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Structure of 3-[*N*-Methyl-*N*-(*S*)-*α*-methylbenzyl]carbamoyl-1,2,4-trimethylpyridinium Iodide

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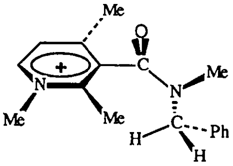
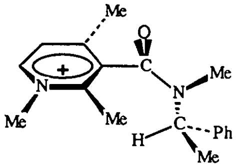
Abstract. $C_{18}H_{23}N_2O^+.I^-$, $M_r = 410{\cdot}30$, orthorhombic, $P2_12_12_1$, $a = 7{\cdot}0112\,(9)$, $b = 14{\cdot}385\,(1)$, $c = 37{\cdot}213\,(2)$ Å, $V = 3753{\cdot}2\,(6)$ Å^3, $Z = 8$, $D_x = 1{\cdot}452$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71073$ Å, $\mu = 16{\cdot}9$ cm^{-1}, $F(000) = 1648$, $T = 294$ K, final $R = 0{\cdot}0458$ for 3596 unique observed reflections. The two independent molecules have identical geometries. There are three planar moities, the pyridinium and phenyl rings, and the carbamoyl fragment in which the carbonyl and *N*-methyl groups are in the *anti* position. In both molecules the I^- ion has a short contact with the N atom of the pyridinium ring: 3·664 (8) [molecule (I)] and 3·590 (8) Å [molecule (II)].

Introduction. In a previous paper we described the crystal structures of 3-(*N*-methyl-*N*-benzyl)carbamoyl-1,2,4-trimethylpyridinium iodide (PYR) and of 3-[*N*-methyl-*N*-(*R*)-*α*-methylbenzyl]carbamoyl-1,2,4-trimethylpyridinium iodide (PYRM) (Kanters, van der Steen, Bastiaansen & de Graaf, 1986).

PYR, which crystallizes in space group $P2_12_12_1$, was found to contain a dissymmetric molecule and it was concluded that spontaneous resolution had occurred on crystallization. The chirality of PYR is caused by the two ring-methyl groups that force the carbonyl group out of the plane of the pyridinium ring, thus causing axial chirality of PYR.



PYR



PYRM

In PYRM additional chirality is introduced by the *α*-methyl group. In both PYR and PYRM the carbonyl group is directed to the *A* side† of the pyridine ring, and the carbonyl oxygen and *N*-methyl group are in a nearly eclipsed *syn* conformation. The *syn* rotamer is preferably formed when the quaternization is carried out at elevated temperature (373 K) in a nonpolar solvent (toluene), whereas the

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† The *A* side of the pyridine ring is the side which faces the observer when the ring is viewed from a direction perpendicular to the plane of the ring and one travels around the ring in a counterclockwise direction when taking the shortest path from the ring N atom to the carboxamide group. The other side is the *B* side.

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anti rotamer is formed by quaternization at low temperature (273 K) in a polar solvent (ethanol) (Bastiaansen, Kanters, van der Steen, de Graaf & Buck, 1986). Next we prepared the diastereo (*S*) *anti* rotamer of PYRM by reacting the acid chloride of 2,4-dimethylnicotinic acid with *N*-(*S*)-*α*-methylbenzylamine followed by treatment of the reaction product with CH_3I in ethanol at 273 K. Despite numerous attempts, no suitable crystals could be obtained. Therefore we decided to synthesize the analogous 2,4-dimethyl derivative, *i.e.* 3-[*N*-methyl-*N*-(*S*)-*α*-methylbenzyl]carbamoyl-2,4-dimethylpyridinium chloride which could be obtained in crystalline form. The structure analysis showed that this compound was indeed the *anti* rotamer and the carbonyl was directed to the *B*-side of the pyridine ring (Bastiaansen, Vermeulen, Buck, Smeets, Kanters & Spek, 1988).

