

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), *ORTEPII* (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 $\alpha,\alpha'$ -*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

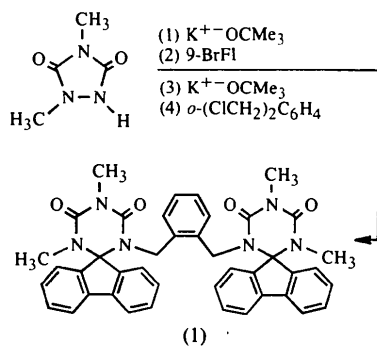
The title compound, C<sub>42</sub>H<sub>36</sub>N<sub>6</sub>O<sub>4</sub>, was synthesized via a sequence of reactions in which the product of the reaction between the potassium salt of 1,4-dimethylurazole and 9-bromofluorene was allowed to react with (a) potassium *tert*-butoxide and (b) *o*-(ClCH<sub>2</sub>)<sub>2</sub>C<sub>6</sub>H<sub>4</sub>. 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 & Sevrance, 1990).

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

an *o*-xylylene linkage. The molecule is flexible owing to the presence of two benzylic C atoms and thus can be used as a sensitive probe of weak molecular forces.



The X-ray structure of (1), with an appropriate atom-numbering scheme, is shown in Fig. 1. Inspection of the structure reveals that the two triazinedione-spirofluorene (TSF) portions (which reside on the left and

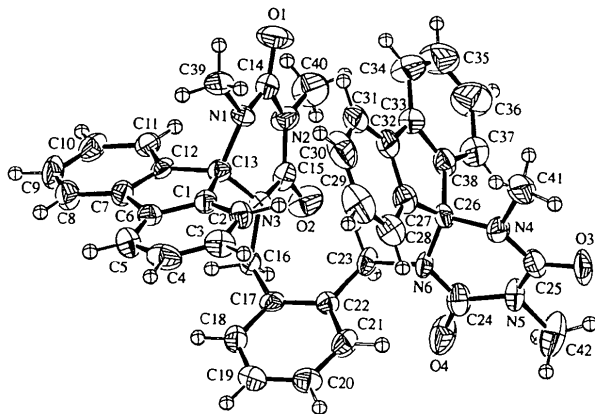


Fig. 1. The molecular configuration and atom-numbering scheme for (1), with displacement ellipsoids at the 50% probability level. H atoms are shown as isotropic spheres of arbitrary radius.

