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МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

Plastics — Small enclosures for conditioning and testing using aqueous solutions to maintain relative humidity at constant value

Plastiques — Petites enceintes de conditionnement et d'essai utilisant des solutions aqueuses pour maintenir l'humidité relative à une valeur constante

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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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 483 was prepared by Technical Committee ISO/TC 61, *Plastics*.

It cancels and replaces ISO Recommendation R 483 : 1966, of which it constitutes a technical revision.

Annex A forms an integral part of this International Standard.

Plastics — Small enclosures for conditioning and testing using aqueous solutions to maintain relative humidity at constant value

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1 Scope

1.1 This International Standard establishes guidelines for the construction and use of enclosures with volumes less than 200 dm³, in order to obtain atmospheres of constant relative humidity at given temperatures, using saturated aqueous salt solutions, glycerol/water solutions or sulfuric acid/water solutions, for conditioning and testing plastics.

It specifies the procedures to be followed to maintain the relative humidities of the conditioning and testing atmospheres within the required tolerances, at the temperatures specified by particular International Standards.

Information is given concerning the methods of producing desired humidities in these enclosures at temperatures from 5 °C to 60 °C. The relative humidity values indicated are average values, in per cent, with permissible deviations of ± 2 .

1.2 The procedures described are intended for conditioning small quantities of materials prior to test, and for such tests as may be carried out entirely within a small enclosure, e.g. electrical tests.

The guidelines described do not apply to enclosures requiring frequent opening.

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International

Standard. At the time of publication, the edition indicated was 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 edition of the standard given below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 291 : 1977, *Plastics — Standard atmospheres for conditioning and testing*.

3 Reagents and solutions

3.1 Salts (see table 1), of recognized analytical grade.

3.2 Glycerol, chemically pure.

3.3 Sulfuric acid, chemically pure.

3.4 Distilled water, or water of equivalent purity.

3.5 Saturated aqueous salt solutions (see table 1), with an excess of salt covered by the solution for the maintenance of saturation.

3.6 Aqueous glycerol solutions (see table 2), of specified concentrations.

3.7 Aqueous sulfuric acid solutions (see table 3), of specified concentrations.

Table 1 — Relative humidity of air over saturated aqueous salt solutions at temperatures between 5 °C and 60 °C

Saturated aqueous salt solutions	Relative humidity (%) at temperature θ									
	5 °C	10 °C	15 °C	20 °C	25 °C	30 °C	35 °C	40 °C	50 °C	60 °C
Potassium hydroxide (KOH) ¹⁾	14	13	10	9	8	7	6	6	6	—
Lithium chloride (LiCl.xH ₂ O) ²⁾	12	12	12	12	12	11	11	11	11	11
Potassium acetate (CH ₃ COOK)	25	24	24	23	22	22	21	20	—	—
Magnesium chloride hexahydrate (MgCl ₂ .6H ₂ O) ²⁾	34	34	34	33	33	33	32	32	31	30
Potassium carbonate dihydrate (K ₂ CO ₃ .2H ₂ O) ²⁾	46	45	44	44	43	42	41	40	38	36
Magnesium nitrate hexahydrate [Mg(NO ₃) ₂ .6H ₂ O] ²⁾	58	57	56	54	53	51	50	48	46	43
Sodium dichromate dihydrate (Na ₂ Cr ₂ O ₇ .2H ₂ O)	59	58	56	55	54	52	51	50	47	—
Ammonium nitrate (NH ₄ NO ₃) ³⁾	—	73	69	65	62	59	55	53	47	42
Sodium nitrite (NaNO ₂) ^{2) 4)}	—	—	—	66	64	63	62	61	60	58
Sodium chloride (NaCl) ²⁾	75	75	75	75	75	75	75	75	75	75
Ammonium sulfate [(NH ₄) ₂ SO ₄] ^{2) 3)}	82	82	81	81	80	80	80	79	79	—
Potassium chloride (KCl)	88	87	86	86	85	84	84	83	81	80
Potassium nitrate (KNO ₃) ²⁾	97	96	95	94	93	91	89	88	85	82
Potassium sulfate (K ₂ SO ₄)	98	98	97	97	97	96	96	96	96	96

