THE CRYSTAL STRUCTURE OF SOME DICARBONYL-ALLYL DERIVATIVES OF SUBSTITUTED CARBONYLS OF MOLYBDENUM I. THE CRYSTAL STRUCTURE OF (ISOTHIOCYANATO)DICARBONYL-2,2'-BIPYRIDINE- π -ALLYLMOLYBDENUM NCS(CO)₂(C₁₀H₈N₂)(π -C₃H₅)Mo

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SUMMARY

The crystal structure and molecular configuration of the complex (isothiocyanato)dicarbonyl-2,2'-bipyridine- π -allylmolybdenum, NCS(CO)₂(C₁₀H₈N₂)-(π -C₃H₅)Mo, have been determined from three-dimensional data collected photographically at 20°. The unit cell is monoclinic, spacegroup P2₁/c, with four formula units in a cell of dimensions $a=8.290\pm0.003$ Å, $b=10.160\pm0.001$ Å, $c=21.631\pm0.007$ Å, $\beta=113^{\circ}$ 58' ±2'. The structure has been refined to R=0.086 by the application of an isotropic/anisotropic block-diagonal-approximation least-squares refinement using 2606 independent non-zero reflections.

The complex has essentially an octahedral arrangement of ligands. If it is viewed such that the 2,2'-bipyridine and dicarbonyl ligands lie in equatorial positions, the allyl and isothiocyanato ligands lie in the axial positions, one on each side of the equatorial plane. The equatorial ligands are each planar and dip away from the plane of the allylic carbon atoms.

The inter-planar angles between these ligands are: 2,2'-bipyridine/dicarbonyl 15.1°; π -allyl/dicarbonyl 8.3°; 2,2'-bipyridine/ π -allyl 22.7°. The central allylic carbon atom is nearer the molybdenum, at 2.196±0.009 Å, than the two terminal atoms at 2.291±0.011 Å and 2.351±0.012 Å. The allylic inter-carbon angle is 115.7 ±1.1° and the mean C-C bond length is 1.440±0.023 Å. The mean Mo-C (carbonyl) and C-O (carbonyl) bond lengths are 1.941±0.007 Å and 1.178±0.014 Å respectively. The thiocyanato ligand is nitrogen-coordinated at a distance of 2.117±0.007 Å with bond lengths N-C=1.163±0.010 Å and C-S=1.643±0.008 Å.

The aims of the investigation of the MCBAS structure were threefold, namely:

- (i) To investigate the orientation of the allyl ligand and to show that it was a π -allyl, and not a σ -allyl, ligand.
- (ii) To determine whether the thiocyanato ligand was sulphur- or nitrogen-coordinated to the molybdenum.
- (iii) To investigate the stereochemical position of the thiocyanato ligand in relation to the other ligands.

In their infra-red work, Hull and Stiddard⁸ observed a strong spectral band at 2080 cm⁻¹ which they assigned to v(C-N) and a weak band at 825 cm⁻¹, assigned to v(C-S). On the basis of the latter mode they inferred that the thiocyanato ligand was N-bonded to the molybdenum.

INTRODUCTION

Many π -allyl complexes of transition metals have been made⁸ and investigated by chemical and spectroscopic^{1,2,3,8} methods. However, very little confirmation of these structures by X-ray methods exists. The allylpalladium chloride dimer (π -C₃H₅PdCl)₂ has been investigated by several workers at both normal^{4,5} and low⁶ temperatures. The structure of the (1,3-dimethylallyl)palladium chloride dimer has also been described⁷.

The group of complexes from which the subject of this and subsequent papers have been chosen are those described and prepared by Hull and Stiddard⁸. The complexes chosen are numbers 4, 9 and 16 in Table 1 of their paper, namely:

Complex 4: (Isothiocyanato)dicarbonyl-2,2'-bipyridine- π -allylmolybdenum Complex 9: Dicarbonyl-2,2'-bipyridinepyridine- π -allylmolybdenum

methallylmolybdenum

Complex 4, henceforth MCBAS, is the subject of this paper and the other two complexes are under investigation. Complex 11 of their paper⁸ is also under consideration but the recrystallisation of this and other halogen derivatives is proving somewhat difficult.

