRODUCTS	WEIGHT	CO2		SrO		La2O3		CeO2	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1	19.16	29.90	19.38	16.67	32.06	4.73	23.15	8.19	22.61
2 F5	10.67	26.70	9.64	13.84	14.82	3.44	9.38	6.11	9.39
3 NF5	13.56	32.20	14.77	11.51	15.66	6.67	23.11	11.78	23.01
4 F3	23.66	32.90	26.33	9.40	22.32	4.35	26.29	7.77	26.49
5 F4	17.85	33.70	20.34	6.30	11.29	2.99	13.63	5.39	13.86
6 NF4	15.10	18.70	9.55	2.54	3.85	1.15	4.44	2.13	4.63
CALCULATED HEAD	100.00	29.57	100.00	9.96	100.00	3.91	100.00	6.94	100.00

PRODUCTS	WEIGHT	REO TOT.		REE+Y		CERICS		YTTRICS	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADEppm	RECOVERY
1 F1	19.16	16.15	22.80	13.13	22.60	13.06	22.61	649.60	21.87
2 F5	10.67	11.94	9.39	9.82	9.41	9.77	9.41	501.50	9.40
3 NF5	13.56	23.06	23.04	18.85	22.96	18.76	22.98	842.70	20.08
4 F3	23.66	15.15	26.42	12.47	26.52	12.41	26.52	646.50	26.88
5 F4	17.85	10.48	13.79	8.65	13.88	8.60	13.87	492.40	15.44
6 NF4	15.10	4.10	4.56	3.41	4.62	3.38	4.61	238.60	6.33
CALCULATED HEAD	100.00	13.57	100.00	11.13	100.00	11.07	100.00	569.13	100.00

PRODUCTS	WEIGHT %	CERICS GRADE	REE+Y / GRADE
1 F1	19.16		.9951
2 F5	10.67		.9949
3 NF5	13.56		.9955
4 F3	23.66		.9948
5 F4	17.85		.9943
6 NF4	15.10		.9930
CALCULATED HEAD	100.00		.9949

TABLE N° 2H05 A : FLOTATION TEST N°5

PRODUCTS	WEIGHT	CO2		SrO		La2O3		CeO2	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1 1 0 0 0 0 0	19.16	29.90	19.38	16.67	32.06	4.73	23.15	8.19	22.61
2 NF1 2 3 4 5 6 0	80.84	29.49	80.62	8.37	67.94	3.72	76.85	6.64	77.39
3 F2 2 3 0 0 0 0	24.23	29.78	24.40	12.54	30.49	5.25	32.48	9.28	32.41
4 NF2 4 5 6 0 0 0	56.61	29.36	56.22	6.59	37.46	3.07	44.36	5.52	44.98
5 F3 4 0 0 0 0 0	23.66	32.90	26.33	9.40	22.32	4.35	26.29	7.77	26.49
6 NF3 5 6 0 0 0 0	32.95	26.83	29.89	4.58	15.14	2.15	18.07	3.90	18.50
7 F4 5 0 0 0 0 0	17.85	33.70	20.34	6.30	11.29	2.99	13.63	5.39	13.86
8 NF4 6 0 0 0 0 0	15.10	18.70	9.55	2.54	3.85	1.15	4.44	2.13	4.63
9 F1+NF5+F3 1 3 4 0 0 0	56.38	31.71	60.47	12.38	70.04	5.04	72.55	8.88	72.11
10 F4+NF4+F5 5 6 2 0 0 0	43.62	26.80	39.53	6.84	29.96	2.46	27.45	4.44	27.89

PRODUCTS	WEIGHT	REO TOT.		REE+Y		CERICS		YTTRICS	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADEppm	RECOVERY
1 F1 1 0 0 0 0 0	19.16	16.15	22.80	13.13	22.60	13.06	22.61	649.60	21.87
2 NF1 2 3 4 5 6 0	80.84	12.96	77.20	10.65	77.40	10.60	77.39	550.05	78.13
3 F2 2 3 0 0 0 0	24.23	18.16	32.43	14.87	32.38	14.80	32.39	692.45	29.48
4 NF2 4 5 6 0 0 0	56.61	10.73	44.76	8.85	45.02	8.80	45.00	489.11	48.65
5 F3 4 0 0 0 0 0	23.66	15.15	26.42	12.47	26.52	12.41	26.52	646.50	26.88
6 NF3 5 6 0 0 0 0	32.95	7.56	18.35	6.25	18.50	6.21	18.49	376.09	21.77
7 F4 5 0 0 0 0 0	17.85	10.48	13.79	8.65	13.88	8.60	13.87	492.40	15.44
8 NF4 6 0 0 0 0 0	15.10	4.10	4.56	3.41	4.62	3.38	4.61	238.60	6.33
9 F1+NF5+F3 1 3 4 0 0 0	56.38	17.39	72.26	14.23	72.09	14.16	72.10	694.74	68.82
10 F4+NF4+F5 5 6 2 0 0 0	43.62	8.63	27.74	7.12	27.91	7.08	27.90	406.77	31.18

TABLE N° 2HD5 B : REO ORE SAMPLE N° 2,GROUND DOWN TO 0.16mm, FRACTION -0.05+0.008mm,MATERIAL BALANCE OF FLOTATION TEST N° 5.

PRODUCTS	WEIGHT	La		Ce		Pr		Nd	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADEppm	RECOVERY	GRADE %	RECOVERY
1 F1	19.16	4.03	23.14	6.67	22.61	6155.00	22.02	1.64	21.68
2 F5	10.67	2.93	9.37	4.97	9.38	4771.00	9.50	1.30	9.57
3 NF5	13.56	5.69	23.12	9.59	23.01	9027.00	22.85	2.42	22.64
4 F3	23.66	3.71	26.30	6.33	26.50	6058.00	26.76	1.65	26.93
5 F4	17.85	2.55	13.64	4.39	13.87	4231.00	14.10	1.16	14.28
6 NF4	15.10	.98	4.43	1.73	4.62	1694.00	4.78	.47	4.90
CALCULATED HEAD	100.00	3.34	100.00	5.65	100.00	5356.77	100.00	1.45	100.00

PRODUCTS	WEIGHT	Sm		Eu		Gd		Tb	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 F1	19.16	1078.00	21.10	170.00	20.13	255.00	22.64	22.30	24.04
2 F5	10.67	884.00	9.63	146.50	9.66	186.50	9.22	15.50	9.31
3 NF5	13.56	1595.00	22.09	258.10	21.63	321.00	20.17	26.30	20.07
4 F3	23.66	1123.00	27.14	190.00	27.79	244.00	26.75	19.20	25.56
5 F4	17.85	809.00	14.75	138.00	15.23	183.60	15.19	15.00	15.07
6 NF4	15.10	343.00	5.29	59.50	5.55	86.20	6.03	7.00	5.95
CALCULATED HEAD	100.00	979.05	100.00	161.77	100.00	215.80	100.00	17.77	100.00

PRODUCTS	WEIGHT %	La GRADE / Ce GRADE
1 F1	19.16	.6042
2 F5	10.67	.5895
3 NF5	13.56	.5933
4 F3	23.66	.5861
5 F4	17.85	.5809
6 NF4	15.10	.5665
CALCULATED HEAD	100.00	.5905

TABLE N° 2H05 B :FLOTATION TEST N°5.

PRODUCTS	WEIGHT	La		Ce		Pr		Nd	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADEppm	RECOVERY	GRADE %	RECOVERY
1 F1 1 0 0 0 0 0	19.16	4.03	23.14	6.67	22.61	6155.00	22.02	1.64	21.68
2 NF1 2 3 4 5 6 0	80.84	3.17	76.86	5.41	77.39	5167.59	77.98	1.40	78.32
3 F2 2 3 0 0 0 0	24.23	4.47	32.49	7.56	32.39	7152.81	32.35	1.93	32.21
4 NF2 4 5 6 0 0 0	56.61	2.62	44.38	4.49	44.99	4317.88	45.63	1.18	46.11
5 F3 4 0 0 0 0 0	23.66	3.71	26.30	6.33	26.50	6058.00	26.76	1.65	26.93
6 NF3 5 6 0 0 0 0	32.95	1.83	18.07	3.17	18.49	3068.37	18.87	.84	19.18
7 F4 5 0 0 0 0 0	17.85	2.55	13.64	4.39	13.87	4231.00	14.10	1.16	14.28
8 NF4 6 0 0 0 0 0	15.10	.98	4.43	1.73	4.62	1694.00	4.78	.47	4.90
9 F1+NF5+F3 1 3 4 0 0 0	56.38	4.29	72.56	7.23	72.13	6805.04	71.62	1.83	71.25
10 F4+NF4+F5 5 6 2 0 0 0	43.62	2.10	27.44	3.61	27.87	3484.85	28.38	.96	28.75

PRODUCTS	WEIGHT	Sm		Eu		Gd		Tb	
	%	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY	GRADEppm	RECOVERY
1 F1 1 0 0 0 0 0	19.16	1078.00	21.10	170.00	20.13	255.00	22.64	22.30	24.04
2 NF1 2 3 4 5 6 0	80.84	955.60	78.90	159.82	79.87	206.51	77.36	16.70	75.96
3 F2 2 3 0 0 0 0	24.23	1281.90	31.73	208.96	31.30	261.77	29.39	21.54	29.38
4 NF2 4 5 6 0 0 0	56.61	815.94	47.18	138.79	48.57	182.86	47.97	14.62	46.58
5 F3 4 0 0 0 0 0	23.66	1123.00	27.14	190.00	27.79	244.00	26.75	19.20	25.56
6 NF3 5 6 0 0 0 0	32.95	595.45	20.04	102.03	20.78	138.96	21.22	11.33	21.02
7 F4 5 0 0 0 0 0	17.85	809.00	14.75	138.00	15.23	183.60	15.19	15.00	15.07
8 NF4 6 0 0 0 0 0	15.10	343.00	5.29	59.50	5.55	86.20	6.03	7.00	5.95
9 F1+NF5+F3 1 3 4 0 0 0	56.38	1221.23	70.33	199.58	69.56	266.26	69.56	21.96	69.68
10 F4+NF4+F5 5 6 2 0 0 0	43.62	666.03	29.67	112.90	30.44	150.59	30.44	12.35	30.32

TABLE N° 2H05 C : RED ORE SAMPLE N° 2,GROUND DOWN TO 0.16mm, FRACTION -0.05+0.008mm,MATERIAL BALANCE OF FLOTATION TEST N° 5.

PRODUCTS	WEIGHT	Dy		Ho		Er		Tm	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1	19.16	65.30	21.30	6.00	21.11	10.20	20.21	.80	20.00
2 F5	10.67	52.00	9.45	5.00	9.79	9.00	9.93	.60	8.35
3 NF5	13.56	86.50	19.97	7.70	19.17	13.30	18.65	1.00	17.70
4 F3	23.66	68.00	27.39	6.00	26.06	11.00	26.91	.90	27.79
5 F4	17.85	50.20	15.26	5.00	16.39	9.10	16.80	.70	16.31
6 NF4	15.10	25.80	6.63	2.70	7.48	4.80	7.50	.50	9.85
CALCULATED HEAD	100.00	58.73	100.00	5.45	100.00	9.67	100.00	.77	100.00

PRODUCTS	WEIGHT	Yb		Lu		Y	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1	19.16	4.50	20.09	.50	18.27	115.00	23.35
2 F5	10.67	3.80	9.45	.60	12.21	82.00	9.27
3 NF5	13.56	5.20	16.43	.60	15.52	123.00	17.68
4 F3	23.66	4.90	27.01	.50	22.57	102.00	25.58
5 F4	17.85	4.30	17.88	.50	17.02	86.00	16.27
6 NF4	15.10	2.60	9.15	.50	14.40	49.00	7.84
CALCULATED HEAD	100.00	4.29	100.00	.52	100.00	94.35	100.00

TABLE N° 2H05 C : FLOTATION TEST N° 5.

PRODUCTS	WEIGHT	Dy		Ho		Er		Tm	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1 1 0 0 0 0 0	19.16	65.30	21.30	6.00	21.11	10.20	20.21	.80	20.00
2 NF1 2 3 4 5 6 0	80.84	57.18	78.70	5.32	78.89	9.54	79.79	.76	80.00
3 F2 2 3 0 0 0 0	24.23	71.31	29.42	6.51	28.96	11.41	28.58	.82	26.05
4 NF2 4 5 6 0 0 0	56.61	51.13	49.28	4.80	49.93	8.75	51.21	.73	53.95
5 F3 4 0 0 0 0 0	23.66	68.00	27.39	6.00	26.06	11.00	26.91	.90	27.79
6 NF3 5 6 0 0 0 0	32.95	39.02	21.89	3.95	23.87	7.13	24.29	.61	26.16
7 F4 5 0 0 0 0 0	17.85	50.20	15.26	5.00	16.39	9.10	16.80	.70	16.31
8 NF4 6 0 0 0 0 0	15.10	25.80	6.63	2.70	7.48	4.80	7.50	.50	9.85
9 F1+NF5+F3 1 3 4 0 0 0	56.38	71.53	68.66	6.41	66.34	11.28	65.78	.89	65.49
10 F4+NF4+F5 5 6 2 0 0 0	43.62	42.19	31.34	4.20	33.66	7.59	34.22	.61	34.51

PRODUCTS	WEIGHT	Yb		Lu		Y	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1 1 0 0 0 0 0	19.16	4.50	20.09	.50	18.27	115.00	23.35
2 NF1 2 3 4 5 6 0	80.84	4.24	79.91	.53	81.73	89.45	76.65
3 F2 2 3 0 0 0 0	24.23	4.58	25.87	.60	27.73	104.95	26.95
4 NF2 4 5 6 0 0 0	56.61	4.10	54.04	.50	53.99	82.82	49.69
5 F3 4 0 0 0 0 0	23.66	4.90	27.01	.50	22.57	102.00	25.58
6 NF3 5 6 0 0 0 0	32.95	3.52	27.03	.50	31.43	69.04	24.11
7 F4 5 0 0 0 0 0	17.85	4.30	17.88	.50	17.02	86.00	16.27
8 NF4 6 0 0 0 0 0	15.10	2.60	9.15	.50	14.40	49.00	7.84
9 F1+NF5+F3 1 3 4 0 0 0	56.38	4.84	63.52	.52	56.36	111.47	66.61
10 F4+NF4+F5 5 6 2 0 0 0	43.62	3.59	36.48	.52	43.64	72.21	33.39

TABLE N° 2H07 : RED ORE SAMPLE GROUND DOWN TO 0.16mm,-0.05+0.008mm FRACTION,MATERIAL BALANCE OF FLOTATION TEST N° 7.

PRODUCTS	WEIGHT	Fe2O3		SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1	25.79	14.30	38.18	8.30	19.46	2.03	34.84	4.81	14.26
2 NF2	37.35	8.90	34.41	14.31	48.59	1.90	47.23	6.11	26.24
3 NF3	20.59	9.26	19.74	12.22	22.87	1.00	13.70	8.40	19.88
4 F3	16.27	4.55	7.66	6.14	9.08	.39	4.22	21.18	39.62
CALCULATED HEAD	100.00	9.66	100.00	11.00	100.00	1.50	100.00	8.70	100.00

PRODUCTS	WEIGHT %	REO TOT. GRADE / SrO GRADE
1 NF1	25.79	.5795
2 NF2	37.35	.4270
3 NF3	20.59	.6874
4 F3	16.27	3.4495
CALCULATED HEAD	100.00	.7907

TABLE N° 2HD7 : FLOTATION TEST N° 7.

PRODUCTS	WEIGHT	Fe203		SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1 1 0 0 0 0 0	25.79	14.30	38.18	8.30	19.46	2.03	34.84	4.81	14.26
2 F1 2 3 4 0 0 0	74.21	8.05	61.82	11.94	80.54	1.32	65.16	10.05	85.74
3 NF2 2 0 0 0 0 0	37.35	8.90	34.41	14.31	48.59	1.90	47.23	6.11	26.24
4 F2 3 4 0 0 0 0	36.86	7.18	27.40	9.54	31.95	.73	17.93	14.04	59.50
5 NF3 3 0 0 0 0 0	20.59	9.26	19.74	12.22	22.87	1.00	13.70	8.40	19.88
6 F3 4 0 0 0 0 0	16.27	4.55	7.66	6.14	9.08	.39	4.22	21.18	39.62

TABLE N° 2H08 : REE ORE SAMPLE N°2 GROUND DOWN TO 0.16mm,-0.05+0.008mm FRACTION,MATERIAL BALANCE OF FLOTATION TEST N°8.

PRODUCTS	WEIGHT	Fe203		SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1	22.40	12.57	32.59	16.39	35.25	1.48	22.50	4.70	10.70
2 NF2	26.94	9.68	30.18	14.77	38.21	2.33	42.61	5.35	14.64
3 NF3	22.68	9.65	25.33	7.89	17.18	1.70	26.17	6.13	14.13
4 F3	17.94	5.17	10.73	4.13	7.11	.62	7.55	9.04	16.48
5 F4	10.05	1.00	1.16	2.32	2.24	.17	1.16	43.14	44.05
CALCULATED HEAD	100.00	8.64	100.00	10.41	100.00	1.47	100.00	9.84	100.00

PRODUCTS	WEIGHT %	REO TOT. GRADE / SrO GRADE
1 NF1	22.40	.2868
2 NF2	26.94	.3622
3 NF3	22.68	.7769
4 F3	17.94	2.1889
5 F4	10.05	18.5948
CALCULATED HEAD	100.00	.9451

TABLE N° 2H08 : FLOTATION TEST N°8.

PRODUCTS	WEIGHT	Fe203		SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1 1 0 0 0 0 0	22.40	12.57	32.59	16.39	35.25	1.48	22.50	4.70	10.70
2 F1 2 3 4 5 0 0	77.60	7.50	67.41	8.69	64.75	1.47	77.50	11.32	89.30
3 NF2 2 0 0 0 0 0	26.94	9.68	30.18	14.77	38.21	2.33	42.61	5.35	14.64
4 F2 3 4 5 0 0 0	50.66	6.35	37.23	5.45	26.54	1.01	34.88	14.50	74.66
5 NF3 3 0 0 0 0 0	22.68	9.65	25.33	7.89	17.18	1.70	26.17	6.13	14.13
6 F3 4 5 0 0 0 0	27.99	3.67	11.90	3.48	9.35	.46	8.71	21.28	60.53
7 F3 4 0 0 0 0 0	17.94	5.17	10.73	4.13	7.11	.62	7.55	9.04	16.48
8 F4 5 0 0 0 0 0	10.05	1.00	1.16	2.32	2.24	.17	1.16	43.14	44.05

TABLE N° 2H09 : REE ORE SAMPLE N°2 GROUND DOWN TO 0.16mm,-0.05+0.008mm FRACTION,MATERIAL BALANCE OF FLOTATION TEST N°9.

PRODUCTS	WEIGHT	Fe203		SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1	15.84	6.13	10.51	20.56	30.03	.76	8.01	10.58	17.72
2 F2	8.80	8.76	8.35	10.02	8.13	.71	4.16	11.97	11.14
3 NF3	32.41	13.75	48.25	12.45	37.20	3.51	75.70	5.86	20.08
4 NF4	23.72	8.93	22.94	7.70	16.84	.59	9.31	8.67	21.75
5 F4	19.23	4.78	9.95	4.40	7.80	.22	2.82	14.41	29.30
CALCULATED HEAD	100.00	9.24	100.00	10.85	100.00	1.50	100.00	9.46	100.00

PRODUCTS	WEIGHT %	REO TOT. GRADE / SrO GRADE
1 F1	15.84	.5146
2 F2	8.80	1.1946
3 NF3	32.41	.4707
4 NF4	23.72	1.1260
5 F4	19.23	3.2750
CALCULATED HEAD	100.00	.8718

TABLE N° 2H09 : FLOTATION TEST N° 9.

PRODUCTS	WEIGHT	Fe203		SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1 1 0 0 0 0 0	15.84	6.13	10.51	20.56	30.03	.76	8.01	10.58	17.72
2 NF1 2 3 4 5 0 0	84.16	9.82	89.49	9.02	69.97	1.64	91.99	9.24	82.28
3 F2 2 0 0 0 0 0	8.80	8.76	8.35	10.02	8.13	.71	4.16	11.97	11.14
4 NF2 3 4 5 0 0 0	75.36	9.94	81.14	8.90	61.84	1.75	87.83	8.93	71.14
5 F3 4 5 0 0 0 0	42.95	7.07	32.89	6.22	24.64	.42	12.13	11.24	51.05
6 NF3 3 0 0 0 0 0	32.41	13.75	48.25	12.45	37.20	3.51	75.70	5.86	20.08
7 F4 5 0 0 0 0 0	19.23	4.78	9.95	4.40	7.80	.22	2.82	14.41	29.30
8 NF4 4 0 0 0 0 0	23.72	8.93	22.94	7.70	16.84	.59	9.31	8.67	21.75

FRACTION,MATERIAL BALANCE OF FLOTATION TEST N°10.

PRODUCTS	WEIGHT %	Fe203		SrO		BaO		REO TOT.	
		GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1	23.17	5.38	13.84	22.01	47.57	2.43	38.27	10.04	23.27
2 F2	15.72	6.98	12.18	13.51	19.81	1.83	19.55	10.93	17.18
3 NF3	12.11	21.52	28.93	5.94	6.71	1.11	9.14	3.59	4.35
4 NF4	10.59	13.00	15.28	9.97	9.85	1.98	14.25	4.17	4.42
5 F4	38.41	6.98	29.76	4.48	16.05	.72	18.80	13.22	50.79
CALCULATED HEAD	100.00	9.01	100.00	10.72	100.00	1.47	100.00	10.00	100.00

PRODUCTS	WEIGHT %	REO TOT. GRADE / SrO GRADE
1 F1	23.17	.4562
2 F2	15.72	.8090
3 NF3	12.11	.6044
4 NF4	10.59	.4183
5 F4	38.41	2.9509
CALCULATED HEAD	100.00	.9328

TABLE N° 2H10 : FLOTATION TEST N° 10.

PRODUCTS	WEIGHT %	Fe203		SrO		BaO		REO TOT.	
		GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1 1 0 0 0 0 0	23.17	5.38	13.84	22.01	47.57	2.43	38.27	10.04	23.27
2 NF1 2 3 4 5 0 0	76.83	10.10	86.16	7.31	52.43	1.18	61.73	9.99	76.73
3 F2 2 0 0 0 0 0	15.72	6.98	12.18	13.51	19.81	1.83	19.55	10.93	17.18
4 NF2 3 4 5 0 0 0	61.11	10.90	73.98	5.72	32.61	1.02	42.18	9.74	59.55
5 F3 4 5 0 0 0 0	49.00	8.28	45.05	5.67	25.90	.99	33.05	11.26	55.20
6 NF3 3 0 0 0 0 0	12.11	21.52	28.93	5.94	6.71	1.11	9.14	3.59	4.35
7 F4 5 0 0 0 0 0	38.41	6.98	29.76	4.48	16.05	.72	18.80	13.22	50.79
8 NF4 4 0 0 0 0 0	10.59	13.00	15.28	9.97	9.85	1.98	14.25	4.17	4.42

TABLE N° 2H11 : REE ORE SAMPLE N°2 GROUND DOWN TO 0.16mm,-0.05+0.008mm FRACTION,MATERIAL BALANCE OF FLOTATION TEST N°11.

PRODUCTS	WEIGHT	Fe2O3		SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1	17.80	11.90	26.87	7.60	12.92	2.30	28.30	4.16	8.48
2 NF2	46.75	7.50	44.49	16.60	74.11	1.80	58.18	6.39	34.22
3 NF3	17.23	7.75	16.94	5.25	8.64	.90	10.72	7.63	15.06
4 NF4	11.10	6.35	8.94	2.80	2.97	.30	2.30	8.06	10.25
5 F4	7.12	3.05	2.76	2.00	1.36	.10	.49	39.23	31.99
CALCULATED HEAD	100.00	7.88	100.00	10.47	100.00	1.45	100.00	8.73	100.00

PRODUCTS	WEIGHT	La2O3		CeO2	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1	17.80	1.22	8.49	2.11	8.49
2 NF2	46.75	1.86	34.00	3.25	34.33
3 NF3	17.23	2.23	15.02	3.87	15.07
4 NF4	11.10	2.34	10.16	4.11	10.31
5 F4	7.12	11.61	32.32	19.77	31.81
CALCULATED HEAD	100.00	2.56	100.00	4.43	100.00

PRODUCTS	WEIGHT %	REO TOT. GRADE / SrO GRADE
1 NF1	17.80	.5474
2 NF2	46.75	.3849
3 NF3	17.23	1.4533
4 NF4	11.10	2.8786
5 F4	7.12	19.6150
CALCULATED HEAD	100.00	.8338

TABLE N° 2H11 : FLOTATION TEST N°11.

PRODUCTS	WEIGHT	Fe2O3		SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1 1 0 0 0 0 0	17.80	11.90	26.87	7.60	12.92	2.30	28.30	4.16	8.48
2 F1 2 3 4 5 0 0	82.20	7.01	73.13	11.09	87.08	1.26	71.70	9.72	91.52
3 NF2 2 0 0 0 0 0	46.75	7.50	44.49	16.60	74.11	1.80	58.18	6.39	34.22
4 F2 2 4 5 0 0 0	64.97	6.82	56.18	12.64	78.44	1.36	60.97	10.27	76.46
5 NF3 3 0 0 0 0 0	17.23	7.75	16.94	5.25	8.64	.90	10.72	7.63	15.06
6 F3 4 5 0 0 0 0	18.22	5.06	11.70	2.49	4.33	.22	2.79	20.24	42.24
7 NF4 4 0 0 0 0 0	11.10	6.35	8.94	2.80	2.97	.30	2.30	8.06	10.25
8 F4 5 0 0 0 0 0	7.12	3.05	2.76	2.00	1.36	.10	.49	39.23	31.99

PRODUCTS	WEIGHT	La2O3		CeO2	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1 1 0 0 0 0 0	17.80	1.22	8.49	2.11	8.49
2 F1 2 3 4 5 0 0	82.20	2.85	91.51	4.93	91.51
3 NF2 2 0 0 0 0 0	46.75	1.86	34.00	3.25	34.33
4 F2 2 4 5 0 0 0	64.97	3.01	76.48	5.21	76.45
5 NF3 3 0 0 0 0 0	17.23	2.23	15.02	3.87	15.07
6 F3 4 5 0 0 0 0	18.22	5.96	42.48	10.23	42.11
7 NF4 4 0 0 0 0 0	11.10	2.34	10.16	4.11	10.31
8 F4 5 0 0 0 0 0	7.12	11.61	32.32	19.77	31.81

TABLE N° 2H12 : REE ORE SAMPLE N°2 GROUND DOWN TO 0.16mm,-0.05+0.008mm FRACTION,MATERIAL BALANCE OF FLOTATION TEST N°12.

PRODUCTS	WEIGHT	Fe2O3		SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1	15.03	15.58	29.89	8.12	11.51	2.38	24.75	3.84	6.03
2 NF2	40.34	7.93	40.83	18.05	68.65	1.79	49.96	5.71	24.07
3 NF3	15.22	9.02	17.52	8.29	11.90	1.59	16.74	7.26	11.55
4 NF4	10.97	6.71	9.40	4.00	4.14	.74	5.62	5.96	6.83
5 F4	18.44	1.00	2.35	2.19	3.81	.23	2.93	26.73	51.51
CALCULATED HEAD	100.00	7.83	100.00	10.61	100.00	1.45	100.00	9.57	100.00

PRODUCTS	WEIGHT %	REO TOT. GRADE / SrO GRADE
1 NF1	15.03	.4729
2 NF2	40.34	.3163
3 NF3	15.22	.8758
4 NF4	10.97	1.4900
5 F4	18.44	12.2055
CALCULATED HEAD	100.00	.9021

TABLE N° 2H12 : FLOTATION TEST N°12.

PRODUCTS	WEIGHT	Fe2O3		SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1 1 0 0 0 0 0	15.03	15.58	29.89	8.12	11.51	2.38	24.75	3.84	6.03
2 F1 2 3 4 5 0 0	84.97	6.46	70.11	11.05	88.49	1.28	75.25	10.58	93.97
3 NF2 2 0 0 0 0 0	40.34	7.93	40.83	18.05	68.65	1.79	49.96	5.71	24.07
4 F2 3 4 5 0 0 0	44.63	5.14	29.27	4.72	19.84	.82	25.29	14.98	69.89
5 NF3 3 0 0 0 0 0	15.22	9.02	17.52	8.29	11.90	1.59	16.74	7.26	11.55
6 F3 4 5 0 0 0 0	29.41	3.13	11.75	2.87	7.94	.42	8.55	18.98	58.35
7 NF4 4 0 0 0 0 0	10.97	6.71	9.40	4.00	4.14	.74	5.62	5.96	6.83
8 F4 5 0 0 0 0 0	18.44	1.00	2.35	2.19	3.81	.23	2.93	26.73	51.51

3.2.3. Flotation of the - 80 + 10 µm gravity preconcentrate

Removal of Fe-Mn containing dolomite by gravity separation leads to low REO losses in light fractions and is expected to promote monazite separation by flotation from the heavy fraction since Fe associated to light carbonates detrimentally affects selectivity of phosphoric esters for monazite.

Operating conditions and flowsheets of flotation tests, conducted in Minemet flotation cells of 2.5 l and 0.8 l capacity, are shown in figures n° 2F1 to 2F16. Relevant material balances are given in tables 2F1 to 2F16. Tests were made on the gravity preconcentrate n° 1 with a moderate content in iron bearing carbonates.

Results of tests reveal that:

- addition of H_2SO_4 and phosphoric ester P301, at a pulp pH of 4.5 results in strong depression of barite while strontianite is slightly depressed (test 1) and affinity of P.E. for monazite is maximum. Comparison with tests 2 to 4 clearly indicates that pH value and nature of the anion control selectivity of P.E. for monazite:

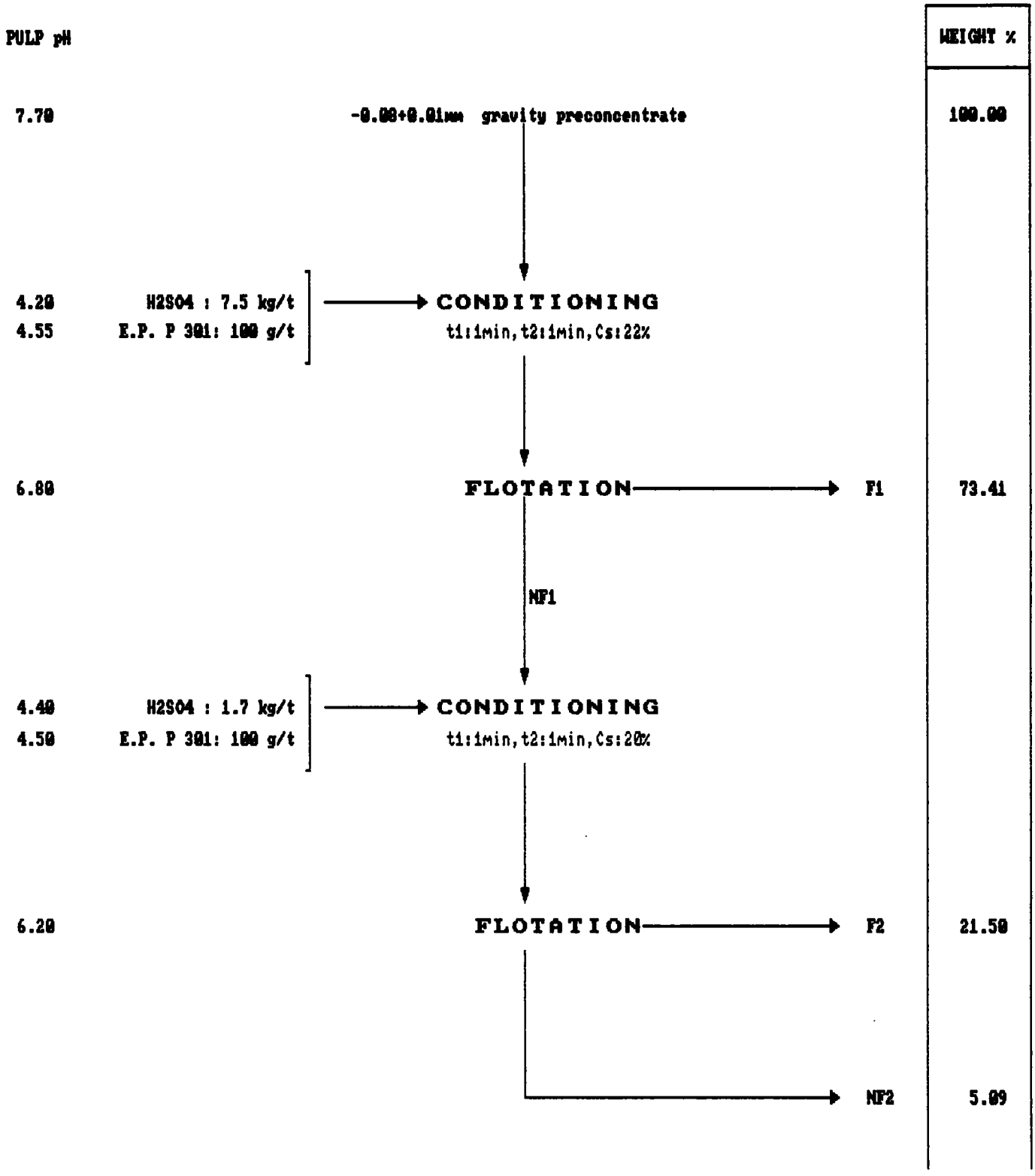


FIGURE N° 2F1 : FLOWSHEET FOR FLOTATION TEST N° 1

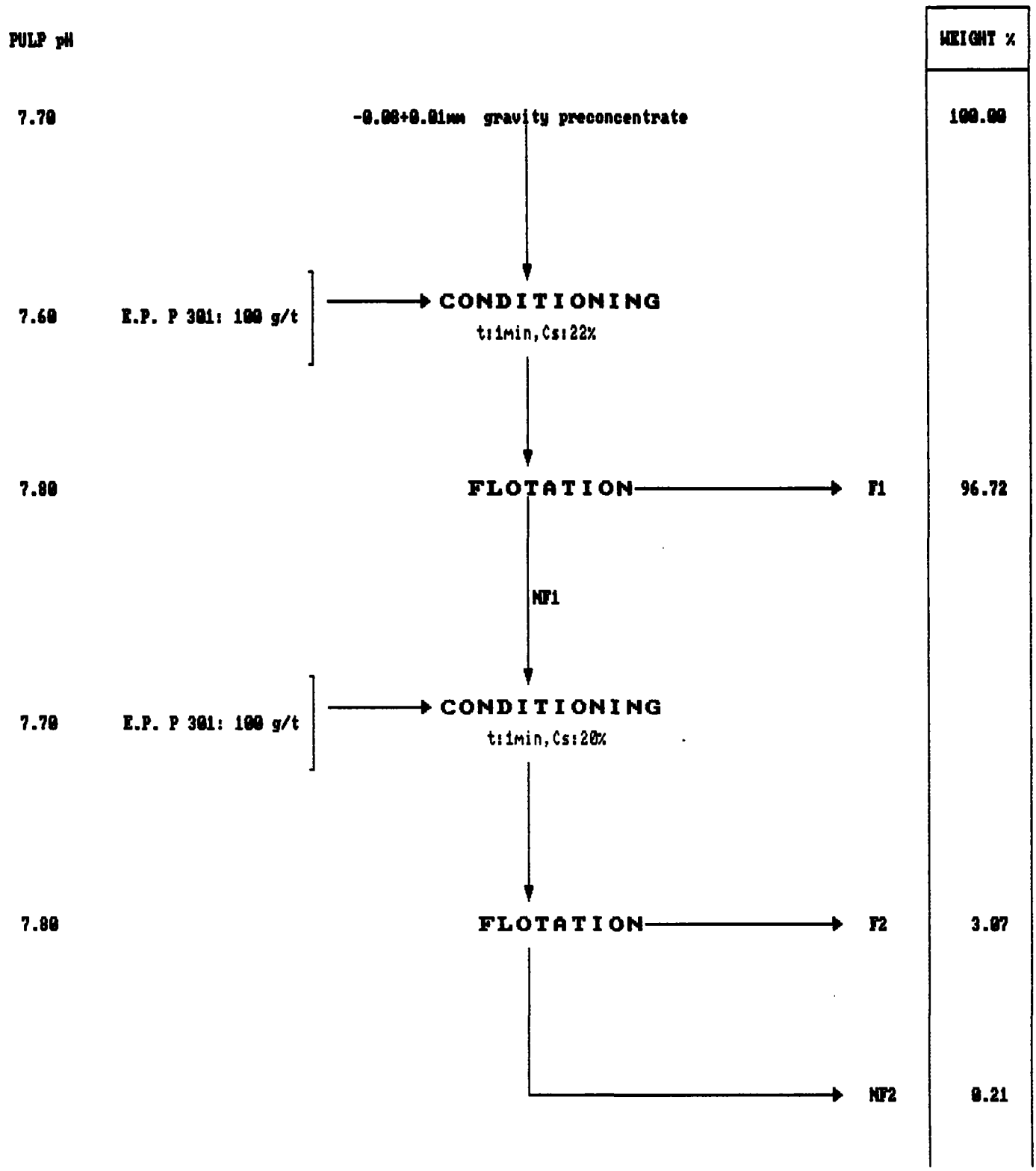


FIGURE N° 2F2 : FLOWSHEET FOR FLOTATION TEST N° 2

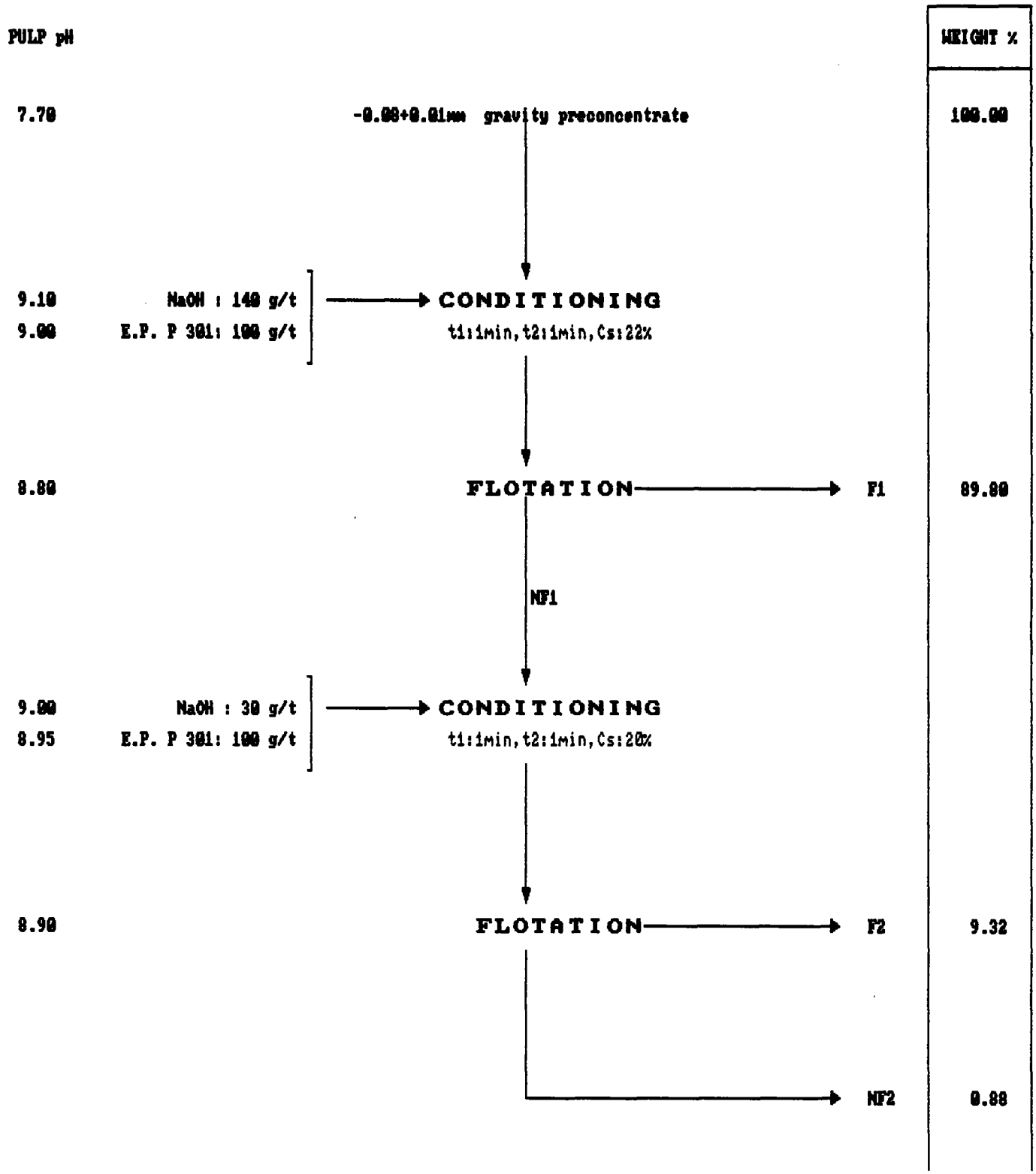


FIGURE N° 2F3 : FLOWSHEET FOR FLOTATION TEST N° 3

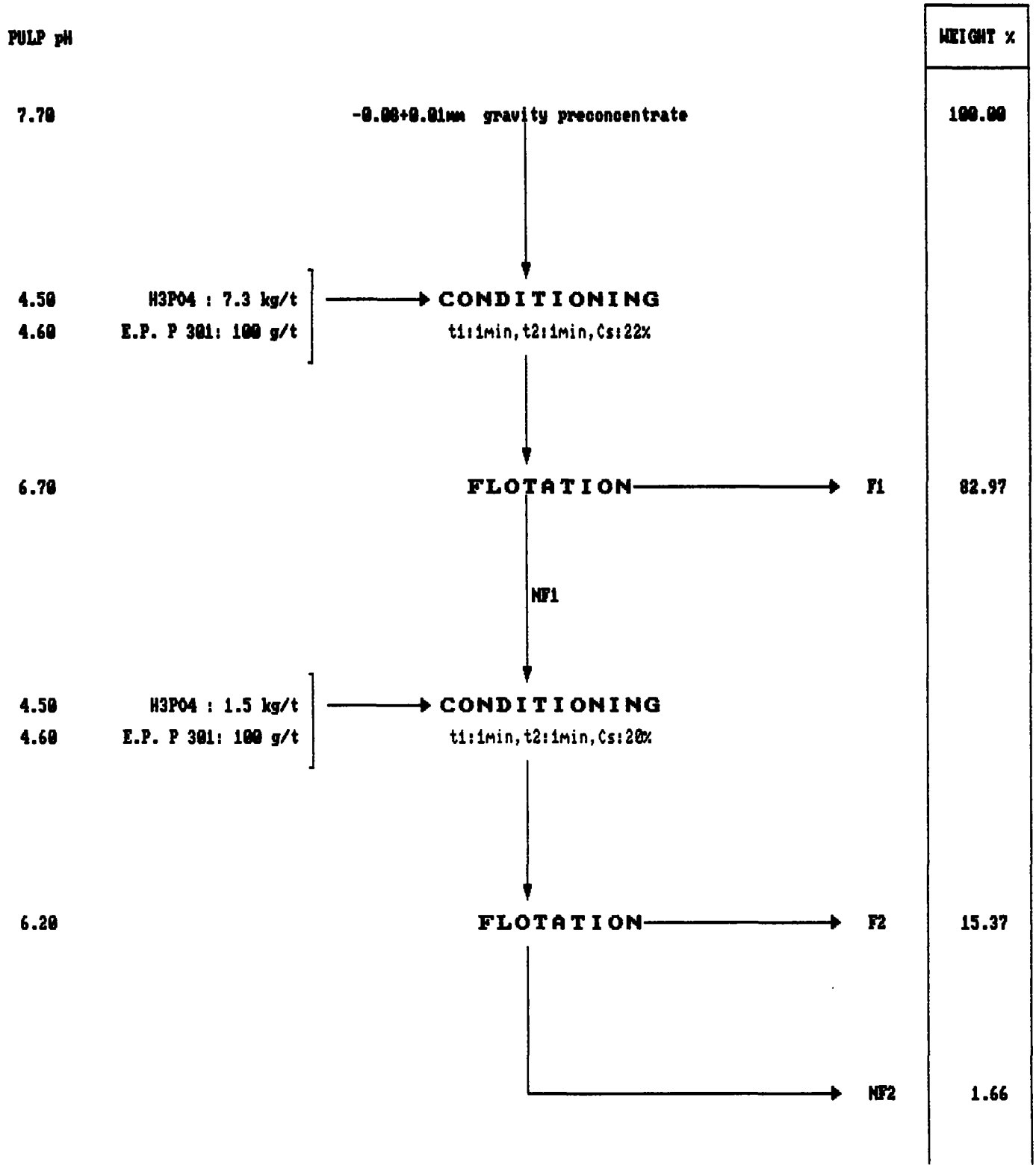


FIGURE N° 2F4 : FLOWSHEET FOR FLOTATION TEST N° 4

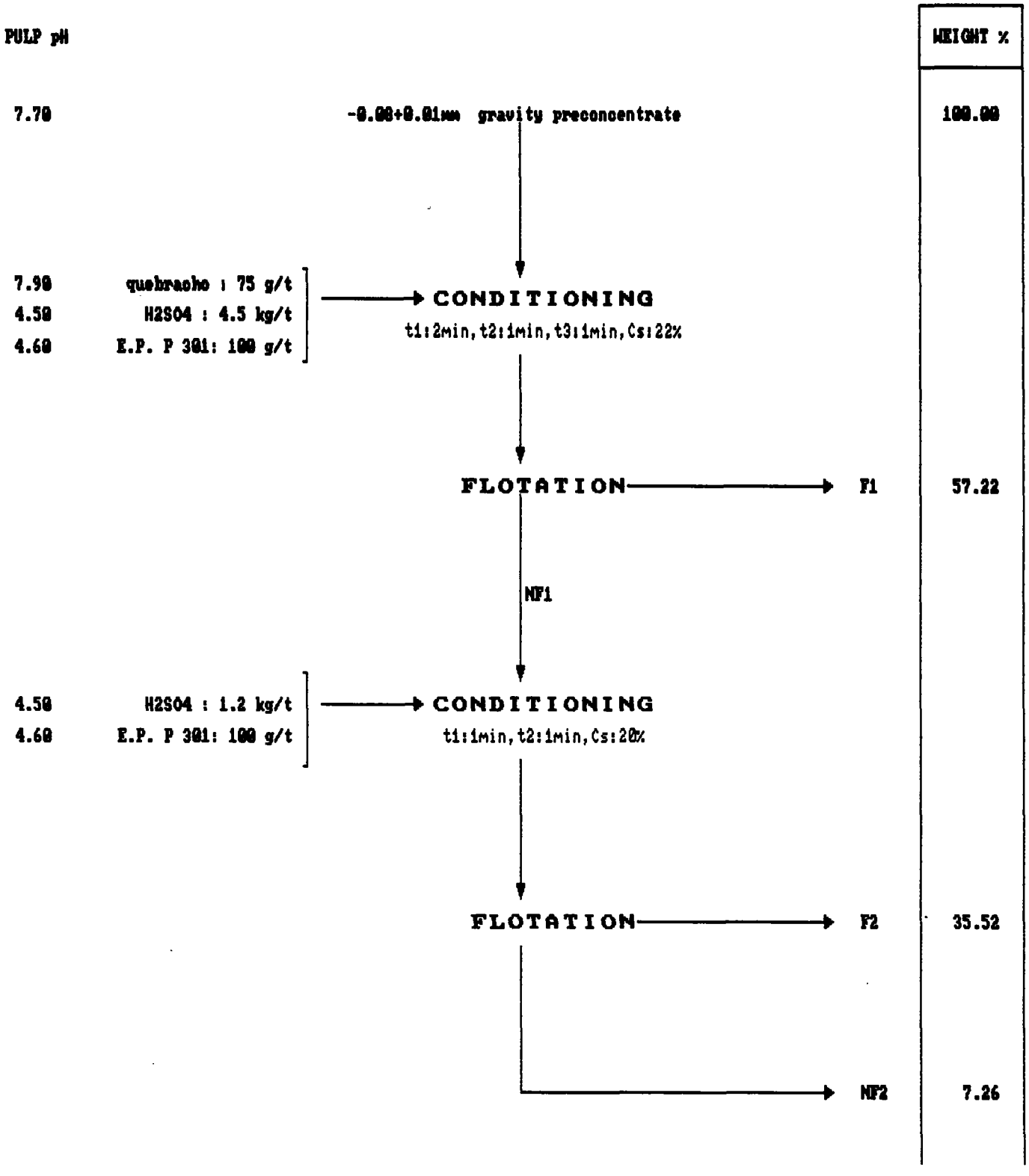


FIGURE N° 2F5 : FLOWSHEET FOR FLOTATION TEST N° 5

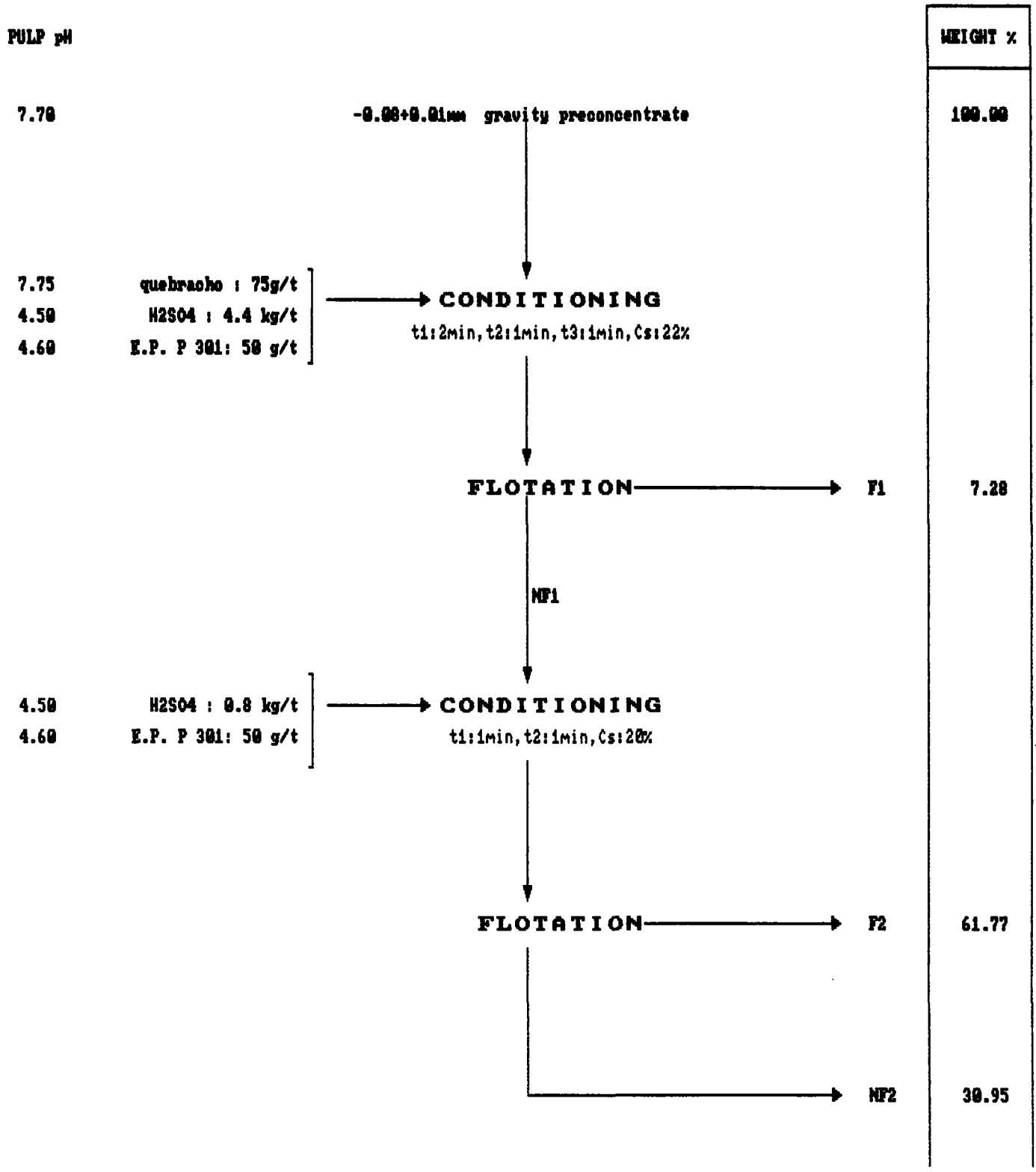


FIGURE N° 2F9 : FLOWSHEET FOR FLOTATION TEST N° 9

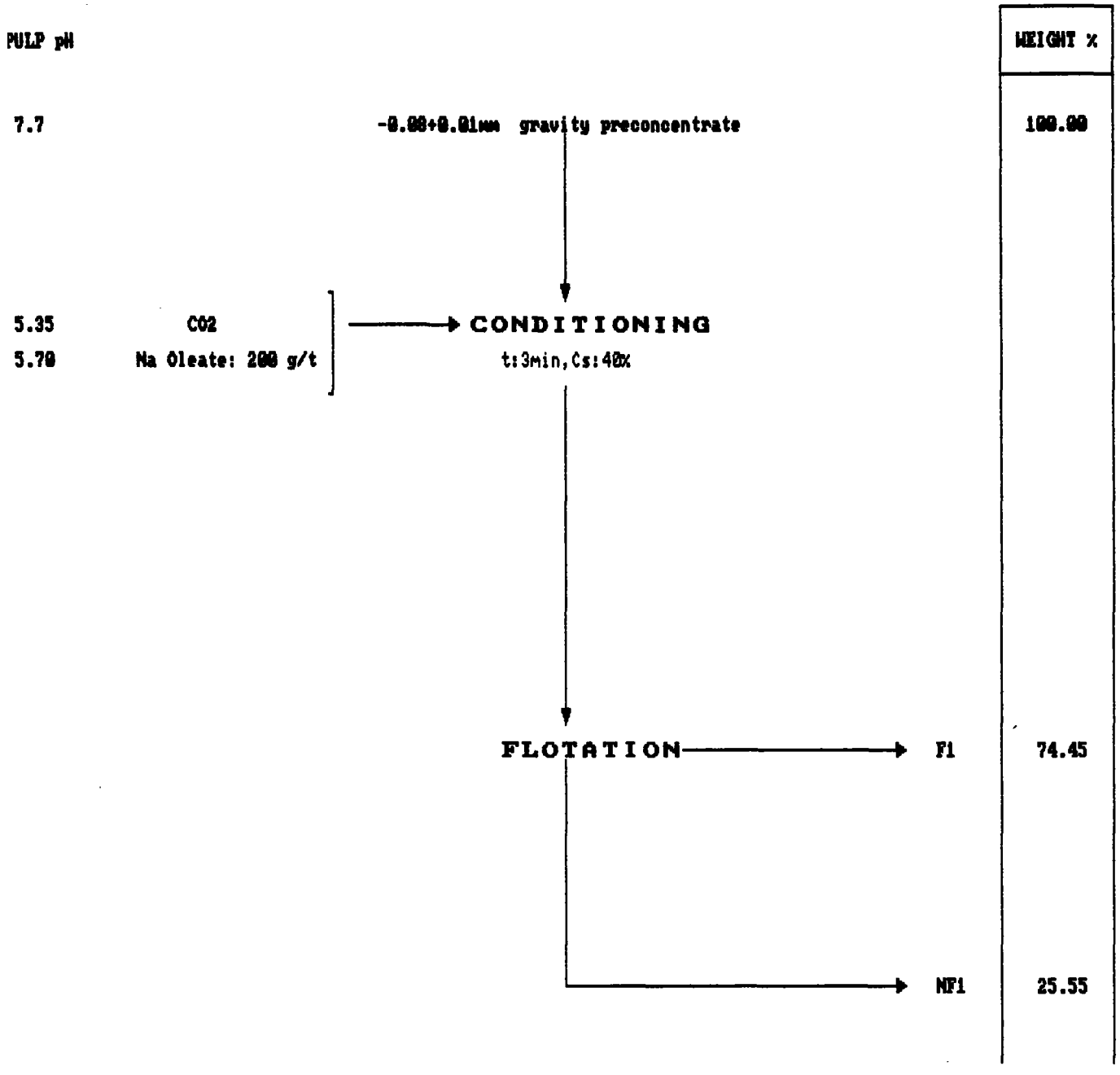


FIGURE N° 2F10 : FLOWSHEET FOR FLOTATION TEST N° 10

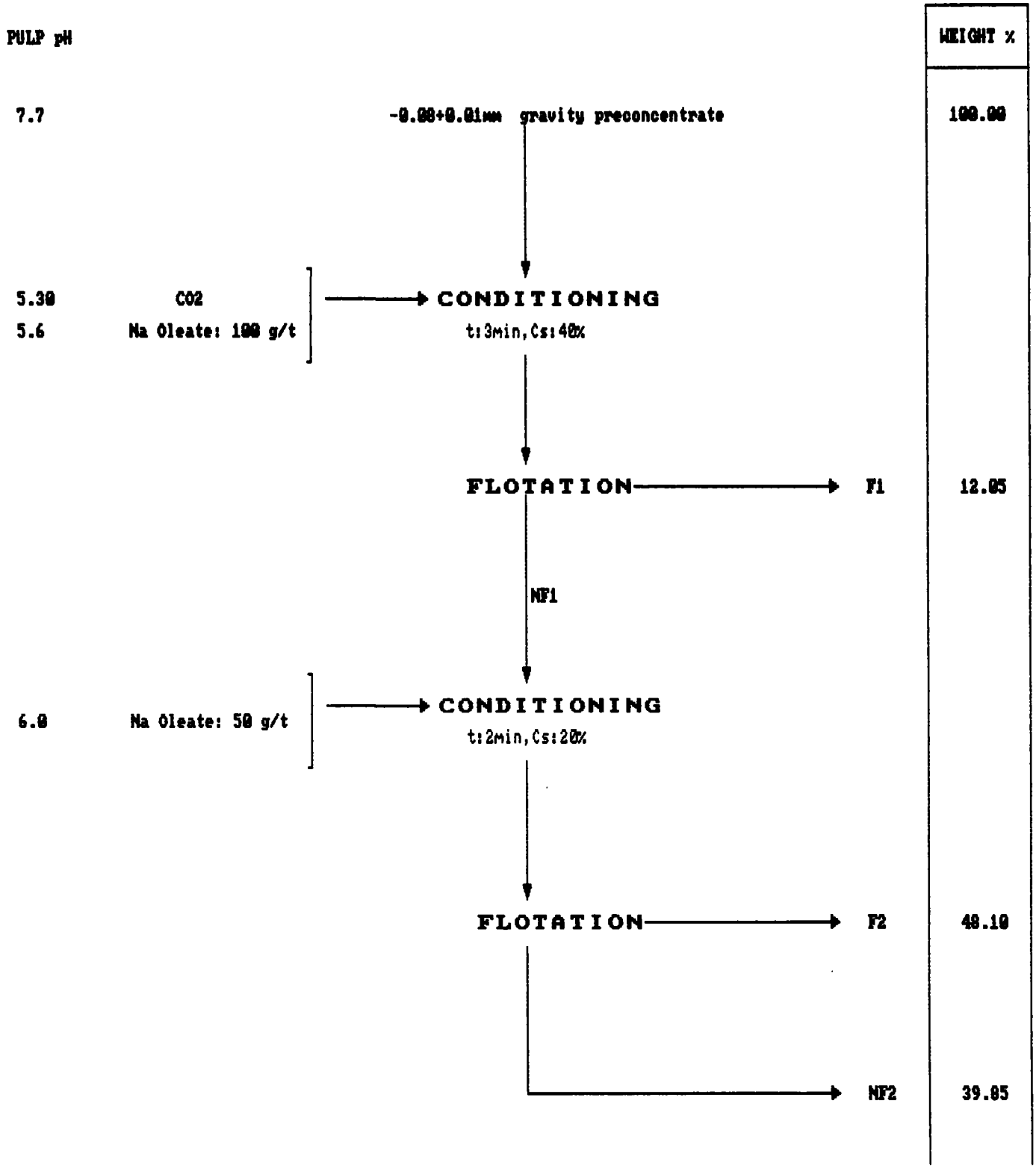


FIGURE N° 2F11 : FLOWSHEET FOR FLOTATION TEST N° 11

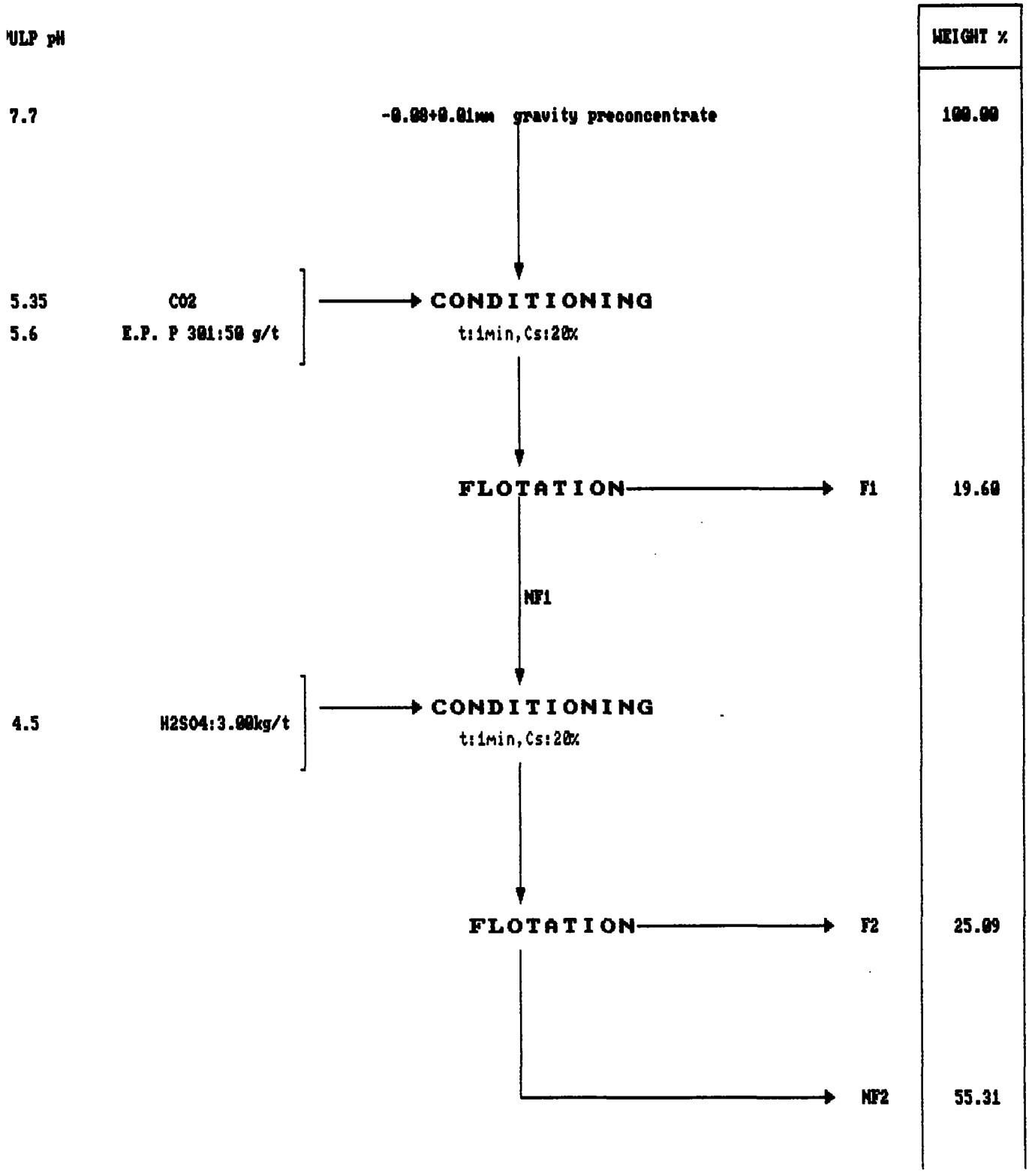


FIGURE N° 2F13 : FLOWSHEET FOR FLOTATION TEST N° 13

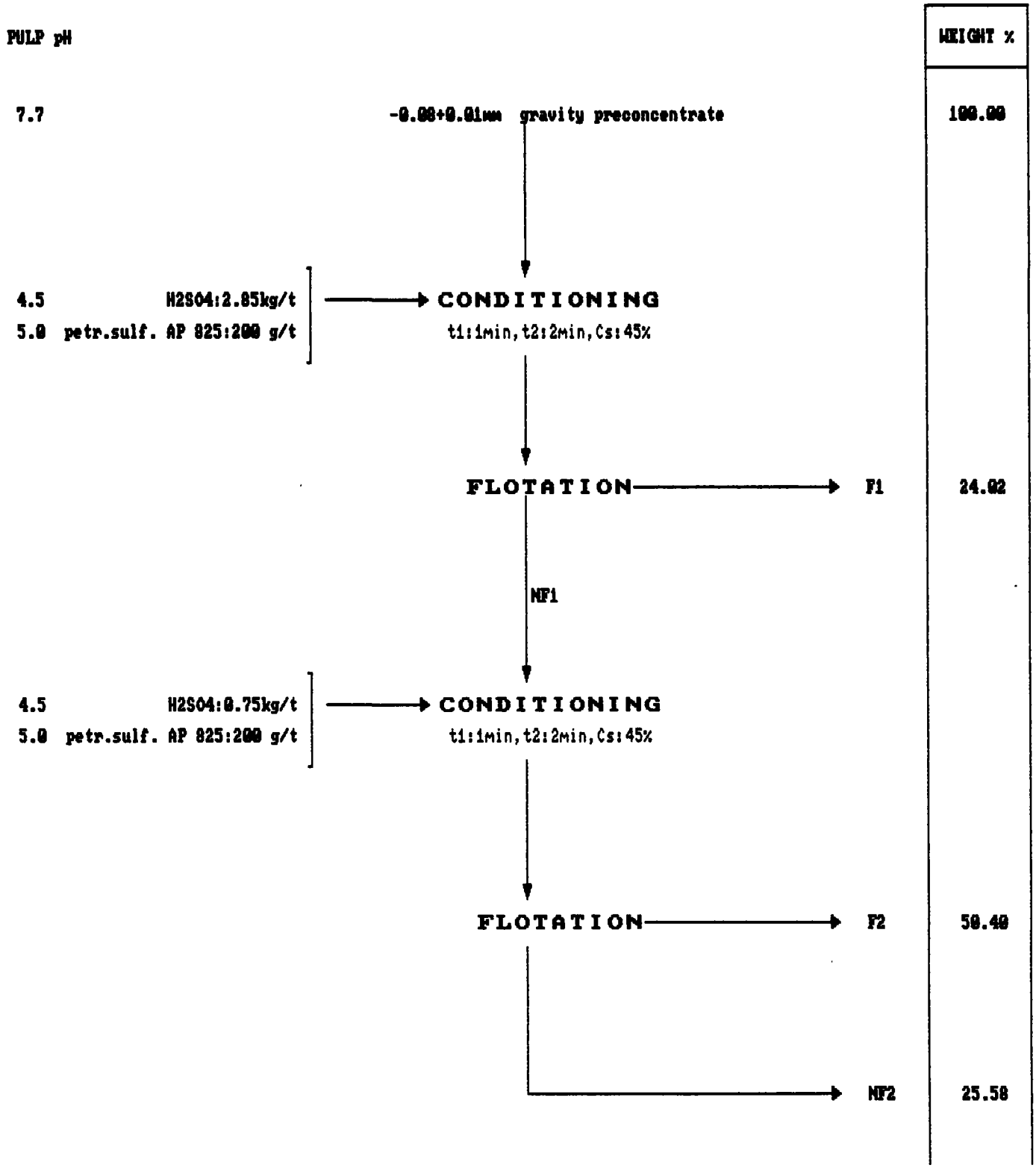


FIGURE N° 2F15 : FLOWSHEET FOR FLOTATION TEST N° 15

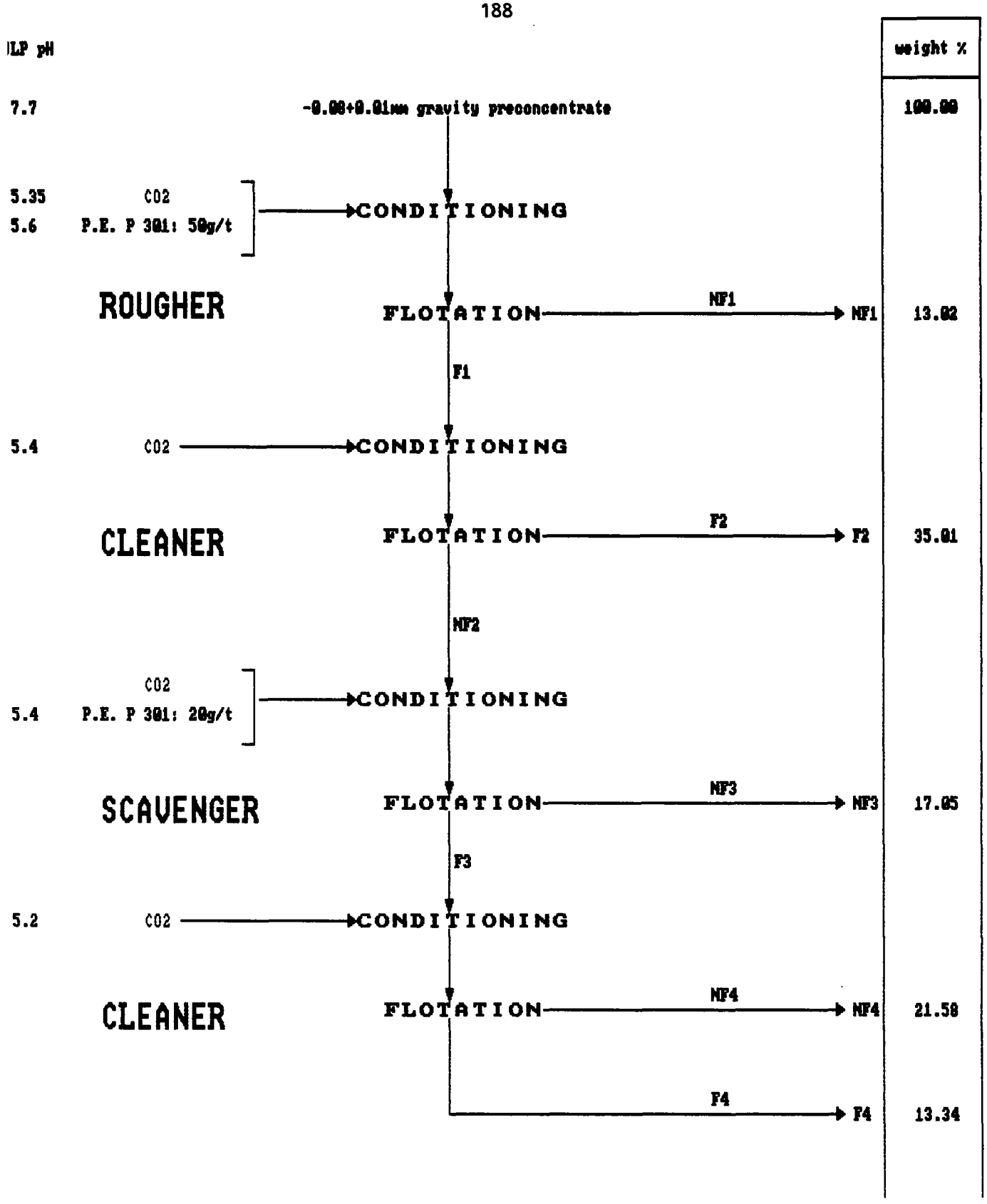


FIGURE N° 2F16 : FLOWSHEET FOR FLOTATION TEST N° 16

TABLE N° 2F1 : REO ORE SAMPLE N°2,-0.08+0.01mm GRAVITY PRECONCENTRATE, MATERIAL BALANCE OF FLOTATION TEST N° 1.

PRODUCTS	WEIGHT	SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1	73.41	10.52	63.83	.75	9.83	57.11	90.79
2 NF1	26.59	16.46	36.17	19.00	90.17	16.00	9.21
CALCULATED HEAD	100.00	12.10	100.00	5.60	100.00	46.18	100.00

PRODUCTS	WEIGHT %	REO TOT. GRADE / SrO GRADE
1 F1	73.41	5.4287
2 NF1	26.59	.9721
CALCULATED HEAD	100.00	3.8166

TABLE N° 2F2 : REO ORE SAMPLE N°2,-0,08+0.01mm GRAVITY PRECONCENTRATE, MATERIAL BALANCE OF FLOTATION TEST N° 2.

PRODUCTS	WEIGHT	SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1	96.76	12.28	96.84	5.20	95.45	42.53	96.72
2 NF1	3.24	11.95	3.16	7.40	4.55	43.10	3.28
CALCULATED HEAD	100.00	12.27	100.00	5.27	100.00	42.55	100.00

PRODUCTS	WEIGHT %	REO TOT. GRADE / SrO GRADE
1 F1	96.76	3.4634
2 NF1	3.24	3.6067
CALCULATED HEAD	100.00	3.4679

TABLE N° 2F3 : REO ORE SAMPLE N°2,-0,08+0.01mm GRAVITY PRECONCENTRATE, MATERIAL BALANCE OF FLOTATION TEST N° 3.

PRODUCTS	WEIGHT	SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1	89.80	12.96	91.13	5.38	88.59	45.76	90.33
2 NF1	10.20	11.10	8.87	6.10	11.41	43.15	9.67
CALCULATED HEAD	100.00	12.77	100.00	5.45	100.00	45.49	100.00

PRODUCTS	WEIGHT %	REO TOT. GRADE / SrO GRADE
1 F1	89.80	3.5309
2 NF1	10.20	3.8874
CALCULATED HEAD	100.00	3.5625

TABLE N° 2F4 : REO ORE SAMPLE N°2,-0.08+0.01mm GRAVITY PRECONCENTRATE, MATERIAL BALANCE OF FLOTATION TEST N° 4.

PRODUCTS	WEIGHT	SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1	82.97	11.81	80.64	5.61	81.63	43.50	82.81
2 NF1	17.03	13.81	19.36	6.15	18.37	44.00	17.19
CALCULATED HEAD	100.00	12.15	100.00	5.70	100.00	43.59	100.00

PRODUCTS	WEIGHT %	REO TOT. GRADE / SrO GRADE
1 F1	82.97	3.6833
2 NF1	17.03	3.1861
CALCULATED HEAD	100.00	3.5871

TABLE N° 2F5 : RED ORE SAMPLE N°2,-0.08+0.01mm GRAVITY PRECONCENTRATE, MATERIAL BALANCE OF FLOTATION TEST N°5.

PRODUCTS	WEIGHT	Fe2O3		SrO		BaO		La2O3	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1	57.22	1.50	41.30	10.50	50.25	.65	6.65	15.01	68.49
2 F2	35.52	3.25	55.55	16.05	47.68	3.10	19.70	11.02	31.21
3 NF2	7.26	.90	3.14	3.40	2.06	56.70	73.65	.51	.30
CALCULATED HEAD	100.00	2.08	100.00	11.96	100.00	5.59	100.00	12.54	100.00

PRODUCTS	WEIGHT	Ce2O3		RED TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1	57.22	26.41	68.20	51.78	68.30
2 F2	35.52	19.65	31.50	38.34	31.40
3 NF2	7.26	.92	.30	1.79	.30
CALCULATED HEAD	100.00	22.16	100.00	43.38	100.00

PRODUCTS	WEIGHT %	RED TOT. GRADE / SrO GRADE
1 F1	57.22	4.9314
2 F2	35.52	2.3888
3 NF2	7.26	.5265
CALCULATED HEAD	100.00	3.6281

TABLE N°2F5 : FLOTATION TEST N°5

PRODUCTS	WEIGHT	Fe203		SrO		BaO		La203	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1 1 0 0 0 0 0	57.22	1.50	41.30	10.50	50.25	.65	6.65	15.01	68.49
2 NF1 2 3 0 0 0 0	42.78	2.85	58.70	13.90	49.75	12.20	93.35	9.24	31.51
3 F1+F2 1 2 0 0 0 0	92.74	2.17	96.86	12.63	97.94	1.59	26.35	13.48	99.70
4 NF2 3 0 0 0 0 0	7.26	.90	3.14	3.40	2.06	56.70	73.65	.51	.30

PRODUCTS	WEIGHT	Ce203		RED TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1 1 0 0 0 0 0	57.22	26.41	68.20	51.78	68.30
2 NF1 2 3 0 0 0 0	42.78	16.47	31.80	32.14	31.70
3 F1+F2 1 2 0 0 0 0	92.74	23.82	99.70	46.63	99.70
4 NF2 3 0 0 0 0 0	7.26	.92	.30	1.79	.30

TABLE N° 2F9 : REO ORE SAMPLE N°2,-0.08+0.01mm GRAVITY PRECONCENTRATE, MATERIAL BALANCE OF FLOTATION TEST N° 9.

PRODUCTS	WEIGHT	SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1	7.28	12.83	8.76	.56	.80	48.80	7.45
2 F2	61.77	10.93	63.34	.82	9.90	56.30	72.94
3 NF2	30.95	9.61	27.90	14.77	89.31	30.21	19.61
CALCULATED HEAD	100.00	10.66	100.00	5.12	100.00	47.68	100.00

PRODUCTS	WEIGHT %	REO TOT. GRADE / SrO GRADE
1 F1	7.28	3.8036
2 F2	61.77	5.1510
3 NF2	30.95	3.1436
CALCULATED HEAD	100.00	4.4728

TABLE N° 2F9 : FLOTATION TEST N°9.

PRODUCTS	WEIGHT	SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1 1 0 0 0 0 0	7.28	12.83	8.76	.56	.80	48.80	7.45
2 NF1 2 3 0 0 0 0	92.72	10.49	91.24	5.48	99.20	47.59	92.55
3 F1+F2 1 2 0 0 0 0	69.05	11.13	72.10	.79	10.69	55.51	80.39
4 NF2 3 0 0 0 0 0	30.95	9.61	27.90	14.77	89.31	30.21	19.61

TABLE N° 2F10: RED ORE SAMPLE N°2,-0.08+0.01mm GRAVITY PRECONCENTRATE, MATERIAL BALANCE OF FLOTATION TEST N°10.

PRODUCTS	WEIGHT	SrO		BaO		RED TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1	74.45	13.51	86.49	3.98	54.89	48.68	78.56
2 NF1	25.55	6.15	13.51	9.53	45.11	38.71	21.44
CALCULATED HEAD	100.00	11.63	100.00	5.40	100.00	46.13	100.00

PRODUCTS	WEIGHT %	RED TOT. GRADE / SrO GRADE
1 F1	74.45	3.6033
2 NF1	25.55	6.2943
CALCULATED HEAD	100.00	3.9669

TABLE N° 2F11: RED ORE SAMPLE N°2,-0.08+0.01mm GRAVITY PRECONCENTRATE, MATERIAL BALANCE OF FLOTATION TEST N°11.

PRODUCTS	WEIGHT	SrO		BaO		RED TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1	12.05	10.53	11.90	2.83	6.88	38.06	10.64
2 F2	48.10	13.36	60.28	1.81	17.56	46.64	52.05
3 NF2	39.85	7.44	27.81	9.40	75.56	40.35	37.31
CALCULATED HEAD	100.00	10.66	100.00	4.96	100.00	43.10	100.00

PRODUCTS	WEIGHT %	RED TOT. GRADE / SrO GRADE
1 F1	12.05	3.6144
2 F2	48.10	3.4910
3 NF2	39.85	5.4234
CALCULATED HEAD	100.00	4.0432

TABLE N° 2F11 : FLOTATION TEST N° 11.

PRODUCTS	WEIGHT	SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1 1 0 0 0 0 0	12.05	10.53	11.90	2.83	6.88	38.06	10.64
2 NF1 2 3 0 0 0 0	87.95	10.68	88.10	5.25	93.12	43.79	89.36
3 F1+F2 1 2 0 0 0 0	60.15	12.79	72.19	2.01	24.44	44.92	62.69
4 NF2 3 0 0 0 0 0	39.85	7.44	27.81	9.40	75.56	40.35	37.31

TABLE N° 2F13: RED ORE SAMPLE N°2,-0.08+0.01mm GRAVITY PRECONCENTRATE, MATERIAL BALANCE OF FLOTATION TEST N°13.

PRODUCTS	WEIGHT	Fe2O3		SrO		BaO		La2O3	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1	19.60	3.85	37.80	11.55	19.01	14.70	51.12	10.79	16.19
2 NF2	55.31	1.95	54.03	15.80	73.40	4.80	47.10	11.38	48.17
3 F2	25.09	.65	8.17	3.60	7.59	.40	1.78	18.56	35.64
CALCULATED HEAD	100.00	2.00	100.00	11.91	100.00	5.64	100.00	13.07	100.00

PRODUCTS	WEIGHT	Ce2O3		RED TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1	19.60	17.56	14.88	35.44	15.35
2 NF2	55.31	20.51	49.03	39.86	48.72
3 F2	25.09	33.28	36.09	64.80	35.93
CALCULATED HEAD	100.00	23.14	100.00	45.25	100.00

PRODUCTS	WEIGHT %	RED TOT. GRADE / SrO GRADE
1 NF1	19.60	3.0684
2 NF2	55.31	2.5228
3 F2	25.09	18.0000
CALCULATED HEAD	100.00	3.8007

TABLE N° 2F13 : FLOTATION TEST N° 13.

PRODUCTS	WEIGHT	Fe203		SrO		BaO		La203	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1 1 0 0 0 0 0	19.60	3.85	37.80	11.55	19.01	14.70	51.12	10.79	16.19
2 F1 2 3 0 0 0 0	80.40	1.54	62.20	11.99	80.99	3.43	48.88	13.62	83.81
3 NF1+NF2 1 2 0 0 0 0	74.91	2.45	91.83	14.69	92.41	7.39	98.22	11.23	64.36
4 F2 3 0 0 0 0 0	25.09	.65	8.17	3.60	7.59	.40	1.78	18.56	35.64

PRODUCTS	WEIGHT	Ce203		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1 1 0 0 0 0 0	19.60	17.56	14.88	35.44	15.35
2 F1 2 3 0 0 0 0	80.40	24.50	85.12	47.64	84.65
3 NF1+NF2 1 2 0 0 0 0	74.91	19.74	63.91	38.70	64.07
4 F2 3 0 0 0 0 0	25.09	33.28	36.09	64.80	35.93

TABLE N° 2F15: REO ORE SAMPLE N°2,-0.08+0.01mm GRAVITY PRECONCENTRATE, MATERIAL BALANCE OF FLOTATION TEST N°15.

PRODUCTS	WEIGHT	Fe2O3		SrO		BaO		La2O3	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1	24.02	.30	3.45	31.80	66.09	11.10	48.24	7.27	12.61
2 F2	50.40	.90	21.74	6.10	26.60	3.90	35.56	17.59	64.00
3 NF2	25.58	6.10	74.80	3.30	7.30	3.50	16.20	12.67	23.40
CALCULATED HEAD	100.00	2.09	100.00	11.56	100.00	5.53	100.00	13.85	100.00

PRODUCTS	WEIGHT	Ce2O3		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1	24.02	12.90	12.53	25.21	12.56
2 F2	50.40	31.20	63.59	60.99	63.74
3 NF2	25.58	23.09	23.88	44.70	23.71
CALCULATED HEAD	100.00	24.73	100.00	48.23	100.00

PRODUCTS	WEIGHT %	REO TOT. GRADE / SrO GRADE
1 F1	24.02	.7928
2 F2	50.40	9.9984
3 NF2	25.58	13.5455
CALCULATED HEAD	100.00	4.1731

TABLE N° 2F15 : FLOTATION TEST N°15.

PRODUCTS	WEIGHT	Fe203		SrO		BaO		La203	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1 1 0 0 0 0 0	24.02	.30	3.45	31.80	66.09	11.10	48.24	7.27	12.61
2 NF1 2 3 0 0 0 0	75.98	2.65	96.55	5.16	33.91	3.77	51.76	15.93	87.39
3 F1+F2 1 2 0 0 0 0	74.42	.71	25.20	14.40	92.70	6.22	83.80	14.26	76.60
4 NF2 3 0 0 0 0 0	25.58	6.10	74.80	3.30	7.30	3.50	16.20	12.67	23.40

PRODUCTS	WEIGHT	Ce203		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1 1 0 0 0 0 0	24.02	12.90	12.53	25.21	12.56
2 NF1 2 3 0 0 0 0	75.98	28.47	87.47	55.51	87.44
3 F1+F2 1 2 0 0 0 0	74.42	25.29	76.12	49.44	76.29
4 NF2 3 0 0 0 0 0	25.58	23.09	23.88	44.70	23.71

TABLE N° 2F16 : REE ORE SAMPLE N°2,-0.08+0.01mm GRAVITY PRECONCENTRATE, MATERIAL BALANCE OF FLOTATION TEST N°16.

PRODUCTS	WEIGHT	Fe2O3		SrO		BaO		La2O3	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1	13.02	5.05	31.94	12.00	12.35	15.50	35.00	8.56	8.22
2 F2	35.01	.53	9.01	3.15	8.72	.25	1.52	19.23	49.66
3 NF3	17.05	3.10	25.68	28.25	38.09	16.50	48.79	5.04	6.34
4 NF4	21.58	2.75	28.83	22.20	37.88	3.80	14.22	10.44	16.62
5 F4	13.34	.70	4.54	2.80	2.95	.20	.46	19.47	19.16
CALCULATED HEAD	100.00	2.06	100.00	12.65	100.00	5.77	100.00	13.56	100.00

PRODUCTS	WEIGHT	Ce2O3		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1	13.02	14.98	8.47	29.42	8.37
2 F2	35.01	32.18	48.90	64.26	49.18
3 NF3	17.05	8.84	6.54	17.35	6.47
4 NF4	21.58	18.18	17.03	35.77	16.88
5 F4	13.34	32.92	19.06	65.49	19.10
CALCULATED HEAD	100.00	23.04	100.00	45.74	100.00

PRODUCTS	WEIGHT %	REO TOT. GRADE / SrO GRADE
1 NF1	13.02	2.4517
2 F2	35.01	20.4000
3 NF3	17.05	.6142
4 NF4	21.58	1.6113
5 F4	13.34	23.3893
CALCULATED HEAD	100.00	3.6170

TABLE N° 2F16 : FLOTATION TEST N° 16.

PRODUCTS	WEIGHT	Fe203		SrO		BaO		La203	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1 1 0 0 0 0 0	13.02	5.05	31.94	12.00	12.35	15.50	35.00	8.56	8.22
2 F1 2 3 4 5 0 0	86.98	1.61	68.06	12.74	87.65	4.31	65.00	14.30	91.78
3 NF1+NF3 1 3 0 0 0 0	30.07	3.94	57.62	21.21	50.44	16.07	83.80	6.56	14.56
4 F2+F3 2 4 5 0 0 0	69.93	1.25	42.38	8.96	49.56	1.34	16.20	16.56	85.44
5 NF1+NF3+NF4 1 3 4 0 0 0	51.65	3.45	86.45	21.63	88.33	10.94	98.02	8.18	31.18
6 F2+F4 2 5 0 0 0 0	48.35	.58	13.55	3.05	11.67	.24	1.98	19.30	68.82
7 NF1+NF2 1 3 4 5 0 0	64.99	2.88	90.99	17.76	91.28	8.74	98.48	10.50	50.34
8 F2 2 0 0 0 0 0	35.01	.53	9.01	3.15	8.72	.25	1.52	19.23	49.66
9 F2+NF 1 2 3 4 0 0	86.66	2.27	95.46	14.16	97.05	6.62	99.54	12.65	80.84
10 F4 5 0 0 0 0 0	13.34	.70	4.54	2.80	2.95	.20	.46	19.47	19.16

PRODUCTS	WEIGHT	Ce203		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1 1 0 0 0 0 0	13.02	14.98	8.47	29.42	8.37
2 F1 2 3 4 5 0 0	86.98	24.24	91.53	48.18	91.63
3 NF1+NF3 1 3 0 0 0 0	30.07	11.50	15.01	22.58	14.84
4 F2+F3 2 4 5 0 0 0	69.93	28.00	84.99	55.70	85.16
5 NF1+NF3+NF4 1 3 4 0 0 0	51.65	14.29	32.04	28.09	31.72
6 F2+F4 2 5 0 0 0 0	48.35	32.38	67.96	64.60	68.28
7 NF1+NF2 1 3 4 5 0 0	64.99	18.11	51.10	35.77	50.82
8 F2 2 0 0 0 0 0	35.01	32.18	48.90	64.26	49.18
9 F2+NF 1 2 3 4 0 0	86.66	21.52	80.94	42.70	80.90
10 F4 5 0 0 0 0 0	13.34	32.92	19.06	65.49	19.10

Test n°	Pulp pH	pH regulator	Rougher float fraction F1			
			Total REO		BaO content %	SrO content %
			content %	recovery %		
1	4.5	H2SO4	57.11	90.79	0.75	10.52
2	7.8	none	42.53	96.72	5.20	12.28
3	9.0	NaOH	45.76	90.33	5.38	12.96
4	4.5	H3PO4	43.50	82.81	5.61	11.81

As expected, H_3PO_4 slightly depresses monazite.

