

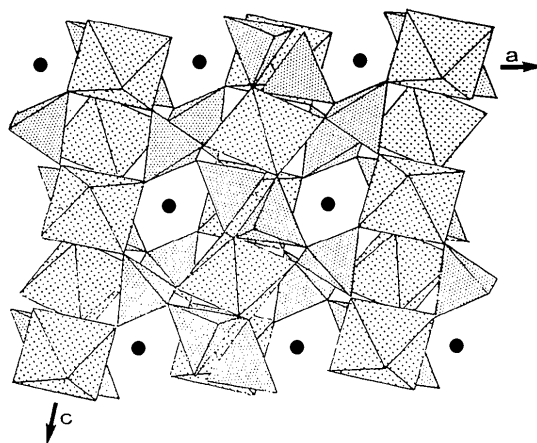
Table 2. Bond lengths (Å) and angles (°)

The $M-O$ distances are on the diagonal; above it are the $O\cdots O$ distances and below it are the $O-M-O$ angles.

P(1)	O(1)	O(2)	O(3)	O(4)		
O(1)	1.503 (5)	2.517 (7)	2.517 (7)	2.522 (7)		
O(2)	112.5 (3)	1.524 (5)	2.467 (7)	2.528 (7)		
O(3)	112.9 (3)	108.5 (3)	1.517 (5)	2.460 (7)		
P(4)	109.2 (3)	108.6 (3)	104.7 (3)	1.589 (5)		
P(2)	O(4)	O(5)	O(6)	O(7)		
O(4)	1.610 (5)	2.541 (7)	2.500 (7)	2.419 (7)		
O(5)	109.1 (3)	1.509 (5)	2.469 (7)	2.529 (7)		
O(6)	106.2 (3)	109.5 (3)	1.515 (5)	2.568 (7)		
O(7)	101.5 (3)	113.7 (3)	116.1 (3)	1.512 (5)		
V(1)	O(1 ⁱ)	O(1 ⁱⁱ)	O(2)	O(2 ⁱⁱⁱ)	O(6)	O(6 ⁱⁱⁱ)
O(1 ⁱ)	1.956 (5)	3.048 (10)	2.792 (7)	3.977 (10)	2.864 (7)	2.712 (7)
O(1 ⁱⁱ)	102.3 (3)	1.956 (5)	3.977 (10)	2.792 (7)	2.712 (7)	2.864 (7)
O(2)	88.4 (2)	166.4 (2)	2.049 (5)	2.699 (10)	2.817 (7)	3.109 (7)
O(2 ⁱⁱⁱ)	166.4 (2)	88.4 (2)	82.4 (3)	2.049 (5)	3.109 (7)	2.817 (7)
O(6)	91.0 (2)	84.9 (2)	86.6 (2)	98.4 (2)	2.059 (5)	4.112 (10)
O(6 ⁱⁱⁱ)	84.9 (2)	91.0 (2)	98.4 (2)	86.6 (2)	173.4 (3)	2.059 (5)
V(2)	O(3 ^{iv})	O(3)	O(5 ^v)	O(5 ^{vi})	O(7 ^{vii})	O(7 ^{viii})
O(3 ^{iv})	1.984 (5)	3.968 (8)	2.929 (8)	2.647 (7)	2.944 (7)	2.717 (8)
O(3)	180.0 (0)	1.984 (5)	2.647 (7)	2.929 (8)	2.717 (8)	2.944 (7)
O(5 ^v)	95.8 (2)	84.2 (2)	1.964 (5)	2.928 (8)	2.637 (8)	2.990 (7)
O(5 ^{vi})	84.2 (2)	95.8 (2)	180.0 (0)	1.964 (5)	2.990 (7)	2.637 (8)
O(7 ^{vii})	94.6 (2)	85.4 (2)	82.8 (2)	97.2 (2)	2.022 (5)	4.044 (8)
O(7 ^{viii})	85.4 (2)	94.6 (2)	97.2 (2)	82.8 (2)	180.0	2.022 (5)
Ba—O(2 ⁱⁱⁱ)	2.799 (5)	Ba—O(5 ^{vi})	2.953 (5)			
Ba—O(2 ^{iv})	2.799 (5)	Ba—O(6 ⁱⁱⁱ)	3.062 (5)			
Ba—O(3 ⁱⁱⁱ)	2.933 (5)	Ba—O(6 ^{viii})	3.062 (5)			
Ba—O(3 ^{iv})	2.933 (5)	Ba—O(7)	2.841 (5)			
Ba—O(5 ⁱ)	2.953 (5)	Ba—O(7 ^v)	2.841 (5)			

