A similar situation has been found in the study of the molecular packing of glycerolipids, where an extreme chain tilt of 60° has been observed in the crystal structure of 2,3-dilauroyl-D-glycerol (Pascher, Sundell & Hauser, 1981). These authors underline that in a bilayer structure the chain tilt increases with the space requirement of the head group according to the relation $\cos\varphi = \Sigma/S$ where φ, Σ and S stand for the chain tilt angle, the area of cross section of the hydrocarbon chain and the area per polar head in the layer plane respectively.

Crystals of a second triclinic phase of SDS have been obtained from aqueous solution and are under structural investigation. Simultaneously work is in progress in order to correlate the structural information obtained from crystal phases with that of micellar aggregates in aqueous solution.

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Oligophosphine Ligands. XVII.* *trans*-Bis(dinitrogen)-*mer*-[bis(3-diphenylphosphinopropyl)phenylphosphine](trimethylphosphine)molybdenum(0)

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Abstract. $[\mathrm{Mo(N_2)_2\{P(CH_3)_3\}\{P(C_6H_5)[(CH_2)_3\text{-}P(C_6H_5)_2]_2\}}]$, $M_r = 790{\cdot}66$, orthorhombic, $P2_12_12_1$, $a = 10{\cdot}893\,(2)$, $b = 15{\cdot}573\,(3)$, $c = 22{\cdot}989\,(5)$ Å, $V = 3900\,(1)$ Å^3, $Z = 4$, $D_x = 1{\cdot}347$ g cm^{-3}, $\lambda(\mathrm{Mo}\,K\alpha) = 0{\cdot}71069$ Å, $\mu(\mathrm{Mo}\,K\alpha) = 4{\cdot}66$ cm^{-1}, $F(000) = 1640$, $T = 293$ K, $R = 0{\cdot}037$ for 4104 observed reflexions. The X-ray study has shown the presence of two dinitrogen ligands in *trans* positions, with the phosphines lying in the equatorial plane of the coordination octahedron. The Mo—N bond lengths are 1·998 (4) and 2·031 (4) Å, and the Mo—P distances within the six-membered chelate rings are 2·438 (1)–2·481 (1) Å. The Mo—$\mathrm{P(CH_3)_3}$ separation is 2·508 (1) Å.

Introduction. The crystal structure of the title compound, synthesized as described in a previous paper (Dahlenburg & Pietsch, 1986), has been determined as part of a broad study of bis(dinitrogen)molybdenum complexes containing flexible trimethylene-linked tris(tertiary phosphine) ligands.

Experimental. Orange, transparent crystals of the compound under investigation were grown from tetrahydrofuran/methanol at room temperature, and a fragment of approximate dimensions 0·2 × 0·2 × 0·3 mm was used for all X-ray measurements. Syntex $P2_1$ diffractometer; graphite-monochromated Mo $K\alpha$ radiation; cell parameters by least-squares refinement of the setting angles of 15 reflexions within $16 < 2\theta < 25°$; intensity measurements by $\theta/2\theta$ scans ($4{\cdot}5 \leq 2\theta \leq 55°$, $0 \leq h \leq 14$, $0 \leq k \leq 20$, $0 \leq l \leq 29$); no significant decline in intensity for three standards monitored every 100 reflexions. 5029 data (all unique) collected out of which 29 systematically absent ($h00$, $0k0$, $00l$ for h, k and l odd, respectively), 893 unobserved [$|F_o| < 4\sigma(F_o)$], and three suppressed (111, 022, and 041 which apparently suffered from secondary extinction). Lorentz–polarization correction, absorption ignored. Structure solved by Patterson and

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* Part XVI: Antberg & Dahlenburg (1986).

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difference Fourier methods and anisotropically refined by full-matrix least-squares procedures on F employing the *SHELX* program (Sheldrick, 1976). H atoms included in geometrically idealized positions (C—H = 0·96 Å). Convergence at R = 0·037 and wR = 0·032 (436 parameters, 4104 data); function minimized $\sum w(|F_o| - |F_c|)^2$ with weights $w = (\sigma^2|F_o| + 0{\cdot}0001|F_o|^2)^{-1}$. Maximum shift/e.s.d. in final cycle = 0·6, largest features on final difference map +0·4 and −0·4 e Å^{-3}. Complex neutral-atom scattering factors as incorporated in *SHELX* for C, H, N, P and, for Mo, from *International Tables for X-ray Crystallography* (1974).

