
**Carbonaceous materials used in the
production of aluminium — Pitch for
electrodes — Determination of C/H ratio in
the quinoline-insoluble fraction**

*Produits carbonés utilisés pour la production de l'aluminium — Brais pour
électrodes — Détermination du rapport C/H dans la fraction insoluble dans
la quinoléine*



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Contents

	Page
1	Scope 1
2	Normative references 1
3	Principle 1
4	Isolation of quinoline-insoluble material for analysis..... 2
4.1	Reagents 2
4.2	Apparatus 2
4.3	Sampling and preparation of the test sample..... 2
4.4	Procedure 2
4.5	Calculation and expression of results..... 5
5	Analysis of quinoline-insoluble material for carbon and hydrogen content..... 5
5.1	Reagents 5
5.2	Apparatus 5
5.3	Preparation of the apparatus..... 7
5.4	Procedure 9
5.5	Calculation and expression of results..... 9
5.6	Ash content 10
6	Precision 11
6.1	Repeatability..... 11
6.2	Reproducibility..... 11
7	Test report 11

Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 12979 was prepared by Technical Committee ISO/TC 47, *Chemistry*, Subcommittee SC 7, *Aluminium oxide, cryolite, aluminium fluoride, sodium fluoride, carbonaceous products for the aluminium industry*.

Carbonaceous materials used in the production of aluminium — Pitch for electrodes — Determination of C/H ratio in the quinoline-insoluble fraction

1 Scope

This International Standard describes a method for the determination of the atomic carbon/hydrogen ratio of quinoline-insoluble material isolated from coal-tar pitches. A procedure for the isolation of the quinoline-insoluble material in a form suitable for the analysis is described.

NOTE The isolation procedure is not an alternative to ISO 6971¹⁾, which is to be used in all cases where the amount of quinoline-insoluble matter present in a pitch is to be determined.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 565, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings.*

ISO 4788, *Laboratory glassware — Graduated measuring cylinders.*

ISO 6257, *Carbonaceous materials used in the production of aluminium — Pitch for electrodes — Sampling.*

3 Principle

The quinoline-insoluble material is isolated from a finely divided sample of the coal-tar pitch by digestion with hot quinoline followed by pressure-filtration through a membrane filter. The isolated material is washed on the filter with hot quinoline then with hot toluene and dried.

The carbon and hydrogen contents of the isolated quinoline-insoluble material are determined by combustion of a weighed aliquot portion of that material in a stream of oxygen and trapping and weighing the carbon dioxide and water which are formed. The atomic carbon/hydrogen ratio of the quinoline-insoluble material is calculated from its carbon and hydrogen contents and the relative atomic masses of carbon and hydrogen.

1) ISO 6971, *Carbonaceous materials for the production of aluminium — Pitch for electrodes — Determination of contents of quinoline-insoluble material.*

4 Isolation of quinoline-insoluble material for analysis

4.1 Reagents

WARNING — Refer to the reagent supplier's Health and Safety data sheets for the precautions which are to be taken for the safe use of quinoline and toluene.

4.1.1 Quinoline, of purity at least 97 %, freshly distilled, boiling between 235 °C and 237 °C at 0,101 3 MPa.

Store the distilled quinoline between 5 °C and ambient temperature in a stoppered bottle of dark glass. Use it within two weeks; after this time period either redistill or discard the quinoline.

4.1.2 Toluene, general purpose reagent grade.

4.1.3 Nitrogen or other inert gas, supplied via a two-stage pressure regulator with a maximum outlet pressure of 0,45 MPa.

4.2 Apparatus

WARNING — Before using pressure-filtration equipment ensure that it is provided with adequate safety devices and that the equipment has been tested to ensure compliance with relevant Health and Safety legislation.

Ordinary laboratory apparatus and the following.

4.2.1 Thermostatically controlled electric hotplate, capable of maintaining liquids in beakers at 70 °C to 80 °C.

4.2.2 Pressure-filtration apparatus, accepting filter discs of 47 mm diameter and designed for safe operation at up to 120 °C with applied gas pressures of up to 1 MPa. A typical pressure-filtration apparatus is shown in Figure 1.

4.2.3 Membrane filter discs, of polytetrafluoroethylene (PTFE), 47 mm in diameter and having a nominal pore size of 0,2 µm.

4.2.4 Hand-held hot-air blower, general-purpose laboratory type, typically of 500 W power rating and giving an air exit temperature of approximately 300 °C.

