APPENDIX J

RUDDER SERVO CONTROL HYDRAULIC FLUID SAMPLE ANALYSIS

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MINISTÈRE DE LA DÉFENSE

DGA

DELEGATION GENERALE
POUR L'ARMEMENT

DIRECTION DES
CENTRES D'EXPERTISE
ET D'ESSAIS

Centre d'essais des
propulseurs

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Bâtiment 153 – Aéroport du Bourget
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(A l'attention de MM. Berthier et Gaubert)

Saclay, le
N° */DCE/CEPr/DEE/IP/R*

BORDEREAU D'ENVOI

NUMÉRO DES PIÈCES	DÉSIGNATION DES PIÈCES	NOMBRE	OBSERVATIONS
	Investigation report n° 37-IP-2002 Subject of investigation : AIRBUS A300-600 registered N 14053. Analysis of hydraulic fluid.	7	Le chef du département « Investigations et Produits » N. Bouchez

DESTINATAIRES

- BEA – (7 ex)
  Sécurité Aviation Civile
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  (A l'attention de J.F Berthier et G. Gaubert)

DIFFUSION INTERNE

- D/St/Courrier
- D ⇨ SDA ⇨ SDT ⇨ DEE ⇨DEE/IP (1 ex)
- DEE/IP/R (Mme Félix –1 ex)

MINISTERE DE LA DEFENSE

DGA

DIRECTION DES CENTRES D'EXPERTISE ET D'ESSAIS

CENTRE D'ESSAIS DES PROPULSEURS
91695 Orsay CEDEX

DEPARTEMENT INVESTIGATIONS-PRODUITS

INVESTIGATION REPORT

37 – IP – 02
OT 4459

Subject of investigation : AIRBUS A300-600 registered N 14053. Analysis of hydraulic fluid.

Références : Request n° 06/2002 of 04/03/2002 by the BEA

Date of reception of products : 04/04/2002
Start of investigation : 04/11/2002
End of l'investigation : 06/06/2002

This report concerns the physical and chemical analysis of samples of hydraulic fluid taken from the three rudder servo-controls from the AIRBUS A300 registered N14053 involved in the accident in New York (USA) on November 12 2001. The circumstances of the accident are related in the appendix.

Analysis of the samples of hydraulic fluid from the three servo-controls shows evidence of the presence of water in greater or lesser quantities according to the sampling. High water quantity is associated with the presence of significant quantities of iron and lesser quantities of magnesium. Corrosion by sea water of steels and alloys could explain these results.

COMPOSITION

Pages	Plates	Appendix	Références bibliographiques
31	21	1	

REPERES D'ARCHIVAGE

Thème d'identification :

Mots clés :

Les Responsables d'Investigations	Le Responsable d'Affaire	Le Responsable Assurance Qualité Investigation
M.C Félix – H. Sylvestre	M.C Félix	C. Nagy

DIFFUSION INTERNE : D⇨SDA⇨SDT⇨DEE⇨IP/St - DEE/EX (M.C Félix) – IP/R (H. Sylvestre) DQC/AQM (M. Nagy)

DIFFUSION EXTERNE :

BEA Sécurité Aviation Civile (A l'attention de MM. J.F Berthier et G. Gaubert) – (7 ex)

SOMMAIRE

1 - INTRODUCTION

This report concerns the physical and chemical analysis of samples of hydraulic fluid taken from the three rudder servo-controls from the AIRBUS A300 registered N14053 involved in the accident in New York (USA) on November 12 2001. The circumstances of the accident are related in the appendix.

Analyses were also performed on the fluids from the test bench, as well as on the filters on these benches, and on various other grease samples.

The samples received in 4 boxes were referenced as follows :

Reference	Label	Container
	Box UPPER SC 1	
1	Graisse sur joint	Glass flask 50 cm^3
2	Graisse sur filetage	Glass flask 50 cm^3
3	Fluid from thread (eau)	Glass flask 50 cm^3
4	Graisse servo-control	Glass flask 50 cm^3
5	Fluid from R	Glass flask 250 (5 cm^3)
6	Fluid from CB	Glass flask 250 (100 cm^3)
7	Fluid on P	Glass flask 250 (1 cm^3)
	Box MIDDLE SC	
8	Fluid from return line	Glass flask 250 cm^3 full
9	Fluid from return line	Glass flask 250 cm^3 full
10	Fluid from CA	Glass flask 250 cm^3 full
11	Fluid from CA	Glass flask 250 (100 cm^3)
12	Fluid from CB	Glass flask 250 cm^3 full
13	Fluid from CB	Glass flask 250 (60 cm^3)
	Box Lower SC	
14	Deposit taken from rear bearing 4-380	Glass flask 50 cm^3 with deposit
15	Fluid from return line filter (after test)	Glass flask 250 cm^3 full
16	Fluid from return line filter (after test)	Glass flask 250 cm^3 full
17	Fluid CA	Glass flask 250 cm^3 full
18	Fluid CA	Glass flask 250 cm^3 (50 cm^3)
19	Fluid CB	Glass flask 250 cm^3 full

Reference	Label	Container
	Box UPPER SC 2 + prélèvements bench	
20	Return line on test bench	Glass flask 250 cm^3 full
21	Return line on test bench	Glass flask 250 cm^3 full
22	Return line on test bench	Glass flask 250 cm^3 full
23	Return line on test bench	Glass flask 250 cm^3 full
24	Return line after complete test	Glass flask 250 cm^3 full
25	Return line after complete test	Glass flask 250 cm^3 full
26	Fluid from CA	Glass flask 250 cm^3 full
27	Supply line on test bench	Glass flask 250 cm^3 (100 cm^3)
28	Test bench	Glass flask 250 cm^3 (200 cm^3)
29	Filtre upper	Cardboard 20 cm
30	Filtre MIDDLE return line	Cardboard 20 cm
31	Filtre LOWER return line	Cardboard 20 cm
32	Sample HYJET 4 new	Glass flask 250 cm^3 full

2 – ANALYSIS OF THE SAMPLES OF HYDRAULIC FLUIDS

2.1 physical and chemical characteristics

The characteristics measured are indicated in plates 1 to 5.

2.1.1 Kinematic viscosity at 40 and 100°C

The viscosity measured on the samples from the three servo-controls were of the same order, at about 6.6 mm2/s at 100°C, which seems normal for used fluid.

The viscosity measured on the samples from the test benches was of the order of 7.8 mm^2/s at 40°C and about 2.7 mm^2/s at 100°C.

2.1.2 Total acid number

The acid numbers were not high on any of the samples (0.2 à 0.3 mgKOH/g) which indicates that the fluid was not subject to high temperatures.

2.1.3 Water content

The samples from the servo-controls contained between 2 and 7 % m/m of water, except for the samples referenced 10 and 11 « Middle SC from CA » and 18 « Lower SC from CA », which contained respectively : 0.32 ; 0.38 and 0.77 %.

The samples from the test benches showed water content of the order of 0.3 % m/m.

It appears that it can be concluded that sea water penetrated the three servo-controls.

2.1.4 Sediment content

Filtering through Teflon membranes with 5µm porosity was performed on 12 fluid samples.
The photos and analysis of the most characteristic deposits are given in paragraph 7.

2.2 Metallic element content

The metallic element content of the samples of hydraulic fluids was determined by rotated disc electrode optical emission spectrometry samples from the servo-controls showed very high iron content from 19 ppm for reference 11 to 30 ppm for references 12 and 13, and above 50 ppm for the other samples, these high values most likely being due to corrosion resulting from the presence of sea water.

The high magnesium content in the samples containing water confirmed the presence of sea water.

The samples from the test bench showed high silicon content and noticeable zinc content.

2.3 Qualitative analysis by gas chromatography linked to mass spectrometry (GC/MS)

The chromatograms of the samples of hydraulic fluid, from the servo-controls and the test bench, are shown in plates 6 to 10, the chromatogram of new fluid being illustrated in plate 6 for purposes of comparison.

It should be noted that the chromatograms for the samples referenced 8 – 15 – 20 – 24 – 27 and 28 taken from the test bench, are essentially identical to that of new fluid.

The chromatograms of the samples referenced 5, 6, 7, 10, 12, 17, 19 and 26 taken from the servo-controls show the disappearance of certain plots, probably due to the fact that these fluids had been used and that the additives had fixed in the metallic parts of the system.

No anomalies in composition were identified during this analysis.

3 – ANALYSIS OF THE SAMPLE REFERENCED 3

The sample referenced 3 « Fluid from thread », is a drop of water in appearance, and analysis using the Karl Fischer method confirmed that it contrained **98,3 %** water.

4 – ANALYSIS OF THE « GREASE » (references 1, 2 and 4)

The grease was analysed by infra red absorption spectrometry, X ray fluorescence spectrometry and by gas chromatography linked to mass spectrometry (GC/MS).

The results obtained by infra-red absorption spectrometry and by X ray fluorescence spectrometry are shown in the following table. The spectra are shown in plates 12, 13, 14 and 15.

Reference	IR ANALYSIS	X RAY ANALYSIS
1	teflon, silicone and traces of Hyjet IV	Cause of the low quantity of available products,not any element was detected
2	teflon, silicone and traces of Hyjet IV	main elements : silicon, aluminium, phosphorus. Trace of iron and silver
4	teflon, silicone and traces of Hyjet IV	Cause of the low quantity of available products, not any element was detected

- **Analysis by GC/MS**

Gas chromatography linked to mass spectrometry did not confirm the presence of Teflon. The resulting chromatograms are in plate 16.

Comments

According to the facts available to us, it apparears that some Teflon, as well as traces of hyjet and silicon, can be found on the three samples.

5 – EXAMINATION OF THE FILTERS AND ANALYSIS OF THE POLLUTING PRODUCTS REFERENCED 29, 30 and 31

The products contained in the filters were extracted by ultrasound in petroleum ether and analysed by infra-red absorption spectrometry.

The extracted product retrieved for reference 30 is shown in the photo in plate 17. The results are recorded in the following table.

Reference	IR ANALYSIS
29 et 31	Not any products were extracted by ultrasound
30	Epoxy resin bisphenol A

Comments

Only the extraction performed on the filter referenced 30 allowed a deposit to be recovered. In order to determine the origin of the recovered product, various parts of the filter were analysed. Analysis of the stick band of the filter revealed the presence of polyamide and trace of Hyjet. Analysis of the mastic revealed the presence of calcium carbonate $CaCO_3$.

It was not possible to prove that the product detected was a part of the composition of the filter.

6 – ANALYSIS OF THE SAMPLE « REFERENCED REPERE 14 »

The deposit referenced 14 appeared in the form of an oxidized and pasty deposit.

The deposit was first washed in petroleum ether then analysed by infra-red absorption spectrometry and by X ray fluorescence spectrométry. The spectra are shown in plate 18.

The washing liquid was analysed by X ray fluorescence spectrometry.

Analysis of the deposit after washing showed the presence of Teflon, carbonate and iron.

The washing liquid contained silicon and iron.

7 – ANALYSIS OF THE DEPOSITS RECOVERED AFTER FILTERING THROUGH MILLIPORE MEMBRANE

Photos of the 4 Millipore filters from which were extracted deposits characteristic of hydraulic fluids referenced 6, 8 and 25 are shown in plates 19 and 20.

The results from the most characteristic references (references 6, 8, 16, 25 and 26) are shown in the following table.

Reference	IR analysis	X ray fluorescence analysis
6 « Fluid from CB »	no significant product	iron, chlorine, nickel, copper, phosphorus, aluminium, silicon, potassium, calcium, sulphur
8 « Fluid from return line »	no significant product	-
16 « Fluid from return line filter »	no significant product	iron, chlorine, nickel, copper, phosphorus, aluminium, silicon, potassium, calcium, sulphur
25 « Return line »	no significant product	-
26 « Fluid from CA »	no significant product	-

NB : Results from references 10, 12, 18, 19, 20, 27 and 28 are not shown in the table :

- Either the quantities were too low to perform the analysis
- or the results obtained revealed no extra information

It should be noted that sodium is not detected with the method used.

