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Hard coal — Froth flotation testing — Part 2: Sequential evaluation

*Houille — Essais de flottation —
Partie 2: Évaluation séquentielle*



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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 8858-2 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 1, *Coal preparation: Terminology and performance*.

ISO 8858 consists of the following parts, under the general title *Hard coal — Froth flotation testing*:

- *Part 1: Laboratory procedure*
- *Part 2: Sequential evaluation*
- *Part 3: Release evaluation*

Introduction

The froth flotation of coal has a widespread application for the recovery of fine coal particles and their separation from unwanted mineral matter. The response of coal to the froth flotation process is measured initially by a laboratory scale test. ISO 8858-1:1990 provides a means of evaluating the general flotation characteristics of a coal under a set of specified conditions and will not necessarily indicate the full potential of that coal. It is accepted that variation of the many parameters in the froth flotation process can be used to effect the beneficiation of the product. This part of ISO 8858 describes a procedure for the more complete determination of the flotation characteristics of a coal, using the apparatus and basic procedures described in ISO 8858-1. The purpose of this extended procedure is to provide information similar to that provided by the sink/float curve, which is the basis for density separations. The data obtained are expressed as a yield/ash curve. The information can be used to define the limitations on the cleaning of fine coal by froth flotation.

The procedures specified in this part of ISO 8858 are of practical significance in the development and evaluation of coal-preparation-plant flotation circuits, although engineering design aspects, such as flotation kinetics and the selection of size and type of cell, are not addressed.

The flotation response curve (yield/ash) indicates the maximum possible yield at any specified ash content. The general shape of the curve indicates the sensitivity of flotation performance to the nature of the coal and to operating conditions.

The procedure may be modified to test and compare the performance of different frother and collector types, the assessment of liberation by grinding, and the comparison of alternative feed size ranges. However, results of such tests should clearly indicate any use of non-standard procedures.

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Hard coal — Froth flotation testing —

Part 2: Sequential evaluation

1 Scope

This part of ISO 8858 sets out a laboratory sequential procedure for the froth flotation testing of fine coal, e.g. coal having a particle size of less than 0,5 mm. The procedure provides a means of evaluating for a coal the flotation characteristics (expressed as a yield/ash relationship) that may be expected from the froth flotation process.

Pulp samples that cannot be dewatered without the use of heat or chemical additives are not covered by this part of ISO 8858; nor does it cover procedures for the investigation of flotation kinetics.

The test is not intended to provide plant design data.

This part of ISO 8858 should be read in conjunction with ISO 8858-1.

2 Normative references

The following referenced documents are indispensable for the application 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 589:2003, *Hard coal — Determination of total moisture*

ISO 1171:1997, *Solid mineral fuels — Determination of ash*

ISO 1213-1:1993, *Solid mineral fuels — Vocabulary — Part 1: Terms relating to coal preparation*

ISO 1953:1994, *Hard coal — Size analysis by sieving*

ISO 7936:1992, *Hard coal — Determination and presentation of float and sink characteristics — General directions for apparatus and procedures*

ISO 8858-1:1990, *Hard coal — Froth flotation testing — Part 1: Laboratory procedure*

ISO 13909-4: 2001, *Hard coal and coke — Mechanical sampling — Part 4: Coal — Preparation of test samples*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 1213-1 and the following apply.

3.1 higher rank coal

coal having a gross specific energy of 21,00 MJ/kg or greater on an ash-free, moist (afm) basis and 27,00 MJ/kg or greater on a dry, ash-free (daf) basis

4 Principle

A sequence of laboratory flotation tests is carried out on a single coal sample, using a procedure based on that described in ISO 8858-1. Variations on that procedure are made to generate a number of products, by refloating concentrates and tailings a number of times. The masses and ash percentages of the various products are used to construct a yield/ash curve showing the flotation response.

5 Sample

The history and method of preparation of samples can affect the flotation characteristics of the coal considerably. The history of the sample should be recorded, and care should be taken to ensure that samples for comparison purposes are prepared in a similar manner and, where applicable, in accordance with the sample preparation procedures specified in ISO 13909-4.

