Synthesis and Electrochemistry of [Mo(NO){HB(mppz)$_3$}X$_2$] (mppz = 3-*p*-methoxyphenylpyrazolyl, X = CO or Cl); Crystal Structure of [Mo(NO){HB(mppz)$_3$}(CO)$_2$]†

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The new tripodal ligand K[HB(mppz)$_3$] (mppz = 3-*p*-methoxyphenylpyrazolyl) and its complexes [Mo(NO){HB(mppz)$_3$}X$_2$] (X = CO or Cl) have been synthesised. The crystal structure of the dicarbonyl complex shows that the molybdenum atom is in an approximately octahedral co-ordination environment and that the three aryl substituents on the tripodal ligand produce some steric compression of the coligand geometry to give (O)N–Mo–C and C–Mo–C angles in the range 83.7(6)–86.0(6)° as compared to 90.45(30)° for the analogous complex containing the HB(pz)$_3$ (pz = pyrazolyl) ligand.

The tripodal ligands HB(pz)$_3^-$ (pz = $C_3H_3N_2$) and HB(dmpz)$_3^-$ (dmpz = 3,5-$Me_2C_3NH_2$) form stable mono and bis complexes with a wide variety of metal centres in a number of oxidation states ranging from 0 in [Mo{HB(dmpz)$_3$}(CO)$_3$]$^-$ to VII in [ReO$_3${HB(pz)$_3$}].[1–5] Consequently they have found application in the modelling of the reactive centres in such diverse metalloenzymes as the iron-containing haemerythrin and the molybdenum-containing sulfite oxidase.[6,7] These applications have exploited the ability of the hydrotris(pyrazolyl)borate ligands to block three facial co-ordination sites on a metal ion and, in the case of HB(dmpz)$_3$, to restrict the co-ordination number of molybdenum(V) to six by exploiting the steric effects of the 3-methyl substituents.[7,8] However, in addition to regulating the chemical environment of the metal centre, the ligand systems in metalloenzymes also control the environment of the substrate, usually by providing a cavity in which the chemical reactions of the substrate occur. In the HB(pz)$_3$ ligand the three pyrazole ring planes are mutually inclined at *ca.* 120° and in order to create a cavity with such a ligand it is necessary to incorporate planar groups in the 3 position which lie approximately orthogonal to the pyrazole ring planes. It might be expected that steric interactions would cause appended aryl groups in the 3 position to adopt such an orientation with respect to the pyrazolyl rings and so form a trigonal cavity adjacent to a co-ordinated metal centre (Fig. 1). Trofimenko *et al.*[9] have shown that with Co^{2+} the phenyl-substituted ligand HB(3-PhC$_3$N$_2$H$_2$)$_3$ affords a five-co-ordinate complex, [Co{HB(3-PhC$_3$N$_2$H$_2$)$_3$}(NCS)(thf)] (thf = tetrahydrofuran). However, the crystal structure of this molecule revealed that the phenyl rings of the tripodal ligand were not oriented so as to form a trigonal cavity.

We have now synthesised two octahedral complexes containing the related ligand HB(mppz)$_3^-$ (mppz = 3-*p*-methoxyphenylpyrazolyl). The crystal structure of one of these complexes has been determined to establish the orientation of the phenyl rings in these six-co-ordinate species.

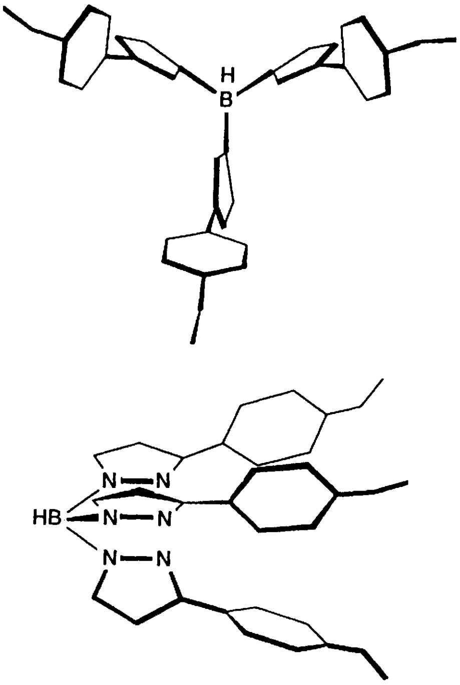

Fig. 1 Possible conformational arrangements of aryl substituents on a tris(pyrazolyl)borate ligand

