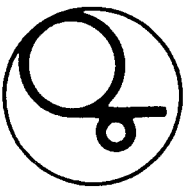


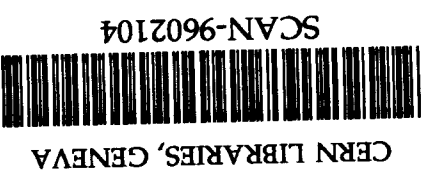
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Scintillation and Phosphorescence of PbWO₄ Crystals

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Abstract

There are two types of crystals with respect to luminescence. In dc measurement, Type A gives a primary emission at 410-430 nm (blue) with a second one at around 500 nm. Type B gives only dominant emission at 480-500 nm (green). This apparent difference can be interpreted as follows; the characteristic scintillation of PbWO₄ is dominantly blue and subdominantly green. Intense phosphorescence with a decay constant of about 15 ms exists only in Type B at about 500 nm with one to two orders of magnitude larger intensity than scintillation, hiding the blue scintillation in its tail. The cause of the phosphorescence may be Mo.

1. Introduction

In recent years, lead tungstate, PbWO₄, has attracted [1-4] much interest as a very dense, fast and possibly radiation-hard material for EM calorimeters at high energies. PbWO₄ is non-hygroscopic and has a density $d=8.2 \text{ g/cm}^3$; its unit radiation length $X_0=0.92 \text{ cm}$ is shorter than any of the well established scintillators[5] such as CdWO₄ ($X_0=1.06 \text{ cm}$), BGO (1.12 cm), Gd₂SiO₅:Ce (1.38 cm), etc. and even the heavy Cherenkov radiator PbF₂ (0.95 cm) which has been developed since several years ago[6]. Soon after the rediscovery of the scintillation of PbWO₄ at room temperature by Derenzo et al.[7], evaluation of the scintillation characteristics was made at KEK [1] and Russia [2]. Radiation hardness was also examined at KEK[1]. According to our meas-

urement[1], the light output is about 4-5% of that in BGO. The luminescence is fast with the decay constant less than 10 ns for 84% and as small as 40 ns for the rest. Degradation in optical transmittance due to γ -ray irradiation is not large up to at least 10^6 rad . Spontaneous recovery of the radiation damage, after irradiation by 10^9 rad , occurs to some extent with a time constant much shorter than 18 days. More extensive measurements [8,9] have been carried out since then, essentially confirming the initial results in [1,2]. Much efforts have also been devoted to improve the crystal growth, and now crystals with the length as long as $25 X_0$ can be grown successfully.

However, there remains an unsettled discrepancy in the peak emission wavelengths. Two different results have been reported[1-3,8-9]; one with the emission peak at $\lambda_{em}=420\text{-}440 \text{ nm}$ (blue) for the excitation at $\lambda_{ex}\sim 330 \text{ nm}$, and the other with $\lambda_{em}=490\text{-}500 \text{ nm}$ (green) for $\lambda_{ex}\sim 310 \text{ nm}$. This confusing result already appeared in the two first measurements at KEK[1] and Russia[2]. Korzhik et al.[8] gave a conjecture that the blue (green) emission is due to WO_4^{2+} (WO_3+F), pointing out a possibility that the green emission may be related to crystal defects on the surface (lack of oxygen). However, as described later, our measurement shows no significant effects of surface on the emission spectra.

In order to solve the above-mentioned discrepancy, we carried out a comparison study of photoluminescence, phosphorescence after UV-excitation, radioluminescence due to X-rays, scintillation due to γ -rays, decay constants, etc. for 15 crystal samples (10 from Furukawa, 5 from Russia[11]), which were grown at different times with different techniques or conditions as listed in Table 1. It is the aim of the present paper to describe the result of the above study and derive a reasonable solution[10] of the above problem. After the description on the preparation of PbWO₄ samples, we will present our result one by one in the order which may help one to reach our final conclusion to the problem mentioned above.

2. PbWO₄ Samples

All the PbWO₄ samples (see Table 1) were grown by the Czochralski method. The nominal purity of raw material was 99.99% except for No.3

(99.999%). No.2 and No.8 were obtained by twice and three times crystallization, respectively. No.4 and No.10[1] were grown by adding a small amount of Cl and Cs, respectively, although the dopants were undetectable in the grown crystals within the sensitivity of 0.3 atomic ppm. No.5-7 are typical Russian samples of the best quality at different times of 1993-1995. No.11 and No.12 are doped with Mo and Cl, respectively. The dopants were added into the melt in a large amount of percent levels. No.13-15 were doped with Nb. All the samples except No.12 have a rectangular shape; the thickness is given in Table 1. Most of them have a size of roughly $10 \times 10 \times (20-30)$ mm³. No.8 had a smaller size $7 \times 10 \times 10$ mm³, while No.12 a larger size $20 \times 20 \times 120$ mm³. No.12 was as small as 5-10 mm in size with an irregular shape and unpolished.

3. Transmission

Transmission spectra measured with a spectrophotometer (Hitachi 330) are shown in Fig. 1 for all the samples except No. 12, which was not polished. We cannot compare the absolute transmittance precisely but only the spectral shape among the samples since the surface quality of the samples was various. As seen in Fig. 1, the cutoff wavelength is approximately 320-340 nm. A slight absorption around 440 nm corresponds to the yellowish colour (the complementary colour to blue). The shape of the spectra is not much different among the samples. Although the 440 nm absorption was negligibly small for some samples (No.4, 5, 6 and 8), we did not find any correlation of it with the crystal growth (doping, multiple crystallization, raw material, etc.)

4. Photoluminescence

We measured photoluminescence using a fluorescence spectrophotometer (Hitachi F4500) in both the surface and the deep modes (see Fig. 2), in order to measure the luminescence from the surface and from deep inside, respectively. In the deep mode, the crystal was tilted three dimensionally so as to prevent the scattered excitation light from entering the detection slit of emission. The obtained spectra are given in Fig. 3 with the surface emission on the left and the deep emission on the right. We can see the existence of two groups which are largely different from each other. In almost all crystals

grown by Furukawa, the contour of the surface emission has a peak typically between $\lambda_{em}=420$ and 480 nm for λ_{ex} around 310 nm, extending towards lower λ_{em} (420-440 nm) and higher λ_{ex} (330nm). The deep emission, however, is usually peaked at $\lambda_{em}=420-440$ nm for $\lambda_{ex}=330$ nm. The above shift can be attributed to the cutoff of PbWO₄ at 320-340 nm. In most of crystals from Russia, however, the emission contour has a similar shape with the centre at $\lambda_{em}=480-500$ nm and $\lambda_{ex}=310$ nm for both the surface and the deep emissions (Table 1). We call the former Type A and the latter Type B throughout the present paper. This assignment is also given in Table 2. The light intensity can be roughly compared from the numbers attached to the contours (see Fig. 3). We can see that the crystals of Type B give one to two orders of magnitude larger light intensity than Type A. The intensity can be compared separately in the surface or the deep emission. Mixed comparison between both emissions is not correct since the absorption of the excitation light in the crystal can be large for the deep emission. In the deep mode, the excitation light has to penetrate a few mm deep inside the crystal to reach the optical axis of the emission slit. The excitation light is attenuated since the excitation wavelengths (330 nm for blue and 315 nm for green emission) are close to the cutoff wavelength (320-340 nm). The reduction of the deep emission than the surface emission by a factor of several to ten, as seen in Fig. 3, can be interpreted in terms of the attenuation of the excitation light mentioned above. Although the emission from the vicinity of the surface can also enter the emission slit, the acceptance is not large.

