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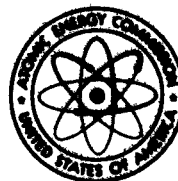
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The previous reports in this series are: MLM-1152
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TABLE OF CONTENTS

	Page
<u>Summary</u>	3
<u>Adhesives Research</u>	
Conductive Adhesives	5
<u>Radioelements Research</u>	
Residue Adsorption	6
Half-life of Tritium	9
Polonium-208-Polonium-209	9
Polonium Process Waste Disposal	10
Actinium-227 Separation	11
Uranium-234 Separation	11
<u>Alpha and Neutron Source Development</u>	
Neutron Measurements	13
Polonium Alpha Sources	14
<u>Analytical</u>	
Calorimetry	15

SUMMARY

Adhesives Research

Conductive Adhesives Three silicone-based conductive adhesives were formulated and evaluated. One of the formulations had a resistivity of 0.089 ohm-centimeter after aging one week. After the same period the other two formulations had resistivities of 0.871 and 38.0 ohm-centimeters.

Radioelements Research

Residue Adsorption Erratic adsorptive behavior of barium-140 has been traced to an impurity in the barium-lanthanum-140 stock solution. Repurification by cation exchange did not improve the reproducibility of the separations, possibly because impurities are being reintroduced by the distilled water. Definite improvement was noted when the barium-140 was separated from lanthanum-140 and other hydrolyzable materials by residue adsorption methods.

Half-life of Tritium A calorimetric measurement of a salt sample over a period of 148 days gave a tritium half-life of 12.361 ± 0.028 years. This value confirmed the earlier value of 12.355 ± 0.028 years.

Polonium-208-Polonium-209 The polonium-208-polonium-209 mixture returned from Argonne National Laboratory is being processed further. Two batches of polonium-contaminated organic material were wet-ashed in nitric and perchloric acids. Further purification will be by techniques similar to those presently used for separating polonium-210 from bismuth. An unidentified gamma activity was found in the polonium which had been recovered from pieces of Monel fluorination apparatus.

Polonium Process Waste Disposal The optimum conditions for decontamination of polonium process waste are being determined. Ferric sulfate was dissolved in the solution, and the pH was adjusted to a value between 7.0 and 12.0 to precipitate ferric hydroxide. However, the alpha and beta activity was not decreased sufficiently to allow dumping of the wastes without further processing.

Actinium-227 Separation A method of separating actinium-227 from a mixture of its daughter products and radium-226 is being developed using ion exchange and solvent extraction techniques. Substituted elements are being used to evaluate the separation procedures prior to the separation of the radioactive isotopes. More than 99 per cent of the thorium in the separation mixture was extracted into a chloroform solution of thenoyltrifluoroacetone at pH 2.

Uranium-234 Separation The separation of uranium-234 from aged plutonium-238 was demonstrated using ion exchange and solvent extraction techniques. One batch of aged plutonium-238 solution was processed, and 14 milligrams of uranium-234 were recovered. A second batch containing an estimated 250 milligrams of uranium-234 in 26.2 grams of plutonium-238 is being processed. More than 99 per cent of the plutonium was removed from the uranium fraction using an ion exchange technique, followed by a hexone extraction.

Alpha and Neutron Source Development

Neutron Measurements Calibrations of neutron sources with the precision long counter are based on counting the unknown and the standard with their geometrical centers at the same position. The percentage by which the detector counting rate for a PuBe source of finite geometry exceeds the counting rate for a point-source was determined for six sources. As a result of the investigation of neutron source calibration, the relative responses of the precision long counter to PuBe secondary standard sources are being restudied. Twenty-two recanned PuBe sources were recalibrated.

Polonium Alpha Sources A new electroplating control unit is being used for plating the gold retention layer on a series of experimental polonium alpha sources. Fabrication of wipe-free, goldplated polonium sources with a specific activity of more than five millicuries per square centimeter was unsuccessful.

Analytical

Calorimetry A quasi-calorimeter is being used to investigate stretching effects of calorimeters. Stretching, or tailing on the calorimeter cooling curve, is affected by the insulation used. To lower the stretching effect in micro-calorimeters an insulation consisting of an air gap, or gaps, separated by foils will be used.

ADHESIVES RESEARCH

Adhesives are being developed for use with explosives and for other specialized applications. The adhesives must have satisfactory working and curing characteristics; they must retain flexibility at low temperatures and adequate tensile strength at elevated temperatures.