CRYSTAL DATA

The red prismatic crystals, prepared⁸ and kindly donated by C. Hull of University College, London, were generally rectangular with well formed faces parallel to the *C*-axis of the holohedral class of the monoclinic system. However, the crystal chosen for this investigation also had four prominent pyramidal faces. The crystal was grown by the slow evaporation of a solution in dimethylformamide, HCON(CH₃)₂, and had dimensions $0.28 \times 0.21 \times 0.26$ mm³.

From the systematically absent reflections on equi-inclination Weissenberg photographs, *i.e.*

$${h0l} - h+l = 2n+1$$

 ${0k0} - k = 2n+1$

the spacegroup was uniquely defined as $P2_1/n$. By a change of indices this is the conventional spacegroup $P2_1/c$. It was however found to be convenient to collect the Weissenberg data as per $P2_1/n$ because the b_n^* axis on the upper level photographs remained almost linear indicating that $\beta_n^* \simeq 90^\circ$ (suffices *n* and *c* will henceforth refer to the spacegroups $P2_1/n$ and $P2_1/c$ respectively).

Only one crystal was used in this analysis and the following zones were collected:

- (i) $(hkN)_n$ Weissenberg zones for N=0, 1, 2, ..., 7; main data
- (ii) $(hNl)_c$ Precession zones for N = 0, 1, 2; correlation data
- (iii) $(0kl)_c$ and $(h0l)_c$ Weissenberg zones for accurate cell parameters' determination.

All Weissenberg data were collected using Ni-filtered CuKa radiation,

tetrafluoroborate Complex 16: (Isothiocyanato)dicarbonyl-1,10-phenanthroline-2-

 $\lambda_{a} = 1.54178$ Å at 30 kV, 20 mA at a temperature of $20 \pm 2^{\circ}$ in packs of five films. The precession data were collected on a Buerger Precession camera using Zr-filtered MoK α radiation, $\lambda_{\vec{a}} = 0.71069$ Å at 45 kV, 15 mA in the same temperature range. All data were measured visually by comparison with a calibrated wedge made from the chosen crystal.

The accurate cell parameters were obtained from the (0kl), and (h0l), Weissenberg zones using fine gold wire, 0.25 mm diameter and 99.998% purity, as a calibrating standard, Powell⁹ and Graham¹⁰. The gold diffraction pattern, using the Weissenberg technique, was superimposed onto the above zones and the calculated parameters were plotted against

$$f(\theta) = \frac{1}{2} \left(\frac{\cos^2 \theta}{\theta} + \frac{\cos^2 \theta}{\sin \theta} \right)$$

Nelson and Riley¹¹. β was obtained from measurements on the h, 0, 2h and $h, 0, \overline{2h}$ type reflections. The data used in this determination and the resulting accurate cell parameters obtained are as follows:

Cell constant for gold $a = 4.0785$	04 Å at 25°, Weyerer ¹²
Copper radiation wavelengths ¹⁵	$\lambda_{a_1} = 1.54051 \text{\AA}$
	$\lambda_{a_{\lambda}} = 1.54433 \text{ Å}$
	$\lambda_{\pi}^{-2} = 1.54178 \text{ Å}$
	$\lambda_{B} = 1.39217 \text{ Å}$
Coefficient of refraction ¹⁵	$\hat{\beta} = 1.86 \times 10^{-4} \text{ Å}$
Coefficient of expansion ¹⁵	$\alpha = 14.1 \times 10^{-6} ^{\circ}\mathrm{C}^{-1}$
Accurate cell parameters ^{9,10} at	$20^{\circ} a = 8.290 \pm 0.003 \text{ Å}$
	$b = 10.160 \pm 0.001 \text{ Å}$
	$c = 21.631 \pm 0.007 \text{ Å}$
	$\beta = 113^{\circ}58' \pm 2'$
Density	$D_{\rm obs} = 1.634 \pm 0.029 {\rm g} \cdot {\rm cm}^{-3}$
	$D_{\rm calc} = 1.623 \pm 0.007 {\rm g} \cdot {\rm cm}^{-3}$
7-1	

Linear absorption coefficients $\mu_{Mo} = 9 \text{ cm}^{-1}; \quad \mu_{Cu} = 80 \text{ cm}^{-1}$

The density of the crystals was determined by the method of flotation in a mixture of bromoform and benzene at 22° .

As MoKa radiation at 45 kV gave a large number of Laue streaks on the Weissenberg zones it was decided to use CuKa radiation for the collection of data in these zones even though no absorption corrections were to be made. Attempts were made to obtain spherical or cylindrical crystals but they proved to be too soft.

