

**Oförstörande provning – Röntgendiffraktions-
mätningar på polykristallina och amorfa material –
Del 3: Instrument**

**Non destructive testing – X-ray diffraction from
polycrystalline and amorphous material –
Part 3: Instruments**

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Appareillage

Zerstörungsfreie Prüfung - Röntgendiffraktometrie von
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Foreword

This document (EN 13925-3:2005) has been prepared by Technical Committee CEN/TC 138 “Non destructive testing”, the secretariat of which is held by AFNOR.

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of an identical text or by endorsement, at the latest by November 2005, and conflicting national standards shall be withdrawn at the latest by November 2005.

This European Standard about “Non destructive testing - X-ray diffraction from polycrystalline and amorphous material” is composed of:

- prEN 1330-11, Terminology - Part 11: X-Ray Diffraction from Polycrystalline and Amorphous Materials
- EN 13925-1, Part 1: General principles
- EN 13925-2, Part 2: Procedures
- EN 13925-3 Part 3: Instruments
- WI 00138070, Reference Materials

In order to explain the relationship between the topics described in the different standards, a diagram illustrating typical operation involved in XRPD is given in Annex A.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

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Introduction

X-ray powder diffraction (XRPD) is a powerful Non-Destructive Testing (NDT) method for determining a range of physical and chemical characteristics of materials. These include the type and quantities of phases present, the crystallographic unit cell and structure, crystallographic texture, macrostress, crystallite size and microstrain, and the electron radial distribution function.

This document aims to describe the general aspects of the XRPD technique and its applications but not to define a specific or detailed standard for each field of application or type of analysis.

The main purposes of the standard are therefore to provide:

- practical guidance, unified concepts and terminology for use of the XRPD technique in the area of Non-Destructive Testing with general information about its capabilities and limitations of relevance to laboratories working at different levels of sophistication, from routine testing to research;
- a basis for Quality Assurance in XRPD laboratories allowing performance testing and monitoring of instruments as well as the comparison of results from different instruments;
- a general basis (without imposing specifications) for further specific NDT product standards and related Quality Assurance applications, with aspects common to most fields of application.

In order to make the standard immediately usable in a wide range of laboratories and applications, diffractometers with Bragg-Brentano geometry are considered in more detail than other instruments.

Radiation Protection: Exposure of any part of the human body to X-rays can be injurious to health. It is therefore essential that whenever X-ray equipment is used, adequate precautions should be taken to protect the operator and any other person in the vicinity. Recommended practice for radiation protection as well as limits for the levels of X-radiation exposure are established by national legislation in each country. If there are no official regulations or recommendations in a country, the latest recommendations of the International Commission on Radiological Protection should be applied.

1 Scope

This document sets out the characteristics of instruments used for X-ray powder diffraction ("powder" as defined in EN 13925-1:2003, Clause 5) as a basis for their control and hence quality assurance of the measurements made by this technique. Performance testing indicators are given for diffractometer performance testing. Different types and makes of X-ray powder diffractometer vary considerably in their design and intended fields of application. This document attempts to cover as much of this range as possible by keeping to common principles. To make the standard more readily applicable, the Bragg-Brentano configuration is addressed in most detail because of its wide use. Additional considerations and adaptations may be necessary to cover some types of instruments or configuration and some fields of application. Some of these types of instrument are described in Annex B.

2 Normative references

The following referenced documents are indispensable for the application 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 13925-2:2003, *Non-destructive testing – X-ray diffraction from polycrystalline and amorphous materials – Part 2: Procedures*

prEN 1330-11:2004, *Non-destructive testing – Terminology – Part 11: X-ray Diffraction from Polycrystalline and Amorphous Materials*

3 Terms and definitions

For the purposes of this document, the terms and definitions of prEN 1330-11:2004 apply.

4 Description of equipment

4.1 General

This description is particularly intended for instruments dedicated to the fields of application described in EN 13925-1. For other applications, additional considerations may be required.

