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3,3″-Dimethyl-1,1′:4′,1″-terphenyl

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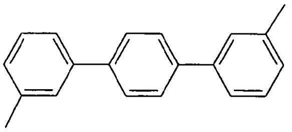
Abstract

The title compound, $C_{20}H_{18}$, is centrosymmetric and adopts a twisted conformation. This result contrasts related unsubstituted systems, where rotational disorder is inevitably found. The different behaviour is rationalized in terms of the absence of π–π interactions in the lattice of the title compound.

Comment

The title compound, (1), was isolated as a by-product from the addition of singlet oxygen to 1-methyl-3-[4-(3-methylphenyl)-1,3-cyclohexadienyl]benzene. The structure of (1) is molecular, with the closest non-hydrogen contact of 3.588 (3) Å occurring between C2′ and C6′^{i} [symmetry code: (i) $-\frac{1}{2}+x$, $-\frac{1}{2}-y$, $-\frac{1}{2}+z$]; there is no evidence of base stacking, *i.e.* π–π interactions. The molecule is centrosymmetric with significant twists between the constituent rings as seen in the dihedral angle between the inner and outer rings of 35.3 (1)°. Consequently, there is little opportunity for conjugation throughout the molecule and this is reflected in the C1—C1′ bond distance of 1.488 (2) Å. This separation compares well with that found in *p*-terphenyl of 1.496 (4) Å (Rietveld *et al.*, 1970). Other geometric parameters are as expected, with the aromatic C—C bonds lying in the range 1.377 (3) to 1.399 (2) Å, and C3′—C3″ is 1.504 (3) Å.



(1)

p-Polyphenyls have received considerable attention owing to their molecular flexibility. Whether a particular structure is planar or twisted depends on a balance between (i) intermolecular forces and (ii) intramolecular repulsions between the *ortho*-H atoms (Saitoh *et al.*, 1993; Baker *et al.*, 1993). Accordingly, while a planar structure is found for *p*-terphenyl and indeed higher homologues at room temperature, these structures undergo phase changes at reduced temperatures to give rise to twisted conformations. In *p*-terphenyl, the dihedral angle between the inner and outer rings at 200 K is 26.6° (Baudour *et al.*, 1986). The absence of extended base stacking in (1) precludes the sort of rotational disorder found in the unsubstituted analogues.

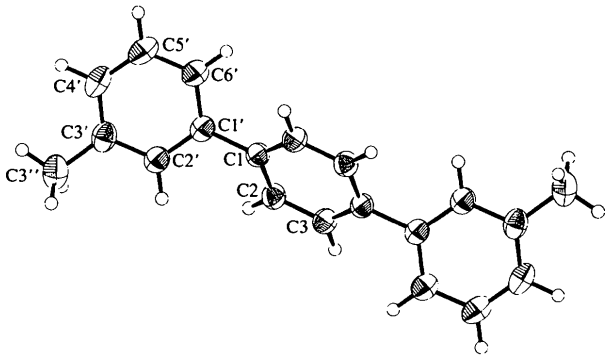


Fig. 1. The title structure is shown with 40% probability displacement ellipsoids (*ORTEP*II; Johnson, 1976).

Experimental

To a solution of 1-methyl-3-[4-(3-methylphenyl)-1,3-cyclohexadienyl]benzene in dichloromethane (100 ml) at 283 K was added rose bengal (5 mg). A gentle stream of oxygen was passed through the solution with concomitant irradiation from a 500 W tungsten halogen lamp at a distance of 10 cm from the reaction vessel. After 4 h, the mixture was concentrated and subjected to column chromatography. Elution with hexane afforded the title compound in 25% yield as a white solid; recrystallization from a chloroform solution of the compound gave colourless crystals with m.p. 412–413 K; literature m.p. 414 K (Kern *et al.*, 1959).

Crystal data

$C_{20}H_{18}$
$M_r = 258.36$
Monoclinic
$P2_1/n$
$a = 7.983\ (2)$ Å
$b = 10.908\ (1)$ Å
$c = 9.264\ (1)$ Å
$\beta = 114.51\ (1)^\circ$
$V = 734.0\ (2)$ Å^3
$Z = 2$
$D_x = 1.169$ Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 53.1\text{–}55.0^\circ$
$\mu = 0.493$ mm^{-1}
$T = 293$ K
Needle
0.44 × 0.11 × 0.11 mm
Colourless

Data collection

Rigaku AFC-6*R* diffractometer
$\omega\text{–}2\theta$ scans
Absorption correction: analytical (De Meulenaer & Tompa, 1965)
$T_{\min} = 0.871$, $T_{\max} = 0.957$
1679 measured reflections
1537 independent reflections

1142 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.049$
$\theta_{\max} = 75.0^\circ$
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 13$
$l = -11 \rightarrow 9$
3 standard reflections every 400 reflections intensity decay: −0.45%

Refinement

Refinement on F
$R = 0.053$
$wR = 0.068$
$S = 2.828$
1142 reflections
91 parameters
H atoms fixed in calculated positions, C—H = 0.97 Å

$w = 1/[\sigma^2(F) + (0.0125F)^2]$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.22$ e Å^{-3}
$\Delta\rho_{\min} = -0.17$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1992). Program(s) used to solve structure: *SIR*92 (Burla *et al.* 1989). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *TEXSAN*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: LN1034). Services for accessing these data are described at the back of the journal.

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2,2′-Bipyridine 1,1′-Dioxide

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Abstract

The title molecule, $C_{10}H_8N_2O_2$, lies on a crystallographic twofold axis. The dihedral angle between the planes of the pyridine groups is 67.5° and the non-bonded distance between the O atoms in the *N*-oxide groups is 3.046 (2) Å. Although the skew conformation of the skeleton is almost the same as that of the coordinated form, the dihedral angle is the largest among the corresponding values for the 2,2′-bipyridine 1,1′-dioxide complexes.

Comment

2,2′-Bipyridine 1,1′-dioxide (bpdo), (I), forms a skew seven-membered chelate ring with a metal ion. Recently, we reported that the tetraammine(bpdo)cobalt(III) complex was resolved into a pair of enantiomers which rapidly racemized in water (Kanno *et al.*, 1997). This result indicates that the skewed bpdo-chelate ring is chiral and changes its conformation easily in solution.