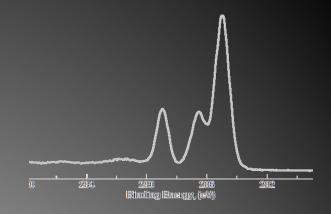
Monochromatic XPS Spectra

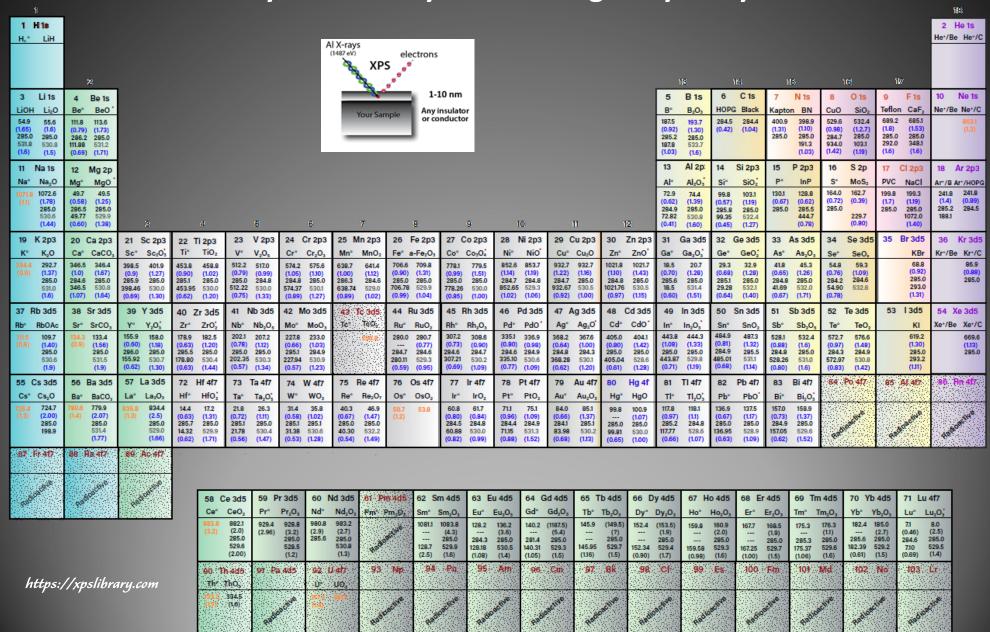


Polymers & Polymers Damaged by X-rays

B. Vincent Crist

XPS International, LLC https://xpslibrary.com

Monochromatic XPS Spectra Polymers & Polymers Damaged by X-rays



Handbooks of Monochromatic XPS Spectra

Volume 4 – *Polymers & Polymers Damaged by X-rays*

B. Vincent Crist, Ph.D.



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APPENDIX "A"



ALPHABETICAL LIST OF XPS SPECTRA IN VOLUME FOUR

POLYMERS & POLYMERS DAMAGED BY X-RAYS

<u>Section 1 – Polymers Purified by the National ESCA and Surface Analysis Center for Biomedical Problems</u>

α-methyl styrene (AMPS)	3
2-chloro ethyl methacrylate (CLMA)	7
2-hydroxy ethyl methacrylate (HEMA)	12
2-hydroxy ethyl methacrylate (HEMA) trimethyl-silane derivative	16
4,4'-dimethoxy benzophenone (4,4-DBP)	20
4-ethoxy styrene (PES)	
4-hydroxy styrene (PHS)	28
4-methyl styrene (PMPS)	33
4-octyl styrene (POS)	37
4-vinyl phenol – trifluoro acetic anhydride derivative	40
Biomer TM (2% in HFIP)	43
cellulose (Whatman™ filter paper) as received	48
dimethyl siloxane (PDMS) as received	51
ether ether ketone (PEEK) pressed wafer	58
ethyl acrylate (PEA)	63
ethyl methacrylate (EMA) film on glass	70
ethylene (PE) freshly pressed	74
ethylene glycol (PEG) 4000 flake	
ethylene terephthalate (PET, Mylar TM) 3X MeOH rinse	83
methyl acrylate (PMA) 3% solution	87
methyl methacrylate (PMMA) on copperfilm on disk	
methylene diisocynate/butane-diol copolymer (MDBD) 1:1 (urethane)	
methylene diisocynate/ butane-diol /propane-diamine terpolymer (MDBP) hard segment	103
methylene di-isocynate/propane-diamine copolymer (MDPD) 1:1	
propylene glycol (PPG) 2000 (from MeOH)	
styrene (PS) 4% in toluene	
tetra fluoro ethylene (PTFE) cleaned (Teflon TM)	
tetra methylene glycol (PTMG) cast from CHCl ₃	
vinyl alcohol (PVA) 2.5% water solution	
vinyl chloride (PVC)	
vinylidene di-fluoride (PVDF) old	140



Section 2 – Commercial Polymers & Polymers Damaged by Long Term (Overnight) Exposure to Monochromatic Aluminium X-rays

1-butene, isotactic (studied for X-ray induced damage) freshly exposed bulk	145
4-methyl-1-pentene (studied for X-ray induced damage) freshly exposed bulk	150
acetal (trioxane) (studied for X-ray induced damage) freshly exposed bulk	155
acrylic acid (no flood gun used to avoid flood gun damage)	163
acrylic acid (studied for X-ray induced damage) film on aluminium foil	168
acrylonitrile (PAN) (studied for X-ray induced damage) as received film	
amide resin (studied for X-ray induced damage) freshly exposed bulk	188
anti-static bag (used to store printed circuit boards, SECO Co.)	199
anti-static bag (used to store printed circuit boards, SEALPAK Co.)	206
bis-phenol carbonate (PC) (studied for X-ray induced damage) freshly exposed bulk	211
cellulose proprionate (freshly exposed bulk)	219
dimethylsiloxane (dimethyl silicone oil)	223
ethylene (HDPE) (studied for X-ray induced damage) freshly exposed bulk	228
ethylene (LLDPE) as received and after O ₂ plasma treatment	233
ethylene oxide (PEO) (studied for X-ray induced damage) film from CHCl ₃ solution	240
ethylene terephthalate (PET, Mylar TM) (studied for X-ray induced damage) freshly exposed bulk	248
ethylene tetra fluoro ethylene (ETFE) as received	257
Glad Wrap TM (as received)	262
Kapton TM (studied for X-ray induced damage) freshly exposed bulk	267
methyl methacrylate (PMMA) (studied for X-ray induced damage) film from CHCl ₃ solution	278
nitrocellulose (filter paper) (studied for X-ray induced damage)	286
Nomex TM (as received)	
Nylon 6 TM (caprolactam) (studied for X-ray induced damage) freshly exposed bulk	304
phenylene sulfide (PPS) (studied for X-ray induced damage) powder on adhesive tape	315
propylene (PP) freshly exposed bulk	
propylene (PP) as received	
propylene (PP) (studied for X-ray induced damage) freshly exposed bulk	331



Section 2 – Commercial Polymers & Polymers Damaged by Long Term (Overnight) Exposure to Monochromatic Aluminium X-rays

Saran Wrap TM (as received)	336
spider web (Black Widow, bunched together, but not sticky)	
styrene (PS) as received film	347
styrene (PS) (studied for X-ray induced damage) freshly exposed bulk	
styrene (PS) Gamma-ray treatment	
styrene (PS) oxidized (O ₂ plasma treatment)	
styrene (PS) oxidized and Gamma-ray treatment	
sulphone resin (studied for X-ray induced damage) freshly exposed bulk	
tetra-fluoro ethylene (PTFE) Teflon TM (studied for X-ray induced damage) thin film	
vinyl acetate (PVA) (studied for X-ray induced damage) freshly exposed bulk	
vinyl chloride (PVC) (studied for X-ray induced damage) beads on adhesive tape	
vinyl methyl ketone (PVMK) film on glass	
vinyl pyridine (as received, bead)	408
vinylidene di-fluoride (studied for X-ray induced damage) powder on adhesive tape	
7 inloc TM (interior surface)	421



INTRODUCTION

This handbook contains wide scan spectra, narrows scan spectra, and some valence band spectra from pure polymers and pure co-polymers. The . The polymer powders used for the X-ray Damage Study were 99% pure as sold by the Scientific Polymer Products Company. The Polymers are non-conductive. As a result the BEs in some of the spectra will be lower than expected due to charge shifting caused by flooding the sample with a low energy beam of electrons (1-10 eV). The correct BE for these samples depends on which reference energy the user prefers to use. We at XPS International prefer to assign the C (1s) BE of the hydrocarbon component to be 285.0 eV and to use this BE value as our means of charge referencing all the other signals generated by that binary oxide that was found to be non-conductive during analysis by XPS. Please refer to section "F" (Energy Scale Reference Energies and Calibration Details) for more details about calibration.

Volume 4 – Polymers and Polymers Damaged by Long Term Exposure to Monochromatic X-rays

Section 1 - Polymers from the National ESCA and Surface Analysis Center for Biomedical Problems (NESAC/BIO) at the University of Washington:

Includes wide scan spectra, high energy resolution carbon (1s) spectra, high energy resolution oxygen (1s) spectra, and other principal signal high energy resolution spectra (i.e. N (1s), F (1s), Cl (2p) S (2p), or others) known as: poly-ethylene (PE), poly-vinyl chloride (PVC), poly-vinylidene di-fluoride (PVDF), poly-tetrafluoroethylene (PTFE), poly-styrene (PS), poly-a-methyl styrene (AMPS), poly-4-methyl styrene (4MPS), poly-4-octylstyrene (POS), poly-4-hydroxystyrene (PHS), poly-4-hydroxystyrene-derviatized with trifluoro acetic anhydride, poly-4-ethoxystyrene (PES), poly-4,4'-dimethoxy benzophenone (4DBP), poly-ether ether ketone (PEEK), poly-ethylene terephthalate (PET), poly-ethyl acrylate (PEA), poly-methyle acrylate (PMA), poly-methyl methacrylate (PMMA) on copper, poly-ethyl methacrylate (EMA), poly-2-hydroxyl methacrylate (HEMA), poly-trimethyl silane hydroxyethyl methacrylate (CLMA), poly-ethylene glycol (PEG), poly-propylene glycol (PPG), poly-tetramethylene glycol (PTMG), poly-vinyl alcohol (PVA),

BiomerTM, poly-methylene di-isocynate/propane-diamine copolymer (MDPD), poly-methylene diisocynate/butane-diol copolymer (MDBD), poly-methylene diisocynate/propane-diamine/butane-diol terpolymer (MDBP), poly-dimethyl siloxane (PDMS) and WhatmanTM filter paper (cellulose)

Section 2 - Polymers Damaged by Long Term Exposure to Monochromatic X-rays:

Includes wide scan spectra, and >15 repetitive cycles of high energy resolution carbon (1s) spectra, high energy resolution oxygen (1s) spectra, other principal signal high energy resolution spectra (i.e. N (1s), F (1s), Cl (2p) S (2p), or others) known as: poly-acetal, poly-acrylonitrile (PAN), poly-amide, poly-1-butene, poly-caprolactam (nylon 6), poly-carbonate bis-phenol A (PC), poly-ethylene (HDPE), poly-ethylene oxide (PEO), poly-ethylene terephthalate (PET), poly-imide (Kapton), poly-methylmethacrylate (PMMA), poly-4-methyl-1-pentene, poly-phenylene sulfide (PPS), poly-propylene (PP), poly-styrene (PS), poly-sulfone resin, poly-tetrafluoroethylene (PTFE), poly-vinyl acetate (PVA), poly-vinyl chloride (PVC), poly-vinylidene fluoride, and nitrocellulose. Each sample was analyzed overnight for >12 hours to observe the X-ray induced changes that occur from extended exposure to X-rays.



ORGANIZATION AND DETAILS OF SPECTRAL SETS

Organization of Spectra

A set of spectra for a particular chemical is located by looking for the chemical formula abbreviation written in the upper right hand corner of each page. For the binary oxide called "aluminium oxide" the user will find the chemical abbreviation " Al_2O_3 " in the upper right corner of the pages that belong to that set of data and spectra. The spectra are organized by using the chemical abbreviation. This means that "Antimony Oxide" spectra will be found by looking for the chemical formula: " Sb_2O_3 ".

Contents of Each Set of Spectra

The spectra are presented exactly as printed by the Spectral Data Processor software provided in each XI SpecMaster Data-Base system. The first page of a set includes the "Detailed Surface Composition Table" which reports the peak assignments, binding energies, relative sensitivity factors, and Atom % abundance of each major signal contained in the wide scan survey spectrum for that chemical. In the title line of this first page the user will find the full chemical name along with other basic information about the chemical, such as Formula Weight, Chemical Abstract Services number, common name, and a few key words about the analysis conditions.

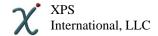
The second page of each set is the wide scan survey spectrum with information about the experimental conditions used to collect the spectrum, the history of the sample, the source of the sample if known, and peak labels. Detailed information about the operating capabilities of the SSI systems and the instrument and analysis conditions used to collect these data are presented in the next section of this book.

The remaining pages of each set are the high energy resolution narrow scan spectra which were obtained by measuring the strongest signals found in the wide scan survey spectrum. These spectra normally contained detailed peak-fit results in a table and display the actual peak-fit results for each spectrum. The binding energies of insulating materials are reported without any charge correction because there is currently no standard method or standard reference energy for charge referencing spectra from insulating materials. The FWHM values for each peak of a high energy resolution spectrum is adjacent to the binding energy for that peak. The percentage numbers given for each peak is a relative percentage that is based on the intensity of that signal only (It is not an atom % value).

In this edition the spectra do not have labels which identify the XPS signal so the reader needs to refer to the energy range to determine which signal has been reported. In many cases valence band region spectra, Auger signal spectra, and spectra from weaker XPS signals are also included, but only for materials which were expected to be commercially pure.

Philosophy of Data Collection Methods

Our philosophy is to collect spectra under analysis conditions that are practical, readily reproduced, and typically used in laboratories that use monochromatic X-ray sources and work under real world practical analysis conditions. We have assumed that the most XPS laboratories need practical reference spectra and will not spend the time or money to produce and to analyze pure, clean surfaces under ultimate energy resolution



conditions. However, we did spend extra time to collect data with above average signal to noise (S/N) ratios which reveal the presence of minor components that might otherwise be missed. In the production of these spectra we did not attempt to produce clean surfaces which would make charge referencing of insulators a difficult task. For practical reasons we used the C (1s) spectra from the naturally formed layer of adventitious hydrocarbons because that signal is the "de facto" standard for charge referencing insulating materials.

The spectral data contained within these handbooks are designed to assist engineers, scientists, analysts, theoreticians, and teachers who use XPS on an everyday basis under practical working conditions. We believe that these spectra will help XPS users to analyze industrial problems, gather reference data, perform basic research, test theories, and teach others. Our spectra are designed to be practical tools for everyday use and were obtained under practical working conditions. We have not actually attempted to produce research grade spectra, but we have, in fact, produced research grade spectra because of our self-consistent methods.

In the production of these spectra no attempt to produce a pure, clean surface, but an effort was made to produce surfaces with a minimum amount of natural surface contamination. When ion etching was used to clean a material that contained more than one element, then ion etching was done with conditions that should minimize preferential sputtering.

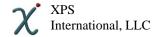
Peak-Fitting (Curve-Fitting) of High Energy Resolution Spectra

Peak-fitting was performed by using the software provided with the Surface Science Instruments XPS system. This software allows the user to control full width at half maxima (FWHM) value of any peak, the binding energy (BE) of any peak, peak areas, the ratio of two peak areas, the energy difference between two peak maxima, the shape of a peak as a sum-function of Gaussian and Lorentzian peak shapes in any peak, and the percentage of asymmetry in any peak...

By empirically peak-fitting the spectra from large sets of closely related materials in a trial and error method and analyzing the trends, it was possible to recognize several fundamental peak-shape and peak-fitting parameters for pure elements, binary oxides, polymers, and semiconductors. We used those empirical results to guide our efforts to peak-fit many of the spectra which had complicated peak shapes. In some cases we used the theoretical ratio of spin-orbit coupled signals to assist the peak-fitting of many spectra and also the energy interval between spin-orbit couple signals which were derived from pure element spectra. No attempt was made to fit the spectra in accordance with theoretical expectations or calculations.

The reduced "chi-squared" value, which indicates the goodness of a peak-fit, was used to determine if a peak-fit was reasonable or not. Based on practical experience a "chi-squared" value between 1 and 2 implies a relatively good peak-fit. A "chi-squared" value between 2 and 4 implies that the fit has not yet been optimized. A "chi-squared" value larger than 4 implies that one or more signals may be missing from the peak-fit effort.

A Shirley-type baseline was used for most peak-fits. Peak shapes for the main XPS signals from chemical compounds (e.g. oxides or polymers,)were typically optimized by using a Gaussian:Lorentzian ratio between 80:20 and 90:10. For pure metals Gaussian:Lorentzian ratio for the main XPS signals was normally between 50:50 and 70:30. For the main XPS signals from semi-conductor materials, the Gaussian:Lorentzian ratio was usually between 70:30 and 80:20.



From the peak-fitting of the binary oxides, we have observed that FWHM for the C (1s), O (1s) and the main metal signal from the binary oxide are usually in range 1.0-1.4 eV. This trend helped us to decide if we had good charge compensation.

Charge Compensation of Insulating Materials

Charge compensation of insulating materials was normally handled by using the patented SSI mesh-screen together with a low voltage flood gun of electrons which used an acceleration voltage that was adjusted to 2-4 eV for optimum results. The mesh-screen device uses a 90% transmission electro-formed mesh made of nickel metal that is supported above the surface of the sample by mounting the mesh on a conductive metal frame that is grounded to the sample mount. To achieve good charge compensation the mesh-screen is positioned so that the distance between the mesh and the surface of the sample is between 0.5 - 1.0 mm. When the distance between the mesh-screen and the surface of the sample is greater than 1.2 mm, the usefulness of the mesh screen flood gun system was null.

The mesh-screen is understood to function as a electron cut-off lense with some tendency to allow incoming flood gun electrons to focus on the area being irradiated with monochromatic X-ray beam because the X-ray beam does not have a uniform flux density of the area of the beam. In effect, the mesh-screen produces a nearly uniform electric potential at the surface of the sample and allows incoming flood-gun electrons to pass through whenever they are needed.

The mesh-screen was used on every insulating material except for a few materials that were analyzed before the mesh-screen method was developed.

Abbreviations Used

Due to the limited space provided to describe each sample in each electronic data-file, it was necessary to use various abbreviations. The abbreviations are:

scr = screen used for charge compensation

scrn = screen used for charge compensation

TOA = take-off-angle for the electrons

Aldr = Aldrich Chemical Co.

RMC = Rare Metallics Co.

SPP = Scientific Polymer Products Co.

MS Co. = Metal Samples Company

FG = flood gun,

1mm = 1 mm height used for the mesh-screen,

semi-con = semi-conductive behavior

conduc. = conductive behavior

Tech = technical grade purity,

Tech = technical grade purity,

pellet = sample pressed into pellet form by pellet press used

pel = pellet

plt = pellet



INSTRUMENT AND ANALYSIS DETAILS USED TO MAKE XPS SPECTRA

A. Instrument Details

Manufacturer: Surface Science Instruments (SSI)

Model: X-Probe

S-Probe (upgraded from M-Probe model 2703)

Software Version; 1.36.05 (Compiled in MS-DOS "C" version 6.0)

Analyzer Type: Fixed Analyzer Transmission (FAT)

Fixed (Constant) Pass Energy = Constant Analyser Energy (CAE)

180° Hemi-spherical (truncated)

Input Lens Field of View: 30° for sample normal to lens axis (1" diameter port)

(always larger than X-ray beam; retarding potential scanned) Al $^{\circ}$ monochromatic (one 2 " diameter thin natural SiO2

crystal wafer glued onto Zerodur substrate heated to 65° C)

X-ray kV and mA Emission: 10 KV, 1.5-22.0 mA (depending on spot size used)

X-ray Energy Defined as: 1486.7 eV (8.3393 Å), Bragg Angle=78.5°

Excitation Source Window: 0.6 μ aluminum in S-Probe (10μ mylar in X-Probe)

Angle of X-ray Incidence: $\alpha = 71^{\circ}$ (relative to sample normal) Electron Emission Angle: $\beta = 0^{\circ}$ (relative to sample normal)

Angle Between X-ray Axis and Electron Analyzer Axis: $\phi = 71^{\circ}$ (fixed, non-variable)

X-ray Type:

Pass Energy of Analyzer: 150 V for Resolution 4 setting

100 V for Resolution 3 setting 50 V for Resolution 2 setting

25 V for Resolution 1 setting

Type & Size of Input Slit: Fixed (2 mm X 35 mm); magnetic compression

Type & Size of Output Slit: None (dispersion limited by hemisphere voltages)

Electron Collection Lens Field of View: $\sim 1 \text{ mm}^2 \text{ for } b = 0^\circ \text{ at } 1000 \text{ eV KE}$

Electron Collection Lens Efficiency: 7% over 2π steradians

Sample Surface to Tip of Electron Collection Lens Distance: ~33 mm

X-ray Crystal to Sample Surface Distance: ~190 mm X-ray Crystal to X-ray Anode Distance: ~190 mm



True Background Count of Noise: <10 electrons/second at -50 eV (shot noise limited)

> Detector Type: SSI Position Sensitive Detector, resistive anode, 40 mm X 40 mm

electronically defined as 128 active channels with max ct rate 1,000,000 Dead Time:

normally zero (unless ion etching pure element while collecting XPS data) 4. x 10⁻¹⁰ torr

Base Pressure: 1.6 x 10⁻⁹ torr Normal Operating Pressure:

~0.25 eV FWHM of X-rays Diffracted by natural SiO2:

> Power Settings: 200 Watts in a 250 x1100 μ X-ray beam 100 Watts in a 150 x 800 μ X-ray beam

45 Watts in a 80 x 350 μ X-ray beam 15 Watts in a 40 x 250 µ X-ray Beam

X-ray Induced Current: 1.1 x 10⁻⁹ amps for a 600 μ spot in X-Probe Converted from amps to watts

Approximate True X-ray Power: ~6 x 10⁻⁶ W in a 600 µ spot

Approximate True X-ray Irradiance: $\sim 8 \text{ W/m}^2$

~7 x 10⁹ photons/sec Approximate True X-ray Photon Flux:

Experimental Details В.

Electron Take-Off-Angle: 90° relative to sample surface (unless otherwise reported)

Pass Energies Used: Wide scans were done at PE = 150 eV

Narrow scans were normally done at PE = 50 eV

Valence band scans were done at PE=150 eV

X-ray Beam Size Used: Wide scans: 250 x 1500 µ ellipse (at 90° TOA)

(for S-Probe) 250 x 1100 μ ellipse (at 35° TOA)

> Narrow Scans: 250 x 1500 μ ellipse (at 90° TOA)

> > 150 x 1000 μ ellipse (at 90° TOA)

SSI Mesh-Screen: A 90% transmission (20 μ diameter wire with 200 μ

spacing) nickel metal mesh screen was adhered to a small 25 mm x 25 mm x 1.5 mm (W x L x H) aluminum plate over a 20 mm x 20 mm aperture. This mesh-screen was placed over all oxide samples so that the distance between the sample surface

and the mesh-screen was <1.0 mm but >0.3 mm.

200 milliseconds/channel (usual setting) Dwell Time (counting time):

> Data Transfer Time: 4 milliseconds

Max. Number of Channels: 5000 (channels = data points)

Scan Time for One Wide Scan: ~ 3.5 minutes (using 1024 data points) Scan Time for One Narrow Scan: ~100 seconds (using 256 data points)



Energy Range: -100 to +1400 eV (BE range)

Typical Step Size: 0.1 eV/step (i.e. 0.1 eV/data point)

C. Data Processing Details

Baseline Subtraction: None, unless S/BG gave a small display. When the

baseline was removed, the intensity of the lowest

point was subtracted from all points.

