

Docket No. SA-533

Exhibit No. 15-B

NATIONAL TRANSPORTATION SAFETY BOARD

WASHINGTON, D.C.

Materials Laboratory Hydraulic Fluid Testing Factual Report

by

Nancy B. McAtee

(35 Pages)

NATIONAL TRANSPORTATION SAFETY BOARD

Office of Research and Engineering
Materials Laboratory Division
Washington, D.C. 20594



August 26, 2009

MATERIALS LABORATORY FACTUAL REPORT

Report No. 09-052

A. ACCIDENT

Place : Lubbock, Texas
Date : January 27, 2009
Vehicle : ATR 42-320
NTSB No. : CEN09MA142
Investigator : Kristi Dunks
AS-WPR

B. COMPONENTS EXAMINED

Hydraulic fluid test report review for testing conducted for the NTSB and the BEA

C. DETAILS OF THE EXAMINATION

Residue samples from the flap actuators, hydraulic lines and hydraulic filters as well as bulk liquid samples from the hydraulic reservoirs were sent to an independent laboratory for testing. A complete listing of the results including chromatograms is found in the attached reports.

The residues samples from the flap actuators were tested by gas chromatography/mass spectrometry (GC/MS) to determine identity. The samples taken from the right outboard actuator demonstrated no positive peaks in the GC/MS chromatogram. The samples taken from the left outboard actuator were found to be positive for SkydroTM hydraulic fluid.

The residue from the hydraulic filters was tested using inductively-coupled plasma-atomic emissions spectroscopy (ICP-AES). Six samples were submitted. The first sample was from the aircraft's "blue" hydraulic system outboard filter. Several metals were detected but were under the reporting limit for the test procedure. The second sample was from the aircraft's "blue" hydraulic system inboard filter. Calcium was found within reporting limits (11.43 ppm¹). The third sample was from aircraft's "green" hydraulic system outboard filter. Several metals were detected but

¹ The unit *ppm* stands for parts per million

were under the reporting limit for the test procedure. The fourth sample was from aircraft's "green" hydraulic system inboard filter. Silver was found within reporting limits (20.42 ppm). The fifth sample was from return filter #1. Silver was found within reporting limits (18.30 ppm). The sixth sample was from return filter #2. Several metals were detected but were under the reporting limit for the test procedure.

The residue samples from the lines were examined using both GC/MS and ICP-AES to determine the identity of the material found in the lines. The right outboard sample gas chromatogram showed the presence of dibutyl phenyl phosphate, which is a component of hydraulic fluid. The ICP analysis for the right outboard sample found aluminum, cadmium, chromium, copper, iron, manganese, molybdenum, nickel, and zinc. The right inboard sample chromatogram had several peaks consistent with phenols as well as large phosphate ester peaks, consistent with the presence of hydraulic fluid. The ICP analysis for the right inboard sample found aluminum, calcium, cadmium, chromium, copper, iron, magnesium, manganese, nickel, titanium and zinc.

The bulk samples were tested for water content, total acid number and particle size distribution. The testing was done to the specifications outlined in Airbus Technical Specification NSA 307110. All three tests were done on the bulk sample from the "blue" system. The total acid number and water content were within acceptable limits of the Airbus specifications. The particle size distribution numbers were higher than acceptable for all sizes. According to the hydraulic fluid manufacturer, it is recommended that approximately 1 pint of fluid be drained from the reservoir prior to taking a sample. Due to damage from the accident, it was not possible to drain fluid from the system prior to taking the sample.

In addition, the BEA submitted a report to the NTSB for review. The report contained the findings of chemical testing that conducted on the behalf of the BEA. The laboratory analyzed nine samples using Fourier-transform Infrared Spectroscopy (FTIR) and GC/MS. Analysis confirmed that the hydraulic fluid used on the aircraft was Skydrol™ 500 B-4, as expected. In several samples, phenolic compounds were detected. A complete listing of the results including copies of the spectra is found in the attached reports.

Nancy B. McAtee
Chemist



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TEST REPORT FOR:
NTSB
490 L'Enfant Plaza
Washington, DC 20594

Attn: Nancy McAtee

DATE IN: June 8, 2009

P/O #: Credit Card

SAMPLE IDENTIFICATION:

Nine (9) samples were submitted for qualitative analysis by direct injection gas chromatography- mass spectroscopy (GC-MS). Samples matrix consisted of swabs, filter paper, and hydraulic fluid and are identified as follows:

- NTSB Sample #1- 5 - Right OB Actuator
- NTSB Sample #6-7 – Left OB Actuator
- NTSB Sample #8-9 – Blank filter paper and swabs similar to those used to collect NTSB Samples #1-7.
- NTSB Sample #10 – 1 qt. can of fresh Skydrol 500B4 hydraulic fluid.

APPROVED:

A handwritten signature in blue ink that reads "Stephen N. Keller".

Stephen N. Keller
Technical Director



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**DIRECT SOLVENT INJECTION
GAS CHROMATOGRAPHY MASS SPECTROSCOPY
(GCMS)**

SAMPLE PREPARATION:

All samples were prepared in the same fashion. Subsample from Samples #1-5 were combined into one vial with 25 mL of methylene chloride and mixed to dissolve potential analytes within the subsamples. Sample #6, 8 and 9 were prepared in the same fashion as Samples #1-5. Sample #9 was also spiked with Skydrol to identify extraction efficiency. Sample #10 (Skydrol) was diluted with methylene chloride to 100ppm in solution and analyzed to establish a chromatographic "fingerprint."

RESULTS:

Samples #1-5 Right OB Actuator

- Samples #1-5 show no evidence positive chromatographic peaks (see Chromatogram 1). Chromatogram 1 is not significantly different to that of Chromatogram 2, which represents the swab blank (see chromatogram 2 attachment).

Sample #6 Left OB Actuator

- Sample #6 does show positive chromatographic peaks (see Chromatogram 3 attachment). Major peaks in said chromatogram do match those of Chromatogram 4 and 5, which represents the Skydrol and the Skydrol "spiked" swab chromatogram respectively.

In addition, Chromatogram 6 is provided to show a stacked view of the Sample #6 and the Swab Spike chromatograms.



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ANALYSTS:

A handwritten signature in blue ink, appearing to read "Chad Miller".

Chad Miller
Senior Scientist

Attachments: PowerPoint Presentation containing six (6) chromatograms.

