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

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Foreword

This translation has been made based on the original Japanese Industrial Standard revised by the Minister of Economy, Trade and Industry through deliberations at the Japanese Industrial Standards Committee as the result of proposal for revision of Japanese Industrial Standard submitted by Japan Analytical Instruments Manufacturers' Association (JAIMA)/Japanese Standards Association (JSA) with the draft being attached, based on the provision of Article 12 Clause 1 of the Industrial Standardization Law applicable to the case of revision by the provision of Article 14.

Consequently **JIS K 0119:1997** is replaced with this Standard.

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General rules for X-ray fluorescence analysis

1 Scope

This Japanese Industrial Standard specifies general rules for the measurement of fluorescent X-ray generating from a specimen using a fluorescent X-ray spectrometric analysis instrument and for performing the qualitative analysis and the quantitative analysis of elements contained in a specimen. Scope covers the thickness determination and the mapping analysis.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this Standard. The most recent editions of the standards (including amendments) indicated below shall be applied.

JIS K 0211 *Technical terms for analytical chemistry (General part)*

JIS K 0212 *Technical terms for analytical chemistry (optical part)*

JIS K 0215 *Technical terms for analytical chemistry (analytical instrument part)*

JIS Q 0030 *Terms and definitions used in connection with reference materials*

3 Terms and definitions

For the purposes of this Standard, the definitions given in **JIS K 0211**, **JIS K 0212**, **JIS K 0215** and **JIS Q 0030**, and the following definitions apply.

3.1 fluorescent X-ray

X-ray emitted when outer-shell electrons transit to holes generated by the excitation of inner-shell electrons of atoms which constitute the substance by irradiating the substance with X-rays, γ -rays, etc.

It has energy specific to the element which constitutes the substance (see **JIS K 0212**).

3.2 primary X-rays

X-rays with which a specimen is irradiated in order to generate the fluorescent X-ray

3.3 wavelength dispersive method

method to disperse X-rays generated from a specimen using a wavelength dispersion element such as an analyzing crystal and a synthetic multi-layer film (see **JIS K 0212** and **JIS K 0215**)

3.4 energy dispersive method

method to disperse (energy selection) X-rays using a detector which generates electric signals proportional to X-ray energy (see **JIS K 0212** and **JIS K 0215**)

3.5 total reflection

phenomenon in which nearly the whole incident X-ray, which strikes on a flat surface, makes total reflection when the glancing angle becomes a certain definite value or under (critical angle)

3.6 total reflection method

method to analyze the surface making use of total reflection

3.7 elemental mapping

analysis method in which an element distribution is obtained as an image

3.8 secondary target method

method in which in order to effectively excite analytical lines, the suitable target is irradiated with X-rays from a tube etc. and the fluorescent X-ray generated from that is employed for excitation (see **JIS K 0215**)

3.9 primary beam filter

filter to be interposed between the primary X-ray source and the specimen in order to change the spectral distribution of primary X-rays

3.10 secondary beam filter

filter to be interposed between the specimen and the detector in order to effectively attenuate X-rays generated from the elements other than the target elements

3.11 energy resolution

energy discrimination capability to a monochrome X-ray

It is expressed by half-width of the peak obtained [full width at half maximum (FWHM)] (the dimension of unit is energy) or the ratio (%) of half-width to peak energy.

3.12 counting loss

phenomenon in which the proportional relation between the incident X-ray intensity and the measured number of counts is broken when the X-ray intensity is measured at a high-counting rate, because the next X-ray photon comes during the dead time of a detector (the length of time from when a detector detects one photon till when the next photon becomes recognizable) (see **JIS K 0215**)

3.13 screening

method which performs the analysis of the concentration or quantity of the specific element in a specimen by focusing only on the magnitude relation with the predetermined reference value and selects the specimen

3.14 fundamental parameter (FP) method

method which obtains the analysis results theoretically from the fluorescent X-ray intensity obtained by measurement using the spectral distribution of primary X-rays, the geometrical arrangement of an instrument, constants of X-ray absorption coefficient of substance, etc.

3.15 specimen for calibration curve

specimen for creating the calibration curve

It is a group of specimens, of which the concentration of a target element is known and the composition is similar to that of the specimen to be measured.

3.16 specimen for calibration of instrument

specimen to ascertain whether the X-ray intensity generated from an instrument falls within the allowable range or not and then to aim for normalization of the instrument

3.17 background

components other than analytical lines generated from the specimen among signals which are detected with a detector

3.18 escape peak

peak which appears at a lower energy side than incident X-ray energy by the energy level specific to a detector

For example, for a silicon semiconductor detector, a peak appears at a lower energy side by 1.74 keV and for an argon proportional counter, it appears at a lower energy side by 2.96 keV.

3.19 sum peak

peak which appears at the energy position of the integral multiple of single photon energy

When two or more photons come into a detector for a short time, pulse height is observed as a sum of energy of each photon.

3.20 statistical error of counting

variation of counting values in radiation counting

This variation follows the Poisson distribution and is expressed by the square root of a counting value (see **JIS K 0215**).

