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Structures of Two Isomers of Dichlorobis(2-phenylazopyridine)ruthenium(II), $[RuCl_2(C_{11}H_9N_3)_2]$

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Abstract. $M_r = 538$. C_2 isomer: $P2_1/c$, $a = 8{\cdot}421\ (8)$, $b = 22{\cdot}88\ (2)$, $c = 12{\cdot}99\ (2)$ Å, $\beta = 116{\cdot}3\ (1)°$, $V = 2243{\cdot}2$ Å^3, $Z = 4$, $F(000) = 1080$, $\lambda(\text{Mo } K\alpha) = 0{\cdot}7107$ Å, $\mu = 9{\cdot}4\ \text{cm}^{-1}$, $D_x = 1{\cdot}59\ \text{g cm}^{-3}$, $R = 0{\cdot}051$ for 3257 observed $[I > 2\sigma(I)]$ reflections. C_1 isomer: $P\bar{1}$, $a = 13{\cdot}371\ (4)$, $b = 15{\cdot}345\ (3)$, $c = 13{\cdot}101\ (4)$ Å, $\alpha = 114{\cdot}75\ (2)$, $\beta = 93{\cdot}75\ (3)$, $\gamma = 67{\cdot}61\ (3)°$, $V = 2242{\cdot}7$ Å^3, $Z = 4$, $F(000) = 1080$, $\lambda(\text{Mo } K\alpha) = 0{\cdot}7107$ Å, $\mu = 9{\cdot}4\ \text{cm}^{-1}$, $D_x = 1{\cdot}59\ \text{g cm}^{-3}$, $R = 0{\cdot}054$ for 3660 observed reflections. With reference to the pairs of Cl, N(py) and N(azo) coordinating with Ru, the C_2 isomer has a *cis–trans–cis* configuration and the C_1 isomer a *cis–cis–cis*. In both structures Ru—N(azo) distances are relatively shorter than Ru—N(py), indicating stronger bonding in the former case, caused by the considerable π-backbonding power of the azo function.

Introduction. The title compound is known to exist in three isomeric forms (Krause & Krause, 1980; Goswami, Chakravarty & Chakravorty, 1981, 1982, 1983), one of which is green and the other two are dark blue. In principle, five isomers are possible. If the coordinating pairs of Cl, N(py) and N(azo) are considered in that order, the configurations of these five are (Fig. 1) *trans–trans–trans* (*ttt*), *trans–cis–cis* (*tcc*), *cis–trans–cis* (*ctc*), *cis–cis–trans* (*cct*) and *cis–cis–cis* (*ccc*). Spectroscopic evidence suggested that the green isomer has the *ttt* configuration. Either the *ctc* or *cct* configuration has been indicated in the case of one blue isomer (herein referred to as the C_2 isomer, owing to the nominal twofold symmetry of the complex), while the other (the C_1 isomer) appears to be of the *ccc* variety (Goswami *et al.*, 1981, 1982, 1983).

This investigation was undertaken with two primary ends in view: (1) to establish unequivocally the structural identities of the two blue isomers; and (2) to ascertain the relative π-backbonding abilities of N(py) and N(azo) towards Ru^{II}.

Experimental. Specimens $0{\cdot}25 \times 0{\cdot}2 \times 0{\cdot}15$ mm (C_2 isomer) and $0{\cdot}25 \times 0{\cdot}2 \times 0{\cdot}1$ mm (C_1 isomer). CAD-4 diffractometer, graphite-monochromatized Mo $K\alpha$ radiation. 25 reflections (to $\theta = 15{\cdot}2°$) for measurement of lattice parameters in each case. D_m not measured since all suitable liquids available were solutions of bromides and iodides with which the crystals reacted. ω–2θ scan, $\theta_{\max} = 25°$ for C_2 isomer [25° for C_1 isomer], h −9 to 8 [−15 to 15], k 0 to 27 [−18 to 16], l 0 to 15 [0 to 15]. 3924 [7980] unique reflections measured (no merging of equivalents), 667 [832] unobserved with $I \le 2\sigma(I)$. Max. value of decay

