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**RADIATION CHEMISTRY OF MATERIALS
USED IN PLUTONIUM PROCESSING**

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RADIATION CHEMISTRY OF MATERIALS USED IN PLUTONIUM PROCESSING

Armen R. Kazanjian and Alan K. Brown

Abstract: There are many materials used in plutonium fabrication which are highly susceptible to radiolytic decomposition. This report summarizes the radiation chemistry of those materials used at Rocky Flats. The radiolysis products are, in general, reactive toward plutonium and could lead to corrosion. However, the extent of radiation damage depends on the exposure conditions.

INTRODUCTION

Purpose – Application to Rocky Flats

The purpose of this report is to evaluate the materials used in plutonium production and fabrication glovebox lines from the standpoint of radiation stability. There are many materials, such as oils, solvents, and plastics, in production areas, which come into contact with plutonium. These substances are therefore subjected to the alpha particle emanations from plutonium and are altered chemically and physically. The extent of the changes will depend on the substance and its exposure (amount and conditions) to the alpha particles.

The radiation damage to these materials could be detrimental to production in two ways. First, some radiolytically generated compounds could be corrosive toward plutonium and thereby affect the product integrity. Second, the irradiation could be prolonged to the point where the function of the material is impaired; for example, an oil may lose its lubricating properties or a plastic holder may lose its structural strength. The first effect is more strongly emphasized in this report because much less irradiation is required to bring about corrosive changes.

The Introduction presents background material as a basis for understanding the next section (list of materials) which is a list of materials that may come into contact with plutonium. This list is current and additions will be made as newer materials are added to the fabrication lines. The uses and potential radiation exposure of each material will first be described and then the known information about its radiation chemistry will be presented. The radiation chemistry of some of these

materials has been previously investigated by others and these data are summarized. Information on some materials has also been obtained from experimental work at Rocky Flats. This work is continuing and we will supplement the present results as additional data are obtained. The last section of each of these radiation studies will describe the possible effects of the radiolysis products on the integrity of the plutonium.

It should be stressed that this report is concerned only with the potential corrosion that could be caused by radiation. Under normal production conditions, radiation effects may be negligible and all corrosion may be due to other causes, e.g., the direct reaction between a contaminant and plutonium.

Radiation Chemistry Background

This section acquaints the reader with some general effects of radiation and a few definitions of terms commonly used in this field. Radiation chemistry or radiolysis is the study of the chemical effects produced in a system by the absorption of ionizing radiation. The types of ionizing radiation are x-rays, γ -rays, electrons, protons, neutrons, α -particles, recoil particles, and other high-velocity charged particles. In this report, γ -rays and α -particles are of most concern.

Almost any material exposed to high-energy radiation will be altered. These changes are due to the energy imparted to matter along the tracks of fast, charged particles. The mode of energy transfer depends on the type of radiation. Gamma rays give up their energy to energetic secondary electrons which lose their energy almost entirely by electrostatic interaction with the electrons of the stopping medium. Alpha particles lose their energy in essentially the same way; however, the track, or rate of energy loss, is very different for the electron and the alpha particle because of the large difference in mass. The linear rate of energy loss is called the linear-energy-transfer (LET), or the stopping power, and is expressed in units of kilo electron-volts per micron, or in units of electron volts per angstrom.

The energy transferred to the stopping material will produce ions and excited atoms or molecules. These primary species may, in turn, decompose or react with

the substrate to effect a chemical change. It is the function of the radiation chemist to determine the nature of these chemical changes. The changes in physical properties are also important; e.g., the viscosity change in an oil or the change in lattice constants of a crystalline solid. At this stage of development in this new field, it is very difficult to accurately predict the effects of radiation on any particular substance. One can only gain information (prior to experimental work) by making comparisons to very similar materials.

The absorbed dose, which is the amount of energy absorbed per unit mass of irradiated material, can be expressed in several different units. The most common are eV/g, eV/cm³, or rads. A rad is equal to an energy absorption of 100 ergs per gram of material and is equivalent to $6.24 \cdot 10^{13}$ eV/g.

The chemical yield is now expressed as a G value, which is defined as the number of molecules consumed or produced per 100 eV of radiant energy absorbed by the system. G values for most compounds range from 1 to 10, although there are many exceptions. There are relatively stable compounds with G values less than 1 as well as compounds which undergo chain reactions to yield G values in the thousands.

The state (gas, liquid, or solid) of the system is also important in radiation chemistry. In general, the gas phase is more highly decomposed by ionizing radiation than are the condensed phases because there is a greater chance for the primary active species to escape from their point of formation before they recombine back into the original compound. A condensed phase, especially the solid phase, greatly enhances recombination. Metals, for example, are the most radiolytically stable materials because the ions produced by radiations such as gamma or beta rays are merely neutralized, so no permanent change results. However, when metals are bombarded with heavy particles such as alpha particles, atoms are actually displaced from their lattice sites, thereby causing physical changes. Large doses of particles are usually necessary before significant defects appear in a metal.

Irradiation of non-metals will change their chemical composition and thereby change their physical properties. For example, the radiolysis of hydrocarbon oils produces polymers which increase the viscosity of the oil. These changes are described in the main body of this report.

Oxygen is a very reactive species in radiation chemistry. Consequently, most of our studies were done both in the presence and in the absence of air. Frequently, large changes in radiolysis are effected by the oxygen in air. Nitrogen is relatively inert.

Comparison of Alpha and Gamma Radiation

The reason for including this topic is that whereas the materials of interest at Rocky Flats are actually being subjected to plutonium alpha particles (the absorbed dose from plutonium gamma rays is negligible compared to that from alpha particles), almost all radiation chemistry has been done with gamma rays. This is due to the much greater availability and ease of using gamma sources. It is necessary to understand the differences so that the information gathered from gamma irradiation studies can be applied to situations where alpha irradiation occurs.

