

Fig. 2. A view down b showing the crystal packing of the title compound. Cl ions are marked with large circles and hydrogen bonds with thick lines. H atoms are omitted for clarity.

drawing showing the conformation of the cation and its atom-labelling system is presented in Fig. 1. A view of unit cell down the b axis is shown in Fig. 2.

Related literature. Crystal structures containing hexahydrocinchonine moieties have been reported by Golankiewicz, Gdaniec, Jaskólski & Kosturkiewicz (1981) and Gdaniec, Kosturkiewicz & Golankiewicz

(1987). The geometry of Cinchona alkaloids is discussed by Oleksyn (1987).

The work was supported by the project RP.II.13.2.13.(MEN).

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Acta Cryst. (1989). **C45**, 1658–1660

Stereochemical Studies of Oligomers. XXV.* *N*-(*o*-Tolyl)phthalimide

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(Received 10 May 1989; accepted 24 May 1989)

Abstract. $C_{15}H_{11}NO_2$, $M_r = 237.3$, monoclinic, $P2_1/c$, $a = 10.010$ (2), $b = 16.250$ (2), $c = 7.658$ (2) Å, $\beta = 104.73$ (3)°, $V = 1204.7$ Å³, $Z = 4$, $D_x = 1.31$ g cm⁻³, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu = 6.71$ cm⁻¹, $F(000) = 496$, room temperature, $R = 0.070$ for 888 observed reflections. Each individual ring is planar but the two rings of the phthalimide group are not perfectly coplanar, the dihedral angle between the mean ring planes being 1.2 (3)°. The O1 and O2 atoms deviate from the mean pyrrole ring by 0.019 (7) and 0.011 (6) Å. The geometrical values for the methyl-substituted ring are normal, the mean C—C distance and C—C—C internal angle being 1.382 (7) Å and

120.0 (9)° respectively. This ring forms a dihedral angle of 70.7 (2)° with the mean phthalimide moiety. The molecules are joined together in the crystals by van der Waals forces only.

Experimental. Colourless prismatic crystal, recrystallized by slow evaporation from methanol, of $0.4 \times 0.5 \times 0.8$ mm used for data collection on a Siemens AED single-crystal diffractometer equipped with an IBM PS2/30 personal computer, in the $3 \leq \theta \leq 70^\circ$ range, using the nickel-filtered Cu $K\alpha$ radiation through a modified version (Belletti, Cantoni & Pasquellini, 1988) of the Lehmann & Larsen (1974) procedure. 28 reflections employed to obtain accurate cell dimensions by least-squares fit. The range in

* Part XXIV: Bocelli, Rizzoli & Ori (1989).

Table 1. Atomic fractional coordinates ($\times 10^4$) and U_{eq} (Hamilton, 1959) ($\text{\AA}^2 \times 10^4$)

	x	y	z	U_{eq}
O1	111 (6)	326 (3)	2602 (9)	758 (26)
O2	4082 (6)	1534 (3)	1979 (8)	595 (24)
N1	2242 (6)	728 (3)	2304 (8)	450 (21)
C1	899 (9)	862 (4)	2463 (12)	536 (34)
C2	746 (8)	1773 (4)	2533 (11)	463 (29)
C3	-364 (9)	2237 (5)	2695 (11)	530 (35)
C4	-195 (11)	3097 (5)	2747 (13)	632 (37)
C5	970 (9)	3442 (5)	2478 (12)	587 (34)
C6	2101 (9)	2970 (5)	2315 (11)	526 (34)
C7	1925 (8)	2126 (4)	2305 (10)	462 (31)
C8	2931 (8)	1477 (4)	2170 (9)	418 (25)
C9	2873 (8)	-77 (4)	2306 (9)	441 (28)
C10	3243 (7)	-523 (4)	3905 (10)	447 (29)
C11	3806 (9)	-1283 (5)	3826 (12)	545 (33)
C12	4041 (10)	-1594 (5)	2206 (14)	659 (40)
C13	3697 (10)	-1133 (5)	671 (14)	674 (41)
C14	3117 (10)	-368 (5)	709 (11)	644 (38)
C15	3031 (13)	-206 (7)	5643 (13)	620 (42)

