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QUALITY ENGINEERING AND CONTROL SEMIANNUAL PROGRESS REPORT NOVEMBER AND DECEMBER 1977 AND JANUARY THROUGH APRIL 1978

Robert L. Carpenter

ANALYTICAL LABORATORIES GROUP

Rockwell International

Energy Systems Group
Rocky Flats Plant
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Golden, Colorado 80401

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for the
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SUBJECT DESCRIPTORS

Abrasive Blasting
Actinides
Alloys
Building Materials
Chemical Effects
Chemical Reactions
Chromium
Decomposition
Defects
Dielectric Materials
Differential Thermal Analysis
Electrolysis
Electron Microscopy
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Manganese
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X-ray Fluorescence Analysis

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QUALITY ENGINEERING AND CONTROL SEMIANNUAL PROGRESS REPORT NOVEMBER AND DECEMBER 1977 AND JANUARY THROUGH APRIL 1978

Robert L. Carpenter

INFRARED SPECTROSCOPY

DIBUTYL PHOSPHATE IN AQUEOUS COLUMN ELUATES

Patricia A. Hyman and Judy A. Laurent

SUMMARY

Using infrared spectroscopy, it is possible to determine the concentration of dibutyl phosphate (DBP) in both aqueous 5.0 normal (*N*) nitric acid (HNO_3) and 1.0 molar (*M*) sodium carbonate (Na_2CO_3) ion column eluates. Nitric acid solutions are extracted directly with carbon tetrachloride (CCl_4), and the alkyl (C-H) absorption of DBP in the CCl_4 extract is measured by infrared spectroscopy. Sodium carbonate solutions must first be acidified with HNO_3 before DBP can be extracted by CCl_4 for subsequent analysis.

EXPERIMENTAL

Dibutyl phosphate is selectively more soluble in CCl_4 than in neutral or acidic aqueous solutions. DBP forms a water-soluble complex in alkaline solutions but can be freed for extraction with CCl_4 by acidifying the basic solution with HNO_3. Sodium carbonate solutions containing DBP are mixed with 1.5 times their volume of $2N$ HNO_3 before CCl_4 extraction. The addition of HNO_3 to Na_2CO_3 solutions is done carefully in order to avoid excessive effervescence of carbon dioxide gas. The method is valid only on the assumption that DBP is the sole CCl_4-soluble, C-H-containing species in the aqueous solution.

Two sets of standard solutions of DBP in $5N$ HNO_3 and in $1.0M$ Na_2CO_3 were prepared in the concentration ranges of 20 to 3600 parts per million by volume (ppm) and 64 to 3200 ppm, respectively. The C-H infrared absorbance at 3.4 micrometres (μm) for the CCl_4 extract of each standard solution was measured using the baseline method. The CCl_4 extracts of the standards were contained in a 10-centimetre path-length glass cell with sodium chloride windows, and the C-H absorbance was measured differentially against CCl_4 contained in a matched reference cell. Beer-Lambert calibration plots of absorbance at 3.4 μm versus concentration of DBP in the CCl_4 extract were constructed for the concentration ranges of 20 to 3600 ppm for the HNO_3 standards and 64 to 3200 ppm for the Na_2CO_3 standards. Because of the partial solubility of DBP in the aqueous media, the Beer-Lambert plots were not linear over the specified concentration ranges. Typical Beer-Lambert plots are given in Figure 1.* The mean relative standard deviation of absorbance measurements on a series of standards is 9 percent. No recovery factor is necessary for this method since the samples are analyzed in exactly the same manner as the standards.

IRRADIATED COMMERCIAL WATCH OILS

Roger S. Cichorz

SUMMARY

A commerical watch oil, a blend of refined dolphin head oil and paraffinic base mineral oil, which had been irradiated by a cobalt-60 gamma source in air, underwent two observable chemical changes. Infrared spectroscopy indicated that ester cleavage of the fatty triglycerides comprising the dolphin head oil was the major radiolytic reaction and that dehydrogenation of both the mineral and dolphin head oils was a minor radiolytic reaction.

*All figures appear at end of text.

EXPERIMENTAL

The study was undertaken in support of a research and development investigation of radiation-induced changes in process materials that have high exposures to plutonium or other radioactive substances. Nye Superior Watch Oil and Nye Celebrated Watch Oil (products of the William F. Nye Company, New Bedford, Massachusetts) are used at Rocky Flats as precision oils on surfaces which are undergoing dimensional tolerance testing. These products traditionally have been used because of their inherent oxidation stability, viscosity characteristics that allow for precision gauging, and ability to be entirely removed from surfaces during routine cleaning procedures.

Both of these products are blends of paraffinic base mineral oil and refined dolphin head oil in the approximate proportion of 1 part mineral oil and 2 parts dolphin head oil. The dolphin head oils (also called dolphin oil, porpoise oil, or blackfish head oil) are naturally-occurring oils in the cavities of the heads of most of the smaller-tooth whales which include dolphins and porpoises. The refined and purified dolphin head oil that is contained in the Nye products is a mixture of fatty triglyceride oils derived from branched-chain, short chain-length fatty acids (principally isovaleric acid). These fatty triglycerides contain a low incidence of unsaturated species, a property which allegedly results in relative stability against oxidation and gumming.

For this study, the Nye oil samples were housed in a 0.1-millimetre path-length potassium bromide cavity cell to obtain relatively-thick film spectra that would show subtle differences between samples. The infrared spectra of the two nonirradiated reference oils were identical and characteristic of a mixture of paraffinic base mineral oil and saturated fatty triglycerides. Upon irradiation, two changes occurred in these products that can be correlated to observed changes in the infrared spectra.

The major change is ester cleavage at one of the three triglyceride sites of the fatty oil with the concomitant formation of a carboxylic acid and diglyceride ester as indicated in the reaction:

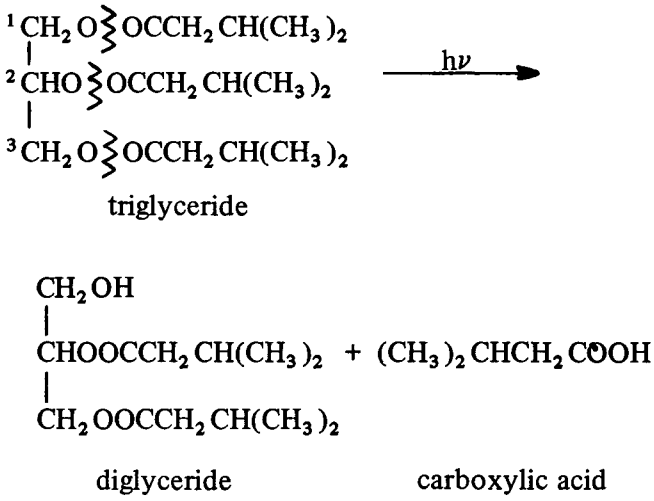


TABLE 1. Additional Absorptions in the Infrared Spectra of Two Irradiated Nye Watch Oils

Absorption (cm^{-1})	Assignment	Attribution	
3600 to 3200[a]	νO-H[b]	diglyceride formation	ester cleavage
3600 to 3050[a]	νO-H[b]	carboxylic acid formation	ester cleavage
3200 to 3000[a]	νC=C[b]	olefinic -C=C formation because of dehydrogenation	
1730 to 1710	νC=O[b]	carboxylic acid formation	
1220	νC-O[b]	diglyceride formation	
970	δC-H[c]	olefinic -C=C- formation	
930	δO-H[c]	carboxylic acid formation	

[a] continuous, broad absorption band.
[b] ν = stretching frequency;
[c] δ = out-of-plane deformation (bending frequency).

The cleavages probably occur randomly at the number 1, 2, and 3 carbon sites of the glycerol; statistically, two of the products shown above should be formed to each diglyceride at the number 2 carbon site. Secondary cleavages to form monoglycerides and eventually glycerol probably also occur, but to a much lesser extent.

A minor change is dehydrogenation. This probably occurs in both the mineral oil and ester oil in a random manner between various alkyl (terminal) and alkylene (chain) carbons. Additionally, it is anticipated that some oxidation of both the ester oil and mineral oil occurs; however, this cannot be observed in the infrared spectra because of the dominant carbonyl contributions of the esters and carboxylic acid products. Significantly, the carbonyl absorption of the ester, upon irradiation, broadened only on the low frequency side, indicating major carboxylic acid formation, the effect of the ester cleavage reaction discussed above. The observed changes in the infrared spectra of the irradiated samples and assignments to structural changes that have occurred because of irradiation are summarized in Table 1. The infrared spectra of samples of the nonradiated commercial watch oil and the irradiated product are shown in Figure 2.

SULFATE IN PLUTONIUM NITRATE FEED SOLUTIONS

Roger S. Cichorz

SUMMARY

The infrared spectroscopy method for determining the sulfate contents of plutonium compounds has been extended to plutonium-nitric acid feed solutions. To assess the accuracy and precision of the method, a series of standard solutions containing sulfate ion (SO_4^{2-}) in the 0.010 to 0.145 molar (*M*) concentration range was prepared and analyzed by this method. Analysis results were all within the range of the calculated (theoretical) SO_4^{2-} concentration, although a high bias appears for low concentrations. The relative standard deviation for replicate analyses of solutions of greater than 0.024*M* SO_4^{2-} was less than 10 percent.