Comments

No unexpected products were brought to light.

Is should also be noted that :

- potassium, chlorine and phosphate content were relatively higher on filter 16 than on filter 6,
- iron content appears higher on reference 6 than on reference 16.

These results appear to corroborate those obtained by rotated disc electrode optical emission spectrometry.

8 – CONCLUSION

Analysis of the samples of hydraulic fluid from the three servo-controls showed evidence of the presence of water in greater or lesser quantities according to the sampling. High water quantity was associated with the presence of significant quantities of iron and lesser quantities of magnesium. Corrosion by sea water of steels and alloys could explain these results.

- The samples from the test benches showed acceptable water and iron content, though the silicon content was high.

- The physical and chemical characteristics of kinematic viscosity and the acid number as well as the qualtitative analysis by gas chromatography linked to mass spectrometry (GC/MS) showed no anomalies in the previously mentioned fluids.

- Analysis of the sediments recovered from the Millipore membrane confirmed the results of the metallic elements performed through optical emission spectrometry.

- Examination of the test bench filters only identified the presence of expoxy resin.

- Analysis of the grease samples showed the presence of Teflon and silicon.

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INVESTIGATION REPORT N° 37 – IP – 02
(n°O.T. 4459)

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PLATE 1

PHYSICAL AND CHEMICALS PROPERTIES

AIRBUS UPPER SC FLUID 1

Property	Unity	reference 5 Fluid from R	reference6 Fluid from CB	ref,20 return line on test bench	ref,21 return line on test bench	ref,22 return line on test bench	ref,23 return line on test bench
Viscosity 40°C 100°C EN ISO 3104	mm2/s	 – –	 6,50 2,23	 8,39 2,95	 – –	 – –	 – –
Total acid number ASTM D 664	mg KOH/g	–	0,21	0,18	–	–	–
Water content NFT 60-154	%m/m	–	4,0	0,27	–	–	–
Contamination gravimétric analysis (5µm)	mg/100cm3	–	* 24	6,94	–	–	–
metallic elements and contaminants	mg/kg						
Fe		>50	>50	4	4	4	4
Ni		1	3	<0,5	<0,5	1	1
Cr		3	2	1	1	1	1
Al		6	2	1	1	2	1
Mg		14	24	2	2	2	2
Cu		14	<0,5	<0,5	<0,5	<0,5	<0,5
Sn		2	1	1	1	1	1
Pb		4	<0,5	<0,5	<0,5	1	<0,5
Ag		<0,5	<0,5	<0,5	<0,5	<0,5	<0,5
Si		18	11	24	20	20	21
Ti		<0,5	<0,5	<0,5	<0,5	<0,5	<0,5
V		1	1	1	1	1	1
Mo		1	1	<0,5	1	1	<0,5
Zn		8	1	5	5	6	5

*filter/ 50 cm3

PLATE 2

PHYSICAL AND CHEMICALS PROPERTIES

AIRBUS UPPER SC FLUID 2

Property	Unity	Ref,24 return line after	Ref,25 return line after	Ref,26 from CA
Viscosity 40°C 100°C EN ISO 3104	mm2/s	 - -	 7,88 2,77	 6,64 2,26
Total acid number ASTM D 664	mg KOH/g	-	0,2	0,33
Water content NFT 60-154	%m/m	-	0,25	2,45
Contamination gravimétric analysis (5µm)	mg/100cm3	-	6,24	8,7
metallic elements and contaminants	mg/kg			
Fe		3	4	>50
Ni		1	1	2
Cr		1	2	2
Al		2	2	3
Mg		1	1	16
Cu		<0,5	<0,5	1
Sn		1	1	1
Pb		<0,5	<0,5	<0,5
Ag		<0,5	<0,5	<0,5
Si		17	19	7
Ti		<0,5	<0,5	<0,5
V		1	1	2
Mo		1	1	1
Zn		6	7	2

PLATE 3

AIRBUS UPPER SC FLUID 3

Property	Unity	Ref, 27 supply line on test bench	Ref,28 test bench	Ref, 32 new	Référence Hyjet IV
Viscosity 40°C 100°C EN ISO 3104	mm2/s	 7,83 2,76	 7,83 2,76	 9,90 3,46	 9 à 12,5 3,00 à 4,00
Total acid number ASTM D 664	mgKOH/g	0,20	0,19	–	<0,15
Water content NFT 60-154	%m/m	0,37	0,23	–	<0,20
Contamination gravimétric analysis (5µm)	mg/100cm3	* 11,74	5,41	–	
metallic elements and contaminants	mg/kg				
Fe		3	2	<0,5	
Ni		1	1	<0,5	
Cr		2	1	<0,5	
Al		2	2	<0,5	
Mg		1	1	<0,5	
Cu		<0,5	<0,5	<0,5	
Sn		1	1	<0,5	
Pb		<0,5	<0,5	<0,5	
Ag		<0,5	<0,5	<0,5	
Si		19	19	<0,5	
Ti		<0,5	<0,5	<0,5	
V		1	1	<0,5	
Mo		1	1	<0,5	
Zn		7	6	<0,5	

*filtration of 50 cm3

PLATE 4

AIRBUS LOWER SC FLUID

Property	Unity	reference 15 from return line line filter (after test)	reference 16 from return line line filter (after test)	ref,17 fluid from CA	ref, 18 fluid from CA	ref, 19 fluid from CB *
Viscosity 40°C 100°C EN ISO 3104	mm2/s	 - -	 7,90 2,77	 7,13 2,48	 **	 6,63 2,30
Total acid number ASTM D 664	mg KOH/g	-	0,18	-	0,28	0,27
Water content NFT 60-154	%m/m	-	0,29	-	0,77	7,00
Contamination gravimetric analysis (5µm)	mg/100cm3	-	8,76	-	7,36 ***	23,88 ***
metallic elements and contaminants	mg/kg					
Fe		6	6	17	17	>50
Ni		1	1	2	2	7
Cr		1	1	6	5	4
Al		2	2	7	8	3
Mg		1	1	4	4	>50
Cu		<0,5	<0,5	4	4	<0,5
Sn		1	1	2	2	<0,5
Pb		<0,5	<0,5	1	1	<0,5
Ag		<0,5	<0,5	<0,5	<0,5	<0,5
Si		19	20	4	4	3
Ti		<0,5	<0,5	<0,5	<0,5	<0,5
V		1	1	2	3	<0,5
Mo		1	1	1	1	<0,5
Zn		7	6	4	3	1

*ref, 19 free water

** too small size of the sample

*** test with 50cm3

PLATE 5

AIRBUS MIDDLE SC FLUID

Property	Unity	reference 8 return line	reference9 return line	ref,10 from CA	ref,11 from CA	ref,12 from CB	ref,13 from CB
Viscosity 40°C 100°C EN ISO 3104	mm2/s	 7,86 2,78	 – –	 6,94 2,49	 –	 6,70 2,36	 - -
Total acid number ASTM D 664	mg KOH/g	0,2	–	0,25	–	0,19	-
Water content NFT 60-154	%m/m	0,28	0,26	0,32	0,38	2,50	2,39
contaminants gravimétric analysis (5µm)	mg/100cm3	30,11	–	6,52	–	3,48	-
metallic elements and contaminants Fe Ni Cr Al Mg Cu Sn Pb Ag Si Ti V Mo Zn	mg/kg	 3 1 1 2 1 <0,5 1 <0,5 <0,5 19 <0,5 1 1 7	 3 1 1 1 1 <0,5 1 <0,5 <0,5 21 <0,5 1 1 7	 2 1 2 3 1 1 2 <0,5 <0,5 3 <0,5 2 1 2	 3 1 2 3 1 1 2 1 <0,5 3 <0,5 2 1 2	 30 1 2 3 16 1 1 <0,5 <0,5 3 <0,5 1 1 2	 31 2 2 3 18 1 2 1 <0,5 3 <0,5 2 1 2

*filtration / 50 cm3

PLATE 6

GAS CHROMATOGRAPHY

LINKED TO MASS SPECTROMETRY
GC/MS

HYJET NEW FLUID

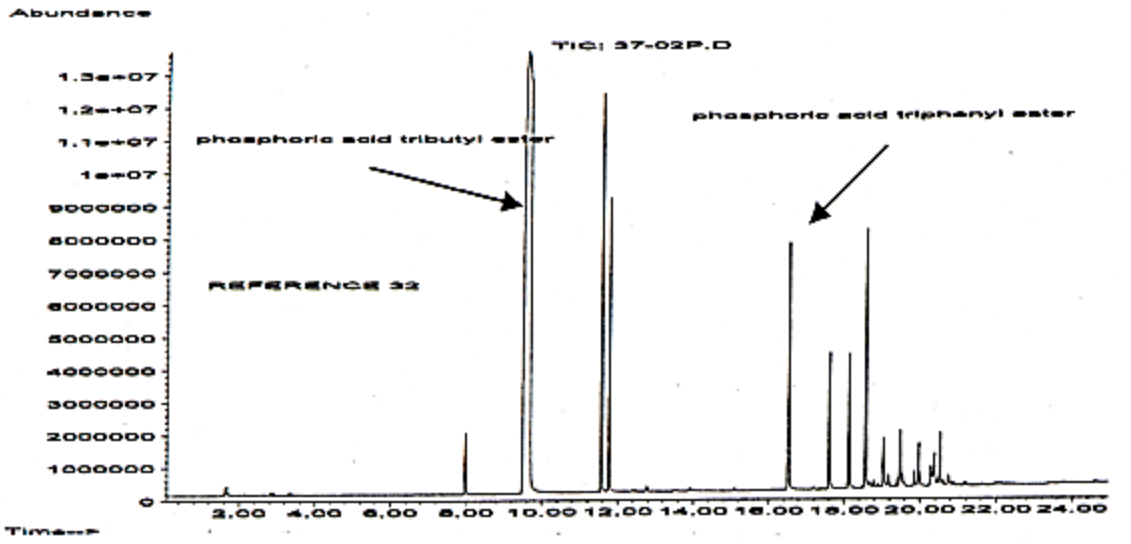
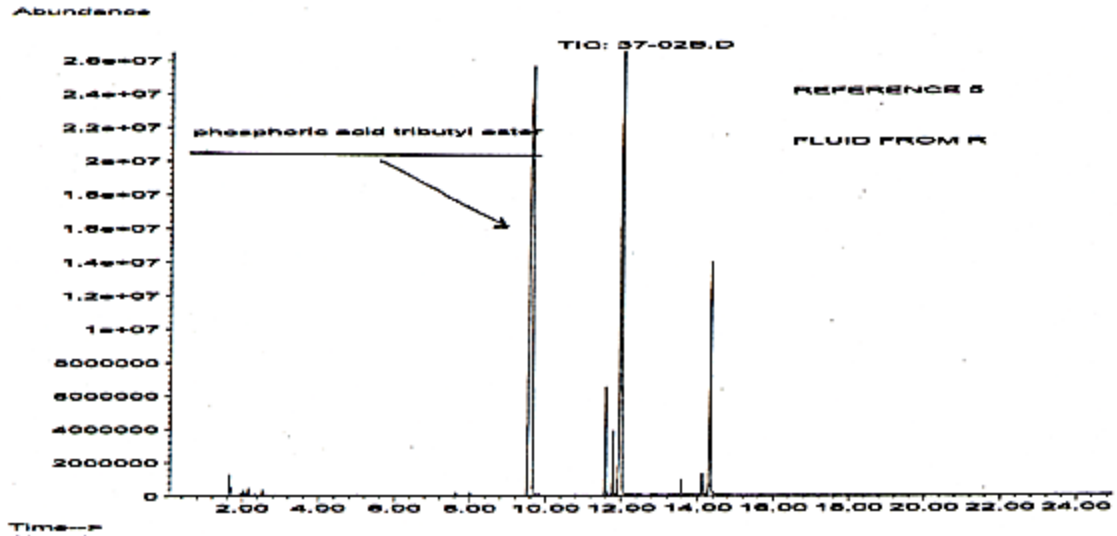
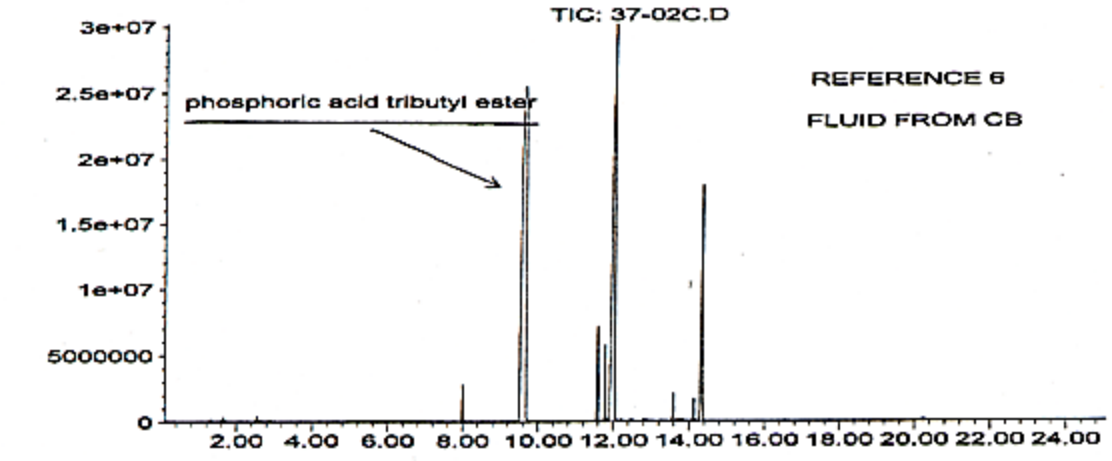


