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QUALITY ENGINEERING AND CONTROL
SEMIANNUAL PROGRESS REPORT
MAY THROUGH OCTOBER 1977

SERVICE LABORATORIES GROUP

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**ROCKWELL INTERNATIONAL
ATOMICS INTERNATIONAL DIVISION
ROCKY FLATS PLANT
P. O. BOX 484
GOLDEN, COLORADO 80401**

**Prepared under Contract EY-76-C-04-3533
for the
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REA

SUBJECT DESCRIPTORS

Actinides	Phosphoric Acid Esters
Alloys	Plutonium
Atomic Absorption Spectroscopy	Plutonium Alloys
Brazing Alloys	Plutonium Compounds
Chemical Properties	Quantitative Chemical Analysis
Chemical Reactions	Radiation Effects
Decomposition	Radiolysis
Differential Thermal Analysis	Radiometric Analysis
Electron Microscopy	Radon
Electropolishing	Reactivity
Fluorides	Salts
Gallium Alloys	Sample Preparation
Gas Analysis	Soils
Ground Water	Specific Ion Electrodes
Incinerator Ash	Sulfates
Infrared Spectroscopy	Surface Cleaning
Liquid Chromatography	Surface Properties
Liquid Scintillation Counting	Thermal Analysis
Mass Spectroscopy	Thermogravimetric Analysis
Materials Compatibility	Topography
Metalworking Fluids	Tritium
Microscopy	Turbidimetry
Oils	Zirconium

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ATOMIC ABSORPTION SPECTROSCOPY

ALUMINA CONTENT OF FIREBRICK

John D. Grooms

Summary

Vendor specifications for alumina in firebrick employed in a rotary kiln can be checked by a rapid, reliable atomic absorption spectroscopy method. Aluminum concentrations of from 20 to 60 weight percent are routinely determined with a relative accuracy of about 10 percent.

Experimental

Composition of firebricks used in a rotary kiln incinerator was suspected as the cause for operational problems. An atomic absorption analytical technique was developed to verify alumina content. Aluminum (Al) concentrations from 20 to 60 weight percent are rapidly and accurately determined by this method.

In the procedure, 5-gram samples are broken from the brick, crushed, and ground to a fine powder in a boron carbide mortar. Two 0.10-gram aliquots of the powder are weighed and intimately mixed with a caustic flux consisting of 1 gram sodium carbonate and 0.8 gram lithium metaborate. The samples are then fused in platinum crucibles at 900 °C for 1 hour. The resultant melt is cooled for 30 minutes then dissolved in aqueous 6 molar hydrochloric acid and brought to 0.1-litre volume with distilled water. Appropriate dilutions are made and the solution is then analyzed for Al by atomic absorption spectroscopy, which permits a relative accuracy of about ± 5 percent of the amount

of Al present. Four brick formulations were analytically determined for Al content. Results obtained from three of the bricks agreed well with the vendor's stated formulation, whereas the fourth brick was found to be 10 percent below the specified alumina content.

GALLIUM AND ZIRCONIUM IN
PLUTONIUM ALLOYS

John D. Grooms

Summary

Zirconium (Zr) and gallium (Ga) have been found by X-ray spectroscopic examination to be preferentially alloyed as a binary impurity in various ternary alloys of plutonium (Pu) produced by the Plutonium Metallurgy Research and Development Group. A technique for separating and analyzing these alloys for Zr-Ga fractions has been developed. Concentrations of these two metals ranging from 50 parts-per-million by weight to 100 weight percent can be determined by conventional atomic absorption spectroscopic techniques with an accuracy of better than relative 10 percent of the amounts present.

Experimental

Duplicate aliquots of the ternary Pu-Ga-Zr alloy are dissolved in aqueous 2 normal (N) hydrochloric acid (HCl). The improperly alloyed Ga-Zr fraction is not soluble in the acid and is subsequently separated from the solution by filtration with a 0.1-micrometre, type VCWP, Millipore® filter. The collected powder, confirmed by emission spectroscopy determination to be a Pu-free Ga-Zr binary

alloy, is dried, weighed, and dissolved in aqueous 6N HCl-1N hydrofluoric acid solution. The resultant stock solutions are then analyzed by conventional atomic absorption spectroscopy.

ELECTRON MICROSCOPY

INFLUENCE OF SUBSTRATE ETCH ON THE TOPOGRAPHY OF PLUTONIUM SURFACES

Davis F. Carpenter

Summary

Prior to the deposition of plutonium (Pu) layers, substrates are subjected to an etch cycle. The purpose of this investigation was to determine the importance the degree of etch has upon the surface topography of the Pu layer. It was found that substrate topography plays a significant role in determining the frequency of domes and furrows in deposited Pu surfaces.

Experimental

Because preservation of the samples was required, a replica technique coupled with an image-reversal procedure was employed to record the surfaces used in this study (refer to previous Progress Report, RFP-2644, July 14, 1977, pages 16-17). The microscopy investigation was conducted upon two sets of surfaces that were etched for different periods of time. All other parameters remained the same. The experimental design of this study consisted of examining the surfaces prior to etching, after etching, and after depositing the Pu layer. Figures 1-a through 1-i are typical micrographs of such a surface that was ultimately influenced by excessive etching of the substrate.

Micrographs shown as Figures 1-a through 1-c illustrate the as-machined surface of the substrate prior to etching. The blemishes that break the continuity of the machined grooves are possibly caused by the cutting tool encountering areas of

high hardness, such as carbide grains. Evidence indicates that these blemishes sometimes survive through the etch cycle and are manifested as super domes such as the one found extending across the left side of the micrograph shown as Figure 1-g. Micrographs shown as Figures 1-d through 1-f are characteristic of a highly etched substrate. The surface has a sintered appearance. The straight, sharply-defined machine cuts are somewhat ragged; however, the craters are most noteworthy. These craters are sites where substrate material has been *scooped* out of the surface. Micrographs shown as Figures 1-g through 1-i depict the surface of the Pu layer. Furrows, which would normally show up at this point in a short etch cycle, are conspicuously absent in these micrographs, which are characterized by a high density of domes.

Conclusions drawn from this study are:

- (1) Long etch cycles form many craters, which in turn form domes in high density; short etch cycles will cause fewer domes to be formed.
- (2) Dome formation is possibly the result of at least two mechanisms: (a) Blemishes in the as-machined surface will tend to form large domes; (b) craters remaining after etching will tend to form the smaller domes.
- (3) Long etch cycles will tend to cause the furrows to be less frequent in the Pu topography. Conversely, short etch cycles will allow the machine tool marks to manifest themselves as furrows in the Pu surface.

