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N-[6-(Dibromomethyl)-2-pyridyl]-2,2-dimethylpropionamide

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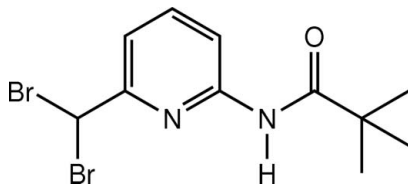
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.006$ Å; R factor = 0.051; wR factor = 0.153; data-to-parameter ratio = 25.4.

In the molecular structure of the title compound, $\text{C}_{11}\text{H}_{14}\text{Br}_2\text{N}_2\text{O}$, the dimethylpropionamide substituent is twisted slightly with respect to the pyridine ring, the interplanar angle being $12.3(2)^\circ$. The dibromomethyl group is orientated in such a way that the two Br atoms are tilted away from the pyridine ring. In the crystal structure, molecules are associated into supramolecular chains by weak $\text{C}-\text{H}\cdots\text{O}$ interactions. The crystal is further stabilized by weak $\text{N}-\text{H}\cdots\text{Br}$ and $\text{C}-\text{H}\cdots\text{N}$ interactions.

Related literature

For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For molecular recognition and *N*-bromosuccinimides, see, for example: Goswami & Mukherjee, (1997); Goswami *et al.* (2000, 2001, 2004). For the stability of the temperature controller used in the data collection, see: Cosier & Glazer (1986).



Experimental

Crystal data

 $\text{C}_{11}\text{H}_{14}\text{Br}_2\text{N}_2\text{O}$
 $M_r = 350.06$

Monoclinic, $P2_1/c$
 $a = 13.2936(7)$ Å
 $b = 8.4660(3)$ Å
 $c = 11.9638(6)$ Å
 $\beta = 99.195(3)^\circ$
 $V = 1329.15(11)$ Å³

$Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 6.08$ mm⁻¹
 $T = 100$ K
 $0.33 \times 0.29 \times 0.24$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2005)
 $T_{\min} = 0.114$, $T_{\max} = 0.233$

12087 measured reflections
 3863 independent reflections
 2954 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.039$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.051$
 $wR(F^2) = 0.153$
 $S = 1.10$
 3863 reflections
 152 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 1.98$ e Å⁻³
 $\Delta\rho_{\text{min}} = -1.13$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|--|--------------|--------------------|-------------|----------------------|
| $\text{N2}-\text{H1N2}\cdots\text{Br1}^{\text{i}}$ | 0.82 (5) | 2.89 (4) | 3.587 (4) | 144 (4) |
| $\text{C3}-\text{H3A}\cdots\text{O1}^{\text{ii}}$ | 0.93 | 2.41 | 3.249 (5) | 151 |
| $\text{C4}-\text{H4A}\cdots\text{O1}$ | 0.93 | 2.34 | 2.886 (5) | 117 |
| $\text{C9}-\text{H9B}\cdots\text{N1}^{\text{iii}}$ | 0.96 | 2.55 | 3.506 (6) | 179 |

Symmetry codes: (i) $-x, y + \frac{1}{2}, -z + \frac{3}{2}$; (ii) $-x + 1, y - \frac{1}{2}, -z + \frac{3}{2}$; (iii) $x, -y + \frac{3}{2}, z - \frac{1}{2}$.

Data collection: APEX2 (Bruker, 2005); cell refinement: SAINT (Bruker, 2005); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL and PLATON (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2385).

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supplementary materials

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N-[6-(Dibromomethyl)-2-pyridyl]-2,2-dimethylpropionamide

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Comment

Bromomethyl aromatic and heteroaromatic compounds (*e.g.* pyridine or naphthyridine derivatives) are important substrates and they have been used as the precursors for pharmacologically active compounds. Bromide compounds have applications in the synthesis of artificial receptors for molecular recognition research (Goswami & Mukherjee, 1997; Goswami *et al.*, 2000). We have also reported the *N*-bromosuccinimide reaction of various heterocycles in the absence or presence of water (Goswami *et al.*, 2001; 2004). We report here the crystal structure of the title compound which is a side-chain substituted with gem-dibromo moiety of pyridine.

