DOUBLE-BEAM METHOD OF DETERMINING THE THERMORADIATION CHARACTERISTICS OF MATERIALS DISSIPATING RADIATION

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A method and optical diagram of a double-beam infrared spectrophotometer developed by the authors to measure the spectral and integral transmissivity and reflectivity of materials dissipating radiation are described. The results of measuring the transmissivity and reflectivity of samples of pinewood of different thickness in the 0.5-10.0 μ spectrum range are presented.

Theoretical investigations of the radiation energy transport process in capillary-porous colloids and various technical materials dissipating radiation [1] pose the problem of developing more precise experimental methods [2] of determining the thermoradiation characteristics, the reflectance R_λ and transmittance T_λ, of the objects under investigation.

Known methods and adapters to double-beam [14] and single-beam [10, 13] infrared spectrophotometers using exposure of the sample to an integral radiation flux which causes it to be heated strongly during the measurement, cannot be used to study the thermoradiation characteristics of moist materials, and of materials whose optical properties are altered during heating.

Double-beam instruments using a monochromatic radiation flux to expose the sample [6, 7] are used in the visible spectrum range (0.40-0.75 μ), but only single-beam instruments [3-13, 15, 16] are presently used in the infrared spectrum range ($\lambda > 1.0\ \mu$).

Among the deficiencies of the single-beam method are the extreme difficulty and the long duration of the measurement and processing of the results obtained, and high demands imposed on maintaining the temperature of the radiation source and the surrounding medium constant, the invariance of the sample and etalon properties and the high accuracy in mounting them at the same place during the measurement, as well as the demands to assure constant gain of the detector–recorder unit.

The double-beam method permits elimination of the mentioned deficiencies to a significant degree, and obtaining a qualitative reflection or transmission spectrum of the sample, and requires a lesser expenditure of time. An advantage of the double-beam method is also the fact that the characteristics of all elements of the instrument should be kept constant only for a time equal to the period of rotation of the mirror interrupter (~0.1 sec). Optical signal compensation permits utilization of a radiation detector with a nonlinear light characteristic, but possessing high response.

Underlying the method of measuring R_λ and T_λ of materials dissipating radiation by means of the double-beam scheme is the method of the Coblentz mirror hemisphere [16], developed and used up to now only in combination with single-beam instruments and primarily for measurement of R_λ.

The optical diagram of a universal double-beam infrared spectrophotometer developed and produced by the authors on the basis of the IKS-14 device, is shown in Fig. 1 in principle. The optical diagram of the device is such that it permits measurement of the spectral R_λ and T_λ as well as the integral R and T of materials dissipating radiation, and making an absorption spectrum analysis by the customary double-beam method.

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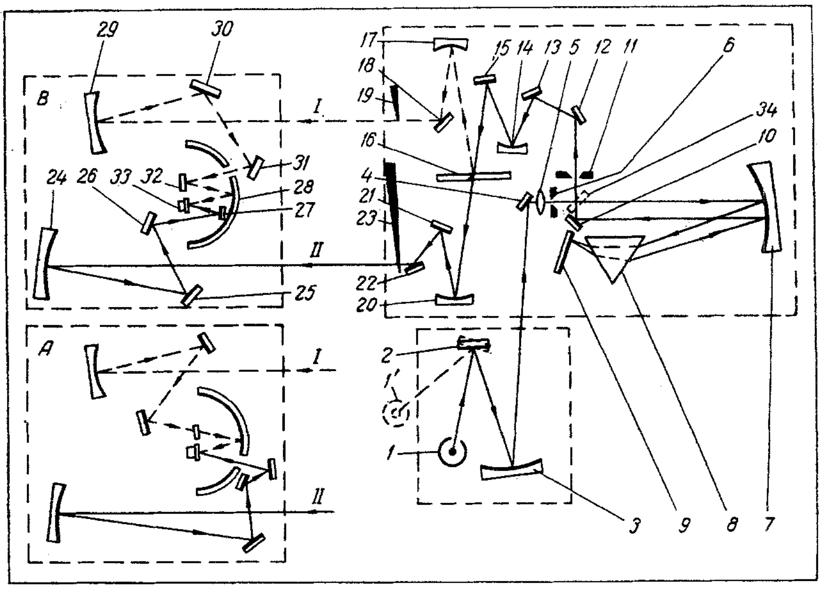

Fig. 1. Optical diagram, in principle, of a double-beam infrared spectrophotometer to measure the reflectance and transmittance of materials dissipating radiation.

