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(Z)-N-(2-Iodophenyl)-4-nitrobenzimidoyl cyanide

Rodolfo Moreno-Fuquen,* Andres C. Garcia, Rodrigo Abonia, Luz M. Jaramillo-Gómez and Alan R. Kennedy

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Keywords: crystal structure; α-iminonitrile; C—H···N interactions.

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Structural data: full structural data are available from iucrdata.iucr.org

In the title molecule, C14H8IN3O2, the cyanide group is anti to the iodide substituent of the adjacent benzene ring. The central segment is essentially planar (r.m.s deviation = 0.034 Å) and it is twisted away from the iodide- and nitro-substituted benzene rings by 69.02 (9) and 15.83 (16)°, respectively. In the crystal, molecules are linked by weak C—H···N interactions, leading to C(8) chains along [010].

Structure description

The α-iminonitriles are an important class of synthetic products with interesting biological activities (Jursic et al., 2002). These compounds are useful precursors for the synthesis of analogues of naturally occurring iminosugars (Ayers & Fleet, 2014), amide-functionality formation (Gualtierotti et al., 2012), and are frequently found in different natural compounds, pharmaceuticals and polymers. Several methodologies have been developed for the synthesis of α-iminonitriles (Fontaine et al., 2008; Gualtierotti et al., 2012; Jursic et al., 2002). In our approach, the (Z)-N-(2-iodophenyl)-4-nitrobenzimidoyl cyanide, (I), was obtained through an oxidative Strecker-type reaction from the imine, previously formed by a condensation reaction of 2-iodoaniline with 4-nitrobenzaldehyde.

A perspective view of the molecule of the title compound, showing the atomic numbering scheme, is given in Fig. 1. The structural parameters of a related ligand, i.e. containing (I) in its backbone, has been reported in an organoruthenium compound (II) (Xiang et al., 2010), and can serve as a comparison with (I). A comparison of the bond lengths in the central segment C1/N1/C8/C7/N2/C9 of (I) and (II), shows an elongation in the C1—N1 [1.452 (3) Å] and a shortening in C8—C9 [1.460 (3) Å] in (II). These differences in bond lengths may be due to the formation of bonds with the ruthenium atom via O atoms appended to the backbone. The cyanide group is anti to the α-iodide.
 substituent in the adjacent benzene ring. The central segment C1/N1/C8/C7/N2/C9 is essentially planar, with an r.m.s deviation of 0.0341 Å and it is twisted away from the iodide- and nitro-substituted benzene rings by 69.02 (9) and 15.83 (16)°, respectively.

In the crystal, molecules of (I) are linked by weak inter- molecular C—H—N interactions, Table 1. These interactions generate C(8) chains of molecules along [010], see Fig. 2.

Synthesis and crystallization
A mixture of 2-iodoaniline (100 mg, 0.46 mmol) and 4-nitrobenzaldehyde (69 mg, 0.46 mmol) was heated at 373 K for 1 h in solvent-free conditions until the starting materials were no longer detected by TLC, to afford a yellow solid (in quantitative yield) corresponding to the imine. Then a mixture of imine (100 mg, 0.28 mmol), potassium cyanide (37 mg, 0.57 mmol), silica gel (50 mg) and acetonitrile (5 mL) was stirred at room temperature for 20 h. After the imine was consumed (monitored by TLC), the solvent was evaporated under reduced pressure and the crude product was purified by column chromatography on silica gel using a hexane–dichloromethane mixture (4:1, v/v) as eluent to afford compound (I) [57% yield, orange solid, m.p. 433 (1) K].

Refinement
Crystal data, data collection and structure refinement details are summarized in Table 2. The maximum and minimum residual electron density peaks of 1.42 and 0.84 eÅ⁻³, respectively, were located 0.86 and 0.79 Å from the I1 atom.

Acknowledgements
RMF, RA, ACG and LMJ are grateful to Colciencias and the Universidad del Valle, Colombia, for partial financial support.

References

Table 1
Hydrogen-bond geometry (Å, °).

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Symmetry code: (i) −x, y + ½, −z + ½

Table 2
Experimental details.

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<td>Tmin, Tmax</td>
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<tr>
<td>No. of measured, independent and observed</td>
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<td>Rm</td>
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<td>(sin θ/λ)max (Å⁻¹)</td>
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| Refinement                 |                             |
| R(F² > 2σ(F²))             | 0.032, 0.087, 1.07          |
| No. of reflections         | 3014                       |
| No. of parameters          | 181                        |
| H-atom treatment          | H-atom parameters constrained |
| Δρmax, Δρmin (e Å⁻³)       | 1.42, −0.84                |

Computer programs: CrysAlis PRO (Oxford Diffraction, 2010), SIR92 (Altomare et al., 1994), SHELXL2014 (Sheldrick, 2015), ORTEP-3 for Windows (Farrugia, 2012), Mercury (Macrae et al., 2006).

full crystallographic data

IUCrData (2016). 1, x160315  [doi:10.1107/S2414314616003151]

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(Z)-N-(2-Iodophenyl)-4-nitrobenzimidoyl cyanide

Crystal data
C_{14}H_{8}IN_{3}O_{2}

$D_{x} = 1.872$ Mg m$^{-3}$

Melting point: $433(1)$ K

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 3743 reflections

$\theta = 4.8$–$28.3^\circ$

$\mu = 2.40$ mm$^{-1}$

$T = 123$ K

Fragment from a large block, orange

$0.25 \times 0.25 \times 0.18$ mm

Data collection

Oxford Diffraction Xcalibur E

diffractometer

6073 measured reflections

Radiation source: fine-focus sealed tube

3014 independent reflections

Graphite monochromator

2602 reflections with $I > 2\sigma(I)$

$\omega$ scans

$R_{int} = 0.049$

Absorption correction: multi-scan

$\theta_{max} = 27.5^\circ$, $\theta_{min} = 4.8^\circ$

(CrysAlis PRO; Oxford Diffraction, 2010)