Results and Discussion

Synthetic Studies.—Following established precedents[9] the new tripodal ligand HB(mppz)$_3^-$ was prepared as its potassium

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† *Supplementary data available:* see Instructions for Authors, *J. Chem. Soc., Dalton Trans.*, 1992, Issue 1, pp. xx–xxv.

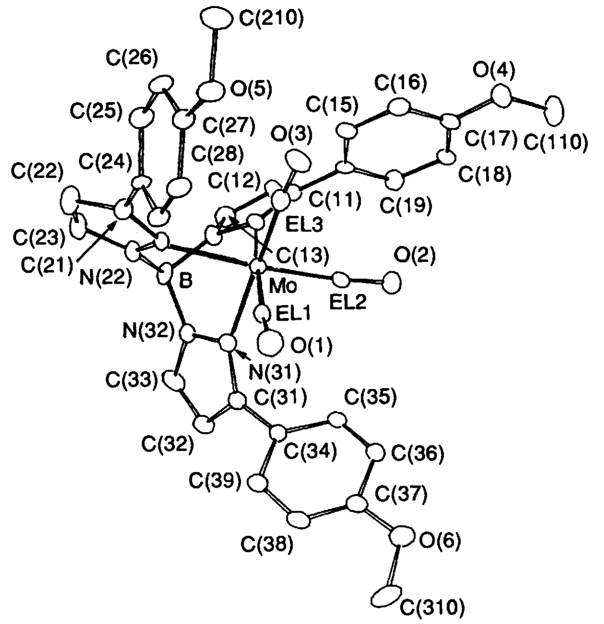

Fig. 2 The structure of [Mo(NO){HB(mppz)$_3$}(CO)$_2$] **1** showing the atom labelling, with 20% probability ellipsoids. Hydrogen atoms have been omitted for clarity

salt in the reaction between KBH_4 and excess of $C_3N_2H_3$-(C_6H_4OMe-*p*)-3. This was then treated with [Mo(CO)$_6$] and butyl nitrite to give [Mo(NO){HB(mppz)$_3$}(CO)$_2$] **1**. Attempts to halogenate **1** using iodine in a reaction analogous to that affording[10] [Mo(NO){HB(dmpz)$_3$}I$_2$] were unsuccessful, leading only to degradation of the complex. Attempts to produce a dichloride complex with NOCl were more successful and gave [Mo(NO){HB(mppz)$_3$}Cl$_2$] **2** in 83% yield.

The IR spectrum of K[HB(mppz)$_3$] contains ν_{BH} at 2350 cm^{-1} compared to values of 2490 and 2500 for **1** and **2** respectively. Respective bands at 1605, 1610 and 1608 cm^{-1} for these three compounds are assigned to the C=C stretching mode of the aryl group and are absent in the spectra of the related compounds containing HB(dmpz)$_3$ in place of HB(mppz)$_3$. The IR spectrum of **1** also contains ν_{CO} at 2010 and 1910 cm^{-1} in addition to ν_{NO} at 1650 cm^{-1}. For **2** ν_{NO} appears at 1710 cm^{-1}.

The ^{1}H NMR spectrum of K[HB(mppz)$_3$] contains signals consistent with trigonal symmetry for the molecule, and with the presence of only one isomer. Since steric interactions would be expected to preclude the presence of three aryl groups in positions adjacent to the BH moiety, we conclude that the pyrazole rings must be substituted in the 3 positions. The ^{1}H NMR spectra of **1** and **2** are qualitatively similar except for some changes in the relative positions of two of the aryl proton signals on replacing CO by Cl. The spectra contain two singlets of relative area 6:3 attributable to the methoxy methyl groups, and two sets of two doublets of relative area 2:2:1:1, attributable to the pyrazolyl protons in positions 4 and 5. Similarly the aryl protons appear as two sets of doublets with relative area 4:4:2:2. These spectra are thus consistent with the presence of a plane of symmetry in the molecules of **1** and **2** and with free rotation of the aryl groups on the NMR time-scale since the two edges of the aryl rings interposed between the Cl or CO and NO ligands are not distinguished.

