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2,4,6-Trimethylphenyl isocyanide

Manuel A. Fernandes, Marcus Layh* and Bernard Omondi

Molecular Sciences Institute, School of Chemistry, University of the Witwatersrand, PO Wits 2050, South Africa
Correspondence e-mail: marcus@aurum.chem.wits.ac.za

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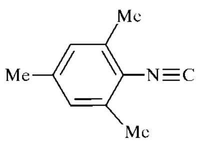
The title compound, $C_{10}H_{11}N$, displays a crystallographic mirror plane that incorporates all the non-H atoms, as well as the H atoms attached to the aromatic ring. The isocyano group is almost linear and shows an N≡C bond distance of 1.158 (3) Å.

Comment

We are currently interested in the reactions of α-H-free isocyanides with trimethylsilyl-substituted lithium amides, alkyls and silyls that have previously led to a wide variety of products. A neutral isocyanide adduct (Caro *et al.*, 1998), a lithium-1-azabuta-1,3-dienylamide (Hitchcock *et al.*, 2001), a silacyclobutene derivative (Hitchcock *et al.*, 1999), and a series of lithium-1-azaallyls (Hitchcock *et al.*, 2001) and lithium-β-diketiminates (Hitchcock *et al.*, 2001) have so far been isolated and crystallographically characterized. The isolated products are believed to depend on, amongst other factors, the lithium starting material, the solvent, and the steric and electronic properties of the isocyanide. As a recent search of the Cambridge Structural Database (CSD, Version 5.22; Allen & Kennard, 1993) has revealed that only a comparatively small number of simple aryl isocyanides have so far been structurally characterized, the majority of which carry electron-withdrawing substituents, we decided to determine the solid-state structure of 2,4,6-trimethylphenyl isocyanide, (I), by X-ray diffraction.



(I)

Compound (I) crystallizes in the orthorhombic space group *Pnma*, with all non-H atoms lying in a mirror plane. Its structure is isomorphous with that of the previously described 2,4,6-trimethylphenyl nitrile (Britton, 1979; CSD refcode MESITN; space group *Pnma*, cell parameters 15.637, 6.998 and 8.256 Å). The molecule of (I) (Fig. 1) is located on a crystallographic mirror plane that includes all the non-H atoms and the H atoms attached to the aromatic ring (atoms H3 and H5). The H atoms of the three methyl groups are staggered relative to the phenyl group, resulting in them being disordered over two symmetry-related positions, above and below the mirror plane.

The N≡C bond distance of 1.158 (3) Å (Table 1) lies well within the range found in aromatic isocyanides (1.153–1.163 Å), but is slightly longer than those found in aliphatic isocyanides (*e.g.* Lane *et al.*, 1994). This has been attributed to the possibility of delocalization of π-electron density from the isocyanide moiety into the aromatic ring, which partially reduces the N≡C bond order in aromatic isocyanides (Colapietro *et al.*, 1984). This notion is supported by a comparatively short N1≡C1 distance of 1.407 (3) Å.

The endocyclic bond angles within the aromatic ring deviate periodically by up to 3° (Table 1) from the expected value of 120° for an ideal hexagon. This has been attributed (Domenicano & Murray-Rust, 1979) to the electronic properties of the methyl (σ-donating) and isocyano substituents (σ-withdrawing and π-donating).

The supramolecular structure of (I) shows parallel layers of isocyanide molecules, which are stacked in an *ABA* pattern along the *b* axis (Fig. 2). Molecules in adjacent layers are arranged in such a fashion that the isocyano groups of two closest molecules point in opposite directions and one of the *o*-methyl groups (C8) is located above or below the aromatic ring of the adjacent isocyanide, with non-bonding intermolecular distances ranging from 3.583 (1) (C8···C1) to 3.843 (1) Å (C8···C4). This arrangement differs from that found in halogenated aryl isocyanides. In 2,4,6-trichlorophenyl isocyanide (Pink *et al.*, 2000) and pentafluorophenyl isocyanide (Lentz & Preugschat, 1993), the isocyano groups of neighbouring molecules point in the same direction and the isocyano and 4-halogeno groups are located in the centre of the aromatic ring of adjacent molecules. A similar arrangement is found for 1,4-diisocyanobenzene (Colapietro *et al.*, 1984). In 4-bromophenyl isocyanide (Britton *et al.*, 1978), 4-iodophenyl isocyanide (Britton *et al.*, 1978) and 2,4,6-tribromophenyl isocyanide (Carter *et al.*, 1977), the isocyano

