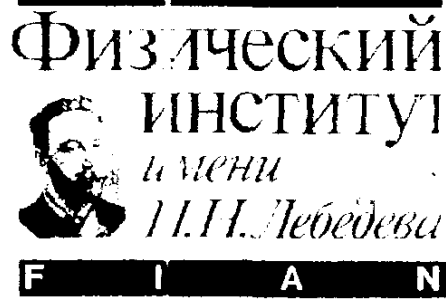


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1995 11 / 10 191 CA 4002  
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PREPRINT

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PBWO<sub>4</sub> CRYSTALS PRODUCED AT  
DIFFERENT GROWING-CONDITIONS**

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Moscow 1995

# CHARACTERISTICS OF SCINTILLATING $\text{PbWO}_4$ CRYSTALS PRODUCED AT DIFFERENT GROWING CONDITIONS

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## ABSTRACT

The characteristics of heavy scintillating  $\text{PbWO}_4$  crystals produced at different growing conditions have been studied. All crystals were grown in All-Russian Research Institute of Synthesis of Mineral Raw Materials. Crystal colours are varied from bright yellow (orange) to slight yellow and almost colourless depending on oxidation potential of the growing atmosphere.

It is shown that colour centers in crystals are created due to the transition of part of lead ions in the melt into three valent state and its further re-entry into a crystal which causes defects involving  $\text{Pb}^{3+}$ .

The measured light yield of the best samples was 6.4 % of that of BGO crystal. Decay times were established to be  $\tau_1 = 1.95$  ns,  $\tau_2 = 6.9$  ns,  $\tau_3 = 28.5$  ns.

All samples were irradiated by  $\gamma$ -quanta from a powerful  $^{60}\text{Co}$  source up to doses of 460 krad and 7 Mrad. For colourless samples the significant improvement in transmission spectra are observed for high dose of irradiation compared with low one as well as almost self annealing after 2 weeks of the exposure for one sample is seen.

## 1 Introduction

Starting with the first communication from S.Derenzo et al. [1] the intensive studies on scintillation properties and radiation hardness of  $\text{PbWO}_4$  crystal are being carried out [2-8]. This scintillating crystal used in e.m. calorimetry has several advantages compared to already known scintillators: fast decay time and emission spectrum with good correspondense to the spectral sensitivity range of most commonly used photodetectors, high density, relatively cheap raw material. Some characteristics of  $\text{PbWO}_4$  crystal are listed in Table 1. Lately the interest for this crystal increased since CMS Collaboration (LHC, CERN, Geneva) is developing on its basis an electromagnetic calorimeter consisting of 93312  $\text{PbWO}_4$  crystals in the barrel and 16128 ones in the end cap regions.

**Table 1. Characteristics of  $\text{PbWO}_4$  crystal.**

density, ( $\text{g}/\text{cm}^3$ )	8.28
radiation length, (cm)	0.89
Moliere radius, (cm)	2.13
light yield, (% of BGO)	5 <sup>[5]</sup>
decay time, (ns)	2.1, 7.5, 26 <sup>[5]</sup>
refractive index	2.16
melting point, ( $^{\circ}\text{C}$ )	1123 $\pm$ 5
hygroscopicity	no

According to [4-8] optical nonuniformity along the full size crystal and shift of emission spectra for different parts of undoped crystal were observed. The introduction of Nb allowed to obtain more optical

uniform crystals and increased its radiation hardness [5-8]. However, for  $\text{PbWO}_4$  doped with Nb wide absorption band around 420 nm is observed which slightly decreases the crystal light yield. Note, that even for the best  $\text{PbWO}_4$  samples light yield doesn't exceed 5 % from that of BGO crystal.

Below we present the first results on X-ray phase studies as well as measurements of scintillation characteristics and radiation hardness of  $\text{PbWO}_4$  crystals grown in All-Russian Research Institute of Synthesis of Mineral Raw Materials.

## 2 Growing technique

$\text{PbWO}_4$  single crystal belongs to tetragonal singony and has a sheelite-type structure, its space group is of a  $J4_1/a$ -type. An elementary cell contains 4 formular units, its parameters are  $\mathbf{a} = 5.45 \text{ \AA}$  and  $\mathbf{c} = 12.043 \text{ \AA}$ . Its structure is made up of lead and tungstene oxigenic polihedrons. The corresponding coordinating polytypes are dodecahedron and tetrahedron slightly flattened on the  $\mathbf{c}$  axis. The crystals have the average cleavage on the  $(111)$  plane and an incomplete one on the  $(001)$  and  $(101)$  planes.

State diagram studies of  $\text{PbO-WO}_3$  system [9] have shown that a crystallization of  $\text{PbWO}_4$  corresponds to its central section of 50 $\text{PbO}$ -50 $\text{WO}_3$  (mol.%) and occurs at  $1123 \pm 5 \text{ }^\circ\text{C}$ . Anyway a slight deviations from the central section (approximately  $\pm 2$  mol.%) appreciably decreases compound melting points and leads to co-crystallization of  $\text{WO}_3$  or another tungstate  $\text{Pb}_2\text{WO}_5$  present in the system [9]. The melting takes place congruently in all the range of  $\text{PbO-WO}_3$  contents and that makes it possible to grow crystals in the system by melting technique.

Besides the tetragonal  $\text{PbWO}_4$  specified above ( $\beta$ -fase, stolzite) other two monoclinic modifications are established in the given tungstate:  $\alpha$ - $\text{PbWO}_4$  (raspite) and high pressure phase of  $\text{PbWO}_4$  - **111** [9]. Polymorphic transition  $\alpha \rightarrow \beta$   $\text{PbWO}_4$  found out at 875 °C in one of previous studies hadn't been confirmed by latest ones (see the list of references in [9]).

Crystals have been grown by Czochralski method in a resistive heating setup using platinum crucibles. The mechanical mixture of  $\text{PbO}$  and  $\text{WO}_3$  oxides taken in stoichiometric ratio is used as a charge. The following standard industry made oxides are used:  $\text{WO}_3$  - special purity for making optical glass;  $\text{WO}_3$  - chemical purity for phosphors;  $\text{PbO}$  - chemical purity for spectral analysis (yellow modification);  $\text{PbO}$  - special purity (red modification). Certified total contents of foreign matters in the initial oxides are: not more than  $162.3 \times 10^{-4}$ ;  $335 \times 10^{-4}$ ;  $118 \times 10^{-4}$  and  $6.5 \times 10^{-4}$  mass % respectively. Crystals have been grown on seeds oriented approx. on forth order axis i.e. on **001**. The process is performed at crucible wall temperature of 1150-1160 °C, pulling rate of 3-6 mm/hour and rotation of 30  $\text{min}^{-1}$ . The atmosphere varied from neutral to oxigenic during experiments.

Crystals 90-100 mm long and 20-22 mm in diameter have been obtained. The crystals are transparent, contain no breaks; occasionally white, finely dispersed inclusions are present at its rear zone. Crystal colour varies from bright orange-yellow to slight lemon-yellow and almost colourless depending on oxidizing potential of gas atmosphere. Very slight gray subcolour is observed in some colourless samples. No dependence on oxide type has been observed within all the range of used oxide types excluding red  $\text{PbO}$ . In the last case in spite of its extreme chemical purity the orange-yellow colour of crystals is of max-

imum intensity. This fact indicates that quality and quantity of foreign matter in charge content don't effect on crystal colour. The content of an atmosphere and introduced lead oxide modification play a crucial role in crystal colour. Therefore, X-ray phase studies of grown material and initial lead oxides are performed taking into account that phase content of crystals themselves and lead content in charge components may produce a colour of  $\text{PbWO}_4$ .