The sample is exposed to a monochromatic modulated radiation flux for the measurements of R_λ and T_λ, which permits elimination of the influence of the intrinsic sample radiation on the instrument readings and significant reduction in the heating during the measurement process.

The image of the radiation source (glow bar) 1 is projected with a ×1.3 magnification on the entrance slit 6 of the monochromator by using flat 2, spherical 3, and flat 4 mirrors. The collecting lens 5 is mounted in front of the entrance slit 6 for uniform exposure of the slit over the height. Passing through the slit, the integral radiation flux is incident on the parabolic mirror 7, and having been reflected therefrom is expanded in a spectrum by the prism 8. Then, having been reflected from the mirror 9 the monochromatic radiation flux passes through the prism a second time and is focused by the mirror 10 onto the exit slit 11. By rotating the mirror radiation fluxes of different wavelengths can be directed at the exit slit, and then focused in the plane of the reflecting interrupter surface 16 by using flat 12, 13, 15, and toroidal 14 mirrors.

As the interrupter rotates (at a 8.8 Hz frequency), the monochromatic radiation flux is directed, by using the toroidal 17 and flat 18 mirrors, to channel I or alternatively to channel II by using the toroidal 20 and flat 22 and 21 mirrors.

A compensating wedge 19 is located in channel I, and a photometric wedge 23 in channel II.

An adapter B is mounted to measure R_λ, and the radiation flux of channel I is focused through an orifice (∅ 20 mm) in the hemisphere with a single magnification onto the outer surface of the sample (etalon) 32 at one of the conjugate points of the equatorial plane of the hemisphere 28 (ρ = 55 mm) by using the spherical 29 and the flat 30, 31 mirrors. Diffusely reflected radiation from the sample is collected at the receiving area of the detector 33 by using the hemisphere.

The radiation flux of the comparison channel II is also focused with a single magnification at the radiation detector 33 by using the spherical 24 and flat 25, 26, 27 mirrors.

An adapter A is mounted for the T_λ measurements, and the radiation flux of channel I is focused with single magnification on the outer surface of the sample (etalon) 32 by using the spherical 29 and flat mirrors 30, 31. Having passed through the sample (etalon), the radiation is collected at the receiving area of the radiation detector 33 by using the mirror hemisphere 28. The radiation flux of the comparison channel II is also focused through an orifice in the hemisphere on the radiation detector 33 by using the spherical 24 and flat 25, 26, 27 mirrors.

Therefore, the radiation of channel I is incident on the detector after reflection from (or passage through) the sample, and the radiation of channel II is directly incident on the radiation detector. The detector records the radiation from channel I after reflection from, or transmission by, the sample and

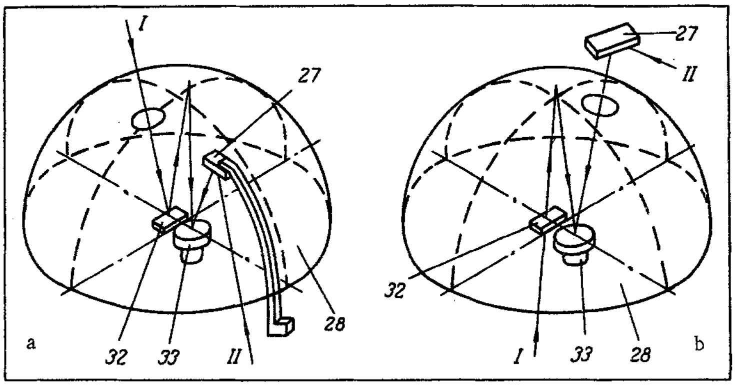

Fig. 2. Diagram of the arrangement of the sample 32, the radiation detector 33, the mirror 27, and the hemisphere 28.

from the comparison channel II alternately at the frequency 8.8 Hz. The difference in the exposure of the detector is compensated by the insertion of a photometric wedge 23 in the comparison channel II, which is connected mechanically to the pen of the recording unit, and a record of the reflection or transmission spectrum is therefore obtained graphically in percent with respect to the etalon.

A mirror with aluminum outer coating deposited on a glass surface is the reflection etalon. An empty cuvette, transmitting substrate, etc., depending on the kind of material to be investigated are the transmission etalons.

