

highly favourable for H abstraction by the β -enone C (Jiang, Scheffer, Secco & Trotter, 1981), yet compound (1) is photo-inert in the solid state. The reason for the lack of reactivity is shown in Fig. 1. Abstraction of H(51) by C(3) is sterically hindered by the methyl group C(42) [H(421)⋯H(51) is 2.2 Å]. Abstraction of H(51) by the α -enone C [C(2)⋯H(51) is 2.84 Å] is not sterically hindered by the methyl H atoms on C(42); however, abstraction by C(2) would lead to an unstable radical on C(3), in an α -position relative to dimethylated C(4).

It is noteworthy that O abstraction of a β -H from C(8) cannot occur because of unsuitable geometry. Molecule (1) adopts a common conformation in which the bulkier substituent on C(8) assumes the pseudo-equatorial position, with H(8) in the pseudo-axial position, 3.92 Å from O(1).

Bond lengths and angles (Table 2) do not deviate significantly from accepted values. C(4)–C(4a) and C(4a)–C(8a) bonds are relatively long [1.554 (9) and 1.557 (8) Å respectively] because of steric effects due to the methyl groups on C(4) and the ester on C(8a).

A stereodiagram of compound (1) and its nearest lattice neighbours is shown in Fig. 2. The centrosymmetrically related molecules X and \bar{X} are ideally arranged to undergo [2 + 2] photodimerization. The potentially reactive enone double bonds are parallel, directly above one another and only slightly offset along the double-bond axis (0.52 Å); the centre-to-centre distance is 3.79 (1) Å. Crystal structures and photochemical studies of a series of *cis*-4a,5,8,8a-tetrahydro-1,4-naphthoquinone derivatives have revealed that the most favourable reaction involves [2 + 2] intermolecular cycloaddition in the solid state for those crystals with parallel C=C bonds in adjacent molecules with a separation of <4.1 Å (Trotter, 1983). Nevertheless, compound (1) is photochemically inert when irradiated in the solid state (Ariel *et al.*, 1984). Inspection of the packing diagram (Fig. 2) reveals the probable answer to the source of this remarkable lack of reactivity in the solid state. As the potentially reactive molecules X and \bar{X} start to move towards one

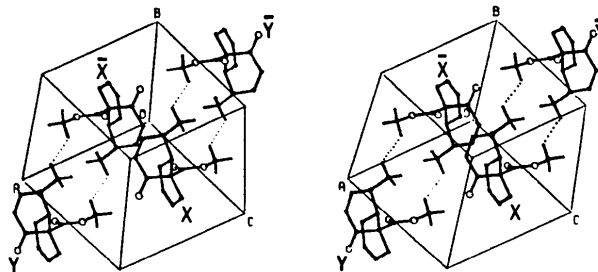


Fig. 2. Stereopacking diagram of (1). Molecules X and \bar{X} are related through a centre of symmetry. Translation of X along \mathbf{a} generates Y , and translation of \bar{X} along $-\mathbf{a}$ generates \bar{Y} . The dotted lines indicate the intermolecular H⋯H repulsion. For clarity of the picture certain atoms are omitted: methyl groups C(42) and C(81), and all H atoms, except those on C(41) and C(9).

another in the initial stages of [2 + 2] photocycloaddition, each experiences increasingly severe steric compression of two of its methyl groups (dotted lines). The key feature of this steric compression is that it is developed, not between the potential reactants X and \bar{X} , but between X and \bar{Y} and \bar{X} and Y . Thus molecules Y and \bar{Y} act as stationary impediments to the dimerization.

We thank Professor H.-J. Liu for the generous sample of compound (1), Professor J. R. Scheffer and Mr S. Askari for their collaborative studies on the solid-state photo-inertness, the Natural Sciences and Engineering Research Council of Canada for financial support, and the University of British Columbia Computing Centre for assistance.

References

- ARIEL, S., ASKARI, S., SCHEFFER, J. R., TROTTER, J. & WALSH, L. (1984). *J. Am. Chem. Soc.* In the press.
 CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* **A24**, 321–324.
 JIANG, Z. Q., SCHEFFER, J. R., SECCO, A. S. & TROTTER, J. (1981). *Tetrahedron Lett.* **22**, 891–894.
 SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
 STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
 TROTTER, J. (1983). *Acta Cryst.* **B39**, 373–381.

