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5-Methoxy-1,3,4-thiadiazol-2(3*H*)-one

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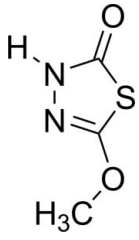
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(N–C) = 0.008 Å; R factor = 0.056; wR factor = 0.133; data-to-parameter ratio = 14.0.

The three molecules in the asymmetric unit of the title compound, $C_3H_4N_2O_2S$, are connected *via* N—H···O hydrogen bonds, forming layers normal to [001]. The rings of the molecules are approximately planar, with r.m.s. deviations of 0.0051 (1), 0.0044 (1) and 0.0111 (1) Å.

Related literature

For background to the applications of the title compound, see: Collier (2004). For the synthesis, see: Zhu *et al.* (2011). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_3H_4N_2O_2S$
M_r = 132.16
Hexagonal, $P6_1$
a = 11.9240 (17) Å
c = 20.111 (4) Å
V = 2476.3 (7) Å^3
Z = 18
Mo $K\alpha$ radiation
μ = 0.49 mm^{-1}
T = 293 K
0.30 × 0.20 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.867, T_{max} = 0.909
3492 measured reflections
3040 independent reflections
2206 reflections with $I > 2\sigma(I)$
R_{int} = 0.039
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.133
S = 1.04
3040 reflections
217 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}
Absolute structure: Flack (1983), 1468 Friedel pairs
Flack parameter: −0.05 (14)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O3^{i}	0.86	1.97	2.796 (7)	160
N3—H3D···O5	0.86	1.96	2.788 (6)	161
N5—H5A···O1^{ii}	0.86	1.99	2.813 (8)	161

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1985); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BQ2331).

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Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Zhu, H. J., Xi, B. B., Feng, M. L., Wang, K., Li, Y. F., Shi, L. & Chen, C. (2011). CN Patent No. 102212076A.

supplementary materials

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5-Methoxy-1,3,4-thiadiazol-2(3*H*)-one

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Comment

The tittle compound, 2-methoxythiazol-5(4*H*)-one is an important intermediate, which can be utilized to synthesize herbicide fluthiacet-ethyl (Collier, 2004). We report here the crystal structure of the title compound, (I).

The molecular structure of (I) is shown in Fig. 1. In the crystal structure, the asymmetric unit contains three molecules of 2-methoxythiazol-5(4*H*)-one and these molecules were connected together via N-H···O intermolecular hydrogen bonds forming stacking layers along c-axis (Fig. 2.). In the crystal structure, the rings are planar, with r.m.s. deviation of 0.06 (1) Å. The bond lengths and angles are within normal ranges (Allen *et al.*, 1987).

Experimental

The title compound, (I) was prepared by a method reported in literature (Zhu *et al.*, 2011). The crystals were obtained by dissolving (I) (0.2 g) in methanol (50 ml) and evaporating the solvent slowly at room temperature for about 10 d.

Refinement

All H atoms were positioned geometrically and constrained to ride on their parent atoms, with 0.86 Å for N—H, 0.96 Å for methyl H, respectively. The U_{iso}(H) = xU_{eq}(C), where x = 1.2 for N—H, and x = 1.5 for methyl H.

Figures

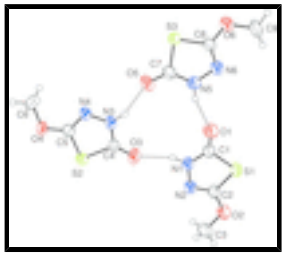


Fig. 1. The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Intermolecular H-bonds are shown with dashed lines.

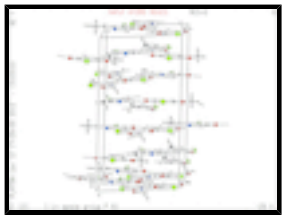


Fig. 2. A packing diagram of (I) showing the stacked layers along c-axis.