### 3 X-ray phase studies of crystals and initial lead oxides

The studies were performed using DRON-4-07 diffractometer ( $\text{CuK}_\alpha$ -irradiation, Ni-filter). Some samples have been studied additionally with differential-temperature method (heating and cooling in air at  $10^\circ/\text{min}$  rate). Material selected from the rear crystal zones as well as from the head ones is analyzed.

According to X-ray diffraction data high temperature orthorhombical phase (massicot) and low temperature tetragonal one (glet) are presented in yellow modification  $\text{PbO}$  at quantities of 80 and 20 mass %, respectively. Red modification lead oxide contains 97-98 mass % of glet, 1-2 mass % of massicot and less than 1 mass % of hydrocerussite  $\text{Pb}_2(\text{CO}_3)_2(\text{OH})_2$ .

Almost all lines in the diffractogramms of all crystal samples correspond to  $\beta$ -phase of  $\text{PbWO}_4$ . Along with that fact the presence of slight additional reflections are observed in some colourless samples and all coloured ones. The number of these reflections for each of diffractogram is low (2-5), its intensity is 0.2-2.5 % compared with the main reflection (**112**) of  $\beta$ - $\text{PbWO}_4$ . As a rule, the number of these additional reflection and its intensity are higher for material from rear zones (espe-

cially when impurities are present) then for one from head and central parts of the crystal. The number of these lines were indentified as corresponding to monoclinic phase of  $\alpha$ -PbWO<sub>4</sub>. An affiliation of the rest part of additional reflections to some known oxide, intermetallic lead or tungsten compound, as well as possible products of reaction of melt components with crucible walls has not been found (all corresponding files from a catalogue were used [10]). The reflections of an unidentified phase are observed on diffractograms of slight-yellow crystals and, in specific cases, of colourless ones. The number and intensity of lines of this phase decrease when intensity of colour increases. The reflections corresponding to  $\alpha$ -PbWO<sub>4</sub> predominate among additional ones of middle- and bright-coloured crystals. The comparison of number and intensity of additional lines on diffractograms with quality and quantity of impurities in initial oxides reveals no correlation between the level of phase nonuniformity of crystals and the presence of some special impurity or its total content in a charge. Along with that, crystals produced with red lead oxide have the maximum number of relatively intense additional reflections.

Differential-thermo analysis has not shown the effect of impurities in charge and phase nonuniformity level on thermal behavior of crystals in the range from room temperature to melting one. Within this range the derivatograms of all samples are the same and don't contain anomalies. Endothermic effect of melting appears at the temperature of 1120-1125 °C which corresponds to less symmetric exothermic effect of crystallization for the cooling curves at the same temperature. No effects have been observed in PbWO<sub>4</sub>-Pb<sub>2</sub>WO<sub>5</sub> and PbWO<sub>4</sub>-WO<sub>3</sub> eutectic melting as well as in polymorphous transformation  $\alpha \rightarrow \beta$  PbWO<sub>4</sub>. Correlation of these data with X-ray diffractometric ones confirms the

absence of  $\text{Pb}_2\text{WO}_5$  and  $\text{WO}_3$  compounds and indicates that  $\alpha \rightarrow \beta$  transformation either really doesn't occur or proceed slowly and with low intensity.

Thus, the composition of all coloured and part of colourless crystals obtained deviates from monophase  $\beta\text{-PbWO}_4$ . These deviations are not large. It seems likely that unidentified phase ( $\alpha$  phase of lead tungstate) creates clusters statistically distributed in  $\beta\text{-PbWO}_4$  matrix. Crystal colour corresponds to a presence of  $\text{PbWO}_4$   $\alpha$ -phase. The unidentified phase doesn't effect crystal colour. Breaking of phase composition is not a result of lack of chemical purity of used oxides. However, these breakings are stronger when crystals are grown under oxigenic atmosphere and, especially, when lead oxide of low temperature tetragonal modification (glet) is used. Phase nonuniformity increases from head zone to rear one along the length of the crystal.

The data obtained indicate that the phase inhomogeneity and yellow colour centers happen during crystal growing and are substantially caused by oxigenic processes in a melt as well as by form of the lead oxide present. The highest oxidation level (+6) of tungsten in  $\text{WO}_3$  as well as lowest one (+2) of lead in  $\text{PbO}$  suggest that lead oxide is the oxidating component of the charge. The proof of that are the data [12] on rather easy  $\text{PbO}$  oxidation to  $\text{Pb}_3\text{O}_4$  (more specifically, to the mixture  $(\text{Pb}_2^{2+}\text{Pb}^{4+})\text{O}_4$ ) and further decay according to the scheme  $\text{Pb}_3\text{O}_4 \leftrightarrow \text{PbO} + \text{Pb}_2\text{O}_3$ . Significantly, the oxidation rate of red  $\text{PbO}$  is higher than that of yellow one [13]. The comparison of our results with the above mentioned data is well founded and allows to consider the creation of colour centers as a consequence of transition into three valent state of part of lead in melt and its further re-entry into crystal yielding defects involving  $\text{Pb}^{3+}$ . In the framework of this scheme the



increasing brightness of yellow colour when using red PbO is explicable by a consequence of high intensity of oxidation process of lead oxide in a melt. Co-crystallization mechanism of secondary phases as well as a connection of colour centers with a presence of  $\text{PbWO}_4$   $\alpha$  phase is not known till now and additional studies are needed.

#### **4 Studies of transmission and X-ray luminescence spectra of different coloured crystals**

The initial crystals have been cut up to cylindrical samples with the following dimensions: 11.0 mm in diameter and 10.0 mm long for spectral studies. The butt surfaces of the samples have been polished. The same way a BGO sample for comparison is manufactured. The position of selected zones in each sample in the crystals has been fixed to determine variation of material characteristics. Transmission spectra in visible and close UV regions (280 - 900 nm) are recorded on spectrometer "Specord-M 40". X-ray luminescence spectra are obtained using special setup based on KSVU-2 assembly ( $\text{MoK}_\alpha$  irradiation, PMT-100) in the range of 330-750 nm. In doing so, the emission is detected at the end sides of the samples placed at an angle of  $45^\circ$  to the orthogonal directions of excitation flux and PMT's optical axis. All measurements were performed at room temperature.

Transmission spectra of crystals obtained are shown on fig. 1. Observed optical absorption boundary of all samples is close to 333 nm. The wide broad absorption band in the range of 375 - 500 nm with the maximum at  $\lambda \approx 423$  nm is characteristic for coloured crystals. The bandwidth is constant, its intensity increases with increasing intensity of yellow colour. In this connection values of absorption coefficient

spectra don't show blue ( $\lambda_{max} = 464$  nm), green (538 nm) and red ( $\sim 700$  nm) band characteristics for  $\text{PbWO}_4$  [15,16], it is evident that resulting spectra are produced by superposition of blue and green components with prevailing contribution of the last one. Traces of the red component are not observed even at maximum setup sensitivity.

Short wavelength part of the green band and all the whole of the blue band are overlapped within the range of 375-500 nm in coloured crystals. Value of corresponding emission loss is determined by its absorption inside a luminescent layer when the mentioned above geometry of obtaining spectra is used. Taking the thickness of the layer as depth of thousandfold attenuation of  $\text{MoK}_\alpha$  irradiation in  $\text{PbWO}_4$  at its falling angle to a sample of  $45^\circ$  and estimating the absorption at the band maximum ( $\lambda = 423$  nm) for obtained  $d = 74 \mu\text{m}$ , one can see that for the extremely bright-coloured crystals the emission losses exceed that for colourless material by a factor not higher than 4 tot%. Such small difference of losses inside thin exciting layer suggests that the observed shift of maximum of X-ray luminescence spectra of crystals with loosening and vanishing of its colour is not as a consequence of absorbing effects but mainly a result of decreasing yield of green luminescence. The last factor determines the accompanying decrease of summed spectra. The most probable reason of all these changes is a decrease of number of centers of green luminescence. It should be noted that absorption losses in 375-500 nm band sharply increase with material layer thickness so when detecting emission from the opposite end of a sample high intensity of luminescence of the coloured crystals is almost completely smeared.

so that to have an independent assembly stable in time. The "STOP" channel was equipped with a diaphragm which allows to adjust the amount of light in the channel operating in photon counting mode. PMT's were arranged orthogonal to each other in order to avoid direct hits of  $\gamma$ -quanta from the  $^{60}\text{Co}$  source in the "STOP" PMT window (see fig. 9). PMT signals enter constant fraction discriminator ORTEC 463 and then 1024 channel TDC KA-317.

