Acta Crystallographica Section E
**Structure Reports**
**Online**
ISSN 1600-5368

# 1,3-Dimethyl-1*H*-indole-2-carbonitrile

**Jiang-Sheng Li* and Peng-Mian Huang**

School of Chemistry and Biological Engineering, Changsha University of Science & Technology, Changsha 410004, People's Republic of China
Correspondence e-mail: js_li@yahoo.com.cn

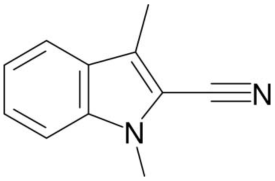
Received 23 June 2009; accepted 27 June 2009

Key indicators: single-crystal X-ray study; $T$ = 113 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.048; $wR$ factor = 0.132; data-to-parameter ratio = 18.1.

The title compound, $C_{11}H_{10}N_2$, crystallizes with two molecules in the asymmetric unit, both of which are essentially planar (r.m.s. deviations = 0.014 and 0.016 Å). In the crystal, aromatic $\pi$–$\pi$ stacking interactions occur [shortest centroid–centroid separation = 3.5569 (11) Å].

## Related literature

For the synthesis, see: Snyder & Eliel (1948).



## Experimental

### *Crystal data*

$C_{11}H_{10}N_2$
$M_r$ = 170.21
Monoclinic, $P2_1/c$
$a$ = 8.8066 (18) Å
$b$ = 15.359 (3) Å
$c$ = 13.480 (3) Å
$\beta$ = 95.67 (3)°
$V$ = 1814.4 (7) Å$^3$
$Z$ = 8
Mo $K\alpha$ radiation
$\mu$ = 0.08 mm$^{-1}$
$T$ = 113 K
0.20 × 0.18 × 0.14 mm

### *Data collection*

Rigaku Saturn CCD area-detector diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
$T_{min}$ = 0.985, $T_{max}$ = 0.990
16175 measured reflections
4303 independent reflections
3462 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.035

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.132
$S$ = 1.03
4303 reflections
238 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.29 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.28 e Å$^{-3}$

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *CrystalStructure* (Rigaku/MSC, 2005).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5013).

## References

Rigaku/MSC (2005). *CrystalClear* and *CrystalStructure*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Snyder, H. R. & Eliel, E. L. (1948). *J. Am. Chem. Soc.* **70**, 1703–1705.

# supplementary materials

*Acta Cryst.* (2009). E**65**, o1759 [ **doi:10.1107/S1600536809024817** ]

# 1,3-Dimethyl-1*H*-indole-2-carbonitrile

**J.-S. Li** and **P.-M. Huang**

## Comment

The asymmetric unit of (I) comprises of two molecules (Fig. 1), in which the indole ring is each almost coplanar with a dihedral angle of 1.32 (7)° and 0.75 (7)°, respectively, between its pyrrole ring and fused benzene ring.

In the crystal packing, strong π-π stacking interactions help establishing the molecular packing.

## Experimental

The title compound was prepared according to the modified method of Snyder & Eliel (1948), as Scheme 1 shows. 1-Methyl-3-dimethylaminomethylindole was added to an ethanolic-aqueous solution (15%, 100 ml) of sodium cyanide (1.87 g, 0.038 mol), and then the resulting mixture was refluxed for 2 h, with the process monitored by TLC. After the reaction ceased, the reaction mixture was extracted with $CH_2Cl_2$ (3 × 50 ml), dried over anhydrous $Na_2SO_4$, and separated by flash chromatograhpy (ethyl acetate-petroleum 10/90 *v*/*v*) to provide the major product 1-methylindole-3-acetonitrile (yield 3.68 g,57%, m.p.328–330 K) and its isomeric substance 1,3-dimethyl-1*H*-indole-2-carbonitrile (yield 0.97 g,15%, m.p.339–340 K). Colourless blocks of (I) were grown from a mixture of ethyl actate and petroleum ether (1:1 *v*/*v*).

