Chemical Processing of Acrylic Samples for U and Th + Determination.

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Neutron Irradiation of the Acrylic Samples.

Samples of acrylic are machined, using either tungsten carbide or high speed steel cutting bits, so that they will fit into plastic pneumatic carriers. The finished samples are about 1 cm diam. by 3 cm long and weigh about 3.5 g. The samples are ultrasonically washed with HCl, HF, and water prior to insertion into the plastic carriers. Irradiations are made using the INC-5 Omega West reactor which has a neutron flux of about $1x10^{13}$ n/cm^2/s. The total irradiation fluence is typically $2.4x10^{17}$ n/cm^2.

Volatilization of the Acrylic.

After irradiation, the samples are taken out of the plastic carrier and placed in a volatilization tube along with ^{231}Pa and ^{236}Np for chemical yield determination. The volatilization tube, 25 mm diam. by 75 mm long, has a 12 mm diam. connection tube 100 mm long terminating in a 14/35 standard taper joint. The tube is connected, through a glass trap cooled in liquid nitrogen, to a vacuum pump. The system is evacuated to less than 0.01 torr and the tube is heated in a small furnace. Initially the temperature is held at 200-220 °C. until the sample melts and quits degassing. During this period the pressure will often increase to greater than 1 torr. When the pressure again drops to less than 0.05 torr, the temperature is the gradually raised to 400-420 ° + C. as the acrylic distills away. This process takes about 3-4 hrs and depends somewhat on the type of acrylic sample. At the end of the volatilization the tube is cooled to room temperature while still under vacuum. In most cases, there is only a small amount of carbonaceous material left in the tube. The tube is removed from the trap then the joint and upper end of the connection tube are cleaned to remove vacuum grease and any soluble high boiling material that may have condensed near the joint.

Digestion of the Residue.

Next 0.5 mℓ of conc. HNO_3 and 0.5 mℓ of conc. $HClO_4$ are added + to the tube. The tube is then heated to boil the acid mixture. More nitric acid is added as it distills from the tube. After most of the carbon is washed from the walls the last of the nitric acid is distilled off and the perchloric acid destroys the carbon yielding a pale green clear solution. The perchloric acid is the distilled down to near dryness, 0.5 mℓ nitric acid is added and distilled off to near dryness. Next 1 mℓ of conc. HCl is added and + distilled off. This is repeated until no more NO_2/N_2O_4 fumes are evolved. The solution is then transferred along with 1 mg of Fe^{+3} to a centrifuge tube and $Fe(OH)_3$ is precipitated with conc. NH_4OH. The $Fe(OH)_3$, carrying the Np and Pa activities, is centrifuged down and the supernant is discarded. The precipitate is then dissolved in conc. HCl, reprecipitated with conc. NH_4OH, and centrifuged. Again the supernant is discarded and the precipitate is dissolved in 0.5 mℓ conc. HCl.

Ion Exchange Separation of Np and Pa.

