BRITISH STANDARD

BS
2000-317:1995

Methods of test for petroleum and its products — BS 2000-317: Determination of residues in liquefied petroleum gases — Low temperature evaporation method

(Identical with IP 317-1995)

ICS 75.160.30

Confirmed
January 2010

BSI
British Standards

National foreword

This British Standard reproduces verbatim IP 317:1995 (confirmed in 2002) and implements it as the UK national standard.

The UK participation in its preparation was entrusted to Technical Committee PTI/13, Petroleum testing and terminology, which has the responsibility to:

— aid enquirers to understand the text;

— present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;

— monitor related international and European developments and promulgate them in the UK.

A list of organizations represented on this committee can be obtained on request to its secretary.

The Institute of Petroleum publishes and sells all Parts of BS 2000, and all BS EN petroleum test methods that would be Part of BS 2000, both in its annual publication "Standard methods for analysis and testing of petroleum and related products and British Standard 2000 Parts" and individually.

Further information is available from:

The Institute of Petroleum, 61 New Cavendish Street,
London W1G 7AR.
Tel: 020 7467 7100. FAX: 020 7255 1472

Cross-references

The British Standards which implement international publications referred to in this document may be found in the *BSI Catalogue* under the section entitled "International Standards Correspondence Index", or by using the "Search" facility of the *BSI Electronic Catalogue* or of British Standards Online.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 16 April 2003

Summary of pages

This document comprises a front cover, an inside front cover, pages 1 to 4, an inside back cover and a back cover.

The BSI copyright date displayed in this document indicates when the document was last issued.

Amendments issued since publication

Amd. No.	Date	Comments

ISBN 0 580 41552 X

IP 317/95 ASTM D2158-92 BS 2000 : Part 317 : 1995

Determination of residues in liquefied petroleum gases – Low temperature evaporation method

This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations.

FOREWORD *This method was adopted as a joint IP – ASTM Standard Method in 1974.*

1 Scope

This standard specifies a method for the determination of the materials weathering above 38 °C which may be present in liquefied petroleum gases (LPG).

NOTE 1 The results obtained when testing liquefied petroleum gases that contain alcohols to enhance their anti-icing behaviour may be affected by the alcohols.

NOTE 2 The result may be expressed in terms of measured volumes or indices derived from these volumes. In either case, the method provides an indication of the quantity and nature of materials in the product that are substantially less volatile than the liquefied petroleum gas hydrocarbons.

NOTE 3 Control over the residue content is of considerable importance in end-use applications. In liquid feed systems residues may lead to troublesome deposits and, in vapour offtake systems, residues that are carried over may foul regulating equipment. Those that remain will accumulate, may be corrosive, and will contaminate any following product. Water, particularly if alkaline, can cause failure of regulating equipment and corrosion of metals.

2 Definitions

For the purposes of this standard the following definitions apply.

2.1 residue: The volume, in millilitres, of the residual material boiling above 38 °C resulting from the evaporation of 100 ml of sample under the specified conditions of this test.

2.2 R number: The residue multiplied by 200.

2.3 oil stain observation: The volume, in millilitres, of solvent–residue mixture required to yield an oil ring that persists for 2 min on a filter paper under specified conditions.

2.4 O number: 10 divided by the oil stain observation.

3 Principle

A 100 ml sample of liquefied petroleum gas is weathered in a 100 ml centrifuge tube. The volume of residue remaining at 38 °C is measured and recorded. The residue is redissolved in an oil-free solvent and measured volumes are spotted onto filter papers. The volume of residue required to produce a persistent oil stain is recorded.

4 Materials

4.1 Solvent, oil-free pentane, except whenever the ambient temperature or altitude is too high to enable the convenient handling of pentane, cyclopentane shall be used.

4.2 Coolant, a suitable liquid such as acetone, propan-2-ol, methanol or ethanol.

4.3 Carbon dioxide, solid, commercial grade, if required (see 7.1).

CAUTION - Safety glasses and gloves should be worn when handling solid carbon dioxide as it can cause burns.