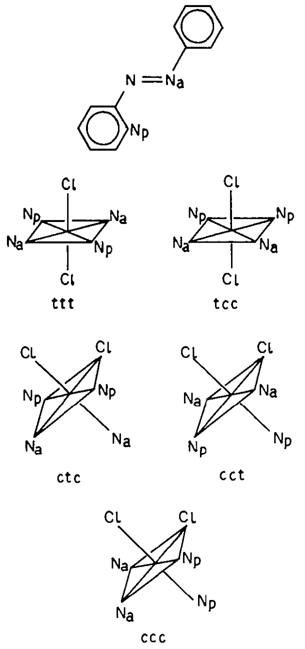

Fig. 1. The ligand and the five geometrically possible isomeric configurations of its dichloro complex with Ru.

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Table 1. *Refined parameters for non-hydrogen atoms*

$B_{eq} = \frac{4}{3}\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$

(*a*) The C_2 isomer

	x	y	z	B_{eq}(Å^2)
Ru	0·16269 (6)	0·17901 (2)	0·08439 (4)	2·03
Cl(1)	0·2575 (2)	0·26628 (7)	0·0251 (1)	3·16
Cl(2)	0·0012 (2)	0·23771 (8)	0·1586 (1)	3·32
N(1)	0·0488 (6)	0·1100 (2)	0·1150 (4)	3·38
N(2)	−0·1082 (6)	0·0944 (2)	0·0452 (4)	3·02
N(3)	−0·0711 (6)	0·1709 (2)	−0·0613 (4)	2·20
N(4)	0·3211 (5)	0·1311 (2)	0·0418 (4)	2·53
N(5)	0·4831 (6)	0·1218 (2)	0·1134 (4)	3·02
N(6)	0·3972 (6)	0·1786 (2)	0·2316 (4)	2·39
C(1)	0·161 (1)	0·1005 (3)	0·3217 (6)	4·14
C(2)	0·232 (1)	0·0671 (4)	0·4211 (6)	5·06
C(3)	0·272 (1)	0·0095 (4)	0·4167 (7)	5·60
C(4)	0·237 (1)	−0·0159 (4)	0·3132 (8)	5·74
C(5)	0·165 (1)	0·0164 (3)	0·2120 (7)	4·35
C(6)	0·1275 (7)	0·0749 (2)	0·2189 (5)	2·94
C(7)	−0·1770 (7)	0·1292 (2)	−0·0527 (5)	2·72
C(8)	−0·3491 (9)	0·1196 (3)	−0·1353 (6)	3·75
C(9)	−0·4131 (8)	0·1546 (3)	−0·2330 (6)	3·62
C(10)	−0·3046 (8)	0·1967 (3)	−0·2438 (5)	3·26
C(11)	−0·1336 (8)	0·2042 (2)	−0·1559 (5)	2·86
C(12)	0·3010 (8)	0·0425 (2)	−0·0674 (5)	3·27
C(13)	0·261 (1)	0·0151 (3)	−0·1718 (6)	4·13
C(14)	0·192 (1)	0·0475 (3)	−0·2717 (6)	4·33
C(15)	0·1625 (9)	0·1065 (3)	−0·2695 (6)	3·83
C(16)	0·2007 (8)	0·1343 (2)	−0·1657 (5)	3·31
C(17)	0·2722 (7)	0·1015 (2)	−0·0656 (5)	2·47
C(18)	0·5275 (7)	0·1485 (3)	0·2187 (5)	2·90
C(19)	0·6984 (9)	0·1440 (3)	0·3061 (7)	4·36
C(20)	0·737 (1)	0·1713 (3)	0·4106 (6)	4·11
C(21)	0·604 (1)	0·2013 (3)	0·4230 (6)	4·10
C(22)	0·4380 (8)	0·2041 (3)	0·3328 (5)	3·33

