

C22	0.2431 (4)	0.8698 (2)	0.5873 (3)	0.0438 (8)
C23	0.1509 (5)	0.9368 (3)	0.6069 (4)	0.0542 (9)
C24	0.2132 (5)	1.0174 (3)	0.6348 (4)	0.0591 (10)
C25	0.3724 (5)	1.0323 (2)	0.6461 (4)	0.0508 (9)
C26	0.4651 (4)	0.9647 (2)	0.6299 (3)	0.0411 (7)
C31	0.6456 (4)	0.8261 (2)	0.4799 (3)	0.0334 (6)
C32	0.6264 (4)	0.9031 (2)	0.4102 (3)	0.0414 (7)
C33	0.7140 (5)	0.9194 (3)	0.3234 (4)	0.0544 (10)
C34	0.8190 (5)	0.8594 (3)	0.3064 (4)	0.0561 (10)
C35	0.8378 (5)	0.7830 (3)	0.3728 (4)	0.0540 (9)
C36	0.7512 (4)	0.7650 (2)	0.4605 (3)	0.0452 (9)

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

Rh—N3	1.964 (3)	P—C31	1.830 (3)
Rh—N1	1.970 (3)	P—C11	1.828 (3)
Rh—N2	1.984 (3)	N1—O1	1.366 (4)
Rh—N4	1.986 (3)	N2—O2	1.315 (4)
Rh—C9	2.119 (4)	N3—O3	1.362 (4)
Rh—P	2.4543 (11)	N4—O4	1.314 (4)
P—C21	1.818 (4)		
N3—Rh—N1	172.59 (13)	N1—Rh—P	91.89 (9)
N3—Rh—N2	100.14 (13)	N2—Rh—P	89.44 (10)
N1—Rh—N2	78.36 (13)	N4—Rh—P	98.27 (9)
N3—Rh—N4	78.74 (12)	C9—Rh—P	176.97 (13)
N1—Rh—N4	101.76 (12)	C21—P—C31	106.8 (2)
N2—Rh—N4	172.3 (2)	C21—P—C11	102.2 (2)
N3—Rh—C9	85.8 (2)	C31—P—C11	99.84 (15)
N1—Rh—C9	86.9 (2)	C21—P—Rh	109.32 (11)
N2—Rh—C9	87.6 (2)	C31—P—Rh	114.76 (11)
N4—Rh—C9	84.7 (2)	C11—P—Rh	122.30 (11)
N3—Rh—P	95.35 (9)		

Data reduction was performed using *XP21* (Pavelčík, 1993). Intensities were corrected for Lorentz and polarization factors using *XP21*. The structure was solved by heavy-atom methods with *XFPS* (Pavelčík, Rizzoli & Andreotti, 1990) and subsequent Fourier syntheses using *SHELXL93* (Sheldrick, 1993). Anisotropic displacement parameters were refined for all non-H atoms. Both hydroxyl H atoms were located from difference Fourier maps and refined with isotropic displacement factors fixed at  $0.06 \text{\AA}^2$ . The H atoms attached to carbon were apparent in difference maps and were included as riding atoms in calculated positions with isotropic displacement factors. Geometrical analysis was performed using *PARST* (Nardelli, 1983) and *SHELXL93*. *ORTEP* (Johnson, 1965) was employed to produce the figure. *SHELXL93* was used to prepare the material for publication.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1053). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## 2,4,6-Tris(2-pyridyl)-1,3,5-triazine-Lithium Tetraphenylborate Bis(ethanol) Solvate

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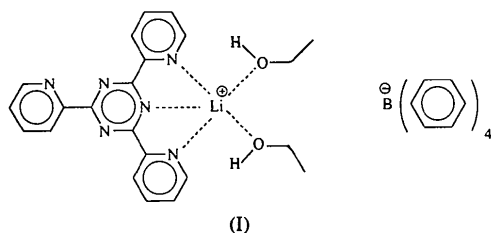
## Abstract

The first crystal structure determination of an alkali metal complex of 2,4,6-tris(2-pyridyl)-1,3,5-triazine reveals a five-coordinate Li cation with three contacts to the N atoms and two more to the O atoms of the ethanol solvent, *i.e.* bis(ethanol-*O*)[2,4,6-tris(2-pyridyl)-*N*<sup>2</sup>,*N*<sup>6</sup>-1,3,5-triazine-*N*<sup>1</sup>]lithium tetraphenylborate, [Li-(C<sub>18</sub>H<sub>12</sub>N<sub>6</sub>)(C<sub>2</sub>H<sub>6</sub>O)<sub>2</sub>][(C<sub>6</sub>H<sub>5</sub>)<sub>4</sub>B]. The tetraphenylborate anion is not in contact with the cation.