The above difference between Type A and Type B cannot be interpreted in terms of the crystal defects on the surface (lack of oxygen, for an example) suggested by Korzhik et al.[8]. We can see from Fig. 3 the similar large difference in intensity between Type A and Type B not only for the surface but also for the deep emission. The above difference essentially remained unchanged even if some of the Furukawa and Russian crystals were cut and polished in the same way by the same company.

Doping of Mo seems to drastically change the emission pattern from Type A to Type B. Among all the Furukawa crystals, only No.11 doped with Mo gives the emission pattern (Type B) similar to Russian samples (see Fig. 3).

Doping of Cl was somewhat an exception; No.12 doped with Cl shows an emission pattern intermediate between Type A and Type B. It belongs to Type B in that the peak emission occurs at green around 480 nm (surface emission) or 500 nm (deep emission), but the intensity is relatively small compared with the other Type B crystals. It does not emit significant phosphorescence either, which is characteristic to the Type B crystals as shown later in Section 6. We don't see any systematic change due to Nb doping. No.13 grown by Furukawa is similar to the other Furukawa undoped crystals. No.14 and No.15, both grown in Russia, are very much different from each other. No.14 resembles to the other Type B undoped crystals, while No.15 to Type A.

5 Scintillation due to γ -rays

In the previous section, we saw a big difference in the luminescence intensity between Type A and Type B when integrated over time. It is interesting to see if such a big difference should exist also in scintillation. We measured the scintillation light output of each crystal for γ -rays from radioactive isotopes (^{60}Co) by mounting the crystal on a 2-inch photomultiplier tube (PMT, Hamamatsu R329, bialkali photocathode) and using a PHA (Lecroy QVT) with a gate width of 1 ms. The 1.25 MeV peak, as an average of 1.17 and 1.33 MeV peaks, appeared on a background in the pulse height spectrum (see an example given in Fig. 4). The relative light intensity can be obtained from the position of the 1.25 MeV peak. The obtained result is given in Table 2. Although the relative light output scatters from 39 to 144 depending on the samples, the minimum of 39 in the large sample No.15 needs a correction to compensate the smaller efficiency of light collection when compared with all the other much smaller samples. The light intensity is then almost the same within a factor of two for all the samples (see Table 2). The number of photoelectrons can be approximately estimated from the width of the 1.25 MeV peak. For an example, from the rms width of 20%(or 26%) for No.8 (No.3) sample, the number of photoelectrons was roughly 20 pe/MeV (12 pe/MeV). To make a comparison with BGO, the PbWO_4 crystal was replaced by a BGO crystal with a similar size $1\times 1\times 3\text{ cm}^3$. If the samples of intermediate light intensity (~ 100 as in No.7 or No.11) are taken, the light output of PbWO_4 corresponds to about 5% of BGO. The above result means that all the crystals are similar in scintillation (fast luminescence) intensity. In order to reconcile

the above result with the result given in the previous section (one to two orders of magnitude larger intensity for Type B than for Type A in the dc measurement), a natural conclusion is that Type B crystals emit much slower emission in addition to the fast one.

We carried out measurement of the decay constant within 1 ms for all the samples using the conventional single photoelectron technique and a setup described in [1]. The start pulse for the time measurement was taken from a small GSO:Ce scintillator mounted on a 2-inch PMT (R329) for a γ -ray emitting isotope (^{60}Co). The stop pulse was the single photoelectron signal of PbWO_4 mounted on another fast 2-inch PMT (Philips XP2020, bialkali). The obtained decay time spectrum similar to that given in [1] was fitted with three exponentials with the time constants τ_1 , τ_2 and τ_3 , respectively, plus a constant background. The fitted result is given in Table 2. No sizable difference was found among all the Furukawa and Russian crystals. The decay constant was smaller than typically 15 ns for more than 80% of the intensity with the remaining part as fast as or faster than several tens ns. This result indicates that the slow component in Type B samples as concluded above should be by at least orders of magnitude slower than ms. It must be of the order of ms or slower.

6. Phosphorescence for UV-excitation

We measured the phosphorescence (excitation-emission spectra and decay constant) for all the samples using the F4500 spectrophotometer. Measurement was started at 2 ms after stopping the excitation. The result confirms the conjecture given in the previous section. As seen in Table 2, phosphorescence was significant in most of the Russian samples No.5,6,7 and 14 and one Furukawa sample (No.11) doped with Mo. All these crystals belong to Type B. Phosphorescence spectra are given in Fig. 5. No sizable phosphorescence was observed in any Type A samples. However, No.3, grown from the raw material of 5N (99.999%) purity, showed weak phosphorescence only in the surface emission but not in the deep emission (see Fig. 5). The decay constant was approximately 15 ms (see Fig. 6). Two samples, No.7 and No.14, have an additional slower component of 81 ms and 127 ms, respectively (see also Table 2).

7. Radioluminescence due to X-rays

We also measured the radioluminescence due to X-rays (50kV, 30A, Cu). The spectral dependence of the quantum efficiency of PMT (Hamamatsu R329) was corrected for. Normalized emission spectra are given in Fig. 7 for all the samples. The peak emission wavelength and the relative intensity are given in Table 2; they are consistent with the result of deep emission obtained for UV-excitation. Since the luminescence due to X-rays is expected to be similar to that due to γ -rays, the above result indicates the similarity of the luminescences for different excitation sources of UV-light, X-rays and γ -rays. If we compare both the deep and the surface emissions for UV-excitation (see Fig. 3) with the radioluminescence, the deep emission has better agreement with the radioluminescence with respect to the emission peaks. This indicates that the deep emission is closer to the actual emission measured in actual detectors rather than the surface emission.

8. Overall Scenario for Luminescence

The possible scenario which can explain all the above experimental results will be as follows;

(i) All the Type B crystals (most of Russian samples and one Furukawa sample No.11), except No.12 doped with Cl, emit phosphorescence with the decay constant of about 15 ms at around 500 nm (green) with the intensity by one to two orders of magnitude larger than scintillation.

(ii) Type A crystals have no sizable phosphorescence. The primary emission at around 420-440 nm (blue) and a secondary one around 500 nm (green) must be the characteristic scintillation of PbWO_4 .

(iii) Similarity of the scintillation (intensity and decay constant) between Type A and Type B crystals suggests that Type B samples should also have the similar characteristic scintillation as Type A (dominantly blue and subdominantly green). The scintillation in Type B must be hidden behind the tail of the intense phosphorescence if measurement is carried out in the dc mode.