the Miller indices hkl was $-12 \leq h \leq 11$, $0 \leq k \leq 19$, $0 \leq l \leq 9$. One check reflection measured every 50 reflections, with a drop of about 22% in intensity, indicated significant crystal decomposition. The intensities were corrected for this decay, for Lorentz and polarization effects but not for absorption. 2478 reflections recorded of which 2267 unique ($R_{int} = 0.013$), 910 considered observed [$I \geq 3.2\sigma(I)$] and used in the refinement. Direct methods (SHELX76; Sheldrick, 1976); full-matrix anisotropic least squares. Most H atoms located in a subsequent ΔF map, the remainder placed in their theoretical positions and all refined isotropically. In the last cycles 22 reflections having large ΔF values were excluded from the refinement, $\sum w\Delta F^2$ minimized, unit weights, $R = 0.070$, $(\Delta/\sigma)_{max} = 0.09$, the final difference electron density map shows peaks from 0.28 to $-0.25 e \text{\AA}^{-3}$. Atomic scattering factors were from SHELX76.

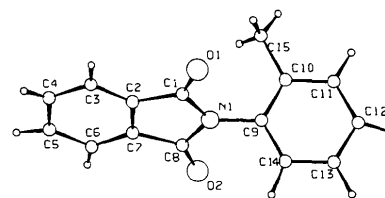
All the calculations were performed on an IBM PS2/80 personal computer with the CRYSRULER package (Rizzoli, Sangermano, Calestani & Andretti, 1987). Atomic parameters are given in Table 1; Table 2 reports bond distances and angles for the molecule which is illustrated in Fig. 1.*

Related literature. The geometry of the phthalimide fragment agrees with that found in phthalimide (Matzat, 1972) and in some of its derivatives (Voliotis, Arrieta & Germain, 1984; Magomedova, Neigauz, Zavodnik & Bel'skii, 1981). The value of the angle between the mean phthalimide group and

* 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 51930 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Bond distances (\AA) and angles ($^\circ$)

O1—C1	1.198 (10)	C5—C6	1.399 (13)
O2—C8	1.201 (11)	C6—C7	1.383 (10)
N1—C1	1.397 (11)	C7—C8	1.480 (11)
N1—C8	1.416 (9)	C9—C10	1.390 (10)
N1—C9	1.452 (9)	C9—C14	1.390 (12)
C1—C2	1.491 (9)	C10—C11	1.365 (11)
C2—C3	1.374 (12)	C10—C15	1.492 (14)
C2—C7	1.363 (12)	C11—C12	1.414 (15)
C3—C4	1.407 (12)	C12—C13	1.362 (14)
C4—C5	1.356 (15)	C13—C14	1.375 (12)
C8—N1—C9	123.7 (6)	C2—C7—C8	109.6 (6)
C1—N1—C9	124.6 (5)	N1—C8—C7	104.8 (6)
C1—N1—C8	111.7 (5)	O2—C8—C7	130.1 (6)
O1—C1—N1	124.3 (6)	O2—C8—N1	125.1 (6)
N1—C1—C2	105.5 (6)	N1—C9—C14	118.6 (6)
O1—C1—C2	130.0 (8)	N1—C9—C10	119.3 (6)
C1—C2—C7	108.3 (6)	C10—C9—C14	122.1 (6)
C1—C2—C3	129.9 (7)	C9—C10—C15	122.7 (7)
C3—C2—C7	121.8 (6)	C9—C10—C11	116.9 (6)
C2—C3—C4	117.0 (8)	C11—C10—C15	120.4 (7)
C3—C4—C5	120.5 (8)	C10—C11—C12	121.7 (7)
C4—C5—C6	122.3 (7)	C11—C12—C13	119.9 (8)
C5—C6—C7	116.1 (8)	C12—C13—C14	119.6 (9)
C2—C7—C6	122.0 (7)	C9—C14—C13	119.7 (7)
C6—C7—C8	128.3 (7)		

Fig. 1. Atom-numbering scheme of *N*-(*o*-tolyl)phthalimide.

the aromatic substituent [$70.7 (2)^\circ$], which is larger than the value observed in the *m*-substituted analogous derivative [$54.3 (2)^\circ$; Chandrasekhar, Vasantha Pattabhi & Swaminathan, 1983], agrees with that [$71.0 (2)^\circ$] found in *N*-(2,4,6-trimethylphenyl)phthalimide (Voliotis *et al.*, 1984), confirming the influence of *o*-substituents on the angle between the two parts of these molecules and justifying the weak π -conjugation between the N atom and the tolyl ring.

