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**General rules for infrared
spectrophotometric 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 0117:2000** is replaced with this Standard.

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General rules for infrared spectrophotometric analysis

1 Scope

This Japanese Industrial Standard specifies the general rules applicable to the qualitative analysis and quantitative analysis of inorganic and organic substances using infrared spectrophotometer¹⁾.

Note ¹⁾ In a broad definition, the term “infrared rays” means electromagnetic waves having wavelength between visible ray and microwaves. In this Standard, however, it is treated as rays in the wavenumber range of 4 000 cm⁻¹ to 400 cm⁻¹ (wavelengths 2.5 μm to 25 μm).

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 0050 *General rules for chemical analysis*

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)*

3 Terms and definitions

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

3.1 interferogram

diagram expressing the signals sent from Michelson interferometer, obtained by plotting the optical path difference of light on abscissa and the light intensity on ordinate

3.2 apodization

mathematical operation to overlap the adequate function on interferogram, in order to reduce the spectrum distortion caused by the finite scanning distance of interferometer

3.3 absorbance

numerical value²⁾ expressed by the common logarithm of the ratio between the intensity (I_0) of the incident light directed at a sample and the intensity (I) of the light transmitted through it

Note ²⁾ Absorbance (Abs) is expressed as follows.

$$\text{Absorbance (Abs)} = -\log_{10}(I/I_0)$$

3.4 Lambert-Bouguer's law/Beer's law

law³⁾ referring to the relation between intensity of incident flux and that of transmitted flux, when luminous flux is transmitted through a medium

It is also referred to as the Lambert-Beer's law.

Note ³⁾ When monochromatic incident luminous flux with an intensity I_0 is transmitted through a medium, the intensity of the light is reduced to I . The relation between I_0 and I is expressed as follows:

$$I = I_0 \times 10^{-\varepsilon c l}$$

ε : molar absorbance coefficient ($\text{cm}^{-1} \text{ mol}^{-1} \text{ L}$)

c : concentration (mol L^{-1})

l : optical path length (cm)

3.5 Kubelka-Munk transformation

method to transform spectrum measured by diffuse reflection method into absorption spectrum

3.6 specular reflection method

method to measure the intensity of reflected light using a regular reflection (specular reflection) on the sample surface

Because reflectance is the function of complex index of refraction, when it is changed to absorption spectrum, Kramers-Kronig transformation is used.

3.7 attenuated total reflection (ATR) method

a type of spectroscopy to measure high absorbing materials or the surface of materials by exploiting the penetration of electromagnetic wave into a low refractive medium which occurs near the total reflection surface when a light enters a high refractive substance at an angle greater than the critical angle and total reflection occurs inside the high refractive material

3.8 electrical direct ratio method

method to compare by electrical means the quantity of light at the sample-side luminous flux with the quantity of light at the contrast-side luminous flux when using double-beam spectroscopic analyser

4 Apparatus

4.1 General

Figures 1 to 3 show examples of construction of infrared spectrophotometer typically used for analysis. Infrared spectrophotometers are largely classified into Fourier transform spectrophotometers which use interference (Figure 1) and dispersion-type infrared spectrophotometers which use dispersion (Figures 2 and 3).

Figure 1 shows an example of construction of Fourier transform infrared spectrophotometer. The apparatus shall be composed of light source unit, sample unit, spectrophotometric unit (interferometer, detector, amplifier, A/D converter, sampling signal generator), Fourier transformation unit, data processing unit, display/recording unit, and

others. Figure 2 shows an example of dispersion-type infrared spectrophotometer utilizing an optical null method for photometric method. The apparatus shall be composed of light source unit, sample unit, spectrophotometric unit (dimmer, sector mirror, spectroscope, detector, amplifier), data processing unit⁴⁾, display/recording unit, and others. Figure 3 shows an example of dispersion-type infrared spectrophotometer utilizing an electrical direct ratio method. The apparatus shall be composed of light source unit, sample unit, spectrophotometric unit (sector mirror, spectroscope, detector, amplifier, processor), data processing unit⁴⁾, display/recording unit, and others.

