

## Piperazinediium bis(2-carboxypyridine-3-carboxylate)

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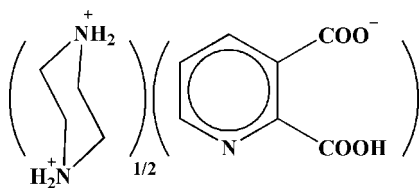
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Key indicators: single-crystal X-ray study;  $T = 100$  K; mean  $\sigma(\text{C}-\text{C}) = 0.001$  Å;  $R$  factor = 0.034;  $wR$  factor = 0.083; data-to-parameter ratio = 17.2.

The asymmetric unit of the title salt,  $\text{C}_4\text{H}_{12}\text{N}_2^{2+} \cdot 2\text{C}_7\text{H}_4\text{NO}_4^-$  or  $\text{pipzH}_2^{2+} \cdot 2(\text{py}-2,3\text{-dcH}^-)$ , prepared by a reaction between pyridine-2,3-dicarboxylic acid (py-2,3-dcH<sub>2</sub>) and piperazine (pipz), contains a monoanion and half of a centrosymmetric dication. The anionic fragment individually has two intramolecular hydrogen bonds, an almost linear O—H···O bond between two carboxylate groups and a C—H···O bond between the aromatic ring and carboxylate group. Other O—H···O, N—H···O and C—H···O hydrogen bonds are responsible for three-dimensional expansion of the structure.

### Related literature

For related literature, see: Aghabozorg *et al.* (2006); Aghabozorg, Daneshvar *et al.* (2007); Aghabozorg, Sadr-khanlou *et al.* (2007); Khalil & Attia (1999); Manteghi *et al.* (2007).



### Experimental

#### Crystal data

$\text{C}_4\text{H}_{12}\text{N}_2^{2+} \cdot 2\text{C}_7\text{H}_4\text{NO}_4^-$   
 $M_r = 420.38$   
 Monoclinic,  $P2_1/c$   
 $a = 8.0116$  (6) Å  
 $b = 11.0588$  (9) Å  
 $c = 10.4621$  (7) Å  
 $\beta = 106.574$  (2)°

$V = 888.42$  (11) Å<sup>3</sup>  
 $Z = 2$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.13$  mm<sup>-1</sup>  
 $T = 100$  (2) K  
 $0.20 \times 0.15 \times 0.15$  mm

#### Data collection

Bruker SMART APEXII diffractometer  
 Absorption correction: none  
 6962 measured reflections

2341 independent reflections  
 2104 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.015$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.034$   
 $wR(F^2) = 0.084$   
 $S = 1.03$   
 2341 reflections

136 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.43$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.22$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H \cdots A$                                       | $D-H$ | $H \cdots A$ | $D \cdots A$ | $D-H \cdots A$ |
|--|-------|--------------|--------------|----------------|
| $\text{O3}-\text{H3O} \cdots \text{O1}$              | 0.85  | 1.57         | 2.4219 (11)  | 175            |
| $\text{N2}-\text{H2A} \cdots \text{O2}^{\text{i}}$   | 0.90  | 1.94         | 2.7778 (12)  | 154            |
| $\text{N2}-\text{H2B} \cdots \text{O1}$              | 0.90  | 1.97         | 2.7571 (11)  | 146            |
| $\text{C3}-\text{H3A} \cdots \text{O4}$              | 0.95  | 2.31         | 2.6773 (13)  | 102            |
| $\text{C8}-\text{H8A} \cdots \text{O2}^{\text{ii}}$  | 0.99  | 2.58         | 3.2982 (13)  | 130            |
| $\text{C9}-\text{H9A} \cdots \text{O4}^{\text{iii}}$ | 0.99  | 2.39         | 3.3491 (14)  | 163            |

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL* and *Mercury* (Version 1.4.2; Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: OM2187).

