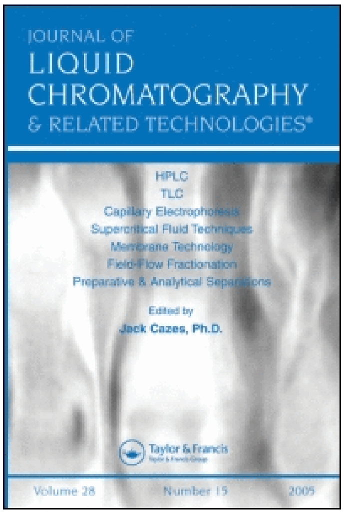

Journal of Liquid Chromatography & Related Technologies
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713597273

Identification of Some Potential Antidiabetic Compounds on Impregnated Silica Gel G Platea as Their π-Complexes

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To cite this Article Jain, Rajeev and Agarwal, D. D.(1983) 'Identification of Some Potential Antidiabetic Compounds on Impregnated Silica Gel G Platea as Their π-Complexes', Journal of Liquid Chromatography & Related Technologies, 6: 14, 2661 — 2664

To link to this Article: DOI: 10.1080/01483918308064935

URL: http://dx.doi.org/10.1080/01483918308064935

JOURNAL OF LIQUID CHROMATOGRAPHY, 6(14), 2661-2664 (1983)

IDENTIFICATION OF SOME POTENTIAL ANTIDIABETIC COMPOUNDS ON IMPREGNATED SILICA GEL G PLATEA AS THEIR π-COMPLEXES

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ABSTRACT

A rapid and convenient procedure for the detection of potential antidiabetics as π complexes with aromatic amines is described. The method could be of immense value even in places where normal laboratory facilities are not available.

INTRODUCTION

The field of charge transfer complexes has been investigated recently by several chromatographic techniques. Unsaturated lipids have been resolved on columns of solica gel impregnated with silver nitrate (1). Thin-layer chromatography has been employed in the investigations of the complexes of silver with terpenes (2), glycerides (3) and silylic-propenylic isomers (4). The separation of the π - complexes of m-dinitrobenzene, 2, 4-dinitrotoluene, 2,4,5-trinitrotoluene and 2,4,6-trinitroanisole with some hydrocarbons on TLC plates has been carried out (5). Nitroaromatic compounds form π - complexes with aromatic donors (6) due to polarization of the nitro group. These π complexes involve hybrid structures with bedding of the filled Sp hybridized orbital on the nitrogen atom in the amine compound with the vacant p orbital on the nitrogen of the nitro group, e.g.

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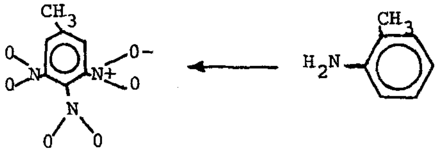
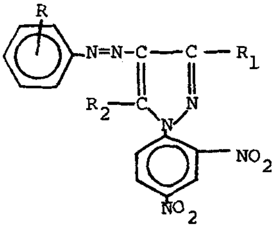


More recently it has been suggested that during the formation of 1:1 π complexes of the m-dinitrobenzene with various aminobenzoic acids only one nitro group plays role (7). The majority of the complexes are unstable. Solvent and adsorption forces, steric hindrance, temperature etc., weaken the charge transfer linkages and make their study difficult.

The present papers describes a simple and convenient method for the identification of trace amounts of 1-(2,4-dinitrophenyl)-3,5-dimethyl/diphenyl-4-arylazopyrazoles(A),which are well known as potential antidiabetic agents, as charge-transfer complexes with aromatic amines employing TLC technique. The resolved π complexes being highly coloured could be located easily on the chromatoplates. It was possible to identify as little as 1-2 μg of these compounds as their π complexes.



(A)

where R represents various substituents, $R_1=R_2+CH_3$ or C_6H_5

EXPERIMENTAL

The TLC plates (thickness 0.5 mm) where prepared by spraying a slurry of a mixture of 50g of silica gel G+1 g of aromatic amine in 100 ml of distilled water and drying for 24 hrs at a constant temperature of 60°C. The compounds (I-XX)

TABLE - 1

Sl No.	R	R_1	R_2	Rf x 100				Detection limit (µg)
				A	B	A	B	
				Plain Silica Gel G		Silica Gen C+p-Toludine		
I	H	CH_3	CH_3	44	52	36	46	2.0
II	2-CH_3	CH_3	CH_3	55	49	29	40	1.5
III	3-CH_3	CH_3	CH_3	37	50	32	32	2.0
IV	4-CH_3	CH_3	CH_3	31	54	24	36	3.5
V	2-CI	CH_3	CH_3	21	28	20	29	2.0
VI	3-CI	CH_3	CH_3	22	34	21	23	1.5
VII	4-CI	CH_3	CH_3	25	35	16	19	1.5
VIII	2-OCH_3	CH_3	CH_3	39	42	41	52	2.5
IX	3-OCH_3	CH_3	CH_3	45	48	45	58	2.5
X	4-OCH_3	CH_3	CH_3	46	51	50	54	1.5
XI	H	C_6H_5	C_6H_5	30	46	28	42	1.0
XII	2-CH_3	C_6H_5	C_6H_5	27	40	23	38	1.5
XIII	3-CH_3	C_6H_5	C_6H_5	29	38	20	28	2.0
IXV	4-CH_3	C_6H_5	C_6H_5	32	32	18	31	2.0
XV	2-CI	C_6H_5	C_6H_5	09	16	07	19	1.5
XVI	3-CI	C_6H_5	C_6H_5	13	17	11	23	1.5
XVII	4-CI	C_6H_5	C_6H_5	15	20	13	16	2.0
XVIII	2-OCH_3	C_6H_5	C_6H_5	26	23	31	45	1.5
XIX	3-OCH_3	C_6H_5	C_6H_5	28	28	26	54	2.0
XX	4-OCH_3	C_6H_5	C_6H_5	33	30	35	51	2.0

* For p-toluidine impregnated plates
a - Average of two identical runs

Solvant Systems: (A) Xylene (60/): Chloroform (30/):Acetone (10/).
(B) Benzene (55/):Chloroform(40/): Ethyl methyl ketone(5/)

Rate of Development: 15 cm in 40 min for plain silica gel plates and 15 cm in 50 min for p-toluidine impregnated silica gel plates.

in acetone (0.05/ w/v) were applied to the chromatographic plates using glass capillary and the chromatograms were eluted with various solvents systems but the best resolution was obtained with solvent systems A and B (Table-1).

After thorough research it was found that a mixture of p-toludine and silica gel gave the most satisfactory separation. The results are presented in Table-1. For comparison the values on plain silica gel plates under similar conditions are also given. It can be seen that out of 20 pyrazoles only 12 can be separated on plain silica gel G plates. Further, it may be pointed out that the Rf-values are changed when a mixture of pryazoles was applied.

RESULTS AND DISCUSSION

The resolution of π complexes was found to be governed by three factors (i) adsorbent (ii) nature of the π donor (aromatic amine) and (iii) irrigating solvent. In general the migration of these compounds were in relation to the effect of electron attracting or repelling groups present in them.

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