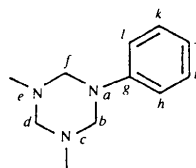


Table 4. Selected geometric parameters (Å, °) for (1b)

N(1)—C(2)	1.444 (4)	C(13)—C(18)	1.395 (4)
N(1)—C(6)	1.462 (5)	C(14)—C(15)	1.380 (4)
N(1)—C(7)	1.420 (2)	C(15)—C(16)	1.378 (4)
N(3)—C(2)	1.482 (3)	C(16)—C(17)	1.380 (3)
N(3)—C(4)	1.471 (4)	C(16)—O(26)	1.369 (3)
N(3)—C(13)	1.416 (3)	C(17)—C(18)	1.376 (4)
N(5)—C(4)	1.450 (3)	C(19)—C(20)	1.387 (3)
N(5)—C(6)	1.463 (3)	C(19)—C(24)	1.395 (3)
N(5)—C(19)	1.421 (3)	C(20)—C(21)	1.386 (3)
C(7)—C(8)	1.380 (3)	C(21)—C(22)	1.375 (4)
C(7)—C(12)	1.398 (3)	C(22)—C(23)	1.388 (3)
C(8)—C(9)	1.397 (3)	C(22)—O(27)	1.381 (4)
C(9)—C(10)	1.379 (3)	C(23)—C(24)	1.368 (4)
C(10)—C(11)	1.389 (3)	C(28)—O(25)	1.418 (4)
C(10)—O(25)	1.368 (3)	C(29)—O(26)	1.413 (5)
C(11)—C(12)	1.379 (3)	C(30)—O(27)	1.405 (5)
C(13)—C(14)	1.394 (3)		
C(2)—N(1)—C(6)	109.8 (2)	N(3)—C(13)—C(18)	119.7 (2)
C(2)—N(1)—C(7)	118.6 (3)	C(14)—C(13)—C(18)	116.8 (2)
C(6)—N(1)—C(7)	117.6 (4)	C(13)—C(14)—C(15)	121.1 (3)
C(2)—N(3)—C(4)	108.9 (2)	C(14)—C(15)—C(16)	121.1 (2)
C(2)—N(3)—C(13)	114.2 (2)	C(15)—C(16)—C(17)	118.6 (3)
C(4)—N(3)—C(13)	116.0 (2)	C(15)—C(16)—O(26)	116.2 (2)
C(4)—N(5)—C(6)	109.8 (2)	C(17)—C(16)—O(26)	125.2 (3)
C(4)—N(5)—C(19)	117.4 (2)	C(16)—C(17)—C(18)	120.4 (3)
C(6)—N(5)—C(19)	115.3 (2)	C(13)—C(18)—C(17)	121.9 (2)
N(1)—C(2)—N(3)	112.0 (2)	N(5)—C(19)—C(20)	124.0 (2)
N(3)—C(4)—N(5)	111.8 (2)	N(5)—C(19)—C(24)	118.6 (3)
N(1)—C(6)—N(5)	112.0 (2)	C(20)—C(19)—C(24)	117.4 (2)
N(1)—C(7)—C(8)	123.0 (3)	C(19)—C(20)—C(21)	121.3 (2)
N(1)—C(7)—C(12)	119.3 (2)	C(20)—C(21)—C(22)	120.4 (2)
C(8)—C(7)—C(12)	117.6 (2)	C(21)—C(22)—C(23)	118.8 (2)
C(7)—C(8)—C(9)	121.6 (2)	C(21)—C(22)—O(27)	125.8 (2)
C(8)—C(9)—C(10)	119.9 (2)	C(23)—C(22)—O(27)	115.4 (2)
C(9)—C(10)—C(11)	119.0 (2)	C(22)—C(23)—C(24)	120.7 (3)
C(9)—C(10)—O(25)	125.3 (2)	C(19)—C(24)—C(23)	121.4 (2)
C(11)—C(10)—O(25)	115.7 (2)	C(10)—O(25)—C(28)	117.1 (2)
C(10)—C(11)—C(12)	120.6 (2)	C(16)—O(26)—C(29)	117.7 (2)
C(7)—C(12)—C(11)	121.1 (2)	C(22)—O(27)—C(30)	117.3 (3)
N(3)—C(13)—C(14)	123.5 (3)		

