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Space Shuttle Cleaning Verification: Applications of the Contamination Analysis Unit to Reusable Solid Rocket Motor Production Scenarios

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Lawrence Livermore National Laboratory

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Work Performed by Lawrence Livermore National Laboratory
in accordance with
Thiokol Aerospace and Industrial Technologies Testplan PTP-0327

Principal Investigator: Michael Meltzer
Pollution Prevention Group

Coinvestigators: Carolyn Koester and Stephanie Ross
Chemistry and Material Sciences Directorate

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EXECUTIVE SUMMARY

The "Contamination Analysis Unit" (CAU) is an instrument that was built at Lawrence Livermore National Laboratory to measure organic contamination on surfaces. The CAU consists of a commercial Transpector mass spectrometer (Leybold Inficon, Inc.) interfaced to a thermal desorption interface, and employs a combination of heat and vacuum to volatilize the surface contaminants being analyzed. After volatilization, the contaminants are drawn into the source of the Transpector and detected. The CAU was employed in this study to analyze soils and cleaner residues on Space Shuttle Reusable Solid Rocket Motor parts.

Full scan mass spectral analysis was performed for the following soils:

Celvacene	Chemlok 205/233
HB polymer (propellant component)	HC-434 (from liner)
Hycar	EA-946 adhesive
HD-2 grease	Molykote
PR-1422	RTV DC 90-006
Teflon tape adhesive	vacuum putty
yellow tape adhesive	

The CAU produced usable mass spectral data for all of the above soils, with the exceptions of Celvacene, HC-434 polymer, Hycar, and Molykote. Although HB polymer was detected by the CAU, the poor detection limits for this soil suggest that the CAU is not the appropriate instrument for detection of propellant contamination.

Full scan mass spectral analysis was also performed for the following cleaners:

Bio-Act 113	Bio-Act 145
Bio-Act PCG	Ecosolve 5
Ionox BC	isopropanol (IPA)
Oxsol 100	PF 145 HP
PF Degreaser	PF d'Ink
PF Ionsol	Reveille
1,1,1 trichloroethane (TCA)	

The CAU produced usable mass spectral data for all of the above cleaners, with the exceptions of isopropanol and TCA. Because of these two chemicals' extremely high volatilities, they completely evaporated before sufficient vacuum was reached to turn on the mass spectrometer, and thus they could not be detected by the CAU.

The CAU was used to compare the cleaning effectiveness of TCA (an existing Thiokol solvent) *versus* alternative cleaners for various soil/substrate combinations. Ionox appeared to better remove EA-946 from a range of surfaces than TCA. The various alternate cleaner candidates showed comparable cleaning abilities to TCA when removing HD-2 grease (TCA *versus* Reveille), Teflon adhesive (TCA *versus* PF Degreaser), Chemlok 205/233 (TCA *versus* Ionox), and vacuum putty (TCA *versus* Reveille) from test surfaces. TCA showed superior cleaning ability to the alternate cleaners used to remove yellow tape adhesive (TCA *versus* PF Degreaser) and RTV 90-006 (TCA *versus* IPA) from test surfaces.

1.0 INTRODUCTION

This report describes work to date on a project to test the capabilities of a mass spectrometer-based system for analyzing *in-situ* organic compounds on a variety of substrates. The system is termed the "Contamination Analysis Unit" (CAU) and employs vacuum and thermal desorption of surface residues, followed by ionization and analysis with a Leybold Inficon Transpector mass spectrometer.

The agreement covering this project was made between the United States government, represented by the Department of Energy (DOE) and Thiokol Aerospace and Industrial Technologies (Thiokol) under contract number M89R020. Work on the project has been performed by Lawrence Livermore National Laboratory (LLNL).

2.0 PROJECT OBJECTIVES

This project examines the CAU's potential application to an assortment of Reusable Solid Rocket Motor (RSRM) production scenarios. The CAU has the capability to provide data relating to both the approximate quantities and species of volatile residues on production components.

Specific objectives of the project include:

1. Evaluate vacuum sealing capability of the CAU

2. Assess contamination detection effectiveness of the CAU on critical soil/substrate combinations

3. Compare TCA and replacement cleaner performance

3.0 TEST DATA REQUIREMENTS

To meet project objectives, the following data were generated:

Task 1. Maximum vacuum level achievable for test coupons with various surface roughnesses.

Task 2. Mass spectra of critical soils.

Task 3. Mass spectra of critical substrates.

Task 4. Mass spectra of TCA replacement candidates.

Task 5. Cleanability study of various soil/substrate combinations. Included CAU analyses and visual inspection of test coupons.

4.0 THE CAU -- DESIGN AND OPERATION

The CAU consists of a commercial Transpector mass spectrometer (Leybold Inficon, Inc.) interfaced, via a gate valve, to a thermal desorption interface (see Figures 1 and 2). A heater, operating in vacuum (~10^{-5} torr) and at temperatures ranging from ambient to 200°C, is used to volatilize the surface contaminants being analyzed. After volatilization, the contaminants are drawn into the source of the Transpector by vacuum, ionized, and mass analyzed.

Figure 1. The Contamination Analysis Unit Front End: Transpector Mass Spectrometer, Turbopump, Gate Valve and Thermal Desorption Interface

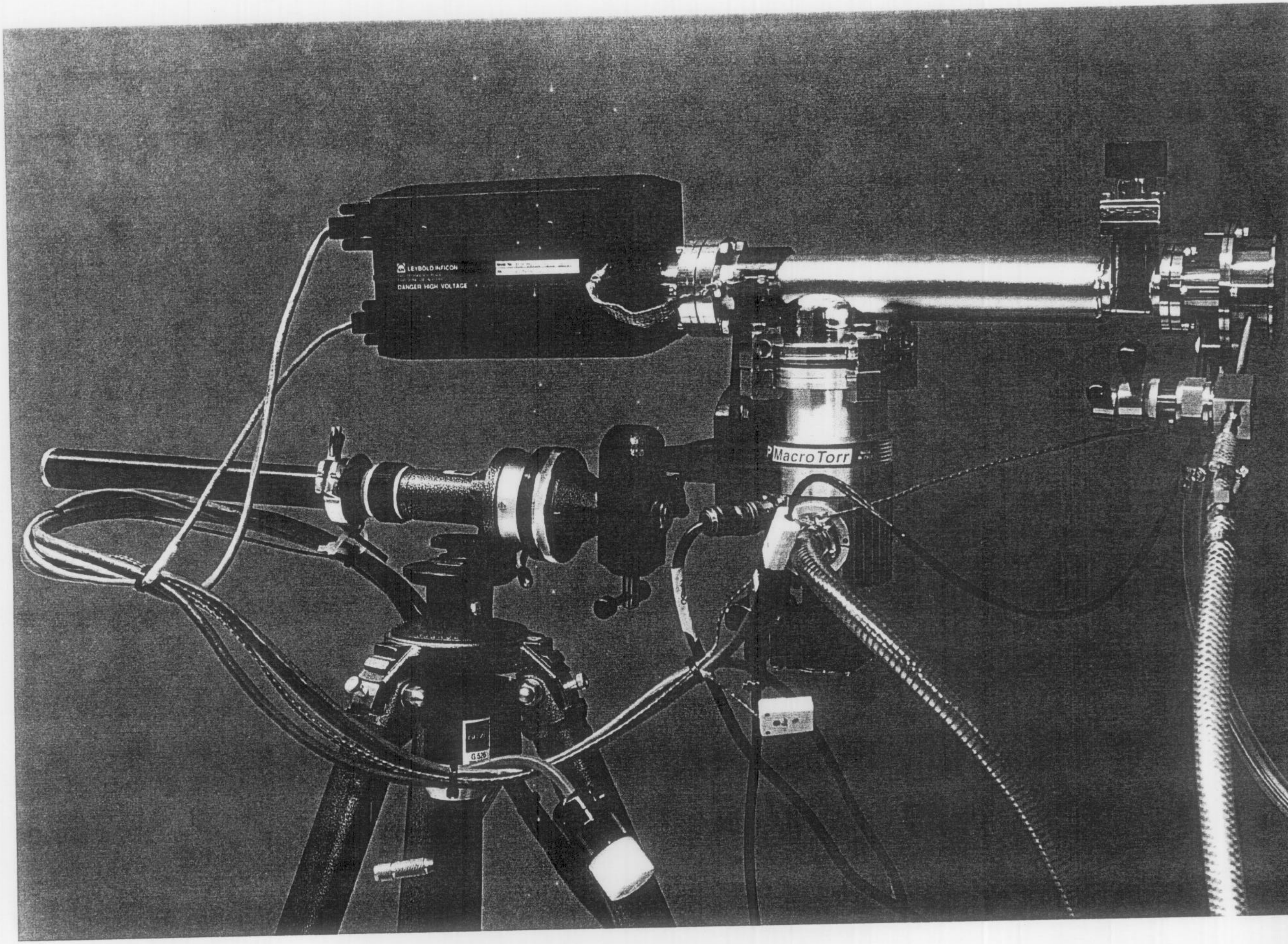
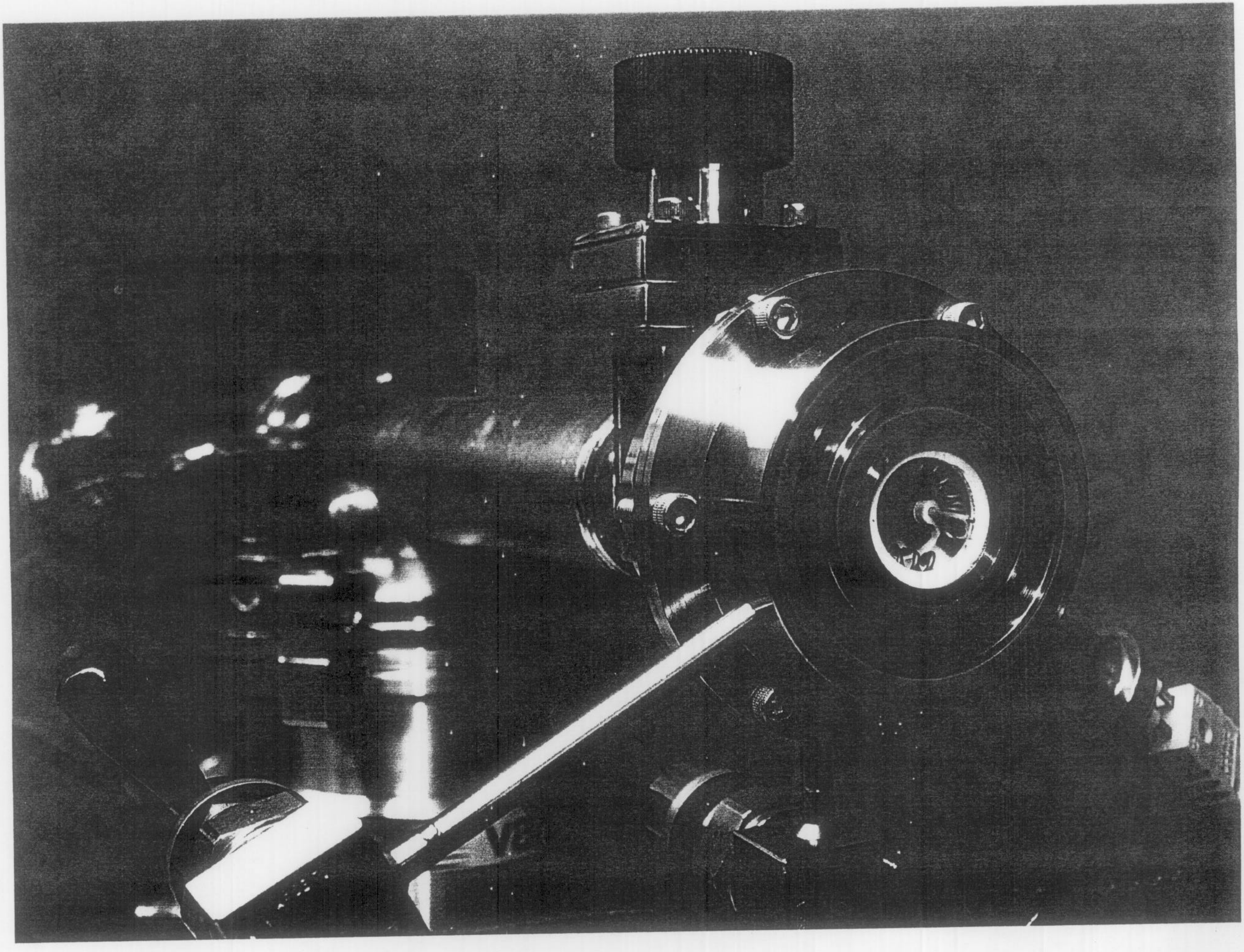

LEYBOLD INFICON
DANGER HIGH VOLTAGE
MacroTorr

Figure 2. CAU, Including Computer Display, Controls, and Roughing Pump

INTERFACE PRESSURE
8489
SAMPLE SURFACE TEMPERATURE
SAMPLE SURFACE HEATER POWER
RESET
HEAT TAPE CONTROLLER
POWER
varian
Turbo-V 70

CAU o-ring assemblies: The CAU can be operated with either a single o-ring or double o-ring sample inlet. The single o-ring inlet works well (i.e., it provides a vacuum of 10^{-5} torr) for the analysis of smooth surfaces. However, when the analysis of rough surfaces is attempted, the single o-ring sample the CAU will not go lower than 10^{-4} torr). To solve this problem, a concentric, double o-ring sample inlet was constructed (see Figure 3). The space between o-rings is evacuated with a roughing pump; this minimizes air leaks into the CAU. The sample analysis area, inside the smaller o-ring, is evacuated using a turbopump. As is evident from vacuum measurements shown in Table 2 of Section 6.0, the double o-ring nose allowed us to improve the vacuum to rough surfaces such that they could be analyzed.

Figure 3. Double O-Ring Configuration



4.1 Modes of Operation

The CAU can be operated in either "full scan" or "trending" mode. In full scan mode, ion current is measured for each integer mass between a predetermined low and high mass (*i.e.*, the entire mass spectrum of a contaminant is collected over a selected mass range). A typical scan range for this mode of operation, which generally captures the species of interest, is *m/z* (mass/charge) 30 to *m/z* 150 atomic mass units (amu). A complete mass spectrum is recorded every seven seconds as the sample inlet is heated from ambient to 200°C. The full scan mode provides information about the mass spectral fingerprint of a contaminant. It also provides information about which ions to use for trending mode analysis.

In trending mode, which is analogous to selected ion monitoring in gas chromatography/mass spectrometry (GC/MS), 10 to 20 pre-selected ions are monitored. As the sample inlet is heated from ambient to 200°C, signals from these selected ions are monitored for dwell times of approximately 30 milliseconds each and data are recorded every two seconds. Trending mode affords better detection limits than full scan mode. Trending mode is most useful in process applications in which known compounds are analyzed. The tradeoff for the improved detection limits offered by trending mode analysis is the loss of wide-range information provided by full scan analysis.

4.2 Strengths and Limitations of the CAU

Compounds amenable to analysis by CAU. The CAU can detect organic compounds of 2 to 200 atomic mass units. Inorganic compounds cannot be detected with the CAU. In order to be analyzed by the CAU, analytes must be sufficiently volatile to be removed by vacuum and thermal desorption from the surface being examined. In addition, the analyte (and the surface) must not be degraded by temperatures of 200° C. Surfaces that outgas significantly (for example, EPDM rubber) or that powder easily (for example, carbon cloth phenolic) cannot be analyzed by the CAU. These substrates will contaminate the CAU, decreasing its sensitivity and requiring it to be disassembled and cleaned. In addition, dust from materials, such as carbon cloth phenolic, will damage the CAU's turbopump. All analytes and surfaces must be evaluated individually in order to determine their applicability to CAU analysis.

Contaminant identification. A strength of the CAU is that it can be used to detect a wide variety of known organic contaminants on a range of metallic and non-metallic surfaces. A <u>mixture</u> of contaminants can, however, be difficult to deconvolute. In contrast to gas chromatography (GC) or gas chromatography/mass spectrometry (GC/MS), no compound separation occurs before analyte detection. With the CAU, all analytes that reach the Transpector are ionized and mass analyzed simultaneously. Thus, the mass spectrum of a cleaner or contaminant mixture is the sum of the ions of the individual compounds present. The CAU is best suited for process applications in which the contaminants are known or for applications for which it is not necessary to elucidate the exact composition of a contaminant mixture.