As mentioned, the primary difference between the tracks produced by gamma and alpha rays is their stopping power or linear-energy-transfer (LET). For example, the mean LET for 1.25 MeV gamma rays from ⁶⁰Co is about 0.3 keV/micron in water, whereas the 5.3 MeV alpha particles from ²¹⁰Po have a mean LET of about 150 keV/micron in water. The 5.1 MeV alpha particles from ²³⁹Pu would have essentially the same LET as the ²¹⁰Po alpha particles. The slower, heavier alpha particles lose much more energy per unit distance because there is greater time for interaction with the electrons of the stopping medium; therefore, the range of the alpha particle is much shorter than that of the gamma ray. For example, a ²³⁹Pu alpha particle will only travel 3.7 cm in air, whereas it is most probable that a 1.25 MeV gamma ray would pass through gloveboxes unimpeded. The importance of this is that even though the specific activity (disintegrations per gram) of ²³⁹Pu is low, all of the alpha energy can be absorbed in a very small amount of material on the surface of plutonium metal. The range of the ²³⁹Pu alpha in an organic material is about 0.004 cm.

The difference in the radiation effects of these two types of rays is due to their difference in LET. Alpha particles create a track of excited ions and radicals so dense that they can react with each other; whereas, the ions and radicals created by a gamma ray (or more accurately, the energetic electrons produced by the gamma ray) will be sufficiently far apart to make ion or radical substrate reactions relatively more prevalent. This LET effect has not been investigated to the point where accurate predictions are feasible; nevertheless, some generalizations can be made.

There is little or no LET effect in the radiolysis of gas, so the information obtained by gamma irradiation can be used in estimating the effects of alpha irradiation of gases. In condensed phases, particularly liquids, LET differences do have a quantitative effect if not a qualitative one. In other words, the products may be the same from both alpha and gamma radiolysis, but the individual product

yields may vary. To generalize further, in most cases the LET is negligible, but there are examples where the yields may vary as much as 20 to 100 percent.

The conclusion is that information that has been acquired from gamma irradiations can be used directly to predict alpha effects, with the introduction of very little error. Even in the exceptional cases where there is a large difference resulting from the linear-energy-transfer, the gamma results will help to predict the major products and any appreciable yields.

Example of Radiolysis – Hexane

To present a quantitative concept of the extent of radiation damage, an example is given in which a relatively simple organic compound, hexane, contacts the surface of plutonium metal. Our object is to utilize the information known about the radiation chemistry of hexane to describe the practical outcome of contacting this liquid with plutonium. Hexane was selected for this example because its radiation chemistry is well known and it is chemically representative of the lubricating oils used in machining operations.

There have been a number of investigations of the radiolysis of liquid hexane and the main products are essentially as follows.

Product	G(Product)
H ₂	5.0
trans - C ₆ H ₁₂	1.2
C ₁₂	2.0
C ₁ -C ₅ compounds	0.1-0.5 for each compound
C ₇ -C ₁₀ compounds	0.1-0.5 for each compound

These irradiations were carried out at room temperature in the absence of air. The presence of air and high temperatures greatly affect the results, but these simpler results will be used. These irradiations were made using 800 keV electrons, which have essentially the same effect as high-energy gamma rays. The product yields, however, are not influenced by LET, so that the above G values can also be used for the alpha particle irradiation from plutonium.

Another simplifying feature of this example is that hydrogen is the only radiolysis product formed from hexane that is reactive with plutonium. To calculate the amount of hydrogen produced from hexane on a plutonium metal surface, the only information required is the above G values, the range of the alpha particles in the liquid, and the rate at which radioactive energy emanates from the surface of plutonium.

The rate of alpha energy release from the surface of either alpha or delta plutonium was calculated to be $2.9 \cdot 10^{13}$ eV/sec cm². The range of the 5.1 MeV alpha particle in hexane, calculated by an empirical formula, is approximately 0.0041 cm or 0.0016 inch. Using these values and assuming that the metal surface is covered with at least 0.0016 inch of hexane, the rate of H₂ production is calculated to be $1.45 \cdot 10^{12}$ molecules per cm² per sec. The rate of production from a thickness less than 0.0016 inch would be proportionately less.

In 30 days the hydrogen build-up would be about $1.2 \cdot 10^{-2}$ mg per cm². Although this is a small amount, it is still sufficient to cause visible pitting of the surface through the formation of plutonium hydride. It must be emphasized that the conditions in fabrication and assembly facilities can differ greatly from the simple example given here; thereby drastically altering the outcome. The reader undoubtedly recognizes that the actual conditions cannot be determined easily, so generalizations have to be made.

GENERAL EXPERIMENTAL PROCEDURE

Sample Preparation

Solids, liquids, and gases have been irradiated and analyzed as part of the radiolysis program at Rocky Flats. The sample material is placed directly into glass irradiation flasks, which can be of any form (see Figure 1). If gas analysis is required, or if irradiation under vacuum is desired, the air above the sample is evacuated while the sample is frozen. A gas handling system was constructed and is used for evacuation and for filling gas samples. When there is concern about stopcock grease contamination, samples are enclosed in break-seal tubes during irradiation.

Sample Irradiation

Materials are gamma irradiated in a Gammacell-220®, a commercial source containing about 3500 curies of Cobalt-60 (see Figure 2). Sample sizes are limited to the size of the irradiation chamber which is a cylinder 6 inches in diameter and 8 inches in depth. The dose rate within the chamber is not uniform, so for more accurate results, dosimetry (the determination of the energy absorbed per unit volume) is done in the same type flask and using the same position for the sample, as used for the actual sample. Dose, dose rate, and temperature can be varied in this apparatus.

FIGURE 1. Glass Flasks Used for Gamma Irradiation.

Beta and alpha sources are also available at Rocky Flats but have not been used for this project. The beta source is a commercial 3M irradiator containing 500 curies of ^{90}Sr - ^{90}Y . It can only be used for irradiating fluids (see Figure 3). An alpha irradiation chamber is also available for irradiating gases. However, the ^{210}Po alpha source for this apparatus must be ordered shortly before use because of its short half life (138 days).