## Refinement

All H atoms were positioned geometrically (C—H = 0.95–0.98 Å)and refined as riding with $U_{iso}(H) = 1.2U_{eq}(CH)$ or $1.5U_{eq}(CH_3)$.

## Figures

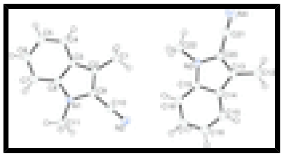


Fig. 1. The two molecular structure of (I) in the asymmetrical unit with the atom-numbering scheme and 50% probability displacement ellipsoids.

Fig. 2. The formation of the title compound.

## 1,3-Dimethyl-1*H*-indole-2-carbonitrile

*Crystal data*

| | |
|---|---|
| $C_{11}H_{10}N_2$ | $F_{000} = 720$ |

$M_r = 170.21$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 8.8066\ (18)$ Å

$b = 15.359\ (3)$ Å

$c = 13.480\ (3)$ Å

$\beta = 95.67\ (3)$°

$V = 1814.4\ (7)$ Å$^3$

$Z = 8$

$D_x = 1.246$ Mg m$^{-3}$

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

Cell parameters from 5208 reflections

$\theta = 2.0$–27.9°

$\mu = 0.08$ mm$^{-1}$

$T = 113$ K

Block, colourless

0.20 × 0.18 × 0.14 mm

## *Data collection*

Rigaku Saturn CCD area-detector diffractometer

Radiation source: rotating anode

Monochromator: confocla

Detector resolution: 7.31 pixels mm$^{-1}$

$T = 113$ K

ω and φ scans

Absorption correction: multi-scan (CrystalClear; Rigaku/MSC, 2005)

$T_{\min} = 0.985$, $T_{\max} = 0.990$

16175 measured reflections

4303 independent reflections

3462 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.035$

$\theta_{\max} = 27.9$°

$\theta_{\min} = 2.0$°

$h = -11 \rightarrow 7$

$k = -20 \rightarrow 20$

$l = -17 \rightarrow 17$

## *Refinement*

Refinement on $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.048$

$wR(F^2) = 0.132$

$S = 1.03$

4303 reflections

238 parameters

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

$w = 1/[\sigma^2(F_o^2) + (0.0783P)^2 + 0.2443P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.003$

