

**Determination of Polyoxyethylene Glycol in Non-Ionic Detergents**

SIR,—When ethylene oxide is polymerised in the presence of a long chain alcohol, ROH, to give a non-ionic detergent,  $R(\text{OCH}_2\text{CH}_2)_m\text{OH}$ , in which  $m$  is the designated number of ethylene oxide units, traces of water in the reaction mixture can lead to the formation of polyoxyethylene glycols as by-products (Ginn, Church and Harris, 1961; Drew and Schaeffer, 1958). Several methods have been devised for estimating the amount of glycol formed (Ginn and others, 1961; Weibull, 1961), but these involve separating the glycol from the detergent. A means of estimating the glycol content without separation, based on two assay methods, is reported here.

The first method is that of Siggia, Starke, Garis and Stahl (1958) and determines the total ethylene oxide content of the mixture of glycol and detergent. In a mixture of weight,  $W_t$ , containing  $W_1$  g. glycol of mean molecular weight  $M_1$ , and  $W_2$  g. detergent of mean molecular weight  $M_2$ , the weight of ethylene oxide,  $z$  g. is

$$z = \frac{W_1(M_1 - 18.01)}{M_1} + \frac{W_2(M_2 - y)}{M_2} \dots \dots \dots (1)$$

where  $y$  = molecular weight of the long chain alcohol. In the present instance the detergents are based on hexadecyl alcohol, m.w. 242.45. The second assay method is the pharmacopoeial method for the assay of hydroxy groups in benzyl alcohol (B.P. 1958) but in which the time of heating is increased until the results are constant (6–8 hr.). This is used to determine the percentage of hydroxy groups present in the mixture. If  $W_t$  g. of mixture contains  $x$  m-equiv. of hydroxyl then:

$$x = 2000W_1/M_1 + 1000W_2/M_2 \dots \dots \dots (2)$$

Remembering  $W_t = W_1 + W_2 \dots \dots \dots (3)$

and substituting (2) in (1), either  $W_1$  and  $M_1$ , or  $W_2$  and  $M_2$  can be eliminated, giving:

$$n_1 = \frac{W_1}{M_1} = \frac{z - W_t + 0.2424x}{466.89} \dots \dots \dots (4)$$

$$n_2 = \frac{W_2}{M_2} = \frac{2W_t - 0.018x - 2z}{466.89} \dots \dots \dots (5)$$

where  $n_1$  and  $n_2$  are the number of moles of glycol and detergent respectively, and the denominator of the right hand side arises by subtracting the molecular weight of water from twice the alcohol molecular weight. Equations (4) and (5) give a means of determining the number of moles of each component in a chosen weight of mixture.

Prepared mixtures of triethylene glycol and hexaoxyethylene monohexadecyl ether were subjected to the proposed procedure with the following results.

Mixture number	.. ..	1	2	3
Mole per cent glycol added	.. ..	7.1	10.1	15.0
Mole per cent glycol found	.. ..	7.0	10.1	15.3

The results were satisfactory considering assay errors of  $\pm 1$  per cent in the OH determination, and  $\pm 0.3$  per cent in the ethylene oxide assay.

A series of commercially produced detergents based on hexadecyl alcohol (H), and containing  $m$  ethylene oxide units, was assayed.

Detergent	.. ..	Hm <sub>4</sub>	Hm <sub>6</sub>	Hm <sub>10</sub>	Hm <sub>16</sub>	Hm <sub>24</sub>
Mole per cent glycol found	..	0.5	1.2	2.5	5.1	7.0

## LETTERS TO THE EDITOR

The mole percentage of glycol increased as the hexadecyl alcohol was reacted with increasing amounts of ethylene oxide. This increase may be due to the presence of traces of water in the ethylene oxide.

Hm<sub>24</sub> corresponds to Cetomacrogol 1000, and the procedure may be used to determine the polyoxyethylene glycol present in this material.

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### REFERENCES

- Drew, H. F. and Shaeffer, J. R. (1958). *Indust. Engng Chem.*, **50**, 1253-1254.  
Ginn, M. E., Church, C. L. and Harris, J. C. (1961). *Analyt. Chem.*, **33**, 143-145.  
Siggia, S., Starke, A. C., Garis, J. J. and Stahl, C. R. (1958). *Ibid.*, **30**, 115-116.  
Weibull, B. (1961). Third International Congress on Surface Activity, Vol. III, p. 121-124. Mainz: University Press.

### Analeptic Activity of Tremor-producing Amino-Alcohols

SIR,—Ahmed, Marshall and Shepherd (1958) described a series of amino-alcohols capable of inducing tremor when injected into mice. In higher doses, these compounds produced convulsions similar to those of picrotoxin, and consequently two of the compounds have now been tested for analeptic activity against pentobarbitone anaesthesia by the mouse-awakening test of Goodwin and Marshall (1945).

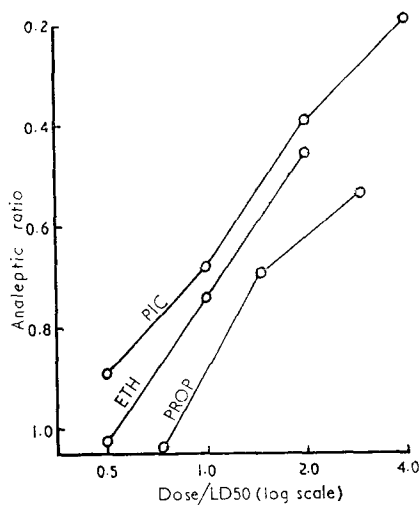


Fig. 1. Awakening time for mice under pentobarbitone anaesthesia. Analeptic ratio = median awakening time for mice receiving analeptic/median awakening time for controls.  
PIC = picrotoxin (from Goodwin and Marshall, 1945);  
ETH = 1,1,2-triphenyl-2-amino-ethanol;  
PROP = 1,1,3-triphenyl-3-amino-propan-1-ol.