In Fig. 1, the O1, N2, C6, C7 atoms lie on the same plane with the maximum deviation of 0.005 (5) Å being for atom C6. The mean plane through these atoms makes the dihedral angle of 12.3 (2)° with the mean plane through pyridine ring. This dihedral angle and the torsion angles C6–N2–C5–C4 = -14.4 (7)°, C5–N2–C6–O1 = 5.2 (7)° and C5–N2–C6–C7 = -174.0 (4)° indicate the orientation of the dimethylpropionamide substituent is slightly twisted with respect to the pyridine ring. The dibromomethyl group on the pyridine ring is orientated in such a way that the two bromine atoms are tilted away from the plane of pyridine ring. A weak intramolecular C4–H4A···O1 contact generates a S(6) ring motif (Bernstein *et al.*, 1995).

The crystal packing shows that the molecules are associated into supramolecular chains via weak C—H···O interactions (Table 1). The crystal is further stabilized by weak interactions of the type N—H···Br and C—H···N (Table 1).

Experimental

To a 100 ml round bottom flask, a mixture of compound 1 (see Fig. 3) (3 g, 0.016 mol) and azobisisobutyronitrile (AIBN) (1.28 g, 7.79 mmol) were added. Dry CCl₄ (30 ml) was added and the reaction mixture was heated to reflux for 30 min with vigorous stirring in the presence of light from a 60 W lamp. When all the materials were dissolved, *N*-bromosuccinimide (NBS) (2.78 g, 0.016 mol) was added slowly and reflux continued for 3 h. The reaction mixture was cooled, crushed ice added, and then extracted with CCl₄ to afford the crude product. The brown liquid was purified by column chromatography over 100–200 mesh silica gel using 3% ethylacetate in petroleum ether (330–350 K) as eluent to yield a white dense liquid of compound 2 (Fig. 3) (2.12 g, yield 50%) and a crystalline solid 3 (2.18 g, yield 40%).

Refinement

The amide-H atom was located in a difference map and refined isotropically; N-H = 0.82 (5)Å. The remaining H atoms were constrained in a riding motion approximation with d(C—H) = 0.93 Å and $U_{iso}=1.2U_{eq}(C)$ for aromatic-H, d(C—H) = 0.98 Å and $U_{iso}=1.2U_{eq}(C)$ for methine-H, and d(C—H) = 0.96 Å and $U_{iso}=1.5U_{eq}(C)$ for methyl-H. A rotating group model was used for the methyl groups. The highest residual electron density peak was located at 0.86 Å from Br1 and the deepest hole was located at 0.86 Å from Br2.

Figures

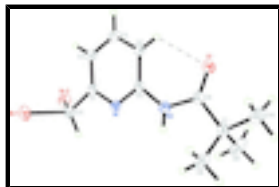


Fig. 1. The molecular structure of (I), showing 50% probability displacement ellipsoids and the atomic numbering. The intramolecular C-H...O contact is drawn as a dashed line.

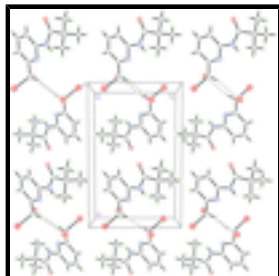


Fig. 2. The crystal packing of the title compound, viewed down the *c* axis showing dimers with $R^2_2(6)$ motifs. Hydrogen bonds were drawn as dashed lines.

