Oxymetallation. Part VIII.[1] The Stereochemistry of Peroxymercuration of Cyclohexene, Norbornene, But-2-ene, and Stilbene; Intramolecular Co-ordination in β-Peroxyalkylmercury Trifluoroacetates

By **A. J. Bloodworth** * and **I. M. Griffin,** The Christopher Ingold Laboratories, Chemistry Department, University College London, 20 Gordon Street, London WC1H 0AJ

The configurations of peroxymercurials, $RCH^A(OOBu^t)CH^B(HgTFA)R$, prepared from mercuric trifluoroacetate have been assigned on the basis of vicinal proton–proton coupling constants (J_{AB}). The structures of the cyclohexene and norbornene products are readily assigned from spectral similarity with other oxymercurials and they result from *trans*- and *cis*-addition respectively. In the but-2-ene (R = Me) and stilbene (R = Ph) products the assignments depend on comparisons between *cis*- and *trans*-derived diastereoisomers. The relative magnitudes of J_{AB} and their behaviour upon addition of pyridine or replacement of trifluoroacetate by bromide indicate that there is intramolecular $Bu^tO \cdots Hg$ co-ordination in the adducts and that the peroxymercurations involve *trans*-addition.

THE hydroxymercuration, methoxymercuration, and (trifluoro)acetoxymercuration of alkenes [equation (1; Y = H, Me, CF_3CO, and CH_3CO respectively)] are stereospecific processes except for reactions with some bicyclo-oct-2-enes [2] and *trans*-cyclodecene.[3] Until recently configurational assignments were limited to

$$R^1CH{=}CHR^2 + HgX_2 + YOH \longrightarrow R^1CH(OY)CH(HgX)R^2 + HX \quad (1)$$

oxymercurials derived from cyclic and bicyclic alkenes. The C_4—C_9 *cis*-cycloalkenes undergo *trans*-oxymercuration [4] whereas *trans*-cyclo-octene, *trans*-cyclononene,[4] and norbornene and related alkenes [5] exhibit *cis*-addition.

The initial evidence for these modes of addition came from studies with cyclohexene and norbornene. *X*-Ray diffraction studies revealed the *trans*-diequatorial disposition of chloromercuri- and methoxy-substituents in the cyclohexene product,[6] and the same configuration for the hydroxymercurial was established by ^1H n.m.r. spectroscopy on the basis of vicinal proton–proton coupling constants.[7,8] Dipole moment comparisons were consistent with a *cis-exo*-configuration for the norbornene methoxymercurial; [9] the presence of an *exo*-hydroxy-group in the hydroxymercurial was demonstrated by reduction and conversion to the known acetate [9] and the *cis-exo*-nature of hydroxymercuration was later confirmed from vicinal proton–proton coupling constants.[7] Stereochemical assignments in related systems have relied largely upon semi-empirical techniques based for methoxymercurials on the change in methoxy chemical shift upon reductive demercuration [10] or solvent change,[11] and for hydroxymercurials on the change in OH stretching frequency upon reductive demercuration.[12]

It was known for a long time that the oxymercuration of acyclic alkenes is stereoselective [5] but until recently the generally accepted view of a *trans*-mode of addition was based mainly on analogy with cyclohexene. Stereospecific *trans*-addition has now been confirmed for the methoxymercuration of [1,2-$^2H_2$]ethylene,[13] methyl 4-methoxycinnamate,[14] and *cis*-2,2,5,5-tetramethylhex-3-ene,[15] but *trans*-2,2,5,5-tetramethylhex-3-ene has provided the first acyclic example of *cis*-addition. Configurational assignments were based on vicinal proton–proton coupling constant data and rely upon the reasonableness of assumed conformational preferences, except for [1,2-$^2H_2$]ethylene where solvent effects establish the *trans*-mode of addition unequivocally.

Evidence pertaining to the stereochemistry of peroxymercuration [equation (1; Y = Bu^tO)] is limited to the reports that methyl cinnamate,[16] but-2-ene,[1] and stilbene [1] react stereospecifically. The configurations of the diastereoisomers from methyl *cis*- and *trans*-cinnamate could not be assigned with confidence because the appropriate vicinal proton–proton coupling constants are too similar.

