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THE STEREOCHEMISTRY OF FORWARD AND REVERSE REACTIONS IN THE ADDITION OF HYDRIDOCOBALOXIME TO (*E*)-1-PHENYLPROPENE

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Summary

The addition of deuteriocobaloxime to (*E*)-1-phenylpropene and to indene is stereoselective, the product of *cis*-addition predominating. When stereoselectively labelled 2-[$^2H_1$]-1-phenylpropylcobaloxime decomposes in the presence of oxygen in hydroxylic solvents, the intermediate organic radicals are intercepted by oxygen and then cobaloxime(II) to give the corresponding racemic organoperoxycobaloxime in good yield. However, insertion of oxygen into the carbon–cobalt bond of 1-phenylpropylcobaloxime takes place stereospecifically in the solid state. These results show that the addition reaction takes place by a homolytic transfer of a hydrogen atom from hydridocobaloxime to the olefin; this is followed substantially by stereospecific *cis*-capture of the new organic radical by the cobaloxime(II) fragment within the solvent cage, but some *trans*-capture by cobaloxime(II) already present in solution also takes place to a limited extent.

Hydridocobaloxime (**1**) is formed reversibly by the reaction of molecular hydrogen on cobaloxime(II) (**2**; eq. 1) [1] and by protonation of the cobaloxime(I) ion (eq. 2) [2]. The pK_a of hydridocobaloxime is not known with precision; that determined for hydridobis(dimethylglyoximato)tributylphosphinecobalt(III) being ca. 10.5 based on multisolvent partition studies [2].

$$\tfrac{1}{2}H_2 + \underset{(\mathbf{2})}{Co^{II}(dmgH)_2py} \rightleftharpoons \underset{(\mathbf{1})}{HCo(dmgH)_2py} \quad (1)$$

$$H_3O^+ + [Co(dmgH)_2py]^- \rightleftharpoons \underset{(\mathbf{1})}{HCo(dmgH)_2py} + H_2O \quad (2)$$

However, there is no doubt that hydridocobaloxime, as distinct from the cobaloxime(I) ion and a proton, adds to activated olefins (eq. 3) [3,4,5] and to activated

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acetylenes (eq. 4) [6,7]. The addition to acetylenes is essentially irreversible, whereas that to olefins is demonstrably reversible. Thus, a mixture of 1-phenylethylcobaloxime(III) (**3**) and phenyl acetylene changes to α-styrylcobaloxime(III) and styrene (eq. 5) [5] under anaerobic conditions, and the temperature dependence of the equilibrium constant of the process shown in eq. 6 (a combination of eq. 1 and 3) along with other thermodynamic quantities, has been used by Halpern to determine the bond dissociation energy for the carbon–cobalt bond in 1-phenylethylcobaloxime(III) [8].

$$PhCH{=}CH_2 + HCo(dmgH)_2py \rightleftharpoons PhCH(CH_3)Co(dmgH)_2py \quad (3)$$
(**3**)

$$PhC{\equiv}CH + HCo(dmgH)_2py \rightarrow PhC(=CH_2)Co(dmgH)_2py \quad (4)$$
(**4**)

$$PhCH(CH_3)Co(dmgH)_2py + PhC{\equiv}CH \rightarrow PhC(=CH_2)Co(dmgH)_2py + PhCH{=}CH_2 \quad (5)$$

$$PhCH{=}CH_2 + \tfrac{1}{2}H_2 + Co^{II}(dmgH)_2py \rightleftharpoons PhCH(CH_3)Co(dmgH)_2py \quad (6)$$

Since both cobaloxime(II) (**2**) and hydridocobaloxime (**1**) react rapidly with molecular oxygen [9], reactions 3 and 6 are driven to the left when complex **3** is dissolved in several solvents under aerobic conditions. Complexes **1** and **2** also react irreversibly, but more slowly, with chlorinated solvents such as deuteriochloroform. However, under a variety of conditions the organoperoxycobaloxime(III) complexes such as **5** are formed irreversibly in the presence of oxygen, and may even become the major product of reaction (eq. 7) [10,11].

$$PhCH(CH_3)Co(dmgH)_2py + O_2 \rightarrow PhCH(CH_3)OOCo(dmgH)_2py \quad (7)$$
(**5**)