We now report the crystal structure of the *anti* rotamer of the (*S*)-analogue of PYRM which was crystallized by slow evaporation of an ethanolic solution in a desiccator above solid potassium hydroxide at 253 K.

Experimental. A needle-shaped crystal, 0·9 × 0·13 × 0·03 mm, was used for data collection on an Enraf–Nonius CAD-4 diffractometer with Zr-filtered Mo *Kα* radiation. Lattice parameters were derived from the angular settings of 25 reflections ($6{\cdot}69 \leq \theta \leq 13{\cdot}73^\circ$). The intensity data of 4600 reflections were collected of which 3596 were above the $2{\cdot}5\sigma(I)$ level [h $0\rightarrow7$, k $0\rightarrow15$, l $-39\rightarrow39$, $2\theta_{\max} = 44^\circ$, ω–2θ scan mode with $\Delta\omega = (0{\cdot}60 + 0{\cdot}35\tan\theta)^\circ$]. The *hkl* and $hk\bar{l}$ Bijvoet pairs were not merged in order to determine the absolute configuration. Three periodically measured standard reflections (114, 120, 034) showed r.m.s. deviations of 1·03, 0·81 and 0·68% respectively. The intensity data were correced for absorption with the *DIFABS* program (Walker & Stewart, 1983) (min. and max. transmission factors 0·87 and 1·12, respectively). The structure was solved by Patterson and Fourier methods. H atoms were placed at calculated positions (C—H 1·00 Å) riding on their carrier atoms with one general isotropic temperature factor [$U = 0{\cdot}08\,(2)$ Å^2]. Anisotropic, weighted blocked full-matrix refinement on F (398 parameters) gave $R = 0{\cdot}0458$, $wR = 0{\cdot}0409$ with $w = 1{\cdot}1777[\sigma^2(F_o) + 0{\cdot}00104F_o^2]^{-1}$, $S = 1{\cdot}14$, $(\Delta/\sigma)_{\rm av} = 0{\cdot}012$, $(\Delta/\sigma)_{\max} = 0{\cdot}051$, $(\Delta\rho)_{\max} = 0{\cdot}77$, $(\Delta\rho)_{\min} = -0{\cdot}73$ e Å^{-3} (around I).

The absolute configuration was ascertained by refinement of the inverted model, resulting in $R = 0{\cdot}0495$ and $wR = 0{\cdot}0536$, which established the configuration at C(7) as (*S*) in accordance with that of its synthetic precursor *N*-(*S*)-α-methylbenzylamine. The scattering factors and anomalous-dispersion corrections were taken from *International Tables for X-ray Crystallography* (1974). Calculations were performed with *SHELX*76 (Sheldrick, 1976) (structure determination and refinement) and the *EUCLID* package (Spek, 1982) (molecular geometry and illustrations).

Table 1. *Final coordinates and equivalent isotropic thermal parameters* (Å^2) *with e.s.d.'s in parentheses*