Conductive Adhesives

Three silicone-based conductive adhesives were formulated and evaluated as shown in Table 1. These data show that silicones may be used as a base for conductive adhesives. The best result was obtained with formulation J2-102, which contains carbon black (Witco Chemical Company) and J-2 Silver Powder (Engelhard Industries) in RTV 102 silicone adhesive (General Electric Company) matrix. The specific resistivity of this material remained constant at about 0.09 ohm-centimeter. A similar formulation (G3-102 which contained G3 Silver Powder (Engelhard Industries) in place of the J-2 powder had a higher original specific resistivity which increased on aging one week. Formulation G3-602 contains carbon black and G3 Silver Powder in a matrix consisting of LVT602 silicone (General Electric Company) cured by a proprietary basic catalyst (SRC-05). This material had a specific resistance of almost 100 ohm-centimeters 16 hours after mixing, which decreased to 38 ohm-centimeters after one week.

Table 1

FORMULATION AND SPECIFIC RESISTIVITY OF SILICONE BASE ADHESIVES

	J2-102 ^a	G3-102 ^a	G3-602 ^a
Continex CF Black (wt %) (Witco Chemical Co.)	7.5	8.2	8.2
J-2 Silver (wt %) (Engelhard Industries)	68.5	-	-
G-3 Silver (wt %) (Engelhard Industries)	-	66.2	66.2
Silicone (wt %)	24.0 ^b	25.6	25.6 ^c
<u>Specific Resistivity^d</u>			
After 16 hr. (ohm-cm)	0.089	0.221	98.7
After 1 wk. (ohm-cm)	0.089	0.871	38.0

^aMound Laboratory designation.

^bRTV 102 Silicone Adhesive (General Electric Co.)

^cConsists of 22.8 per cent silicone LTV 602 and 2.8 per cent SRC-05 catalyst.
(General Electric Co.)

^dSamples allowed to stand at room temperature.

RADIOELEMENTS RESEARCH

Basic and applied research on a number of radioelements is being conducted to determine physical properties, develop analytical techniques, and study the basic radiochemistry involved. Of particular interest are alpha emitters, their decay chains, their isotopes, and their chemical homologs.

Residue Adsorption

Erratic results obtained in previous work on the desorption of alkaline earth elements from hydroxide residues were attributed to impurities in the diluted ammonium hydroxide.¹ In a continuing effort to isolate and, if possible, identify the presumed impurities, tests have been carried out with barium-lanthanum-140 as the radioactive source material.

The separation procedure has been described in detail.² In general, it consists of depositing on a flat surface, previously cleaned with alcoholic potassium hydroxide and dilute nitric acid, a sample of the carrier-free radioactive material sufficient to cover an area defined by a methyl methacrylate ring (15 mm in diameter). The sample is dried at 90°C, covered with distilled water, dried again, and covered with dilute ammonium hydroxide. When the solution has evaporated to dryness, the residue is leached with distilled water, and the gamma spectrum of the residual activity is compared with the original spectrum or with that of a standard sample.

An example of the separation of barium-140 and lanthanum-140 from a platinum surface is illustrated in Figures 1 and 2. In Figure 1, the original (upper) spectrum is compared with the residual (lower) spectrum. In Figure 2 the original spectrum is compared with the spectrum of the desorbed radioactivity, which is nearly pure barium-140. The degree of separation is shown by the relative peak heights at 0.162 Mev (due to barium-140) and beyond 0.7 Mev (due to lanthanum-140). In the particular example shown, the residual radioactivity included 15.7 per cent of the original barium-140 and 94.4 per cent of the original lanthanum-140, as estimated from the areas under the peaks at 0.162 Mev and 1.60 Mev (not shown). These results are comparable to those previously obtained with strontium-90 and yttrium-90.

The results have not been consistent, as seen in Table 2, where the adsorptive behavior of barium and lanthanum-140 on various surfaces is compared.

A new source of barium-lanthanum-140 was obtained from Oak Ridge National Laboratory. The solution, which was 2.35 normal in hydrochloric acid, was evaporated to dryness in a glass vial. There was a heavy residue, which was expected from the fact that the analysis supplied with the shipment indicated the presence of 25 milligrams of nonvolatile solids. The residue was leached with distilled water, and the solution was evaporated to dryness. An estimated 90 per cent of the solids was transferred with the distilled water, with approximately 50 per cent of the gamma radioactivity. The spectra showed no significant separation of barium-140 from lanthanum-140.