A size analysis of the sample shall be carried out in accordance with ISO 1953.

6 Apparatus

The apparatus shall be as specified in ISO 8858-1.

7 Flotation conditions

7.1 Collector dosage

To establish well-defined flotation response curves, it is necessary to extend the range of conditions specified in ISO 8858-1. Different coals will require different conditions to display the attainable range of yields and corresponding product ash levels. To obtain a preliminary guide to the appropriate flotation conditions for this extended test, it is recommended that a sample of the coal be first tested according to the procedure specified in ISO 8858-1.

NOTE It is suggested that the collector increments referred to in 8.3 be selected on the basis of the result of the ISO 8858-1 test in Table 1.

Table 1

ISO 8858-1 yield %	ISO 8858-1 suggested collector dosage (fraction of dosage used in ISO 8858-1)
< 40	As in ISO 8858-1 = 1 ml/kg
≥ 40 < 60	25 % = 0,25 ml/kg
≥ 60 ≤ 80	10 % = 0,10 ml/kg
> 80	2,5 % = 0,025 ml/kg

The final result is largely independent of the selected increment size. Experienced operators may prefer to use other dosage rates.

7.2 Frother dosage

Frother addition at each stage may be made at approximately half the dosage specified in ISO 8858-1, i.e. at 0,05 ml of 4-methyl-2-pentanol (formerly called methyl isobutyl carbinol MIBC) (50 ml of 0,1 % aqueous solution) per kilogram of dry solids in the initial sample.

NOTE Frother dosages are not critical beyond the initial separation. Operator judgment may be used to maintain an adequate froth.

7.3 Solids content

The solids content for the first flotation stage shall be as specified in ISO 8858-1. For all subsequent stages, the mass of solids shall be that which results from the previous flotation stage.

7.4 Air flow rate

The air flow rate shall be as specified in ISO 8858-1.

7.5 Test temperature

The test temperature shall be as specified in ISO 8858-1 and shall be reported with the test results.

8 Procedure

8.1 Initial procedure

The moisture content, size analysis and other parameters specified in ISO 8858-1 shall be determined.

8.2 Initial separation

The initial separation shall be carried out generally following the principles given in ISO 8858-1 with the exceptions that

- a) no collector shall be used,
- b) no conditioning time is required, and
- c) no frother shall be used.

Both concentrate and tailings shall be retained as pulps for subsequent refloatation.

NOTE Experience has shown that omission of frother and collector permits selective recovery of low-ash rapid floating particles and better definition of the low-yield end of the curve.

To ensure that the cell volume for subsequent tests is not exceeded, care should be taken to use a minimum of wash water in collecting the two products.

8.3 Subsequent separations

After performing an initial flotation, the resultant tailings shall be successively refloatated, generally following the procedure given in ISO 8858-1, with incremental collector addition to recover any floatable particles that may not have been collected with the concentrate. This process shall be continued until it is estimated that refloatation of the successive tailings fraction has resulted in nil or negligible concentrate mass (approximately less than 5 % of the initial feed sample mass).

Each concentrate fraction, resulting from these successive tailings flotations, shall then be refloatated as many times as necessary, until further flotation does not result in additional removal of mineral matter (as judged by the presence of solids in the tailings), or the concentrate sample to be floated shall have nil or negligible mass (approximately less than 5 % of the initial feed sample mass).

After completion of these flotations, each concentrate shall be repeatedly refloatated until all entrained mineral matter is removed. Each tailings fraction resulting from refloating of the concentrates shall then be subjected to further flotations until nil or negligible concentrate is produced.

Essentially, the initial feed mass is fractionated by successive refloatations without subsequent recombination of individual concentrate or tailings fractions.

NOTE 1 It will be rare for any branch of the process sequence to require more than four flotation operations. Usually two or three stages will suffice.

NOTE 2 For some coals, further testing may be required to achieve separations yielding down to 2 % of the initial feed mass.

