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# 4-Amino-2-chlorobenzoic acid

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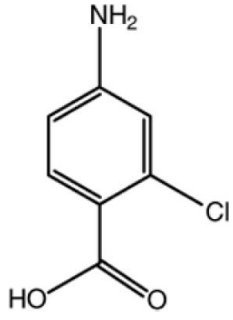
Key indicators: single-crystal X-ray study; $T$ = 296 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.029; $wR$ factor = 0.077; data-to-parameter ratio = 10.5.

The title compound, $C_7H_6ClNO_2$, crystallizes with two roughly planar molecules in the asymmetric unit (r.m.s. deviations = 0.073 and 0.074 Å). The amine H atoms of the two molecules have opposite orientations. In the crystal, molecules are linked into dimers by pairs of O—H···O hydrogen bonds, generating $R_2^2(8)$ loops. N—H···N and N—H···Cl hydrogen bonds link the dimers into a three-dimensional network. The crystal studied was found to be a racemic twin.

## Related literature

For an isomer (2-amino-4-chlorobenzoic acid) of the title compound, see: Farag *et al.* (2011). For hydrogen-bond motifs, see: Bernstein *et al.* (1995).




## Experimental

### Crystal data

$C_7H_6ClNO_2$
$M_r$ = 171.58
Monoclinic, $P2_1$
$a$ = 3.9595 (2) Å
$b$ = 22.6656 (11) Å
$c$ = 8.0285 (4) Å
$\beta$ = 104.257 (2)°
$V$ = 698.32 (6) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.49 mm$^{-1}$
$T$ = 296 K
0.28 × 0.13 × 0.12 mm

### Data collection

Bruker APEXII CCD diffractometer
3009 measured reflections
2295 independent reflections
2197 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.011

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.077
$S$ = 1.07
2295 reflections
218 parameters
7 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.21 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.23 e Å$^{-3}$
Absolute structure: Flack (1983), 514 Freidel pairs
Flack parameter: 0.50 (6)

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O1—H*O1*···O4$^{i}$ | 0.82 (3) | 1.84 (3) | 2.650 (3) | 171 (3) |
| N1—H*N2*···N2 | 0.86 (3) | 2.60 (3) | 3.375 (4) | 150 (3) |
| O3—H*O3*···O2$^{ii}$ | 0.81 (3) | 1.84 (3) | 2.650 (3) | 173 (3) |
| N2—H*N3*···Cl2$^{iii}$ | 0.86 (3) | 2.81 (3) | 3.374 (2) | 125 (2) |
| N2—H*N4*···N1$^{iv}$ | 0.86 (3) | 2.47 (3) | 3.302 (4) | 163 (2) |

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6334).

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# supplementary materials

*Acta Cryst.* (2011). E**67**, o2247 [ **doi:10.1107/S1600536811030728** ]

# 4-Amino-2-chlorobenzoic acid

**M. H. Khan, I. U. Khan and M. Akkurt**

## Comment

The title compound, (I), is used as a starting material for the synthesis of various sulphonamides.

As shown in Fig. 1, the asymmetric unit of the title compound contains two independent molecules. Both have the bond lengths and angles as expected for a molecule of this kind (Farag *et al.*, 2011).

The amine H atoms of the two molecules have opposite orientations. In the crystal, the molecules form dimers *via* intermolecular O—H···O hydrogen bonds, forming a graph-set motif $R^2_2(8)$ (Bernstein *et al.*, 1995; Table 1, Fig. 2). Furthermore, C—H···O, N—H···N and N—H···Cl interactions stabilize the crystal structure.

## Experimental

To a 100-ml round bottom flask equipped with a reflux condenser, was placed 0.5 g (2.486 mmol) of 2-chloro-4-amino benzoic acid and 0.447 g m of granulated tin. Then, 30 ml of concentrated HCl in was added in six intervals (5 ml each time with cooling in ice). The reaction is highly exothermic and the reaction mixture is keept under control by keeping it in ice water. When all the HCl was added and the temprature of the reaction mixture was stable, the round bottom flask was placed on water bath for 90 min under reflux.

