New Folder Name Organic Molecules

LIGO PROJECT

CALIFORNIA INSTITUTE OF TECHNOLOGY
PASADENA, CALIFORNIA 91125

FACSIMILE COVER SHEET

LIGO FAX (818) 304-9834 TELEPHONE CONFIRMATION (818) 395-2966

TO	Marty Tellalian CBITSC	DATE FAX NUMBER OFFICE NUMBER	November 4, 1994

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FROM	Larry K. Jones California Institute of Technology 102–33 Bridge Laboratory Pasadena, CA 91125	OFFICE NUMBER	(818) 395-2970

Rai's input for this morning's meeting; sorry about the quality of the photos.

APPENDIX C

ORGANIC MOLECULES

Statistical Group Properties, m in amu

type	$R(x/H_2)$	P(300K) torr	P(77K) torr	RGA peaks amu > 10% max	
H_pC_q	$(0.62+0.115m)m^{0.25}$	$1.3\times10^{3}e^{-m/17.88}$	$3\times10^{-2}e^{-m/29.35}$	57,55,53,51,43,42,41	*
$H_pC_qO_k$	$(0.81+0.09m)m^{0.25}$			m,55,53,51,43,42,41	**
$H_pC_qHlgn_k$	$(1.37+0.06m)m^{0.25}$			m,51,43,42,41,39	***

* Oils and waxes

** plasticizers,alcohols and esters

*** aromatic compounds

References:

International Critical Tables, Landolt - Bornstein (Indices of Refraction)

Handbook of Chemistry and Physics, CRC Press (Vapor Pressure)

API/NBS Mass Spectra Tables, NBS (RGA spectra)

Eight Peak Index of Mass Spectra, UK Atomic Energy Authority (RGA spectra)

The Wiley/NBS Registry of Mass Spectral Data, Wiley (RGA spectra amu 2 - 2724)

As a general rule the ionization cross section for H_p C_q hydrocarbons increases linearly with m and the partition of the molecule into different mass peaks increases as m as well. On average therefore, the ion current at the major peaks (> 10% max peak) stays constant with m. As m increases more amu peaks become populated at small % of the max peak. The sensitivity of an RGA in terms of positive ion current/pressure of the neutral molecule (the gauge factor) stays approximately constant if one records several of the more common hydrocarbon fraction mass peaks.

Phase noise from organic gases (H_pC_q)

$$P_{pq} = \frac{P_{H_2}}{R^2}$$

Cut off in organic gas mass at m >602 amu since vapor pressure at 300K for higher masses satisfies phase noise goal.

amu	$P_{pq}(H_2(equiv)$ torr	J(end pump) t l/s cm^2	J(9 pump) t l/s cm^2	J(btd) ss t l/s cm^2	J(stack) ss t l/s cm^2
50	3.5×10^{-12}	4.0×10^{-17}	2.5×10^{-15}	$< 2.6 \times 10^{-18}$	$< 1.8 \times 10^{-17}$
100	6.8×10^{-13}	5.6×10^{-18}	3.5×10^{-16}	$< 1.8 \times 10^{-18}$	$< 1.3 \times 10^{-17}$
200	1.3×10^{-13}	7.5×10^{-19}	4.8×10^{-17}	$< 1.3 \times 10^{-18}$	$< 9.1 \times 10^{-18}$
300	4.7×10^{-14}	2.2×10^{-19}	1.4×10^{-17}	$< 1.1 \times 10^{-18}$	$< 7.7 \times 10^{-18}$
400	2.3×10^{-14}	9.4×10^{-20}	6.0×10^{-18}	$< 9.3 \times 10^{-19}$	$< 6.5 \times 10^{-18}$
500	1.3×10^{-14}	4.7×10^{-20}	3.0×10^{-18}	$< 8.3 \times 10^{-19}$	$< 5.8 \times 10^{-18}$
600	8.4×10^{-15}	2.8×10^{-20}	1.8×10^{-18}	$< 7.6 \times 10^{-19}$	$< 5.3 \times 10^{-18}$

Assumptions:

In BTD measurements amu 41 and amu 55 equal the pressure of the heavy hydrocarbon.

Pumping speed of BTD: $F \approx 1.1 \times 10^3/\sqrt{m}$ liters/sec.

LIGO pumping speed limited only by tube conductance.

Results of Surface Analysis of Cleaned LIGO Tube Steel

N. Mavalvala and R. Weiss, May 3, 1994

Goals for the measurements:

1. Determine which of the field applicable cleaning methods currently under consideration leaves the minimum hydrocarbon contamination on the surface.
2. Make a correspondence between surface contamination and hydrocarbon outgassing rate.
3. Establish if the current cleaning techniques are adequate for the LIGO outgassing requirements.

Samples and Techniques:

Samples were tested for surface and subsurface hydrocarbon contamination after cleaning by several field applicable techniques. The samples and cleaning techniques used were:

1. Oakite 33 (102F)
2. Steam (122 - 132F)
3. Steam and Mirachem 500 Cleaner/Degreaser (122 - 132F)

The samples measured had been covered with SAE 30 non-detergent motor oil prior to cleaning.

To gain insight and for calibration, additional samples were analysed, these are:

4. CBI steam cleaned followed by multiple cleaning in boiling isopropyl alcohol and acetone - oxide coated super clean sample.
5. Flycut bare metal sample followed by super cleaning procedure.
6. Beamtube demonstration project original steel sample which had been Oakite 33 cleaned by the project (1990?) and whose outgassing properties were measured in the beamtube demonstration project. This sample is the only connection we currently have relating the surface measurements to a hydrocarbon outgassing rate.

Surface analysis methods used:

1. Auger electron analysis - elemental composition of the surface and subsurface.
2. Secondary Ion Mass Spectroscopy (SIMS) - mass spectrum of surface and subsurface (2 - 500 amu)

3. X-ray Photoelectron Spectroscopy (XPS) - elemental composition of surface and subsurface with some bonding information. The technique was tested but abandoned after the SIMS measurments showed better sensitivity.

Handling of samples:

Samples were cleaned at CBI in 1 by 18 inch strips and packed in "Ameristat" plastic film for shipment. The sample were cut into 1 cm squares at MIT for surface analysis. The cutting was carried out by placing the samples in aluminium holder clamps that had been dry surface machined and multiple cleaned in isopropyl alcohol and acetone. The samples were cut with cleaned hack saw blades while in the holders. The samples were transported and maintained in these holders. "Nitrilite" gloves and masks were used while handling the samples with degreased tools.

Results :

Of the three cleaning techniques tried, steam leaves the smallest hydrocarbon contamination on the surface followed closely by steam with detergent. The Oakite - 33 left a marked contamination. A summary of the Auger data is shown in figure 1 and the SIMS data in figure 2.

Figure 1 is a plot of the Auger carbon peak counts averaged over three areas of the sample vs time of Argon ion beam etching. The rate of etching is approximately 50A per minute

Figure 2 shows the counts in the amu 12 channel (Carbon, negative ions) when the ion emission is stimulated by 10Kev positive Cesium ions. The data is averaged over three areas on the sample. The Cesium both etches the surface at an estimated rate of 50A/scan as well as coats the surface thereby enhancing the emission of negative ions. The rise in counts at amu 12 as well as at most other mass numbers (not shown in the figure) after more than 10 scans is due to this enhancement and confuses the measurement.

The mass scans extended to amu 250, all samples showed peaks attributable to hydrocarbons between amu 40 through 100. These peaks were generally under 5% of the amu 12 peak. No peaks above background were seen in any of the samples above amu 160.

The Auger and SIMS scans before etching show carbon peaks for all samples: both CBI cleaned and laboratory cleaned as well as the oxide coated and the bare metal surfaces. This initial layer is most likely due to adsorbed CO_2, CO and loosely bound airborne hydrocarbons. None of the surface measurement facilities at MIT currently are able to bake the samples in vacuum at 140C before surface analysis. This would be a useful step in the surface evaluation since it will be more characteristic of the surfaces in the LIGO and a bake would be expected to remove these loosely bound contaminants.

The data after some etching, especially in the Auger analysis, differentiates the samples even though the oxide coated surface is irregular and the etching rate is variable depending on the area of the sample chosen.

Both Auger and SIMS show that the Oakite 33 cleaned surfaces has carbon on the surface that penetrates into the oxide coated surface by several 100 Angstroms. The oxide is

estimated to be 500 ±200 A thick. In the Auger analysis the surface is etched by argon ion bombardment to make the depth assay. There was clear evidence for mobile surface films that moved into the etched regions indicating oil or wax remaining on the surface. Such a phenomena was not experienced in the other samples.

The Auger analysis showed tracer peaks of sodium in the steam and detergent cleaned samples attributed to the Mirachem 500 degreasing compound that penetrated 50 to 100 A into the surface.

Oxide coated surfaces show more hydrocarbon contamination irrespective of the cleaning technique (including the super cleaning) than the bare metal. The estimated surface density of hydrocarbons on the steam cleaned oxidized surface is less than 0.1 monolayers.

Relation of surface contamination measurements to hydrocarbon outgassing rate: A prediction of the hydrocarbon outgassing rate from basic principles given the measurement of the surface contamination can, with our present state of knowledge, only be guessed. The critical factor is the binding energy of the contaminant to the surface. We are trying to estimate this to set bounds. A somewhat more reliable approach comes from the measurements of the beam tube demonstration steel sample for which upper limits of the hydrocarbon outgassing rate after the bake were determined. Providing that the samples measured were not further contaminated over the 3 years during which they were stored in polyethelyne bags, the hydrocarbon contamination on these samples seems to be comparable to that on the other oxide coated samples as measured by the SIMS and higher when measured by the Auger technique after a few minutes of Argon etching. A reasonable assumption would then be that the outgassing rate of the steam and the steam with detergent cleaned samples will be comparable or lower than the btd. A new piece of information from the SIMS measurments is that the amu of the hydrocarbon contaminants is most likely smaller than 160 so that beamtube demonstration upper limits for the phase noise from hydrocarbons has margin.

Conclusions and recommendations: Steam cleaning of the oxidized steel leaves comparable contamination on the surface as the super cleaning technique. It is therefore unlikely that any new cleaning techniques will do better on the oxidized steel than steam cleaning. We recommend that steam cleaning be adopted for the qualification test.

