INTERNATIONAL STANDARD

ISO 3262-18

Second edition
2023-07

Extenders — Specifications and methods of test —

Part 18: Precipitated sodium aluminium silicate

Matières de charge — Spécifications et méthodes d'essai —
Partie 18: Silicoaluminate de sodium précipité

Reference number
ISO 3262-18:2023(E)

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

Page

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 procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

ISO draws attention to the possibility that the implementation of this document may involve the use of (a) patent(s). ISO takes no position concerning the evidence, validity or applicability of any claimed patent rights in respect thereof. As of the date of publication of this document, ISO had not received notice of (a) patent(s) which may be required to implement this document. However, implementers are cautioned that this may not represent the latest information, which may be obtained from the patent database available at www.iso.org/patents. ISO shall not be held responsible for identifying any or all such patent rights.

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 256, *Pigments, dyestuffs and extenders*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 298, *Pigments and extenders*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 3262-18:2000), which has been technically revised.

The main changes are as follows:

— the first part of the title has been changed to “Extenders”;

— the normative references have been updated.

A list of all parts in the ISO 3262 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Extenders — Specifications and methods of test —

Part 18:
Precipitated sodium aluminium silicate

1 Scope

This document specifies requirements and corresponding methods of test for precipitated sodium aluminium silicate.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 648, *Laboratory glassware — Single-volume pipettes*

ISO 787-2, *General methods of test for pigments and extenders — Part 2: Determination of matter volatile at 105 °C*

ISO 787-5, *General methods of test for pigments and extenders — Part 5: Determination of oil absorption value*

ISO 787-9, *General methods of test for pigments and extenders — Part 9: Determination of pH value of an aqueous suspension*

ISO 3262-1, *Extenders — Specifications and methods of test — Part 1: Introduction and general test methods*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 5794-1:2022, *Rubber compounding ingredients — Silica, precipitated, hydrated — Part 1: Non-rubber tests*

ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

ISO 18451-1, *Pigments, dyestuffs and extenders — Terminology — Part 1: General terms*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18451-1 and the following apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
precipitated sodium aluminium silicate
amorphous silicate precipitated by the reaction of sodium silicate solution with a mineral acid and/or an aluminium salt

4 Requirements and test methods

For precipitated sodium aluminium silicate complying with this document, the essential requirements are specified in Table 1 and the conditional requirements are listed in Table 2. The test methods listed in Tables 1 and 2 shall apply.

Table 1 — Essential requirements

Characteristic	Unit	Requirement	Test method
Silica content, min.	% mass fraction	75	See Clause 6 or 7
Oxide content — Na_2O		3 to 10	See Clause 7
— Al_2O_3		5 to 15	
Residue on 45 µm sieve, max.	% mass fraction	2	See Clause 8
Lightness, min.	%	95	Test method in preparation
Matter volatile at 105 °C, max.	% mass fraction	10	ISO 787-2
Loss on ignition	% mass fraction	3 to 12	ISO 3262-1
Oil absorption value [a], min.	g/100 g	70	ISO 787-5
pH value of aqueous suspension	—	7 to 12	ISO 787-9

[a] A test method with higher reproducibility and repeatability is described in ASTM D2414. However, the results cannot be compared directly with oil absorption values determined in accordance with ISO 787-5.

Table 2 — Conditional requirements

Characteristic	Unit	Requirement	Test method
Particle size distribution (instrumental method)	% mass fraction	To be agreed between the interested parties	To be agreed between the interested parties
Specific surface area	m^2/g		ISO 5794-1:2022, Annex D

5 Sampling

Take a representative sample of the product to be tested, in accordance with ISO 15528.

6 Determination of silica content

6.1 Principle

A test portion is repeatedly treated with hydrochloric acid and evaporated to dryness. To render the dehydrated silicic acid thus formed as insoluble as possible, it is then heated for 2 h at (140 ± 5) °C. Any chlorides present are removed by extracting the precipitate with hot dilute hydrochloric acid.