TLC check after 90 min showed completion of reaction. Reaction mixtures was treated with 60% NaOH solution followed by the addition of NaCl solution and extration with di-ethyl ether.

Diethyl ether was evaporated on rotary evaporator and reddish brown precipates of the required product were obtained. This was recrystallized in methanol to yield reddish brown prisms of (I).

## Refinement

The H atoms of the $NH_2$ and OH groups in the title compound were located in a difference map and refined with the distance restraint N——H = 0.86 (1) and O——H = 0.82 (1) Å; their $U_{iso}$ values were constrained to be $1.5U_{eq}$ of the carrier atom for hydroxyl H atoms and $1.2U_{eq}$ for amine H atoms. The remaining aromatic H atoms were positioned geometrically with C—H = 0.93 Å, and refined using a riding model, with $U_{iso}$(H) = $1.2U_{eq}$(C). The crystal studied was a racemic twin [Flack parameter = 0.50 (6)].

## Figures

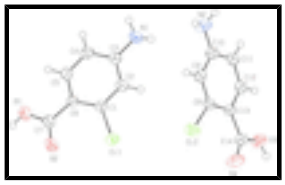

Fig. 1. The molecule of the title compound, with displacement ellipsoids for non-H atoms drawn at the 50% probability level.

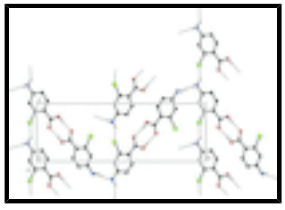

Fig. 2. Partial view of the dimers by two O—H···O hydrogen bonds and the packing in the crystal. Hydrogen atoms that not involved in the hydrogen-bonding (dashed lines) have been omitted for clarity.

## 4-Amino-2-chlorobenzoic acid

*Crystal data*

| | |
|---|---|
| $C_7H_6ClNO_2$ | $F(000) = 352$ |
| $M_r = 171.58$ | $D_x = 1.632$ Mg m$^{-3}$ |
| Monoclinic, $P2_1$ | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| Hall symbol: P 2yb | Cell parameters from 3211 reflections |
| $a = 3.9595$ (2) Å | $\theta = 2.8–28.3°$ |
| $b = 22.6656$ (11) Å | $\mu = 0.49$ mm$^{-1}$ |
| $c = 8.0285$ (4) Å | $T = 296$ K |
| $\beta = 104.257$ (2)° | Prism, reddish brown |
| $V = 698.32$ (6) Å$^3$ | 0.28 × 0.13 × 0.12 mm |
| $Z = 4$ | |

*Data collection*

| | |
|---|---|
| Bruker APEXII CCD diffractometer | 2197 reflections with $I > 2\sigma(I)$ |
| Radiation source: sealed tube | $R_{int} = 0.011$ |
| graphite | $\theta_{max} = 28.3°$, $\theta_{min} = 2.8°$ |
| φ and ω scans | $h = -5\rightarrow5$ |
| 3009 measured reflections | $k = -18\rightarrow30$ |
| 2295 independent reflections | $l = -10\rightarrow10$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.029$ | H atoms treated by a mixture of independent and constrained refinement |
| $wR(F^2) = 0.077$ | $w = 1/[\sigma^2(F_o^2) + (0.0454P)^2 + 0.092P]$ |

| | |
|---|---|
| | where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.07$ | $(\Delta/\sigma)_{max} < 0.001$ |
| 2295 reflections | $\Delta\rho_{max} = 0.21$ e Å$^{-3}$ |
| 218 parameters | $\Delta\rho_{min} = -0.23$ e Å$^{-3}$ |
| 7 restraints | Absolute structure: Flack (1983), 514 Freidel pairs |
| Primary atom site location: structure-invariant direct methods | Flack parameter: 0.50 (6) |