For example, GC/MS data indicate that there are three distinct organic compounds present in the cleaner Ionox--a furan, a dioxalane, and an alcohol. Each of these compounds has a unique mass spectrum--the furan's mass spectrum shows predominant ions of *m/z* 43, and 71; the dioxalane's mass spectrum shows ions of *m/z* 43, 73, and 101; and the alcohol's mass spectrum shows ions of *m/z* 43, 59, and 101. However, when Ionox is analyzed by the CAU, the resulting mass spectrum is a composite of all ions present and contains ions at *m/z* 43, 59, 71, 73, and 101. We have experimented with adapting commercial deconvolution software to the CAU. While results were encouraging, the CAU is not able at this time to identify individual components of the above mixture.

5.0 CAU DETECTION LIMITS AND PRECISION

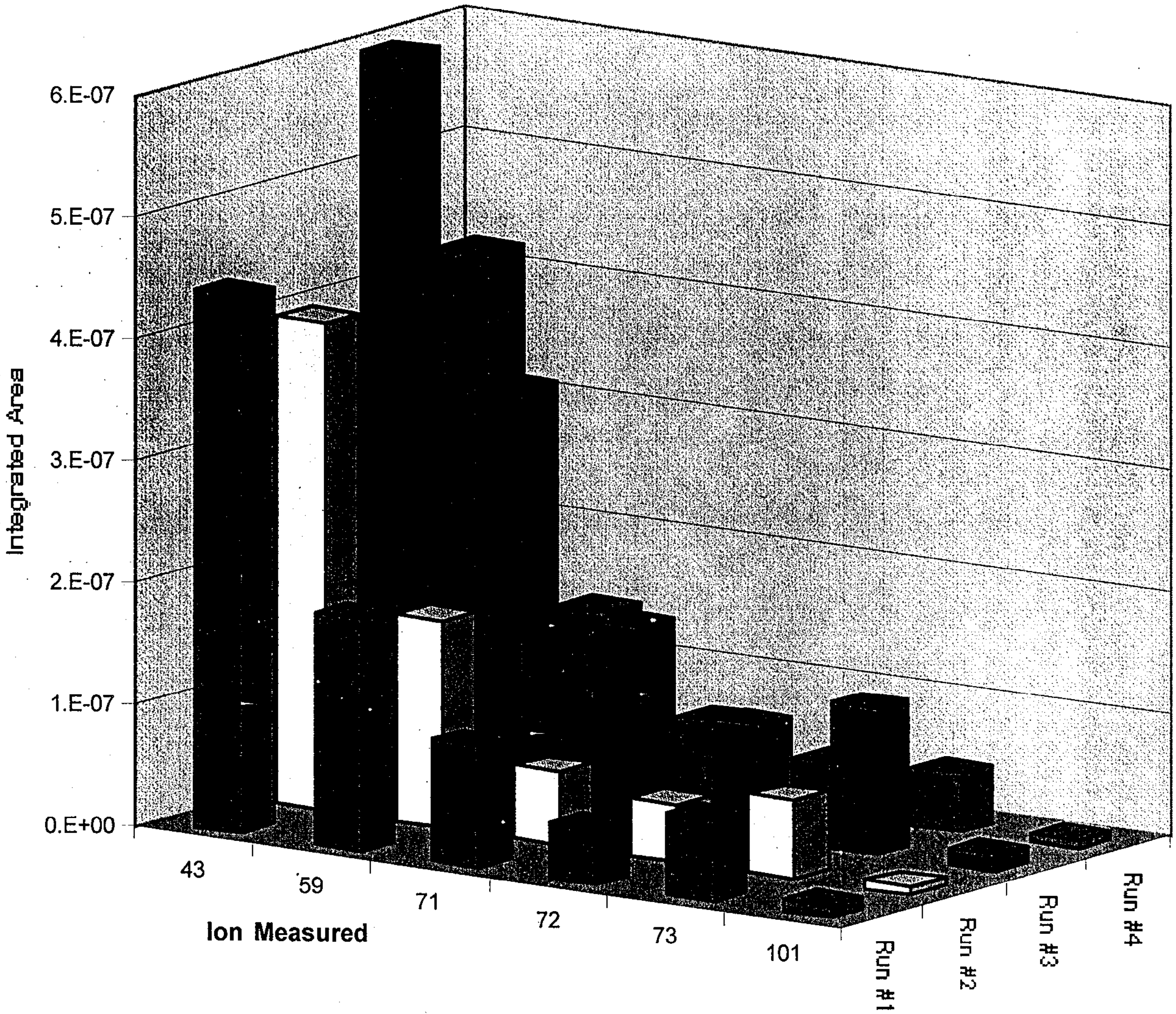
5.1 Determining CAU Detection Limits

The detection limit for a particular analyte is defined as the minimum concentration of that analyte which will produce a signal that is a factor of ten greater than that of the background. For a contaminant to be positively detected by the CAU, a minimum of two of its ions must be detected. The CAU yields its best detection limits in trending mode. Thus, all detection limit studies are performed in this mode of operation.

5.2 Example of Detection Limit Determination

Detection limits for the aqueous cleaner Ionox were conducted with both the single o-ring and double o-ring configurations, using the portable CAU. For the single o-ring nose, the detection limit for Ionox on a smooth, stainless steel surface was approximately 0.5 $\mu g/cm^2$ (± 90%). The detection limit for Ionox on a slightly roughened (60 microinch) stainless steel surface, using the double o-ring nose ranged from 3 to 5 $\mu gm/cm^2$ (± 90%). It is unclear at present why double o-ring detection limits are not as good as those observed with the single o-ring sample inlet. The difference might be caused by the design of the double o-ring itself or by the rougher surface material onto which the Ionox was applied, possibly leading to contaminants bleeding out of the analysis area under the o-ring.

Figure 4. Portable CAU Within-Day Precision 0.5 $\mu g/cm^2$ Ionox 10/8/98



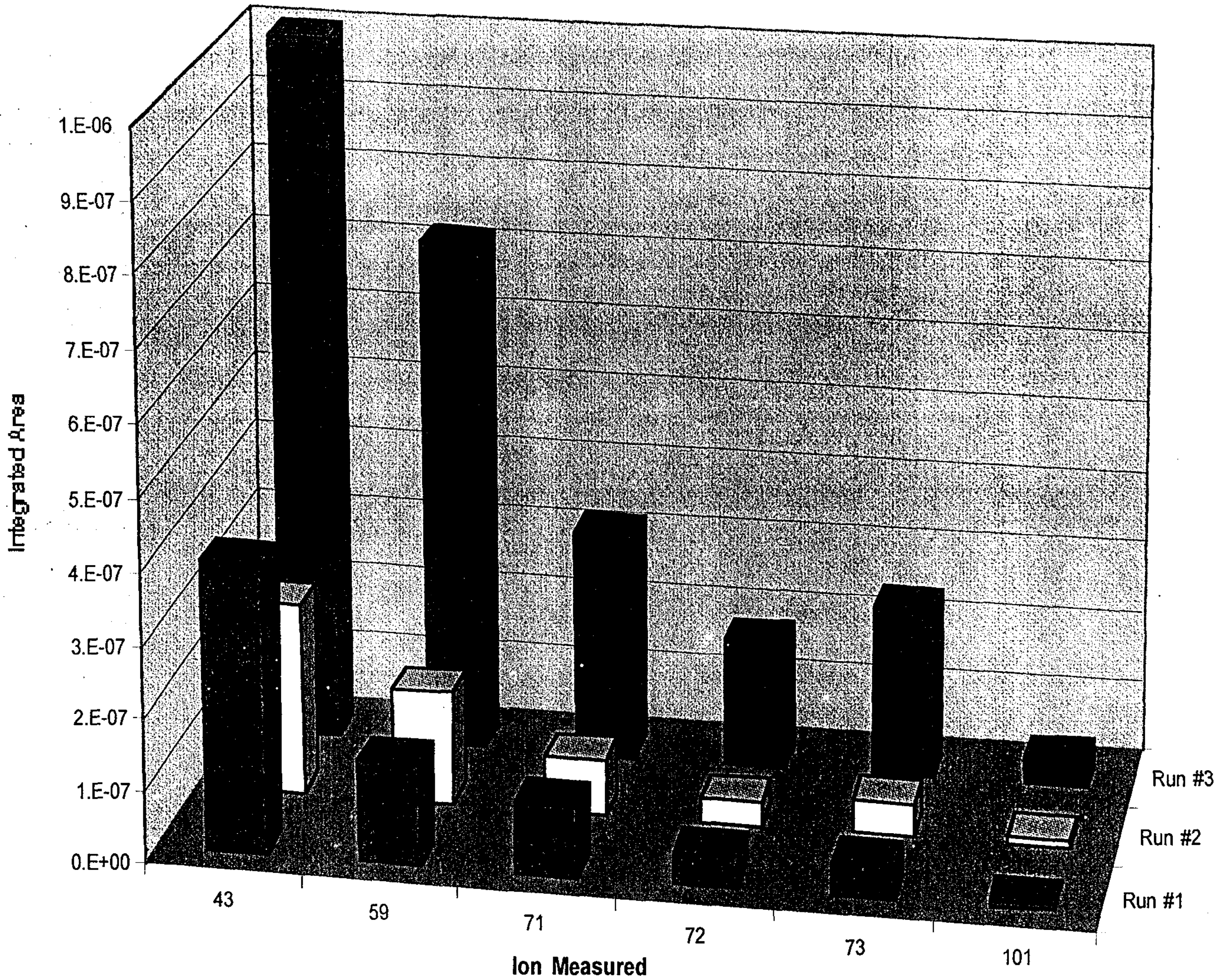
These data are intended to provide an estimate of the detection capabilities of the portable CAU. Instrument detection limits are a function of several factors that may include the roughness and composition of the surface from which the contaminant is desorbed, the "background" ion signals from the instrument, and the characteristics of the analytes (such as their volatilities and surface interactions). The area of the surface sampled also affects detection limits.

5.3 Precision (Studied with Single O-Ring Sample Inlet)

Experiments were performed to study within-day and day-to-day changes in instrument response. To perform these analyses, replicate plates were spotted with the equivalent of 0.5 $\mu g/cm^2$ and 2.6 $\mu g/cm^2$ of Ionox.

Within-day precision. Figures 4 and 5 show the integrated areas measured for various ion signals produced by replicate analyses. In general, if the response

Figure 5. Portable CAU Within-Day Precision 0.5 μg/cm² Ionox 10/15/98



of one ion was low relative to that of a previous analysis, the responses of all ions monitored during that analysis were also proportionately low.

The results of the within-day precision studies are summarized in Table 1. Depending on the ion monitored, the relative standard deviations (RSDs) for measurements of 0.5 μg/cm² Ionox made with the portable CAU ranged from 42 to 101%. This precision is comparable to that of the non-portable CAU (prototype instrument). Using a liberal definition, a quantitative analytical measurement should have a relative standard deviation less than 15-20%. Good quantitative techniques, such as GC/MS, have precisions of less than 2% relative standard deviation. In light of the observed precision of the instrument, the CAU should be considered a semi-quantitative instrument.

Table 1. Summary of Ionox Same-day Precision Data

	Relative Standard Deviations (RSDs) for Replicate Measurements of Ionox		
m/z	**Non-Portable CAU 6 μg/cm² n=5**	**Portable CAU 0.5 μg/cm² n=4 10/9/98**	**Portable CAU 0.5 μg/cm² n=3 10/15/98**
43	85	ND	70
59	73	42	97
71	69	49	81
72	80	ND	96
73	71	43	101
101	73	38	93
Average of all ions	**75**	**43**	**90**

Note: RSD (relative standard deviation) is the standard deviation of each measurement divided by the average of the measurement, expressed as a percent. "ND" indicates that the ion was not detected because the signal it produced was not significantly greater than background.

Day-to-day variations in instrument response. Day-to-day variations in the response of the portable CAU were studied by measuring the signals produced from coatings of 0.5 μg/cm² Ionox and 2.6 μg/cm² Ionox on stainless steel plates. As shown in Figures 5 and 6, instrument responses, as measured by integrated peak areas produced by specific ions during a trending mode analysis, varied by factors of 1 (essentially equivalent responses) to 8. The data for the analyses of 0.5 μg/cm² Ionox indicate that the CAU was most sensitive (*i.e.* showed the greatest response) on 10/8/98 and showed approximately 5-fold less sensitivities on 10/9/98 and 10/20/98 (see Figure 6). The data for the analyses of 2.6 μg/cm² Ionox also showed that the CAU was most sensitive on 10/8/98 (see Figure 7). The response of the CAU on 10/20/98 was almost equivalent to its response measured on 10/8/98. The CAU's response on 10/9/98 was approximately a factor of 5 less than that its response on either 10/8/98 or 10/20/98. While instrument response often changes from day to day, note that the amplitudes of the ion signals, *relative to each other on a given day,* are significantly more consistent.

Within-day variability in CAU response is sufficiently high (as mentioned above, relative standard deviations of signals for replicate measurements range from 42 to101%) that there are large uncertainties in our measurement of day-to-day CAU variations. These results suggest the need for frequent analysis of a coupon containing a known amount of a standard contaminant, in order to assess instrument response and performance on a given day. Several possible causes for the lack of consistent instrument response include the instability of the Transpector (not likely), the inconsistency of the thermal desorption process, and loss of analytes to the vacuum system.

