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ORGANOMETALLIC COMPOUNDS IN ORGANIC SYNTHESIS

XVIII *. REMOVAL OF OMe FROM SOME SUBSTITUTED TRICARBONYLCYCLOHEXADIENEIRONS TO FORM SUBSTITUTED TRICARBONYLCYCLOHEXADIENYLIRON SALTS

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Summary

The structures of tricarbonylcyclohexadienyliron salts resulting from the action of acids on 1- or 2-OMe derivatives of the tricarbonylcyclohexadieneiron series indicate the operation of two alternative processes for removal of OMe. The major one results in a mesomeric cation complex with the complexed system terminating at the carbon initially carrying OMe. The minor process results in a system terminating at the adjacent carbon.

The labelling introduced by the use of deutero-acid relates the initial stages to the Fe-mediated protonations involved in the catalysed rearrangements of such complexes. In the 1-OMe series deuterium is found at the 1-position; in the 2-OMe series a β-D is introduced. Evidence from D-labelled precursors is adduced that in the 2-OMe series there is transfer of H (or D) across the β-face from the 5-position to the 2-position, in permitting cation formation.

The attachment of complexed transition metal atoms to olefinic systems to produce superimposed lateral control of reactivities and structures for organic synthetic purposes has been discussed [2]. Particularly useful compounds are cyclohexadiene-$Fe(CO)_3$ complexes which are, in the form of the derived dienyl salts, synthetic equivalents of chiral cyclohexenone cations and of aromatic cations. They permit, in fully optically resolved form, the production of new asymmetric centres of known absolute configurations [3]. To employ them widely requires a range of methods for their preparations as defined structural isomers, and informa-

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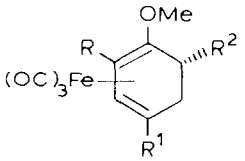
tion on the compatibility of synthetic procedures with retention of optical activity. Some aspects have been discussed [4]. One promising method involves the acid-catalysed removal of OMe from a broad range of neutral complexes available from precursors made by the Birch reduction of anisoles [5].

Results and discussion

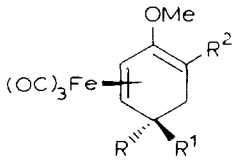
In the first examples studied [5], (**1a** or **b**) and (**2a** or **b**), the mesomeric cationic charge in the product was found to terminate on the position originally bearing OMe (in the major product), whether originally in the 1- or the 2-position. Further examination of these and other complexes, especially using deuterated acids, has shown that the situation is not as simple as appeared, and that mixed products can result, depending on the precursor and the choice of acid.

The mechanisms suggested are shown below for the examples **1b** (Scheme 1) and **2b** (Scheme 2), using D_2SO_4 to provide markers. Justifications of the product structures are given below.



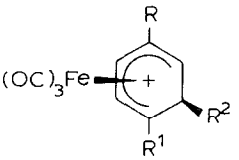
(1)

(**a**, R = R^1 = R^2 = H;
b, R = R^2 = H, R^1 = Me;
c, R = H, R^1 = Me, R^2 = D;
d, R = R^1 = Me, R^2 = H;
e, R = R^2 = H, R^1 = OMe;
f, R = R^2 = H, R^1 = OPr^i)



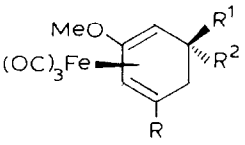
(2)

(**a**, R = R^1 = R^2 = H;
b, R = Me, R^1 = R^2 = H;
c, R = D, R^1 = R^2 = H;
d, R = Me, R^1 = D, R^2 = H;
e, R = D, R^1 = Me, R^2 = H;
f, R = R^2 = Me, R^1 = H)



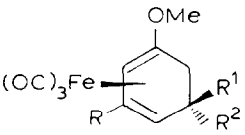
(3)

(**a**, R = D, R^1 = Me, R^2 = H;
b, R = Me, R^1 = D, R^2 = H;
c, R = R^2 = H, R^1 = Me;
d, R = Me, R^1 = H, R^2 = D;
e, R = Me, R^1 = R^2 = D;
f, R = R^2 = H, R^1 = D;
g, R = D, R^1 = R^2 = H;
h, R = OMe, R^1 = Me, R^2 = H;
i, R = Me, R^1 = R^2 = H;
j, R = R^1 = H, R^2 = D;
k, R = H, R^1 = R^2 = D;
l, R = R^1 = R^2 = H)