8.4 Analysis of concentrate and tailings

All products may be filtered. All products shall be air-dried, weighed and analysed for moisture and ash as specified in ISO 589 and ISO 1171 respectively, and reported on a dry basis.

NOTE Drying in an air oven at 40 °C may be carried out, provided that equilibration with ambient conditions is achieved before weighing.

9 Calculation of results

The mass of reconstituted feed (m_R), in grams, shall be calculated on a dry basis from the following equation:

$$m_R = \sum_{i=1}^N (m_C)_i + \sum_{j=1}^M (m_T)_j \quad (1)$$

where

N is the total number of concentrates;

$(m_C)_i$ is the mass of concentrate i , in grams;

M is the total number of tailings;

$(m_T)_j$ is the mass of tailing j , in grams.

The percentage mass distribution (D) for a product is given by the following equations:

Percentage mass distribution for concentrate i ,

$$D_i = 100(m_C)_i / m_R \quad (2)$$

Percentage mass distribution for tailings j ,

$$D_j = 100(m_T)_j / m_R \quad (3)$$

The distribution for all products (concentrate and tailings) and the ash percentage of each product shall be determined and the results listed in a single table in ascending order of percentage of ash. Progressive cumulative distribution (i.e. yield) and corresponding cumulative ash percentage values shall then be calculated according to the following equations:

Cumulative percentage yield ($Y_{\text{cum},n}$) in the first n products,

$$Y_{\text{cum},n} = \sum_{k=1}^N D_k \quad (4)$$

where D_k is the distribution to the k th product in the ordered list

Cumulative percentage of ash ($A_{\text{cum},n}$) of the first n products,

$$A_{\text{cum},n} = \sum_{k=1}^N \frac{(D_k \times A_k)}{Y_{\text{cum},n}} \quad (5)$$

where A_k is the ash percentage on a dry basis of the k th product in the ordered list

To determine a quantitative performance measure, organic efficiency and ash error may be used in accordance with ISO 7936.

10 Test report

The test report shall contain the following information:

- a) a reference to this part of ISO 8858, i.e. ISO 8858-2;
- b) complete identification of the sample;
- c) history of the sample;
- d) date on which the test was carried out;
- e) particle size analysis of the sample, carried out in accordance with ISO 1953;
- f) mass of each feed, concentrate and tailings, expressed on a dry basis;
- g) the difference between the initial feed mass and the reconstituted feed mass (where the initial dried sample mass is able to be determined);
- h) mass distribution;
- i) ash percentage of each feed, concentrate and tailings on a dry basis;
- j) the yield/ash relationship in tabular and graphical forms;
- k) the test temperature and other properties as required;
- l) air flow rate.

NOTE 1 A worked example of the calculation of results is given in Annex A.

NOTE 2 A recommended form for the presentation of results, together with a graphical representation, is given in Annex B.

Annex A (informative)

Worked example of calculation of results

A.1 Scope

This Annex sets out a worked example of the calculation of the results of the testing in accordance with Clause 9. The process sequence for this example is given in Figure A.1.

A.2 Procedure

The sequential procedure effectively divides the original sample into fractions of varying ash. After drying, each fraction is weighed and its percentage of ash is determined. A cumulative yield/ash curve is generated from these data by mathematically recombining the various fractions in order of increasing ash. Figure A.1 shows a sequential flotation procedure conducted on a sample of minus 0,5 mm coal. A "C" prefix is used to denote a concentrate, and the "T" prefix is used to denote a tailing. The numerical portion of the label refers to the order in which the flotations are conducted, with rectangular boxes indicating an intermediate stream and ovals indicating an actual fraction. Table A.1 indicates how a yield/ash curve is generated from the experimental data.

A sample of approximately 400 g, initial air-dry mass, is subjected to repeated flotation according to the sequential procedure. (A preliminary test, following the procedure given in ISO 8858-1, gave a yield of 73 %.) Accordingly (see 7.1), a collector increment of 0,1 ml is used in all but the initial stage. Frother, at a dosage of 20 ml of 0,1 % aqueous solution, is added at each stage in accordance with 7.2.