Figure 6. Daily Variation in Instrument Response Portable CAU 0.5 ug/cm2 Ionox

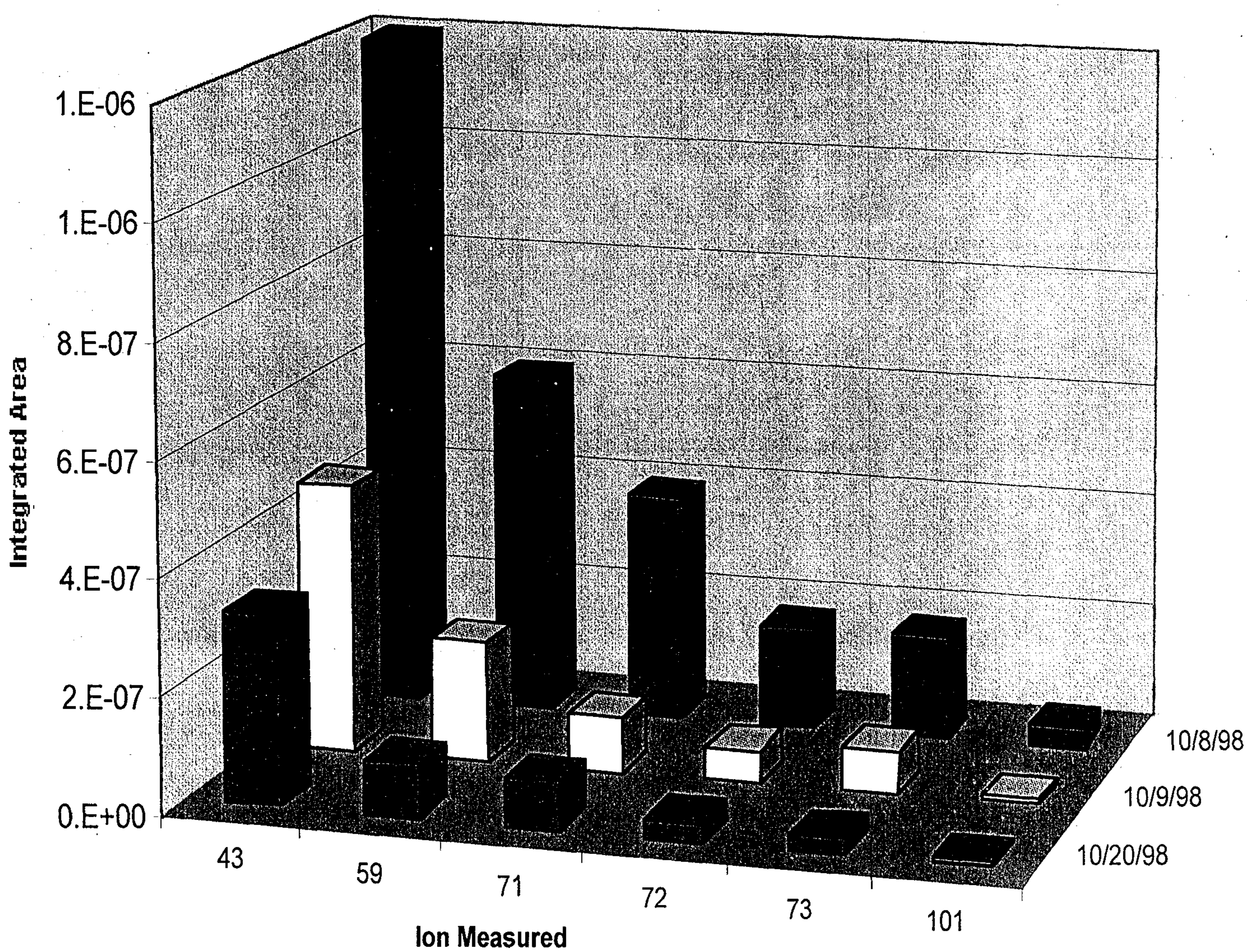
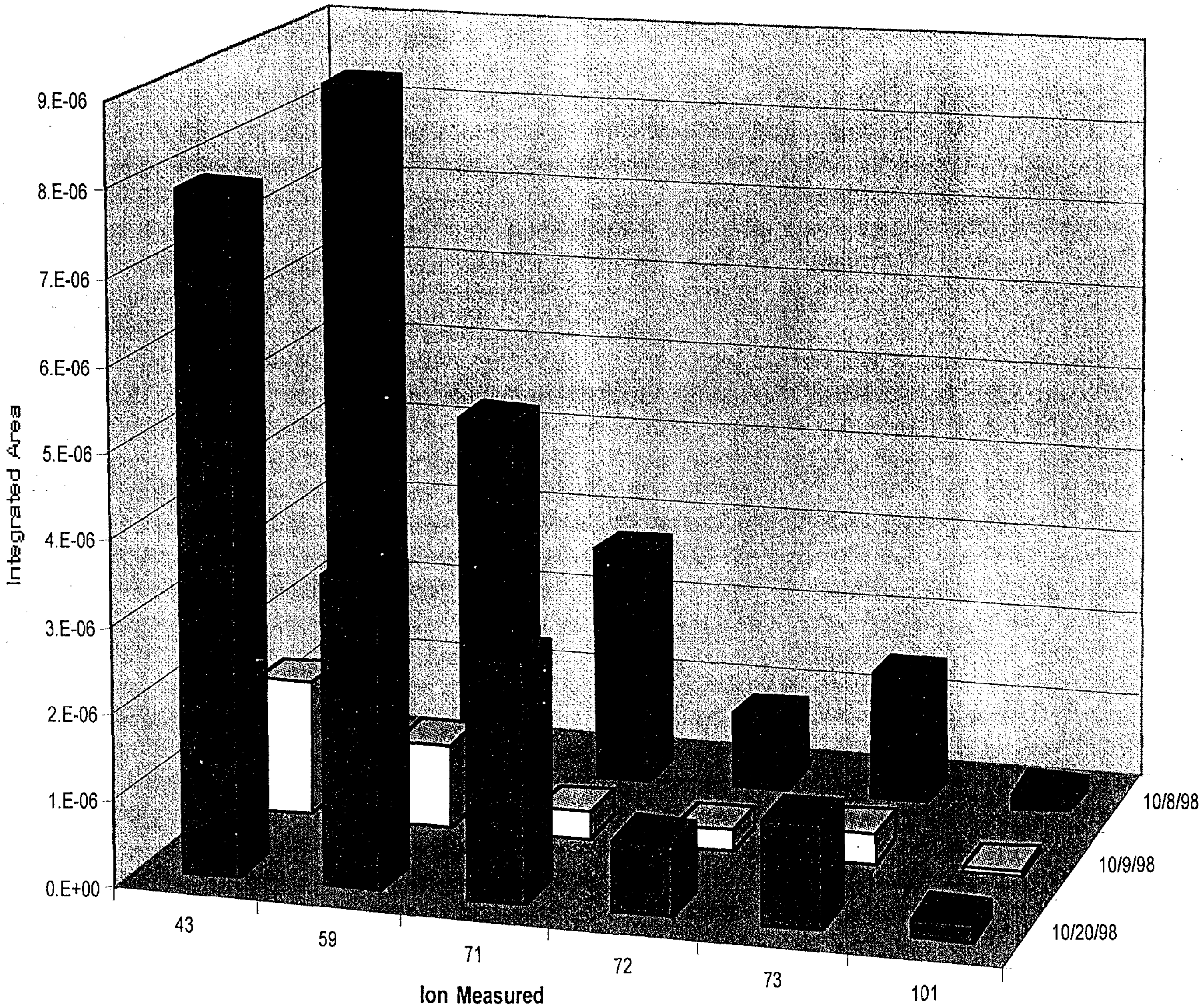


Figure 7. Daily Variation in Instrument Response Portable CAU 2.6 $\mu g/cm^2$ Ionox



6.0 DISCUSSION OF RESULTS

6.1 Task 1: Maximum Vacuum Level Achievable

All mass spectrometric methods require low vacuum. Obtaining a sufficient vacuum on rough surfaces was a concern in this project. Good vacuum seals around the substrate analysis area are necessary for the CAU's Transpector mass spectrometer to operate without damage. Vacuum seals are provided by either single o-ring or concentric double o-ring configurations. Both Viton and silicon o-rings are employed, although silicone o-rings were used in this study as they were determined to provide the best seal.

Measurements of maximum vacuum attainable were made for various surfaces with both the single and double o-ring sample inlets, and are listed in Table 2. Vacuums greater than 1 E-4 indicate that the pressure was too high to allow the Transpector's filament to be lit and to obtain an accurate vacuum measurement. It can be seen from Table 2 that a single o-ring vacuum seal is adequate for smooth, rigid surfaces such as D6 Steel, painted steel, or glass bead-finished stainless steel, but a double o-ring configuration is required on rougher surfaces such as grit-blasted stainless steel, glass-cloth phenolic and NBR rubber.

For all of the surfaces analyzed, the double o-ring sample inlet provided the low vacuum necessary for CAU analyses, if vacuum was not sufficient with the single o-ring assembly.

Table 2. Vacuum Measured on Various Surfaces

Surface	Roughness (microinches)	Single O-Ring Vacuum (torr)	Double O-Ring Vacuum (torr)
D6 steel[a]		8.6E-5	
Aluminum[a]		6.0E-5	
Teflon coated aluminum[a]		5.0E-5	
Painted steel[a]		2.5E-5	
Painted aluminum[a]		1.7E-4	
D6 steel, glass bead[b]	40	7.6 E-5	1.2 E-5
D6 steel, light grit[b]	60	>1 E-4	1.2 E-5
D6 steel, heavy grit[b]	110	>1 E-4	1.6 E-5
Glass cloth phenolic[b]	-----	>1 E-4	5.3 E-5
NBR insulation[b]	-----	>1 E-4	5.1 E-5

a – Values obtained with nonportable CAU
b – Values obtained with portable CAU

6.2 Critical Soil Mass Spectra

Full scan mass spectra were collected with the nonportable CAU (prototype instrument). Hycar, Celvacene, Molykote, and HC-434 (liner component) yielded no mass spectral data and, as a result, cleanability studies were not performed. The following soils yielded useful mass spectral data: EA-946, HD-2 grease, Teflon adhesive, RTV 90-006, Chemlok 205/233, yellow tape adhesive, vacuum putty, PR-1422, and HB Polymer (propellant component). See Appendix A for mass spectra.

HB polymer (present at about 10% in propellant) produced a detectable mass spectrum. However, trending mode analysis failed to detect 10 $\mu g/cm^2$ of HB polymer on a surface. Because this amount of HB polymer would correspond to 100 $\mu g/cm^2$ of propellant contamination (a large amount), we do not believe that the CAU would be the appropriate instrument to use to measure propellant contamination.

6.3 Substrate Background Spectra

None of the substrates tested (D6 steel, aluminum, Teflon-coated aluminum, painted steel, painted aluminum, NBR, and glass-cloth phenolic (GCP)) yielded mass spectral signals above the instrument's background. These spectra are included in Appendix B. In many of these spectra, ions of *m/z* 32, 40, and 44 are predominant. These peaks arise from the air background in the CAU, and represent, oxygen, argon, and carbon dioxide, respectively. In addition, ions of *m/z* 55 and 57 are also visible. These arise from hydrocarbon contamination in the CAU, most probably from the roughing pump. These hydrocarbons are especially noticeable in the background spectra for NBR and the glass cloth phenolic – these were collected using the portable CAU with the double o-ring assembly.

3.1 Solvent/Cleaner Spectra

Full scan mass spectra were collected with the nonportable CAU (prototype instrument). Trichloroethane and isopropyl alcohol yielded no mass spectral data (they volatized as the CAU vacuum was being pumped down, leaving no residue). The following cleaners yielded useful mass spectral data: Ionox, Reveille, PF Degreaser, PF 145 HP, Bio-Act 113, PF Ionsol, PF d'Ink, Bio-Act PCG, OxSol 100, Bio-Act 145, and Exosolve 5. See Appendix C for their mass spectra.

6.5 Performance Comparison of TCA with Replacement Cleaner Candidates

Once the mass spectral data of the soils, cleaners and substrates were collected, the cleaning performances of different cleaning chemicals were compared. Substrates were contaminated with various critical soils, then cleaned using a wipe of either 1,1,1 trichloroethane (TCA) or its proposed replacement. The CAU, operated in trending mode, was used to determine if measurable contamination from the soil or cleaner remained on each coupon after cleaning. Table 3 summarizes the results of these comparisons. Appendix D lists the ions monitored for each soil/cleaner.

Table 3 - Cleanability Summary

	EA-946		HD-2		Teflon Adhesive		Yellow Adhesive		Chemlok 205/233	
	TCA	Ionox	TCA	Reveille	TCA	PF Degr.	TCA	PF Degr.	TCA	Ionox
Steel	No	No	No	No	No	No	No	No [2]	Yes	No
Aluminum	Yes	No	No	No; Cleaner	No	No	No	No [2]	No	No
Teflon Al			No	No			No	No		
Painted Al	No	No	No	No	No [2]	No [2]	No [2]	Yes [2]		
Painted Steel	No	No	No	No	No [2]	No [2]	Yes [2]	No [2]	Yes	Yes & Cleaner
NBR (dblo-ring)	Yes [1,2]	No [1]	Yes [1]	Yes [1]	No [1]	No [1]	No [1]	No; Cleaner [1,2]	Yes [1]	Yes [1]
GCP (dblo-ring)	Yes [2]	No [2]	Yes [1,2]	Yes [1,2]	No [1,2]	No [1,2]	No [1]	No [2,3]	No [1]	No [1]

	RTV 90-006		Vacuum Putty	
	TCA	IPA	TCA	Reveille
Steel	No	No [2]	No	No
Aluminum	No	No [2]	No	No
Teflon Al	No	No		
Painted Al	No	No	No [2]	No [2]
Painted Steel	No	No	No [2]	No [2]
NBR (dblo-ring)	No [1]	No [1,2]	Yes [1,2]	Yes & Cleaner [1,2]
GCP (dblo-ring)	No [1,2]	No [2]	No [2]	Yes & Cleaner [2]

Criteria for detection: (1) Signal must be approximately one order of magnitude above that of the blank (2) at least two specific contaminant ions must be above baseline
Yes = contamination detected; No = no contamination detected; Cleaner = cleaner detected
1 = high pressure or pressure hold; run not completed to 200 C
2 = contaminant still visually observable on substrate
3 = does not meet criteria for detection; however, displays unique cleaner/contaminant desorption profiles

Discussion of Table 3

For ease of comprehension, we will explain the data summarized in Table 3 in order of contaminant.

EA-946. After cleaning with Ionox, no residues were detected on any of the substrates. For TCA cleaning, however, EA-946 residues were detected on aluminum, NBR rubber and GCP. This suggests that Ionox removes EA-946 from substrates more effectively than TCA. Residue was visually observed on NBR and GCP after cleaning with TCA, but only on GCP after cleaning with Ionox. Obviously, the CAU could not detect the visible, non-volatile residues remaining on GCP.

HD-2. TCA and Reveille appear to be comparable cleaners. For both cleaners, the CAU detected HD-2 residues on NBR and GCP. Visual residues were observed on GCP. On Teflon-coated aluminum, Reveille, but not HD-2, was detected by the CAU.

Teflon adhesive. No Teflon adhesive residues were detected by the CAU after cleaning with either TCA or PF Degreaser. Residue was visually observed on three of the substrates (painted aluminum, painted steel, and GCP) when cleaned either with TCA or PF Degreaser.

Yellow adhesive. The CAU detected adhesive residue on painted steel cleaned with TCA, and on painted aluminum cleaned with PF degreaser. PF Degreaser residue was detected on the NBR coupon. CAU performance data for the two cleaners appear comparable. Adhesive residues, however, were visually observed on *six* of the substrates cleaned with PF degreaser, but on only two substrates cleaned with TCA, indicating that TCA performed better than PF Degreaser in this test.

Chemlok 205/233. The CAU detected soil residue on three of the TCA-cleaned substrates (steel, painted steel, and NBR), but on only two of the Ionox-cleaned coupons (painted steel and NBR). On NBR, the CAU also detected Ionox residue. No visible residue was detected on any of the coupons.

RTV 90-006. No soils were detected by the CAU on substrates cleaned either with TCA, or with isopropyl alcohol (IPA). Visual residues for coupons cleaned with TCA were only observed on the GCP coupon. For IPA-cleaned coupons, visual residue was observed on steel, aluminum, NBR, and GCP, indicating that TCA performed better than IPA in this test.

Vacuum putty. The CAU detected putty residue on NBR cleaned with TCA, and on NBR and GCP that was cleaned with Reveille. Residues were visually observed on the painted aluminum, painted steel, NBR, and GCP substrates cleaned with TCA, as well as on the <u>same</u> substrates cleaned with Reveille.

7.0 REVIEW OF RESULTS

The CAU provided mass spectral data for soils of Chemlok 205/233, HB polymer, EA-946 adhesive, HD-2 grease, PR-1422, RTV DC 90-006, Teflon tape adhesive, vacuum putty, and yellow tape adhesive. The CAU also provided mass spectral data for the cleaners Bio-Act 113, Bio-Act 145, Bio-Act PCG, Ecosolve 5, Ionox BC, Oxsol 100, PF 145 HP, PF degreaser, PF d'Ink, PF Ionsol, and Reveille.

The CAU was used to determine that Ionox removed EA-946 from various surfaces better than TCA. The appropriate alternate cleaner showed comparable cleaning abilities to TCA when removing HD-2 (TCA *versus* Reveille), Teflon tape adhesive (TCA *versus* PF Degreaser), Chemlok 205/233 (TCA *versus* Ionox), and vacuum putty (TCA *versus* Reveille) from test surfaces. TCA showed superior cleaning ability to the alternate cleaners used to remove yellow tape adhesive (TCA *versus* PF Degreaser) and RTV 90-006 (TCA *versus* IPA) from test surfaces.

8.0 FUTURE WORK

Extensions to Thiokol Project

In certain cleaning applications, it was possible to determine if TCA or the alternate cleaner best removed the contaminant of interest from a surface. The next question to address would be "at what *approximate* concentrations did the soils remain on the surfaces?" It will also be important to determine at what detection limits soils can be measured by the CAU (*i.e.*, does the CAU have good enough detection limits to be useful to Thiokol in "real-life" production scenarios?). Given the *semi-quantitative* nature of the CAU, is the CAU useful to Thiokol without further modification (see below)?