INTRODUCTION

The concentration of SO_4^{2-} in plutonium nitrate feed solution has been determined for several years by a method which involves evaporating a known aliquot of the feed solution at <100 °C and sampling a portion of the residue. Quantitative sulfate determination is accomplished by subjecting the residue to the established alkali halide pellet infrared spectroscopic technique,[1,2] obtaining the SO_4^{2-}concentration from a Beer-Lambert calibration plot, and calculating the resultant sulfate concentration taking into consideration the residue weight found and aliquot volume employed.

This method had never been verified for accuracy, although when originally developed, it appeared to offer reasonably reliable results for the following reasons:

(1) The sulfate in the feed solution should be quantitatively converted to $Pu(SO_4)_2 \cdot 4H_2O$ at the relatively low temperatures employed for evaporating the solutions to dryness. Since $Pu(SO_4)_2 \cdot 4H_2O$ is relatively stable to 167 °C,[3] and $Pu(SO_4)_2$ is relatively stable to above 600 °C,[4] no loss of sulfate should be experienced in the evaporation step.

(2) The infrared spectroscopic alkali halide method for sulfate in plutonium compounds, notably PuO_2 and PuF_4, had been established, and the matrix effect was found to be relatively insignificant.[5] This would enable the existing Beer-Lambert calibration plots to be valid for a plutonium nitrate complex with a minimum of error.

---

[1] A. J. Johnson and E. Vejvoda. "Determination of Sulfate and Nitrate in Plutonium Compounds by Infrared Spectroscopy." *Talanta* 13:81. January 1966.

[2] R. S. Cichorz and E. R. Cocetti. "An Infrared Spectroscopy Method for Sulfate in Plutonium Compounds." *RFP-1541.* May 28, 1970. Rocky Flats Division, The Dow Chemical Company, Golden, Colorado 80401. Pages 6-8. (Classified Report)

[3] K. J. Grossaint. Internal Communication. Rocky Flats Plant. September 1977.

[4] C. E. Pietri. "Plutonium Sulfate Tetrahydrate, A Proposed Primary Analytical Standard for Plutonium." *Analytical Chemistry* 34:1604. November 1962.

[5] R. S. Cichorz and E. R. Cocetti, *op. cit.*

(3) Since the absorption frequencies of either the the nitrate ion or the hexanitratoplutonium(IV) species do not occur immediately at that of the sulfate ion (circa 1075 cm^{-1}), little or no interference by the plutonium nitrate species is anticipated.

EXPERIMENTAL

Seven sulfate standards, consisting of a known concentration of (SO_4^{2-}) in approximately 4*M* nitric acid (HNO_3) containing Pu at a concentration of approximately 100 grams per litre (g/l), were prepared. The SO_4^{2-} concentration ranged from 0.010 to 0.145 *M*. In addition, a single standard of 0.048*M* SO_4^{2-} with a Pu concentration of approximately 50 g/l was prepared in 4*M* HNO_3.

Samples are prepared by transferring 1.00-millilitre aliquots of the standard solutions to tared beakers and allowing the liquid to dry for 24 to 48 hours at 60 °C in a desiccator that has been adapted to allow an air flow to pass over the beakers. After a sample has been air dried to a residue of constant weight, the nonvolatile residue is physically scraped from the beaker and mixed thoroughly to ensure homogeneity. An aliquot of this residue of about 2 milligrams is carefully weighed to the nearest 0.1 milligram (mg) with an analytical balance accurate to ±0.01 mg and transferred to a preweighed amount (300 ±3 mg) of dry *Spectroscopy Grade* potassium bromide (KBr). The sample is then mixed with the KBr for 60 seconds using a mechanical grinder, the mixture quantitatively transferred to a 13-millimetre diameter die, and a transparent pellet pressed at a pressure of 25,000 pounds per square inch. All sample preparation is done inside a glovebox line. The KBr pellet containing the sample is scanned over the 2.5 to 15.0 micrometre (μm) region of the infrared spectrum with a recording infrared spectrophotometer contained inside the glovebox line. The absorbance of the sulfate absorption maximum at about 1075 cm^{-1} is measured by either the empirical ratio (*peak-shoulder*) method or baseline method and related to the SO_4^{2-} concentration plots prepared from $Pu(SO_4)_2$ standards.[6] The concentration of sulfate in the solution can then be calculated by considering the residue aliquot weight, the total weight of nonvolatile residue, and the amount of solution aliquot originally pipetted from the sample.

TABLE 2. Accuracy and Reproducibility of the Sulfate Method

Sample	Calculated Sulfate Concentration (Molarity)	Experimental Sulfate Concentration (Molarity)*
2-0979	0.145	0.135 ±0.016
2-0978	0.097	0.110 ±0.019
2-0977	0.073	0.076 ±0.002
2-0976	0.048	0.052 ±0.005
2-0980	0.048	0.044 ±0.006
2-0975	0.024	0.028 ±0.003
2-0974	0.010	0.015 ±0.011

*Mean results of four replicate analyses given at the 95 percent confidence level (±2 standard deviations).

In this investigation, duplicate analyses of the eight standards were performed. In addition, a sample of plutonium nitrate solution was analyzed to determine if the relatively high nitrate concentration would affect the background absorbance levels of the sulfate. It was found that at high concentrations of sulfate, the nitrate provided no interference and the calculations were routine. At relatively low concentrations of sulfate (<0.05M); the hexanitratoplutonium(IV) absorbance at 1050 cm^{-1} enhanced the absorbance of the sulfate standard, and an absorbance correction factor had to be applied.

The experimental results for a series of four determinations by the baseline technique are summarized in Table 2 and compared to the calculated (theoretical) sulfate concentrations of the standards. Agreement is good throughout the concentration range tested, although the mean results appear high at the lowest concentration levels. This is anticipated because the hexanitratoplutonium(IV) absorption at 1050 cm^{-1} has an enhancing effect on the sulfate absorption at 1075 cm^{-1}, since the latter diminishes in intensity as the concentration is lessened. In the case of the higher concentrations, the intensity of the sulfate absorption is sufficient to overcome nitrate enhancement.

[6] *Ibid.*

The relative standard deviation of the mean results of the lowest concentration standard was 34 percent. However, the relative standard deviation for the mean results of the other six standards ranged from 1.1 to 8.5 percent, a range consistent with the 5 percent relative reproducibility generally attributed to infrared spectroscopy quantitative measurements. Possible methodological errors contributing to decreased reproducibility of results include pipetting errors, errors in three weighing steps (e.g., beaker taring, sample aliquot weighing, and weighing the KBr), and instrument reproducibility. Indeterminate errors could arise from interferring species, operator carelessness, and moisture being introduced into the matrix system. Nonvolatile residue determinations of aliquots of 14 samples (2 each of the 7 standards containing about 100 g/l Pu)--which take into account the pipetting, beaker taring, and final beaker weighing steps in addition to variation between the standard preparation and evaporation steps--had a mean result of 215.1 ±3.72 (at the 2σ 95 percent confidence level) or a relative standard deviation of 1.73 percent. This indicates that methodological errors were relatively minor in this investigation.

MASS SPECTROSCOPY

HIGH-TEMPERATURE OUTGAS ANALYSIS

Tom L. McFeeters

SUMMARY

Dynamic mass spectrometric determination of volatile surface contamination from samples of large geometric surface areas has been accomplished at high temperature (to 650 °C) by use of a special container and a vacuum-brazing furnace.

EXPERIMENTAL

In the past, outgas analyses at temperatures of greater than 300 °C had been restricted to relatively small samples, since the only routine method of providing the vacuum and heating requirements consisted of quartz tubes and a cylindrical external furnace. Samples of greater than 1 to 2 inches in diameter were limited to heating-mantle temperatures because of limitations in the size of bake-out pots and furnaces. Large-volume, high-temperature vacuum furnaces, such as those used in brazing operations, were available, but the large internal surface area and volume contributed excessive background contamination and greatly increased minimum detectable limits.

A special bake-out pot capable of handling samples of up to 10 inches in diameter has been fabricated from stainless steel. A metal gasket flange maintains high vacuum capability through temperature cycles from 25 to 700 °C. Thermocouples may be attached externally, and a thermocouple inlet allows measurement of pot and sample temperature. This apparatus, depicted in Figure 3, successfully allows the outgassing of large samples at 650 °C.

The vacuum furnace has been further modified by replacing the thermocouple inlet with a vacuum line of ¼-inch stainless-steel tubing connected externally to the mass spectrometer ion source. The sample is placed in the prebaked pot, the lid sealed, and the pot then situated in the vacuum furnace chamber. Thermocouples are then placed on the pot and through the pot lid in order to contact the sample. The vacuum line is connected to the pot, and the pot evacuated by the mass spectrometer. The chamber atmosphere around the pot is then evacuated and the sample heating cycle started. After these operations, analysis can be performed by routine dynamic analysis techniques. This apparatus has been successfully used to monitor surface contamination of beryllium parts too large to analyze by routine methods.

MASS SPECTROMETER DEVELOPMENT PROJECT

Jerry R. Turbett

The Analytical Laboratories are participating in a *first* in sharing costs for a major analytical Department of Energy (DOE) Mass Spectrometer Development Program with other DOE weapons contractors.

V-G Micromass of England, one of the world's foremost manufacturers of mass spectrometers, has been awarded the contract to develop a new gas analysis instrument for nuclear applications. The contract outlines increased performances in the areas of sensitivity, resolution, and quantitative accuracy over present instrumentation.