Acta Cryst. (1984). **C40**, 2086–2088

Structure of 4-*p*-Chlorophenyl-1-methyl-2,6-diphenylpyridinium Iodide, C₂₄H₁₉ClN⁺.I⁻

BY F. FLORENCIO AND S. GARCÍA-BLANCO

Departamento de Rayos X, Instituto Rocasolano, CSIC, Serrano 119, Madrid, Spain

(Received 8 June 1984; accepted 3 September 1984)

Abstract. $M_r = 438.733$, monoclinic, $P2_1/n$, $a = 1.35(2) \text{ g cm}^{-3}$, $\text{Mo K}\alpha$, $\lambda = 0.7107 \text{ \AA}$, $\mu = 10.404(9)$, $b = 10.745(2)$, $c = 19.982(3) \text{ \AA}$, $\beta = 16.781 \text{ cm}^{-1}$, $F(000) = 860$, room temperature, final $R = 0.073$ for 2675 reflections. The molecule shows an

0108-2701/84/122086-03\$01.50

© 1984 International Union of Crystallography

approximate *m* symmetry passing through Cl, N and the methyl C atom. The bond lengths and angles conform to the values given in the literature for similar compounds. The positive charge is localized on the nitrogen atom. The packing in the crystal is due to Coulombic and van der Waals forces.

Introduction. The structure analysis of the title compound is part of a study on pyridinium iodide derivatives with pharmacological properties carried out in this Laboratory. The present compound was obtained by Professor J. Alvarez-Buillas of the Facultad de Farmacia of Alcalá de Henares, Spain.

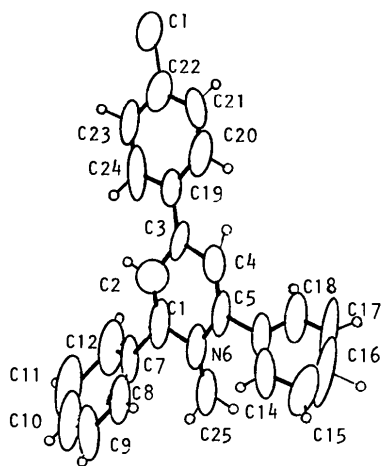


Fig. 1. The structure of the title compound with the atomic numbering.

Table 1. Atomic parameters for non-H atoms

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* \cos(\mathbf{a}_i, \mathbf{a}_j).$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{eq}</i> (Å ² × 10 ⁴)
I	0.10520 (9)	0.29614 (9)	0.91110 (5)	538 (3)
Cl	0.2639 (4)	0.9642 (3)	-0.05778 (20)	616 (14)
C(1)	0.4056 (11)	0.2987 (11)	0.1502 (5)	348 (35)
C(2)	0.3594 (11)	0.4024 (10)	0.1108 (6)	319 (36)
C(3)	0.4466 (10)	0.4968 (9)	0.1010 (5)	275 (32)
C(4)	0.5825 (13)	0.4811 (10)	0.1351 (6)	387 (42)
C(5)	0.6298 (10)	0.3759 (10)	0.1745 (5)	304 (34)
N(6)	0.5397 (8)	0.2846 (9)	0.1789 (4)	330 (29)
C(7)	0.3088 (10)	0.2007 (11)	0.1602 (6)	337 (34)
C(8)	0.3011 (12)	0.1651 (12)	0.2265 (6)	432 (42)
C(9)	0.2062 (14)	0.0783 (13)	0.2347 (8)	589 (58)
C(10)	0.1183 (13)	0.0261 (14)	0.1782 (10)	642 (63)
C(11)	0.1244 (14)	0.0602 (13)	0.1119 (9)	602 (57)
C(12)	0.2178 (13)	0.1478 (12)	0.1031 (7)	478 (46)
C(13)	0.7743 (11)	0.3632 (10)	0.2105 (6)	324 (37)
C(14)	0.8139 (12)	0.3485 (11)	0.2821 (6)	422 (43)
C(15)	0.9510 (15)	0.3446 (13)	0.3151 (7)	600 (53)
C(16)	1.0451 (13)	0.3470 (14)	0.2774 (9)	623 (58)
C(17)	1.0097 (11)	0.3649 (13)	0.2084 (8)	514 (50)
C(18)	0.8734 (12)	0.3733 (12)	0.1739 (6)	464 (45)
C(19)	0.3973 (11)	0.6075 (10)	0.0586 (6)	318 (35)
C(20)	0.4864 (12)	0.6757 (11)	0.0298 (5)	384 (40)
C(21)	0.4463 (11)	0.7819 (11)	-0.0076 (6)	378 (38)
C(22)	0.3148 (13)	0.8264 (10)	-0.0140 (6)	415 (41)
C(23)	0.2248 (13)	0.7607 (11)	0.0139 (7)	469 (48)
C(24)	0.2675 (12)	0.6519 (12)	0.0503 (7)	445 (45)
C(25)	0.5931 (12)	0.1615 (10)	0.2085 (6)	408 (41)