Data Smoothing: None Energy Shifting: None Intensity Scaling: None

D. Sample Details

The "Description" given on each XPS spectrum reports the empirical elemental formula for the oxide, purity, source, production lot number, a note, if appropriate, about being conductive or semi-conductive, the abbreviation "scrn" which means that the SSI mesh-screen was used, and a number, e.g. 90 which reports the electron take-off-angle used to collect the data for that sample. Abbreviations used in the description and their full meaning include: Aldr = Aldrich Chemical Co., RMC = Rare Metallics Co., semi-con = semi-conductive behavior, scrn = SSI mesh-screen used, TOA = electron Take-Off-Angle, Tech = technical grade purity, pellet = sample pressed into pellet form, plt = pellet, pel = pellet, MS Co. = Metal Samples Company in Munford, Alabama USA (Tel 205-358-4202), SPP = Scientific Polymer Products Inc. in Ontario, New York state, USA (Tel 716-265-0413)

Sources of Elements and Chemical Compounds Used for Element Series

The pure element samples were obtained from various sources without any specific information about sample purity so pure element samples must be assumed to be pure at the 99% level. The "halide" salts used to produce spectra from gaseous or highly reactive elements were also obtained from various sources. These halide samples were obtained as crystalline "windows" which are normally used in Infrared spectroscopy and have purities at the 99% level. The Boron Nitride (BN) sample was a white ceramic standoff which was fractured in air. The copper foil material, which was always used to determine reference energies, were obtained as 99% pure foil which was designed as a multiple purpose foil for use around the home. The gold ingot material, which was also used to determine reference energies was obtained as a 99.999% pure sample from Aldrich Chem. Co..

Source of Polymer Materials

A special kit (#205) of the 100 polymer materials was obtained from Scientific Polymer Products, Inc. which is located at 6265 Dean Parkway, Ontario, New York, USA 13519 (Tel 716-265-0413).



Source of Alloys

A special kit of 54 metallic alloys was obtained from the Metal Samples Co., which is located at Route #1, Box 152, Munford, Alabama, USA, 36268 (Tel 205-358-4202). This kit includes a materials analysis report on each alloy in weight percents. The National Research Institute for Metals in Tsukuba Japan has provided a series of various binary alloys made of AuCu and CoNi alloys.

Sources of Semi-Conductor Materials

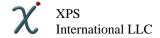
Over the course of many years, many people in the Japanese semi-conductor business have given samples of various semi-conductor materials in crystalline wafer form. Various samples were donated by the Oki Electric Company, Mitsubishi Materials, Canon, and various universities. The source of each material is included with the individual sample descriptions whenever that information was provided.

Sources of Binary Oxide Samples

Most of the commercially pure binary oxides were purchased from the Aldrich Chem. Co.. Many packages from the Aldrich Chemical Co. included an "Analytical Information" sheet which described an ICP or AA analysis summary, a production lot number, the Aldrich product number, sample purity number (e.g. 99+%), sample appearance (color and physical form), date of chemical analysis, formula weight and a label on the bottle that reports the melting point, toxicity, Chemical Abstracts registry number and density. The samples from Aldrich were generally quite pure at the surface. Other oxide samples were obtained from either Cerac Inc. (USA) or Rare Metallics Co., Ltd. (Japan). The packages from Cerac Inc. included a "Certificate of Analysis" with an ICP or AA analysis summary, a production lot number, a product number, purity (e.g. 99+%),and mesh size. The packages from Rare Metallics Co. did not include analytical data reports, but instead had stock numbers and a purity statement. Two samples (i.e. SiO2 natural crystal and Al2O3 fused plate) were obtained from in-house sources and do not have any purity reports.

Powdered Samples Pressed into 3mm Diameter Pellet

Until analyzed, all finely powdered samples were kept stored in their original glass or plastic containers, which were packaged inside of plastic-lined aluminum bags. Just prior to XPS analysis, each bottle was opened in the normal air of the room where the XPS system was kept, and a small 50-100 mg portion of the sample was removed via a clean nichrome spatula and placed in the compression chamber of a hand-operated, stainless steel pellet press. All finely powdered samples were compressed without any chemical treatments, which, if done, may have introduced unusual contamination or produced some change in the samples. The resulting pellets varied in thickness from 0.3 - 0.8 mm. To avoid iron and /or chromium contamination from the anvil, a thin sheet of paper was placed over the sample in the compression chamber. Any powders, which were clumped together, were very gently pressed into a powder just prior to compression. To avoid unnecessary heat-



induced oxidation, those samples which were hard and granular were very gently ground into a fine powder in a agate marble mortar and pestle. As soon as each sample was removed from the compression chamber, it was mounted onto silver (Ag°) paint inside of a 5mm wide round brass boat which was 1.3 mm in height. Silver paint was used so that conductive oxides could behave as true conductors thereby providing true electron binding energies for those oxides that were indeed conductive. In general, each oxide was exposed to room air for <15 min.

Benefits of Pressing Powders into Pellets (increased counts and simple charge control)

A comparison of the electron counts obtained from powdered samples pressed onto double-sided adhesive tape and positioned at a 35° electron take-off-angle with the electron counts obtained from hand-pressed glossy or semi-glossy pellets positioned at a 90° electron take-off-angle (TOA) revealed that a pellet at a 90° electron TOA produces 3-5 times higher electron counts than a powdered sample pressed onto double-sided tape at a 35° electron TOA.

By pressing the finely powdered oxides into pellets, it was also found the surface charging behavior of these glossy or semi-glossy samples was very easy to control by using the mesh-screen electron flood-gun combination with the flood gun set to 4-6 eV acceleration energy and approximately 0.5 mA filament current.

Problems Caused by Pressing Samples into Pellets

By pressing the finely powdered oxides into pellets, the surface of the resulting samples were usually smooth enough to appear glossy or semi-glossy, but some samples had iron or chromium contamination which indicated that the oxide had undergone a pressure induced reaction with the stainless steel anvil. Very strong hand pressure caused some oxides to react with the stainless steel anvil, but medium hand pressure usually did not produce undesired iron and chromium contamination. All analyses that showed any unexpected contamination were repeated. Other forms of accidental contamination (chlorine or previously analyzed oxides) were caused by insufficient cleaning of the stainless steel anvil, which was normally cleaned with a metal polishing solution (Pikal) and rinsed with distilled water and isopropanol. All analyses that showed any unexpected contamination were repeated.

Solution to Pressure Induced Contamination of Pellets

Experiments on ways to avoid the pressure-induced iron or chromium contamination, produced pellets with semi-smooth non-glossy surfaces which required more effort to produce good charge control. These non-glossy surfaces also gave electron count rates that were about 10-50% lower than the glossy or semi-glossy surfaces. As a result, it appears that very smooth surfaces, which appear glossy or semi-glossy, greatly simplify efforts to control surface charging under the charge-control mesh-screen and also enhance the electron count rate by 10-50% more than a pellet that has a semi-rough non-glossy appearance.

Extensive experiments on different methods to avoid contamination of the pellets revealed that contamination is minimized or avoided by using freshly cleaned aluminum foil as a "buffer" between the oxide powders and the metals in the steel anvil components. The aluminum foil, which is sold as a kitchen wrap material, is cleaned with 100% isopropanol (isopropyl alcohol) just prior to use. The foil is cut to a size that is readily useful with the pellet press device after it is cleaned. Alternately, we have also used a type of "glycine" paper which is commonly used to as a paper to hold powders when weighing a powdered sample. This "weighing" paper is common in many chemical laboratories and can be substituted for the aluminum foil whenever the pressing results with the aluminum foil produce undesired binding results. The glycine paper method sometimes introduces very small amounts of contaminants which produce a N (1s) and C (1s) signals. The amount of these contaminants is much smaller than the amount of contaminants that occur by simply pressing the powder without any sort of paper or aluminum foil buffers.



Source of Pellet Press Equipment

"Qwik Handi-Press" from Barnes Analytical Division, Spectra-Tech, Inc.652 Glenbrook Road, Stamford, Connecticut, 06906 (FAX 203-357-0609) Kit: Part # 0016-111 to 0016-121 contains 1,3, and 7 mm die sets. Originally purchased through Aldrich Chem. Co. in 1989.

E. Energy Resolution Details

Table 1: Experimentally Observed Relation Between Energy Resolution (FWHM) and Resolution Variables

Element (XPS signal)	Resulting FWHM	Resolution Setting	Pass Energy	X-ray Spot Size
Si (2p _{3/2}) crystal - fractured edge	0.38 eV	5	10 eV	40 x 250μ
Si (2p _{3/2}) crystal - fractured edge	0.43 eV	1	25 eV	80 x 350μ
Au (4f _{7/2}) foil - ion etched clean	0.64 eV	5	10 eV	250 x 1000μ
Au (4f _{7/2}) foil - ion etched clean	0.79 eV	1	25 eV	250 x 1000μ
Au (4f _{7/2}) foil - ion etched clean	0.86 eV	2	50 eV	250 x 1000μ
Au (4f _{7/2}) foil - ion etched clean	1.40 eV	4	150 eV	250 x 1000μ
Ag (3d _{5/2}) foil - ion etched clean	0.42 eV	5	10 eV	40 x 250μ
Ag (3d _{5/2}) foil - ion etched clean	0.64 eV	1	25 eV	40 x 250μ
Ag (3d _{5/2}) foil - ion etched clean	0.75 eV	2	50 eV	40 x 250μ
Ag (3d _{5/2}) foil - ion etched clean	1.00 eV	3	100 eV	40 x 250μ
Ag (3d _{5/2}) foil - ion etched clean	1.30 eV	4	150 eV	40 x 250μ
Cu (2p _{3/2}) foil - ion etched clean	0.85 eV	5	10 eV	250 x 1000μ
Cu (2p _{3/2}) foil - ion etched clean	0.94 eV	1	25 eV	250 x 1000μ
Cu (2p _{3/2}) foil - ion etched clean	1.06 eV	2	50 eV	250 x 1000μ
Cu (2p _{3/2}) foil - ion etched clean	1.60 eV	4	150 eV	250 x 1000μ
Cu (2p _{3/2}) foil - ion etched clean	0.85 eV	5	10 eV	150 x 800μ
Cu (2p _{3/2}) foil - ion etched clean	0.96 eV	1	25 eV	150 x 800μ
Cu (2p _{3/2}) foil - ion etched clean	1.05 eV	2	50 eV	150 x 800μ
Cu (3s) foil - ion etched clean	2.35 eV	2	50 eV	250 x 1000μ

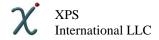


Table 2: Theoretical Analyzer Resolution versus Pass Energy Settings

Theoretical Analyser Resolution	Pass Energy	Effective Detector Width
0.25 eV	25.0 eV	3.5 eV
0.50	50	7.0
1.00	100	14.0
1.50	150	21.0

F. Energy Scale Reference Energies and Calibration Details

From May 1986 to January 1993

Energy Scale Reference Energies: 932.47 eV for Cu (2p_{3/2}) signal

122.39 eV for Cu (3s) signal

83.96 eV for Au (4f_{7/2}) signal

Binding Energy Uncertainty: less than ±0.08 eV Digital-to-Analog (DAC) Conversion Setting: 163.88

After January 1993

Energy Scale Reference Energies: 932.67 <±0.05 eV for Cu (2p_{3/2}) signal

 $122.45 < \pm 0.05$ eV for Cu (3s) signal $83.98 < \pm 0.05$ eV for Au $(4f_{7/2})$ signal

Observed Reference Energy: 75.01 <±0.05 eV for Cu (3p3) signal

Binding Energy Uncertainty: less than ±0.08 eV Digital-to-Analog (DAC) Conversion Setting: 163.87

Reference Energies of Adventitious Hydrocarbon Contaminants

From May 1986 to January 1993 the electron binding energy of adventitious hydrocarbons was assumed to occur at 284.6 eV based on SSI and C. D. Wagner's research and recommendations. Publications by P.Swift (Surface and Interface Analysis 4, 47 (1982), S. Kohiki and K. Oki (J. Electron Spectrosc. Related Phenom. 33, 375-380 (1984), and G. Barth, R. Linder and C. E. Bryson, III (Surface and Interface Analysis 11, 307-311 (1988) have shown that the electron binding energy for various hydrocarbon contaminants and polymers is not necessarily a constant number. Research by this author indicates that the electron binding energy for adventitious hydrocarbons lies somewhere between 284.4 and 287.0 eV depending on the underlying oxide materials. By taking a simple average of all available binding energies, the author has found that 285.0 eV is preferred for



hydrocarbons on ion etched metals where the hydrocarbon is many hours old. For naturally-formed native oxides the preferred binding energy is 285.2 eV. Oxide based materials at the far left of the periodic element table (columns 1-4) tend to have higher values (285.2-287.0 eV, while most of the transition metal oxides center around 285.0 eV. Near the far right of the periodic table, the binding energy again rises to a 285.2-286.5 eV range (columns 12-14). In routine practice, this author prefers to use the 285.0 eV number. Some potential factors that may cause this rather large range of electron binding energies for adventitious hydrocarbon contamination includes the dipole moment at the surface of the oxide material, which is expected to be much stronger than the dipole moment of a pure metal, and also, in the case of naturally formed native oxide films, the thickness of the native oxide, any physical or chemical treatments, the thickness of the adventitious hydrocarbon layer, and the type of instrument used to analyze the sample. The type of instrument being used may cause different shifts in the observed binding energy of the adventitious hydrocarbon contamination because the source may or may not generate different amounts of low energy secondary electrons from the window that protects the X-ray source. The heat from the source and contamination that degases from a just turned on source may also influence the observed binding energy. Electron flood guns may or may not influence the binding energy as well.

Instrument Stability and Long Term Calibration

Initially each of the three SSI systems, that we have used, was calibrated 2-3 times per week because its ability to maintain accurate voltage settings was unknown. Once it was determined that the systems could maintain reliable voltage settings for 1-3 months, it was decided that good calibration could be maintained by checking and, if necessary, correcting the pass energies of the system on a 2-4 week basis. Each of the three SSI XPS instruments, that we have used, have been calibrated on a routine basis every 2-4 weeks by using SSI's reference energies. By using this method over several years time, it was found that the maximum uncertainty (error in pass energies) was normally $<\pm0.10\,\mathrm{eV}$, but a few times rose to $\pm0.15\,\mathrm{eV}$ or less. In a very rare case, the uncertainty rose to $0.20\,\mathrm{eV}$. Long term use of the SSI systems has shown that the DAC circuit does not change enough to be observed unless the room temperature changes by more than 10 deg Centigrade. If the room temperature changes within a few hours time by more than 10 deg or the temperature of the DAC chip is changed by more than 10 deg, then a $>0.1\,\mathrm{eV}$ shift, which is much smaller than the reliability of almost all literature BEs, can be observed. Variables, which seem to cause pass energy settings to change slightly, include building line-voltages, ion etching conditions, and the addition or removal of some electrical device.

G. Electron Counting and Instrument Response Function Details (for the X-Probe System only)

Instrument Response Functions

Instrument Response Function: $Q(E)=E^{+0.27}$ for 150 eV PE (ref.3) Instrument Response Function: $Q(E)=E^{+1.0}$ for 50 eV PE (ref.3)

Signal/Background Ratios for Ion Etched Silver using a 250x1000 μ Spot*

Pass Energy	25 eV	50 eV	100 eV	150 eV
S/BG ratio**	>140	>110	>70	>50

^{*} Using a 90° electron take-off-angle and a smooth Ag°/mylar film.



^{**} The S/BG ratio is a simple numerical ratio of electrons counts at the peak maximum relative to the average electron counts observed at approximately 10 eV lower BE.

Lens Voltage Settings Available via Software under Instrument Calibration

Pass Energy*	29.6-29.8	54.7-54.9	105.1-105.3	155.9-156.2
Detector Widths	3.743	7.486	14.954	22.297
Sensitivity Exponent	0.7	1.1	1.3	1.5
V1 Offset	30	55	105	155
V1 Slope	0.600	0.611	0.676	0.709

^{*} These pass energies include corrections for instrument work function. True pass energies were set to 25, 50, 100, and 150 eV ±0.1 eV.

H. Effects of Poorly Focussing the Distance between the Sample and the Electron Lens

If the focus distance between the sample surface and the electron collection lens is poorly adjusted, then the number of electron counts drops very quickly. A 0.5mm error in focus produces a >300% decrease in counts, but does not produce any observable error in binding energies, which is a common problem with many other instruments. A 0.1mm error in focus produces a 15% decrease in peak area counts and is easily observed as a horizontal displacement in the static (un-scanned mode) XPS signal as observed on the standard CRT display of the detector response. Such a decrease in signal intensity generally causes the operator to correct the focus error so as to maximize the electron count rate. In this manner, the operator has avoided any chance of obtaining false BE readings and has accurately reproduced a nearly absolute focus point which greatly increases the quantitative accuracy of any unknown sample. Experiments with the Bragg angle alignment of the crystal indicated that the maximum error due to an unusual bad alignment of the crystal would be <0.1 eV. To observe an error greater than 0.1 eV, the electron counts were found to decrease by >50%.

I. Quantitation Details and Choice of "Sensitivity Exponents"

By default, the SSI software uses a 0.7 number as the sensitivity exponent factor for each pass energy setting which are used in an equation that modifies theoretically calculated atomic photo-ionization cross-sections (John H. Scofield) to generate relative sensitivity factors that are valid for this XPS systems and which can be used to generate valid atomic percentages. The 0.7 value produces a $\pm 10\%$ accuracy in quantitative results for XPS signals obtained by using a 150 eV pass energy and occur within the 0-700 eV BE range. For signals that occur at higher BEs, it is generally necessary to change the sensitivity exponent factor to a 1.1 or higher value. To measure signals obtained by using other pass energies for quantitation, it is necessary to use other sensitivity exponent factors, if the user desires to maximize quantitative accuracy. To determine useful sensitivity exponents, it is possible to use freshly ion etched poly-crystalline copper foil to test the validity of the sensitivity exponent for larger BE ranges and different pass energies. By integrating the peak areas of the Cu (2p1), Cu (2p3), Cu (3s), Cu (3p) and Cu (3d) signals with a modest amount of attention to baseline end points it is possible to perform trial and error choices of the sensitivity exponents until a useful number is determined. Once a useful number has been entered into the computer software routine, then the software can generate fictional atomic percentages for each of the integrated copper signals which will generate 20 atom % values with a uncertainty of ± 1 -2 atom %. If the exponent factor is severely wrong then the atomic percentages will generate numbers such as 10%, 11%, 26%, 24%, and 29% or perhaps 31%, 28%, 14%, 13%, and 14%. This trial-and-error approach may require 1-2 hours time and can be done on either wide scan data or more preferably narrow scan data for each of the 4-5 pass energies. This method, in effect, assumes that all five of the relative sensitivity factors for copper are reasonably correct. If wide scan data are used, this method requires a little extra effort to avoid the satellites associated with the Cu (2p) signals. This method, in effect, pretends that the pure copper sample is a standard material that is composed of 5 components which are present in 20 atomic % concentration. The objective is to change the sensitivity exponent until the software



generates a 20 atom % result for each of the five copper signals. After useful sensitivity exponents are found, they are tested by analyzing freshly exposed bulk regions of crystalline materials such as SiO2, Al2O3, and NaCl.

The high and low BE signals of the NaCl crystal are especially useful to test the validity of the sensitivity exponents. As further checks, the freshly exposed bulk of common polymers (e.g. mylar or PMMA) or a thin film of high purity silicone oil can also be analyzed. Teflon has repeatedly given slightly larger than desirable error by comparison to the other materials listed above. For that reason Teflon is a less desirable material to test the sensitivity exponents.

J. Crude Tests of the Reliability of Relative Sensitivity Factors

Crude testing of Scofield's numbers are included in atomic percentage composition tables that give atomic percentages for only one element. This testing used the software's automatic peak area integration software that is reasonably accurate. The results indicate that some of the relative sensitivity factors for some of the weaker signals are less reliable. If, however, all factors are taken into account, then Scofield's numbers are reliable to a 95% accuracy level for truly homogeneous materials.

K. Traceability Details

The definition of traceability reported by Martin P. Seah and Cedric J. Powell in the J. Vac. Soc. Technol. Vol 8, p.736 (1990) publication is: "The property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons."

Traceability of Reference Binding Energies (Calibration)

At this time, there are no international standards for binding energies or reference energies. Numbers which are considered to be standard binding energies (BE), which would lead to traceability in BEs, include (a) those provided by Martin P. Seah at the National Physical Laboratory (NPL) in the United Kingdom (England), and (b) those provided by the ASTM in the USA "Standard Practice for Checking the Operating Characteristics of XPS Spectrometers" designated as "E 902-88". Other nations also have similar national standards, which tend to imitate those set by the USA and the UK. Recently, many people in the world have been using NPL's reference energies, which have become "de facto" standards but have not yet been accepted by the International Standards Organization (ISO). There are still many workers and researchers using various numbers provided by the instrument makers. The author of this book was using Surface Science Instruments (SSI) Co. reference energies until December 1992 and then switched to NPL BEs in January 1993. SSI reference energies came from Hewlett-Packard (HP). SSI and HP both used high precision voltage meters from HP to calibrate their ESCA machines (i.e. X, M, and S-Probe and HP-5950 A-type and B-type, resp.). Hewlett Packard was the first company to offer a commercial ESCA system, which used reference energies developed in cooperation with Kai Siegbahn at Uppsala, who effectively developed ESCA into a useful science and received the Nobel Prize. In a recent effort to improve the accuracy of BEs obtained from pure elements, the S-Probe pass energies were checked and corrected, if needed, almost every work-day for two months to obtain high precision and high accuracy BEs for the pure elements that are metals. This study used the NPL reference energies with Cu (2p3) at 932.67 eV with +/-0.02 uncertainty and Au (4f7) 83.98 eV with +/-0.02 uncertainty by using 0.02 eV/pt. steps for the calibrations. To determine the "true" BE of each of the pure elements, which were scraped clean in air and t



valence (Fermi edge) band, and narrow scans of the main signals for each metal at 50, 25 and 10 eV pass energies. Each repetitive experiment run lasted about 4 hours. Therefore, if NPL's BE numbers are accepted as "de facto" international standards, then the ultimate traceability of BEs in this data collection can be related to NPL BE numbers for Cu (2p3) and Au (4f7). In a different, but similar manner, the BEs used to calibrate the S-Probe are traceable to Siegbahn's work and HP's high precision, high voltage meters.

Traceability Transfer from Pure Metals to Non-conductive Binary Oxides

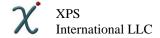
A question that should be posed is traceability to the oxide BEs. Traceability begins with NPL's BEs for pure copper and gold as state above. Traceability then transfers to pure element BEs which are based on NPL reference BEs. Traceability then transfers to pure element BEs based on SSI's reference BEs, and then the naturally formed native oxide data published in Volume 2 of our XPS Spectral Handbook series where BEs were measured from pure element signals and also the naturally formed native oxide signals. Naturally formed native oxides typically have thin oxide films (10-80Å) which, in general, behave as good or true electrical conductors, which allows a direct measure of the true binding energy of many, but not all, binary oxides. To determine if traceability can indeed be transferred to true binary oxides, it was necessary to study the behavior of the naturally formed native oxides by applying various flood gun settings with the samples grounded and insulated. The results from that study can be used to transfer traceability to the experimentally observed BEs of pure binary oxides. The most difficult transfer of traceability occurs for the naturally formed native oxide systems. If the flood gun study was not done, then it is difficult to transfer traceability in a reliable manner from a conductive metal to one of its corresponding non-conductive binary oxides.

Traceability of Instrument Response (Throughput) Function

Copper, gold and silver data obtained from the M-Probe system were submitted to Martin P. Seah at the NPL for a round robin test on transmission function; the results of which were published in Surface and Interface Analysis, p.243 (1993). In that publication, M-Probe data, which we contributed, were attributed to group #35. That paper reported that instrument has a Q(E) = $E^{0.27}$ for Rex 4 pass energy (PE=150 V) and a Q(E) = $E^{1.0}$ for the Res 2 pass energy (PE=50 V). If the NPL method is accepted as a "de-facto" standard, even though it is not an internationally recognized standard, then the transmission function and quantitation results of the S-Probe system are traceable to the "metrology spectrometer" at NPL.