4.2.5 Test sieve, of 250 µm nominal aperture, conforming to the requirements of ISO 565, and having a lid and receiver.

4.2.6 Watch glass, approximately 70 mm in diameter, dried at 105 °C to 110 °C for 1 h and cooled in a desiccator.

4.2.7 Measuring cylinder, of borosilicate glass, of 50 ml capacity with pouring spout, conforming to the requirements of ISO 4788.

4.3 Sampling and preparation of the test sample

Prepare a representative sample of approximately 10 g of the pitch by the method described in ISO 6257. Grind the sample with a pestle and mortar until it all passes the test sieve (4.2.5).

4.4 Procedure

4.4.1 Weigh a 100 ml borosilicate glass beaker to the nearest 0,1 mg. Transfer approximately 1 g of the test sample (4.3) to the beaker and weigh it to the nearest 0,1 mg. Using the measuring cylinder (4.2.7), add 25 ml of the quinoline (4.1.1) and stir the mixture with a glass rod to break up any agglomerates. Cover the beaker with a watch glass and place it on the hotplate (4.2.1). Allow the mixture to digest at 70 °C to 80 °C for 20 min. During the

digestion stir the contents of the beaker at approximately 5 min intervals. While the digestion is proceeding, heat the liquid reservoir and filter-support assembly of the pressure-filtration apparatus (4.2.2; see Figure 1) to approximately 100 °C, either in an oven or by use of the hot-air blower (4.2.4).

When the digestion is approaching completion, weigh a membrane filter disc (4.2.3) to the nearest 0,1 mg and insert it into the heated filter-support assembly. Assemble the filtration unit ensuring that it is satisfactorily gas-tight.

NOTE The membrane filter disc tends to curl up when placed on the heated filter-support assembly. It is helpful to place the membrane filter disc and the silicone rubber O-ring seal which lies above it on to the heated filter-support assembly in a single operation, thus holding the membrane filter disc flat during the subsequent assembly of the apparatus.

4.4.2 Place the measuring cylinder (4.2.7) under the outlet of the pressure filtration-assembly. Transfer the digested mixture of pitch and quinoline from the beaker to the liquid reservoir of the pressure-filtration apparatus, using a small glass funnel to prevent any of the liquid from entering the gas line. Close the filter unit and pressurize it with the inert gas (4.1.3), gradually increasing the applied pressure until the filtrate begins to flow into the graduated receiver.

NOTE 1 The filtrate typically begins to flow when the applied gas pressure reaches 0,12 MPa to 0,14 MPa.

Allow the filtration to proceed using the minimum applied gas pressure which is consistent with a satisfactory flow rate. Use the volume of the collected filtrate as a guide to the progress of filtration. If filtration is slow, maintain the temperature of the liquid-containing part of the pressure-filtration apparatus at approximately 70 °C to 80 °C by use of the hot-air blower.

NOTE 2 The filtration rate which is achieved depends very markedly on the characteristics of the pitch being tested. Electrode binder pitches which have had little or no thermal polymerization usually give solutions which filter readily in under 1 min with an applied gas pressure of 0,13 MPa. However, solutions of pitches which have had extensive thermal treatment usually filter more slowly, with filtration times up to 1 h being common. In these cases, it is essential to keep the filter unit hot during filtration and occasional depressurization and repressurization of the filter unit may prove helpful. The use of applied pressures above approximately 0,2 MPa does not normally produce any useful improvement in the rate of filtration.