*Special details*

**Geometry**. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

**Refinement**. Refinement on $F^2$ for ALL reflections except those flagged by the user for potential systematic errors. Weighted *R*-factors *wR* and all goodnesses 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 observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating -*R*-factor-obs *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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Cl1 | 0.23950 (15) | 0.81998 (3) | 0.56801 (6) | 0.0427 (2) |
| O1 | −0.2726 (5) | 0.65233 (9) | 0.6710 (2) | 0.0502 (6) |
| O2 | −0.0913 (6) | 0.70711 (10) | 0.4820 (2) | 0.0577 (7) |
| N1 | 0.5447 (5) | 0.83421 (11) | 1.2139 (3) | 0.0431 (7) |
| C1 | 0.2193 (5) | 0.78948 (10) | 0.7631 (3) | 0.0277 (5) |
| C2 | 0.3803 (5) | 0.82200 (12) | 0.9065 (3) | 0.0316 (5) |
| C3 | 0.3834 (5) | 0.80158 (10) | 1.0704 (3) | 0.0306 (6) |
| C4 | 0.2106 (6) | 0.74934 (11) | 1.0871 (3) | 0.0349 (6) |
| C5 | 0.0515 (6) | 0.71758 (11) | 0.9432 (3) | 0.0338 (6) |
| C6 | 0.0508 (5) | 0.73617 (10) | 0.7767 (3) | 0.0294 (6) |
| C7 | −0.1085 (5) | 0.69776 (11) | 0.6301 (3) | 0.0341 (6) |
| Cl2 | 0.80948 (15) | 0.96180 (3) | 0.66956 (7) | 0.0442 (2) |
| O3 | 0.3027 (6) | 1.13060 (8) | 0.7718 (2) | 0.0511 (6) |
| O4 | 0.4834 (7) | 1.07555 (10) | 0.5823 (2) | 0.0581 (7) |
| N2 | 1.1098 (6) | 0.94780 (12) | 1.3131 (3) | 0.0491 (8) |
| C8 | 0.7883 (5) | 0.99282 (10) | 0.8643 (3) | 0.0293 (5) |
| C9 | 0.9466 (5) | 0.96044 (12) | 1.0074 (3) | 0.0321 (5) |
| C10 | 0.9516 (5) | 0.98080 (11) | 1.1714 (3) | 0.0349 (6) |
| C11 | 0.7808 (6) | 1.03355 (12) | 1.1881 (3) | 0.0356 (6) |
| C12 | 0.6218 (6) | 1.06497 (11) | 1.0437 (3) | 0.0347 (6) |
| C13 | 0.6238 (5) | 1.04643 (10) | 0.8776 (3) | 0.0295 (6) |
| C14 | 0.4640 (5) | 1.08498 (11) | 0.7309 (3) | 0.0336 (6) |
| HO1 | −0.335 (8) | 0.6315 (13) | 0.585 (3) | 0.0640* |
| H2 | 0.48700 | 0.85770 | 0.89330 | 0.0380* |
| HN1 | 0.607 (7) | 0.8126 (13) | 1.303 (3) | 0.0510* |

| | | | | |
|---|---|---|---|---|
| HN2 | 0.707 (6) | 0.8583 (11) | 1.203 (4) | 0.0510* |
| H4 | 0.20270 | 0.73590 | 1.19550 | 0.0420* |
| H5 | −0.05990 | 0.68240 | 0.95690 | 0.0410* |
| HO3 | 0.220 (8) | 1.1532 (14) | 0.694 (4) | 0.0660* |
| HN3 | 1.163 (7) | 0.9675 (13) | 1.407 (3) | 0.0530* |
| HN4 | 1.262 (6) | 0.9232 (11) | 1.296 (4) | 0.0530* |
| H9 | 1.05120 | 0.92460 | 0.99420 | 0.0390* |
| H11 | 0.77480 | 1.04740 | 1.29640 | 0.0430* |
| H12 | 0.50820 | 1.09990 | 1.05690 | 0.0420* |