1) Potassium hydroxide solution is corrosive and should not be allowed to come into contact with the skin.
 2) These salts are recommended for particular humidity ranges because the change with temperature is very small over the range 20 °C to 30 °C.
 3) Ammonium salts may cause corrosion of copper parts.
 4) The addition of 1 % to 2 % sodium tetraborate has been found to reduce discoloration of sodium nitrite solutions. The relative humidity is reduced by 1 % R.H. at 20 °C by this addition.

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Table 2 — Relative humidity of air over aqueous glycerol solutions at temperatures between 0 °C and 70 °C

Approximate glycerol concentration % (m/m)	Refractive index at 25 °C n_D^{25}	Relative humidity (%) at temperature θ			
		0 °C	25 °C	50 °C	70 °C
10	1,346 3	97,7	98,0	98,2	98,4
15	1,356 0	95,6	96,0	96,4	96,7
25	1,360 2	94,5	95,0	95,5	95,8
35	1,377 3	89,2	90,0	90,7	91,2
45	1,390 5	84,0	85,0	85,9	86,6
52	1,401 5	78,8	80,0	81,1	81,8
58	1,410 9	73,7	75,0	76,2	77,0
64	1,419 1	68,6	70,0	71,3	72,2
69	1,426 4	63,4	65,0	66,4	67,3
74	1,432 9	58,4	60,0	61,4	62,5
77	1,438 7	53,3	55,0	56,5	57,6
81	1,444 0	48,3	50,0	51,5	52,6
84,5	1,448 6	43,3	45,0	46,6	47,7
87,5	1,452 9	38,3	40,0	41,6	42,7

Table 3 — Relative humidity of air over aqueous sulfuric acid solutions at temperatures between 5 °C and 50 °C

Sulfuric acid concentration % (m/m)	Density at 25 °C g/cm ³	Density at 23 °C g/cm ³	Relative humidity (%) at temperature θ										
			5 °C	10 °C	15 °C	20 °C	23 °C	25 °C	30 °C	35 °C	40 °C	45 °C	50 °C
5	1,030 0	1,030 7	98	98	98	98	98	98	98	98	98	98	98
10	1,064 0	1,064 8	95,5	95,5	95,5	95,5	95,5	95,5	95,5	95,5	95,5	95,5	95,5
15	1,099 4	1,100 5	92,5	92,5	92,5	92	92	92	92,5	92,5	92,5	92	92
20	1,136 5	1,137 6	87,5	88	88	88	88	88	88	88	88,5	88,5	88,5
25	1,175 0	1,176 4	81,5	82	82	82	82,5	82,5	82,5	83	83	83	83
30	1,215 0	1,216 4	74	74,5	74,5	75	75	75	75,5	76	76	76,5	76,5
35	1,256 3	1,257 7	64,5	65	65,5	66	66,5	66,5	67	67,5	67,5	68	68,5
40	1,299 1	1,300 5	54	55	55,5	56	56,5	56,5	57	57,5	58	58,5	59
45	1,343 7	1,345 2	43	44	44,5	45	45,5	46	46,5	47	48	48,5	49
50	1,391 1	1,397 2	32,5	33	34	34,5	35	35	35,5	36,5	37	37,5	38,5
55	1,441 2	1,442 8	22,5	23	24	24,5	25	25	25,5	26	27	27,5	28
60	1,494 0	1,495 7	14	14,5	15	15,5	16	16	17	17,5	18	18,5	19
65	1,549 0	1,550 7	8	8	8,5	9	9	9,5	9,5	10	10,5	11	11,5
70	1,605 9	1,607 7	3,5	4	4	4	4,5	4,5	4,5	5	5	5,5	6

4 Apparatus

4.1 If the volume of the enclosure is less than 15 dm³, the enclosure shall be a **container**, of simple shape, with internal walls which are easily cleaned and are inert with regard to the solutions used.