INFRARED SPECTROSCOPY

SODIUM CARBONATE IN INCINERATOR ASH

Karl N. Kirchner and Mary L. Grooms

Summary

Fluidized bed incinerator ash samples are analyzed for sodium carbonate (Na_2CO_3) content by infrared spectroscopy. Two different wavelengths are used and allow for a concentration range from 0.2 to 100 percent.

FIGURE 1. Typical Micrographs of Surface Ultimately Influenced by Excessive Etching of the Substrate (Magnification 200X)

Machined Surface Prior to Etching

a. Waist

b. Mid

c. Pole

Machined Surface After Etching

d. Waist

e. Mid

f. Pole

Etched Surface Deposited with Plutonium

g. Waist

h. Mid

i. Pole

Experimental

Standards were prepared by adding dry Na_2CO_3 [0.01-0.15 milligrams (mg) for lower concentrations; 0.1-1 mg for higher concentrations] to dry potassium bromide, pressing pellets, and recording the infrared spectra. The absorbances of the lower concentration pellets were measured at 6.95 micrometres (μm); the higher concentration pellets at 11.4 μm . Two separate Beer-Lambert plots were made at these wavelengths, using the baseline method for measuring absorbances. The reproducibility of the standards was better than 10 percent over both wavelength ranges.

A trial pellet of the dried sample was pressed and scanned to approximate the concentration of Na_2CO_3 . The actual weight of sample to use was determined from the trial pellet. The percentage of the Na_2CO_3 was calculated from the standard Beer-Lambert plots and known sample weights.

IRRADIATED PARAFFINIC BASE MINERAL OIL

Roger S. Cichorz

Summary

An additive-free paraffinic base mineral oil (Shell Vitrea® 46 Oil) that had been irradiated with a cobalt 60 gamma source in air underwent two principal chemical changes. Infrared spectroscopy indicated that both oxidation and dehydrogenation had occurred in the irradiated oil compared to the nonirradiated reference oil.

Experimental

The oil 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. The Shell Vitrea samples were housed in a 0.1-millimetre path-length potassium bromide

cavity cell in order to obtain relatively-thick film spectra that would show subtle differences between the samples. The observed changes in the infrared spectrum of the irradiated oil when compared to that of the reference oil indicated that only oxidation and dehydrogenation had occurred. The oxidation is indicated by the appearance of a broad carbonyl stretching frequency ($\nu_{\text{C=O}}$); dehydrogenation is indicated by the appearance of an olefinic stretching frequency ($\nu_{\text{C=C}}$) and several olefinic bending frequencies ($\delta_{\text{C-H}}$). The observed changes in the infrared spectrum of the irradiated oil are summarized in Table I and depicted in Figure 2.

The formation of olefinic ($-\text{C}=\text{C}-$) bonds because of dehydrogenation of saturated carbons of the linear skeletal chain (i.e., linear alkene formation) and of branched alkyl carbons off the skeletal chain (i.e., linear alkene formation and terminal methylene group formation) of the oil is more pronounced than dehydrogenation of the terminal alkyl groups (i.e., terminal vinyl group formation). This is probably because of statistical considerations: there are relatively fewer terminal alkyl C-C sites compared to alkyl C-C sites in both the main skeletal chain and secondary branched chains available for olefin formation.

TABLE I. Frequencies in the Infrared Spectrum of Irradiated Mineral Oil Attributed to the Effects of Radiolysis

Absorption (cm^{-1})	Assignment	Attribution
1750 to 1690 (broad)	$\nu_{\text{C=O}}$	Oxidation (formation of ketones, esters, carboxylic acids).
1675 to 1645 (broad)	$\nu_{\text{C=C}}$	Olefinic $-\text{C}=\text{C}-$ formation because of dehydrogenation.
1020	$\delta_{\text{C-H}}$	$\text{RHC}=\text{CHR}$ (olefinic double bonds in other than terminal carbons of the skeletal and $>\text{C}_2$ branched chains).
990, 910	$\delta_{\text{C-H}}$	$\text{RHC}=\text{CH}_2$ (terminal vinyl groups).
890	$\delta_{\text{C-H}}$	$\text{RR}'\text{C}=\text{CH}_2$ (terminal methylene groups).

ν = stretching frequency; δ = bending frequency

FIGURE 2. Infrared Spectra of Shell Vitrea Oils

DETERMINATION OF DIBUTYLDIETHYLCARBAMYL PHOSPHATE IN AQUEOUS NITRIC ACID SOLUTIONS

Patricia A. Hyman

Summary

A method has been developed for ascertaining the solubility of dibutyldiethylcarbaryl phosphate (DBDECP) in various molarity solutions of nitric acid (HNO₃). The concentration of trace quantities of DBDECP in an aqueous nitric acid solution can be determined by extracting the solution with carbon tetrachloride (CCl₄) and quantitatively measuring, by infrared spectroscopy, the alkyl (C-H) absorption of DBDECP in the CCl₄ extract.

Experimental

Nonpolar solvents quantitatively extract DBDECP from aqueous solutions, since the phosphate ester is only slightly soluble in water. Being both nonpolar and an excellent solvent for infrared spectroscopic organic determinations, CCl₄ was consequently chosen as the extraction medium for DBDECP in aqueous nitric acid. The DBDECP that is extracted into the CCl₄ can be subsequently determined by infrared spectroscopy by measuring its C-H absorption and relating it to a standard Beer-Lambert calibration plot. This method, however, is valid only when DBDECP is the sole extractable C-H component in the aqueous solution.

Standard solutions of DBDECP in the concentration range of 80 to 4800 parts per million by volume (ppm) were prepared in 15 percent HNO_3 . Each standard was quantitatively extracted with CCl_4 , and the C-H infrared absorbance of the CCl_4 extracts at 3.4 micrometres (μm) was measured by the baseline method. The standard extracts were each 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. The Beer-Lambert plot of absorbance at 3.4 μm versus

concentration of DBDECP in CCl_4 was linear over the concentration range of 800 to 2400 ppm. A typical Beer-Lambert plot is given in Figure 3.

Since the samples are analyzed in exactly the same manner as the standards, a recovery factor was unnecessary. The reproducibility for the standard determinations was ± 2 percent. The overall uncertainty in the procedure was calculated from recovery studies to be ± 9 relative percent.