PLATE 7

UPPER SC





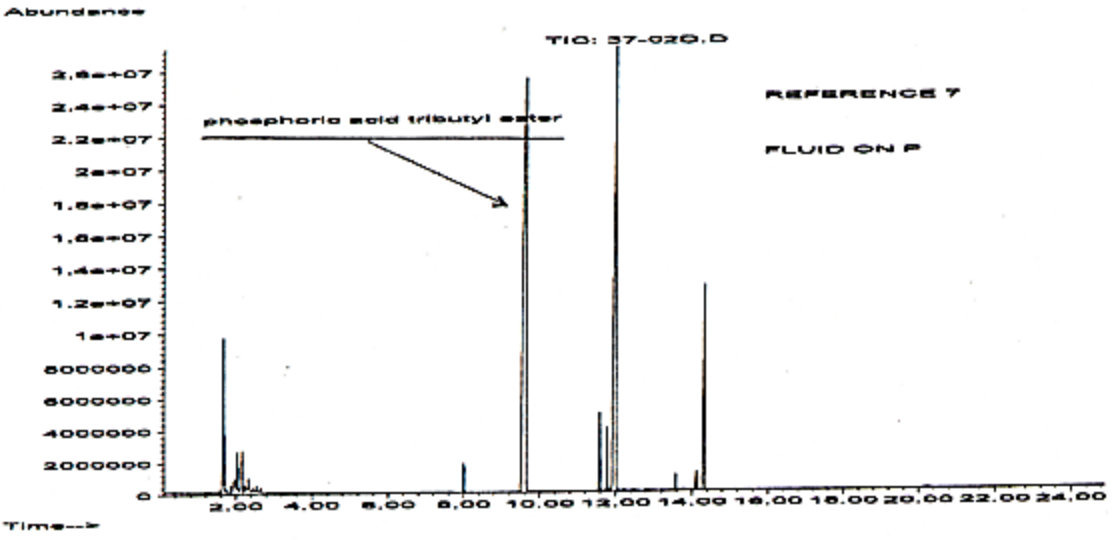
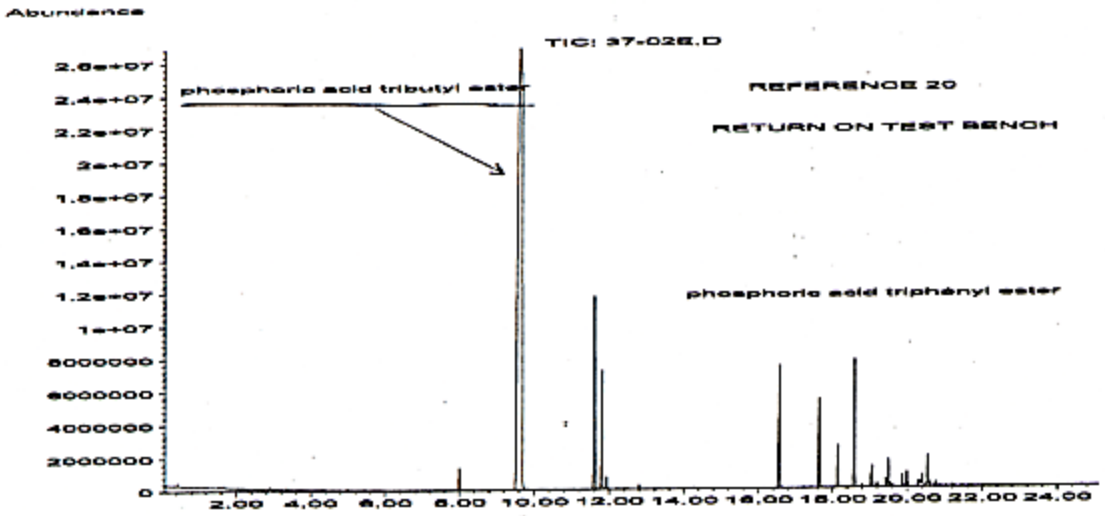
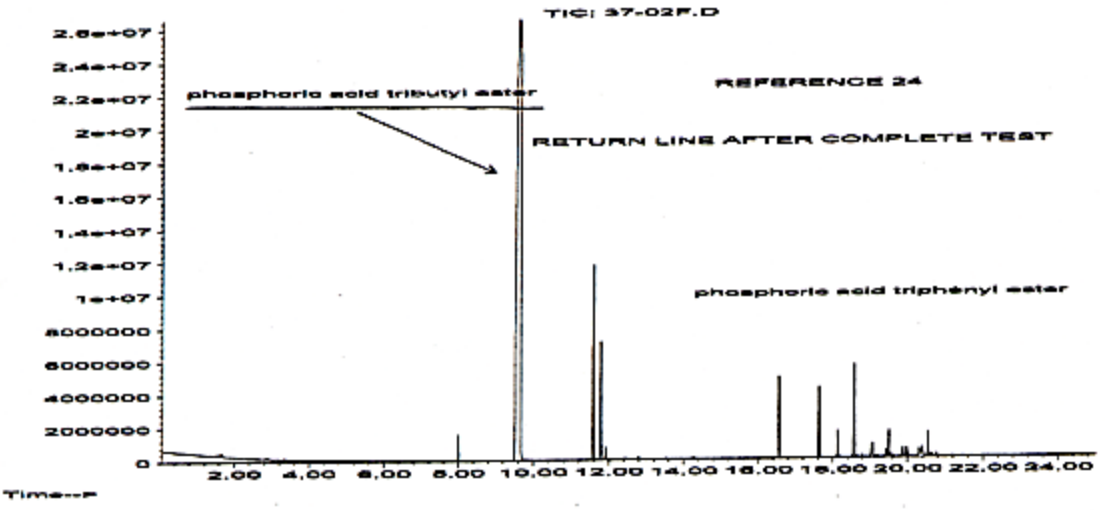