***N*-[6-(Dibromomethyl)-2-pyridyl]-2,2-dimethylpropionamide**

Crystal data

$C_{11}H_{14}Br_2N_2O$

$M_r = 350.06$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 13.2936$ (7) Å

$b = 8.4660$ (3) Å

$c = 11.9638$ (6) Å

$\beta = 99.195$ (3)°

$V = 1329.15$ (11) Å³

$Z = 4$

$F(000) = 688$

$D_x = 1.749$ Mg m⁻³

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

Cell parameters from 3863 reflections

$\theta = 1.6$ – 30.0 °

$\mu = 6.08$ mm⁻¹

$T = 100$ K

Block, colorless

$0.33 \times 0.29 \times 0.24$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer

Radiation source: sealed tube graphite

φ and ω scans

Absorption correction: multi-scan (*SADABS*; Bruker, 2005)

$T_{\min} = 0.114$, $T_{\max} = 0.233$

12087 measured reflections

3863 independent reflections

2954 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.039$

$\theta_{\max} = 30.0$ °, $\theta_{\min} = 1.6$ °

$h = -16 \rightarrow 18$

$k = -9 \rightarrow 11$

$l = -16 \rightarrow 16$

Refinement

Refinement on F^2

Primary atom site location: structure-invariant direct methods

| | |
|---------------------------------|--|
| Least-squares matrix: full | Secondary atom site location: difference Fourier map |
| $R[F^2 > 2\sigma(F^2)] = 0.051$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.153$ | H atoms treated by a mixture of independent and constrained refinement |
| $S = 1.10$ | $w = 1/[\sigma^2(F_o^2) + (0.0946P)^2]$ |
| 3863 reflections | where $P = (F_o^2 + 2F_c^2)/3$ |
| 152 parameters | $(\Delta/\sigma)_{\max} < 0.001$ |
| 0 restraints | $\Delta\rho_{\max} = 1.98 \text{ e } \text{\AA}^{-3}$ |
| | $\Delta\rho_{\min} = -1.13 \text{ e } \text{\AA}^{-3}$ |

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

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 (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|-------------|-------------|----------------------------------|
| Br1 | -0.01634 (3) | 0.15918 (5) | 0.82585 (4) | 0.02738 (15) |
| Br2 | 0.08741 (3) | 0.31287 (5) | 1.05799 (4) | 0.02696 (15) |
| O1 | 0.4376 (2) | 0.5484 (4) | 0.6250 (3) | 0.0403 (9) |
| N1 | 0.1855 (2) | 0.4274 (4) | 0.7746 (3) | 0.0188 (6) |
| N2 | 0.2728 (3) | 0.5490 (4) | 0.6511 (3) | 0.0233 (7) |
| C1 | 0.1747 (3) | 0.3175 (4) | 0.8522 (4) | 0.0205 (8) |
| C2 | 0.2499 (3) | 0.2067 (5) | 0.8910 (4) | 0.0250 (9) |
| H2A | 0.2416 | 0.1345 | 0.9474 | 0.030* |
| C3 | 0.3376 (3) | 0.2089 (5) | 0.8420 (4) | 0.0269 (9) |
| H3A | 0.3887 | 0.1351 | 0.8643 | 0.032* |
| C4 | 0.3500 (3) | 0.3196 (4) | 0.7602 (4) | 0.0230 (8) |
| H4A | 0.4084 | 0.3216 | 0.7267 | 0.028* |
| C5 | 0.2719 (3) | 0.4275 (5) | 0.7302 (3) | 0.0191 (7) |
| C6 | 0.3540 (3) | 0.6069 (5) | 0.6056 (4) | 0.0237 (8) |
| C7 | 0.3295 (3) | 0.7544 (5) | 0.5291 (4) | 0.0278 (9) |
| C8 | 0.3325 (5) | 0.8986 (6) | 0.6075 (5) | 0.0463 (13) |
| H8A | 0.3962 | 0.9001 | 0.6584 | 0.070* |
| H8B | 0.2774 | 0.8923 | 0.6502 | 0.070* |
| H8C | 0.3260 | 0.9933 | 0.5629 | 0.070* |
| C9 | 0.2253 (4) | 0.7427 (6) | 0.4534 (5) | 0.0441 (14) |