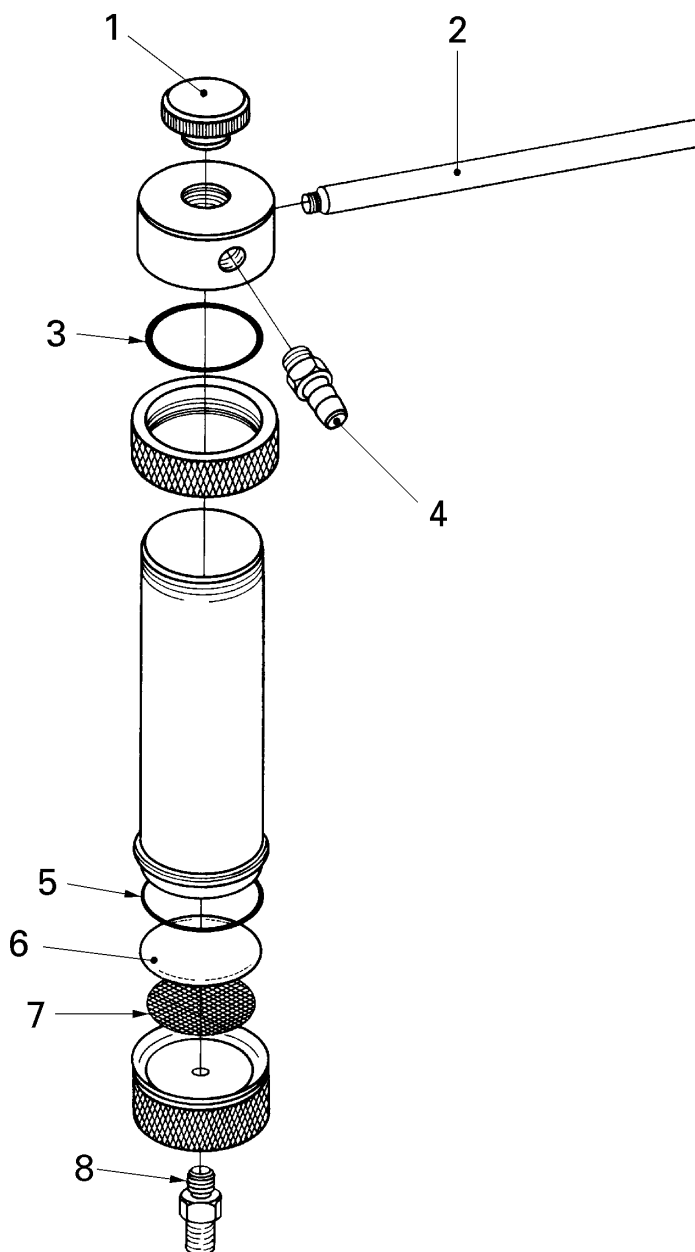
4.4.3 When the filtration of the quinoline solution is complete, depressurize the filtration apparatus, rinse the beaker with approximately 5 ml of quinoline at 70 °C to 80 °C and transfer the washings via the glass funnel to the filtration apparatus. Close the apparatus and gradually repressurize it until filtration commences. This will typically occur at approximately 0,1 MPa. Adjust the applied pressure to achieve a satisfactory flow rate. Repeat the rinsing of the beaker and the washing of the insoluble matter on the filter a further nine times using approximately 5 ml of quinoline at 70 °C to 80 °C for each operation, by which stage the washings passing into the graduated receiver should be very little darker than the distilled quinoline (4.1.1). If the washings are still deeply coloured, continue washing with further portions of hot quinoline until they show no further lightening of colour. Wash the insoluble matter in the pressure-filtration apparatus with ten successive approximately 5 ml portions of the toluene (4.1.2) at 70 °C to 80 °C, using an applied gas pressure reduced to approximately 0,01 MPa to 0,02 MPa to achieve a controllable flow rate. Allow the final washings to drain for 1 min to 2 min.

4.4.4 Weigh the watch glass (4.2.6) to the nearest 0,1 mg. Transfer the membrane filter disc from the pressure-filtration apparatus to the watch glass. If any significant amount of the quinoline-insoluble matter adheres to the O-ring seal, place this on the watch glass also. Place the watch glass and contents in an oven maintained at 105 °C to 110 °C for 2 h, then transfer them to a desiccator to cool.

If quinoline-insoluble material has adhered to the O-ring seal, gently brush this on to the watch glass. Weigh the watch glass with the membrane filter disc and the quinoline-insoluble matter to the nearest 0,1 mg. Using forceps, hold the membrane filter disc, which will have rolled into a tube, over the watch glass and detach as much adhering quinoline-insoluble matter as possible by sharply tapping the forceps. Hold the membrane filter disc flat on a clean, smooth piece of paper and remove any residual quinoline-insoluble material by carefully brushing.

Transfer the recovered quinoline-insoluble material to a clean vial of suitable size to await analysis.

NOTE An artist's sable brush, size 5 has been found to be suitable for brushing off the quinoline-insoluble material.



Key

- | | | | |
|---|-------------------------|---|--------------------------|
| 1 | Cap | 5 | Silicone O-ring |
| 2 | Clamping rod | 6 | Perforated support plate |
| 3 | Silicone O-ring | 7 | Coarse mesh support |
| 4 | Pressure hose connector | 8 | Outlet connector |

Figure 1 — Typical pressure-filtration apparatus

4.4.5 If the amount of recovered quinoline-insoluble material is insufficient for the ensuing analysis, repeat the extraction with amounts of pitch and quinoline increased proportionately up to a maximum of 5 g of pitch and 125 ml of quinoline for a single extraction. In the event that the increased amounts still yield an inadequate quantity of quinoline-insoluble matter, repeat the entire extraction procedure using further 5 g portions of the test sample until the required amount is obtained.