PLATE 8

UPPER SC





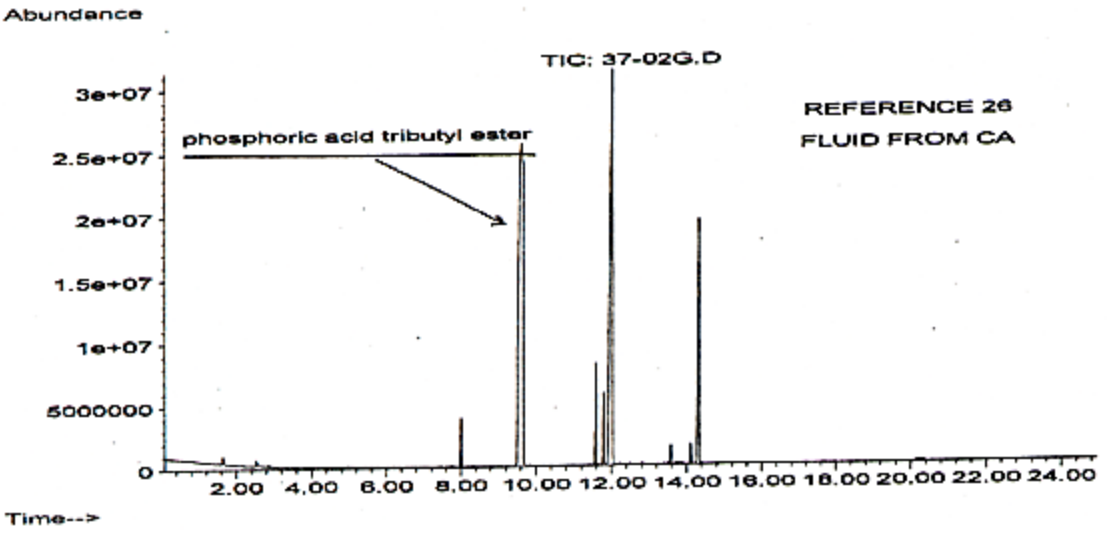


PLATE 9

UPPER SC

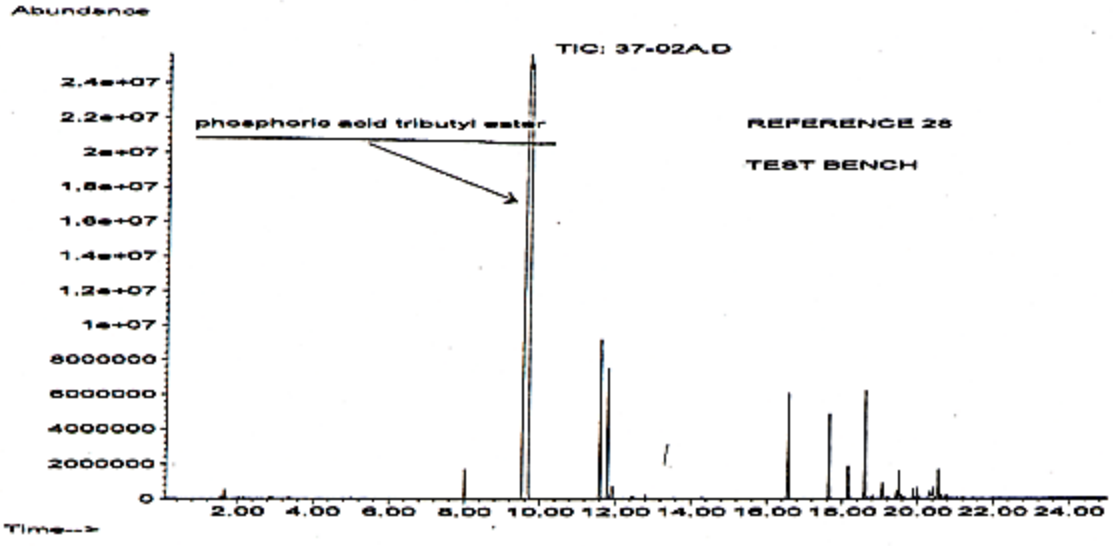
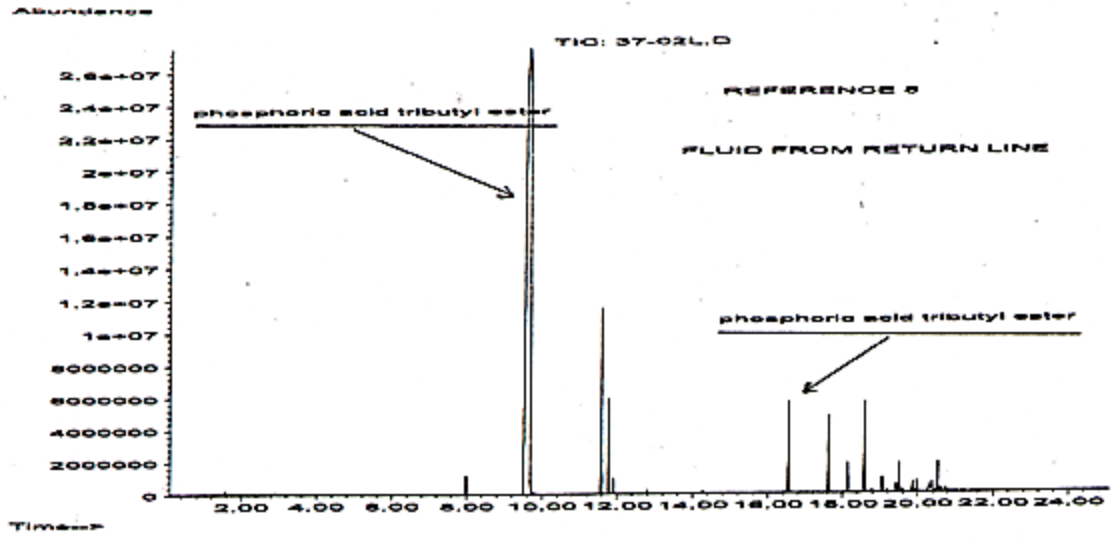
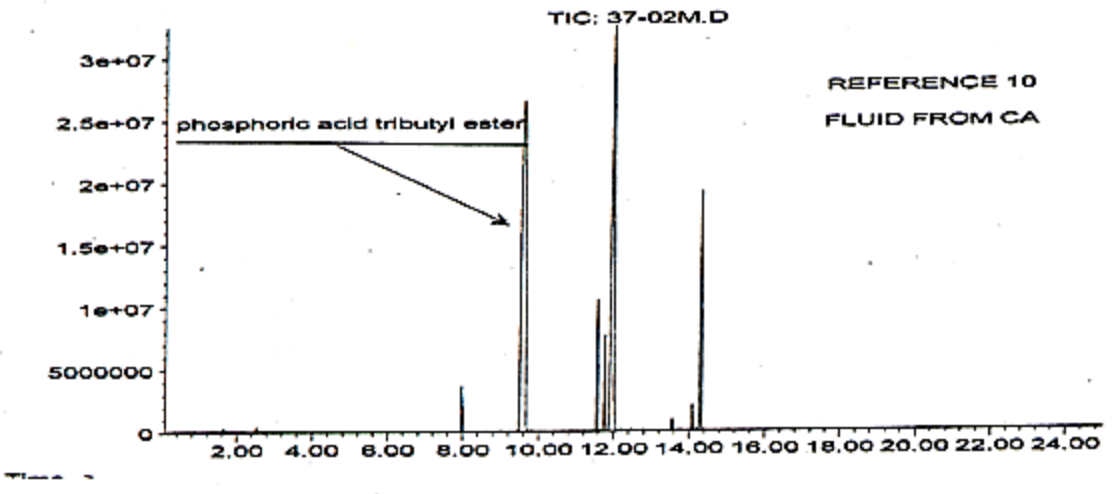


PLATE 10

MIDDLE SC





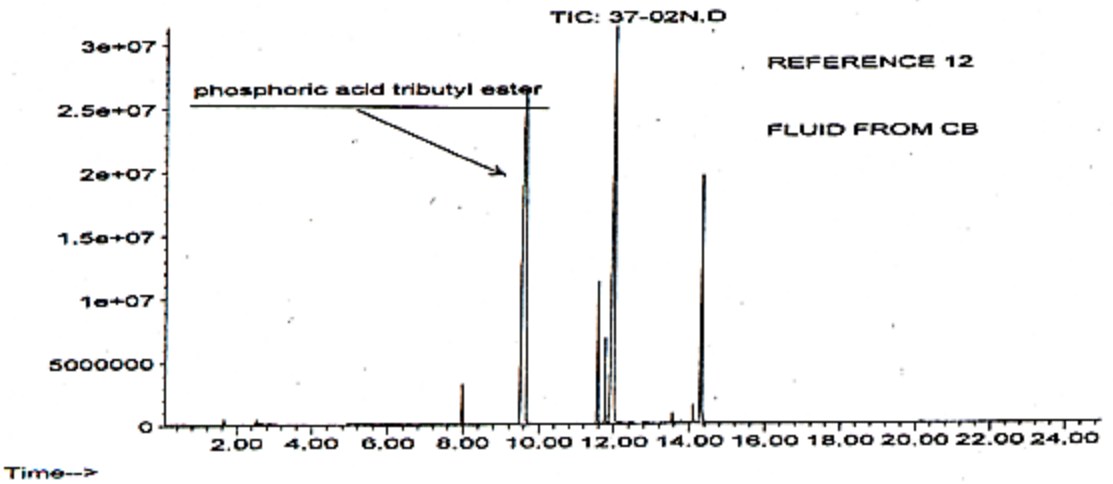
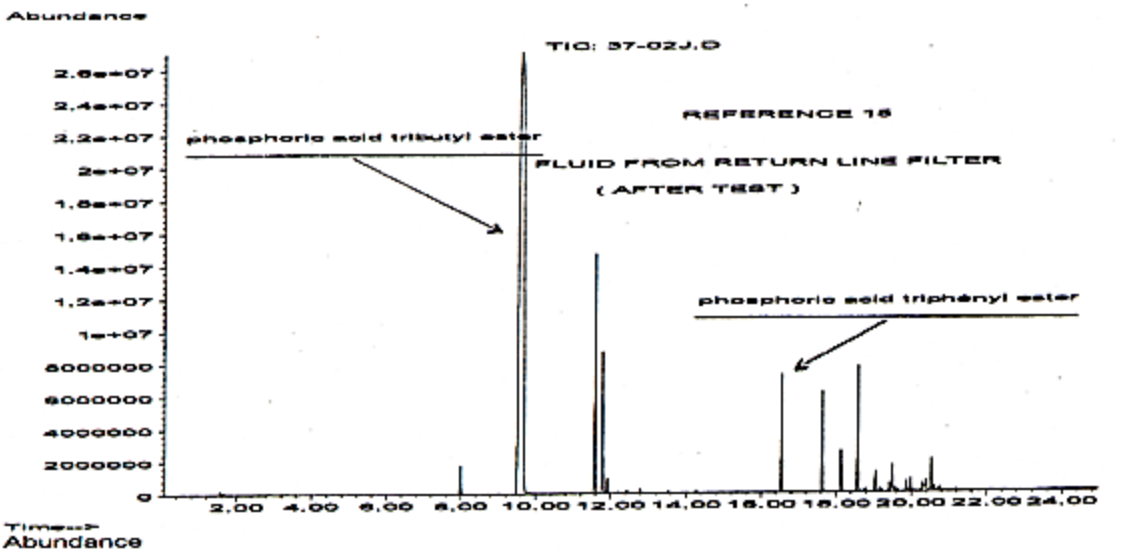
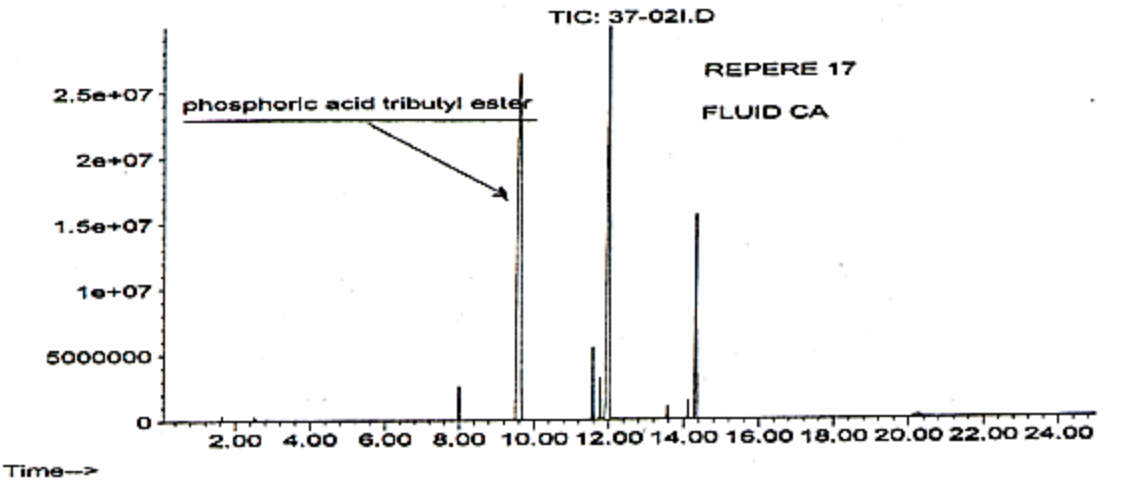