(*b*) The C_1 isomer

	x	y	z	B_{eq} or B(Å^2)
Ru(1)	0·09672 (9)	0·19522 (8)	0·11667 (9)	3·6
Ru(2)	0·38521 (9)	0·66474 (8)	0·34636 (9)	4·1
Cl(1)	0·1283 (3)	0·2691 (3)	0·3121 (3)	4·3
Cl(2)	−0·0652 (3)	0·1818 (3)	0·1652 (3)	5·4
Cl(3)	0·4963 (3)	0·7319 (3)	0·2931 (3)	5·4
Cl(4)	0·2654 (3)	0·6909 (3)	0·2088 (3)	4·9
N(1)	0·2396 (8)	0·1791 (7)	0·0649 (8)	2·7 (1)
N(2)	0·3275 (8)	0·0995 (8)	0·0554 (8)	3·4 (2)
N(3)	0·2011 (8)	0·0563 (7)	0·1172 (8)	3·3 (2)
N(4)	0·0048 (8)	0·3246 (8)	0·0982 (8)	3·3 (2)
N(5)	−0·0201 (9)	0·3177 (9)	−0·0007 (9)	4·4 (2)
N(6)	0·0707 (8)	0·1416 (8)	−0·0480 (8)	3·3 (2)
N(7)	0·4986 (8)	0·6195 (8)	0·4367 (8)	3·4 (2)
N(8)	0·5905 (9)	0·5386 (8)	0·3890 (9)	3·9 (2)
N(9)	0·4935 (8)	0·5218 (7)	0·2310 (8)	3·2 (2)
N(10)	0·2826 (8)	0·7999 (8)	0·4581 (9)	3·8 (2)
N(11)	0·1924 (9)	0·8076 (9)	0·4994 (9)	4·3 (2)
N(12)	0·2758 (8)	0·6243 (7)	0·3965 (8)	3·1 (2)
C(1)	0·225 (1)	0·349 (1)	0·093 (1)	4·6 (2)
C(2)	0·243 (1)	0·414 (1)	0·049 (1)	5·9 (3)
C(3)	0·290 (1)	0·370 (1)	−0·059 (1)	6·1 (3)
C(4)	0·319 (1)	0·266 (1)	−0·128 (1)	6·3 (3)
C(5)	0·304 (1)	0·200 (1)	−0·089 (1)	4·0 (2)
C(6)	0·2582 (9)	0·2453 (9)	0·022 (1)	3·2 (2)
C(7)	0·305 (1)	0·0349 (9)	0·091 (1)	3·9 (2)
C(8)	0·393 (1)	−0·050 (1)	0·095 (1)	5·0 (3)
C(9)	0·365 (1)	−0·110 (1)	0·133 (1)	6·6 (3)
C(10)	0·264 (1)	−0·093 (1)	0·157 (1)	6·2 (3)
C(11)	0·178 (1)	−0·008 (1)	0·149 (1)	5·3 (3)
C(12)	−0·082 (1)	0·446 (1)	0·285 (1)	4·1 (2)
C(13)	−0·131 (1)	0·550 (1)	0·370 (1)	5·9 (3)
C(14)	−0·133 (1)	0·630 (1)	0·346 (1)	6·5 (3)
C(15)	−0·090 (1)	0·614 (1)	0·249 (1)	6·8 (3)
C(16)	−0·042 (1)	0·512 (1)	0·161 (1)	5·9 (3)
C(17)	−0·040 (1)	0·430 (1)	0·185 (1)	3·8 (2)
C(18)	0·019 (1)	0·215 (1)	−0·083 (1)	4·6 (2)
C(19)	0·001 (1)	0·195 (1)	−0·195 (1)	5·9 (3)
C(20)	0·042 (1)	0·091 (1)	−0·274 (1)	6·5 (3)
C(21)	0·094 (1)	0·014 (1)	−0·240 (1)	5·4 (3)
C(22)	0·108 (1)	0·039 (1)	−0·126 (1)	4·6 (2)
C(23)	0·413 (1)	0·704 (1)	0·629 (1)	4·4 (2)
C(24)	0·412 (1)	0·768 (1)	0·745 (1)	6·3 (3)
C(25)	0·499 (1)	0·798 (1)	0·780 (1)	6·9 (3)
C(26)	0·585 (1)	0·768 (1)	0·704 (1)	6·3 (3)
C(27)	0·586 (1)	0·708 (1)	0·590 (1)	4·9 (3)
C(28)	0·501 (1)	0·6774 (9)	0·555 (1)	3·7 (2)
C(29)	0·586 (1)	0·4858 (9)	0·272 (1)	3·6 (2)
C(30)	0·678 (1)	0·400 (1)	0·208 (1)	4·7 (2)
C(31)	0·672 (1)	0·350 (1)	0·094 (1)	4·9 (2)
C(32)	0·577 (1)	0·384 (1)	0·048 (1)	4·7 (3)
C(33)	0·486 (1)	0·472 (1)	0·120 (1)	4·0 (2)
C(34)	0·368 (1)	0·922 (1)	0·551 (1)	6·5 (3)
C(35)	0·379 (1)	1·018 (1)	0·576 (1)	7·3 (4)
C(36)	0·312 (1)	1·080 (1)	0·531 (1)	7·8 (4)
C(37)	0·233 (1)	1·059 (1)	0·472 (1)	8·4 (4)
C(38)	0·215 (1)	0·967 (1)	0·447 (1)	6·6 (3)
C(39)	0·288 (1)	0·901 (1)	0·487 (1)	4·2 (3)
C(40)	0·189 (1)	0·710 (1)	0·467 (1)	3·8 (2)
C(41)	0·101 (1)	0·701 (1)	0·508 (1)	4·8 (3)
C(42)	0·102 (1)	0·603 (1)	0·474 (1)	5·0 (3)
C(43)	0·188 (1)	0·516 (1)	0·399 (1)	4·5 (2)
C(44)	0·273 (1)	0·529 (1)	0·363 (1)	4·0 (2)