## Comment

2,4,6-Tris(2-pyridyl)-1,3,5-triazine (TPTZ) has been used for the spectrophotometric analytical determination of iron(II), cobalt(II) and ruthenium(II) (Janmohamed & Ayres, 1972). However, very few structures of its transition metal complexes have been determined so far. TPTZ acts as mono-, bi- or tridentate ligand, in

some instances simultaneously (Halfpenny & Small, 1982), contrary to theoretical predictions (Durham, Frost & Hart, 1969). Recently, the DNA-cleavage ability of a TPTZ-complexed aquaruthenium(II) reagent was reported (Gupta *et al.*, 1993). The lack of any structural studies on alkali metal or alkali earth metal complexes of TPTZ prompted us to prepare the lithium complex, (I), and to determine its structure.



The Li cation is positioned close to the direction of the C3—N1 axis of the triazine ring and is  $\eta^3$ -coordinated by the TPTZ half-shell ligand with two significantly different distances to the pyridyl N atoms. The rather long Li1—N20 distance of 2.461 (5) Å to one pyridyl ring is obviously a result of rotation by 9° around the C—C bond to the triazine ring. The second pyridyl ring, with a shorter coordination contact (by 0.21 Å), is twisted by only 2° out of plane. The Li1—N1 contact of 2.074 (5) Å to the triazine N atom is significantly shorter, in agreement with analogous structural results for transition metal complexes with the TPTZ ligand (Faus, Julve, Amigó & Debaerdemaeker, 1989). In the coordination sphere of the lithium complex, two additional contacts to the ethanol solvent atoms O1 and O2 are observed, at the expected distances of 1.910 (5)

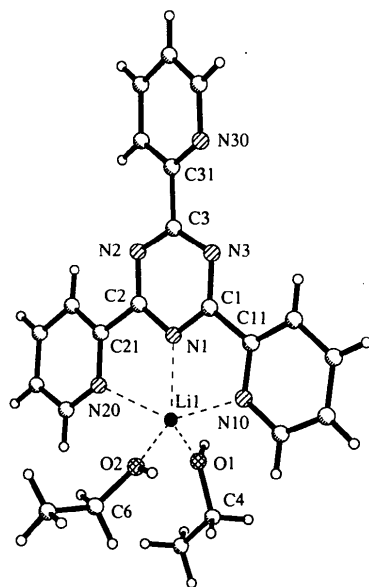


Fig. 1. View of the five-coordinate lithium cation (Li contacts are shown as dotted lines).

and 1.973 (5) Å, respectively. The Li<sup>+</sup> cation is located within the triangular plane formed by the coordination sites O1, O2 and N1 and the sum of angles around Li to O1, O2 and N1 is 360°. The angle N10—Li1—N20 [146.4 (2)°] leads to a distorted trigonal bipyramidal conformation.

The TPTZ ligand displays expected bond lengths and angles (Table 2), but is flattened in comparison with the free ligand molecule (Bock, Van & Bats, 1995) in which the pyridyl rings are twisted by 32, 19 and 16° out of the triazine ring plane. Within standard deviations, the triazine ring is planar. The third and non-coordinating pyridyl ring is rotated by 14° out of the triazine plane. Its N atom, N30, forms a weak hydrogen bridge [N30...O1A 2.903 (3) Å, N...H1A—O1A 154°] to an ethanol O—H group generated by symmetry operation  $-x + 1, -y, -z + 1$ .