4.5 Calculation and expression of results

Calculate the yield of quinoline-insoluble material, w_{qi} , expressed as a percentage by mass, using the equation:

$$w_{qi} = \frac{100(m_1 - m_2 - m_3)}{m_4 - m_5} \quad (1)$$

where

m_1 is the combined mass of the watch glass, the membrane filter disc and the isolated quinoline-insoluble material, in grams;

m_2 is the mass of the watch glass, in grams;

m_3 is the mass of the membrane filter disc, in grams;

m_4 is the mass of the beaker containing the pitch sample, in grams;

m_5 is the mass of the empty beaker, in grams.

Express the result of the calculation to two significant figures.

NOTE This result should not be quoted as the content of quinoline-insoluble matter present in the pitch because the quinoline-insoluble matter is empirical. ISO 6791 should be used for the determination of quinoline-insoluble matter.

5 Analysis of quinoline-insoluble material for carbon and hydrogen content

5.1 Reagents

Warning — Refer to the reagent supplier's Health and Safety data sheets for the precautions which are to be taken for the safe use of magnesium perchlorate and sodium hydroxide supported on synthetic silicate.

5.1.1 Magnesium perchlorate, anhydrous, analytical reagent grade, of particle size range approximately 0,7 mm to 1,2 mm.

5.1.2 Sodium hydroxide on synthetic silicate support, analytical reagent grade, of particle size range approximately 1,5 mm to 3,0 mm.

5.1.3 Silver metal woven mesh, of wire diameter approximately 0,3 mm and mesh aperture approximately 0,8 mm.

5.1.4 Silver metal wool, microanalytical quality, typically of wire diameter approximately 0,05 mm.

5.1.5 Quartz wool.

5.1.6 Oxygen, of purity at least 99,5 %, supplied via a two-stage pressure regulator and a needle-valve capable of controlling the flow rate in the range 0 ml/min to 500 ml/min.

5.2 Apparatus

Ordinary laboratory apparatus and the following.

5.2.1 Carbon/hydrogen analyser, comprising the items in 5.2.2 to 5.2.8.

5.2.2 Combustion boat, of porcelain, approximately 60 mm long, 10 mm wide and 8 mm high, and having an eyelet at one end to allow it to be coupled to the transport mechanism (5.2.6).

5.2.3 Combustion tube, of impermeable alumina, 900 mm long, 23 mm external diameter and 18 mm internal diameter, capable of working satisfactorily at temperatures up to 1 400 °C.

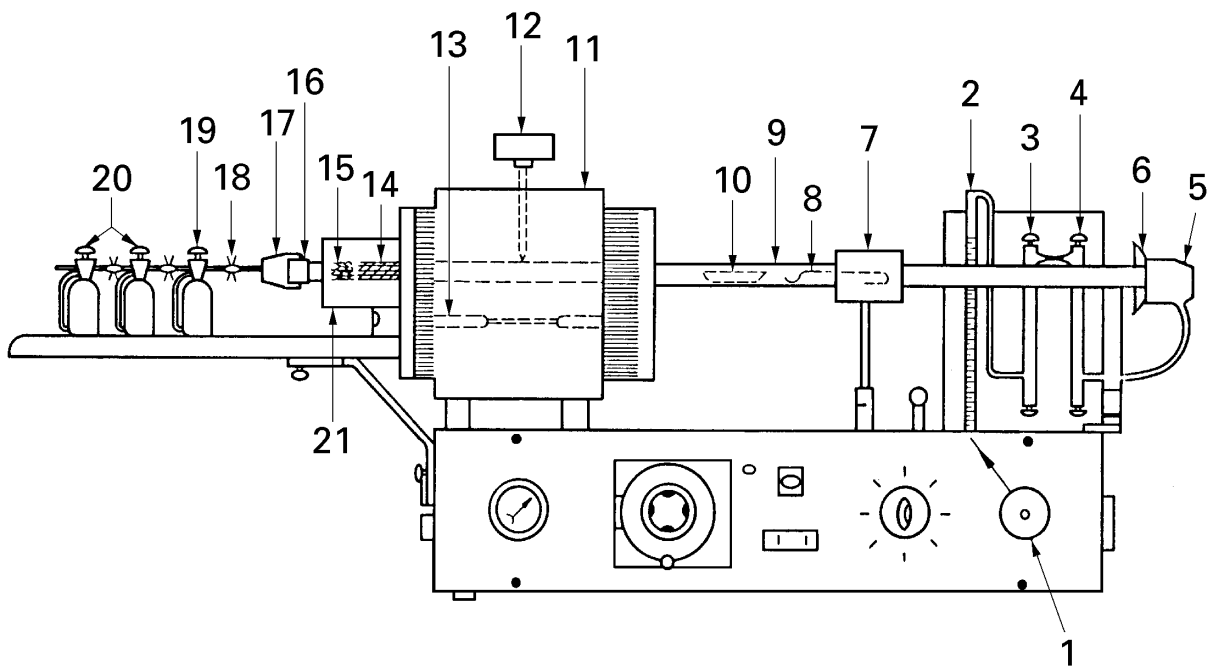
5.2.4 Combustion furnace, electrically heated, capable of maintaining a heated zone of the combustion tube (5.2.3) a minimum of 100 mm long to a maximum temperature of 1 400 °C and equipped with means of indicating the operating temperature.

