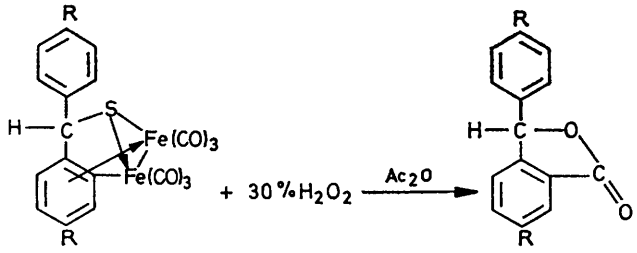
A Novel Lactone Synthesis

By HOWARD ALPER*† and WILLIAM G. ROOT

(*Department of Chemistry, State University of New York at Binghamton, Binghamton, New York* 13901)

Summary Cleavage of sulphur-donor ligand *ortho*-metallated complexes by 30% hydrogen peroxide or by *m*-chloroperbenzoic acid provides a novel synthesis of lactones.

SULPHUR-DONOR ligand *ortho*-metallated complexes [*e.g.* (I)] are useful precursors to benzo[*c*]thiophen derivatives,[1] and to aromatic compounds[2,3] which are difficult or impossible to obtain by other means. We describe a novel synthesis of lactones, obtained by treatment of these complexes with 30% hydrogen peroxide or with *m*-chloroperbenzoic acid (MCBA).



(I) (II)

Reaction of (I; R = H, Me, or OMe) with excess of 30% H_2O_2 in acetic anhydride affords the lactones (II) in 45—73% yields (Table), which were characterized by i.r.[ν_{CO} (neat) 1765—1778 cm^{-1}],[4] n.m.r., and mass spectral data as well as by elemental analyses.‡ Lactones were also formed by treatment of (I) with MCBA in dry benzene at room temperature for 2·5 h, but work-up proved to be less simple than with 30% H_2O_2.

TABLE

Yields of lactones obtained by reaction of (I) with 30% H_2O_2 or MCBA[b]

R	Reagent	M.p. (°C) (II)	Yield (%)[a]
OMe	30% H_2O_2	133—135	73
OMe	MCBA	133—134	57
Me	30% H_2O_2	109—110	45
H	30% H_2O_2	103—105	49

[a] Yields are of pure material. [b] Typically, H_2O_2 (30%; 5·0 ml) was added dropwise to a stirred, ice-cold solution of (I; R = Me) (1·38 g) in Ac_2O (50 ml). The mixture was then allowed to warm to room temperature, and left for several days. After filtration and evaporation *in vacuo*, the resulting oil was treated with benzene, filtered, and the filtrate was concentrated and chromatographed on neutral alumina. Elution with benzene gave the lactone, (II; R = Me).

We thank the National Institutes of Health for support.

(*Received, 25th July* 1974; *Com.* 935.)

† Department of Chemistry, University of Ottawa, Ottawa, Ontario, Canada K1N 6N5.

‡ Satisfactory (±0·4%) analytical data were obtained for all new compounds.

[1] H. Alper and A. S. K. Chan, *J. Amer. Chem. Soc.*, 1973, **95**, 4905.
[2] H. Alper and W. G. Root, *Tetrahedron Letters*, 1974, 1611.
[3] H. Alper, *J. Organometallic Chem.*, 1973, **61**, C62.
[4] R. M. Silverstein, G. C. Bassler, and T. C. Morrill, 'Spectrometric Identification of Organic Compounds,' 3rd edn., Wiley, New York, 1974, p. 149.