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LUMINESCENT ANALYSIS OF OPTICAL GLASSES

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[Drawings and tables are appended]

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In 1936 at the suggestion of S. I. Vavilov the GOI, undertook to develop a method and apparatus for grading optical glasses by luminescence. Preliminary experiments showed that considerably more brilliant luminescence of optical glasses can be attained by short-wave ultra violet rays (wavelength about 250 mμ), than by using radiations of longer wave length (wavelength about 365 mμ). The two independent characteristics observable are a prolonged (about 10^{-2} second) glow emitted by the glass, and one of shorter duration (less than 10^{-5} second), which will be referred to as phosphorescence and fluorescence, respectively. The apparatus constructed for this purpose - a spark fluorophosphoroscope /1/, is shown in Figure 1 (Diagram of the fluorophosphoroscope). This device makes possible successive observations of fluorescence and phosphorescence, (without altering the position of the object) merely upon switching the voltage by means of a commutator. A high voltage condenser spark flashed between iron electrodes F located at the lower part of the apparatus is a fairly rich source of short-wave ultra violet rays. The segregation of the short-wave portion of the spectrum (wavelength about 250 ~ 300 mμ) for the purpose of producing fluorescence is attained by the use of a slitless monochromator consisting of a quartz lens L and a quartz prism P. Observation of phosphorescence is made possible by the use of a spark phosphoroscope placed at the upper part of the apparatus. In the phosphoroscope the voltage is transmitted to a stationary electrode E and to the movable electrodes E_1, E_2, E_3 and E_4 which are rotated by motor M. The phosphorescence is observed by means of mirrors S_1 and S_2. The apertured disk D, revolving on the axis of the motor, screens the flash of the spark and the object A so illuminated from the eye of the observer. Thus the observer views the object through the apertures of disk D only during intervals between flashes. This permits observation of phosphorescence for not less than 10^{-3} second

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duration. The two features, first the consecutive observations of fluorescence and phosphorescence, and second the powerful light source rich in far ultraviolet radiations (from the condenser spark between iron electrodes), advantageously differentiate this device from earlier apparatus for grading optical glasses by luminescence [2,3]. This fluorophosphoroscope tried out at a number of plants made possible an efficient and rapid inspection of the glasses employed in manufacturing operations.

The spark fluorophosphoroscope can also find applications in mineralogy. It is known that the vast majority of native minerals are natural phosphors , possessing the property of glowing upon exposure to ultraviolet rays - from a fraction of a second up to several hours. All native minerals absorb short-wave ultraviolet rays more readily than they do longer wave radiations, and consequently the luminescence intensity of minerals in the fluorophosphoscope is considerably greater than in a conventional apparatus consisting of a mercury lamp and a wood filter. Furthermore, analysis by phosphorescence has the added advantage of eliminating entirely any background interference, since the glowing object is observed only after the action of the excitant source has ceased.

However, the spark fluorophosphoroscope has one substantial disadvantage: It is actuated by a dangerous high-voltage condenser spark (10,000 volts, transformer capacity 0.2 – 0.25 kilowatts). Though provided with an automatic blocking system, the apparatus can therefore be recommended for operation only at laboratories staffed with adequately trained personnel. Consequently it was most desirable to develop, for the luminescence analysis of optical glasses, apparatus using a low-voltage source of excitation. To this effect PRK-2, PRK-4, and SVD mercury quartz lamps, produced by domestic manufacturers, were tested. It was found that lamps PRK-2 and PRK-4 display sufficiently intense lines in the short-wave region of the ultraviolet spectrum (wavelength equals

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= 254~313 mμ) and are quite satisfactory as a source of luminescence excitation of optical glasses. The SVD lamp, in which the resonance line of mercury (wavelength equals 254 mμ) is greatly weakened as a result of self-reversal, is considerably less suitable for this purpose. Therefore, in the new apparatus a PRK-4 mercury quartz lamp serves as the source of ultraviolet radiation. Figure 2 (Diagram of the Luminoscope) shows schematically the luminoscope developed by us, and designed for grading optical glasses under plant conditions. In this apparatus, the same as in the above-described fluorophosphoroscope, the luminescence method of grading, consists in comparing the luminescence of the specimens tested with that of standards made from optical glass of a known variety. A PRK-4 quartz bulb, mercury lamp operating under normal conditions at 110 volts and 4 amperes, is the source (L) of ultra violet rays.