- scavenger flotation with addition of H_2SO_4 as a depressant and P.E. as a collector (test 5) leads to concentration of barite in the non float fraction which is low in REO (1.79 %). It should be pointed out that strontianite and iron containing carbonates are floated together with monazite, this shows that H_2SO_4 is not satisfactory as a depressant for strontianite and iron bearing minerals

- sodium oleate used as a collector in conjunction with CO_2 in a slightly acidic pulp (about 5.4) results in flotation of both monazite and strontianite while barite is slightly depressed (tests 10 and 11)

- petroleum sulfonate AP 825 at pH 4.5, adjusted by H_2SO_4, has a good affinity for strontianite, it is interesting to note that iron bearing carbonates are not floated together with strontianite and barite (test n° 15) in the rougher and scavenger stages. However, as strontianite concentration drops in the material to be floated, affinity of sulfonate for monazite increases, selectivity for strontianite in the scavenger flotation stage is poor

- conditioning with CO_2 and a small addition of P.E. P301 (50 g/t), followed by rougher and cleaner flotations in a CO_2 containing pulp, depresses gangue minerals: iron bearing carbonates, barite and strontianite while monazite is floated (test n° 16). Scavenger flotation followed by a cleaner stage under the same operating conditions (CO_2 containing pulp and a small addition of P.E. P301-20 g/t) leads to a high monazite concentrate. Separation of strontianite as a non float fraction appears to be a slow rate process, the final composite concentrate has a SrO content in excess of 3 % though the monazite content is high:

	Composite concentrate F2 + F4
Fe_2O_3 assay %	0.58
BaO assay %	0.24
SrO assay %	3.05
total REO content %	64.60
total REO recovery %	68.28
monazite content %	93.60

Recirculation of middlings in a continuous flotation circuit would probably lead to increased monazite recoveries in final concentrates.

3.2.4. Flotation of the - 40 + 10 µm gravity preconcentrate

Tests conducted on the - 160 + 10 µm, - 50 + 10 µm fractions and the - 80 + 10 µm gravity preconcentrate have revealed important features relevant to the amenability of the REO ore sample to flotation:

- strong depression of barite with H_2SO_4 when floating with P.E. as a collector
- flotation of strontianite with a petroleum sulfonate as a collector and H_2SO_4 as a pH regulator and depressant
- depression of gangue minerals: iron bearing carbonates, barite and strontianite, in a CO_2 containing pulp and multi stage flotation of monazite by a P.E. as a collector. Application of the process to a gravity preconcentrate leads to a high monazite flotation concentrate. However, removal of strontianite is poor in the refining stages (cleaner flotation), thereby the process would deserve to be improved in order to get better concentration performance for monazite.

More complex flowsheets, involving combinations of the features mentioned above, were evaluated on the - 40 + 10 µm gravity preconcentrate. It should be pointed out that the Fe_2O_3 content of the feed to flotation is much higher than that determined on the - 80 + 10 µm preconcentrate, this indicates a higher content in iron bearing carbonates which are difficult to separate from monazite.

Operating conditions and flowsheets of flotation tests, conducted in Minemet flotation cells of 2.5 and 0.8 l capacity, are shown in figures 2F20 to 2F29, relevant material balances are given in tables 2F20 to 2F29.

Four basic types of flowsheets were evaluated:

- flowsheet I (test n° 20), involving flotation with P.E. 301 as a collector and H_2SO_4 as a pH regulator and barite depressant, followed by conditioning with CO_2 and multi-stage flotation of monazite with small additions of P.E.
- flowsheet II (test n° 21), involving flotation with P.E. 301 a collector and H_2SO_4 as a pH regulator and barite depressant to produce a barite concentrate as a final non float fraction. The float fraction is first attritioned at high pulp density and acidic pH to clean the surface of the particles. The cleaned material is conditioned with H_2SO_4 and sulfonate AP 825 to float strontianite. The non float fraction is conditioned with CO_2 and P.E. to separate monazite from residual impurities by multi-stage flotation
- flowsheet III (tests n° 22 to 28), consisting of:
  - stage 1: flotation with H_2SO_4 and sulfonate AP825 to separate strontianite in rougher-scavenger-cleaner steps, leaving a SrO enriched float fraction, cleaner tailings and scavenger tailings

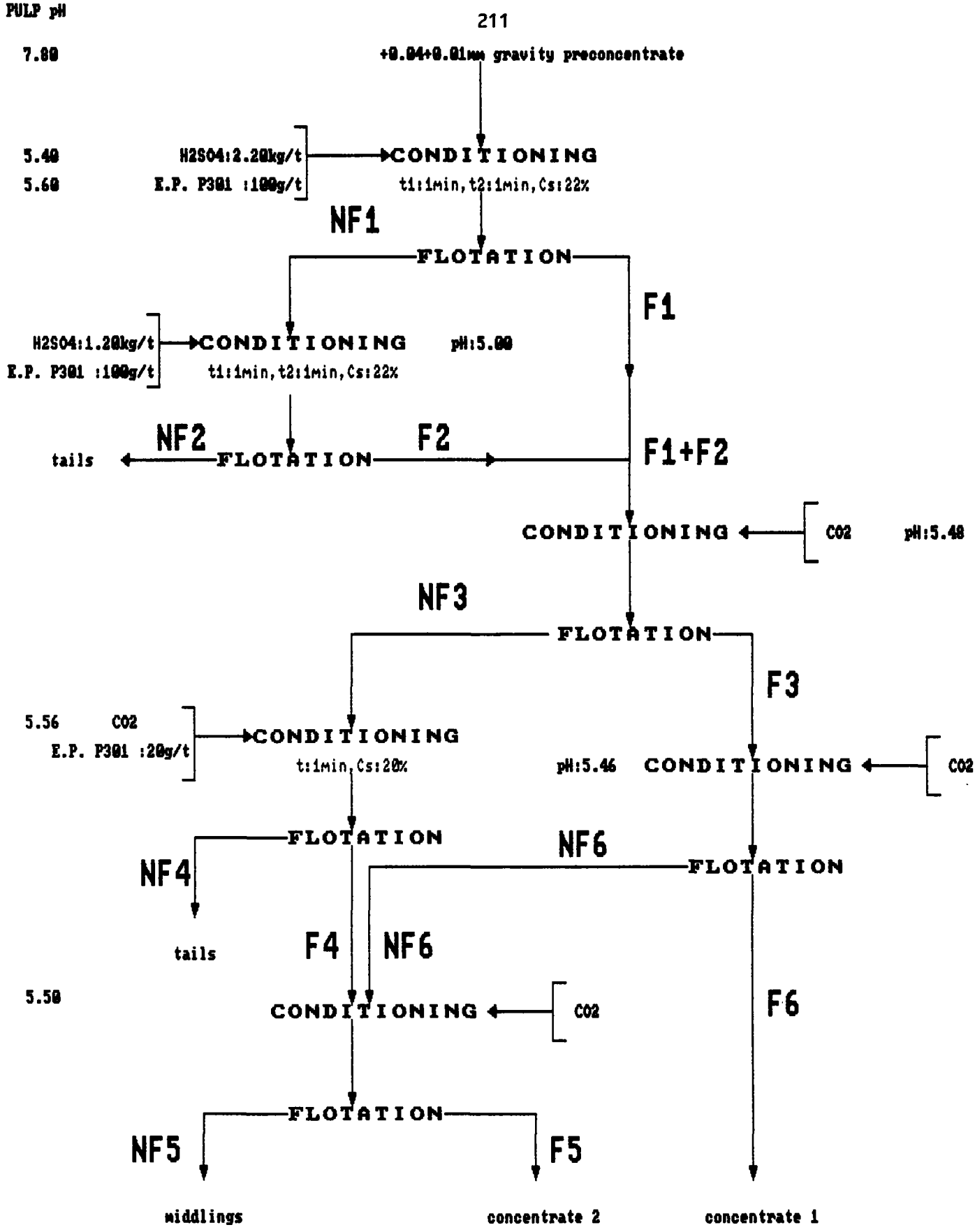
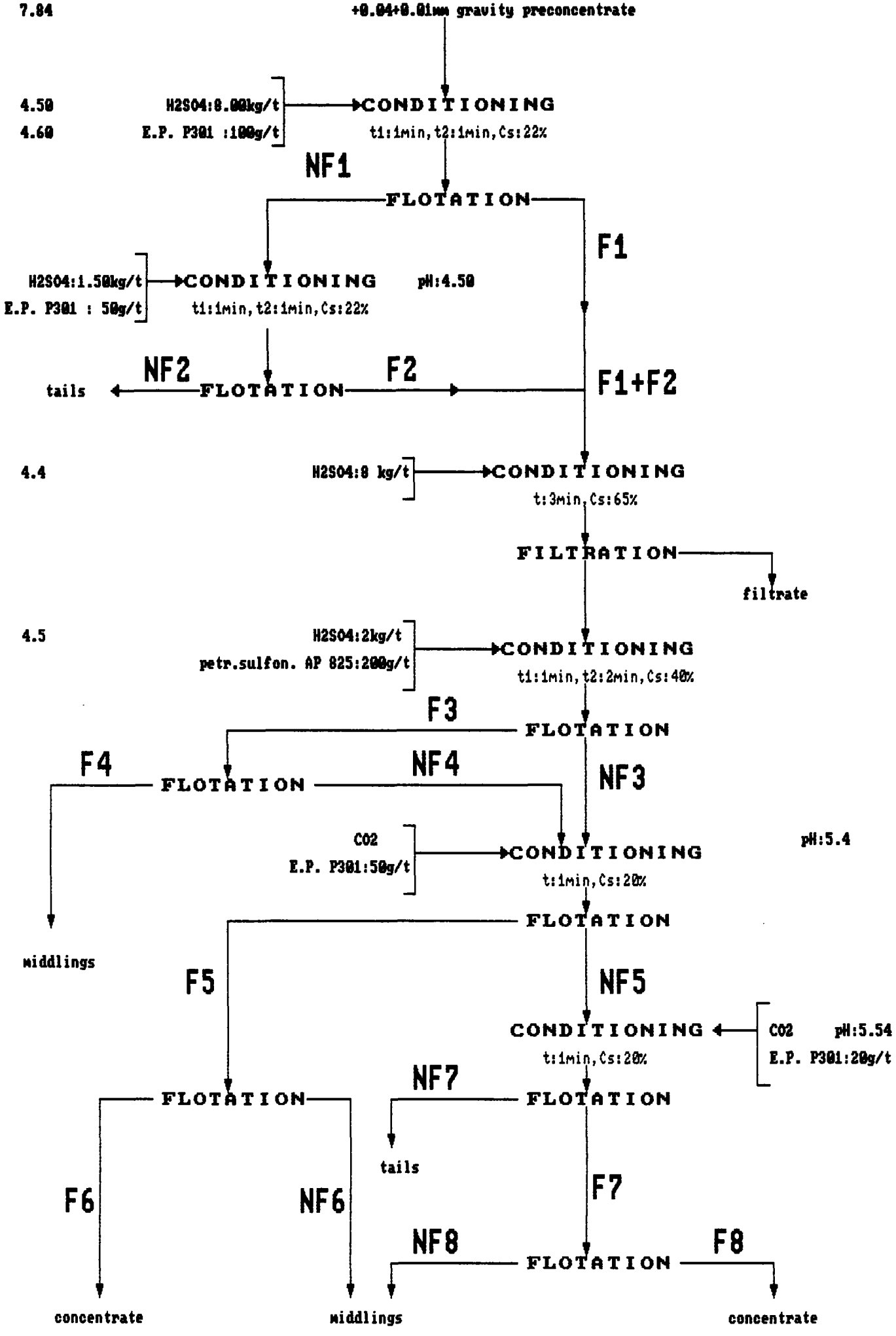


FIGURE N° 2F20 : FLOWSHEET OF FLOTATION TEST N° 20

PULP pH

FIGURE N° 2F21 : FLOWSHEET OF FLOTATION TEST N° 21



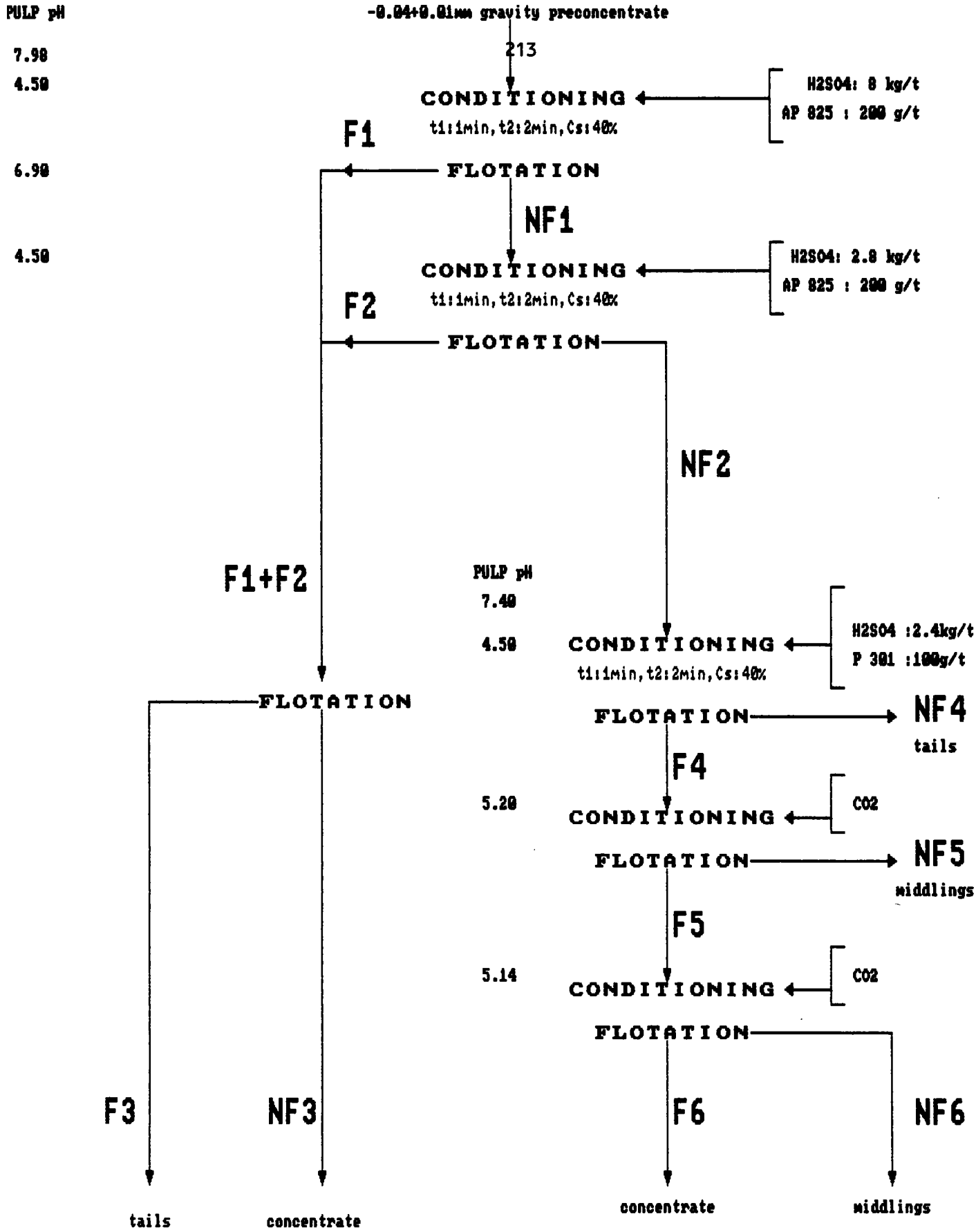


FIGURE N° 2F22 : FLOWSHEET OF FLOTATION TEST N°22.

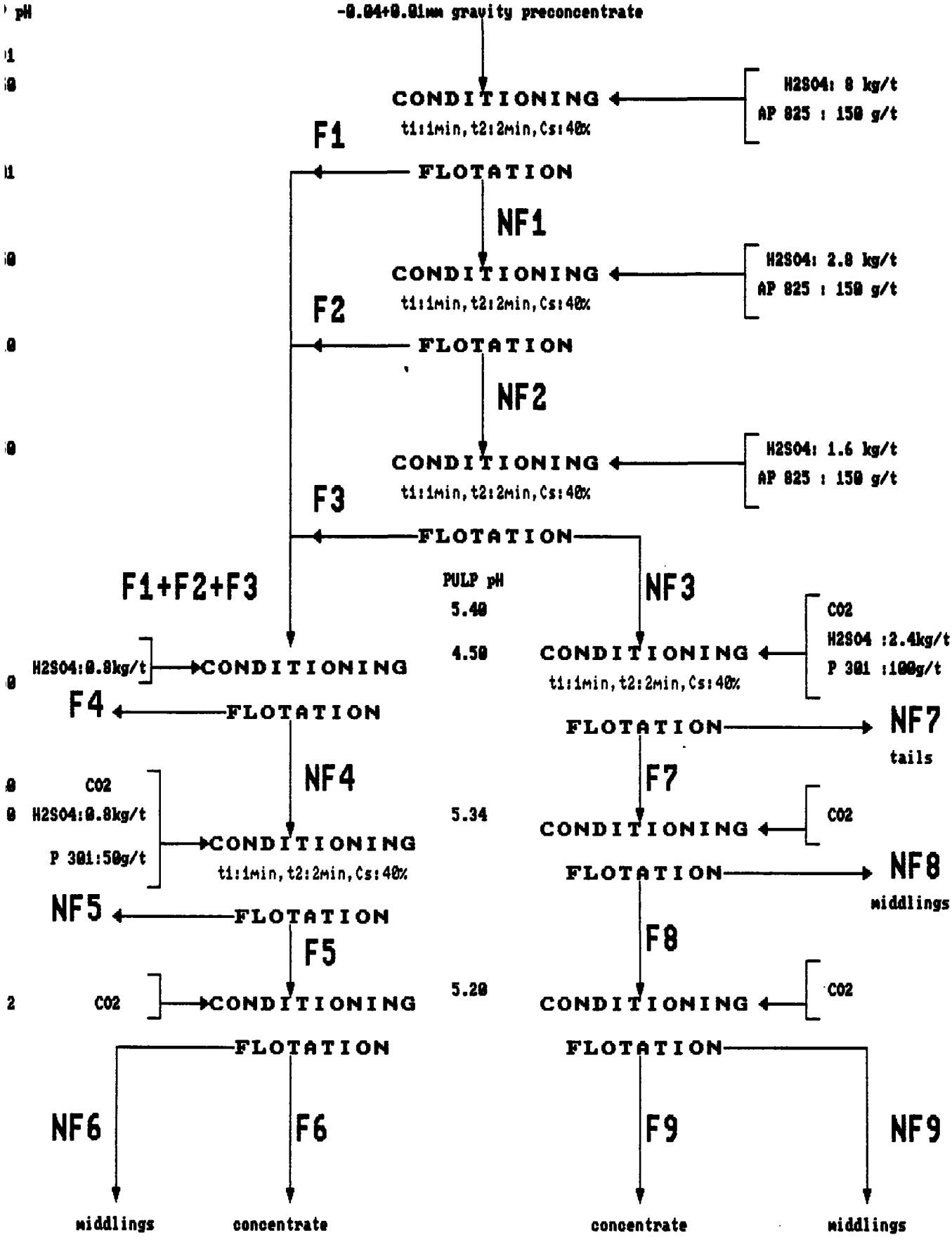


FIGURE N° 2F23 : FLOWSHEET OF FLOTATION TEST N°23.

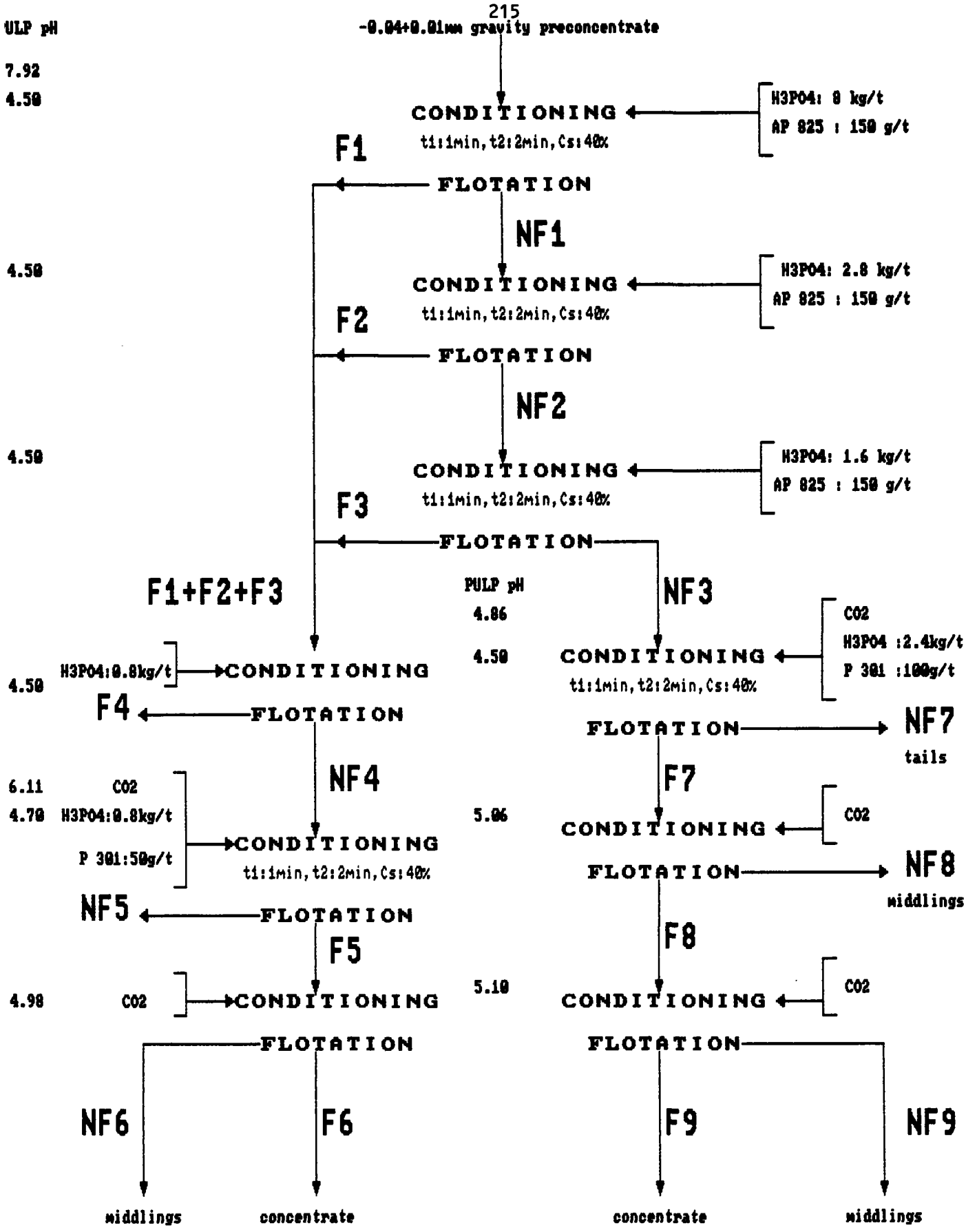


FIGURE N° 2F24 : FLOWSHEET OF FLOTATION TEST N°24.

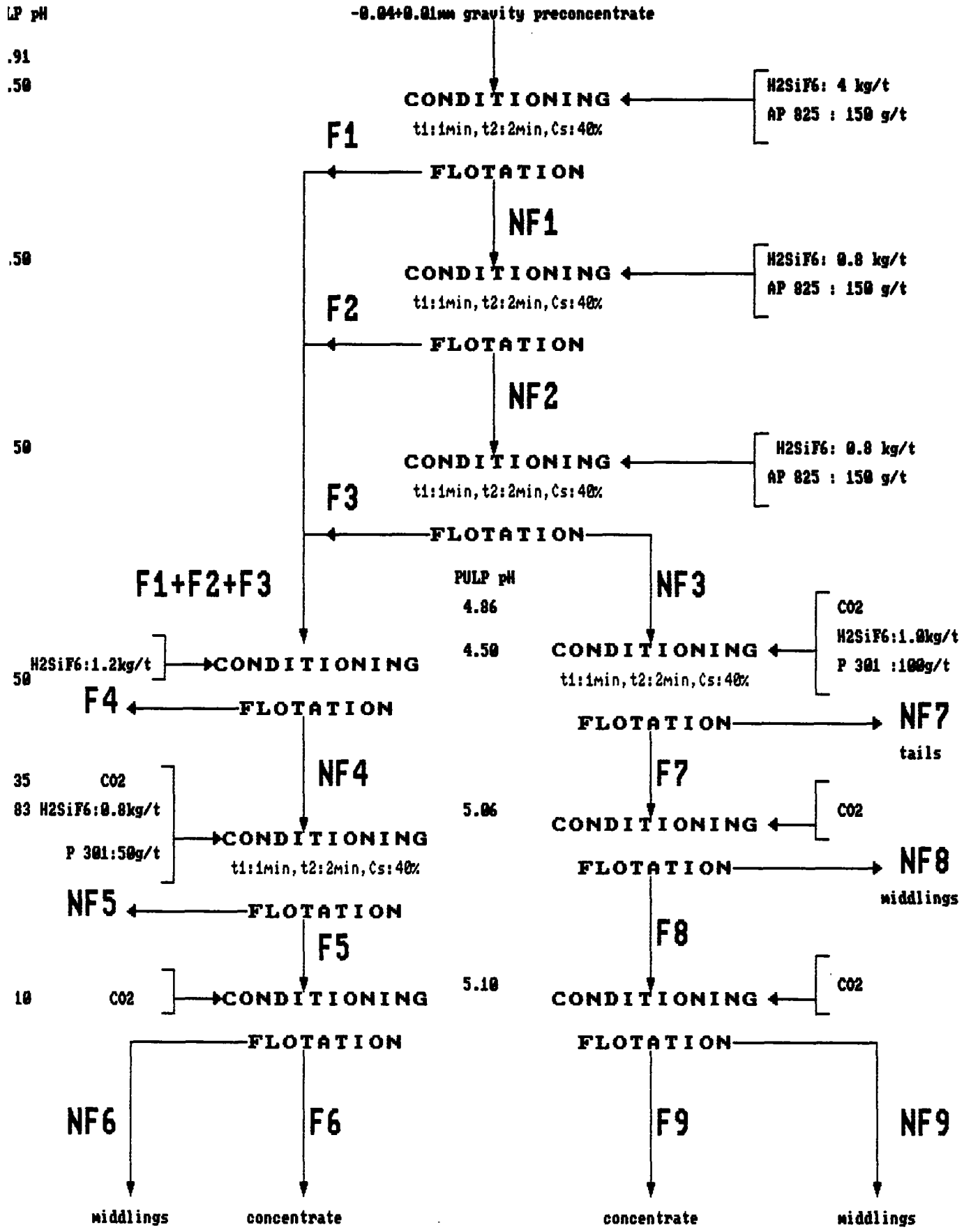


FIGURE N° 2F25 : FLOWSHEET OF FLOTATION TEST N°25.

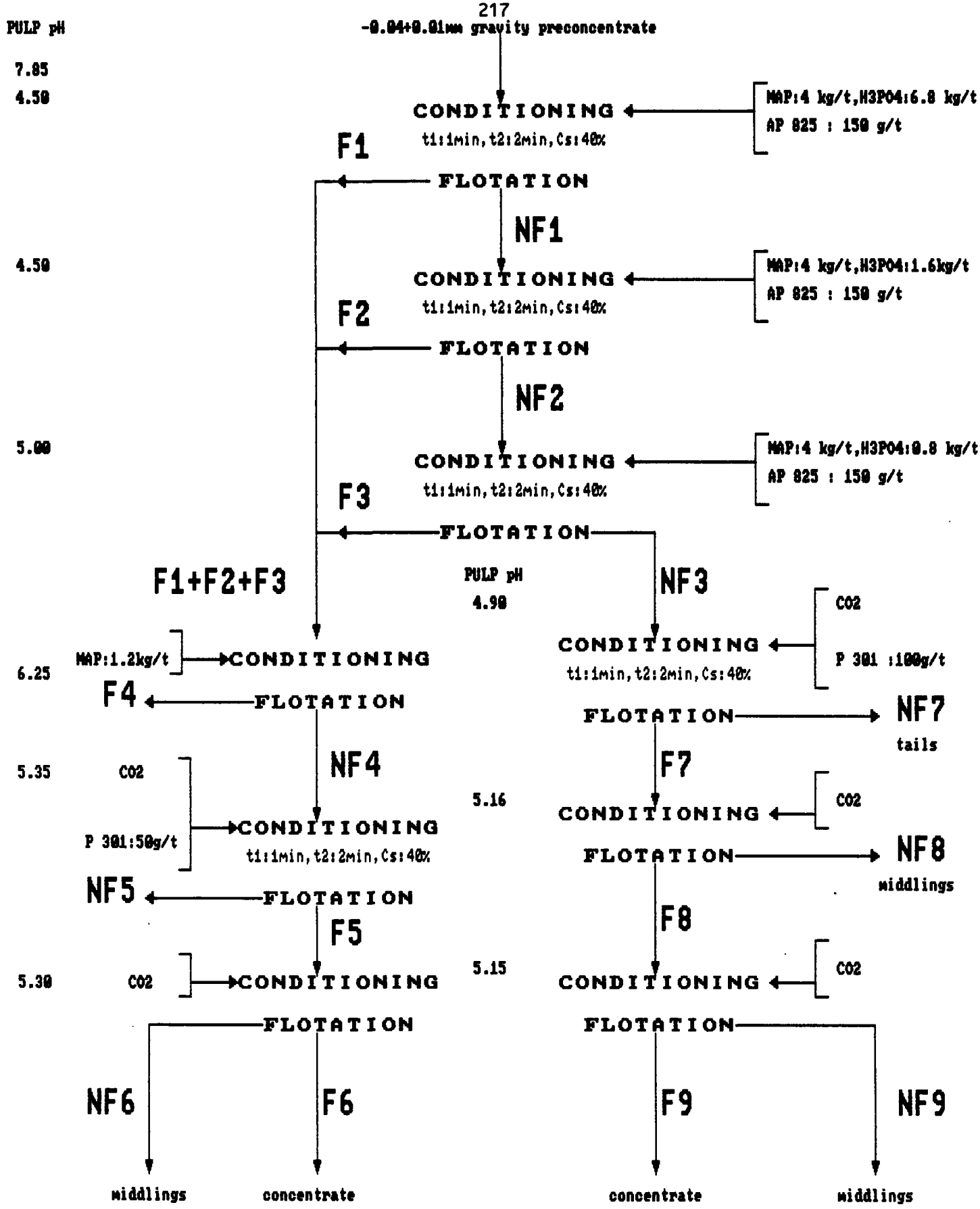


FIGURE N° 2F26 : FLOWSHEET OF FLOTATION TEST N°26.

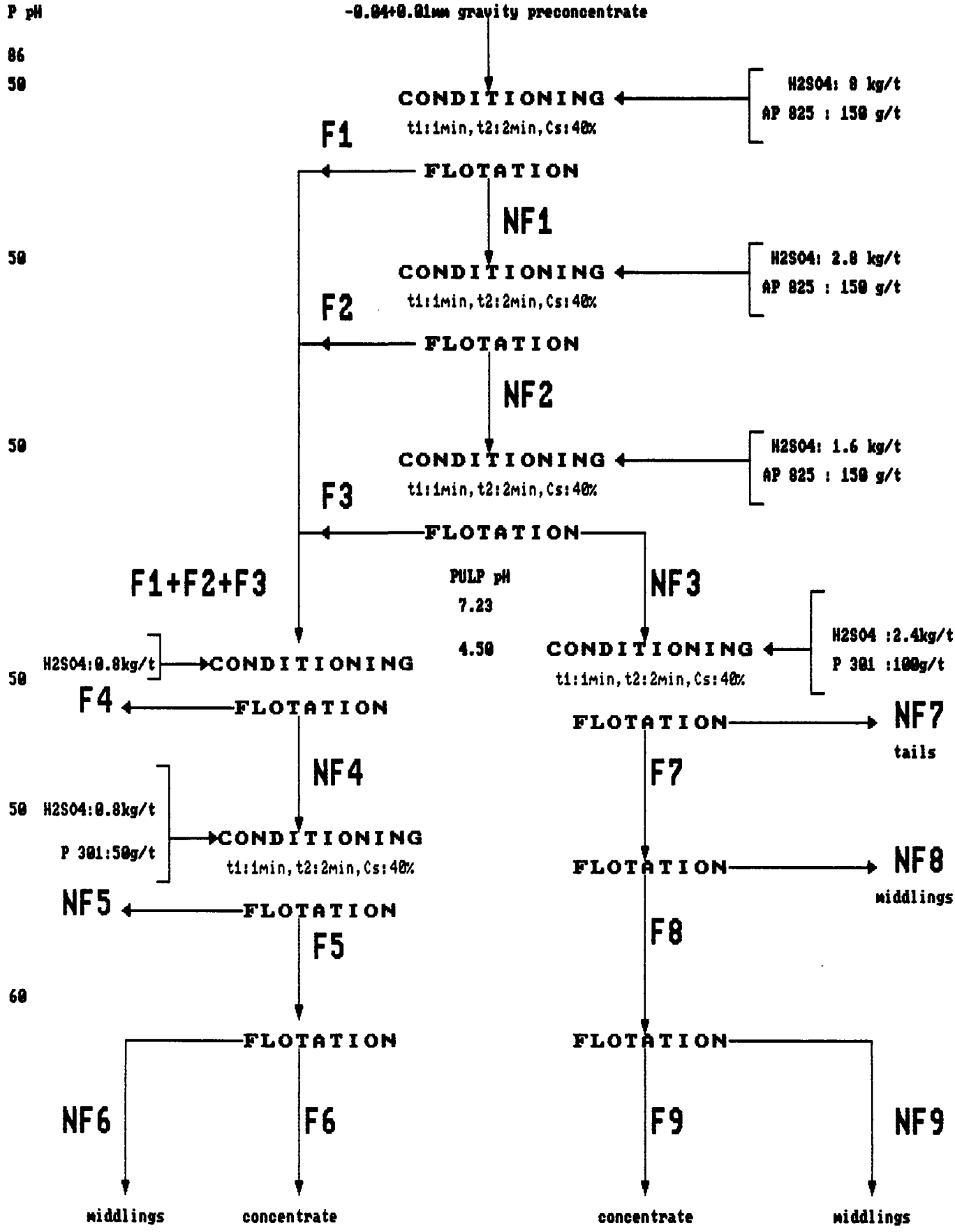


FIGURE N° 2F27 : FLOWSHEET OF FLOTATION TEST N°27.

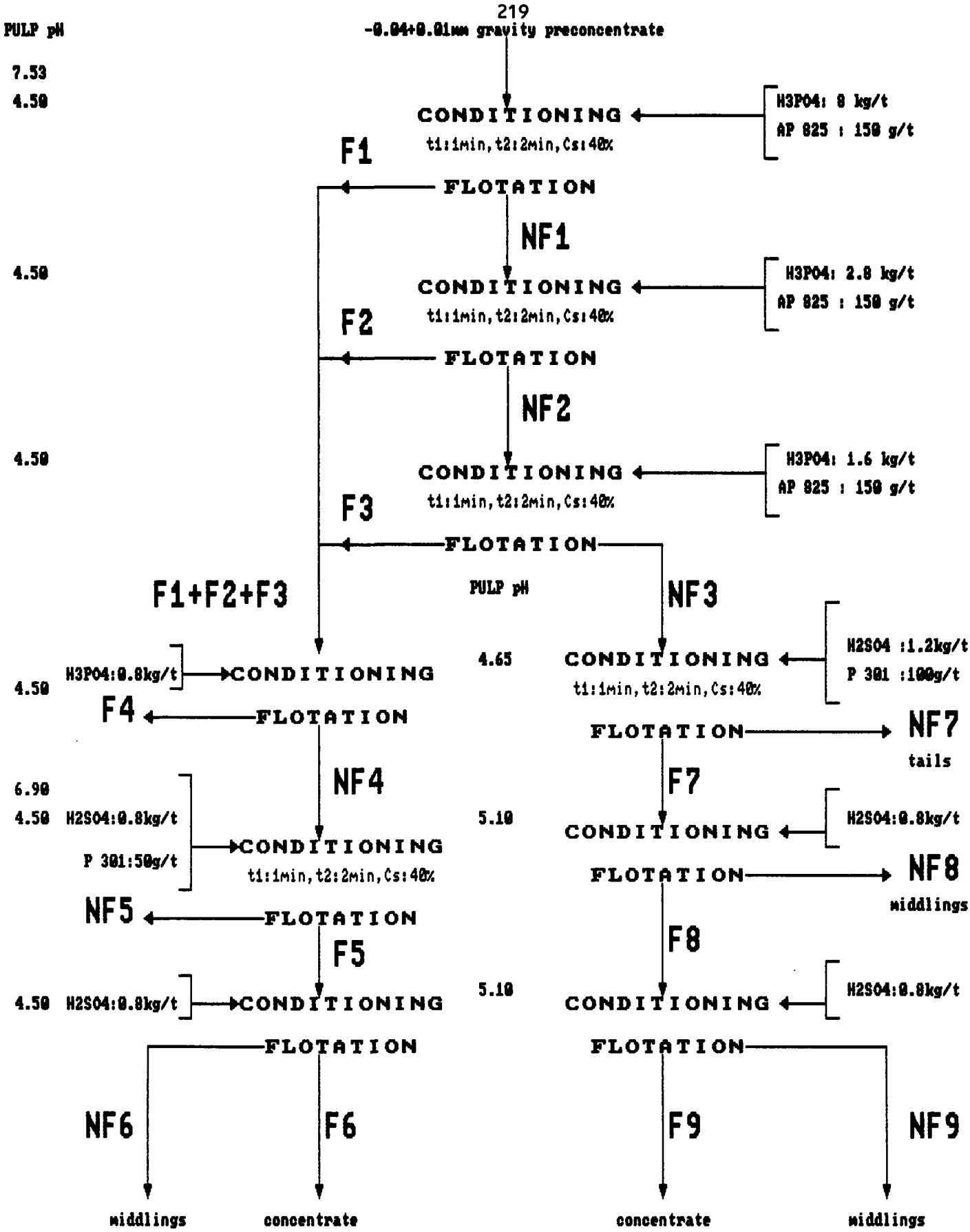


FIGURE N° 2F28 : FLOWSHEET OF FLOTATION TEST N°28.

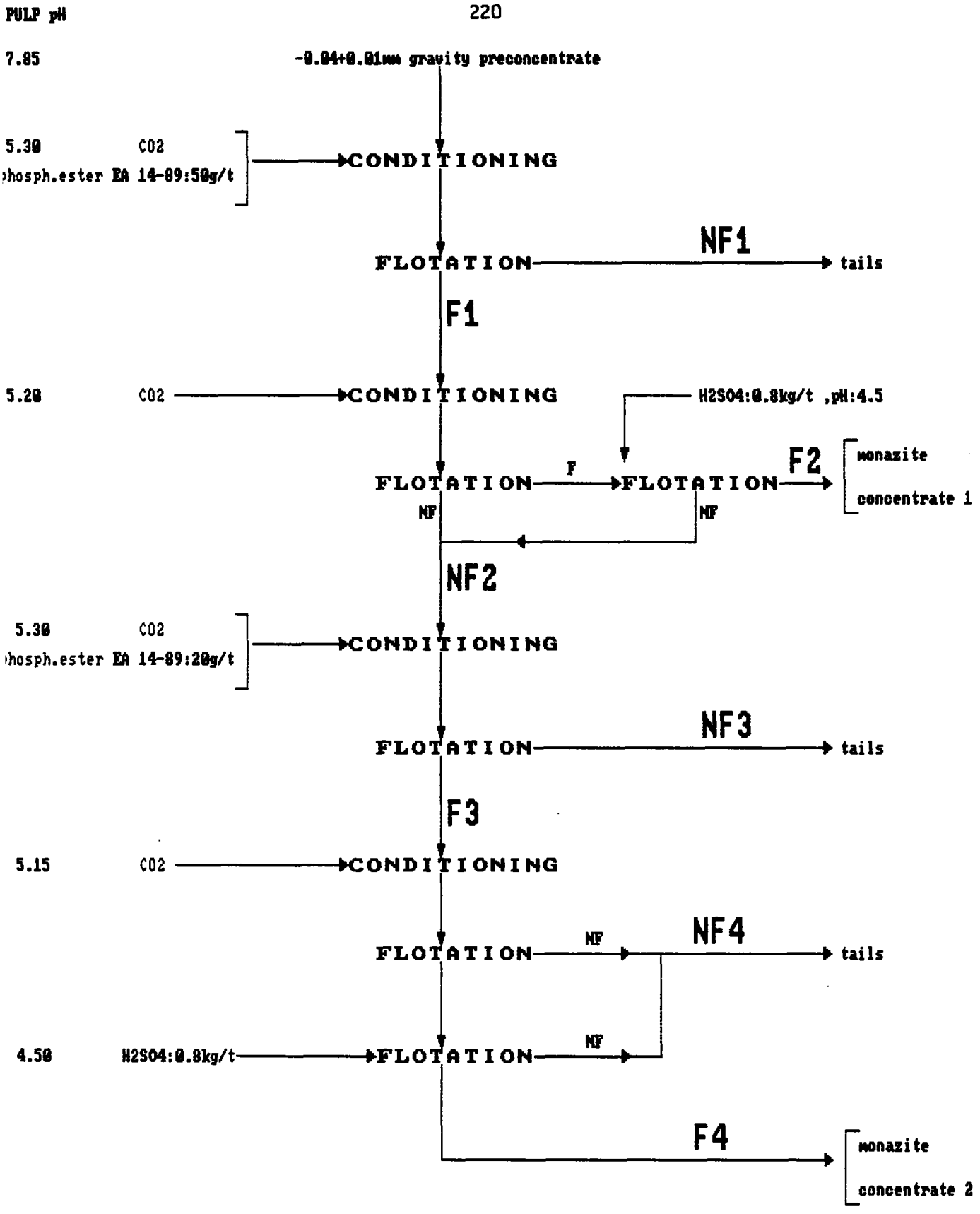


FIGURE N° 2F29 : FLOWSHEET OF FLOTATION TEST N°29 ON THE -0.04+0.01mm GRAVITY PRECONCENTRATE SEPARATED FROM THE REO ORE SAMPLE N°2 GROUND DOWN TO 0.16mm.

TABLE N° 2F20 : RED ORE SAMPLE N°2,-0.04+0.01mm GRAVITY PRECONCENTRATE, MATERIAL BALANCE OF FLOTATION TEST N°20.

PRODUCTS	WEIGHT	Fe2O3		SrO		BaO		RED TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF2	.45	9.50	.59	4.10	.15	34.00	3.17	2.50	.04
2 NF4	5.57	11.20	8.61	14.01	6.16	17.93	20.67	11.64	2.09
3 F6	40.85	3.45	19.46	8.28	26.72	.76	6.43	43.21	56.85
4 F5	30.30	9.54	39.91	12.42	29.73	2.68	16.81	31.19	30.44
5 NF5	22.83	9.97	31.43	20.65	37.24	11.20	52.93	14.41	10.59
CALCULATED HEAD	100.00	7.24	100.00	12.66	100.00	4.83	100.00	31.05	100.00

PRODUCTS	WEIGHT %	RED TOT. GRADE / SrO GRADE
1 NF2	.45	.6098
2 NF4	5.57	.8308
3 F6	40.85	5.2186
4 F5	30.30	2.5113
5 NF5	22.83	.6978
CALCULATED HEAD	100.00	2.4529

TABLE N° 2F20 : FLOTATION TEST N° 20.

PRODUCTS	WEIGHT	Fe203		SrO		BaO		RED TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF2 1 0 0 0 0 0	.45	9.50	.59	4.10	.15	34.00	3.17	2.50	.04
2 F1+F2 2 3 4 5 0 0	99.55	7.23	99.41	12.70	99.85	4.70	96.83	31.18	99.96
3 F4+NF6 4 5 0 0 0 0	53.13	9.72	71.34	15.96	66.97	6.34	69.73	23.98	41.03
4 F5+F6 3 4 0 0 0 0	71.15	6.04	59.37	10.04	56.45	1.58	23.23	38.09	87.28
5 NF2+NF4+NF5 1 2 5 0 0 0	28.85	10.20	40.63	19.11	43.55	12.85	76.77	13.69	12.72

TABLE N° 2F21 : REO ORE SAMPLE N°2,-0.04+0.01mm GRAVITY PRECONCENTRATE, MATERIAL BALANCE OF FLOTATION TEST N°21.

PRODUCTS	WEIGHT	Fe203		SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF2	6.38	8.13	7.34	5.96	3.24	31.07	46.87	3.82	.79
2 F4	36.33	3.77	19.37	7.76	24.05	1.24	10.65	42.31	49.53
3 NF7	9.84	18.23	25.37	18.91	15.88	8.51	19.80	9.89	3.14
4 F6	9.60	3.40	4.62	16.39	13.42	1.60	3.63	28.66	8.87
5 NF6	19.05	6.15	16.57	12.69	20.62	1.85	8.33	32.29	19.82
6 F8	6.01	1.00	.85	6.88	3.53	.45	.64	55.35	10.72
7 NF8	12.80	14.30	25.89	17.63	19.25	3.33	10.08	17.31	7.14
CALCULATED HEAD	100.00	7.07	100.00	11.72	100.00	4.23	100.00	31.03	100.00

PRODUCTS	WEIGHT %	REO TOT. GRADE / SrO GRADE
1 NF2	6.38	.6409
2 F4	36.33	5.4523
3 NF7	9.84	.5230
4 F6	9.60	1.7486
5 NF6	19.05	2.5445
6 F8	6.01	8.0451
7 NF8	12.80	.9818
CALCULATED HEAD	100.00	2.6476

TABLE N° 2F21 : FLOTATION TEST N° 21.

PRODUCTS	WEIGHT	Fe2O3		SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF2 1 0 0 0 0 0	6.38	8.13	7.34	5.96	3.24	31.07	46.87	3.82	.79
2 F1+F2 2 3 4 5 6 7	93.62	7.00	92.66	12.11	96.76	2.40	53.13	32.88	99.21
3 F4+F8 2 6 0 0 0 0	42.34	3.38	20.22	7.64	27.58	1.13	11.29	44.16	60.25
4 F6+ SUM(NF) 1 3 4 5 7 0	57.66	9.78	79.78	14.72	72.42	6.51	88.71	21.39	39.75
5 F7 6 7 0 0 0 0	18.81	10.05	26.74	14.20	22.78	2.41	10.72	29.46	17.86
6 NF7 3 0 0 0 0 0	9.84	18.23	25.37	18.91	15.88	8.51	19.80	9.89	3.14
7 F5 4 5 0 0 0 0	28.65	5.23	21.19	13.93	34.05	1.77	11.96	31.07	28.69
8 NF5 6 7 3 0 0 0	28.65	12.86	52.11	15.81	38.66	4.50	30.52	22.74	21.00
9 NF3+NF4 3 4 5 6 7 0	57.29	9.04	73.29	14.87	72.70	3.14	42.48	26.91	49.68

TABLE N° 2F22 : REO ORE SAMPLE N°2,-0.04+0.01mm GRAVITY PRECONCENTRATE, MATERIAL BALANCE OF FLOTATION TEST N°22.

PRODUCTS	WEIGHT	Fe2O3		SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F3	12.67	1.00	1.79	25.00	30.79	3.25	9.44	20.65	8.09
2 NF3	30.05	3.09	13.09	14.22	41.54	3.64	25.07	33.23	30.89
3 NF4	8.64	22.13	26.96	4.59	3.86	13.33	26.40	10.80	2.89
4 NF5	17.26	15.08	36.71	6.95	11.66	7.20	28.49	16.39	8.75
5 F6	18.78	1.00	2.65	2.33	4.25	.51	2.20	64.22	37.31
6 NF6	12.60	10.58	18.80	6.44	7.89	2.91	8.40	30.95	12.06
CALCULATED HEAD	100.00	7.09	100.00	10.29	100.00	4.36	100.00	32.32	100.00

PRODUCTS	WEIGHT %	REO TOT. GRADE / SrO GRADE
1 F3	12.67	.8260
2 NF3	30.05	2.3368
3 NF4	8.64	2.3529
4 NF5	17.26	2.3583
5 F6	18.78	27.5622
6 NF6	12.60	4.8059
CALCULATED HEAD	100.00	3.1426

TABLE N° 2F22 : FLOTATION TEST N°22.

PRODUCTS	WEIGHT	Fe_2O_3		SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1+F2 1 2 0 0 0 0	42.72	2.47	14.88	17.42	72.34	3.52	34.51	29.50	38.99
2 NF2 3 4 5 6 0 0	57.28	10.54	85.12	4.97	27.66	4.99	65.49	34.43	61.01
3 NF4 3 0 0 0 0 0	8.64	22.13	26.96	4.59	3.86	13.33	26.40	10.80	2.89
4 F4 4 5 6 0 0 0	48.64	8.48	58.15	5.03	23.81	3.51	39.09	38.63	58.13
5 NF5 4 0 0 0 0 0	17.26	15.08	36.71	6.95	11.66	7.20	28.49	16.39	8.75
6 F5 5 6 0 0 0 0	31.38	4.85	21.45	3.98	12.14	1.47	10.60	50.86	49.38

TABLE N° 2F23 : REO ORE SAMPLE N°2,-0.04+0.01mm GRAVITY PRECONCENTRATE, MATERIAL BALANCE OF FLOTATION TEST N°23.

PRODUCTS	WEIGHT	Fe203		SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F4	11.60	2.51	4.45	25.00	24.35	2.09	4.85	21.47	7.60
2 NF5	3.20	9.58	4.69	16.78	4.51	6.19	3.96	18.90	1.85
3 F6	10.49	4.47	7.17	16.06	14.15	3.47	7.29	38.58	12.35
4 NF6	7.18	1.00	1.10	7.86	4.74	1.46	2.10	29.51	6.47
5 NF7	14.47	11.20	24.77	21.61	26.26	21.10	61.12	7.86	3.47
6 NF8	11.61	14.47	25.68	11.75	11.45	4.76	11.06	18.35	6.50
7 F9	25.12	1.08	4.15	2.45	5.17	.60	3.02	58.63	44.95
8 NF9	16.33	11.22	28.00	6.84	9.38	2.02	6.60	33.72	16.81
CALCULATED HEAD	100.00	6.54	100.00	11.91	100.00	5.00	100.00	32.76	100.00

PRODUCTS	WEIGHT %	REO TOT. GRADE / SrO GRADE
1 F4	11.60	.8588
2 NF5	3.20	1.1263
3 F6	10.49	2.4022
4 NF6	7.18	3.7545
5 NF7	14.47	.3637
6 NF8	11.61	1.5617
7 F9	25.12	23.9306
8 NF9	16.33	4.9298
CALCULATED HEAD	100.00	2.7510

TABLE N° 2F23 : FLOTATION TEST N° 23.

PRODUCTS	WEIGHT	Fe2O3		SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF3 5 6 7 8 0 0	67.53	8.00	82.60	9.22	52.26	6.05	81.80	34.80	71.73
2 F1+F2+F3 1 2 3 4 0 0	32.47	3.51	17.40	17.51	47.74	2.80	18.20	28.52	28.27
3 NF4 2 3 4 0 0 0	20.87	4.06	12.95	13.35	23.39	3.20	13.35	32.44	20.67
4 F4 1 0 0 0 0 0	11.60	2.51	4.45	25.00	24.35	2.09	4.85	21.47	7.60
5 NF5 2 0 0 0 0 0	3.20	9.58	4.69	16.78	4.51	6.19	3.96	18.90	1.85
6 F5 3 4 0 0 0 0	17.67	3.06	8.26	12.73	18.88	2.65	9.38	34.89	18.82
7 NF7 5 0 0 0 0 0	14.47	11.20	24.77	21.61	26.26	21.10	61.12	7.86	3.47
8 F7 6 7 8 0 0 0	53.06	7.13	57.83	5.84	26.00	1.95	20.68	42.15	68.26
9 NF8 6 0 0 0 0 0	11.61	14.47	25.68	11.75	11.45	4.76	11.06	18.35	6.50
10 F8 7 8 0 0 0 0	41.45	5.07	32.15	4.18	14.55	1.16	9.62	48.82	61.76
11 F4+NF7 1 5 0 0 0 0	26.07	7.33	29.22	23.12	50.61	12.64	65.97	13.92	11.07
12 NF5+NF6 2 4 0 0 0 0	10.38	3.65	5.78	10.61	9.25	2.92	6.06	26.24	8.31
13 NF8+NF9 6 8 0 0 0 0	27.94	12.57	53.68	8.88	20.83	3.16	17.67	27.33	23.31
14 F6+F9 3 7 0 0 0 0	35.61	2.08	11.31	6.46	19.31	1.45	10.30	52.72	57.30

TABLE N° 2F24 : RED ORE SAMPLE N°2,-0.04+0.01mm GRAVITY PRECONCENTRATE, MATERIAL BALANCE OF FLOTATION TEST N°24.

PRODUCTS	WEIGHT	Fe2O3		SrO		BaO		RED TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F4	5.74	1.00	.81	25.00	13.53	8.67	10.26	11.50	2.00
2 NF5	5.42	4.55	3.49	12.67	6.48	9.07	10.13	31.86	5.24
3 F6	9.27	1.00	1.31	25.00	21.86	6.94	13.26	21.67	6.10
4 NF6	4.67	1.23	.81	17.64	7.77	8.75	8.42	20.10	2.85
5 NF7	12.09	28.07	48.08	3.34	3.81	3.76	9.37	18.90	6.94
6 NF8	14.29	12.84	26.00	4.07	5.49	3.16	9.31	33.75	14.65
7 F9	26.26	1.00	3.72	10.87	26.92	4.18	22.63	40.38	32.21
8 NF9	22.26	5.00	15.77	6.74	14.15	3.62	16.61	44.38	30.00
CALCULATED HEAD	100.00	7.06	100.00	10.60	100.00	4.85	100.00	32.93	100.00

PRODUCTS	WEIGHT %	RED TOT. GRADE / SrO GRADE
1 F4	5.74	.4600
2 NF5	5.42	2.5146
3 F6	9.27	.8668
4 NF6	4.67	1.1395
5 NF7	12.09	5.6587
6 NF8	14.29	8.2924
7 F9	26.26	3.7148
8 NF9	22.26	6.5846
CALCULATED HEAD	100.00	3.1052

TABLE N° 2F24 : FLOTATION TEST N° 24.

PRODUCTS	WEIGHT	Fe2O3		SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF3 5 6 7 8 0 0	74.90	8.82	93.57	7.13	50.36	3.75	57.92	36.84	83.80
2 F1+F2+F3 1 2 3 4 0 0	25.10	1.81	6.43	20.97	49.64	8.13	42.08	21.25	16.20
3 NF4 2 3 4 0 0 0	19.36	2.05	5.62	19.77	36.10	7.97	31.82	24.14	14.20
4 F4 1 0 0 0 0 0	5.74	1.00	.81	25.00	13.53	8.67	10.26	11.50	2.00
5 NF5 2 0 0 0 0 0	5.42	4.55	3.49	12.67	6.48	9.07	10.13	31.86	5.24
6 F5 3 4 0 0 0 0	13.94	1.08	2.13	22.53	29.63	7.55	21.69	21.14	8.95
7 NF7 5 0 0 0 0 0	12.09	28.07	48.08	3.34	3.81	3.76	9.37	18.90	6.94
8 F7 6 7 8 0 0 0	62.81	5.11	45.48	7.86	46.56	3.75	48.55	40.29	76.86
9 NF8 6 0 0 0 0 0	14.29	12.84	26.00	4.07	5.49	3.16	9.31	33.75	14.65
10 F8 7 8 0 0 0 0	48.52	2.84	19.49	8.98	41.07	3.92	39.24	42.22	62.21
11 F4+NF7 1 5 0 0 0 0	17.83	19.36	48.89	10.31	17.34	5.34	19.63	16.52	8.94
12 NF5+NF6 2 4 0 0 0 0	10.09	3.01	4.31	14.97	14.25	8.92	18.56	26.42	8.10
13 NF8+NF9 6 8 0 0 0 0	36.55	8.07	41.76	5.70	19.63	3.44	25.92	40.22	44.65
14 F6+F9 3 7 0 0 0 0	35.53	1.00	5.03	14.56	48.78	4.90	35.89	35.50	38.31

TABLE N° 2F25 : REO ORE SAMPLE N°2,-0.04+0.01mm GRAVITY PRECONCENTRATE, MATERIAL BALANCE OF FLOTATION TEST N°25.

PRODUCTS	WEIGHT	Fe2O3		SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F4	29.72	1.00	4.20	17.38	43.73	5.22	34.10	33.14	31.30
2 NF5	9.84	7.44	10.34	9.75	8.12	5.53	11.96	29.18	9.12
3 F6	17.82	1.00	2.52	9.76	14.73	2.58	10.11	45.10	25.55
4 NF6	15.54	2.48	5.44	11.26	14.81	6.14	20.97	32.47	16.03
5 NF7	4.37	31.59	19.51	5.67	2.10	4.01	3.86	14.35	1.99
6 NF8	6.11	27.02	23.31	8.68	4.49	4.85	6.51	12.17	2.36
7 F9	8.27	10.97	12.82	5.65	3.96	2.70	4.91	35.61	9.36
8 NF9	8.32	18.58	21.85	11.44	8.06	4.14	7.58	16.15	4.27
CALCULATED HEAD	100.00	7.08	100.00	11.81	100.00	4.55	100.00	31.47	100.00

PRODUCTS	WEIGHT %	REO TOT. GRADE / SrO GRADE
1 F4	29.72	1.9068
2 NF5	9.84	2.9928
3 F6	17.82	4.6209
4 NF6	15.54	2.8837
5 NF7	4.37	2.5309
6 NF8	6.11	1.4021
7 F9	8.27	6.3027
8 NF9	8.32	1.4117
CALCULATED HEAD	100.00	2.6639

TABLE N° 2F25 : FLOTATION TEST N° 25.

PRODUCTS	WEIGHT	Fe203		SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF3 5 6 7 8 0 0	27.08	20.26	77.50	8.12	18.61	3.84	22.86	20.91	17.99
2 F1+F2+F3 1 2 3 4 0 0	72.92	2.18	22.50	13.18	81.39	4.81	77.14	35.39	82.01
3 NF4 2 3 4 0 0 0	43.20	3.00	18.30	10.30	37.66	4.53	43.04	36.93	50.70
4 F4 1 0 0 0 0 0	29.72	1.00	4.20	17.38	43.73	5.22	34.10	33.14	31.30
5 NF5 2 0 0 0 0 0	9.84	7.44	10.34	9.75	8.12	5.53	11.96	29.18	9.12
6 F5 3 4 0 0 0 0	33.36	1.69	7.96	10.46	29.54	4.24	31.08	39.22	41.58
7 NF7 5 0 0 0 0 0	4.37	31.59	19.51	5.67	2.10	4.01	3.86	14.35	1.99
8 F7 6 7 8 0 0 0	22.71	18.08	57.99	8.59	16.51	3.81	19.00	22.17	16.00
9 NF8 6 0 0 0 0 0	6.11	27.02	23.31	8.68	4.49	4.85	6.51	12.17	2.36
10 F8 7 8 0 0 0 0	16.60	14.79	34.67	8.55	12.02	3.42	12.49	25.85	13.64
11 F4+NF7 1 5 0 0 0 0	34.09	4.92	23.71	15.88	45.83	5.06	37.96	30.73	33.30
12 NF5+NF6 2 4 0 0 0 0	25.38	4.40	15.78	10.67	22.93	5.90	32.93	31.19	25.16
13 NF8+NF9 6 8 0 0 0 0	14.43	22.15	45.16	10.27	12.55	4.44	14.09	14.47	6.64
14 F6+F9 3 7 0 0 0 0	26.10	4.16	15.34	8.46	18.69	2.62	15.02	42.09	34.91

TABLE N° 2F26 : REO ORE SAMPLE N°2,-0.04+0.01mm GRAVITY PRECONCENTRATE, MATERIAL BALANCE OF FLOTATION TEST N°26.

PRODUCTS	WEIGHT	Fe2O3		SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF5	2.12	17.05	5.34	13.52	2.63	17.28	7.87	20.35	1.33
2 F6	7.99	1.00	1.18	25.00	18.30	5.45	9.36	17.02	4.19
3 NF6	3.12	3.46	1.59	20.67	5.91	5.60	3.75	19.28	1.86
4 NF7	23.25	19.20	65.91	6.80	14.48	3.73	18.64	20.43	14.65
5 NF8	20.48	6.49	19.62	6.29	11.80	3.58	15.76	45.32	28.63
6 F9	24.20	1.00	3.57	15.00	33.25	5.38	27.98	34.80	25.98
7 NF9	18.84	1.00	2.78	7.90	13.63	4.11	16.64	40.20	23.36
CALCULATED HEAD	100.00	6.77	100.00	10.92	100.00	4.65	100.00	32.42	100.00

PRODUCTS	WEIGHT %	REO TOT. GRADE / SrO GRADE
1 NF5	2.12	1.5052
2 F6	7.99	.6808
3 NF6	3.12	.9328
4 NF7	23.25	3.0044
5 NF8	20.48	7.2051
6 F9	24.20	2.3200
7 NF9	18.84	5.0886
CALCULATED HEAD	100.00	2.9698

TABLE N° 2F26 : FLOTATION TEST N° 26.

PRODUCTS	WEIGHT	Fe2O3		SrO		BaO		RED TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF3 4 5 6 7 0 0	86.77	7.17	91.89	9.21	73.17	4.24	79.01	34.61	92.62
2 F1+F2+F3 1 2 3 0 0 0	13.23	4.15	8.11	22.14	26.83	7.38	20.99	18.09	7.38
3 NF5 1 0 0 0 0 0	2.12	17.05	5.34	13.52	2.63	17.28	7.87	20.35	1.33
4 F5 2 3 0 0 0 0	11.11	1.69	2.77	23.78	24.21	5.49	13.11	17.65	6.05
5 NF7 4 0 0 0 0 0	23.25	19.20	65.91	6.80	14.48	3.73	18.64	20.43	14.65
6 F7 5 6 7 0 0 0	63.52	2.77	25.98	10.09	58.69	4.42	60.38	39.79	77.97
7 NF8 5 0 0 0 0 0	20.48	6.49	19.62	6.29	11.80	3.58	15.76	45.32	28.63
8 F8 6 7 0 0 0 0	43.04	1.00	6.35	11.89	46.89	4.82	44.62	37.16	49.34
9 NF5+NF6 1 3 0 0 0 0	5.24	8.96	6.93	17.78	8.53	10.33	11.63	19.71	3.19
10 NF8+NF9 5 7 0 0 0 0	39.32	3.86	22.41	7.06	25.43	3.83	32.40	42.87	51.99
11 F6+F9 2 6 0 0 0 0	32.19	1.00	4.75	17.48	51.55	5.40	37.34	30.39	30.17

TABLE N° 2F27 : RED ORE SAMPLE N°2,-0.04+0.01mm GRAVITY PRECONCENTRATE, MATERIAL BALANCE OF FLOTATION TEST N°27.

PRODUCTS	WEIGHT	Fe203		SrO		BaO		RED TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F4	8.69	1.00	1.34	25.00	18.35	1.36	2.60	21.43	6.01
2 NF5	3.84	6.74	4.00	18.73	6.07	5.06	4.27	19.34	2.40
3 F6	8.52	1.00	1.32	17.73	12.76	1.31	2.46	36.08	9.92
4 NF6	6.76	1.96	2.05	14.92	8.52	2.44	3.63	32.81	7.16
5 NF7	16.12	13.41	33.41	13.67	18.61	16.66	59.09	10.27	5.34
6 NF8	14.48	17.21	38.51	8.86	10.83	5.50	17.52	18.99	8.88
7 F9	27.42	1.00	4.24	6.76	15.65	.52	3.14	50.76	44.92
8 NF9	14.17	6.91	15.13	7.70	9.21	2.34	7.30	33.61	15.37
CALCULATED HEAD	100.00	6.47	100.00	11.84	100.00	4.55	100.00	30.98	100.00

PRODUCTS	WEIGHT %	RED TOT. GRADE / SrO GRADE
1 F4	8.69	.8572
2 NF5	3.84	1.0326
3 F6	8.52	2.0350
4 NF6	6.76	2.1991
5 NF7	16.12	.7513
6 NF8	14.48	2.1433
7 F9	27.42	7.5089
8 NF9	14.17	4.3649
CALCULATED HEAD	100.00	2.6163

TABLE N° 2F27 : FLOTATION TEST N° 27.

PRODUCTS	WEIGHT	Fe2O3		SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF3 5 6 7 8 0 0	72.19	8.18	91.29	8.91	54.31	5.48	87.04	31.98	74.51
2 F1+F2+F3 1 2 3 4 0 0	27.81	2.03	8.71	19.46	45.69	2.12	12.96	28.40	25.49
3 NF4 2 3 4 0 0 0	19.12	2.49	7.36	16.94	27.35	2.46	10.36	31.56	19.48
4 F4 1 0 0 0 0 0	8.69	1.00	1.34	25.00	18.35	1.36	2.60	21.43	6.01
5 NF5 2 0 0 0 0 0	3.84	6.74	4.00	18.73	6.07	5.06	4.27	19.34	2.40
6 F5 3 4 0 0 0 0	15.28	1.42	3.36	16.49	21.27	1.81	6.08	34.63	17.08
7 NF7 5 0 0 0 0 0	16.12	13.41	33.41	13.67	18.61	16.66	59.09	10.27	5.34
8 F7 6 7 8 0 0 0	56.07	6.68	57.88	7.54	35.70	2.27	27.95	38.22	69.17
9 NF8 6 0 0 0 0 0	14.48	17.21	38.51	8.86	10.83	5.50	17.52	18.99	8.88
10 F8 7 8 0 0 0 0	41.59	3.01	19.37	7.08	24.87	1.14	10.43	44.92	60.29
11 F4+NF7 1 5 0 0 0 0	24.81	9.06	34.75	17.64	36.95	11.30	61.69	14.18	11.35
12 NF5+NF6 2 4 0 0 0 0	10.60	3.69	6.05	16.30	14.59	3.39	7.90	27.93	9.56
13 NF8+NF9 6 8 0 0 0 0	28.65	12.12	53.65	8.29	20.05	3.94	24.82	26.22	24.25
14 F6+F9 3 7 0 0 0 0	35.94	1.00	5.55	9.36	28.41	.71	5.59	47.28	54.84

TABLE N° 2F28 : REO ORE SAMPLE N°2,-0.04+0.01mm GRAVITY PRECONCENTRATE, MATERIAL BALANCE OF FLOTATION TEST N°28.

PRODUCTS	WEIGHT	Fe2O3		SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F4	4.79	1.00	.65	25.00	10.88	8.26	8.39	9.62	1.47
2 NF5	2.48	7.39	2.48	13.80	3.11	6.02	3.17	19.13	1.52
3 F6	10.81	1.00	1.47	24.00	23.58	7.66	17.56	25.53	8.83
4 NF6	8.77	1.47	1.75	23.62	18.82	7.34	13.65	19.56	5.49
5 NF7	12.82	27.66	48.07	4.01	4.67	3.37	9.16	18.21	7.47
6 NF8	16.42	14.58	32.45	5.63	8.40	3.57	12.43	28.04	14.73
7 F9	22.04	1.00	2.99	7.10	14.22	3.82	17.86	47.52	33.50
8 NF9	21.87	3.42	10.14	8.21	16.32	3.83	17.77	38.58	26.99
CALCULATED HEAD	100.00	7.38	100.00	11.00	100.00	4.71	100.00	31.26	100.00

PRODUCTS	WEIGHT %	REO TOT. GRADE / SrO GRADE
1 F4	4.79	.3848
2 NF5	2.48	1.3862
3 F6	10.81	1.0638
4 NF6	8.77	.8281
5 NF7	12.82	4.5411
6 NF8	16.42	4.9805
7 F9	22.04	6.6930
8 NF9	21.87	4.6991
CALCULATED HEAD	100.00	2.8407

TABLE N° 2F28 : FLOTATION TEST N° 28.

PRODUCTS	WEIGHT	Fe203		SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF3 5 6 7 8 0 0	73.15	9.44	93.65	6.56	43.61	3.69	57.22	35.34	82.69
2 F1+F2+F3 1 2 3 4 0 0	26.85	1.74	6.35	23.11	56.39	7.51	42.78	20.15	17.31
3 NF4 2 3 4 0 0 0	22.06	1.91	5.70	22.70	45.51	7.35	34.38	22.44	15.83
4 F4 1 0 0 0 0 0	4.79	1.00	.65	25.00	10.88	8.26	8.39	9.62	1.47
5 NF5 2 0 0 0 0 0	2.48	7.39	2.48	13.80	3.11	6.02	3.17	19.13	1.52
6 F5 3 4 0 0 0 0	19.58	1.21	3.21	23.83	42.40	7.52	31.22	22.86	14.32
7 NF7 5 0 0 0 0 0	12.82	27.66	48.07	4.01	4.67	3.37	9.16	18.21	7.47
8 F7 6 7 8 0 0 0	60.33	5.57	45.58	7.10	38.94	3.76	48.06	38.98	75.22
9 NF8 6 0 0 0 0 0	16.42	14.58	32.45	5.63	8.40	3.57	12.43	28.04	14.73
10 F8 7 8 0 0 0 0	43.91	2.21	13.13	7.65	30.54	3.82	35.63	43.07	60.50
11 F4+NF7 1 5 0 0 0 0	17.61	20.41	48.72	9.72	15.55	4.70	17.56	15.87	8.94
12 NF5+NF6 2 4 0 0 0 0	11.25	2.78	4.23	21.46	21.93	7.05	16.82	19.47	7.01
13 NF8+NF9 6 8 0 0 0 0	38.29	8.21	42.59	7.10	24.72	3.72	30.20	34.06	41.72
14 F6+F9 3 7 0 0 0 0	32.85	1.00	4.45	12.66	37.80	5.08	35.42	40.28	42.33

TABLE N° 2F29 : REE ORE SAMPLE N°2,-0.04+0.01mm GRAVITY PRECONCENTRATE, MATERIAL BALANCE OF FLOTATION TEST N°29.

PRODUCTS	WEIGHT	Fe203		SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1	34.61	14.87	71.99	18.45	50.67	8.50	63.90	13.02	13.37
2 F2	26.22	1.00	3.67	1.52	3.16	.67	3.82	65.75	51.15
3 NF3	9.78	8.53	11.67	20.25	15.71	5.15	10.94	11.45	3.32
4 NF4	16.12	4.80	10.82	22.00	28.14	5.60	19.61	14.30	6.84
5 F4	13.27	1.00	1.86	2.20	2.32	.60	1.73	64.30	25.32
CALCULATED HEAD	100.00	7.15	100.00	12.60	100.00	4.60	100.00	33.70	100.00

PRODUCTS	WEIGHT %	REO TOT. GRADE / SrO GRADE
1 NF1	34.61	.7057
2 F2	26.22	43.2566
3 NF3	9.78	.5654
4 NF4	16.12	.6500
5 F4	13.27	29.2273
CALCULATED HEAD	100.00	2.6743

TABLE N° 2F29 : FLOTATION TEST N° 29.

PRODUCTS	WEIGHT	Fe203		SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1 1 0 0 0 0 0	34.61	14.87	71.99	18.45	50.67	8.50	63.90	13.02	13.37
2 F1 2 3 4 5 0 0	65.39	3.06	28.01	9.51	49.33	2.54	36.10	44.65	86.63
3 NF1+NF3 1 3 0 0 0 0	44.39	13.47	83.65	18.85	66.38	7.76	74.85	12.67	16.69
4 F2+F3 2 4 5 0 0 0	55.61	2.10	16.35	7.62	33.62	2.08	25.15	50.49	83.31
5 NF1+NF3+NF4 1 3 4 0 0 0	60.51	11.16	94.48	19.69	94.52	7.19	94.45	13.11	23.53
6 F2+F4 2 5 0 0 0 0	39.49	1.00	5.52	1.75	5.48	.65	5.55	65.26	76.47
7 NF1+NF2 1 3 4 5 0 0	73.78	9.33	96.33	16.54	96.84	6.00	96.18	22.31	48.85
8 F2 2 0 0 0 0 0	26.22	1.00	3.67	1.52	3.16	.67	3.82	65.75	51.15

- stage 2: conditioning of cleaner tailings with CO_2 and P.E. P301 followed by rougher-cleaner flotations to separate a monazite concentrate n° 2 as a final float fraction, rougher tailings and cleaner middlings

- stage 3: conditioning of the scavenger tailings from stage 1, with CO_2 and P.E. P301 to separate a monazite concentrate n° 1 as a float fraction, by rougher-cleaner flotations, tailings and cleaner flotations, rougher tailings and cleaner-recleaner middlings.

Stage 2 was suppressed in flotation n° 22. A combination of CO_2 and H_2SO_4 was used as a depressant in stages 2 and 3 of flotation n° 23. A similar combination was used in flotation tests n° 24 and 25, except that H_3PO_4 or H_2SiF_6 were substituted for H_2SO_4. H_3PO_4 and H_2SiF_6 were also substituted for H_2SO_4 in stage 1.

A mixture of H_3PO_4 and MAP (Mono Ammonium Phosphate: $NH_4H_2PO_4$) was used in stage 1 of flotation test 26.

H_2SO_4 was substituted for CO_2 in stages 2 and 3 of flotation test n° 27, the same applied to flotation 28, except that H_3PO_4 was substituted for H_2SO_4 in stage 1.

- flowsheet IV (test n° 29).

Phosphoric ester EA 14-89 was added as a collector for monazite on a CO_2 containing pulp so as to depress most of the gangue minerals.

Final tails were separated in the rougher stage, the rougher concentrate was cleaned first in a CO_2 containing pulp then after addition of H_2SO_4 as a barite depressant, the final cleaner float constitued a monazite concentrate 1.

The non float fraction separated in the first cleaner stage was subjected to scavenger flotation using CO_2 as a gangue depressant and phosphoric ester EA 14-89 as a monazite collector to produce a float fraction enriched in monzazite, and final tailings.

Cleaner flotation of the monazite enriched fraction was accomplished on CO_2 then H_2SO_4 conditioned pulps to complete removal of $BaSO_4$. The second cleaner float constituted a monazite concentrate 2.

Material balances show that:

- excess of P.E. detrimentally affects selectivity, strontianite is floated together with monazite for high addition of P.E. (test n° 20), separation of iron bearing carbonates is also not satisfactory

- flotation of strontianite with sulfonate AP 825 in test n° 21 leads to poor selectivity, this could be attributed to poor cleaning of particle surfaces after flotation with P.E. Occurrence of residual P.E. causes monazite to be floated and also probably hinders adsorption of sulfonate on to the surface of strontianite particles

- strontianite flotation with sulfonate under slightly acidic pH results in heavy REO losses in strontianite tailings (float fractions) or middlings

- additions of H_3PO_4 and H_2SiF_6 are detrimental to selectivity: barite cannot be separated, this also applies to the mixture of MAP and H_3PO_4, evaluated in test n° 26. MAP was found to depress flotation of strontianite since weight recovery of the cleaner float F4 was nil after addition of MAP

- CO_2 is needed to depress strontianite in scavenger and cleaner flotations using P.E. as a collector for monazite. Comparison of tests 23 and 27 clearly reveals this major finding:

Test n°	Depressant	Monazite concentrate F9				
		total REO		SrO content %	BaO content %	Fe_2O_3 content %
		content %	recovery %			
23	CO_2-H_2SO_4	58.63	44.95	2.45	0.60	1.08
27	H_2SO_4	50.76	44.92	6.76	0.52	1.00

- the best concentration performance is achieved using P.E. EA 14-89 as a unique collector, CO_2 as a gangue depressant and H_2SO_4 as a complementary barite depressant. Collector requirement is very low: 50 g/t in the rougher stage, 20 g/t in the scavenger stage. High monazite concentrates are obtained at a monazite recovery which could probably be improved through retreatment of the middlings:

	Composite monazite concentrate F2+F4
Fe_2O_3 assay %	1.00
BaO assay %	0.65
SrO assay %	1.75
total REO content %	65.26
total REO recovery %	76.47
monazite content %	94.60

3.3. PRELIMINARY TESTS ON THE REO ORE SAMPLE N° 3

Tests were made on:

the - 125 + 10 μm (t) fraction
the - 10 μm (l) fraction which is high in REE

using the Agitair laboratory flotation cells. Flotation reagents used were the following:

- H_2SO_4 technical grade from Prolabo as a pH regulator and barite depressant
- $BaCl_2$ technical grade from Prolabo as a barite modifier (activator)
- Na silicate from Rhône-Poulenc, aqueous solution of density 1.33, with SiO_2/Na_2O= 3.4 to 3.5, as a slimes dispersant and silicates depressant
- citric acid, technical grade from Prolabo, as a fluorite and iron depressant
- CO_2, technical grade from L'Air Liquide, as a barite and fluorite modifier, as a carbonate depressant
- phosphoric ester collector: P 301 from Gerland-CECA, a detailed characterization is shown in chapter 4
- petroleum sulfonate AP 825, from American Cyanamid, as a barite collector.

3.3.1. Preliminary tests on the - 125 + 10 µm fraction

Attempts were made to separate fluorite and barite preconcentrates using:

- phosphoric ester P 301 as a fluorite collector
- petroleum sulfonate AP 825 as a barite collector, flotation products were also analyzed for La and Ce so as to determine the response of REE bearing minerals to flotation.

Operating conditions and flowsheets of flotation tests are shown in figures 3F01 to 3F03, relevant material balances are given in tables 3F01 to 3F03.

Material balances show that:

- barite is not satisfactorily depressed with H_2SO_4 when fluorite is floated with P.E. as a collector in a slightly acidic pulp
- selectivity of P.E. for fluorite improves in a CO_2 containing pulp
- multi-stage flotation with small additions of P.E. also leads to increased F contents in cleaner concentrates.

A maximum fluorite content of 93.7 % in cleaner concentrate F7 separated in flotation test 3 indicates the potentiel of the process based upon utilization of phosphoric esters as fluorite collectors. Further optimization studies could possibly lead to improved concentration performance.

As a preliminary result, a composite preconcentrate assaying 87.2 % fluorite was obtained at a fluorine recovery of 84.9 %.

- concentration of barite either as a non float fraction separated from the fluorite flotation stage or as a float fraction separated by flotation with a sulfonate collector, has led to poor concentration performance: maximum barite content in preconcentrates does not exceed 76.3 % at Ba recoveries of 42.9-44.7 %. However, it should be pointed out that these preconcentrates were not upgraded by cleaner flotation. The composite product F1+F2 separated in flotation test 3 assays 67.1 % $BaSO_4$ at a barite recovery of 83.25 %

- the highest concentration ratios for La and Ce were achieved in test 3. However, selectivity for separation of REE bearing minerals appears to be very poor either with sulfonate under alkaline pH or with P.E. under acidic pH. Difficulties encountered in REE separation can be attributed to improper conditions for flotation and poor liberation of REE minerals.

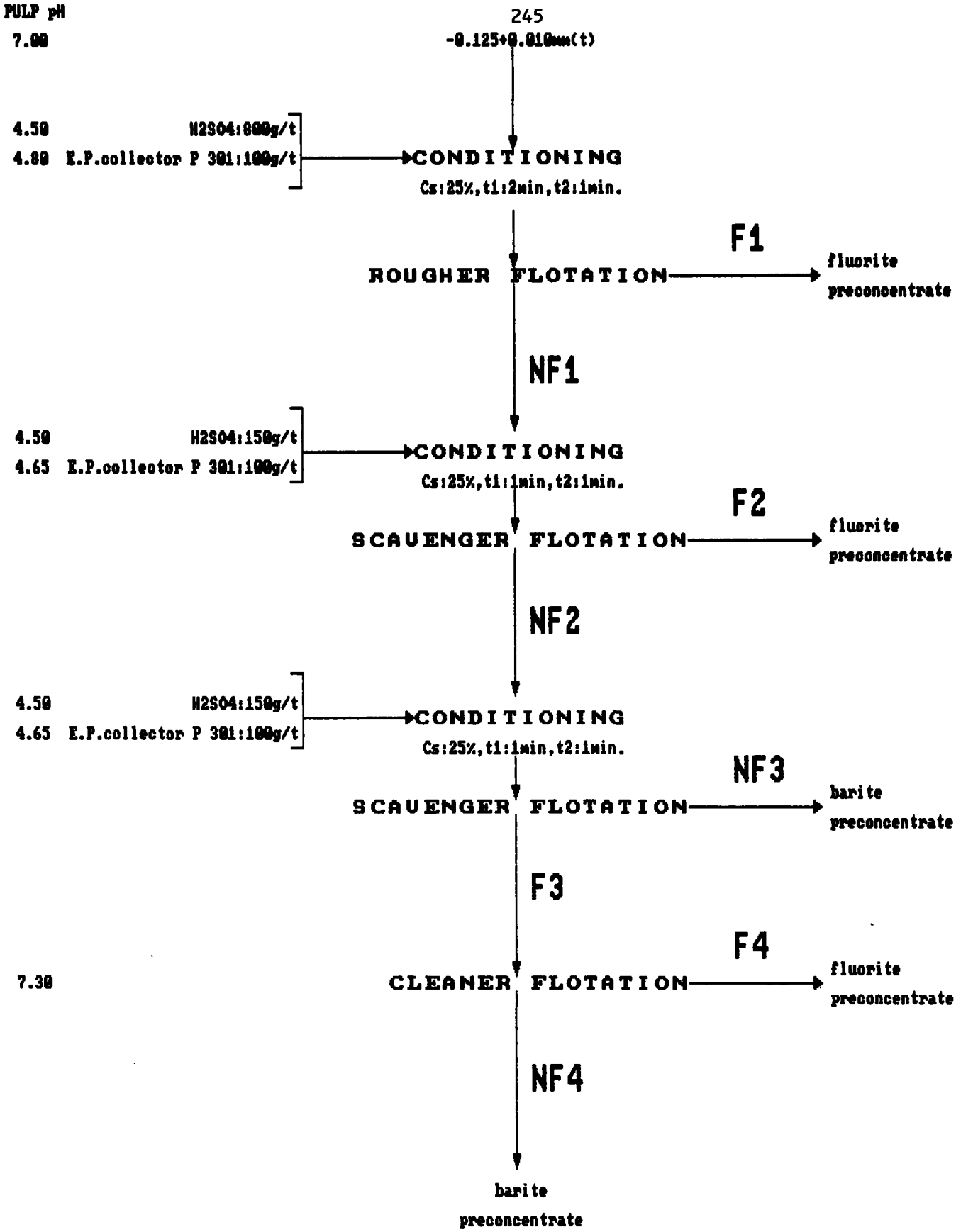


FIGURE N° 3F01 : FLOWSHEET OF FLOTATION N° 1 ON THE -0.125+0.010mm(t) FRACTION SEPARATED FROM THE REO ORE SAMPLE N°3.

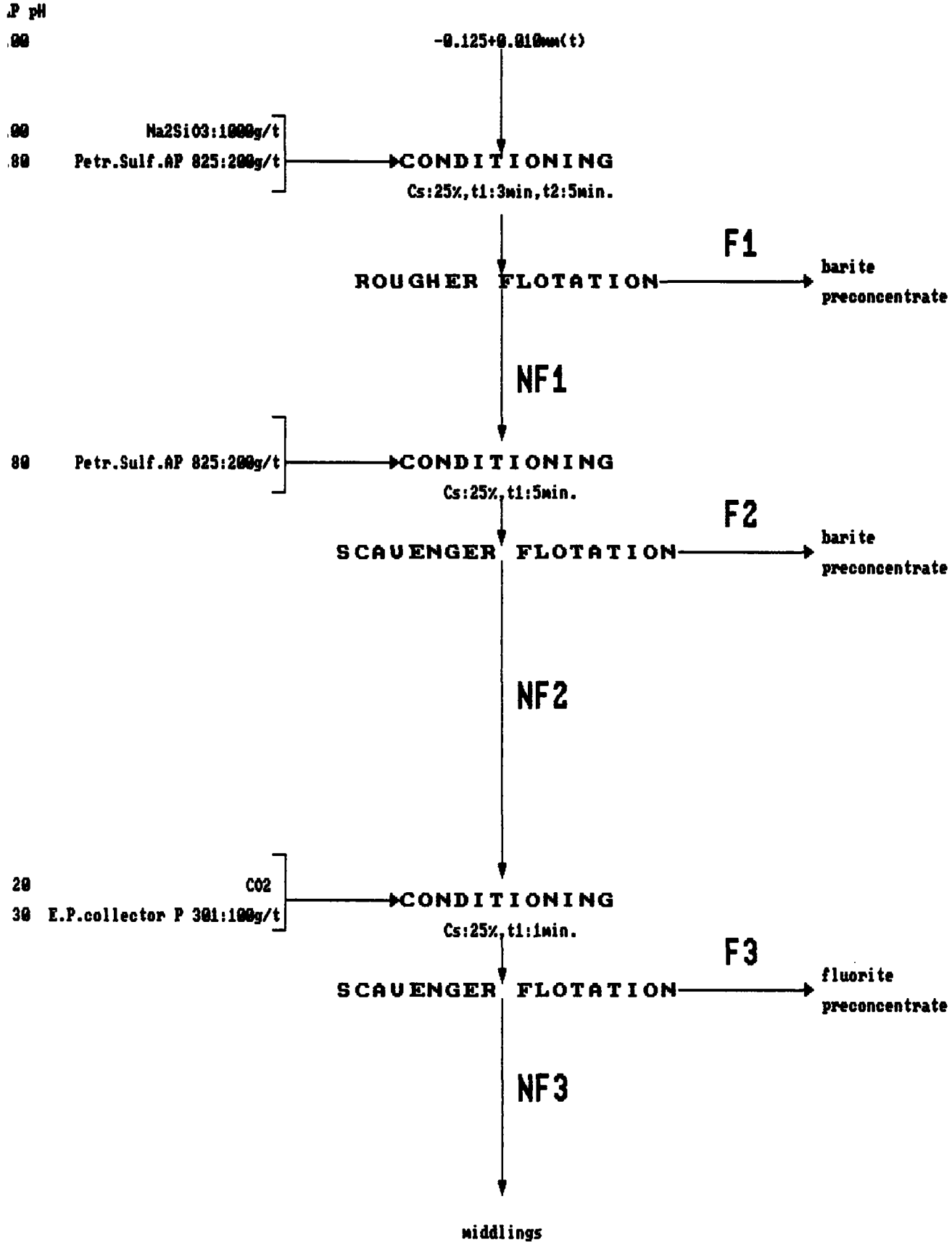


FIGURE N° 3F02 : FLOWSHEET OF FLOTATION N° 2 ON THE -0.125+0.010mm(t) FRACTION SEPARATED FROM THE REO ORE SAMPLE N°3.

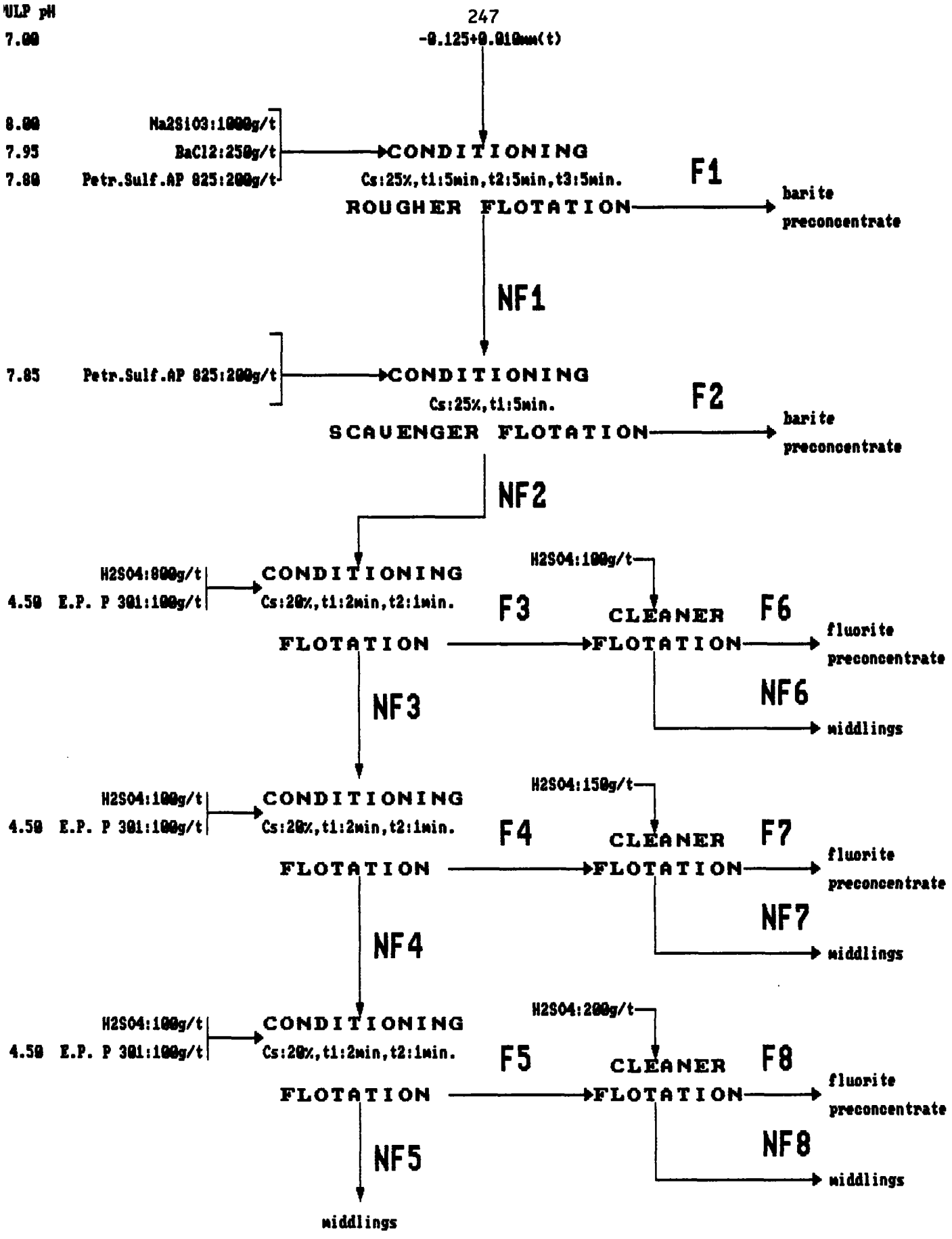


FIGURE N° 3F03 : FLOWSHEET OF FLOTATION TEST N° 3 ON THE -0.125+0.010mm(t) FRACTION SEPARATED FROM THE REO ORE SAMPLE N°3.

TABLE N° 3F01 : RED ORE SAMPLE N°3,MATERIAL BALANCE OF FLOTATION N°1
ON THE -.125+0.010mm(t) FRACTION SEPARATED FROM THE ORE GROUND DOWN TO 0.125mm.

