185
5-8-78
RFP-2652
April 14, 1978

14.37
RFP-2652
April 14, 1978

EFFECT OF RADIATION AND ACIDS ON
THERMAL STABILITY OF ION EXCHANGE RESINS

A. R. Kazanjian

Chemistry Research and Development
PRODUCT INTEGRITY AND SURVEILLANCE

Rockwell International

Atomics International Division
Rocky Flats Plant
P.O. Box 464
Golden, Colorado 80401

U. S. DEPARTMENT OF ENERGY
CONTRACT EY-76-C-04-3533

MASTER

MASTER

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

LEGAL NOTICE

This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the Department of Energy, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, expressed or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

Printed in the United States of America

Available from the

National Technical Information Service

U. S. Department of Commerce

Springfield, Virginia 22161

Price: Printed Copy \$4.50 Microfiche \$3.00

Price Is Subject to Change Without Notice

Printed
April 14, 1978

RFP-2652
UC-4 CHEMISTRY
TID-4500-R66

**EFFECT OF RADIATION AND ACIDS ON
THERMAL STABILITY OF ION EXCHANGE RESINS**

A. R. Kazanjian

Chemistry Research and Development
PRODUCT INTEGRITY AND SURVEILLANCE

SUBJECT DESCRIPTORS

Ion Exchange Materials
Radiation Effects
Thermal Analysis
Inorganic Acids

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

NOTICE
This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Department of Energy, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

**Prepared under Contract EY-76-C-04-3533
for the
Albuquerque Operations Office
U. S. Department of Energy**

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

CONTENTS

Abstract	1
Introduction	1
Experimental	1
Results	1
Effect of Gamma Radiation	1
Effect of Alpha Radiation	2
Effect of Acids	6
Nature of Thermal Degradation	7
Product Analysis by Mass Spectrometry	7
Conclusions	8
References	8

ACKNOWLEDGMENT

The aid of R. S. Cichorz (Rocky Flats Analytical Laboratory) in doing all of the differential thermal analyses and infrared analyses is greatly appreciated.

EFFECT OF RADIATION AND ACIDS ON THERMAL STABILITY OF ION EXCHANGE RESINS

A. R. Kazanjian

Abstract. An investigation was made of the effects of high-energy radiation and strong acids on the thermal stability of ion exchange resins. Dowex® 1-X4 (anion exchange resin) and Dowex 50-X8 (cation exchange resin) were subjected to gamma and alpha radiation in addition to nitric and hydrochloric acids. The resins then were analyzed by differential thermal analysis and infrared analysis to examine the changes in thermal stability. Gamma irradiation (up to 1.6×10^9 rads) did not affect the thermal stability of Dowex 50-X8 in hydrochloric or nitric acid. Gamma irradiation (about 10^8 rads) of Dowex 1-X4 in nitric acid lowers the exotherm temperature approximately 50 degrees Celsius. The effect of alpha radiation on Dowex 1-X4 is about the same as that of gamma radiation, whereas alpha particles are more damaging than gamma rays to Dowex 50-X8. Contacting the unirradiated resins with hydrochloric or nitric acid did not cause degradation, but both resins reacted with nitric acid at elevated temperatures, which created a thermal instability.

INTRODUCTION

In recent years there have been several accidents involving ion exchange resins in the nuclear industry. Causes and descriptions of these accidents have been summarized previously.^{1,2} The common factors in these occurrences have been resin in the nitrate form, a high concentration of nitric acid, and a source of heat. The most recent accident occurred in August 1976 at Hanford and it was the first accident involving a cation exchange resin. A high concentration of nitric acid was present even though the resin was obviously not in the nitrate form. It is now well established that ion exchange resins can react with nitric acid (at elevated temperatures) and other oxidizing agents, and that special precautions must be taken to prevent this occurrence.

More understanding of reactions of ion exchange resins would be helpful in avoiding future accidents. The effects of radiation and acids on the thermal

stability of resins are of interest as is the cause of thermal excursions. Both subject areas have been examined in this study. The resins examined were Dowex 50-X8 (cation exchange) and Dowex 1-X4 (anion exchange). The most convenient measure of thermal stability and the one used in this study was differential thermal analysis (DTA). Infrared spectroscopy also was used for analysis in an effort to relate the structural changes in the resin to the thermal stability.