The glass vial in which the original evaporation had taken place, and from which 90 per cent of the solid residue had been removed by leaching with distilled water, was leached with one normal nitric acid. The nitric acid solution removed approximately two-thirds of the total gamma radioactivity. A sample of this solution, deposited on platinum and treated by the standard residue adsorption procedure, showed 16.3 per cent barium-140 retention and 98.8 per cent lanthanum-140 retention.

The radioactivity remaining in the original vial was combined with that leached by distilled water and loaded on a small column of Dowex-50 cation exchange resin. The column was washed several times with one normal nitric acid, and the barium-140 was eluted with three normal nitric acid. The eluate was diluted to a suitable volume with one normal nitric acid and used as a source of barium-140.

¹MLM-1157.

²H. W. Kirby, *J. Inorg. Nucl. Chem.*, 25, 483-99 (1963).

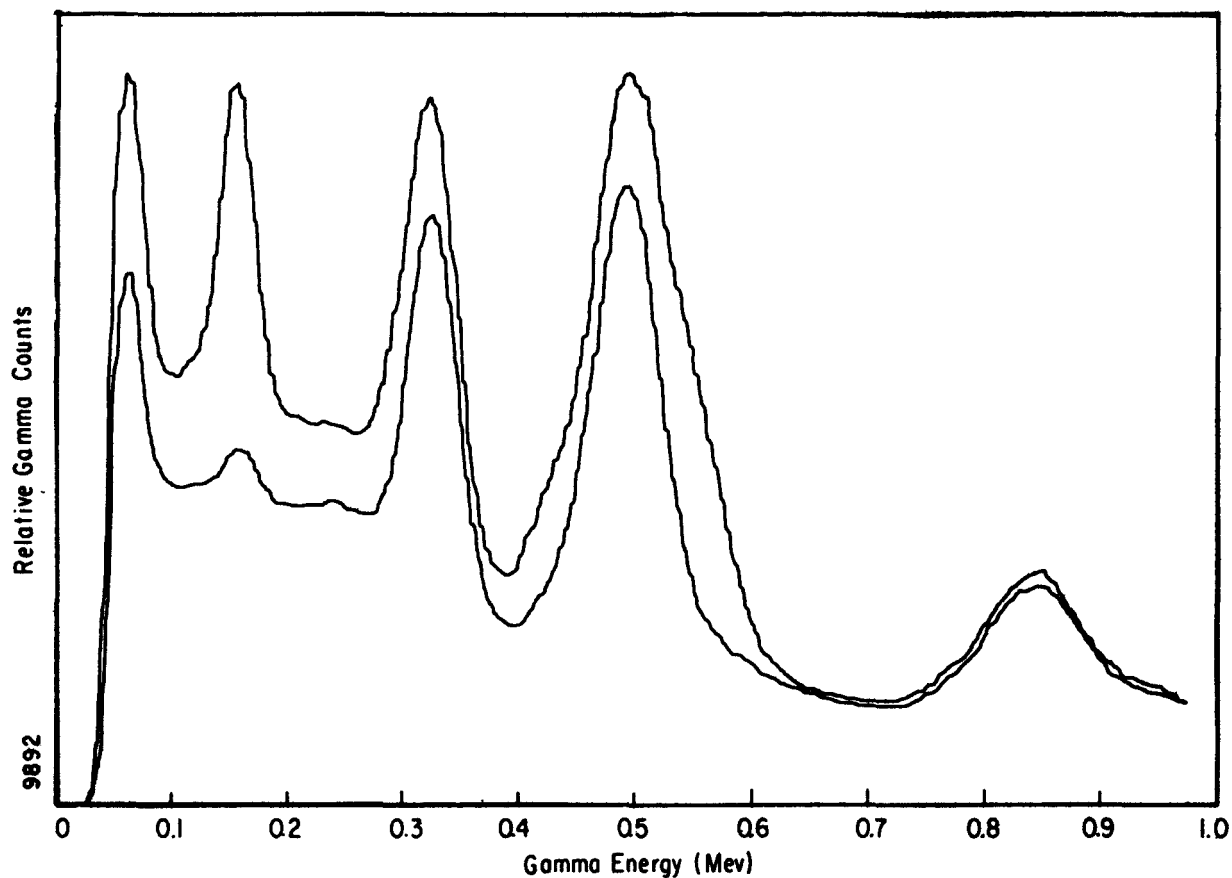


Figure 1. Residue Adsorption of Barium-Lanthanum-140 (Retained Fraction).