9. Summary and Discussions

We summarize the above as follows;

(1) We carried out a comparison study of photoluminescence, phospho-

rescence after UV-excitation, radioluminescence due to X-rays, scintillation due to γ -rays, decay constants, etc. for 15 different crystal samples.

(2) There are two types of crystals with respect to luminescence. In dc measurement, Type A gives a primary emission at 420-430 nm (blue) with a second one at around 500 nm. Type B gives only dominant emission at 490-500 nm (green) with an intensity of one to two orders of magnitude larger than scintillation. The scintillation intensity, however, is almost similar in all the crystals.

(3) The characteristic scintillation of PbWO_4 is dominantly blue and subdominantly green. Intense green phosphorescence with a decay constant of about 15 ms exists only in Type B at about 500 nm with one to two orders of magnitude larger intensity than scintillation, hiding the blue scintillation in the short wavelength tail.

It is well known [12] that at low temperatures PbWO_4 have two emissions, blue and green. As the temperature increases, both emissions begin to quench at around 150°K, and are almost entirely quenched at room temperature. It is possible[12] that blue emission is less quenched at room temperature than green one in consistency with the observed dominance of blue emission.

The phosphorescence in Type B crystals could be tolerated in actual EM calorimeters at high energy as far as the pulse rate is small. The decay constant (about 15 ms) is longer than scintillation by 10^6 . Even if the integrated intensity of phosphorescence is two orders of magnitude larger than scintillation, the amplitude of phosphorescence is as small as 0.01% of the scintillation. However, the phosphorescence can accumulate and become a problem if the signal rate is as large as kHz or larger.

The cause of the phosphorescence in Type B crystals may be Mo contamination in PbWO_4 . There exist a few facts supporting this hypothesis. Among ten samples from Furukawa, only one sample, No.11, doped with Mo belongs to Type B in all the relevant properties such as the dominant green emission and very intense light output in the dc measurement, phosphorescence wavelength, intensity and decay constant, etc. Moreover, GDMS (Glow Discharge Mass Spectroscopy) analysis of the grown crystals made for several

among total 15 samples revealed the existence of a significant amount of Mo in Type B samples. All the analyzed Russian samples (No.5, No.7, No.14, all belonging to Type B) contain 14 to 25 atomic ppm of Mo, while all the Furukawa samples (No.1, No.10, No.12, all belonging to Type A) did not contain Mo within the sensitivity of 0.27 to 3.8 atomic ppm depending on the samples. However, the above conjecture does not mean that PbMoO_4 should be the cause of the phosphorescence. It is known[12,13] that PbMoO_4 does not give intense luminescence at room temperature. We also confirmed this from our own measurement of a PbMoO_4 crystal[11]. Although the traces of all the elements from Li(Z=3) to Bi(Z=83) were measured in GDMS, there was no systematic difference between Type A and Type B samples except the Mo amount. If the above hypothesis on the role of Mo should be correct, it follows that we detected the effects of Mo in the occurrence of intense phosphorescence. Contamination of Mo has to be minimized for fast operation of PbWO_4 calorimeters. Practically, Mo sometimes coexists with W; its chemical or physical separation is not necessarily a simple job. In this sense, absence of Mo in a group of PbWO_4 crystals (grown by Furukawa) is remarkable.

The effect of Cl contamination is somewhat ambiguous. We cannot completely exclude a possibility that Cl may have some relation to the luminescence type (A or B), although the correlation between the amount of Cl and the type of luminescence is less clear compared with the case of Mo. Among Type B samples, GDMS data were taken for 4 samples (No.5,7,12 and 14). No.7 and No.14 contain large amount of Cl, 28 and 66 atomic ppm, respectively, but No.5 only 1.3 ppm. No.12 doped with Cl contains a large amount of Cl (24 atomic ppm) but emits no or only weak phosphorescence. Type A crystals, No.1 and 10 for an example, contain a few ppm of Cl. Further studies are necessary before deriving any conclusions on the effect of Cl.

After completion of the present work, the authors noticed a few papers[14-16] (submitted to SCINT95 International Conference) which are related to the present problem (discrepancy of the emission wavelength of PbWO_4). Ref.[14] describes the very slow green emission and attributes the

fast component to mainly blue emissions in consistency with the result of the present work. Ref.[15] reports that annealing in vacuum enhances blue emission relative to green one, in contradiction to the conjecture of Korzhik et al.[8] on the role of surface (lack of oxygen) on the green emission. Ref.[16] proposes a hypothesis to attribute the green luminescence to a monoclinic structure which may contaminate into the main tetragonal structure of PbWO_4 .

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Figure Captions

- Fig. 1 Transmission spectra across the thickness (roughly 1 cm for most of samples) given in Table 1.
- Fig. 2 Sketch of two configurations in luminescence measurement, the surface and the deep modes.
- Fig. 3 Excitation-emission spectra for UV excitation. The numbers attached to the contour give rough intensity; it can be compared within the surface or the deep emissions separately. Comparison between the two modes is not correct due to absorption of light in the deep mode (see the text).
- Fig. 4 An example of γ -ray spectrum for a small sample No.8 (7x10x10 mm³). The FWHM resolution of 47% corresponds to the light output of about 20 pe/MeV.
- Fig. 5 Phosphorescence spectra for the surface and the deep emissions. Phosphorescence was undetectable in the other samples either in the surface or the deep emission.
- Fig. 6 Decay time spectra of phosphorescence for the surface emission. Measurement was carried out at λ_{ex} =320 and λ_{em} =490 nm, which is approximately the excitation and the emission peak of all the crystals that emit phosphorescence.
- Fig. 7 Radioluminescence spectra for X-rays (50kV, 30A on Cu target).

Table 1 List of PbWO₄ samples tested. The quality of surface polishing is various.

Sample	Thickness	Colour	Mfg. Co.	Remarks
No.1	10 mm	slightly yellow	Furukawa	
No.2	10 mm	" "	"	Twice crystallized
No.3	10 mm	" "	"	5N material
No.4	10 mm	" "	"	Cl-added
No.5	10 mm	colourless	Russia	
No.6	10 mm	slightly whitish	"	
No.7	20 mm	slightly yellow	"	
No.8	7 mm	" "	Furukawa	3-times crystallized
No.9	10 mm	" "	"	WO ₃ of 2-nd company
No.10	10 mm	" "	"	Cs-added
No.11	10 mm	" "	"	Mo-doped
No.12	~5 mm	" "	"	Cl-doped, unpolished
No.13	10 mm	slightly yellow	Furukawa	Nb-doped
No.14	10 mm	" "	Russia	"
No.15	20 mm	" "	"	"

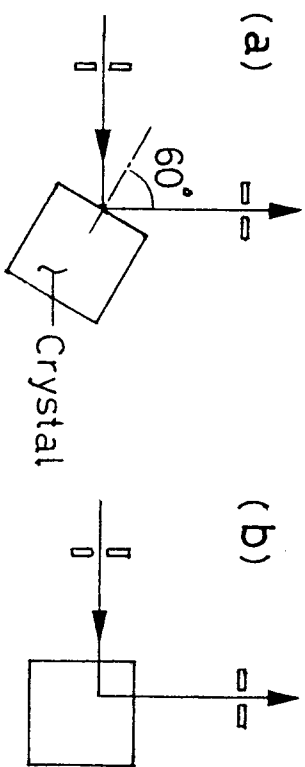
Table 2 Comparison of PbWO₄ samples with respect to photoluminescence, phosphorescence, radioluminescence and scintillation (intensity and decay constant). Dotted lines (---) indicates absence of phosphorescence peaks. Wavelengths in parentheses give second peaks. Intensities are relative and can be compared only within each category (separately for deep emission, surface emission, etc.; see the text). Type A or B (see the text) of luminescence is also given.