The usual Lorentz, polarisation and spot-distortion¹³ corrections were applied to the raw data and the initial layer scale factors were computed from the reflections occurring on more than one layer, using the method of Hamilton, Rollett and Sparks¹⁴. However, individual layer scale factors were refined in the least-squares routine.

THE STRUCTURE DETERMINATION

Using the $P2_1/n$ data, a three-dimensional Patterson synthesis yielded the molybdenum position. These parameters were refined using a block-diagonalapproximation least-squares method in which $\Sigma(\omega \cdot \Delta^2) = \Sigma[\omega \cdot (K \cdot F_o - F_c)^2]$ was .

TABLE I

(a) the final positional parameters and standard deviations $\times \ 10^4$

	x	У	Z
Мо	0.1300(0)	0.3193(0)	0.3661 (0)
N10	0.2209 (9)	0.1223(7)	0.3780(3)
C10	0.2798(10)	0.0166(7)	0.3871 (4)
S	0.3741(4)	-0.1290(2)	0.4018(2)
C1	0.1721(16)	0.3298(10)	0.2861(6)
01	0.1980(14)	0.3327 (9)	0.2346(5)
C2	0.3745(12)	0.3820(9)	0.4017(5)
O2	0.5171(12)	0.4193(10)	0.4203 (5)
C3	-0.0382(15)	0.4834(10)	0.2974(6)
C4	0.0183(14)	0.4989 (9)	0.3653(5)
C5	0.1600(19)	0.5300(12)	0.4145(7)
N11	0.0846(9)	0.2591 (6)	0.4581 (3)
C11	0.2059 (14)	0.2779 (10)	0.5216(5)
C21	0.1854(15)	0.2309(12)	0.5797(6)
C31	0.0257(15)	0.1659(9)	0.5670(6)
C41	- 0.0993(15)	0.1512(10)	0.5021(6)
C51	- 0.0617(14)	0.1959(8)	0.4489(5)
N12	- 0.1270(9)	0.2212(6)	0.3297(3)
C12	- 0.1866(13)	0.1762(8)	0.3770(5)
C22	- 0.3475(14)	0.1187(12)	0.3601 (6)
C32	- 0.4515(17)	0.0825(14)	0.2915(7)
C42	- 0.3962(16)	0.1305(13)	0.2445(6)
C52	- 0.2448(14)	0.1960(10)	0.2638(6)

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$T = \exp - (h^2 \cdot B_{11})$	$+k^{2} \cdot B_{22} + l^{2}$	$B_{33}+k\cdot l\cdot l$	$B_{23} + h \cdot l \cdot E_{23}$	$B_{13} + h \cdot k \cdot B$	12)
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	B ₁₁	B ₂₂	B ₃₃	B ₂₃	B ₁₃	B ₁₂
Мо	0.01097	0.00325	0.00223	0.00078	0.00447	- 0.00070
N10	0.00921	0.00718	0.00180	0.00059	0.00384	0.00350
C10	0.00823	0.00478	0.00182	-0.00068	0.00435	- 0.00187
S	0.01931	0.00586	0.00414	0.00213	0.01006	0.00570
CI	0.01847	0.01039	0.00263	- 0.00046	0.00776	- 0.00240
01	0.03142	0.01603	0.00382	0.00092	0.01028	- 0.00488
C2	0.00868	0.00788	0.00341	0.00044	0.00396	- 0.00681
O2	0.02223	0.01847	0.00388	- 0.00093	0.00833	0.00608
C3	0.02065	0.00754	0.00377	0.00008	0.00711	0.00178
C4	0.02095	0.00620	0.00304	0.00095	0.00626	0.00554
C5	0.02968	0.00981	0.00363	- 0.00208	0.00815	0.00053
N11	0.01088	0.00542	0.00193	0.00065	0.00347	0.00271
C11	0.01728	0.00947	0.00279	0.00004	0.00661	0.00101
C21	0.02010	0.01092	0.00284	- 0.00129	0.00636	- 0.00070
C31	0.01961	0.00822	0.00265	0.00069	0.00644	- 0.00131
C41	0.01811	0.00909	0.00304	- 0.00085	0.00861	0.00062
C51	0.01524	0.00679	0.00269	0.00041	0.00671	0.00216
N12	0.01084	0.00482	0.00202	-0.00046	0.00282	0.00312
C12	0.01106	0.00867	0.00231	- 0.00071	0.00427	- 0.00149
C22	0.01475	0.01062	0.00364	- 0.00055	0.00461	0.00383
C32	0.02088	0.01430	0.00440	0.00076	0.00874	- 0.00061
C42	0.01809	0.01232	0.00372	0.00029	0.00660	0.00216
C52	0.01525	0.00899	0.00288	0.00004	0.00437	- 0.00118

