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Note

Detection of organosulphur compounds on thin layers using Dragendorff's reagent

DIPAKRANJAN MAL* and GOUTAM BISWAS
Department of Chemistry, Bose Institute, Calcutta 700 009 (India)
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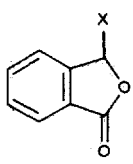
Organosulphur compounds are important intermediates in organic synthesis[1] and their utilization, particularly that of sulphones[2] and sulphoxides[3], in forming new carbon–carbon bonds and thus offering promise for synthesizing complex molecules is well documented. Their thin-layer chromatographic detection, however, is confined to a narrow choice of suitable spray reagents. In mid-1970s, acetic acid–chromium trioxide solution[4] and ammonium hexanitratocerate(IV) solution[5] were introduced as spray reagents for α-polychlorosulphoxides, sulphides, thiols and sulphinate esters, but they are not effective with organic sulphones. In this paper, we describe the application of Dragendorff's reagent for the detection of certain classes of organosulphur compounds.

Dragendorff's reagent[6] in its various forms has found widespread application in the detection of nitrogen-containing compounds such as alkaloids, quaternary ammonium salts, lactams, phenothiazines and Rh^{3+}–amine complexes. It is also useful for α-pyrones and polyethylene glycols. Trézl *et al.*[7] reported crown ethers can develop characteristic colours with Dragendorff's reagent.

During the course of investigations on the synthetic utility of phthalide sulphones[3], we observed that phthalide sulphone 1d produced a bright orange-red spot on treatment with Dragendorff's reagent on a thin-layer plate. Based on this observation, we further proceeded to test under the same conditions various sulphur compounds as shown.



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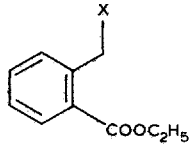

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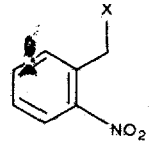

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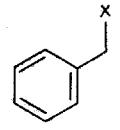

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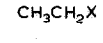

5

a, X = H; b, X = $S-C_6H_5$; c, X = $S(=O)-C_6H_5$; d, X = $S(=O)_2-C_6H_5$

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EXPERIMENTAL

The sulphides, sulphoxides and sulphones were prepared by standard procedures[3]. Thin-layer plates (20 cm × 10 cm) with silica gel G layers were spotted with compounds 1–5 and developed with appropriate solvents. After air drying they were sprayed with a freshly prepared solution of Dragendorff's reagent[8]. Immediate colour formation on a yellow background was observed, as indicated in Table I.

TABLE I

RESULTS FOR SULPHONES, SULPHOXIDES, SULPHIDES AND OTHERS

Type	*Compound*	*Solvent*	*Colour**	R_F	*Amount applied (μg per spot)*
Sulphones	1d	Chloroform	R	0.60	15
	2d		R	0.43	25
	3d		R	0.58	23
	4d		R	0.62	30
	5d		R	0.51	26
Sulphoxides	1c	Chloroform–ethyl	R	0.45	19
	2c	acetate (99:1)	R	0.42	20
	3c		R	0.66	40
	4c		R	0.55	35
	5c		R	0.73	36
Sulphides	1b	Chloroform–ethyl	R	0.82	25
	2b	acetate (99:1)	R	0.72	35
	3b		R	0.85	33
	4b		LO	0.87	50
	5b		LO	0.90	65
Others	1a	Chloroform	R	0.73	23
	p-Toluenesulphonic acid		C	–	50
	Thiophenol		C	–	55
	Thioglycolic acid		C	–	53

* Colour developed at room temperature. R = orange-red; LO = light orange-red; C = no characteristic colour.

RESULTS AND DISCUSSION

All the sulphones, sulphoxides and sulphides produced orange-red spots with Dragendorff's reagent, irrespective of the oxidation state of the sulphur atoms in the molecules. Sulphides 4 and 5, however, gave weakly coloured spots. Thiophenol, thioglycolic acid and *p*-toluenesulphonic acid, on the other hand, did not respond to this test. It is conceivable that the acidic groups of these substrates are responsible for preventing the colour reaction with Dragendorff's reagent. Unlike the aromatic ester 2a, the parent phthalide 1a gave an orange-red spot with the reagent. Although only one example of this class of compounds was investigated, it appears that such behaviour of phthalides could be of general character.

In comparison with chromic acid–acetic acid reagent, Dragendorff's reagent seems to be equally or more sensitive and produces a sharp, stable colour contrast on the thin layers. More important, it gives colours with organosulphones, which cannot be detected with the chromic acid–acetic acid system or with ammonium hexanitratocerate(IV) solution.

In conclusion, the use of Dragendorff's reagent constitutes a complementary method of detecting organosulphur compounds in thin-layer chromatography.

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