### References

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**supplementary materials**

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## Piperazinediium bis(2-carboxypyridine-3-carboxylate)

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### Comment

Pyridinedicarboxylic acids are of great interest in medicinal chemistry because of the wide variety of their physiological properties displayed by natural as well as synthetic acids. These acids are present in many natural products, such as alkaloids, vitamins and coenzymes. Pyridinedicarboxylic acid metal complexes are therefore, especially interesting model systems (Khalil & Attia, 1999). Pyridine-2,3-dicarboxylic acid (py-2,3-dcH<sub>2</sub>) and piperazine (pipz) with other acids and bases are found in many ion pairs, such as (pipzH<sub>2</sub>)<sub>2</sub>(pydc) (Aghabozorg *et al.*, 2006) and (pnH<sub>2</sub>)(py-2,3-dc).H<sub>2</sub>O (Manteghi *et al.*, 2007). Moreover, a polymeric complex {(pipzH<sub>2</sub>)[Zn(py-2,3-dc)<sub>2</sub>].4H<sub>2</sub>O}<sub>n</sub> including fragments of the title zwitterion has been synthesized (Aghabozorg, Daneshvar *et al.*, 2007). In all mentioned compounds, piperazine is biprotonated and pyridine-2,3-dicarboxylic acid is doubly deprotonated. But in the title ion pair and a complex formulated as [Zn(py-2,3-dcH)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>] (Aghabozorg, Sadr-khanlou *et al.*, 2007), pyridine-2,3-dicarboxylic acid is singly deprotonated. Fig. 1 shows the monoanion and the dication and the strong intramolecular hydrogen bond (see table of hydrogen-bond geometry). Fig. 2 illustrates the hydrogen bonded layers parallel to *bc* plane. The title ion pair has three C—H...O hydrogen bonds amongst them the C3—H3A...O4 has a short distance (H...O, 2.31 Å) compared with common C—H...O bonds, although its angle is far from linearity. Additionally, as shown in Fig. 3, the ion pair has a  $\pi$ - $\pi$  stacking at the distance of 3.6623 (7) Å between the  $\pi$ -rings (symmetry code: 1 - *x*, -*y*, 1 - *z*).

### Experimental

The title compound was synthesized *via* reaction of 1.67 g (10 mmol) of pyridine-2,3-dicarboxylic acid with 0.86 g (20 mmol) piperazine in a tetrahydrofuran (THF) solution (50 ml). After a while, the obtained white precipitate was filtered out and dissolved in water to recrystallize. Colorless crystals of the title compound were obtained after 1 week.

### Refinement

The H(N) and H(O) atoms were found from difference Fourier map. The H(C) atom positions were calculated. All the hydrogen atoms were refined in isotropic approximation within riding model with the  $U_{\text{iso}}(\text{H})$  parameters equal to 1.2Ueq(Ci), 1.2Ueq(Nj) and 1.5Ueq(O) where U(Ci), U(Cj) and U(O) are the equivalent thermal parameters of the carbon, nitrogen and oxygen atoms correspondingly to which corresponding H atoms are bonded.

### Figures

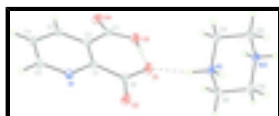


Fig. 1. A view of the ion pair drawn at 50% probability level. Symmetry related atoms are labeled "i" and are generated by 2 - *x*, 1 - *y*, 1 - *z*. Some of the hydrogen bonds are shown and are depicted by dashed lines.

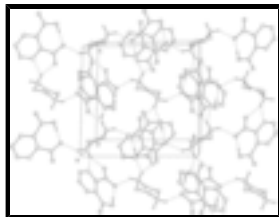


Fig. 2. Hydrogen bonded layers parallel to *bc* plane. Hydrogen atoms are omitted for clarity except those involved in hydrogen bonding.

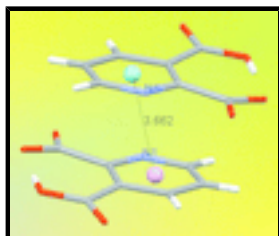
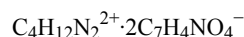


Fig. 3. The  $\pi$ - $\pi$  stacking at the distance of 3.6623 (7) Å (symmetry code: 1 - *x*, -*y*, 1 - *z*).