Instrument Modifications

Currently, the major area for improvement of the CAU is in reproducibility of measurements. It is possible that precision might be improved by developing a more efficient thermal desorption interface. Also, incorporating additional heaters into the nose of the CAU might help to keep the ceramics cleaner and minimize the instrument's background signals, which would improve the signal-to-noise ratio. A smaller orifice connecting the CAU's sample inlet with the analyzer manifold might also improve detection limits.

APPENDIX A. MASS SPECTRA OF SOILS

Analysis2
C:\Labview5\CAU\DESORB2\Analys2.llb\Analysis2
Last modified on 8/5/98 at 8:09 AM
Printed on 11/9/98 at 9:26 AM

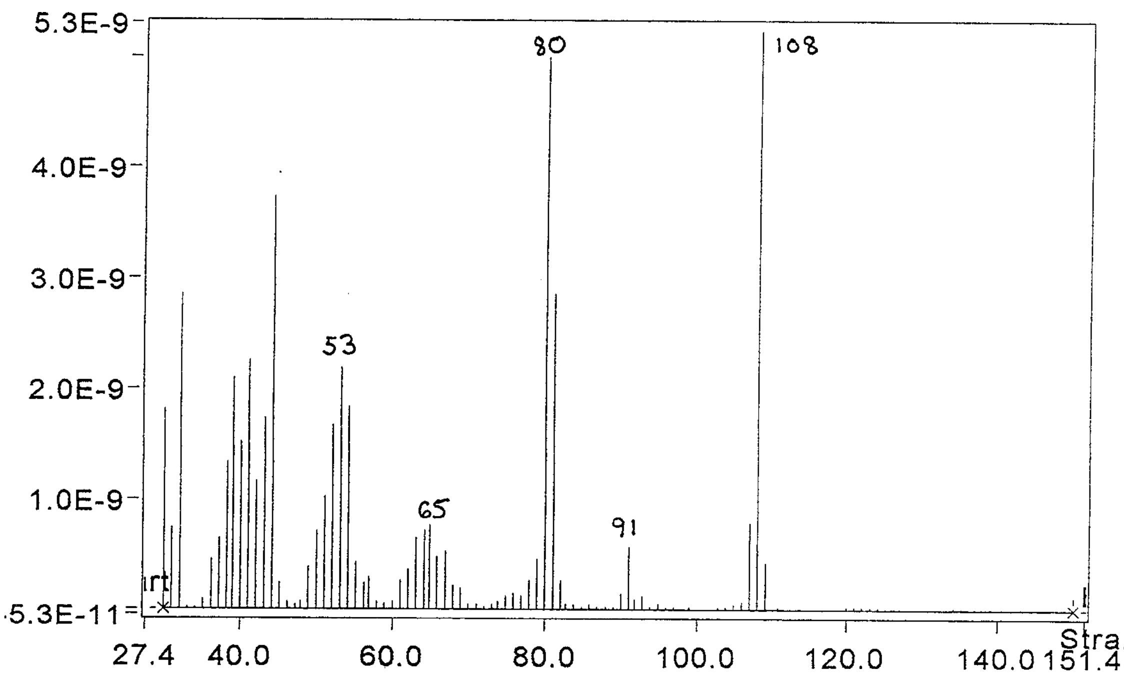
Sample ID: EA-946 neat

File path: C:\CAUDATA\THIOKOL\Table II\

Spectrum

EXIT

Y Mapping Linear



Records: 21

Rec No.

21

Sample Temp 215.38

Strart AMU 30

End AMU 150

Start	30.00	0.00E+0
End	150.00	0.00E+0

Baseline correction Above baseline

Baseline Pts 2

Analysis2
C:\Labview5\CAU\DESORB2\Analys2.llb\Analysis2
Last modified on 8/5/98 at 8:09 AM
Printed on 11/9/98 at 9:24 AM

Sample ID: HD-2 Grease - cured

File path: C:\CAUDATA\THIOKOL\Table II\

Spectrum

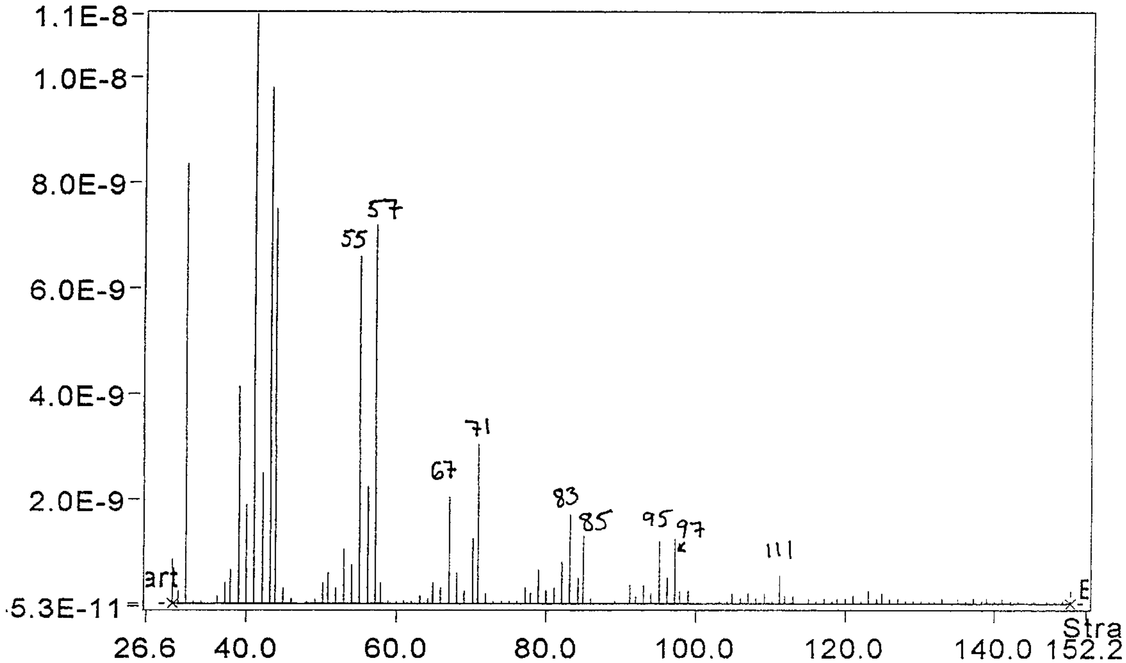
EXIT

Y Mapping Linear

Records: 22

Rec No. 22

Sample Temp 211.50



Strart AMU 30 End AMU 150

Start	30.00	0.00E+0
End	150.00	0.00E+0

Baseline correction Above baseline

Baseline Pts 2

Analysis2
C:\Labview5\CAU\DESORB2\Analys2.llb\Analysis2
Last modified on 8/5/98 at 8:09 AM
Printed on 11/9/98 at 9:28 AM

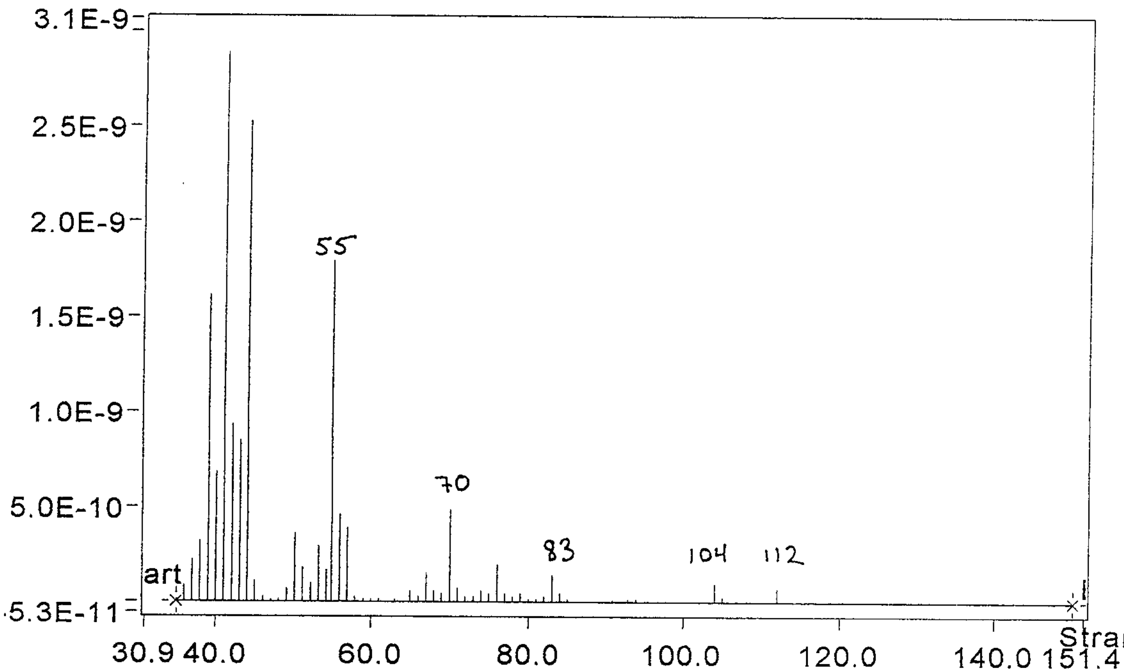
Sample ID: Teflon Tape Adhesive

File path: C:\CAUDATA\THIOKOL\Table II\52898f

Spectrum

EXIT

Y Mapping Linear



Records: 22

Rec No.

22

Sample Temp 210.08

Strart AMU 35   End AMU 150

Start	35.00	0.00E+0
End	150.00	0.00E+0

Baseline correction Above baseline

Baseline Pts 2

Analysis2
C:\Labview5\CAU\DESORB2\Analys2.llb\Analysis2
Last modified on 8/5/98 at 8:09 AM
Printed on 11/9/98 at 9:34 AM

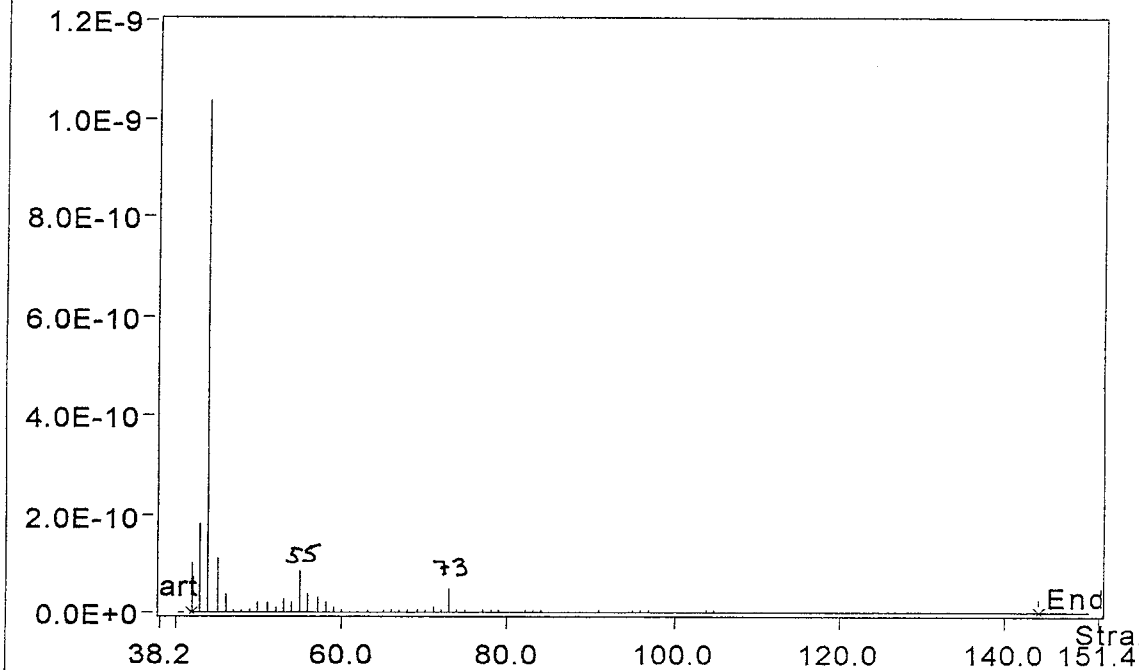
Sample ID: RTV 90-006 full scan

File path: C:\CAUDATA\THIOKOL\Table II\

Spectrum

EXIT

Y Mapping Linear



Records: 23

Rec No.

23

Sample Temp 214.43

Strart AMU 42 End AMU 150

Start	42.00	0.00E+0
End	144.00	0.00E+0

Baseline correction Above baseline

Baseline Pts 2

Analysis2
C:\Labview5\CAU\DESORB2\Analys2.llb\Analysis2
Last modified on 8/5/98 at 8:09 AM
Printed on 11/9/98 at 9:36 AM

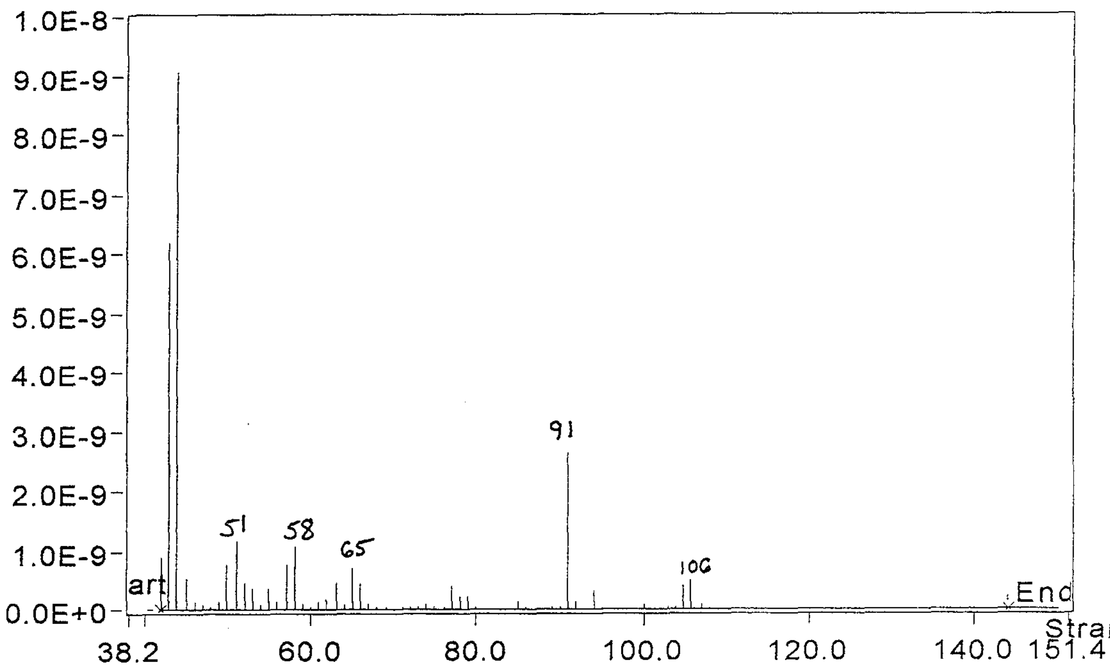
Sample ID: chemlok 205/233; full scan

File path: C:\CAUDATA\THIOKOL\Table II\

Spectrum

EXIT

Y Mapping Linear



Records: 19

Rec No.