We here assign configurations to the diastereoisomers from but-2-ene and stilbene, and to the peroxymercurials from cyclohexene and norbornene. The results show that the stereochemistry of peroxymercuration parallels that of other oxymercurations, *i.e.* *cis*-addition to norbornene and *trans*-addition to the unstrained alkenes. This is not unexpected and perhaps the more important outcome of the work is the illustration that the possibility of intramolecular co-ordination must be considered if correct configurational assignments are to be made in acyclic systems on the basis of vicinal proton–proton coupling constants.

---

[1] Part VII, A. J. Bloodworth and I. M. Griffin, *J.C.S. Perkin I*, 1975, 195.

[2] R. D. Bach and R. F. Richter, *Tetrahedron Letters*, 1973, 4099.

[3] V. I. Sokolov, L. L. Troitskaya, and O. A. Reutov, unpublished work quoted in ref. 4.

[4] W. L. Waters, T. G. Traylor, and A. Factor, *J. Org. Chem.*, 1973, **38**, 2306.

[5] W. Kitching, *Organometallic Chem. Rev.*, 1968, **3**, 61.

[6] A. G. Brook and G. F. Wright, *Acta Cryst.*, 1951, **4**, 50.

[7] M. M. Anderson and P. M. Henry, *Chem. and Ind.*, 1961, 2053.

[8] S. Wolfe and P. G. C. Campbell, *Canad. J. Chem.*, 1965, **43**, 1184.

[9] T. G. Traylor and A. W. Baker, *Tetrahedron Letters*, 1959, No. 19, 14.

[10] W. L. Waters, *Tetrahedron Letters*, 1969, 3769.

[11] R. F. Richter, J. C. Philips, and R. D. Bach, *Tetrahedron Letters*, 1972, 4327.

[12] T. G. Traylor and A. W. Baker, *J. Amer. Chem. Soc.*, 1963, **85**, 2746.

[13] T. Ibusuki and Y. Saito, *J. Organometallic Chem.*, 1973, **56**, 103.

[14] M. C. Cabaleiro, A. D. Ayala, and M. D. Johnson, *J.C.S. Perkin II*, 1973, 1207.

[15] R. D. Bach and R. F. Richter, *J. Org. Chem.*, 1973, **38**, 3442.

[16] A. J. Bloodworth and I. M. Griffin, *J.C.S. Perkin I*, 1974, 688.

RESULTS

Proton–proton and ^{199}Hg–H coupling constants of the β-peroxyalkylmercury trifluoroacetates (and related bromides) formed by peroxymercuration of cyclohexene,

$$\mathrm{RCH{=}CHR + Hg(TFA)_2 + HOOBu^t \longrightarrow RCH(OOBu^t)CH(HgTFA)R} \quad (2)$$

$$\mathrm{TFA = CF_3CO_2}$$

norbornene, but-2-ene, and stilbene [equation (2)] are listed in the Table. For the but-2-ene and stilbene derivatives the same measurements in the presence of one to three molar equivalents of pyridine are also recorded. An example of the spectra from which these data were derived is shown in Figure 1.

Vicinal coupling constants of β-mercurioperoxides $R^1CH_A(OOBu^t)CH_B(HgX)R^2$ in deuteriochloroform and in the presence of pyridine

