(as Barry, Peterson and King[1] showed recently) by way of the ammonia compound back to native cellulose. Moreover, Hess and Gundermann[2] have described the appearance of native cellulose along with hydrate cellulose during the washing out of alkali cellulose at 100° C. We find, however, that it is possible to convert hydrate cellulose directly into native cellulose.

When strongly stretched hydrate cellulose in the form of viscose silk 'Lilienfeld silk' is heated for half an hour in water at 200° C., the X-ray diagram shows interferences characteristic of native cellulose as well as those of hydrate cellulose. The same diagram is obtained after treating viscose with boiling formamide (200° C. for half an hour). If the fibres are heated in formamide to 140° C., less native cellulose is formed than at 200° C.; after half an hour in glycerine at 250° C. the hydrate cellulose is converted almost completely into the native form. If the fibres are left for eight days in water at 200° C., very little more native cellulose is formed than after half an hour.

The same experiments were made with hydrate cellulose obtained by the mercerization of ramie. It was found that under the same conditions less native cellulose was formed from this material than from Lilienfeld silk.

Dry viscose heated for twenty minutes in a high vacuum at 200° C. remains unchanged; but after ten minutes at 300° C. a little native cellulose is formed. Water and other dipole-containing liquids appear to increase the rate of transformation very considerably.

Native cellulose (ramie) after heating for five days in water at 150° C. remains unchanged. We conclude that at least over the range of temperature covered by these experiments *native cellulose is the stable form, and hydrate cellulose the non-stable.*

As already indicated, Hess and Gundermann obtained native cellulose by the action of water at 100° C. on alkali cellulose. We have been able to detect native cellulose in preparations which were washed out at 60° C. If decomposition and washing out take place at 20° C. and the preparation is afterwards heated, no native cellulose is formed even at 100° C. On dipping fibres of alkali cellulose into formamide at 140° C. the product obtained is almost entirely native cellulose. In all these cases, therefore, the temperature necessary for the formation of native cellulose is considerably lower than that required for the transformation of hydrate cellulose as such into the native form.

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[1] *J. Amer. Chem. Soc.*, **58**, 333 (1936).
[2] *Ber. deutsch. chem. Ges.*, **70**, 527 (1937); see also Hess and collaborators in *Z. phys. Chem.*, B, **7**, 7 (1930).

Isolation of Ascorbic Acid from Urine

HINSBERG and Ammon[1] have been unsuccessful in an attempt to isolate ascorbic acid from urine. Recently E. C. Noyons[2] has described a method for the isolation of ascorbic acid from tomato juice involving chromatographic adsorption, and has announced his intention of applying a similar procedure to the isolation of ascorbic acid from urine. We have been engaged for a considerable time on this problem and have finally isolated from urine a small amount of a crystalline dinitro-phenyl hydrazine derivative which appears to be that of ascorbic acid. Its melting point (269–271 uncorr.) agrees with that of the similar derivative prepared from pure ascorbic acid (m.p. 270–272) and the crystalline forms appear identical. A mixed m.p. with the dinitro-phenyl hydrazine derivative of the pure vitamin shows no depression (m.p. 271–272). The reliability of the m.p. determinations has been checked by taking a mixed m.p. of the 2 : 4 dinitro-phenyl hydrazine derivatives of ascorbic acid and salicylaldehyde (m.p. 249). A very marked depression was observed, both in this case and in another where a mixed melting point of two different hydrazones with very similar melting points was taken.

This work is being continued, and we hope shortly to be in a position to give a more complete account of this investigation.

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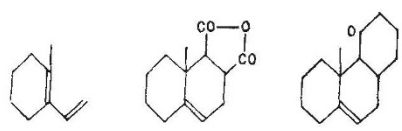
[1] Hinsberg, K., and Ammon, R., *Biochem. Z.*, **288**, 102 (1936).
[2] Noyons, E. C., *Acta. Brev. Neerlandica*, **7**, 79 (1937).

A Diene Synthesis applicable to the Sterol Group

THIS method is regarded as the most promising yet adumbrated in that it reduces the problem of the synthesis of cholesterol or its stereoisomerides and analogous substances to the much simpler one of preparing a monocyclic and a bicyclic intermediate. It seems to be especially important that the double bond of cholesterol is placed by this synthetic process in the correct position and that unlike many other diene syntheses the method can be used to introduce the angle-methyl groups.

1-Methyl-2-vinylcyclohexene (I), obtained from the known 2-methylcyclohexenylethyl alcohol by an application of the xanthogenate reaction, condenses with maleic anhydride in benzene solution with the formation of the anhydride (II), m.p. 111·5°. This and the related dibasic acid, m.p. 171°, afforded analytical values tallying with theoretical anticipations.

With crotonaldehyde the diene furnishes an adduct the dinitrophenylhydrazone of which has m.p. 192° and analysis of which agrees with the formula $C_{19}H_{24}O_4N_4$. The 2 : 4-dinitrophenylhydrazone of the adduct (III) from the diene and cyclohexenone has m.p. 164° and gives satisfactory figures on analysis.



(I) (II) (III)
Fig. 1.

The constitutions of the intermediates now required for the synthesis of substances containing the cholane skeleton are obvious on inspection, and attempts to make some of them are in progress.

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