supplementary materials

| | | | | |
|------|------------|------------|------------|-------------|
| H9A | 0.2227 | 0.6484 | 0.4084 | 0.066* |
| H9B | 0.2152 | 0.8332 | 0.4045 | 0.066* |
| H9C | 0.1727 | 0.7391 | 0.4998 | 0.066* |
| C10 | 0.4117 (4) | 0.7706 (7) | 0.4552 (5) | 0.0452 (14) |
| H10A | 0.4150 | 0.6753 | 0.4124 | 0.068* |
| H10B | 0.4763 | 0.7887 | 0.5022 | 0.068* |
| H10C | 0.3959 | 0.8579 | 0.4043 | 0.068* |
| C11 | 0.0748 (3) | 0.3265 (4) | 0.8950 (3) | 0.0212 (8) |
| H11A | 0.0433 | 0.4283 | 0.8713 | 0.025* |
| H1N2 | 0.224 (3) | 0.609 (6) | 0.638 (4) | 0.023 (12)* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|---------------|--------------|---------------|
| Br1 | 0.0269 (2) | 0.0317 (3) | 0.0252 (2) | -0.00904 (17) | 0.00912 (17) | -0.00740 (16) |
| Br2 | 0.0314 (3) | 0.0321 (3) | 0.0176 (2) | 0.00285 (17) | 0.00477 (17) | 0.00017 (15) |
| O1 | 0.0224 (16) | 0.0365 (19) | 0.064 (3) | 0.0035 (14) | 0.0128 (16) | 0.0234 (17) |
| N1 | 0.0202 (16) | 0.0166 (15) | 0.0189 (15) | -0.0010 (12) | 0.0012 (13) | -0.0006 (12) |
| N2 | 0.0215 (17) | 0.0221 (17) | 0.0271 (18) | 0.0045 (14) | 0.0064 (14) | 0.0044 (14) |
| C1 | 0.0213 (19) | 0.0155 (18) | 0.025 (2) | -0.0025 (14) | 0.0030 (16) | -0.0011 (14) |
| C2 | 0.027 (2) | 0.0176 (18) | 0.031 (2) | 0.0014 (15) | 0.0064 (17) | 0.0041 (16) |
| C3 | 0.026 (2) | 0.0172 (19) | 0.037 (2) | 0.0033 (16) | 0.0029 (18) | 0.0031 (17) |
| C4 | 0.0197 (19) | 0.0169 (19) | 0.033 (2) | 0.0014 (14) | 0.0070 (17) | 0.0001 (15) |
| C5 | 0.0186 (18) | 0.0168 (18) | 0.0219 (18) | -0.0021 (14) | 0.0031 (15) | -0.0005 (14) |
| C6 | 0.0211 (19) | 0.0207 (19) | 0.030 (2) | -0.0017 (15) | 0.0057 (16) | 0.0011 (16) |
| C7 | 0.027 (2) | 0.024 (2) | 0.034 (2) | 0.0004 (17) | 0.0103 (18) | 0.0048 (17) |
| C8 | 0.065 (4) | 0.022 (2) | 0.053 (3) | 0.003 (2) | 0.010 (3) | 0.000 (2) |
| C9 | 0.043 (3) | 0.035 (3) | 0.051 (3) | -0.003 (2) | -0.004 (2) | 0.025 (2) |
| C10 | 0.046 (3) | 0.043 (3) | 0.052 (3) | 0.012 (2) | 0.023 (3) | 0.022 (3) |
| C11 | 0.024 (2) | 0.0194 (19) | 0.0206 (19) | -0.0010 (15) | 0.0044 (15) | -0.0021 (14) |