Traceability of Relative Sensitivity Factors used for Quantitation

Scofield's theoretically calculated photo-ionization cross-sections are internationally used as the "de-facto" standard theoretical numbers, except in Russia and a few other places, where Band's numbers are preferred but are almost identical to Scofields. The SSI system uses a very simple equation that modifies Scofield's numbers to generate relative sensitivity factors that are used by the SSI software to calculate atom %s. That equation corrects for pass energy differences, transmission function differences, and inelastic mean free path versus kinetic energy dependency. The SSI system relies on Scofield numbers and that simple equation. Other instrument makers prefer to blend Scofield's numbers and experimentally determined numbers.



Traceability of Sample Purity

The purity of the commercially pure (99+%) binary oxides can be traced to Aldrich's ICP or AA analyses performed by Aldrich. Copies of their results are included in the handbook at the beginning of each group of spectra. Similar data sheets were also obtained for samples bought from Cerac. A set of gold, copper, and silver samples, i.e. "Reference Metal Samples SCAA90" set, kit #367, was obtained from the NPL and used to test the instrument response function of the M-Probe system. Binding energies obtained from those gold, copper, and silver samples were identical to binding energies obtained from our commonplace gold, copper, and silver samples within the expected uncertainty of ± 0.08 eV used for routine instrument calibration.

L. Reference Papers Describing Capabilities of X-Probe, M-Probe, and S-Probe XPS systems

1. Robert L. Chaney, Surface and Interface Analysis, 10, 36-47 (1987)

2. Noel H. Turner, Surface and Interface Analysis, 18, 47-51 (1992)

3. M. P. Seah, Surface and Interface Analysis, 20, 243-266 (1993)

4. L.T. Weng et al, Surface and Interface Analysis, 20, 179-192 (1993)

5. L.T. Weng et al, Surface and Interface Analysis, 20, 193-205 (1993)

6. B. Vincent Crist, Surface Science Spectra, 1, 292-296 (1993)

7. B. Vincent Crist, Surface Science Spectra, 1, 376-380 (1993)

[re: X-Probe]

[re: Quantitation]

[re: Response Function]

[re: Response Function]

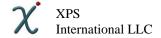
[re: Response Function]

[re: KBr spectra]

[re: Ar/C spectra]



Spectra of Polymers & Polymers Damaged by X-rays



Polymers in NECSA/BIO Collection (at the University of Washington, USA)

HC Polymers	Acronym	HCON Polymers	Acronym
Poly-4-methyl styrene	4MPS	Biomer™	
Poly-8-octyl styrene	POS	Methylene di-isocynate/butane diol co-polyme	r
Poly-ethylene	PE	Methylene di-isocynate/propane diamine co-polymer	
Poly-styrene	PS	Methylene di-isocynate/propane diamine/butan	e diol ter-polymer
Poly-α-methyl styrene	AMPS		-
		HCX Polymers (X=Cl, F)	
HCO Polymers		•	
		Poly-2-chloro ethyl methacrylate	CLMA
Poly-2-hydroxy ethyl methacrylate	PHEMA	Poly-tetra-fluoro ethylene	PTFE
Poly-4-ethoxy styrene	PES	Poly-vinyl chloride	PVC
Poly-4-hydroxy styrene	PHS	Poly-vinylidene di-fluoride	PVDF
Poly-ether ether ketone	PEEK		
Poly-ethyl acrylate	PEA		
Poly-ethyl methacrylate	PEMA	Miscellaneous	
Poly-ethylene glycol	PEG		
Poly-ethylene terephthalate	PET	Cellulose (filter paper)	
Poly-methyl acrylate	PMA	4,4'-Di-methoxy benzophenone	4DBP
Poly-methyl methacrylate	PMMA	Poly-4-hydroxy styrene -	
Poly-propylene glycol	PPG	tri-fluoro acetic anhydride derivative	TFAA
Poly-tetra-methylene glycol	PTMG	Poly-di-methyl siloxane	PDMS
Poly-vinyl alcohol	PVA	Poly-hydroxy ethyl methacrylate -	
		tri-methyl silane derivative	HTMS



Poly - α-Methyl Styrene (4% in toluene)

Detailed Surface Composition Table

File name: AMPS SU.MRS

Region:

Description: Poly(alpha-methyl styrene), 4% IN TOLUENE, 4/11/88

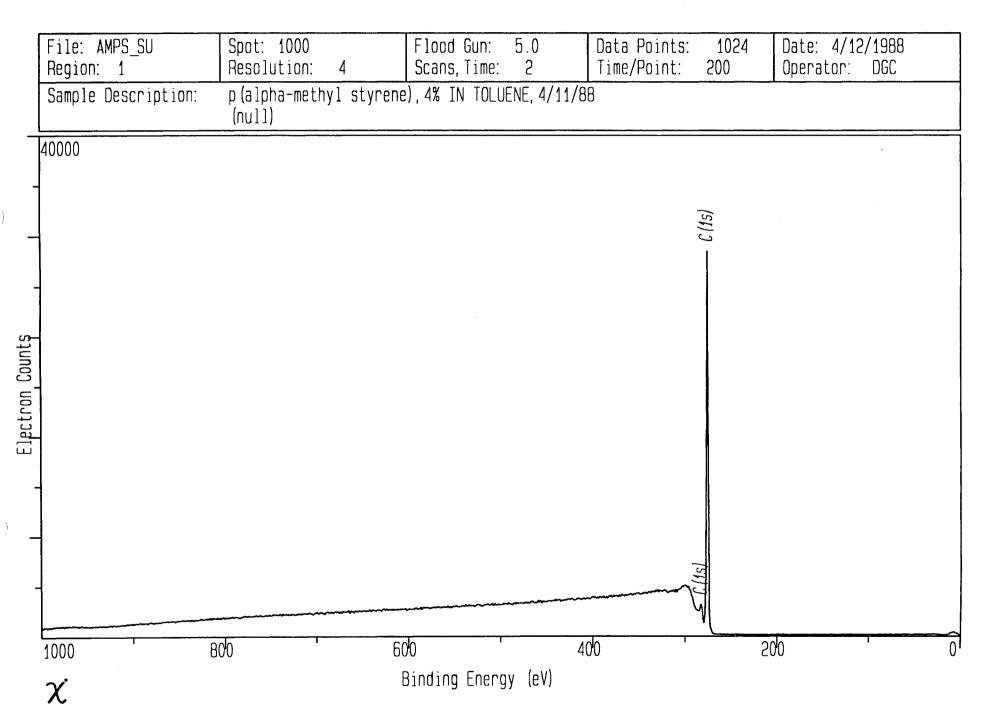
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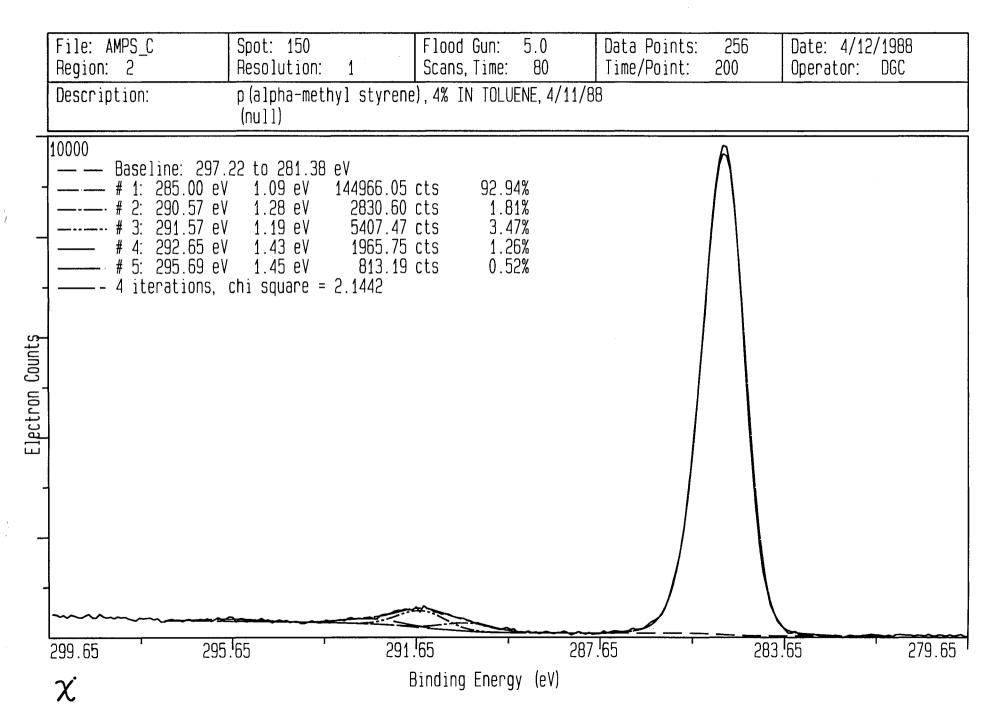
DGC

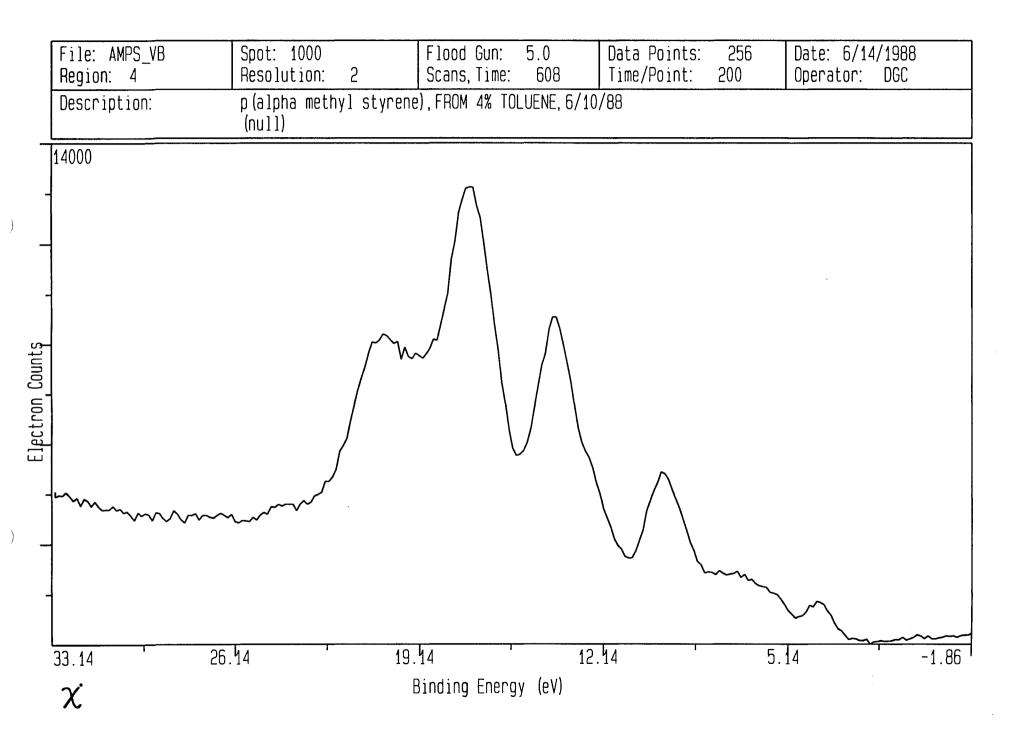
Date:

Tue Apr 11 1988

	Corrected	Exper.	Sens	Norm	Relative	
Signal	BE	BE	Factor	Area	Area	Atom %
* C 1s	280.0	275.0	1.01	153353	152501	94.37
* C 1s	287.5	282.5	1.00	9102	9091	5.63







Poly 2-Chloro Ethyl Methacrylate (3% solution)

Detailed Surface Composition Table

File name: CLMA SU.MRS

Region:

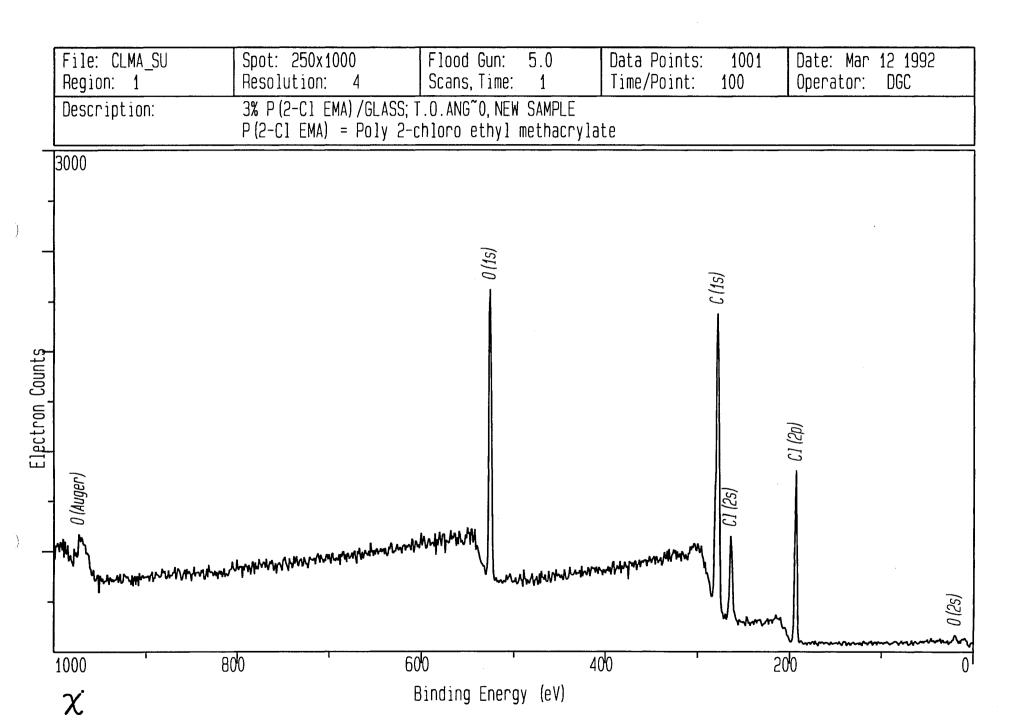
Description: 3% P(2-Cl EMA)/GLASS; TOA~0, NEW SAMPLE, P(2-Cl-EMA) = Poly 2-chloro ethyl

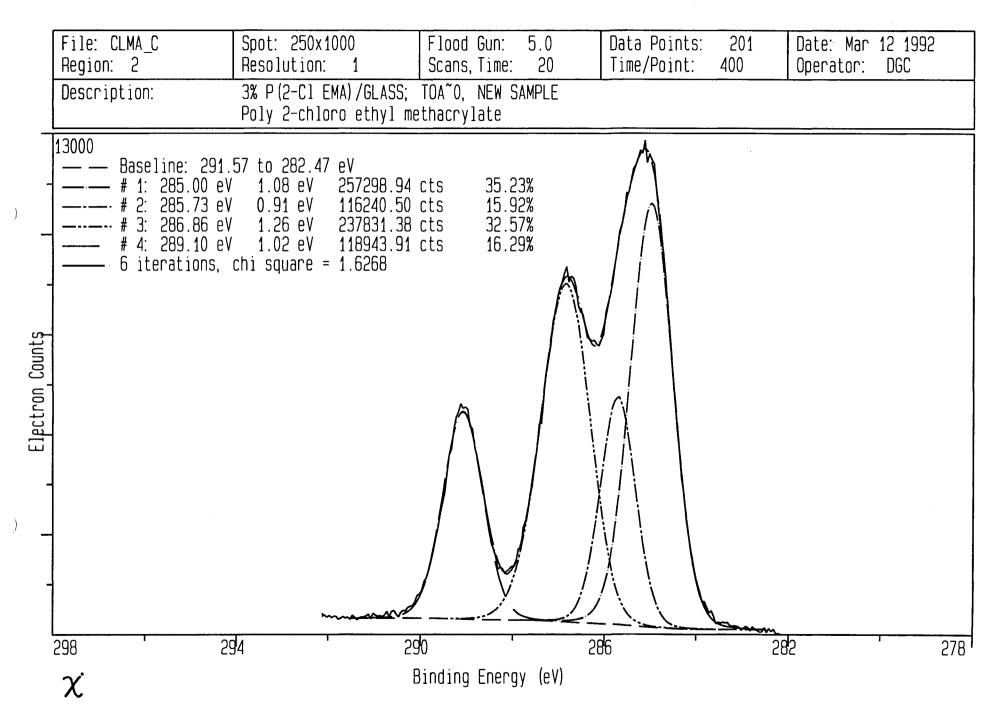
methacrylate

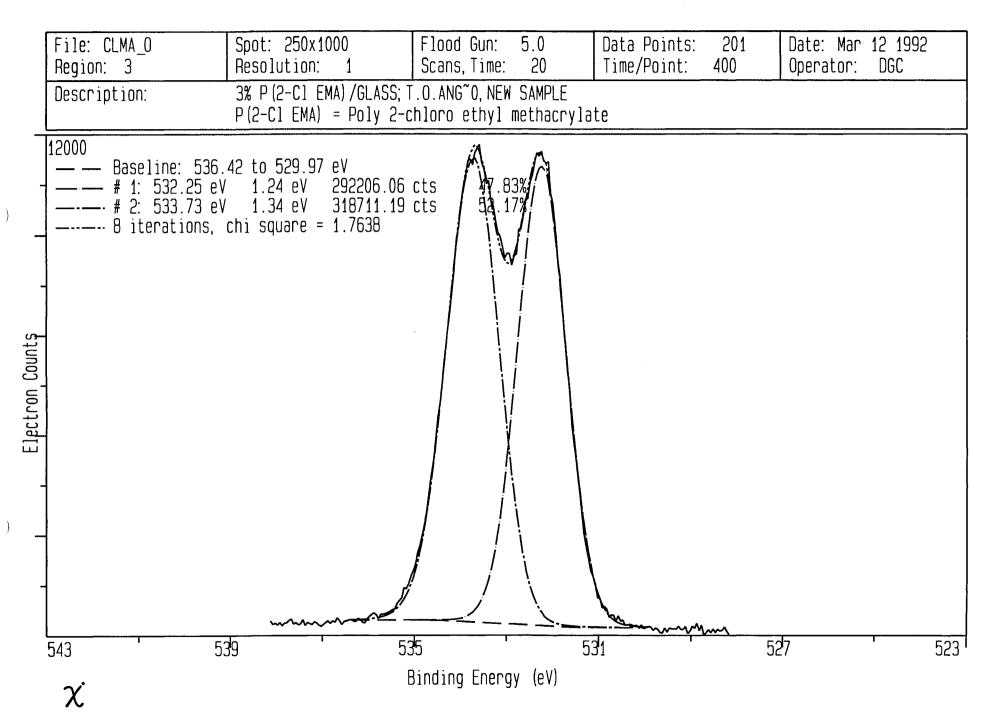
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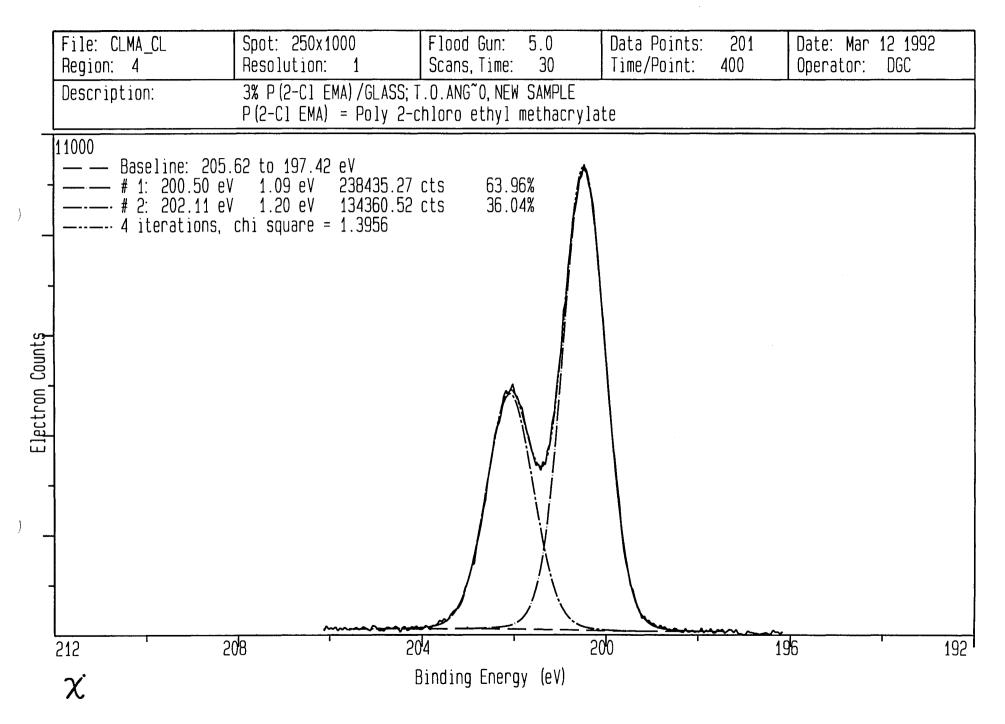
Date: Thu Mar 12 14:13 1992

	Corrected	${\tt Exper.}$	Sens	Norm	Relative	
Signal	BE	BE	Factor	Area	Area	Atom 8
O Auger	$97\overline{7.6}$	$9\overline{72}.6$	0.00	6548	o	
* 0 1s	529.8	524.8	2.45	57206	23337	21.66
* C 1s	282.3	277.3	1.00	72311	71964	66.81
Cl2s	268.2	263.2	1.71	18551	10823	
* Cl2p	197.3	192.3	2.42	30110	12420	11.53
0 2s	24.2	19.2	0.16	1988	12105	









Poly 2-Hydroxyethyl Methacrylate

Detailed Surface Composition Table

File name: HEMA SU.MRS

Region:

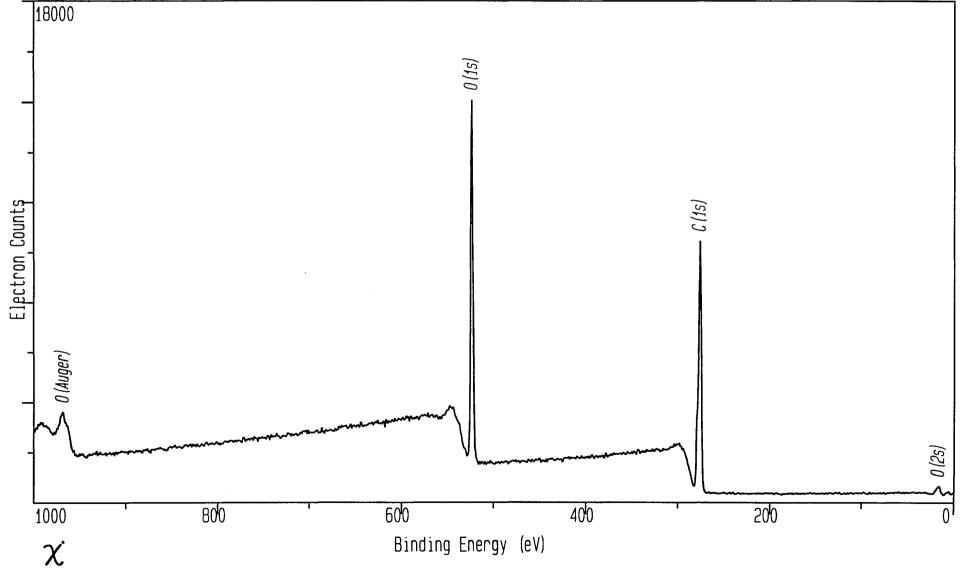
Description: NAKAHAMA, WC11-18-8, AH92-26, pHEMA = Poly 2-hydroxyethyl methacrylate