The temperature shall be kept constant and uniform at all points by placing the container in an oven, a water bath, or a laboratory conditioned at constant temperature.

A thermometer shall be placed inside the container to take temperature readings during the test to ± 1 K.

Air circulation is generally recommended, particularly when hygroscopic materials are being tested. Uniform relative humidity can only be obtained by using a fan with blades located just above the aqueous solution.

If the enclosure is intended only for conditioning practically non-hygroscopic materials or for conditioning over a long time in relation to the water-absorption capacities of the specimens placed in it, air circulation may not be necessary.

Without air circulation, the height of the container shall not exceed the smallest dimension of the free surface of the solution. In containers with air circulation, this height may reach 1,5 times the smallest dimension of this free surface.

4.2 If the volume of the enclosure is between 15 dm³ and 200 dm³, the **enclosure** shall also be of simple shape, with internal walls that are easily cleaned and are inert with regard to the solutions used.

The enclosure shall, however, be insulated from the surrounding environment in such manner that its internal regulation apparatus and its air circulation can maintain a constant temperature at all points of its usable volume within the tolerance specified or within the tolerance on this temperature as required by the tolerance on the relative humidity.

The internal height of the usable volume shall not exceed 1,5 times the smallest dimension of the free surface of the solution.

The cover of the enclosure shall be of an electrically insulating material and shall be equipped with wet-and-dry-bulb thermometers readable to 0,1 K from the exterior.

The air shall circulate around the bulbs of these thermometers at a velocity of about 3 m/s, after regulation of its relative humidity by passage between the surface of the aqueous solution and a suitable air duct. To achieve this result, air circulation may, for example, be arranged in such a manner that the passage over the surface of the solution is preceded by heating, remote-controlled by a contact thermometer located in the usable volume of the enclosure at such a point that the temperature specified at this location is maintained in accordance with the specifications. (See also annex A.)

At the end of the circuit, after passage through the usable volume and before passage through the heater, the air shall be cooled by suitable means to a temperature above the dew point, at least when the enclosure and its contents are in equilibrium. For example, for the temperature and relative humidity specified in ISO 291 (23 °C and 50 % relative humidity), the temperature of the cooled air shall be greater than, or equal to, 12 °C.

5 Procedure

5.1 General

5.1.1 Enclosure load

5.1.1.1 Overload in a small enclosure can lower the rate of reaching the specified relative humidity in the container so that it may be necessary to wait a very long time for a state of equilibrium to be established. In view of the fact that the load limit depends on the conditioned material's water absorption capacity and the difference between its moisture content and the humidity of the enclosure, this limit cannot be specified. Only periodic measurements of the mass of the specimens and periodic readings of the enclosure's temperature and relative humidity measurement instruments can indicate when the required state of equilibrium has been reached.

NOTES

1 Even without any variation in the moisture content of the air, differences in the temperature of the air from one point of the usable volume to another will produce differences in the relative humidity. The relative humidity will be low at points at which the temperature is high, and high at points at which the temperature is low. For example, for standard test atmospheres, a difference of 0,5 K causes a difference of nearly 2 % relative humidity; at 90 % relative humidity, the corresponding difference is approximately 3 % R.H.

2 Variations of the temperature at a given point with time will tend to produce a similar effect. If the fluctuations are slow, the changes in relative humidity will be reduced by the action of the saturated solution.

5.1.1.2 It is recommended that the following guidelines be observed.

Specimens with a large volume (V) and small surface area (S) (e.g. a sphere) have a high ratio of V/S . In enclosures without air circulation, the total surface areas of the specimens in this case should not exceed the surface area of the solution.

With smaller values of V/S , the sum of the total surface areas of the specimens may be increased proportionately.

For specimens with a small value of V/S (e.g. a film), the total surface area may be up to three times the surface area of the solution.

In enclosures with air circulation, the total surface area of the specimens can be three times the area recommended for enclosures without air circulation.

It is necessary to maintain cleanliness of the surface of the solution and of the interior walls of the enclosure.

