SLOVENSKI STANDARD
oSIST prEN 6042:2021

01-september-2021

Aeronavtika - Organske spojine - Preskusna metoda - Analiza z infrardečo spektroskopijo

Aerospace series - Organic compounds - Test method - Analysis by infrared spectroscopy

Luft- und Raumfahrt - Organische Verbindungen - Prüfverfahren - Analyse durch Infrarot-Spektroskopie

Série aérospatiale - Composés organiques - Méthode d'essai - Analyse par spectroscopie infra-rouge

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49.025.40 Guma in polimerni materiali Rubber and plastics

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Aerospace series - Organic compounds - Test method - Analysis by infrared spectroscopy

Série aérospatiale - Composés organiques - Méthode d'essai - Analyse par spectroscopie infra-rouge

Luft- und Raumfahrt - Organische Verbindungen - Prüfverfahren - Analyse durch lnfrarot-Spektroskopie

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Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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European foreword

This document (prEN 6042:2021) has been prepared by the Aerospace and Defence Industries Association of Europe — Standardization (ASD-STAN).

After enquiries and votes carried out in accordance with the rules of this Association, this document has received the approval of the National Associations and the Official Services of the member countries of ASD-STAN, prior to its presentation to CEN.

This document is currently submitted to the CEN Enquiry.

1 Scope

This test method describes the principles applicable to infrared transmission spectrophotometric analysis of organic compounds (elastomers, basic resins, resin mixes or resin systems) used as the matrix in reinforced polymers, adhesives, bonding primers and, in general terms, all organic compounds.

The method could also be applied to some inorganic products.

It is intended to be used jointly with special test conditions specified in the materials specification invoking the test.

This document does not give any directions necessary to meet the health and safety requirements. It is the responsibility of the user of this document to adopt appropriate health and safety precautions.

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.

EN 2743, *Aerospace series - Fibre reinforced plastics - Standard procedures for conditioning prior to testing unaged materials*

3 Terms and definitions

For the purposes of this document, the following terms and definitions 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 https://www.electropedia.org/

3.1
base resin
main component of a resin system

3.2
resin mix
resin system
neat resin
resin
base resin + fillers + additive + catalytic systems + hardener + accelerator + thinner

3.3
prepreg resin
resin obtained from the prepreg

4 Principle of the method

4.1 General

Organic molecules consist of atoms bonded together. Many bonds vibrate at a characteristic frequency in the infrared (IR) range.

If a monochromatic IR beam impinges on the molecule and its frequency corresponds to a natural vibration frequency between functional groups, energy from the beam is absorbed.

Varying the wavelength of the beam therefore generates a series of absorption lines corresponding to the various molecular bonds. This set of lines forms a spectrum. The sample is subjected to a beam at all frequencies of interest and a computer determines which wavelengths have been absorbed. The preferred method is Fourier Transform Infrared (FTIR).

The IR absorption spectrum is reproducible and not greatly affected by the apparatus. It can be used to:

- identify the main organic functional groups of the molecule (carbonyl, ether, amine, epoxy, etc.),
- identify a material by comparison with reference spectra.

4.2 The Beer-Lambert law (Method of tangents)

For any absorption line in the JR spectrum (see Figure 1), a line can be drawn tangential to the transmission maxima on either side of the band. The absorption Beer's law is then written:

$$A = log\frac{I_o}{I} = \varepsilon \times C \times L$$

where:

A is the absorbance,

I is the transmittance at the maximum absorption within the ban (see Figure 1),

I_0 is the transmittance read from the tangent at the maximum absorption wavelength (see Figure 1),

ε is the factor of absorption (characteristic of the bond generating the absorption),

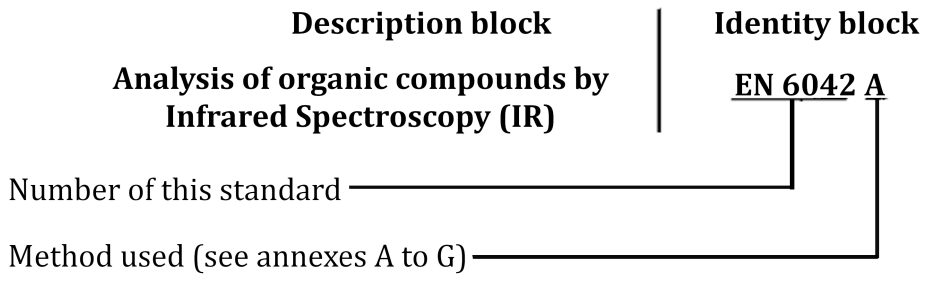
C is the concentration,

L is the length of the optical path in the sample.

For a given absorption band, ε and L are constant and absorbance A is therefore directly proportional to concentration C.

5 Designation of the method

The designation of the method used shall be drawn up according to the following example:



6 Apparatus

6.1 Spectrophotometer

6.1.1 General

Two types of spectrophotometer are used covering the range 400 cm^{-1} to 4 000 cm^{-1} (2,5 µm to 25 µm). These instruments give the same type of spectrum.