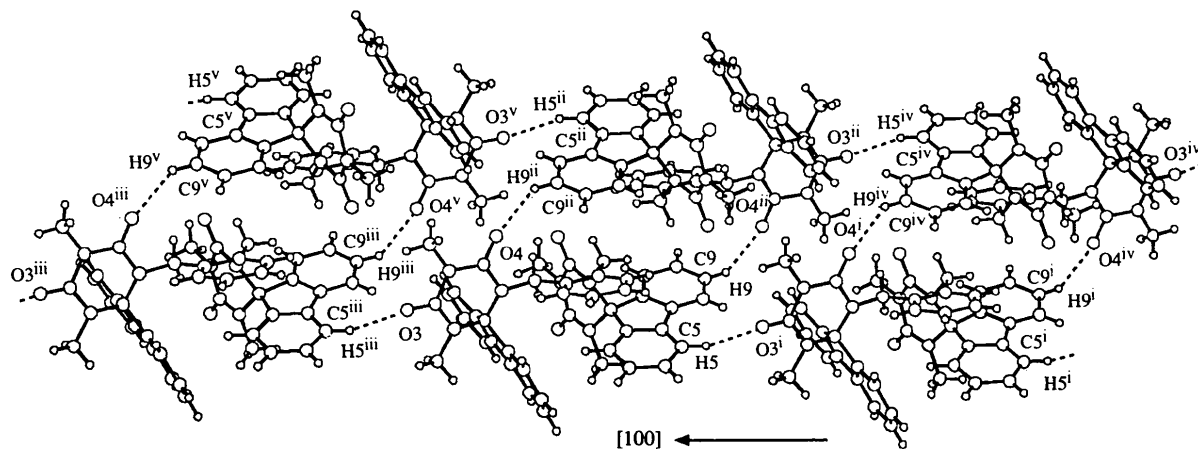


Fig. 2. The hydrogen-bonding scheme for (1) showing infinite cross-linked double chains propagating parallel to [100] [symmetry codes: (i)  $x-1, y, z$ ; (ii)  $1-x, -y, 2-z$ ; (iii)  $1+x, y, z$ ; (iv)  $-x, -y, 2-z$ ; (v)  $2-x, -y, 2-z$ ].

right sides of Fig. 1) are asymmetrical. Specifically, the left TSF portion is folded and atom N3 is out of the xylene plane [torsion angle N3—C16—C17—C18 127.1(6)°], while the right TSF portion is extended and N6 is in the xylene plane [C21—C22—C23—N6 -3.1(9)°]. The angles between the fluorene plane and the xylene-benzene plane are 20.7(3) and 64.2(3)° on the left and right sides, respectively.

Two lines of evidence support the notion that the two fluorene rings in (1) assemble into a tilted T-geometry (for an explanation of the tilted T-geometry see Paliwal, Geib & Wilcox, 1994). Firstly, the centroid-centroid distance between the nearest benzene rings within the left- and right-hand fluorenyl moieties (Fig. 1) is 4.853(5) Å, a value in reasonable agreement with the theoretically derived centroid-centroid distance of *ca* 5.5 Å between two interacting benzene molecules (Jorgensen & Severance, 1990). Also, comparisons of the distance between the C2 atom and the centroid of the nearest benzene ring in the right-hand portion of (1) [3.503(8) Å], as well as the distance between atom H2 and the centroid of the right-hand portion of (1) (2.62 Å) (also referred to as a  $\pi$ -hydrogen bond; Cochran, Parrott, Whitlock & Whitlock, 1992), are in good agreement with literature values for analogous distances within *cis*-1,4-dihydro-4-tritylbiphenyl (3.55 and 2.57 Å, respectively; Grossel, Cheetham, Hope & Weston, 1993). Secondly, the edge of the left-hand fluorene ring points towards the face of its right-hand counterpart and the angle between the planes of the two fluorene rings is 69.05(18)°. Published literature (Grossel, Cheetham, Hope & Weston, 1993) indicates that analogous angles greater than 50°, within a separation distance between 3.4 and 6.5 Å, demonstrates the existence of intramolecular edge-to-face aromatic interactions.

Further structural examination indicates that the two triazinedione rings within (1) adopt half-chair conformations, with the spiro-C13 and spiro-C26 atoms displaced from their respective planes as defined by the

other five members of each heterocycle. The triazine-dione ring on the right (Fig. 1) is significantly more flattened than its counterpart on the left, as reflected by the C26—N4—C25—N5 and C13—N1—C14—N2 torsion angles of  $-13.4$  (9) and  $-21.7$  (9) $^\circ$ , respectively. In the absence of the proposed aromatic edge-to-face interaction, these two torsion angles would be expected to be equal. The difference in torsion angles is therefore additional evidence for the proposed edge-to-face interactions between the left- and right-side fluorene rings.

Aromatic interactions in compound (1) cause molecular distortions which produce chirality and, in this case, result in the formation of a conformationally racemic compound. Molecular chirality was also shown to be present in 1,2-bis(*N*-benzoyl-*N*-methylamino)-benzene and is expected in all compounds with restricted conformational interconversion (Azumaya, Yamaguchi, Okamoto, Kagechika & Shudo, 1995).

The hydrogen-bonding scheme for (1) is shown in Fig. 2. Each molecule is hydrogen-bonded to two molecules of identical chirality and one molecule of opposite chirality *via* weak C—H $\cdots$ O intermolecular interactions. The result is an infinite cross-linked double chain of molecules propagating in the [100] direction. The hydrogen-bond geometry is given in Table 3.

