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Plastics — Polyethylene (PE) and polypropylene (PP) thermoplastics — Determination of metal content by ICP-OES

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC *61*, *Plastics*, Subcommittee SC 9, *Thermoplastic materials*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Metal elements in polyethylene (PE) and polypropylene (PP) can lead to increased ash content and can impact material mechanical properties. In addition, some elements are also environmentally hazardous. This document provides a method to determine the metal content in PE and PP and it can serve as a measure for additive monitoring, quality control and post-reactor studies.

In this document, inductively coupled plasma optical emission spectrometry (ICP-OES) is used to determine the concentrations of certain elements in PE and PP. The main advantages of ICP-OES over atomic absorption spectroscopy (AAS) include multi-element measurement capability, longer linear dynamic range and less condensed phase interferences.

Plastics — Polyethylene (PE) and polypropylene (PP) thermoplastics — Determination of metal content by ICP-OES

WARNING — Persons using this document should be familiar with normal laboratory practice, if applicable. This document does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices.

1 Scope

This document specifies a method for the determination of metal content in polyethylene (PE) and polypropylene (PP) by inductively coupled plasma optical emission spectrometry (ICP-OES).

This document is applicable to the determination of the content of magnesium (Mg), aluminium (Al), calcium (Ca), zinc (Zn), chromium (Cr), titanium (Ti), iron (Fe), antimony (Sb), copper (Cu), lead (Pb), cadmium (Cd), etc. This document is not applicable to the determination of mercury (Hg) due to its volatility.

This method is suitable for base polymers of PE, PP, PE/PP copolymer and their blends.

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 648, *Laboratory glassware — Single-volume pipettes*

ISO 1042, *Laboratory glassware — One-mark volumetric flasks*

ISO 3451-1, *Plastics — Determination of ash — Part 1: General methods*

3 Terms and definitions

No terms and definitions are listed in this document.

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

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

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

4 Principle

PE and PP samples are incinerated and digested. Metal elements within are dissolved in aqueous solution. The solution is converted into aerosol via a nebulizer, and then injected into argon supported ICP-OES. At high temperature, the plasma atomizes metal elements in the solution and gives characteristic optical emission. The intensity of emission is proportional to the concentration of element. The content of metal elements in the sample is then calculated by comparing the emission intensities of the sample with the calibration curve.

5 Reagents

Use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

5.1 Concentrated nitric acid, density 1,42 g/ml.

WARNING — Highly corrosive. Handle with suitable skin and eye protection in a fume hood.

5.2 Metal element standard solutions, 1 000 mg/l of Mg, Al, Ca, Zn, Cr, Ti, Fe, Sb, Cu, Pb and Cd for each individual standard solution, or multi-element standard solution containing 100 mg/l Mg, Al, Ca, Zn, Cr, Ti, Fe, Sb, Cu, Pb and Cd.

5.3 Cleaning agent, for example a volume fraction of 10 % of nitric acid solution.

6 Apparatus

Usual laboratory apparatus and glassware and, in particular, the following.

6.1 Inductively coupled plasma optical emission spectrometer, atomic emission wavelength measurement range: 160 nm to 760 nm.

6.2 Analytical balance, accurate to 0,1 mg.

6.3 Beaker, quartz, 250 ml.

6.4 Watch glass, quartz.

6.5 Hot plate, or other appropriate heat source. Working temperature up to (500 ± 25) °C.

6.6 Muffle furnace, capable of being maintained at (750 ± 50) °C.

6.7 Pipette, glass, of suitable capacity, ISO 648 class A.

6.8 Measuring cylinder, glass, 5 ml and 10 ml.

6.9 Volumetric flask, glass, 25 ml and 100 ml, ISO 1042 class A.

6.10 Fume hood, or other suitable means of ventilation.

6.11 Desiccator.

7 Sample preparation

WARNING — It is necessary that sample preparation procedure shall be carried out in a well-ventilated fume hood.

7.1 Cleaning glassware

Clean all glassware using cleaning agent (5.3).

7.2 Preparation of working standard solutions

7.2.1 Prepare a volume fraction of 3 % to 5 % of nitric acid solution from concentrated nitric acid (5.1) used as blank solution.

7.2.2 Prepare a series of five (5) working standard solutions from metal element standard solutions (5.2). The final concentrations of each element of Mg, Al, Ca, Zn, Cr , Ti, Fe, Sb, Cu, Pb and Cd in the working standard solutions are 0,50 mg/l, 1,00 mg/l, 5,00 mg/l, 10,00 mg/l and 20,00 mg/l. Concentrated nitric acid (5.1) shall be added during preparation, so that the volume fraction of nitric acid in the working standard solutions are 3 % to 5 %.

NOTE Commercially available standard solution can be used as well.

7.3 Preparation of test solution

7.3.1 Weigh 25 g (accurate to 0,1 mg) of the test sample into a beaker (6.3). The amount of test sample shall not reach the half volume of the beaker.

NOTE Adjustment of sample mass can apply according to the metal content.

7.3.2 Heat the beaker with sample on a hot plate (6.5) or other suitable heating devices. Adjust the heating power so that the sample is burnt slowly until the sample is well charred.

7.3.3 Remove the beaker from its heating device and cover with a watch glass (6.4). Introduce the beaker (6.3) into a muffle furnace (6.6) pre-heated to (750 ± 50) °C, which is chosen from ISO 3451-1. Calcine the sample for 3 h.

NOTE Depending on the content of metal elements in the sample, alternative methods of sample digestion such as pressure, microwave or wet acid digestion can be taken into account, too.

7.3.4 Remove the beaker (6.3) with the watch glass (6.4) from the muffle furnace (6.6) and allow it to cool in the desiccator (6.11).

7.3.5 Gently add 5 ml of concentrated nitric acid (5.1) and 5 ml of water to the beaker (6.3) and heat it on the hot plate (6.5) to dissolve the inorganics. When the solution is condensed to about 1,00 ml, take the beaker off the plate and cool it to room temperature.

7.3.6 Transfer the solution to a 25 ml volumetric flask (6.9), wash the residue twice by water and top up to final volume with water.

NOTE Turbid solution can be left to settle overnight, or centrifuged to get clear supernatant.

8 Procedure

8.1 Instrument set up

Turn on the ICP-OES (6.1) and warm up to readiness. Set up the ICP-OES parameters and detection wavelength for each element. Recommended wavelengths see Table 1, and other recommended instrumental parameters of the ICP-OES see Table 2.

8.2 Preparation of calibration curve

Introduce the working standard solutions (7.2) into the ICP-OES, record the emission line intensity of the blank solution and for a series of 0,50 mg/l, 1,00 mg/l, 5,00 mg/l, 10,00 mg/l and 20,00 mg/l working standard solutions in the ICP-OES. Plot the emission line intensity against the concentration