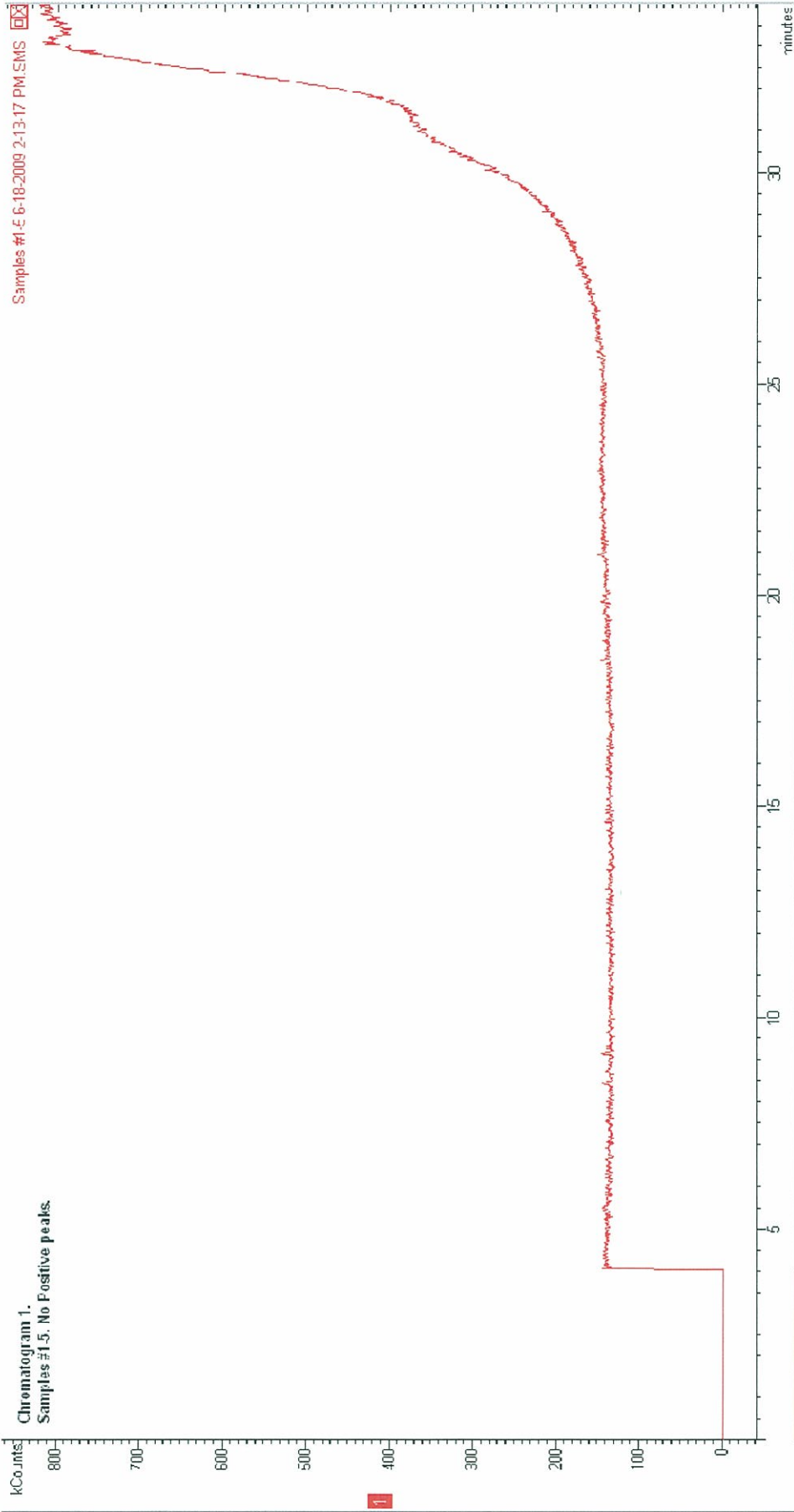


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SO: 72836

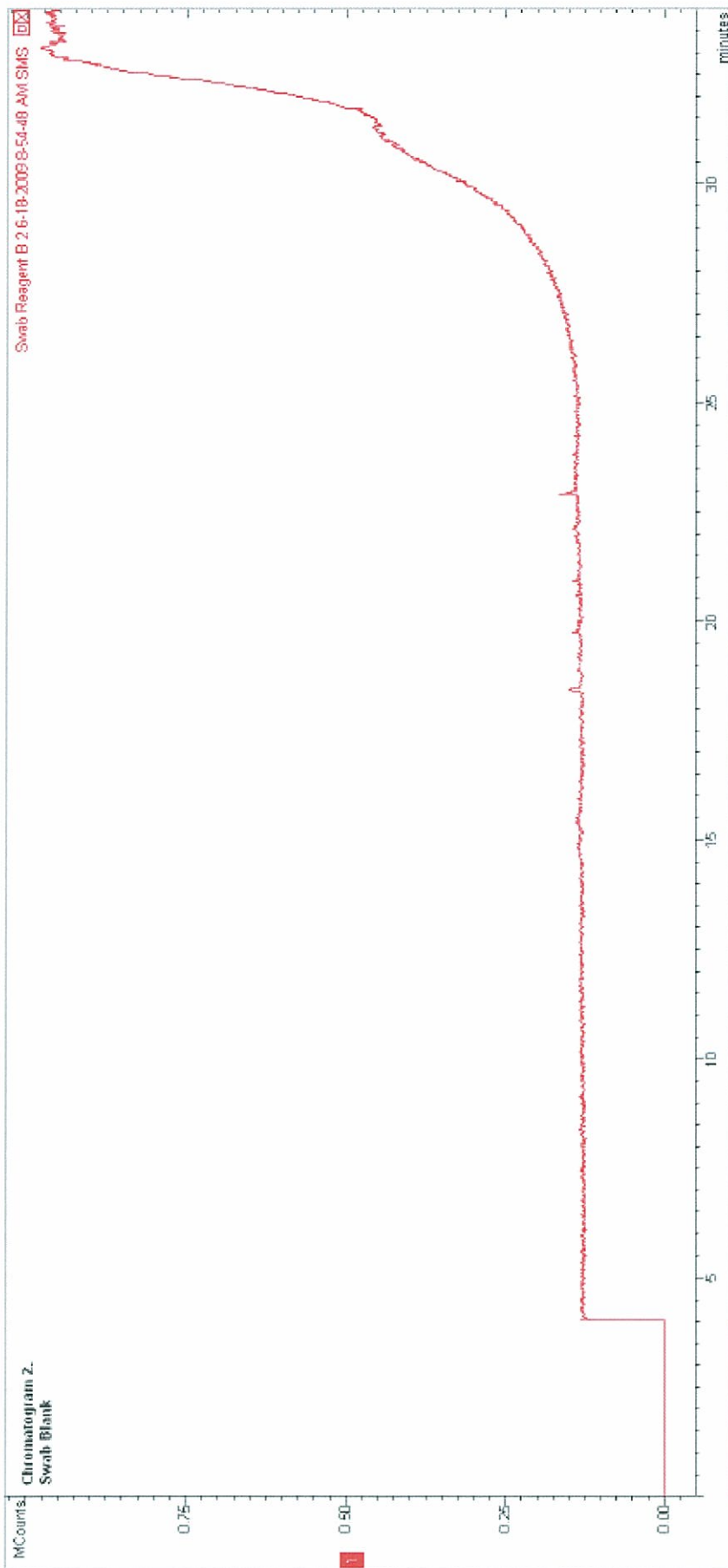
Chromatogram 1: Samples #1-5



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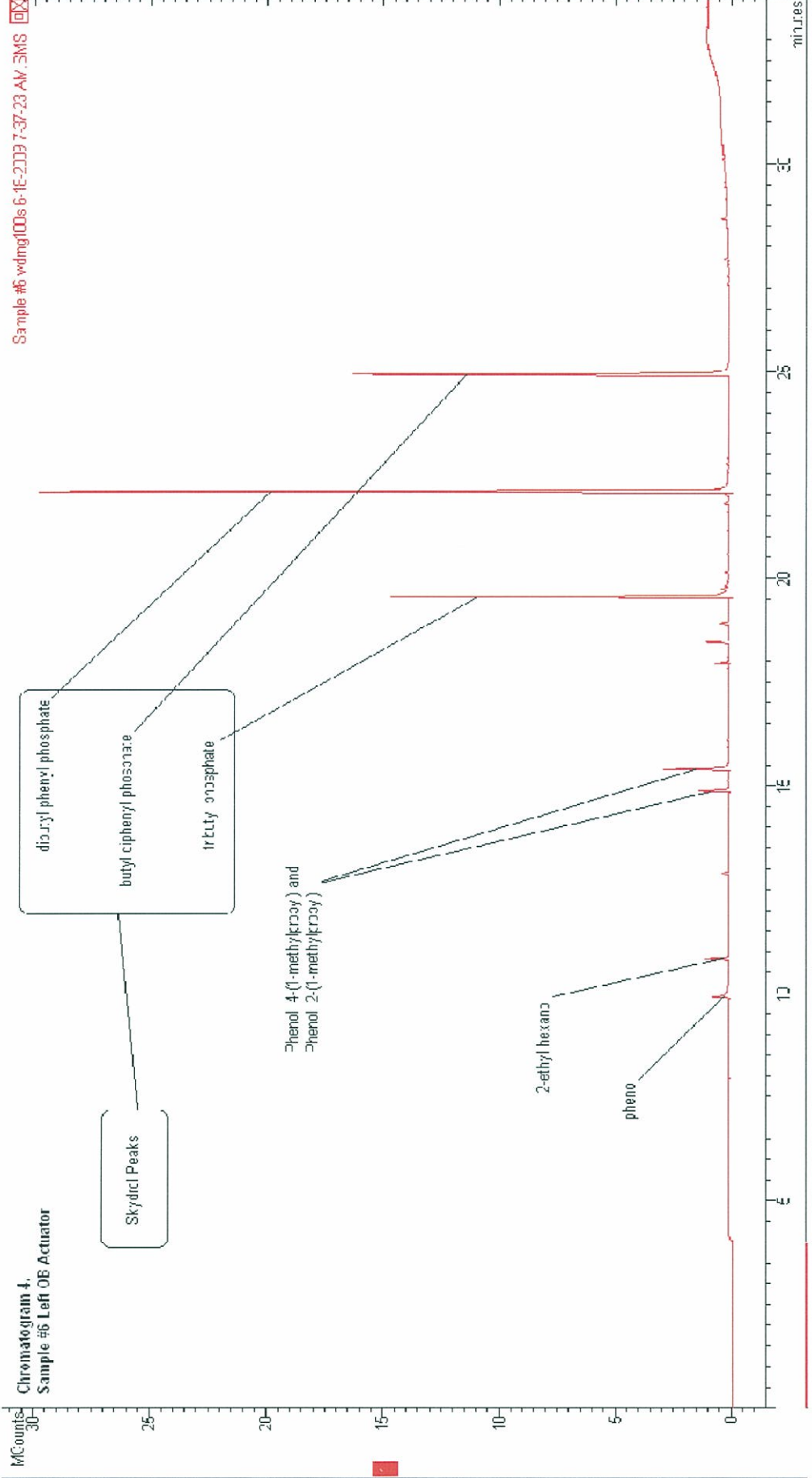
Chromatogram 2: Swab Blank



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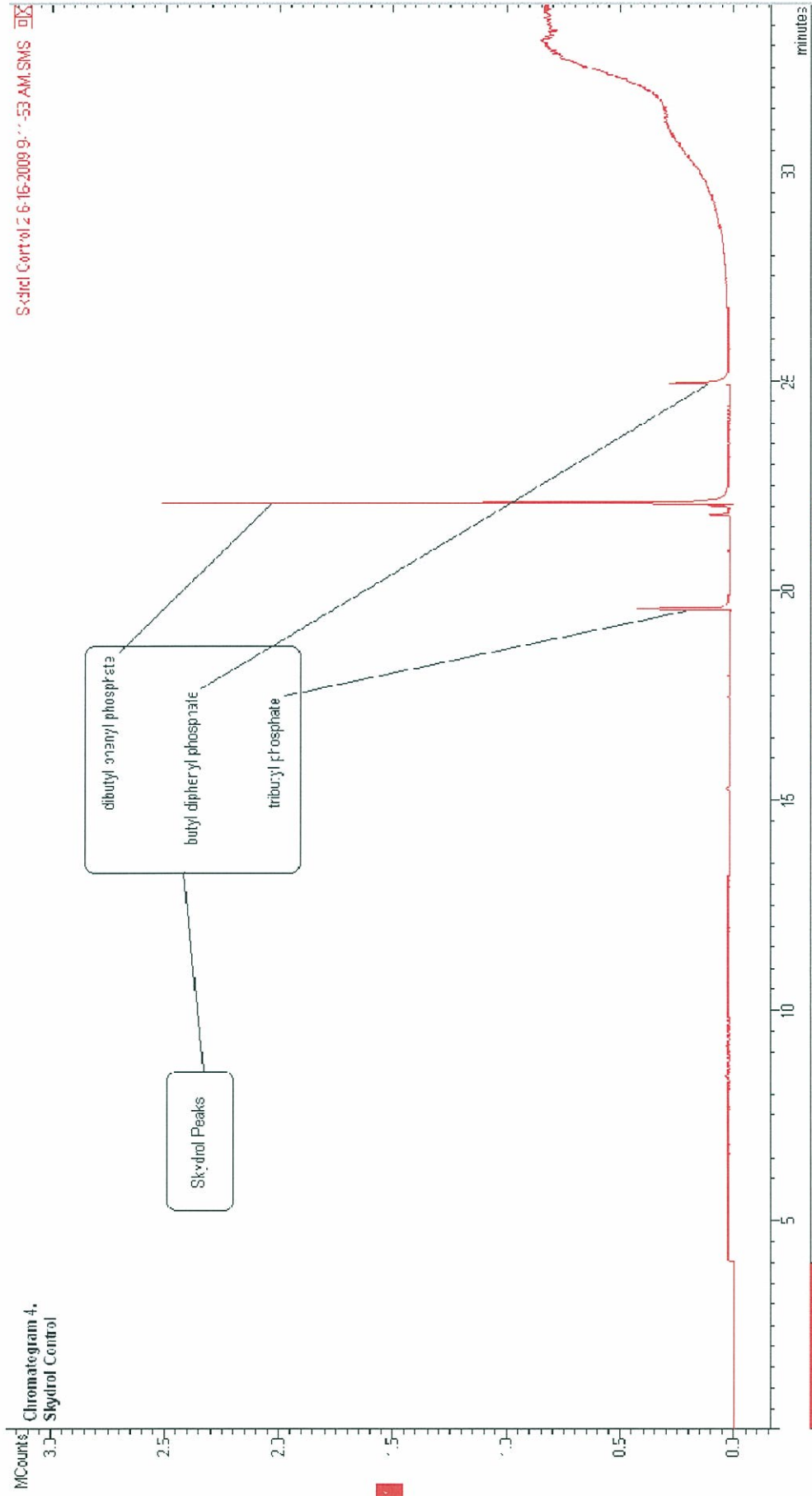
Chromatogram 3: Swab Blank



