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Specifications for use of poly(lactic acid) based filament in additive manufacturing applications

Spécifications pour l'utilisation de filaments à base de poly(acide lactique) dans les applications de fabrication additive

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Contents

Page

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Foreword

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This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 14, *Environmental aspects.*

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Introduction

Poly(lactic acid) (PLA) is a thermoplastic polymer material that is commonly used in additive manufacturing/3D Printing. This document specifically addresses the following:

a) specify various parameters such as the appearance, dimensions, chemical and physical properties;

b) this document is expected to improve the quality management of PLA based filament for additive manufacturing applications;

c) provide information on end-of-life management, use of renewable biobased carbon feedstocks vs. fossil based feedstocks, Carbon and Environmental Footprint, and methodology of a circular economy.

The document is expected to provide benefits to both consumers and industry. Consumers may profit from this document by relying on standardized specification. This document is also important for the development and growth of a new environmentally responsible polymer material industry based on PLA.

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Specifications for use of poly(lactic acid) based filament in additive manufacturing applications

1 Scope

This document specifies the technical requirements, test methods, detection rules, marking/labelling, packaging, transportation and storage of poly(lactic acid) (PLA) based filament for use in specific additive manufacturing technology, such as materials extrusion (MEX).

The document applies to PLA based filament for MEX additive manufacturing applications.

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2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 527-2, *Plastics — Determination of tensile properties — Part 2: Test conditions for moulding and extrusion plastics*

ISO 1133-1:2022, *Plastics — Determination of the melt mass-flow rate (MFR) and the melt volume-flow rate (MVR) of thermoplastics — Part 1: Standard method*

ISO 2859-1, *Sampling procedures for inspection by attributes — Part 1: Sampling schemes indexed by acceptance quality limit (AQL) for lot-by-lot inspection*

ISO 8124-3, *Safety of toys — Part 3: Migration of certain elements*

ISO 12219-2, *Interior air of road vehicles — Part 2: Screening method for the determination of the emissions of volatile organic compounds from vehicle interior parts and materials — Bag method*

ISO 15512:2019, *Plastics — Determination of water content*

ISO 16620 (all parts), *Plastics — Biobased content*

ISO 22526 (all parts), *Plastics — Carbon and environmental footprint of biobased plastics*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
additive manufacturing
AM
process of joining materials to make parts from 3D model data, usually layer upon layer, as opposed to subtractive manufacturing and formative manufacturing methodologies

Note 1 to entry: This is also commonly referred to as 3D-printing.

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[SOURCE: ISO/ASTM 52900:2021, 3.1.2, modified — Note 1 to entry replaced and Note 2 to entry removed.]

3.2
material extrusion
MEX
additive manufacturing (3.1) process, where an object is built by selectively depositing melted material in a pre-determined path layer-by-layer

Note 1 to entry: More specifically, MEX, refers to a process in which a filament material (i.e. PLA) is heated to melt in a printer and extruded through a nozzle (with certain bore diameter), that moves following a preset computer slice model to continuously deposit a layer on, and bond to a previous layer after solidification to form the final product.

3.3
melt mass-flow rate
MFR
rate of extrusion of a molten resin through a die of specified length and diameter under prescribed conditions of temperature, load and piston position in the cylinder of an extrusion plastometer

Note 1 to entry: The rate is determined as the mass extruded over a specified time.

Note 2 to entry: MFR can be expressed in milligrams per second (mg/s) or grams per 10 min (g/10 min).

3.4
total volatile organic compounds
TVOC
group of compounds that are present in emissions or ambient air, which include benzene, toluene, butyl acetate, ethylbenzene, *p*-(*m*-)xylene, styrene, *o*-xylene, undecane, etc.

Note 1 to entry: Indoor building and decoration materials are the main sources of TVOC in the air.

3.5
3D printing
3DP
type of rapid prototyping and digital model file-based technology using powdered metal or plastic and other adhesive materials to construct object layer-by-layer

4 Labelling

The product shall be labelled with the specifications: barcode, country of production, build temperature, name of material, 3D filament, colour and net mass as shown in Figure 1.

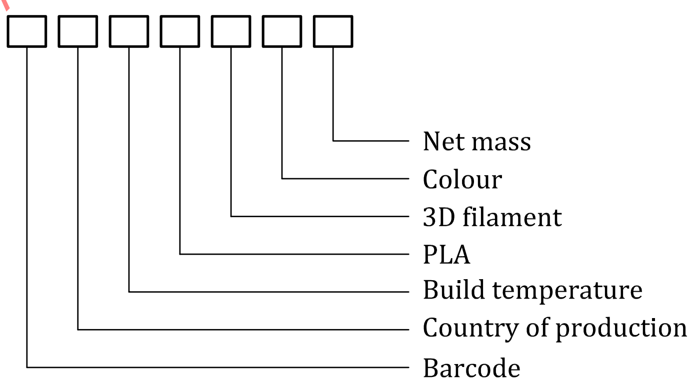

Figure 1 — Labelling of product

EXAMPLE A blue PLA filament material having diameter of 1,75 mm, with net mass of 1 kg shall be designated as 1,75 mm PLA 3D filament blue: 1 kg as shown in Figure 2.