PRODUCTS	WEIGHT	F		Ba		SiO2		La2O3	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1+F2	52.83	35.17	59.94	6.55	24.13	1.35	39.22	2.82	79.58
2 NF3	11.49	6.40	2.37	44.90	35.98	5.40	34.12	.89	5.46
3 F4	25.83	40.20	33.50	7.80	14.05	1.00	14.20	.71	9.80
4 NF4	9.85	13.20	4.19	37.60	25.83	2.30	12.46	.98	5.16
CALCULATED HEAD	100.00	31.00	100.00	14.34	100.00	1.82	100.00	1.87	100.00

PRODUCTS	WEIGHT	CeO2	
	%	GRADE %	RECOVERY
1 F1+F2	52.83	3.13	79.02
2 NF3	11.49	.91	5.00
3 F4	25.83	.83	10.24
4 NF4	9.85	1.22	5.74
CALCULATED HEAD	100.00	2.09	100.00

PRODUCTS	WEIGHT %	Ba GRADE / F GRADE
1 F1+F2	52.83	.1862
2 NF3	11.49	7.0156
3 F4	25.83	.1940
4 NF4	9.85	2.8485
CALCULATED HEAD	100.00	.4625

TABLE N° 3F01 : FLOTATION TEST N°1.

PRODUCTS	WEIGHT	F		Ba		SiO2		La2O3	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1+F2+F4 1 3 0 0 0 0	78.66	36.82	93.43	6.96	38.19	1.24	53.42	2.13	89.38
2 NF3+NF4 2 4 0 0 0 0	21.34	9.54	6.57	41.53	61.81	3.97	46.58	.93	10.62
3 F3 3 4 0 0 0 0	35.68	32.75	37.69	16.03	39.88	1.36	26.66	.78	14.95
4 NF2 2 3 4 0 0 0	47.17	26.33	40.06	23.06	75.87	2.34	60.78	.81	20.42

PRODUCTS	WEIGHT	CeO2	
	%	GRADE %	RECOVERY
1 F1+F2+F4 1 3 0 0 0 0	78.66	2.37	89.26
2 NF3+NF4 2 4 0 0 0 0	21.34	1.05	10.74
3 F3 3 4 0 0 0 0	35.68	.94	15.99
4 NF2 2 3 4 0 0 0	47.17	.93	20.98

TABLE N° 3F02 : RED ORE SAMPLE N°3,MATERIAL BALANCE OF FLOTATION N°2
ON THE -.125+0.010mm(t) FRACTION SEPARATED FROM THE ORE GROUND DOWN TO 0.125mm.

PRODUCTS	WEIGHT	F		Ba		SiO2		La2O3	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1	15.27	9.70	4.85	44.70	44.74	.75	6.29	1.00	8.19
2 F2	15.76	18.30	9.45	32.70	33.78	.90	7.79	1.62	13.69
3 F3	17.46	42.60	24.37	2.40	2.75	1.00	9.58	2.12	19.85
4 NF3	51.51	36.35	61.34	5.55	18.74	2.70	76.34	2.11	58.28
CALCULATED HEAD	100.00	30.53	100.00	15.26	100.00	1.82	100.00	1.87	100.00

PRODUCTS	WEIGHT	CeO2	
	%	GRADE %	RECOVERY
1 F1	15.27	1.37	9.99
2 F2	15.76	1.78	13.39
3 F3	17.46	2.26	18.84
4 NF3	51.51	2.35	57.79
CALCULATED HEAD	100.00	2.09	100.00

PRODUCTS	WEIGHT %	Ba GRADE / F GRADE
1 F1	15.27	4.6082
2 F2	15.76	1.7869
3 F3	17.46	.0563
4 NF3	51.51	.1527
CALCULATED HEAD	100.00	.4998

TABLE N° 3F02 : FLOTATION TEST N° 2.

PRODUCTS	WEIGHT	F		Ba		SiO2		La2O3	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1+F2 1 2 0 0 0 0	31.03	14.07	14.30	38.61	78.52	.83	14.07	1.31	21.88
2 NF2 3 4 0 0 0 0	68.97	37.93	85.70	4.75	21.48	2.27	85.93	2.11	78.12

PRODUCTS	WEIGHT	CeO2	
	%	GRADE %	RECOVERY
1 F1+F2 1 2 0 0 0 0	31.03	1.58	23.38
2 NF2 3 4 0 0 0 0	68.97	2.33	76.62

TABLE N° 3F03 : REO ORE SAMPLE N°3,MATERIAL BALANCE OF FLOTATION N°3
ON THE -.125+0.010mm(t) FRACTION SEPARATED FROM THE ORE GROUND DOWN TO 0.125mm.

PRODUCTS	WEIGHT	F		Ba		SiO2		La2O3	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
F1	14.71	8.75	4.09	44.90	42.91	.65	4.97	.98	7.72
F2	17.74	16.60	9.37	35.00	40.34	.90	8.30	1.51	14.35
F6	3.98	42.80	5.42	1.45	.37	1.35	2.79	2.66	5.67
F7	21.80	45.60	31.61	.85	1.20	1.40	15.87	1.46	17.05
F8	7.63	40.60	9.85	5.05	2.50	2.05	8.13	1.45	5.93
NF5+NF8	4.61	11.20	1.64	25.15	7.53	13.00	31.16	2.01	4.96
NF6	10.35	38.60	12.70	2.35	1.58	1.55	8.34	4.86	26.95
NF7	19.18	41.50	25.31	2.85	3.55	2.05	20.44	1.69	17.37
CALCULATED HEAD	100.00	31.45	100.00	15.39	100.00	1.92	100.00	1.87	100.00

PRODUCTS	WEIGHT	CeO2	
	%	GRADE %	RECOVERY
F1	14.71	1.31	9.21
F2	17.74	1.92	16.28
F6	3.98	2.79	5.31
F7	21.80	1.43	14.90
F8	7.63	1.64	5.98
NF5+NF8	4.61	2.32	5.11
NF6	10.35	5.05	24.98
NF7	19.18	1.99	18.24
CALCULATED HEAD	100.00	2.09	100.00

PRODUCTS	WEIGHT %	Ba GRADE / F GRADE
F1	14.71	5.1314
F2	17.74	2.1084
F6	3.98	.0339
F7	21.80	.0186
F8	7.63	.1244
NF5+NF8	4.61	2.2455
NF6	10.35	.0609
NF7	19.18	.0687
CALCULATED HEAD	100.00	.4895

TABLE N° 3F03 : FLOTATION TEST N°3.

PRODUCTS	WEIGHT	F		Ba		SiO2		La2O3	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1+F2 1 2 0 0 0 0	32.45	13.04	13.46	39.49	83.25	.79	13.27	1.27	22.07
2 NF2 3 4 5 6 7 8	67.55	40.29	86.54	3.82	16.75	2.47	86.73	2.15	77.93
3 F3 3 7 0 0 0 0	14.33	39.77	18.12	2.10	1.96	1.49	11.13	4.25	32.62
4 NF3 4 8 5 6 0 0	53.22	40.43	68.42	4.28	14.79	2.73	75.60	1.59	45.31
5 F4 4 8 0 0 0 0	40.98	43.68	56.93	1.79	4.76	1.70	36.31	1.57	34.42
6 NF4 6 5 0 0 0 0	12.24	29.53	11.49	12.62	10.04	6.17	39.29	1.66	10.89
7 F6 to F8+NF7 3 4 5 8 0 0	52.59	43.17	72.19	2.23	7.63	1.73	47.23	1.63	46.01
8 NF5+NF6+NF8 6 7 0 0 0 0	14.96	30.16	14.35	9.38	9.11	5.08	39.50	3.98	31.91
9 F6toF8+NF6+NF7 3 4 5 7 8 0	62.94	42.42	84.90	2.25	9.21	1.70	55.57	2.16	72.96

PRODUCTS	WEIGHT	CeO2	
	%	GRADE %	RECOVERY
1 F1+F2 1 2 0 0 0 0	32.45	1.64	25.49
2 NF2 3 4 5 6 7 8	67.55	2.31	74.51
3 F3 3 7 0 0 0 0	14.33	4.42	30.28
4 NF3 4 8 5 6 0 0	53.22	1.74	44.23
5 F4 4 8 0 0 0 0	40.98	1.69	33.14
6 NF4 6 5 0 0 0 0	12.24	1.90	11.09
7 F6 to F8+NF7 3 4 5 8 0 0	52.59	1.77	44.42
8 NF5+NF6+NF8 6 7 0 0 0 0	14.96	4.21	30.09
9 F6toF8+NF6+NF7 3 4 5 7 8 0	62.94	2.31	69.40

3.3.2. Preliminary tests on the - 10 µm primary fraction

Due to the fine size of the crystals which constitute the bastnaesite particles with a loose texture, a major proportion of the REE reports to the - 10 µm (1) slimes generated by attrition-scrubbing, this fraction is high in REE since La_2O_3 and CeO_2 contents are 13.1 and 14.7 %.

Attempts were made to separate REE minerals from fluorite using phosphoric ester P301 as a collector, Na_2SiO_3 as a dispersant, citric acid as a fluorite depressant. Flotation was effected at a pulp pH of 4.5 adjusted with H_2SO_4 (test n° 5), at a pulp pH of 7 (natural pH, after addition of Na_2SiO_3, citric acid and P.E. test n° 7). Determinations of La, Ce in flotation products showed no significant differences, the largest differences were obtained in flotation test n° 6.

The basic flowheet of test n° 6 is shown in figure n° 3F04, the relevant material balance in table 3F04. It can be seen that no separation has occurred in terms of fluorite, barite, silicates or REE bearing minerals, this could result from the extremely fine distribution of the material (median diameter d50= 2.7 µm, d80= 4.8 µm, d20= 1.5 µm) which detrimentally affects selectivity of flotation.

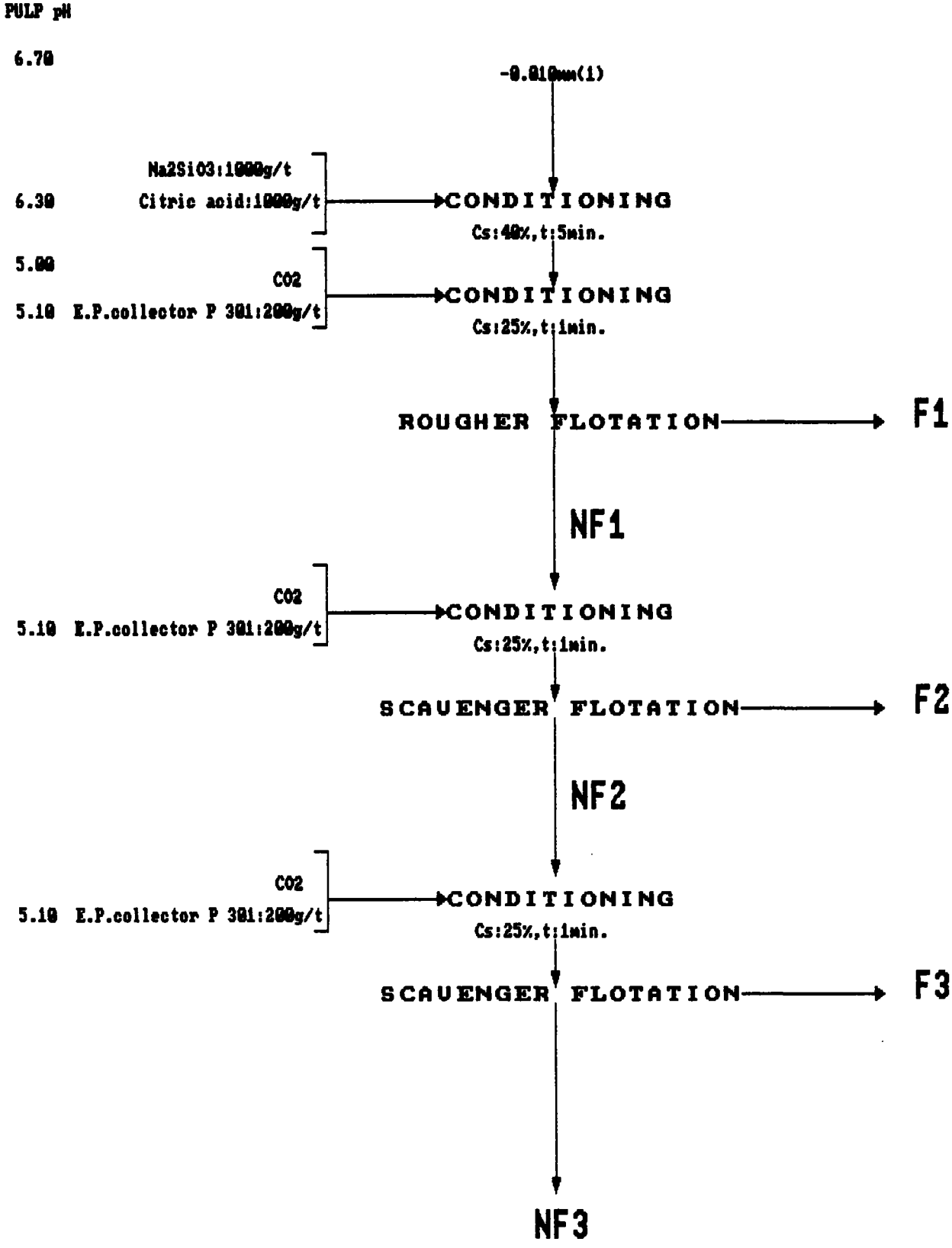


FIGURE N° 3F04 : FLOWSHEET OF FLOTATION N° 6 ON THE PRIMARY SLIMES FRACTION SEPARATED FROM THE REO ORE SAMPLE N°3.

TABLE N° 3F04 :RED ORE SAMPLE N°3 GROUND DOWN TO 0.125mm,MATERIAL BALANCE OF FLOTATION N°6 ON THE -0.010mm(1) SLIMES.

PRODUCTS	WEIGHT	F		Ba		SiO2		La2O3	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1+F2	39.59	20.20	38.40	1.90	38.35	4.30	38.85	13.60	41.01
2 F3	19.85	21.10	20.11	1.80	18.22	4.20	19.03	13.37	20.22
3 NF3	40.56	21.30	41.49	2.10	43.43	4.55	42.12	12.55	38.77
CALCULATED HEAD	100.00	20.82	100.00	1.96	100.00	4.38	100.00	13.13	100.00

PRODUCTS	WEIGHT	CeO2	
	%	GRADE %	RECOVERY
1 F1+F2	39.59	15.36	41.34
2 F3	19.85	15.11	20.39
3 NF3	40.56	13.88	38.27
CALCULATED HEAD	100.00	14.71	100.00

PRODUCTS	WEIGHT %	Ba GRADE / F GRADE
1 F1+F2	39.59	.0941
2 F3	19.85	.0853
3 NF3	40.56	.0986
CALCULATED HEAD	100.00	.0942

TABLE N° 3F04 : FLOTATION TEST N°6.(-0.010mm(1)).

PRODUCTS	WEIGHT	F		Ba		SiO2		La2O3	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 F1+F2 1 0 0 0 0 0	39.59	20.20	38.40	1.90	38.35	4.30	38.85	13.60	41.01
2 NF2 2 3 0 0 0 0	60.41	21.23	61.60	2.00	61.65	4.43	61.15	12.82	58.99

PRODUCTS	WEIGHT	CeO2	
	%	GRADE %	RECOVERY
1 F1+F2 1 0 0 0 0 0	39.59	15.36	41.34
2 NF2 2 3 0 0 0 0	60.41	14.28	58.66

3.3.3. Comparative separation tests using gravimetry on the - 125 + 10 μm fraction

Gravity concentration tests using the centrifugal separator M.G.S. (Multi Gravity Separator) from Mozley were conducted on the - 125 + 50 μm and - 50 + 10 μm fractions separated from the - 125 + 10 μm material, the objectives of the tests were:

- to determine the amenability of the material to gravity separation
- to compare concentration performances achieved by gravity and flotation.

Weight recoveries were: 39.2 % in - 125 + 50 μm and 60.8 % in - 50 + 10 μm fraction.

It is interesting to note that values of specific gravity for the main minerals to be separated are as follows:

barite:	4.4 to 4.5 g/cm^3
bastnaesite:	4.9 to 5.2 g/cm^3
fluorite:	3.1 to 3.2 g/cm^3
silicates:	2.6 to 3.3. g/cm^3

The concentration criterion relevant to barite-fluorite separation is low= 1.60, this should be compared with minimum values given by the standard curve, (2.25 at 120 μm, 2.65 at 65 μm, 3.35 at 10 μm), corresponding to values at which gravity separation becomes virtually impossible, using conventional gravity concentration equipments operated under the gravity field of forces.

Basic flowsheets of gravity separation tests are shown in figures 3G01 and 3G02, the M.G.S. separator (first stage) was operated under the following conditions:

	-125 +50 μm	-50 +10 μm
S.R.: speed of rotation of the drum r.p.m.	150	168
Fcy : frequency of stroke s.p.m.	300	300
slope of the separating drum, degrees	6.5	6.5
Cs : solids concentration in feed pulp %	18.7	23.3
QF : flow rate of feed pulp l/min	3.5	3.5
QW : flow rate of wash water l/min	8.2	8.5

Separations were accomplished in two stages, the light material separated in stage 1 was deslimed at about 10 μm, using a 25 mm inner diameter hydrocyclone under an inlet pressure of 2 bars, middlings and redeslimed lights were combined and treated in stage 2.

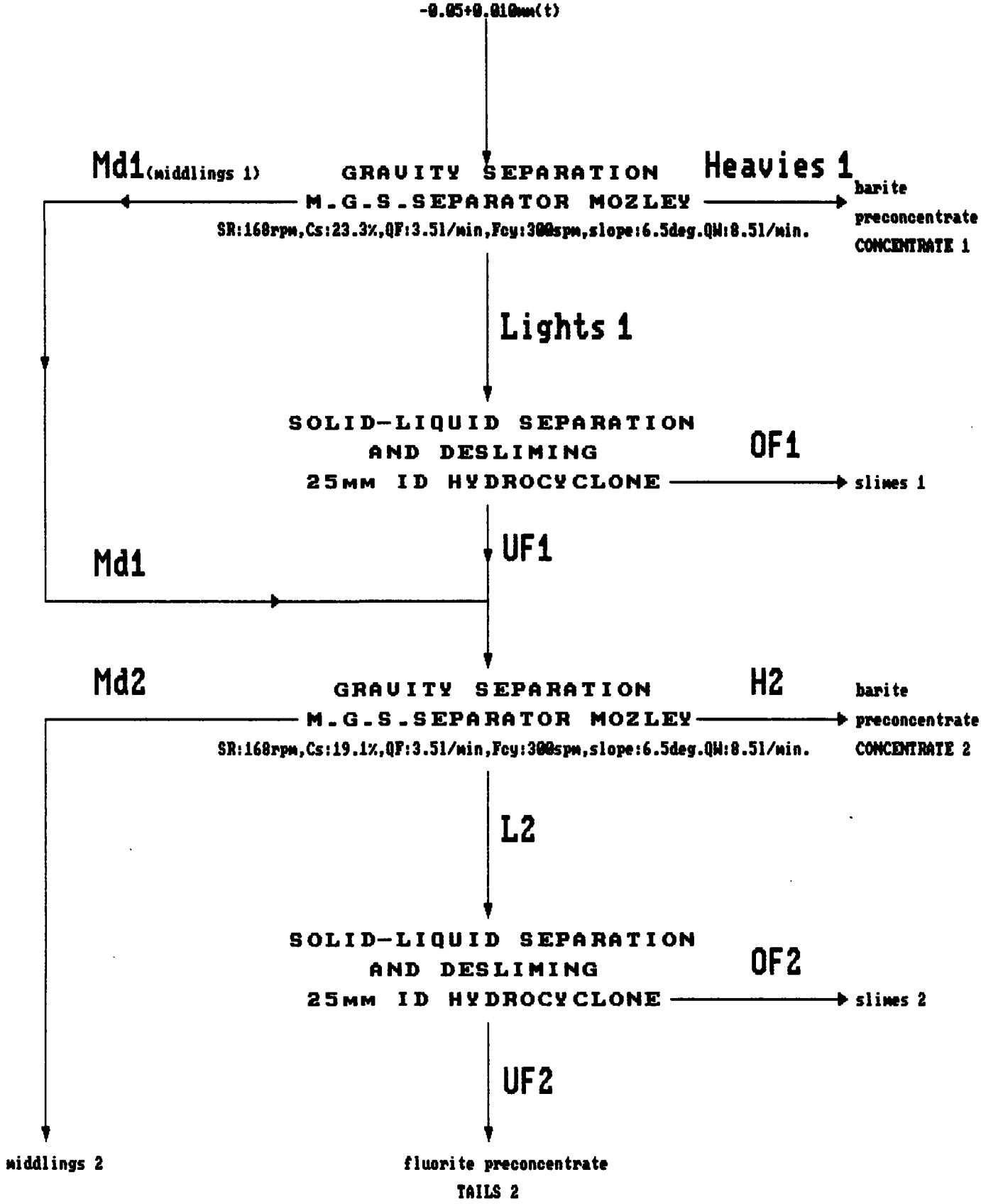


FIGURE N° 3G01 : FLOWSHEET OF GRAVITY SEPARATION TESTS ON THE -0.05+0.01mm(t) FRACTION SEPARATED FROM THE REO ORE SAMPLE N°3 GROUND DOWN TO 0.125mm.

TABLE N° 3G01 : RED ORE SAMPLE N°3,GROUND DOWN TO 0.125mm,MATERIAL BALANCE FOR GRAVITY CONCENTRATION TEST ON THE-.125+0.05mm(t),USING THE M.G.S.

PRODUCTS	WEIGHT	F		Ba		La203		CeO2	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 CONCENTRATE 1	6.76	11.40	2.21	41.45	24.08	.72	7.89	.81	7.16
2 CONCENTRATE 2	1.52	16.90	.74	34.85	4.55	.60	1.48	.81	1.61
3 MIDDLINGS 2	7.49	17.40	3.73	34.40	22.14	.66	8.02	.78	7.63
4 TAILS 2	78.36	38.60	86.68	6.90	46.47	.59	74.99	.74	75.78
5 SLIMES 1+2	5.87	39.50	6.64	5.45	2.75	.80	7.62	1.02	7.82
CALCULATED HEAD	100.00	34.90	100.00	11.64	100.00	.62	100.00	.77	100.00

PRODUCTS	WEIGHT %	Ba GRADE / F GRADE
1 CONCENTRATE 1	6.76	3.6360
2 CONCENTRATE 2	1.52	2.0621
3 MIDDLINGS 2	7.49	1.9770
4 TAILS 2	78.36	.1788
5 SLIMES 1+2	5.87	.1380
CALCULATED HEAD	100.00	.3334

TABLE N° 3G01 :GRAVITY CONCENTRATION TEST N°1.(-0.125+0.05mm(t)).

PRODUCTS	WEIGHT	F		Ba		La203		CeO2	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 C1+C2+MID. 1 2 3 0 0 0	15.77	14.78	6.68	37.47	50.78	.68	17.39	.80	16.40
2 TAILS+SLIM. 4 5 0 0 0 0	84.23	38.66	93.32	6.80	49.22	.60	82.61	.76	83.60

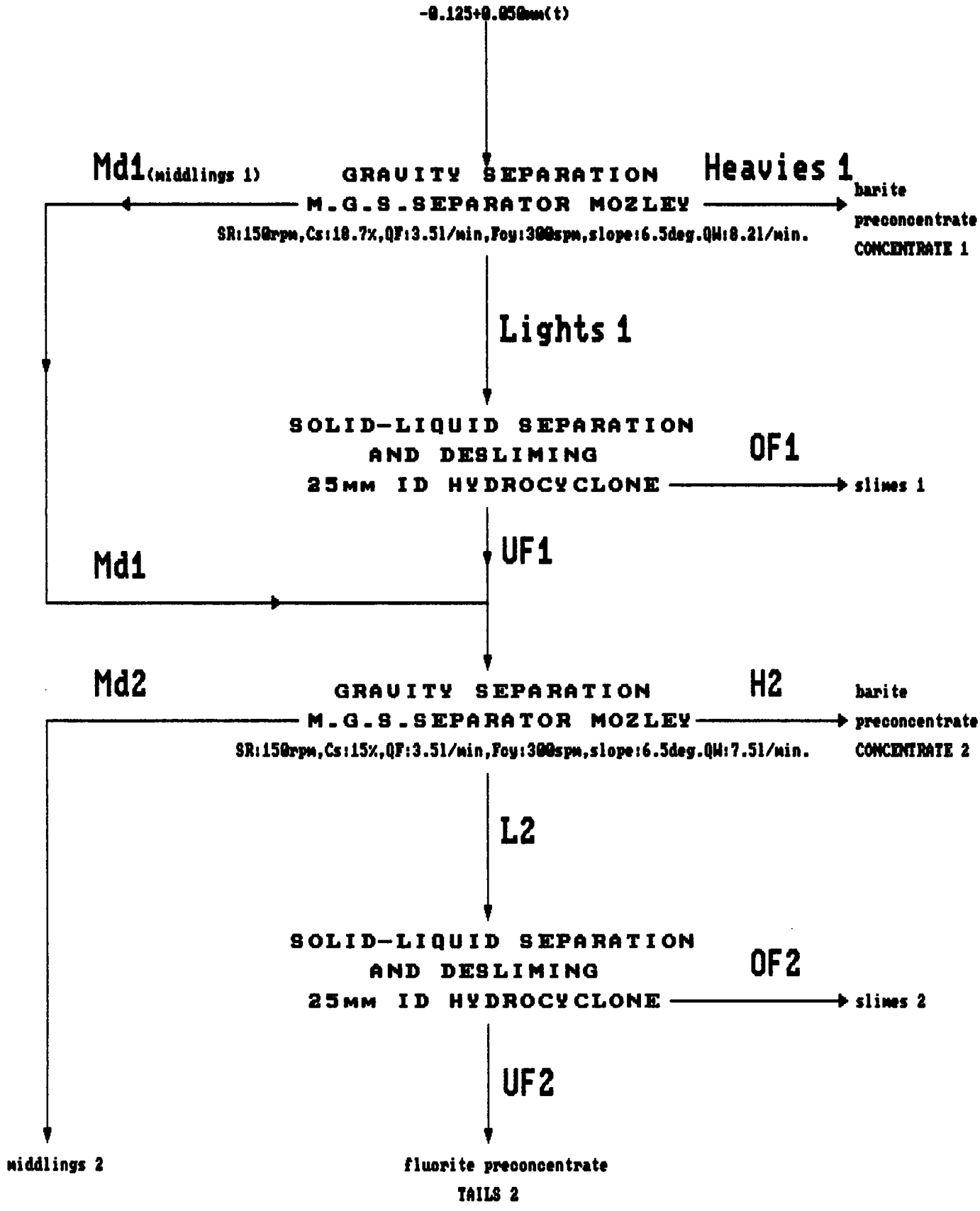


FIGURE N° 3G02 : FLOWSHEET OF GRAVITY SEPARATION TESTS ON THE -0.125+0.05mm(t) FRACTION SEPARATED FROM THE REO ORE SAMPLE N°3 GROUND DOWN TO 0.125mm.

TABLE N° 3G02 : RED ORE SAMPLE N°3,GROUND DOWN TO 0.125mm,MATERIAL BALANCE FOR GRAVITY CONCENTRATION TEST ON THE-0.05+0.01mm(t),USING THE M.G.S.

PRODUCTS	WEIGHT	F		Ba		La203		CeO2	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 CONCENTRATE 1	21.75	5.50	4.28	49.10	61.37	.75	6.11	.89	6.56
2 CONCENTRATE 2	2.50	8.15	.73	44.50	6.39	1.15	1.08	1.39	1.18
3 MIDDLINGS 2	3.57	14.20	1.82	35.45	7.27	2.23	2.98	2.45	2.96
4 TAILS 2	62.67	37.20	83.50	5.80	20.89	2.86	67.11	3.19	67.76
5 SLIMES 1+2	9.51	28.40	9.67	7.45	4.07	6.38	22.72	6.68	21.53
CALCULATED HEAD	100.00	27.92	100.00	17.40	100.00	2.67	100.00	2.95	100.00

PRODUCTS	WEIGHT %	Ba GRADE / F GRADE
1 CONCENTRATE 1	21.75	8.9273
2 CONCENTRATE 2	2.50	5.4601
3 MIDDLINGS 2	3.57	2.4965
4 TAILS 2	62.67	.1559
5 SLIMES 1+2	9.51	.2623
CALCULATED HEAD	100.00	.6232

TABLE N° 3G02 : GRAVITY CONCENTRATION TEST N°2.(-0.05+0.01mm(t)).

PRODUCTS	WEIGHT	F		Ba		La203		CeO2	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 C1+C2 1 2 0 0 0 0	24.25	5.77	5.01	48.63	67.77	.79	7.18	.94	7.74
2 TAILS 2 4 0 0 0 0 0	62.67	37.20	83.50	5.80	20.89	2.86	67.11	3.19	67.76
3 MID.+SLIM. 3 5 0 0 0 0	13.08	24.52	11.49	15.09	11.34	5.25	25.70	5.53	24.50

Materials balances, shown in tables 3G01 and 3G02, reveal that:

- the fine fraction - 50 + 10 μm is higher in REE and barite

- no separation of La or Ce occurs in treating the coarse fraction - 125 + 50 μm. Lack of liberation of REE bearing minerals or high porosity of REE containing particles (as porous aggregates), is shown by increasing La and Ce contents with decreasing specific gravities of fractions separated from the - 50 + 10 μm fraction. It can also be seen that higher La and Ce contents in slimes account for the low strength of the REE containing aggregates and small sizes of the crystals which constitute the aggregates

- significant improvements in barite-fluorite separations occur in the - 50 + 10 μm fraction, this is probably related to better liberation of minerals in the fine sized fraction.

	F		Ba	
	content %	recovery %	content %	recovery %
- 50 + 10 μm concentrate	5.77	5.01	48.63	67.77
- 50 + 10 μm tails	37.20	83.50	5.80	20.89
- 50 + 10 μm middlings + slimes	24.52	11.49	15.09	11.34
- 125 + 50 μm concentrate	12.43	2.95	40.25	28.63
- 125 + 50 μm tails	38.60	86.68	6.90	46.47
- 125 + 50 μm middlings + slimes	27.09	10.37	21.68	24.89
- 125 + 50 μm concentrate + middlings	14.78	6.68	37.47	50.78
- 125 + 50 μm tails + slimes	38.66	93.32	6.80	49.22

The composite fluorite preconcentrate - 125 + 10 μm, separated by gravity assays 77.75 % fluorite at a fluorine recovery of 87.87 %, the composite barite preconcentrate - 125 + 10 μm, separated by gravity, assays 77.1 % barite at a barite recovery of 62.64 %. Concentration performance for fluorite, achieved by gravity is inferior to that achieved by flotation, the reverse applies to barite.

4. CHARACTERIZATION AND EVALUATION OF PHOSPHORIC ESTER PROMOTERS

Phosphoric ester promoters were synthetized and characterized by CECA (ex Gerland-SHPC). Preliminary flotation tests conducted at BRGM laboratories led to the definition of basic flowsheets which were applied to REO ore samples n° 1 and n° 2 with the purpose to:

- compare P.E. collectors in terms of selectivity and concentration performances for REE bearing minerals: REE and Y containing apatite in REO ore n° 1, monazite in REO ore n° 2
- determine characteristics of the P.E. collectors which affect flotation performance for REE minerals.

4.1. CHARACTERIZATION OF PHOSPHORIC ESTERS PROMOTERS

Synthesis of phosphoric ester collectors involves phosphatation of alcohol, ether or phenol, including o to n oxialkylene groups, with $POCl_3$, PCl_5, PCl_3 P_2O_5, H_3PO_4 or condensed phosphoric acids. Phosphoric ester promoters are complex mixtures of mono and diesters:

where: - r is an hydrophobic radical: alkyl, aryl or alkyl - aryl
- (K)n are oxialkylene groups (alkyloxi).

associated with residual acids or non ionic products which affect frothing properties and collecting power: residue of phosphatation agents (pyrophosphoric and phosphoric acids, as examples), residues of alcohol or alkylphenol, esters of pyro or meta phosphoric acids ...

Frothing and collection properties, hydrophobicity of the collector coating, as well as selectivity of adsorption, can be affected by numerous parameters, such as the nature of the R chain: linear chain, chains including aromatic rings, branched chains, distribution of molecular weights (or lengths) of initial alcohols subjected to phosphatation (fatty and synthetic alcohols), the nature and number of the oxialkylene groups, the nature and proportions of the residual products, the relative proportions of mono and diesters. Most of these parameters strongly depend on the operating conditions followed in the phosphatation of the initial raw material (alcohol, alkyl-phenol ...) as well as conditions used for purifying the reaction products.

Samples of phosphoric esters submitted for flotation tests were produced by C.E.C.A.-S.H.P.C. at laboratory scale, using proprietary procedures which led to control the basic parameters such as the ratio monoester/diester, the proportions of residual non phosphated substances and free acids.

Ten phosphoric ester collectors were tested and evaluated:

collector	coded composition	order for flotation tests: REO ore 1	order for flotation tests: REO ore 2
MELIORAN D2-45E	D2-45E	1	1
MELIORAN D7-61E	D7-61E	2	2
PE 89124	IM7-58		3
P312	D9 PS2*	3	4
B110	D4-92	4	5
B110 88094	D4-70E	5	6
MELIORAN T4-55E	T4-55E	6	7
P301	T6 PS**	7	8
B110 69032	D4-66	8	
PE 89122	EA14-89		9

* or D9-50 ** or T6-59

Coded composition appears as: Xn - Y-E where:

X: is the hydrophobic radical
n: is the number of ethylene oxide groups
Y: is the percentage of monoester on the basis of 100 % for the total of the orthoesters
E: is added to coded composition of collectors with low residual non phosphated matter (most of the reagents withlow non phosphated matter were refined to separate the residical impurities)
X: corresponds to the initial alcohol or alcohol derivative which is the most abundant in the initial material to be phosphatated
D: means Dodecyl alcohol
T: Tridecylic alcohol
IM: Isomyristic alcohol
EA: Ether alcohol (C_{21} alcohol).

A number of analytical and separation technique were used to determine mono and di orthophosphoric esters, pyrophosphoric esters, free orthophosphoric acid, free pyrophosphoric acid, residual non phosphated substances, non ionic products, total P_2O_5:

- separations by ion exchange resins and by organic solvents ($CHCl_3$ + Bu OH/H_2O)
- potentiometry, liquid and gas phases chromatography, colorimetry, high pressure liquid chromatography.

Standard procedures are referenced as CFD 110 - A - 157, and CFD 110 - A - 163 methods.

The main analytical procedures used by CECA-GERLAND laboratories are described in the appendix 5.

Characteristics of the ten P.E. collectors are shown in tables 8 and 9.

	MELIORAN D2-45E	MELIORAN D7-61E	PE 89124	P312	B110
R	L.A.	L.A.	B.S.A.	L.A.	L.A.
X	D	D	I.M.	D	D
K	E.O.	E.O.	E.O.	E.O.	E.O.
NbC	12	12	14	12	12
n	2	7	7	9	4
Mono Ester Orthophosphoric %	34.6	51.2	51.0	43.6	71.9
Di Ester Orthophosphoric %	41.3	32.3	34.3	43.2	6.5
Non Phosphated Products %	4.5	7.7	15.7	9.4	23.2
Free H_3PO_4 %	1.1	1.5	0.8	0.7	3.5
Free $H_4P_2O_7$ %	1.2	0.8	0.2	0.2	0
Mono Ester Pyrophosphoric %	11.7	14.1	2.7	3.0	0
Di Ester Pyrosphoric symmetrical %	16.3	8.9	4.5	4.3	0
Refining procedure	Yes	Yes	No	No	No
Coded composition	D2-45E	D7-61E	IM7-58	D9 PS2 or D9-50	D4-92

Table n° 8 : Characteristics of Phosphoric Ester collectors

	B110 88094	MELIORAN T4-55E	PE 301	B110 69032	PE89122 B110
R	L.A.	B.S.A.	B.S.A.	L.A.	E.A.
X	D	T	T	D	E.A.
K	E.O.	E.O.	E.O.	E.O.	E.O.
NbC	12	13	13	12	21
n	4	4	6	4	14
Mono Ester Orthophosphoric %	63.0	46.3	46.9	54.5	44.
Di Ester Orthophosphoric %	24.9	31.0	32.2	25.9	6.3
Non Phosphated Products %	4.5	4.8	15.6	20.3	33.6
Free H_3PO_4 %	2.3	1.2	0.7	1.7	1.1
Free $H_4P_2O_7$ %	0.5	1.0	0.8	0.1	0.9
Mono Ester Pyrophosphoric %	4.9	7.4	5.9	1.5	8.7
Di Ester Pyrophosphoric symmetrical %	3.7	13.5	2.6	2.9	0.5
Refining procedure	Yes	Yes	No	No	No
Coded composition	D4-70E	T4-55E	T6-PS or T6-59	D4-66	EA14-89

Table n° 9: Characteristics of Phosphoric Ester collectors

R: describes the nature of the hydrophobic radical where:

- L.A.: Linear fatty Alcohol
- B.S.A.: Branched Chain, synthetic Alcohol
- E.A.: Ether Alcohol

n: statistical number of Ethylene Oxide groups (E.O.)
NbC: Number of Carbons in the hydrophobic Chain
MEO: content in Mono Ester Orthophosphoric
DEO: content in Di Ester Orthophosphoric
NPP: content in Non Phosphated Products
FOPA: content in Free Orthosphoric Acid
FFPA: content in Free Pyrophosphoric Acid
MEP: content in Mono Ester Pyrophosphoric
DEP: content in DiEster Pyrophosphoric (symmetrical).

Variables describing the main characteristics of P.E. promoters have been used in the analysis of flotation data.

4.2. COMPARATIVE FLOTATION TESTS ON REO ORE SAMPLE N° 1 WITH PHOSPHORIC ESTERS AS COLLECTORS

4.2.1. Flotation test results

Flowsheet used for comparative tests was similar to the type 2b flowsheet used in preliminary flotation tests except that a solution of Na silicate and Na citrate (in a 5/1 weight ratio) was added as an iron depressant at the rougher flotation stage, scavenger stage and cleaner stages, with the purpose to improve selectivity of apatite flotation in all separation stages.

Flowsheet used in flotation tests 30 to 37 is shown in figure 1F30.

Flotation products were analyzed for P_2O_5, concentrates F3 and F4 corresponding to the best concentration efficiencies were analyzed for P_2O_5, CaO, SiO_2, Fe_2O_3, Al_2O_3, MgO, LOI, Y and REE.

Material balances relevant to flotation tests 30 to 37 are shown in tables 10 and 11.

Flotation performances of the P.E. collectors tested are represented as grade-recovery curves for P_2O_5 in figure n° 4.

It can be seen that P.E. collector B110 88094 leads to the best concentration performances while the worst was achieved with P312.

It should be pointed out that addition of the Na silicate-citrate depressant prior to each of the flotation stages significantly improves selectivity with respect to previous tests.

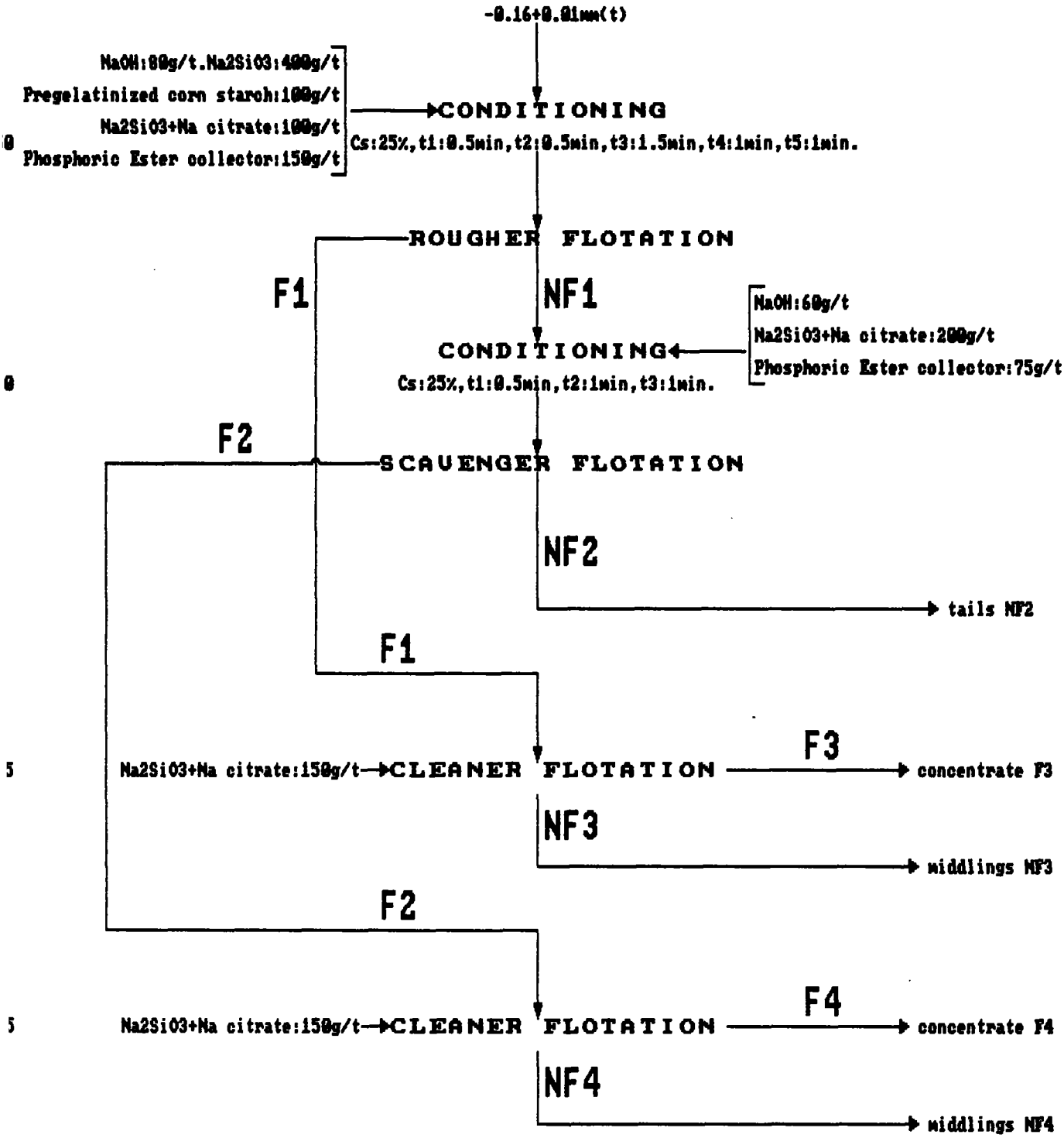


figure n° 1F30 : basic flowsheet used for the evaluation of phosphoric ester promoters on the REO ore sample n°1.

flotation test	P.E. collector	weight %	P2O5 content %	P2O5 recovery %
n° 30	n° 5			
NF2	B110	83.72	0.28	6.90
F3	88094	6.63	33.10	64.54
NF3		3.40	8.50	8.51
F4	D4-70E	2.15	27.50	17.39
NF4		4.10	2.20	2.65
calculated head		100.00	3.40	100.00
F3 + F4		8.78	31.73	81.93
n° 31	n° 4			
NF2	B110	83.95	0.63	16.12
F3		6.38	31.95	62.15
NF3		4.18	7.81	9.94
F4	D4-92	1.15	27.00	9.47
NF4		4.34	1.75	2.32
calculated head		100.00	3.28	100.00
F3 + F4		7.53	31.19	71.60
n° 32	n° 8			
NF2	B110	82.51	0.37	9.13
F3	69032	6.42	32.40	62.21
NF3		3.79	8.35	9.47
F4	D4-66	1.95	28.30	16.51
NF4		5.33	1.68	2.68
calculated head		100.00	3.34	100.00
F3 + F4		8.37	31.44	78.72
n° 33	n° 6			
NF2	MELIORAN	80.92	0.35	8.26
F3		6.10	32.15	57.18
NF3		3.55	6.62	6.85
F4	T4-55E	2.35	30.33	20.78
NF4		7.08	3.36	6.93
calculated head		100.00	3.43	100.00
F3 + F4		8.45	31.64	77.96
n° 34	n° 1			
NF2	MELIORAN	87.13	1.12	29.39
F3		5.15	32.70	50.72
NF3		2.63	7.75	6.13
F4	D2-45E	1.35	27.75	11.29
NF4		3.74	2.19	2.47
calculated head		100.00	3.32	100.00
F3 + F4		6.50	31.67	62.01

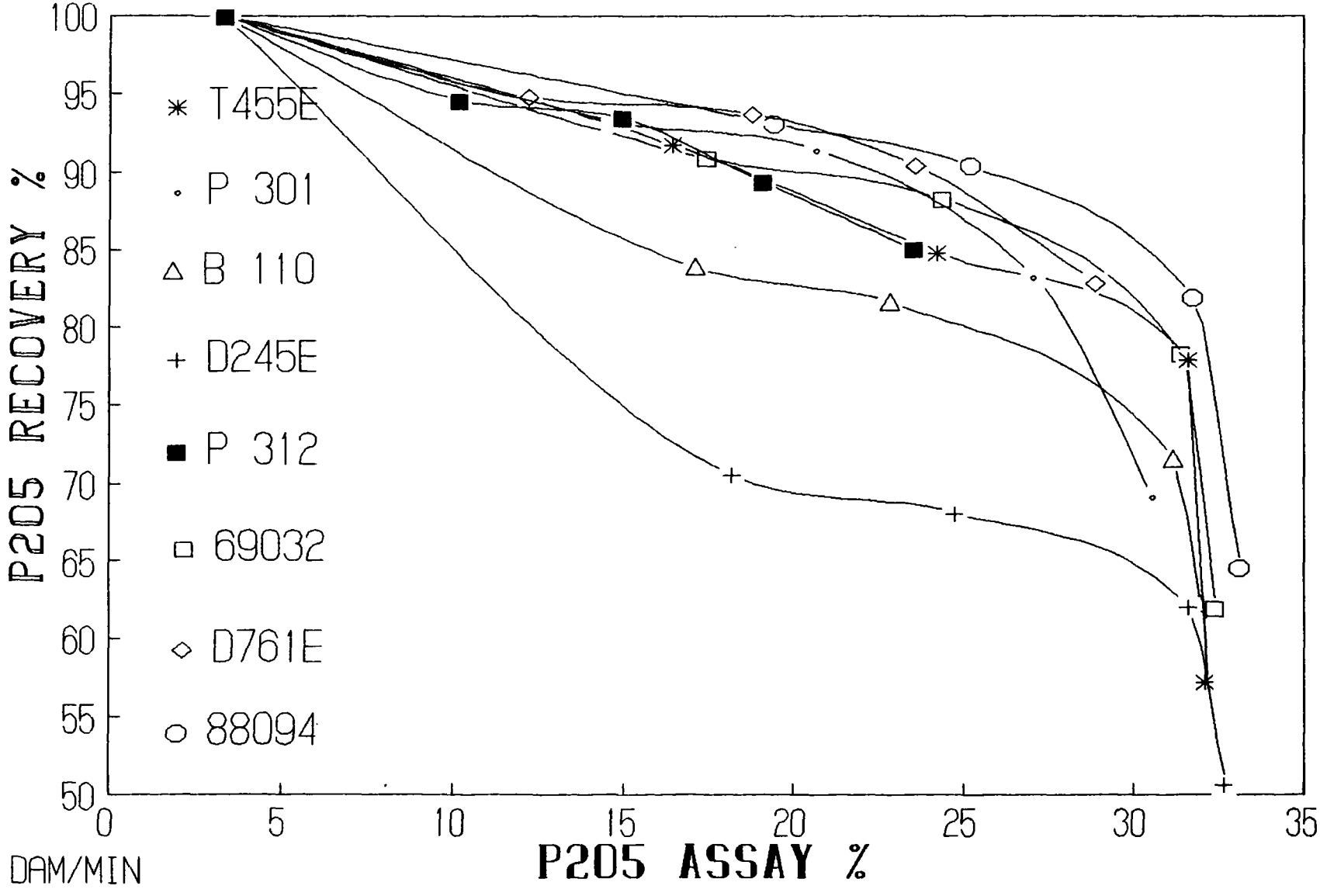
Table n° 10: Material balances of comparative flotation tests on REO ore sample n° 1

flotation test	P.E. collector	weight %	P2O5	
			content %	recovery %
n° 35	n° 2			
NF2	MELIORAN	73.82	0.24	5.23
F3		9.72	28.92	82.92
NF3		3.95	2.85	3.32
F4	D7-61E	3.25	7.81	7.49
NF4		9.26	0.38	1.04
calculated head		100.00	3.39	100.00
F3 + F4		12.97	23.63	90.41
n° 36	n° 7			
NF2	P301	77.98	0.30	6.74
F3		7.84	30.60	69.14
NF3		4.67	6.10	8.20
F4	T6 PS	2.82	17.39	14.13
NF4	(T6-59)	6.69	0.93	1.79
calculated head		100.00	3.47	100.00
F3 + F4		10.66	27.10	83.27
n° 37	n° 8			
NF2	P312	69.12	0.26	5.40
F3		12.02	23.55	85.01
NF3		5.15	2.62	4.05
F4	D9 PS2	3.54	4.12	4.38
NF4	(D9-50)	10.17	0.38	1.16
calculated head		100.00	3.33	100.00
F3 + F4		15.56	19.13	89.39

Table n° 11: Material balances of comparative flotation tests on REO ore sample n° 1

FIGURE N 4.

R.E.O. ORE SAMPLE 1.FLOTATION TESTS
PERFORMANCE OF PHOSPHORIC ESTERS



Chemical compositions of apatite concentrates separated using the phosphoric ester collector B110 88094 with low dosages of 150 g/t (rougher flotation) and 75 g/t (scavenger flotation), are given below:

	Apatite concentrates		Feed to flotation
	F3 + F4	F3	
P_2O_5 %	31.82	33.10	3.42
CaO %	45.72	46.35	7.74
SiO_2 %	6.92	5.75	62.56
Fe_2O_3 %	1.20	0.95	5.92
Al_2O_3 %	0.55	0.42	8.69
MgO %	0.53	0.50	1.64
L.O.I. %	3.10	2.31	1.09
La ppm	7 650	7 610	1 679
Ce ppm	14 800	14 220	3 009
Nd ppm	7 030	6 830	1 124
Sm ppm	1 525	1 485	217.5
Eu ppm	149.9	155	20.2
Gd ppm	1 640	1 650	209.2
Dy ppm	1 420	1 480	185.1
Er ppm	853	870	102.5
Yb ppm	783	792	89.7
Y ppm	8 015	8 080	1 006.2
Total REE + Y ppm	43 865.9	43 172	7 642.4
CaO/P_2O_5	1.437	1.400	2.263
R_2O_3/P_2O_5	0.055	0.041	4.272
T.C.P. %	69.53	72.32	7.47
P_2O_5 recovery %	81.93	64.54	100.00
$(R_2O_3 + MgO)/P_2O_5$	0.0716	0.0565	4.7515
total REE + Y recovery %	50.40	37.45	100.00
Eu recovery %	65.15	50.87	100.00
Y recovery %	69.94	53.24	100.00
apatite content %	88.90	92.5	9.55

Flotation with B110 88094 leads to apatite concentrates which meet specifications for their potential use as a phosphate material to be converted into wet process phosphoric acid:

	F3	F3+F4
P_2O_5 content %	33.10	31.82
Equivalent in Tri Calcium Phosphate %	72.32	69.53
R_2O_3 content %	1.37	1.75
MgO content %	0.50	0.53
$(R_2O_3 + MgO)/P_2O_5$	0.0565	0.0716
CaO/P_2O_5	1.400	1.437

Low levels in R_2O_3 and MgO might probably allow superphosphoric acid to be produced.

Satisfactory concentrates can be separated at acceptable phosphate recoveries: 64.54 and 81.93 % on flotation feed basis. Lower REE+Y, Y and Eu recoveries account for REE and Y associated to silicates or silico-phosphates which are not floated with P.E. as collectors.

Flotation in a continuous circuit would probably lead to increased phosphate recovery in composite phosphate concentrate F3+F4 due to recirculation of middlings NF3.

4.2.2. Analysis of flotation test data

Assessment of test data was made using selectivity index S and concentration efficiency E defined by the expressions:

$$S = (c-h) / (cmax - h)$$

$$E = [(c - h) / (cmax - h)].R$$

where c: P_2O_5 content % in concentrate
h: P_2O_5 content % in feed
cmax: maximum P_2O_5 content % in concentrate obtainable at 100 % recovery, this corresponds to P_2O_5 content in apatite i.e. 35.8 %
R: phosphate recovery % in concentrate.

Flotation test data and main characteristics of the P.E. collector used are shown in table 12 where:

- the row number corresponds to the number of the P.E. collector shown in paragraph 4.1. as well as in tables 10 and 11
- NbC, n, MEO, DEO, NPP, FOPA, FPPA, MEP, DEP are variables corresponding to characteristics of phosphoric ester collectors shown in tables 8 and 9 paragraph 4.1.
- tP_2O_5 FC, RP205 FC, EP205 FC, SP205 FC are P_2O_5 content and recovery, concentration efficiency for P_2O_5, selectivity index for P_2O_5 relevant to the composite concentrate F3+F4

File RE01SY01 Page 1-1

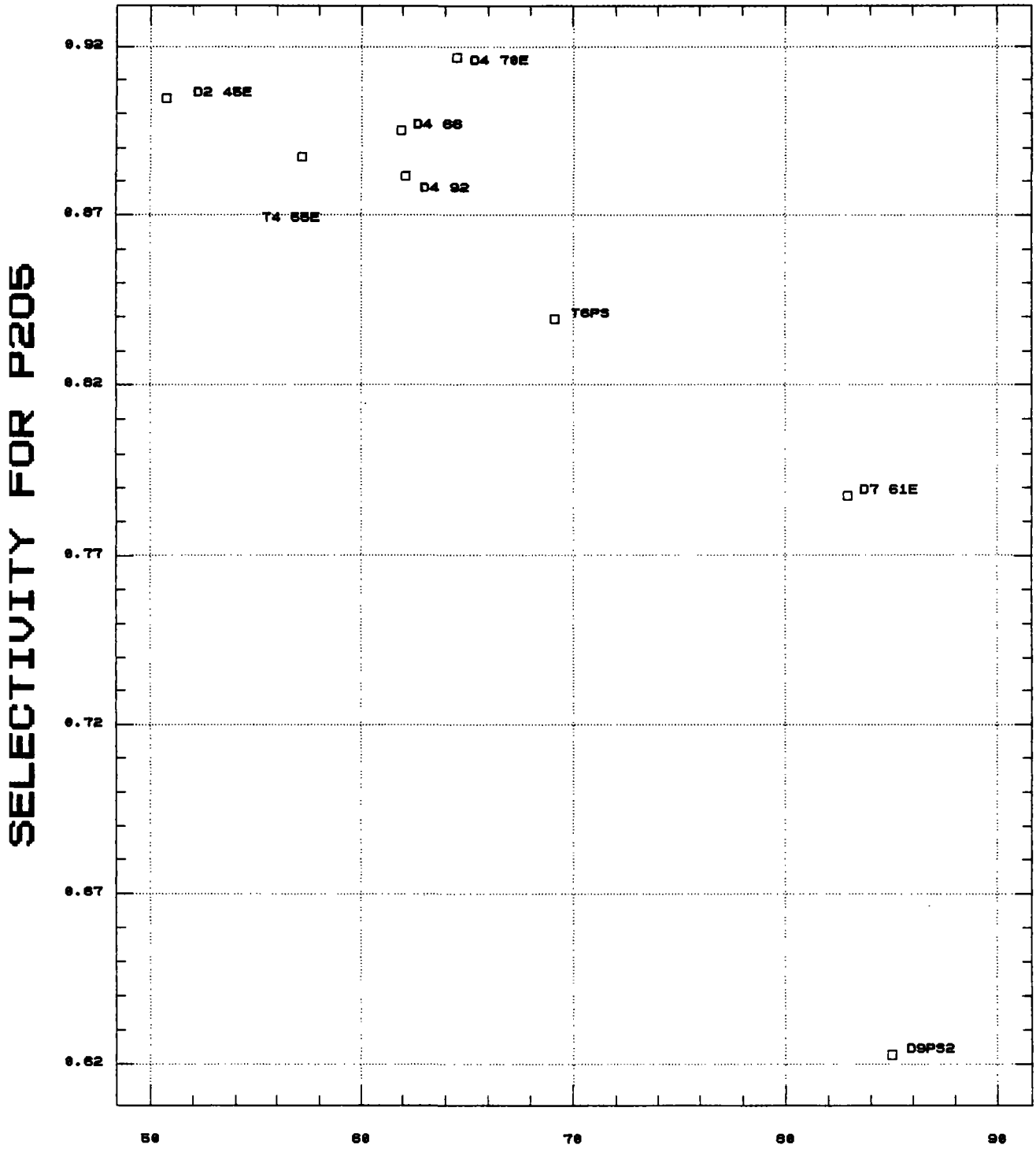
row	NbC	n	MEO	DEO	NPP	FOPA	FPPA	MEP	DEP	tP205FC	RP205FC	EP205FC	SP205FC	tP205F4	RP205F4	EP205F4	SP205F4
1	12	2	34.6	41.30	4.5	1.1	1.2	11.7	16.3	31.67	62.01	54.13	0.8728	27.75	26.17	20.02	0.7650
2	12	7	51.2	32.30	7.7	1.5	0.8	14.1	8.9	23.63	90.41	56.46	0.6245	7.81	54.43	11.22	0.2062
3	12	9	43.6	43.20	9.4	0.7	0.2	3.0	4.3	19.13	89.39	43.50	0.4866	4.12	40.04	4.17	0.1041
4	12	4	71.9	6.59	23.2	3.5	0.0	0.0	0.0	31.19	71.60	61.45	0.8582	27.00	33.94	25.35	0.7470
5	12	4	63.0	24.90	4.5	2.3	0.5	4.9	3.7	31.73	81.93	71.64	0.8744	27.50	64.50	49.11	0.7614
6	13	4	46.3	31.00	4.8	1.2	1.0	7.4	13.5	31.64	77.96	67.94	0.8715	30.33	57.77	48.59	0.8411
7	13	6	46.9	32.20	15.6	0.7	0.8	5.9	2.6	27.10	83.27	60.86	0.7309	17.39	62.38	29.47	0.4725
8	12	4	54.5	25.90	20.3	1.7	0.1	1.5	2.9	31.44	78.33	67.81	0.8657	28.30	58.29	45.71	0.7842

File RE01SY01 Page 1-2

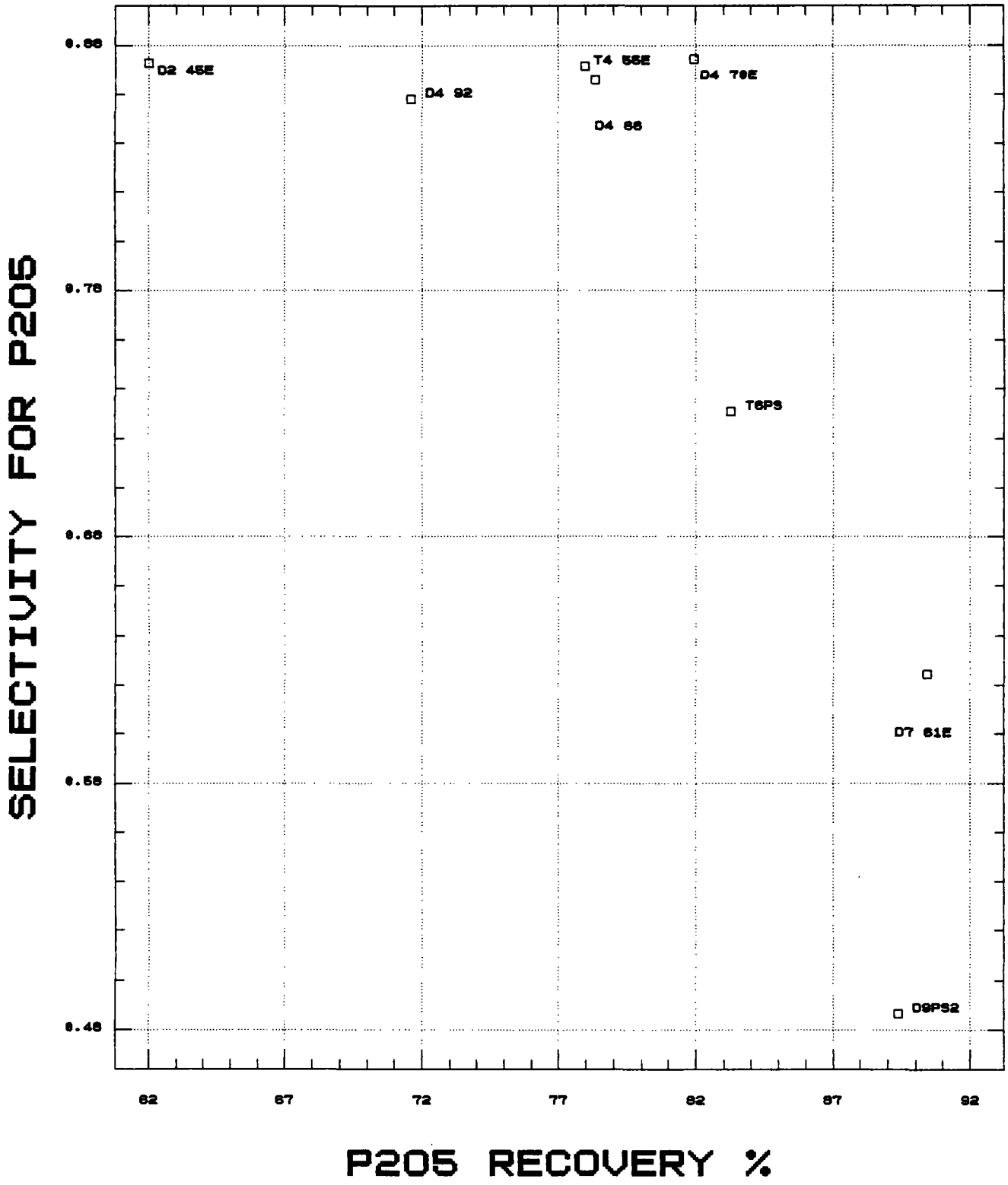
row	MDRATIO	PROMOTER	tP205F3	RP205F3	EP205F3	SP205F3
1	0.837772	D2 45E	32.70	50.72	45.88	0.9046
2	1.585139	D7 61E	28.92	82.92	65.32	0.7877
3	1.009259	D9PS2	23.55	85.01	52.94	0.6227
4	10.910470	D4 92	31.95	62.15	54.79	0.8816
5	2.530120	D4 70E	33.10	64.54	59.16	0.9167
6	1.493548	T4 55E	32.15	57.18	50.73	0.8872
7	1.456521	T6PS	30.60	69.14	58.02	0.8392
8	2.104247	D4 66	32.40	61.91	55.43	0.8953

Table 12: Flotation tests on the REO ore sample 1 with phosphoric esters as collector. Summary table

REO ORE SAMPLE 1
PERFORMANCE OF P.E.COLLECTORS (F3)



REO ORE SAMPLE 1
PERFORMANCE OF P.E.COLLECTORS (FC)



- F3 and F4 are cleaned rougher and cleaned scavenger concentrates, relevant P_2O_5 contents and recoveries, concentration efficiencies and selectivities are identified as tP_2O_5, RP205, EP205, SP205

- MD RATIO is the mono orthophosphoric ester to di orthophosphoric ester ratio value.

Treatment of the data includes:

- determinations of correlation coefficients between variables listed in table 12
- regression analysis to explain flotation results with characteristics of the collectors as the independant variables.

Correlation coefficients between all 21 variables are listed in table of the appendix 6, the most interesting correlations excerpted from this table are shown below:

Variables	Correlation coefficient r
t P205FC - n	- 0.9518
R P205FC - n	0.8743
E P205FC - n	- 0.5944
S P205FC - n	- 0.9521
E P205FC - MEO	0.5006
E P205FC - FOPA	0.4341
t P205F3 - n	- 0.9196
R P205F3 - n	0.9610
S P205F3 - n	- 0.9204
E P205F3 - n	0.4742
E P205F3 - MEO	0.4626
MEO - FOPA	0.9000
DEO - NPP	- 0.6521
NPP - FPPA	- 0.7211
MEP - NPP	- 0.6891
MDRATIO - FOPA	0.8926
DEP-NPP	- 0.7396

P_2O_5 content in concentrates and selectivity for P_2O_5 are detrimentally affected by high n values whereas P_2O_5 recovery appears to increase with n.

A stepwise multiple linear regression program was used to explain flotation performance with characteristics of the P.E. as the explicative variables.

Data relevant to P.E. with branched and linear alkyl chains were not treated separately since no major difference was revealed through comparison of flotation results obtained with T455E and D466 promoters which have the same number (4) of ethylene oxide groups.

The best fits to flotation data were obtained by the following regression equations:

- EP2O5FC= 28.82 - 4.63 n + 1.62 MEO - 18.11 FOPA
  with df: degree of freedom for error: 4
  R^2(A): R - squared Adjusted for d.f. : 0.82207
  F ratio: 11.7804 (P= 0.0187)
  S.E.: standard error of estimate: 3.839

- SP2O5FC= 0.95 - 0.06237 n + 0.0026 MEO
  df: 5
  R^2(A): 0.929
  F ratio: 46.84 (P: 0.0006)
  S.E.: 0.0392

or SP2O5FC= 0.813 - 0.07523 n + 0.0096 MEO - 0.0995 FOPA
  df: 4
  R^2(A): 0.98
  F ratio: 118.65
  S.E.: 0.0205

- tP2O5FC= 34.189 - 2.0232 n + 0.0848 MEO
  df: 5, R^2(A): 0.9281
  F: 46.15 (P= 0.0006)
  S.E.: 1.281

or tP2O5FC= 29.694 - 2.4446 n + 0.3136 MEO - 3.2622 FOPA
  df: 4, R^2(A): 0.9807
  F: 119.4
  S.E.: 0.664

- RP2O5FC= 60.8625 + 3.7 n
  df: 6, R^2(A): 0.7252
  F: 19.48
  S.E.: 4.889

Quality of the fit to flotation data provided by the regression equations is illustrated by tables and diagrams shown at the end of the paragraph 4.2.2.

From regression equations it can be concluded that:

- phosphoric esters with high n values exhibit a strong collecting power at the expense of selectivity. Ethylene oxide groups strongly

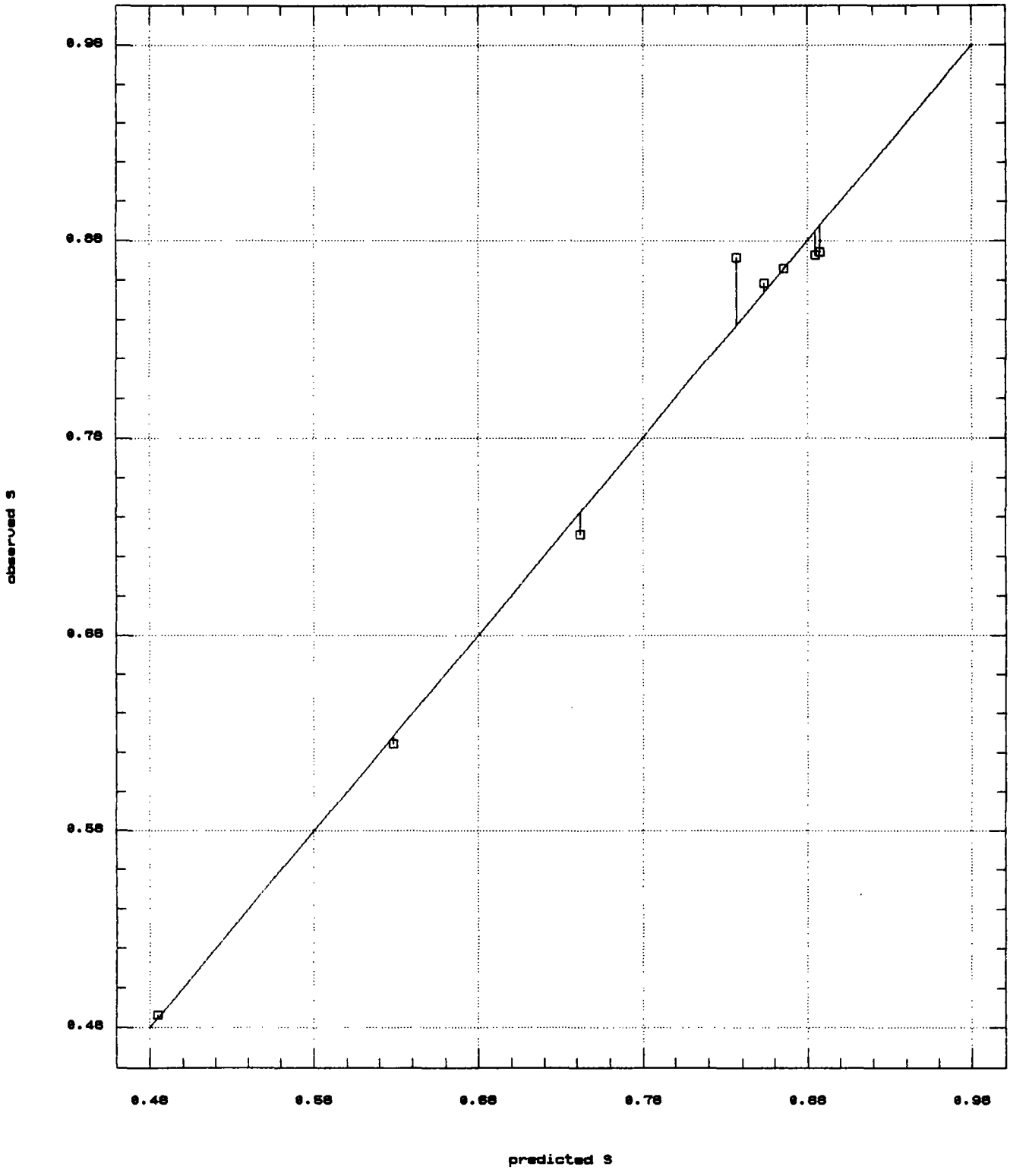
contribute to the collecting power of phosphoric esters. It should be noted that the length of the hydrophobic radical reflected by the number of Carbons (NbC) was found to exert no influence on flotation data , due probably to the fact that the variation of NbC is too slight

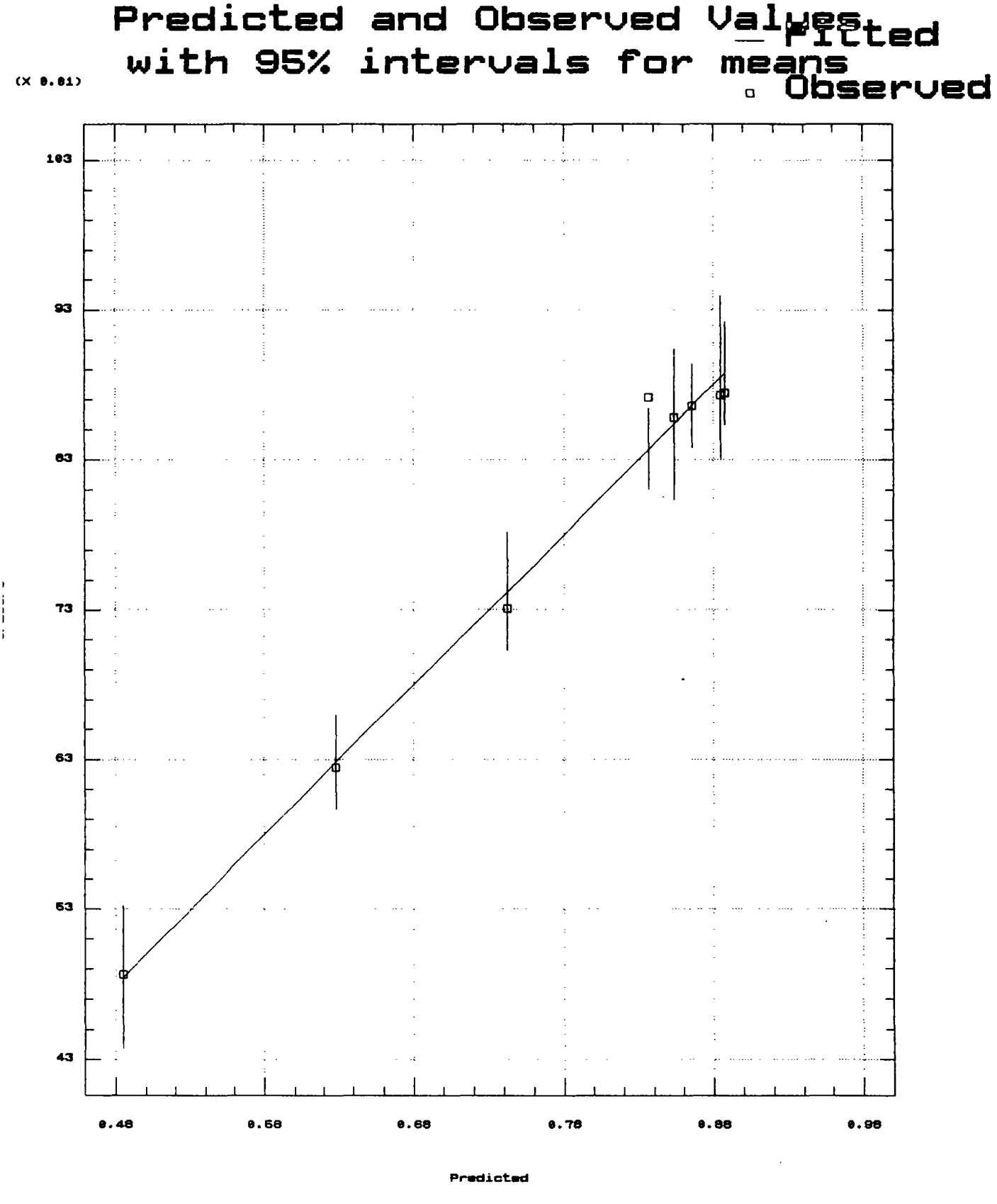
- content in mono-orthophosphoric ester (MEO) exerts a significant positive influence on both selectivity and concentration efficiency for REE and Y bearing apatite. High contents of MEO in P.E. collectors promote high selectivity and concentration efficiency

- free orthophosphoric acid detrimentally affects both concentration efficiency and selectivity, this is not surprising since H_3PO_4 is known as a strong depressant for apatite.

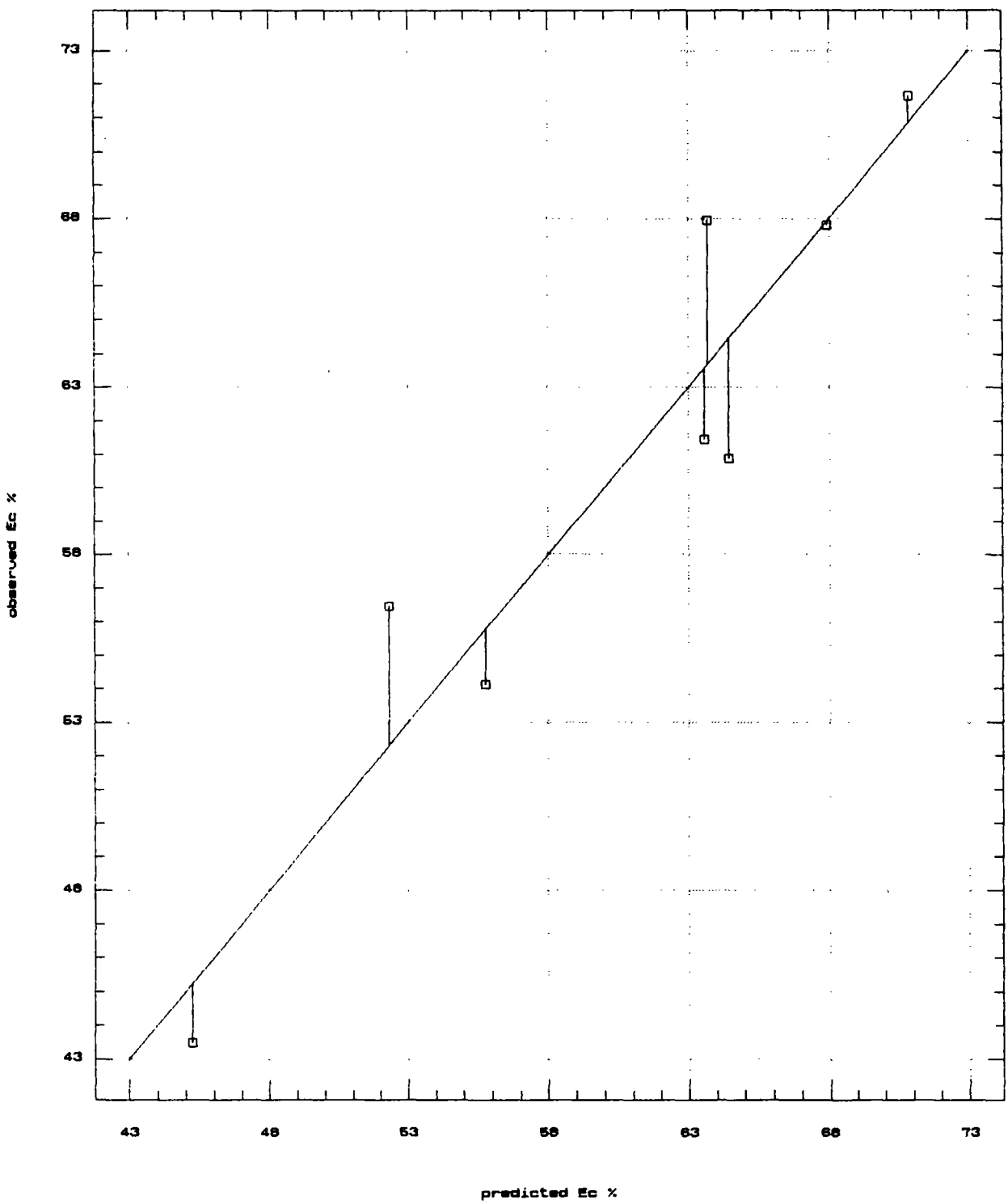
It appears that selective P.E. collectors for REE and Y bearing apatites should have a high content in mono orthophosphoric ester, a low concentration in residual H_3PO_4 and an appropriate number n of ethylene oxide groups leading to satisfactory apatite recovery with moderate detrimental effect on apatite content in the concentrates.

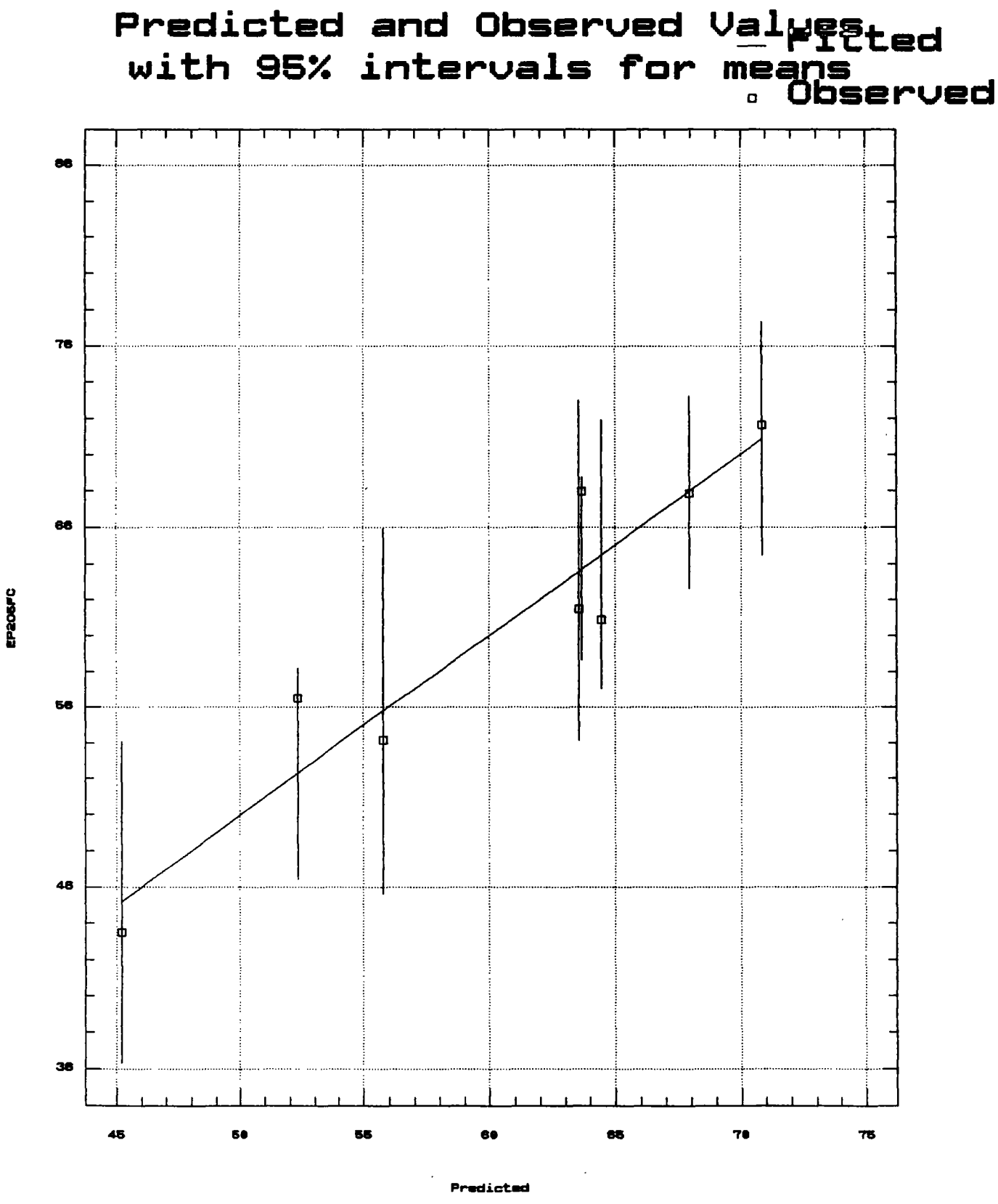
REO ORE SAMPLE 1
SELECTIVITY FOR P205 (F3+F4)




Predicted and Observed Values
with 95% intervals for means
Fitted
Observed
(X 0.01)
103
93
83
73
63
53
43
0.48
0.58
0.68
0.78
0.88
0.98
Predicted

REO ORE SAMPLE 1
CONCENTRATION EFFICIENCY FOR P205(F3+F4)




Predicted and Observed Values
with 95% intervals for means
Fitted
Observed
EP205FC
Predicted
85
75
65
55
45
35
45
50
55
60
65
70
75

4.3. COMPARATIVE FLOTATION TESTS ON REO ORE SAMPLE N° 2 WITH PHOSPHORIC ESTERS AS COLLECTORS

4.3.1. Flotation test results

Comparative flotation tests were conducted on the 80 + 10 µm gravity preconcentrate n° 2, using a basic flowsheet developed in preliminary tests and previously evaluated in flotation test n° 16.

This flowsheet is shown in figure 2F30, CO_2 was used as a depressant for Fe-Mn containing dolomite and strontianite while different types of phosphoric esters were used as monazite collectors. Operating conditions of tests are summarized as follows:

Flotation test n°	Order for evaluation	P.E. collector used	Collector dosage g/t	
			rougher stage	scavenger stage
40	1	MELIORAN D2-45E	50	20
41	2	MELIORAN D7-61E	50	20
34	3	PE89124 IM7-58	50	20
35	4	P312 D9PS2	50	20
36	5	B110 D4-92	50	20
38	6	B110 88094 D4-70E	50	20
39	7	MELIORAN T4-55E	50	20
16	8	P301 T6PS	50	20
33	9	PE89122 EA14-89	50	20
30		MELIORAN D2-45E	100	20
31		MELIORAN D7-61E	100	20
32		MELIORAN T4-55E	100	20

Products from flotation tests 30 to 39 were analyzed for Fe_2O_3, SrO, BaO, La_2O_3, CeO_2, total REO contents were calculated from La_2O_3, CeO_2, total REO contents were calculated from La and Ce contents.

Material balances of tests 30 to 39 are shown in tables 2F30 to 2F39. Material balance for test 16 is shown in table 2F16 in paragraph 3.2.3.

Products from flotation tests 40 and 41 were only analyzed for La and Ce, total REO contents were derived from La and Ce determinations.

Material balances of tests 40 and 41 are given in the following table:

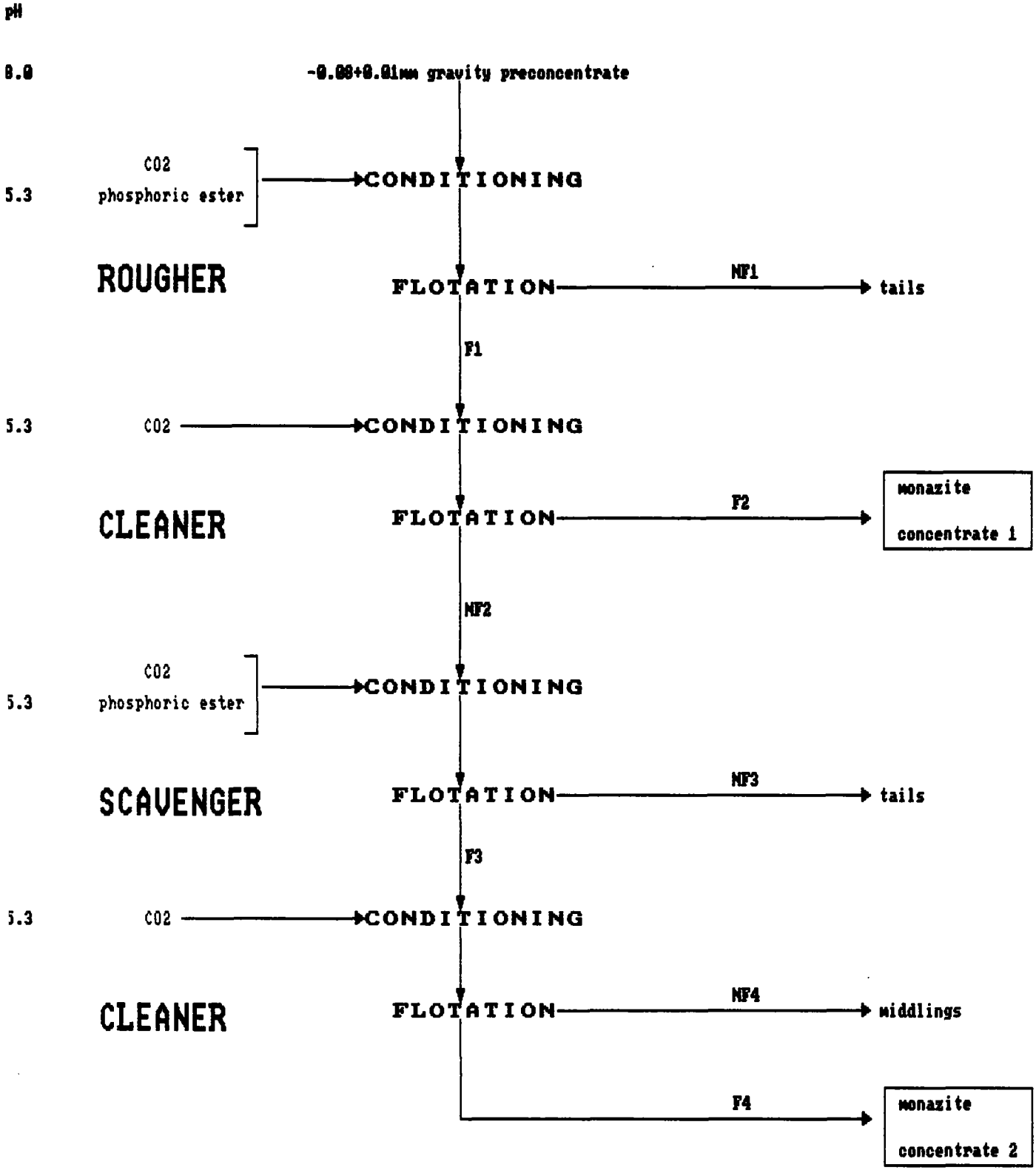


figure n° 2F30 : basic flowsheet for flotation of -0.08+0.01mm gravity preconcentrates separated from the R.E.O. ore sample n°2 ground down to -0.16mm. (evaluation of phosphoric ester promoters)

TABLE N° 2F30 : REE ORE SAMPLE N°2,-0.08+0.01mm GRAVITY PRECONCENTRATE, MATERIAL BALANCE OF FLOTATION TEST N°30.

PRODUCTS	WEIGHT	Fe2O3		SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1	39.25	7.38	63.68	10.72	42.95	6.37	51.94	34.30	33.75
2 F2	32.39	1.00	7.12	3.81	12.60	1.41	9.49	57.77	46.91
3 NF3	12.84	5.86	16.54	22.77	29.84	7.86	20.96	18.90	6.08
4 NF4	10.75	4.86	11.49	12.26	13.45	7.34	16.39	31.12	8.39
5 F4	4.77	1.12	1.17	2.38	1.16	1.23	1.22	40.72	4.87
CALCULATED HEAD	100.00	4.55	100.00	9.80	100.00	4.81	100.00	39.89	100.00

PRODUCTS	WEIGHT %	REO TOT. GRADE / SrO GRADE
1 NF1	39.25	3.1996
2 F2	32.39	15.1627
3 NF3	12.84	.8300
4 NF4	10.75	2.5383
5 F4	4.77	17.1092
CALCULATED HEAD	100.00	4.0716

TABLE N° 2F30 : FLOTATION TEST N° 30.

PRODUCTS	WEIGHT	Fe203		SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1 1 0 0 0 0 0	39.25	7.38	63.68	10.72	42.95	6.37	51.94	34.30	33.75
2 F1 2 3 4 5 0 0	60.75	2.72	36.32	9.20	57.05	3.81	48.06	43.50	66.25
3 NF1+NF3 1 3 0 0 0 0	52.09	7.01	80.22	13.69	72.79	6.74	72.90	30.50	39.83
4 F2+F3 2 4 5 0 0 0	47.91	1.88	19.78	5.56	27.21	2.72	27.10	50.09	60.17
5 NF1+NF3+NF4 1 3 4 0 0 0	62.84	6.64	91.71	13.45	86.24	6.84	89.29	30.61	48.22
6 F2+F4 2 5 0 0 0 0	37.16	1.02	8.29	3.63	13.76	1.39	10.71	55.58	51.78
7 NF1+NF2 1 3 4 5 0 0	67.61	6.25	92.88	12.66	87.40	6.44	90.51	31.32	53.09
8 F2 2 0 0 0 0 0	32.39	1.00	7.12	3.81	12.60	1.41	9.49	57.77	46.91

TABLE N° 2F31 : REE ORE SAMPLE N°2,-0.08+0.01mm GRAVITY PRECONCENTRATE, MATERIAL BALANCE OF FLOTATION TEST N°31.

