1980) [average As—C 1·948 (9) Å, average CAsC angle 91·5 (16)°] quaternization has decreased the As—C_{ar} distance and increased the C_{ar}—As—C_{ar} angle. In arsatriptycene itself (Freijee & Stam, 1980), As—C is 1·94 Å and C—As—C is 90°, but these results are less reliable because of disorder. There is good agreement between corresponding bonds and angles in the three benzene rings. As in 7-phenylarsatriptycene there is a tendency for the inner C—C bonds to be longer and for the outer C—C bonds to be shorter than average. The benzene rings [(I) = C(1)–C(6); (II) = C(7)–C(12); (III) = C(13)–C(18)] are planar within 0·005 (I), 0·008 (II) and 0·015 Å (III). The As atom deviates significantly from all three planes [0·06 (I), 0·09 (II) and 0·05 Å (III)]. The corresponding deviations for C(19) are 0·01, 0·01 and 0·06 Å. The angles between the planes are: (I)–(II) 120·4, (I)–(III) 120·4 and (II)–(III) 119·3°.

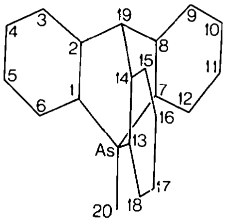

Fig. 1. Atomic numbering and shape of the molecule.

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2-Benzylidene-1-indanone

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Abstract. $C_{16}H_{12}O$, M_r = 220·3, monoclinic, $P2_1/a$, $Z = 4$, a = 23·949 (2), b = 7·990 (1), c = 6·095 (1) Å, β = 91·18 (4)°, V = 1166·0 Å^3, D_m = 1·27, D_c = 1·25 Mg m^{-3}. The final R = 0·062 and R_w = 0·058 for 1035 non-zero reflections. The molecule has a *trans-s-cis* configuration.

Introduction. Spectroscopic investigations (IR, UV, NMR) of arylidene derivatives of 1-tetralone and 4-chromanone showed that an increase in the electron interactions of electron-donor substituents in a conjugated bond system leads to the flattening of the molecules (Orlov, Borovoi & Lavrushin, 1976; Orlov, Borovoi, Surov & Lavrushin, 1976).

In order to confirm this conclusion we are undertaking the X-ray study of a series of benzylidene derivatives of 1-tetralone, 1-indanone and 4-chromanone with electron-donor (Rabinovich, 1970) and electron-acceptor (Rabinovich, Schmidt & Shakked, 1970) substituents in the benzylidene rings.

We began our investigations with 2-benzylidene-1-tetralone (Kałuski, Skrzypczak-Jankun, Orlov & Borovoi, 1978) and now present the results of the determination of the molecular and crystal structure of 2-benzylidene-1-indanone. Crystals of this compound, synthesized as in Orlov, Borovoi & Lavrushin (1976) and Orlov, Borovoi, Surov & Lavrushin (1976), were obtained as light-yellow monoclinic plates elongated along the c axis by evaporating its solution in an ethanol–water mixture (3:1 v/v).

A crystal 0·1 × 0·3 × 0·5 mm was investigated on a Syntex $P2_1$ diffractometer to obtain precise lattice constants and to measure the diffraction pattern in the range 2θ = 0–114°. The θ–2θ technique was used with graphite-monochromated Cu $K\alpha$ radiation.

In order to determine the structure model, 1035 reflections with $I > 1{\cdot}96\sigma I$ were used; these were corrected for Lorentz and polarization factors. No absorption or extinction corrections were made. The structure was solved directly by the application of

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MULTAN (Germain, Main & Woolfson, 1971) and refined by the full-matrix least-squares program *FMLS* from the *XTL* system (Syntex, 1976). The positions of the H atoms found on a difference Fourier map were used in the structure factor calculations but they were not included in the refinement. The result of the calculations for 1035 reflections was $R = 0{\cdot}065$. The function minimized was $\sum w(F_o - F_c)^2$ where $w = (1/\sigma_{F_o})^2$. In the last few cycles of refinement the following weighting scheme was used: $w = (F_o/F_{\rm low})^2$ if $F_o < F_{\rm low}$; $w = 1$ if $F_{\rm low} < F_o < F_{\rm high}$; $w = (F_{\rm high}/F_o)^2$ if $F_o > F_{\rm high}$; where $F_{\rm low} = 4{\cdot}2$, $F_{\rm high} = 12{\cdot}4$.