PLATE 11

LOWER SC





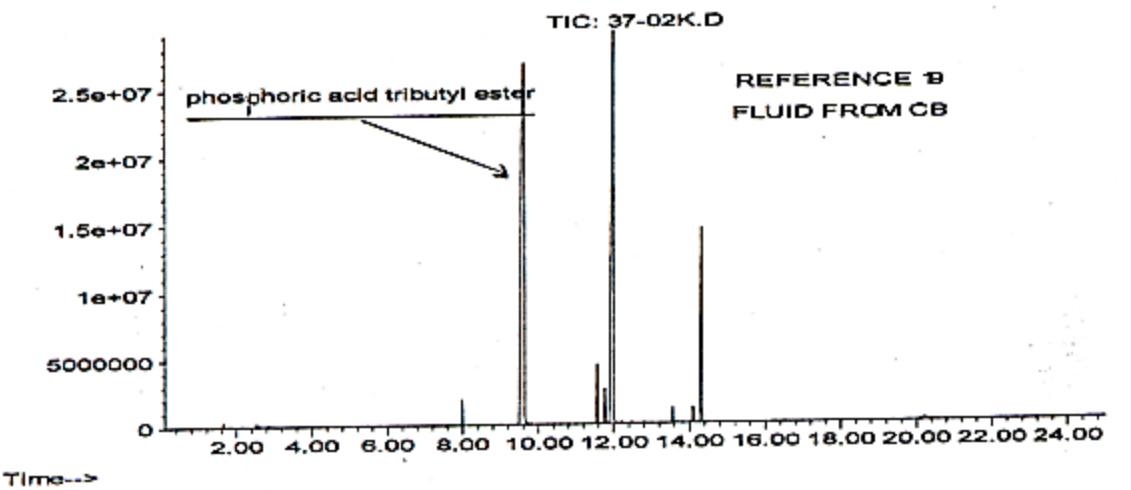
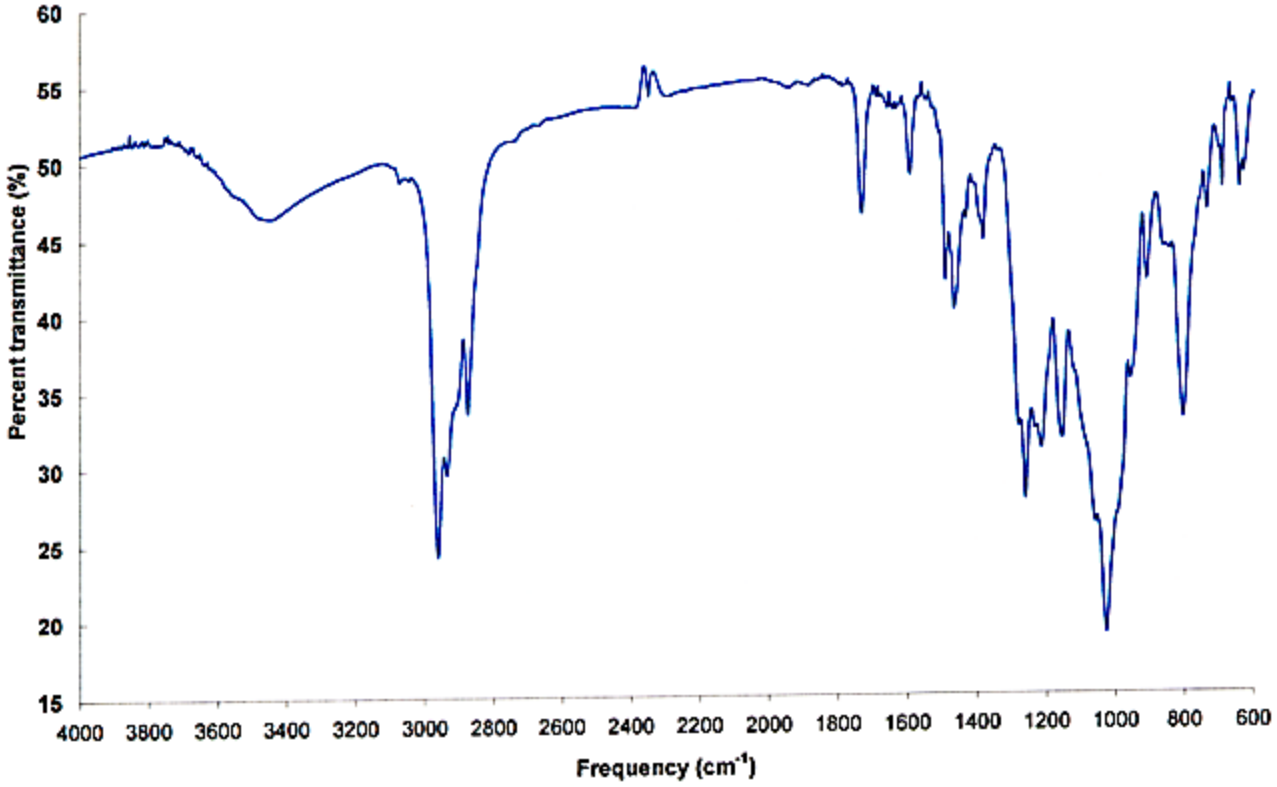
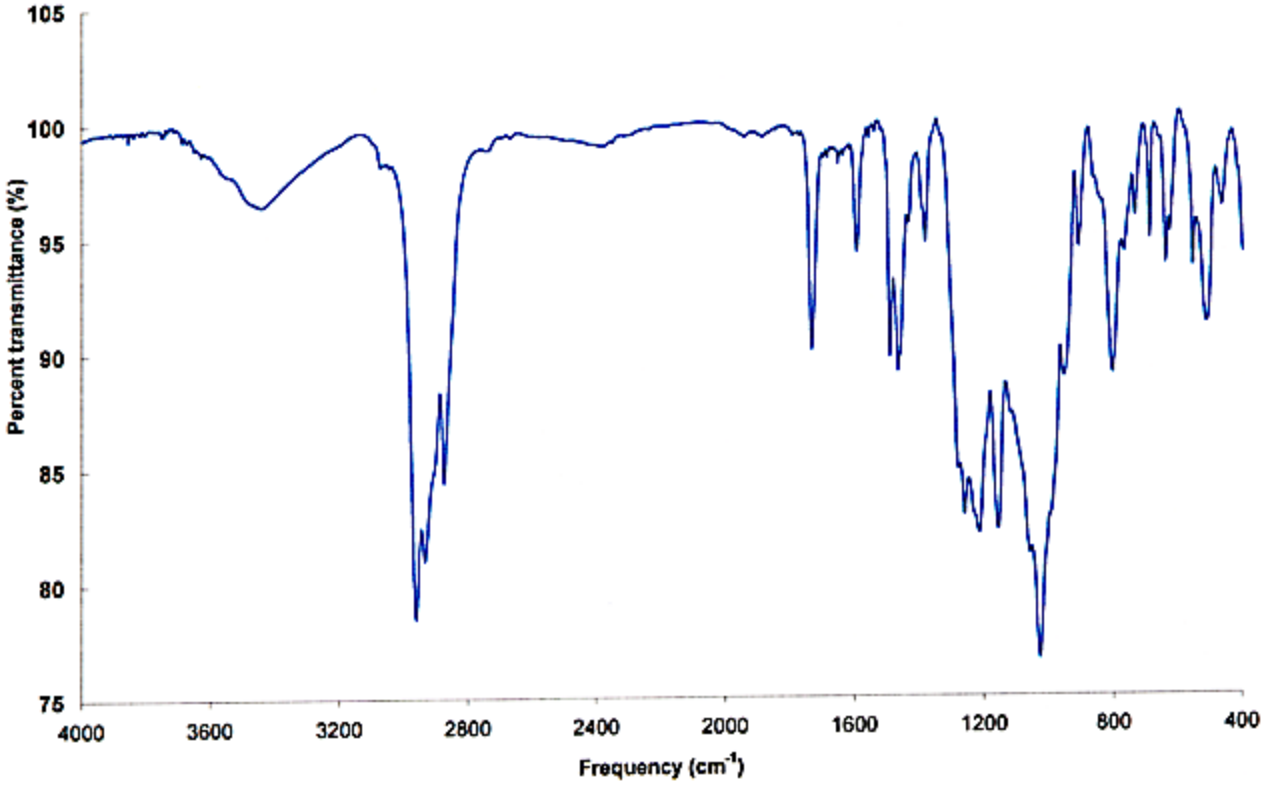


PLATE 12
Infrared Spectra

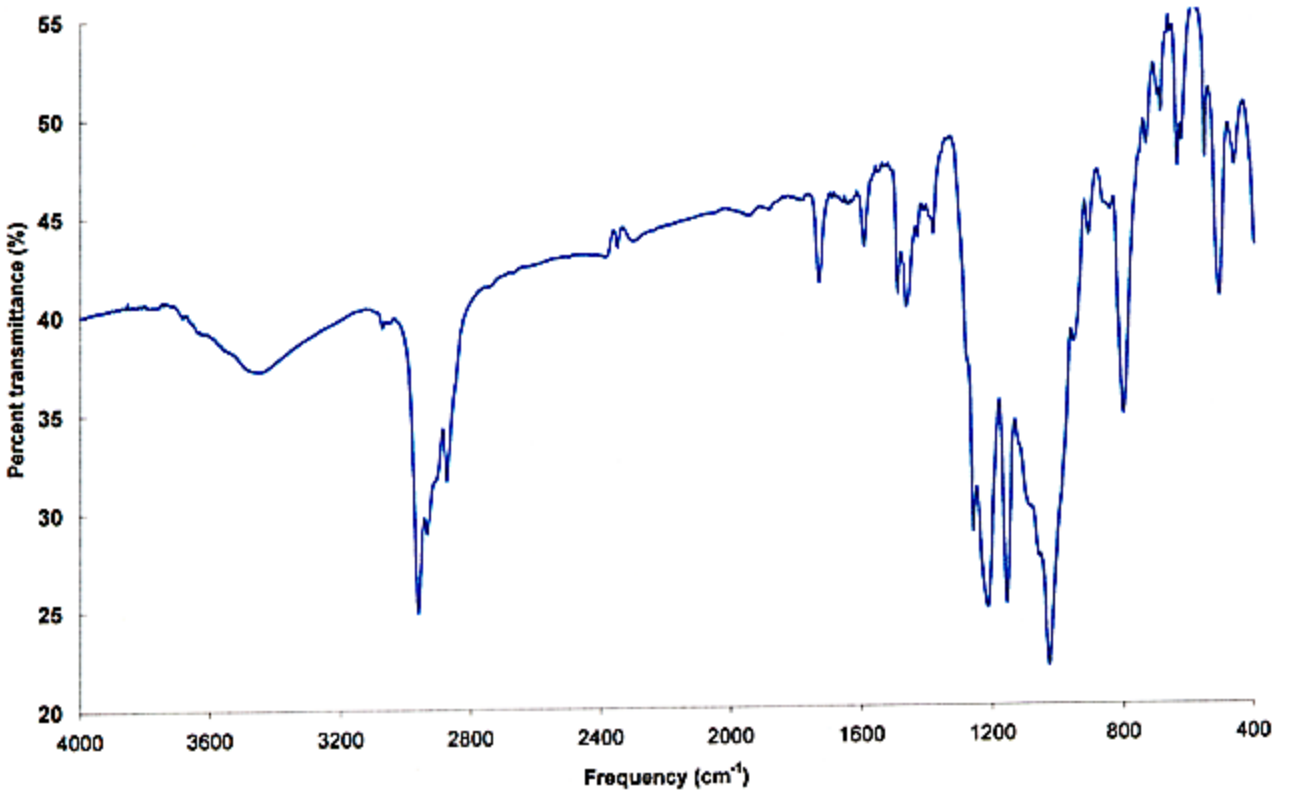


Spectrum 1 : Reference 1 (joint grease)

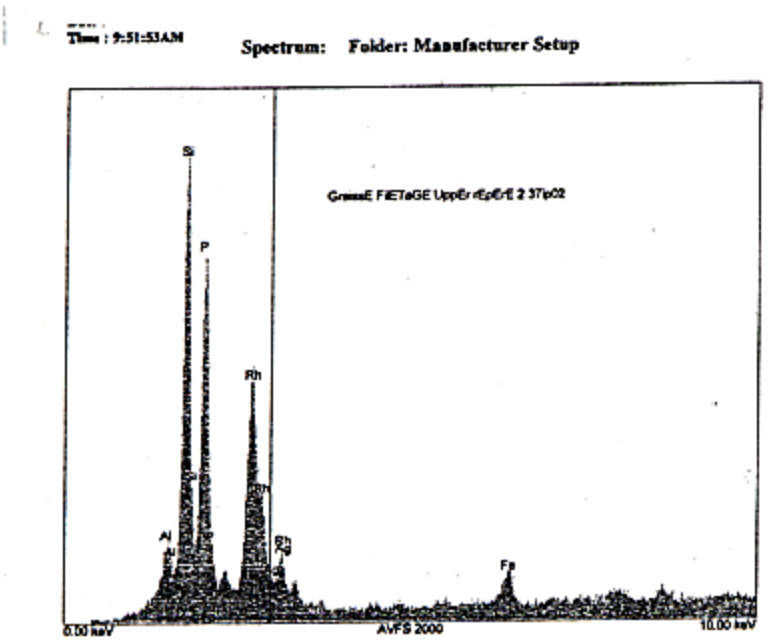


Spectrum 2 : Reference 4 (servo- control grease)

