Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)*

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

	x	*y*	*z*	U_{eq}
N1	−0.0878 (2)	0.8283 (3)	−0.0201 (2)	0.0177 (6)
C2	−0.0158 (3)	0.8987 (3)	0.0608 (2)	0.0183 (8)
O2	0.0318 (2)	0.8405 (2)	0.1410 (2)	0.0232 (5)
N3	−0.0031 (3)	1.0298 (3)	0.0495 (2)	0.0179 (6)
C4	−0.0611 (3)	1.0916 (3)	−0.0364 (3)	0.0171 (7)
N4	−0.0627 (3)	1.2209 (3)	−0.0380 (2)	0.0273 (7)
C5	−0.1159 (3)	1.0206 (3)	−0.1265 (2)	0.0172 (7)
C6	−0.1295 (3)	0.8901 (3)	−0.1147 (2)	0.0163 (7)
C7	−0.1862 (3)	0.8253 (3)	−0.2145 (3)	0.0209 (8)
C8	−0.2018 (4)	0.9385 (3)	−0.2969 (3)	0.0291 (9)
C9	−0.1633 (3)	1.0651 (3)	−0.2384 (3)	0.0230 (8)
C1′	−0.1263 (4)	0.6926 (3)	−0.0010 (3)	0.0212 (8)
C2′	−0.0065 (4)	0.5982 (3)	0.0265 (3)	0.0218 (8)
C3′	−0.0480 (3)	0.5404 (3)	0.1370 (3)	0.0199 (7)
O3′	−0.0104 (2)	0.4071 (2)	0.1433 (2)	0.0277 (6)
O4′	−0.2240 (2)	0.6866 (2)	0.0869 (2)	0.0221 (6)
C4′	−0.2062 (3)	0.5622 (3)	0.1375 (3)	0.0204 (7)
C5′	−0.2788 (3)	0.5585 (3)	0.2481 (3)	0.0235 (8)
O5′	−0.2226 (2)	0.6426 (2)	0.3296 (2)	0.0268 (6)

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

C2—N1—C1′—O4′	64.4 (3)	C1′—O4′—C4′—C3′	39.6 (3)
O4′—C1′—C2′—C3′	0.1 (3)	C2′—C3′—C4′—O4′	−37.8 (3)
C1′—C2′—C3′—C4′	22.4 (3)	O4′—C4′—C5′—O5′	−65.5 (3)
C2′—C1′—O4′—C4′	−24.6 (3)	C3′—C4′—C5′—O5′	53.6 (4)

The assignment of absolute configuration was made on chemical grounds, with the base having a β-configuration with respect to the deoxyribose sugar.

Data collection: *MADNES* (Pflugrath & Messerschmidt, 1990); further details from Darr, Drake, Hursthouse & Malik (1993). Cell refinement: *MADNES*. Data reduction: *MADNES*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEX* (McArdle, 1993). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BM1048). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1996). C**52**, 2334–2337

(*E*)-2,2,5,5-Tetramethyl-3,4-bis[4-(tribromomethyl)phenyl]hex-3-ene

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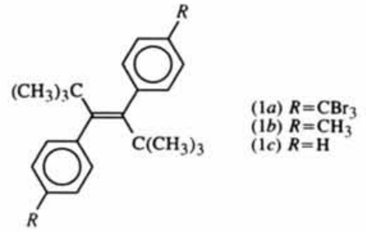
Abstract

Steric repulsion of the *tert*-butyl groups of the title compound, $C_{24}H_{26}Br_6$, causes the phenyl rings to rotate out of the plane of the central double bond eliminating the conjugation between the three π systems, yet the central double bond is normal, 1.33 (1) Å. The molecules pack together to maximize Br···Br and *tert*-butyl···*tert*-butyl interactions forming 'planes' of Br atoms and *tert*-butyl groups. The results are supplemented by MOPAC calculations.