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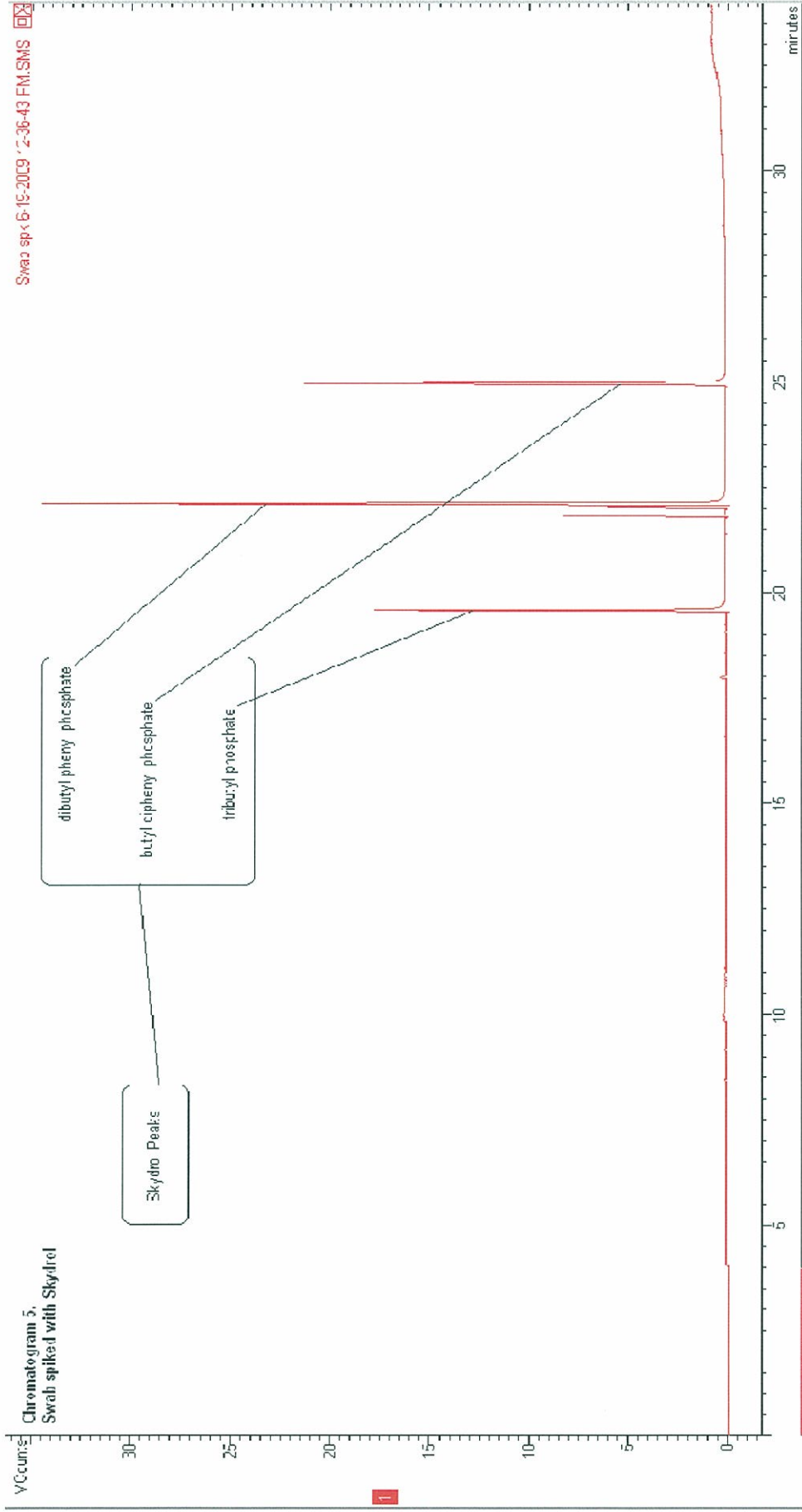
Chromatogram 4: Skydrol Control



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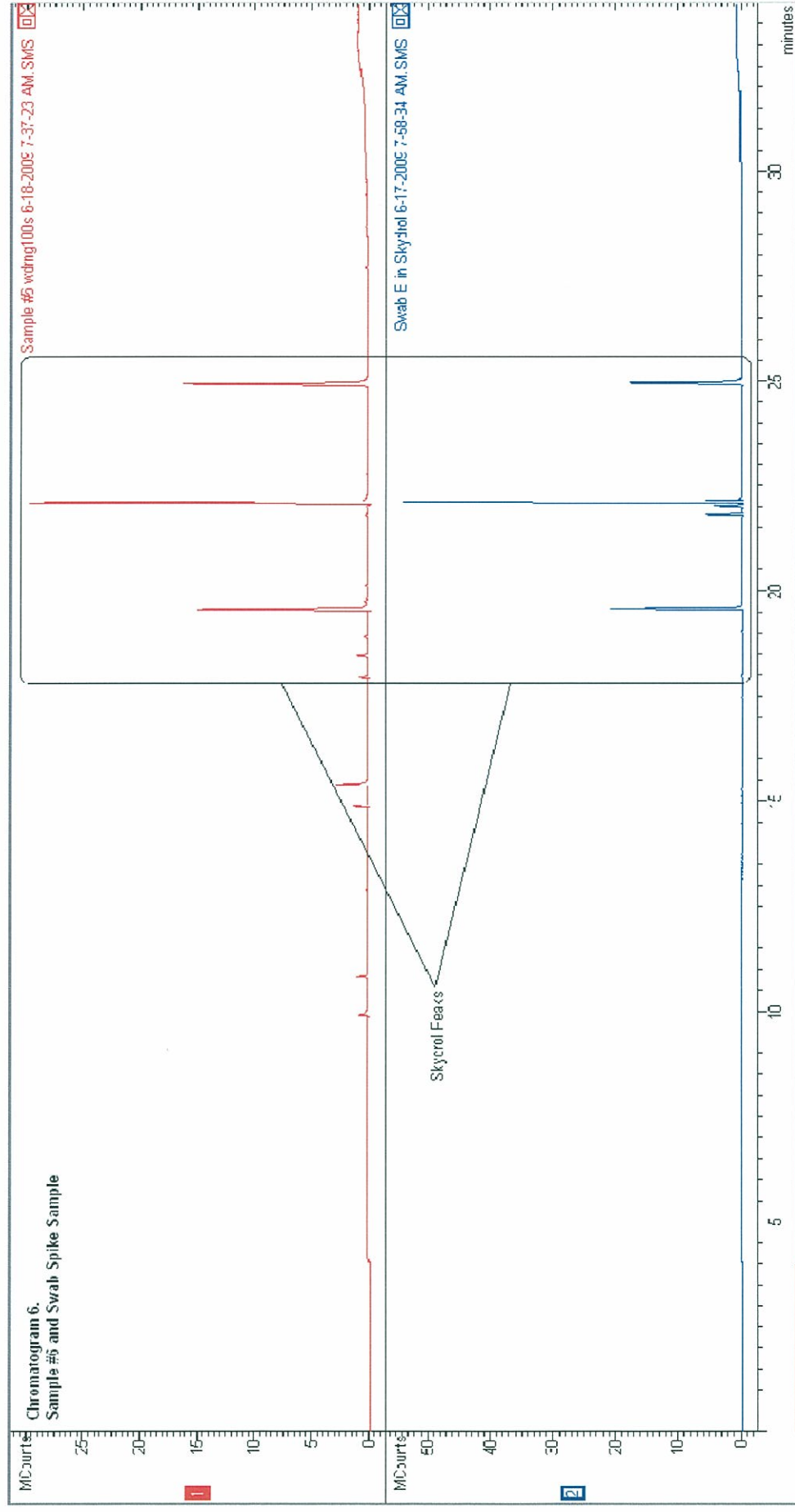
Chromatogram 5: Swab Spike w/Skydrol



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Chromatogram 6: Sample #6 and Swab Spike Stacked



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TEST REPORT (AMENDED) FOR:

NTSB
490 L'Enfant Plaza
Washington, DC 20594

Attn: Nancy McAtee

DATE IN: June 8, 2009

P/O #: Credit Card

SAMPLE IDENTIFICATION:

Six (6) sample filters were submitted for the examination of the hydraulic fluid content for elements (metals) via inductively coupled plasma-optical emissions spectroscopy (ICP-OES). Samples are identified as follows:

- Sample 1: NTSB Sample #13- Blue system OB filter
- Sample 2: NTSB Sample #14- Blue system IB filter
- Sample 3: NTSB Sample #15- Green system OB filter
- Sample 4: NTSB Sample #16- Green system IB filter
- Sample 5: NTSB Sample #17- Return filter #1
- Sample 6: NTSB Sample #18- Return filter #2

APPROVED:

A handwritten signature in blue ink that reads "Stephen N. Keller".

Stephen N. Keller
Technical Director



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SAMPLE PREPARATION:

An aliquot of hydraulic fluid was taken from each sample filter and digested by closed vessel microwave power with a nitric acid / hydrogen peroxide reagent. Digestate was then diluted and subsequently analyzed by ICP-OES.

RESULTS: Quantitative elemental analysis of hydraulic fluid by ICP-OES (ASTM E1613). Results are reported as parts per million (ppm).

| Element-Wavelength(nm) | NTSB #13 | NTSB #14 | NTSB #15 | NTSB #16 | NTSB #17 | NTSB #18 |
|------------------------|----------|----------|----------|----------|----------|----------|
| Ag 328.068 | 1.23U | 0.77U | 0.30 U | 20.42 | 18.30 | 0.37 U |
| Al 308.215 | ND | 0.31 U | ND | ND | ND | ND |
| As 188.980 | ND | ND | ND | ND | ND | ND |
| Ba 585.367 | ND | 0.19 U | ND | ND | ND | ND |
| Be 313.107 | ND | ND | ND | ND | ND | 0.00 |
| Ca 315.887 | 10.00 U | 11.43 | 6.88 U | 7.12 U | 6.06 U | 6.80 U |
| Cd 226.502 | 1.36 U | 0.50 U | 0.23 U | 0.27 U | 0.32 U | 0.23 U |
| Co 228.615 | ND | ND | ND | ND | ND | ND |
| Cr 267.716 | 0.42 U | 0.25 U | 0.27 U | 0.26 U | 0.35 U | 0.33 U |
| Cu 324.754 | ND | ND | ND | ND | ND | ND |
| Fe 261.187 | 0.70 U | 0.71 U | 0.77 U | 0.48 U | 0.35 U | 0.30 U |
| Mg 279.078 | 0.31 U | 0.44 U | ND | ND | ND | ND |
| Mn 257.610 | ND | ND | ND | ND | ND | ND |
| Mo 202.032 | 1.23 U | 0.79 U | ND | 0.31 U | 0.25 U | ND |
| Ni 231.604 | ND | ND | ND | ND | ND | ND |
| Pb 220.353 | 0.30 U | 0.25 U | ND | ND | 0.32 U | ND |
| Sb 206.834 | ND | ND | ND | ND | ND | ND |
| Se 196.026 | 1.60 U | 1.84 U | ND | 3.00 U | 0.68 U | 0.89 U |
| Ti 337.280 | 0.57 U | 2.19 U | 1.46 U | 2.56 U | 1.80 U | 0.53 U |
| Tl 190.794 | ND | 0.03 U | ND | ND | ND | ND |
| V 311.837 | ND | ND | ND | 0.02 U | ND | 0.02 U |
| Zn 206.200 | 1.23 U | ND | 0.25 U | ND | ND | ND |

U: Results detected but under the reporting limit.
 *ND: None Detect: Below reporting limit.





