The B(11)—N(12) distance of 1·606 (2) Å is close to those in $Me_3NBH_2CO_2H$, 1·589 Å (Spielvogel *et al.*, 1976), and H_3NBH_2CN, 1·577 Å (McPhail *et al.*, 1978). The shortest intermolecular distance (excluding H atoms) is an N(11)···O(11) separation of 3·013 (2) Å, which is a weak hydrogen bond with an N(11)—H(111)···O(11) angle of 163 (2)°.

References

BEURSKENS, P. T., BEURSKENS, G., STRUMPEL, M. & NORDMAN, C. E. (1987). *Patterson and Pattersons*, edited by J. P. GLUSKER, B. K. PATTERSON & M. ROSSI, pp. 356–367. Oxford Univ. Press.

BEURSKENS, P. T., BOSMAN, W. P., DOESBURG, H. M., VAN DEN HARK, TH. E. M., PRICK, P. A. J., NOORDIK, J. H., BEURSKENS, G., GOULD, R. O. & PARTHASATHI, V. (1983). *Conformation in Biology*, edited by R. SRINIVASAN & R. H. SARMA, pp. 389–406. New York: Adenine Press.

BEURSKENS, P. T., GOULD, R. O., BRUINS SLOT, H. J. & BOSMAN, W. P. (1987). *Z. Kristallogr.* **179**, 127–159.

DAS, M. K. & MUKERJEE, P. (1985). *J. Chem. Res. (S)*, pp. 66–67.

DAS, M. K. & MUKERJEE, P. (1987). *J. Chem. Res. (M)*, pp. 2981–2983.

GRANT, D. F. & GABE, E. J. J. (1978). *J. Appl. Cryst.* **11**, 114–120.

HALL, I. H., GILBERT, C. J., MCPHAIL, A. T., MORSE, K. W., HASSETT, K. & SPIELVOGEL, B. F. (1985). *J. Pharm. Sci.* **74**, 755–758.

HALL, I. H., GRIFFIN, T. S., DOCKS, E. L., BROTHERTON, R. J. & FUTCH, G. (1986). *J. Pharm. Sci.* **75**, 706–710.

HALL, I. H., SPIELVOGEL, B. F., SOOD, A., AHMED, F. & JAFRI, S. (1987). *J. Pharm. Sci.* **76**, 359–365.

HALL, I. H., STARNES, C. O., SPIELVOGEL, B. F., WISIAN-NEILSON, P., DAS, M. K. & WOJNOWICH, L. (1979). *J. Pharm. Sci.* **68**, 685–688.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

LEHMANN, M. S. & LARSEN, F. K. (1974). *Acta Cryst.* **A30**, 580–584.

MCPHAIL, A. T., ONAN, K. D., SPIELVOGEL, B. F. & WISIAN-NEILSON, P. (1978). *J. Chem. Res. (S)*, pp. 205–208.

MOTHERWELL, W. D. S. (1976). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.

NORTH, A. C. T., PHILIPS, D. C. & MATHEWS, F. S. (1968). *Acta Cryst.* **A24**, 351–359.

SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

SPIELVOGEL, B. F., AHMED, F. U., MORSE, K. W. & MCPHAIL, A. T. (1984). *Inorg. Chem.* **23**, 1776–1777.

SPIELVOGEL, B. F., DAS, M. K., MCPHAIL, A. T., ONAN, K. D. & HALL, I. H. (1980). *J. Am. Chem. Soc.* **102**, 6343–6344.

SPIELVOGEL, B. F., WOJNOWICH, L., DAS, M. K., MCPHAIL, A. T. & HARGRAVE, K. D. (1976). *J. Am. Chem. Soc.* **98**, 5702–5703.

SUR, P., ROY, D. K. & DAS, M. K. (1981). *IRCS Med. Sci.* **9**, 1066–1067.

WALKER, N. & STUART, D. (1983). *Acta Cryst.* **A39**, 158–166.

