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3,3''-Dimethyl-1,1':4',1''-terphenyl

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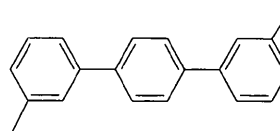
Abstract

The title compound, C₂₀H₁₈, 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.

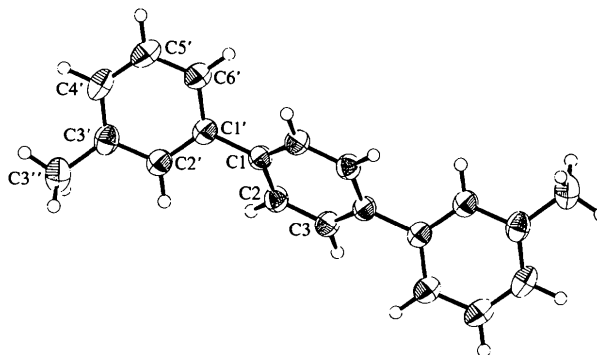


Fig. 1. The title structure is shown with 40% probability displacement ellipsoids (ORTEPII; Johnson, 1976).

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

$C_{20}H_{18}$	Cu $K\alpha$ radiation
$M_r = 258.36$	$\lambda = 1.5418 \text{ \AA}$
Monoclinic	Cell parameters from 25 reflections
$P2_1/n$	$\theta = 53.1\text{--}55.0^\circ$
$a = 7.983 (2) \text{ \AA}$	$\mu = 0.493 \text{ mm}^{-1}$
$b = 10.908 (1) \text{ \AA}$	$T = 293 \text{ K}$
$c = 9.264 (1) \text{ \AA}$	Needle
$\beta = 114.51 (1)^\circ$	$0.44 \times 0.11 \times 0.11 \text{ mm}$
$V = 734.0 (2) \text{ \AA}^3$	Colourless
$Z = 2$	
$D_x = 1.169 \text{ Mg m}^{-3}$	
D_m not measured	

Data collection

Rigaku AFC-6R diffractometer	1142 reflections with $I > 2\sigma(I)$
ω -2 θ scans	$R_{\text{int}} = 0.049$
Absorption correction: analytical (De Meulenaer & Tompa, 1965)	$\theta_{\text{max}} = 75.0^\circ$
$T_{\text{min}} = 0.871$, $T_{\text{max}} = 0.957$	$h = 0 \rightarrow 9$
1679 measured reflections	$k = 0 \rightarrow 13$
1537 independent reflections	$l = -11 \rightarrow 9$
	3 standard reflections every 400 reflections
	intensity decay: -0.45%

Refinement

Refinement on F	$w = 1/[\sigma^2(F) + (0.0125F)^2]$
$R = 0.053$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$wR = 0.068$	$\Delta\rho_{\text{max}} = 0.22 \text{ e \AA}^{-3}$
$S = 2.828$	$\Delta\rho_{\text{min}} = -0.17 \text{ e \AA}^{-3}$
1142 reflections	Extinction correction: none
91 parameters	Scattering factors from <i>International Tables for Crystallography</i> (Vol. C)
H atoms fixed in calculated positions, C—H = $0.97 \text{ \AA}$	

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: *SIR92* (Burla *et al.* 1989). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEPII* (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 $3.046 (2) \text{ \AA}$. 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.