## Experimental

The title compound was prepared by layering ethanolic solutions of lithium tetraphenylborate and 2,4,6-tris(2-pyridyl)-1,3,5-triazine dihydrochloride. After 3 days yellow blocks were obtained.

### Crystal data

[Li(C<sub>18</sub>H<sub>12</sub>N<sub>6</sub>)(C<sub>2</sub>H<sub>6</sub>O)<sub>2</sub>]-  
(C<sub>24</sub>H<sub>20</sub>B)  
*M<sub>r</sub>* = 730.62  
Triclinic  
*P* $\bar{1}$   
*a* = 10.253 (1) Å  
*b* = 13.061 (1) Å  
*c* = 16.593 (1) Å  
 $\alpha$  = 112.94 (1)°  
 $\beta$  = 99.40 (1)°  
 $\gamma$  = 91.09 (1)°  
*V* = 2010.5 (3) Å<sup>3</sup>  
*Z* = 2  
*D<sub>x</sub>* = 1.207 Mg m<sup>-3</sup>

Mo *K*α radiation  
 $\lambda$  = 0.71073 Å  
Cell parameters from 48 reflections  
 $\theta$  = 7.5–18.5°  
 $\mu$  = 0.074 mm<sup>-1</sup>  
*T* = 200 (2) K  
Block  
0.68 × 0.44 × 0.40 mm  
Yellow

### Data collection

Siemens *P4* four-circle diffractometer  
 $\omega$ -scans  
Absorption correction: none  
7478 measured reflections  
7057 independent reflections  
5134 observed reflections  
[*I* > 2σ(*I*)]

*R*<sub>int</sub> = 0.0197  
 $\theta_{\text{max}}$  = 25.50°  
*h* = 0 → 12  
*k* = -14 → 14  
*l* = -20 → 19  
4 standard reflections monitored every 100 reflections  
intensity decay: negligible

### Refinement

Refinement on *F*<sup>2</sup>  
*R*[*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.060  
*wR*(*F*<sup>2</sup>) = 0.155  
*S* = 1.073  
7055 reflections

Extinction correction: *SHELXL93* (Sheldrick, 1993)  
Extinction coefficient: 0.015 (5)

510 parameters

$$w = 1/[\sigma^2(F_o^2) + (0.0792P)^2 + 1.2450P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} < 0.001$$

$$\Delta\rho_{\max} = 0.373 \text{ e } \text{Å}^{-3}$$

$$\Delta\rho_{\min} = -0.247 \text{ e } \text{Å}^{-3}$$

Atomic scattering factors  
from *International Tables  
for Crystallography* (1992,  
Vol. C, Tables 4.2.6.8 and  
6.1.1.4)

Li1—N10	2.248 (5)	C1—C11	1.490 (3)
Li1—N20	2.461 (5)	C2—C21	1.483 (3)
O1—C4	1.423 (3)	C3—C31	1.489 (3)
C4—C5	1.470 (5)	C11—C12	1.374 (3)
O2—C6	1.447 (5)	C12—C13	1.391 (3)
C6—C7	1.417 (7)	C13—C14	1.370 (4)
N1—C1	1.330 (3)	C14—C15	1.376 (4)
N1—C2	1.340 (3)	C21—C22	1.380 (3)
N2—C2	1.328 (3)	C22—C23	1.373 (4)
N2—C3	1.334 (3)	C23—C24	1.371 (4)
N3—C1	1.339 (3)	C24—C25	1.377 (4)
N3—C3	1.346 (3)	C31—C32	1.385 (3)
N10—C15	1.337 (3)	C32—C33	1.387 (4)
N10—C11	1.349 (3)	C33—C34	1.362 (4)
N20—C25	1.334 (3)	C34—C35	1.384 (4)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{Å}^2$ )