A diffractometer generally comprises:

- goniometer;
- X-ray source;
- incident beam optics which may include monochromatisation or filtering, collimation and/or focusing or parallelism of the beam;
- diffracted beam optics which may include monochromatisation or filtering, collimation and/or focusing or parallelism of the beam;
- specimen stage;
- detector;
- data collection system.

These parts of the instrument are considered in more detail below. A data processing system is also required to produce measurements from the instrument. Data processing systems, whether manual or computerised, shall be included with the data processing procedures of EN 13925-2.

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A well-controlled environment (temperature and pressure) is strongly recommended for analysis where reproducible measurement of line profile position, width and shape is required.

Humidity is may be important because compounds in the specimen may react with water or absorb it with a consequent change in their lattice constants, e.g. clay minerals.

The X-ray beam is partially scattered and attenuated by the air in the beam path with consequential effects on the detected diffraction pattern background and intensity. This effect has sometimes been minimised by use of an evacuated or helium filled beam path.

For all the items described in this clause the corresponding main characteristics to be controlled are given in Clause 5.

4.2 X-ray sources

4.2.1 General

There are several types of x-ray source that can be used for XRPD measurements ranging from conventional laboratory sources to intense and well-collimated synchrotron sources. Each source exhibits characteristics that make it more suitable for particular types of analysis. The main source types are described below.

4.2.2 Conventional X-ray sources (sealed tubes and rotating anode sources)

X-rays are obtained by bombarding a metal anode with electrons emitted by the thermoionic effect and accelerated in a strong electric field produced by a high-voltage generator. Most of the kinetic energy of the electrons is converted to heat, which limits the power of the tubes and requires efficient anode cooling. An increase of about two orders of magnitude in brilliance can be obtained using rotating anodes instead of sealed tubes. Microfocus sources operate at relatively low power settings but maintain brightness by electrostatically or magnetically steering the beam inside the X-ray tube onto the target. The spectrum emitted by a conventional X-ray source operating at sufficiently high voltage consists firstly of a continuous background of polychromatic radiation with a sharp cut off at short wavelengths determined by the maximum voltage applied. Upon this is superimposed a limited number of narrow characteristic lines whose wavelengths are characteristic of the anode material. The emitted radiation is not polarised.

The type of X-ray source and the electron emission current, and accelerating voltage applied to it by the high voltage generator have a marked effect on X-ray intensity and its energy distribution. The emission current and accelerating voltage of a conventional X-ray tube normally give reproducible adjustment of the X-ray beam intensity and energy spectrum on a time scale of days. However, experience has shown that the absolute X-ray intensity differs significantly for nominally equal sources and that it decreases with source age.

4.2.3 Synchrotron radiation sources

A beam of charged particles strongly accelerated in an electric field and deflected in a magnetic field emits a continuous spectrum of X-rays that is as much as 10^{13} times as brilliant as sealed X-ray tubes. It is called "synchrotron radiation". This increased brilliance relates to the total energy spectrum. Monochromatisation of the beam typically results in diffraction intensities one or two orders of magnitude greater than from conventional sources.

The main advantages of using synchrotron radiation for XRPD measurements are:

- nearly parallel-beam diffraction geometry;
- highly monochromatised and tunable radiation;
- very small and almost symmetric contribution of the instrument to the observed line shape that leads to simpler characterisation of line profiles and very good angular resolution.

This type of X-ray radiation source requires beam flux monitoring with time (the beam flux can decrease significantly during the experiment) and wavelength calibration. The emitted radiation is strongly polarised in the plane of deflection of the charged particles.

4.3 Incident and diffracted X-ray beam optics

4.3.1 General

The main characteristics of the incident and diffracted beams are their wavelength spectrum, their direction of propagation, their cross-sectional area and shape at the specimen as well as their degree of collimation (axial and equatorial divergence) and focusing. The items of equipment that determine these characteristics are called "beam optics". The effects of these items are referred to by the general term "beam conditioning".

The items of equipment described below are used to obtain different degrees of radiation purity and different geometries, i.e. a so-called "focused" beam, "monochromatised" beam, "collimated" beam, or "parallel" beam.

4.3.2 Monochromators

4.3.2.1 General

The wavelengths conventionally used with laboratory sources correspond to the characteristic spectral lines from specific anode materials.

