# organic compounds

# 4-(6-Chloroimidazo[1,2-*b*]pyridazin-3-yl)benzonitrile

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In the title compound, $C_{13}H_7ClN_4$, the imidazopyridazine ring system is essentially planar [maximum deviation 0.015 (1) Å]. It is inclined to the benzene ring of the benzonitrile group by 11.31 (2)°. In the crystal, molecules are linked *via* C—H···Cl and C—H···N interactions.

## Related literature

For related structures, see Kia *et al.* (2009); Khan *et al.* (2010); Xue (2010); Zhao *et al.* (2009).
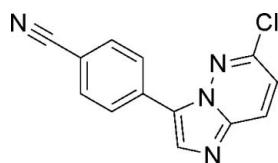


## Experimental

### Crystal data

$C_{13}H_7ClN_4$
$M_r = 254.68$
Tetragonal, $I4_1/a$
$a = 13.5513$ (12) Å
$c = 24.566$ (3) Å
$V = 4511.3$ (7) Å$^3$

$Z = 16$
Mo $K\alpha$ radiation
$\mu = 0.32$ mm$^{-1}$
$T = 150$ K
$0.25 \times 0.15 \times 0.10$ mm

### Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
$T_{min} = 0.659$, $T_{max} = 0.746$

140841 measured reflections
18107 independent reflections
11833 reflections with $I > 2\sigma(I)$
$R_{int} = 0.032$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.141$
$S = 1.08$
18107 reflections
191 parameters

7 restraints
All H-atom parameters refined
$\Delta\rho_{max} = 0.85$ e Å$^{-3}$
$\Delta\rho_{min} = -0.34$ e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C2—H2···N1$^i$ | 1.08 (1) | 2.60 (1) | 3.3502 (7) | 126 (1) |
| C3—H3···Cl1$^{ii}$ | 1.08 (1) | 2.70 (1) | 3.7389 (6) | 161 (1) |
| C5—H5···N4$^{iii}$ | 1.08 (1) | 2.31 (1) | 3.3341 (7) | 157 (1) |
| C8—H8···N4$^{iii}$ | 1.08 (1) | 2.52 (1) | 3.6049 (8) | 177 (1) |
| C12—H12···N2 | 1.08 (1) | 2.25 (1) | 2.9975 (6) | 125 (1) |

Symmetry codes: (i) $x, y + 1, z$; (ii) $-y + \frac{3}{4}, x + \frac{1}{4}, z + \frac{1}{4}$; (iii) $-y + \frac{3}{4}, x - \frac{1}{4}, -z + \frac{3}{4}$.

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1999); software used to prepare material for publication: *SHELXL97* and *publCIF* (Westrip, 2010).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG5227).

## References

Bruker (2008). *SADABS*, *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
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Kia, R., Fun, H.-K. & Kargar, H. (2009). *Acta Cryst.* E**65**, o660–o661.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
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**supplementary materials**

## 4-(6-Chloroimidazo[1,2-*b*]pyridazin-3-yl)benzonitrile

### Y. Zhao and C. K. L. Ng

### Comment

The title compound, $C_{13} H_7 Cl_1 N_4$, was newly synthesized, crystallized and analysed at high resolution sin theta/lambda 1.1. The bond lengths are as expected. The crystal packing reveals that in one unit cell, every four molecules stack in parallel position by a number of non-classical hydrogen bonds. Hydrogen atoms are fixed at 1.083 A as determined from neutron diffraction, and the temperature factors were refined isotropically.

### Experimental

To a thick wall borosilicate glass vial were added 6-Chloro-3-iodo-imidazo[1,2-*b*]pyridazine (0.05 g, 1.0 equiv.), 4-cyan-ophenylboronic acid (0.032 g, 1.2 equiv.), $Pd(PPh_3)_4$ (0.02 g, 10 mol%), $K_2CO_3$ (0.037 g, 1.5 equiv.), and EtOH/H2O (4 : 1, 5 ml). The vial was sealed with a silicon septum and the reaction mixture was pre-stirred for 1 min. The reaction mixture was subjected to microwave irradiation at 90 °C for 14 min at the maximum power of 300 W with sufficient stirring. The reaction mixture was cooled to room temperature and solvent was removed under reduced pressure. The crude product was purified by flash column chromatography, eluting with ethyl acetate/hexane (1 : 1) to give the titled compound as a bright yellow solid (0.038 g, 83 %).

