Tableau 2. *Distances* (Å) *et angles dans l'ion thioéthanoate* CH_3COS^-

Les écarts types sont entre parenthèses.

C(1)–S	1,703 (16)	O——C(1)–S	124°37 (50)′
C(1)–O	1,231 (19)	C(2)–C(1)–S	118°48 (49)′
C(1)–C(2)	1,528 (25)	C(2)–C(1)–O	116°58 (34)′

L'ion thioéthanoate est plan, les distances obtenues dans KCH_3COS sont voisines de celles données par Gordy (1946) pour l'acide thioéthanoïque [C–S = 1,78(2), C–O = 1,24(4) Å].

Le potassium est entouré par trois atomes d'oxygène et quatre atomes de soufre. Le polyèdre de coordination est un prisme trigonal dont une face rectangulaire est remplacée par une pyramide qui a pour sommet un atome d'oxygène. Un environnement à sept atomes a été également trouvé par Philippot & Lindqvist (1970) dans la structure de $K_2CS_3.H_2O$ avec un atome d'oxygène à 2,74 Å et une valeur moyenne de 3,289 Å pour les six distances K–S.

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μ-Dinitrogen-bis{(π-mesitylene)[1,2-bis(dimethylphosphino)ethane]molybdenum}

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Abstract. $C_{30}H_{56}Mo_2N_2P_4$, $M = 760{\cdot}6$, orthorhombic, *Pbca* (D_{2h}^{15}, No. 61), $a = 13{\cdot}309$ (7), $b = 16{\cdot}582$ (9), $c = 16{\cdot}438$ (9) Å, $Z = 4$, $D_{calc} = 1{\cdot}39$ g cm^{-3}. Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å. The molecules lie on centres of symmetry and consist of a dinitrogen bridge between two molybdenum-mesitylene-phosphine groupings.

Introduction. Single crystals of octahedral habit were obtained by the method of Silverthorn & Green (1974) and mounted under dry nitrogen in Lindemann glass capillary tubes. A crystal of dimensions approximately $0{\cdot}3 \times 0{\cdot}3 \times 0{\cdot}3$ mm was selected for intensity measurements, and was set up about $[10\bar{1}]$ on a Hilger and Watts PDP8-controlled four-circle diffractometer. Accurate cell dimensions and orientation matrix were obtained by a least-squares fit to the setting angles of 20 reflexions.

The intensities of two equivalents of each independent reflexion with $\sin\theta/\lambda < 0{\cdot}7$ were measured with an $\omega/2\theta$ scan and the ordinate analysis method (Watson, Shotton, Cox & Muirhead, 1970). Mo $K\alpha$ radiation was used with a graphite monochromator. Reflexions with intensity less than 4σ, where σ is the standard deviation derived from simple counting statistics, or whose apparent centre was more than 0·2° from the predicted position were not included in subsequent calculations. Lorentz and polarization corrections, and an empirical absorption correction (North, Phillips & Mathews, 1968), were applied, leading to a final set of 3291 independent structure amplitudes.

The structure (Fig. 1) was solved by Patterson and Fourier techniques; hydrogen atoms were located from difference syntheses. Refinement was by the least-squares method, with a large block approximation to the normal matrix: a 2×2 block was calculated from the derivatives of the scale and dummy overall temperature factor, two blocks from those of the positional parameters of the mesitylene ligand and the remainder of the molecule respectively, and two blocks from those of the corresponding temperature factors. In the final

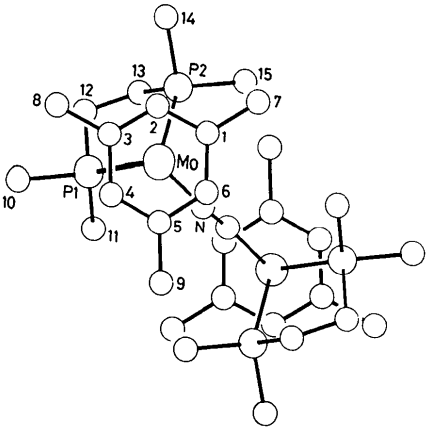

Fig. 1. The molecule in projection onto the best plane of the mesitylene ring. There is a centre of symmetry midway between the two nitrogen atoms.

cycles non-hydrogen atoms were permitted anisotropic temperature factors, whilst hydrogen atoms were refined isotropically and, in addition, the C–H lengths were constrained (Waser, 1963; Rollett, 1969) to their mean with a standard deviation of 0·03 Å. The final value of this mean was 0·98 Å. Each reflexion was assigned a weight according to the expression $W^{-1} = 1 + \{(F-32)/15\}^2$ and the final R was 0·031. All calculations were performed on the Oxford University ICL 1906A computer with the Oxford package of crystallographic programs (Carruthers & Rollett, 1973). Complex neutral-atom scattering factors were taken from *International Tables for X-ray Crystallography* (1962).

