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Berkeley, California
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SOLID-STATE COUNTERS FOR IMMEDIATE ANALYSIS DURING COLUMN CHEMISTRY

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Solid-State Counters for Immediate Analysis
During Column Chemistry

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ABSTRACT

A reliable method, using solid-state detectors, is described for the immediate determination of actinide and lanthanide elution peak positions during separation of transplutonium elements and rare earths.
A pressing need has existed for some method of quickly determining elution peak positions during the separation of transplutonium elements and rare earths.

The previous method was drop-by-drop counting in counters remote from the water shield and hot enclosure. The time this consumed and the excessive handling of highly radioactive samples had to be eliminated.

A reliable method which gives an immediate determination of the actinide and lanthanide elution peak positions on an ammonium α-hydroxy isobutyrate column has been developed during the past year at the Lawrence Radiation Laboratory in Berkeley.

Conditions that influenced this development stemmed from the requirement that many elution fractions and assays must be taken and later analyzed. In many cases an unsatisfactory separation still occurred because of the lack of immediate determination of the beginning and ending positions of the eluting fractions.

Attempts were made to analyze these fractions by transferring the elution cones to beta-gamma or neutron counters, but this entailed dangerously fast and excessive handling of the precious and high-level radioactive materials and consumed more time than was allowed to correctly operate a column.

Surface-barrier, solid-state detectors were then considered. A small, suitably packaged, lithium-drifted silicon detector was supported near the column tip for analyzing the surface of each drop for alpha and beta-gamma activity. It was found that a 13-λ drop having about $10^4$ disintegrations per minute of alpha could be detected above the background in the alpha, beta-gamma gloved-box by such a counter located 3/8 in. from the drop.

A system was then developed that not only measured the surface activity of the drops, but counted each drop, distinguished between beta-gamma and alpha activity, gave audible and visual indications of counting rates, and plotted all the information on a single strip chart. A solid-state neutron detector was also provided for rough column surveillance.

At first there was some doubt about the survival of these sensitive detectors in the hostile environment of the separation enclosure. Time has shown that simple precautions are adequate for long life -- such as gold
plating exposed metal parts, putting a gold-coated Mylar cover over the detector face, and providing for changing the Mylar in case of contamination.

Figure 1 shows the column assembly as it is used. The important parts are (1) heater block, (2) column, (3) drop counter, and (4) detector. Ideally the column is positioned with sufficient accuracy by having the shoulder of the column rest against a shoulder provided on the heater block; however, the shoulder on the column varies considerably about its circumference and some means was required to compensate for this variation in column tip position. Figure 2 shows an exploded view of the detector and the hood that corrects for column tip position. The reason that the column tip must be precisely located is that the tip becomes contaminated and would cause a very high background. The slot in the hood is designed so that the hood can be rotated and, at some position, the detector can see the drop as it forms, but not the tip.

Originally we intended to have a preamplifier in the detector holder, mainly to reduce cable losses, but it was not possible to do so without seriously affecting the signal-to-noise ratio. Instead, Nuvistor preamps were mounted outside the enclosure and their low noise properties more than offset the cable losses. Lately, field-effect transistor (FET) preamps have been used in place of the Nuvistors because of their much-improved signal-to-noise ratio.

Figure 3 is a block diagram of the electronics for the alpha, beta-gamma detector. Amplified pulses from the detector appear at both discriminators. Those pulses above the noise but below the alpha discriminator are fed to two one-shots where they are shaped, delayed, and appear at the base of the coincidence input transistor. The pulses above the alpha discriminator are fed to a one-shot that has two outputs -- one to the coincidence input transistor emitter, which clamps it off, and the other to the alpha CRM. The CRM outputs are fed to separate pens of a dual-channel recorder so that both beta-gamma and alpha count rates are simultaneously recorded. Some pulses from degraded alphas look like beta pulses and are counted as such; also, beta pile-up can cause pulses to form that look like alpha pulses; however, this does not occur to a serious degree and can be taken into account when interpreting the strip chart.
Fig. 1. Column assembly
(1) Heater block
(2) Column
(3) Drop counter
(4) Detector.
Fig. 2. Exploded view of the detector and the hood that corrects for column tip position.
Fig. 3. Block diagram of electronics for alpha, beta-gamma detector.
Figure 4 (top) is a typical display on a strip chart as the drops elute from the column. The column is an ammonium α-hydroxy isobutyrate column. The chart shows the drops progressing from right to left. From bottom to top is a four-decade log scale starting from $10^3$ to $10^7$ counts/min. Lower traces represent the alpha per drop and the upper the beta-gamma per drop. The small peaks show the activity buildup as the drop forms on the tip of the column, while the sharp decline reflects the drop falling from the tip of the column into the collecting cone. The overall peaks show the transcurium elution fractions. One can see the Fm elution peak with the beginning of the Es peak shown in the last half of the top chart. Below, the remainder of the Es peak and Cf peak is illustrated. One can notice the associated beta-gamma eluting in coincidence with the Cf fraction. The interrupted section two-thirds of the way through the Cf elution represents the time required to load the column with more butyrate solution, thus interrupting the regularity of drop formation.

Figure 5 (bottom) shows the last half of the elution. The top shows the Bk fraction (a beta-gamma emitter) and the bottom the Cm alpha peak, again with its associated beta-gamma. The pen marks on the edge of the chart indicate the drop counter display.

An alpha detector with a 0.06-in.-thick polyethylene foil placed on the surface of the detector is used for neutron counting. This device has moderate sensitivity to fast-neutrons via the proton-recoil interaction. The important factor, in this type of fast-neutron counter, is that its neutron sensitivity is unidirectional. The neutrons coming from the loaded columns strike the back of the converter foil, and the resulting recoil proton proceeds out the front face and away from the detector; thus the intense background caused by neutrons coming from the column or other neutron sources in an enclosure does not effect the detector. However, the recoil protons formed by neutrons striking the front face of the foil travel toward the detector and are counted.

Care should be taken to keep the detector free of contamination, by means of the changeable Mylar foil and retainer ring, and to position the column tip to give the correct shielding effect, thus eliminating background from tip contamination during operation. These detectors can be either stationary or portable, and allow the operator to detect the progress of fast neutrons or beta-gamma emitting isotopes along the eluting column.
Fig. 4. First half of strip chart recording of an ammonium α-hydroxy isobuterate column, showing eluting elements.
Fig. 5. Second half of strip chart recording of an ammonium α-hydroxy isobutirate column, showing eluting elements.
During the past year this system has proven itself by giving chemists an insight into the changing characteristics of an ion-exchange column, thus permitting exact separations of the radioactive fractions, lessening hours of handling and assaying, and decreasing the delivery time of short-lived radioisotope fractions to researchers.
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