Table 2

RETENTION OF BARIUM-140 AND LANTHANUM-140 HYDROXIDES BY VARIOUS SURFACES

Sample Number	Ba Retained (%)	La Retained (%)	Surface
356 A	14.8	93.2	Platinum-clad stainless steel
361 B	19.9	93.8	Platinum-clad stainless steel
361-26	29.8	94.1	Platinum
361 A	14.8	79.7	Gold-plated stainless steel
361-2	18.2	73.2	Gold
362-4	32.4	93.5	Gold
356 AA	31.3	98.2	Type 430 stainless steel
356 AP	27.5	96.3	Type 304 stainless steel
357 A	29.8	90.9	Pyrex
359 C	21.2	82.8	Pyrex (Desiccote ring)
360 A	26.7	88.9	Lime glass vial

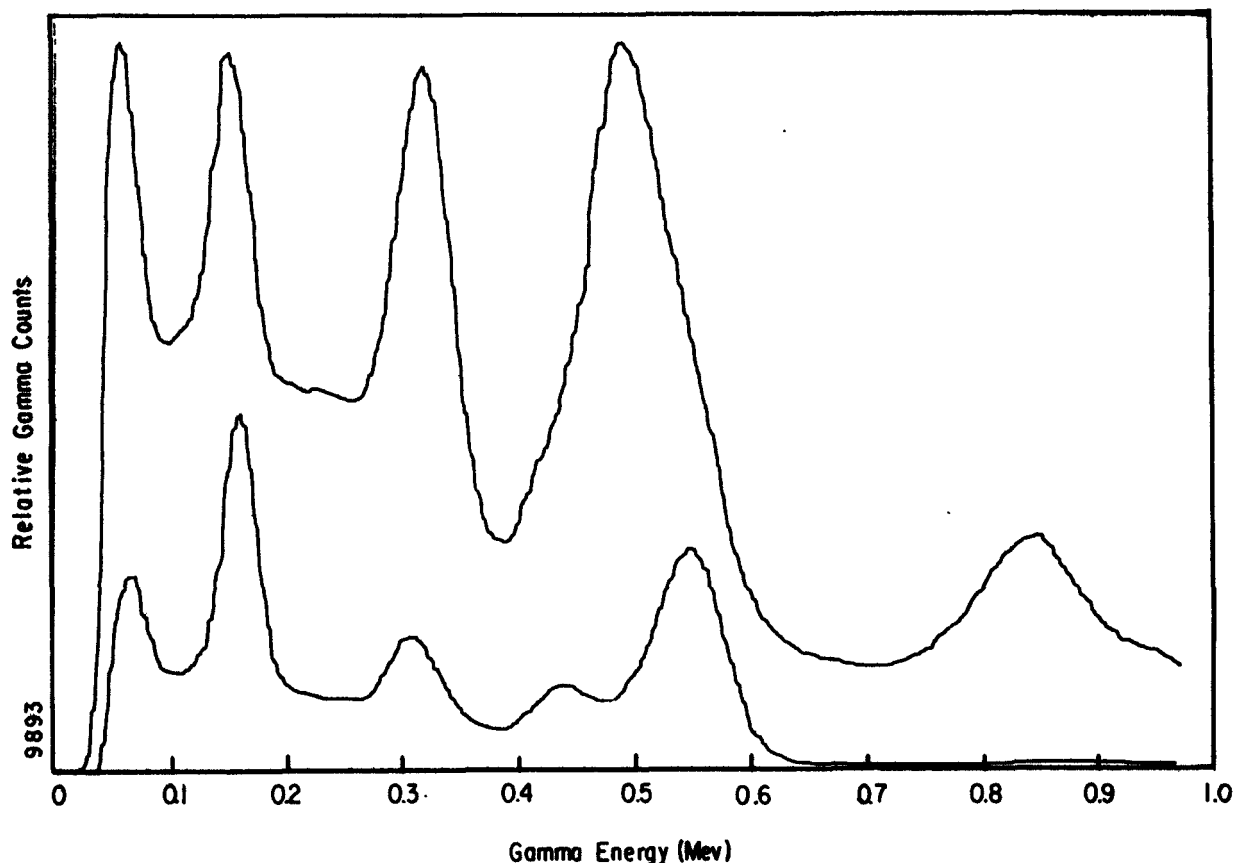


Figure 2 Residue Adsorption of Barium-Lanthanum-140 (Desorbed Fraction).