$h = -14 \rightarrow 15$

$T_{min} = 0.850$, $T_{max} = 1.000$

$k = -11 \rightarrow 8$

$l = -17 \rightarrow 16$

Refinement

Refinement on $F^2$

Secondary atom site location: difference Fourier map

Least-squares matrix: full

Hydrogen site location: inferred from neighbouring sites

$R[F^2 > 2\sigma(F^2)] = 0.032$

H-atom parameters constrained

$wR(F^2) = 0.087$

$w = 1/[\sigma^2(F^2) + (0.0437P)^2]$

where $P = (F^2 + 2F_c^2)/3$

$(\Delta\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 1.42$ e Å$^{-3}$

$\Delta\rho_{min} = -0.84$ e Å$^{-3}$

$3014$ reflections

$181$ parameters
**Special details**

**Experimental.** IR (FT–IR SHIMADZU IR-Affinity-1 spectrophotometer; KBr): cm\(^{-1}\), 3072, 2954, 2225 (CN), 1593, 1510 (NO\(_2\)), 1340 (NO\(_2\)), 1201, 1002. \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\): 8.04 (dd, \(J = 7.9, 1.3\) Hz, 2H), 7.54 (btd, \(J = 7.7, 1.3\) Hz, 2H), 7.23 (dd, \(J = 7.9, 1.4\) Hz, 2H), 7.13 (btd, \(J = 7.7, 1.5\) Hz, 2H) ppm. \(^13\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\): 150.4, 149.3, 139.7, 138.2, 138.0, 129.6, 129.5, 129.4, 124.3, 118.5, 110.0, 93.00 ppm. MS (70 eV) m/z (%): 379, 378, 377 (2.4, 18, 100) \([\text{M}^+]\), 250 (17), 203 (47), 204 (68). Crystals of (I) suitable for single-crystal X-ray diffraction were grown by slow evaporation, at ambient temperature and in air, from a solution in chloroform.

**Geometry.** All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

**Refinement.** Refinement of \(F^2\) against ALL reflections. The weighted \(R\)-factor \(wR\) and goodness of fit \(S\) are based on \(F^2\), conventional \(R\)-factors \(R\) are based on \(F\), with \(F\) set to zero for negative \(F^2\). The threshold expression of \(F^2 > \sigma(F^2)\) is used only for calculating \(R\)-factors (gt) etc. and is not relevant to the choice of reflections for refinement. \(R\)-factors based on \(F^2\) are statistically about twice as large as those based on \(F\), and \(R\)-factors based on ALL data will be even larger.

**Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å\(^2\))**

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**Atomic displacement parameters (Å\(^2\))**

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**Geometric parameters (Å, °)**

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C3—C2—I1   119.3 (2)  C11—C10—H10  120.0
C1—C2—I1   120.1 (2)  C9—C10—H10  120.0
C2—C3—C4   119.5 (3)  C12—C11—C10  118.2 (3)
C2—C3—H3   120.2     C12—C11—H11  120.9
C4—C3—H3   120.2     C10—C11—H11  120.9
C5—C4—C3   119.9 (3)  C11—C12—C13  123.1 (3)
C5—C4—H4   120.1     C11—C12—N3   119.2 (3)
C3—C4—H4   120.1     C13—C12—N3   117.6 (3)
C4—C5—C6   121.2 (3)  C12—C13—C14  118.5 (3)
C4—C5—H5   119.4     C12—C13—H13  120.7
C6—C5—H5   119.4     C14—C13—H13  120.7
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C5—C6—H6   120.4     C13—C14—H14  120.2
C1—C6—H6   120.4     C9—C14—H14  120.2

C8—N1—C1—C2  −120.1 (3)  N1—C8—C9—C10  −164.4 (3)
C8—N1—C1—C6  66.2 (4)  C7—C8—C9—C10  15.2 (4)
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N1—C1—C2—C3  −174.2 (3)  C8—C9—C10—C11  −177.1 (3)
C6—C1—C2—I1  −177.9 (2)  C9—C10—C11—C12  1.3 (5)
N1—C1—C2—I1  8.4 (4)  C10—C11—C12—C13  −3.1 (5)
C1—C2—C3—C4  1.3 (5)  C10—C11—C12—N3  176.3 (3)
I1—C2—C3—C4  178.7 (2)  O2—N3—C12—C11  −174.6 (3)
C2—C3—C4—C5  −1.0 (5)  O1—N3—C12—C11  4.5 (4)
C3—C4—C5—C6  0.0 (5)  O2—N3—C12—C13  4.8 (4)
C4—C5—C6—C1  0.7 (5)  O1—N3—C12—C13  −176.1 (3)
C2—C1—C6—C5  −0.5 (5)  C11—C12—C13—C14  2.4 (5)
N1—C1—C6—C5  173.2 (3)  N3—C12—C13—C14  −176.9 (3)
C1—N1—C8—C7  7.3 (4)  C12—C13—C14—C9  0.0 (5)
C1—N1—C8—C9  −173.1 (3)  C10—C9—C14—C13  −1.7 (5)
N1—C8—C9—C14  17.5 (4)  C8—C9—C14—C13  176.5 (3)
C7—C8—C9—C14  −162.9 (3)

Hydrogen-bond geometry (Å, °)

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Symmetry code: (i) −x, y+1/2, −z+1/2.