A major change in hydrocarbon contamination could come from removing the oxide. The hydrocarbon outgassing of the steam cleaned oxidized steel will be one of the results of the qualification test at CBI. There is logic in pursuing a parallel program in comparing the hydrocarbon outgassing rates of steam cleaned oxidized and bare steel in the event that the qualification tests are unsatisfactory. The benefits of keeping the oxide coating, however, are not small. They are the attenuation of stray light in the tubes, the known hydrogen outgassing properties of the steel and the economical factor of minimizing the processing cost of the steel.

Legend for figures

ok = Oakite 33 cleaned

s = steam cleaned

sd = steam cleaned with Mirachem 500

ssc = steam cleaned followed by boiling isopropyl alcohol and acetone

fsc = flycut followed by boiling isopropyl alcohol and acetone

btd = beam tube demonstration original steel (yellow oxide)

file:clnagenda110494.txt
to: Larry, Al, Gerry, Gary
from: R. Weiss November 3, 1994
concerning: Agenda for phone call on cleaning problems

1) Set problem into context

   a) Beamtube demonstration data on outgassing rates for hydrocarbons and projection to LIGO (refer to table 1 on printer)

   b) Connection to surface measurements with Auger and SIMS fig 1 and SIMS data of largest amu seen in surface analysis, surface analysis document in printer

2) The current situation

   a) Marginal but not devastating - surface analysis comparisons: Fig 1 through Fig 7. How well can one do on the oxide coated surface, fig 1 SSC sample.

   b) Known oil contamination fig 15 and 16. Missing analysis of effluent to determine global extent. Data only from "weepers".

   c) "Weepers". Not remarkable in surface analysis fig 8 through fig 14. Concentrated on oxide, associated with depressions in surface, surface coverage 10^-5. Guess that the contamination in the "weepers" is the same as on the general surface.

   d) Steam only cleaned tube does not pass water break test and is visibly dirty, shows fluorescence.

   e) Detergent and steam followed by steam does pass water break test is not visibly dirty but still shows attenuated fluorescence.

3) What one would like

   a) More margin than we now have.

      i) Improve cleaning technique

      ii) Use option of distributed pumps for advanced interferometers

          Hydorcarbon column density ~ (L^2)*sqrt(amu)

   b) Make cleaning technique more robust to allow for more tolerance in fabrication

   c) Develop techniques for quality assurance of the cleaning by direct measurement rather than only demanding adherence to a prescription.

4) Constraints

   a) Cleaning chemistry should not increase the hydrogen outgassing

   b) Need to maintain surface roughness and optical absorption of the tube to avoid phase noise from scattering

   c) Must be field applicable

      i) costs

      ii) schedule (time)

iii) environmentally compatible and safe for personnel

5) Cleaning improvement options open to us

a) Improve procedure with detergent and steam

i) Foam concept

ii) Higher detergent surface dwell time and larger concentration to increase surface wetting (reduction in surface tension).

b) Introduce solvent cleaning step after detergent/steam cleaning
May violate items 4 c) i through iii. Need input from CBI.

6) My recommendation for a procedure

The only on line analysis tools available at CBI are the fluorescence technique and the water break test. The FTIR and surface analysis cost about $700 and about 4 days (when all falls into place) per attempt. To go forward we must call it quits at some point and get to the end point - the actual hydrocarbon outgassing after bakeout associated with a particular process.

Suggest that CBI make a test run of the technique recommended by the Mirachem Corporation (most likely the foam technique) and on the steam/detergent/solvent technique providing that it is economically feasible in the field. Then adopt that process which gives the smallest (not necessarily zero) fluorescence and clean both QT tubes with it. They collect effluent samples for FTIR and have a witness piece in the tube for surface analysis which are analysed to establish a baseline for future reference while they go on to the rest of the QT. The chances are reasonable but not certain that the tube will pass our criterion for hydrocarbon outgassing.

The project implement the long standing plan to setup a hydrocarbon outgassing test bench as insurance against the possibility that the QT does not pass the hydrocarbon outgassing requirement.

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Cambridge, Massachusetts 02139

MIT GRAVITY GRP. FAX #617-253-7014
CONFIRMATION # 617-253-4824

Facsimile Cover Sheet

DATE: Nov 3, 1994 TIME: 9:25 PM (E.T.)

TO: L. JONES FAX#: _____

_____ ADDRESS: _____

_____ _____

NUMBER OF PAGES (including this cover sheet): 15

FROM: R. WEISS OFFICE #: (617)253-_____

Massachusetts Institute of Technology
Room 20B-145
Cambridge, Massachusetts 02139

NOTES:

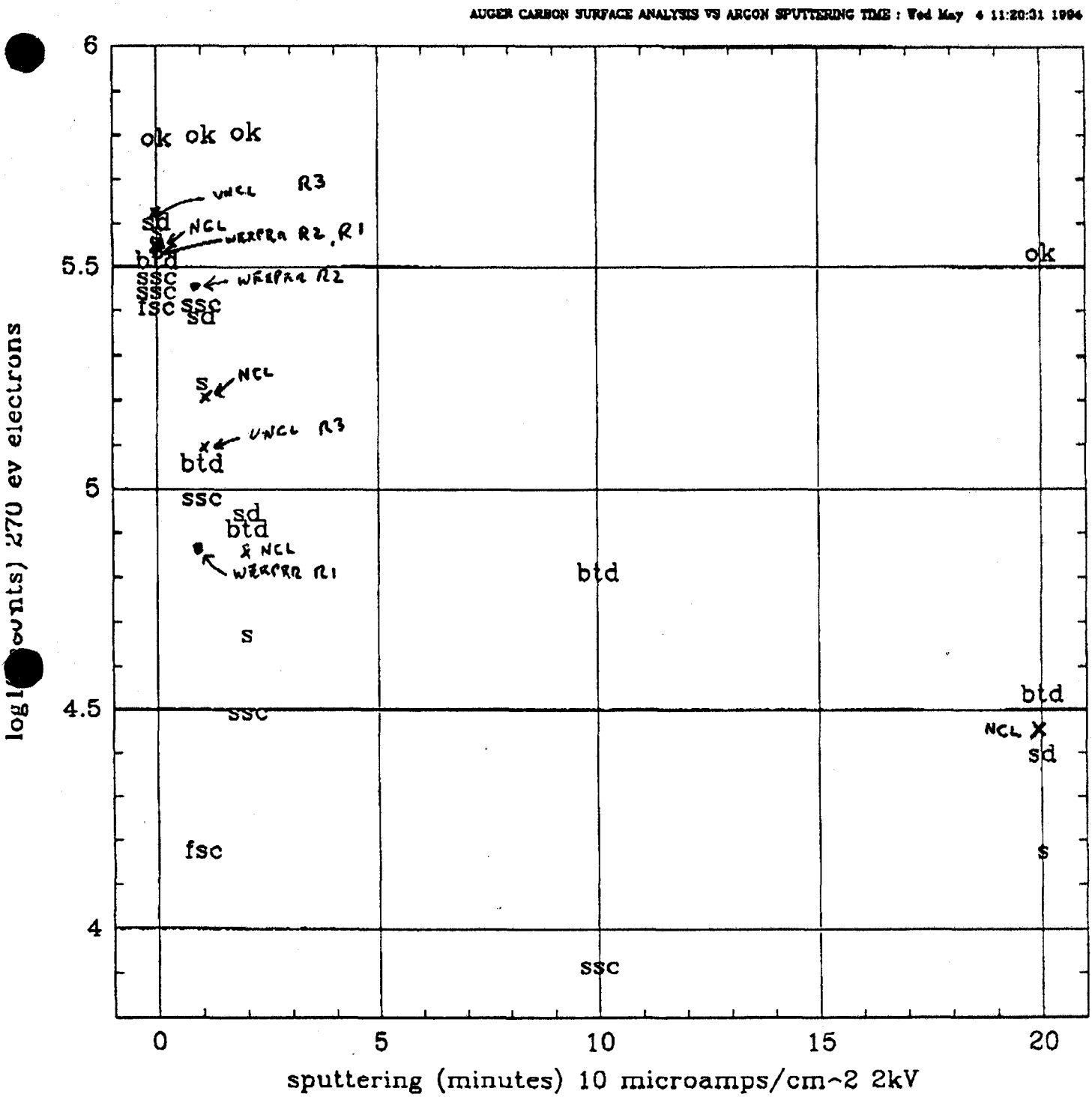
FOR MORNING MEETING ON CLEANING

DISTRIBUTE TO

AL LAZZARINI GARY SANDERS

GERRY STAFFER

FIG 1



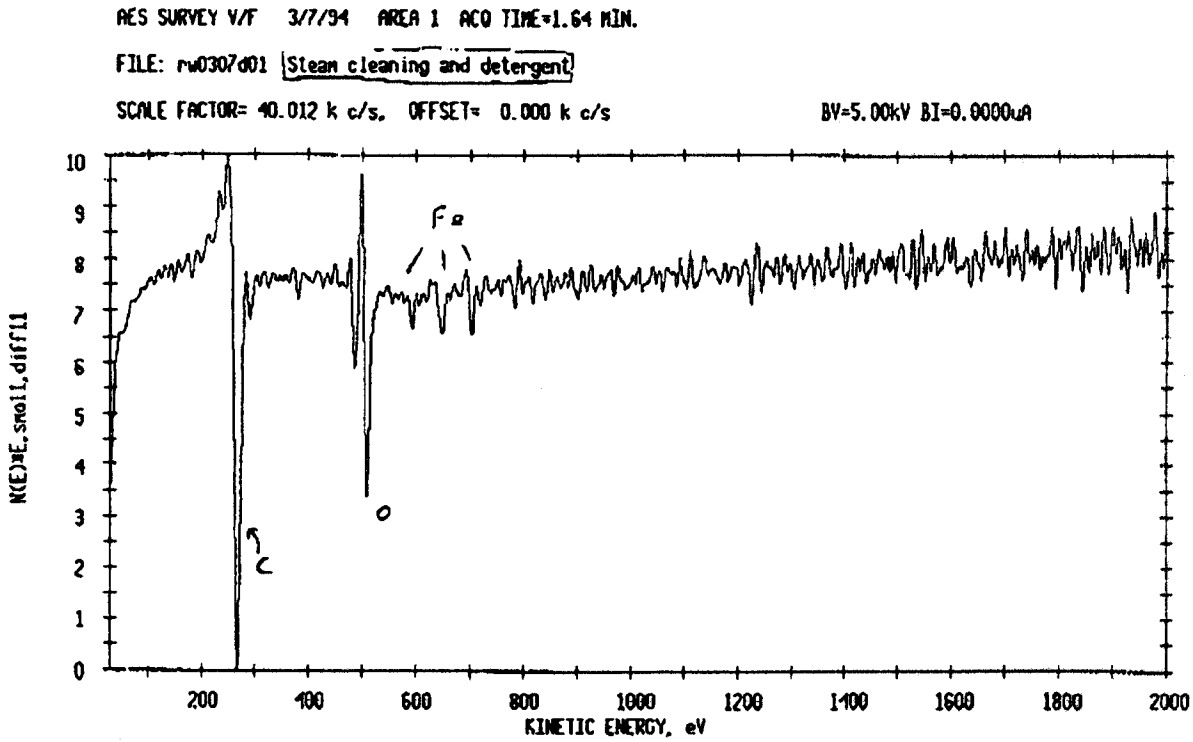
LEGEND

OK	OAKITE 33	CB1
S	STEAM ONLY	
SD	STEAM + MIRACHEM 500	
SSC	OKIDE COATED	SUPER CLEANED
FSC	FLY CUT STEEL	SUPER CLEANED
BTD	BEAM TUBE DEMONSTRATION	STEEL