The precipitate is ignited at 1 000 °C, giving impure silicon dioxide, which is treated with sulfuric and hydrofluoric acid. The silicon tetrafluoride formed is evaporated off and the silica content is calculated from the resulting loss in mass.

6.2 Reagents

Use only reagents of recognized analytical grade and only water of at least grade 3 purity as specified in ISO 3696.

6.2.1 Hydrochloric acid, CAS Registry Number®[1] 7647-01-0, concentrated, (approximately 32 % mass fraction, $\rho \approx 1{,}16$ g/ml).

6.2.2 Hydrochloric acid, CAS Registry Number® 7647-01-0, diluted 1 + 1.

Add 1 part by volume of concentrated hydrochloric acid (6.2.1) slowly to 1 part by volume of water.

6.2.3 Sulfuric acid, CAS Registry Number® 7664-93-9, diluted 1 + 1.

Add 1 part by volume of concentrated sulfuric acid, (approximately 96 % mass fraction, $\rho \approx 1{,}84$ g/ml), slowly to 1 part by volume of water.

6.2.4 Hydrofluoric acid, CAS Registry Number® 7664-39-3, concentrated, (approximately 40 % mass fraction, $\rho \approx 1{,}13$ g/ml).

6.3 Apparatus

Use ordinary laboratory apparatus and glassware, together with the following.

6.3.1 Dish

6.3.2 Platinum crucible

6.3.3 Water bath, capable of being maintained at 100 °C.

6.3.4 Infrared evaporator

6.3.5 Muffle furnace, capable of being maintained at (1 000 ± 20) °C.

6.3.6 Drying oven, capable of being maintained at (140 ± 5) °C.

6.3.7 Filter paper

The filter paper used for filtration of the silica shall be of such texture as to retain the smallest particles of precipitate and nevertheless permit rapid filtration. For example, it is possible to use Whatman No. 40 or 41 or Schleicher und Schüll No. 589/2 “Weißband”.[2]

6.3.8 Desiccator, containing magnesium perchlorate as desiccant.

6.3.9 Balance, with an accuracy of 0,000 1 g.

6.4 Procedure

6.4.1 Number of determinations

Carry out the determination in duplicate.

1) Chemical Abstracts Service (CAS) Registry Number® is a trademark of the American Chemical Society (ACS). This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

2) Whatman No. 40 or 41 or Schleicher und Schüll No. 589/2 "Weißband" are examples of suitable products available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of these products.

6.4.2 Test portion

Weigh, to the nearest 0,2 mg, approximately 1 g (m_0) of the sample (see Clause 5) into a dish (6.3.1).

6.4.3 Determination

Add slowly 20 ml of concentrated hydrochloric acid (6.2.1) and evaporate to dryness under the infrared evaporator (6.3.4). Add again 20 ml of concentrated hydrochloric acid and evaporate to dryness. Repeat this step once more. After the third evaporation, place the dish in the drying oven (6.3.6), maintained at (140 ± 5) °C, and leave for 2 h.

Remove the dish from the oven and allow to cool. Add 50 ml of 1 + 1 hydrochloric acid (6.2.2) to the residue in the dish and warm it for approximately 20 min on the water bath (6.3.3) at 100 °C. Filter through a suitable filter paper (6.3.7) and wash the residue on the filter with hot water until the washings are neutral.

Pour the filtrate and washings into the original dish and evaporate to dryness. Repeat this evaporation step another two times, adding each time 10 ml of concentrated hydrochloric acid to the residue. After the third evaporation, heat at (140 ± 5) °C for 2 h in the drying oven.

Add 20 ml of 1 + 1 hydrochloric acid to the residue in the dish and warm it for approximately 10 min on the water bath at 100 °C. Filter through a fresh filter paper and wash the residue on the filter with hot water until the washings are neutral.

