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Thermal Analysis and Chemical Compatibility of Reillex^{TM} HPQ Resin and Nitric Acid with Ethylene Glycol

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Recent processing activities at the Savannah River Site (SRS) involved purifying neptunium solutions using anion exchange (Reillex^{TM} HPQ resin) columns. The addition of cooling coils and the use of antifreeze in a chemical make-up tank led to an evaluation of chemical incompatibility accident scenarios between the antifreeze agent, ethylene glycol, and other chemicals used in the anion exchange operation, as well as a risk of possible fouling of the resin with ethylene glycol. Therefore, calorimetric and spectroscopy work was conducted to evaluate reactions and thermal hazards.

Ethylene glycol oxidizes readily in 6 M nitric acid (at 60°C) and in 40wt% potassium permanganate. The magnitude of the maximum pressure rate data indicates that the reactions are non-deflagrating and non-explosive. In the presence of reductants such as hydrazine and ferrous sulfamate (both at 0.06 M), ethylene glycol did not oxidize in permanganate. Ethylene glycol reversibly sorbed on Reillex^{TM} HPQ resin to a limited extent in the time period observed. Therefore, ethylene glycol will not significantly affect the physical shape of the Reillex^{TM} HPQ resin beads.

Keywords chemical compatibility; ethylene glycol; ferrous sulfamate; hydrazine

BACKGROUND

Recent processing activities in the HB-Line facility at the Savannah River Site involved neptunium purification by anion exchange followed by precipitation as neptunium oxalate and calcination to generate NpO_2. The purified NpO_2 is slated for use as a precursor for the production of ^{238}Pu, a heat-generating isotope used to power deep space missions.

Wash solutions for washing the ion exchange column are prepared in a tank that contains a set of coils for cooling the solutions. The chemicals for making the wash and stripping solutions are stored nearby the tank and include aqueous solutions of 64 wt% nitric acid, 40 wt% sodium permanganate, 30 wt% hydrazine mononitrate, 30 wt% ferrous sulfamate, 30 wt% sodium nitrite, 0.15 M potassium fluoride, and oxalic acid solids. Normally, to make the wash solution, chemicals are retrieved and mixed in a tank to generate a make-up solution. As the solution is compounded its temperature rises (due to heat of mixing) to 40°C. Then the process uses a coil (with commercial antifreeze as the cooling agent) to bring the solution temperature to 22°C after which hydrazine and ferrous sulfamate may be added. To limit the chemical reaction between nitric acid and ferrous sulfamate, the solution must be at room temperature. The make-up solution is compounded to 6.4 M nitric acid, 0.05 M ferrous sulfamate, and 0.05 M hydrazine mononitrate. The solution is then used to wash the anion exchange columns packed with Reillex^{TM} HPQ which is used to purify the solution containing ^{237}Np. The washed bed is rinsed (upflow) with an 8 M nitric acid decontamination solution followed by a downflow elution with dilute nitric acid-hydrazine solution to remove the ^{237}Np.

A safety basis evaluation of the process assessed the accident scenarios of mixing antifreeze (resulting from a leak in the coils) with the cold feed chemicals, with the make-up solution, with the ion exchange resin, and with 64 wt% nitric acid. To address the different chemical incompatability scenarios, a literature survey and experimental studies to evaluate the possible reactions were conducted to evaluate and quantify the energy and byproducts generated. The safety basis evaluation acknowledged that nitric acid is known to oxidize organics (1–4). Depending on the starting organic chemical, the oxidation reaction may yield energetic materials (5–7).

The researchers envisioned two accidental leaks of cooling agent (30 wt% ethylene glycol) during the preparation of the wash solution. One scenario considers the cooling agent leaking into the wash solution tank before 14 M nitric acid is added to water to make the concentration target of 6.4 M nitric acid. A second scenario considers the cooling agent leaking into the wash solution tank after the addition of hydrazine and ferrous sulfamate to 6.4 M nitric acid. In both scenarios, researchers conservatively assume that all the cooling agent leaks into the full tank yielding a volumetric ratio of 3.4:1 wash solution to the

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cooling agent solution (calculated from the volume of antifreeze and the volume of wash tank).

- To determine if an accidental leak of ethylene glycol into the wash solution during preparation creates a hazardous scenario or an energetic solution, the authors assessed the following two mixing tests:
  - i. Ethylene glycol with 6.4 M nitric acid, and
  - ii. ethylene glycol with 6.4 M nitric acid, hydrazine, and ferrous sulfamate.

  For chemicals not associated with the wash solution that may leak into the common sump, researchers examined the chemical compatibilities in the following series of mixing tests:
  - i. 30 wt% ethylene glycol and 40 wt% potassium permanganate solution, and
  - ii. 30 wt% ethylene glycol and 30 wt% sodium nitrite solution.
- To determine if ethylene glycol affects the performance of the Reillex^{TM} HPQ resin, researchers conducted a series of mixing tests between the resin, ethylene glycol, and wash tank solutions. From these tests, researchers determined the impact of 30 wt% ethylene glycol on resin size and shape (e.g., swelling).

