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# 3-(4-Iodophenyl)pentanedinitrile

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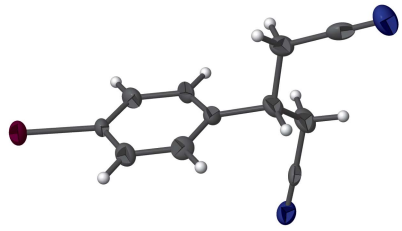
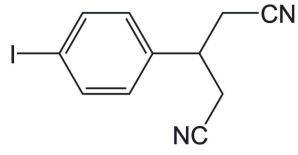
In the title pentanedinitrile derivative, $C_{11}H_9IN_2$, the iodophenyl group is connected at the 3-position. The central propylene chain of the pentanedinitrile moiety contains one *gauche* conformation as a result of steric repulsion with the phenyl ring. Intermolecular close contacts in the crystal comprise a weak $Csp^3$—H···N hydrogen bond and a C—I···N halogen bond.

**3D view**



**Chemical scheme**




## Structure description

The title compound, $C_{11}H_9IN_2$, is a pentanedinitrile (glutaronitrile) derivative, in which the iodophenyl group is connected at the 3-position. Pentanedinitrile derivatives are used as precursors in the synthesis of 2,6-diaminopyridines, which are used as raw materials for insecticides (Kato *et al.*, 1989).

The title compound (Fig. 1) was obtained in a condensation reaction between 4-iodebenzaldehyde and cyanoacetic acid. The central propylene chain of the pentanedinitrile group contains one *gauche* conformation where the torsion angle of C1—C2—C3—C4 is 161.3 (9)°. Similarly, the conformations of related compounds (see *e.g.* Al-Arab *et al.*, 1988; Lorente *et al.*, 1995; Percino *et al.*, 2014) also do not show all-*anti* conformations. In the title compound, steric repulsion between the C1—N1 cyano group and the phenyl ring is thought to result in the *gauche* conformation.

In the crystal, intermolecular halogen bonds are formed between inversion-related molecules to give a dimeric structure (Fig. 2), where the distance of C—I···N$^{i}$ [symmetry code: (i) $-x+1, -y, -z+1$] is 3.369 (7) Å and the C—I···N$^{i}$ angle is 166.2 (2)°. Here, the intermolecular distance is shorter by 4.6% than the sum of the van der Waals radii of the nitrogen and iodine atoms, such that this halogen bond is classified as a weak interaction. Pairs of weak intermolecular $Csp^3$—H···N1$^{ii}$ hydrogen bonds also form between inversion-related dimers (Fig. 2, Table 1).

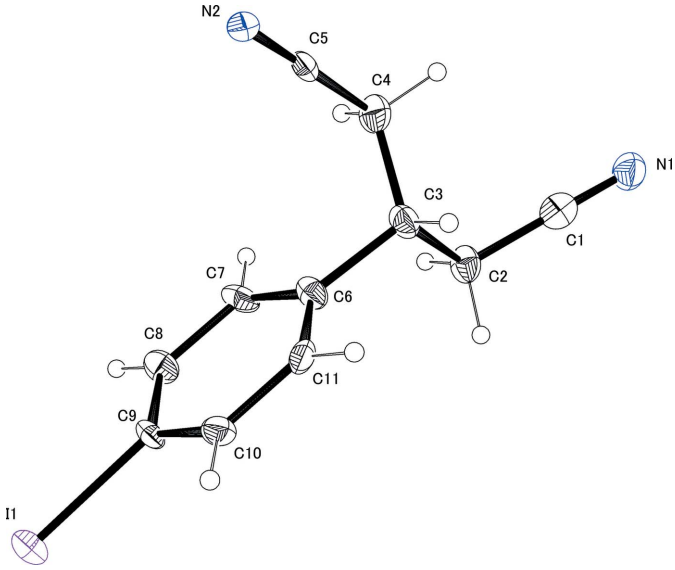



**Figure 1**
The molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level, with H atoms shown as small spheres.

## Synthesis and crystallization

A solution of 4-iodobenzaldehyde (1.0 g, 4.3 mmol) and cyanoacetic acid (0.73 g, 8.6 mmol) in acetonitrile (30 ml) was refluxed overnight with piperidine (0.22 ml) as a basic catalyst. The solution was condensed using a rotary evaporator, and the residual yellowish oil was dissolved in chloroform (90 ml). The solution was washed with $HCl_{aq}$ (1.0 *M*) and water. It was dried over $Na_2SO_4$ and condensed under reduced pressure. The residue was purified by recrystallization from a hexane-ethanol (*v*:*v* = 7:1) solution to give 3-(4-iodophenyl)pentanedinitrile in a yield of 41% (0.52 g).

