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RADIOLYTIC GAS GENERATION FROM CALCINED
NUCLEAR WASTE

G. L. Tingey
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August 1977

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RADIOLYTIC GAS GENERATION FROM CALCINED NUCLEAR WASTE

Garth L. Tingey
W. Dale Felix

INTRODUCTION

The handling and storage of high level nuclear waste presents problems spanning many scientific and technological disciplines. Among these problems is the need to evaluate the interaction of the ionizing radiation released by radioactive decay of the waste with compounds present in the storage media. Decomposition of some of these compounds may potentially lead to a variety of species. In this study, the formation of gaseous species which result in pressurization when the waste is contained in a closed system is examined. We have restricted our work to calcined inorganic oxides which are selected to simulate a typical fission product mixture which has been converted to oxides with relatively high chemical and thermal stability. Small quantities of nitrate salts and water remain in the calcine produced at 500°C, the normal operating temperature of the calciner. Unless these gas generating components are removed (i.e., heating up to 900°C) they will decompose both thermally and radiolytically to yield gaseous products.

RADIOLYSIS OF WATER

The radiolysis of water has been studied more thoroughly than any other compound and over the past several years a number of excellent books and reviews have been published.⁽¹⁻⁸⁾ The radiation induced decomposition of pure water has historically been a complex and bewildering problem. For example, Fricke⁽⁹⁾ showed, in an early study, that x-rays applied to an enclosed vessel of water appeared to result in essentially no decomposition at all. In the most highly purified water available at that time, only traces of hydrogen were found. However, in another study, irradiation of water enclosed with a large free volume in a vacuum system gave large yields of hydrogen.⁽¹⁰⁾ It was not until Allen's⁽²⁾ definitive work that some order could be brought to this chaotic picture. Later studies have further clarified the picture. Four main factors affect the decomposition of water: 1) Pure water is extremely difficult to obtain, and the decomposition of water by radiation is dependent on the impurities present. 2) The amount of decomposition is a function of

free volume above the water. 3) A reduction in LET (linear energy transfer) results in a shift in the equilibrium toward a greater recombination of products in the gas phase. Conversely, the yield of molecular products increases relative to the free radical yield at higher LET.⁽¹¹⁾ For example, the amount of water decomposed by nuclear reactor radiation reaches an equilibrium composition at a fairly low level of hydrogen concentration. 4) Water decomposition is a function of pH.

The radiolytic decomposition of water, as presently understood, can be explained in terms of ionization and subsequent formation of free hydrogen atoms and OH radicals. The ions and free radicals are formed heterogeneously in spurs along the ionizing particle track. Reactions of these species lead to molecular products. Some of the reactions which can occur are:

The yields (defined as molecules formed or changed per 100eV energy absorbed) for the primary products, OH, H, e^-_{aq} , and HOOH, have been determined by kinetic techniques. Molecular product yields are a function of the primary yields as defined by the reaction mechanism. In extremely pure solutions, under controlled conditions, molecular product yields can be predicted quite accurately from the established primary yields.

As noted above, LET differences may cause substantial differences in product yield. This is shown by comparing alpha radiolysis (high LET) with gamma or beta radiolysis (low LET). High yields of hydrogen are observed from α radiolysis (high LET) of pure water⁽¹²⁾ giving a constant $G(H_2) = 1.2$ molecules/100eV independent of total dose; lower LET radiation as from γ results in a rapidly reached steady-state with the ultimate H_2 , O_2 , and H_2O_2 yields being very small. The lower yield from low LET radiation types has been ascribed to radiation induced recombination reactions occurring in the gas phase. This phenomenon is obviously of importance to radiolysis in nuclear wastes since the radiation will be a mix of many types and energies. A major factor in the LET effect may be that the gaseous products removed from the condensed phase are out of range of the α particles but the x-rays and to a lesser extent β particles penetrate into the gas phase and cause reaction. Thus, in the mixed radiation field, the gas phase reactions of the product will occur and prevent the continuous pressure build up. If this postulate proves to be correct, the presence of gamma rays will tend to limit the pressure which might otherwise occur in a purely α radiation flux.