(footnote)

*) The second peak in the parenthesis is comparable with the first peak at $\lambda_{ex}=328$ nm, $\lambda_{em}=425$ nm at the top of the boule but almost disappears at the bottom.

+) Since No.15 is large, this number has to be roughly doubled in order to compare with the other much smaller samples (see the text).

	No.1	No.2	No.3	No.4	No.5	No.6	No.7	No.8	No.9	No.10	No.11	No.12	No.13	No.14	No.15
<u>Photoluminescence including phosphorescence</u>															
Deep emission															
λ_{ex} (nm)	328	333	331	330(310)	303	313	335(310)	328	330	330	310	320	328	322	328(305)*
λ_{em} (nm)	425	425	425	420(500)	498	490	490	425	425	425	510	500	415	490	425(520)*
Intensity	16	16	14	8	90	300	320	5	5.5	6.5	120	65	7	420	7
Surface emission															
λ_{ex} (nm)	311	311	311	310	308	313	312	315	315	310	310	310	312	312	302
λ_{em} (nm)	470	440	490	470	494	492	490	470	415	450	490	480	418	490	460
Intensity	75	60	200	90	630	4800	2500	70	30	32	1700	250	45	4000	45
<u>Phosphorescence after UV-excitation</u>															
Deep emission															
λ_{ex} (nm)	---	---	---	---	310	310	320	---	---	---	310	---	---	320	---
λ_{em} (nm)	---	---	---	---	490	490	490	---	---	---	500	---	---	490	---
Intensity	<0.2	<0.1	<0.2	<0.2	0.8	1.2	2.5	<0.2	<0.2	<0.2	0.8	<0.4	<0.1	18	<0.1
Surface emission															
λ_{ex} (nm)	---	---	315	---	310	310	315	---	---	---	310	---	---	320	---
λ_{em} (nm)	---	---	500	---	490	490	490	---	---	---	490	---	---	490	---
Intensity	<0.2	<0.2	4.1	<0.2	3.6	32	17	<0.2	<0.4	<0.2	7.5	<0.2	<0.2	135	<0.2
Decay τ (ms)	---	---	15	---	15	15	11(40%) 81(60%)	---	---	---	17	---	---	10(11%) 127(89%)	---
<u>Radioluminescence due to X-rays</u>															
λ_{em} (nm)	430 (505)	435 (510)	430 (505)	430 (510)	500 (450)	505	500	440	430 (520)	430 (490)	510	505 (440)	425 (510)	505	435 (510)
Intensity	40	60	70	81	110	140	260	108	49	40	228	220	44	1250	32
<u>Scintillation for γ-rays</u>															
Decay constants															
τ_1 (ns)	3.3(43%)	3.8(43%)	2.8(26%)	5.2(61%)	4.4(65%)	3.7(50%)	3.5(59%)	3.1(59%)	2.3(60%)	2.5(41%)	2.7(41%)	1.2(52%)	7.6(80%)	2.7(41%)	4.1(50%)
τ_2 (ns)	14.5(46%)	15.5(42%)	12.3(50%)	17.7(31%)	17.1(25%)	15.5(34%)	11.6(33%)	12.8(34%)	8.4(32%)	9.6(39%)	10.6(41%)	6.3(38%)	28.6(14%)	10.4(45%)	12.4(42%)
τ_3 (ns)	62.1(11%)	84.3(15%)	52.4(24%)	65.4(8%)	77.0(10%)	68.0(16%)	53.9(8%)	106(7%)	32(8%)	28(20%)	70.7(17%)	51.5(10%)	126(6%)	67.3(14%)	79.2(8%)
Intensity	74	70	83	70	88	121	102	132	85	71	100	143	71	144	39*
Type	A	A	A	A	B	B	B	A	A	A	B	B	A	B	A