PRODUCTS	WEIGHT	Fe203		SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1	8.97	12.66	29.16	7.93	8.13	8.95	16.19	17.61	3.76
2 F2	61.47	2.00	31.57	3.90	27.40	1.02	12.64	57.55	84.10
3 NF3	6.15	8.97	14.17	14.40	10.12	14.68	18.21	9.90	1.45
4 NF4	16.13	5.31	22.00	25.00	46.08	15.52	50.48	8.96	3.44
5 F4	7.28	1.66	3.10	9.94	8.27	1.69	2.48	41.97	7.26
CALCULATED HEAD	100.00	3.89	100.00	8.75	100.00	4.96	100.00	42.07	100.00

PRODUCTS	WEIGHT %	REO TOT. GRADE / SrO GRADE
1 NF1	8.97	2.2207
2 F2	61.47	14.7564
3 NF3	6.15	.6875
4 NF4	16.13	.3584
5 F4	7.28	4.2223
CALCULATED HEAD	100.00	4.8072

TABLE N° 2F31 : FLOTATION TEST N° 31.

PRODUCTS	WEIGHT	Fe203		SrO		BaO		RED TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1 1 0 0 0 0 0	8.97	12.66	29.16	7.93	8.13	8.95	16.19	17.61	3.76
2 F1 2 3 4 5 0 0	91.03	3.03	70.84	8.83	91.87	4.57	83.81	44.47	96.24
3 NF1+NF3 1 3 0 0 0 0	15.12	11.16	43.33	10.56	18.25	11.28	34.39	14.47	5.20
4 F2+F3 2 4 5 0 0 0	84.88	2.60	56.67	8.43	81.75	3.83	65.61	46.98	94.80
5 NF1+NF3+NF4 1 3 4 0 0 0	31.25	8.14	65.32	18.01	64.33	13.47	84.88	11.63	8.64
6 F2+F4 2 5 0 0 0 0	68.75	1.96	34.68	4.54	35.67	1.09	15.12	55.90	91.36
7 NF1+NF2 1 3 4 5 0 0	38.53	6.92	68.43	16.49	72.60	11.24	87.36	17.36	15.90
8 F2 2 0 0 0 0 0	61.47	2.00	31.57	3.90	27.40	1.02	12.64	57.55	84.10

TABLE N° 2F32 : REE ORE SAMPLE N°2,-0.08+0.01mm GRAVITY PRECONCENTRATE, MATERIAL BALANCE OF FLOTATION TEST N°32.

PRODUCTS	WEIGHT	Fe2O3		SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1	4.98	11.27	14.72	6.00	3.20	4.17	4.31	18.23	2.15
2 F2	68.39	2.38	42.69	5.72	41.94	1.25	17.74	55.44	89.85
3 NF3	7.21	8.87	16.77	14.78	11.43	15.60	23.34	7.29	1.25
4 NF4	14.52	6.06	23.08	25.00	38.92	16.69	50.29	9.46	3.25
5 F4	4.90	2.14	2.75	8.58	4.51	4.25	4.32	30.16	3.50
CALCULATED HEAD	100.00	3.81	100.00	9.33	100.00	4.82	100.00	42.20	100.00

PRODUCTS	WEIGHT %	REO TOT. GRADE / SrO GRADE
1 NF1	4.98	3.0383
2 F2	68.39	9.6923
3 NF3	7.21	.4932
4 NF4	14.52	.3784
5 F4	4.90	3.5152
CALCULATED HEAD	100.00	4.5246

TABLE N° 2F32 : FLOTATION TEST N° 32.

PRODUCTS	WEIGHT	Fe2O3		SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1 1 0 0 0 0 0	4.98	11.27	14.72	6.00	3.20	4.17	4.31	18.23	2.15
2 F1 2 3 4 5 0 0	95.02	3.42	85.28	9.50	96.80	4.85	95.69	43.46	97.85
3 NF1+NF3 1 3 0 0 0 0	12.19	9.85	31.49	11.19	14.63	10.93	27.65	11.76	3.40
4 F2+F3 2 4 5 0 0 0	87.81	2.98	68.51	9.07	85.37	3.97	72.35	46.43	96.60
5 NF1+NF3+NF4 1 3 4 0 0 0	26.71	7.79	54.56	18.70	53.55	14.06	77.94	10.51	6.65
6 F2+F4 2 5 0 0 0 0	73.29	2.36	45.44	5.91	46.45	1.45	22.06	53.75	93.35
7 NF1+NF2 1 3 4 5 0 0	31.61	6.91	57.31	17.13	58.06	12.54	82.26	13.56	10.15
8 F2 2 0 0 0 0 0	68.39	2.38	42.69	5.72	41.94	1.25	17.74	55.44	89.85

TABLE N° 2F33 : REE ORE SAMPLE N°2,-0.08+0.01mm GRAVITY PRECONCENTRATE, MATERIAL BALANCE OF FLOTATION TEST N°33.

PRODUCTS	WEIGHT	Fe2O3		SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1	35.86	8.65	70.73	17.52	63.78	12.09	82.60	18.47	15.49
2 F2	39.76	1.00	9.07	1.33	5.37	.75	5.68	68.88	64.04
3 NF3	5.19	7.66	9.07	21.47	11.31	7.11	7.03	13.91	1.69
4 NF4	6.91	5.29	8.34	24.05	16.87	2.44	3.21	17.82	2.88
5 F4	12.28	1.00	2.80	2.14	2.67	.63	1.47	55.39	15.91
CALCULATED HEAD	100.00	4.39	100.00	9.85	100.00	5.25	100.00	42.77	100.00

PRODUCTS	WEIGHT %	REO TOT. GRADE / SrO GRADE
1 NF1	35.86	1.0542
2 F2	39.76	51.7895
3 NF3	5.19	.6479
4 NF4	6.91	.7410
5 F4	12.28	25.8832
CALCULATED HEAD	100.00	4.3415

TABLE N° 2F33 : FLOTATION TEST N° 33.

PRODUCTS	WEIGHT	Fe203		SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1 1 0 0 0 0 0	35.86	8.65	70.73	17.52	63.78	12.09	82.60	18.47	15.49
2 F1 2 3 4 5 0 0	64.14	2.00	29.27	5.56	36.22	1.42	17.40	56.35	84.51
3 NF1+NF3 1 3 0 0 0 0	41.05	8.52	79.80	18.02	75.09	11.46	89.63	17.89	17.18
4 F2+F3 2 4 5 0 0 0	58.95	1.50	20.20	4.16	24.91	.92	10.37	60.08	82.82
5 NF1+NF3+NF4 1 3 4 0 0 0	47.96	8.06	88.13	18.89	91.96	10.16	92.84	17.88	20.06
6 F2+F4 2 5 0 0 0 0	52.04	1.00	11.87	1.52	8.04	.72	7.16	65.70	79.94
7 NF1+NF2 1 3 4 5 0 0	60.24	6.62	90.93	15.47	94.63	8.22	94.32	25.53	35.96
8 F2 2 0 0 0 0 0	39.76	1.00	9.07	1.33	5.37	.75	5.68	68.88	64.04

TABLE N° 2F34 : REE ORE SAMPLE N°2,-0.08+0.01mm GRAVITY PRECONCENTRATE, MATERIAL BALANCE OF FLOTATION TEST N°34.

PRODUCTS	WEIGHT	Fe2O3		SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1	32.13	8.55	55.26	11.95	38.55	9.73	60.12	28.45	22.07
2 F2	33.16	1.58	10.54	1.72	5.73	.75	4.78	64.61	51.72
3 NF3	15.33	5.97	18.41	22.59	34.77	9.26	27.30	17.92	6.63
4 NF4	11.20	6.28	14.15	17.36	19.52	3.08	6.63	29.39	7.95
5 F4	8.18	1.00	1.65	1.75	1.44	.74	1.16	58.91	11.63
CALCULATED HEAD	100.00	4.97	100.00	9.96	100.00	5.20	100.00	41.42	100.00

PRODUCTS	WEIGHT %	REO TOT. GRADE / SrO GRADE
1 NF1	32.13	2.3808
2 F2	33.16	37.5640
3 NF3	15.33	.7933
4 NF4	11.20	1.6930
5 F4	8.18	33.6629
CALCULATED HEAD	100.00	4.1588

TABLE N° 2F34 : FLOTATION TEST N° 34.

PRODUCTS	WEIGHT	Fe2O3		SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1 1 0 0 0 0 0	32.13	8.55	55.26	11.95	38.55	9.73	60.12	28.45	22.07
2 F1 2 3 4 5 0 0	67.87	3.28	44.74	9.02	61.45	3.06	39.88	47.56	77.93
3 NF1+NF3 1 3 0 0 0 0	47.46	7.72	73.67	15.39	73.32	9.58	87.42	25.05	28.70
4 F2+F3 2 4 5 0 0 0	52.54	2.49	26.33	5.06	26.68	1.25	12.58	56.21	71.30
5 NF1+NF3+NF4 1 3 4 0 0 0	58.66	7.44	87.82	15.76	92.84	8.34	94.05	25.88	36.65
6 F2+F4 2 5 0 0 0 0	41.34	1.47	12.18	1.73	7.16	.75	5.95	63.48	63.35
7 NF1+NF2 1 3 4 5 0 0	66.84	6.65	89.46	14.05	94.27	7.41	95.22	29.92	48.28
8 F2 2 0 0 0 0 0	33.16	1.58	10.54	1.72	5.73	.75	4.78	64.61	51.72

TABLE N° 2F35 : REE ORE SAMPLE N°2,-0.08+0.01mm GRAVITY PRECONCENTRATE, MATERIAL BALANCE OF FLOTATION TEST N°35.

PRODUCTS	WEIGHT	Fe2O3		SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1	28.61	10.08	62.02	14.87	42.85	12.82	68.69	17.99	12.24
2 F2	37.71	1.00	8.11	1.65	6.27	.77	5.44	67.78	60.80
3 NF3	8.39	7.09	12.79	23.36	19.74	11.22	17.63	13.90	2.77
4 NF4	11.69	5.63	14.15	23.65	27.84	3.00	6.57	20.41	5.68
5 F4	13.60	1.00	2.92	2.41	3.30	.66	1.68	57.23	18.51
CALCULATED HEAD	100.00	4.65	100.00	9.93	100.00	5.34	100.00	42.04	100.00

PRODUCTS	WEIGHT %	REO TOT. GRADE / SrO GRADE
1 NF1	28.61	1.2098
2 F2	37.71	41.0788
3 NF3	8.39	.5950
4 NF4	11.69	.8630
5 F4	13.60	23.7469
CALCULATED HEAD	100.00	4.2343

TABLE N° 2F35 : FLOTATION TEST N° 35.

PRODUCTS	WEIGHT	Fe203		SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1 1 0 0 0 0 0	28.61	10.08	62.02	14.87	42.85	12.82	68.69	17.99	12.24
2 F1 2 3 4 5 0 0	71.39	2.47	37.98	7.95	57.15	2.34	31.31	51.68	87.76
3 NF1+NF3 1 3 0 0 0 0	37.00	9.40	74.81	16.80	62.59	12.46	86.31	17.06	15.02
4 F2+F3 2 4 5 0 0 0	63.00	1.86	25.19	5.90	37.41	1.16	13.69	56.71	84.98
5 NF1+NF3+NF4 1 3 4 0 0 0	48.69	8.50	88.97	18.44	90.43	10.19	92.88	17.87	20.69
6 F2+F4 2 5 0 0 0 0	51.31	1.00	11.03	1.85	9.57	.74	7.12	64.98	79.31
7 NF1+NF2 1 3 4 5 0 0	62.29	6.86	91.89	14.94	93.73	8.11	94.56	26.46	39.20
8 F2 2 0 0 0 0 0	37.71	1.00	8.11	1.65	6.27	.77	5.44	67.78	60.80

TABLE N° 2F36 : REE ORE SAMPLE N°2,-0.08+0.01mm GRAVITY PRECONCENTRATE, MATERIAL BALANCE OF FLOTATION TEST N°36.

PRODUCTS	WEIGHT	Fe2O3		SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1	42.43	9.78	77.30	11.79	49.56	7.54	59.00	33.27	34.38
2 F2	19.68	1.00	3.67	1.95	3.80	1.53	5.55	63.63	30.50
3 NF3	10.25	6.29	12.01	22.21	22.55	10.74	20.30	16.06	4.01
4 NF4	12.70	1.79	4.23	16.95	21.33	4.76	11.15	31.95	9.88
5 F4	14.94	1.00	2.78	1.86	2.75	1.45	4.00	58.31	21.22
CALCULATED HEAD	100.00	5.37	100.00	10.09	100.00	5.42	100.00	41.05	100.00

PRODUCTS	WEIGHT %	REO TOT. GRADE / SrO GRADE
1 NF1	42.43	2.8219
2 F2	19.68	32.6308
3 NF3	10.25	.7231
4 NF4	12.70	1.8850
5 F4	14.94	31.3495
CALCULATED HEAD	100.00	4.0675

TABLE N° 2F36 : FLOTATION TEST N° 36.

PRODUCTS	WEIGHT	Fe2O3		SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1 1 0 0 0 0 0	42.43	9.78	77.30	11.79	49.56	7.54	59.00	33.27	34.38
2 F1 2 3 4 5 0 0	57.57	2.12	22.70	8.84	50.44	3.86	41.00	46.79	65.62
3 NF1+NF3 1 3 0 0 0 0	52.68	9.10	89.32	13.82	72.12	8.16	79.30	29.92	38.39
4 F2+F3 2 4 5 0 0 0	47.32	1.21	10.68	5.95	27.88	2.37	20.70	53.45	61.61
5 NF1+NF3+NF4 1 3 4 0 0 0	65.38	7.68	93.55	14.43	93.44	7.50	90.45	30.32	48.28
6 F2+F4 2 5 0 0 0 0	34.62	1.00	6.45	1.91	6.56	1.50	9.55	61.33	51.72
7 NF1+NF2 1 3 4 5 0 0	80.32	6.44	96.33	12.09	96.20	6.38	94.45	35.52	69.50
8 F2 2 0 0 0 0 0	19.68	1.00	3.67	1.95	3.80	1.53	5.55	63.63	30.50

TABLE N° 2F38 : REE ORE SAMPLE N°2,-0.08+0.01mm GRAVITY PRECONCENTRATE, MATERIAL BALANCE OF FLOTATION TEST N°38.

PRODUCTS	WEIGHT	Fe2O3		SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1	43.46	9.53	75.46	11.83	51.49	7.52	64.18	30.39	32.22
2 F2	23.00	1.00	4.19	1.91	4.40	.85	3.84	66.64	37.39
3 NF3	9.34	7.32	12.46	19.30	18.05	9.31	17.08	17.64	4.02
4 NF4	12.52	2.53	5.77	18.87	23.66	5.29	13.01	30.56	9.33
5 F4	11.68	1.00	2.13	2.05	2.40	.83	1.90	59.76	17.03
CALCULATED HEAD	100.00	5.49	100.00	9.99	100.00	5.09	100.00	40.99	100.00

PRODUCTS	WEIGHT %	REO TOT. GRADE / SrO GRADE
1 NF1	43.46	2.5689
2 F2	23.00	34.8901
3 NF3	9.34	.9140
4 NF4	12.52	1.6195
5 F4	11.68	29.1512
CALCULATED HEAD	100.00	4.1049

TABLE N° 2F38 : FLOTATION TEST N° 38.

PRODUCTS	WEIGHT	Fe203		SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
NF1 00000	43.46	9.53	75.46	11.83	51.49	7.52	64.18	30.39	32.22
F1 34500	56.54	2.38	24.54	8.57	48.51	3.23	35.82	49.13	67.78
NF1+NF3 30000	52.80	9.14	87.91	13.15	69.54	7.84	81.25	28.13	36.24
F2+F3 45000	47.20	1.41	12.09	6.44	30.46	2.02	18.75	55.37	63.76
NF1+NF3+NF4 34000	65.32	7.87	93.68	14.25	93.20	7.35	94.26	28.60	45.58
F2+F4 50000	34.68	1.00	6.32	1.96	6.80	.84	5.74	64.32	54.42
NF1+NF2 34500	77.00	6.83	95.81	12.40	95.60	6.36	96.16	33.33	62.61
F2 00000	23.00	1.00	4.19	1.91	4.40	.85	3.84	66.64	37.39

TABLE N° 2F39 : REE ORE SAMPLE N°2,-0,08+0.01mm GRAVITY PRECONCENTRATE, MATERIAL BALANCE OF FLOTATION TEST N°39.

PRODUCTS	WEIGHT	Fe2O3		SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1	18.98	9.95	44.93	10.42	20.03	9.07	32.80	30.31	13.32
2 F2	39.43	1.39	13.04	2.35	9.38	.84	6.31	64.03	58.44
3 NF3	12.53	6.78	20.21	18.06	22.92	15.22	36.33	14.24	4.13
4 NF4	17.45	4.59	19.06	24.81	43.85	6.87	22.84	19.48	7.87
5 F4	11.61	1.00	2.76	3.25	3.82	.78	1.73	60.43	16.24
CALCULATED HEAD	100.00	4.20	100.00	9.87	100.00	5.25	100.00	43.20	100.00

PRODUCTS	WEIGHT %	REO TOT. GRADE / SrO GRADE
1 NF1	18.98	2.9088
2 F2	39.43	27.2468
3 NF3	12.53	.7885
4 NF4	17.45	.7852
5 F4	11.61	18.5938
CALCULATED HEAD	100.00	4.3751

TABLE N° 2F39 : FLOTATION TEST N° 39.

PRODUCTS	WEIGHT	Fe2O3		SrO		BaO		REO TOT.	
	%	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY	GRADE %	RECOVERY
1 NF1 1 0 0 0 0 0	18.98	9.95	44.93	10.42	20.03	9.07	32.80	30.31	13.32
2 F1 2 3 4 5 0 0	81.02	2.86	55.07	9.75	79.97	4.35	67.20	46.22	86.68
3 NF1+NF3 1 3 0 0 0 0	31.51	8.69	65.14	13.46	42.95	11.52	69.13	23.92	17.45
4 F2+F3 2 4 5 0 0 0	68.49	2.14	34.86	8.22	57.05	2.37	30.87	52.07	82.55
5 NF1+NF3+NF4 1 3 4 0 0 0	48.96	7.23	84.20	17.50	86.79	9.86	91.96	22.34	25.32
6 F2+F4 2 5 0 0 0 0	51.04	1.30	15.80	2.55	13.21	.83	8.04	63.21	74.68
7 NF1+NF2 1 3 4 5 0 0	60.57	6.03	86.96	14.77	90.62	8.12	93.69	29.64	41.56
8 F2 2 0 0 0 0 0	39.43	1.39	13.04	2.35	9.38	.84	6.31	64.03	58.44

	Weight %	REO TOTAL	
		content %	recovery %
test n° 40			
NF1	58.71	38.07	53.47
F2	17.71	60.90	25.80
NF3	8.73	21.21	4.43
NF4	9.04	41.38	8.95
F4	5.81	52.90	7.35
calculated head	100.00	41.80	100.00
test n° 41			
NF1	31.37	18.17	13.70
F2	33.33	66.30	53.12
NF3	8.44	15.28	3.10
NF4	14.73	32.14	11.38
F4	12.13	64.15	18.70
calculated head	100.00	41.60	100.00

Flotation performances for collector additions of 50 g/t and 20 g/t in rougher and scavenger stages are shown as grade-recovery curves for total REO in figure n° 5.

It can be seen that the best performances were achieved with PE89122 EA14-89, a high grade composite concentrate (FC= F2+F4) was produced at acceptable REO recovery (on flotation feed basis):

Fe_2O_3 content %:	1.00
SrO content %:	1.52
BaO content %:	0.72
total REO content %:	65.70
total REO recovery %:	79.94
monazite content %:	95.22

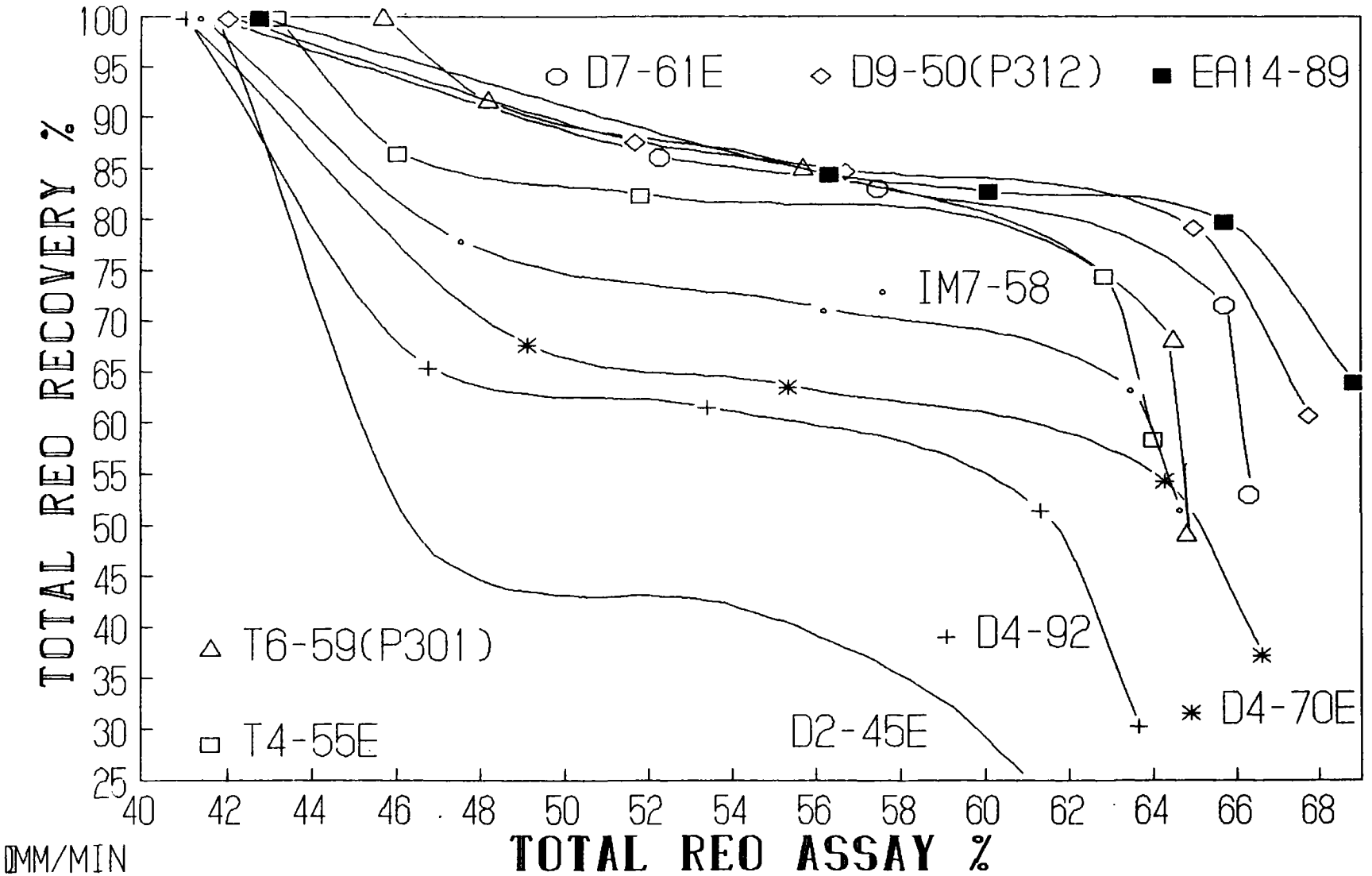
improvement in REO recovery can be expected if flotation is performed in a continuous circuit involving recirculation of middlings and optionally regrinding, to enhance liberation of monazite.

It should be pointed out that flotation of monazite requires extremely low additions of phosphoric ester collector: 70 g/t. Results of this series of tests also confirm the effectiveness of CO_2 as a depressant for iron bearing carbonates (dolomite-siderite), strontianite and barite. Further development of the flotation process in combination with magnetic separation could be devoted to production of barite and strontianite concentrates.

FIGURE N 5

REO ORE SAMPLE 2. FLOTATION TESTS

PERFORMANCE OF PHOSPHORIC ESTERS



DMM/MIN

4.3.2. Analysis of flotation test data

Besides conventional indicators of concentration as REO grade and recovery, selectivity index S and concentration efficiency E were taken into consideration in the analysis of flotation test data, cmax corresponding to maximum REO content in a monazite concentrate obtainable at 100 % recovery was set at 69 % as an average. However, it should be noted that flotation data indicates that a portion of monazite could be slightly higher in REO.

Flotation test data and main characteristics of the Phosphoric Ester collectors are listed in table 13 where:

- the row number corresponds to the number of the P.E. collector as previously shown in paragraph 4.1. and paragraph 4.3.1. as the order for evaluation

- tREOFC, RREOFC, EREOFC, SREOFC stand for REO contents and recovery, concentration efficiency and selectivity index for REO relevant to the composite concentrate F2+F4 produced in the rougher and scavenger stages. Similar indices relevant to rougher-cleaner concentrate alone (F2) and scavenger-cleaner concentrate alone (F4) are also given in table n° 13

- variables relevant to P.E. collector reagents have already been described in paragraph 4.1. MDRATIO is the mono-orthophosphoric ester to di-orthophosphoric ester ratio value (MEO/DEO).

Data of table n° 13 was processed to:

- determine correlation coefficients between variables

- explain flotation results: tREO, RREO, EREO, SREO with characteristics of the collectors as the independent variables.

Correlation coefficients between all 22 variables: 10 variables relevant to P.E. characterization, 12 variables relevant to REO concentration performance in F2, FC and F4, are listed in a table shown in appendix 6. The most interesting correlations excerpted from this table are shown below:

File REO2SY03 Page 1-1

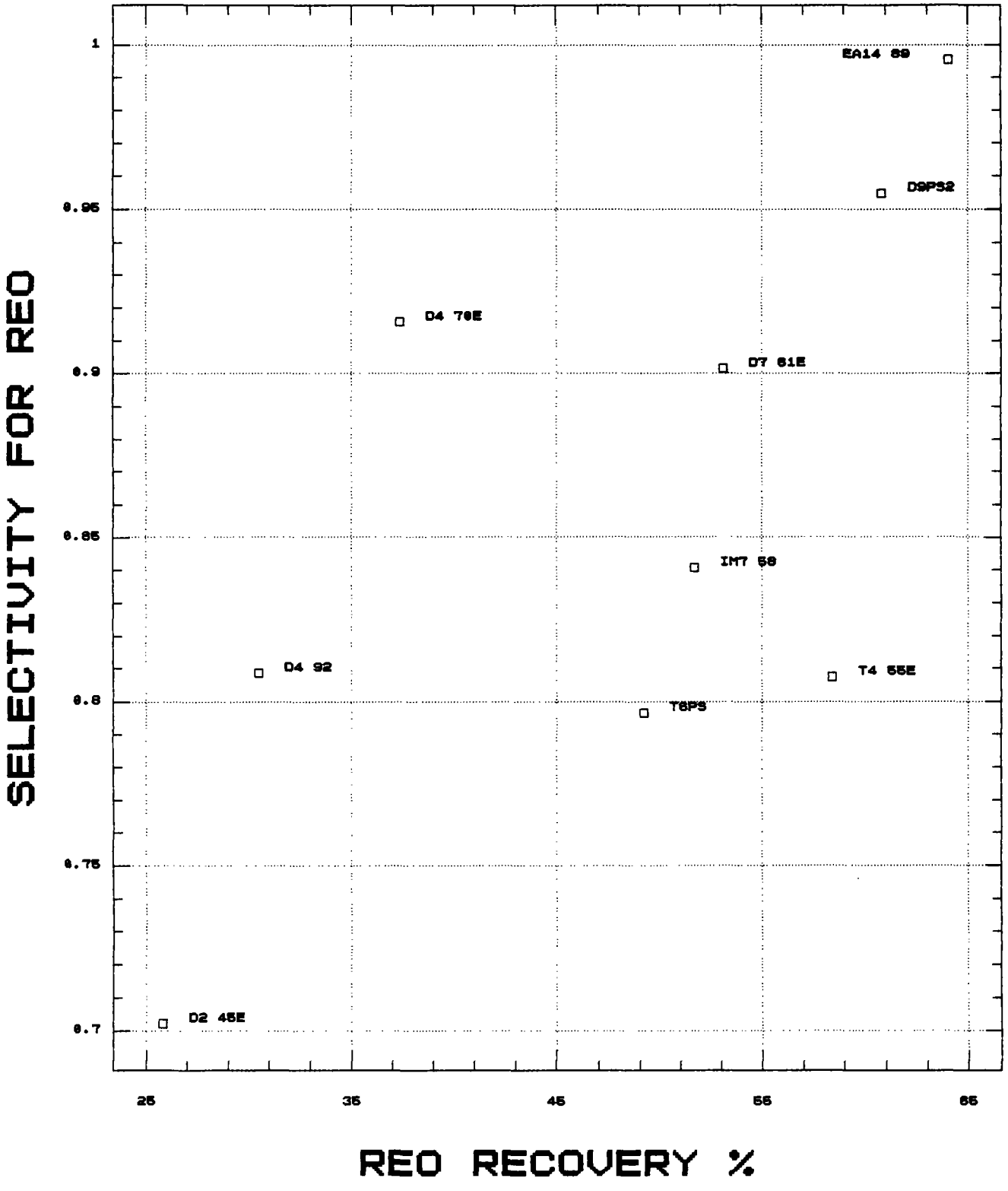
row	NbC	n	MEO	DEO	NPP	FOPA	FPPA	MEP	DEP	tREOF2	RREOF2	EREOF2	SREOF2	tREOFC	RREOFC	EREOFC	SREOFC	tREOF4
1	12	2	34.6	41.3	4.5	1.1	1.2	11.7	16.3	60.90	25.80	18.12	0.7022	58.91	33.15	20.85	0.6290	52.9
2	12	7	51.2	32.3	7.7	1.5	0.8	14.1	8.9	66.30	53.12	47.89	0.9015	65.72	71.82	63.22	0.8803	64.2
3	14	7	51.0	34.3	15.7	0.8	0.2	2.7	4.5	64.61	51.72	43.49	0.8408	63.47	63.35	50.65	0.7995	58.9
4	12	9	43.6	43.2	9.4	0.7	0.2	3.0	4.3	67.78	60.80	58.05	0.9547	64.98	79.31	67.48	0.8509	57.2
5	12	4	71.9	6.5	23.2	3.5	0.0	0.0	0.0	63.65	30.50	24.66	0.8086	61.33	51.72	37.53	0.7256	58.3
6	12	4	63.0	24.9	4.5	2.3	0.5	4.9	3.7	66.64	37.39	34.24	0.9157	64.32	54.42	45.33	0.8329	59.8
7	13	4	46.3	31.0	4.8	1.2	1.0	7.4	13.5	64.03	58.44	47.18	0.8074	62.84	74.68	56.85	0.7612	60.4
8	13	6	46.9	32.2	15.6	0.7	0.8	5.9	2.6	64.26	49.20	39.19	0.7966	64.60	68.30	55.40	0.8112	65.5
9	21	14	44.5	6.3	33.6	1.1	0.9	8.7	0.5	68.88	64.04	63.75	0.9954	65.71	79.95	69.92	0.8746	55.4

File REO2SY03 Page 1-2

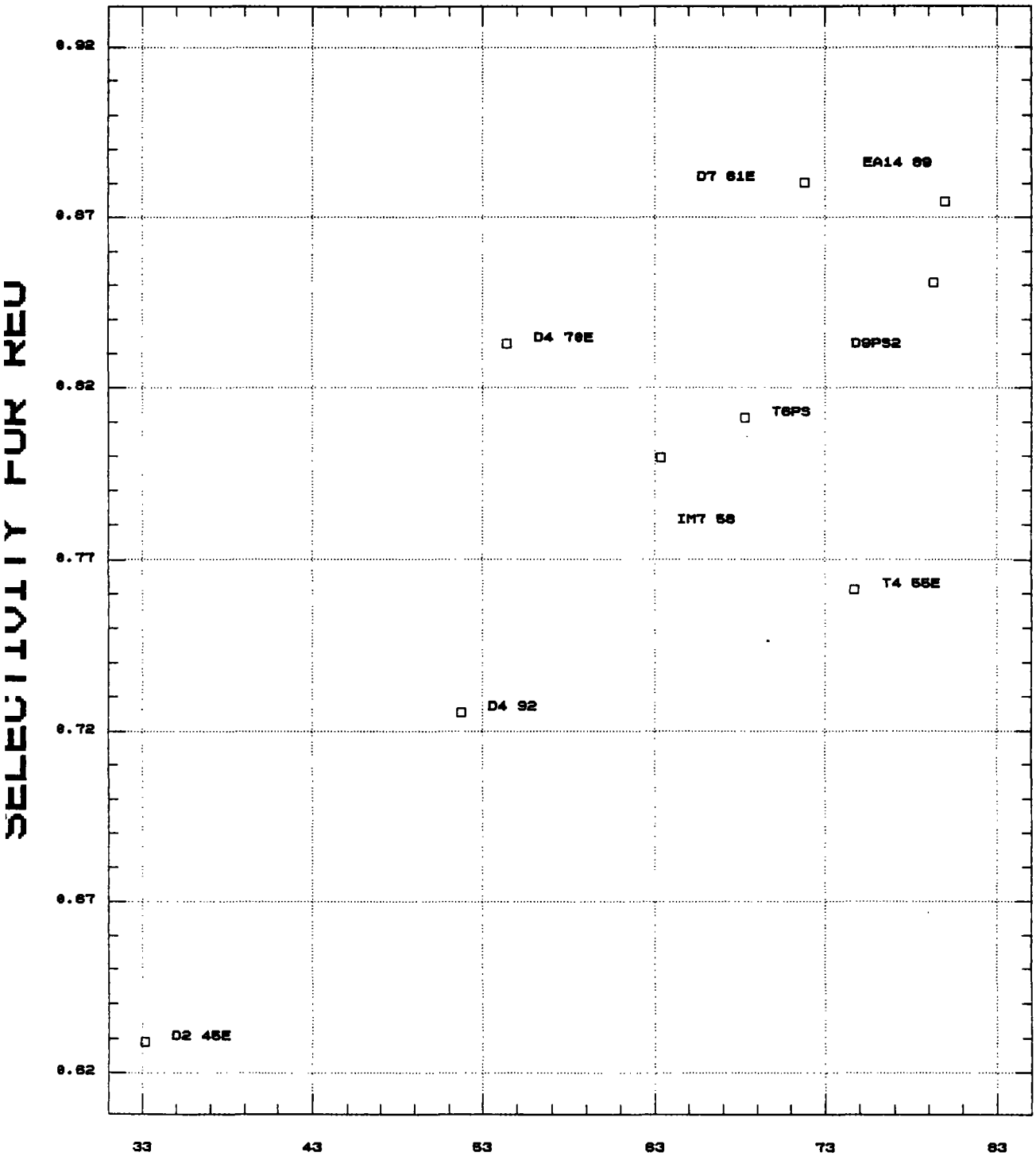
row	RREOF4	EREOF4	SREOF4	MDRATIO	PROMOTER
1	35.46	17.76	0.5008	0.83777	D2 45E
2	56.36	47.22	0.8378	1.58514	D7 61E
3	44.37	32.50	0.7325	1.48688	DM7 58
4	68.66	45.80	0.6670	1.00925	D9PS2
5	60.42	39.55	0.6546	11.06153	D4 92
6	56.04	39.79	0.7100	2.53012	D4 70E
7	57.51	45.09	0.7840	1.49354	T4 55E
8	44.99	40.01	0.8894	1.45652	T6PS
9	77.72	45.75	0.5887	7.06349	EA14 89

Table 13: Flotation tests on REO ore sample 2 with phosphoric esters as collectors. Summary table

REO ORE SAMPLE 2
PERFORMANCE OF P.E.COLLECTORS (F2)



REO ORE SAMPLE 2
PERFORMANCE OF P.E.COLLECTORS (FC)



Variables	correlation coefficient	
tREOF2 - n	0.8288	(P= 0.0058)
tREOF2 - DEP	- 0.5853	(0.0985)
RREOF2 - n	0.7714	(0.0149)
RREOF2 - FOPA	- 0.5786	(0.1026)
EREOF2 - n	0.8690	(0.0024)
SREOF2 - n	0.8134	(0.0077)
SREOF2 - DEP	- 0.5722	(0.1074)
tREOFC - n	0.7282	(0.0261)
RREOFC - n	0.7455	(0.0211)
EREOFC - n	0.8012	(0.0094)
SREOFC - n	0.7321	(0.0249)
MEO - FOPA	0.8674	(0.0024)
DEO - NPP	- 0.7744	(0.0143)
NPP - FPPA	- 0.2327	(0.5468)
MEP - NPP	- 0.2741	(0.4754)
DEP - NPP	- 0.7190	(0.0290)
MDRATIO - FOPA	0.7494	(0.02)

Comparison of correlation coefficients (between characteristics of the P.E. collectors) drawn from first (REO sample 1) and second (REO sample 2) series, indicates that introduction of PE89122 EA14-89 which derives from ether-alcohol, strongly decreases some correlations as shown below:

Variables	1st series	2nd series including EA14-89	2nd series excluding EA14-89
MEO - FOPA	0.900	0.8674	0.8655
DEO - NPP	- 0.6521	- 0.7744	- 0.6053
NPP - FPPA	- 0.7211	- 0.2327	- 0.6844
MEP - NPP	- 0.6891	- 0.2741	- 0.6579
DEP - NPP	- 0.7396	- 0.7190	- 0.7350
MDRATIO - FOPA	0.8926	0.7494	0.8942

With reference to the REO ore sample 1 series of tests, it is worthy to note that both selectivity and concentration efficiency for monazite are positively correlated to n (number of Ethylene oxide groups), this also applies to REO content and recovery, whereas for the REO ore sample 1:

- P_2O_5 content and n were negatively correlated
- P_2O_5 recovery and n were positively correlated
- selectivity for P_2O_5 and n were negatively correlated.

These differences in the effect of n on selectivity and concentration efficiency could be attributed to a fundamental change in the mechanism of P.E. adsorption on to the surface of monazite in CO_2 containing pulp, with respect to that prevailing for apatite. It

should also be kept in mind that flotation of apatite was conducted at high pulp pH (about 9.5) instead of a pulp pH of 5.3 used to float monazite.

Predictor variables were selected by stepwise multiple linear regression applied to data shown in table 13.

Regression equations shown below, led to the best fits to flotation data:

* EREOF2= 19.85 + 5.008 n - 0.735 NPP
  with df: degree of freedom for error: 6
  R^2(A): R-squared Adjusted for d.f.: 0.8447
  F ratio: 22.785 (P=0.0016)
  S.E.: standard error of estimate: 5.80

* SREOF2= 0.4844 + 0.0362 n + 0.0046 MEO - 0.0065 NPP
  df= 5, R^2(A)= 0.9286
  F= 35.68 (P= 0.0008)
  S.E.= 0.0244

* tREOF2= 55.66 + 0.9615 n + 0.1137 MEO - 0.1698 NPP
  df= 5, R^2(A)= 0.9328
  F= 38.03
  S.E.= 0.628

* RREOF2= 22.9408 + 4.3372 n - 0.72 NPP
  df= 6, R^2(A)= 0.6518
  F= 8.49 (P= 0.0178)
  S.E.= 8.03

* EREOFC= 30.4272 + 4.9090 n - 0.7263 NPP
  df= 6, R^2(A) = 0.6711
  F= 9.16 (P= 0.015)
  S.E.= 8.95

* SREOFC= 0.4712 + 0.0323 n + 0.0042 MEO - 0.0068 NPP
  df= 5, R^2(A)= 0.8374
  F= 14.74 (P= 0.0065)
  S.E.= 0.0325

* tREOFC= 49.787 + 0.6146 n + 0.2614 MEO - 0.1032 NPP - 2.0986 FOPA + 0.1662 MEP
  df= 3, R^2(A)= 0.8408
  F= 9.45
  S.E.= 0.893

or tREOFC= 55.380 + 0.8632 n + 0.1003 MEO - 0.1780 NPP
  df= 5, R^2(A)= 0.7295
  F= 8.19
  S.E.= 1.164

* RREOFC= 43.8219 + 3.1983 n
  df= 7, R^2(A)= 0.4924
  F= 8.76 (P= 0.0211)
  S.E.= 10.91

Quality of the fit to flotation data (EREOFC and SREOFC) provided by the regression equations is shown in tables and diagrams at the end of the paragraph 4.3.2.

Conclusions drawn from regression analysis are as follows:

- REO recovery in rougher-cleaner concentrate F2 or composite concentrate (FC= F2+F4) are not satisfactorily explained by variables relevant to characterization of P.E. collectors. This could be ascribed to variables not taken into consideration in the regression models such as the occurrence of composite particles related to the degree of liberation of the minerals in the gravity preconcentrates, it is interesting to note that the fit to flotation performance data sharply drops for FC with respect to F2, this could indicate increasing variability due to response of composite particles to flotation in the scavenger-cleaner stage. Liberated particles float readily in the rougher-cleaner stage. It should also be pointed out that poorly controlled variables such as the froth depth or height of the froth-slurry interface, the manual froth removing process, could affect monazite recovery.

The number n of Ethylene Oxide groups in P.E. strongly affects REO recovery, increase in REO recovery with n reflects enhancement of collecting power of P.E. However, it should be noted that n and NbC are highly correlated (0.8112), hence the effect could be attributed to both n and NbC. REO or monazite recovery is also detrimentally affected by increasing contents of non phosphated products (NPP), this effect could be expected since the proportion of collector molecules (phosphoric ester molecules) decreases with increasing contents of residual NPP.

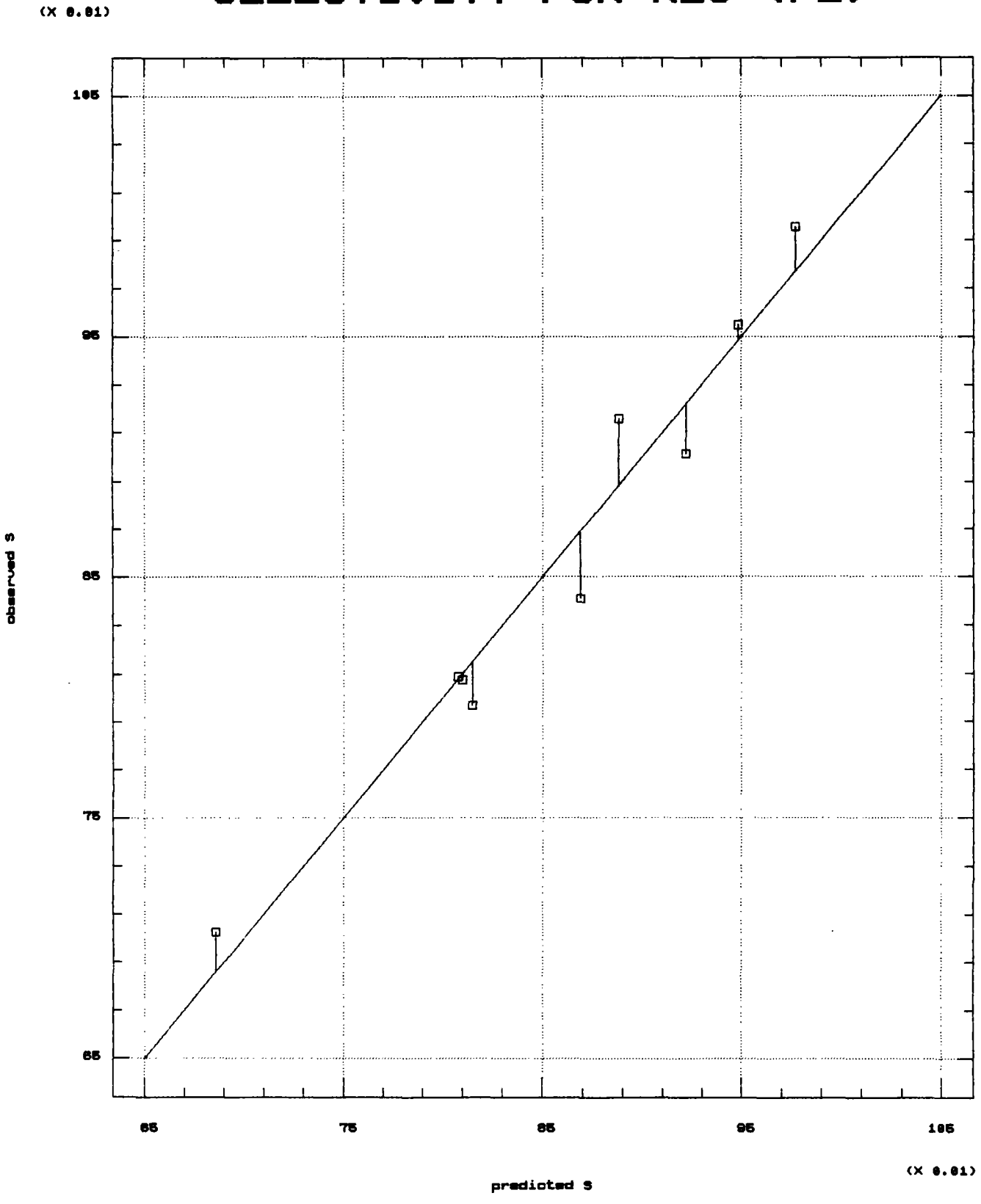
Concentration efficiency E for REO reflects both selectivity and recovery, since recovery appears to be not well explained by variables related to the collector reagents, the latter are not satisfactory explicative variables for E, particularly for FC concentrates. However, the regression equation for EREOF2 highlights how characteristics of the P.E. affect concentration efficiency: E improves with increasing n and decreasing NPP.

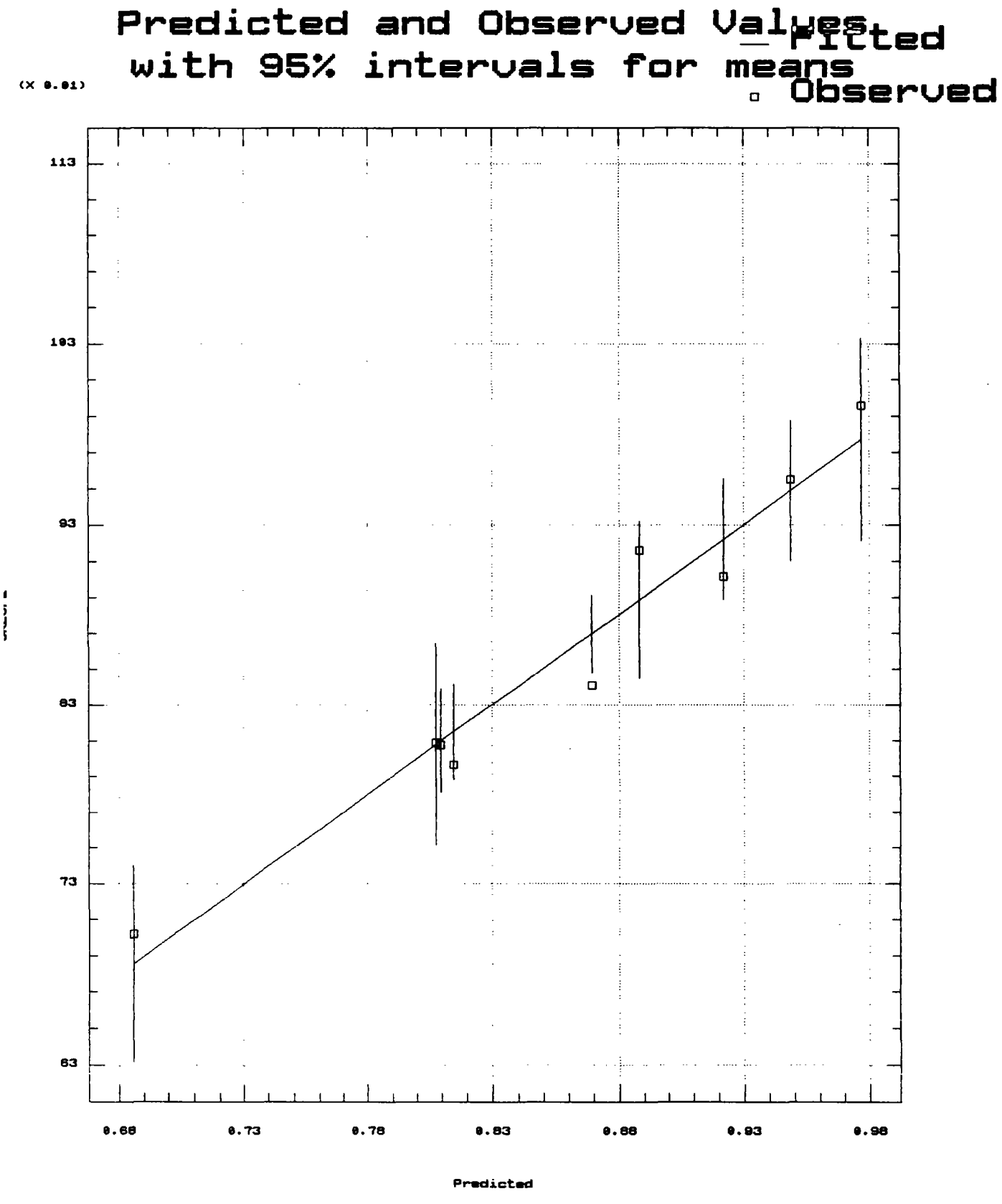
- High n values within the experimental range also appears to be beneficial to REO content and selectivity. Low contents of residual non phosphated products also favours high REO contents in concentrates, thus low NPP improves selectivity for REO.

Two major characteristics of P.E.: n and NPP exert parallel beneficial influences on flotation results: REO contents and recoveries, these effects are not commonly observed. This reflects higher adsorption density of the collector on to the surface of monazite particles at high n values whereas adsorption density on gangue particles is little affected by n. It is worthy to note that adsorption of P.E. on to the surface of monazite involves REE-phosphoric ester interactions whereas adsorption of P.E. on to the surface of gangue particles involves Ca, Mg, Ba, Sr - phosphoric ester interactions. Increasing NPP merely decreases phosphoric esters concentration which is detrimental to both recovery and selectivity.

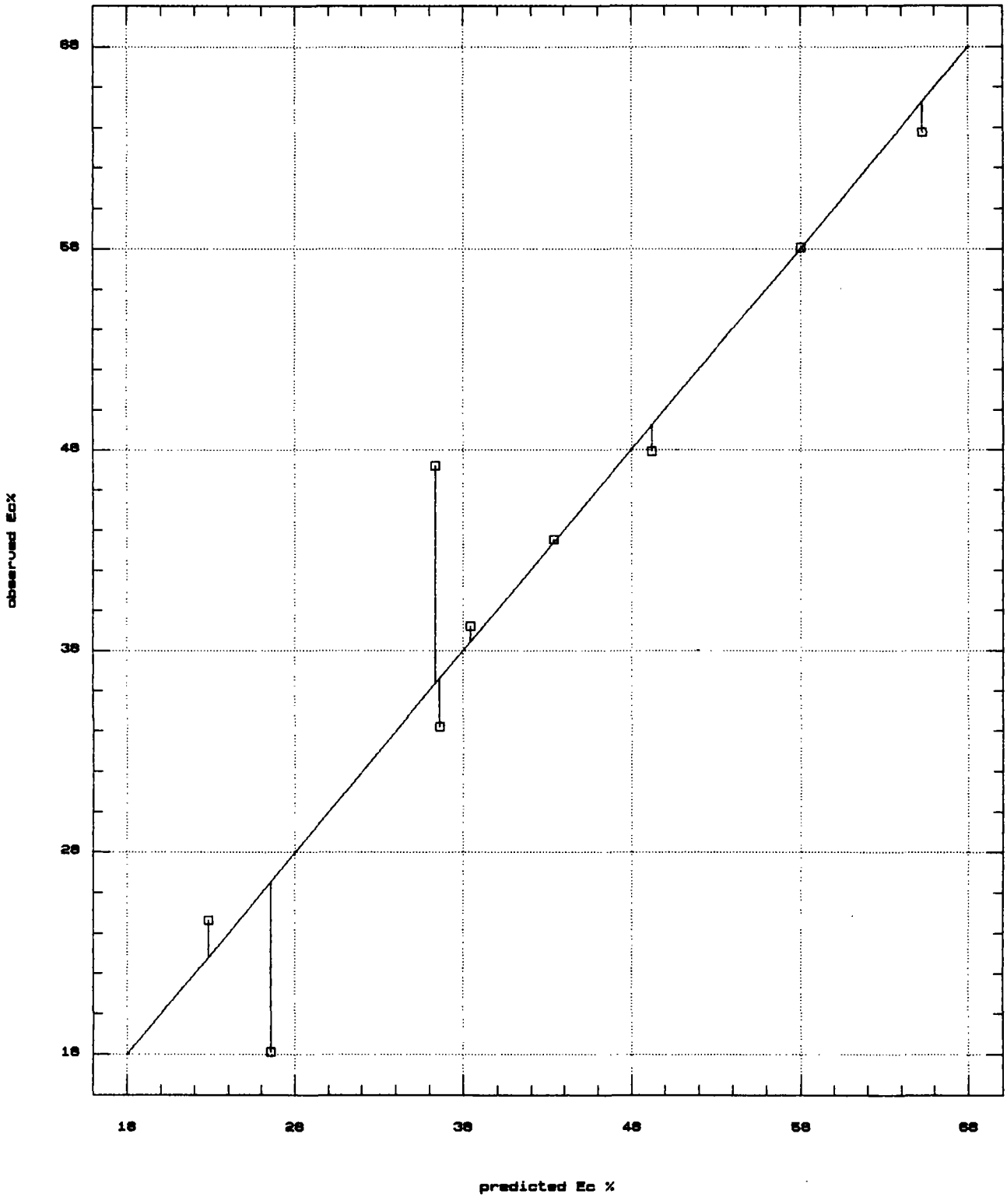
- REO contents in concentrates F2 or FC as well as selectivities increase with mono orthophosphoric ester concentration (MEO) in collector. High concentration of free orthophosphoric acid (FOPA) appears to lower REO content in concentrates, this is consistent with the depressant effect of H_3PO_4 on phosphate minerals. At relatively low pulp pH (about 5.3), high concentrations of mono esters (ortho and pyro) appear to favour selectivity for monazite with respect to carbonate minerals.

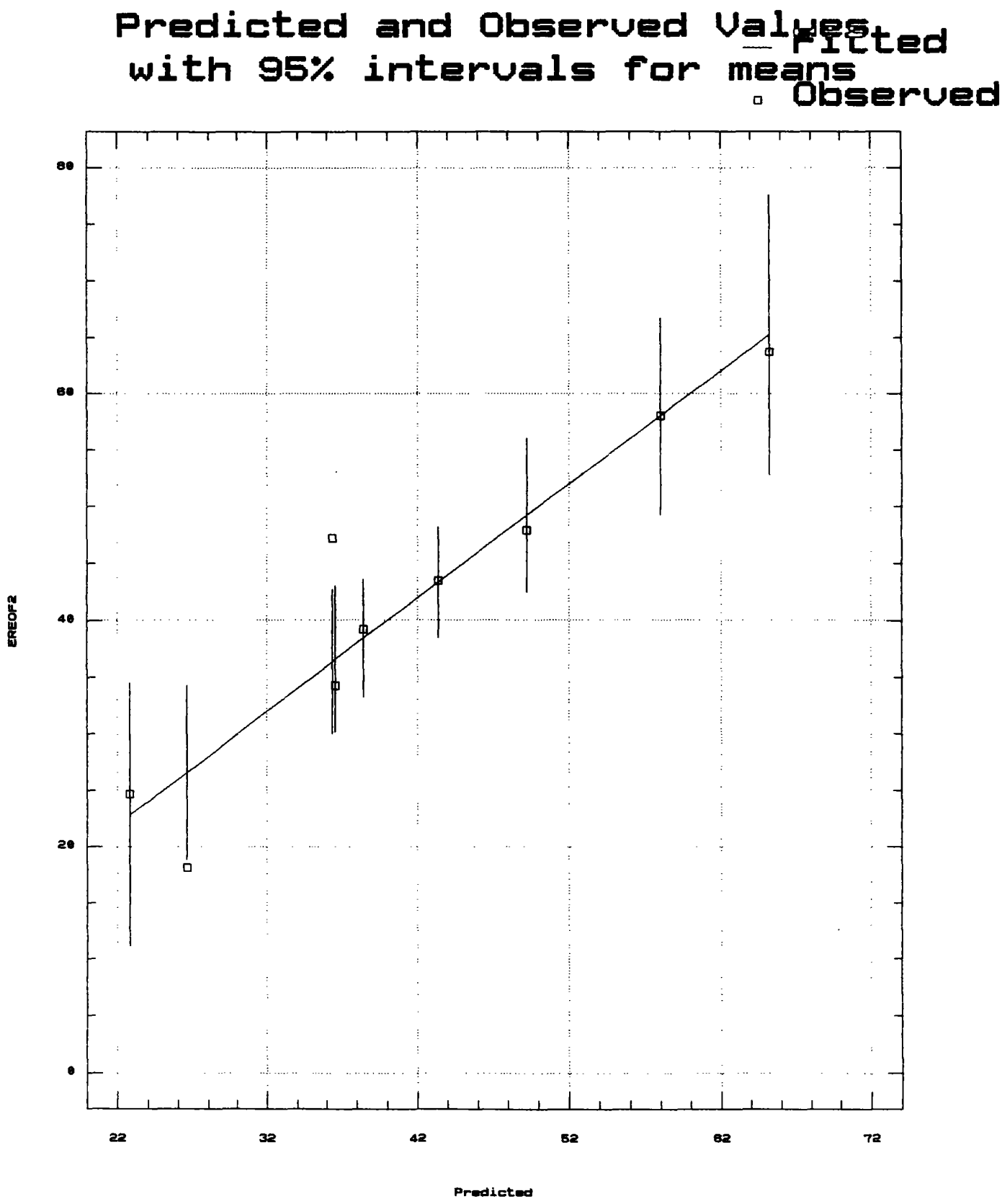
REO ORE SAMPLE 2
SELECTIVITY FOR REO (F2)

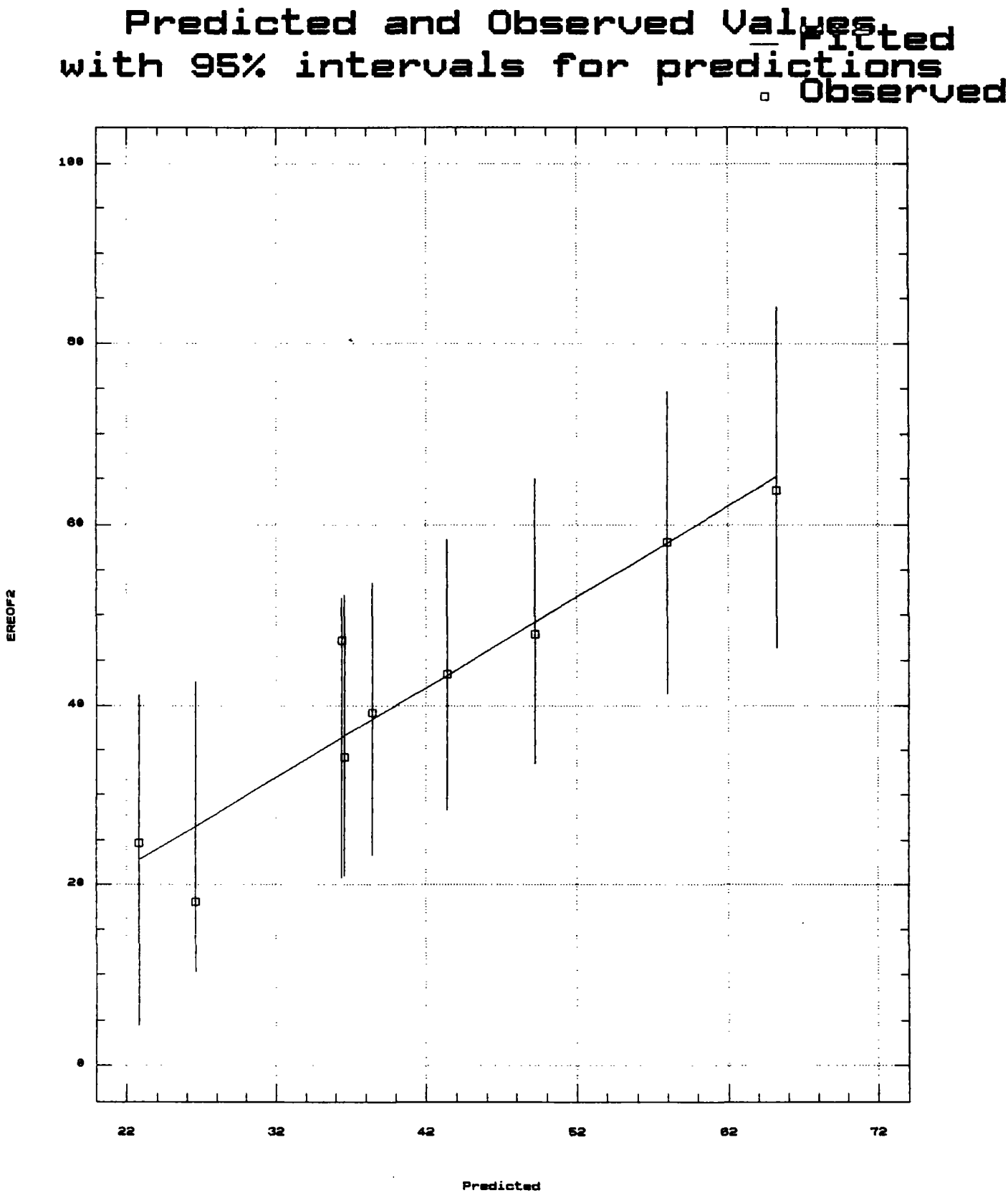



Predicted and Observed Values
with 95% intervals for means
Fitted
Observed
(X 0.01)
113
103
93
83
73
63
0.68
0.73
0.78
0.83
0.88
0.93
0.98
Predicted

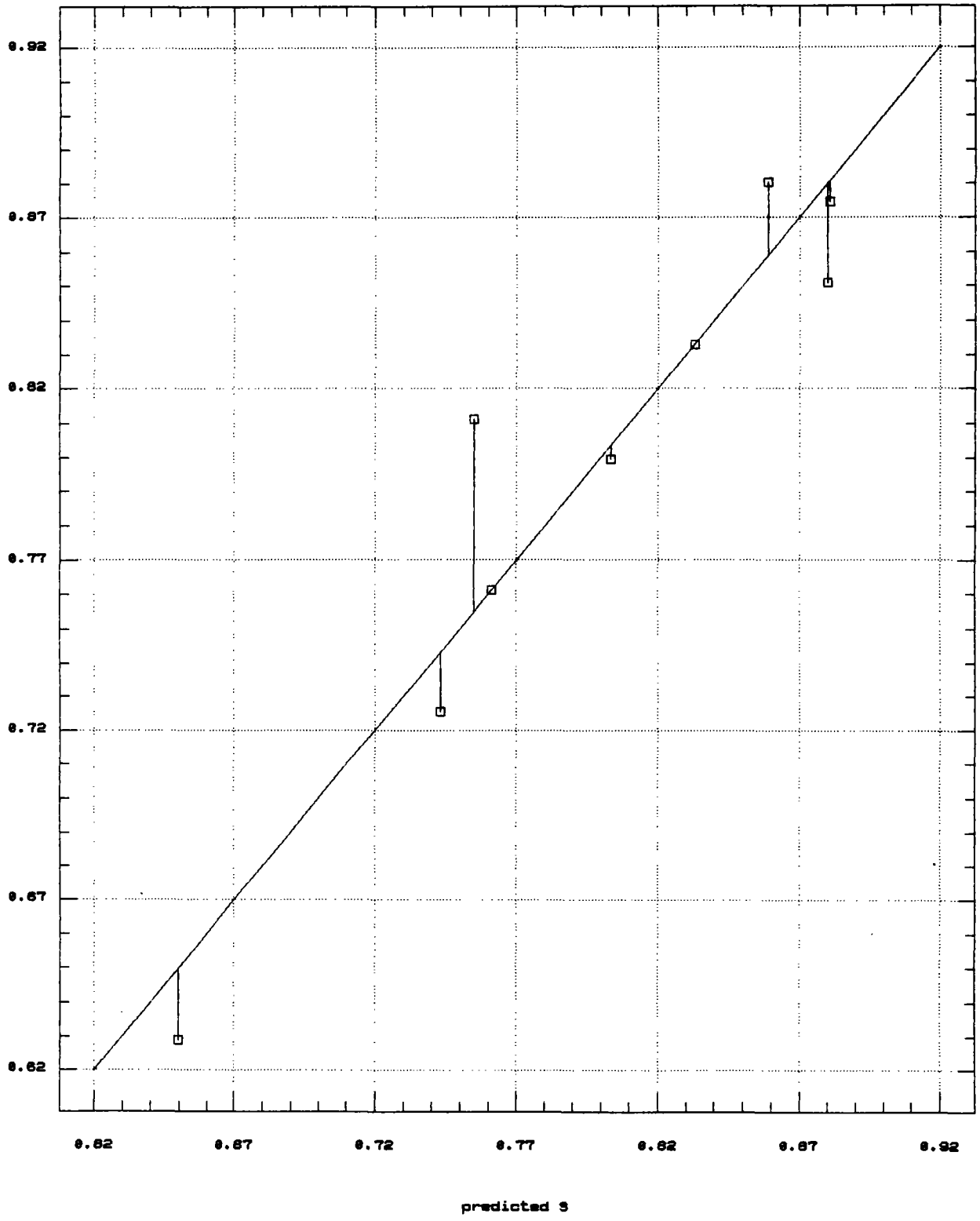
REO ORE SAMPLE 2
CONCENTRATION EFFICIENCY FOR REO

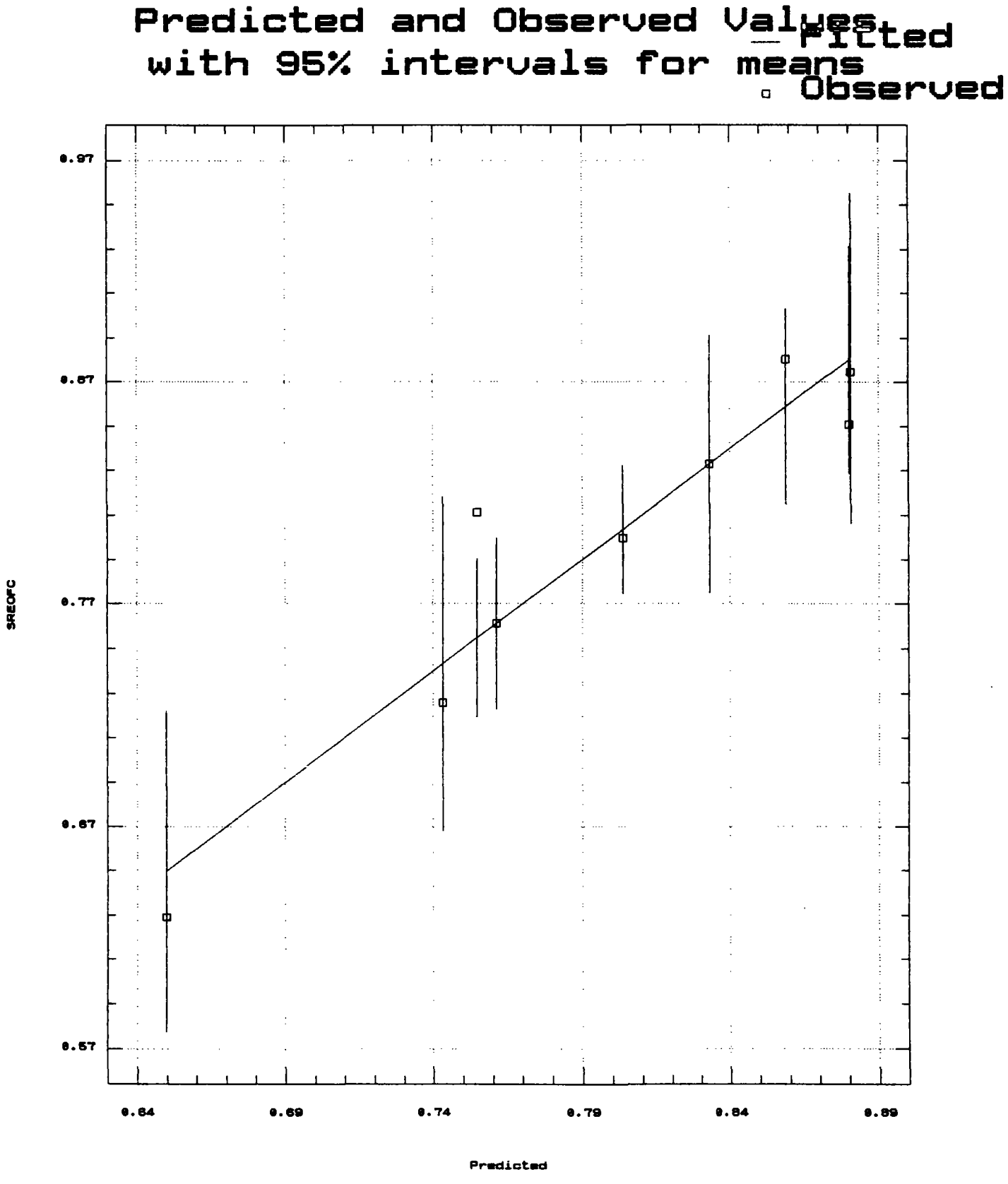



Predicted and Observed Values
with 95% intervals for means
Fitted
Observed
80
60
40
20
0
EREOF2
22
32
42
52
62
72
Predicted

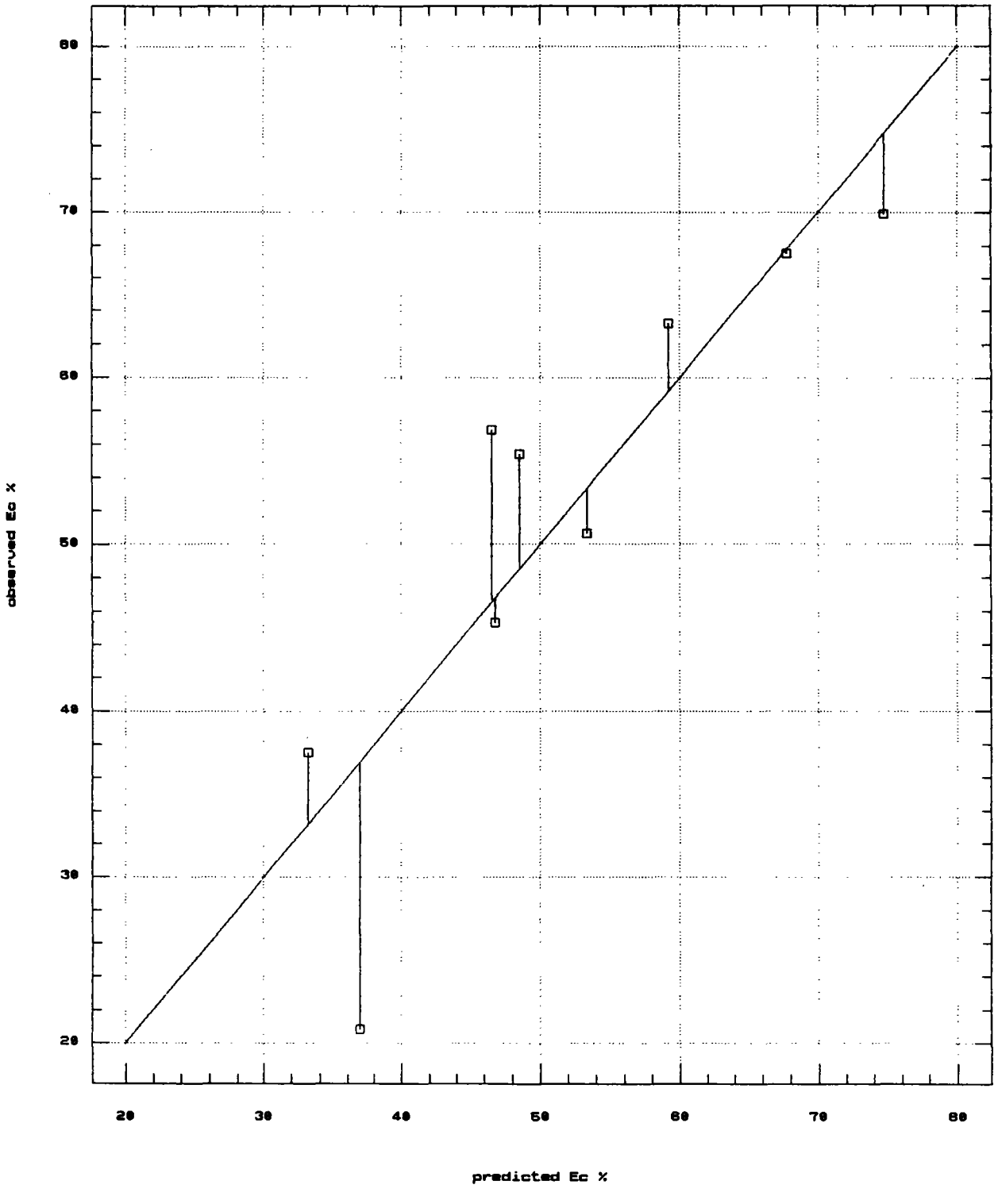

Predicted and Observed Values
with 95% intervals for predictions
Fitted
Observed
EREOF2
100
80
60
40
20
0
22
32
42
52
62
72
Predicted

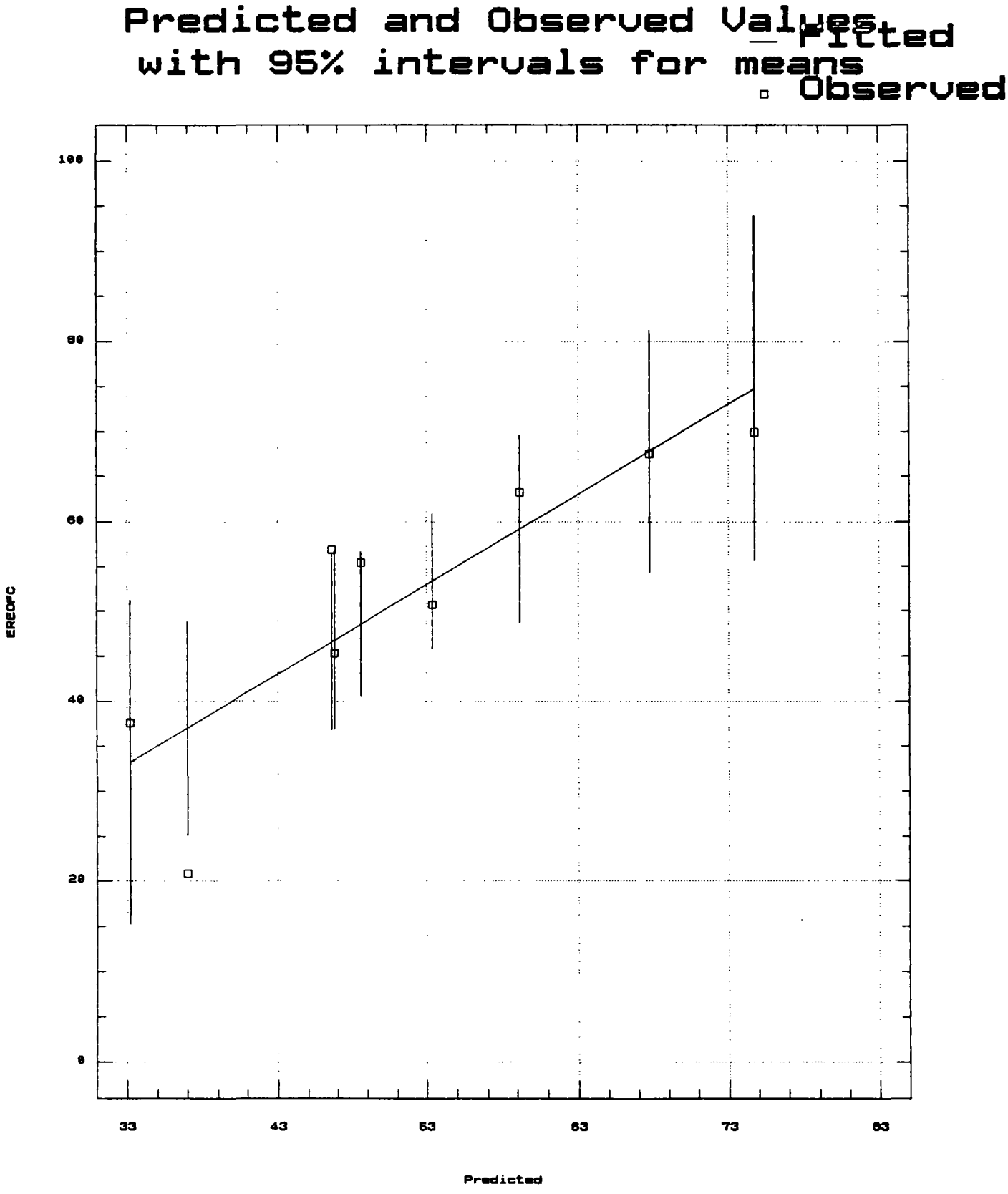
REO ORE SAMPLE 2
SELECTIVITY FOR REO (F2+F4)




Predicted and Observed Values
with 95% intervals for means
Fitted
Observed
0.97
0.87
0.77
0.67
0.57
SREOFC
0.64
0.69
0.74
0.79
0.84
0.89
Predicted

REO ORE SAMPLE 2
ONCENTRATION EFFICIENCY FOR REO (F2+F4)




Predicted and Observed Values
with 95% intervals for means
Fitted
Observed
100
80
60
40
20
0
EREOFC
33
43
53
63
73
83
Predicted

5. CONCLUSION

Evaluation of phosphoric ester collectors for flotation of rare earth minerals was conducted on three ore samples:

- REO ore sample 1 consisting of: Y and REE containing apatites as the major REE bearing minerals, REE bearing silicates: Y-britholite, Ce-britholite, allanite as minor REE bearing minerals, clinopyroxene, magnetite, biotite, albite, amphibola, ilmenite, zircon, iron carbonate, pyrite and clay minerals: kaolinite, smectite, chlorite, sepiolite and talc as gangue minerals

- REO ore sample 2 consisting of: monazite as the major REE bearing mineral, accounting for more than 95 % of the total REE contained in the ore, apatite and traces of bastnaesite or parisite as minor REE bearing minerals, carbonate minerals: Mn and Fe containing dolomite, strontianite, siderite, calcite, dialogite as major gangue minerals, barite, manganese oxides, magnetite and quartz as other gangue minerals

- REO ore sample 3 consisting of: bastnaesite as the major REE bearing mineral, REE containing phosphates as minor REE minerals, fluorite and barite as potentially valuable minerals, muscovite, phlogopite, chlorite, quartz, calcite, dolomite, iron and manganese oxides as major gangue minerals.

Contents of REE and Y in ore samples are as follows:

	REO ore sample		
	n° 1	n° 2	n° 3
(REE + Y) %	0.746	4.483	7.512
(Eu + Y) / (REE + Y)	0.1329	0.0033	0.0063

The REO ore sample n° 1 with the lowest content of total REE exhibits the most favourable composition in terms of proportions of Eu and Y.

Performances of separations by flotation were estimated on the basis of a maximum P_2O_5 content of 35.8 % in apatite concentrates and maximum total REO content of 69-71 % in monazite concentrates.

Flotation tests were carried out on:

- the - 160 + 10 μm fraction separated from the attrition-scrubbed, rod-milled down to about 160 μm and deslimed REO ore sample 1, purged of magnetite by low intensity magnetic separation

- fractions-separated from the attrition-scrubbed, rod-milled down to about 160 μm, and deslimed REO ore sample 2:
  - - 160 + 10 μm and - 50 + 10 μm
  - - 80 + 10 μm and - 40 + 10 μm gravity preconcentrates.
- fractions separated from the attrition-scrubbed, rod-milled down to about 125 μm, REO ore sample 3:
  - - 125 + 10 μm and - 10 μm primary slimes.

Preliminary flotation tests were effected so as to assess the amenability of ores to flotation using conventional flotation reagents and phosphoric ester collectors then define standard flowsheets to compare performances of various types of phosphoric ester promoters.

Preliminary tests on the - 160 + 10 μm fraction from the REO ore sample n° 2 led to a standard flotation procedure using phosphoric ester as an apatite collector in an alkaline pulp pH of about 9.5, a solution of sodium silicate and citrate in combination with corn starch as a gangue depressant.

Tests made on the - 160 + 10 μm and - 50 + 10 μm fractions from the REO ore sample n° 2 were unsuccessful to separate monazite from a high carbonate gangue. Removal of part of the Fe-Mn containing dolomite by gravity separation led to gravity preconcentrates amenable to flotation. A standard flotation procedure was developed to separate monazite from residual dolomite, siderite, strontianite and barite, monazite was floated with phosphoric ester as a collector in a slightly acidic pulp pH of about 5.3, using CO_2 as a strontianite, barite and gangue depressant.

Attempts to separate bastnaestite by flotation from the - 125 + 10 μm and - 10 μm (1) fractions of the REO ore sample n° 3, were unsuccessful, this is attributed to poor liberation of bastnaesite in the coarsest fraction and poor selectivity of phosphoric esters for bastnaesite in the primary slimes fraction which is extremely fine with a median diameter of 2.7 μm. However, preliminary flotation tests showed that phosphoric ester could be used as a fluorite collector in a slightly acidic pulp pH.

Ten phosphoric ester collectors were evaluated in systematic flotation tests conducted on REO ore samples n° 1 and 2:

Collector	coded composition	order for flotation tests	
		REO ore 1	REO ore 2
MELIORAN D2-45E	D2-45E	1	1
MELIORAN D7-61E	D7-61E	2	2
PE 89124	IM7-58		3
P312	D9PS2 (D9-50)	3	4
B110	D4-92	4	5
B110 88094	D4-70E	5	6
MELIORAN T4-55E	T4-55E	6	7
P301	T6PS (T6-59)	7	8
B110 69032	D4-66	8	
PE 89122	EA14-89		9

Eight types of phosphoric ester collectors, listed in the previous table, were compared for their performance to concentrate Y and REE containing apatite by flotation, using the standard flowsheet developed on the - 160 + 10 μm fraction of the REO ore sample n° 1, under preliminary tests. The collector B110 88094 led to the best concentration performance with low dosages of 150 g/t in rougher flotation and 75 g/t in scavenger flotation. Apatite concentrates meet specifications for their potential use as a phosphatic material suitable for conversion into wet process phosphoric acid:

	concentrate F3	composite concentrate F3 + F4	feed to flotation
P_2O_5 content %	33.10	31.82	3.42
Equivalent in Tri Calcium phosphate %	72.32	69.53	7.47
R_2O_3 % content %	1.37	1.75	14.61
MgO content %	0.50	0.53	1.64
$(R_2O_3 + MgO) / P_2O_5$	0.0565	0.0716	4.7515
CaO/P_2O_5	1.400	1.437	2.263
total REE + Y content %	4.3172	4.3866	0.7642
Eu content %	0.0155	0.0150	0.0020
Y content %	0.8080	0.8015	0.1006
total REE + Y recovery %	37.45	50.40	100
Eu recovery %	50.87	65.15	100
Y recovery %	53.24	69.94	100
apatite content %	92.5	88.9	9.55
P_2O_5 recovery %	64.54	81.93	100

Satisfactory grade concentrates were produced at acceptable phosphate recoveries: 64.54 and 81.93 % on flotation feed basis, or 62.71 and 79.61 % on head basis. Lower REE + Y, Y and Eu recoveries account for REE and Y associated to silicates or silico-phosphates which were not floated with phosphoric esters as collectors.

Flotation in a continuous circuit would probably lead to increased phosphate recovery in the composite phosphate concentrate F3 + F4 due to recirculation of middlings.

Nine types of phosphoric ester collectors were compared for their performance to concentrate monazite by flotation from the - 80 + 10 μm gravity preconcentrate (separated from the REO ore sample 2, using the standard flowsheet developed under preliminary tests. The best concentration performances were achieved with PE89122, EA14-89, for extremely low additions of 50 g/t in rougher flotation and 20 g/t in scavenger flotation:

	concentrate F2	composite concentrate F2 + F4	feed to flotation
Fe_2O_3 content %	< 1	1	4.39
SrO content %	1.33	1.52	9.85
BaO content %	0.75	0.72	5.25
total REO content %	68.88	65.70	42.77
total REO recovery %	64.04	79.94	100.00
monazite content %	~ 97	95.22	61.98

flotation in a continuous circuit including recirculation of middlings or optionally regrinding to improve liberation of monazite, would probably increase REO recovery in the composite concentrate.

Stepwise multiple linear regression techniques applied to analysis of flotation test data have shown that:

- the collecting power of phosphoric ester collectors strongly increases with the number n of ethylene oxide groups in the molecule.

Apatite or monazite recoveries in flotation concentrates separated from the REO ore samples n° 1 or n° 2, increase with n. However, P_2O_5 content in concentrates produced from the ore n° 1, concentration efficiency and selectivity for P_2O_5, were found to be detrimentally affected at high n values, whereas n was found to exert a positive influence on both REO contents in concentrates produced from the ore n° 2 and selectivity for REO.

Differences in the effects of n on selectivity for apatite and for monazite might be attributed to differences in the mechanism of phosphoric ester adsorption on to the surface of apatite and monazite,

the former involves predominant Ca - phosphoric ester interactions at high pulp pH (9.5) while the latter involves REE - phosphoric ester interactions at lower pulp pH (5.3).

- REO recovery in flotation concentrates separated from the ore n° 2 increases as the content in residual non phosphated matter (NPP) of the phosphoric ester collector decreases. This could be related to increasing concentrations of phosphoric esters in the commercial products as the non phosphated matter is reduced

- selectivity for P_2O_5 or REO as well as P_2O_5 or REO contents in flotation concentrates separated from the ore n° 1 or the ore n° 2, increase with concentration of mono orthophosphoric ester (MEO) in commercial collectors

- selectivity for P_2O_5 is detrimentally affected by high concentration of free orthophosphoric acid (FOPA) in commercial collectors. This reflects the depressant effect of H_3PO_4 for apatite or other phosphated minerals. To a lower extent FOPA is also found to impair selectivity for monazite

- selectivity for REO decreases as content in NPP increases, this is related to reduction of MEO concentration in commercial collectors and possibly to increasing depressant effect on monazite.

- characteristics of the phosphoric ester collectors provide variables to satisfactorily explain variations relevant to selectivity. Recovery of valuable minerals (apatite or monazite) and concentration efficiency are not so well explained by characteristics of the collectors. Occurrence of composite particles and variations in operating conditions of flotation might also account for part of variations observed in recovery of valuable minerals

- concentration efficiency for P_2O_5 strongly depends on the selectivity term while concentration efficiency for REO is governed by the recovery term.

The most efficient phosphoric ester collectors for REE and Y containing apatite should have a high content in MEO, a low concentration in residual H_3PO_4 and an appropriate number n of ethylene oxide groups giving satisfactory apatite recovery with moderate detrimental effect on the apatite content of the concentrates.

Within the experimental range explored, the most effective phosphoric ester collectors for monazite are characterized by high n values, low concentrations in NPP and H_3PO_4 and high concentrations in MEO.

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SERVICE REPROGRAPHIE

APPLICATION OF PHOSPHORIC ESTERS TO THE FLOTATION OF FINELY DIVIDED OXIDIZED ORES:

SYNTHESIS REPORT

G. Baudet

March 1991

BUREAU DE RECHERCHES GEOLOGIQUES ET MINIERES
Direction Mines Moyens
Département Minéralurgie
B.P. 6009 - 45060 Orléans Cedex 2 - France - Tel. : (33) 38 64 34 34

G. BAUDET — Bureau de Recherches Géologiques et Minières - Mineral Technology Department
Orléans - France

G. MORTEANI — Technische Universität MUNCHEN
Lehrstuhl Für Angewandte Mineralogie und Geochemie
Munich F.R.G.

M.P. STRUB — C.E.C.A.
Laboratoire Recherche Application
CORBEHEM - France

C.E.C. RESEARCH CONTRACT MAIM - 0059 - C

1. OBJECTIVES:

The objectives of the Research programme are:

- to develop new phosphoric ester (P.E.) derived flotation collectors with optimum performance for concentration of finely divided oxidized Rare Earth Elements bearing ores (R.E.E.)

- to define R.E.E. bearing minerals occurring in typical ores

- to develop flotation processes using P.E. derivatives, alone or in conjunction with other promoters, as collectors applicable to difficult to beneficiate R.E.E. ores. This could promote the development of new deposits which would constitute new potential R.E.E. sources for European producers.

2. INTRODUCTION:

Preliminary laboratory studies have shown that particular types of phosphoric esters, jointly developed by GERLAND (at present C.E.C.A.) and BRGM [2] [3], for separating carbonates from phosphate by flotation of sedimentary high carbonate phosphate rock, exhibit good selectivity and strong collecting properties for certain finely oxidized divided minerals. This specially applies to rare earth oxides (R.E.O.), as an example a high R.E.O. concentration ratio value of 11 was achieved using a P.E. as a collector, on a - 20 μm stabilized slimes sample containing less than 1 % total R.E.O. as monazite [1], associated with clays, silica, feldspar and iron minerals.

Despite a R.E.O. world production well below the mining capacity, it appears that R.E.O. supply to Europe and production of refined derivatives are sensitive to the following factors:

- increased semi-refining and refining capacities in R.E.O. concentrates producing countries,

- fluctuating demand in certain rare earth derivatives depending upon end uses and new developing applications. This is related to the relative amounts of rare earth elements contained in bastnaesite, monazite and xenotime concentrates,

- occurrence of radio active Th in high amount in most of the monazite concentrates, which are widely used in Europe, along with a significantly lower total R.E.O. content with respect to bastnaesite concentrates dominantly used by the refining plants in the U.S.A.,

- insufficiently diversified sources of concentrates and low acceptable grade ore reserves,

- emergence of China as a major producer of both R.E.O. concentrates, with high Eu content, and refined oxides.