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ANALYSTS:

Joel Ford

Joel Ford
Laboratory Technician



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TEST REPORT FOR:
NTSB
490 L'Enfant Plaza SW
Washington, DC 20594

Attn: Nancy McAtee

DATE IN: June 8, 2009

P/O #: Credit Card

SUBMISSION IDENTIFICATION: Two (2) hydraulic fluid samples designated as N902FX "Blue System #11" and N902FX "Green System #12" were submitted for the following analysis; Total Acid Number (ASTM D664), Water By Karl Fischer (ASTM D1744), and Particle Size Distribution by HIAC Royco NAS 1630/ISO 4406.

We offer our results and observations.

APPROVED:

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Stephen N. Keller
Technical Director



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Page 1 of 3



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RESULTS:

Sample Identification: N902FX Blue System #11

| Parameter | Method | Test Value | Test Unit (s) | Note |
|----------------------------------|------------|------------|---------------|------|
| Total Acid Number | ASTM D664 | <0.05 | mgKOH/g | |
| Water Content by Karl Fischer | ASTM D1744 | 0.6 | % | |
| Particle Size Distribution/100ml | HIAC ROYCO | 0.00 | | |
| 5-15 microns | HIAC ROYCO | 561000 | | |
| 16-25 microns | HIAC ROYCO | 144000 | | |
| 26-50 microns | HIAC ROYCO | 52600 | | |
| 51-100 microns | HIAC ROYCO | 10400 | | |
| >100 microns | HIAC ROYCO | 1650 | | |

Sample Identification: N902FX Green System #12

| Parameter | Method | Test Value | Test Unit (s) | Note |
|----------------------------------|------------|------------|---------------|------------------------------|
| Total Acid Number | ASTM D664 | * | mgKOH/g | * Insufficient Sample Volume |
| Water Content by Karl Fischer | ASTM D1744 | 0.7 | % | |
| Particle Size Distribution/100ml | HIAC ROYCO | 0.00 | | |
| 5-15 microns | HIAC ROYCO | 238000 | | |
| 16-25 microns | HIAC ROYCO | 20400 | | |
| 26-50 microns | HIAC ROYCO | 12100 | | |
| 51-100 microns | HIAC ROYCO | 4000 | | |
| >100 microns | HIAC ROYCO | 790 | | |



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PERFORMED BY:

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Chad Miller
Senior Scientist

SAMPLE DISPOSITION: Samples consumed during analysis.



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TEST REPORT FOR:
NTSB
490 L'Enfant Plaza
Washington, DC 20594

Attn: Nancy McAtee

DATE IN: August 4, 2009

P/O #: Credit Card

SAMPLE IDENTIFICATION:

Two (2) samples were submitted for qualitative analysis by direct injection gas chromatography- mass spectroscopy (GC-MS), and quantitative elemental analysis by inductively coupled plasma-optical emissions spectroscopy (ICP-OES). Sample matrix consisted of unknown residue, black in color, contained in respective test tubes inside of glass jars padded with paper towels. Sample identification is as follows:

- Right Outboard Actuator (ROB) Residue Sample
- Right Inboard Actuator (RIB) Residue Sample

APPROVED:

A handwritten signature in black ink, appearing to read "Stephen N. Keller".

Stephen N. Keller
Technical Director



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**DIRECT SOLVENT INJECTION
GAS CHROMATOGRAPHY MASS SPECTROSCOPY
(GCMS)**

SAMPLE PREPARATION:

All samples were prepared in the same fashion. A Small subsample of residue was transferred to 1 mL of methylene chloride (dichloromethane) and shaken to facilitate dissolution. Subsequently, 1 μ L of solution was injected into the GC for analysis.

RESULTS:

Right Outboard Actuator (ROB) Residue Sample

- The ROB sample chromatogram expresses only one small identifiable peak for dibutyl phenyl phosphate. All other peaks were not identifiable and are barely above background level (see attachment).

Right Inboard Actuator (RIB) Residue Sample

- The RIB sample chromatogram (see attachment) expresses many positive peaks mainly consisting of phenols. There are also large phosphate ester peaks in the chromatogram, which may indicate the presence of hydraulic fluid.



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**QUANTITATIVE ELEMENTAL ANALYSIS BY INDUCTIVELY COUPLED PLASMA-OPTICAL
 EMISSIONS SPECTROSCOPY
 (ICP-OES)**

SAMPLE PREPARATION:

An aliquot of hydraulic fluid was taken from each sample filter and digested by closed vessel microwave power with a nitric acid / hydrogen peroxide reagent. Digestate was then diluted and subsequently analyzed by ICP-OES.

RESULTS: Quantitative elemental analysis of hydraulic fluid by ICP-OES (ASTM E1613). Results are reported as parts per million (ppm).

| Element-Wavelength(nm) | RIB | ROB |
|-------------------------|-----------|-----------|
| Reporting Limit: | 61 | 53 |
| Ag 328.068 | ND | ND |
| Al 308.215 | 1227 | 65 |
| As 188.980 | ND | ND |
| Ba 585.367 | ND | ND |
| Be 313.107 | ND | ND |
| Ca 315.887 | 113 | ND |
| Cd 226.502 | 175 | 4600 |
| Co 228.615 | ND | ND |
| Cr 267.716 | 1414 | 3347 |
| Cu 324.754 | 89 | 204 |
| Fe 261.187 | 5763 | 17083 |
| Mg 279.078 | 254 | ND |
| Mn 257.610 | 217 | 335 |
| Mo 202.032 | ND | 59 |
| Ni 231.604 | 778 | 2279 |
| Pb 220.353 | ND | ND |
| Sb 206.834 | ND | ND |
| Se 196.026 | ND | ND |
| Ti 337.280 | 24 | ND |
| Tl 190.794 | ND | ND |
| V 311.837 | ND | ND |
| Zn 206.200 | 1215 | 1060 |

ND: None Detect: Below reporting limit.



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ANALYSTS:

A handwritten signature in blue ink, appearing to read "Chad Miller".

Chad Miller
Senior Scientist

Joel Ford

Joel Ford
Laboratory Technician

Attachments: PowerPoint Presentation containing two (4) chromatograms.