Comment

Stilbenes bearing *tert*-butyl groups on the central C atoms have received significant attention because of their unusual geometry (Gano, Park, Pinkerton & Lenoir, 1990, 1991; Gano, Park, Subramaniam, Lenoir & Gleiter, 1991; Laali, Gano, Lenoir & Gundlach, 1994; Lenoir, Gano & McTague, 1986). Although the crystal structure of a *Z* isomer appeared some time ago (Gano, Park, Pinkerton & Lenoir, 1991), crystallographic information on the *E* isomers has proven to be elusive (Ermer, 1977). Herein is provided the first report of a crystallographic investigation of an (*E*)-di-*tert*-butylstilbene, (*E*)-2,2,5,5-tetramethyl-3,4-bis[4-(tribromomethyl)phenyl]hex-3-ene, (1*a*). Stilbene (1*a*) was prepared by bromination of stilbene (1*b*), whose preparation followed the procedure for preparation of the parent stilbene (1*c*) (Lenoir *et al.*, 1986).

Recrystallization of (1*a*) from chloroform produced crystals suitable for X-ray diffraction measurements. Although the results immediately suggested that additional rotational isomers were needed to accommo-



date disorder, the number of necessary conformations was not clear. This problem was solved by plotting the electron density across a plane perpendicular to the *tert*-butyl axis. That clearly demonstrated that only two rotamers with 0.5:0.5 occupancy ratio, related by *ca* 50° rotation, were observed.

The features of the tribromomethyl group are of some interest. Of the four compounds bearing the tribromomethyl group whose crystal structures have been reported (Hovmoeller, Smith & Kennard, 1978; Irving & Irving, 1986; Mandel & Donohue, 1972; Porter & Doedens, 1984; Snaauw & Wiebenga, 1942), none have the group attached to an aromatic ring, as does (1*a*). One structure has it attached to a carbonyl group (Porter & Doedens, 1984). In (1*a*), one C—Br bond is almost 'perpendicular' to the ring, angle C(8)—C(7)—C(12)—Br(3) = 75 (1)°, and one is almost coplanar, angle C(8)—C(7)—C(12)—Br(2) = −164 (1)°. MOPAC calculations on tribromomethylbenzene (Dewar, Zoebisch, Healy & Stewart, 1985) show a very small rotational barrier, 0.2 kcal mol^{-1} (1 kcal mol^{-1} = 4.184 kJ mol^{-1}), about the sixfold axis and a preferred conformation where the Br atom is essentially eclipsed with the adjacent ring. It is thus not surprising that MOPAC calculations on (1*a*) reveal a series of conformations of very similar energies, range 0.25 kcal mol^{-1}, and differing CBr_3 rotational angles.

The general features of (1*a*) are depicted in Fig. 1. As expected from earlier work and computational studies, the phenyl groups rotate to a position perpendicular to the molecular plane, C(3′)—C(3)—C(4)—C(5) = 90 (1)°, in order to avoid the large *tert*-butyl groups. In addition, the C═C—^{t}Bu angle is opened, C(3′)—C(3)—C(2) = 128.9 (8)°, the Me_3C—C bond is stretched, C(2)—C(3) = 1.53 (2) Å, and the Ph—C bond is stretched, C(3)—C(4) = 1.54 (1) Å.

The central C═C bond is planar and of normal length, C(3)—C(3′) = 1.33 (1) Å, for an unconjugated C═C bond (Ogawa, Harada & Tomoda, 1995).

The bromomethyl group lies −0.15 (1) Å below the plane of the phenyl ring on the side of the Br atom most bent into and conjugated with the ring π orbitals (Fig. 1*b*), a feature also reflected in the calculated structure.