EXPERIMENTAL

The instrument used for analysis was the DuPont 900 Differential Thermal Analyzer. The procedure followed was to place a few milligrams of resin in a capillary tube and heat it at a constant, predetermined rate. Any exotherms or endotherms occurring in the sample were recorded on a baseline of increasing temperature. These temperature excursions were usually small. Their sizes (areas) were not determined by integration; the relative sizes were given as small, medium, and large. The plotted charts are not included in this report. The results reported are temperatures at the onset of exotherms and endotherms and the maxima or minima of the peaks.

The analyses were made in open and in closed capillary tubes. Open tubes allowed the resin to combine with air; the sealed capillaries did not. It has not been determined whether results from the open or closed tubes are more applicable to large-scale column operation. Samples were prepared for infrared analyses by grinding resin in a mortar and pestle and then forming resin-potassium bromide pellets.

RESULTS

Effect of Gamma Radiation

Resins were irradiated with gamma rays at a dose rate of 0.65 megarads per hour and were analyzed

by DTA and infrared spectroscopy. The irradiated resins were also contacted with nitric and nitrous acids to roughly measure their reactivity. The resins were prepared by washing in acids and filtering through a sintered disc. The following three samples were irradiated:

Dowex 50-X8 in 0.5N hydrochloric acid (HCl)

Dowex 50-X8 in 3N nitric acid (HNO_3)

Dowex 1-X4 in 7N HNO_3

Differential Thermal Analyses

Almost all of the trials were made in duplicate with results as shown in Tables 1 and 2.

The endotherms that appeared in the open capillary measurements were attributed to the liquid-vapor transition of the aqueous phase. These disappear with lengthy irradiation because of the loss of water. Endotherms are relatively unimportant because it is the exotherms that measure the thermal instability of the material. Results from the open and closed capillary trials show that irradiation in the 10^8 -rad region produces lower exotherm temperatures, which indicates lower thermal stability. Previous experiments have shown that this 10^8 -rad dose region is where the resin is degraded as indicated by loss of exchange capacity.³ Further irradiation into the 10^9 -rad region appeared to restore the thermal stability to its original state. This reversible effect, however, was considered to be spurious and was probably caused by evaporation of the nitric acid solution. If the irradiated resin was wet with 7N HNO_3 before analysis (see the last experiment in Table 1), the exotherm temperature was lowered again in the closed capillary. The endotherm also reappeared in the open capillary.

The results in Table 2 show that Dowex 50-X8 is very stable in nitric acid and in hydrochloric acid and that radiation does not affect its thermal stability. The endotherms can be neglected because they are attributed to the loss of water.

Infrared Analyses

There was no change in the infrared spectra of Dowex 50-X8 irradiated in HCl and HNO_3 compared to the unirradiated resin. This corroborates the

results of the differential thermal analysis. There were changes, however, in the spectra of Dowex 1-X4 irradiated in HNO_3 . These changes are shown in Figure 1. The middle spectrum (1.6×10^8 rads) shows several significant differences. There is a broad band from 1800 to 1670 cm^{-1} , actually a shoulder on the 1630 cm^{-1} absorption, and another absorption from 1220 to 1150 cm^{-1} . Both absorptions indicate that oxidation occurred. Two absorptions that appear at 1600 to 1500 cm^{-1} and 1380 to 1230 cm^{-1} are in the regions of nitrogen-oxygen stretching and may have been caused by formation of nitration species.

These differences increase with irradiation and the spectrum changes until, as in the top spectrum, it is hardly recognizable as the spectrum of Dowex 1-X4 resin. Unfortunately, there are no distinct peaks that lend themselves to a direct measure of the amount of degradation. It seems difficult to relate these results to those obtained by differential thermal analysis (DTA) and therefore difficult to use infrared peaks as a simple measure of thermal stability of the resin.

Reactivity With Acids

The irradiated resins were immersed in acids to determine the relative reactivity of irradiated resin. The acids were concentrated HNO_3 and 7N HNO_3 plus NaNO_2 (added to produce some HNO_2). The reactivity was examined visually.