Time decay spectra processing was carried out using special program which minimises  $\chi^2$  distribution. To approximate initial data the function consisting of a sum of three exponents and a line reflecting background was chosen. The fit parameters were used to estimate the contribution of each decay component into the total light yield of a crystal under investigation. All results are listed in Table 2.

**Table 2. Decay times of various  $\text{PbWO}_4$  crystals.**

sample	$\tau_1$	$\tau_2$	$\tau_3$
PWO 1.E (yellow)	2.9 (34.1 %)	9.5 (37.8 %)	33.0 (28.1 %)
PWO 3.E (yellow)	2.6 (34.3 %)	10.8 (38.9 %)	76.9 (28.4 %)
PWO 1.6 (yellow)	2.9 (35.6 %)	10.9 (35.9 %)	91.3 (28.4 %)
PWO 3.8 (colourless)	2.0 (23.4 %)	6.9 (48.4 %)	28.5 (28.1 %)
PWO 3.85 (colourless)	2.2 (28.4 %)	7.9 (47.6 %)	33.9 (23.9 %)

According to Table 2 colourless  $\text{PbWO}_4$  samples have shorter decay times compared with yellow ones (especially  $\tau_3$ ). Our results on decay time measurements of colourless PWO crystals are similar to [5].

## 5 Light yield measurements

For measuring the  $\text{PbWO}_4$  light yield samples were coated with white teflon tape and coupled to XP-1911 photomultiplier by "Rhodorsil" optical grease. The signal from the samples irradiated by  $^{137}\text{Cs}$  source enters the amplifier and then ADC ORTEC AD811. Pulse height spectra for all samples under investigation are presented in fig. 7. It is evident that photopeaks corresponding to yellow colour samples (1.E, 3.E, 1.6) have a smaller amplitude than those of colourless ones (3.8, 3.85).

Absolute light yield of  $\text{PbWO}_4$  samples was measured by comparing it with that of BGO crystal of the same size. For this comparison the  $\text{PbWO}_4$  3.85 sample was chosen and QDC KA-010 was used having gate width of 400 ns. When measuring BGO pulse height spectrum the attenuation was 16 dB. Spectra for  $\text{PbWO}_4$  and BGO are shown in fig. 8. By comparing these spectra one can calculate that  $\text{PbWO}_4$  3.85 sample light yield is 6.4 % of that of BGO sample. Note that in accordance with S.Derenzo et al. [1] light yield of  $\text{PbWO}_4$  is 3.6 % and with M.Kobayashi et al. [3] it is 4.4 % of that of BGO.

## 6 Decay time measurements of $\text{PbWO}_4$ crystals

Decay times were measured using "delay coincidence" [17] and "separate scintillators" methods [18]. A small piece of YAP scintillating crystal coupled to a PMT was used in the "START" channel. A crystal under investigation was placed at a distance of 4 cm from "STOP" PMT inside its frame. The investigated crystal and YAP were irradiated by  $^{60}\text{Co}$  source which was located inside "START" PMT frame

## 7 Radiation hardness of $\text{PbWO}_4$ crystals

Radiation hardness of  $\text{PbWO}_4$  samples was studied by comparing transmission spectra of the samples before and after irradiation. The irradiation was carried out using  $^{60}\text{Co}$  source (maximum power is about 1 krad/s). All  $\text{PbWO}_4$  crystals were sequentially irradiated with two doses: 460 krad and 7 Mrad. Transmission spectra measurements were performed just after irradiation. The results are plotted in figs. 10-14. From the analysis of the spectra one can conclude that with the increase of the irradiation dose the transmission drops down for yellow coloured samples 1.E, 3.E, and 1.6. Evidently the saturation of radiation damages has taken place because the difference in transmission for two doses is small. The behaviour of radiation damages of colourless crystals 3.8 and 3.85 is different. After the samples were irradiated by a low dose the decrease in transmission is about 5 %. After the high dose irradiation the transmission increases, for 3.85 sample more noticeably. In addition, the transmission spectra of all samples were measured within 2 weeks after high dose irradiation (7 Mrad). The self annealing of radiation damages is quite pronounced for all samples and the transmission is completely recovered for the sample 3.85.

## 8 Conclusion

The first results on X-ray phase analysis, transmission and X-ray luminescence spectra, light yield, decay time and radiation hardness of  $\text{PbWO}_4$  crystals grown in All-Russian Research Institute of Synthesis of Mineral Raw Material have been published. We plan improve in future crystal growth technology in order to increase light yield as well

as radiation hardness of  $\text{PbWO}_4$  crystals.

We consider it necessary to express our thanks to Dr. V.Makhov for fruitful discussions.

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## Figure captions

1. Transmission spectra of different  $\text{PbWO}_4$  crystals: 1 - colourless; 2 - slight yellow; 3 - yellow; 4 - bright orange-yellow.
2. Variation of optical absorption coefficient  $\mathbf{k}$  of a material along a crystal length  $\mathbf{L}$ : yellow coloured crystals 1 -  $\lambda = 423$  nm; 2 -  $\lambda = 500$  nm, colourless crystals 3 -  $\lambda = 423$  nm.
3. X-ray luminescence spectra of yellow  $\text{PbWO}_4$  crystals: 1 - head part of a crystal; 2 - middle part; 3 - rear part; 4 - rear part with inclusions
4. X-ray luminescence spectra of  $\text{PbWO}_4$  crystals grown using yellow modification of  $\text{PbO}$  raw material; 1 - oxidation atmosphere; 2,3 - neutral atmosphere; 4 - X-ray luminescence spectrum of BGO crystal.
5. X-ray luminescence spectra of  $\text{PbWO}_4$  crystals: 1 - bright orange-yellow colour (red  $\text{PbO}$  raw material); 2 - bright orange-yellow colour (red and yellow mixture of  $\text{PbO}$  raw material); 3 - yellow colour (yellow  $\text{PbO}$  raw material).
6. Dependence of position of maximum of X-ray luminescence spectrum on yellow colour intensity (absorption coefficient at  $\lambda = 423$  nm) of  $\text{PbWO}_4$  crystals.
7. Pulse height spectra of different  $\text{PbWO}_4$  crystals (10 mm in diameter, 10 mm long) obtained using  $^{137}\text{Cs}$  source : 1 - sample 1.6; 2 - sample 1.E; 3 - sample 3.E; 4 - sample 3.8; 5 - sample 3.85.