Using deuterium labelling it has been shown not only that the addition of hydridocobaloxime to phenylacetylene (eq. 4) is of α-regiospecificity (with respect to cobalt) and of total *cis*-stereospecificity (in contrast with the sequential addition of cobaloxime(I) and a proton which is of β-regiospecificity and total *trans*-stereospecificity) but also that the exchange of the hydridic hydrogen of hydridocobaloxime with neutral protic solvents is slow [6]. Earlier studies of the addition of deuteriocobaloxime to (*E*)-1-phenylpropene also suggested that 1-phenylpropylcobaloxime (**6D**) was formed with α-regiospecificity and *cis*-stereospecificity [5]. However, with the equipment available at that time, it was unclear whether the addition reaction was completely stereospecific or merely stereoselective (eq. 8).

$$PhCH{=}CHCH_3 + DCo(dmgH)_2py \rightarrow PhCH(CHDCH_3)Co(dmgH)_2py \quad (8)$$
(**6D**)

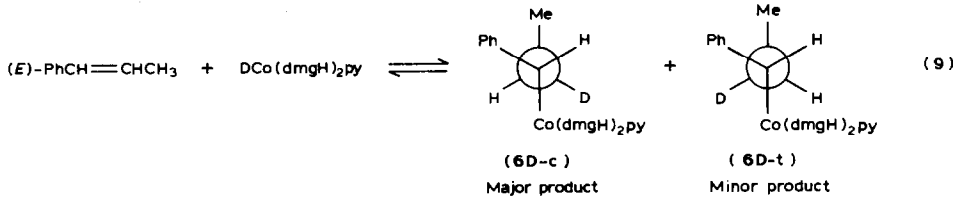
In this paper are described studies of the addition of hydridocobaloxime to (*E*)-1-phenylpropene and to indene, and of the reverse reactions promoted by oxygen and other reagents, in order to establish the stereochemistry of the addition and peroxidation processes and to cast further light on the mechanism of these processes.

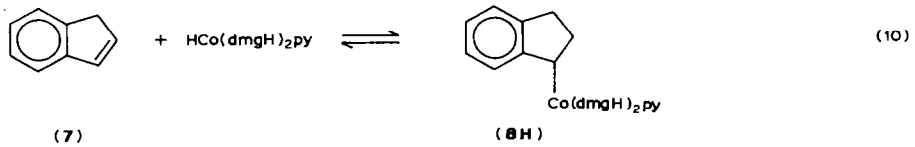
Results and discussion

Regio and stereo-specificity of reaction of hydridocobaloxime with indene and (E)-1-phenylpropene

