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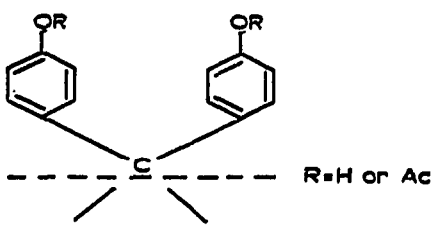
Thin-layer chromatographic investigations of some synthetic compounds having laxative properties

In the past few years, synthetic laxatives have been more widely used in pharmacy than those derived from plants. (The reasons are too obvious to be discussed here.) 4,4'-Dihydroxydiphenylisatin or its acetyl derivatives, well-known for their laxative properties, and a few other related substances are studied herein. Quality control was necessary at various stages of production, and thin-layer chromatography (TLC) proved to be a very suitable method for this purpose. Because very few publications[1–4] have appeared in this field, a systematic investigation of such substances by TLC was considered worthwhile. In all these compounds, the dihydroxy- (or diacetoxy-)diphenyl group was a common feature, as shown below.



Experimental

Thin-layer plates approx. 0.25 mm thick were prepared manually[5] or using an applicator. The adsorbent used was Silica Gel GF Woelm TLC. The plates could be used after drying at room temperature for about 24 h. TLC plates of Woelm precoated with Silica Gel F 254/366 could be used with equal success.

All substances, except phenolphthalein, were synthesized. While thiazolyl derivatives (compounds 8–11) were prepared according to GEIGER *et al.*[6], 4,4'-dihydroxydiphenyl-pyrid-2-yl-methane and its diacetyl derivative were synthesized according to SEEGER AND KOTTLER[7].

The following solvent systems were employed: (I) chloroform–acetone (1:1); (II) chloroform–cyclohexane–ethyl methyl ketone (1:1:1); (III)chloroform–benzene–ethyl methyl ketone (1:1:1); (IV) carbon tetrachloride–cyclohexane–ethyl methyl ketone (1:1:1); (V) carbon tetrachloride–benzene–ethyl methyl ketone (1:1:1); (VI) chloroform–benzene–acetone (1:1:1).

The solvent front was allowed to advance 10 cm from the starting line. After development, the plates were dried and then viewed at 254 mμ under an UV lamp. The separated substances showed up as dark spots against a greenish fluorescent background. Table I shows the R_F values of various substances on plates prepared manually.

Discussion

In these experiments, it was found that for compounds 1–7 and compounds 8–11 solvent systems VI and II, respectively, proved to be most suitable. The separations were equally good whether the thin-layer plates were prepared manually or using an applicator or whether TLC plates of Woelm precoated with Silica Gel

TABLE I

No.	*Substances*	*Solvent systems*					
		I	*II*	*III*	*IV*	*V*	*VI*
1	Phenolphthalein	0.80	0.39	0.61	0.28	0.64	0.57
2	Diacetoxyphenolphthalein	0.93	0.75	0.86	0.58	0.82	0.79
3	4,4'-Dihydroxydiphenyl-pyrid-2-yl-methane	0.76	0.14	0.19	0.13	0.22	0.35
4	4,4'-Diacetoxydiphenyl-pyrid-2-yl-methane	0.94	0.59	0.65	0.51	0.57	0.74
5	4,4'-Dihydroxydiphenylisatin	0.66	0.15	0.20	0.10	0.27	0.29
6	4,4'-Diacetoxydiphenylisatin (Diphesatin)	0.91	0.50	0.61	0.35	0.57	0.69
7	4,4'-Diacetoxydiphenyl-N-acetoxyisatin (Trisatin)	0.94	0.83	0.92	0.74	0.88	0.86
8	4,4'-Diacetoxydiphenyl-4-methylthiazol-2-yl-methane	0.84	0.65	0.79	0.49	0.72	0.85
9	4,4'-Dihydroxy-3,3'-dimethyldiphenyl-4,5-dimethylthiazol-2-yl-methane	0.69	0.40	0.53	0.27	0.44	0.63
10	4,4'-Dihydroxydiphenyl-4-ethyl-5-methyl-thiazol-2-yl-methane	0.66	0.35	0.49	0.23	0.42	0.59
11	4,4'-Dihydroxydiphenyl-4-methyl-5-ethyl-thiazol-2-yl-methane	0.61	0.30	0.42	0.20	0.36	0.53

F 254/366 were used. R_F values on plates prepared manually were slightly higher than on precoated plates or on those prepared using an applicator. Acetyl derivatives showed R_F values greater than those of their corresponding hydroxyl compounds. The increasing number of acetyl groups on the molecule enhanced the corresponding R_F values.

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