

JIS

JAPANESE INDUSTRIAL STANDARD

**General rules for
X-ray diffractometric analysis**

JIS K 0131—1996

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General rules for
X-ray diffractometric analysis

K 0131-1996

1. Scope This Japanese Industrial Standard specifies the general rules for the measurements of diffracted X-ray using a X-ray diffractometer in order to make the identification and determination, the accurate measurement of lattice constant, and the measurement of crystallinity degree, of substances.

Remarks 1. The standards cited in this Standard are as follows:

- JIS K 0050 General rules for chemical analysis
- JIS K 0119 General rules for X-ray fluorescence spectrometric analysis
- JIS K 0211 Technical terms for analytical chemistry (general part)
- JIS K 0215 Technical terms for analytical chemistry (analytical instrument part)
- JIS Z 9101 Safety colours and safety signs
- JIS Z 9104 Safety signs — General specification

2. The units given in { } in this Standard are based on the traditional units, and are appended for informative reference.

2. Matters in common The matters in common shall follow JIS K 0050.

3. Definitions For the purpose of this Standard, in addition to the definitions in JIS K 0119, JIS K 0211, and JIS K 0215, the definitions given below apply.

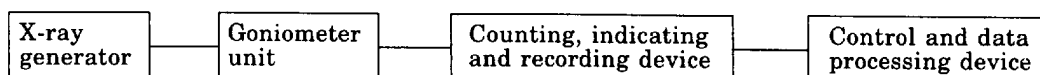
- (1) specimen rotation stage The specimen stage to uniform the influences of grain size on to the diffracted X-ray intensity by means of rotating the specimen around the axis vertical to measurement surface.
- (2) fiber specimen stage Specimen stage to measure the orientation of fiber sample, film, etc. Generally, it has a mechanism to rotate the specimen around the axis which is vertical to the lengthwise direction of the specimen.
- (3) monochromator A spectrometer to carry out the selection of wavelength of X-ray.
- (4) absorption effect (of X-ray) The decrement of diffracted X-ray intensity due to the absorption of X-ray in specimen.
- (5) preferred orientation The phenomena that specific diffracted X-ray only is strongly observed because of the orientational inclination of crystallites toward special direction.
- (6) reference material (for X-ray diffraction) A chemically stable and highly purified substance whose characteristic such as lattice constant has been accurately measured and which can be used for the standard of X-ray measurement or of analysis.
- (7) lattice constants The constants by which the size and shape [the length of an edge and angle between edges] of unit lattice in a crystal are specified.

- (8) mass absorption coefficient The quotient of the coefficient which expresses absorption degree of X-ray (absorption coefficient) by the density of the substance.
- (9) Rietveld method The method by which the most reliable value of a lattice constant or structural parameter is obtained by means of treatment of the powder diffraction data with a non-linear method of least squares. The process to get the most reliable value is called "to make something precise".
- (10) Bragg equation The equation that gives the condition for occurrence of X-ray diffraction.
- (11) Miller indices The indices by which the lattice plane can be indicated.
- (12) coherent scattering This means the X-ray which is scattered when a X-ray is incident on a substance and which has the same wavelength as the incident X-ray. Alternatively, it is called "elastic scattering".
- (13) incoherent scattering This means the X-ray which is scattered when a X-ray is incident on a substance and which has the longer wavelength than that of the incident X-ray. Alternatively, it is called "inelastic scattering".
- (14) crystallite Fine particles of single crystal in the body of polycrystal. They are usually not more than 0.1 μm and have the effect to widen the width of diffracted X-ray.
- (15) inhomogeneous strain Ununiform strain of lattice caused by the stress added to or left in a crystal grain.
- (16) residual stress The stress which remains inside of a polycrystal body under such conditions that a stress is first generated by the elastic deformation of polycrystal body caused by an external force and then the external force is removed. When the deformation takes place as by tension in a direction parallel to specimen surface, it is called "tensile stress", and when the deformation takes place as by compression, it is called "compressive stress".
- (17) radial distribution The distribution of distances from an arbitrary atom, thought to be a center, to other atoms.
- (18) texture Assembling condition of crystallites in a polycrystal body.
- (19) polar net The net made by stereographic projection of longitudes and latitudes, staying on a spherical surface, onto the equator plane.
- (20) stereographic projection In order to indicate the direction of a crystal, place the crystal at a center of sphere, and project the intersecting point (called "pole") between the spherical surface and normal line at crystal surface on the plane.
- (21) pole figure The figure which is a stereographical projection of pole position and density distribution of a specific crystal face onto a plane which is perpendicular to a certain direction of a specimen.