EXPERIMENTAL

Researchers needed to decide whether to use a commercial antifreeze solution or a pure solution of ethylene glycol to evaluate the reactivity of the 30 wt% antifreeze solution used as the cooling agent. A typical commercial antifreeze solution contains between 45 to 50 wt% ethylene glycol, 3 wt% diethylene glycol, and 3 wt% of either sodium 2-ethylhexanoic acid, potassium 2-ethylhexanoate, and potassium phosphate dibasic. A literature survey of the aliphatic organic acids used in antifreeze revealed that they are inert in nitric acid since they are the byproduct of the oxidation reaction between glycol or alcohols and nitric acid (8). Diethylene glycol is readily oxidized to diglycolic acid (9) and, therefore, the energetic and gas production from this reaction is similar to the oxidation of ethylene glycol in nitric acid. Based on this survey, researchers decided to use pure ethylene glycol to make the 30 wt% ethylene glycol solution as a more conservative solution than antifreeze to study the oxidation reactions between the cooling agent and oxidizers like nitric acid and potassium permanganate. Limited confirmatory tests, using commercial antifreeze, were included to validate this assumption.

Thermal reactivity tests occurred in multiple instruments, including the Advanced Reactive System Screening Tool (ARSST^{TM}) from Fauske & Associates LLC. The ARSST is a nearly adiabatic 350-mL sealed calorimeter in which an open (i.e., vented), well-insulated, 12-mL glass test cell is heated at a programmed rate while the sample temperature and the calorimeter pressure data are recorded. The ARSST is equipped with a high-pressure syringe, so that the system can be sealed before reactive chemicals are mixed. The high pressure syringe attachment was used for several tests in this study. For the ARSST tests, the calorimeter was pressurized with 10 to 50 psig of nitrogen gas prior to testing.

The adiabatic thermal tests used an Accelerated Rate Calorimeter (ARC) from TIAX LLC (ARC2000^{TM}). The ARC is an adiabatic 10-mL sealed vessel connected to a pressure sensor. The vessel is heated at a programmed rate and if the sample self-heats (for example, undergoes exothermic reactions such as oxidation), the heater will input power to the vessel such that the temperature of the surroundings (vessel jacket) matches the temperature of the sample. No heat escapes from the sample to the calorimeter during the sample reaction. All ARC tests were pressurized with air before heating the sample to suppress boiling of the solution. There is no significant effect of the initial pressure on the thermal reactivity of the solutions.

To test an ethylene glycol leak to a tank containing acid, researchers performed initial ARSST and ARC tests. These tests mixed 30 wt% ethylene glycol, as used in the HB-Line, with 6–7 M nitric acid at a volumetric ratio of 3.4:1 acid to glycol. The HB-Line uses 64 wt% nitric acid to form a 6.4 M nitric acid wash solution in the tank of interest. In the first test, researchers added 70 wt% nitric acid to 30 wt% ethylene glycol and water to achieve ~7 M HNO_3 at a volumetric ratio of ~3.4:1 acid to glycol with no external heat added. In the second test, researchers heated the resulting acid/ethylene glycol mixture to 95°C at a programmed rate of 3°C/min. Finally, researchers prepared a mixture of 6.4 M nitric acid and 30 wt% ethylene glycol at a 3.1:1 volumetric ratio. Researchers sealed this mixture inside the ARSST, heated to 40°C, and held at that temperature for 17 hours.

To test an ethylene glycol leak into the reductive wash solution used in HB-Line, researchers prepared a solution of 6.4 M nitric acid/0.05 M hydrazine/0.05 M ferrous sulfamate mixed with 30 wt% ethylene glycol at a 3.4:1 volumetric ratio. Researchers sealed this mixture inside the ARSST or ARC, then heated and kept at 90°C for two hours.

Finally, researchers performed ARSST tests to determine the effect of multiple spills in the cold chemical portion of the HB-Line facility. In one test, researchers added 7.22 grams of 30 wt% sodium nitrite to 7.16 grams of 30 wt% ethylene glycol. After about 15 minutes, researchers heated the mixture 50°C while monitoring the temperature and pressure of the system.

In a final test, researchers added 8.76 grams of 40 wt% sodium permanganate to 3.90 grams of 30 wt% ethylene glycol. For both tests, researchers sealed the ARSST initially with an ethylene glycol solution in the test cell. Then they used a high-pressure syringe to add either nitrite or the permanganate solution so that immediate temperature and pressure changes could be recorded.

Some tests used a Perkin-Elmer PE-7 Differential Scanning Calorimeter (DSC). DSC tests used sealed stainless steel pans with programmed heating to 90°C. The DSC records the amount of heat a sample gives off or takes in during a reaction. Testing included measuring the heat of reaction between 7 M nitric acid and 30 wt% ethylene glycol at a 3.4 to 1 volumetric ratio.