The contract has been divided into three phases: basic design definition, experimental-design demonstration, and the construction, testing, and delivery of a working instrument. The first phase has been satisfactorily completed, and the experimental-design demonstration phase is in progress with expected completion about October 1978. If the results of the second phase are satisfactory, the third phase is expected to begin in November 1978.

POLYCHLORINATED BIPHENYLS AS IMPURITIES IN DIMETHYL SILICONE TRANSFORMER FLUIDS

Keith J. Grossaint

SUMMARY

A probe-outgas technique was developed to determine the amount of residual polychlorinated biphenyl (PCB) compounds remaining in the dimethyl silicone fluid used as a replacement dielectric medium in electrical transformers at Rocky Flats. While other instrumental and classical chemical techniques were ineffectual for this environmental monitoring consideration, the mass spectroscopy method provided rapid, specific, and quantitative results for Aroclor® 1260 in Dow Corning® 561 Fluid.

EXPERIMENTAL

Environmental restrictions on the production, use, and disposal of PCB compounds have resulted in their discontinuance at Rocky Flats. For example, the PCB dielectric fluid in electrical transformers was recently replaced with a dimethyl silicone fluid. To determine the effects of flushing the PCB's with the replacement fluid and to assess any potential environmental effect of future disposal of this dielectric medium, it became necessary to develop a method for the determination of PCB's in silicone fluid. The mass spectral probe technique was selected because it provided rapid, specific, and quantitative results while efforts to use solvent extraction, infrared spectroscopic, chromatographic, and PARR-bomb pyrolysis techniques were unsuccessful.

Selective removal of PCB from the silicone by solvent extraction was unsuccessful because of mutual solubility. Indirect analysis by determining total chloride after PARR bomb pryolysis was not possible, because of the difficulty in obtaining complete ignition of the silicone oil and because of interference by trichlorobenzene. The concentration levels of PCB were too low for detection by differential infrared spectroscopy. The PCB failed to elute from a gas chromatography column when the silicone fluid was injected directly onto the column, indicating severe interference from the presence of the silicone.

Although the mass spectral probe-outgas technique does not usually lend itself to quantitative analysis, it proved adequate for this PCB determination. The technique required accurately weighing a small amount (0.5 to 2.0 mg) of the silicone fluid into the probe capillary, outgassing the PCB completely at 180 °C, and computerized control of both data acquisition and mass scanning. Under these conditions, reproducible ratios of integrated ion intensity to amount of PCB introduced were obtained for standard solutions of PCB in silicone fluid. The fragment ions of silicone fluid did not interfere with the PCB spectral-ion peaks (i.e., appear at same nominal masses). An additional advantage of the technique was that the chlorine distribution (PCB type) and other impurity components could be determined simultaneously. Some disadvantages observed were errors introduced from inaccurate weighings or transfer of the very small sample sizes, relatively large standard deviations resulting from *peak clipping,* uneven heating rates, and other characteristics inherent to the mass spectral probe technique.

TABLE 3. Response Factors for Polychlorinated Biphenyls (PCB's) in Silicone Fluid by Mass Spectral Probe Outgas at 180 °C

Sample	Percent PCB's	Weight (grams X 10^{-5})	Integrated Ion Intensity (X 10^{5})	Response* (X 10^{8})
HI	3.2	1.344	7.681	0.57
LOW	0.37	0.7326	2.28	0.31
LOW 1	0.37	0.3404	20.98	6.2
HI 2	3.2	2.304	165.89	7.2
LOW 2	0.37	0.7141	28.31	4.0
LOW 3	0.37	0.1480	8.980	6.1
HI 3	3.2	2.784	206.98	7.4

*Response is given in terms of integrated ion intensity per milligram of PCB.

The mass spectral probe-outgas technique indicated the PCB content of the replacement silicone oil was at 0.31 percent on the day of exchange and increased to 3.3 percent one week later. This increase in PCB concentration, consistent with predictions by the supplier of the silicone oil, was attributed to extraction of PCB fluid from elastomer gaskets and insulation material in the transformer as its temperature was raised during operation. Mass spectral analysis indicated the PCB impurity contained from 5 to 9 chlorines on the biphenyl group, which agrees with the chlorine distribution of the type of PCB (Aroclor® 1260) used in the previous dielectric fluid. Mass spectral analysis also indicated the presence of quantities of trichlorobenzene, with its concentration approximately doubling in the week following exchange. The previous commercial dielectric fluid was Innertween,® which consists of 60 percent Aroclor 1260 and 40 percent trichlorobenzene.

For this analysis, standard solutions of PCB (Aroclor 1254) in Dow Corning® 561 Silicone Fluid were prepared containing PCB's in approximately the concentrations expected in the transformer fluid. Several determinations of integrated ion intensity per milligram PCB introduced were obtained and are summarized in Table 3. The prominent ions of all molecular species were used for the ion-intensity summation (i.e., all isotopic contributions of molecules containing 4, 5, 6, and 7 chlorines). The same ion-formation efficiencies were assumed for the Aroclor 1260 in the unknown, and the appropriate adjustment for weight of PCB was made.

The data indicated the technique has acceptable reproducibility (6.2 ±2.0 X 10^{8} ions per milligram of PCB) for sets of determinations taken eight days apart. A third set of data indicated an order of magnitude lower response, but a known condition accounted for this (i.e., the ion source was changed and instrument retuned). Subtle changes in instrument response could be ignored if standards are run in conjunction with unknowns.

MICROSCOPY

DEFECT ANALYSIS OF GLOVEBOX GLOVES

John K. Fraser

SUMMARY

Defects and cuts in glovebox gloves can be examined by microscopy and photography in an effort to determine origins. Details of glove damage generally are determined with a Spencer stereo microscope and defects recorded with a Polaroid® MP-4 Land Camera.

EXPERIMENTAL

Entire glovebox gloves with cuts or defects, submitted to the Chemical Operations Support Laboratory, are initially examined for the location of damage and the size and general appearance of the defect. Both sides of the defect are analyzed in an effort to determine from which side the damage occurred.

If information on the entire glove is necessary to the investigation, photographs are taken of the glove placed inside a hood. Polaroid 55 P/N film is used in conjunction with a Crown Graflex Camera equipped with a 135-millimetre lens.

Following this initial examination, the defective area is sectioned from the glove and placed in a *hot cell* for examination at higher magnification. The hot cell is a 2-part cylindrical container about 15 centimetres both in diameter and height, fabricated from polymethyl methacrylate resin (Lucite®). The sample rests on a platform in the hot cell. The top section of the hot cell is fabricated from optical glass and slip fits over the bottom section to contain radioactive contamination. The sectioned sample is then examined with a stereo microscope for abrasions, inclusions of foreign matter in or around the defect, cut surface appearance, or any other evidence needed to determine the origin or cause of the defect.

The stereo microscope, with a 10 to 40 magnification factor, provides an enlarged 3-dimensional view of the defect, which then can be photographed with a Polaroid MP-4 camera. Magnifications of from less then one to approximately 15 times can be made with a series of 17- to 135-millimetre lenses. Enlargements of up to 25 times, required for some investigations, can be made from the negatives. Photomicrographs are made of the glove defect with the glove in flattened-out and folded positions. Horseshoe magnets are used to hold folded glove sections in place while they are undergoing microscopic examination.

Figures 4 through 7 are photomicrographs (7.3 enlargement factor) of gloves supplied by the Charleston Manufacturing Company that were damaged during their use at Rocky Flats. These photomicrographs are examples of a few of the wide variety of defected gloves submitted for examination. Figure 4 depicts the folded section of an 8YLY-S6 glove that was cut from the observed side by a sharp knife blade. Figure 5 shows the folded section of an 8YLY-S6 glove that was punctured from the observed side by a 3-millimetre blade of a screwdriver. In Figure 6, the folded section of an 8YLY-S2 glove was partially cut from the contaminated side of the glove, permitting acid degradation of the lead oxide layer and subsequent tearing of the thin, noncontaminated outer layer of chlorosulfonated-polyethylene resin (Hypalon®). Figure 6 shows this glove from its noncontaminated outer layer. Figure 7 shows the folded section of a glove defect probably resulting from pinching. The view is from its noncontaminated, outer layer side; the cut does not go completely through this glove.

SCANNING ELECTRON MICROSCOPY INVESTIGATIONS OF GRIT-BLAST SURFACES

Davis F. Carpenter

SUMMARY

Application of the replication and image-reversal technique as a quality-control step to the grit-blast operation has effectively brought an out-of-control process under control. Image analysis of grit-blast surfaces has enabled Rocky Flats personnel to study the effects of excursion of various control parameters upon these surfaces.

EXPERIMENTAL

Until recently the effectiveness of the grit-blast operation was assumed to be dependent upon only two parameters: (1) the consistency of weight removed from the waist to pole of a part and (2) the total amount of weight removed. During the course of other studies of a similar nature, the investigator developed a nondestructive technique

for the examination of surface roughness on large parts. This procedure is called a replication and image-reversal technique (refer to earlier Progress Report, RFP-2644, July 14, 1977, pages 16-17).

Application of the replication and image-reversal technique has enabled a more complete characterization of grit-blast surfaces. As a result of these microscopy investigations, four additional surface parameters were found to be significant in determining the overall quality of the product. These parameters, which can be effectively examined and monitored, are the type of roughness, the degree of roughness, the homogeneity of roughness, and the depth factor which correlates to the amount of relief in the surface. Successful analysis of an image requires the *practiced eye* of an experienced microscopist. The following series of micrographs constitutes a pictorial analysis of the surface roughness as it relates to the grit-blast operation.