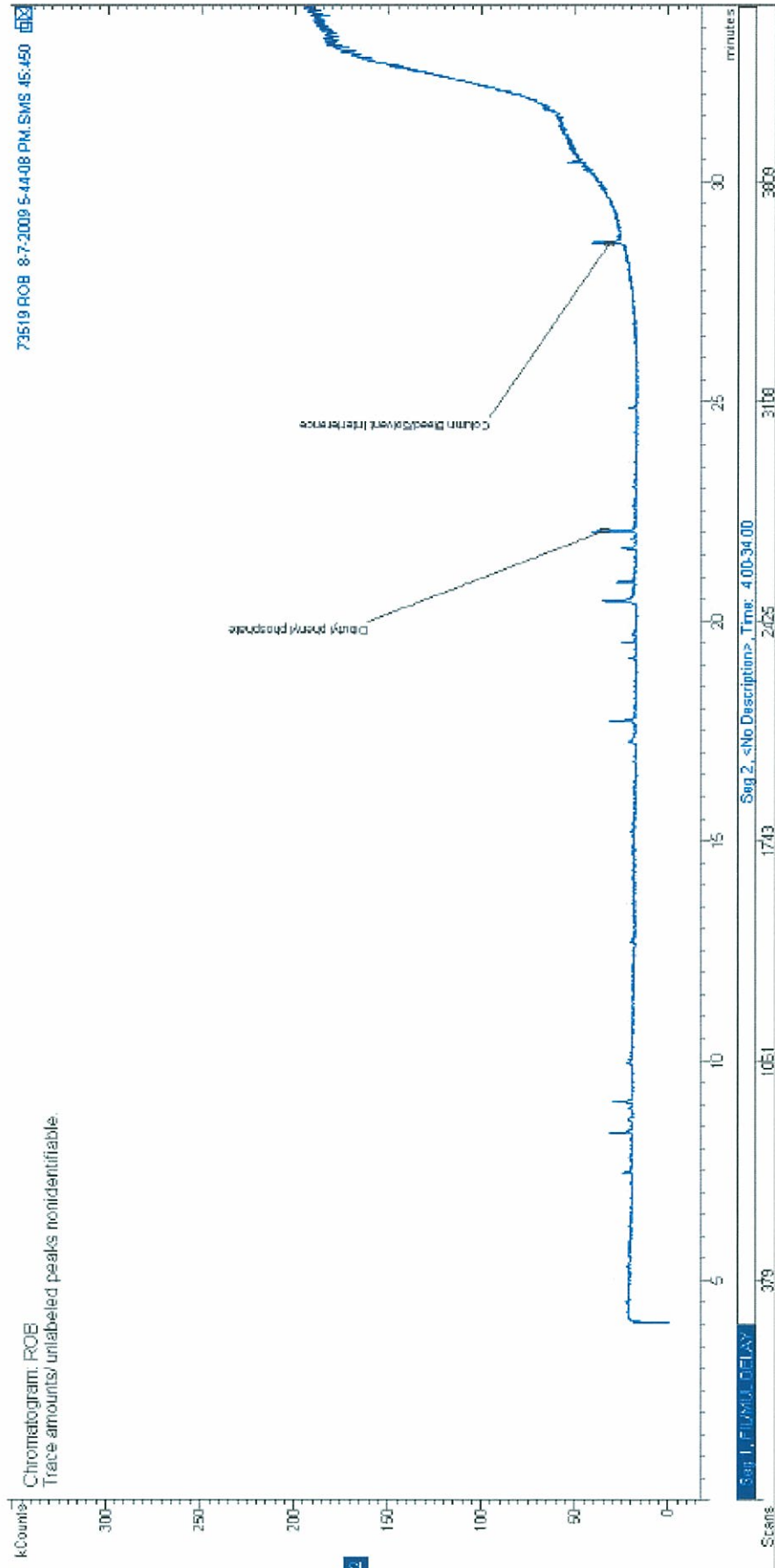


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SO: 73519

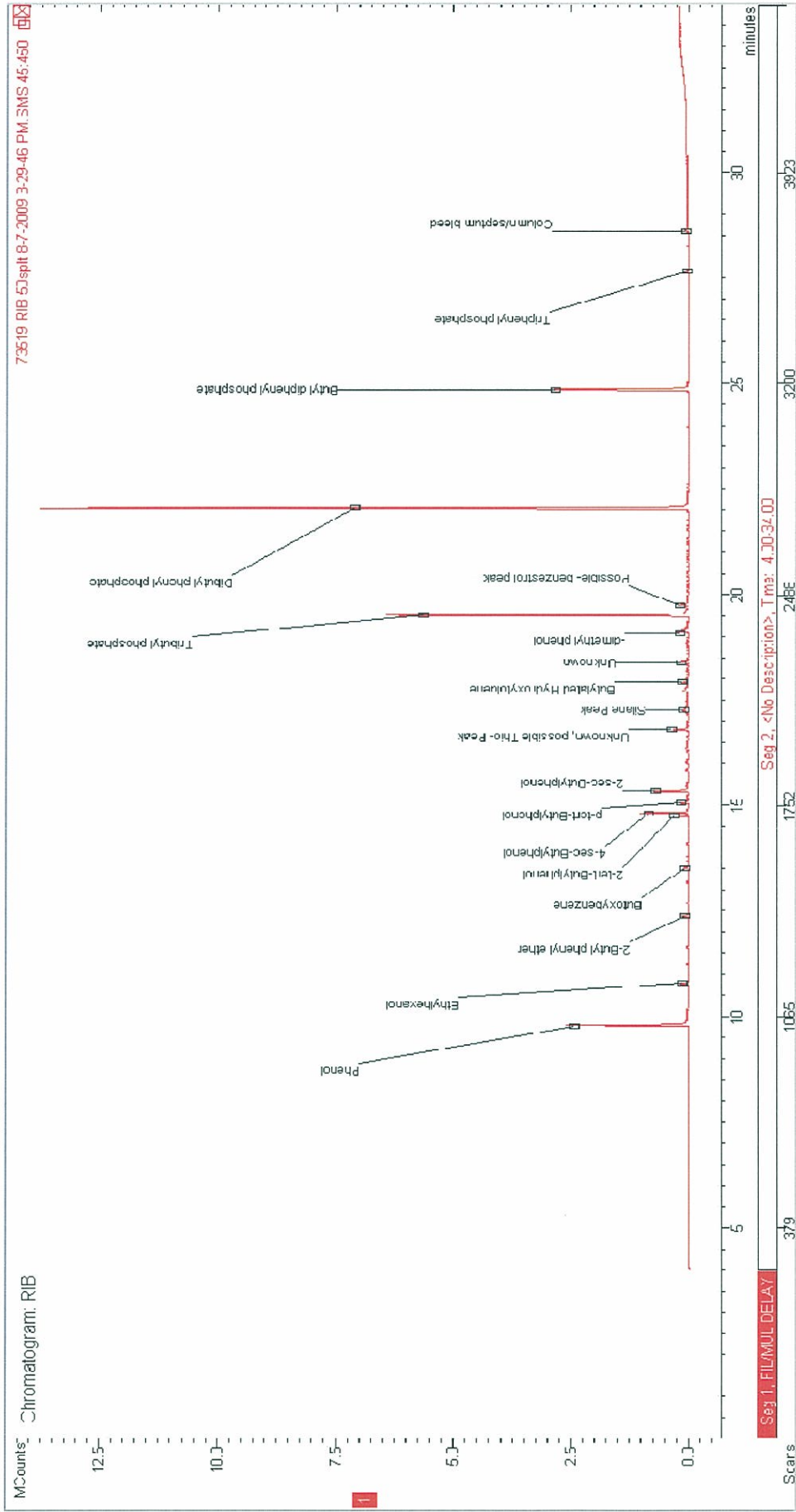
Chromatogram 1: Right Outboard Actuator



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SO: 73519

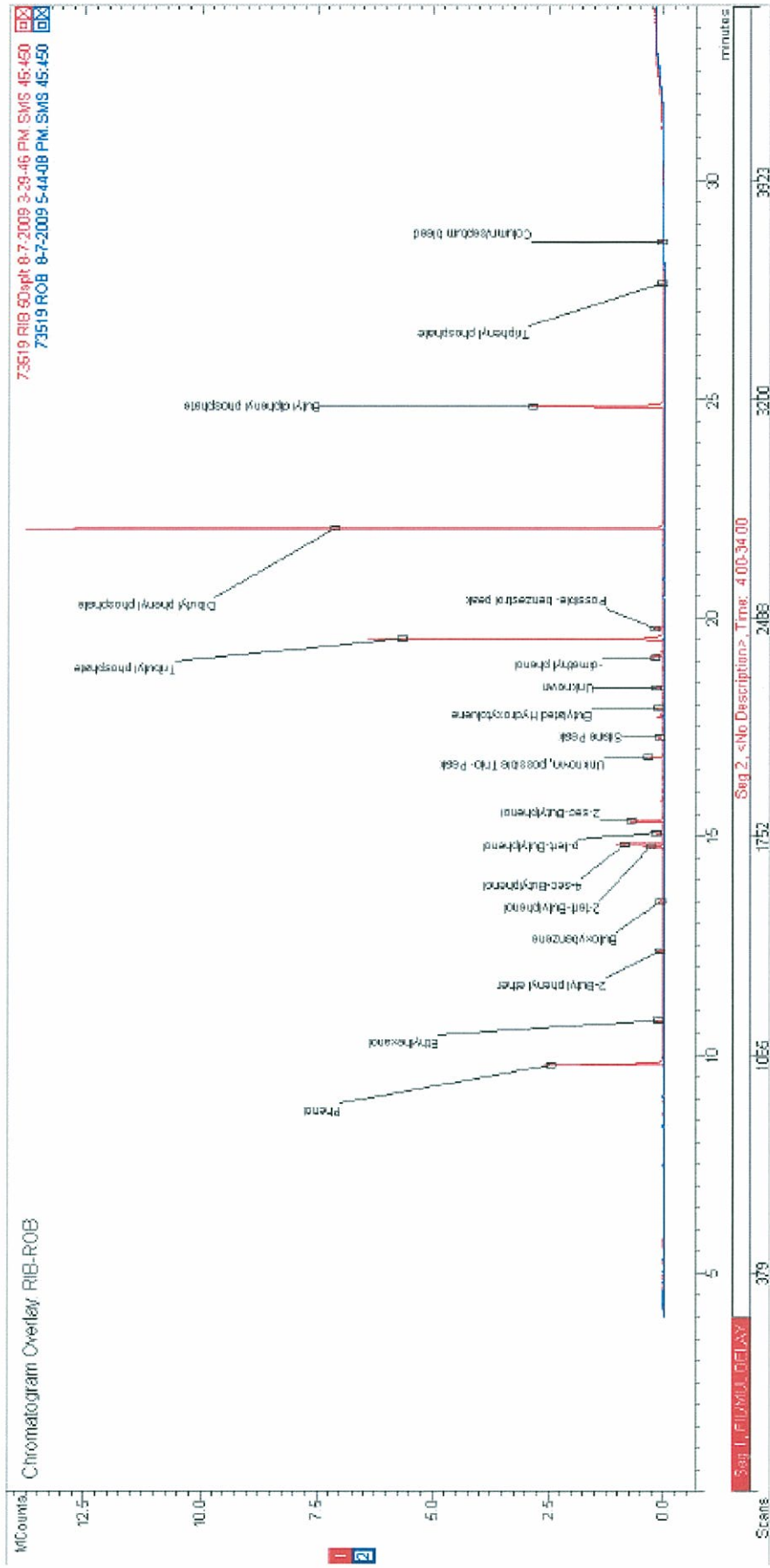
Chromatogram 2: Right Inboard Actuator



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SO: 73519

Chromatogram 3: Overlay: RIB/ROB



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SO: 73519 Chromatogram 4: Blank



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INVESTIGATION REPORT

39 - DAI - 09
OT N°5353

Investigation object : analysis of 9 samples of hydraulic fluid in use on ATR42, registered N902FX, crashed on the 01-27-09, in AD Lubbock (USA)

References : BEA Enquiry request N°2009/07 from the 04-08-09, from Ch. Menez.

Date of sample arrival : 04-09-09 and 05-12-09
Beginning of investigation : 04-24-09
End of investigation : 05-13-09

We were required to analyse nine samples of hydraulic fluid, mainly to check that the expected hydraulic fluid in use on ATR42 was skydrol 500 B-4, to detect possible pollutions. According to NTSB request, the nine samples were analysed by FTIR spectrometrical analysis.

RESULTS SUMMARY

Chromatographycal and spectrometrical analysis confirmed that the hydraulic fluid used on the ATR 42, registered N902FX, was skydrol 500 B-4, as expected. No intial organic pollution was detected.

In chromatograms and spectra from samples 6, 7 (hydraulic jack 3) and 9 (hydraulic jack 4), phenol and derivatives were detected. They probably resulted from the degradation of skydrol compounds during combustion, when the plane caught fire.

Results given in this tests report concern only the samples received by the laboratory. Reproduction of this report is authorized only in the form of an integral photographic facsimile. THIS ENGLISH COPY IS ONLY FOR INFORMATION. THE ORIGINAL COPY IS IN FRENCH.

COMPOSITION


| | | | |
|-------------|-------------|---------------|-------------------------------|
| Pages 10 | Tables 2 | Appendix - | Bibliographic references - |
|-------------|-------------|---------------|-------------------------------|

ARCHIVING REFERENCES

Identification theme: ATR 42 - hydraulic fluid - skydrol 500 B-4

Key words : hydraulic fluid - skydrol 500 B-4 - GC/MS - FTIR

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EXTERNAL DISTRIBUTION : BEA/SAC : Ch. Menez, J-F. Berthier, G. Romeuf (3 ex + 1 fichier PDF : photos JPEG).

1 - INTRODUCTION

Following the crash of the ATR 42 (registration n° N902FX) on the 01.27.09, we were requested by the BEA to analyse nine samples of hydraulic fluid, with the following objectives.

The aim of the labwork was :

- to check if that the hydraulic fluid in use in ATR42 was skydrol 500 B-4,
- to detect potential pollutions,
- to analyse the nine samples with FTIR spectrometrical analysis according to NTSB request.

2 - SAMPLES IDENTIFICATION

The samples we received were labelled as follows :

- Sample 1 : thick black liquid 1a, sampled from "small room",
- Sample 2 : black liquid 1b, sampled from "large room",
- Sample 3 : clear mauve liquid 1c sampled from "large room",
- Sample 4 : residue 2, sampled from "small room",
- Sample 5 : residue 2, sampled from "large room",
- Sample 6 : thick black liquid 3,
- Sample 7 : black liquid 3,
- Sample 8 : thick black liquid 4, sampled from "small room",
- Sample 9 : black liquid 4, sampled from "large room".

Sample 32 (file 107DAI07) : reference of skydrol 500 B-4 (supplied by BEA).

As samples 4 and 5 were solids, neither chromatographical analysis, nor spectrometrical analysis could had been performed.

3 - METHODOLOGY

The samples were analysed by gas chromatography coupled with a mass spectrometer (GC/MS) and by Fourier Transform Infra-Red (FTIR) spectrometrical analysis.

4 - FINDINGS AND COMMENTS

3.1 Gas chromatography coupled with a mass spectrometer (GC/MS)

Chromatograms were shown on table 1 (reference of skydrol 500 B-4 was picture 1 and the analysed samples were from picture 2 to picture 9).

Chromatograms were all in compliance with the referenced hydraulic fluid skydrol 500 B-4 one, analysed under the same conditions.