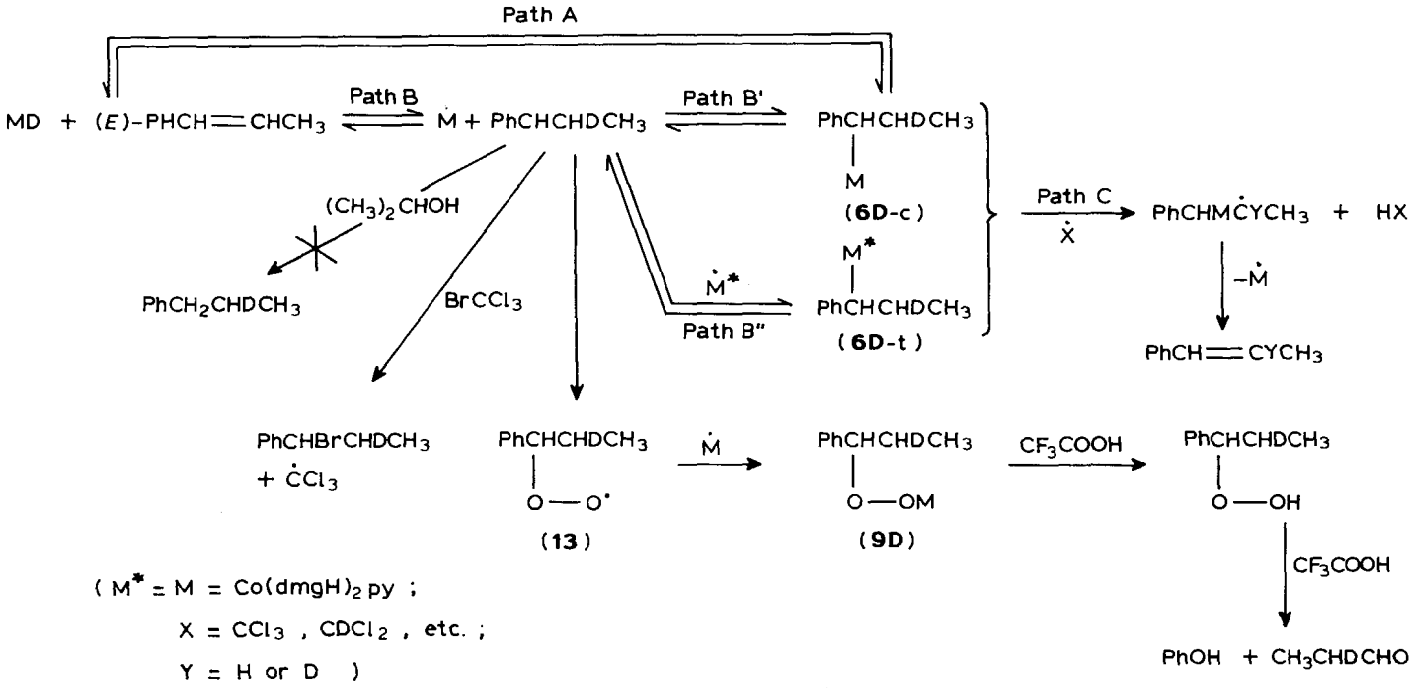
The addition of hydridocobaloxime to (*E*)-1-phenylpropene in 1/1 aqueous methanol gave a good yield (ca. 70%) of 1-phenylpropylbis(dimethylglyoximato)-pyridinecobalt(III) (**6H**; Scheme 1) having an NMR spectrum characterised by a methyl resonance of δ 0.58 (t, 3H) ppm, the α-proton resonance at δ 3.38 (q, *J* 12, and 4 Hz) ppm, multiplets at δ 1.18 and 1.61 ppm for the two β-protons, a pair of singlet resonances for the diastereotopic diagonal pairs of equatorial methyl groups at δ 1.92 and 1.99 ppm, and relatively high field phenyl resonances at δ 6.88 (d, 2H), 6.99 (m, 2H) and ca. 7.25 ppm. In the corresponding reaction of deuteriocobaloxime in a mixture of D_2O and MeOD (2/1 to 1/1) in the presence or absence of an equimolar amount of boric acid, the spectrum of the product showed substantially a doublet resonance (*J* 12 Hz) at δ 3.38 ppm corresponding to one of the diastereoisomers of **6D** with a variable but minor amount (5–20%) of that diastereoisomer having *J* 4 Hz at essentially the same chemical shift. The relative intensity of the β-proton resonance at δ 1.61 ppm was similarly much greater than that at δ 1.18 ppm. Similar product mixtures were formed whether the reaction was carried out by addition of the olefin to preformed hydride or vice versa, at temperatures from −5 to 20°C. In each case, however, the organocobaloxime precipitated out during reaction. No changes in the proportions of the two doublets at δ 3.38 ppm or of the β-proton resonances at δ 1.61 and 1.18 ppm was detected over more than 1 h at 0°C in deuteriochloroform.

Clearly, the addition of deuteriocobaloxime to (*E*)-1-phenylpropene is stereo-selective under the above conditions and not stereospecific as previously supposed [5]. We can assign the configuration of the major and minor diastereoisomers **6D**-c and **6D**-t respectively on the basis of the coupling constants between the α- and β-protons. The large difference in both δ and *J* observed for these protons in **6H** show that there is one conformation that is particularly favoured in **6**; i.e. that with the methyl group *trans* to the cobaloxime moiety. The major diastereoisomer (**6D**-c) with *J* 12 Hz is thus that formed by a *cis*-addition of the metal deuteride to the olefin (eq. 9). The corresponding addition of hydridocobaloxime to indene **7** at 0°C



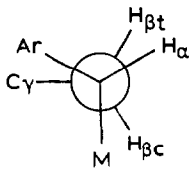


gave a precipitate of indanylcobaloxime (**8H**). As in the case of complex **6H**, the two multiplets of the β-proton resonances are well separated in the NMR spectrum, at δ