The electrochemical properties of complexes **1** and **2** were investigated by cyclic voltammetry. In the case of **1** only an irreversible electrochemical oxidation process, for which no corresponding cathodic wave was present, was observed at $E_p{}^a = +1.04$ V *vs.* saturated calomel electrode (SCE). In the case of **2** a quasi-reversible reduction wave was observed at $E_f = +0.08$ V which may be compared to the value of +0.14 V found under similar conditions for [Mo(NO){HB(dmpz)$_3$}Cl$_2$]. The reason for this small cathodic shift in replacing a methyl by an aryl substituent is uncertain. Since it is counter to the expected electronic trend it may reflect increased steric interactions in **2** leading to a weaker interaction between the HB(mppz)$_3$ ligand and Mo than between HB(dmpz)$_3$ and Mo in [Mo(NO){HB(dmpz)$_3$}Cl$_2$]. The Mo–N{HB(mppz)$_3$} bond distances (mean 2.25 Å) in **1** (see below) are longer compared to those of 2.213(4) Å found in [Mo(NO){HB(pz)$_3$}(CO)$_2$].[11]

Structural Studies.—There are four possible isomers of complex **1** which contain a plane of symmetry as required by the NMR spectrum, but only one of these may be deemed likely on steric grounds since the location of a *p*-MeOC$_6$H$_4$ group adjacent to the BH moiety should be energetically unfavourable. However, ligand reorganisation reactions are known[12–14] with the poly(pyrazolyl)borates and a crystal structure of **1** was undertaken to establish the structure unequivocally.

The molecular structure is shown in Fig. 2 and atomic coordinates are presented in Table 1 with selected bond distances and angles in Table 2. It was not possible to distinguish with certainty the nitrosyl nitrogen and carbonyl carbon atoms in the structure. Accordingly these atoms are all identified by the symbol EL in the Tables and Fig. 2. The *p*-MeOC$_6$H$_4$ substituents are all located in the 3 positions of the pyrazolyl rings, as expected, and the molybdenum is in an approximately octahedral co-ordination environment. The aryl rings are inclined at angles of 52.0(4) [C(14)–C(19)], 61.9(4) [C(24)–C(29)] and 55.0(3)° [C(34)–C(39)] [average 56.5(4)°] to their respective attached pyrazolyl ring planes. Thus, although these rings are in an approximately C_3 relationship, they are not truly orthogonal to the mirror planes which would be associated with strict C_{3v} point symmetry and so do not form a perfect trigonal cavity.

The largest deviations from the ideal *cis*-octahedral angle of 90° at Mo are for N(11)–Mo–N(21) which is reduced to 81.4(3)° compared to values of 83.3(3) and 83.6(3)° for the other two angles made by the pyrazolyl N^2 atoms at Mo. This may suggest that the NO group is actually located in this smaller angle between N(11) and N(21). The *cis*-EL–Mo–N angles fall into two groups of three values. The first group [EL1–Mo–N(31), EL2–Mo–N(11), EL3–Mo–N(21)] range from 91.3(5) to 94.5(5)° with a mean of 92.5° and the second [EL1–Mo–N(21), EL2–Mo–N(31), EL3–Mo–N(11)] from 98.6(5) to 101.6(5)° with a mean of 100.1°. These correspond with the orientations of the *p*-MeOC$_6$H$_4$ substituents, the larger angles apparently relieving steric interactions between EL–O and the aryl group which has *o*-CH groups oriented towards EL–O.