Acta Cryst. (1989). **C45**, 1459–1461

Structure of an Enone Derivative

BY TOSHIO ABE, TOMITAKE TSUKIHARA,* MASARU YASUI, HIROYUKI SAIMOTO AND YOSHIHIRO SHIGEMASA

Department of Industrial Chemistry, Faculty of Engineering, Tottori University,
Koyama Minami 4-101, *Tottori* 680, *Japan*

(*Received* 18 *January* 1989; *accepted* 13 *March* 1989)

Abstract. 2-Oxo-1,4,4-triphenyl-3-butenyl acetate, $C_{24}H_{20}O_3$, $M_r = 356\cdot42$, orthorhombic, $P2_12_12_1$, $a = 9\cdot717\ (1)$, $b = 21\cdot078\ (2)$, $c = 9\cdot054\ (2)$ Å, $V = 1854\cdot4\ (5)$ Å^3, $Z = 4$, $D_x = 1\cdot277$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1\cdot5418$ Å, $\mu = 6\cdot00$ cm^{-1}, $F(000) = 752$, room temperature, $R = 0\cdot054$ for 1553 observed reflections [$F_o > 3\sigma(F_o)$]. There are no short intermolecular contacts less than the van der Waals contact distances. The phenyl groups have higher temperature factors than the ethylene group.

Experimental. Light-yellow crystals obtained from ethyl ether. Crystal of dimensions 0·2 × 0·2 × 0·2 mm rounded by a crystal grinder. Rigaku AFC-5 rotating-anode four-circle diffractometer, graphite-monochromated Cu $K\alpha$ radiation. Cell dimensions determined from 20 2θ angles in the range $57 < 2\theta < 60°$. Intensities collected to $(\sin\theta)/\lambda = 0\cdot570$ Å^{-1} in h 0/11, k 0/25 and l 0/10, θ–2θ scans, θ-scan width $(1\cdot5 + 0\cdot15\tan\theta)°$, three standard reflections monitored every 100 reflections showed no significant variation in intensity. 1827 unique reflections measured, 1553 intensities observed [$F_o < 3\sigma(F_o)$ and two very strong reflections rejected], no absorption correction. Structure solved by *MULTAN* (Germain, Main & Woolfson, 1971). H atoms located on a difference map. Positional parameters of all atoms, anisotropic thermal parameters for C and O atoms, and isotropic thermal parameters for H atoms refined by block-diagonal least squares (Ashida, 1973). $\sum w|\Delta F|^2$ minimized with $w = 1\cdot0$ for $0 < F_o < 20$ and $w = [1\cdot0 + 0\cdot1(F_o - 20)]^{-1}$ for $F_o > 20$. Final

---

* Author to whom correspondence should be addressed.

0108-2701/89/091459-03$03.00

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters* (Å²) *with e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{4}{3}(B_{11}a^2 + B_{22}b^2 + B_{33}c^2).$$