Final atomic parameters are given in Tables 1 and 2; Table 3 shows bond lengths and angles involving non-hydrogen atoms, with estimated standard deviations calculated from the full variance-covariance matrix.*

Table 1. *Fractional coordinates and isotropic temperature factors for hydrogen atoms*

To determine the serial number of the carbon atom to which any particular hydrogen atom is bonded, delete the least significant digit of the hydrogen-atom serial number. Thus H(21) is bonded to C(2) and H(103) is bonded to C(10).

	x	y	z	U(iso)
H(21)	0·204 (3)	0·199 (2)	0·196 (2)	0·06 (1)
H(41)	0·317 (3)	0·143 (2)	−0·030 (2)	0·06 (1)
H(61)	0·027 (2)	0·212 (2)	−0·013 (2)	0·04 (1)
H(71)	0·016 (4)	0·299 (2)	0·166 (3)	0·08 (2)
H(72)	−0·058 (2)	0·222 (3)	0·151 (4)	0·10 (2)
H(73)	0·009 (5)	0·220 (3)	0·206 (2)	0·12 (2)
H(81)	0·358 (5)	0·171 (5)	0·190 (2)	0·16 (3)
H(82)	0·407 (4)	0·215 (2)	0·145 (4)	0·11 (2)
H(83)	0·408 (5)	0·111 (3)	0·116 (5)	0·15 (3)
H(91)	0·188 (4)	0·231 (2)	−0·159 (3)	0·10 (2)
H(92)	0·209 (3)	0·145 (3)	−0·148 (3)	0·10 (2)
H(93)	0·101 (3)	0·176 (4)	−0·158 (3)	0·12 (2)
H(101)	0·410 (3)	−0·091 (2)	0·034 (3)	0·06 (1)
H(102)	0·393 (3)	−0·008 (2)	−0·011 (2)	0·06 (1)
H(103)	0·408 (3)	−0·008 (2)	0·076 (2)	0·05 (1)
H(111)	0·216 (4)	−0·089 (3)	−0·073 (2)	0·07 (2)
H(112)	0·238 (2)	−0·158 (2)	−0·016 (2)	0·03 (1)
H(113)	0·131 (2)	−0·122 (3)	−0·013 (3)	0·06 (1)
H(121)	0·254 (3)	−0·152 (2)	0·136 (3)	0·06 (1)
H(122)	0·274 (3)	−0·068 (2)	0·188 (2)	0·08 (2)
H(131)	0·123 (3)	−0·120 (2)	0·237 (2)	0·05 (1)
H(132)	0·084 (3)	−0·121 (2)	0·144 (2)	0·06 (1)
H(141)	0·106 (3)	0·102 (2)	0·301 (3)	0·07 (1)
H(142)	0·204 (2)	0·046 (3)	0·290 (3)	0·10 (2)
H(143)	0·112 (4)	0·015 (3)	0·335 (2)	0·11 (2)
H(151)	−0·079 (3)	−0·014 (3)	0·151 (2)	0·08 (2)
H(152)	−0·055 (3)	−0·035 (2)	0·246 (2)	0·04 (1)
H(153)	−0·070 (3)	0·062 (2)	0·205 (3)	0·07 (1)

Discussion. The coordination around the molybdenum atom is of the type which Churchill & O'Brien (1969) likened to that of a three-legged piano stool, and is typical of other cases where the metal is bonded to a cyclic π-ligand on one side and to three monodentate ligands, or their equivalent, on the other, *e.g.* $(\pi\text{-}C_7H_7)Mo(CO)_3^+$ (Clark & Palenik, 1973), $(\pi\text{-}C_7H_7)Mo(CO)_2(\sigma\text{-}C_6F_5)$ (Churchill & O'Brien, 1969).