For many XRPD applications it is advantageous to eliminate all other spectral components emitted from the X-ray source or the specimen. When using the $K_{\alpha 1,2}$ doublet is sometimes helpful to additionally suppress the $K_{\alpha 2}$ -line, i.e. to work with highly monochromatic $K_{\alpha 1}$ -radiation.

Monochromatisation devices can be used singly or in combination.

4.3.2.2 Filters and β filters

Partial monochromatisation can be obtained using K_{β} filters, i.e. foils made of a metal selected as having an absorption edge between the wavelengths of the K_{α} and K_{β} radiation emitted by the source.

Standard K_{β} filters are designed to reduce the K_{β} intensity to about two orders of magnitude less than the K_{α} intensity. Such a filter also attenuates the K_{α} doublet intensity, typically by a factor of 2, and reduces the polychromatic radiation from the source. Two filters are sometimes combined to form so-called "balanced filters" [1].

Spectral filters shall be used with caution because the absorption edges of the filter material cause significant steps in the background close to each of the observed diffraction lines. Such steps impede accurate analysis when the complete line profile(s) are used, for example, in line-broadening analysis.

In some cases it is useful to attenuate the incident or the diffracted beam. Then a foil is used that is made of a metal (often aluminium) selected as having no absorption edge near the radiation wavelength used.

4.3.2.3 Crystal Monochromators

Monochromatisation of radiation is most often accomplished by diffraction from a crystal. We may distinguish:

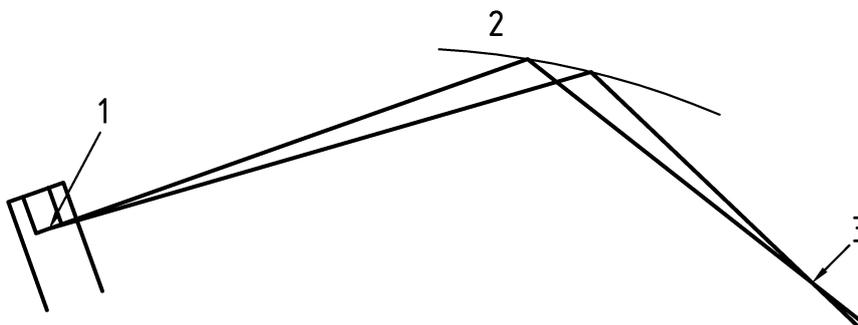
a) a mosaic crystal. The relative energy or wavelength resolution ($\Delta E/E$ and $\Delta \lambda/\lambda$, respectively) is typically a few parts per hundred. Most of the K_{β} radiation and of the continuous polychromatic radiation is eliminated. The orientations of the coherently diffracting parts of the mosaic crystal (generally graphite) are spread over about 0,4 degrees for many commercially available monochromators. For such crystals more than 50 % of the incident intensity of the $K_{\alpha 1,2}$ doublet can be retained. Mosaic crystal monochromators are usually installed in the diffracted beam. In this configuration, they also eliminate specimen fluorescence from all elements except the one that constitutes the anode.

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b) a single crystal. The energy resolution is typically better than two parts per thousand. The two components of the $K_{\alpha 1,2}$ doublet can be separated and the $K_{\alpha 2}$ radiation eliminated. This type of monochromatisation causes a large loss in intensity as typically only about 15 % of the incident intensity of a spectral line is retained. Single crystal monochromators (usually almost perfect single crystals of quartz, silicon or germanium) are generally used on the primary beam often in a focusing arrangement. The intensity loss may be largely offset by the associated reduction in axial divergence of the beam, eliminating the need for one set of Soller slits (see 4.3.3.3.2).

Two or more crystals (e.g. [2]) may be combined to achieve much better resolution than one, but the use of multiple crystals may yield insufficient intensity for normal powder diffraction. Care shall be taken to ensure that the harmonics of the chosen wavelength are eliminated by the positioning of suitable apertures (slits) within the monochromator, or by other means.