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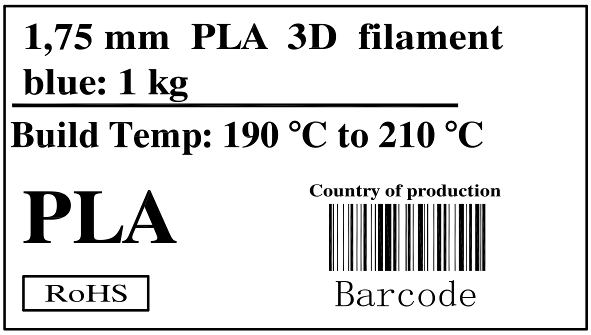

Figure 2 — Label example

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5 Requirements

5.1 Appearance

In general, the filament material shall be of uniform colour consistency, without burrs, protuberances, visible crease, scratches, or bubbles. However, filaments can also be multicolour or contain foreign matter such as foaming agents, fillers, metals powders and cellulose particles. The filament material shall be of uniform colour consistency if marked as a single colour filament. In the case of multicolour filament, two or three basic colours should be mentioned.

Appearance requirements shall be assessed in accordance with 6.2.

5.2 Specification

5.2.1 Diameter and ovality tolerance

Filament diameters of 1,75 mm and 2,85 mm are the most common (80 % to 90 %) of filament produced. Other diameter sizes can also occur. Filament diameter and filament ovality shall meet the requirements of Table 1, when measured according to 6.3.

Table 1 — Diameter and roundness

Nominal diameter (ND)	Tolerance		Ovality
	Premium product	Qualified product	
2,85 mm	±0,03 mm	±0,05 mm	≤0,07 mm
1,75 mm	±0,03 mm	±0,03 mm	≤0,05 mm
Other values	±1,7 %ND	±1,1 %ND	≤±2,6 %ND

NOTE Filament products are classified into premium products and qualified products based on nominal diameter tolerance and ovality tolerance.

5.2.2 Net mass and tolerance

The net mass of filament shall be 0,5 kg or 1,0 kg with tolerance of +3 % when measured according to 6.4. Spools can range from 0,75 kg to 6 kg.

5.2.3 Volatile matter limit

Follow the reporting requirements in ISO 12219-2.

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5.2.4 Biobased (carbon) content

Follow the reporting requirements in the ISO 16620 series.

5.2.5 Carbon and environmental footprint

Follow the reporting requirements in the ISO 22526 series.

5.3 Property requirements

Filament shall meet the property requirements in Table 2.

Table 2 — Properties of filament

Item		Requirements	Test method
Build temperature		(200 ± 20) °C	Device settings
Moisture content		≤0,5 %	6.6
Line tensile load	1,75 mm	≥125 N	6.7
	2,85 mm	≥340 N	6.7
	Other value	Negotiated by manufacture and user	
Line elongation at break	1,75 mm	≥10 %	6.7
	2,85 mm	≥6 %	6.7
	Other value	Negotiated by manufacture and user	

5.4 Limit quantity of substances and impurities

Appropriate limitations should be met according to, for example, ISO 8124, ISO 17088, etc.

6 Test methods

6.1 Conditioning

Conditioning of filament samples on the spool shall be done according to ISO 291 under condition of 23 °C ± 2 °C. The time of conditioning shall be at least 48 h, but no longer than 96 h.

6.2 Appearance of filament material

Take a whole roll of the sample, under the condition of sufficient light with the help of a magnifying glass, the eyes are (30~50) cm away from the sample to directly observe the colour difference, burr, protrusion, creases, foreign bodies, scratches, bubbles, etc. of the sample, and the observation time is (2 s~3 s). A colour chart shall be used.

6.3 Diameter and ovality of filament material

Use a micrometer or other non-contact detection tool such as an infrared calliper or a laser calliper with an error limit of 0,001 mm.

The filament diameter shall be measured by at least three different directions, at least 50° apart. All values shall attend the diameter tolerance.

The ovality is measured by measuring diameters of at least three different directions, at least 50° apart, and by subtracting the minimum value from the maximum value.

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The ovality result is used to determine whether the filament is cylindrical, and the basis for the determination shall meet the ovality requirements in Table 1.

6.4 Net mass and tolerance of filament material

This shall be measured by using a calibrated balance with maximum error limit no larger than 0,001 kg.