Results obtained with the ion exchange purified material were as erratic as those reported in Table 2. Barium-140 retention ranged from approximately 12 per cent to as high as 54 per cent.

To determine whether the erratic behavior was due to the reagents or to some intrinsic characteristic of barium-140, a sample of barium-lanthanum-140 was deposited on a platinum-clad stainless steel disk and treated in the usual manner. The desorbed fraction was transferred to another disk, dried, and covered with one normal nitric acid. This solution was dried and treated as a newly deposited sample of barium-140. The same procedure was repeated until the barium-140 had been desorbed from four successive platinum surfaces. Of the barium-140 originally present on each surface, the following percentages were retained: 30.9; 10.4; 9.4; 8.3 per cent. Thus, it would appear that barium-140, once it is freed from whatever inhibiting impurity is present in the original stock solution, behaves as strontium-90.

One possible source of the unknown contaminant is the distilled water now being piped to the laboratories. Conductivity measurements show that the water contains ionizable material equivalent to approximately 2.5 parts per million of sodium chloride, more than twice as high as the impurities present during the period when the strontium-90 residue adsorption work was being carried out. Any repurification of the barium-lanthanum-140 stock solution would prove futile if the purified material were subsequently diluted with distilled water or reagents containing adsorbable solids. The total nonvolatile solids content of the distilled water is being determined, and the residue will be analyzed spectrographically. As further insurance against the introduction of contaminants by the reagents, future work will be done with reagents, prepared from distilled water which has been further purified by passage through a resin demineralizer.

Half-life of Tritium

Further half-life data have confirmed the previously reported half-life for tritium as 12.355 ± 0.01 years. A salt sample was assayed 45 times over a period of 148 days in Calorimeter 90, which has an automatic loading device and a constant current controller for the heater current. The value determined was 12.361 ± 0.028 years.

Polonium-208-Polonium-209

The polonium-208-polonium-209 mixture returned from Argonne National Laboratory is being purified. All of the polonium, which was mixed with filter paper and cardboard cartons (used to contain the sample) has been successfully wet-ashed. The wet-ashing was carried out by boiling the contaminated organic material with an equivolume mixture of concentrated nitric and perchloric acids. A few milligrams of potassium dichromate were added to catalyze the reaction and to serve as an indicator.

As the mixture boiled, nitric acid oxidized the easily oxidizable material and gradually distilled out of the mixture, leaving dilute perchloric acid. As heating continued, the perchloric acid concentration increased to about 72 per cent, a constant boiling (203°C) azeotrope, and the more difficultly oxidized materials were removed. When the reaction was complete, the chromium indicator changed from green (Cr^{+3}) to orange (Cr^{+7}).

The mixture was heated in a distillation flask, and condensed distillate was continuously monitored for polonium alpha activity. No polonium activity was detected in the distillate at any time.

Two batches of polonium-contaminated organic material were wet-ashed. The first batch was free of inorganic salts, but the second lot contained large quantities of copper and nickel salts. It was assumed that the first lot of polonium residues resulted from washing the Monel fluorination apparatus with a dilute, non-oxidizing acid; the second batch was produced when the equipment was rinsed with a stronger acid, which also attacked the Monel. Small aliquots of both batches, after wet-ashing, have been treated further, to determine what additional radiochemistry is required for purification of the polonium.

The residue, after wet-ashing of the first lot of material, consisted of a clear, light green, perchloric acid solution and a dark, red-brown precipitate. All of the polonium was contained in the precipitate and could not be removed by washing with water. On treatment with dilute (3M) hydrochloric acid, however, the red-brown precipitate was decolorized, giving an orange-yellow solution containing all the polonium and an unidentified white precipitate. The appearance and chemical properties of the red-brown precipitate resemble those of a polonium chromate reported by Bagnall.³ Further characterization of this compound will be explored when the polonium has been completely purified. It is expected that the chemical operations on this batch of material will be performed carrier-free, following the procedure used to separate polonium-210 from irradiated bismuth.