12

Sample Temp 134.37

Strart AMU 42

End AMU 150

Start	42.00	0.00E+0
End	144.00	0.00E+0

Baseline correction Above baseline

Baseline Pts 2

Analysis2
C:\Labview5\CAU\DESORB2\Analys2.llb\Analysis2
Last modified on 8/5/98 at 8:09 AM
Printed on 11/9/98 at 9:43 AM

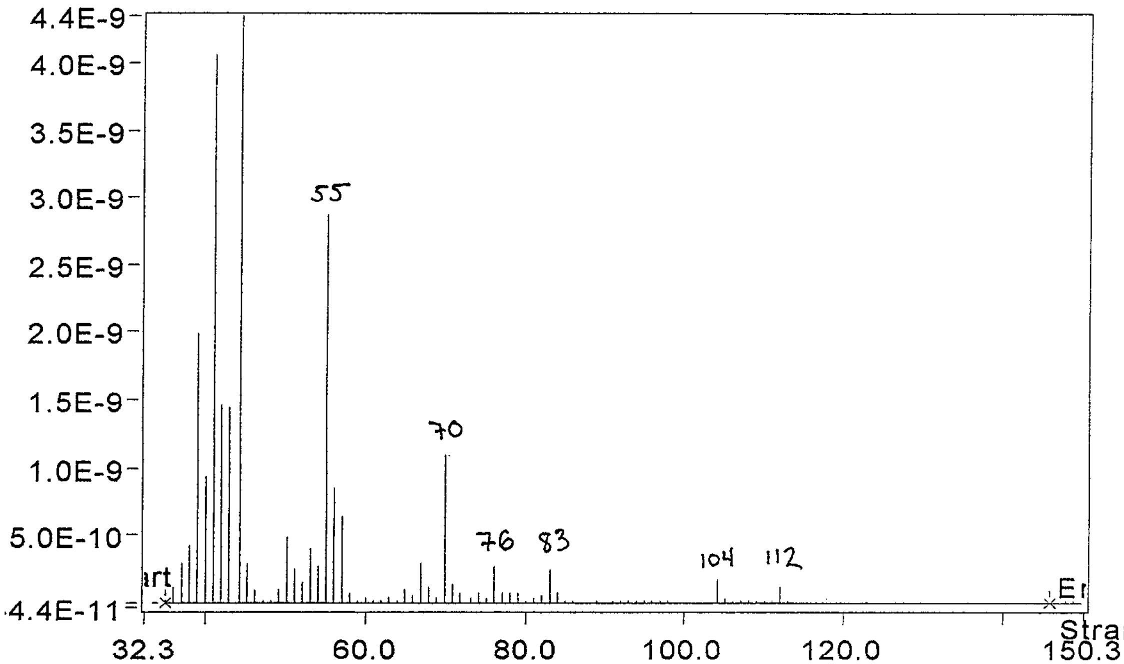
Sample ID: Permacel Yellow Tape Adhesive

File path: C:\CAUDATA\THIOKOL\Table II\

Spectrum

EXIT

Y Mapping Linear



Records: 22

Rec No.

22

Sample Temp 210.57

Strart AMU 35

End AMU 150

Start	35.00	0.00E+0
End	146.00	0.00E+0

Baseline correction Above baseline

Baseline Pts 2

Analysis2
C:\Labview5\CAU\DESORB2\Analys2.llb\Analysis2
Last modified on 8/5/98 at 8:09 AM
Printed on 11/9/98 at 9:45 AM

Sample ID: LLNS SS vacuum putty; full scan

File path: C:\CAUDATA\THIOKOL\Table II\

Spectrum

EXIT

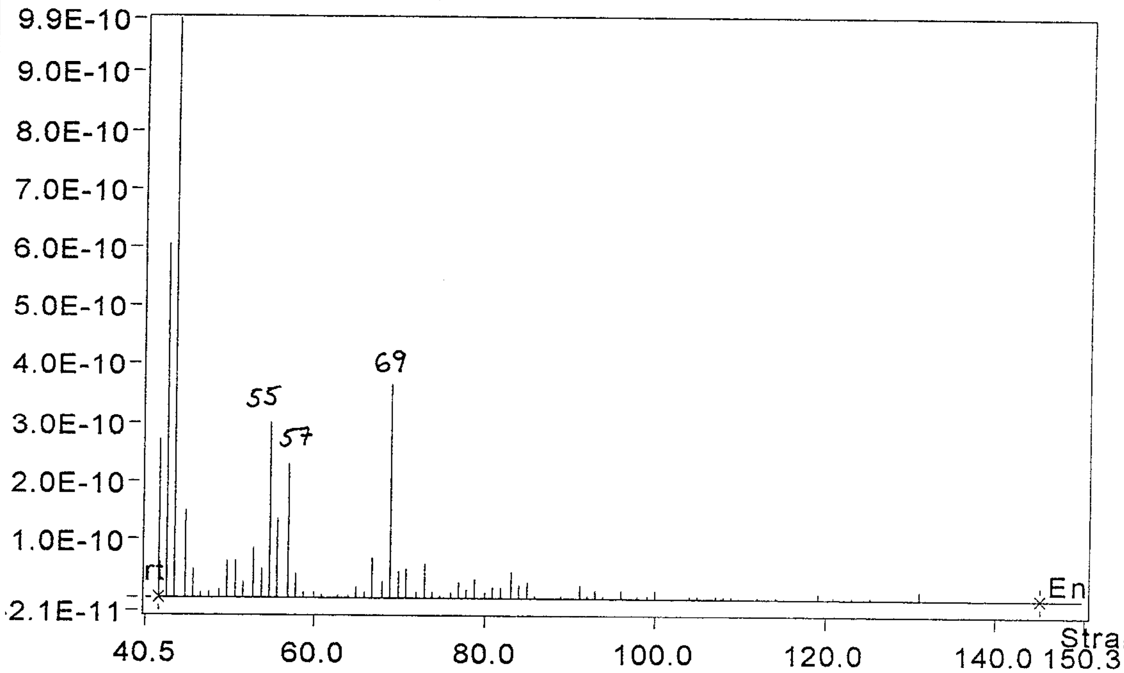
Y Mapping Linear

Records: **20**

Rec No.

18

Sample Temp 184.79



Strart AMU 42 End AMU 150

Start	42.00	0.00E+0
End	145.00	0.00E+0

Baseline correction Above baseline

Baseline Pts 2

Analysis2
C:\Labview5\CAU\DESORB2\Analys2.llb\Analysis2
Last modified on 8/5/98 at 8:09 AM
Printed on 11/9/98 at 9:52 AM

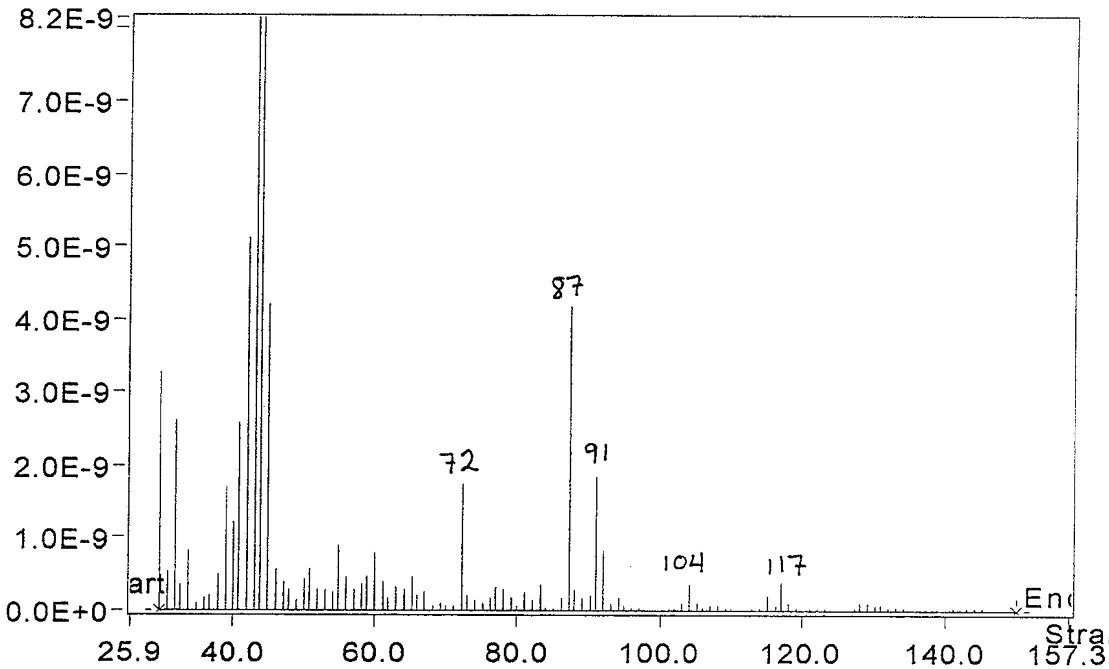
Sample ID: PR-1422 neat

File path: C:\CAUDATA\THIOKOL\Table II\

Spectrum

EXIT

Y Mapping Linear



Records: 21

Rec No.

20

Sample Temp 203.71

Strart AMU 30

End AMU 150

Start	30.00	0.00E+0
End	150.00	7.98E-12

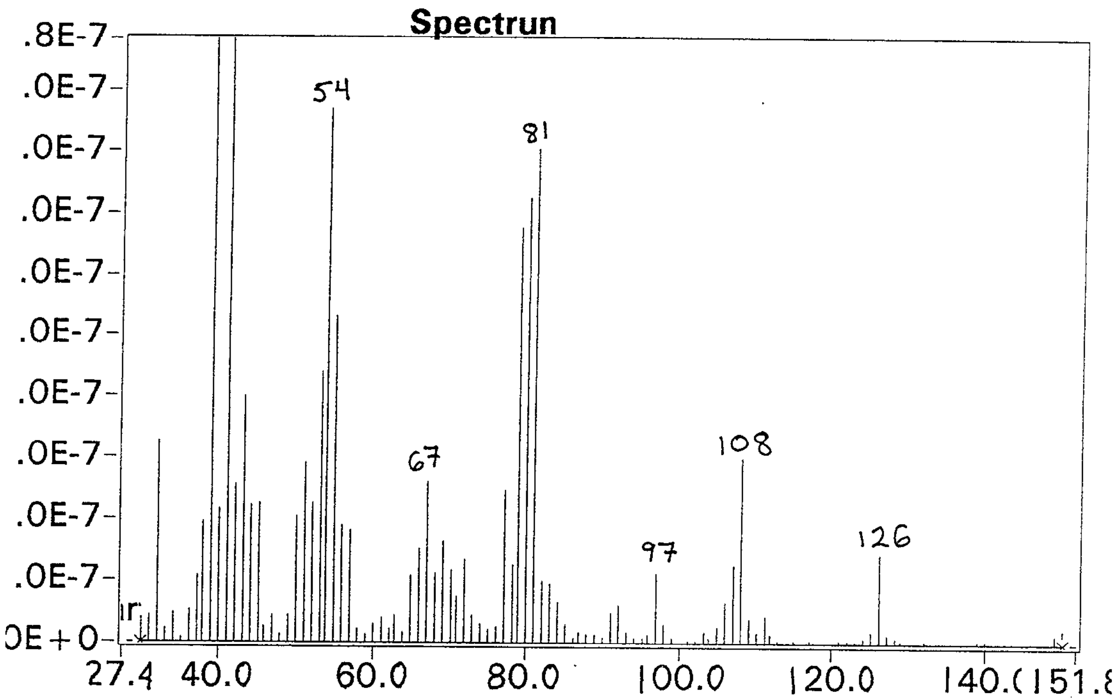
Baseline correction Above baseline

Baseline Pts 2

Analysis2
11/03/98 12:26 PM

Sample I HB Polymer   File pat C:\CAUDATA\TABLEII\110598B

Spectrum   EXIT



Y Mapping Linear

Records: 15

Rec No. 15

Sample Temp .29.1

Start	30.00	0.00E+0
End	50.0	2.09E-9

Strart AM 30   End AM 150   Baseline correc Above baseline   Baseline F 2

APPENDIX B. MASS SPECTRA OF SUBSTRATES

Analysis2
C:\Labview5\CAU\DESORB2\Analys2.llb\Analysis2
Last modified on 8/5/98 at 8:09 AM
Printed on 11/9/98 at 10:57 AM

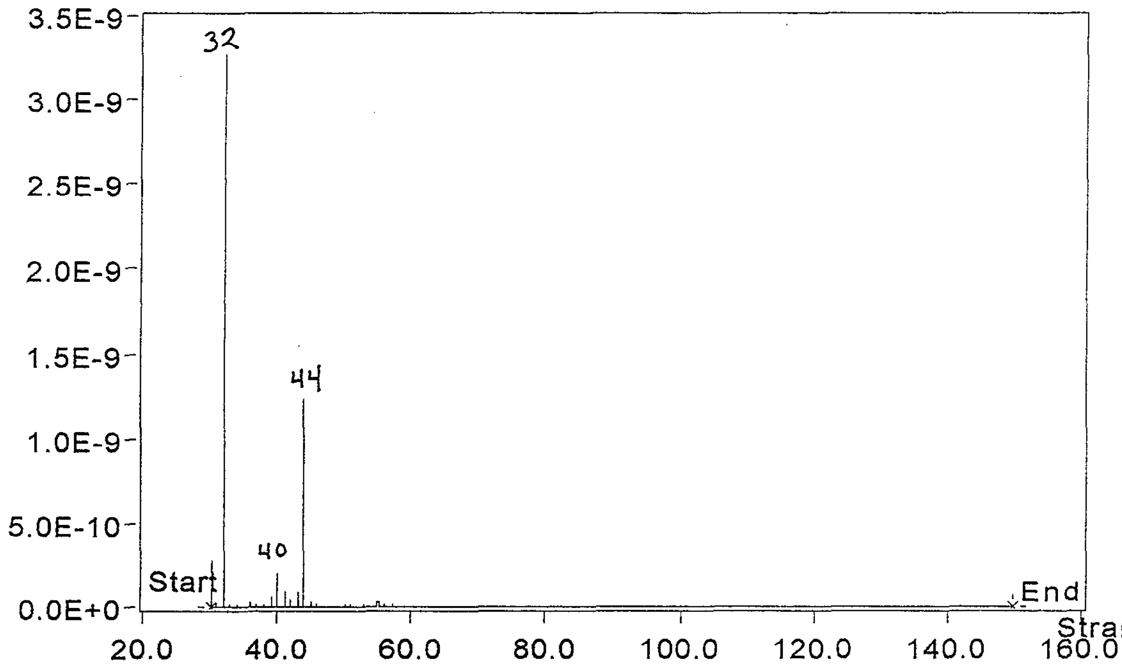
Sample ID: D6 Steel

File path: C:\CAUDATA\THIOKOL\Table III\

Spectrum

EXIT

Y Mapping Linear



Records: **22**

Rec No.

22

Sample Temp 209.91

Strart AMU 30

End AMU 150

Start	30.00	0.00E+0
End	150.00	0.00E+0

Baseline correction Above baseline

Baseline Pts 2

Analysis2
C:\Labview5\CAU\DESORB2\Analys2.llb\Analysis2
Last modified on 8/5/98 at 8:09 AM
Printed on 11/9/98 at 10:57 AM

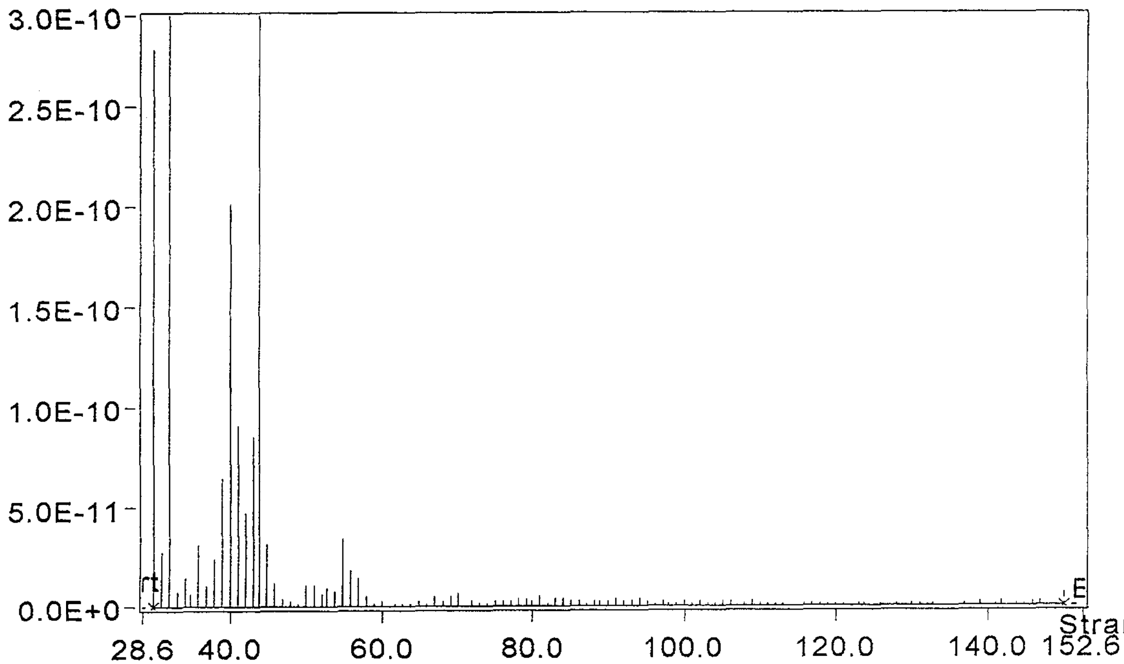
Sample ID: D6 Steel (detailed look at baseline)

File path: C:\CAUDATA\THIOKOL\Table III\

Spectrum

EXIT

Y Mapping Linear



Records: 22

Rec No.