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

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cl1 | 0.0554 (3) | 0.0449 (3) | 0.0274 (2) | −0.0095 (3) | 0.0097 (2) | 0.0044 (2) |
| O1 | 0.0723 (11) | 0.0413 (11) | 0.0365 (9) | −0.0205 (9) | 0.0123 (8) | −0.0066 (8) |
| O2 | 0.0901 (14) | 0.0528 (12) | 0.0312 (8) | −0.0299 (11) | 0.0170 (9) | −0.0109 (9) |
| N1 | 0.0473 (10) | 0.0514 (15) | 0.0276 (9) | −0.0042 (9) | 0.0033 (8) | −0.0061 (9) |
| C1 | 0.0280 (8) | 0.0302 (11) | 0.0252 (9) | 0.0023 (8) | 0.0073 (7) | 0.0022 (8) |
| C2 | 0.0313 (8) | 0.0335 (11) | 0.0290 (9) | 0.0005 (9) | 0.0058 (7) | −0.0018 (10) |
| C3 | 0.0292 (8) | 0.0340 (12) | 0.0281 (9) | 0.0038 (8) | 0.0061 (7) | −0.0020 (8) |
| C4 | 0.0407 (10) | 0.0384 (13) | 0.0267 (10) | 0.0060 (9) | 0.0103 (9) | 0.0025 (9) |
| C5 | 0.0384 (10) | 0.0301 (12) | 0.0332 (11) | −0.0001 (9) | 0.0097 (8) | 0.0019 (9) |
| C6 | 0.0316 (9) | 0.0288 (11) | 0.0274 (10) | 0.0026 (8) | 0.0068 (7) | 0.0004 (8) |
| C7 | 0.0355 (9) | 0.0323 (12) | 0.0328 (11) | −0.0013 (8) | 0.0053 (8) | −0.0034 (9) |
| Cl2 | 0.0540 (3) | 0.0475 (3) | 0.0305 (3) | 0.0080 (3) | 0.0091 (2) | −0.0060 (3) |
| O3 | 0.0750 (12) | 0.0388 (11) | 0.0384 (9) | 0.0208 (10) | 0.0121 (8) | 0.0072 (9) |
| O4 | 0.0890 (14) | 0.0535 (12) | 0.0324 (9) | 0.0303 (11) | 0.0163 (9) | 0.0112 (9) |
| N2 | 0.0545 (11) | 0.0603 (17) | 0.0320 (10) | 0.0120 (11) | 0.0095 (9) | 0.0132 (11) |
| C8 | 0.0304 (8) | 0.0325 (11) | 0.0253 (9) | −0.0041 (8) | 0.0073 (7) | −0.0026 (9) |
| C9 | 0.0319 (8) | 0.0304 (10) | 0.0335 (9) | 0.0030 (9) | 0.0072 (7) | 0.0026 (10) |
| C10 | 0.0322 (9) | 0.0419 (13) | 0.0298 (10) | −0.0042 (9) | 0.0062 (8) | 0.0083 (9) |
| C11 | 0.0419 (10) | 0.0379 (13) | 0.0266 (10) | −0.0022 (9) | 0.0077 (9) | −0.0037 (9) |
| C12 | 0.0421 (10) | 0.0308 (11) | 0.0314 (10) | −0.0001 (9) | 0.0094 (8) | −0.0029 (9) |
| C13 | 0.0310 (9) | 0.0290 (11) | 0.0278 (10) | −0.0019 (8) | 0.0057 (8) | 0.0033 (8) |
| C14 | 0.0391 (10) | 0.0322 (12) | 0.0291 (10) | 0.0004 (9) | 0.0078 (8) | 0.0039 (9) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| Cl1—C1 | 1.732 (2) | C3—C4 | 1.390 (3) |
| Cl2—C8 | 1.735 (2) | C4—C5 | 1.375 (3) |
| O1—C7 | 1.302 (3) | C5—C6 | 1.401 (3) |
| O2—C7 | 1.226 (3) | C6—C7 | 1.475 (3) |
| O1—HO1 | 0.82 (3) | C2—H2 | 0.9300 |
| O3—C14 | 1.299 (3) | C4—H4 | 0.9300 |
| O4—C14 | 1.233 (3) | C5—H5 | 0.9300 |
| O3—HO3 | 0.81 (3) | C8—C9 | 1.377 (3) |
| N1—C3 | 1.386 (3) | C8—C13 | 1.395 (3) |
| N1—HN1 | 0.85 (3) | C9—C10 | 1.391 (3) |
| N1—HN2 | 0.86 (3) | C10—C11 | 1.396 (4) |