Surface Emission Deep Emission

Fig. 2

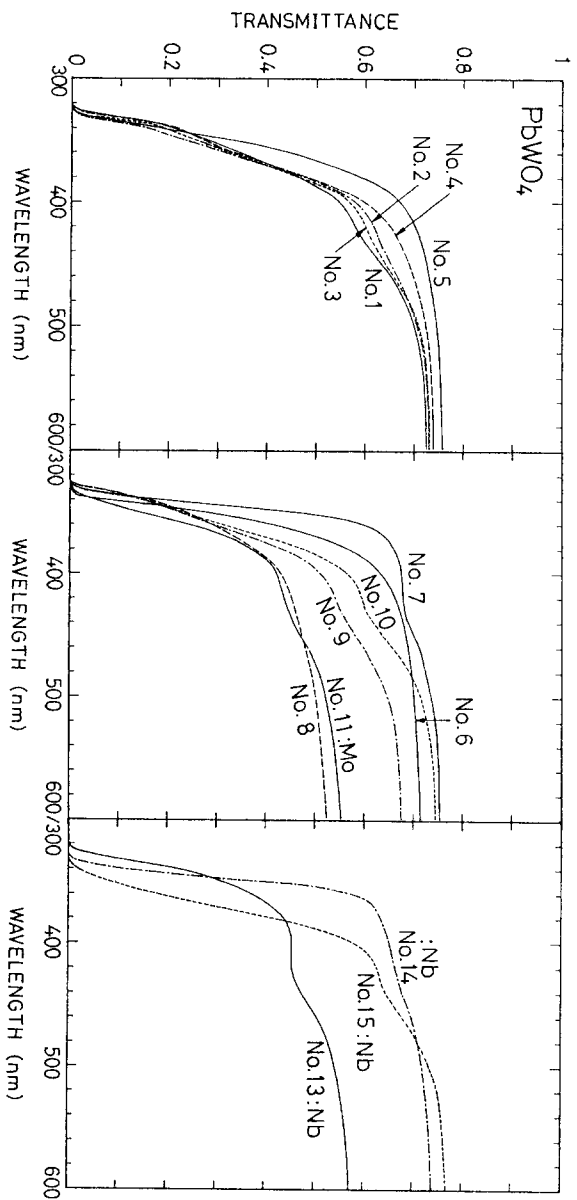


Fig. 1

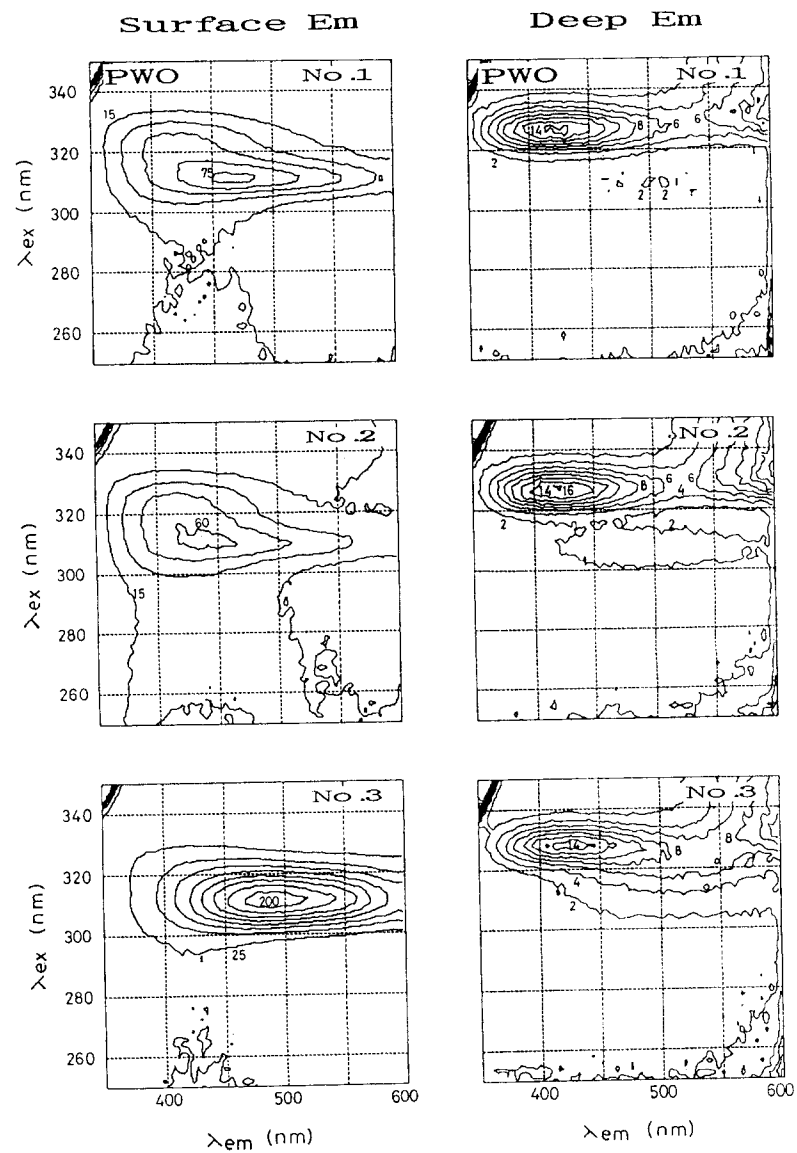


Fig. 3a

