

INTERNATIONAL STANDARD



3549

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Zinc dust pigment for paints

Poussière de zinc pour peintures

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up has the right to be represented on that Committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 3549 was drawn up by Technical Committee ISO/TC 35, *Paints and varnishes*, and circulated to the Member Bodies in October 1974.

It has been approved by the Member Bodies of the following countries :

Austria	Israel	Spain
Brazil	Netherlands	Sweden
Bulgaria	New Zealand	Switzerland
Czechoslovakia	Poland	Turkey
France	Portugal	United Kingdom
Iran	Romania	Yugoslavia
Ireland	South Africa, Rep. of	

The Member Body of the following country expressed disapproval of the document on technical grounds :

Germany

Zinc dust pigment for paints

0 INTRODUCTION

This International Standard deals with zinc dust pigment for use in protective coatings. The material complying with this International Standard is quite suitable for the usual types of zinc paint but it does not necessarily cover the requirements of industries other than the paint industry. The possibility of including a requirement for particle size has been carefully considered, and it has been decided to include a clause that particle size distribution limits may be specified but both the method and limits should be agreed between the interested parties. With regard to the analytical methods given in this International Standard, more up-to-date methods such as atomic absorption methods are currently being developed by Technical Committee ISO/TC 18, *Zinc and zinc alloys*, and Technical Committee ISO/TC 35; when these are finalized, they may be included in any future revision. Meanwhile, such methods may be used by agreement between the interested parties.

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies requirements for zinc dust pigment manufactured by a distillation process, suitable for use in protective coatings. It does not cover zinc pigments of other physical form such as zinc flake, as this would make the document unduly complex.

2 REFERENCES

ISO 713, *Zinc — Determination of lead and cadmium contents — Polarographic method.*

ISO 714, *Zinc — Determination of iron content — Photometric method.*

ISO 787/XVIII, *General methods of test for pigments — Part XVIII: Determination of residue on sieve by a mechanical flushing procedure.*

ISO 842, *Raw materials for paints and varnishes — Sampling.*

ISO 2590, *General method for the determination of arsenic — Silver diethyldithiocarbamate photometric method.*

3 REQUIRED CHARACTERISTICS AND THEIR TOLERANCES

Zinc dust pigment for paints shall have the characteristics shown in table 1.

4 RESIDUE ON SIEVE AND PARTICLE SIZE [OPTIONAL]

The material shall meet the sieve residue requirements shown in table 2 when examined by the method described in ISO 787/XVIII, or other methods as agreed between the interested parties. A 50 g test portion should be used for the test.

If required, mean particle size or the particle size distribution may be specified but both the limits and the method shall be agreed between the interested parties.

TABLE 1 — Composition of zinc dust pigment

Characteristic	Requirement % (m/m)	Test method
Total zinc content expressed as zinc (Zn)	98 min.	Clause 6
Metallic zinc content (Zn)	94 min.	Clause 7 or 8; the particular method to be agreed between the interested parties
Lead (Pb)	0,2 max.	ISO 713 and clause 9 ¹⁾
Cadmium (Cd)	0,2 max.	ISO 713 and clause 9 ¹⁾
Iron (Fe)	0,2 max.	ISO 714 and clause 10 ¹⁾
Arsenic (As)	0,000 4 (4 mg/kg) max.	ISO 2590 and clause 11
Matter insoluble in acid	0,2 max.	Clause 12

1) or other suitable methods such as atomic absorption methods, as agreed between the interested parties.

NOTE — If the zinc oxide content is required, this should be calculated from the difference between the total zinc content and the metallic zinc content.

TABLE 2 — Residue on sieve requirements

Nominal size of sieve aperture μm	Residue on sieve % max.
125	0
90	0,1
45	3,0

NOTE — For certain uses, zinc dust is required with lower residues on the 90 and 45 μm sieves than those shown in table 2. In these cases the residue on sieve limits should be agreed between the interested parties.

5 SAMPLING

A representative sample of the material shall be taken in accordance with ISO 842.

NOTE — It is pointed out that the sample should on no account be dried before testing and any portion of the sample not used should not be returned to the sample container.

METHODS OF TEST

During the analysis, use only reagents of recognized analytical reagent grade, and only distilled water or water of equivalent purity.