$\Delta\rho_{\max} = 0.29$ e Å$^{-3}$

$\Delta\rho_{\min} = -0.27$ e Å$^{-3}$

Extinction correction: none

## *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.

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

| | $x$ | $y$ | $z$ | $U_{\rm iso}$*/$U_{\rm eq}$ |
|---|---|---|---|---|
| N1 | 0.82816 (12) | 0.53565 (7) | 0.89373 (8) | 0.0220 (2) |
| N2 | 0.54466 (13) | 0.37505 (8) | 0.91305 (9) | 0.0323 (3) |
| N3 | 0.68373 (12) | 0.02746 (7) | 0.89897 (8) | 0.0234 (3) |
| N4 | 0.88484 (17) | −0.16898 (9) | 0.90325 (11) | 0.0447 (4) |
| C1 | 0.94864 (16) | 0.30650 (8) | 0.85689 (11) | 0.0294 (3) |
| H1A | 1.0143 | 0.2841 | 0.9142 | 0.044* |
| H1B | 0.9950 | 0.2936 | 0.7954 | 0.044* |
| H1C | 0.8482 | 0.2786 | 0.8544 | 0.044* |
| C2 | 0.93090 (14) | 0.40280 (8) | 0.86704 (9) | 0.0210 (3) |
| C3 | 1.04387 (14) | 0.46744 (8) | 0.85879 (9) | 0.0201 (3) |
| C4 | 1.19845 (15) | 0.46347 (8) | 0.84089 (9) | 0.0237 (3) |
| H4 | 1.2462 | 0.4092 | 0.8308 | 0.028* |
| C5 | 1.27868 (15) | 0.54002 (9) | 0.83840 (10) | 0.0286 (3) |
| H5 | 1.3834 | 0.5382 | 0.8271 | 0.034* |
| C6 | 1.20941 (16) | 0.62085 (9) | 0.85218 (10) | 0.0285 (3) |
| H6 | 1.2680 | 0.6725 | 0.8488 | 0.034* |
| C7 | 1.05797 (15) | 0.62722 (8) | 0.87060 (9) | 0.0247 (3) |
| H7 | 1.0114 | 0.6820 | 0.8800 | 0.030* |
| C8 | 0.97653 (14) | 0.54941 (8) | 0.87480 (9) | 0.0200 (3) |
| C9 | 0.80186 (14) | 0.44673 (8) | 0.88817 (9) | 0.0211 (3) |
| C10 | 0.65833 (15) | 0.40909 (8) | 0.90293 (10) | 0.0242 (3) |
| C11 | 0.71576 (15) | 0.60235 (9) | 0.91016 (10) | 0.0278 (3) |
| H11A | 0.7664 | 0.6514 | 0.9463 | 0.042* |
| H11B | 0.6382 | 0.5779 | 0.9495 | 0.042* |
| H11C | 0.6670 | 0.6226 | 0.8458 | 0.042* |
| C12 | 0.43756 (19) | −0.16808 (9) | 0.85253 (11) | 0.0339 (3) |
| H12A | 0.4196 | −0.1801 | 0.7825 | 0.051* |
| H12B | 0.3426 | −0.1699 | 0.8818 | 0.051* |
| H12C | 0.5057 | −0.2110 | 0.8837 | 0.051* |
| C13 | 0.50716 (16) | −0.07991 (8) | 0.86744 (9) | 0.0239 (3) |
| C14 | 0.43132 (15) | 0.00185 (8) | 0.86448 (9) | 0.0223 (3) |
| C15 | 0.27685 (15) | 0.02613 (9) | 0.84775 (9) | 0.0258 (3) |
| H15 | 0.1994 | −0.0167 | 0.8352 | 0.031* |
| C16 | 0.24020 (16) | 0.11338 (9) | 0.85001 (10) | 0.0280 (3) |
| H16 | 0.1363 | 0.1307 | 0.8392 | 0.034* |
| C17 | 0.35452 (16) | 0.17728 (9) | 0.86808 (10) | 0.0277 (3) |
| H17 | 0.3258 | 0.2369 | 0.8689 | 0.033* |
| C18 | 0.50620 (16) | 0.15567 (8) | 0.88453 (10) | 0.0244 (3) |
| H18 | 0.5827 | 0.1991 | 0.8961 | 0.029* |
| C19 | 0.54362 (14) | 0.06698 (8) | 0.88352 (9) | 0.0205 (3) |
| C20 | 0.66012 (16) | −0.06165 (8) | 0.88826 (9) | 0.0241 (3) |
| C21 | 0.78416 (17) | −0.12122 (9) | 0.89733 (11) | 0.0306 (3) |

| | | | | |
|---|---|---|---|---|
| C22 | 0.83057 (15) | 0.07062 (9) | 0.91704 (11) | 0.0300 (3) |
| H22A | 0.8496 | 0.1053 | 0.8585 | 0.045* |
| H22B | 0.9112 | 0.0268 | 0.9295 | 0.045* |
| H22C | 0.8301 | 0.1088 | 0.9753 | 0.045* |