$$U_{\rm eq} = \tfrac{1}{3}(U_{11} + U_{22} + U_{33}).$$

	x	y	z	$U_{\rm eq}$
Molecule (I)				
I(1)	0·72458 (10)	0·49740 (6)	0·15398 (2)	0·0557 (2)
O(1)	0·1325 (11)	0·6884 (5)	0·0493 (2)	0·060 (3)
N(1)	0·2923 (13)	0·5514 (6)	0·0486 (2)	0·055 (4)
N(2)	0·4993 (13)	0·7256 (5)	0·1435 (2)	0·045 (3)
C(1)	0·1202 (17)	0·3372 (8)	0·0136 (3)	0·063 (5)
C(2)	0·0511 (22)	0·2550 (9)	0·0269 (3)	0·087 (7)
C(3)	−0·0516 (22)	0·2504 (10)	0·0578 (4)	0·096 (7)
C(4)	−0·0783 (19)	0·3306 (10)	0·0768 (3)	0·088 (6)
C(5)	−0·0092 (18)	0·4142 (8)	0·0638 (3)	0·061 (5)
C(6)	0·0862 (15)	0·4206 (7)	0·0324 (3)	0·050 (4)
C(7)	0·1650 (15)	0·5152 (7)	0·0204 (2)	0·052 (4)
C(8)	0·4517 (16)	0·4935 (8)	0·0611 (3)	0·078 (5)
C(9)	0·2653 (17)	0·6398 (7)	0·0596 (2)	0·042 (4)
C(10)	0·4163 (15)	0·6791 (6)	0·0842 (2)	0·033 (4)
C(11)	0·5743 (16)	0·7181 (7)	0·0701 (3)	0·060 (5)
C(12)	0·6243 (16)	0·7146 (8)	0·0311 (2)	0·085 (6)
C(13)	0·7021 (16)	0·7595 (8)	0·0952 (3)	0·067 (5)
C(14)	0·6589 (16)	0·7621 (8)	0·1303 (3)	0·057 (5)
C(15)	0·4652 (17)	0·7269 (8)	0·1832 (2)	0·071 (5)
C(16)	0·3761 (15)	0·6815 (6)	0·1209 (3)	0·043 (4)
C(17)	0·1976 (13)	0·6384 (7)	0·1382 (2)	0·056 (4)
C(18)	0·2702 (16)	0·5134 (7)	−0·0158 (2)	0·072 (5)
Molecule (II)				
I(21)	0·01948 (11)	0·36295 (5)	0·29897 (2)	0·0508 (2)
O(21)	0·3671 (10)	0·1881 (5)	0·2080 (2)	0·065 (3)
N(21)	0·5125 (12)	0·1189 (5)	0·1612 (2)	0·047 (3)
N(22)	0·7562 (12)	0·0090 (6)	0·2668 (2)	0·044 (3)
C(21)	0·3356 (16)	0·0793 (8)	0·0738 (3)	0·061 (5)
C(22)	0·2786 (17)	0·0063 (11)	0·0512 (3)	0·072 (5)
C(23)	0·2038 (18)	−0·0726 (9)	0·0659 (4)	0·075 (6)
C(24)	0·1812 (19)	−0·0773 (9)	0·1022 (4)	0·087 (7)
C(25)	0·2286 (16)	−0·0054 (8)	0·1241 (3)	0·060 (4)
C(26)	0·3099 (16)	0·0746 (8)	0·1110 (3)	0·056 (5)
C(27)	0·3653 (15)	0·1520 (8)	0·1359 (3)	0·059 (5)
C(28)	0·6741 (15)	0·0680 (7)	0·1467 (3)	0·074 (5)
C(29)	0·4984 (17)	0·1409 (7)	0·1962 (2)	0·050 (4)
C(30)	0·6559 (14)	0·1102 (6)	0·2217 (2)	0·035 (4)
C(31)	0·8209 (16)	0·1621 (7)	0·2243 (3)	0·049 (5)
C(32)	0·8543 (15)	0·2473 (7)	0·2026 (3)	0·070 (5)
C(33)	0·9560 (16)	0·1325 (7)	0·2496 (3)	0·055 (4)
C(34)	0·9221 (17)	0·0560 (8)	0·2699 (3)	0·059 (5)
C(35)	0·7297 (17)	−0·0750 (6)	0·2894 (2)	0·063 (5)
C(36)	0·6246 (13)	0·0325 (6)	0·2423 (2)	0·030 (3)
C(37)	0·4482 (15)	−0·0266 (7)	0·2387 (2)	0·056 (5)
C(38)	0·4349 (19)	0·2431 (7)	0·1177 (3)	0·100 (6)

Discussion. The atomic coordinates and equivalent isotropic thermal parameters of the title compound are listed in Table 1.* The molecular geometries are given in Table 2. A perspective view of one of the independent molecules together with atom numbering is shown in Fig. 1. The independent molecules have similar geometries as follows from the r.m.s. deviations of corresponding distances,

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51940 (34 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

angles and exocyclic torsion angles involving non-H atoms, which amount to 0·017 (9) Å, 1·2 (8) and 5 (3)°, respectively. The fitting by the quaternion transformation method (Mackay, 1984) resulted in r.m.s. deviations of 0·188 Å for the 1,2,4-trimethylpyridinium fragment, 0·078 Å for the *N,N*-dimethylcarbamoyl fragment and 0·038 Å for the phenyl fragment respectively.