The steric consequences of the presence of these substituents may be best assessed by comparing the structure of [Mo(NO){HB(mppz)$_3$}(CO)$_2$] with that of its unsubstituted counterpart [Mo(NO){HB(pz)$_3$}(CO)$_2$].[11] The Mo–EL distance, mean 1.907 Å, is slightly shorter than that of 1.930(4) Å found in [Mo(NO){HB(pz)$_3$}(CO)$_2$]. The Mo–N(pyrazolylborate) distances show a larger difference with a mean value of 2.253 Å for **1** compared to 2.213(4) Å in [Mo(NO){HB(pz)$_3$}(CO)$_2$]. There is no significant difference between the N–Mo–N angles in these two compounds but the angle at boron shows a slight difference with a value of 107.81(28)° in [Mo(NO){HB(pz)$_3$}(CO)$_2$] compared to a mean of 109.6° in **1**. The most significant difference between the two structures lies in the EL–Mo–EL angles which are considerably smaller in **1** (mean 85.0) compared to 90.45(30)° in [Mo(NO){HB(pz)$_3$}(CO)$_2$]. This may be attributed to the steric compression of the Mo(ELO)$_3$ moiety by the bulky 3-*p*-MeOC$_6$H$_4$ substituents as compared to the situation with the unsubstituted tris(pyrazolyl)borate ligand.

Conclusion

The results described here show that, when complexed to Mo^0, the ligand HB[3-(C_6H_4OMe)-$C_3N_2H_2$]$_3$ can accept a co-ordination number of six. However, there do appear to be some

Table 1 Atomic coordinates for $[Mo(NO)\{HB(mppz)_3\}(CO)_2]$ **1**; EL = C or N

Atom	*x*	*y*	*z*
Mo	0.5251(1)	0.2500(0)	0.9797(1)
B	0.5132(10)	0.2611(9)	1.3534(10)
EL1	0.4290(9)	0.1856(5)	0.8249(11)
O(1)	0.3725(8)	0.1499(5)	0.7209(9)
EL2	0.4948(8)	0.3369(6)	0.8320(10)
O(2)	0.4865(7)	0.3875(5)	0.7372(9)
EL3	0.6586(8)	0.2245(5)	0.8747(9)
O(3)	0.7335(7)	0.2087(5)	0.7976(9)
N(11)	0.6220(8)	0.3347(6)	1.1658(11)
N(12)	0.5954(9)	0.3279(7)	1.3122(12)
C(11)	0.6857(10)	0.4048(7)	1.1667(13)
C(12)	0.6958(13)	0.4417(8)	1.3072(17)
C(13)	0.6387(14)	0.3910(9)	1.3957(16)
C(14)	0.7374(9)	0.4345(7)	1.0304(13)
C(15)	0.8107(8)	0.3853(6)	0.9531(11)
C(16)	0.8556(9)	0.4155(6)	0.8269(11)
C(17)	0.8283(12)	0.4931(8)	0.7744(16)
C(18)	0.7569(9)	0.5422(6)	0.8491(12)
C(19)	0.7098(10)	0.5127(6)	0.9770(12)
O(4)	0.8772(9)	0.5168(6)	0.6496(12)
C(110)	0.8469(14)	0.5956(7)	0.5898(13)
N(21)	0.5806(8)	0.1595(5)	1.1684(11)
N(22)	0.5618(10)	0.1779(6)	1.3149(13)
C(21)	0.6456(11)	0.0901(8)	1.1790(15)
C(22)	0.6681(15)	0.0633(9)	1.3329(16)
C(23)	0.6169(15)	0.1184(8)	1.4159(18)
C(24)	0.6952(8)	0.0549(6)	1.0459(13)
C(25)	0.8235(10)	0.0507(7)	1.0595(12)
C(26)	0.8781(9)	0.0223(6)	0.9378(12)
C(27)	0.8038(12)	−0.0014(8)	0.7985(15)
C(28)	0.6755(10)	−0.0015(6)	0.7896(11)
C(29)	0.6233(9)	0.0270(6)	0.9109(11)
O(5)	0.8461(8)	−0.0242(6)	0.6696(12)
C(210)	0.9786(11)	−0.0235(7)	0.6774(14)
N(31)	0.3613(7)	0.2702(4)	1.1010(8)
N(32)	0.3808(7)	0.2713(4)	1.2596(8)
C(31)	0.2373(8)	0.2771(5)	1.0560(11)
C(32)	0.1811(9)	0.2827(6)	1.1840(12)
C(33)	0.2746(10)	0.2786(6)	1.3096(12)
C(34)	0.1735(8)	0.2770(5)	0.8937(10)
C(35)	0.1998(8)	0.3338(6)	0.7857(12)
C(36)	0.1375(8)	0.3339(6)	0.6383(12)
C(37)	0.0429(8)	0.2775(6)	0.5913(12)
C(38)	0.0119(8)	0.2245(6)	0.6946(13)
C(39)	0.0782(8)	0.2231(6)	0.8476(12)
O(6)	−0.0142(7)	0.2823(5)	0.4372(8)
C(310)	−0.1223(10)	0.2352(10)	0.3908(14)