8. Comparison of pulse height spectra of  $\text{PbWO}_4$  and BGO crystals (10 mm in diameter, 10 mm long) obtained using  $^{137}\text{Cs}$  source : 1 -  $\text{PbWO}_4$  (sample 3.85); 2 - BGO (attenuation 16 dB).
9. Scheme of the setup for decay time measurements: 1 - YAP crystal; 2 - studying crystal; 3 -  $^{60}\text{Co}$  source; 4 - diaphragm; 5 - shield.
10. Transmission spectra of  $\text{PbWO}_4$  crystal (sample 3.8 - 10 mm in diameter, 10 mm long): 1 - before irradiation; 2 - after 460 krad ( $^{60}\text{Co}$  source); 3 - after 7.0 Mrad; 4 - self annealing after 2 weeks after irradiation
11. Transmission spectra of  $\text{PbWO}_4$  crystal (sample 3.85 - 10 mm in diameter, 10 mm long): 1 - before irradiation; 2 - after 460 krad ( $^{60}\text{Co}$  source); 3 - after 7.0 Mrad; 4 - self annealing after 2 weeks after irradiation.
12. Transmission spectra of  $\text{PbWO}_4$  crystal (sample 1.E - 10 mm in diameter, 10 mm long): 1 - before irradiation; 2 - after 460 krad ( $^{60}\text{Co}$  source); 3 - after 7.0 Mrad; 4 - self annealing after 2 weeks after irradiation
13. Transmission spectra of  $\text{PbWO}_4$  crystal (sample 3.E - 10 mm in diameter, 10 mm long): 1 - before irradiation; 2 - after 460 krad ( $^{60}\text{Co}$  source); 3 - after 7.0 Mrad; 4 - self annealing after 2 weeks after irradiation
14. Transmission spectra of  $\text{PbWO}_4$  crystal (sample 1.6 - 10 mm in diameter, 10 mm long): 1 - before irradiation; 2 - after 460 krad ( $^{60}\text{Co}$  source); 3 - after 7.0 Mrad; 4 - self annealing after 2 weeks after irradiation

