Synthesis and Electrochemistry of $[\{Mo(NO)[HB(3,5\text{-}Me_2C_3HN_2)_3]Cl\}_2\{1,2\text{-}(NH)_2C_6H_4\}]$ and the Crystal Structure of $[\{Mo(NO)[HB(3,5\text{-}Me_2C_3HN_2)_3]I\}_2\{1,2\text{-}(NH)_2C_6H_4\}]$†

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Cyclic voltammetry of the 1,2-phenylenediamide-bridged bimetallic complex $[\{Mo(NO)L(Cl)\}_2\{1,2\text{-}(NH)_2C_6H_4\}]$ [L = $HB(3,5\text{-}Me_2C_3HN_2)_3$] has revealed two one-electron reduction processes separated by 770 mV, a value which is in accord with the presence of strongly interacting molybdenum centres but intermediate between those found for the corresponding complexes containing 1,4- and 1,3-disubstituted aryl bridging groups. The crystal structure of $[\{Mo(NO)L(I)\}_2\{1,2\text{-}(NH)_2C_6H_4\}]$ has been determined. It contains Mo–N(arylamide) distances of 1.957(6) Å and reveals a non-planar structure for the MoN(amide)–C–C–N(amide)Mo moiety which may account for the lower than expected Mo $\cdots$ Mo interaction detected electrochemically.

The co-ordinatively unsaturated $\{Mo(NO)L(X)\}^+$ [L = $HB(3,5\text{-}Me_2C_3HN_2)_3$] moiety forms remarkably stable alkyl and aryl amide complexes.[1] In these compounds the electron deficiency of the formally 16-electron molybdenum centre is relieved to some extent by N $p_\pi \rightarrow$ Mo d_π charge donation, as indicated by the short Mo–N(aryl) bond distances.[2,3] Electrochemical studies have shown[4,5] that complexes of the type [Mo(NO)L(X)(NHR)] (X = Cl or I, R = Ph or substituted aryl) undergo a one-electron reduction process at potentials which vary with the nature of R. The EPR studies of related systems indicate that the added electron is largely molybdenum centred.[6,7] Electrochemical studies of bimetallic complexes containing bifunctional aryl amides reveal strong metal–metal interactions through the aryl bridge. Thus, rather than one two-electron reduction process, these complexes exhibit two one-electron processes which are separated by a potential difference reflecting the extent of metal–metal interaction. In the case of $[\{Mo(NO)L(Cl)\}_2\{1,4\text{-}(NH)_2C_6H_4\}]$ this separation was found to be 850 mV, while for the 1,3-substituted compound in which the sites of substitution on the aryl ligand are not mesomerically linked this value falls to 390 mV.[8,9] In order to complete the series of aryl substitutional isomers we have synthesised $[\{Mo(NO)L(Cl)\}_2\{1,2\text{-}(NH)_2C_6H_4\}]$ and determined its electrochemical properties. We have previously reported[10] the synthesis of $[\{Mo(NO)L(I)\}_2\{1,2\text{-}(NH)_2C_6H_4\}]$ but were unable to assess the coupling between the metal centres by electrochemistry because of the irreversible and polyelectronic nature of the second reduction process. This may be associated with I^- dissociation from the monoreduced species as has been observed previously.[11,12] However, we have been able to obtain single crystals of this material and have carried out an X-ray diffraction study. This represents the first such study of a bimetallic aryl bridged complex of this type containing two $\{Mo(NO)L(I)\}^+$ moieties.

Experimental

All commercial reagents were used as supplied; $[Mo(NO)LCl_2]$ was prepared according to previously described methods.[13] Toluene used as the reaction medium was dried and freed of oxygen before use by distillation from sodium–benzophenone under nitrogen. Reactions were carried out under nitrogen but purification procedures were carried out in air. Silica gel 60 (70–230 mesh) was used as the stationary phase for column chromatography. Elemental analyses were carried out by the Microanalytical Laboratory, University of Birmingham.

Infrared spectra were recorded using KBr pellets and a Perkin Elmer 297 spectrometer, 270 MHz ^1H NMR spectra from solutions in $CDCl_3$ using a JEOL GX270 spectrometer at the University of Birmingham and mass spectra using a Kratos MS80RF spectrometer. Cyclic voltammetric measurements were made using a platinum-bead electrode with a PAR 174A polarograph and 10^{-3} mol dm^{-3} solutions of complex in CH_2Cl_2 containing 0.2 mol dm^{-3} $[NBu_4][BF_4]$ as base electrolyte. Values are referred to the saturated calomel electrode (SCE) but ferrocene (E_f = 0.55 V, ΔE_p = 90 mV) was used as an internal standard. The number of electrons involved was assessed by comparison with isomeric complexes.[8,9] A scan rate of 200 mV s^{-1} was used and the effect of scan rate on diffusion current was assessed over the range 10–500 mV s^{-1}.