6 DETERMINATION OF TOTAL ZINC CONTENT

6.1 Reagents

6.1.1 Hydrochloric acid, ρ 1,18 g/ml.

6.1.2 Hydrochloric acid, 1 + 4 solution.

Dilute 200 ml of the hydrochloric acid (6.1.1) to 1 l with water.

6.1.3 Sulphuric acid, 1 + 1 solution.

Slowly add 1 part by volume of sulphuric acid, ρ 1,84 g/ml, to 1 part by volume of water.

6.1.4 Nitric acid, ρ 1,42 g/ml.

6.1.5 Ammonia solution, ρ 0,880 g/ml.

6.1.6 Buffer solution.

Dissolve 200 g of hydroxylammonium chloride ($\text{NH}_2\text{OH}\cdot\text{HCl}$) in approximately 300 ml of water. Dissolve 28 g of sodium hydroxide in approximately 300 ml of water. Combine the two solutions, cool and dilute to 1 l with water.

6.1.7 Ethylenedinitrilotetraacetic acid (EDTA), disodium salt, dihydrate, solution, zinc factor T , in g Zn/ml, i.e. 1 ml of disodium EDTA solution will form a complex with T g of zinc.

Dissolve about 20 g of disodium EDTA, dihydrate, in water and make up to 1 l with water. Store in a polyethylene bottle.

Standardize against a 0,05 M zinc sulphate solution.

6.1.8 Xylenol orange, 1 g/l indicator solution of the sodium salt in water.

6.1.9 Bromothymol blue, 1 g/l indicator solution in ethanol 96 % (V/V).

6.2 Procedure

6.2.1 Test portion

Weigh, to the nearest 0,001 g, 1,5 g of the sample into a 250 ml beaker.

6.2.2 Determination

Dissolve the test portion in 20 ml of the hydrochloric acid (6.1.1) and add 1 or 2 ml of the nitric acid (6.1.4) to ensure that any lead present is dissolved. Add 5 ml of the sulphuric acid (6.1.3) and evaporate until strongly fuming.

Cool, then add 100 ml of water, boil for a short time and allow to stand until the following day.

Filter the solution through a sintered glass or silica crucible; wash the crucible, collecting the filtrate in a 500 ml one-mark volumetric flask. Make the solution up to the mark and mix thoroughly.

By means of a pipette, transfer 50 ml of the solution to a 500 ml conical flask; add 200 ml of water and 3 drops of the bromothymol blue indicator (6.1.9), then add ammonia solution (6.1.5), drop by drop, until the colour turns blue. Then add the hydrochloric acid (6.1.2) until a yellow colour returns. Then add 20 ml of the buffer solution (6.1.6) and 3 drops of the xylenol orange indicator (6.1.8) and titrate with the disodium EDTA solution (6.1.7) until the red colour changes to pale yellow.

6.3 Expression of results

Calculate the total zinc content, as a percentage by mass, by the formula

$$\frac{1\,000\,VT}{m}$$

where

m is the mass, in grams, of the test portion;

T is the zinc factor, in grams per millilitre, of the disodium EDTA solution;

V is the volume, in millilitres, of the disodium EDTA solution used.

Report the result to one decimal place.

7 DETERMINATION OF METALLIC ZINC BY THE GAS EVOLUTION METHOD

7.1 Reagents

7.1.1 Hydrochloric acid/copper(I) chloride reagent.

Dissolve 0,05 g of copper(I) chloride (Cu_2Cl_2) in 700 ml of hydrochloric acid, ρ 1,18 g/ml, and dilute to 1 000 ml with water.

7.1.2 Solution for levelling bottle.

Acidify some water with a few drops of hydrochloric acid and add sufficient methyl orange solution to render the colour of the water in the apparatus easily visible.

7.2 Apparatus

7.2.1 Gas measuring apparatus, consisting of a water-jacketed gas burette of capacity 400 ml, graduated from 300 ml, and a water-cooled reaction flask of capacity 400 ml. Any suitable apparatus may be used, but the figure illustrates one convenient type.

