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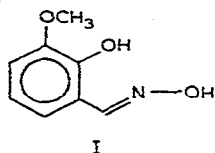
o-Vanillin oxime as a chromatographic spray reagent for metal ions

M. L. DHAR, S. M. JAIN and RAVINDER RAINA

Department of Chemistry, University of Jammu, Jammu-1 (India)

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o-Vanillin oxime (I) has been employed as a gravimetric reagent for the estimation of palladium¹. It has now been found to give colour reactions with 25 metal ions on a paper chromatogram, and is sensitive for the detection of at least 13 of these ions. The compound can therefore be employed as a useful chromatographic spray reagent.



EXPERIMENTAL

o-Vanillin oxime was prepared by the method of Singh and co-workers¹⁻³ by refluxing *o*-vanillin with hydroxylamine hydrochloride. The product *o*-vanillin oxime (I) was purified by crystallization from water until a single spot was obtained on thin-layer chromatography (TLC); the melting point of the compound was 123° (ref. 1, 123°).

A 1% solution of the reagent I in methanol was used for spraying. Whatman No. 3 filter paper was used in each case. The solvent system employed was *n*-butanol-6 *N* hydrochloric acid (1:1). The chromatograms were exposed to ammonia before spraying with the reagent. Ascending chromatography was employed. Sensitivity limits were determined by the usual procedure in visible and UV light.

RESULTS AND DISCUSSION

The colours given by the 25 metal ions in visible and UV light are listed in Table I. All of the metal ions showed coloured spots on spraying with the reagent. The transition-metal ions titanium(IV), vanadium(III), manganese(II), cobalt(II), nickel(II), copper(II), molybdenum(VI), palladium(II), platinum(IV), thorium(IV) and uranium(VI) gave particularly distinct colour reactions. Non-transition-metal ions were not complexed by the reagent to the same extent and the spots shown were either weak in intensity or white, *e.g.*, in the cases of calcium(II) and strontium(II).

TABLE I

COLOUR REACTIONS OF METAL IONS IN VISIBLE AND UV LIGHT

lmy = Lemon yellow, og = olive green, yg = yellow green, grg = grey green, c = chocolate, lg = light green, lb = light brown, ly = light yellow, b = brown, gr = grey, y = yellow, br = brick red, w = white, lv = light violet, dv = dark violet, v = violet and fbw = fluorescent bluish white.

No.	Metal ion	Spot colour	
		Visible	UV
1	Titanium(IV)	lmy	lv
2	Vanadium(IV)	og	dv
3	Chromium(III)	yg	v
4	Manganese(II)	grg	dv
5	Iron(III)	c	v
6	Cobalt(II)	c	dv
7	Nickel(II)	lg	v
8	Copper(II)	lb	dv
9	Molybdenum(VI)	ly	v
10	Palladium(II)	ly	v
11	Tungsten(VI)	b	v
12	Platinum(IV)	gr	lv
13	Gold(III)	gr	lv
14	Mercury(II)	gr	lv
15	Tin(II)	gr	lv
16	Lead(II)	gr	lv
17	Antimony(V)	gr	lv
18	Arsenic(V)	gr	lv
19	Bismuth(III)		lv
20	Selenium(IV)	ly	lv
21	Thorium(IV)	y	lv
22	Uranium(VI)	br	dv
23	Calcium(II)	w	fbw
24	Strontium(II)	w	fbw
25	Beryllium(II)	yg	fbw

TABLE II

SENSITIVITY (μg) OF METAL IONS WITH VARIOUS SPRAY REAGENTS

Reagents: I = *o*-vanillin oxime; II = quercetin; III = 8-hydroxyquinoline; IV = rubeanic acid; V = morin; VI = thoronol; VII = chromotropic acid; VIII = diphenylcarbazide; IX = quinalizarin.

No.	Metal ions	Reagent								
		I	II	III	IV	V	VI	VII	VIII	IX
		Visible	UV	Visible	Visible	Visible	Visible	Visible	Visible	Visible
1	Titanium(IV)	10	6	—	—	—	0.01	—	0.5	—
2	Vanadium(IV)	10	4	0.25	0.5	0.25	—	—	—	—
3	Chromium(III)	50	8	—	—	—	—	—	0.25	—
4	Manganese(II)	10	6	—	—	—	—	—	—	—
5	Cobalt(II)	10	8	—	—	0.03	—	—	—	—
6	Nickel(II)	10	6	—	—	0.012	—	—	—	—
7	Copper(II)	10	8	—	0.4	0.006	—	—	—	—
8	Palladium(II)	10	4	—	—	—	—	—	—	—
9	Selenium(IV)	10	6	—	—	—	—	—	—	—
10	Thorium(IV)	10	6	—	—	—	—	545	—	—
11	Uranium(VI)	10	6	3	10	3	—	—	—	—
12	Iron(III)	10	4	3	10	—	—	—	—	—
13	Beryllium(II)	50	10	—	—	—	0.07	—	—	0.14

The sensitivity limits for 13 of the ions are given in Table II. In the UV, this reagent is quite effective for concentrations ranging from 4 to 10 μg . For comparison, the corresponding limits where known⁴ are given for the reagents quercetin, 8-hydroxyquinoline, rubeanic acid, morin, thoronol, chromatropic acid, diphenylcarbazide and quinalizarin.

The compound can be used as a spray reagent for the identification and differentiation of metal ions by paper chromatography with suitable solvent systems. The nature and composition of some of the chelates formed by the reagent are under investigation.

REFERENCES

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