## Piperazinedium bis(2-carboxypyridine-3-carboxylate)

### Crystal data



$M_r = 420.38$

Monoclinic,  $P2_1/c$

Hall symbol: -P 2ybc

$a = 8.0116$  (6) Å

$b = 11.0588$  (9) Å

$c = 10.4621$  (7) Å

$\beta = 106.574$  (2)°

$V = 888.42$  (11) Å<sup>3</sup>

$Z = 2$

$F(000) = 440$

$D_x = 1.571$  Mg m<sup>-3</sup>

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

Cell parameters from 2095 reflections

$\theta = 2.7$ – $28.6$ °

$\mu = 0.13$  mm<sup>-1</sup>

$T = 100$  K

Prism, colourless

$0.20 \times 0.15 \times 0.15$  mm

### Data collection

Bruker SMART APEXII  
diffractometer

Radiation source: fine-focus sealed tube  
graphite

$\varphi$  and  $\omega$  scans

6962 measured reflections

2341 independent reflections

2104 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.015$

$\theta_{\text{max}} = 29.0$ °,  $\theta_{\text{min}} = 2.7$ °

$h = -10 \rightarrow 10$

$k = -15 \rightarrow 15$

$l = -14 \rightarrow 12$

### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.034$

$wR(F^2) = 0.084$

$S = 1.03$

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: mixed

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.035P)^2 + 0.450P]$

2341 reflections

136 parameters

0 restraints

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} < 0.001$$

$$\Delta\rho_{\max} = 0.43 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.22 \text{ e } \text{\AA}^{-3}$$

### 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.

### Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

|     | <i>x</i>     | <i>y</i>     | <i>z</i>     | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|--------------|----------------------------------|
| O1  | 0.88615 (11) | 0.17340 (7)  | 0.51531 (8)  | 0.02036 (18)                     |
| O2  | 0.98274 (10) | 0.08122 (7)  | 0.70930 (8)  | 0.02041 (18)                     |
| O3  | 0.72179 (11) | 0.13267 (7)  | 0.28657 (8)  | 0.02033 (18)                     |
| H3O | 0.7842       | 0.1467       | 0.3657       | 0.030*                           |
| O4  | 0.54664 (10) | -0.00934 (8) | 0.17740 (8)  | 0.02126 (18)                     |
| N1  | 0.77161 (11) | -0.10247 (8) | 0.63974 (9)  | 0.01409 (18)                     |
| N2  | 0.98062 (11) | 0.40793 (8)  | 0.59237 (8)  | 0.01352 (17)                     |
| H2A | 1.0047       | 0.4441       | 0.6726       | 0.016*                           |
| H2B | 0.9555       | 0.3300       | 0.6036       | 0.016*                           |
| C1  | 0.76624 (12) | -0.02324 (8) | 0.54104 (10) | 0.01174 (18)                     |
| C2  | 0.65530 (12) | -0.04141 (9) | 0.41030 (10) | 0.01209 (19)                     |
| C3  | 0.54476 (13) | -0.14260 (9) | 0.39063 (10) | 0.0147 (2)                       |
| H3A | 0.4655       | -0.1565      | 0.3052       | 0.018*                           |
| C4  | 0.54919 (13) | -0.22223 (9) | 0.49329 (11) | 0.0161 (2)                       |
| H4A | 0.4736       | -0.2902      | 0.4802       | 0.019*                           |
| C5  | 0.66802 (14) | -0.19939 (9) | 0.61616 (10) | 0.0160 (2)                       |
| H5A | 0.6760       | -0.2552      | 0.6867       | 0.019*                           |
| C6  | 0.88903 (13) | 0.08412 (9)  | 0.59330 (10) | 0.01317 (19)                     |
| C7  | 0.63871 (13) | 0.03075 (9)  | 0.28268 (10) | 0.01448 (19)                     |
| C8  | 1.13286 (13) | 0.40868 (9)  | 0.53727 (10) | 0.0146 (2)                       |
| H8A | 1.1067       | 0.3580       | 0.4560       | 0.018*                           |
| H8B | 1.2353       | 0.3736       | 0.6036       | 0.018*                           |
| C9  | 0.82508 (13) | 0.46401 (9)  | 0.49653 (10) | 0.0147 (2)                       |
| H9A | 0.7270       | 0.4659       | 0.5365       | 0.018*                           |
| H9B | 0.7892       | 0.4149       | 0.4141       | 0.018*                           |

