

tion. This is a "time-honored method capable of consistent accuracy, but only in the hands of an experienced analyst [1]". We have, therefore, previously converted mercuric cyanide to its tetracyano complex and titrated the latter with cetylpyridinium chloride [5] according to Eq (1). However, because this method required handling fair amounts of potassium cyanide, we decided to modify the method of Overman [2]. In this procedure mercuric ion is titrated with iodide, using an iodide ISE. This method can easily be adapted to the microscale and works in aqueous solution and in 0.1 N nitric acid. For mercuric cyanide, however, a more acidic medium is required. Optimum results were obtained in 1.8 to 2.4 M nitric acid solutions, or solutions containing between 45 and 60 mmol of nitric acid per 25 ml of solution. Ferric ion decomplexes some of the mercuric cyanide; in the presence of 0.2 mmol of ferric solution per 25 ml, the acidity may be reduced to 1 M nitric acid for satisfactory results. After acidification with nitric acid the solutions may be immediately titrated; no heating or waiting is required for removal of the hydrogen cyanide. However, acidification and titration of the samples should be carried out in a well-ventilated hood.

If solutions are heated with nitric acid in order to digest organic material, it is then mandatory to remove the oxides of nitrogen with sulfamic acid because these oxides will interfere in the titration with iodide.

Using 0.01 M sodium iodide as titrant, the endpoint breaks are several hundred mV. The relative percentage standard deviation for the normality of a typical titrant solution was 0.31% (4 replicates). A typical sample analysis yielded a mean value of 32.23% mercuric ion with a standard deviation of 0.08% for 4 repliates.

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Application of the ion-exchanger Cellex P for the selective separation of some metal ions

Part 5. Separation of gold from platinum and rhodium*

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Anwendung des Ionenaustauschers Cellex P zur selektiven Trennung von Metallionen

Teil 5. Trennung des Goldes von Platin und Rhodium

In an earlier paper we described the separation of platinum group metals by means of the system Cellex P/amines [1]. In the present paper we deal with the application of this system to the separation of gold from platinum and rhodium. The technique is useful for a separation prior to trace metal determination by AAS.

Reagents, apparatus and testing method were the same as described previously [1].

Without the presence of amines gold is only slightly retained, whereas quantitative sorption is achieved by adding amines. The best results were obtained with 1,3-diaminopropane and the pH-values as given below. In test experiments for the separation of Au and Pt 0.016–2.32 mg Au and 1–6 mg Pt were taken and were found with errors of ± 0.0004 –0.06 resp. 0.02–0.06 mg. The method was also applied to the separation of gold

from rhodium chloride. In this case $12.9 \times 10^{-3}\%$ Au were found by direct AAS and $11.2 \pm 0.7\%$ Au by the ion-exchange method.

Column separation. The solution of a mixture of Au and Pt or Rh in the presence of 1,3-diaminopropane (amine: Au = 50:1) was given into the column with a volume of 5 ml. Bed height of Cellex P was 1 cm for 0.10 g of resin and 2 cm for 0.20 g of resin. The pH was kept within the range of 6.0 to 9.5 for Au/Pt and 6.0 to 7.0 for Au/Rh. Gold is retained while the others are carried through. Pt or Rh remaining in void or interstitial spaces were washed out with 10 ml of twice-distilled water. Gold was eluted with 50 ml of 1 M HCl at a flow rate of 3 ml/min.

Determination of Au in rhodium chlorides. Weighed samples of RhCl_3 or $\text{RhCl}_3 \cdot 3\text{H}_2\text{O}$ were dissolved in 20 ml of 2 M HCl, and transferred to 25 ml flasks and made up to volume. 5 ml of the solution in the presence of 1,3-diaminopropane (amine: Au = 50:1) at suitable pH (see above) were introduced into the column filled with 0.10 g of Cellex P. Gold was eluted as above.

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* Part 4: see [1]
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