



SLOVENSKI STANDARD SIST ISO/TR 11422:2002

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Železne rude -- Priporočene postopke za raztopitev železnih rud s pomočjo
kislote ali alkalije

Iron ores -- Recommended procedures for iron ore dissolution using either acid digestion
or alkali fusion

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Minerais de fer -- Méthodes recommandées de dissolution des minerais de fer par
digestion acide ou fusion alcaline

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Železove rude

Iron ores

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TECHNICAL REPORT

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Iron ores — Recommended procedures for iron ore dissolution using either acid digestion or alkali fusion

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de fer par digestion acide ou fusion alcaline*

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The main task of technical committees is to prepare International Standards, but in exceptional circumstances a technical committee may propose the publication of a Technical Report of one of the following types:

- type 1, when the required support cannot be obtained for the publication of an International Standard, despite repeated efforts;
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- type 2, when the subject is still under technical development or where for any other reason there is the future but not immediate possibility of an agreement on an International Standard;
- type 3, when a technical committee has collected data of a different kind from that which is normally published as an International Standard (“state of the art”, for example).

Technical Reports of types 1 and 2 are subject to review within three years of publication, to decide whether they can be transformed into International Standards. Technical Reports of type 3 do not necessarily have to be reviewed until the data they provide are considered to be no longer valid or useful.

ISO/TR 11422, which is a Technical Report of type 3, was prepared by Technical Committee ISO/TC 102, *Iron ores*, Subcommittee SC 2, *Chemical analysis*.

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Iron ores — Recommended procedures for iron ore dissolution using either acid digestion or alkali fusion

1 Background

From time to time in ISO/TC 102/SC 2, the view had been expressed that some degree of unification of procedures for the dissolution of iron ore test portions, based on selected established SC 2 procedures, would be of considerable benefit in the development of new analytical methods. This objective, coupled with the need to investigate possible procedure changes when increasing the mass of test portion in trace element methods, led to formation of the Dissolution Study Group in 1982.

The activities of this Study Group and the details and results of an interlaboratory dissolution test were reported in document ISO/TC 102/SC 2 N772 E (annexes A and B to this report). Based on these results and consideration of certain dissolution procedures already in use in SC 2, several proposed dissolution procedures for various test portion masses were presented in document ISO/TC 102/SC 2 N887 E, constituting a consolidated study group report for the period 1982 to 1986.

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Before these procedures could be finalized as acceptable models for use in future analytical methods, certain discrepancies of detail in the conduct of acid digestions had to be resolved. Accordingly, SC 2 members' opinions were invited in the form of an explanatory note and questionnaire (documents ISO/TC 102/SC 2 N886 E and N888 E). A summary of the replies is appended (annex C), from which the conclusions to be drawn are as follows:

- a) In acid digestions, the beaker should be totally covered; in evaporations it should be partially covered.
- b) Temperatures (obtained in a test beaker) should be, for the initial digestion, 95°C and, for subsequent digestion and evaporation, 105°C.
- c) The evaporation to dehydrate silica should continue to dryness with 15 min further heating.
- d) The fused residue should be leached in the crucible, and not in the reserved main solution.

These conclusions have been incorporated in the procedures presented in this report. It is not possible to recommend only one method for acid digestion, because of the slightly different conditions required for varying test portion masses. Accordingly, methods are proposed for 2 g, 1 g and 0,5 g test portions, based on SC 2 methods applicable to or using such test portions.

An important conclusion from the interlaboratory test was that not all of the parameters temperature, time of heating and target volume can be specified simultaneously. The test results showed that, if target volume needs to be specified, temperature and target volume take precedence, and heating time should be given only as a guide.

2 Sources of the recommended procedures

Acid digestion	4.1.1	2 g test portion	:	ISO 9685: 1991,
"	4.1.2	1 g test portion	:	ISO 9685: 1991,
"	4.1.3	0.5 g test portion	:	ISO 9682: 1991,
Alkali fusion	4.2.1		:	ISO 9682: 1991,
"	4.2.1		:	ISO 4687: 1991,
"	4.2.2		:	ISO 9683: 1991,
Alkali sinter fusion	4.3.1		:	ISO 7834: 1987,
"	4.3.2		:	ISO 9683: 1991,

NOTE 1 To take into account the survey responses and other considerations, the recommended procedures differ in minor detail from those in the above sources.