Parent alkene	R^1	R^2	X	Concn. % w/v	Equiv. py [a]	J_{H-H}/Hz (±0·2 Hz) AB	AR^1	BR^2	$J_{^{199}Hg-H}$/Hz (±3 Hz) A	R^2
Cyclohexene	$[CH_2]_4$		TFA [b]	43	0	10·5	*c*		104	
			Br	43	0	10·0	*c*			
Norbornene			TFA	34	0	6·7	~0	2·4	108	
			Br	29	0	6·7	~0	2·7		
cis-But-2-ene	Me	Me	TFA	46	0	6·2	6·2	7·8	272	302
				46	1	4·8	6·0	7·7	295	290
				46	2	4·7	6·2	7·8		288
				46	3	4·7	6·2	7·8		288
			Br	36	0	5·6	5·6	7·6		277
				46	1	5·9	5·9	7·7		279
trans-But-2-ene	Me	Me	TFA	46	0	3·4	6·3	7·9	344	306
				46	1	4·4	6·3	8·2 [d]	369	298
				46	2	4·6	5·9	7·8		296
				46	3	4·9	6·0	7·8		296
			Br	48	0	3·6	6·1	7·6		281
				46	1	3·8	6·2	7·7		284
cis-Stilbene	Ph	Ph	TFA	46	0	6·7				
				44	1	5·7				
				41	2	5·3				
				39	3	5·2				
			Br	37	0	5·7				
				34	1	5·9				
trans-Stilbene	Ph	Ph	TFA	47	0	6·0				
				44	1	8·9				
				42	2	9·3				
			Br	33	0	7·1				
				34	1	7·5				

[a] Number of equivalents of pyridine present. [b] TFA = $OCOCF_3$. [c] First-order analysis of the H_A doublet of triplet gives $J_{AB} = J_{AH_{ax}}$, and $J_{AH_{eq}}$ 4·4 Hz. [d] Measured only from distorted R^2 doublet.

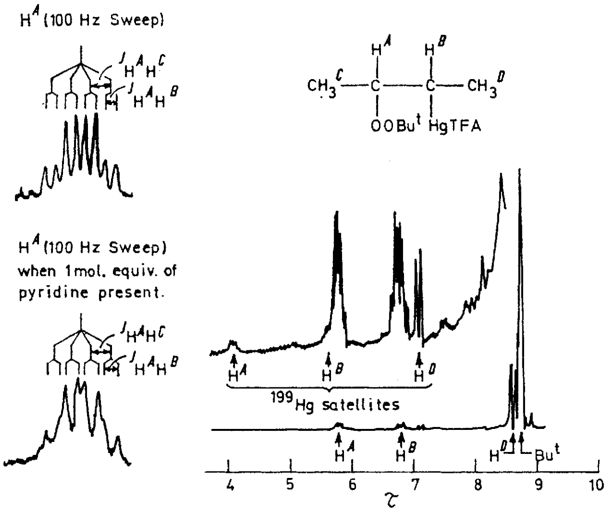
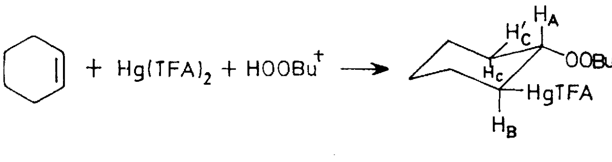


FIGURE 1 100 MHz ^{1}H N.m.r. spectrum of the peroxymercurial derived from *trans*-but-2-ene

DISCUSSION

Using vicinal proton–proton coupling constants, the assignment of configurations to the cyclohexene and norbornene peroxymercurials is relatively straightforward because of the rigidity imposed by the cyclic systems. The products from the acyclic alkenes present a greater problem, and assignments depend upon establishing the nature of conformational preferences.

Cyclohexene and Norbornene.—In the cyclohexene peroxymercurial [equation (3)], the methine proton (H_A) geminal to the peroxy-group appears as a doublet of triplet at τ 5·94. First-order analysis gives the



(3)

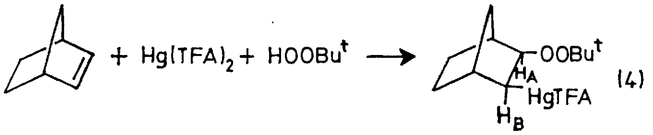
triplet coupling as 10·5 and the doublet splitting as 4·4 Hz. Although this is only an approximation it clearly indicates that H_A has two *anti* (H_B and H_C) and one *gauche* (H_C') vicinal proton, and hence that the peroxy- and trifluoroacetoxymercuri-substituents are *trans*-diequatorial. Similar conclusions were drawn for the hydroxymercurial [7] and trifluoroacetoxymercurial [17]

---

[17] H. C. Brown, M-H. Rei, and K-T. Liu, *J. Amer. Chem. Soc.*, 1970, **92**, 1760.