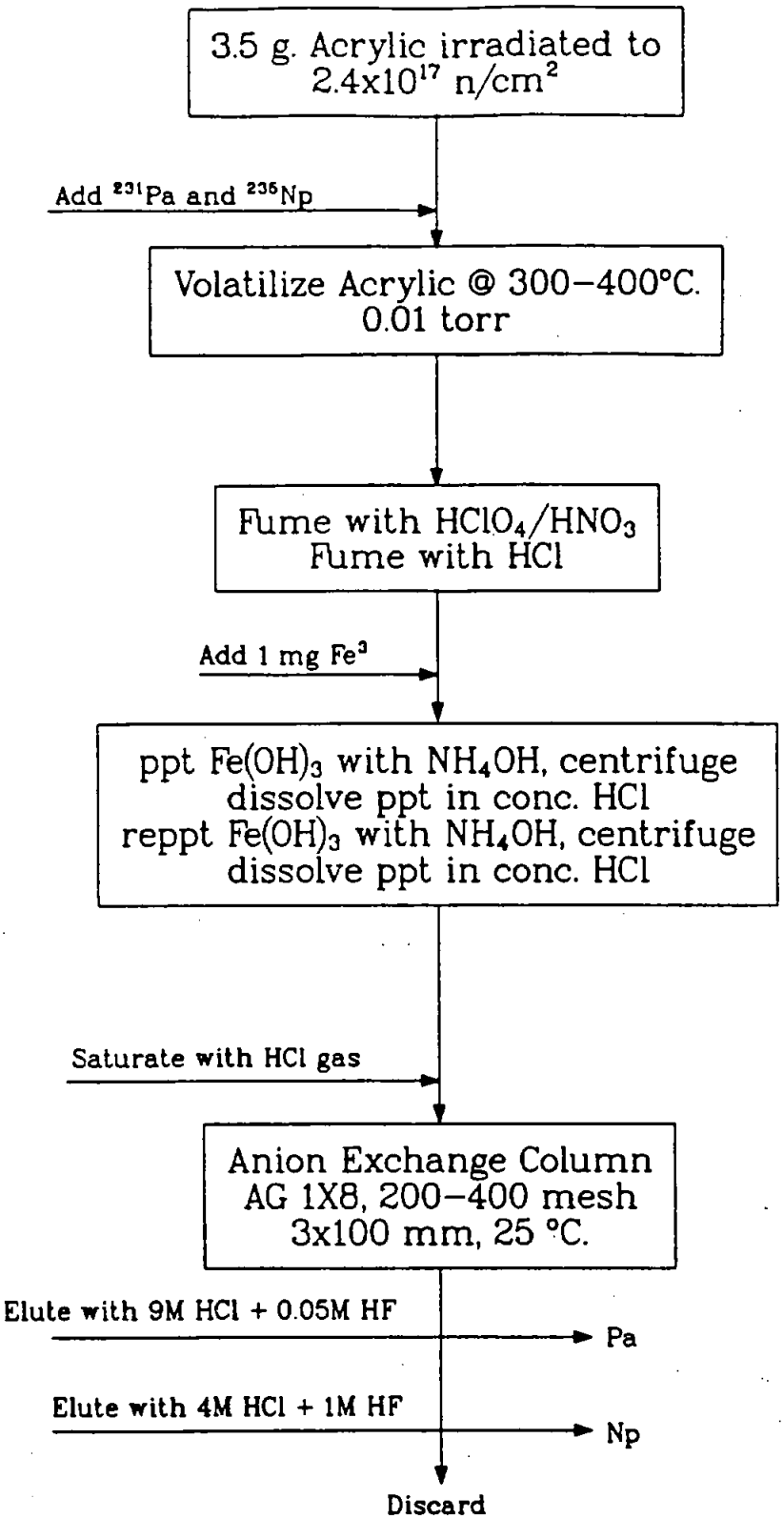
^{239}Np and ^{233}Pa are separated on a small column with AG 1X8 anion exchange resin (200-400 mesh). The column is a plastic tube 3.5 mm diam. and + 100 mm long that holds 1 cm^3 of resin. The column is pretreated with water then with 3 m +*ell* of conc. HCl. The solution from the digestion step is saturated with HCl gas the transferred to the column with 4 washes of 0.5 mℓ of conc. HCl. The Pa activity is eluted from the column with 3 mℓ of 9M HCl that contains 0.05M + HF. After the Pa is collected, the Np activity is eluted with 2 mℓ of 4M HCl containing 1M HF. Both the Pa and Np are collected directly in small counting vials. The chemical yield has been improving with time and is now about 70% for both Np and Pa.

Counting of the Np and Pa Activities.

The activities are measured with a well type intrinsic Ge detector. The detector is made with low background components and has a NaI(Tl) compton suppression shield. The background of the detector is about 3 c/hr/keV at 300 keV. With our sample size and irradiation conditions, 1 ppt of U in the acrylic gives a counting rate of 10.7 c/h while 1 ppt of Th gives a counting rate of 1.7 c/h assuming unity chemical yields.

In our irradiation conditions, with a poor cadmium ratio, the formation cross section for ^{233}Pa is about 9 barns while that for ^{239}Np is about 13 barns. Both of these effective cross sections are quite a bit larger than for the (n,γ) reactions with thermal neutrons. (7.4 ± 2.7 b)

The Th is estimated from the intensity of the 312 keV transition associated with the decay of ^{233}Pa and the U concentration comes from the intensity of the 228 keV transition in the decay of ^{239}Np.


3.5 g. Acrylic irradiated to 2.4x10^17 n/cm^2
Add 231Pa and 235Np
Volatilize Acrylic @ 300–400°C. 0.01 torr
Fume with HClO4/HNO3
Fume with HCl
Add 1 mg Fe3
ppt Fe(OH)3 with NH4OH, centrifuge
dissolve ppt in conc. HCl
reppt Fe(OH)3 with NH4OH, centrifuge
dissolve ppt in conc. HCl
Saturate with HCl gas
Anion Exchange Column
AG 1X8, 200–400 mesh
3x100 mm, 25 °C.
Elute with 9M HCl + 0.05M HF
Pa
Elute with 4M HCl + 1M HF
Np
Discard