5.2.5 Auxiliary furnace, electrically heated, capable of maintaining a heated zone of the combustion tube (5.2.3) a minimum of 80 mm long at a temperature of (730 ± 5) °C.

The auxiliary furnace is positioned after the combustion furnace (5.2.4) on the same axis.

5.2.6 Transport mechanism, (see Figure 2) capable of moving the combustion boat (5.2.2) containing the quinoline-insoluble material (4.4.4) within the combustion tube (5.2.3) at a rate of 20 mm/min without external mechanical linkage, to position the combustion boat finally at the centre of the heated zone of the combustion furnace.

Typically this apparatus will comprise a cylindrical soft-iron armature fitting inside the combustion tube and connected by a heat-resistant metal hook to the eyelet of the combustion boat. Motion is transmitted to the soft-iron armature by a cylindrical electrical solenoid which is moved along the outside of the combustion tube by means of a screw-feed mechanism driven by a variable-speed electric motor.



Key		
1 Oxygen valve	8 Soft-iron armature and hook	15 Silver metal wool
2 Flowmeter	9 Combustion tube	16 Silicone O-ring
3 H ₂ O absorber	10 Combustion boat	17 Hemispherical ground joint
4 CO ₂ absorber	11 Combustion furnace	18 Clipped (hemispherical) ground joint
5 Hemispherical ground joint	12 Control thermocouple	19 H ₂ O absorption tower
6 Static electricity inhibitor	13 Silicon carbide heating elements	20 CO ₂ absorption towers
7 Electric solenoid	14 Silver metal woven mesh	21 Auxiliary furnace

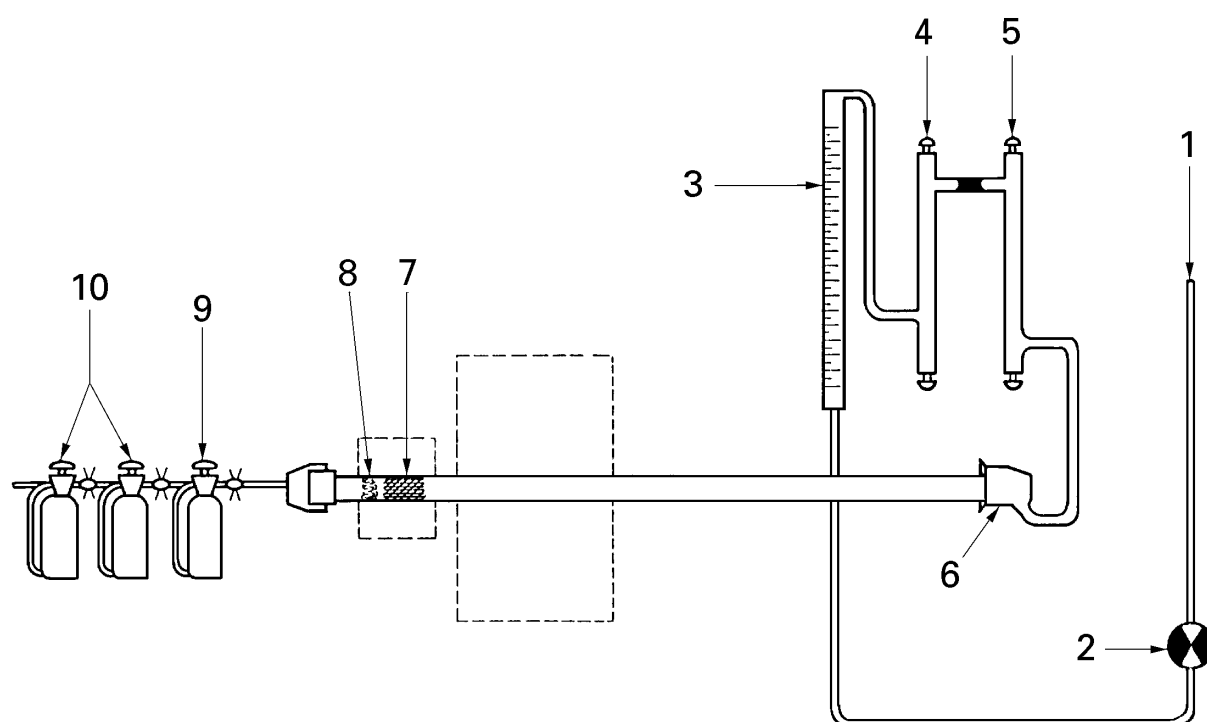
Figure 2 — Typical arrangement of a carbon/hydrogen analyser