minimised. The weighting factor, ω , was given by $\omega = [1 + (K \cdot F_o - b)^2 a^{-2}]^{-1}$ where the final values of *a* and *b* were 1100 and 500 respectively on a scale fifty times absolute. All the atomic scattering factors were taken from International Tables¹⁶. Conversion to $P2_1/c$ was effected at this point.

The first three-dimensional electron density distribution located the two carbonyl ligands, the allylic carbon atoms, the thiocyanato ligand and one half of the 2,2'-bipyridine. After further refinement a second Fourier synthesis located the remaining atoms and a Difference Fourier synthesis indicated anisotropic thermal vibrations of both the molybdenum and sulphur atoms.

Further refinement, including layer scale-factors, gave a residual, defined by $R = \Sigma |K \cdot F_o - F_c| \cdot \Sigma |K \cdot F_o| = 0.090$ and mean $\Sigma (\omega \cdot \Delta^2) = 5.9$ on an absolute scale. An

TABLE 2

THE R.M.S. THERMAL DISPLACEMENTS, \overline{U} , and the direction cosines l, m, n with respect to the reciprocal axes a^* , b^* and c^*

	Ū	1	m	n		Ū	I	m	n
Mo	0.2318	0.0635	0.6678	0.7036	C11	0.1396	0.0509	-0.6389	0.7221
	0.1124	-0.6590	-0.5308	0.2201		0.2747	0.0565	0.7699	0.6041
	0.1434	0.7490	-0.5236	0.6756		0.2219	-0.9972	0.0109	-0.3364
	0.2922	0.7256	0.4652	0.7592	C21	0.1529	0.1444	-0.5711	0.7971
N10	0.1104	0.0975	-0.7121	0.6757		0.2856	-0.2139	0.7800	0.4510
	0.2221	0.5523	0.6210	0.7333		0.2388	0.9662	0.2578	0.4012
	0.1840	0.8282	0.3304	0.0778	C31	0.2796	-0.0037	0.8239	0.5172
C10	0.2157	-0.2711	0.6846	0.5086		0.1374	-0.1955	-0.5575	0.6579
	0.0877	-0.0265	-0.7093	0.6339		0.2099	0.9807	-0.1080	0.5474
	0.1537	0.9623	0.1733	0.5830	C41	0.1047	0.1203	-0.6286	0.7510
S	0.3241	0.2139	0.6796	0.7290		0.2904	-0.0568	0.7690	0.5591
	0.1336	-0.0269	-0.7152	0.6281		0.2190	-0.9914	-0.1205	-0.3508
	0.1649	-0.9772	0.1618	-0.2692	C51	0.2682	0.1261	0.7570	0.6373
C1	0.1116	-0.0149	-0.5835	0.7360		0.1204	0.0447	-0.6508	0.7108
	0.2838	-0.1981	0.7986	0.4396		0.1872	-0.9913	0.0669	-0.2969
	0.2292	0.9806	0.1526	0.5148	N12	0.2181	0.0911	0.6697	0.7107
01	0.1581	-0.1603	0.5384	0.6909		0.1351	0.6524	-0.6002	0.6888
	0.3483	-0.1423	0.8429	0.4178		0.1752	-0.7572	-0.4392	0.1431
	0.2908	0.9769 ·	0.0345	0.5902	C12	0.1349	0.0120	-0.7642	0.5944
C2	0.1211	-0.4517	-0.8475	0.0735		0.2377	-0.4239	0.5812	0.4632
	0.2694	-0.0880	0.3561	0.8144		0.2091	0.9056	0.2821	0.6573
	0.2185	0.8881	0.3958	0.5757	C22	0.2858	-0.2389	0.4863	0.6710
O 2	0.1690	-0.0698	-0.6850	0.6347		0.1816	-0.1853	-0.8732	0.3379
	0.3325	-0.6848	0.5623	0.1462		0.2358	0.9523	-0.0479	0.6601
C3	0.3036	0.0495	0.7029	0.6691	C32	0.1750	0.0757	-0.7481	0.5721
	0.1778	0.2475	-0.6981	0.7149		0.3220	0.0860	0.6547	0.7213
	0.2013	-0.9676	-0.1432	-0.2024		0.2745	-0.9937	0.1140	-0.3901
C4	0.2915	0.2212	0.7736	0.6330	C42	0.1762	0.0642	-0.7649	0.6121
	0.1651	0.5765	-0.5958	0.7458		0.2964	0.1820	0.6414	0.7551
	0.1798	-0.7855	-0.2204	0.2095		0.2536	-0.9814	0.0689	-0.2340
C5	0.3316	-0.0948	0.8300	0.4649	C52	0.2610	-0.0541	0.6586	0.6640
	0.1729	0.4422	0.4608	0.8827		0.1769	0.1256	-0.7509	0.5417
	0.2370	-0.8918	0.3168	-0.0670		0.2171	0.9909	-0.0592	0.5152
N11	0.2238	0.2852	0.7001	0.7143					
	0.1362	0.1012	-0.7025	0.6858					
	0.1633	-0.9535	0.1348	-0.1397					

