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Note

Detection and significance of glycols in drug screening

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Drug screening, in a forensic toxicology laboratory, is most conveniently done by thin-layer chromatography (TLC) of biological extracts. The solvent systems and drug-locating sprays are now fairly standard. The procedure adopted in our laboratory[1] has given us a slight "bonus" in that glycol derivatives can also be detected.

Basic/neutral drugs are run on silica gel 60 F_{254} (Merck precoated glass plates) in ammonia–methanol (1:99) and are detected by a sequential spray technique, part of which involves spraying with Dragendorff's spray followed by 5% sodium nitrite. The Dragendorff's spray will react with the glycols forming an orange-red spot similar to a basic drug. The nitrite will cause the spot to change to deep purple, then to a grey, and finally fading to a yellow (the last stage often taking hours).

Drugs are extracted directly from blood or from a tissue hydrolysate using chloroform. The glycols will partition into chloroform under any pH condition and consequently will be found with the neutral compounds. Polyethylene glycols and glycol ethers are the two groups which have been encountered and studied.

POLYETHYLENE GLYCOLS

Polyethylene glycols (PEGs) are polymers of ethylene oxide having the general formula: $HO–CH_2–(CH_2–O–CH_2)_n–CH_2–OH$. Commercial PEGs are mixtures of glycols of various molecular weights normally distributed about a mean value and are named according to this mean value (*e.g.*, PEG-400). They are water soluble, viscous, relatively non-toxic and excellent vehicles for drug preparations. The polymers may also increase drug absorption and activity *in vivo*.

When located on a TLC plate, these glycols appear as a trail, usually extending to R_F 0.45. The trail is inevitable because of the mixed nature of the glycol. Concentration on the plate plays a major part in determining R_F values (*cf.* Table I).

The quantities given in Table I represent overloading of the plate but are the amounts which would normally be seen during screening. The sensitivity of the sprays is quite good: Dragendorff's spray will locate about 10 μg of PEG and as little as 1 μg can be seen if nitrite is used. Monoethylene glycol (MEG), diethylene glycol (DEG) and triethylene glycol (TEG) react poorly with the sprays —a yellow-orange spot is observed after spraying. Tetraethylene glycol (Tetra) is the first in the series to exhibit the purple/grey reaction with nitrite. Typical R_F values for 1-μg quantities are listed in Table II.

TABLE I

R_F VALUES FOR DIFFERENT AMOUNTS OF PEG-600 APPLIED TO A TLC PLATE

	Amount applied (μl)					
	0.1	*0.2*	*0.3*	*0.5*	*0.8*	*1.0*
R_F value	0.23	0.29	0.33	0.35	0.38	0.38

TABLE II

R_F VALUES FOR 1-μg AMOUNTS OF SEVERAL POLYETHYLENE GLYCOLS APPLIED TO A TLC PLATE

	MEG	*DEG*	*TEG*	*Tetra*	*PEG-200*	*PEG-400*	*PEG-600*	*PEG-800*	*PEG-1500*
R_F value	0.51	0.51	0.51	0.50	0.48	0.42	0.38	0.38	0.12

GLYCOL ETHERS

Glycol ethers have the general formula: $HO–CH_2–(CH_2–O–CH_2)_n–CH_2–OX$, where X is usually a methyl, ethyl, or butyl group. Hydraulic fluids are commonly composed of glycol ethers and n rarely exceeds 5. A brake fluid poisoning case brought this group of glycols to our notice when it was found that TLC spray reactions were similar to those of the PEGs. No pure standards have been available but useful results have been obtained with a formulated brake fluid. Three distinct spots were observed, one at an R_F value of 0.50 corresponding to the low-molecular-weight PEGs while the two higher R_F values (0.67, 0.79) would be due to the glycol ethers: The principal constituents (65%) were triethylene glycol methyl ether and tetraethylene glycol methyl ether.

CONCLUSION

Glycols may be detected with Dragendorff's reagent and in drug screening their reaction is similar to a basic drug. If sodium nitrite is sprayed immediately after Dragendorff's reagent a distinctive reaction is observed. Awareness of this reaction has broadened the scope of our drug screening: Evidence of PEGs suggests that a pharmaceutical preparation has been taken, while glycol ethers suggest hydraulic fluids or an industrial solvent. It is assumed that propylene glycols will behave similarly.

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1 A. E. Hodda, *Proc. Nat. Symp. Forensic Sci., 3rd, Sydney, 1973.*