PLATE 13
infrared and fluorescence X spectrum item 2

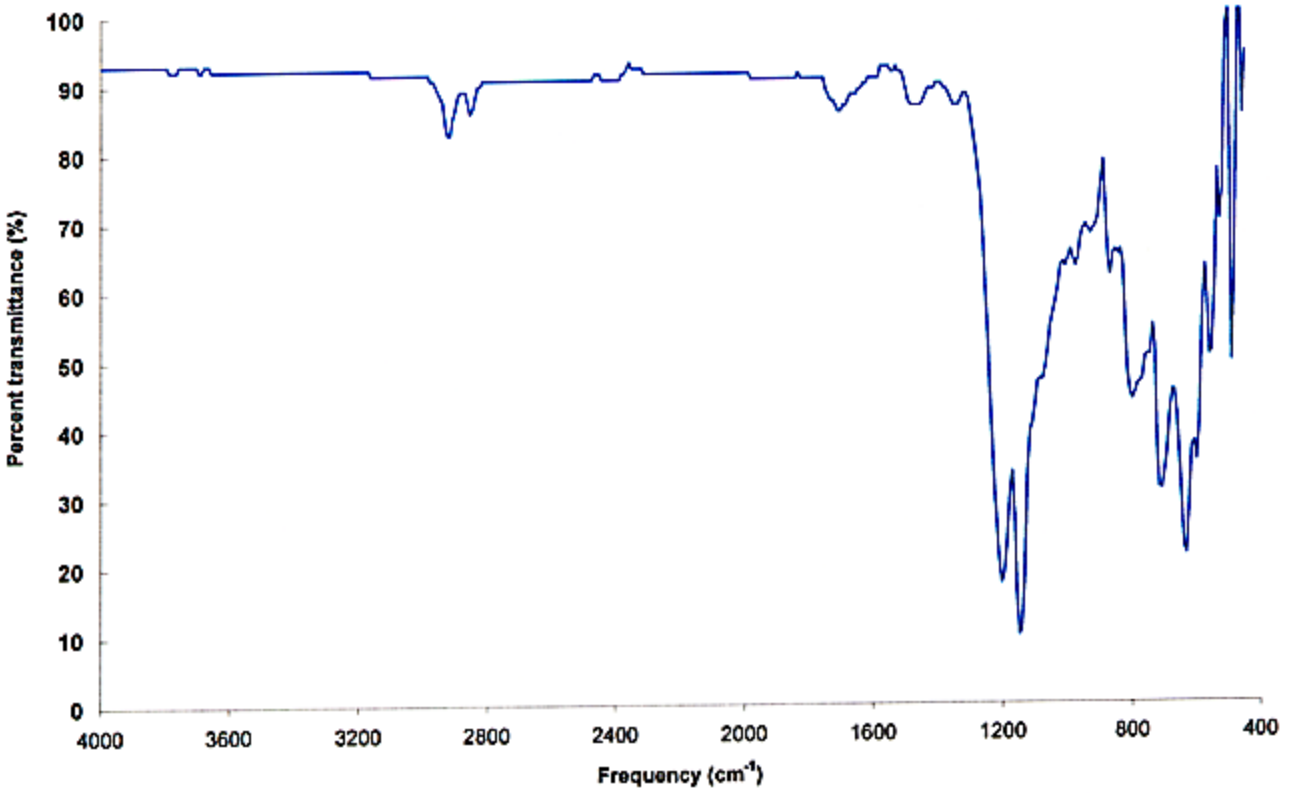


Spectrum 3 : Infrared spectrum reference 2 (thread grease)

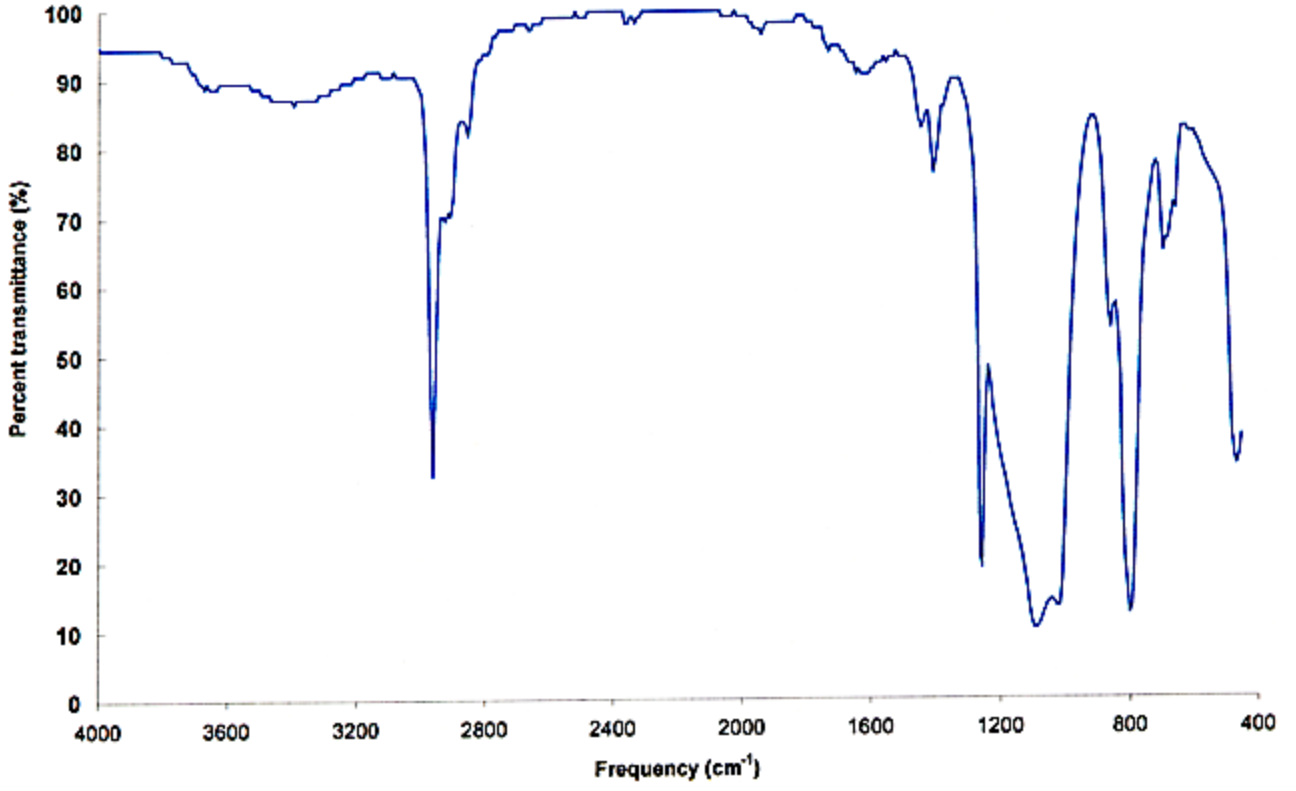


Spectrum 4 : fluorescence X spectrum item 2 (thread grease)

PLATE 14
Infrared spectra

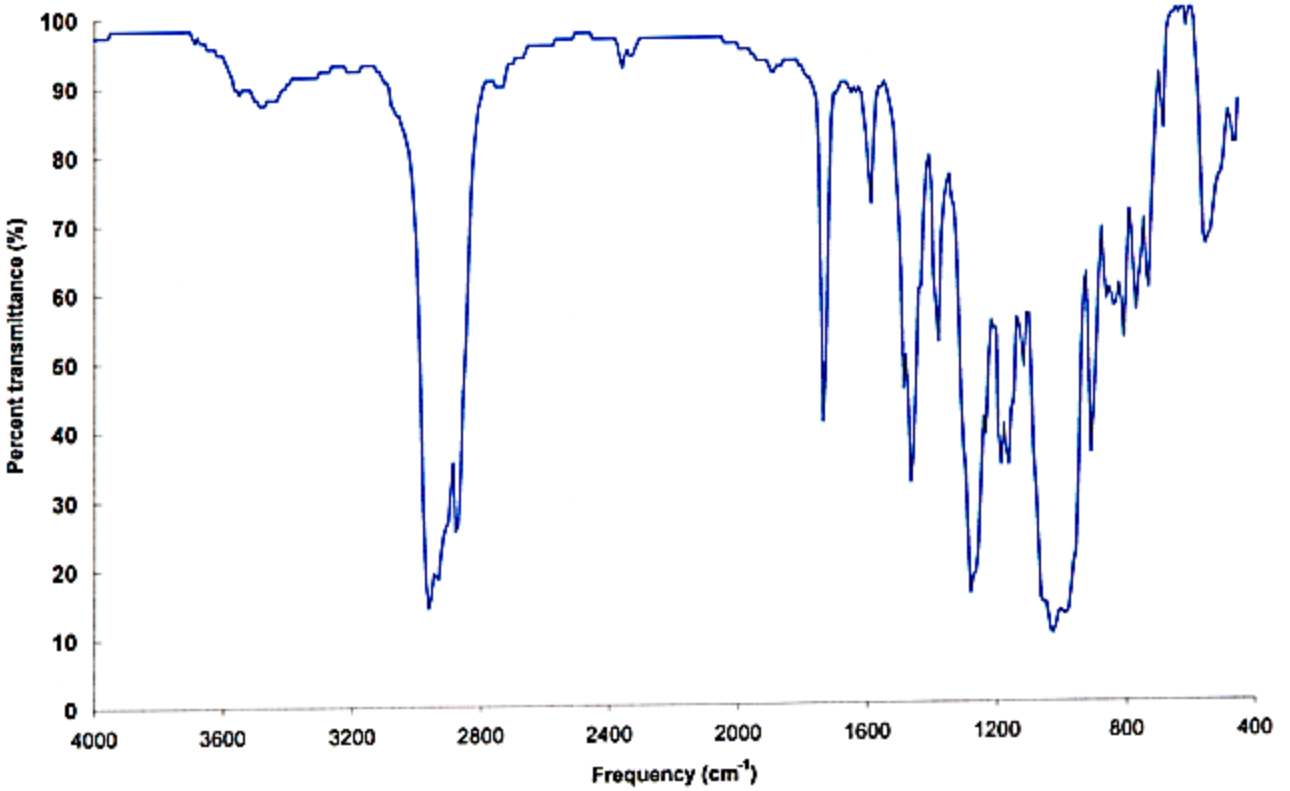


Spectrum 5 : Polytétrafluoroéthylène référence (PTFE)