Geometric parameters (\AA , $^\circ$)

| | | | |
|---------|-----------|----------|-----------|
| Br1—C11 | 1.959 (4) | C4—H4A | 0.9300 |
| Br2—C11 | 1.934 (4) | C6—C7 | 1.551 (6) |
| O1—C6 | 1.205 (5) | C7—C10 | 1.518 (6) |
| N1—C1 | 1.338 (5) | C7—C9 | 1.532 (7) |
| N1—C5 | 1.341 (5) | C7—C8 | 1.536 (7) |
| N2—C6 | 1.374 (5) | C8—H8A | 0.9600 |
| N2—C5 | 1.400 (5) | C8—H8B | 0.9600 |
| N2—H1N2 | 0.82 (5) | C8—H8C | 0.9600 |
| C1—C2 | 1.395 (6) | C9—H9A | 0.9600 |
| C1—C11 | 1.500 (6) | C9—H9B | 0.9600 |
| C2—C3 | 1.386 (6) | C9—H9C | 0.9600 |
| C2—H2A | 0.9300 | C10—H10A | 0.9600 |
| C3—C4 | 1.384 (6) | C10—H10B | 0.9600 |
| C3—H3A | 0.9300 | C10—H10C | 0.9600 |
| C4—C5 | 1.385 (5) | C11—H11A | 0.9800 |

| | | | |
|--------------|------------|---------------|-------------|
| C1—N1—C5 | 117.9 (3) | C9—C7—C6 | 112.4 (4) |
| C6—N2—C5 | 128.5 (4) | C8—C7—C6 | 107.2 (4) |
| C6—N2—H1N2 | 110 (3) | C7—C8—H8A | 109.5 |
| C5—N2—H1N2 | 120 (3) | C7—C8—H8B | 109.5 |
| N1—C1—C2 | 123.2 (4) | H8A—C8—H8B | 109.5 |
| N1—C1—C11 | 113.6 (3) | C7—C8—H8C | 109.5 |
| C2—C1—C11 | 123.2 (4) | H8A—C8—H8C | 109.5 |
| C3—C2—C1 | 117.2 (4) | H8B—C8—H8C | 109.5 |
| C3—C2—H2A | 121.4 | C7—C9—H9A | 109.5 |
| C1—C2—H2A | 121.4 | C7—C9—H9B | 109.5 |
| C4—C3—C2 | 120.7 (4) | H9A—C9—H9B | 109.5 |
| C4—C3—H3A | 119.6 | C7—C9—H9C | 109.5 |
| C2—C3—H3A | 119.6 | H9A—C9—H9C | 109.5 |
| C3—C4—C5 | 117.5 (4) | H9B—C9—H9C | 109.5 |
| C3—C4—H4A | 121.3 | C7—C10—H10A | 109.5 |
| C5—C4—H4A | 121.3 | C7—C10—H10B | 109.5 |
| N1—C5—C4 | 123.4 (4) | H10A—C10—H10B | 109.5 |
| N1—C5—N2 | 111.6 (3) | C7—C10—H10C | 109.5 |
| C4—C5—N2 | 124.9 (4) | H10A—C10—H10C | 109.5 |
| O1—C6—N2 | 122.4 (4) | H10B—C10—H10C | 109.5 |
| O1—C6—C7 | 123.0 (4) | C1—C11—Br2 | 113.7 (3) |
| N2—C6—C7 | 114.6 (3) | C1—C11—Br1 | 110.0 (3) |
| C10—C7—C9 | 109.2 (4) | Br2—C11—Br1 | 109.32 (19) |
| C10—C7—C8 | 109.4 (4) | C1—C11—H11A | 107.9 |
| C9—C7—C8 | 110.2 (4) | Br2—C11—H11A | 107.9 |
| C10—C7—C6 | 108.2 (4) | Br1—C11—H11A | 107.9 |
| C5—N1—C1—C2 | 1.9 (6) | C5—N2—C6—O1 | 5.2 (7) |
| C5—N1—C1—C11 | -179.1 (3) | C5—N2—C6—C7 | -174.0 (4) |
| N1—C1—C2—C3 | -2.8 (6) | O1—C6—C7—C10 | 19.8 (6) |
| C11—C1—C2—C3 | 178.3 (4) | N2—C6—C7—C10 | -161.0 (4) |
| C1—C2—C3—C4 | 1.6 (7) | O1—C6—C7—C9 | 140.5 (5) |
| C2—C3—C4—C5 | 0.3 (7) | N2—C6—C7—C9 | -40.3 (6) |
| C1—N1—C5—C4 | 0.3 (6) | O1—C6—C7—C8 | -98.2 (5) |
| C1—N1—C5—N2 | -179.7 (3) | N2—C6—C7—C8 | 81.0 (5) |
| C3—C4—C5—N1 | -1.3 (6) | N1—C1—C11—Br2 | -133.6 (3) |
| C3—C4—C5—N2 | 178.6 (4) | C2—C1—C11—Br2 | 45.5 (5) |
| C6—N2—C5—N1 | 165.5 (4) | N1—C1—C11—Br1 | 103.5 (3) |
| C6—N2—C5—C4 | -14.4 (7) | C2—C1—C11—Br1 | -77.5 (5) |