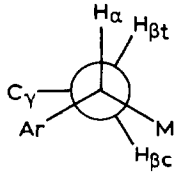

Path A
MD + (E)-PHCH=CHCH3
Path B
M + PhCHCHDCH3
Path B'
PhCHCHDCH3
M
(6D-c)
M*
PhCHCHDCH3
(6D-t)
Path B"
(CH3)2CHOH
PhCH2CHDCH3
BrCCl3
Path C
X
PhCHMCYCH3 + HX
-M
PhCH=CYCH3
PhCHBrCHDCH3
+ CCl3
PhCHCHDCH3
O—O
(13)
M
PhCHCHDCH3
O—OM
(9D)
CF3COOH
PhCHCHDCH3
O—OH
CF3COOH
PhOH + CH3CHDCHO
(M* = M = Co(dmgH)2 py ;
X = CCl3 , CDCl2 , etc. ;
Y = H or D)

SCHEME 1

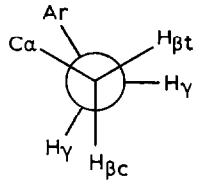
0.63 (*J* 15, 8, 8, and 7 Hz) ppm and at δ 1.76 (*J* 15, 5, 3, and 1 Hz) ppm, but only the larger coupling of 7 Hz is evident in the α-proton resonance at δ 3.56 ppm. Examination of the conformers of **8H** about C_α–C_β, and about C_β–C_γ for the *exo*- and *endo*-conformations (**8**-*exo*-a, **8**-*exo*-b, **8**-*endo*-a, and **8**-*endo*-b) show that only the *exo*-conformation has a dihedral angle of 90° between H_α and H_β, consistent



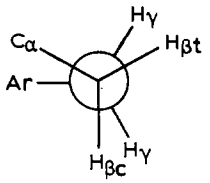
(8-*exo*-a)



(8-*endo*-a)

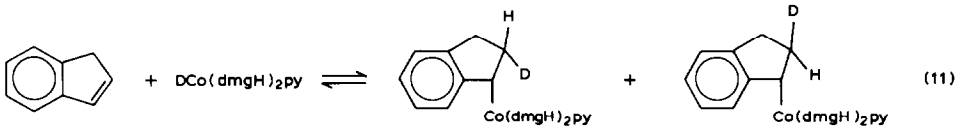


(8-*exo*-b)



(8-*endo*-b)

with the very small coupling constant observed for one of the β-protons. We assume, therefore, that the *exo*-conformation predominates but that the coupling constants are modified by the presence of a small proportion of the *endo*-conformation. Thus for $H_{\beta\text{-}cis}$ with coupling constants of 1 (to H_α), 5 and 3 (to H_γ), and 15 Hz (*geminal*) the resonance occurs at δ 1.76 ppm; and for $H_{\beta\text{-}trans}$ with coupling constants of 7 (to H_α), 8 and 8 (to H_γ), and 15 Hz (*geminal*), the resonance occurs at δ 0.63 ppm. It is thus evident from the NMR spectrum of the product, **8D**, of addition of deuteriocobaloxime to indene, that this reaction is even less stereoselective than that with (*E*)-1-phenylpropene. The resonances at δ 0.63 and 1.76 ppm appear in the ratio 63/37, implying a maximum of 63% of the presumed *cis*-addition product **8D**-c.



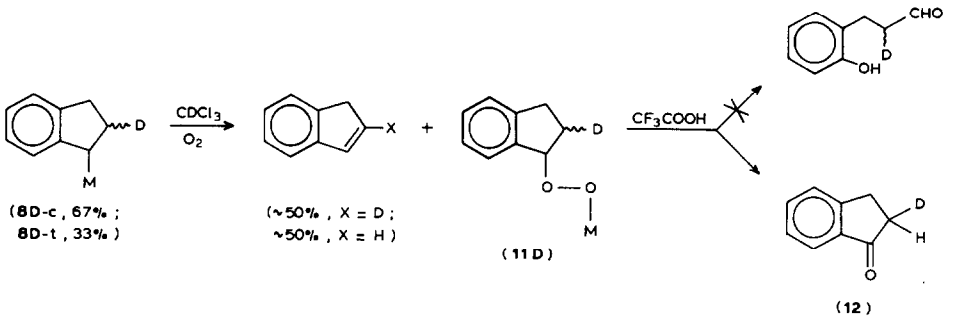
(11)

(8D-c) (8D-t)

No organocobaloxime could be obtained from the reaction of hydridocobaloxime with either 2-methylindene or 2,2′-dimethylindene.