Table 2. *Distances* (Å) *and angles* (°) *in the co-ordination octahedra*

(*a*) The C_2 isomer

Ru–Cl(1)	2·401 (1)	Ru–Cl(2)	2·397 (1)
Ru–N(1)	1·977 (4)	Ru–N(3)	2·045 (4)
Ru–N(4)	1·984 (4)	Ru–N(6)	2·051 (4)
Cl(1)–Ru–Cl(2)	89·52 (6)	Cl(1)–Ru–N(6)	89·0 (1)
Cl(1)–Ru–N(3)	95·6 (1)	Cl(1)–Ru–N(4)	90·5 (1)
Cl(2)–Ru–N(1)	87·3 (1)	Cl(2)–Ru–N(3)	86·4 (1)
Cl(2)–Ru–N(6)	96·3 (1)	N(1)–Ru–N(3)	76·1 (2)
N(1)–Ru–N(4)	93·5 (2)	N(1)–Ru–N(6)	99·2 (2)
N(3)–Ru–N(4)	100·5 (2)	N(4)–Ru–N(6)	76·6 (2)

(*b*) The C_1 isomer

Ru(1)–Cl(1)	2·410 (3)	Ru(1)–Cl(2)	2·401 (4)
Ru(1)–N(1)	1·960 (9)	Ru(1)–N(3)	2·059 (9)
Ru(1)–N(4)	2·003 (9)	Ru(1)–N(6)	2·020 (9)
Ru(2)–Cl(3)	2·379 (4)	Ru(2)–Cl(4)	2·389 (4)
Ru(2)–N(7)	1·958 (9)	Ru(2)–N(9)	2·069 (9)
Ru(2)–N(10)	1·96 (1)	Ru(2)–N(12)	2·028 (9)
Cl(1)–Ru(1)–Cl(2)	92·2 (1)	Cl(1)–Ru(1)–N(1)	91·6 (3)
Cl(1)–Ru(1)–N(3)	84·2 (2)	Cl(1)–Ru(1)–N(4)	100·7 (3)
Cl(2)–Ru(1)–N(3)	94·8 (2)	Cl(2)–Ru(1)–N(4)	89·6 (3)
Cl(2)–Ru(1)–N(6)	89·9 (3)	N(1)–Ru(1)–N(3)	76·2 (4)
N(1)–Ru(1)–N(4)	98·8 (4)	N(1)–Ru(1)–N(6)	86·6 (4)
N(3)–Ru(1)–N(6)	98·6 (4)	N(4)–Ru(1)–N(6)	76·1 (4)
Cl(3)–Ru(2)–Cl(4)	91·1 (2)	Cl(3)–Ru(2)–N(7)	88·1 (3)
Cl(3)–Ru(2)–N(9)	84·2 (3)	Cl(3)–Ru(2)–N(10)	96·3 (3)
Cl(4)–Ru(2)–N(9)	93·5 (3)	Cl(4)–Ru(2)–N(10)	87·2 (3)
Cl(4)–Ru(2)–N(12)	85·4 (3)	N(7)–Ru(2)–N(9)	76·1 (4)
N(7)–Ru(2)–N(10)	103·0 (4)	N(7)–Ru(2)–N(12)	96·2 (4)
N(9)–Ru(2)–N(12)	101·9 (4)	N(10)–Ru(2)–N(12)	77·5 (4)