5.1.2 Start of conditioning period

The duration of conditioning indicated in specifications for the specimens is counted, or testing in a specified atmosphere is started, after the introduction of the specimens, only from the time at which the enclosure has regained its equilibrium state within the allowable tolerances, as indicated by readings of the thermometers and hygrometer.

In any case, the time required for the container or empty enclosure to reach steady-state conditions must be known, on the basis of clearly defined initial and final conditions, in order to determine, without opening, the minimum required residence time to obtain satisfactory conditioning, depending on the type and moisture content of the specimens placed in it. Testing of the change in mass of these specimens, combined with observation of the thermometers and hygrometer, will make it possible to determine this state of equilibrium.

The temperature shall be constant to ± 1 K.

5.2 Method A: Saturated aqueous salt solutions

This method is preferred whenever there is a salt whose saturated aqueous solution produces the desired relative humidity at the given temperature (see clause 3 and table 1).

The saturated aqueous salt solution shall be placed in the enclosure sufficiently in advance to ensure that the atmosphere has reached equilibrium at the time of measurement.

An excess of solid salt shall be in contact with the solution throughout the entire duration of conditioning or testing.

The enclosure shall be cleaned frequently between use in order to remove salt deposits (creeping salts) outside the tray used to hold the solution.

5.3 Method B: Aqueous glycerol solutions

This method requires more attention than the preceding one because the concentration of the glycerol/water mixture must be maintained practically constant. Specimens that absorb a large amount of water can cause significant change in the concentration. This may also occur if the load in the enclosure is too large in relation to the amount of solution used.

The concentration of an aqueous glycerol solution, as indicated by its refractive index at 25 °C, is related to the desired relative humidity and temperature (table 2) and may be calculated between 0°C and 70 °C by the formula

$$[715,3 (n_D^{25} - 1,3333) + A]^2 = (100 + A)^2 + A^2 - (RH + A)^2$$

where

A is a parameter related to temperature θ by the equation

$$A = 25,60 - 0,1950 \theta + 0,0008 \theta^2$$

RH is the relative humidity expressed in per cent;

n_D^{25} is the refractive index of the aqueous glycerol solution at 25 °C for the sodium D line;

θ is the temperature, in degrees Celsius, of the solution.

This formula makes it possible to calculate the desired relative humidity with an accuracy of $\pm 0,2$ % R.H. at a temperature of 25 °C.

The refractive index of the aqueous glycerol solution shall be regularly checked with a refractometer covering the range 1,33 to 1,47 with a precision of $\pm 0,001$, and in any case before and after each test or conditioning.

For the conditioning atmospheres generally used, the corresponding refractive indexes are given in clause 6 (tables 4 and 5).

5.4 Method C: Aqueous sulfuric acid solutions

WARNING — Sulfuric acid is corrosive and attacks the skin. Before handling sulfuric acid, it is essential to observe the relevant safety rules. Always wear safety goggles when handling concentrated sulfuric acid. Attacks by sulfuric acid penetrate very deep. Immediately wash the affected parts with copious quantities of water. In case of eye damage, or severe attack of the skin, immediately call a doctor, because sulfuric acid continues to penetrate into the flesh.

The mixing of concentrated sulfuric acid and water leads to considerable liberation of heat. Sulfuric acid must always be added to the water and never the opposite, because of the danger of splattering when water is added to the acid.

The enclosure shall be a container that is non-absorbent to water, gas-tight and corrosion-resistant, and with a height generally not exceeding one-quarter of the circumference of the base. The capacity of the enclosure shall be a maximum of 1 dm³. The total surface area of the specimens shall not exceed that of the sulfuric acid solution.

The values given in table 3 for the relative humidity (RH) of the air above different aqueous sulfuric acid solutions have been taken from the literature.

They are mean values, in per cent, with permissible deviations of ± 2 .

