Short Communication

A new chemical filter for the isolation of the 366 nm mercury emission line

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1. *Introduction*

Many photochemical reactions are carried out at the high intensity 366 nm line of the medium pressure mercury arc, the isolation of which is often necessary, especially for quantum yield determinations. To our knowledge, only filters containing two solutions separated by a planar highly selective glass window have been described for its isolation (refs. 1, 2, p. 734, and 3). The use of such filters is thus limited to linear irradiation systems. They should not be incorporated in large preparative cylindrical photoreactors.

In this paper a single-solution chemical filter able to isolate the 366 nm mercury emission line in the UV region is described.

2. *Experimental details*

Benzalphthalide was prepared by the reaction of phthalic anhydride with phenylacetic acid in the presence of sodium acetate [4]. 2,3-Diphenyl-1-indenone was obtained from benzalphthalide using the procedure of Allen *et al.* [5] (melting point, 162 °C). No difference in behaviour from that of a commercial sample (Aldrich, Belgium) was found. Azulene (Puriss grade, Fluka, Switzerland) was used without further purification. The solvent was spectroscopic grade *n*-hexane (Uvasol, Merck, F.R.G.).

The filter solutions were irradiated in the presence of air at room temperature by a 125 W medium pressure mercury lamp (HPK 125, Philips, The Netherlands) in a cylindrical Pyrex photoreactor. The total energy radiated was passed through a solution of volume 250 ml and with a path length of 10 mm.

The transmission spectra were recorded on a Beckman spectrophotometer (model DB).

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3. *Results and discussion*

A typical medium pressure mercury lamp such as the Philips HPK 125 burner emits several lines in the UV and visible region with a maximum intensity at 366 nm [6] (Table 1). It is relatively easy to eliminate the emission below 300 nm by the use of UV-absorbing glasses or solvents. In many cases it is not necessary to remove the green (546 nm) and the yellow (578 nm) lines which are not absorbed by most chemical reagents, *e.g.* ferrioxalate actinometric solution [7]. The main problem therefore consists in separating the 366 nm line from the intense 313 nm, 404 nm (violet) and 436 nm (blue) lines.

We found that such a separation can be effected by a solution of 2,3-diphenyl-1-indenone in *n*-hexane at a suitable concentration (0.22 g l^{-1}). The transmission spectra in Fig. 1 and in Table 2 show that the total UV emis-

TABLE 1

Emission maxima of the Philips HPK 125 lamp [6]

Emission wavelength λ_{max} (nm)	*Intensity* (relative values)
313	0.61
334	0.08
366	1.00
404	0.35
436	0.69
546	0.86
578	0.69

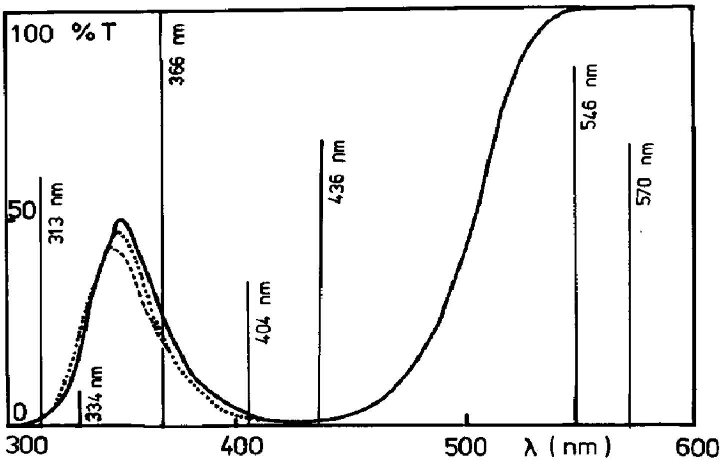

Fig. 1. Transmission spectrum of the filter solution 2,3-diphenyl-1-indenone–*n*-hexane (concentration, 0.22 g l^{-1}; cell length, 10 mm): ——, before irradiation; · · · · ·, after 1 h of irradiation; – – –, between 24 and 100 h of irradiation. The vertical lines represent the relative values of the intensities of the mercury emission lines (Philips HPK 125 W lamp).

