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Liquid-Liquid Extraction of Antimony (III) with Mesityl Oxide

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NOTE

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Abstract

Mesityl oxide [$(CH_3)_2C{=}CHCOCH_3$] was used for the liquid–liquid extraction of antimony. It can be quantitatively extracted from 3 *M* hydrochloric acid within 10 sec of equilibration with 15 ml of mesityl oxide. The metal from the organic phase was stripped with 10 ml of water and was determined photometrically as its iodide complex at 425 nm. The probable composition of the extractable species at 3 *M* hydrochloric acid was either $SbCl_3 \cdot 3MeO$ or [$HSbCl_4 \cdot (MeO)_3$] where MeO is mesityl oxide. The method is simple, rapid, and selective, as it was possible to separate antimony from a large number of elements which are associated with it in alloys and minerals. The method is applicable for the analysis of antimony in solder. The standard deviation is ±1%.

Mesityl oxide (4-methyl-3-pentene-2-one) has been successfully utilized for the solvent extraction of several transition elements (*1*). The extension of these studies revealed that it is possible to quantitatively extract antimony (III) with mesityl oxide from 3 *M* hydrochloric acid. The metal can be stripped from the organic phase with water and it can be determined photometrically as its iodide complex at 425 nm.

The various chelating agents used for the solvent extraction of antimony are described in a recent monograph (*2*). In addition to these, a

TABLE 1

Extraction as a Function of Acidity and Mesityl Oxide Concentration: Sb(III) = 300 μg

Mesityl oxide concentration	HCl initial (M)	% Extraction, E	Distribution ratio, D
100% (8.7 M)	0.5	23.07	0.5007
	1.0	77.00	5.590
	1.5	84.60	9.175
	2.0	88.45	12.790
	2.5	94.23	27.270
	3.0	100.00	∞
75% (6.53 M)	0.5	19.23	0.398
	1.0	46.15	1.43
	1.5	57.70	2.278
	2.0	73.15	4.550
	2.5	73.15	4.550
	3.0	77.00	5.590
50% (4.35 M)	0.5	7.70	0.140
	1.0	23.07	0.5007
	1.5	26.92	0.615
	2.0	34.62	0.8845
	2.5	38.47	1.044
	3.0	46.15	1.430
35% (3.04 M)	0.5–1.5	—	—
	0.2	7.7	0.140
	2.5	15.40	0.2838
	3.0	19.23	0.3976
25% (2.17 M)	0.5–1.5	—	—
	2.0	3.85	0.0668
	2.5	5.77	0.1023
	2.0	7.70	0.14

number of oxygenated solvents were also described (*3*) for the solvent extraction of antimony. The method described in this paper is simple, rapid, and selective. It is possible to accomplish clean-cut separation of antimony from several elements such as copper, indium, tin, lead, and bismuth, which are associated with it in alloys. The method is also applicable to the separation of antimony in fusible alloys such as solder.

GENERAL PROCEDURE

An aliquot of solution containing 300 μg of antimony was mixed with an appropriate volume of hydrochloric acid to give a desired acid concentration of 3 M in a total volume of 25 ml. The solution was then shaken for just 20 sec with 15 ml of pure mesityl oxide. The layers were allowed to settle and separate. Antimony was stripped from the organic phase with 10 ml of water. It was determined photometrically as its iodide complex (*4*) at 425 nm.

RESULTS AND DISCUSSION

Effect of Acidity and Mesityl Oxide Concentration

Antimony (III) was extracted from 0.5 to 3 *M* hydrochloric acid with 25–100% (2.17–8.70 *M*) mesityl oxide in toluene (Table 1). The results showed that the quantitative extraction of antimony was possible from

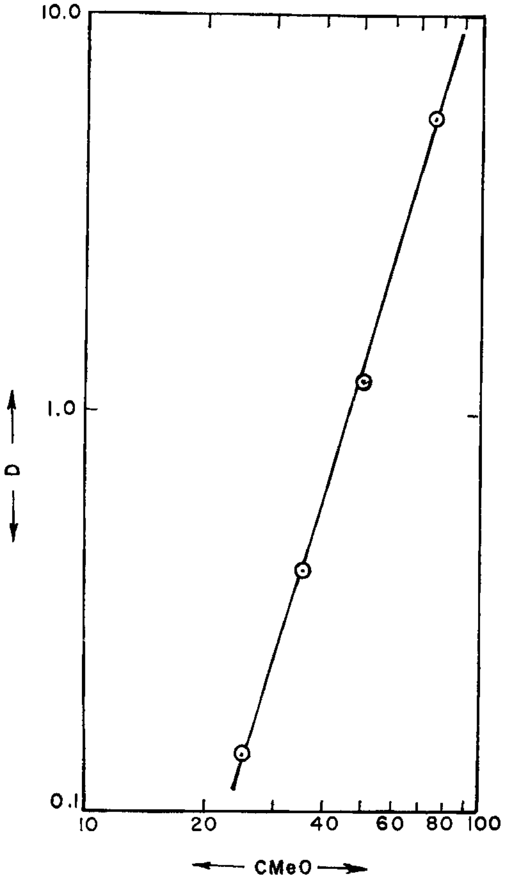

Fig. 1. Extraction as a function of MeO concentration.

