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Formation of Lactones from Dienes *via* Iron Carbonyl Complexes

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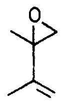
Summary A number of dienes were monoepoxidised and treated with $Fe(CO)_5$ to form intermediate complexes which were subsequently oxidised to lactones.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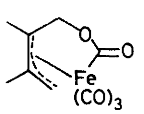
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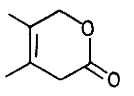
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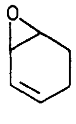
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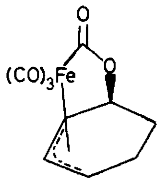
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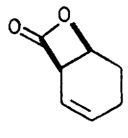
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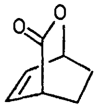
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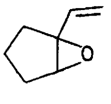
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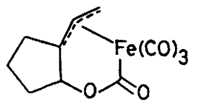
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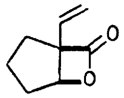
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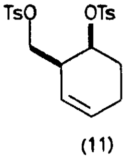
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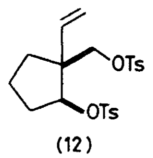
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Ts = p-$MeC_6H_4SO_2$

RECENT studies[1] on the preparation of δ-lactones from dienes prompts us to report our results in a related area. We were attracted by the known propensity for carbonyl group insertion into vinyloxirans by pentacarbonyliron[2] and reasoned that the complexes formed would lead to lactones on oxidation. Irradiation of the monoepoxide (**1**),[3] from 2,3-dimethylbutadiene, in the presence of $Fe(CO)_5$ gave the iron complex (**2**), which showed structurally significant i.r. bands at 2050, 1990, and 1660 cm^{-1}. The n.m.r. spectrum was also in agreement with the proposed structure (**2**). Subsequent oxidation with ceric ammonium nitrate at 60 °C in ethanol–water (50 : 50) gave the lactone (**3**) in 38% yield.

The known iron complex (**5**)[2] was similarly prepared from 1,3-cyclohexadiene monoepoxide (**4**); however on oxidation with ceric ammonium nitrate at room temperature (**4**) afforded the β-lactone (**6**) in 65—70% yield, and the lactone (**7**)[4] was not detected. The β-lactone (**6**) was readily characterized from its spectral data {*e.g.* ν(C=O) 1820 cm^{-1}, typical of β-lactones; δ 5·6—6·35 (2H, m, vinylic), 4·75—5·0 [1H, m, –CH–O–C(:O)–], 4·12 [1H, dd *J* 6 Hz, –C(:O)–CH–C=], and 1·4—2·5 (4H, m, aliphatic)}. Decoupling experiments are in accord with the above assignments and in addition these data are similar to those of recently prepared β-lactones.[5]

The monoepoxide (**8**) on treatment with $Fe(CO)_5$ gave the iron complex (**9**) (79%) which showed typical carbonyl absorptions at ν_{max} 2050, 2000, 1990, and 1660 cm^{-1}. Oxidation of this complex afforded exclusively the β-lactone (**10**) in 40% yield. Structural assignment follows from the spectral data, ν_{max} 1820 cm^{-1}, δ 1·0—2·2 (m, 6H, aliphatic), 4·78 (d, 1H *J* 2 Hz, –CH–O–), 5·1—5·55 (m, 2H, methylene), and 5·65—6·15 (m, 1H, vinylic). Very few examples of similar α-vinyl β-lactones appear to be known in the literature.[6]

As both the β-lactones (**6**) and (**10**) were rather unstable, they were reduced with lithium aluminium hydride to the corresponding diols which in turn were characterised as their ditosylates (**11**) and (**12**).

The above procedure constitutes the formation of lactones from dienes *via* iron carbonyl complexes.

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