Micrographs shown as Figures 8a, 8b, and 8c are of the waist, mid, and pole areas of a part which had undergone grit blasting with consideration to only the first two control parameters. There are large differences in surface texture which were typical of parts produced at the beginning of the program. Micrographs shown as Figures 8d, 8e, and 8f are representative of the best texture characteristics that could be achieved with the grit blaster operating in its standard configuration. Micrographs shown as Figures 8g, 8h, and 8i represent the first attempt at alteration of the standard grit-blaster configuration in order to achieve the goal of uniform roughness. In this case, the grit blaster was hand held. A vast improvement in texture was achieved over the surfaces pictured in the preceding set of micrographs.

Micrographs shown as Figures 9a, 9b, and 9c depict an attempt to reproduce the texture found in Figures 8g, 8h, and 8i. The same grit-blasting conditions were used here as on the previous part, and there is a high degree of reproduction of textures. Micrographs shown as Figures 9d, 9e, and 9f are the surface of one half of another part that was also hand grit blasted. Micrographs shown as Figures 9g, 9h, and 9i are representative of the surface of the other half of this part. The uniformity and reproducibility of texture over the entire area of the part is notable.

Micrographs shown as Figures 10a, 10b, and 10c are of surfaces of another part that had undergone a first try at hand grit blasting and are illustrative of macro roughness. This feature had not been seen previously on any of the hand grit blast series. Micrographs shown as Figures 10d, 10e, and 10f depict the results of a second grit blast effort on the same part. The texture shown in this series of micrographs is intermediate between that of the first try and that of an ideally-textured surface. The final two micrographs, shown as Figures 10g and 10h, are of still another part and represent an ideally-textured surface because they demonstrate that the surface meets the criteria of all six control parameters.

RADIOMETRIC ANALYSIS

IMPROVED SEPARATION OF ACTINIDES IN SOIL SAMPLES

David R. Guhlow, John R. Stevens, Lewis B. Yoder, and Terry F. Rees

SUMMARY

A sequential method involving only four aliquots per sample has been developed for determining thorium (Th), uranium (U), neptunium (Np), plutonium (Pu), americium (Am), and curium (Cm) in soils. This sequential procedure has the same accuracy and precision as the previous method of Guhlow et al. (refer to previous Progress Report RFP-2699, March 27, 1978, page 12) and the advantages of reduction in time for sample preparation and of ease of chemical separations.

EXPERIMENTAL

In this procedure, a weighed sample of soil is fused with potassium pyrosulfate. The fusion product is dissolved in aqueous 0.5 normal (*N*) nitric acid (HNO_3) and then eluted through a column containing cation-exchange resin, which results in the retention of Am and Cm on the resin. The column effluent, containing Th, Pu, Np, and U, is made 8*N* in HNO_3 and then eluted through a column containing anion-exchange resin. The Th and Pu is retained on this resin, and the effluent is taken to dryness. The resultant residue is dissolved in 12*N*

hydrochloric acid and passed through a column containing anion-exchange resin in order to render Np and U into determinable form.

The sequential method offers several advantages over the previous procedure. Sample-preparation time has been substantially reduced, since all separations come from only four weighed aliquots instead of twelve and only four separation schemes are utilized instead of ten. Additionally, for the determination of U, Am, and Cm, this procedure has resulted in better precision and accuracy. The lower detection limits for each element in disintegrations per minute per gram remain as before: Th = 0.2, U = 0.2, Np = 0.2, Pu = 0.2, Am = 0.6, and Cm = 0.6.

THERMAL ANALYSIS

COMPATIBILITIES OF COMMERCIAL PACKING MATERIALS WITH NITRIC ACID

Roger S. Cichorz

SUMMARY

Several packing materials composed of polytetrafluoroethylene resin or graphite were evaluated for their feasibility for use in a system in which contact with seven molar (*M*) nitric acid (HNO_3) was inevitable. Testing was accomplished by sealed-capillary differential thermal analysis (DTA), and the onset temperatures of exothermic reactions were used as indicators of the ability of the packing materials to react in the system.

In general, polytetrafluoroethylene resin or graphite was inert to reaction with 7*M* HNO_3; however, plasticizers, extenders, and other organic additives in the formulations did react exothermically in DTA experiments. Packings containing these additives could be used provided that the bulk of the additives were removed by a solvent-extraction or an HNO_3-preconditioning step. From an economics standpoint, the commercial packings in the untreated state passing the DTA evaluation were more highly recommended. The data obtained in this investigation were useful in predicting the behavior of compositionally-similar packings.

EXPERIMENTAL

The employment of sealed-capillary DTA testing as a technique for examining thermal reactivities of mixtures of organic resins and HNO_3 has been discussed in an earlier Progress Report (refer to RFP-2644, July 14, 1977, pages 23-25). In this investigation, the DTA method was applied to several commerical packing materials to assess their compatibilities with 7*M* HNO_3. The study was undertaken to evaluate the performance of these packings in a system designed to contain and transfer 7*M* HNO_3 from a central storage tank to several outlets scattered throughout a production facility. Since interaction of the packing material and HNO_3 is considered inevitable at some time during the lifetime of the system, the engineering and maintenance design personnel involved in the project were interested in obtaining packing that would remain chemically unreactive to HNO_3 during prolonged periods of contact.

Commercial packings are composed of organic resins, generally bulk elastomeric resins or nonelastic resins that have been rendered flexible by design or by the addition of plasticizers or both. Designed flexibility is accomplished by fabricating a non-elastic resin in the form of filaments, strands, fibers, or braids. Frequently incorporated into these packing materials are *inert* reinforcing agents, pigments, or fillers, such as fiberglass, carbon black, graphite, silica, or titanium dioxide, designed to impart additional properties or appearance to the product.

For the particular HNO_3 system considered for Rocky Flats, it is necessary to avoid use of the relatively-reactive, conventional elastomeric packings composed of neoprene, polyisoprene, nitrile rubber, plasticized vinyl resins, polyesters, or styrenated resins, because HNO_3 typically reacts exothermically with these resins at slightly elevated temperatures. Additionally, HNO_3 typically reacts exothermically at slightly elevated temperatures with some common additives, such as ester plasticizers or mineral oil extenders, that are incorporated into commercial packing materials. Also, these additives can be leached out by HNO_3 at room temperature, rendering the packing brittle and ineffective after short periods of use. Consequently, only packings fabricated from materials known to be relatively inert to HNO_3, such as

graphite and polytetrafluoroethylene resins of designed flexibility, have been considered for use in the HNO_3 system and subjected to the DTA evaluation studies.

Seven commercial packings composed of polytetrafluoroethylene resin, two composed of graphite, and one composed of polyisobutylene were submitted for compatability evaluation. Analyses and tests prior to the DTA tests included complete compositional analysis of resins, additives, and fillers by infrared spectroscopic and other methods, and extractions of organic additives with carbon tetrachloride, ethyl ether, acetone, and methanol. The packings were submerged for 24 hours in 7*M* HNO_3 at 60 °C for observation of resin discoloration or other signs of decompositon or reaction. The HNO_3 extraction media were also examined to determine if any additives were removed by the acid (i.e., if any residues remained after subsequent evaporation of HNO_3). The DTA testing was performed both on the packing after 24-hour submersion in HNO_3 and on equal volume mixtures of as-received packing and 7*M* HNO_3.

In general, the packing composed of polytetrafluoroethylene resin or graphite resisted reaction with HNO_3. However, many of the packings contained plasticizers, oil extenders, and other organic processing materials which reacted exothermically with HNO_3 above 100 °C. In several cases, enough organic additives were present in the resin to cause a reaction between the HNO_3-treated packing material after it was air dried. Apparently, enough HNO_3 remained physically sorbed in the resin system to react with organic matter that had not been leached during the contact-immersion period.

Of the ten packings tested, only the polyisobutylene-compounded resin reacted at room temperature when contacted with 7*M* HNO_3, and this reaction was attributed to the alkaline nature of its inorganic mineral silicate filler. After the initial reaction had subsided, the packing was tested by DTA and an exothermic reaction occurred starting at 271 °C. This packing was not deemed acceptable for use because of the vigorous reaction that occurred on initial contact with HNO_3.

Of the nine other packings tested, only one appeared unsatisfactory because of its relative reactivity with HNO_3. This was attributed to its plasticizer/dispersant system, a mixture of polymeric alcohol and polyamide, rather than the polytetrafluoroethylene resin, based on the rather high potential for reactivity between HNO_3 and alcohols. The other eight packings have compositional characteristics that place them into one of two general reactivity groupings. The first group is completely inert to HNO_3 and is comprised of the packings that do not contain any organic matter other than a polytetrafluoroethylene resin or graphite. The second group is relatively inert to HNO_3 and is comprised of packings that contain both polytetrafluoroethylene resin or graphite and other organic additives that are subject to extraction by HNO_3 and react with HNO_3 at elevated temperatures. The packings in the group can become acceptable for use if they are treated with appropriate organic solvents or HNO_3 prior to their application. This step is costly and tedious, however, and the packings in the first grouping are generally preferred.

One packing investigated contained some organic additives but did not undergo an exothermic reaction during DTA testing; apparently, the concentration of the additives was not enough for sufficient chemical interaction with HNO_3. The results of the DTA testing and compositional analyses of the ten packings submitted are summarized in Table 4.