Symmetry code: (i) $-x, -y+1, -z$; (ii) $+x, 1-y, \frac{1}{2}+z$; (iii) $-x, y, \frac{1}{2}-z$; (iv) $-x, -y, -z$; (v) $-\frac{1}{2}+x, -\frac{1}{2}+y, +z$; (vi) $\frac{1}{2}-x, \frac{1}{2}-y, -z$; (vii) $-\frac{1}{2}+x, \frac{1}{2}-y, -\frac{1}{2}+z$; (viii) $-\frac{1}{2}+x, -\frac{1}{2}+y, \frac{1}{2}-z$; (ix) $\frac{1}{2}+x, \frac{1}{2}-y, \frac{1}{2}+z$; (x) $\frac{1}{2}-x, \frac{1}{2}-y, 1-z$.

to approach it in a time limited to 60 s. 1023 reflections with $I/\sigma(I) > 3$ used to solve and refine the structure. Correction made for Lorentz, polarization, secondary extinction and absorption ($T_{\min} = 0.04$, $T_{\max} = 0.13$). Structure solved by heavy-atom method. All subsequent calculations on a MicroVAX II computer with the SDP System (B. A. Frenz &

Fig. 1. Structure of $BaV_2(P_2O_7)_2$ onto the ac plane.

Associates, Inc., 1982). All atoms refined anisotropically on F . Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). ($\Delta/\sigma_{\max} = 0.05$, $S = 0.994$, $\Delta\rho_{\max} = 0.93 \text{ e } \text{Å}^{-3}$, $R = 0.038$, $wR = 0.044$, $w = f(\sin\theta/\lambda)$). Atomic parameters in Table 1, bond distances and angles in Table 2.*

A view of the structure of $BaV_2(P_2O_7)_2$ onto the ac plane is shown in Fig. 1.

Related literature. Title compound is isostructural with $BaTi_2(P_2O_7)_2$ (Wang & Hwu, 1991).

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54309 (7 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Refinement of the Structure of Si_2N_2O

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Abstract. Silicon nitride oxide, $M_r = 100.18$, orthorhombic, Cmc_2 , $Z = 4$, $\lambda(\text{Cu K}\alpha) = 1.54178 \text{ Å}$, $a =$

$8.8717 (6)$, $b = 5.4909 (16)$, $c = 4.8504 (7) \text{ Å}$, $V = 236.28 (12) \text{ Å}^3$, $D_x = 2.816 \text{ g cm}^{-3}$, $\mu = 114 \text{ cm}^{-1}$, $F(000) = 200$, $R = 0.0222$ for 24 parameters and 192 unique observed reflections at $T = 296 \text{ K}$ and $a =$

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8.8742 (10), $b = 5.4894$ (15), $c = 4.8464$ (14) Å, $V = 236.09$ (16) Å³, $D_x = 2.818$ g cm⁻³, $\mu = 114$ cm⁻¹, $F(000) = 200$, $R = 0.0205$ for 24 parameters and 136 unique observed reflections at $T = 123$ K. The crystal structure of Si₂N₂O has been refined from X-ray diffraction data obtained by a rotating anode X-ray source. Our results confirm the structure determined by earlier X-ray work [Idrestedt & Brosset (1964). *Acta Chem. Scand.* **18**, 1879–1886] and provide an improved precision in the determination of the atomic coordinates. The influence of temperature on coordinates and temperature factors is negligible.

