C4	0.28810 (15)	−0.08968 (12)	0.33002 (13)	0.0223 (3)
C5	0.1769 (2)	−0.03551 (12)	0.39276 (12)	0.0226 (3)
C6	0.06617 (15)	0.00934 (11)	0.31501 (12)	0.0196 (3)
C7	−0.02465 (14)	−0.07047 (12)	0.25265 (13)	0.0198 (3)
C8	0.2040 (2)	−0.20865 (13)	0.03576 (14)	0.0263 (3)
O9	0.07558 (12)	−0.23217 (10)	−0.01145 (10)	0.0290 (3)
C10	−0.0231 (2)	−0.18267 (13)	0.06005 (13)	0.0236 (3)
C11	0.2361 (2)	0.05157 (15)	0.46984 (15)	0.0314 (4)
O12	0.12095 (11)	0.08252 (8)	0.23336 (10)	0.0215 (2)
C13	−0.1339 (2)	−0.00990 (14)	0.1889 (2)	0.0292 (3)
O14	0.05349 (12)	−0.25180 (8)	0.23963 (10)	0.0223 (2)
C1*S*	0.3996 (2)	0.1480 (2)	0.1667 (2)	0.0335 (4)
Cl1 †	0.4220 (6)	0.1114 (12)	0.0217 (5)	0.079 (2)
Cl2 †	0.5430 (9)	0.1095 (8)	0.2509 (10)	0.0521 (13)
Cl3 †	0.3618 (5)	0.2738 (4)	0.1917 (10)	0.0654 (12)
Cl1′ ‡	0.4096 (3)	0.1599 (6)	0.0084 (2)	0.0563 (10)
Cl2′ ‡	0.5473 (8)	0.1078 (7)	0.2249 (5)	0.0418 (6)
Cl3′ ‡	0.3601 (4)	0.2787 (3)	0.2151 (3)	0.0418 (6)

† Occupancy = 0.47 (2).
‡ Occupancy = 0.53 (2).

Table 2. *Selected geometric parameters* (Å, °)

C1—O14	1.443 (2)	C5—C6	1.533 (2)
C1—C10	1.529 (2)	C5—C11	1.533 (2)
C1—C2	1.535 (2)	C6—O12	1.430 (2)
C1—C7	1.550 (2)	C6—C7	1.541 (2)
C2—C3	1.500 (2)	C7—C13	1.528 (2)
C2—C8	1.527 (2)	C8—O9	1.435 (2)
C3—C4	1.327 (2)	O9—C10	1.437 (2)
C4—C5	1.504 (2)		
O14—C1—C10	104.23 (11)	C4—C5—C11	108.19 (14)
O14—C1—C2	109.00 (11)	C6—C5—C11	109.78 (13)
C10—C1—C2	100.11 (11)	O12—C6—C5	109.32 (12)
O14—C1—C7	109.89 (11)	O12—C6—C7	111.58 (12)
C10—C1—C7	116.06 (12)	C5—C6—C7	116.68 (12)
C2—C1—C7	116.50 (11)	C13—C7—C6	108.09 (12)
C3—C2—C8	111.77 (12)	C13—C7—C1	114.63 (13)
C3—C2—C1	116.01 (12)	C6—C7—C1	115.92 (12)
C8—C2—C1	101.62 (12)	O9—C8—C2	106.23 (12)
C4—C3—C2	127.19 (14)	C8—O9—C10	109.23 (11)
C3—C4—C5	129.36 (14)	O9—C10—C1	106.11 (12)
C4—C5—C6	116.54 (12)		

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O12—H12···O14^{i}	0.80 (2)	1.98 (2)	2.774 (2)	170 (2)
O14—H14···O9^{ii}	0.77 (2)	2.13 (2)	2.837 (2)	153 (2)
C1*S*—H1*S*···O12	0.90 (2)	2.21 (2)	3.043 (2)	153 (2)

Symmetry codes: (i) $-x, \frac{1}{2}+y, \frac{1}{2}-z$; (ii) $x, -\frac{1}{2}-y, \frac{1}{2}+z$.