The wet-ash residue of the second batch of polonium-contaminated organic matter was a clear, light blue-green perchloric acid solution and a large quantity of very dark green solid. Once again, the polonium was with the solid material and could not be removed with water. The precipitate was treated with three molar hydrochloric acid solution, giving a dark gray precipitate and a clear, yellow-orange solution, which contained all the polonium. The solution containing the polonium will be combined with that of the first batch for further processing.

³K. W. Bagnall et al., *The Polonium Chemistry Project*, AERE C/R 2566, 1958.

In addition to the organic matter several pieces of the Monel fluorination apparatus itself were returned from Argonne National Laboratory. One four-inch length of Monel pipe which contained approximately 100 microcuries of polonium-208-polonium-209 was selected for trial separations. The Monel was effectively decontaminated (95% recovery) with several rinsings of dilute (3 M) hydrochloric acid. Some surface oxidation of the Monel had occurred prior to its treatment at Mound Laboratory. Thus nickel and copper, the major constituents of Monel, were also dissolved in the dilute acid, giving a clear, dark green solution. The polonium was completely removed from the solution by spontaneous electrodeposition on a bed of powdered bismuth metal. Further purification of the polonium will probably follow the chemistry used in the polonium process at Mound Laboratory.

In the spontaneous electrodeposition of the polonium recovered from the Monel a small amount of radioactivity (about 2.5% of the polonium activity) appeared in the effluent. It was first thought that this activity was the polonium-208-polonium-209 mixture, but on closer examination, the gamma rays associated with the radioactive decay in the solution were found to have energies of approximately 100 and 300 kev. (The main gamma rays of the polonium-208-polonium-209 mixture occur at 260 and 75 kev.) Further work is in progress to identify this radioactivity which has not been observed in the polonium separated from the organic material.

Polonium Process Waste Disposal

An investigation was initiated to determine the optimum conditions for decontamination of polonium process waste after the first precipitation of bismuth hydroxide at pH 12-14. The batch of high-level wastes used for the work was designated HH27; bismuth had been removed from this batch by precipitation of bismuth hydroxide at pH 13.8. Samples containing 100 milliliters were treated as follows: The pH was adjusted to 4.0 with sulfuric acid. Solid ferric sulfate, equivalent to 25 pounds per 300 gallons of waste solution, was dissolved in the solution. The pH was adjusted to a range of values between 7.0 and 12.0, precipitating ferric hydroxide. The mixtures were stirred for 30 minutes and then centrifuged. Aliquots of the supernatant solution were counted for alpha, beta, and gamma radioactivity. The results are given in Table 3, along with the decontamination factors calculated from the counting data.

As in previous work, correlations between the various counting methods are not satisfactory. For example, the decontamination factor from gamma counting data increases from pH 7 to pH 11; the increase is due mainly to the more effective removal of silver-110m with increasing pH. The general trend of the beta decontamination factors is in the opposite direction, and the alpha decontamination is clearly decreasing. This trend is due to the nature of the radioactive species present in the polonium process wastes, and to the fact that some solid material acting as an absorber is always present in evaporated counting samples. This solid material causes erroneous results in alpha and beta counting. Several radioactive isotopes common in polonium process wastes decay chiefly by gamma-ray emission, with low-energy (thus, uncountable) beta particles. Zinc-65 and silver-110m are examples of such nuclides.

In none of the experiments was the alpha or beta activity decreased sufficiently to allow dumping without further dilution and treatment. The poor decontamination was attributed to the fact that the amount of bismuth available for precipitation of bismuth hydroxide was much lower than usual in HH27; most of the spent liquor arose from polonium reprocessing operations. Ordinarily, reprocessing solutions do not contain bismuth. Thus, the first precipitation was not as effective in decontaminating the wastes. Further studies are planned on more representative polonium process wastes.