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

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| N1 | 0.0207 (5) | 0.0209 (5) | 0.0245 (6) | 0.0012 (4) | 0.0027 (4) | −0.0009 (4) |
| N2 | 0.0231 (6) | 0.0319 (6) | 0.0425 (7) | −0.0034 (5) | 0.0063 (5) | −0.0043 (5) |
| N3 | 0.0229 (6) | 0.0213 (5) | 0.0262 (6) | 0.0001 (4) | 0.0027 (4) | 0.0008 (4) |
| N4 | 0.0502 (9) | 0.0360 (7) | 0.0480 (9) | 0.0171 (7) | 0.0053 (7) | 0.0000 (6) |
| C1 | 0.0278 (7) | 0.0219 (6) | 0.0387 (8) | 0.0004 (5) | 0.0039 (6) | 0.0009 (5) |
| C2 | 0.0196 (6) | 0.0219 (6) | 0.0211 (6) | −0.0003 (5) | 0.0001 (5) | 0.0009 (5) |
| C3 | 0.0202 (6) | 0.0211 (6) | 0.0189 (6) | −0.0001 (5) | 0.0007 (5) | 0.0012 (4) |
| C4 | 0.0210 (6) | 0.0268 (6) | 0.0232 (6) | 0.0013 (5) | 0.0023 (5) | 0.0017 (5) |
| C5 | 0.0211 (6) | 0.0385 (8) | 0.0264 (7) | −0.0046 (6) | 0.0028 (5) | 0.0034 (6) |
| C6 | 0.0296 (7) | 0.0278 (7) | 0.0277 (7) | −0.0104 (6) | 0.0010 (6) | 0.0027 (5) |
| C7 | 0.0283 (7) | 0.0212 (6) | 0.0242 (7) | −0.0019 (5) | 0.0008 (5) | −0.0004 (5) |
| C8 | 0.0202 (6) | 0.0220 (6) | 0.0174 (6) | 0.0005 (5) | 0.0001 (5) | 0.0007 (4) |
| C9 | 0.0199 (6) | 0.0222 (6) | 0.0210 (6) | −0.0018 (5) | 0.0011 (5) | 0.0010 (5) |
| C10 | 0.0226 (6) | 0.0245 (6) | 0.0254 (7) | 0.0011 (5) | 0.0022 (5) | −0.0022 (5) |
| C11 | 0.0246 (7) | 0.0275 (6) | 0.0321 (7) | 0.0058 (5) | 0.0064 (6) | −0.0014 (5) |
| C12 | 0.0435 (9) | 0.0250 (7) | 0.0330 (8) | −0.0067 (6) | 0.0024 (6) | −0.0010 (5) |
| C13 | 0.0305 (7) | 0.0211 (6) | 0.0201 (6) | −0.0028 (5) | 0.0028 (5) | 0.0001 (5) |
| C14 | 0.0252 (7) | 0.0236 (6) | 0.0182 (6) | −0.0034 (5) | 0.0030 (5) | −0.0010 (5) |
| C15 | 0.0237 (7) | 0.0317 (7) | 0.0220 (7) | −0.0034 (6) | 0.0015 (5) | −0.0013 (5) |
| C16 | 0.0234 (7) | 0.0355 (7) | 0.0248 (7) | 0.0037 (6) | 0.0013 (5) | 0.0005 (5) |
| C17 | 0.0300 (7) | 0.0254 (6) | 0.0276 (7) | 0.0055 (6) | 0.0024 (6) | 0.0018 (5) |
| C18 | 0.0266 (7) | 0.0213 (6) | 0.0252 (7) | −0.0016 (5) | 0.0019 (5) | 0.0010 (5) |
| C19 | 0.0204 (6) | 0.0220 (6) | 0.0192 (6) | −0.0001 (5) | 0.0018 (5) | 0.0009 (4) |
| C20 | 0.0296 (7) | 0.0200 (6) | 0.0231 (6) | 0.0026 (5) | 0.0045 (5) | 0.0013 (5) |
| C21 | 0.0382 (8) | 0.0249 (6) | 0.0292 (7) | 0.0052 (6) | 0.0053 (6) | −0.0001 (5) |
| C22 | 0.0216 (7) | 0.0292 (7) | 0.0384 (8) | −0.0025 (6) | −0.0013 (6) | 0.0030 (6) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| N1—C8 | 1.3722 (16) | C9—C10 | 1.4217 (17) |
| N1—C9 | 1.3860 (16) | C11—H11A | 0.9800 |
| N1—C11 | 1.4566 (16) | C11—H11B | 0.9800 |
| N2—C10 | 1.1493 (17) | C11—H11C | 0.9800 |
| N3—C19 | 1.3724 (16) | C12—C13 | 1.4917 (18) |
| N3—C20 | 1.3897 (16) | C12—H12A | 0.9600 |
| N3—C22 | 1.4522 (17) | C12—H12B | 0.9600 |
| N4—C21 | 1.1474 (19) | C12—H12C | 0.9600 |
| C1—C2 | 1.4949 (17) | C13—C20 | 1.3774 (19) |
| C1—H1A | 0.9800 | C13—C14 | 1.4211 (18) |
| C1—H1B | 0.9800 | C14—C15 | 1.4071 (19) |
| C1—H1C | 0.9800 | C14—C19 | 1.4123 (18) |

