

p*-Diisocyanotetramethylbenzene*Matthias Zeller, Allen D. Hunter* and Cynthia L. Perrine**

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Key indicators

Single-crystal X-ray study

 $T = 100\text{ K}$ Mean $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$

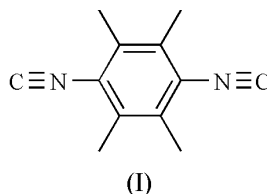
Disorder in main residue

 R factor = 0.046 wR factor = 0.131

Data-to-parameter ratio = 18.2

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.*p*-Diisocyanotetramethylbenzene, $p\text{-C}_6(\text{CH}_3)_4(\text{NC})_2$, crystallizes in the monoclinic space group $C2/c$. One-half of the molecule is crystallographically independent, with the other half being generated by an inversion centre located at the centre of the molecule.**Comment**

In the course of our work on isonitrile-bridged and capped molybdenum complexes, we isolated and structurally characterized *p*-diisocyanotetramethylbenzene, (I). The bisisonitrile is located on an crystallographic inversion centre, with only one-half of the molecule crystallographically independent. Both crystallographically independent methyl groups show disorder of the H atoms; these have been refined as idealized disordered methyl groups with the two positions rotated from each other by 60° and the site distributions have been refined to be approximately 0.90:0.10 (2) and 0.72:0.28 (2). Bond lengths and angles are in the expected ranges for aromatic isonitriles.



The compound was found to be isostructural with its cyanoisomer, dicyanodurene, $p\text{-C}_6(\text{CH}_3)_4(\text{NC})_2$ (Britton & Van Rij, 1991). Cell parameters and the orientation of the molecules are essentially identical.

Experimental

1,4-Diisocyano-2,3,5,6-tetramethylbenzene was synthesized from the corresponding formamide by reacting tetramethyl-*p*-phenylenediamine with 75% formic acid under reflux for two hours. Addition of water precipitated the formamide, which was washed with water until neutral and dried *in vacuo*. The isocyanide was synthesized as described by Efraty *et al.* (1980) for 1,4-diisocyanobenzene. The isocyanide was purified by sublimation *in vacuo*. Single crystals were grown by sublimation *in vacuo*.

Crystal data $\text{C}_{12}\text{H}_{12}\text{N}_2$ $M_r = 184.24$ Monoclinic, $C2/c$ $a = 17.1518(17)\text{ \AA}$ $b = 5.1324(5)\text{ \AA}$ $c = 12.3670(12)\text{ \AA}$ $\beta = 112.010(2)^\circ$ $V = 1009.32(17)\text{ \AA}^3$ $Z = 4$ $D_x = 1.221\text{ Mg m}^{-3}$ Mo $K\alpha$ radiation

Cell parameters from 4202

reflections

 $\theta = 2.6\text{--}28.3^\circ$ $\mu = 0.07\text{ mm}^{-1}$ $T = 100(2)\text{ K}$

Block, colourless

 $0.50 \times 0.35 \times 0.35\text{ mm}$

Data collection

Bruker AXS SMART APEX CCD diffractometer
 ω scans
 Absorption correction: none
 4935 measured reflections
 1257 independent reflections

1181 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.080$
 $\theta_{\text{max}} = 28.3^\circ$
 $h = -22 \rightarrow 21$
 $k = -6 \rightarrow 6$
 $l = -16 \rightarrow 16$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.046$
 $wR(F^2) = 0.131$
 $S = 1.05$
 1257 reflections
 69 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0698P)^2 + 0.6662P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.016$
 $\Delta\rho_{\text{max}} = 0.32 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.27 \text{ e } \text{\AA}^{-3}$
 Extinction correction: *SHELXL*
 Extinction coefficient: 0.011 (4)

Table 1

Selected geometric parameters (\AA , $^\circ$).

N1—C1	1.1628 (14)	C3—C5	1.5106 (13)
N1—C2	1.4045 (12)	C4—C6	1.5105 (13)
C3—C4	1.4008 (13)	C2—C4 ⁱ	1.4065 (13)
C3—C2	1.4070 (13)		
C1—N1—C2	178.30 (10)	C3—C4—C6	121.39 (8)
C4—C3—C2	117.65 (9)	C2 ⁱ —C4—C6	120.69 (9)
C4—C3—C5	121.14 (8)	N1—C2—C4 ⁱ	117.59 (8)
C2—C3—C5	121.20 (9)	N1—C2—C3	117.99 (8)
C3—C4—C2 ⁱ	117.92 (9)	C4 ⁱ —C2—C3	124.42 (9)

Symmetry code: (i) $\frac{1}{2} - x, \frac{3}{2} - y, 1 - z$.

Both crystallographically independent methyl groups show rotational disorder. The H atoms were refined as idealized disordered methyl groups, with the two positions rotated from each other by 60° . They were geometrically positioned with a fixed C—H distance of 0.98 \AA . Isotropic displacement parameters are 1.5 times U_{eq} of the parent atom. The major site occupancies were refined to be approximately 0.90 (2) and 0.72 (2).

The s.u. values of the cell parameters are derived from the software, and are unreasonably small (Herbstein, 2000).

Data collection: *SMART* (Bruker 1997–2000); cell refinement: *SAINT-Plus* (Bruker 1997–1999); data reduction: *SAINT-Plus*;

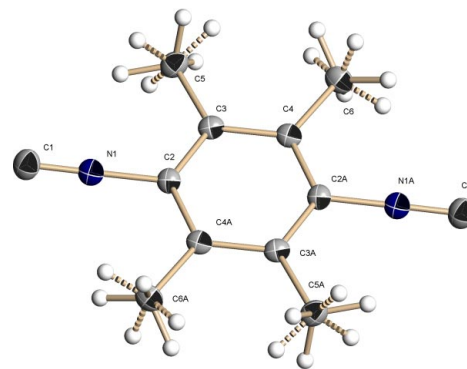


Figure 1

Molecular structure of (I), showing 30% probability displacement ellipsoids.

program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXL97*; software used to prepare material for publication: *SHELXL97*.

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