For one ARC test, researchers prepared a reductive wash solution with ethylene glycol. The solution contained 6.2 M HNO_3/0.06 M ferrous sulfamate/0.05 M hydrazine. researchers tested a volumetric ratio of 3:1 wash solution to 30 wt% ethylene glycol.

To test the effect of ethylene glycol on the Reillex^{TM} HPQ resin (lot Number 80302 MA received in December 1998), researchers contacted weighed amounts of resin (0.278 grams on a dried basis since Reillex^{TM} HPQ resin is 55 wt% free water as determined by a thermogravimetry measurement) with measured volumes (10 mL) of wash solution with (at a volume ratio of 3.4:1) and without 30 wt% ethylene glycol for 48 hours. A similar contact test between 0.21 grams (on a dried basis) of the resin with 5 mL of 30 wt% ethylene glycol was conducted for 24 hours.

All thermal tests and resin contacts with ethylene glycol were conducted only once.

RESULTS

Ethylene Glycol and Nitric Acid (6.4 M) Reaction

In the first acid/glycol test performed in the ARSST, after the addition of 15.8 M (70 wt%) nitric acid to a mixture of ethylene glycol and water, a sharp temperature increase from 22 to 35°C occurred. Only a slight pressure increase occurred (from 18 to 19 psig), which is attributed to a pressure increase from the syringe addition and to the temperature increase. After the initial temperature rise, no T or P increase occurred for 12 minutes. The mixture began to gradually cool. The final mixture contained a 3.4:1 volumetric ratio of ~7 M HNO_3 to 30 wt% ethylene glycol solution.

After modest cooling, researchers heated the resulting acid/glycol mixture to 95°C at a programmed rate of 3°C/min. Figure 1 shows the results of this test. No reaction occurred initially and during heating until the mixture reached 95°C, which is well above the expected process temperature of 40°C. At 95°C, an energetic reaction occurred. Results of tests with the same solution composition in the ARC are shown in Fig. 2. Inspection of Fig. 2 clearly shows an exothermic (oxidation reaction) between ethylene glycol and 6.4 M nitric acid occurred at 60°C. At this temperature, a rapid increase in pressure (~158 atm or 1033 kPa) and temperature (~200°C) is noted. The reaction initiation temperature was lowered in the ARC due to the adiabatic conditions, smaller free volume above the solution, and the use of air as the starting gas (nitrogen was used in the ARSST tests which is an inert gas).

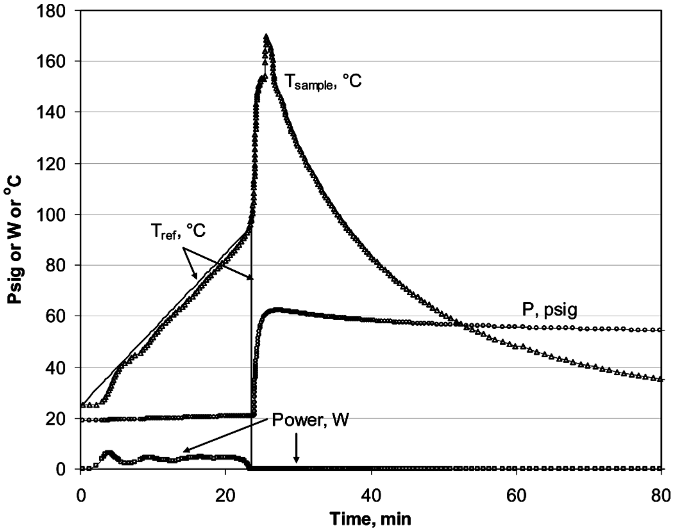


FIG. 1. Mixture of 30 wt% ethylene glycol and ~7 M HNO_3 heated at 3°C/min to 95°C.

To evaluate process conditions (where the maximum observed working temperature is approximately 40°C), researchers performed a third test mixing 6.4 M HNO_3 with 30 wt% ethylene glycol at a 3.1:1 volumetric ratio and heated to 40°C for an extended time. The results indicate that no significant pressure increase was observed over the course of the extended test at 40°C. Therefore, the extent of reaction over the 17 hours period was not significant.