| | | | |
|---|---|---|---|
| C2—C9 | 1.3754 (17) | C15—C16 | 1.3794 (19) |
| C2—C3 | 1.4177 (17) | C15—H15 | 0.9500 |
| C3—C4 | 1.4072 (18) | C16—C17 | 1.410 (2) |
| C3—C8 | 1.4172 (17) | C16—H16 | 0.9500 |
| C4—C5 | 1.3739 (19) | C17—C18 | 1.3730 (19) |
| C4—H4 | 0.9500 | C17—H17 | 0.9500 |
| C5—C6 | 1.404 (2) | C18—C19 | 1.4019 (17) |
| C5—H5 | 0.9500 | C18—H18 | 0.9500 |
| C6—C7 | 1.384 (2) | C20—C21 | 1.4209 (19) |
| C6—H6 | 0.9500 | C22—H22A | 0.9800 |
| C7—C8 | 1.3978 (17) | C22—H22B | 0.9800 |
| C7—H7 | 0.9500 | C22—H22C | 0.9800 |

| | | | |
|---|---|---|---|
| C8—N1—C9 | 107.39 (10) | N1—C11—H11C | 109.5 |
| C8—N1—C11 | 126.43 (11) | H11A—C11—H11C | 109.5 |
| C9—N1—C11 | 126.07 (11) | H11B—C11—H11C | 109.5 |
| C19—N3—C20 | 107.26 (11) | C13—C12—H12A | 109.4 |
| C19—N3—C22 | 126.58 (11) | C13—C12—H12B | 109.5 |
| C20—N3—C22 | 126.05 (11) | H12A—C12—H12B | 109.5 |
| C2—C1—H1A | 109.5 | C13—C12—H12C | 109.5 |
| C2—C1—H1B | 109.5 | H12A—C12—H12C | 109.5 |
| H1A—C1—H1B | 109.5 | H12B—C12—H12C | 109.5 |
| C2—C1—H1C | 109.5 | C20—C13—C14 | 105.86 (11) |
| H1A—C1—H1C | 109.5 | C20—C13—C12 | 126.36 (12) |
| H1B—C1—H1C | 109.5 | C14—C13—C12 | 127.78 (13) |
| C9—C2—C3 | 105.86 (11) | C15—C14—C19 | 119.34 (12) |
| C9—C2—C1 | 126.89 (12) | C15—C14—C13 | 133.08 (12) |
| C3—C2—C1 | 127.24 (11) | C19—C14—C13 | 107.58 (12) |
| C4—C3—C8 | 119.41 (11) | C16—C15—C14 | 118.59 (12) |
| C4—C3—C2 | 132.95 (11) | C16—C15—H15 | 120.7 |
| C8—C3—C2 | 107.62 (11) | C14—C15—H15 | 120.7 |
| C5—C4—C3 | 118.40 (12) | C15—C16—C17 | 121.03 (13) |
| C5—C4—H4 | 120.8 | C15—C16—H16 | 119.5 |
| C3—C4—H4 | 120.8 | C17—C16—H16 | 119.5 |
| C4—C5—C6 | 121.49 (13) | C18—C17—C16 | 121.76 (12) |
| C4—C5—H5 | 119.3 | C18—C17—H17 | 119.1 |
| C6—C5—H5 | 119.3 | C16—C17—H17 | 119.1 |
| C7—C6—C5 | 121.70 (12) | C17—C18—C19 | 117.34 (12) |
| C7—C6—H6 | 119.2 | C17—C18—H18 | 121.3 |
| C5—C6—H6 | 119.2 | C19—C18—H18 | 121.3 |
| C6—C7—C8 | 117.01 (12) | N3—C19—C18 | 129.57 (12) |
| C6—C7—H7 | 121.5 | N3—C19—C14 | 108.50 (11) |
| C8—C7—H7 | 121.5 | C18—C19—C14 | 121.94 (12) |
| N1—C8—C7 | 129.87 (11) | C13—C20—N3 | 110.79 (11) |
| N1—C8—C3 | 108.18 (10) | C13—C20—C21 | 127.91 (12) |
| C7—C8—C3 | 121.96 (12) | N3—C20—C21 | 121.29 (12) |
| C2—C9—N1 | 110.94 (11) | N4—C21—C20 | 178.97 (16) |
| C2—C9—C10 | 126.43 (12) | N3—C22—H22A | 109.5 |
| N1—C9—C10 | 122.63 (11) | N3—C22—H22B | 109.5 |
| N2—C10—C9 | 176.77 (14) | H22A—C22—H22B | 109.5 |