Sample Analysis

Since the irradiated samples are not radioactively contaminated, they can be analyzed by any of the analytical tools that are available at Rocky Flats. These include instrumentation such as infrared spectroscopy, gas chromatography, and mass spectrometry. Mass spectro-

metry is very suitable for measuring radiolytic product gases and is the most widely used technique for our radiation studies.

LIST OF MATERIALS

The following materials are used in the radiation environment at Rocky Flats. The first and major part of the list consists of materials which merit the closest study because they are the ones most likely to adversely affect plutonium metal. These materials were chosen by applying the following criteria: (1) the quantity of material and time it is in contact with plutonium, (2) the radiation stability of the material, and

FIGURE 2. Gamma Irradiation Source.

FIGURE 3. Beta Irradiation Facility.

(3) its radiolytic decomposition products. A brief explanation concerning the use of the material is also included.

Solvents

CARBON TETRACHLORIDE — used as a solvent to remove organic contamination from plutonium.

TRICHLOROETHYLENE — same use as above.

Diisopropylamine (DIA) — used as an inhibitor for acid in trichloroethylene.

Diisopropylamine hydrochloride — an acid salt of DIA.

TRICHLOROTRIFLUOROETHANE (Freon TF) — used as a density-measurement medium for plutonium.

Lubricants

LATHE COOLANT (Texaco Regal Oil A) — used during plutonium machining.

LATHE COOLANT and Carbon tetrachloride.

DOW CORNING-550 Silicone Oil.

RYKON GREASE — used to lubricate the chain conveyors in the fabrication glovebox lines.

NYES WATCH OIL — a lubricant applied to parts before gaging.

Plastics and Rubber

POLYETHYLENE (PE) — used as a bag material in waste-bags, small storage bags, and on part carriers.

POLYVINYL CHLORIDE (PVC) — used as a bag material on gloveboxes.

TEFLON — used as a glovebox liner and in part-cleaning baskets.

NEOPRENE — used in glovebox gloves and vacuum chucks.

Air

Miscellaneous Materials

Materials that are found in gloveboxes that could possibly contact the plutonium, and materials that are placed in contact with plutonium for a very short period of time.

PERCHLOROETHYLENE — used as an organic solvent.

1,1,1, Trichloroethane — same use as above.

ETHANOL — same use as above.

MONOBROMOBENZENE — used as an energy transfer medium in ultrasonic testing. A possible production requirement.

METHYL-ISOBUTYL KETONE (MIBK) — used as a paint solvent.

DOW SILICONE VACUUM GREASE — used on vacuum chucks.

RTV-20 SILICONE RUBBER COMPOUND — used during inspection of parts.

PLEXIGLASS — used in drybox window.

ALUMINUM FOIL

MOLYKOTE Z

PAPER PRODUCTS

RADIOLYSIS OF PROCESS MATERIALS

Solvents

Carbon Tetrachloride

Use and Exposure

Carbon tetrachloride is used extensively to remove lubricants from plutonium. The solvent is re-used until its lubricant content becomes excessive. This mixture (CCl_4 + lubricant) or CCl_4 alone will be subjected to alpha radiation during the cleaning operation, and afterward if a large quantity of solvent is left on the plutonium. Therefore, exposure times could conceivably be extremely varied.

Radiation Chemistry

Only CCl_4 will be discussed here. Experimental work on the CCl_4 -lubricant mixture is presented in the section devoted to lubricants. The radiolysis of both degassed and aerated CCl_4 has been studied using ^{60}Co radiation.^(1,2)

(1) T. H. Chen, K. Y. Wong, and F. J. Johnston, *J. Phys. Chem.*, **64**, 1023 (1960).

(2) Z. Spurny and I. Janovsky, *Nature*, **190**, 624 (1961).

The radiolysis of degassed CCl_4 is relatively simple as only two products are detected. They are chlorine, produced with a G value of 0.66, and hexachloroethane (a white solid) for which no yield was determined. Irradiation in the presence of air (oxygen) produces a third product, phosgene (COCl_2), with a G of 12.1⁽²⁾ and increases the Cl_2 yield to 3.6. Moisture is also an important factor in the irradiation as it leads to the formation of hydrochloric acid (via the reaction, $\text{COCl}_2 + \text{H}_2\text{O} \rightarrow 2\text{HCl} + \text{CO}_2$) as well as the previously mentioned products.⁽³⁾

Mund, et al.,⁽⁴⁾ investigated the radiolysis of CCl_4 vapor using the alpha particles from Radon gas. Chlorine and C_2Cl_6 were the products identified and $G(\text{Cl}_2)$ was determined to be about 0.4, which is essentially the same as the yield in gamma irradiated liquid.

Possible Effects

Both of the gaseous products, Cl_2 and COCl_2 , are very reactive and would be corrosive toward plutonium. Hydrochloric acid, formed in the presence of moisture, would also be corrosive. The increase in product yields in the presence of oxygen is dependent on the amount of air that is in intimate contact with the CCl_4 during irradiation. The effect of ionizing radiation on the corrosivity of several metals in CCl_4 has been studied by Byalobzhetskii and Lukinskaya.⁽³⁾ They found that radiation considerably intensified the corrosion in all the metals. Plutonium would be expected to behave in a similar manner.

Trichloroethylene

Use and Exposure

Alk-Tri® (Dow) a commercial trichloroethylene (TCE), is widely used as a cleaning solvent in many plutonium production and processing applications. Consequently, it is exposed to alpha irradiation from plutonium. Exposure periods could vary greatly depending on the particular operation. Quality assurance tests made on Alk-Tri ensure that only those batches whose purity has met the prescribed standards will be used.

Radiation Chemistry

Very little work had been done previously on the radiolysis of TCE so an investigation was started. There

were a few studies of the two-phase system, trichloroethylene-water,⁽⁵⁾ but this is not exactly what is required. The TCE used in our work was of Baker reagent grade and it did not contain diisopropylamine, a common additive used to inhibit the light-initiated breakdown of TCE.