The crystal analysis of the title compound was undertaken to determine its molecular structure and to compare it with other compounds previously studied (Smith-Verdier, Florencio & García-Blanco, 1983).

Experimental. Enraf-Nonius CAD-4 automatic four-circle diffractometer, graphite-monochromated Mo K α , $\omega/2\theta$ scan mode. Crystal 0.10 × 0.15 × 0.20 mm. Lattice parameters measured by least squares from 25 reflections. 6171 independent reflections up to $\theta = 30^\circ$. Range of *hkl* -14,0,0 to 14,15,28. Two standard reflexions measured every 100 min showed no variation

Table 2. Bond lengths (Å), bond angles (°) and torsion angles (°)

Cl-C(22)	1.73 (1)	C(10)-C(11)	1.39 (3)
C(1)-C(2)	1.38 (2)	C(11)-C(12)	1.39 (2)
C(1)-N(6)	1.29 (1)	C(13)-C(14)	1.40 (2)
C(1)-C(7)	1.50 (2)	C(13)-C(18)	1.41 (2)
C(2)-C(3)	1.41 (2)	C(14)-C(15)	1.41 (2)
C(3)-C(4)	1.42 (2)	C(15)-C(16)	1.38 (2)
C(3)-C(19)	1.48 (1)	C(16)-C(17)	1.35 (2)
C(4)-C(5)	1.40 (1)	C(17)-C(18)	1.41 (2)
C(5)-N(6)	1.44 (1)	C(19)-C(20)	1.41 (2)
C(5)-C(13)	1.50 (1)	C(19)-C(24)	1.40 (2)
N(6)-C(25)	1.53 (1)	C(20)-C(21)	1.37 (2)
C(7)-C(8)	1.40 (2)	C(21)-C(22)	1.43 (2)
C(7)-C(12)	1.41 (2)	C(22)-C(23)	1.40 (2)
C(8)-C(9)	1.40 (2)	C(23)-C(24)	1.39 (2)
C(9)-C(10)	1.38 (2)		
N(6)-C(1)-C(7)	119.5 (10)	C(10)-C(11)-C(12)	119.9 (14)
C(2)-C(1)-C(7)	119.5 (10)	C(7)-C(12)-C(11)	121.1 (12)
C(2)-C(1)-N(6)	121.0 (11)	C(5)-C(13)-C(18)	121.3 (10)
C(1)-C(2)-C(3)	121.2 (10)	C(5)-C(13)-C(14)	120.3 (10)
C(2)-C(3)-C(19)	121.2 (10)	C(14)-C(13)-C(18)	118.3 (11)
C(2)-C(3)-C(4)	116.6 (9)	C(13)-C(14)-C(15)	119.0 (12)
C(4)-C(3)-C(19)	122.2 (10)	C(14)-C(15)-C(16)	121.2 (14)
C(3)-C(4)-C(5)	122.3 (11)	C(15)-C(16)-C(17)	120.8 (13)
C(4)-C(5)-C(13)	120.8 (10)	C(16)-C(17)-C(18)	119.3 (13)
C(4)-C(5)-N(6)	115.5 (10)	C(13)-C(18)-C(17)	121.2 (12)
N(6)-C(5)-C(13)	123.7 (9)	C(3)-C(19)-C(24)	121.8 (10)
C(1)-N(6)-C(5)	123.3 (9)	C(3)-C(19)-C(20)	118.9 (10)
C(5)-N(6)-C(25)	111.8 (9)	C(20)-C(19)-C(24)	119.2 (10)
C(1)-N(6)-C(25)	123.8 (9)	C(19)-C(20)-C(21)	119.3 (11)
C(1)-C(7)-C(12)	120.9 (10)	C(20)-C(21)-C(22)	118.6 (11)