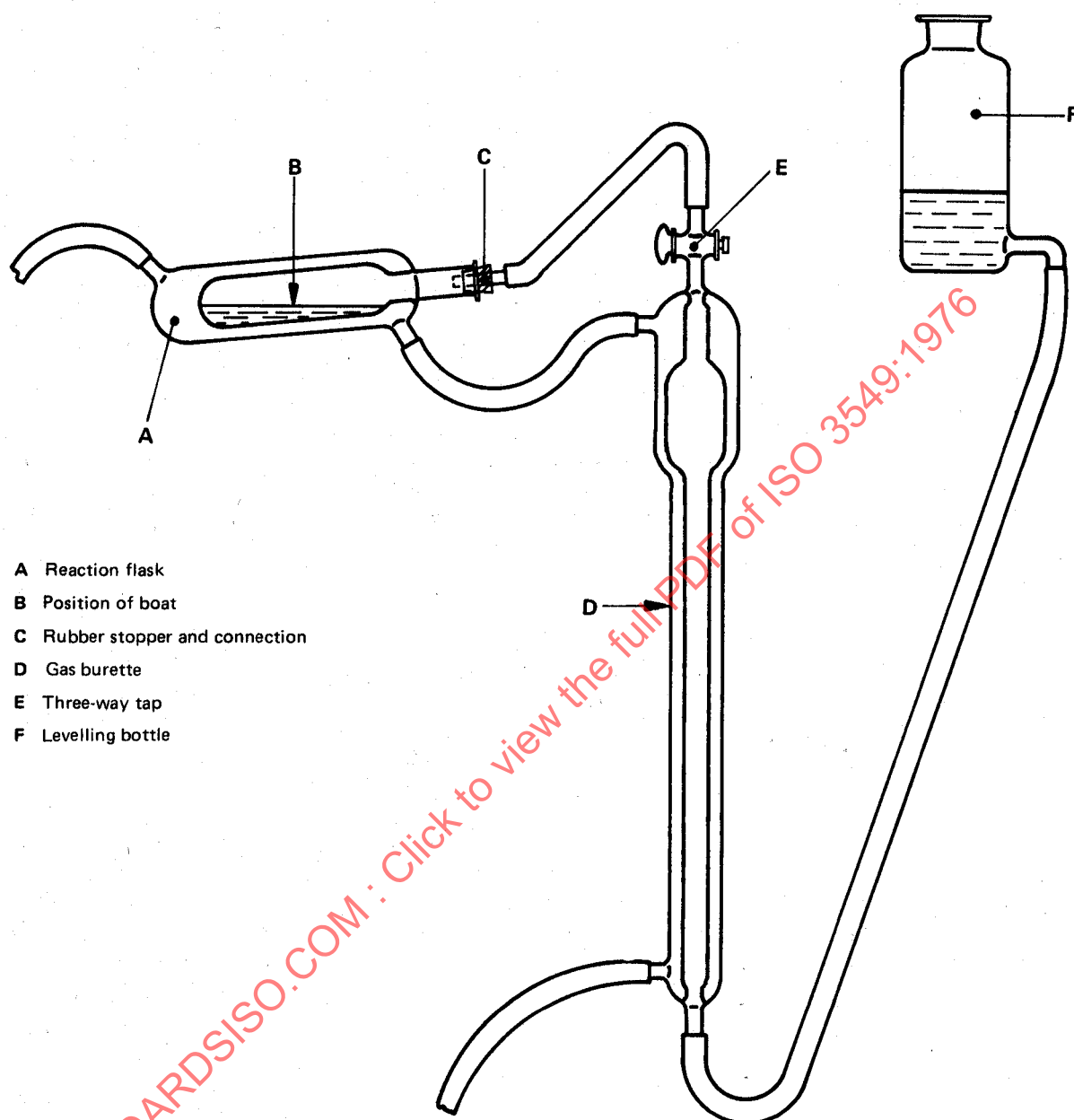


FIGURE — Suitable apparatus for the determination of metallic zinc by the gas evolution method

7.2.2 Boat, of a suitable plastics material or porcelain, with lid.

7.3 Procedure

NOTE — For convenience, the procedure given below refers to the apparatus illustrated in the figure. Other suitable apparatus may require modification of the procedure described.

7.3.1 Test portion

Weigh, to the nearest 0,001 g, about 1 g of the sample into the boat and cover with the lid.

7.3.2 Determination

Pass a steady flow of tap water through the two water jackets. Introduce 30 ml of the hydrochloric acid/copper(I) chloride reagent (7.1.1) into the reaction flask (A) and clamp it in an almost horizontal position. Introduce the covered boat into the neck of the flask. Take care that the zinc does not come in contact with the acid.

Fit the connecting stopper (C) to the flask (A). After the circulating tap water has been running long enough to establish a constant temperature, raise the level of the water in the gas burette (D) by opening the three-way tap (E) to atmosphere and raising the levelling bottle (F) until the level is at a convenient height (about eye level).