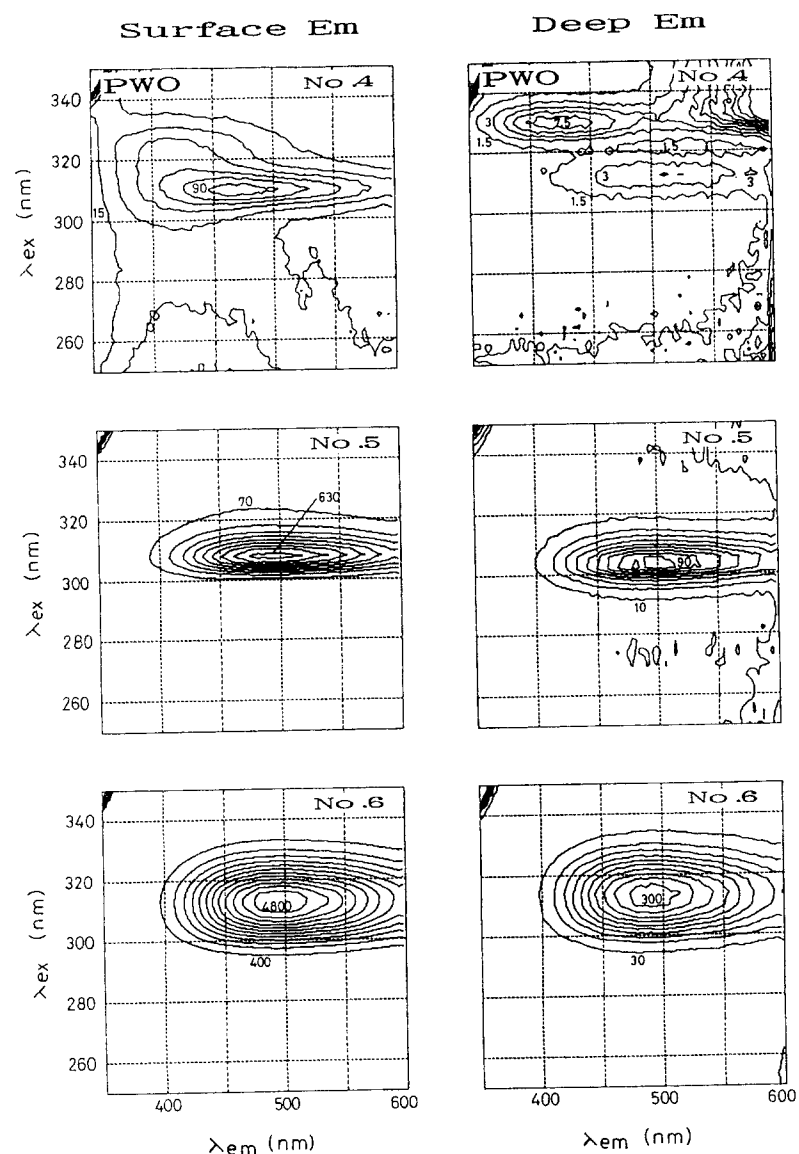


Fig. 3b

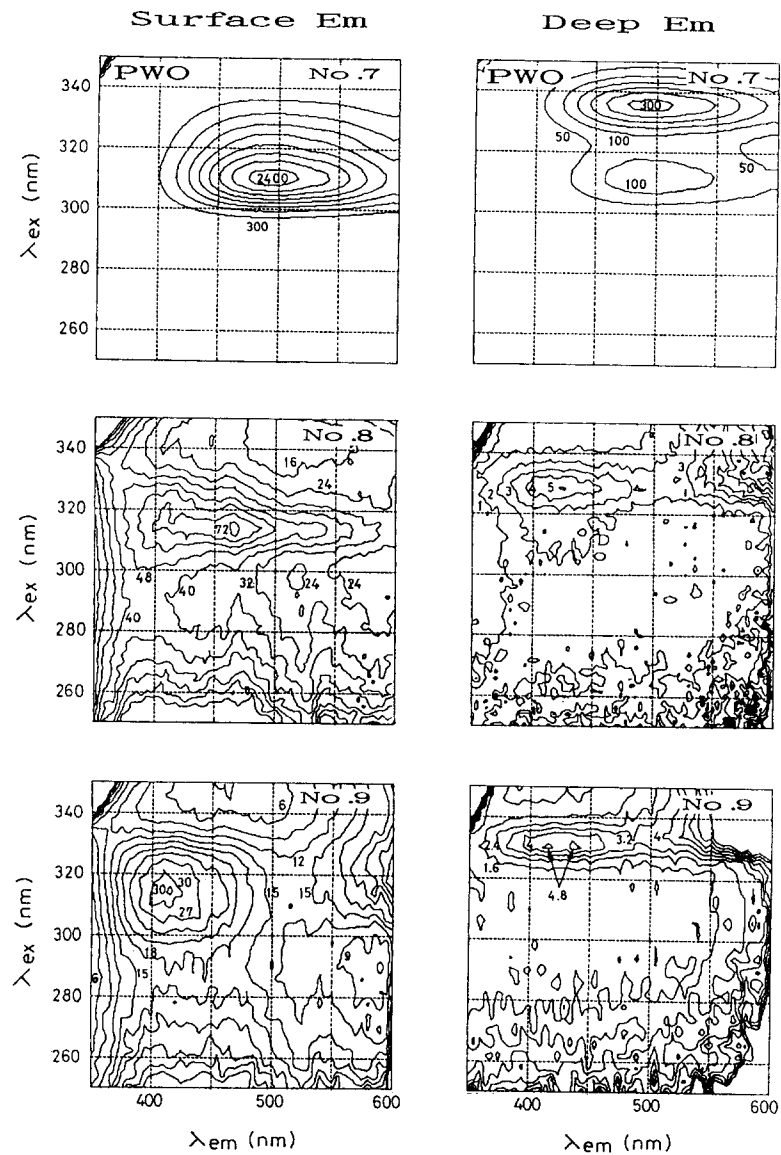


Fig. 3c

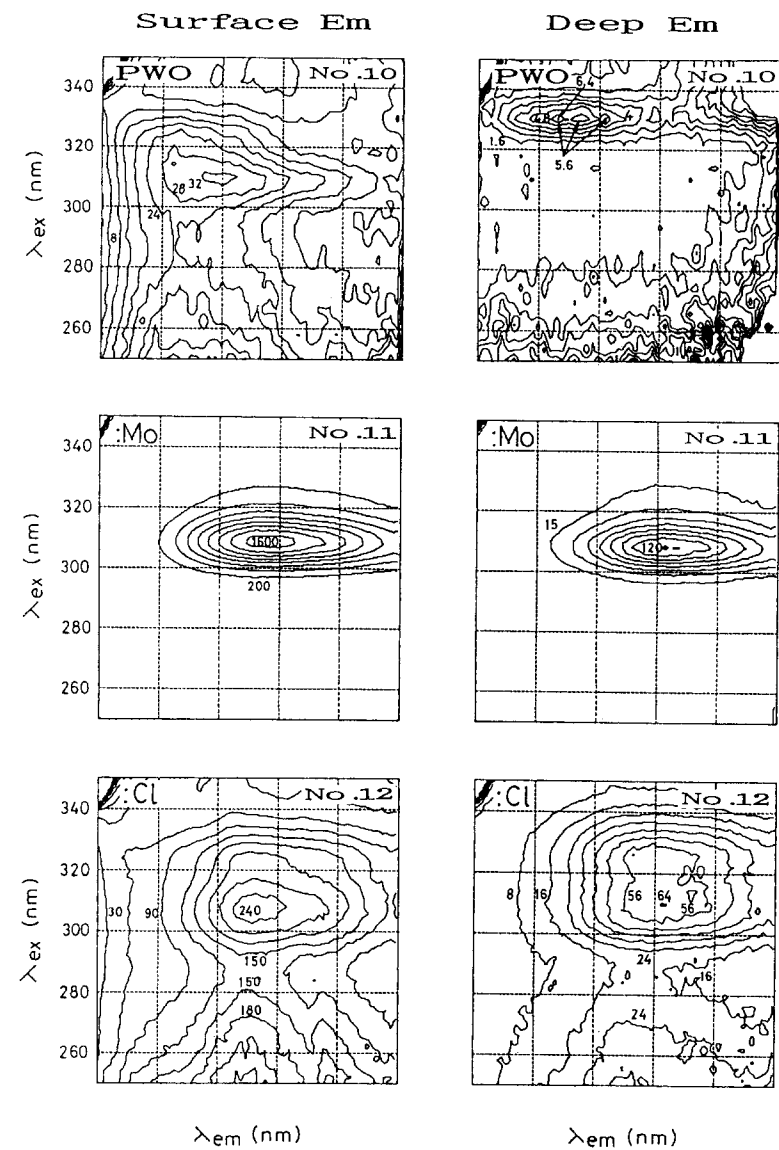


Fig. 3d