C(1)-C(7)-C(8)	121.1 (10)	Cl-C(22)-C(21)	119.7 (9)
C(8)-C(7)-C(12)	118.0 (11)	C(21)-C(22)-C(23)	121.4 (11)
C(7)-C(8)-C(9)	120.3 (12)	Cl-C(22)-C(23)	118.9 (9)
C(8)-C(9)-C(10)	121.1 (14)	C(22)-C(23)-C(24)	118.5 (12)
C(9)-C(10)-C(11)	119.5 (14)	C(19)-C(24)-C(23)	121.3 (12)
N(6)-C(1)-C(7)-C(8)	57.7 (15)	C(13)-C(5)-N(6)-C(25)	-12.1 (15)
C(2)-C(1)-C(7)-C(8)	-123.6 (12)	C(1)-C(7)-C(12)-C(11)	-177.6 (12)
N(6)-C(1)-C(7)-C(12)	-126.2 (12)	C(1)-C(7)-C(8)-C(9)	176.9 (12)
C(2)-C(1)-C(7)-C(12)	52.5 (16)	C(8)-C(7)-C(12)-C(11)	-1.4 (18)
C(2)-C(1)-N(6)-C(25)	-166.9 (10)	C(12)-C(7)-C(8)-C(9)	0.7 (18)
C(7)-C(1)-N(6)-C(5)	-175.8 (10)	C(7)-C(8)-C(9)-C(10)	-0.1 (21)
C(2)-C(1)-N(6)-C(5)	5.5 (16)	C(8)-C(9)-C(10)-C(11)	0.2 (23)
N(6)-C(1)-C(2)-C(3)	-2.0 (16)	C(9)-C(10)-C(11)-C(12)	-0.9 (23)
C(7)-C(1)-C(2)-C(3)	179.3 (10)	C(10)-C(11)-C(12)-C(17)	-1.5 (21)
C(7)-C(1)-N(6)-C(25)	11.8 (15)	C(5)-C(13)-C(18)-C(17)	178.1 (11)
C(1)-C(2)-C(3)-C(4)	-1.8 (16)	C(5)-C(13)-C(14)-C(15)	-176.1 (11)
C(1)-C(2)-C(3)-C(19)	179.7 (10)	C(14)-C(13)-C(18)-C(17)	2.0 (18)
C(2)-C(3)-C(19)-C(20)	-157.7 (11)	C(18)-C(13)-C(14)-C(15)	0.1 (18)
C(2)-C(3)-C(19)-C(24)	27.0 (16)	C(13)-C(14)-C(15)-C(16)	-4.2 (20)
C(2)-C(3)-C(4)-C(5)	2.4 (16)	C(14)-C(15)-C(16)-C(17)	6.3 (23)
C(4)-C(3)-C(19)-C(20)	23.9 (16)	C(15)-C(16)-C(17)-C(18)	-4.1 (22)
C(4)-C(3)-C(19)-C(24)	-151.4 (12)	C(16)-C(17)-C(18)-C(13)	-0.1 (20)
C(19)-C(3)-C(4)-C(5)	-179.1 (10)	C(3)-C(19)-C(24)-C(23)	175.9 (11)
C(3)-C(4)-C(5)-N(6)	0.8 (17)	C(3)-C(19)-C(20)-C(21)	-177.6 (10)
C(3)-C(4)-C(5)-C(13)	-179.4 (10)	C(20)-C(19)-C(24)-C(23)	0.6 (18)
C(4)-C(5)-N(6)-C(1)	-4.8 (15)	C(24)-C(19)-C(20)-C(21)	-2.2 (17)
C(4)-C(5)-C(13)-C(14)	121.1 (13)	C(19)-C(20)-C(21)-C(22)	3.7 (17)
C(4)-C(5)-C(13)-C(18)	-54.9 (16)	C(20)-C(21)-C(22)-Cl	177.0 (9)
C(4)-C(5)-N(6)-C(25)	167.7 (10)	C(20)-C(21)-C(22)-C(23)	-3.7 (18)
C(13)-C(5)-N(6)-C(1)	175.4 (10)	C(21)-C(22)-C(23)-C(24)	2.1 (18)
N(6)-C(5)-C(13)-C(14)	-59.1 (15)	Cl-C(22)-C(23)-C(24)	-178.5 (10)
N(6)-C(5)-C(13)-C(18)	124.9 (12)	C(22)-C(23)-C(24)-C(19)	-0.6 (19)

