IUCrData

ISSN 2414-3146

Received 5 February 2016
Accepted 24 February 2016

Edited by E. R. T. Tiekink, Sunway University, Malaysia

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

CCDC reference: 1455354

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

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

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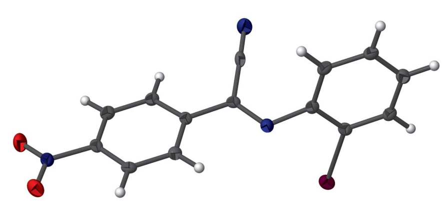
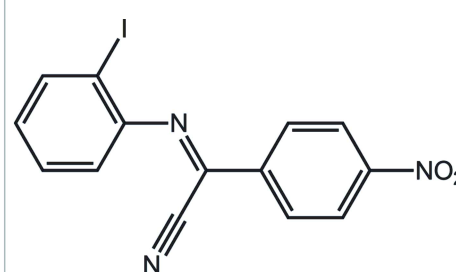
In the title molecule, $C_{14}H_8IN_3O_2$, the cyanide group is *anti* to the iodide substituent of the adjacent benzene ring. The central segment is essentially planar (r.m.s deviation = 0.0341 Å) and it is twisted away from the iodide- and nitro-substituted benzene rings by 69.02 (9) and 15.83 (16)°, respectively. In the crystal, molecules are linked by weak C—H···N interactions, leading to *C*(8) chains along [010].

3D view



Chemical scheme



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 *o*-iodide

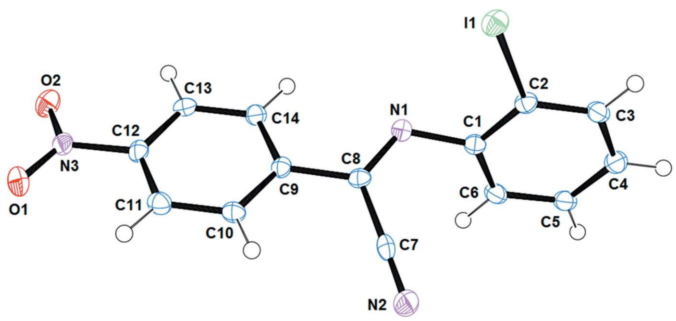


Figure 1
The molecular structure of (I) with displacement ellipsoids drawn at the 50% probability level.

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 intermolecular 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].

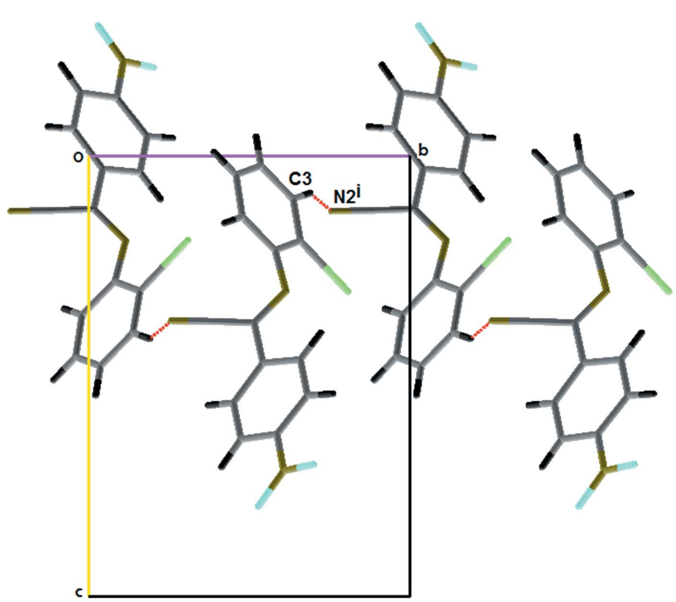


Figure 2
Part of the crystal structure of (I), showing the formation of *C*(8) chains of molecules along [010]. [Symmetry code: (i) $-x, y+\frac{1}{2}, -z+\frac{1}{2}$.]

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C3—H3⋯N2^{i}	0.95	2.61	3.485 (4)	153

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

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.

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Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{14}H_8IN_3O_2$
M_r	377.13
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	123
a, b, c (Å)	11.9620 (7), 9.0103 (5), 13.2047 (8)
β (°)	109.893 (6)
V (Å^3)	1338.29 (13)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	2.40
Crystal size (mm)	0.25 × 0.25 × 0.18
Data collection	
Diffractometer	Oxford Diffraction Xcalibur E
Absorption correction	Multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
T_{min}, T_{max}	0.850, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	6073, 3014, 2602
R_{int}	0.049
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.650
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.032, 0.087, 1.07
No. of reflections	3014
No. of parameters	181
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (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).