The $CDCl_3$ molecule is rotationally disordered about the C—D bond axis by about 12°. Cl atoms are disordered over two sites with relative Cl/Cl′ occupancies of 0.47/0.53. The refined C—H distances range between 0.90 (2) and 1.01 (2) Å.

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *XS* in *SHELXTL/PC* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *XP* in *SHELXTL/PC*. Software used to prepare material for publication: *SHELXL*93.

This research was supported by NSERC Canada, the A. P. Sloan Foundation, the E. W. R. Steacie Memorial Fund, Bio-Mega/Boehringer Ingelheim Inc., the Eli Lilly Grantee Program, the Petroleum Research Fund administered by the American Chemical Society, and the University of Toronto.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CR1194). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Ashcroft, M. R. & Hoffman, H. M. R. (1978). *Org. Synth.* **58**, 17–24.
Broka, C. A., Lee, W. J. & Shen, T. (1988). *J. Org. Chem.* **53**, 1336–1388.
Broka, C. A. & Shen, T. (1989). *J. Am. Chem. Soc.* **111**, 2981–2984.
Chiu, P. (1994). PhD thesis. *Ring-Opening Reactions of Oxabicyclic Compounds: Unsymmetrical Substrates and Reduction*, p. 76. Univ. of Toronto, Canada.
Clarke, R. D. & Heathcock, C. H. (1976). *J. Org. Chem.* **41**, 636–643.
Ladd, M. F. C. & Palmer, R. A. (1985). *Structure Determination by X-ray Crystallography*, 2nd ed., pp. 352–354. New York: Plenum Press.
Lautens, M. & Kumanovic, S. (1995). *J. Am. Chem. Soc.* **117**, 1954–1964.
Rigby, J. H. (1988). *Studies in Natural Products Chemistry*, Vol. 1, edited by Atta-ur-Rahman, pp. 545–576. Amsterdam: Elsevier.
Sato, T. & Noyori, R. (1978). *Bull. Chem. Soc. Jpn*, **51**, 2745–2746.
Seyferth, D. & Andrews, S. B. (1971). *J. Organomet. Chem.* **30**, 151–166.
Sheldrick, G. M. (1990). *SHELXTL/PC Users Manual*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1993). *SHELXL*93. *Program for the Refinement of Crystal Structures*. Univ. of Göttingen, Germany.
Siemens (1994). *XSCANS. X-ray Single Crystal Analysis System*. Version 2.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Still, W. C. (1978). *J. Am. Chem. Soc.* **100**, 1481–1486.
Still, W. C., McDonald, J. H., Collum, D. B. & Mitra, A. (1979). *Tetrahedron Lett.* pp. 593–594.
Still, W. C. & Mitra, A. (1978). *J. Am. Chem. Soc.* **100**, 1927–1928.

Acta Cryst. (1995). C**51**, 2372–2374

N-(*p*-Tolyl)phthalimide

GABRIELE BOCELLI AND ANDREA CANTONI

Centro di Studio per la Strutturistica Diffrattometrica del CNR, Viale delle Scienze, I-43100 Parma, Italy

PIETRO COZZINI

Dipartimento di Chimica Generale e Inorganica, Chimica Analitica e Chimica Fisica, Università di Parma, Viale delle Scienze, I-43100 Parma, Italy

(*Received* 12 *April* 1995; *accepted* 30 *May* 1995)

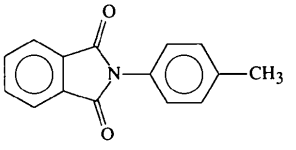
Abstract

The title compound, $C_{15}H_{11}NO_2$, was structurally analyzed in order to clarify the influence of the substituents on the conformational change, which has an

effect on the synthesis process. The *p*-tolyl and phthalimide groups are folded towards each other, making an angle of 55.7 (1)°.