The gamma radiolysis of TCE in the absence of air was relatively simple, as there were only two major products; hydrochloric acid and chloroacetylene, each with a G value of about 0.25. This is a relatively low yield so that the pure compound can be regarded as fairly radiation stable.

The gamma radiolysis of TCE in the presence of air was complicated by a chain reaction involving oxygen. Extremely high yields were obtained but the products were difficult to analyze because of their high reactivity. For identification, the analytical techniques of mass spectrometry, infrared spectrometry, gas chromatography, and acid-base titration were used as well as comparison to the previous work that had been done on the radiolysis of tetrachloroethylene.^(6,7)

The total acid yield was measured by shaking the irradiated solvent with water and titrating the mixture with standardized NaOH solution. The $G(\text{H}^+)$ obtained was usually between 2000 and 3000, depending on the irradiation conditions.

There can be a very large variation in the yields depending on the amount of air (oxygen) that is available to the compound as it is being irradiated. These high variable yields are indicative of a chain reaction.

The major product has been determined to be dichloroacetyl chloride, CHCl_2COCl . Phosgene (COCl_2), hydrochloric acid, chloral, and other possible products, have not been identified. Both CHCl_2COCl and COCl_2 are highly reactive organic chlorides which makes them difficult to analyze. However, their rapid reaction with water to form HCl makes it possible to analyze for total acidity.

For comparison, Alk-Tri, the commercial brand of TCE that contains diisopropylamine, was gamma irradiated in the presence of air. The $G(\text{H}^+)$ for this experiment was 1600. Although this value is less than that for uninhibited TCE, it probably varies with irradiation time or dose. If the low concentration of additive were to be depleted

(3) A. V. Byalobzhetskii and V. N. Lukinskaya, *Soviet Atomic Energy*, 11, 805 (1961).

(4) W. Mund, P. Huyskens, and J. Debaisieux, *B CSAR BELG*, 41, 929 (1955).

(5) R. A. Sasse, *Health Physics*, 13, 1015 (1967).

(6) J. W. Sutherland and J. W. Spinks, *Can. J. Chem.*, 37, 79 (1959).

(7) G. Jeffrey and T. Boozalis, *Dow Report TOS-85*, Oct. 1965.

with continued irradiation, the acid yields would be expected to increase. More work remains to be done in this area.

Possible Effects

Since TCE used in production processes is exposed to alpha particles in the presence of air, the radiolytic decomposition of TCE can be a major corrosion factor. The compound undergoes a chain reaction with oxygen, producing acidic G values in the thousands, an enormous yield. Not only would CHCl_2COCl and COCl_2 react with plutonium but they are just as likely to react with atmospheric moisture to produce HCl , which can also corrode plutonium.

There are two ways to prevent the formation of these acidic products other than by minimizing the exposure of the solvent to plutonium. One is to exclude oxygen by using an inert atmosphere, and the other is to add large amounts of basic stabilizers to TCE. The amounts and effectiveness of these inhibitors would have to be evaluated. However, the advisability of taking the above steps is questionable. The results indicate that the extremely poor radiation stability of TCE in air can be a major contributor to the plutonium corrosion problem.

Diisopropylamine and Diisopropylamine Hydrochloride

Use and Exposure

Diisopropylamine (DIA) is used as a stabilizer in Alk-Tri (Dow's commercial brand of trichloroethylene) and is thus subjected to the same radiation dose as Alk-Tri. Alk-Tri is a solvent widely used for cleaning plutonium. Since the DIA concentration in the solvent is only about 1500 ppm it would not appear to be a significant contributor to the radiolytically induced corrosion. However, DIA and its hydrochloric acid salt, diisopropylamine hydrochloride ($\text{DIA}\cdot\text{HCl}$) have been found as residues on plutonium so they have to be considered as an important corrosion factor.

Radiation Chemistry

An investigation was undertaken to determine the radiation stability of DIA and $\text{DIA}\cdot\text{HCl}$ as there was no previous work published on the subject. The results of both compounds are reported together because of the similarity in structures and radiation chemistry. The DIA used was obtained from Eastman and the $\text{DIA}\cdot\text{HCl}$ was synthesized by passing anhydrous hydrogen chloride through DIA (a liquid). The resultant white solid was then dried in a vacuum line. Special handling care had to

be taken with the $\text{DIA}\cdot\text{HCl}$ because it is extremely hygroscopic.

Both compounds were irradiated with gamma rays in the presence and in the absence of air. The gaseous yield data are shown in Table I.

TABLE I.

Compound	Gaseous Product	In Vacuum	In Air
DIA	H_2	4.0	3.6
	CH_4	0.6	0.5
	CO	--	0.5
	CO_2	--	< 0.1
$\text{DIA}\cdot\text{HCl}$	H_2	2.0	1.8
	CH_4	0.08	0.07
	CO	--	0.3
	CO_2	--	0.07

The radiolytic decomposition of DIA and $\text{DIA}\cdot\text{HCl}$ appears to be relatively simple and essentially identical to each other. Apparently the ionically bonded HCl is less sensitive to radiation, whereas the organic portion of the $\text{DIA}\cdot\text{HCl}$ molecule is decomposed the same way as DIA. Air does not appear to play a major role in the radiolysis except in the formation of CO and, to a lesser extent, CO_2 . Hydrogen is the principal product and is formed at a rate which is considered typical for the radiolysis of organic systems.

Besides the gaseous products listed, acetone and the double-bonded amine are probably formed, but the yields of these two compounds have not been determined.

Possible Effects

Both DIA and $\text{DIA}\cdot\text{HCl}$ undergo "average" radiation decomposition producing H_2 as the major product. The H_2 yield is about the same as that from hydrocarbons such as the Texaco lubricating oil. Hydrogen is one of the major plutonium corroding agents because it forms the hydride. With the small amount of DIA that could be exposed to plutonium (because of its low concentration), it does not seem likely that the irradiation of DIA could result in a major corrosion problem. However, there is the possibility that $\text{DIA}\cdot\text{HCl}$, formed by reaction between DIA and the decomposition products of trichloroethylene, would remain on plutonium because of its low volatility and thus receive far greater radiation exposure than the more volatile solvents and lubricants.