5 Apparatus

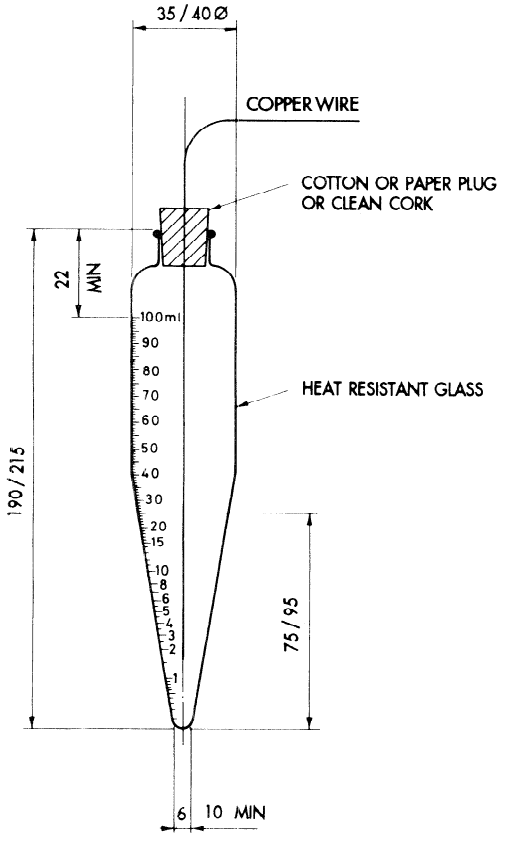
5.1 Centrifuge tube, 100 ml graduated, conforming to the dimensions given in figure 1. The first 0,5 ml shall be graduated in 0,05 ml increments.

The taper shall be uniform and the bottom shall be rounded as shown in figure 1. Tubes shall be made of thoroughly annealed heat-resistant glass and conform to the volumetric graduation tolerances, based on air-free water at 20 °C, given in table 1.

Table 1 – Centrifuge tube graduation tolerances

Range ml	Scale division ml	Volume tolerance ml
0 to 0,1	0,05	±0,02
Above 0,1 to 0,3	0,05	±0,03
Above 0,3 to 0,5	0,05	±0,05
Above 0,5 to 1,0	0,1	±0,05
Above 1,0 to 3,0	0,1	±0,1
Above 3,0 to 5,0	0,5	±0,2
Above 5,0 to 25,0	1,0	±0,5
Above 25,0 to 100,0	1,0	±1,0

5.2 Stopper, either slotted cork or a cotton wool or cleansing tissue plug, loosely fitting into the mouth of the centrifuge tube (5.1).



Dimensions are in millimetres

Figure 1 – 100 ml graduated centrifuge tube.

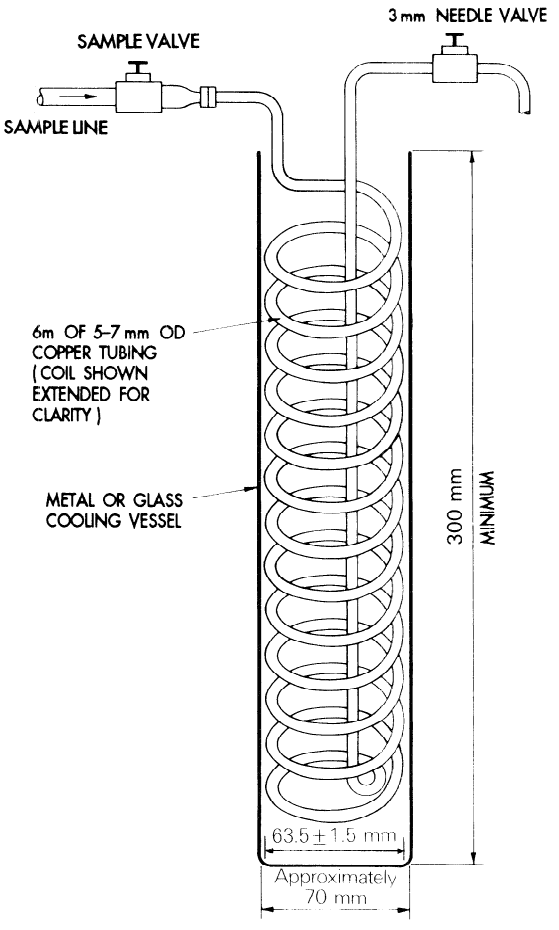


Figure 2 – Precooling equipment.