Commercial Antifreeze and Nitric Acid Reaction

Researchers tested the compatibility of a commercial antifreeze solution from Texaco Extended Life Coolant (Cat$^{®}$ ELC^{TM})—this antifreeze is typical of current inventory—with nitric acid. Researchers added 70 wt% HNO_3 to a 30 wt% antifreeze/coolant to obtain 6 M HNO_3 at a 3:1 acid to coolant volumetric ratio, and no P increase occurred at ambient temperature, indicating no immediate reaction. A second test with 30 wt% coolant, mixed 7.3 M HNO_3 and the antifreeze to achieve a 3.5:1 acid to coolant ratio. Heating the mixture to 40°C two times did not cause a reaction. When the same mixture was further heated at 3°C/min, a reaction occurred, initiating at 84–85°C, as shown in Fig. 3. In comparison to the

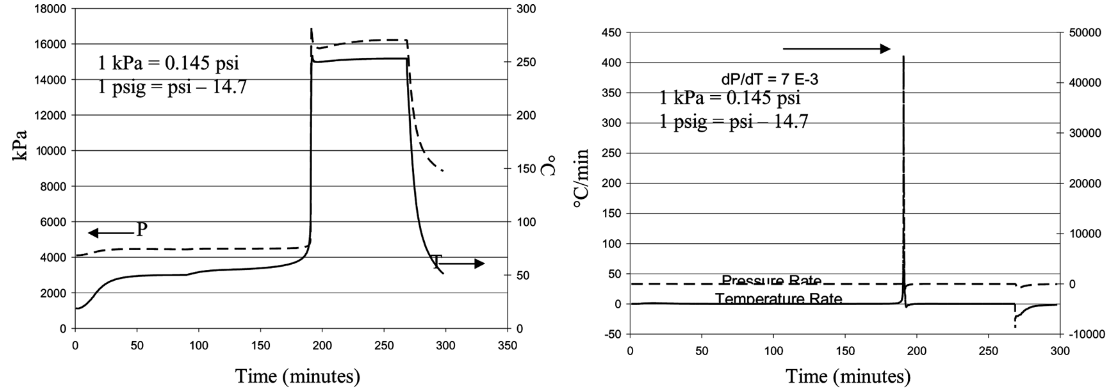


FIG. 2. Mixture of 30 wt% ethylene glycol and 6.4 M HNO_3 heated at 3°C/min to 95°C at a volume ratio of 1 to 3.4. The onset reaction temperature measured is 60°C. Figure on the right shows the first derivative with respect to time of the pressure and temperature rate of the data the left figure.

results shown in Fig. 1, the reaction initiation temperature in Fig. 3 is about 10°C lower, and the magnitude of the reaction is also lower, as indicated by smaller temperature and pressure increases.

Researchers also tested the thermal stability of this commercial antifreeze with 6.4 M nitric acid in an ARC unit. The oxidation of the antifreeze started around 60°C, comparable to the earlier ARC test for the ethylene glycol mixture (i.e., Fig. 2). At this temperature a rapid increase in pressure (102 atms or 10,335 kPa) and temperature (165°C) is noted. The pressure rise rate measured (0.002 MPsi/min) is comparable with the pressure rise rate for the comparable ARC test with ethylene glycol. Also note that after the temperature reached 220°C (or after 248 minutes) a leak developed in the vessel and correspondingly the pressure decreased. This leak did not affect the slow temperature rise seen after the initial oxidation, nor did it affect the initial temperature and pressure rise associated with the initial oxidation reaction.

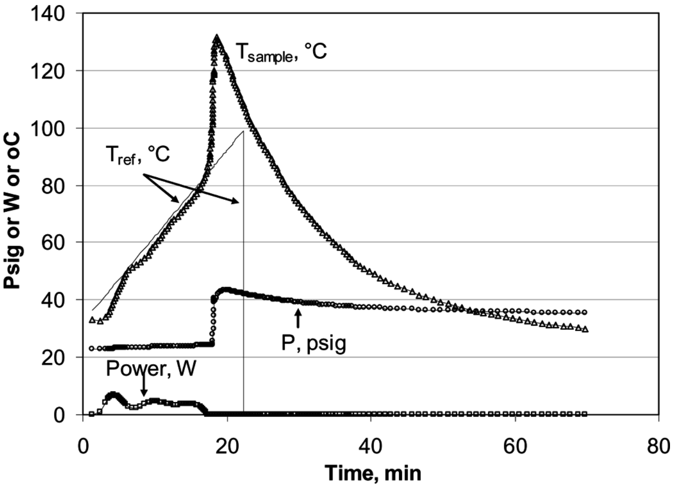


FIG. 3. Mixture of commercial antifreeze and ~7 M HNO_3 heated at 3°C/min to 95°C.