Decomposition of phenylpropyl-, indanyl-, and phenylethyl-cobaloximes

When phenylethylcobaloxime (0.01–0.1 *M*) is dissolved in methanol or in mixtures of methanol and methylene chloride, or isopropanol and methylene



SCHEME 2

chloride (1/1) and air or oxygen is bubbled through the solution phenylethylperoxycobaloxime (**5**) is formed cleanly and near-quantitatively. Phenylpropyl- and indanyl-cobaloximes react similarly to give phenylpropylperoxycobaloxime (**9**) and indanylperoxycobaloxime (**11**), respectively. The reaction of the stereochemically enriched mixture of the diastereoisomeric complexes **6D**-c and **6D**-t (85/15) in the presence of oxygen gave an equimolar mixture of the diastereoisomeric complexes **9D**-c and **9D**-t. Treatment of the latter mixture with trifluoroacetic acid in $CDCl_3$ (4%) gave initially the hydroperoxide **10** and subsequently a mixture of phenol and regiospecifically labelled 2-[$^2H_1$]-propionaldehyde, confirming the exclusive monodeuteriation of the cobaloximes **6D** and **9D** at position-2. The stereochemistry of the indanylperoxycobaloxime **11D**, formed on reaction of oxygen with the less stereochemically enriched mixture of **8D**-c and **8D**-t was not examined in detail, but further reaction with trifluoroacetic acid in $CDCl_3$ gave 2-[$^2H_1$]-indanone (**12**) rather than the expected 3-(*ortho*-hydroxyphenyl)-propionaldehyde (Scheme 2); thus confirming the monodeuteriation of the complexes **8D** and **11D** at position-2.

The insertion of oxygen into the carbon–cobalt bond of phenylethyl- and phenylpropyl-cobaloximes also took place in the solid state over several weeks, and care was necessary in establishing the identity of these substrates before commencing work described here and in the following paper [17]. In the case of the stereochemically enriched mixture of **6D**-c and **6D**-t, the product of insertion of oxygen showed a substantial excess of that phenylpropylperoxycobaloxime, either **9D**-c or **9D**-t, showing a coupling constant of 5 Hz between H_α and H_β. However, in view of the small difference between the coupling constants of the two diastereoisomers (5 and 8.5 Hz) and the problems of assigning the predominant conformations of these complexes, we are unable to state which diastereoisomer predominates in this mixture. However, it seems likely that an oxygen insertion in the solid state would occur with retention rather than with inversion of configuration at the α-carbon, and hence give preferentially **9D**-c.

In our earliest studies of the reactions of the above cobaloximes with oxygen in solution, which were carried out using $CDCl_3$ as solvent, we observed that no matter how high the concentration of oxygen or air in the solution, the olefin was always formed in at least 50% yield. Indeed, the yield of the phenylpropylperoxycobaloxime was lower when the reaction was carried out in the presence of oxygen than in the presence of air.

Mechanism of addition of hydridocobaloxime to activated olefins

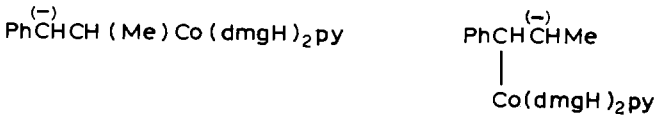
Since the addition of hydridocobaloxime to styrene, indene and phenylpropene is reversible, information about its mechanism can be gained from a consideration of both the forward and reverse reactions, with due regard to any changes of conditions, especially of solvent. Thus, the exclusive formation of phenylpropylperoxycobaloxime **9**, and in particular the formation of racemic **9D** from the diastereoisomerically enriched mixture of **6D**-c and **6D**-t, is indicative of the capture of the phenylpropyl radical by oxygen and the subsequent recapture of the organoperoxyradical **13** (Scheme 1) by cobaloxime(II) either within or without the solvent cage, in a manner similar to that proposed for the formation of racemic sec-octylperoxycobaloxime in the photochemical reaction of oxygen with optically active sec-octylcobaloxime [10,11]. Under our conditions no olefin could be detected, even though any hydridocobaloxime that might have been formed by a concerted elimination process (Scheme 1; path A) would also have been rapidly removed by reaction with oxygen. Since Espenson has shown that the rate of decomposition of the neutral complex **3** in aqueous methanol in the presence of oxygen (the product of which was not determined) [12] is identical with that calculated for the anaerobic decomposition in acidic solution where the olefin is the main product and both cobaloxime(II) and hydridocobaloxime are destroyed by other means, the capture of the organic radical by oxygen and the formation of olefin must occur after the rate-determining cleavage of the carbon–cobalt bond. We conclude, therefore, that the non-radical concerted part A is of negligible importance and that, under other conditions where cobaloxime(II) or hydridocobaloxime are removed, the free radical decomposition to olefin (Paths B and B′) predominates.

