

The crystal structure analysis shows the molecule to be (II) and that it adopts the expected chair-chair conformation. The bond lengths are all within the accepted ranges and the N–O length of 1.28 Å in the nitroxide group agrees with previous crystal structure analyses of these radicals [N–O (av.) = 1.28 Å] (Rassat & Rey, 1973).

The geometry of the nitroxide group is of interest as this group has not been extensively studied. A summary of the results obtained so far (Rassat & Rey, 1973) suggests that in non-planar rings the nitroxide group is pyramidal, with the N–O bond at 19(2)° to the C–N–C plane. The present structure shows an angle of 17.0(5)°. The distortion from planarity is outwards from the body of the molecule as this does most to relieve steric interactions with the carbonyl group. By comparison, the carbonyl group is planar within experimental error.

The conformation of the molecule, although the expected twin-chair, shows some distortion from ideal geometry even when the effects of the  $sp^2$  hybridized C(3) and N are considered. The bond angles at C(2), C(4), C(6), and C(8) are all opened by 3–4° and the torsional angles in the rings suggest considerable flattening of the rings, particularly round the nitroxide

group, where they are some 10° smaller than near the carbonyl group. This might be due to a greater flexibility in the C–N–C system of nitroxide radicals. Additional evidence for the distortion of the ring system is shown by the interplanar angle C(2)C(3)C(4)/C(6)NC(8) of 41.3(7)° which for an ideal 3,7-dione system would be less than 10°.

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## Tetraamminepalladium(II) Pyrazine-2,5-dicarboxylate

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**Abstract.** [Pd(NH<sub>3</sub>)<sub>4</sub>]C<sub>4</sub>N<sub>2</sub>H<sub>2</sub>(CO<sub>2</sub>)<sub>2</sub>, C<sub>6</sub>H<sub>14</sub>O<sub>4</sub>N<sub>6</sub>Pd, M.W. 340.6, triclinic,  $P\bar{1}$ ,  $a = 7.145$  (1),  $b = 10.685$  (1),  $c = 3.771$  (1) Å,  $\alpha = 99.10$  (1),  $\beta = 98.78$  (1),  $\gamma = 97.90$  (1)°, 23°C,  $Z = 1$ ,  $D_x = 2.041$ ,  $D_m = 2.040$  g cm<sup>-3</sup>,  $V = 277.07$  Å<sup>3</sup>. Full-matrix least-squares refinement of 1268 observed reflections collected with  $\theta$ - $2\theta$  scans on a Syntex  $P2_1$  yielded a final agreement index,  $R$ , of

0.038. Multiple hydrogen bonds are formed between the square-planar tetraamminepalladium(II) cations and the pyrazine-2,5-dicarboxylate anions.

**Introduction.** The unit-cell dimensions were determined by a least-squares refinement of 29 high-angle reflections ( $\text{Cu } K\alpha_1 = 1.54050$ ,  $\text{Cu } K\alpha_2 = 1.54433$  Å). The

Table 1. Atomic coordinates and thermal parameters with *e.s.d.*'s

Positional parameters are  $\times 10^4$ ;  $\beta_{11}$ ,  $\beta_{22}$  and  $\beta_{12}$  are  $\times 10^4$ ;  $\beta_{33}$ ,  $\beta_{13}$  and  $\beta_{23}$  are  $\times 10^3$ .

The thermal parameters are of the form  $\exp[-(h^2\beta_{11} + k^2\beta_{22} + l^2\beta_{33} + 2hk\beta_{12} + 2hl\beta_{13} + 2kl\beta_{23})]$ .

	$x$	$y$	$z$	$\beta_{11}$	$\beta_{22}$	$\beta_{33}$	$\beta_{12}$	$\beta_{13}$	$\beta_{23}$
Pd	0	0	0	63 (1)	42 (1)	44 (1)	20 (1)	4 (0)	0 (0)
O(1)	4918 (6)	1699 (4)	5808 (12)	200 (11)	48 (4)	82 (4)	25 (5)	6 (2)	-2 (1)
O(2)	7884 (6)	2781 (4)	6438 (13)	162 (11)	84 (5)	99 (5)	58 (6)	16 (2)	-1 (1)
N(1)	2731 (7)	-146 (5)	-762 (14)	103 (10)	83 (6)	71 (5)	42 (6)	10 (2)	-1 (1)
N(2)	842 (7)	1904 (5)	1974 (14)	134 (11)	64 (5)	63 (5)	22 (6)	6 (2)	2 (1)
N(3)	3676 (7)	3863 (4)	8759 (14)	102 (10)	51 (5)	85 (5)	13 (6)	8 (2)	-3 (1)
C(1)	6155 (9)	2682 (5)	6752 (15)	156 (14)	56 (6)	45 (5)	44 (7)	5 (2)	2 (1)
C(2)	5530 (8)	3913 (5)	8482 (15)	108 (12)	48 (5)	43 (4)	25 (6)	6 (2)	1 (1)
C(3)	6812 (8)	5039 (6)	9732 (18)	96 (12)	57 (6)	84 (6)	20 (7)	11 (2)	-1 (2)