Sealed-capillary DTA testing has been adequately demonstrated as a means of examining the behavior of chemicals and commercial formulations in given chemical environments. For example, the similarities among formulations of commercial packings permit DTA testing to be a tenable means of evaluating these products in an acid environment. With the data base established in an abbreviated investigation as the one reported in this work, the behavior of other packing materials as they are submitted for evaluation can be predicted with a knowledge only of their chemical compositon. Several additional polytetrafluoroethylene resin-based packing materials have been analyzed for additives, and their reactivities in an HNO_3 system were predicted from the DTA results of compositionally-similar packings that had been previously tested. Agreement on a qualitative basis was reasonably good; i.e., the predicted exotherm onset temperature was within 10 °C of that obtained by DTA testing.

TABLE 4. Summary of Compositional Analyses of Commercial Packing Materials and Differential Thermal Analysis (DTA) Results of 7 Molar Nitric Acid Compatibility Testing

Packing Material	Compositional Analysis	DTA Exothermic Reactivity 1	DTA Exothermic Reactivity 2	Comments for Use in Systems Containing Nitric Acid (HNO_3)
Sacomo Style 705 (Sacomo-Sierra, Inc.)	graphite-filled polytetrafluoroethylene yarn	none	none	Recommended; no reactive organic additives present.
Unichem 6313 (Martin Merkel KG)	polytetrafluoroethylene yarn, silica filler; fatty amide, alkyl phenol-polyoxyethylene condensate, dimethyl silicone oil additives	none	none	Pretreatment recommended to remove extractable organic components.
Chesterton Style One (Chesterton Company)	powdered graphite-filled graphite fibers, trace resin additive	230 °C	none	Recommended; no reactive organic additives present; graphite powder removable on flexing.
Crane (braided)	graphite-impregnated polytetrafluoroethylene yarn, fiberglass laminate reinforcement; alkyl phenol-polyoxyethylene condensate, polyamide additives	236 °C	none	Pretreatment recommended to remove extractable organic and inorganic components.
Crane (flat sheet)	graphite sheet, inorganic oxide filler, mineral oil additive	290 °C	none	
Crane (ribbed) (John Crane Packing Co.)	graphite sheet, inorganic oxide filler, dioctyl phthalate additive	none	none	
Chesterton No. 375	graphite fibers; alkyl phenol-polyoxyethylene condensate, polyamide additives	250 °C	none	Pretreatment recommended to remove extractable organic components.
UNIVERDIT 7005 (Martin Merkel KG)	polytetrafluoroethylene resin; alkyl phenol-polyoxyethylene condensate, aromatic and naphthenic hydrocarbon resin additives	250 °C	none	Pretreatment recommended to remove extractable organic components.
Lanacid 5986 (Martin Merkel KG)	graphite-coated polytetrafluoroethylene yarn; fatty amide, alkyl phenol-polytetrafluoroethylene condensate, dimethyl silicone, paraffin wax additives	142 °C	none	Pretreatment recommended to remove extractable organic components.
UNIVERDIT 7000	polytetrafluoroethylene resin; polyoxyethylene ether, naphthenic and aromatic hydrocarbon resin additives	140 °C	--	Pretreatment recommended to remove extractable organic components.
Alchem II (Martin Merkel KG)	polytetrafluoroethylene resin-impregnated polytetrafluoroethylene fibers, silica filler; alkyl phenol-polyoxyethylene condensate and polyamide additives	115 °C	115 °C	Not compatible with regard to reactivity considerations.
ItS Din 3754 (Frenzelli Packings)	mineral silicate-filled polyisobutylene resin	<25 °C	271 °C	Not compatible with regard to reactivity considerations.

DTA exothermic reactivity: Column 1 results represent the exothermic behavior of an equal volume mixture of packing and 7 molar HNO_3 during sealed capillary trials. (The exotherm onset temperature is listed in °C when it occurred; *none* means no exotherms occurred to 450 °C) Column 2 results represent the sealed-capillary exothermic behavior of packing after it had been immersed in 7 molar HNO_3 for 24 hours, removed, rinsed with distilled water, and dried. (If an exothermic reaction occurred, the onset temperature is listed; *none* means no exotherms occurred to 450 °C.)

NOTE: The author is making no recommendations, expressed or implied, and assumes no legal responsiblity for the use of any of the commercially-available packings discussed in this report. The study has been presented as a survey of analysis results, obtainable by a particular thermal analysis technique, that are useful for the evaluation of potential long-term performance of products being considered for a specific system. Reference to a company or product name does not imply approval or recommendation of the product by Rockwell International or the U.S. Department of Energy to the exclusion of others that may be suitable.

X-RAY FLUORESCENCE SPECTROSCOPY

STATISTICAL EVALUATION OF THE XRD-6 X-RAY UNIT

Jack L. Long and James A. Montano

SUMMARY

The General Electric XRD-6 crystal dispersive X-ray unit was evaluated using liquid uranium (U)-niobium (Nb) standards and solid stainless steel standards. The correlation coefficients for Nb in U-Nb alloys and for chromium (Cr) and nickel (Ni) in stainless steels were 0.995 or better for each element.

EXPERIMENTAL

The Nb in U ranges from 5.5 to 6.5 weight percent. The relative standard deviations based upon 45 measurements of Nb and U were 0.44 and 0.40 percent, respectively. The evaluation showed that the Nb could be determined to within ±0.1 percent at concentration levels of from 5.50 to 6.51 weight percent. The mean of five 20-second counts was used for both Nb and U, and the Nb-U count ratio was correlated to the concentration of Nb. The correlation coefficient for Nb was 0.9953 as determined from 45 measurements each of U and Nb intensities.

The ranges of Cr and Ni in stainless steel standards used were 9.3 and 12.5 weight percent and 12.8 to 18.8 weight percent, respectively. The relative standard deviations for Cr and Ni were 0.23 percent and 0.15 percent, respectively, for four standards based on 80 measurements of each element. The mean count based on a counting time of 20 seconds for each element was correlated to the concentration of the element present. The correlation coefficients were 0.9994 for Cr and 0.9976 for Ni, based on the 80 measurements on each element.

STATISTICAL EVALUATION OF THE QANTA METRIX X-RAY UNIT

Jack L. Long

SUMMARY

The Qanta Metrix energy dispersive X-ray unit was evaluated using six stainless steel standards. The elements determined were chromium (Cr), manganese (Mn), and nickel (Ni). For standards ranging in concentrations of from 12.8 to 20.5 weight percent Cr, 0.5 to 8.8 weight percent Mn, and 6.8 to 12.5 weight percent Ni, the corresponding relative standard deviations were 0.88, 9.5, and 1.18 percent, respectively. Correlation coefficients of 0.994 or better for each of these three elements were obtained by total area measurement.

EXPERIMENTAL

The stability of the unit was determined by an overnight single sample run. Only a single position on the sample turntable was used, and 264 measurements were obtained for the stability testing. Since the run was unattended, the standardization function was not in operation. This function corrects for spectral drift by comparing the area under the curve produced by a given element with an area assigned when the computer was initially programmed. However, there appeared to be insignificant drift, so the results probably would not have been improved by operation of the standardization function. The relative standard deviations obtained were 0.46, 1.17, and 1.18 percent for Cr, Mn, and Ni, respectively.

The relative standard deviations for these three elements were determined on six standards, utilizing six positions on the turntable and the multiple regression software program. The standardization function was operated after each cycle of the six standards. The relative standard deviation obtained from 15 cycles, consisting of 90 measurements on each element, were 0.88 percent for Cr, 9.5 percent for Mn, and 1.18 percent for Ni. A large relative standard deviation for manganese was obtained because only a small amount of the element is present in most of the standards (i.e., three of the standards contained less than 1.0 weight percent Mn and another contained just over 1.0 weight percent Mn). For example, for a standard containing 0.53 weight percent Mn, eight measurements yielded a mean analysis of 0.45 weight percent with a relative standard deviation of 22.3 percent. The correlation coefficients were determined by total area measurement. A total of 264 measurements were made on each of the three elements, and the resultant correlation coefficients for Cr, Mn, and Ni were 0.9993, 0.9942, and 0.9942, respectively.

ANALYSIS OF STAINLESS STEELS FOR CHROMIUM AND NICKEL

Harvey L. Bramlet

SUMMARY

Chromium (Cr) and nickel (Ni) concentrations in stainless-steel samples can be determined by energy-dispersive X-ray fluorescence spectrometry regardless of plutonium contamination. Samples are prepared by making a flat surface with similiar roughness to two or more standards which bracket the composition of the sample. Energy-dispersive X-ray spectra, taken over 100-second count intervals, are processed directly by computer, and the analysis results in weight percent are printed out on a teletype console.

EXPERIMENTAL

Equipment corrosion problems are encountered frequently during plutonium-processing operations. In order to solve these problems, it is often necessary to determine the alloy employed in construction of the equipment. Presently in use is an energy-dispersive X-ray fluorescence system that provides the capability of analyzing plutonium-contaminated equipment which is larger in size than the conventional 1¼-inch diameter coupon samples. With this system, samples of up to 8 inches in diameter and 5 pounds in weight can be conveniently handled.

The system uses a Philips XRG-5000 X-ray Generator with its top enclosed in a large interlocked radiation shield. The enclosed area contains a vertical goniometer fitted with a lightweight remotely-fed lithium-drifted silicon [Si(Li) cryogenic detector. X-ray counting data are taken with a Nuclear Data 4420 computer programmed for multichannel pulse-height analysis. The computer utilizes 24 K of core and has integrated-system dead-time correction. Programming for complete data handling is done in extended BASIC language. The current software provides the capability for both energy-dispersive X-ray diffraction or quantitative energy-dispersive X-ray fluorescence analysis.