Discussion. Final positional and thermal parameters are given in Table 1, and selected bond distances and angles are reported in Table 2.* Fig. 1 shows a view of the molecule and illustrates the atom-labeling scheme chosen.

The overall molecular geometry about the central molybdenum atom corresponds to distorted octahedral with the tris(tertiary phosphine) spanning meridional coordination sites and the dinitrogen ligands occupying *trans* positions. Bond lengths and angles derived from the analysis are much more reliable than those of the closely related species *mer,trans*-$Mo(N_2)_2[PhP(CH_2CH_2CH_2PPh_2)_2](PMe_2Ph)$, the structural model of which has previously been found to refine in an unsatisfactory manner (Dahlenburg & Pietsch, 1986). The structural features of the *trans*-NN—Mo—NN moiety of the title complex, *i.e.* the Mo—N bond lengths, 1·998 (4) and 2·031 (4) Å, the NN distances, 1·119 (5) and 1·123 (5) Å, as well as the Mo—N—N angles, 176·7 (5) and 176·9 (4)°, compare well with those observed for other dinitrogen complexes of Mo^0 containing *trans*-$Mo(N_2)_2$ fragments, *e.g.*, *trans*-$Mo(N_2)_2(Ph_2PCH_2CH_2PPh_2)_2$ (Uchida, Uchida, Hidai & Kodama, 1975) and *mer*-$Mo(N_2)_3(n\text{-}Pr_2PPh)_3$ (Anderson, Hughes & Richards, 1984). The Mo—P distances within the six-membered chelate rings, 2·438 (1)–2·481 (1) Å, are close to those [2·445 (1) and 2·462 (1) Å] found for $Mo(N_2)_2(Ph_2PCH_2CH_2PPh_2)_2$ (Uchida *et al.*, 1975), whereas the Mo—PMe_3 separation of 2·508 (1) Å is considerably longer than that of, *e.g.*, *trans*-$MoCl(N_2)(PMe_3)_4$ [2·461 (1) Å] (Carmona, Marin, Poveda, Atwood & Rogers, 1983*a*). This difference nonwithstanding, the Mo—PMe_3 length of the title compound should also be considered normal since the range covered by d(Mo—P) is wide (2·3–2·6 Å) as has been pointed out by others (Carmona, Marin, Poveda, Atwood & Rogers, 1983*b*).

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Table 1. *Positional and thermal parameters*

$U_{eq} = \frac{1}{3}$ (trace of the orthogonalized U_{ij} tensor).