FIGURE 3. Beer-Lambert Calibration Plot for Dibutyldiethylcarbaryl Phosphate in Carbon Tetrachloride

LIQUID CHROMATOGRAPHY

DETECTION OF BROMACIL IN SOIL

Karl N. Kirchner and William A. Gies

Summary

Bromacil (5-bromo-3-*sec*-butyl-6-methyluracil) is a component in several commercial herbicides used at the Rocky Flats Plant. As part of an environmental sampling program, soil samples are analyzed for residual bromacil after periods of herbicide application. High-performance liquid chromatography (HPLC) is used to routinely separate and detect bromacil at a lower limit of detection of 4 nanograms.

Experimental

Bromacil standards, prepared in hexane, are injected into a Waters liquid chromatograph, equipped with a μ -Bondapak-C₁₈® column, using a solvent system of 50-percent methanol and 50-percent water, and a flow rate of 1 millilitre per minute. It has been determined that the 280-nanometre wavelength ultraviolet detector permits a lower detection limit of 4 nanograms bromacil.

An extraction step is employed in the analysis of soil samples for bromacil. Soil samples weighing from 40 to 45 grams are treated with 15-percent dichloromethane and 85-percent hexane in a Soxhlet apparatus. The solvent containing extracted herbicide is evaporated to one-tenth of its original volume, and the concentrate injected into the liquid chromatograph. For this method, the lower detection limit for bromacil in soil is approximately 100 parts per billion.

NITROSAMINES IN SYNTHETIC CUTTING FLUIDS

Mary L. Grooms

Summary

N-nitrosodiethanolamine (NDEIA) has been detected in a commercial cutting fluid concentrate (Cincinnati

Milacron Cimcool® CX3OS) used at Rocky Flats. The method for detection of nitrosamines in metalworking fluids involves an initial separation on silica gel followed by additional separation and detection with liquid chromatography.

Experimental

A 1-millilitre (ml) portion of cutting fluid concentrate was mixed with 10 grams magnesium sulfate. The mixture was rinsed several times with ethyl acetate and dried with anhydrous sodium sulfate. The ethyl acetate solution was concentrated to approximately 1 ml and then loaded onto a silica gel column.¹ The column was rinsed with ethyl acetate to elute some components of the sample. The remaining components, including the NDEIA, were eluted with acetone. The acetone fraction was concentrated and injected into a liquid chromatograph equipped with an ultraviolet detector and a μ -Porasil column. The solvent system consisted of 5 volume percent methanol and 95 volume percent methylene chloride.

The concentration of NDEIA was calculated from the peak height of the sample on the chromatogram versus that of a known standard solution. Both sample and standard peaks eluted at the same retention time. Recovery studies were performed, and 70 percent recovery was attained for standard NDEIA solution in a mixture of 5 weight percent triethanolamine and 5 weight percent sodium nitrite in water.

The separatory steps and liquid chromatographic determination may be adapted to various cutting fluids to check for nanogram quantities of NDEIA. The potentially-carcinogenic NDEIA is a suspect impurity in synthetic cutting fluids composed of triethanolamine and alkali nitrites. This method has been developed to screen the fluids currently in use or proposed for use at Rocky Flats in order to prevent any undue employee exposure.

¹Tsi Y. Fan *et al.* "N-Nitrosodiethanolamine in Synthetic Cutting Fluids: A Part-Per Hundred Impurity". *Science* 196:70. April 1, 1977.

LIQUID SCINTILLATION COUNTING

TRITIUM IMPURITY LEVELS IN PLUTONIUM METALS AND OXIDES

Donald P. Anderson and Ronald N. Morris

Summary

Tritium in plutonium samples is converted to tritiated water vapor by heating the samples to 950 °C in a stream of moist oxygen gas. The evolved tritiated water is subsequently collected and measured by liquid scintillation counting techniques. The method allows for the detection of picocurie amounts of total tritium regardless of its form.

Experimental

Plutonium metal aliquots or plutonium oxide aliquots sealed in a tin capsule are dropped into a tube furnace heated to 950 °C. A flow of moist oxygen gas through the system oxidizes any tritium gas to tritiated water vapor. The oxygen flow sweeps all the evolved tritiated water vapor into a measured volume of water, and the tritium collected in water is then measured by liquid scintillation counting techniques.

The furnace set-up is patterned after a similar arrangement of the Los Alamos Laboratory. Exchange samples have been analyzed at Los Alamos and Rocky Flats with comparable results. After collecting the tritiated water vapor, the distilled water medium is checked for plutonium contamination. If a 0.5 millilitre aliquot measures less than 12 disintegrations per minute, a sample is submitted to the General Analytical Laboratory for liquid scintillation counting.

Accuracy of the method was checked with the assistance of the Rocky Flats Standards Laboratory. A weighed quantity of United States National Bureau of Standards tritiated water standard was encapsulated and dropped into the furnace. The collected tritium was measured by liquid scintillation counting, and the recovery was 78 percent. Replicate analyses of 10 plutonium oxide aliquots

indicate a relative standard deviation of ± 20 percent for this method. The advantage to the furnace technique is that it allows for the determination of total tritium in the sample regardless of its form.

MASS SPECTROSCOPY

DETERMINATION OF CARBON MONOXIDE AND NITROGEN FROM RESOLVED HIGH-RESOLUTION SPECTRA

Harold O. Pitts

Summary

Most samples of gas received for analysis by the Mass Spectrometry Laboratory contain varied amounts of carbon monoxide (CO) and Nitrogen (N_2). A method is described that improves the determination accuracy for certain CO- N_2 ratios when close control of these gases is required.

Experimental

When the partial pressures of CO and N_2 are equal or nearly equal in a mixture of gases, the quantity of each can be calculated from the following equations:

$$X + Y = T \quad (1)$$

$$AW + BY = F \quad (2)$$

Where X = peak height at mass-to-charge ratio (m/e) 28 due to N_2 ,

Y = peak height at m/e 28 due to CO,

A = cracking pattern or ratio of m/e 14 to m/e 28 when only N_2 is present (average value is about 0.08),

B = cracking pattern or ratio of m/e 14 to m/e 28 when only CO is present (average value is about 0.008),

T = net peak height at m/e 28, in the spectrum of the sample being analyzed, and

F = net peak height at m/e 14 in the spectrum of the sample being analyzed.

$$\text{And } V = 100 X \div S_N P \quad (3)$$

$$W = 100 Y \div S_{CO} P$$

Where V = volume percent of N₂ in mixture,

W = volume percent of CO in mixture,

S_N = instrument sensitivity of N₂,

S_{CO} = instrument sensitivity of CO, and

P = sum of all partial pressures in the mixture being analyzed.