5-Methoxy-1,3,4-thiadiazol-2(3*H*)-one

Crystal data

$C_3H_4N_2O_2S$	$D_x = 1.595$ Mg m^{-3}
$M_r = 132.16$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hexagonal, $P6_1$	Cell parameters from 25 reflections
Hall symbol: P 61	$\theta = 9$–$13°$
$a = 11.9240$ (17) Å	$\mu = 0.49$ mm^{-1}
$c = 20.111$ (4) Å	$T = 293$ K
$V = 2476.3$ (7) Å^3	Block, colourless
$Z = 18$	0.30 × 0.20 × 0.20 mm
$F(000) = 1224$	

Data collection

Enraf–Nonius CAD-4 diffractometer	2206 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.039$
graphite	$\theta_{max} = 25.4°$, $\theta_{min} = 2.0°$
$\omega/2\theta$ scans	$h = 0 \rightarrow 12$
Absorption correction: ψ scan (North *et al.*, 1968)	$k = 0 \rightarrow 12$
$T_{min} = 0.867$, $T_{max} = 0.909$	$l = -24 \rightarrow 24$
3492 measured reflections	3 standard reflections every 200 reflections
3040 independent reflections	intensity decay: 1%

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.056$	$w = 1/[\sigma^2(F_o^2) + (0.0696P)^2 + 0.1426P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.133$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.04$	$\Delta\rho_{max} = 0.27$ e Å^{-3}
3040 reflections	$\Delta\rho_{min} = -0.38$ e Å^{-3}
217 parameters	Extinction correction: *SHELXL97* (Sheldrick, 2008)
1 restraint	Extinction coefficient: 0.0009 (2)
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), **1468 Friedel pairs**
Secondary atom site location: difference Fourier map	Flack parameter: −0.05 (14)

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.82079 (12)	0.67453 (12)	0.52166 (9)	0.0559 (4)
N1	0.8459 (4)	0.4789 (4)	0.5285 (3)	0.0500 (12)
H1A	0.8783	0.4288	0.5317	0.060*
C1	0.9238 (5)	0.6063 (5)	0.5255 (3)	0.0473 (14)
O1	1.0407 (3)	0.6672 (4)	0.5228 (3)	0.0709 (14)
N2	0.7133 (4)	0.4265 (4)	0.5265 (3)	0.0487 (12)
O2	0.5724 (3)	0.5049 (3)	0.5201 (2)	0.0638 (12)
C2	0.6895 (5)	0.5184 (5)	0.5232 (3)	0.0440 (13)
C3	0.4662 (5)	0.3737 (6)	0.5148 (4)	0.071 (2)
H3A	0.3860	0.3742	0.5130	0.107*
H3B	0.4659	0.3248	0.5528	0.107*
H3C	0.4759	0.3348	0.4751	0.107*
S2	−0.10862 (12)	0.04825 (12)	0.51466 (7)	0.0452 (4)
O3	−0.1023 (4)	0.2749 (4)	0.5220 (3)	0.0646 (12)
O4	0.0620 (3)	−0.0301 (3)	0.5161 (2)	0.0564 (11)
N3	0.0884 (4)	0.2708 (4)	0.5190 (3)	0.0497 (12)
H3D	0.1382	0.3535	0.5191	0.060*
N4	0.1407 (4)	0.1911 (4)	0.5184 (3)	0.0443 (11)
C4	−0.0410 (5)	0.2188 (5)	0.5194 (3)	0.0431 (13)
C5	0.0477 (4)	0.0735 (5)	0.5165 (3)	0.0429 (12)
C6	0.1950 (5)	−0.0021 (6)	0.5226 (4)	0.0588 (16)
H6A	0.1968	−0.0817	0.5216	0.088*
H6B	0.2302	0.0416	0.5640	0.088*
H6C	0.2456	0.0521	0.4864	0.088*
S3	0.52008 (12)	0.74536 (12)	0.53340 (7)	0.0513 (4)
O5	0.2932 (4)	0.5245 (3)	0.5299 (3)	0.0760 (14)
O6	0.5965 (3)	0.9929 (3)	0.5329 (2)	0.0622 (12)
N5	0.2976 (4)	0.7178 (4)	0.5261 (3)	0.0519 (12)
H5A	0.2149	0.6839	0.5233	0.062*
N6	0.3771 (4)	0.8501 (4)	0.5268 (3)	0.0500 (11)
C7	0.3507 (5)	0.6438 (5)	0.5296 (3)	0.0486 (14)
C8	0.4939 (4)	0.8748 (5)	0.5309 (3)	0.0447 (13)
C9	0.5707 (6)	1.0982 (5)	0.5339 (5)	0.074 (2)

