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\$ -RESORCYLALDEHYDE AS A CHROMATOGRAPHIC SPRAY REAGENT FOR METAL IONS

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β -RESORCYLALDEHYDE or 2,4-dihydroxybenzaldehyde has been employed as a specific chromatographic colour reagent for amino acids¹, and some of its derivatives have been shown to possess antibacterial² and antimalarial³ activity. It has also been employed as a spectrophotometric reagent for the determination⁴ of iron(III). In the present study it has been found to give colour reactions with 32 metal ions on a paper chromatogram in the visible and UV light. The colour reactions are particularly intense with transition metal ions: Ti(IV), V(V), Mn(II), Fe(II), Fe(III), Co(II), Ni(II), Cu(II), Ce(III) and U(VI). The sensitivity limits of detection of these metal ions with the reagent have been determined. The compound can therefore be employed as a chromatographic spray reagent for the identification of metal ions in macro and micro quantities.

β -Resorcylaldehyde was prepared and purified, m.p. 135°C (Lit. 135°-136°C) by the usual procedure⁵.

Metal ion aqueous solutions of appropriate concentrations were prepared from AnalaR grade soluble salts of the respective metals.

Ascending paper chromatography, using Whatman No. 3 filter paper, was used for the development of chromatograms. n-Butanol: 6N HCl (1:1 V/V) solvent system was employed in all cases. The chromatograms were developed at room temperature ($\approx 20^{\circ}$ C) under optimum conditions of development. They were dried, exposed to ammonia and then sprayed with the reagent solution (0.5%) in 95% ethanol. The chromatograms were scanned in visible and UV light.

The colour reactions given by the 32 metal ions in the visible and UV light are listed in table 1. Table 2 shows the sensitivity limits with reference to the reagent and the corresponding values with other reagents wherever cited in the literature.

TABLE 1

Colour reactions of the metal ions in visible and UV light

```
ygr \equiv yellow grey; ly = light yellow; y = yellow, gr = grey;
lgr \equiv light grey; c = chocolate; yg = yellow green; fty = faint yellow;
brr \equiv brick red; fbl = fluorescent blue; blw = blue white;
fw \equiv fluorescent white; b = brown; dv = dark violet; v = violet;
lbl \equiv light blue; lb = light brown; vgr = violet grey; db = dark brown;
yw \equiv yellow, white; dg = dark green.
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M_n^+ (visible, UV)

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Ag<sup>+</sup>(-, lbl), Al<sup>3+</sup>(ly, fw), As<sup>3+</sup>(-, y<sub>2</sub>w), Ba<sup>2+</sup>(ly, dg),
Be<sup>2+</sup>(ygr, fbl), Ca<sup>2+</sup>(-, blw), Ce<sup>3+</sup>(lgr, vgr), Ce<sup>4+</sup>(lgr, gr),
Co<sup>2+</sup>(lgr, vgr), Cr<sup>3+</sup>(ly, lb), Cs (-, yg), Cu<sup>2+</sup>(yg, db), Fe<sup>2+</sup>(c, dv),
Fe<sup>3+</sup>(c, dv), Hg<sup>2+</sup>(-, lbl), La<sup>3+</sup>(ly, yg), Mg<sup>2+</sup>(-, blw), Mn<sup>2+</sup>(lgr, dv),
Nd<sup>3+</sup>(fty, brr), Ni<sup>2+</sup>(lgr, vgr), Pr<sup>3+</sup>(fty, brr), Rb (-, yg),
Sb<sup>3+</sup>(-, yw), Sn<sup>2+</sup>(-, blw), Th<sup>4+</sup>(ly, db), Ti<sup>4+</sup>(y, b), U<sup>6+</sup>(brr, db),
V<sup>4+</sup>(gr, dv), V<sup>5+</sup>(lgr, v), Y<sup>3-</sup>(fty, lbl), Zn<sup>2+</sup>(-, fbl), Zr<sup>4+</sup>(fty, b).
```

0.5

0.01

Sensitivity limits (µg) of metal ions with various spray reagents									
Metal Ions	Reagent								
	I		II		III	Ϊ́V	V	VI	VII
	Visible	UV	Visible	UV	Vis	Vis	Vis	Vis	Vis
Ce(III)	20	8.0				 -			
Co(II)	10	2.0	10	8			0.03		
Cu(II)	15	5.0	10	8		0.4	0.006		
Fe(II)	1	0.5			-	_	 `	_	

Table 2

Sensitivity limits (µg) of metal ions with various spray reagents

Reagents: I β -resorcylaldehyde; II = o-vanillin oxime; III = quercetin; IV = 8-hydroxyquinoline; V = rubeanic acid; VI = morin; VII = chromatropic acid.

10

10

10

10

10

All the metal ions showed coloured spots on spraying with the reagent. The transition metal ions gave intense colour reactions.

10

10

30

Fe(III)

Mn(II)

Ni(II)

Ti(IV)

U(VI)

V(V)

0.6

5.0

2.0

1.0

10

The results of the sensitivity limits for the ten metal ions reveal that in the UV light the reagent is quite effective for concentrations ranging from $0.5 \mu g$ to $10 \mu g$. For comparison, the corresponding limits are given for the reagents o-vanillin oxime⁶, quercetin⁷, 8-hydroxyquinoline⁷, rubeanic acid⁷, morin⁷ and chromatropic acid⁷.

The compound can, therefore, be used as a useful spray reagent for the identification and distinction of metal ions by paper chromatography using proper solvent systems. The possible use of the reagent as a spectrophotometric and gravirnetric reagent for the determination of metal ions and the nature of the coloured complexes formed by the reagent is under investigation.

The results of this paper were presented at the Annual Convention of Chemists in Jaipur, 1977.

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10.0

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THE OCCURRENCE OF FIBROUS ATRETIC FOLLICLES IN THE OVARIES OF RANA CYANOPHLYCTIS AND RANA TIGERINA

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FOLLICULAR atresia in the vertebrate ovary has recently been reviewed 1+2. While it is a common feature of vertebrate ovaries the mode of follicular atresia differs 2. The common type of atresia involves the hypertrophy and hyperplasia of granulosa cells; they become phagocytic, invade the egg/oocyte and digest its contents leaving behind pigment matter and thecal elements 2. Occasionally, 'bursting atresia' has been observed in some non-mammalian vertebrates other