BTPB exists in the form (1*A*) and the distances C(6)—C(15) of 3·455 (6) Å and C(5)—C(8) of 5·005 (7) Å agree well with those of similar compounds in which C(3) is disubstituted (Fitzgerald *et al.*, 1978). The geometry around P in BTPB is as expected in a four-membered ring. The internal angle at P is 81·1 (2) with adjacent angles 86·2 (1) and 89·1 (3)°, and the internal angle opposite P is 96·8 (2)°. These angles match very well with those found in other substituted phosphetanes. Bond lengths and angles are comparable with the observed values in related structures.

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4-Benzylidene-2,3,4,5-tetrahydro-1-benzoxepin-5-one

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Abstract. $C_{17}H_{14}O_2$, $M_r = 250{\cdot}3$, orthorhombic, $P2_12_12_1$, $a = 11{\cdot}208\,(1)$, $b = 12{\cdot}828\,(2)$, $c = 8{\cdot}987\,(1)$ Å, $V = 1292{\cdot}1\,(3)$ Å^3, $Z = 4$, $D_x = 1{\cdot}290\,(6)$ g cm^{-3}, $\lambda(\text{Cu}\,K\alpha) = 1{\cdot}54178$ Å, $\mu(\text{Cu}\,K\alpha) = 5{\cdot}82$ cm^{-1}, $F(000) = 528$, $T = 292$ K, $R = 0{\cdot}047$ for 992 unique reflections. The tetrahydrooxepin ring is in a twist-boat conformation. The conformation of the *cisoid* 1,3-enone bridge between the two phenyl rings is modified from that in 3-benzylidene-4-chromanone by the tetrahydrooxepin ring. The inclination angle between the two planar phenyl rings is 20·2 (5)°.

Introduction. The present investigation is a continuation of our previous X-ray studies of α,β-unsaturated ketones (Katrusiak, Ratajczak-Sitarz, Kałuski & Orlov, 1987, and references cited therein). 4-Benzylidenetetrahydrooxepinone [hereinafter referred to as (I)] distinguishes itself from the ketones investigated previously by its seven-membered tetrahydrooxepin ring, which introduces strains in the molecular skeleton and modifies its conformation. The detailed spectroscopic studies of (I) and its analogues have been reported by Orlov, Borovoi & Lavrushin (1976) and Orlov, Mihedkina & Shandula (1984).

Experimental. Recrystallization from ethanol as colourless prisms, crystal fragment 0·40 × 0·31 × 0·21 mm, Syntex $P2_1$ diffractometer, graphite-monochromated Cu *K*α radiation. Unit cell from least-squares fit to 15 reflections ($13 \le 2\theta \le 29°$); 2θ–θ mode, variable scan speed: 2·0 to 29·3° min^{-1}, two control reflections monitored every 50 measurements; 1027 reflections measured up to $2\theta = 115°$; index range *h* 0/12, *k* 0/13 and *l* 0/9; profile analysis method of Lehmann & Larsen (1974), 993 independent reflections ($R_{\text{int}} = 0{\cdot}025$) with $I \ge 1{\cdot}96\sigma_I$; Lp corrections. Structure solved with *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980); H atoms from difference Fourier map, only their isotropic temperature factors were initially refined, but then fixed in the final cycles. The structure refinements were carried out for both enantiomorphs – the presented enantiomorph was chosen in the test of the *R*-factor ratio: $\mathscr{R} = wR_q/wR_0 = 1{\cdot}164$ compared to $\mathscr{R}_{171,820,0.01} = 1{\cdot}153$

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Table 1. *Final fractional coordinates and equivalent isotropic thermal parameters of non-*H *atoms*

$U_{eq} = (U_{11}U_{22}U_{33})^{1/3}$.