If necessary, check the filtrate for any silicon which may have passed through the filter.

Place the two filter papers with the washed precipitates in the platinum crucible (6.3.2). Dry, reduce to ashes at low temperature, ignite in the muffle furnace (6.3.5) at (1 000 ± 20) °C to constant mass (this should take approximately 1 h) and allow to cool in the desiccator (6.3.8). Weigh the ignited precipitate to the nearest 0,2 mg (m_1).

Wet the ignited precipitate in the platinum crucible with 2 ml to 3 ml of water, add 1 ml of 1 + 1 sulfuric acid (6.2.3) and 15 ml of hydrofluoric acid (6.2.4) and evaporate to a syrup, taking care to avoid loss by spitting. Allow to cool and wash the sides down with small quantities of water. Then add a further 10 ml of hydrofluoric acid and evaporate to dryness. If the evaporation of the silicon tetrafluoride is not complete, add a further 10 ml of hydrofluoric acid and evaporate to dryness again.

Heat the residue until white fumes are no longer evolved, then ignite for 30 min in the muffle furnace at (1 000 ± 20) °C. Remove from the furnace, allow to cool in the desiccator and weigh to the nearest 0,2 mg (m_2).

6.4.4 Determination of the total loss on ignition

Weigh, to the nearest 0,2 mg, approximately 1 g (m_3) of the sample (see Clause 5) into a platinum crucible.

NOTE Weighing out the test portions for the determination of the silica content (see 6.4.2) and the total loss on ignition can be carried out at the same time.

Ignite the test portion to constant mass in the muffle furnace at (1 000 ± 20) °C (this should take approximately 2 h) and allow to cool in the desiccator. Weigh the ignited test portion to the nearest 0,2 mg (m_4).

Calculate the total loss on ignition w_{TLI}, expressed as a percentage by mass, using the Formula (1):

$$w_{TLI} = \frac{m_3 - m_4}{m_3} \times 100 \quad (1)$$

where

m_3 is the mass, expressed in grams, of the test portion before ignition;

m_4 is the mass, expressed in grams, of the ignited test portion.

Calculate the mean of the two determinations and report the result to the nearest 0,1 %.

6.5 Expression of results

Calculate the silica content $w(SiO_2)$, expressed as a percentage by mass, using the Formula (2):

$$w(SiO_2) = \frac{(m_1 - m_2)}{m_0 \times \left[1 - \frac{w_{TLI}}{100}\right]} \times 100 \quad (2)$$

where

m_0 is the mass, expressed in grams, of the test portion (see 6.4.2);

m_1 is the mass, expressed in grams, of the dehydrated impure silica after ignition at (1 000 ± 20) °C to constant mass (see 6.4.3);

m_2 is the mass, expressed in grams, of the silica after treatment with hydrofluoric acid and ignition to constant mass (see 6.4.3);

w_{TLI} is the total loss on ignition determined in 6.4.4.

Calculate the mean of the two determinations and report the result to the nearest 0,1 %.

6.6 Precision

6.6.1 Repeatability, *r*

The repeatability, *r*, is the value below which the absolute difference between two single test results, each the mean of duplicates, can be expected to lie when this method is used under repeatability conditions. In this case, the test results are obtained on identical material by one operator in one laboratory within a short interval of time. For this document, *r* is 0,6 %, with a 95 % probability.

6.6.2 Reproducibility, *R*

No reproducibility data are currently available.

7 Determination of the composition by spectrometry

7.1 Principle

This spectrometric method gives the aluminium oxide and sodium oxide contents as well as the aluminium silicate content, although the precision of the determination of the aluminium silicate content can be lower than that of the method described in Clause 6.

The elements aluminium, sodium and silicon are analysed using either flame atomic absorption spectrometry (FAAS) or inductively coupled plasma atomic emission spectrometry (ICP-AES), depending on which type of instrument is available in the laboratory.

