X, 23. Tin.—*Discussion.* In acid solution, toluene-3,4-dithiol (**dithiol**) forms a red compound when warmed with stannous salts (compare Molybdenum, Section **X, 18**). Stannic tin also reacts, but more slowly than stannous tin; thioglycollic acid may be employed to reduce tin(IV) to tin(II). The reagent is not stable, being easily reduced, and hence should be prepared as required. A dispersant, such as Lorol or Teepol X, is generally added to the solution under test.

Many heavy metals react with dithiol to give coloured precipitates, *e.g.*, bismuth, iron(III), copper, nickel, cobalt, silver, mercury, lead, cadmium, arsenic, etc.; molybdate and tungstate also react. Of the various interfering elements, only arsenic distils over with the tin when a mixture is distilled from a medium of concentrated sulphuric acid and concentrated hydrobromic acid in a current of carbon dioxide. If arsenic is present in quantities larger than that of the tin it should be removed.

Reagents. *Dithiol reagent.* Dissolve 0·1 g. of dithiol in 2·5 ml. of 5*M*-sodium hydroxide solution. Add 0·5 ml. of thioglycollic acid, and dilute to 50 ml. with water. Prepare fresh daily.

Lorol solution. Prepare a 1 per cent aqueous solution of sodium lauryl sulphate.

Standard tin solution. Dissolve 1·000 g. of A.R. tin in 100 ml. of 1 : 1 hydrochloric acid and dilute with the same concentration of acid to 1 litre: 1 ml. contains 1 mg. of Sn. Prepare more dilute solutions as required (*e.g.*, 0·01 mg. Sn per ml.) by dilution with 1 : 1 hydrochloric acid.

Procedure. Transfer a 10-ml. aliquot of the sample solution, which should be 0·5*N* in hydrochloric acid and contain not more than 0·25 mg. of tin, to a 20-ml. volumetric flask, and add in the order given 1 drop of thioglycollic acid, 2·0 ml. of concentrated hydrochloric acid, 0·5 ml. of the Lorol solution, and 1·0 ml. of the dithiol reagent with thorough mixing after each addition. Place the flask in a water bath at 60° C. for 10 minutes, cool, and dilute the contents to the mark. Measure the optical density at 545 mμ against a reagent blank.

Construct a titration curve with the aid of the standard tin solution.

Procedure (**tin in canned foods**). The procedure provides for the removal of interfering copper by the addition of diethylammonium diethyldithiocarbamate in chloroform reagent.*

Weigh 5 or 10 g. of the sample, depending on the expected tin content, into a small porcelain crucible. Dry and char the sample on a hot plate; heat to ash in a muffle furnace at 600° C. Add 1 g. of fusion mixture (3 parts Na_2CO_3 + 1 part KCN by weight) and fuse this with the ash by holding the crucible with nickel tongs over a Bunsen or Meker burner. Cool the crucible, place it in a small beaker, and cover the latter with a watch glass. Add 10 ml. of water, and run 10 ml. of dilute hydrochloric acid (1 : 1) cautiously into the crucible (*Fume Cupboard!*) Boil the contents of the beaker gently for 30 minutes. Cool and filter: wash the beaker, crucible, and filter with water.

If copper is known to be absent or present only in negligible proportions, dilute the solution with water to 50 ml. in a volumetric flask, and continue as detailed below. Otherwise, transfer the solution to a small separatory funnel and add 5 ml. of the diethylammonium diethyldithiocarbamate in chloroform reagent (diluted (1 + 20) with chloroform when required). Shake and run off the chloroform layer, extract the aqueous layer with successive 1-ml. portions of the reagent until the chloroform layer is colourless; finally, wash the aqueous layer with a few ml. of chloroform. Dilute the aqueous solution with water to 50 ml. in a volumetric flask.

To 10·0 ml. of the solution thus prepared add 0·5 ml. of dilute hydrochloric acid (1 : 1) and proceed as above. Measure the absorption at 545 mμ, or use an Ilford No. 604 green filter with an absorptiometer.

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