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full crystallographic data

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

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Crystal data

$C_{14}H_8IN_3O_2$
M_r = 377.13
Monoclinic, $P2_1/c$
a = 11.9620 (7) Å
b = 9.0103 (5) Å
c = 13.2047 (8) Å
β = 109.893 (6)°
V = 1338.29 (13) Å^3
Z = 4
$F(000)$ = 728
D_x = 1.872 Mg m^{-3}
Melting point: 433(1) K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3743 reflections
θ = 4.8–28.3°
μ = 2.40 mm^{-1}
T = 123 K
Fragment from a large block, orange
0.25 × 0.25 × 0.18 mm

Data collection

Oxford Diffraction Xcalibur E diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
T_{min} = 0.850, T_{max} = 1.000
6073 measured reflections
3014 independent reflections
2602 reflections with $I > 2\sigma(I)$
R_{int} = 0.049
θ_{max} = 27.5°, θ_{min} = 4.8°
$h = -14 \rightarrow 15$
$k = -11 \rightarrow 8$
$l = -17 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.087
S = 1.07
3014 reflections
181 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0437P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 1.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.84 e Å^{-3}

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, $CDCl_3$) δ: 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, $CDCl_3$) δ: 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) [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)

	x	y	z	U_{iso}*/U_{eq}
I1	0.04067 (2)	0.30747 (2)	0.19197 (2)	0.02256 (10)
O1	0.4118 (2)	0.0280 (3)	−0.29485 (18)	0.0305 (6)
O2	0.5255 (2)	0.2000 (3)	−0.1999 (2)	0.0255 (5)
N1	0.2669 (2)	0.1140 (3)	0.1927 (2)	0.0185 (5)
N2	0.1607 (3)	−0.2436 (4)	0.1256 (2)	0.0271 (7)
N3	0.4470 (2)	0.1070 (3)	−0.2146 (2)	0.0199 (6)
C1	0.2371 (3)	0.0802 (3)	0.2856 (2)	0.0164 (6)
C2	0.1477 (3)	0.1590 (3)	0.3068 (2)	0.0172 (6)
C3	0.1250 (3)	0.1364 (4)	0.4016 (2)	0.0190 (6)
H3	0.0627	0.1891	0.4151	0.023*
C4	0.1942 (3)	0.0358 (4)	0.4772 (2)	0.0209 (7)
H4	0.1801	0.0208	0.5430	0.025*
C5	0.2828 (3)	−0.0417 (4)	0.4564 (2)	0.0200 (7)
H5	0.3296	−0.1102	0.5083	0.024*
C6	0.3052 (3)	−0.0219 (4)	0.3611 (3)	0.0199 (7)
H6	0.3662	−0.0770	0.3472	0.024*
C7	0.2035 (3)	−0.1303 (4)	0.1247 (2)	0.0190 (6)
C8	0.2576 (3)	0.0148 (3)	0.1211 (2)	0.0164 (6)
C9	0.3007 (2)	0.0423 (3)	0.0300 (2)	0.0154 (6)
C10	0.2656 (3)	−0.0495 (4)	−0.0605 (2)	0.0179 (6)
H10	0.2106	−0.1277	−0.0657	0.021*
C11	0.3108 (3)	−0.0264 (4)	−0.1428 (2)	0.0202 (7)
H11	0.2864	−0.0866	−0.2056	0.024*
C12	0.3920 (3)	0.0861 (4)	−0.1312 (2)	0.0173 (6)
C13	0.4262 (3)	0.1811 (3)	−0.0439 (3)	0.0172 (6)
H13	0.4802	0.2599	−0.0399	0.021*
C14	0.3799 (3)	0.1588 (3)	0.0377 (2)	0.0180 (6)
H14	0.4020	0.2226	0.0986	0.022*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.02007 (14)	0.02419 (15)	0.02160 (15)	0.00280 (8)	0.00473 (10)	0.00532 (8)
O1	0.0444 (15)	0.0318 (14)	0.0182 (12)	−0.0017 (12)	0.0143 (11)	−0.0027 (10)
O2	0.0223 (12)	0.0294 (14)	0.0269 (13)	−0.0027 (10)	0.0113 (10)	0.0048 (10)
N1	0.0215 (12)	0.0176 (14)	0.0178 (13)	−0.0002 (11)	0.0087 (10)	0.0004 (11)
N2	0.0313 (15)	0.0248 (16)	0.0302 (17)	−0.0088 (14)	0.0169 (13)	−0.0046 (14)
N3	0.0247 (13)	0.0194 (15)	0.0169 (12)	0.0068 (11)	0.0087 (10)	0.0045 (11)
C1	0.0174 (13)	0.0147 (15)	0.0165 (14)	−0.0051 (12)	0.0051 (11)	−0.0040 (12)
C2	0.0181 (14)	0.0161 (15)	0.0158 (15)	−0.0007 (12)	0.0038 (12)	0.0008 (12)
C3	0.0200 (14)	0.0167 (16)	0.0210 (16)	0.0015 (13)	0.0079 (12)	−0.0017 (13)
C4	0.0261 (16)	0.0195 (16)	0.0173 (15)	−0.0023 (13)	0.0074 (13)	−0.0011 (13)
C5	0.0237 (15)	0.0156 (16)	0.0206 (16)	0.0015 (13)	0.0073 (13)	−0.0001 (13)
C6	0.0211 (15)	0.0165 (16)	0.0230 (16)	0.0024 (13)	0.0089 (12)	−0.0020 (13)
C7	0.0209 (14)	0.0253 (18)	0.0123 (14)	0.0020 (14)	0.0077 (12)	−0.0012 (13)
C8	0.0167 (13)	0.0142 (15)	0.0170 (15)	0.0025 (12)	0.0041 (11)	0.0024 (12)
C9	0.0149 (13)	0.0165 (15)	0.0148 (14)	0.0024 (12)	0.0050 (11)	0.0027 (12)
C10	0.0189 (14)	0.0153 (16)	0.0189 (15)	−0.0034 (12)	0.0057 (12)	−0.0031 (12)
C11	0.0228 (15)	0.0186 (16)	0.0168 (15)	0.0011 (13)	0.0036 (12)	−0.0015 (13)
C12	0.0180 (13)	0.0188 (16)	0.0159 (14)	0.0061 (12)	0.0068 (11)	0.0054 (12)
C13	0.0182 (14)	0.0126 (15)	0.0191 (16)	0.0006 (11)	0.0041 (12)	0.0022 (11)
C14	0.0223 (15)	0.0163 (16)	0.0144 (15)	−0.0001 (12)	0.0051 (12)	−0.0003 (12)