Table 3

**DECONTAMINATION OF POLONIUM PROCESS WASTES BY
PRECIPITATION OF FERRIC HYDROXIDE AT VARIOUS pH VALUES**

Sample	pH	cpm/ml			Decontamination Factor		
		Alpha	Beta	Gamma	Alpha	Beta	Gamma
HH27F1	-	51,000	105,000	26,682	-	-	-
HH27F1a	7.0	13,100	6,200	818.3	3.89	17.5	32.6
HH27F1b	8.0	17,900	16,600	632.3	2.85	6.3	42.2
HH27F1c	9.0	25,300	8,000	634.1	2.02	13.1	42.1
HH27F1d	10.0	28,000	9,100	452.6	1.82	11.5	59.0
HH27F1e	11.0	32,900	8,000	191.6	1.55	13.1	139.2
HH27F1f	12.0	33,900	12,200	370.2	1.50	8.6	72.1

Actinium-227 Separation

Thenoyltrifluoroacetone (TTA) was used as a chelating agent with carbon tetrachloride and chloroform as solvents in the extraction and purification of actinium-227. The naturally occurring elements listed below were used as substitutes for highly radioactive actinium-227 and its daughter products. Standard solutions of one gram of the substitute element per liter of two molar nitric acid were prepared and used in the extraction studies. Approximately one gram per liter concentration is expected for each radioactive element in solution.

Isotope	Substitute Elements
Actinium-227	Lanthanum
Thorium-227	Thorium-230
Radium-223, -226	Barium
Lead-207, -211	Lead
Bismuth	Bismuth

Carbon tetrachloride was unsatisfactory as the solvent for TTA due to interface problems in the lanthanum extraction. The use of chloroform as solvent eliminated interface problems under the extraction conditions. Greater than 99 per cent of the thorium is extracted at pH 2 or above. Lanthanum begins to extract at pH 2.5 and is 98 per cent extracted at pH 6. The literature indicated that actinium extracts at a higher pH than lanthanum.

Uranium-234 Separation

A program to separate and purify milligram quantities of uranium-234 from aged plutonium-238 solutions is in progress at Mound Laboratory. The uranium is separated from the bulk of the plutonium in nitrate solution by an anion exchange process. Extraction of the uranium into hexone (methyl isobutyl ketone) further reduces the concentration of plutonium and most other impurities. Finally, thenoyltrifluoroacetone (TTA) extraction removes the last traces of plutonium from the uranium. One batch of feed solution (B63) has been processed according to this scheme, and 14 milligrams of uranium-234 were recovered. A second batch (5H) has been processed through the hexone extraction step.

Solution 5HB was prepared for hexone extraction by adding an equal volume of two molar aluminum nitrate solution. The plutonium concentration of the resulting feed solution was 0.225 milligrams per milliliter, and the acidity was 1.8 normal. A small amount of ferrous sulfamate solution was added to Feed Solution 5HB, producing the blue color characteristic of trivalent plutonium.

A few drops of hydroxylamine nitrate solution was added to each 70-milliliter batch of Feed Solution 5HB before the solution was combined with an equal volume of hexone. The loaded organic phase was washed with 20 milliliters of a solution one molar in aluminum nitrate and one normal in nitric acid; the solution also contained a few drops of hydroxylamine nitrate. Scrub and feed solutions were combined and contacted an additional three times with 70-milliliter portions of hexone in a batch countercurrent manner. A sample of the loaded organic from processing on the same day the ferrous sulfamate was added was determined to contain one per cent of the plutonium concentration of the feed solution. A similar sample, taken from processing on a subsequent day, contained over 70 per cent of the original plutonium concentration. This behavior was attributed to the failure of the hydroxylamine to reduce the plutonium to the trivalent state. This hypothesis is substantiated by the lack of blue color of trivalent plutonium upon the addition of the hydroxylamine reducing agent.

Hundred-milliliter batches of loaded organic were stripped by contacting three times with 50 milliliter portions of 0.35 normal nitric acid in a batch countercurrent manner. The resulting aqueous solution was designated 5HC and is being evaporated to a smaller volume in preparation for the TTA extraction step.

ALPHA AND NEUTRON SOURCE DEVELOPMENT

Mound Laboratory is responsible for producing alpha and neutron sources, manufactured from polonium-210 and plutonium-239, which cannot be produced by American industry at the present time. The techniques of fabricating these unusual sources are being developed and publicized.

Neutron Measurements

Calibrations of neutron sources with the precision long counter have been based on counting the unknown and the standard with their geometrical centers at the same position. Neutrons produced in the far side of the source with respect to the counter, however, are attenuated more by the source material than those produced in the near side. The effective center of neutron emission is closer to the counter than the geometrical center, the amount varying with the physical size of the source. A comparison between a standard and an unknown of different physical size will then be in error.