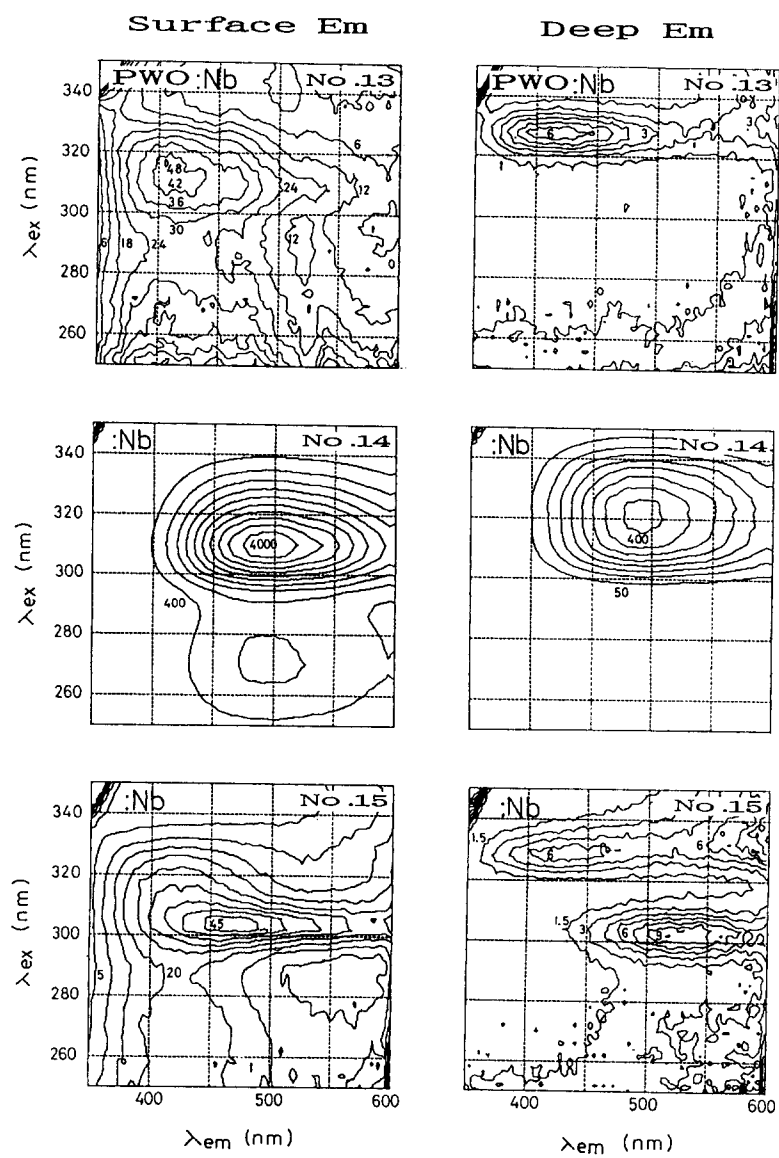


Fig. 3e

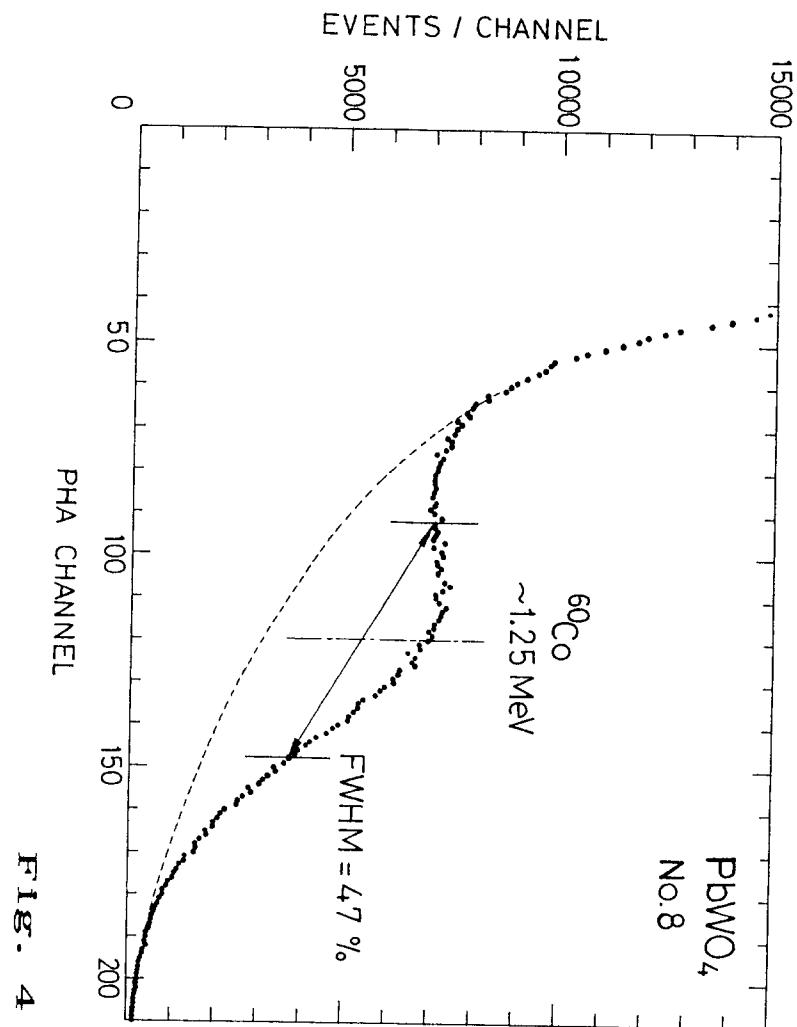


FIG. 4

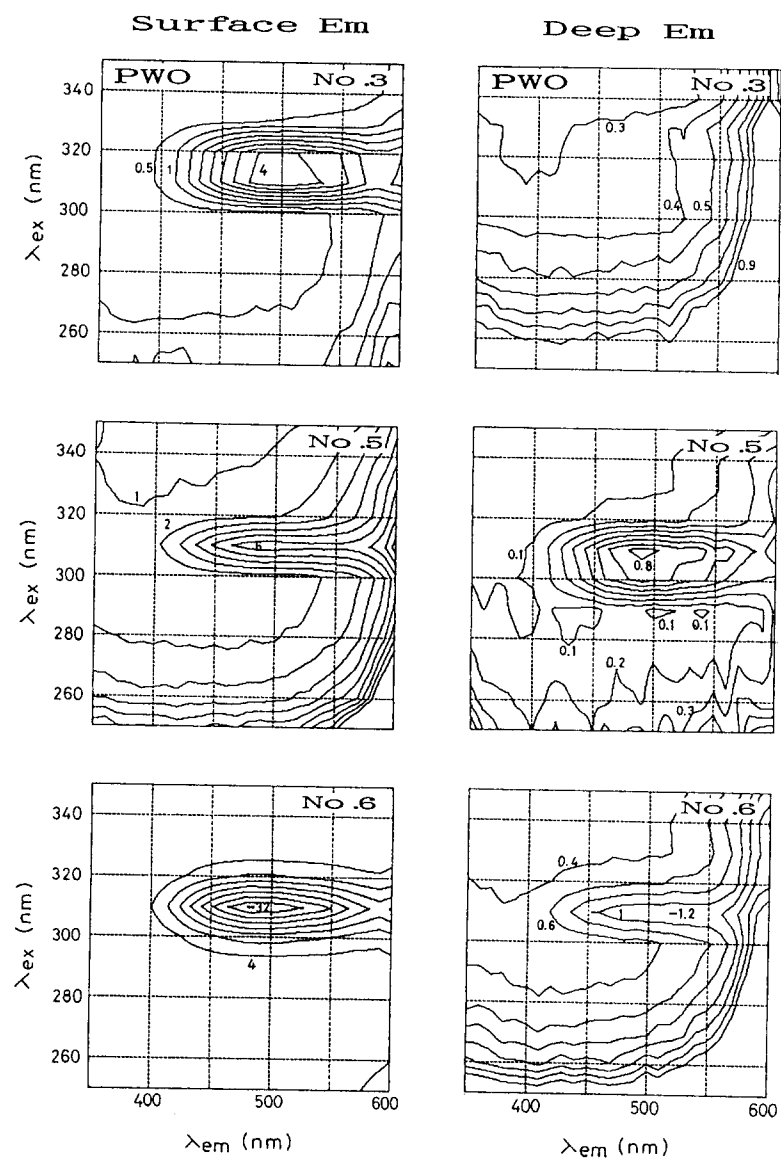


Fig. 5a