Table 2. *Bond distances* (Å), *bond angles* (°) *and selected torsion angles* (°)

	(I)	(II)
O(1)—C(9)	1·23 (1)	1·23 (1)
N(1)—C(7)	1·47 (1)	1·48 (1)
N(1)—C(8)	1·47 (1)	1·45 (1)
N(1)—C(9)	1·35 (1)	1·34 (1)
N(2)—C(14)	1·33 (1)	1·35 (1)
N(2)—C(15)	1·50 (1)	1·48 (1)
N(2)—C(16)	1·36 (1)	1·34 (1)
C(1)—C(2)	1·37 (2)	1·40 (2)
C(1)—C(6)	1·41 (1)	1·40 (2)
C(2)—C(3)	1·36 (2)	1·37 (2)
C(3)—C(4)	1·37 (2)	1·36 (2)
C(4)—C(5)	1·38 (2)	1·36 (2)
C(5)—C(6)	1·35 (2)	1·37 (2)
C(6)—C(7)	1·53 (1)	1·50 (2)
C(7)—C(18)	1·54 (1)	1·55 (2)
C(9)—C(10)	1·51 (1)	1·52 (1)
C(10)—C(11)	1·35 (1)	1·38 (1)
C(10)—C(16)	1·39 (1)	1·37 (1)
C(11)—C(12)	1·49(1)	1·49(1)
C(11)—C(13)	1·42(2)	1·40(1)
C(13)—C(14)	1·34(2)	1·36(1)
C(16)—C(17)	1·54(1)	1·51(1)
C(7)—N(1)—C(8)	119·1(8)	118·1(8)
C(7)—N(1)—C(9)	117·7(8)	119·4(9)
C(8)—N(1)—C(9)	123·0(9)	122·4(9)
C(14)—N(2)—C(15)	119·6(9)	117·8(8)
C(14)—N(2)—C(16)	119·3(9)	121·7(9)
C(15)—N(2)—C(16)	120·9(9)	120·3(8)
C(2)—C(1)—C(6)	120(1)	121(1)
C(1)—C(2)—C(3)	122(1)	119(1)
C(2)—C(3)—C(4)	118(1)	119(1)
C(3)—C(4)—C(5)	120(1)	122(1)
C(4)—C(5)—C(6)	122(1)	122(1)
C(1)—C(6)—C(5)	117(1)	117(1)
C(1)—C(6)—C(7)	123·3(9)	123(1)
C(5)—C(6)—C(7)	119·4(9)	121(1)
N(1)—C(7)—C(6)	108·9(7)	109·6(9)
N(1)—C(7)—C(18)	109·9(8)	109·3(9)
C(6)—C(7)—C(18)	114·4(8)	116·0(9)
O(1)—C(9)—N(1)	123·3(9)	122·2(9)
O(1)—C(9)—C(10)	120·6(8)	118·9(7)
N(1)—C(9)—C(10)	116·0(9)	118·9(9)
C(9)—C(10)—C(11)	119·7(8)	119·7(8)
C(9)—C(10)—C(16)	117·5(9)	117·9(8)
C(11)—C(10)—C(16)	122·5(9)	122·4(9)
C(10)—C(11)—C(12)	124(1)	122·7(9)
C(10)—C(11)—C(13)	116(1)	116·7(9)
C(12)—C(11)—C(13)	120(1)	121(1)
C(11)—C(13)—C(14)	121(1)	120(1)
N(2)—C(16)—C(10)	119·2(9)	118·4(8)
N(2)—C(14)—C(13)	123(1)	121(1)
N(2)—C(16)—C(17)	116·4(9)	118·9(7)
C(10)—C(16)—C(17)	124·4(9)	122·7(8)
C(8)—N(1)—C(7)—C(6)	55(1)	48(1)
C(8)—N(1)—C(7)—C(18)	−71(1)	−81(1)
C(9)—N(1)—C(7)—C(6)	−130·7(9)	−135·9(9)
C(9)—N(1)—C(7)—C(18)	103(1)	96(1)
C(7)—N(1)—C(9)—O(1)	6(1)	0(1)
C(7)—N(1)—C(9)—C(10)	−170·9(8)	−177·2(8)
C(8)—N(1)—C(9)—O(1)	−180(1)	176(1)
C(8)—N(1)—C(9)—C(10)	4(1)	−1(1)
C(1)—C(6)—C(7)—N(1)	−118(1)	−118(1)
C(1)—C(6)—C(7)—C(18)	5(1)	6(2)
C(5)—C(6)—C(7)—N(1)	57(1)	62(1)
C(5)—C(6)—C(7)—C(18)	−180 (1)	−173(1)
O(1)—C(9)—C(10)—C(11)	−91(1)	−95(1)
O(1)—C(9)—C(10)—C(16)	83(1)	83(1)
N(1)—C(9)—C(10)—C(11)	86(1)	82 (1)
N(1)—C(9)—C(10)—C(16)	−101(1)	−100(1)