4. Apparatus

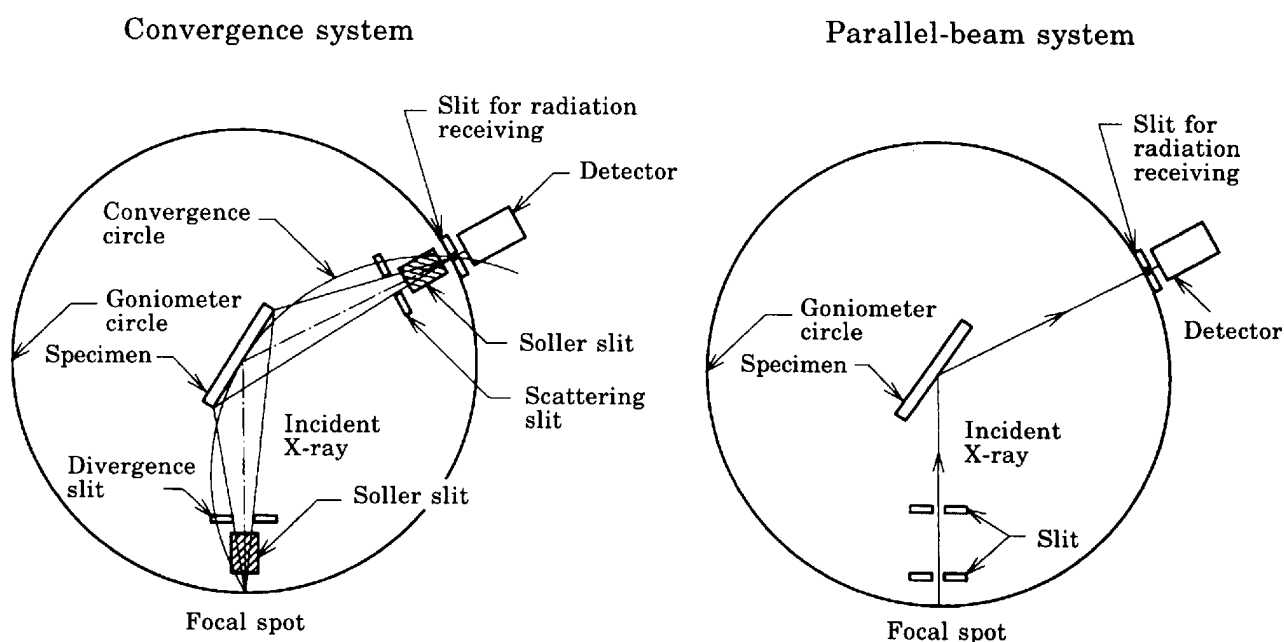
- 4.1 Configuration of apparatus The basic configuration of an X-ray diffractometer is exemplified in Fig. 1.

Fig. 1. Basic configuration of X-ray diffractometer (example)



- (1) X-ray generator X-ray generator consists of an X-ray tube, high voltage power source, and control unit.
- (1.1) X-ray tube A tube which is capable of generating characteristic X-ray and which has a capacity enough to prepare diffracted X-ray suitable for measurements. There are two types, a sealed-type X-ray tube and open-type X-ray tube ⁽¹⁾.
- Note ⁽¹⁾ Separately from these types, there is a synchrotron orbital radiation for an X-ray source.
- (1.2) High voltage power source A power source which feeds voltage and current to an X-ray tube, and which has a sufficient capacity.
- (1.3) Control unit The unit which controls voltage and current to be applied to an X-ray tube, and which consists of the following components:
- (a) Controller of tube voltage and tube current
 - (b) Stabilizing mechanism for tube voltage and tube current
 - (c) Safety mechanism
- (2) Goniometer unit This is a unit for measurement of the diffraction angle of diffracted X-ray, and shall have a sufficient accuracy on the angle and of reproducibility. There are convergence system and parallel-beam system in the optical system of the goniometer unit (refer to Fig. 2).

