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Preliminary Communication

ISOLATION, CHARACTERISATION AND OXIDATION OF ISOMERIC FERRELACTONE COMPLEXES.

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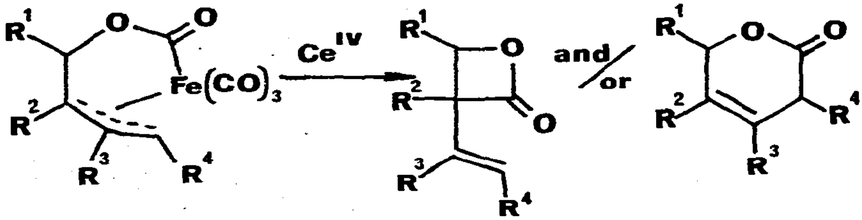
Summary

Irradiation of 1,2-epoxy-1-vinylcyclopentane (1) in the presence of $Fe(CO)_5$ leads to the formation of isomeric *syn*- and *anti*- ferrelactones (2) and (3). The structures of these complexes were determined by X-ray crystallographic methods and on oxidation gave different ratios of β- and δ-lactones.

Ferrelactone complexes can be prepared by a variety of methods[1–6] although under photolytic conditions their formation from vinyloxiranes is reported to be stereospecific.[4]

We have recently reported that ferrelactone complexes can be converted to lactones on oxidation with Ce^{IV}.[7] Surprising however was the predominant formation of the more strained β-lactone relative to the less strained δ-lactone.



We now show that it is possible to prepare isomeric ferrelactones which on separate oxidation lead to different product ratios.

Reaction of the vinyloxirane (1) with pentacarbonyl iron affords a separable mixture of isomeric ferrelactones (2) and (3) in the ratio of 6 : 1 in 79% yield.

As the structure assignment of these complexes was not conclusive by spectroscopic means we resorted to X-ray crystallographic methods. The compounds (2) and (3) are referred to as *syn*- and *anti*-isomers respectively to indicate the position of the iron moiety relative to the bridge oxygen atom.

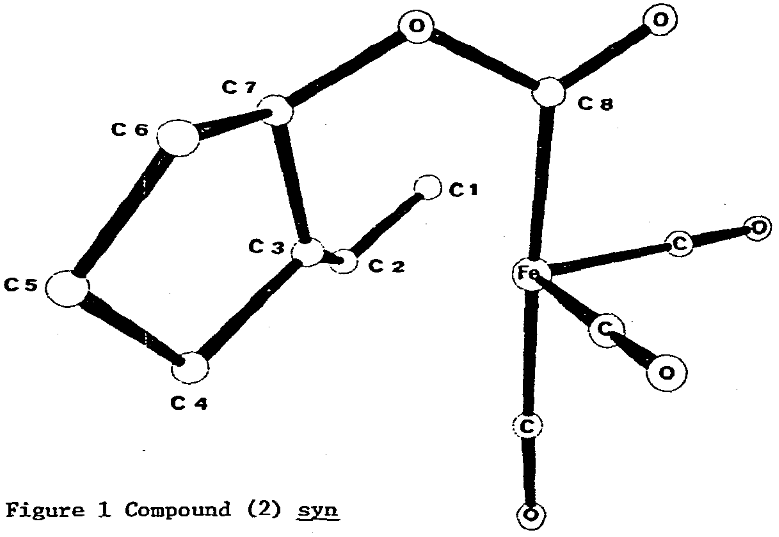
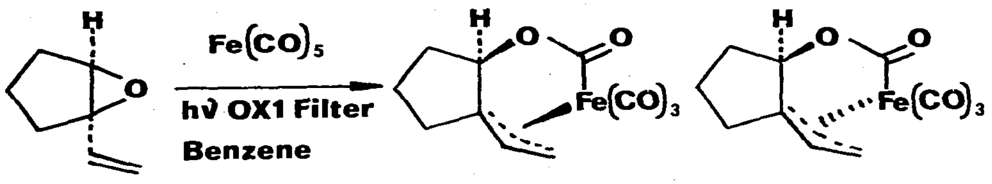


Figure 1 Compound (2) *syn*

X-ray parameters:- Triclinic *a* = 13.737(2), *b* = 13.128(2), *c* = 6.733(1)Å, α = 85.08(2), β = 96.15(2), γ = 107.67(2)°. Space group $P\bar{1}$, Z = 4 (2 independent but conformationally identical molecules in asymmetric unit). 2787 reflections to $\theta \leqslant 60°$ (734 unobserved) with Cu-*K* radiation; R = 0.072. Fe-C1 2.17(1); Fe-C2 2.11(1); Fe-C3 2.13(1); Fe-C8 1.99(1)Å.