With a view to increase diversification and quality of European sources of R.E.O. concentrates it would be desirable to evaluate new potentially interesting deposits the development of which closely depends upon performance of beneficiation. This applies to flotation which appears as the most flexible and promising process for recovering R.E.O. values from difficult to beneficiate ores containing finely divided R.E.O.

The collaborative research project between BRGM, the Technical University of Munich, C.E.C.A. laboratories was developed to investigate the response to flotation of R.E.E. minerals with P.E. as collectors.

The research program covers the following:

BRGM and the Technical University of MUNICH	. selection and characterization of R.E.E. ore samples, with special attention given to determinations of R.E.E. contents in R.E.E. bearing minerals
BRGM	. preparation of R.E.E. ores to liberate minerals to be separated
	. laboratory flotation tests to correlate compositions of phosphoric esters to their performance for R.E.E. concentration
C.E.C.A. and BRGM	. synthesis and characterization of new phosphoric ester collectors

. analysis of flotation data in order to select the more selective P.E. and optimize their composition

3. EXPERIMENTAL WORK:

3.1. Materials:

Three samples taken from deposits under the exploration-evaluation phase, containing different major R.E.E. bearing minerals have been selected:

- ore 1: containing R.E.E. and Y rich apatite, from NORTH AMERICA

- ore 2: containing low Th monazite, from AFRICA

- ore 3: containing bastnaesite, from TURKEY.

Ores 1 and 2 have been collected and characterized by the BRGM, ore 3 by the Technical University of Munich (T.U.M.).

3.2. Methods:

3.2.1. Characterization of materials:

3.2.1.1. Microscopy - Ore mineralogy

- Optical microscopy. Observations by both reflected and transmitted light on polished sections and polished thin sections

- Scanning electron microscopy, for qualitative or semi-quantitative chemical determinations of minerals. An EDAX analysis system coupled with the S.E.M. was used by the T.U.M.

- Analysis by the electron microprobe. Quantitative chemical determinations of minerals were made by BRGM laboratories, using a CAMECA microprobe and data processing specially designed for R.E.E. analysis. P, Ca, Si, Fe, Ce, Pr, Nd, La, Sm, Gd, Y (or Dy, Yb and Er substituted for Ce, Pr and La) were determined on R.E.E. bearing minerals ; S, Ba, Sr, Cu, Ti, Mn, Mg, La, Fe, Ce, Si, P, on gangue minerals.

- X ray diffraction analysis

3.2.1.2. Chemical analysis or ores and products from the treatment

A number of techniques were used including:

- Induced coupled plasma associated to mass spectrometry (I.C.P./M.S.) for determinations of R.E.E. and other trace elements (T.U.M. and B.R.G.M.) X Ray Fluorescence for R.E.E. (T.U.M.)

- XRF on glass beads for determinations of major elements (BRGM and T.U.M.)

- Wet chemical analysis (B.R.G.M. and T.U.M.)

- FADCP, DCP, DCPA, FAA and GFAA methods (T.U.M.)

- Part of the flotation products were analyzed for Fe, Sr, Ba, La, Ce by a portable XRF analyzer (SYRANO) using a radio-active source.

3.2.1.3. Size analysis and fractionations

Laser light diffractometry and sieving.

3.2.2. Characterization of phosphoric esters (C.E.C.A.):

A number of analytical and separation techniques were used to determine mono and di orthophosphoric esters, pyrophosphoric esters, free orthophosphoric acid, free pyrophosphoric acid, residual non phosphated substances, non ionic products, total P_2O_5:

- separations by ion exchange resins and by organic solvents ($CHCl_3$ + Bu OH/H_2O),

- potentiometry, liquid and gas phases chromatography, colorimetry, high pressure liquid chromatography.

Main chemical procedures are referenced as CFD 110 - A - 157, and CFD 110 - A - 163 methods.

3.2.3. Synthesis of phosphoric esters:

Synthesis of phosphoric ester collectors involves phosphatation of alcohol, ether or phenol, including o to n oxialkylene groups, with $POCl_3$, PCl_5, PCl_3, P_2O_5, H_3PO_4 or condensed phosphoric acids. Phosphoric ester promoters are complex mixtures of mono and diesters:

where: - R is an hydrophobic radical: alkyl, aryl or alkyl - aryl

- (K)n are oxialkylene groups (alkyloxi)

associated with residual acids or non ionic products which affect frothing properties and collecting power: residue of phosphatation agents (pyrophosphoric and phosphoric acids, as examples), residues of alcohol or alkylphenol, esters of pyro or meta phosphoric acids ...

Frothing and collection properties, hydrophobicity of the collector coating, as well as selectivity of adsorption, are affected by numerous parameters, such as the nature of the R chain: linear chain, chains including aromatic rings, branched chains, distribution of molecular weights (or lengths) of initial alcohols subjected to phosphatation

(fatty and synthetic alcohols), the nature and number of the oxialkylene groups, the nature and proportions of the residual products, the relative proportions of mono and diesters. Most of these parameters strongly depend on the operating conditions followed in the phosphatation of the initial raw material (alcohol, alkyl-phenol ...) as well as conditions used for purifying the reaction products.

Samples of phosphoric esters submitted for flotation tests were produced by C.E.C.A. at laboratory scale, using proprietary procedures which led to control the basic parameters such as the ratio monoester/diester, the proportions of residual non phosphated substances and free acids.

Ten phosphoric ester collectors were tested and evaluated:

B 110 D 4 - 92, B 110 69032, B 110 D 4 - 70 E, (PE 88094), P 301, P 312, T 4 - 55 E, D 2 - 45 E, D 7 - 61 E, IM 7 - 58 (PE 89124), EA 14 - 89 (PE 89122).

Main characteristics of the collectors are given below:

	MELIORAN D2-45E	MELIORAN D7-61E	PE 89124	P312	B110
R	L.A.	L.A.	B.S.A.	L.A.	L.A.
X	D	D	I.M.	D	D
K	E.O.	E.O.	E.O.	E.O.	E.O.
NbC	12	12	14	12	12
n	2	7	7	9	4
Mono Ester Orthophosphoric %	34.6	51.2	51.0	43.6	71.9
Di Ester Orthophosphoric %	41.3	32.3	34.3	43.2	6.5
Non Phosphated Products %	4.5	7.7	15.7	9.4	23.2
Free H_3PO_4 %	1.1	1.5	0.8	0.7	3.5
Free $H_4P_2O_7$ %	1.2	0.8	0.2	0.2	0
Mono Ester Pyrophosphoric %	11.7	14.1	2.7	3.0	0
Di Ester Pyrosphoric symmetrical %	16.3	8.9	4.5	4.3	0
Refining procedure	Yes	Yes	No	No	No
Coded composition	D2-45E	D7-61E	IM7-58	D9 PS2 or D9-50	D4-92

	B110 88094	MELIORAN T4-55E	P301	B110 69032	PE89122
R	L.A.	B.S.A.	B.S.A.	L.A.	E.A.
X	D	T	T	D	E.A.
K	E.O.	E.O.	E.O.	E.O.	E.O.
NbC	12	13	13	12	21
n	4	4	6	4	14
Mono Ester Orthophosphoric %	63.0	46.3	46.9	54.5	44.5
Di Ester Orthophosphoric %	24.9	31.0	32.2	25.9	6.3
Non Phosphated Products %	4.5	4.8	15.6	20.3	33.6
Free H_3PO_4 %	2.3	1.2	0.7	1.7	1.1
Free $H_4P_2O_7$ %	0.5	1.0	0.8	0.1	0.9
Mono Ester Pyrophosphoric %	4.9	7.4	5.9	1.5	8.7
Di Ester Pyrophosphoric symmetrical %	3.7	13.5	2.6	2.9	0.5
Refining procedure	Yes	Yes	No	No	No
Coded composition	D4-70E	T4-55E	T6-PS or T6-59	D4-66	EA14-89

R: describes the nature of the hydrophobic radical where:

L.A.: Linear fatty Alcohol
B.S.A.: Branched Chain, synthetic Alcohol
E.A.: Ether Alcohol

n: statistical number of Ethylene Oxide groups (E.O.)
NbC: Number of Carbons in the hydrophobic Chain
MEO: content in Mono Ester Orthophosphoric
DEO: content in Di Ester Orthophosphoric
NPP: content in Non Phosphated Products
FOPA: content in Free Orthosphoric Acid
FFPA: content in Free Pyrophosphoric Acid
MEP: content in Mono Ester Pyrophosphoric
DEP: content in Di Ester Pyrophosphoric (symmetrical).

Variables describing the main characteristics of P.E. promoters have been used in the analysis of flotation data.

3.2.4. Preparation of ore samples for flotation tests:

Batch preparation was accomplished at laboratory scale using a combination of:

stage - crushing, attrition - scrubbing, wet sizing and desliming, stage rod milling down to about 160 μm (ores n° 1 and 2) and 125 μm (ore n° 3).

Rod milled materials were deslimed at 10 μm prior to be used as feeds to flotation.

Gravity preconcentrates - 80 + 10 μm or - 40 + 10 μm separated by combination of centrifugal separation and tabling from the R.E.E. ore sample 2 were also subjected to flotation.

3.2.5. Laboratory flotation tests:

Tests were made using conventional laboratory mechanical flotation cells: AGITAIR or 1.5 and 5 l capacity, MINEMET of 0.8 and 2.5 l capacity.

4. RESULTS:

4.1. Characterization of R.E.E. ore samples

4.1.1. Ore 1: Most of the R.E.E. mineralization occurs as apatite with small contents in heavy R.E.O., assaying 9.04% R.E.O., and 1.74% Y_2O_3. Other minor R.E.O. bearing minerals were found to be :

	total REO content %	Y_2O_3 content %
- a Cerium-britholite	50.39	4.55
- a Yttrium-britholite	11.13	27.12
- allanite	22.93	0
- a Cerium-apatite	41.71	0.49
- other R.E.O. bearing minerals (batnaesite, parisite)	61.74	1.18
- a secondary apatite with a low R.E.O. content	n.d.	n.d.

Gangue minerals consist of clinopyroxene, magnetite, biotite, albite, amphibola, ilmenite, zircon, iron carbonate, pyrite and clay minerals: kaolinite, smectite, chlorite, sepiolite and talc.

4.1.2. Ore 2: R.E.O. bearing minerals occur as:

	total REO content %
- monazite (major R.E.O. bearing mineral)	69 to 69.5
- apatite	4.51
- trace amounts of bastnaesite and parisite	n.d.

Gangue minerals or potentially valuable minerals occur as:

- carbonates:
  - . Mn and Fe containing dolomite
  - . stontianite
  - . siderite containing Mn, Ca, Mg
  - . calcite
- sulfate : baryte
- oxides : - manganese oxides (psilomelane) containing varying amounts of Fe, Ba, Ce, Sr
  - magnetite;
- quartz

4.1.3. Ore 3: is a mixture of two types of ores (composite ore TU):

TU1: fluorite-baryte-REE minerals
TU2: high carbonate ore

- The first type consists of:

fluorite: 15-60%, baryte: 10-50%, bastnaesite: up to 20%, Fe-Mn oxides: up to 10%, white mica and phlogopite: up to 15%, calcite and dolomite: up to 20%, chlorite, quartz, chalcedony and other R.E.E.-minerals: up to 3%.

Most of the bastnaesite occurs in extremely fine grained felty aggregates which will generate slimes on grinding and will cause problems for its concentration and recovery by flotation.

- The second type consists of:

calcite and dolomite: 40-50%, fluorite: 15-25%, baryte: 15-20%, bastnaesite: up to 15%, white micas and phlogopite, Fe-Mn oxides and other R.E.E.-minerals (R.E.O. containing phosphates): up to 3%.

Similarly to the first type, most of the basnaesite grains exhibit a felty texture characterizing associations of fine sized crystals as aggregates.

The low carbonate ore TU1 is predominant in the composite sample TU which contains fluorite: about 58.7%, barite: about 19.3%, bastnaesite: about 11.8% and gangue minerals (silicates, carbonates, iron oxides, silica): about 10.2%.

Chemical compositions of R.E.E. ore samples are shown in table 1.

		R.E.O. ORE SAMPLE		R.E.O. ORE SAMPLE	
		n°1	n°2	n°3	
P_2O_5	%	3.13	3.56	SiO_2 %	1.85
CaO	"	7.32	16.77	P_2O_5 "	0.55
SiO_2	"	56.51	4.61	Fe_2O_3 "	2.53
Fe_2O_3	"	14.01	7.31	Al_2O_3 "	0.78
Al_2O_3	"	7.88	0.96	SO_4 "	8.10
MgO	"	1.67	12.94	Ca "	27.50
TiO_2	"		0.06	F "	29.63
LOI	"	0.93	30.71	Ba "	11.36
Na_2O	"		< 0.20		
K_2O	"		< 0.05		
MnO	"		1.89		
CO_2	"		30.48		
H_2O^-	"		0.23		
H_2O^+	"		1.29		
SrO	"		9.98		
BaO	"		1.28		
SO_3	"		0.95		
La	ppm	1 532	14 045	La ppm	32 500
Ce	"	2 988	23 044	Ce "	34 500
Nd	"	1 142	6 466	Nd "	4 822
Sm	"	217	715	Sm "	316
Eu	"	20	88	Eu "	74
Gd	"	207	261	Gd "	143
Dy	"	196	103	Dy "	66
Er	"	100	30	Er "	39
Yb	"	86	11	Yb "	28
Y	"	971	61	Y "	398
				Ho "	13
				Lu "	4
				Pr "	2 194
				Tb "	18
				Tm "	5
Total REE+Y ppm		7 459	44 824	Total REE+Y ppm	75 120
(Eu+Y)/(REE+Y)		0.1329	0.0033	(Eu+Y)(REE+Y)	0.0063

TABLE 1 : Chemical compositions of R.E.E. ore samples

4.2. <u>Ore preparation:</u>

- 160 + 10 μm fractions, separated from the ground ores n° 1 and 2, had the following compositions:

	- 160 + 10 µm fractions	
	Ore n° 1	Ore n° 2
Weight recovery %	95.36	88.81
P_2O_5 recovery %	97.17	90.19
Total R.E.O. recovery %	95.94	93.10
Y recovery %	95.30	90.34
P_2O_5 assay %	3.19	3.62
Ca O assay %	7.41	16.95
$Si O_2$ assay %	57.30	4.51
Fe_2O_3 assay %	13.22	6.40
Al_2O_3 assay %	7.91	0.76
Mg O assay %	1.49	13.29
Sr O assay %		10.39
Ba O assay %		1.27
CO_2 assay %		31.29
TOTAL R.E.O. assay ppm	6528	46929
Y assay ppm	970	62

Preparation of ore 3 gave rise to production of a high proportion of - 10 µm slimes, highly enriched in R.E.E.:

	Total - 125 + 10 µm	Total - 10 µm slimes
Weight recovery %	74.57	25.43
F recovery %	78.09	21.91
Ba recovery %	93.79	6.21
Total R.E.E. recovery %	36.85	63.15
Y recovery %	57.36	42.64
F assay %	31.03	25.53
Ba assay %	14.34	2.79
Total R.E.E. assay ppm	36899	185442
Y assay ppm	306	668

Concentration of R.E.E. was even higher in the primary - 10 µm (1) slimes generated by attrition - scrubbing, total R.E.E. content was 241369 ppm, accounting for 52.85 % of the total R.E.E. contained in the initial material, recoveries of light R.E.E. (from La to Sm) in - 10 µm (1) slimes was higher: 50.3 to 53.5 %, than those of heavy R.E.E. ranging from 30 to 47.6 %. Since most of the R.E.E. bearing minerals report to the - 10 µm attrition and grinding slimes, further concentration by flotation or other physical separation process appeared to be extremely difficult.

4.3. Flotation tests - Evaluation of phosphoric ester collectors:

4.3.1. Ore 1:

Prior to flotation, most of the magnetite was removed from the - 160 + 10 µm fraction by a low intensity magnetic drum separator, preliminary tests conducted on the - 160 + 10 µm NM fraction have shown that:

- selectivity of separation of the Y containing apatite by flotation, using certain types of phosphoric ester promoters, was superior to that obtained through conventional apatite flotation using direct anionic flotation of apatite with an aqueous emulsion of tall oil, diesel oil and an emulsifier reagent, sodium silicate as depressant and dispersant of the siliceous gangue and corn starch as a depressant of iron bearing minerals.

- addition of specific depressants for both silicates and iron containing minerals was necessary to produce low R_2O_3 and MgO apatite concentrates, using phosphoric esters as collectors for apatite at pulp pH of about 9.5. The best depressant was a solution of sodium silicate and citrate (with 5/1 concentration ratio) in combination with dextrin or corn starch.

Preliminary testwork has led to the definition of a standard flotation flowsheet for separating R.E.E. and Y containing apatite from silicates and iron bearing minerals.

Basic flowsheet shown in figure 1 was applied to the - 0.16 + 0.01 mm ore, using 8 types of phosphoric ester collectors, collector dosage was 225 g/ton of flotation feed.

Results of tests are shown, in a synthetic manner, as grade-recovery curves for P_2O_5 in diagrams of the figure n° 2.

Positions of grade-recovery curves reflect performances of phosphoric ester collectors in terms of selectivity and apatite recovery, it can be seen that the B 110 - 88094 provides the best response of the ore to flotation.

Performances of phosphoric esters are in the following decreasing order: 88094 - 69032 - T455E - B110 - D245E - D761E - P301 - P312.

The collecting power increases with n, while selectivity appears to be detrimentally affected at high n values.

Optimum performances are obtained for n = 4, 70 % monoester and low amount (about 4.5 %) of residual non phosphated products, occurrence of free phosphoric acid appears to impair selectivity.

Chemical compositions of apatite concentrates separated using the phosphoric ester reagent 88094 as a promoter are given below:

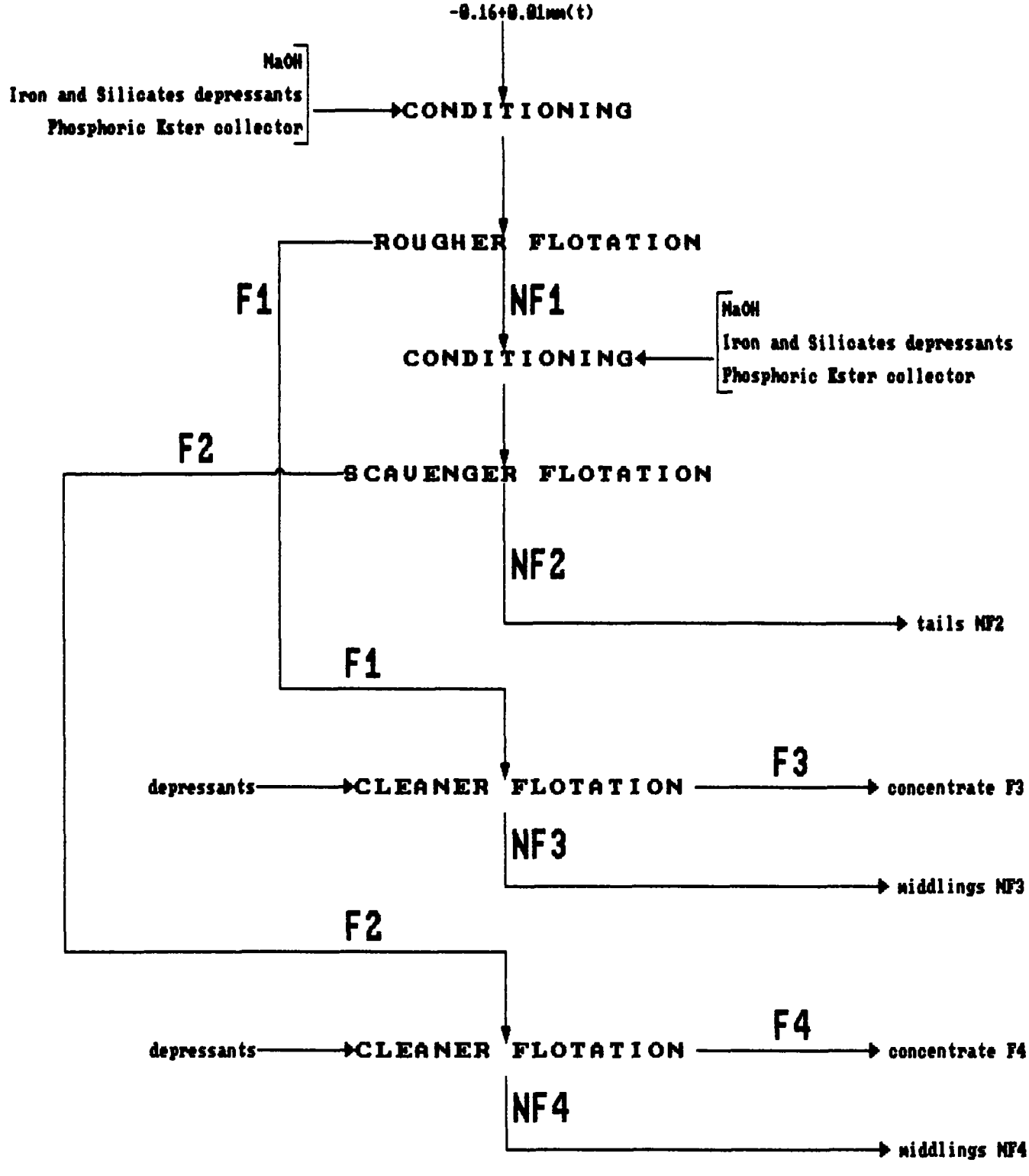
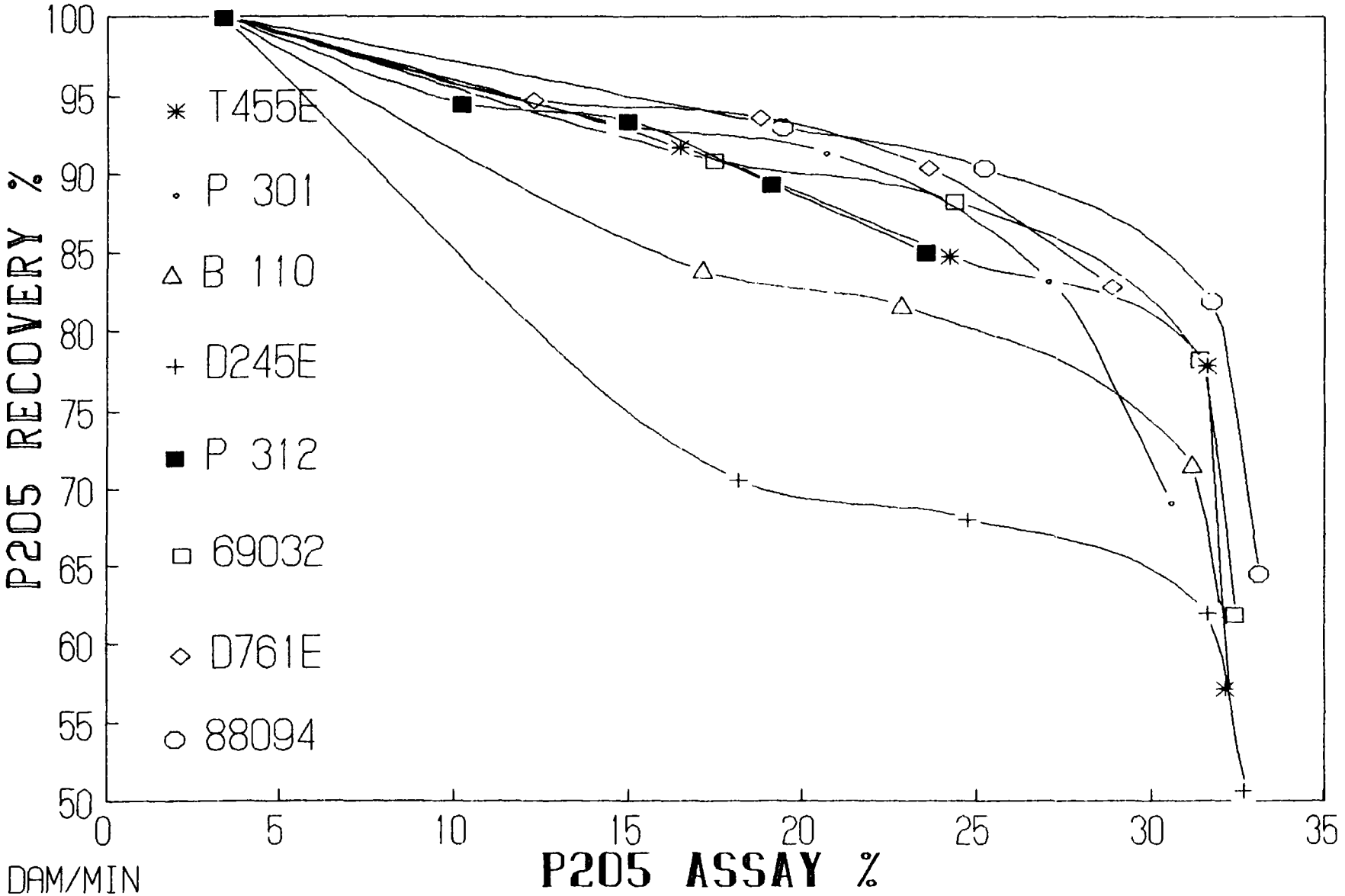


FIGURE Nº 1 : BASIC FLOWSHEET FOR FLOTATION OF THE REO SAMPLE Nº1.

FIGURE N 2.

R.E.O. ORE SAMPLE 1.FLOTATION TESTS
PERFORMANCE OF PHOSPHORIC ESTERS



	Apatite concentrates		Feed to flotation
	F 3 + F 4	F 3	
P_2O_5 %	31.82	33.10	3.42
CaO %	45.72	46.35	7.74
SiO_2 %	6.92	5.75	62.56
Fe_2O_3 %	1.20	0.95	5.92
Al_2O_3 %	0.55	0.42	8.69
MgO %	0.53	0.50	1.64
L.O.I. %	3.10	2.31	1.09
La ppm	7650	7610	1679
Ce ppm	14800	14220	3009
Nd ppm	7030	6830	1124
Sm ppm	1525	1485	217.5
Eu ppm	149.9	155	20.2
Gd ppm	1640	1650	209.2
Dy ppm	1420	1480	185.1
Er ppm	853	870	102.5
Yb ppm	783	792	89.7
Y ppm	8015	8080	1006.2
Total R.E.E. ppm	43865.9	43172	7642.4
CaO/P_2O_5	1.437	1.400	2.263
R_2O_3/P_2O_5	0.055	0.041	4.272
T.C.P. %	69.53	72.32	7.47
P_2O_5 recovery %	81.93	64.54	100

Separation of apatite with phosphoric ester leads to marketable grade phosphate concentrates with low contents in contaminants such as R_2O_3 and MgO. Conversion of P_2O_5 values to marketable wet process phosphoric acid appears to be possible besides recovery of rare earth elements and yttrium.

The enrichment ratio for P_2O_5 ranges between 9.3 and 9.7, the enrichment ratio is 5.74 - 5.65 for R.E.E. + Y, 7.96 - 8.93 for Y.

Phosphate concentrates assaying 31.8 - 33.1 % P_2O_5, 4.39 - 4.32 % R.E.E. + Y, 0.8 - 0.81 % Y can be produced at satisfactory recoveries on flotation feed basis: 81.93 - 64.54 % for P_2O_5, 50.4 - 37.5 % for R.E.E. + Y and 69.9 - 53.2 % for Y.

Differences between R.E.E. - Y and P recoveries account for R.E.E. and Y losses in flotation tails as R.E.E. and Y containing silicates: britholite and allanite which were identified by the mineralogical study. R.E.E. and Y bearing silicates are not amenable to flotation using phosphoric esters as collectors.

4.3.2. Ore 2

Extensive laboratory teswork was effected on:

- the - 0.05 + 0.01 mm size fraction separated from the ore ground down to 0.16 mm

- gravity preconcentrates separated by shaking tables and the MOZLEY MGS separator from the - 0.08 + 0.01 mm and - 0.04 + 0.01 mm fractions of the ore ground down to 0.16 mm

Flotation involves separation of monazite from: Mn - Fe containing dolomite, strontianite, calcite and baryte.

Results have shown that:

. separation of baryte as a non float product is possible, using H_2SO_4 as a baryte depressant and pH regulator, phosphoric ester P301 as a collector for monazite and strontianite

. strontianite could be floated with pretroleum sulfonate as a collector

. conditioning with CO_2 can depress Fe - Mn dolomite, siderite, calcite and strontianite when monazite is floated with phosphoric ester in a weak acidic pulp. Separation of monazite from strontianite requires a high number of cleaning stages.

- Performance of flotation on the - 0.05 + 0.01 mm fraction of the ore with a monazite content of 13 %, was poor in terms of R.E.E. recovery: the best concentrate assays 11.61 % La_2O_3 and 19.77 % CeO_2 at 32.3 and 31.8 % La and Ce recoveries. This corresponds to a 40.9 % content in total R.E.O. or a monazite content of 58.7 %. Further enrichment of this preconcentrate appears to be possible using multiple cleaning stages in an attempt to remove residual iron bearing dolomite.

- Response to flotation of the gravity preconcentrate with a monazite content of 68.5 %, is promising since the flotation concentrate assays 19.3 % La_2O_3 and 32.38 % CeO_2 at 68.8 and 68 % La and Ce recoveries, for a phosphoric ester consumption of 70 g/t. The flotation concentrate assays 67.4 % total R.E.O., corresponding to a monazite content of 96.7 %.

Retreatment of middling products could probably lead to improved monazite recoveries in concentrates.

It should be pointed out that magnetic separation applied to this gravity preconcentrate does not lead to a satisfactory monazite concentrate due to the fact that Fe - Mn dolomite, siderite, and monazite have similar magnetic susceptibilities.

Phosphoric ester collectors were evaluated for their capability to float monazite from the - 80 + 10 μm gravity preconcentrate, using the basic flowsheet shown in figure n° 3.

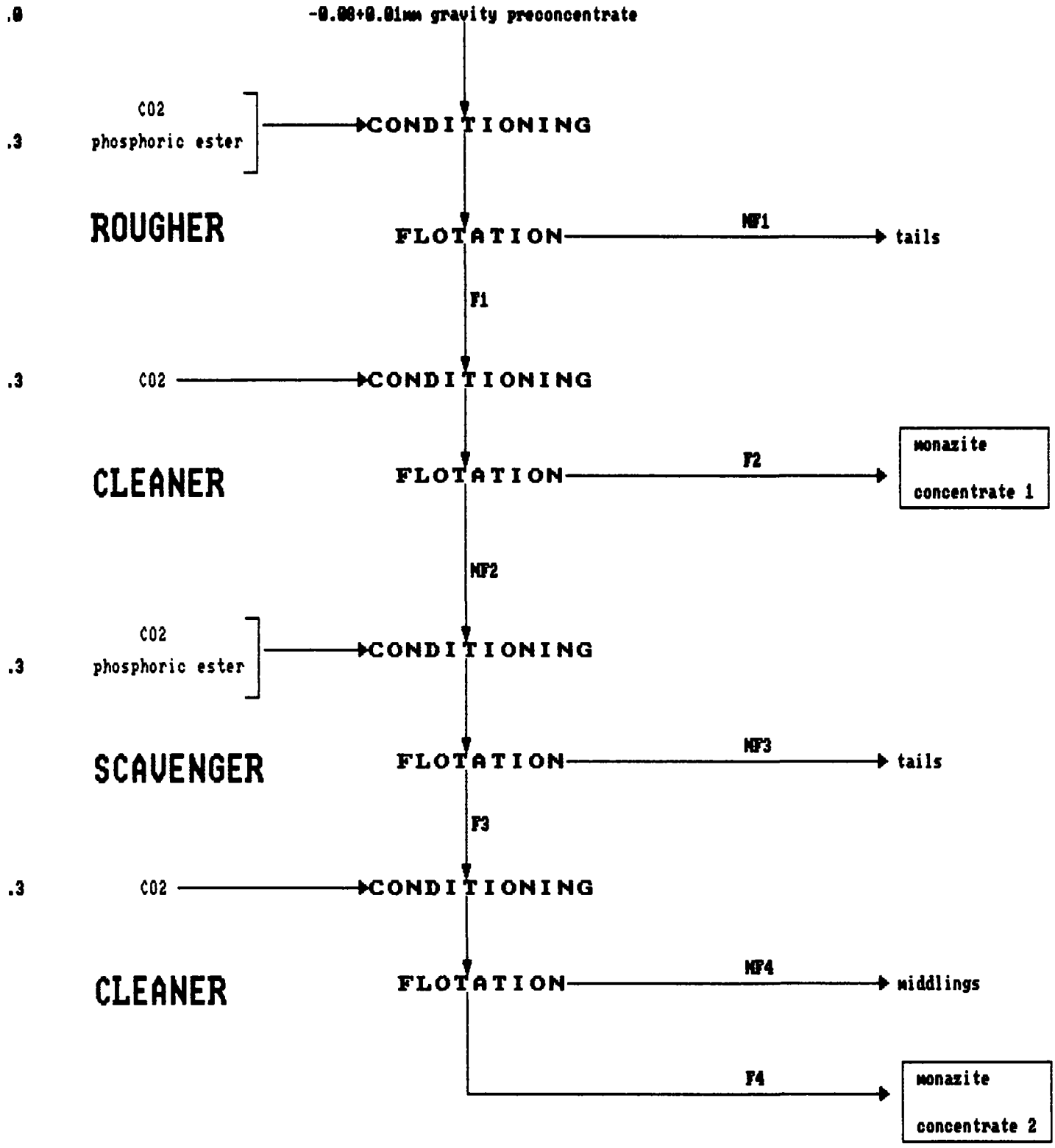


figure n° 3 :basic flowsheet for flotation of -0.08+0.01mm gravity preconcentrates separated from the R.E.O. ore sample n°2 ground down to -0.16mm. (evaluation of phosphoric ester promoters).

Flotation performance is shown as grade - recovery curves in figure n° 4, phosphoric ester dosages were 70 g/t.

Data of figure 4 indicate that the best performances were obtained using EA 14-89 and D9PS2 reagents at low dosages of 70 g/t, concentrates assaying 94.2-95.3 % monazite at 79.3-80 % recovery were separated in batch operation, excluding retreatment of middling products.

	Concentrate F2	Composite concentrate F2 + F4	Feed to flotation
Fe_2O_3 content %	< 1	1	4.39
SrO content %	1.33	1.52	9.85
BaO content %	0.75	0.72	5.25
total REO content %	68.88	65.70	42.77
total REO recovery %	64.04	79.94	100.00
monazite content %	- 97	95.22	61.98

flotation in a continuous circuit including recirculation of middlings or optionally regrinding to improve liberation of monazite, would probably increase REO recovery in the composite concentrate.

High number of oxialkylene group appears to promote both high selectivity and collecting power. Selectivity for monazite increases with increasing mono ester content and decreasing contents of residual non phosphated products and free H_3PO_4.

4.3.3. Ore 3

Flotation of the - 125 + 10 µm fraction with phosphoric esters led to moderate concentration of fluorite and R.E.E. minerals in float fractions, however, selectivity for R.E.E. was poor, probably due to the fact that the R.E.E. minerals are insufficiently liberated as shown by results of heavy liquid separations.

Results of flotation tests on - 10 µm primary slimes also showed no significant concentration of R.E.E., this could be related to the extremely fine size distribution of the flotation feed material : 89.1% by weight minus 6 µm, 55.5% minus 3 µm, 33.5% minus 2 µm.

5. ANALYSIS OF FLOTATION DATA

Stepwise multiple linear regression techniques applied to analysis of flotation test data have shown that:

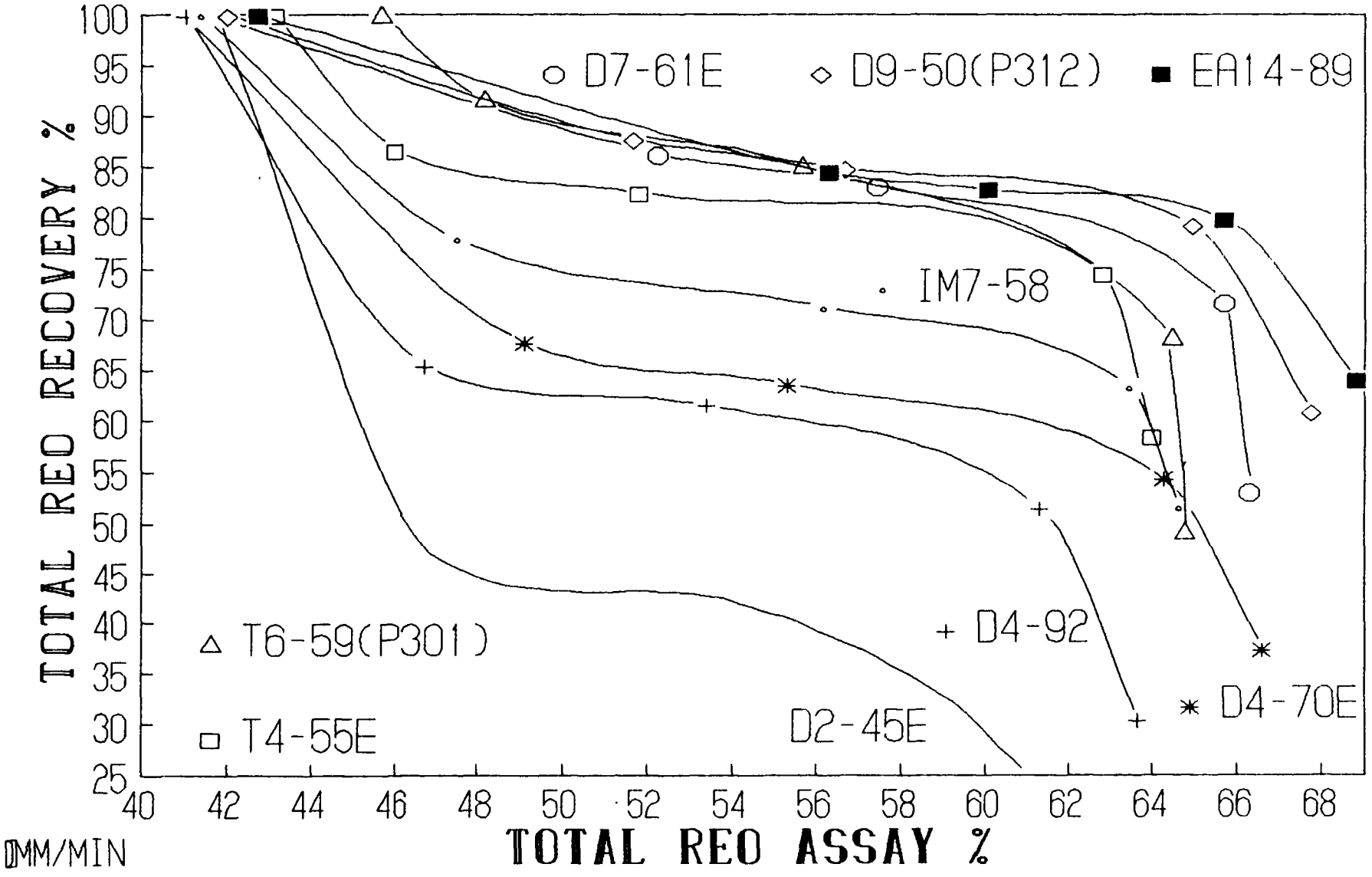
- the collecting power of phosphoric ester collectors strongly increases with the number n of ethylene oxide groups in the molecule.

Apatite or monazite recoveries in flotation concentrates separated from the REO ore samples n° 1 or n° 2, increase with n. However, P_2O_5

FIGURE N 4

REO ORE SAMPLE 2.FLOTATION TESTS

PERFORMANCE OF PHOSPHORIC ESTERS



DMM/MIN

content in concentrates produced from the ore n° 1, concentration efficiency and selectivity for P_2O_5, were found to be detrimentally affected at high n values, whereas n was found to exert a positive influence on both REO contents in concentrates produced from the ore n° 2 and selectivity for REO.

Differences in the effects of n on selectivity for apatite and for monazite might be attributed to differences in the mechanism of phosphoric ester adsorption on to the surface of apatite and monazite the former involves predominant Ca - phosphoric ester interactions at high pulp pH (9.5) while the latter involves REE - phosphoric ester interactions at lower pulp pH (5.3).

- REO recovery in flotation concentrates separated from the ore n° 2 increases as the content in residual non phosphared matter (NPP) of the phosphoric ester collector decreases. This could be related to increasing concentrations of phosphoric esters in the commercial products as the non phosphated matter is reduced

- selectivity for P_2O_5 or REO, as well as P_2O_5 or REO contents in flotation concentrates separated from the ore n° 1 or the ore n° 2, increase with concentration of mono orthophosphoric ester (MEO) in commercial collectors

- characteristics of the phosphoric ester collectors provide variables to satisfactorily explain variations relavant to selectivity. Recovery of valuable minerals (apatite or monazite) and concentration efficiency are not so well explained by characteristics of the collectors. Occurrence of composite particles and variations in operating conditions of flotation might also account for part of variations observed in recovery of valuable minerals

- concentration efficiency for P_2O_5 strongly depends on the selectivity term while concentration efficiency for REO is governed by the recovery term.

6. CONCLUSIONS

Laboratory investigations conducted on three R.E.E. ore samples differing by the nature of the main R.E.E. bearing minerals clearly show that certains types of phosphoric esters exhibit high affinity for Y and R.E.E. containing apatite as well as for monazite, and could be used as collectors for concentrating these minerals by flotation provided that the compositions of the gangue allows selectivity to be achieved. Results of flotation tests on the monazite ore indicate that monazite could be efficiently separated from strontianite, dolomite, baryte, siderite, iron and manganese oxides, whereas concentration performance sharply drops in the presence of high proportions of Mn-Fe containing dolomite which responds quite well to flotation whith phosphoric esters. Removing most of this contaminant by tabling leads to a gravity preconcentrate which is refined by flotation to produce concentrates assaying 94 to 97 % monazite. Results of flotation tests on the bastnaesite ore suggest that phosphoric esters have low affinity for bastnaesite.

Separation of Y and R.E.E. containing apatite from silicates and iron minerals by flotation with a phosphoric ester collector has led to marketable grade apatite concentrates with acceptable contents of residual MgO and R_2O_3.

This makes possible conversion of P_2O_5 values to marketable wet process phosphoric acid besides recovery of rare earth elements and yttrium.

The most efficient phosphoric ester collectors for R.E.E. and Y containing apatite should have a high content in mono orthophosphoric ester, a low concentration in residual H_3PO_4 and an appropriate number n of ethylene oxide groups giving satisfactory apatite recovery with moderate detrimental effect on the apatite content of the concentrates.

Within the experimental range explored, the most effective phosphoric ester collectors for monazite are characterized by high n values, low concentrations in non phosphated products and H_3PO_4 and high concentrations in mono orthophosphoric ester.

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COMMISSION
OF THE EUROPEAN COMMUNITIES DG XII
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APPLICATION OF PHOSPHORIC ESTERS TO FLOTATION OF FINELY DIVIDED OXIDIZED ORES

Final Report

C.E.C. Research Contract MA1M-0059-C

G. Baudet (BRGM)
G. Morteani (TUM)
M.-P. Strub (CECA)

Appendix

January 1992
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BRGM
DIRECTION DES ACTIVITÉS MINIÈRES
Département Minéralurgie
B.P. 6009 - 45060 ORLÉANS CEDEX 2 - France - Tél. : (33) 38.64.34.34

A P P E N D I X 1

Mineralogic study of the REO ore sample 1 by O. LEGENDRE (BRGM)

ETUDE MINERALOGIQUE D'UN MINERAI DE PHOSPHATES DE TERRES-RARES

(échantillon n°1)

Par O. LEGENDRE DT/ANA

RESUME :

L'étude de l'échantillon de minerai de terres rares a montré que :

- l'essentiel de la minéralisation en oxydes de terres rares est exprimée sous forme d'apatite. Cette apatite ne contient pratiquement pas d'oxydes de terres rares lourdes.

- Les autres minéraux porteurs de terres rares sont la cérium-britholite, l'yttrium-britholite, l'allanite, une cérium-apatite ainsi qu'un mélange d'oxydes de terres rares lourdes.

Il semble, au vu des clichés réalisés au microscope électronique, que ces minéraux résultent de la destabilisation de l'apatite primaire. Cette déstabilisation entraîne d'une part la formation de ces minéraux, et d'autre part la formation d'une apatite secondaire pratiquement dépourvue d'oxydes de terres rares.

L'abondance de ces minéraux ainsi que les teneurs en oxydes de terres rares sont rappelées dans le tableau ci-dessous :

	Y_2O_3	La_2O_3	Ce_2O_3	Pr_2O_3	Nd_2O_3	Sm_2O_3	Gd_2O_3	Dy_2O_3	Er_2O_3	Yb_2O_3
Apatite (A)	1.74	1.76	3.74	0.63	2.02	0.33	0.29	0.15	0.02	0.10
Ce Apatite (TR)	0.49	14.19	18.08	3.01	5.54	0.68	0.21	ND	ND	ND
Ce Britholite (F)	4.55	9.58	21.61	3.67	11.79	2.02	1.72	ND	ND	ND
Y Britholite (R)	27.12	ND	ND	ND	0.73	0.79	1.85	2.69	2.47	2.60
Allanite (R)	0.00	4.46	10.97	1.92	4.89	0.59	0.10	ND	ND	ND
Oxydes (R)	1.18	14.17	26.56	4.64	12.90	1.98	1.19	ND	ND	ND

Teneurs en oxydes de terres rares des différentes phases minérales rencontrées.
(nd = non dosé). L'estimation de leur abondance est indiquée entre parenthèse (A : abondante ; F : faible ; R : rares ; Tr : très rares.) Ces estimations sont des estimations visuelles).

D'un point de vue pratique, les observations montrent que :

- les minéraux de terres rares* sont à de rares exceptions près, toujours associés à l'apatite.

- la taille moyenne de ces minéraux se situe aux alentours de 50 µm.

- la quasi-totalité de l'apatite semble libérée à une granulométrie de 500µm.

Les phases stériles qui accompagnent le minerai sont :

- Clinopyroxène
- Biotite
- Albite
- Amphibole
- Ilménite
- Zircon
- Carbonate de fer
- Pyrite

Parmi celle-ci, les phases les plus fréquemment rencontrées étant le clinopyroxène, la biotite, le plagioclase et l'amphibole.

* Dans le texte "minéraux de terres rares" fait toujours référence aux minéraux porteurs de terres rares, excepté l'apatite primaire.

INTRODUCTION :

Cette étude, réalisée sur un échantillon broyé d'une granulométrie de - 1mm à 0.02 mm, a pour but de déterminer la nature et la composition des phases porteuses de terres rares, ainsi que d'effectuer une estimation de la minéralogie de la partie stérile. Compte tenu de la nature de l'échantillon, (granulométrie variable, et très fine) il n'a pas été possible de procéder à une estimation quantitative de la proportion des différentes phases en présence.

TECHNIQUE ANALYTIQUE :

L'échantillon a été préparé sous forme de section polie (la finesse et l'hétérogénéité des grains qui le compose étaient trop importantes pour entreprendre la confection d'une lame mince, qui aurait permis une meilleure approche de la minéralogie de l'échantillon).

L'étude a été menée en trois étapes successives :

- un repérage au microscope métallographique des phases favorables à la présence d'éléments de terres rares.

- une observation au microscope électronique à balayage, ceci pour une sélection définitive des minéraux contenant des oxydes de terres rares en vue de l'analyse à la microsonde électronique. De même, cette observation permet une approche des associations paragénétiques des différentes phases minérales.

  La nature des minéraux composant la gangue a aussi été déterminée au microscope électronique à balayage.

- Une étude du chimisme des différents minéraux à la microsonde électronique. Compte tenu du comportement particulier des éléments de terres rares en émission de rayons X (Raies d'émissions très proches les unes des autres, pouvant occasionner des phénomènes d'interférences, il a été nécessaire de procéder avant l'analyse proprement dite, à une détermination qualitative fine des éléments présents dans chacun des minéraux à analyser. A la suite de ces déterminations nous avons retenu les éléments suivants :

  P, Ca, Si, Fe, Ce, Pr, Nd, La, Sm, Gd et Y

Pour une série de minéraux nous avons remplacé Ce, Pr et La par Dy, Yb et Er.

RESULTATS

I - MINERALOGIE

Les minéraux à terres rares :

La paragénèse de ces minéraux est assez restreinte. En fait, six espèces principales porteuses de terres rares ont été identifiées :

Apatite : Dans la section étudiée, ce minéral est le porteur essentiel des oxydes de terres rares. Et c'est sans doute celui qui est à l'origine de la présence des autres minéraux de terres rares ; en effet, on observe deux types d'apatite :

- une apatite riche en oxyde de terres rares (elle apparaît en clair sur les clichés MEB) ; Les teneurs vont, pour les terres rares les plus abondantes de 2,01 à 5,7 % de Ce_2O_3, 0,81 % à 3,28 % de Nd_2O_3, 0,80 % à 2,83 % de La_2O_3, 0,80 % à 2,72 % de Y_2O_3, Sm_2O_3, Gd_2O_3 et Pr_2O_3 sont présents en quantités généralement inférieures à 1 % pondéral (Tableau 1a). Les terres rares lourdes qui ont pu être analysées -i.e. Gd_2O_3, Dy_2O_3, Er_2O_3 et Yb_2O_3 ne sont jamais présentes en quantités supérieures à 1 % pondéral (tableau 1b).

- Une apatite pratiquement dépourvue d'oxydes de terres rares. Les photos réalisées au microscope électronique montrent que cette apatite semble résulter de la destabilisation partielle de l'apatite à oxydes de terres rares, probablement sous l'action de fluides hydrothermaux ; les oxydes de terres rares libérés par cette destabilisation se reconcentrent au sein même de ces apatites, sous forme de minéraux dont les oxydes de terres rares sont les composants majeurs.

Un minéral semblant appartenir au groupe des apatites a été rencontré en remplissage de fissure d'une apatite à terres rares, celui ci est très riche en Ce_2O_3 et en La_2O_3 (respectivement 18,08 % et 14,2 %, Tableau 1c). Sa présence dans le minerai reste toutefois anecdotique. (Par commodité, nous l'avons appelé "Cerium - apatite".)

Allanite : ce minéral est assez peu fréquent, il a été trouvé soit sous forme de minéral d'apparence précoce, bien individualisé (photo B), soit comme minéral secondaire, en inclusion dans un clinopyroxène associé à une apatite à terres rares (photo C, figure D). Les teneurs en oxydes de terres rares de ces deux types d'allanite sont cependant assez proches, les plus abondants étant Ce_2O_3, La_2O_3 et Nd_2O_3 (tableau 2).

Cérium-britholite : Il s'agit d'un silico-phosphate de terres rares, dont la formule théorique est : $(Ce, Ca)_5 (SiO_4, PO_4)_3 (OH,F)$. C'est le plus fréquent des minéraux de terres rares, apatite non comprise, que nous ayons rencontré dans cet échantillon. La cérium-britholite est toujours en inclusion dans les apatites (photos E et F, figure G ; photo H, figure I et photo J), en plages d'une dizaine de µm environ. La composition de ce minéral est assez variable. Cette variation est due essentiellement aux substitutions interélémentaires nombreuses -e.g. Ca-Ce-Y, Si-P... La variation des teneurs est pour les quatre principaux oxydes de terres rares : Ce_2O_3 15,36 % à 26,29 %, La_2O_3 7,79 % à 12,47 %, Nd_2O_3 7,65 à 16,03 % et Y_2O_3 2,62 % à 12,32 % (tableau 3). Nous avons inclus dans ce groupe un minéral non répertorié, qui n'est vraisemblablement pas une cérium-britholithe, mais dont la composition en est assez proche (tableau 3, point 5).

Yttrium-britholite : Pôle riche en yttrium de la britholite, dans ce cas la substitution Si-P joue en faveur du silicium. Ce minéral n'a été rencontré que dans un seul grain, le clinopyroxène associé à l'apatite, et dans lequel nous avons observé l'allanite en inclusion (photo C, figure d). Dans ce grain l'yttrium-britholite est en remplissage des clivages du pyroxène, elle semble plus tardive que l'allanite. La formule théorique de ce minéral est $Ca_2 Y_3 Si_3 O_{12} (OH)$, en fait ce minéral contient en plus de l'yttrium, des terres rares lourdes en quantité non négligeable i.e Dy_2O_3 et Yb_2O_3 en teneurs de l'ordre de 2 à 3 % pondéral. Les terres rares légères sont pratiquement absente (Tableau 4). La et Ce n'ont pas été détectés lors des déterminations qualitatives.

Oxydes de terres rares : Ces oxydes n'ont été trouvés que dans un seul grain, comme la plupart des autres minéraux de terres rares, ceux-ci sont associés à

l'apatite (photo A). Il s'agit d'un mélange d'oxydes de terres rares légères, principalement Ce, La et Nd (Tableau 5). Le calcium et le phosphore présents dans les analyses sont dus à l'apatite qui entoure ces grains.

Les minéraux stériles :

Les minéraux accompagnateurs des minéraux à oxydes de terres rares sont pour l'essentiel des silicates. Au microscope électronique, nous avons pu déterminer la présence des minéraux suivants :

- clinopyroxène
- biotite
- albite
- amphibole
- ilménite
- zircon
- carbonate de fer
- pyrite

Les minéraux les plus fréquents sont le clinopyroxène, la biotite, l'albite et l'amphibole. Les analyses à la microsonde électronique de ces quatre minéraux sont données dans le tableau 6. Les autres minéraux sont occasionnels et n'ont pas été analysés.

II - GRANULOMETRIE

Bien que nous n'ayons pas pu mesurer précisément la maille de libération des minéraux de terres rares, l'observation de l'échantillon montre que :

- Si les minéraux à terres rares autre que l'apatite sont généralement de taille très faible (de 1µm à 50 µm environ), ces minéraux sont pratiquement toujours associés intimement à l'apatite (l'exception est l'yttrium-britholite que nous avons trouvé en remplissage des clivages d'un clinopyroxène).

- Les fragment d'apatite que nous avons observés sont pratiquement tous libérés à une granulométrie de 500 µm.

ANNEXES

10 samples, 11 elements

	2	3	8	14	23	24	26	31	32	Moy
Fe2O3	0.05	0.09	0.22	0.14	0.29	0.11	-	0.10	0.08	0.12
SiO2	2.01	2.06	1.82	6.09	6.05	5.77	5.67	2.62	2.70	3.87
Ce2O3	1.99	1.90	1.77	6.06	5.89	5.44	5.35	2.56	2.75	3.74
Pr2O3	0.34	0.29	0.40	1.09	0.71	0.96	1.00	0.23	0.61	0.63
P2O5	39.59	39.21	39.68	34.16	34.44	34.35	34.33	38.99	38.89	37.07
CaO	49.34	48.99	49.05	41.34	41.06	42.44	43.04	47.38	47.15	45.53
Nd2O3	0.82	1.27	0.87	3.18	3.28	3.01	2.79	1.38	1.58	2.02
Y2O3	0.86	0.80	0.98	2.72	2.55	2.58	2.61	1.24	1.33	1.74
La2O3	0.90	0.86	0.80	2.82	2.83	2.57	2.57	1.24	1.27	1.76
Sm2O3	0.20	0.22	0.18	0.41	0.69	0.41	0.37	0.19	0.27	0.33
Gd2O3	0.35	0.06	0.19	0.46	0.47	0.43	0.35	0.33	-	0.29
Total	96.46	95.75	95.97	98.46	98.25	98.08	98.08	96.26	96.64	97.11
	-	-	-	-	-	-	-	-	-	-
#Fe+3	0.01	0.01	0.03	0.02	0.04	0.01	-	0.01	0.01	0.02
#Si+4	0.35	0.36	0.32	1.10	1.09	1.04	1.02	0.46	0.47	0.69
#Ce+3	0.13	0.12	0.11	0.40	0.39	0.36	0.35	0.16	0.18	0.24
#Pr+3	0.02	0.02	0.03	0.07	0.05	0.06	0.07	0.01	0.04	0.04
#P	5.82	5.81	5.85	5.21	5.25	5.24	5.23	5.78	5.76	5.55
#Ca+2	9.18	9.19	9.15	7.98	7.92	8.19	8.30	8.88	8.84	8.63
#Nd+3	0.05	0.08	0.05	0.20	0.21	0.19	0.18	0.09	0.10	0.13
#Y	0.08	0.07	0.09	0.26	0.24	0.25	0.25	0.12	0.12	0.17
#La+3	0.06	0.06	0.05	0.19	0.19	0.17	0.17	0.08	0.08	0.12
#Sm+3	0.01	0.01	0.01	0.03	0.04	0.03	0.02	0.01	0.02	0.02
#Gd+3	0.02	0.00	0.01	0.03	0.03	0.03	0.02	0.02	-	0.02
#TOTAL	15.73	15.74	15.71	15.49	15.44	15.56	15.61	15.62	15.62	15.61
#O-2	25.00	25.00	25.00	25.00	25.00	25.00	25.00	25.00	25.00	25.00

TABLEAU 1a :

Analyses à la microsonde électronique des apatites à terres rares. Chaque numéro correspond à une analyse, la localisation de ces analyses est rappelée dans la légende des photos et des figures.

3 samples, 11 elements

	B3	B11	Moy.
Fe2O3	0.13	0.22	0.17
SiO2	1.86	1.81	1.83
CaO	51.06	50.83	50.95
Nd2O3	1.05	0.94	1.00
P2O5	40.59	40.58	40.58
Sm2O3	0.13	-	0.06
Y2O3	0.87	1.21	1.04
Gd2O3	0.33	0.50	0.42
Dy2O3	0.15	0.13	0.14
Yb2O3	0.08	0.11	0.10
Er2O3	0.04	-	0.02
Total	96.30	96.32	96.31
	-	-	-
#Fe+3	0.02	0.03	0.02
#Si+4	0.32	0.31	0.31
#Ca+2	9.36	9.32	9.34
#Nd+3	0.06	0.06	0.06
#P	5.88	5.88	5.88
#Sm+3	0.01	-	0.00
#Y	0.08	0.11	0.09
#Gd+3	0.02	0.03	0.02
#Dy+3	0.01	0.01	0.01
#Yb+3	0.00	0.01	0.00
#Er+3	0.00	-	0.00
#TOTAL	15.76	15.75	15.76
#O-2	25.00	25.00	25.00

TABLEAU 1b :

Analyses à la microsonde électronique des terres rares lourdes dans les apatites.

3 samples, 11 elements

	37	38	Moy.
Fe2O3	0.99	0.71	0.85
SiO2	7.16	5.49	6.32
Ce2O3	14.91	21.25	18.08
Pr2O3	2.67	3.34	3.01
P2O5	34.62	32.59	33.61
CaO	22.61	15.48	19.05
Nd2O3	4.80	6.27	5.54
Y2O3	0.76	0.22	0.49
La2O3	11.97	16.42	14.19
Sm2O3	0.49	0.88	0.68
Gd2O3	0.01	0.40	0.21
Total	100.99	103.05	102.02
	-	-	-
#Fe+3	0.14	0.11	0.12
#Si+4	1.35	1.11	1.23
#Ce+3	1.03	1.57	1.30
#Pr+3	0.18	0.25	0.21
#P	5.52	5.56	5.54
#Ca+2	4.57	3.34	3.95
#Nd+3	0.32	0.45	0.39
#Y	0.08	0.02	0.05
#La+3	0.83	1.22	1.03
#Sm+3	0.03	0.06	0.05
#Gd+3	0.00	0.03	0.01
#TOTAL	14.06	13.71	13.88
#O-2	25.00	25.00	25.00

TABLEAU 1c :
Analyses à la microsonde électronique des cérium-apatites.

6 samples, 11 elements

	9	10	11	12	13	Moy.
Fe2O3	25.54	27.39	20.80	20.61	20.92	23.05
SiO2	30.33	34.22	31.93	31.87	30.99	31.87
Ce2O3	10.81	10.42	11.64	9.12	12.85	10.97
Pr2O3	2.07	1.66	2.26	1.62	1.99	1.92
P2O5	0.22	0.22	0.67	0.27	1.94	0.66
CaO	8.43	8.81	9.02	8.15	8.94	8.67
Nd2O3	3.70	4.62	5.23	5.39	5.53	4.89
Y2O3	-	-	-	-	-	-
La2O3	5.48	4.38	4.21	3.26	4.98	4.46
Sm2O3	0.51	0.56	0.57	0.73	0.60	0.59
Gd2O3	0.09	0.31	0.08	0.02	-	0.10
Total	87.19	92.58	86.39	81.05	88.75	87.19
	-	-	-	-	-	-
#Fe+3	4.31	4.25	3.51	3.61	3.47	3.83
#Si+4	6.81	7.06	7.16	7.43	6.83	7.06
#Ce+3	0.89	0.79	0.96	0.78	1.04	0.89
#Pr+3	0.17	0.12	0.18	0.14	0.16	0.16
#P	0.04	0.04	0.13	0.05	0.36	0.12
#Ca+2	2.03	1.95	2.17	2.03	2.11	2.06
#Nd+3	0.30	0.34	0.42	0.45	0.44	0.39
#Y	-	-	-	-	-	-
#La+3	0.45	0.33	0.35	0.28	0.40	0.36
#Sm+3	0.04	0.04	0.04	0.06	0.05	0.05
#Gd+3	0.01	0.02	0.01	0.00	-	0.01
#TOTAL	15.04	14.94	14.92	14.83	14.85	14.92
#O-2	25.00	25.00	25.00	25.00	25.00	25.00

TABLEAU 2 :
Analyses à la microsonde électronique des allanites.

17 samples, 11 elements

	15	16	17	18	19	20	21	22	27	28
Fe2O3	2.96	3.07	2.66	2.89	2.85	3.12	2.81	2.57	2.77	2.44
SiO2	24.78	27.32	24.16	24.52	26.52	23.83	24.82	20.34	24.11	21.37
Ce2O3	26.29	22.44	23.40	24.36	20.18	25.81	22.10	20.19	21.38	22.99
Pr2O3	4.49	3.76	4.02	3.95	3.51	4.53	4.18	3.54	3.74	3.63
P2O5	0.85	0.94	4.23	3.86	2.25	1.29	4.14	11.02	2.10	1.06
CaO	5.45	4.25	9.86	7.59	5.76	5.79	6.78	13.71	6.01	5.15
Nd2O3	16.03	13.23	13.87	12.80	11.86	15.85	12.05	12.12	10.83	11.63
Y2O3	2.62	2.79	2.30	3.72	5.81	3.04	3.08	2.83	8.61	4.55
La2O3	10.19	8.60	9.06	9.89	8.05	9.40	9.48	8.18	10.24	10.39
Sm2O3	2.27	2.13	2.11	2.00	2.18	2.58	1.93	1.86	1.84	2.18
Gd2O3	1.18	0.95	1.46	1.23	1.91	1.36	1.16	1.49	2.26	2.51
Total	97.10	89.50	97.13	96.81	90.87	96.58	92.52	97.87	93.88	87.89
	-	-	-	-	-	-	-	-	-	-
#Fe+3	0.58	0.61	0.49	0.54	0.55	0.62	0.53	0.44	0.54	0.53
#Si+4	6.49	7.21	5.92	6.06	6.79	6.28	6.27	4.64	6.25	6.22
#Ce+3	2.52	2.17	2.10	2.21	1.89	2.49	2.04	1.69	2.03	2.45
#Pr+3	0.43	0.36	0.36	0.36	0.33	0.43	0.39	0.29	0.35	0.38
#P	0.19	0.21	0.88	0.81	0.49	0.29	0.89	2.13	0.46	0.26
#Ca+2	1.53	1.20	2.59	2.01	1.58	1.63	1.84	3.35	1.67	1.61
#Nd+3	1.50	1.25	1.21	1.13	1.08	1.49	1.09	0.99	1.00	1.21
#Y	0.36	0.39	0.30	0.49	0.79	0.43	0.41	0.34	1.19	0.71
#La+3	0.98	0.84	0.82	0.90	0.76	0.91	0.88	0.69	0.98	1.12
#Sm+3	0.20	0.19	0.18	0.17	0.19	0.23	0.17	0.15	0.16	0.22
#Gd+3	0.10	0.08	0.12	0.10	0.16	0.12	0.10	0.11	0.19	0.24
#TOTAL	14.89	14.52	14.97	14.78	14.61	14.93	14.60	14.82	14.83	14.95
#O-2	25.00	25.00	25.00	25.00	25.00	25.00	25.00	25.00	25.00	25.00

	29	30	33	34	35	36	5	Moy.
Fe2O3	3.13	3.16	-	0.95	-	0.07	0.13	2.22
SiO2	24.13	24.02	24.38	19.97	21.21	16.68	35.96	23.26
Ce2O3	22.93	23.74	20.90	17.25	16.43	15.36	17.13	21.61
Pr2O3	4.12	3.94	3.34	2.72	2.62	2.67	3.04	3.67
P2O5	1.61	1.63	2.50	3.81	9.86	16.53	8.13	4.23
CaO	5.85	6.32	11.21	11.62	17.54	22.50	7.10	9.09
Nd2O3	11.40	12.33	10.28	8.30	8.45	7.65	8.49	11.79
Y2O3	5.99	5.22	12.32	12.37	12.29	8.81	1.05	6.02
La2O3	12.47	11.79	10.85	9.15	7.74	7.79	8.79	9.58
Sm2O3	2.28	2.19	1.97	1.62	1.56	1.61	1.40	2.02
Gd2O3	1.68	1.51	2.26	2.52	2.21	1.78	0.99	1.72
Total	95.59	95.85	100.02	90.26	99.89	101.46	92.20	95.20
	-	-	-	-	-	-	-	-
#Fe+3	0.61	0.62	-	0.19	-	0.01	0.02	0.43
#Si+4	6.28	6.24	5.93	5.31	4.69	3.49	7.53	5.88
#Ce+3	2.18	2.26	1.86	1.68	1.33	1.18	1.31	2.00
#Pr+3	0.39	0.37	0.30	0.26	0.21	0.20	0.23	0.34
#P	0.35	0.36	0.51	0.86	1.85	2.93	1.44	0.84
#Ca+2	1.63	1.76	2.92	3.31	4.16	5.04	1.59	2.36
#Nd+3	1.06	1.14	0.89	0.79	0.67	0.57	0.63	1.07
#Y	0.83	0.72	1.59	1.75	1.45	0.98	0.12	0.80
#La+3	1.20	1.13	0.97	0.90	0.63	0.60	0.68	0.89
#Sm+3	0.20	0.20	0.16	0.15	0.12	0.12	0.10	0.18
#Gd+3	0.15	0.13	0.18	0.22	0.16	0.12	0.07	0.14
#TOTAL	14.88	14.93	15.32	15.43	15.26	15.23	13.73	14.93
#O-2	25.00	25.00	25.00	25.00	25.00	25.00	25.00	25.00

TABLEAU 3 :
Analyses à la microsonde électronique des cérium-britholites, l'analyse 5 n'a pas été prise en compte dans le calcul de la moyenne (cf. texte).

8 samples, 11 elements

	B4	B5	B6	B7	B8	B9	B10	Moy.
Fe203	0.86	0.87	1.07	1.07	0.38	0.23	0.43	0.70
Si02	36.48	37.29	36.65	35.56	36.70	36.46	36.78	36.56
CaO	16.75	16.68	16.64	16.23	16.45	16.78	16.72	16.61
Nd203	0.31	0.49	0.43	0.80	0.80	1.39	0.90	0.73
P205	2.68	2.19	2.02	1.98	2.17	2.23	2.14	2.20
Sm203	0.52	0.87	0.85	0.96	0.73	0.91	0.68	0.79
Y203	28.17	27.47	26.95	24.93	27.67	26.34	28.32	27.12
Gd203	1.84	2.15	1.70	2.06	1.71	1.83	1.66	1.85
Dy203	2.91	2.72	2.92	2.80	2.78	2.48	2.22	2.69
Yb203	2.51	2.77	2.76	2.61	2.77	1.97	2.80	2.60
Er203	2.35	2.29	2.69	2.41	2.51	2.65	2.37	2.47
Total	95.39	95.77	94.69	91.40	94.67	93.27	95.02	94.31
	-	-	-	-	-	-	-	-
#Fe+3	0.10	0.10	0.13	0.13	0.05	0.03	0.05	0.09
#Si+4	5.83	5.95	5.93	5.96	5.94	5.96	5.92	5.93
#Ca+2	2.87	2.85	2.89	2.91	2.85	2.94	2.88	2.88
#Nd+3	0.02	0.03	0.02	0.05	0.05	0.08	0.05	0.04
#P	0.36	0.30	0.28	0.28	0.30	0.31	0.29	0.30
#Sm+3	0.03	0.05	0.05	0.06	0.04	0.05	0.04	0.04
#Y	2.40	2.33	2.32	2.22	2.38	2.29	2.43	2.34
#Gd+3	0.10	0.11	0.09	0.11	0.09	0.10	0.09	0.10
#Dy+3	0.15	0.14	0.15	0.15	0.14	0.13	0.12	0.14
#Yb+3	0.12	0.13	0.14	0.13	0.14	0.10	0.14	0.13
#Er+3	0.12	0.11	0.14	0.13	0.13	0.14	0.12	0.13
#TOTAL	12.10	12.10	12.13	12.13	12.11	12.12	12.13	12.12
#O-2	20.00	20.00	20.00	20.00	20.00	20.00	20.00	20.00

TABLEAU 4 :
Analyses à la microsonde électronique des yttrium-britholites.

3 samples, 11 elements

	4	7	Moy.
Fe2O3	-	0.01	0.00
SiO2	0.40	0.62	0.51
Ce2O3	26.18	26.94	26.56
Pr2O3	4.57	4.71	4.64
P2O5	4.26	10.73	7.49
CaO	9.58	10.19	9.88
Nd2O3	13.08	12.72	12.90
Y2O3	0.84	1.51	1.18
La2O3	14.49	13.84	14.17
Sm2O3	1.95	2.02	1.98
Gd2O3	1.04	1.34	1.19
Total	76.38	84.64	80.51
	-	-	-
#O-2	-	-	-
#Fe+3	-	0.00	0.00
#Si+4	0.18	0.21	0.19
#Ce+3	4.25	3.40	3.82
#Pr+3	0.74	0.59	0.66
#P	1.60	3.13	2.36
#Ca+2	4.55	3.76	4.15
#Nd+3	2.07	1.57	1.82
#Y	0.20	0.28	0.24
#La+3	2.37	1.76	2.06
#Sm+3	0.30	0.24	0.27
#Gd+3	0.15	0.15	0.15
#TOTAL	16.39	15.10	15.74
#O-2	24.00	24.00	24.00

TABLEAU 5 :
Analyses à la microsonde électronique des mélanges d'oxydes de terres rares.

	Y2O3	La2O3	Ce2O3	Pr2O3	Nd2O3	Sm2O3	Gd2O3	Dy2O3	Er2O3	Yb2O3
Apatite(A)	1.74	1.76	3.74	0.63	2.02	0.33	0.29	0.15	0.02	0.10
Ce Apatite(TR)	0.49	14.19	18.08	3.01	5.54	0.68	0.21	nd	nd	nd
Ce Britholite(F)	4.55	9.58	21.61	3.67	11.79	2.02	1.72	nd	nd	nd
Y Britholite(R)	27.12	nd	nd	nd	0.73	0.79	1.85	2.69	2.47	2.60
Allanite(R)	0.00	4.46	10.97	1.92	4.89	0.59	0.10	nd	nd	nd
Oxydes(R)	1.18	14.17	26.56	4.64	12.90	1.98	1.19	nd	nd	nd

Tableau 6 :
Teneurs en oxydes de terres rares des différentes phases minérales rencontrées. L'estimation de leur abondance est indiquée entre parenthèse (A: abondantes, F: faible, R: rares, TR : très rares. Ces estimations sont des estimations visuelles).

4 samples, 22 elements

	-1-	-2-	-3-	-4-
SiO2	46.87	36.74	67.32	37.84
TiO2	0.80	3.21	-	1.02
Al2O3	3.65	15.90	19.24	10.77
FeO	18.86	30.22	-	24.76
MnO	-	-	-	-
MgO	10.24	0.14	-	9.75
CaO	17.52	-	0.02	9.77
Na2O	0.64	0.10	6.42	2.10
K2O	0.70	9.01	7.12	1.92
Total	99.28	95.32	100.12	97.93
	-	-	-	-
#Si+4	3.08	2.67	3.75	2.61
#Ti+4	0.04	0.18	-	0.05
#Al+3	0.28	1.36	1.26	0.87
#Fe+2	1.04	1.83	-	1.43
#Mn+2	-	-	-	-
#Mg+2	1.00	0.02	-	1.00
#Ca+2	1.23	-	0.00	0.72
#Na+1	0.08	0.01	0.69	0.28
#K	0.06	0.83	0.51	0.17
#TOTAL	6.81	6.90	6.22	7.13
#O-2	10.00	10.00	10.00	10.00

TABLEAU 7 :

Analyses à la microsonde électronique des principaux silicates rencontrés dans l'échantillon. 1 : Clinopyroxène, 2 : biotite, 3 : Plagioclase, 4 : amphibole. L'analyse de chacun des minéraux correspond à une moyenne sur cinq analyses.

LEGENDES DES PHOTOS ET DES FIGURES

Toutes les photographies ont été réalisées au microscope électronique à balayage, en images d'électrons secondaires. Dans ce type d'imagerie, les différences de teintes que l'on observe sur les clichés s'expliquent par la variation du numéro atomique moyen des différentes phases, plus celui-ci est élevé (présence d'éléments lourds), plus les teintes sont claires.

PHOTO A :
Fragment d'apatite, l'apatite primaire apparaît en gris clair, l'apatite secondaire apparaît en gris plus foncé et les minéraux de terres rares en blanc. Points d'analyses 2 à 7.

PHOTO B :
Assemblage de cristaux d'allanite, les points blancs sont des oxydes de fer. Points d'analyses 11, 12 et 13.

PHOTO C :
Cristal de clinopyroxène (gris foncé), associé à un fragment d'apatite. Les minéraux blanc qui remplissent les clivages et les fractures du clinopyroxène sont essentiellement des yttrium-britholite. Points d'analyses 8, 9 et 10 et B2 à B11.

FIGURE D :
Localisation des points d'analyses correspondants à la photo C.

PHOTO E :
Association d'apatite primaire riche en terres rares (gris clair), d'apatite secondaire sans terres rares (gris foncé) et de cérium-britholite (blanc).

PHOTO F :
Détail de la photo précédente. points d'analyses 14 à 23.

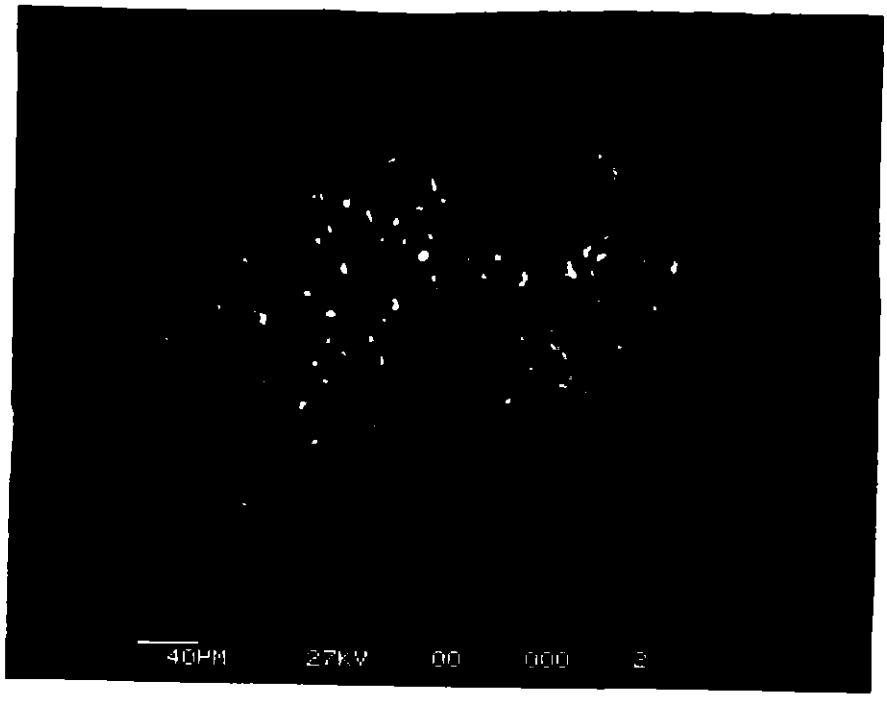
PHOTO G :
Localisation des points d'analyses correspondants à la photo F.

PHOTO H :
Fragment d'apatite primaire. Ce fragment est traversé par un filonnet d'apatite secondaire (gris foncé) dans lequel se trouvent des plages de cérium-britholite. Points d'analyses 24 à 30.

FIGURE I :
Localisation des points d'analyses correspondants à la photo H.

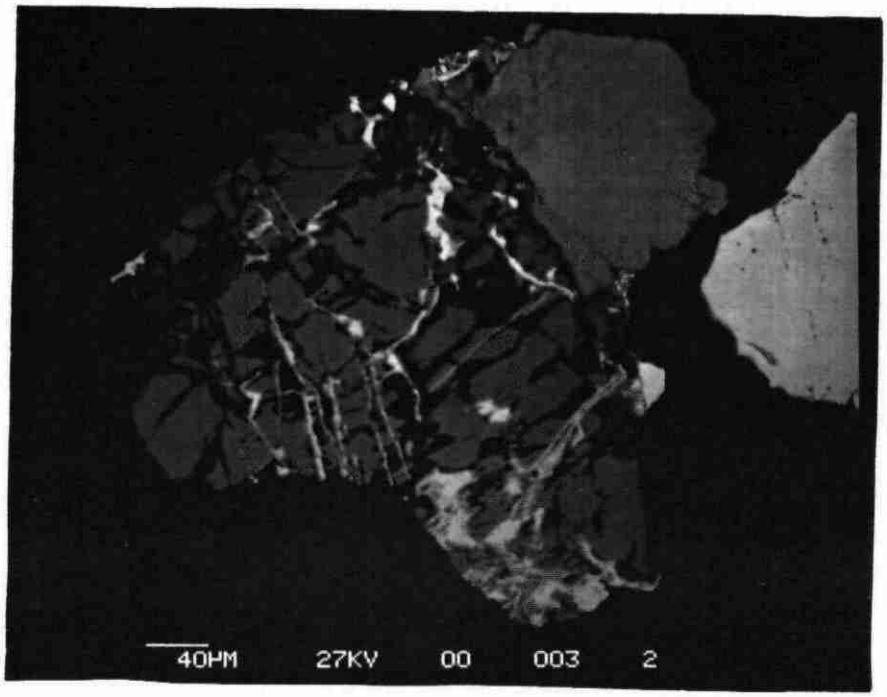
PHOTO J :
Fragment d'apatite primaire et d'apatite secondaire. On retrouve la cérium-britholite en inclusion dans l'apatite secondaire (plages blanches). Points d'analyses 31 à 36.

PHOTO K :
Fragment d'apatite primaire. Les minéraux blancs qui remplissent les fissures sont des cérium apatites. Points d'analyses 37 et 38.



A

B



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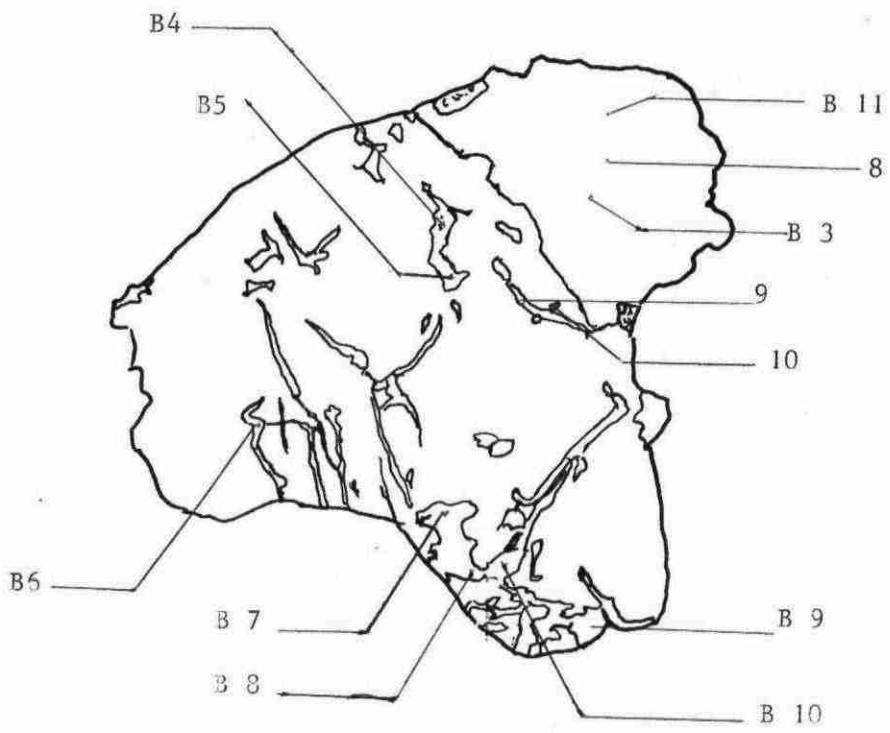

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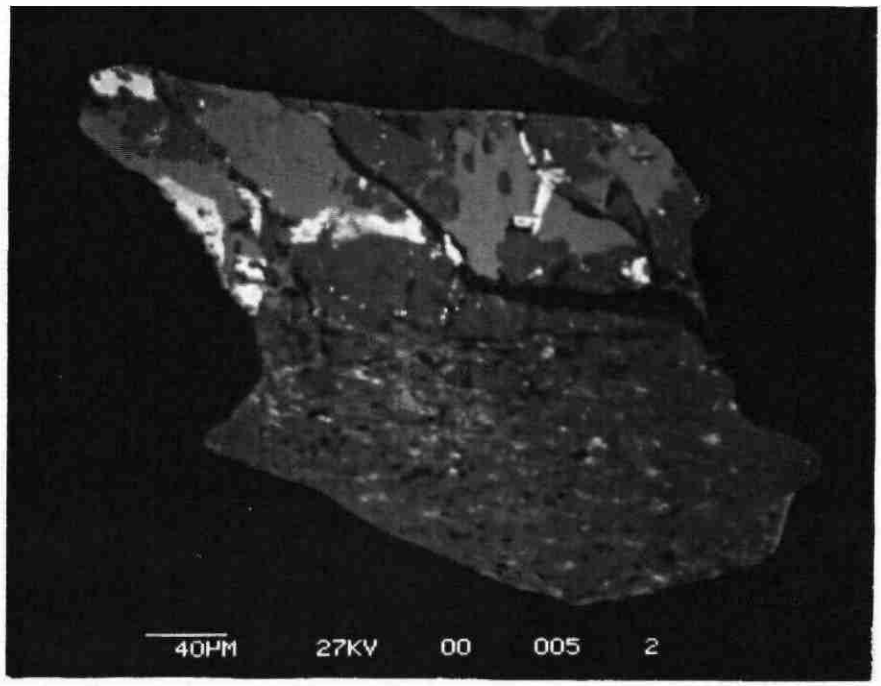

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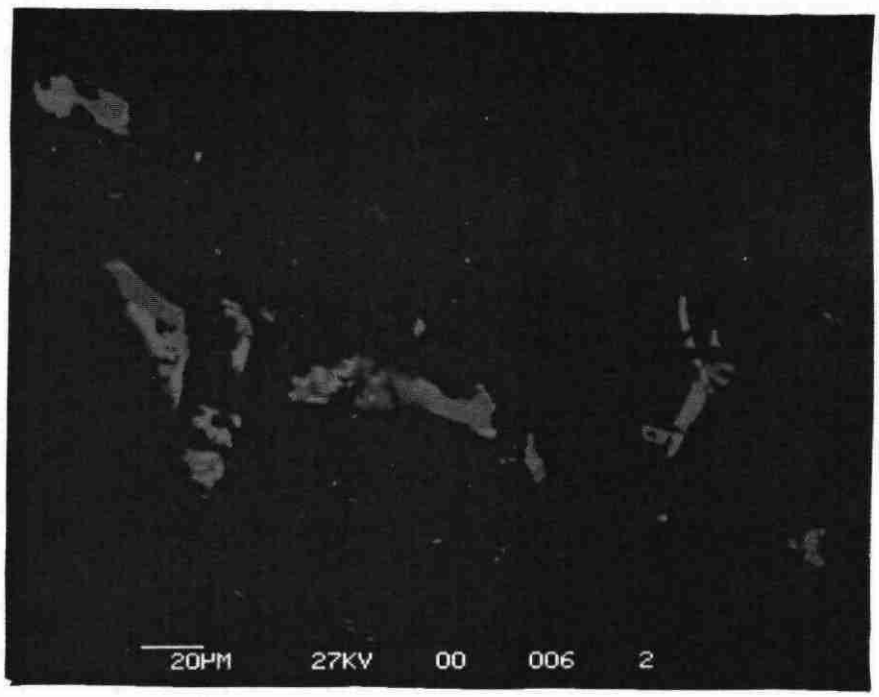

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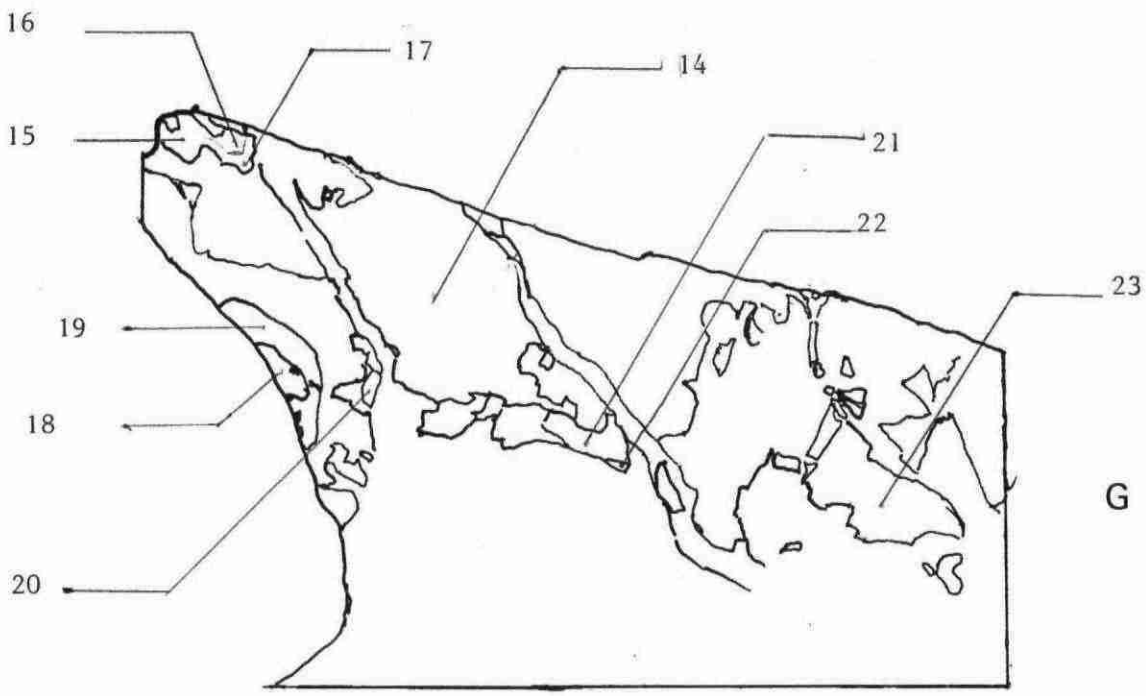

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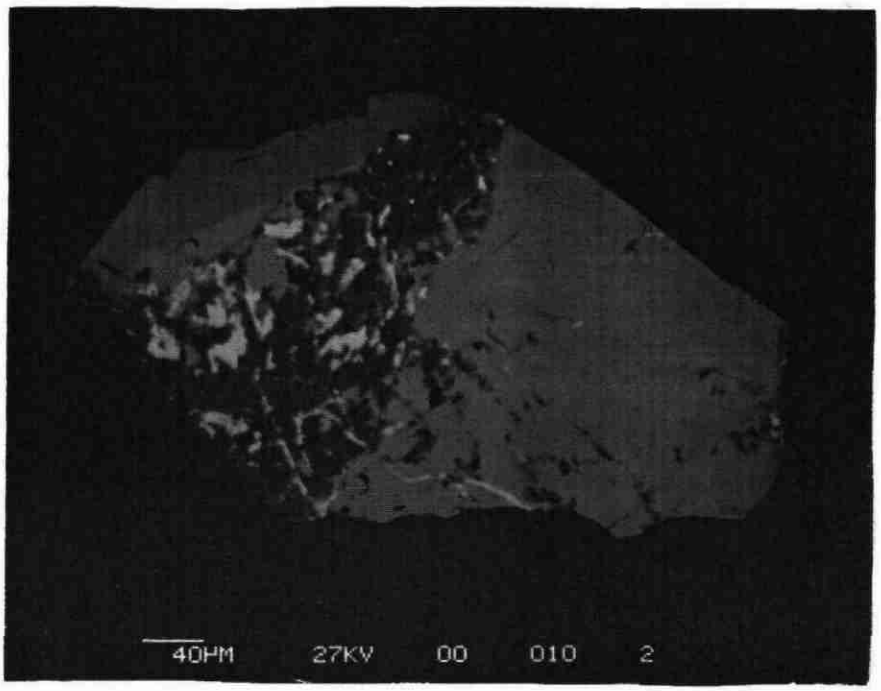

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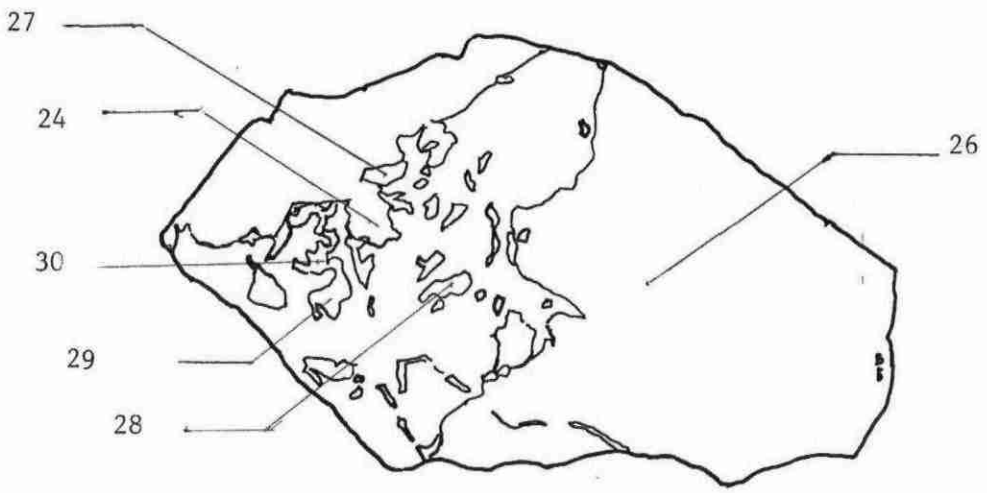

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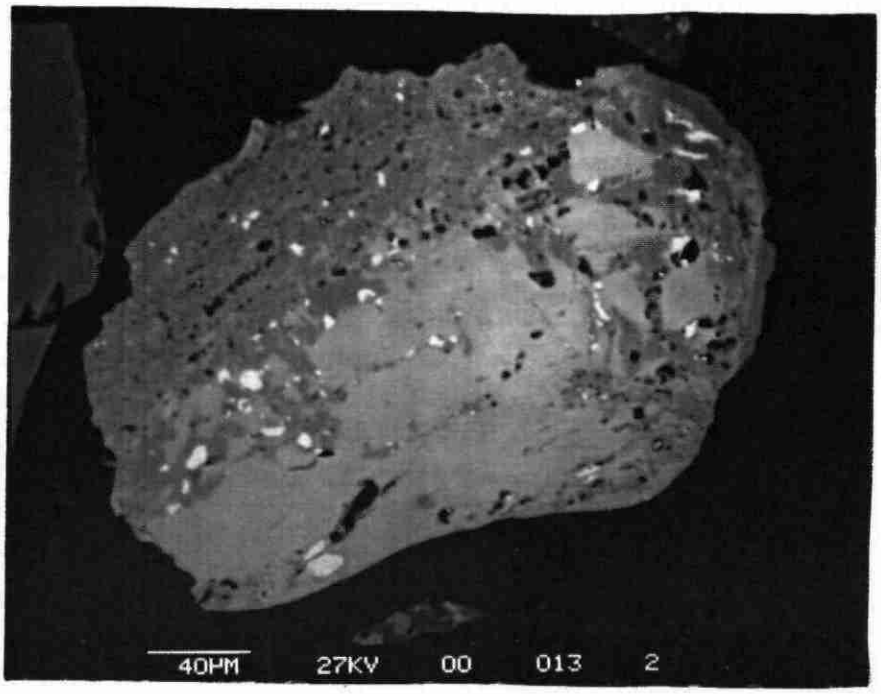
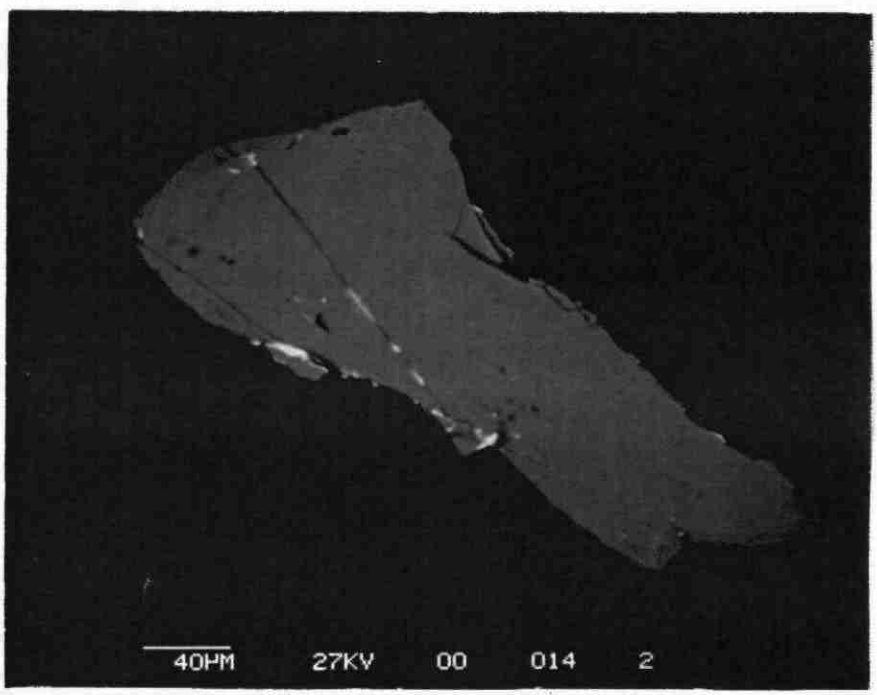


J



K

BRGM

DIRECTION DE LA TECHNOLOGIE

Département analyse

RÉSULTATS D'ÉTUDES

ETUDE M 1364 C

Orléans, le 10 août 1988

Demandeur M. BARON
M. BAUDET DAM/MIN

DETERMINATION MINERALOGIQUE D'ECHANTILLONS EN POUDRE

Méthode employée : Diffractométrie de rayons X (appareillage Philips)
- rayonnement CoKα, monochromateur arrière en graphite
- sensibilité : 2000 c/s
- domaine d'exploration du spectre : 4 à 84° 2θ
- échelle d'importance : TA très abondant - A abondant - P présent - F faible - Tr traces.