Table 2 Bond distances (Å) and angles (°) in the molybdenum co-ordination sphere of $[Mo(NO)\{HB(mppz)_3\}(CO)_2]$

Mo–N(11)	2.26(1)	Mo–EL3	1.89(1)
Mo–N(21)	2.23(1)	O(1)–EL1	1.17(6)
Mo–N(31)	2.27(1)	O(2)–EL2	1.17(2)
Mo–EL1	1.90(1)	O(3)–EL3	1.18(2)
Mo–EL2	1.93(1)		
Mo–EL1–O(1)	175(1)	EL2–Mo–N(11)	91.3(5)
Mo–EL2–O(2)	174(1)	EL2–Mo–N(21)	172.0(5)
Mo–EL3–O(3)	174(1)	EL2–Mo–N(31)	98.6(5)
EL1–Mo–EL2	86.0(6)	EL3–Mo–N(11)	100.0(5)
EL1–Mo–EL3	85.2(6)	EL3–Mo–N(21)	94.5(5)
EL2–Mo–EL3	83.7(6)	EL3–Mo–N(31)	175.9(5)
EL1–Mo–N(11)	173.9(5)	N(11)–Mo–N(21)	81.4(3)
EL1–Mo–N(21)	101.6(5)	N(11)–Mo–N(31)	83.3(3)
EL1–Mo–N(31)	91.7(5)	N(21)–Mo–N(31)	83.6(3)

steric effects on the geometry of the coligand binding compared to the situation with $HB(pz)_3$.[11] The three anisolyl groups in $[Mo(NO)\{HB(mppz)_3\}(CO)_2]$ tend to form a cavity close to the molybdenum centre in a manner not observed[9] in the five-co-ordinate complex $[Co\{HB(3\text{-}PhC_3N_2H_2)_3\}(NCS)(thf)]$ suggesting that the aryl orientation can vary better to accommodate differing co-ordination numbers. Some chemical consequences are observed in replacing 3-H, 3-Me or 3-Pr^i substituents of the tris(pyrazolyl)borate ligand with aryl groups. Whereas $[Mo(NO)L(CO)_2]$ [L = $HB(pz)_3$, $HB(dmpz)_3$ or $HB(impz)_3$ (impz = 3-isopropyl-5-methylpyrazolyl)] may be halogenated to form $[Mo(NO)LI_2]$,[3,10,13] in the case of $[Mo(NO)\{HB(mppz)_3\}(CO)_2]$ only the dichloride derivative could be isolated. Attempts to prepare the analogous compound containing the larger iodide ligand were unsuccessful.

Experimental

Synthetic and Spectroscopic Studies.—Commercial reagents were used as supplied. 3-*p*-Methoxyphenylpyrazole was prepared using the procedure described previously for 3-phenylpyrazole[9] but substituting 4-methoxyacetophenone for acetophenone. Solvents used as reaction media were dried according to standard methods and freed of oxygen before use. All reactions involving molybdenum complexes were carried out under nitrogen but purification procedures were carried out in air. Activated alumina UGI (100 mesh) or silica gel 60 (70–230 mesh) were used as the stationary phase for column chromatography.