H9A	0.6511	1.1787	0.5356	0.111*
H9B	0.5198	1.0907	0.5725	0.111*
H9C	0.5240	1.0955	0.4945	0.111*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0353 (7)	0.0322 (7)	0.1008 (12)	0.0171 (6)	0.0044 (9)	0.0039 (8)
N1	0.038 (2)	0.035 (2)	0.082 (4)	0.023 (2)	−0.001 (3)	−0.003 (3)
C1	0.034 (3)	0.039 (3)	0.069 (4)	0.019 (3)	0.003 (3)	−0.004 (3)
O1	0.033 (2)	0.050 (2)	0.125 (4)	0.0174 (18)	0.004 (3)	−0.004 (3)
N2	0.035 (2)	0.035 (2)	0.076 (4)	0.0171 (19)	0.001 (2)	0.000 (3)
O2	0.0308 (18)	0.043 (2)	0.117 (4)	0.0178 (16)	0.004 (2)	−0.003 (3)
C2	0.031 (2)	0.033 (3)	0.066 (4)	0.014 (2)	0.008 (3)	0.007 (3)
C3	0.036 (3)	0.053 (4)	0.115 (7)	0.015 (3)	−0.006 (4)	−0.011 (4)
S2	0.0315 (6)	0.0334 (6)	0.0664 (9)	0.0132 (5)	−0.0002 (6)	−0.0008 (6)
O3	0.059 (3)	0.047 (2)	0.101 (3)	0.036 (2)	−0.005 (3)	−0.004 (2)
O4	0.045 (2)	0.0305 (19)	0.096 (3)	0.0208 (16)	−0.003 (2)	−0.001 (2)
N3	0.042 (2)	0.025 (2)	0.079 (4)	0.0145 (18)	−0.006 (3)	0.001 (2)
N4	0.035 (2)	0.029 (2)	0.066 (3)	0.0139 (17)	0.000 (3)	0.004 (2)
C4	0.043 (3)	0.031 (2)	0.057 (4)	0.020 (2)	−0.002 (3)	0.000 (3)
C5	0.038 (3)	0.037 (3)	0.052 (3)	0.017 (2)	0.002 (3)	0.003 (3)
C6	0.044 (3)	0.055 (3)	0.088 (5)	0.032 (3)	0.001 (4)	0.016 (4)
S3	0.0317 (7)	0.0351 (7)	0.0863 (12)	0.0160 (6)	−0.0036 (7)	−0.0017 (7)
O5	0.045 (2)	0.029 (2)	0.139 (4)	0.0071 (19)	−0.003 (3)	−0.001 (3)
O6	0.036 (2)	0.0323 (19)	0.110 (4)	0.0112 (17)	0.001 (2)	0.001 (2)
N5	0.029 (2)	0.041 (2)	0.084 (4)	0.016 (2)	0.004 (3)	−0.001 (3)
N6	0.036 (2)	0.034 (2)	0.078 (3)	0.0164 (19)	0.003 (3)	0.001 (2)
C7	0.036 (3)	0.037 (3)	0.067 (4)	0.014 (2)	0.003 (3)	−0.005 (3)
C8	0.033 (3)	0.032 (3)	0.064 (4)	0.012 (2)	0.003 (3)	−0.002 (3)
C9	0.059 (4)	0.027 (3)	0.127 (6)	0.016 (3)	0.009 (4)	0.004 (4)

Geometric parameters (Å, °)

S1—C2	1.733 (5)	N3—N4	1.373 (5)
S1—C1	1.782 (5)	N3—H3D	0.8600
N1—C1	1.328 (6)	N4—C5	1.282 (6)
N1—N2	1.380 (5)	C6—H6A	0.9600
N1—H1A	0.8600	C6—H6B	0.9600
C1—O1	1.209 (6)	C6—H6C	0.9600
N2—C2	1.264 (6)	S3—C8	1.721 (5)
O2—C2	1.324 (6)	S3—C7	1.762 (6)
O2—C3	1.443 (7)	O5—C7	1.232 (6)
C3—H3A	0.9600	O6—C8	1.326 (6)
C3—H3B	0.9600	O6—C9	1.434 (7)
C3—H3C	0.9600	N5—C7	1.321 (6)
S2—C5	1.734 (5)	N5—N6	1.375 (6)
S2—C4	1.777 (5)	N5—H5A	0.8600
O3—C4	1.214 (5)	N6—C8	1.274 (6)