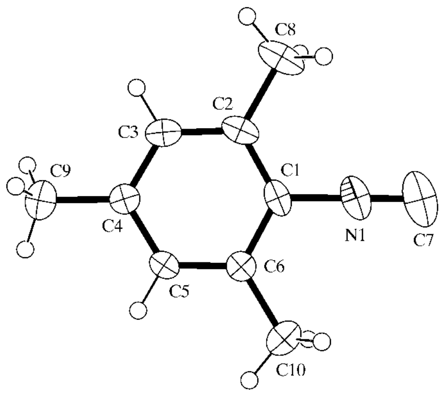


Figure 1
A view of the molecule of (I) showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

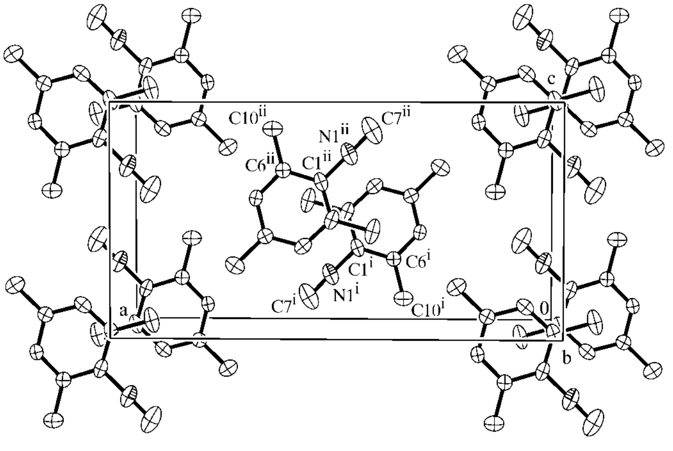


Figure 2
The packing of (I) viewed along the *b* axis. H atoms have been omitted for clarity [symmetry codes: (i) $\frac{1}{2} - x, -y, z + \frac{1}{2}$; (ii) $x + \frac{1}{2}, \frac{1}{2} - y, \frac{1}{2} - z$].

groups of molecules in different layers point in opposite directions, and there are close contacts between the isocyano group of one molecule and the halogen substituent in the 4-position of the adjacent molecule.

Experimental

Compound (I) was prepared from 2,4,6-trimethylaniline and formic acid, followed by treatment of the product with phosgene, as described by Ugi *et al.* (1965). Crystals of (I) suitable for an X-ray diffraction study were obtained from a solution of the isocyanide in hexane at 213 K.

Crystal data

$C_{10}H_{11}N$
$M_r = 145.20$
Orthorhombic, *Pnma*
$a = 15.7210$ (19) Å
$b = 6.8582$ (8) Å
$c = 8.2338$ (10) Å
$V = 887.75$ (18) Å^3
$Z = 4$
$D_x = 1.086$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 932 reflections
$\theta = 2.6$–24.5°
$\mu = 0.06$ mm^{-1}
$T = 173$ (2) K
Plate, colourless
0.36 × 0.22 × 0.10 mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.977$, $T_{\max} = 0.994$
5953 measured reflections

1189 independent reflections
590 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.087$
$\theta_{\max} = 28.3°$
$h = -20 \rightarrow 20$
$k = -9 \rightarrow 4$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.115$
$S = 0.91$
1189 reflections
70 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0547P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.15$ e Å^{-3}
$\Delta\rho_{\min} = -0.26$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C7	1.158 (3)	C2—C8	1.502 (3)
N1—C1	1.407 (3)	C3—C4	1.386 (3)
C1—C2	1.394 (3)	C4—C5	1.390 (3)
C1—C6	1.402 (3)	C4—C9	1.509 (3)
C2—C3	1.386 (3)	C5—C6	1.381 (3)
C7—N1—C1	179.0 (3)	C3—C4—C5	118.57 (19)
C2—C1—C6	123.10 (18)	C6—C5—C4	122.10 (19)
C3—C2—C1	116.92 (19)	C5—C6—C1	117.04 (18)
C2—C3—C4	122.26 (19)		

H atoms were treated as riding atoms with C—H distances in the range 0.95–0.98 Å.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT-Plus* (Bruker, 1999); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Bruker, 1999); program(s) used to refine structure: *SHELXTL*; molecular graphics: *PLATON* (Spek, 1990) and *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXTL*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: GD1206). Services for accessing these data are described at the back of the journal.

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