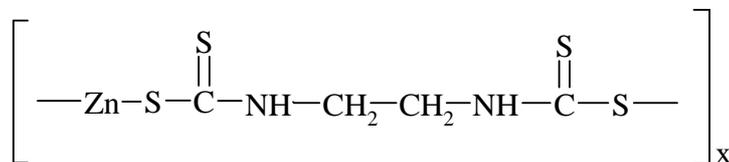


ZINEB
25



<i>ISO common name</i>	Zineb
<i>Chemical name</i>	Zinc ethylenebis(dithiocarbamate) (IUPAC); [1,2-ethanediy]bis(dithiocarbamato)(2-)zinc (CA; 12122-67-7)
<i>Empirical formula</i>	C ₄ H ₆ N ₂ S ₄ Zn
<i>RMM</i>	275.7
<i>m.p.</i>	Decomposes
<i>v.p.</i>	Less than 10 × 10 ⁻⁵ Pa
<i>Solubility</i>	In water 10 mg/l. Slightly soluble in pyridine; practically insoluble in most common solvents
<i>Description</i>	Light-coloured powder
<i>Stability</i>	Somewhat unstable to light, heat and moisture
<i>Formulations</i>	Wettable powders, waterdispersible granules and dustable powders

ZINEB TECHNICAL
***25/TC/M/-**

1 Sampling. Take at least 200 g.

2 Identity tests.

2.1 CS₂ evolution. MT 153, CIPAC F, p 360

2.2 Spot test. MT 130, CIPAC F, p 320

2.3 UV absorption test. MT 165, CIPAC F, p 411

2.4 Identification of amines MT 152, CIPAC F, p 357

2.5 Zinc MT 154, CIPAC F, p 361

* CIPAC method 1964; revised 1967.

3 Zineb. As for maneb technical **61/TC/M/3** except:

(c) *Calculation*

$$\text{Zineb content} = \frac{137.9 \times N \times (t - b)}{w} \text{ g/kg}$$

4 Zinc. As for mancozeb **34/1/M/7.4**, CIPAC H, p 195.

5 Arsenic

SCOPE

The method is suitable for the determination of arsenic in products containing more than 100 mg/kg of arsenic.

OUTLINE OF METHOD Arsenic is liberated from an acid solution of the sample by nascent hydrogen. The arsenic is absorbed in silver diethyldithiocarbamate/pyridine reagent and determined spectrophotometrically.

REAGENTS

Hydrochloric acid density 1.18

Hydrochloric acid density 1.10

Nitric acid. Dilute nitric acid (1 volume density 1.42) with distilled water (2 volumes).

Potassium iodide solution 50% w/v in distilled water

Silver diethyldithiocarbamate RE 44. Dissolve 0.5 g in pyridine (100 ml); store away from light. Discard after two days.

Tin(II)chloride 2aq. 33% w/v solution in hydrochloric acid (80 ml of density 1.18 and 20 ml water)

Sulphuric acid density 1.84. Dilute 25% by volume (density 1.84) in distilled water.

Zinc shot 16 to 30 mesh.

Standard arsenic solutions:

Stock solution. Dissolve arsenic trioxide (1.33 g) in the minimum volume of sodium hydroxide c (Na OH) = 5 mol/l (about 4 ml) and dilute to 100 ml with distilled water (1 ml contains 10 mg As).

Standard solution. Dilute the stock solution (10 ml) to 100 ml with arsenic-free distilled water (1 ml contains 1 mg As).

Zinc sulphate low in arsenic

Lead acetate wool. Soak cotton wool in 20% lead acetate solution and dry in warm air.

Note: All reagents should be analytical quality and should be arsenic free.

APPARATUS

Volumetric flasks, 100 ml

Kjeldahl flasks, 300 ml

Kjeldahl rack

Test tubes, internal diameter 1.3 cm, length 15 cm, graduated at 10 ml

Conical flask, 100 ml

Gutzeit apparatus. See Fig. 4. Fisons Scientific Apparatus Ltd. apparatus, modified by sealing 5 cm of glass tube of a suitable diameter to the top of the reagent tube.

Spectrophotometer

PROCEDURE

(a) *Digestion of sample*. Accurately weigh not more than 1 g of the well mixed sample with an approximately known zinc content and transfer to the Kjeldahl flask. Add dilute nitric acid (10 ml) and as soon as any initial reaction subsides, heat gently until further vigorous reaction ceases, and then cool the mixture. Partially immerse the flask in cold water and add, gradually, the sulphuric acid (5 ml of density 1.84), at such a rate as not to cause excessive frothing or heating. Heat the flask gently over a small flame until the flask is free of brown fumes, or the first signs of charring, in which case add more nitric acid, slowly, in small portions (1 ml of density 1.42), heating after each addition until oxidation of organic matter is complete. Do not heat so strongly that charring is excessive or loss of arsenic will occur. Continue heating until copious white fumes of sulphuric acid are evolved. Allow to cool, add distilled water (10 ml) and boil again to white fumes of sulphuric acid. Repeat this procedure with a further 5 ml of distilled water. Finally, cool, dilute to about 50 ml with distilled water, transfer, quantitatively, to a 100 ml volumetric flask, make up to volume at 10 °C.