## supplementary materials

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### Atomic displacement parameters ( $\text{\AA}^2$ )

|    | $U^{11}$   | $U^{22}$   | $U^{33}$   | $U^{12}$    | $U^{13}$   | $U^{23}$    |
|----|------------|------------|------------|-------------|------------|-------------|
| O1 | 0.0283 (4) | 0.0139 (3) | 0.0167 (4) | -0.0056 (3) | 0.0029 (3) | 0.0024 (3)  |
| O2 | 0.0234 (4) | 0.0200 (4) | 0.0146 (4) | -0.0074 (3) | 0.0003 (3) | 0.0013 (3)  |
| O3 | 0.0268 (4) | 0.0184 (4) | 0.0135 (3) | -0.0025 (3) | 0.0021 (3) | 0.0034 (3)  |
| O4 | 0.0197 (4) | 0.0294 (4) | 0.0129 (4) | -0.0027 (3) | 0.0019 (3) | 0.0005 (3)  |
| N1 | 0.0165 (4) | 0.0130 (4) | 0.0133 (4) | 0.0001 (3)  | 0.0051 (3) | 0.0006 (3)  |
| N2 | 0.0147 (4) | 0.0136 (4) | 0.0120 (4) | -0.0018 (3) | 0.0033 (3) | 0.0006 (3)  |
| C1 | 0.0120 (4) | 0.0110 (4) | 0.0129 (4) | 0.0007 (3)  | 0.0047 (3) | -0.0008 (3) |
| C2 | 0.0119 (4) | 0.0128 (4) | 0.0123 (4) | 0.0023 (3)  | 0.0047 (3) | -0.0005 (3) |
| C3 | 0.0124 (4) | 0.0162 (4) | 0.0158 (5) | 0.0002 (3)  | 0.0044 (4) | -0.0040 (4) |
| C4 | 0.0160 (4) | 0.0131 (4) | 0.0209 (5) | -0.0022 (3) | 0.0082 (4) | -0.0034 (4) |
| C5 | 0.0202 (5) | 0.0125 (4) | 0.0171 (5) | -0.0004 (4) | 0.0084 (4) | 0.0011 (4)  |
| C6 | 0.0136 (4) | 0.0123 (4) | 0.0145 (4) | -0.0005 (3) | 0.0054 (3) | -0.0009 (3) |
| C7 | 0.0130 (4) | 0.0181 (5) | 0.0128 (4) | 0.0035 (3)  | 0.0044 (3) | 0.0017 (4)  |
| C8 | 0.0143 (4) | 0.0156 (4) | 0.0143 (4) | 0.0032 (3)  | 0.0046 (4) | 0.0000 (3)  |
| C9 | 0.0113 (4) | 0.0181 (5) | 0.0138 (4) | -0.0013 (3) | 0.0023 (3) | 0.0003 (4)  |