(*c*) The azo bond distances in the ligands

C_2 isomer		C_1 isomer	
N(1)–N(2)	1·279 (7)	N(1)–N(2)	1·30 (1)
N(4)–N(5)	1·283 (6)	N(4)–N(5)	1·29 (1)
		N(7)–N(8)	1·31 (1)
		N(10)–N(11)	1·30 (2)

correction based on variation of intensity of standards 1·076 [1·053]. No absorption correction. Structures solved by Patterson and successive Fourier syntheses. Least-squares refinement (on F). Atomic scattering factors and f' and f'' for Ru from *International Tables for X-ray Crystallography* (1974). Block-diagonal approximation after the introduction of anisotropic thermal parameters. Towards the end of refinement, the weighting scheme was modified so as to reflect the trends in $|\Delta F|$, by adopting the following values of $\sigma(F)$ (Seal & Ray, 1981): C_2 isomer: for $|F_o| \leq 9{\cdot}49$, $\sigma(F) = 0{\cdot}225F_o$; for $9{\cdot}49 < |F_o| \leq 12{\cdot}98$, $\sigma(F) = 0{\cdot}133F_o$; for $12{\cdot}98 < |F_o| \leq 16{\cdot}55$, $\sigma(F) = 0{\cdot}10F_o$; for $16{\cdot}55 < |F_o| \leq 20{\cdot}81$, $\sigma(F) = 0{\cdot}089F_o$; for $20{\cdot}81 < |F_o| \leq 25{\cdot}08$, $\sigma(F) = 0{\cdot}089F_o$; for $25{\cdot}08 <$

$|F_o| \le 30{\cdot}65$, $\sigma(F) = 0{\cdot}073F_o$; for $30{\cdot}65 < |F_o| \le 37{\cdot}10$, $\sigma(F) = 0{\cdot}06F_o$; for $37{\cdot}10 < |F_o| \le 45{\cdot}20$, $\sigma(F) = 0{\cdot}057F_o$; for $45{\cdot}20 < |F_o| \le 62{\cdot}701$, $\sigma(F) = 0{\cdot}044F_o$; for $|F_o| > 62{\cdot}701$, $\sigma(F) = 0{\cdot}037F_o$; C_1 isomer: for $|F_o| \le 16{\cdot}34$, $\sigma(F) = 0{\cdot}21F_o$; for $16{\cdot}34 < |F_o| \le 21{\cdot}32$, $\sigma(F) = 0{\cdot}15F_o$; for $21{\cdot}32 < |F_o| \le 26{\cdot}80$, $\sigma(F) = 0{\cdot}11F_o$; for $26{\cdot}80 < |F_o| \le 39{\cdot}55$, $\sigma(F) = 0{\cdot}10F_o$; for $39{\cdot}55 < |F_o| \le 48{\cdot}02$, $\sigma(F) = 0{\cdot}08F_o$; for $48{\cdot}02 < |F_o| \le 57{\cdot}98$, $\sigma(F) = 0{\cdot}07F_o$; for $57{\cdot}98 < |F_o| \le 77{\cdot}31$, $\sigma(F) = 0{\cdot}06F_o$; for $|F_o| > 77{\cdot}31$, $\sigma(F) = 0{\cdot}04F_o$. In both the cases, H atoms were kept fixed at their calculated positions. In the case of the C_2 isomer, anisotropic thermal parameters of all non-hydrogen atoms were refined along with the positional parameters, using 3257 observed reflections. Refinement converged $[(\Delta/\sigma)_{\max} = 0{\cdot}534]$ at $R = 0{\cdot}051$, $wR = 0{\cdot}070$ with $S = 1{\cdot}265$. Final difference map showed max. and min. peaks of 1·5 and $-0{\cdot}8$ e Å^{-3} respectively. In the case of the C_1 isomer, anisotropic thermal parameters only of Ru(1), Ru(2), Cl(1) and Cl(2), and isotropic thermal parameters of the C and N atoms were refined along with the positional parameters. Only 3660 observed reflections, extending to $\theta = 20°$, were used and refinement was stopped when $R = 0{\cdot}054$ and $wR = 0{\cdot}074$, with $S = 0{\cdot}965$. Max. and min. peaks in difference map 0·8 and $-1{\cdot}7$ e Å^{-3} respectively. All calculations carried out on a Burroughs 6700 computer. The *X-RAY ARC* program system, compiled by B. L. Vickery, D. Bright and P. R. Mallinson, was used with several local modifications. Programs *EXFFT* and *SEARCH* of the *MULTAN*78 system (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) for weighted Fourier syntheses.

