Radio thin layer chromatography of some aromatic phosphorus and arsenic compounds

In a previous paper[1] the determination of R_F values of some organic phosphorus and arsenic compounds by thin layer chromatography has already been reported. These experiments, which have proved the TLC technique to be an efficient tool for the separation and simultaneous identification of these compounds by isotopic indication, have been continued and information on further types of compounds and adsorbents has been obtained.

Experimental

For coating the glass plates silica gel G (Merck), neutral alumina (Woelm) and silica gel (Merck), finely ground for this purpose, were used in turn. The coated plates were dried, in the case of gypsum content at 120°, for starch at 105°, each for 2 h. The R_F values were determined on 20 cm × 20 cm glass plates and in addition on 3 cm × 20 cm glass strips used in the radiometric evaluation. No appreciable difference was found between the R_F values obtained on plates of various sizes. The ascending technique was employed in glass tanks with ground-in lids. The time of development varied from 30 to 60 min. Elementary iodine was used for the detection of spots. The radio thin-layer chromatograms were scanned by an automatic recorder.

Results

In Tables I and II the R_F values of the compounds chromatographed on silica gel G and neutral alumina are shown as obtained for various solvent systems. In

TABLE I

R_F × 100 VALUES ON SILICA GEL G ADSORBENT

Compound	R_F × 100								
	S_1	S_2	S_3	S_4	S_5	S_6	S_7	S_8	S_9
Triphenyl phosphate	93	79	9	24	94	87	79	91	93
Triphenyl phosphite	95	81	18	—	—	—	—	—	—
Triphenylphosphine oxide	84	54	0	0	55	9	16	70	64
Triphenylphosphine	94	73	21	92	95	95	94	95	95
Tricresyl phosphate	95	90	0	84	90	88	87	89	94
Triphenylarsine	96	75	39	89	95	94	93	91	94

Solvent systems: S_1 = acetone; S_2 = acetone ("oversaturated"); S_3 = petroleum ether; S_4 = benzene; S_5 = benzene–acetone (1:1); S_6 = benzene–acetone (9:1); S_7 = chloroform; S_8 = chloroform–acetone (6:4); S_9 = chloroform–acetone (9:1).

Table III the R_F values of triphenylphosphine oxide are listed for various adsorbents and acetone solvent. In the experiments silica gel G proved to give the most satisfactory coating mainly because of very good mechanical behaviour (uniform, very adhesive to glass and resistant to solvents).

TABLE II

$R_F \times 100$ VALUES ON NEUTRAL ALUMINA ADSORBENT

Compound	$R_F \times 100$								
	S_1	S_2	S_3	S_4	S_5	S_6	S_7	S_8	S_9
Triphenyl phosphate	93	73	0	36	94	90	35	90	91
Triphenyl phosphite	92	70	17	—	—	—	—	—	—
Triphenylphosphine oxide	85	53	0	0	68	13	65	84	75
Triphenylphosphine	95	74	54	93	94	91	91	92	92
Tricresyl phosphate	90	70	0	32	88	88	91	93	91
Triphenylarsine	94	78	75	93	91	94	90	93	94

For the solvent systems see Table I.

TABLE III

$R_F \times 100$ VALUES OF TRIPHENYLPHOSPHINE OXIDE ON VARIOUS ADSORBENTS WITH ACETONE SOLVENT

Adsorbent	$R_F \times 100$
Silica gel G	87
Silica gel G (activated at 160°)	81
Silica gel + 5% gypsum	82
Silica gel + 3% starch	76
Alumina + 3% starch	80
Silica gel–alumina–gypsum (14:14:2)	

Central Research Institute for Physics, Budapest (Hungary)

KLÁRA BEREI

1 K. BEREI, *Közp. Fiz. Kut. Int. Közl.*, 13 (1965) 49.

Received May 11th, 1965

J. Chromatog., 20 (1965) 406–407

Separation of vitamin D esters by thin-layer chromatography*

There is evidence that vitamin D, like other sterols[1,2], is esterified during intestinal absorption[3]. The nature of the esters thus formed is currently being investigated in this laboratory with ^{14}C vitamin D_3 in rats. This communication describes methods for separating esters of vitamins D_2 and D_3 with thin-layer chromatography.

Methods

Esters were prepared by a modification of the method of KUKSIS AND BEVERIDGE[4] either from commercially available fatty acid chlorides (butyryl, hexanoyl,

* Supported (in part) by U.S.P.H.S. Grant No. AM 09016-01 and (in part) by U.S.P.H.S. General Research Support Grant No. 1-S01-FR-05070-01.