

CHROM. 9947

## Note

---

### Modified thin-layer chromatographic method for the detection of phenols

B. R. CHHABRA, R. S. GULATI, R. S. DHILLON and P. S. KALSI

*Department of Chemistry, Punjab Agricultural University, Ludhiana (India)*

(Received November 23rd, 1976)

A number of methods for the detection of phenols on silica gel G thin-layer plates are known<sup>1</sup>. Detection with iron(III) chloride-hexacyanoferrate(III) reagent has the disadvantage that the reacted reagent cannot be washed from the water-sensitive silica gel G plates, and a black background colour rapidly develops; also, this reagent fails in the detection of monohydric phenols. Spraying with sodium hexanitritocobaltate(III) in acetic acid<sup>2</sup> gives yellow-brown spots on a blue background after heating for 5 min at 105°, but this method is not very sensitive for the detection of phenols that have a carbonyl group in the *ortho*-position<sup>2</sup>. Other spray reagents such as diazotized benzidine, sulphanilamide and sulphanilic acid are suitable for the detection of phenols that are capable of undergoing coupling. However, in the diazotization method the reagent has to be prepared at a low temperature and is stable for only 2-3 h.

To overcome the last shortcoming, we propose here a modification of the diazotization technique in which silica gel G layers impregnated with anilinium chloride are used and the chromatogram is developed by spraying with sodium nitrite solution.

## EXPERIMENTAL

### *Preparation of silica gel G impregnated with anilinium chloride*

Aniline (analytical-reagent grade, 10 g) was mixed with an excess of concentrated hydrochloric acid (about 30 ml). The mixture was cooled, filtered, washed with a small volume of diethyl ether and dried.

Silica gel G (E. Merck, Darmstadt, G.F.R.; 100 g) was added to a solution of anilinium chloride (5 g) in water (200 ml) and the mixture was stirred thoroughly to give a fine slurry. This mixture was then coated on glass plates (20 × 5 cm) to a thickness of about 0.25 mm. The plates were air dried (15 min) and activated for ½ h at 120°. After spotting, the plates were developed for 35 min in a chromatographic tank (25 × 11 × 9 cm) lined with filter-paper to a distance of 15 cm (ambient temperature, 35°), then air dried and sprayed with 5% (w/v) sodium nitrite solution.

## RESULTS

The spots appear instantaneously and the plates are stable for many days.

TABLE I

$R_f$  VALUES OF PHENOLS ON IMPREGNATED SILICA GEL G PLATES WITH BENZENE-ETHYL ACETATE AS SOLVENT

Compound	Benzene-ethyl acetate			Colour
	9:1	3:1	1:1	
$\beta$ -Naphthol	0.47	0.69	0.74	Crimson
$\alpha$ -Naphthol	0.44	0.66	0.71	Pink
Phenol	0.59	0.64	0.73	Dark brown
Pyrogallol	0.03	0.27	0.46	Grey
2,4-Dinitrophenol	0.68	0.70	0.76	Yellow
<i>o</i> -Nitrophenol	0.61	0.65	0.73	Light brown
<i>p</i> -Nitrophenol	0.30	0.57	0.68	Yellow
Resorcinol	0.19	0.40	0.48	Brown
Salicylic acid	0.41	0.43	0.55	Light pink
Thymol	0.70	0.75	0.86	Orange

Ten phenols were tested, using benzene-ethyl acetate in different proportions (9:1, 3:1 and 1:1) as solvent. The colours obtained are reported in Table I at 2- $\mu$ g concentrations of the phenols. Of particular interest is the successful separation of  $\alpha$ - and  $\beta$ -naphthol.

Similar results were obtained by using the hydrochloride of sulphanilic acid as the impregnating reagent.

## REFERENCES

- 1 E. Stahl, *Thin Layer Chromatography: A Laboratory Handbook*, Springer, New York, 1969, p. 705.
- 2 I. S. Bhatia, K. J. Bajaj, A. K. Verma and J. Singh, *J. Chromatogr.*, 62 (1971) 471.