In contrast to its (*R*) *syn* rotamer (PYRM), the carbonyl group of the title compound is directed towards the *B* side of the pyridine ring, and the carbonyl oxygen and *N*-methyl groups now have the *anti* conformation with torsion angles C(8)—N(1)—C(9)—O(1) of 180 (1) and 176·0 (9)° for molecules (I) and (II), respectively. In this respect the (*R*)- and (*S*)-compounds should be considered as diastereomeric rotamers.

The independent molecules each have three nearly planar fragments; the pyridinium, the *N,N*-dimethylcarbamoyl and phenyl groups for which the σ_{plane} values are 0·018, 0·017 and 0·019 Å [molecule (I)] and 0·017, 0·024 and 0·017 Å [molecule (II)] respectively. The bulky pyridinium and phenyl rings are attached to the planar *N,N*-dimethylcarbamoyl fragment in the *anti* position [torsion angles C(7)—N(1)—C(9)—C(10) −170·9 (8) and −177·2 (8)° for (I) and (II), respectively] which is much more favourable than the *syn* position in PYRM where this angle amounts to 0·6 (5)°. The differences between the conformations of the title compound and PYRM result in a stretched flattened form for the title compound, and a folded conformation with eclipsing C(7) and C(10) atoms for PYRM. Together with the distinct mode of preparation of the *syn* and *anti* rotamers, this conformational difference indicates that the *anti* rotamer is more stable. As may be expected there is a strong correspondence between the structures of the title compound and the 2,4-dimethylpyridinium chloride analogue mentioned in the *Introduction*. The two independent molecules in the latter structure also have a flattened form with torsion angles C(7)—N(1)—C(9)—C(10) of −171·8 (4) and −172·7 (3)°, respectively. As in PYRM there are short intermolecular contact distances between I^-

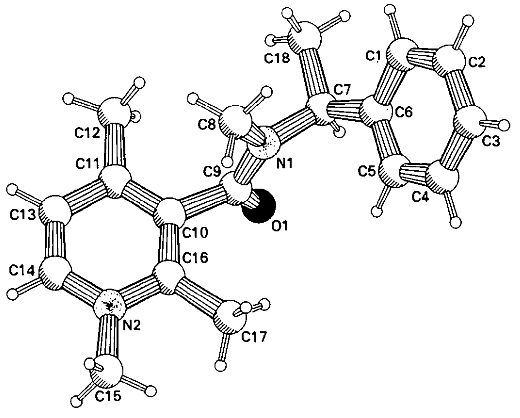

Fig. 1. Perspective view and atomic numbering of the title compound. Only one of the independent molecules is shown.