For the example, a minimum yield limit of 5 % of the initial feed mass is specified for the concentrates to restrict the number of flotation tests required.

The procedure adopted for this example is as follows.

a) Initial separation (see 8.1)

No collector, no frother, and no conditioning time allowed. Concentrate (C1) is set aside. Tailings (T1) are retained in the cell.

b) Second flotation (see 8.2)

Pulp is conditioned with 0,1 ml of collector and then add frother in this and all subsequent stages. Concentrate (C2) is set aside. Tailings (T2) are retained in cell. Neither product is judged to be less than the minimum limits.

c) Third flotation

Concentrate (C3) is set aside. Neither product is judged to be less than the minimum limits. Tailings (T3) are retained in cell.

d) Fourth flotation

Concentrate (C4) is set aside. Tailings (T4) are retained in cell. Neither product is judged to be less than the minimum limits.

e) Fifth flotation

Concentrate (C5) is set aside (judged to be less than the minimum limits). Tailings (T5) are removed from the cell and retained.

f) Sixth flotation

Concentrate (C5) from the fifth flotation was reintroduced to the cell and refloated. Each product was judged to be less than the minimum limits and they are therefore removed from the cell and retained as concentrate (C6) and tailings (T6), respectively.

g) Seventh flotation

Concentrate (C4) from the fourth flotation is reintroduced to the cell and refloated. Each product is judged to be less than the minimum limits and they are therefore removed from the cell and retained as concentrate (C7) and tailings (T7), respectively.

h) Eighth flotation

Concentrate (C3) from the third flotation is reintroduced to the cell and refloated. Concentrate (C8) is judged to be less than the minimum limits, and retained. Tailings (T8) are retained in the cell.

i) Ninth flotation

Each product is judged to be less than the minimum limits. Removed and retained as concentrate (C9) and tailings (T9), respectively.

j) Tenth flotation

Concentrate (C2) from the second flotation is reintroduced to the cell and refloated. Concentrate (C10) is judged to be less than the minimum limits, and retained. Tailings (T10) are retained in the cell.

k) Eleventh flotation

Concentrate (C11) is judged to be less than the minimum limits, and retained. Tailings (T11) are retained in the cell.

l) Twelfth flotation

Each product is judged to be less than the minimum limits. Removed and set aside as concentrate (C12) and tailings (T12) respectively.

m) Thirteenth flotation

Concentrate (C1) from the first flotation is reintroduced to the cell and refloated. Concentrate (C13) is judged to be less than the minimum limits, and saved. Tailings (T13) are retained in the cell.

n) Fourteenth flotation

Concentrate (C14) is judged to be less than the minimum limits, and saved. Tailings (T14) are retained in the cell.

o) Fifteenth flotation

Concentrate (C15) is judged to be less than the minimum limits, and retained. Tailings (T15) are retained in the cell.

p) Sixteenth flotation

Each product is judged to be less than the minimum limits. Removed from the cell and set aside as concentrate (C16) and tailings (T16), respectively. Separation is now considered to be effectively complete.

A.3 Test results

The results of the test are given in Table A.1.

Table A.1 — Results of froth flotation test example

Product	Mass (d)	Ash (d)
	g	%
C6	12,0	24,37
C7	33,4	12,80
C8	16,4	7,93
C9	60,7	8,22
C10	23,8	3,98
C11	83,8	4,10
C12	9,7	7,76
C13	14,0	2,98
C14	28,3	3,12
C15	46,1	3,05
C16	11,0	7,03
T5	27,4	82,28
T6	2,7	61,87
T7	1,7	43,88
T9	1,3	46,08
T12	1,5	49,15
T16	2,0	63,02

A.4 Calculations

The yield/ash relationship is calculated as follows:

a) Mass of reconstituted feed, m_R

$$m_R = \sum_{i=1}^N (m_C)_i + \sum_{j=1}^M (m_T)_j \tag{A.1}$$

where

N = total number of concentrates

= 11

$(m_C)_i$ = mass of concentrate i

For example, for concentrate C13 ($i = 13$), $(m_C)_i = 14,0$ g

M = total number of tailings

= 6

$(m_T)_j$ = mass of tailing j

For example for tailing T5 ($j = 5$), $(m_T)_j = 27,4$ g

Thus

$$m_R = 375,8 \text{ g}$$

The difference between m_R and the initial feed mass is considered acceptable.