The sample holder is an old-style AMR metallurgical sample platform, about 4 inches square, fitted with a combined leveler and vertical position gauge. The omega- or theta-to-two-theta angle is variable, and the detector-to-sample distance is adjustable with two thumb screws.

For distinguishing Inconel,® Monel,® and stainless steel from one another without differentiation as to the specific type, a flat surface is not necessary. In these cases, the cleaned metal surface need only be placed approximately in the X-ray beam and a spectrum taken for about one minute. For determining the specific type of stainless steel, the analysis is done by preparing a flat surface approximately 0.5 inch in diameter and then placing the sample in a plastic bag onto which a Mylar® window has been placed. Samples are counted 100 seconds and the total Ni Kα and Cr Kα counts extracted by the computer. Results are computer calibrated by counting two or more standards that bracket the Ni and Cr concentrations in the unknown. The calibration plot is a straight line if the two standards are just several percent different in these two elements. The slope and intercept are determined by a standard linear regression calculation and are retained in the computer for future analysis of unknown samples.

Typical Cr calibration data based on two Brammer Standards[7] are: 51,202 counts for 17.24 weight percent (%) Cr and 64,967 counts for 22.81% Cr; slope = 4.064×10^{-4} and intercept = −3.479. Typical Ni calibration data based on these two same standards are: 15,892 counts for 8.16% Ni and 27,423 counts for 13.16% Ni; slope = 4.336×10^{-4} and intercept = 1.269. The mean results and 95 percent confidence intervals of ten replicate analyses of a third Brammer Standard,[8] the Cr and Ni contents of which were within the range of the two calibration standards, are: Cr = 18.48 ±0.35% and Ni = 8.14 ±0.32%.

[7] Brammer Standard 80C, stainless steel type 303, Cr = 17.24% Ni = 8.16% and Brammer Standard 82A, stainless steel type 309, Cr = 22.81%, Ni = 13.16%, British Chemical Standards issued by the Bureau of Analysed Samples, Ltd., Brammer Standards Company, Newham Hall, Middlesbrough, England.

[8] Brammer Standard 81D, stainless steel type 304, Cr = 18.33%, Ni = 8.46%

ILLUSTRATIONS
(Figures 1 through 10)

FIGURE 1. Beer-Lambert Calibration Plots for Dibutyl Phosphate (DBP) in Aqueous Nitric Acid and Sodium Carbonate Solutions

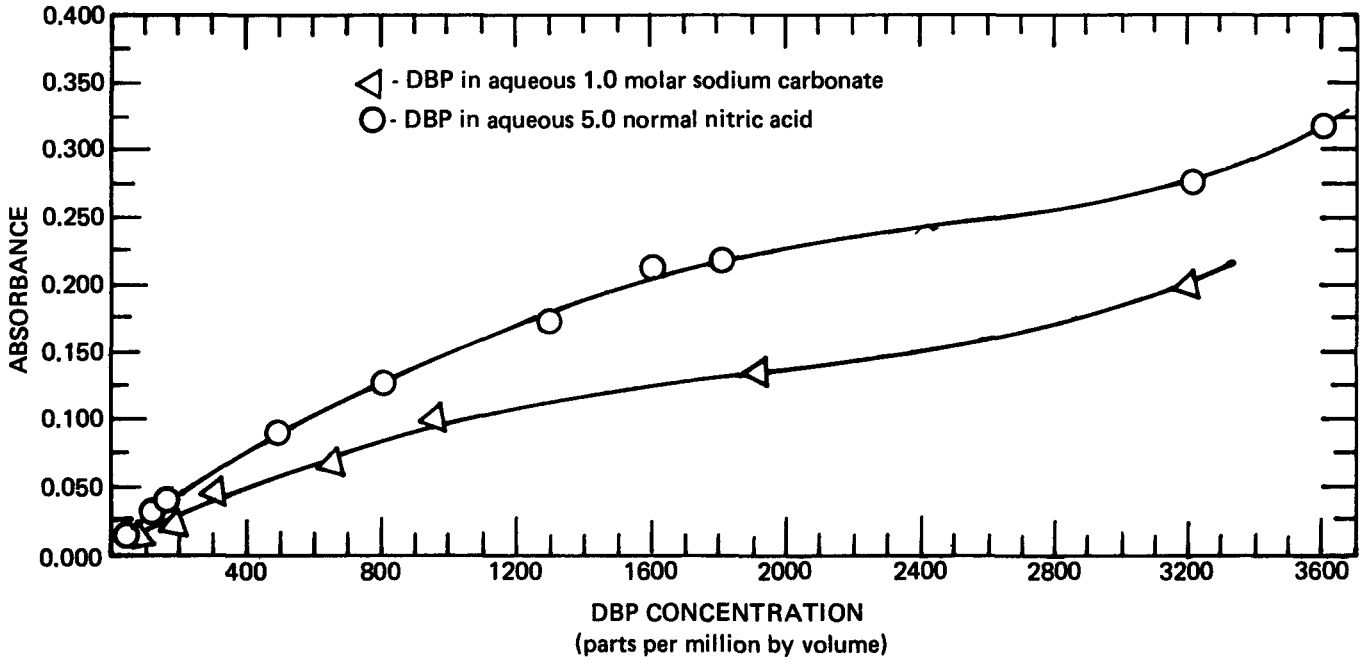


FIGURE 2. Infrared Spectra of Samples of Irradiated and Nonradiated Commercial Watch Oils

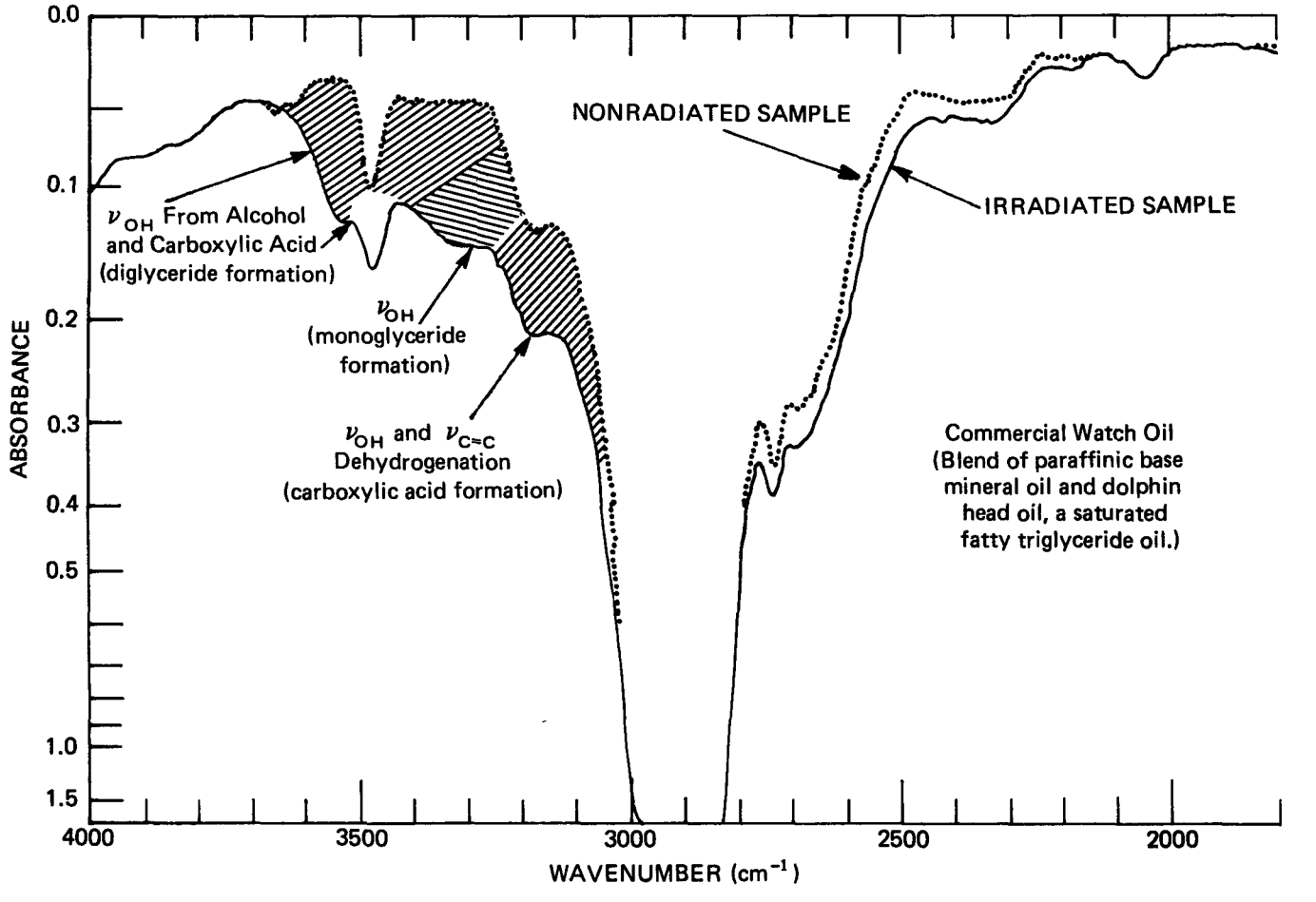
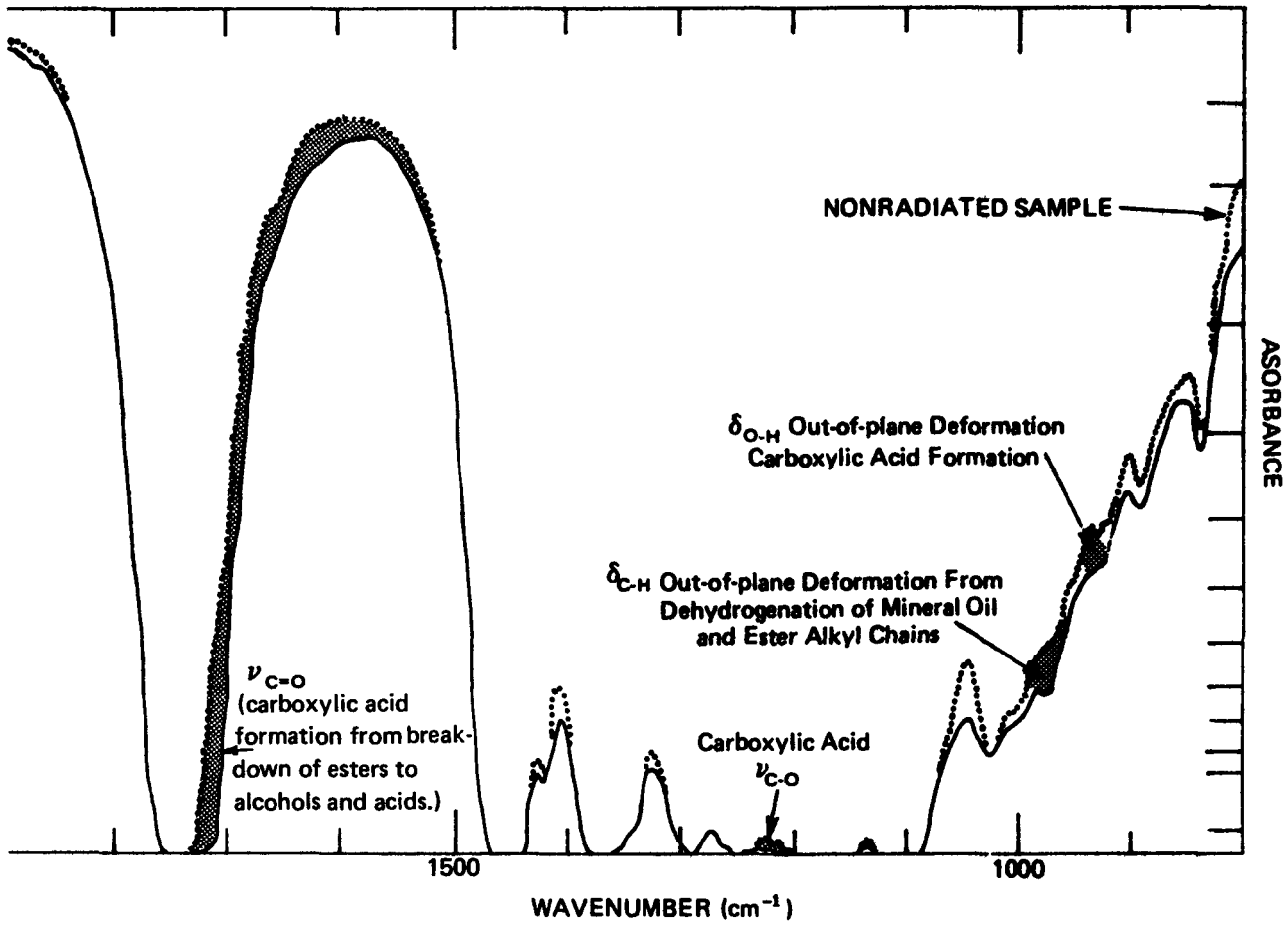