Table 5. Conformational angles (°)



$\theta$  is the angle between planes *dagj* and *aghijkl*,  $\gamma$  is between the *a—g* bond and the *fab* plane and  $\zeta$  is between the *a—g* bond and the *ghijkl* plane.

Compound	$\theta$	$\gamma$	$\zeta$
Compound (1a)			
N(3) equatorial	40.0 (3)	44.9 (3)	3.7 (3)
N(1) axial	38.4 (3)	30.8 (3)	5.3 (3)
N(5) axial	32.8 (3)	35.5 (3)	6.0 (3)
Compound (1b)			
N(3) equatorial	63.3 (3)	43.1 (3)	3.5 (3)
N(1) axial	63.6 (3)	35.0 (3)	6.5 (3)
N(5) axial	62.4 (3)	39.5 (3)	1.4 (3)

Data collection: *CAD-4 Operations Manual* (Enraf–Nonius, 1979). Cell refinement: *CAD-4 Operations Manual*. Data reduction: *GX* (Mallinson & Muir, 1985). Program(s) used to solve structure: *MITHRIL* (Gilmore, 1984). Program(s) used to refine structure: *GX*. Molecular graphics: *ORTEP* (Johnson, 1971). Software used to prepare material for publication: *GX*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1074). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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*Acta Cryst.* (1995). **C51**, 249–251

## Non-Sinusoidal Structure of the 1:1 Complex of Phenothiazine and 7,7,8,8-Tetracyanoquinodimethane

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(Received 23 April 1993; accepted 8 July 1994)

## Abstract

The title compound,  $C_{12}H_9NS.C_{12}H_4N_4$ , has a different space group to that reported by Kobayashi [*Acta Cryst.* (1974), **B30**, 1010–1017] and does not present any disorder or sinusoidal modulation, in contrast to the first study of this compound. In particular, no  $N \cdots N$  distance of about 3.3 Å was observed.

### Comment

The title compound, (I), was studied previously by Kobayashi (1974) and proved very interesting as a result of a sinusoidal modulation and an ambiguity about the space group (*C2/c* or *Cc*). These characteristics led us to suppose that some structural changes might occur at lower temperature; we had previously undertaken a crystal structure analysis of this complex at room temperature. Fig. 1 presents an ORTEP (Johnson, 1965) drawing of (I) with the atom-numbering scheme and Fig. 2 shows a stereoscopic view of the unit cell.

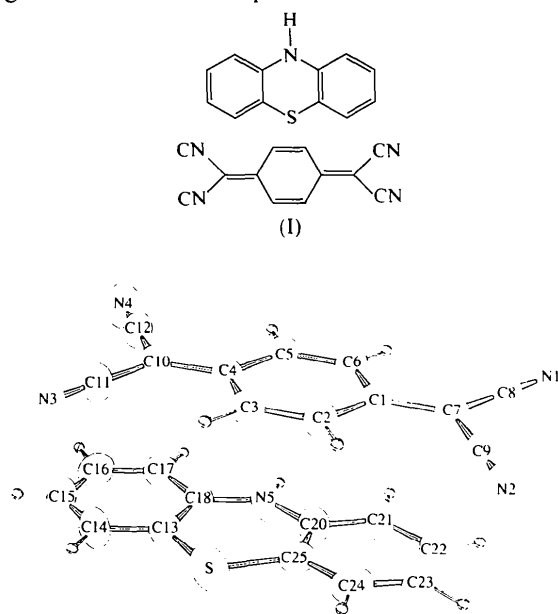


Fig. 1. ORTEP (Johnson, 1965) view of the title compound with the atom-numbering scheme. Displacement ellipsoids are plotted at the 50% probability level.