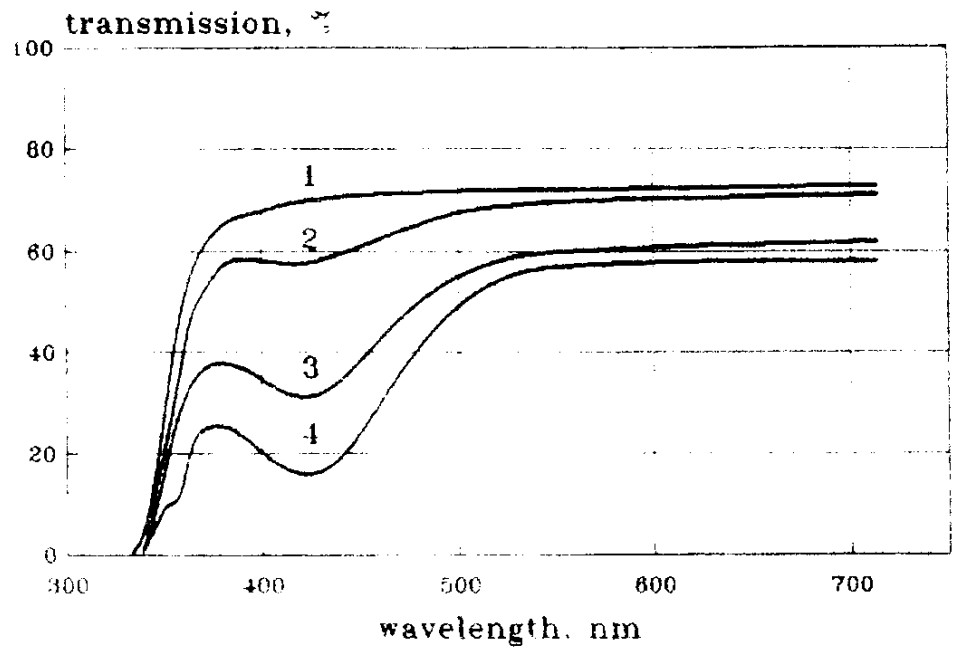


Fig. 1

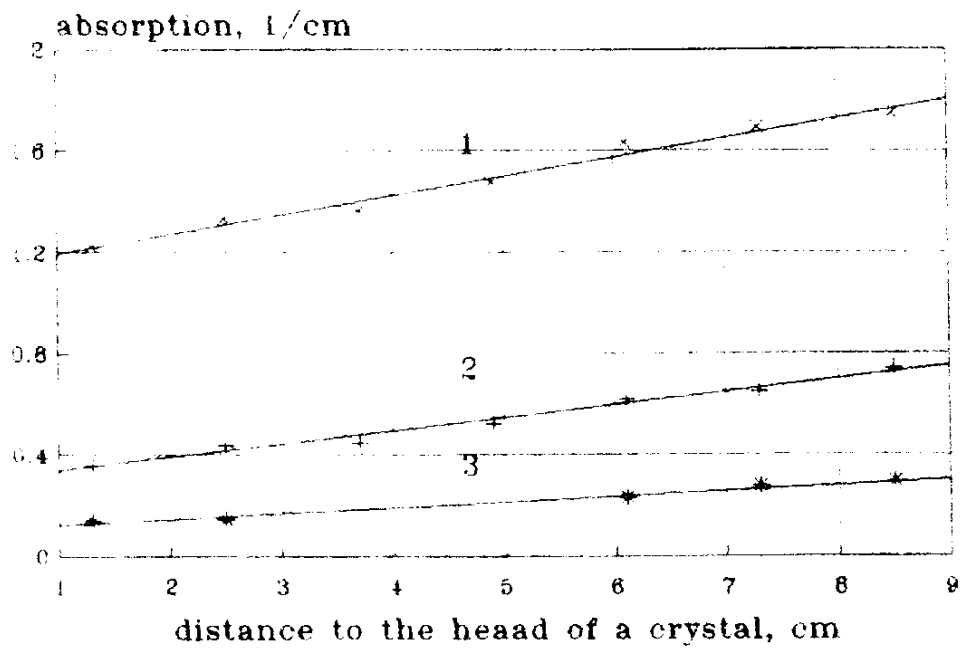


Fig. 2

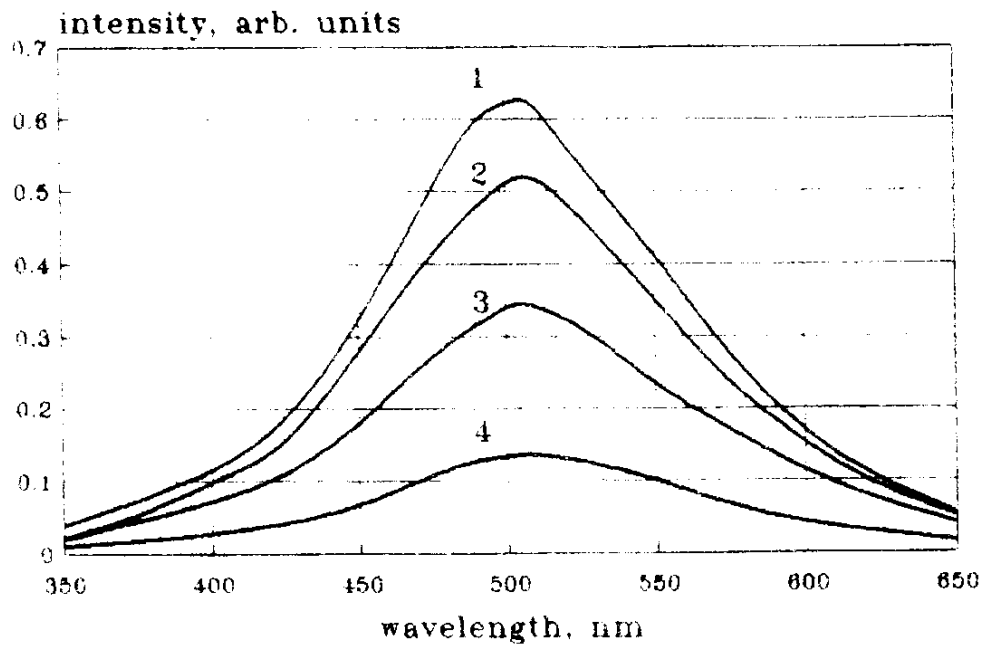


Fig. 3

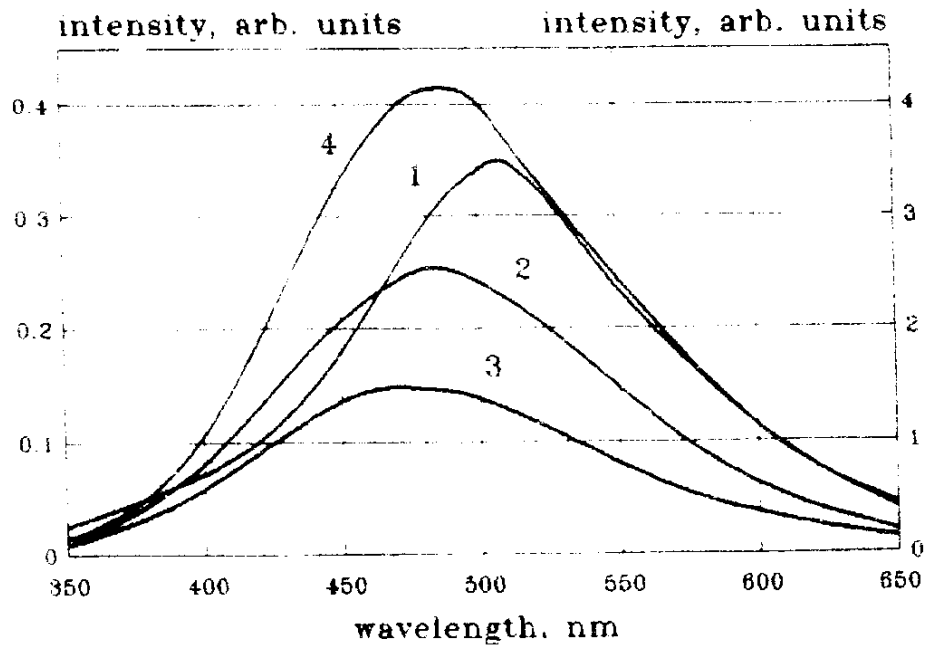