Note ⁴⁾ It is not an essential component of the apparatus and therefore may be omitted in some spectrophotometers.

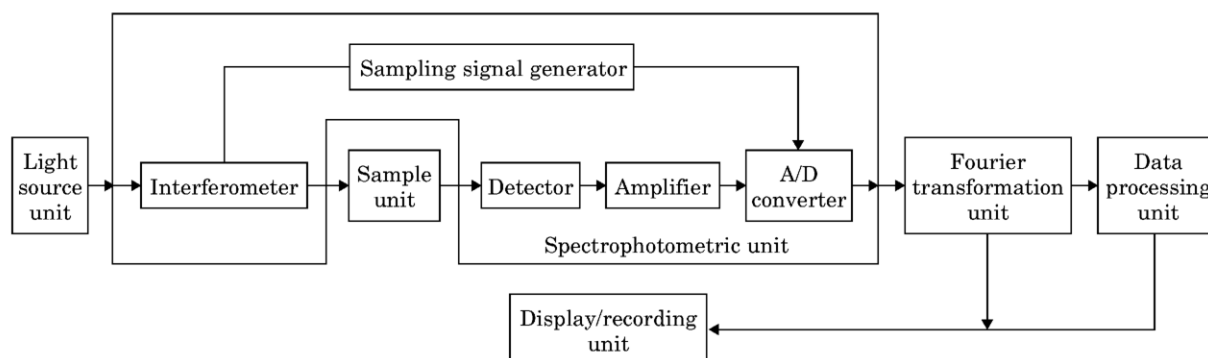


Figure 1 Example of construction of Fourier transform infrared spectrophotometer

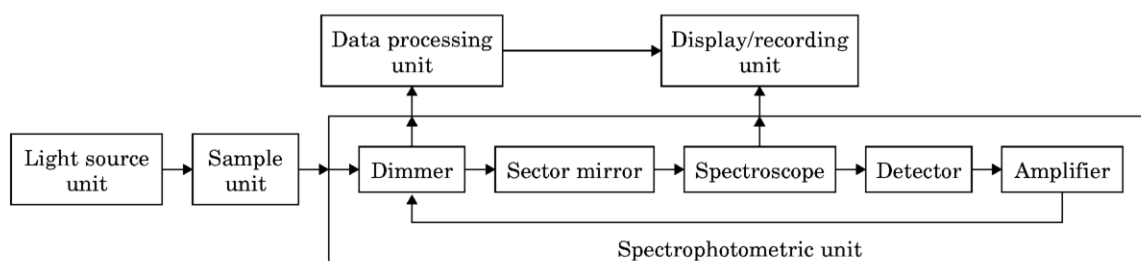


Figure 2 Example of construction of dispersion-type infrared spectrophotometer (optical null method)

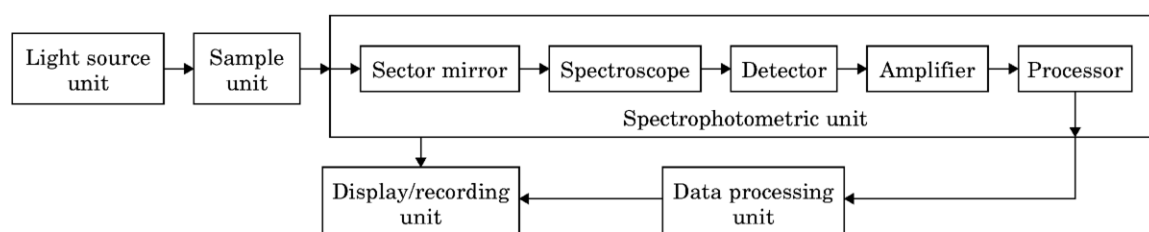


Figure 3 Example of construction of dispersion-type infrared spectrophotometer (electrical direct ratio method)

4.2 Infrared spectrophotometer

4.2.1 Fourier transform infrared spectrophotometer

Figure 4 shows an example of construction of optical system of Fourier transform infrared spectrophotometer. The infrared rays radiated from the infrared light source is reflected by an ellipsoidal mirror, condensed and passed through an aperture, transformed to parallel light by parabolic mirror and enter Michelson interferometer. The infrared rays interfered by Michelson interferometer is condensed by the parabolic mirror in front of the sample chamber and irradiated to the sample. The residual infrared rays, which are passed through the sample and absorbed by the sample, are condensed by the ellipsoidal mirror in front of the detector and converted to electric signal by detector.