Infrared spectra were recorded on a Perkin-Elmer 297 spectrometer with samples as KBr discs, 270 MHz ^1H NMR spectra on a JEOL GX 270 spectrometer and mass spectra using a Kratos MS80RF spectrometer. Elemental analyses were carried out by the Microanalytical Laboratory, University of Birmingham.

Potassium hydrotris(p-*methoxyphenylpyrazol*-1-*yl*)*borate*, $K[HB(mppz)_3]$. A mixture of KBH_4 (0.4 g, 8.0 mmol) and Hmppz (3.87 g, 22.0 mmol) was heated with stirring. Hydrogen evolution commenced when the mixture reached *ca.* 140 °C and vigorous evolution of gas occurred while the temperature was increased to 170 °C. After *ca.* 400 cm^3 of H_2 had been collected the reaction temperature was raised to *ca.* 190 °C and maintained between 190 and 210 °C until the stoichiometric volume of hydrogen (520 cm^3) had been evolved. The white solid obtained on cooling the mixture was suspended in heptane (100 cm^3) and collected by filtration. The residual excess of pyrazole was removed by sublimation to afford the product (2.68 g, 64%) (Found: C, 63.5; H, 5.1; N, 14.8. $C_{30}H_{28}BKN_6O_3$ requires C, 63.1; H, 4.9; N, 14.7%); $\nu_{max}(BH)$ 2350 cm^{-1}; $\delta_H(CDCl_3)$ 7.68 and 6.89 [2 H, d, *J*(HH) 9; 2 H, d, *J*(HH) 9, C_6H_4], 7.43 and 6.38 [1 H, d, *J*(HH) 2; 1 H, d, *J*(HH) 2 Hz, $C_3N_2H_2$], and 3.75 (3 H, s, OCH_3).

$[Mo(NO)\{HB(mppz)_3\}(CO)_2]$. To a solution of $K[HB(mppz)_3]$ (1.7 g, 3.0 mmol) in tetrahydrofuran (20 cm^3) was added $[Mo(CO)_6]$ (0.76 g, 3.0 mmol). The mixture was heated under reflux until no further gas evolution was observed and the orange solution cooled. An excess of butyl nitrite (0.527 cm^3, 4.5 mmol) was then added and the mixture heated under reflux until gas evolution ceased. The solution was poured into water (100 cm^3) and the product extracted into dichloromethane and purified by column chromatography on alumina with dichloromethane as the eluent. The major orange band was collected and afforded a yellow solid which was further purified by recrystallisation from methanol to give the pure product (0.9 g, 40%) (Found: C, 53.7; H, 3.8; N, 13.7. $C_{32}H_{28}BMoN_7O_6$ requires C, 53.9; H, 4.0; N, 13.7%); $\nu_{max}(BH)$ 2490, $\nu_{max}(CO)$ 2010, 1910, $\nu_{max}(NO)$ 1650s cm^{-1}; $\delta_H(CDCl_3$, 270 MHz) 6.14, 6.32, 7.67 and 7.78 [1 H, d, *J*(HH) 2; 2 H, d, *J*(HH) 2, 1 H, d, *J*(HH) 2; 2 H, d, *J*(HH) 2, $C_3N_2H_2$], 6.86, 6.93, 7.20 and 7.44 [2 H, d, *J*(HH) 9; 4 H, d, *J*(HH) 9; 2 H, d, *J*(HH) 9; 4 H, d, *J*(HH) 9 Hz, C_6H_4], 3.78 and 3.80 (3 H, s; 6 H, s, OCH_3). *m/z* 657 $(M - 2CO)^+$. $E_p{}^c$ (irreversible, CH_2Cl_2, *vs.* SCE) = +1.04 V, $E_f\{[Fe(C_5H_5)_2]^+–[Fe(C_5H_5)_2]\}$ = +0.55 V.