### Figures
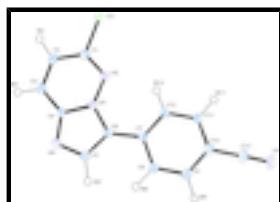


Fig. 1. , The *ORTEP* view of the title compound with 50% probability displacement elipsoids for non-H atoms.

### 4-(6-Chloroimidazo[1,2-*b*]pyridazin-3-yl)benzonitrile

*Crystal data*

| | |
|---|---|
| $C_{13}H_7ClN_4$ | $D_x = 1.500$ Mg m$^{-3}$ |
| $M_r = 254.68$ | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| Tetragonal, $I4_1/a$ | Cell parameters from 500 reflections |
| $a = 13.5513$ (12) Å | $\theta = 1.7$–62.3° |
| $c = 24.566$ (3) Å | $\mu = 0.32$ mm$^{-1}$ |
| $V = 4511.3$ (7) Å$^3$ | $T = 150$ K |
| $Z = 16$ | Block, colourless |
| $F(000) = 2080$ | $0.25 \times 0.15 \times 0.10$ mm |

## Data collection

| | |
|---|---|
| Bruker SMART APEXII CCD diffractometer | 18107 independent reflections |
| Radiation source: fine-focus sealed tube | 11833 reflections with $I > 2\sigma(I)$ |
| graphite | $R_{int} = 0.032$ |
| ω scans | $\theta_{max} = 62.3°$, $\theta_{min} = 1.7°$ |
| Absorption correction: multi-scan (*SAINT*; Bruker, 2008) | $h = -33 \rightarrow 32$ |
| $T_{min} = 0.659$, $T_{max} = 0.746$ | $k = -33 \rightarrow 31$ |
| 140841 measured reflections | $l = -58 \rightarrow 60$ |

## Refinement

| | |
|---|---|
| Refinement on $F^2$ | Primary atom site location: structure-invariant direct methods |
| Least-squares matrix: full | Secondary atom site location: difference Fourier map |
| $R[F^2 > 2\sigma(F^2)] = 0.043$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.141$ | All H-atom parameters refined |
| $S = 1.08$ | $w = 1/[\sigma^2(F_o^2) + (0.0756P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| 18107 reflections | $(\Delta/\sigma)_{max} = 0.001$ |
| 191 parameters | $\Delta\rho_{max} = 0.85$ e Å$^{-3}$ |
| 7 restraints | $\Delta\rho_{min} = -0.34$ e Å$^{-3}$ |

## Special details

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

Reflections -1 11 4, -2 4 0, -5 7 10, 4 4 0, 2 4 2, 1 8 13, -4 8 6, 1 3 16, -2 4 22, -1 10 1, -1 6 1, 0 0 24, -4 6 10, 4 5 11, -2 5 7 were omitted due to bad agreement statistics.

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

| | $x$ | $y$ | $z$ | $U_{iso}*/U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.39217 (3) | 0.36640 (3) | 0.159032 (17) | 0.01678 (5) |
| C2 | 0.37908 (3) | 0.45664 (3) | 0.187490 (19) | 0.01942 (6) |
| C3 | 0.36436 (3) | 0.45225 (3) | 0.242477 (19) | 0.01919 (6) |