3 *M* hydrochloric acid with undiluted mesityl oxide. It was not possible to extract antimony quantitatively ($<77\%$) with diluted mesityl oxide. The plot of log *D* vs log *C* (mesityl oxide) at 3 *M* hydrochloric acid concentration (Fig. 1) indicated a slope of 3.27. This showed that the probable composition of the extractable species was either $SbCl_3 \cdot 3MeO$ or $[HSbCl_4 \cdot (MeO)_3]$ where MeO is mesityl oxide. In order to ensure quantitative extraction of antimony, it is necessary to use 15 ml of mesityl oxide.

Effect of Salting-Out Agent

The effect of varying concentrations of salting-out agents such as chlorides of lithium, potassium, ammonium, and calcium on the extraction of antimony was studied. Pure mesityl oxide was used for the extraction of antimony from 0.5 to 3 *M* hydrochloric acid. A plot of percentage extraction vs molarity of hydrochloric acid (Fig. 2) showed that it was possible to quantitatively extract antimony at 2.5 *M* hydrochloric acid concentration in the presence of 2 *M* lithium, potassium, or ammonium chloride. In addition, it was possible to extract it at lower acidity, such as 1.5 *M* hydrochloric acid, in the presence of 1 *M* calcium or aluminum chloride as the salting-out agents.

Period of Extraction

The period of shaking was varied from 10 sec to 10 min. It was observed that extraction was quantitative within 10 sec of equilibration. It was further observed that prolonged shaking had an adverse effect on extractibility of antimony, possibly on account of dissociation of the ion-associated complex. This was analogous to other extraction systems involving mesityl oxide (*5*) as an extractant.

Effect of Diverse Ions

The various ions usually associated with antimony in alloys were tested for interference. The tolerance limit was set at the amount required to cause ±2% error in antimony recovery (*5*). The results showed that antimony can be extracted in the presence of fortyfold excess of several ions (Table 2) while ions such as lead, mercury, silver, copper, platinum metals, tin, germanium, arsenate, vanadate, and thiosulfate were tolerated in twentyfold ratios, but platinum and tellurite interfered seriously. Thus it was possible to accomplish the clean-cut separation of antimony from most of the elements which are associated with it in fusible alloys and minerals.

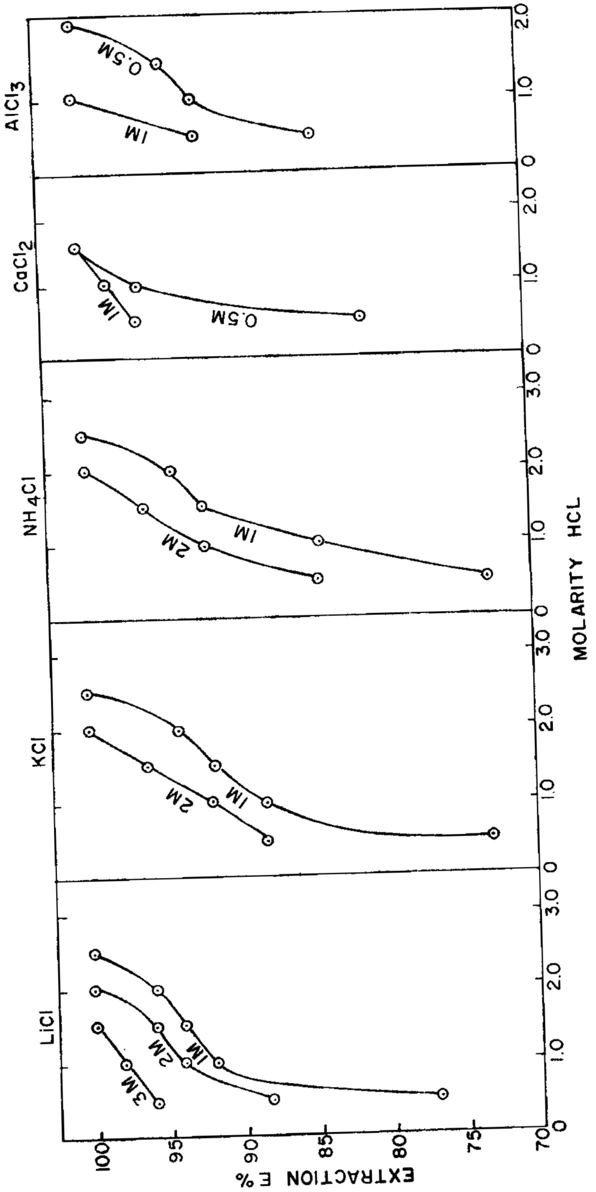

FIG. 2. Effect of salting-out agent.