where, in each case, the proton geminal to the mercury substituent is an apparent doublet of triplet of similar magnitude. Further evidence that the peroxymercurial is isostructural with other cyclohexene oxymercurials comes from the ^{199}Hg–H$_A$ coupling constant (104 Hz) which closely resembles that for the methoxymercurial (99 Hz)[18] and trifluoroacetoxymercurial (*ca.* 100 Hz).[17]

For the norbornene peroxymercurial [equation (4)] the configurational assignment is again based on spectral similarity with other oxymercurials.[7,17] The features



diagnostic of *cis-exo*-addition are the appearance of H$_A$ as a doublet and of H$_B$ as a doublet of doublet, and the magnitude of the H$_A$–H$_B$ and ^{199}Hg–H$_A$ coupling constants (Table).

But-2-ene and Stilbene.—If the vicinal coupling constants (J_{AB}) of both diastereoisomers of a compound RCH^{A}X·CH^{B}Y·R are known, configurational assignments can often be made on the basis of the criterion $J_{erythro}$ * $> J_{threo}$. These coupling constants are averaged quantities which depend upon relative populations in the respective conformational equilibria. The relative magnitudes arise because the Karplus equation predicts larger coupling constants for conformations with *anti*-disposed protons than for those with *gauche*-disposed protons, and repulsions between R groups cause the conformation with *anti*-protons to be more highly populated in the *erythro*-isomer. Thus, for example, the methoxymercurials from methyl *cis*- and *trans*-4-methoxycinnamate, 4-MeOC$_6$H$_4$CH^A(OMe)CH^B(HgOAc)-CO$_2$Me, had J_{AB} 6·8 and 10·0 Hz respectively indicating respective *threo*- and *erythro*-configurations and hence *trans*-oxymercuration.[14] As the difference in coupling constants becomes smaller, the method becomes less reliable and this will arise if repulsions involving X and Y groups become comparable with those originating from the R groups.

If the criterion were to be applied to the products of peroxymercuration of but-2-ene and stilbene [equation (2; R = Me or Ph); see Table], it would be concluded that *cis*-addition takes place, an unusual result for an unstrained alkene and at variance with our observation for cyclohexene. We believe that the mode of addition is *trans* and that the observation that $J_{erythro} < J_{threo}$ arises because *attractive* interactions as well as the usual steric repulsions influence the conformational preferences.

The attractive interaction is the intramolecular co-ordination of the t-butylperoxy-group to mercury (Figure 2). In the *erythro*-isomer this favours conformations with *gauche*-protons (E_2 and E_3) and thus reduces J_{AB}, whereas in the *threo*-isomer one of the favoured conformations (T_2) has *anti*-protons so that J_{AB} is increased. It appears that in butene peroxymercurials the influence of intramolecular co-ordination outweighs that of methyl–methyl repulsion, for the coupling constant of the *trans*-derived product (3·4 Hz) is considerably smaller than that of the *cis*-derived product (6·2 Hz). However there is a marked residual steric influence in the stilbene peroxymercurials where the coupling constants (*cis* 6·7 and *trans* 6·0 Hz) are much closer together.

Intramolecular co-ordination of the t-butylperoxy-group to mercury has been suggested before in compounds of the type XHgCH$_2$CR(OOBu^t)COY on the basis of chemical shift data for the diastereoisotopic methylene protons.[19] In the butene and stilbene peroxymercurials, which have the advantage of not containing other potential donor groups, the evidence for intramolecular co-ordination is based on the observed

