

the reaction is completed in a further 5 min, as indicated by the manometer, which should then read 1 mm or less. No loss in the efficiency of the catalyst after numerous hydrogenations was observed. The reaction efficiency is better than 99 per cent based on hydrogen. The mixture of ethane and excess ethylene is used as the counting gas.

International Atomic Energy Agency      H. PERSCHKE  
 Laboratory Seibersdorf  
 Niederoesterreich  
 Austria

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**Production of Carrier-Free I<sup>131</sup>-Iodate**

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CARRIER-FREE iodate solutions, frequently required e.g. in radiation chemical, biochemical etc. experiments, where the results may be affected even by trace contaminations, are usually produced with the use of oxidizing agents, like cerium(IV),<sup>(1)</sup> chromium(VI),<sup>(2)</sup> manganese(VIII),<sup>(3)</sup> elementary chlorine and nitric acid.<sup>(4)</sup> Oxidization with nitric acid yields products free from metal ions, provided the excess HNO<sub>3</sub> is removed in total darkness by vacuum distillation. In any oxidization technique the complete removal of oxidizing agents is a critical problem.

In the simple method to be described here no oxidizing agents are used. It is based on the adsorption of iodine ions on Pt-surface with subsequent electrochemical oxidation of the adsorbed iodine. I<sup>131</sup> is adsorbed in carrier-free form from strongly agitated sulphuric acid solution (about 0.5 M), onto Pt-adsorbent reduced previously by cathodic polarization.<sup>(5)</sup> The adsorption is performed in 20-30 min. The maximum adsorption per 1 cm<sup>2</sup> macroscopic surface is 0.7 × 10<sup>-6</sup> g iodine which means about 100 mc I<sup>131</sup>. This latter value might decrease in case of contaminating the I<sup>131</sup> with inactive iodine. After attaining equilibrium the Pt-adsorbent is first rinsed in distilled water, then immersed into a solution of desired composition—eventually redistilled water—and anodically coupled to a Pt-electrode of smaller surface. The applied voltage must be such that the

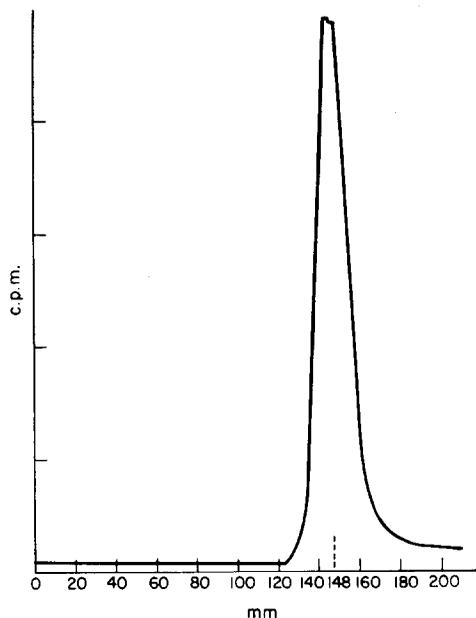


FIG. 1. Paper electrophoretogram of iodide ions.

polarization potential at the Pt-adsorbent anode should be at least 1.5 V. I<sup>131</sup> adsorbed as elementary iodine, passes then into the solution and is oxidized simultaneously to iodate. The electrochemical oxidation yield was checked by paper electrophoresis.<sup>(6)</sup> The migration rate of the ionic species, (established by paper electrophoretography and radiometry on a solution containing radioiodine in iodide

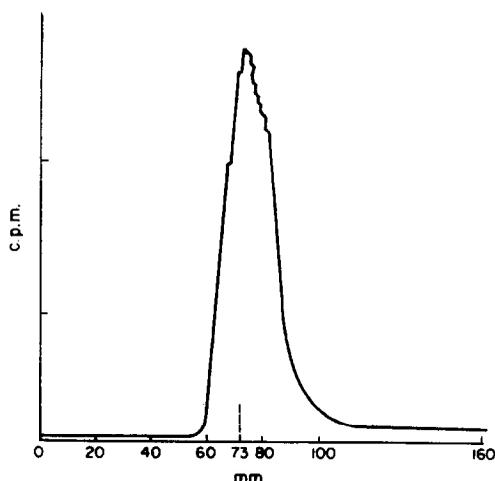


FIG. 2. Paper electrophoretogram of iodate ions.

form) was found to be 148 mm/20 min (Fig. 1). Paper electrophoretography of the electrochemically oxidized solution showed the presence of iodate ( $\text{IO}_3^-$ ) the migration rate of which proved to be 73 mm/20 min (Fig. 2).