3 Assessment of acid digestion vs alkali fusion or sinter fusion Alkali fusion and sinter fusion processes have the distinct advantage of providing one-step dissolution which does not require time-consuming residue treatment; hence they are the first choice wherever possible. They have the major disadvantage, however, of adding very materially to the salt concentration of the solution, imposing difficulties with atomic absorption and ICP measurement techniques. To address this problem, the "8-4-5" low flux ratio procedure (4.2.1) has been devised to provide the lowest flux-to-sample ratio so far available in iron ore dissolution (2,4 flux to 1 test portion). Where the method can permit a separation of the fusion salts from the analyte, or where spectrophotometric techniques are involved and the salt concentration is accordingly less relevant, the flux ratio adopted in 4.3.1 (3 flux to 1 test portion) and in 4.2.2 (4 flux to 1 test portion) can then become acceptable.

Alkali fusion or sinter fusion is, in general, limited to test portion masses not greater than 1 g. Where trace element determinations requiring 2 g test portions are involved, acid digestion tends to be preferred. However, it would not be impossible to devise an alkali sinter fusion method with a 2 g test portion and 6 g of sodium peroxide if separation of the analyte from the high concentration of salts could be contemplated.

4 Recommended procedures

4.1 Acid digestion procedure

4.1.1 2 g test portion

A Reagents

During the procedure, use only water of a grade that complies with ISO 3696:1987, *Water for analytical laboratory use—Specification and test methods*.

A.1 Sodium carbonate (Na_2CO_3), anhydrous.

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A.2 Sodium tetraborate ($\text{Na}_2\text{B}_4\text{O}_7$), anhydrous.

A.3 Hydrochloric acid, ρ 1,16 g/ml to 1,19 g/ml.

A.4 Hydrochloric acid, ρ 1,16 g/ml to 1,19 g/ml, diluted 1 + 1.

A.5 Hydrochloric acid, ρ 1,16 g/ml to 1,19 g/ml, diluted 2 + 100.

A.6 Nitric acid, ρ 1,4 g/ml.

A.7 Sulfuric acid, ρ 1,84 g/ml.

A.8 Sulfuric acid, ρ 1,84 g/ml, diluted 1 + 1.

A.9 Hydrofluoric acid, ρ 1,13 g/ml, 40% (m/m), or ρ 1,19 g/ml, 48% (m/m).

B Apparatus

Ordinary laboratory equipment, and

B.1 Glass beakers, 250 ml capacity.

B.2 Platinum crucibles, minimum capacity 20 ml.

B.3 Hotplate, calibrated using a partial-immersion thermometer, to produce in a 10 mm depth of sulfuric acid (A.7) in a 250 ml test beaker, temperatures of 95°C and 105°C.

C Procedure

Transfer a 2,0 g test portion to a 250 ml beaker (B.1) and moisten with water. Add 50 ml of hydrochloric acid (A.3), cover with a watch glass and heat on a hotplate (B.3) for about 1 h at a temperature of 95°C.

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NOTE 2 If after this digestion the amount of insoluble residue is high, raise the hotplate temperature to about 105°C and continue heating, avoiding boiling.

Add 5 ml of nitric acid (A.6) and 0,2 ml of sulfuric acid (A.8), and digest for 15 min at 105°C. Displace the cover to provide a 6 mm gap and evaporate the solution at the 105°C hotplate setting to dryness. Continue heating for a further 15 min.

NOTE 3 If the test sample contains significant amounts of barium, omit the addition of sulfuric acid.

Add 30 ml of hydrochloric acid (A.4) and heat to dissolve the salts. Add 20 ml of water and mix, then wash the watch glass and wall of the beaker and filter the solution through a close-texture filter paper containing filter pulp (0,3 g to 0,4 g dry mass) into a 200 ml beaker. Carefully remove all adhering particles with a rubber-tipped glass rod or piece of moistened filter paper and transfer to the filter. Wash the paper with hydrochloric acid (A.5) until visibly free from iron, then finally wash with three or four portions of warm water. Reserve the filtrate and washings as the main solution.