| | | | | |
|---|---|---|---|---|
| C4 | 0.36372 (3) | 0.35883 (3) | 0.267513 (16) | 0.01652 (5) |
| C5 | 0.35942 (3) | 0.23262 (3) | 0.319699 (16) | 0.01807 (5) |
| C6 | 0.37353 (3) | 0.19353 (3) | 0.267716 (15) | 0.01514 (5) |
| C7 | 0.37986 (3) | 0.09040 (3) | 0.251063 (15) | 0.01514 (5) |
| C8 | 0.38792 (3) | 0.01863 (3) | 0.292379 (18) | 0.01890 (6) |
| C9 | 0.38734 (4) | −0.08101 (3) | 0.27984 (2) | 0.02124 (7) |
| C10 | 0.38037 (3) | −0.11125 (3) | 0.22543 (2) | 0.01965 (6) |
| C11 | 0.37593 (3) | −0.04085 (3) | 0.183904 (18) | 0.01980 (6) |
| C12 | 0.37476 (3) | 0.05927 (3) | 0.196657 (16) | 0.01772 (5) |
| C13 | 0.37580 (4) | −0.21460 (3) | 0.21251 (2) | 0.02413 (8) |
| N1 | 0.37014 (5) | −0.29780 (4) | 0.20267 (3) | 0.03142 (10) |
| N2 | 0.39085 (2) | 0.27773 (2) | 0.179828 (13) | 0.01557 (4) |
| N3 | 0.37627 (2) | 0.27594 (2) | 0.234420 (13) | 0.01445 (4) |
| N4 | 0.35305 (3) | 0.33273 (3) | 0.319431 (15) | 0.01907 (5) |
| Cl1 | 0.413952 (9) | 0.370824 (9) | 0.089725 (5) | 0.02143 (3) |
| H2 | 0.3846 (8) | 0.5243 (4) | 0.1643 (3) | 0.031 (2)* |
| H3 | 0.3542 (7) | 0.5204 (4) | 0.2647 (4) | 0.030 (2)* |
| H5 | 0.3569 (8) | 0.1898 (7) | 0.3569 (2) | 0.034 (3)* |
| H8 | 0.3999 (8) | 0.0435 (7) | 0.33372 (15) | 0.031 (2)* |
| H9 | 0.3914 (7) | −0.1341 (6) | 0.3127 (3) | 0.029 (2)* |
| H11 | 0.3662 (8) | −0.0628 (9) | 0.14188 (16) | 0.042 (3)* |
| H12 | 0.3662 (7) | 0.1155 (5) | 0.1657 (3) | 0.030 (2)* |

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

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.01593 (12) | 0.01674 (12) | 0.01767 (13) | 0.00052 (9) | −0.00065 (9) | 0.00152 (9) |
| C2 | 0.02065 (14) | 0.01551 (12) | 0.02209 (15) | 0.00112 (10) | −0.00169 (11) | 0.00123 (10) |
| C3 | 0.02072 (14) | 0.01484 (12) | 0.02199 (15) | 0.00197 (10) | −0.00191 (11) | −0.00164 (10) |
| C4 | 0.01706 (12) | 0.01544 (11) | 0.01706 (12) | 0.00196 (9) | −0.00169 (9) | −0.00230 (9) |
| C5 | 0.02051 (14) | 0.01867 (13) | 0.01504 (11) | 0.00221 (10) | −0.00118 (10) | −0.00095 (9) |
| C6 | 0.01548 (11) | 0.01474 (11) | 0.01520 (11) | 0.00104 (8) | −0.00108 (8) | −0.00048 (8) |
| C7 | 0.01486 (11) | 0.01468 (11) | 0.01587 (11) | 0.00048 (8) | −0.00133 (8) | −0.00043 (8) |
| C8 | 0.02312 (15) | 0.01589 (12) | 0.01768 (13) | 0.00058 (10) | −0.00269 (11) | 0.00081 (10) |
| C9 | 0.02647 (18) | 0.01571 (13) | 0.02155 (15) | 0.00072 (11) | −0.00397 (13) | 0.00104 (11) |
| C10 | 0.02066 (14) | 0.01475 (12) | 0.02353 (16) | 0.00046 (10) | −0.00355 (12) | −0.00161 (11) |
| C11 | 0.02297 (16) | 0.01711 (13) | 0.01932 (14) | −0.00005 (11) | −0.00236 (11) | −0.00292 (10) |
| C12 | 0.02068 (14) | 0.01622 (12) | 0.01625 (12) | −0.00001 (10) | −0.00164 (10) | −0.00097 (9) |
| C13 | 0.02702 (19) | 0.01604 (14) | 0.0293 (2) | 0.00087 (12) | −0.00495 (16) | −0.00242 (13) |
| N1 | 0.0399 (3) | 0.01696 (15) | 0.0374 (3) | 0.00031 (15) | −0.0067 (2) | −0.00451 (15) |
| N2 | 0.01569 (10) | 0.01588 (10) | 0.01514 (9) | 0.00048 (7) | −0.00012 (7) | 0.00018 (7) |
| N3 | 0.01422 (9) | 0.01436 (9) | 0.01476 (9) | 0.00097 (7) | −0.00081 (7) | −0.00084 (7) |
| N4 | 0.02264 (13) | 0.01842 (12) | 0.01614 (11) | 0.00297 (9) | −0.00162 (9) | −0.00302 (9) |
| Cl1 | 0.02372 (5) | 0.02314 (5) | 0.01744 (4) | 0.00047 (3) | 0.00083 (3) | 0.00373 (3) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| C1—N2 | 1.3058 (5) | C7—C12 | 1.4033 (5) |
| C1—C2 | 1.4197 (6) | C7—C8 | 1.4100 (6) |