(b) *Preparation of calibration graph*. Accurately weigh 2.2 g quantities of zinc sulphate (0.5 g Zn) and transfer to Kjeldahl flasks. To each sample add portions of the standard arsenic solution (0.5 to 4 mg arsenic). Add water to adjust the aqueous volume to 7 ml and then add 3.3 ml of nitric acid (density 1.42). Digest the mixture as described for the samples. Transfer portions (1 ml) containing from 5 to 40 µg arsenic to conical flasks, pre-reduce and transfer to the Gutzeit apparatus for reduction to arsine, as described below, and construct a calibration curve.

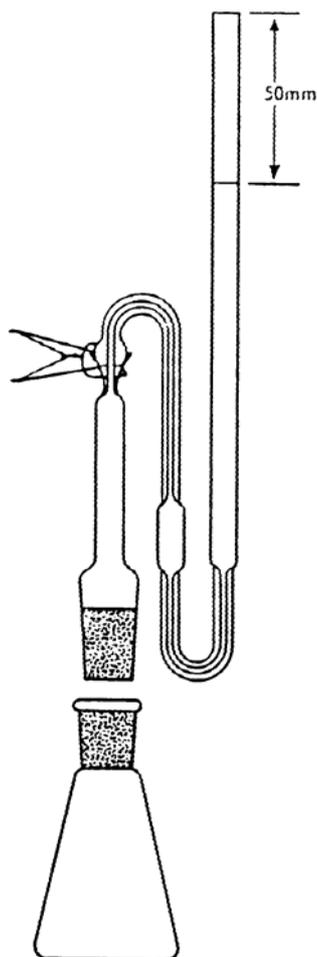


Fig. 4 Gutzeit apparatus showing modified reagent tube.

(c) *Determination.* Transfer an aliquot containing about 5 to 30 μg of arsenic but not more than 0.025 g of zinc (normally 5 ml) to the 100 ml conical flask, add potassium iodide (5 ml), and swirl to dissolve. Add tin(II)chloride solution (2 ml) and hydrochloric acid (10 ml), and make up the contents of the flask to about 50 ml, with distilled water.

Place a small wad of dry lead acetate wool in the end of the guard tube, and attach it to the reagent tube by means of the joint clip. Pipette 10 ml of the silver diethyldithiocarbamate solution into the reagent tube. Add zinc shot (10 g) quickly to the contents of the flask, and quickly place the assembled bubbler in position. Check that the joints are gas tight.

Allow the reaction to proceed for 45 min at room temperature. If the lead acetate treated cotton wool plug is markedly stained, low recoveries of arsenic will be found. Low recoveries of arsenic also occur if nitric acid or other oxidizing agents are present. Nitric oxides produce a transient blue colour with reagent. At the end of the reaction time, disconnect the bubbler at the ball joint and tilt to and fro to dissolve any complex on the capillary walls and to mix the reagent thoroughly.

Immediately measure the absorbance of the solution in a 1 cm cell at a wavelength of 540 nm and deduct the reagent "blank absorbency" obtained, by repeating the whole procedure but omitting the sample.

Calculate the quantity of arsenic present in the sample by reference to a calibration graph.

If more than 30 µg arsenic per aliquot are found, repeat the reduction procedure with a smaller aliquot. If present in the sample, 1000 mg/kg antimony would be reported as less than 20 mg/kg arsenic by this procedure. The recovery of arsenic from different samples should be checked, similarly.

ZINEB WETTABLE POWDERS

*25/WP/M/-

1 Sampling. Take at least 500 g.

2 Identity tests. As for zineb technical 25/TC/M/2.

3 Zineb. As for zineb technical 25/TC/M/3.

4 Zinc. As for mancozeb technical 34/1/M/7.4, CIPAC H, *p* 195.

5 Arsenic. As for zineb technical 25/TC/M/5.

6 Suspensibility. As for maneb wetttable powders 61/WP/M/5, except: Mass of zineb in the 25 ml of suspension = $0.1379 \times N \times (t - b)$ g.

ZINEB WATER DISPERSIBLE GRANULES

*25/WG/M/-

1 Sampling. Take at least 500 g.

2 Identity tests. As for zineb technical 25/TC/M/2.

3 Zineb. As for zineb technical 25/TC/M/3.

4 Zinc. As for mancozeb technical 34/1/M/7.4, CIPAC H, *p* 195.

5 Arsenic. As for zineb technical 25/TC/M/5.

* CIPAC method 1964; revised 1967.

6 Suspensibility. As for maneb wettable powders 61/WP/M/5, except: Mass of zineb in the 25 ml of suspension = $0.1379 \times N \times (t - b)$ g.

ZINEB DUSTABLE POWDERS

*25/DP/M/-

1 Sampling. Take at least 1 kg.

2 Identity tests. As for zineb technical 25/TC/M/2.

3 Zineb. As for zineb technical 25/TC/M/3.

4 Zinc. As for mancozeb technical 34/1/M/7.4, CIPAC H, p 195.

5 Arsenic. As for zineb technical 25/TC/M/5.

6 Suspensibility. As for maneb wettable powders 61/WP/M/5, except: Mass of zineb in the 25 ml of suspension = $0.1379 \times N \times (t - b)$ g.

* CIPAC method 1964; revised 1967.