Turn the three-way tap to connect flask (A) with the gas burette and move the levelling bottle until the liquids in the tube and bottle are at the same level. Keep the bottle steady until the tap is turned to atmosphere.

Raise the bottle and expel all the air from the gas burette. Turn the tap to connect the flask with the gas burette and place the bottle on the bench.

Take the flask out of the clamp and allow the boat to drop into the acid. Shake the flask well until the reaction has virtually ceased. When the volume of gas evolved becomes constant and the temperature returns to the initial value, level off the liquids in the gas burette and bottle and record the volume V .

Record the barometric pressure and the temperature of the cooling water. Correct the barometer reading for the temperature of the brass scale (only valid for a special barometer) by using table 3. Read the saturated water vapour pressure at the temperature of the cooling water from table 4.

PRECAUTIONARY NOTES

1 Careful initial and vigorous final shakings of the reaction flask are essential.

2 Check that the apparatus is air-tight by opening the tap to connect the flask (A), levelling the solution in the levelling bottle, stoppering the bottle and lowering it. The level of the solution in the gas burette should remain constant.

TABLE 3 — Barometer corrections for brass scales

Temperature °C	Barometer reading, kPa (mmHg)						
	82,6 (620)	86,6 (650)	90,6 (680)	93,3 (700)	97,3 (730)	101,3 (760)	104,0 (780)
1	0,01 (0,1)	0,01 (0,1)	0,01 (0,1)	0,01 (0,1)	0,01 (0,1)	0,01 (0,1)	0,01 (0,1)
2	0,03 (0,2)	0,03 (0,2)	0,03 (0,2)	0,03 (0,2)	0,03 (0,2)	0,03 (0,2)	0,04 (0,3)
3	0,04 (0,3)	0,04 (0,3)	0,04 (0,3)	0,04 (0,3)	0,05 (0,4)	0,05 (0,4)	0,05 (0,4)
4	0,05 (0,4)	0,05 (0,4)	0,05 (0,4)	0,07 (0,5)	0,07 (0,5)	0,07 (0,5)	0,07 (0,5)
5	0,07 (0,5)	0,07 (0,5)	0,08 (0,6)	0,08 (0,6)	0,08 (0,6)	0,08 (0,6)	0,08 (0,6)
6	0,08 (0,6)	0,08 (0,6)	0,09 (0,7)	0,09 (0,7)	0,09 (0,7)	0,09 (0,7)	0,11 (0,8)