Experimental. Single crystals of Si₂N₂O were grown from a gas-phase reaction between SiO and N₂ at 1723 K according to the procedure described by Idrestedt & Brosset (1964), with minor modifications. The low O₂ partial pressure needed was maintained by the equilibrium between Si(l) and SiO₂. In the heterogeneous product, colourless elongated crystals were found, which by Hägg–Guinier film techniques were identified as Si₂N₂O. A rectangular prism crystal with dimensions 0.112 × 0.025 × 0.025 mm was selected for data collection at 296 and 123 K on a Rigaku AFC6R diffractometer using graphite-monochromated Cu $K\alpha$ radiation from an RU200 rotating anode operated at 9 kW (50 kV; 180 mA). Because the space group is polar, data were collected for Bijvoet pairs \bar{h}, \bar{k}, l and h, k, \bar{l} , giving a total of 223 unique reflections ($R_{\text{int}} = 0.012$) at room temperature, out of 252 measured using the $\omega/2\theta$ scan mode with an ω -scan rate of 16° min⁻¹ and a scan width of $(1.70 + 0.30 \tan \theta)^\circ$. The experimental condition only permitted 187 reflections to be measured at 123 K, of which 164 were unique ($R_{\text{int}} = 0.015$). Of the 223 reflections, collected at room temperature with $10 < 2\theta < 124^\circ$, 192 had $I > 3\sigma(I)$ and were considered observed. At 123 K, the number of observed reflections was 136. Because of the high linear absorption coefficient ($\mu = 114$ cm⁻¹), an analytical absorption correction was applied (de Meulenaer & Tompa, 1965) which resulted in transmission factors ranging from 0.79 to 0.90. Cell constants from refinement of setting angles for 25 reflections in the range $80 < 2\theta < 100^\circ$ for the data collected at 296 K, while those for the 123 K data set were determined by refinement of setting angles of 11 reflections with $15 < 2\theta < 65^\circ$.

As a starting point for refinement, coordinates from the previous determination (Idrestedt & Brosset, 1964) were used. Atomic scattering factors and f' and f'' values were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). All refinements were carried out with the *TEXSAN* (1989) crystallographic programs. The least-squares refinements were carried out on F and the weights, w , were calculated as $w = 4F_o^2/\sigma^2(F_o^2)$. Refinements

including anisotropic thermal parameters for all atoms gave final $R = 0.022$, $wR = 0.031$ and $S = 1.72$ for 24 parameters and 192 reflections for the data set collected at 296 K (as compared to $wR = 0.032$ for the reversed polar direction). Max. and min. heights in the final $\Delta\rho$ map were 0.26 and -0.30 e Å⁻³, respectively, and max. Δ/σ was less than 0.002. For the refinement based on data collected at 123 K, the corresponding values were: $R = 0.020$, $wR = 0.029$ and $S = 1.69$ for 136 reflections; max. and min. residual electron densities were 0.28 and -0.31 e Å⁻³, respectively; max. $\Delta/\sigma < 0.03$. Throughout the data collection, the intensities for three standard reflections ($\bar{1}\bar{1}\bar{2}$, $\bar{3}10$, $\bar{3}\bar{1}0$) remained constant within 0.8% at room temperature and within 2.1% at 123 K.

The results of the refinements for both temperatures are given in Table 1.* The atomic positions are almost identical for the two temperatures. The difference in temperature factors for the two temperatures is, at most, just outside the e.s.d.'s. Atomic distances and angles are given in Table 2. The structural features of Si₂N₂O are confirmed, including the distorted SiON₃ tetrahedron and the almost planar NSi₃ unit, see Fig. 1.

