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N-{3-[3-(2,2-Dimethylpropionyl)-thioureido]propyl}-2,2-dimethylpropionamide

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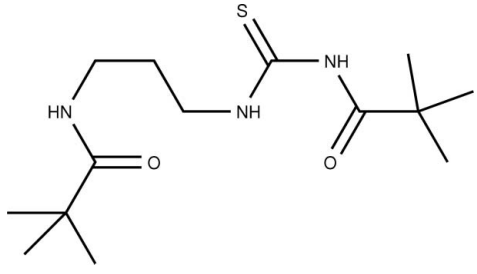
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.060; wR factor = 0.153; data-to-parameter ratio = 17.7.

The title compound, $C_{14}H_{27}N_3O_2S$, is a pivaloyl thiourea derivative and there are two intramolecular N—H···O hydrogen bonds, together with a C—H···S interaction. In the crystal structure, molecules are stabilized by intermolecular N—H···S and N—H···O hydrogen bonds, forming a two–dimensional network.

Related literature

For related crystal structures and the potential biological activity of the title compound, see: Yusof *et al.* (2006, 2007); Vig *et al.* (1998); Vankatachalam *et al.* (2001).



Experimental

Crystal data

$C_{14}H_{27}N_3O_2S$
M_r = 301.46
Monoclinic, $P2_1/c$
a = 9.205 (3) Å
b = 20.667 (7) Å
c = 10.071 (3) Å
β = 115.707 (5)°
V = 1726.3 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.19 mm^{-1}
T = 298 (2) K
0.49 × 0.24 × 0.17 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.911, T_{max} = 0.968
9243 measured reflections
3210 independent reflections
2048 reflections with $I > 2\sigma(I)$
R_{int} = 0.042

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.153
S = 1.04
3210 reflections
181 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O1	0.86	1.96	2.631 (3)	134
N2—H2A···O2	0.86	2.38	3.025 (4)	132
C7—H7B···S1	0.97	2.71	3.082 (4)	103
N1—H1A···S1^{i}	0.86	2.75	3.581 (3)	163
N3—H3A···O1^{ii}	0.86	2.48	3.051 (4)	125
N3—H3A···O2^{ii}	0.86	2.48	3.161 (4)	137

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RK2065).

References

Bruker (2000). *SADABS* (Version 2.01), *SMART* (Version 5.630) and *SAINT* (Version 6.36a). Bruker AXS Inc., Madison, Wisconsin, USA.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Sheldrick, G. M. (1997*a*). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*b*). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Vankatachalam, T. K., Sudbeck, E. A., Mao, C. & Uckun, F. M. (2001). *Bioorg. Med. Chem. Lett.* **11**, 523–528.
Vig, R., Mao, C., Vankatachalam, T. K., Tuel-Ahlgren, L., Sudbeck, E. A. & Uckun, F. M. (1998). *Bioorg. Med. Chem.* **6**, 1789–1797.
Yusof, M. S. M., Ramadzan, N. I. A. & Yamin, B. M. (2006). *Acta Cryst.* E**62**, o5513–o5514.
Yusof, M. S. M., Saadum, Z. I. M. & Yamin, B. M. (2007). *Acta Cryst.* E**63**, o3781.

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supplementary materials

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N-{3-[3-(2,2-Dimethylpropionyl)thioureido]propyl}-2,2-dimethylpropionamide

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Comment

Thiourea and dithio compounds, such as dithiocarbazates and dithiocarbamates, are potential biologically active compounds (Vig *et al.*, 1998; Vankatachalam *et al.*, 2001). The title compound, (**I**), is a simple analogue of thiourea derivatives (Fig. 1). The bond lengths and angles are in normal ranges and comparable with other thiourea derivatives (Yusof *et al.*, 2006, 2007). The central thiourea (S1/N1/N2/C6) are planar, with a maximum deviation of 0.023 (3) Å for atom N2 from the mean plane.

