INTERNATIONAL STANDARD

ISO 9120

First edition
1997-12-01

Corrected and reprinted
1998-05-15

Petroleum and related products — Determination of air-release properties of steam turbine and other oils — Impinger method

Pétroles et produits connexes — Détermination de l'aptitude à la désaération des huiles pour turbine à vapeur et autres huiles — Méthode Impinger

Reference number
ISO 9120:1997(E)

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.

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.

International Standard ISO 9120 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

Annex A forms an integral part of this International Standard.

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X.400 c=ch; a=400net; p=iso; o=isocs; s=central

Printed in Switzerland

Petroleum and related products — Determination of air-release properties of steam turbine and other oils — Impinger method

WARNING — The use of this International Standard may involve hazardous materials, operations and equipment. This International Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this International Standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard specifies a method for the estimation of the ability of a petroleum-type steam turbine oil to be separated from entrained air.

NOTES

1 The impinger method described in this International Standard is used for determining the air-release ability of the oils tested. Air bubbles dispersed in the oil influence the compressibility and can cause malfunctions. This method may not be suitable for ranking oils in applications where residence times are short and gas contents high.

2 Agitation of lubricating oil with air in equipment such as bearings, couplings, gears, pumps and oil return lines, may produce a dispersion of finely divided air bubbles in the oil. If the residence time in the reservoir is too short to allow the air bubbles to rise to the oil surface, a mixture of air and oil will circulate through the lubricating oil system. This may result in an inability to maintain oil pressure (particularly with centrifugal pumps), incomplete oil films in bearings and gears, and poor hydraulic system performance or failure.

3 This test measures the time for the entrained air content to fall to the relatively low value of 0,2 % (*V/V*) under a standardized set of test conditions, and hence permits the comparison of the ability of oils to separate entrained air under conditions where a separation time is available. Although the significance of the test results is not fully established, sponginess and lack of sensitivity of the control systems of some turbines may be related to the air release properties of the oil. System design and system pressure are other variables.

4 For the purposes of this International Standard, the expression "%(*V/V*)" is used to represent the volume fraction of a material.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standards. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 3170:1988, *Petroleum liquids — Manual sampling.*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

ISO 4259:1992, *Petroleum products — Determination and application of precision data in relation to methods of test.*

ISO 6353-2:1983, *Reagents for chemical analysis — Part 2: Specifications — First series.*

ISO 6353-3:1987, *Reagents for chemical analysis — Part 3: Specifications — Second series*.

ISO 6743-4:1982, *Lubricants, industrial oils and related products (class L) — Classification — Part 4: Family H (Hydraulic systems).*

3 Definitions

For the purposes of this International Standard, the following definition applies.

3.1 air release: Time, in minutes, in which air dispersed in the oil is reduced to 0,2 % of the volume, at a specified temperature.

4 Principle

Air is blown into the oil under pressure at a test temperature of 25 °C, 50 °C or 75 °C. After the air flow is stopped, the escape of the dispersed air bubbles from the oil is followed by determinations of density as a function of time. The time for the dispersed air content to reduce to 0,2 % (*V*/*V*) is recorded from this graphical representation. Manual and automated apparatus are available and suitable.

5 Reagents and materials

During the analysis, unless otherwise stated, use only reagents specified in ISO 6353-2 and ISO 6353-3, or reagents of analytical reagent grade and water conforming to grade 3 of ISO 3696:1987.

5.1 Methylbenzene (toluene)

5.2 Acetone

5.3 Methanol

5.4 Gum solvent, consisting of equal volumes of toluene (5.1), acetone (5.2), and methanol (5.3).

NOTE — Formerly, 1,1,1-trichloroethane was specified as the gum solvent, but because of its toxicity and environmental harmfulness, alternatives should be used. Any acceptable solvent system that effectively removes the gum deposits from the glassware may be used.