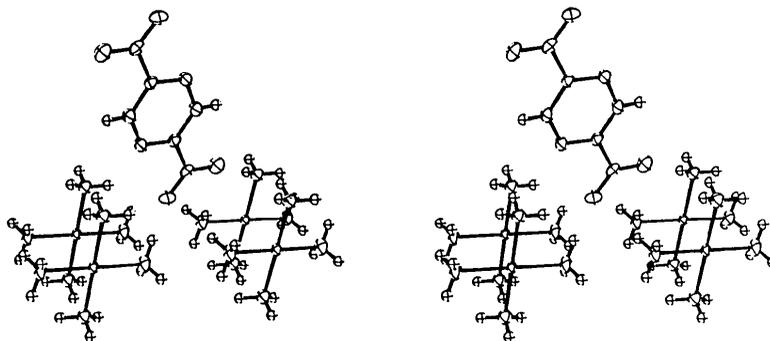


Fig. 2. Partial packing diagram.

Fig. 1 also shows (dashed lines) some of the inter-ionic distances between possible hydrogen-donor atoms, N(1) and N(2), and hydrogen-acceptor atoms, O(1), O(2) and N(3); the dashed lines represent all distances less than 3.3 Å. In addition Fig. 2, a partial packing diagram, shows the relative orientation of the hydrogen atoms attached to N(1) and N(2) and gives a clearer indication of possible hydrogen bonds.

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### The Crystal Structure of Tetra-(2-thienyl)silane

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**Abstract.**  $(C_4H_3S)_4Si$ , tetragonal,  $P\bar{4}2_1c$ ,  $a = 11.368$  (9),  $c = 6.535$  (5) Å,  $Z = 2$ ,  $D_c = 1.42$ ,  $D_o = 1.42$  (1) g cm<sup>-3</sup> (by flotation in aqueous KI solution). The structure is orientationally disordered. Each thienyl ring has two conformations rotated 179° with respect to the Si–C(1) bond. The structure was refined by rigid-body least-squares methods to  $R = 9.1\%$ .

**Introduction.** Colorless, elongated needle crystals of tetra-(2-thienyl)silane were grown by slow evaporation of a benzene solution from a sample kindly provided by Dr L. Spialter of the Air Force Materials Laboratory, Wright–Patterson Air Force Base, Ohio. A single crystal of approximate dimensions 0.12 × 0.12 × 0.25 mm was used. From indexed Weissenberg photographs the systematic absences  $h00$  for  $h$  odd and  $hhl$  for  $l$  odd uniquely determined the space group to be  $P\bar{4}2_1c$ .

Three-dimensional intensity data were collected on a Picker FACS-1 diffractometer equipped with a scin-

tillation counter and pulse-height analyzer; zirconium-filtered Mo  $K\alpha$  ( $\lambda = 0.71069$  Å) radiation was used. In total, 1159 reflections out to 55° in  $2\theta$  were measured using the  $\theta$ – $2\theta$  scan mode. In  $P\bar{4}2_1c$  the  $hkl$  and  $khl$  reflections are equivalent by symmetry for  $h \neq k$ . Averaging of the corresponding equivalent  $hkl$  and  $khl$  pairs yielded 540 reflections. There were an additional 79  $hhl$  type reflections to give a total of 619 independent reflections. Of these, 497 had  $I > \sigma(I)$  and were used in subsequent calculations.

The space group  $P\bar{4}2_1c$  has eight general positions which requires that the two silicon atoms per unit cell be constrained at special positions of  $\bar{4}$  symmetry. There is only one thienyl ring in the asymmetric unit. An electron-density map phased by the silicon atom revealed the essential features of the independent thienyl ring. However, attempts to improve the positions of the ring atoms with a series of difference Fourier syntheses proved unsuccessful. Each successive map gave consistently unrealistic bond distances