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{eq}}$		$U_{\text{eq}}$		$U_{\text{eq}}$
Li1	0.4829 (5)	0.0246 (4)	0.3005 (3)	0.0548 (11)	O1—Li1—O2	111.8 (2)	N2—C3—N3	125.3 (2)
O1	0.6640 (2)	-0.0083 (2)	0.31115 (12)	0.0606 (5)	O1—Li1—N1	125.5 (3)	N2—C3—C31	116.7 (2)
C4	0.7265 (3)	-0.0588 (3)	0.2363 (2)	0.0667 (8)	O2—Li1—N1	122.6 (3)	N3—C3—C31	118.0 (2)
C5	0.8171 (5)	-0.1408 (3)	0.2462 (3)	0.0928 (12)	O1—Li1—N10	95.2 (2)	N10—C11—C12	123.3 (2)
O2	0.4430 (2)	0.1106 (2)	0.2260 (2)	0.0772 (7)	O2—Li1—N10	105.9 (2)	N10—C11—C1	114.2 (2)
C6	0.5346 (5)	0.1798 (4)	0.2067 (3)	0.1068 (15)	N1—Li1—N10	74.7 (2)	C12—C11—C1	122.5 (2)
C7	0.5478 (6)	0.1398 (6)	0.1162 (4)	0.153 (3)	O1—Li1—N20	103.9 (2)	C11—C12—C13	118.4 (3)
N1	0.3383 (2)	-0.0312 (2)	0.35176 (12)	0.0397 (5)	O2—Li1—N20	92.4 (2)	C14—C13—C12	119.1 (2)
N2	0.1693 (2)	-0.1645 (2)	0.33974 (13)	0.0412 (5)	N1—Li1—N20	71.7 (2)	C13—C14—C15	118.6 (2)
N3	0.2509 (2)	-0.0150 (2)	0.47956 (13)	0.0411 (5)	N10—Li1—N20	146.4 (2)	N10—C15—C14	123.9 (3)
N10	0.5031 (2)	0.1488 (2)	0.44220 (13)	0.0436 (5)	O1—C4—C5	113.0 (3)	N20—C21—C22	123.4 (2)
N20	0.3632 (2)	-0.1474 (2)	0.18290 (14)	0.0515 (6)	C7—C6—O2	114.3 (5)	N20—C21—C2	115.8 (2)
N30	0.1045 (2)	-0.1153 (2)	0.55704 (14)	0.0573 (6)	C1—N1—C2	115.5 (2)	C22—C21—C2	120.8 (2)
C1	0.3311 (2)	0.0199 (2)	0.43745 (15)	0.0371 (5)	C2—N2—C3	115.4 (2)	C23—C22—C21	118.8 (3)
C2	0.2555 (2)	-0.1237 (2)	0.30562 (15)	0.0375 (5)	C1—N3—C3	114.0 (2)	C24—C23—C22	119.0 (3)
C3	0.1725 (2)	-0.1088 (2)	0.4268 (2)	0.0396 (5)	C15—N10—C11	116.7 (2)	C23—C24—C25	118.3 (3)
C11	0.4221 (2)	0.1232 (2)	0.4895 (2)	0.0387 (5)	C25—N20—C21	116.3 (2)	N20—C25—C24	124.2 (3)
C12	0.4238 (3)	0.1858 (2)	0.5781 (2)	0.0494 (6)	C35—N30—C31	116.8 (2)	N30—C31—C32	123.2 (2)
C13	0.5130 (3)	0.2807 (2)	0.6213 (2)	0.0522 (7)	N1—C1—N3	125.3 (2)	N30—C31—C3	116.8 (2)
C14	0.5962 (3)	0.3078 (2)	0.5745 (2)	0.0498 (7)	N1—C1—C11	116.0 (2)	C32—C31—C3	120.0 (2)
C15	0.5882 (3)	0.2400 (2)	0.4860 (2)	0.0479 (6)	N3—C1—C11	118.6 (2)	C31—C32—C33	118.5 (2)