Trichloro fluoroethane (Freon TF)*Use and Exposure*

Freon TF is sometimes used as a density-measurement medium for plutonium in various forms. Its radiation exposure depends on its length of service. Normal use is a 20-minute immersion and usually 30 immersions are made before the oil is changed.

Radiation Chemistry There are no reported radiation studies on this compound and very little work has been done on Freons. One report⁽⁸⁾ states that irradiation of Freon TF forms corrosive products in small yields. Since there is inadequate information on these compounds, the radiolysis of Freon TF will be investigated as soon as time permits.

Possible Effects

It is expected that long exposure to alpha particles from plutonium would produce corrosive products, but more information is required to predict the extent of damage to plutonium.

Lubricants

A number of lubricants and oil-bath fluids are used in the fabrication glovebox lines. Some of them come into direct contact with plutonium during the fabrication process. Others, such as chain-conveyor grease, are not supposed to contact plutonium but sometimes do. In any case, the general PuO_2 contamination of the lines will cause some radiolytic decomposition.

Good hydrocarbon lubricants can generally withstand large doses of radiation before their physical properties are significantly degraded. Our primary objective, therefore, was to determine what compounds are radiolytically produced that are corrosive to plutonium metal. Oxidation drastically reduces the life of a lubricant, and radiation accelerates oxidation; therefore, the lubricants were irradiated in the presence of oxygen (air) and under vacuum.

Lathe Coolant (Texaco Regal Oil A)*Use and Exposure*

A light hydraulic oil, Texaco Regal Oil A, is used as a cutting or machining oil during plutonium machining

operations. The large surface area of plutonium lathe turnings provides high radiation exposure to the oil in the lathe box.

Radiation Chemistry

Samples of Texaco Regal A were irradiated under vacuum or sealed under 500 torr air. A Bendix Time-of-Flight mass spectrometer was used to analyze gaseous products. Nearly all of the radiolytically generated gas was hydrogen. Table II shows the radiolytic production of hydrogen from deaerated oil with respect to absorbed radiation (G-values).

TABLE II. The Radiolytic Production of Hydrogen from Texaco Regal Oil A (Vacuum)

Radiation Dose (rads $\times 10^{-6}$)	G(H ₂)
8.4	2.3
17.0	2.3
44.0	2.4
98.0	1.6

Results from the oil samples irradiated in the presence of oxygen are shown in Table III. Yields are given for the consumption of oxygen and the production of hydrogen.

TABLE III. The Radiolytic Oxidation of Texaco Regal Oil A

Radiation Dose (rads $\times 10^{-6}$)	G(-O ₂)	G(H ₂)
1.43	3.0	2.1
2.39	3.2	2.2
4.78	3.6	2.3
7.17	1.2	2.0
8.36	1.6	1.8
9.55	0.9	1.3

Evidence that the oil had been oxidized was given by infrared spectroscopic analysis of the residual oil after irradiation. Carbonyl groups and polymers were found in the irradiated oil.

A comparison of the G(H₂) values in each table shows that hydrogen is produced at the same rate whether oxygen is present or not. It is difficult to explain why the rate of oxygen consumption decreases with increasing dose. One explanation could be that the oil is not a simple aliphatic compound. It contains corrosion inhibitors that could result in some undetected oxygen-producing reaction.

(8) M. Silver, G. Haston, and P. Rudolph, "Radiation Damage to Freon," CF-56-8-198, August 1956.

Carbon Tetrachloride-Texaco Regal Oil Mixture

Use and Exposure

After the machining operation, carbon tetrachloride is used to remove the residual cutting oil from the plutonium. This rinsing results in a mixture of oil, plutonium turnings, and carbon tetrachloride in the lathe gloveboxes.

Radiation Chemistry

It was believed that the decomposition of this mixture would produce hydrogen chloride. As previously discussed in this section (under Solvents) chlorine is known to be produced with $G(\text{Cl}_2) = 0.66^{(9)}$ during the radiolysis of CCl_4 . This chlorine should combine with the hydrogen produced from the oil to form hydrogen chloride.

Samples of a 1:1 mixture of carbon tetrachloride and oil were irradiated in the presence and in the absence of oxygen. The tubes containing the irradiated solution were opened under water. The mixture was shaken and titrated with 0.04N NaOH to determine the yield of water-soluble acid. The results are shown in Table IV.

TABLE IV. The Water Soluble Acid Produced During the Radiolysis of a Carbon Tetrachloride - Texaco Regal Oil A Mixture

Oxygen Absent		Oxygen Present	
Dose (rads $\times 10^{-6}$)	G(Acid)	Dose (rads $\times 10^{-6}$)	G(Acid)
1.41	9.8	1.20	16.7
2.39	8.1	2.39	17.7
4.54	9.2	3.58	14.3
4.77	9.0	4.77	15.5
9.55	8.5	4.77	16.3
11.5	9.2		

As can be seen in Table IV, the $G(\text{acid})$ nearly doubles when the radiolysis is conducted in the presence of oxygen. This can be explained as follows. Chlorine and phosgene have been reported to be produced with $G(\text{Cl}_2) = 3.6$ and $G(\text{COCl}_2) = 12.1$ during the radiolysis of carbon tetrachloride in the presence of oxygen. The presence of oxygen not only causes phosgene to be produced at a high rate but the $G(\text{Cl}_2)$ increases from 0.66 to 3.6. When the irradiated oil-carbon tetrachloride solution is shaken in water, phosgene is hydrolyzed and HCl is a product:

⁽⁹⁾ T. H. Chen, K. Y. Wong, and F. J. Johnston, *J. Phys. Chem.*, **64**, 1023 (1960).