Related literature. In the phase system AlN–Si₃N₄–SiO₂–Al₂O₃, only a few structures of nitrogen-containing compounds determined from single crystals, other than Si₂N₂O, have been reported, viz AlN (Schulz & Thiemann, 1977), α -Si₃N₄ (Kato, Inoue, Kijima, Kawada, Tanaka & Yamane, 1975) and β -Si₃N₄ (Grün, 1979). Powder techniques are becoming increasingly important, allowing not only for structure solution and refinement of complex phases, but also for studies of structural changes under varying external conditions, e.g. high-temperature and high-pressure studies. Such information is available for Si₂N₂O (Srinivasa, Cartz, Jorgensen, Worlton, Beyerlein & Billy, 1977; Billy, Labbe, Selvaraj & Roult, 1980; Baraton, Billy, Labbe, Quintard & Roult, 1988). In general, good single-crystal data are required as references to the structural refinements based on powder data.

Since the previous determination of the structure of Si₂N₂O (Idrestedt & Brosset, 1964), technical improvements have made further refinement possible, enabling extended and more precise determination of structural parameters. In particular, anisotropic temperature factors have been added. Calculated isotropic temperature factors (B) for data collected at 296 K, 0.74 (7), 0.44 (3) and 0.46 (7) Å²

* Lists of structure factors and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54297 (7 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Positional and equivalent isotropic displacement parameters (Å²)
$$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i \cdot a_j$$

	x	y	z	B _{eq}		
T = 296 K						
O	0.000	0.2127 (7)	0.2300 (0)	0.8 (2)		
Si	0.1767 (1)	0.1511 (2)	0.2815 (8)	0.41 (4)		
N	0.2191 (4)	0.1228 (5)	0.6267 (12)	0.5 (2)		
	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
O	0.004 (2)	0.009 (2)	0.016 (3)	0.00	0.00	0.001 (2)
Si	0.004 (1)	0.006 (1)	0.006 (1)	0.000 (1)	-0.001 (1)	0.000 (1)
N	0.004 (2)	0.011 (2)	0.006 (3)	-0.001 (1)	-0.003 (2)	0.001 (2)
T = 123 K						
O	0.000	0.2128 (8)	0.2300 (0)	0.7 (2)		
Si	0.1768 (1)	0.1512 (2)	0.2813 (11)	0.41 (6)		
N	0.2192 (4)	0.1227 (6)	0.627 (2)	0.5 (2)		
	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
O	0.001 (2)	0.008 (2)	0.016(4)	0.00	0.00	0.000 (3)
Si	0.003 (1)	0.006 (1)	0.006 (1)	0.001 (1)	-0.001 (1)	-0.001 (1)
N	0.002 (2)	0.012 (3)	0.005 (3)	0.002 (2)	0.001 (2)	0.001 (2)

Table 2. Interatomic distances (Å) and bond angles (°) for Si₂N₂O

	T = 298 K		T = 123 K	
Si—O	1.623 (1)		1.624 (2)	
Si—N ^{vii}	1.720 (3)		1.718 (4)	
Si—N ⁱ	1.723 (3)		1.724 (4)	
Si—N ^{iv}	1.723 (3)		1.709 (4)	
	N—N/O distance		N—N/O distance	
N ⁱ —Si—N ^{vii}	111.8 (1)	2.852 (3)	111.8 (1)	2.850 (4)
N ⁱ —Si—N ^{viii}	107.3 (2)	2.775 (3)	107.2 (2)	2.772 (4)
N ^{iv} —Si—N ^{vii}	108.8 (1)	2.800 (2)	108.9 (2)	2.799 (2)
N ^{vii} —Si—O	107.6 (2)	2.698 (4)	107.6 (2)	2.697 (4)
N ⁱ —Si—O	112.3 (2)	2.778 (5)	112.3 (2)	2.781 (6)
N ^{iv} —Si—O	109.0 (2)	2.725 (4)	109.0 (2)	2.725 (5)
	Si—Si distance		Si—Si distance	
Si ⁱ —N—Si ^{iv}	117.0 (2)	2.939 (1)	117.0 (2)	2.938 (1)
Si ⁱ —N—Si ^{vii}	118.4 (2)	2.958 (1)	118.3 (2)	2.956 (1)
Si ^{iv} —N—Si ^{vii}	123.8 (2)	3.038 (1)	124.0 (3)	3.037 (1)
Si ^{iv} —O—Si ⁱⁱ	150.0 (3)	3.136 (2)	150.0 (3)	3.138 (2)