The final discrepancy factors are $R = 0{\cdot}062$ and $R_w = 0{\cdot}058$ for 1035 reflections;* of these, 209 corresponded to $F_o < F_{\rm low}$ and 234 corresponded to $F_o > F_{\rm high}$. The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). All calculations were carried out on a Nova 1200 computer using programs included in the Syntex *XTL-XTL/E* structure determination system (Syntex, 1976). The atomic coordinates and equivalent isotropic thermal parameters are given in Table 1.

Table 1. *Fractional atomic coordinates* (×10^4 *for nonhydrogen*, ×10^3 *for hydrogen atoms*) *and isotropic thermal parameters*

The isotropic thermal parameters for H are 5·0 Å^2.

	x	y	z	B_j(Å^2)
C(1)	604 (2)	1802 (6)	3372 (7)	4·7 (3)
C(2)	896 (2)	2580 (5)	5291 (7)	4·0 (2)
C(3)	459 (2)	3139 (6)	6907 (7)	4·0 (2)
C(4)	−86 (2)	2792 (6)	5714 (7)	4·5 (3)
C(5)	−625 (2)	3157 (6)	6336 (8)	5·3 (3)
C(6)	−1068 (2)	2751 (7)	4941 (10)	5·7 (3)
C(7)	−978 (2)	1953 (7)	2934 (10)	5·7 (3)
C(8)	−441 (2)	1557 (6)	2287 (8)	5·1 (3)
C(9)	−1 (2)	2007 (5)	3698 (7)	3·9 (2)
C(10)	1452 (2)	2741 (6)	5309 (7)	4·4 (2)
C(11)	1832 (2)	3522 (6)	6906 (7)	4·4 (2)
C(12)	2396 (2)	3676 (6)	6336 (8)	5·1 (2)
C(13)	2780 (2)	4421 (7)	7758 (10)	5·8 (3)
C(14)	2613 (2)	5034 (7)	9762 (10)	5·7 (3)
C(15)	2060 (2)	4876 (7)	10341 (9)	5·5 (3)
C(16)	1668 (2)	4129 (6)	8951 (7)	5·1 (3)
O(1)	824 (1)	1142 (4)	1809 (5)	5·5 (2)
H(3)	50	451	753	
H(3′)	50	233	850	
H(5)	−67	367	800	
H(6)	−150	300	550	
H(7)	−133	167	150	
H(8)	−35	83	61	
H(10)	167	200	400	
H(12)	258	298	468	
H(13)	329	441	740	
H(14)	300	567	1050	
H(15)	200	567	1200	
H(16)	121	401	954	

Discussion. Experimental values of bond lengths and valence angles (Fig. 1) in the molecule of 2-benzylidene-1-indanone are in a good agreement with the values obtained for the very similar structure of 2-benzylidene-1-tetralone (Kałuski *et al.*, 1978). The mean value of the C—C bonds in the aromatic rings is 1·391 Å with root-mean-square deviations of 0·007 Å. The mean values of the 1,3-enone bond system agree well with those observed for chalcone derivatives (Rabinovich, Schmidt & Shakked, 1970, 1973). Torsion angles (Table 2) show that the molecule as a whole is non-planar. It can be clearly seen that the pentagonal ring cannot be considered planar. The conjugated double-bond system is also non-planar. The values obtained for torsion angles along C(1)—C(2) [O, C(1), C(2), C(10)], C(2)—C(10) [C(1), C(2), C(10), C(11)] and C(10)—C(11) [C(2), C(10), C(11), C(16)] lead to the conclusion that these bonds are twisted to

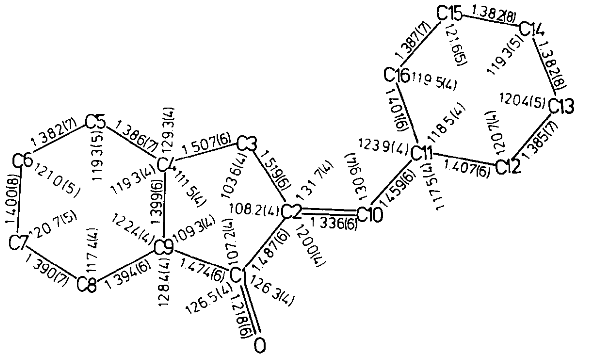

Fig. 1. Bond lengths (Å) and valency angles (°) with their e.s.d.'s.