| | | | |
|---|---|---|---|
| C1—Cl1 | 1.7291 (5) | C8—C9 | 1.3849 (6) |
| C2—C3 | 1.3668 (7) | C8—H8 | 1.0824 (10) |
| C2—H2 | 1.0819 (10) | C9—C10 | 1.4014 (7) |
| C3—C4 | 1.4074 (6) | C9—H9 | 1.0824 (10) |
| C3—H3 | 1.0816 (10) | C10—C11 | 1.3980 (7) |
| C4—N4 | 1.3315 (6) | C10—C13 | 1.4374 (6) |
| C4—N3 | 1.3970 (5) | C11—C12 | 1.3925 (6) |
| C5—N4 | 1.3593 (6) | C11—H11 | 1.0825 (10) |
| C5—C6 | 1.3957 (5) | C12—H12 | 1.0823 (10) |
| C5—H5 | 1.0827 (10) | C13—N1 | 1.1557 (7) |
| C6—N3 | 1.3847 (5) | N2—N3 | 1.3558 (5) |
| C6—C7 | 1.4588 (5) | | |
| | | | |
| N2—C1—C2 | 126.74 (4) | C9—C8—C7 | 120.80 (4) |
| N2—C1—Cl1 | 114.80 (3) | C9—C8—H8 | 120.9 (6) |
| C2—C1—Cl1 | 118.45 (3) | C7—C8—H8 | 118.2 (6) |
| C3—C2—C1 | 117.87 (4) | C8—C9—C10 | 119.84 (4) |
| C3—C2—H2 | 124.6 (5) | C8—C9—H9 | 118.8 (6) |
| C1—C2—H2 | 117.5 (5) | C10—C9—H9 | 121.4 (6) |
| C2—C3—C4 | 118.16 (4) | C11—C10—C9 | 119.96 (4) |
| C2—C3—H3 | 118.7 (5) | C11—C10—C13 | 120.14 (4) |
| C4—C3—H3 | 123.2 (5) | C9—C10—C13 | 119.89 (4) |
| N4—C4—N3 | 110.92 (3) | C12—C11—C10 | 120.08 (4) |
| N4—C4—C3 | 131.17 (4) | C12—C11—H11 | 118.7 (7) |
| N3—C4—C3 | 117.91 (4) | C10—C11—H11 | 120.9 (7) |
| N4—C5—C6 | 112.52 (4) | C11—C12—C7 | 120.44 (4) |
| N4—C5—H5 | 122.4 (6) | C11—C12—H12 | 121.9 (5) |
| C6—C5—H5 | 125.0 (6) | C7—C12—H12 | 117.6 (5) |
| N3—C6—C5 | 103.77 (3) | N1—C13—C10 | 178.50 (7) |
| N3—C6—C7 | 127.26 (3) | C1—N2—N3 | 113.92 (3) |
| C5—C6—C7 | 128.92 (4) | N2—N3—C6 | 127.06 (3) |
| C12—C7—C8 | 118.82 (4) | N2—N3—C4 | 125.38 (3) |
| C12—C7—C6 | 123.52 (3) | C6—N3—C4 | 107.54 (3) |
| C8—C7—C6 | 117.62 (3) | C4—N4—C5 | 105.24 (3) |
| | | | |
| N2—C1—C2—C3 | 0.66 (7) | C8—C7—C12—C11 | 1.11 (6) |
| Cl1—C1—C2—C3 | −178.41 (3) | C6—C7—C12—C11 | −176.50 (4) |
| C1—C2—C3—C4 | 0.41 (6) | C11—C10—C13—N1 | −112 (3) |
| C2—C3—C4—N4 | 178.65 (4) | C9—C10—C13—N1 | 67 (3) |
| C2—C3—C4—N3 | −1.24 (6) | C2—C1—N2—N3 | −0.74 (6) |
| N4—C5—C6—N3 | 0.34 (5) | Cl1—C1—N2—N3 | 178.35 (3) |
| N4—C5—C6—C7 | −177.28 (4) | C1—N2—N3—C6 | −178.44 (4) |
| N3—C6—C7—C12 | −10.13 (6) | C1—N2—N3—C4 | −0.23 (5) |
| C5—C6—C7—C12 | 166.96 (4) | C5—C6—N3—N2 | 178.38 (3) |
| N3—C6—C7—C8 | 172.24 (4) | C7—C6—N3—N2 | −3.94 (6) |
| C5—C6—C7—C8 | −10.67 (6) | C5—C6—N3—C4 | −0.09 (4) |
| C12—C7—C8—C9 | −2.26 (6) | C7—C6—N3—C4 | 177.58 (4) |
| C6—C7—C8—C9 | 175.48 (4) | N4—C4—N3—N2 | −178.70 (4) |
| C7—C8—C9—C10 | 1.11 (7) | C3—C4—N3—N2 | 1.21 (6) |
| C8—C9—C10—C11 | 1.22 (7) | N4—C4—N3—C6 | −0.19 (4) |

