

graphically different side groups are identical within experimental error. The bond lengths to the H atoms not shown range from 0.92 to 1.08 Å. Bond lengths in the polymer chain are consistent with a poly-en-yne structure where triple bonds are conjugated with double bonds. Within 0.01 to 0.02 Å the phenyl rings and the polymer backbone are planar. The equations of the calculated best planes and the angles between the different planes are listed in Table 2.

Calculations were performed at the Rechenzentrum der Universität Freiburg and were supported by the Deutsche Forschungsgemeinschaft. We gratefully acknowledge the assistance of Dr E. Keller in obtaining the intensities.

*Acta Cryst.* (1980), **B36**, 1956–1958

## Structure of 3,3-Di(*o*-tolyl)phthalide

BY C. J. WANG, M. TH. WU AND Y. J. CHEN

*Chemistry Department, Chung Shan Institute of Science and Technology, PO Box 1–4, Lung Tan, Taiwan*

AND T. H. HSEU

*National Tsing Hua University, Hsinchu, Taiwan*

(Received 17 October 1979; accepted 16 January 1980)

**Abstract.**  $C_{22}H_{18}O_2$ ,  $M_r = 314.18$ , monoclinic,  $P2_1/c$ ,  $a = 8.614$  (2),  $b = 7.210$  (2),  $c = 27.620$  (6) Å,  $\beta = 93.98$  (11)°,  $Z = 4$ ,  $\mu(Cu K\alpha) = 0.632$  mm<sup>-1</sup>,  $D_c = 1.248$ ,  $D_m = 1.245$  Mg m<sup>-3</sup>. The structure was refined to  $R = 0.064$ ,  $R_w = 0.065$ , for 2252 independent reflections. The angle between the planes of the two tolyl groups is 81.7 (6)°, and the angles between the plane of the phthalide ring and the planes of each of the tolyl groups are 77.8 (3) and 65.1 (5)°. The two C–O bonds in the  $\gamma$ -lactone group differ significantly in length, 1.477 (4) and 1.368 (4) Å, the shorter being adjacent to the carbonyl group.

**Introduction.** 3,3-Di(*o*-tolyl)phthalide was synthesized according to the method of Bartlett & Jones (1942). It was recrystallized from nitromethane by slow evaporation at room temperature. Preliminary crystal data were determined by photographic methods. A transparent crystal 0.3 × 0.3 × 0.4 mm was used on a computer-controlled Picker diffractometer; 2252 independent reflections with  $I > 3\sigma(I)$  were collected up to  $2\theta = 120^\circ$  with Ni-filtered Cu  $K\alpha$  radiation and the

## References

- DECLERCQ, J. P., GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1973). *Acta Cryst.* **A29**, 231–234.  
*International Tables for X-ray Crystallography* (1968). Vol. III, 2nd ed. Birmingham: Kynoch Press.  
 JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.  
 STEWART, J. M., MACHIN, P. A., DICKINSON, C., AMMON, H. L., HECK, H. & FLACK, H. (1976). The XRAY system – version of March 1976. Tech. Rep. TR-446. Computer Science Center, Univ. of Maryland, College Park, Maryland.  
 STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.  
 WEGNER, G. (1972). *Makromol. Chem.* **154**, 35–48.  
 WEGNER, G. (1979). *Molecular Metals*, edited by W. E. HATFIELD, pp. 209–242. New York: Plenum.

$\omega$ – $2\theta$  scanning technique. The cell dimensions were obtained by a least-squares fit to the  $2\theta$  values of 20 reflections. Lorentz and polarization corrections were applied, but none were made for absorption.

The structure was solved by *MULTAN* (Germain, Main & Woolfson, 1971). The  $E$  map revealed all the nonhydrogen atoms. Full-matrix isotropic least-squares refinement gave  $R = 0.108$ . All H atoms were then located on a difference map. The final  $R = 0.064$ .\*

**Discussion.** The final positional parameters are given in Table 1. Fig. 1 shows a perspective view of the molecule. The bond lengths and angles are given in Fig. 2.

