An Isolatable α-Lactone made by Direct Epoxidation

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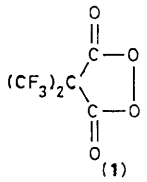
Epoxidation of perfluoro-[(1-ethyl-1-methylpropyl)(1-methylpropyl)]keten with sodium hypochlorite–acetonitrile afforded a high yield of perfluoro-[(1-ethyl-1-methylpropyl)(1-methylpropyl)]-ethanolide, the most stable α-lactone yet to be isolated.

There has been much interest in α-lactones; they have been postulated[1] as reaction intermediates, and many attempts[2–5] have been made to isolate them, using a wide variety of approaches. However, in only one case[5] so far was a product obtained with any degree of stability. Photolysis of the peroxide (**1**), in solution in carbon tetrachloride at −15 °C, gave a solution containing the α-lactone (**2**). At 24 °C in the gas phase, (**2**) had a half-life of 8 hours, but the pure compound could not be isolated. Predictions[3,5,6] have been made that α-lactones and related species require bulky electron-attracting substituents for maximum stability.

It seemed to us that such a compound could be made by extension to ketens of our direct epoxidations of cyclic[7] and acyclic[8] fluorocarbon alkenes, for which we have used a modification of the sodium hypochlorite system introduced[9] by Kolenko *et al.* One of the oligomers[10] of tetrafluoroethylene, the major hexamer isomer (**3**), readily gives[11] the keten (**4**) by alkaline hydrolysis.

When this keten (**4**) was treated with sodium hypochlorite–acetonitrile, the desired α-lactone (**5**) was obtained, in yields around 85%, as a colourless mobile liquid at room temperature.† ^{19}F N.m.r. spectroscopy showed the presence of the intact fluorocarbon groups, and indicated that two diastereo-

† Satisfactory elemental analyses for C and F were obtained.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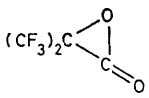
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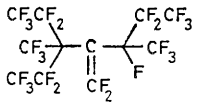
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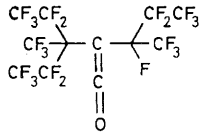
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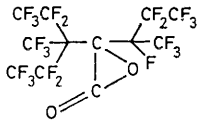
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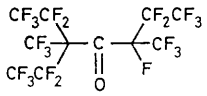
(6)

isomers were present. The i.r. spectrum had a complex band with main peaks at 1945 and 1990 cm^{-1} [(**2**) had a band[5] centred at 1975 cm^{-1}] whereas the keten (**4**) had a sharp band at 2160 cm^{-1}. The α-lactone (**5**) was stable at least for several days at room temperature and for 2 months at −15 °C, but on being heated to 60—80 °C for 15 min it lost carbon monoxide in a quantitative conversion into the ketone (**6**), b.p. 150—152 °C.† The broad i.r. band in the region 1900—2070 cm^{-1} in the spectrum of the lactone (**5**) had completely disappeared in (**6**), being replaced by a sharp carbonyl absorption at 1755 cm^{-1}. ^{19}F N.m.r. and mass spectrometry confirmed the structure of (**6**).

Thus we have a simple route to an α-lactone which is certainly the most stable reported so far.

Received, 21st December 1981; Com. 1437

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