O4—C5	1.328 (5)	C9—H9A	0.9600
O4—C6	1.454 (6)	C9—H9B	0.9600
N3—C4	1.345 (6)	C9—H9C	0.9600
C2—S1—C1	88.1 (2)	N4—C5—O4	125.1 (4)
C1—N1—N2	120.3 (4)	N4—C5—S2	117.2 (4)
C1—N1—H1A	119.9	O4—C5—S2	117.7 (3)
N2—N1—H1A	119.9	O4—C6—H6A	109.5
O1—C1—N1	128.8 (5)	O4—C6—H6B	109.5
O1—C1—S1	125.1 (4)	H6A—C6—H6B	109.5
N1—C1—S1	106.1 (4)	O4—C6—H6C	109.5
C2—N2—N1	108.2 (4)	H6A—C6—H6C	109.5
C2—O2—C3	115.9 (4)	H6B—C6—H6C	109.5
N2—C2—O2	125.2 (4)	C8—S3—C7	87.5 (2)
N2—C2—S1	117.3 (4)	C8—O6—C9	116.3 (4)
O2—C2—S1	117.5 (4)	C7—N5—N6	118.7 (5)
O2—C3—H3A	109.5	C7—N5—H5A	120.7
O2—C3—H3B	109.5	N6—N5—H5A	120.7
H3A—C3—H3B	109.5	C8—N6—N5	108.2 (4)
O2—C3—H3C	109.5	O5—C7—N5	126.5 (5)
H3A—C3—H3C	109.5	O5—C7—S3	125.4 (4)
H3B—C3—H3C	109.5	N5—C7—S3	108.1 (4)
C5—S2—C4	88.1 (2)	N6—C8—O6	124.6 (4)
C5—O4—C6	114.7 (4)	N6—C8—S3	117.5 (4)
C4—N3—N4	119.6 (4)	O6—C8—S3	117.8 (3)
C4—N3—H3D	120.2	O6—C9—H9A	109.5
N4—N3—H3D	120.2	O6—C9—H9B	109.5
C5—N4—N3	108.3 (4)	H9A—C9—H9B	109.5
O3—C4—N3	127.9 (5)	O6—C9—H9C	109.5
O3—C4—S2	125.4 (4)	H9A—C9—H9C	109.5
N3—C4—S2	106.7 (3)	H9B—C9—H9C	109.5
N2—N1—C1—O1	−175.3 (7)	N3—N4—C5—S2	0.5 (7)
N2—N1—C1—S1	1.7 (7)	C6—O4—C5—N4	4.0 (10)
C2—S1—C1—O1	176.1 (6)	C6—O4—C5—S2	−175.8 (5)
C2—S1—C1—N1	−1.0 (5)	C4—S2—C5—N4	−1.8 (5)
C1—N1—N2—C2	−1.5 (8)	C4—S2—C5—O4	178.0 (5)
N1—N2—C2—O2	179.6 (6)	C7—N5—N6—C8	0.2 (9)
N1—N2—C2—S1	0.5 (8)	N6—N5—C7—O5	179.6 (7)
C3—O2—C2—N2	−5.2 (10)	N6—N5—C7—S3	−0.8 (7)
C3—O2—C2—S1	174.0 (5)	C8—S3—C7—O5	−179.5 (7)
C1—S1—C2—N2	0.3 (6)	C8—S3—C7—N5	0.9 (5)
C1—S1—C2—O2	−178.9 (5)	N5—N6—C8—O6	−179.9 (6)
C4—N3—N4—C5	1.7 (8)	N5—N6—C8—S3	0.6 (8)
N4—N3—C4—O3	177.5 (7)	C9—O6—C8—N6	3.4 (11)
N4—N3—C4—S2	−2.9 (7)	C9—O6—C8—S3	−177.1 (6)
C5—S2—C4—O3	−178.0 (6)	C7—S3—C8—N6	−0.9 (6)
C5—S2—C4—N3	2.4 (5)	C7—S3—C8—O6	179.6 (6)
N3—N4—C5—O4	−179.3 (6)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···O3^{i}	0.86	1.97	2.796 (7)	160.
N3—H3D···O5	0.86	1.96	2.788 (6)	161.
N5—H5A···O1^{ii}	0.86	1.99	2.813 (8)	161.