| C8—C9—C10—C13 | −177.53 (5) | C3—C4—N3—C6 | 179.72 (4) |
| C9—C10—C11—C12 | −2.37 (7) | N3—C4—N4—C5 | 0.39 (5) |
| C13—C10—C11—C12 | 176.38 (4) | C3—C4—N4—C5 | −179.51 (4) |
| C10—C11—C12—C7 | 1.19 (7) | C6—C5—N4—C4 | −0.46 (5) |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
| --- | --- | --- | --- | --- |
| C2—H2···N1[i] | 1.08 (1) | 2.60 (1) | 3.3502 (7) | 126.(1) |
| C3—H3···Cl1[ii] | 1.08 (1) | 2.70 (1) | 3.7389 (6) | 161.(1) |
| C5—H5···N4[iii] | 1.08 (1) | 2.31 (1) | 3.3341 (7) | 157.(1) |
| C8—H8···N4[iii] | 1.08 (1) | 2.52 (1) | 3.6049 (8) | 177.(1) |
| C12—H12···N2 | 1.08 (1) | 2.25 (1) | 2.9975 (6) | 125.(1) |

Symmetry codes: (i) *x*, *y*+1, *z*; (ii) −*y*+3/4, *x*+1/4, *z*+1/4; (iii) −*y*+3/4, *x*−1/4, −*z*+3/4.

**Fig. 1**