The average C–C distances and the mean C–C–C angles of the phenyl rings in the two *o*-tolyl groups are 1.391 (13) Å, 120.0 (12)° and 1.392 (14) Å,

\* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34986 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional coordinates ( $\times 10^4$ ) (for H  $\times 10^3$ )

	x	y	z
O(1)	2342 (3)	3676 (3)	3763 (1)
O(2)	4008 (3)	5452 (3)	4212 (1)
C(1)	2031 (4)	1255 (5)	4310 (1)
C(2)	1582 (5)	-257 (6)	4571 (1)
C(3)	2218 (6)	-435 (7)	5046 (2)
C(4)	3249 (5)	856 (7)	5255 (2)
C(5)	3677 (5)	2390 (7)	4997 (1)
C(6)	3050 (4)	2546 (5)	4522 (1)
C(7)	1554 (4)	1865 (5)	3793 (1)
C(8)	3247 (4)	4065 (5)	4177 (1)
C(9)	-2673 (5)	3559 (7)	3808 (2)
C(10)	-3455 (5)	2067 (8)	3600 (2)
C(11)	-2629 (5)	538 (8)	3460 (2)
C(12)	-997 (4)	555 (6)	3521 (1)
C(13)	-209 (4)	2066 (5)	3725 (1)
C(14)	-1038 (4)	3589 (5)	3878 (1)
C(15)	-319 (6)	5277 (8)	4138 (3)
C(16)	2237 (4)	677 (5)	3398 (1)
C(17)	3002 (4)	-980 (5)	3517 (1)
C(18)	3664 (5)	-2064 (7)	3169 (2)
C(19)	3575 (6)	-1479 (8)	2694 (2)
C(20)	2818 (5)	154 (8)	2570 (2)
C(21)	2124 (4)	1272 (6)	2909 (1)
C(22)	1307 (7)	3033 (9)	2741 (2)
H(2)	77 (4)	-107 (5)	443 (1)
H(3)	186 (6)	-127 (7)	523 (2)
H(4)	380 (5)	74 (6)	560 (2)
H(5)	441 (5)	345 (7)	513 (2)
H(9)	-338 (6)	451 (7)	392 (2)
H(10)	-469 (5)	199 (5)	359 (1)
H(11)	-320 (6)	-67 (8)	334 (2)
H(12)	-37 (7)	-51 (10)	343 (2)
H(17)	308 (4)	-145 (5)	382 (1)
H(18)	429 (5)	-314 (7)	327 (2)
H(19)	424 (6)	-209 (8)	244 (2)
H(20)	280 (5)	57 (6)	223 (2)
H(15A)	-109 (6)	616 (8)	426 (2)
H(15B)	6 (6)	584 (7)	389 (2)
H(15C)	18 (6)	478 (8)	450 (2)
H(22A)	156 (10)	416 (12)	282 (3)
H(22B)	10 (8)	324 (10)	285 (2)
H(22C)	104 (7)	306 (10)	239 (3)

