SLOVENSKI STANDARD
oSIST prEN ISO 787-28:2020

01-junij-2020

Splošne metode preskušanja pigmentov in polnil - 28. del: Določevanje celotnih polikloriranih bifenilov (PCB) z raztapljanjem, čiščenjem in plinsko kromatografijo z masno selektivnim detektorjem (GC-MS) (ISO 787-28:2019)

General methods of tests for pigments and extenders - Part 28: Determination of total content of polychlorinated biphenyls (PCB) by dissolution, cleanup and GC-MS (ISO 787 -28:2019)

Allgemeine Prüfverfahren für Pigmente und Füllstoffe - Teil 28: Bestimmung des Gesamtgehalts an polychlorierten Biphenylen in organischen Pigmenten durch Auflösung, Reinigung und GC/MS (ISO 787-28:2019)

Méthodes générales d'essai des pigments et matières de charge - Partie 28: Détermination de la teneur totale en biphényles polychlorés dans les pigments organiques par dissolution, purification et CG-SM (ISO 787-28:2019)

Ta slovenski standard je istoveten z: **prEN ISO 787-28**

ICS:

87.060.10	Pigmenti in polnila	Pigments and extenders

oSIST prEN ISO 787-28:2020 **en,fr,de**

INTERNATIONAL STANDARD

ISO 787-28

First edition
2019-05

General methods of tests for pigments and extenders —

Part 28: Determination of total content of polychlorinated biphenyls (PCB) by dissolution, cleanup and GC-MS

Méthodes générales d'essai des pigments et matières de charge —

Partie 28: Détermination de la teneur totale en biphényles polychlorés dans les pigments organiques par dissolution, purification et CG-SM

Reference number
ISO 787-28:2019(E)

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Published in Switzerland

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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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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 256, *Pigments, dyestuffs and extenders*.

A list of all parts in the ISO 787 series can be found on the ISO website.

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 https://www.iso.org/members.html.

Introduction

A number of methods to quantify PCBs in “environmental samples” or oil residues prove inadequate for pigments due to being merely extractive on the particle surface without taking into account occlusions of contaminants in the crystal lattice of pigments (see References [1] to [3]).

Occurrence and formation principles are referred to in References [5], [6] and [8].

General methods of tests for pigments and extenders —

Part 28: Determination of total content of polychlorinated biphenyls (PCB) by dissolution, cleanup and GC-MS

1 Scope

This document specifies a method for determining the total content of polychlorinated biphenyls (PCBs), checking for all 209 possible congeners in pigment materials.

This document is applicable to a working range from1 mg/kg to 150 mg/kg. The lower quantitation limit of this method is 1 mg/kg per congener. Results below 1 mg/kg are considered to be qualitative only.

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 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

ISO 18451-1, *Pigments, dyestuffs and extenders — Terminology — Part 1: General terms*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18451-1 and the following apply.

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/

3.1
polychlorinated biphenyls
PCB
209 congeners, from mono- through deca-chlorinated biphenyls, which may be subdivided into homologue groups comprising PCB congeners with the same degree of chlorination, i.e. same gross formula

Note 1 to entry: The general CAS-Number for polychlorinated biphenyls is 1336-36-3. For a comprehensive congener list with CAS-Numbers, see Annex A or Reference [5].

3.2
internal reference material
mixture of defined quantities of ^{13}C-Isotope-labelled PCBs added directly into the freshly weighed pigment sample

Note 1 to entry: No subsequent additions of internal reference materials are permitted. Surrogate standards reference materials can be added to assess recovery rates only, but these cannot be deemed an internal reference materials, nor can these be used for quantitation.

Note 2 to entry: Mass spectrometry assumes a vast linear range of detector sensitivity, thus a calibration with a single quantity will usually suffice.

Note 3 to entry: In case of major deviations between analytes and reference material concentrations, the quantitation might need to be confirmed by a second analysis (from the beginning) using an adequately adapted sample weight.

4 Principle

The principles have been developed from References [1] to [4], [6] and [7].

The use of solid surface extraction methods risks underestimating the actual content by orders of magnitude. Such errors are often further enhanced when the PCBs quantitation is carried out by using external or internal reference materials which do not undergo the same losses as the analytes that are occluded in the crystal lattice of the pigment.

5 Sampling

Take a representative sample of the product to be tested, as described in ISO 15528.

Samples shall be of dry powder consistency. Volatiles should be less than 1 % (mass fraction), determined with a sample portion separated from the sample portions used for PCB analyses.

Samples shall be kept in the dark and in capped glass bottles or vials.

Samples suspected of having high impurities content shall be handled in dedicated glassware and kept separately from other laboratory equipment; sample mass may be reduced and internal reference materials amounts doubled in order to cope with the possible elevated levels of interferences by impurities. A screening pre-run is recommended to avoid detector overload.

Bromine detection or presence of partially brominated samples (e.g. C.I. Pigment Red 168, C.I. Pigment Green 36) require caution due to occurrence of "mixed" halogenated aromatic compounds. Analysis can proceed if results are checked for non-interference (see 9.5).

6 Procedure

6.1 General

This method requires a strict sequential application of the following steps:

— sample weighing;

— addition of internal reference material and thorough mixing;

— dissolution in concentrated sulfuric acid from 92 % (mass fraction) to 95 % (mass fraction);

— sonicate the mixture until fully dissolved and a homogenous dark solution is obtained in the sulfuric acid phase. The temperature shall be kept lower than 50 °C at all times, e.g. by circulating or continuously exchanging the water in the sonication bath.

NOTE 1 Using this concentration range avoids major disintegration and rearrangement of mono- and bichlorinated biphenyls.

NOTE 2 Sufficient dissolution is indicated by absence of the "tyndall-effect" (i.e. no scattering of a preferably blue light beam by the solution).

Upon dissolution in concentrated sulphuric acid, pigment molecules are protonated and may be considered in a simplified way as being pulled out of their crystal lattice one by one in drawer-like fashion, until all the solid matter has dissolved, without cleavage occurring at the molecular (chromophor) level.

