IUCrData

ISSN 2414-3146

4,5-Dichloro-3*H*-1,2-dithiol-3-one

Khaled Boukebbous,[a]* El Adoui Laifa,[a] Aimery De Mallmann[b] and Mostafa Taoufik[b]

[a]Department of Chemistry, University of Constantine, BP 325, Route de Ain El Bey, Constantine 25017, Algeria, and [b]C2P2 (CNRS-UMR 5265), COMS group, Lyon 1 University, ESCPE Lyon, 43 Boulevard du 11 Novembre 1918, Villeurbanne 69626, France. *Correspondence e-mail: boukebbous.khaled@gmail.com

Received 25 October 2016
Accepted 13 November 2016

Edited by S. Bernès, Benemérita Universidad Autónoma de Puebla, México

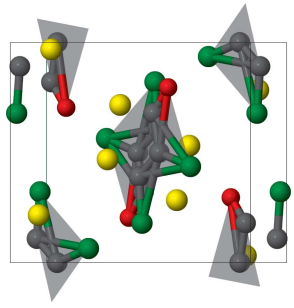
Keywords: crystal structure; 1,2-dithiol-3-one; sulfur organic compounds; heterocyclic compounds.

CCDC reference: 1517056

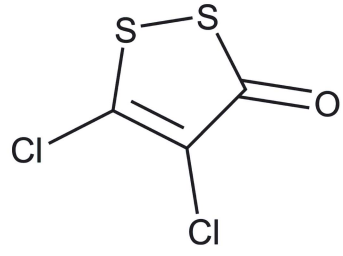
Structural data: full structural data are available from iucrdata.iucr.org

The crystallization from an ethyl acetate solution of the title compound, also known as dichloro-1,2-dithiacyclopentenone, $C_3Cl_2OS_2$, leads to a monoclinic system with $P2_1/n$ space group. The molecule displays an almost planar geometry, with a torsion angle of −2.1 (1)° for the endocyclic C—C—S—S fragment. The S—S and C═O bond lengths are 2.0521 (8) and 1.212 (3) Å, respectively. The endocyclic S—C—C angle sustained by the carbonyl group, 110.94 (16)°, deviates from the expected value of 120° for an sp^2-hybridized C atom. In the crystal, short S···S, S···Cl, S···O and Cl···Cl contacts are observed.

3D view



Chemical scheme



Structure description

The title compound belongs to a family of bioactive compounds (He *et al.*, 2004). Its crystallization from an ethyl acetate solution leads to a monoclinic system with $P2_1/n$ space group (Figs. 1 and 2). The molecule is almost planar, with torsion angles of −0.3 (3), 0.6 (3), −2.1 (1), −0.8 (2) and −0.6 (3)° for the Cl6—C5═ C4—Cl7, Cl7—C4—C3═O8, C4—C3—S2—S1, C4═C5—S1—S2 and S1—C5═C4—C3 fragments, respectively. Bond lengths of 2.0521 (8), 1.728 (2), 1.785 (2), 1.696 (2), 1.707 (2), and 1.212 (3) Å, respectively, for the S1—S2, C5—S1, C3—S2, C5—Cl6, C4—Cl7 and C3═O8 bonds are observed; furthermore, values of 93.88 (8) and 97.60 (8)° for the C5—S1—S2 and S1—S2—C3 angles, respectively, are noted. The endocyclic angle at C3, 110.94 (16)°, deviates from the value of 120° expected for an sp^2-hybridized carbon atom (C3═O8 bond); similarly, minor deviations (less than 2°) are observed for the angles S1—C5═C4 and C3—C4═C5 from the expected value of 120° for these sp^2 C atoms (C4═C5 bond).

In the crystal, short S···S [3.5929 (11) Å], S···Cl [3.4162 and 3.5219 (11) Å], S···O [3.153 (2) and 3.140 (2) Å] and Cl···Cl [3.3390 (11) Å] contacts are observed (Fig. 3).

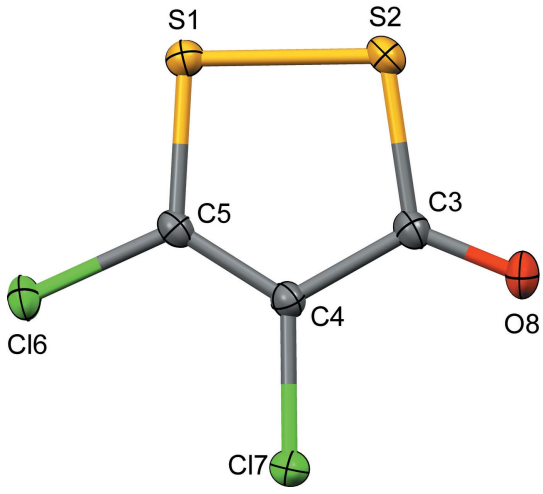


Figure 1
The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level.