NCL NEW STEEL MIRACHEM AND H_2O BOIL 1 MIN / 2 MIN

UNCL UNCLEANED EXCEPT TO EXPOSE WRKPRAS WITH PROPANOL

FIG 2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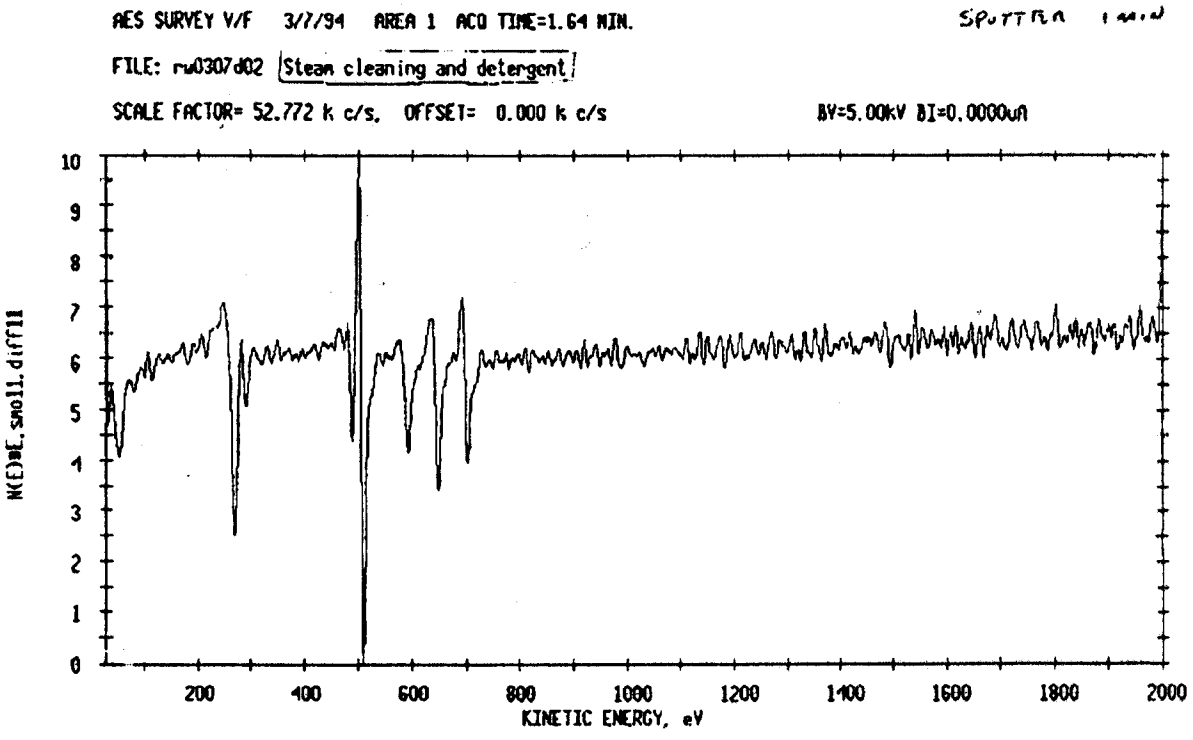
N(E)dE, smo11, diff11
KINETIC ENERGY, eV
Fe
O
C


AES SURVEY V/F 3/7/94 AREA 1 ACQ TIME=1.64 MIN.
FILE: nw0307d02 Steam cleaning and detergent
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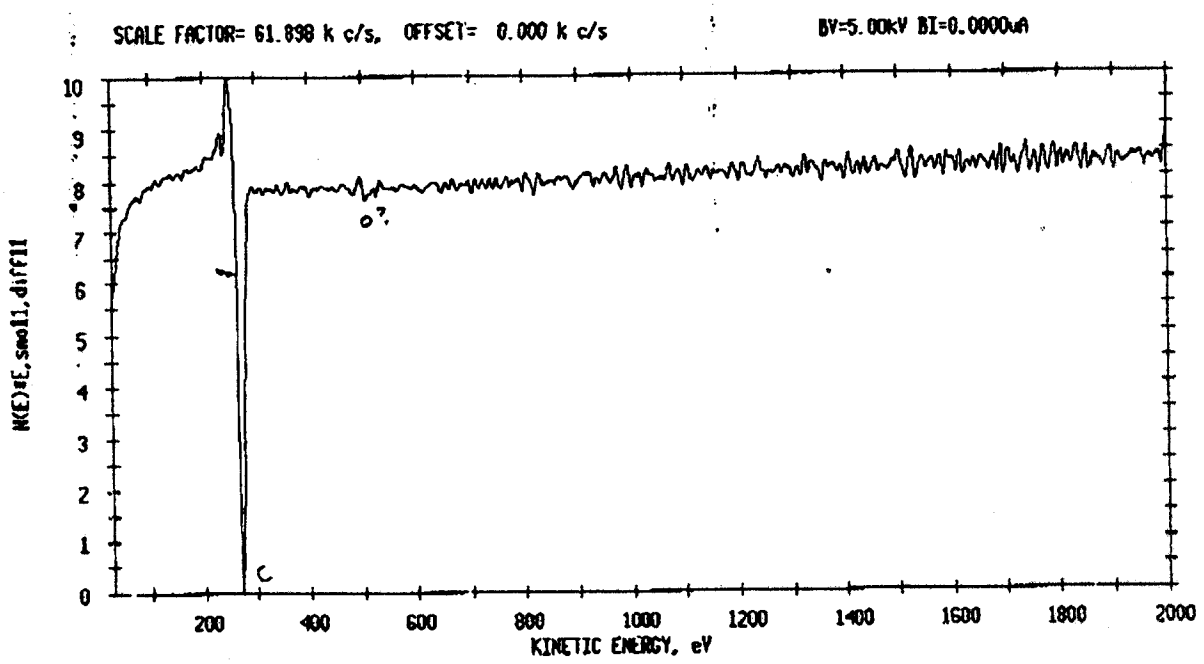
BV=5.00kV BI=0.0000uA
N(E)dE, smo11, diff11
KINETIC ENERGY, eV