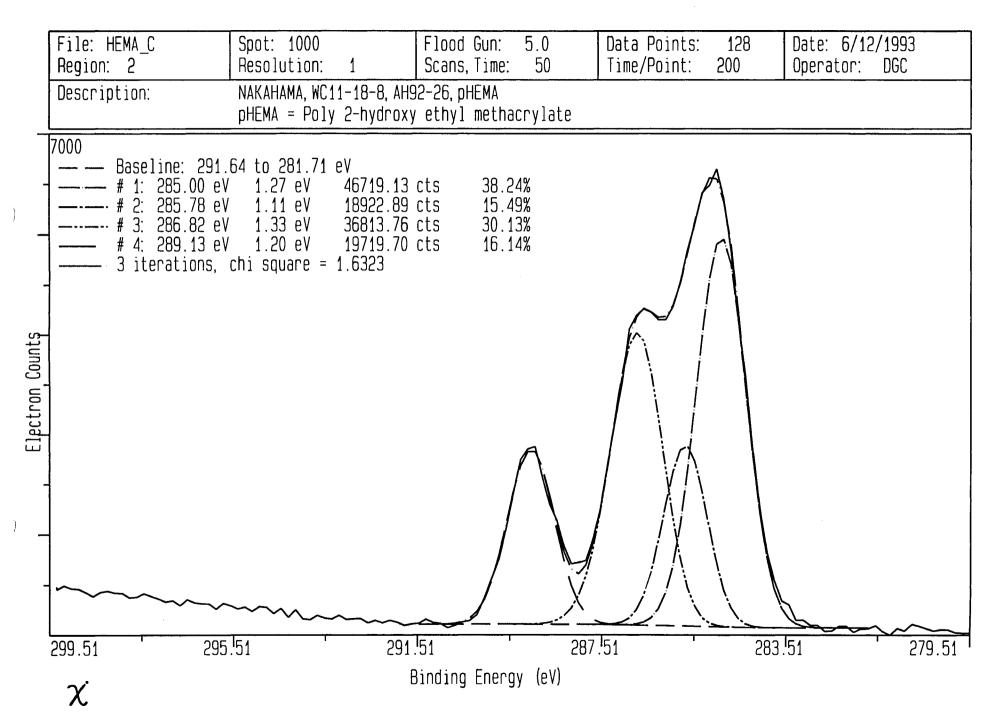
Operator: DGC

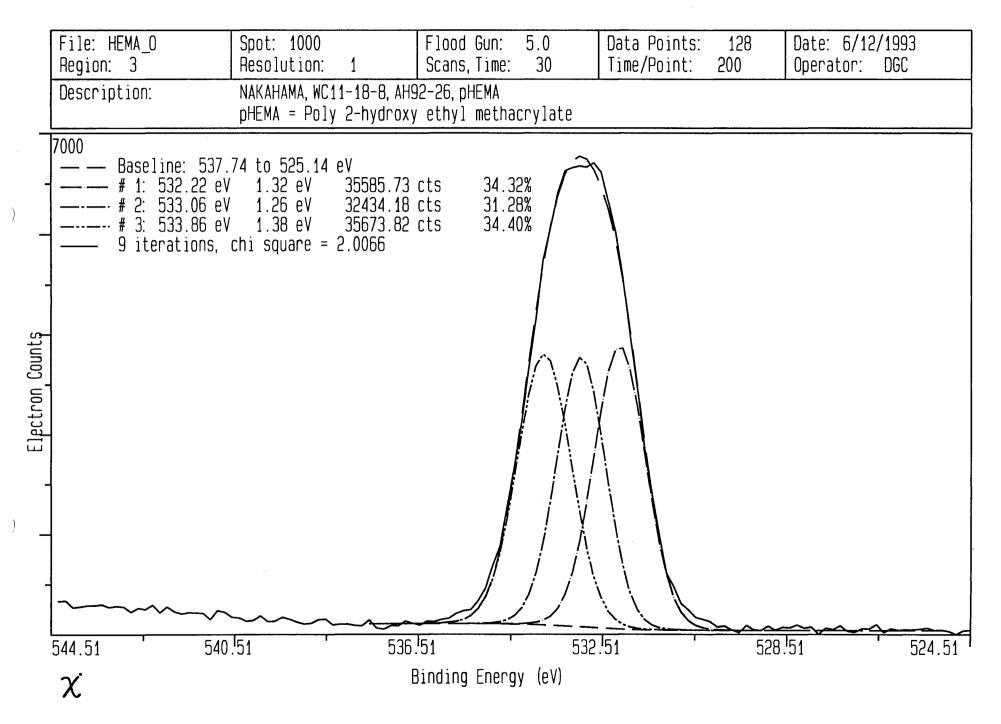
Date: Sat Jun 12 1993

	Corrected	Exper.	Sens	Norm	Relative	
Signal	BE	BE	Factor	Area	Area	Atom &
O Auger	973.0	9 68 .0	0.00	15262	О	
* 0 1s	527.8	522.8	2.51	49057	19542	31.46
* C 1s	280.1	275.1	1.01	42803	42566	68.54
0 2s	20.4	15.4	0.16	1892	11734	

File: HEMA_SU	Spot: 1000	Flood Gun: 5.0	Data Points:	1024	Date: 6/12/1993
Region: 1	Resolution: 4	Scans, Time: 4	Time/Point:	200	Operator: DGC
Description:	NAKAHAMA, WC11-18-8, pHEMA = Poly 2-hydr	AH92-26, pHEMA oxyethyl methacrylate			







Poly 2-Hydroxy Ethyl Methacrylate, trimethyl silane

Detailed Surface Composition Table

File name: HTMS_SU.MRS

Region:

Description: NAKAHAMA, WC11-18-9, AH92-26', Poly 2-hydroxy ethyl methacrylate, trimethyl

derivative

DGC

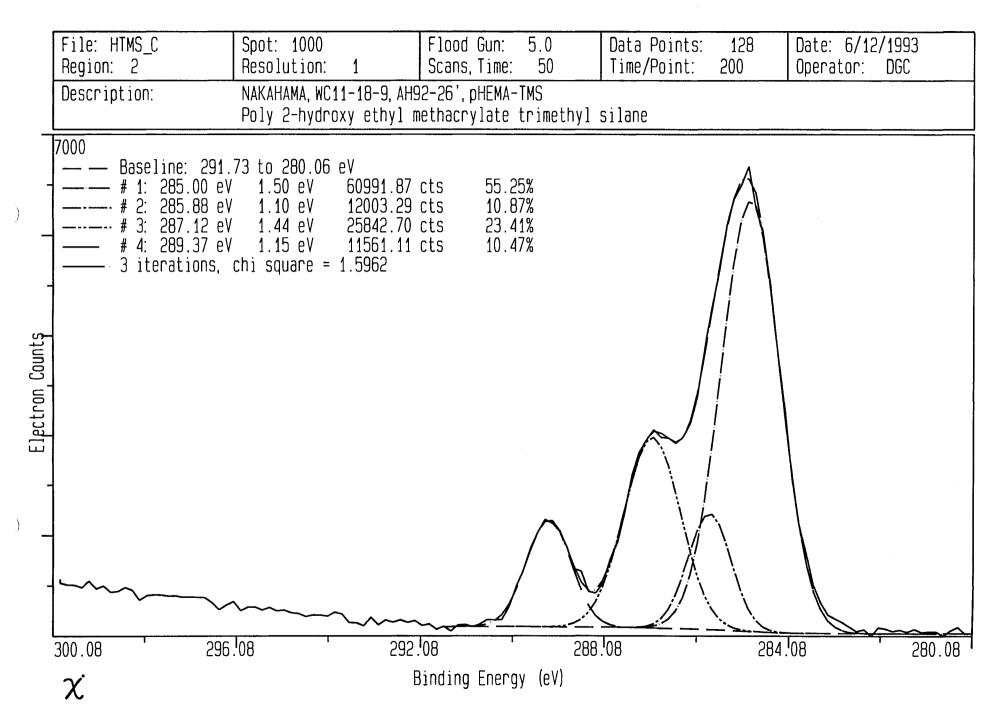
Operator:

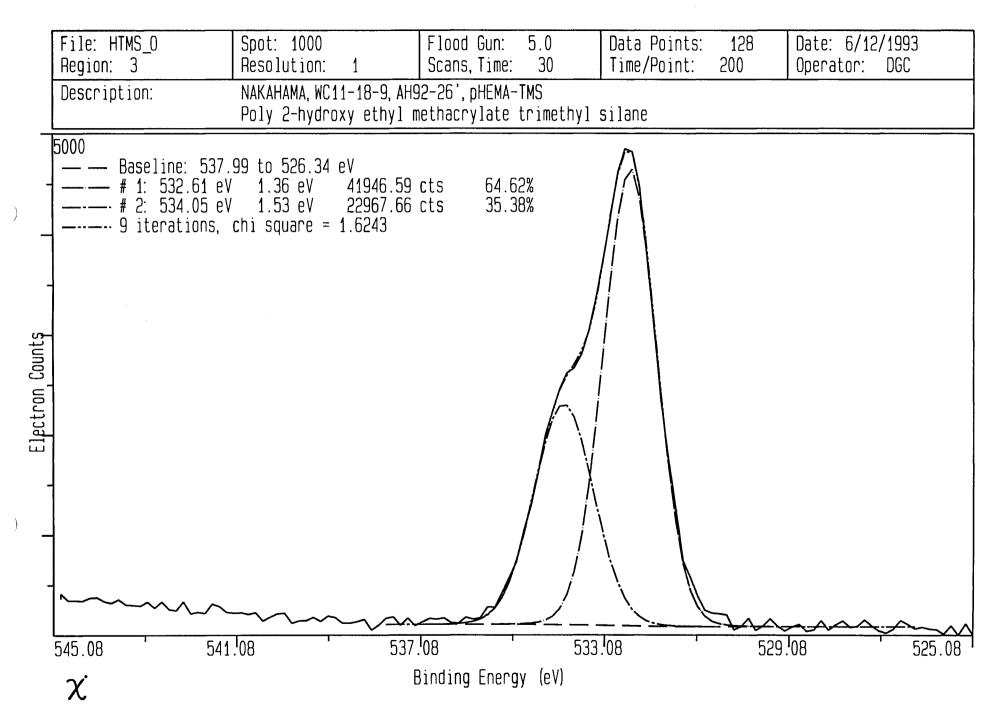
Date:

Sat Jun 12 1993

	Corrected	Exper.	Sens	Norm	Relative	
Signal	BE	BE	Factor	Area	Area	Atom 8
0 Auger	99 6. 5	$9\overline{91.5}$	0.00	1390	0	_
O Auger	974.3	969.3	0.00	9353	0	
* 0 1s	527.0	522.0	2.51	30369	12090	21.89
* C 1s	279.0	274.0	1.01	38435	38200	69.17
* Si2s	146.5	141.5	1.03	5099	4935	8.94
Si2p	95.2	90.2	0.91	4532	4994	
0 2s	19.7	14.7	0.16	1652	10240	

			· · · · · · · · · · · · · · · · · · ·				
File: HTMS_SU	Spot: 1000			Data Points:	1024	Date: 6/12/1993	
Region: 1	Resolution	4 Scans,	Time: 4	Time/Point:	200	Operator: DGC	
Description:	NAKAHAMA, W	C11-18-9, AH92-26',	DHEMA-TMS				
		roxy ethyl methacr		silane			
12000					<u></u>		
12000							
4							
			2)	-	C (1s)		
4			0 (1s)	(3		
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E lectron Counts							
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2							
U/Auger) • O (Auger)					İ		
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$-\sqrt{\lambda}$		and the same of th		^		Si (2s) Si (2p)	
Landens	white the market of the same		V	Mark Branch Branch	<u> </u>	5	
-			Product M. sans				0 (2s)
				1	/ h)0
						سالس	~~~~
1000	80'0	600	' 4	10'0	50	<i>'</i> '	01
√.		Bindina	Energy (eV)				
χ		- J	3,				





4,4' Dimethoxybenzophenone (FW=242.27)

Detailed Surface Composition Table

File name: 4DBP_SU.MRS

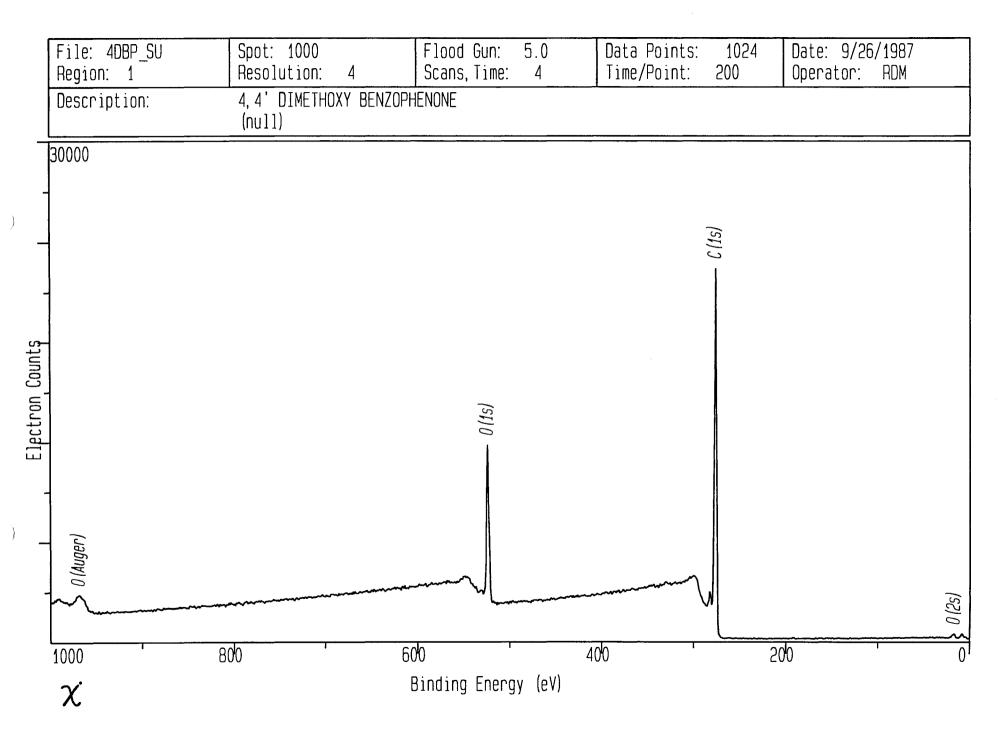
Region:

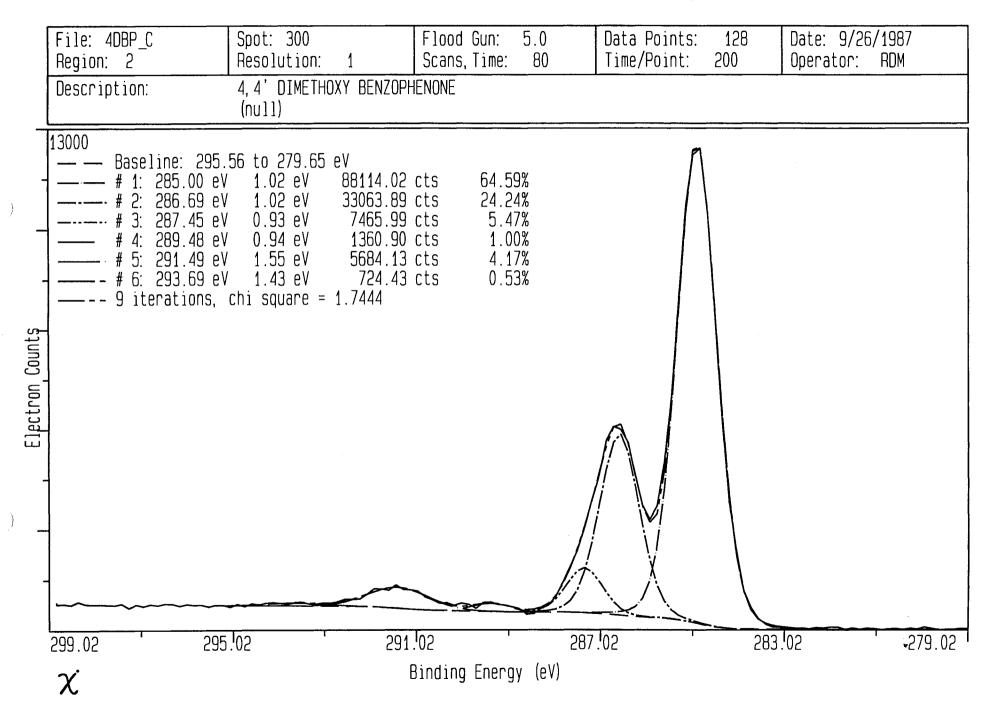
Description: 4,4' DIMETHOXY BENZOPHENONE

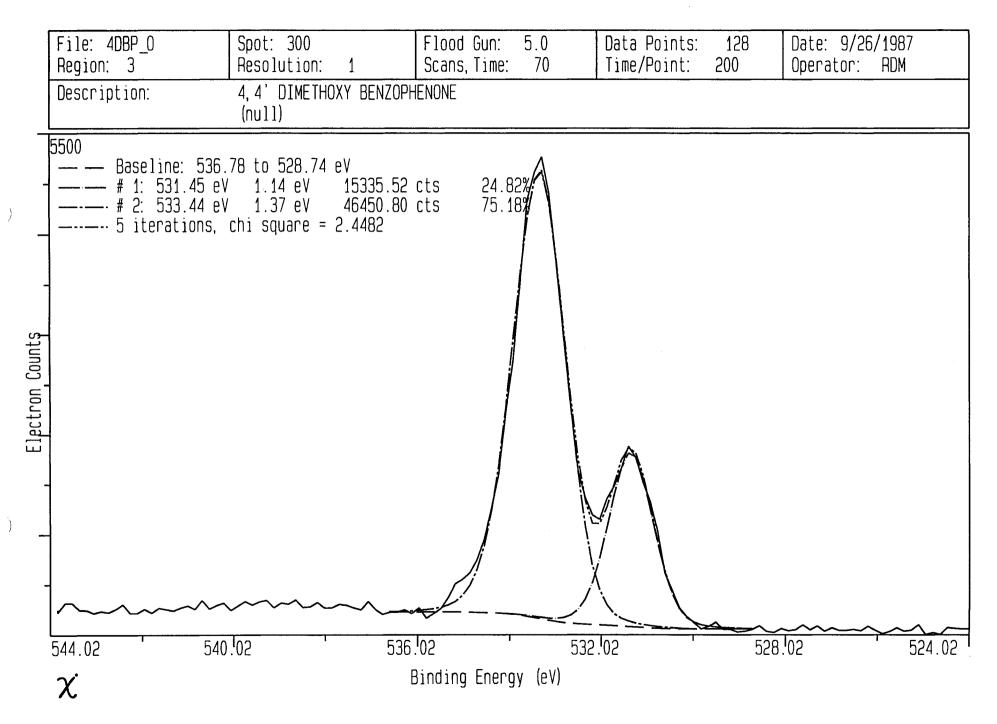
Operator: RDM

Date: Sat Sep 26 1987

	Corrected	Exper.	Sens	Norm	Relative	
Signal	BE	BE	Factor	Area	Area	Atom 8
O Auger	97 3. 3	9 68 .3	0.00	10654	о	
* 0 1s	528.7	523.7	2.51	37165	14815	15.57
* C 1s	280.1	275.1	1.01	80786	80343	84.43
0 2s	21.0	16.0	0.16	1105	6852	







Poly - 4-Ethoxy Styrene

Detailed Surface Composition Table

File name: PES SU.MRS

Region: 1

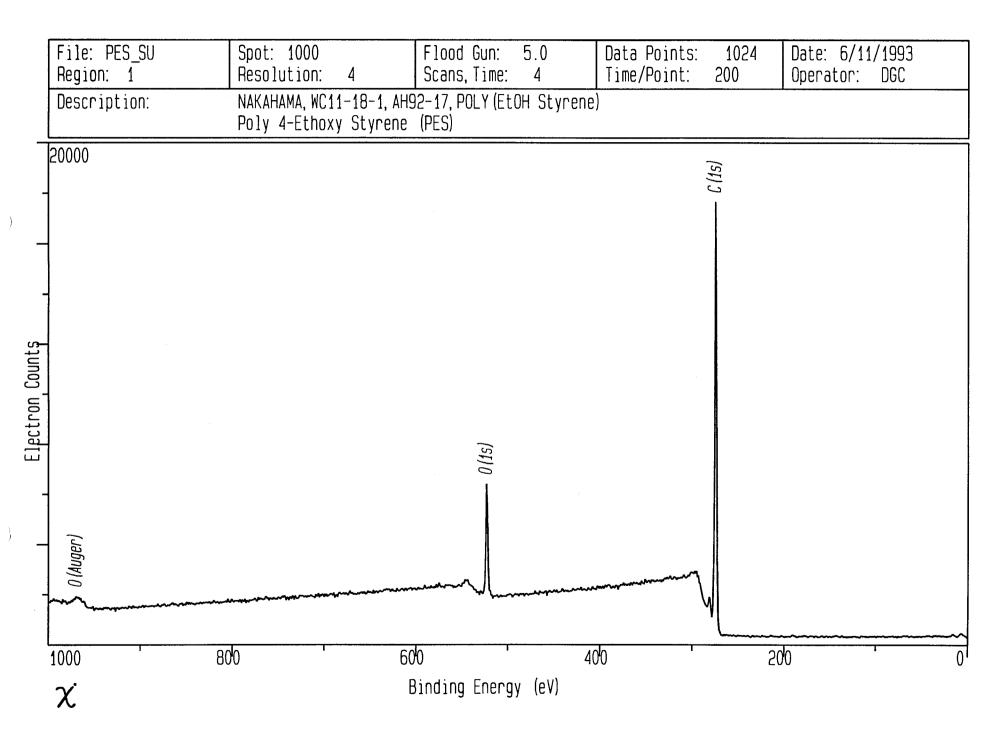
Description: NAKAHAMA, WC11-18-1, AH92-17, POLY(EtOH Styrene)

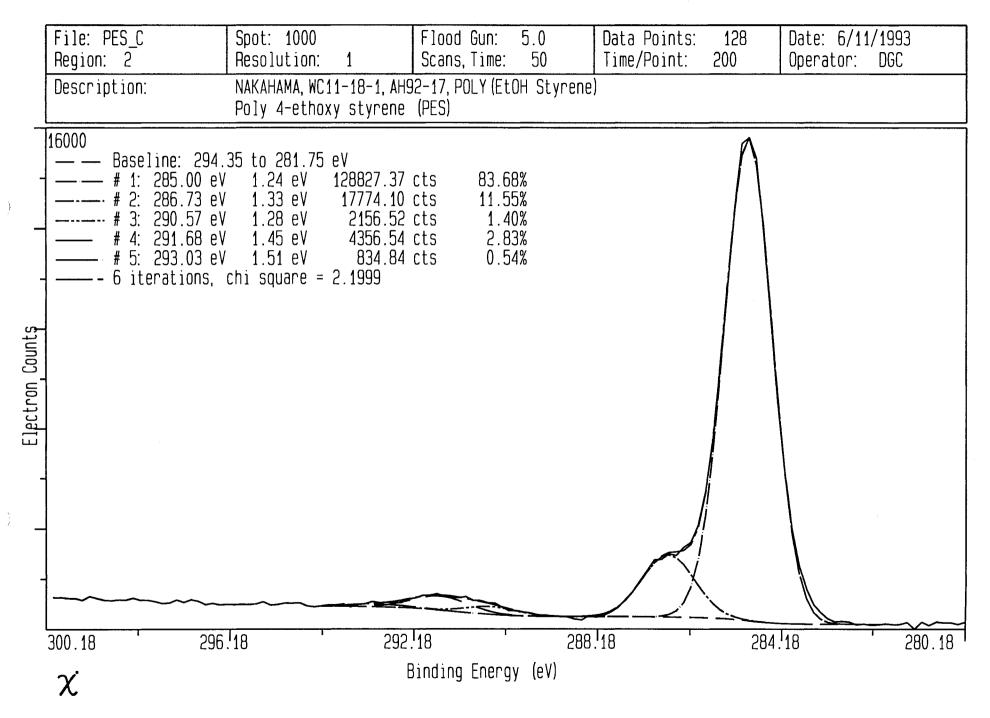
Poly 4-Ethoxy Styrene (PES)