(1) (2) Syn (3) Anti

The X-ray data[8] for both structures (Figures 1 and 2) also shows that the carbon atom at the terminus of the η^3 allyl system, i.e. C3, is some 0.24Å out of plane relative to the neighbouring carbon atoms, C2, C4 and C7, thus indicating its high sp^3 character.[9]

The *syn* complex (2) on oxidation with ceric ammonium nitrate in acetonitrile gave a separable mixture of *cis* fused β-lactone (4)

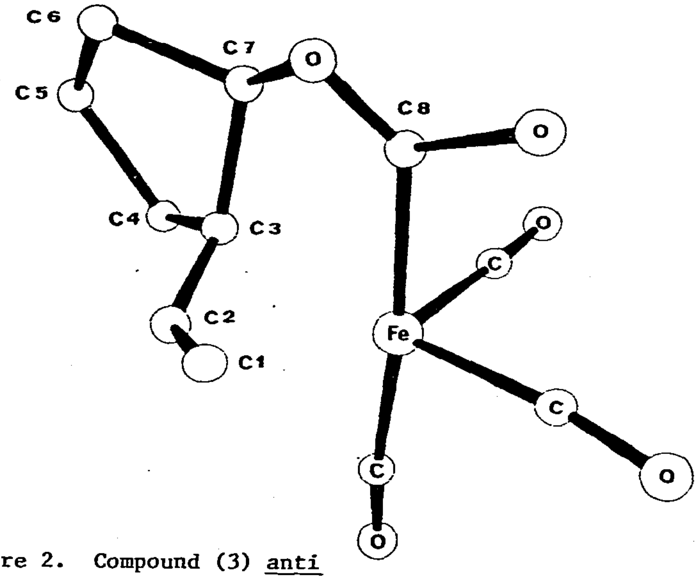
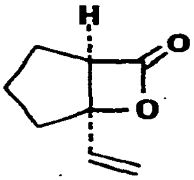


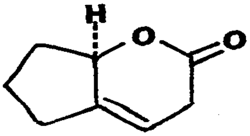
Figure 2. Compound (3) *anti*

X-ray parameters: Monoclinic a = 7.617(1), b 12.560(2), c = 12.990(2)Å, β = 109.44(2)°. Space groups $P2_1/c$, Z = 4 1206 reflections to θ ≤ 50° (318 unobserved), with Cu-K radiation; R = 0.107 (without absorption correction). Fe-C1 2.14(2); Fe-C2 2.03(3); Fe-C3 2.12(2); Fe-C8 2.06(3)Å.

Atomic coordinates for both structures have been deposited with the Cambridge Crystallographic Data Centre, University Chemical Laboratory, Cambridge, CB2 1EW, U.K.



(4)



(5)

and δ-lactone (5) in a ratio of 1.8 : 1 in 80% overall yield. However similar oxidation of (3) gave the δ-lactone (5) exclusively.

The ring strain of the corresponding *trans*-fused β-lactone from (3) precludes its formation.

These results suggest that during the oxidation of ferrelactones the stereochemical integrity of the starting material dominates the formation of the products.[10]

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References and Footnotes.

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8. For full X-ray data for the ferrelactone reported in reference 4, see M.R. Churchill and K-N. Chen, *Inorg. Chem.*, 1976, *15*, 788.
9. For other η^3 allyl complexes see R.B. King in " The Organic Chemistry of Iron" , Vol. 1, Ed. E.A. Koerner von Gustorf, F-W. Grevels, and I. Fischler, Academic Press, 1978, p. 463.
10. A full discussion of the mechanism of oxidation of these and other complexes will be described in the full paper (in preparation).