CHAPTER VII

Distribution Coefficient of Antimony in Anisole-Formic acid system.

Liquid liquid extraction is a convenient method for the separation and determination of inorganic elements in complex mixtures. Radiotracers offer simple and rapid methods of analysis for the species extracted. Applications of various extractors for the determination of antimony is summarized in Table XXXV. A reference to this table shows that no studies have been reported of the extraction of Sb and Sn in anisole from formic acid systems. Our studies on the separation of Sn and Sb had shown that anisole-formic acid system is very efficient for the separation of Sn and Sb. If we could find out the distribution coefficients of Sn and Sb in anisole-aqueous formic acid systems this may help in the correlation of Rf values with the distribution coefficient and it may give us a new pair of solvent systems for the extraction of tin and antimony. The present work was therefore undertaken to determine the distribution
coefficients of tin and antimony in anisole-formic acid systems. The tracer technique was used in these studies since it is simple, fast and elegant.

**TABLE - XXXV**

**L - L Extraction of Antimony and Tin.**

<table>
<thead>
<tr>
<th>Element extracted</th>
<th>Separation From</th>
<th>Aqueous phase</th>
<th>Organic Phase</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sb</td>
<td>Mo</td>
<td>Citric acid, Methyl Violet</td>
<td>Benzene, Toluene</td>
<td>(16)</td>
</tr>
<tr>
<td></td>
<td>Many cations</td>
<td>4-11M HCl, HCl-Brilliant green</td>
<td>T.B.P.</td>
<td>(28)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>HCl</td>
<td>Benzene</td>
<td>(15)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>7M HCl, 6M H₃PO₄</td>
<td>Isopropyl ether</td>
<td>(36)</td>
</tr>
<tr>
<td></td>
<td>Fission products</td>
<td>M₁ BK</td>
<td>Diisobutyl carbinol-n Heptane</td>
<td>(17)</td>
</tr>
<tr>
<td>Mg Alloys</td>
<td>Na-Diethyl dithiocarbamate</td>
<td>Ethylacetate, Butyl acetate, 4-Methyl 2-Pentanol</td>
<td>(20)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>7M HCl</td>
<td>Isobutyl acetate, Bisisobutyl Carbinol</td>
<td>(14, 17), (29)</td>
<td></td>
</tr>
</tbody>
</table>

... Contd. on next page ...
eM HCl - Carbon Tetra Chloride (9), (24),
Rhodamine - Chlorobenzene (32)

eM HCl - Brilliant green (1), (31)
Na NO₂ - Toluene

KI, K₂SO₄ - MIBK (7)
or HCl

As, Bi - H₂SO₄, MIBK (33)
Kf

Many Metals - HCl diff Ethyl acetate conc.
" - TBP. (25)
" - MIBK (8)

H₂SO₄, Benzene (26)
HClO₄

Many cations - 1-2M HCl Ethyl acetate (40)
Mg

Tl-Ir - H₂SO₄, Chloroform (18)
Cupferron

Many cations - pH 9.2 EDTA - Sod. diethyl dithio carbamate chloroform (2)
NaCN

9N H₂SO₄ Benzene (39)
KI

W, V, Fe - 3-8M HCl " (19), (23), (38)

Sn - HCl - Amm. Carbontetra
Zn - Acetate chloride

Many Elements - HCl Tris(2-ethyl hexyl) phosphene oxide (27)
in cyclohexane

... Contd. on next page ...
<table>
<thead>
<tr>
<th>Element/Condition</th>
<th>Solvent</th>
</tr>
</thead>
<tbody>
<tr>
<td>Most HClO₄, NaI, Elements NaClO₄</td>
<td>Benzene</td>
</tr>
<tr>
<td>Soil HCl, ScH ascorbic acid</td>
<td>Ethyl acetate</td>
</tr>
<tr>
<td>Fe HCl, Ascorbic acid</td>
<td>MIBK</td>
</tr>
<tr>
<td>Nuclear Reactor pH 0.8,20% NH₄Cl 8-Quinonol</td>
<td>Chloroform</td>
</tr>
<tr>
<td></td>
<td>Alizarine Blue,N HCl Cyclohexanone Ethylacetate</td>
</tr>
<tr>
<td>In-Sb 4M HI, KI, H₂SO₄</td>
<td>Ether</td>
</tr>
<tr>
<td>Pb 7-8 M HCl</td>
<td>Amberlite LA2/ Xylene</td>
</tr>
<tr>
<td>Sb H₂SO₄, NH₄CN</td>
<td>Ethyl acetate</td>
</tr>
<tr>
<td></td>
<td>IM HNO₃ Butyl phosphoric acid Butyl ether</td>
</tr>
<tr>
<td>Many Metals 4.6M HF</td>
<td>Ethyl ether</td>
</tr>
<tr>
<td></td>
<td>6.9M HI Ethyl ether</td>
</tr>
<tr>
<td></td>
<td>KI-1.5M</td>
</tr>
<tr>
<td></td>
<td>H₂SO₄</td>
</tr>
<tr>
<td></td>
<td>DH 2.5-6 3-Quinonol in Chloroform</td>
</tr>
</tbody>
</table>
EXPERIMENTAL

Reagents: Antimony-125 trichloride was obtained from Atomic Energy Establishment Bombay. Ten ml of solution contained 540 microcuries. E. Merck hydrochloric acid and formic acid were used. Anisole (Bush) was distilled before use.