7	0,09 (0,7)	0,09 (0,7)	0,11 (0,8)	0,11 (0,8)	0,11 (0,8)	0,12 (0,9)	0,12 (0,9)
8	0,11 (0,8)	0,12 (0,9)	0,12 (0,9)	0,12 (0,9)	0,13 (1,0)	0,13 (1,0)	0,13 (1,0)
9	0,12 (0,9)	0,12 (0,9)	0,13 (1,0)	0,13 (1,0)	0,14 (1,1)	0,14 (1,1)	0,14 (1,1)
10	0,13 (1,0)	0,14 (1,1)	0,14 (1,1)	0,14 (1,1)	0,16 (1,2)	0,16 (1,2)	0,17 (1,3)
11	0,14 (1,1)	0,16 (1,2)	0,16 (1,2)	0,17 (1,3)	0,17 (1,3)	0,18 (1,4)	0,18 (1,4)
12	0,16 (1,2)	0,17 (1,3)	0,17 (1,3)	0,18 (1,4)	0,18 (1,4)	0,20 (1,5)	0,20 (1,5)
13	0,17 (1,3)	0,18 (1,4)	0,18 (1,4)	0,20 (1,5)	0,21 (1,6)	0,21 (1,6)	0,23 (1,7)
14	0,18 (1,4)	0,20 (1,5)	0,20 (1,5)	0,21 (1,6)	0,23 (1,7)	0,23 (1,7)	0,24 (1,8)
15	0,20 (1,5)	0,21 (1,6)	0,23 (1,7)	0,23 (1,7)	0,24 (1,8)	0,25 (1,9)	0,25 (1,9)
16	0,21 (1,6)	0,23 (1,7)	0,24 (1,8)	0,24 (1,8)	0,25 (1,9)	0,27 (2,0)	0,27 (2,0)
17	0,23 (1,7)	0,24 (1,8)	0,25 (1,9)	0,25 (1,9)	0,27 (2,0)	0,28 (2,1)	0,29 (2,2)
18	0,24 (1,8)	0,25 (1,9)	0,27 (2,0)	0,28 (2,1)	0,28 (2,1)	0,29 (2,2)	0,31 (2,3)
19	0,25 (1,9)	0,27 (2,0)	0,28 (2,1)	0,29 (2,2)	0,31 (2,3)	0,32 (2,4)	0,32 (2,4)
20	0,27 (2,0)	0,28 (2,1)	0,29 (2,2)	0,31 (2,3)	0,32 (2,4)	0,33 (2,5)	0,33 (2,5)
21	0,28 (2,1)	0,29 (2,2)	0,31 (2,3)	0,32 (2,4)	0,33 (2,5)	0,35 (2,6)	0,36 (2,7)
22	0,29 (2,2)	0,31 (2,3)	0,32 (2,4)	0,33 (2,5)	0,35 (2,6)	0,36 (2,7)	0,37 (2,8)
23	0,31 (2,3)	0,32 (2,4)	0,33 (2,5)	0,35 (2,6)	0,36 (2,7)	0,39 (2,9)	0,39 (2,9)
24	0,32 (2,4)	0,33 (2,5)	0,36 (2,7)	0,36 (2,7)	0,39 (2,9)	0,40 (3,0)	0,41 (3,1)
25	0,33 (2,5)	0,35 (2,6)	0,37 (2,8)	0,39 (2,9)	0,40 (3,0)	0,41 (3,1)	0,43 (3,2)
26	0,35 (2,6)	0,36 (2,7)	0,39 (2,9)	0,40 (3,0)	0,40 (3,0)	0,43 (3,2)	0,44 (3,3)
27	0,36 (2,7)	0,37 (2,8)	0,40 (3,0)	0,41 (3,1)	0,41 (3,1)	0,45 (3,4)	0,45 (3,5)
28	0,37 (2,8)	0,40 (3,0)	0,41 (3,1)	0,43 (3,2)	0,44 (3,4)	0,47 (3,5)	0,48 (3,6)
29	0,39 (2,9)	0,41 (3,1)	0,43 (3,2)	0,44 (3,3)	0,47 (3,5)	0,48 (3,6)	0,49 (3,7)
30	0,40 (3,0)	0,43 (3,2)	0,44 (3,3)	0,45 (3,4)	0,48 (3,6)	0,49 (3,7)	0,51 (3,8)