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

|            |             |                         |             |
|------------|-------------|-------------------------|-------------|
| O1—C6      | 1.2769 (12) | C2—C3                   | 1.4053 (14) |
| O2—C6      | 1.2319 (12) | C2—C7                   | 1.5284 (14) |
| O3—C7      | 1.3036 (13) | C3—C4                   | 1.3815 (15) |
| O3—H3O     | 0.8501      | C3—H3A                  | 0.9500      |
| O4—C7      | 1.2209 (13) | C4—C5                   | 1.3872 (15) |
| N1—C5      | 1.3348 (13) | C4—H4A                  | 0.9500      |
| N1—C1      | 1.3456 (12) | C5—H5A                  | 0.9500      |
| N2—C8      | 1.4903 (13) | C8—C9 <sup>i</sup>      | 1.5129 (14) |
| N2—C9      | 1.4933 (13) | C8—H8A                  | 0.9900      |
| N2—H2A     | 0.9000      | C8—H8B                  | 0.9900      |
| N2—H2B     | 0.9000      | C9—C8 <sup>i</sup>      | 1.5129 (14) |
| C1—C2      | 1.4169 (13) | C9—H9A                  | 0.9900      |
| C1—C6      | 1.5394 (13) | C9—H9B                  | 0.9900      |
| C7—O3—H3O  | 110.0       | N1—C5—H5A               | 118.5       |
| C5—N1—C1   | 119.93 (9)  | C4—C5—H5A               | 118.5       |
| C8—N2—C9   | 110.87 (7)  | O2—C6—O1                | 122.99 (9)  |
| C8—N2—H2A  | 112.1       | O2—C6—C1                | 118.55 (9)  |
| C9—N2—H2A  | 110.9       | O1—C6—C1                | 118.44 (9)  |
| C8—N2—H2B  | 107.1       | O4—C7—O3                | 120.92 (9)  |
| C9—N2—H2B  | 108.1       | O4—C7—C2                | 118.56 (9)  |
| H2A—N2—H2B | 107.5       | O3—C7—C2                | 120.51 (9)  |
| N1—C1—C2   | 121.50 (9)  | N2—C8—C9 <sup>i</sup>   | 110.92 (8)  |
| N1—C1—C6   | 110.63 (8)  | N2—C8—H8A               | 109.5       |
| C2—C1—C6   | 127.85 (9)  | C9 <sup>i</sup> —C8—H8A | 109.5       |
| C3—C2—C1   | 116.74 (9)  | N2—C8—H8B               | 109.5       |
| C3—C2—C7   | 113.19 (8)  | C9 <sup>i</sup> —C8—H8B | 109.5       |

|             |             |                          |              |
|-------------|-------------|--------------------------|--------------|
| C1—C2—C7    | 130.06 (9)  | H8A—C8—H8B               | 108.0        |
| C4—C3—C2    | 121.22 (9)  | N2—C9—C8 <sup>i</sup>    | 110.15 (8)   |
| C4—C3—H3A   | 119.4       | N2—C9—H9A                | 109.6        |
| C2—C3—H3A   | 119.4       | C8 <sup>i</sup> —C9—H9A  | 109.6        |
| C3—C4—C5    | 117.58 (9)  | N2—C9—H9B                | 109.6        |
| C3—C4—H4A   | 121.2       | C8 <sup>i</sup> —C9—H9B  | 109.6        |
| C5—C4—H4A   | 121.2       | H9A—C9—H9B               | 108.1        |
| N1—C5—C4    | 122.94 (9)  |                          |              |
| C5—N1—C1—C2 | -1.52 (14)  | N1—C1—C6—O2              | 5.34 (13)    |
| C5—N1—C1—C6 | 176.91 (9)  | C2—C1—C6—O2              | -176.36 (10) |
| N1—C1—C2—C3 | 3.28 (14)   | N1—C1—C6—O1              | -173.07 (9)  |
| C6—C1—C2—C3 | -174.85 (9) | C2—C1—C6—O1              | 5.23 (15)    |
| N1—C1—C2—C7 | -174.82 (9) | C3—C2—C7—O4              | -6.01 (13)   |
| C6—C1—C2—C7 | 7.04 (16)   | C1—C2—C7—O4              | 172.15 (10)  |
| C1—C2—C3—C4 | -2.13 (14)  | C3—C2—C7—O3              | 174.22 (9)   |
| C7—C2—C3—C4 | 176.30 (9)  | C1—C2—C7—O3              | -7.62 (16)   |
| C2—C3—C4—C5 | -0.66 (15)  | C9—N2—C8—C9 <sup>i</sup> | 57.28 (11)   |
| C1—N1—C5—C4 | -1.56 (15)  | C8—N2—C9—C8 <sup>i</sup> | -56.84 (11)  |
| C3—C4—C5—N1 | 2.64 (15)   |                          |              |

Symmetry codes: (i)  $-x+2, -y+1, -z+1$ .