| | | | |
|---|---|---|---|
| N1—C11—H11A | 109.5 | N3—C22—H22C | 109.5 |
| N1—C11—H11B | 109.5 | H22A—C22—H22C | 109.5 |
| H11A—C11—H11B | 109.5 | H22B—C22—H22C | 109.5 |
| C9—C2—C3—C4 | 178.15 (14) | C20—C13—C14—C15 | −179.38 (13) |
| C1—C2—C3—C4 | −0.8 (2) | C12—C13—C14—C15 | −0.1 (2) |
| C9—C2—C3—C8 | −0.48 (14) | C20—C13—C14—C19 | 0.25 (14) |
| C1—C2—C3—C8 | −179.42 (12) | C12—C13—C14—C19 | 179.58 (13) |
| C8—C3—C4—C5 | −0.77 (19) | C19—C14—C15—C16 | 0.34 (18) |
| C2—C3—C4—C5 | −179.27 (13) | C13—C14—C15—C16 | 179.94 (13) |
| C3—C4—C5—C6 | −0.7 (2) | C14—C15—C16—C17 | 0.36 (19) |
| C4—C5—C6—C7 | 1.1 (2) | C15—C16—C17—C18 | −0.3 (2) |
| C5—C6—C7—C8 | −0.1 (2) | C16—C17—C18—C19 | −0.5 (2) |
| C9—N1—C8—C7 | 179.28 (12) | C20—N3—C19—C18 | −178.96 (13) |
| C11—N1—C8—C7 | 2.9 (2) | C22—N3—C19—C18 | −2.4 (2) |
| C9—N1—C8—C3 | −0.91 (14) | C20—N3—C19—C14 | 0.89 (14) |
| C11—N1—C8—C3 | −177.24 (11) | C22—N3—C19—C14 | 177.41 (12) |
| C6—C7—C8—N1 | 178.39 (12) | C17—C18—C19—N3 | −178.95 (12) |
| C6—C7—C8—C3 | −1.40 (19) | C17—C18—C19—C14 | 1.23 (19) |
| C4—C3—C8—N1 | −177.98 (11) | C15—C14—C19—N3 | 178.98 (11) |
| C2—C3—C8—N1 | 0.87 (14) | C13—C14—C19—N3 | −0.71 (14) |
| C4—C3—C8—C7 | 1.85 (19) | C15—C14—C19—C18 | −1.17 (19) |
| C2—C3—C8—C7 | −179.30 (11) | C13—C14—C19—C18 | 179.14 (11) |
| C3—C2—C9—N1 | −0.08 (14) | C14—C13—C20—N3 | 0.30 (14) |
| C1—C2—C9—N1 | 178.86 (12) | C12—C13—C20—N3 | −179.04 (12) |
| C3—C2—C9—C10 | 179.90 (12) | C14—C13—C20—C21 | −178.78 (13) |
| C1—C2—C9—C10 | −1.2 (2) | C12—C13—C20—C21 | 1.9 (2) |
| C8—N1—C9—C2 | 0.62 (14) | C19—N3—C20—C13 | −0.75 (14) |
| C11—N1—C9—C2 | 176.97 (12) | C22—N3—C20—C13 | −177.29 (12) |
| C8—N1—C9—C10 | −179.36 (11) | C19—N3—C20—C21 | 178.41 (12) |
| C11—N1—C9—C10 | −3.01 (19) | C22—N3—C20—C21 | 1.86 (19) |
| C2—C9—C10—N2 | −13 (3) | C13—C20—C21—N4 | 103 (10) |
| N1—C9—C10—N2 | 167 (3) | N3—C20—C21—N4 | −76 (10) |