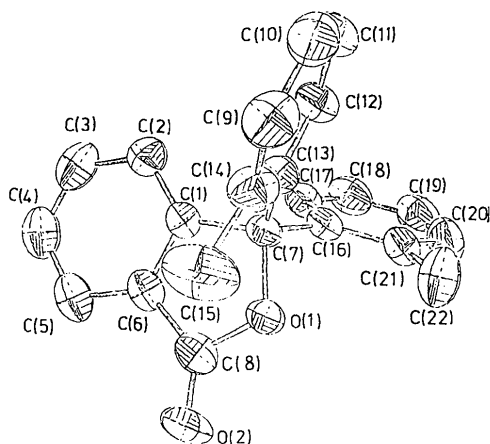
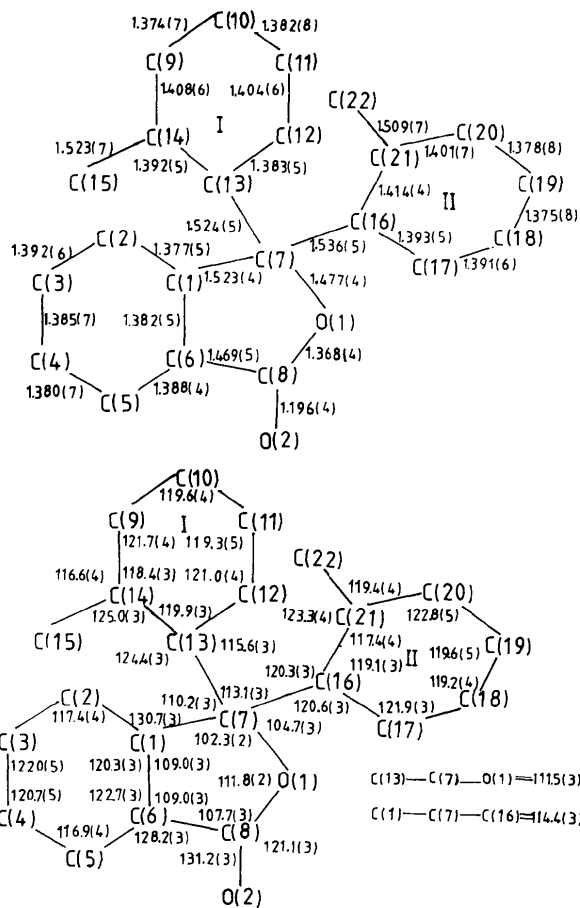


Fig. 1. ORTEP drawing (Johnson, 1965) of the molecule with the thermal ellipsoids of the non-hydrogen atoms at 50% probability.

Fig. 2. Bond lengths (Å) and angles ( $^{\circ}$ ) in 3,3-di(*o*-tolyl)phthalide; values in parentheses denote the e.s.d.'s.

120.0 (20) $^{\circ}$ , respectively, for tolyl groups (I) and (II). The corresponding distance and angle in the six-membered part of the phthalide group are 1.384 (5) Å and 120.0 (24) $^{\circ}$  respectively.

Generally, the dimensions of the phthalide group are similar to those in *meso*-3,3'-bis(*p*-bromophenyl)-3,3'-biphtalidyl (Kalyani, Manohar & Mani, 1967) and 3-(*p*-bromophenyl)phthalide (Kalyani & Vijayan, 1969). The  $\gamma$ -lactone group exhibits the expected characteristics (Karle & Karle, 1966; McConnell, Mathieson & Schoenborn, 1964; Schoenborn & McConnell, 1962). The two C—O bonds on the ring differ significantly in length, C(7)—O(1) 1.477 (4) and C(8)—O(1) 1.368 (4) Å, the shorter being adjacent to the carbonyl group. The group is nearly planar. The internal angles in the five-membered ring have a mean of 108 (3) $^{\circ}$ . The two exocyclic angles of the carbonyl C atom differ by 10.1 $^{\circ}$  [O(2)—C(8)—O(1) 121.1 (3) $^{\circ}$  and O(2)—C(8)—C(6) 131.2 (3) $^{\circ}$ ].

The equations for the least-squares planes in the molecule are: tolyl group (I):  $1.0387x + 3.0564y - 25.0174z = -8.7357$ ; tolyl group (II):  $7.3276x + 3.6334y + 2.8689z = 2.8520$ ; phthalide group:

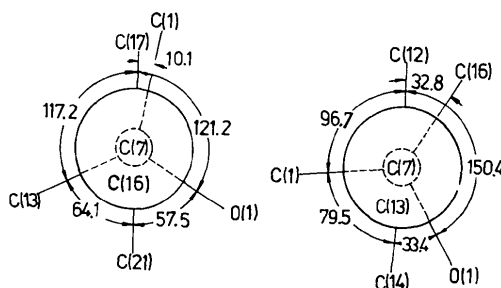


Fig. 3. Newman projections showing the conformations about C(7)–C(13) and C(7)–C(16).