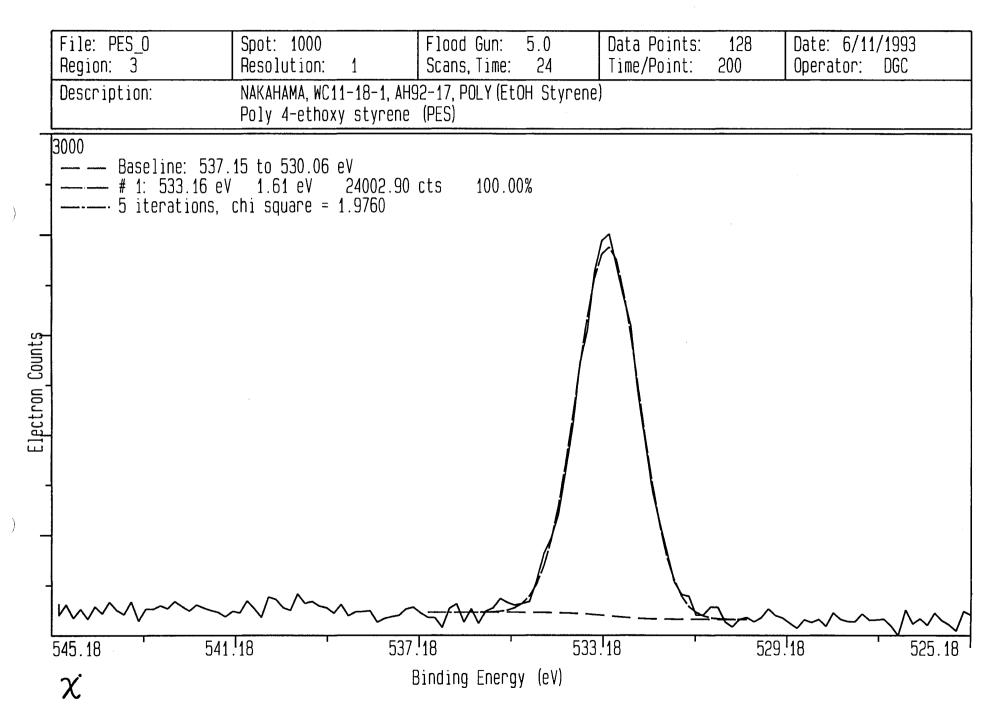
Operator: DGC

Date: Fri Jun 11 1993

	Corrected	Exper.	Sens	Norm	Relative	
Signal	BE	BE	Factor	Area	Area	Atom %
0 Auger	$97\overline{4.5}$	969.5	0.00	1970	0	
* 0 1s	527.2	522.2	2.51	14984	5966	10.55
* C 1s	278.8	273.8	1.01	50911	50593	89.45







Poly - 4-Hydroxy Styrene

Detailed Surface Composition Table

File name: PHS_SU.MRS

Region:

Description: COMMERCIAL poly (4 hydroxystyrene)

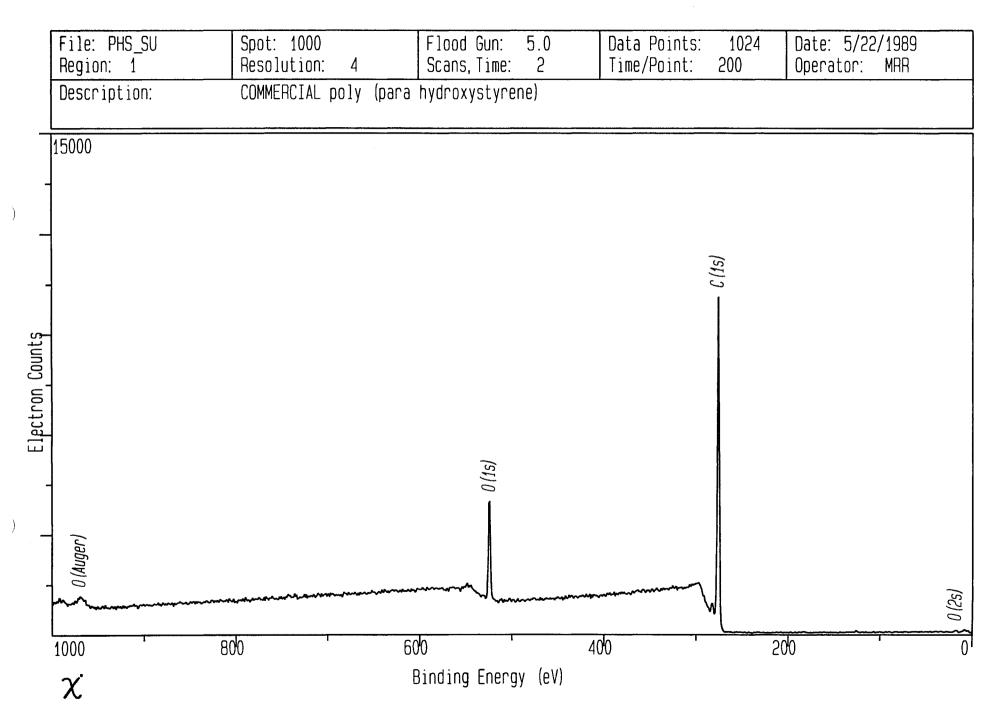
Operator:

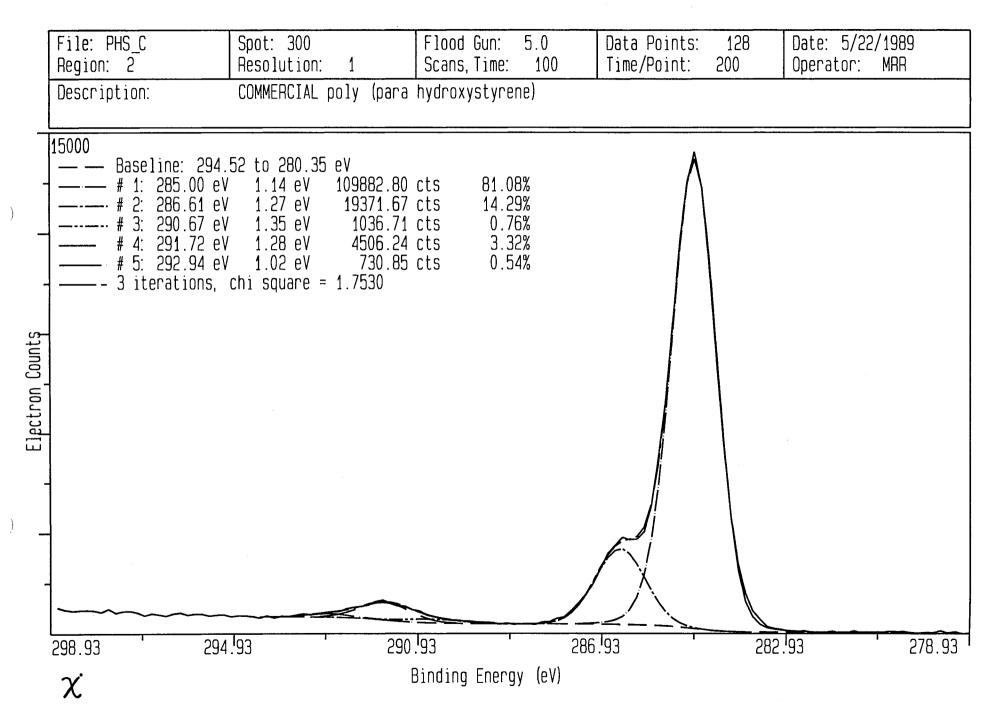
Date:

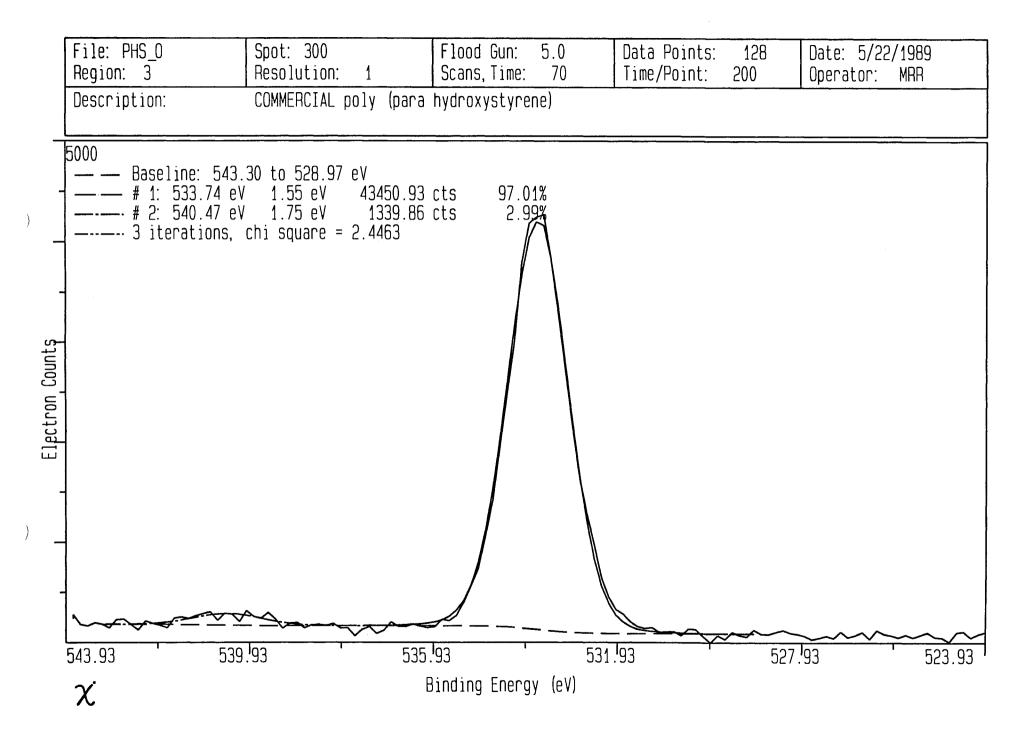
Mon May 22 1989

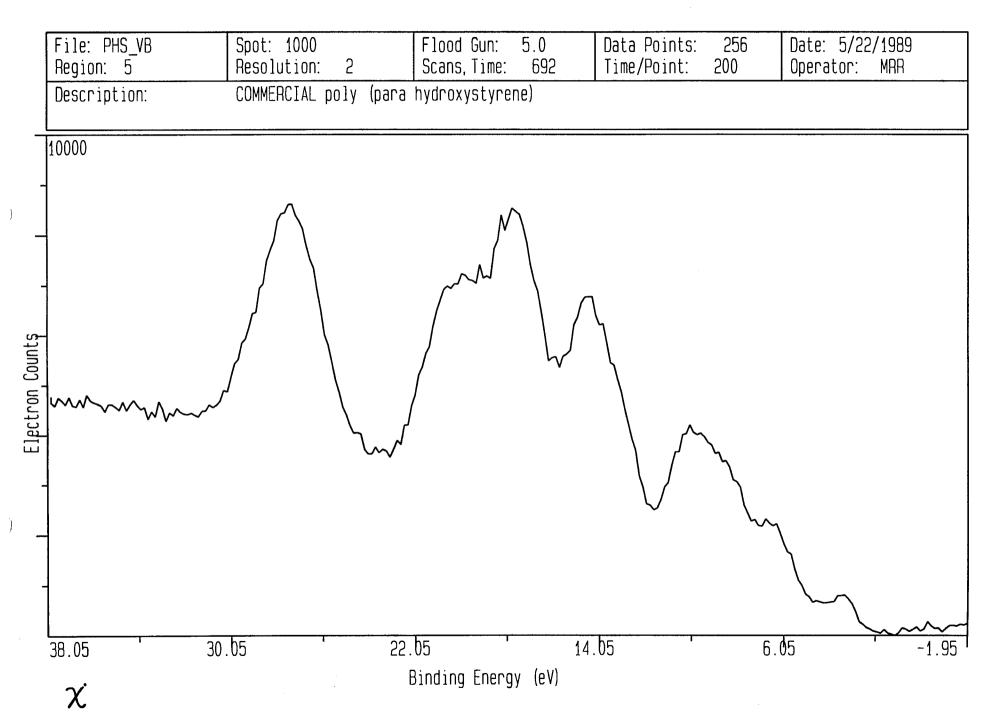
MRR

	Corrected	Exper.	Sens	Norm	Relative	
Signal	BE	BE	Factor	Area	Area	Atom %
O Auger	974.4	969.4	0.00	5077	0	
* 0 1s	529.0	524.0	2.51	20720	8261	11.65
* C 1s	280.0	275.0	1.01	62999	62650	88.35
0 2s	21.4	16.4	0.16	482	2989	









Poly - 4-Methyl Styrene (2% w/v toluene)

[CAS# 24936-41-

Detailed Surface Composition Table

File name: 4MPS_SU.MRS

Region:

Description: POLY (4-METHYL STYRENE); DISSOLVED IN 2% W/V IN TOLUENE; MeOH Precipitated

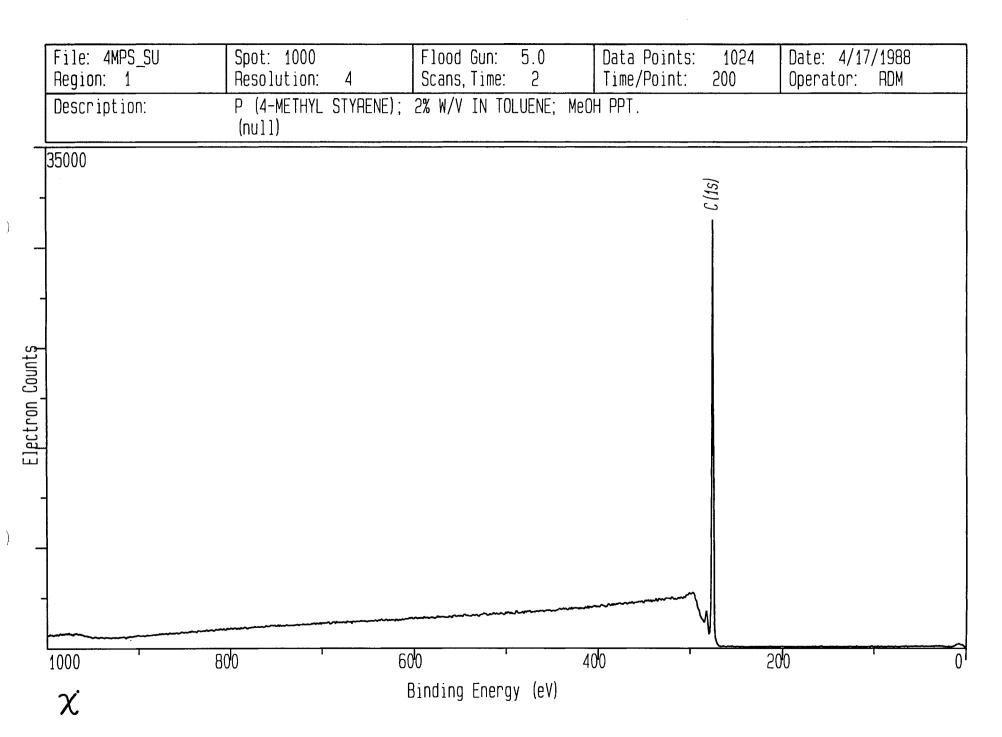
Operator: RDM

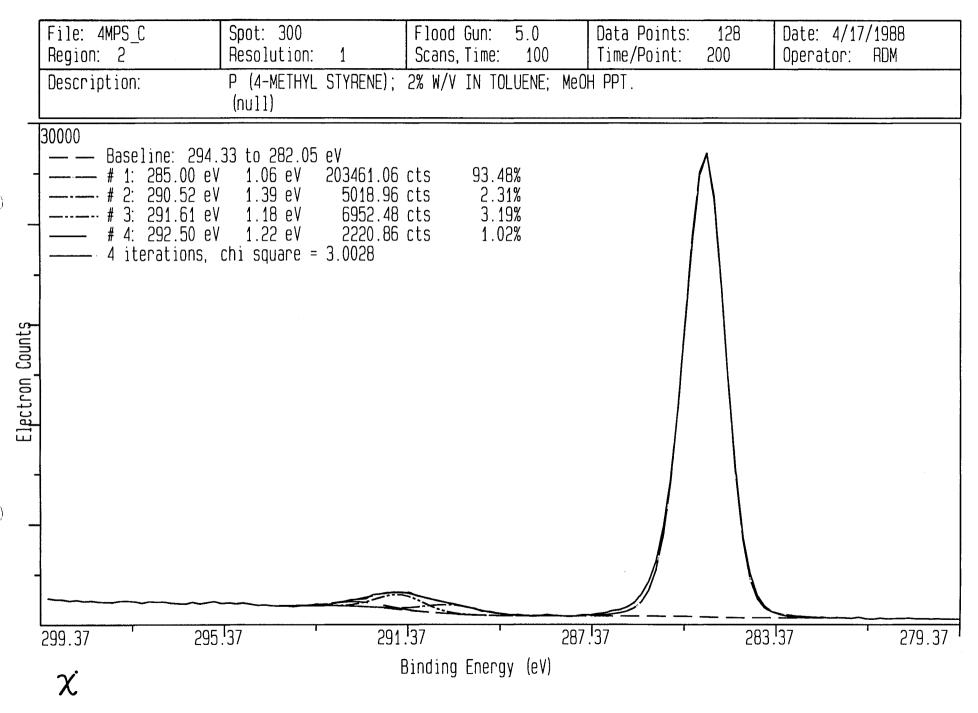
Date: Sun April 17 1988

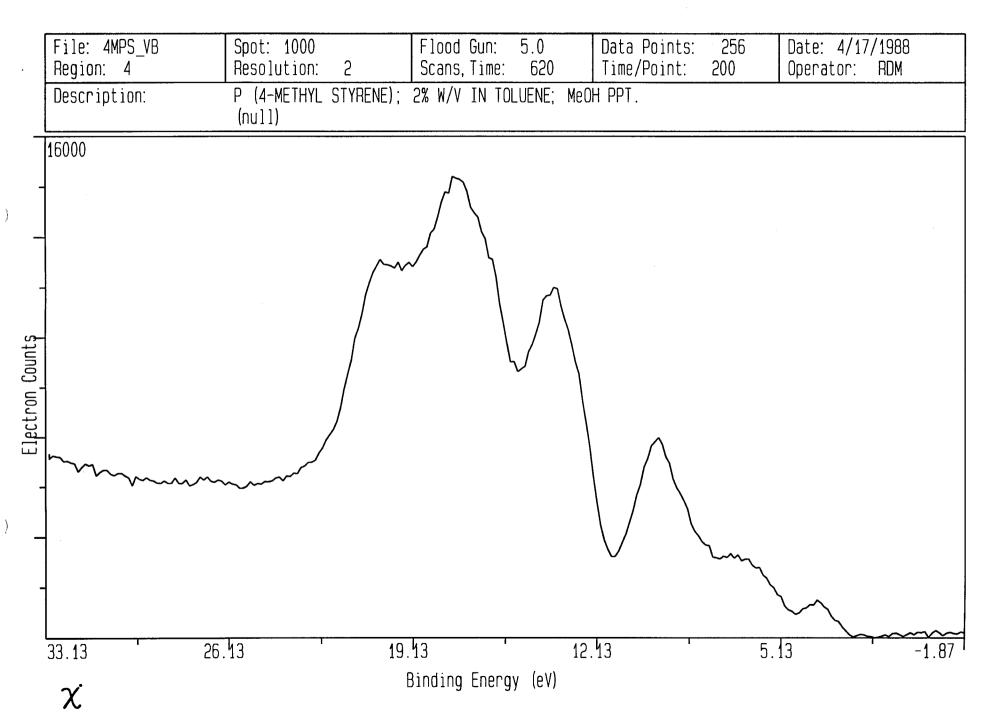
 Corrected
 Exper.
 Sens
 Norm
 Relative

 Signal
 BE
 BE
 Factor
 Area
 Area
 Atom %

 * C 1s
 280.0
 275.0
 1.01
 151943
 151098
 100.00







Poly – 4-Octyl Styrene

Detailed Surface Composition Table

File name: POS SU.MRS

Region:

1

Description: NAKAHAMA, WC11-18-10, NH-49, poly 4-Octyl-Styrene

Operator:

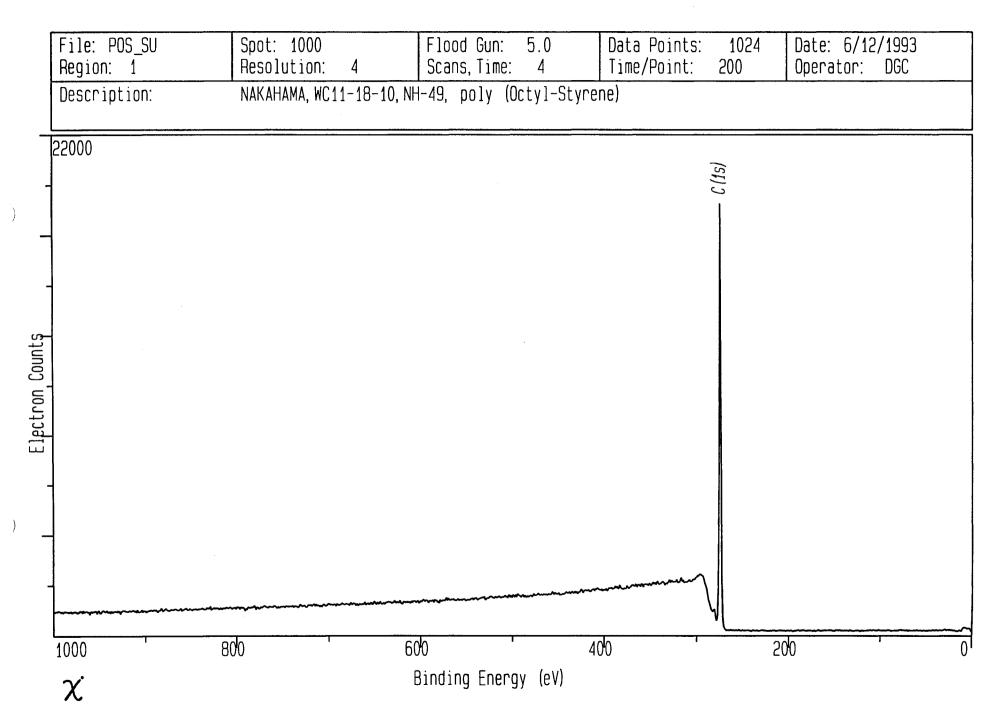
DGC

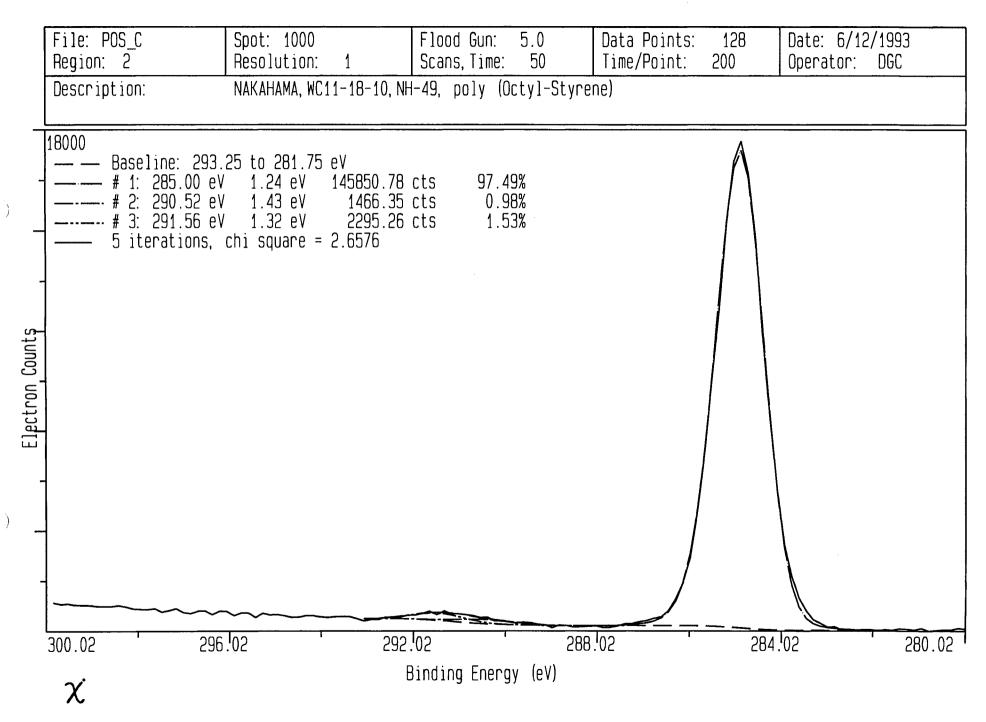
Date:

Sat Jun 12 1993

Corrected Exper. Sens Norm Relative

<u>Signal</u> <u>BE</u> <u>BE</u> <u>Factor</u> <u>Area</u> <u>Area</u> <u>Atom</u> <u>%</u> * C 1s 278.9 273.9 1.01 49584 49277 100.00





Poly - 4-Vinyl Phenol (TFAA derivative)

Detailed Surface Composition Table

File name: TFAA SU.MRS

Region:

Description: Poly(4-VINYL PHENOL)-TFAA Deriv. FROM 4% DIOXANE

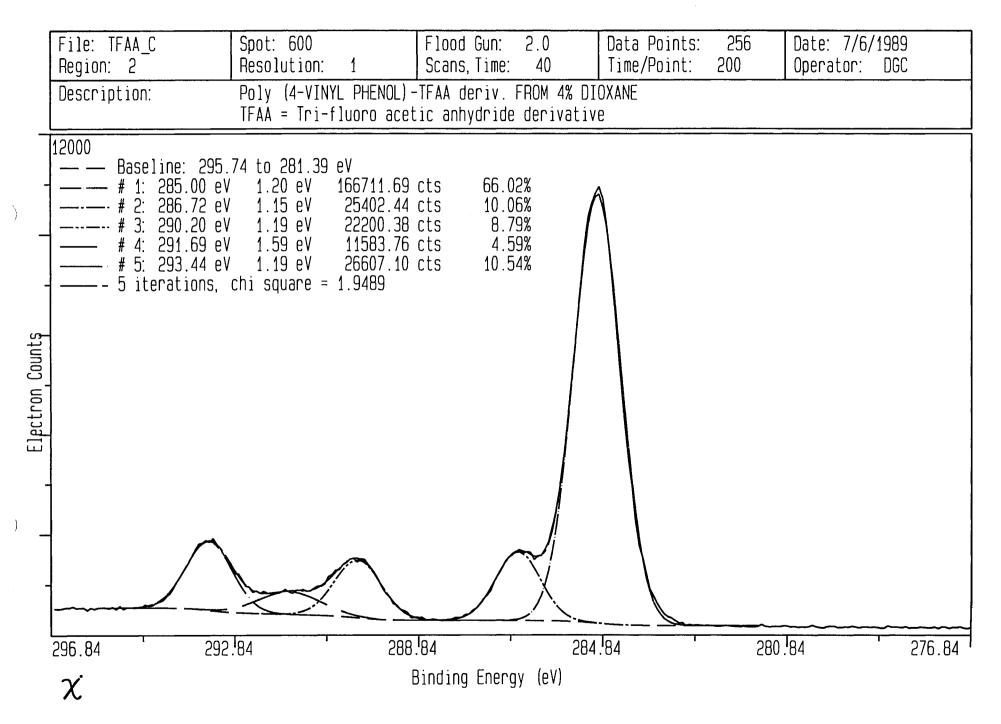
TFAA = Tri-fluoro acetic anydride derivative

Operator: DGC

Date: Thu Juul 6 1989

	Corrected	Exper.	Sens	Norm	Relative	
Signal	BE	BE	Factor	Area	Area	Atom %
O Auger	976.3	$9\overline{74.3}$	0.00	4604	0	
F Auger	859.0	857.0	0.00	5413	0	
F Auger	831.0	829.0	0.00	31521	0	
* F 1s	685.6	683.6	3.34	64890	19427	20.90
* 0 1s	530.5	528.5	2.50	28943	11577	12.46
* C 1s	281.0	279.0	1.00	62128	61926	66.64
F 2s	29.3	27.3	0.24	6224	25874	

	File: TFAA_SU Region: 1	Spot: 1000 Resolution:	4	Flood Gun: Scans, Time:	2.0	Data Points: Time/Point:	1024 200	Date: 7/6/1989 Operator: DGC	
	Description:	Poly(4-VINYL TFAA = Tri-fl	PHENOL) -1	FAA Deriv. F	ROM 4% DIC	XANE		1 450, 4401. 800	
_	12000								
<u>-</u>		F (15)							
)									
_						-	C (15)		
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Electron, Counts									
ron C				<u></u>					
Elect				- 0(15)					
_	Auger) F (Auger) >> F (Auger)								·
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-		•	Lymmer	mondy	markemannen	Manney			F (2s)
	1000 8	00	60)	4	00	2	00	<u>~~</u>
	χ			Binding Energ					



Biomer (2% in HFIP)

Detailed Surface Composition Table

File name: BIOM SU.MRS

Region:

Description: WC8-15-VII-1,2% BIOMER IN HFIP (2X PPTED) ON GLASS (HFIP = Hexafluoro isopropanol)

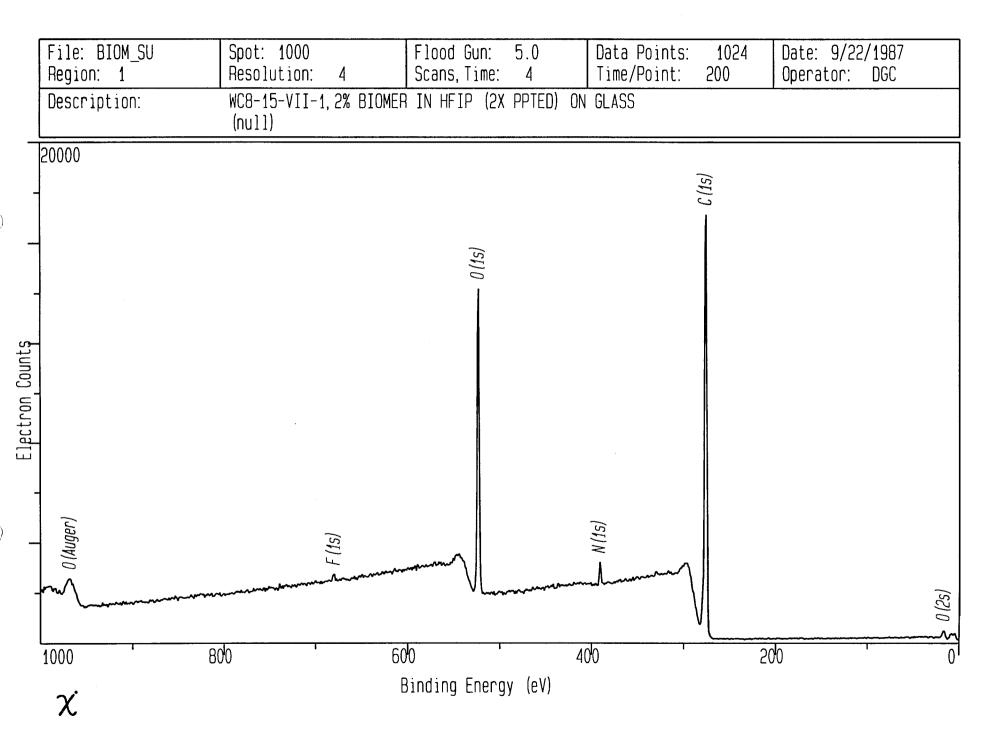
Operator:

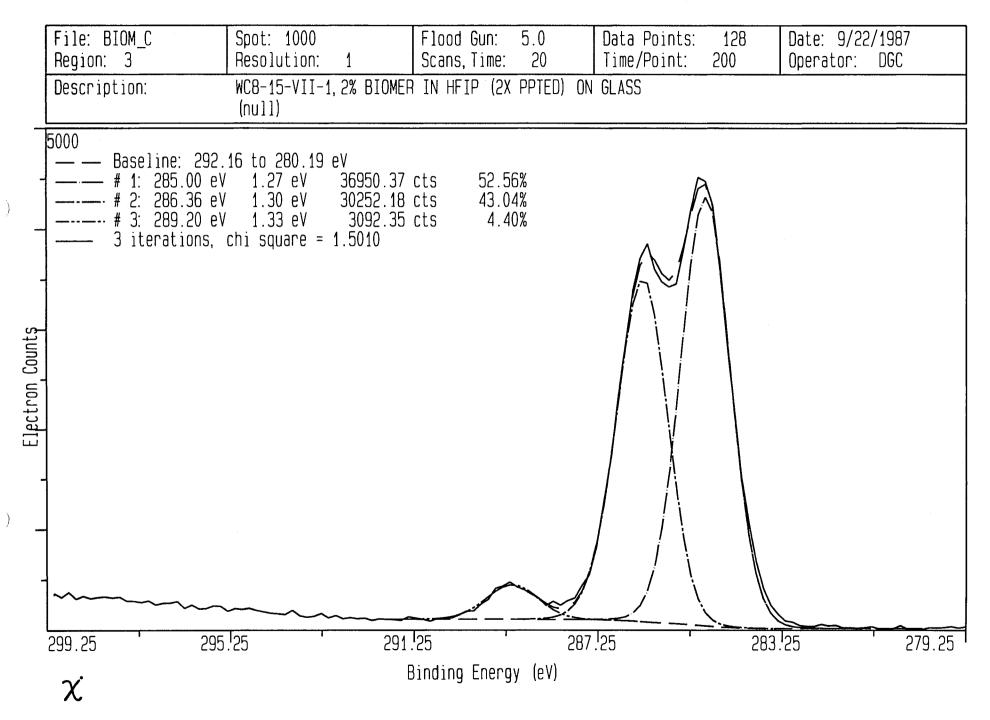
DGC

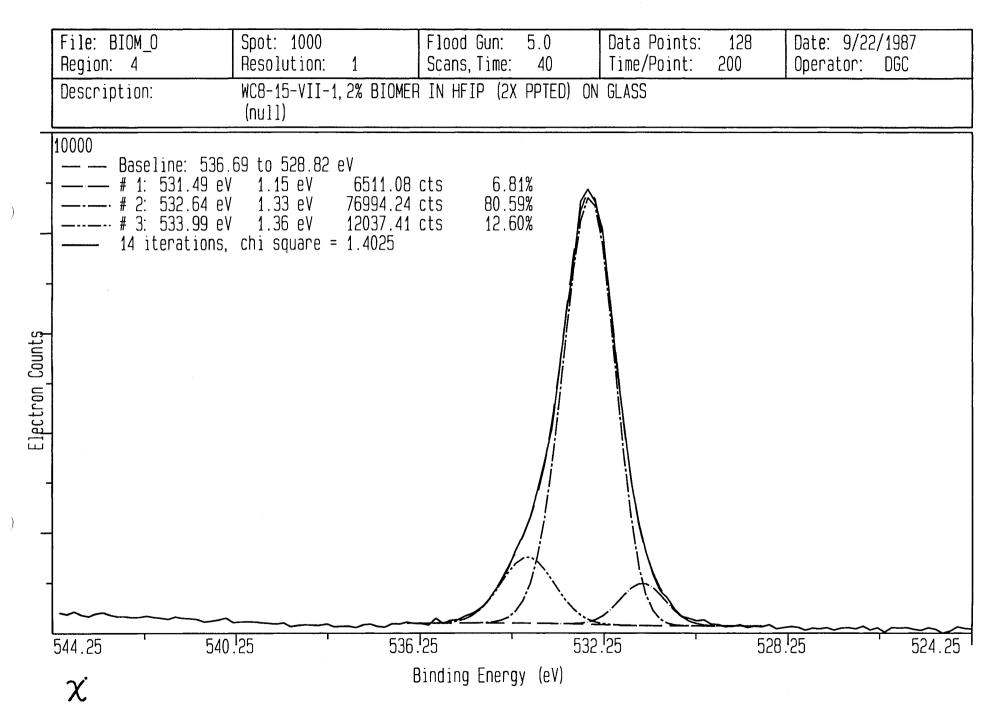
Date:

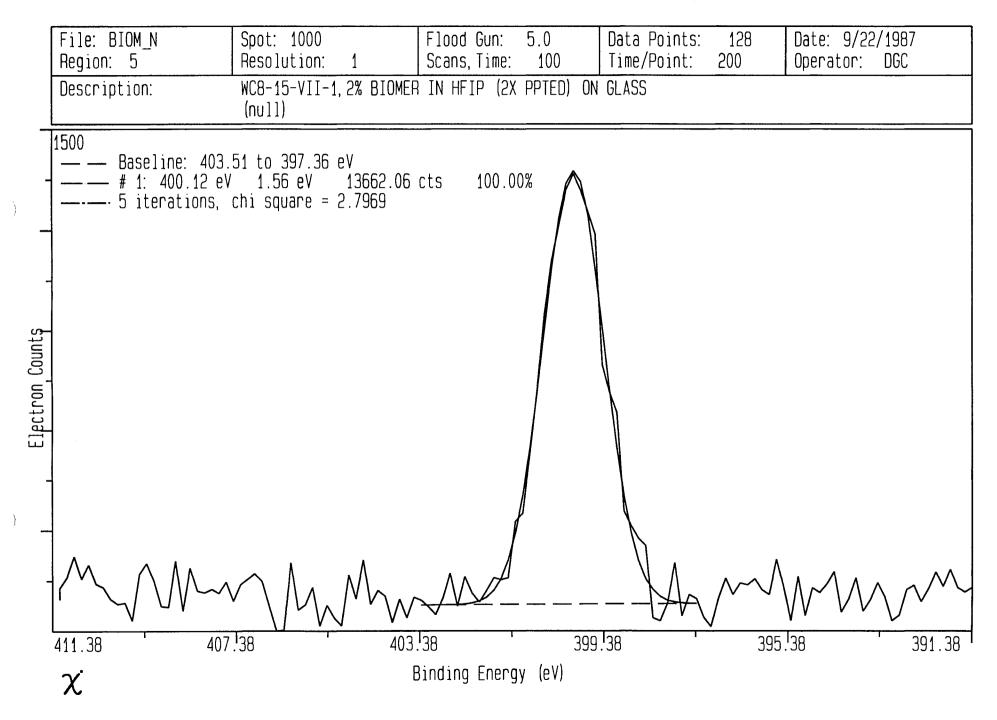
Tue Sep 22 1987

	Corrected	Exper.	Sens	Norm	Relative	
Signal	BE	BE	Factor	Area	Area	Atom %
0 Auger	$97\overline{2.9}$	9 67 .9	0.00	11289	o	
* F 1s	684.5	679.5	3.36	979	291	0.34
* 0 1s	527.9	522.9	2.51	40547	16153	19.07
* N 1s	395.2	390.2	1.70	2430	1433	1.69
* C 1s	280.4	275.4	1.01	67207	66848	78.90
0 2s	20.8	15.8	0.16	1447	8976	









Cellulose (filter paper)

Detailed Surface Composition Table

File name: CELL_SU.MRS

Region:

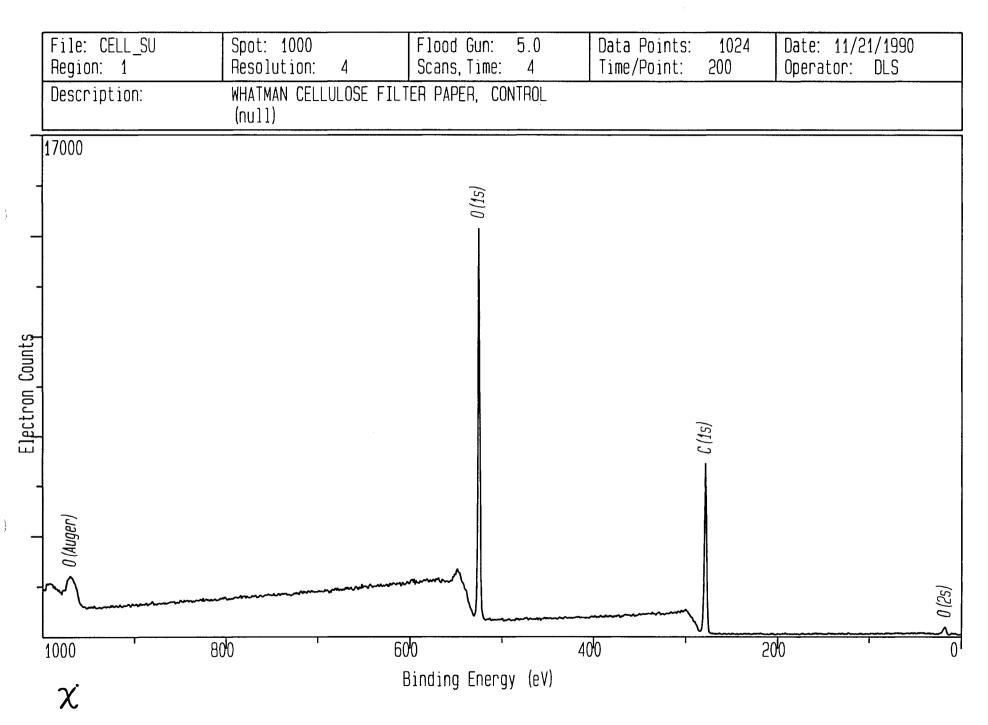
Description: WHATMAN™ CELLULOSE FILTER PAPER, CONTROL

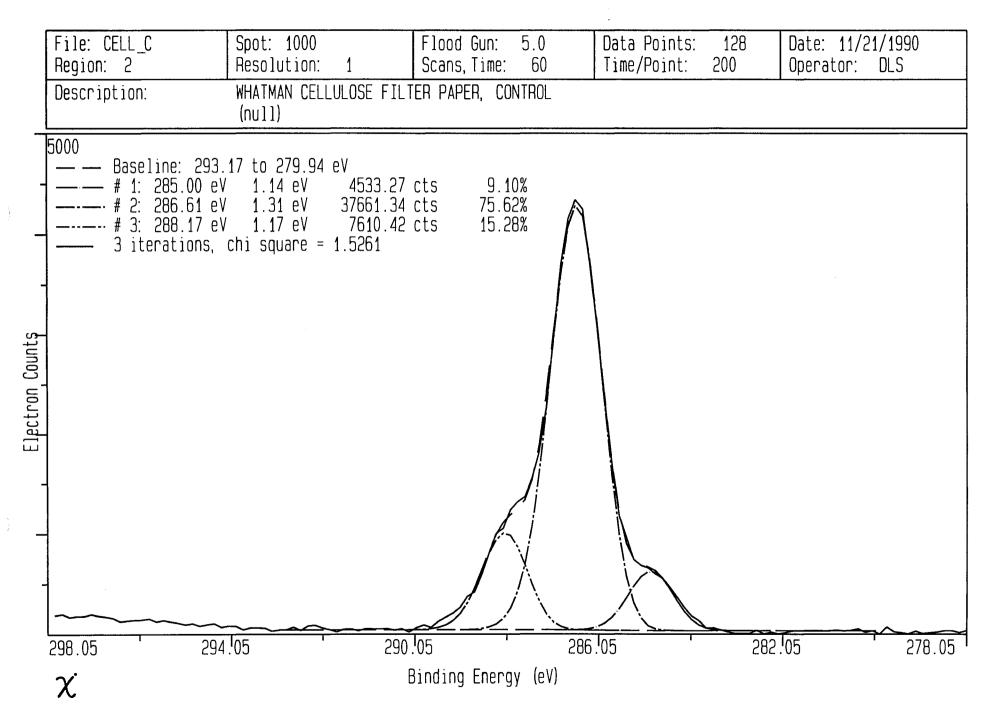
Operator:

DLS

Date: Wed Nov 21 1990

	Corrected	Exper.	Sens	Norm	Relative	
Signal	BE	BE	Factor	Area	Area	Atom &
O Auger	$97\overline{4.7}$	9 69 .7	0.00	11021	o	
* 0 1s	529.4	524.4	2.51	39563	15779	43.71
* C 1s	282.8	277.8	1.00	20401	20320	56.29
0.26	21 8	16.8	0.16	1540	9555	





Poly - Dimethylsiloxane (as received)

Detailed Surface Composition Table

File name: PDMS SU.MRS

Region:

Description: PDMS VB & Hi Res, NIH STANDARD, TOA=0

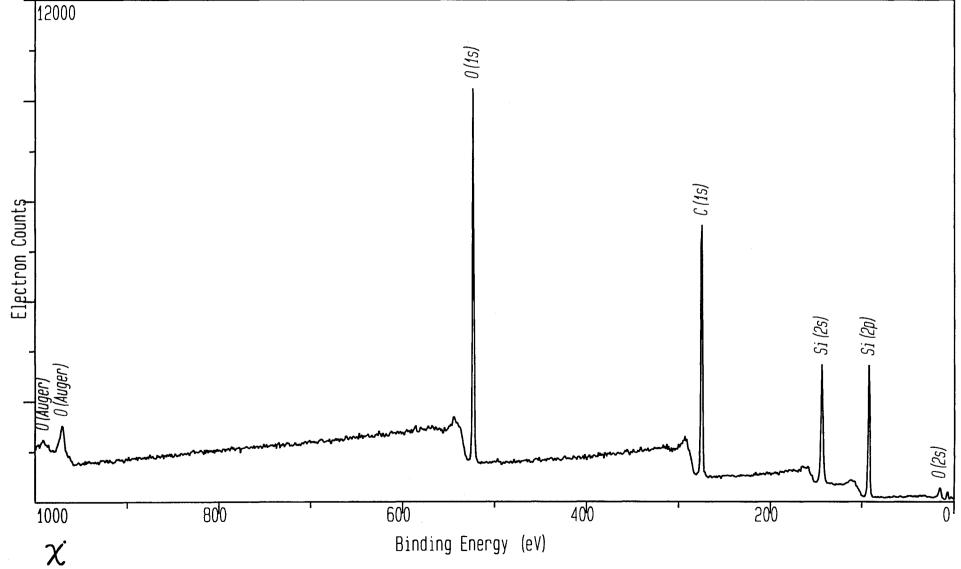
Poly-dimethyl siloxane (National Institute Health)