The test solution is prepared by fusing a test portion of precipitated sodium aluminium silicate with lithium metaborate and dissolving the melt in a mixture of nitric acid and tartaric acid.

The advantages of the ICP-AES method include its wide dynamic range and multi-element capabilities. Both methods (FAAS and ICP-AES) are relative analytical techniques. For quantitative analytical results, both measurement techniques shall be calibrated using standard matching solutions.

7.2 Reagents and materials

Use only reagents of recognized analytical grade and only water of at least grade 3 purity as defined in ISO 3696.

7.2.1 Lithium metaborate, CAS Registry Number® 13453-69-5.

7.2.2 Acid mixture

Add 50 ml of nitric acid, CAS Registry Number® 7697-37-2, (approximately 65 % mass fraction, $\rho \approx 1{,}40$ g/ml) and then 17 g of tartaric acid, CAS Registry Number® 526-83-0, to 500 ml of water. After dissolution, make up to 1 000 ml with water and mix well.

7.2.3 Caesium chloride buffer solution

Dissolve 50 g of caesium chloride, CAS Registry Number® 7647-17-8, in approximately 500 ml of water and add 50 ml of 30 % mass fraction hydrochloric acid, CAS Registry Number® 7647-01-0, ($\rho \approx 1{,}15$ g/ml). Make up to 1 000 ml with water and mix well.

7.2.4 Standard stock solutions, containing 1,000 g/l of aluminium, sodium and silicon, respectively.

Store each solution in a fluorinated-polyethylene/polypropylene (FEP) bottle.

7.2.5 Matrix solution, free from aluminium, sodium and silicon.

Dissolve 2 g of lithium metaborate, CAS Registry Number® 13453-69-5, in 60 ml of the acid mixture (7.2.2) in a 200 ml polypropylene one-mark volumetric flask (7.3.7). Make up to 200 ml with water and mix well.

7.2.6 Acetylene (C_2H_2), CAS Registry Number® 74-86-2, commercial grade, in a steel cylinder.

7.2.7 Compressed air

7.2.8 Dinitrogen oxide (N_2O), CAS Registry Number® 10024-97-2, commercial grade, in a steel cylinder.

7.3 Apparatus

Use ordinary laboratory apparatus and glassware, together with the following.

7.3.1 Flame atomic absorption spectrometer (FAAS), suitable for measurements at the following wavelengths:

— 309,3 nm for aluminium (Al),

— 589,0 nm for sodium (Na),

— 251,6 nm for silicon (Si),

fitted with a suitable burner fed with:

— an N_2O/C_2H_2 mixture for Al and Si determinations,

— a C_2H_2/air mixture for Na determinations,

and also fitted with hollow-cathode lamps for the elements Al, Na and Si and a deuterium background corrector.

7.3.2 Inductively coupled plasma atomic emission spectrometer (ICP-AES), preferably with high resolution (≤0,01 nm), automatic control of all plasma operating functions and a computer-controlled signal compensation system.

7.3.3 Platinum crucible

7.3.4 Muffle furnace, capable of being maintained at (1 000 ± 20) °C.

7.3.5 Magnetic stirrer, with a stirrer bar coated with polytetrafluoroethylene (PTFE).

7.3.6 PTFE beaker with cover, volume approximately 250 ml and diameter approximately 6 cm.

7.3.7 200 ml one-mark volumetric flasks, made of polypropylene.

7.3.8 Pipettes, calibrated, of suitable volumes (see 7.4), complying with the requirements of ISO 648.

7.3.9 Balance, with an accuracy of 0,000 1 g.

7.4 Procedure

7.4.1 Preparation of standard matching solutions

Introduce, with pipettes (7.3.8), suitable volumes of the standard stock solutions (7.2.4) into a series of several 50 ml one-mark volumetric flasks. Add the required volume of matrix solution (7.2.5), make up to the mark with water and mix well. Prepare the blank matching solution by dilution of the required volume of matrix solution with water in a 50 ml one-mark volumetric flask.