Fig. 2. Examples of optical system i.e. convergence system and parallel-beam system



- (2.1) Convergence system A system which consists of a divergence slit, specimen holder, and so on.
- (a) Divergence slit A slit by which divergent X-ray can be obtained.
 - (b) Specimen holder A holder capable of holding the portion of specimen to be analyzed on the circumference of the convergence circle.
- (2.2) Parallel-beam system A system which consists of a collimator or slit, specimen holder, and so on.
- (a) Collimator An apparatus capable of getting parallel fine X-ray beam.
 - (b) Slit A slit capable of getting parallel X-ray.
 - (c) Specimen holder A holder capable of holding the portion of specimen to be analyzed on incident X-ray beam.
- (3) Counting, indicating and recording device This device detects the scattered X-ray generated from the specimen, selects the diffracted X-ray necessary for analysis, and counts its intensity. Its constitution shall be as follows.
- (a) Detector An apparatus which detects X-ray, and generates pulses proportional to its intensity and proportional counter, scintillation counter, semiconductor detector, position-sensitive proportional counter, and so on are used as the detector.
 - (b) High voltage power source for detector A power source which can feed stably the voltage to be applied to a detector.
 - (c) Linear amplifier An amplifier capable of amplifying the pulse from a detector at a certain amplification.
 - (d) Pulse-height analyzer An analyzer which has the function to select the energy of X-ray by identifying the difference between pulse heights.
 - (e) Scaler An apparatus for counting the number of pulses, with enough capacity for counting.
 - (f) Timer This is an apparatus used for fixed time method and fixed counting method, and shall have a high accuracy of repeatability.
 - (g) Counting ratemeter A meter capable of continuously monitoring the intensity of X-ray ⁽²⁾.

Note ⁽²⁾ Some device is not equipped with a counting ratemeter.

- (h) Indicator Such as a recorder, printer, cathode ray tube, or display can be employed.
- (4) Control and data processing device The device controls an X-ray generator, goniometer unit, and counting device, and collects the data of diffraction. This device makes various types of correction on the data, and has at least one function out of identification of substance, quantitative analysis, making crystal construction more precise by Rietveld method, and other analytical functions. It consists of the parts stated below.
- (a) Control mechanism
 - (b) Data processing unit

4.2 Accessory To widen the scope of analysis, there are various accessory apparatus. They shall be used if necessary.

- (1) Heating and cooling apparatus
- (2) Specimen rotating stage
- (3) Specimen rotating and vibrating stage
- (4) Measuring apparatus of pole figure
- (5) Fiber specimen stage
- (6) Measuring apparatus of residual stress
- (7) Measuring apparatus of thin film specimen
- (8) Measuring apparatus of microrange
- (9) Monochromator
- (10) X-ray filter ($K\beta$ filter)
- (11) Automatic exchanger of specimen

4.3 Additional functions There are the following additional functions. They shall be added, if necessary.

- (1) Smoothing
- (2) Correction of absorption
- (3) Correction of Lorenz polarization factor
- (4) Correction of background
- (5) Peak searching
- (6) Calculation of integral intensity, peak height, half-width, integral width, and so on.
- (7) Separation of peaks
- (8) Repeated measurement
- (9) Correction of systematic error of diffraction angle
- (10) Correction of counting loss
- (11) Adjustment of goniometer
- (12) Adjustment of counting device
- (13) Setting of slit or slit width

5. Sample and its preparation

5.1 Powdery sample

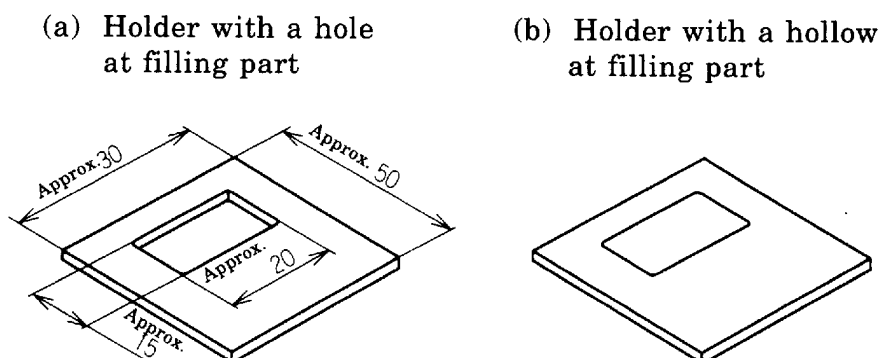
5.1.1 Grain size adjustment of powdery sample If the sample includes large size grains, grind it with a mortar or the like, manually or by use of exclusive implement in order to prepare 10 μm or less grain size. However, pay full attention to the chemical change, construction change, or contamination, probably brought by grinding.

