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# 5-Anilino-4-chloro-3*H*-1,2-dithiol-3-one

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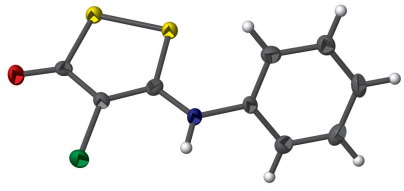
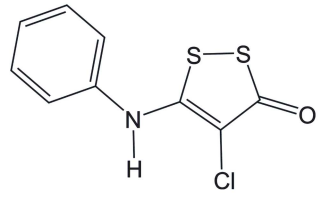
**Structural data**: full structural data are available from iucrdata.iucr.org

In the title compound, $C_9H_6ClNOS_2$, the two rings subtend a dihedral angle of 51.9 (7)°. The S—S bond has a length of 2.061 (2) Å. In the crystal, hydrogen-bonding interactions and $\pi$–$\pi$ stacking [centroid–centroid distance = 3.927 (2) Å] contacts link the molecules into a three-dimensional network.

**3D view**



**Chemical scheme**




## Structure description

The title compound, $C_9H_6ClONS_2$, is a derivative of 1,2-dithiole-3-one, a family of bioactive compounds (He *et al.*, 2004). It crystallizes from mixture of ethanol and dichloromethane in the monoclinic space group $P2_1/n$ (Fig. 1). The molecule is composed of two rings with a dihedral angle of 51.9 (7)° between them. The length of the S—S bond is 2.061 (2) Å and the angles C9—N8—C3, C3—S2—S1, S2—S1—C5 and C5—C4—C3 are 126.2 (4), 94.5 (2), 96.8 (2) and 120.5 (4)°, respectively.

In the crystal (Figs. 2 and 3), the three-dimensional molecular packing is sustained by hydrogen-bonding interactions (C10—H101···O6$^{i}$, N8—H81···O6$^{ii}$ with H···*A* lengths of 2.55 and 1.99 Å, respectively; Table 1) and parallel-displaced $\pi$–$\pi$ aromatic-stacking [centroid–centroid distance = 3.927 (2) Å] contacts between successive molecules in the [100] direction.

## Synthesis and crystallization

To a methanol solution (50 ml) of 4,5-dichloro-1,2-dithiol-3-one ($C_3Cl_2OS_2$, 1 g) and $NaHCO_3$ (0.5 g), 0.6 g of aniline was added. The mixture was stirred for 20 h at room temperature. Then, 100 ml of distilled water was added, and the formed precipitate was filtered and washed 3 times with distilled water and dried. The product was crystallized in an ethyl acetate solution in 80% yield. The recrystallization process was performed from a 1:1 mixture of ethanol and dichloromethane solution.

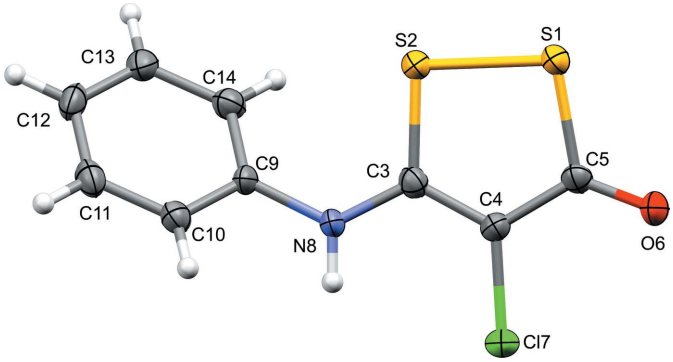



**Figure 1**
The title compound, with displacement ellipsoids drawn at the 50% probability level. H atoms are shown as spheres of arbitrary radius.

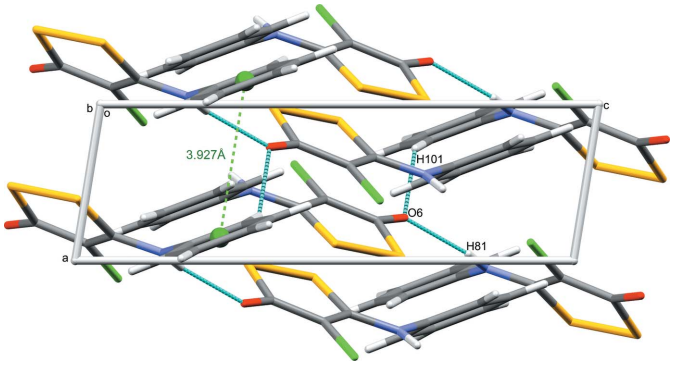



**Figure 2**
A view along the *b* axis of the crystal packing. Hydrogen-bonding interactions are shown as dashed blue lines. Centroids are shown as green dots. The centroid–centroid distance is shown as a light-green dashed line.