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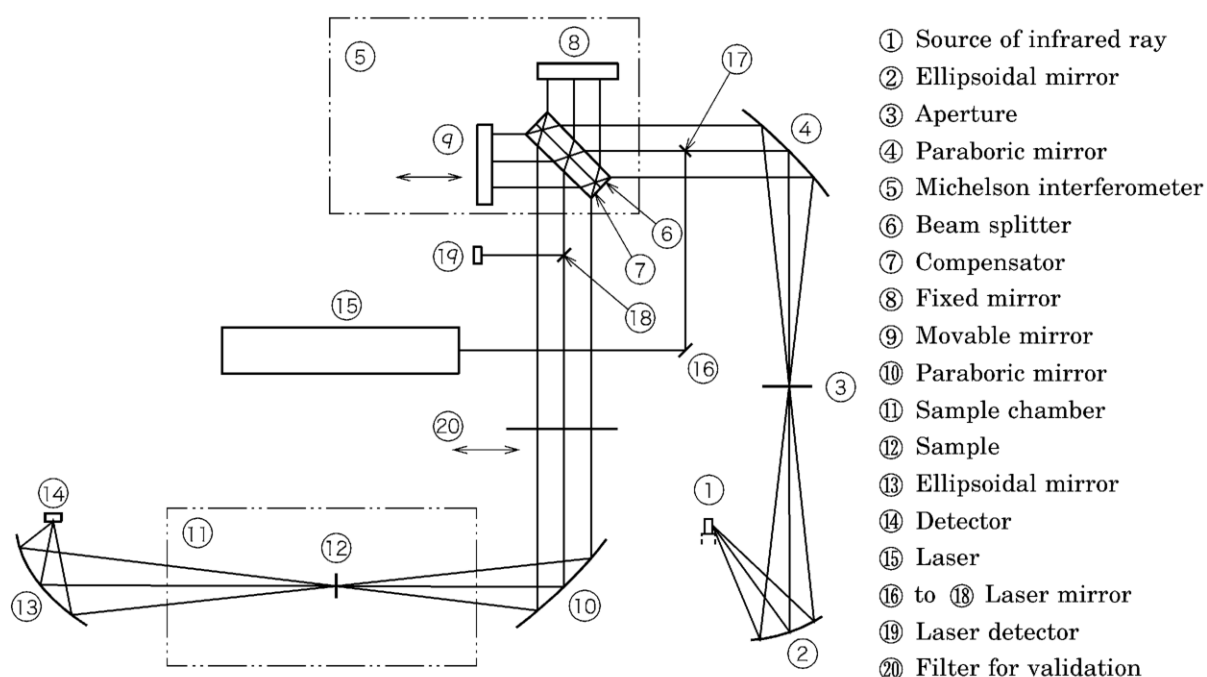


Figure 4 Example of construction of optical system of Fourier transform infrared spectrophotometer

a) **Light source unit** The light source unit shall be composed of a radiator, a power source, and a concave mirror for condensation of light.

- 1) The infrared radiator shall be made of silicon carbide, ceramics, etc., and shall be capable of stably radiating the intended infrared ray.
- 2) The power source of light source shall be capable of supplying stable voltage and current so that stable radiation of infrared ray can be obtained.
- 3) The concave mirror used for condensation of light shall be capable of condensing the infrared ray radiated by the radiator through the aperture, or of transforming the infrared ray into parallel light so that it can enter the Michelson interferometer. Configuration of this converging mirror includes elliptical, parabolic, spherical, or others.