## Experimental

Compound (1) was prepared from 1,4-dimethylurazole in four sequential steps, namely, deprotonation with potassium *tert*-butoxide in dimethyl sulfoxide, treatment with 9-bromo-fluorene, base-promoted ring expansion and substitution with  $\alpha,\alpha'$ -dichloro-*o*-xylene. The desired product precipitated from the dimethyl sulfoxide solution with an overall yield of 90% and was recrystallized from acetone–chloroform solution (m.p. 576–578 K). X-ray quality racemic crystals were obtained *via* slow evaporation of an ethanol–chloroform solution.

### Crystal data

$C_{42}H_{36}N_6O_4$	Mo $K\alpha$ radiation
$M_r = 688.78$	$\lambda = 0.71069 \text{ \AA}$
Monoclinic	Cell parameters from 25 reflections
$P2_1/n$	$\theta = 10.0\text{--}10.4^\circ$
$a = 14.153$ (6) $\text{\AA}$	$\mu = 0.081 \text{ mm}^{-1}$
$b = 14.054$ (4) $\text{\AA}$	$T = 296 \text{ K}$
$c = 17.959$ (4) $\text{\AA}$	Plate
$\beta = 102.90$ (2) $^\circ$	$0.45 \times 0.41 \times 0.07 \text{ mm}$
$V = 3482$ (2) $\text{\AA}^3$	Colorless
$Z = 4$	
$D_x = 1.314 \text{ Mg m}^{-3}$	
$D_m$ not measured	

### Data collection

Rigaku AFC-5S diffractometer	2177 observed reflections
$\omega$ scans (rate $4^\circ \text{ min}^{-1}$ in $\omega$ , 3 repetitions maximum)	$[I > 1.75\sigma(I)]$
	$R_{\text{int}} = 0.052$
	$\theta_{\text{max}} = 25^\circ$

Absorption correction:  
 $\psi$  scans (North, Phillips & Mathews, 1968)  
 $T_{\text{min}} = 0.97$ ,  $T_{\text{max}} = 1.00$   
 6685 measured reflections  
 6411 independent reflections

$h = 0 \rightarrow 16$   
 $k = 0 \rightarrow 16$   
 $l = -21 \rightarrow 21$   
 3 standard reflections monitored every 150 reflections  
 intensity decay: 1.8%

### Refinement

Refinement on  $F$   
 $R = 0.063$   
 $wR = 0.056$   
 $S = 1.57$   
 2177 reflections  
 469 parameters  
 H-atom parameters not refined (riding with C—H = 0.95  $\text{\AA}$ )  
 $w = 4F_o^2/\sigma^2(F_o^2)$

