VANILLIN DERIVATIVE SPRAY REAGENTS FOR IDENTIFICATION OF ORGANIC COMPOUNDS ON THIN LAYER CHROMATOGRAMS.

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ABSTRACT

Several derivatives of vanillin have been synthesized and tested as spray reagents for identification of some natural products on thin layer chromatograms. Colours produced on chromatographic plates were of shades of the spectrum, and characteristic for a good number of the compounds tested. The series of spray reagents are suitable, not only for identification, but also, for preparative chromatography. The reagents may also find application in the analysis of food and pharmaceutical products.

INTRODUCTION

In spite of many spray reagents described as visualisation reagents for the identification of organic compounds, we have found too few reports on derivatives of vanillin. This motivated us to carry out the present study so as to widen the scope of colour spectrum for vanillin and vanillin derivative spray reagents.

The use of conventional visualisation reagents like $KMnO_4$, H_2SO_4 etc and ultraviolet light is routine[1,2,3], but the sensitivity, specificity and convenience of colour - producing spray reagents like vanillin and vanillin mixtures[4,5,6] make the latter more attractive and are often preferred to the conventional spray reagents.

Encouraged by the extensive investigation carried out by Picman et al.[4] on vanillin, we have prepared and tested other derivatives of vanillin. These derivatives widened the scope of colour reactions obtainable on thin layer chromatograms (tlc) hitherto not obtainable with vanillin itself. The organic compounds identified with the aid of these vanillin derivative spray reagents include those earlier tested by picman and coworkers. In addition, natural products have been identified and isolated directly from crude extracts on preparative basis with the aid of these spray reagents.[4]

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EXPERIMENTAL

General

The natural compounds used as test samples were either isolated fresh or supplied. Chemicals were used without further purification and all solvents were redistilled before use. Infrared (ir) spectra were recorded on a Beckman infrared spectrophotometer. Melting points were determined on a digital melting point apparatus. Qualitative tlc was run with precoated Merck silica gel plates (20 x 20cm, 0.2mm layer thickness: Merck, Darmstadt, Germany).

Synthesis

The synthesis of the various vanillin derivatives is as shown in Fig. 1.

Vanillin derivatives

Vanillic acid (II)

Vanillin (I) (1.5g, 0.0098 mole) was dissolved in 3cm^3 acetone. The resulting solution was cooled in ice-water and oxidised with 1.2cm^3 cold solution of acidified sodium dichromate. After the usual work-up, a pink crystalline solid, m.p. 208-209°C (Lit. 207 - 210°C) was obtained, ir (KBr, cm^{-1}) 3400 -

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