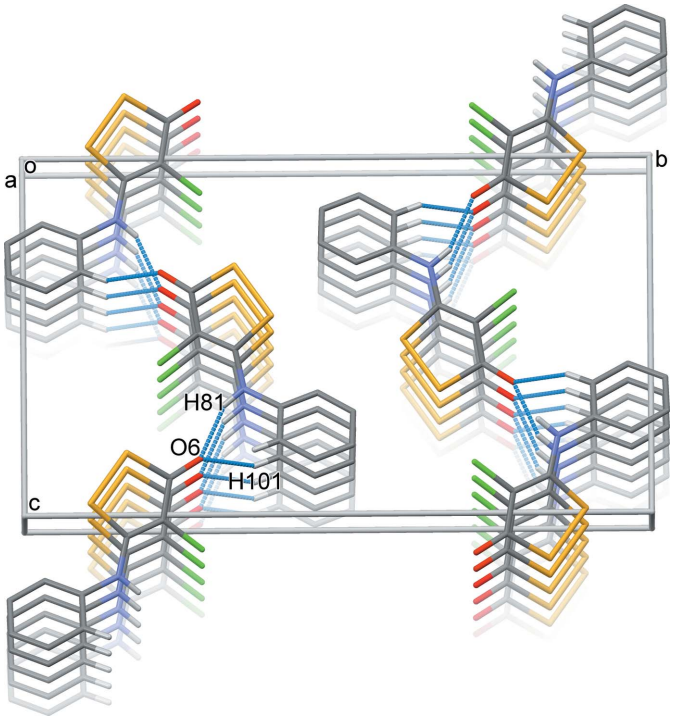



**Figure 3**
A view along the *a* axis of the crystal packing. Displacement ellipsoids drawn at the 50% probability level. Hydrogen-bonding interactions are shown as dashed blue lines.

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C10—H101···O6$^{i}$ | 0.93 | 2.50 | 3.320 (7) | 147 (1) |
| N8—H81···O6$^{ii}$ | 0.86 | 1.99 | 2.794 (7) | 155 (2) |

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

**Table 2**
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | $C_9H_6ClNOS_2$ |
| $M_r$ | 243.74 |
| Crystal system, space group | Monoclinic, $P2_1/n$ |
| Temperature (K) | 150 |
| $a, b, c$ (Å) | 3.9268 (6), 20.752 (3), 12.3431 (19) |
| $\beta$ (°) | 99.182 (14) |
| $V$ (Å$^3$) | 992.9 (3) |
| $Z$ | 4 |
| Radiation type | Mo $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 0.77 |
| Crystal size (mm) | 0.38 × 0.14 × 0.09 |
| Data collection | |
| Diffractometer | Rigaku Xcalibur Atlas Gemini ultra |
| Absorption correction | Analytical [*CrysAlis PRO* (Rigaku OD, 2015), based on expressions derived by Clark & Reid (1995)] |
| $T_{min}$, $T_{max}$ | 0.915, 0.968 |
| No. of measured, independent and observed [$I > 2.0\sigma(I)$] reflections | 2403, 2403, 1929 |
| $R_{int}$ | 0.040 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.696 |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.066, 0.155, 1.01 |
| No. of reflections | 2393 |
| No. of parameters | 131 |
| No. of restraints | 3 |
| H-atom treatment | H atoms treated by a mixture of independent and constrained refinement |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.79, −1.02 |

Computer programs: *CrysAlis PRO* (Rigaku OD, 2015), *SIR97* (Altomare *et al.*, 1999), *CRYSTALS* (Betteridge *et al.*, 2003), *CAMERON* (Watkin *et al.*, 1996). Weighting scheme: Chebychev polynomial (Watkin, 1994; Prince, 1982).

## Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

## Acknowledgements

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# full crystallographic data

*IUCrData* (2016). **1**, x161736 [https://doi.org/10.1107/S2414314616017363]

## 5-Anilino-4-chloro-3*H*-1,2-dithiol-3-one

**Khaled Boukebbous, El Adoui Laifa, Aimery De Mallmann and Mostafa Taoufik**

**(I)**

*Crystal data*

$C_9H_6ClNOS_2$
$M_r$ = 243.74
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a$ = 3.9268 (6) Å
$b$ = 20.752 (3) Å
$c$ = 12.3431 (19) Å
$\beta$ = 99.182 (14)°
$V$ = 992.9 (3) Å$^3$
$Z$ = 4
$F(000)$ = 496
$D_x$ = 1.630 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 2296 reflections
$\theta$ = 3.6–29.1°
$\mu$ = 0.77 mm$^{-1}$
$T$ = 150 K
Needle, light yellow
0.38 × 0.14 × 0.09 mm