TABLE 2

Effect of Diverse Ions:
Sb(III) = 300 μg, 3 *M* HCl, 100% Mesityl Oxide

Foreign ion	Added as	Tolerence limit (μg)
Pb^{2+}	$PbCl_2$	6,000
Hg^{2+}	$HgCl_2$	6,000
Tl^+	Tl_2SO_4	15.000
Ag^+	Ag_2SO_4	6.000
Cu^{2+}	$CuSO_4 \cdot 5H_2O$	6,000
Cd^{2+}	$3CdSO_4 \cdot 8H_2O$	5,000
Bi^{3+}	$Bi(NO_3)_3 \cdot 5H_2O$	3,000
Sn^{2+}	$SnCl_2 \cdot 2H_2O$	6,000
Ru^{3+}	$RuCl_3 \cdot 3H_2O$	5,000
Rh^{3+}	$RhCl_3 \cdot 3H_2O$	5,000
Pd^{2+}	$PdCl_2 \cdot 2H_2O$	3,000
Pt^{4+}	$H_2PtCl_6 \cdot xH_2O$	None
Fe^{3+}	$FeCl_3 \cdot 6H_2O$	9,000
Cr^{3+}	$CrCl_3 \cdot 6H_2O$	10,000
Al^{3+}	$AlCl_3 \cdot 6H_2O$	20,000
Ti^{4+}	$Ti_2(SO_4)_3 \cdot 6H_2O$	10,000
Be^{2+}	$BeSO_4 \cdot 4H_2O$	10,000
Th^{4+}	$Th(NO_3)_4 \cdot 4H_2O$	10,000
Zr^{4+}	$Zr(NO_3)_4 \cdot 4H_2O$	10,000
U^+	$UO_2(NO_3)_2 \cdot 6H_2O$	10,000
Zn^{2+}	$ZnSO_4 \cdot 7H_2O$	10,000
Mn^{2+}	$MnCl_2 \cdot 6H_2O$	10,000
Co^{2+}	$CoCl_2 \cdot 6H_2O$	10,000
Ni^{2+}	$NiCl_2 \cdot 6H_2O$	10,000
Ca^{2+}	$CaCl_2 \cdot 6H_2O$	20,000
Ba^{2+}	$BaCl_2 \cdot 2H_2O$	10,000
Mg^{2+}	$MgCl_2 \cdot 2H_2O$	20,000
Au^{3+}	$HAuCl_4 \cdot xH_2O$	3,000
In^{3+}	$InCl_3$	3,000
Ge^{4+}	$GeCl_4$	6,000
Rb^+	$RbCl$	5,000
Cs^+	$CsCl$	5,000
ReO_4	$KReO_4$	10,000
$VO_3{}^{4-}$	NH_4VO_3	6,000
$SeO_3{}^{2-}$	Na_2SeO_3	10,000
$TeO_3{}^{2-}$	Na_2TeO_3	None
$AsO_4{}^{3-}$	Na_3AsO_4	6,000
$Mo_7O_{24}{}^{6-}$	$(NH_4)_6 \cdot Mo_7O_{24}$	3,000
F^-	NaF	10,000
CN^-	KCN	10,000
$NO_3{}^-$	$NaNO_3$	20,000
SCN	NH_4SCN	10,000
$SO_4{}^{2-}$	$Na_2SO_4 \cdot 10H_2O$	10,000
$S_2O_3{}^{2-}$	$Na_2S_2O_3 \cdot 5H_2O$	5,000
$PO_4{}^{3-}$	$Na_3PO_4 \cdot 12H_2O$	10,000
$Acet^-$	CH_3COONa	10,000
$Ascorb^-$	Ascorbic acid	10,000
$Oxal^{2-}$	$H_2C_2O_4 \cdot 2H_2O$	10,000
Mal^{2-}	Malonic acid	10,000
Cit^{3-}	Citric acid	10,000
$Tart^{3-}$	Tartaric acid	10,000
$EDTA^{4-}$	EDTA (disodium)	10,000

Application to Analysis of Solder

A known weight (~0.25 g) of solder was dissolved in 10 ml of concentrated hydrochloric acid containing 0.5 ml of concentrated nitric acid. It was diluted to 50 ml with water. The small amount of lead which was precipitated as lead chloride in dilute solution was filtered out. About 1 ml of an aliquot was taken and was adjusted to 3 *M* with hydrochloric acid. It was then extracted and determined according to the general procedure. The results from the duplicate analysis of solder were 1.45 and 1.50% of antimony (reported as 1.50% of antimony). Thus the method for the analysis of solder is simple and rapid, as it is possible to directly extract antimony in the presence of lead and tin. The previous method (*6*) for the determination of antimony by its extraction with malachite green in toluene was cumbersome and time-consuming as it involved too many operational steps prior to the actual extraction of antimony.

The average recovery from 10 runs was 99.5 ± 0.5%. The relative standard deviation was ±1%. Since equilibrium is rapidly attained, the entire operation of extraction and photometric determination takes about ten minutes. The method permits separation of antimony from a large excess of many metals, and it is applicable for the analysis of solder.

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