There are two intramolecular hydrogen bond, N2—H2A···O1 and N2—H2A···O2 (forming a pseudo-six-membered ring: O1···H2A—N2—C6—N1—C5═O1). In the crystal structure, the molecules are linked by intermolecular interaction, N1—H1a···S1^{i}, N3—H3A···O1^{ii} and N3—H3A···O2^{ii} to form two–dimensional network (Fig. 2). Symmetry codes: (i) $-x + 2, -y, -z + 1$; (ii) $x, -y + 1/2, z - 1/2$.

Experimental

To a stirring acetone solution (75 ml) of pivaloyl chloride (2.0 g, 17 mmol) and ammoniumthiocyanate (1.3 g, 17 mmol), 1,3-diaminopropane (0.67 g, 8.5 mmol) in 40 ml of acetone was added dropwise. The solution mixture was refluxed for 1 h. The resulting solution was poured into a beaker containing some ice blocks. The white precipitate was filtered off and washed with distilled water and cold ethanol before dried under vacuum. Good quality crystals were obtained by recrystallization from THF.

Refinement

After their location in the difference map, all H atoms were fixed geometrically at ideal positions and allowed to ride on the parent C or N atoms with C—H = 0.93–0.97Å and N—H = 0.86Å with U_{iso}(H) = 1.2U_{eq}(C, N)(CH_2 and NH) or 1.5U_{eq}(C)(CH_3).

Figures

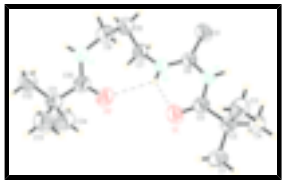

Fig. 1. The molecule of **I** showing the atom–labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are presented as a spheres of arbitrary radius. Dashed lines indicate intramolecular hydrogen bonds.

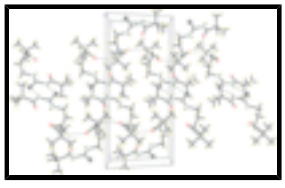

Fig. 2. Packing diagram of **I**, viewed down the a–axis. The dashed lines denote the N—H···S and N—H···O hydrogen bonds.

N-{3-[3-(2,2-Dimethylpropionyl)thioureido]propyl}-2,2-dimethylpropionamide

Crystal data

$C_{14}H_{27}N_3O_2S$	F_{000} = 656
M_r = 301.46	D_x = 1.160 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 873 reflections
a = 9.205 (3) Å	θ = 2.0–25.5º
b = 20.667 (7) Å	μ = 0.19 mm^{-1}
c = 10.071 (3) Å	T = 298 (2) K
β = 115.707 (5)º	Plate, colourless
V = 1726.3 (10) Å^3	0.49 × 0.24 × 0.17 mm
Z = 4	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	3210 independent reflections
Radiation source: fine–focus sealed tube	2048 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.042
Detector resolution: 83.66 pixels mm^{-1}	θ_{max} = 25.5º
T = 298(2) K	θ_{min} = 2.0º
ω scans	h = −11→10
Absorption correction: multi-scan (SADABS; Bruker, 2000)	k = −25→23
T_{min} = 0.911, T_{max} = 0.968	l = −9→12
9243 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.060	H-atom parameters constrained
$wR(F^2)$ = 0.153	$w = 1/[\sigma^2(F_o^2) + (0.0671P)^2 + 0.4147P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.04	$(\Delta/\sigma)_{max}$ = 0.001
3210 reflections	$\Delta\rho_{max}$ = 0.22 e Å^{-3}
181 parameters	$\Delta\rho_{min}$ = −0.14 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All s.u. (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u. are taken into account individually in the estimation of s.u. in distances, angles and torsion angles; correlations between s.u. in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u. is used for estimating s.u. 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 $F^2 > 2\sigma(F^2)$ 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	0.83718 (10)	0.02231 (4)	0.28573 (9)	0.0686 (3)
O1	0.5861 (3)	0.10775 (11)	0.5413 (2)	0.0737 (7)
O2	0.4227 (3)	0.21923 (10)	0.3053 (2)	0.0725 (7)
N1	0.7725 (3)	0.04792 (11)	0.5112 (2)	0.0514 (6)
H1A	0.8543	0.0232	0.5568	0.062*
N2	0.6253 (3)	0.10530 (11)	0.2977 (2)	0.0500 (6)
H2A	0.5827	0.1242	0.3483	0.060*
N3	0.4857 (3)	0.26693 (11)	0.1385 (3)	0.0631 (7)
H3A	0.4529	0.2911	0.0613	0.076*
C1	0.6136 (4)	0.05377 (17)	0.7934 (4)	0.0713 (9)
H1B	0.5977	0.0997	0.7931	0.107*
H1C	0.6453	0.0372	0.8911	0.107*
H1D	0.5149	0.0335	0.7269	0.107*
C2	0.7451 (3)	0.03940 (13)	0.7441 (3)	0.0476 (7)
C3	0.9013 (4)	0.07277 (15)	0.8487 (3)	0.0664 (9)
H3B	0.8838	0.1186	0.8475	0.100*
H3C	0.9842	0.0641	0.8173	0.100*
H3D	0.9341	0.0566	0.9468	0.100*
C4	0.7706 (4)	−0.03363 (13)	0.7446 (3)	0.0593 (8)
H4A	0.6718	−0.0540	0.6787	0.089*
H4B	0.8030	−0.0501	0.8425	0.089*
H4C	0.8531	−0.0427	0.7130	0.089*
C5	0.6927 (3)	0.06821 (14)	0.5911 (3)	0.0480 (7)
C6	0.7378 (3)	0.06233 (13)	0.3648 (3)	0.0465 (7)
C7	0.5671 (4)	0.12330 (14)	0.1433 (3)	0.0564 (8)
H7A	0.4543	0.1354	0.1046	0.068*
H7B	0.5750	0.0862	0.0878	0.068*
C8	0.6611 (4)	0.17867 (17)	0.1220 (4)	0.0739 (10)
H8A	0.7737	0.1664	0.1609	0.089*
H8B	0.6229	0.1865	0.0172	0.089*
C9	0.6481 (4)	0.24037 (16)	0.1951 (4)	0.0731 (10)
H9A	0.6867	0.2324	0.2999	0.088*
H9B	0.7183	0.2725	0.1831	0.088*