and N(2) of the pyridinium ring: for molecule (I) the N(2)···I(x, y, z) distance is 3·664 (8) Å, for molecule (II) N(2)···I($1 - x, y - \frac{1}{2}, \frac{1}{2} - z$) is 3·590 (8) Å.

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Structure of 6-*tert*-Butyl-3-cyclohexyl-3,4-dihydro-2-(4-tolyloxy)-2*H*-1,3,2-benzoxazaphosphorine 2-Oxide

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Abstract. $C_{24}H_{32}NO_3P$, $M_r = 413{\cdot}5$, monoclinic, $P2_1/c$, $a = 9{\cdot}045$ (4), $b = 20{\cdot}507$ (5), $c = 12{\cdot}571$ (2) Å, $\beta = 102{\cdot}60$ (1)°, $V = 2275{\cdot}6$ Å^3, $Z = 4$, $D_x = 1{\cdot}21$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 12{\cdot}4$ cm^{-1}, $F(000) = 888$, $T = 297$ K, $R = 0{\cdot}057$ for 3178 observed reflections. The oxazaphosphorine ring adopts a conformation midway between a half-chair and a sofa. The phosphoryl O(2) atom occupies an axial position. The *tert*-butyl and phenyl groups are *trans* to each other and occupy equatorial positions on the phosphorine ring. The cyclohexyl ring adopts a chair conformation with the N atom [N(5)] equatorial. The molecular geometry is normal. The molecular packing involves only van der Waals contacts.

Introduction. The title compound was prepared and supplied by M. S. R. Naidu & Nagaraju, Department of Chemistry, S. V. University, Tirupathi, India. This type of phosphorus heterocycle has been found to possess significant antitumour activity (Chugani Pharmaceutical Co., 1966; Arnold, Bourseaux & Brock, 1961; Friedman, Papanastassiou & Levi, 1963) and for this reason its X-ray structure is of great interest to our continuing investigations.

Experimental. Transparent, colourless needle-shaped crystals were grown from ethanol. A specimen with approximate dimensions 0·30 × 0·40 × 0·55 mm was selected. Unit-cell parameters and their e.s.d.'s derived from a least-squares treatment of 25 reflections ($35 < \theta < 45°$), intensity data were collected on an Enraf–Nonius CAD-4 single-crystal X-ray diffractometer, graphite-crystal-monochromatized Cu $K\alpha$ radiation, $\omega/2\theta$ scan mode. Intensities of reflections with $4 \leq 2\theta \leq 120°$ (h $-11 \rightarrow 10$, k $0 \rightarrow 24$, l $0 \rightarrow 15$) were measured. Two standard reflections for every 100 observations, <2% variation. Of the 3496 independent reflections collected, 3056 were judged significant [$I > 3\sigma(I)$]; intensities not corrected for absorption. Structure by direct methods using *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Positional and thermal parameters of non-H atoms refined by full-matrix least-squares using *SHELX*76 (Sheldrick, 1976). H atoms were located from a difference Fourier map and not refined. In the final stage of the refinement, weights were introduced resulting in $R = 0{\cdot}057$, $wR = 0{\cdot}063$, $w = 1/[\sigma^2(F) + 0{\cdot}00026F^2]$, $S = 2{\cdot}9$. $(\Delta/\sigma)_{\max} = 0{\cdot}008$. Final difference Fourier map featureless, with $\Delta\rho$ within $\pm 0{\cdot}13$ e Å^{-3}. Atomic scattering factors as provided in *SHELX*76. Computer programs: *PARST* (Nardelli, 1983) for

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