	x	y	z	U_{eq}(Å^2)
O(1)	0·4728 (2)	0·2202 (2)	1·0030 (2)	0·050
C(2)	0·5634 (3)	0·1505 (3)	1·0583 (4)	0·052
C(3)	0·6732 (3)	0·2061 (2)	1·1172 (3)	0·044
C(4)	0·6466 (2)	0·3026 (2)	1·2056 (4)	0·041
C(5)	0·6094 (3)	0·3970 (2)	1·1189 (3)	0·047
O(5)	0·6066 (3)	0·4834 (2)	1·1747 (3)	0·066
C(6)	0·6029 (3)	0·4640 (3)	0·8603 (4)	0·057
C(7)	0·5638 (3)	0·4601 (3)	0·7126 (4)	0·057
C(8)	0·4971 (3)	0·3752 (3)	0·6653 (4)	0·055
C(9)	0·4715 (2)	0·2939 (3)	0·7606 (4)	0·052
C(10)	0·5103 (2)	0·2982 (3)	0·9076 (3)	0·042
C(11)	0·5758 (2)	0·3835 (2)	0·9593 (3)	0·044
C(12)	0·6619 (3)	0·3142 (2)	1·3516 (3)	0·042
C(13)	0·6973 (3)	0·2364 (2)	1·4648 (3)	0·042
C(14)	0·7723 (3)	0·2678 (2)	1·5797 (4)	0·049
C(15)	0·8080 (3)	0·1987 (3)	1·6891 (4)	0·054
C(16)	0·7654 (3)	0·0957 (3)	1·6868 (4)	0·058
C(17)	0·6886 (3)	0·0647 (3)	1·5747 (4)	0·055
C(18)	0·6557 (3)	0·1354 (2)	1·4637 (4)	0·049

(Hamilton, 1974). Minimization of $\sum w(|F_o| - |F_c|)^2$ where $w = [\sigma^2(F_o) + 0{\cdot}0075F_o^2]^{-1}$, final $R = 0{\cdot}047$, $wR = 0{\cdot}055$, $S = 0{\cdot}84$, max. $\Delta/\sigma = 0{\cdot}10$, max. and min. densities in final difference Fourier map: 0·18 and −0·19 e Å^{-3}. One strong low-angle reflection omitted in the last cycles of refinement. The final atomic parameters are listed in Table 1.* Scattering factors including real and imaginary parts of anomalous dispersion from *International Tables for X-ray Crystallography* (1974); Riad-32 computer and *SHELX76* program (Sheldrick, 1976).

Discussion. Fig. 1 shows a perspective view of the molecule of (I) with the atom numbering, bond lengths and valency angles. The bond lengths in the 1,3-enone system are in very good agreement with those observed for 3-benzylidene-4-chromanone (Katrusiak, Ratajczak-Sitarz, Kałuski & Orlov, 1987) and indicate a good localization of the single and double bonds (Allmann, 1977). The torsion angles are listed in Table 2. There are no conformationally significant distortions from planarity in the two phenyl rings: χ^2 for ring C(6)–C(7)–C(8)–C(9)–C(10)–C(11) is 22·4 and for ring C(13)–C(14)–C(15)–C(16)–C(17)–C(18) −28·7. The tetrahydrooxepin ring has a distorted twist-boat conformation (Bucourt, 1974) with its element of pseudo-symmetry – a twofold axis – passing through C(2) and between C(5) and C(11). C(3) is the furthest atom from the plane fitted to the adjacent phenyl ring, 1·601 (3) Å, the height of the other atoms of the tetrahydrooxepin ring above this plane are: O(1) −0·114 (2), C(2) 1·030 (3), C(4) 0·566 (3) and C(5) −0·144 (3) Å. In this conformation, bonds C(2)—C(3) and C(3)—C(4) lie in the plane almost perpendicular to the axis of the molecule and consequently the H atoms bonded to C(3) are directed outwards (see Fig. 1), not towards the benzylidene substituent, as they are in 3-benzylidene-4-chromanone (Katrusiak, Ratajczak-Sitarz, Kałuski & Orlov, 1987). The shortest intermolecular contact of H(18), bonded to C(18), is to C(3) of the tetrahydrooxepin ring and is 2·87 Å, which is commensurate with the sum of the van der Waals radii of these atoms (Kitaigorodski, 1976). Similar intramolecular contacts, but with the H atoms of the pyrone ring, were observed in 3-benzylidene-4-chromanone (Katrusiak, Ratajczak-Sitarz, Kałuski & Orlov, 1987) and with the H atoms of the hexenone ring in 2-benzylidene-1-tetralone (2-benzylidene-1,2,3,4-tetrahydro-1-naphthalenone) (Kałuski, Skrzypczak-Jankun, Orlov & Borovoi, 1978) and were considered as responsible for the large angle between the planes fitted to the phenyl rings observed in both those molecules. This angle is 20·2 (5)° for (I) compared to 59·1 (4)° in 3-benzylidene-4-chromanone and 52·9 (8)° in 2-benzylidene-1-tetralone. The main conformational difference between (I) and 3-benzylidene-4-chromanone or 2-benzylidene-1-tetralone in the region of the *cisoid* 1,3-enone bridge connecting two phenyl rings is

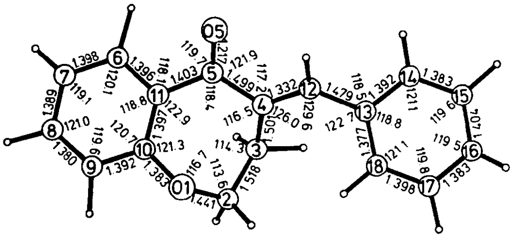

Fig. 1. A perspective view of the molecule of (I) with the bond distances and valency angles. The e.s.d.'s for the bond distances range from 0·004 to 0·005 Å, for the valency angles 0·2–0·3°.

