Separation of Sixth B Group Elements (S, Se, Te, Po) with Anion Exchange Resin

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Selenium (IV), tellurium (IV) and polonium are known to form chloride complex anions of the type MCl_5^- or MCl_6^{--} in conc. hydrochloric acid solutions. This communication reports the complete separation of sulfur, selenium, tellurium and polonium by anion exchange chromatography of these chloride complexes.

The resin used was Dowex 1 X-4, porous strongly-basic type resin, ground less than 180 mesh, and was settled in a cylindrical bed 0.6 $cm^2 \times 3.8$ cm. Weighed quantities of 4 mg. of selenium, 6 mg. of tellurium and ca. $5 \times 10^{-3} \mu$C. of polonium were mixed and oxidized with fuming nitric acid. The mixture solution was evaporated to dryness, and dissolved in 1 cc. of 12 N hydrochloric acid. Besides them, the amount of 3 mg. of sulfur was added to the mixture in the form of sulfuric acid, as sulfur is volatile in its quadrivalent oxidation state in strong acidic solution.

The solution was placed at the top of the bed and the liquid level was allowed to fall to the resin, which was previously conditioned with 12 N hydrochloric acid. When the mixture solution was passing through the column, yellowish-green and yellow sharp adsorption bands due to selenium and tellurium respectively were clearly observed and distinguished from each other.

Elution was performed with 12 N hydrochloric acid at flow rate of about 0.2 cc./cm^2/min. at room temperature, and every 0.5 cc. fraction of effluent was collected and analyzed.

Sulfate was effectively not adsorbed to the resin and immediately appeared in the effluent, whereas the adsorption band of selenium moved only 1.5 cm. after the addition of 2.6 cc. of eluent (elution constant $E=0.3$),[1] and in the effluent the latter was not detected by the thiourea test.

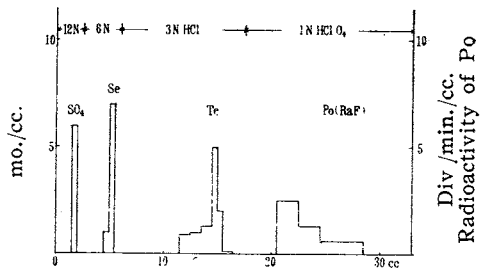

Fig. 1. Elution curve

Succeedingly, selenium was eluted completely with 3.5 cc. of 6 N hydrochloric acid, and then tellurium with 12 cc. of 2 N acid. Finally, polonium was desorbed with 1 N perchloric acid or 1N nitric acid.[2]

Sulfuric acid was determined as barium sulfate. Selenium and tellurium were reduced to their elementary states with sulfur dioxide and determined gravimetrically. Radioactivity of polonium was measured with the use of the Lauritsen electroscope. As shown in the figure, a nearly complete separation was obtained.

Within several hours after the adsorption of the sample, perchloric acid was effective to elute polonium. But after fifty hours, 3 N perchloric acid could elute only 10% of the total quantity of polonium; on the other hand, 3 N nitric acid was still effective. It was very likely that polonium was reduced in the bed or reacted irreversibly with ion exchange resin.

1) K.A. Kraus, G.E. Moore, *J. Am. Chem. Soc.*, **73**, 9 (1951), ibid., **75**, 1460 (1953).

2) T. Ishimori, : Abstracts of the Second Annual Meeting of The Japan Society for Analytical Chemistry., Oct. 1953 at Tokyo.

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