The features of the molecular packing appear to be dominated by the strong Br···Br interactions (Desiraju, 1989). The molecular packing is characterized by ^{t}Bu···^{t}Bu, CBr_3···CBr_3 and Br···Ph interactions,

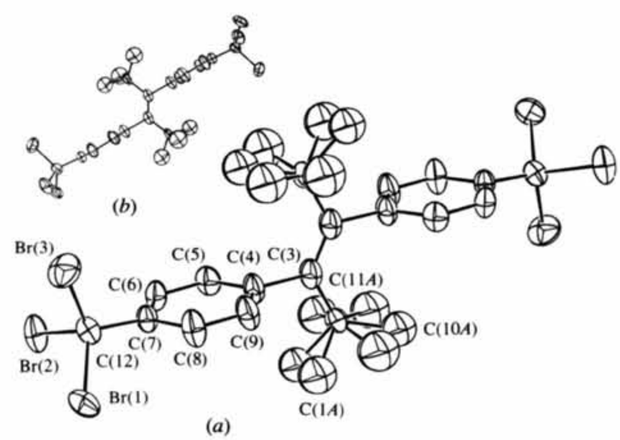


Fig. 1. *ORTEP*II plots (Johnson, 1976), 50% probability, showing (*E*)-2,2,5,5-tetramethyl-3,4-bis[4-(tribromomethyl)phenyl]hex-3-ene, (1*a*): (*a*) Molecular orientation selected to reveal atomic numbering. The crystal was disordered by rotation of the *tert*-butyl group. (*b*) Molecular orientation selected to emphasize the perpendicular relationship between the benzene rings and central π bond.

Fig. 2. Each molecule interlocks its CBr_3 group into a 'plane' of interlocking CBr_3 groups allowing each Br atom to maximize the number of Br···Br contacts. The 'plane' of interlocking tribromomethyl groups is shown separately in Fig. 2(*a*). Although the other reported structures for tribromomethyl compounds seem to maximize the Br···Br contacts, none shows a plane of Br atoms. Each phenyl ring has a close contact, Fig. 2(*b*), with a Br atom lying above it, C(6)—Br(3^{ii}) = 3.4 Å [symmetry operation: (ii) $x-\frac{1}{2}, \frac{1}{2}-y, z-\frac{1}{2}$]. Finally, a second plane through the crystal is formed by *tert*-butyl groups which pile upon one another like stacked cans, Fig. 2(*b*). Presumably these groups do not fit tightly together, thus accounting for the considerable disorder. The aromatic rings do not approach one another due, presumably, to steric restrictions.

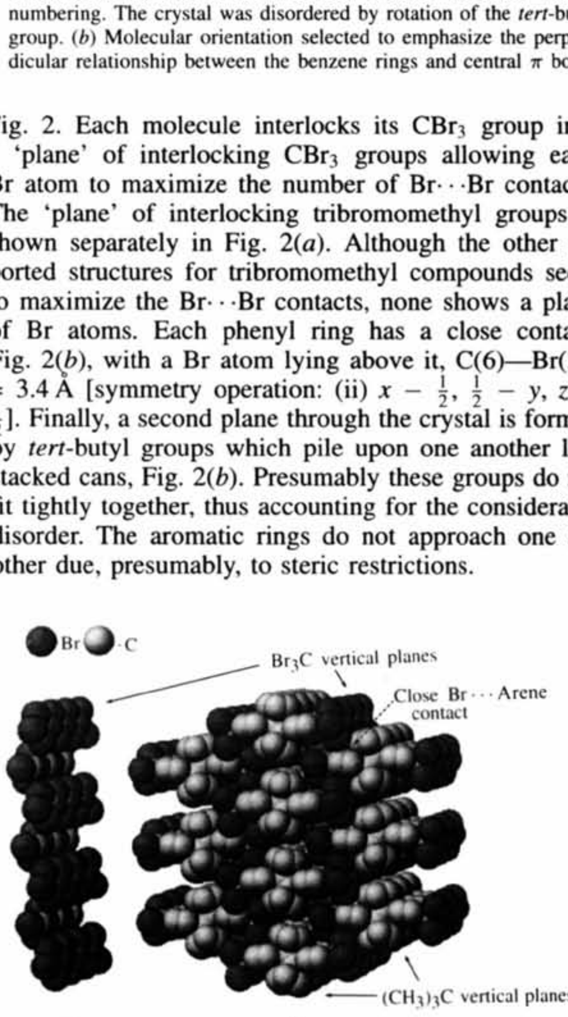


Fig. 2. Representation of the packing of (*E*)-2,2,5,5-tetramethyl-3,4-bis[4-(tribromomethyl)phenyl]hex-3-ene, (1*a*): (*a*) a copy of the left 'planes' of tribromomethyl groups in crystal (*b*) moved left out of the crystal to show the bromine interactions; (*b*) crystal with molecules selectively removed to show crystal planes and packing.