1H NMR (400 MHz): $\delta$ 7.73 (*d*, *J* = 8.4 Hz, 2H), 7.19 (*d*, *J* = 8.4 Hz, 2H), 3.39 (*m*, 1H), 2.91 (*d*, *J* = 7.4 Hz, 4H)

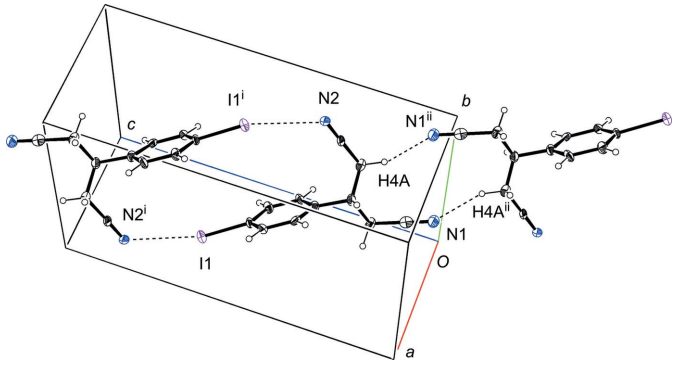



**Figure 2**
A view of the intermolecular interactions in the title compound [symmetry codes:(i) $-x+1, -y, -z+1$; (ii) $-x, -y, -z$].

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

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C4—H4*B*···N1$^{i}$ | 0.99 | 2.61 | 3.387 (12) | 135 |

Symmetry code: (i) $-x, -y, -z$.

**Table 2**
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | $C_{11}H_9IN_2$ |
| $M_r$ | 296.11 |
| Crystal system, space group | Monoclinic, $P2_1/c$ |
| Temperature (K) | 93 |
| *a*, *b*, *c* (Å) | 6.508 (4), 10.524 (6), 16.047 (10) |
| $\beta$ (°) | 90.507 (10) |
| *V* (Å$^3$) | 1099.0 (11) |
| *Z* | 4 |
| Radiation type | Mo $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 2.88 |
| Crystal size (mm) | 0.15 × 0.10 × 0.02 |
| Data collection | |
| Diffractometer | Rigaku Saturn724+ |
| Absorption correction | Numerical (*NUMABS*; Rigaku, 1999) |
| $T_{min}$, $T_{max}$ | 0.744, 0.944 |
| No. of measured, independent and observed [$F^2 > 2.0\sigma(F^2)$] reflections | 7204, 1920, 1618 |
| $R_{int}$ | 0.065 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.595 |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S* | 0.068, 0.095, 1.21 |
| No. of reflections | 1920 |
| No. of parameters | 127 |
| H-atom treatment | H-atom parameters constrained |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 1.02, −0.83 |

Computer programs: *CrystalClear* (Rigaku, 2008), *SIR92* (Altomare *et al.*, 1994), *SHELXL2013* (Sheldrick, 2015), *ORTEP-3 for Windows* (Farrugia, 2012) and *CrystalStructure* (Rigaku, 2014).

## Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

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# full crystallographic data

*IUCrData* (2019). **4**, x190282 [https://doi.org/10.1107/S2414314619002827]

## 3-(4-Iodophenyl)pentanedinitrile

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### 3-(4-Iodophenyl)pentanedinitrile

*Crystal data*

$C_{11}H_9IN_2$
$M_r$ = 296.11
Monoclinic, $P2_1/c$
$a$ = 6.508 (4) Å
$b$ = 10.524 (6) Å
$c$ = 16.047 (10) Å
$\beta$ = 90.507 (10)°
$V$ = 1099.0 (11) Å$^3$
$Z$ = 4
$F(000)$ = 568.00
$D_x$ = 1.790 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71075 Å
Cell parameters from 2574 reflections
$\theta$ = 2.5–25.0°
$\mu$ = 2.88 mm$^{-1}$
$T$ = 93 K
Prism, colorless
0.15 × 0.10 × 0.02 mm

*Data collection*

Rigaku Saturn724+ diffractometer
Detector resolution: 7.111 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: numerical (NUMABS; Rigaku, 1999)
$T_{min}$ = 0.744, $T_{max}$ = 0.944
7204 measured reflections
1920 independent reflections
1618 reflections with $F^2 > 2.0\sigma(F^2)$
$R_{int}$ = 0.065
$\theta_{max}$ = 25.0°, $\theta_{min}$ = 3.1°
$h$ = −7→6
$k$ = −12→12
$l$ = −18→19

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.068
$wR(F^2)$ = 0.095
$S$ = 1.21
1920 reflections
127 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
$w = 1/[\sigma^2(F_o^2) + (0.P)^2 + 7.5421P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.02 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.83 e Å$^{-3}$

*Special details*

**Geometry**. ENTER SPECIAL DETAILS OF THE MOLECULAR GEOMETRY

**Refinement**. Refinement was performed using all reflections. The weighted R-factor (wR) and goodness of fit (S) are based on F$^2$. R-factor (gt) are based on F. The threshold expression of F$^2$ > 2.0 sigma(F$^2$) is used only for calculating R-factor (gt).