The unirradiated resins did not react with concentrated HNO_3 for long periods at room temperature. Resins irradiated 10 and 33 days did not appear to react either immediately or after standing a few days. Resins irradiated 75 and 104 days did not appear to react immediately, but they were all completely "dissolved" in a few days. The reaction was faster in concentrated HNO_3 , which indicated that HNO_3 concentration is a more important factor than the nitrous acid. It is difficult to explain how an organic polymer (resin) can form a single phase without an overt reaction. Perhaps radiation produces sufficient hydrophilic groups (COOH , C=O , OH) to solubilize the polymer.

Effect of Alpha Radiation

Dowex 50-X8 loaded with americium (Am) and Dowex 1-X4 loaded with plutonium (Pu) were

TABLE 1. Dowex 1-X4 in 7N Nitric Acid

	<u>Closed Capillary*</u>	<u>Open Capillary*</u>
Unirradiated (nitrate form) as-received resin	Small exotherm at 310, 310 (384, 387)	Small exotherm at 265, 261 (320, 318)
Unirradiated wet with 7N HNO ₃	Medium exotherm at 182, 182 (219, 225) Very small exotherm at 345 (383)	Small exotherm at 185, 184 (214, 218) Small exotherm at 264, 263 (294, 293) Small endotherm at 123, 121 (138, 135)
Irradiated 10 days 1.6×10^8 rads	Medium exotherm at 150, 155 (210, 210) Medium exotherm at 335, 327 (356, ---)	Small exotherm at 140, 145 (181, 187) Small exotherm at 255, 257 (325, 325) Small endotherm at 122, 125 (135, 140)
Irradiated 33 days 5.1×10^8 rads	Small exotherm at 130, 131 (159, 162) Medium exotherm at 323 (361)	Small exotherm at 142, 143 (168, 168) Small exotherm at 305, 303 (330, 329)
Irradiated 75 days 1.2×10^9 rads	Small exotherm at 310, 312 (348, 360)	Small exotherm at 307, 307 (348, 345)
Irradiated 104 days 1.6×10^9 rads	Small exotherm at 320, 320 (357, 355)	Small exotherm at 308, 314 (345, 351)
Irradiated 104 days wet with 7N HNO ₃	Medium exotherm at 122 (149) Small exotherm at 195 (222)	Small exotherm at 293 (334) Very large endotherm at 114 (135)

*Numbers outside the parentheses are temperatures (°C) at the beginning of a thermal excursion. Numbers (temperatures) inside the parentheses are the maxima or minima of the peaks.

TABLE 2. Dowex 50-X8 in 0.5N Hydrochloric Acid and 3N Nitric Acid

	<u>Closed Capillary*</u>	<u>Open Capillary*</u>
Unirradiated	No exotherm to 450	Large endotherm at 110, 110 (130, 130)
Irradiated 10 days 1.6×10^8 rads	No exotherm to 450	Large endotherm at 110, 110 (130, 130)
Irradiated 33 days 5.1×10^8 rads	No exotherm to 450	Large endotherm at 115, 115 (128, 128)
Irradiated 75 days 1.2×10^9 rads	No exotherm to 450	Small endotherm HCl-142, 140 (160, 155); HNO ₃ -117, 120 (137, 134)
Irradiated 104 days 1.6×10^9 rads	No exotherm to 450	Very small endotherm HCl-138, 142 (156, 158); HNO ₃ -147, 140 (167, 165)

*Numbers outside the parentheses are temperatures (°C) at the beginning of a thermal excursion. Numbers (temperatures) inside the parentheses are the maxima or minima of the peaks.

FIGURE 1. Infrared Spectra of Dowex 1-X4

stored and periodically analyzed for thermal stability by DTA. The Am-Dowex 50-X8 sample was prepared by mixing 10 cm^3 of resin with 1 g Am in 2N HCl and filtering the mixture through a stainless steel screen. The loading was calculated to be 0.75 g Am per 10 cm^3 resin using the analyzed Am content of the effluent. The dose rate at this

loading was 4.4×10^6 rads per hour. The Pu-Dowex 1-X4 sample was prepared by mixing 10 cm^3 of resin with 1.7 g Pu in 7N HNO_3 and filtering. The loading was calculated to be 1 g Pu per 10 cm^3 resin using the analyzed Pu content of the effluent. The dose rate at this loading was 1.1×10^5 rads per hour. The DTA results are shown in Tables 3 and 4.