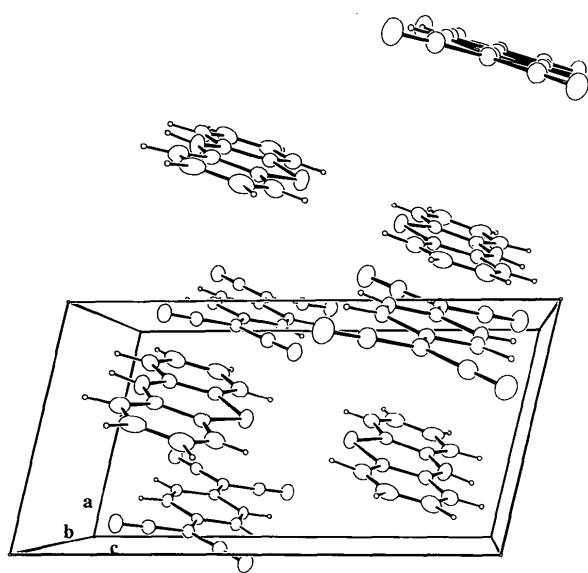


Fig. 2. Packing diagram of the unit cell.

The geometry of the title compound in this crystallographic phase is quite homogeneous and, in particular, does not show either an N...N distance of about 3.3 Å, as in the reported sinusoidal phase, or paired columns due to the hydrogen-bond network. Preliminary studies with photographic films failed to reveal the sinusoidal aspect observed previously. There is no geometrical relationship between the monoclinic sinusoidal phase and the triclinic ordered phase. Furthermore, the shapes of the samples are quite different (needle or prism). The possibility of formation of a disordered sinusoidal phase may arise from particular unpublished parameters applied by Kobayashi during the growth method.

### Experimental

Phenothiazine (PTZ) was purified by liquid chromatography under red safety lights, sublimed and further purified by multiple zone refining (Karl, 1980). 7,7,8,8-Tetracyanoquinodimethane (TCNQ), which cannot be melted without decomposition, was only purified by repeated directional vacuum sublimation in a temperature gradient. Single crystals of PTZ:TCNQ (typical size up to 6 × 2 × 2 mm) were grown from the separate components *in vacuo* by the plate-sublimation technique described elsewhere (Karl, 1989).

#### Crystal data

C<sub>12</sub>H<sub>9</sub>NS.C<sub>12</sub>H<sub>4</sub>N<sub>4</sub>

*M<sub>r</sub>* = 403.5

Triclinic

*P* $\bar{1}$

*a* = 7.066 (3) Å

*b* = 10.657 (6) Å

*c* = 13.046 (7) Å

$\alpha$  = 91.99 (5)°

$\beta$  = 103.15 (5)°

$\gamma$  = 90.52 (4)°

*V* = 956 (1) Å<sup>3</sup>

*Z* = 2

*D<sub>x</sub>* = 1.40 Mg m<sup>-3</sup>

Mo K $\alpha$  radiation

$\lambda$  = 0.7107 Å

Cell parameters from 25 reflections

$\theta$  = 7–11°

$\mu$  = 1.82 mm<sup>-1</sup>

*T* = 294 K

Prism

0.20 × 0.20 × 0.20 mm

Black

#### Data collection

Enraf–Nonius CAD-4

diffractometer

$\omega/2\theta$  scans

Absorption correction:

none

3663 measured reflections

2040 independent reflections

2040 observed reflections

[*I* > 3 $\sigma$ (*I*)]