The Mo–N length of 2·04 Å is close to the value (2·01 Å) observed in *trans*-$Mo(N_2)_2(Ph_2PCH_2CH_2PPh_2)_2$ (Uchida, Uchida, Hidai & Kodama, 1971), and the N–N bond (1·14 Å) is similar to those in two other bridged dinitrogen complexes: $[\mu\text{-}N_2\text{-}\{Ru(NH_3)_5\}_2](BF_4)_4$ (1·12 Å; Treitel, Flood, Marsh & Gray, 1969) and $\mu\text{-}N_2\text{-}[\{(C_6H_{11})_3P\}_2Ni]_2$ (1·12 Å; Jolly, Jonas, Kruger & Tsay, 1971). Both these complexes also contain essentially linear M–N–N–M skeletons.

The molybdenum–carbon contacts range from 2·24 to 2·31 Å, similar to those found in π-cyclopentadienyl

* A table of observed structure amplitudes and calculated structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30549 (31 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 2. *Fractional coordinates and anisotropic temperature factors*

The $U(ij)$'s $\times 10^4$ for Mo and P and $\times 10^3$ for C and N.

	x	y	z	$U(11)$	$U(22)$	$U(33)$	$U(23)$	$U(13)$	$U(12)$
Mo	0·14459 (2)	0·08474 (1)	0·06520 (1)	385 (1)	275 (1)	389 (1)	−2 (1)	−43 (1)	−48 (1)
P(1)	0·24104 (5)	−0·03772 (4)	0·05427 (5)	398 (4)	388 (3)	507 (4)	12 (3)	12 (3)	22 (3)
P(2)	0·09036 (6)	0·01529 (5)	0·18516 (5)	535 (4)	484 (4)	399 (4)	−4 (3)	42 (3)	−57 (3)
C(1)	0·0944 (3)	0·2123 (2)	0·0999 (2)	80 (3)	29 (1)	89 (3)	−13 (2)	15 (2)	−6 (2)
C(2)	0·1894 (4)	0·1942 (2)	0·1368 (2)	115 (3)	39 (2)	54 (2)	−4 (1)	−11 (2)	−27 (2)
C(3)	0·2742 (3)	0·1725 (2)	0·0903 (2)	63 (2)	41 (2)	89 (3)	14 (2)	−27 (2)	−22 (2)
C(4)	0·2618 (3)	0·1644 (2)	0·0050 (2)	59 (2)	38 (1)	79 (2)	7 (2)	6 (2)	−13 (1)
C(5)	0·1705 (3)	0·1821 (2)	−0·0330 (2)	80 (2)	37 (1)	55 (2)	9 (1)	−10 (2)	−17 (2)
C(6)	0·0880 (3)	0·2050 (2)	0·0160 (2)	60 (2)	33 (1)	86 (2)	7 (2)	−15 (2)	−2 (1)
C(7)	0·0078 (5)	0·2435 (3)	0·1500 (5)	138 (5)	59 (3)	186 (7)	−43 (3)	83 (4)	−9 (3)
C(8)	0·3766 (4)	0·1632 (3)	0·1291 (5)	94 (3)	78 (3)	184 (6)	36 (3)	−76 (4)	−42 (3)
C(9)	0·1619 (5)	0·1843 (3)	−0·1248 (3)	172 (5)	72 (3)	57 (2)	21 (2)	−15 (3)	−35 (3)
C(10)	0·3772 (3)	−0·0400 (3)	0·0362 (3)	44 (2)	84 (3)	103 (3)	−3 (3)	6 (2)	5 (2)
C(11)	0·2011 (3)	−0·1119 (2)	−0·0211 (3)	72 (3)	46 (2)	78 (3)	−19 (2)	−6 (2)	11 (2)
C(12)	0·2342 (3)	−0·0976 (2)	0·1489 (2)	77 (2)	54 (2)	68 (2)	16 (2)	−2 (2)	14 (2)
C(13)	0·1305 (3)	−0·0917 (2)	0·1834 (3)	87 (3)	53 (2)	69 (2)	22 (2)	15 (2)	0 (2)
C(14)	0·1311 (4)	0·0465 (4)	0·2861 (2)	108 (4)	109 (4)	43 (2)	−5 (2)	−7 (2)	−21 (3)
C(15)	−0·0451 (3)	0·0036 (3)	0·2031 (3)	60 (2)	91 (3)	72 (3)	−4 (2)	21 (2)	−14 (2)
N	0·0321 (2)	0·0194 (1)	0·0124 (1)	40 (1)	30 (1)	41 (1)	0 (1)	−4 (1)	0 (1)