Comment

N-Substituted phthalimides containing electron-donor and electron-acceptor fragments simultaneously are an important class of aromatic derivatives used in the synthesis of heat-resistant polymers (Magomedova, Dzyabchenko, Zavodnik & Bel'skii, 1980; Magomedova, Neigauz, Zavodnik & Bel'skii, 1981), many biological derivatives (Ribar, Stankovic, Herak, Halasi & Djuric, 1974; Ribar, Stankovic & Halasi, 1976; Geita, Dalberga, Medne & Aren, 1970; Benetollo, Del Pra, Orsini & Baiocchi, 1993) and inclusion compounds (Herbstein & Kaftory, 1981; Kaftory, 1978). Their geometrical features play an important role in the synthesis process and the X-ray analysis of the title compound, (I), constitutes a further step in improving the understanding of their structural characteristics, the effects of the substituents and the conformational changes in this family of compounds.



(I)

Fig. 1 shows a perspective view of the title molecule. The phthalimide moiety is not exactly planar, the dihedral angle between the mean planes of the two individual rings being 2.0 (1)°. The *p*-tolyl and phthalimide groups are folded towards each other, making an angle of 55.7 (1)°, which is in good agreement with the corresponding angles found in other *N*-(*p*-substituted phenyl)phthalimides, for example, 58° for bromine-substituted (Ribar *et al.*, 1976), 56° for iodine-substituted (Ribar *et al.*, 1974), 64.5° for dimethylamino-substituted (Magomedova *et al.*, 1980) and 58.4° for an unsubstituted aromatic ring (Magomedova *et al.*, 1981). The shortening of the N—C bonds with respect to the normal C_{sp^3}—N_{sp^3} length (1.47 Å) and the sum of bond angles about the N1 atom of 360°, both indicate the inclusion of the unshared pair of electrons on the N atom in the conjugated bicyclic system of the phthalimide acceptor moiety.

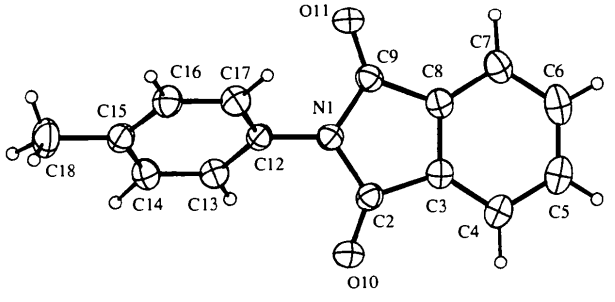


Fig. 1. *ORTEP* plot (Johnson, 1965) of the title compound showing 30% probability displacement ellipsoids.

The molecules in the crystal are stacked perpendicular to the *a* axis (Fig. 2).

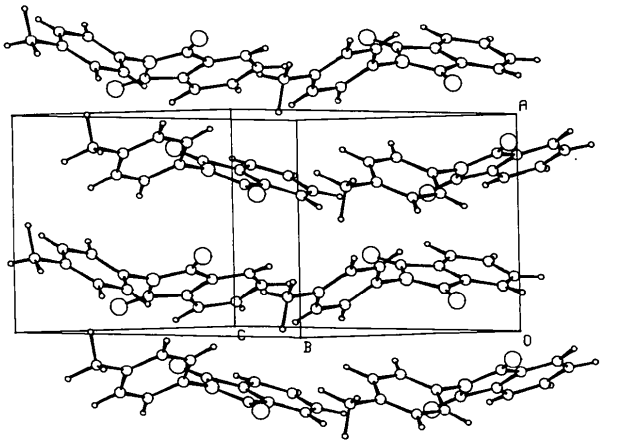


Fig. 2. Packing of the title compound.

Experimental

The title compound was prepared according to the procedures of Grenier-Loustalot (1995).