- b) **Sample unit** The sample unit shall be composed of a sample cell, a sample holder and a jig to which accessories can be attached. Most of the Fourier transform infrared spectrophotometers are single-beam spectrophotometers. In the case of double-beam spectrophotometer, two sample holders are set up, one in the optical path for sample and the other in the optical path for contrast sample.
- 1) The sample unit is usually provided with a barrier and a door for isolating the optical path of infrared rays and sample from the surrounding environment, and in some cases, provided with an introducing opening for dry gas which enables gas displacement.
 - 2) In the entrance of the sample unit, the parabolic mirror for condensing the parallel light which went out from the Michelson interferometer in the sample unit is provided.
 - 3) In some apparatuses, validation mechanism is provided in which a polyethylene film, neutral filter or the like is inserted in the path of infrared rays so as to enable easy and sure performance validation of the apparatus.
- c) **Spectrophotometric unit** The spectrophotometric unit shall be composed of an interferometer, detector, amplifier, A/D converter, and a sampling signal generator described in the following.
- 1) **Interferometer** Michelson interferometer shall generally be used. In Michelson interferometer, the incident parallel light is split into transmitted light and reflected light by a beam splitter. These lights are then reflected by two plane mirrors, namely, a fixed mirror and a movable mirror, and then allowed to enter the beam splitter again so that they are superimposed and interfere with each other. The movable mirror is always scanned at a fixed angle to the beam splitter so that the two lights are superimposed and interference between them occurs. When the distances from the beam splitter to the fixed mirror and to the movable mirror are equal, the lights of all wavelengths enhance each other, and with the moving mirror scanning, lights of specific wavelengths enhance and interfere with each other so that interference pattern called the interferogram is observed.

In Fourier transform infrared spectrophotometer, the resolution of spectrum is determined by the moving distance of the movable mirror, and when the optical path difference between the movable mirror and the fixed mirror is L , the resolution is $1/L$. For example, if the optical path difference is 0.5 cm, the resolution is 2 cm^{-1} .

For the beam splitter, a germanium thin film which is vapour-deposited on a KBr substrate is generally provided in the mean infrared region, with a KBr substrate of the same thickness but without the vapour-deposited thin film set up as a compensator so that the optical path lengths of the fixed mirror side and the movable mirror side are equal in all wavelengths at which the measurement is made. Replacement of the substrate of the beam splitter by CsI will expand the measurement area to the long wavelength region, and the use of CaF_2 substrate with the ZnSe thin film vapour-deposited will expand it to the short wavelength region.

Interferogram signals are obtained by sampling the signals from the sinusoidal wave generated by the interference of He-Ne laser light built in the interferometer at accurately equal distance interval, transforming the signals into digital signals by means of an A/D converter, and input into the computer. The interference signal of the He-Ne laser light is also utilized for controlling so that the scanning of the movable mirror is performed at constant speed.

In the interferometer, it is important that the angle of the fixed mirror and the movable mirror to the beam splitter is always kept constant, and for collection of high quality data, it is essential that the beam splitter or the mirror be adjusted to an optimum angle before measurement. In some apparatuses, automatic and continuous adjustment of the interferometer is performed through monitoring the phase of interferogram of He-Ne laser light, and electrically adjusting the mirror accordingly.

In the usual measurement, the linear scanning measurement mode in which data is collected by moving the movable mirror at a constant speed is used. In some apparatuses that are capable of time-resolved measurement where the change of sample within sub-millisecond is measured or of measurement of the pulse-modulated sample, the step scanning measurement mode in which data is collected by stopping the movable mirror at each sampling position can also be provided.

At the entrance of a Michelson interferometer, the parabolic mirror or the like for transforming the radiation light from the light source into the parallel light that is required in the Michelson interferometer shall be provided.

Some apparatuses are provided with an aperture for restricting the size of incident light for the purpose of high resolution measurement.

- 2) **Detector** The detector, used for converting the incident light into electrical signals based on its intensity, shall be e.g. a pyroelectric detector, semiconductor-type detector, photoacoustic detector. The detector used for Fourier transform infrared spectrophotometer is required to satisfy the conditions indicated in the following. However, since a detector satisfying all of the following conditions cannot be expected to be available, the optimum detector shall be selected in accordance with the intended purpose of analysis.