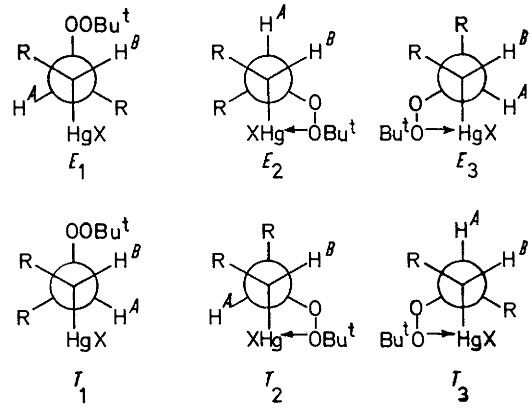


FIGURE 2 Conformations of *erythro*-(E) and *threo*-(T) peroxymercurials (X = OCOCF$_3$)

changes in J_{AB} upon adding pyridine to each isomer. Thus the addition of one molar equivalent of pyridine to a solution of the *cis*-but-2-ene derivative caused J_{AB} to be reduced from 6·2 to 4·8 Hz, whereas similar treatment of the *trans*-but-2-ene derivative *increased* J_{AB} from 3·4 to 4·4 Hz. The corresponding changes for the peroxymercurials derived from *cis*- and *trans*-stilbene were 6·7 to 5·7 and 6·0 to 8·9 Hz respectively. These changes are precisely as predicted if the intramolecular co-ordination which is mainly responsible for conformational preferences in these systems is broken, or at least appreciably weakened, by intermolecular co-ordination of pyridine to the mercury [equation (5)].

$$\mathrm{RCH(OOBu^t)CH(HgTFA)R} + \mathrm{py} \longrightarrow$$

$$\mathrm{RCH(OOBu^t)CHR{\cdot}Hg(py)TFA} \quad (5)$$

(I)

Consistent with these ideas are the vicinal coupling constants for the corresponding methoxymercurials

---

* The term *erythro* is used here to describe the configuration arising from *trans*-addition to a *trans*-alkene.

[18] M. M. Kreevoy and J. F. Schaefer, *J. Organometallic Chem.*, 1966, **6**, 589.

[19] A. J. Bloodworth and R. J. Bunce, *J. Chem. Soc.* (*C*), 1971, 1453.

where no intramolecular co-ordination is expected.* Thus for stilbene,[1] $J_{AB} = 3{\cdot}9$ from *cis* and 7·3 Hz from *trans*, and for but-2-ene perdeuteriomethoxymercurials, $J_{AB} = 4{\cdot}3$ from *cis* and 4·2 Hz from *trans*. Here the comparison between isomers is similar to that in the peroxymercurials when a molar equivalent of pyridine is present. In keeping with the above picture the addition of further molar equivalents of pyridine to the peroxymercurials has comparatively little effect upon J_{AB} (Table).

The nature of the interaction of pyridine with the *trans*-but-2-ene peroxymercurial has been confirmed by isolating a crystalline 1 : 1 complex (I; R = Me). Treatment of this compound with mercuric trifluoroacetate [equation (6)] regenerates the free peroxymercurial in which J_{AB} reverts to its original value.

$$\begin{aligned}&2\text{MeCH(OOBu}^t\text{)CHMe}{\cdot}\text{Hg(py)TFA} + \text{Hg(TFA)}_2 \longrightarrow \\ &2\text{MeCH(OOBu}^t\text{)CH(HgTFA)Me} + \text{py}_2\text{Hg(TFA)}_2 \quad (6)\end{aligned}$$

We are unaware of any reports describing complexes of organomercurials with pyridine, although complexes with more basic amines are known.[20] In fact 1 : 1 complexes with trichloromethylmercury bromide [21] and methylmercury perchlorate [22] appear to be the only previously known complexes of pyridine with organomercury salts. It is noteworthy that in both examples there is a strongly electronegative group attached to mercury thereby enhancing its acceptor properties. The trifluoroacetoxy-group probably performs the same function here.

Coupling constant data certainly indicate that mercury is a stronger acceptor in β-peroxyalkylmercury trifluoroacetates than in the corresponding bromides. Replacement of trifluoroacetate by bromide results in similar though less marked changes in J_{AB} to those observed upon adding pyridine, thus suggesting a weakening of intramolecular co-ordination. The effect of added pyridine upon J_{AB} in the bromides is small and similar for all four compounds indicating little intermolecular interaction.