22

Sample Temp 209.91

Start AMU 30

End AMU 150

Start	30.00	0.00E+0	
End	150.00	0.00E+0	

Baseline correction Above baseline

Baseline Pts 2

Analysis2
C:\Labview5\CAU\DESORB2\Analys2.llb\Analysis2
Last modified on 8/5/98 at 8:09 AM
Printed on 11/9/98 at 10:58 AM

Sample ID: Aluminum

File path: C:\CAUDATA\THIOKOL\Table III\

Spectrum

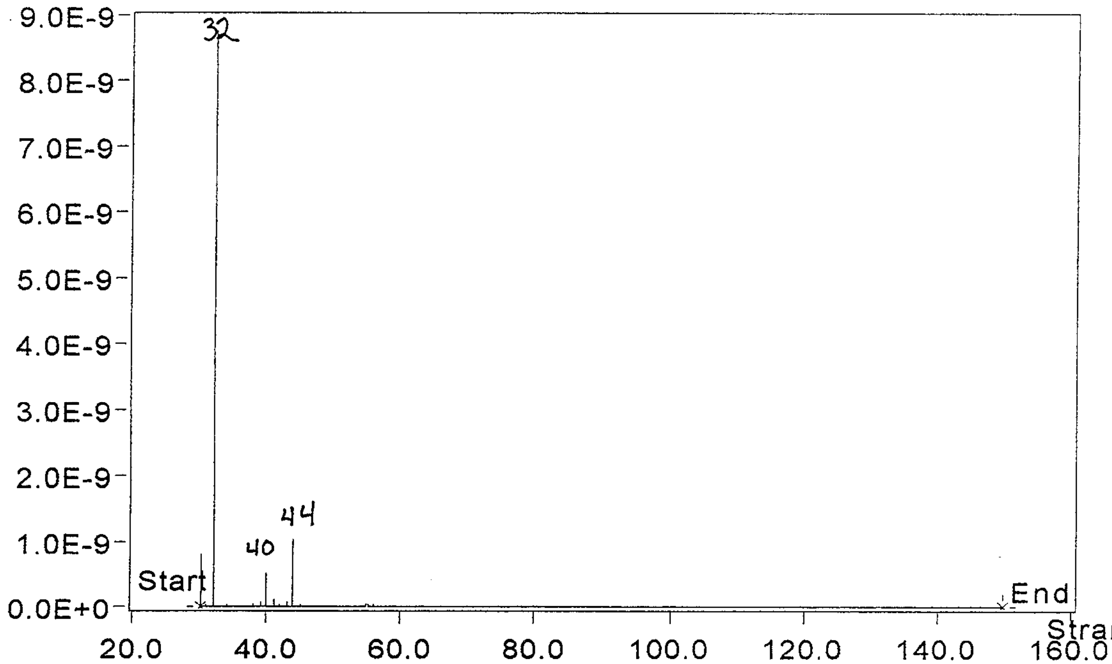
EXIT

Y Mapping Linear

Records: 21

Rec No. 1

Sample Temp 40.04



Strart AMU 30 End AMU 150

Start	30.00	0.00E+0
End	150.00	0.00E+0

Baseline correction Above baseline

Baseline Pts 2

Analysis2
C:\Labview5\CAU\DESORB2\Analys2.llb\Analysis2
Last modified on 8/5/98 at 8:09 AM
Printed on 11/9/98 at 10:59 AM

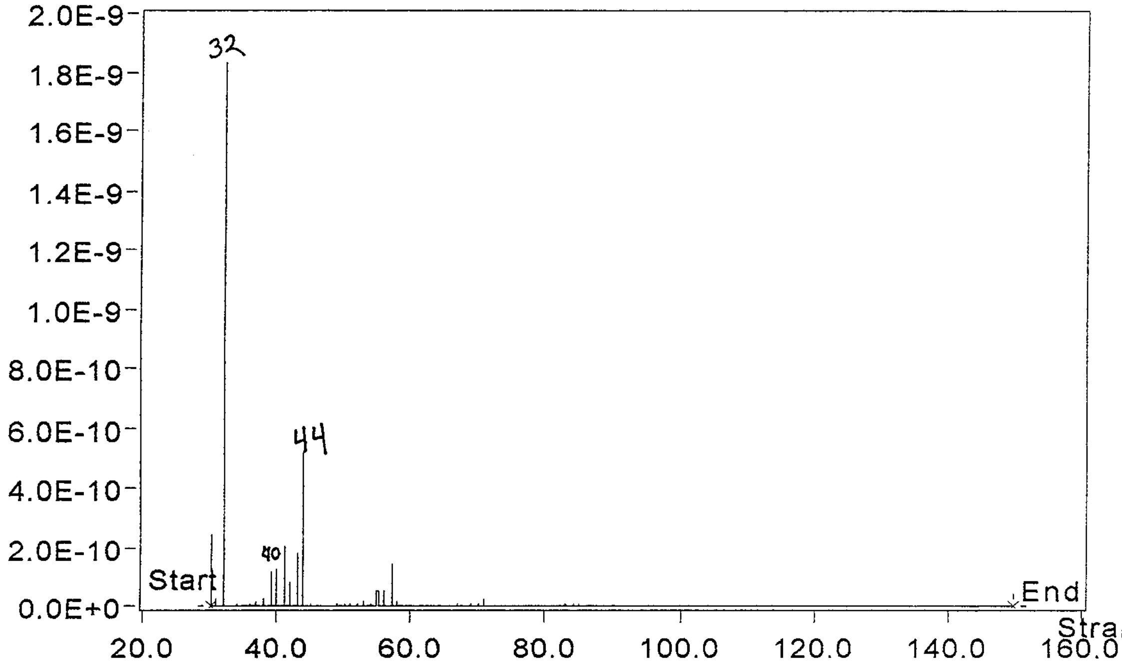
Sample ID: Teflon Coated Aluminum

File path: C:\CAUDATA\THIOKOL\Table III\

Spectrum

EXIT

Y Mapping Linear



Records: 22

Rec No. 1

Sample Temp 34.40

Strart AMU 30 End AMU 150

Start	30.00	0.00E+0
End	150.00	0.00E+0

Baseline correction Above baseline

Baseline Pts 2

Analysis2
C:\Labview5\CAU\DESORB2\Analys2.llb\Analysis2
Last modified on 8/5/98 at 8:09 AM
Printed on 11/9/98 at 11:05 AM

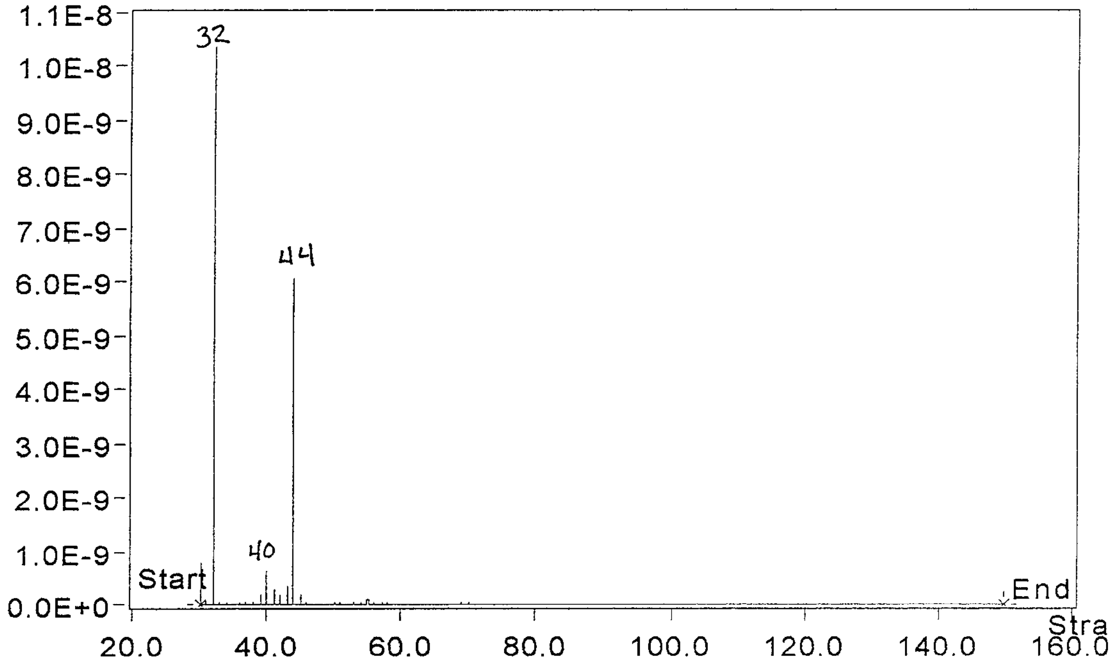
Sample ID: Painted Steel

File path: C:\CAUDATA\THIOKOL\Table III\

Spectrum

EXIT

Y Mapping Linear



Records: **15**

Rec No. 1

Sample Temp 46.22

Strart AMU 30 End AMU 150

Start	30.00	0.00E+0
End	150.00	0.00E+0

Baseline correction Above baseline

Baseline Pts 2

Analysis2
C:\Labview5\CAU\DESORB2\Analys2.llb\Analysis2
Last modified on 8/5/98 at 8:09 AM
Printed on 11/9/98 at 11:00 AM

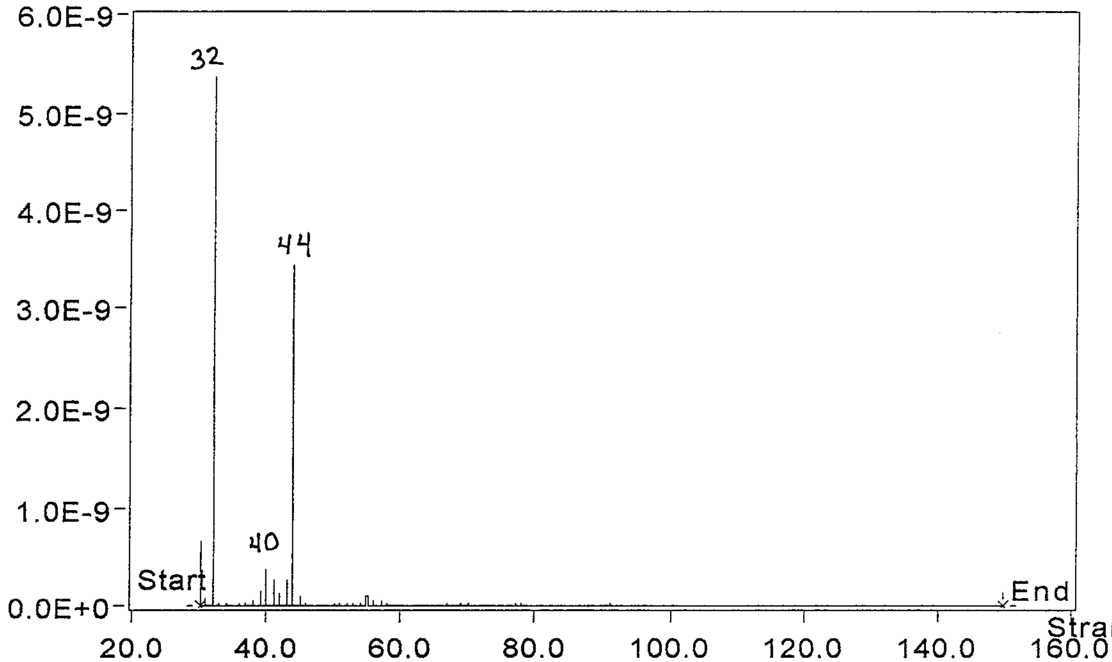
Sample ID: Painted Aluminum

File path: C:\CAUDATA\THIOKOL\Table III\

Spectrum

EXIT

Y Mapping Linear



Records: 16

Rec No. 1

Sample Temp 35.08

Strart AMU 30 End AMU 150

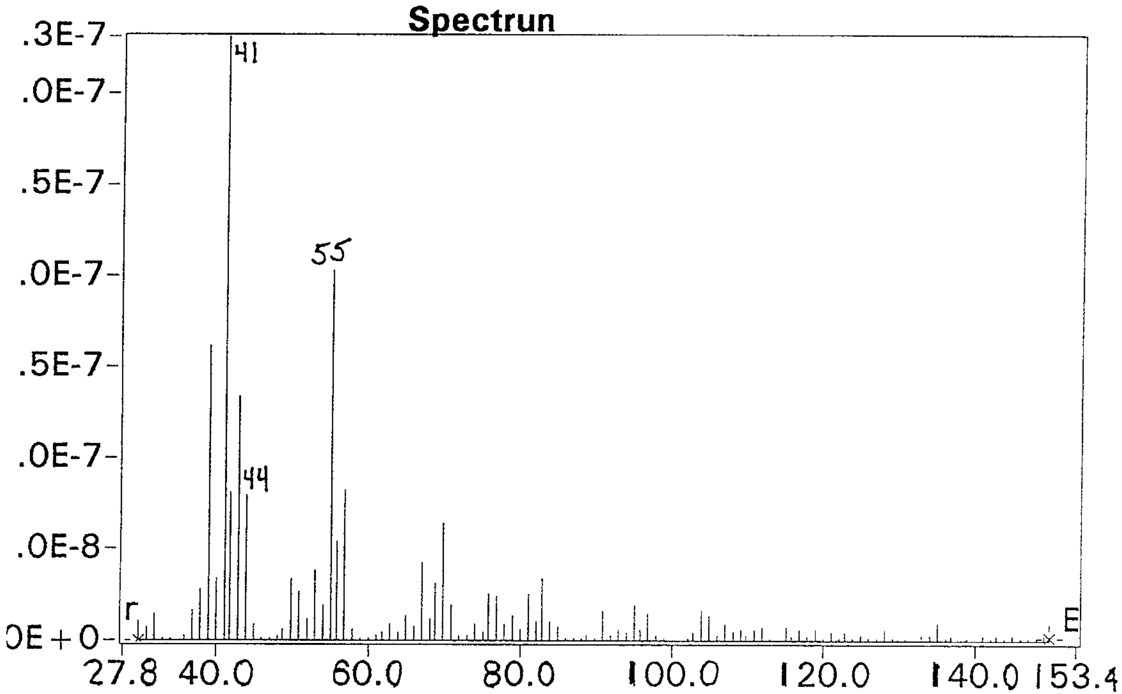
Start	30.00	0.00E+0
End	150.00	0.00E+0

Baseline correction Above baseline

Baseline Pts 2

Sample I NBR/Double File pat C:\CAUDATA\TABLEV\102698B

Spectrum EXIT



Y Mappin Linear

Records: 19

Rec No. 19

Sample Temp .67.0

Start	30.00	0.00E+0	
End	150.0	3.09E-10	

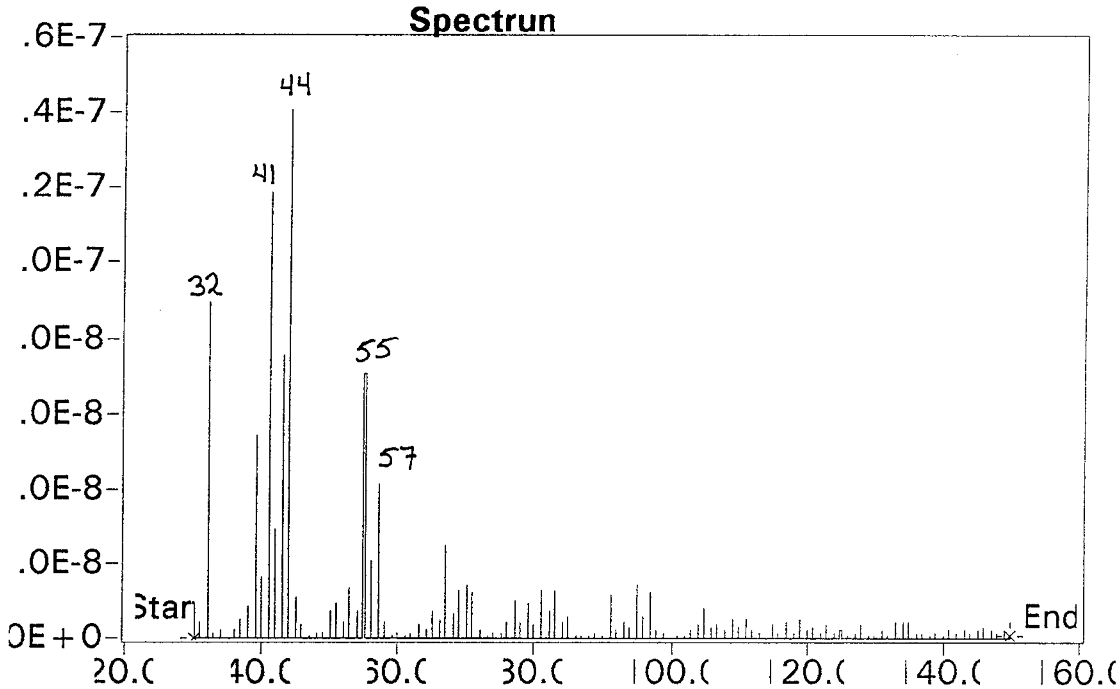
Strart AM 30 End AM 150

Baseline correc Above baseline Baseline F 2

Analysis2
11/03/98 12:35 PM

Sample I GCP/Double File pat C:\CAUDATA\TABLEV\102698C

Spectrum EXIT



Y Mapping Linear

Records: 7

Rec No. 7

Sample Temp 82.77

Start	30.00	0.00E+0
End	150.0	4.93E-10

Strart AM 30 End AM 150

Baseline correc Above baseline Baseline F 2

APPENDIX C. MASS SPECTRA OF CLEANERS

Analysis2
C:\Labview5\CAU\DESORB2\Analys2.llb\Analysis2
Last modified on 8/5/98 at 8:09 AM
Printed on 11/9/98 at 10:20 AM