The detector used shall

- have a high sensitivity;
- have a wavelength sensitivity characteristic which is flat within the measurement wavelength range;
- have a frequency characteristic which is flat within the Fourier frequency;
- have a signal output intensity which is in linear relation to the incident light intensity over a wide range;
- have a sensitive area of the size corresponding to the beam diameter of the optical system;
- be simple in handling.

In front of the detector, an ellipsoidal mirror or the like for condensing the infrared rays from the sample unit to the detector shall be provided.

- 3) **Amplifier** The amplifier has a function of amplifying the electric signals from the detector to a size in which they can be easily processed in the subsequent processing system.
- 4) **A/D converter** The A/D converter has a function of converting the analogue signals from the amplifier into digital signals so that they can be processed by computer.

Since handling of the interferogram signals detected by the detector of the Fourier transform infrared spectrophotometer requires a large dynamic range, an A/D converter with a high resolution of about 24 bits is used in some apparatuses.

- 5) **Sampling signal generator** The sampling signal generator has a function of generating a reference signal used in data sampling in interferogram measurement. Generally, for a monochromatic light source for obtaining the interferogram of the reference signal, a helium-neon laser is used. In other cases, a solid laser may be used.
- d) **Fourier transformation unit** The Fourier transformation unit has the function of transforming the digitalized interferogram to the spectrums by Fourier transformation.
- e) **Data processing unit** The data processing unit carries out the transformation and analysis of the spectral data obtained. On the two dimensional data collected by the imaging measurement function such as an infrared microscope, the unit performs the multivariate analysis such as the main component analysis, the multivariate spectral resolution and the image analysis of imaging data such as measurement of the position, number, and area of the region occupied by each component, as well as the data processing of individual spectrums.
- f) **Display/recording unit** The display/recording unit is the unit in which displaying the analytical and data-processing results on the monitor, printing them via a printer and storing them in the external storage is performed, normally by the computer together with Fourier transformation and data processing.

4.2.2 Dispersion-type infrared spectrophotometer

- a) **Light source unit**, in accordance with 4.2.1 a).
- b) **Sample unit**, in accordance with 4.2.1 b). This spectrophotometer shall normally be a double-beam spectrophotometer where a sample holder is set up each in the optical path for sample and in the optical path for contrast sample.
- c) **Spectrophotometric unit** The spectrophotometric unit shall be composed of a dimmer, spectroscopy, detector, amplifier, processor, and others.
 - 1) **Dimmer** The dimmer is an optical element used to decrease light in the optical null method. This element is set up in the luminous flux for contrast and controls the intensities of the luminous flux which passed through the sample and the luminous flux which passed through the contrast sample so that they are equal.
 - 2) **Sector mirror** The sector mirror is a rotating mirror for switching between the luminous flux of sample and that of contrast.

- 3) **Spectroscope** The spectroscope is composed of a slit, mirror, dispersion element, and others. It is the instrument in which the light from one light source is dispersed and focused into a slit image on one focal surface in the order of wavelength so that the spectrum can be observed. The dispersion element shall be composed of a prism, diffraction grating or an optical system combining these. It may be a monochromator which takes out a specific wavelength of monochromatic light, or a polychromator which takes out lights of multiple wavelengths simultaneously. Generally, a diffraction grating⁵⁾ is used.

Note ⁵⁾ The diffraction grating spectroscope is generally used in combination with the wavenumber selection filter in order to separate the light of higher order.

- 4) **Detector** The detector, used for transforming the incident light into electrical signals according to its intensity, shall be a vacuum thermocouple, a pyroelectric detector, or a semiconductor-type detector.
- 5) **Amplifier** The amplifier has a function to amplify the signals from the detector to a size in which they can be easily processed in the subsequent processing system. In optical null method, it is composed of a preset amplifier, main amplifier, synchronous rectifier, modulator, and power amplifier. In electrical direct ratio method, it is composed of a preset amplifier, main amplifier, and synchronous rectifier.
- 6) **Processor** The processor is a signal processing system used for the electrical direct ratio method, and can separate electric signal by sample luminous flux from that by contrast sample, and calculate the ratio between their signal intensities.

Figure 5 illustrates an example of the optical system of the dispersion-type infrared spectrophotometer (optical null method).