3.21 thickness determination

analysis method which obtains film thickness from the relational expression of fluorescent X-ray intensity and film mass thickness (mass per unit area)

3.22 maximum limit of thickness determination

maximum film thickness of a measurable specimen in thickness determination

4 Outline

The fluorescent X-ray spectrometric analysis method is a generic name of various methods which analyze constituent elements of the specimen by measuring fluorescent X-rays specific to elements emitted when the specimen is irradiated with primary X-rays. The qualitative analysis concerning the composition of elements contained in a specimen can be performed by measuring the wavelength or energy of fluorescent X-rays, and the quantitative analysis and semi-quantitative analysis can be performed by measuring the fluorescent X-ray intensity. The fluorescent X-ray spectrometric

analysis method is classified into the wavelength dispersive method in which the wavelength of fluorescent X-rays is measured using an analyzing crystal, etc. and the energy dispersive method in which the energy of fluorescent X-rays is measured using a detector with a high-energy resolution. Among the latter, there is also an instrument for exclusive use of the total reflection fluorescent X-ray spectrometric analysis method of which the main purpose is to perform microanalysis or surface analysis. Furthermore, the micro part analysis, the two-dimensional or three-dimensional elemental mapping are also performed. The transportable instrument which incorporates the battery-driven device, the small-sized X-ray source, etc. and is designed to be compact and lightweight so as to enable easy portability is also used, and the application range has been expanded along with the enhanced performance.

5 Instrument

5.1 Constitution of instrument

The example of basic constitution of fluorescent X-ray spectrometric analysis instrument is shown in figure 1.

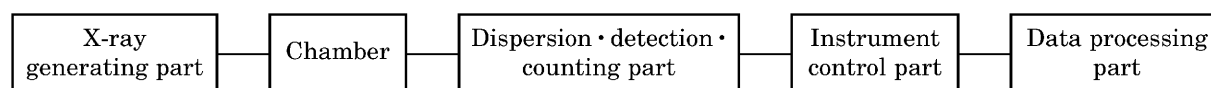


Figure 1 Basic constitution of fluorescent X-ray spectrometric analysis instrument (example)

The constitution of each part of instrument differs depending on the dispersion method to be used and the irradiation method of primary X-rays on the specimen. The dispersion methods are classified into the wavelength dispersive method that uses a spectroscope and the energy dispersive method that uses a detector with high resolution for X-ray energy. The energy dispersive method is classified into the ordinary irradiation method and the total reflection method which utilizes total reflection phenomena of X-rays on the specimen surface by irradiating with primary X-rays at not more than critical angle. The ordinary irradiation method instrument is for analyzing elements in the specimen, and the total reflection method instrument is for analyzing elements on the specimen surface and is used for the measurement of contaminants on the specimen surface and others. The instrument generally includes the large-sized instrument and the desktop instrument as well as the transportable instrument. The transportable instrument includes the portable type and the stationary type. Furthermore, there are also an instrument for analyzing the specified location on the specimen and for obtaining the image of elemental mapping. The constitution and function of each part by these methods are given as follows.

- a) **Wavelength dispersive method** The X-ray generating part consists of a X-ray tube for generating primary X-rays, a high-voltage power source to provide power to it, and a control part to adjust them. In the chamber, fluorescent X-rays are generated by irradiating the specimen with primary X-rays. The dispersion • detection part consists of a spectroscope for selecting analytical lines from fluorescent X-rays, a detector in which the X-ray intensity of analytical lines is transduced into electric signals (pulses) proportionally to the intensity and others. The spectroscope includes a parallel method [a) of figure 2] in which a Soller slit is

combined with a plane dispersion element and a concentration method [(b) of figure 2] in which a slit is combined with a curved dispersion element. The detector uses a proportional counter, a scintillation counter and others. The arrangement methods of a spectroscope and a detector are classified into a scanning method which automatically or manually sets one detector for each element to be analyzed by using one spectroscope, and a fixed method in which detectors are set respectively for each element to be analyzed. In the case of the latter, there are two methods having one spectroscope and two or more spectroscopes. The counting part consists of a proportional amplifier amplifying the pulse from the detector, a pulse-height analyzer (single channel type) selecting pulses in the specific pulse-height range, a scaler that counts the number of pulses, and others. The instrument control part outputs control signals to the X-ray generating part and the dispersion · detection · counting part. The data processing part consists of a computer and others performing various calculations such as correction.

- b) **Energy dispersive method** The X-ray generating part uses a radioisotope ray source in addition to an X-ray tube. The state inside the chamber can be made to be atmospheric air, gas displacement or vacuum according to the purpose. The dispersion · detection part consists of a detector, preamplifier thereof and others (see figure 3). The detector uses a semiconductor detector with high energy resolution, a proportional counter tube, and others. The counting part consists of a proportional amplifier, a pulse-height analyzer, a scaler, a multi-channel pulse-height analyzer, and others. The multi-channel pulse-height analyzer is capable of classifying the pulse-height range, counting pulses for every channel at the same time and storing its value in a memory. The single-channel pulse-height analyzer is also capable of measuring the pulse-height distribution by scanning the pulse-height values. The data processing part often shares a part of the counting part. For the transportable fluorescent X-ray spectrometric analysis instrument, the instrument is designed to be compact and the instrument without a chamber is also provided.