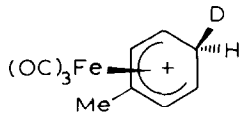
(4)

(**a**, R = R^1 = Me, R^2 = H;
b, R = Me, R^1 = R^2 = H)

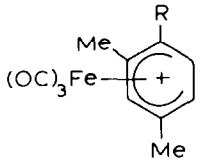


(5)

(**a**, R = R^1 = Me, R^2 = H;
b, R = R^2 = Me, R^1 = H)

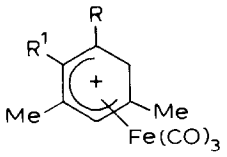


(6)



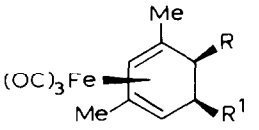
(7)

(**a**, R = H;
b, R = D)



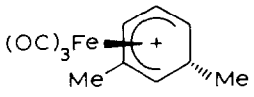
(8)

(**a**, R = R^1 = H;
b, R = D, R^1 = H;
c, R = H, R^1 = D)



(9)

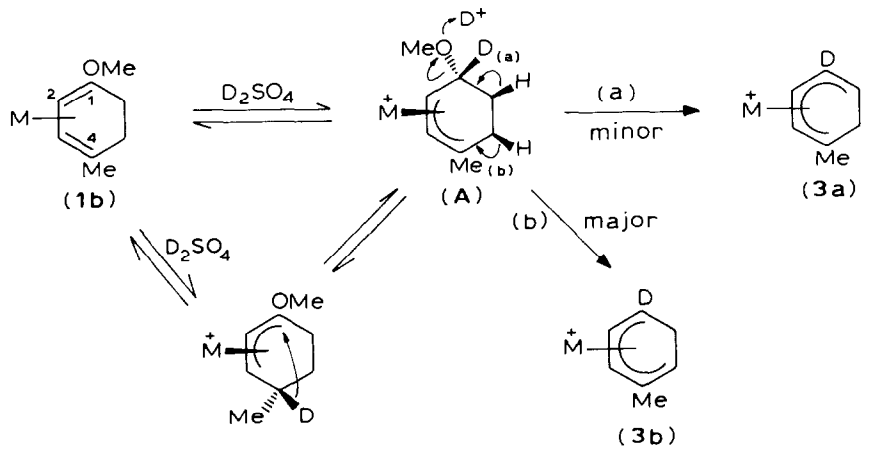
(**a**, R = R^1 = H;
b, R = D; R^1 = H;
c, R = R^1 = D)



(10)

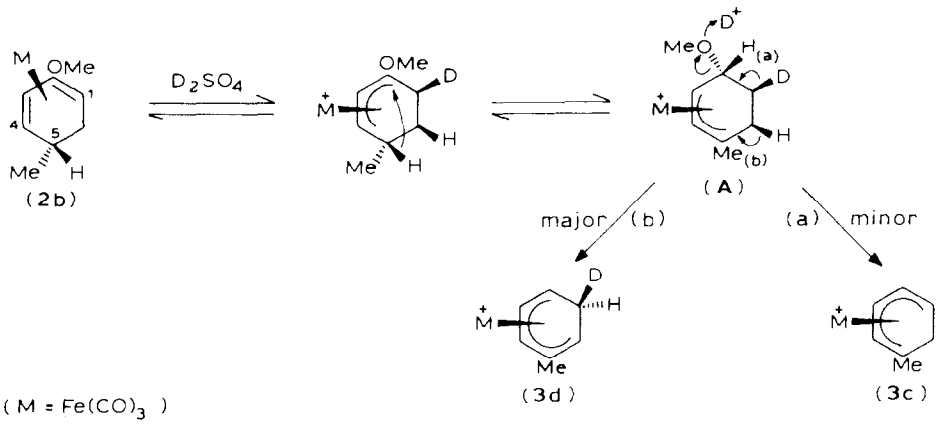
Although many of the formulae represent chiral molecules, only racemic forms were used here. Acid-catalysed isomerisation processes are known [6] to be capable of leading to some racemisation of neutral complexes: for example protonation of **2b** at the 4-position would lead to a symmetrical intermediate allyl cation [6].

SCHEME 1. Demethoxylation in the 1-OMe series.