$[Mo(NO)\{HB(mppz)_3\}Cl_2]$. Nitrosyl chloride was bubbled through a suspension of $[Mo(NO)\{HB(mppz)_3\}(CO)_2]$ (0.9 g, 1.2 mmol) in dichloromethane (250 cm^3). The solution became orange then red and finally violet after *ca.* 15 min. At this time the solvent was evaporated off and the solid residue suspended in hexane (50 cm^3). The product was filtered off, washed with hexane (3 × 10 cm^3) and dried to give a violet solid (0.72 g, 83%) (Found: C, 49.6; H, 3.8; N, 13.4. $C_{30}H_{28}BCl_2MoN_7O_3$ requires C, 49.5; H, 3.9; N, 13.5%); ν_{max}(BH) 2500, ν_{max}(NO) 1710 cm^{-1}; δ_H($CDCl_3$, 270 MHz) 6.22, 6.37, 7.72 and 7.93 [1 H, d, *J*(HH) 2; 2 H, d, *J*(HH) 2; 1 H, d, *J*(HH) 2; 2 H, d, *J*(HH) 2, $C_3N_2H_2$], 6.67, 6.88, 7.18 and 7.46 [2 H, d, *J*(HH) 9; 4 H, d, *J*(HH) 9; 4 H, d, *J*(HH) 9; 2 H, d, *J*(HH) 9 Hz, C_6H_4], 3.72 and 3.80 (3 H, s; 6 H, s, OCH_3). *m/z* 725 (M^+), 690 $(M - Cl)^+$ and $(M - NO)^+$ (both ions are encompassed by the *m/z* range of this ion cluster), and 657 $(M - 2Cl)^+$. E_f (quasi-reversible, CH_2Cl_2, *vs.* SCE) = +0.08 V, ΔE_p = 115 mV, $E_f\{[Fe(C_5H_5)_2]^+–[Fe(C_5H_5)_2]\}$ = +0.55 V, $\Delta E_p\{[Fe(C_5H_5)_2]^+–[Fe(C_5H_5)_2]\}$ = 115 mV.

Structural Studies.—Diffraction data were measured on a kappa diffractometer operating in the $\omega–2\theta$ mode with graphite-monochromated Mo-Kα radiation (λ = 0.710 69 Å) up to $\theta = 25°$ from yellow crystals of size 0.3 × 0.3 × 0.2 mm coated in epoxy resin. Three standard reflections were monitored periodically to check the stability of the system. 4965 Unique reflections were scanned and 2508 with $I > 2\sigma(I)$ were considered observed and used in the analyses.

Crystal data. $C_{32}H_{28}BMoN_7O_6$, M_r = 713.4, monoclinic, space group $P2_1$, a = 10.972(1), b = 16.520(3), c = 8.743(1) Å, β = 100.13(9)°, U = 3123.8(9) Å^3, Z = 2, D_c = 1.52 g cm^{-3}, μ(Mo-Kα) = 4.63 cm^{-1}, $F(000)$ = 728.

The cell dimensions were refined by least-squares fitting of the values of 25 reflections. The structure was solved by Patterson and Fourier methods using the X-Ray 80 system[14] and refined by least squares. The *y* coordinate of the Mo atom was held fixed. The N atom of the nitrosyl ligand and the C atoms of the carbonyl ligands became indistinguishable. The positions corresponding to these three atoms were considered occupied by C and N atoms with population factors of 0.66 and 0.34 respectively. The intensities were corrected for Lorentz and polarisation effects. Scattering factors for neutral atoms and anomalous dispersion corrections for Mo were taken from ref. 15. An empirical absorption correction[16] was applied at the end of the isotropic refinement and final refinement was made with anisotropic thermal motion for the non-hydrogen atoms and fixed isotropic thermal parameters and calculated coordinates for hydrogen atoms. Refinement was terminated when all shift/e.s.d. ratios were < 0.12 : 1 and R, R' = 0.04, 0.043, unit weights were used.

Additional material available from the Cambridge Crystallographic Data Centre comprises H-atom coordinates, thermal parameters, and remaining bond lengths and angles.

Acknowledgements

We are grateful to Direccion General de Investigaccion Cientifica y Tecnica of the Ministry of Education and Science of Spain for support through the PB89-0127 project.

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Received 3rd *March* 1992; *Paper* 2/01133F