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Note

UV-induced silica gel GF photoluminescence studied by liquid scintillation spectrophotometry

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The techniques and problems in thin-layer radiochromatographic analyses were described in a recent monograph devoted to radiochromatography[1]. One problem which is not discussed in that reference is the possibility of spontaneous light emissions which can occur during liquid scintillation counting of 254 nm UV-irradiated silica gel GF thin-layer zones. This potential problem is apparently recognized by many radiochromatographers[2], but literature which describe thin-layer zonal analysis techniques[3–6] make no specific mention of this UV-induced photoluminescent effect. Based on some preliminary findings[7] and literature which describe the measurement of spontaneous light emissions by liquid scintillation counting[8–10] we investigated the nature of 254 nm UV-induced silica gel GF photoluminescence. The present report describes the results of our findings.

EXPERIMENTAL

Materials

Pre-coated silica gel G and silica gel GF thin-layer plates (thickness 0.25 mm) were purchased from Analtech (Newark, DE, U.S.A.) and used without modification. E. Merck silica gel GF, Type 60, was purchased from MCB Reagents (Cincinnati, OH, U.S.A.) and applied onto glass plates (thickness 0.25 mm) as an aqueous slurry with a Desaga applicator. A sample of the Analtech manganese-activated zinc silicate powder was generously provided by Dr. Herman Felton, Analtech. Woelm fluorescent green indicator was purchased from ICN Pharmaceuticals, Life Science Group (Cleveland, OH, U.S.A.). The scintillator solutions employed in these studies included Scintiverse (Fisher Scientific, Fairlawn, NJ, U.S.A.), Bray's solution[11], and a toluene cocktail made with 90 mg/l PPO and 4.50 g/l POPOP. All solvents and reagents used in these experiments were purchased from Fisher Scientific, except naphthalene, which was purchased from New England Nuclear Corporation (Boston, MA, U.S.A.).

Instrumentation

Ambient temperature liquid scintillation counting was performed with a

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Beckman LS 100-C instrument operated with a gain setting of 339 mV. Channel 1 had a present discriminator setting to monitor tritium, channel 2 had preset discriminator settings to monitor tritium–carbon-14 or carbon-14 and channel 3 was preset to monitor tritium through phosphorus-32 β-energy levels. The instrument was operated in near darkness within a temperature range of 24.5–26.5°C. Instrumental efficiency for carbon-14 was $> 95\%$ in channel 2, and $> 46\%$ for tritium in channel 1. Samples were counted for 5, 10 or 20 min with a preset error of 2% and the instrument was operated in the continuous cycle mode for up to 4 days. Discriminator settings for the various energy levels in the Beckman LS 100-C were established by counting UV-irradiated silica gel GF samples in a Beckman 7500 liquid scintillation counter. Discriminator settings for the Beckman 7500 instrument were 0–377 for tritium and 377–700 for carbon-14.

Subambient photoluminescent effects were recorded with a Packard Model 3225 liquid scintillation counter operated at 15–18°C with gain settings of 500 for tritium and 100 for carbon-14. The instrument was operated with discriminator settings of 0–300 for the tritium channel (channel 1) and 300–1000 for the carbon-14 channel (channel 2). Samples were counted for 5, 10 or 20 min in the cycle repeat mode for up to fourteen cycles per sample. Counting efficiency for tritium in this instrument was $> 42\%$ in channel 1 and $> 90\%$ for carbon-14 in channel 2.

Sample preparation

Sampling of the Analtech and Merck silica gel GF thin-layer plates has been described elsewhere[7]. Sampling of the manganese-activated zinc silicate powder was carried out under yellow lights and consisted of the following: (a) 1 g of the respective indicators were mixed with 99 g of Analtech silica gel G in a rotary mixer. The samples were divided into 80 ± 10 mg aliquots, transferred to glassine paper, then irradiated for 10 sec with a Mineralight Model UVS-11 short-wave UV lamp (Ultraviolet Products, San Gabriel, CA, U.S.A.) positioned 12 cm above the silica gel surface; (b) 1.5 ± 0.4 mg aliquots of the Analtech and Woelm zinc silicate indicators were placed on glassine paper and irradiated for 10 sec with the 254 nm UV lamp. Non-irradiated silica gel G, silica gel GF, zinc silicate indicator samples and solvents served as controls in procedure (a) and (b). Following UV irradiation, samples were added to glass scintillation vials along with 10 ml of the respective scintillator solutions. Both the control and UV-irradiated samples were mechanically shaken for 5 min prior to liquid scintillation analyses, and all experiments were performed in either duplicate or triplicate.

RESULTS AND DISCUSSION

Photoluminescent effects in samples of 254 nm UV-irradiated Analtech and Merck silica gel GF thin-layer powder and Analtech and Woelm manganese-activated zinc silicate indicators were studied by liquid scintillation spectrophotometry at 26°C and 18°C. The gel and indicator samples were added to organic scintillators composed of xylene, toluene or dioxan and UV-induced spontaneous light emissions were recorded with scintillation spectrophotometer discriminator settings corresponding to tritium, tritium–carbon-14, carbon-14 or tritium–phosphorus-32 β-energy levels. The intensity of the photoluminescent decay curves recorded

for the various UV-irradiated gels and indicators ranged from $> 10^5$ cpm at 26°C for the Analtech silica gel GF powder to > 700 cpm at 18°C for the Merck silica gel GF and Woelm zinc silicate powders. The duration of the UV-induced photoluminescent effects ranged from 86 h for the Analtech silica gel GF at 18°C, to 12 h for the Merck and Woelm materials. These results are summarized in Tables I and II. Tables I and II also show that 254 nm UV-induced silica gel GF photoluminescence should not interfere with liquid scintillation counting of carbon-14, provided that the counting channels have discriminator settings of > 300.