(M = $Fe(CO)_3$)

SCHEME 2. Demethoxylation in the 2-OMe series.



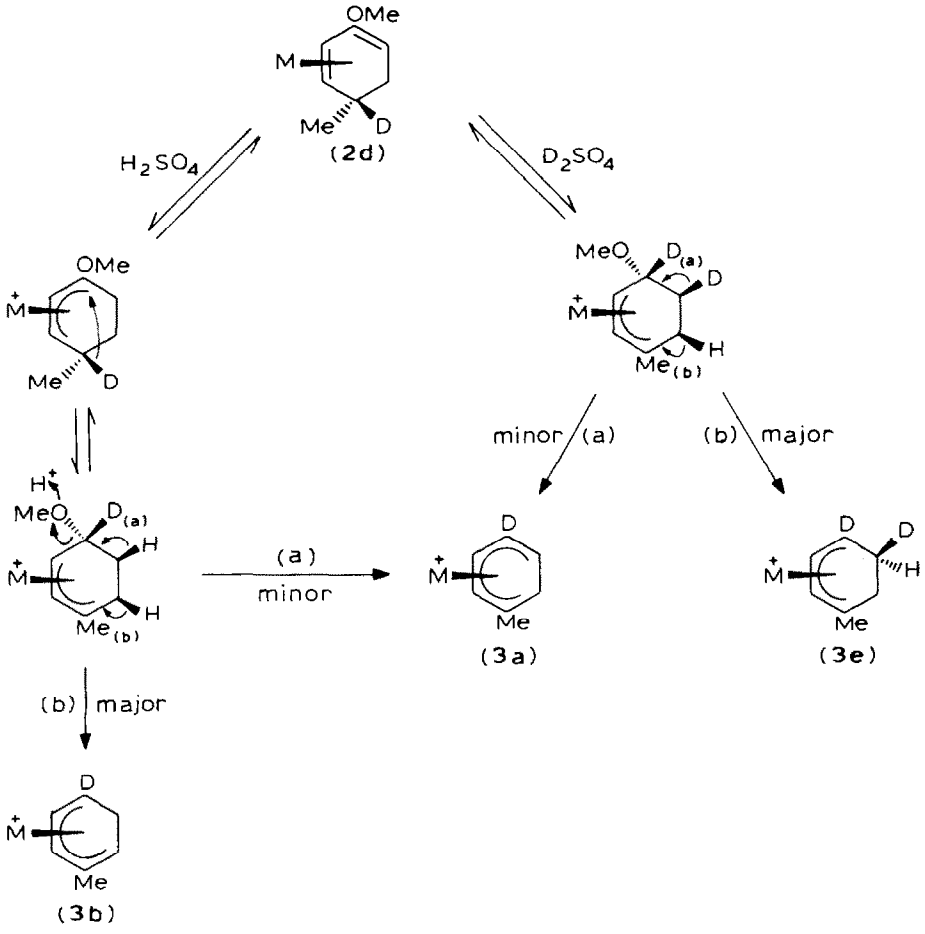
Equilibration between the 1- and 2-OMe complexes **1a** and **2a** can be induced under less vigorous acidic conditions than used here. For instance, the action of TFA at 0°C on either isomer yields a mixture (7/4 in favour of 1-OMe) of the two isomers as the neutral product, a ratio similar to a thermally induced one [7]. Only minor, if any, equilibration seems to occur under the more drastic conditions used in OMe removal with H_2SO_4.

The sequences consummated by loss of OMe would be expected to require the same type of protonated intermediate, i.e. **A** in Schemes 1 and 2. This can be generated as shown from 1-methoxy complexes of type **1** either directly by addition of a $\beta(endo)$-proton to the 1-terminus, or indirectly through the 4-terminus with migration (Scheme 1) by analogy with the route for 2-OMe complexes of type **2** (see below). Both processes would lead with D_2SO_4 through a similar intermediate (**A**) as shown, to the observed products. The sequence from 2-OMe complexes was initially not obvious. The products from D_2SO_4 on **2b** were chiefly the D_1 cation (**3d**), and the undeuterated cation (**3c**), the formation of which may be due to the alternative 1,2-elimination pathway (a), or to traces of proton-acid present or formed by methanol elimination. In a similar case the isotope effect observed is very high [8].