$[\{Mo(NO)L(Cl)\}_2\{1,2\text{-}(NH)_2C_6H_4\}]$.—The complex $[Mo(NO)LCl_2]$ (0.5 g, 1.01 mmol) and 1,2-diaminobenzene (48 mg, 0.5 mmol) were added to dry toluene (35 cm^3). Triethylamine (0.2 cm^3) was added and the mixture heated under reflux for 16 h. After this time the mixture was cooled and filtered and the solvent removed from the filtrate by evaporation under reduced pressure. The residue was purified by column chromatography on silica gel using dichloromethane as the eluent. Four bands were observed: the first (yellow) and second (purple) bands contained very little material. The third (purple) band contained the product and the fourth (orange) band decomposed on the column. The third band was collected and further purified by recrystallisation using dichloromethane to dissolve the material followed by the dropwise addition of hexane and slow evaporation of the solvent *in vacuo* (yield 36 mg, 6%) (Found: C, 43.9; H, 5.7; N, 21.5. Calc. for $C_{36}H_{50}B_2Cl_2Mo_2N_{16}O_2\cdot 0.33C_6H_{14}$: C, 43.4; H, 5.3; N, 21.3%).

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

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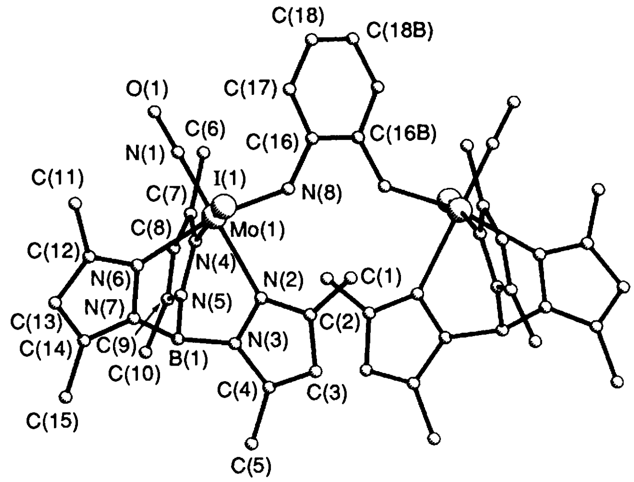

Fig. 1 A view of $[\{Mo(NO)L(I)\}_2\{1,2\text{-}(NH)_2C_6H_4\}]$ showing the atom numbering; hydrogen atoms have been omitted for clarity

Table 1 Fractional atomic coordinates (× 10^5 for Mo and I, × 10^4 for other atoms) with e.s.d.s in parentheses

Atom	x	y	z
Mo(1)	42 992(3)	26 664(4)	41 901(5)
I(1)	53 622(3)	28 041(4)	58 973(4)
N(1)	3 939(4)	3 591(4)	4 361(5)
N(2)	4 750(3)	1 502(3)	3 951(4)
N(3)	4 296(3)	866(3)	3 749(4)
N(4)	3 388(3)	2 315(3)	2 984(5)
N(5)	3 135(3)	1 571(3)	2 908(5)
N(6)	3 657(3)	2 006(3)	4 930(4)
N(7)	3 427(3)	1 269(4)	4 611(4)
N(8)	4 822(4)	3 043(3)	3 353(5)
O(1)	3 703(4)	4 178(4)	4 475(5)
B(1)	3 499(5)	964(5)	3 683(6)
C(1)	6 054(5)	1 734(5)	4 183(7)
C(2)	5 406(6)	1 246(5)	3 979(5)
C(3)	5 342(5)	441(5)	3 785(6)
C(4)	4 667(5)	230(4)	3 662(5)
C(5)	4 327(6)	−551(5)	3 441(8)
C(6)	3 048(6)	3 569(5)	2 090(7)
C(7)	2 939(4)	2 723(5)	2 256(6)
C(8)	2 407(4)	2 225(5)	1 713(6)
C(9)	2 537(4)	1 519(5)	2 140(6)
C(10)	2 109(5)	768(6)	1 918(8)
C(11)	3 571(5)	2 898(6)	6 194(7)
C(12)	3 432(5)	2 148(4)	5 655(6)
C(13)	3 068(5)	1 509(5)	5 824(6)
C(14)	3 052(5)	966(5)	5 143(6)
C(15)	2 686(5)	183(5)	4 945(7)
C(16)	4 932(4)	3 738(4)	2 947(6)
C(17)	4 905(6)	4 448(5)	3 390(8)
C(18)	4 961(8)	5 137(5)	2 942(12)

The complex $[\{Mo(NO)L(I)\}_2\{1,2\text{-}(NH)_2C_6H_4\}]$ was prepared as described previously,[10] and crystals were obtained by slow evaporation of a solution in CH_2Cl_2–hexane.