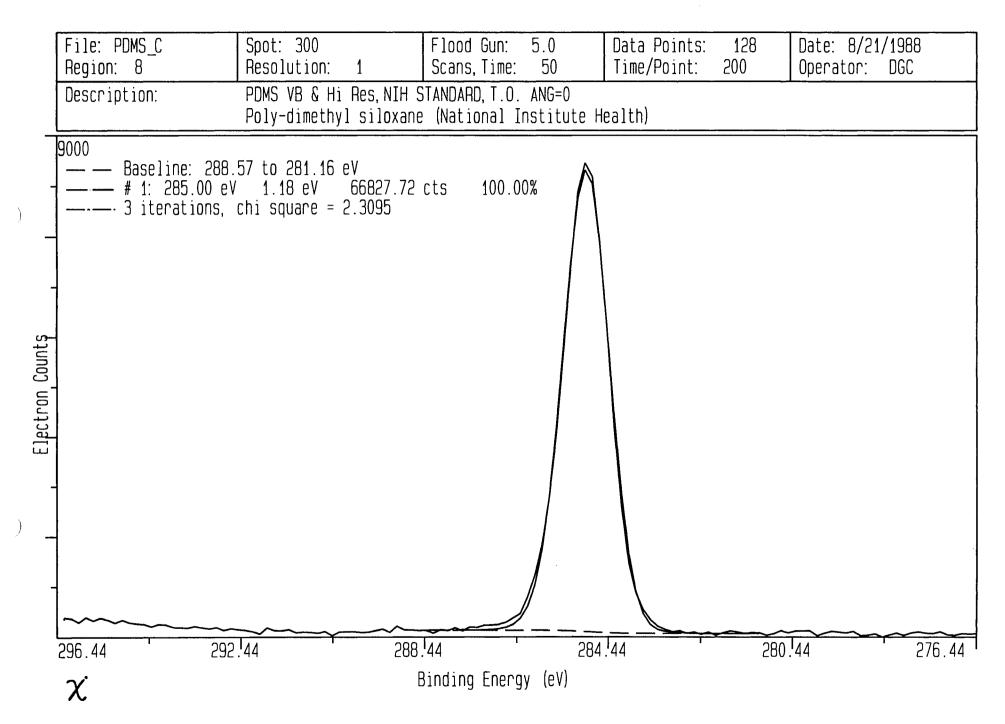
Operator: DGC

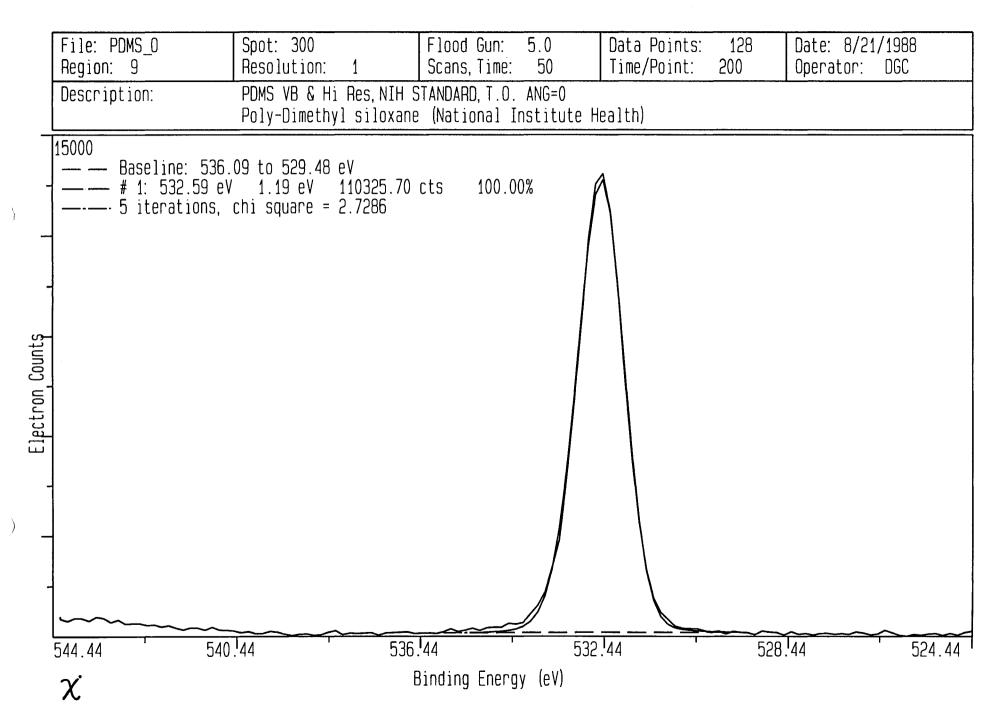
Date: Sun Aug 21 1988

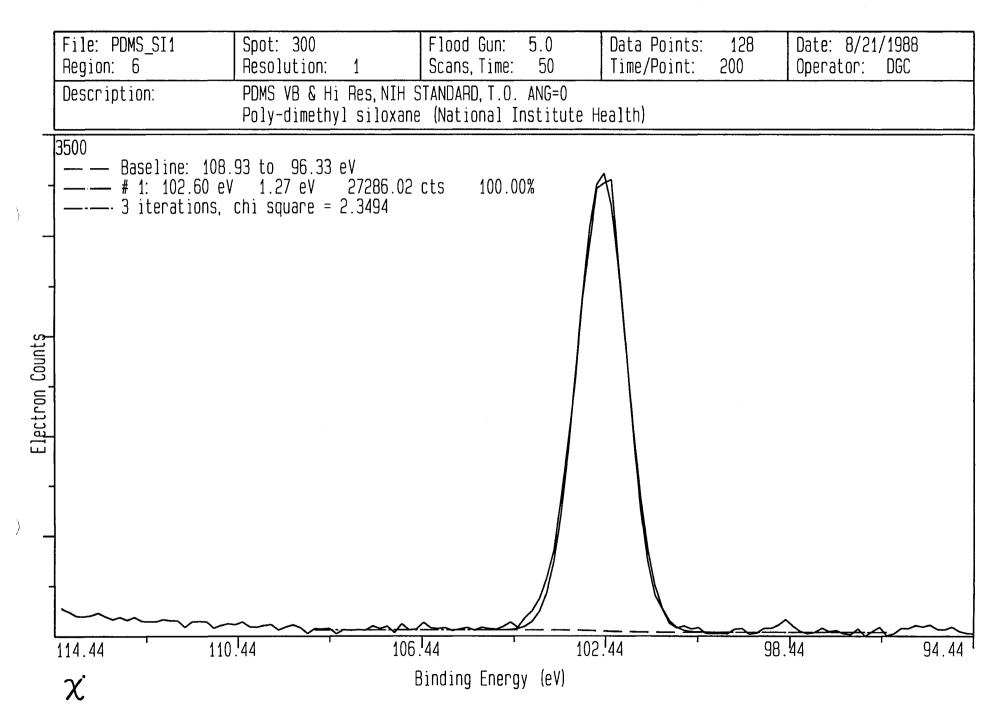
	Corrected	Exper.	Sens	Norm	Relative	
Signal	BE	BE	Factor	Area	Area	Atom %
O Auger	$99\overline{6.1}$	$9\overline{91}.1$	0.00	4784	0	
O Auger	975.4	970.4	0.00	13996	0	
* 0 1s	527.5	522.5	2.51	45868	18267	24.64
* C 1s	279.4	274.4	1.01	36432	36217	48.86
* Si2s	147.9	142.9	1.03	20283	19645	26.50
Si2p	96.8	91.8	0.91	17827	19662	
0 2s	19.4	14.4	0.16	2480	15373	

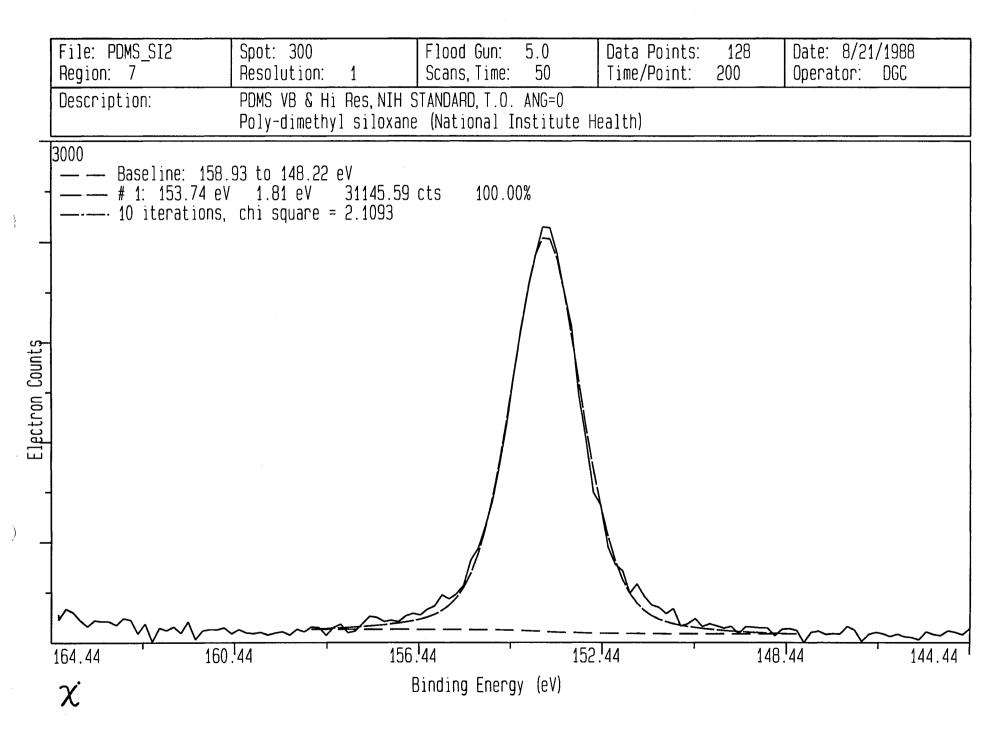
File: PDMS_SU	Spot: 1000	Flood Gun: 5.0	Data Points:	1024	Date: 8/21/1988
Region: 1	Resolution: 4	Scans, Time: 2	Time/Point:	200	Operator: DGC
Description:	•	H STANDARD, T.O. ANG=0 ane (National Institute	e Health)		



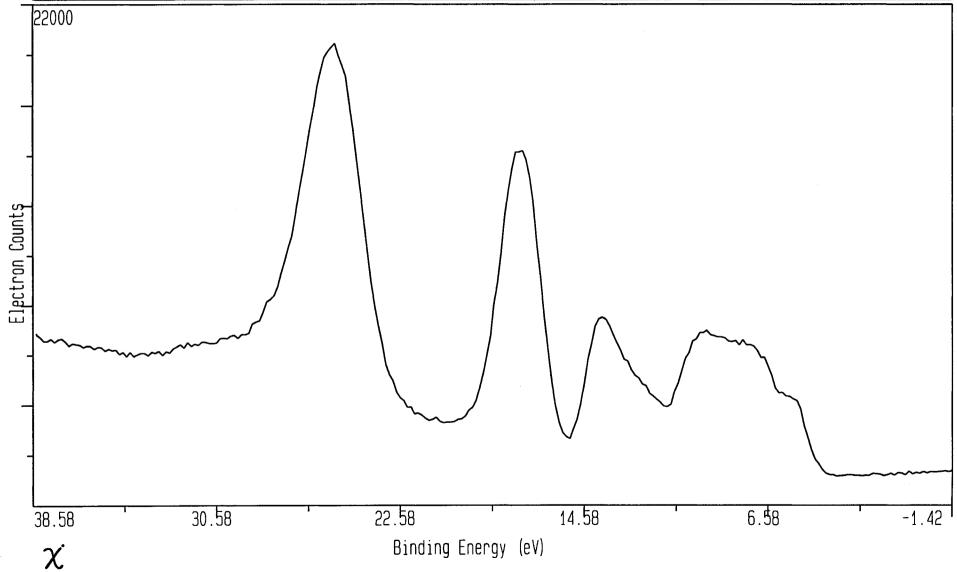








File: PDMS_VB	Spot: 1000	Flood Gun: 5.0	Data Points: 256	Date: 8/21/1988
Region: 11	Resolution: 2	Scans, Time: 530	Time/Point: 200	Operator: DGC
Description:	PDMS VB & Hi Res, NIH S Poly-dimethyl siloxano	STANDARD, T.O. ANG=0 e (National Institute H	lealth)	



Poly - Ether Ether Ketone (pressed wafer)

Detailed Surface Composition Table

File name: PEEK SU.MRS

DGC

Region:

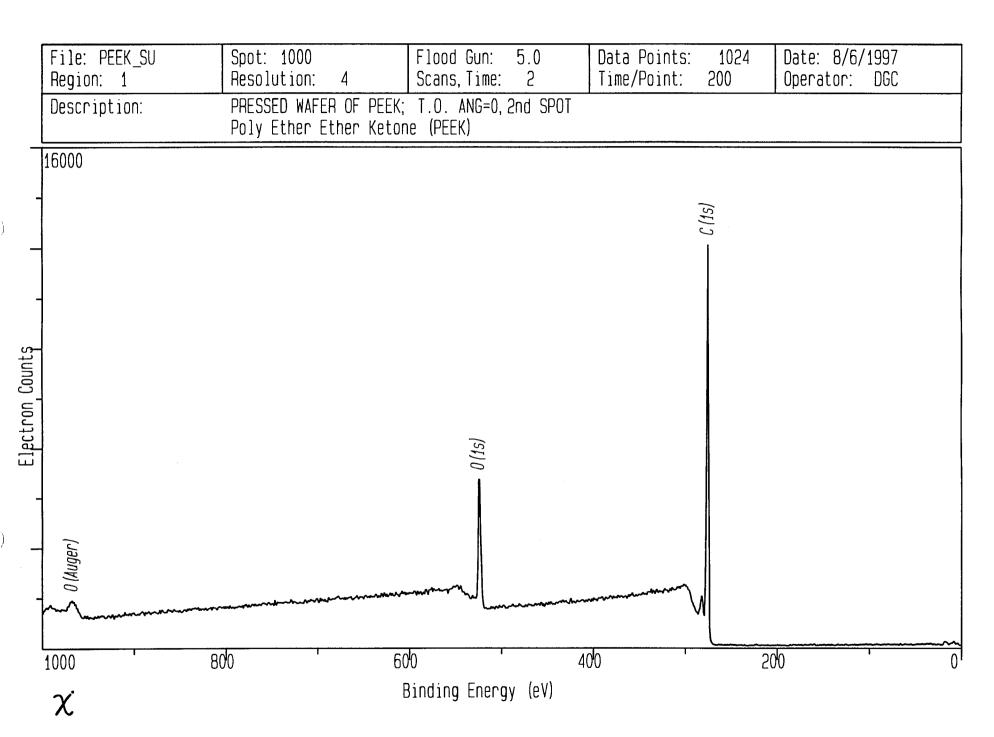
Description: PRESSED WAFER OF PEEK; TOA=0, 2nd SPOT, PEEK = Poly Ether Ether Ketone

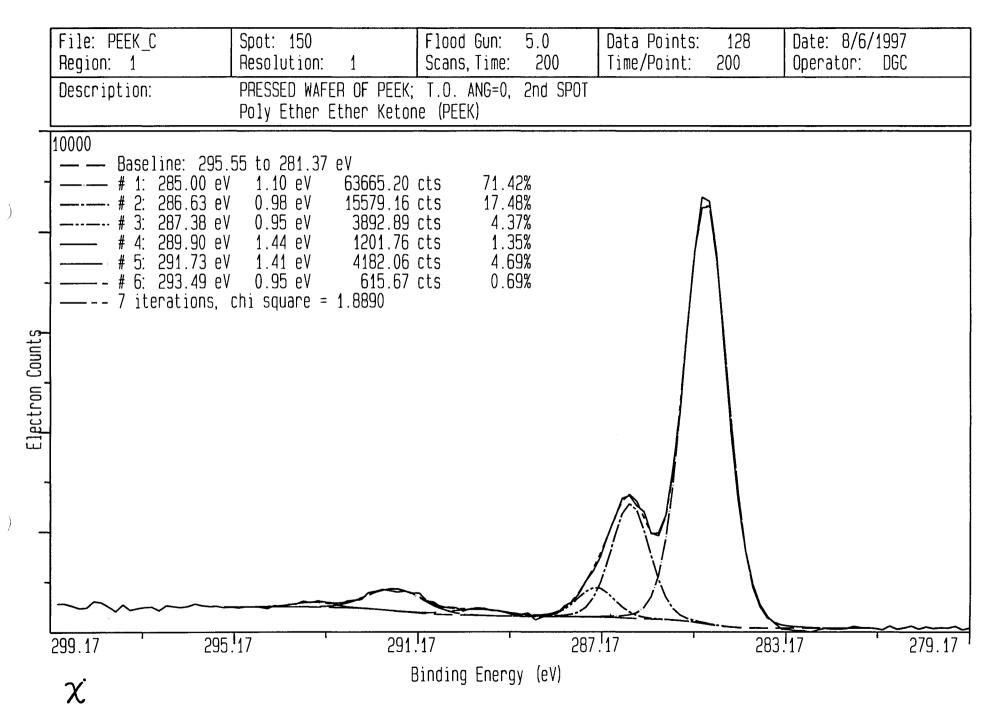
Operator:

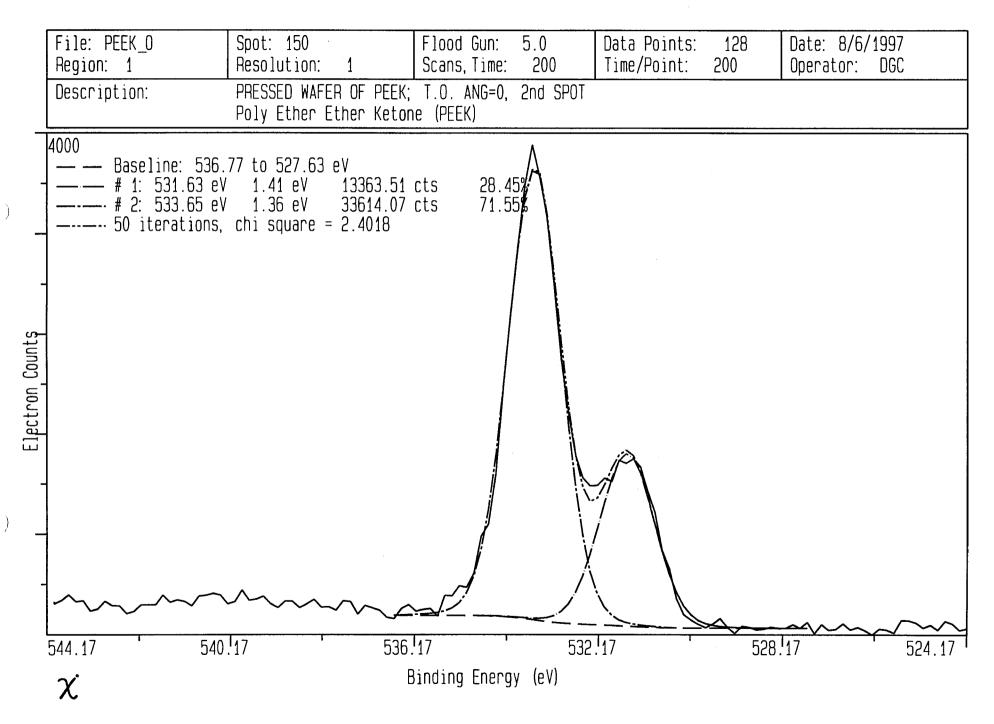
Date:

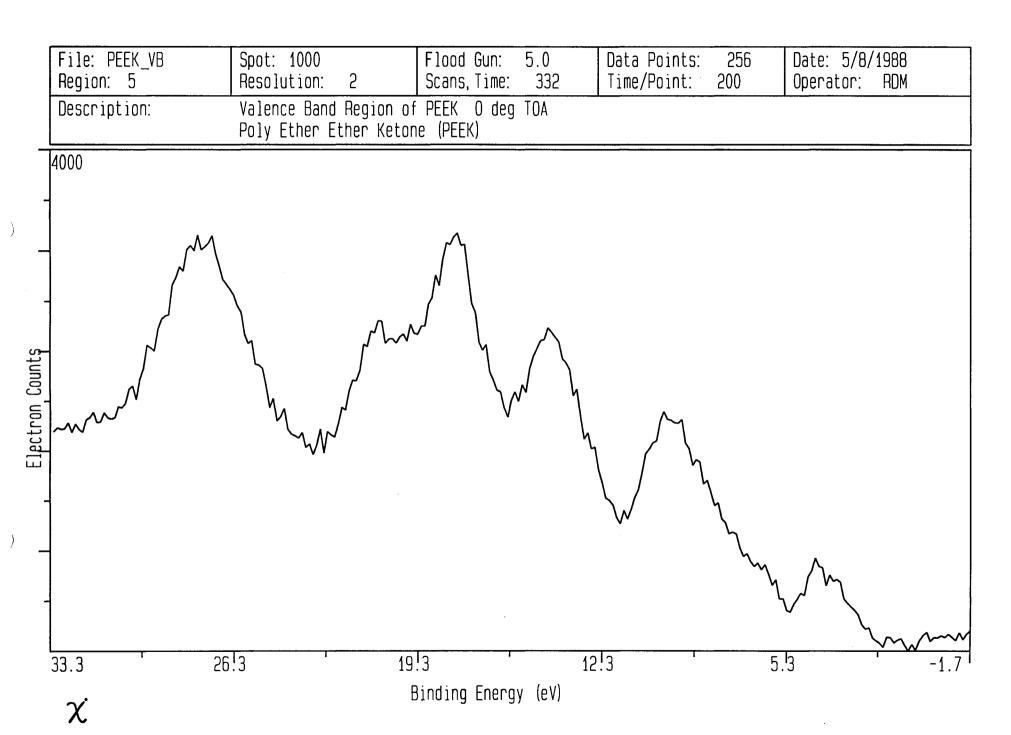
Wed Aug 6 1997

	Corrected	Exper.	Sens	Norm	Relative	
Signal	BE	BE	Factor	Area	Area	Atom %
0 Auger	972.8	967.8	0.00	10580	0	
* 0 1s	529.0	524.0	2.51	34489	13751	14.10
* C 1s	280.1	275.1	1.01	84211	83748	85.90









Poly - Ethyl Acrylate

Detailed Surface Composition Table

File name: PEA_SU.MRS

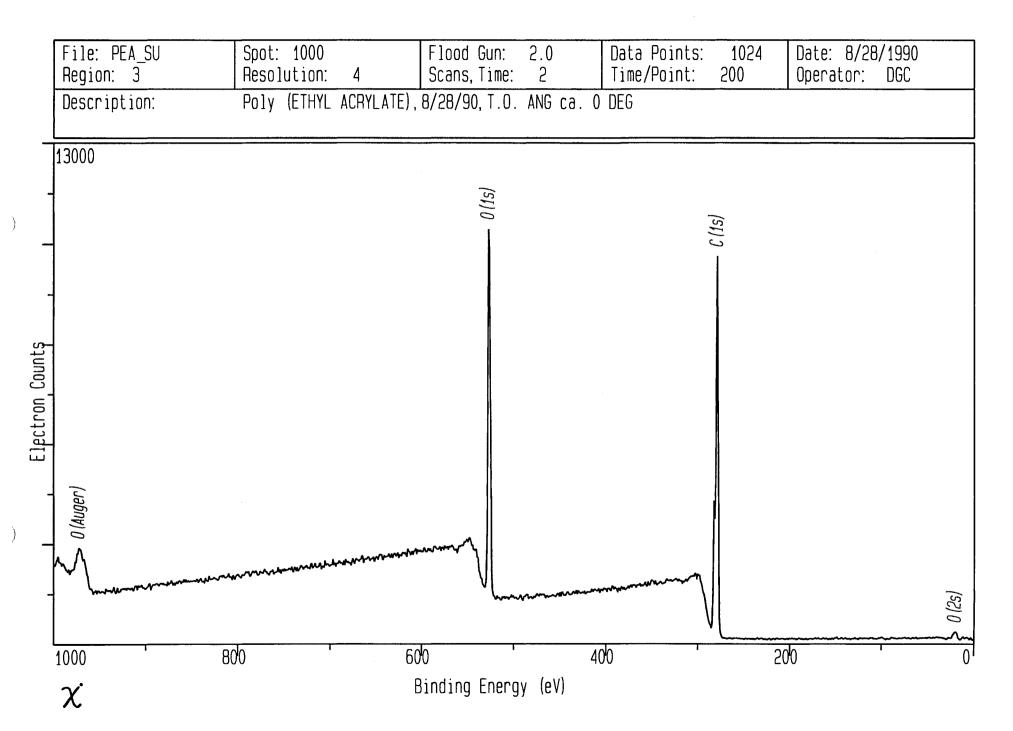
Region: 3

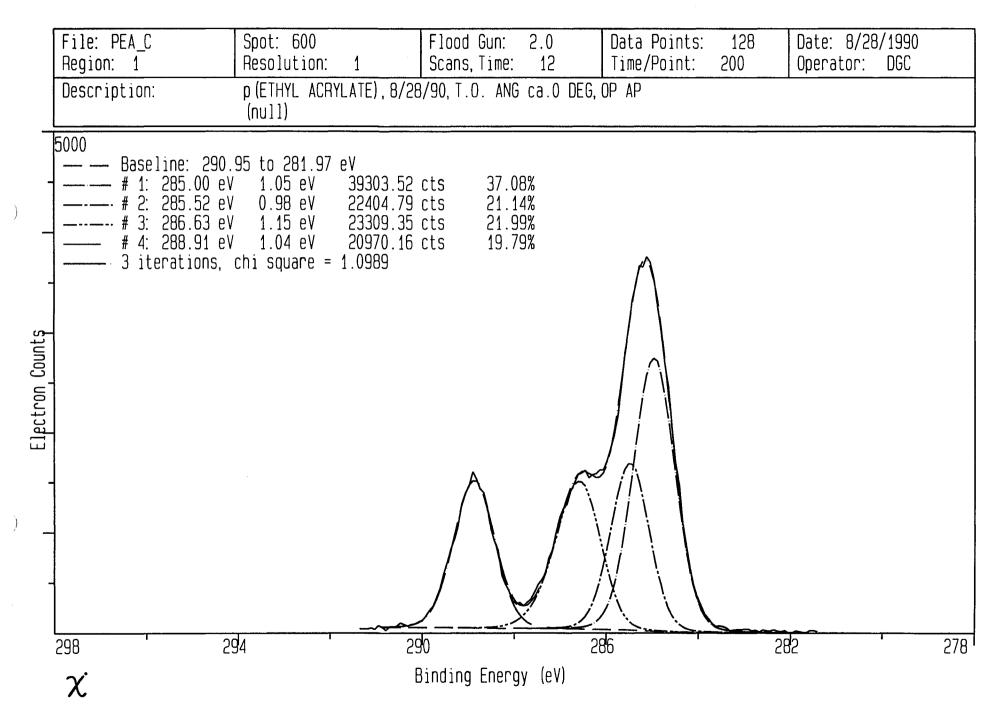
Description: Poly (ETHYL ACRYLATE), 8/28/90, TOA = 0 ca.

