NOTES.

Electrolytic Reduction of αα′-Dicyano-ββ-dimethylglutarimide.

By G. Gratton and G. R. Ramage.

As a preliminary to the preparation of bridged piperidonecarboxylic acids, which appeared likely to prove of value as a starting point for the synthesis of substituted *cyclo*butane acids, the electrolytic reduction of αα′-dicyano-ββ-dimethylglutarimide was investigated. This gave in poor yield the *lactam* of *δ-amino-αγ-dicyano-ββ-dimethylvaleric acid,* from which by heating with hydrochloric acid a substance was obtained having the properties of a pyridinecarboxylic acid. This was probably the *monoamide* of *6-hydroxy-2 : 4-dimethylpyridine-3 : 5-dicarboxylic acid,* hydrolysis being accompanied by dehydrogenation and the wandering of a methyl group to the α-position in the pyridine nucleus. Since attempts to prepare the normal hydrolysis product failed, this method of synthesis was abandoned.

αα′-Dicyano-ββ-dimethylglutarimide (4 g.) in ethyl alcohol (70 c.c.) and concentrated sulphuric acid (7 c.c.) was reduced with a rotating lead cathode (5 amps.; 125 sq. cm. immersed) for 8 hours, the cell being cooled with ice. The cathode liquid was added to water (500 c.c.), and the precipitated unchanged imide (3 g.) recovered. The free acid was exactly neutralised with potassium hydroxide, and the solution evaporated to dryness. The residue was extracted with alcohol (Soxhlet), and on removal of the solvent a gum remained which crystallised when rubbed with water. The *lactam* crystallised from alcohol–benzene in prisms, m. p. 194° (Found : C, 60·7; H, 6·3; N, 23·5. $C_9H_{11}ON_3$ requires C, 61·0; H, 6·2; N, 23·7%). The lactam (3 g.) and concentrated hydrochloric acid (20 c.c.) were heated in a sealed tube for 5 hours at 130—140°; the resulting amic *acid* crystallised from hot water, in which it was very sparingly soluble, in thin plates, decomp. above 360° (Found : C, 51·7; H, 5·0; N, 13·6. $C_9H_{10}O_4N_2$ requires C, 51·4; H, 4·8; N, 13·3%). It was insoluble in all the ordinary organic solvents, lost ammonia when digested with alkali, and gave a red colour with aqueous ferric chloride.

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Substituted Phenyldihydroresorcinols.

By L. E. Hinkel, E. E. Ayling, and J. F. J. Dippy.

The requisite intermediate substituted benzylideneacetones were obtained by the following general procedure : *o*-Chlorobenzaldehyde (20 c.c.), acetone (50 c.c.), 10% aqueous sodium hydroxide (20 c.c.), and water (500 c.c.) were shaken together for 24 hours, and the mixture was then saturated with sodium chloride. Ether extracted a liquid (17 g.), b. p. 160—163°/17 mm., which solidified in a freezing mixture and crystallised from well-cooled light petroleum (b. p. 40—60°) in stout colourless needles, m. p. 24·5° (Found : Cl, 19·3. Calc. : Cl, 19·7%) (Vorländer, *Annalen,* **1897**, **294**, 291, describes *o*-chlorobenzylideneacetone as a liquid, b. p. 189°/30 mm.).

5-o-*Chlorophenyldihydroresorcinol.*—Molecular proportions of *o*-chlorobenzylideneacetone (24 g.), ethyl malonate, and sodium ethoxide in alcohol were condensed by heating on a water-bath for 10 hours. The subsequent procedure, similar to that described for 5-*iso*propyldihydroresorcinol by Crossley and Pratt (J., 1915, **107**, 173), yielded 5-o-*chlorophenyldihydroresorcinol* (8 g.; 27%), which crystallised from aqueous alcohol in fine white plates, m. p. 149° (decomp.) (Found: Cl, 15·7. $C_{12}H_{11}O_2Cl$ requires Cl, 16·0%).

5-o-*Methoxyphenyldihydroresorcinol.*—*o*-Methoxybenzylideneacetone (20 g., prepared in 68% yield by the above procedure) was condensed as described above, but with heating for 12 hours. Acidification of the dihydroresorcylic acid yielded an oil, which solidified on cooling, the solution also depositing a small quantity of crystals, and crystallisation from aqueous alcohol gave 5-o-*methoxyphenyldihydroresorcinol* in colourless prisms, m. p. 145° (Found: C, 71·7; H, 6·4. $C_{13}H_{14}O_3$ requires C, 71·6; H, 6·4%).

5-p-*Methoxyphenyldihydroresorcinol.*—*p*-Methoxybenzylideneacetone (37 g., prepared in 72% yield by the above procedure) was condensed as described above, but with heating for 9 hours, to yield 5-*p*-methoxyphenyldihydroresorcinol (27 g., 59%), which crystallised from acetone in fine colourless leaflets and from alcohol in colourless prisms, m. p. 175° (decomp.) (Vorländer and Erig, *Annalen*, 1897, **294**, 310, record m. p. about 185°) (Found: C, 71·1; H, 6·4. Calc.: C, 71·6; H, 6·4%).

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Note on " The Conductivity of Methoxides and Ethoxides." By W. F. K. WYNNE-JONES.

IN a paper with the above title (J., 1934, 1197) Jones and Hughes reported the results of careful measurements on the conductivity of methoxides, ethoxides, and methyl and ethyl carbonates in their respective alcohols. Their results are in good agreement with the data published by the author (*J. Physical Chem.*, 1927, **31**, 1647) for the conductivity of sodium methoxide and methyl carbonate in methyl alcohol, as is shown below.

NaOMe: $\Lambda = 98{\cdot}4 - 236c^{1/2}$ (J. and H.); $\Lambda = 98{\cdot}3 - 223c^{1/2}$ (W.-J.).
$NaMeCO_3$: $\Lambda = 91{\cdot}0 - 277c^{1/2}$ (J. and H.); $\Lambda = 90{\cdot}5 - 265c^{1/2}$ (W.-J.).

In their discussion of the solvent correction, Jones and Hughes state: " Wynne-Jones has corrected conductivity data for sodium methoxide in methyl alcohol on the assumption that the carbon dioxide concentration can be calculated from the conductivity of the solvent." Actually, the correction was applied for both strong and weak bases quite empirically, and for strong bases such as sodium methoxide the equation used (see p. 1656) was $\Lambda_{\text{obs.}} = (1 - x)\Lambda_{\text{true}} + x\Lambda_{\text{NaMeCO}_3}$, which is identical with that deduced by Jones and Hughes (p. 1202).

The author also outlined a more general empirical method of correcting the conductivity data for strong acids and bases, using the equation $\Lambda_{\text{obs.}} = \Lambda_0 - bc^{1/2} + k/c$, which, as indicated elsewhere (Wynne-Jones, *J. Amer. Chem. Soc.*, 1932, **54**, 2130), can be applied to data for aqueous solutions.

With regard to Jones and Hughes's statement that " it has been found that ammonia is present in quantities of the order of $2 \times 10^{-7}N$," it seems that this is an hypothesis rather than an experimental fact. In order to account for a specific conductivity of 10^{-7} ohm^{-1}, it would be necessary to assume an ammonia concentration of $1 \times 10^{-6}N$: for reasons already discussed by the author, the presence of such amounts of ammonia in the methyl alcohol used by Goldschmidt and Hartley and their students is improbable.—FRICK CHEMICAL LABORATORY, PRINCETON UNIVERSITY, PRINCETON, NEW JERSEY. [*Received, January 18th,* 1935.]