Experimental

The title compound was prepared by bromination of (*E*)-2,2,5,5-tetramethyl-3,4-bis(4-methylphenyl)hex-3-ene (Gano & Sekher, 1996) and recrystallized from chloroform.

Crystal data

$C_{24}H_{26}Br_6$
$M_r = 793.93$
Monoclinic
$P2_1/n$
$a = 6.283$ (1) Å
$b = 22.113$ (2) Å
$c = 9.691$ (2) Å
$\beta = 91.10$ (1)°
$V = 1346.2$ (6) Å^3
$Z = 2$
$D_x = 1.96$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 6$–11°
$\mu = 8.874$ mm^{-1}
$T = 294$ K
Plate
0.50 × 0.10 × 0.05 mm
Light orange

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical *via* ψ scans (North, Phillips & Mathews, 1968)
$T_{min} = 0.4200$, $T_{max} = 0.9980$
2962 measured reflections
2717 independent reflections

1187 observed reflections [$I > 3.0\sigma(I)$]
$R_{int} = 0.083$
$\theta_{max} = 25.97°$
$h = 0 \rightarrow 7$
$k = 0 \rightarrow 27$
$l = -11 \rightarrow 11$
3 standard reflections
frequency: 50 min
intensity decay: 1.0%

Refinement

Refinement on F
$R = 0.056$
$wR = 0.061$
$S = 1.732$
1187 reflections
134 parameters
H atoms riding, $U = 1.3 \times U$(bonding atom)
$w = 4F_o^2/[\sigma^2(F_o^2) + 0.0016 F_o^4]$

$(\Delta/\sigma)_{max} = 0.005$
$\Delta\rho_{max} = 0.76$ e Å^{-3}
$\Delta\rho_{min} = -0.87$ e Å^{-3}
Extinction correction: isotropic (Zachariasen, 1963)
Extinction coefficient: 0.57×10^{-6}
Atomic scattering factors from Cromer (1974)

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}
Br(1)	0.0970 (2)	0.17773 (6)	0.8759 (2)	0.0653 (4)
Br(2)	−0.3292 (2)	0.18624 (5)	1.0357 (2)	0.0644 (4)
Br(3)	0.1060 (2)	0.20567 (6)	1.1945 (2)	0.0611 (4)
C(2)	−0.092 (2)	0.5027 (5)	0.800 (1)	0.047 (3)
C(3)	−0.029 (2)	0.4830 (5)	0.946 (1)	0.043 (4)
C(4)	−0.033 (2)	0.4140 (5)	0.967 (1)	0.042 (3)
C(5)	−0.216 (2)	0.3858 (4)	1.009 (1)	0.042 (3)
C(6)	−0.225 (2)	0.3234 (4)	1.022 (1)	0.041 (3)
C(7)	−0.050 (1)	0.2879 (4)	0.995 (1)	0.033 (3)
C(8)	0.131 (2)	0.3160 (5)	0.949 (1)	0.049 (4)
C(9)	0.140 (2)	0.3784 (5)	0.936 (1)	0.051 (4)
C(12)	−0.048 (2)	0.2214 (5)	1.022 (1)	0.040 (3)
C(1*A*)	−0.041 (4)	0.453 (1)	0.694 (3)	0.09 (1)
C(10*A*)	0.010 (4)	0.558 (1)	0.731 (3)	0.076 (9)
C(11*A*)	−0.338 (4)	0.513 (1)	0.800 (3)	0.10 (1)
C(1*B*)	−0.210 (4)	0.452 (1)	0.715 (3)	0.074 (8)
C(10*B*)	0.117 (4)	0.512 (1)	0.715 (4)	0.10 (1)
C(11*B*)	−0.209 (4)	0.562 (1)	0.776 (3)	0.09 (1)