$(\Delta/\sigma)_{\text{max}} = 0.0002$   
 $\Delta\rho_{\text{max}} = 0.31 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.32 \text{ e \AA}^{-3}$   
 Extinction correction: none  
 Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.3.1)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	$x$	$y$	$z$	$U_{\text{eq}}$
O1	0.5750 (4)	0.2901 (4)	0.7694 (3)	0.072 (3)
O2	0.6636 (4)	0.0263 (4)	0.9121 (3)	0.061 (2)
O3	1.1846 (3)	0.2464 (4)	1.1232 (3)	0.059 (2)
O4	0.9480 (3)	0.0325 (4)	1.1270 (3)	0.067 (2)
N1	0.5465 (4)	0.2855 (4)	0.8886 (3)	0.035 (2)
N2	0.6099 (4)	0.1541 (5)	0.8378 (3)	0.046 (3)
N3	0.5946 (3)	0.1485 (4)	0.9650 (3)	0.0294 (19)
N4	1.0289 (4)	0.2925 (4)	1.0808 (3)	0.043 (2)
N5	1.0672 (4)	0.1339 (4)	1.1133 (3)	0.042 (2)
N6	0.9037 (4)	0.1811 (4)	1.0834 (3)	0.034 (2)
C1	0.5111 (5)	0.2787 (5)	1.0173 (4)	0.031 (3)
C2	0.5808 (5)	0.3328 (5)	1.0634 (4)	0.037 (3)
C3	0.5579 (5)	0.3775 (5)	1.1261 (4)	0.043 (3)
C4	0.4661 (6)	0.3675 (6)	1.1402 (4)	0.054 (3)
C5	0.3951 (5)	0.3141 (5)	1.0926 (4)	0.045 (3)
C6	0.4187 (5)	0.2692 (5)	1.0309 (4)	0.034 (3)
C7	0.3598 (5)	0.2101 (5)	0.9704 (4)	0.036 (3)
C8	0.2642 (5)	0.1815 (5)	0.9583 (4)	0.048 (3)
C9	0.2265 (5)	0.1242 (6)	0.8967 (5)	0.055 (3)
C10	0.2831 (5)	0.0951 (5)	0.8470 (4)	0.048 (3)
C11	0.3796 (5)	0.1246 (5)	0.8587 (4)	0.039 (3)
C12	0.4167 (4)	0.1812 (4)	0.9204 (4)	0.032 (3)
C13	0.5199 (4)	0.2238 (5)	0.9460 (4)	0.034 (3)
C14	0.5754 (5)	0.2476 (6)	0.8275 (5)	0.046 (3)
C15	0.6241 (5)	0.1039 (6)	0.9058 (4)	0.042 (3)
C16	0.5943 (4)	0.0891 (5)	1.0324 (4)	0.036 (3)
C17	0.6484 (5)	0.1281 (4)	1.1095 (4)	0.031 (2)
C18	0.5984 (5)	0.1330 (5)	1.1672 (4)	0.037 (3)
C19	0.6434 (5)	0.1604 (5)	1.2404 (4)	0.046 (3)
C20	0.7408 (6)	0.1823 (5)	1.2559 (4)	0.046 (3)
C21	0.7920 (5)	0.1794 (5)	1.1991 (4)	0.043 (3)
C22	0.7465 (5)	0.1529 (4)	1.1250 (4)	0.029 (2)
C23	0.8012 (4)	0.1496 (5)	1.0619 (4)	0.037 (3)
C24	0.9701 (5)	0.1116 (6)	1.1084 (4)	0.046 (3)
C25	1.0985 (5)	0.2269 (6)	1.1062 (4)	0.044 (3)
C26	0.9283 (4)	0.2674 (5)	1.0460 (4)	0.036 (3)
C27	0.8614 (5)	0.3498 (5)	1.0540 (4)	0.040 (3)
C28	0.8509 (5)	0.3933 (6)	1.1206 (4)	0.050 (3)
C29	0.7922 (7)	0.4745 (6)	1.1135 (6)	0.067 (4)
C30	0.7477 (6)	0.5076 (6)	1.0437 (7)	0.072 (4)
C31	0.7572 (6)	0.4649 (6)	0.9770 (5)	0.061 (3)
C32	0.8154 (5)	0.3849 (6)	0.9823 (4)	0.046 (3)
C33	0.8444 (5)	0.3277 (6)	0.9233 (4)	0.049 (3)
C34	0.8206 (6)	0.3338 (7)	0.8441 (5)	0.074 (4)
C35	0.8651 (8)	0.2705 (9)	0.8026 (5)	0.091 (5)
C36	0.9309 (8)	0.2041 (8)	0.8386 (5)	0.088 (4)
C37	0.9535 (5)	0.1958 (6)	0.9168 (5)	0.059 (3)

C38	0.9101 (5)	0.2589 (6)	0.9588 (4)	0.042 (3)
C39	0.5014 (5)	0.3797 (5)	0.8780 (4)	0.049 (3)
C40	0.6399 (6)	0.1076 (6)	0.7736 (4)	0.078 (4)
C41	1.0626 (5)	0.3851 (6)	1.0595 (4)	0.067 (3)
C42	1.1410 (5)	0.0626 (6)	1.1442 (5)	0.078 (4)

Table 2. Selected geometric parameters (Å, °)