TABLE 2

Percentage transmission at different wavelengths *versus* the time of irradiation of the chemical filter 2,3-diphenyl-1-indenone–*n*-hexane (concentration, 0.22 g l^{-1}; path length, 10 mm)

Time of irradiation (h)	*Percentage transmission*							
	313 nm	*334 nm*	λ_{max}	*366 nm*	*404 nm*	*436 nm*	*546 nm*	*578 nm*
Unirradiated	0.7	22	50 (350 nm)	28	3	1	100	100
1	0.7	24	47 (349 nm)	25	2	1	100	100
24	0.7	24	43 (346 nm)	25	1	1	100	100
56	0.7	24	43 (346 nm)	25	1	1	100	100
100	0.7	24	43 (346 nm)	25	1	1	100	100

sion (including the 313 nm line) and the 404 and 436 nm lines are almost entirely absorbed while 25% of the 366 nm line is transmitted for an absorption thickness of 10 mm.

The stability of this filter solution was checked by irradiating it for 100 h under the conditions described in Section 2. The height and the position of the absorption maximum near 350 nm decrease slightly within 24 h of irradiation but then remain unchanged for up to 100 h. The transmission values at the different mercury emission wavelengths reach stability after 1 h (Table 2). In addition we did not notice any change in the spectrum of the unirradiated solution after it had been kept in sealed bottles for 3 years. The weak residual transmittances at 313, 404 and 436 nm can be decreased to zero by increasing the concentration of the solution, but the transmittance at 366 nm then falls below 10%.

In many cases it is not necessary to eliminate the very weak 334 nm line. Nevertheless we tried to remove it by adding to the solution either a suitable cut-off filter glass or a second solute which absorbed strongly at 334 nm and weakly at 366 nm. The removal was effected initially by enclosing the lamp in a cylinder made of Jena glass containing some yellow cerium salt (GWC glass, Glasswerke Wertheim, F.R.G.), the transmission of which was 0% at 340 nm and 80% at 400 nm. The compounds added to the solution are listed in Table 3. Only pyrene and azulene have satisfactory transmittance values for the different mercury lines, but the pyrene-containing solution is not stable against irradiation. Therefore we studied the diphenylindenone–azulene–hexane solution in more detail. Table 4 gives evidence for the stability of this filter during the first stage of irradiation. From 18 to 96 h, the whole transmission spectrum (Table 4 and Fig. 2) changes progressively; however, the transmissions at 334 and 366 nm are only slightly affected. This alteration can be attributed to simultaneous decreases in both the absorption of diphenylindenone and that of azulene.

An attempt to substitute the solvent toluene, which absorbs UV better and is less volatile, for *n*-hexane failed because a green unidentified precipitate appeared from the start of irradiation.

In order to show that, when necessary, it is possible not only to eliminate the 546 and 570 nm lines but also to maintain simultaneously a reasonable value of transmittance at 366 nm, we added to the diphenylindenone–azulene filter a second filter solution of iodine in CCl_4, the stability of which has been tested by other workers (ref. 2, p. 736). When an I_2–CCl_4 solution containing 2 g I_2 l^{-1} is used in a 10 mm cell, all the mercury emission lines are absorbed except the 366 nm line for which the transmittance is 20% (Fig. 3).

4. Conclusion

A one-component solution of diphenylindenone in *n*-hexane at a given absorbance was tested for stability against an intense emission from a medium pressure mercury lamp. In practical use, this solution yields an efficient filter for the important 366 nm line. It may advantageously compete for

TABLE 3

Percentage transmission of some binary solutions at different wavelengths (path length, 10 mm)

Solute 1 (concentration)	*Solute 2 (concentration)*	*Solvent*	*Time of irradiation* (h)	*Percentage transmission*				
				313 nm	*334 nm*	*366 nm*	*404 nm*	*436 nm*
2,3-diphenyl-1-indenone ($0.22\ g\ l^{-1}$)	Anthraquinone ($0.05\ g\ l^{-1}$)	Benzene	0	0	3	28	3	2
2,3-diphenyl-1-indenone ($0.22\ g\ l^{-1}$)	Xanthone ($0.05\ g\ l^{-1}$)	Benzene	0	0	0	32	5	3
2,3-diphenyl-1-indenone ($0.22\ g\ l^{-1}$)	Pyrene ($0.01\ g\ l^{-1}$)	Benzene	0	0	0	27	3	1
2,3-diphenyl-1-indenone ($0.22\ g\ l^{-1}$)	Pyrene ($0.01\ g\ l^{-1}$)	Benzene	100	0	2	27	20	19
2,3-diphenyl-1-indenone ($0.22\ g\ l^{-1}$)	Azulene ($0.15\ g\ l^{-1}$)	Hexane	0	0	0	24	1.5	1
2,3-diphenyl-1-indenone ($0.22\ g\ l^{-1}$)	Azulene ($0.15\ g\ l^{-1}$)	Hexane	96	0	0	23	2	2