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analysis of the mean  $\Sigma(\omega \cdot \Delta^2)$  for the different  $|K \cdot F_o|$  ranges indicated no modification of *a* and *b* in the weighting factor.

A final Difference Fourier synthesis was computed to investigate any further atoms which might require anisotropic thermal vibrations. Small, but not insignificant, positive and negative regions around all atomic sites indicated the anisotropic thermal vibration of the remaining atoms. After the anisotropic refinement of these parameters the residual finally became 0.086 and the mean  $\Sigma(\omega \cdot \Delta^2) = 4.9$ . A final Difference Fourier synthesis showed no residual peaks in excess of  $\pm 0.5 \text{ e} \cdot \text{Å}^{-3}$  except in the neighbourhood of the molybdenum atom where peaks of  $-2 \text{ e} \cdot \text{Å}^{-3}$  were noted. The peaks were oriented along the crystal axes thus indicating probable absorption effects for which no correction had been made. It was also noted that the negative regions extended along the bonds towards the nearest atoms to the molybdenum. This may be indicative of an electron density distribution around the molybdenum and neighbouring atoms which is more complex than the postulated ellipsoids.

The conclusion of the refinement was judged to have been reached when the parameter shifts were less than the corresponding standard deviations in magnitude and when  $\Sigma(\omega \cdot \Delta^2)$  would reduce no further. The final positional parameters, standard deviations and thermal parameters are given in Table 1. The root-mean-square thermal displacements are listed in Table 2 and the observed and calculated structure factors, on an absolute scale, are given in Table 3.

Figure 1 shows four molecules of the complex projected onto the 010 electron density distribution.

### THE MOLECULAR STRUCTURE

If the allyl ligand is considered to occupy one coordination position about the molybdenum atom and the 2,2'-bipyridine ligand two then the complex may be considered to have a distorted octahedral arrangement of ligands. The main distortion is of the angles at the molybdenum formed (*i*) by the 2,2'-bipyridine nitrogens and (*ii*) by the carbonyl atoms. These angles have been found to be significantly different from 90°, being N11-Mo-N12=73.0 $\pm$ 0.3° and C1-Mo-C2=78.3 $\pm$ 0.5° respectively. The angles about the thiocyanate ligand, however, do approach 90°, being closer for the carbonyl carbons than the 2,2'-bipyridine nitrogens; 79.3 $\pm$ 0.3° and 81.9 $\pm$ 0.3° for the latter and 88.6 $\pm$ 0.4° and 89.9 $\pm$ 0.3° for the former. Fig. 2 shows an idealised structure for this complex giving the atomic designation.

The main feature of the MCBAS molecular configuration is the planarity of the 2,2'-bipyridine and dicarbonyl ligands. With respect to the orthogonal axes X, Y, Z given by

 $X = a \cdot x + c \cdot z \cdot \cos \beta$   $Y = b \cdot y$  $Z = c \cdot z \cdot \sin \beta$ 

the equations of the respective least-squares best fit planes are:

(i) bipy: 0.4882X - 0.8727Y - 0.0037Z + 3.9042 = 0

(*ii*) dicarb: -0.2854X + 0.9473Y - 0.1453Z - 2.6537 = 0

The mean deviations of the component atoms from their respective planes are (i)



Fig. 2. An idealised structure for the MCBAS complex, and the atomic designation.