To test the mechanism proposed for type **2** it is necessary to establish the migration of 5β-D and lack of migration of 5α-D. The 5α-deuterated precursor **2c** was obtained by two methods. The Birch reduction product of 4-D-anisole (kindly supplied by Dr. W.M.P. Johnson) on complexation gave a mixture of the 1- and 2-OMe complexes, separated by chromatography. The latter was the 5$\alpha(exo)$-D isomer (**2c**), identical (^{1}H NMR) with the borodeuteride reduction product of the 2-OMe cation salt. The $\alpha(exo)$-disposition of the label was further demonstrated by its complete removal by trityl cation. Reaction with H_2SO_4 gave deuterated salts (**3f**) (1 part) and (**3g**) (5 parts), explicable by the alternative routes (a) and (b), leaving the D in place as expected (cf. Scheme 2).

The 5$\beta(endo)$-D derivative (**2d**) was produced in admixture with isomers **2e** and **1c** by the non-stereoselective borodeuteride reduction of the 2-OMe-5-Me cation (**3h**). The mixed steric course of such reductions is discussed elsewhere [9]. Isomer **1c** was removed by chromatography and from the mixture of the remaining two isomers, trityl fluoroborate selectively removed **2e** to re-form the initial salt. The isomer **2d** is inert to hydride abstraction under the usual conditions since it has no

SCHEME 3. Proposed migration of 5β-D.

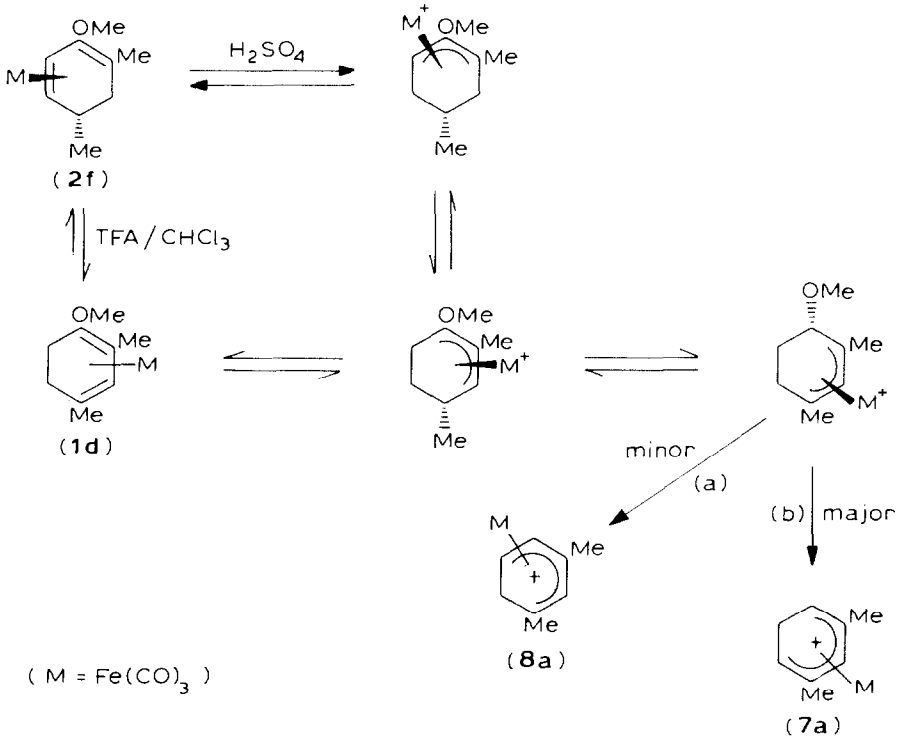


5α(*exo*)-D and the 6α(*exo*)-H is sterically protected by the adjacent Me [4]. With H_2SO_4 or D_2SO_4 (**2d**) underwent the transformations shown (Scheme 3).

Proton additions and removals are presumably stereospecifically β(*endo*)- as in the general series lacking OMe [4,10]. Evidence for the necessity of loss of β-H in both the 1- and 2-OMe series is found in the lack of cation formation from the complexes **4a** and **5a**, where the process is blocked by the presence of β-Me in a situation from which β-H should move. Instead, equilibration of the two isomers was observed yielding predominantly **5a** (about 7/3) with an "outer" OMe and an "inner" Me [4] *. The 5α-Me isomer **5b** with D-TFA gave only the cation with deuterium at the terminus originally occupied by OMe (**8b**) showing that in this case

* The lack of formation of the 2,6α-dimethyl cation via a 1,2-demethoxylation process (e.g. path (a) in Scheme 1) is puzzling. It may indicate that either C(4) protonation occurs with subsequent internal transfer of H to C(1) which in the case of **5a** may be hindered by the β-Me, or that 1,2- and 1,5-demethoxylations are not related by a common intermediate like A (Scheme 1).