	x	y	z	B_{eq}
O(1)	0·6354 (4)	0·2482 (1)	1·0998 (3)	6·2 (1)
O(2)	0·3705 (4)	0·1605 (2)	1·1723 (4)	7·3 (1)
O(3)	0·5295 (3)	0·1442 (1)	0·9969 (3)	4·8 (1)
C(1)	0·4859 (5)	0·1997 (2)	0·9269 (5)	4·4 (1)
C(2)	0·5437 (5)	0·2542 (2)	1·0083 (5)	4·6 (1)
C(3)	0·4899 (5)	0·3113 (2)	0·9540 (5)	5·0 (1)
C(4)	0·5161 (5)	0·3694 (2)	0·9963 (5)	4·4 (1)
C(5)	0·5190 (7)	0·0737 (2)	1·1893 (6)	7·9 (2)
C(6)	0·4620 (6)	0·1309 (2)	1·1245 (5)	5·6 (1)
C(7)	0·5351 (4)	0·1975 (2)	0·7674 (4)	4·0 (1)
C(8)	0·4424 (5)	0·2030 (2)	0·6563 (5)	5·6 (1)
C(9)	0·4880 (6)	0·2026 (2)	0·5094 (5)	6·6 (2)
C(10)	0·6209 (6)	0·1968 (2)	0·4772 (5)	6·2 (1)
C(11)	0·7141 (6)	0·1910 (3)	0·5885 (6)	6·9 (2)
C(12)	0·6722 (5)	0·1906 (2)	0·7355 (5)	5·7 (1)
C(13)	0·4644 (5)	0·4201 (2)	0·9103 (5)	4·7 (1)
C(14)	0·4228 (5)	0·4736 (2)	0·9704 (6)	5·5 (1)
C(15)	0·3741 (6)	0·5225 (2)	0·8865 (7)	6·7 (2)
C(16)	0·3685 (6)	0·5162 (2)	0·7348 (7)	7·0 (2)
C(17)	0·4088 (7)	0·4637 (2)	0·6711 (6)	7·7 (2)
C(18)	0·4559 (6)	0·4155 (2)	0·7537 (6)	6·6 (2)
C(19)	0·5935 (5)	0·3849 (2)	1·1328 (5)	4·5 (1)
C(20)	0·5617 (6)	0·3576 (2)	1·2672 (5)	6·2 (2)
C(21)	0·6279 (8)	0·3754 (3)	1·3967 (6)	8·6 (2)
C(22)	0·7256 (7)	0·4189 (3)	1·3939 (8)	9·3 (2)
C(23)	0·7633 (6)	0·4460 (3)	1·2643 (9)	9·0 (2)
C(24)	0·6952 (5)	0·4293 (2)	1·1301 (7)	6·4 (2)

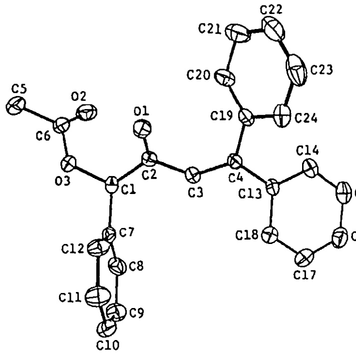

Fig. 1. Perspective view with the atomic numbering system. The absolute configuration of the molecule is arbitrary.

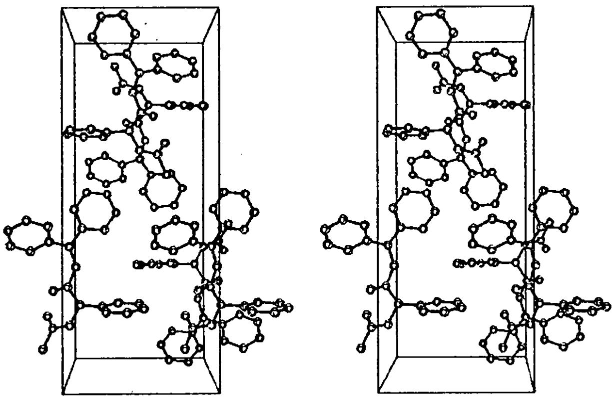

Fig. 2. A stereoview of the unit-cell packing viewed along the *a* axis.

Table 2. *Bond lengths* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

O(1)—C(2)	1·224 (6)	C(10)—C(11)	1·361 (9)
O(2)—C(6)	1·185 (7)	C(11)—C(12)	1·391 (8)
O(3)—C(1)	1·443 (5)	C(7)—C(12)	1·372 (7)
O(3)—C(6)	1·360 (6)	C(13)—C(14)	1·363 (7)
C(1)—C(2)	1·520 (6)	C(14)—C(15)	1·402 (8)
C(1)—C(7)	1·522 (6)	C(15)—C(16)	1·381 (9)
C(2)—C(3)	1·451 (7)	C(16)—C(17)	1·354 (10)
C(3)—C(4)	1·362 (7)	C(17)—C(18)	1·379 (10)
C(4)—C(13)	1·453 (7)	C(13)—C(18)	1·423 (8)
C(4)—C(19)	1·486 (6)	C(19)—C(20)	1·392 (7)
C(5)—C(6)	1·500 (9)	C(20)—C(21)	1·396 (9)
C(7)—C(8)	1·356 (7)	C(21)—C(22)	1·351 (10)
C(8)—C(9)	1·403 (8)	C(22)—C(23)	1·367 (11)
C(9)—C(10)	1·329 (8)	C(23)—C(24)	1·433 (10)
		C(19)—C(24)	1·392 (8)

