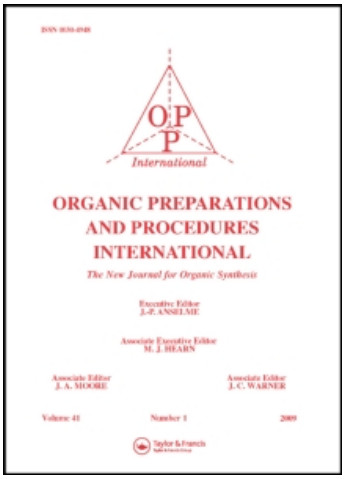

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A PREPARATIVE AVICEL-CELITE TLC

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the temperature indicated in Table 1, until complete disappearance of 1 occurred (TLC, silica gel, 60:40 hexane-tetrahydrofuran as eluent). Diethyl ether (600 ml) and water (200 ml) were added and after stirring (10 min), the organic layer was separated and evaporated in vacuo. The residue was then crystallized from a suitable solvent or purified by silica chromatography.

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A PREPARATIVE AVICEL-CELITE TLC

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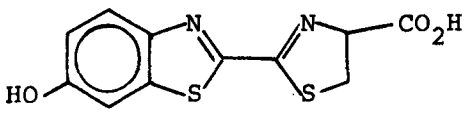
The reproducibility of R_f values is excellent on cellulose paper chromatograms (PC) but spots are apt to enlarge and only a very small

amount of sample can be applied. In contrast, good separations and spots are expected in Avicel thin layer chromatograms (TLC) but reproducibility of R_f values and the sample size are not satisfactory. Thick layer Avicel TLC plates for preparative scale are not suitable because of crack formation during their preparation. These cracks may result from the prevention of water-evaporation caused by the formation of surface films. We have obtained uncracked and good quality Avicel thick layers for preparative scale chromatography (thickness: 1 mm) by using Avicel mixed with Celite to improve ventilation, and found that these layers are useful for the separations of firefly luciferin (1), oxyluciferin (2a), and dehydroluciferin (2b), with good reproducibility of R_f values.[1]



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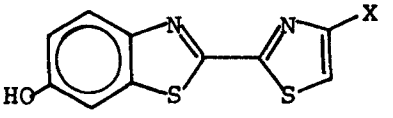

2 a) X = OH
b) X = CO_2H

PROCEDURE

Avicel (E. Merck A. G. or Funakoshi Pharm. Co.: microcrystalline cellulose, 30 g) and Hyfro-super-cel (Nakarai Chem. Co.: Celite: 15 gals/sq. ft/hr; washed with water and dried at 110° for 12 hrs before use, 10 g) were mixed thoroughly in a mortar with water (140 ml), spread with an applicator (Yazawa Scientific Instr. Co.: thickness: 1.0 mm) on glass plates (20 x 20 cm), and dried slowly in a wooden box with small slits, for 2 days (20-25°). Alternatively, the plates could be dried in a ventilated electric oven at 60° for 1 hr after being air-dried overnight.

R_f values and the order of elution on the preparative Avicel-Celite plates were closely similar to that obtained on analytical Avicel plates. Thus, the merit of the present method is that the results of analytical TLC were applicable to preparative TLC. Although TLC on Avicel-Kieselguhr was

reported by Lee independently of our results [S. C. Lee, J. Chromatogr., 93, 480 (1974)], our material is superior because of the correspondence between R_f values for analytical and preparative TLC.

Quantities of the samples applied ranged from 20-30 mg at maximal conditions up to 100 mg per plate. A fluorescent lamp (366 nm) was used for detection and elution was carried out with ether or methanol. The same developing solvents applicable on PC could be used.

TABLE I. R_f Values of 1-3 on Preparative Avicel-Celite TLC

Solvents	R_f Value		
	1	2	3
MeOH-H_2O (4:5)	0.92	0.53	0.77
95% EtOH-1N NH_4OAc (9:1)	0.64	0.88	0.25

Avicel-Celite (30:10); thickness: 1.0 mm on glass plate (20 x 20 cm).

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1. This method is an improvement upon that which was briefly commented upon in a previous paper [N. Suzuki and T. Goto, Tetrahedron, 28, 4075 (1972)].