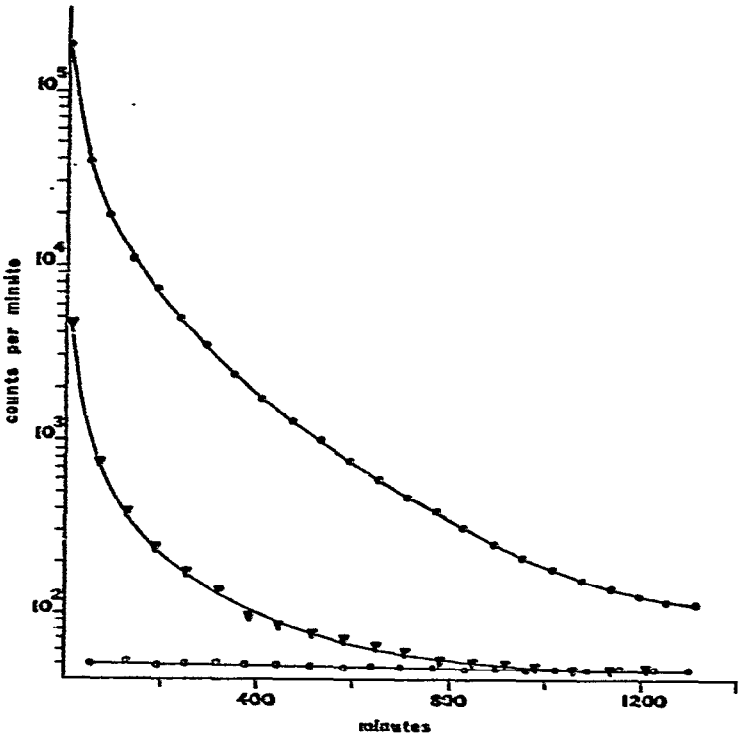

Fig. 1. Duration and intensity of 254 nm UV-induced photoluminescence from Analtech and Woelm manganese-activated zinc silicate indicators in toluene. ●, Analtech zinc silicate indicator; ▼, Woelm zinc silicate indicator; ○, background counts per minute. Similar results were obtained with xylenes and dioxan solvents.

One interesting result was recorded at 26°C. Discriminator settings of 0–1000 were used to monitor photoluminescence decay processes of UV-irradiated Analtech and Woelm zinc silicate samples in xylenes, dioxan or toluene which did not contain added organic scintillators. These results are shown in Fig. 1. It is evident from those decay curves that Analtech zinc silicate powder photoluminescences with more intensity and with longer duration than corresponding amounts of Woelm zinc silicate powder. These results may prove to be useful in some types of radio-luminescence analyses[12,13] which employ silica gel GF.

TABLE I

INTENSITY AND DURATION OF AMBIENT TEMPERATURE PHOTOLUMINESCENCE RECORDED FOR 254 nm UV-IRRADIATED SILICA GEL GF AND ZINC SILICATE SAMPLES

Scintillator mixtures	*Counting channel*	*Analtech silica gel GF*		*Analtech silica gel G and Analtech Indicator*		*Merck silica gel GF*		*Analtech silica gel G and Woelm Indicator*	
		*cpm**	*hours***	*cpm**	*hours***	*cpm**	*hours***	*cpm**	*hours***
Scintiverse	1	>10,000	60	>1000	36	>4000	10	500	12
	1–2	>10,000	60	>1000	36	>4000	10	500	12
	1–3	>10,000	60	>1000	36	>4000	10	500	12
	2	***				***		***	
Bray's solution	1	>10,000	60	>1000	36	>4000	10	>500	12
	1–2	>10,000	60	>1000	36	>4000	10	>500	12
	1–3	>10,000	60	>1000	36	>4000	10	>500	12
	2	***				***		***	
Toluene solution	1	>10,000	60	>1000	36	>500	10	500	12
	1–2	>10,000	60	>1000	36	>500	10	500	12
	1–3	>10,000	60	>1000	36	>500	10	500	12
	2	***				***		***	

* Data represent the lowest cpm recorded from either duplicate or triplicate samples approximately 2 h after sample irradiation.

** Represents the minimum number of hours required to reach background levels of 20–60 cpm.

*** Photoluminescence was not detected.

TABLE II

INTENSITY AND DURATION OF SUBAMBIENT TEMPERATURE PHOTOLUMINESCENCE FOR 254 nm UV-IRRADIATED SILICA GEL GF AND MANGANESE-ACTIVATED ZINC SILICATE SAMPLES

Solvent	*Counting channel*	*Analtech silica gel GF*		*Analtech silica gel G and Analtech indicator*		*Merck silica gel GF*		*Analtech silica gel G and Woelm indicator*	
		*cpm**	*hours***	*cpm**	*hours***	*cpm**	*hours***	*cpm**	*hours***
Scintiverse	1	>3000	>86	<1000	48	<1000	11	>700	12
	1–2	>3000	>86	<1000	48	<1000	11	>700	12
	2	***		***		***		***	
Bray's solution	1	>3000	>86	<1000	48	<1000	11	>700	12
	1–2	>3000	>86	<1000	48	<1000	11	>700	12
	2	***		***		***		***	12
Toluene solution	1	>3000	>86	<1000	48	<1000	11	>700	12
	1–2	>3000	>86	<1000	48	<1000	11	>700	12
	2	***		***		***		***	

*–*** As in Table I.

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