TABLE 3. Americium on Dowex 50-X8

	<u>Closed Capillary*</u>	<u>Open Capillary*</u>
Unirradiated	No exotherm to 450	Large endotherm at 110 (130)
Irradiated 7 days 7.4×10^8 rads	No exotherm to 450	Large endotherm at 110 (130)
Irradiated 23 days 2.4×10^9 rads	Small exotherm 278, 267 (305, 285)	Large endotherm at 105, 105 (130, 130)
Irradiated 42 days 4.4×10^9 rads	Small exotherm 265, 239 (319, --)	Large endotherm at 105, 105 (129, 129)
Irradiated 70 days 7.4×10^9 rads	No exotherm to 450	Large endotherm 98, 98 (120, 120)

*Numbers outside the parentheses are temperatures ($^{\circ}\text{C}$) at the beginning of a thermal excursion. Numbers (temperatures) inside the parentheses are the maxima or minima of the peaks.

TABLE 4. Plutonium on Dowex 1-X4

	<u>Closed Capillary*</u>	<u>Open Capillary *</u>
Unirradiated	Small exotherm at 310, 310 (384, 387)	Small exotherm at 265, 261 (320, 318)
Immediately after Pu addition (wet with HNO_3)	Small exotherm at 195, 200 (219, 233) Medium exotherm at 279 (354)	Large endotherm at 90, 90 (107, 108) Small exotherm at 185, 175 (219, 222) Large exotherm at 250, 255 (270, 290)
Irradiated 38 days 1.0×10^8 rads	Large exotherm at 188, 183 (212, 227) Medium exotherm at 204 (231) Medium exotherm at 250, 268 (268, 294) Small exotherm at 271 (295)	Large exotherm at 185, 185 (208, 204)
Irradiated 86 days 2.3×10^8 rads	Large exotherm at 178, 157 (208, 197) Medium exotherm at 255, 244 (278, 278)	Large exotherm at 185, 183 (207, 206) Medium exotherm at 246, 252 (270, 276)
Irradiated 135 days 3.7×10^8 rads	Large exotherm at 155, 170 (194, 204) Medium exotherm at 247, 245 (280, 270)	Very large exotherm at 170, 168 (204, 203) Small exotherm at 247 (270)
Irradiated 135 days, then eluted with 0.35N HNO_3	Small exotherm at 135, 135 (166, 160) Small exotherm at 264, 245 (297, 285)	Small exotherm at 153, 145 (193, 183) Medium exotherm at 234, 230 (279, 278) Very large endotherm at 90, 91 (114, 110)

*Numbers outside the parentheses are temperatures ($^{\circ}\text{C}$) at the beginning of a thermal excursion. Numbers (temperatures) inside the parentheses are the maxima or minima of the peaks.

Dowex 50-X8 showed greater thermal stability than Dowex 1-X4 before and after irradiation. A small exotherm was created after a large dose of 2×10^9 rads. Further contact to a dose of 7.4×10^9 rads

restored the thermal stability so that no thermal excursions were observed up to 450°C . This effect would probably be attributed to the evaporative loss of acid solution if it were not for the occurrence

TABLE 5. Resins in Acid Solutions

	<u>Closed Capillary*</u>	<u>Open Capillary*</u>
<u>Dowex 50-X8 in 0.5N HCl</u>		
Original sample	No exotherm to 450	Large endotherm at 110, 110 (130, 130)
75 days storage	No exotherm to 450	Large endotherm at 112, 112 (126, 125)
136 days storage	No exotherm to 450	Large endotherm at 115, 113 (131, 126)
<u>Dowex 50-X8 in 1N HNO₃</u>		
Original sample	Very small exotherm at 192 (212)	Large endotherm at 110, 110 (130, 130)
75 days storage	Very small exotherm at 195, 192 (208, 207)	Large endotherm at 111, 113 (124, 126)
136 days storage	Small exotherm at 188, 185 (207, 207) Small exotherm at 295, 280 (318, 323)	Large endotherm at 109, 115 (129, 130)
<u>Dowex 1-X4 in 7N HNO₃</u>		
Original sample	Medium exotherm at 182, 182 (219, 225) Very small exotherm at 345 (383)	Medium endotherm at 123, 121 (138, 135) Small exotherm at 185, 184 (214, 218) Small exotherm at 264, 263 (294, 293)
75 days storage	Small exotherm at 202, 202 (228, 232)	Large endotherm at 117, 115 (127, 128) Small exotherm at 189, 190 (210, 212) Small exotherm at 267, 263 (336, 326)
136 days storage	Medium exotherm at 200, 197 (250, 231) Small exotherm at 345 (378)	Large endotherm at 113, 113 (128, 129) Small exotherm at 187, 188 (212, 209) Small exotherm at 263, 260 (320, 323)