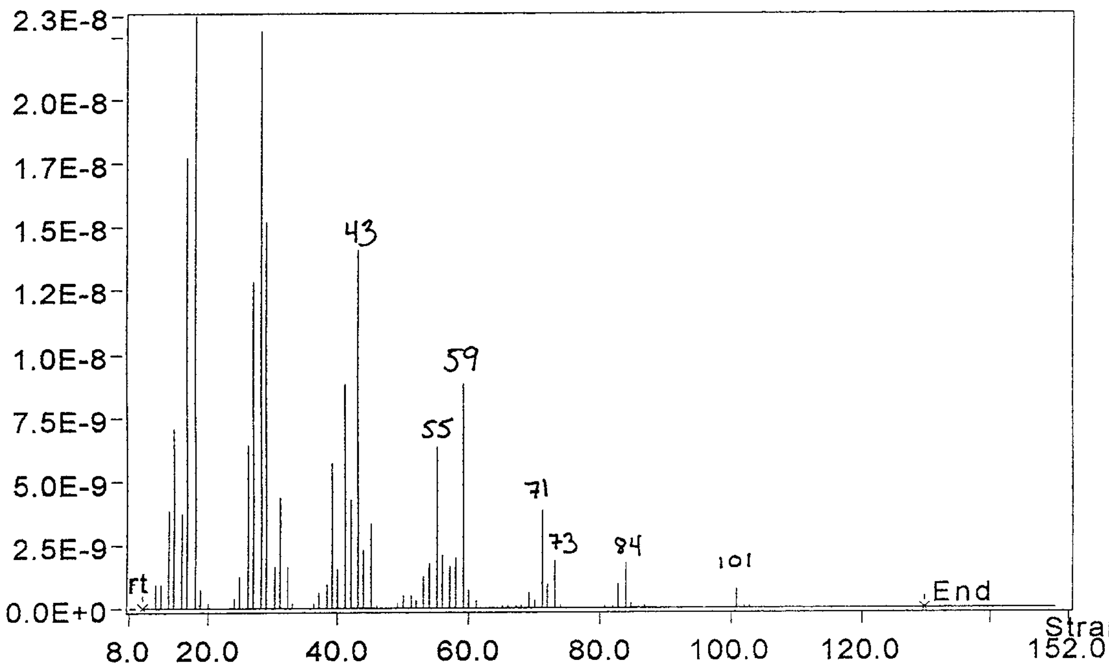
Sample ID: Ionox

File path: C:\CAUDATA\THIOKOL\Table IV\

Spectrum

EXIT

Y Mapping Linear



Records: 23

Rec No.

23

Sample Temp 215.06

Strart AMU 10 End AMU 150

Start	10.00	0.00E+0	
End	130.00	0.00E+0	

Baseline correction Above baseline

Baseline Pts 2

Analysis2
C:\Labview5\CAU\DESORB2\Analys2.llb\Analysis2
Last modified on 8/5/98 at 8:09 AM
Printed on 11/9/98 at 10:15 AM

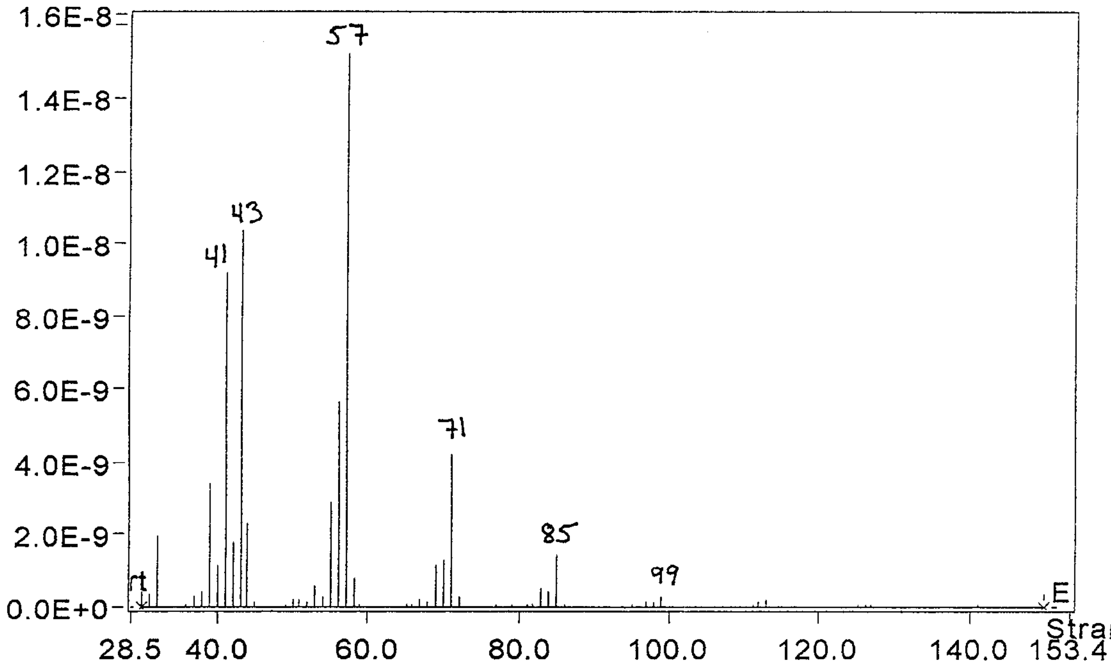
Sample ID: Reveille

File path: C:\CAUDATA\THIOKOL\Table IV\

Spectrum

EXIT

Y Mapping Linear



Records: 23

Rec No.

23

Sample Temp 216.38

Strart AMU 30

End AMU 150

Start	30.00	0.00E+0
End	150.00	0.00E+0

Baseline correction Above baseline

Baseline Pts 2

Analysis2
C:\Labview5\CAU\DESORB2\Analys2.llb\Analysis2
Last modified on 8/5/98 at 8:09 AM
Printed on 11/9/98 at 10:03 AM

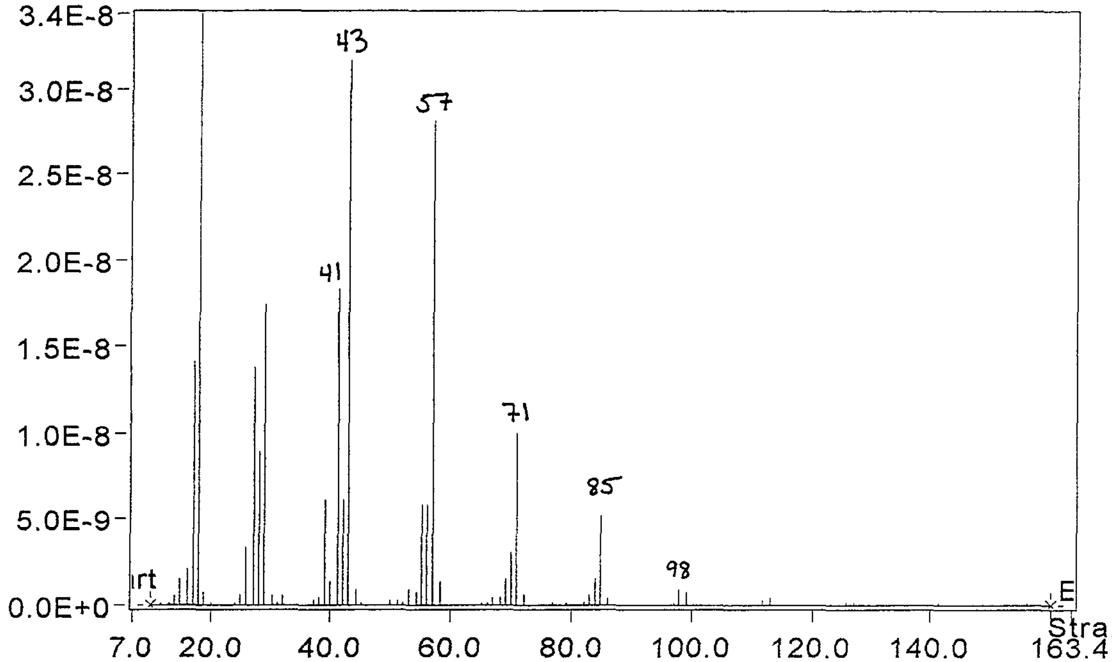
Sample ID: PF Degreaser

File path: C:\CAUDATA\THIOKOL\Table IV\

Spectrum

EXIT

Y Mapping Linear



Records: 15

Rec No. 12

Sample Temp 131.47

Strart AMU 10

End AMU 160

Start	10.00	0.00E+0
End	160.00	3.25E-12

Baseline correction: Above baseline

Baseline Pts: 2

Analysis2
C:\Labview5\CAU\DESORB2\Analys2.llb\Analysis2
Last modified on 8/5/98 at 8:09 AM
Printed on 11/9/98 at 10:31 AM

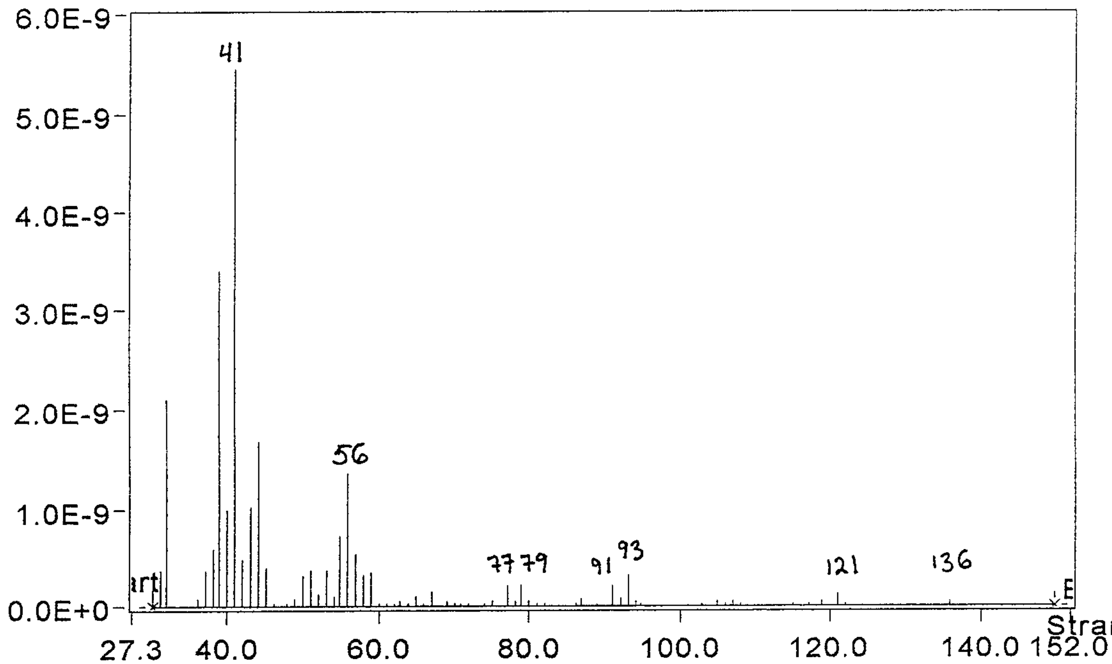
Sample ID: Bio-Act 113 neat

File path: C:\CAUDATA\THIOKOL\Table IV\

Spectrum

EXIT

Y Mapping Linear



Records: 22

Rec No.

22

Sample Temp 212.26

Strart AMU 30

End AMU 150

Start	30.00	0.00E+0
End	150.00	0.00E+0

Baseline correction Above baseline

Baseline Pts 2

Analysis2
C:\Labview5\CAU\DESORB2\Analys2.llb\Analysis2
Last modified on 8/5/98 at 8:09 AM
Printed on 11/9/98 at 10:38 AM

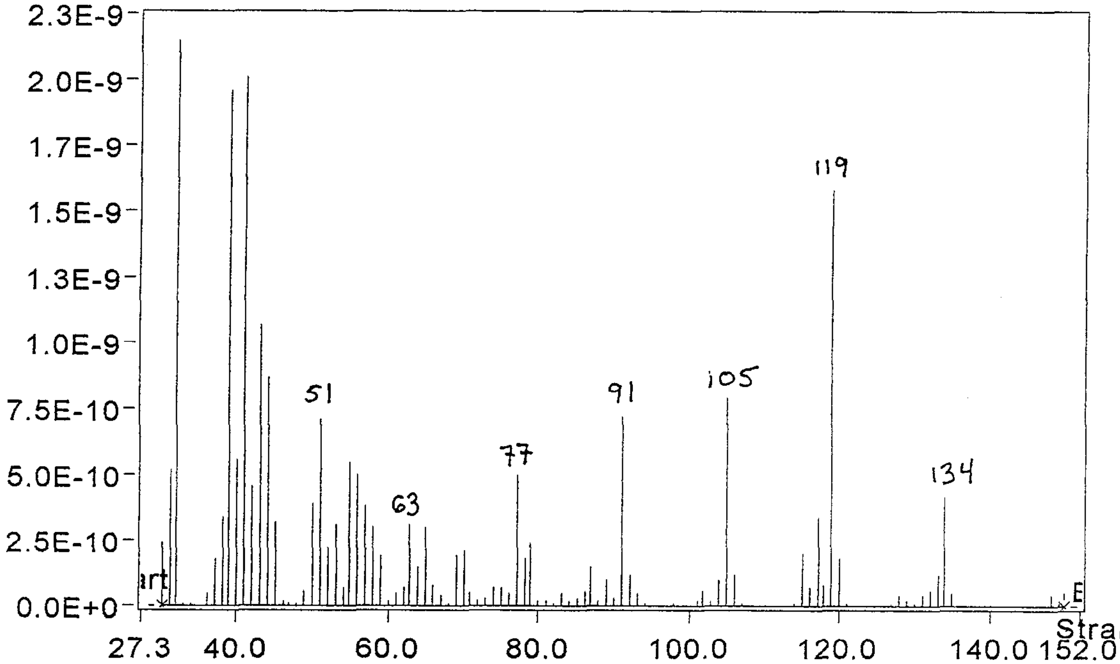
Sample ID: PF Ionsol neat

File path: C:\CAUDATA\THIOKOL\Table IV\

Spectrum

EXIT

Y Mapping Linear



Records: 22

Rec No.