Fig. 4

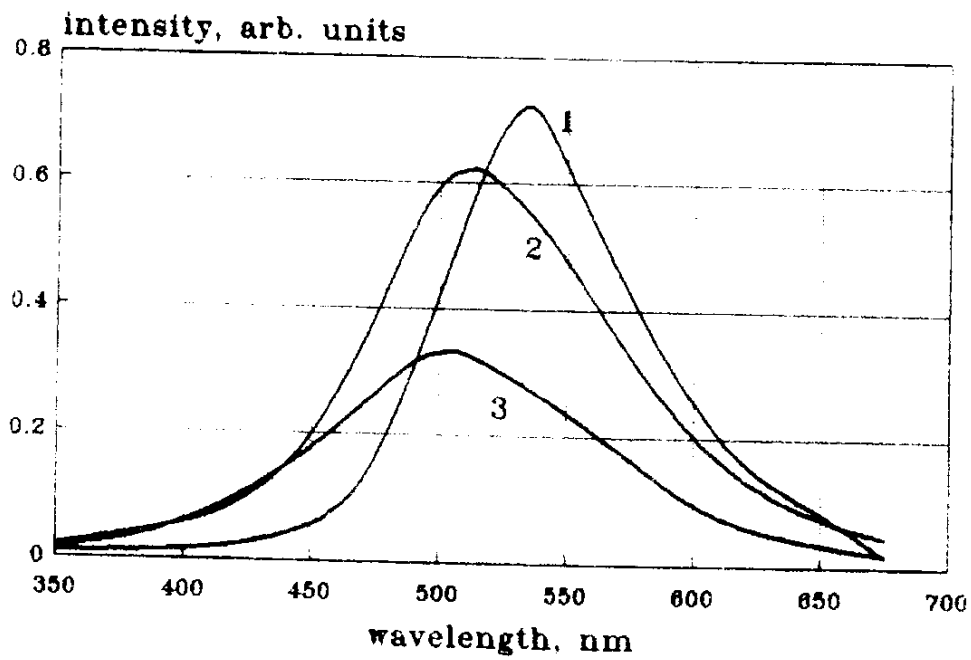


Fig. 5

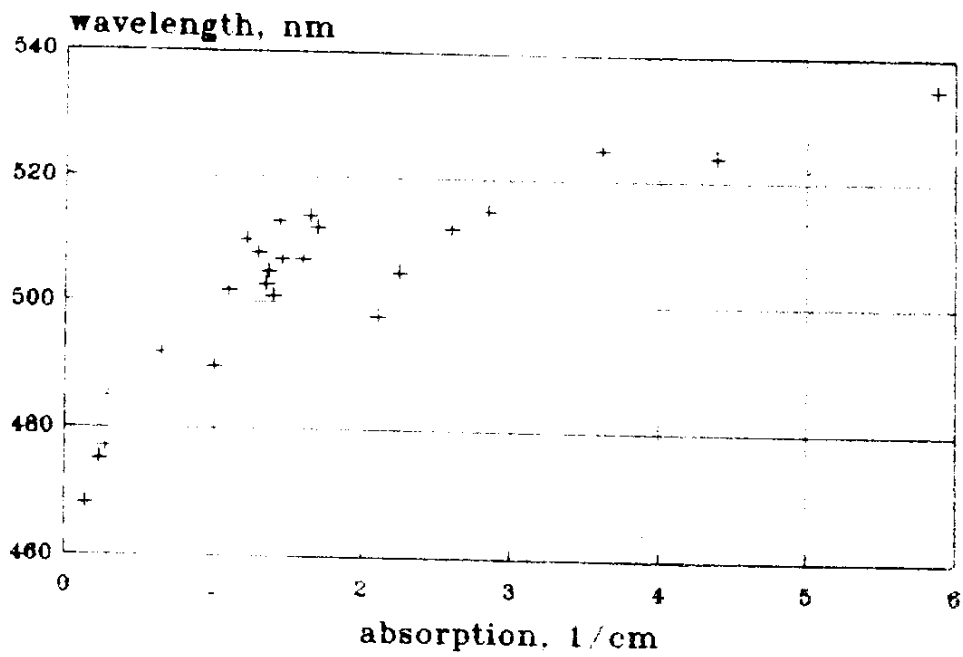


Fig. 6

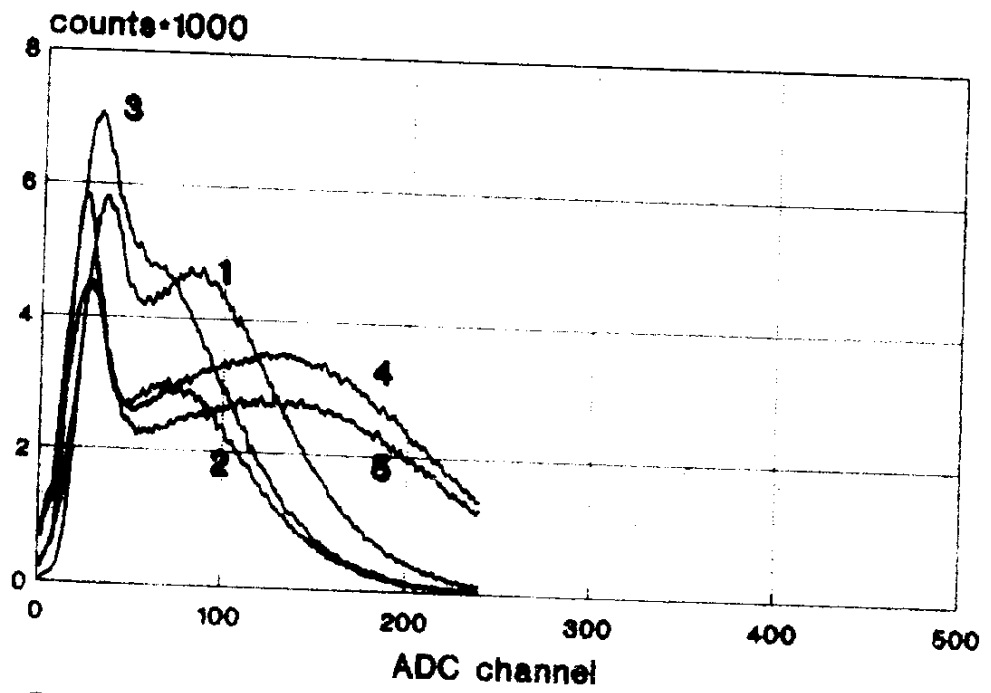


Fig. 7