Préparation : Pastillage à sec sous faible pression

Résultats : Apatite USA

N° 07

Quartz Tr
Plagioclase F
Amphibole Tr
Calcite Tr
Sidérite infra Tr
Magnétite P/F
Apatite (probable) infra Tr

Phase argileuse ou phylliteuse P
Kaolinite Tr/F
Mica et/ou Illite Tr
Smectite + Chlorite (probable) F
Sépiolite Tr
Talc (probable) Tr

N° 09

Quartz Tr(+)
Plagioclase F/P
Amphibole infra Tr
Calcite infra Tr
Magnétite (Probable) infra Tr
Apatite (probable) infra Tr
Phase argileuse ou phylliteuse P ou P(-)
- Mica et Illite F
- Smectite et chlorite (probable) F
- Kaolinite Tr
- Sépiolite (probable) infra Tr

Conclusion :

La minéralogie de ces deux échantillons est assez complexe, en particulier dans le domaine des argiles. Une étude plus approfonde sur lames orientées serait nécessaire pour plus de précisions dans ce domaine.

F. PILLARD

P.o

Coordonnateur des Études
Département analyse

A P P E N D I X 2

Mineralogic study of the REO ore sample 2

by O. LEGENDRE (BRGM)

Etude du minerai carbonaté à monazite du Malawi

Par O. Legendre

DT/ANA. Etude N 010707

1 INTRODUCTION

Cette étude sur le minerai carbonaté à monazite du Malawi a pour but de principal de localiser dans les minéraux de la gangue des éléments spécifiques accompagnateurs des oxydes de terres rares. Ces éléments sont le strontium, le baryum et le manganèse.

Bien que le porteur principal des oxydes de terres rares soit bien défini -il s'agit de la monazite-, nous avons effectué un contrôle sur le chimisme de celui-ci, et recherché d'autres phases susceptible de contenir des oxydes de terres rares.

2 TECHNIQUES ANALYTIQUES

Cette étude à été réalisée sur le tout-venant non déshlamé broyé à une maille inférieure à 2 mm (échantillon n° 01). Pour effectuer l'étude minéralogique et chimique ponctuelle, cet échantillon a été préparé sous forme de lame mince polie.

Un deuxième échantillon (n° 10), provenant de l'alimentation flottation (granulométrie comprise entre 160 µm et 10 µm) a été préparé sous forme de section polie pour effectuer un contrôle sur la minéralogie.

L'étude à été menée en trois étapes successives :

- un repérage au microscope pétrographique des différentes phases minérales du minerai.
  une observation au microscope électronique à balayage, ceci pour une sélection définitive des minéraux contenant

des oxydes de terres rares et des minéraux composant la gangue en vue de l'analyse à la microsonde électronique.

- une étude du chimisme des différents minéraux à la microsonde électronique. Compte tenu du comportement particulier des éléments de terres rares en émission de rayons X (Raies d'émission très proches les unes des autres, pouvant occasionner des phénomènes d'interférences, il a été nécessaire de procéder, avant l'analyse proprement-dite, a une détermination qualitative fine des éléments présents dans chacun des minéraux à analyser.
  Nous avons retenu les éléments suivants :
  F, P, Ca, Fe, Ce, Pr, Nd, La, Sm, Gd, et Mn.
  Il n'a cependant pas été possible de contourner complètement ces phénomènes d'interférences dans l'analyse des monazites. Ceci explique les totaux au alentours de 105% pondéral pour l'analyse de ces minéraux.
  Pour les minéraux composant la gangue, nous avons retenu, a la suite des observations au microscope électronique les éléments suivants :
  S, Ba, Sr, Ca, Ti, Mn, Mg, La, Fe, Ce

3 RESULTATS

3.1 Les minéraux à oxydes de terres rares

La paragénèse des minéraux à oxydes de terres rares dans le minerai du Malawi est assez simple. En fait seuls trois porteurs de ces oxydes ont été rencontrés dans l'échantillon étudié.

3.1.1 La monazite

Ce minéral était déjà connu pour être le porteur principal des oxydes de terres rares dans ce minerai.

Elle semble représenter plus de 95% des minéraux porteurs de terres rares.

Elle se présente généralement en plages bien automorphes, d'une taille moyenne d'environ 100 à 500 µm. Ces plages sont très souvent en inclusions dans les carbonates (photo 1 et 3).

Les analyses des monazites, réalisées à la microsonde électronique sont présentées dans le tableau 1. Les oxydes de terres rares présents dans ces monazites sont, par ordre d'abondance Ce_2O_3 (35% en poids), La_2O_3 (18,4% en poids), Nd_2O_3 (11,9% en poids), et dans une moindre mesure Pr_2O_3, Gd_2O_3 et Sm_2O_3 (en teneurs allant de 5 à 1% pondéral).

Ces teneurs fluctuent assez peu sur l'ensemble des monazites analysées. On notera cependant, pour certaines d'entre elles, une zonation chimique marquée par un enrichissement relatif de Ce_2O_3 par rapport à La_2O_3, en allant vers la périphérie des grains (Photo 2).

3.1.2 L'apatite

Elle ne représente que quelques pour-cent de la masse des minéraux de terres rares.

Elle se présente en plages de quelques centaines de micromètres, soit fibreuse, soit monocristalline ; dans ce cas elle est généralement automorphe.

Les analyses de ces apatites à la microsonde électronique (tableau 2) montrent des teneurs en oxydes de terres rares assez variables. En général, les variétés fibreuses en sont pratiquement dépourvues.

Comme dans les monazites, les terres rares présentes sont par ordre d'abondance Ce_2O_3, La_2O_3 et Nd_2O_3, avec des teneurs pondérales maximum de 8,20% Ce_2O_3, 4,40% La_2O_3 et 3,22% Nd_2O_3.

3.1.3 Minéral de terres rares non identifié

Ce minéral a révélé lors des analyses qualitatives, de très fortes teneurs en oxydes de terres rares. En fait, il s'agit très probablement d'un carbonate de terres rares (Bastnaesite ou Parisite).

Nous n'avons observé ce minéral qu'une seule fois. Il se présente sous forme de gerbes fibroradiées (photo 4) en inclusions dans un carbonate (Calcite).

3.2 Les minéraux de la gangue

La paragénèse des minéraux de la gangue est assez monotone. Ce sont pour l'essentiel des carbonates, mais on observe aussi des sulfates, des oxydes et des silicates.

3.2.1 Les carbonates

Ils sont subdivisés en quatre familles minéralogiques bien distinctes, en fonction de leur chimisme :

- Carbonate de calcium et de magnésium (Dolomite)
  Ce carbonate est le plus abondant.
  Les analyses à la microsonde électronique son présentées dans le tableau 3. Outre le calcium et le magnésium, ce carbonate présente parfois des teneurs non négligeables en fer (jusqu'à 7,43% pondéral de $FeCO_3$) et de manganèse (jusqu'à 3,73% de $MnCO_3$).

- Carbonate de Strontium (Strontianite)
  Ce carbonate est fréquemment rencontré, il représente peut-être 25 à 30% de la masse des carbonates
  Les analyses ponctuelles de ces strontianites (tableau 4) montrent qu'elles sont composées presque exclusivement de $SrCO_3$ (!), les teneurs sont de l'ordre de 97% en poids de $SrCO_3$.
  Les seuls autres éléments présents sont le calcium (3% de $CaCO_3$) et le baryum, en trace (inférieur à 0,5% pondéral de $BaCO_3$).

- Carbonate de fer (Sidérite)
  Ce carbonate est moyennement présent (5 à 10% de la masse des carbonates)

En fait ce carbonate n'est pas une sidérite *sensu stricto* car les analyses (Tableau 5a) montrent des teneurs assez importantes de calcium, de manganèse et de magnésium (5 à 10% en poids de $MnCO_3$, $CaCO_3$ ou $MgCO_3$).

Une seule analyse de ce carbonate a été réalisée, le total nettement supérieur à 100% s'explique par le fait que ces sidérites sont presque systématiquement mélangées à des oxydes de fer (elles apparaissent opaques au microscope pétrographique).

- Carbonate de calcium seul (Calcite)

  Cette calcite est le moins abondant des carbonates rencontrés dans ce minerai.

  Les analyses (tableau 5b) montrent qu'elle présente des teneurs $FeCO_3$ de l'ordre de 6% en poids et en $SrCO_3$ d'environ 5% en poids.

3.2.2 Les sulfates

En fait de sulfates il s'agit uniquement de **barytine.** Elle est assez faiblement présente, elle compte pour environ 5% de la masse du minerai.

Les analyses réalisées à la microsonde électronique (Tableau 6) montrent qu'il s'agit d'une barytine tout à fait classique. On notera cependant quelques traces de strontium (teneurs inférieures à 5% de $SrSO_4$ en poids).

Morphologiquement, elle se présente en grand cristaux (en forme de latte) de plusieurs centaines de micromètres de longueur.

3.2.3 Les oxydes

Ce sont pour l'essentiel des oxydes de manganèse, très probablement proches du psilomélane.

Ces oxydes contiennent des quantités très variables de Fe_2O_3 et de BaO, on notera aussi des teneurs allant jusqu'à 3% de Ce_2O_3 et de SrO.

Ce type d'oxyde est généralement hydraté, ce qui expliquerait que le total de l'analyse soit inférieur à 100%.

Le sodium et le potassium, souvent présents dans les psilomélanes, n'ont pas été détectés dans ceux de cette étude lors des observations au microscope électronique à balayage.

Ces oxydes sont présent sous forme de grains xénomorphes de taille assez importante (jusqu'à 1 mm de longueur) dans lesquels on peut retrouver en inclusion, des carbonates et des apatites.

Ces oxydes de manganèse représentent plusieurs pour-cent de la masse du minerai.

3.2.4 Les silicates

Le seul silicate rencontré dans cet échantillon est le quartz (bien que certaines classifications le rangent parmi les oxydes !). Il est en proportion inférieure à 10% de la masse du minerai.

4 CONCLUSIONS

Cette étude, menée sur le minerai carbonaté à monazite du Malawi a montré que :

- La paragénèse des minéraux de terres rares est assez simple. Elle est constituée de monazite, d'apatite (enrichie en oxydes de terres rares) et d'un carbonate de terres rares (probablement bastnaesite ou parisite).
  Cependant, compte tenu des teneurs modérées en oxydes de terres rares et de la faible abondance de l'apatite d'une part, et d'autre part de la rareté du carbonate de terres rares, il semble que la monazite seule présente un intérêt du point de vue minéralurgique dans ce gisement.

- Les minéraux de la gangue sont en nombre assez restreints, ce sont pour l'essentiel des carbonates répartis en quatre

	12	13	14	15	16	17	18	19	33
F	0.0042	0.0054	0.0063	0.0043	0.0046	0.0036	0.0041	0.0045	0.0023
PR	0.0537	0.0487	0.0600	0.0531	0.0483	0.0559	0.0537	0.0525	0.0561
P	0.3108	0.3062	0.2935	0.3039	0.3072	0.2989	0.2978	0.3019	0.3090
CE	0.3372	0.3498	0.3563	0.3442	0.3456	0.3488	0.3458	0.3482	0.3544
ND	0.1316	0.1183	0.1183	0.1326	0.1222	0.1034	0.1016	0.0957	0.1234
CA	0.0038	0.0037	0.0030	0.0052	0.0037	0.0037	0.0040	0.0042	0.0018
SM	0.0137	0.0144	0.0108	0.0128	0.0114	0.0099	0.0097	0.0068	0.0103
LA	0.1654	0.1808	0.1857	0.1694	0.1808	0.2022	0.2085	0.2043	0.1766
GD	0.0310	0.0306	0.0313	0.0329	0.0291	0.0375	0.0320	0.0358	0.0313
MN	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
FE	0.0000	0.0008	0.0009	0.0008	0.0000	0.0009	0.0000	0.0000	0.0000
F =0	-0.0018	-0.0023	-0.0026	-0.0018	-0.0019	-0.0015	-0.0017	-0.0019	-0.0010
	1.0496	1.0565	1.0634	1.0573	1.0509	1.0633	1.0555	1.0520	1.0643
PR	0.1475	0.1343	0.1681	0.1466	0.1333	0.1552	0.1498	0.1460	0.1535
P	1.9840	1.9609	1.9097	1.9505	1.9697	1.9269	1.9308	1.9491	1.9630
CE	0.9308	0.9686	1.0024	0.9553	0.9584	0.9724	0.9695	0.9721	0.9737
ND	0.3544	0.3196	0.3247	0.3591	0.3307	0.2812	0.2779	0.2606	0.3308
CA	0.0307	0.0302	0.0249	0.0422	0.0301	0.0306	0.0332	0.0341	0.0146
SM	0.0357	0.0376	0.0285	0.0335	0.0296	0.0261	0.0256	0.0180	0.0267
LA	0.4601	0.5044	0.5263	0.4738	0.5051	0.5678	0.5889	0.5746	0.4888
GD	0.0774	0.0768	0.0798	0.0826	0.0730	0.0946	0.0813	0.0905	0.0779
MN	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
FE	0.0000	0.0050	0.0056	0.0048	0.0000	0.0058	0.0000	0.0000	0.0000
	4.0206	4.0374	4.0700	4.0483	4.0299	4.0606	4.0569	4.0449	4.0292
F	0.0997	0.1292	0.1527	0.1036	0.1102	0.0867	0.0986	0.1093	0.0541

Tableau 1: ../..

	34	35		AVERAGE FOR 11 ANALYSIS
F	0.0039	0.0045	F	0.0043
PR	0.0547	0.0539	PR	0.0537
P	0.3144	0.3158	P	0.3054
CE	0.3586	0.3602	CE	0.3499
ND	0.1264	0.1318	ND	0.1187
CA	0.0015	0.0015	CA	0.0033
SM	0.0132	0.0091	SM	0.0111
LA	0.1782	0.1760	LA	0.1844
GD	0.0345	0.0305	GD	0.0324
MN	0.0000	0.0000	MN	0.0000
FE	0.0000	0.0000	FE	0.0003
F =O	-0.0016	-0.0019	F =O	-0.0018
	1.0838	1.0815		1.0616
PR	0.1471	0.1449	PR	0.1478
P	1.9639	1.9709	P	1.9527
CE	0.9689	0.9722	CE	0.9677
ND	0.3331	0.3470	ND	0.3199
CA	0.0121	0.0117	CA	0.0268
SM	0.0334	0.0231	SM	0.0289
LA	0.4849	0.4787	LA	0.5139
GD	0.0844	0.0745	GD	0.0812
MN	0.0000	0.0000	MN	0.0000
FE	0.0000	0.0000	FE	0.0019
	4.0278	4.0230		4.0408
F	0.0905	0.1040	F	0.1035

Tableau 1 (Suite): Analyses à la microsonde éléctronique des monazites. La colonne supérieure donne les poucentages en poids des différents oxydes. Les totaux supérieurs à 100 sont dus à de nombreux phénomènes d'interférences sur les raie d'emission X des terres rares. La colonne inférieure correspond aux pourcentage atomique calculé sur la base de 8 oxygènes.

	1	3	4	5	9	10	11	21	22
F	0.0498	0.0527	0.0282	0.0285	0.0287	0.0317	0.0201	0.0293	0.0302
PR	0.0039	0.0031	0.0000	0.0000	0.0076	0.0037	0.0130	0.0030	0.0000
P	0.4042	0.4045	0.4100	0.3946	0.3935	0.3889	0.3864	0.4229	0.4218
CE	0.0237	0.0199	0.0046	0.0059	0.0243	0.0149	0.0820	0.0056	0.0042
ND	0.0138	0.0148	0.0073	0.0040	0.0159	0.0132	0.0322	0.0058	0.0032
CA	0.4984	0.4720	0.5194	0.5029	0.4822	0.4804	0.4038	0.5205	0.5206
SM	0.0008	0.0019	0.0013	0.0009	0.0024	0.0000	0.0047	0.0014	0.0014
LA	0.0099	0.0097	0.0026	0.0011	0.0091	0.0055	0.0440	0.0024	0.0011
GD	0.0024	0.0025	0.0000	0.0005	0.0028	0.0025	0.0097	0.0016	0.0000
MN	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0005	0.0000
FE	0.0006	0.0005	0.0000	0.0006	0.0001	0.0000	0.0000	0.0117	0.0206
H2O	0.0000	0.0000	0.0038	0.0031	0.0030	0.0013	0.0067	0.0038	0.0034
F =O	-0.0210	-0.0222	-0.0119	-0.0120	-0.0121	-0.0133	-0.0085	-0.0124	-0.0127
	0.9866	0.9596	0.9653	0.9299	0.9576	0.9288	0.9941	0.9962	0.9937
PR	0.0244	0.0196	0.0000	0.0000	0.0498	0.0250	0.0878	0.0186	0.0000
P	5.8513	5.9298	6.0568	6.0468	6.0207	6.0546	6.0517	6.0728	6.0602
CE	0.1484	0.1264	0.0293	0.0392	0.1610	0.1004	0.5551	0.0347	0.0261
ND	0.0846	0.0914	0.0458	0.0257	0.1028	0.0869	0.2128	0.0354	0.0192
CA	9.1317	8.7573	9.7089	9.7533	9.3376	9.4662	8.0031	9.4602	9.4667
SM	0.0046	0.0114	0.0079	0.0054	0.0150	0.0000	0.0297	0.0085	0.0081
LA	0.0623	0.0622	0.0165	0.0071	0.0606	0.0374	0.3003	0.0151	0.0069
GD	0.0135	0.0146	0.0000	0.0033	0.0167	0.0152	0.0594	0.0090	0.0000
MN	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0069	0.0000
FE	0.0086	0.0075	0.0000	0.0086	0.0017	0.0000	0.0000	0.1655	0.2921
	15.3293	15.0202	15.8651	15.8894	15.7660	15.7856	15.2999	15.8267	15.8795
F	2.6959	2.8854	1.5556	1.6295	1.6392	1.8439	1.1748	1.5736	1.6199
OH	0.0000	0.0000	0.4444	0.3705	0.3608	0.1561	0.8252	0.4264	0.3801

Tableau 2 : ../..

	23	24		AVERAGE FOR 11 ANALYSIS
F	0.0339	0.0276	F	0.0328
PR	0.0029	0.0018	PR	0.0035
P	0.4214	0.4019	P	0.4046
CE	0.0064	0.0126	CE	0.0186
ND	0.0041	0.0092	ND	0.0112
CA	0.5352	0.5096	CA	0.4950
SM	0.0014	0.0002	SM	0.0015
LA	0.0021	0.0034	LA	0.0083
GD	0.0000	0.0004	GD	0.0020
MN	0.0023	0.0071	MN	0.0009
FE	0.0072	0.0105	FE	0.0047
H2O	0.0017	0.0040	H2O	0.0028
F =O	-0.0143	-0.0116	F =O	-0.0138
	1.0043	0.9768		0.9721
PR	0.0180	0.0112	PR	0.0231
P	6.0135	5.9544	P	6.0102
CE	0.0398	0.0810	CE	0.1219
ND	0.0244	0.0576	ND	0.0715
CA	9.6653	9.5547	CA	9.3005
SM	0.0081	0.0015	SM	0.0091
LA	0.0131	0.0217	LA	0.0548
GD	0.0000	0.0026	GD	0.0122
MN	0.0295	0.0947	MN	0.0119
FE	0.1016	0.1538	FE	0.0672
	15.9133	15.9332		15.6826
F	1.8084	1.5293	F	1.8141
OH	0.1916	0.4707	OH	0.3296

Tableau 2 (Suite) : Analyses à la microsonde électronique des apatites. Les pourcentages atomiques sont calculés sur la base de 25 oxygènes et 1 H2O.

	3	4	14	15	22	23	24	25	26
S	0.0013	0.0000	0.0014	0.0000	0.0000	0.0000	0.0000	0.0012	0.0000
BA	0.0000	0.0020	0.0022	0.0000	0.0023	0.0000	0.0026	0.0000	0.0000
SR	0.0038	0.0025	0.0117	0.0021	0.0064	0.0050	0.0161	0.0072	0.0077
CA	0.4871	0.4888	0.5042	0.5207	0.5149	0.5233	0.5175	0.5274	0.4840
TI	0.0000	0.0017	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
MN	0.0269	0.0434	0.0000	0.0100	0.0067	0.0188	0.0004	0.0063	0.0303
MG	0.3796	0.3332	0.4258	0.3514	0.4258	0.4092	0.4378	0.4211	0.3736
LA	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0004	0.0000	0.0005
FE	0.0468	0.0847	0.0000	0.0000	0.0150	0.0051	0.0002	0.0055	0.0657
CE	0.0004	0.0005	0.0000	0.0000	0.0007	0.0001	0.0008	0.0000	0.0000
	0.9460	0.9568	0.9453	0.8841	0.9718	0.9614	0.9757	0.9687	0.9618
S	0.0020	0.0000	0.0022	0.0000	0.0000	0.0000	0.0000	0.0018	0.0000
BA	0.0000	0.0020	0.0022	0.0000	0.0022	0.0000	0.0025	0.0000	0.0000
SR	0.0052	0.0034	0.0155	0.0030	0.0083	0.0065	0.0208	0.0094	0.0103
CA	0.9683	0.9775	0.9882	1.0985	0.9854	1.0129	0.9851	1.0104	0.9527
TI	0.0000	0.0027	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
MN	0.0466	0.0755	0.0000	0.0184	0.0112	0.0317	0.0006	0.0105	0.0519
MG	0.8957	0.7911	0.9905	0.8800	0.9672	0.9401	0.9894	0.9577	0.8728
LA	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0003	0.0000	0.0005
FE	0.0804	0.1463	0.0000	0.0000	0.0247	0.0085	0.0003	0.0091	0.1117
CE	0.0004	0.0005	0.0000	0.0000	0.0007	0.0001	0.0008	0.0000	0.0000
	1.9987	1.9991	1.9987	1.9999	1.9998	1.9999	1.9997	1.9989	1.9998

	38	39	40	41	42	43
S	0.0000	0.0005	0.0000	0.0001	0.0000	0.0018
BA	0.0012	0.0037	0.0020	0.0000	0.0006	0.0000
SR	0.0080	0.0057	0.0075	0.0064	0.0057	0.0063
CA	0.5295	0.5554	0.5200	0.5190	0.4969	0.4853
TI	0.0000	0.0001	0.0000	0.0000	0.0000	0.0008
MN	0.0025	0.0036	0.0062	0.0035	0.0373	0.0345
MG	0.4332	0.3951	0.4108	0.4392	0.3521	0.3581
LA	0.0000	0.0000	0.0012	0.0000	0.0000	0.0000
FE	0.0057	0.0094	0.0171	0.0047	0.0696	0.0743
CE	0.0000	0.0000	0.0003	0.0000	0.0000	0.0001
	0.9802	0.9734	0.9652	0.9728	0.9622	0.9610
S	0.0000	0.0008	0.0000	0.0001	0.0000	0.0028
BA	0.0012	0.0036	0.0020	0.0000	0.0006	0.0000
SR	0.0103	0.0075	0.0098	0.0082	0.0077	0.0084
CA	1.0019	1.0659	1.0049	0.9867	0.9823	0.9592
TI	0.0000	0.0002	0.0000	0.0000	0.0000	0.0012
MN	0.0041	0.0059	0.0105	0.0057	0.0643	0.0593
MG	0.9731	0.9000	0.9425	0.9913	0.8262	0.8401
LA	0.0000	0.0000	0.0011	0.0000	0.0000	0.0000
FE	0.0093	0.0155	0.0286	0.0077	0.1188	0.1268
CE	0.0000	0.0000	0.0002	0.0000	0.0000	0.0001
	1.9999	1.9994	1.9997	1.9998	1.9999	1.9980

AVERAGE FOR 15 ANALYSIS

S	0.0004
BA	0.0011
SR	0.0068
CA	0.5116
TI	0.0002
MN	0.0154
MG	0.3964
LA	0.0001
FE	0.0269
CE	0.0002
	0.9591
S	0.0007
BA	0.0011
SR	0.0090
CA	0.9987
TI	0.0003
MN	0.0264
MG	0.9172
LA	0.0001
FE	0.0458
CE	0.0002
	1.9994

Tableau 3 : Analyses à la microsonde électronique des dolomites. La colonne du haut correspond aux pourcentages en poids de carbonates (e.g. FeCO3).

	8	9	10	11	27	28	37	AVERAGE FOR 7 ANALYSIS	
S	0.0000	0.0008	0.0001	0.0015	0.0000	0.0000	0.0003	S	0.0004
BA	0.0016	0.0001	0.0035	0.0032	0.0015	0.0032	0.0021	BA	0.0022
SR	0.9603	0.9818	0.9646	0.9706	0.9669	0.9744	0.9608	SR	0.9685
CA	0.0240	0.0202	0.0295	0.0228	0.0293	0.0316	0.0229	CA	0.0258
TI	0.0009	0.0019	0.0000	0.0001	0.0000	0.0000	0.0000	TI	0.0004
MN	0.0000	0.0000	0.0010	0.0000	0.0004	0.0000	0.0000	MN	0.0002
MG	0.0002	0.0000	0.0000	0.0006	0.0000	0.0000	0.0000	MG	0.0001
LA	0.0000	0.0000	0.0000	0.0000	0.0000	0.0008	0.0000	LA	0.0001
FE	0.0000	0.0000	0.0000	0.0000	0.0000	0.0008	0.0000	FE	0.0001
CE	0.0000	0.0000	0.0000	0.0000	0.0029	0.0000	0.0007	CE	0.0005
	0.9871	1.0048	0.9986	0.9990	1.0009	1.0107	0.9867		0.9983
S	0.0000	0.0019	0.0002	0.0036	0.0000	0.0000	0.0006	S	0.0009
BA	0.0024	0.0001	0.0052	0.0048	0.0021	0.0046	0.0031	BA	0.0032
SR	1.9230	1.9326	1.9060	1.9204	1.9068	1.9010	1.9270	SR	1.9167
CA	0.0710	0.0585	0.0860	0.0667	0.0853	0.0910	0.0677	CA	0.0752
TI	0.0022	0.0045	0.0000	0.0002	0.0000	0.0000	0.0000	TI	0.0010
MN	0.0000	0.0000	0.0024	0.0000	0.0011	0.0000	0.0000	MN	0.0005
MG	0.0006	0.0000	0.0000	0.0022	0.0000	0.0000	0.0000	MG	0.0004
LA	0.0000	0.0000	0.0000	0.0000	0.0000	0.0011	0.0000	LA	0.0002
FE	0.0000	0.0000	0.0000	0.0000	0.0000	0.0020	0.0000	FE	0.0003
CE	0.0000	0.0000	0.0000	0.0000	0.0040	0.0000	0.0010	CE	0.0007
	1.9993	1.9976	1.9998	1.9979	1.9993	1.9997	1.9994		1.9990

Tableau 4 : Analyses à la microsonde électronique des strontianites. La colonne du haut correspond aux pourcentages en poids de carbonates (e.g. FeCO3).

Sidérites

	2
S	0.0000
BA	0.0026
SR	0.0005
CA	0.0855
TI	0.0000
MN	0.0577
MG	0.0750
LA	0.0000
FE	0.8676
CE	0.0000
	1.0889
S	0.0000
BA	0.0027
SR	0.0006
CA	0.1752
TI	0.0000
MN	0.1030
MG	0.1824
LA	0.0000
FE	1.5359
CE	0.0000
	1.9999

Calcites

	16
S	0.0005
BA	0.0012
SR	0.0539
CA	0.8298
TI	0.0000
MN	0.0000
MG	0.0000
LA	0.0043
FE	0.0659
CE	0.0072
	0.9628
S	0.0009
BA	0.0013
SR	0.0785
CA	1.7830
TI	0.0000
MN	0.0000
MG	0.0000
LA	0.0044
FE	0.1223
CE	0.0075
	1.9978

Tableau 5a et b : Analyses à la microsonde électronique d'une sidérite et d'une calcite. La colonne du haut correspond aux pourcentages en poids de carbonates (e.g. FeCO3).

	18	19	20	21	44	45	46	AVERAGE FOR 7 ANALYSIS	
S	0.3491	0.3435	0.3430	0.3442	0.3402	0.3327	0.3475	S	0.3429
BA	0.6410	0.6465	0.6496	0.6641	0.6581	0.6692	0.6485	BA	0.6538
SR	0.0063	0.0064	0.0074	0.0019	0.0002	0.0009	0.0000	SR	0.0033
CA	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	CA	0.0000
TI	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	TI	0.0000
MN	0.0000	0.0003	0.0000	0.0009	0.0000	0.0000	0.0007	MN	0.0003
MG	0.0000	0.0001	0.0000	0.0000	0.0000	0.0000	0.0000	MG	0.0000
LA	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	LA	0.0000
FE	0.0014	0.0001	0.0003	0.0000	0.0000	0.0000	0.0000	FE	0.0002
CE	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	CE	0.0000
	0.9977	0.9970	1.0003	1.0111	0.9986	1.0028	0.9966		1.0006
S	2.0113	2.0002	1.9967	1.9926	1.9946	1.9741	2.0116	S	1.9973
BA	1.9285	1.9658	1.9744	2.0072	2.0146	2.0730	1.9602	BA	1.9891
SR	0.0281	0.0290	0.0332	0.0085	0.0010	0.0042	0.0000	SR	0.0149
CA	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	CA	0.0000
TI	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	TI	0.0000
MN	0.0000	0.0018	0.0000	0.0059	0.0000	0.0000	0.0045	MN	0.0017
MG	0.0000	0.0017	0.0000	0.0000	0.0000	0.0000	0.0000	MG	0.0002
LA	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	LA	0.0000
FE	0.0088	0.0005	0.0017	0.0000	0.0000	0.0000	0.0000	FE	0.0016
CE	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	CE	0.0000
	3.9768	3.9990	4.0060	4.0142	4.0103	4.0513	3.9763		4.0049

Tableau 7: Analyses à la microsonde électronique des barytines. La colonne du haut correspond aux pourcentages en poids d'oxydes. La colonne du bas correspond aux pourcentages atomiques calculés sur la base de 8 atomes d'oxygène.

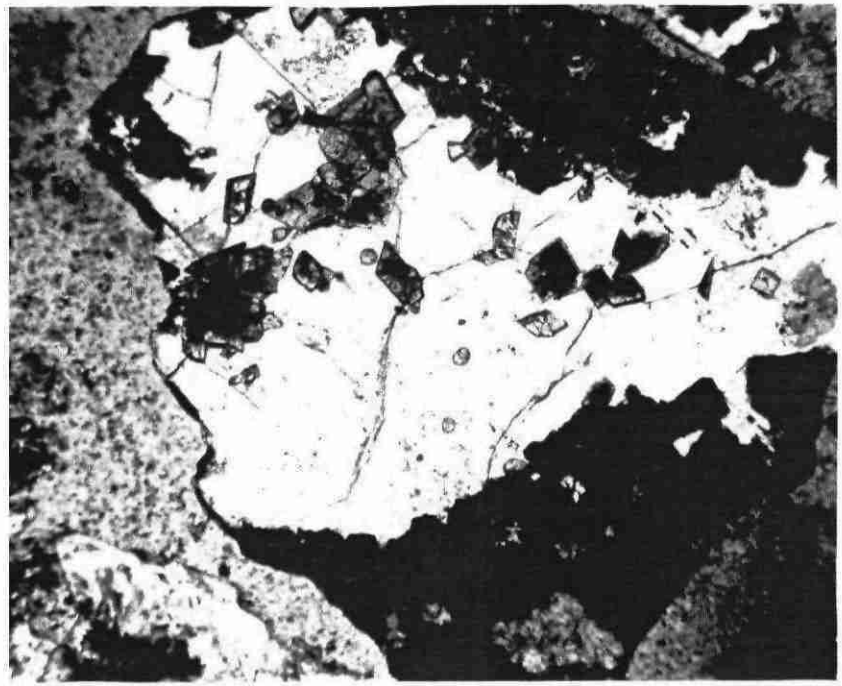
Legende de la planche photographique

Photo 1 : Inclusions de cristaux de monazites automorphes dans une strontianite, les phases opaques en periphérie de la strontianite sont des sidérites mélangées d'oxydes de fer (Photographie au microscope pétrographique X63)

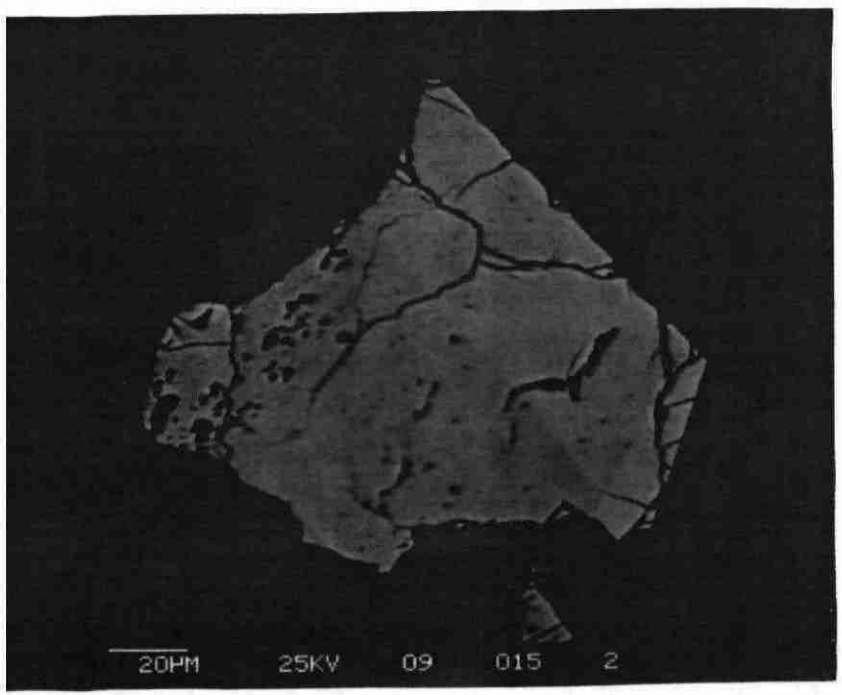
Photo 2 : Fragment d'un cristal de monazite, on observe une zonation vers le bord supérieur droit (zone plus claire) qui correspond a un enrichissement en cerium par rapport au lanthane (Photographie MEB).

Photo 3 : Grand cristal de strontianite (gris moyen), avec au centre une inclusion de dolomite (gris foncé), et deux inclusions de monazite (blanc), sur le bord supérieur droit on observe une grande plage blanche de barytine (photographie MEB).

Photo 4 : Gerbes d'un carbonate (probable) de terres rares (bastnaesite ou parisite), en inclusion dans une calcite (photographie MEB).

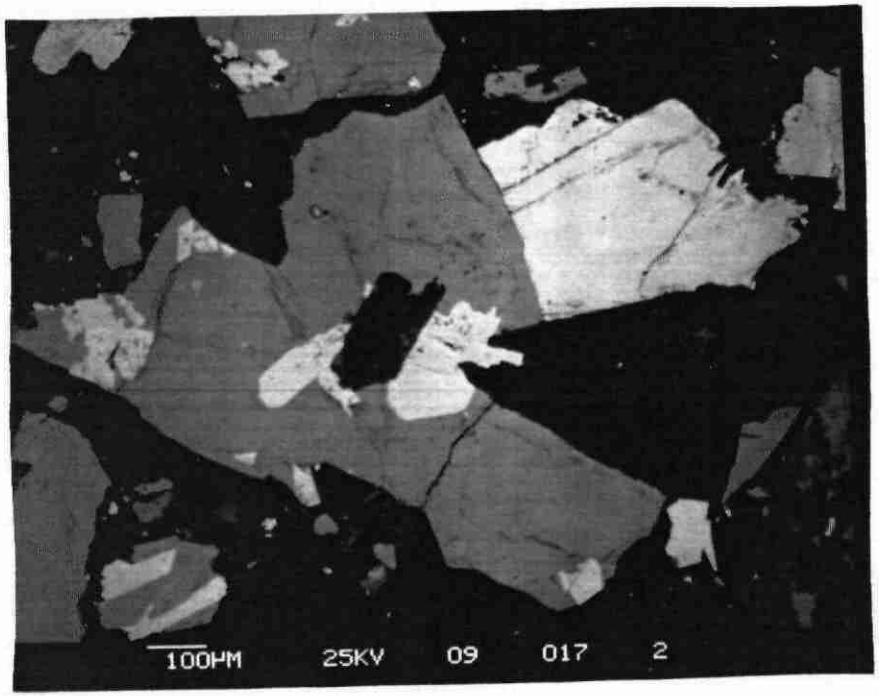


1



2

3



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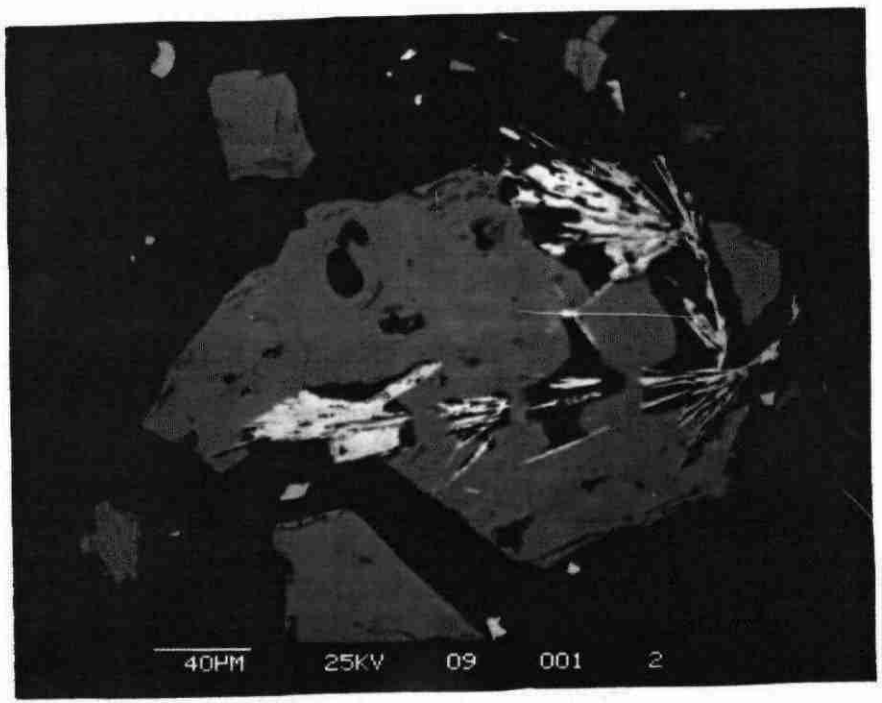

A P P E N D I X 3

Characterization studies on the RED ore sample 3 by Dr. G. MORTEANI (T.U.M.)

Contents:

1. Introduction

An extensive fluorite, baryte, and bastnaesite mineralization was discovered in 1977 in the neighbourhood of Kizilcaören (approx. 80 km east of Eskisehir and 100 km west of Ankara, see fig. 1).
This discovery was due to the detection of a radiometric thorium anomaly by a countrywide exploration for radioactive minerals carried out by the MTA (in translation: the Mineral Research and Exploration Institute of Turkey, Ankara).
All important data published up to now, including 1987, were produced by the MTA.

There are only two minerals known which are economically viable for the economic production of rare earth elements: monazite and bastnaesite. Since 1949 the USA has been the largest producer of bastnaesite and light REE on world scale, from the deposit of Mountain Pass (Calif.). The deposit of Kizilcaören reveals mineralization strikingly similar to that of Mountain Pass in the Sierra Nevada Mountains deposit (see below).

26 samples from the deposit near Kizilcaören (fig.1, tab. 1) were investigated by microscopy, x-ray fluorescence analysis, and, in same cases, more detailed examination of individual ore grains under the S.E.M..

As many samples resulted to be intensely altered, it proved impossible to prepare thin sections of all of them.
Most of the samples have been analysed using x-ray fluorescence (see appendix, pp. C,D).

Particular analytical care was taken for the sample "TU". This sample is a bulk composite ore sample of 72 kg.
This sample should represent the typical REE-ore of the Kizilcaören district and on this sample the flotation experiments at BRGM have to be performed.

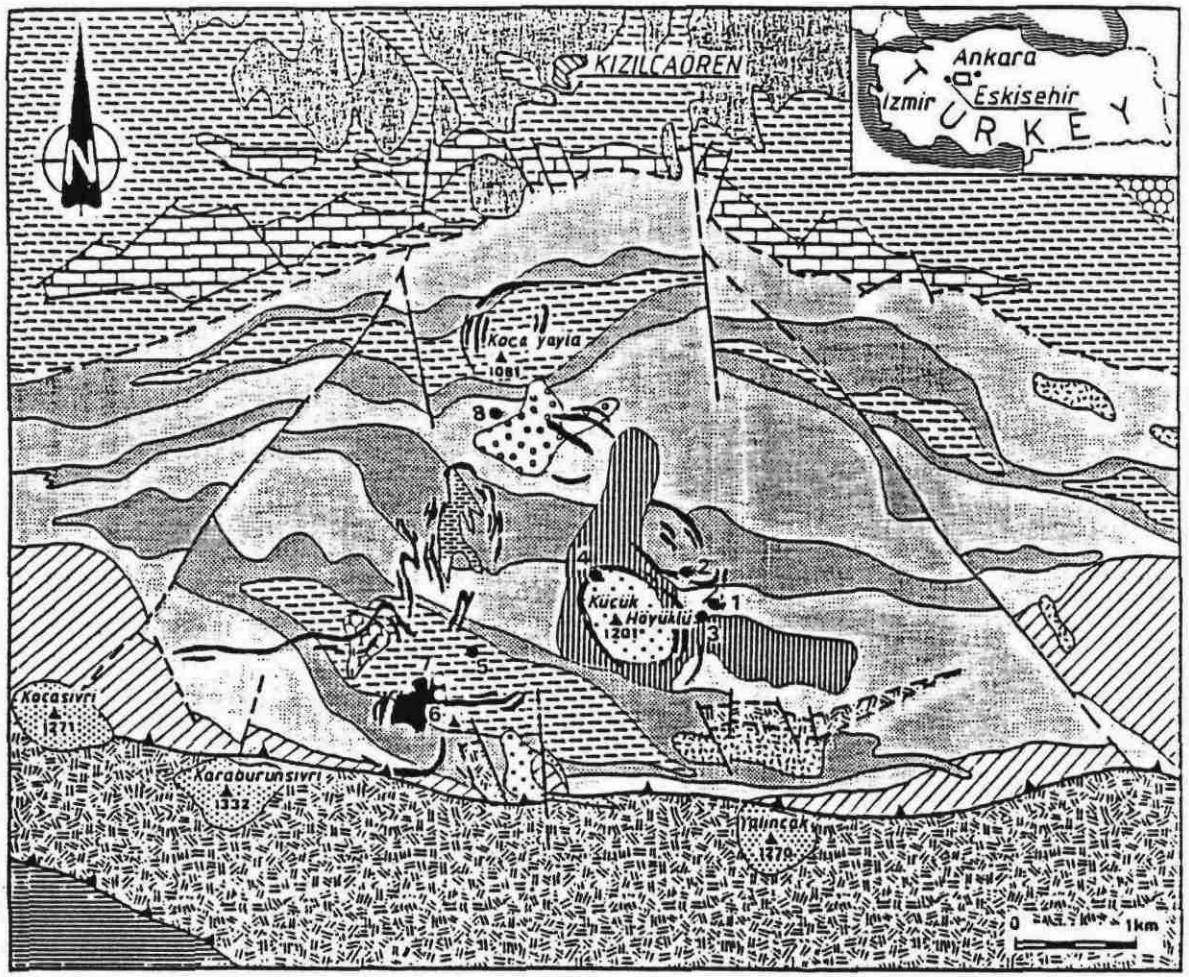

Fig 1: Geological map of the area showing sample localities (1 - 8) - see also fig.2. Sample numbers and adit names corresponding to localities 1 - 8 are listed in table 1.

Tab. 1: Lokality	Sample numbers	adit
1	7025,30,31,32,33,34,36,37	HG4
2	7041,52,53	HG2
3	7038,39,40,54	HG3
4	7042,43	HG5
5	7045,48,49,50	adit 6
6	7044,46,55	G1
7	7047	G1
8	7051	Kocayayla

2. Geology

The geological map and stratigraphic column shown in fig. 2 were produced by detailed mapping of the area containing the radiometric anomaly by KAPLAN (1977), KULAKSIZ (1977), and DELALOYE & ÖZGENC (1983), supplemented by our investigations.
The basement consists of lower Palaeozoic anchimetamorphic rocks e.g. greywackes, sandstones, phyllites, glaucophane schists, calcareous schists, and marbles (KUPFAHL 1954, WEINGART 1954).

There is a tectonic contact between the basement and the overlying Kizilcaören Formation, which is made up of pelites, sandstones, mudstones, and arenites and intruded by diorites and spilites.

Imbricated Triassic meta-ophiolites are situated between the basement and the Kizilcaören Formation. These consist of intensely serpentinised peridotites and minor pyroxenites - the extent of serpentinisation increases from the South to the North of the area.

The area was subsequently overlain by transgressive Jurassic conglomerates and fossiliferous limestones.

Volcanic activity dominated the Tertiary, producing phonolites, trachytes, tuffs, agglomerates and volcanic breccias. The phonolites form mountain peaks up to 1332 m a.s.l. in the southwestern sector of the area. The trachytic dykes are observed mainly in the Kizilcaören Formation.

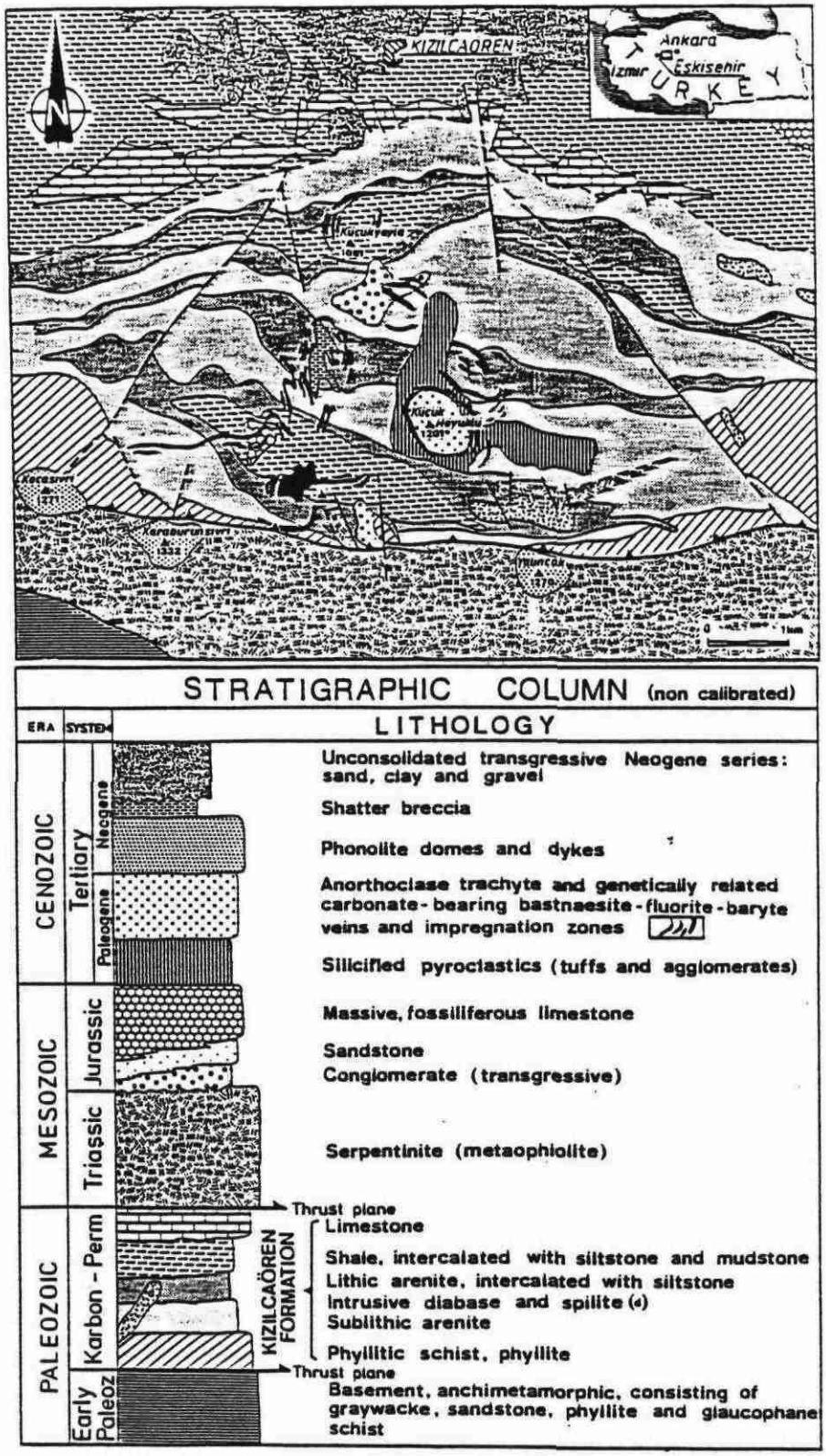

Fig 2: Geological map of the investigated area near Kizilcaören. The lithologies are represented in the accompanying stratigraphic column.

The carbonate dykes and the fluorite-baryte-REE-mineralisation are probably genetically associated with the volcanic rocks.

The youngest sequence in the area consists of the transgressive Neogene sandstones, pebbly sandstones and conglomerates.

The entire area belongs to the Izmir - Ankara Ophiolite Zone (BRINKMANN 1966), which is situated between the Anatolian - Taurian Zone in the south, and the Pontic Zone to the north.

It represents the remains of a lower Palaeozoic - Triassic ocean, which closed during the upper Palaeocene - late Tertiary along a north dipping subduction zone (SENGÖR 1979, SENGÖR & YILMAZ 1981).

3. Mineralisation

3.1. Structures

The deposits occur in the form of layers and lenses, impregnation zones, brecciated zones up to 40 m in thickness, and veins (up to 20 m thick), which all cut the Kizilcaören Formation.
The veins are oriented for the main part either NW-SE or NE-SW, according to STUMPFL & KIRIKOGLU (1985).
The deposits are mainly associated with the trachytes and phonolites, which cut the Kizilcaören Formation. The vein ores exhibit a striking tended structure.

3.2. Ores

The macroscopical and the microscopical studies of the ores have shown that there are at least two important types of REE ores in the Kizilcaören District. The most important one is the fluorite-baryte-dominated REE-ore. The second, subordinate type is a REE-ore with carbonate matrix. The carbonate ores have a more massive, veined character, and the fluorite-baryte-REE-minerals - ores mostly have a banded structure.

3.2.1. Fluorite-baryte-REE-minerals - ores
(sample nos.: 7030-7050,7053-7056)

Macroscopically, the ores have either banded or schlieren texture, which is caused by alternation, on a mm- or cm-scale, between fluorite-rich layers which are often purplish in colour, and schlieren or lenses of light-coloured baryte-rich material.
Occasional Fe-Mn-oxide layers and REE-mineral layers also contribute to the banded apperance of the ores.
The bands may be "cross-bedded", gently folded or faulted.
The banding has been destroyed in places, by recristallization and deformation processes, causing the rocks to appear massive in hand specimen.

Dark purple fluorite cristals up to 2 cm in diameter, which have been cataclastically deformed, are conspicuous - these are usually concentrated in layers, and only seldom randomly scattered throughout the ore.

The colour of the ores depends on the proportions of the ore minerals listed above, and ranges from pale beige/brown to dark purple.

Mineral contents:

Fluorite:	15 - 60 %
Baryte:	10 - 50 %
Bastnaesite:	up to 20 %
Fe-Mn oxides:	up to 10 %
White mica and phlogopite:	up to 15 %
Calcite, dolomite:	up to 20 %

Chlorite, quartz, chalcedony and other REE-minerals: up to 3%

Fluorite determines the texture of the ore. It can be easily seen under the microscope by its isotropism, purple colour, and zoning (fig.3). There are at least three generations of fluorite:

i) The oldest generation consists of the cataclastic deformed grains (up to 2 cm in diam.) mentioned above (fig.3 and 4). This generation usually exhibits an extensive, zoned purple colour.

ii) During a second phase of mineralisation following a tectonism the older generation was impregnated starting from ruptures by a very fine-grained generation of fluorite (with associated barytes) (fig.4). Its colour is much less extensive, and ranges from colourless through yellowish pink to pale purple. Individual cristals can hardly be distinguished, as they are at the most 5-10 µm in diameter.

iii) In the youngest phase of mineralisation colourless fluorite cristallized, probably in tiny pore spaces (up to 1 mm diameter), along with chlorite and REE-minerals (fig.5).

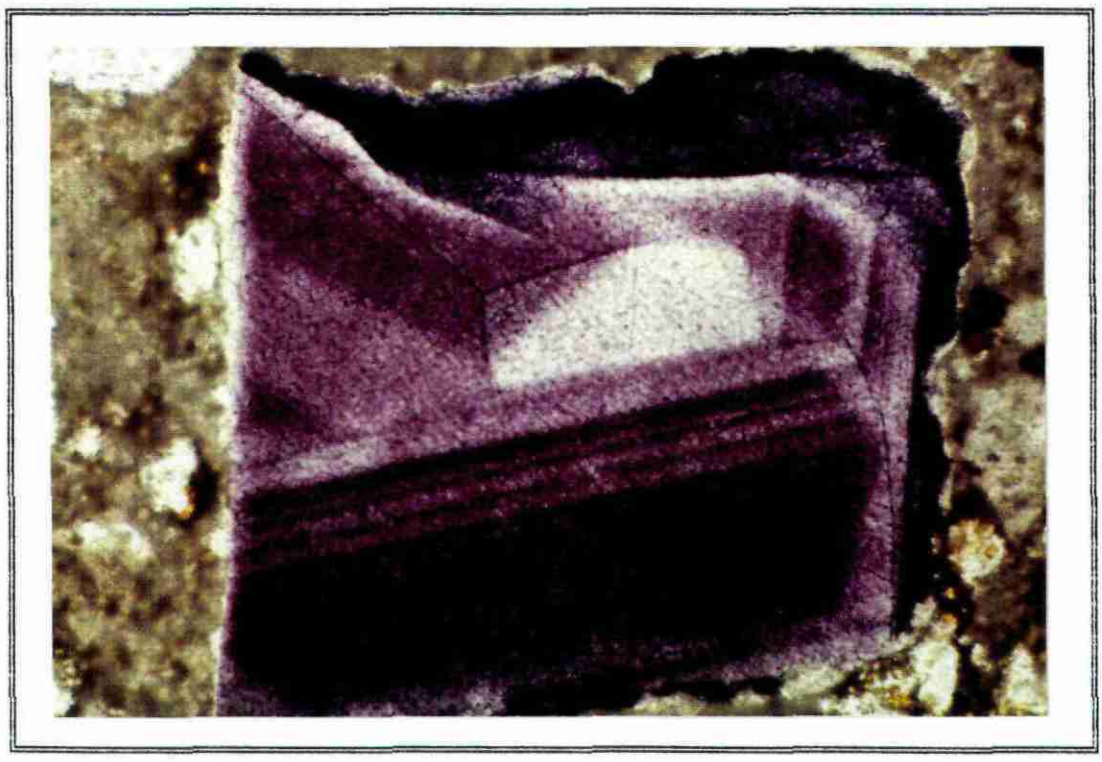

Fig. 3: Broken zoned fluorite crystal
(sample no.: 7037)

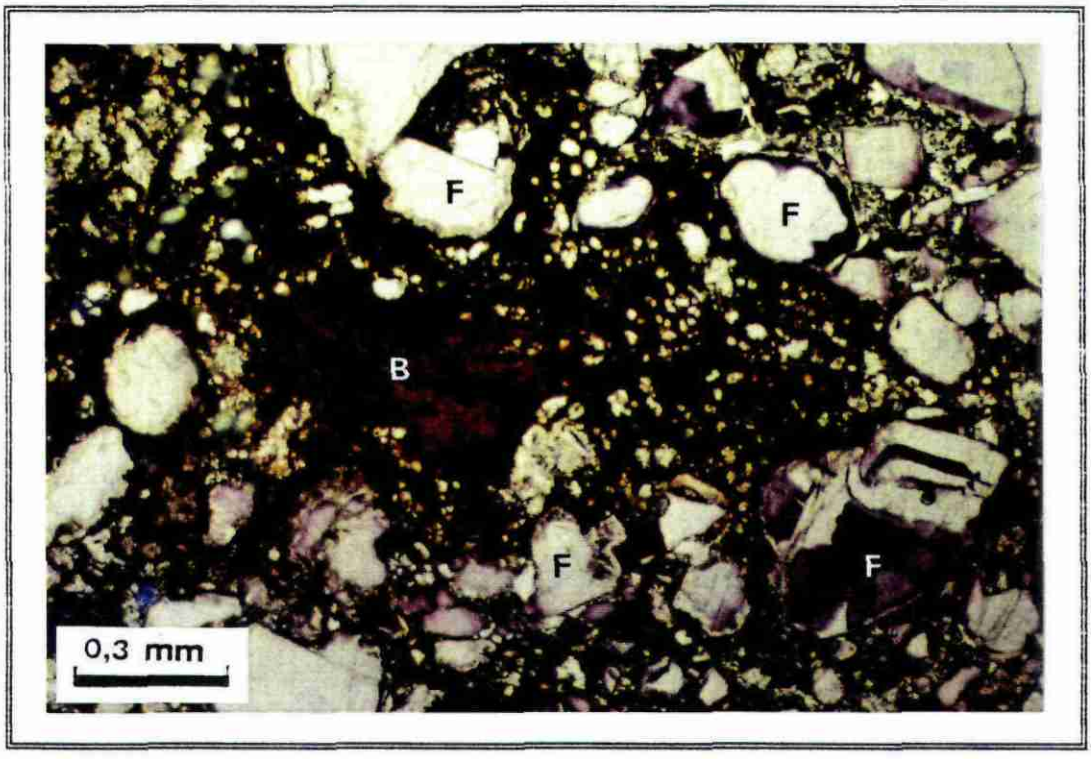

Fig. 4: Large cataclastically deformed fluorite grains (F) in an extremely finegrained fluorite-baryte matrix, with brown bastnaesite, which is also deformed (B). (sample no.: 7032)

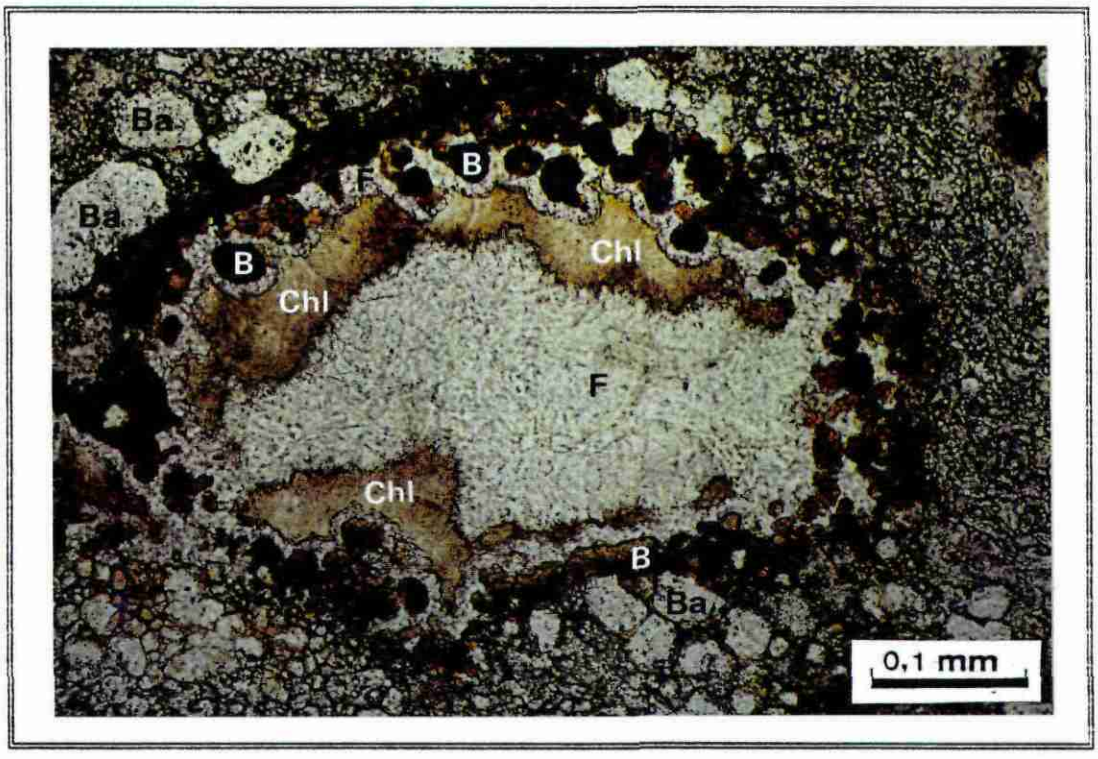

Fig. 5: Late pore-filling, consisting of bastnaesite (B), fluorite (F) and chlorite (Chl) in a massive baryte-fluorite ore. (sample no.: 7041)

Baryte occurs, like fluorite, in at least three generations. In the mineralisation sequence described above, the first baryte generation corresponds to the first fluorite generation (fig.6). It occurs in the form of largish (up to 1 cm diam.) round cataclastically deformed crystals.

The second generation is also equivalent to the second fluorite generation and is associated with it as a very fine grained matrix component. This second generation is volumetrically the most important.

The third generation is relatively rare in comparison to the third generation of fluorite, and is usually found in the "carbonate ores" (see 3.2.2.).

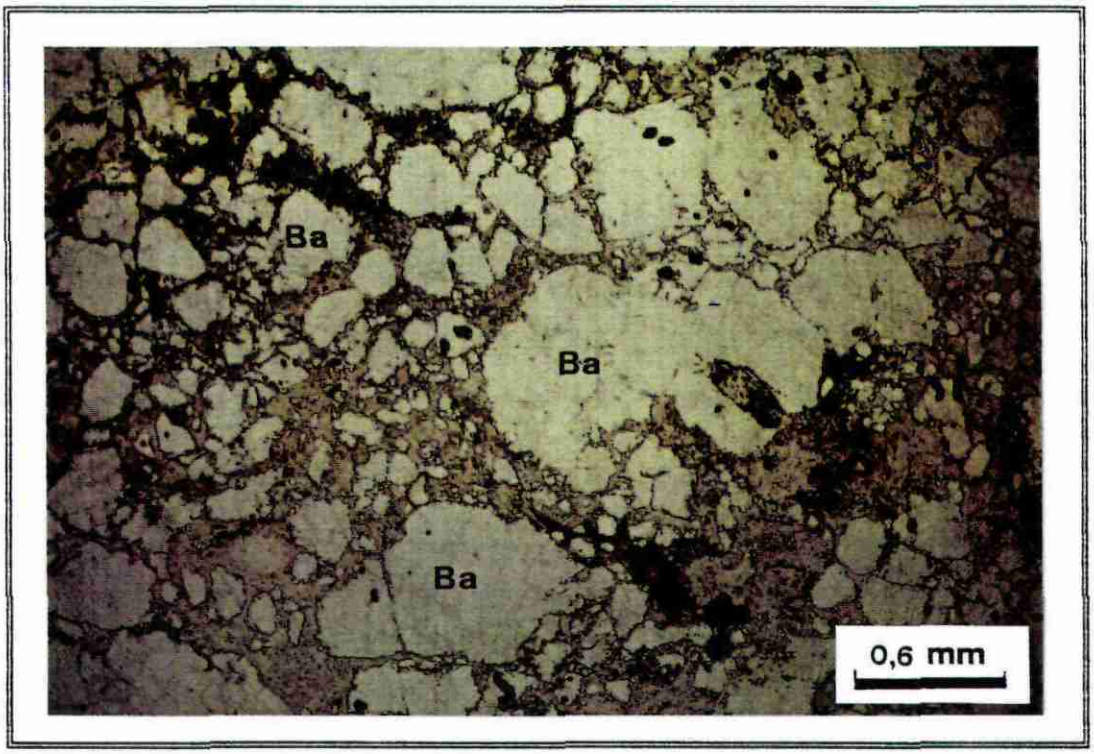

Fig. 6: Large cataclastically deformed baryte cristals (Ba) in a very finegrained fluorite matrix (sample no.:7041)

As the ore grains of bastnaesite and other REE-minerals are so tiny it is very difficult and often impossible to distinguish between the various REE-minerals. It is also difficult to tell them apart from the very finegrained iron oxides, as they sometimes occure together in aggregates. Therefore in selected thin sections REE mineral composition was investigated qualitatively using the S.E.M. (see 3.5).

Bastnaesite occurs predominantly in extremely finegrained brownish felty aggregates (fig. 4 and 7), which are up to 1 cm long and 0.5 cm wide. In places, up to 20 % of the ore may be made up of these bastnaesite schlieren. As rectangular relics have been observed, it is thought that these aggregates represent old deformed and later on recristallized and bastnaesite grains.

Round grains also occur in pores, associated with fluorite and chlorite (fig. 5 and 8). From the fact that the X-ray fluorescence analyses, and several S.E.M. analyses indicate that the bastnaesite contains thorium it can be assumed that the bastnaesite is a thoro-bastnaesite. Microscopically, in this ores, it is not possible to distinguish thoro-bastnaesite and bastnaesite, which is free of thorium.

As revealed by S.E.M. analyses (see 3.4.) there are some REE-minerals, which cannot be distinguished microscopically from bastnaesite, containing REE, Ca, P, Sr, Si, Al and Th. as reveale. According to STUMPFL & KIRKOGLU (1985), the ores contain accessory brockite ($CaTh(PO_4)*H_2O$), florencite ($CeAl_3((OH)_6/(PO_4)_2)$), and monazite ($CePO_4$). This problem requires some additional effort.

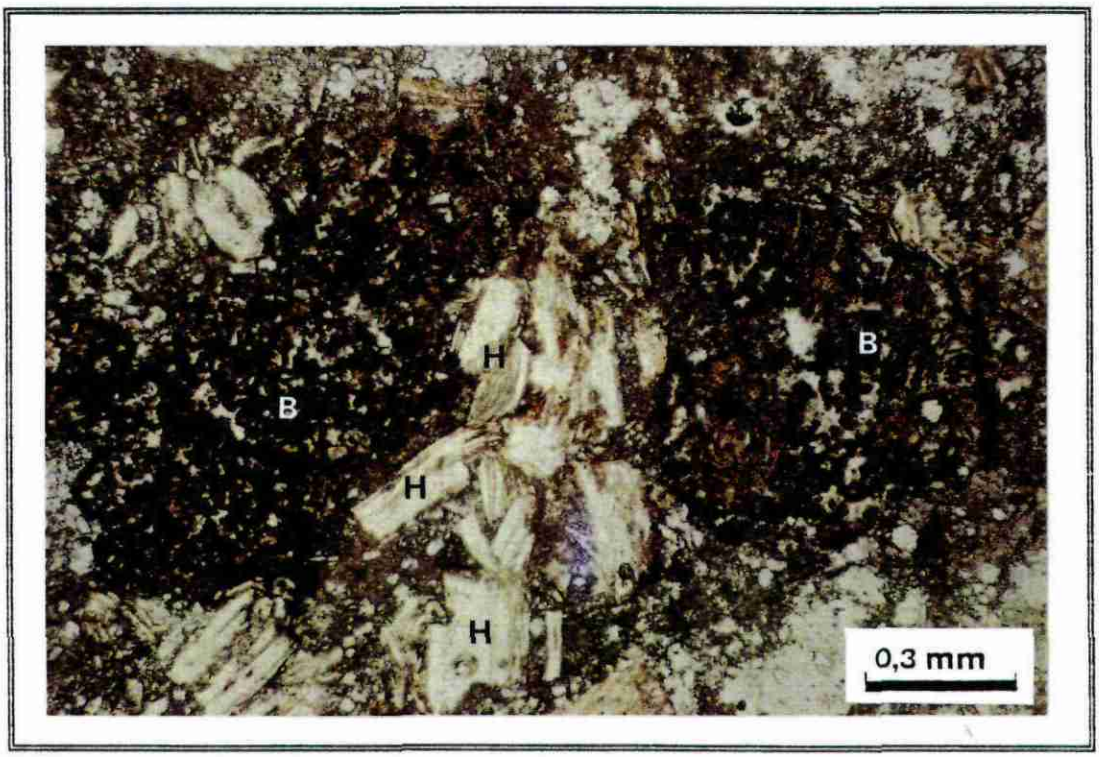

Fig. 7: Felty aggregates of bastnaesite (B),set in a matrix of flattened deformed white mica grains (H). (sample no.: 7041)

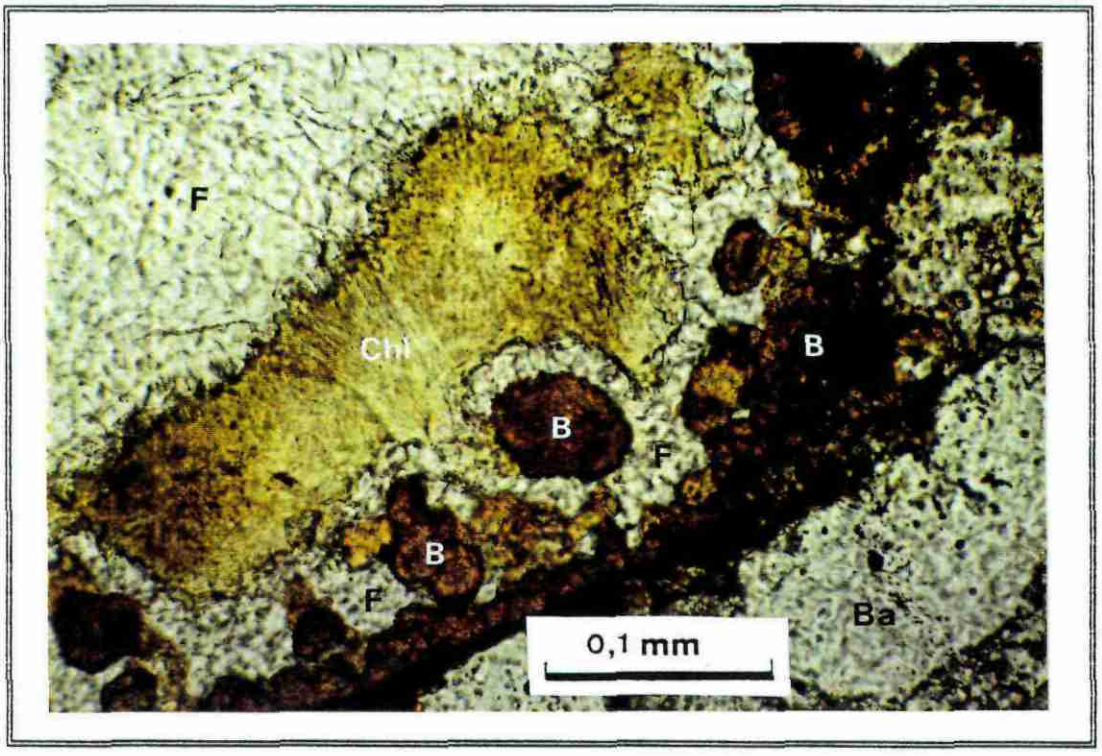

Fig. 8: A detail of fig. 5: small spherical grains of bastnaesite (B) which contain Ca, Sr, and Th. These are overgrown by fluorite (F). Chlorite (Chl) and fluorite crystallized later. (sample no.: 7041)

The white mica and phlogopite grains may achieve lengths up to 2 mm parallel to the cristallographic c-axis (on average approx. 0.1 - 0.3 mm). The lath shaped crystals are either scattered throughout the ore or concentrated in clusters or layers (fig. 9). Many of the mica grains have been deformed, as revealed by rounded corners, bent and broken grains (fig. 7). This deformation is presumably the same which led to cataclasis of the fluorite and baryte, which means that the micas must also belong to the first phase of mineralization.

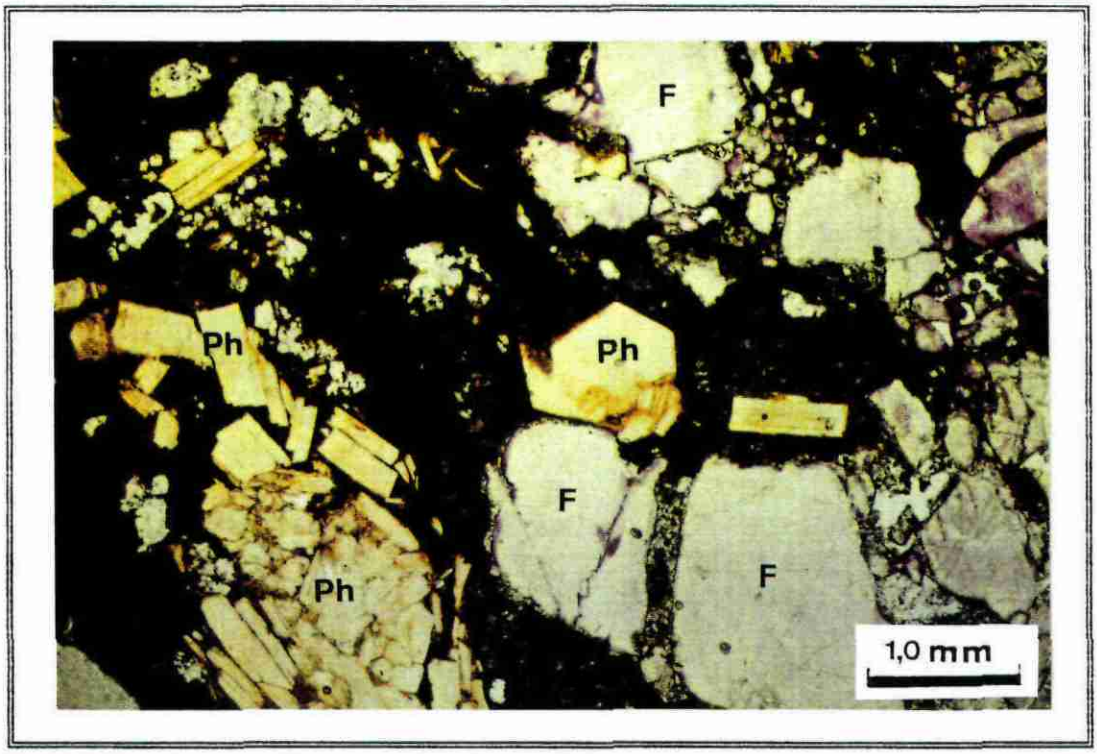

Fig. 9: Clusters of phlogopite (Ph) in dark FeOOH-REE-mineral matrix, with pale purple cataclastically deformed fluorite cristals (F). (sample no.: 7037)

Calcite and dolomite occur rarely as a pore filling, in aggregates or in secondary veins. Calcite rarely exceeds 5 %, and is not present at all in some samples.

Fe-Mn oxides are often concentrated in diffuse aggregates, lenses or layers. Form relics of pyrite cubes (up to 2 cm diam.) are rare (fig. 10).

Sample 7043 contains almost 50 % Fe-Mn oxides and is the only sample to contain significant amounts of quartz or chalcedony.

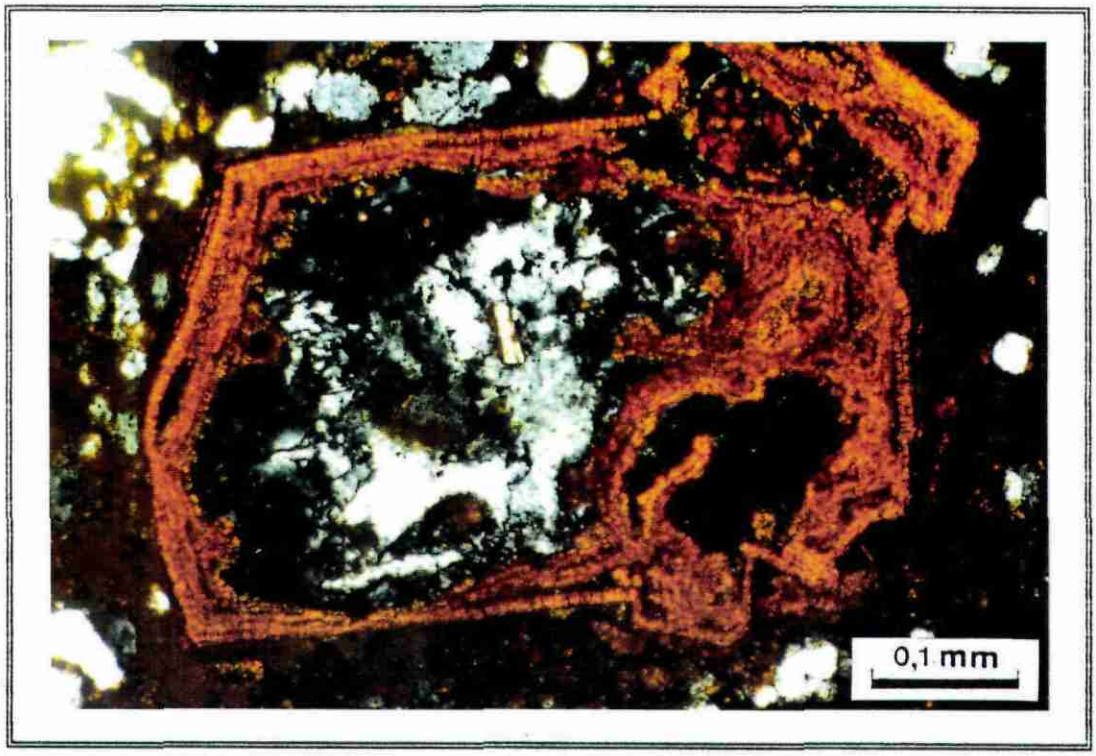

Fig. 10: Probable form relic of pyrite, now limonitized.

The quartz mentioned above crystallized in finegrained lenses and, as chalcedony, in tiny pore spaces.
As its relief is similar to that of baryte, tiny grains of quartz may easily escape notice.

Finally, an iron-rich chloride has been observed, which occurs in the above-mentioned pore spaces in association with fluorite and REE-minerals (Fig. 5).

Sample 7054 is exceptional in that it contains several serpentinite clasts, which must have been transported by the hydrothermal fluid as passes through the ophiolite units.

3.2.2. Carbonate ores

(Sample Nos.: 7025, 7051 and 7052)

These ores are massive and irregular in texture, in contrast to the ores described in chapt. 3.2.1..
Broken crystals of baryte and flourite (up to 0.5 cm in diam.) are enclosed in a carbonate matrix. Texturally, the ore is a breccia. The fluorite crystals are usually colourless, but occasionally pale purple. Brownish patches suggest the presence of Fe-Mn oxides and bastnaesite.

Mineral content:	calcite, dolomite:	40 - 50 %
	fluorite:	15 - 25 %
	baryte:	15 - 20 %
	bastnaesite:	up to 15 %

White micas and phlogopite, Fe-Mn oxides and the other REE-minerals: up to 3 %

The calcite (and dolomite) (0.1 - 0.5 mm in diam.) occur in particular as matrix components, in a "brecciated" irregular texture formed by fluorite, baryte and bastnaesite. This generation of calcite seems very dusty, because it contains many inclusions. A young, clear generation of calcite which is poor in inclusions is mainly found filling cracks in the broken baryte and fluorite grains.

Both the baryte and the usually colourless fluorite have been cataclastically deformed and are present as angular grains. The grainsize in both cases is approx. 0.2 - 1 mm in diam.. Grains of the 2nd and 3rd generation as described in 3.2.1. are usually lacking. Occasional fillings of tiny pores are an exception.
Only in sample 7025, containing only 10 % ore minerals (90 % calcite and dolomite), also subhedral baryte lining pore spaces was found (fig. 11).

The distinctively brown bastnaesite may exhibit rectangular or square form relics, but these are composed internally of felty aggregates (fig. 12).
Light and dark micas, in the form of laths up to 0.5 mm in length, and REE-phosphates, which cristallized in pore spaces, are present as accessory phases. Minor Fe-Mn oxides occur in diffuse patches in the matrix (up to 0.2 mm in diam.).

The above mentioned carbonatite sample (7025) also contains Sr, P, Ca-bearing and zoned REE-minerals (in particular Ce, La and Nd; fig. 13, see also chapt. 3.4.).

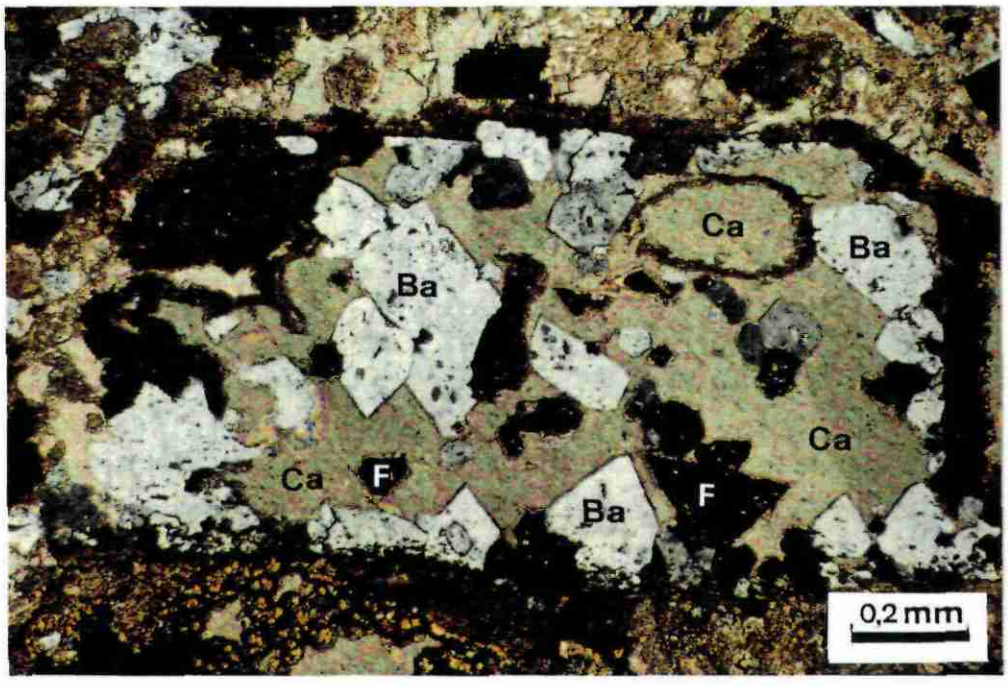

Fig. 11: Subhedral to euhedral barytes (Ba) which has cristallized, along with fluorite (F) and clear calcite (Ca), which is poor in inclusions, in a pore space during the 2nd phase of mineralization (sample no.: 7025).

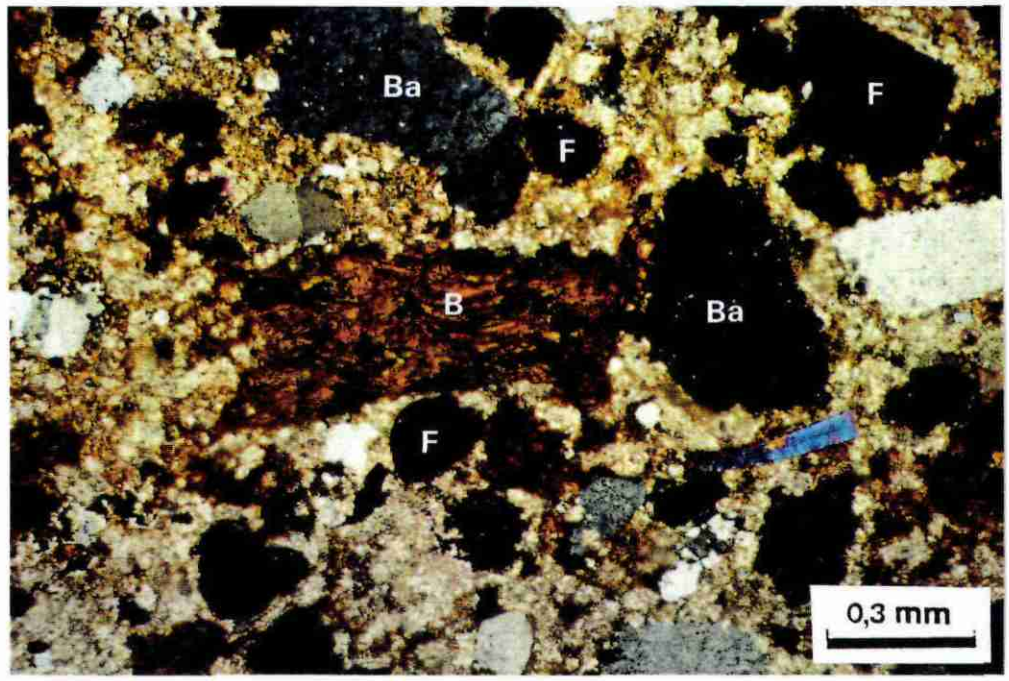

Fig. 12: Fine grained wisps recrystallized Ca-, Sr- and Th- bearing bastnaesite (B) , in association with baryte (Ba) and fluorite (F) in a coarsegrained calcite matrix. (sample no.: 7054)

Fig. 13: Zoned REE-minerals (yellowish), with dark cores which are very rich in Ce (possibly Ce-bastnaesite, fluocerite or cerium oxide) and paler P-Sr-rich margins (possibly belovite, see chap. 3.4.2).
An older inclusion-rich generation of calcite (Ca1) can be distinguished from a younger clear calcite generation (Ca2) in this "carbonatite" The opaque radial aggregates are probably composed of hollandite (Ba-Mn-oxide) as mentioned above. (sample no.: 7025).