22

Sample Temp 210.99

Strart AMU 30

End AMU 150

Start	30.00	0.00E+0	
End	150.00	0.00E+0	

Baseline correction Above baseline

Baseline Pts 2

Analysis2
C:\Labview5\CAU\DESORB2\Analys2.llb\Analysis2
Last modified on 8/5/98 at 8:09 AM
Printed on 11/9/98 at 10:39 AM

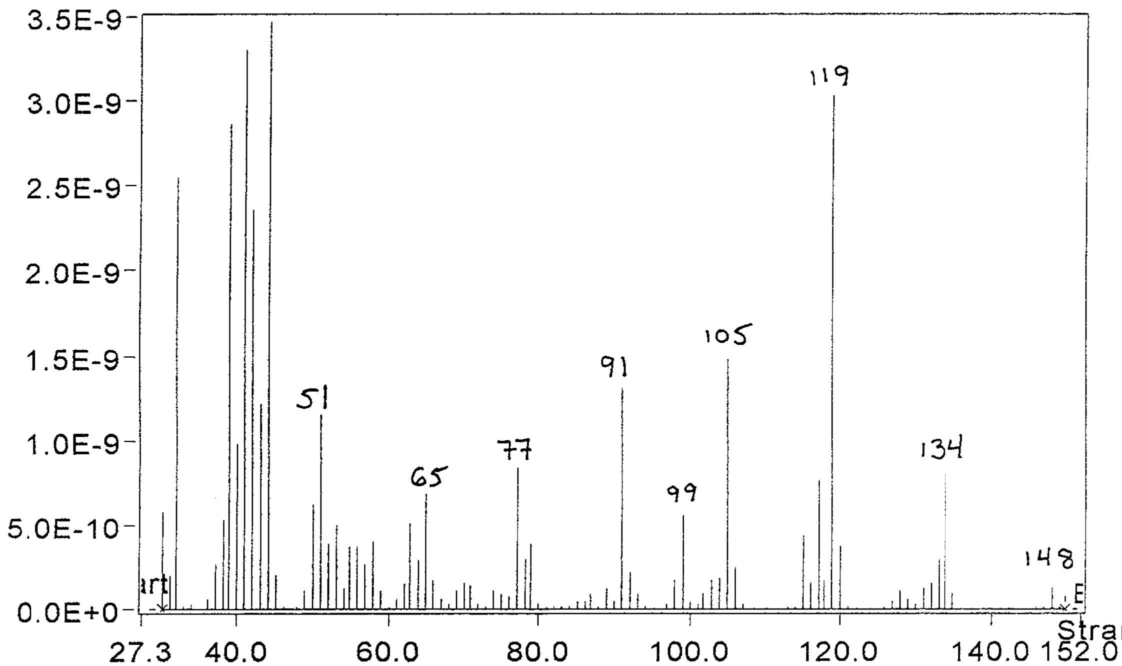
Sample ID: PF d'INK neat

File path: C:\CAUDATA\THIOKOL\Table IV\

Spectrum

EXIT

Y Mapping Linear



Records: 22

Rec No.

22

Sample Temp 215.19

Strart AMU 30

End AMU 150

Start	30.00	0.00E+0
End	150.00	0.00E+0

Baseline correction Above baseline

Baseline Pts 2

Analysis2
C:\Labview5\CAU\DESORB2\Analys2.llb\Analysis2
Last modified on 8/5/98 at 8:09 AM
Printed on 11/9/98 at 10:41 AM

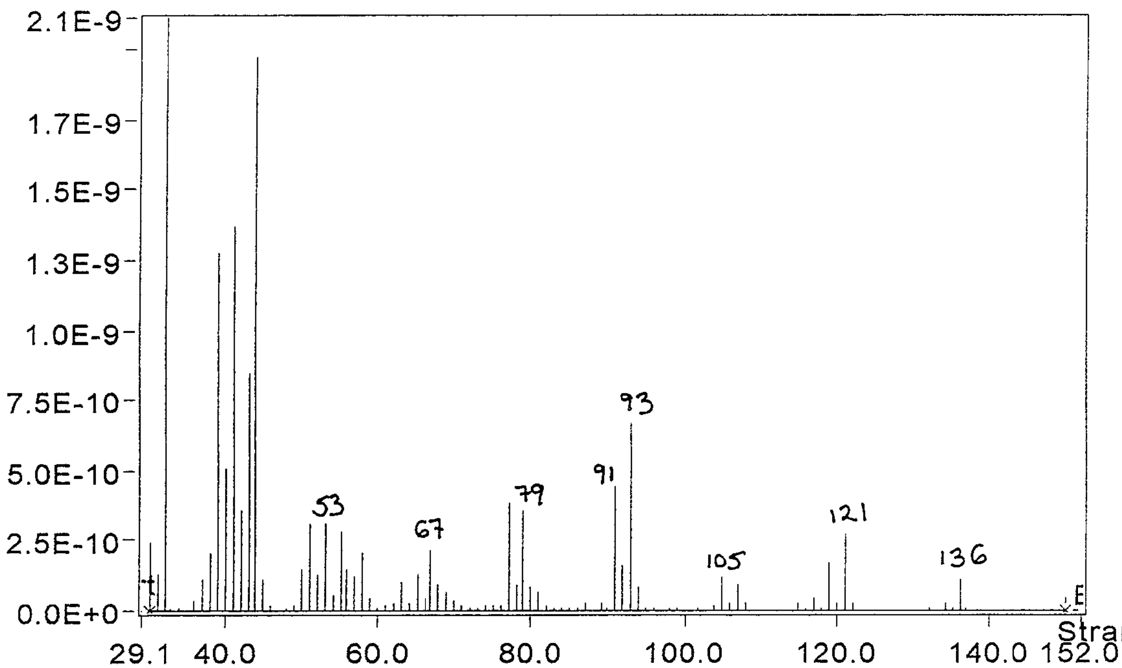
Sample ID: BIOACT PCG neat

File path: C:\CAUDATA\THIOKOL\Table IV\

Spectrum

EXIT

Y Mapping Linear



Records: 22

Rec No.

22

Sample Temp 207.08

Strart AMU 30

End AMU 150

Start	30.00	0.00E+0
End	150.00	0.00E+0

Baseline correction Above baseline

Baseline Pts 2

Analysis2
C:\Labview5\CAU\DESORB2\Analys2.llb\Analysis2
Last modified on 8/5/98 at 8:09 AM
Printed on 11/9/98 at 10:47 AM

Sample ID: Oxsol - 100 neat

File path: C:\CAUDATA\THIOKOL\Table IV\

Spectrum

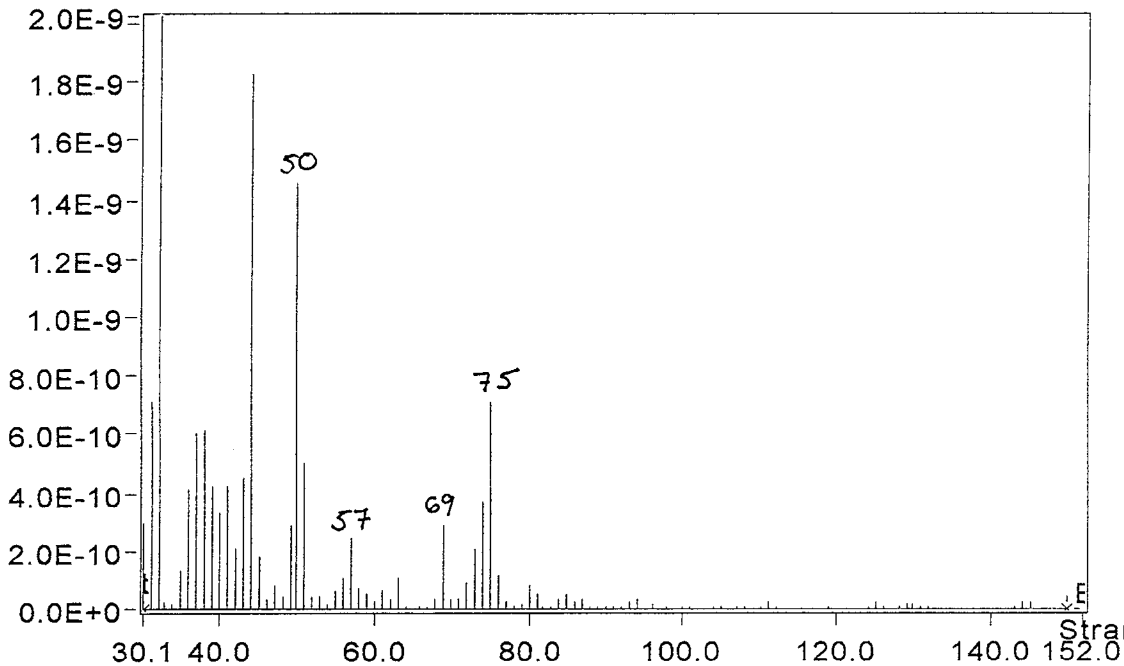
EXIT

Y Mapping Linear

Records: 22

Rec No. 16

Sample Temp 158.13



Strart AMU 30 End AMU 150

Start	30.00	0.00E+0
End	150.00	0.00E+0

Baseline correction Above baseline

Baseline Pts 2

Analysis2
C:\Labview5\CAU\DESORB2\Analys2.llb\Analysis2
Last modified on 8/5/98 at 8:09 AM
Printed on 11/9/98 at 10:50 AM

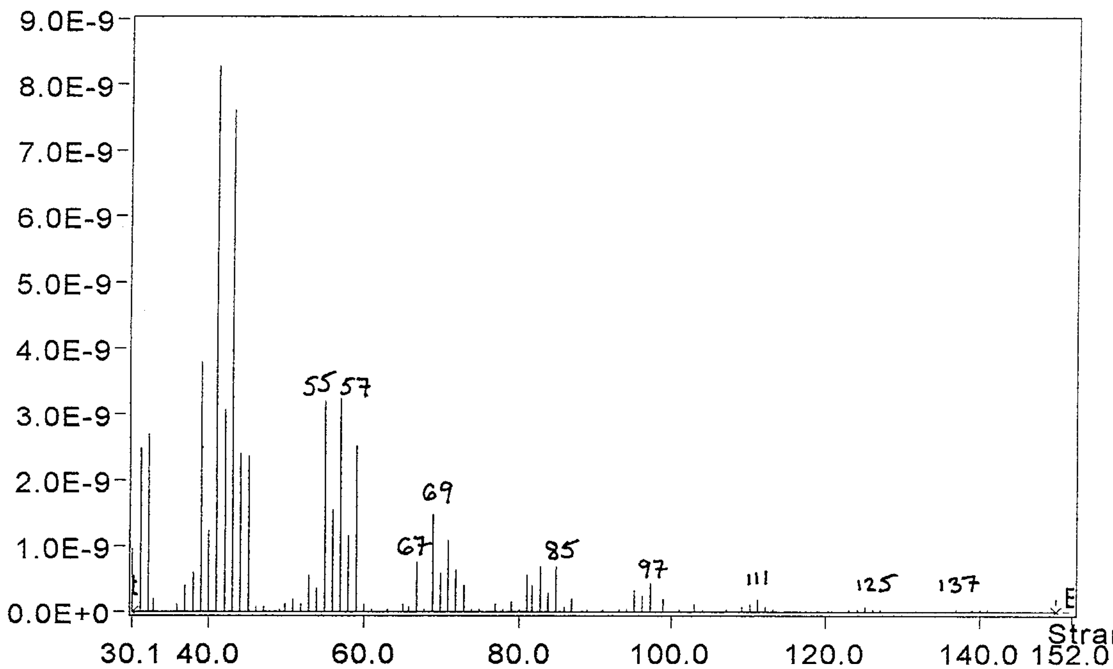
Sample ID: BIOACT 145 neat

File path: C:\CAUDATA\THIOKOL\Table IV\

Spectrum

EXIT

Y Mapping Linear



Records: 23

Rec No.

23

Sample Temp 214.87

Strart AMU 30 End AMU 150

Start	30.00	0.00E+0
End	150.00	0.00E+0

Baseline correction Above baseline

Baseline Pts 2

Analysis2
C:\Labview5\CAU\DESORB2\Analys2.llb\Analysis2
Last modified on 8/5/98 at 8:09 AM
Printed on 11/9/98 at 10:50 AM

Sample ID: Eco-Solv - 5

File path: C:\CAUDATA\THIOKOL\Table IV\

Spectrum

EXIT

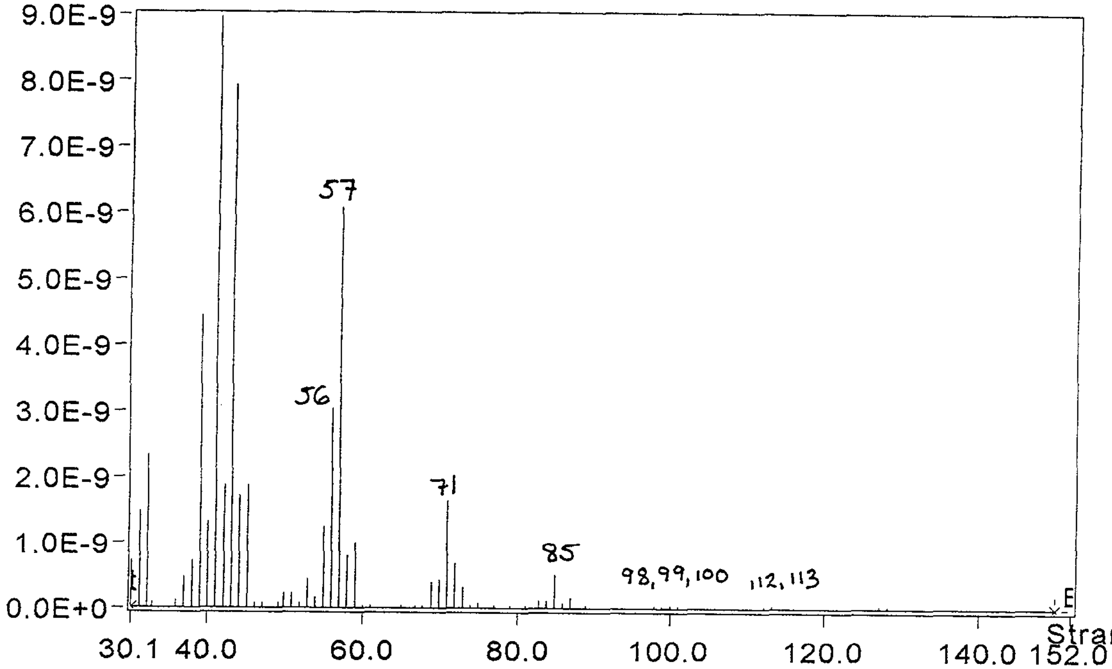
Y Mapping Linear

Records: 23

Rec No.

23

Sample Temp 211.96



Strart AMU 30 End AMU 150

Start	30.00	0.00E+0
End	150.00	0.00E+0

Baseline correction Above baseline

Baseline Pts 2

APPENDIX D – IONS REPRESENTING CLEANER (in red) or CONTAMINANT (in blue)

Chemlok & Ionox	43, 59, 65, 71, 72, 73, 77, **91, 101, 105, 106**
RTV 90-006 & Isopropyl Alcohol	55, 57, 67, 73
EA-946 & Ionox	43, 59, 65, 71, **80, 81, 91/91, 101, 108**
HD–2 & Reveille	55, 57, 67, 71/71, **95, 97, 105, 111**
Tape Adhesive & PF Degreaser	55, 57, 68, 70, 76, **98, 104, 112, 113**
Vacuum Putty & Reveille	57, 69, 70, 71, **98, 99, 100, 119, 131**