The standard matching solutions for FAAS and ICP-AES analysis shall have the same matrix solution concentration as the test solution (see 7.4.4).

Each diluted standard matching solution shall be stored in FEP bottles and shall not be used for more than a few days.

In cases of samples with high concentrations of sodium or aluminium which require a larger dilution, add, for example, caesium chloride buffer solution (7.2.3) in accordance with the instrument manufacturer's instruction manual when using the FAAS method.

7.4.2 Spectrometric measurement

7.4.2.1 Spectrometric measurement using FAAS

Install the required burner. Adjust the flow of C_2H_2 (7.2.6) and N_2O (7.2.8) or compressed air (7.2.7) to suit the characteristics of the burner (see Table 3) and light the burner. Install the spectral source in the spectrometer (7.3.1) and optimize the conditions for the determination of the relevant element (Al, Na or Si). Adjust the instrument in accordance with the manufacturer's instructions in order to obtain the maximum absorbance. The test parameters are listed in Table 3.

Table 3 — FAAS test parameters

Element	Spectral resonance line nm	Flame	Measurement range µg of oxide/ml
Al	309,3	C_2H_2/N_2O	4 to 100
Na	589,0	C_2H_2/Air	0,05 to 0,8
Si	251,6	C_2H_2/N_2O	10 to 200

Set the scale expansion, if fitted, so that the standard matching solution with the highest concentration gives an absorbance of about 0,55.

Aspirate into the flame each of the standard matching solutions in ascending order of concentration. Repeat with the solution of lowest concentration to verify that the instrument is stable.

Aspirate water into the flame after each measurement.

7.4.2.2 Spectrometric measurement using ICP-AES

Introduce the standard matching solutions into the plasma using an air atomizer, having set the instrument to the manufacturer's recommended operating conditions and using spectral resonance lines as given in Table 4.

Table 4 — ICP-AES test parameters

Element	Spectral resonance line nm	Measurement range µg of oxide/ml
Al	308,22	1 to 100
Na	589,59	
Si	212,41	

7.4.3 Calibration graph

Correct each absorbance by subtracting the reading obtained from the blank matching solution and plot for each element a graph having the masses, in micrograms, of the element contained in 1 ml of standard matching solution as abscissae and the corresponding values of the absorbance as ordinates.

7.4.4 Preparation of the test solution

Carry out the determinations in duplicate.

Weigh, to the nearest 0,1 mg, approximately 200 mg (m_0) of the sample (see Clause 5) into a platinum crucible (7.3.3). Add 2 g of lithium metaborate (7.2.1) and fuse the mixture by heating for approximately 30 min at (1 000 ± 20) °C in the muffle furnace (7.3.4) until a clear melt is obtained. Allow the crucible to cool to room temperature and immerse it in 60 ml of the acid mixture (7.2.2) in a PTFE beaker (7.3.6). Place the stirrer bar of the magnetic stirrer (7.3.5) in the beaker and stir until complete dissolution is obtained (probably overnight). Remove the platinum crucible and the stirrer bar, rinse with water, transfer the solution to a 200 ml polypropylene volumetric flask (7.3.7), make up to the mark with water and measure using the FAAS method (7.4.2.1) or the ICP-AES method (7.4.2.2).

7.4.5 Determination

First, adjust the spectrometer as described in 7.4.2.1 or 7.4.2.2 and measure the absorbance of the blank matching solution (see 7.4.1) in the spectrometer (7.3.1 or 7.3.2). Then measure the absorbance of the test solution (see 7.4.4) three times and, afterwards, that of the blank matching solution again. Finally, redetermine the absorbance of the standard matching solution with the highest concentration in order to verify that the response of the apparatus has not changed.