SCHEME 4. Reaction of tricarbonyl-1,5α-dimethyl-2-methoxycyclohexa-1,3-dieneiron with H_2SO_4.



demethoxylation is faster than rearrangement, which would leave some deuterium in the alternative 6β-position (cf. Scheme 2).

The complex **4b** with D_2SO_4 showed no sign of rearrangement, producing **6**. Complex **2f** was examined to discover the effect of substitution on a situation which would otherwise be expected to undergo protonation. The Birch reduction product of 2,4-dimethylanisole gave with $Fe(CO)_5$ a mixture of **1d** and **2f**, separated by chromatography. Reaction of **2f** with H_2SO_4 yielded **7a** and **8a** (3/1) presumably via the mechanism shown in Scheme 4. Neither of these corresponds to a direct product from **2f**, so that initial isomerisation to **1d** must have occurred. TFA in chloroform converts **2f** into an equilibrium ratio (1/9) with the more stable isomer **1d** with 75% recovery.

The cation resulting from such acid-catalysed reactions does not necessarily, therefore, correspond directly in structure to the precursor, and checks are required for synthetic uses. The salts, once formed, are stable to the conditions. The general method provides salts not readily available by the classical hydride removal method [11]. In many cases the mixture of 1-OMe and 2-OMe isomers obtained in the initial complexation process from a 1,4-diene can be used without separation, provided deuterated derivatives are not the objective, since they usually lead to what is structurally the same cation.

Preparation of the 3-OMe cation, reported elsewhere [12], subsequently led to the conclusion that in general F_3CCO_2H yields only one cation by a 1,5-process (route (b)) in contrast to H_2SO_4, which in the 3-OMe case produces a mixture containing

about 10% of cation formed by 1,2-elimination of MeOH (route (a)) [12]. The generality of this advantageous result is supported by the observation that while **1b** or **2b** with H_2SO_4 give the 2-Me cation **3i** contaminated with the 1-Me isomer **3c**, TFA gives only the former. Complex **5b** with TFA yields pure **8a** or **8b** using D-TFA, but with H_2SO_4 a mixture of salts. The contrasting results may reflect differing extents of protonation of OMe, or differences in intermediates from TFA, which has a coordinating anion [13], compared with H_2SO_4 which should favour charge-separated species. The nature of the products is also related to factors which include the effects of type and positions of substituents on formations and stabilities of alternative intermediates. The 1,4-dialkoxy complexes (**1e** or **1f**) with either H_2SO_4 or TFA react almost exclusively via the 1,2-elimination mechanism to produce tricarbonyl-2,4-cyclohexadienoneiron [12] rather than a methoxy cation. This is presumably because of involvement of alkoxy oxygen *p*-electrons in deciding the nature of the chosen allylic intermediate.

An example of inhibition of protonation by an Me terminally attached to a complexed diene, uncomplicated by OMe loss, is provided by **9a**, obtained by the standard procedure from *m*-xylene. With D_2SO_4 at 0°C for 5 min, conditions which produced only the D_2-product from the unsubstituted cyclohexadiene complex, a mixture was obtained of **9b** (1 part) and **9c** (4 parts). More prolonged treatment gave only the latter. The introduction of deuterium is postulated to involve the equilibria shown, the first involving reaction at an unmethylated terminus to give **9b**. The second involves reaction at a methylated position, in order to move the complexed system. The relative slowness of the second stage permits detection of the intermediate. No deuterium could be detected in any position other than those shown in **9b** and **9c**.

SCHEME 5. Deuteration of tricarbonyl-1,3-dimethylcyclohexa-1,3-dieneiron.

