NEUTRON ACTIVATION ANALYSIS OF PALLADIUM, PLATINUM AND RHODIUM IN LEAD FOAM

K. S. PARK,* R. GIJBELS,** J. HOSTE

Institute for Nuclear Chemistry, University of Ghent, Ghent (Belgium)

(Received July 17, 1969)

The determination of palladium, platinum and rhodium in industrial concentrates such as lead foam and raw lead by neutron activation analysis is described. The noble elements are separated from the matrix by spontaneous deposition on amalgamated copper powder prior to activation. After the determination of palladium and platinum, rhodium is coprecipitated on iron hydroxide, and the precipitate irradiated for the determination of rhodium. The results are compared with those obtained by fire assay.

Introduction

In the present paper the determination of palladium, platinum and rhodium in industrial samples, such as lead foam and raw lead is described. Typical compositions of such samples are given in Table 1. It is important for the industry to know not only the quite high gold and silver contents of these materials, but also the concentrations of the other noble metals. The classical fire assay, followed by cupellation of the lead bead, does not result in complete rhodium recoveries, as it has been indicated elsewhere. More reliable analyses can be obtained by a modified fire assay (reduction of CuO and collection of the noble metals in copper), followed by a separation of copper on a cation exchanger and determination of the noble metals in the eluate by atomic absorption. Nevertheless it is highly desirable to check the above routine method by a completely different

Table 1
Composition of the lead samples

<table>
<thead>
<tr>
<th>Lead samples</th>
<th>Ag, ppm</th>
<th>Au, ppm</th>
<th>Pb, %</th>
<th>Cu, %</th>
<th>Bi, %</th>
<th>As, %</th>
<th>Sb, %</th>
<th>Sn, %</th>
<th>Si, %</th>
<th>Se, %</th>
<th>Te, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lead foam 1</td>
<td>5.603</td>
<td>152.5</td>
<td>56.39</td>
<td>13.15</td>
<td>0.020</td>
<td>2.58</td>
<td>7.90</td>
<td>6.00</td>
<td>4.37</td>
<td>0.22</td>
<td>—</td>
</tr>
<tr>
<td>Lead foam 2</td>
<td>2.800</td>
<td>45.5</td>
<td>53.66</td>
<td>27.19</td>
<td>0.008</td>
<td>2.76</td>
<td>1.45</td>
<td>5.30</td>
<td>1.00</td>
<td>0.10</td>
<td>0.03</td>
</tr>
<tr>
<td>Raw lead</td>
<td>10.348</td>
<td>151.9</td>
<td>94.60</td>
<td>0.16</td>
<td>0.55</td>
<td>0.10</td>
<td>2.60</td>
<td>0.83</td>
<td>—</td>
<td>—</td>
<td>0.34</td>
</tr>
</tbody>
</table>

* Present address: Atomic Energy Research Institute, Seoul, Korea.
** Research Associate of I.I.K.W., Belgium.
technique, all the more so as interelement interferences of the platinum metals occur in atomic absorption.\textsuperscript{3} The method involves the separation of these elements from the sample before neutron irradiation using amalgamated copper powder, as described previously.\textsuperscript{1} Although pre-separation is not really required, it is recommended to avoid manipulation with high activities induced in the samples due to antimony and silver isotopes. A pre-separation is also preferable because of the relatively short half-lives of the platinum, palladium and especially rhodium isotopes as compared with the time required for chemical separation.

\textbf{Separation scheme}

The separation scheme is represented in Fig. 1, and the behaviour of some of the elements involved is summarized in Table 2, as determined by radioactive tracer experiments. As it appears from Table 2, with the exception of rhodium, the platinum metals are not appreciably coprecipitated on lead, antimony and silver chloride, and they are almost quantitatively reduced by amalgamated copper powder.\textsuperscript{1} During this deposition, an additional decontamination from base metals such as sodium, nickel and a small amount of lead, antimony, tin and tellurium is obtained.

After irradiation for 6 hrs at a thermal neutron flux of $8.5 \cdot 10^{10} \text{n} \cdot \text{cm}^{-2} \cdot \text{sec}^{-1}$ in the Thetis reactor, the filter paper is carbonized and the mercury volatilized from the amalgamated copper powder by gentle heating in a ‘Main Smith’ quartz crucible.

The residual copper is dissolved in aqua regia, in the presence of palladium and platinum carriers. The elements are converted to the chlorides and the solution diluted to 1N HCl. If a small precipitate of AgCl is formed, it is filtered off. Rhodium tends to coprecipitate, particularly if the sample has been previously heated (compare Rh in fractions 1 and 7 in Table 2).

\begin{table}[h]
\centering
\begin{tabular}{|l|c|c|c|c|c|c|c|}
\hline
\textbf{Element} & \textbf{1} & \textbf{2} & \textbf{3} & \textbf{4} & \textbf{5} & \textbf{6} & \textbf{7} & \textbf{8} \\
\hline
Pd & < 0.3 & >99.7 & >99.7 & >99.7 & >99.7 & --- & --- & --- \\
Pt & < 0.2 & >99.8 & >99.8 & >99.8 & >99.8 & --- & --- & --- \\
Rh & < 4.5 & >95.5 & >93.4 & >93.4 & >93.4 & \sim 50 & --- & --- \\
Au & < 0.1 & >99.9 & >99.9 & >99.9 & >99.9 & --- & --- & --- \\
Ag & >99 & < 1 & < 1 & < 1 & < 1 & --- & --- & --- \\
Ir & < 1 & >99 & >99 & >99 & >99 & --- & --- & --- \\
Hg & --- & --- & 100 & -- & -- & --- & --- & --- \\
Sb & >99 & < 1 & < 0.01 & < 0.01 & < 0.01 & --- & --- & --- \\
\hline
\end{tabular}
\caption{Behaviour of the elements in the separation}
\end{table}

\textit{J. Radioanal. Chem. 5 (1970)}