Kieselgel G treated with 6 *N* HCl produced lower migrations of the compounds compared to Kieselgel G when xylene and toluene were employed as solvents.

All the three types of alumina proved to be excellent adsorbents for 2,4-dinitrophenyl derivatives. In general the mobilities of these compounds on basic alumina were lower than on neutral alumina.

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The detection and identification of antioxidants containing nitrogen in synthetic aviation lubricants after separation by thin layer chromatography

A paper chromatographic method for the detection and identification of nitrogen-containing antioxidants has been reported previously[1]. Subsequently, it was found that a disadvantage of this method was that Aroclor 1254, which is sometimes present in synthetic aviation lubricants as an extreme pressure (E.P.) agent, interferes with the detection of some of the antioxidants. Other additives and complex organic molecules which are present, *e.g.* polyalkylene oxides, esters derived from mono- and dibasic acids, triaryl phosphates and silicone anti-foaming agents do not interfere. To overcome the difficulty of detecting antioxidants in the presence of Aroclor 1254, a thin-layer chromatographic method has been developed.

Experimental and results

Preliminary experiments using neutral and basic layers of silica gel showed that better separations, and increased R_F values, could be obtained on basic layers. The antioxidants studied and the R_F values obtained using two solvent systems are given in Table I.

TABLE I

COMPARISON OF R_F VALUES OF ANTIOXIDANTS USING NEUTRAL AND BASIC LAYERS OF SILICA GEL
Solvent A = chloroform–cyclohexane (30:70, v/v); solvent B = benzene–*n*-hexane (40:60, v/v).

Antioxidant	*Solvent A*		*Solvent B*	
	Basic	*Neutral*	*Basic*	*Neutral*
Diphenylamine	0.50	0.37	0.39	0.34
Di-(*p*-octylphenyl)amine	0.65	0.53	0.65	0.59
Phenothiazine	0.34	0.22	0.25	0.22
3,7-Dioctylphenothiazine	0.59	0.43	0.52	0.47
N-Phenyl-α-naphthylamine	0.53	0.37	0.43	0.36
N-Phenyl-β-naphthylamine	0.46	0.31	0.34	0.30
Di-2-pyridylamine	0.04	0.0	0.01	0.0

Preparation of chromatoplates. Glass plates, 20 × 10 cm, were coated with Kieselgel "G" prepared by slurrying 1 part gel with 2 parts of aqueous sodium hydroxide (*N*/2). The thin layers (275 μ) were activated for 30 min at 110°, and stored in the presence of silica gel desiccant until required.

Solvent system. Various solvent combinations were examined for the separation of the antioxidants on thin layers of basic silica gel. The solvent system benzene–*n*-hexane (40:60, v/v) gave the best separation, and it was employed in the subsequent investigations.

Spray reagents. Location of the zones was achieved by spraying the chromatoplate with a solution of 20% v/v antimony pentachloride in carbon tetrachloride. More highly coloured derivatives were obtained with this reagent than with *p*-nitrobenzenediazonium fluoborate which was used in the paper chromatographic method.

Procedure. A reference blend of the antioxidants in acetone was prepared to contain approximately 2 μg of each antioxidant per μl of solution. The lubricants under investigation were also diluted with acetone to give approximately the same concentration of antioxidants as in the reference blend.

Chromatography was carried out in the usual way, using approximately 1 μl of each solution, and a solvent development of 10 cm. The separating tank was lined with filter paper and 20 min. were allowed for equilibration of the solvent and vapour phases.

The antioxidants were revealed as brightly coloured spots on spraying with

TABLE II

THE DETECTION AND IDENTIFICATION OF SOME NITROGEN-CONTAINING ANTIOXIDANTS

Antioxidant	*Colour-$SbCl_5$ spray*	*R_F value*
Diphenylamine	Blue turns green	0.39
Di-(*p*-octylphenyl)amine	Brown	0.65
Phenothiazine	Brown-green	0.25
3,7-Dioctylphenothiazine	Red-orange	0.52
N-Phenyl-α-naphthylamine	Blue	0.43
N-Phenyl-β-naphthylamine	Dark grey	0.34
Di-2-pyridylamine	Yellow	0.01

antimony pentachloride solution. Sensitivity tests showed that 1 μg of each antioxidant was readily detected.

The R_F values and colours obtained on the reference blend of antioxidants are given in Table II.

TABLE III

THIN LAYER CHROMATOGRAPHIC ANALYSIS OF FOUR SYNTHETIC AVIATION LUBRICANTS

Lubricant	*Observations*			*Inference*
	No. of spots	*Colour-$SbCl_5$ spray*	*R_F value*	
1	1	Brown-green	0.25	Phenothiazine
2	2	Brown-green	0.25	Phenothiazine
		Blue	0.43	N-Phenyl-α-naphthylamine
3	2	Brown-green	0.25	Phenothiazine
		Blue→green	0.39	Diphenylamine
4*	2	Red-orange	0.52	3,7-Dioctylphenothiazine
		Brown	0.65	Di-(*p*-octylphenyl)amine

* The antioxidants in this lubricant could not be detected by the paper chromatographic method[1], because of the presence of Aroclor 1254.

The results obtained on four synthetic aviation lubricants are given in Table III. The lubricants were selected because they contained different base esters, E.P. agents (including Aroclor 1254) and other additives. There was no interference during chromatography, as evidenced by the R_F values, or with the method of detection.

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