5.2.7 Oxygen purification train, comprising in the direction of gas flow, an excess pressure relief device, a flowmeter with a flow range of 50 ml/min to 500 ml/min, an absorption tube containing anhydrous magnesium perchlorate (5.1.1) and an absorption tube containing sodium hydroxide supported on synthetic silicate (5.1.2).

The outlet of the last tube is linked by flexible tubing to a gas-tight connector which fits the inlet end of the combustion tube (5.2.3). A typical arrangement for the oxygen purification train is shown in Figure 3.

5.2.8 Gas absorption train, comprising three absorption towers connected in series, the first in the direction of the gas flow containing anhydrous magnesium perchlorate (5.1.1) and the remaining two containing sodium hydroxide supported on synthetic silicate (5.1.2).

The inlet of the tower containing anhydrous magnesium perchlorate is connected via a hemispherical ground-glass joint to a glass tube terminating in a silicone rubber seal to the outlet of the combustion tube (5.2.3). The dimensions and packing arrangement of a typical absorption tower are shown in Figure 4.



Key

- | | |
|---------------------------------------------------|-------------------------------------------------|
| 1 Oxygen inlet from excess-pressure relief device | 6 Hemispherical ground joint to combustion tube |
| 2 Oxygen flow control | 7 Silver metal woven mesh |
| 3 Flowmeter | 8 Silver metal wool |
| 4 H ₂ O absorber | 9 H ₂ O absorption tower |
| 5 CO ₂ absorber | 10 CO ₂ absorption towers |

Figure 3 — Oxygen flow circuit in a typical carbon/hydrogen analyser

5.3 Preparation of the apparatus

The arrangement of the apparatus is illustrated in Figure 2.