The C-bound H atoms were placed at ideal positions and were refined as riding on their parent C atoms. $U_{iso}$(H) values of the H atoms were set at 1.2$U_{eq}$(parent atom for C).

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| I1 | 0.77853 (9) | 0.16532 (6) | 0.51640 (4) | 0.0351 (2) |
| N1 | 0.2457 (12) | 0.0590 (8) | −0.0356 (5) | 0.045 (2) |
| N2 | 0.0921 (11) | −0.2452 (7) | 0.2869 (4) | 0.0362 (19) |
| C1 | 0.2949 (14) | 0.0783 (8) | 0.0303 (6) | 0.035 (2) |
| C2 | 0.3609 (14) | 0.1037 (8) | 0.1178 (5) | 0.035 (2) |
| C3 | 0.3499 (13) | −0.0126 (9) | 0.1745 (5) | 0.034 (2) |
| C4 | 0.1348 (14) | −0.0614 (9) | 0.1804 (5) | 0.041 (2) |
| C5 | 0.1166 (12) | −0.1689 (9) | 0.2395 (5) | 0.0301 (19) |
| C6 | 0.4440 (13) | 0.0237 (8) | 0.2588 (5) | 0.030 (2) |
| C7 | 0.6281 (12) | −0.0277 (8) | 0.2834 (5) | 0.030 (2) |
| C8 | 0.7244 (13) | 0.0096 (9) | 0.3576 (5) | 0.032 (2) |
| C9 | 0.6311 (12) | 0.0997 (8) | 0.4067 (5) | 0.0248 (19) |
| C10 | 0.4428 (13) | 0.1489 (9) | 0.3852 (5) | 0.035 (2) |
| C11 | 0.3499 (13) | 0.1095 (8) | 0.3110 (5) | 0.034 (2) |
| H2A | 0.2728 | 0.1714 | 0.1411 | 0.0425* |
| H2B | 0.50393 | 0.13561 | 0.11769 | 0.0425* |
| H3 | 0.43663 | −0.08104 | 0.14959 | 0.0402* |
| H4A | 0.04378 | 0.00848 | 0.19859 | 0.0497* |
| H4B | 0.08771 | −0.08939 | 0.12454 | 0.0497* |
| H7 | 0.69157 | −0.08991 | 0.24927 | 0.0363* |
| H8 | 0.85222 | −0.02667 | 0.37399 | 0.0389* |
| H10 | 0.37682 | 0.20873 | 0.42025 | 0.0416* |
| H11 | 0.21879 | 0.14254 | 0.29604 | 0.0412* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| I1 | 0.0355 (3) | 0.0376 (3) | 0.0322 (3) | 0.0030 (3) | −0.0112 (2) | −0.0077 (3) |
| N1 | 0.041 (5) | 0.064 (6) | 0.030 (5) | 0.002 (4) | 0.000 (4) | 0.002 (4) |
| N2 | 0.040 (5) | 0.040 (5) | 0.029 (4) | −0.017 (4) | 0.000 (3) | 0.001 (4) |
| C1 | 0.036 (5) | 0.020 (5) | 0.047 (6) | 0.009 (4) | 0.008 (5) | 0.005 (4) |
| C2 | 0.046 (6) | 0.032 (5) | 0.028 (5) | 0.000 (4) | 0.003 (4) | 0.006 (4) |
| C3 | 0.039 (5) | 0.039 (5) | 0.023 (4) | −0.001 (4) | −0.005 (4) | 0.006 (4) |
| C4 | 0.047 (6) | 0.051 (6) | 0.025 (5) | −0.006 (5) | −0.004 (4) | 0.007 (4) |
| C5 | 0.037 (5) | 0.033 (5) | 0.020 (4) | −0.011 (5) | −0.007 (4) | −0.007 (4) |
| C6 | 0.044 (6) | 0.025 (5) | 0.021 (4) | −0.004 (4) | −0.007 (4) | 0.002 (4) |
| C7 | 0.032 (5) | 0.035 (5) | 0.024 (5) | −0.002 (4) | 0.011 (4) | −0.001 (4) |