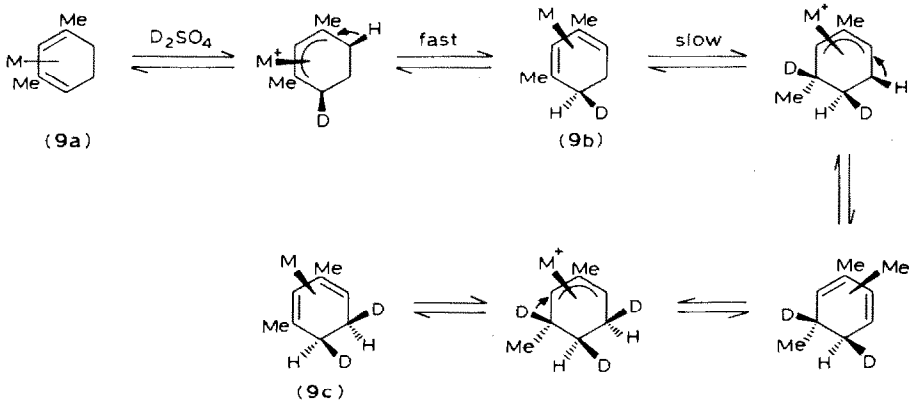


Table 1 summarises the conversions described. To it are appended details of the determinations of structures and the principles used in defining the ratios of cations.

Structures depend largely on ^1H NMR spectra of which characteristic peaks were integrated for ratios. Interpretation was aided by comparisons of the cations containing deuterium with their hydrogen substituted equivalents. Reduction of the

TABLE 1

FORMATIONS OF SALTS AND EQUILIBRATIONS OF COMPLEXES

Precursor [a]	Conditions [b]	Products [c]
1a	H_2SO_4 or FSO_3H	[5]
1a	F_3CCO_2H/30 min/0°C	**1a**/**2a** (7/4) [d]
1a	D_2SO_4	**3f** (3 pts) **3k** (1 pt) [e]
2a	H_2SO_4 or FSO_3H	[5]
2a	F_3CCO_2H/30 min/0°C	**1a**/**2a** (7/4) [d]
2a	D_2SO_4	**3j** (4 pts) **3l** (1 pt) [f]
1b	F_3CCO_2H	**3i** [g]
2b	F_3CCO_2H	**3i** [g]
2b	D_2SO_4	**3c** (3 pts) **3d** (7 pts) [h]
1b	D_2SO_4	**3b** (20 pts) **3a** (1 pt) [i]
2c	H_2SO_4	**3g** (5 pts) **3f** (1 pt) [j]
1d	H_2SO_4	**7a** (3 pts) **8a** (1 pt) [k]
1d	D_2SO_4	**7b** (3 pts) **8c** (1 pt) [m]
2f	H_2SO_4	**7a** (3 pts) **8a** (1 pt) [n]
2d	H_2SO_4	**3b** (7 pts) **3a** (3 pts) [o]
2d	D_2SO_4	**3e** (7 pts) **3a** (3 pts) [p]
4b	D_2SO_4	**6** [q]
9a	D_2SO_4 (5 min)	**9c** (4 pts) **9b** (1 pt) [r]
4a	F_3CCO_2H	**4a** (3 pts) **5a** (7 pts) [s]
2f	$F_3CCO_2H/CHCl_3$	**2f** (1 pt) **1d** (9 pts) [t]
5b	F_3CCO_2H	**8a** [u]
5b	F_3CCO_2D	**8b** [v]
5b	H_2SO_4	**8a** (7 pts) **10** (1 pt) [w]