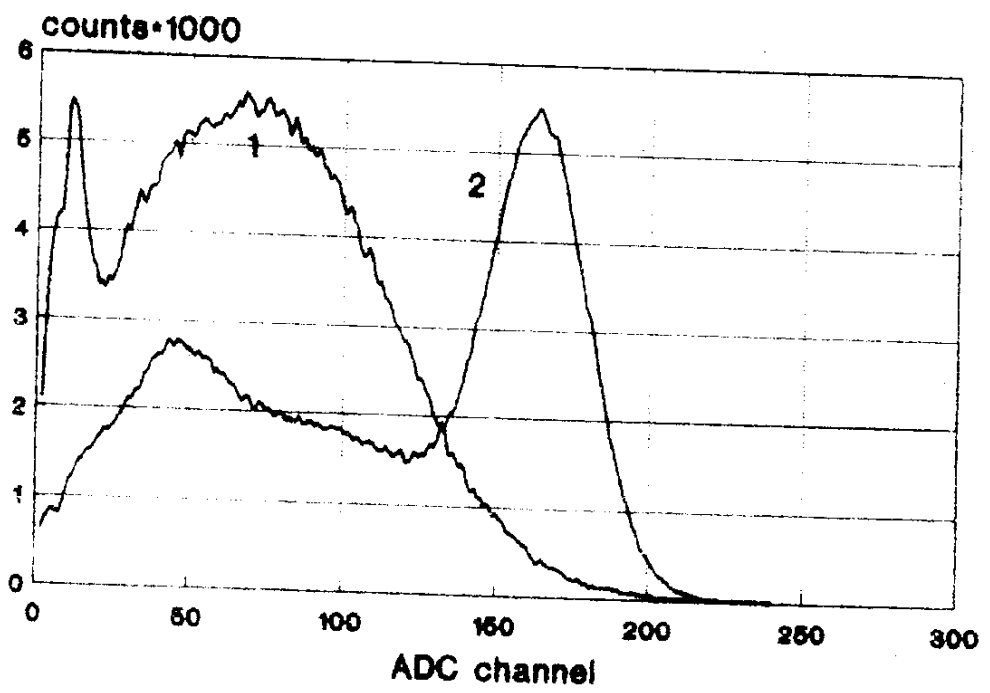


Fig. 8

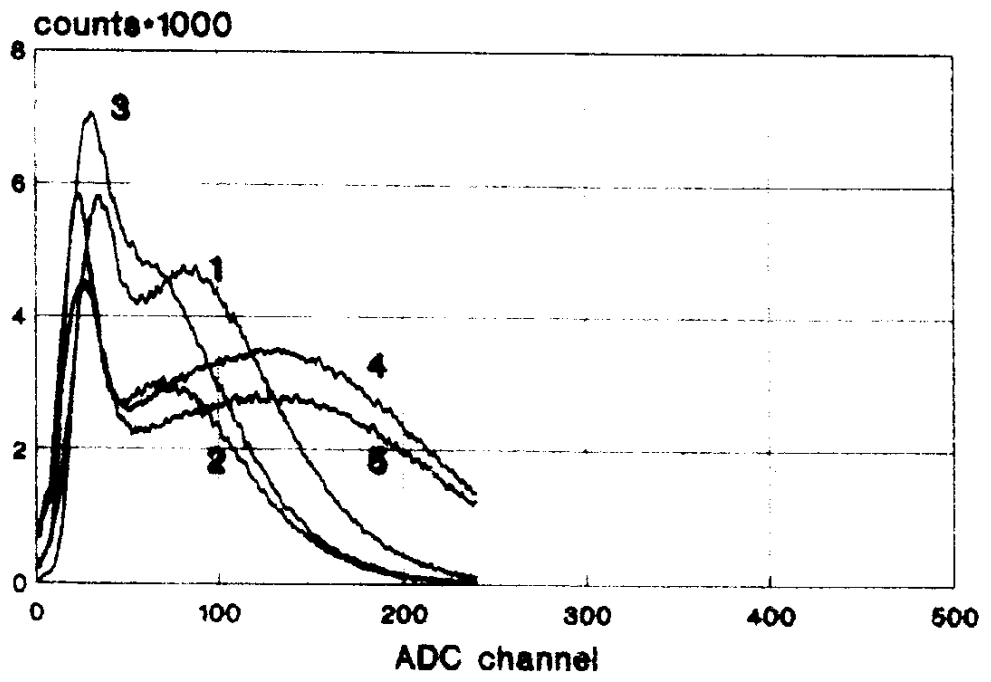


Fig. 7

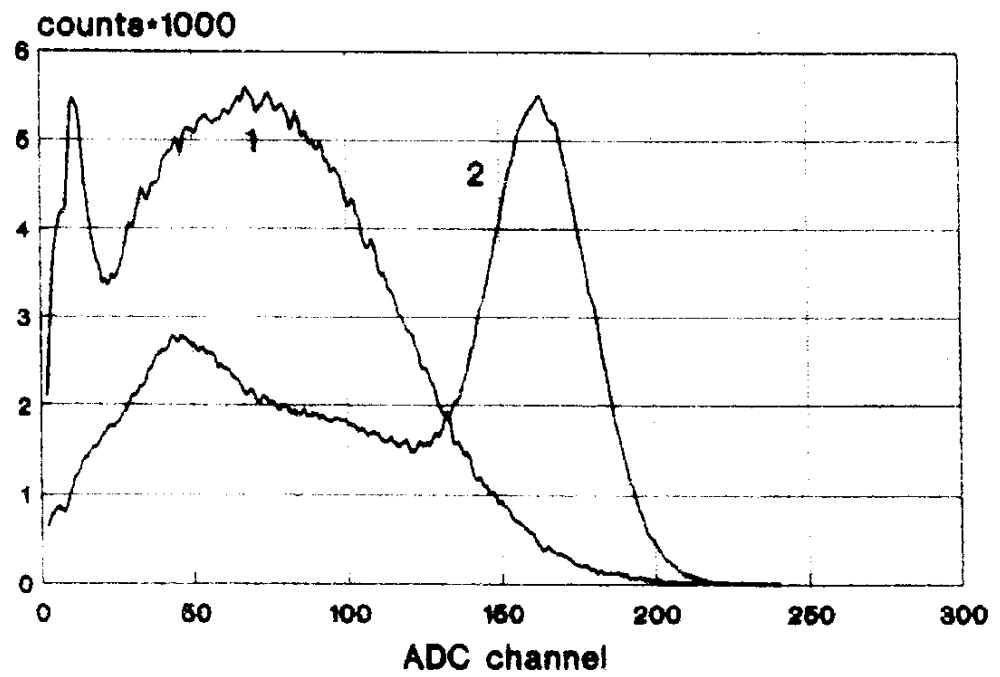
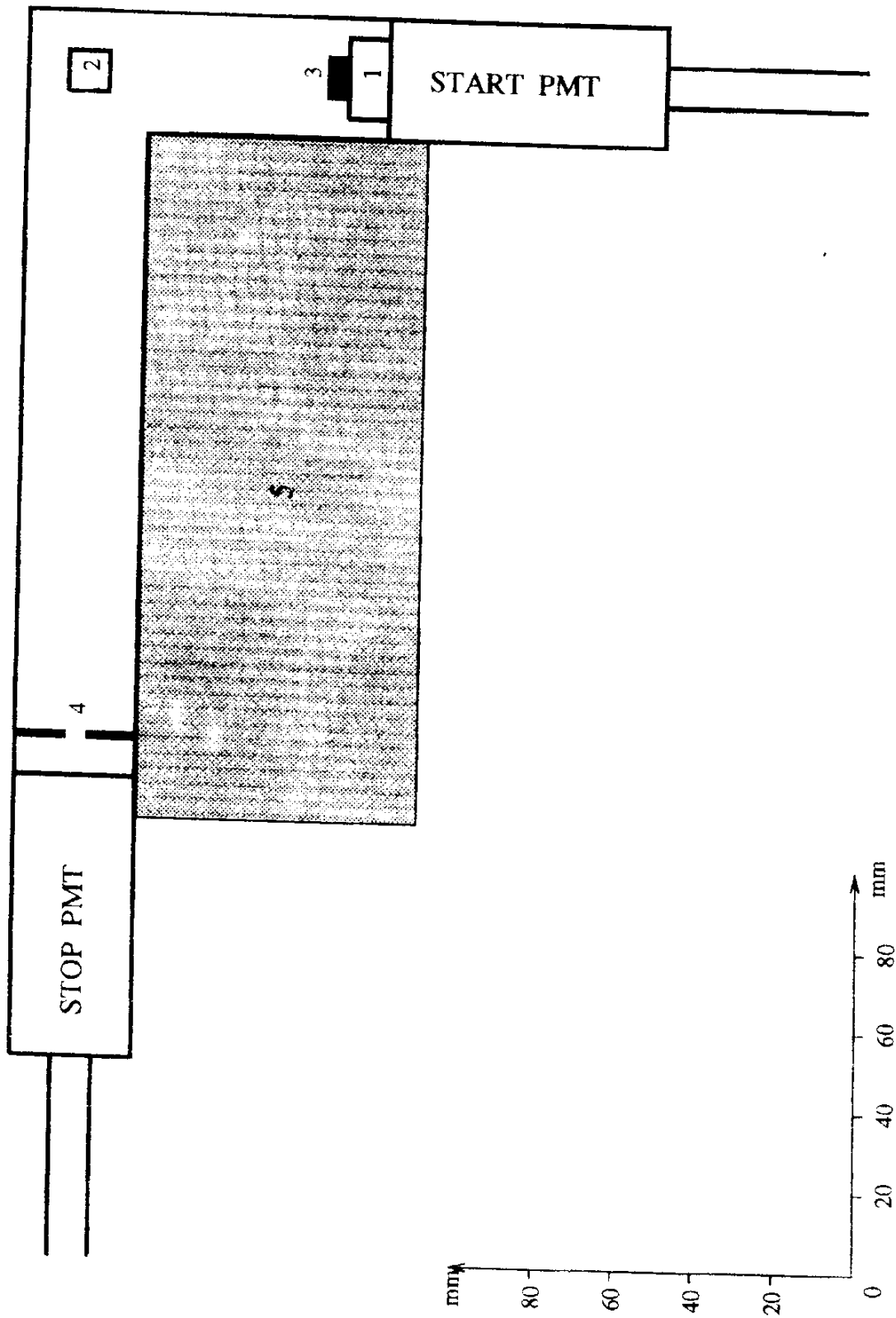