RADIOLYSIS OF NITRATES

Although there have been a number of studies on the irradiation of inorganic nitrates, the decomposition mechanism is still open to question. The chemical effect of irradiation can, however, be summarized by a few general observations:⁽¹³⁾

1. The net chemical reaction is

with a significant radiation induced back reaction

2. There does not appear to be a dose rate effect; thus, competition reactions of primary or intermediate species (i.e., $O + O \rightarrow O_2$) must not be significant.
3. LET effects are apparently due to the production of high localized temperatures; and
4. There is very little, if any, decomposition of the nitrite ion.

Initial $G(\text{NO}_2^-)$ values of solid nitrate salts which vary from 0.2 to 3.1 molecules/100eV⁽¹³⁾ are dependent on the cation of the salt and the radiolysis temperature. An increase in temperature is generally reflected in an increase in yield.⁽¹⁴⁾ Oxygen production is generally co-linear with NO_2^- production in the correct stoichiometric ratio. The detectable amount of oxygen will decline with respect to NO_2^- when reaction of oxygen with metal atoms is favorable.⁽¹⁵⁾ The presence of impurities, such as $\text{Ca}(\text{NO}_3)_2$ causes an initial increase in yield which then falls off to the normally expected yield after an absorbed dose of about 1.0×10^{21} eV/g.⁽¹⁶⁾ A dramatic increase in yield is noted when the nitrates are liquified. For example, in the molten state, the initial G-value for NO_2^- production is 5.5 ± 0.5 with an extrapolated steady state NO_2^- concentration of 33 moles per mole of LiNO_3 when irradiated with ^6Li .⁽¹⁶⁾ This value, however, is not in agreement with a reportedly much lower G-value using gamma radiation.⁽¹⁷⁾

Most results reported are for the initial G-value. Steady state concentrations are expected to be strongly dependent upon experimental conditions. In one of the only studies available for which equilibrium or long term products were measured for nitrates irradiated in a nuclear reactor, the initial G-value for the production of O_2 ⁽¹⁸⁾ is consistent with the above reports. The equilibrium yields were, however, dependent upon irradiation period and flux. Thus, extrapolation of the results to long term irradiations or different flux conditions will probably lead to incorrect conclusions.

RADIOLYSIS OF SORBED MOLECULES OR SPECIES INCORPORATED IN SOLIDS

In nuclear waste, we are primarily concerned with water sorbed on the surface of solid particles or hydrates and nitrates incorporated within the oxide matrix. In this instance the radiolytic behavior will likely differ significantly from that in a homogeneous system, whether liquid or gas. The cause for the different behavior is three-fold. First, energy transfer can occur from the solid thereby causing much higher energy deposition to the sensitive molecule than would be expected in the homogeneous system. Second, the behavior of the active species (atoms, radicals, or ions) may be altered. Stabilization by the solid matrix may increase their lifetimes by orders of magnitude thus resulting in an entirely different reaction sequence or perhaps even in totally inert behavior. Third, sorbed impurities may play a much greater role than would a comparable concentration homogeneously distributed.

Krylova and Dolin⁽¹⁹⁾ reported on the radiolysis of water sorbed on silica gel. They found H_2 yields up to 40 molecules/100 eV indicative of energy transfer from the solid to the water molecules. Oxygen from the water decomposition is liberated as CO_2 which is evidently formed by interaction with organic impurities on the silica surface. In work with the complicated solids generated in calcination of nuclear wastes, these effects are of great interest and must be carefully considered.

EXPERIMENTAL APPROACH

DESCRIPTION OF CALCINED SOLID

The formulation used in the radiation experiment was representative of a typical high-level waste resulting from reprocessing spent LWR fuel. The sample was prepared by calcining a solution of nitrate salts in a fluidized bed at about 500°C. The nominal composition of the resulting oxide mixture is given in Table 1. In addition, about 5.1% NO_3^- and 0.3% H_2O remained in the mixture. The calcined powder was sieved to yield a powder with irregular shaped particles ranging in size from 125 to 175 μm .

GAMMA RADIOLYSIS

The gamma radiolyses were performed in a gamma flux generated by a 260,000 curie ^{60}Co source under about 14 feet of water. Access to the source was through 2-inch diameter dry tubes. Various gamma dose rates could be achieved by adjusting the distance from the sample to the center of the source. The dose rates were determined using ceric sulfate dosimetry in conjunction with electronic dose rate meters. The dose rate was corrected for decay with time using the known half life of ^{60}Co (5.7 years). 5,27?