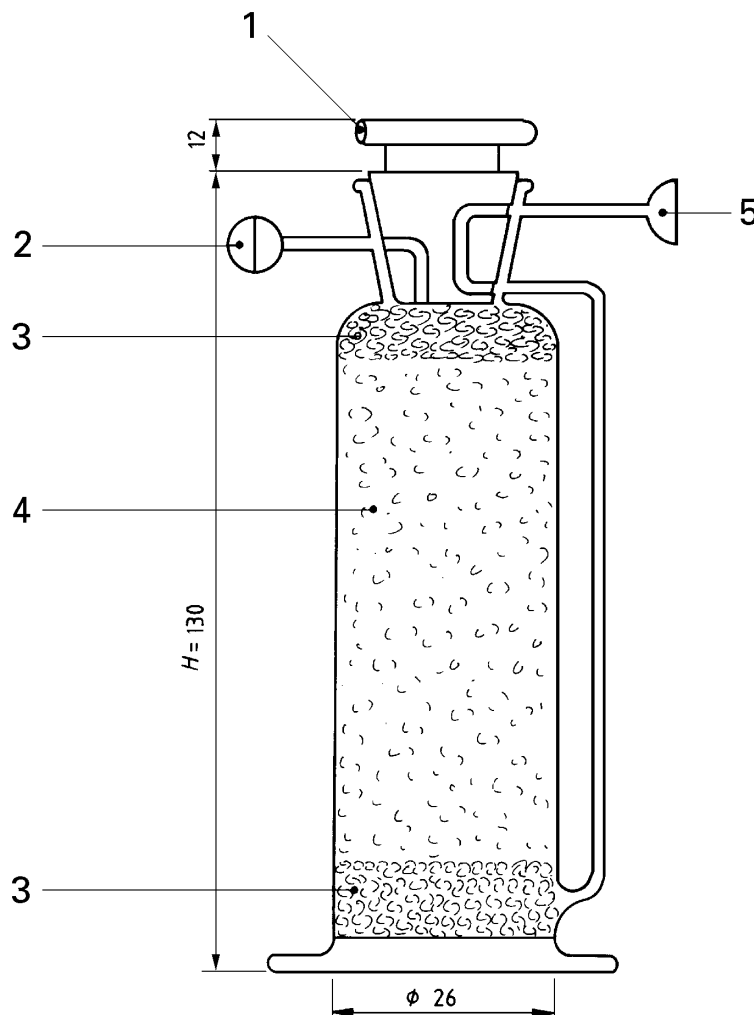
Take a piece of the silver metal woven mesh (5.1.3), 100 mm by 100 mm, and fold it in half, then roll it up to make a cylinder 50 mm long and approximately 18 mm in diameter. Insert the rolled silver metal mesh into the combustion tube (5.2.3) so that it is in light contact with the walls and leaves approximately 60 mm of space between the end of the mesh and the end of the combustion tube. Push a small plug of the silver metal wool (5.1.4) into the tube in

contact with the silver mesh to act as a back-up in case the silver metal mesh in time becomes deformed and allows the combustion gases to pass through preferentially. Replace the silver metal mesh and silver metal wool with fresh materials after 70 determinations have been made.

Place the combustion tube (5.2.3) into the two furnaces (5.2.4 and 5.2.5) so that the silver metal mesh (5.1.3) and the silver metal wool (5.1.4) are centrally placed within the auxiliary furnace (5.2.5). Connect the inlet end of the combustion tube (5.2.3) to the oxygen purification train (5.2.7) and the outlet end, which protrudes from the auxiliary furnace, to the gas absorption train (5.2.8).

Set the temperature controller of the combustion furnace (5.2.4) to 1 200 °C and that of the auxiliary furnace (5.2.5) to (730 ± 5) °C Start the oxygen flow. Leave the apparatus running for about 2 h until the temperatures of the two furnaces have stabilized at the required values.

Dimensions in millimetres



- Key**
- | | |
|-------------------------------------------------|-------------------------------------------------------------------------------------------|
| 1 Tap to close inlet and outlet by 90° rotation | 4 Magnesium perchlorate (5.1.1) or sodium hydroxide support on synthetic silicate (5.1.2) |
| 2 Hemispherical ground joints | 5 Hemispherical ground joints |
| 3 Quartz wool (5.1.5) | |

Figure 4 — Typical gas absorption tower

5.4 Procedure

5.4.1 Conditioning

Insert the combustion boat (5.2.2) into the combustion tube (5.2.3) and connect it to the armature of the transport mechanism (5.2.6), then reconnect the oxygen flow. Move the combustion boat into the combustion furnace (5.2.4) and leave it to condition for approximately 20 min. Disconnect the oxygen supply and withdraw the combustion boat, then place it in a desiccator to cool for 15 min. Close and disconnect the three absorption towers constituting the gas absorption train (5.2.8). Weigh the three towers separately to the nearest 0,1 mg, record the masses, then reconnect them to the combustion tube in the original sequence. Weigh the combustion boat to the nearest 0,1 mg, then introduce (190 ± 10) mg of the quinoline-insoluble material (4.4.4) into the boat in a uniform layer. Weigh the boat and its contents to the nearest 0,1 mg. Record the masses.

5.4.2 Determination

Using forceps, insert the combustion boat (5.2.2) containing the test sample into the combustion tube (5.2.3). Connect the hook on the armature of the transport mechanism (5.2.6) to the eye of the combustion boat and gently move the assembly along the combustion tube until the armature is located correctly inside the solenoid of the magnetic drive mechanism. Rapidly reconnect the oxygen supply, open the taps on the absorption towers and raise the oxygen flow rate smoothly to 300 ml/min. Switch on the solenoid and the drive motor of the transport mechanism (5.2.6) and set the rate of movement of the combustion boat into the combustion furnace (5.2.4) at 20 mm/min.