O1—C14	1.201 (10)	N3—C15	1.377 (9)
O2—C15	1.219 (10)	N3—C16	1.471 (9)
O3—C25	1.219 (9)	N4—C25	1.352 (10)
O4—C24	1.223 (10)	N4—C26	1.465 (8)
N1—C13	1.459 (9)	N4—C41	1.466 (10)
N1—C14	1.362 (10)	N5—C24	1.392 (9)
N1—C39	1.464 (9)	N5—C25	1.396 (10)
N2—C14	1.400 (11)	N5—C42	1.463 (10)
N2—C15	1.386 (9)	N6—C23	1.483 (8)
N2—C40	1.468 (10)	N6—C24	1.359 (10)
N3—C13	1.481 (8)	N6—C26	1.466 (9)
C13—N1—C14	120.5 (6)	N3—C13—C12	111.6 (5)
C13—N1—C39	117.0 (6)	C1—C13—C12	101.3 (5)
C14—N1—C39	116.7 (6)	O1—C14—N1	124.2 (8)
C14—N2—C15	124.8 (7)	O1—C14—N2	121.0 (8)
C14—N2—C40	118.0 (6)	N1—C14—N2	114.8 (7)
C15—N2—C40	117.1 (7)	O2—C15—N2	120.6 (7)
C13—N3—C15	118.2 (6)	O2—C15—N3	123.2 (7)
C13—N3—C16	117.5 (5)	N2—C15—N3	116.1 (7)
C15—N3—C16	115.7 (6)	N3—C16—C17	116.7 (5)
C25—N4—C26	123.0 (6)	N6—C23—C22	115.6 (5)
C25—N4—C41	116.1 (6)	O4—C24—N5	119.8 (7)
C26—N4—C41	116.4 (6)	O4—C24—N6	122.9 (7)
C24—N5—C25	122.2 (6)	N5—C24—N6	117.4 (7)
C24—N5—C42	118.7 (7)	O3—C25—N4	122.9 (8)
C25—N5—C42	117.6 (6)	O3—C25—N5	120.5 (7)
C23—N6—C24	115.9 (6)	N4—C25—N5	116.7 (6)
C23—N6—C26	116.5 (5)	N4—C26—N6	108.2 (5)
C24—N6—C26	121.9 (6)	N4—C26—C27	110.0 (6)
N1—C13—N3	107.7 (5)	N4—C26—C38	112.3 (5)
N1—C13—C1	112.1 (6)	N6—C26—C27	111.8 (5)
N1—C13—C12	113.2 (5)	N6—C26—C38	113.6 (6)
N3—C13—C1	110.9 (5)		

Table 3. Hydrogen-bonding geometry (Å, °)

D—H...A	D—H	H...A	D...A	D—H...A
C5—H5...O3 <sup>i</sup>	0.95	2.37	3.288 (9)	163.1
C9—H9...O4 <sup>ii</sup>	0.95	2.44	3.265 (9)	145.1

Symmetry codes: (i)  $x - 1, y, z$ ; (ii)  $1 - x, -y, 2 - z$ .

Data collection: *MSCIAFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSCIAFC Diffractometer Control Software*. Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN LS*. Molecular graphics: *ORTEP* (Johnson, 1965) in *TEXSAN*. Software used to prepare material for publication: *TEXSAN FINISH*, *PLATON* (Spek, 1990).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: FG1188). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Packing of Four Independent Molecules: 1-Methyl-*N,N'*-bis(salicylidene)-2,4-phenylenediamine

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## Abstract

The four independent molecules in the asymmetric unit of the title compound, C<sub>21</sub>H<sub>18</sub>N<sub>2</sub>O<sub>2</sub>, share similar geometry with two coplanar rings and the third making an angle of 20–40° with the others. In the crystal, pairs of molecules are aligned parallel, apart from the diverging third rings.

## Comment

The title compound (I) was prepared for evaluation as a potential ligand, and the crystal structure of the uncomplexed molecule was determined for comparative purposes. The dimensions appear standard, though few closely similar structures have been reported (Allen *et al.*, 1991); the central toluene–nitrogen core was reported by Zhou, Chen & Shi (1984). The correspond-