Table 2. *Geometric parameters (Å, °)*

Br(1)—C(12)	1.96 (1)	C(3)—C(4)	1.54 (1)
Br(2)—C(12)	1.94 (1)	C(3)—C(3^i)	1.33 (1)
Br(3)—C(12)	1.94 (1)	C(4)—C(5)	1.38 (1)
C(2)—C(3)	1.53 (2)	C(4)—C(9)	1.38 (1)
C(2)—C(1*A*)	1.54 (3)	C(5)—C(6)	1.39 (1)
C(2)—C(10*A*)	1.53 (3)	C(6)—C(7)	1.38 (1)
C(2)—C(11*A*)	1.57 (3)	C(7)—C(8)	1.37 (1)
C(2)—C(1*B*)	1.56 (3)	C(7)—C(12)	1.50 (1)
C(2)—C(10*B*)	1.58 (3)	C(8)—C(9)	1.39 (1)
C(2)—C(11*B*)	1.51 (3)		
C(3)—C(2)—C(1*A*)	111 (1)	C(3)—C(4)—C(9)	121.6 (9)
C(3)—C(2)—C(10*A*)	122 (1)	C(5)—C(4)—C(9)	118.1 (9)
C(3)—C(2)—C(11*A*)	106 (1)	C(4)—C(5)—C(6)	120.8 (9)
C(3)—C(2)—C(1*B*)	113 (1)	C(5)—C(6)—C(7)	121.0 (9)
C(3)—C(2)—C(10*B*)	108 (1)	C(6)—C(7)—C(8)	118.2 (9)
C(3)—C(2)—C(11*B*)	120 (1)	C(6)—C(7)—C(12)	121.8 (9)
C(1*A*)—C(2)—C(10*A*)	101 (2)	C(8)—C(7)—C(12)	119.8 (9)
C(1*A*)—C(2)—C(11*A*)	109 (2)	C(7)—C(8)—C(9)	120.9 (9)
C(10*A*)—C(2)—C(11*A*)	108 (2)	C(4)—C(9)—C(8)	121 (1)
C(1*B*)—C(2)—C(10*B*)	102 (2)	Br(1)—C(12)—Br(2)	106.8 (5)
C(1*B*)—C(2)—C(11*B*)	108 (2)	Br(1)—C(12)—Br(3)	107.6 (5)
C(10*B*)—C(2)—C(11*B*)	103 (2)	Br(1)—C(12)—C(7)	111.0 (7)
C(2)—C(3)—C(4)	113.2 (9)	Br(2)—C(12)—Br(3)	108.1 (5)
C(2)—C(3)—C(3^i)	128.9 (8)	Br(2)—C(12)—C(7)	113.7 (7)
C(4)—C(3)—C(3^i)	117.9 (9)	Br(3)—C(12)—C(7)	109.4 (7)
C(3)—C(4)—C(5)	120.2 (9)		