5.1.2 Filling powdery sample into specimen holder Specimen holder shall be a glass or metal plate which has a hole or hollow on it (Fig. 3). Fill the sample uniformly and flatly in the specimen holder so that the surface of sample coincides with the holder surface ⁽³⁾.

Note ⁽³⁾ If there is a possibility of dropping out of the filled sample powder during the measurement due to incorrect filling, a little amount of grease or paste may be used.

Fig. 3. Example of specimen holder

Unit: mm



5.1.3 Sample with orientation At filling of sample into a specimen holder, the sample which is likely to cause preferred orientation shall be treated as follows.

- (1) Mix amorphous substance ⁽⁴⁾, as an diluent, with powdered sample.

Note ⁽⁴⁾ Use silica gel powder or the like.

- (2) Employ a specimen rotating and vibrating stage.

5.1.4 Small amount of sample If the filling of sample into an ordinary-type specimen holder is impossible due to small amount, carry out the following procedures:

- (1) Fix it on a reflection-free specimen plate or glass plate.
- (2) Fill it into a glass capillary.
- (3) Disperse it in a solvent, and fix it on the filter paper by filtering.

5.2 Solid sample (thin film, rod, plate, etc.) Prepare a specimen holder suitable for each type of sample, and fix the sample to the holder by using compound or grease, if necessary.

In the case of thin film, paste it together with a baseplate on a specimen holder ⁽⁵⁾, and in the case of the sample in the shape of rod or plate, cut it to make suitable size if necessary and fix it on a specimen holder ⁽⁶⁾.

Notes ⁽⁵⁾ In order to secure the number of crystals which contribute to the diffraction inside thin film, it is preferable to employ an optical system for measuring thin film in which the incident angle can be fixed 5° or less.

⁽⁶⁾ Be careful with the influence of adhesives or others, which have been used for fixing, on diffracted X-ray.

5.3 Piled dust and floating dust

5.3.1 Piled dust

- (1) Pass piled dust through a 75 μm sieve to make its grain size uniform. If necessary, treat it by ashing. In this case, be careful about chemical change caused by ashing.
- (2) Prepare sample according to 5.1.

5.3.2 Floating dust

- (1) Gather the sample on the filter by sacking and filtering the sample using an air sampler, and fix it on a filtered specimen holder, to subject it to the measurement.

In gathering of sample, a sizing apparatus may be used to meet the purpose ⁽⁷⁾.

Note ⁽⁷⁾ When inhalant dust (the dust with grain size which invades into human's respiratory organ by inhaling, and deposits there) except asbestos is gathered, employ a sizing apparatus.

(2) Preparation of reference sample for floating dust

- (a) Dust-raising method Raise dust from the sample using a dust-raiser, allow it to stand for several minutes until larger particles settle, and suck and deposit it on a filter using an air sampler, to gather it.
- (b) Suspension method Put the sample in a dispersion liquid (volatile solvent), mix them by stirring, then disperse the sample uniformly by using an ultrasonic cleaner. Gather the specified quantity of dust from this suspension on a filter by sucking and filling this liquid. Put the filter on a glass plate with its sample side upwards, and dry it naturally by allowing it in a desiccator to stand for at least 24 h.
- (c) Sprinkling method Stick double-coated adhesive tape punched out into a circular form on a specimen holder, sprinkle the sample uniformly on the tape by using absorbent cotton or the like.

6. Reference material

6.1 Reference material for accurate measurement of diffraction angle Employ a material which is chemically stable, whose lattice constants have been accurately measured, and which has a high symmetry about the profile of diffracted X-ray as the reference material. The points to be considered at selection of reference material are as stated below.

- (1) No overlapping with diffracted X-ray of unknown sample to be measured ⁽⁸⁾.

Note ⁽⁸⁾ It is preferable that the ray exists as near as possible.

- (2) Its mass absorption coefficient is as close as possible to that of unknown sample.
- (3) Select a material of as high purity as possible ⁽⁹⁾.