Open-type PS-AI photoresistors and others whose threshold sensitivity is 10- to 1000-fold greater than bolometers, RTE, opticoacoustic, and other thermal radiation detectors are used as radiation detectors. Utilization of cooled photoresistors, bolometers, RTE, etc., is possible to expand the operating range of the instrument and for integral measurements.

In order for the absorption spectrum of atmospheric gas vapors (H_2O, CO_2, etc.) not to be superposed on the sample spectrum, the distances between the interrupter 16 and the radiation detector in channels I and II should be equal. It would moreover be necessary to achieve an equal number of reflections from the mirror surfaces since the mirrors also absorb part of the incident radiation. All this was achieved by taking the following precautions.

1. An additional flat mirror 21, which compensates radiation absorption in channel I by the mirror surface of the interrupter 16, is mounted after the interrupter in channel II.
2. The radiation flux of channel I is incident on the detector after reflection from (or passage through) the sample and reflection from the mirror hemisphere, i.e., traverses an additional path equal to two radii of the hemisphere 28 (in our case $2\rho = 110$ mm). To obtain an image of the slit on the detector with single magnification in channels I and II it is necessary that the radius of curvature of the spherical mirror 29 be 55 mm less than the radius of the mirror 24.
3. An additional mirror 27, which compensates radiation absorption by the mirror surface of the hemisphere 28 in channel I, is mounted in channel II.

Upon compliance with these measures, the radiation fluxes in channels I and II traverse equal distances and undergo an equal number of reflections up to the time of incidence on the radiation detector.

Therefore, the superposition of absorption spectra of atmospheric gas vapors and absorption by mirrors are excluded.

Meanwhile, utilization of the mirror hemisphere does not assure absolute values of the diffuse reflection or transmission coefficients will be obtained as a result of the measurements. The qualitative reflection or transmission spectrum turns out to be correct, but the quantities R_λ and T_λ are obtained reduced as compared to their true values. This is because not all the radiation energy reflected from or transmitted through the sample reaches the detector, but only some part. The fundamental reasons for losses in the measurements of R_λ are: a) loss of reflected radiation through the entrance orifice; b) the screening effect of the mirror 27 set within the hemisphere and the mounting of the radiation detector; c)

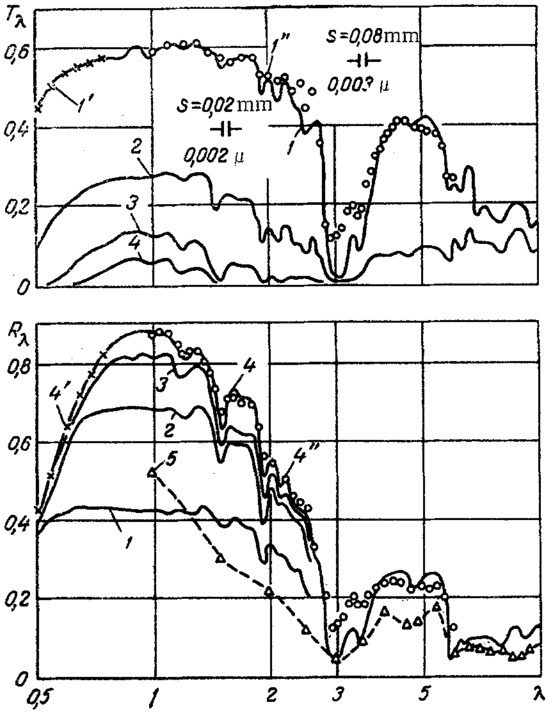

Fig. 3. Dependence of the spectral transmittance T_λ and reflectance R_λ of pinewood on the wavelength λ for different sample thicknesses: 1) 0.14 mm; 2) 0.46; 3) 1.05; 4) 2.26; 5) 3.00; [1-4) data obtained by the described double-beam method]; 1',4') data obtained by the double-beam method of an integrating sphere in an SF-10; 1",4") data obtained by a single-beam method on an IKS-12 with a Selyukov adapter [5]; 5) data obtained by a single-beam method [4], λ, μ.

aberration of the optical system whereupon the image exaggerates the size of the receiving area of the radiation indicator.

Losses of the reflected radiation through an entrance orifice of 20 mm diameter are around 3% of all the reflected energy for diffuse reflection. Losses of the specular component can turn out to be significant since the specular component through the entrance orifice can emerge almost completely beyond the limits of the hemisphere.