*Numbers outside the parentheses are temperatures (°C) at the beginning of a thermal excursion. Numbers (temperatures) inside the parentheses are the maxima or minima of the peaks.

of a large endotherm (caused by moisture) in the open capillary trial.

The addition of plutonium to Dowex 1-X4 lowered the thermal stability because of the presence of 7N HNO₃. Extending the contact time (radiation dose) continued to lower the exotherm temperatures. The change in thermal stability with dose approximated that observed with the gamma irradiation. After 135 days of contact time, the plutonium was eluted from the resin with 0.35N HNO₃. Analysis of the resin by DTA indicated a rewetting of the resin because of the appearance of a large endotherm in the open capillary trials. The exotherm temperatures were lowered even further—probably the result of a new nitric acid supply.

Effect of Acids

Dowex 1-X4 and Dowex 50-X8 were contacted with acids for long periods and were analyzed for

thermal stability by DTA and infrared analyses. The following acid concentrations were used because they are commonly used loading media.

- Sample #1 6 g Dowex 50-X8 + 3 cm³ 0.5N HCl
 Sample #2 8 g Dowex 50-X8 + 4 cm³ 1N HNO₃
 Sample #3 6 g Dowex 1-X4 + 3 cm³ 7N HNO₃

Contact times up to 136 days produced no changes in the infrared spectra of either resin. This indicated that the acids did not chemically degrade the resins.

The DTA results are shown in Table 5. They show that the thermal stability of Dowex 50-X8 is not affected by 0.5N HCl, but that this stability is lowered by 1N HNO₃. The thermal stability of Dowex 1-X4 (nitrate form) is also lowered by 7N HNO₃; however, increasing the contact time of both resins in nitric acid does not decrease the exotherm temperatures further. Nitric acid reacts with these

resins at elevated temperatures but does not degrade the resins appreciably at room temperature. Actually, nitric acid does react slowly with Dowex 1-X4 over long periods liberating carbon dioxide and nitrous oxide.

Nature of Thermal Degradation

A number of experiments were carried out in which resin was heated in the Differential Thermal Analyzer and removed at various temperatures (relevant to exotherms) for infrared analysis.

Dowex 1-X4, Nitrate Form

The anticipated start of an exotherm is 260 to 300 °C. Resin was heated to 250 °C in open and closed capillaries and was then analyzed. The infrared spectra showed only the following, minor changes compared to the spectrum of the original (unheated) resin: a decrease in water, an increase in nitrate ion, and a slight oxidation (shoulder absorption occurring at 1690 cm^{-1}).

Resin was heated to 450 °C in an open capillary and analyzed. A small exotherm had ended at 354 °C. The resin was a charred black color. The infrared spectrum was no longer recognizable as that of a styrene-divinylbenzene copolymer (Dowex 1-X4). Some features of the spectrum were a complete loss of water and nitrate ion absorptions, appearance of a strong absorption at 825 cm^{-1} (indicative of a parasubstituted benzene), and appearance of an absorption at 220 cm^{-1} (indicative of the cyanide group). Both alkyl and aryl absorption bands remained in the spectrum, and a relatively intense absorption at 1700 cm^{-1} (characteristic of oxidized organics) was present.

Dowex 1-X4 Wet with 7N Nitric Acid

There was a large exotherm in the sealed capillaries starting at 191 °C and reaching a maximum at 224 °C. Resin was heated to 200 °C and analyzed. The infrared spectrum resembled that of the Dowex 1-X4 nitrate-form resin except for loss of water, appearance of an oxidation ($\text{C}=\text{O}$) band at 1700 cm^{-1} , and changes in relative intensities of several absorptions in the 1100- to 650- cm^{-1} region.