The changes in J_{AB} induced by adding pyridine to the trifluoroacetates or upon replacing trifluoroacetate by bromide must arise from conformational changes rather than alterations in the coefficients of the Karplus equation as a result of the mercury substituent changing. For such an effect is expected to be small and in the same direction for both diastereoisomers. This was confirmed experimentally for the butene derivatives by simultaneously monitoring the other vicinal coupling constant which is affected by the mercury substituent (J_{BR^2}; Table).

A model of a peroxymercurial which assumes a Hg–C bond length of 2·13 Å [23] and an O–O bond length and angle at oxygen the same as in hydrogen peroxide [24] indicates that the shortest possible $\text{Bu}^t\text{O} \cdots \text{Hg}$ distance in a fully staggered conformation (E_2, E_3, T_2, or T_3) is 2·3 Å which is 0·6 Å less than the sum of the van der Waals radii.[25] This is very favourable towards our suggested intramolecular co-ordination.

In contrast a similar model indicates that intramolecular co-ordination is very improbable in methoxymercurials where the $\text{MeO} \cdots \text{Hg}$ distance is 2·8 Å in the fully *eclipsed* conformation. Likewise no intramolecular co-ordination is expected in aminomercurials and this has been confirmed by *X*-ray diffraction studies for the compound $\text{Et}_2\text{NCH}_2\text{CH}_2\text{HgCl}$.[23] This would appear to invalidate the principal argument upon which the configurational assignments to products of intramolecular aminomercuration were based.[26]

In the β-peroxyalkylmercury trifluoroacetates derived from but-2-ene and stilbene the vicinal H^A–H^B coupling constants and their behaviour upon addition of pyridine or conversion to organomercury bromides present a coherent and compelling case for intramolecular co-ordination and *trans*-peroxymercuration. We were also able to measure the vicinal ^{199}Hg–H^A coupling constants in the diastereoisomers from but-2-ene (Table). However, these are not expected to be as useful as J_{AB} in diagnosing conformational preferences due to intramolecular co-ordination since in such preferred conformations there is one *anti* (E_2 and T_3) and one *gauche* (E_3 and T_2) relationship for *both* diastereoisomers (Figure 2). It is therefore surprising that the ^{199}Hg–H^A coupling constants should differ by as much as 72 Hz. The effect upon J ^{199}Hg–H^A of breaking intramolecular co-ordination by adding pyridine should be the same in both diastereoisomers. This is found, but both coupling constants *increase* instead of decreasing as expected on the basis of increased population of E_1 and T_1 conformations. The effect of the change in the mercury substituent upon co-ordination was again monitored *via* the vicinal coupling to R^2 (Table) and cannot account for this result.

The peroxymercurials investigated here were prepared using a one-fold excess of t-butyl hydroperoxide in dichloromethane.[1] There was no change in the product from *trans*-but-2-ene in either dichloromethane or dioxan when the excess of hydroperoxide was omitted. Thus the stereochemistry of peroxymercuration in inert solvents is the same as that of hydroxy- and methoxy-mercuration where the conditions involve a large excess of hydroxylic reagent.

EXPERIMENTAL

Peroxymercuration.—The preparation and chemical shift data of the compounds investigated here were described in Part VII.[1]

---

* It has been shown that the preferred conformation in 2-methoxyethylmercury chloride is that with the *anti*-disposition of chloromercuri- and methoxy-groups.[13]

[20] G. Spengler and A. Weber, *Brennstoff-Chem.*, 1962, **43**, 234.

[21] A. N. Nesmeyanov, R. Kh. Freidlina, and F. K. Velichko, *Izvest. Akad. Nauk S.S.S.R., Otdel. Khim. Nauk*, 1958, 40.

[22] G. E. Coates and A. Lauder, *J. Chem. Soc.*, 1965, 1857.

[23] K. Toman and G. G. Hess, *J. Organometallic Chem.*, 1973, **49**, 133.

[24] F. A. Cotton and G. Wilkinson, 'Advanced Inorganic Chemistry,' 3rd edn., Wiley Interscience, New York, 1972, p. 415.

[25] D. Grdenić, *Quart. Rev.*, 1965, **19**, 303.

[26] J. Roussel, J. J. Perie, J. P. Laval, and A. Lattes, *Tetrahedron*, 1972, **28**, 701.