[a] Precursor preparations are in the literature [11], apart from those recorded in the experimental section. Sometimes it was necessary to separate mixtures by the general method now noted. [b] For conditions see Experimental. [c] The assignments of structures and proportions of products depend on the general principles and procedures already discussed. The indicative ^1H NMR resonances used for structure assignment or analysis by integration are quoted. With deuterated salts, borohydride reduction and mass spectra were used to supplement the NMR. [d] MeO resonances at δ 3.43 (1-OMe) and 3.60 ppm (2-OMe) [11]. [e] The key resonances in the undeuterated unsubstituted cation are: δ 7.4 (t, H(3)), 5.9 (t, H(2,4)); 4.2 (t, H(1,5)); 3.16 (dt, H(6β)); 2.2 (d, J 16 Hz, H(6α)). The spectrum of a mixture was generally compared with that of the corresponding D_0 cation. Decrease of 4.22 in the present mixture indicates the proportion of 1-D isomer; the H(6β) resonance also becomes in part a dd indicating the adjacent D(1). The lower intensity of H(6β) (by about 25%) indicates the presence of some D there. The assigned β-stereochemistry is supported both by the NMR and by the sequence: borohydride reduction, reaction with trityl fluoroborate, borohydride reduction then mass spectrum ($M-CO$). No D was lost. However, the ($M-3CO-2H$ or D) showed large loss of D. This β-assignment of D(6) is also supported by partial collapse of the doublet (H(6)) at 2.2 to a singlet. The mass spectrum of the borohydride product ($M-CO$) showed about 75% D_1 and 25% D_2; with loss of about 90% deuterium in ($M-3CO-2H$). This result also excludes the possibility of the NMR spectrum being due to two alternative D_1 isomers. We estimate the accuracy of analyses at not more than 5%. The other mixtures below are discussed in outline only. [f] The ratio was estimated from 3.16 (H(6β)) to 2.20 (H(6α)); the D_1 content by mass spectrum of the reduced product. [g] Showed 2.29 (s, Me(2)): no 1.95 (s, Me(1)). [h] Presence 1.95 (Me(1)) and 2.29 (Me(2)), compared with undeuterated drop of intensity 3.06 (H(6β)) collapse of 4.22 (d, H(1)) to s and of 4.40(t) to d. Mass spectrum shows about 80% D_1 and therefore **3c** probably contains some D_1. [i] Me(2) is found at 2.29(s); compared with D_0 4.22t has disappeared 5.8 (t, H(4)) has collapsed to d and 3.06 (dt, H(6β)) to dd (J 7, 17 Hz); mass spectrum shows no D_2. Me(1) at 1.95(s). [j] The intensities of 5.96 (H(2,4)) and 4.42 (H(1.5)) compared with D_0 suggests 80% D at the 2-position and 20% at the 1-position. [k] 4.18 (H(1,5)) 3.04 (H(6)) 5.96 (H(4)) used in conjunction with integration of Me singlet resonances. (Me(1) 1.95; Me(2) 2.29; Me(3) 2.86). Also by comparison with *u*. [m] 2.28 (Me_2(2,4) of **7b**) 2.86 (Me(3)) of **8c** and 1.96 (Me(1)) of **8c**. The position of D by comparison with *k*, 4.18 shows one terminal H only, 3.04 (H(6)) has become dd. [n] Product identical with *k*. [o] Main product identical with *i*; ratios by 2.29 (Me(2)) and 1.96 (Me(1)); presence of D(5) in **3b** shown by loss of 4.22 (H(5)); mass

involatile salts with sodium borohydride gave neutral diene complexes which were examined by mass spectrometry. The $(M-(CO)_n)$ $(n=0-3)$ peaks in a mixture permitted deduction of the ratio $D_0/D_1/D_2$. The $(M-3CO-2H$ or D) peaks, due to an aromatic ring attached to Fe, led to the assessment in the reduced product of $\beta(endo)$-D, which is lost preferentially in aromatisation [10], other deuterium remaining in the ion. The conclusions from this procedure do not apply without correction to a cation with a terminal D, since $\alpha(exo)$-reduction at that terminus yields further $\beta(endo)$-D in the product.

An appropriate combination of well-known spectral assignments with methods permits structural definitions of components in mixtures with a high degree of certainty if above about 10% content of an individual isomer is present. The ratios of more abundant isomers quoted are estimated to be correct within $\pm 5\%$.

Experimental

The general methods and equipment were those used in other parts of this Series. Preparative and analytical chromatographic separations (including TLC) were carried out on the neutral complexes in light petroleum solution, containing up to 10% of ether if required. Evidence of the homogeneity of separated complexes was sought in all cases by TLC, ^{1}H NMR and GLC in a glass column (6 ft. by 0.25 in.) packed with acid-washed Chrom-W carrying Carbowax 20M (5%). Deuterated complexes, including borohydride reduction products of all cations or cation mixtures were examined in a mass spectrometer (MS 902) for $(M-n\mathrm{CO})$ $(n=0-3)$ and $(M-3\mathrm{CO}-2\mathrm{H}$ or D) peaks. Analyses of borohydride reduction products of cations by GLC were often not very useful because of mixtures resulting from lack of regioselectivity.

The substituted cyclohexadiene complexes were obtained from the Birch reduction products of the appropriate benzenes, as previously described [11]. However, better yields (70–90% of total complexed product) were obtained by the general experimental procedure described for the methoxy derivatives [14]. This involved intermediate cleaning of the cooled reaction solution by passage through a column of silica and resumption of heating with the addition of $Fe(CO)_5$ (2 equiv.). Two, or at most three periods (16 h) of heating produced complete disappearance of the initial diene.