Phosphate glass colored by nickel and cobalt is used as the light filter (c). Its transmittance curve is shown in Figure 3 (Transmission Curve of the Light Filter). By means of a light condenser (K) the source of ultraviolet radiation is projected onto the tested specimens of optical glass located on stage (E). Fluorescence is observed visually with filter (S) in position. This filter has a fairly high transmittance in the 405 mμ region (Figure 3). Therefore, when testing weakly luminescent glass samples it is advantageous to supplement this filter with a liquid filter consisting of an aqueous solution of nickel sulfate contained in a plane-parallel quartz cell. Such a liquid filter of 1 centimeter thickness, with a 15 percent concentration of nickel sulfate, transmits well in the wave range of about 250 - 300 mμ, and absorbs completely the violet portion of the spectrum. If the glass being tested does not fluoresce in the blue-violet position of the spectrum, then, to eliminate the effect of dispersed violet rays, it is recommended to substitute for the nickel sulfate solution a light yellow filter, placed in front of the observer's eye.

Observation of phosphorescence is made by means of a phosphoroscope

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consisting of a disk provided with four symmetrical apertures and fitted to the axis of motor M. The speed of the motor is regulated by rheostat P_1, and reaches 2000 revolutions per minute. Excitation of phosphorescence is produced by unfiltered radiations of the same source of ultraviolet rays (L), the filter (S) being removed from the path of the radiation. Color comparison of the phosphorescence of the tested specimen with that of the standard is made by a photometric system consisting of biprism (B), objective (O) and eyepiece (G). This system gives within the field of vision as seen through the eyepiece, juxtaposed images of the phosphorescent objects, i.e., the tested sample and the standard. The observer's eye sees the biprism illuminated at the moments when the rotating disk discloses the phosphorescent objects and at the same time screens the source of radiation. Thus one half of the biprism is illuminated by the glow of the phosphorescent sample being tested, and the other half by that of the phosphorescent standard. For convenience of observation the optical axis of the photometer is turned about by means of mirror T.

This apparatus was used to study the fluorescence and phosphorescence of a large number of optical glasses produced by our industry in accordance with GOST 3514-47. It is known that depending on their chemical composition, optical glasses are classified into several types having definite refractive indices and dispersion. Table 1 (Classification of Optical Glasses) shows the classification of optical glasses according to OST .

We had available a set of standards made from different types of glass.

Table 2, (Luminescence Characteristics of Optical Glasses) showing the luminescence characteristics of the glasses studied, is the key to the luminescence method for grading optical glasses.

In this table optical glasses of different types are divided into four basic groups according to the color of their fluorescence (columns 1-2). In addition each group is divided into subgroups (column 3), embracing glasses having identical shades of fluorescence color. The color of phosphorescence makes possible further subdivision of glasses grouped by the color of their fluorescence. Thus the collection of glasses which were available to us, was divided, according to their fluorescence into 23 groups. Further subdivisions by the color of the phosphorescense yielded the data shown in column 4. In column 5 are shown the varieties of glass which can be definitely identified by the luminescence method, as well as those which cannot be differentiated by this method. Thus, for example, glass varieties K4, K6, K9, and BK6, cannot be differentiated on the basis of their luminescence characteristics. For these varieties, as well as for others listed in the table as not identifiable by luminescence, it is necessary to conduct additional refractometric tests. However, the segregation of these glasses into a separate group characterized by definite luminescence properties greatly expedites their final identification, by the index of refraction.