*Data collection*

Rigaku Xcalibur Atlas Gemini ultra diffractometer
Radiation source: fine-focus sealed X-ray tube, Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 10.4685 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: analytical [CrysAlis PRO (Rigaku OD, 2015), based on expressions derived by Clark & Reid (1995)]
$T_{min}$ = 0.915, $T_{max}$ = 0.968
2403 measured reflections
2403 independent reflections
1929 reflections with $I > 2.0\sigma(I)$
$R_{int}$ = 0.040
$\theta_{max}$ = 29.6°, $\theta_{min}$ = 3.4°
$h$ = −5→5
$k$ = 0→28
$l$ = 0→15

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.066
$wR(F^2)$ = 0.155
$S$ = 1.01
2393 reflections
131 parameters
3 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: difference Fourier map
H atoms treated by a mixture of independent and constrained refinement
Method, part 1, Chebychev polynomial, (Watkin, 1994; *P*rince, 1982) [*w*eight] = 1.0/[$A_0$*$T_0$(x) + $A_1$*$T_1$(x) ··· + $A_{n-1}$]*$T_{n-1}$(x)] where $A_i$ are the Chebychev coefficients listed below and x = $F$ /$F$max Method = Robust Weighting (*P*rince, 1982) W = [*w*eight] * [1-(delta$F$/6*sigma$F$)$^2$]$^2$ $A_i$ are: 0.173E + 04 0.271E + 04 0.145E + 04 406.
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.79 e Å$^{-3}$
$\Delta\rho_{min}$ = −1.02 e Å$^{-3}$

*Special details*

**Experimental**. The crystal was placed in the cold stream of an Oxford Cryosystems open-flow nitrogen cryostat (Cosier & Glazer, 1986) with a nominal stability of 0.1 K.
Absorption correction: CrysAlisPro 1.171.38.43 (Rigaku OD, 2015) Analytical numeric absorption correction using a multifaceted crystal model based on expressions derived by Clark and Reid (1995). Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