In an attempt to confirm this explanation, a series of oil-carbon tetrachloride samples was irradiated and analyzed by infrared spectroscopy and gas chromatography. The results are shown in Table V.

TABLE V. The Radiolytic Production of Phosgene (Dose 4.77×10^6 rads)

Sample	Phosgene (μg)
CCl_4 , Vacuum	12
CCl_4 , Vacuum	61
1:1 CCl_4 , Oil, Vacuum	72
1:1 CCl_4 , Oil, Vacuum	11
CCl_4 , 400 torr air	483
CCl_4 , 400 torr air	425
1:1 CCl_4 , Oil, 400 torr air	420
1:1 CCl_4 , Oil, 400 torr air	370

It is obvious that phosgene is produced when oxygen is present. The relatively small amount of phosgene found in the deaerated samples is within the analytical error. Therefore only the relative amounts should be considered.

Possible Effects

From these data, it is apparent that the radiolytic rates of production of hydrogen, hydrogen chloride, and organic acids, are high enough to be considered as a dangerous source of plutonium corrosion in the glovebox lines.

Dow Corning-550 Silicone Oil

Use and Exposure

Dow Corning-550 silicone oil is used in various high temperature applications that involve its exposure to plutonium. The major effect of radiation on silicones is an increase in viscosity. Prolonged use (1 month) of DC-550 at high temperature in the radiation environment causes the oil to gel. The gelled oil is difficult to remove from the gloveboxes.

Radiation Chemistry

Another silicone fluid, General Electric SF-1093, was proposed as a replacement for DC-550. A high temperature radiation study was conducted to compare the two fluids. Samples of both oils were given a total dose of 2×10^7 rads. The molecular weight of the SF-1093 increased 62%, whereas the molecular weight of DC-550 increased only 8%. At this point the SF-1093 oil was just starting to gel. No data were collected concerning the radiolytic products from the silicones.

A comparison was made between the rates of corrosion of plutonium metal in DC-550 and SF-1093 at elevated

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temperature. Neither oil was excessively corrosive to plutonium. The DC-550 was found to be the more compatible with plutonium. Since no substitute has been found for DC-550, frequent oil changes are necessary to prevent gelling.

Rykon Grease

Use and Exposure

A general purpose industrial lubricant, Rykon 2 (manufactured by American Oil Co.), is used on the chain-conveyor system that moves plutonium through the glovebox lines. In the past, grease has been found on the plutonium but this has been eliminated by the use of covered carriers. Since large quantities of the grease are used inside the glovebox lines, it is necessary to have some knowledge of its stability to radiation.

Radiation Chemistry

A lubricating grease is a gel formed by dispersing a solid in an oil. Rykon consists of a straight-chain hydrocarbon oil gelled with an isocyanamide compound. Radiation effects on grease would be expected to correspond to those on liquid hydrocarbons such as machining oil.

Samples of Rykon were irradiated under vacuum and in the presence of oxygen (air). Gaseous products were analyzed by mass spectrometry. The gas yield was low and consisted mostly of hydrogen. The approximate G-value for hydrogen was $G(H_2) = 1$. Water was also found in the gas phase. The water yield did not appear to be significant but mass spectrometry is not very reliable for water analysis. Further tests may be necessary to determine the exact water yield.

It appeared that oxygen was being consumed in the grease samples irradiated in the presence of air. This indicates that the Rykon oxidizes in a radiation field. The grease was observed to be less viscous after absorbing a low dose of radiation. Softening of grease at low doses is characteristic; at very high doses a grease generally solidifies.

Possible Effects

Rykon grease appears to be reasonably stable to radiation, but since large quantities are used on the chain-conveyors and the grease is found nearly everywhere in the lines, it is difficult to determine the contribution it is making to plutonium corrosion.

Nyes Watch Oil

Nyes Watch Oil is a precision lubricant used on plutonium during inspection. It prevents gage tips from scratching

the metal. The oil is applied by the use of an oil-spiked cheese cloth. Carbon tetrachloride is used to remove the oil immediately after the inspection. Plutonium is then put through the standard ultrasonic cleaning procedure.

Nyes Watch Oil is sperm-whale oil. Its composition is mainly glycerides which are esters of fatty acids. Esters are known to oxidize when irradiated in the presence of air. As long as the oil is completely removed from the plutonium immediately after inspection, it is doubtful corrosion would be a problem.

Plastics and Rubber

Polyethylene

Use and Exposure

Plutonium metal is frequently enclosed in plastic bags to facilitate handling. The two most common types of plastics used in contact with plutonium are polyethylene (bags for contaminated waste) and polyvinyl chloride (used in the glovebox bag-out procedure). Both materials have been used as wrapping for plutonium during storage.

Radiation Chemistry

Polyethylene has been the subject of many radiation studies because of its simplicity. The following work was conducted on the material used at Rocky Flats.

Fourteen samples of polyethylene sheet weighing from 10 to 20g were irradiated. Hydrogen was the only significant product. The G-value for the production of hydrogen was $G(H_2) = 2.2$. This is in fair agreement with the $G(H_2) = 2.03$ reported by Karpov⁽¹⁰⁾ but slightly lower than the 3.1 reported by Dole.⁽¹¹⁾

Other gaseous products were small amounts of fragments of chain molecules containing one to four carbon atoms.

Polyethylene oxidizes when irradiated in the presence of oxygen. The $G(-O_2)$ for the polyethylene used at Rocky Flats was found to be 8.1. This value is in fair agreement with the maximum value of $G(-O_2) = 10$ reported by

(10) V. L. Karpov, Conf. Acad. Sci., USSR, Peaceful Uses of Atomic Energy, 1955, Div. Chem. Sci., 3-32.

(11) M. Dole, T. F. Williams, and A. J. Arvia, Proceedings of the Second International Conference on the Peaceful Uses of Atomic Energy, Geneva, 1958, Vol 29, p 171, United Nations, New York (1959).

Matsuo.⁽¹²⁾ G values for oxygen consumption depend on film thickness and oxygen pressure.