The formation of substantial quantities of olefin in solvent $CDCl_3$, even in the presence of an excess of oxygen, is clearly an artifact peculiar to the use of that solvent, a result of the formation of polyhalogenomethyl radicals from the solvent and their attack directly on the organocobaloxime (Scheme 1; Path C). Such a reaction may occur at a β-hydrogen atom with synchronous or subsequent cleavage of the carbon–cobalt bond. Indeed, both the indene formed from **8D** and the phenylpropene formed from **6D** under these conditions contain approximately 50% deuterium at position-2, consistent with a near-random abstraction of a hydrogen or deuterium atom from that position, and different from that expected in the reverse of the addition process, for which an isotope effect and/or a conformational preference should exist. Cobaloxime(II) and organic radicals are known to react with polyhalogenomethanes [13], and it is not surprising, therefore, that some 1-bromo-1-phenylpropane is formed when bromotrichloromethane is also present.

Despite the presence of phenylethyl, phenylpropyl, and indanyl radicals during the decomposition of the above cobaloximes, no hydrogen atom abstraction by these radicals from, for example isopropanol, giving ethylbenzene, propylbenzene or indane, could be detected. Capture of the organic radical by oxygen and the transfer of a hydrogen atom to the cobaloxime(II) radical within the solvent cage appear to be the faster processes.

As far as the addition process is concerned, the formation of a *trans*-addition product (**6D**-t or **8D**-t) is clearly not the result of a concerted one-step addition (Scheme 1; path A). This could imply either that there is the intrusion of an ionic path in which the cobaloxime(I) ion adds and the carbanion so-formed rapidly removes a proton from the solvent, or that the free radical addition allows the

formation of both *cis*- and *trans*-addition products. The carbanionic mechanism is unlikely on two counts: first the proportion of *trans*-addition product, though variable, is not markedly changed when the pH of the solution is lowered by the addition of boric acid. Secondly, the regiospecificity of such an addition would be expected to be different on account of the far greater stability of the intermediate carbanion (**14**) than of the alternative 1-phenylprop-2-ylcarbanion (**15**). Such a change of regiospecificity is observed in the corresponding addition of hydridocobaloxime to acrylonitrile when the pH is lowered [4].



(**14**) (**15**)

The markedly higher proportion of *trans*-addition product in the reaction of hydridocobaloxime with indene (ca. 33%) must thus result from the free radical path. Whereas rotation about the C_α–C_β bond in the intermediate organic radical could account for the small amount of *trans*-addition to phenylpropene, it cannot account for any of the *trans*-addition to indene. The significant extent of the latter thus indicates that the organic radical, whether indanyl or phenylpropyl, is also captured from the opposite side by a different cobaloxime(II) radical (shown in Scheme 1 path B as M*) already present in solution because of incomplete reduction of the reagent chlorocobaloxime(III). Such a process would maintain the amount of free cobaloxime(II) in solution, since the consumption of external cobaloxime(II) sets free the cobaloxime(II) derived from the hydridocobaloxime. The higher proportion of *trans*-addition to indene than to phenylpropene also suggests that the indanyl radical persists longer and has a greater chance of capture by the small proportion of external cobaloxime(II). The need to consider a concerted addition (Scheme 1; path A) is thus obviated.