Losses of the specular component are eliminated by a special arrangement of the entrance orifice in the hemisphere as shown in Fig. 2. In this case the angles of incidence of radiation specularly reflected by the sample and of radiation from the comparison channel directed by the mirror 27 to the detector are approximately identical. Then the signal of the radiation detector for reflection from the sample will be proportional to the recorded energy

$$N_{\mathrm{I}} \equiv Q_0 R_\lambda R_{\mathrm{hemi}}(1 - R_{\mathrm{det}} + R_\lambda R_{\mathrm{hemi}}^2 R_{\mathrm{det}} - R_\lambda R_{\mathrm{hemi}}^2 R_{\mathrm{det}}^2 + \ldots). \quad (1)$$

The signal for recording of the radiation energy of the comparison channel will be proportional to the following value:

$$N_{\mathrm{II}} \equiv Q_0 R_{\mathrm{mirror}}(1 - R_{\mathrm{det}} + R_\lambda R_{\mathrm{hemi}}^2 R_{\mathrm{det}} - R_\lambda R_{\mathrm{hemi}}^2 R_{\mathrm{det}}^2 + \ldots). \quad (2)$$

Such an arrangement of the sample, detector, and hemisphere permits elimination of the influence of multiple reflection between the sample and the detector on the measured value of R_λ since the ratio between the signals $N_{\mathrm{I}}/N_{\mathrm{II}}$ equals $R_\lambda R_{\mathrm{hemi}}/R_{\mathrm{mirror}}$, which equals the reflectance R_λ of the sample when R_{hemi} and R_{mirror} are equal. The quantity R_λ determined in this manner is less than the true value only because of the radiation loss.

The second reason evokes a large radiation loss. The dimensions of the mirror 27 within the hemisphere are 10 × 25 mm. The mirror 27 is fastened to a rectangular 6 × 8 mm rod as shown in Fig. 2. The radiation losses because of screening by the rod and the mirror are about 6%. The use of open-type photoresistors permitted cutting down the radiation losses because of screening by the detector mounting, from 50% [5] and 25% [3] to 7%.

Therefore, the total radiation losses because of screening and leakage through the orifice are about 16% by computation, which was later verified experimentally.

The aberrations of the optical system were reduced to a minimum by compliance with the condition $s \le 0.1\rho$ [3].

TABLE 1

Material	Measured quantity, %		Calculated, %	
	R	*T*	*R*	*T*
Pinewood (1.5 mm thick layer)	70,5	5,3	72,8	5,7
Plastic foam PS-4 (8.0 mm thick layer)	78,5	—	80,5	—
White enamel AS-2	77,2	—	78,9	—

The total radiation losses in the T_λ measurements are less and are about 10% since the mirror 27 is brought out of the hemisphere. To eliminate the influence of multiple reflections on the quantity T_λ to be measured, the sample, the radiation detector, and the entrance orifice of the hemisphere are arranged as shown in Fig. 2b. In such an arrangement the detector signals will be proportional to the radiation energies in channels I and II (Fig. 2a and b):

$$N_I \equiv Q_0 T_\lambda R_{hemi}(1 - R_{det} + R_\lambda R_{hemi}^2 R_{det} - R_\lambda R_{hemi}^2 R_{det}^2 + \ldots); \quad (3)$$

$$N_{II} \equiv Q_0 R_{mirror}(1 - R_{det} + R_\lambda R_{hemi}^2 R_{det} - R_\lambda R_{hemi}^2 R_{det}^2 + \ldots). \quad (4)$$

When R_{hemi} and R_{mirror} are equal, the ratio N_I/N_{II} equals the value of T_λ of the sample, which is less than the true value only because of the radiation losses.

Therefore, the proposed double-beam method permits determination of the qualitative spectrum of diffuse transmission and reflection. The absolute values of R_λ and T_λ are found by multiplying the ordinate of the spectrum by the appropriate coefficients k_R and k_T. The value of the correction coefficient k_R is 1.16 and of k_T is 1.10. It must be noted that as the sample thickness increases, the quantity k_T will also increase in measurements of T_λ (for $T_\lambda < 50\%$) since radiation losses can appear because of an increase in the size of the radiation spot issuing from the sample. The spot dimensions can exceed the detector dimensions, and part of the radiation will not be incident on the detector. In this case the quantity k_T must be determined from a comparison of the T_λ obtained by this method and the method of a detector with a large receiving area [2, 5].