	x	y	z	U_{eq}(Å^2)
Mo	0·54450 (3)	0·49183 (2)	0·55772 (2)	0·0357 (3)
P(1)	0·4278 (1)	0·3793 (1)	0·5027 (1)	0·0536 (13)
P(2)	0·4878 (1)	0·4295 (1)	0·6532 (1)	0·0429 (11)
P(3)	0·6377 (1)	0·6118 (1)	0·6096 (1)	0·0428 (11)
P(4)	0·6074 (1)	0·5660 (1)	0·4669 (1)	0·0442 (12)
N(1)	0·6984 (4)	0·4183 (2)	0·5592 (2)	0·049 (4)
N(2)	0·7840 (4)	0·3784 (3)	0·5629 (2)	0·076 (6)
N(3)	0·3964 (4)	0·5675 (2)	0·5599 (2)	0·048 (4)
N(4)	0·3167 (4)	0·6127 (3)	0·5623 (2)	0·078 (6)
C(1)	0·4074 (8)	0·2728 (3)	0·5321 (3)	0·115 (11)
C(2)	0·2660 (5)	0·4057 (5)	0·4864 (3)	0·099 (9)
C(3)	0·4781 (6)	0·3456 (4)	0·4303 (3)	0·100 (9)
C(4)	0·3382 (4)	0·3736 (3)	0·6623 (2)	0·050 (5)
C(5)	0·2336 (5)	0·4164 (4)	0·6436 (2)	0·060 (6)
C(6)	0·1190 (5)	0·3790 (4)	0·6479 (3)	0·072 (7)
C(7)	0·1071 (6)	0·2981 (4)	0·6709 (3)	0·078 (8)
C(8)	0·2087 (6)	0·2528 (4)	0·6880 (3)	0·080 (8)
C(9)	0·3240 (5)	0·2915 (4)	0·6849 (2)	0·064 (6)
C(10)	0·5921 (5)	0·3496 (3)	0·6856 (2)	0·052 (5)
C(11)	0·6243 (5)	0·2776 (4)	0·6537 (3)	0·065 (6)
C(12)	0·6963 (6)	0·2116 (4)	0·6761 (3)	0·072 (7)
C(13)	0·7421 (6)	0·2185 (4)	0·7313 (3)	0·089 (9)
C(14)	0·7135 (7)	0·2888 (5)	0·7636 (3)	0·101 (10)
C(15)	0·6403 (6)	0·3544 (4)	0·7420 (3)	0·079 (8)
C(16)	0·8043 (4)	0·6186 (3)	0·6249 (2)	0·049 (5)
C(17)	0·8514 (6)	0·6698 (5)	0·6675 (3)	0·088 (8)
C(18)	0·9772 (6)	0·6795 (5)	0·6743 (3)	0·106 (10)
C(19)	1·0565 (5)	0·6397 (4)	0·6382 (3)	0·076 (7)
C(20)	1·0116 (5)	0·5910 (4)	0·5933 (3)	0·062 (6)
C(21)	0·8858 (5)	0·5798 (4)	0·5868 (2)	0·058 (6)
C(22)	0·7606 (4)	0·5426 (3)	0·4375 (2)	0·045 (4)
C(23)	0·7928 (5)	0·4575 (3)	0·4289 (2)	0·058 (6)
C(24)	0·9035 (5)	0·4357 (4)	0·4042 (2)	0·069 (6)
C(25)	0·9856 (5)	0·4969 (4)	0·3881 (3)	0·078 (7)
C(26)	0·9558 (7)	0·5806 (4)	0·3958 (3)	0·099 (9)
C(27)	0·8439 (5)	0·6034 (4)	0·4201 (3)	0·081 (7)
C(28)	0·5160 (5)	0·5576 (4)	0·3988 (2)	0·054 (5)
C(29)	0·3919 (5)	0·5791 (4)	0·4022 (3)	0·066 (6)
C(30)	0·3196 (6)	0·5765 (4)	0·3522 (3)	0·082 (8)
C(31)	0·3673 (5)	0·5521 (3)	0·3005 (2)	0·101 (6)
C(32)	0·4886 (7)	0·5315 (6)	0·2967 (3)	0·121 (12)
C(33)	0·5642 (6)	0·5347 (4)	0·3453 (2)	0·089 (8)
C(34)	0·4638 (4)	0·5042 (3)	0·7145 (2)	0·055 (4)
C(35)	0·5664 (5)	0·5700 (3)	0·7253 (2)	0·061 (6)
C(36)	0·5679 (5)	0·6408 (3)	0·6799 (2)	0·058 (6)
C(37)	0·6187 (5)	0·7187 (3)	0·5760 (2)	0·057 (5)
C(38)	0·6742 (5)	0·7304 (3)	0·5167 (2)	0·062 (6)
C(39)	0·6041 (7)	0·6845 (5)	0·4689 (3)	0·061 (10)

Table 2. *Selected bond lengths* (Å) *and bond angles* (°)

P(1)—Mo	2·508 (1)	N(1)—Mo	2·031 (4)
P(2)—Mo	2·478 (1)	N(3)—Mo	1·998 (4)
P(3)—Mo	2·438 (1)	N(1)—N(2)	1·123 (5)
P(4)—Mo	2·481 (1)	N(3)—N(4)	1·119 (5)
P(2)—Mo—P(1)	92·69 (5)	N(1)—Mo—P(4)	92·8 (1)
P(3)—Mo—P(1)	173·48 (5)	N(3)—Mo—P(1)	90·9 (1)
P(3)—Mo—P(2)	88·33 (5)	N(3)—Mo—P(2)	90·4 (1)
P(4)—Mo—P(1)	92·34 (5)	N(3)—Mo—P(3)	82·6 (1)
P(4)—Mo—P(2)	174·83 (5)	N(3)—Mo—P(4)	88·3 (1)
P(4)—Mo—P(3)	86·54 (5)	N(3)—Mo—N(1)	176·9 (2)
N(1)—Mo—P(1)	91·9 (1)	N(2)—N(1)—Mo	176·7 (5)
N(1)—Mo—P(2)	88·3 (1)	N(4)—N(3)—Mo	176·9 (4)
N(1)—Mo—P(3)	94·6 (1)		