In chromatograms from samples 1, 6, 7, 8, and 9, some peacks were very reduced by comparison with the reference. And, they were only detected as traces. Those fluids may had been altered when plane burned. Therefore, we could say that no initial organic pollution was detected.

In chromatograms from samples 6 and 7 (hydraulic jack 3) and sample 9 (hydraulic jack 4), phenol and derivates were detected. They probably resulted from degradation of skydrol compounds during combustion, when the plane caught fire.

3.2 Fourier Transform Infra-Red spectrometrical analysis

Spectra were shown on table 2, from picture 1 to picture 8.

Spectra of samples 1, 2, 3, 8 and 9 were in compliance with the spectrum of the hydraulic fluid skydrol 500 B-4 one, supplied by BEA as a reference for comparison.

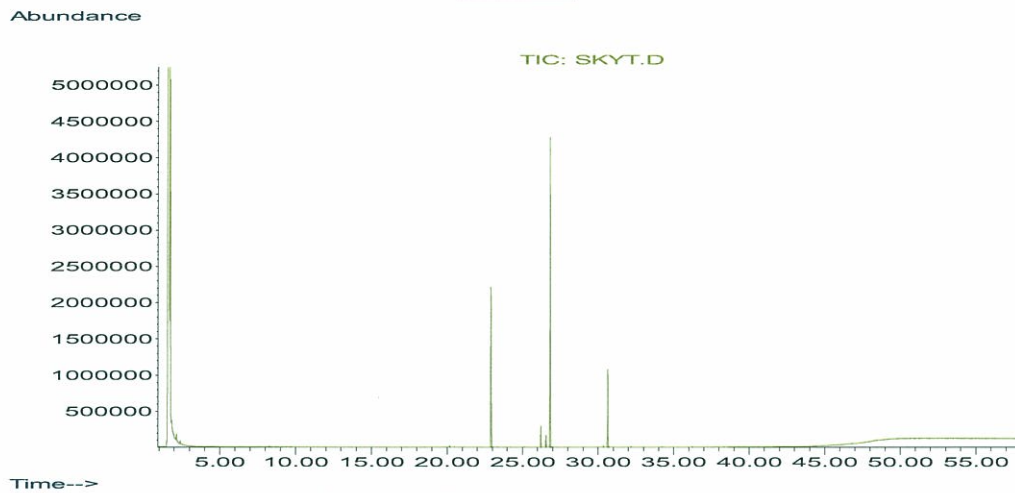
Spectra of samples 6 and 7 (hydraulic jack 3) showed significant differences mainly in the wavelengths from 2500 to 3200 cm^{-1} and from 1000 to 1300 cm^{-1} . All this pointed out a degradation of the original hydraulic fluid, probably when the plane caught fire.

5 - CONCLUSION

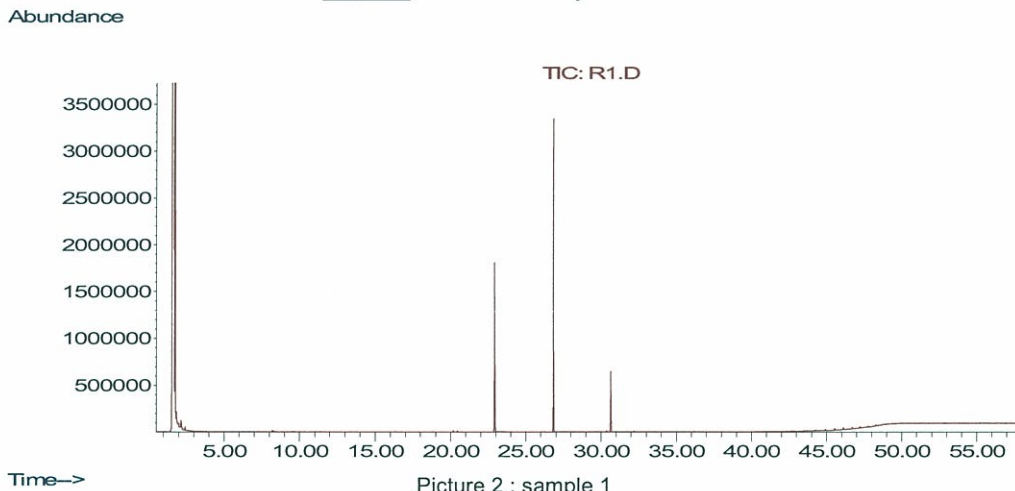
Chromatographycal and spectrometrical analysis confirmed that the hydraulic fluid used on ATR 42, registered N902FX, was skydrol 500 B-4, as expected.
No initial organic pollution was detected.

In chromatograms and spectrum from samples 6, 7 (hydraulic jack 3) and sample 9 (hydraulic jack 4), phenol and derivates were detected. They probably resulted from the degradation of skydrol compounds during combustion, when the plane caught fire.

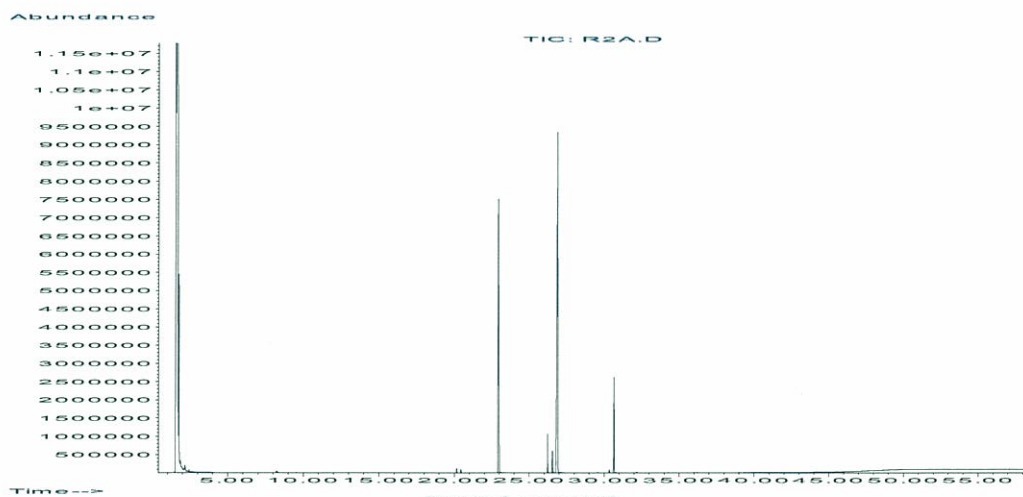
TABLE 1



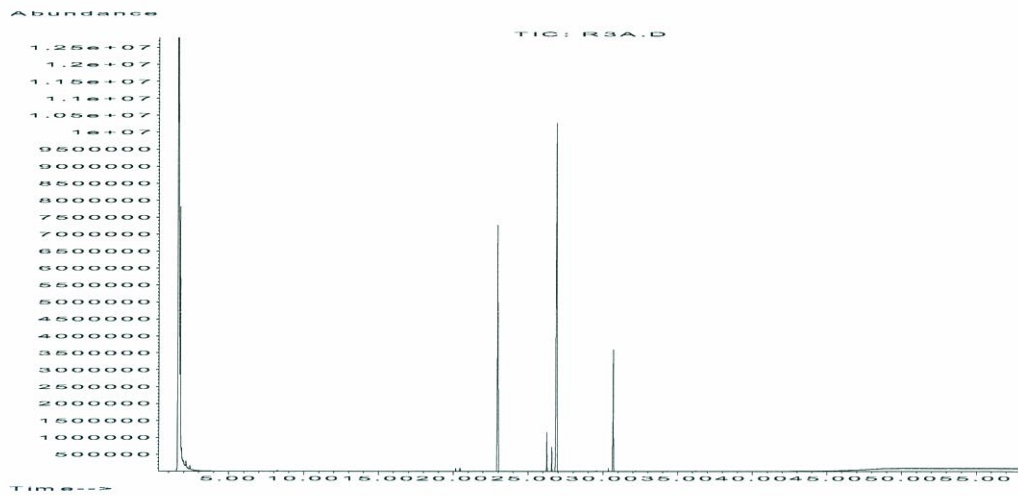
Picture 1 : reference of skydrol 500 B-4