Ethylene Glycol and Wash Solution (HNO_3, N_2H_2, $Fe(NH_2SO_3)_2$) Reaction

To test the reactivity of ethylene glycol with the combined components of a reductive wash solution, researchers performed two additional tests. The targeted solution concentrations for the wash solution were 6.4 M HNO_3/0.05 M hydrazine/0.05 M ferrous sulfamate. However, in making the small portions needed for this test, researchers prepared 6.2 M HNO_3/0.06 M hydrazine/0.06 M ferrous sulfamate. This wash solution was mixed with 30 wt% ethylene glycol at a 3.4:1 volumetric ratio, heated, and held at 90°C for two hours. No significant reaction occurred. After cooling, the solution was re-heated to 50°C and held at that temperature for 17 hours. Again, no exothermic reaction or gas generation occurred. Researchers heated a solution of similar composition in the ARC and detected no reaction during the adiabatic heating. The drop in pressure between 75 and 90°C is possibly due to a leak or (most likely) the formation of reducing species that reacted with oxygen in the air causing the overall pressure to drop. The addition of 30 wt% ethylene glycol lowers the nitric acid concentration from 6.4 to 4.9 M. This concentration change does not significantly change the reactivity of 6.4 M nitric with ethylene glycol. The reactivity of lower volumes of 30 wt% ethylene glycol in 6.4 M nitric acid (less than 3.4 times the volume of 6.4 M nitric acid) is bounded by the reactivity measured in this study where all the cooling solution is leaked.

Ethylene Glycol Reaction with Sodium Nitrite and Potassium Permanganate

In the test adding 30 wt% sodium nitrite solution to 30 wt% ethylene glycol solution and heating to 50°C, no exothermic reaction or gas generation was observed. Similarly, adding 30 wt% sodium nitrite to 30 wt% ethylene glycol in the DSC resulted in no detectable heat of reaction. However, when researchers added 40 wt% sodium permanganate to 30 wt% ethylene glycol inside the ARSST, an immediate, energetic reaction occurred, causing significant temperature

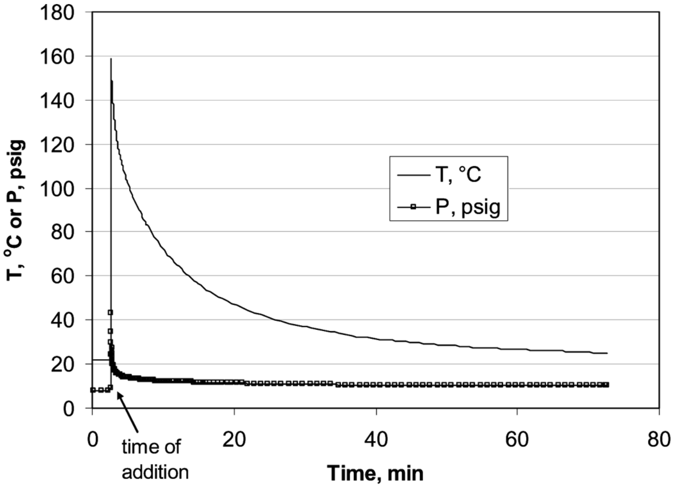


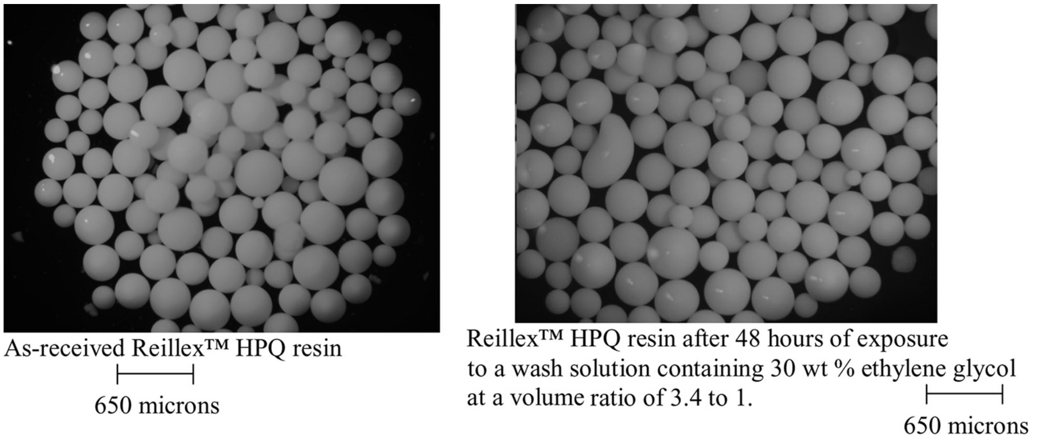
FIG. 4. Reaction after addition of 40 wt% $NaMnO_4$ to 30 wt% ethylene glycol.

and pressure spikes inside the calorimeter. Temperature and pressure data are shown in Fig. 4. The gas produced by the permanganate test appears to be largely condensable (gases other than CO_2/CO), since the pressure rapidly increased and quickly decreased. The pressure rate calculated using a standard method (10) is 0.01 Mpsi/min, which is less than the criteria values set for explosive and deflagrating materials (11). The reaction produced a dark solid material (likely MnO_2) and expelled a significant amount of solid reaction product from the test cell which stuck to the top and upper walls of the inside of the calorimeter. The observed reaction is consistent with that reported by Smith (12). The reaction condition is simpler than the one observed by Smith, which included HNO_3.