Crystal data

$C_{15}H_{11}NO_2$
M_r = 237.25
Orthorhombic
$Pna2_1$
a = 7.625 (3) Å
b = 11.262 (2) Å
c = 13.883 (2) Å
V = 1192.2 (5) Å^3
Z = 4
D_x = 1.322 Mg m^{-3}
Cu $K\alpha$ radiation
λ = 1.54178 Å
Cell parameters from 26 reflections
θ = 11.9–41.3°
μ = 0.717 mm^{-1}
T = 293 (2) K
Prism
0.64 × 0.52 × 0.33 mm
Colorless

Data collection

Siemens AED diffractometer
Lehman & Larsen (1974) scans
Absorption correction: none
1350 measured reflections
1185 independent reflections
1129 observed reflections [$I > 2\sigma(I)$]
θ_{max} = 70.07°
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 13$
$l = 0 \rightarrow 16$
1 standard reflection monitored every 100 reflections
intensity decay: none

Refinement

Refinement on F^2
$R(F)$ = 0.0304
$wR(F^2)$ = 0.0806
S = 1.027
1182 reflections
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0251 (20)

207 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0562P)^2 + 0.0750P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = -0.544$
$\Delta\rho_{max} = 0.10$ e Å^{-3}
$\Delta\rho_{min} = -0.09$ e Å^{-3}
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: Flack (1983) parameter = −0.17 (33)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
N1	0.7501 (2)	0.12066 (13)	0.1047	0.0470 (4)
C2	0.8198 (3)	0.0273 (2)	0.0498 (2)	0.0501 (5)
C3	0.8165 (2)	0.0693 (2)	−0.0520 (2)	0.0502 (5)
C4	0.8669 (3)	0.0120 (2)	−0.1360 (2)	0.0618 (6)
C5	0.8417 (4)	0.0734 (3)	−0.2222 (2)	0.0730 (7)
C6	0.7692 (4)	0.1856 (3)	−0.2241 (2)	0.0711 (7)
C7	0.7196 (3)	0.2430 (2)	−0.1388 (2)	0.0616 (6)
C8	0.7459 (2)	0.1825 (2)	−0.0539 (2)	0.0499 (5)
C9	0.7058 (3)	0.2187 (2)	0.0469 (2)	0.0487 (5)
O10	0.8679 (2)	−0.06734 (13)	0.0813 (2)	0.0680 (5)
O11	0.6453 (2)	0.31108 (14)	0.0756 (2)	0.0677 (5)
C12	0.7293 (3)	0.1184 (2)	0.2071 (2)	0.0454 (4)
C13	0.6327 (3)	0.0286 (2)	0.2498 (2)	0.0545 (5)
C14	0.6110 (3)	0.0271 (2)	0.3484 (2)	0.0592 (5)
C15	0.6835 (3)	0.1149 (2)	0.4067 (2)	0.0549 (5)
C16	0.7780 (3)	0.2047 (2)	0.3621 (2)	0.0576 (5)
C17	0.8021 (3)	0.2071 (2)	0.2635 (2)	0.0533 (5)
C18	0.6605 (5)	0.1129 (4)	0.5151 (2)	0.0842 (9)

Table 2. *Selected geometric parameters* (Å, °)

N1—C2	1.404 (3)	C7—C8	1.376 (3)
N1—C9	1.406 (3)	C8—C9	1.489 (3)
N1—C12	1.431 (2)	C9—O11	1.206 (3)
C2—O10	1.209 (3)	C12—C13	1.384 (3)
C2—C3	1.490 (3)	C12—C17	1.385 (3)
C3—C8	1.385 (3)	C13—C14	1.378 (3)
C3—C4	1.387 (3)	C14—C15	1.392 (3)
C4—C5	1.395 (4)	C15—C16	1.388 (3)
C5—C6	1.379 (5)	C15—C18	1.514 (3)
C6—C7	1.402 (4)	C16—C17	1.382 (3)
C2—N1—C9	111.6 (2)	C3—C8—C9	108.3 (2)
C2—N1—C12	124.7 (2)	O11—C9—N1	125.5 (2)
C9—N1—C12	123.7 (2)	O11—C9—C8	128.7 (2)
O10—C2—N1	125.4 (2)	N1—C9—C8	105.8 (2)
O10—C2—C3	128.9 (2)	C13—C12—C17	119.9 (2)
N1—C2—C3	105.7 (2)	C13—C12—N1	119.9 (2)
C8—C3—C4	121.4 (2)	C17—C12—N1	120.2 (2)
C8—C3—C2	108.5 (2)	C14—C13—C12	119.9 (2)
C4—C3—C2	130.2 (2)	C13—C14—C15	121.4 (2)
C3—C4—C5	116.9 (2)	C16—C15—C14	117.6 (2)
C6—C5—C4	121.7 (2)	C16—C15—C18	120.9 (3)
C5—C6—C7	120.9 (3)	C14—C15—C18	121.4 (3)
C8—C7—C6	117.2 (2)	C17—C16—C15	121.7 (2)
C7—C8—C3	121.9 (2)	C12—C17—C16	119.5 (2)
C7—C8—C9	129.8 (2)		