O(1)—C(2)—C(1)	120·8 (4)	C(8)—C(7)—C(12)	120·0 (4)
O(1)—C(2)—C(3)	125·9 (5)	C(4)—C(13)—C(18)	120·0 (5)
O(2)—C(6)—C(5)	126·8 (6)	C(4)—C(13)—C(14)	123·8 (4)
O(2)—C(6)—O(3)	123.7 (5)	C(13)—C(14)—C(15)	123·4 (5)
C(1)—O(3)—C(6)	114·4 (4)	C(14)—C(15)—C(16)	118·4 (6)
O(3)—C(1)—C(2)	110·6 (4)	C(15)—C(16)—C(17)	119·9 (6)
O(3)—C(1)—C(7)	107·3 (3)	C(16)—C(17)—C(18)	121·7 (7)
O(3)—C(6)—C(5)	109·6 (5)	C(13)—C(18)—C(17)	120·4 (6)
C(1)—C(2)—C(3)	113·1 (4)	C(14)—C(13)—C(18)	116·2 (5)
C(2)—C(3)—C(4)	130·9 (5)	C(13)—C(4)—C(19)	116·3 (4)
C(3)—C(4)—C(13)	120·6 (4)	C(4)—C(19)—C(20)	121.0 (4)
C(3)—C(4)—C(19)	123·1 (4)	C(4)—C(19)—C(24)	120·2 (4)
C(1)—C(7)—C(8)	119·5 (4)	C(19)—C(20)—C(21)	120·8 (5)
C(1)—C(7)—C(12)	120·6 (4)	C(20)—C(21)—C(22)	120·4 (7)
C(7)—C(8)—C(9)	119·5 (5)	C(21)—C(22)—C(23)	121.2 (7)
C(8)—C(9)—C(10)	121·0 (5)	C(22)—C(23)—C(24)	119·4 (7)
C(9)—C(10)—C(11)	119·6 (6)	C(19)—C(24)—C(23)	119.7 (6)
C(10)—C(11)—C(12)	120·9 (5)	C(20)—C(19)—C(24)	118·5 (5)
C(7)—C(12)—C(11)	119·0 (5)		

$R = 0{\cdot}054$, $wR = 0{\cdot}061$ and $S = 0{\cdot}691$. Maximum positive and maximum negative electron densities in final difference Fourier synthesis are 0·48 and $-0{\cdot}50\ \text{e Å}^{-3}$. $\Delta/\sigma_{\max}$ in the final cycle = 0·3 (z coordinate of H atom). Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). All computations performed on a HITAC 280D at the Tottori University Computing Center and on a FACOM M780/30 at the Data Processing Center of Kyoto University. Final atomic coordinates and equivalent isotropic temperature factors are given in Table 1. Bond distances and angles are listed in Table 2.* A perspective view of the moelcule with atomic numbering and a stereoview of crystal packing drawn by *DCM*-3 (Takenaka, 1977) are shown in Figs. 1 and 2, respectively. There are no short intermolecular contacts less than the van der Waals contact distances. The phenyl groups have higher temperature factors than the

---

* Lists of structure factors, anisotropic temperature factors of the non-H atoms, and isotropic temperature factors and positional parameters of H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51804 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

ethylene group. The absolute configuration was not determined in the present study.

Related literature. The detailed synthetic method and spectral data of the enone derivatives will be published by Shigemasa (1989).

The authors thank Professor N. Yasuoka of the Himeji Institute of Technology for the use of the X-ray diffractometer.

References

Ashida, T. (1973). *HBLS-V. The Universal Crystallographic Computing System, Osaka*, pp. 55–61. Computing Center, Osaka Univ., Japan.

Germain, G., Main, P. & Woolfson, M. M. (1971). *Acta Cryst.* A27, 368–376.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Shigemasa, Y. (1989). In preparation.