Resin was heated to 233 °C and analyzed. The spectrum had additional changes in the relative intensities of absorption bands in the 1100- to 650- cm^{-1} region. Two new bands appeared and one band disappeared between 800 and 650 cm^{-1} . The oxidation band at 1708 cm^{-1} had become more intense, and the major nitrate-ion absorbance, although still relatively intense, had its maximum shifted from 1380 to 1350 cm^{-1} . It appeared that the organic species were being degraded by oxidation while the ionic nitrate species were changing.

Irradiated Dowex 1-X4 Wet with 7N Nitric Acid

Dowex 1-X4, gamma irradiated for 104 days (1.6×10^9 rads), was wet with 7N HNO_3 and heated in sealed capillaries. An exothermic reaction started at 140 °C and ended at 195 °C. Resin was heated to 150 °C and analyzed. The infrared spectrum was similar to that of the irradiated resin except for (1) the appearance of an oxidation band at 1710 cm^{-1} , (2) broadening of the OH stretching band in the 3600 to 2400 cm^{-1} region, and (3) loss of water. The nitrate ion absorptions (1389 and 821 cm^{-1}) were still present. Resin was heated to 200 °C and analyzed. The infrared spectrum bore little resemblance to the spectrum of the irradiated resin. The principal changes included the following: loss of water, an intense broad absorption band at about 1700 cm^{-1} [indicative of oxidation ($\text{C}=\text{O}$)], broadening of the OH stretching band in the 3600- to 2400- cm^{-1} region (indicative of carboxylic acids), appearance of an absorption band at 1100 cm^{-1} [indicative of oxidation ($\text{C}-\text{O}$)], and complete disappearance of absorptions due to nitrate ion. Disappearance of the nitrate ion and extensive oxidation of the resin matrix between 150 and 200 °C suggested that the first DTA exotherm observed in the closed capillary trials corresponds to nitrate ion oxidation of organic species.

Product Analyses by Mass Spectrometry

Irradiated resin was analyzed by mass spectrometry to determine if there were any intermediate molecular weight products of irradiation. It has been previously established that radiation produces trimethylamine in Dowex 1-X4, sulfate ion in Dowex 50-X8, and small amounts of hydrogen in

both resins.³ No significant amounts of other products were found upon heating the resin to 400 °C, thus indicating that except for the loss of exchange groups, there is no fragmentation of the reacting resin polymer.

CONCLUSIONS

1. Differential thermal analysis was established as an effective measurement of the thermal stability of resins. Closed capillary results were judged to be somewhat more useful than open capillary results because small exotherms are more easily detected, temperatures could be raised to higher levels, and endotherms caused by aqueous transitions were absent in the closed capillary trials.
2. Infrared analysis revealed in a general way the oxidation and nitration occurring in the resin. The method is not suitable, however, as a quantitative measure of the amount of degradation or the change in thermal stability.
3. Gamma irradiation (up to 1.6×10^9 rads) does not affect the thermal stability of Dowex 50-X8 in hydrochloric or nitric acid, but it does lower the thermal stability of Dowex 1-X4 in nitric acid. Irradiation of Dowex 1-X4 in the range of 10^8 rads lowers the exotherm temperature approximately 50 °C.

4. Alpha irradiation of Dowex 50-X8 produces a small thermal instability after a large dose (greater than 10^9 rads). Alpha radiation lowers the thermal stability of Dowex 1-X4. The change in stability with dose is approximately the same as that observed with gamma radiation.
5. Contacting the resins with hydrochloric or nitric acid does not cause degradation, but both resins react with nitric acid at elevated temperatures—thereby creating a thermal instability.
6. The first exotherm in a DTA trial is a measure of thermal instability and is caused by the oxidation of the organic polymer by nitrate ion.

REFERENCES

1. F. W. Miles. "Ion-Exchange-Resin System Failures in Processing Actinides." *Nuclear Safety*, Vol. 9, Page 5. September 1968.
2. C. E. Baldwin and C. C. Perry. Rocky Flats Plant. Private communication.
3. A. R. Kazanjian and D. R. Horrell. *Radiation Effects on Ion-Exchange Resins Part II: Gamma Irradiation of Dowex 1*. RFP-2354. Dow Chemical U.S.A., Rocky Flats Division. February 27, 1975.