6.5 Melt mass-flow rate

Measure melt mass-flow rate method A of ISO 1133-1 under test condition D [temperature (190 ± 1) °C, load (2,16 ± 0,01) kg].

6.6 Moisture content

6.6.1 General

Moisture content shall be measured using one of the following methods given in 6.6.2 to 6.6.4.

6.6.2 Method A

Measure moisture in accordance with ISO 15512:2019, method B.

6.6.3 Method B

Take (10,0 ± 0,5) g of sample and spread it on the bottom of the weighing pan and place it in a halogen fast moisture meter whose temperature is raised to (105 ± 1) °C. Press the "Display" key to allow an 80 s of judgment before the moisture measurement is completed, when the instrument will automatically stop heating, and an alarm will sound to record the display data in the form of a % sign. Each sample was tested at least three times and the average value taken.

6.6.4 Method C

Take 1,0 g of sample (tolerance of ±0,1 g), put it in weighing bottle (w_2) that was dried to constant mass at (110 ± 1) °C and heat the bottle at (70 ± 1) °C for 4 h in a dryer before cooling to room temperature and weigh it (w_1). Moisture content shall be calculated using Formula (1).

$$\Delta w = \frac{w-(w_1-w_2)}{w} \times 100\,\% \tag{1}$$

where

Δw is the moisture content, expressed as a percentage (%);

w is the measured mass of sample, in gram (g);

w_1 is the mass of sample and weighing bottle after cooling to room temperature, in gram (g);

w_2 is the mass of weighing bottle, in gram (g).

In an arbitration experiment, the moisture test shall be carried out according to Method C.

6.7 Tensile load and elongation at break of filament

Take filament test sample directly with fixture spacing of (350 ± 5) mm for testing in accordance with ISO 527-2 at a tensile speed of (50 ± 2) mm/min. Five test samples shall be tested and the average is reported as result. The numerical deviation of the 5 samples should be no more than 20 % from the mean value.

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6.8 Impurities limit

Measurement of impurities content shall be performed according to ISO 8124-3.

6.9 Volatile matter limit

Measurement of formaldehyde, xylene, styrene and other TVOC shall be done in accordance with ISO 12219-2.

6.10 Biobased (carbon) content

Report on biobased (carbon) content using the ISO 16620 series.

6.11 Carbon and environmental footprint

Report on the carbon and environmental footprint of the filament material and/or the target product made by 3D printing/AM using the ISO 22526 series.

6.12 End-of-life options

Options for the filament waste could be industrial composting, mechanical recycling, or chemical depolymerization.

One should consider application appropriateness for end-of-life options. If products are being designed with industrial composting as end of life, they should comply separately to the appropriate compostability standard (e.g. ISO 17088).

6.13 Discharge ability

This test is optional (see Annex A)

7 Verification

7.1 General

Unless required for specific applications, for example, as absorbable material for implant manufacturing, material verification shall be done according to 7.2 to 7.3.

7.2 Sampling

7.2.1 General

Samples for testing shall be drawn randomly in accordance with ISO 2859-1, adopting single sampling plan for normal inspection, by general inspection level I and acceptance level 6,5 (see Table 3).

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Table 3 — Sampling scheme

Unit: roll

Lot size	Sample size	Acceptance number *Ac*	Rejection number *Re*
Up to 15	2	0	1
	2	-	-
16 to 25	3	-	-
	3	0	1
26 to 90	5	-	-
	5	-	-
91 to 150	8	1	2
	8	-	-
151 to 280	13	2	3
	13	1	2
281 to 500	20	3	4
	20	2	3
501 to 1 200	32	5	6
	32	3	4
1 201 to 3 200	50	7	8
	50	5	6
>3 200	80	10	11
	80	8	9

7.2.2 Determination of acceptability

The sample size for the first inspection should be equal to the first sample size given by the protocol. If the number of nonconforming items found in the first sample is less than or equal to the first acceptance number, the lot shall be considered acceptable; if the number of nonconforming items found in the first sample is greater than or equal to the first rejection number, the batch should be considered unacceptable. If the number of nonconforming items found in the first sample is between the first accepted and the first rejected, a second sample of the sample size given by the protocol shall be inspected and accumulated in the first and second samples the number of nonconforming items found. If the cumulative number of non-conforming products is less than one thousand or equal to the second acceptance number, the batch is determined to be acceptable; if the cumulative number of non-conforming products is greater than or equal to the second rejection number, the batch is determined to be unacceptable.

7.3 Type inspection

Type inspection shall include checking all the items specified in this document and shall be undertaken in case of one of the following:

a) trial production of new product and finalization of products;

b) when the raw material, formula and production line are changed, which may influence the properties of products;

c) in-cycle periodic inspection during normal production process or after a certain number of products have been accumulated (at least one time a year);

d) when production resumes after suspension of production for more than half a year;

e) when the delivery inspection is significantly different from the last type of inspection.