Mixtures of 1- and 2-methoxy-dienes can generally be separated by column chromatography in light petroleum on silica, the 2-OMe complex emerging first.

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spectrum confirms ratios. [p] Estimation of **3a** by comparison with *o*; **3e** by comparison with **3b** *i*,*o*; loss of 3.16 (H(6β)) and 2.2 (H(6α)) as *s* indicate D(6). [q] By comparison with D_0, 2.90 (H(6β)) has disappeared and 2.00 (d, H(6α)) collapsed to s; 4.21t becomes d (H(1,5)). Reduction followed by treatment with trityl cation does not remove D; some content ($<10\%$) of undeuterated cation cannot be ruled out. [r] Reaction with trityl cation resulted in no loss of D. The resulting salt was compared with the D_0 salt: 4.12 (t, H(5)) and 2.98 (dd, H(6β)) have disappeared while 2.30(d, H(6α)) and 5.96 (d, H(4)) have collapsed to *s*. In the product of brief reaction 4.12 (d, H(5)) is reduced to about 20% compared with D_0; whichever end (5- or 6-) loses H(α) in the original abstraction, only outer H will remain. The mass spectrum $(M-\mathrm{CO})$ indicated about 20% D_1; virtually all D is lost from $(M-3\mathrm{CO}-2\mathrm{H})$. [s] Separated by chromatography using silica gel light petroleum/dichloromethane (4/1). ^{1}H NMR from ref. 11. [t] Separated by chromatography using silica gel light petroleum (see Experimental section). [u] δ(H) 1.78 (Me(1)), 2.72 (Me(3)), 5.92 (d, H(4)), 5.65 (s, H(2)), 4.10 (dt, H(4)), 2.90 (dd, (H(6β), 2.14 (d, H(6α)). [v] δ 1.78 (Me(1)), 2.72 (Me(3)), 4.10 (0.3H), H(5); 0.7 (D) compared with *u*. δ(C) 24.17 (Me(1)), 19.88 (Me(3)). [w] Identified by comparison of ^{1}H NMR spectrum with that of **8a**, see *u*. 2.3 (Me(2)).

The only complexes not previously noted, at any rate as components of mixtures, are those from 1-methoxy-2,4-dimethylcyclohexa-1,4-diene, obtained by Birch reduction of 2,4-dimethylanisole. Complexation of the diene (4.0 g) yielded tricarbonyl-2-methoxy-1,5α-dimethylcyclohexa-1,3-dieneiron (**2f**) (2.1 g) and tricarbonyl-1-methoxy-2,4-dimethylcyclohexa-1,3-dieneiron (**1d**) (3.2 g), separated by chromatography as above. The 2-OMe complex had the resonances (CCl_4) 4.90 (1H, d, *J* 6 Hz, H(3)), 3.70 (3H, s, OMe), 2.52 (1H, q, *J* 6.3 Hz, H(4)), 2.12 (2H, m, H(5,6)), 1.58 (3H, Me(1)), 1.18 (1H, d, *J* 12 Hz, H(6)), 0.90 (3H, d, *J* 6 Hz, Me(5)). The 1-OMe structure was supported by the lack of any resonance (3.0) due to "outer" H on the complexed system and the presence of "inner" H(3) (5.0) and the expected singlets at 2.07, 1.59 (Me) and 3.42 (OMe).

Each complex showed *m/e* 278 (M^+) with consecutive loss of 3CO. The α-configuration of the Me in the 2-MeO case was demonstrated by the lack of reactivity of the compound to trityl fluoroborate.

Acid-catalysed transformations

The complex (200 mg) was dissolved in conc. H_2SO_4 (or 98% D_2SO_4) (0.5 ml) at 0°C and left at about 15°C for 15 min. The mixture was diluted with ether in which the salt is insoluble, and the cation finally precipitated with NH_4PF_6 as the hexafluorophosphate from aqueous solution. Because of known large isotope effects, considerable precautions were taken with the deuterium experiments to exclude moisture. Spectra were examined in trifluoroacetic acid. An alternative procedure for direct examination of spectra was to dissolve the starting material in FSO_3H at −70°C. Demethoxylation using TFA has been described elsewhere [12].

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