Symmetry code: (i) x, y, z; (ii) -x, y, z; (iii) -x, -y, $\frac{1}{2} + z$; (iv) x, -y, $\frac{1}{2} + z$; (v) $\frac{1}{2} + x$, $\frac{1}{2} + y$, z; (vi) $\frac{1}{2} - x$, $\frac{1}{2} + y$, z; (vii) $\frac{1}{2} - x$, $\frac{1}{2} - y$, $\frac{1}{2} + z$; (viii) $\frac{1}{2} + x$, $\frac{1}{2} - y$, $\frac{1}{2} + z$.

for O, Si and N, respectively, differ somewhat from the previous results for Si₂N₂O, but both isotropic and anisotropic temperature factors can be compared to those found in studies on Si—O—Si bridges in silicates (Liebau, 1985) and on Si₃N₄ (Grün, 1979; Kato, Inoue, Kijima, Kawada, Tanaka & Yamane, 1975). The largest and most directed atomic displacements are found for oxygen, as expected.

The nearly planar configuration of the NSi₃ unit is a characteristic feature of structures containing Si—N bonds, as in the AlN—Si₃N₄—SiO₂—Al₂O₃ system, which seems, from powder-diffraction

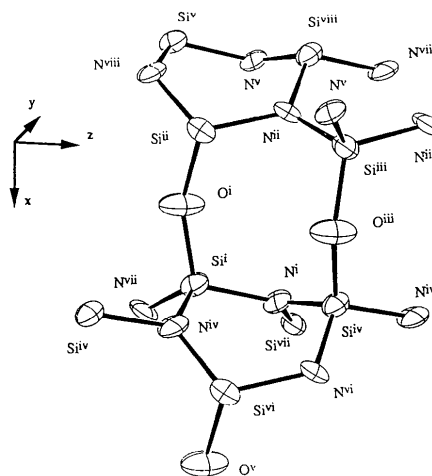


Fig. 1. An ORTEP (Johnson, 1976) drawing of a fragment of the structure of Si₂N₂O. The thermal ellipsoids are drawn for a 98% probability level. Equivalent positions are given in Table 2.

studies on phases where Al and O replaces Si and N, not to be restricted to Si—N bonds (Gillott, Cowlam & Bacon, 1981; Lindqvist, Sjöberg, Hull & Pompe, 1991). While the situation of possible contribution from π bonds between silicon and nitrogen (e.g., Morgan, 1977; Domashevskaya, Timoshenko, Terekhov, Desyatirikova, Bulycheva & Seleznev, 1989) is not clear (e.g. Robertson, 1981), the electron density on either side of the plane — a result of the nitrogen p_z orbital not taking part in the σ bonds — would have some influence on the stability of the configuration, especially when a substantial fraction of the N atoms are replaced by oxygen as in Si_{6-z}Al_zN_{8-z}O_z, 0 < z < 4 (Ekström, Käll, Nygren & Olsson, 1989), or, to a lesser extent, as in Si_{2-x}Al_xN_{2-x}O_{x-1}, 0 < x < 0.2 (Trigg & Jack, 1987). The configuration may also be influenced by non-bonded Si...Si interactions (O'Keeffe & Hyde, 1978). Expected distances between Si atoms in contact are ≈ 3.06 Å, as compared to the range 2.93–3.14 Å observed for Si₂N₂O.