b) Percentage mass distribution, D

Distribution to concentrate i is given by

$$D_i = 100(m_C)_i / m_R \quad (\text{A.2})$$

For example, distribution (of reconstituted feed) to concentrate C13 is given by

$$D = 100(14,0)/375,8 = 3,7 \%$$

Similarly, the distribution of feed to tailings product T5 is given by

$$D = 100(27,4)/375,8 = 7,3 \%$$

The distribution of feed to all products is listed in Figure B.1, in which the products have been arranged in the required ascending order of percentage of ash (see Clause 9). Also given in Figure B.1 are the progressive cumulative distribution and ash percentage values, $Y_{\text{cum},n}$ and $A_{\text{cum},n}$, calculated according to the equations given in Clause 9.

For example, the third ($n = 3$) value in the cumulative yield column is given by the equation

$$\begin{aligned} Y_{\text{cum},n} &= \sum_{k=1}^N D_k \\ &= 3,7 + 12,3 + 7,5 \quad (n = 3 \text{ terms}) \\ &= 23,5 \% \end{aligned} \quad (\text{A.3})$$

and the corresponding value in the cumulative ash column is given by the equation

$$\begin{aligned} A_{\text{cum},n} &= \sum_{k=1}^N \frac{(D_k \times A_k)}{Y_{\text{cum},n}} \\ &= [(3,7 \times 2,98) + (12,3 \times 3,05) + (7,5 \times 3,12)]/23,5 \\ &= (11,03 + 37,52 + 23,40)/23,5 \\ &= 3,06 \% \end{aligned} \quad (\text{A.4})$$