When equations 1 and 2 are solved,

$$X = 13.9 F - 0.1 T \quad (4)$$

$$\text{and } Y = T - X. \quad (5)$$

The m/e 14 and m/e 28 peak heights in equation 4 are net values with the contributions from methane, ethane, carbon dioxide, other hydrocarbons, and background values deducted from the gross amount measured. The volume percents calculated from the above equations are dependent upon:

- (1) the cracking patterns of the gases used to remove other contributions to T and F,
- (2) the m/e 14 cracking patterns of CO and N₂ used for calculating the numerical coefficients of F in equation 4, and
- (3) the recording and measurement parameters of the spectrum detection system.

In the cases of some mixtures where the N₂-CO ratio is approximately 95 to 5 rather than 1 to 1, occurrence of error from one or more of the above variables can distort or obscure the lower result. When knowledge of the quantity of CO and N₂ is critical in a mixture, a second analysis and additional calculations can reduce the variability in equations 1 and 2. The second step is performed by analyzing the sample with the mass spectrometer adjusted for high resolution rather than high sensitivity. This permits separation and measurement of each of the compounds contributing to m/e 28. Probable peaks at m/e 28 are listed below.

<u>Compound or Fragment</u>	<u>*Molecular Weight (atomic mass units)</u>
Carbon Monoxide (CO ⁺)	27.99491
Nitrogen (N ₂ ⁺)	28.00614
Hydrocarbons (C ₂ H ₄ ⁺)	28.03128

*Based on carbon 12 standard.

After measuring, totaling, and normalizing the *resolved* peaks, the normalized values can be applied to the total m/e 28 peak height obtained from the normal or higher sensitivity analysis. This provides a direct calculation of the contribution to m/e 28 from CO, N₂, and hydrocarbons (if present), rather than having to depend upon the simultaneous equation as done in the first calculation method. Replicate analysis results for CO₂ in three mixtures are presented for statistical comparison of the two methods. Two analyses were performed without resolving the samples, and the third, exchange sample CS-6, was done by resolving the samples and using the alternate method of calculation.

<u>Sample</u>	<u>Carbon Monoxide (volume percent)</u>	<u>Number of Trials</u>	<u>Standard Deviation</u>
C1	20.4	20	0.93
C2	5.08	16	0.33
CS-6	0.92	15	0.07

SOLVENT PERMEATION RATES BY DYNAMIC TECHNIQUES

Tom L. McFeeters

Summary

Techniques of dynamic mass spectrometry have been applied to the determination of solvent permeation rates through various drybox glove materials. Time-related phenomena and stable permeation rates have been determined for carbon tetrachloride and Viton® fluoroelastomer.

Experimental

The permeation manifold for gas-permeation rate determinations was used. The gas supply line was

disconnected from each sample holder and the upper portion filled to a depth of 2.54 centimetres by pouring liquid-phase solvent directly on the sample. The upper portion of the manifold (including the pressure gauge) was replaced and evacuated to remove air. When only solvent vapor remained, the vacuum system was isolated, allowing direct measurement of solvent vapor pressure.

The manifold was then connected to the inlet of the Bendix Model MA-3 time-of-flight mass spectrometer previously calibrated in the dynamic mode of operation by vacuum-service permeation tubes² corresponding to the solvent used. Solvent permeation rates were then determined in the same general manner as gas permeation rates.

Solvent permeation of polymers may vary with time because of physical changes such as swelling or other solvent-polymer interactions, in addition to the more commonly recognized parameters such as surface area, pressure, and polymer thickness. A pattern for these time-related phenomena was observed for the solvent carbon tetrachloride and Viton samples. This pattern is illustrated in Figure 4 as a generalized depiction of the variation of carbon tetrachloride permeation with time. The time phases of permeation appear to be a function of sample thickness or volume and may be related to swelling of the polymer. Table II lists approximate values for the time phases of the permeation pattern and the values of the resulting stable permeation rate for eight Viton samples.

CLEANING OF HIGH-VACUUM METAL COMPONENTS BY ELECTROPOLISHING

Duane I. Hunter

Summary

An electropolishing technique to clean and polish the metal components of mass spectrometer ion-source parts has been adapted for use at the Rocky

Flats Plant. Source parts can be cleaned and polished with no scratching and essentially no metal removal.

Discussion

The electropolishing method developed by Peele and Brent³ has been adapted to clean and polish surfaces of 300 series stainless steel, including mass spectrometer ion-source components. The cleaning of stainless steel parts results in no surface abrasion and removal of less than 0.005 millimetre of metal surface. Therefore, parts with critical dimension and edges can be cleaned without damage. Electropolishing removes deposits from irregular surfaces which can not be cleaned by conventional methods. The smooth surface produced by electropolishing increases the homogeneity of the electrical fields around the ion-source parts and does not attract surface contamination as readily as a rough scratched surface.

Electropolishing equipment consists of a standard battery jar with lead plates hung on a copper rod around the jar edge. The size of the battery jar can vary to accommodate the part undergoing cleaning. A second copper rod holds a brass test tube that serves as the part holder. The rod and clamp are arranged to submerge the part in the electropolish solution. The temperature of the bath is controlled using a quartz immersion heater and a variable stirrer. Power is supplied by a plating power supply fabricated at Rocky Flats. In this arrangement, the work piece acts as the anode and the lead plates as the cathode.

Ashland Chemical Company Glowbrite® Electro-polish Solution No. 4 is used as the electropolishing medium. Chemical analysis of this product indicates a composition of approximately 60 volume percent (%) ortho-phosphoric acid, 25% sulfuric acid, and 15% water. Each type of metal or alloy requires a specific time, temperature, and current flow to achieve a clean polished surface with an absolute minimum of metal removal. For example, parts fabricated from 316 stainless steel polish best at a

²Tom L. McFeeters. "Permeation Tubes for Dynamic Mass Spectrometry Calibration," RFP-2521. Paper presented at the Eighteenth Annual Rocky Mountain Spectroscopy Conference. Denver, Colorado. August 2-3, 1976.

³Gerald L. Peele and David A. Brent. "Cleaning of Mass Spectrometer Ion Sources by Electropolishing." *Analytical Chemistry* 49:4. April 1977.