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Structure of *trans*-[Rh(CO)Cl{P(C₆H₅)₃}₂]: a Centrosymmetric Triclinic Phase

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Abstract. *trans*-[Rh(CO)Cl{P(C₆H₅)₃}₂], $M_r = 690.96$, triclinic, $P\bar{1}$, $a = 9.182$ (3), $b = 9.640$ (2), $c = 10.384$ (2) Å, $\alpha = 107.56$ (2), $\beta = 89.70$ (2), $\gamma = 110.48$ (2)°, $V = 815.7$ (9) Å³, $Z = 1$, $D_x = 1.41$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 7.2$ cm⁻¹, $F(000) = 352$, $T = 298$ K, $R = 0.036$ for 2878 reflections with $I > 3\sigma(I)$. The molecule lies at an inversion center with a disordered Cl atom and CO group. Rh—C(10) = 1.759 (1), Rh—Cl = 2.380 (2), Rh—P = 2.328 (1) Å. The molecular structure is similar to the previously reported noncentrosymmetric triclinic ($P1$) structure. However, unlike the previous report, the distance between C(10) and O(10), C—O = 1.14 (1) Å, is well defined and reasonable.

Experimental. Yellow crystals of *trans*-carbonylchlorobis(triphenylphosphino)rhodium(I), *trans*-Rh(CO)Cl(PPh₃)₂, were grown from CH₂Cl₂/Et₂O. A suitable column-like crystal with dimensions 0.15 × 0.17 × 0.32 mm was mounted on a glass fiber on an Enraf-Nonius CAD-4 diffractometer equipped with monochromated Mo $K\alpha$ radiation. Cell constants were derived from least-squares refinement of 25 reflections having $19 < 2\theta < 24^\circ$. Intensity data were collected at room temperature using the $\theta/2\theta$ scan technique with $2 < 2\theta < 55^\circ$ ($h = -11$ to 11; $k = 0$ to 12; $l = -13$ to 12). Three standard reflections were monitored every 3600 s, and only small (<2%) random variations were observed. A total of 3728 unique reflections were measured, of which 2878

were observed [$I > 3\sigma(I)$]. Lorentz and polarization corrections were applied. An empirical correction based on a series of ψ scans was applied to the data; $T_{\min} = 0.87$, $T_{\max} = 0.99$.

The atomic position of Rh was fixed at (0,0,0). The remaining non-H atoms were located in successive difference Fourier syntheses. H-atom positions were calculated using ideal geometry but were not included in the least-squares refinements. Atomic scattering factors including anomalous dispersion were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). The structure was refined by full-matrix least squares using *Personal SDP* (Frenz, 1989) on an 80386-based IBM compatible PC. All non-H atoms were refined anisotropically. $\sum w(|F_o| - |F_c|)^2$ was minimized, where $w = 4F_o^2/[\sigma^2(I) + 0.02(F_o)^2]$. A secondary-extinction coefficient refined to a value of $\chi = 5.21 \times 10^{-7}$, at which point the correction factor $(1 + \chi I)^{-1}$ was applied to F_c . Based on the centrosymmetric space group $P\bar{1}$, final $R = 0.036$, $wR = 0.045$, $R_{\text{all}} = 0.059$ and $S = 1.617$ obtained using 206 variables. The largest shift/e.s.d. was 0.02; maximum and minimum residual electron densities in the final difference Fourier map were 0.52 and -0.40 e Å⁻³.

The atoms Cl—Rh—C(10)—O(10) are almost colinear [angle of Cl—Rh—C(10) is 179.7 (3)°]. The disordered model in $P\bar{1}$ leads to three atom sites for these ligands on each side of the Rh. The sites are sufficiently well resolved that atomic positional and anisotropic displacement parameters can be refined satisfactorily. Refinements based on the non-centrosymmetric space group $P1$ were also carried out. CO and Cl remained disordered, and the

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