THE SEPARATION OF MERCURY BY AMALGAMATION FROM OXIDISING SOLUTION

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For the separation of mercury from other compounds amalgamation has frequently been used[1-7]. When large amounts of insoluble material are present such as in tablet mixtures these procedures cannot generally be used because the reaction between the metal—usually zinc—used and the acid or alkali present causes the formation of hydrogen *in statu nascendi*. This, by direct action, or through reaction with other reducible compounds in the solution reduces ionic mercury to the metallic state or to mercurous salt and precipitates it partially on or in particles of the insoluble material from which it can be separated only with difficulty. This can be avoided by carrying out the amalgamation in a solution containing free bromine. The bromine prevents the precipitation of any mercury except on the surface of the zinc itself where both bromine and mercury are reduced.

The two following procedures were tried for the determination of mercury in tablets containing 18·5 mg. of 3-chloro-mercuri-2-methoxy-propylcarbamide and other substances (starch, lactose, talcum, gelatin, colour) to a total weight of 185 mg.

Procedure 1. Tablets containing about 500 mg. of 3-chloromercuri-2-methoxy-propyl-carbamide were ground and weighed into a 150-ml. Kjeldahl flask. 100 ml. of water and 0·5 ml. of bromine were added and the solution was mixed well. 10 ml. of glacial acetic acid and 1 g. of granulated zinc, 20 mesh, were then added. The mixture was heated to boiling temperature under strong swirling over a burner and then it was placed on a gas-heated Kjeldahl digestion stand over a very small flame during 1 hour. The size of the flame was adjusted so that it held the solution in constant movement, but no appreciable boiling occurred. The colour of the bromine disappeared during the digestion. After swirling, the solution with the suspended tablet-mixture was decanted from the amalgam and the latter was washed several times with much water which also was decanted. 10 ml. of concentrated nitric acid was then added under a hood and when all was dissolved 1 per cent. potassium permanganate solution was added until it was no longer decolorised, and then 5 ml. in excess. The solution was shaken well and transferred with water to a 250-ml. Erlenmeyer flask. A few drops of 30 per cent. hydrogen peroxide were added to destroy the permanganate and after mixing and cooling, 1 ml. of ferric ammonium sulphate indicator was added and the solution was titrated with ammonium thiocyanate to a faint brown colour. The recoveries obtained in a series of determinations are given in Table 1 under the heading Procedure 1.

Procedure 2. Tablets corresponding to about 500 mg. of the pure mercury compound were placed in a 50-ml. test-tube with glass stopper. 25 ml. of water, 0·5 ml. of bromine and 1 ml. of concentrated hydrochloric acid were added. The tube was stoppered and shaken to obtain a good mixing of the reagents, and then it was allowed to stand for 10 minutes. 2 g. of granulated zinc, 20 mesh, was added and the joint of the tube was rinsed with 3 ml. of glacial acetic acid, which was allowed to flow into the tube. The tube was then shaken strongly in a laboratory shaker during 1 hour. The mixture was decanted from the amalgam, the amalgam was washed, dissolved and titrated as described in Procedure 1. A series of results obtained according to this procedure is given in Table I under the heading Procedure 2.

TABLE I

RECOVERIES OF MERCURY

Procedure 1		Procedure 2	
Sample	Recovery per cent.	Sample	Recovery per cent.
30 tablets (303·5 mg. Hg)	99·9	Pure chloromercurimethoxypropyl-carbamide, 488·7 mg. (267·3 mg. Hg)	99·3
" " "	97·6		
" " "	98·9	Pure chloromercurimethoxypropyl-carbamide, 767·2 mg. (419·6 mg. Hg)	99·2
" " "	97·2		
" " "	96·6	Pure chloromercurimethoxypropyl-carbamide, 577·5 mg. (315·8 mg. Hg) + indifferentia corresponding to 30 tablets	99·8
" " "	98·4		
		Pure chloromercurimethoxypropyl-carbamide, 815·0 mg. (445·7 mg. Hg) + indifferentia corresponding to 30 tablets	99·9
		30 tablets (303·5 mg. Hg)	99·8
		" "	99·8
		" "	99·9

Procedure 2 appears to give more accurate and more reliable results, than Procedure 1, in which the results probably are affected by losses of mercury through volatilisation.

Amalgamation without the addition of bromine gave acceptable results (97 to 99 per cent. recovery) with pure 3-chloromercuri-2-methoxypropyl-carbamide, but quite unacceptable results (70 to 80 per cent. recovery) with the tablet mixture. The compound is hydrolysed rather quickly in acid solution.

It appears that amalgamation from oxidising solution should be a useful method for the separation of mercury also in many other instances.

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