Transfer the filter paper and residue to a platinum crucible (B.2). Dry and char the paper at a low temperature, then ignite at 750°C to 800°C. Allow the crucible to cool. Add 0,3 ml of sulfuric acid (A.8) and 5 ml of hydrofluoric acid (A.9), evaporate slowly to remove silicon dioxide, then continue heating to remove the sulfuric acid. Ignite at 800°C for several

minutes and cool. Add 0,8 g of sodium carbonate (A.1) and 0,4 g of sodium tetraborate (A.2) and mix. Heat at 1000°C in a muffle furnace or over a pressurized air burner for a period sufficient to produce a clear melt, then allow the crucible to cool.

Add 10 ml of hydrochloric acid (A.4) and heat to dissolve the melt and expel carbon dioxide. Cool, combine the solution with the evaporated and cooled main solution, transfer to a 100 ml volumetric flask, dilute to volume and mix. (This is the test solution.)

4.1.2 1 g test portion

A Reagents

During the analysis use only water of a grade that complies with ISO 3696:1987.

A.1 Sodium carbonate (Na_2CO_3), anhydrous.

A.2 Sodium tetraborate ($\text{Na}_2\text{B}_4\text{O}_7$), anhydrous.

A.3 Hydrochloric acid, ρ 1,16 g/ml to 1,19 g/ml.

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A.4 Hydrochloric acid, ρ 1,16 g/ml to 1,19 g/ml, diluted 1 + 1.

A.5 Hydrochloric acid, ρ 1,16 g/ml to 1,19 g/ml, diluted 2 + 100.

A.6 Nitric acid, ρ 1,4 g/ml.

A.7 Sulfuric acid, ρ 1,84 g/ml.

A.8 Sulfuric acid, ρ 1,84 g/ml, diluted 1 + 1.

A.9 Hydrofluoric acid, ρ 1,13 g/ml, 40% (m/m), or ρ 1,19 g/ml, 48% (m/m).

B Apparatus

Ordinary laboratory equipment, and

B.1 Glass beakers, 250 ml capacity.

B.2 Platinum crucibles, minimum capacity 20 ml.

B.3 Hotplate, calibrated using a partial-immersion thermometer, to produce in a 10 mm depth of sulfuric acid (A.7) in a 250 ml test beaker, temperatures of 95°C and 105°C.

C Procedure

Transfer a 1,0 g test portion to a 250 ml tall-form beaker (B.1) and moisten with water. Add 25 ml of hydrochloric acid (A.3), cover with a watch glass and heat on a hotplate (B.3) for about 1 h at a temperature of 95°C.

NOTE 4 If after this digestion the amount of insoluble residue is high, raise the hotplate temperature to about 105°C and continue heating, avoiding boiling.

Add 2 ml of nitric acid (A.6) and 0,2 ml of sulfuric acid (A.8), and digest for 15 min at 105°C. Displace the cover to provide a 6 mm gap and evaporate the solution at the 105°C hotplate setting to dryness. Continue heating for a further 15 min.

NOTE 5 If the test sample contains significant amounts of barium, omit the addition of sulfuric acid.

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Add 20 ml of hydrochloric acid (A.4) and heat to dissolve the salts. Add 20 ml of water and mix, then wash the watch glass and wall of the beaker and filter the solution through a close-texture filter paper containing filter pulp (0,3 g to 0,4 g dry mass) into a 200 ml beaker. Carefully remove all adhering particles with a rubber-tipped glass rod or piece of moistened filter paper and transfer to the filter. Wash the paper with hydrochloric acid (A.5) until visibly free from iron, then finally wash with three or four portions of warm water. Reserve the filtrate and washings as the main solution.

Transfer the filter paper and residue to a platinum crucible (B.2). Dry and char the paper at a low temperature, then ignite at 750°C to 800°C. Allow the crucible to cool. Add 0,3 ml of sulfuric acid (A.8) and 5 ml of hydrofluoric acid (A.9), evaporate slowly to remove silicon dioxide, then continue heating to remove the sulfuric acid. Ignite at 800°C for several minutes and cool. Add 0,8 g of sodium carbonate (A.1) and 0,4 g of sodium tetraborate (A.2) and mix. Heat at 1000°C in a muffle furnace or over a pressurized air burner for a period sufficient to produce a clear melt, then allow the crucible to cool.