Table 3. *Bond lengths* (Å) *and angles* (°)

Mo—P(1)	2·409 (3)	Mo—C(3)	2·295 (4)
Mo—P(2)	2·395 (3)	Mo—C(4)	2·271 (4)
Mo—N	2·042 (4)	Mo—C(5)	2·309 (4)
Mo—C(1)	2·290 (4)	Mo—C(6)	2·280 (4)
Mo—C(2)	2·243 (4)	N—N^{I}	1·145 (7)
P(1)—Mo—P(2)	79·4 (1)	Mo—N—N^{I}	175·6 (4)
P(1)—Mo—N	85·0 (1)	P(2)—Mo—N	82·7 (1)
P(1)—C(10)	1·837 (5)	P(2)—C(13)	1·853 (5)
P(1)—C(11)	1·824 (5)	P(2)—C(14)	1·820 (5)
P(1)—C(12)	1·848 (5)	P(2)—C(15)	1·837 (5)
C(12)-C(13)	1·496 (6)		
Mo—P(1)—C(10)	123·7 (3)	Mo—P(2)—C(13)	111·1 (4)
Mo—P(1)—C(11)	117·6 (3)	Mo—P(2)—C(14)	121·6 (4)
Mo—P(1)—C(12)	111·3 (4)	Mo—P(2)—C(15)	118·6 (4)
C(10)-P(1)—C(11)	99·4 (4)	C(13)-P(2)—C(14)	101·6 (5)
C(11)-P(1)—C(12)	101·3 (4)	C(14)-P(2)—C(15)	100·1 (5)
C(12)-P(1)—C(10)	100·0 (4)	C(15)-P(2)—C(13)	100·6 (5)
P(1)—C(12)-C(13)	109·2 (7)	P(2)—C(13)-C(12)	109·5 (7)
C(1)—C(2)	1·434 (6)	C(6)—C(1)	1·387 (5)
C(2)—C(3)	1·410 (6)	C(1)—C(7)	1·508 (6)
C(3)—C(4)	1·418 (5)	C(3)—C(8)	1·512 (6)
C(4)—C(5)	1·398 (5)	C(5)—C(9)	1·515 (6)
C(5)—C(6)	1·412 (5)		
C(2)—C(1)—C(6)	117·1 (7)	C(4)—C(3)—C(8)	120·8 (8)
C(2)—C(1)—C(7)	121·0 (7)	C(3)—C(4)—C(5)	121·5 (6)
C(6)—C(1)—C(7)	121·8 (8)	C(4)—C(5)—C(6)	118·5 (7)
C(1)—C(2)—C(3)	122·0 (7)	C(4)—C(5)—C(9)	121·1 (7)
C(2)—C(3)—C(4)	117·8 (7)	C(6)—C(5)—C(9)	120·2 (7)
C(2)—C(3)—C(8)	121·3 (7)	C(5)—C(6)—C(1)	122·8 (7)

Symmetry operator superscript: I $-x, -y, -z$.

complexes of molybdenum (Prout, Allison, Delbaere & Gore, 1972; Prout, Cameron, Forder, Critchley, Denton & Rees, 1974). The carbon skeleton of the mesitylene ligand is not quite planar (Table 4). The three methyl carbon atoms lie significantly out of the mean plane of the C_6-ring on the opposite side to the molybdenum whilst the ring carbon atoms themselves deviate by up to 5σ.

Table 4. *Planarity of mesitylene ligand*

The equation of the least-squares best plane through atoms C(1) to C(6) is

$$3{\cdot}519x + 15{\cdot}908y - 1{\cdot}625z = 3{\cdot}544$$

where x, y, z are fractional coordinates referred to the crystallographic axes.

Deviations (Å) of atoms from the plane

Constituent atoms		Non-constituent atoms	
C(1)	0·002	Mo	−1·793
C(2)	−0·011	C(7)	0·113
C(3)	0·018	C(8)	0·167
C(4)	−0·016	C(9)	0·160
C(5)	0·006		
C(6)	0·001		

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