C(1*A*)—C(2)—C(3)—C(4)	−27 (2)
C(1*A*)—C(2)—C(3)—C(3^i)	152 (1)
C(10*A*)—C(2)—C(3)—C(4)	−145 (1)
C(10*A*)—C(2)—C(3)—C(3^i)	34 (2)
C(11*A*)—C(2)—C(3)—C(4)	91 (1)
C(11*A*)—C(2)—C(3)—C(3^i)	−89 (2)
C(1*B*)—C(2)—C(3)—C(4)	17 (2)
C(1*B*)—C(2)—C(3)—C(3^i)	−164 (1)
C(10*B*)—C(2)—C(3)—C(4)	−95 (2)
C(10*B*)—C(2)—C(3)—C(3^i)	84 (2)
C(11*B*)—C(2)—C(3)—C(4)	147 (1)
C(11*B*)—C(2)—C(3)—C(3^i)	−34 (2)
C(2)—C(3)—C(4)—C(5)	−91 (1)
C(2)—C(3)—C(4)—C(9)	86 (1)
C(3^i)—C(3)—C(4)—C(5)	90 (1)
C(3^i)—C(3)—C(4)—C(9)	−93 (1)
C(3)—C(4)—C(5)—C(6)	177 (1)
C(9)—C(4)—C(5)—C(6)	0 (2)
C(3)—C(4)—C(9)—C(8)	−177 (1)
C(5)—C(4)—C(9)—C(8)	0 (2)
C(4)—C(5)—C(6)—C(7)	1 (2)
C(5)—C(6)—C(7)—C(8)	−2 (2)
C(5)—C(6)—C(7)—C(12)	173 (1)
C(6)—C(7)—C(8)—C(9)	2 (2)
C(12)—C(7)—C(8)—C(9)	−173 (1)
C(6)—C(7)—C(12)—Br(1)	141 (1)
C(6)—C(7)—C(12)—Br(2)	21 (1)
C(6)—C(7)—C(12)—Br(3)	−100 (1)
C(8)—C(7)—C(12)—Br(1)	−44 (1)
C(8)—C(7)—C(12)—Br(2)	−164 (1)
C(8)—C(7)—C(12)—Br(3)	75 (1)
C(7)—C(8)—C(9)—C(4)	−1 (2)

Symmetry code: (i) $-x, 1-y, 2-z$.

To accommodate disorder, the solution included two conformations for the *tert*-butyl groups. The atoms of the disordered group were refined isotropically with an occupancy factor of 0.5. H atoms were calculated on ideal positions and included in the refinement as riding atoms with $U_{iso} = 1.3 \times U$(bonding atoms).

Data collection: *CAD*-4 (Enraf–Nonius, 1977). Cell refinement: *CAD*-4. Data reduction: *PROCESS MolEN* (Fair, 1990). Program(s) used to solve structure: *MULTAN* (Main *et al.*, 1980). Program(s) used to refine structure: *LSFM MolEN*. Molecular graphics: *CAChe* (CAChe Scientific, 1993), *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *CIF VAX MolEN*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1154). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Racemic α,α'-*o*-Xylylene-1,1′-bis[(3,5-dimethyl-5,6-dihydro-1,3,5-triazine-6-spiro-9′-fluorene)-2,4(1*H*,3*H*)-dione]

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Abstract

The title compound, $C_{42}H_{36}N_6O_4$, was synthesized *via* a sequence of reactions in which the product of the reaction between the potassium salt of 1,4-dimethylurazole and 9-bromofluorene was allowed to react with (*a*) potassium *tert*-butoxide and (*b*) *o*-$(ClCH_2)_2C_6H_4$. The X-ray crystal structure of the title compound features a tilted T-shaped edge-to-face aromatic interaction between the two fluorene moieties and the resultant molecular distortion produces chirality which leads to the formation of a racemic structure. The distortion also causes marked differences in the conformations of the two half-chair triazinedione rings. Intermolecular C—H···O interactions produce infinite cross-linked double chains of hydrogen-bonded molecules.

Comment

The present study investigates the effects of aromatic interactions between large aromatic rings. Although such interactions are not unexpected, examples are rarely seen. Non-covalent interactions are known to be important in protein folding and molecular recognition. Interactions between aromatic moieties have been postulated as important factors in protein stabilization (Burley & Petsko, 1985). Stabilizing non-covalent interactions between aromatic moieties have been observed in *cis*-1,4-dihydro-4-tritylbiphenyl and its 4′-bromo derivative (Grossel, Cheetham, Hope & Weston, 1993), dibenzodiazocine esters (Paliwal, Geib & Wilcox, 1994) and a trianilide derivative (Yamaguchi *et al.*, 1991). Calculations aimed at understanding interactions between aromatic rings suggest that benzene rings favor a slightly tilted T-shaped edge-to-face aromatic interaction, with a centroid–centroid distance of 5.5 Å (Jorgensen & Severance, 1990).

The molecular structure of the title compound, (1) (synthesized as shown below), consists of two triazinedione-spiro-fluorene moieties, connected through