| | | | |
|---|---|---|---|
| N2—C10 | 1.377 (3) | C11—C12 | 1.374 (3) |
| N2—HN4 | 0.86 (3) | C12—C13 | 1.400 (3) |
| N2—HN3 | 0.86 (3) | C13—C14 | 1.478 (3) |
| C1—C6 | 1.398 (3) | C9—H9 | 0.9300 |
| C1—C2 | 1.383 (3) | C11—H11 | 0.9300 |
| C2—C3 | 1.392 (3) | C12—H12 | 0.9300 |
| C7—O1—HO1 | 108 (2) | C5—C4—H4 | 120.00 |
| C14—O3—HO3 | 116 (2) | C3—C4—H4 | 120.00 |
| C3—N1—HN1 | 111.7 (19) | C4—C5—H5 | 119.00 |
| HN1—N1—HN2 | 112 (3) | C6—C5—H5 | 119.00 |
| C3—N1—HN2 | 117 (2) | C9—C8—C13 | 121.7 (2) |
| C10—N2—HN4 | 115 (2) | Cl2—C8—C9 | 115.01 (18) |
| HN3—N2—HN4 | 117 (3) | Cl2—C8—C13 | 123.30 (18) |
| C10—N2—HN3 | 114.0 (19) | C8—C9—C10 | 120.7 (2) |
| Cl1—C1—C2 | 115.19 (18) | N2—C10—C11 | 121.2 (2) |
| Cl1—C1—C6 | 123.03 (18) | C9—C10—C11 | 118.7 (2) |
| C2—C1—C6 | 121.8 (2) | N2—C10—C9 | 120.0 (2) |
| C1—C2—C3 | 120.3 (2) | C10—C11—C12 | 119.7 (2) |
| C2—C3—C4 | 119.0 (2) | C11—C12—C13 | 122.6 (2) |
| N1—C3—C4 | 120.8 (2) | C8—C13—C12 | 116.5 (2) |
| N1—C3—C2 | 120.2 (2) | C8—C13—C14 | 124.8 (2) |
| C3—C4—C5 | 120.0 (2) | C12—C13—C14 | 118.7 (2) |
| C4—C5—C6 | 122.5 (2) | O3—C14—C13 | 114.2 (2) |
| C5—C6—C7 | 118.9 (2) | O4—C14—C13 | 123.5 (2) |
| C1—C6—C7 | 124.5 (2) | O3—C14—O4 | 122.3 (2) |
| C1—C6—C5 | 116.5 (2) | C8—C9—H9 | 120.00 |
| O2—C7—C6 | 123.8 (2) | C10—C9—H9 | 120.00 |
| O1—C7—C6 | 114.0 (2) | C10—C11—H11 | 120.00 |
| O1—C7—O2 | 122.1 (2) | C12—C11—H11 | 120.00 |
| C1—C2—H2 | 120.00 | C11—C12—H12 | 119.00 |
| C3—C2—H2 | 120.00 | C13—C12—H12 | 119.00 |
| Cl1—C1—C2—C3 | −179.44 (17) | Cl2—C8—C9—C10 | 179.22 (17) |
| C6—C1—C2—C3 | 1.1 (3) | C13—C8—C9—C10 | −0.9 (3) |
| Cl1—C1—C6—C5 | −179.00 (17) | Cl2—C8—C13—C12 | 178.57 (17) |
| Cl1—C1—C6—C7 | 4.2 (3) | Cl2—C8—C13—C14 | −3.3 (3) |
| C2—C1—C6—C5 | 0.4 (3) | C9—C8—C13—C12 | −1.3 (3) |
| C2—C1—C6—C7 | −176.4 (2) | C9—C8—C13—C14 | 176.8 (2) |
| C1—C2—C3—N1 | −180.0 (2) | C8—C9—C10—N2 | 179.8 (2) |
| C1—C2—C3—C4 | −2.5 (3) | C8—C9—C10—C11 | 2.6 (3) |
| N1—C3—C4—C5 | 179.9 (2) | N2—C10—C11—C12 | −179.2 (2) |
| C2—C3—C4—C5 | 2.5 (3) | C9—C10—C11—C12 | −2.0 (3) |
| C3—C4—C5—C6 | −1.0 (4) | C10—C11—C12—C13 | −0.2 (4) |
| C4—C5—C6—C1 | −0.5 (3) | C11—C12—C13—C8 | 1.9 (3) |
| C4—C5—C6—C7 | 176.5 (2) | C11—C12—C13—C14 | −176.4 (2) |
| C1—C6—C7—O1 | −175.4 (2) | C8—C13—C14—O3 | 175.1 (2) |
| C1—C6—C7—O2 | 4.4 (4) | C8—C13—C14—O4 | −5.7 (4) |
| C5—C6—C7—O1 | 7.8 (3) | C12—C13—C14—O3 | −6.8 (3) |
| C5—C6—C7—O2 | −172.3 (2) | C12—C13—C14—O4 | 172.4 (2) |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1—HO1···O4$^{i}$ | 0.82 (3) | 1.84 (3) | 2.650 (3) | 171 (3) |
| N1—HN2···N2 | 0.86 (3) | 2.60 (3) | 3.375 (4) | 150 (3) |
| O3—HO3···O2$^{ii}$ | 0.81 (3) | 1.84 (3) | 2.650 (3) | 173 (3) |
| N2—HN3···Cl2$^{iii}$ | 0.86 (3) | 2.81 (3) | 3.374 (2) | 125 (2) |
| N2—HN4···N1$^{iv}$ | 0.86 (3) | 2.47 (3) | 3.302 (4) | 163 (2) |

