

Anwar Usman,^a Ibrahim Abdul Razak,^a Hoong-Kun Fun,^{a*} Suchada Chantrapromma,^{a†} Min Zhang^b and Jian-Hua Xu^b^aX-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, and ^bDepartment of Chemistry, Nanjing University, Nanjing 210093, People's Republic of China

† Permanent address: Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand

Correspondence e-mail: hkfun@usm.my

Key indicators

Single-crystal X-ray study
T = 213 K
Mean $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$
R factor = 0.058
wR factor = 0.137
Data-to-parameter ratio = 13.4For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

5-(1,4-Dimethyl-4-phenyl-1,2,3,4-tetrahydro-1-naphthyl)-1,2,4-benzenetricarbonitrile

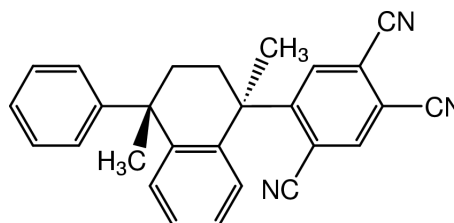
In the title compound, $\text{C}_{27}\text{H}_{21}\text{N}_3$, the tetrahydrobenzene ring adopts a half-chair conformation. The orientations of the attached phenyl and tricyanophenyl substituents are determined by the sp^3 state of the tetrahydrobenzene atoms.

Received 11 March 2002

Accepted 21 March 2002

Online 28 March 2002

Comment

1,2,4,5-Tetracyanobenzene (TCB) is one of the compounds of interest in photo-induced electron transfer (PET) reactions, owing to its extremely strong electron-withdrawing ability. In our ongoing studies on PET reactions, we have found that PET reaction between TCB and an excess amount of α -methylstyrene in a polar solvent (acetonitrile) afforded the title compound, (I), as one of the stereoisomeric products. As a part of these studies, we have undertaken the X-ray crystallographic analysis of the title compound in order to elucidate its conformation and configuration.

(I)

The bond lengths and angles in the title compound are within normal ranges (Allen *et al.*, 1987), and are in good agreement with the related compound 4-phenyl-1-(3,4,6-tricyanophenyl)-1,2,3,4-tetrahydronaphthalene [(II); Zhang *et al.*, 2002]. The $\text{C}_{\text{ar}}-\text{C}$ distances of the carbonitriles in the title compound are slightly longer [1.440 (4)–1.446 (4) Å] than the typical $\text{C}_{\text{sp}}-\text{C}_{\text{sp}^2}$ bond distance (Allen *et al.*, 1987); they are comparable with those observed in the related compounds (II) [1.442 (4)–1.446 (4) Å; Zhang *et al.*, 2002] and 1,2,4,5-benzenetetracarboxitrile–acridine (1/1) [1.444 (7) Å; Toupet *et al.*, 1989].

In the title structure (Fig. 1), the tetrahydrobenzene ring adopts a half-chair conformation, with puckering parameters (Cremer & Pople, 1975) $Q = 0.487$ (3) Å, $\theta = 48.7$ (4)° and $\varphi = 78.8$ (4)°, and atoms C15 and C16 deviate from the C7/C8/C13/C14 plane by -0.243 (3) and 0.487 (3) Å, respectively. In the tetrahydronaphthalene, the mean plane through the tetrahydrobenzene ring makes a dihedral angle of 5.5 (1)° with the benzene ring. The methyl groups attached to the tetrahydronaphthalene unit have different torsion angles [C13–C8–C7–C27 -98.5 (3)° and C8–C13–C14–C26 108.2 (3)°], as a

result of the different substituents at C7 and C14. The orientation of the phenyl and tricyanophenyl substituents are determined by the sp^3 state of the tetrahydrobenzene atoms. The tricyanophenyl moiety attached at C7 and the phenyl ring attached at C14 make dihedral angles of 70.1 (1) and 75.1 (1) $^\circ$, respectively, with the mean plane through the tetrahydrobenzene ring.