FIG 3

'Ebeam 5kV, 100nA
point
Ibeam 2kV, 20μA/c



after 1 min sputter

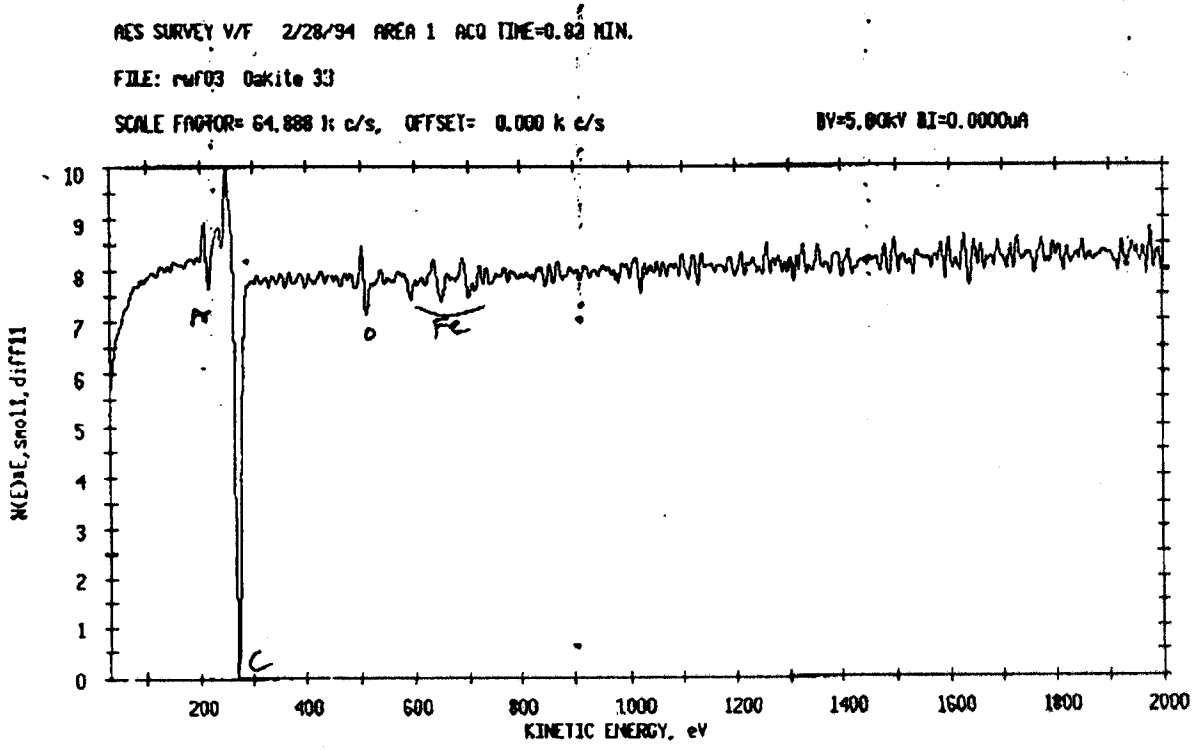
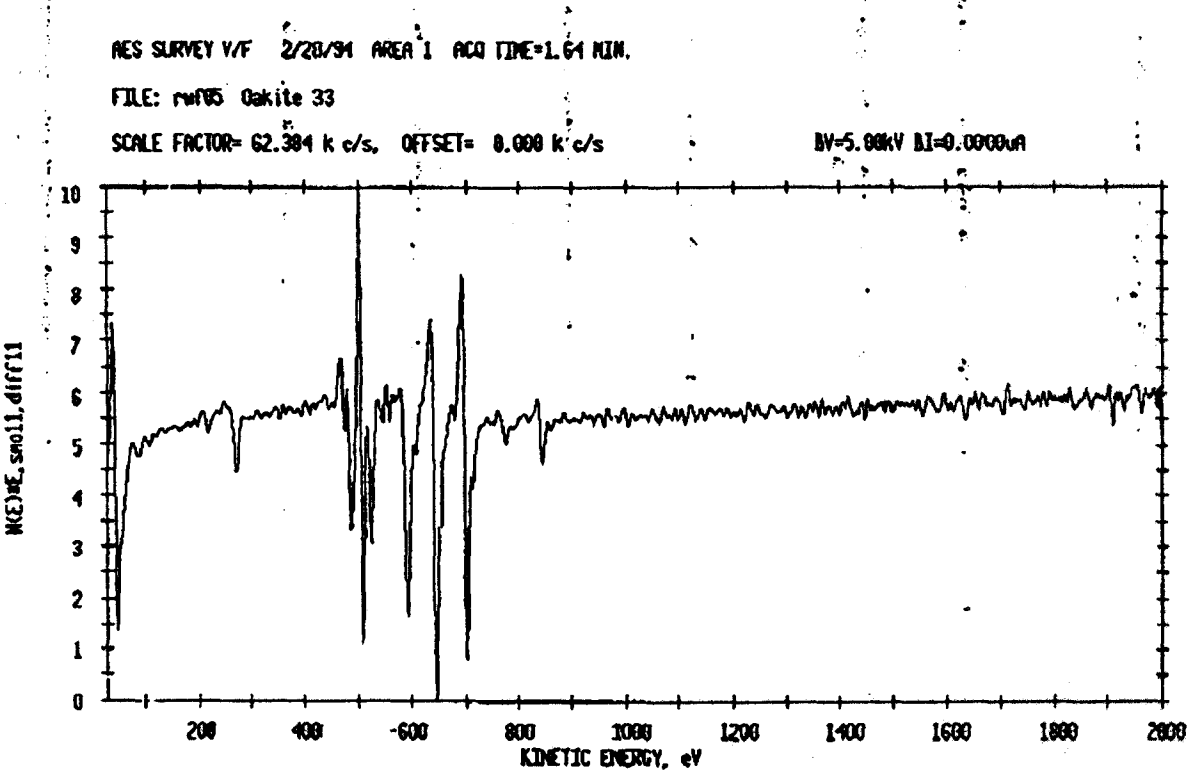


FIG 4

after 20 min



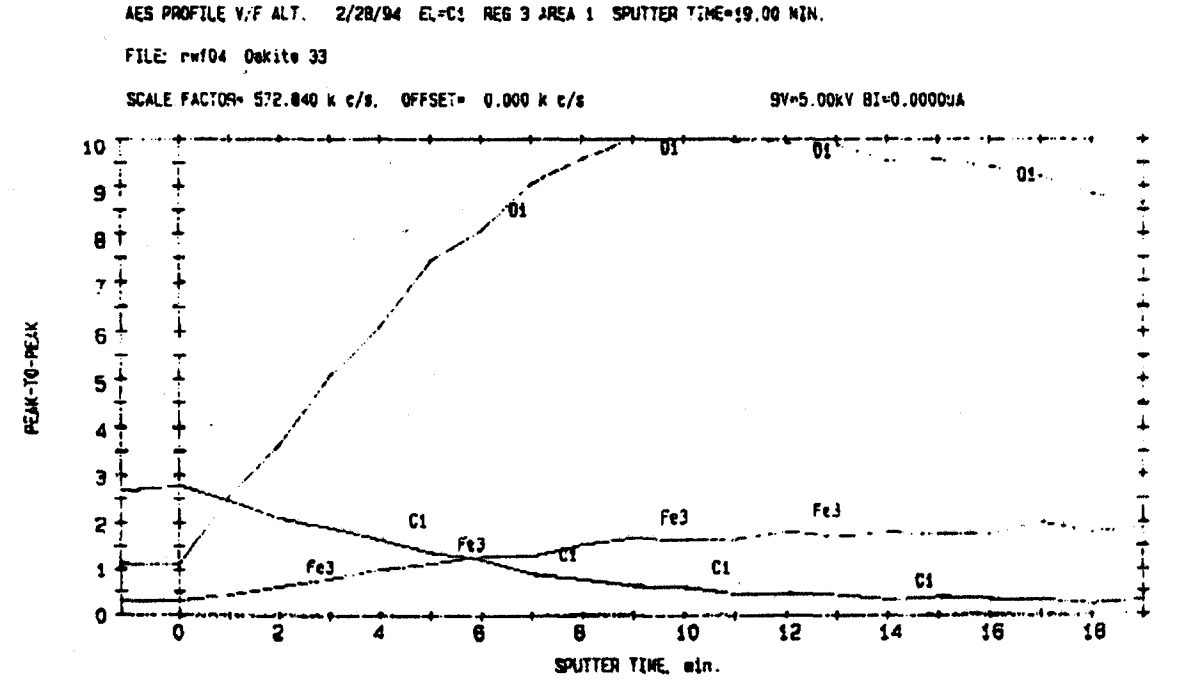
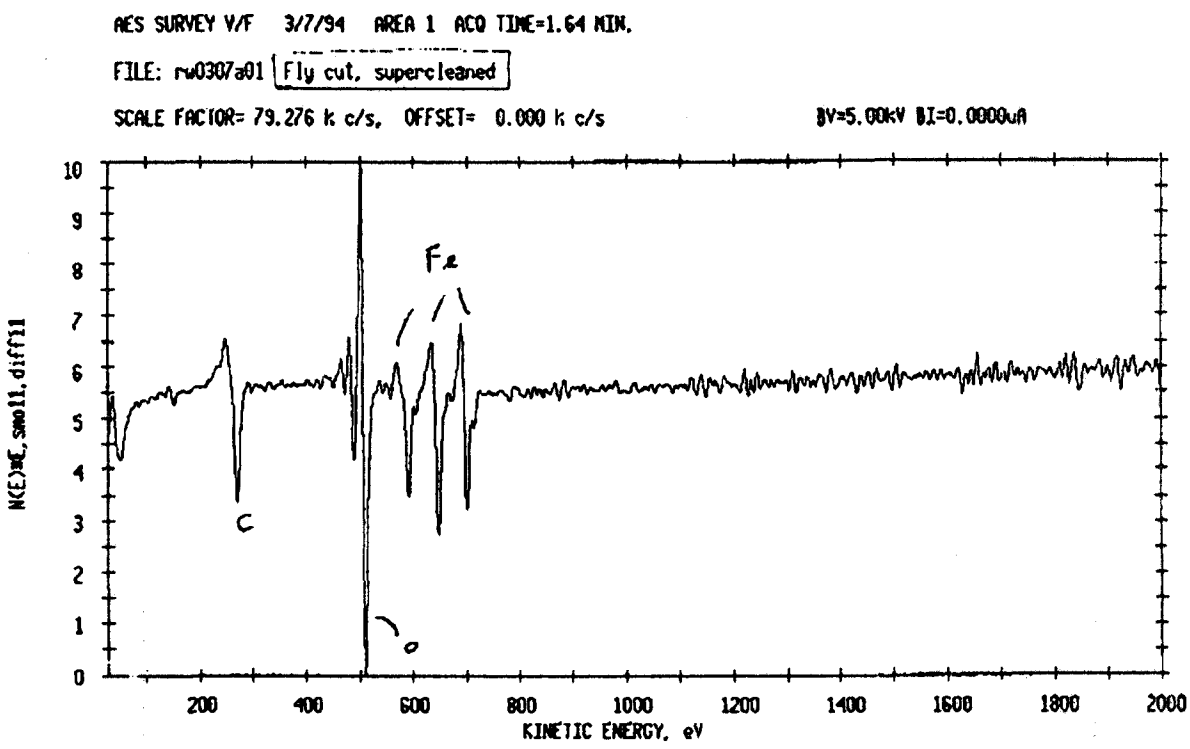


FIG 5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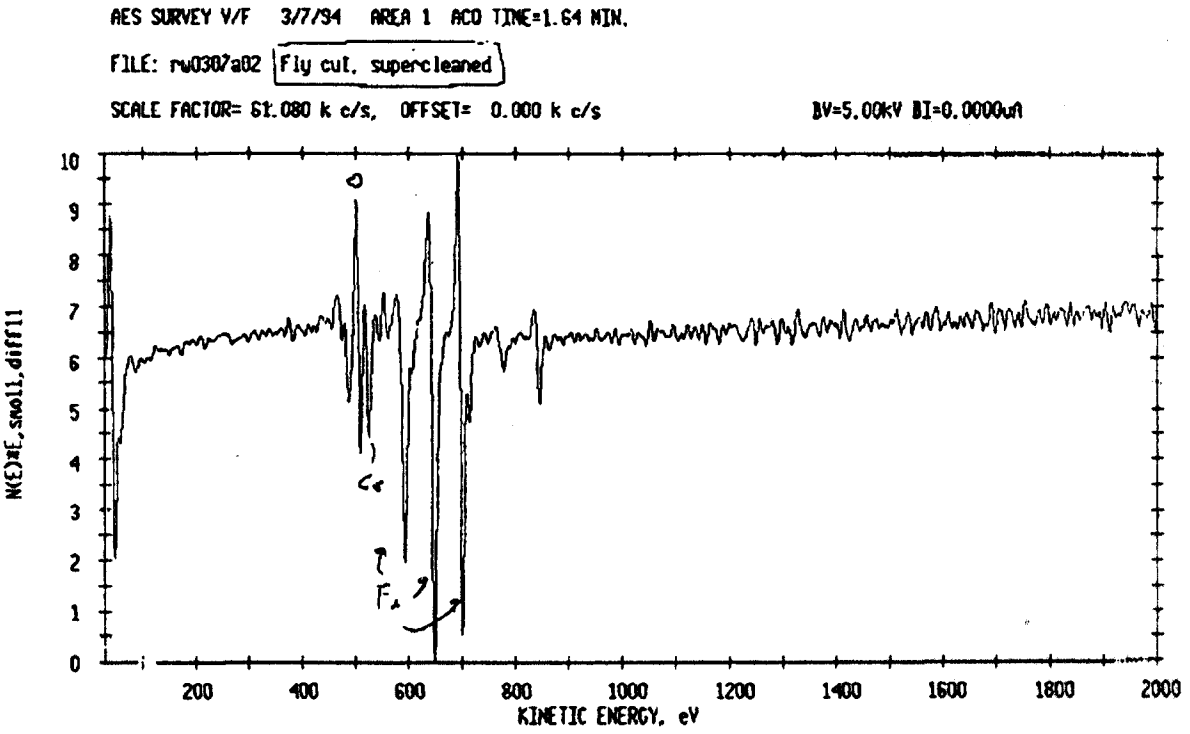
N(E)*E, smo11, diff11
KINETIC ENERGY, eV
Fe
C
O