5.3 Copper wire, (1,6 ± 0,1) mm diameter, (300 ± 10) mm long.

5.4 Cooling coil, of minimum length 6 m, and of 5 mm to 7 mm outside diameter (O.D.) copper tubing wound to a diameter of (63,5 ± 1,5) mm O.D., and assembled in a suitable cooling bath (5.5). See figure 2.

5.5 Cooling bath, of suitable dimensions to receive the cooling coil and to accommodate any frothing that may occur when filled with a suitable liquid (4.2).

5.6 Syringe, 2 ml (ordinary medical) syringe, graduated in 0,1 ml and fitted with a needle (200 ± 5) mm long, or a 2 ml pipette, graduated in 0,1 ml.

5.7 Thermometers, conforming to IP Appendix A or ASTM E1.

5.7.1 Low range, −80 °C to +20 °C IP 2C/ASTM 6C.

5.7.2 High range, −35 °C to +70 °C IP 74C or −20 °C to +50 °C ASTM 57C.

NOTE 4 For routine testing a general purpose thermometer with 0,5 °C subdivisions and a maximum scale error of 0,5 °C may be used.

5.8 Filter paper, qualitative grade, white, 125 mm diameter.

5.9 Solvent wash bottle.

5.10 Water bath, controlled at (38 ± 2) °C.

NOTE 5 A 250 ml beaker with an appropriate heating source is suitable.

NOTE 6 When a thermometer and/or a water bath are not available, e.g. when performing a field test, a satisfactory alternative for screening is to warm the tip of the centrifuge tube with the hand.

5.11 Clamp, suitable for holding the centrifuge tube during weathering.

5.12 Mechanical refrigerator, with coolant temperature in the range −46 °C to −48 °C, if required (see 7.1).

6 Preparation of apparatus

Wash all glassware that is to be used in the test in the selected solvent (4.1). Add 10 ml of a fresh sample of solvent to the centrifuge tube (5.1). Mark the centre of the filter paper (5.8) with a pencil. Fill the syringe (5.6) with a portion of the solvent drawn from the centrifuge tube and direct 0,1 ml of the solvent to the mark on the paper. Hold the filter paper level during the solvent addition by such means as placing it on a 250 ml beaker. Allow the solvent to evaporate and note the persistence

of an oil ring by holding the dry filter paper between the eye and a bright incandescent light or strong daylight. Add further solvent in increments of 0,1 ml to cover a circle of approximately 30 mm – 35 mm in diameter on the filter paper with each addition. If no oil ring appears after 1,5 ml of solvent has been added, the solvent and glassware are satisfactory. If unsatisfactory, reclean the apparatus in accordance with the procedure given in this clause.

NOTE 7 The appearance of an oil ring indicates either improperly cleaned glassware or contaminated solvent.

7 Procedure

7.1 Insert the cooling coil (5.4) in the cooling bath (5.5). Add sufficient coolant (4.2) to cover the coil. Using either solid carbon dioxide (4.3) or a mechanical refrigerator (5.12) adjust the temperature of the bath and the coil to at least 2 °C below the boiling point of the material being sampled, e.g. −4 °C for butane and −44 °C for propane. Connect the coil to the sampling point and flush with the liquid material being sampled.

7.2 Using the cooled coil, rinse the centrifuge tube (5.1) with the material being sampled and then fill to the 100 ml mark. Immediately after filling insert the copper wire (5.3) into the tube and stopper the tube with either the loosely fitting cork or the cotton wool plug (5.2).

NOTE 8 The wire helps to prevent superheating and resulting bumping (erratic or excessive boiling), and the stopper keeps out air or moisture while the sample is weathering.

If more than 10 ml of sample is lost during this operation due to bumping, reject the sample and refill the tube.

7.3 Clamp the tube (5.11) and allow the sample to weather at ambient temperature.

NOTE 9 Artificial heating may be required if the ambient temperature is below 5 °C.