3.3. X-ray fluorescence analysis

3.3.1. Introduction

Lists of analysis results and details of procedures (e.g. detection levels) are presented in the appendix (pp.C,D,E). The results are presented as oxide wt%, and ppm in the case of elements.
The elements are calculated as oxides for the total contents.

As the ore samples contain carbonates, the loss of ignition (GV) expresses not only the H_2O but olso the CO_2 content.

The total content of the XRF analyses is often below 100%, because of the high fluorine content (fluorite !), which cannot be measured with the XRF.

3.3.2. Results

3.3.2.1. Major elements or oxides

The main element contents confirm the microscopic analyses and reveal that CaO, which is a component of both the carbonate and the fluorite generations, is the only oxide to reach over 10 wt%. CaO reaches 30-40 wt% in most analyses.

All other main elements, with the exception of SiO_2 in samples 7040 and 7043, have weight %s less than 10 %.

MgO seldom achieves more than 1 wt%, and is only present in large amounts in the carbonatite ores (7051 a. 7052), with approx. 7 wt%. Microscopy has shown that this reflects the Mg contained in calcite and dolomite.

Samples 7040 and 7043 are the only ones to contain significant amounts of free quartz. The other SiO_2 contents (0.5-5 wt%) correspond roughly with the mica contents. The same goes for Al_2O_3 and K_2O.

The Fe_2O_3 contents range from 0.5 - 5 wt%, with the exception of sample 7043 with 19 wt%, as a result of the large amount of opaque components in the ore.

MnO is very variable - it ranges from <0.01 to 3 wt%.

TiO_2 varies similarly from 0.07 to 1.78 wt%.

The P_2O_5 content (0.15 - 4,51 wt%) is also interesting - S.E.M. investigations have shown that REEs may occur in complex phosphate minerals.
Finally the SO_4 content (up to 21 wt%) may be correlated with the Ba (up to 30 wt%) in barytes.

3.3.2.2. Trace elements

Several analysed elements (Rb, Zr, Co, Ni, and Cr) are either below the detection limit of XRF or barely above it. Cr is the only one of these which achieves values above 100 ppm. The La, Ce, Nd, Th, Y, and Sr contents are graphically represented in Fig.14.

Sr is present in barytes, REE-minerals and in fluorite, up to 9000 ppm (on average 3500 ppm). According to STUMPFL & KIRKOGLU (1985), strontzianite is present in the ores.

Zn is usually under 200 ppm, but several samples contain more than 500 ppm (the maximum value, of 682 ppm, was detected in sample 7048).

Nb is seldom present in amounts above the detection limit. However values of 695 and 411 ppm were measured , in samples 7037 and 7040 respectively.

La and Ce have the highest concentrations of all the REEs, up to almost 50000 ppm (5 wt.%). They are usually present in equal amounts. Differences up to 1 % are possible. Average contents of both Ce and La are approx. 2.5 wt%.

Nd and Y may be incorporated into the REE-minerals instead of Ce and La, but occur in concentrations which are one (Nd up to 7000 ppm) or two (Y up to 900 ppm) orders of magnitude smaller.

Th concentrations average are several 100 ppm, and reach up to 0.2 wt% in several samples (e.g. sample 7052). U is less important - it is usually below the detection limit, and only occasionally above 100 ppm.

Mo peaks have been observed in several samples, but exact values cannot be reported due to lack of standards (see also chap. 3.4).

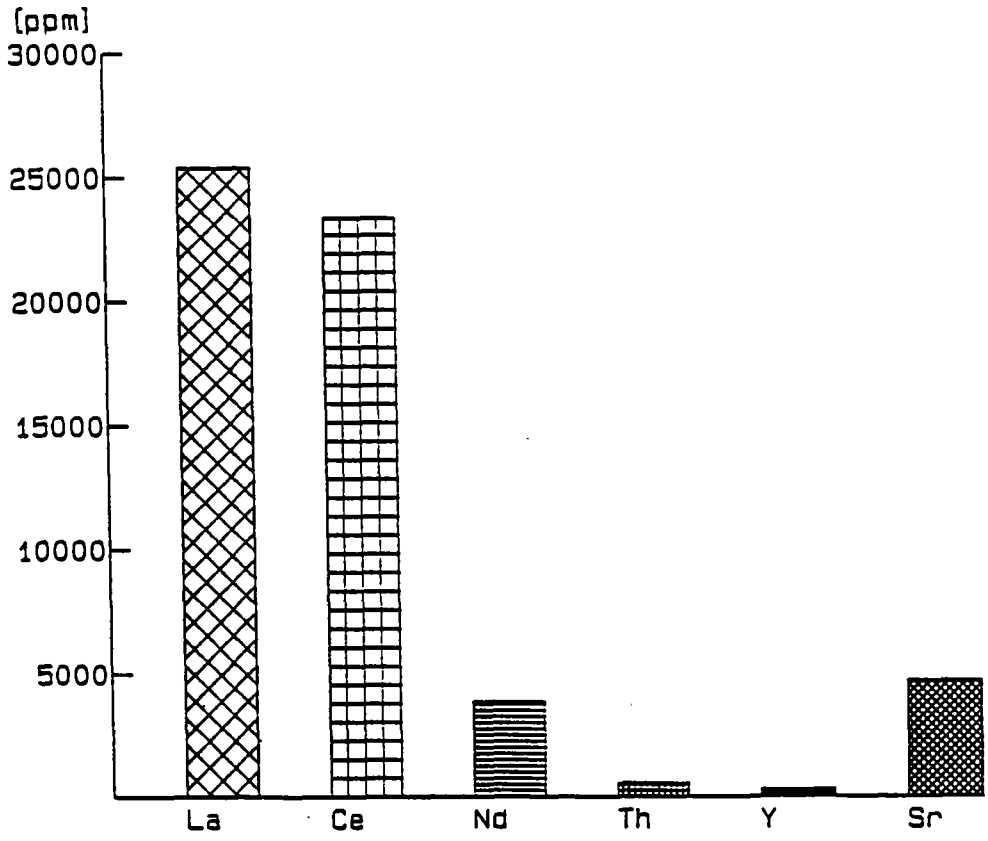

Fig. 14: Average concentrations of REEs and other trace elements in the analysed ores as analysed by XRF (in ppm)

3.4. Composite sample analysis

3.4.1. Introduction

In addition to the x-ray fluorescence analyses of different ore samples a full analysis (66 components) of a composite ore sample (TU) representative ore sample has been made, by ICP-MS, XRF, wet chemical analysis, FADCP, DCP, DCPA, FAA, and GFAA.

For this analysis a 72 kg ore sample (approx. 60% CaF_2, 20% $BaSO_4$ and 20% REE-minerals in macroscopic view) was reduced by hand to a 10 kg ore sample.
This representative sample (TU) was crushed and split in a sample divider, to give two 100 g ore samples (TU1 and TU2).
After milling, 50 g of each sample were taken for analysis.

The results of the full analysis with details of the methods of analysis and detection limits are given in the appendix (pp.B,E).
Reference analysis of 26 components by x-ray fluorescence were made of the same sample material to test the ICP-MS data. The XRF data are given in brackets in the appendix (p.B), too.

3.4.2. Results

3.4.2.1. Major elements

Since the bulk sample consists only of fluorite-baryte-REE-mineral ores (without carbonate; see also the analysis of CO_2) calcium is given in the appendix (p.B) as Ca.

The results for the major elements (more than 1 wt.%) are about identical to the x-ray fluorescence analyses discussed in chapter 3.3.:

SiO_2 (2 wt.%), Fe_2O_3 (3 wt.%), SO_4 (9 wt.%), Ba (12 wt.%), Ca (30 wt.%), F (35 wt.%), H_2O (1 wt.%), and CO_2 (2.4 wt.%).
Calculation of mineral stoichiometry shows that:

i) the CO_2 - content can be accomodated by bastnaesite, so that no significant amount of calcite or dolomite is in the sample.
ii) Ca reaches ca. 30 wt.% and because of i), it is probably contained in fluorite.

iii) the fluorine is not only a constituent of fluorite, but also of bastnaesite and other possible REE - minerals.

iiii) there are stoichiometric problems in the amounts of Ca and F. As shown in i) - iii) the F and Ca must be contained in bastnaesite and fluorite. However the measured F content is at least 5 wt.% too high (or Ca too low) to satisfy the mineral stoichiometry. We will check the Ca and F contents with a wet chemical determination of Ca to resolve this problem.

3.4.2.2. Trace elements

3.4.2.2.1. Rare earth elements

The ICP-MS analyses of the ore samples TU1 and TU2 agree very well with the XRF - analyses for the elements La, Ce and Nd (see appendix p.B).

La and Ce have the highest concentrations of all the REE with about 3,2 - 3,6 wt.%.

Pr (0,24 wt.%) and Nd (0,6 wt.%) are one order of magnetude smaller.

Sm (310 ppm) and Gd (118 ppm) are the next in sequence of absolute amounts.

REEs between 10 and 100 ppm are: Eu (61 ppm), Dy (60), Er (28), Yb (27), Tb (22), and Ho (11).
Tm (4,2 ppm) and Lu (3,7) are the REEs with the lowest absolute amounts.
All REEs together achieve nearly 8 wt.%.

Fig. 15 shows the REE distribution pattern of the ores in form of a chondrite-normalized diagram. The chondrite values used for normalization are from HANSON (1980) and MASUDA, NAKAMURA & TONAKA (1973).

The diagram shows for both samples an immense enrichment of the light REE and a much weaker enrichment of the heavy REE with respect to chondrite values.

This kind of REE distribution is typical for carbonatitic ores. There is essentially no negative Europium anomaly, which is a further important indication of carbonatitic development of the Kizilcaören ores.

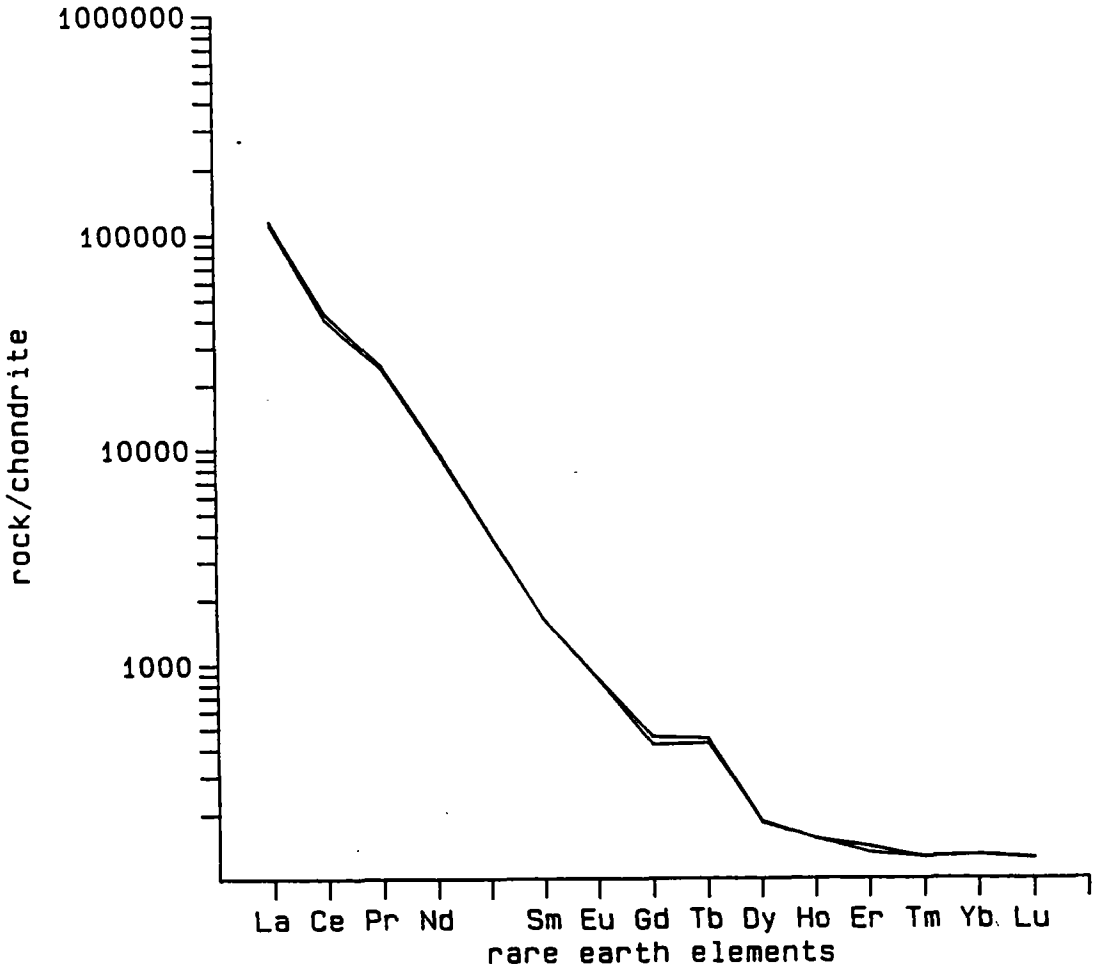

Fig. 15: Chondrite - normalized diagram of TU1 and TU2 (bulk sample)

3.4.2.2.2. Other trace elements

The following elements are significantly enriched over the crustal average ("Clarke values"):

Thorium (700 - 1100 ppm) and Uranium (150 ppm) are probably contained in bastnaesite in the form of thorobastnaesite.

Sr contents are approximately 4000 - 5000 ppm.

The metals Mo, Pb, Zn, As and Mn range between 100 ppm and 150 ppm, and V reaches 300 ppm.
The Au content (60 ppb) is about approx. 20 times the Clarke value.
All other analysed elements (with exception of Li (160 ppm) and Ga (60 ppm) are not essentially enriched or are below the detection limits.

3.5. Scanning electron microscope investigations

3.5.1. Introduction

The results produced by the EDAX analysis system which is coupled with the S.E.M. are only semiquantitative. No standards are available for REE-minerals, and important components of the REE-mineral group are not measurable (e.g. fluorine, CO_3, H_2O and O_2).

The analysis programme calculates automatically all values to total 100%, so that only the element proportions are reflected in the results.
Therefore the lists of analyses are not reported.

3.5.2. Results

At least six significantly different REE-minerals can be distinguished.

1) Zoned REE-minerals with cores composed mainly of Ce and La, and

2) margins composed of La, Ce, Sr, P, and Nd (sample no.: 7025).

3) Grains which contain significant Ca, as well as Ce, La, Sr, P, and Nd (samples no.: 7025, 7041, 7052 and 7054).

4) REE-minerals with Ce, La, Ca, and Nd, but low values of P and Sr (samples no.: 7052 and 7054).

5) REE-aggregates with composition similar to 4) but without P. (samples 7052 and 7054).

6) REE-particles which contain not only Ce, La and Nd, but also Si and Al (sample no.: 7054).

Thorium concentrations in the REE-minerals 3)-6) often reach approx. 2 wt.%.

Possible minerals in 1)-6) are:

Oxides: Cerianite ($(Ce,Th)O_2$)

Carbonates: Bastnaesite ($(Ce,La,Th)CO_3F$); Fluocerite ($(Ce,La)F_3$)
Synchisyte ($(Ce,La)Ca(CO_3)_2F$);
Parisite ($Ca(Ce,La)_2(CO_3)_3F_2$);
Carbocernaite ($(Na,Ca,Sr,Ce)CO_3$);
Burbankite ($(Na,Ca,Sr,Ba,Ce)_6(CO_3)$);
Ambatoarinite ($Sr(Ce,La,Nd)O(CO_3)_3$);
Cordylite ($Ba(Ce,La,Nd)_2(F_2/CO_3)_3$);
Ancylite ($(Ce,La)_4(Sr,Ca)_3(CO_3)_7(OH)_4*3H_2O$);

Phosphates: Belovite ($(Sr,Ce,Na,Ca)_5(PO_4)_3(OH)$);
Fermorite ($(Ca,Sr)_5((As,P)O_4)_3(F,OH)$;

Silicates: Toernbohmite ($(Ce,La,Al)_3(OH/(SiO_4)_3)$);

4. Summary

The investigation of the ore samples reveals that the fluorite and baryte are the main ore minerals. Both are present in at least three generations:

1) The oldest, cataclastically deformed coarsegrained generation.

2) A younger, very finegrained generation which impregnates the older one.

3) The youngest subhedral to euhedral generation which fills pore spaces.

The sequence of generations is also described in the following cristallization - deformation diagram (fig.16):

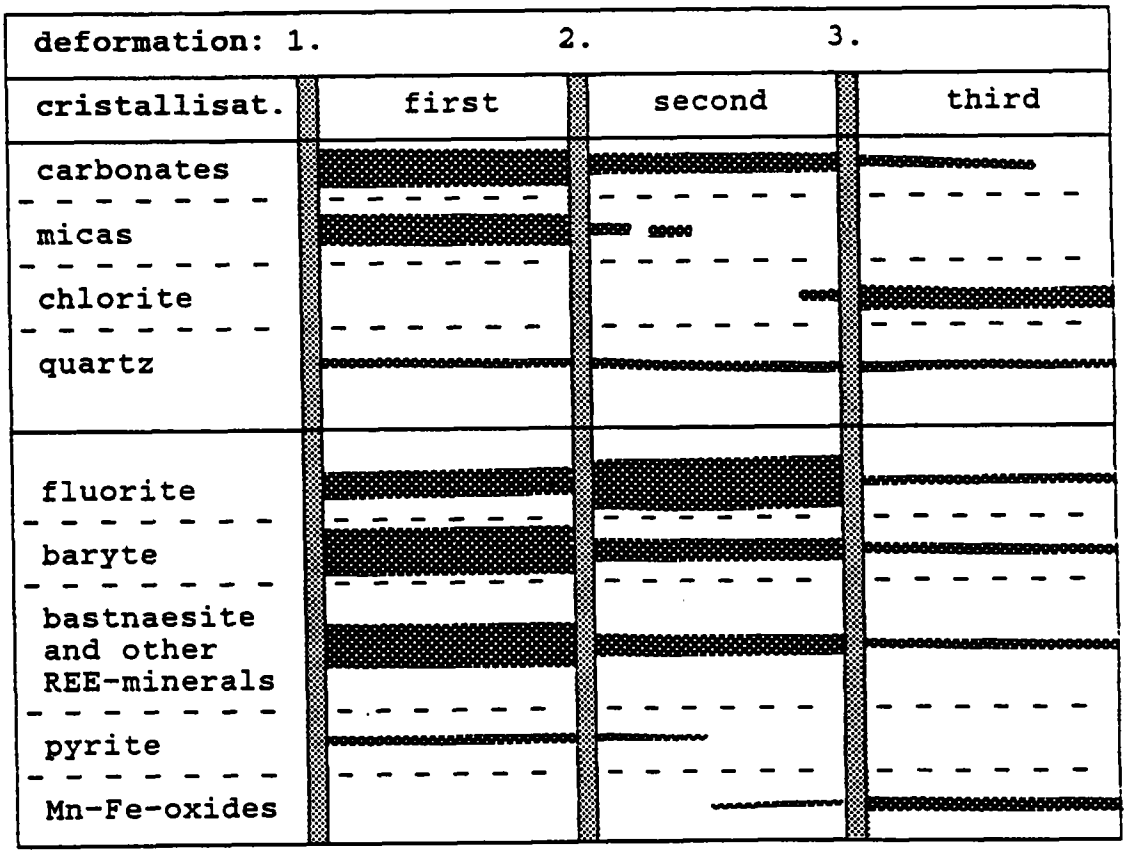

Fig.16: Cristallization - deformation diagram of the REE-ores of the Kizilcaören District

The REE-minerals, in particular bastnaesite, occur in:

1) the carbonate ores, as tiny spheres and schlieren of recrystallized aggregates.

2) in the fluorite-baryte-REE ores, as fine schlieren and aggregates, and

3) rare, but beautifully developed, REE-mineral concentrations in the secondary filled pore spaces.

The X-ray fluorescence analysis and the ICPMS analysis results indicate the following average concentrations of the most important REEs:

Ce:	3.0 - 3.5 wt-%
La:	3.0 - 3.5 wt-%
Nd:	0.4 - 0.6 wt-%
Pr:	0.2 - 0.3 wt-%
Sm:	300 ppm
Gd:	110 ppm

Along with the SEM investigations, the bulk chemical analysis shows that bastnaesite is the most important (at least 95 %), but not the only REE-mineral. A large spectrum of compounds (with e.g. Sr, P, Ca, and Th), which cannot be precisely defined, is present, too.
Th (up to 0.2%) is probably incorporated mainly in the bastnaesite, forming thorobastnaesite.

The occurence of carbonate ores and the normalized chondrite diagram support the suggestion (see e.g. STUMPFL & KIRIKOGLU 1985) that the mineralization is genetically associated with the tertiary volcanism.

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A P P E N D I X

SAMPLE LIST

Sample number:	Ore type:
7025	carbonate ore
7030	fluorite-baryte-REE-minerals - ores (FBR - ore)
7031	FBR - ore
7032	FBR - ore
7033	FBR - ore
7034	FBR - ore
7036	FBR - ore
7037	FBR - ore
7038	FBR - ore
7039	FBR - ore
7040	FBR - ore, with high amount of quartz
7041	FBR - ore
7042	FBR - ore
7043	FBR - ore
7044	FBR - ore
7045	FBR - ore
7046	FBR - ore
7047	FBR - ore
7048	FBR - ore
7049	FBR - ore
7650	FBR - ore
7051	carbonate ore
7052	carbonate ore
7053	FBR - ore
7054	FBR - ore
7055	FBR - ore
7056	FBR - ore

BULK SAMPLE ANALYSES

sample:		TU1		TU2	
Na_2O	[%]	0,03	[<0,4]	0,02	[<0,4]
MgO	[%]	0,36	[0,20]	0,22	[0,23]
Al_2O_3	[%]	0,76	[0,81]	0,76	[0,71]
SiO_2	[%]	2,25	[1,90]	2,00	[1,72]
P_2O_5	[%]	0,57	[0,56]	0,57	[0,53]
SO_4	[%]	8,93	[7,58]	9,08	[7,11]
K_2O	[%]	0,17	[0,18]	0,16	[0,18]
TiO_2	[%]	0,11	[0,14]	0,12	[0,13]
Fe_2O_3	[%]	3,08	[2,46]	3,14	[2,41]
H_2O	[%]	1,20		1,10	
CO_2	[%]	2,40		2,40	
F	[%]	34,30		35,80	
Cl	[ppm]	<50		<50	
Ca	[%]	29,59	[24,36]	29,02	[23,94]
Mn	[ppm]	150	[137]	140	[125]
Ba	[%]	10,30	[9,67]	10,60	[9,41]
Sr	[ppm]	4350	[4061]	4100	[3929]
Rb	[ppm]	10	[<6]	13	[<6]
Nb	[ppm]	3	[<6]	12	[<6]
La	[ppm]	34500	[37552]	36200	[36123]
Ce	[ppm]	32700	[35080]	35000	[34101]
Pr	[ppm]	2390		2480	
Nd	[ppm]	5840	[5383]	6100	[5293]
Sm	[ppm]	310		308	
Eu	[ppm]	61,6		61,0	
Gd	[ppm]	118		108	
Tb	[ppm]	21,9		21,4	
Dy	[ppm]	60,4		58,8	
Ho	[ppm]	10,8		10,5	
Er	[ppm]	29,6		27,6	
Tm	[ppm]	4,3		4,1	
Yb	[ppm]	27,1		26,9	
Lu	[ppm]	3,6		3,8	
Hf	[ppm]	2		5	

		TU1		TU2	
Y	[ppm]	290	[291]	285	[280]
Th	[ppm]	1100		766	
U	[ppm]	156	[64]	162	[63]
Li	[ppm]	170		150	
Be	[ppm]	39		36	
B	[ppm]	<10		10	
Sc	[ppm]	20		15	
V	[ppm]	290		300	
Cr	[ppm]	10	[61]	10	[71]
Co	[ppm]	3	[<8]	2	[<8]
Ni	[ppm]	56	[<8]	63	[<8]
Cu	[ppm]	6		5	
Zn	[ppm]	130	[64]	130	[61]
Ga	[ppm]	64,1		57,1	
Ge	[ppm]	10		20	
As	[ppm]	94		77	
Se	[ppm]	<0,5		<0,5	
Zr	[ppm]	29	[<6]	34	[<6]
Mo	[ppm]	117		123	
Au	[ppb]	51		56	
Ag	[ppm]	<0,5		<0,5	
Cd	[ppm]	<0,2		<0,2	
In	[ppm]	<0,5		<0,5	
Sn	[ppm]	2		6	
Sb	[ppm]	0,7		1,4	
Cs	[ppm]	<1		<1	
Ta	[ppm]	<1		<1	
W	[ppm]	2		6	
Hg	[ppm]	29		24	
Tl	[ppm]	0,4		0,5	
Pb	[ppm]	98		80	
Bi	[ppm]	<0,1		<0,1	

values in [ ] are RFA - data, to check the full analysis

sample	7030	7031	7033	7034	7036	7037	7038
SiO2	0.97	1.60	1.13	1.60	2.01	5.72	7.08
Al2O3	0.53	0.38	0.55	0.38	1.00	2.79	7.79
Fe2O3	1.47	1.09	1.47	2.30	4.13	1.23	0.52
MgO	0.22	0.06	0.07	0.97	0.20	1.31	0.42
CaO	39.59	38.30	39.30	37.80	35.00	31.30	34.20
Na2O	1.82	<N	0.44	<N	0.45	<N	<N
K2O	0.05	0.07	0.07	0.07	0.22	0.81	0.15
TiO2	0.11	0.14	0.12	0.22	0.15	1.78	0.11
P2O5	0.34	0.95	0.45	1.63	0.36	1.91	4.51
MnO	0.01	0.01	0.01	0.01	0.02	0.04	0.01
SO4	9.53	9.63	9.60	7.37	9.45	9.07	4.02
Gv.	4.80	5.20	3.60	6.80	4.70	9.70	10.10
I [wt.%]	82.27	80.31	77.48	81.28	80.39	88.10	82.47
Rb	<N	<N	<N	<N	14	56	9
Sr	4056	4436	3972	4827	4882	6761	9034
Ba	147511	137677	137845	108008	139829	140266	80004
Cr	40	40	73	98	42	66	27
Zr	<N	<N	<N	<N	<N	<N	<N
Co	<N	<N	<N	<N	<N	<N	<N
Ni	<N	<N	<N	25	<N	19	<N
Zn	38	46	56	74	108	99	92
Y	329	357	653	817	252	292	256
Nb	17	<N	<N	<N	11	695	<N
U	58	69	44	123	93	96	56
Th	752	691	996	1159	568	341	328
La	29482	31560	25974	41545	32697	29974	17131
Ce	29449	31793	22842	38121	30740	28721	17310
Nd	4807	4602	4152	5670	5051	4527	3191

sample	7039	7040	7041	7042	7043	7044	7045
SiO2	3.90	17.90	0.62	1.33	44.10	0.32	0.41
Al2O3	3.00	5.14	0.40	1.00	12.10	0.09	0.21
Fe2O3	3.49	1.90	1.36	2.72	19.70	0.46	0.83
MgO	1.29	2.73	<N	<N	0.69	0.67	0.47
CaO	20.90	5.84	30.40	27.50	1.65	51.70	35.40
Na2O	<N	0.71	<N	1.15	<N	0.85	0.90
K2O	0.57	3.70	0.03	0.01	9.46	<N	<N
TiO2	0.29	0.51	0.20	0.26	0.57	0.07	0.13
P2O5	3.23	0.54	0.19	0.58	0.15	0.48	0.76
MnO	0.03	0.08	0.10	3.04	2.91	0.30	0.60
SO4	13.60	21.10	19.29	16.20	<N	4.63	10.90
Gv.	7.10	3.80	2.00	4.40	5.50	1.30	3.40
I [wt.%]	89.34	98.60	86.26	88.34	97.56	70.87	77.18
Rb	38	102	<N	8	173	<N	<N
Sr	8297	1715	2329	4038	237	3880	3393
Ba	195433	306151	288613	250283	4254	71888	158214
Cr	100	70	<N	63	161	<N	42
Zr	<N	<N	<N	<N	127	<N	14
Co	10	<N	<N	37	52	<N	13
Ni	11	<N	20	33	116	<N	<N
Zn	118	135	40	302	468	86	147
Y	327	169	476	980	165	79	92
Nb	35	411	<N	21	36	13	11
U	173	44	<N	<N	<N	<N	45
Th	233	653	663	1268	75	68	92
La	47026	15417	4208	18096	336	10257	31533
Ce	42975	12936	11769	15805	390	8521	24347
Nd	6495	2748	4425	3009	129	1096	2990

oxides [wt.%] & elements [ppm]

C

sample	7046	7047	7048	7049	7050	7051	7052
SiO2	0.90	1.10	1.44	0.92	0.60	0.69	0.73
Al2O3	0.22	0.47	0.40	0.21	0.20	0.25	0.15
Fe2O3	1.54	3.72	4.14	4.16	2.01	1.45	1.43
MgO	0.09	<N	<N	<N	<N	7.39	6.32
CaO	40.30	32.80	35.00	34.59	39.09	31.50	28.00
Na2O	1.56	<N	<N	<N	<N	<N	<N
K2O	0.03	<N	0.12	<N	0.03	0.03	0.05
TiO2	0.15	0.17	0.13	0.13	0.23	0.15	0.18
P2O5	1.04	0.28	1.12	0.69	1.20	0.54	0.23
MnO	0.79	1.99	0.62	0.55	0.71	1.00	1.11
SO4	9.22	14.20	9.55	10.60	9.04	9.38	9.71
Gv.	3.00	3.50	6.90	6.70	5.00	23.60	20.90
Σ [wt.%]	79.16	82.60	83.54	78.98	77.48	93.39	90.65
Rb	10	<N	15	<N	<N	<N	<N
Sr	3575	3348	4096	3335	3155	9027	6738
Ba	138441	217333	134138	150728	133907	132913	141569
Cr	26	<N	94	53	17	6	29
Zr	19	25	6	16	7	40	<N
Co	<N	<N	<N	<N	<N	<N	<N
Ni	<N	<N	<N	9	<N	<N	<N
Zn	350	296	682	579	423	132	69
Y	79	356	270	162	236	183	385
Nb	34	<N	17	13	33	34	<N
U	79	<N	136	85	94	<N	<N
Th	88	414	268	221	330	271	1800
La	24700	4539	48177	30870	24771	12352	20840
Ce	19647	10396	34293	24326	19228	10931	28568
Nd	2405	3063	3323	3154	2778	2123	7213

sample	7054	7055	7056
SiO2	7.63	1.67	5.62
Al2O3	3.63	0.42	2.62
Fe2O3	1.38	1.21	1.14
MgO	0.12	<N	1.54
CaO	35.09	33.59	31.70
Na2O	0.45	<N	<N
K2O	0.03	0.03	0.88
TiO2	0.09	0.18	1.64
P2O5	0.17	0.88	1.89
MnO	0.01	0.64	0.04
SO4	7.94	14.70	9.21
Gv.	9.10	3.60	7.50
Σ [wt.%]	84.90	85.17	85.34
Rb	<N	<N	58
Sr	3818	3515	6386
Ba	119606	215072	136456
Cr	46	21	55
Zr	<N	8	<N
Co	<N	<N	<N
Ni	<N	<N	<N
Zn	51	218	95
Y	386	159	284
Nb	<N	33	611
U	46	82	96
Th	1442	241	236
La	26387	28006	28462
Ce	25714	22264	26715
Nd	4431	3020	4408

oxides [wt.%] & elements [ppm]

RFA - detection limits

Na_2O	0,435%	Th	14 ppm
MgO	0,055%	U	18 ppm
Al_2O_3	0,010%	Cr	5 ppm
SiO_2	0,100%	Co	10 ppm
P_2O_5	0,100%	Ni	9 ppm
K_2O	0,003%	Zn	7 ppm
CaO	0,004%	Rb	6 ppm
TiO_2	0,004%	Sr	7 ppm
Fe_2O_3	0,008%	Y	7 ppm
MnO	0,001%	Zr	7 ppm
SO_4	0,002%	Nb	8 ppm
		Ba	75 ppm
		La	37 ppm
		Ce	115 ppm
		Nd	69 ppm

Detection limits of the bulk analyses

	METHOD	DETECTION LIMIT
AU PPB	FADCP	1.
LI PPM	AA	1.
BE PPM	DCP	1.
B PPM	DCP	10.
NA2O %	DCPA	0.01
MGO %	DCPA	0.01
AL2O3 %	DCPA	0.01
SIO2 %	DCPA	0.01
P2O5 %	DCPA	0.01
S %	XRF	0.01
K2O %	DCPA	0.01
CAO %	DCPA	0.01
SC PPM	ICPMS	1.
TIO2 %	DCPA	0.01
V PPM	DCP	2.
CR PPM	DCP	2.
MN PPM	DCP	2.
FE2O3 %	DCPA	0.01
CO PPM	ICPMS	1.
NI PPM	DCP	1.
CU PPM	DCP	0.5
ZN PPM	DCP	0.5
GA PPM	ICPMS	0.1
GE PPM	DCP	10.
AS PPM	FAA	0.1
SE PPM	GFAA	0.5
RB PPM	ICPMS	1.
SR PPM	DCPA	10.
Y PPM	DCPA	10.
ZR PPM	ICPMS	1.
NB PPM	ICPMS	1.
H2O+ %	WET	0.1
CO2 %	WET	0.01
F %	WET	0.01
CL PPM	XRF	50.

	METHOD	DETECTION LIMIT
MO PPM	ICPMS	1.
AG PPM	DCP	0.5
CD PPM	DCP	0.2
IN PPM	ICPMS	0.5
SN PPM	XRF	2.
SB PPM	FAA	0.2
CS PPM	ICPMS	1.
BAO %	DCPA	0.01
LA PPM	ICPMS	0.1
CE PPM	ICPMS	0.1
PR PPM	ICPMS	0.1
ND PPM	ICPMS	0.1
SM PPM	ICPMS	0.1
EU PPM	ICPMS	0.05
GD PPM	ICPMS	0.1
TB PPM	ICPMS	0.1
DY PPM	ICPMS	0.1
HO PPM	ICPMS	0.05
ER PPM	ICPMS	0.1
TM PPM	ICPMS	0.1
YB PPM	ICPMS	0.1
LU PPM	ICPMS	0.05
HF PPM	ICPMS	1.
TA PPM	ICPMS	1.
W PPM	ICPMS	1.
HG PPB	WET	5.
TL PPM	ICPMS	0.1
PB PPM	DCP	2.
BI PPM	ICPMS	0.1
TH PPM	ICPMS	0.5
U PPM	ICPMS	0.1

A P P E N D I X 4

REO ore sample 1

Statistical study of REE distributions

Sample Correlations

	RP2O5	RCaO	RSiO2	RFe2O3	RAl2O3	RMgO
RP2O5	1.0000	.8538	.2576	.2773	.2479	.2435
	(15)	(15)	(15)	(15)	(15)	(15)
	.0000	.0001	.3541	.3170	.3730	.3819
RCaO	.8538	1.0000	.7170	.7346	.7100	.7067
	(15)	(15)	(15)	(15)	(15)	(15)
	.0001	.0000	.0026	.0018	.0030	.0032
RSiO2	.2576	.7170	1.0000	.9946	.9999	.9929
	(15)	(15)	(15)	(15)	(15)	(15)
	.3541	.0026	.0000	.0000	.0000	.0000
RFe2O3	.2773	.7346	.9946	1.0000	.9946	.9959
	(15)	(15)	(15)	(15)	(15)	(15)
	.3170	.0018	.0000	.0000	.0000	.0000
RAl2O3	.2479	.7100	.9999	.9946	1.0000	.9939
	(15)	(15)	(15)	(15)	(15)	(15)
	.3730	.0030	.0000	.0000	.0000	.0000
RMgO	.2435	.7067	.9929	.9959	.9939	1.0000
	(15)	(15)	(15)	(15)	(15)	(15)
	.3819	.0032	.0000	.0000	.0000	.0000
RLOI	.4426	.8241	.9319	.9503	.9317	.9539
	(15)	(15)	(15)	(15)	(15)	(15)
	.0985	.0002	.0000	.0000	.0000	.0000
RLa	.7825	.9794	.7532	.7847	.7468	.7526
	(15)	(15)	(15)	(15)	(15)	(15)
	.0006	.0000	.0012	.0005	.0014	.0012
RCe	.8253	.9911	.7246	.7524	.7177	.7208
	(15)	(15)	(15)	(15)	(15)	(15)
	.0002	.0000	.0022	.0012	.0026	.0024
RNd	.9202	.9807	.5834	.6116	.5753	.5783
	(15)	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0224	.0154	.0248	.0239
RSm	.9566	.9645	.5127	.5369	.5041	.5040
	(15)	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0507	.0391	.0554	.0554
REu	.9659	.9569	.4855	.5091	.4771	.4773
	(15)	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0665	.0526	.0722	.0720
RGd	.9708	.9507	.4675	.4917	.4588	.4590
	(15)	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0789	.0627	.0854	.0852
RDy	.9800	.9363	.4293	.4526	.4206	.4207
	(15)	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.1103	.0903	.1185	.1184

Coefficient (sample size) significance level

	RLOI	RLa	RCe	RNd	RSm	REu
RP2O5	.4426	.7825	.8253	.9202	.9566	.9659
	(15)	(15)	(15)	(15)	(15)	(15)
	.0985	.0006	.0002	.0000	.0000	.0000
RCaO	.8241	.9794	.9911	.9807	.9645	.9569
	(15)	(15)	(15)	(15)	(15)	(15)
	.0002	.0000	.0000	.0000	.0000	.0000
RSiO2	.9319	.7532	.7246	.5834	.5127	.4855
	(15)	(15)	(15)	(15)	(15)	(15)
	.0000	.0012	.0022	.0224	.0507	.0665
RFe2O3	.9503	.7847	.7524	.6116	.5369	.5091
	(15)	(15)	(15)	(15)	(15)	(15)
	.0000	.0005	.0012	.0154	.0391	.0526
RAl2O3	.9317	.7468	.7177	.5753	.5041	.4771
	(15)	(15)	(15)	(15)	(15)	(15)
	.0000	.0014	.0026	.0248	.0554	.0722
RMgO	.9539	.7526	.7208	.5783	.5040	.4773
	(15)	(15)	(15)	(15)	(15)	(15)
	.0000	.0012	.0024	.0239	.0554	.0720
RLOI	1.0000	.8616	.8394	.7345	.6714	.6509
	(15)	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0001	.0018	.0061	.0086
RLa	.8616	1.0000	.9966	.9629	.9282	.9138
	(15)	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0000	.0000	.0000	.0000
RCe	.8394	.9966	1.0000	.9797	.9533	.9412
	(15)	(15)	(15)	(15)	(15)	(15)
	.0001	.0000	.0000	.0000	.0000	.0000
RNd	.7345	.9629	.9797	1.0000	.9938	.9881
	(15)	(15)	(15)	(15)	(15)	(15)
	.0018	.0000	.0000	.0000	.0000	.0000
RSm	.6714	.9282	.9533	.9938	1.0000	.9980
	(15)	(15)	(15)	(15)	(15)	(15)
	.0061	.0000	.0000	.0000	.0000	.0000
REu	.6509	.9138	.9412	.9881	.9980	1.0000
	(15)	(15)	(15)	(15)	(15)	(15)
	.0086	.0000	.0000	.0000	.0000	.0000
RGd	.6347	.9070	.9354	.9860	.9977	.9996
	(15)	(15)	(15)	(15)	(15)	(15)
	.0110	.0000	.0000	.0000	.0000	.0000
RDy	.6023	.8847	.9162	.9757	.9925	.9975
	(15)	(15)	(15)	(15)	(15)	(15)
	.0175	.0000	.0000	.0000	.0000	.0000

	RGd	RDy	REr	RYb	RY
RP2O5	.9708	.9800	.9621	.9394	.9654
	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0000	.0000	.0000
RCaO	.9507	.9363	.9538	.9606	.9523
	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0000	.0000	.0000
RSiO2	.4675	.4293	.4804	.5185	.4755
	(15)	(15)	(15)	(15)	(15)
	.0789	.1103	.0699	.0477	.0732
RFe2O3	.4917	.4526	.5039	.5426	.5000
	(15)	(15)	(15)	(15)	(15)
	.0627	.0903	.0555	.0366	.0577
RAl2O3	.4588	.4206	.4721	.5107	.4674
	(15)	(15)	(15)	(15)	(15)
	.0854	.1185	.0756	.0518	.0790
RMgO	.4590	.4207	.4718	.5125	.4700
	(15)	(15)	(15)	(15)	(15)
	.0852	.1184	.0758	.0508	.0771
RLOI	.6347	.6023	.6471	.6869	.6483
	(15)	(15)	(15)	(15)	(15)
	.0110	.0175	.0091	.0047	.0090
RLa	.9070	.8847	.9080	.9219	.9071
	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0000	.0000	.0000
RCe	.9354	.9162	.9351	.9446	.9349
	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0000	.0000	.0000
RNd	.9860	.9757	.9817	.9808	.9841
	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0000	.0000	.0000
RSm	.9977	.9925	.9915	.9840	.9946
	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0000	.0000	.0000
REu	.9996	.9975	.9970	.9897	.9986
	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0000	.0000	.0000
RGd	1.0000	.9984	.9959	.9864	.9978
	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0000	.0000	.0000
RDy	.9984	1.0000	.9960	.9849	.9974
	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0000	.0000	.0000

Sample Correlations

	RP2O5	RCaO	RSiO2	RFe2O3	RAl2O3	RMgO
REr	.9621	.9538	.4804	.5039	.4721	.4718
	(15)	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0699	.0555	.0756	.0758
RYb	.9394	.9606	.5185	.5426	.5107	.5125
	(15)	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0477	.0366	.0518	.0508
RY	.9654	.9523	.4755	.5000	.4674	.4700
	(15)	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0732	.0577	.0790	.0771

Coefficient (sample size) significance level

	RLOI	RLa	RCe	RNd	RSm	REu
REr	.6471	.9080	.9351	.9817	.9915	.9970
	(15)	(15)	(15)	(15)	(15)	(15)
	.0091	.0000	.0000	.0000	.0000	.0000
RYb	.6869	.9219	.9446	.9808	.9840	.9897
	(15)	(15)	(15)	(15)	(15)	(15)
	.0047	.0000	.0000	.0000	.0000	.0000
RY	.6483	.9071	.9349	.9841	.9946	.9986
	(15)	(15)	(15)	(15)	(15)	(15)
	.0090	.0000	.0000	.0000	.0000	.0000

	RGd	RDy	REr	RYb	RY
REr	.9959	.9960	1.0000	.9957	.9981
	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0000	.0000	.0000
RYb	.9864	.9849	.9957	1.0000	.9928
	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0000	.0000	.0000
RY	.9978	.9974	.9981	.9928	1.0000
	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0000	.0000	.0000

Sample Correlations

	P2O5	CaO	SiO2	Fe2O3	Al2O3	MgO
P2O5	1.0000	.9953	-.9531	-.5317	-.9716	-.6243
	(15)	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0000	.0414	.0000	.0129
CaO	.9953	1.0000	-.9562	-.5349	-.9816	-.6168
	(15)	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0000	.0399	.0000	.0143
SiO2	-.9531	-.9562	1.0000	.2685	.9778	.3899
	(15)	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0000	.3332	.0000	.1508
Fe2O3	-.5317	-.5349	.2685	1.0000	.4074	.8850
	(15)	(15)	(15)	(15)	(15)	(15)
	.0414	.0399	.3332	.0000	.1317	.0000
Al2O3	-.9716	-.9816	.9778	.4074	1.0000	.5347
	(15)	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0000	.1317	.0000	.0400
MgO	-.6243	-.6168	.3899	.8850	.5347	1.0000
	(15)	(15)	(15)	(15)	(15)	(15)
	.0129	.0143	.1508	.0000	.0400	.0000
LOI	.2141	.2605	-.4401	.3936	-.3451	.5036
	(15)	(15)	(15)	(15)	(15)	(15)
	.4435	.3484	.1007	.1466	.2078	.0556
La	.9334	.9602	-.9273	-.4874	-.9647	-.5569
	(15)	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0000	.0654	.0000	.0310
Ce	.9572	.9777	-.9455	-.4913	-.9761	-.5718
	(15)	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0000	.0629	.0000	.0259
Nd	.9831	.9934	-.9599	-.5035	-.9841	-.5901
	(15)	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0000	.0557	.0000	.0206
Sm	.9938	.9979	-.9614	-.5155	-.9836	-.6021
	(15)	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0000	.0492	.0000	.0175
Eu	.9968	.9979	-.9639	-.5092	-.9817	-.6023
	(15)	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0000	.0525	.0000	.0175
Gd	.9958	.9976	-.9643	-.5075	-.9821	-.5998
	(15)	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0000	.0535	.0000	.0181
Dy	.9977	.9965	-.9669	-.4966	-.9812	-.5941
	(15)	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0000	.0597	.0000	.0195

Coefficient (sample size) significance level

	LOI	La	Ce	Nd	Sm	Eu
P2O5	.2141	.9334	.9572	.9831	.9938	.9968
	(15)	(15)	(15)	(15)	(15)	(15)
	.4435	.0000	.0000	.0000	.0000	.0000
CaO	.2605	.9602	.9777	.9934	.9979	.9979
	(15)	(15)	(15)	(15)	(15)	(15)
	.3484	.0000	.0000	.0000	.0000	.0000
SiO2	-.4401	-.9273	-.9455	-.9599	-.9614	-.9639
	(15)	(15)	(15)	(15)	(15)	(15)
	.1007	.0000	.0000	.0000	.0000	.0000
Fe2O3	.3936	-.4874	-.4913	-.5035	-.5155	-.5092
	(15)	(15)	(15)	(15)	(15)	(15)
	.1466	.0654	.0629	.0557	.0492	.0525
Al2O3	-.3451	-.9647	-.9761	-.9841	-.9836	-.9817
	(15)	(15)	(15)	(15)	(15)	(15)
	.2078	.0000	.0000	.0000	.0000	.0000
MgO	.5036	-.5569	-.5718	-.5901	-.6021	-.6023
	(15)	(15)	(15)	(15)	(15)	(15)
	.0556	.0310	.0259	.0206	.0175	.0175
LOI	1.0000	.3483	.3325	.2967	.2702	.2566
	(15)	(15)	(15)	(15)	(15)	(15)
	.0000	.2033	.2260	.2829	.3301	.3559
La	.3483	1.0000	.9911	.9738	.9578	.9534
	(15)	(15)	(15)	(15)	(15)	(15)
	.2033	.0000	.0000	.0000	.0000	.0000
Ce	.3325	.9911	1.0000	.9925	.9789	.9729
	(15)	(15)	(15)	(15)	(15)	(15)
	.2260	.0000	.0000	.0000	.0000	.0000
Nd	.2967	.9738	.9925	1.0000	.9960	.9918
	(15)	(15)	(15)	(15)	(15)	(15)
	.2829	.0000	.0000	.0000	.0000	.0000
Sm	.2702	.9578	.9789	.9960	1.0000	.9977
	(15)	(15)	(15)	(15)	(15)	(15)
	.3301	.0000	.0000	.0000	.0000	.0000
Eu	.2566	.9534	.9729	.9918	.9977	1.0000
	(15)	(15)	(15)	(15)	(15)	(15)
	.3559	.0000	.0000	.0000	.0000	.0000
Gd	.2666	.9540	.9736	.9919	.9973	.9994
	(15)	(15)	(15)	(15)	(15)	(15)
	.3368	.0000	.0000	.0000	.0000	.0000
Dy	.2529	.9466	.9685	.9900	.9973	.9989
	(15)	(15)	(15)	(15)	(15)	(15)
	.3632	.0000	.0000	.0000	.0000	.0000

	Gd	Dy	Er	Yb	Y
P2O5	.9958	.9977	.9789	.9494	.9933
	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0000	.0000	.0000
CaO	.9976	.9965	.9791	.9513	.9896
	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0000	.0000	.0000
SiO2	-.9643	-.9669	-.9472	-.9195	-.9543
	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0000	.0000	.0000
Fe2O3	-.5075	-.4966	-.4957	-.4877	-.5082
	(15)	(15)	(15)	(15)	(15)
	.0535	.0597	.0602	.0651	.0531
Al2O3	-.9821	-.9812	-.9630	-.9375	-.9704
	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0000	.0000	.0000
MgO	-.5998	-.5941	-.5897	-.5800	-.6048
	(15)	(15)	(15)	(15)	(15)
	.0181	.0195	.0207	.0234	.0169
LOI	.2666	.2529	.2792	.2883	.2488
	(15)	(15)	(15)	(15)	(15)
	.3368	.3632	.3135	.2973	.3712
La	.9540	.9466	.9335	.9181	.9286
	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0000	.0000	.0000
Ce	.9736	.9685	.9450	.9203	.9493
	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0000	.0000	.0000
Nd	.9919	.9900	.9625	.9322	.9743
	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0000	.0000	.0000
Sm	.9973	.9973	.9714	.9397	.9863
	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0000	.0000	.0000
Eu	.9994	.9989	.9783	.9482	.9895
	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0000	.0000	.0000
Gd	1.0000	.9986	.9826	.9541	.9917
	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0000	.0000	.0000
Dy	.9986	1.0000	.9787	.9480	.9920
	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0000	.0000	.0000

Sample Correlations

	P2O5	CaO	SiO2	Fe2O3	Al2O3	MgO
Er	.9789	.9791	-.9472	-.4957	-.9630	-.5897
	(15)	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0000	.0602	.0000	.0207
Yb	.9494	.9513	-.9195	-.4877	-.9375	-.5800
	(15)	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0000	.0651	.0000	.0234
Y	.9933	.9896	-.9543	-.5082	-.9704	-.6048
	(15)	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0000	.0531	.0000	.0169

Coefficient (sample size) significance level

	LOI	La	Ce	Nd	Sm	Eu
Er	.2792	.9335	.9450	.9625	.9714	.9783
	(15)	(15)	(15)	(15)	(15)	(15)
	.3135	.0000	.0000	.0000	.0000	.0000
Yb	.2883	.9181	.9203	.9322	.9397	.9482
	(15)	(15)	(15)	(15)	(15)	(15)
	.2973	.0000	.0000	.0000	.0000	.0000
Y	.2488	.9286	.9493	.9743	.9863	.9895
	(15)	(15)	(15)	(15)	(15)	(15)
	.3712	.0000	.0000	.0000	.0000	.0000

	Gd	Dy	Er	Yb	Y
Er	.9826	.9787	1.0000	.9914	.9934
	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0000	.0000	.0000
Yb	.9541	.9480	.9914	1.0000	.9736
	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0000	.0000	.0000
Y	.9917	.9920	.9934	.9736	1.0000
	(15)	(15)	(15)	(15)	(15)
	.0000	.0000	.0000	.0000	.0000

Regression Analysis - Linear model: Y = a+bX

Dependent variable: RLa Independent variable: RP2O5

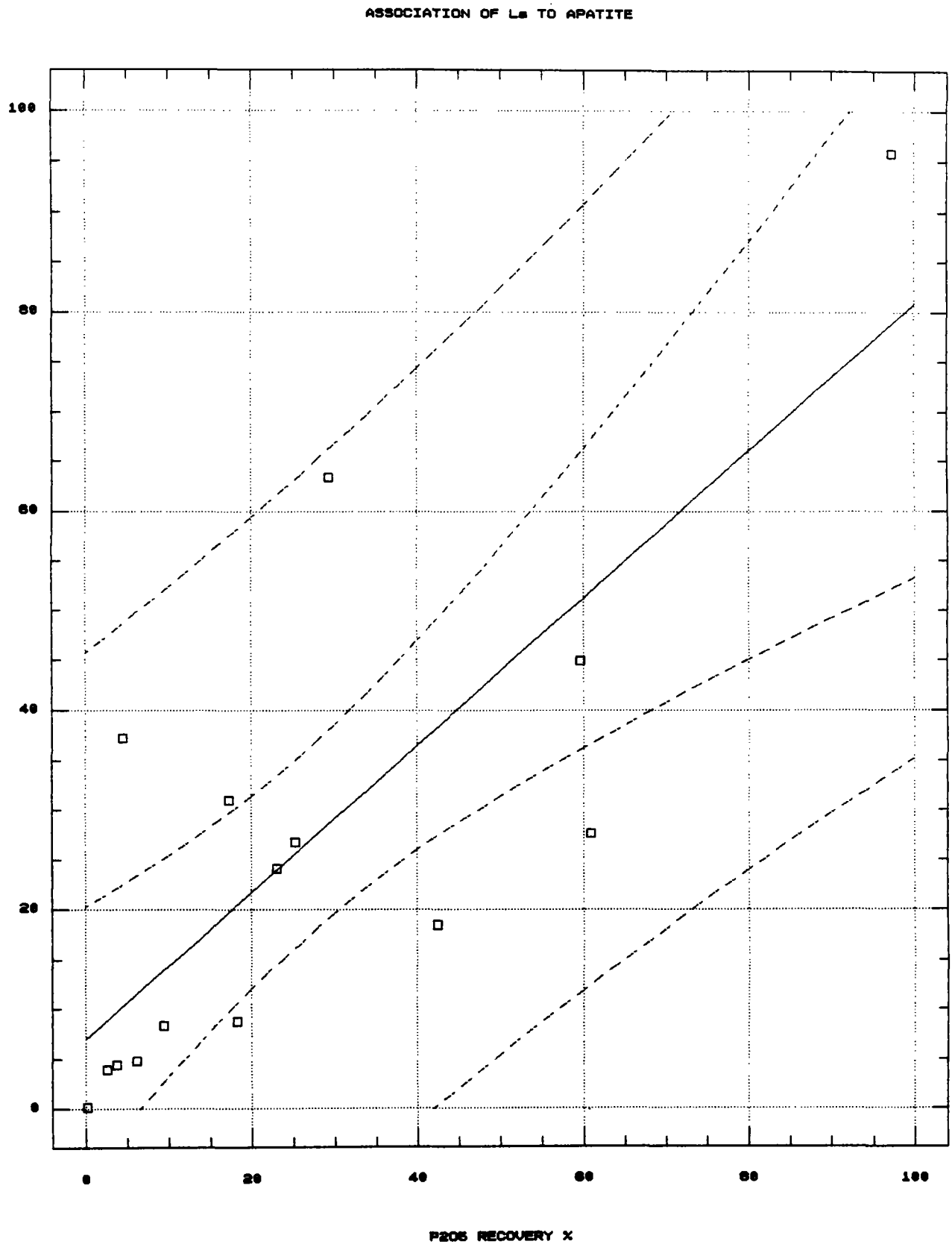
Parameter	Estimate	Standard Error	T Value	Prob. Level
Intercept	6.97223	6.14868	1.13394	.27730
Slope	0.738505	0.162995	4.53083	.00056

Analysis of Variance

Source	Sum of Squares	Df	Mean Square	F-Ratio	Prob. Level
Model	5823.4793	1	5823.4793	20.528	.00056
Error	3687.8260	13	283.6789		
Total (Corr.)	9511.3053	14			

Correlation Coefficient = 0.782476 R-squared = 61.23 percent
Stnd. Error of Est. = 16.8428

REO ORE SAMPLE 1



Model fitting results for: RLa

Independent variable	coefficient	std. error	t-value	sig.level
CONSTANT	0.868721	2.666032	0.3258	0.7502
RP2O5	0.594865	0.070052	8.4917	0.0000
RSiO2	0.372538	0.046794	7.9612	0.0000

R-SQ. (ADJ.) = 0.9280 SE= 6.994462 MAE= 4.690954 DurbWat= 2.282
Previously: 0.0000 0.000000 0.000000 0.000
15 observations fitted, forecast(s) computed for 0 missing val. of dep. var.

Analysis of Variance for the Full Regression

Source	Sum of Squares	DF	Mean Square	F-Ratio	P-value
Model	8924.24	2	4462.12	91.2079	.0000
Error	587.070	12	48.9225		
Total (Corr.)	9511.31	14			

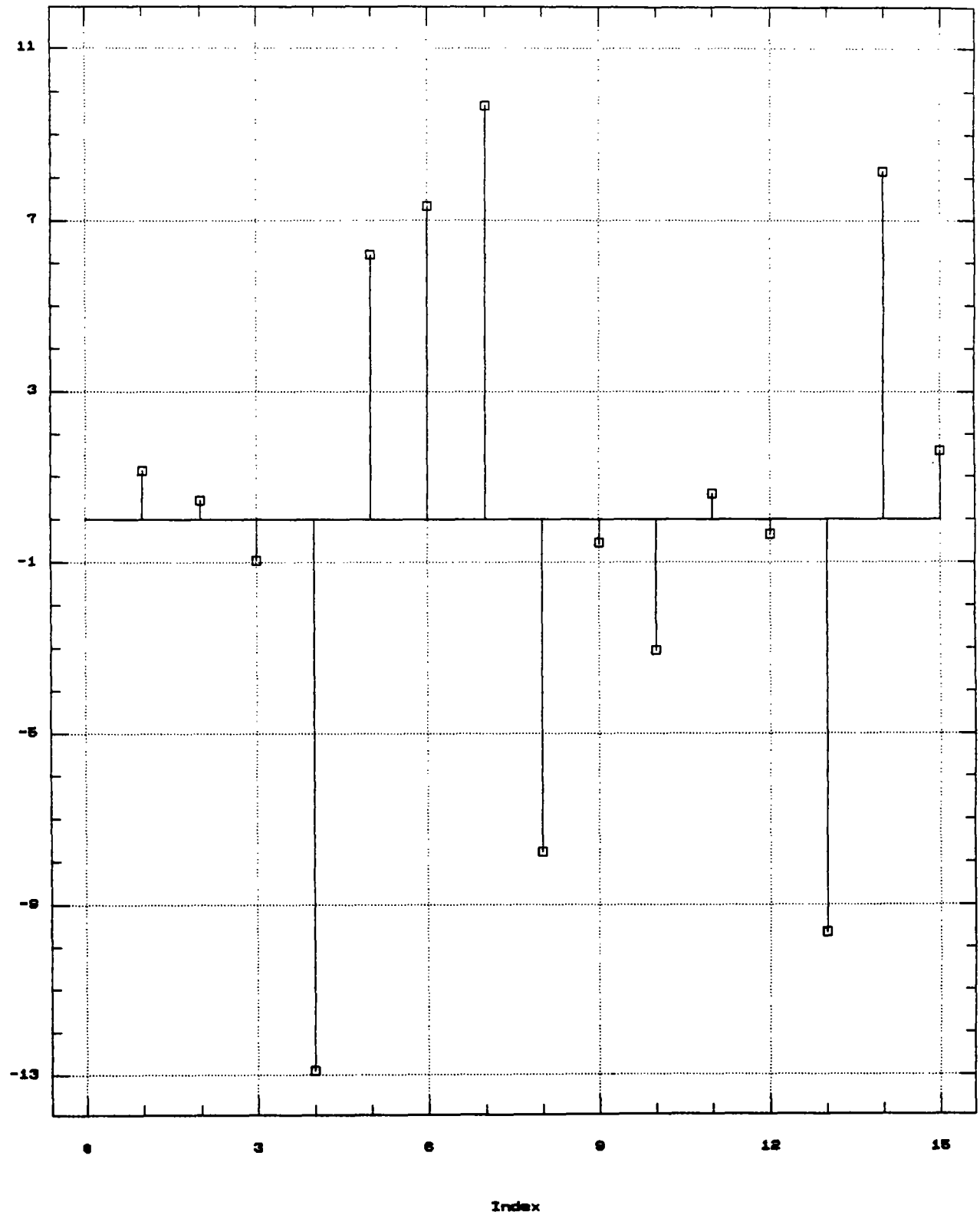
R-squared = 0.938277 Stnd. error of est. = 6.99446
R-squared (Adj. for d.f.) = 0.927989 Durbin-Watson statistic = 2.28176

Further ANOVA for Variables in the Order Fitted

Source	Sum of Squares	DF	Mean Sq.	F-Ratio	P-value
RP2O5	5823.47931	1	5823.4793	119.03	.0000
RSiO2	3100.75596	1	3100.7560	63.38	.0000
Model	8924.23527	2			

row	LaOBSERVED	LaFITTED	LaRESIDUAL
1	95.84	94.69	1.15
2	3.95	3.49	0.46
3	0.21	1.16	-0.95
4	30.97	43.86	-12.89
5	24.13	17.93	6.20
6	44.90	37.55	7.35
7	63.40	53.72	9.68
8	18.51	26.28	-7.77
9	4.84	5.36	-0.52
10	8.78	11.85	-3.07
11	4.47	3.87	0.60
12	37.21	37.55	-0.34
13	27.67	37.31	-9.64
14	26.78	18.63	8.15
15	8.34	6.73	1.61

Residual Plot for RLa



Regression Analysis - Linear model: Y = a+bX

Dependent variable: RCe Independent variable: RP2O5

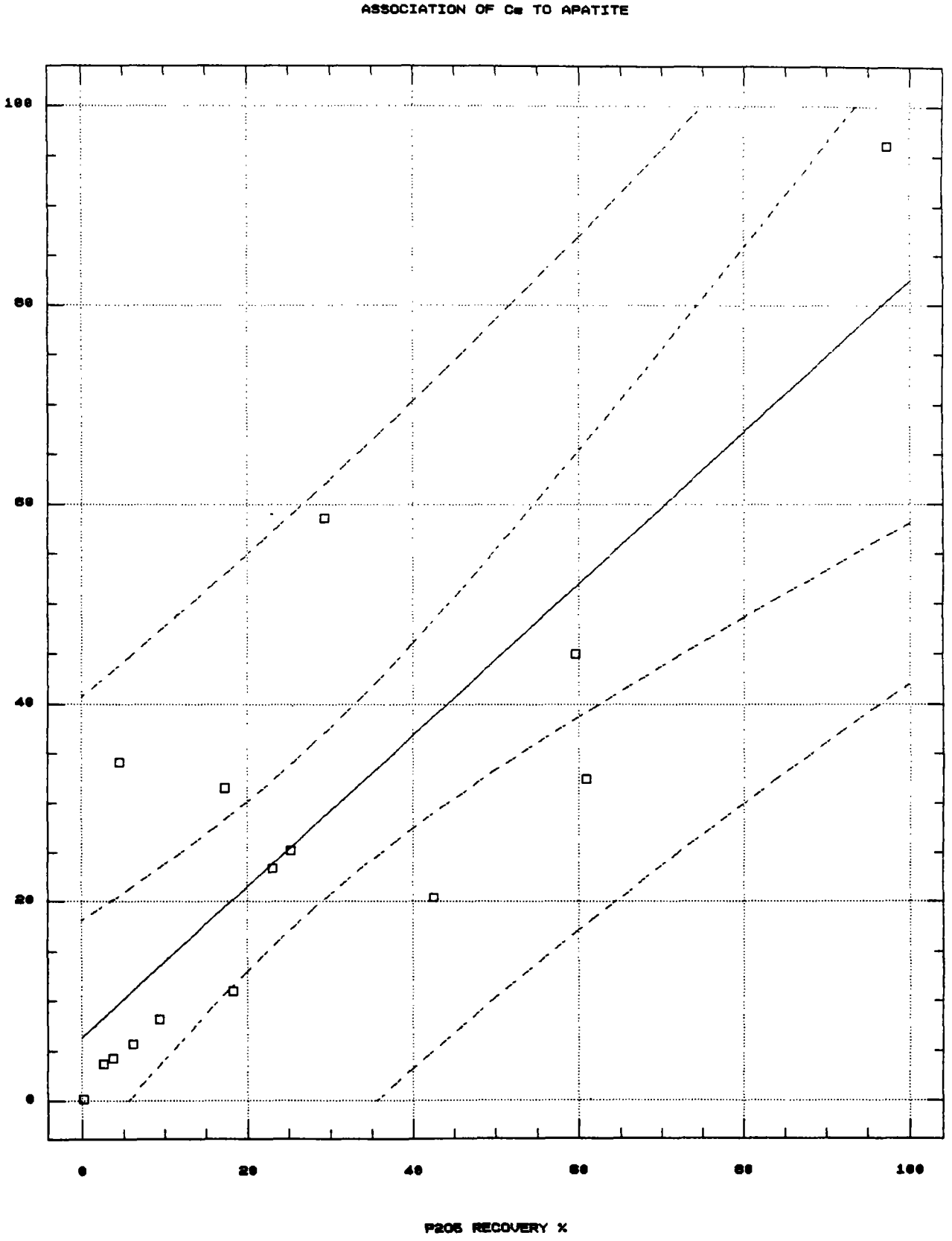
Parameter	Estimate	Standard Error	T Value	Prob. Level
Intercept	6.36057	5.45046	1.16698	.26418
Slope	0.76139	0.144486	5.26964	.00015

Analysis of Variance

Source	Sum of Squares	Df	Mean Square	F-Ratio	Prob. Level
Model	6190.0057	1	6190.0057	27.769	.00015
Error	2897.8295	13	222.9100		
Total (Corr.)	9087.8352	14			

Correlation Coefficient = 0.825307 R-squared = 68.11 percent
Stnd. Error of Est. = 14.9302

REO ORE SAMPLE 1



Model fitting results for: RCe

Independent variable	coefficient	std. error	t-value	sig.level
CONSTANT	0.823207	2.045614	0.4024	0.6944
RP2O5	0.631074	0.05375	11.7409	0.0000
RSiO2	0.337983	0.035905	9.4134	0.0000

R-SQ. (ADJ.) = 0.9556 SE= 5.366766 MAE= 3.579688 DurbWat= 2.343
Previously: 0.0000 0.000000 0.000000 0.000
15 observations fitted, forecast(s) computed for 0 missing val. of dep. var.

Analysis of Variance for the Full Regression

Source	Sum of Squares	DF	Mean Square	F-Ratio	P-value
Model	8742.21	2	4371.10	151.763	.0000
Error	345.626	12	28.8022		
Total (Corr.)	9087.84	14			

R-squared = 0.961968 Stnd. error of est. = 5.36677
R-squared (Adj. for d.f.) = 0.95563 Durbin-Watson statistic = 2.3426

Further ANOVA for Variables in the Order Fitted

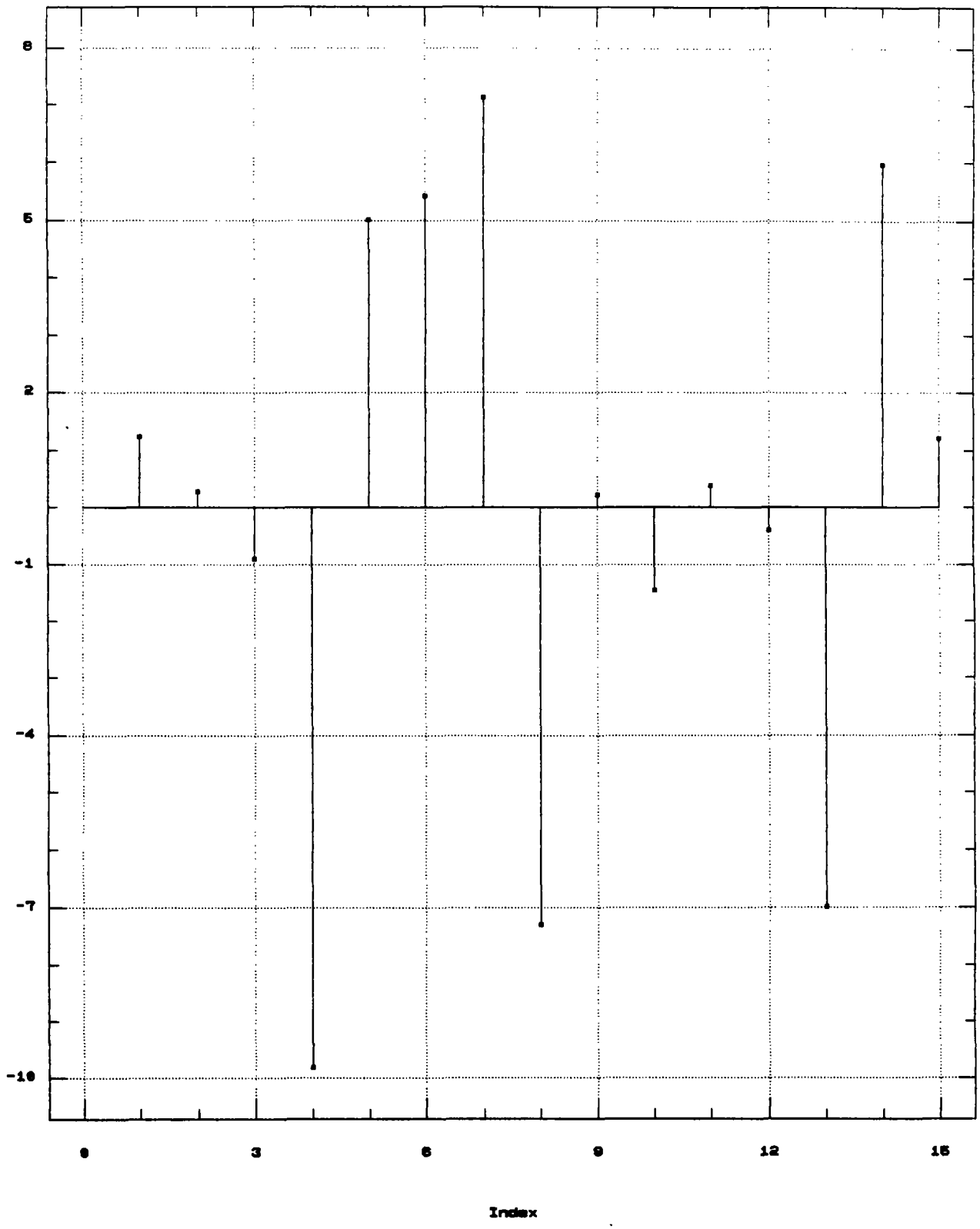
Source	Sum of Squares	DF	Mean Sq.	F-Ratio	P-value
RP2O5	6190.00572	1	6190.0057	214.91	.0000
RSiO2	2552.20339	1	2552.2034	88.61	.0000
Model	8742.20910	2			

95 percent confidence intervals for coefficient estimates

	Estimate	Standard error	Lower Limit	Upper Limit
CONSTANT	0.82321	2.04561	-3.63495	5.28137
RP2O5	0.63107	0.05375	0.51393	0.74822
RSiO2	0.33798	0.03590	0.25973	0.41623

row	CeOBSERVED	CeFITTED	CeRESIDUAL
1	96.06	94.82	1.24
2	3.72	3.44	0.28
3	0.21	1.11	-0.90
4	31.59	41.40	-9.81
5	23.43	18.41	5.02
6	44.98	39.56	5.42
7	58.58	51.45	7.13
8	20.45	27.76	-7.31
9	5.68	5.47	0.21
10	11.01	12.46	-1.45
11	4.27	3.89	0.38
12	34.11	34.51	-0.40
13	32.47	39.45	-6.98
14	25.20	19.24	5.96
15	8.22	7.00	1.22

Residual Plot for RCa



Regression Analysis - Linear model: Y = a+bX

Dependent variable: REu — Independent variable: RP2O5

Parameter	Estimate	Standard Error	T Value	Prob. Level
Intercept	3.22521	2.46516	1.30832	.21343
Slope	0.879011	0.0653491	13.451	.00000

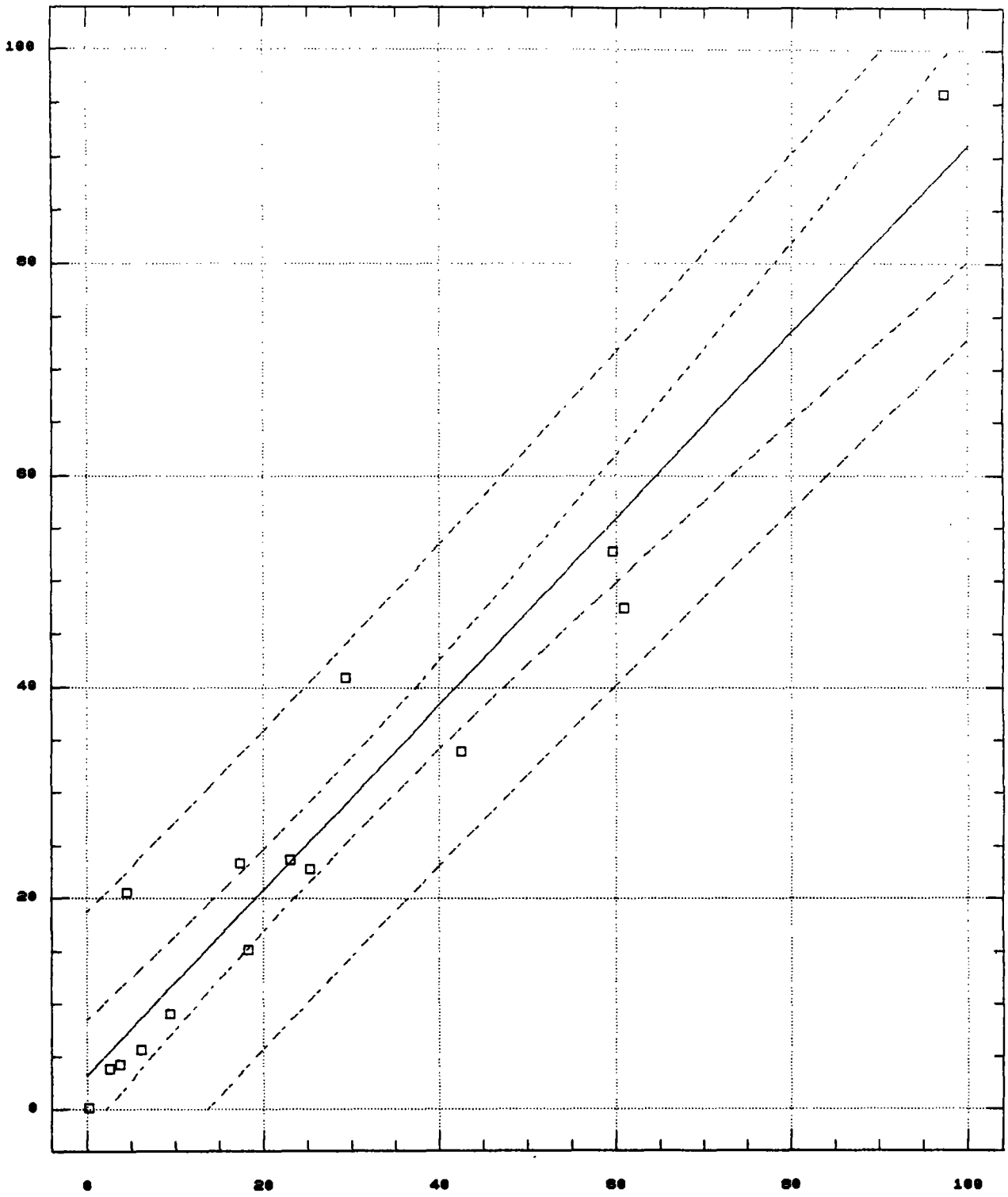
Analysis of Variance

Source	Sum of Squares	Df	Mean Square	F-Ratio	Prob. Level
Model	8250.1986	1	8250.1986	180.930	.00000
Error	592.78652	13	45.59896		
Total (Corr.)	8842.9851	14			

Correlation Coefficient = 0.965901 R-squared = 93.30 percent
Stnd. Error of Est. = 6.7527

REO ORE SAMPLE 1

ASSOCIATION OF Eu TO APATITE



Model fitting results for: REu

Independent variable	coefficient	std. error	t-value	sig.level
CONSTANT	0.699628	0.86543	0.8084	0.4346
RP2O5	0.819573	0.02274	36.0413	0.0000
RSiO2	0.154153	0.01519	10.1484	0.0000

R-SQ. (ADJ.) = 0.9918 SE= 2.270496 MAE= 1.536093 DurbWat= 2.217
Previously: 0.0000 0.000000 0.000000 0.000
15 observations fitted, forecast(s) computed for 0 missing val. of dep. var.

Analysis of Variance for the Full Regression

Source	Sum of Squares	DF	Mean Square	F-Ratio	P-value
Model	8781.12	2	4390.56	851.685	.0000
Error	61.8618	12	5.15515		
Total (Corr.)	8842.99	14			

R-squared = 0.993004 Stnd. error of est. = 2.2705
R-squared (Adj. for d.f.) = 0.991838 Durbin-Watson statistic = 2.21674

Further ANOVA for Variables in the Order Fitted

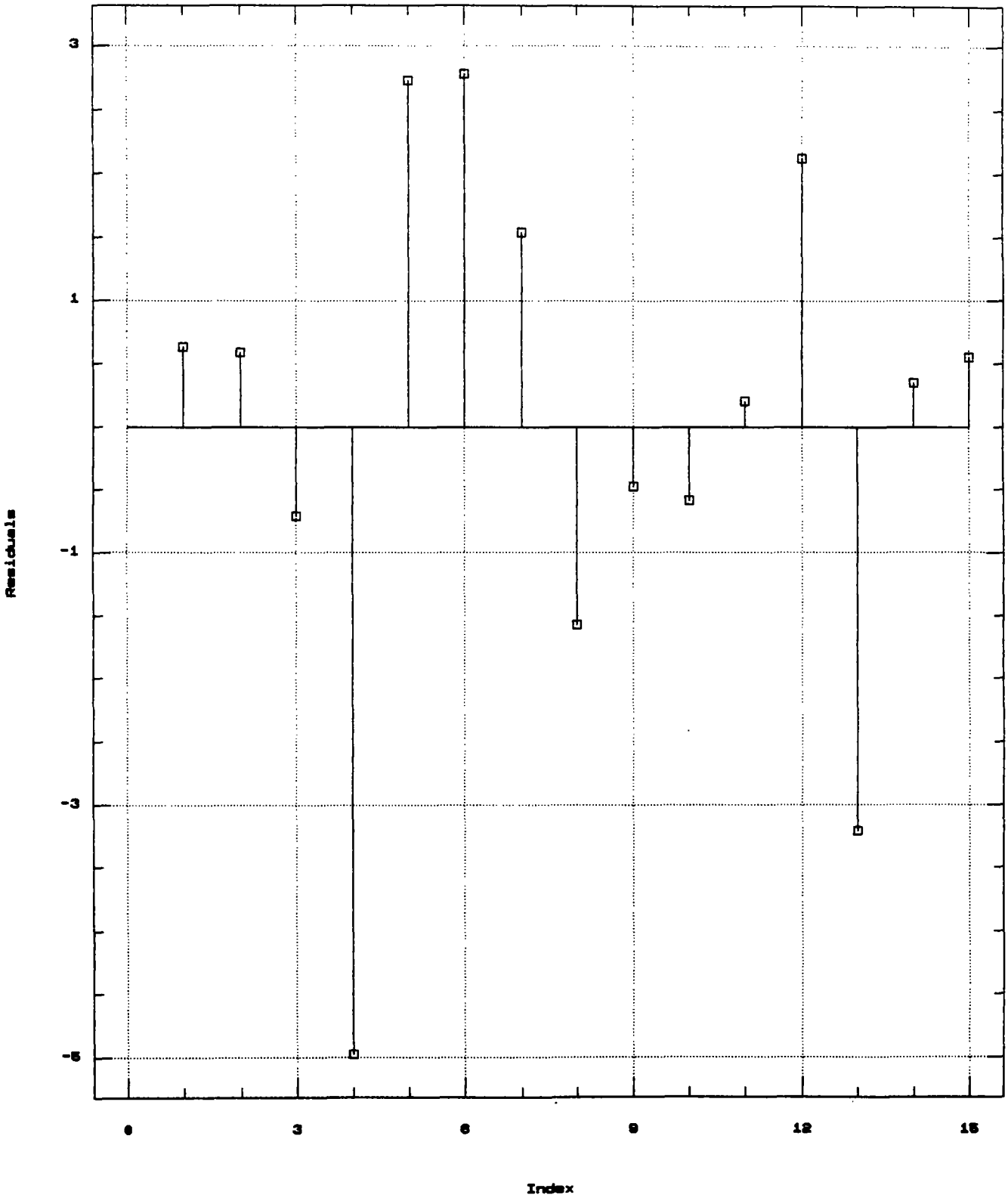
Source	Sum of Squares	DF	Mean Sq.	F-Ratio	P-value
RP2O5	8250.19862	1	8250.1986	1600.38	.0000
RSiO2	530.92471	1	530.9247	102.99	.0000
Model	8781.12333	2			

95 percent confidence intervals for coefficient estimates

	Estimate	Standard error	Lower Limit	Upper Limit
CONSTANT	0.69963	0.86543	-1.18647	2.58572
RP2O5	0.81957	0.02274	0.77001	0.86913
RSiO2	0.15415	0.01519	0.12105	0.18726

row	REuOBSERV.	REuFITTED	REuRESIDU.
1	95.88	95.24	0.64
2	3.88	3.29	0.59
3	0.23	0.94	-0.71
4	23.42	28.39	-4.97
5	23.71	20.98	2.73
6	52.88	50.10	2.78
7	40.93	39.39	1.54
8	34.00	35.57	-1.57
9	5.63	6.10	-0.47
10	15.15	15.73	-0.58
11	4.29	4.08	0.21
12	20.57	18.45	2.12
13	47.49	50.70	-3.21
14	22.87	22.52	0.35
15	9.07	8.52	0.55

Residual Plot for REu



Regression Analysis - Linear model: Y = a+bX

Dependent variable: RY — Independent variable: RP205

Parameter	Estimate	Standard Error	T Value	Prob. Level
Intercept	3.2645	2.48181	1.31537	.21111
Slope	0.877562	0.0657904	13.3388	.00000

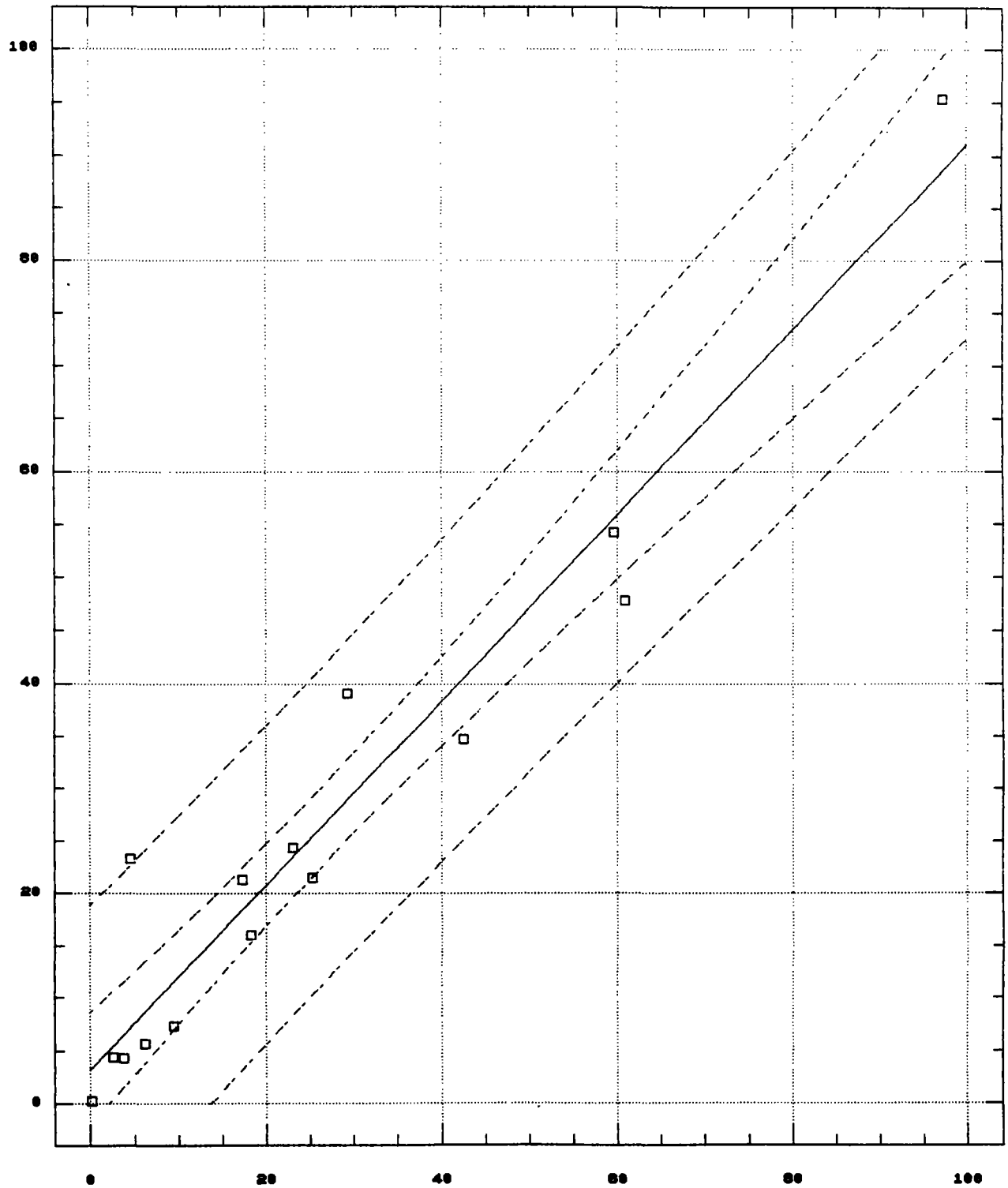
Analysis of Variance

Source	Sum of Squares	Df	Mean Square	F-Ratio	Prob. Level
Model	8223.0343	1	8223.0343	177.922	.00000
Error	600.82039	13	46.21695		
Total (Corr.)	8823.8547	14			

Correlation Coefficient = 0.965355 R-squared = 93.19 percent
Stnd. Error of Est. = 6.79831

REO ORE SAMPLE 1

ASSOCIATION OF Y TO APATITE



Model fitting results for: RY

Independent variable	coefficient	std. error	t-value	sig.level
CONSTANT	0.846789	1.176267	0.7199	0.4854
RP2O5	0.820664	0.030907	26.5524	0.0000
RSiO2	0.147569	0.020646	7.1477	0.0000

R-SQ. (ADJ.) = 0.9849 SE= 3.085992 MAE= 1.933574 DurbWat= 2.218
Previously: 0.9556 5.366766 3.579688 2.343
15 observations fitted, forecast(s) computed for 0 missing val. of dep. var.

Analysis of Variance for the Full Regression

Source	Sum of Squares	DF	Mean Square	F-Ratio	P-value
Model	8709.57	2	4354.79	457.275	.0000
Error	114.280	12	9.52335		
Total (Corr.)	8823.85	14			

R-squared = 0.987049 Stnd. error of est. = 3.08599
R-squared (Adj. for d.f.) = 0.98489 Durbin-Watson statistic = 2.21825

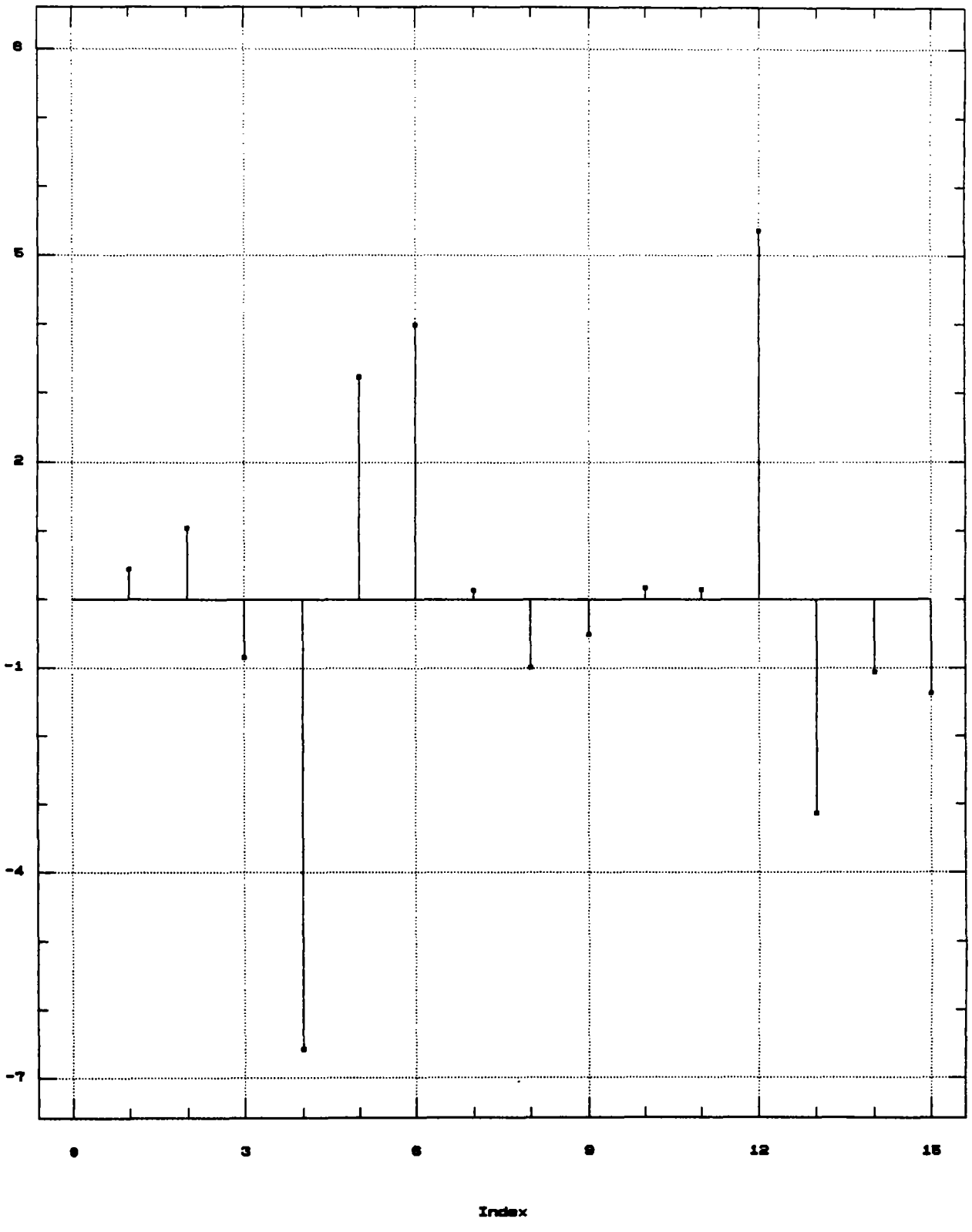
Further ANOVA for Variables in the Order Fitted

Source	Sum of Squares	DF	Mean Sq.	F-Ratio	P-value
RP2O5	8223.03431	1	8223.0343	863.46	.0000
RSiO2	486.54022	1	486.5402	51.09	.0000
Model	8709.57453	2			

95 percent confidence intervals for coefficient estimates

	Estimate	Standard error	Lower Limit	Upper Limit
CONSTANT	0.84679	1.17627	-1.71674	3.41032
RP2O5	0.82066	0.03091	0.75331	0.88802
RSiO2	0.14757	0.02065	0.10257	0.19256

row	YOBSERVED	YFITTED	YRESIDUAL
1	95.30	94.86	0.44
2	4.46	3.42	1.04
3	0.24	1.09	-0.85
4	21.41	27.98	-6.57
5	24.32	21.09	3.23
6	54.28	50.29	3.99
7	39.08	38.94	0.14
8	34.77	35.76	-0.99
9	5.73	6.24	-0.51
10	16.07	15.90	0.17
11	4.36	4.22	0.14
12	23.35	18.00	5.35
13	47.78	50.91	-3.13
14	21.57	22.64	-1.07
15	7.29	8.67	-1.38


Residual Plot for RY
8
5
2
-1
-4
-7
0
3
6
9
12
15
Index

A P P E N D I X 5

Main analytical procedures used by CECA to characterize phosphoric ester collectors

REACTIFS DE FLOTTATION

INTRODUCTION

Les traitements de flottation des terres rares effectués au Laboratoire du BRGM permettent de tester notamment quelques esters phosphoriques (mono et diesters) synthétisés par la Société GERLAND Chimie.