AES SURVEY V/F 3/7/94 AREA 1 ACQ TIME=1.64 MIN.
FILE: rw0307a02
Fly cut, supercleaned
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BV=5.00kV BI=0.0000uA
N(E)*E, smo11, diff11
KINETIC ENERGY, eV
O
Cr
Fe

FIG 6

AES SURVEY V/F 11/2/94 AREA 1 ACQ TIME=1.64 MIN.

FILE: ru1102a2 CLN 112 spot 100ms 5K V

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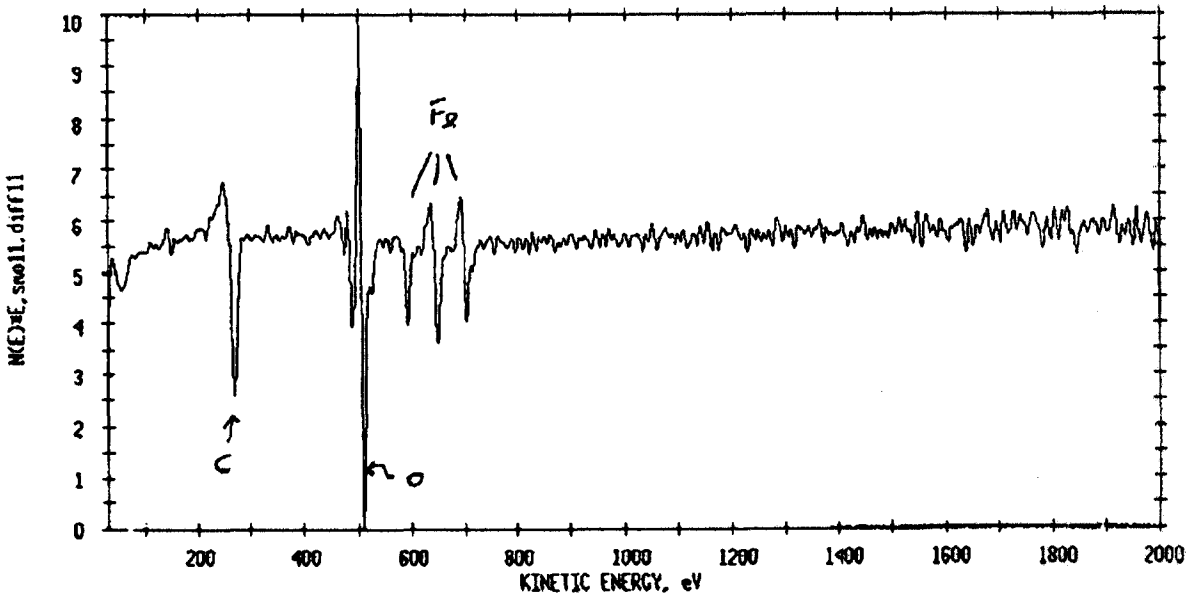

Spot After 1 minute of sputtering 5Kv 10μa/cm²

AES SURVEY V/F 11/2/94 AREA 1 ACQ TIME=1.64 MIN.

FILE: ru1102a4 CLN 112

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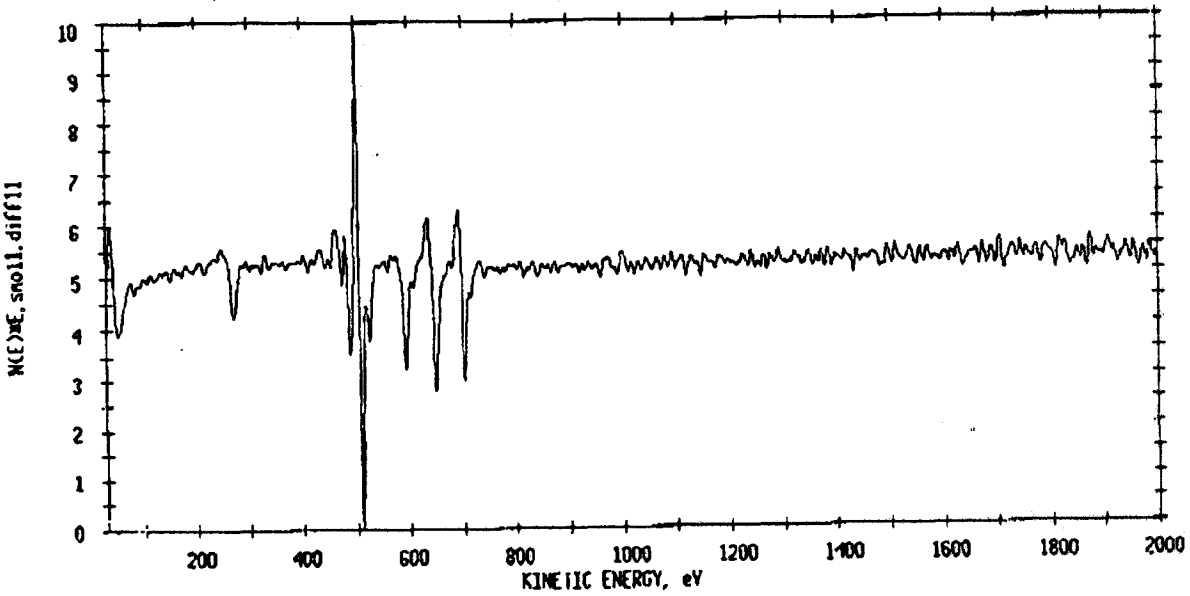

FIG 7

Sputtering at
2kV 25 μA/cm²
Spot

AES PROFILE V/F ALT. 11/2/94 EL=C1 REG 3 AREA 1 SPUTTER TIME=28.00 MIN.

FILE: rw1102a3 CLN 112

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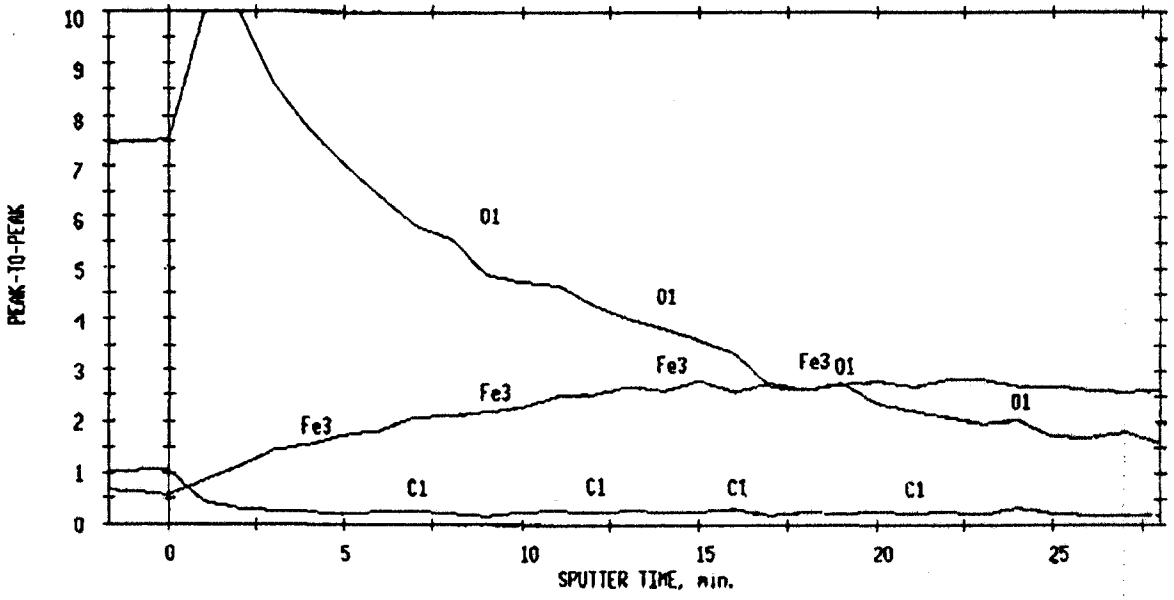
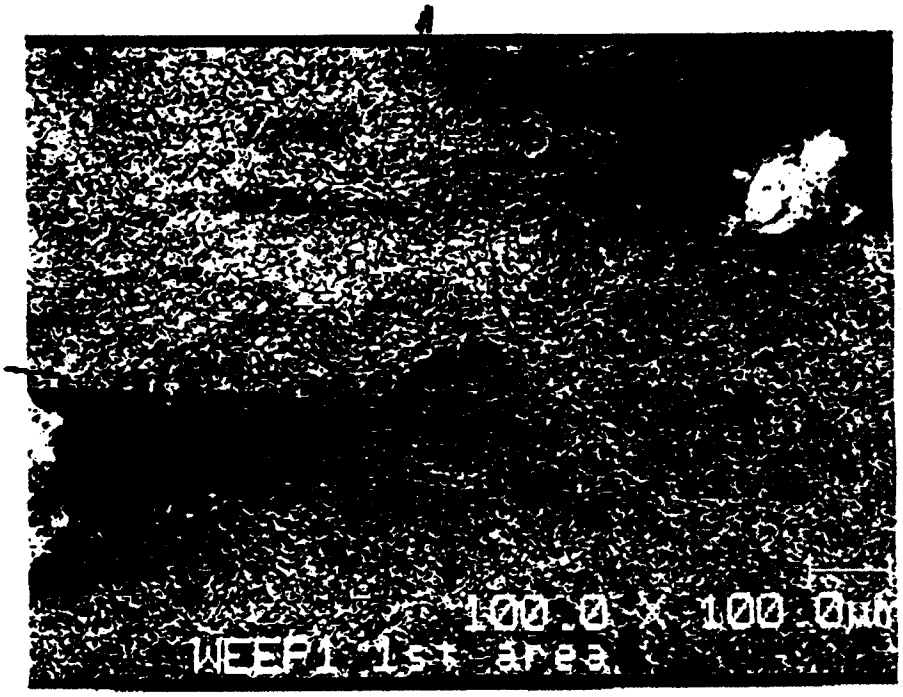
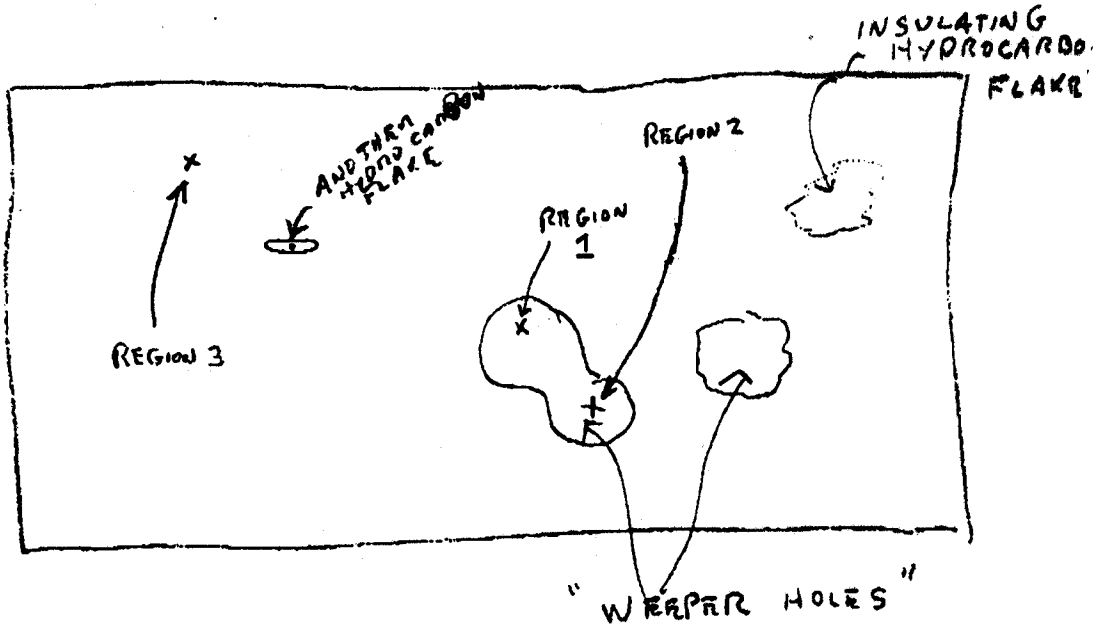