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Acta Cryst. (1989). **C45**, 1660–1661

Stereochemical Studies of Oligomers. XXVI.* Bis[4-(4-aminophenoxy)phenyl]-dimethylmethane

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(Received 10 May 1989; accepted 24 May 1989)

Abstract. $C_{27}H_{26}N_2O_2$, $M_r = 410.5$, monoclinic, Pc , $a = 15.138$ (2), $b = 7.269$ (3), $c = 10.386$ (2) Å, $\beta = 99.60$ (4)°, $V = 1126.9$ Å³, $Z = 2$, $D_x = 1.21$ g cm⁻³, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu = 5.69$ cm⁻¹, $F(000) = 436$, room temperature, final $R = 0.075$ for 1349 observed reflections. As expected the four rings C4...C9 (*A*), C10...C15 (*B*), C16...C21 (*C*) and C22...C27 (*D*) are planar and their reciprocal dihedral angles are $A-B = 103.3$ (3), $A-C = 91.6$ (2), $A-D = 7.6$ (2), $B-C = 11.8$ (3), $B-D = 110.4$ (3) and $C-D = 98.8$ (2)°.

Experimental. Colourless prismatic crystal $0.4 \times 0.5 \times 0.8$ mm, Siemens AED single-crystal diffractom-

eter equipped with an IBM PS2/30 personal computer, nickel-filtered Cu $K\alpha$ radiation, intensities measured with a modified version (Belletti, Cantoni & Pasquinelli, 1988) of the Lehmann & Larsen (1974) procedure, $3 \leq \theta \leq 70^\circ$ range, 26 reflections with $11.37 \leq \theta \leq 44.15^\circ$ employed for refinement of lattice dimensions, index range $-18 \leq h \leq 18$, $0 \leq k \leq 8$, $0 \leq l \leq 12$, significant crystal decomposition revealed by a decrease in intensity of about 17% of one check reflection measured every 50, intensities

* Part XXV: Bocelli & Cantoni (1989).

Table 1. Atomic fractional coordinates ($\times 10^4$) and U_{eq} ($\times 10^4$ Å²)

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq} *
O1	8719 (4)	-76 (9)	1648 (6)	727 (22)
O2	2955 (4)	4434 (9)	210 (6)	782 (23)
N1	10257 (7)	-7137 (17)	2296 (14)	1105 (46)
N2	1450 (6)	11469 (12)	228 (13)	953 (43)
C1	5876 (11)	2469 (13)	4217 (11)	625 (29)
C2	6254 (9)	4052 (22)	5078 (12)	1001 (56)
C3	5504 (8)	1013 (19)	5073 (12)	859 (45)
C4	6622 (5)	1659 (12)	3559 (8)	578 (28)
C5	6952 (5)	-99 (13)	3717 (10)	591 (31)
C6	7650 (6)	-771 (14)	3120 (9)	686 (35)
C7	8014 (5)	351 (12)	2303 (8)	580 (27)
C8	7687 (6)	2150 (13)	2096 (10)	692 (34)
C9	7013 (6)	2764 (13)	2722 (9)	654 (33)
C10	9088 (6)	-1855 (13)	1840 (9)	663 (33)
C11	9669 (7)	-2232 (15)	2942 (10)	774 (40)
C12	10086 (7)	-3961 (16)	3103 (13)	819 (43)
C13	9863 (6)	-5266 (13)	2146 (11)	693 (37)
C14	9288 (6)	-4905 (15)	1055 (12)	720 (40)
C15	8886 (7)	-3147 (16)	860 (11)	822 (43)
C16	5102 (5)	3095 (11)	3157 (8)	619 (27)
C17	4700 (6)	1848 (13)	2242 (10)	686 (33)
C18	3990 (6)	2324 (14)	1284 (12)	777 (38)
C19	3673 (5)	4099 (11)	1202 (8)	602 (27)
C20	4060 (6)	5361 (12)	2114 (9)	718 (32)
C21	4779 (4)	4916 (10)	3083 (8)	510 (23)
C22	2576 (5)	6205 (11)	187 (8)	635 (31)
C23	1977 (6)	6545 (12)	1024 (10)	460 (33)
C24	1598 (7)	8274 (12)	964 (11)	773 (36)
C25	1824 (5)	9691 (12)	198 (9)	687 (34)
C26	2380 (6)	9205 (17)	-676 (9)	778 (38)
C27	2779 (5)	7474 (12)	-666 (9)	658 (29)