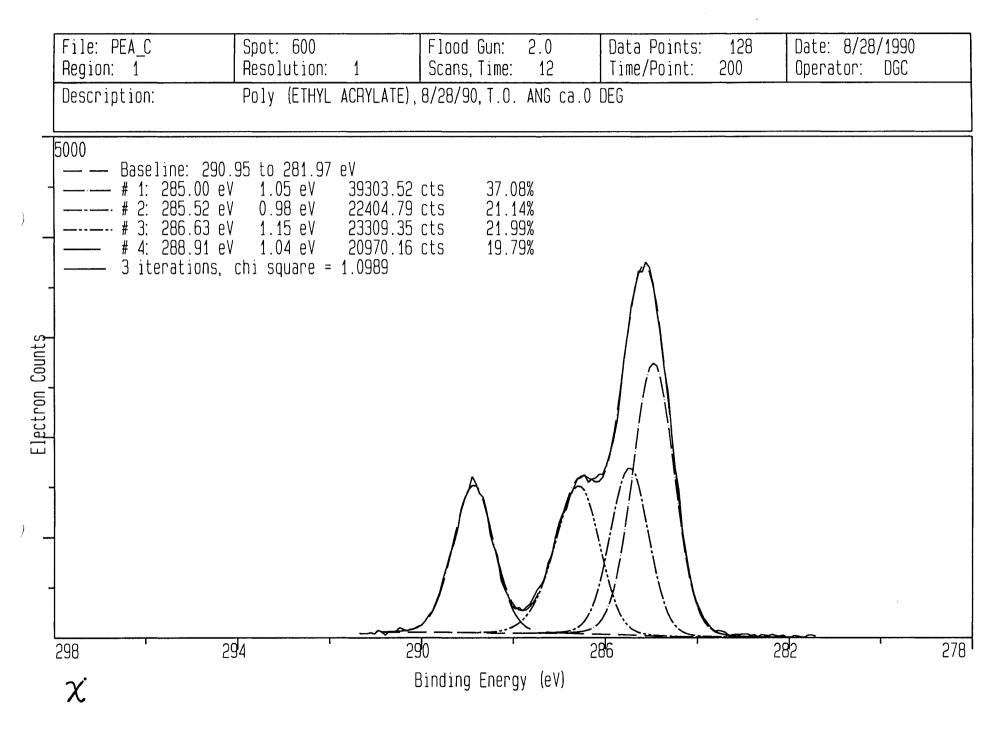
Operator: DGC

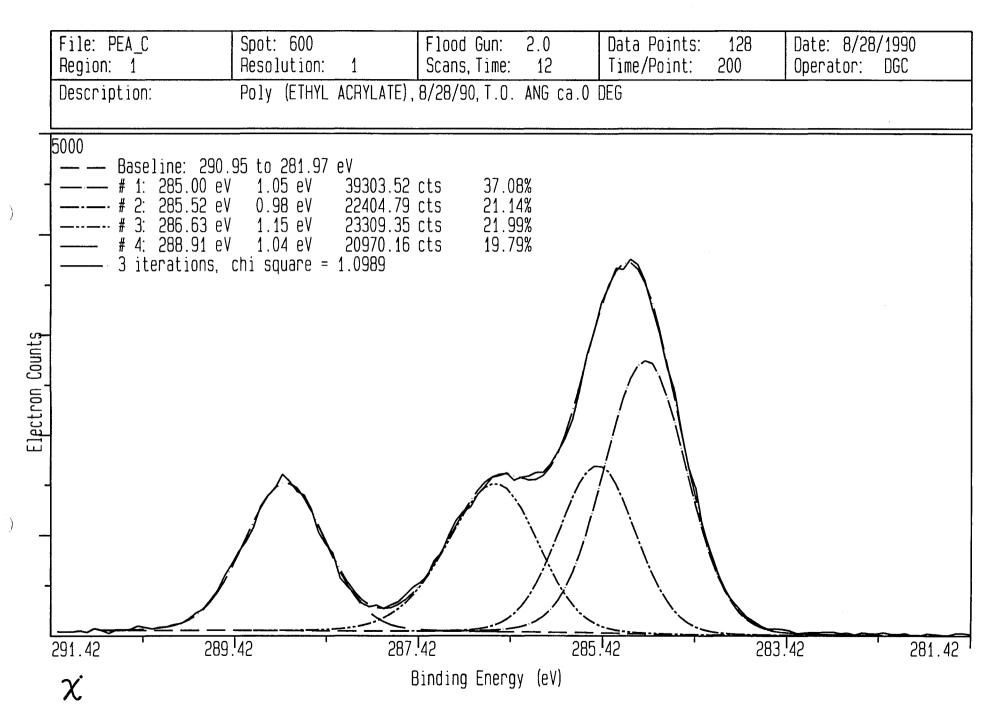
Date: Tue Aug 28 1990

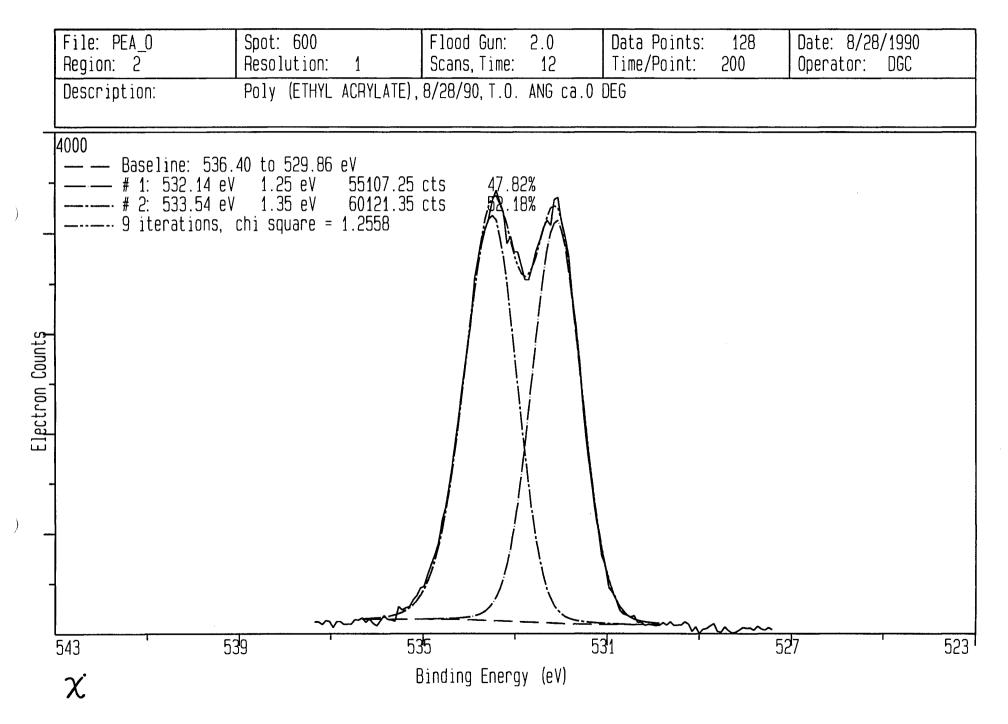
	Corrected	Exper.	Sens	Norm	Relative	
Signal	BE	BE	Factor	Area	Area	Atom %
O Auger	$97\overline{4.3}$	972.3	0.00	24375		
* 0 1s	528.0	526.0	2.50	74990	29944	27.33
* C 1s	280.1	278.1	1.00	79934	79 6 31	72.67
0 2s	21.4	19.4	0.16	3014	18721	



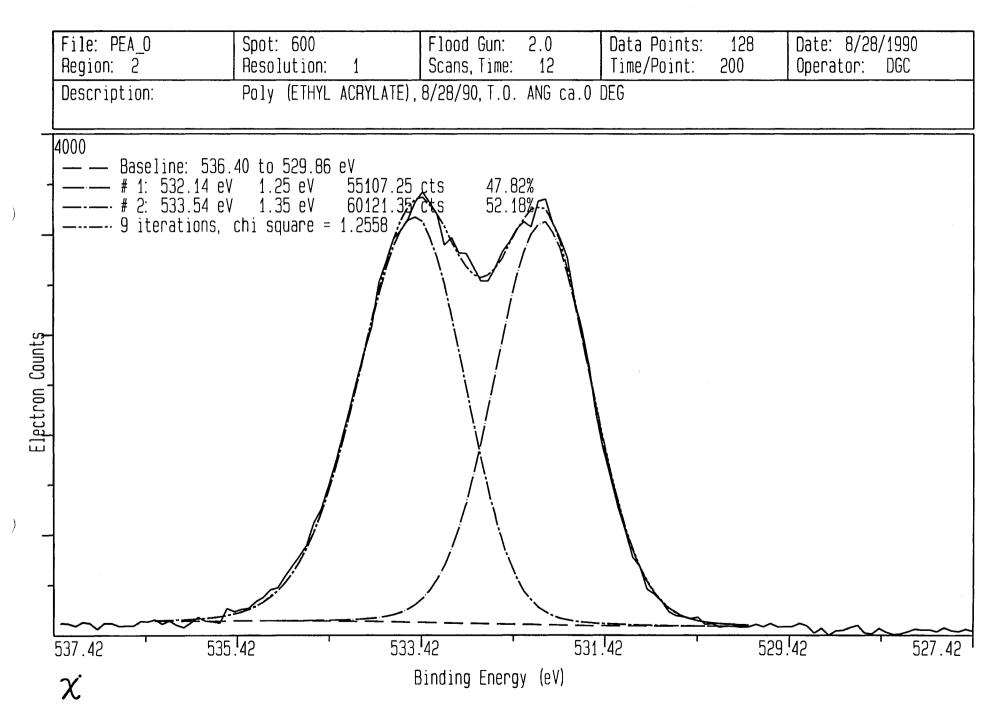








67



Poly Ethyl Methacrylate (film on glass)

Detailed Surface Composition Table

File name: EMA_SU.MRS

Region: 1

Description: poly-EMA (HOMEMADE) ON GLASS, WC9-96-2, TOA = 55

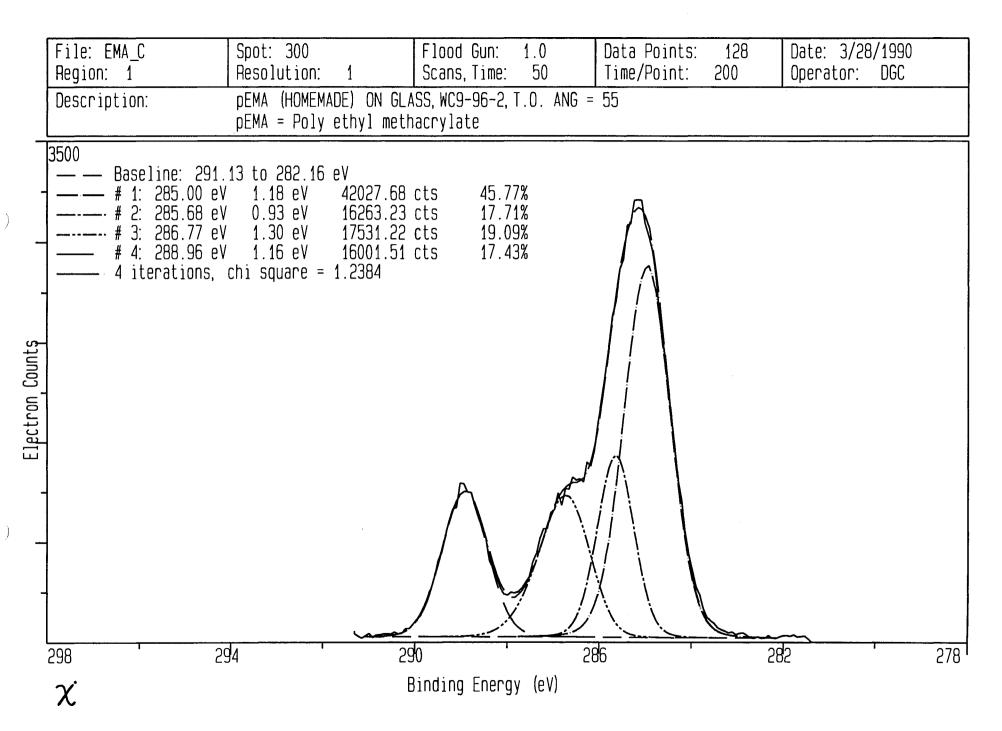
PEMA = Poly Ethyl Methacrylate

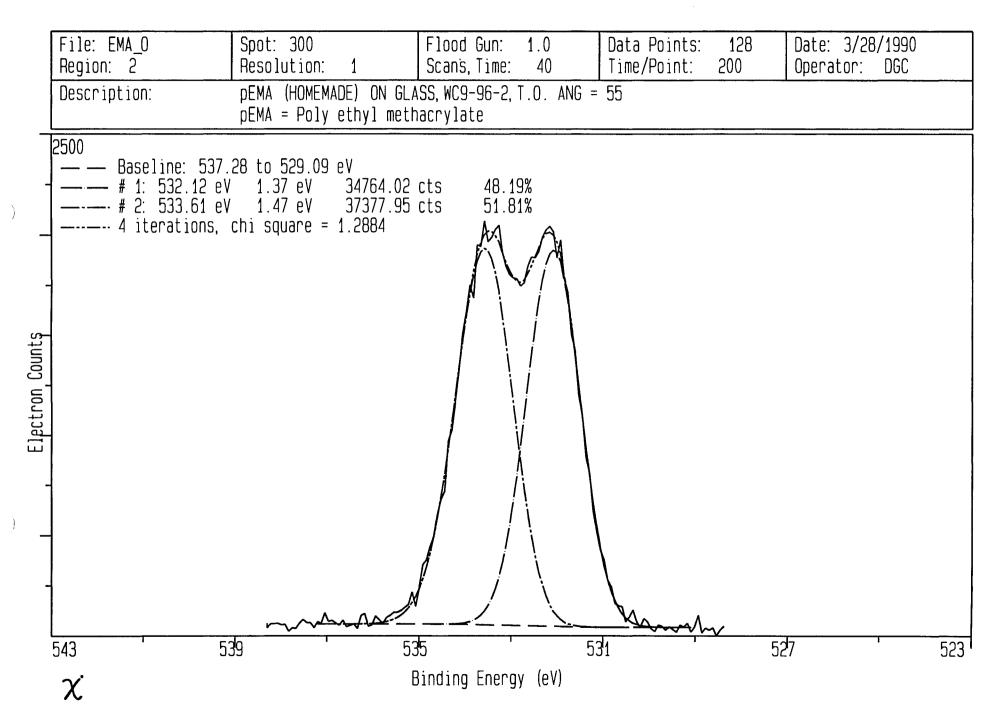
Operator: DGC

Date: Wed Mar 28 1990

	Corrected	Exper.	Sens	Norm	Relative	
Signal	BE	BE	Factor	Area	Area	Atom %
0 Auger	974.6	973.6	0.00	11165	—_о	
* 0 1s	528.5	527.5	2.50	32313	12916	23.83
* C 1s	280.5	279.5	1.00	41412	41291	76.17
0 2s	22.3	21.3	0.16	1343	8349	

File: EMA_SU Region: 1	Spot: 1000 Resolution:	Flood Gun: 4 Scans, Time:	1.0 Data Po		Date: 3/28/1990 Operator: DGC
Description:	pEMA (HOMEMAD	E) ON GLASS, WC9-96-2, thyl Methacrylate		5111.	- oper deer
6000					
_		- 0(15)		— C (1s)	
Electron Counts					
O (Auger)		a southware			
- Ammuni	shipmenth programment of the programment of the second	annyment !	wayungangangangangangan	manny	0 (25)
1000	80'0	600	400	2	
X		Binding Energ	gy (eV)		





Poly - Ethylene (freshly pressed)

Detailed Surface Composition Table

File name: PE_SU.MRS

Region: 1

Description: Poly-Ethylene - Freshly pressed between Al sheets

Operator:

DGC

Date:

Tue Nov 8 1988

Corrected Exper. Sens Norm Relative

<u>Signal</u> <u>BE</u> <u>BE</u> <u>Factor</u> <u>Area</u> <u>Area</u> <u>Atom</u> <u>%</u>
* C 1s 280.7 275.7 1.01 86076 85634 100.00

Appendix

Full Overlay of Spectra from 14 Hour Damage Studies

Index by Monomer Name

Acetal (PA)	
Acrylic Acid (PAA)	A-6
Acrylonitrile (PAN)	A-9
Amide Resin.	A-12
Butene (PB)	A-14
Carbonate (PC)	A-16
Ethylene (PE, HDPE)	A-19
Ethylene Oxide (PEO)	A-21
Ethylene Terephthalate (PET, Mylar TM)	A-24
Imide (Kapton TM)	A-27
Isohexene	A-31
Methyl Methacrylate (PMMA)	A-32
Nitrocellulose (filter paper)	A-30
Nylon 6	
Phenylene Sulfide (PPS)	A-44
Propylene (PP)	A-47
Styrene (PS)	A-49
Sulfone (PSu)	A-54
Tetra-Fluoro Ethylene (PTFE, Teflon™)	A-53
Vinyl Acetate (PVA)	A-5:
Vinyl Chloride (PVC)	A-58
Vinylidene di-Fluoride (PVdF)	A-60

APPENDIX "A"

ALPHABETICAL INDEX OF XPS SPECTRA IN VOLUME FOUR POLYMERS & POLYMERS DAMAGED BY X-RAYS

POLYMERS & POLYMERS DAMAGED BY X-RAYS

Section 1 – Polymers Purified by the National ESCA and Surface Analysis Center for Biomedical Problems

α-methyl styrene (AMPS)	
2-chloro ethyl methacrylate (CLMA)	7
2-hydroxy ethyl methacrylate (HEMA)	12
2-hydroxy ethyl methacrylate (HEMA) trimethyl-silane derivative	16
4,4'-dimethoxy benzophenone (4,4-DBP)	20
4-ethoxy styrene (PES)	24
4-hydroxy styrene (PHS)	28
4-methyl styrene (PMPS)	33
4-octyl styrene (POS)	37
4-vinyl phenol – trifluoro acetic anhydride derivative	40
Biomer TM (2% in HFIP)	43
cellulose (Whatman™ filter paper) as received	
dimethyl siloxane (PDMS) as received	51
ether ether ketone (PEEK) pressed wafer	58
ethyl acrylate (PEA)	63
ethyl methacrylate (EMA) film on glass	70
ethylene (PE) freshly pressed	
ethylene glycol (PEG) 4000 flake	
ethylene terephthalate (PET, Mylar TM) 3X MeOH rinse	
methyl acrylate (PMA) 3% solution	
methyl methacrylate (PMMA) on copperfilm on disk	
methylene diisocynate/butane-diol copolymer (MDBD) 1:1 (urethane)	
methylene diisocynate/ butane-diol /propane-diamine terpolymer (MDBP) hard segment	
methylene di-isocynate/propane-diamine copolymer (MDPD) 1:1	
propylene glycol (PPG) 2000 (from MeOH)	
styrene (PS) 4% in toluene	
tetra fluoro ethylene (PTFE) cleaned (Teflon TM)	
tetra methylene glycol (PTMG) cast from CHCl ₃	
vinyl alcohol (PVA) 2.5% water solution	
vinyl chloride (PVC)	
vinylidene di-fluoride (PVDF) old	140

Section 2 - Commercial Polymers & Polymers Damaged by Long Term (Overnight) Exposure to Monochromatic Aluminium X-rays

1-butene, isotactic (studied for X-ray induced damage) freshly exposed bulk	145
4-methyl-1-pentene (studied for X-ray induced damage) freshly exposed bulk	150
acetal (trioxane) (studied for X-ray induced damage) freshly exposed bulk	
acrylic acid (no flood gun used to avoid flood gun damage)	
acrylic acid (studied for X-ray induced damage) film on aluminium foil	
acrylonitrile (PAN) (studied for X-ray induced damage) as received film	
amide resin (studied for X-ray induced damage) freshly exposed bulk	
anti-static bag (used to store printed circuit boards, SECO Co.)	
anti-static bag (used to store printed circuit boards, SEALPAK Co.)	206
bis-phenol carbonate (PC) (studied for X-ray induced damage) freshly exposed bulk	
cellulose proprionate (freshly exposed bulk)	
dimethylsiloxane (dimethyl silicone oil)	
ethylene (HDPE) (studied for X-ray induced damage) freshly exposed bulk	228
ethylene (LLDPE) as received and after O ₂ plasma treatment	233
ethylene oxide (PEO) (studied for X-ray induced damage) film from CHCl ₃ solution	
ethylene terephthalate (PET, Mylar TM) (studied for X-ray induced damage) freshly exposed bulk	248
ethylene tetra fluoro ethylene (ETFE) as received	257
Glad Wrap TM (as received)	262
Kapton™ (studied for X-ray induced damage) freshly exposed bulk	267
methyl methacrylate (PMMA) (studied for X-ray induced damage) film from CHCl ₃ solution	278
nitrocellulose (filter paper) (studied for X-ray induced damage)	286
Nomex TM (as received)	
Nylon 6 TM (caprolactam) (studied for X-ray induced damage) freshly exposed bulk	304
phenylene sulfide (PPS) (studied for X-ray induced damage) powder on adhesive tape	315
propylene (PP) freshly exposed bulk	
propylene (PP) as received	327
propylene (PP) (studied for X-ray induced damage) freshly exposed bulk	331
Saran Wrap™ (as received)	
spider web (Black Widow, bunched together, but not sticky)	341
styrene (PS) as received film	
styrene (PS) (studied for X-ray induced damage) freshly exposed bulk	351
styrene (PS) Gamma-ray treatment	
styrene (PS) oxidized (O ₂ plasma treatment)	
styrene (PS) oxidized and Gamma-ray treatment	
sulphone resin (studied for X-ray induced damage) freshly exposed bulk	366

Section 2 - Commercial Polymers & Polymers Damaged by Long Term (Overnight) Exposure to Monochromatic Aluminium X-rays

tetra-fluoro ethylene (PTFE) Teflon TM (studied for X-ray induced damage) thin film
VIIVI ACEIAIC IT VAITSIUUICU IOI A-IAV IIIUUCCU UAIIIAECI IICSIIIV CADOSCU DUIK
vinyl chloride (PVC) (studied for X-ray induced damage) beads on adhesive tape
vinyl methyl ketone (PVMK) film on glass40
vinyl pyridine (as received, bead)
vinylidene di-fluoride (studied for X-ray induced damage) powder on adhesive tape41
Ziploc TM (interior surface)