FIG 8 LOW RESOLUTION SEM PICTURE OF UNCLEANED SAMPLE WITH "WEEPER" SITES





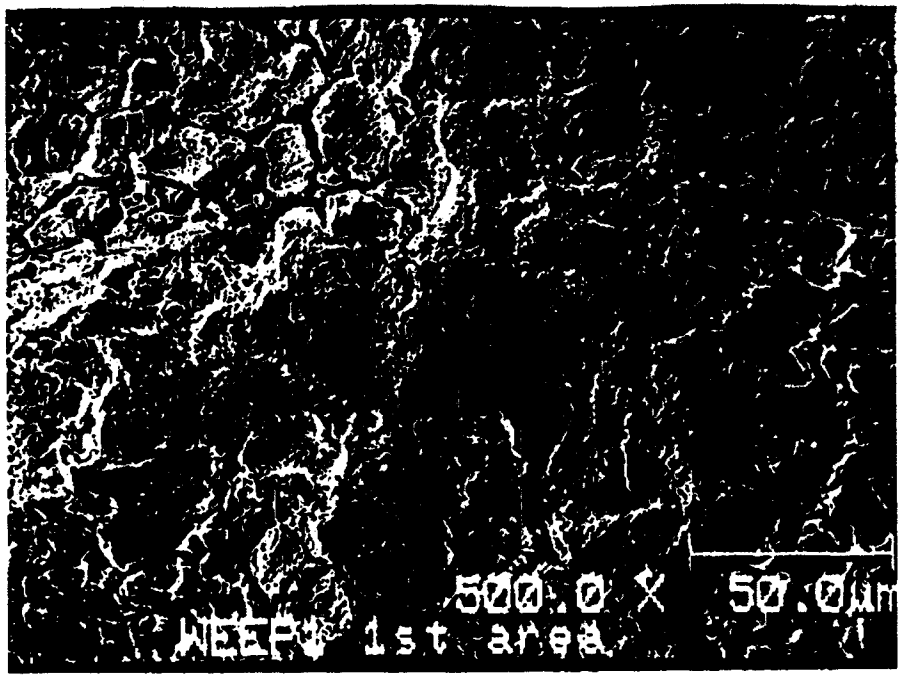


FIG 9 REGION 1 SEM
NOTE FLAT REGION

FIG 11 REGION 2 K-SOP →

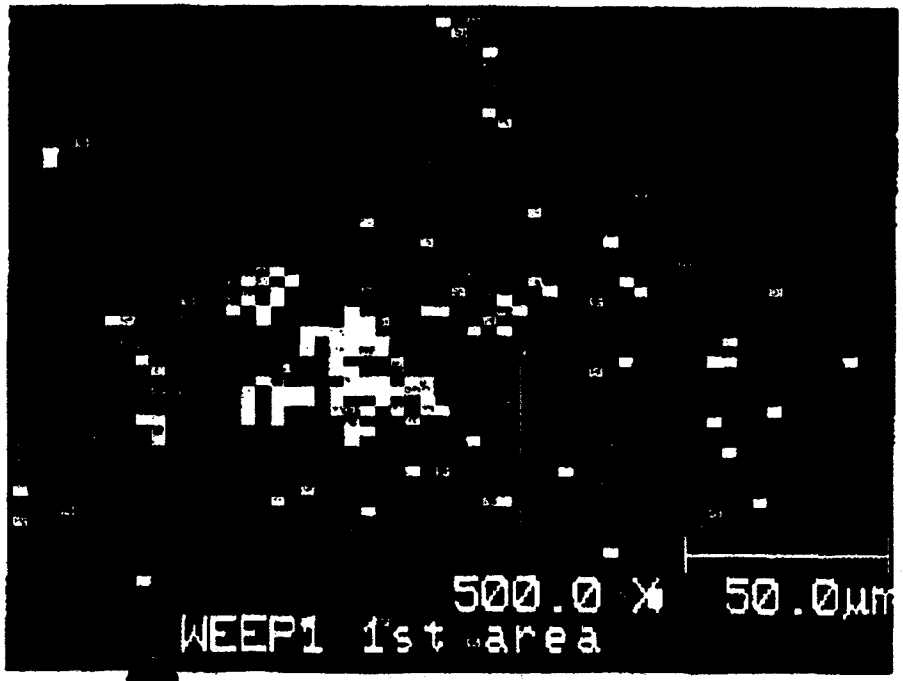


FIG 10 [illegible] MAP OF REGION 1

FIG 12

AES SURVEY V/F 11/3/94 AREA 1 ACQ TIME=1.64 MIN.

FILE: ru1103q1 NEEP1

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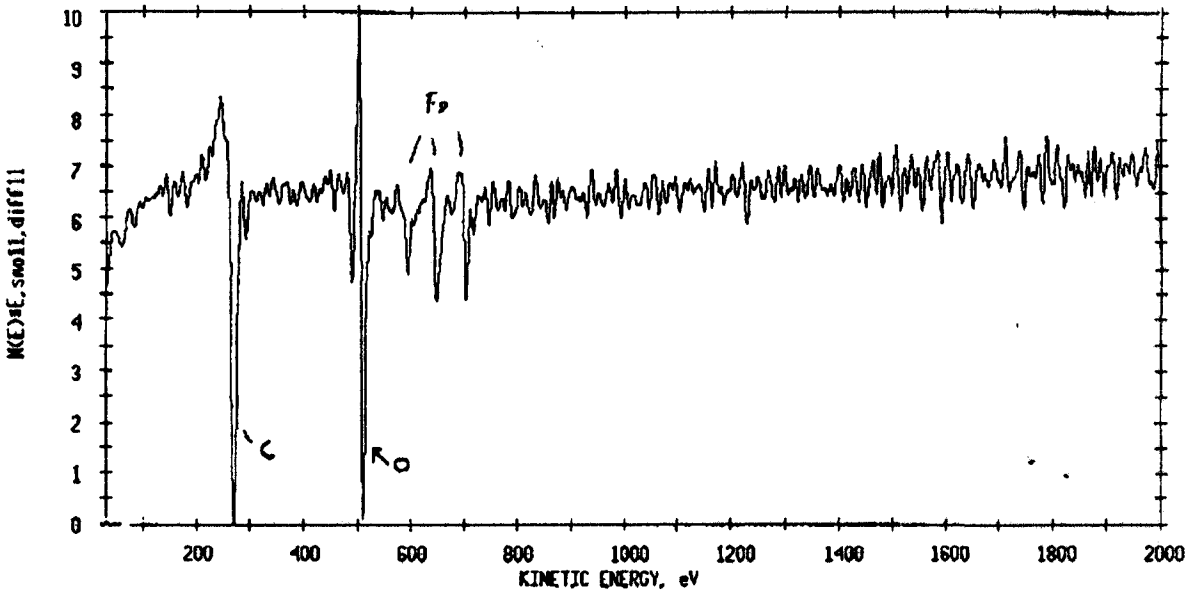