C10	0.3853 (4)	0.25604 (14)	0.1990 (3)	0.0521 (7)
C11	0.2226 (4)	0.29009 (14)	0.1346 (3)	0.0566 (8)
C12	0.1978 (4)	0.33658 (17)	0.0085 (4)	0.0777 (10)
H12A	0.2014	0.3129	−0.0720	0.117*
H12B	0.2815	0.3686	0.0421	0.117*
H12C	0.0949	0.3574	−0.0238	0.117*
C13	0.2149 (5)	0.32914 (18)	0.2609 (4)	0.0865 (11)
H13A	0.2305	0.3007	0.3413	0.130*
H13B	0.1115	0.3497	0.2270	0.130*
H13C	0.2980	0.3615	0.2936	0.130*
C14	0.0924 (4)	0.23851 (19)	0.0830 (4)	0.0915 (12)
H14A	0.0978	0.2143	0.0039	0.137*
H14B	−0.0114	0.2587	0.0491	0.137*
H14C	0.1086	0.2099	0.1633	0.137*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0749 (6)	0.0882 (6)	0.0544 (5)	0.0298 (5)	0.0389 (4)	0.0081 (4)
O1	0.0895 (16)	0.0901 (16)	0.0502 (13)	0.0438 (14)	0.0386 (12)	0.0160 (11)
O2	0.1040 (18)	0.0741 (15)	0.0546 (13)	0.0240 (12)	0.0487 (13)	0.0200 (11)
N1	0.0519 (14)	0.0640 (15)	0.0412 (14)	0.0189 (12)	0.0230 (11)	0.0104 (11)
N2	0.0616 (15)	0.0546 (14)	0.0389 (13)	0.0128 (12)	0.0264 (12)	0.0044 (11)
N3	0.0664 (17)	0.0600 (16)	0.0716 (18)	0.0110 (13)	0.0382 (15)	0.0209 (13)
C1	0.082 (2)	0.089 (2)	0.060 (2)	0.0127 (19)	0.0477 (18)	0.0075 (17)
C2	0.0503 (17)	0.0552 (17)	0.0405 (16)	0.0005 (13)	0.0226 (13)	0.0016 (13)
C3	0.071 (2)	0.069 (2)	0.0478 (18)	−0.0079 (17)	0.0158 (16)	−0.0026 (15)
C4	0.0657 (19)	0.0583 (19)	0.0573 (19)	−0.0033 (15)	0.0299 (16)	0.0048 (14)
C5	0.0498 (17)	0.0530 (17)	0.0423 (17)	0.0033 (14)	0.0209 (14)	−0.0010 (13)
C6	0.0482 (16)	0.0527 (17)	0.0432 (16)	0.0042 (14)	0.0243 (13)	0.0045 (13)
C7	0.072 (2)	0.0614 (19)	0.0405 (17)	0.0159 (16)	0.0287 (15)	0.0064 (13)
C8	0.081 (2)	0.085 (3)	0.070 (2)	0.0243 (19)	0.047 (2)	0.0262 (18)
C9	0.062 (2)	0.065 (2)	0.098 (3)	0.0002 (17)	0.040 (2)	0.0167 (18)
C10	0.066 (2)	0.0496 (17)	0.0461 (17)	0.0010 (14)	0.0297 (16)	−0.0016 (14)
C11	0.066 (2)	0.0601 (19)	0.0516 (18)	0.0049 (16)	0.0324 (16)	0.0032 (14)
C12	0.085 (2)	0.088 (2)	0.067 (2)	0.028 (2)	0.0401 (19)	0.0209 (18)
C13	0.114 (3)	0.087 (3)	0.077 (2)	0.027 (2)	0.059 (2)	0.0006 (19)
C14	0.078 (2)	0.108 (3)	0.086 (3)	−0.019 (2)	0.033 (2)	−0.002 (2)