Data were corrected for Lorentz and polarization effects, but not for absorption. All the calculations were performed on a Dell 466De computer with the *CRYSRULER* (Rizzoli, Sangermano, Calestani & Andreetti, 1987) package.

Data collection: Belletti, Cantoni & Pasquinelli (1992). Cell refinement: Belletti, Cantoni & Pasquinelli (1992). Data reduction: Belletti, Cantoni & Pasquinelli (1992). Program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP* (Johnson, 1965).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1133). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Altomare, A. M., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435–436.
Belletti, D., Cantoni, A. & Pasquinelli, G. (1992). *Gestione On Line di Diffrattometro a Cristallo Singolo Siemens AED con Personal Computer*. Internal report 1/92. Centro di Studio per la Strutturistica Diffrattometrica del CNR, Parma, Italy.
Benetollo, F., Del Pra, A., Orsini, F. & Baiocchi, L. (1993). *J. Crystallogr. Spectrosc. Res.* **23**, 987–992.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881
Geita, L. S., Dalberga, I. E., Medne, K. K. & Aren, A. K. (1970). *Izv. Akad. Nauk. Latv. SSR Ser. Khim.* pp. 572–576.
Grenier-Loustalot, M. F. (1995). Private communication.
Herbstein, F. H. & Kaftory, M. (1981). *Z. Kristallogr.* **157**, 1–25.
Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
Kaftory, M. (1978). *Acta Cryst.* B**34**, 471–475.
Lehmann, M. S. & Larsen, F. K. (1974). *Acta Cryst.* A**30**, 580–589.
Magomedova, N. S., Dzyabchenko, A. V., Zavodnik, V. E. & Bel'skii, V. K. (1980). *Cryst. Struct. Commun.* **9**, 713–715.
Magomedova, N. S., Neigauz, M. G., Zavodnik, V. E. & Bel'skii, V. K. (1981). *Kristallografiya*, **26**, 841–844.
Ribar, B., Stankovic, S. & Halasi, R. (1976). *Cryst. Struct. Commun.* **5**, 919–922.
Ribar, B., Stankovic, S., Herak, R., Halasi, R. & Djuric, S. (1974). *Cryst. Struct. Commun.* **3**, 669–672.
Rizzoli, C., Sangermano, V., Calestani, G. & Andreetti, G. D. (1987). *J. Appl. Cryst.* **20**, 436–439.
Sheldrick, G. M. (1993). *SHELXL*93. *Program for the Refinement of Crystal Structures*. Univ. of Göttingen, Germany.

Acta Cryst. (1995). C**51**, 2374–2376

Dimethyl 4,4-Bis(ethoxycarbonyl)-1,2-cyclopentanediylidenediacetate

GABRIELE BOCELLI AND ANDREA CANTONI

Centro di Studio per la Strutturistica Diffrattometrica del CNR, Viale delle Scienze, I-43100 Parma, Italy

MIRCO COSTA

Dipartimento di Chimica Organica e Industriale, Università di Parma, Viale delle Scienze, I-43100 Parma, Italy

(*Received* 6 *March* 1995; *accepted* 18 *May* 1995)

Abstract

The title compound, $C_{17}H_{22}O_8$, is an example of a product obtained from a cyclodicarbonylation reaction