Symmetry codes: (i) −*x*, *y*−1/2, −*z*+1; (ii) −*x*, *y*+1/2, −*z*+1; (iii) *x*, *y*, *z*+1; (iv) *x*+1, *y*, *z*.

**Fig. 1**

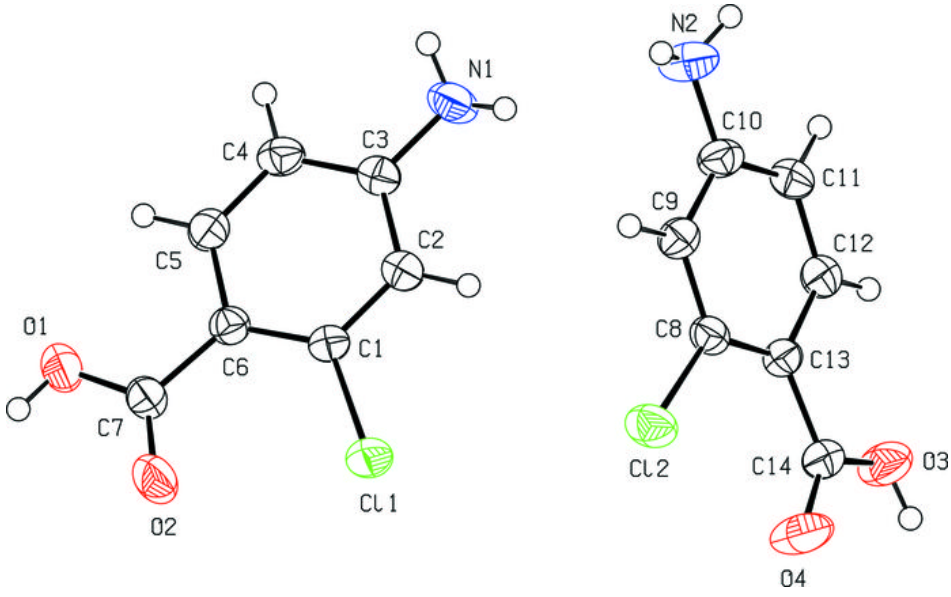
N1
C4
C3
C5
C2
O1
C6
C1
C7
O2
Cl1
N2
C10
C11
C9
C12
C8
C13
Cl2
C14
O3
O4

**Fig. 2**