The value given in the table shall be subtracted from the barometric pressure reading if the temperature of the gas in the gas burette is above room temperature and added to the reading if the temperature is below room temperature.

TABLE 4 — Saturated water vapour pressures, kPa (mmHg)

Temperature °C	0,0	0,1	0,2	0,3	0,4	0,5	0,6	0,7	0,8	0,9
0	0,61 (4,6)	0,61 (4,6)	0,61 (4,6)	0,62 (4,7)	0,62 (4,7)	0,62 (4,7)	0,63 (4,8)	0,63 (4,8)	0,63 (4,9)	0,63 (4,9)
1	0,65 (4,9)	0,66 (5,0)	0,66 (5,0)	0,66 (5,0)	0,67 (5,1)	0,67 (5,1)	0,67 (5,1)	0,69 (5,2)	0,69 (5,2)	0,71 (5,3)
2	0,71 (5,3)	0,71 (5,3)	0,72 (5,4)	0,72 (5,4)	0,72 (5,4)	0,73 (5,5)	0,73 (5,5)	0,75 (5,6)	0,75 (5,6)	0,75 (5,6)
3	0,76 (5,7)	0,76 (5,7)	0,77 (5,8)	0,77 (5,8)	0,77 (5,8)	0,78 (5,9)	0,78 (5,9)	0,80 (6,0)	0,80 (6,0)	0,83 (6,2)
4	0,81 (6,1)	0,81 (6,1)	0,83 (6,2)	0,83 (6,2)	0,84 (6,3)	0,84 (6,3)	0,85 (6,4)	0,85 (6,4)	0,87 (6,5)	0,87 (6,5)
5	0,87 (6,5)	0,88 (6,6)	0,88 (6,6)	0,89 (6,7)	0,89 (6,7)	0,90 (6,8)	0,90 (6,8)	0,92 (6,9)	0,92 (6,9)	0,93 (7,0)
6	0,93 (7,0)	0,95 (7,1)	0,95 (7,1)	0,96 (7,2)	0,96 (7,2)	0,97 (7,3)	0,97 (7,3)	0,99 (7,4)	0,99 (7,4)	1,00 (7,5)
7	1,00 (7,5)	1,01 (7,6)	1,01 (7,6)	1,03 (7,7)	1,04 (7,8)	1,04 (7,8)	1,05 (7,9)	1,05 (7,9)	1,05 (7,9)	1,07 (8,0)
8	1,07 (8,0)	1,08 (8,1)	1,09 (8,2)	1,09 (8,2)	1,11 (8,3)	1,11 (8,3)	1,12 (8,4)	1,12 (8,4)	1,13 (8,5)	1,15 (8,6)
9	1,15 (8,6)	1,16 (8,7)	1,16 (8,7)	1,17 (8,8)	1,17 (8,8)	1,19 (8,9)	1,20 (9,0)	1,20 (9,0)	1,21 (9,1)	1,21 (9,1)
10	1,23 (9,2)	1,24 (9,3)	1,24 (9,3)	1,25 (9,4)	1,27 (9,5)	1,27 (9,5)	1,28 (9,6)	1,29 (9,7)	1,29 (9,7)	1,31 (9,8)
11	1,31 (9,8)	1,32 (9,9)	1,33 (10,0)	1,33 (10,0)	1,35 (10,1)	1,36 (10,2)	1,36 (10,2)	1,37 (10,3)	1,39 (10,4)	1,39 (10,4)
12	1,40 (10,5)	1,41 (10,6)	1,43 (10,7)	1,43 (10,7)	1,44 (10,8)	1,45 (10,9)	1,45 (10,9)	1,47 (11,0)	1,48 (11,1)	1,49 (11,2)
13	1,49 (11,2)	1,51 (11,3)	1,52 (11,4)	1,53 (11,5)	1,53 (11,5)	1,55 (11,6)	1,56 (11,7)	1,57 (11,8)	1,57 (11,8)	1,59 (11,9)
14	1,60 (12,0)	1,61 (12,1)	1,61 (12,1)	1,62 (12,2)	1,64 (12,3)	1,65 (12,4)	1,66 (12,5)	1,66 (12,5)	1,68 (12,6)	1,69 (12,7)
15	1,71 (12,8)	1,72 (12,9)	1,73 (13,0)	1,73 (13,0)	1,75 (13,1)	1,76 (13,2)	1,77 (13,3)	1,79 (13,4)	1,80 (13,5)	1,80 (13,5)
16	1,81 (13,6)	1,83 (13,7)	1,84 (13,8)	1,85 (13,9)	1,86 (14,0)	1,87 (14,1)	1,89 (14,2)	1,91 (14,3)	1,91 (14,3)	1,92 (14,4)
17	1,93 (14,5)	1,95 (14,6)	1,96 (14,7)	1,97 (14,8)	1,99 (14,9)	2,00 (15,0)	2,01 (15,1)	2,03 (15,2)	2,04 (15,3)	2,05 (15,4)
18	2,07 (15,5)	2,08 (15,6)	2,09 (15,7)	2,11 (15,8)	2,12 (15,9)	2,13 (16,0)	2,15 (16,1)	2,16 (16,2)	2,17 (16,3)	2,19 (16,4)
19	2,20 (16,5)	2,21 (16,6)	2,23 (16,7)	2,24 (16,8)	2,25 (16,9)	2,27 (17,0)	2,28 (17,1)	2,29 (17,2)	2,31 (17,3)	2,32 (17,4)
20	2,33 (17,5)	2,35 (17,6)	2,36 (17,7)	2,39 (17,9)	2,40 (18,0)	2,41 (18,1)	2,43 (18,2)	2,44 (18,3)	2,45 (18,4)	2,47 (18,5)
21	2,49 (18,7)	2,51 (18,8)	2,52 (18,9)	2,53 (19,0)	2,55 (19,1)	2,56 (19,2)	2,57 (19,3)	2,60 (19,5)	2,61 (19,6)	2,63 (19,7)
22	2,64 (19,8)	2,65 (19,9)	2,68 (20,1)	2,69 (20,2)	2,71 (20,3)	2,72 (20,4)	2,75 (20,6)	2,76 (20,7)	2,77 (20,8)	2,79 (20,9)
23	2,81 (21,1)	2,83 (21,2)	2,84 (21,3)	2,87 (21,5)	2,88 (21,6)	2,89 (21,7)	2,91 (21,8)	2,93 (22,0)	2,95 (22,1)	2,96 (22,2)
24	2,99 (22,4)	3,00 (22,5)	3,03 (22,7)	3,04 (22,8)	3,05 (22,9)	3,08 (23,1)	3,10 (23,2)	3,10 (23,2)	3,13 (23,5)	3,15 (23,6)