7.4 After weathering, and when the tube has attained ambient temperature, place the tip of the tube in the water bath (5.10) maintained at 38 °C. After 5 min withdraw the tube and examine. Record the volume of any residue to the nearest 0,05 ml and note if any adventitious matter is present.

7.5 Using a wash bottle (5.9), add solvent (4.1) to the tube and fill to the 10 ml mark, taking care to wash the complete surface of the inside of the tube. Using the syringe needle or pipette (5.6) stir the solvent–residue to ensure mixing.

As the oil-free solvent may become contaminated if stored in a plastics wash bottle, any solvent transferred to the wash bottle for the purposes of carrying out the test shall either be used in testing during the same day or be discarded.

7.6 Mark the centre of the filter paper (5.8) with a pencil. Fill the syringe or pipette (5.6) and place 1,5 ml of the solvent–residue onto the filter paper at such a rate to produce a wetted circle of approximately 30 mm to 35 mm in diameter. Hold the filter paper level during the solvent addition by such means as placing it on a 250 ml beaker.

7.7 Except when testing materials in the gasoline boiling range, after 2 min examine the filter paper by holding it in front of a bright light. If no oil ring persists discontinue the test and report the O number as zero. If a ring is discernible after 2 min, determine the minimum volume of the solvent–residue mixture, to the nearest 0,1 ml, which results in an oil ring persisting for 2 min, by adding varying volumes of the solvent–residue mixture to a series of clean filter papers. Record the minimum volume, to the nearest 0,1 ml, required to produce a persistent oil ring as the oil stain observation.

7.8 As materials in the gasoline boiling range leave an oil stain after 2 min at ambient temperatures below 5 °C, for these materials either conduct the test in an area where the temperature is above 5 °C, or if this is not possible, examine the filter paper for oil stain after 10 min. If the filter paper is examined after 10 min this fact shall be reported.

7.9 An acceptable alternative procedure for use in those cases where a product specification limit has been established is to continue incremental additions of the solvent–residue up to the volume equivalent to the limiting specification. If no persistent oil ring is produced by this volume the test is discontinued and the product reported as 'passing'.

8 Calculation

8.1 R number

Multiply the volume of residue obtained in 7.4 by 200.

8.2 O number

Divide 10 by the oil stain observation obtained in 7.6. If the oil stain observation equals or exceeds 1,5 ml, the result is recorded as zero.

9 Expression of results

9.1 Volumetric

Report the residue on evaporation to the nearest 0,05 ml, and the oil stain observation to the nearest 0,1 ml.

9.2 Normalized

Report the R number to the nearest 10, and the O number to the nearest 1.

10 Precision

The precision is expressed in terms of the normalized reporting units only; the data relating to the derivation of these values are no longer available.

The precision of the method is as given in tables 2 and 3.

Table 2 – Repeatability (*r*)

O number	*r*	R number	*r*
0 to 20	4	0 to 20	5
Above 20 to 40	6	Above 20 to 40	10
Above 40 to 100	8	Above 40 to 60	20

Table 3 – Reproducibility (*R*)

O number	*R*	R number	*R*
0 to 20	6	0 to 20	10
Above 20 to 40	8	Above 20 to 40	10
Above 40 to 100	12	Above 40 to 60	30

11 Test report

The test report shall contain at least the following information.

a) sufficient details for complete identification of the product tested;

b) a reference to this standard;

c) the result of the test (see clause 9);

d) any deviation, by agreement or otherwise, from the procedure specified;

e) the date of the test.

blank

BS 2000 Series

The Institute of Petroleum

61. New Cavendish Street
London
W1G 7AR

Tel: +44 (0)20 7467 7100
Fax: +44 (0)20 7255 1472
www.petroleum.co.uk

Buying Parts of BS 2000

Orders for BS 2000 publications should be addressed to:

The Library at the Institute of Petroleum

British Standards Institution — Customer services
389 Chiswick High Road
London
W4 4AL

Tel: +44 (0)20 8996 9001
Fax: +44 (0)20 8996 7001
www.bsi-global.com

Order hard copy standards securely via:
www.bsi-global.com/bsonline

Copyright

IP
THE INSTITUTE OF PETROLEUM