Une des phases importantes au delà de la synthèse des produits concerne l'analyse des divers constituants permettant de mettre en ralation les résultats de flottation et la composition de tels réactifs.

I) METHODE D'ANALYSE DES ESTERS PHOSPHORIQUES TESTES

Les méthodes employées sont CFD 110 - A - 157 et CFD 110 - A - 163 (méthodes potentiométriques et colorimétriques).
Un rappel de ces méthodes est reproduit en annexe : ANALYSE DES REACTIFS.

Pour les produits industrialisés, les valeurs indiquées correspondent à la moyenne des différents lots synthétisés.

II) RESULTATS

Le tableau suivant indique les valeurs obtenues par ces méthodes.

III) DISCUSSION

La série D4 présente un double intérêt d'étude :

- rapport diester/monoester
- teneur en matières non phosphatées.

Les produits industriels D9 PS2 et T6 PS, proches en composition d'esters présentent l'intérêt d'étudier :

- l'influence de la viscosité du produit
- l'incidence de la teneur en pyrophosphates.

.../...

IV) CONCLUSIONS ET POURSUITE DE L'ETUDE

Bien que la méthode potentiométrique - colorimétrique soit au point et considérée comme reproductible, l'inconvénient principal reste un temps important de traitement et d'analyse.

Une étude importante débutant maintenant et ayant pour thème l'analyse HPLC des non-ioniques phosphatés fera l'objet du compte rendu suivant.

Cette méthode permettrait un temps d'analyse beaucoup plus faible que la méthode exposée dans le présent compte rendu.

ANALYSE DES REACTIFS DE FLOTTATION PAR LES METHODES
CFD 110-A-157 ET CFD 110-A-163

PRODUIT:	D9PS2	T6PS	D4-92	D4-66	D4-70E
SYNTHESE	IND.	IND.	LABO	LABO	LABO
ASPECT	PATEUX	LIQUIDE	LIQUIDE	LIQUIDE	LIQUIDE
ANALYSE:					
H3PO4 %	0.7	0.7	3.5	1.7	2.3
H4P2O7 %	0.2	0.8		0.1	0.5
MONOESTER ORTHO %	43.6	46.9	71.9	54.5	63.0
MONOESTER PYRO %	3.0	5.9		1.5	4.9
DIESTER ORTHO %	43.2	32.2	6.5	25.9	24.9
DIESTER PYRO. SYM. %	4.3	2.6		2.9	3.7
DIESTER PYRO ASSYM. %					
NON PHOPHATE %	9.4	15.6	23.2	20.3	4.5
TOTAL	104.4	104.7	105.1	106.9	103.8
RAPPORT MONO/DIESTER	50/50	60/40	92/8	66/34	70/30

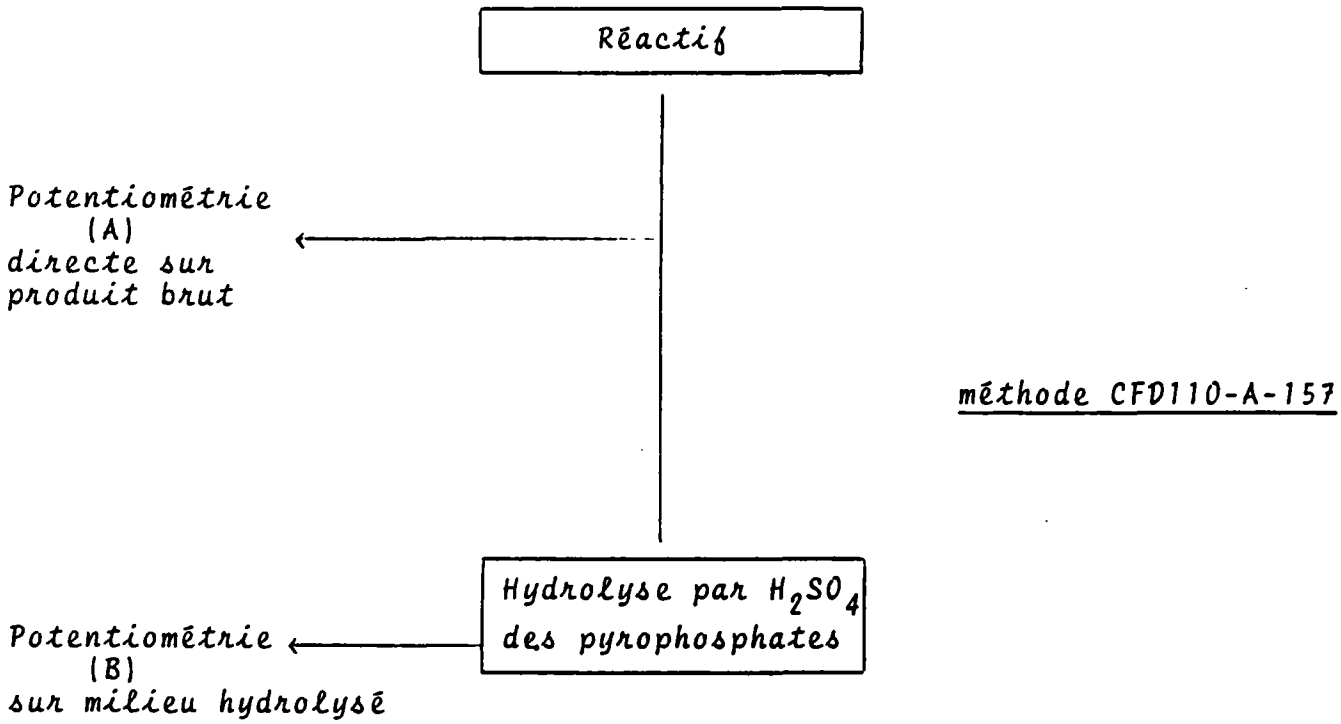
REMARQUE: LE TOTAL SUPERIEUR A 100 % EST DU A UNE INCERTITUDE EXPERIMENTALE SUR LA METHODE DE CARACTERISATION

ANALYSE DES REACTIFS

A/ Schéma de l'analyse des esters phosphoriques

1) Vérification de la présence d'esters pyrophosphoriques.

Ex: (R O P(=O)(OH)-O P(=O)(OH)-O R)



2 possibilités se présentent alors :

* A ≠ B ⟶ présence d'esters pyrophosphoriques.

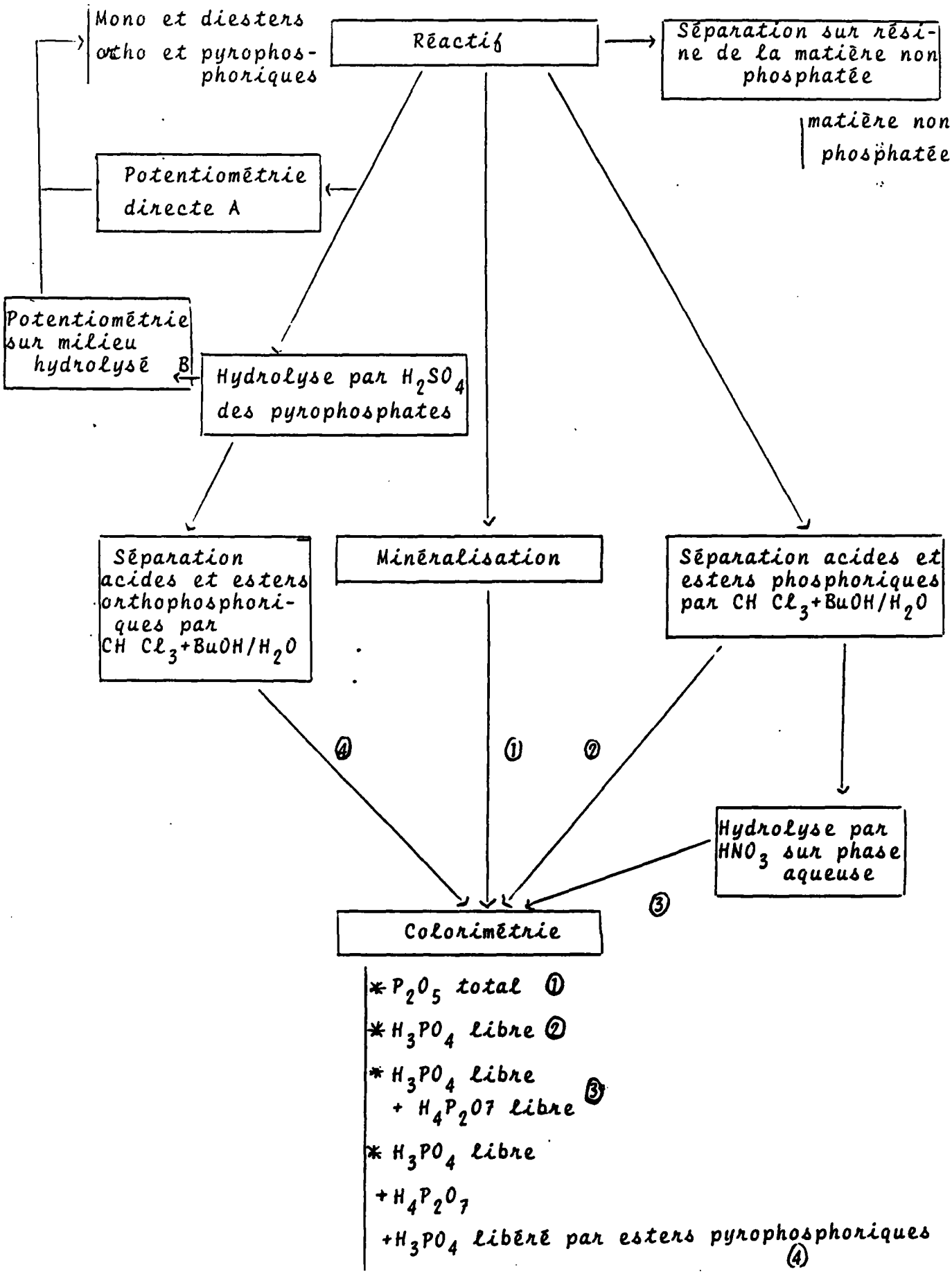
Continuation de la méthode CFD 110-A-157 (Annexe III) voir (2)

* A = B ⟶ pas d'esters pyrophosphoriques.

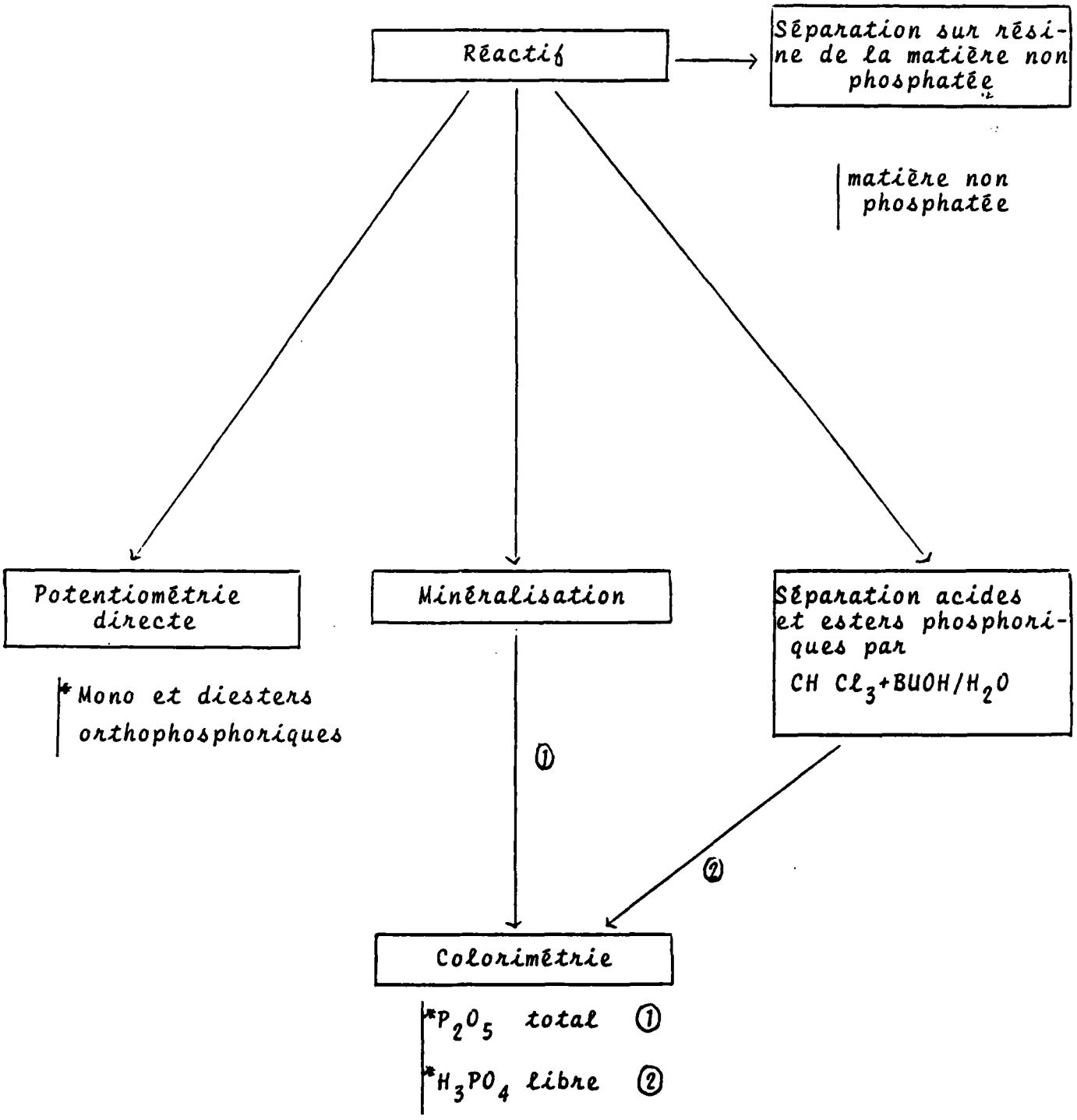
Utilisation de la méthode CFD 110-A-163 (Annexe I) voir (3)

../...

2) Présence d'esters pyrophosphoriques (CFD 110-A-157)



3) Absence d'esters pyrophosphoriques (Méthode CFD 110-A-163)



PHOSPHATES D'ALKYLES

ANALYSE DU PHOSPHATE DE STEARYLE

I - DETERMINATION DU PENTOXYDE DE PHOSPHORE TOTAL (organique et minéral)

1 . Principe

Minéralisation par digestion sulfo nitro perchlorique puis détermination colorimétrique au moyen du complexe jaune (phospho vanado molybdique).

Note 1 . L'attention des utilisateurs de la méthode est attirée sur les dangers que représente l'utilisation de l'acide perchlorique.

Notamment, ne pas minéraliser un échantillon susceptible de contenir des alcools volatils (méthanol, éthanol, isopropanol etc...)

On consultera avec profit les manuels traitant de l'utilisation de l'acide perchlorique (ex. NFT, 01 010 précautions concernant l'utilisation de l'acide perchlorique dans les laboratoires).

2. 2. Réactifs

Ils sont tous de qualité analytique reconnue

- 2-1. acide sulfurique $d\ ^{20}_{4} = 1,83$
- 2-2. acide nitrique $d\ ^{20}_{4} = 1,38$
- 2-3. acide perchlorique $d\ ^{20}_{4} = 1,615$
- 2-4. vanadate d'ammonium
- 2-5. molybdate d'ammonium-
- 2-6. phosphate monopotassique
- 2-7. solution de soude 10 N préparée et stockée dans du polyéthylène exclusivement
- 2-8. fluorure de sodium à 30 g/l
- 2-9. solution éthanolique de phénolphtaléine à 1 %

3. Matériel

Verrerie courante de laboratoire et notamment

- 3-1. matras de 100 cm3
- 3-2. fioles jaugées de 100, 250, 500 cm3
- 3-3. spectrophotomètre ou colorimètre et accessoires
- 3-4. pipettes de précision de 5, 10, 25 cm3
- 3-5. balance analytique sensible à 1/10 mg

.../...

4. Mode opératoire

4-1. Préparation des solutions

4-1.1. Solution étalon d'orthophosphate

- Solution mère

Dissoudre 7,164 g de phosphate monopotassique anhydre dans de l'eau distillée - compléter à 1000 cm3.

- Solution étalon

Diluer la solution mère au 1/50 (20 cm3 dans 1 litre).
1 cm3 de solution étalon contient :
- 0,10 mg d'ion orthophosphate

4-1.2. Solution nitro vanado molybdique

Dans 500 cm3 d'eau distillée introduire :
- métavanadate d'ammonium : 1 g
  puis
- acide nitrique : 55 cm3
  laisser dissoudre
  puis introduire :
- molybdate d'ammonium : 20 g
  laisser dissoudre puis compléter à 1 litre

4-2. Minéralisation

- Peser avec précision une prise d'essai contenant au moins 0,05 mg de phosphore et de 100 mg au minimum pour une balance au 1/10 de mg puis, l'introduire dans le matras de 100 cm3 (3-1)
- Verser sur l'échantillon avec précaution
  H_2SO_4 pur (2 cm3)
  puis le mélange nitroperchlorique (75/25 volume-volume) 25 cm3
- Porter le contenu du ballon à l'ébullition et chasser le mélange nitroperchlorique de façon à ramener au volume d'acide sulfurique (l'opération dure environ 1 h avec une prise d'essai de 100 mg).

.../...

- Refroidir puis introduire :
  eau distillée ou déminéralisée 50 cm3
  A ce stade la solution obtenue doit être incolore et limpide.
- Faire bouillir 1/2 heure.

Cette ébullition a pour but : d'hydrolyser les acides polyphosphoriques éventuellement formés et de s'assurer de la qualité de la minéralisation.
Refroidir, puis neutraliser exactement au moyen de soude 10 N en présence de phénolphtaléine.
Transvaser quantitativement le contenu du matras dans la fiole de 100 cm3 destinée à la colorimétrie (cas où la quantité de phosphore est minimale) ou dans une fiole jaugée de volume appropriée aux dilutions nécessaires (cf. paragraphe 6).

4-3. .Colorimétrie

4-3.1. Gamme étalon

Dans une série de fioles jaugées de 100 cm3 introduire :
0 - 1 - 2... 10 cm3 de solution étalon de phosphate monopotassique (4.1.1.) correspondant à :
0 - 0,1 - 0,2 ... 1 mg de PO_4^{3-}

Verser dans l'ordre :
10 cm3 de fluorure de sodium (2-8) et 25 cm3 de solution nitro vanado molybdique (4.1.2.). Agiter entre chaque introduction puis compléter à 100 cm3 par de l'eau distillée.

Note 2 : En l'absence de silice, ce qui est le cas, l'utilisation de fluorure n'est pas nécessaire, on peut l'éliminer sans inconvénient

4-3.2. Echantillon

Pour l'échantillon procéder comme ci-dessus en substituant une aliquote adéquate de l'échantillon à la solution étalon.

4-3.3. Développement du complexe

Laisser développer la coloration à la température ambiante pendant 1 heure.

.../...

Note 3 : En raison de l'influence critique de la température, placer les fioles à proximité immédiate de l'instrument de mesure pendant le temps de développement.

En l'absence de thermostatisation de la salle de mesure, veiller à ne pas modifier les conditions thermiques au cours des mesures de longue durée (mise en service d'appareils notamment, etc...)

4-3.4 - Mesure

Déterminer la densité optique à 360 nm en cuve de 1 cm des solutions étalons et des échantillons (opérer par rapport au 0 de la gamme étalon) La courbe densité optique en fonction de la quantité de phosphate doit être linéaire. Déterminer la pente de cette courbe en effectuant la moyenne arithmétique des quotiens De/P où De est la densité optique de la solution étalon et P la quantité de phosphate en milligramme correspondante.

5. Expression des résultats

$$P_2O_5\ \% = \frac{E_1 \quad 0{,}7472 \quad 100}{K \quad M_1 \quad F_1}$$

où E_1 = densité optique de l'échantillon

K = pente de la courbe d'étalonnage

M_1 = prise d'essai en milligrammes

F_1 = facteur de dilution de la prise d'essai initiale (paragraphe 6)

0,7472 : facteur de transformation de PO_4 en P_2O_5

6. Tableau de dilution pour le dosage de l'anhydride phosphorique total

Teneur en P_2O_5 présumée (%)	Volume de solution (cm3)	Aliquote mise en oeuvre (cm3)	Facteur de dilution final
1	100	50	0,5
5	100	10	0,1
10	100	5	0,05
15	250	10	0,04
20	250	5	0,02
25	250	5	0,02
30	250	5	0,02
35	500	5	0,01
40	500	5	0,01

(Ces dilutions sont calculées pour une prise d'essai de 100 mg environ)

.../...

II - DETERMINATION DE LA TENEUR EN PENTOXYDE DE PHOSPHORE MINERAL

1. Principe

Séparation par extraction liquide-liquide des phosphates d'alkyles et de l'acide phosphorique minéral. Ce dernier est déterminé au moyen de la méthode colorimétrique déjà décrite pour la détermination de l'anhydride phosphorique total.

2. Réacitfs

2-1. Solution aqueuse de nitrate d'ammonium molaire
2-2. Mélange chloroforme - n Butanol (50% V/V)
2-3. Phénolphtaléine à 1 %. dans l'éthanol
2-4. Solution aqueuse d'hydroxyde de sodium normal

3. Matériel

3-1. Tubes à centrifuger tronconiques de 10 cm3 gradués par 0,1 cm3
3-2. Centrifugeuse pour tube de 10 cm3
3-3. Pipettes de 5 cm3
3-4. Pipettes de 2 cm3
3-5. Bain marie régulé à 100°C

4. Mode opératoire

4.1. Détermination de H_3PO_4

Dans un tube à centrifuger de 10 cm3 (3-1.), peser exactement une masse d'ester phosphorique correspondant à environ 1 mg d'anhydride phosphorique à l'état de H_3PO_4. Dissoudre dans 5 cm3 de mélange chloroforme-butanol (2-3.). Ajouter 5 cm3 de solution de nitrate d'ammonium (2-1.) agiter vigoureusement le contenu du tube pendant 1 minute afin de mélanger convenablement les 2 phases. Centrifuger puis prélever 2 cm3 de la phase supérieure, les introduire dans 1 fiole jaugée de 100 cm3. Se reporter à la rubrique (I.4.3.) dosage de l'anhydride phosphorique total pour la partie colorimétrie.

.../...

5. Expression des résultats

$$H_3PO_4 \text{ en g\%g} = \frac{E_2 \cdot 1{,}032 \quad 100}{K \cdot M_2 \cdot F_2}$$

$$H_3PO_4 \text{ en mmo/1\%g} = \frac{E_2 \cdot 10{,}531 \times 100}{K \cdot M_2 \cdot F_2}$$

Formules pour lesquelles les symboles sont les suivants :

E_2 = densité optique correspondant à la colorimétrie des ortho phosphates (4-1.)

K = pente de la courbe d'étalonnage (I-4-3-5)

M_2 = masse de la prise d'essai (milligrammes) correspondant à la détermination de H_3PO_4 (4-1.)

F_2 = facteur de dilution

10,531 facteur de transformation de PO_4^{3-} en mmol de H_3PO_4

6. Tableau de dilution

P_2O_5 minéral présumé (%)	M (mg)	Prélèvement (5ème)	Volume de solution (cm3)	Aliquote (cm3)	Facteur de dilution final
0,5	200	2,0	-	-	0,4
1	100	"	-	-	0,4
2,5	100	"	100	50	0,2
5	100	"	100	25	0,1
7,5	100	"	100	20	0,08
10	100	"	100	10	0,04
15	100	"	100	10	0,04

III - DETERMINATION DE LA TENEUR EN NON-IONIQUE

1. Note 4.

Le non-ionique comprend essentiellement l'alcool non phosphaté ainsi qu'un peu d'ester phosphorique neutre. Ce dernier pouvant être déter miner éventuellement sur la base de la teneur en phosphore de la fra non-ionique. Par ailleurs, la présence de produits de déshydratation de l'alcool gras (ether-oxyde, oléfine) est également possible.

2. Principe

Fixation des esters phosphoriques acides au moyen d'une résine forte basique (hydroxyde de triméthylammonium sur matrice polystyrène divinylbenzène). macroporeuse.

3. Réactifs

3-1. Soude 2,5 N en solution aqueuse (préparée et stockée dans du polyéthylène).

3-2. Alcool isopropylique

3-3. Résine fortement basique 60 - 150 mesh (LEWATIT MP 5080 de MER

4. Appareillage

Matériel courant de laboratoire et notamment :

4-1. Capsule antigrimpante

4-2. Agitateur magnétique avec platine chauffante régulée

4-3. Thermomètre - 10 + 100°

4-4. Entonnoir fritté porosité 4 diamètre 60 mm

4-5. Fiole à vide de 500 cm3

4-6. Evaporateur rotatif sous vide ou dispositif équivalent

4-7. Balance analytique au 1/10 de mg

4-8. Béchers de 250 cm3

4-9. Ballon de 100 cm3

5. Mode opératoire

5-1. Préparation de la résine (transformation de la forme saline en forme hydroxyde)

Dans un bécher de 250 cm3 (4-8.), peser environ 25 g de résine (3-3) sous la forme commerciale(chlorure principalement), ajouter 100 cm3 de soude 2,5N (3-1.) agiter lentement pendant 1/2 heure à 20°C au moyen de l'agitateur magnétique. Filtrer la résine sur un entonnoir fritté (4-4.) sous vide. Laver abondamment par de l'eau distillée jusqu'à réaction neutre et fin d'ions chlorures dans le filtrat. Transférer ensuite la résine du filtre au bécher et effectuer un nouvel échange dans les mêmes conditions. Après la série de lavages à neutralité, pratiquer 4 rinçages par fractions de 50 cm3 d'isopropanol à 50°C. La résine est alors prête à l'emploi.

Remarque : Il est possible de préparer en une seule opération la résine nécessaire à plusieurs déterminations en effectuant l'opération ci-dessous sur une masse quelconque de résine. Cette dernière sera éventuellement stockée après le dernier lavage aqueux. Laver l'échangeur d'ions par l'isopropanol immédiatement avant usage.

5-2. - Séparation du non-ionique

Dans un bécher de 250 cm3 (4-8), peser exactement une masse d'ester correspondant à 7 meq maximum d'acidité libre (détermination à la rubrique IV) puis les dissoudre dans 100 cm3 d'isopropanol (3-2.)à 50°C Introduire alors 25 g de résine (5-1.) et agiter à 50°C pendant 1/2 heur au moyen de l'agitateur magnétique chauffant. La température est contrôlée à l'aide d'un thermomètre (4-3.) et l'évaporation du solvant limitée par l'emploi d'une capsule antigrimpante (4-1.) remplie d'eau froide et placée sur le bécher. Filtrer ensuite la résine et recueillir le filtrat dans la fiole à vide de 500 cm3 (4-5.). Pratiquer les rinçages nécessaires par 4 fois 50 cm3 d'isopropanol à 50°C. Evaporer le filtrat et peser le résidu.

.../...

6. Expression des résultats

$$NI = \frac{R_1 \times 100}{M_3}$$

R_1 = masse du résidu non ionique obtenue (grammes)

M_3 = prise d'essai d'ester- phosphoriques (grammes)

7. Remarques

En règle générale, admettre que la totalité du non ionique est constituée d'alcool gras non phosphaté, sinon déterminer la teneur en P_2O_5 de cette fraction (I) et en déduire la teneur en ester neutre (connaissant le poids moléculaire de l'alcool) et, celle de l'alcool non phosphaté par différence. Pour les faibles teneurs en non ionique on peut opérer sur des quantités plus importantes en prenant soin d'utiliser des quantités de réactifs également plus grandes.

.../...

V - DETERMINATION DE LA REPARTITION EN MONO ET DI STEARYLE PHOSPHATE

1. Principe

Interprétation de la courbe de neutralisation, en admettant que le phosphate de stearyle ne contient pas d'esters pyrophosphoriques.

Si ceux-ci sont présents en quantités notables, ils fausseront l'analyse et l bilan matière sera supérieur à 100 % (1 mole de pyrophosphate de mono octadecyle est équivalent sur le plan acidimétrique à 1 mole de mono octadécylphosphate + 1 mole de di octadécyle phosphate).

Note 5 : Point critique Les mono esters phosphoriques ont la propriété de donner des sels insolubles répondant à la formule générale $[ROPO_3H_2, ROPO_3 HNa]$ (dénomination anglo saxonne : quater salt)

La formation de ce sel lors de la détermination de l'acidité forte occasionn un surdosage du monoester phosphorique et un sous dosage du diester.

En conséquence, veillez à ce que la solution soit exempte d'insoluble lorsque le premier point équivalent est atteint.

Dans le cas contraire, recommencer la détermination en tiédissant (40-45°) ou en diluant.

2. Réactifs

2-1 Isopropanol

2-2 Tetrahydrofurane

2-3 Solution étalonnée d'hydroxyde de Sodium en solution aqueuse décinormale solution ethanolique de phenolphtaleine à 1 g/l

3. Matériel

- bechers de 400 cm3
- pH mètre
- electrodes de verre et calomel
- agitateur magnetique
- burette de 25 cm3 (graduation pour 0,05 cm3)

.../...

4. Mode opératoire

4.1 Dans un becher de 400 cm3, peser environ exactement une quantité de phosphate de stearyle correspondant à 1,25 mmol de phosphore.

Dissoudre dans 30 cm3 de tétrahydrofurane en tiédissant (40° C env.)
Diluer par 150 cm3 d'isopropanol (tiédir également si apparition de cristaux).
Ajouter 50 cm3 d'eau et titrer par la solution de soude decinormale en suivant l'évolution du pH (pH mètre - electrodes - verre - calomel) et en agitant continuellement au moyen d'un agitateur magnétique.
Tracer la courbe pH : $\mathcal{f}$ (cm3 NaOH) versé et déterminer les 2 points équivalents V_1 correspondant aux premières dissociations et V_2 correspondant aux deuxièmes dissociations.

4.2 Détermination de l'acidité correspondant aux solvants

Note 6 :

Cette acidité est une acidité faible imputable au gaz carbonique et aux acides organiques éventuellement présents dans l'isopropanol et le tetrahydrofurane.
Dans un becher de 400 cm3 introduire : 30 cm3 de tétrahydrofurane, 150 cm3 d'isopropanol, 50 cm3 d'eau, 2 à 3 gouttes de solution éthanolique de phenolphtaléine (2-4) et titrer par la solution de soude décinormale jusqu'au virage rose pâle de la solution : soit Vo

5. Expression des résultats

Acidité libre : en millequivalents % g = AL_1

$$AL_1 = \frac{t. v_1}{10\ M_4} \times 1000$$

V_1 = volume en cm3 de soude decinormale versé pour atteindre le premier point équivalent

t = titre en normalité de la soude utilisée

M_4 = poids en gramme de la prise d'essai pour la détermination de l'acidité libre et totale.

.../...

Acidité totale en millequivalents % g = AT_2

$$AT_2 = \frac{t.\ V_2 - Vo}{10\ M_4}$$

V_2 = volume en cm3 de soude décinormale versé pour atteindre le second point équivalent.

Vo = volume de soude décinormale utilisé pour l'essai témoin (acidité des solvants)

Phosphate de mono stearyle

en mmol % g = $AT_1 - AL_1 - P_1$

en g % g = $(AT_1 - AL_1 - P_1)\left\{\frac{PM_1}{1000}\right\}$

avec PM_1 = poids moléculaire moyen du phosphate de mono stearyle

Phosphate di-stearyle

en mmol % g = $2\,AL_1 - AT_1$

en g % g = $(2\,AL_1 - AT_1)\left\{\frac{PM_2}{1000}\right\}$

avec PM_2 = poids moléculaire moyen du phosphate de di-stearyle.

A N N E X E

Cette annexe décrit de façon exhaustive et selon les recommandations de l'AFNOR ([102]) la méthode générale d'analyse des orthophosphates d'alkyles telle que nous l'avons mise au point au cours de la présente étude.

I - DETERMINATION DU PENTOXYDE DE PHOSPHORE

1. Principe

Minéralisation par digestion sulfo nitro perchlorique puis détermination colorimétrique au moyen du complexe jaune (phospho vanado molybdique).

2. Réactifs

Ils sont tous de qualité analytique reconnue.

2-1. acide sulfurique $d^{20}_4 = 1,83$
2-2. acide nitrique $d^{20}_4 = 1,38$
2-3. acide perchlorique $d^{20}_4 = 1,615$
2-4. vanadate d'ammonium
2-5. molybdate d'ammonium
2-6. phosphate monopotassique
2-7. solution de soude 10 N préparée et stockée dans du polyéthylène exclusivement
2-8. fluorure de sodium à 30 g/l
2-9. solution éthanolique de phénolphtaléine à 1%.

3. Matériel

Verrerie courante de laboratoire et notamment -

3-1. matras de 100 cm3
3-2. fioles jaugées de 100, 250, 500 cm3.
3-3. spectrophotomètre ou colorimètre et accessoires
3-4. pipettes de précision de 5, 10, 25 cm3
3-5. balance analytique sensible à 1/10 mg.

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4. Mode opératoire

4.1. - Préparation des solutions

4.1.1. - Solution étalon d'orthophosphate

- Solution mère

Dissoudre 7,164 g de phosphate monopotassique anhydre dans de l'eau distillée - compléter à 1000 cm3.

- Solution étalon

Diluer la solution mère au 1/50 (20 cm3 dans 1 litre).
1 cm3 de solution étalon contient:
- 0,10 mg d'ion orthophosphate

4.1.2. - Solution nitro vanado molybdique

Dans 500 cm3 d'eau distillée introduire:
- métavanadate d'ammonium: 1 g
puis
- acide nitrique: 55 cm3
laisser dissoudre
puis introduire:
- molybdate d'ammonium: 20 g
laisser dissoudre puis compléter à 1 litre

4.2. - Minéralisation

- Peser avec précision une prise d'essai contenant au moins 0,05 mg de phosphore et de 100 mg au minimum pour une balance au 1/10 de mg puis, l'introduire dans le matras de 100 cm3 (3-1)
- Verser sur l'échantillon avec précaution
H_2SO_4 pur (2 cm3)
puis le mélange nitroperchlorique (75/25 volume-volume) 25 cm3
- Porter le contenu du ballon à l'ébullition et chasser le mélange nitroperchlorique de façon à ramener au volume d'acide sulfurique (l'opération dure environ 1 h avec une prise d'essai de 100 mg).

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- Refroidir puis introduire:
  eau distillée ou déminéralisée 50 cm3
  A ce stade la solution obtenue doit être incolore et limpide.
- Faire bouillir 1/2 heure.

Cette ébullition a pour but: d'hydrolyser les acides polyphosphoriques éventuellement formés et de s'assurer de la qualité de la minéralisation.
Refroidir, puis neutraliser exactement au moyen de soude 10 N en présence de phénolphtaléine.
Transvaser quantitativement le contenu du matras dans la fiole de 100 cm3 destinée à la colorimétrie (cas où la quantité de phosphore est minimale) ou dans une fiole jaugée de volume appropriée aux dilutions nécessaires (cf. paragraphe 6).

4.3. - Colorimétrie

4.3.1. - Gamme étalon

Dans une série de fioles jaugées de 100 cm3 introduire:
0 - 1 - 2 ... 10 cm3 de solution étalon de phosphate monopotassique (4.1.1.) correspondant à:
0 - 0,1 - 0,2 ... 1 mg de PO_4^{3-}
Verser dans l'ordre:
10 cm3 de fluorure de sodium (2-8) et 25 cm3 de solution nitro vanado molybdique (4.1.2.). Agiter entre chaque introduction puis compléter à 100 cm3 par de l'eau distillée.

4.3.2. - Echantillon

Pour l'échantillon procéder comme ci-dessus en substituant une aliquote adéquate de l'échantillon à la solution étalon.

4.3.3. - Développement du complexe

Laisser développer la coloration à la température ambiante pendant 1 heure. En raison de l'influence critique de la température, placer les fioles à proximité immédiate de l'instrument de mesure pendant le temps de développement.

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En l'absence de thermostatisation de la salle de mesure, veiller à ne pas modifier les conditions thermiques au cours des mesures de longue durée (mise en service d'appareils notamment, etc ...)

4.3.4. - Mesure

Déterminer la densité optique à 360 nm en cuve de 1 cm des solutions étalons et des échantillons (opérer par rapport au 0 de la gamme étalon) La courbe densité optique en fonction de la quantité de phosphate doit être linéaire. Déterminer la pente de cette courbe en effectuant la moyenne arithmétique des quotients $\frac{De}{P}$ où De est la densité optique de la solution étalon et P la quantité de $PO_4^{\equiv}$ en milligrammes correspondante.

5. Expression des résultats

$$P_2O_5\% = \frac{E_1 \times 0,7472 \times 100}{K \times M_1 \times F_1}$$

où E_1 = densité optique de l'échantillon

K = pente de la courbe d'étalonnage

M_1 = prise d'essai en milligrammes

F_1 = facteur de dilution de la prise d'essai initiale (paragraphe 6)

0,7472: facteur de transformation de $PO_4^{\equiv}$ en P_2O_5

6. Tableau de dilution pour le dosage de l'anhydride phosphorique total

Teneur en P_2O_5 présumée (%)	Volume de solution (cm3)	Aliquote mise en oeuvre (cm3)	Facteur de dilution final
1	100	50	0,5
5	100	10	0,1
10	100	5	0,05
15	250	10	0,04
20	250	5	0,02
25	250	5	0,02
30	250	5	0,02
35	500	5	0,01
40	500	5	0,01

(Ces dilutions sont calculées pour une prise d'essai de 100 mg environ)

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Introduire alors 25 g de résine (5-1.) et agiter à 50°C pendant 1/2 heure au moyen de l'agitateur magnétique chauffant. La température est contrôlée à l'aide d'un thermomètre (4-3.) et l'évaporation du solvant limitée par l'emploi d'une capsule antigrimpante (4-1.) remplie d'eau froide et placée sur le bécher. Filtrer ensuite la résine et recueillir le filtrat dans la fiole à vide de 500 cm3 (4-5.). Pratiquer les rinçages nécessaires par 4 fois 50 cm3 d'isopropanol à 50°C. Evaporer le filtrat et peser le résidu ainsi qu'il l'est décrit (rubrique II4.)

6. Expression des résultats

$$NI = \frac{R_2 \times 100}{M_3}$$

R_2 = masse du résidu non ionique obtenue (grammes)

M_3 = prise d'essai d'esters phosphoriques (grammes)

7. Remarques

En règle générale, admettre que la totalité du non ionique est constituée d'alcool gras non phosphaté, sinon déterminer la teneur en P_2O_5 de cette fraction (I) et en déduire la teneur en ester neutre (connaissant le poids moléculaire de l'alcool) et celle de l'alcool non phosphaté par différence. Pour les faibles teneurs en non ionique on peut opérer sur des quantités plus importantes en prenant soin d'utiliser des quantités de réactifs également plus grandes.

IV - DETERMINATION DE LA TENEUR EN PENTOXYDE DE PHOSPHORE MINERAL

1. Principe

Séparation par extraction liquide-liquide des phosphates et pyrophosphates d'alkyles et des acides phosphoriques minéraux (H_3PO_4 et $H_4P_2O_7$). Ces derniers sont déterminés au moyen de la méthode colorimétrique déjà décrite pour la détermination de l'anhydride phosphorique total.

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2. Réactifs

2-1. Solution aqueuse de nitrate d'ammonium molaire

2-2. Solution aqueuse d'acide nitrique normal

2-3. Mélange chloroforme - n Butanol (50% V/V)

2-4. Phénolphtaléine à 1 °/oo dans l'éthanol

2-5. Solution aqueuse d'hydroxyde de sodium normal

3. Matériel

3-1. Tubes à centrifuger tronconiques de 10 cm3 gradués par 0,1 cm3

3-2. Centrifugeuse pour tube de 10 cm3

3-3. Pipettes de 5 cm3

3-4. Pipettes de 2 cm3

3-5. Bain marie régulé à 100°C

4. Mode opératoire

4.1. Détermination de H_3PO_4

Dans un tube à centrifuger de 10 cm3 (3-1.), peser exactement une masse d'ester phosphorique correspondant à environ 1 mg d'anhydride phosphorique à l'état de H_3PO_4. Dissoudre dans 5 cm3 de mélange chloroforme-butanol (2-3.). Ajouter 5 cm3 de solution de nitrate d'ammonium (2-1.) agiter vigoureusement le contenu du tube pendant 1 minute afin de mélanger convenablement les 2 phases. Centrifuger puis prélever 2 cm3 de la phase supérieure, les introduire dans 1 fiole jaugée de 100 cm3. Se reporter à la rubrique dosage de l'anhydride phosphorique total pour la partie colorimétrie.

4.2. - Détermination du pentoxyde de phosphore ortho et condensé

Dans un tube à centrifuger de 10 cm3 (3-1.) peser exactement une quantité d'ester phosphorique correspondant à environ 1 mg d'anhydric phosphorique minéral total. Dissoudre dans 5 cm3 de mélange chloroforme-butanol (2-3.).

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Ajouter 5 cm3 de solution d'acide nitrique normal (2-2.). Agiter pendant 1 minute le contenu du tube afin de mélanger les 2 phases. Centrifuger puis prélever 2 cm3 de la phase supérieure qu'on introduit dans un tube à centrifuger (3-1.) et chauffer à 100°C pendant 1 heure à l'aide d'un bain marie en compensant l'évaporation par de l'eau distillée. Refroidir à 20°C, introduire 2 gouttes de phénolphtaléine (2-4) et neutraliser exactement par la solution d'hydroxyde de sodium (2-5.). Transvaser quantitativement le contenu du tube dans une fiole jaugée de 100 cm3 et pratiquer le dosage colorimétrique ainsi qu'il est décrit en première partie.

5. Expression des résultats

$$H_3PO_4 \text{ en g\%g} = \frac{E_2 . 1,032 \times 100}{K . M_4 . F_2}$$

$$H_3PO_4 \text{ en mmol\%g} = \frac{E_2 . 10,531 \times 100}{K \times M_4 \times F_2} \qquad P_1$$

$$H_4P_2O_7 \text{ en g\%g} = \frac{0,9470 . 100}{K} \left[\frac{E_3}{M_5 . F_3} - \frac{E_2}{M_4 . F_2} \right]$$

$$H_4P_2O_7 \text{ en mmol\%g} = \frac{5,265 \times 100}{K} \left[\frac{E_3}{M_5 . F_3} - \frac{E_2}{M_4 . F_2} \right] \qquad P_2$$

Formules pour lesquelles les symboles sont les suivants:

E_2 = densité optique correspondant à la colorimétrie des ortho phosphates (4-1.)

K = pente de la courbe d'étalonnage (I-4-3-5)

M_4 = masse de la prise d'essai (milligrammes) correspondant à la détermination de H_3PO_4 (4-1).

F_2 = facteur de dilution

E_3, M_5, F_3 mêmes significations mais relatives à l'anhydride phosphorique minéral total (4-2.)

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1,032 facteur de transformation de PO_4^{3-} à H_3PO_4

0,9470 " " " " à $H_4P_2O_7$

10,531 " " " " au nombre de mmol de H_3PO_4

5,265 " " " " " " " " de $H_4P_2O_7$

6. Tableau de dilution

P_2O_5 minéral présumé (%)	M (mg)	Prélèvement (5ème)	Volume de solution(cm3)	Aliquote (cm3)	Facteur de dilution final
0,5	200	2,0	-	-	0,4
1	100	"	-	-	0,4
2,5	100	"	100	50	0,2
5	100	"	100	25	0,1
7,5	100	"	100	20	0,08
10	100	"	100	10	0,04
15	100	"	100	10	0,04

V - DETERMINATION DE LA REPARTITION EN MONO ET DIESTERS ORTHO ET PYROPHOSPHORIQUES

1. Principe

On admet pour effectuer cette détermination que les phosphates d'alkyles fabriqués par phosphorylation des alcools au moyen de l'anhydride ou de l'acide polyphosphorique sont essentiellement et uniquement composés de mono et diesters ortho et pyrophosphoriques ainsi que d'acides ortho et pyrophosphorique non estérifiés. On procède par mesure des acidités libres (correspondant à la première acidité de H_3PO_4) et totale (correspondant aux 2 premières acidités phosphoriques) avant et après hydrolyse des pyrophosphates. La mesure des teneurs en P_2O_5 minéral correspondant à H_3PO_4, $H_4P_2O_7$ et H_3PO_4 libéré par l'hydrolyse des pyrophosphates de monoalkyles et de dialkyles asymétriques, est également nécessaire à l'interprétation quantitative des mesures acidimétriques.

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2. Réactifs

2-1. Dioxanne

2-2. Isopropanol

2-3. Acide sulfurique 2,4 N (122 g/l d'acide sulfurique, $d_4^{20} = 1,84$)

2-4. Solution de soude déci-normale

2-5. Phénolphtaléine à 1 °/oo dans l'alcool éthylique

2-6. Tétrahydrofurane

Réactifs nécessaires au dosage de P_2O_5 forme minérale (se reporter à IV - 2.)

3. Matériel

3-1. Ballons de 100 cm3

3-2. Réfrigérant à air adaptable sur le ballon de 100 cm3

3-3. Chauffe-ballon de 100cm3 ou bain-marie

3-4. Pipettes de précision de 2, 5, 10, 20 et 25 cm3

3-5. Fioles jaugées de 100, 200 ou 250 cm3

3-6. pH mètre

3-7. Electrodes de verre et calomel

3-8. Béchers de 400 cm3

3-9. Agitateur magnétique -3 -10 burette de 25 cm3 (graduation par 0,05 cm3)

4. Modes opératoires

4-1. Détermination de l'acidité libre et totale avant hydrolyse des pyrophosphates

Dans un bécher de 400 cm3 (3-8.) peser environ exactement une quantité de phosphate d'alkyle correspondant à 1,25 mmol de phosphore, puis dissoudre dans 200 cm3 d'eau et titrer par la soude déci-normale (2-4.) en suivant le dosage par pH-mètre (électrodes verre-calomel 3-7.) et en agitant continuellement la solution au moyen de l'agitateur magnétique (3-9.). Tracer la courbe pH = f (cm3) NaOH versé et déterminer les 2 points équivalents.

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4.2. Remarques

4.2.1. - Dans le cas des phosphates d'alkyles insolubles dans l'eau, dissoudre la prise d'essai dans 10 cm3 de tétrahydrofurane anhydre (2-6.) ajouter 150 cm3 d'isopropanol (2-2.) puis 50 cm3 d'eau et titrer. La consommation de NaOH liée à l'utilisation d'isopropanol doit être connue (cf. 4-6.)

4.2.2. - Dans tous les cas, titrer rapidement après la mise en solution dans les solvants protiques (iso PrOH et eau) afin d'éviter l'hydrolyse ou l'alcoolyse des pyrophosphates. Ne chauffer en aucun cas en ces milieux solvants pour faciliter la mise en solution (utiliser comme prévu ci-dessus la mise en solution préalable dans le TH

4-3. Calcul des acidités libre et totale avant hydrolyse

$$\text{Acidité libre (meq \% g)} = \frac{100 \times \Theta \times N_1}{M_6} \qquad AL_1$$

M_6 = masse de la prise d'essai (grammes)

Θ = titre de la soude en normalité

N_1 = volume de soude versé pour atteindre le premier point équivalen

Acidité totale (meq % g)

$$\frac{100 \times \Theta \times \left[N_2 + V_0 - V_1 \right]}{M_6} \qquad AT_1$$

N_2 : volume de NaOH versé au deuxième point équivalent

V_0 et V_1 : volume de NaOH utilisé pour les témoins (cf. 4-6.)

4-4. Détermination de l'acidité libre et totale après hydrolyse des pyrophosphates

Dans un ballon de 100 cm3, peser exactement une masse d'ester phosphorique correspondant à 12,5 mmol de phosphore, dissoudre dans 20 d'eau et ajouter exactement 10 cm3 d'acide sulfurique 2,4 N (2-3). Adapter le réfrigérant à air et chauffer à l'ébullition pendant 1h3 Refroidir, puis transvaser quantitativement dans une fiole jaugée d 100 cm3 et compléter à la marque, par de l'eau.

Prélever 10 cm3 de cette solution et les introduire dans le bécher de 400 cm3, ajouter 170 cm3 d'eau et 25 cm3 exactement de soude décinormale (2-4.). Agiter la solution et titrer par NaOH 0,1 N (2-4.) en suivant le dosage au pH mètre. Tracer la courbe pH = f(cm3) NaOH versé. Déterminer les 2 points équivalents.

4.5. - Remarque

Pour les phosphates d'alkyles insolubles dans l'eau, effectuer l'hydrolyse avec 20 cm3 de dioxanne (2-1.) au lieu de 20 cm3 d'eau et après celle-ci transvaser dans une fiole de volume plus important si nécessaire (200 ou 250 cm3) avec de l'isopropanol (2-2.) puis compléter à la marque avec ce solvant. Prélever une aliquote d'un dixième (20 ou 25 cm3) pour effectuer le dosage titrimétrique en tenant compt de la remarque 4.2.1..

4.6. - Témoins

4.6.1. - Introduire exactement 10 cm3 d'acide sulfurique 2,4 N et 20 cm3 d'eau (ou de dioxanne) dans un ballon de 100 cm3. Chauffer à l'ébullition pendant 1h30. Refroidir puis transvaser quantitativemer dans une fiole jaugée de 100 cm3, compléter à la marque par de l'eau Sur une aliquote d'un dixième (10 cm3) titrer par NaOH, 0,1 N en présence de quelques gouttes de phénolphtaléine (2-5.). Soit V_0 le volume de NaOH versé pour obtenir le virage de la phénolphtaléine.

4.6.2. - Dans le cas de l'utilisation d'isopropanol, effectuer sur une autre aliquote d'un dixième (10 cm3) une détermination en présen de 150 cm3 d'isopropanol (2-2.). Soit V_1, le volume de NaOH versé pour obtenir le virage de la phénolphtaléine.

4.7. - Calcul de l'acidité libre et totale après hydrolyse des pyrophosphates

Acidité libre en meq % g

$$\frac{1000 \times \theta\ (N_3 + 25 - V_0)}{M_7} \qquad AL_2$$

./.

N_3 = volume de NaOH versé au premier point équivalent
M_7 = masse de la prise d'essai (grammes)
V_0 = volume de NaOH utilisé pour le témoin 4-6-1

Acidité totale (meq % g)
Pour les phosphates d'alkyles hydrosolubles

$$\frac{1000 \times \theta \times (N_4 + 25 - V_0)}{M_7} \qquad AT_2$$

Pour les phosphates d'alkyles solubilisés dans l'isopropanol

$$\frac{1000 \times \theta \times (N_4 + 25 - V_1)}{M_7} \qquad AT_2$$

N_4 = volume de NaOH versé au 2ème point équivalent
V_1 = volume de NaOH utilisé pour le témoin 4-6-2

4.8. - Détermination de l'acide orthophosphorique après hydrolyse

Prélever exactement 2 cm3 de la solution d'ester phosphorique après hydrolyse sulfurique (4.4.) et les introduire dans un tube à centrifuger de 10 cm3. Ajouter 2 cm3 de chloroforme butanol et 5 cm3 de nitrate d'ammonium molaire. Agiter vigoureusement le contenu du tube afin de mélanger les 2 phases. Centrifuger et prélever 2 cm3 de la phase aqueuse supérieure. Introduire dans une fiole jaugée de 100 cm neutraliser exactement par de la soude 0,1 N en présence de phénolphtaléine. Doser colorimétriquement H_3PO_4 (se reporter à I.4-3).

4.9. Remarques

4.9.1. - En fonction de la teneur en acide phosphorique et de la pris d'essai initiale, les volumes ci-dessus peuvent être modifiés. Toutefois, le volume total ne saurait excéder 10 cm3.

4.9.2. - Lors de l'utilisation d'isopropanol, celui-ci se partageant entre les 2 phases, noter le volume de la phase aqueuse supérieure afin d'en tenir compte dans les calculs.

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4.10. - Calcul de la teneur en acide phosphorique après hydrolyse

H_3PO_4 (mmol % g)

$$\frac{E_4 \times 10,531 \times V_f \times Va}{K \times 40 \times M_8} \qquad (P3)$$

E_4: densité optique à 360 nm

K : pente de la courbe d'étalonnage

V_f: volume auquel a été amenée la prise d'essai après hydrolyse

Va: volume de la phase aqueuse lors de l'extraction de H_3PO_4

4.11. - Expressions des résultats

4.11.1. - Teneur en pyrophosphate de dialkyles asymétrique

$$AL_2 - AL_1 \qquad \text{(mmol \% g)} \qquad (Z)$$

$$\frac{Z}{1000} \times PM_Z \qquad \text{(g \% g)}$$

PM_Z = poids molaire du pyrophosphate de dialkyle asymétrique

4.11.2. - Teneur en pyrophosphate de monoalkyle

$$P_3 - \left[P_1 + 2\,P_2 + Z\right] \qquad \text{(mmol \% g)} \qquad (V)$$

$$\frac{V}{1000} \cdot PM_V \qquad \text{(g \% g)}$$

PM_V = poids du pyrophosphate de monoalkyle

4.11.3. - Teneur en pyrophosphate de dialkyle symétrique

$$\frac{\Delta - (P_2 + V)}{2} \qquad \text{(mmol \% g)} \qquad (W)$$

$$\frac{W \cdot PM_W}{1000} \qquad \text{(g \% g)}$$

PM_W = poids molaire du pyrophosphate de dialkyle symétrique ($\neq PM_Z$)

$\Delta = (AT_2 + AL_1) - (AT_1 + AL_2)$

./.

4.11.4. - Teneur en ortho phosphate de monoalkyle

$$AT_1 - \left[AL_1 + P_1 + P_2 + Z + V\right] \qquad \text{(mmol \% g)} \quad \text{(S)}$$

$$\frac{S \times PM_S}{1000} \qquad \text{(g \% g)}$$

PM_S = poids molaire de l'orthophosphate de monoalkyle

4.11.5. - Teneur en orthophosphate de dialkyle

$$AL_1 - \left[P_1 + 2\,P_2 + Z + 2\,W + S + V\right] \qquad \text{(mmol \% g)} \quad \text{(T)}$$

$$\frac{T \cdot PM_T}{1000} \qquad \text{(g \% g)}$$

PM_T = poids molaire de l'orthophosphate de dialkyle

P_1 et P_2 sont définis en rubrique IV-5.

APPENDIX 6

Correlation matrices relevant to characteristics of phosphoric ester collectors and flotation performances

REO ore sample 1

Sample Correlations

	NbC	n	MEO	DEO	NPP	FOPA
NbC	1.0000	.0000	-.2591	.1047	-.0866	-.4198
	(8)	(8)	(8)	(8)	(8)	(8)
	.0000	1.0000	.5355	.8052	.8385	.3004
n	.0000	1.0000	-.0877	.3271	.0199	-.3804
	(8)	(8)	(8)	(8)	(8)	(8)
	1.0000	.0000	.8363	.4291	.9627	.3526
MEO	-.2591	-.0877	1.0000	-.9264	.5502	.9000
	(8)	(8)	(8)	(8)	(8)	(8)
	.5355	.8363	.0000	.0009	.1576	.0023
DEO	.1047	.3271	-.9264	1.0000	-.6521	-.9210
	(8)	(8)	(8)	(8)	(8)	(8)
	.8052	.4291	.0009	.0000	.0797	.0012
NPP	-.0866	.0199	.5502	-.6521	1.0000	.4671
	(8)	(8)	(8)	(8)	(8)	(8)
	.8385	.9627	.1576	.0797	.0000	.2432
FOPA	-.4198	-.3804	.9000	-.9210	.4671	1.0000
	(8)	(8)	(8)	(8)	(8)	(8)
	.3004	.3526	.0023	.0012	.2432	.0000
FPPA	.4526	-.2925	-.6884	.5489	-.7211	-.5374
	(8)	(8)	(8)	(8)	(8)	(8)
	.2601	.4820	.0590	.1589	.0435	.1696
MEP	.0744	-.0372	-.5783	.5398	-.6891	-.4389
	(8)	(8)	(8)	(8)	(8)	(8)
	.8611	.9303	.1332	.1673	.0587	.2766
DEP	.1628	-.3521	-.7263	.5808	-.7396	-.4514
	(8)	(8)	(8)	(8)	(8)	(8)
	.7000	.3923	.0413	.1311	.0360	.2616
tP2O5FC	.1200	-.9518	.2892	-.5021	.0952	.4787
	(8)	(8)	(8)	(8)	(8)	(8)
	.7771	.0003	.4872	.2048	.8225	.2301
RP2O5FC	.0829	.8743	.0894	.1912	-.0696	-.2854
	(8)	(8)	(8)	(8)	(8)	(8)
	.8453	.0045	.8333	.6502	.8699	.4932
EP2O5FC	.2663	-.5944	.5006	-.5618	.0937	.4341
	(8)	(8)	(8)	(8)	(8)	(8)
	.5238	.1202	.2064	.1473	.8254	.2825
SP2O5FC	.1179	-.9521	.2897	-.5025	.0956	.4798
	(8)	(8)	(8)	(8)	(8)	(8)
	.7809	.0003	.4864	.2044	.8218	.2289
tP2O5F4	.1557	-.9289	.2512	-.4642	.0940	.4366
	(8)	(8)	(8)	(8)	(8)	(8)
	.7128	.0009	.5485	.2466	.8248	.2795

Coefficient (sample size) significance level

	FPPA	MEP	DEP	tP2O5FC	RP2O5FC	EP2O5FC
NbC	.4526	.0744	.1628	.1200	.0829	.2663
	(8)	(8)	(8)	(8)	(8)	(8)
	.2601	.8611	.7000	.7771	.8453	.5238
n	-.2925	-.0372	-.3521	-.9518	.8743	-.5944
	(8)	(8)	(8)	(8)	(8)	(8)
	.4820	.9303	.3923	.0003	.0045	.1202
MEO	-.6884	-.5783	-.7263	.2892	.0894	.5006
	(8)	(8)	(8)	(8)	(8)	(8)
	.0590	.1332	.0413	.4872	.8333	.2064
DEO	.5489	.5398	.5808	-.5021	.1912	-.5618
	(8)	(8)	(8)	(8)	(8)	(8)
	.1589	.1673	.1311	.2048	.6502	.1473
NPP	-.7211	-.6891	-.7396	.0952	-.0696	.0937
	(8)	(8)	(8)	(8)	(8)	(8)
	.0435	.0587	.0360	.8225	.8699	.8254
FOPA	-.5374	-.4389	-.4514	.4787	-.2854	.4341
	(8)	(8)	(8)	(8)	(8)	(8)
	.1696	.2766	.2616	.2301	.4932	.2825
FPPA	1.0000	.8428	.8501	.1507	-.2445	-.0045
	(8)	(8)	(8)	(8)	(8)	(8)
	.0000	.0086	.0075	.7216	.5594	.9916
MEP	.8428	1.0000	.7619	-.1326	.0205	-.1882
	(8)	(8)	(8)	(8)	(8)	(8)
	.0086	.0000	.0280	.7542	.9615	.6553
DEP	.8501	.7619	1.0000	.1570	-.3807	-.1296
	(8)	(8)	(8)	(8)	(8)	(8)
	.0075	.0280	.0000	.7105	.3521	.7597
tP2O5FC	.1507	-.1326	.1570	1.0000	-.7321	.7942
	(8)	(8)	(8)	(8)	(8)	(8)
	.7216	.7542	.7105	.0000	.0389	.0186
RP2O5FC	-.2445	.0205	-.3807	-.7321	1.0000	-.1682
	(8)	(8)	(8)	(8)	(8)	(8)
	.5594	.9615	.3521	.0389	.0000	.6906
EP2O5FC	-.0045	-.1882	-.1296	.7942	-.1682	1.0000
	(8)	(8)	(8)	(8)	(8)	(8)
	.9916	.6553	.7597	.0186	.6906	.0000
SP2O5FC	.1492	-.1339	.1568	1.0000	-.7330	.7933
	(8)	(8)	(8)	(8)	(8)	(8)
	.7244	.7520	.7107	.0000	.0386	.0188
tP2O5F4	.1047	-.2299	.1627	.9830	-.7423	.7609
	(8)	(8)	(8)	(8)	(8)	(8)
	.8051	.5839	.7003	.0000	.0349	.0283

	SP2O5FC	tP2O5F4	RP2O5F4	EP2O5F4	SP2O5F4	tP2O5F2
NbC	.1179	.1557	.4474	.3506	.1532	.1368
	(8)	(8)	(8)	(8)	(8)	(8)
	.7809	.7128	.2663	.3945	.7172	.7467
n	-.9521	-.9289	.1737	-.6005	-.9275	-.9196
	(8)	(8)	(8)	(8)	(8)	(8)
	.0003	.0009	.6808	.1154	.0009	.0012
MEO	.2897	.2512	.2108	.3228	.2540	.2865
	(8)	(8)	(8)	(8)	(8)	(8)
	.4864	.5485	.6163	.4355	.5438	.4915
DEO	-.5025	-.4642	-.0776	-.3888	-.4659	-.4879
	(8)	(8)	(8)	(8)	(8)	(8)
	.2044	.2466	.8552	.3412	.2446	.2200
NPP	.0956	.0940	-.0632	.0190	.0943	.0624
	(8)	(8)	(8)	(8)	(8)	(8)
	.8218	.8248	.8817	.9644	.8242	.8834
FOPA	.4798	.4366	-.1424	.2572	.4390	.4485
	(8)	(8)	(8)	(8)	(8)	(8)
	.2289	.2795	.7366	.5385	.2766	.2650
FPPA	.1492	.1047	.0030	.0146	.1007	.2454
	(8)	(8)	(8)	(8)	(8)	(8)
	.7244	.8051	.9943	.9726	.8124	.5580
MEP	-.1339	-.2299	-.0351	-.2860	-.2328	.0249
	(8)	(8)	(8)	(8)	(8)	(8)
	.7520	.5839	.9342	.4923	.5790	.9534
DEP	.1568	.1627	-.2775	-.0479	.1611	.1606
	(8)	(8)	(8)	(8)	(8)	(8)
	.7107	.7003	.5057	.9103	.7031	.7040
tP2O5FC	1.0000	.9830	.0650	.7886	.9825	.9681
	(8)	(8)	(8)	(8)	(8)	(8)
	.0000	.0000	.8784	.0200	.0000	.0001
RP2O5FC	-.7330	-.7423	.5917	-.2236	-.7402	-.6452
	(8)	(8)	(8)	(8)	(8)	(8)
	.0386	.0349	.1223	.5945	.0357	.0840
EP2O5FC	.7933	.7609	.6271	.9437	.7618	.8258
	(8)	(8)	(8)	(8)	(8)	(8)
	.0188	.0283	.0961	.0004	.0280	.0115
SP2O5FC	1.0000	.9833	.0631	.7880	.9827	.9676
	(8)	(8)	(8)	(8)	(8)	(8)
	.0000	.0000	.8820	.0202	.0000	.0001
tP2O5F4	.9833	1.0000	.0348	.8094	1.0000	.9089
	(8)	(8)	(8)	(8)	(8)	(8)
	.0000	.0000	.9349	.0149	.0000	.0018

	RP2O5F2	EP2O5F2	SP2O5F2	MDRATIO
NbC	-.1827	-.0962	.1345	-.2335
	(8)	(8)	(8)	(8)
	.6649	.8208	.7508	.5778
n	.9610	.4742	-.9204	-.2109
	(8)	(8)	(8)	(8)
	.0001	.2352	.0012	.6161
MEO	.0275	.4626	.2872	.8081
	(8)	(8)	(8)	(8)
	.9485	.2484	.4904	.0152
DEO	.2437	-.2619	-.4885	-.8972
	(8)	(8)	(8)	(8)
	.5608	.5309	.2193	.0025
NPP	.0084	.1397	.0631	.6610
	(8)	(8)	(8)	(8)
	.9842	.7414	.8820	.0743
FOPA	-.2442	.1818	.4499	.8926
	(8)	(8)	(8)	(8)
	.5600	.6666	.2633	.0029
FPPA	-.3143	-.2286	.2439	-.5779
	(8)	(8)	(8)	(8)
	.4483	.5861	.5605	.1335
MEP	.0565	.1311	.0236	-.5477
	(8)	(8)	(8)	(8)
	.8943	.7569	.9557	.1600
DEP	-.3750	-.4814	.1603	-.5286
	(8)	(8)	(8)	(8)
	.3600	.2271	.7046	.1780
tP2O5FC	-.9212	-.3243	.9686	.2962
	(8)	(8)	(8)	(8)
	.0012	.4333	.0001	.4763
RP2O5FC	.8948	.7386	-.6467	-.2915
	(8)	(8)	(8)	(8)
	.0027	.0364	.0831	.4835
EP2O5FC	-.5367	.1886	.8252	.1747
	(8)	(8)	(8)	(8)
	.1703	.6547	.0117	.6790
SP2O5FC	-.9214	-.3256	.9681	.2972
	(8)	(8)	(8)	(8)
	.0011	.4313	.0001	.4747
tP2O5F4	-.9391	-.4429	.9096	.2817
	(8)	(8)	(8)	(8)
	.0005	.2718	.0017	.4991

Sample Correlations

	NbC	n	MEO	DEO	NPP	FOPA
RP2O5F4	.4474	.1737	.2108	-.0776	-.0632	-.1424
	(8)	(8)	(8)	(8)	(8)	(8)
	.2663	.6808	.6163	.8552	.8817	.7366
EP2O5F4	.3506	-.6005	.3228	-.3888	.0190	.2572
	(8)	(8)	(8)	(8)	(8)	(8)
	.3945	.1154	.4355	.3412	.9644	.5385
SP2O5F4	.1532	-.9275	.2540	-.4659	.0943	.4390
	(8)	(8)	(8)	(8)	(8)	(8)
	.7172	.0009	.5438	.2446	.8242	.2766
tP2O5F2	.1368	-.9196	.2865	-.4879	.0624	.4485
	(8)	(8)	(8)	(8)	(8)	(8)
	.7467	.0012	.4915	.2200	.8834	.2650
RP2O5F2	-.1827	.9610	.0275	.2437	.0084	-.2442
	(8)	(8)	(8)	(8)	(8)	(8)
	.6649	.0001	.9485	.5608	.9842	.5600
EP2O5F2	-.0962	.4742	.4626	-.2619	.1397	.1818
	(8)	(8)	(8)	(8)	(8)	(8)
	.8208	.2352	.2484	.5309	.7414	.6666
SP2O5F2	.1345	-.9204	.2872	-.4885	.0631	.4499
	(8)	(8)	(8)	(8)	(8)	(8)
	.7508	.0012	.4904	.2193	.8820	.2633
MDRATIO	-.2335	-.2109	.8081	-.8972	.6610	.8926
	(8)	(8)	(8)	(8)	(8)	(8)
	.5778	.6161	.0152	.0025	.0743	.0029

Coefficient (sample size) significance level

	FPPA	MEP	DEP	tP2O5FC	RP2O5FC	EP2O5FC
RP2O5F4	.0030	-.0351	-.2775	.0650	.5917	.6271
	(8)	(8)	(8)	(8)	(8)	(8)
	.9943	.9342	.5057	.8784	.1223	.0961
EP2O5F4	.0146	-.2860	-.0479	.7886	-.2236	.9437
	(8)	(8)	(8)	(8)	(8)	(8)
	.9726	.4923	.9103	.0200	.5945	.0004
SP2O5F4	.1007	-.2328	.1611	.9825	-.7402	.7618
	(8)	(8)	(8)	(8)	(8)	(8)
	.8124	.5790	.7031	.0000	.0357	.0280
tP2O5F2	.2454	.0249	.1606	.9681	-.6452	.8258
	(8)	(8)	(8)	(8)	(8)	(8)
	.5580	.9534	.7040	.0001	.0840	.0115
RP2O5F2	-.3143	.0565	-.3750	-.9212	.8948	-.5367
	(8)	(8)	(8)	(8)	(8)	(8)
	.4483	.8943	.3600	.0012	.0027	.1703
EP2O5F2	-.2286	.1311	-.4814	-.3243	.7386	.1886
	(8)	(8)	(8)	(8)	(8)	(8)
	.5861	.7569	.2271	.4333	.0364	.6547
SP2O5F2	.2439	.0236	.1603	.9686	-.6467	.8252
	(8)	(8)	(8)	(8)	(8)	(8)
	.5605	.9557	.7046	.0001	.0831	.0117
MDRATIO	-.5779	-.5477	-.5286	.2962	-.2915	.1747
	(8)	(8)	(8)	(8)	(8)	(8)
	.1335	.1600	.1780	.4763	.4835	.6790

	SP2O5FC	tP2O5F4	RP2O5F4	EP2O5F4	SP2O5F4	tP2O5F2
RP2O5F4	.0631	.0348	1.0000	.5938	.0355	.1710
	(8)	(8)	(8)	(8)	(8)	(8)
	.8820	.9349	.0000	.1207	.9335	.6857
EP2O5F4	.7880	.8094	.5938	1.0000	.8106	.7587
	(8)	(8)	(8)	(8)	(8)	(8)
	.0202	.0149	.1207	.0000	.0147	.0291
SP2O5F4	.9827	1.0000	.0355	.8106	1.0000	.9075
	(8)	(8)	(8)	(8)	(8)	(8)
	.0000	.0000	.9335	.0147	.0000	.0018
tP2O5F2	.9676	.9089	.1710	.7587	.9075	1.0000
	(8)	(8)	(8)	(8)	(8)	(8)
	.0001	.0018	.6857	.0291	.0018	.0000
RP2O5F2	-.9214	-.9391	.1908	-.6167	-.9375	-.8466
	(8)	(8)	(8)	(8)	(8)	(8)
	.0011	.0005	.6508	.1034	.0006	.0080
EP2O5F2	-.3256	-.4429	.6082	-.0588	-.4422	-.1377
	(8)	(8)	(8)	(8)	(8)	(8)
	.4313	.2718	.1096	.8900	.2726	.7451
SP2O5F2	.9681	.9096	.1689	.7583	.9082	1.0000
	(8)	(8)	(8)	(8)	(8)	(8)
	.0001	.0017	.6892	.0292	.0018	.0000
MDRATIO	.2972	.2817	-.3209	.0269	.2831	.2363
	(8)	(8)	(8)	(8)	(8)	(8)
	.4747	.4991	.4384	.9496	.4969	.5732

	RP2O5F2	EP2O5F2	SP2O5F2	MDRATIO
RP2O5F4	.1908	.6082	.1689	-.3209
	(8)	(8)	(8)	(8)
	.6508	.1096	.6892	.4384
EP2O5F4	-.6167	-.0588	.7583	.0269
	(8)	(8)	(8)	(8)
	.1034	.8900	.0292	.9496
SP2O5F4	-.9375	-.4422	.9082	.2831
	(8)	(8)	(8)	(8)
	.0006	.2726	.0018	.4969
tP2O5F2	-.8466	-.1377	1.0000	.2363
	(8)	(8)	(8)	(8)
	.0080	.7451	.0000	.5732
RP2O5F2	1.0000	.6425	-.8473	-.1608
	(8)	(8)	(8)	(8)
	.0000	.0858	.0079	.7037
EP2O5F2	.6425	1.0000	-.1390	.0555
	(8)	(8)	(8)	(8)
	.0858	.0000	.7427	.8962
SP2O5F2	-.8473	-.1390	1.0000	.2376
	(8)	(8)	(8)	(8)
	.0079	.7427	.0000	.5710
MDRATIO	-.1608	.0555	.2376	1.0000
	(8)	(8)	(8)	(8)
	.7037	.8962	.5710	.0000

REO ore sample 2

Sample Correlations

	NbC	n	MEO	DEO	NPP	FOPA
NbC	1.0000	.8112	-.2266	-.5608	.7829	-.2433
	(9)	(9)	(9)	(9)	(9)	(9)
	.0000	.0080	.5577	.1162	.0126	.5281
n	.8112	1.0000	-.1985	-.3329	.6945	-.3613
	(9)	(9)	(9)	(9)	(9)	(9)
	.0080	.0000	.6087	.3813	.0379	.3395
MEO	-.2266	-.1985	1.0000	-.5952	.1941	.8674
	(9)	(9)	(9)	(9)	(9)	(9)
	.5577	.6087	.0000	.0909	.6167	.0024
DEO	-.5608	-.3329	-.5952	1.0000	-.7744	-.6381
	(9)	(9)	(9)	(9)	(9)	(9)
	.1162	.3813	.0909	.0000	.0143	.0644
NPP	.7829	.6945	.1941	-.7744	1.0000	.1398
	(9)	(9)	(9)	(9)	(9)	(9)
	.0126	.0379	.6167	.0143	.0000	.7198
FOPA	-.2433	-.3613	.8674	-.6381	.1398	1.0000
	(9)	(9)	(9)	(9)	(9)	(9)
	.5281	.3395	.0024	.0644	.7198	.0000
FPPA	.2282	-.0732	-.6829	.2021	-.2327	-.4279
	(9)	(9)	(9)	(9)	(9)	(9)
	.5547	.8516	.0426	.6021	.5468	.2506
MEP	.1202	.0352	-.5738	.2524	-.2741	-.3490
	(9)	(9)	(9)	(9)	(9)	(9)
	.7581	.9283	.1062	.5123	.4754	.3573
DEP	-.3674	-.5377	-.5833	.6197	-.7190	-.3069
	(9)	(9)	(9)	(9)	(9)	(9)
	.3307	.1354	.0992	.0751	.0290	.4218
tREOF2	.5206	.8288	.1081	-.2904	.3866	-.1250
	(9)	(9)	(9)	(9)	(9)	(9)
	.1507	.0058	.7919	.4484	.3041	.7486
RREOF2	.5183	.7714	-.3162	.0289	.2628	-.5786
	(9)	(9)	(9)	(9)	(9)	(9)
	.1529	.0149	.4071	.9411	.4944	.1026
EREOF2	.5908	.8690	-.2581	-.0699	.3456	-.4919
	(9)	(9)	(9)	(9)	(9)	(9)
	.0939	.0024	.5026	.8581	.3622	.1786
SREOF2	.5101	.8134	.1365	-.3096	.3830	-.0687
	(9)	(9)	(9)	(9)	(9)	(9)
	.1606	.0077	.7262	.4175	.3090	.8606
tREOFC	.3771	.7282	.0692	-.1267	.2602	-.2761
	(9)	(9)	(9)	(9)	(9)	(9)
	.3170	.0261	.8595	.7453	.4989	.4720

Coefficient (sample size) significance level

	FPPA	MEP	DEP	tREOF2	RREOF2	EREOF2
NbC	.2282	.1202	-.3674	.5206	.5183	.5908
	(9)	(9)	(9)	(9)	(9)	(9)
	.5547	.7581	.3307	.1507	.1529	.0939
n	-.0732	.0352	-.5377	.8288	.7714	.8690
	(9)	(9)	(9)	(9)	(9)	(9)
	.8516	.9283	.1354	.0058	.0149	.0024
MEO	-.6829	-.5738	-.5833	.1031	-.3162	-.2581
	(9)	(9)	(9)	(9)	(9)	(9)
	.0426	.1062	.0992	.7919	.4071	.5026
DEO	.2021	.2524	.6197	-.2904	.0289	-.0699
	(9)	(9)	(9)	(9)	(9)	(9)
	.6021	.5123	.0751	.4484	.9411	.8581
NPP	-.2327	-.2741	-.7190	.3866	.2628	.3456
	(9)	(9)	(9)	(9)	(9)	(9)
	.5468	.4754	.0290	.3041	.4944	.3622
FOPA	-.4279	-.3490	-.3069	-.1250	-.5786	-.4919
	(9)	(9)	(9)	(9)	(9)	(9)
	.2506	.3573	.4218	.7486	.1026	.1786
FPPA	1.0000	.8381	.6640	-.2398	.0422	-.0188
	(9)	(9)	(9)	(9)	(9)	(9)
	.0000	.0048	.0511	.5343	.9141	.9617
MEP	.8381	1.0000	.6176	-.0619	.0799	.0718
	(9)	(9)	(9)	(9)	(9)	(9)
	.0048	.0000	.0764	.8742	.8380	.8544
DEP	.6640	.6176	1.0000	-.5843	-.2028	-.3100
	(9)	(9)	(9)	(9)	(9)	(9)
	.0511	.0764	.0000	.0985	.6007	.4169
tREOF2	-.2398	-.0619	-.5843	1.0000	.7179	.8484
	(9)	(9)	(9)	(9)	(9)	(9)
	.5343	.8742	.0985	.0000	.0294	.0038
RREOF2	.0422	.0799	-.2028	.7179	1.0000	.9708
	(9)	(9)	(9)	(9)	(9)	(9)
	.9141	.8380	.6007	.0294	.0000	.0000
EREOF2	-.0188	.0718	-.3100	.8484	.9708	1.0000
	(9)	(9)	(9)	(9)	(9)	(9)
	.9617	.8544	.4169	.0038	.0000	.0000
SREOF2	-.2772	-.0699	-.5722	.9935	.6741	.8189
	(9)	(9)	(9)	(9)	(9)	(9)
	.4702	.8581	.1074	.0000	.0464	.0069
tREOFC	-.1427	.0538	-.5205	.8855	.7846	.8319
	(9)	(9)	(9)	(9)	(9)	(9)
	.7142	.8906	.1508	.0015	.0123	.0054

	SREOF2	tREOFC	RREOFC	EREOFC	SREOFC	tREOF4
NbC	.5101	.3771	.4365	.4607	.3681	-.2926
	(9)	(9)	(9)	(9)	(9)	(9)
	.1606	.3170	.2401	.2120	.3297	.4448
n	.8134	.7282	.7455	.8012	.7321	-.0731
	(9)	(9)	(9)	(9)	(9)	(9)
	.0077	.0261	.0211	.0094	.0249	.8517
MEO	.1365	.0692	-.1093	-.0995	.1277	.2855
	(9)	(9)	(9)	(9)	(9)	(9)
	.7262	.8595	.7795	.7989	.7434	.4564
DEO	-.3096	-.1267	-.0916	-.1002	-.1562	.0849
	(9)	(9)	(9)	(9)	(9)	(9)
	.4175	.7453	.8147	.7976	.6882	.8281
NPP	.3830	.2602	.3046	.3162	.2640	-.1661
	(9)	(9)	(9)	(9)	(9)	(9)
	.3090	.4989	.4254	.4071	.4924	.6693
FOPA	-.0687	-.2761	-.3958	-.3885	-.2088	-.0320
	(9)	(9)	(9)	(9)	(9)	(9)
	.8606	.4720	.2916	.3014	.5898	.9348
FPPA	-.2772	-.1427	-.0902	-.0915	-.2075	-.0606
	(9)	(9)	(9)	(9)	(9)	(9)
	.4702	.7142	.8175	.8149	.5921	.8770
MEP	-.0699	.0538	-.0269	.0368	.0337	.0470
	(9)	(9)	(9)	(9)	(9)	(9)
	.8581	.8906	.9453	.9250	.9314	.9045
DEP	-.5722	-.5205	-.3538	-.4001	-.5402	-.1886
	(9)	(9)	(9)	(9)	(9)	(9)
	.1074	.1508	.3503	.2860	.1332	.6270
tREOF2	.9935	.8855	.7696	.8495	.9143	.1101
	(9)	(9)	(9)	(9)	(9)	(9)
	.0000	.0015	.0153	.0037	.0006	.7780
RREOF2	.6741	.7846	.9634	.9462	.7531	.2504
	(9)	(9)	(9)	(9)	(9)	(9)
	.0464	.0123	.0000	.0001	.0192	.5158
EREOF2	.8189	.8319	.9453	.9602	.8211	.1384
	(9)	(9)	(9)	(9)	(9)	(9)
	.0069	.0054	.0001	.0000	.0067	.7226
SREOF2	1.0000	.8430	.7215	.8066	.8856	.0398
	(9)	(9)	(9)	(9)	(9)	(9)
	.0000	.0043	.0282	.0086	.0015	.9189
tREOFC	.8430	1.0000	.8506	.9196	.9917	.5241
	(9)	(9)	(9)	(9)	(9)	(9)
	.0043	.0000	.0037	.0004	.0000	.1475

	RREOF4	EREOF4	SREOF4	MDRATIO
NbC	.5517	.2362	-.2670	.3442
	(9)	(9)	(9)	(9)
	.1236	.5407	.4874	.3644
n	.7367	.5476	-.0407	.1948
	(9)	(9)	(9)	(9)
	.0236	.1270	.9172	.6155
MEO	.2023	.2982	.2143	.6439
	(9)	(9)	(9)	(9)
	.6017	.4357	.5797	.0613
DEO	-.5502	-.3243	.1579	-.9168
	(9)	(9)	(9)	(9)
	.1248	.3946	.6849	.0005
NPP	.5321	.2497	-.1945	.7374
	(9)	(9)	(9)	(9)
	.1403	.5171	.6160	.0234
FOPA	.1537	.0609	-.1425	.7497
	(9)	(9)	(9)	(9)
	.6930	.8764	.7146	.0200
FPPA	-.2418	-.2277	-.0717	-.4022
	(9)	(9)	(9)	(9)
	.5309	.5557	.8545	.2832
MEP	-.1662	-.0960	.0001	-.4133
	(9)	(9)	(9)	(9)
	.6691	.8059	.9998	.2689
DEP	-.5310	-.4652	-.1447	-.6023
	(9)	(9)	(9)	(9)
	.1413	.2070	.7103	.0861
tREOF2	.8398	.7700	.1279	.1239
	(9)	(9)	(9)	(9)
	.0046	.0152	.7430	.7508
RREOF2	.6045	.7228	.3662	-.2053
	(9)	(9)	(9)	(9)
	.0847	.0278	.3324	.5962
EREOF2	.7321	.7450	.2302	-.1011
	(9)	(9)	(9)	(9)
	.0249	.0213	.5513	.7958
SREOF2	.8491	.7376	.0555	.1604
	(9)	(9)	(9)	(9)
	.0038	.0233	.8873	.6801
tREOFC	.6042	.8253	.5436	-.0918
	(9)	(9)	(9)	(9)
	.0848	.0062	.1303	.8143

Sample Correlations

	NbC	n	MEO	DEO	NPP	FOPA
RREOFC	.4365	.7455	-.1093	-.0916	.3046	-.3958
	(9)	(9)	(9)	(9)	(9)	(9)
	.2401	.0211	.7795	.8147	.4254	.2916
EREOFC	.4607	.8012	-.0995	-.1002	.3162	-.3885
	(9)	(9)	(9)	(9)	(9)	(9)
	.2120	.0094	.7989	.7976	.4071	.3014
SREOFC	.3681	.7321	.1277	-.1562	.2640	-.2088
	(9)	(9)	(9)	(9)	(9)	(9)
	.3297	.0249	.7434	.6882	.4924	.5898
tREOF4	-.2926	-.0731	.2855	.0849	-.1661	-.0320
	(9)	(9)	(9)	(9)	(9)	(9)
	.4448	.8517	.4564	.8281	.6693	.9348
RREOF4	.5517	.7367	.2023	-.5502	.5321	.1537
	(9)	(9)	(9)	(9)	(9)	(9)
	.1236	.0236	.6017	.1248	.1403	.6930
EREOF4	.2362	.5476	.2982	-.3243	.2497	.0609
	(9)	(9)	(9)	(9)	(9)	(9)
	.5407	.1270	.4357	.3946	.5171	.8764
SREOF4	-.2670	-.0407	.2143	.1579	-.1945	-.1425
	(9)	(9)	(9)	(9)	(9)	(9)
	.4874	.9172	.5797	.6849	.6160	.7146
MDRATIO	.3442	.1948	.6439	-.9168	.7374	.7497
	(9)	(9)	(9)	(9)	(9)	(9)
	.3644	.6155	.0613	.0005	.0234	.0200

Coefficient (sample size) significance level

	FPPA	MEP	DEP	tREOF2	RREOF2	EREOF2
RREOFC	-.0902	-.0269	-.3538	.7696	.9634	.9453
	(9)	(9)	(9)	(9)	(9)	(9)
	.8175	.9453	.3503	.0153	.0000	.0001
EREOFC	-.0915	.0368	-.4001	.8495	.9462	.9602
	(9)	(9)	(9)	(9)	(9)	(9)
	.8149	.9250	.2860	.0037	.0001	.0000
SREOFC	-.2075	.0337	-.5402	.9143	.7531	.8211
	(9)	(9)	(9)	(9)	(9)	(9)
	.5921	.9314	.1332	.0006	.0192	.0067
tREOF4	-.0606	.0470	-.1886	.1101	.2504	.1384
	(9)	(9)	(9)	(9)	(9)	(9)
	.8770	.9045	.6270	.7780	.5158	.7226
RREOF4	-.2418	-.1662	-.5310	.8398	.6045	.7321
	(9)	(9)	(9)	(9)	(9)	(9)
	.5309	.6691	.1413	.0046	.0847	.0249
EREOF4	-.2277	-.0960	-.4652	.7700	.7228	.7450
	(9)	(9)	(9)	(9)	(9)	(9)
	.5557	.8059	.2070	.0152	.0278	.0213
SREOF4	-.0717	.0001	-.1447	.1279	.3662	.2302
	(9)	(9)	(9)	(9)	(9)	(9)
	.8545	.9998	.7103	.7430	.3324	.5513
MDRATIO	-.4022	-.4133	-.6023	.1239	-.2053	-.1011
	(9)	(9)	(9)	(9)	(9)	(9)
	.2832	.2689	.0861	.7508	.5962	.7958

	SREOF2	tREOFC	RREOFC	EREOFC	SREOFC	tREOF4
RREOFC	.7215	.8506	1.0000	.9835	.8209	.3884
	(9)	(9)	(9)	(9)	(9)	(9)
	.0282	.0037	.0000	.0000	.0067	.3016
EREOFC	.8066	.9196	.9835	1.0000	.9003	.3849
	(9)	(9)	(9)	(9)	(9)	(9)
	.0086	.0004	.0000	.0000	.0009	.3063
SREOFC	.8856	.9917	.8209	.9003	1.0000	.4698
	(9)	(9)	(9)	(9)	(9)	(9)
	.0015	.0000	.0067	.0009	.0000	.2020
tREOF4	.0398	.5241	.3884	.3849	.4698	1.0000
	(9)	(9)	(9)	(9)	(9)	(9)
	.9189	.1475	.3016	.3063	.2020	.0000
RREOF4	.8491	.6042	.6949	.7142	.6363	-.1094
	(9)	(9)	(9)	(9)	(9)	(9)
	.0038	.0848	.0378	.0307	.0654	.7794
EREOF4	.7376	.8253	.8695	.8695	.8201	.5143
	(9)	(9)	(9)	(9)	(9)	(9)
	.0233	.0062	.0023	.0023	.0068	.1566
SREOF4	.0555	.5436	.4767	.4521	.4850	.9801
	(9)	(9)	(9)	(9)	(9)	(9)
	.8873	.1303	.1945	.2218	.1857	.0000
MDRATIO	.1604	-.0918	-.0612	-.0693	-.0483	-.2110
	(9)	(9)	(9)	(9)	(9)	(9)
	.6801	.8143	.8758	.8595	.9019	.5857

	RREOF4	EREOF4	SREOF4	MDRATIO
RREOFC	.6949	.8695	.4767	-.0612
	(9)	(9)	(9)	(9)
	.0378	.0023	.1945	.8758
EREOFC	.7142	.8695	.4521	-.0693
	(9)	(9)	(9)	(9)
	.0307	.0023	.2218	.8595
SREOFC	.6363	.8201	.4850	-.0483
	(9)	(9)	(9)	(9)
	.0654	.0068	.1857	.9019
tREOF4	-.1094	.5143	.9801	-.2110
	(9)	(9)	(9)	(9)
	.7794	.1566	.0000	.5857
RREOF4	1.0000	.7805	-.0968	.4827
	(9)	(9)	(9)	(9)
	.0000	.0131	.8043	.1881
EREOF4	.7805	1.0000	.5369	.1947
	(9)	(9)	(9)	(9)
	.0131	.0000	.1361	.6157
SREOF4	-.0968	.5369	1.0000	-.2929
	(9)	(9)	(9)	(9)
	.8043	.1361	.0000	.4444
MDRATIO	.4827	.1947	-.2929	1.0000
	(9)	(9)	(9)	(9)
	.1881	.6157	.4444	.0000