C21	0.2623 (2)	-0.1832 (2)	0.2100 (2)	0.0403 (6)	N2—C2—N1	124.4 (2)	C34—C33—C32	119.0 (2)
C22	0.1670 (3)	-0.2678 (2)	0.1539 (2)	0.0530 (7)	N2—C2—C21	118.9 (2)	C33—C34—C35	118.6 (3)
C23	0.1754 (3)	-0.3186 (2)	0.0655 (2)	0.0648 (8)	N1—C2—C21	116.7 (2)	N30—C35—C34	123.9 (3)
C24	0.2788 (3)	-0.2845 (2)	0.0362 (2)	0.0592 (8)				
C25	0.3690 (3)	-0.1990 (3)	0.0968 (2)	0.0619 (8)				
C31	0.0822 (2)	-0.1558 (2)	0.4681 (2)	0.0433 (6)				
C32	-0.0164 (3)	-0.2398 (2)	0.4146 (2)	0.0481 (6)				
C33	-0.0989 (3)	-0.2816 (2)	0.4553 (2)	0.0511 (7)				
C34	-0.0786 (3)	-0.2402 (2)	0.5459 (2)	0.0603 (8)				
C35	0.0242 (3)	-0.1581 (3)	0.5940 (2)	0.0669 (9)				
B1	1.0879 (3)	0.3592 (2)	0.2177 (2)	0.0384 (6)				
C41	1.1941 (2)	0.3693 (2)	0.1563 (2)	0.0428 (6)				
C42	1.2717 (3)	0.4694 (2)	0.1787 (2)	0.0516 (7)				
C43	1.3578 (3)	0.4832 (3)	0.1270 (2)	0.0623 (8)				
C44	1.3717 (3)	0.3957 (3)	0.0497 (2)	0.0702 (9)				
C45	1.2984 (4)	0.2960 (3)	0.0254 (2)	0.0753 (9)				
C46	1.2111 (3)	0.2835 (2)	0.0777 (2)	0.0594 (7)				
C51	0.9569 (2)	0.4259 (2)	0.20336 (15)	0.0407 (6)				
C52	0.8415 (3)	0.4128 (2)	0.2353 (2)	0.0524 (7)				
C53	0.7305 (3)	0.4696 (3)	0.2258 (2)	0.0615 (8)				
C54	0.7307 (3)	0.5408 (2)	0.1824 (2)	0.0634 (8)				
C55	0.8407 (3)	0.5550 (2)	0.1493 (2)	0.0566 (8)				
C56	0.9510 (3)	0.4985 (2)	0.1599 (2)	0.0450 (6)				
C61	1.0416 (2)	0.2258 (2)	0.1871 (2)	0.0405 (6)				
C62	1.1110 (3)	0.1563 (2)	0.2214 (2)	0.0479 (6)				
C63	1.0799 (3)	0.0420 (2)	0.1876 (2)	0.0587 (7)				
C64	0.9764 (3)	-0.0092 (2)	0.1177 (2)	0.0612 (8)				
C65	0.9040 (3)	0.0556 (2)	0.0826 (2)	0.0632 (8)				
C66	0.9356 (3)	0.1701 (2)	0.1169 (2)	0.0554 (7)				
C71	1.1617 (2)	0.4140 (2)	0.32312 (15)	0.0379 (5)				
C72	1.2896 (2)	0.3877 (2)	0.3496 (2)	0.0452 (6)				
C73	1.3508 (3)	0.4243 (2)	0.4386 (2)	0.0513 (7)				
C74	1.2894 (3)	0.4925 (2)	0.5053 (2)	0.0532 (7)				
C75	1.1667 (3)	0.5236 (2)	0.4823 (2)	0.0514 (7)				
C76	1.1049 (2)	0.4849 (2)	0.3934 (2)	0.0429 (6)				