$6.8986x - 3.5963y - 10.7298z = -3.7006$ ; lactone group:  $6.9209x - 3.4707y - 11.2527z = -3.8651$ ; where  $x, y, z$  are fractional coordinates and the right-hand side is the distance of the plane from the cell origin. The root-mean-square displacements of the atoms in these groups from their corresponding least-squares planes are 0.026, 0.0055, 0.027 and 0.018 Å, respectively.

The two tolyl planes are almost normal to each other. The angle between the two planes is  $81.7(6)^\circ$ . The angles between the phthalide plane and the planes of the tolyl groups are  $77.8(3)^\circ$  (I) and  $65.1(5)^\circ$  (II). Fig. 3 shows the conformations about C(7)–C(13) and C(7)–C(16). The angles at C(7) indicate some significant deviations from the ideal  $109.5^\circ$ . The smallest angle occurs at O(1)–C(7)–C(1) ( $102^\circ$ ); this is probably due to the strain in the lactone ring caused by the shortening of O(1)–C(8). The larger angles at C(16)–C(7)–C(13) ( $113^\circ$ ) and C(16)–C(7)–C(1) ( $114^\circ$ ) are apparently the result of steric hindrance between tolyl group (II) and tolyl group (I) as well as the phthalide group.

The general geometry of the molecule is roughly that the lactone  $-\text{O}-\text{C}(=\text{O})-$  and the two tolyl methyl groups lie on one side of the molecule and three phenyl rings on the other. The non-bonded intramolecular distances between the ether O atom and the two tolyl methyl C atoms are O(1)–C(15) =  $2.827(7)$  Å and O(1)–C(22) =  $2.937(6)$  Å. The molecular arrangement is shown in Fig. 4. The short intermolecular contact distances are given in Table 2. All these contacts involve the carbonyl O and phenyl C atoms of neighboring molecules. However, none is abnormally

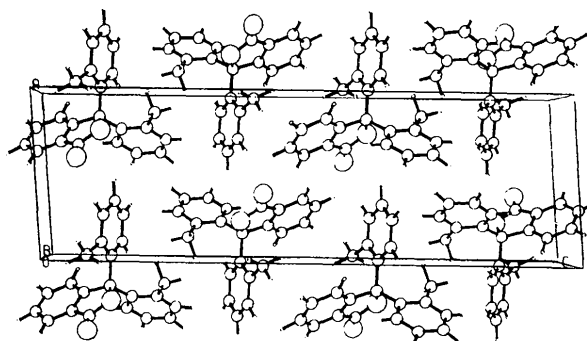


Fig. 4. Crystal structure of the molecule viewed along *b*.

Table 2. Short intermolecular contacts (Å)

Symmetry code: (I) $1 + x, y, z$ ; (II) $x, 1 + y, z$ ; (III) $1 - x, 1 - y, 1 - z$ ; (IV) $-x, -y, 1 - z$ .			
O(2)···C(9)I	3.425 (5)	O(2)···C(5)III	3.251 (4)
O(2)···C(17)II	3.290 (4)	C(2)···C(3)IV	3.545 (7)
O(2)···C(18)II	3.388 (6)		

short. Seemingly, the crystal packing is decided principally by the shape of the molecule.

We thank Mr Y. F. Wang for supplying the sample and Dr Y. Wang of the National Research Council, Ottawa, for the data collection.

#### References

- BARTLETT, P. D. & JONES, J. E. (1942). *J. Am. Chem. Soc.* **64**, 1837–1842.  
 GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* **A27**, 368–376.  
 JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.  
 KALYANI, V., MANOHAR, H. & MANI, N. V. (1967). *Acta Cryst.* **23**, 272–281.  
 KALYANI, V. & VIJAYAN, M. (1969). *Acta Cryst.* **B25**, 1281–1288.  
 KARLE, I. L. & KARLE, J. (1966). *Acta Cryst.* **20**, 555–559.  
 MCCONNELL, J. F., MATHIESON, A. M. & SCHOENBORN, B. P. (1964). *Acta Cryst.* **17**, 472–477.  
 SCHOENBORN, B. P. & MCCONNELL, J. F. (1962). *Acta Cryst.* **15**, 779–785.