Under shop conditions, by using our apparatus it is possible to examine not less than 1000 glass samples within 8 hours. The shape and degree of polish of the glass surface are of no importance. When examining samples of frosted glass it is preferable, however, to compare their glow with that of a frosted surface of the standard. Comparisons of the luminescence of optical glass samples of the same variety but of different batches, have shown the occurence of variations within a given variety, depending on the batch. Thus, for example, the shade of the luminescence of K8 glass varies noticeably from one batch to another. Some of the other varieties, such as BK 10, display rather constant colors of luminescence. The table given cannot be considered as final. It is intended merely as a guide for a more detailed study of glass luminescence; which should be conducted under plant conditions. Such a study may lead to certain corrections of the tabulated data. Basically, however, the table shown, apparently, will remain essentially unaltered. This assumption rests on results obtained from comparing a series of domestically produced glasses with the corresponding varieties, of foreign manufacture, having identical optical constants. Of the K1 varieties thus tested, 10 were identical in the color of the glow; the hues of the remaining 4 were approximately the same, and differed only in the finer shades of glow coloration. These facts indicate that the luminescence of optical glasses is determined, apparently, not by adventitious admixtures, which may vary in batches of different origin, but by the essential constituents of the glass.

Experience gained on using the above described apparatus at a number of plants has proven that the luminescence method of glass grading is most useful in controlling optical details at various stages of manufacturing processes. The apparatus described, as well as the spark fluorophosphoroscope, may also be very useful in luminescence studies of minerals.
State Optical Institute.

Literature Cited:

1. E. M. BRUMBERG and Z. M. SVERDLOV, Journal of optico-mechanical industry, 10-11 (1938).

2. S. M. TOKMACHEV. Ibid., 4-5 (1931).

3. J. GRANT. The Glass Industry 19,387,(1938)

Discussion of Brumberg, Sverdlov and Timofeyeva's paper:

S. I. Vavilov. - I wish to point out that the method of photoluminescence analysis of optical glasses just described constitutes a record-breaking example of grading analysis. Without resorting to spectroscopy, merely by a color comparison, it is possible to identify, quite definitely, approximately 40 varieties of optical glass. Grading analysis in its simplest form, utilizing only visual color sensitivity, has been strikingly illustrated here, insofar as its potentialities and advantages are concerned.

Table 1.

OST Classification of Optical Glasses.

Type of Glass	Designation	Characteristic Compounds of the Type
Crowns	K	BaO and PbO < 3%
Barium crowns	BK	BaO 3 - 30%
Heavy crowns	TK	BaO > 30%
Crown flints	KF	PbO 3 - 15%
Light flints	LF	PbO 15 - 40%
Barium flints	BF	BaO and PbO > 3%
Flints	F	PbO 40 - 50%
Heavy flints	TF	PbO > 50%

Table 2.

LUMINESCENCE CHARACTERISTICS OF OPTICAL GLASSES

NATURE OF LUMINESCENCE

GROUP NUMBER	FLUORESCENCE: Basic Color	FLUORESCENCE: Subgroup by Color	PHOSPHORESCENCE: Color	GLASS VARIETIES
1	2	3	4	5
		I-A	Red	K1
			Pink	K2
		I-B	Pink	K3
1	Yellow		Yellowish	BK4
			Greenish	K4,K6,K9,BK6.
		I-B	Pink	K5,K8.
		I-G	None	F3,F4,F5,
		I-D	Weak red	O2.
		I-E	Pink	K7.
		I-Zh	Red	K11.
		II-A	Very weak red	TK8
			Weak red	TK4,TK9
			Pink	BK5,TK2
			Yellow	BK7

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(Table 2 continued)