Synthesis and crystallization

A commercial sample (Matrix Scientific) of 4,5-dichloro-3*H*-1,2-dithiol-3-one was used. The crystallization was performed using an ethyl acetate solution with a slow evaporation process, affording suitable single crystals.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1.

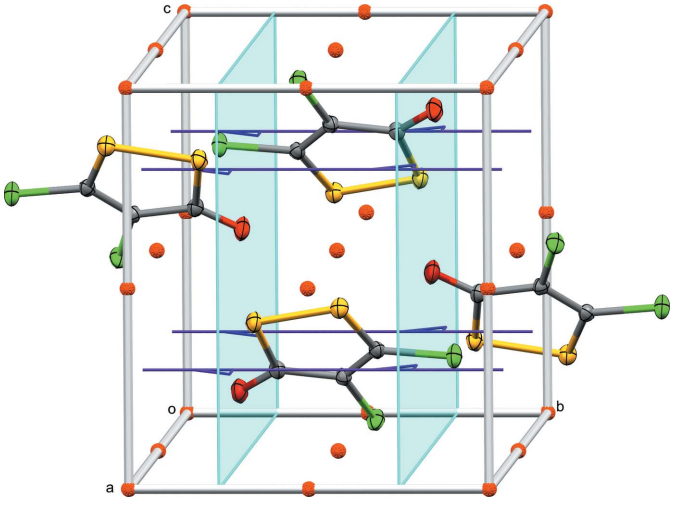


Figure 2
Crystal packing view of the title compound with displacement ellipsoids drawn at the 50% probability level. The inversion centre at [0,0,0] with symmetry operation $(-x, -y, -z)$ is shown as orange dots. The twofold screw axis parallel to [010] at $[\frac{1}{4}, y, \frac{1}{4}]$ with symmetry operation $(\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z)$ is shown as purple lines. The glide plane perpendicular to [010] with glide component $(\frac{1}{2}, 0, \frac{1}{2})$ and symmetry operation $(\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z)$ is shown as light-blue planes.

Table 1
Experimental details.

Crystal data	
Chemical formula	$C_3Cl_2OS_2$
M_r	187.07
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	150
a, b, c (Å)	8.678 (2), 7.9332 (14), 8.7340 (18)
β (°)	98.62 (2)
V (Å^3)	594.5 (2)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	1.67
Crystal size (mm)	0.51 × 0.19 × 0.15
Data collection	
Diffractometer	Agilent Xcalibur, Atlas, Gemini ultra
Absorption correction	Analytical (*CrysAlis PRO*; Rigaku OD, 2015)
T_{min}, T_{max}	0.603, 0.822
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	2973, 1377, 1228
R_{int}	0.036
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.675
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.032, 0.094, 0.99
No. of reflections	1373
No. of parameters	74
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.45, −0.41

Computer programs: *CrysAlis PRO* (Rigaku OD, 2015), *SIR97* (Altomare *et al.*, 1999), *CRYSTALS* (Betteridge *et al.*, 2003) and *Mercury* (Macrae *et al.*, 2008).

Acknowledgements

We are grateful to The Centre National de la Recherche Scientifique (CNRS, France) for the financial support.

References

Altomare, A., Burla, M. C., Camalli, M., Cascarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.

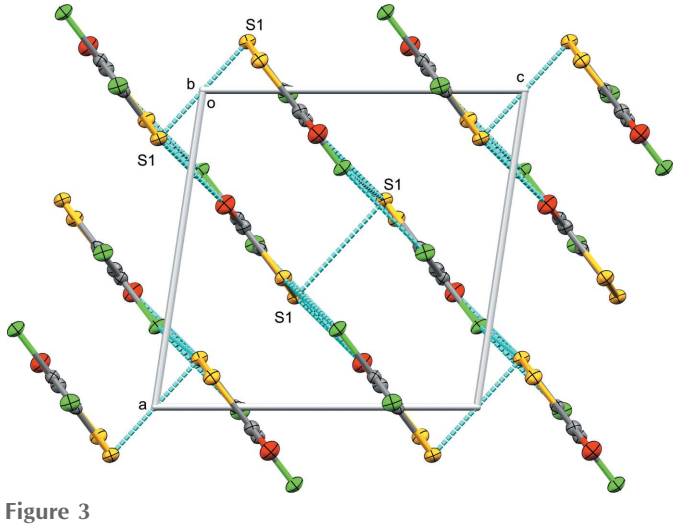


Figure 3
A view along the *b* axis showing the molecular packing. Displacement ellipsoids are drawn at the 50% probability level. Short interactions are shown as dashed blue lines.