7.4 Expression of results

Calculate the metallic zinc content, as a percentage by mass, by the formula

$$\frac{1,048 V (p_b - p_s)}{m (t + 273)}$$

where

m is the mass, in grams, of the test portion;

p_b is the barometric pressure, in kilopascals;

p_s is the saturated water vapour pressure, in kilopascals, at t °C;

t is the temperature, in degrees Celsius, of the cooling water;

V is the volume, in millilitres, of hydrogen liberated.

Report the result to one decimal place.

8 DETERMINATION OF METALLIC ZINC BY THE OXIDIMETRIC METHOD

8.1 Principle

Iron(III) sulphate solution and a solution of a copper salt (used as a catalyst) are added to the test portion. The zinc goes into solution as zinc sulphate, and the resulting iron(II) sulphate is estimated with a standard volumetric potassium permanganate solution.

8.2 Reagents

8.2.1 Carbon dioxide, in cylinder.

8.2.2 Orthophosphoric acid, at least 85 % (m/m).

8.2.3 Sulphuric acid, 1 + 19 solution.

Slowly add 1 part by volume of sulphuric acid, ρ 1,84 g/ml, to 19 parts by volume of water.

8.2.4 Copper(II) sulphate solution.

Dissolve 200 g of copper(II) sulphate pentahydrate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) in 1 l of water.

8.2.5 Iron(III) sulphate solution.

Dissolve, as far as possible, 330 g of iron(III) sulphate in 1 l of cold water. Heat until dissolution is complete. Allow to cool to room temperature, then filter.

8.2.6 Potassium permanganate, standard volumetric solution.

Prepare as follows :

Weigh 20 g of potassium permanganate (KMnO_4) into a 3 or 4 l flask and add 2 l of water. Boil for 1 h, allow to cool and stand until the following day.

Decant into a 2 000 ml one-mark volumetric flask and make up to the mark. Thoroughly mix the solution, allow to settle and decant through a filter of glass wool or a sintered glass filter into a brown glass stock bottle fitted with a glass stopper.

Standardize the potassium permanganate solution before use by the following method :

Weigh, to the nearest 0,001 g, 0,72 g of sodium oxalate (which has been dried for 1 h at 120°C) into a 500 ml flask. Dissolve in approximately 200 ml of the sulphuric acid solution (8.2.3).

Heat to boiling point and titrate with the potassium permanganate solution until a pink colour just appears.

Carry out the titration three times. Calculate the mean of the three masses of sodium oxalate used and the mean of the three titrations. Use these means as m_1 and V_2 in the final calculation (8.4).

8.3 Procedure

8.3.1 Test portion

Take a dry 750 ml conical flask and fill it with carbon dioxide. Stopper the flask carefully with a rubber bung. Weigh, to the nearest 0,001 g, about 0,4 g of the sample and quickly wash it into the flask with 4 or 5 ml of water from a water jet. Immediately replace the rubber bung. Prevent the formation of agglomerates as far as possible.

8.3.2 Determination

Add to the flask 10 ml of the copper(II) sulphate solution (8.2.4) and shake vigorously for about 1 min to prevent the formation of agglomerates. Then, using a pipette, wash down the neck of the flask to remove any metallic particles with 50 ml of the iron(III) sulphate solution (8.2.5) which has previously been deoxygenated by passing through it a stream of carbon dioxide (8.2.1) for 10 min.

Carry out the procedures in 8.3.1 and 8.3.2 as rapidly as possible and ensure that the flask is kept stoppered as much as possible.

Allow the flask to stand, shaking frequently until dissolution is complete. This takes from 15 to 30 min.

When dissolution is complete, add 20 ml of the orthophosphoric acid (8.2.2) and 200 ml of the sulphuric acid solution (8.2.3) and titrate immediately with the potassium permanganate solution (8.2.6) until a faint pink colour is obtained.

Carry out a blank test omitting the test portion.