Effects of Ethylene Glycol on Reillex™ HPQ Resin

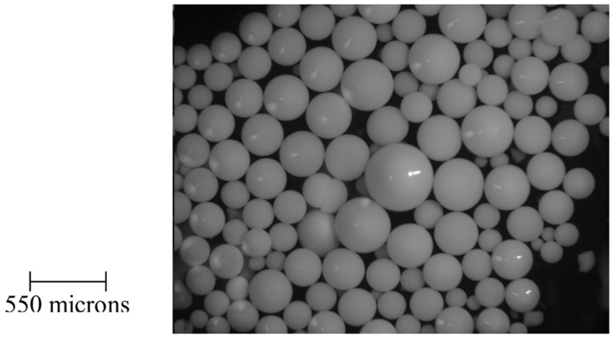
Researchers shook Reillex™ HPQ resin in 30 wt% ethylene glycol solutions as well as in wash tank solution (6.2 M HNO_3/0.06 M hydrazine/0.06 M ferrous sulfamate) mixed with 30 wt% ethylene glycol at a 3.4:1 volumetric ratio for 24 and 48 hours, respectively in a rotary shaker (the times reflect the maximum time the resin is expected to be in contact with ethylene glycol). The resins from each test were observed with an optical microscope.

Figure 5 shows pictures of portions of the resin after contacting the wash tank solution and a solution of 30 wt% ethylene glycol. Inspection of the pictures clearly



As-received Reillex™ HPQ resin

Reillex™ HPQ resin after 48 hours of exposure to a wash solution containing 30 wt % ethylene glycol at a volume ratio of 3.4 to 1.



Reillex™ HPQ resin after 24 hours of exposure to a solution containing 30 wt % ethylene glycol.

FIG. 5. An optical picture of the Reillex™ HPQ resin after exposure to various solutions. All pictures taken at the same magnification (26×).

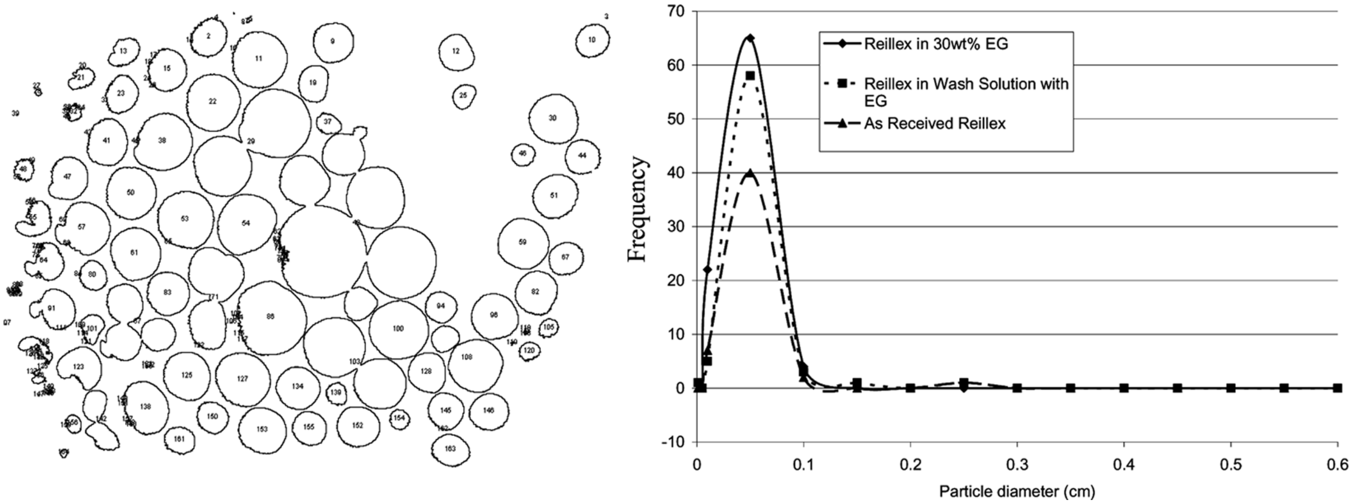


FIG. 6. An illustration of the segmentation from a typical picture as obtained in Fig. 5 (left) and the corresponding particle size distribution (right) not normalized. The figure on the right clearly shows no appreciable changes in the particle size distribution due to the presence of ethylene glycol.

shows no obvious increase or decrease in particle diameter or particle shape deformation due to ethylene glycol. The particle size distribution was determined from these pictures using imaging software "ImageJ 1.37v" from the National Institute of Health. If ethylene glycol physically affected the resin, a shift or a high diameter shoulder in the case of swelling will be noticed. Figure 6 shows a picture of the edges of the resin particle as detected by the imaging software. Also shown in Fig. 6 is the particle size distribution of the resin after exposures to an ethylene glycol solution and the wash tank solution containing ethylene glycol. All particle size distribution curves are similar to each other and, therefore, researchers conclude ethylene glycol had no measurable impact on the physical size or shape of the Reillex^{TM} HPQ resin.

