INTERNATIONAL STANDARD

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Optics and photonics — Optical coatings —

Part 3: Environmental durability

Optique et photonique — Traitements optiques —

Partie 3: Durabilité environnementale

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Foreword

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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 9211-3 was prepared by Technical Committee ISO/TC 172, *Optics and photonics*, Subcommittee SC 3, *Optical materials and components*.

This second edition cancels and replaces the first edition (ISO 9211-3:1994) subclauses 3.1 to 3.5 of which have been technically revised or deleted and renumbered; Table 1 of which has been expanded and technically revised and Table 2 of which has been deleted and replaced by new informative Annex A.

ISO 9211 consists of the following parts, under the general title *Optics and photonics — Optical coatings*:

— *Part 1: Definitions*

— *Part 2: Optical properties*

— *Part 3: Environmental durability*

— *Part 4: Specific test methods*

Optics and photonics — Optical coatings —

Part 3: Environmental durability

1 Scope

ISO 9211 identifies surface treatments of components and substrates excluding ophthalmic optics (spectacles) by the application of optical coatings and gives a standard form for their specification. It defines the general characteristics and the test and measurement methods whenever necessary. It is not intended to define the process method.

This part of ISO 9211 specifies categories of use for optical coatings and identifies which environmental tests are necessary to prove that the coatings meet the required specification. Definitions and the extent of testing are given in ISO 9022-1.

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 9022-1, *Optics and optical instruments — Environmental test methods — Part 1: Definitions, extent of testing*

ISO 9022-2, *Optics and optical instruments — Environmental test methods — Part 2: Cold, heat and humidity*

ISO 9022-4, *Optics and optical instruments — Environmental test methods — Part 4: Salt mist*

ISO 9022-6, *Optics and optical instruments — Environmental test methods — Part 6: Dust*

ISO 9022-9:1994, *Optics and optical instruments — Environmental test methods — Part 9: Solar radiation*

ISO 9022-11, *Optics and optical instruments — Environmental test methods — Part 11: Mould growth*

ISO 9022-12, *Optics and optical instruments — Environmental test methods — Part 12: Contamination*

ISO 9022-14, *Optics and optical instruments — Environmental test methods — Part 14: Dew, hoarfrost, ice*

ISO 9211-4, *Optics and optical instruments — Optical coatings — Part 4: Specific test methods*

3 Categories of use

3.1 Definitions of categories

Five categories of use are defined. Each category requires either different environmental tests and/or different severity of testing. These categories are listed below in order of severity of requirement.

Category A

This category refers to components in applications which would normally only apply when they are to be mounted internally within sealed units. In this category, handling is in a protected and controlled environment and should only take place with extreme care. Physical contact with the optically coated surface is discouraged.

Category B

This category refers to applications where components will be exposed only to a controlled environment. Such applications may involve mild abrasion such as occurs with carefully controlled cleaning.

Category C

This category refers to applications where components will be exposed to normal outdoor ambient conditions and cleaning but without severe abrasion and scratching.

Category D

This category refers to applications where components will be exposed to severe outdoor ambient conditions and uncontrolled cleaning with the risk of severe abrasion and scratching.

Category O

This category refers to applications which require special, non standard, specifications. Since the specification of the components in such cases will not exactly fit into one of the categories A to D, the recommended way to specify in such a case is to indicate first the category in which most requirements are satisfied. The exceptional requirements can then be specified from other categories or by indicating the test degree of severity.

EXAMPLE "Category C; Abrasion, Humidity: Category B; Adhesion: 03".

3.2 Operating and storage conditions

The temperature specifications listed in Table 1 are meant as storage conditions. For some types of coating, e.g. bandpass filters and accurate edge filters, it may be necessary that spectral tolerances shall be maintained within a certain temperature range. This should be specified separately, according to the requirements of the application.

3.3 Influence of the substrate

It should be kept in mind that it is not the coating but the entire coating-substrate combination which determines the category of use.

For instance, coatings on glass, normally satisfying category C, might not do so when applied to sensitive or unstable substrates. This is likely to become apparent with the rain, solubility, humidity, heat, and salt spray tests, for example.

3.4 Cemented coatings

This part of ISO 9211 does not apply to cemented coatings. The environmental stability of such a substrate-coating-cement-substrate combination depends too much on properties of the cement, as well as the (relative) properties, e.g. thermal expansion, of the two substrate components involved.

4 Specifications

The mechanical and chemical properties of coated optical elements, and more generally their environmental durability, can be assessed by a variety of methods. Test methods selected to give meaningful results representative of actual exposure of optical elements in their operating environment are listed in Table 1. The practical severity of any test listed may be limited by the substrate.

The requirements for coatings as listed in Table 1 are typically not accumulative and can be tested individually.