8.4 Expression of results

Calculate the metallic zinc content, as a percentage by mass, by the formula

$$\frac{48,79 \times m_1 (V_0 - V_1)}{m_2 \times V_2}$$

where

m_1 is the mass, in grams, of sodium oxalate used in the standardization;

m_2 is the mass, in grams, of the test portion;

V_0 is the volume, in millilitres, of potassium permanganate solution used in the determination;

V_1 is the volume, in millilitres, of potassium permanganate solution used in the blank test;

V_2 is the volume, in millilitres, of potassium permanganate used in standardizing.

Report the result to one decimal place.

9 DETERMINATION OF LEAD AND CADMIUM

Use the method given in ISO 713 with the following modifications to sub-clauses 7.2 and 7.3 of that document :

[7.2] Plotting of the calibration curve

Prepare a calibration curve as follows :

[7.2.1] Into a series of 100 ml beakers, transfer 25 ml of the zinc chloride solution (4.10) corresponding to 5 g of metallic zinc.

Add 2,5 ml, 5 ml and 10 ml respectively of the standard solution of lead and cadmium No. 1 (4.7), corresponding to 0,05, 0,10 and 0,20 % of lead and cadmium. Additional calibration points may be used if desired.

[7.2.2] Evaporate to a syrupy consistency and proceed as outlined in 7.3.3 to 7.3.5, then polarograph as outlined in 7.4.

[7.3] Determination

[7.3.4] Transfer to a 100 ml volumetric flask and dilute to the mark.

10 DETERMINATION OF IRON

Use the method given in ISO 714 with the following modification to sub-clause 7.1 of that document :

[7.1] Test portion

Weigh 5 g of the test sample to the nearest 0,01 g.

11 DETERMINATION OF ARSENIC

Determine the arsenic content in accordance with ISO 2590, but modify the following clauses of that document :

[6.1] Test portion and preparation of the test solution

Transfer 5 g, weighed to the nearest 0,01 g, of the zinc dust into the conical flask (5.1.1) and add 25 ml of the hydrochloric acid (4.1). After dissolution of the zinc, dilute to approximately 40 ml with water.

[7] EXPRESSION OF RESULTS

Replace the second paragraph by the following :

Calculate the arsenic content, as a percentage by mass, by the formula

$$\frac{m_1}{m_2} \times 100$$

where

m_1 is the mass, in grams, of arsenic in the test solution;

m_2 is the mass, in grams, of the test portion.

If m_1 is expressed in micrograms, the formula becomes

$$\frac{m_1}{m_2} \times 10^{-4}$$

If the arsenic content is to be expressed in milligrams per kilogram (parts per million), and m_1 is expressed in micrograms, then the formula becomes

$$\frac{m_1}{m_2}$$

12 DETERMINATION OF MATTER INSOLUBLE IN ACID

12.1 Reagents

12.1.1 Hydrochloric acid, 1 + 9 solution.

Dilute 100 ml of hydrochloric acid, ρ 1,18 g/ml, with 900 ml of water.

12.1.2 Nitric acid, ρ 1,42 g/ml.

12.2 Apparatus

Normal laboratory apparatus and glassware, and in particular :

12.2.1 Clock glass, fitting on a 2 000 ml beaker.

12.2.2 Sintered glass filter crucible, porosity grade P 40 (pore size index 16 to 40 μ m).

12.3 Procedure

12.3.1 Test portion

Weigh, to the nearest 0,1 g, 20 g of the sample.

12.3.2 Determination

Place the test portion (12.3.1) in a 2 000 ml beaker with 1 000 ml of the hydrochloric acid (12.1.1). Cover with the clock glass (12.2.1) and heat on a hot-plate at $80 \pm 2^\circ\text{C}$ for 24 h.

Add 2 ml of the nitric acid (12.1.2) and boil for 1 h.

Filter through the tared filter (12.2.2), wash the filter thoroughly, dry it at $110 \pm 2^\circ\text{C}$ and weigh.

12.4 Expression of results

Calculate the content of matter insoluble in acid, as a percentage by mass, by the formula

$$\frac{m_1}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of the residue.

Report the result to one decimal place.

13 TEST REPORT

The test report shall contain at least the following information :

- a reference to this International Standard or a corresponding national standard;
- the type and identification of the product tested;
- the results of the tests, and whether or not the product complies with the relevant specification limits;
- any deviation, by agreement or otherwise, from the procedures specified;
- the date of the test.