Experimental

The title compound was the product of a photo-induced reaction between 1,2,4,5-benzenetetracarbonitrile and an excess of α -methylstyrene in an acetonitrile solution; it was isolated by column chromatography on silica gel. Single crystals suitable for X-ray diffraction analysis were obtained by slow evaporation of a chloroform–ethyl acetate (4:1) solution.

Crystal data

$C_{27}H_{21}N_3$	$D_x = 1.223 \text{ Mg m}^{-3}$
$M_r = 387.47$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 3599 reflections
$a = 9.0481 (4) \text{ \AA}$	$\theta = 2.5\text{--}28.4^\circ$
$b = 8.2368 (4) \text{ \AA}$	$\mu = 0.07 \text{ mm}^{-1}$
$c = 28.4787 (13) \text{ \AA}$	$T = 213 (2) \text{ K}$
$\beta = 97.384 (1)^\circ$	Slab, colorless
$V = 2104.84 (17) \text{ \AA}^3$	$0.18 \times 0.18 \times 0.14 \text{ mm}$
$Z = 4$	

Data collection

Siemens SMART CCD area-detector diffractometer	1901 reflections with $I > 2\sigma(I)$
ω scans	$R_{\text{int}} = 0.098$
Absorption correction: none	$\theta_{\text{max}} = 25.0^\circ$
9600 measured reflections	$h = -10 \rightarrow 10$
3659 independent reflections	$k = -9 \rightarrow 9$
	$l = -33 \rightarrow 20$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0361P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.058$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.137$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 0.85$	$\Delta\rho_{\text{max}} = 0.25 \text{ e \AA}^{-3}$
3659 reflections	$\Delta\rho_{\text{min}} = -0.21 \text{ e \AA}^{-3}$
274 parameters	Extinction correction: <i>SHELXTL</i>
H-atom parameters constrained	Extinction coefficient: 0.0150 (16)

Table 1

Selected interatomic distances (\AA).

C2—C23	1.442 (4)	C7—C16	1.547 (3)
C3—C24	1.440 (4)	C14—C26	1.541 (4)
C5—C25	1.446 (4)	C15—C16	1.514 (3)
C7—C27	1.545 (4)		

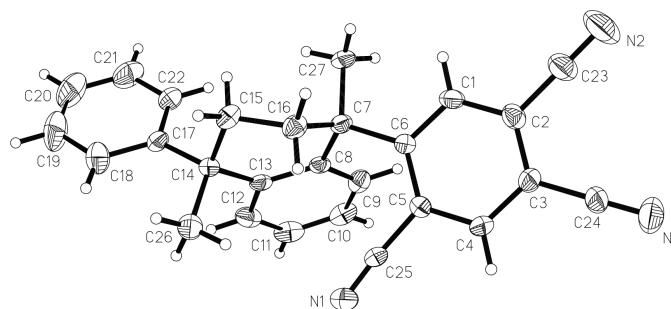


Figure 1

The structure of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme.

The H atoms were fixed geometrically and were treated as riding on the parent C atoms, with C—H distances in the range 0.93–0.97 \AA .

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT* and *SADABS* (Sheldrick, 1996); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 1990).

The authors thank the Malaysian Government and Universiti Sains Malaysia for research grant R&D No. 305/PFIZIK/610961. AU thanks Universiti Sains Malaysia for a Visiting Postdoctoral Fellowship.

References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
- Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
- Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
- Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
- Sheldrick, G. M. (1997). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
- Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Spek, A. L. (1990). *Acta Cryst.* **A46**, C-34.
- Toupet, L., Minewicz, A. & Ecolivet, C. (1989). *Acta Cryst.* **C45**, 1044–1047.
- Zhang, M., Usman, A., Razak, I. A., Fun, H.-K., Chantrapromma, S. & Xu, J.-H. (2002). *Acta Cryst.* **E58**, o132–o133.