**Fig. 1**

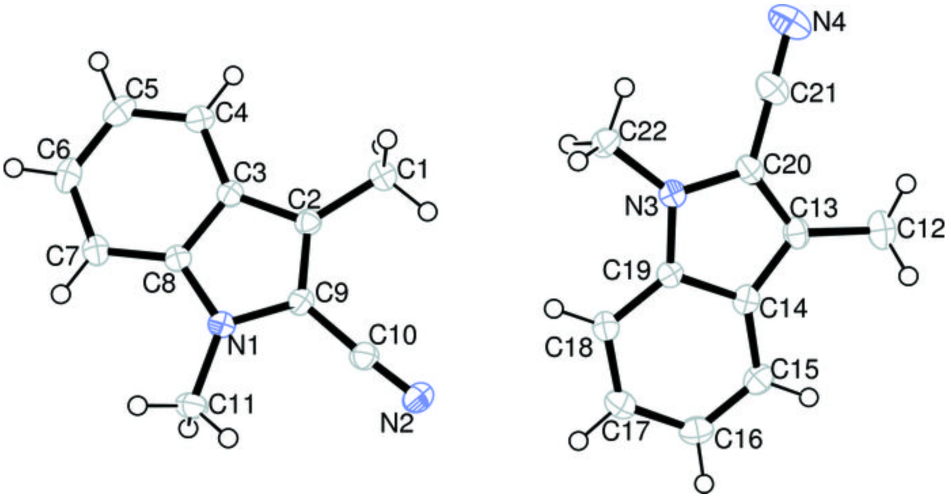
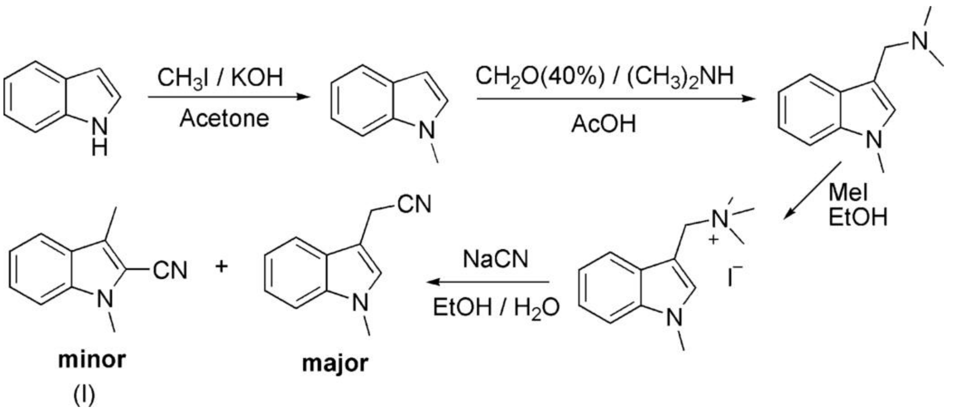

C5
C4
C6
C3
C1
C2
C7
C8
C9
N1
C10
C11
N2
N4
C21
C22
C20
N3
C13
C12
C19
C14
C18
C15
C17
C16

**Fig. 2**


CH3I / KOH
Acetone
CH2O(40%) / (CH3)2NH
AcOH
MeI
EtOH
NaCN
EtOH / H2O
minor
(I)
major