#### TABLE 4

THE DISTANCES OF ATOMS FROM THE LEAST-SQUARES PLANES

Planes defined by:  $l \cdot X + m \cdot Y + n \cdot Z + d = 0$ .

(I) allylic carbon atoms; (II) dicarbonyl atoms; (III) 2,2'-bipyridine ligand; (IV) pyridine (1); (V) pyridine (2).

Plane	1	m	n	d
(I) (II) (III) (IV)	-0.1454 -0.2854 0.4882 0.4871	0.9762 0.9473 -0.8727 -0.8717	-0.1609 -0.1453 -0.0037 -0.0527	-4.2747 -2.6537 3.9042 4.3918
(V)	0.4885	0.8705	-0.0603	-3.5118

PERPENDICULAR DISTANCES, IN Å, OF ATOMS FROM PLANES

Plane	Atom	distance	Atom	distance
(I)	Мо	-1.961		
(II)	Мо	-0.021		
	C1	0.009	<b>O</b> 1	-0.006
	C2	-0.009	O2	0.006
(III)	Mo	-0.002		
	N11	0.050	C11	-0.002
	C21	0.077	C31	0.063
	C41	-0.029	C51	-0.042
	N12	-0.010	C12	0.058
	C22	-0.126	C32	0.074
	C42	0.077	C52	0.025
(IV)	Mo	0.140		
• •	N11	0.000	C11	-0.014
	C21	0.009	C31	0.008
	C41	0.021	C51	0.018
(V)	Mo	-0.078		
• •	N12	-0.018	C12	-0.029
	C22	0.062	C32	- 0.050
	C42	0.005	C52	0.031

 $\pm 0.053$  Å and (*ii*)  $\pm 0.007$  Å and the interplanar angle is about 15.1°. The distances of the individual atoms from the planes are given in Table 4, together with the equations of each of the two pyridine planes. The mean bond lengths within the 2,2'-bipyridine ligand at C-C(ring)=1.385\pm0.016 Å, C-N=1.356±0.013 Å and C-C(bridging)= 1.491±0.014 Å, agree well with the respective values given by Schomaker and

Pauling¹⁶ for pyridine, pyrazine and related six-member heterocyclic molecules; Merritt and Schroeder¹⁷ for 2,2'-bipyridine; Barclay, Hoskins and Kennard¹⁸ for iodobis(2,2'-bipyridine)copper(II) iodide; and the bridging bond between the two benzene rings in biphenyl, Pauling¹⁹. The bond angles approach the 120° for an ideal hexagonal ring but because of the variation in bond lengths the angles lay in the range 115° to 127°. The Mo–N bond lengths are  $2.256 \pm 0.007$  Å and  $2.189 \pm 0.007$  Å but the difference may not be significant in view of the respective thermal vibrations, Busing and Levy²⁰.

The mean Mo–C(carbonyl) and C–O bond lengths in the carbonyl ligand are  $1.943 \pm 0.010$  Å and  $1.172 \pm 0.015$  Å respectively. The latter is in good agreement with the C–O bond lengths as given for carbonyl complexes by other workers, namely:

1.16 $\pm$ 0.05 Å in Cr(CO)₆, Brockway, Ewens and Lister²¹ 1.15 $\pm$ 0.03 Å in Ni(CO)₄, Ladell, Post and Fankuchen²² 1.16 $\pm$ 0.01 Å in Mn₂(CO)₁₀, Dahl and Rundle²³ 1.137 $\pm$ 0.004 Å in Cr(CO)₆, Whittaker and Jeffery²⁴