The approximate errors introduced into the calibration of neutron sources by assuming point-source geometries have been established for several PuBe sources of different sizes.^{4,5} The percentage by which the detector counting rate for a PuBe source of finite geometry exceeds the counting rate for a point-source is given below (source to counter distance is 100 cm):

Source Size			Percentage by which counting rate for a finite source exceeds that for a point source
Diameter (cm)	Height (cm)	Mass of Pu (g)	
2.5	2.5	13	0.15
2.6	3.3	16	0.17
2.6	3.6	16	0.16
2.7	11.4	80	0.20
3.3	6.9	80	0.32
3.9	10.7	160	0.46

The net calibration error would be the difference between two values in the last column — one value for the source being calibrated and one for the secondary standard source. Sources are usually compared with the standard which most closely resembles it in size and shape. The maximum error in a calibration by assuming a point-source geometry would be for the 160-gram source (shown above) when it is compared with the 80-gram standard (3.3 cm x 6.9 cm). Including possible inaccuracies in the above analysis, this error is estimated at 0.2 per cent or less. When comparing two sources which are more closely related in size, the error due to assuming point-source geometry will be 0.1 per cent or less.

As a result of the investigation above, the relative precision long counter responses to the two PuBe secondary standard sources, M-591 (80 g of Pu, 3.3 cm diameter x 6.9 cm high) and M-977 (13 g Pu, 2.5 cm diameter x 2.5 cm high) are being restudied. The response is dependent on the neutron energy spectra of the two sources as well as the effective source-counter distance. Based on published spectra reports a one per cent difference in the counter response to the two sources would be expected. A counting rate difference of about 0.5 per cent has been measured, assuming a point-source geometry. When corrections are applied to account for the differences in the finite geometries of the two sources, the difference becomes about one-third of one per cent. This is significantly different from the one per cent predicted from

⁴ MLM-1155.

⁵ MLM-1160.

spectra measurements. One explanation is that the spectra of PuBe sources of different sizes may not be as different as previously reported. The neutron spectra of sources being calibrated will have to be studied further in order to improve the accuracy of neutron calibrations with the long counter.

Twelve recanned PuBe neutron sources were returned to their users. Ten additional recanned sources have been recalibrated and await shipment

Polonium Alpha Sources

A new electroplating control unit is being used for plating the gold retention layer for a series of experimental polonium alpha sources.

Fabrication of wipe-free, goldplated polonium sources with a specific activity of more than the five millicuries per square centimeter was not successful. Preliminary tests of the use of 0.0003-inch thick gold foil in direct contact with the activity were not successful.

ANALYTICAL

Methods of analyzing elements and compounds are being developed to support other programs at Mound Laboratory. These methods include instrumental techniques and classical wet methods.

Calorimetry

A quasi-calorimeter is being used to investigate stretching effects of calorimeters.⁶ The stretching effect is a tailing on the logarithmically plotted cooling curve of a calorimeter. Santocel, a commercial, high-temperature insulator used around the thermocouples, caused the highest stretching effect of any of the materials studied.

Perlite and Styrofoam had considerably less stretching effect, but the effect was still greater than desired in a high-precision calorimeter. End baffles for the calorimeters probably should be constructed of multiple layers of foil spaced about $\frac{1}{4}$ inch apart and supported by a framework of plastic. The stretching effect of such a baffle will be measured. Since a stretching effect has been measured in Lucite, Kel-F will probably be used in the baffle. The thermal ends of Calorimeter-90 were made of Kel-F; this calorimeter has a very small stretching effect, with a low magnitude of slow variations.⁷ Slow variations are changes in calorimetric readings over a period of time; the cause is undetermined. However, the two effects seem to be related.

It will be relatively easy to reduce the stretching effect in macro-calorimeters by proper choice of construction materials without loss of other features. For micro-calorimeters the situation is more difficult. Good insulating materials are needed in the gradient medium to increase the thermal resistance of radiation and convection. Santocel was used in Micro-Calorimeters 60 and 67, and both have large stretching effects and slow variations. The slow variations are about ten times the short time stability of a calorimeter. Thus, the precision of a micro-calorimeter may be improved by using a much poorer insulator than Santocel as long as the insulator has a low stretching effect. An air gap of less than one centimeter, or several air gaps separated by foils, is probably the best insulation for micro-calorimeters at present.

⁶ MLM-1146.

⁷ MLM-1138.