	Basic Color	Subgroup by Color	Color	
1	2	3	4	5
		II-B	Red	BK8,TK1
II	Lilac		Pink	K10
		II-V	Weak pink	O1
			Green-yellow	BK2
		II-G	Red	TK3
			Green-yellow	BK3
			None	LF3
		II-G	Red	TK3
			Green-yellow	BK3
			None	LF5
		II-D	Weak pink	TK5,TK6,TK7,TK10
		III-A	None	TF2
		III-B	None	BF14,BF15
III	Azure	III-V	None	TF1
		III-G	None	F1,F2,F3,F4.
		III-D	Very weak pink	BF12,O3.
		III-E	None	BF16
		III-Zh	Red	LF7
		IV-A	Weak Red	BF13,TK11
			Red	LF5,LF6
			Pink	BF8,LF4
		IV-B	Weak Pink	BF4,BF5,LF1
			Pink	BF6, BF11
IV	Blue	IV-V	Red	KF2
			Yellow	KF1
			Yellow Green	BF1
			Greenish	BF3,KF3
		IV-G	Red	BK9,BK10,K12,BF7
			Bright red	BF9

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Figure 1. Diagram of the Fluorophosphoroscope.

A- Object; F-holder of iron electrodes, for the excitation of fluorescence; L - quartz lens; P quartz prism; E-stationary, and E_1 and E_2 mobile, electrodes, for the excitation of phosphorescence; D-disk for screening the spark at the moment of discharge; M-motor; S_1 and S_2 - mirrors; G - commutator.

Figure 1.

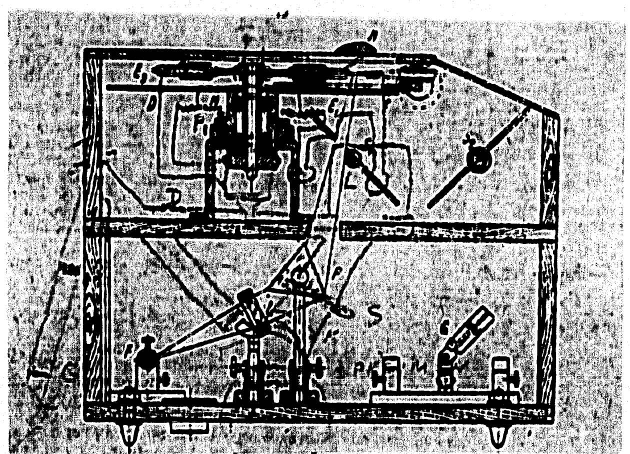

Figure 2. Diagram of the Luminoscope.

L- mercury lamp, LPK-4; S- light filter; K-condenser; E- sample stage; B- biprism; T- mirror; O-objective; G- eyepiece; D- disk; M-motor; P_1 and P_2 rheostats; A- ammeter.

Figure 3. Transmission curve of the light filter (thickness 2 millimeters).

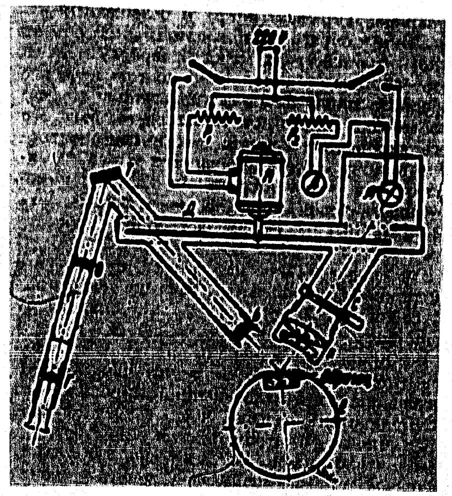

Figure 2

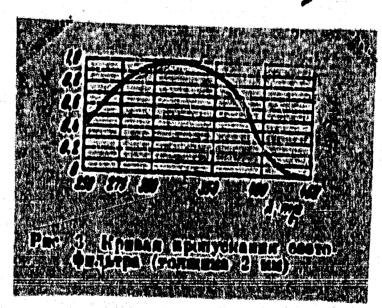

Figure 3

Curve of Transmittance of the Light Filter (Thickness: 2 mm).

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