FIGURE 4. Variation of Carbon Tetrachloride Permeation with Time

TABLE II. Carbon Tetrachloride-Viton® Elastomer Permeation Rates

Sample	Thickness (metres $\times 10^{-4}$)	T_d (minutes)	T_m (minutes)	Q_m^* ($\times 10^{13}$) (kg/Pa·sec·m ²)	Q_s^* ($\times 10^{13}$) (kg/Pa·sec·m ²)
SP117-B-1-qq	5.1816	435	5760	5.54	1.73
SP117-B-1-u	5.3086	3230	29100	3.69	1.27
SP117-B-1-hhh	7.6708	6180	71760	3.69	1.62
SP117-B-1-ggg	7.5946	435	29100	1.39	0.84
SP117-C-1-E	8.3312	10080	72120	1.39	0.89
SP117-E-1-C	8.7376	10080	72120	1.39	0.77
SP117-B-1-ww	9.9568	6180	72120	1.96	1.39
SP117-B-1-zz	9.9822	6180	72120	2.42	1.50

T_d = time to detection, in minutes.

T_m = time to maximum permeation rate, in minutes.

Q_m^* = maximum permeation rate, in kilograms per pascal-second-metre².

Q_s^* = stable permeation rate, in kilograms per pascal-second-metre².

bath temperature of 70 to 80° C and 10 to 20 amperes current for 1 to 3 minutes. The current is varied depending on the size of the part; i.e., large parts require more current than parts of smaller dimensions. This method is limited to 300 and 400 series stainless steels and nickel-chromium alloy. Other metals require different electropolishing media and conditions. After electropolishing, the parts are rinsed in water, chromic acid cleaning solution, water, 1,1,2-trichloro-1,2,2-trifluoroethane or acetone, and methanol in that order. The parts are then dried using an infrared heat lamp or oven.

RADIOMETRIC ANALYSIS

ACTINIDES IN SOIL SAMPLES

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

Summary

A procedure has been developed for determining thorium (Th), uranium (U), neptunium (Np), plutonium (Pu), americium (Am), and curium (Cm) using only three separation schemes per sample. The method has the same accuracy and precision as the previous method and the advantage of less time required for the chemical separations.

Experimental

The first separation scheme involves taking a weighed aliquot of soil and digesting it in a nitric acid (HNO_3)-hydrofluoric acid mixture. The Th, Np, and Pu are sequentially separated from this digestion solution and from each other by employing a series of separation steps on a column loaded with anion exchange resin. The second separation scheme involves the potassium pyrosulfate fusion of another weighed aliquot of soil. The fusion product is dissolved in 0.5 normal HNO_3 , and Am and Cm separated from the rest of the soil matrix by means of cation-exchange resin chromatography. The third separation scheme also involves fusion of

a weighed aliquot of soil in potassium pyrosulfate. The fusion product is dissolved in 8 normal HNO_3 , and U separated from the balance of the soil matrix by elution on a column loaded with anion exchange resin.

These separations offer several advantages over previous techniques. Sample preparation time has been reduced since the number of aliquots has decreased from 22 per sample to only 10. The sequential isolation of 3 elements (Th, Np, and Pu) from 1 set of aliquots also reduces the time required for the chemical separations. This method has the same precision and accuracy as the individual separations used previously. The lower detection limits for each element by this procedure are in disintegrations per minute per gram: Th = 0.2, U = 0.2, Np = 0.2, Pu = 0.2, Am = 0.6, and Cm = 0.6.

RADON IN GROUND AND WATER

Jack L. Long, Terry F. Rees, Duane I. Hunter

Summary

The activity and behavior of naturally-occurring radon gas in ground water taken from an outside sump were examined by displacing the gas by nitrogen sparging and applying liquid scintillation counting techniques. Sample activity attributable to radon after 4 days decreased by a factor of 50 times.

Experimental

Radon (Rn) gas was determined in sump water by nitrogen sparging and collecting the displaced gas phase in a Lucas scintillation cell. An initial increase in count rate with time followed by a decrease was observed. This phenomenon was attributed to the growth of radioactive daughters. After secular equilibrium was established, the cell was evacuated, and the activity remaining in the cell decreased at a rate equivalent to a shorter half life than the equilibrated mixtures. A 20- to 25-minute half life was observed, which is consistent with a mixture of lead²¹⁴ and bismuth²¹⁴, the expected products

from the decay of Rn^{222} . The presence of radium²²⁶, the precursor of Rn^{222} , was confirmed by gamma-ray spectroscopy.

The next step in the investigation involved air sampling of the sump from which the water samples were taken. The air sample was obtained by means of a tube of stainless steel that terminated within 6 inches of the water's surface. After the tube was purged for 5 minutes by a diaphragm pump, the evacuated Lucas cell was filled. The sample activity was 0.2 microcuries (4×10^5 disintegrations per minute) per cubic metre at ambient conditions.

The air sampling of the sump was repeated 4 days later. The pump, which removed the water from the sump, had stopped between the 2 samplings, and the water level had risen approximately 5 feet. This time, the activity was found to be 0.004 microcuries (9×10^3 disintegrations per minute) per cubic metre. The drop in activity was probably caused by the reduced flow of water, either because Rn is not transported to the sump from its source, or because of lack of agitation without pumping allows Rn to remain in the liquid, or both.

THERMAL ANALYSIS

CHARACTERIZATION OF VOLATILES FROM INORGANIC GLASS FORMULATIONS

Keith J. Grossaint

Summary

The decomposition temperatures were determined for seven mixtures of alkali salts. The volatile components were identified and correlated with pertinent temperature ranges to establish which salts were decomposed. Maximum rates of decomposition-volatilization and the relative amounts of various gases evolved from the mixtures were determined at specific temperatures.

Experimental

The technique of combined thermal analysis-mass spectrometric analysis (TA-MS) was used to identify component gases evolved during formation of experimental inorganic glass formulations. The glass

formulation is intended for encapsulation of radioactively-contaminated waste in storage applications. The determination of evolved gases was needed for environmental reasons, as well as for equipment design considerations.

The formulations tested included mixtures of alkali carbonate, alkali phosphate, alkali nitrate, and an ash recovered from incineration of nonradioactive waste. The incinerated waste was typical of waste generated in nuclear materials handling, and represented ash generated at Rocky Flats from incineration of contaminated rags, paper, gloves, plastic bags, and other combustible process materials.

The thermogravimetric analysis (TGA) thermograms in Figure 5 illustrate the volatilization behavior of the various test mixtures. Dynamic mass spectral analysis of the effluent gases, as they evolved, enabled the correlation of compound decomposition with temperature. The maximum rate of gas evolution was determined for each thermal event, from the thermograms. A compilation of percent weight loss, component evolved, rate, and the compound which decomposed is given in Table III.