Geometric parameters (Å, º)

I1—C2	2.099 (3)	C5—H5	0.9500
O1—N3	1.226 (3)	C6—H6	0.9500
O2—N3	1.223 (4)	C7—C8	1.466 (4)
N1—C8	1.279 (4)	C8—C9	1.482 (4)
N1—C1	1.421 (4)	C9—C14	1.395 (4)
N2—C7	1.144 (4)	C9—C10	1.395 (4)
N3—C12	1.475 (4)	C10—C11	1.384 (4)
C1—C2	1.390 (4)	C10—H10	0.9500
C1—C6	1.397 (4)	C11—C12	1.376 (4)
C2—C3	1.383 (4)	C11—H11	0.9500
C3—C4	1.393 (4)	C12—C13	1.381 (4)
C3—H3	0.9500	C13—C14	1.382 (4)
C4—C5	1.373 (4)	C13—H13	0.9500
C4—H4	0.9500	C14—H14	0.9500
C5—C6	1.385 (4)		
C8—N1—C1	120.2 (3)	N2—C7—C8	178.9 (3)
O2—N3—O1	123.6 (3)	N1—C8—C7	121.9 (3)
O2—N3—C12	118.8 (3)	N1—C8—C9	121.1 (3)
O1—N3—C12	117.5 (3)	C7—C8—C9	117.0 (3)
C2—C1—C6	119.7 (3)	C14—C9—C10	120.5 (3)
C2—C1—N1	120.0 (3)	C14—C9—C8	118.8 (3)

C6—C1—N1	120.0 (3)	C10—C9—C8	120.6 (3)
C3—C2—C1	120.6 (3)	C11—C10—C9	119.9 (3)
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
C5—C6—C1	119.1 (3)	C13—C14—C9	119.6 (3)
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)
C6—C1—C2—C3	−0.5 (5)	C14—C9—C10—C11	1.0 (5)
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, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···N2^{i}	0.95	2.61	3.485 (4)	153

Symmetry code: (i) −*x*, *y*+1/2, −*z*+1/2.