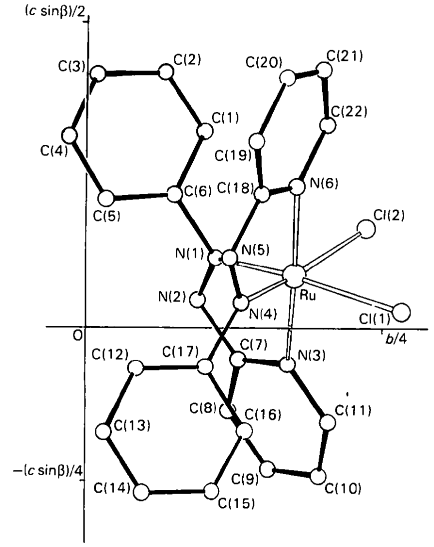

Fig. 2. Asymmetric unit of the structure of the C_2 isomer projected down the *a* axis.

Discussion. The refined positional parameters are presented in Table 1 and a few selected distances and angles in Table 2.* Projections of the asymmetric units of the two structures are shown in Figs. 2 and 3. The C_2 isomer is found to have the *ctc* configuration. The asymmetric unit of the structure of the C_1 isomer consists of two independent *ccc* complexes differing in orientation and some other minor aspects.

In the coordination octahedra, normal Ru—Cl distances are observed and the angles Cl—Ru—Cl are very near to 90°. However, N(py)—Ru—N(azo), the bite angle of the ligand, is found to be ~76° in both structures, thus introducing a considerable distortion in the octahedra.

A surprising feature of both structures is the relative shortness of the Ru—N(azo) distance as compared to Ru—N(py). Normally the pyridine N would be expected to be a better donor than the azo N. Evidently the

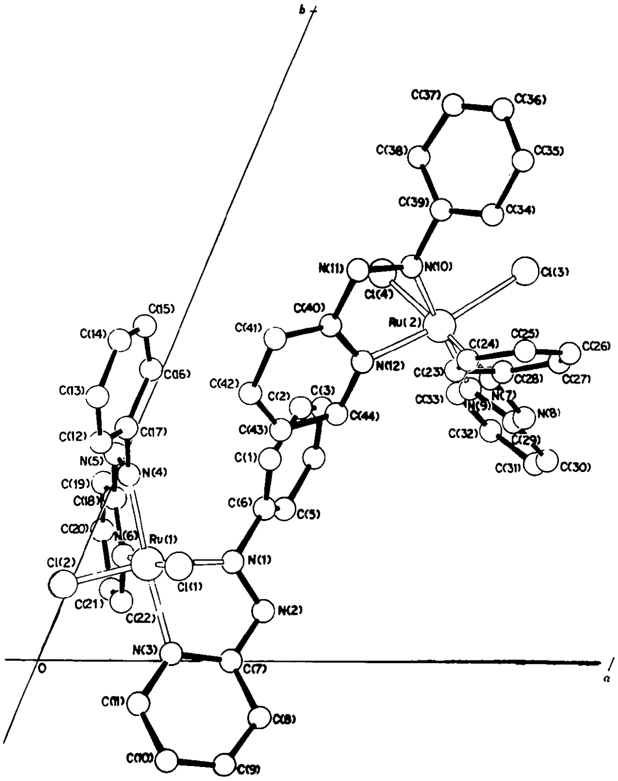

Fig. 3. Asymmetric unit of the structure of the C_1 isomer projected on the *ab* plane.