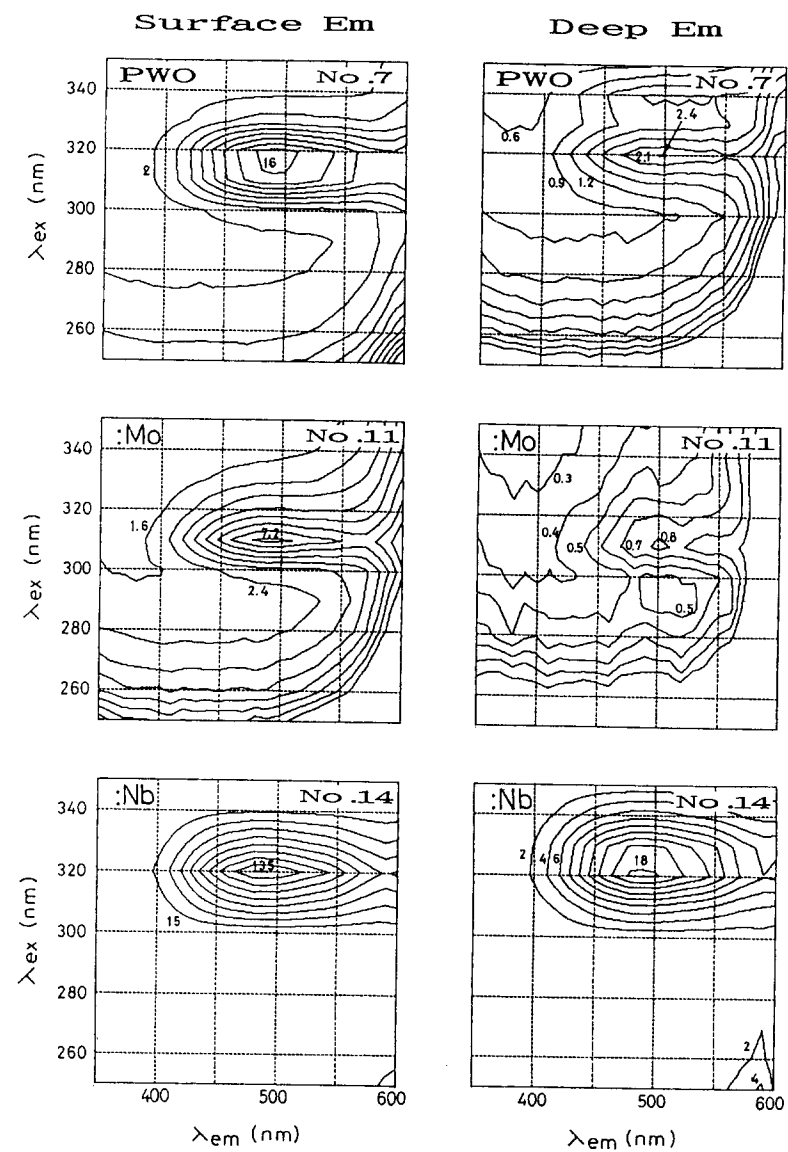


Fig. 5b

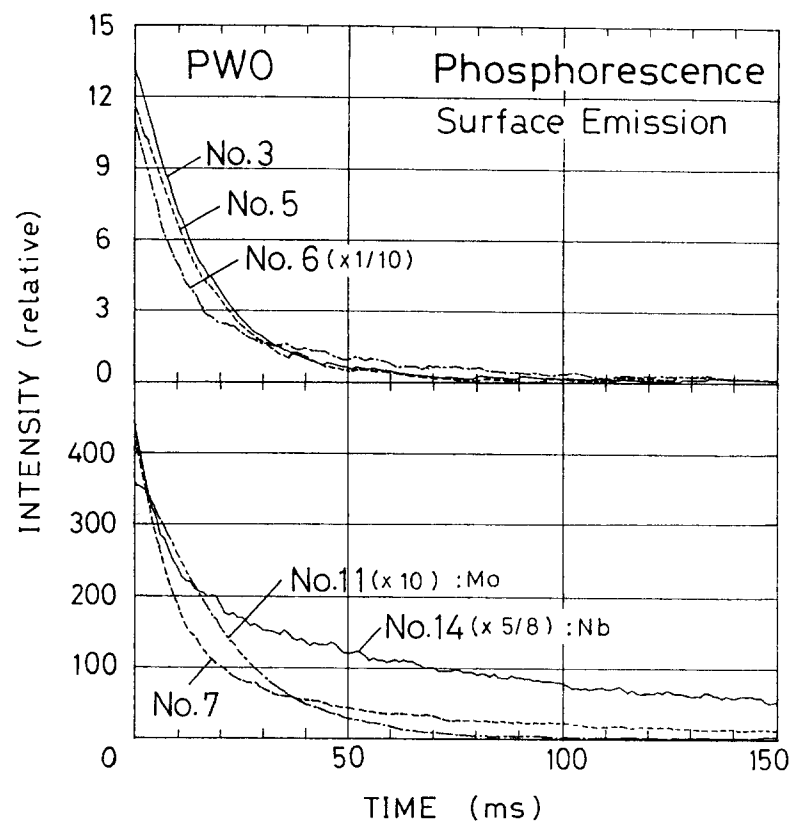


Fig. 6

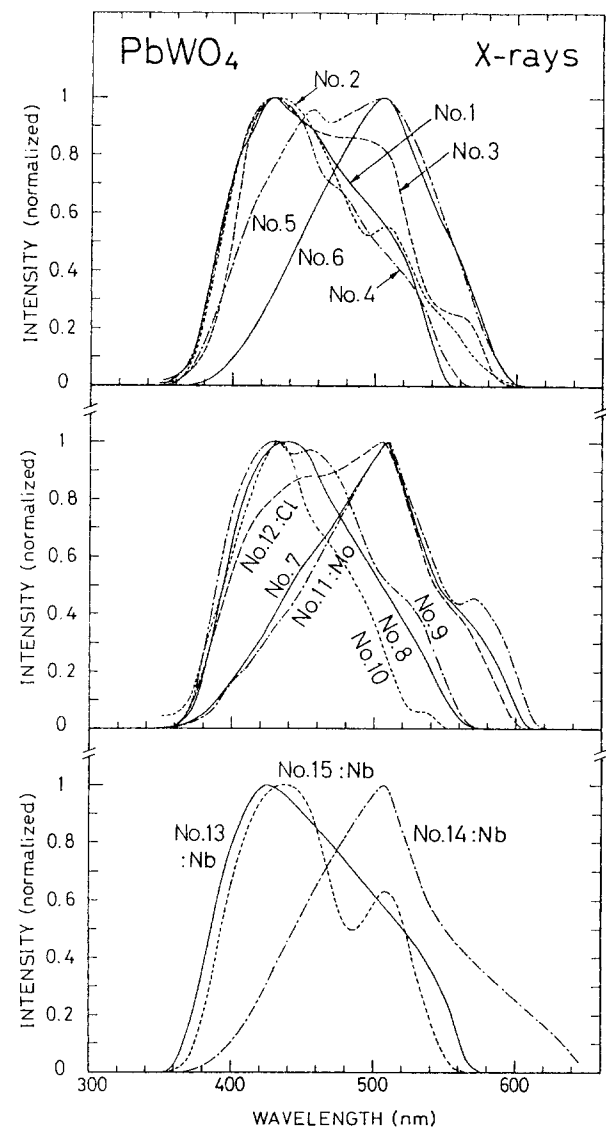


Fig. 7