The tests in Table 1 are subdivided into degrees of severity, where appropriate. The severity of the test requirement increases with the number. The description is condensed information about the test method only. The full test procedure shall be taken from appropriate International Standards, referenced in Table 1, or can be specified by mutual agreement between customer and manufacturer.

An individual test performed on a one-test-on-one-sample basis can give information about that single property of an optical coating reflected by that test and can be particularly useful for the manufacturer. In reality, optical coatings will face a variety and range of severity of environmental exposures, which can be simulated by certain test sequences. Inevitably, such test sequences represent accumulative requirements. Requirements without any listed categories in Table 1 are to be considered for optional use in category O, and are subject to agreement between customer and manufacturer.

Table 1 — Environmental tests for optical coatings

No.	Test	Degree of severity	Description	Categories of use					Reference[a]
				A	B	C	D	O	
1	Abrasion	01	50 strokes cheesecloth		x				ISO 9211-4
		02	100 strokes cheesecloth			x			
		03	20 strokes eraser				x		
		04	40 strokes eraser						
2	Adhesion	01	Slow tape removal		x				ISO 9211-4
		02	Quick tape removal			x	x		
		03	Snap tape removal						
		—	Crosshatch test						
3	Dust/sand	01 to 03	Expose to the dust-laden air of velocity 8 m/s to 10 m/s, at a temperature of 18 °C to 28 °C and of relative humidity below 25 % for 6 h. The dust concentration is 5 g/m^3 to 15 g/m^3, and the size distribution of dust particle is given by: Dust particle size distribution Size (µm) 140 to 100, 100 to 71, 71 to 45, < 45 % (by mass) 2 8 15 75 SiO_2 content > 97 %						ISO 9022-6

Table 1 (*continued*)

No.	Test	Degree of severity	Description	Categories of use					Reference[a]
				A	B	C	D	O	
4	Solubility	01	Immerse in distilled or deionized water at a temperature of 23 °C ± 2 °C for 6 h			x			ISO 9211-4
		02	Immerse in distilled or deionized water at a temperature of 23 °C ± 2 °C for 24 h				x		
		03	Immerse in distilled or deionized water at a temperature of 23 °C ± 2 °C for 96 h						
		04	Immerse in salt water (45 g NaCl/l) at a temperature of 23 °C ± 2 °C for 6 h						
		05	Immerse in salt water (45 g NaCl/l) at a temperature of 23 °C ± 2 °C for 24 h				x		
		06	Immerse in salt water (45 g NaCl/l) at a temperature of 23 °C ± 2 °C for 96 h						
		07	Immerse in boiling distilled or deionized water for 5 min			x			
		08	Immerse in boiling distilled or deionized water for 15 min				x		
		09	Immerse in boiling salt water (45 g NaCl/l) for 5 min				x		
		10	Immerse in boiling salt water (45 g NaCl/l) for 15 min						
		11	Immerse in boiling salt water (45 g NaCl/l) for 60 min						
		12	Immerse in boiling distilled or salt water (45 g NaCl/l) for 2 min. Then immerse in distilled water at room temperature for 1 min						
5	Damp heat	06	Expose to climatic conditions of 90 % to 95 % relative humidity and 55 °C ± 2 °C for 6 h		x				ISO 9022-2
		07	Expose to climatic conditions of 90 % to 95 % relative humidity and 55 °C ± 2 °C for 16 h			x			
		03	Expose to climatic conditions of 90 % to 95 % relative humidity and 40 °C ± 2 °C for 10 d				x		
6	Cold	05	Expose to a temperature of − 25 °C ± 3 °C for 16 h	x					ISO 9022-2
		07	Expose to a temperature of −35 °C ± 3 °C for 16 h		x	x			
		09	Expose to a temperature of −55 °C ± 3 °C for 16 h				x		
			(The temperature change rate should be less than 3 °C/min)						
7	Dry heat	03	Expose to an atmosphere of 55 °C ± 2 °C (below 40 % relative humidity) for 16 h	x					ISO 9022-2
		05	Expose to an atmosphere of 70 °C ± 2 °C (below 40 % relative humidity) for 6 h		x	x			
		06	Expose to an atmosphere of 85 °C ± 2 °C (below 40 % relative humidity) for 6 h				x		
			(The temperature change rate should be less than 5 °C/min)						
8	Slow temperature change	02	− 25 °C ± 3 °C to + 55 °C ± 2 °C		x				ISO 9022-2
		05	− 35 °C ± 3 °C to + 63 °C ± 2 °C			x			
		07	− 50 °C ± 3 °C to + 70 °C ± 2 °C				x		
			Test chamber temperature change rate: between 0,2 °C/min and 2 °C/min						