The calcined powder was placed in a quartz sample tube which was connected to a capacitance manometer with stainless steel capillary tubing. The sample was outgassed under vacuum at 200°C until no further gas release was observed.

The pressure and temperature were monitored continuously while the sample was in the radiation zone. Gas samples were withdrawn periodically via a gas sampling valve and analyzed by mass spectrometry.

TABLE 1: Nominal Composition of PW 7-2 Calcine*

<u>Oxide</u>	<u>Composition (Weight %)</u>	<u>Oxide</u>	<u>Composition (Weight %)</u>
CeO ₂	18.2	NiO	1.8
La ₂ O ₃	16.1	Gd ₂ O ₃	1.5
Nd ₂ O ₃	12.6	TeO	1.3
MoO ₃	11.1	Y ₂ O ₃	0.82
ZrO ₂	8.7	Cr ₂ O ₃	0.60
Fe ₂ O ₃	8.4	Co ₃ O ₄	0.53
P ₂ O ₅	4.7	Dy ₂ O ₃	0.37
Pr ₆ O ₁₁	3.8	Eu ₂ O ₃	0.30
BaO	2.7	Na ₂ O	0.20
Sm ₂ O ₃	2.0	CdO	0.17
K ₂ O	2.0	Ag ₂ O	0.15
SrO	1.9	Er ₂ O	0.07
		Ho ₂ O ₃	<u>0.04</u>
		TOTAL	100.00

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*Calculated from composition of solution prior to calcining
assuming all compounds are converted to the oxide.

ALPHA RADIOLYSIS

The alpha radiolysis experiment was performed to evaluate the expected differences between α and γ radiation. For this test 0.01 g of Cm^{244} was added to 5.55 g of a calcined waste powder, both having a particle diameter less than 40 μm . Cm^{244} is an α emitting radioactive isotope with a half life of 18.1 years. The powders were thoroughly mixed in order to obtain a mixture as nearly homogeneous as possible. The powder mixture was placed in a tube and attached to a capacitance manometer where the pressure was monitored continuously. Gas samples were taken periodically and analyzed by mass spectrometry.

RESULTS AND INTERPRETATION

GAMMA RADIOLYSIS EXPERIMENT

When the sample of the calcined mixture was placed in the gamma flux, a pressure rise was observed. Removal of the sample from the radiation zone stopped the gas generation thereby indicating that the pressure increase resulted from the radiolysis. Figure 1 is a plot of the gas generated as a function of gamma dose from 23.6g sample of calcined waste. Two separate dose rates (5×10^5 and 1.6×10^7 R/hour) were used in this study. The points lie on a smooth curve irrespective of the dose rate indicating an insignificant dose rate effect in this flux range.

The rate of pressure generation initially appeared to be nearly constant with fluence but as the gaseous product concentration increased the rate of generation decreased. This decreasing rate with time was expected because of the potential for reaction of the product gases and/or adsorption of the gas on the high surface area calcined powder. Radiolysis of the product gases was reduced because about one-half of the gas volume was outside the radiation flux. In addition, some of the rate decrease with time may be attributed to depletion of the gas generating species in the calcine since nearly 15% of the NO_3^- was decomposed during the course of the run.

A sample of the gas was collected for analysis by mass spectrometry; the composition is shown in Table II.

Figure 1. Gamma Radiolysis of Calcined Solid

TABLE II: Composition of gas from γ -Radiolysis
of Calcined Solid

O_2	= 98%
N_2	= 1.2%
CO_2	= 0.5%
H_2	= 0.3%
CO	< 0.1%
CH_4	< 0.01%

From these data we conclude that the gas almost certainly results from radiolytic decomposition of NO_3^- salts via reaction (1). Since the nitrite ion (NO_2^-) is resistant to further decomposition it is not surprising that NO_x gases are not observed. The N_2 may be a product of the reaction but the limited quantity observed could also be due to residual air in the system. The small quantities of carbon containing gases, CO_2 , CO , and CH_4 , apparently are formed from decomposition of organics or carbonates in the system or from reaction of oxygen with residual carbon. The hydrogen is probably due to decomposition of water of hydration or sorbed water.