Table 2. *Selected torsion angles* (°)

Oxepin ring			
O(1)–C(2)–C(3)–C(4)	40·7 (3)	C(5)–C(11)–C(10)–O(1)	−0·3 (3)
C(2)–C(3)–C(4)–C(5)	−75·0 (3)	C(11)–C(10)–O(1)–C(2)	−72·3 (3)
C(3)–C(4)–C(5)–C(11)	13·1 (3)	C(10)–O(1)–C(2)–C(3)	47·9 (3)
C(4)–C(5)–C(11)–C(10)	39·3 (4)		
Exocyclic torsion angles			
C(7)–C(6)–C(11)–C(5)	−174·0 (3)	C(12)–C(13)–C(14)–C(15)	179·7 (3)
C(6)–C(11)–C(5)–O(5)	33·5 (4)	C(17)–C(18)–C(13)–C(12)	−178·3 (3)
C(6)–C(11)–C(5)–C(4)	−146·1 (3)	C(18)–C(13)–C(12)–C(4)	−39·7 (4)
O(5)–C(5)–C(4)–C(12)	8·5 (4)	C(13)–C(12)–C(4)–C(3)	−5·9 (4)
C(11)–C(5)–C(4)–C(12)	−171·9 (3)	C(12)–C(4)–C(3)–C(2)	110·6 (3)
C(5)–C(4)–C(12)–C(13)	179·7 (3)	C(2)–O(1)–C(10)–C(9)	113·4 (3)
C(4)–C(12)–C(13)–C(14)	142·5 (4)	O(1)–C(10)–C(9)–C(8)	173·7 (3)

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* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43382 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

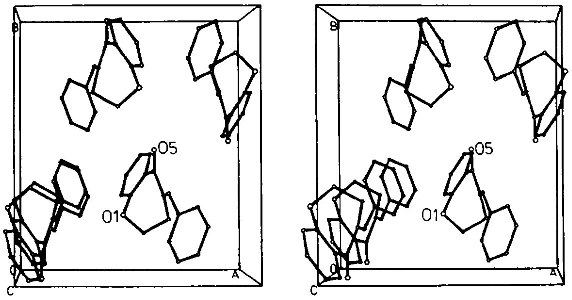

Fig. 2. A stereoscopic view of the unit-cell contents. H atoms are omitted for clarity.

observed in torsion angles O(5)—C(5)—C(11)—C(6) and O(5)—C(5)—C(4)—C(12) (Table 2) – the corresponding torsion angles in 3-benzylidene-4-chromanone are −0·7 (2) and −21·8 (2)° respectively. These two torsion angles are part of the tetrahydro-oxepin ring and presumably the strains imposed on this fragment of the 1,3-enone bridge by the seven-membered ring are responsible for their different magnitudes.

A stereoscopic view of the unit-cell contents (Motherwell & Clegg, 1978) is shown in Fig. 2. Only van der Waals intermolecular contacts are observed for this crystal.

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Structures of *N*-Trityl-L-aspartate Dibenzyl Ester and *N*-Trityl-L-leucine Benzyl Ester

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Abstract. $C_{37}H_{33}NO_4$ (I), $M_r = 555·7$, monoclinic, $P2_1$, $a = 11·211$ (7), $b = 7·983$ (4), $c = 17·582$ (14) Å, $\beta = 103·88$ (6)°, $V = 1527$ (2) Å^3, $Z = 2$, $D_x = 1·208$ (1) g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0·844$ cm^{-1}, $F(000) = 588$, room temperature, $R = 0·0479$ for 1544 unique observed reflections. $C_{32}H_{33}NO_2$ (II), $M_r = 463·7$, monoclinic, $C2$, $a = 28·531$ (12), $b = 11·730$ (3), $c = 16·885$ (10) Å, $\beta = 106·39$ (4)°, $V = 5421$ (4) Å^3, $Z = 8$, $D_x = 1·136$ (1) g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0·756$ cm^{-1}, $F(000) = 1984$, room temperature, $R = 0·0958$ with 2573 observed reflections. The structure of (I) clearly demonstrates that the α-benzyl ester approaches the bulky trityl group and this could explain

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