



Products and systems for the protection and repair of concrete structures — Test methods — Infrared analysis

The European Standard EN 1767:1999 has the status of a
British Standard

ICS 91.010.30; 91.080.40

National foreword

This British Standard is the English language version of EN 1767:1999.

The UK participation in its preparation was entrusted by Technical Committee B/517, Concrete, to Subcommittee B/517/8, Repair and protection of concrete, which has the responsibility to:

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- present to the responsible European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
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Summary of pages

This document comprises a front cover, an inside front cover, the EN title page, pages 2 to 4, an inside back cover and a back cover.

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English version

Products and systems for the protection and repair of concrete structures — Test methods — Infrared analysis

Produits et systèmes pour la protection et la réparation des structures en béton — Méthodes d'essais — Analyse par spectrométrie infrarouge

Produkte und Systeme für den Schutz und die Instandsetzung von Betontragwerken — Prüfverfahren — Infrarotanalyse

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart 36, B-1050 Brussels

Foreword

This European Standard has been prepared by Technical Committee CEN/TC 104, Concrete (performance, production, placing and compliance criteria), the Secretariat of which is held by DIN.

This European Standard describes a test method. Specifications for the products and systems for the repair and the protection of concrete structures will be subject of separate standards.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by December 1999, and conflicting national standards shall be withdrawn at the latest by December 1999.

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1 Scope

This European Standard describes a method to record an infrared spectrum. It applies to products based on synthetic resins before their application and more particularly, in the case of two-component products, to each component separately before mixing. It also applies to emulsions of polymers (latex) that are to be mixed with hydraulic binders.

2 Normative reference

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

EN 21512, *Paints and varnishes — Sampling of products in liquid or paste form.*
(ISO 1512:1991)

3 Principle

3.1 General principle of infrared spectroscopy

Organic molecules can enter into vibration at definite frequencies situated in the range of the infrared. If infrared radiation encounters the molecule and the frequency of the radiation matches a frequency of vibration of the molecule, some of the energy of the radiation is absorbed.

Varying the wavelength of the infrared radiation reveals a series of absorption bands. The set of absorption bands constitutes an infrared spectrum. This spectrum is characteristic for the chemical nature of a particular compound.

3.2 Application to mixed products

The test consists of recording the infrared spectrum of the polymer binder of the product.

When the product is an emulsion or contains solvents, pigment and filler, these constituents, which would affect the analysis of the spectrum, shall first be removed.

The test result is an infrared spectrum of the polymer binder. Details of procedure making it possible to reproduce the test under the same conditions, shall be reported.

4 Apparatus

- *Infrared spectrophotometer.* The reference spectral range shall be at least $4\,000\text{ cm}^{-1}$ – 625 cm^{-1} .
- *Accessories of infrared spectrometry:* chamber of fixed or variable thickness, NaCl, CsI, or KRS-5 windows, pelletizing press. Accessory for analysis by attenuated total reflection (ATR).

- *Glass crystallizers,* 70 mm in diameter and 40 mm high.

- *Heating plate,* adjustable to $(60 \pm 2)^\circ\text{C}$.

- *Desiccator.*

- *Vacuum oven,* adjustable to a temperature of $(50 \pm 2)^\circ\text{C}$ and a pressure of 16 mbar to 33 mbar.

NOTE This vacuum is provided by a water-jet pump.

- *Oven,* adjustable to $(105 \pm 2)^\circ\text{C}$.

- *Centrifuge.*

- *Agitators, glassware, etc.*

- *Device to homogenize the sample* (electric agitator, etc.).

5 Reagents

- *Organic solvents.*

- *Potassium bromide powder* (spectroscopic grade).

6 Sampling

A representative and homogeneous sample of the product to be tested shall be taken according to EN 21512.

7 Procedure

7.1 Measurement of products without solvent or filler

The infrared spectrum is determined directly.

The choice of technique for producing the spectrum (spreading on slide, capillary film between two slides, pelletizing at 1 % in KBr) depends on the consistency of the sample.

7.2 Measurement of products in solution or emulsion without a mineral filler

When possible first of all an IR-spectrum of the original substance is recorded.

Evaporate the solvent or the water as given below:

- place approximately 1 g of product into a crystallizer with a small glass agitator, then place the crystallizer on a heating plate until the solvent or water is removed;
- after evaporation of the solvent or water, break the skin that may have formed and put the crystallizer into a vacuum oven for one hour;
- let the crystallizer cool to ambient temperature in a desiccator.
- Record the infrared spectrum of the dry extract so produced.
- Check, that the solvent has really evaporated entirely by using suitable IR-bands.

If evaporating the solvent is difficult, because of the consistency of the mixture, the process can be accelerated by adding about 25 ml of an organic solvent in the crystallizer. Thoroughly homogenize the mixture using the agitator, put the crystallizer back on the heating plate, and proceed as above.

The dry extract produced may not be homogeneous. To obtain a representative infrared spectrum, evaporate the product on a slide that is transparent in the infrared by placing the slide on a heating plate. In the case of an aqueous solution or emulsion, a water-resistant KRS-5 slide should be used. Using an ATR-crystal the sample can be dried with a fan.

Products which contain volatile matter (e.g. silanes) other than solvents will produce an IR-spectrum of the original substance only.

7.3 Measurement of products in solution or emulsion with mineral filler and product containing only a mineral filler

Before recording the infrared spectrum, eliminate the filler as follows

- place approximately 5 g of product into a centrifuge tube; add about 30 ml of an organic solvent and mix thoroughly using a glass agitator;
- centrifuge; collect the supernatant solution in a crystallizer;
- treat the centrifuging residue with another 30 ml of organic solvent, then mix thoroughly and centrifuge; add the supernatant solution to the first solution being evaporated;
- wash the residue once again and add the washing solution to the crystallizer then proceed as in 7.2.

Then record the infrared spectrum of the dry extract produced. It should be taken care, that the solvent is removed entirely by using suitable absorption bands.

8 Test report

The test report shall at least include the following information:

- a) a reference to this European Standard;
- b) all details necessary to identify the product tested;
- c) the type of preparation of the sample;
- d) the infrared spectrum with details of the execution of the spectrum;
- e) the solvent used for extraction of the polymer binder;
- f) any deviation, by agreement or otherwise, from the procedure described;
- g) the date of the test.

Annex A (informative)

Bibliography

- [1] DIN 51 451, 1986, Prüfung von Mineralölerzeugnissen und verwandten Produkten — Infrarotspektrometrische Analyse — Allgemeine Arbeitsgrundlagen.
- [2] NF T 51-500, 1989, Plastiques — Résines thermodurcissables — Analyse par spectrophotométrie infrarouge.

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