Whilst, in principle, it should be possible to carry out a series of experiments in which the proportions of cobaloxime(II) and hydridocobaloxime are varied during the preparative addition reaction, the high concentrations necessary for the isolation of the phenylpropylcobaloxime and the instability of the hydridocobaloxime under these conditions are such that it is not practicable to vary with certainty, or to determine with accuracy, the relative concentrations of the two cobaloxime species. The noted variations in the yield of the *trans*-addition product are, however, consistent with the presence of variable proportions of cobaloxime(II) during reaction. Such problems indicate that the study of the reverse reaction, under conditions where cobaloxime(II) and hydridocobaloxime are both destroyed as they are formed, should give a better verification of the stereospecificity of the true addition process involving pure hydridocobaloxime.

The above results all depend upon the characteristic reluctance of the cobaloxime(II) radical to dimerise or disproportionate. They are in accord with the suggestion by Halpern that homolysis of the carbon–cobalt bond followed by hydrogen atom transfer within the solvent cage can explain the close correspondence between the calculated bond dissociation energy in 1-phenylethylcobaloxime and the experimentally determined activation energy for its decomposition into styrene and hydrogen (eq. 6) in toluene under nitrogen [8]. They are also in accord

with the observations of Espenson on the decomposition of 1-phenylethylcobaloxime in acidic aqueous methanol, though in that case a concurrent non-radical concerted elimination path could not be discounted [12].

The assumption that forward and reverse reactions proceed by the same mechanism is certainly valid except in one particular respect caused by the change in conditions. During the addition reaction there may be additional cobaloxime(II) present in the system which can undergo capture of the intermediate organic radical and thus reduce the stereoselectivity. During the decomposition reaction in the presence of reagents (such as O_2 and H_3O^+) which destroy cobaloxime(II), the *trans*-addition path (Scheme 1; paths B and B″) is no longer available. The *cis*-elimination reaction (Scheme 1; paths B and B′) must then be the only path available to either diastereoisomer **6D**-c or **6D**-t, thus making the elimination process stereospecific and showing that the mere stereoselectivity of the addition reaction is an artifact of the conditions required for preparative isolation of the organocobaloxime.

Experimental

Preparation of organocobaloximes

(a) To a suspension of chlorobis(dimethylglyoximato)pyridinecobalt(III) (1 mmol) and the olefin (1 mmol; styrene, (*E*)-1-phenylpropene, indene, 2-methylindene, or 2,2-dimethylindene) in H_2O/MeOH or D_2O/MeOD (25 ml; 1/1 or 2/1) at a temperature of from -5 to $+20°C$ under argon, was added a concentrated solution of $NaBH_4$ or $NaBD_4$ (2 mmol) in H_2O or D_2O (2 ml). In the case of styrene, (*E*)-1-phenylpropene and indene, a precipitate formed which, after stirring the mixture for from 2 to 20 minutes, was filtered off, washed copiously with cold water and pentane, dried in vacuo, and stored under argon. (b) To a suspension of the chlorocobaloxime (1 mmol) in H_2O/MeOH or in D_2O/MeOD, as above, was added $NaBH_4$ or $NaBD_4$ until the suspension remained dark blue-black. The olefin was added and the precipitate was isolated as described above. The phenylethyl- [8,12,14], phenylpropyl- [5,14] and indanyl-cobaloximes [15] have all been described previously. The NMR spectra described in this work were determined using Bruker 250 MHz and Varian 200 MHz instruments, with very dilute solutions at $-20°C$ in $CDCl_3$.

Decomposition of organocobaloximes

The decomposition of the organocobaloximes was studied by the direct observation of the NMR spectral changes in very dilute solutions in $CDCl_3$ under anaerobic and aerobic conditions. The products were separated by column chromatography on silica gel (Malincrodt CC7) and by TLC. Indanyl- and phenylethylperoxycobaloximes have been described [15,16]; and 1-phenylpropylperoxybis(dimethylglyoximato)pyridinecobalt(III) had the NMR spectrum δ 0.67 (t, 3H), 2.09 and 2.22 (2s, 6H), 3.96 (q, α-CH; J 5.3 and 8.4 Hz), 7.22 (m, 5H) ppm; pyridine resonances at δ 7.3, 7.7 and 8.4 ppm. In the reactions carried out in other solvents (such as isopropanol/methylene chloride) the product was poured into a large excess of water and extracted with methylene chloride before working up as above.

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