| C8 | 0.029 (5) | 0.038 (5) | 0.030 (5) | −0.004 (4) | −0.005 (4) | 0.003 (4) |
| C9 | 0.031 (5) | 0.022 (4) | 0.020 (4) | −0.001 (4) | −0.009 (4) | −0.002 (4) |
| C10 | 0.035 (5) | 0.032 (5) | 0.037 (5) | 0.003 (4) | −0.013 (4) | 0.002 (4) |
| C11 | 0.029 (5) | 0.035 (5) | 0.039 (5) | −0.008 (4) | −0.020 (4) | 0.005 (4) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| I1—C9 | 2.113 (8) | C9—C10 | 1.371 (11) |
| N1—C1 | 1.120 (12) | C10—C11 | 1.393 (12) |
| N2—C5 | 1.118 (11) | C2—H2A | 0.990 |
| C1—C2 | 1.489 (13) | C2—H2B | 0.990 |
| C2—C3 | 1.526 (12) | C3—H3 | 1.000 |
| C3—C4 | 1.495 (12) | C4—H4A | 0.990 |
| C3—C6 | 1.529 (11) | C4—H4B | 0.990 |
| C4—C5 | 1.482 (13) | C7—H7 | 0.950 |
| C6—C7 | 1.369 (12) | C8—H8 | 0.950 |
| C6—C11 | 1.379 (12) | C10—H10 | 0.950 |
| C7—C8 | 1.398 (11) | C11—H11 | 0.950 |
| C8—C9 | 1.378 (12) | | |
| | | | |
| N1—C1—C2 | 179.8 (9) | C3—C2—H2A | 108.805 |
| C1—C2—C3 | 113.8 (7) | C3—C2—H2B | 108.800 |
| C2—C3—C4 | 111.2 (7) | H2A—C2—H2B | 107.673 |
| C2—C3—C6 | 107.9 (7) | C2—C3—H3 | 108.068 |
| C4—C3—C6 | 113.4 (7) | C4—C3—H3 | 108.070 |
| C3—C4—C5 | 112.6 (7) | C6—C3—H3 | 108.068 |
| N2—C5—C4 | 175.0 (9) | C3—C4—H4A | 109.087 |
| C3—C6—C7 | 119.9 (7) | C3—C4—H4B | 109.085 |
| C3—C6—C11 | 121.6 (7) | C5—C4—H4A | 109.082 |
| C7—C6—C11 | 118.5 (7) | C5—C4—H4B | 109.080 |
| C6—C7—C8 | 121.3 (8) | H4A—C4—H4B | 107.844 |
| C7—C8—C9 | 119.0 (8) | C6—C7—H7 | 119.373 |
| I1—C9—C8 | 120.1 (6) | C8—C7—H7 | 119.368 |
| I1—C9—C10 | 119.0 (6) | C7—C8—H8 | 120.514 |
| C8—C9—C10 | 120.9 (7) | C9—C8—H8 | 120.523 |
| C9—C10—C11 | 118.9 (8) | C9—C10—H10 | 120.539 |
| C6—C11—C10 | 121.4 (8) | C11—C10—H10 | 120.550 |
| C1—C2—H2A | 108.800 | C6—C11—H11 | 119.287 |
| C1—C2—H2B | 108.802 | C10—C11—H11 | 119.285 |
| | | | |
| C1—C2—C3—C4 | −61.3 (9) | C3—C6—C11—C10 | −175.6 (7) |
| C1—C2—C3—C6 | 173.8 (6) | C7—C6—C11—C10 | 3.2 (12) |
| C2—C3—C4—C5 | −176.1 (6) | C11—C6—C7—C8 | −2.9 (12) |
| C2—C3—C6—C7 | −110.6 (8) | C6—C7—C8—C9 | 0.1 (12) |
| C2—C3—C6—C11 | 68.2 (9) | C7—C8—C9—I1 | −177.0 (6) |
| C4—C3—C6—C7 | 125.8 (8) | C7—C8—C9—C10 | 2.6 (12) |
| C4—C3—C6—C11 | −55.4 (10) | I1—C9—C10—C11 | 177.3 (5) |
| C6—C3—C4—C5 | −54.3 (9) | C8—C9—C10—C11 | −2.3 (12) |
| C3—C6—C7—C8 | 175.9 (7) | C9—C10—C11—C6 | −0.7 (12) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C4—H4*B*···N1$^{i}$ | 0.99 | 2.61 | 3.387 (12) | 135 |

Symmetry code: (i) −*x*, −*y*, −*z*.