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

Fig. 1

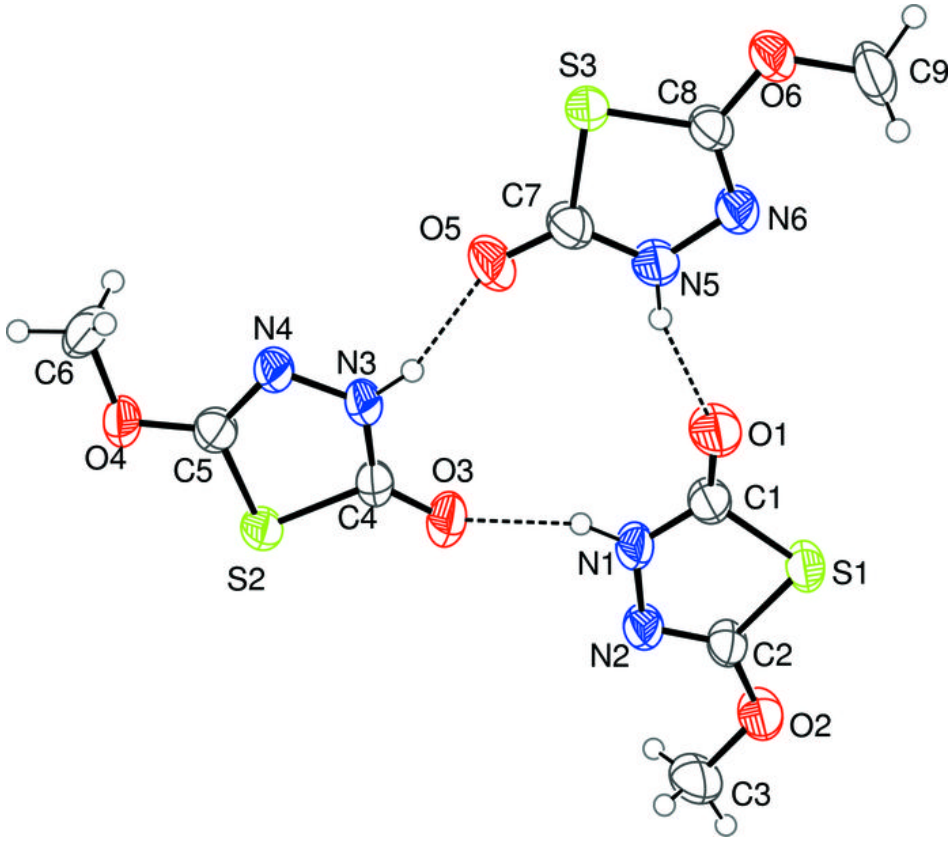
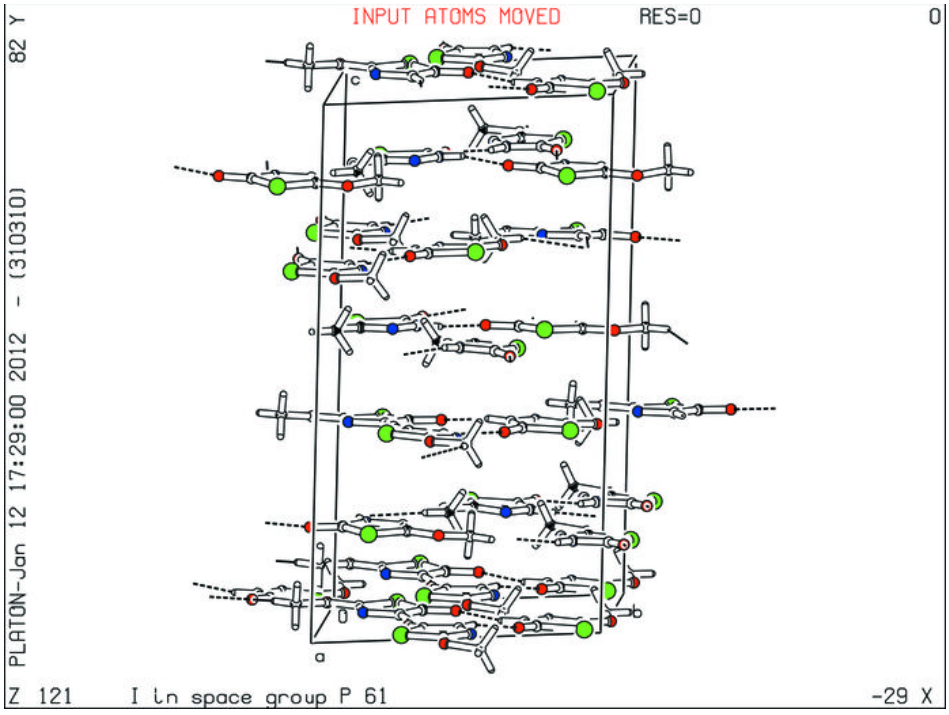


Fig. 2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Z 121
I in space group P 61
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