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

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

Reference number
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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 483 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 6, *Ageing, chemical and environmental resistance*.

This second edition cancels and replaces the first edition (ISO 483:1988), in which the values of the relative humidity above the saturated salt solutions have been corrected to the values given in Reference [1] (see the Bibliography) which are generally accepted as the most reliable values by national physical laboratories.

Introduction

The properties of many plastics are strongly influenced by the relative humidity of the surrounding air.

This International Standard describes small cabinets for conditioning and testing of specimens at constant temperature and constant relative humidity above aqueous solutions of salts, sulfuric acid and glycerol.

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

1 Scope

This International Standard establishes guidelines for the construction and use of enclosures with volumes less than 200 dm^3, 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.

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 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 291, *Plastics — Standard atmospheres for conditioning and testing*

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

3 Principle

In a relatively small, closed container, aqueous solutions of specified concentration can be used to produce atmospheres with specific relative humidities under equilibrium conditions. Such atmospheres can be generated by using binary saturated aqueous salt solutions (see method A and Table 1), by using specific concentrations of aqueous glycerol solutions (see method B and Table 2) or by using aqueous sulfuric acid solutions (see method C and Table 3). In the case of method B and method C, it is necessary to measure and control the concentrations of these solutions.

Information is given concerning the methods of producing desired relative humidities in these enclosures at temperatures from 0 °C to 70 °C.

The relative-humidity values indicated have been taken from the literature [1]. The uncertainties involved are discussed in Clause 9.

4 Materials

4.1 Salts, of recognized analytical grade.

4.2 Glycerol, of reagent quality.

4.3 Sulfuric acid, of reagent quality.

4.4 Water, grade 3 as defined in ISO 3696:1987.

NOTE The exact purity of the chemicals used when the original conditions were developed is not known, but any differences between those chemicals and reagent-grade chemicals are expected to be negligible.

5 Aqueous solutions

5.1 Saturated aqueous salt solutions (see Table 1), with an excess of salt covered by the solution in order to keep the solution saturated.

5.2 Aqueous glycerol solutions (see Table 2), of specified concentrations.

5.3 Aqueous sulfuric acid solutions (see Table 3), of specified concentrations.

Table 1 (for use with method A) — Relative humidity of air over saturated aqueous salt solutions at temperatures between 5 °C and 70 °C

	Saturated aqueous salt solution	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	70 °C
1	Potassium hydroxide (KOH) [a]	14	12	11	9	8	7	7	6	6	6	
2	Lithium chloride ($LiCl{\cdot}xH_2O$) [b]	11	11	11	11	11	11	11	11	11	11	
3	Potassium acetate (CH_3COOK)		23	23	23	23	22					
4	Magnesium chloride hexahydrate ($MgCl_2{\cdot}6H_2O$) [b]	34	34	33	33	33	32	32	32	31	29	
5	Potassium carbonate dihydrate ($K_2CO_3{\cdot}2H_2O$) [b]	43	43	43	43	43	43					
6	Magnesium nitrate hexahydrate [$Mg(NO_3)_2{\cdot}6H_2O$] [b]	59	57	56	54	53	51	50	48	45		
7	Sodium bromide (NaBr)	64	62	61	59	58	56	55	53	51	50	
8	Potassium iodide (KI)	73	72	71	70	69	68	67	66	65	63	62
9	Sodium chloride (NaCl) [b]	76	76	76	75	75	75	75	75	74	75	
10	Ammonium sulfate [$(NH_4)_2SO_4$] [b, c]	82	82	82	81	81	81	80	80	79		
11	Potassium chloride (KCl)	88	87	86	85	84	84	83	82	81	80	
12	Potassium nitrate (KNO_3) [b]	96	96	95	95	94	92	91	89	85		
13	Potassium sulfate (K_2SO_4)	99	98	98	98	97	97	97	96	96		

[a] Potassium hydroxide solution is corrosive and should not be allowed to come into contact with the skin.

[b] These salts are recommended for particular humidity ranges because the change with temperature is very small over the range 20 °C to 30 °C.

[c] Ammonium salts may cause corrosion of copper parts.

Table 2 (for use with method B) — Relative humidity of air over aqueous glycerol solutions at temperatures between 0 °C and 70 °C

Approximate glycerol concentration % (by mass)	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	98	98	98	98
20	1,356 0	96	96	96	97
25	1,360 2	95	95	96	96
35	1,377 3	89	90	91	91
45	1,390 5	84	85	86	87
52	1,401 5	79	80	81	82
58	1,410 9	74	75	76	77
64	1,419 1	69	70	71	72
69	1,426 4	63	65	66	67
74	1,432 9	58	60	61	63
77	1,438 7	53	55	57	58
81	1,444 0	48	50	52	53
84,5	1,448 6	43	45	47	48
87,5	1,452 9	38	40	42	43

Table 3 (for use with method C) — Relative humidity of air over aqueous sulfuric acid solutions at temperatures between 5 °C and 50 °C