Fig. 8



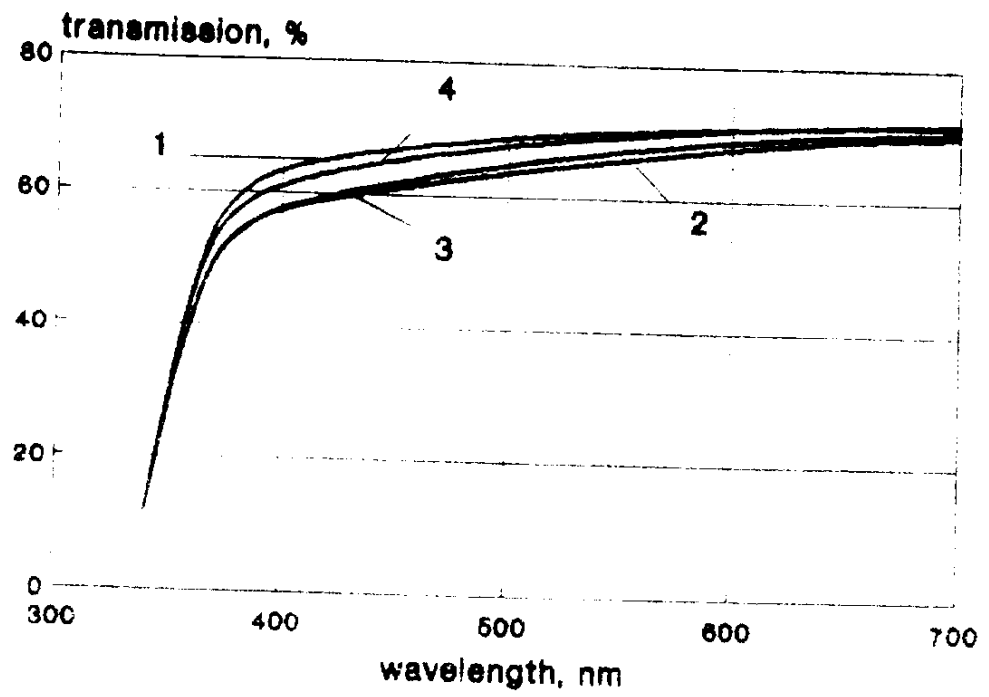


Fig. 10

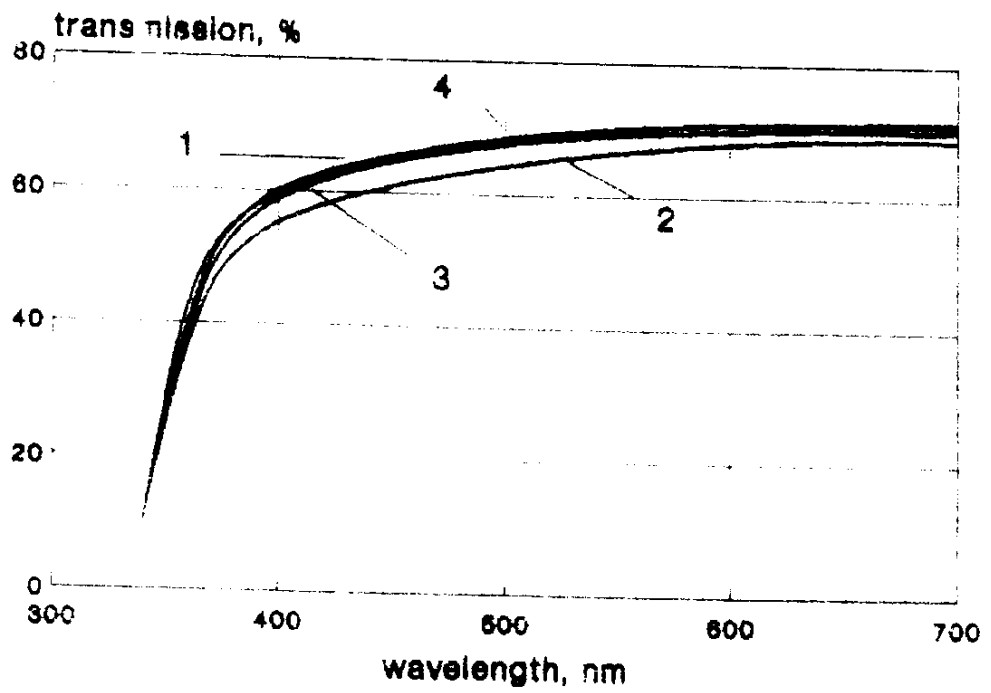


Fig. 11



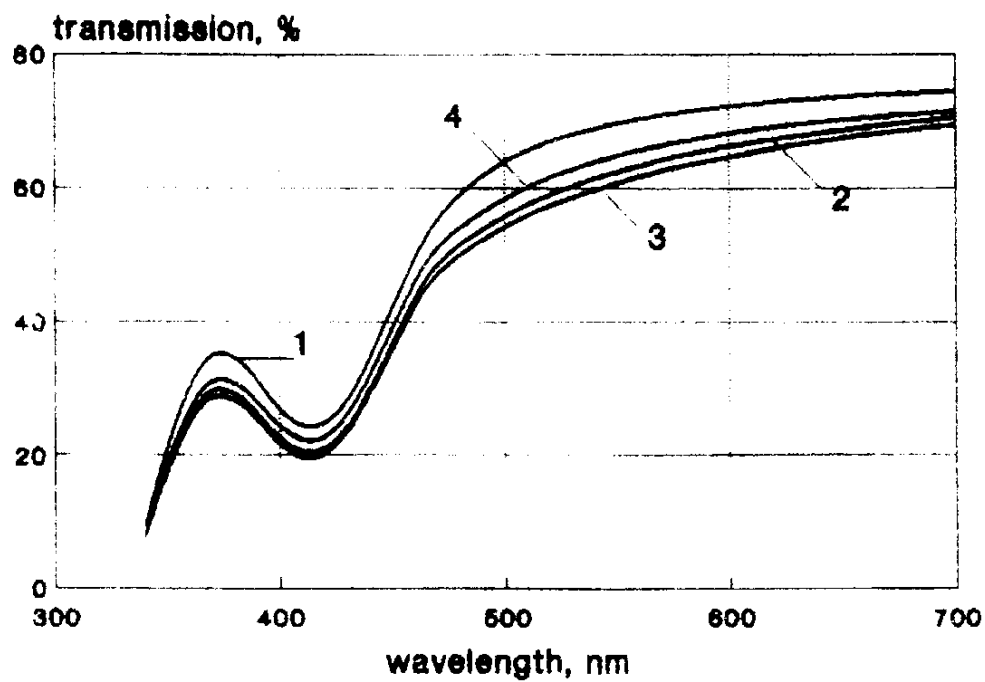


Fig. 12

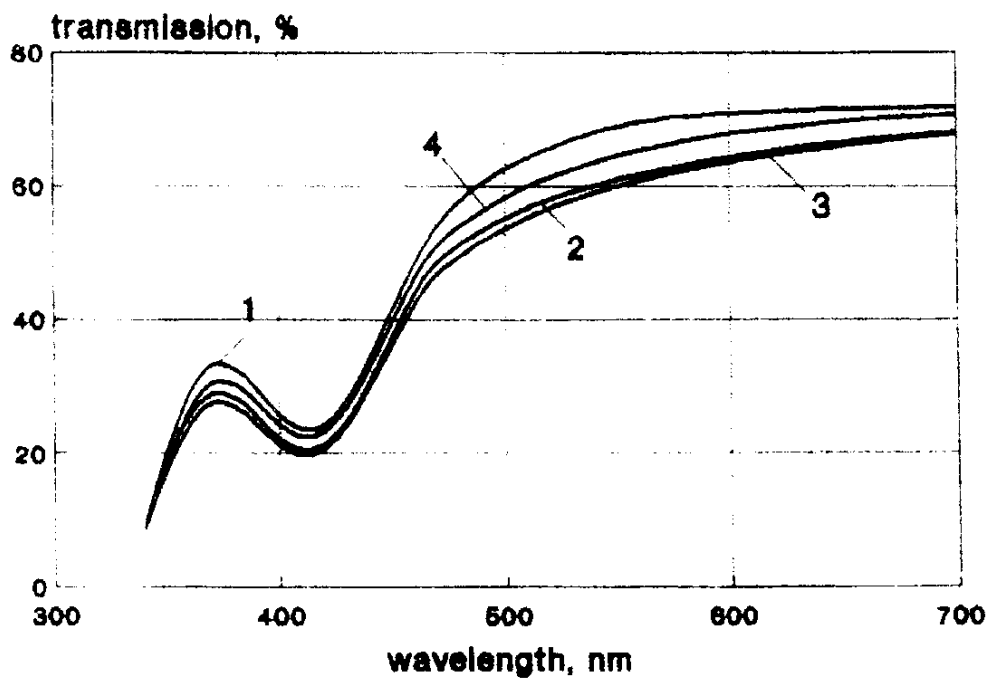


Fig. 13

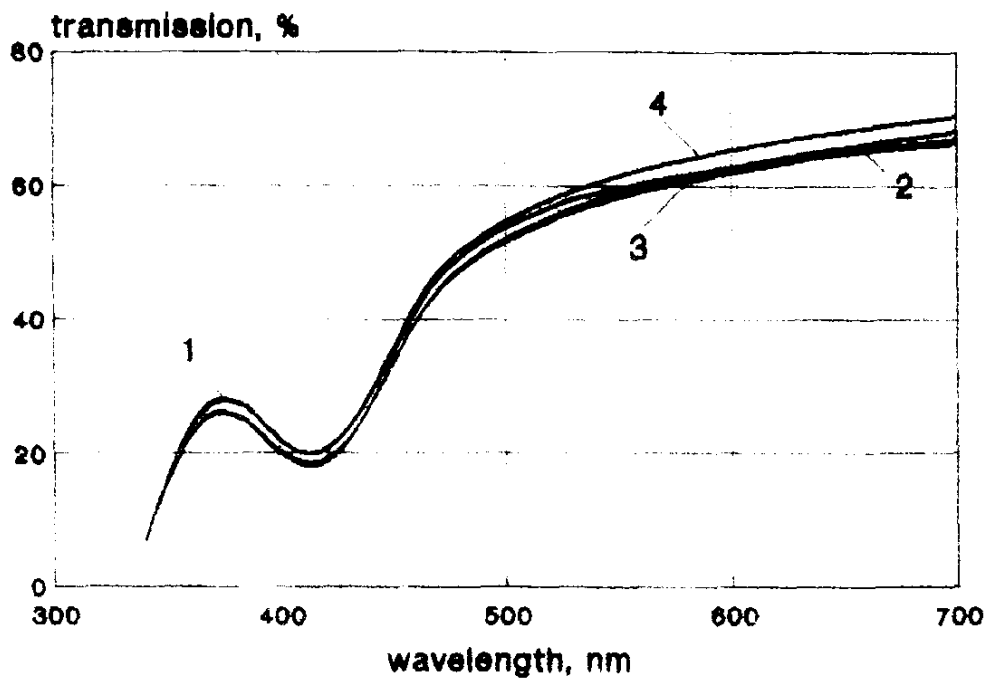


Fig. 14

Подписано в печать 26 октября 1995 г.

Заказ № 262. Тираж 60 экз. П.л. 1,6

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Отпечатано в РИИС ФИАН.

Москва, В-333, Ленинский проспект, 53