*R*<sub>int</sub> = 0.016

$\theta_{\max}$  = 25°

*h* = 0 → 8

*k* = -12 → 12

*l* = -15 → 15

3 standard reflections

frequency: 60 min

intensity decay: 0.3%

#### Refinement

Refinement on *F*<sup>2</sup>

*R* = 0.057

*wR* = 0.056

*S* = 0.81

2040 reflections

311 parameters

Only coordinates of H atoms refined

$w = 1/[\sigma^2(I) + (0.04F_o^2)^2]^{1/2}$

( $\Delta/\sigma$ )<sub>max</sub> = 0.3

$\Delta\rho_{\max}$  = 0.80 e Å<sup>-3</sup>

$\Delta\rho_{\min}$  = 0.05 e Å<sup>-3</sup>

Atomic scattering factors

from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ )
$$B_{\text{eq}} = (4/3) \sum_i \sum_j \beta_{ij} \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	$B_{\text{eq}}$
S	0.5400 (2)	0.2585 (1)	0.60652 (9)	5.29 (3)
N1	0.0693 (6)	-0.1333 (3)	0.9300 (3)	5.13 (9)
N2	-0.1170 (6)	-0.1338 (3)	0.5880 (3)	5.5 (1)
N3	0.1930 (6)	0.6103 (4)	0.5806 (3)	5.7 (1)
N4	0.3884 (6)	0.6327 (3)	0.9208 (3)	5.27 (9)
N5	0.6877 (5)	0.2407 (3)	0.8475 (3)	4.88 (8)
C1	0.0773 (5)	0.1177 (3)	0.7603 (3)	2.94 (8)
C2	0.0474 (5)	0.1815 (3)	0.6639 (3)	3.29 (8)
C3	0.0968 (5)	0.3034 (3)	0.6614 (3)	3.27 (8)
C4	0.1866 (5)	0.3731 (3)	0.7573 (3)	3.00 (8)
C5	0.2127 (5)	0.3096 (3)	0.8552 (3)	3.35 (8)
C6	0.1635 (5)	0.1879 (3)	0.8572 (3)	3.37 (8)
C7	0.0259 (5)	-0.0074 (3)	0.7616 (3)	3.25 (8)
C8	0.0507 (6)	-0.0761 (4)	0.8558 (3)	3.71 (9)
C9	-0.0543 (6)	-0.0781 (4)	0.6649 (3)	3.93 (9)
C10	0.2386 (5)	0.4976 (3)	0.7540 (3)	3.41 (8)
C11	0.2118 (6)	0.5606 (4)	0.6575 (3)	3.86 (9)
C12	0.3225 (6)	0.5719 (3)	0.8475 (3)	3.69 (9)
C13	0.6555 (6)	0.3747 (4)	0.6984 (3)	3.70 (9)
C14	0.6828 (6)	0.4921 (4)	0.6604 (4)	4.9 (1)
C15	0.7683 (7)	0.5889 (4)	0.7299 (4)	5.7 (1)
C16	0.8229 (7)	0.5698 (4)	0.8353 (4)	5.4 (1)
C17	0.7982 (6)	0.4551 (4)	0.8735 (3)	4.6 (1)
C18	0.7110 (6)	0.3552 (4)	0.8060 (3)	3.60 (8)
C20	0.6034 (6)	0.1331 (4)	0.7940 (3)	3.79 (9)
C21	0.5885 (6)	0.0256 (4)	0.8490 (4)	5.0 (1)
C22	0.5080 (6)	-0.0848 (4)	0.7960 (5)	5.9 (1)
C23	0.4372 (7)	-0.0873 (4)	0.6892 (4)	5.8 (1)
C24	0.4447 (6)	0.0190 (4)	0.6348 (4)	5.0 (1)
C25	0.5304 (6)	0.1296 (4)	0.6848 (3)	3.86 (9)
H'N5	0.734 (6)	0.235 (4)	0.916 (3)	