Perdeuteriomethoxymercuration.—*cis*-But-2-ene was bubbled for 5 min through a solution of mercuric trifluoroacetate (2·5 mmol) and perdeuteriomethanol (5 mmol) in dichloromethane (3·75 ml) containing one drop of aqueous perchloric acid. The solution was washed with water, dried ($MgSO_4$), and the volatile material removed *in vacuo* to afford an oil (64%), $CH_3^ACH^B(OCD_3)CH^C(HgTFA)CH_3^D$, τ ($CDCl_3$; 32%) 6·57 (dq, J_{BC} 4·3 Hz, H^B), 7·06 (dq, H^C), 8·50 (d, J_{CD} 7·1, $J^{199}Hg$–H^D 305 Hz, H^D), and 8·76 (d, J_{AB} 5·8 Hz, H^A).

trans-But-2-ene similarly gave the diastereoisomer (63%), τ ($CDCl_3$; 32%) 6·2—6·8 (m, H^B and H^C; from 100 MHz spectrum J_{BC} 4·2, $J^{199}Hg$–H^B 440, $J^{199}Hg$–H^C 220 Hz), 8·60 (d, J_{CD} 7·5, $J^{199}Hg$–H^D 314 Hz, H^D), and 8·72 (d, J_{AB} 6·0 Hz; H^A).

^1H *N.m.r. Spectra.*—Peroxymercurials were weighed into the n.m.r. tube and deuteriochloroform and pyridine added by syringe; for solutions of the trifluoroacetates from but-2-ene the total volume of deuteriochloroform plus pyridine was constant (0·5 ml).

Spectra of the trifluoroacetates from cyclohexene, norbornene, *cis*-but-2-ene, and *trans*-but-2-ene were recorded at 27° on a Varian HA 100 spectrometer (100 MHz); the remaining spectra were recorded at 35° on a Perkin-Elmer R12 instrument (60 MHz). ^{199}Hg–H Coupling constants were measured from a 10 p.p.m. sweep and H–H coupling constants by first-order analysis of signals obtained with a 100 Hz sweep.

The spectra of the but-2-ene derivatives (*e.g.* Figure 2) were analysed as follows. (i) The two coupling constants (J_{AB} and J_{BR^2}) were measured from the H^B doublet of quartet. (ii) J_{BR^2} was measured from the R^2 doublet which was identified by its β-coupling to ^{199}Hg; hence J_{AB} was obtained. (iii) The two coupling constants (J_{AB} and J_{AR^1}) were measured from the H^A signal to give J_{AR^1}.

Pyridine Complex.—(a) *Formation.* Heat was evolved when pyridine (5 mmol) was added to a stirred solution of *erythro*-2-t-butylperoxy-3-trifluoroacetoxymercuriobutane (5 mmol) in dichloromethane (4·6 ml). The solvent was removed at 12 mmHg to afford a white crystalline solid (99%), m.p. 57—60° (Found: C, 32·5; H, 3·9; N, 2·5. $C_{15}H_{22}F_3HgNO_4$ requires C, 33·5; H, 4·1; N, 2·6%).

(b) *Decomposition.* A solution of mercuric trifluoroacetate (1·25 mmol) in dichloromethane (8 ml) was added to a solution of the pyridine complex (2·5 mmol) in the same solvent (2·5 ml). Needles began to separate after 5 min and the mixture was cooled in ice until crystallization was complete. The mixture was filtered and the solvent removed at 12 mmHg. The resultant white slurry was extracted with light petroleum (b.p. 40—60°; 2 × 5 ml) and the solvent removed from the extract at 12 then 0·05 mmHg to afford pure (n.m.r.) *erythro*-$MeCH(OOBu^t)$-$CH(HgTFA)Me$ (69%). The solid which had crystallized was identified as $py_2Hg(TFA)_2$ by comparison with an authentic sample.[27]

One of us (I. M. G.) thanks his wife and the S.R.C. for financial support.

[4/1799 *Received*, 29*th August*, 1974]

[27] H. C. Brown and M-H. Rei, *Chem. Comm.*, 1969, 1296.