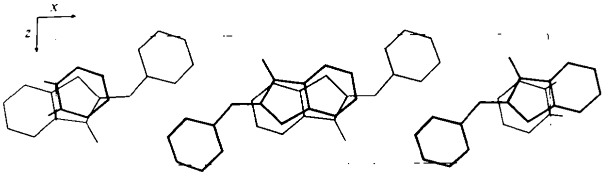

Fig. 2. A projection of the unit-cell contents along the *b* axis.

Table 2. *Torsion angles* (°) *with their e.s.d.'s*

C(1)–C(2)–C(3)–C(4)	−5·2 (4)
C(2)–C(3)–C(4)–C(9)	3·9 (5)
C(3)–C(4)–C(9)–C(1)	−1·1 (4)
C(4)–C(9)–C(1)–C(2)	−2·3 (5)
C(9)–C(1)–C(2)–C(3)	4·8 (4)
O–C(1)–C(2)–C(10)	6·5 (6)
C(1)–C(2)–C(10)–C(11)	176·4 (7)
C(2)–C(10)–C(11)–C(16)	7·3 (7)

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35054 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

form a spiral. The angles between the least-squares planes of the attached phenyl ring [C(4), C(5), C(6), C(7), C(8), C(9)] and those defined by C(9), C(1), O, C(2) and C(1), C(2), C(3), C(10) are 3·2 and 8·0° respectively. The planar phenyl rings are twisted with respect to each other at an angle of 15·3°. This value is copmparable with the 11·4° for chalcone (Rabinovich, 1970) and differs markedly from the 52·9° for 2-benzylidene-1-tetralone (Kałuski *et al.*, 1978).

The arrangement of molecules in the unit cell (projection *xOz*) is shown in Fig. 2. The closest intermolecular contacts are: $O(x,y,z)\cdots C(8)(-x,-y,-z) = 3{\cdot}410\ (6)$ Å and $O(x,y,z)\cdots C(3)(x, y, z-1) = 3{\cdot}482\ (5)$ Å. All other interactions are of van der Waals type.

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Structure of 2,11-Dimethoxybenzo[1,2-*b*;4,3-*b*′]bis(benzofuran)*

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Abstract. $C_{20}H_{14}O_4$, orthorhombic, $P2_12_12_1$, $a = 18{\cdot}100\ (3)$, $b = 15{\cdot}026\ (2)$, $c = 5{\cdot}509\ (2)$ Å, $Z = 4$, $V = 1498$ Å^3, $D_x = 1{\cdot}41$ Mg m^{-3}, $\mu(\text{Cu}\ K\alpha) = 82$ mm^{-1}. The final R value is 0·035 from 1158 data points for which $I_{\text{net}} \geq 4\sigma(I_{\text{net}})$. The molecule is not quite planar; the angles between adjacent ring planes vary from 0·8 to 2·2°. The distortion from a planar configuration is due to intramolecular overcrowding between H atoms.

Introduction. The crystal structure analysis of the title compound is part of a program of studies of quinone condensation products. The structure of the dimer 2,8-dimethoxydibenzofuran has been reported (part I). We now describe the molecular and crystal structure of the trimer 2,11-dimethoxybenzo[1,2-*b*;4,3-*b*′]bis(benzofuran).

X-ray intensities were collected on an automatic Philips PW 1100 diffractometer with monochromated Cu $K\alpha$ radiation, using the $\theta/2\theta$ scan technique. Of the 1345 reflexions collected ($\theta \leq 60°$), 1138 having $I_{\text{net}} \leq 4\sigma(I_{\text{net}})$ were considered observed. Lorentz and polarization corrections were applied but the intensities were not corrected for absorption effects [$\mu(\text{Cu}\ K\alpha) = 82$ mm^{-1}; crystal dimensions $0{\cdot}50 \times 0{\cdot}10 \times 0{\cdot}075$ mm]. The unit-cell parameters were obtained from a least-squares fit of the angular coordinates for 25 reflexions measured on the diffractometer. The structure was solved with *MULTAN* (Germain, Main & Woolfson, 1971). The most probable E map showed 19 out of the 24 non-hydrogen atoms. The remaining atoms, including the hydrogens, were obtained from successive difference Fourier maps. The structure was refined by standard full-matrix least-squares methods, using unit weights, to an R value of 0·035. The H atoms were included in the refinement with fixed isotropic thermal parameters, equal to those of their parent atoms. Scattering factors for C and O were taken from Cromer & Mann (1968) and for H from

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* Quinone Oligomerization. II. Part I: Berg, Karlsson, Pilotti & Söderholm (1978).

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