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## Note

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### Separation and identification of some pyridine derivatives

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As part of a larger investigation into the metabolism of sodium pyridine-1-oxide-2-thiol it became necessary to establish a method for the identification and separation of some pyridine derivatives. Previous workers<sup>1</sup> have reported that TLC systems can be used to identify and separate sodium pyridine-1-oxide-2-thiol, 2-mercaptopyridine, pyridyl-2,2'-disulphide-1,1'-dioxide, pyridyl-2,2'-disulphide and pyridine-1-oxide-2-sulphonic acid.

Attempts to repeat this work were only partially successful, in so far as an effective separation of only four of the five compounds could be achieved. The purpose of this work is to show that all of these compounds can be separated and identified by paper chromatography.

### EXPERIMENTAL

The following materials were obtained from the Olin Chem. Corp., Chemicals Group Research, New Haven, Conn., U.S.A.: sodium pyridine-1-oxide-2-thiol (I), 2-mercaptopyridine (II), pyridyl-2,2'-disulphide-1,1'-dioxide (III), pyridyl-2,2'-disulphide (IV), and pyridine-1-oxide-2-sulphonic acid (V).

The IR spectra were taken in KCl discs (2%) and were as expected. The melting points of compounds I–IV were determined on a Büchi apparatus and agreed with published data<sup>2–5</sup>. The melting point of compound V was found to be 235° (decomposition) (ref. 6: 240–241°). Combustion analysis of compound V showed 8.18% N, 18.3% S; C<sub>5</sub>H<sub>5</sub>NO<sub>4</sub>S requires 8.0% N, 18.3% S.

The chromatography was carried out by an ascending technique on Whatman No. 1 paper strips (26 × 10 cm) in a closed circular tank (16 cm diameter × 32 cm). The compounds were dissolved in an appropriate solvent (absolute ethanol or distilled water, see Table I) and an inoculum of 1 μl was used (microcaps).

The solvent systems used were: (A) *n*-butanol–glacial acetic acid–water (5:1:2), and (B) isobutanol–*n*-propanol–0.880 ammonia (1:4:2). The chromatograms were then developed at room temperature with the solvent systems for 6.5 h. After development the chromatograms were dried at 50° in an air oven for 30 min

and sprayed with an 0.1% w/v solution of sodium fluorescein in absolute ethanol, after drying again at 50° they were examined under long UV in a 'Fluotest Universal' light cabinet.

## RESULTS AND DISCUSSION

The chromatographic results are shown in Table I.

Each value represents the mean of three determinations, each series of three determinations only showed differences in the last figure, which was regarded as being within the limits of experimental error.

Several other solvent systems were studied but the chromatograms thus obtained had the disadvantage of incomplete separation of compounds IV and V which would be a serious handicap in metabolic studies.

TABLE I  
CHROMATOGRAPHY OF COMPOUNDS I-V

Compounds I-IV were dissolved in absolute ethanol, compound V was dissolved in distilled water.

Compound	R <sub>F</sub>	
	Solvent system A	Solvent system B
I	0.89	0.38
II	0.76	0.87
III	0.76	0.68
IV	0.97	0.85
V	0.19	0.25

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