Researchers also conducted spectroscopic analysis of the resin after exposure to the wash tank solution containing ethylene glycol and to the 30 wt% ethylene glycol solution. Figure 7 shows the FTIR spectra of Reillex^{TM} HPQ after exposure to the wash tank solution containing ethylene glycol. Inspection of Fig. 7 shows that the ethylene glycol signal (seen on the top spectrum) was removed by rinsing

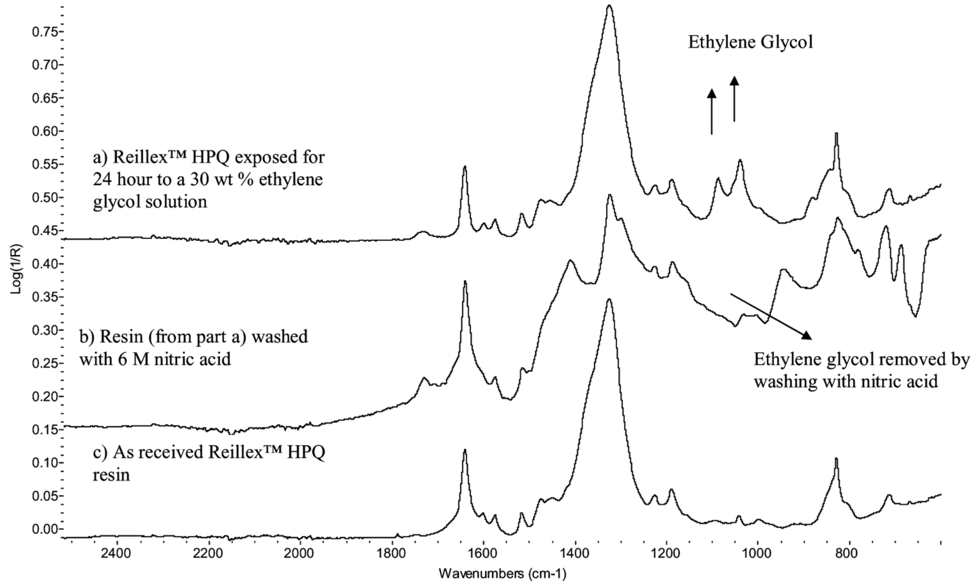


FIG. 7. The FTIR spectrum of the Reillex^{TM} HPQ bead after exposure to a 30 wt% solution of ethylene glycol for 24 hours and after washing with 6 M nitric acid showing the easy removal of ethylene glycol from resin. The spectra were obtained from both the beads and crushed beads.

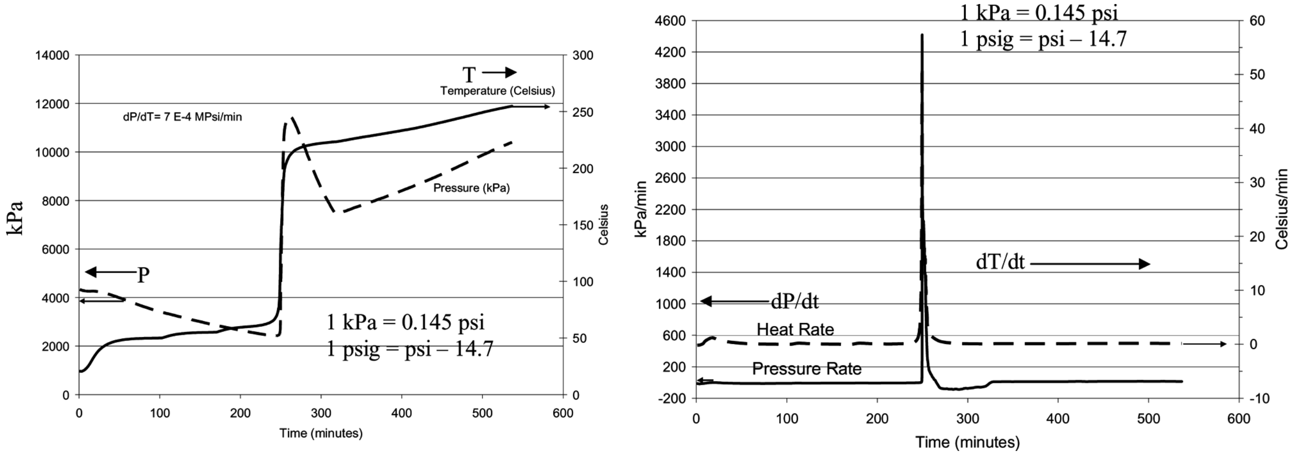


FIG. 8. The thermal activation of the oxidation reaction between ethylene glycol and 6.4 M nitric acid (3.4 to 1 volume ratio) in the presence of 200 mg (on a dried basis) of Reillex^{TM} HPQ resin.

the resin with 6 M nitric acid. Researchers shook 0.12 grams of resin on a dry basis in 0.5 mL of 6 M nitric for one minute. FTIR analysis of the inside of the Reillex^{TM} HPQ resin beads revealed that no ethylene glycol was found inside the resin beads. Similarly, the FTIR spectra of the Reillex^{TM} HPQ resin exposed to 30 wt% ethylene glycol clearly shows that rinsing the resin with 6 M nitric acid removed the ethylene glycol. No ethylene glycol was found inside the crushed resin beads. Since ethylene glycol does not diffuse into the resin within the timeframe of this experiment and it is easily removed from the beads, researchers believe ethylene glycol will have no effect on the actinide sorption and desorption (capacity and rate) of Reillex^{TM} HPQ resin. In fact, ethylene glycol is also easily removed with water.