FIGURE 2. (Continued)



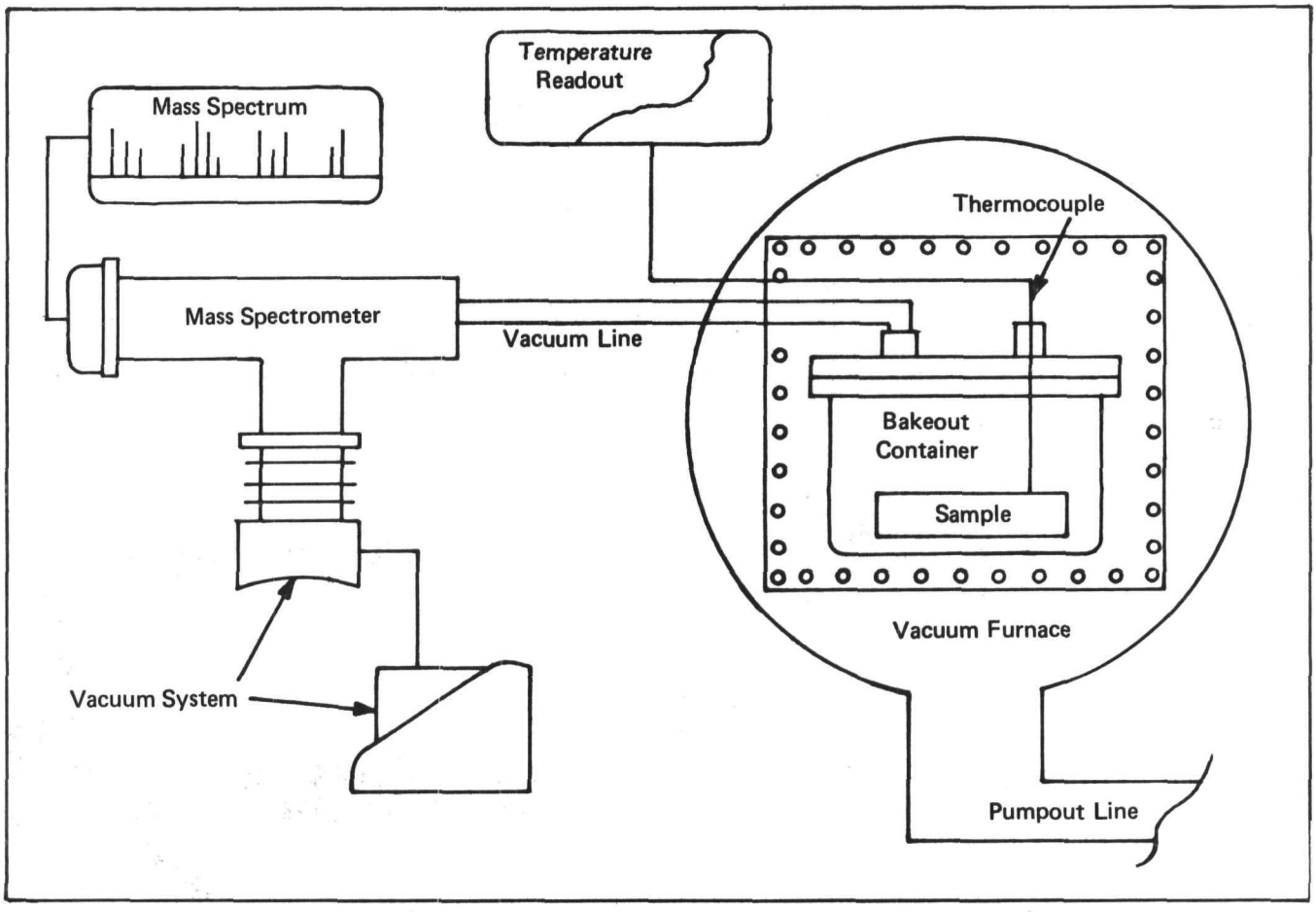


FIGURE 3. High-Temperature Dynamic Outgas Apparatus

FIGURE 4. Photomicrograph of a Knife-Blade Cut of an 8YLY-S6 Glovebox Glove

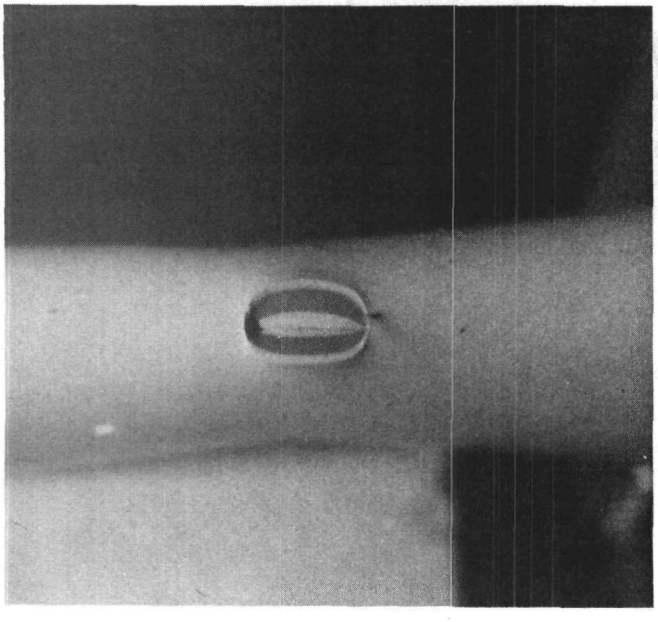
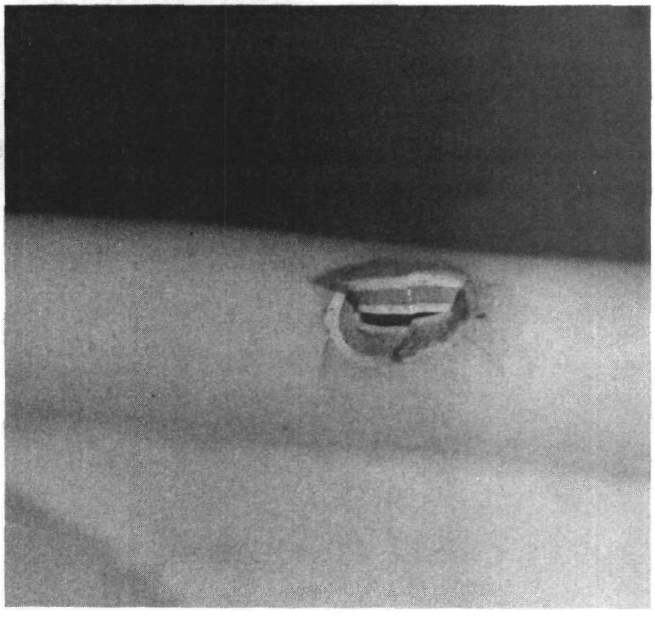