TABLE 4

Percentage transmission at different wavelengths *versus* the time of irradiation of the chemical filter 2,3-diphenyl-1-indenone (concentration, 0.22 g l^{-1})–azulene (concentration, 0.15 g l^{-1}) in *n*-hexane (path length, 10 mm)

Time of irradiation (h)	*Percentage transmission*							
	313 nm	*334 nm*	λ_{max} *(360 nm)*	*366 nm*	*404 nm*	*436 nm*	*546 nm*	*578 nm*
Unirradiated	0	0	28	24	1.5	1	52	46
18	0	0	28	24	1.5	1	52	46
24	0	0	23	20	1.5	1	52	47
48	0	0	23	20	1.5	1	55	51
96	0	0	26	23	2	2	65	62

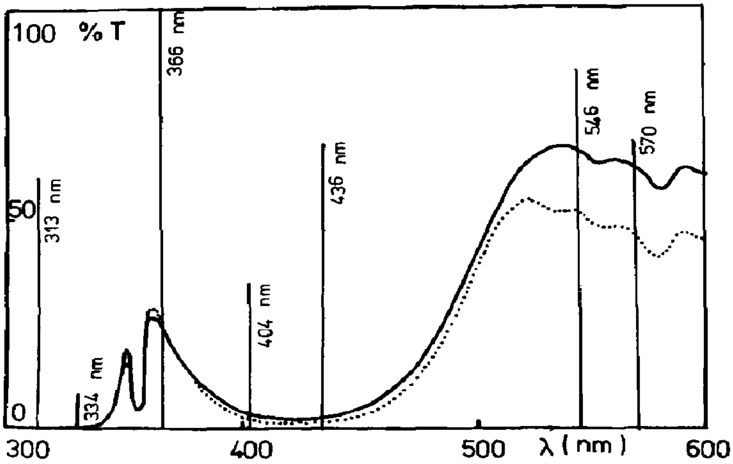

Fig. 2. Transmission spectrum of the filter solution 2,3-diphenyl-1-indenone (concentration, 0.22 g l^{-1})–azulene (concentration, 0.15 g l^{-1}) in *n*-hexane (cell length, 10 mm): ·····, before irradiation and after 18 h of irradiation; ——, after 96 h of irradiation. The vertical lines represent the relative values of the intensities of the mercury emission lines (Philips HPK 125 W lamp).

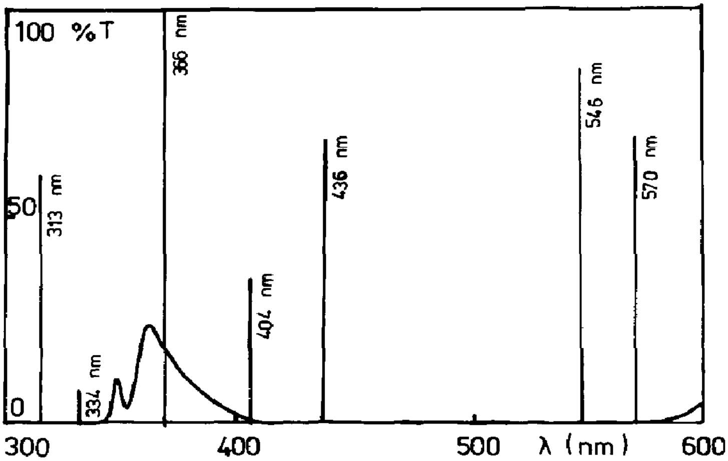

Fig. 3. Transmission spectrum of the added filter solutions 2,3-diphenyl-1-indenone (concentration, 0.22 g l^{-1})–azulene (concentration, 0.15 g l^{-1}) in *n*-hexane (cell length, 10 mm) and iodine in CCl_4 (concentration, 2 g l^{-1}; cell length, 10 mm). The vertical lines represent the relative values of the intensities of the mercury emission lines (Philips HPK 125 W lamp).

transmittance and stability with previously described filters. In addition, it is easier to prepare and to handle. In special cases, where the 334, 546 or 570 nm emissions are disturbing, the monochromaticity of this filter can be improved by the addition of a second solute or a second one-component solution.

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