5.5 Air, a regulated supply of dry, filtered and oil-free air.

5.6 Cleaning solution, Chromosulfuric acid or another strongly oxidizing cleaning solution.

NOTE — A strong oxidizing cleaning solution is required to remove traces of silicones that are frequently present in oils as anti-foam agents, and would seriously affect the results of this test.

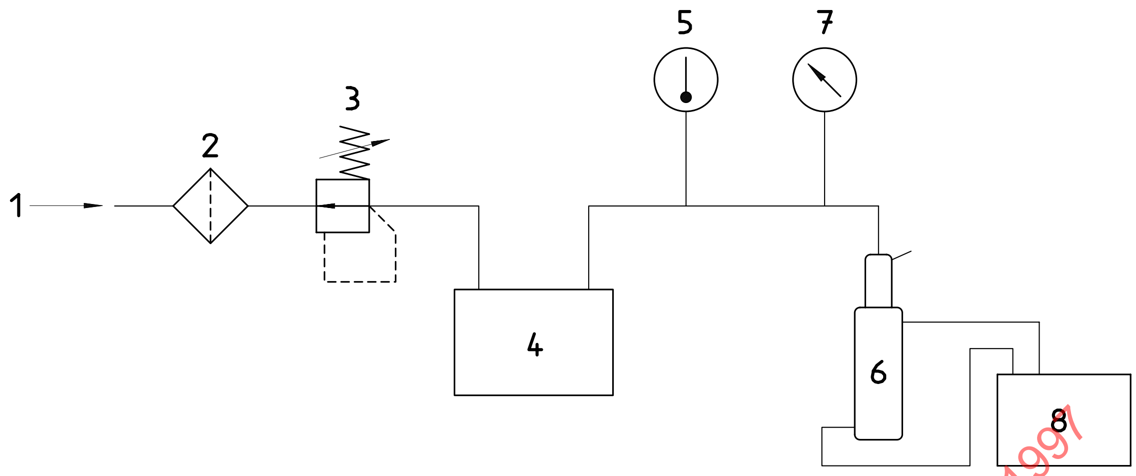
WARNING — Chromosulfuric acid is a health hazard. It is toxic, a recognized carcinogen as it contains Cr(VI) compounds, highly corrosive and potentially hazardous in contact with organic materials. When using chromosulfuric acid cleaning solution, eye protection and protective clothing are essential. Never pipette the cleaning solution by mouth. After use, do not pour cleaning solution down the drain, but neutralize it with great care owing to the concentrated sulfuric acid present, and dispose of it in accordance with standard procedures for toxic laboratory waste (chromium is highly dangerous to the environment).

Non-chromium containing, strongly oxidizing acid cleaning solutions are also highly corrosive and potentially hazardous in contact with organic materials, but do not contain chromium which has its own special disposal problems.

6 Apparatus

6.1 A schematic diagram of the apparatus is shown in figure 1 and consists of the parts specified in 6.1.1 to 6.1.5.

6.1.1 Test vessel, of borosilicate glass, of the design illustrated in figure 2. It consists of a jacketed sample tube fitted with an air inlet capillary, baffle plate and air outlet tube. The two parts of each test vessel shall be marked in such a manner as to assure correct connections.



1	Compressed air	5	Pressure gauge
2	Air filter	6	Test vessel
3	Pressure reducing valve	7	Thermometer
4	Heater	8	Circulating bath

Figure 1 — Assembly of apparatus

6.1.2 Pressure gauge, with a range of 0 kPa to 35,5 kPa.

6.1.3 Temperature sensors

6.1.3.1 Thermometer, conforming to the specifications given in annex A, or a temperature sensor of at least equivalent performance, for monitoring the compressed air temperature.

6.1.3.2 Temperature sensor, having a range of 20 °C to 100 °C, with an accuracy of 0,1 °C, for monitoring the sample temperature (see note in 9.1).