* Hamilton (1959).

Table 2. Bond distances (Å) and angles (°)

O1—C7	1.393 (11)	C10—C15	1.381 (15)
O1—C10	1.410 (11)	C11—C12	1.403 (16)
O2—C19	1.389 (9)	C12—C13	1.375 (16)
O2—C22	1.408 (10)	C13—C14	1.335 (14)
N1—C13	1.482 (15)	C14—C15	1.415 (16)
N2—C25	1.413 (12)	C16—C17	1.379 (12)
C1—C2	1.510 (16)	C16—C21	1.409 (11)
C1—C3	1.548 (15)	C17—C18	1.381 (13)
C1—C4	1.532 (9)	C18—C19	1.374 (13)
C1—C16	1.537 (8)	C19—C20	1.377 (12)
C4—C5	1.372 (13)	C20—C21	1.392 (11)
C4—C9	1.387 (13)	C22—C23	1.379 (14)
C5—C6	1.399 (14)	C22—C27	1.349 (12)
C6—C7	1.358 (13)	C23—C24	1.379 (13)
C7—C8	1.402 (13)	C24—C25	1.379 (14)
C8—C9	1.373 (14)	C25—C26	1.383 (14)
C10—C11	1.350 (13)	C26—C27	1.395 (15)
C7—O1—C10	116.9 (6)	C12—C13—C14	121.6 (9)
C19—O2—C22	116.1 (6)	N1—C13—C14	117.6 (9)
C4—C1—C16	109.0 (4)	C13—C14—C15	120.2 (10)
C3—C1—C16	107.9 (5)	C10—C15—C14	118.1 (9)
C3—C1—C4	111.1 (6)	C1—C16—C21	122.2 (6)
C2—C1—C16	111.5 (6)	C1—C16—C17	119.7 (6)
C2—C1—C4	108.8 (6)	C17—C16—C21	118.1 (7)
C2—C1—C3	108.6 (6)	C16—C17—C18	122.2 (8)
C1—C4—C9	118.7 (6)	C17—C18—C19	120.1 (9)
C1—C4—C5	125.8 (6)	O2—C19—C18	115.6 (7)
C5—C4—C9	115.4 (7)	C18—C19—C20	118.6 (8)
C4—C5—C6	124.0 (8)	O2—C19—C20	125.7 (7)
C5—C6—C7	118.8 (8)	C19—C20—C21	122.3 (7)
O1—C7—C6	126.5 (7)	C16—C21—C20	118.7 (7)
C6—C7—C8	119.1 (8)	O2—C22—C27	120.0 (7)
O1—C7—C8	114.4 (7)	O2—C22—C23	117.5 (7)
C7—C8—C9	120.1 (8)	C23—C22—C27	122.5 (7)
C4—C9—C8	122.5 (8)	C22—C23—C24	116.6 (8)
O1—C10—C15	119.1 (8)	C23—C24—C25	124.4 (8)
O1—C10—C11	119.5 (8)	N2—C25—C24	122.1 (8)
C11—C10—C15	121.3 (9)	C24—C25—C26	115.1 (8)
C10—C11—C12	119.9 (9)	N2—C25—C26	122.6 (8)
C11—C12—C13	118.8 (10)	C25—C26—C27	122.4 (8)
N1—C13—C12	120.8 (10)	C22—C27—C26	118.4 (8)