There were several features of interest. The early decomposition of nitrate ion involved formation of nitrite with evolution of oxygen gas, but subsequent decomposition progressed with evolution of nitric oxide, nitrogen, and nitrous oxide gases. The different decomposition paths of nitrate were not distinguishable in the TGA thermograms, but were detected by the dynamic TA-MS technique.⁴ Temperature ranges for loss of hydrated water were established, carbonate decomposition temperatures were noted, and volatilization of alkali oxide and sodium hydroxide was confirmed. The volatilization of alkali oxide resulted in chemical reaction with quartz components of the TGA balance beam. No evidence for evolution of chlorides (from the ash) or volatile phosphorous compounds was found. Traces of oxygenated organics detected in one sample were attributed to incomplete incineration of the ash.

⁴ Keith J. Grossaint. *Gaseous Pyrolysis Products Identified by Combined Thermal and Mass Spectrometric Analysis*. RFP-2297. Rocky Flats Plant, Rockwell International, Golden, Colorado. May 13, 1975.

FIGURE 5. Thermogravimetric Analysis Results (Thermograms) for Experimental Salt Mixtures

TABLE III. Thermal Analysis-Mass Spectrometric Results for Mixtures of Inorganic Compounds

Sample	Temperature Range (°C)	Weight Loss (Weight Percent)	Components Evolved	Maximum Rate (milligrams per minute)	Decomposition
1	25 to 180	41.1	water	23.	water desorption
	180 to 250	2.84	water	2.0	and hydrated water
	250 to 490	0.00	---	---	
	490 to 800	2.84	oxygen, nitric oxide, nitrogen	0.3	nitrate
	800 to 1030	1.42	nitric oxide, nitrous oxide	0.2	nitrate
2	25 to 160	31.0	water	6.0	water desorption
					and hydrated water
	160 to 240	0.00	---	---	
	240 to 340	4.18	water	1.0	hydrated water
	340 to 530	0.00	---	---	
	530 to 660	2.51	nitrogen, nitric oxide, oxygen	0.5	nitrate
3	25 to 150	41.2	water	20.	water desorption
	150 to 220	2.44	water	0.75	and hydrated water
	220 to 380	0.00	---	---	
	380 to 660	2.82	nitrogen, nitric oxide, oxygen	0.3	nitrate
	660 to 1000	0.00	---	---	
4	25 to 160	5.62	water	1.2	water desorption
	160 to 500	0.00	---	---	
	500 to 870	13.0	nitric oxide, oxygen, nitrogen, nitrous oxide	1.0	nitrate
	870 to 970	3.90	water, sodium oxide	0.5	alkali hydroxide
5	25 to 160	5.84	water	1.5	water desorption
	160 to 400	0.78	carbon dioxide, nitric oxide	0.2	bicarbonate, nitrate
	400 to 860	13.5	nitrous oxide, carbon dioxide	1.2	nitrate, carbonate
6	25 to 160	8.91	water	10.	water desorption
	160 to 320	0.18	water		water desorption
	320 to 500	1.78	carbon dioxide, alcohols and carboxylic acids (trace)	0.1	bicarbonate and organics
	500 to 760	9.45	nitric oxide, nitrogen, oxygen, nitrous oxide	0.5	nitrate
	760 to 980	2.14	carbon dioxide	0.1	carbonate
7	25 to 210	6.19	water	1.8	water desorption
	210 to 410	2.03	water	0.1	water desorption
	410 to 900	2.03	water, nitric oxide, nitrogen, oxygen, sodium oxide	7.0	nitrate and alkali hydroxide

TABLE IV. Relative Abundances of Gaseous Components above Inorganic Mixtures at Specified Temperatures

Sample	Temperature (°C)	Components (Volume Percent)								total hydrocarbons
		water	carbon dioxide	nitric oxide	nitrous oxide	oxygen	nitrogen	hydrogen	argon	
1	950	49		0.66	0.82	38.0	3.3	7.3	0.04	1.0
2			not analyzed							
3	950	5.7	16.1	59.5	1.4	0.3	13.5	3.1	0.02	0.43
4	800	3.1	93.3	0.58		0.07	0.56	2.0	trace	0.39
5	800	0.92	83.6	10.5	1.0	0.08	3.2	0.45	0.02	0.12
6	900	trace	77.6	18.6			3.6	0.23		trace
7	500	34.5		0.54	2.0	2.4	19.5	0.40	0.06	0.0

Sample was evacuated to 10 torr or less prior to heating. [For SI (metric) use: 1 torr = 133 pascals.] Hydrated water was lost from some samples during evacuation. Total hydrocarbons are reported as detectable hydrocarbons, including methane, ethane, propane, butane, and benzene.

The relative amounts of volatiles generated by heating in a closed container at specified temperature are listed in Table IV. The data in Tables III and IV will be used to determine scrubbers and additional system designs required for the process of encapsulation.

REACTIONS OF ALCOHOLS WITH NITRIC ACID

Roger S. Cichorz

Summary

Sealed-capillary differential thermal analysis (DTA) experiments were performed on equal volume mixtures of alcohols and nitric acid (HNO₃) to assess their relative reactivities. The DTA results indicate that for 8 molar HNO₃, the nitration (esterification) reaction is prevalent over the oxidation reaction.

Experimental

The sealed-capillary DTA technique was employed in this investigation to insure intimate contact of the materials throughout the experiment. This study was done to support a research and development investigation on the relative reactivities of alcohols. The alcohols tested were methanol, ethanol, propanol, isopropanol, butanol, and ethylene glycol (1,2-ethanediol). For this study,

8 molar (50 percent aqueous concentrated) HNO₃ solution was chosen because it is prevalent in the various processing areas at Rocky Flats. Equal volumes of HNO₃ and the test alcohol were used for these DTA experiments.

In the DTA trials, all of the alcohols reacted exothermically with HNO₃ above room temperature. The exotherms, in general, were medium to large over a relatively narrow temperature range, indicating the reactions occur rapidly once the critical reaction temperature is reached. Of the alcohols tested, isopropanol appears the most reactive while methanol is the most stable. The results of duplicate DTA trials are given in Table V.

