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

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

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In a previous paper 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 ozone formation 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. 105–106°C/1 mm, identified by its infrared spectrum and its phenylhydrzone, m.p. 105–106°C, and 2,4-dinitrophenylhydrzone, m.p. 207–208°C. The result consequently corroborates 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 % O₃, 15.7 mg O₃/min). The solution was left to stand for two days, after which the water was evaporated under diminished pressure at 20°C. The evaporation residue, an almost colourless oil, was distilled under reduced pressure and gave two fractions. The first fraction, b.p. 80–103°C/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°C/1 mm, (2.94 g) had the infrared spectrum of pure laevulic acid. The fraction yielded a phenylhydrzone, m.p. 105–106°C, and a 2,4-dinitrophenylhydrzone, m.p. 207–208°C, both prepared in the usual way. Neither derivative gave any m. p. depression with authentic material from pure laevulic acid.

2. Fischer, E. Ann. 236 (1886) 126.

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