Polyvinyl Chloride

Radiation Chemistry

Nine samples of polyvinyl chloride (PVC) bag material were irradiated in the same manner as was the polyethylene. The hydrogen yield was $G(H_2) = 0.11$. The tubes containing the irradiated PVC were opened under water, shaken, and titrated with 0.04 N NaOH to determine the yield of water-soluble acid. The acid yield, most of which was HCl, was $G(HCl) = 0.21$.

Polyvinyl chloride becomes highly colored (dark brown) upon irradiation.

Possible Effects

Hydrogen, and possibly acids, can be produced by the radiolytic decomposition of polyethylene and polyvinyl chloride in the glovebox lines. It is recommended that plutonium metal not be stored (months) in contact with either of these materials, especially if it is necessary to maintain the integrity of the surface of the metal.

Teflon

Use and Exposure

Teflon (polytetrafluoroethylene) is a material that is finding increasing use inside plutonium gloveboxes. It is used in some plutonium carriers, cleaning baskets, and recently as a coating on the inside of gloveboxes.

Radiation Chemistry

Teflon has many excellent physical and chemical properties, but it has the lowest stability to radiation of all plastics and rubbers. It is particularly sensitive to radiation when irradiated in the presence of oxygen and eventually loses all mechanical strength and crumbles to a powder.⁽¹³⁾

The radiolysis products have been identified as small amounts of CF_4 and C_2F_6 and a high proportion of

COF_2 .⁽¹⁴⁾ Average gas yields have been reported to be $G(\text{gas}) = 0.05$.⁽¹⁵⁾ Gas measurements may not be meaningful because of the high reactivity of F_2 .

Possible Effects

Teflon powder will be found on the surface of plutonium metal that has been in contact with Teflon. The surface of Teflon will flake-off in an alpha radiation environment but no significant corrosion problems will result.

Neoprene

There are thousands of Neoprene rubber gloves on the gloveboxes at Rocky Flats. Very little information could be found concerning the radiolysis products of Neoprene. Oxygen appears to play a minor role in radiation-induced damage. The radiolytic production of gas has not been measured. The Neoprene polymer molecule, 2 Chloro-1, 2-butadiene, could possibly yield hydrogen chloride during radiolysis; therefore a radiation study is planned.

It is known that chlorinated and other polar solvents have a swelling effect on Neoprene. Swelling caused by carbon tetrachloride would probably shorten the life of a glove considerably. Handling plutonium metal with gloves that have been subjected to a radioactive contaminated, chlorinated solvent would not be safe. This would probably leave a corrosive residue on the metal.

Air

Exposure

There are air atmospheres in all the gloveboxes in the assembly lines. The air is dried so that the moisture content is about 100-300 ppm. This slightly moist air is constantly subjected to alpha radiation from plutonium. However, there is a continuous turnover as ventilation usually displaces the air in the box every few minutes.

Radiation Chemistry

The radiation chemistry of oxygen and mixtures of oxygen and nitrogen has been extensively

(12) H. Matsuo and M. Dole, *J. Phys. Chem.*, **63**, 837 (1959).

(13) L. A. Wall and R. F. Florin, *J. App. Polymer Sci.*, **2**, 251 (1959).

(14) J. H. Golden, *J. Polymer Sci.*, **45**, 534 (1960).

(15) V. L. Karpov, *Conf. Acad. Sci., USSR, Peaceful Uses of Atomic Energy*, 1955, Div. Chem. Sci., 3-32.

studied. (16,17,18) Although the mechanisms have been fairly well established, the product yields are variable depending on the experimental conditions. For example, in a static system the yield of ozone is very small, whereas in a flowing system the yield is much larger and depends on the flow rate and, inversely, on the dose rate. (16) There are two kinds of yields to be considered. One is the rate of product formation with increasing dose, and the other is the equilibrium concentration of products.

The radiolysis products from dry air are ozone and the oxides of nitrogen, NO_2 , N_2O , and NO . The G values for their initial formation in static systems are in the neighborhood of 1. These yields are increased two to three fold using a flow system. Because of efficient back reactions in the mechanism, the concentration of ozone at equilibrium is very small, on the order of a few ppm. On the other hand, the equilibrium concentrations of NO_2 and N_2O can be as large as a few percent. The NO yields are much smaller.

When moisture is present the main product is nitric acid, which is formed until the water vapor is exhausted. G values are around 1, but vary with water concentration.

Possible Effects

Although exposure conditions in the gloveboxes are variable, generalizations can be made about possible corrosion effects caused by irradiated air. The radiolyses products, O_3 , NO_2 , and HNO_3 (formed with H_2O present) would be very corrosive toward plutonium. However, little or no damage would be expected because the very small product concentrations formed in the gas phase would be displaced by the normal air flow through the boxes.

One condition which would greatly increase the corrosion probability is a large increase in the moisture content of the air. If the H_2O concentration were increased to thousands of ppm (quite possible with H_2O in the line), the nitric acid production could be increased to the point where it would corrode plutonium. This effect could be responsible for the increased rate of plutonium air oxidation in the presence of water vapor.

(16) S. C. Lind and D. C. Bardwell, J. Am. Chem. Soc. 51, 2751 (1929).

(17) P. Hartech and S. Dondes, J. Chem. Phys. 27, 546 (1957).

(18) P. Hartech and S. Dondes, J. Phys. Chem. 63, 956 (1959).

Miscellaneous Materials

The following materials are considered to be of secondary importance in the radiation environment at Rocky Flats. The quantities used and exposure times are much less than that of the materials in the first part of the list. However, they are included because: (1) they do make a minor contribution to the radiation-corrosion effect, and (2) there is always the possibility that their use will be greatly increased.

Perchloroethylene

Perchloroethylene is used in some operations as a solvent to remove organic contamination from plutonium. Its radiation chemistry is very similar to that of trichloroethylene (TCE) so the section on TCE can be referred to for further information. In general, radiolysis of perchloroethylene in the presence of oxygen produces an extremely high yield of acidic products which are capable of corroding plutonium.