Researchers then investigated if an accidental introduction of 6 M nitric acid containing ethylene glycol (30 wt% added to give a volume ratio of 1 to 3.4) to the column containing Reillex^{TM} HPQ resin (added 0.2 grams on a dried basis) could pose a thermal hazard given that both ethylene glycol (at 60°C) and Reillex^{TM} HPQ (at 145°C) oxidize in nitric acid (12). Specifically, a risk exists that the temperature rise from the oxidation of ethylene glycol may start the oxidation of the resin. Figure 8 shows the ARC result from the thermal activation of Reillex^{TM} HPQ resin in a nitric acid solution containing ethylene glycol. Inspection of Fig. 8 clearly shows the oxidation of ethylene glycol as noted by the sharp temperature increase from 60°C to 240°C. This temperature increase is similar to the temperature increase seen in Fig. 2. This observation indicates that the resin did not oxidize despite the temperature reaching more than 200°C. In addition, a visual inspection of the resin after the heating experiment revealed the resin had a mild brown coloration but no severe changes such as severe discoloration, tar formation, or resin deformation. A complete oxidation of 0.2 grams of Reillex^{TM} HPQ resin would have yielded a significant temperature rise of more than 10°C. Given an oxidation enthalpy of more than 3000 J/gram of resin (13,14) and a thermal inertia factor of 3.4 (Eq. [1] and Table 1), the temperature under adiabatic conditions (Eq. [3]) is well above the sensitivity of the ARC instrument used for this measurement.

$$thermal\ inertia = \frac{M_{sample} \times C_{sample} + M_{vessel} \times C_{vessel}}{M_{sample} \times C_{sample}} \tag{1}$$

$$C_{sample} = \frac{M_{acid} \times C_{acid} + M_{\mathrm{resin}} \times C_{\mathrm{resin}}}{M_{acid} \times C_{\mathrm{resin}}} \tag{2}$$

$$\Delta T = \frac{\Delta H_{oxidation\ reaction} \times M_{\mathrm{resin}}}{thermal\ inertia \times C_{sample} \times M_{sample}} \tag{3}$$

In addition, the kinetics of Reillex^{TM} HPQ resin oxidation in 8 M nitric acid is 100°C/min (15). This rate of reaction would be easily seen within the time scale of the experiment conducted in the ARC. Therefore, resin oxidation was not clearly observed.

TABLE 1
Values of parameters used in Eqs. [1–3]

M_{sample} (grams)	3
M_{vessel} (grams)	17
M_{resin} (grams)	0.2
C_{acid} (J/g°C)	4.22
C_{resin} (J/g°C) (16)	2.11
C_{vessel} (J/g°C)	1.9 (steel)
Thermal Inertia	3.4

CONCLUSIONS

Researchers examined the chemical compatibility and thermal behavior of ethylene glycol mixed with wash tank solution used in the ion exchange system of the HB-Line Phase II at SRS.

The conclusions from this work were:

1. Ethylene glycol oxidizes in 6 M nitric acid at 60°C under adiabatic conditions and at 95°C under near-adiabatic conditions. The maximum rate of gas generation observed due to oxidation was 0.007 Mpsi/min. This quantity is much less than the criteria values set for detonation and deflagration of materials heated in confined spaces (>0.25 Mpsi/min) (11). The reaction is classified as non-deflagrating and non-explosive.
2. Ethylene glycol does not oxidize in 6 M nitric acid solution containing 0.06 M hydrazine and 0.06 M ferrous sulfamate. The reducing power of the added chemicals appears to prevent the oxidation of ethylene glycol. The added chemicals scavenge the nitrous acid and can be used to prevent the oxidation of other organics.
3. Ethylene glycol readily reacts with 40 wt% potassium permanganate. The maximum rate of gas generation observed due to oxidation was 0.01 Mpsi/min. This value is less than the criteria values set for detonation and deflagration of materials heated in confined spaces (>0.25 Mpsi/min) (11).
4. Ethylene glycol reversibly sorbs on the surfaces of the Reillex^{TM} HPQ resin beads but does not diffuse inside the resin bead within timeframes of process interest. Ethylene glycol sorbed on the resin is easily removed with 6 M nitric acid or water.

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