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* Tables of structure factors, anisotropic thermal parameters, and H-atom positional and isotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42899 (26 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

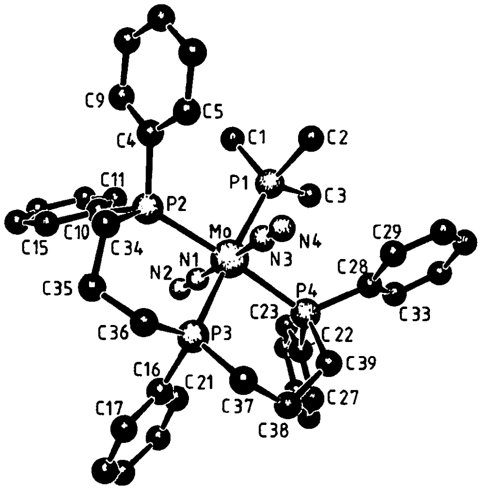

Fig. 1. Perspective view of the molecule $Mo(N_2)_2[PhP(CH_2CH_2CH_2PPh_2)_2](PMe_3)$ [*SCHAKAL* drawing (Keller, 1981)].

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Oligophosphine Ligands. XVIII.* Chlorohydrido[tris(3-dimethylphosphinopropyl)-phosphine]ruthenium(II)

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Abstract. $[Ru(Cl)(H)\{P[(CH_2)_3P(CH_3)_2]_3\}]$, $M_r = 477{\cdot}88$, trigonal, space group $P3_2$ (or its enantiomorph $P3_1$), $a = 9{\cdot}896\,(3)$, $c = 19{\cdot}589\,(5)$ Å, $V = 1661\,(1)$ Å^3, $Z = 3$, $D_x = 1{\cdot}433$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu(\text{Mo } K\alpha) = 10{\cdot}05$ cm^{-1}, $F(000) = 744$, $T = 293$ K, $R = 0{\cdot}0495$ for 4168 observed reflexions. The X-ray analysis has revealed a *trans*-H—Ru—P(CH$_2$—)Me$_2$ moiety within the title complex, the overall geometry of which corresponds to distorted *cis*-octahedral. The Ru—P bond lengths vary considerably [2·217 (2)–2·343 (2) Å] and the Ru—Cl distance is 2·532 (2) Å.

Introduction. $Ru(Cl)(H)[P(CH_2CH_2CH_2PMe_2)_3]$ was prepared according to Antberg & Dahlenburg (1986). Since it was not possible from ^{1}H and ^{31}P NMR spectra to deduce whether the hydride ligand was *trans* to the bridging P(CH$_2$—)$_3$ fragment or *trans* to one of the terminal Me$_2$P substituents, we decided to establish this structural feature by means of a single-crystal diffraction study.

Experimental. Small, colourless crystals of the complex grew from hexane at room temperature, and a specimen of approximate dimensions 0·1 × 0·1 × 0·15 mm was chosen for X-ray work. Syntex $P2_1$ diffractometer; graphite-monochromated Mo $K\alpha$ radiation; crystal data from single-crystal diffractometry using 14 medium-angle reflexions within the range $15 < 2\theta < 22°$; collection of the intensity data by the $\theta/2\theta$ scan technique ($5 \leq 2\theta \leq 55°$, $0 \leq h \leq 12$, $-12 \leq k \leq 12$, $-26 \leq l \leq 26$); no unusual variation of intensity for three checks measured every 100 reflexions. 8323 data obtained, 5043 independent ($R_{\text{int}} = 0{\cdot}0163$), 4168 considered observed [$|F_o| > 4\sigma(F_o)$]. Lorentz and polarization corrections, no absorption correction. Laue symmetry ($\bar{3}$) and systematic absences ($00l$ with $l \neq 3n$) compatible with space group $P3_1$ or its enantiomorph $P3_2$. Structure solved by heavy-atom method and initially refined in $P3_1$ by full-matrix least-squares procedures on F assigning anisotropic thermal

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