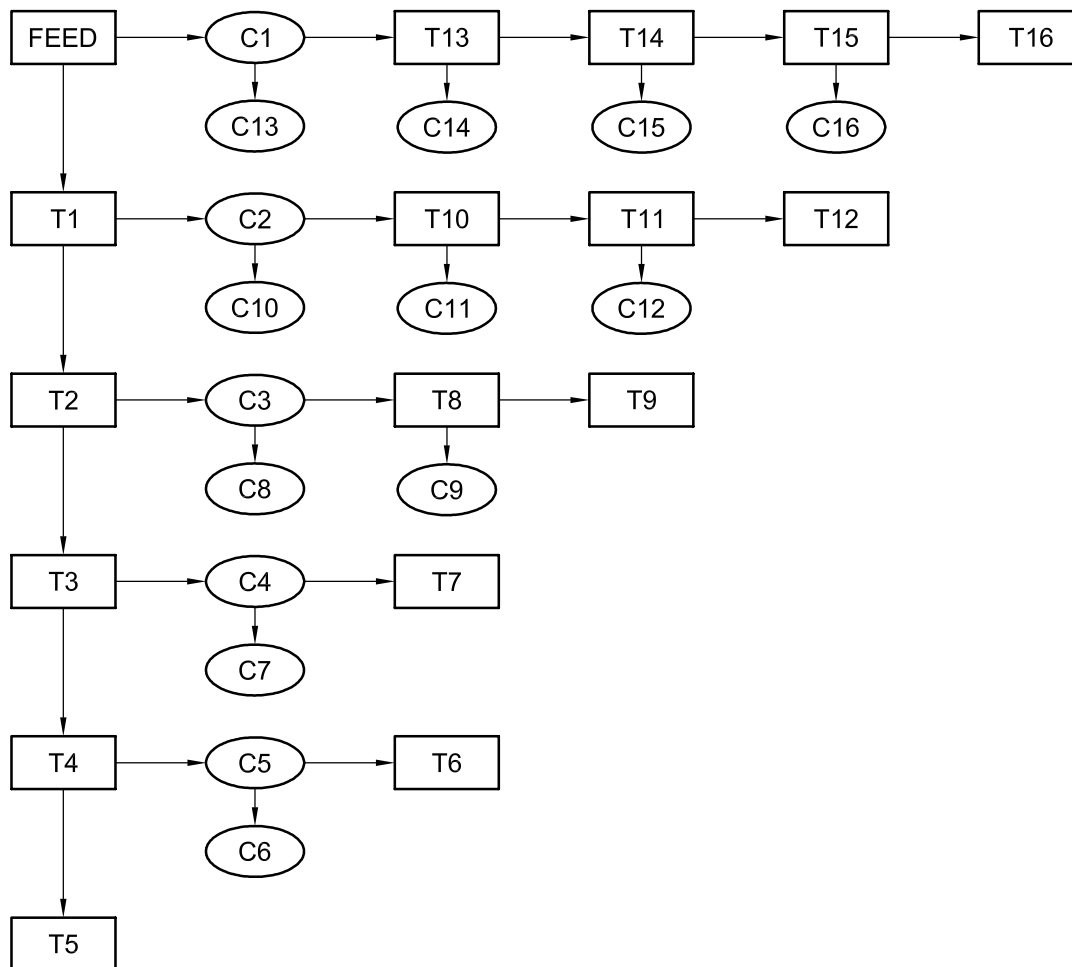


Figure A.1 —Process sequence for worked example

Annex B **(informative)**

Presentation of results

B.1 Tabulated data

A suggested format for the presentation of results is given in Figure B.1.

B.2 Graphical presentation

It is highly recommended that, in addition to the tabular format shown in Figure B.1, the results be presented graphically, as shown in Figure B.2, where cumulative yield is plotted against cumulative ash on rectangular coordinates. A smooth curve drawn through the data points facilitates interpolation, but extrapolation into the low-ash region is not possible.

Froth flotation testing

Date of report: Initials:

Date of test:

Sample identification:

Sample history:

Apparatus:

Sample size analysis

Particle size square aperture		Mass
mm		%
-1,000	+1,000	
-0,500	+0,500	
-0,250	+0,250	
-0,125	+0,125	
-0,063	+0,063	