Spectrum 6 : silicone référence

PLATE 15
Infrared spectrum



Spectrum 7 : New Hyjet IV référence

PLATE 16

UPPER SC

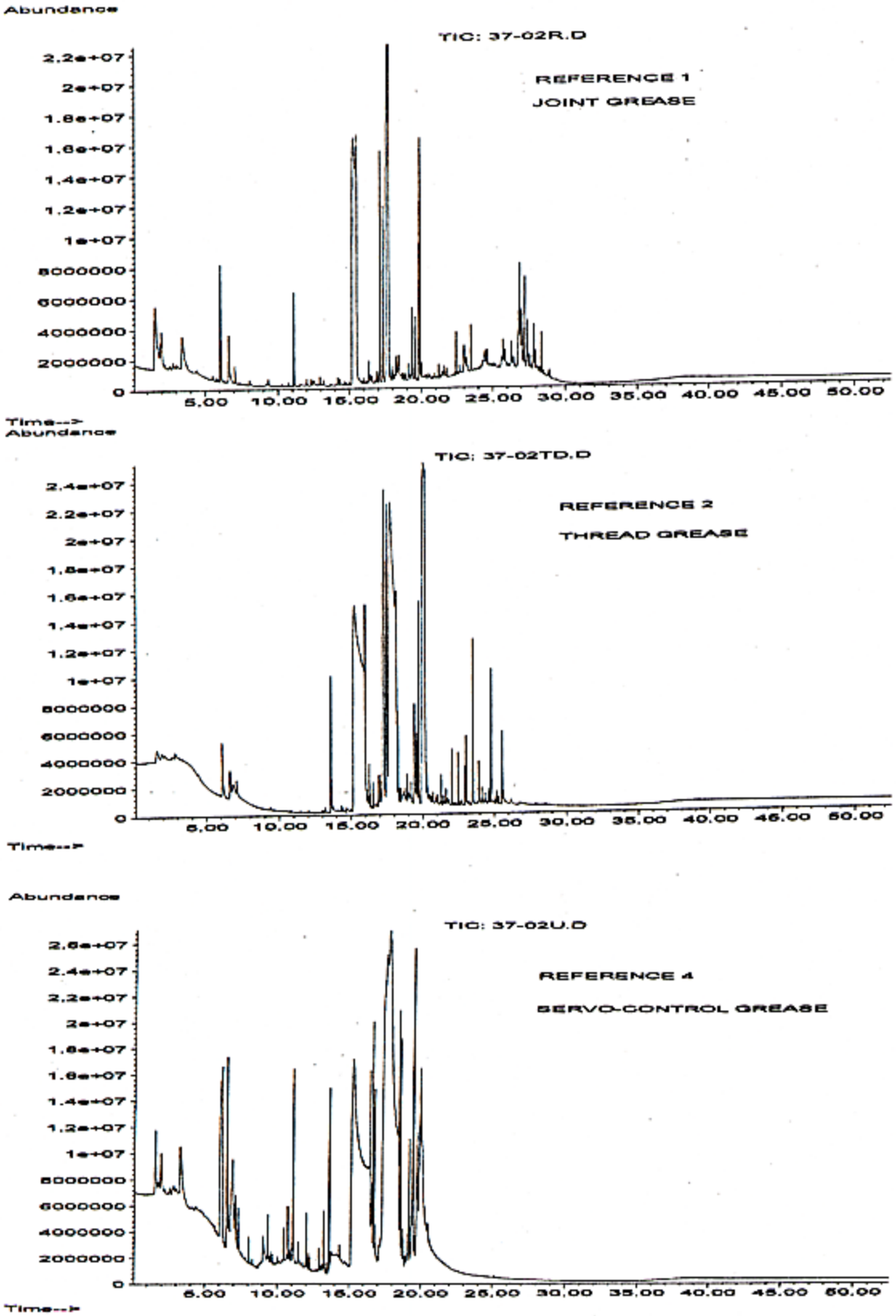
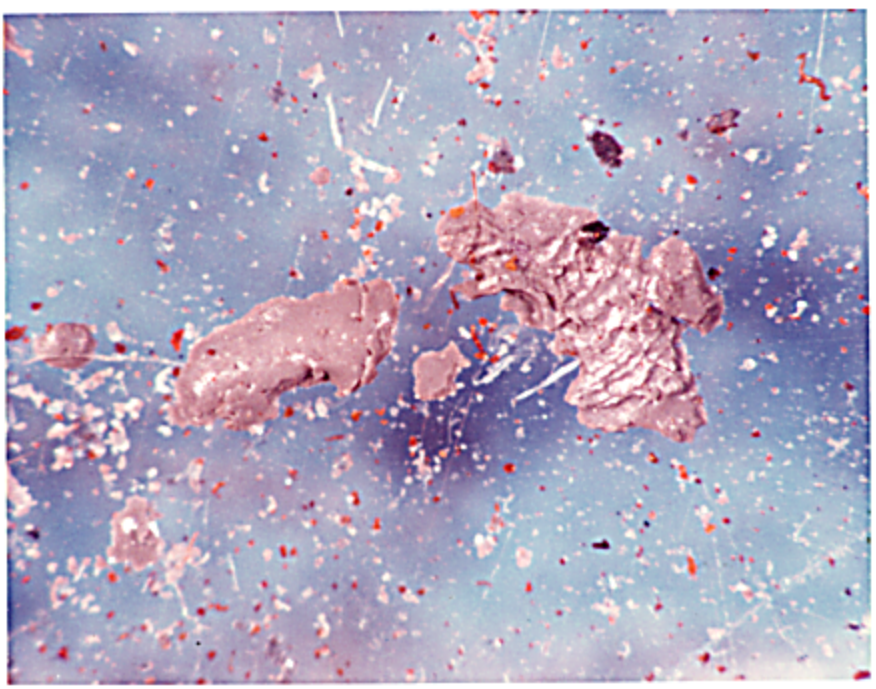
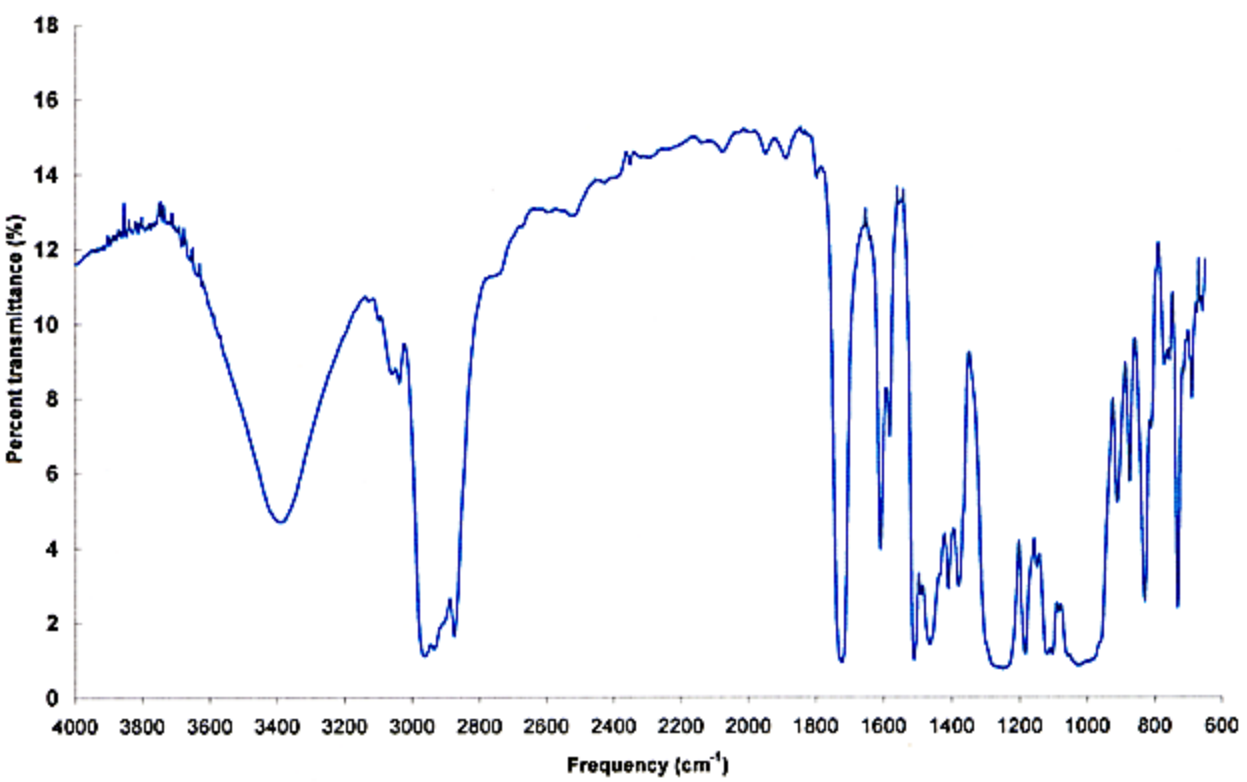


PLATE 17
Sediments recovered on the filter n° 30

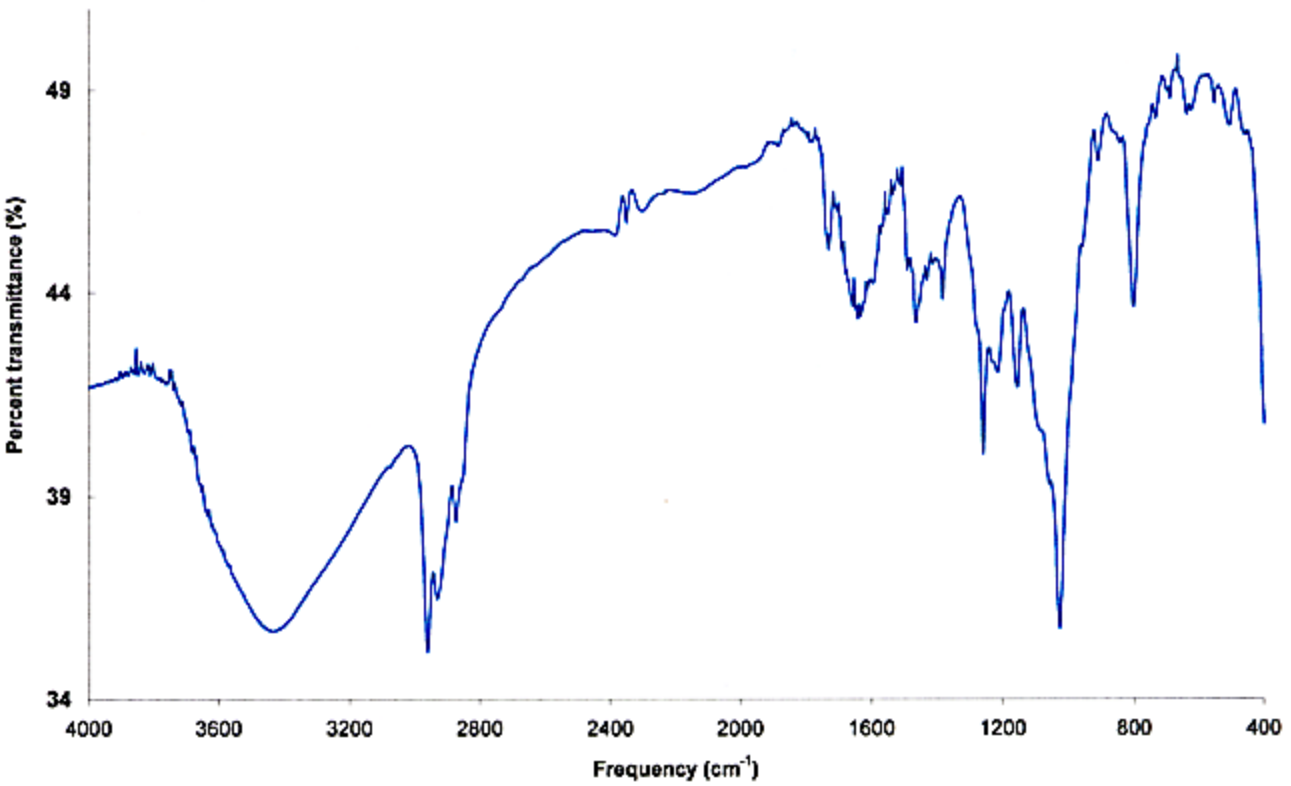


Photography 1 : Sediments photography recovered on the filter n° 30 (× 50)