Special spectrum sweep detents and slit openings were fabricated for operation with the prism F-I in the 0.40-0.75 μ spectrum range. In the 0.75-15 μ spectrum range, linear spectrum sweep detents in the wave numbers and wavelengths applicable to the IKS-14 instrument, and specially fabricated detents for the slit opening were used.

Presented in Fig. 3 are reflection and transmission spectra of pinewood samples of different thickness, obtained by the described double-beam method of a mirror hemisphere (solid lines), by the double-beam method of of an integrating sphere (points 1' and 4') by using an SF-10 spectrophotometer, and by the single-beam method [4, 5] of a mirror hemisphere (points 1", 4", and 5). It is seen from the figure that the agreement between the values of R_λ and T_λ obtained by these methods is quite satisfactory. The advantages of the proposed double-beam method are the possibility of obtaining a qualitative spectrum of the material, and the high resolution of the instrument which permitted more correct values of R_λ and T_λ to be obtained in the region of the absorption bands of the material and the atmosphere, especially near the 30 μ wavelength (Fig. 3). The working slit width is 0.02-0.06 mm. Under these conditions, the half width of the spectrum interval $\Delta\lambda/2$ extracted by the slit equals 0.002-0.006 μ for a monochromator with F-I prism, and 0.0007-0.002 μ with a LiF prism. Such results have been obtained only in the spectrum range where the maximum radiation intensity of the glow bar is located during operation with vacuum bolometers by the ordinary method. The slit width in known apparatus [3-5] is 0.2-1.5 mm in this spectrum range, i.e., the resolution is one-tenth that in the apparatus described.

The double-beam method described permits shortening the duration of the experiment considerably as compared with the known adapter to the IKS-12 [5], from 6 h to 10-25 min with high measurement accuracy.

The accuracy of determining R_λ and T_λ depends on many factors, including the nature of the reflection and transmission. Reproducibility of the quantities R_λ and T_λ for the same sample is obtained with not more than a ±1% spread. The accuracy of measuring R_λ and T_λ by the method described is higher than the accuracy of measurements by known single-beam methods because of the 10- to 1000-fold increase in the sensitivity of the instrument, the exposure of the sample to modulated, monochromatic radiation flux, the diminution in the magnitude of the correction inserted in the measurement results from 1.8 [11], 1.5 [5], and 1.25 [3] to 1.16, the use of optical compensation of the signal and the total elimination of the influence of radiation absorption by water vapor and carbon dioxide gas of the atmosphere on the instrument readings.

To measure T_λ by the customary double-beam method, the sample is mounted in channel I and the etalon in channel II, and the spectrum is recorded. A verification showed that the transmission spectra obtained by the described and the customary double-beam methods, agree completely for 0.035 mm thick polystyrene film.

To measure the integral thermoradiation characteristics R and T of materials, the radiation source used 1' is mounted at a distance from the mirror 2 equal to the distance between the glow bar and the mirror 2. The mirror 2 is rotated so that an image of the source 1 would be on the entrance slit 6. A flat mirror 34, which reflects the integral radiation flux onto the exit slit 11, is mounted behind the monochromator entrance slit. In such an apparatus the integral radiation flux from the radiation source used is directed to channels I and II alternately at the frequency 8.8 Hz.

The integral reflectance and transmittance of various materials were measured by using the apparatus described.

Presented in Table 1 are values of R and T for certain materials relative to radiation of an NIK-220-1000 lamp, obtained by the method described and calculated by averaging the quantities R_λ and T_λ over the spectrum of this lamp.

As is seen, the measured and calculated values of R and T of the same materials differ slightly, which indicates the confidence of the results obtained by the method described.

Therefore, by using the described double-beam infrared spectrophotometer, the spectral and integral thermoradiation characteristics of materials dissipating radiation can be measured and an absorption analysis of absorbing media can be carried out.

NOTATION

R, T	are the reflectance and transmittance of a layer of finite thickness;
N	is the radiation detector signal;
Q_0	is the radiation flux density;
k	is the coefficient taking account of radiation losses.

Subscripts

λ	denotes the spectral radiation;
hemi, mirror, det	denote the coatings of the hemisphere, flat mirror, and detector receiving area, respectively;
R, T	denote the sample reflectance and transmittance.

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