in intensity. 2675 observed reflections with $I \geq 2\sigma(I)$. Structure solved by Patterson analysis, refined by full-matrix least squares, $\sum w(|F_o| - |F_c|)^2$ minimized, anisotropic thermal parameters for non-H atoms. All H atoms located in difference Fourier synthesis and positional parameters included in further refinement with fixed isotropic temperature factor. Final $R = 0.073$, $R_w = 0.080$; empirical weighting scheme giving no trends in $\langle w\Delta^2F \rangle$. No correction for absorption. No significant features in final ΔF synthesis. Max. $\Delta/\sigma = 0.06$. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Calculations performed with *XRAY70* (Stewart, Kundell & Baldwin, 1970), programs *PARST* (Nardelli, 1982) and *PESOS* (Martinez-Ripoll & Cano, 1975) on a Univac 1108 computer.

Discussion. The structure of the title compound with the atomic numbering is shown in Fig. 1 (*ORTEP*; Johnson, 1965). Positional parameters and the equivalent U values of the anisotropic temperature factor are given in Table 1. Bond lengths and angles and torsion angles are given in Table 2.*

Bond distances are in good agreement with those found in the literature and with other pyridinium iodides previously reported (Smith-Verdier, Florencio & García-Blanco, 1983).

* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and deviations from the molecular plane have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39705 (46 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The average torsion angles in the *A, B, C* and pyridine rings are 2.2 (18), 2.8 (20), 0.8 (20) and 2.9 (16)°, respectively. The torsion angles around the C(1)–C(7), C(3)–C(19) and C(5)–C(13) bonds are 55.1 (14), 25.5 (14) and 57.0 (14)°, respectively. Consequently, there is no electronic interaction in the molecule and the positive charge is localized on the N atom.

The environment of the I⁻ ion is approximately tetrahedral with I...N distances about 4.6 Å. The nearest contacts between the molecule and the unattached I⁻ ion are I...Cl (x, y, z) = 3.914 (4) Å; I...C(18) ($-x+1, -y+1, -z+1$) = 3.968 (13) Å.

The molecular symmetry approximates to an m plane defined by Cl, C(22), C(19), C(3), N(6) and C(25). The crystal packing is mainly determined by Coulombic and van der Waals forces. The intermolecular non-bonding distances correspond to standard van der Waals values.

References

- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press.
- JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee.
- MARTÍNEZ-RIPOLL, M. & CANO, F. H. (1975). *PESOS*. Instituto Rocasolano, CSIC, Madrid, Spain.
- NARDELLI, M. (1982). *PARST*. Instituto di Chimica Generale. Univ. of Parma, Italy.
- SMITH-VERDIER, P., FLORENCIO, F. & GARCÍA-BLANCO, S. (1983). 8th Eur. Crystallogr. Meet., 2–12 August 1983, Liège, Belgium, Abstract 2b, 16-P.
- STEWART, J. M., KUNDELL, F. A. & BALDWIN, J. C. (1970). The *XRAY70* system. Computer Science Center, Univ. of Maryland. College Park, Maryland.

Acta Cryst. (1984). **C40**, 2088–2091

Structure of 2-[4-(Acetylaminosulphonyl)phenylcarbamoyl]benzoic Acid (Phthalylsulacetamide), C₁₆H₁₄N₂O₆S

BY T. P. SINGH,* URMILA PATEL AND M. HARIDAS

Department of Physics, Sardar Patel University, Vallabh Vidyanagar 388 120, India

(Received 20 March 1984; accepted 27 July 1984)

Abstract. $M_r = 362.37$, monoclinic, $P2_1/c$, $a = 8.01$ (2), $b = 13.00$ (2), $c = 18.04$ (3) Å, $\beta = 111.6$ (5)°, $V = 1747$ (6) Å³, $Z = 4$, $D_m = 1.386$ (5), $D_x = 1.378$ (5) Mg m⁻³, $\lambda(\text{Cu K}\alpha) = 1.5418$ Å, $\mu = 1.98$ mm⁻¹, $F(000) = 816$, $T = 297$ K, final $R = 0.092$

* To whom correspondence should be addressed. Present address: Department of Biophysics, All India Institute of Medical Sciences, New Delhi-110029, India.

for 1803 observed reflections. The two planar benzene rings in the structure make an angle of 62.1 (5)° with each other. The molecules are arranged in the form of hydrogen-bonded loops. There are no intermolecular interactions in the interior of the loop. The density of crystals is lower than that observed in other sulphonamides. The molecule adopts a coiled conformation about the a axis.