*Structural Studies.**—*Crystal data.* $C_{36}H_{50}B_2I_2Mo_2N_{16}O_2$, $M = 1206.2$, monoclinic, space group $C2/c$, $a = 19.62(2)$, $b = 17.15(2)$, $c = 14.913(9)$ Å, $\beta = 108.26(7)°$, $U = 4765$ Å^3, $Z = 4$, $D_c = 1.681$ g cm^{-3}, $F(000) = 2376$. μ(Mo-Kα) = 1.869 mm^{-1}, $\lambda = 0.710\,69$ Å. A crystal (0.4 × 0.4 × 0.5 mm) was mounted on an Enraf-Nonius CAD-4 diffractometer; cell dimensions and intensities were measured by ω–2θ scans with graphite-monochromated Mo-Kα radiation. 4201 Unique reflections were scanned within $2 < \theta < 25°$. Three standard reflections measured every 2 h showed no significant variation in intensity. 2837 Structure amplitudes with $F > 5\sigma(F)$ were considered observed and used in the analysis.

The structure was determined[14] by Patterson and Fourier methods and refined[15] by least-squares methods using anisotropic thermal parameters for the heavier atoms. Hydrogen atoms were placed in calculated positions riding on their respective bonded atoms with isotropic thermal parameters $U = 0.15$ Å^2 for methyl hydrogen atoms and 0.10 Å^2 for the other H atoms. Weights, $w = 1/\sigma^2(F)$, which resulted in a satisfactory weighting analysis, were used in the least-squares refinement. The refinement converged to $R = 0.043$, $R' = 0.064$ with a maximum shift/error ratio of 0.08 in the final refinement cycle. The residual electron density in a final difference map was within the range −1.04 to 0.69 e Å^{-3}. The data were corrected for absorption by means of an empirical technique.[16] Computations were carried out on the University of Birmingham IBM 3090 computer and at Manchester Computing Centre with the SHELXS 86,[14] SHELX 76,[15] DIFABS[16] and PLUTO[17] programs.

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

Discussion

Synthesis and Electrochemistry.—The bimetallic complex $[\{Mo(NO)L(Cl)\}_2\{1,2\text{-}(NH)_2C_6H_4\}]$ was prepared from the reaction of $[Mo(NO)LCl_2]$ and $1,2\text{-}(NH_2)_2C_6H_4$ following established procedures.[8] Its yield was lower than those found for the 1,3 and 1,4 isomers[8,9] or of the corresponding iodide complex.[10] This may be the result of ligand-degradation reactions, which have been observed previously in reactions with 1,2-disubstituted aryl ligands,[10,18] and which might compete more strongly in this case because of the less facile substitution of chloride compared to iodide. The IR spectrum of the chloro complex contained ν_{BH} at 2540 and ν_{NO} at 1645 cm^{-1} in addition to other bands attributable to the presence of the aryl and dimethylpyrazolyl groups. The 270 MHz ^{1}H NMR spectrum contained six signals of relative area 3 at δ 1.80, 2.05, 2.32, 2.35, 2.41 and 2.60 together with three signals of relative area 1 at δ 5.39, 5.58 and 5.81 attributable to the presence of the ligand L. The amide NH protons appeared as a broad singlet of relative area 1 at δ 12.16 and the aryl protons appeared as two quartets each of relative area 1 at δ 7.37 and 8.23 ($^3J = 5.9$, $^4J = 3.5$ Hz). Signals attributable to some residual hydrocarbon solvent were also present. The spectrum was consistent with the presence of two $\{Mo(NO)L(Cl)NH\}$ substituents in the 1,2 positions of the aryl ring in a structure which has C_2 symmetry as found in the structural study (see below).

