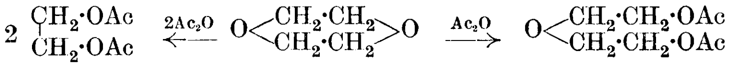
NOTES.

The Acetylation of Diethylene Oxide. By Morna Macleod.

Experiments carried out by Knoevenagel (*Annalen*, 1913, **402**, 133) on the interaction of acetic anhydride with various ethers in the presence of anhydrous ferric chloride suggested a method by which the presence of two oxide rings in ngaione (McDowall, J., 1925, **127**, 2200) might be confirmed and investigated. Some tests were therefore made with compounds of known constitution.

Knoevenagel's results for cineole were verified, and the reaction was then applied to diethylene oxide. Besides some unchanged diethylene oxide, small quantities of glycol diacetate and ββ'-*di-acetoxyethyl ether* were obtained.



$$2\begin{matrix}CH_2\cdot OAc\\ CH_2\cdot OAc\end{matrix}\ \xleftarrow{2Ac_2O}\ O\Big\langle\begin{matrix}CH_2\cdot CH_2\\ CH_2\cdot CH_2\end{matrix}\Big\rangle O\ \xrightarrow{Ac_2O}\ O\Big\langle\begin{matrix}CH_2\cdot CH_2\cdot OAc\\ CH_2\cdot CH_2\cdot OAc\end{matrix}$$

The latter compound was a moderately viscous, sweet-smelling oil, b. p. 110—135°/16 mm., $d_{20^\circ}^{20^\circ}$ 1·123, $n_D^{20^\circ}$ 1·4348, whence $[R_L]_D =$ 44·12 (Found : C, 50·9 ; H, 7·3 ; sap. value, 582. $C_8H_{14}O_5$ requires C, 50·5 ; H, 7·4% ; $[R_L]_D$, 43·98 ; sap. value, 589).—The Ralph Forster Laboratories of Organic Chemistry, University College, London. [*Received, October 8th*, 1928.]

The Nitration of o-*Bromoacetanilide.* By Charles Stanley Gibson and John Dobney Andrew Johnson.

The nitration of *o*-bromoacetanilide proceeds readily according to the method of Franzen and Engel (*J. pr. Chem.*, 1921, **102**, 188), but their description of the separation of the isomeric mononitration products is a little confused. They agitated the finely divided crude nitration product with ice-cold " Witt–Utermann solution " (75 g. of potassium hydroxide, 475 c.c. of water, 100 c.c. of ethyl alcohol). 2-Bromo-6-nitroacetanilide was said to dissolve, and 2-bromo-4-nitroacetanilide was left undissolved. It was further stated that when the solution of the 6-nitro-compound was kept at room temperature for 24 hours, hydrolysis took place and 2-bromo-6-nitroaniline was precipitated.

It has now been found that the crude nitration product is almost completely soluble in the " Witt–Utermann solution," only 2% remaining undissolved. The filtrate becomes turbid and after 15—20 minutes a yellow solid begins to separate, precipitation being complete after 24 hours. This crude product had m. p. 94—98° and after crystallisation from benzene–ligroin (b. p. 60—80°) had m. p. 104°, the melting point of 2-bromo-4-nitroaniline. The nature of this compound was further verified by bromination in acetic acid solution, the dibromonitroaniline, m. p. 206°, obtained being identical with 2 : 6-dibromo-4-nitroaniline prepared by the bromination of *p*-nitroaniline.

The filtrate remaining after the separation of the 2-bromo-4-nitroaniline from the " witt Utermann solution," on treatment with glacial acetic acid gave a precipitate of a substance which crystallised from methyl alcohol in long needles m. p. 193°, the melting point of 2-bromo-6-nitroacetanilide. On boiling a solution of this substance (5 g.) in alcohol (24 c.c.) and hydrochloric acid (12 c.c.) for 3 hours, hydrolysis took place, the 2-bromo-6-nitroaniline having m. p. 74—75° after recrystallisation from ligroin (b. p. 40—60°) (compare Meldola and Streatfeild, J., 1898, **73**, 686, who give m. p. 73—74°).

Chattaway, Orton, and Evans (*Ber.*, 1900, **33**, 3061) obtained 2-bromo-4-nitroacetanilide (m. p. 129°) by the action of acetyl chloride in acetic acid on 2-bromo-4-nitroaniline. Körner (*Atti R. Accad. Lincei*, 1914, **22**, i, 825), using acetic anhydride, obtained a monoacetyl derivative, m. p. 114°, and a diacetyl derivative, m. p. 132°. On repeating the work of the former authors, it was found that the monoacetyl derivative is indeed formed and has m. p. 132—133° (Found : N, 11·1. Calc. : N, 10·8%. The diacetyl derivative requires N, 9·3%). This monoacetyl derivative, contrary to the statement of Franzen and Engel, is readily soluble in " Witt–Utermann solution."

All the *o*-bromoacetanilide used in the work was prepared from *o*-bromoaniline, b. p. 110·5°/19 mm. From 204 g. of pure *o*-bromoacetanilide, 66 g. of pure 2-bromo-4-nitroaniline and 80 g. of pure 2-bromo-6-nitroacetanilide were obtained.—GUY'S HOSPITAL MEDICAL SCHOOL (UNIVERSITY OF LONDON), S.E. 1. [*Received, September 17th,* 1928.]