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* Tables of observed and calculated structure factors, anisotropic thermal parameters, calculated coordinates of the H atoms, bond distances and angles in the ligands not presented in Table 2 and equations of some mean planes have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39218 (63 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

π-backbonding $[d\pi(Ru) \rightarrow \pi^*(azo)]$ power of the azo function is very considerable, making the net $(\sigma + \pi)$ bond order larger in Ru—N(azo) compared to Ru—N(py). The relatively longer N—N distance [1·28–1·31 Å, compared, for example, to 1·257 (2) and 1·254 (2) Å in the structure of 1-phenylazoacetaldehyde oxime (Roy & Sengupta, 1980)] is in full accord with the π-backbonding hypothesis. Certain chemical, electrochemical and spectroscopic data also indirectly suggested the presence of this kind of bonding (Krause & Krause, 1982; Goswami, Chakravarty & Chakravorty, 1983).

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Bis(*tert*-butylthio)bis(η^5-cyclopentadienyl)molybdenum(IV), $[Mo(C_5H_5)_2(C_4H_9S)_2]$

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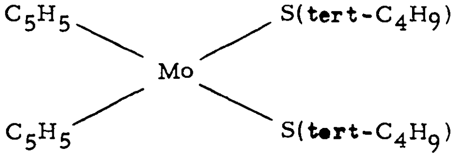
Abstract. $M_r = 404{\cdot}48$, orthorhombic, $P2_12_12_1$, $a = 9{\cdot}3424\ (8)$, $b = 13{\cdot}461\ (1)$, $c = 14{\cdot}884\ (1)$ Å, $V = 1871{\cdot}78$ Å^3, $Z = 4$, $D_x = 1{\cdot}43$ g cm^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}5418$ Å, $\mu = 77{\cdot}89$ cm^{-1}, $F(000) = 840$, $T = 298$ K. Final $R(F) = 0{\cdot}035$ for 1870 observed reflections. The Mo coordination is a distorted tetrahedron formed by the two S atoms of the *tert*-butylthio ligands and the ring normals to the cyclopentadienyl rings. These adopt an eclipsed orientation, giving the molecule an approximate C_{2v} symmetry. The Mo atom is at an average distance from the cyclopentadienyl rings of 2·006 (7) Å and the angle between the ring normals is 130·4 (3)°. The Mo—S bond lengths are 2·501 (1) and 2·491 (1) Å and the S—Mo—S bond angle is 71·1 (1)°.

Introduction. The structure analysis of (I) is a continuation of the investigation of complexes of the type $[M(\eta^5\text{-}C_5H_5)_2LL']$. The structure of the $[Ti(\eta^5\text{-}C_5H_5)_2(SCH_3)_2]$ complex was previously reported by Carrondo & Jeffrey (1983).



(I)

Experimental. $[Mo(\eta^5\text{-}C_5H_5)_2\{S(tert\text{-}C_4H_9)\}_2]$ prepared by Clementina Teixeira, Centro de Química Estrutural (Dias, Martinho Simões & Teixeira, 1981). D_m estimated by flotation but not measured. Laue symmetry and systematic absences consistent with $P2_12_12_1$, crystal $0{\cdot}28 \times 0{\cdot}13 \times 0{\cdot}067$ mm used to collect 1948 *hkl* intensities (range of *hkl*: h 0→11, k 0→16, l 0→17), 75 with $F_o < 3\sigma(F_o)$, θ–2θ scans, $\theta_{max} = 66°$, CAD-4 diffractometer, graphite-monochromated Cu $K\alpha$ radiation. Unit-cell parameters refined from 37 reflections, $40 < \theta < 48°$. $30\bar{3}$, 204, 040 used as standards, intensities showed no decay throughout data collection. Mo-atom position from