#### TABLE 5

INTERATOMIC DISTANCES AND BOND ANGLES

Bond lengths ( standard devia	Å) and ations ( $\times 10^3$ )	Bond angles and standard deviations	s ( × 10)
Mo-C1	1.904(11)	C1-Mo-C2	78.3(5)
Mo-C2	1.963 (8)	N11-Mo-N12	73.0(3)
Mo-C3	2.291 (11)	C3-Mo-C4	36.8(4)
Mo-C4	2.197(9)	C3-Mo-C5	63.4(5)
Mo-C5	2.351 (12)	C4-Mo-C5	37.3(4)
Mo-N10	2.119(7)	C1-Mo-N10	88.6(4)
Mo-N11	2.256(7)	C2-Mo-N10	89.9(3)
Mo-N12	2.189(7)	N11-Mo-N10	79.3(3)
		N12-Mo-N10	81.9(3)
N11-C11	1.347(12)	Mo-N11-C51	118.3(6)
C11-C21	1419(16)	Mo-N12-C12	118.2(5)
C21-C31	1.403(16)	N11C11C21	123.1(10)
C31C41	1.373(16)	C11C21C31	115.4(10)
C41-C51	1.387(15)	C21-C31-C41	120.8 (10)
C51-N11	1.313(12)	C31C41C51	118.8(10)
N12-C12	1.382(13)	C41-C51-N11	122.7 (9)
C12-C22	1.363(14)	C41-C51-C12	121.8(9)
C22-C32	1.432(18)	C22-C12-C51	121.7(10)
C32-C42	1.362(19)	N11-C51-C12	115.4(9)
C42C52	1.329(16)	N12-C12-C51	115.1(8)
C52-N12	1.387(13)	N12-C12-C22	123.2(9)
C51-C12	1.491 (14)	C12-C22-C32	120.0(11)
		C22-C32-C42	115.9(12)
		C32-C42-C52	120.4(12)
		C42-C52-N12	126.6(11)
C1-01	1.216(16)	Mo-C1-O1	177.6(7)
C2O2	1.145(12)	Mo-C2-O2	176.9 (8)
N10-C10	1.163(10)	Mo-N10-C10	175.8(5)
C10-S	1.644(8)	N10-C10-S	176.4(7)

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The carbon and oxygen atoms are almost co-linear with the molybdenum, the mean angle being  $177.3 \pm 0.7^{\circ}$ .

The isothiocyanato ligand is also almost linear, the Mo–N–C and N–C–S angles being  $175.8\pm0.5^{\circ}$  and  $176.4\pm0.7^{\circ}$  respectively. The ligand is co-ordinated to the molybdenum via the nitrogen at a distance of  $2.119\pm0.007$  Å. The N–C and C–S bond lengths at  $1.163\pm0.010$  Å and  $1.644\pm0.007$  Å agree well with values obtained by Ferrari *et al.*²⁵, Nardelli *et al.*²⁶ and Brown and Lingafelter²⁷ in isothiocyanates and with Brown and Lingafelter²⁸ in a thiocyanate.

The plane of the allylic carbon atoms is given by -0.1454X + 0.9672Y-0.1609Z - 4.2747 = 0. This plane thus makes an angle of 8.3° with the dicarbonyl plane and -22.7° with the 2,2'-bipyridine plane. The mean C-C bond length is  $1.440 \pm 0.017$  Å with an inter-carbon angle of  $115.7 \pm 1.1°$ . Both of these values are in agreement with the corresponding values given for the allylpalladium chloride dimer by Smith⁶, Levdik and Porai-Koshits⁵, and Oberhansli and Dahl⁴. As observed by these workers the central carbon atom is nearer the heavy atom, in this case molybdenum, at  $2.197 \pm 0.009$  Å, than the two terminal carbons at  $2.291 \pm 0.011$  Å and  $2.351 \pm 0.012$  Å respectively. A full list of bond lengths and angles is given in Table 5.

#### TABLE 6

INTERMOLECULAR DISTANCES; CLOSEST APPROACHES FOR THE OUTER ATOMS

Atoms	Distance (Å)	Atoms	Distance (Å)
O1-C21(f)	3.371	C4-C21(c)	3.494
O1-C31(i)	3.313	C5-C31 (c)	3.546
O1-C42(g)	3,385	C41-C41 (d)	3.504
O2-C5(d) S-C11(e) C3-C10(g)	3.525 3.531 3.683	C22–C10(a)	3.526

The coordinates of those atoms marked a-g are related to those in Table 1 by: a, (x - 1, y, z); b, (-x, -y, 1-z); c, (-x, 1-y, 1-z); d, (1-x, 1-y, 1-z); e, (1-x, y, 1-z); f,  $(x, \frac{1}{2}+y, \frac{1}{2}+z)$ ; g,  $(-x, \frac{1}{2}-y, \frac{1}{2}+z)$ .

Differences in equivalent bond lengths and angles are not claimed to be significant in view of the lack of knowledge concerning the effects of thermal vibrations, Busing and Levy²⁰. Also, the two carbonyl ligands have differing environments as do the two pyridine halves of the 2,2'-bipyridine ligand. These are indicated by the shortest intermolecular distances listed in Table 6 and by reference to Fig. 1.

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