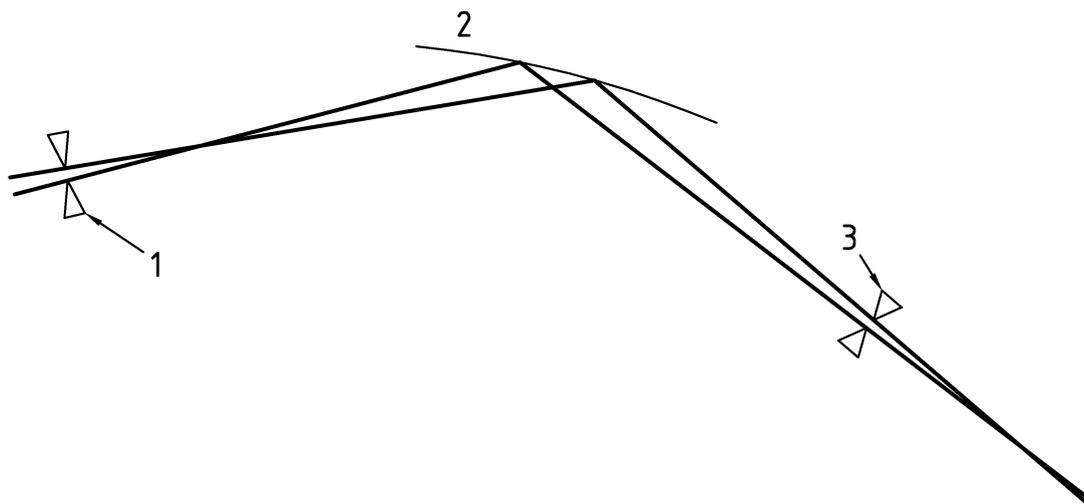
These devices can be used in the incident and/or diffracted beams (see Figures 1 and 2).



Key

- 1 Focus of X-ray tube
- 2 Monochromator
- 3 Apparent source (on the goniometer circle)

Figure 1 - Positioning the monochromator on the incident beam

**Key**

- 1 Receiving slit
- 2 Monochromator
- 3 Detector slit

Figure 2 - Positioning the monochromator on the diffraction beam

4.3.2.4 Electronic filters

Electronic filtering uses photon-counting detectors (see 4.4) and is often referred to as pulse height discrimination or “discrimination” or “energy discrimination”. It electronically selects the pulses arising from the photons of the radiation chosen for the experiment.

An effect similar to partial monochromatisation can be achieved by the use of a solid-state detector, e.g. Peltier-cooled. The relative energy resolution of these systems is of the same order as that of a mosaic crystal monochromator (about one part per 100), i.e. it is sufficient to eliminate the K_{β} component and most of the continuous background, but not to separate the $K_{\alpha 1,2}$ doublet. Detectors of this type typically have high counting efficiencies but are susceptible to dead time effects (prEN 1330-11). Such effects result in a non-linear response above about 10^4 counts per second, including all wavelengths registered by the detector, whether or not used for the measurement. Care shall therefore be taken to limit the maximum count rate observed to a level at which the detector function properly. If necessary an appropriate dead time correction shall be applied.

4.3.2.5 Multilayer mirrors

Multilayer mirrors usually consist of alternating thin layers of two elements (e.g. W and C) deposited onto an appropriately curved substrate. The layers have a thickness of the order of a few times the wavelength of the X-radiation used [3].

Multilayer mirrors for X-radiation are primarily used to reconfigure a divergent beam into an intense parallel or convergent beam (see 4.3.3.3.4). The mirror unit also acts as a monochromator by suppressing the K_{β} and polychromatic radiation from the source. The thickness of the layers deposited on the surface varies along the length of the mirror in such a way that Bragg's law is satisfied over a range of beam divergence angles and the corresponding incidence angles at the mirror. These systems reach a reflectivity close to 100 % and thereby provide high intensity beams. A common configuration uses a parabolic graded mirror to transform the divergent incident radiation from the source into an intense nearly parallel beam. Elliptic graded mirrors may be used to transform the divergent radiation from the source into a convergent (focused) beam. For special applications several mirrors can be combined in the incident and/or diffracted beams.