Apparatus: The radiotracer was measured with a micro-pipette and the separation of the layers was performed in 50 ml separating funnels. β-emission was measured from a counter. Supplied by A.E.E. Bombay. Planchets of diameter 2 cms with a cavity of diameter 1 cm were kept on an aluminium plate which was inserted in the counter.

Rubber gloves were used to avoid the contact of radio active material with the skin. Cellotape was used to cover the planchets. Standardization of antimony and hydrochloric acid solutions was made with sodium hydroxide and potassium permanganate solutions.
Measured quantities of hydrochloric acid were added to the anisole-formic acid (1:1) mixture, to study the separation of layers. Almost equal layers of the organic and aqueous phases were formed when 14 ml of anisole-formic acid (1:1) were mixed with 1 ml of hydrochloric acid. 0.1 M solutions of antimony trichloride in hydrochloric acid ranging from 3 - 8 M were prepared. 1 ml of each solution was shaken with 15 ml of anisole-formic acid mixture and 0.1 ml of isotope solution. On attainment of the equilibrium 0.1 ml portions of the liquids from the aqueous and organic phases were taken out, poured in the clean planchets, dried with a lamp and subjected to $\beta$ counting. Results are recorded in Table XXXVI.
Distribution of antimony in hydrochloric acid-anisole-formic acid system.

<table>
<thead>
<tr>
<th>Strength of HCl</th>
<th>Counts in ac. phase</th>
<th>Counts in organic phase</th>
<th>% of Sb</th>
<th>% of Sb</th>
</tr>
</thead>
<tbody>
<tr>
<td>3 M</td>
<td>338</td>
<td>222</td>
<td>39.8</td>
<td>60.2</td>
</tr>
<tr>
<td>4 M</td>
<td>301</td>
<td>220</td>
<td>42.2</td>
<td>57.8</td>
</tr>
<tr>
<td>6 M</td>
<td>460</td>
<td>261</td>
<td>36.2</td>
<td>63.8</td>
</tr>
<tr>
<td>8 M</td>
<td>462</td>
<td>210</td>
<td>31.2</td>
<td>68.8</td>
</tr>
</tbody>
</table>

A more detailed study was performed to confirm the fact that 3 or 4 M hydrochloric acid gives highest percentage of antimony in organic phase. Nine samples of hydrochloric acid (2-8 M) were prepared. Strength of antimony was fixed at 0.2 M and 0.5 ml of hydrochloric acid with antimony was added to 7 ml (1:1) mixture of anisole-formic acid. 0.1 ml of the radiotracer was added and 0.1 ml portions of the separated layers were taken out for the β counting. Results are summarized in Table XXXVII.
**TABLE - XXXVII**

Distribution of antimony in 7 ml anisole formic acid and 0.5 ml hydrochloric acid.

<table>
<thead>
<tr>
<th>Strength of HCl</th>
<th>Counts in aqueous phase /2 mnts.</th>
<th>Counts in organic phase /2 mnts.</th>
<th>% of antimony Organic Phase</th>
<th>Aq. phase</th>
</tr>
</thead>
<tbody>
<tr>
<td>2 M</td>
<td>1998</td>
<td>948</td>
<td>31.8</td>
<td>68.2</td>
</tr>
<tr>
<td>3 M</td>
<td>1366</td>
<td>826</td>
<td>37.6</td>
<td>62.4</td>
</tr>
<tr>
<td>3.5 M</td>
<td>1680</td>
<td>999</td>
<td>36.8</td>
<td>63.2</td>
</tr>
<tr>
<td>4 M</td>
<td>1566</td>
<td>766</td>
<td>32.5</td>
<td>67.5</td>
</tr>
<tr>
<td>4.5 M</td>
<td>1723</td>
<td>745</td>
<td>30.0</td>
<td>70.0</td>
</tr>
<tr>
<td>5 M</td>
<td>1933</td>
<td>686</td>
<td>25.6</td>
<td>74.4</td>
</tr>
<tr>
<td>6 M</td>
<td>1877</td>
<td>733</td>
<td>27.9</td>
<td>72.1</td>
</tr>
<tr>
<td>7 M</td>
<td>1784</td>
<td>748</td>
<td>28.9</td>
<td>71.1</td>
</tr>
<tr>
<td>8 M</td>
<td>1683</td>
<td>748</td>
<td>30.6</td>
<td>69.4</td>
</tr>
</tbody>
</table>

Background counts in Tables XXXVI and XXXVII were 32 and 40 per two minutes respectively.

Results of these tables were plotted in Figures 12 and 13.
A reference to table XII shows that antimony is extracted to a considerable extent by the organic phase. The concentration of antimony in the organic phase depends to some extent on the concentration of hydrochloric acid originally present in the antimony sample. About 3 M hydrochloric acid is best for the maximum extraction of antimony in the organic phase. Similar experiments with Sn$^{+2}$ will be required before the Rf values of Sn$^{+2}$ and Sb$^{+3}$ in anisole-formic acid systems can be correlated with the distribution coefficient.
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