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

Li1—O1	1.910 (5)	N20—C21	1.338 (3)
Li1—O2	1.973 (5)	N30—C35	1.335 (4)
Li1—N1	2.074 (5)	N30—C31	1.337 (3)

Data were corrected for Lorentz and polarization effects. All non-H atoms were refined with anisotropic displacement parameters. The positions of H1 and H2 were taken from the difference map. The other H atoms were located from the difference map, but then placed in geometrically ideal positions. These H atoms were refined with free isotropic displacement parameters using a riding model.

Data collection: XSCANS (Siemens, 1994). Cell refinement: XSCANS. Data reduction: XSCANS. Program(s) used to solve structure: SHELXS86 (Sheldrick, 1990a). Program(s) used to refine structure: SHELXL93 (Sheldrick, 1993). Molecular graphics: SHELXTL/PC (Sheldrick, 1990b). Software used to prepare material for publication: SHELXL93.

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: NS1001). 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**, 2004–2005

## Dimethanolbis(*N*-nitroso-*N*-phenylhydroxylaminato-*O,O'*)nickel(II)

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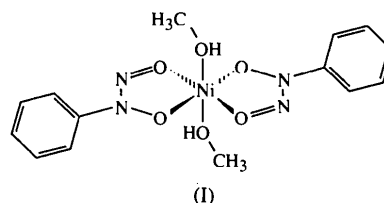
### Abstract

The title compound, [Ni(C<sub>6</sub>H<sub>5</sub>N<sub>2</sub>O<sub>2</sub>)<sub>2</sub>(CH<sub>3</sub>OH)<sub>2</sub>], contains an Ni atom in a six-coordinate state. The Ni atom displays distorted octahedral coordination. The *N*-nitroso-*N*-phenylhydroxylaminato anions lie in a *trans* position to each other, forming the equatorial plane, and the methanol ligands occupy the axial positions.

### Comment

Many metal complexes of *N*-nitroso-*N*-phenylhydroxylamine have been investigated in solution for analytical purposes, but their composition, properties and reactions have not yet been elucidated, except for a few X-ray crystallographic studies on the iron(III), copper(II) and copper(I) complexes (van der Helm, Merrit & Degeilh, 1965; Charalambous, Haines, Harris, Hendrick & Taylor, 1984). It is important to determine the crystal structures of metal complexes of *N*-nitroso-*N*-phenylhydroxylamine, not only from the analytical standpoint, but also from the biophysical one because *N*-nitroso compounds such as dimethylnitrosoamine or nitrosoourea have serious cytotoxicity as carcinogens (Nishimura *et al.*, 1985; Iishi, Tatsuta, Baba, Uehara & Nakazumi, 1994). In the present study, the crystal structure of the Ni<sup>II</sup> complex of *N*-nitroso-*N*-phenylhydroxylamine prepared in methanol solution, dimethanolbis(*N*-nitroso-*N*-phenylhydroxylaminato)nickel(II), (I), was determined

in order to clarify the binding mode of the nitroso group to the nickel(II).



The Ni coordination environment is slightly distorted octahedral. Each of the two *N*-nitroso-*N*-phenylhydroxylaminato anions is bound to the Ni atom through the O atoms of the *N*-nitroso and hydroxy groups (the latter being deprotonated), which lie in the same equatorial plane in a *trans* position to each other. Two methanol molecules at the axial sites complete the distorted octahedral arrangement around the Ni atom. The molecular packing (Fig. 2) is stabilized by

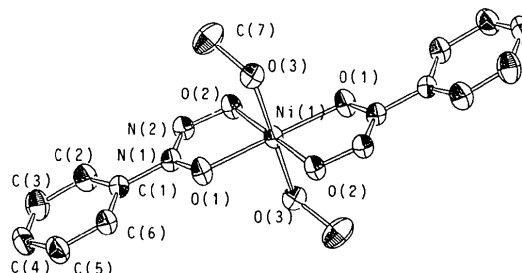


Fig. 1. ORTEP (Johnson, 1976) drawing of the title compound with the atomic numbering scheme, viewed along the *b* axis. Ellipsoids correspond to 50% probability.

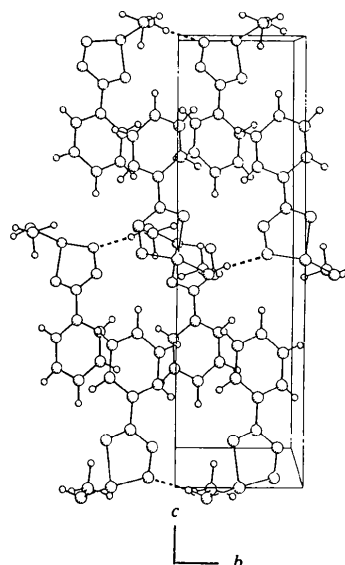


Fig. 2. Packing diagram of the title compound viewed along the *a* axis of the unit cell. Intermolecular hydrogen bonds are represented by dashed lines. The origin of the cell is in the rear left bottom corner.