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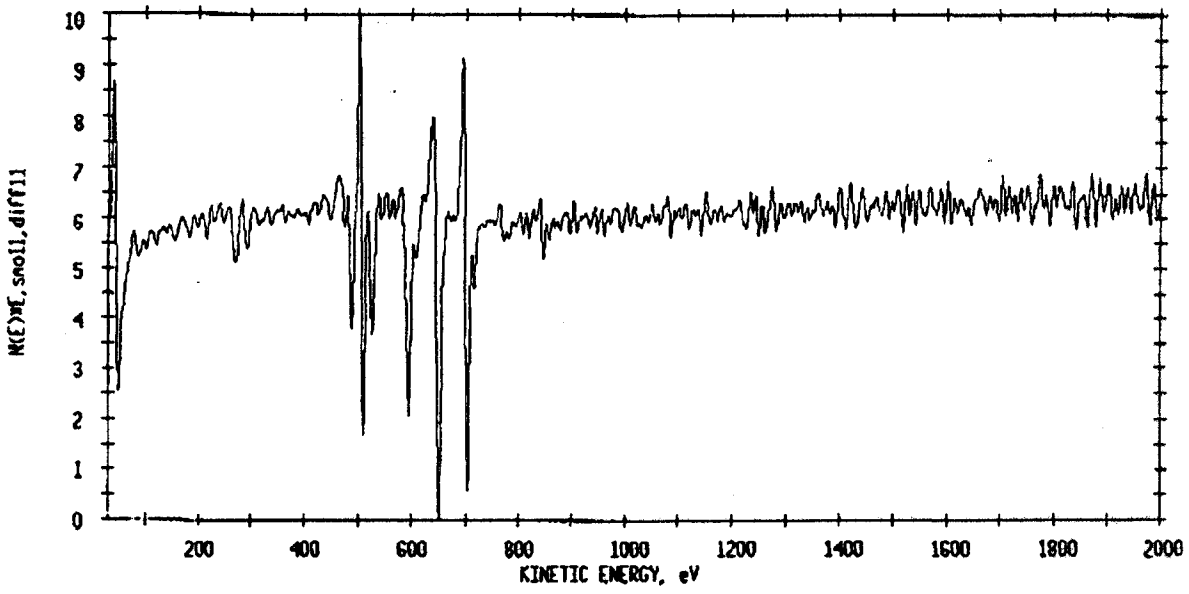

FIG 1

Before sputtering

AES SURVEY V/F 11/3/94 AREA 1 ACQ TIME=1.64 MIN.

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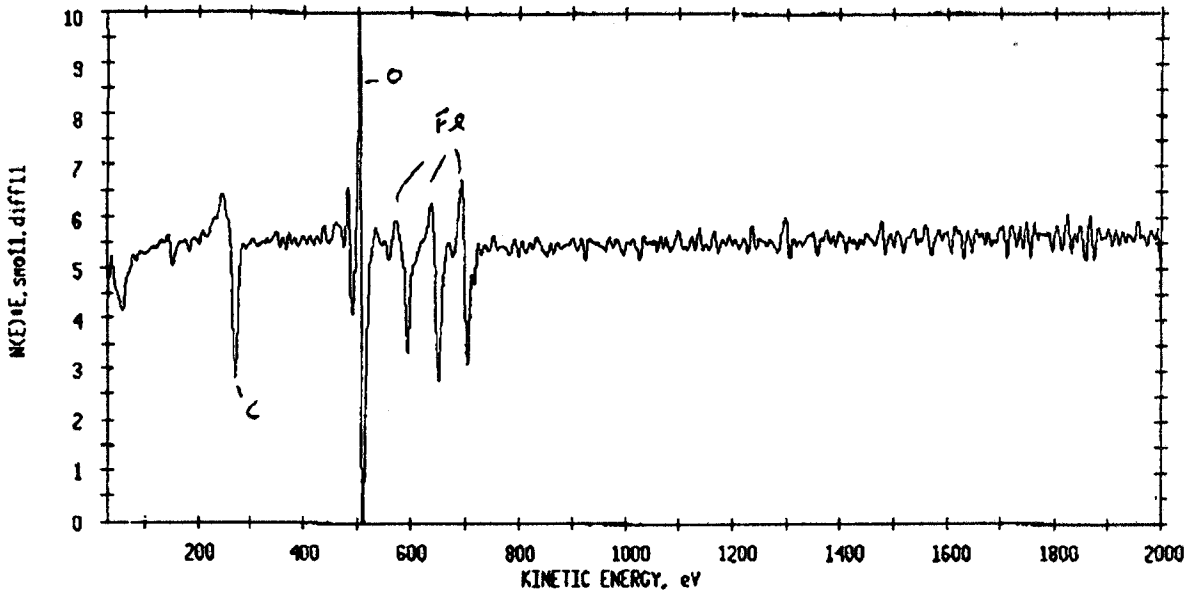

after 1 minute of sputtering

AES SURVEY V/F 11/3/94 AREA 1 ACQ TIME=1.64 MIN.

FILE: ru1103a6 KEEP1, second area

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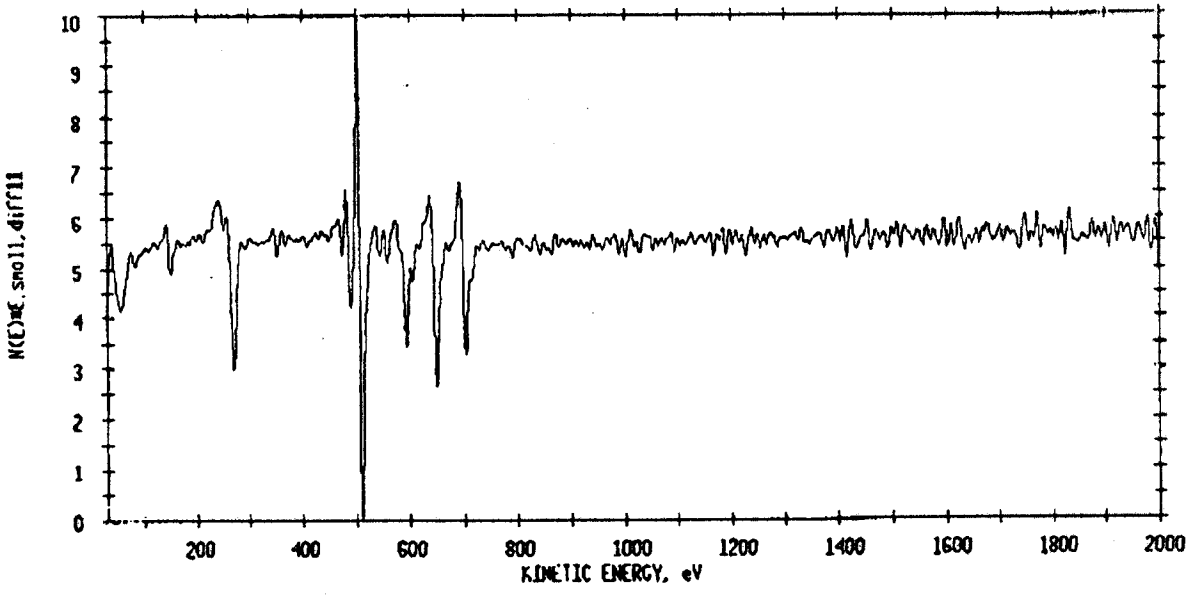


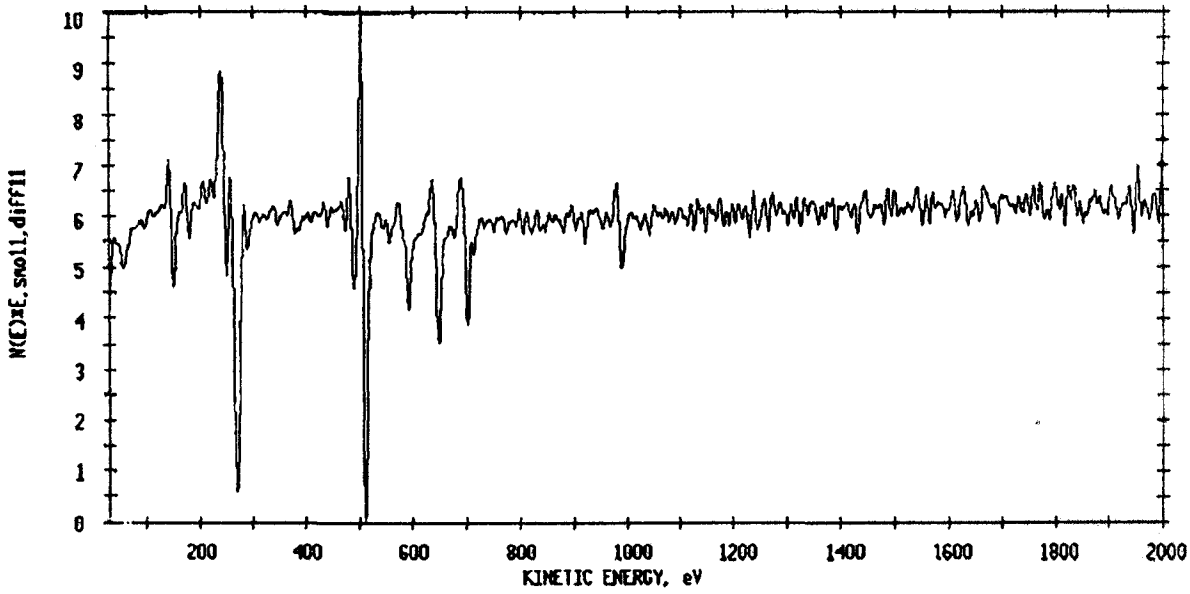
=16 14

Nb2 oxygen area

AES SURVEY V/F 11/3/94 AREA 1 ACQ TIME=1.64 MIN.