Because of the complexity of the possible reactions and since the energy sorbed can be accommodated in many ways, radiolytic yields cannot be assigned to specific reactions. Nevertheless, estimation of the radiolytic yield provides some further understanding of the process. For this calculation, we have assumed the initial yield to be equal to the average yield up to 2×10^8 R. The $G(O_2)$, based on the total energy sorbed in the solid, was then determined to be 0.1 molecule/100 eV. If the yield is calculated based on the energy adsorbed in the NO_3^- only, a $G(O_2)$ of 2.0 molecules/100 eV is obtained. These $G(O_2)$ values appear to be in reasonable agreement with the published 100 eV yield of pure nitrate salts. For example, a $G(NO_2^-)$ of 1.5 has been reported⁽¹³⁾ for KNO_3 . From stoichiometric considerations the corresponding $G(O_2)$ would be 0.75 molecules/100 eV. Our observed $G(O_2)$, based on energy sorbed in NO_3^- only, is thus about twice that expected for decomposition of pure KNO_3 thus indicating that energy initially sorbed in other solid components is partially transferred to the nitrate radical.

ALPHA RADIOLYSIS EXPERIMENT

The calcined material used for the α -radiolysis experiment was similar in composition to that shown in Table I but had less than 1% nitrate and had been mixed with a small amount of Cm^{244} to supply the α -radiation. Upon closing the valve to isolate the system from the vacuum pump, the pressure increased relatively rapidly at a rate which decreased with time. Figure 2 is a plot of the gas generated as a function of dose. Each time the system was outgassed and the valve closed the same pattern of pressure increase was observed. Several simultaneously occurring processes could account for this behavior.

- (1) Radiolysis of the NO_3^- and H_2O to produce gaseous products.
- (2) Desorption of gases from the surface causing a rapid initial pressure rise.
- (3) Radiolysis of the product gases yielding condensed phase products.
- (4) Resorption of gaseous products on the surface. This process increases proportionately to the gas pressure.

This type of experiment is complicated by our inability to separate the radiolytic gas generation from outgassing. In the gamma radiolysis the sample could be thoroughly outgassed prior to placing it in the radiation field. This is not possible with the internal α -radiation source and thus less definitive results are obtained.

The gaseous products were separated into two fractions, about 30% condensable at -78°C and 70% which remained gaseous at that temperature. The condensable fraction was not analyzed further but was assumed to be primarily H_2O . The non-condensable fraction was analyzed by mass spectrometry; the composition is shown in Table III.

TABLE III: Gaseous Products Formed by Alpha Radiolysis of Calcined Solid

<u>Product</u>	<u>Concentration, %</u>
O_2	54%
NO_x	35%
CO_2	4.6%
H_2	3.3%
N_2	2.9%

Figure 2. Alpha Radiolysis of Calcined Waste

CONCLUSIONS

The gas generation rate for both the gamma and alpha radiolysis experiments appear to be about the same. However, there is a clear difference between alpha and gamma radiolysis in the gases generated. The α experiment indicates much more complex reaction events than indicated in the gamma tests. This is demonstrated by a wider range of products from the α -radiolysis experiment. Although the major product was oxygen, significant quantities of gas condensable at -78°C , probably water, and fairly large fractions of nitrogen oxides are produced. By contrast, the gamma radiolysis experiment yielded nearly 100% O_2 demonstrating that the reaction is almost entirely due to reaction 1.

A steady state level should be achieved before all of the NO_3^- salts are decomposed because of the occurrence of the reverse reaction

The pressure at which this will occur is presently unknown. However, as shown in Figure 1, the rate of pressure increase is still substantial at a pressure well above 1 atmosphere. Furthermore, in practice the canister will probably be maintained at higher temperature; therefore, the pressure will be further increased by the PV/T relationship.

From our results, we conclude that radiolysis of NO_3^- or H_2O will pose no insurmountable problems in the shipping and storing of waste calcines even though some additional pressurization will occur from the decomposition of residual nitrate salts. Release of gas by thermal decomposition or thermal desorption will produce gas pressures much more rapidly than radiolysis if the calcine is subjected to high temperatures which could result from accidents during transport or storage where fires result. Such conditions are expected to lead to pressures of hundreds of atmospheres in closed containers. An evaluation of thermal gas release has been made and reported earlier. (22) - ?

We have clearly shown the potential of radiolytic reactions in nuclear wastes. It is important that these processes be considered and evaluated whether the waste be a calcine, glass matrix, or other form. It is hoped that this paper will lay the groundwork for other studies dealing with the importance of radiolytic reactions in nuclear waste processing and storage.

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