The cyclic voltammogram of $[\{Mo(NO)L(Cl)\}_2\{1,2\text{-}(NH)_2C_6H_4\}]$ contained an irreversible oxidation process at +1.45 V and two one-electron reduction processes at −0.560 (ΔE_p = 105) and −1.330 V (ΔE_p = 100 mV). The reduction processes are chemically reversible on the cyclic voltammetry time-scale but the scan-rate dependence of the first reduction wave showed that the electron transfer exhibited some kinetic control. The separation of 770 mV between these two processes may be compared with values of 850 and 390 mV for $[\{Mo(NO)L(Cl)\}_2\{1,4\text{-}(NH)_2C_6H_4\}]$ and $[\{Mo(NO)L(Cl)\}_2\{1,3\text{-}(NH)_2C_6H_4\}]$ respectively.[8,9] This result was unexpected in that the interaction between $\{Mo(NO)L(Cl)\}$ moieties linked by an aryl bridge would normally be expected to be larger for the 1,2-disubstituted aryl derivative than for the 1,4-disubstituted compound. However, this expectation presupposes that the aryl(NH–Mo)$_2$ moiety is essentially planar, allowing efficient mesomeric contact to be established between the metal centres through the sp^2 amide nitrogen and the aryl π system. Although we have been unable to obtain crystals of $[\{Mo(NO)L(X)\}_2\{1,4\text{-}$

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* Contribution from the Crystallography Unit, The Universities of Aston and Birmingham.

Table 2 Selected bond distances (Å) and angles (°) in [{Mo(NO)L(I)}$_2$-{1,2-(NH)$_2$C$_6$H$_4$}] with e.s.d.s in parentheses

Mo(1)–I(1)	2.750(1)	N(1)–O(1)	1.142(8)
Mo(1)–N(1)	1.786(7)	N(8)–C(16)	1.384(9)
Mo(1)–N(2)	2.258(6)	C(16)–C(17)	1.395(11)
Mo(1)–N(4)	2.187(7)	C(16)–C(16B)	1.438(15)
Mo(1)–N(6)	2.227(6)	C(17)–C(18)	1.378(14)
Mo(1)–N(8)	1.957(6)	C(18)–C(18B)	1.375(27)
I(1)–Mo(1)–N(1)	90.6(2)	I(1)–Mo(1)–N(8)	99.5(2)
I(1)–Mo(1)–N(2)	90.1(2)	N(1)–Mo(1)–N(8)	96.1(3)
N(1)–Mo(1)–N(2)	179.1(3)	N(2)–Mo(1)–N(8)	83.2(2)
N(2)–Mo(1)–N(4)	83.5(2)	N(4)–Mo(1)–N(8)	91.4(3)
I(1)–Mo(1)–N(4)	166.6(2)	N(6)–Mo(1)–N(8)	167.1(2)
N(1)–Mo(1)–N(4)	96.0(3)	Mo(1)–N(1)–O(1)	179.1(7)
I(1)–Mo(1)–N(6)	88.1(2)	Mo(1)–N(8)–C(16)	138.5(5)
N(1)–Mo(1)–N(6)	94.1(3)	N(8)–C(16)–C(17)	120.6(7)
N(2)–Mo(1)–N(6)	86.5(2)	C(16B)–C(16)–C(17)	119.0(10)
N(4)–Mo(1)–N(6)	79.8(2)	C(16B)–C(16)–N(8)	120.4(9)

(NH)$_2$C$_6$H$_4$}] (X = I or Cl) suitable for structural studies, the crystal structure of [Mo(NO)L(I)(NHC$_6$H$_4$Me-4)] has been obtained.[19] This reveals that the Mo–N–C(aryl) moiety in this monometallic complex is almost coplanar with the aryl ring, the Mo atom lying only 0.012 Å from the plane of the aryl ring. Thus a near-planar arrangement for the Mo–NH–aryl–NH–Mo core in [{Mo(NO)L(Cl)}$_2${1,4-(NH)$_2$C$_6$H$_4$}] is possible. However, the structure of [{Mo(NO)L(I)}$_2${1,2-(NH)$_2$C$_6$H$_4$}] (see below) shows that steric interactions result in the Mo atoms being substantially removed from the aryl ring plane, reducing opportunities for mesomeric interaction to occur through the aryl π system. Assuming that the structure of [{Mo(NO)L(Cl)}$_2$-{1,2-(NH)$_2$C$_6$H$_4$}] is not substantially different from that of its iodide analogue, this finding would account for the observation that the electrochemically detected five-bond Mo–Mo interaction in [{Mo(NO)L(Cl)}$_2${1,2-(NH)$_2$C$_6$H$_4$}] is smaller than that of the seven-bond interaction in [{Mo(NO)L(Cl)}$_2${1,4-(NH)$_2$C$_6$H$_4$}].[8] However it is larger than the six-bond interaction in [{Mo(NO)L(Cl)}$_2${1,3-(NH)$_2$C$_6$H$_4$}] where no mesomeric interaction is possible.[9]

