

(1:1) without any solvent at 80° C for 2 hours. On cooling, crystallized 2-methyl-5-phenyl- $\Delta^{2,3}$ -oxadiazine-4,6-dione quantitatively.

(Found: C-58, 37%; H-4, 08% and N-13, 95%).

(Calculated: C-58, 82%, H-3, 92%, N-13, 72% and O-23, 5%).

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1. "The analogy of this compound can be seen from the table 12, Vllrich, H., *Cycloaddition Reactions of Heterocumulenes* (Academic Press, 1967), see also Goerdeler, J. and Schenk, H., *Chem. Ber.* 1965, 98, 2954 and 3831.
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### 3-PHENYL-4, 5, 7-TRIHYDROXY-COUMARIN AS A CHROMATOGRAPHIC SPRAYING REAGENT IN INORGANIC CHEMICAL ANALYSIS

3-Phenyl-4, 5, 7-trihydroxycoumarin gives coloured complexes with ten transition metal ions and serves as a valuable spraying reagent in their chromatographic analysis.

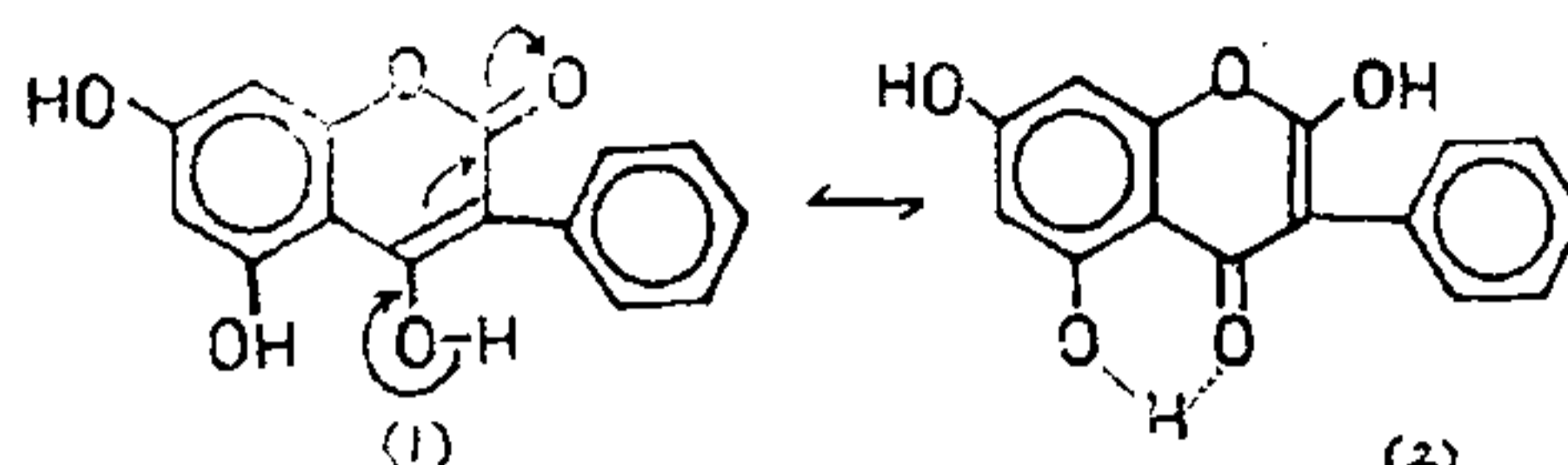
3-Phenyl-4-hydroxycoumarins<sup>1</sup> have long been known synthetically but their recognition in nature was made only in 1964<sup>1</sup>. Since that time the chemistry of such compounds has been studied more intensively. A very important property of this system is that it is tautomeric with 2-hydroxyisoflavone.<sup>2</sup> The latter structure seems to be preferred particularly when 5-hydroxyl is present because chelation stabilises it. Hence it was considered to study the colour reactions of 3-phenyl-4, 5, 7-trihydroxycoumarin with various metal ions. A systematic study indicated that a number of transitional metal ions give varied and very vivid colours as shown in Table I.

Consequently 3-phenyl-4, 5, 7-trihydroxycoumarin was tested for its use as a spraying reagent in the chromatographic analysis of these metal ions. It was observed that in each of the ten cases the metal ions could be spotted out by this reagent. The advantages of this reagent are the following:

(i) Generally a single spraying reagent is difficult to be found for the above-mentioned ten metal ions; whereas 3-phenyl-4, 5, 7-trihydroxycoumarin offers an advantage in

that many of these metal ions together can be detected on a single chromatogram.

TABLE I



Metal ion		Colour with the reagent	
1	Titanium (IV)	..	Golden yellow
2	Vanadium (V)	..	Slate grey
3	Manganese (II)	..	Greenish yellow
4	Iron (II)	..	Chocolate
5	Iron (III)	..	Chocolate
6	Copper (II)	..	Dirty green
7	Molybdenum (VI)	..	Lemon yellow
8	Gold (III)	..	Greyish violet
9	Uranium (VI)	..	Light orange
10	Cerium (III)	..	Dark brown

(ii) The following mixtures which are generally difficult to be identified may be easily done so with the use of this reagent; (a) cerous and ceric, (b) cerium and thorium, (c) thorium and uranium, (d) ferrous and ferric, (e) molybdenum in the presence of chromium and tungsten, and (f) copper and gold in the presence of silver.

(iii) The reagent may be used for quick chromatographic identification of certain metal ions in the common qualitative analysis scheme. For example, in group II gold, copper and molybdenum may be identified in the presence of all other elements of this group; similarly in group III-A iron, titanium, cerous, manganese, vanadium and uranium and in group III-B manganese alone.

Paper chromatography either circular or ascending was done by using different solvent systems described in literature<sup>2</sup>. 3-Phenyl-4, 5, 7-trihydroxycoumarin was prepared according to the method given by McGookin and Robertson<sup>3</sup>. Its 0.5-1.0% alcoholic solution was employed as the spraying reagent.

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