6.1.2 Wavelength dispersion spectrophotometer

An infrared polychromatic source generates a beam which is partially absorbed in the sample and then enter a monochromator. The output from the monochromator is a monochromatic beam with wavelength λ.

A detector measures the intensity of this beam and transmits the result to a recorder. The monochromator scans the wavelength and the recorder thus produces the sample IR absorption spectrum. The spectral resolution at 3 000 cm^{-1} shall be better than 5 cm^{-1} and at 1 000 cm^{-1} better than 3 cm^{-1}.

6.1.3 Fourier Transform Infrared spectrophotometer (FTIR)

The Fourier transform is a basic mathematical operation which converts a time periodic function into a frequency function.

In the FTIR spectrophotometer, the optical dispersion system is replaced by an interferometer. The absorption spectrum $A = f(\lambda)$ is the Fourier transform of the interference diagram obtained; this operation is performed by a computer connected to the spectrophotometer. FTIR spectroscopy offers the following advantages:

- faster,

- better resolution (1-2) cm^{-1},
- more sensitive since the energy loss is lower and the detectors used are more sensitive,
- more suitable for sensitive materials that change with time.

6.1.4 Calibration

The wavelength and absorption shall be calibrated with the standards recommended by the instrument supplier (e.g. polystyrene film).

6.2 Sampling method

Sampling procedures together with method specific apparatus and reagents are defined in Annexes A to G.

7 Test specimen

7.1 Preparation

In general terms, the sample analyzed shall be representative of the entire substance, i.e. the quantity of each component it contains shall be reproducible.

With reinforced products, it is usually necessary to eliminate the support (fibres or fabric), any mineral fillers and solvents using an appropriate process (extraction of the solvent, evaporation, centrifuging, etc.) to isolate the resin system.

Subsequently, take all precautions to obtain a homogeneous sample from a mixture of compounds, some of which tend to segregate. A resin system can contain some insolubles, some partially solubles and some entirely solubles.

7.2 Storage

The sample for analysis shall be stored under conditions such that it does not change between sampling and analysis or between two analyses.

8 Procedure

8.1 General

The test shall be carried out at (23 ± 2) °C and (50 ± 5) % relative humidity (EN 2743 B conditions).

Several infrared spectrophotometry methods can be used. The differences lie in the preparation and processing of the sample or the type of result expected.

The Annexes describe special features of each method.

8.2 Pelletization

This method is applicable to solid substances that are insoluble or difficult to dissolve, such as elastomers and cured materials.

It is described in Annex A.

It offers qualitative and, possibly, semi-quantitative results.

8.3 Deposit on a plate

This is a method frequently used for pasty, viscous organic substances. The deposit may be heterogeneous and difficult to reproduce.

The method is described in Annex B.

It will only give qualitative results.

8.4 Deposit between two plates

This is a method frequently used for pasty and viscous organic substances. The deposit takes the form of a homogeneous, reproducible thin film.

The method is described in Annex C.

It provides qualitative and semi-quantitative results.

8.5 Dissolution and pelletization

This method can be applied to solid or pasty substances even if not totally soluble.

It is described in Annex D.

It provides qualitative and semi-quantitative results.

8.6 Total dissolution

This method can be used to determine the proportion of a component in a resin system by comparing the absorbance with that obtained by previous calibration using a solution of known concentration.

The method is described in Annex E.

It provides qualitative, semi-quantitative and quantitative results.

8.7 Liquid cell method

This method is applicable to liquid or dissolved substances.

It is described in Annex F.

It provides qualitative, semi-quantitative and quantitative results.

8.8 Gas cell method

This method is applicable to gaseous substances.

It is described in Annex G.

It provides qualitative results and can be used to determine the proportion of the various components in the gaseous mixture by comparing the absorbance with that obtained by previous calibration using mixture of known concentration (quantitative results).

9 Analysis and interpretation of spectra

9.1 Introduction

Figure 2 gives an example of a typical spectrum.

The results can be interpreted in several ways, depending on how the sample analyzed was prepared.

9.2 Qualitative interpretation

This method is used to check, within the limits imposed by the method, the product by comparison with a reference spectrum given in the product specification or held on computer. The features checked are the appearance, disappearance or changes to the absorption bands. It is also possible to identify characteristic bands (functional groups) and thus usually identify, the major components.

9.3 Semi-quantitative interpretation

9.3.1 General

Semi-quantitative interpretation involves measuring the intensity of the bands and then calculating the infrared indices.

9.3.2 Calculation of the infrared index

The infrared index expresses the concentration of one function relative to another. The index is determined from the absorbance ratio.

- Let E be the absorption band to be determined quantitatively.
- A reference band R in the infrared spectrum is determined.
  - This band shall be invariable: the bond shall be non-reactive and its intensity shall be independent of changes to the system.
  - This band shall possess an absorbance of the same magnitude that of band E.

The infrared index i_E corresponding to band E is then calculated as follows:

- Calculate the absorbance $A_E = \frac{I_{oE}}{I_E}$ (see Figure 1).