Table 1 (*continued*)

No.	Test	Degree of severity	Description	Categories of use					Reference[a]
				A	B	C	D	O	
9	Salt mist	—	Expose to salt spray fog of 35 °C ± 2 °C for 24 h Salt solution (concentration: 5 % ± 1 %, pH 6,5 to 7,2) is injected by compressed air ($0,4 \times 10^5$ Pa to $1,7 \times 10^5$ Pa) from 0,5 ml/h to 3,0 ml/h per 80 cm^2 in fallout rate				x		ISO 9022-4[b]
10	Solar radiation	01	Expose to the radiation in a de-ozonised atmosphere of 25 °C ± 2 °C to 55 °C ± 2 °C for 72 h Irradiance on the surface of sample is 1 $kW/m^2 \pm 0,1$ kW/m^2 The radiation source and spectral energy distribution shall be in accordance with Table 1 of ISO 9022-9:1994						ISO 9022-9
11	Icing/frosting	01 and 02	Expose to the following atmospheres in succession (conditioning method 77): Step 1 Initial temperature: − 15 °C ± 3 °C Step 2 Temperature of icing/frosting: − 5 °C ± 2 °C Step 3 De-icing temperature and humidity: 30 °C ± 2 °C and 80 % to 95 % relative humidity						ISO 9022-14
12	Chemical durability		Immerse in the following agents for respective tests (conditioning method 87):						
12-1	Acid corrosion	01 to 04	1) Sulphuric acid (H_2SO_4) 2) Nitric acid (HNO_3)						
12-2	Alkaline corrosion	01 to 04	1) Potassium hydroxide (KOH)						ISO 9022-12
12-3	Solvent solubility	01	1) Acetone (CH_3COCH_3)		x	x	x		
		01	2) Ethanol (C_2H_5OH)		x	x	x		
		02 to 04	1) Acetone (CH_3COCH_3)						
		02 to 04	2) Ethanol (C_2H_5OH)						
13	Mould growth[c]	01	Spray the spore suspension Number of spores in the suspension: 1 000 000/ml ± 200 000/ml Temperature and humidity conditions: 29 °C ± 1 °C, 96 % ± 2 % relative humidity Number of spores on test item surface: 15 000/cm^2 ± 3 000/cm^2 Test duration: 28 d Test fungi to be specified						ISO 9022-11
14	[d]								

[a] Referenced International Standards are normative test procedures, unless otherwise indicated.

[b] Exposure time is different from any degree of severity in ISO 9022-4.

[c] Mould growth (fungus): the resistance of a coating to damage by mould growth shall be specified, not the prevention of mould growth.

[d] Additional durability tests for special applications can be required, such as: rain impact/erosion, sand slurry abrasion, corrosive gases, fluids, etc.

Annex A
(informative)

Examples of test sequences for Categories of use A, B, C and D

Table A.1 — Example of a test sequence for Category of use A (one test sample is used)

Test step	No. (from Table 1)	Test	Degree of severity	Test sample 1
1	6	Cold	05	x
2	7	Dry heat	03	x

Table A.2 — Example of a test sequence for Category of use B (two test samples are used)

Test step	No. (from Table 1)	Test	Degree of severity	Test sample 1	Test sample 2
1	2	Adhesion	01	x	x
2	1	Abrasion	01	x	
3	6	Cold	07		x
4	7	Dry heat	05		x
5	5	Damp heat	06		x
6	8	Slow temperature change	02		x
7	12-3	Solvent solubility	01	x	
8	2	Adhesion	01	x	x

Table A.3 — Example of a test sequence for Category of use C (three test samples are used)

Test step	No. (from Table 1)	Test	Degree of severity	Test sample 1	Test sample 2	Test sample 3
1	2	Adhesion	02	x	x	x
2	1	Abrasion	02	x		
3	4	Solubility	01			x
4	6	Cold	07		x	
5	7	Dry heat	05		x	
6	5	Damp heat	07		x	
7	8	Slow temperature change	05		x	
8	12-3	Solvent solubility	01	x		
9	2	Adhesion	02	x	x	x

Table A.4 — Example of a test sequence for Category of use D (four test samples are used)

Test step	No. (from Table 1)	Test	Degree of severity	Test sample 1	Test sample 2	Test sample 3	Test sample 4
1	2	Adhesion	02	x	x	x	x
2	1	Abrasion	03	x			
3	4	Solubility	02			x	
4			05			x	
5			08			x	
6			09			x	
7	6	Cold	09		x		
8	7	Dry heat	06		x		
9	5	Damp heat	03		x		
10	8	Slow temperature change	07		x		
11	9	Salt mist	—				x
12	12-3	Solvent solubility	01	x			
13	2	Adhesion	02	x	x	x	x

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