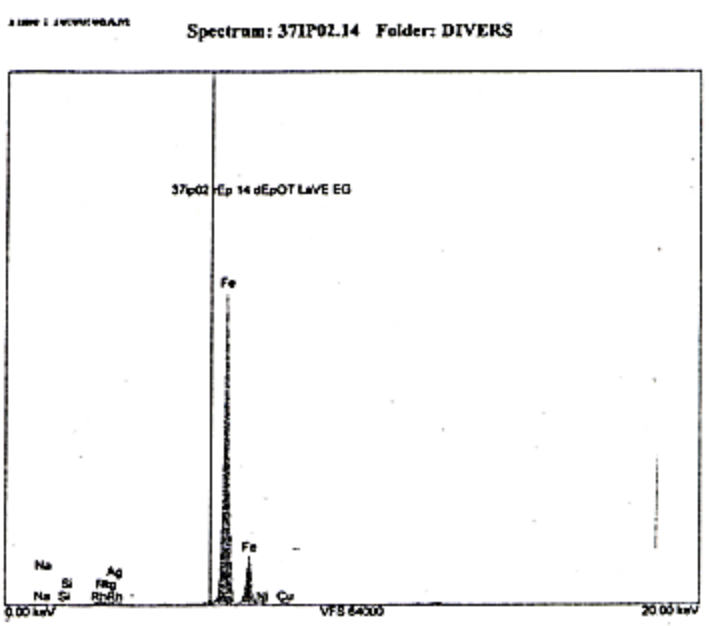


Spectrum 8 : Sediments infrared spectrum

PLATE 18
Infrared and fluorescence X Spectrum item 14

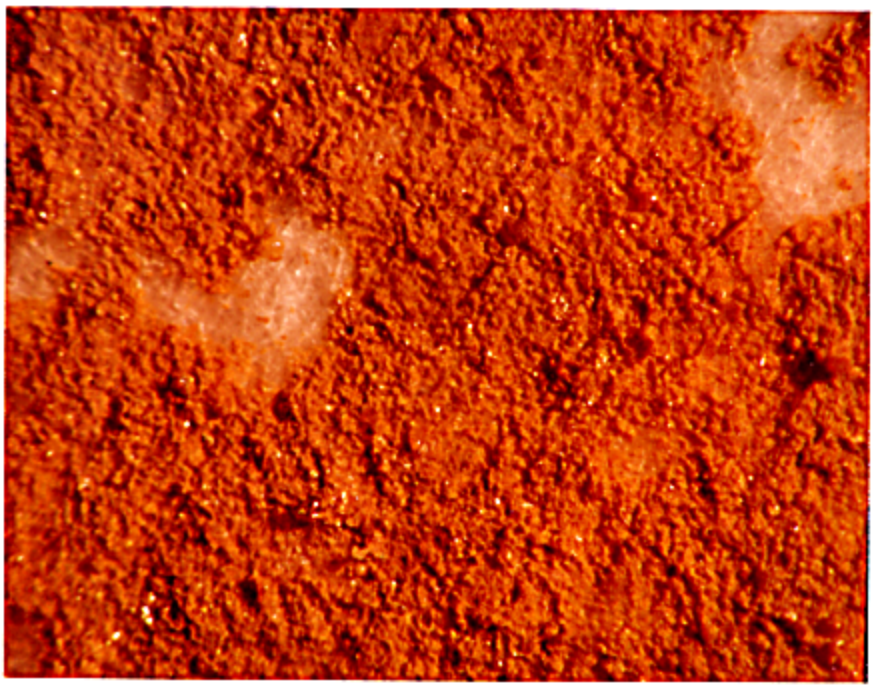


Spectrum 9 : Infrared Spectrum

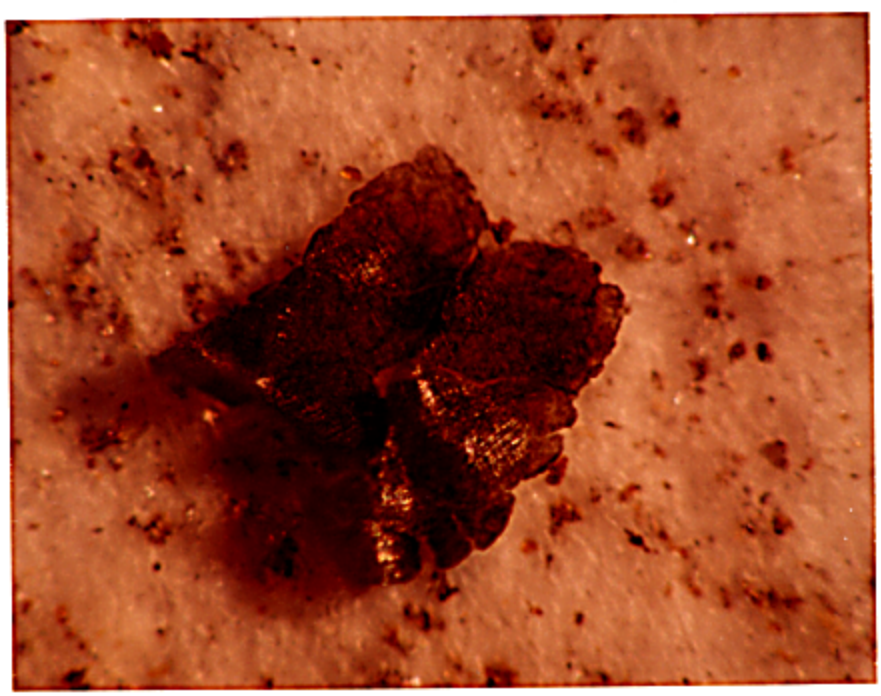


Spectrum 10 : X ray fluorescence spectrum

PLATE 19
Sediments extracted from hydraulic fluids

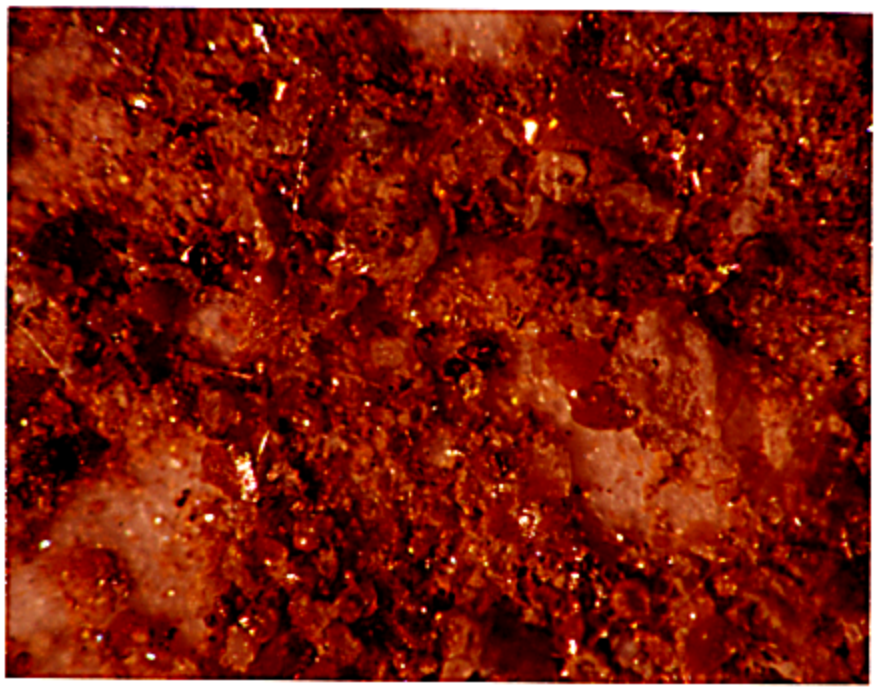


Photography 2 : Reference 6 photography (× 50)

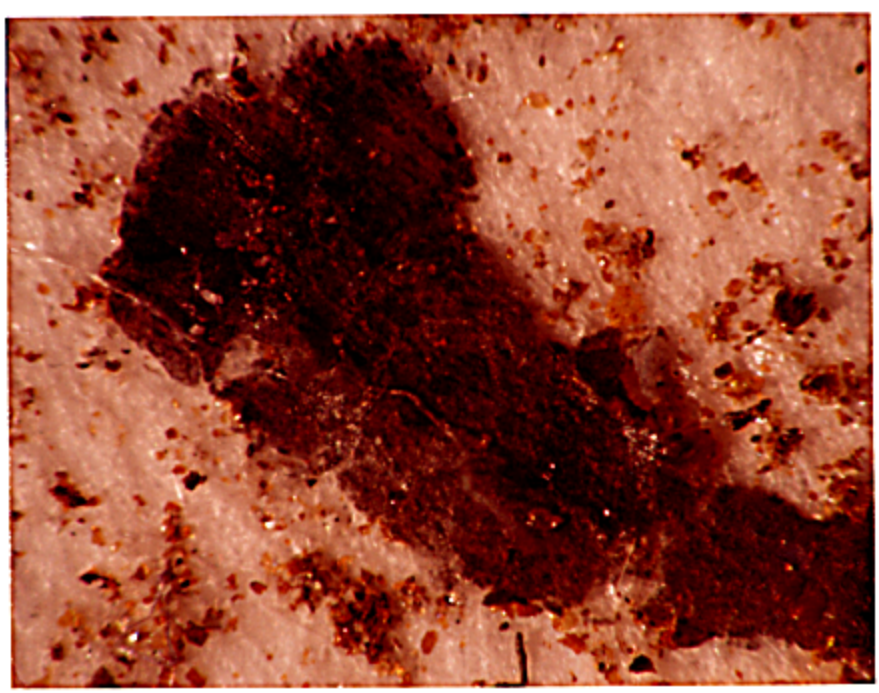


Photography 3 : Reference 8 Photography (× 50)

PLATE 20
Sediments extracted from hydraulic fluids

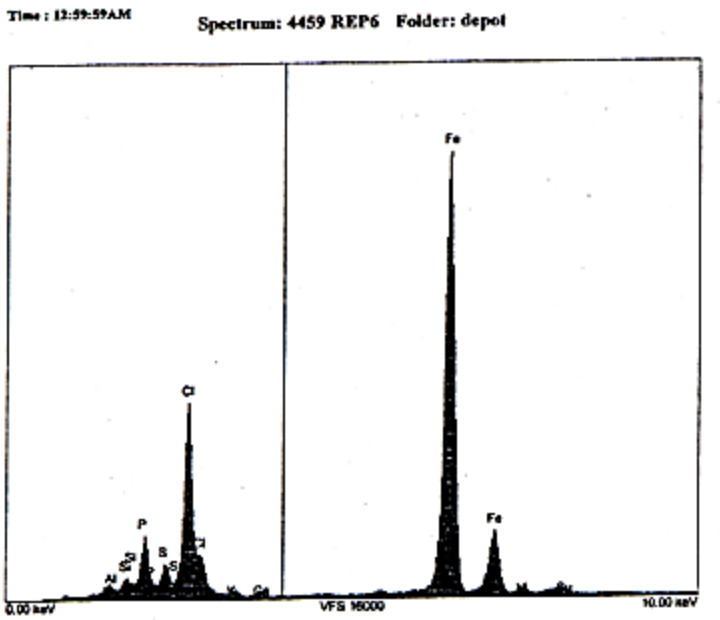


Photography 4 : Reference 16 photography (×50)

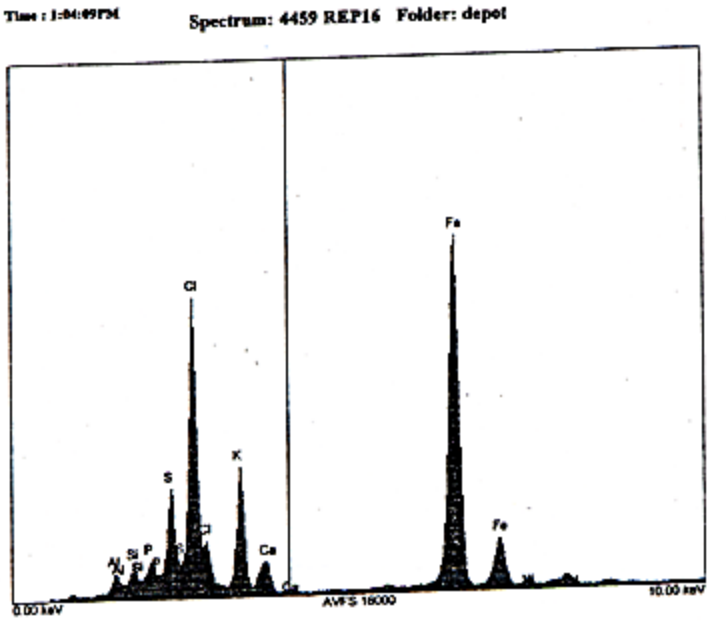


Photography 5 : Reference 25 photography (× 50)

PLATE 21
Sediments extracted from hydraulic fluids



Spectrum 11 : X ray fluorescence spectrum reference 6



Spectrum 12 : X ray fluorescence spectrum reference 16

APPENDIX

BEA

Accident de l'Airbus A 300-600

Immatriculé N14053

survenu le 12 novembre 2001

à New-York (USA)

-:-:-

Peu après le décollage, l'appareil s'écrase après avoir perdu en vol la dérive et la gouverne de direction. Celles-ci sont tombées dans la baie de New York (eau de mer) et ont été récupérées quelques heures plus tard. Ces éléments ont été entreposés à l'air libre après un nettoyage sommaire.

Les trois servocommandes de la gouverne de direction ont été examinées chez le fabricant TRW. Pendant ces examens, divers échantillons de fluides ont été prélevés dans les servocommandes. Les filtres des bancs de tests utilisés ainsi que les filtres des servocommandes, ont également été prélevés.

Le but des travaux confiés au CEPr est de déterminer la nature ainsi que la qualité des fluides prélevés. La pollution des fluides et des filtres sera détaillée.

Les travaux suivants seront réalisés :

Analyse qualitative par chromatographie en phase gazeuse couplée à la spectrométrie de masse (CG/MS),
Viscosité cinématique (mm^2/s) à 40 et 100 °C (EN ISO 3104),
Indice d'acide (mg KOH/g) ASTM D 664,
Teneur en eau (%) NF ISO 6296,
Teneur en sédiments ($mg/100\ cm^3$) gravimétrie 1µm,
Analyse qualitative des sédiments,
Teneur en éléments métalliques (ppm) (BAIRD).