Sulfuric acid concentration % (by mass)	Density at 23 °C g/cm^3	Density at 25 °C g/cm^3	Relative humidity (%) at temperature θ				
			5 °C	15 °C	25 °C	35 °C	50 °C
5	1,030 7	1,030 0	98	98	98	98	98
10	1,064 8	1,064 0	96	96	96	96	96
15	1,100 5	1,099 4	93	93	92	93	92
20	1,137 6	1,136 5	88	88	88	88	89
25	1,176 4	1,175 0	82	82	83	83	83
30	1,216 4	1,215 0	74	75	75	76	77
35	1,257 7	1,256 3	65	66	67	68	69
40	1,300 5	1,299 1	54	56	57	58	59
45	1,345 2	1,343 7	43	45	46	47	49
50	1,397 2	1,391 1	33	34	35	37	39
55	1,442 8	1,441 2	23	24	25	26	28
60	1,495 7	1,494 0	14	15	16	18	19
65	1,550 7	1,549 0	8	9	10	10	12
70	1,607 7	1,605 9	4	4	5	5	6

6 Apparatus

6.1 If the volume of the enclosure is less than 15 dm^3, the enclosure shall be a container, of simple shape, with internal walls that are easily cleaned and are inert with regard to the solutions used.

The temperature shall be kept constant and uniform by placing the enclosure in an oven, a water bath or a laboratory conditioned at constant temperature. Any oven or room in which the enclosure is placed shall be capable of maintaining the temperature to within ± 1 °C.

Place a thermometer that is capable of reading to ± 0,1 °C inside the enclosure. Read the temperature 1 h after the chamber has been closed and at least once every 24 h thereafter. The temperatures recorded shall be within ± 1 °C of the temperature set. Any temperatures recorded outside these limits shall be indicated in the test report.

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 the free surface.

6.2 If the volume of the enclosure is between 15 dm^3 and 200 dm^3, the enclosure shall also be of simple shape, with internal walls that are easily cleaned and are inert with regard to the solutions used.

Place at least one thermometer that is capable of reading to ± 0,1 °C inside the enclosure. If several thermometers are used, distribute them as uniformly as possible throughout the usable volume. Read the temperature of each thermometer 1 h after the enclosure has been closed and at least once every 24 h thereafter. The temperatures recorded for each thermometer shall be within ± 1 °C of the temperature set. Any temperatures recorded outside these limits shall be indicated in the test report.

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 °C from the exterior.

Annex A provides information about the construction of large enclosures.

6.3 Method B requires a refractometer covering the range 1,330 to 1,470, reading to the nearest 0,001 units.

6.4 Method C requires a calibrated hydrometer accurate to ± 0,001 g/ml.

7 Procedure

7.1 General

7.1.1 Enclosure load

Unless otherwise specified, use the following as limits for specimen volume and/or surface area.

Specimens with a large volume V and small surface area S (e.g. spheres) have a high V/S ratio. In enclosures without air circulation, the total surface areas of such specimens shall 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. films), 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 may be three times the area recommended for enclosures without air circulation.

It is necessary to maintain the cleanliness of the surface of the solution and of the interior walls of the enclosure. Clean all interior surfaces of the chamber prior to each use. If the conditioning period is more than one week long, clean the interior surfaces and replace the salt solution at least weekly. During the cleaning process, the specimens shall be wrapped in a polyethylene film of at least 0,5 mm thickness and maintained at the conditioning temperature. If cleaning takes longer than 3 h, the test is invalidated.

7.1.2 Start of conditioning period

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

For each material conditioned/tested, determine the time necessary to reach the equilibrium state by placing the specimens in the enclosure set at the desired temperature and humidity and periodically removing and weighing the specimens until their mass is constant (to within ± 0,5 %) for three successive weighings.

7.2 Method A: Using saturated aqueous salt solutions

Because the atmosphere above a saturated solution is easy to reproduce, this method is preferred whenever a salt can be found whose saturated aqueous solution produces the desired relative humidity at the given temperature (see Clause 3 and Table 1).

Unless otherwise specified, place the saturated aqueous salt solution in the chamber 24 h prior to placing the specimens in the chamber. If data are available to show that the temperature and relative humidity reach the required values in less than 24 h, a shorter time may be used.

An excess of solid salt shall be in contact with the solution throughout the entire duration of conditioning or testing. When at rest, the undissolved salt shall always be covered by at least 1 mm of water.

7.3 Method B: Using 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 changes 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 relative humidity and temperature (see Table 2).

To produce the required relative humidity, the refractive index of the aqueous glycerol solution at 25 °C at the wavelength of the sodium D-line, n_D^{25}, is adjusted to the correct value. This is done by first calculating the refractive index at the wavelength of the sodium D-line, n_D^{25}, using the following equation:

$$n_\mathrm{D}^{25} = \frac{\sqrt{(100+A)^2 - U(2A+U)} - A}{715,3} + 1,333$$

where

U is the relative humidity, expressed in percent;

A is a parameter related to temperature θ of the solution (in degrees Celsius) by the equation:

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

The required concentration c of an aqueous glycerol solution can then be determined from the equation:

$$c = 699{,}93 n_{\mathrm{D}}^{25} - 929{,}31$$

Unless otherwise specified, check the refractive index at least once per week with a refractometer covering the range from 1,330 to 1,470 and reading to the nearest ± 0,001. If the refractive index deviates from the desired value by more than 0,002 units, replace the glycerol solution.