**Refinement**. The H atoms were initially refined with soft restraints on the bond lengths and angles to regularize their geometry (C—H in the range 0.93–0.98 and N—H in the range 0.86–0.89?Å) and Uiso(H) in the range 1.2–1.5 times Ueq of the parent atom, after which the positions were refined with riding constraints (Cooper *et al.*, 2010). The hydrogen atom bonded to N was refined with a restraint on the bond length [N8—H81 = 0.82?(2)?Å]. The bond angles C3—N8—H81 and C9—N8—H81 were restrained to be equal with an e.s.d. of 2.0) and the isotropic displacement parameter of H81 was restrained to 1.2Ueq of N8 with an e.s.d of 0.002.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| S1 | 0.9772 (3) | 0.66331 (5) | 0.65293 (9) | 0.0245 |
| S2 | 0.8932 (3) | 0.60518 (5) | 0.51592 (9) | 0.0236 |
| C3 | 0.6605 (12) | 0.6630 (2) | 0.4331 (4) | 0.0220 |
| C4 | 0.6087 (12) | 0.7208 (2) | 0.4813 (4) | 0.0212 |
| C5 | 0.7448 (12) | 0.7319 (2) | 0.5925 (4) | 0.0216 |
| O6 | 0.7259 (10) | 0.78060 (16) | 0.6465 (3) | 0.0299 |
| Cl7 | 0.3850 (3) | 0.78231 (5) | 0.40834 (9) | 0.0294 |
| N8 | 0.5500 (11) | 0.64918 (18) | 0.3272 (3) | 0.0232 |
| C9 | 0.6041 (13) | 0.5905 (2) | 0.2735 (3) | 0.0218 |
| C10 | 0.7287 (13) | 0.5929 (2) | 0.1738 (4) | 0.0257 |
| C11 | 0.7676 (13) | 0.5366 (3) | 0.1182 (4) | 0.0288 |
| C12 | 0.6940 (14) | 0.4775 (2) | 0.1607 (4) | 0.0290 |
| C13 | 0.5678 (13) | 0.4752 (2) | 0.2596 (4) | 0.0274 |
| C14 | 0.5209 (13) | 0.5316 (2) | 0.3163 (4) | 0.0266 |
| H101 | 0.7838 | 0.6327 | 0.1459 | 0.0308* |
| H111 | 0.8502 | 0.5382 | 0.0512 | 0.0351* |
| H121 | 0.7281 | 0.4394 | 0.1235 | 0.0353* |
| H131 | 0.5129 | 0.4352 | 0.2879 | 0.0330* |
| H141 | 0.4324 | 0.5298 | 0.3825 | 0.0320* |
| H81 | 0.458 (14) | 0.6801 (12) | 0.2864 (18) | 0.0279* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S1 | 0.0318 (6) | 0.0215 (5) | 0.0180 (5) | 0.0009 (5) | −0.0023 (4) | −0.0008 (4) |
| S2 | 0.0313 (6) | 0.0198 (5) | 0.0184 (5) | 0.0034 (4) | −0.0001 (4) | −0.0008 (4) |
| C3 | 0.025 (2) | 0.023 (2) | 0.0180 (19) | −0.0024 (18) | 0.0046 (16) | 0.0000 (16) |
| C4 | 0.027 (2) | 0.0169 (19) | 0.020 (2) | 0.0009 (17) | 0.0025 (17) | 0.0003 (15) |
| C5 | 0.027 (2) | 0.0182 (18) | 0.0195 (19) | −0.0046 (18) | 0.0039 (16) | 0.0024 (15) |
| O6 | 0.043 (2) | 0.0218 (16) | 0.0222 (16) | 0.0002 (15) | −0.0040 (15) | −0.0048 (12) |
| Cl7 | 0.0421 (7) | 0.0216 (5) | 0.0231 (5) | 0.0071 (5) | 0.0003 (5) | 0.0029 (4) |
| N8 | 0.036 (2) | 0.0176 (16) | 0.0144 (16) | 0.0023 (16) | −0.0012 (15) | −0.0022 (13) |
| C9 | 0.031 (2) | 0.0161 (18) | 0.0167 (19) | −0.0006 (18) | −0.0023 (16) | −0.0028 (15) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C10 | 0.031 (2) | 0.025 (2) | 0.0195 (19) | −0.0011 (19) | −0.0005 (17) | −0.0004 (17) |
| C11 | 0.030 (2) | 0.036 (3) | 0.020 (2) | 0.003 (2) | 0.0036 (17) | −0.0052 (19) |
| C12 | 0.033 (2) | 0.023 (2) | 0.029 (2) | 0.003 (2) | −0.0006 (19) | −0.0103 (18) |
| C13 | 0.033 (2) | 0.023 (2) | 0.025 (2) | 0.004 (2) | −0.0014 (18) | −0.0012 (18) |
| C14 | 0.032 (2) | 0.024 (2) | 0.022 (2) | −0.003 (2) | 0.0008 (18) | 0.0028 (17) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| S1—S2 | 2.0605 (15) | C9—C14 | 1.391 (6) |
| S1—C5 | 1.789 (5) | C10—C11 | 1.376 (7) |
| S2—C3 | 1.738 (5) | C10—H101 | 0.932 |
| C3—C4 | 1.367 (6) | C11—C12 | 1.382 (7) |
| C3—N8 | 1.342 (5) | C11—H111 | 0.936 |
| C4—C5 | 1.410 (6) | C12—C13 | 1.390 (7) |
| C4—Cl7 | 1.721 (4) | C12—H121 | 0.934 |
| C5—O6 | 1.220 (5) | C13—C14 | 1.391 (6) |
| N8—C9 | 1.417 (5) | C13—H131 | 0.940 |
| N8—H81 | 0.859 (19) | C14—H141 | 0.939 |
| C9—C10 | 1.396 (6) | | |
| | | | |
| S2—S1—C5 | 96.84 (15) | C10—C9—C14 | 120.1 (4) |
| S1—S2—C3 | 94.51 (16) | C9—C10—C11 | 119.5 (5) |
| S2—C3—C4 | 116.8 (3) | C9—C10—H101 | 119.4 |
| S2—C3—N8 | 118.9 (3) | C11—C10—H101 | 121.2 |
| C4—C3—N8 | 124.3 (4) | C10—C11—C12 | 121.2 (5) |
| C3—C4—C5 | 120.5 (4) | C10—C11—H111 | 119.4 |
| C3—C4—Cl7 | 121.5 (3) | C12—C11—H111 | 119.4 |
| C5—C4—Cl7 | 118.0 (3) | C11—C12—C13 | 119.3 (4) |
| S1—C5—C4 | 111.4 (3) | C11—C12—H121 | 120.6 |
| S1—C5—O6 | 120.2 (3) | C13—C12—H121 | 120.2 |
| C4—C5—O6 | 128.4 (4) | C12—C13—C14 | 120.5 (5) |
| C3—N8—C9 | 126.2 (4) | C12—C13—H131 | 119.4 |
| C3—N8—H81 | 116.9 (14) | C14—C13—H131 | 120.1 |
| C9—N8—H81 | 116.6 (14) | C9—C14—C13 | 119.4 (4) |
| N8—C9—C10 | 118.8 (4) | C9—C14—H141 | 120.5 |
| N8—C9—C14 | 121.0 (4) | C13—C14—H141 | 120.1 |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C10—H101···O6$^{i}$ | 0.93 | 2.50 | 3.320 (7) | 147 (1) |
| N8—H81···O6$^{ii}$ | 0.86 | 1.99 | 2.794 (7) | 155 (2) |

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