Takenaka, A. (1977). *DCM-3*. Program for drawing crystal and molecular structures. Tokyo Institute of Technology, Japan.

Acta Cryst. (1989). C45, 1461–1463

Structure of Hexa-2,4-diyne-1,6-diyl Bis(4-methoxybenzoate)

By Michael J. Barrow, Alan J. Lough and G. Harry W. Milburn

Department of Applied Chemical Sciences, Napier Polytechnic of Edinburgh, Edinburgh EH10 5DT, Scotland

(*Received* 16 *March* 1989; *accepted* 3 *May* 1989)

Abstract. $C_{22}H_{18}O_6$, $M_r = 378·4$, monoclinic, $C2/c$, $a = 23·084$ (4), $b = 4·269$ (11), $c = 19·071$ (3) Å, $\beta = 97·82$ (1)°, $V = 1861·9$ Å^3, $Z = 4$, $D_x = 1·35$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0·092$ mm^{-1}, $F(000) = 792$, room temperature, $R = 0·030$ and $wR = 0·032$ for 981 diffractometer reflections with $F_o > 6\sigma(F_o)$. Only one crystal form was obtained from a variety of solvents. The crystal surfaces become red on exposure to X-rays, but the bulk crystal does not undergo polymerization even after prolonged exposure to heat or X- or γ-radiation. The monomer molecules form a regular inclined stack along the crystal y direction.The distance between reactive 1···4′ acetylenic C atoms in adjacent molecules is 4·00 Å, the angle between the diacetylene chain and the stacking direction is 59·4°.

Experimental. The title compound was prepared at room temperature by the dropwise addition of 4-methoxybenzoyl chloride in tetrahydrofuran to a stirred mixture of hexa-2,4-diyne-1,6-diol and triethylamine in tetrahydrofuran. Recrystallization from ethanol gave pale-yellow crystals; yield 77%, m.p. 387–388 K, found C = 69·8, H = 4·8%; $C_{22}H_{18}O_6$ requires C = 69·8, H = 4·8%. Further recrystallization by room-temperature evaporation of acetone, methanol, chloroform, diethyl ether or dichloromethane solutions gave colourless needles. These were examined optically but all specimens appeared to have the same crystal form. Differential scanning calorimetry (Mettler TA 3000) over the range 303–453 K in a nitrogen atmosphere showed no reaction exotherm before or after the melting point; samples started to decompose at 453 K. There was no obvious polymerization when samples were subjected to pressure in a KBr disc press. When crystals were exposed to X-rays they developed red surfaces suggesting some polymerization or other surface reaction, but there were no detectable changes to the diffraction angles or intensities of X-ray reflections. A sample of single crystals was also exposed to γ-radiation (50 Mrads over 7 d) at the Scottish Universities Reactor, East Kilbride. The surfaces of these crystals became black but again oscillation and Weissenberg X-ray photography showed no change to the space group, nor any detectable changes to cell parameters or relative intensities of reflections.

Two crystals grown from acetone solution (and not previously subjected to X- or γ-radiation) were used for X-ray measurements. Crystal (1) had dimensions 0·48 × 0·40 × 0·08 mm and was mounted along the b axis. Crystal (2) had dimensions 0·40 × 0·38 × 0·10 mm and was mounted along the $[1\bar{1}0]$ face diagonal. The space group and approximate cell parameters were calculated from oscillation and Weissenberg photographs (Cu $K\alpha$ radiation). Accurate cell parameters and intensity data were measured using a Stoe Stadi-2 diffractometer with graphite-monochromated Mo $K\alpha$ radiation. Cell parameters determined from ω and 2θ angles of 20 $h0l$ reflections ($21 < 2\theta < 29°$) from crystal (1) and 14 nnl reflections ($19 < 2\theta < 35°$) from crystal (2). Intensity data were measured for the layers h, $0\rightarrow5$, l from crystal (1), and $m+n$, n, l for $m = 0,2,4,6$ from crystal (2). (Diagonal layers with m = odd are systematically