To check the proper identification of iodate, two other paper electrophoretograms were analysed. One of them was taken of  $\text{KIO}_3$  in macro volume and the position of iodate identified by sodium iodide starch test from the blue colour. The other solution containing iodate was oxidized from  $\text{I}^{131}$  by chlorinated water. The peaks identified on two electrophoretograms by radiometry and the position of iodine starch blue were found to be in good agreement.

This simple technique lends itself well for the rapid production of carrier-free  $\text{I}^{131}$  iodate solutions at any pH required.

G. TÓTH  
AGNES G. NAGY

*Institute of Isotopes  
Central Research Institute for Physics  
Budapest*

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### The Determination of Copper in Quartz by Neutron Activation Analysis

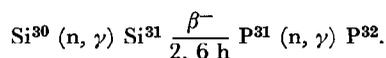
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COPPER is one of the so-called semiconductor poisons and has to be avoided in all procedures used for the preparation of semiconductors. Quartz is a universally used material in the preparation of semiconductor devices. It has been found that copper from quartz ampoules is very effectively introduced into GaAs (a semiconductor of the  $\text{A}^{\text{III}} \text{B}^{\text{V}}$  type) during heat treatment steps such as diffusion, zone refining, etc.<sup>(1,2)</sup> Whereas there are several reports concerning the copper content in semiconductor materials, to our knowledge only one reference exists<sup>(2)</sup> which gives upper limits for the copper content of

two types of quartz. The determinations were made by emission spectroscopy and neutron activation technique. In our experiments, four types of commercially available quartz were analysed for their copper concentration by neutron activation followed by a chemical separation.

#### Experimental

1-2 g of each of the different quartz samples were irradiated in a nuclear reactor for 24 hr with a thermal neutron flux of  $2,3 \times 10^{13} \text{ n/cm}^2/\text{sec}$ . For standards, known amounts in the order of 1 mg of metallic copper were sealed in quartz ampoules and were irradiated together with the quartz. After the irradiation and a cooling time of 26 hr (for the  $\text{Si}^{31}$  radioactivity) the quartz was heated in aqua regia to remove any surface contaminants. Then the quartz was powdered and weighed in a platinum dish. 2 ml of a  $\text{Cu}(\text{NO}_3)_2$  solution containing 100 mg of copper were added as carrier. The quartz was brought into solution with 5 ml  $\text{HNO}_3$  and 20 ml HF which was added in portions. The solution was then evaporated to dryness to remove the  $\text{SiO}_2$  and the residue was brought into solution with 5 ml  $\text{HNO}_3$ . 10 mg each of  $\text{Na}_2\text{CO}_3$  and  $\text{K}_2\text{CO}_3$  and 100 mg of  $(\text{NH}_4)_2\text{HPO}_4$  were added as holdback carrier for the  $\text{Na}^{24}$ ,  $\text{K}^{42}$  and  $\text{P}^{32}$  radioactivity. These nuclides were either formed from naturally occurring sodium and potassium impurities in the quartz or by the reaction



The copper of the solutions was electroplated on a platinum cathode. It was then removed from the cathode by bringing it into solution with a few ml of diluted  $\text{HNO}_3$ . To this solution about 100 mg  $\text{FeCl}_3$  was added. The solution was then heated to boiling. A  $\text{Fe}(\text{OH})_3$  precipitation step was carried out by dropwise adding of  $\text{NH}_4\text{OH}$  until the deep blue colour of the  $\text{Cu}(\text{NH}_3)_6^{2+}$  complex appeared. The  $\text{Fe}(\text{OH})_3$  precipitation step was undertaken to scavenge unknown radioactive trace impurities. It always showed small amounts of radioactivity. The  $\text{Fe}(\text{OH})_3$  precipitate was filtered and the filtrate containing the copper was acidified with  $\text{HNO}_3$  and the copper electrodeposited again. From the weight increase of the electrode the chemical yield was derived. For counting, the copper was removed from the cathode with 5 ml  $\text{HNO}_3$  and this solution was counted in a NaI Tl-activated well-type crystal with a single-channel pulse-height analyser. The 0,51 MeV peak of the  $\text{Cu}^{64}$  annihilation radiation was used for the measurements. The count rate was compared with the standard which was prepared by bringing the irradiated metallic copper into solution and aliquoting and diluting the solution to a concentration of