This method makes it possible to calculate the desired relative humidity with an accuracy of ± 2 % RH.

The refractive indexes corresponding to the conditioning atmospheres generally used are given in Tables 4 and 5.

7.4 Method C: Using aqueous sulfuric acid solutions

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

Mixing concentrated sulfuric acid and water leads to considerable liberation of heat. The sulfuric acid must always be added to the water and never the other way round, because of the danger of spattering if water is added to the acid.

The enclosure shall be a container that is non-absorbent to water, is gas-tight and corrosion-resistant, and has a height generally not exceeding one-quarter of the perimeter of the base. The capacity of the enclosure shall be, at the most, 1 dm^3. The total surface area of the specimens shall not exceed that of the sulfuric acid solution.

The values of the relative humidity of the air above different aqueous sulfuric acid solutions are given in Table 3.

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 checked 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 calibrated hydrometer. The accuracy of the hydrometer shall be 0,001 g/ml. Analysis by titration is a more accurate method of determining the sulfuric acid concentration.

The temperature of the solution and of the air shall be measured before the start of conditioning/testing and during conditioning/testing inside the enclosure without causing any disturbance to the atmosphere. The surface of the solution shall be free of dust and debris.

The densities corresponding to the conditioning atmospheres generally used are given in Tables 4 and 5.

8 Tolerances on standard atmospheres

8.1 General

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

8.2 Normal tolerances

To achieve these standard atmospheres with normal tolerances by method B or C, maintain the refractive index of the glycerol solution or the density of the sulfuric acid solution within the ranges given in Table 4.

Table 4 — Normal tolerances

Temperature °C	**Relative humidity** %	**Refractive index of aqueous glycerol solution** n_D^{25}	**Sulfuric acid density at 25 °C** g/cm^3
23 ± 2	50 ± 10	1,444 ± 0,011	1,32 ± 0,04
27 ± 2	65 ± 10	1,427 ± 0,016	1,26 ± 0,05

NOTE When, in special cases, an atmosphere of (20 ± 2) °C and (65 ± 10) % relative humidity is needed, the value of n_D^{25} is 1,426 ± 0,016 (method B) and the value of the sulfuric acid density is (1,26 ± 0,05) g/cm^3 (method C).

8.3 Close tolerances

To achieve these standard atmospheres with close tolerances by method B or C, maintain the refractive index of the glycerol solution or the density of the sulfuric acid solution within the ranges given in Table 5.

Table 5 — Close tolerances

Temperature °C	**Relative humidity** %	**Refractive index of aqueous glycerol solution** n_D^{25}	**Sulfuric acid density at 25 °C** g/cm^3
23 ± 1	50 ± 5	1,444 ± 0,005	1,32 ± 0,02
27 ± 1	65 ± 5	1,427 ± 0,007	1,26 ± 0,02

NOTE When, in special cases, an atmosphere of (20 ± 1) °C and (65 ± 5) % relative humidity is needed, the value of n_D^{25} is 1,426 ± 0,007 (method B) and the value of the sulfuric acid density is (1,26 ± 0,02) g/cm^3 (method C).

9 Precision and bias

The uncertainties in the relative humidities given in the literature for the methods described are theoretical values. Temperature equilibrium between the air in the enclosure and the aqueous solution and pressure equilibrium between the water vapour in the air in the enclosure and the water vapour pressure of the aqueous solutions are taken for granted. These theoretical uncertainties are generally smaller than 2 % RH. They do not consider e.g. the influence of variations in the temperature of the air and the influence of test specimens that absorb or release even small amounts of water.

The relative humidity in the enclosure is strongly influenced by any variation in air temperature. Even without any variation in the moisture content of the air, differences in the temperature of the air from one point in the enclosure to another will produce differences in the relative humidity. Variations in the temperature at a given point with time will tend to produce the same effect. The relative humidity tends to decrease when the temperature increases and to increase when the temperature decreases.

Therefore, in the equilibrium state, the maximum permitted variation in temperature (in position and in time) is 1 °C. The resulting variation in the relative humidity depends on the temperature and humidity ranges and is shown in Table 6 [2].

Table 6 — Variation in relative humidity from desired value at different temperatures resulting from a temperature variation of ± 1,0 °C

Relative humidity	Variation in relative humidity (%) at temperature θ			
%	5 °C	25 °C	50 °C	70 °C
30	2,1	1,8	1,5	1,3
50	3	3	3	2,2
70	5	5	4	3
90	6	5	4	4

10 Test report

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

a) a reference to this International Standard;

b) the conditioning and testing atmosphere specified (temperature and relative humidity);

c) the characteristics of the enclosure used (volume, air circulation system);

d) the solution used:

   1) for saturated aqueous salt solutions, identify the salt used (by giving the number in Table 1),

   2) for aqueous glycerol solutions, give the refractive index,

   3) for aqueous sulfuric acid solutions, give the density;

e) any details not given in this International Standard and any incidents that occurred which are liable to have influenced the results;

f) the duration and the date of the exposure.