*Hydrogen-bond geometry (Å, °)*

| <i>D</i> —H $\cdots$ <i>A</i>     | <i>D</i> —H | H $\cdots$ <i>A</i> | <i>D</i> $\cdots$ <i>A</i> | <i>D</i> —H $\cdots$ <i>A</i> |
|-----------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| O3—H3O $\cdots$ O1                | 0.85        | 1.57                | 2.4219 (11)                | 175                           |
| N2—H2A $\cdots$ O2 <sup>ii</sup>  | 0.90        | 1.94                | 2.7778 (12)                | 154                           |
| N2—H2B $\cdots$ O1                | 0.90        | 1.97                | 2.7571 (11)                | 146                           |
| C3—H3A $\cdots$ O4                | 0.95        | 2.31                | 2.6773 (13)                | 102                           |
| C8—H8A $\cdots$ O2 <sup>iii</sup> | 0.99        | 2.58                | 3.2982 (13)                | 130                           |
| C9—H9A $\cdots$ O4 <sup>iv</sup>  | 0.99        | 2.39                | 3.3491 (14)                | 163                           |

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

Fig. 1

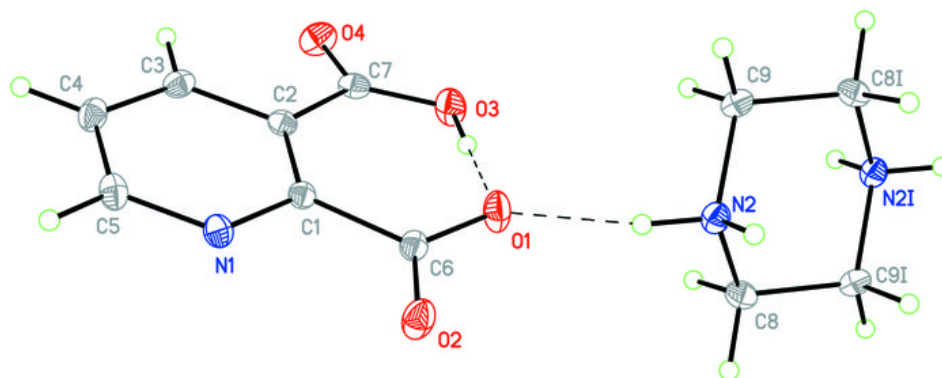




Fig. 2

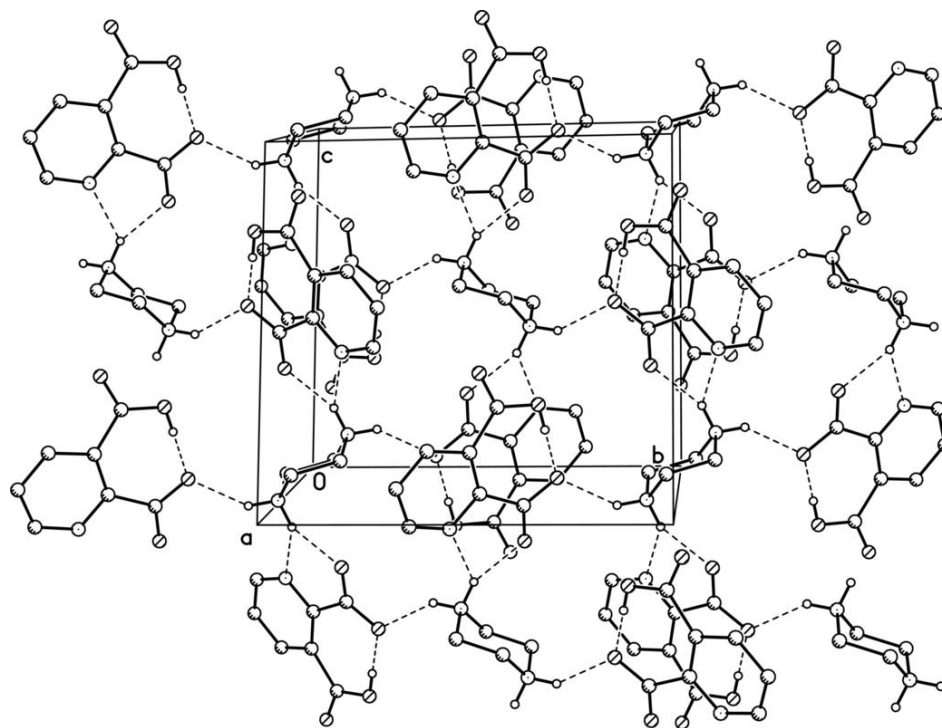


Fig. 3