Theoretically, there can occur at least two reactions in this acid-alcohol system: esterification to alkyl nitrites and oxidation. The nitration reaction (1) proceeds by way of an S_N1 (unimolecular nucleophilic substitution) mechanism, even in aqueous solutions:⁵

⁵ Carl R. Noller. *Chemistry of Organic Compounds*. Third Edition. W. B. Saunders Company, Philadelphia. 1965. Page 146.

TABLE V. Differential Thermal Analysis Results for 1 to 1 by Volume Mixtures of Alcohols and 8 Molar Nitric Acid

Alcohol	Trial	Exotherm		
		Start (°C)	Maximum (°C)	End (°C)
isopropanol	1	78	117	150
	2	74	104	148
ethanol	1	118	133	154
	2	97	109	128
propanol	1	130	145	158
	2	78	108	115
ethylene glycol	1	125	145	155
	2	130	149	167
butanol	1	144	149	159
	2	150	159	170
methanol	1	173	188	205
	2	168	190	207

The rate of an S_N1 reaction is affected by electronic factors rather than steric factors. Consequently, the order of reactivity should be the same as the order of stability of carbonium ions; i.e., tertiary > secondary > primary ($\text{CH}_3\text{CH}_2- > \text{CH}_3-$). This is consistent with the observed DTA results, in which isopropanol, a secondary alcohol, is the most reactive, and methanol, with the smallest possible alkyl group on a primary alcohol, is the least reactive of the alcohols tested. Infrared spectroscopic examination of the reaction products confirms that the nitration reaction is predominant over the oxidation reaction when equal volume mixtures of HNO_3 and alcohol are employed.

The oxidation of alcohols by HNO_3 apparently occurs only when there is an excess of strong oxidizing agent. Primary alcohols are oxidized by HNO_3 to produce carboxylic acids without loss of carbon. This reaction (2) proceeds through an aldehyde intermediate:

$$\text{R}_2\text{CHOH} \xrightarrow{(\text{O})} \text{R}_2\text{CO} \quad (3) \text{ Tertiary alcohols are generally stable to this type of oxidation. In general, DTA results for the } \text{HNO}_3 \text{-oxidation of organic materials, when the nitration-esterification reaction does not occur, are characterized by small- to}$$

medium-sized exotherms that start above 200°C and proceed over a broad temperature range, probably because of nonspecific oxidation-decomposition mechanism.

METAL-CARBON TETRACHLORIDE MIXTURES

Patricia A. Hyman and Roger S. Cichorz

Summary

Sealed-capillary differential thermal analysis (DTA) experiments were performed on a series of metal-carbon tetrachloride (CCl_4) mixtures to assess the reactivity of a particular metal with CCl_4 . The experiments were designed to determine whether reaction onset temperatures were affected by the physical state (i.e., liquid or vapor) of the CCl_4 in contact with the metal, since it was anticipated that exothermic-reaction onset temperatures would be significantly lower for cases in which the metal contacted CCl_4 vapor. The DTA data indicated no significant differences in reaction onset temperatures between the vapor and liquid phase experiments with the various metals tested (plutonium, aluminum, gallium, brass, copper, and zinc).

Experimental

A particular metal and CCl_4 liquid are placed in a quartz capillary tube which is then sealed. For the experiment requiring the metal to be in contact with CCl_4 liquid, a maximum amount of CCl_4 is added to the capillary before sealing. Even at elevated temperatures (greater than 76.7°C , the boiling point of CCl_4), not all the CCl_4 can vaporize, and the metal remains in contact with liquid CCl_4 throughout the analysis. For the vapor phase experiments, enough CCl_4 is added to just wet the metal, so that at temperatures above 76.7°C , the CCl_4 completely vaporizes and the metal is in contact only with CCl_4 vapor throughout the analysis.

FIGURE 6. Experimental Conditions for Sealed-Capillary Differential Thermal Analyses of Metal-Carbon Tetrachloride Mixtures

The capillary loading arrangements for this investigation are given in Figure 6.

All metals used, with the exception of aluminum foil, were in the form of freshly-filed fines to prevent using partially oxidized metals. Replicate DTA runs were made to 450 °C at a heating rate of 20 °C per minute on a DuPont 900® Thermal Analyzer.

A summary of the reactivity data is presented in Table VI. The data indicate that the physical state

of the CCl_4 does not significantly alter the onset temperatures of the observed exotherms. An effort has been made to minimize sample preparation time and to prevent excessive oxidation to the metals, thus increasing the reproducibility of the DTA data. Results for the aluminum- CCl_4 reaction have traditionally been difficult to reproduce. Table VI indicates that minimal exposure of the aluminum to the atmosphere aided in reducing the relative standard deviation of the mean exotherm temperature in 5 replicate analyses to approximately ± 1 to ± 4 percent.

MISCELLANEOUS PROJECTS

FLUORIDE CONTENTS OF PLUTONIUM FLUORIDES

Reuben J. Sironen and Robert A. Sandberg

Summary

A fluoride specific-ion electrode has been used to determine the fluoride content of plutonium fluorides following pyrohydrolysis. This method is faster and more specific than the titration method previously used.

Experimental

Historically, fluoride in plutonium fluorides has been determined at Rocky Flats by passing steam over the sample which is heated to 800 °C in a tube furnace. The hydrogen fluoride (HF) gas that results is swept and collected in a water trap and subsequently titrated with standard base. Recently, a substitute method was introduced that involves direct measurement of fluoride ion (F^-) by specific-ion electrode following pyrohydrolysis. This is faster than the titration method and is not influenced by any other source of hydrogen ion, such as from an impure plutonium fluoride. Prior to measuring F^- concentration, the sample is buffered to a pH of from 5 to 6 in order to destroy any HF and HF_2^- complexes which would cause a low F^- measurement. The samples are compared to buffered standard F^- solutions of comparable total ionic strength.