6.1.4 Heater, for the compressed air. A coil of copper tubing immersed in the circulating bath (6.1.5) is suitable at 25 °C, but additional heating is necessary at 50 °C and 75 °C. Use an additional bath or a separate steam or electric heat exchanger at these higher temperatures. The temperature of the air shall be measured as close as possible to the test vessel (6.1.1).

6.1.5 Circulating bath, approximately 10 l capacity with a flow rate of 10 l/min, and capable of maintaining the test cell at the specified temperature ± 0,1 °C. It shall not be constructed of glass.

6.2 Density balance, accurate to 0,5 kg/m^3, with a sinker of 5 ml or 10 ml displacement, with a round or tapered bottom. The length of the sinker shall be 80 mm ± 1,5 mm.

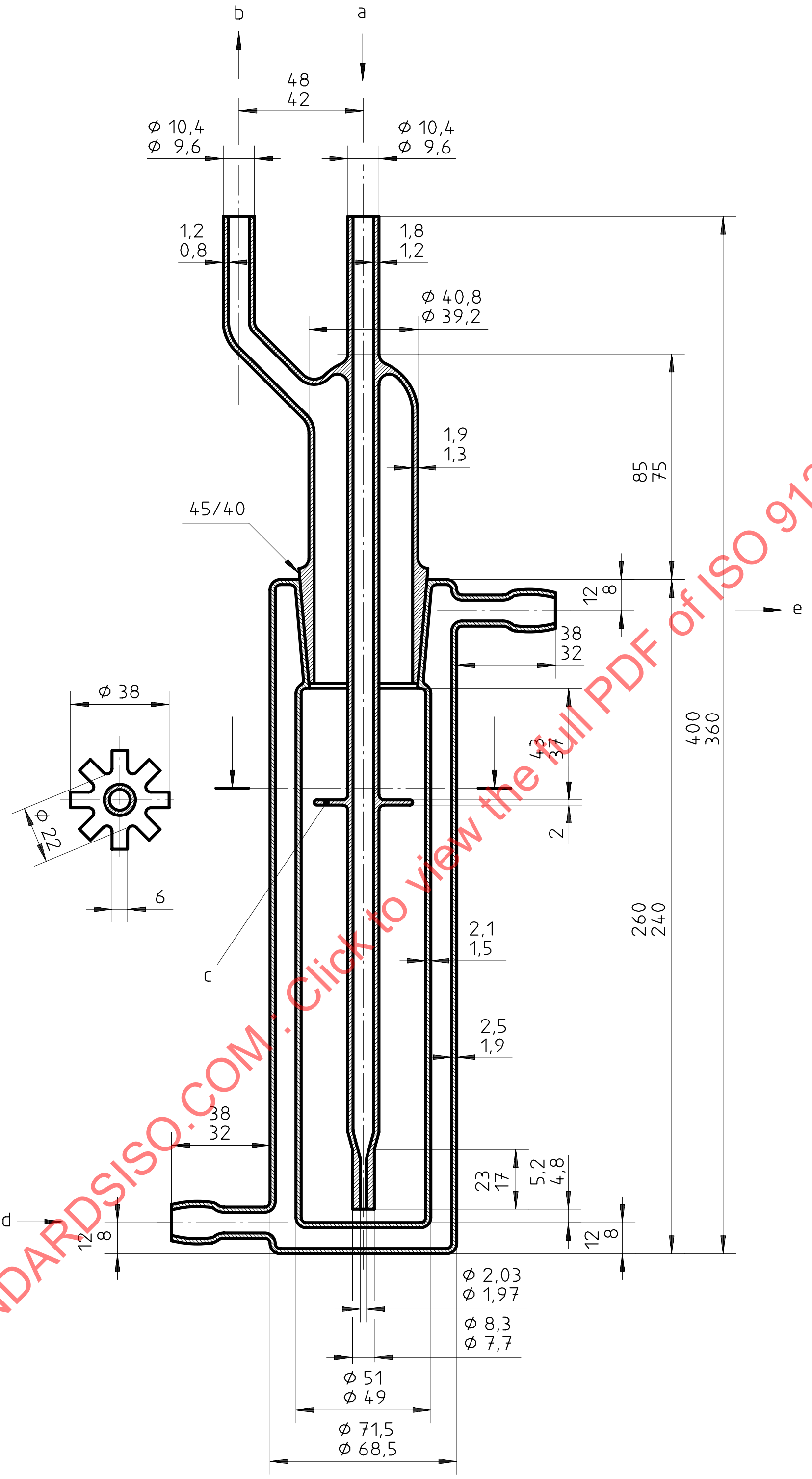
6.3 Oven, capable of controlling temperatures up to 100 °C.