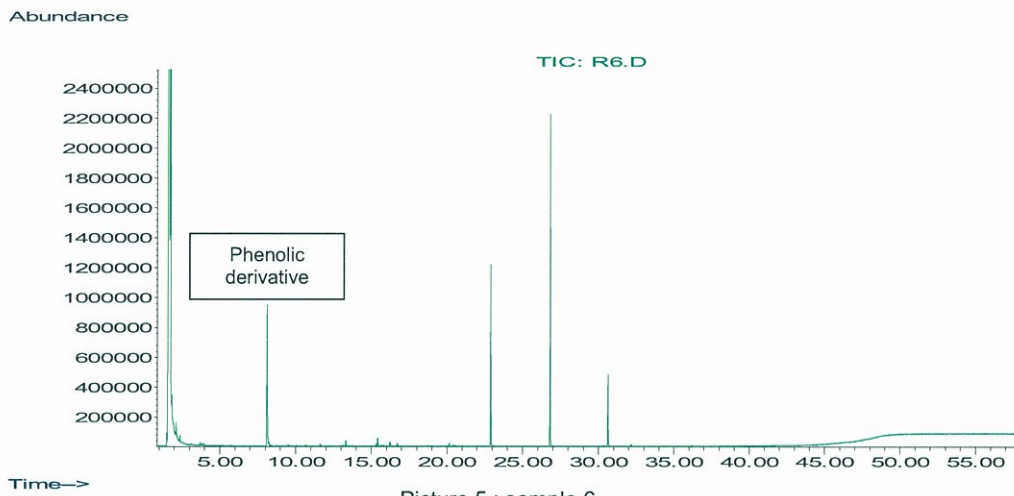
Picture 2 : sample 1



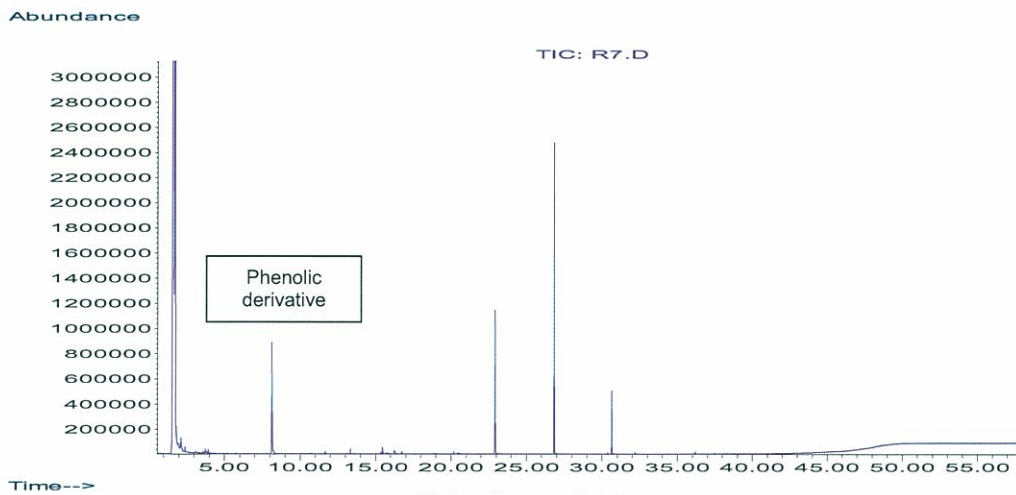
Picture 3 : sample 2



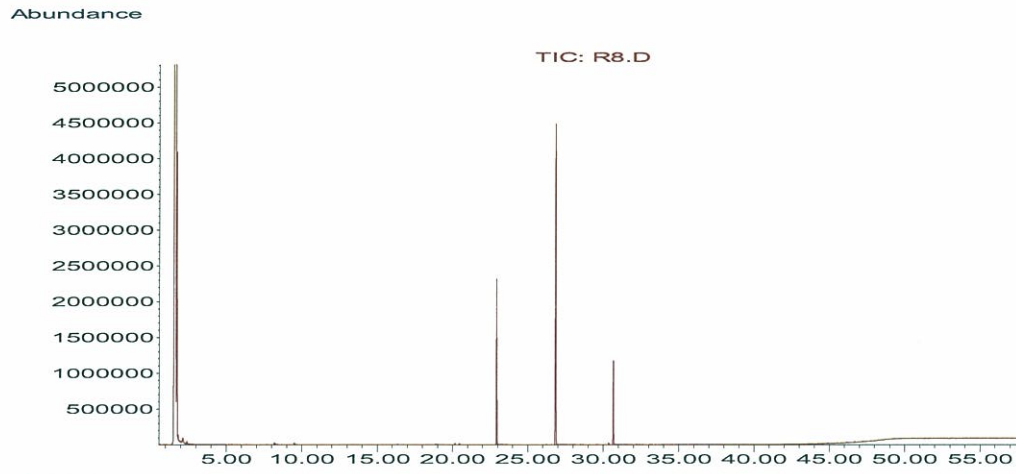
Picture 4 : sample 3



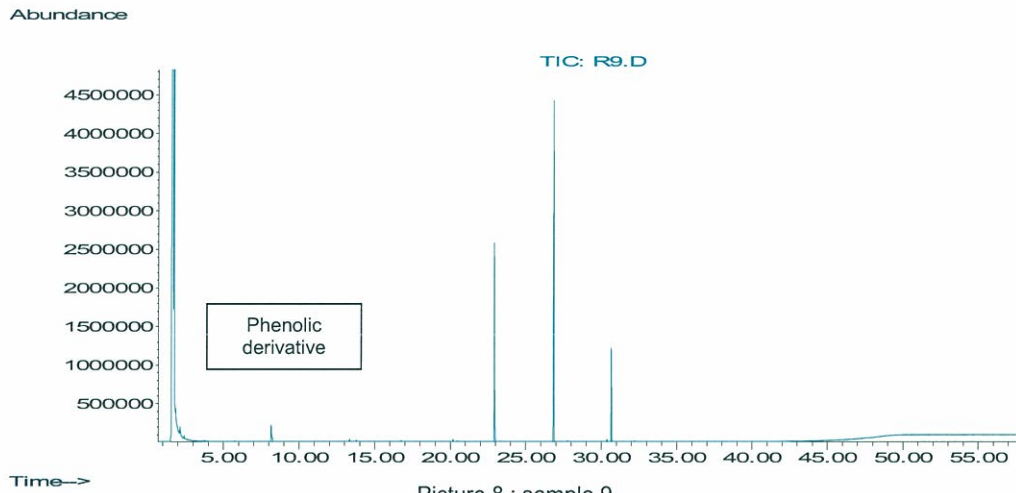
Picture 5 : sample 6



Picture 6 : sample 7

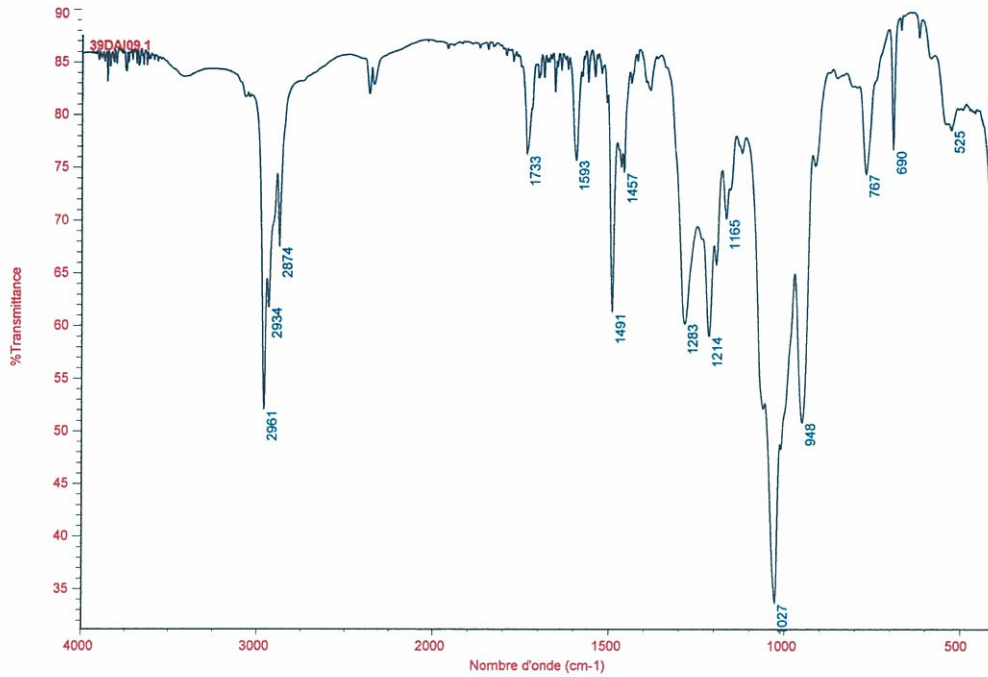


Picture 7 : sample 8

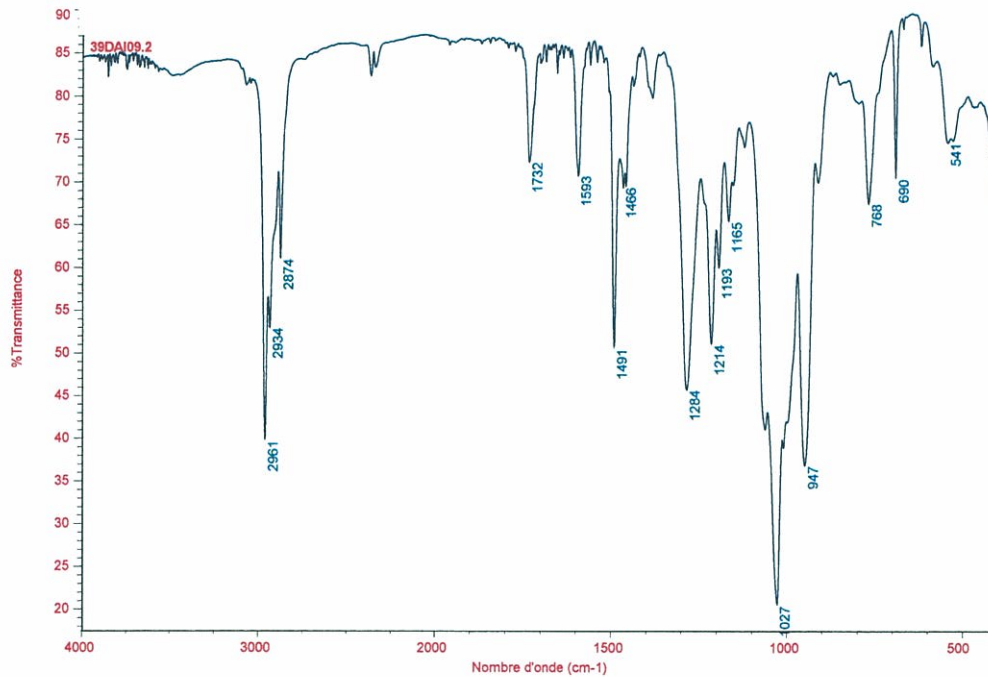


Picture 8 : sample 9

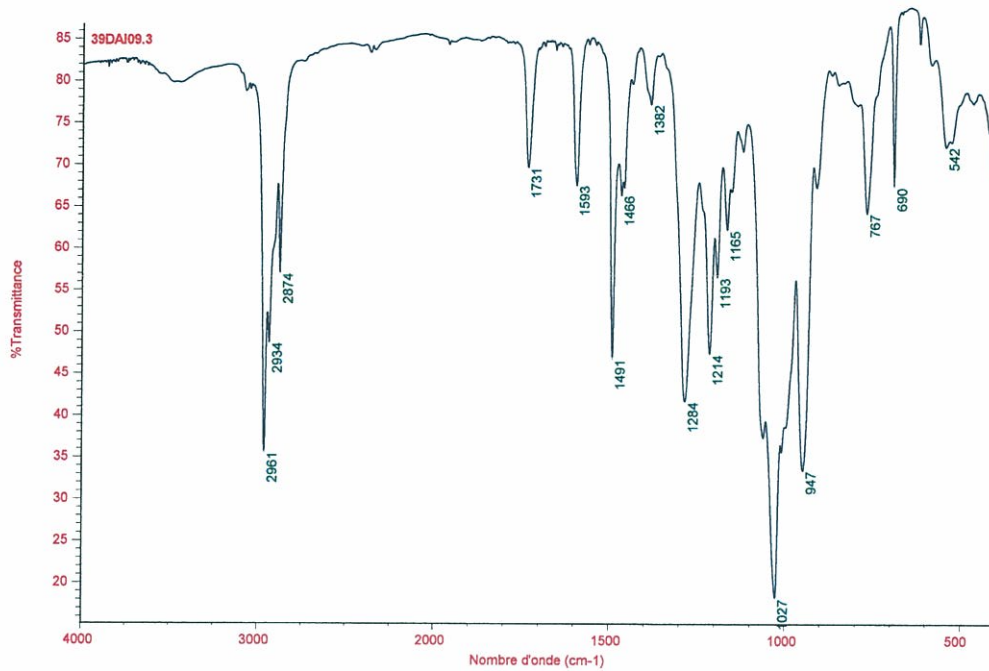
TABLE 2



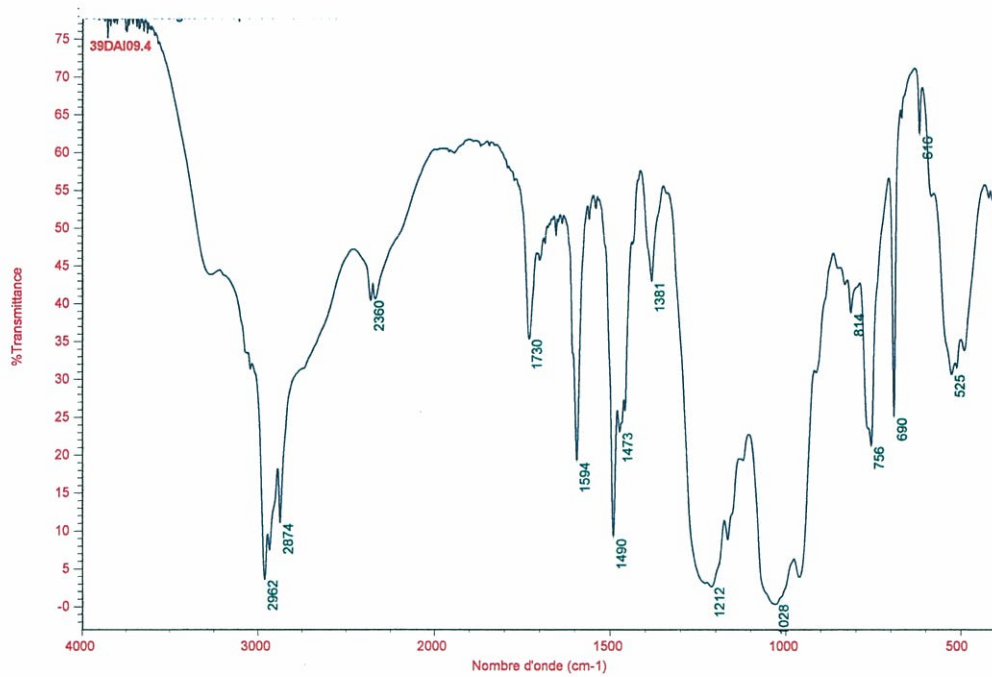
Spectrum 1 : thick black liquid 1a, sampled from "small room" (sample 1)



