

The exact mathematical and theoretical treatment and technical details omitted from this preliminary note will be presented in a further paper.

I wish to thank Professor Artturi I. Virtanen, Director of the Biochemical Institute, for his encouragement, for valuable suggestions during this work and for the excellent facilities put at my disposal in the laboratory of the Foundation for Chemical Research. I also wish to thank Dr. Matti Kreula for his valuable help and suggestions in planning and building the mechanical parts of this counter-current apparatus.

This work has been supported partly by the *Rockefeller Foundation* and partly it belongs to the research project under U. S. Public Law No. 480, 83rd Congress.

1. Jantzen, E. *Dechema-Monographie* 48, Verlag Chemie, Berlin 1932.
2. Cornish, R. E., Archibald, R. C., Murphy, E. A. and Evans, H. M. *Ind. Eng. Chem.* 26 (1934) 397.
3. Craig, L. C. *J. Biol. Chem.* 155 (1944) 519.
4. Craig, L. C., Hausmann, W., Ahrens, Jr., E. H. and Harfenist, E. J. *Anal. Chem.* 23 (1951) 1326.
5. v. Metzsch, F. A. *Chem. Ing. Tech.* 25 (1953) 66.
6. Rauen, H. M. and Stamm, W. *Gegenstromverteilung*, Springer-Verlag, Berlin-Göttingen-Heidelberg 1953.
7. Hecker, E. *Verteilungsverfahren im Laboratorium*, Verlag Chemie, Weinheim 1955.
8. Weygand, F., Wacker, A. and Dellwegg, H. *Z. Naturforsch.* 6B (1951) 130.
9. Stene, S. *Arkiv Kemi, Mineral. Geol.* 18A (1945) 18.

Received December 4, 1959.

### Ozonisation of 3-Methylcyclopent-2-en-2-ol-1-one

JOHAN B. SON BREDEBERG

*Department of Chemistry, Institute of Technology, Helsingfors, Finland*

In a previous paper<sup>1</sup> it was shown by physical methods that 3-methylcyclopenta-1,2-dione exists as the enol 3-methyl-

cyclopent-2-en-2-ol-1-one. This structure should be readily attacked by ozone to yield laevulic acid. The other possible enol structure for the compound, 5-methylcyclopent-2-en-2-ol-1-one, should, on the other hand, give methylsuccinic acid. The enol structure can therefore be confirmed also by ozonisation of the compound.

The compound was ozonised in aqueous solution at room temperature and the water evaporated. The evaporation residue, which had an infrared spectrum of almost pure laevulic acid, but with a small amount of starting material, was distilled and gave a high yield of pure laevulic acid, b.p. 103–105°/1 mm, identified by its infrared spectrum and its phenylhydrazone, m.p. 105–106°, and 2,4-dinitrophenylhydrazone, m.p. 207–208°. The result consequently corroborated the evidence obtained by physical methods.

*Experimental.* 3-Methylcyclopent-2-en-2-ol-1-one (Fluka A.-G.) (3.73 g) was dissolved in water (150 ml) and ozonised at room temperature for 2 h (5.1 % O<sub>3</sub>, 15.7 mg O<sub>3</sub>/min). The solution was left to stand for two days, after which the water was evaporated under diminished pressure at 20°. The evaporation residue, an almost colourless oil, was distilled under reduced pressure and gave two fractions. The first fraction, b.p. 80–103°/1 mm, (0.53 g) which partly crystallised, consisted, according to the infrared spectrum, of the starting material and laevulic acid. The second fraction, b.p. 103–105°/1 mm, (2.94 g) had the infrared spectrum of pure laevulic acid. The fraction yielded a phenylhydrazone, m.p. 105–106°, and a 2,4-dinitrophenylhydrazone, m.p. 207–208°, both prepared in the usual way<sup>2,3</sup>. Neither derivative gave any m.p. depression with authentic material from pure laevulic acid.

1. Bredenberg, J. B. *Acta Chem. Scand.* 13 (1959) 1733.
2. Fischer, E. *Ann.* 236 (1886) 126.
3. Cowley, M. A. and Schuette, H. A. *J. Am. Chem. Soc.* 55 (1933) 3463.

Received December 16, 1959.