6.4 Timer, electronic or manual, graduated and accurate to 1 s or better.

6.5 Graduated cylinder, 250 ml capacity.

6.6 Platinum wire

Dimensions in millimetres



a	Air inlet
b	Air outlet
c	Baffle plate
d	Circulating fluid in
e	Circulating fluid out

Figure 2 — Test vessel

7 Sampling

Unless otherwise specified, obtain the sample by the procedure described in ISO 3170.

8 Preparation of apparatus

8.1 Clean the interior of the test vessel, including the air inlet and sinker, and all other glassware coming into contact with the sample, in the manner described in 8.1.1 to 8.1.4.

8.1.1 Rinse away the oil and gum residue with gum solvent (5.4), and dry by blowing with a gentle stream of air (see note to 8.1.4).

8.1.2 Immerse the apparatus in cleaning solution (5.6) for a minimum of 12 h.

8.1.3 Rinse thoroughly with tap water followed by water as mentioned in clause 5.

8.1.4 Rinse with acetone, and dry in a gentle stream of air.

NOTE — Evaporation of solvents by air blowing should be carried out in an area where fumes are dispersed (vented).

9 Procedure

9.1 Assemble the apparatus to the configuration shown in figure 1. Establish the conditions that give a sample temperature ± 0,1 °C of the test temperature. Set the compressed air temperature to within 0,2 °C of the test temperature and set the circulating bath to give a sample temperature ± 0,1 °C of the specified test temperature. The test temperature shall be related to the kinematic viscosity at 40 °C of the oil under test in the following manner.

Oils with a kinematic viscosity of below 9 mm^2/s	25 °C
Oils with a kinematic viscosity from 9 mm^2/s to 90 mm^2/s	50 °C
Oils with a kinematic viscosity above 90 mm^2/s	75 °C

NOTE — The sample temperature can most conveniently be measured by means of a temperature sensor (6.1.3.2) consisting of a fine resistance or thermocouple probe fed through the outlet pipe.

9.2 Warm approximately 200 ml of the oil to be tested in the oven (6.3) controlled at a temperature 10 °C above the test temperature.

9.3 Pour 180 ml of the warmed sample into the test vessel (see 9.11).

9.4 Allow the sample to reach the test temperature.

NOTE — 20 min is sufficient time for most samples.

9.5 Warm the sinker of the density balance to the test temperature in an air bath for at least 20 min. When the sinker has reached the test temperature, immerse it in the sample ensuring that no air bubbles adhere to it. Attach the sinker to the beam of the density balance by means of the platinum wire (6.6) so that the bottom of the sinker is 10 mm ± 2 mm from the bottom of the test vessel.

9.6 Read and record the density of the oil to the nearest 0,5 kg/m^3.

9.7 Return the sinker to the air bath and replace it with the air inlet tube (see figure 2). After 5 min, start the supply of air at a gauge pressure of 20 kPa at the test temperature. Start the timer (6.4) and maintain the pressure and temperature throughout the test.

9.8 After 420 s ± 1 s, shut off the air and quickly remove the air inlet tube from the test vessel. Immediately re-start the timer and immerse the sinker in the manner described in 9.5. Follow the procedure given in 9.9 or 9.10 below.