Flotation test results

Mass of feed (d) =
 Moisture (d) =
 Ash percent of feed (d) =

Operating conditions

Temperature	
Collector	
Frother	

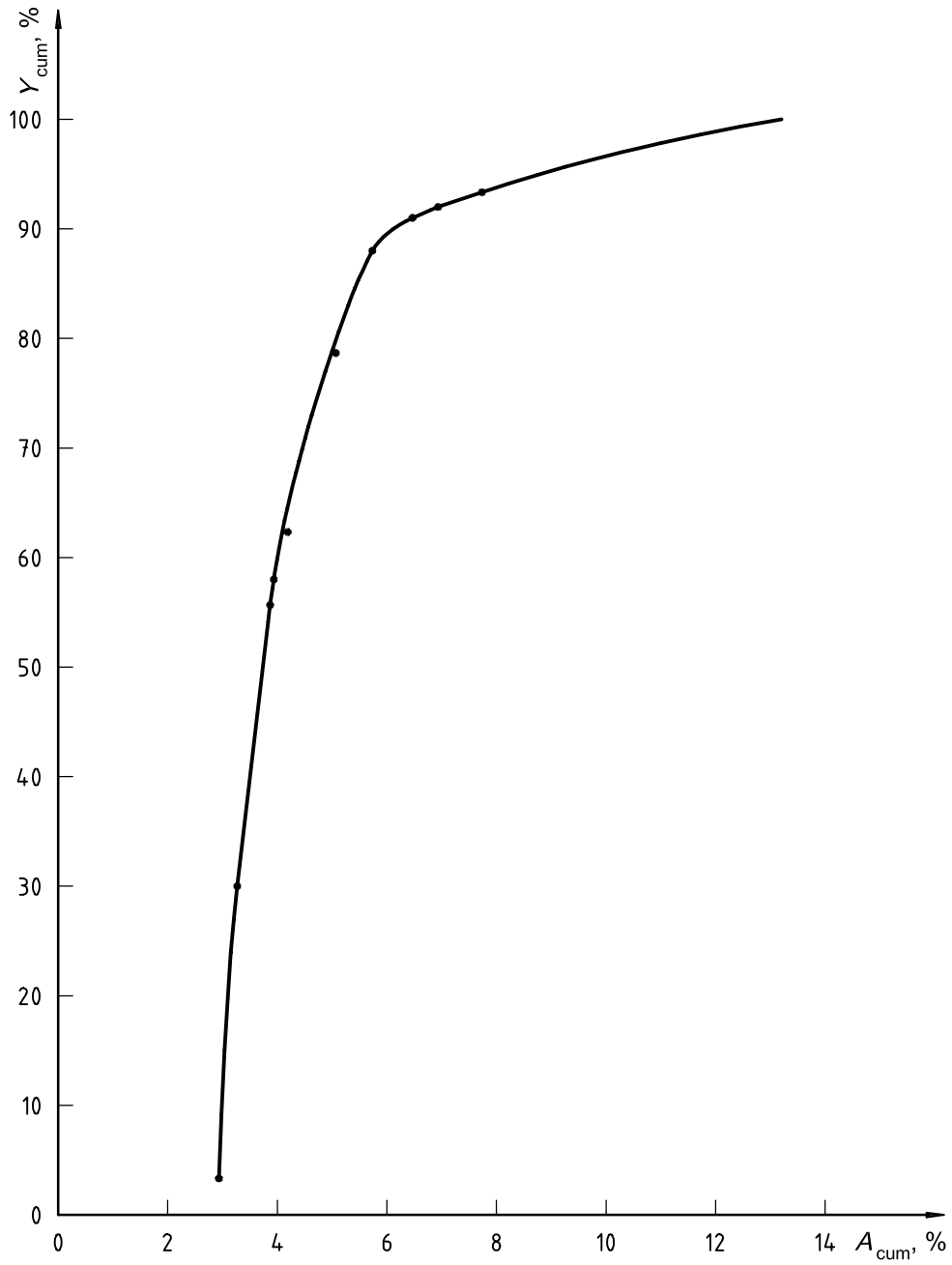
Results

Fraction	Mass	Ash (d)	Mass of ash	Cum. mass	Cum. ash	Cum. yield	Ash (d)
	g	%	g	g	g	%	%
C13	14,0	2,98	0,42	14,0	0,42	3,7	2,98
C15	46,1	3,05	1,41	60,1	1,82	16,0	3,03
C14	28,3	3,12	0,88	88,4	2,71	23,5	3,06
C10	23,8	3,98	0,95	112,2	3,65	29,9	3,26
C11	83,8	4,10	3,44	196,0	7,09	52,2	3,62
C16	11,0	7,03	0,77	207,0	7,86	55,1	3,80
C12	9,7	7,76	0,75	216,7	8,62	57,7	3,98
C8	16,4	7,93	1,30	233,1	9,92	62,0	4,25
C9	60,7	8,22	4,99	293,8	14,91	78,2	5,07
C7	33,4	12,80	4,28	327,2	19,18	87,1	5,86
C6	12,0	24,37	2,92	339,2	22,10	90,3	6,52
T7	1,7	43,88	0,75	340,9	22,85	90,7	6,70
T9	1,3	46,08	0,60	342,2	22,45	91,1	6,85
T12	1,5	49,15	0,74	343,7	24,19	91,5	7,04
T6	2,7	61,87	1,67	346,4	25,86	92,2	7,46
T16	2,0	63,02	1,26	348,4	27,12	92,7	7,78
T5	27,4	82,28	22,54	375,8	49,66	100,0	13,22
TOTAL	375,8						

Products arranged in order of ascending percentage of ash.

Comment:

Figure B.1 — Example of a form for presentation of results



Key

- Y_{cum} Cumulative yield
- A_{cum} Cumulative ash (d)

Figure B.2 — Yield/ash relationship

ICS 73.040

Price based on 13 pages