FIGURE 5. Photomicrograph of a Screwdriver-Blade Puncture of an 8YLY-S6 Glovebox Glove



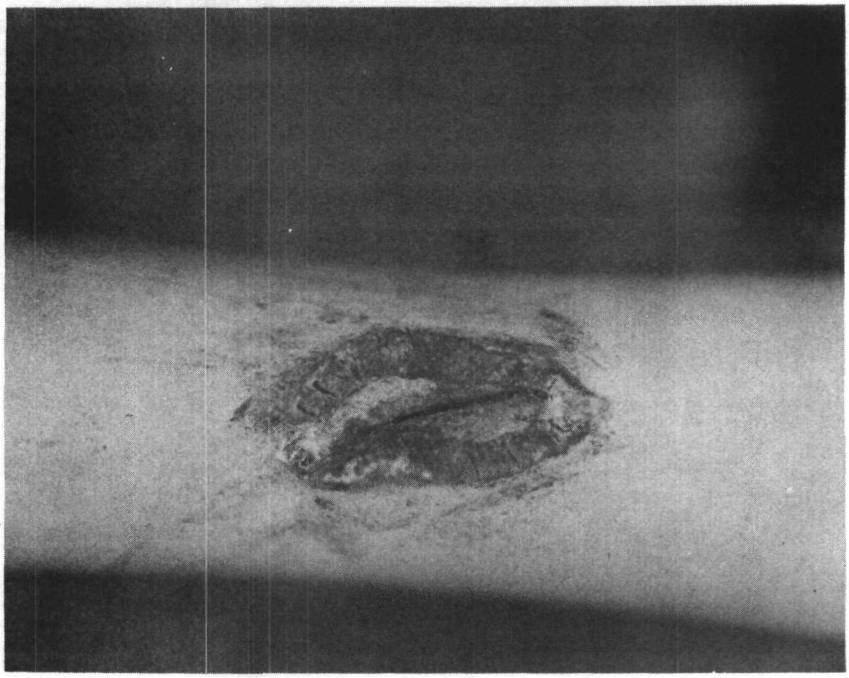


FIGURE 6. Photomicrograph of Deterioration of the Outer Layer of an 8YLY-S6 Glovebox Glove

FIGURE 7. Photomicrograph of a Glove Defect Resulting From Pinching

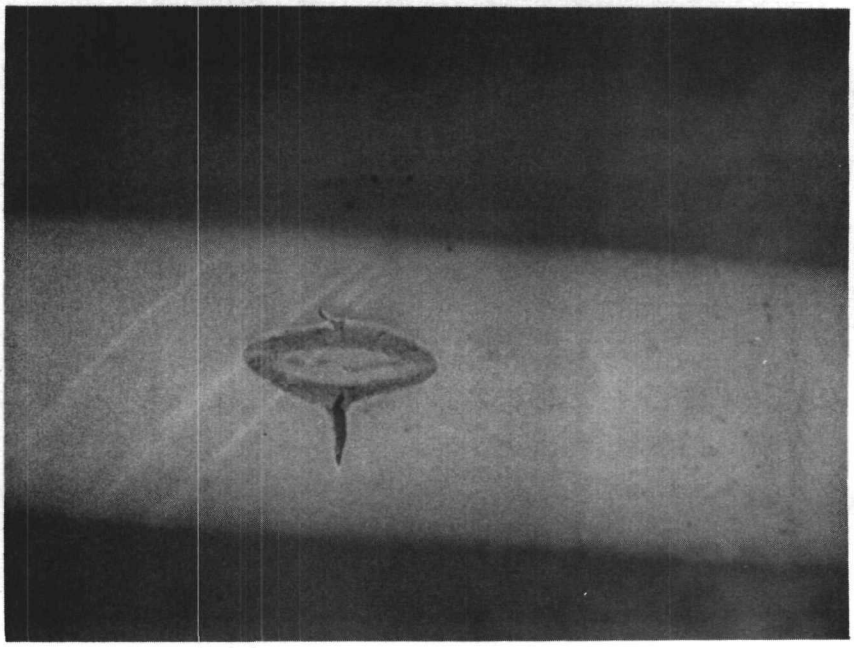
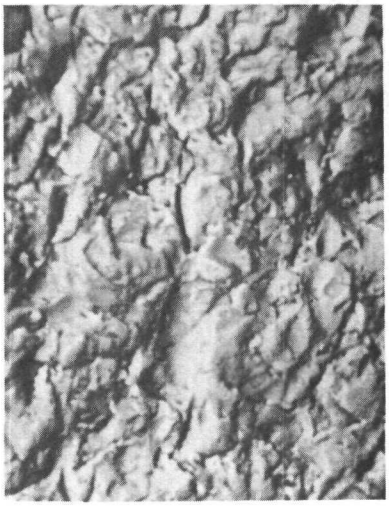


FIGURE 8. Micrographs (1000X Scale Expansion) of Grit-Blast Surfaces



a. Waist.

b. Mid.

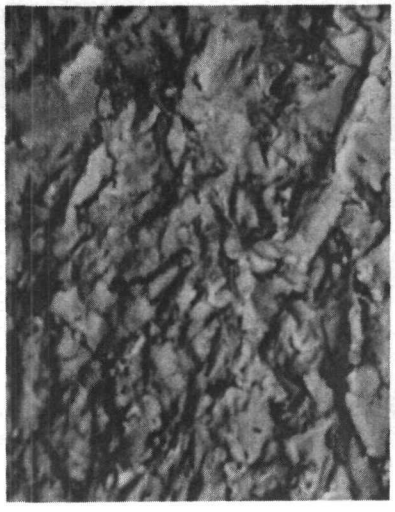
c. Pole.

d. Waist.

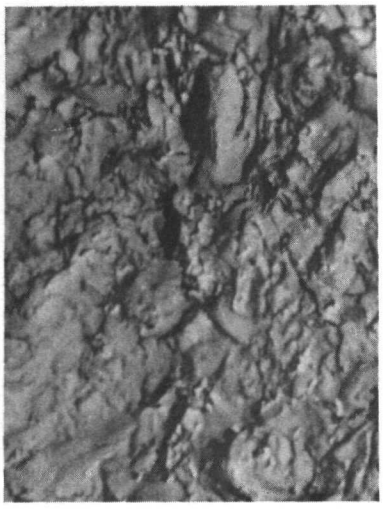
e. Mid.

f. Pole.

g. Waist.



h. Mid.



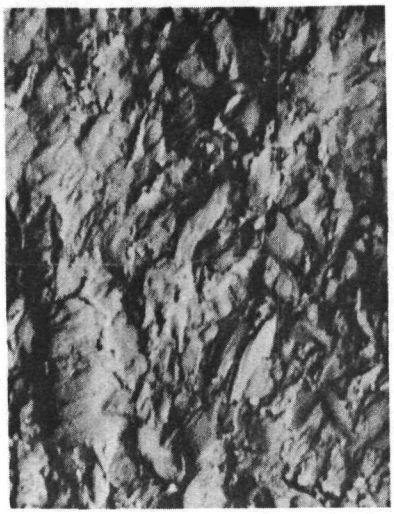
i. Pole.

FIGURE 9. Micrographs (1000X Scale Expansion) of Grit-Blast Surfaces

a. Waist.

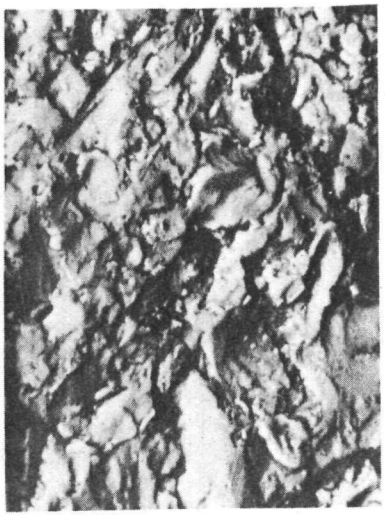
b. Mid.

c. Pole.



d. Waist.

e. Mid.



f. Pole.

g. Waist

h. Mid.

i. Pole.

FIGURE 10. Micrographs (1000X Scale Expansion) of Grit-Blast Surfaces

a. Waist.

b. Mid.

c. Pole.

d. Waist.

e. Mid.

f. Pole.

g. Waist.

h. Pole.