1,1,1-Trichloroethane

1,1,1-Trichloroethane is used infrequently as a cleaning solvent. This compound is fairly similar to Freon TF. Unfortunately, the radiation chemistry of these compounds has not been investigated. Hydrochloric acid would be expected as a major radiolysis product; however, it would be difficult to predict anything further.

Ethanol

Ethanol is also used as a cleaning solvent. Irradiation with gamma rays produces hydrogen, acetaldehyde, and 2,3-butanediol. (19) In air-saturated ethanol, hydrogen peroxide is also produced in moderate amounts. This material can be considered to be equivalent to hydrocarbons such as machining oil with respect to its radiation-corrosion properties.

Monobromobenzene

Monobromobenzene is used as the liquid energy-transfer medium in an ultrasonic testing operation. Irradiation of this compound produces small yields of HBr and Br_2 as well as a number of aromatic compounds. (20,21) Bromine

(19) E. Hayon and J. J. Weiss, J. Chem. Soc. 3962 (1961).

(20) S. U. Choi and J. Willard, J. Phys. Chem. 66, 1041 (1962).

(21) A. F. Everard and G. A. Swan, Proc. Chem. Soc. 1961, 212.

and HBr can corrode plutonium; however, the immersion periods are short and no problems have been experienced. The direct corrosion of plutonium by monobromobenzene may be more important than radiation-induced corrosion. Even though a direct reaction is known to occur, the contact times have apparently been too short for any appreciable corrosion.

Methyl Isobutyl Ketone

Methyl isobutyl ketone (MIBK) is used as a paint remover in some gloveboxes. The exposure to alpha radiation may be negligible for this compound as well as some of the other compounds that are in the last part of this list. However, they are all considered because of their proximity to plutonium. The radiation chemistry of MIBK has not been investigated, but there is sufficient information available from the radiolysis of very similar compounds to be able to predict that the irradiation of MIBK will produce moderate amounts of H_2 , CO, and hydrocarbons. Hydrogen is reactive with plutonium, whereas CO will react with plutonium only at elevated temperatures.

Dow Silicone Vacuum Grease

This material is used on vacuum chucks. Silicones in general have inferior radiation stability although there are silicone greases which have relatively high stabilities because of their higher aromatic content. G values for gas production may vary from 0.1 to 3. Product analyses of silicone greases have not been made so there is insufficient information to make valid predictions.

RTV-20 Silicone Rubber Compound

This material is used to make impression moldings of plutonium during inspection, and residues of the RTV-20 have been found on the parts. Its radiation stability would be similar to that of the silicone vacuum grease, but there is very little information available on the silicone rubber compound.

Plexiglas

Use and Exposure

Polymethylmethacrylate (Plexiglas, Perspex, Lucite) is used in glovebox windows. It is, therefore, constantly exposed to radiation from the plutonium and americium contamination in the boxes.

Radiation Chemistry

Plexiglass can withstand large doses (10^7 - 10^8 rads) at room temperature though it will eventually crack. On irradiation, the main side chains of the polymer rupture

and decompose with the evolution of gas. Heating irradiated Plexiglas causes it to swell into a foamy mass as the result of gas evolution.⁽²²⁾ The gas consists of hydrogen, carbon monoxide, carbon dioxide, and methane. Irradiation of Plexiglas in the presence of oxygen seems to decrease the rate of degradation.⁽²³⁾

Possible Effects

It is doubtful that the radiolysis of Plexiglas glovebox windows significantly contributes to plutonium corrosion. Since the windows are one-half-inch-thick, alpha particles have little effect; although windows on plutonium-238 boxes at Mound Laboratory have become nearly opaque as the result of alpha radiation.

Aluminum Foil

Plutonium is wrapped in aluminum foil for protection during the final cleaning steps. This foil is not believed to be contributing organic contamination to plutonium. A quality assurance program, conducted by the Analytical Laboratories, ensures that hydrocarbon contamination on the surface of new foil is <0.5 mg/ft².

Molykote Z (MoS_2)

Molykote Z (manufactured by Dow Corning) is a pure, dry, solid lubricant used to lubricate gages in the plutonium inspection gloveboxes. Once MoS_2 contacts the surface of a metal, it is very difficult to remove (a stripper must be used). The standard ultrasonic cleaning procedure probably does not remove the MoS_2 , although the quantity of lubricant is very small. The residual MoS_2 on the surface of plutonium metal was taken into consideration as a potential source of radiation-induced corrosion.

Samples of Molykote were irradiated under vacuum to high total doses (2×10^8 rads). Gas evolution was insignificant. The good radiation stability of Molykote is not surprising since it is a pure inorganic compound.

(22) P. Alexander, A. Charlesby and M. Ross, Proc. Roy. Soc., 329 (1954).

(23) L. A. Wall and D. W. Brown, J. Phys. Chem., 61, 129 (1957).

Paper Products

These include cardboard fiber packs, ice cream cartons, Kimwipes, cardboard inserts on part carriers, masking tape, nylon gloves, cotton gloves.

Although these materials are used in the gloveboxes, it is doubtful they contribute to the corrosion of plutonium. In most cases, these items are not directly involved with fabrication. The cellulose in paper products degrades rapidly under radiation, and the paper becomes brittle, and powders easily. Small amounts of water, hydrogen, carbon dioxide, and methane are evolved.

CONCLUSIONS

The information presented in this report shows that there are many materials in the production-fabrication lines which are highly susceptible to radiolytic decomposition. The radiolysis products, in general, are reactive toward plutonium and could lead to corrosion. However, the extent of radiation damage depends on the exposure conditions. Under "normal" conditions, i.e. when the contact time between material and plutonium is very short and there is an appreciable air flow through the box which will remove the radiolysis products, the amount of corrosion from this source is expected to be negligible. This report merely emphasizes the possibility of extensive corrosion damage caused by prolonged exposure.