Structural Studies.—Atomic coordinates are listed in Table 1 and geometric parameters in Table 2. The numbering scheme used is illustrated in Fig. 1. The complex possesses C_2 symmetry, a crystallographic two-fold axis passing through the midpoint of the C(16)–C(16B) and C(18)–C(18B) bonds. The coordination geometry about molybdenum is essentially octahedral, with maximum angular deviation from ideal octahedral 13.4°, I(1)–Mo–N(4) 166.6(2)°, mean deviation 5.6°. These deviations, however, show a consistent pattern when compared with analogous structures based on the Mo(NO){HB(3,5-Me$_2$C$_3$HN$_2$)$_3$} moiety. Thus in [Mo(NO)L(I)(NC$_4$H$_8$)][7] corresponding angles at the metal differ by a maximum of only 5.6°, with a mean difference of 2.2°, and comparison with the dimeric complex[20] [{Mo(NO)L(Cl)}$_2$(NC$_4$H$_8$N)] gives a maximum difference of 5.7° and a mean difference of 2.3°. Some general features emerge. For example the N–Mo–N angles between the nitrogen atoms of the tris(3,5-dimethylpyrazolyl)borato ligand are typically in the range 80–88°, whilst the *cis* angles between the nitrosyl nitrogen atom and these nitrogen atoms are greater than 90° (range 94–101°). With respect to the *trans* angles, that involving the nitrosyl atom is closer to 180° than are the other two *trans* angles, which are generally in the range 160–170°.

The bonds to the pyrazolyl rings (2.187–2.258 Å) are similar in length to those found previously[2,3,7,20,21] in this type of complex. The bond *trans* to the nitrosyl group is usually long, and is, in the present structure, the longest of the three Mo–N(pyrazolyl) bonds. This has been attributed[2] to the *trans* influence of the strongly π-accepting nitrosyl ligand. The short Mo–N(nitrosyl) length, 1.786(7) Å, is also in good agreement with previous results and is consistent with a strong bonding interaction. The Mo–N(pyrazolyl) bond *trans* to the iodo substituent is the shortest of these, reflecting the lack of significant π interaction between the halogen and the metal. The Mo–N(8) distance of 1.957(6) Å falls well within the range of 1.935–1.990 Å measured[2,3,7,20,21] for Mo–N(amido) bonds in a number of complexes of this type. These distances indicate a significant degree of p_π–d_π donation from the ligand to the metal. Consistent with this is the large 138.5(5)° angle at the amido nitrogen atom, although steric effects may also be operative here, the near-planarity of the nitrosyl–Mo–N(amido)–C(phenyl) fragment [torsion angle N(1)–Mo–N(8)–C(16) 1.1(8)°], and also the relatively long *trans* Mo–N(pyrazolyl) bond at 2.227(6) Å. The phenyl ring is, however, twisted out of this plane by 29°, and the molybdenum and amido nitrogen atoms deviate respectively by ±0.905(5) and ±0.15(1) Å from the phenyl ring plane. The Mo ··· Mo separation is 6.455(2) Å. The aromatic C(16)–C(16B) bond of the central N(8)–C(16)–C(16B)–N(8B) moiety is long [1.438(15) Å], and the N–C(aromatic) bonds are short [1.384(9) Å], consistent with some electron delocalisation across the aromatic ring. More extensive electron delocalisation is, presumably, prevented by the non-planarity of the Mo–N–C–C–N–Mo moiety. Planarity of this fragment appears to be prevented by steric factors. Calculations show that if the Mo–N–C–C–N–Mo residue is constrained to be planar, the two rather rigid MoL moieties of the dimeric molecule would be rotated in such a way that the pyrazolyl rings *trans* to the nitrosyl groups (see Fig. 1) would be brought into impossibly close contact, with the distance between atom C(3) and atom C(3B) across the two-fold symmetry axis only 2.46 Å. In the actual structure all atom to atom distances between these two pyrazolyl rings are greater than 3.6 Å. A search of the Cambridge Structural Database (version of January 1992) revealed no previous example of a crystal structure containing the C$_6$H$_4$(NH–Mo)$_2$-1,2 residue with which the structure described here could be compared.

The pyrazolyl rings are planar to within the limits of experimental error. The methyl groups and the molybdenum and boron atoms deviate from their respective planes by up to 0.10, 0.14 and 0.19 Å respectively. The interplanar angles, 125.6, 127.2 and 107.1°, show the pattern usually,[20,21] but not invariably,[3] found, in which the smallest angle occurs between the two rings encompassing the relatively small nitrosyl group.

The results described here provide a demonstration that, in bimetallic complexes, steric effects can have a pronounced effect on metal–metal interactions if they are able to distort the molecular structure in a manner which reduces the efficiency of mesomeric contact between the metal centres.

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