NOTE — In certain oils, the volume of air/oil dispersion may cause the top of the sinker to be immersed in foam, and thus initial results are correspondingly in error.

9.9 Air release value. Set the density balance to a value that corresponds to 0,2 % (*V*/*V*) air in the sample. Record the time from the air shut-off to the null point on the balance to the nearest 0,1 min. If the null point is not reached in 30 min, discontinue the test (see 9.11).

9.10 Air release/time curve. For some applications, the shape of the air release/time curve is required. In such a case, read the density at 1 min intervals for the first 15 min, and at 2 min intervals thereafter, to the nearest 0,5 kg/m^3. Discontinue the test when two readings agree to within 0,5 kg/m^3.

9.11 Certain oils, e.g. HFAC, HFAS and HFC fluids according to ISO 6743-4, may lose light components during the air saturation (see 9.7) thus changing their effective density, ρ_E. This will be noted if the null point is reached or exceeded instantaneously, or if the final reading in 9.10 is above the density value obtained in 9.6. Where an air release value is required for such oils, the density after 10 min of release in 9.9 can be determined, or the final density in 9.10 used in place of the initial density, ρ_0, in clause 10, and this fact reported (see 11.3). If the loss causes any part of the sinker to be exposed, discontinue the test and repeat using 190 ml of sample.

10 Calculation

The percentage volume of dispersed air in the air-in-oil dispersion, *L*, is calculated from the following equation:

$$L = \frac{100\,(\rho_0 - \rho_x)}{\rho_0 - \rho_t}$$

where

ρ_0 is the density of the sample free from air, in kilograms per cubic metre (9.6);

ρ_x is the density of the air-in-oil dispersion after *x* minutes, in kilograms per cubic metre;

ρ_t is the density of air at the test temperature, in kilograms per cubic metre.

NOTE — An approximation of (ρ_0 - 1,7) kg/m^3 for (ρ_0 - ρ_x) relates to a 0,2 % (*V*/*V*) dispersion of air in an oil of a density of 850 kg/m^3 at the test temperature. The 1,7 value changes by 0,1 for every 50 kg/m^3 in oil density, assuming that the initial and final densities are the same.

11 Expression of results

11.1 Report the air release value, to the nearest 0,1 min, together with the test temperature. If the test was discontinued after 30 min, report the result as “greater than 30 min”.

11.2 When required, report a graphical representation of air release plotted against time, with the test temperature clearly appended.

11.3 When the effective density, ρ_E, has been used in place of the initial density, ρ_0, to express the air release value, append to the result “modified density”.

12 Precision

The precision, as determined by statistical examination of inter-laboratory test results in accordance with ISO 4259, is given in 12.1 and 12.2.

NOTE — The precision values in 12.1 and 12.2 may not be attained by manual operation on oils of very low kinematic viscosity at 40 °C, e.g. shock absorber oils. Such oils were not included in the sample matrix deriving this precision statement.

12.1 Repeatability. The difference between successive test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the normal and correct operation of the test method, exceed the value below only in one case in twenty.

$$r = 0{,}5X^{0{,}5}$$

where X is the mean of the values being compared.

12.2 Reproducibility. The difference between two single and independent results obtained by different operators working in different laboratories on nominally identical test material would, in the normal and correct operation of the test method, exceed the value below only in one case in twenty.

$$R = 1{,}3X^{0{,}5}$$

where X is the mean of the values being compared.

NOTE — Comparison of these precision values with those developed by DIN over the three bands covering 0 min to 5 min, 5 min to 10 min and 10 min to 15 min, show a slightly higher reproducibility, but a lower repeatability than those of DIN.

13 Test report

The test report shall contain at least the following information:

a) a reference to this International Standard;

b) the type and complete identification of the product tested;

c) the result of the test (see clause 11);

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

e) the date of the test.