For the total reflection method, the substance surface shall be irradiated with X-rays at a very low incidence angle so that total reflection occurs on the surface. For that reason, it is necessary to provide the mechanism which is capable of controlling the incidence angle of X-rays precisely and the high-flatness specimen table. An example of constitution of the total reflection fluorescent X-ray spectrometric analysis instrument is shown in figure 4. In this example, the specimen is irradiated with X-rays generated from an X-ray tube which are monochromatized by a monochromator.

For the instrument with elemental mapping function, the specimen table control device is equipped on the specimen table, the measurement location on the specimen table is constituted to be controllable, and both methods of the wavelength dispersive method and the energy dispersive method are provided. A concrete example of constitution of instrument around the specimen table is shown in figure 5. In order to perform elemental mapping, a condenser or a collimator for making the measurement diameter small is provided. Although the example where X-rays with which the specimen is irradiated is focused is shown in figure 5, there is also a method in which the detecting diameter on the specimen is made small from the detection side.

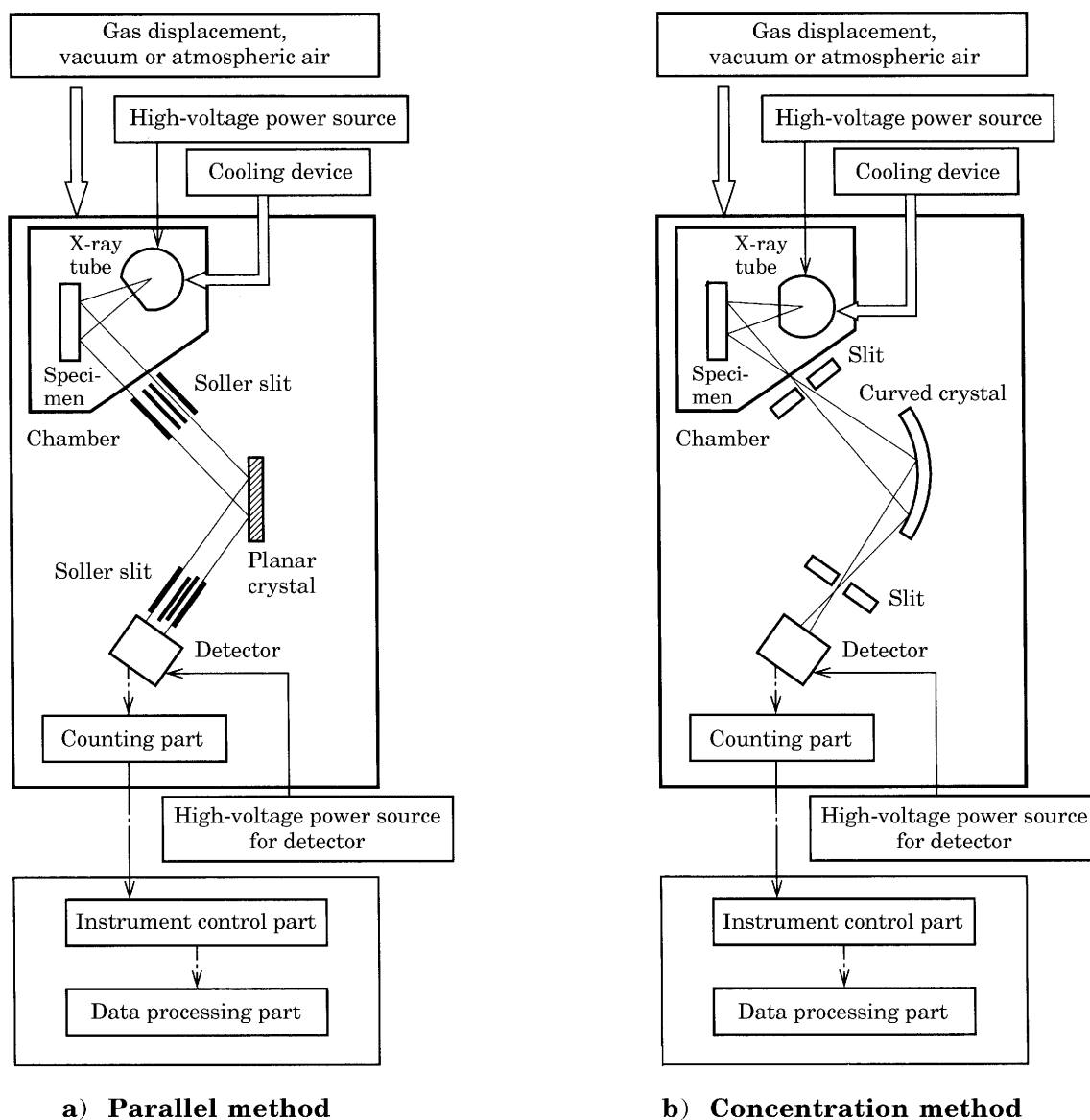


Figure 2 Constitution of fluorescent X-ray spectrometric analysis instrument for wavelength dispersive method (example)