TABLE VI: Differential Thermal Analysis
Data for Metal-Carbon Tetrachloride Mixtures

Metal	Phase	No. of Exotherms	Exotherm ^a		
			Start (°C)	Maximum (°C)	End (°C)
aluminum fines	liquid	1	285 ± 8	302 ± 4	310 ± 9
	vapor	1	283 ± 4	300 ± 5	322 ± 14
aluminum foil	liquid	1	348	358	378
	vapor	1	344	355	366
gallium fines	liquid	2	145 ± 27	168 ± 23	199 ± 25
			273 ± 14	306 ± 5	331 ± 19
	vapor	2	150 ± 30	170 ± 32	202 ± 16
			291 ± 6	323 ± 14	353 ± 23
brass fines	liquid	3	160	169	182
			243	263	296
			338	352	378
	vapor	3	156	168	186
			240	257	276
			322	363	407
copper fines	liquid	4	134 ± 10	160 ± 3	184 ± 2
			238 ± 8	282 ± 11	307 ± 7
			327 ± 14	347 ± 11	377 ± 8
			374	392	406
	vapor	4	144 ± 5	162 ± 0.6	180 ± 3
			233 ± 12	281 ± 7	315 ± 7
			330	349	378
			390	407	416
zinc fines	liquid	1	328	339	382
	vapor	1	324	330	385
plutonium fines	liquid	1	263	no data	no data
	vapor	1	268 ± 5	304 ± 7	368 ± 16

^aThe ± values given for exotherm data represent one standard deviation for a series of replicate analyses. The data given without ± variations represent the mean temperatures of duplicate analyses.

Application of this method to the determination of F^- in plutonium (IV) fluoride (PuF_4) precipitated from nitric acid-hydrochloric acid solutions gave a mean result of 20.9 percent F^- . This corresponds well to the theoretical F^- content of $2PuF_4 \cdot 5H_2O$, which is reported⁶ to be the precipitation product from dilute aqueous solutions. Production samples, determined by this method to have less than theoretical F^- contents, were found to include enough impurities, such as oxide, nitrate, and sulfate, to approximate a material balance.

IMPROVED TURBIDIMETRIC SULFATE METHOD

Robert D. Koelmel and Jack L. Long

Summary

The reproducibility of the standard turbidimetric method for sulfate ion (SO_4^{2-}) was improved and the analysis time decreased through the addition of tetrahydroquinone (THQ) and elimination of a conditioning step. The relative standard deviation of the modified procedure was better than 6 percent at the concentration levels tested, and recoveries of standard additions ranged from 84 to 116 percent.

Experimental

The standard method for the turbidimetric determination of SO_4^{2-} in water⁷ has a time-consuming conditioning period that was eliminated by the addition of THQ. This did not affect the linearity of the Beer calibration plot over the SO_4^{2-} concentration range of from 1 to 40 milligram per litre (mg/l) for which this procedure was designed.

After addition of THQ, 6 replicate analyses were performed on stock SO_4^{2-} solutions to determine

⁶J. K. Dawson *et al.* "The Preparation and Some Properties of Plutonium Fluorides." Page 554. *Journal of the Chemical Society (London, England)*. 1954.

⁷Michael J. Taras, Arnold E. Greenberg, R. D. Hoak, and M. C. Rand, editors. *Standard Methods for the Examination of Water and Wastewater*. Thirteenth Edition. American Public Health Association, New York, N. Y. 1975. page 496.

the reproducibility of the method. The relative standard deviations at 3, 8, 20, and 40 mg/l SO_4^{2-} concentrations were 3.8, 5.5, 4.9, and 3.2 percent, respectively. Recoveries of standard additions of SO_4^{2-} solutions to well waters ranged from 84 to 116 percent.

In this procedure, a known aliquot of sample is taken and diluted to 25 millilitres (ml) so that the resulting stock solution contains SO_4^{2-} from 3 to 40 mg/l. Two ml of aqueous stock solution, containing 400 grams per litre (g/l) barium chloride and 10 g/l THQ, are added, and the resultant solution mixed for 60 seconds. The absorbances of this solution and the sample solution to which no THQ solution has been added (*blank*) are measured at 450 nanometres. The absorbance of the *blank* solution is subtracted from that of the THQ-treated solution to correct for turbidity in the water. The SO_4^{2-} concentration in the sample can then be obtained directly from a Beer calibration plot.

PALLADIUM, SILVER, AND COPPER IN PALCUSIL

Amelia M. Miller

Summary

The chemical analysis of the brazing alloy Palcusil[®] involves a series of gravimetric separations of its three principal metallic components, silver (Ag), copper (Cu), and palladium (Pd). Details of the separations and gravimetric procedures are given.

Experimental

Palcusil is a brazing alloy composed of 65 percent (%) Ag, 20% Cu, and 15% Pd. The alloy is dissolved in nitric acid, and Ag is precipitated with hydrochloric acid as silver chloride ($AgCl$). The $AgCl$ is filtered, washed, and dissolved in concentrated ammonium hydroxide, then reprecipitated, filtered dried, and weighed. The filtrates are combined and evaporated to remove excess acid. The Pd is reduced to the metal by boiling in an aqueous solution of

formic acid and sodium formate and filtered. The Cu is electroplated from the filtrate, dried, and weighed. Any residual Pd is precipitated by addition of dimethyl glyoxime. The combined precipitates of Pd are ignited at 800 °C to palladium oxide (PdO). If heated to a higher temperature, PdO decomposes with the loss of oxygen. The relative precision for each element by this method is ± 0.3 percent. A material balance of 99.8 percent for the alloy has been obtained.

TRITIUM SAMPLING LABORATORY

Duane I. Hunter

Summary

An auxiliary laboratory has been set up for the sampling of shipping containers for tritium and other contaminant gases prior to the opening of the container. Liquid scintillation counting is used for tritium determinations while general gas analyses are performed by mass spectrometry or gas chromatography or both. This facility has been established as a screening device to prevent inadvertent release of toxic or noxious materials to the environment.

Discussion

The new laboratory area results in the consolidation of several sampling methods and analysis techniques

used to screen incoming shipments and parts for possible tritium contamination. Further gas analyses may be used to determine complete atmospheric compositions prior to opening or unpacking operations. The laboratory is equipped to sample drums of up to 80-gallon capacity, pressure cookers (P12081 and P14103 containers), produce cans of all sizes, sealed-metal tubing (stainless steel or copper), and other miscellaneous containers. Sampling methods are used which isolate the gas in a sealed system until the analysis is complete; consequently, no release is made to the box or room atmospheres.

Liquid scintillation counting for tritium analysis permits a lower detection limit of 2 microcuries per cubic metre in the gas phase at standard temperature and pressure. This method requires less than 7 standard cubic centimetres of gas for analysis and a total sampling and analysis time of less than 15 minutes. Gas samples placed in a separate sample vial can be transferred to the Gas Mass Spectroscopy or Gas Chromatography Laboratories for complete analysis. Head space and original pressure in the container can be measured during the sampling operations. The survey gas analysis can detect the presence of flammable or noxious gases such as hydrogen or oxides of nitrogen. This complete set of analytical data gives the customer information needed to safely open or unpack a shipment and prevent releases of toxic or noxious substances to the environment.

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