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N,*N*′,*N*″,*N*‴-Tetrakis(2,6-dimethylphenyl)hexane-2,3,4,5-tetraimine

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The oligomerization of 2,6-dimethylphenyl isocyanide by successive insertion of 2,6-dimethylphenyl isocyanide with MeLi reagent gave the title compound, $C_{38}H_{42}N_4$, which crystallizes in a centrosymmetric structure and possesses normal geometric parameters.

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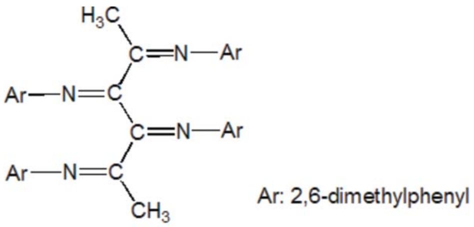
Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.060
wR factor = 0.135
Data-to-parameter ratio = 16.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

The addition of organolithium compounds to isonitriles is subject to various side reactions, notably α-deprotonation (Walborsky *et al.*, 1970, 1971; Niznik *et al.*, 1974; Hirowatari & Walborsky, 1974; Niznik & Walborsky, 1974; Periasamy & Walborsky, 1974). In the absence of α-H atoms, however, isonitriles show carbene-like reactivity, giving lithioimines; subsequent reaction with electrophiles gives imine derivatives, which may be desired in their own right (Walborsky & Roman, 1978; Barluenga *et al.*, 1988; Ito *et al.*, 1984). In an attempted reaction of the corresponding lithium aldimine (obtained from the α-addition of methyllithium to 2,6-dimethylphenyl isocyanide), a diethyl ether solution of this compound was treated at 195 K with a hexane solution of diphenylboron chloride (Ph_2BCl), resulting in the formation of the title compound, (I), as a side product.



(I)

The title compound has half of the molecule in the asymmetric unit; the molecule is located on an inversion centre (Fig. 1). Notably, C—H···N hydrogen bonds are absent from the structure of (I). Its X-ray crystal structure reveals a dimeric compound where α-C atoms C2 and C2^{i} may represent the core of the molecule. Significant bond lengths, angles and torsion angles are shown in Table 1. Similar phenyl groups are situated in alternate positions in order to prevent high repulsion between them. The C4—N1 and C1—C2, C12—N2 and C2—C2^{i} bonds are antiperiplanar, while the C3—C1 and C2—N2 bonds are synclinal. All the geometric parameters for (I) lie within their expected ranges (Allen *et al.*, 1987). The crystal structure consists of molecular stacks formed by unit-cell translation along the a axis (Fig. 1).

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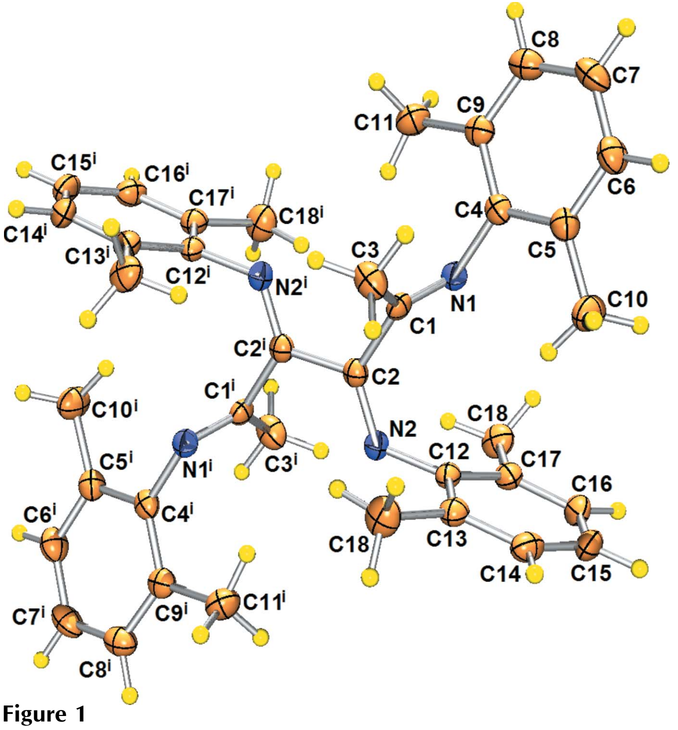

Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids [symmetry code: (i) $1 + x, 1 - y, 1 + z$]. Only one set of the disordered H atoms is shown.

Experimental

For the preparation of compound (I), a solution of 2,6-dimethylphenyl isonitrile (1.000 g, 7.62 mmol) in diethyl ether (10 ml) was treated with methyllithium (4.8 ml, 1.6 *M* in hexanes, 7.64 mmol) at 195 K. After 10 min, a solution of diphenylboron chloride (52.49 mg, 3.81 mmol) in hexane (10 ml) was added dropwise over a period of 30 min and the solution was allowed to warm to room temperature overnight. The solution was filtered, and the solvent and volatile components were removed under vacuum. The main product of (I) was extracted with two portions (20 ml) of diethyl ether, and crystallized from dimethylformamide by slow evaporation to give light-yellow block-shaped crystals of (I). A suitable crystal was selected from the resulting batch. CHN analysis of a handpicked sample of (I) found: C 81.07, H 7.14, N 9.97%; calculated: C 82.27, H 7.63, N 10.10%.

Crystal data

$C_{38}H_{42}N_4$
$M_r = 554.76$
Monoclinic, $P2_1/c$
$a = 8.5355\ (12)$ Å
$b = 24.172\ (3)$ Å
$c = 7.6566\ (11)$ Å
$\beta = 93.696\ (12)^\circ$
$V = 1576.4\ (4)$ Å^3
$Z = 2$
$D_x = 1.169$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.07$ mm^{-1}
$T = 150\ (2)$ K
Block, light yellow
0.18 × 0.14 × 0.10 mm

Data collection

Bruker SMART APEX CCD diffractometer with an Oxford Cryosystems low-temperature device
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.984$, $T_{\max} = 1.000$
8901 measured reflections
3228 independent reflections
2996 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.024$
$\theta_{\max} = 26.4^\circ$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.061$
$wR(F^2) = 0.135$
$S = 1.14$
3228 reflections
191 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0437P)^2 + 0.9545P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.26$ e Å^{-3}
$\Delta\rho_{\min} = -0.17$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C1	1.265 (2)	C1—C3	1.503 (2)
N1—C4	1.427 (2)	C1—C2	1.509 (2)
N2—C2	1.271 (2)	C2—C2^{ii}	1.497 (3)
N2—C12	1.430 (2)		
C1—N1—C4	119.58 (15)	C3—C1—C2	117.35 (15)
C2—N2—C12	121.34 (15)	N2—C2—C2^{ii}	117.52 (19)
N1—C1—C3	126.83 (16)	N2—C2—C1	126.65 (16)
N1—C1—C2	115.81 (15)	C2^{ii}—C2—C1	115.83 (18)
N1—C1—C2—N2	76.5 (2)	N1—C1—C2—C2^{ii}	−103.2 (2)
C3—C1—C2—N2	−104.5 (2)	C3—C1—C2—C2^{ii}	75.8 (2)

Symmetry code: (ii) $-x + 1, -y + 1, -z + 1$.

H atoms were included in the refinement at calculated positions, in the riding-model approximation, with C—H distances between 0.95 and 0.98 Å. The $U_{\rm iso}$(H) values were set equal to 1.5 (idealized disordered methyl H atoms) or 1.2 (other H atoms) times $U_{\rm eq}$(C).

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1994); software used to prepare material for publication: *SHELXL97*.

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