Hydrogen-bond geometry (\AA , $^\circ$)

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|-----------------------------------|----------|-------------|-------------|---------------|
| N2—H1N2 \cdots Br1 ⁱ | 0.82 (5) | 2.89 (4) | 3.587 (4) | 144 (4) |
| C3—H3A \cdots O1 ⁱⁱ | 0.93 | 2.41 | 3.249 (5) | 151 |
| C4—H4A \cdots O1 | 0.93 | 2.34 | 2.886 (5) | 117 |
| C9—H9B \cdots N1 ⁱⁱⁱ | 0.96 | 2.55 | 3.506 (6) | 179 |

Symmetry codes: (i) $-x, y+1/2, -z+3/2$; (ii) $-x+1, y-1/2, -z+3/2$; (iii) $x, -y+3/2, z-1/2$.

Fig. 1

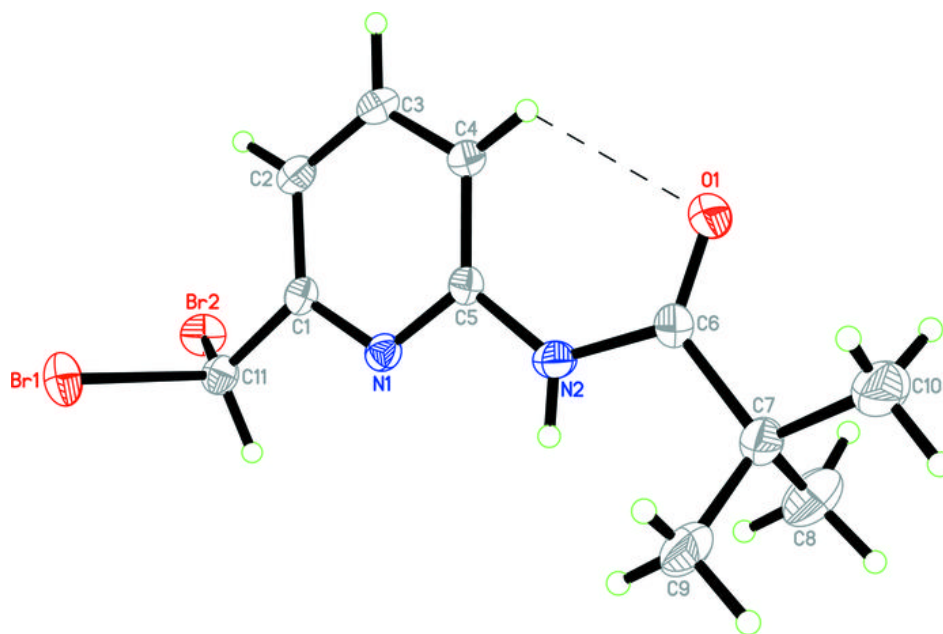


Fig. 2