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8 Mark, packaging, transportation and stock

8.1 Mark

The external packaging shall have obvious marks including trademark, name and address of manufacturer, standard number, product name, specification, batch number and net content.

8.2 Packaging

Use sealed and moisture-proof packaging, to protect from incidental environmental contamination occurring during storage and transport. Handling conditions (from production to packaging, pre -and post-sampling) should be clean, cool and dry.

8.3 Transportation

The transport facility shall be clean and dry. High temperature (higher than 45 °C) shall be avoided. The products shall be lightly loaded and unloaded during transportation, avoiding damage to packaging, sun and rain; do not mix with strong alkali and strong acids, toxic, corrosive or inflammable materials.

8.4 Stock

The filament products shall be stored in a clean, cool (5 °C to 30 °C), dry (humidity: 25 % to 65 %) ventilated warehouse, away from incompatible materials. The products shall be kept away from heat source. Stacking the products in open air is prohibited. No direct compression of the product is allowed. If applicable and relevant, shelf life should be communicated to downstream users.

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Annex A
(informative)

Tester and testing procedure for discharge property test

A.1 Discharge ability tester

The equipment of the discharge tester used for testing volatile chemicals, continuous discharge and stable discharge shall meet the following requirements.

a) The PLA filament wound on the spool or bobbin is installed on a bearing type filament stand, and the PLA filament is fed to the nozzle by a filament feeder (feeder) equipped with a rated torque motor.

b) A feed rate measuring device (encoder) is installed between the PLA filament stand and the feeder, and a device for measuring the feed rate of the PLA filament by this encoder is provided.

c) When an external load is applied at the set feed rate, a device is provided to pass the load current to the rated torque motor in order to keep the torque constant.

d) By measuring the load current related to this rated torque motor, the pressure load related when the PLA filament is sent to the nozzle is evaluated. That is, when no external load is applied, a device for monitoring the load factor by defining the percentage of the load current with respect to the rated current flowing as the load factor is provided.

e) The nozzle used is equipped with a mechanism that can be easily changed.

f) A device for constantly monitoring the nozzle temperature with a temperature sensor or the like is provided.

For reference, a schematic diagram of the discharge tester is shown in Figure A.1.

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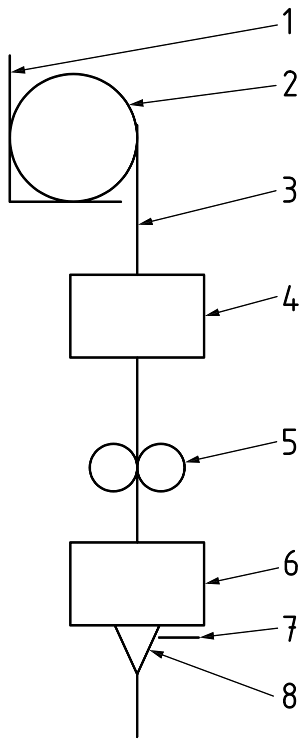

Key

1 PLA filament stand
2 spool
3 PLA filament
4 encoder
5 feeder
6 heater
7 temperature sensor
8 nozzle

Figure A.1 — Schematic diagram of the discharge tester

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A.2 Testing procedure of discharge property

a) Set the specimen with the individual packaging opened in the discharge tester specified in this annex installed in an environment with a temperature of 20 °C ± 15 °C and a relative humidity of 85 % or less on the PLA filament stand.

b) Attach a nozzle with a nozzle hole diameter of 0,4 µm to the heater.

c) Set the nozzle temperature to the recommended moulding temperature[1)] and the charging speed to 100 mm/min. If there is a range of recommended moulding temperatures, the median value is used, and if there is no recommended moulding temperature, the temperature is set to 200 °C.

d) Operate the device, when the nozzle temperature has reached the set temperature ±1 °C, perform a preliminary operation for 10 min.

e) After running for 30 min, run for another 30 min and record the load factor.

f) Read the maximum value $P_{(0\text{-}30)\max}$ and the minimum value $P_{(0\text{-}30)\min}$ of the load factor for 0 to 30 min, and the maximum value $P_{(30\text{-}60)\max}$ and the minimum value $P_{(30\text{-}60)\min}$ of the load factor for 30 min to 60 min.

g) Calculate the difference between the maximum value and the minimum value of the load factor[3)] for 0 min to 30 min $\Delta P_{(0\text{-}30)}$ using Formula (A.1), and the difference between the maximum value and the minimum value of the load factor[3)] for 30 min to 60 min $\Delta P_{(30\text{-}60)}$ using Formula (A.2).

1) The nozzle temperature recommended by the PLA filament manufacturer.

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