If the absorbance of the test solution is higher than the absorbance of the standard matching solution with the highest concentration, dilute the test solution with a known volume of the required matrix solution (see 7.4.1). Note the dilution factor, *D*.

7.5 Expression of results

7.5.1 Concentration of Al, Na and Si

Read from the calibration graph (see 7.4.3) the Al, Na and Si concentrations corresponding to the absorbances measured.

7.5.2 Calculation of the oxide content for each element

Calculate the content of each oxide (Al_2O_3, Na_2O, SiO_2), w_{oxide}, expressed as a percentage by mass, using the Formula (3):

$$w_{oxide} = \frac{c_t \times V_0 \times D \times f}{m_0 \times \left[1 - \frac{w_{TLI}}{100}\right]} \times 100 \quad (3)$$

where

c_t is the concentration, expressed in micrograms per millilitre, of the element concerned (Al, Na or Si) in the solution;

V_0 is the volume, expressed in millilitres, of the test solution (200 ml);

D is the dilution factor (if applicable, see 7.4.5);

f is a conversion factor, for calculation of the oxide content rather than the content of the element, as given in Table 5;

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

w_{TLI} is the total loss on ignition as determined in 6.4.4.

Calculate the mean of the two determinations and report the result to the nearest 0,1 %.

Table 5 — Values of conversion factor *f*

Element	Oxide	Conversion factor
Al	Al_2O_3	1,889 5
Na	Na_2O	1,348
Si	SiO_2	2,139

7.6 Precision

7.6.1 Repeatability, *r*

The repeatability, *r*, is the value below which the absolute difference between two single test results, each the mean of duplicates, can be expected to lie when this method is used under repeatability conditions. In this case, the test results are obtained on identical material by one operator in one laboratory within a short interval of time using the standardized test method. In this document, *r* is 0,8 % for Al_2O_3; 0,3 % for Na_2O; and 1,7 % for SiO_2, each with a 95 % probability.

NOTE These values were obtained in preliminary tests.

7.6.2 Reproducibility, *R*

No reproducibility data are currently available.

8 Determination of residue on sieve

8.1 Principle

The extender under test is suspended in water and the suspension is poured onto a sieve. Water is sprayed onto the sieve by means of a spray head, gently dispersing any agglomerates and flushing the fine particles through the sieve. The residue on the sieve is dried and weighed.

NOTE The spray method described in this document involves medium-level agglomerate-dispersing forces; these are necessary because of the particular physical characteristics of precipitated sodium aluminium silicate.

8.2 Materials

8.2.1 Tap water, filtered, at a pressure of approximately 200 kPa above atmospheric pressure. The pressure shall be adjusted to give a flow rate of (13,5 ± 0,3) l/min from the spray head.

8.3 Apparatus

Use ordinary laboratory apparatus and glassware, together with the following.

8.3.1 Sieve, consisting of a metal frame and wire gauze made from phosphor bronze or stainless steel, mesh size 45 µm, diameter 200 mm.

Loss of suspension by splashing during the sieving procedure can be prevented by using a splash guard made of a cylindrical metal sheet made from phosphor bronze or stainless steel. The metal sheet shall have a height of approximately 200 mm and a diameter slightly less than 200 mm. It shall fit into the top of the sieve.

8.3.2 Sieve holder

8.3.3 Spray head, with a diameter of 60 mm, having 76 holes drilled with a diameter of (1,0 ± 0,1) mm.

8.3.4 Weighing bottle

8.3.5 Drying oven, capable of being maintained at (105 ± 2) °C.

8.3.6 Balance, with an accuracy of 0,000 1 g.

8.4 Procedure

8.4.1 Number of determinations

Carry out the determination in duplicate.

8.4.2 Test portion

Weigh, to the nearest 0,1 mg, a quantity (m_0) of the sample (see Clause 5) such that a sufficient residue is obtained on the sieve (8.3.1). Place this test portion in a suitable beaker. Generally, a test portion of 10 g to 100 g is necessary.