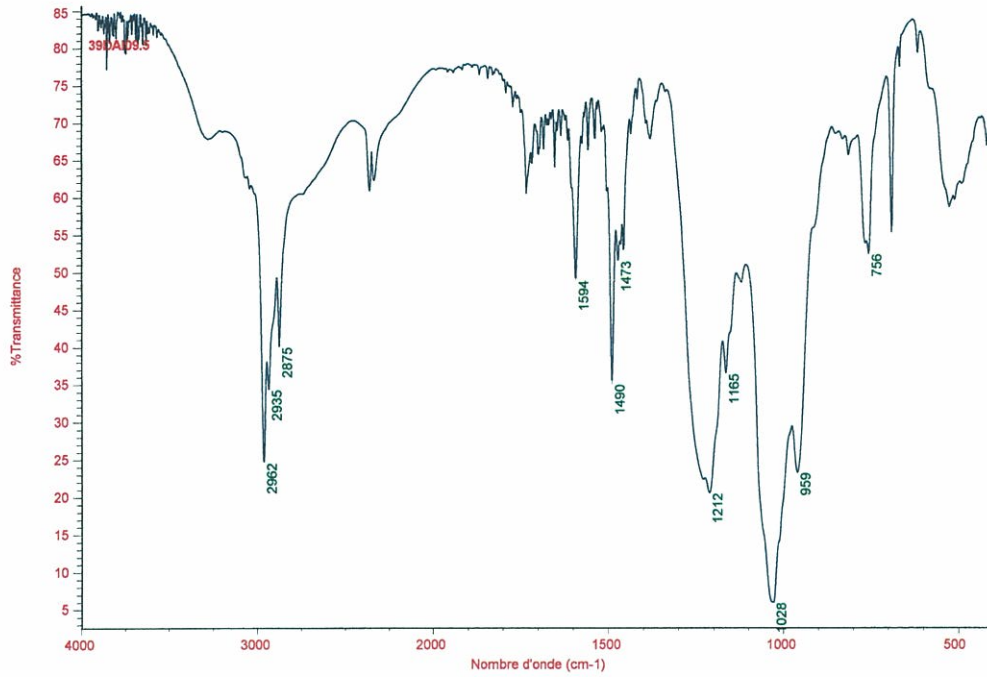
Spectrum 2 : black liquid 1b, sampled from "large room" (sample 2)



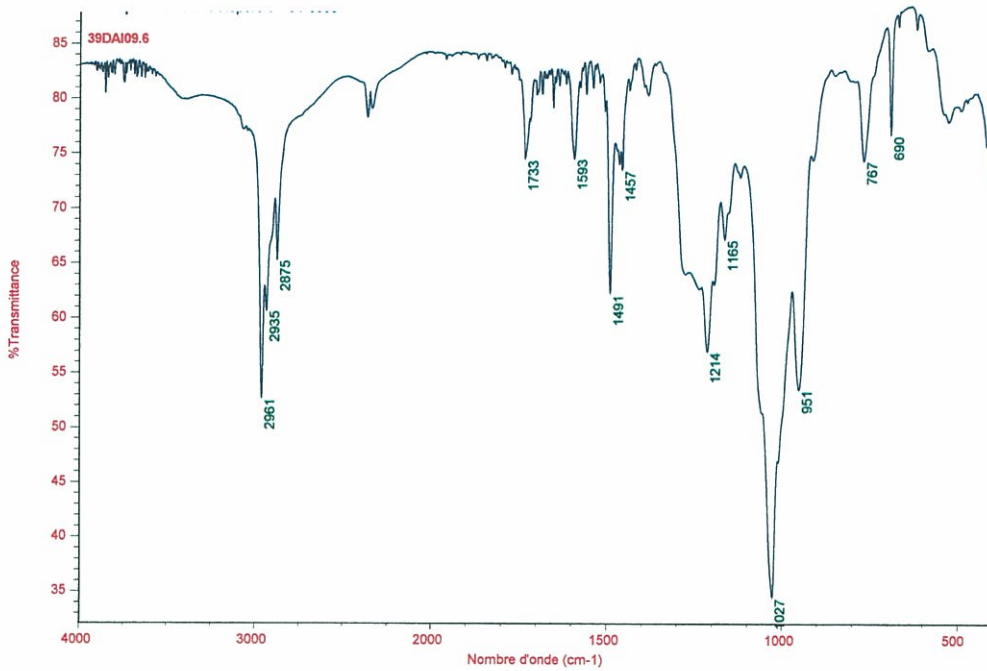
Spectrum 3 : clear mauve liquid 1c sampled from "large room" (sample 3)



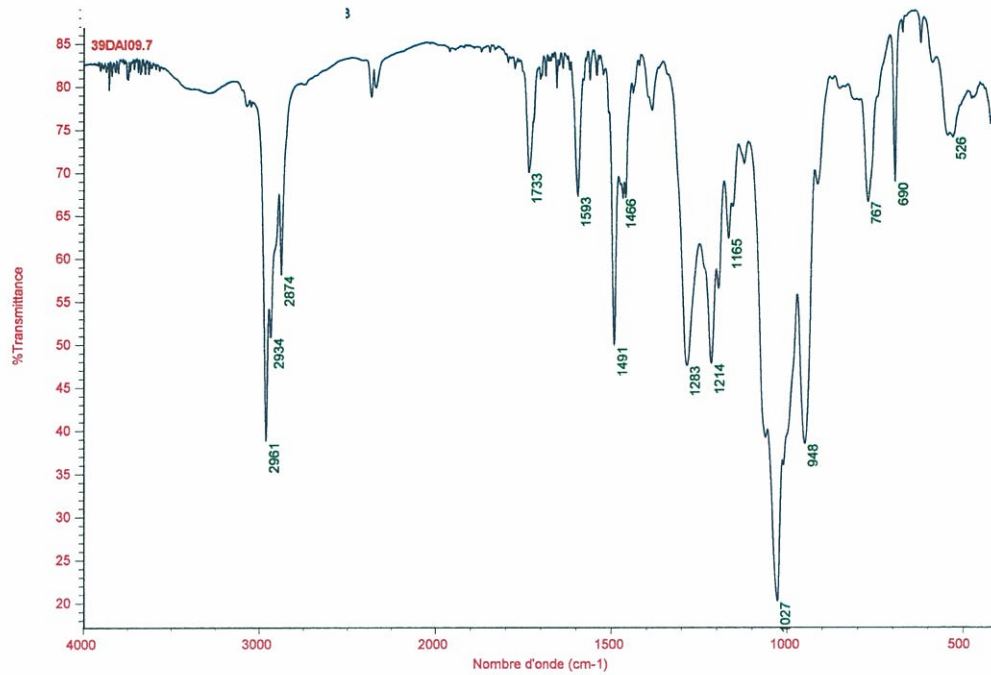
Spectrum 4 : thick black liquid 3 (sample 6)



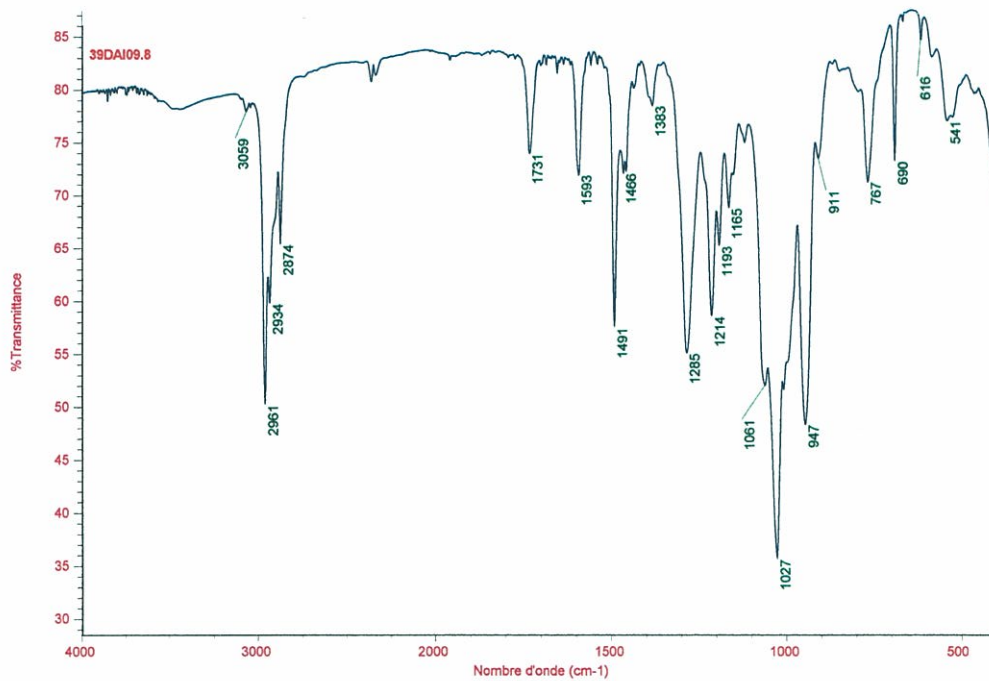
Spectrum 5 : black liquid 3 (sample 7)



Spectrum 6 : black thick liquid 4, sampled from "small room" (sample 8)



Spectrum 7 : black liquid 4, sampled from "large room", (sample 9)



Spectrum 8 : reference of skydrol 500 B-4 supplied from BEA