These sulfuric acid concentrations are given as an aid in the preparation of the required aqueous sulfuric acid solutions. The effective concentration of the aqueous sulfuric acid solution depends on the absorption of water from and release of water to the surrounding atmosphere. The concentration shall be determined at suitable intervals and, if necessary, corrected by the addition of water or sulfuric acid (see warning above).

The sulfuric acid concentration can be checked by measuring the density with a hydrometer. The accuracy of the calibrated hydrometer shall be $\pm 0,001$ g/ml. Analysis by titration is a more accurate method for determining the sulfuric acid concentration.

The temperature of the solution and of the air shall be measured before the start of a test and during a test in the enclosure, without causing any disturbance in the test atmosphere. The surface of the solution shall be free of pollutants.

6 Tolerances

To obtain standard atmospheres as defined in ISO 291, normal or close tolerances may be required.

6.1 Normal tolerances

Normal tolerances on the temperature and relative humidity and on the refractive index of the aqueous glycerol solution shall be as given in table 4.

Table 4 — Normal tolerances

Temperature °C	Relative humidity %	Refractive index of aqueous glycerol solution n_D^{25}
23 \pm 2	50 \pm 5	1,444 \pm 0,005
27 \pm 2	65 \pm 5	1,426 \pm 0,007

6.2 Close tolerances

Close tolerances on the temperature and relative humidity and on the refractive index of the aqueous glycerol solution shall be as given in table 5.

Table 5 — Close tolerances

Temperature °C	Relative humidity %	Refractive index of aqueous glycerol solution n_D^{25}
23 \pm 1	50 \pm 2	1,444 \pm 0,002
27 \pm 1	65 \pm 2	1,426 \pm 0,003

NOTES

1 In general, the permissible temperature variation in the enclosure will be less than the tolerance given in column 1 of tables 4 and 5 if the relative humidity is to be maintained within the limits given in column 2.

2 When, in special cases, an atmosphere of 20 °C and 65 % relative humidity is needed, the value of n_D^{25} is 1,426.

7 Test report

The report of a test in which small conditioning and testing enclosures are used shall include the following information:

- a reference to this International Standard;
- the conditioning and testing atmosphere specified (temperature and relative humidity);
- the characteristics of the enclosure used (volume, air circulation system);
- the solution used:
 - for saturated aqueous salt solutions, identify the salt used,
 - for aqueous glycerol solutions, give the refractive index,
 - for aqueous sulfuric acid solutions, give the specific gravity;
- any details not given in this International Standard and any incidents that occurred which are liable to have influenced the results;
- duration of exposure and date.

Annex A (normative)

General features of a conditioning enclosure with a volume of 15 dm³ or greater

The enclosure, preferably rectangular in shape, shall be constructed from inert and non-absorbent material fabricated so that any joints are leakproof and not subject to corrosion. The interior surface shall be easy to clean. Rounded joints facilitate cleaning.

The door shall have a double-glazed observation window, and shall be rigid to give an efficient seal when the door is closed. A sealing gasket shall be provided that can be easily replaced when necessary.

The tray for the salt solution in the bottom of the enclosure shall be made of impermeable material that does not craze and is unaffected by any saturated salt solution used. The surface area of the tray shall be as large as possible to obtain the maximum rate of moisture transfer. The tray shall be easy to wash completely free of the solution, when necessary.

A suitable fan and air ducts shall provide adequate air circulation over the surface of the salt solution and to all parts of the

working space. If wet-and-dry-bulb thermometers are used for measuring temperature and relative humidity, the air velocity at the measurement point shall be about 3 m/s. It is recommended that the fan be switched off automatically when the door is opened.

Provision shall be made for connections, such as leads used for electrical tests. Efficient seals shall be provided at the entry points of such leads, as well as the entry points of the thermometers, the fan shaft, etc.

Heating elements controlled by a thermostat and tubes for cooling water or another cooling agent shall be provided. The enclosures shall be thermally insulated from the external atmosphere. Condensation shall not occur on any surface exposed to the air stream inside to enclosure.

The temperature may be controlled by a thermostat in the air stream or by a thermostat in a water jacket surrounding the enclosure, depending on the type of apparatus.

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