Table 2. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

S—C13	1.752 (4)	C7—C8	1.429 (6)
S—C25	1.750 (4)	C7—C9	1.442 (6)
N1—C8	1.145 (5)	C10—C11	1.423 (6)
N2—C9	1.144 (5)	C10—C12	1.436 (6)
N3—C11	1.132 (5)	C13—C14	1.390 (6)
N4—C12	1.140 (5)	C13—C18	1.391 (5)
N5—C18	1.376 (5)	C14—C15	1.388 (7)
N5—C20	1.378 (5)	C15—C16	1.364 (8)
N5—H'N5	0.88 (5)	C16—C17	1.360 (7)
C1—C2	1.425 (5)	C17—C18	1.401 (6)
C1—C6	1.451 (5)	C20—C21	1.388 (6)
C1—C7	1.380 (5)	C20—C25	1.399 (6)
C2—C3	1.345 (5)	C21—C22	1.392 (7)
C3—C4	1.444 (5)	C22—C23	1.368 (8)
C4—C5	1.443 (5)	C23—C24	1.365 (7)
C4—C10	1.378 (5)	C24—C25	1.391 (6)
C5—C6	1.343 (5)		
C13—S—C25	102.8 (2)	N3—C11—C10	179.1 (5)
C18—N5—C20	127.3 (4)	N4—C12—C10	178.8 (4)
C18—N5—H'N5	116 (3)	S—C13—C14	117.1 (3)
C20—N5—H'N5	116 (3)	S—C13—C18	122.9 (3)
C2—C1—C6	118.1 (3)	C14—C13—C18	120.0 (4)
C2—C1—C7	121.0 (3)	C13—C14—C15	119.7 (5)
C6—C1—C7	120.8 (3)	C14—C15—C16	120.3 (5)
C1—C2—C3	121.7 (4)	C15—C16—C17	120.6 (5)
C2—C3—C4	120.5 (4)	C16—C17—C18	120.8 (5)
C3—C4—C5	117.9 (3)	N5—C18—C13	122.2 (4)
C3—C4—C10	120.2 (3)	N5—C18—C17	119.2 (4)
C5—C4—C10	121.9 (3)	C13—C18—C17	118.6 (4)
C4—C5—C6	121.3 (4)	N5—C20—C21	119.7 (4)
C1—C6—C5	120.4 (4)	N5—C20—C25	121.5 (4)
C1—C7—C8	123.6 (3)	C21—C20—C25	118.8 (4)
C1—C7—C9	120.8 (3)	C20—C21—C22	120.6 (5)
C8—C7—C9	115.6 (3)	C21—C22—C23	120.2 (5)
N1—C8—C7	178.6 (4)	C22—C23—C24	119.8 (5)
N2—C9—C7	179.5 (4)	C23—C24—C25	121.5 (5)

C4—C10—C11	122.0 (4)	S—C25—C20	123.3 (3)
C4—C10—C12	122.2 (4)	S—C25—C24	117.6 (4)
C11—C10—C12	115.8 (3)	C20—C25—C24	119.1 (4)

All calculations were performed on a Digital MicroVAX 3100 computer with the *MolEN* package (Fair, 1990).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, including H-atom geometry, have been deposited with the IUCr (Reference: PA1062). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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*Acta Cryst.* (1995). C51, 251–253

## cis-6-Methoxycarbonyl-2,10-dioxo-1-phosphabicyclo[4.4.0]decane 1-Oxide

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(Received 20 May 1994; accepted 20 July 1994)

## Abstract

The crystal structure of the title compound,  $\text{C}_9\text{H}_{15}\text{O}_5\text{P}$ , is described. The molecule consists of two *cis*-fused six-membered rings, both in the chair conformation.

## Comment

The crystal structure of the title compound, (I), was investigated in order to establish the stereochemistry of the methoxycarbonyl group relative to the phosphoryl group. Since the two analogous model compounds, decalin and 2,10-dioxabicyclo[4.4.0]decane (Descotes,