Thirty minutes after starting the transport mechanism reduce the oxygen flow rate to 50 ml/min then quickly close and disconnect the gas absorption towers. Allow the towers to cool for 15 min then weigh each individually to the nearest 0,1 mg. Record the masses.

5.4.3 Residual ash

Manually return the sample transport mechanism (5.2.6) to its starting position then switch off the solenoid. Remove the combustion boat (5.2.2) using forceps and place it in a desiccator to cool. Weigh the cooled boat and record the mass. Remove any ash residue before re-using the boat.

5.4.4 Blank determination

Perform a blank determination to obtain a hydrogen correction by repeating 5.4.1 and 5.4.2 using an empty boat without a test sample and weighing only the anhydrous magnesium perchlorate absorption tower (5.2.8) at the start and end of the determination. Record these masses.

NOTE It is recommended that the combustion tube be continuously purged with a slow oxygen flow if it is not planned to perform another determination immediately.

5.5 Calculation and expression of results

Calculate the hydrogen content w_H , as a percentage by mass, of the quinoline-insoluble material using the equation:

$$w_H = 11,9 \frac{(m_6 - m_7) - (m_8 - m_9)}{(m_{10} - m_{11})} \quad (2)$$

where

m_6 is the mass, in grams, of the anhydrous magnesium perchlorate tower after the main determination (5.4.2);

m_7 is the mass, in grams, of the anhydrous magnesium perchlorate tower before the main determination (5.4.1);

m_8 is the mass, in grams, of the anhydrous magnesium perchlorate tower after the blank determination (5.4.4);

m_9 is the mass, in grams, of the anhydrous magnesium perchlorate tower before the blank determination (5.4.4);

m_{10} is the mass, in grams, of the combustion boat containing the test sample (5.4.1);

m_{11} is the mass, in grams, of the empty combustion boat (5.4.1).

Calculate the carbon content w_C , expressed as a percentage by mass, of the quinoline-insoluble material using the equation:

$$w_C = 27,29 \frac{(m_{12} - m_{13}) - (m_{14} - m_{15})}{(m_{10} - m_{11})} \quad (3)$$

where

m_{10} and m_{11} are as defined in equation (2);

m_{12} is the mass, in grams, of the first sodium hydroxide absorption tower after the determination (5.4.2);

m_{13} is the mass, in grams, of the first sodium hydroxide absorption tower before the determination (5.4.1);

m_{14} is the mass, in grams, of the second sodium hydroxide absorption tower after the determination (5.4.2);

m_{15} is the mass, in grams, of the second sodium hydroxide absorption tower before the determination (5.4.1).

Use the above values of w_H and w_C to calculate the atomic carbon/hydrogen ratio R of the quinoline-insoluble material using the equation:

$$R = \frac{1,008 \times w_C}{12,011 \times w_H} \quad (4)$$

Express the results for w_H , w_C and R as the mean of duplicate determinations, to two decimal places.

5.6 Ash content

Calculate the ash content w_{ash} , as a percentage by mass, of the quinoline-insoluble material using the equation:

$$w_{\text{ash}} = 100 \frac{(m_{16} - m_{11})}{(m_{10} - m_{11})} \quad (5)$$

where

m_{10} and m_{11} are as defined in equation (2);

m_{16} is the mass, in grams, of the combustion boat containing any ash residue remaining after the main determination (5.4.3).

Express the result as the mean of duplicate determinations, to two significant figures.

6 Precision

6.1 Repeatability

The results of duplicate determinations, carried out in the same laboratory by the same operator with the same apparatus but at different times on representative test portions taken from the test sample, shall not differ by more than by more than 0,31.

6.2 Reproducibility

The means of the results of duplicate determinations carried out in each of two laboratories on representative test portions taken from the same sample after the last stage of sample preparation, shall not differ by more 0,35.

7 Test report

The test report shall include the following information:

- a) a complete identification of the sample of pitch;
- b) a reference to this International Standard, i.e. ISO 12979:1999;
- c) the yield of quinoline-insoluble material calculated and expressed in accordance with 4.5;
- d) the carbon and hydrogen contents of the quinoline-insoluble material and its atomic carbon/hydrogen ratio calculated and expressed in accordance with 5.5;
- e) the yield of ash residue from the quinoline-insoluble material remaining after the carbon/hydrogen determination, calculated and expressed in accordance with 5.6;
- f) any unusual features noted during the isolation or the determination;
- g) any operation not included in this International Standard or regarded as optional.