Betteridge, P. W., Carruthers, J. R., Cooper, R. I., Prout, K. & Watkin, D. J. (2003). *J. Appl. Cryst.* **36**, 1487.
He, X., Reeve, A. M. E., Desai, U. R., Kellogg, G. E. & Reynolds, K. A. (2004). *Antimicrob. Agents Chemother.* **48**, 3093–3102.
Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
Rigaku OD (2015). *CrysAlis PRO*. Rigaku Americas Corporation, The Woodlands, Texas, USA.

full crystallographic data

IUCrData (2016). **1**, x161820 [https://doi.org/10.1107/S2414314616018204]

4,5-Dichloro-3*H*-1,2-dithiol-3-one

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

4,5-Dichloro-3*H*-1,2-dithiol-3-one

Crystal data

$C_3Cl_2OS_2$
M_r = 187.07
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 8.678 (2) Å
b = 7.9332 (14) Å
c = 8.7340 (18) Å
β = 98.62 (2)°
V = 594.5 (2) Å^3
Z = 4
$F(000)$ = 368
D_x = 2.090 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1441 reflections
θ = 3.5–28.4°
μ = 1.67 mm^{-1}
T = 150 K
Plate, light yellow
0.51 × 0.19 × 0.15 mm

Data collection

Agilent 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}
ω scans
Absorption correction: analytical (CrysAlis PRO; Rigaku OD, 2015)
T_{min} = 0.603, T_{max} = 0.822
2973 measured reflections
1377 independent reflections
1228 reflections with $I > 2.0\sigma(I)$
R_{int} = 0.036
θ_{max} = 28.7°, θ_{min} = 3.5°
h = −6→11
k = −10→10
l = −11→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.094
S = 0.99
1373 reflections
74 parameters
0 restraints
0 constraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
[*w*eight] = 1/[A_0*T_0(x) + A_1*T_1(x) ··· + A_{n-1}]*T_{n-1}(x)]
where A_i are Chebychev coefficients and x = *F*/*F*max; *w* = [*w*eight]*[1-(delta*F*/6*sigma*F*)^2]^2 A_i coefficient are: 0.248E + 04 0.417E + 04 0.267E + 04 0.120E + 04 299.
$(\Delta/\sigma)_{max}$ = 0.0004321
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}
Extinction correction: Larson, Equation 22
Extinction coefficient: 17 (4)

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems open-flow nitrogen cryostat with a nominal stability of 0.1 K.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.65286 (6)	0.53082 (7)	0.38502 (6)	0.0173
S2	0.59380 (7)	0.28367 (7)	0.33822 (7)	0.0182
C3	0.4325 (2)	0.3208 (3)	0.1897 (2)	0.0161
C4	0.4052 (2)	0.4996 (3)	0.1666 (2)	0.0145
C5	0.5024 (2)	0.6089 (3)	0.2519 (2)	0.0145
Cl6	0.48670 (7)	0.82139 (7)	0.23385 (7)	0.0207
Cl7	0.25439 (6)	0.56383 (7)	0.03087 (6)	0.0195
O8	0.3614 (2)	0.2041 (2)	0.1230 (2)	0.0232

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0155 (3)	0.0155 (3)	0.0199 (3)	0.00018 (18)	−0.0002 (2)	−0.00018 (19)
S2	0.0196 (3)	0.0133 (3)	0.0210 (3)	0.00319 (19)	0.0008 (2)	0.00174 (19)
C3	0.0178 (10)	0.0132 (10)	0.0175 (10)	0.0016 (8)	0.0032 (8)	−0.0001 (8)
C4	0.0151 (9)	0.0133 (9)	0.0149 (10)	0.0020 (8)	0.0018 (8)	−0.0007 (8)
C5	0.0152 (9)	0.0131 (10)	0.0156 (10)	0.0022 (7)	0.0039 (8)	0.0008 (8)
Cl6	0.0220 (3)	0.0111 (3)	0.0280 (3)	−0.00152 (18)	0.0007 (2)	−0.00077 (19)
Cl7	0.0199 (3)	0.0157 (3)	0.0207 (3)	0.00089 (19)	−0.0038 (2)	0.00105 (19)
O8	0.0283 (9)	0.0126 (8)	0.0270 (9)	−0.0034 (6)	−0.0018 (7)	−0.0025 (6)

Geometric parameters (Å, º)

S1—S2	2.0521 (8)	C3—O8	1.212 (3)
S1—C5	1.728 (2)	C4—C5	1.353 (3)
S2—C3	1.785 (2)	C4—Cl7	1.707 (2)
C3—C4	1.447 (3)	C5—Cl6	1.696 (2)
S2—S1—C5	93.88 (8)	C3—C4—Cl7	118.83 (17)
S1—S2—C3	97.60 (8)	C5—C4—Cl7	122.75 (18)
S2—C3—C4	110.94 (16)	S1—C5—C4	119.11 (18)
S2—C3—O8	120.71 (17)	S1—C5—Cl6	117.45 (13)
C4—C3—O8	128.3 (2)	C4—C5—Cl6	123.43 (17)
C3—C4—C5	118.4 (2)		