FILE: rw1103a8 KEEP 1, third area

SCALE FACTOR= 65.570 k c/s, OFFSET= 0.000 k c/s BV=5.00kV BI=0.0000uA

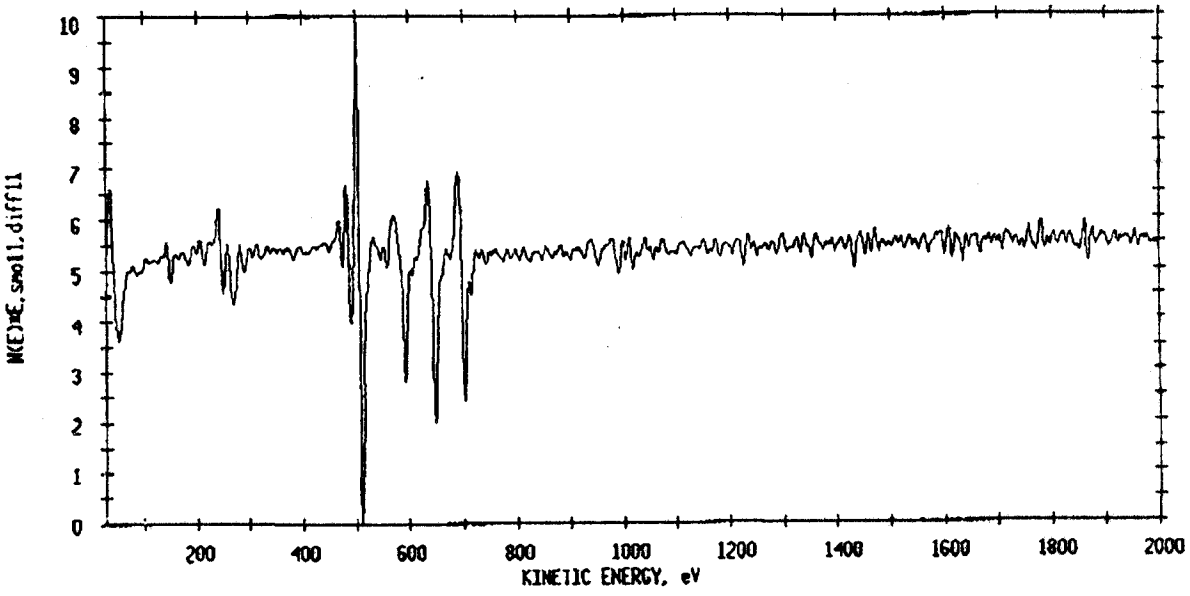


Sputter for 1 min

AES SURVEY V/F 11/3/94 AREA 1 ACQ TIME=1.64 MIN.

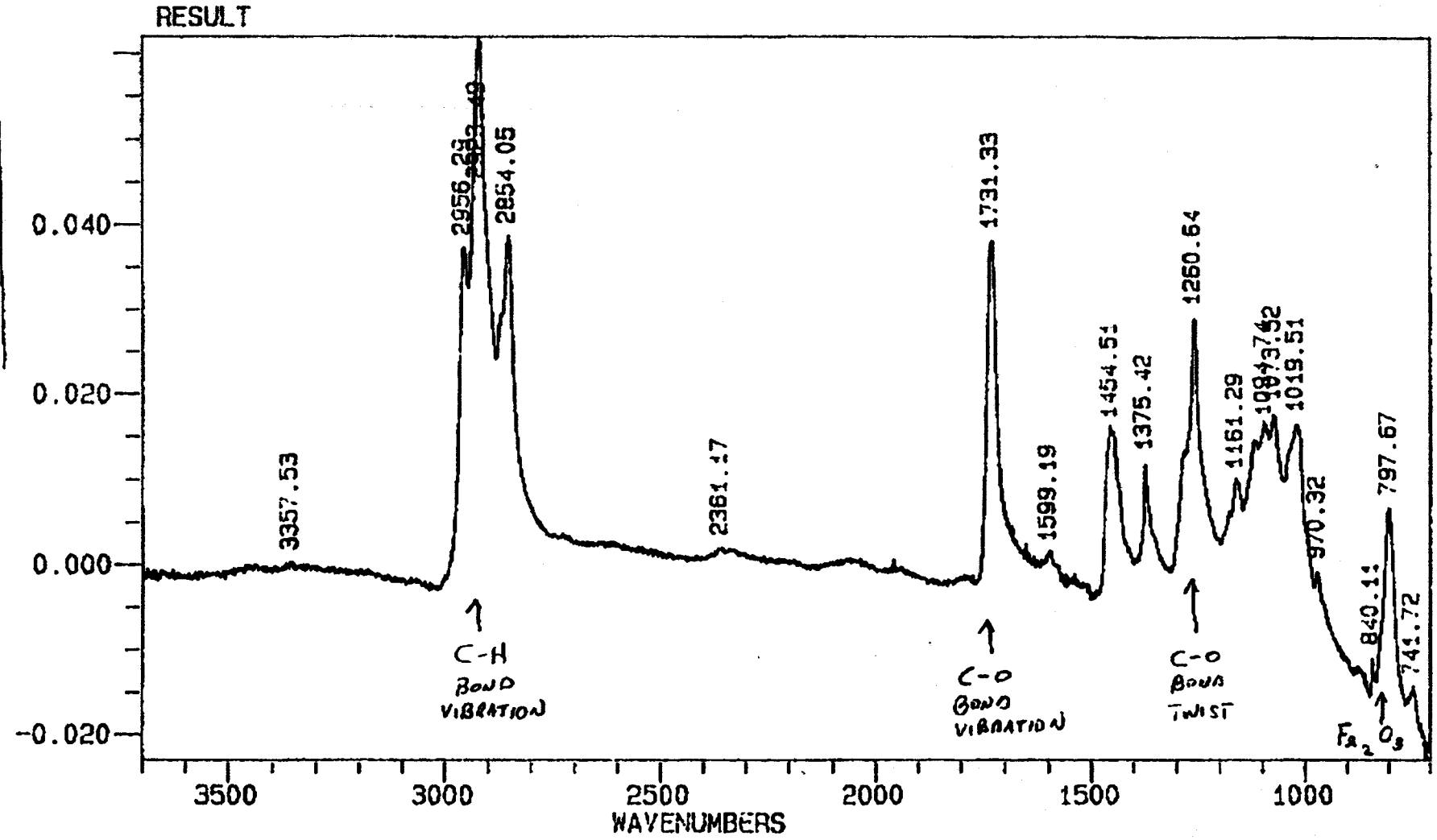
FILE: rw1103a10 KEEP 1, third area

SCALE FACTOR= 106.352 k c/s, OFFSET= 0.000 k c/s BV=5.00kV BI=0.0000uA



FTIR AT MIT

FIG 15

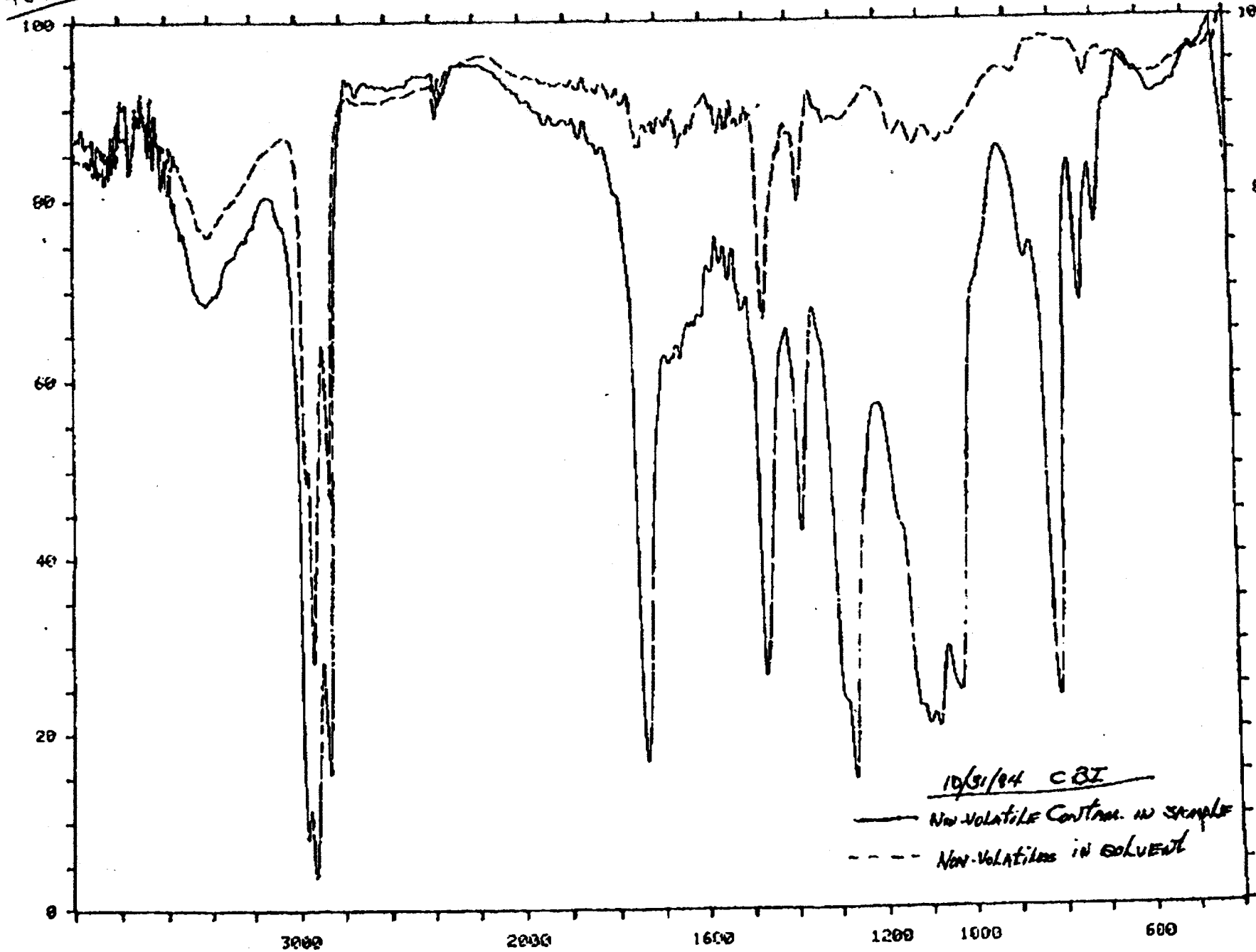


STRONG LINES → MATCHÉ SPECTRA OF:
NUJOL, MOTOR OIL, CASTOR OIL

Nicolet Instrument Corporation

SAMPLE IN THICKER AREA ON NaCl FROM "WEEPERS"
BASELINE CORRECTED
SCANS: 50 RES: 2.0 TIME: 09/24/94 12:20:38 FILE: SAMP3.IRD
cm⁻¹

FIG 16



FT.IR CBI