Geometric parameters (Å, °)

S1—C6	1.670 (3)	C4—H4B	0.9600
O1—C5	1.207 (3)	C4—H4C	0.9600
O2—C10	1.234 (3)	C7—C8	1.505 (4)
N1—C5	1.369 (3)	C7—H7A	0.9700
N1—C6	1.398 (3)	C7—H7B	0.9700
N1—H1A	0.8600	C8—C9	1.503 (5)
N2—C6	1.308 (3)	C8—H8A	0.9700
N2—C7	1.456 (3)	C8—H8B	0.9700

N2—H2A	0.8600	C9—H9A	0.9700
N3—C10	1.329 (4)	C9—H9B	0.9700
N3—C9	1.457 (4)	C10—C11	1.522 (4)
N3—H3A	0.8600	C11—C14	1.517 (4)
C1—C2	1.524 (4)	C11—C12	1.528 (4)
C1—H1B	0.9600	C11—C13	1.535 (4)
C1—H1C	0.9600	C12—H12A	0.9600
C1—H1D	0.9600	C12—H12B	0.9600
C2—C5	1.522 (4)	C12—H12C	0.9600
C2—C4	1.527 (4)	C13—H13A	0.9600
C2—C3	1.529 (4)	C13—H13B	0.9600
C3—H3B	0.9600	C13—H13C	0.9600
C3—H3C	0.9600	C14—H14A	0.9600
C3—H3D	0.9600	C14—H14B	0.9600
C4—H4A	0.9600	C14—H14C	0.9600
C5—N1—C6	128.2 (2)	N2—C7—H7B	109.2
C5—N1—H1A	115.9	C8—C7—H7B	109.2
C6—N1—H1A	115.9	H7A—C7—H7B	107.9
C6—N2—C7	124.5 (2)	C9—C8—C7	113.7 (3)
C6—N2—H2A	117.7	C9—C8—H8A	108.8
C7—N2—H2A	117.7	C7—C8—H8A	108.8
C10—N3—C9	123.8 (3)	C9—C8—H8B	108.8
C10—N3—H3A	118.1	C7—C8—H8B	108.8
C9—N3—H3A	118.1	H8A—C8—H8B	107.7
C2—C1—H1B	109.5	N3—C9—C8	114.5 (3)
C2—C1—H1C	109.5	N3—C9—H9A	108.6
H1B—C1—H1C	109.5	C8—C9—H9A	108.6
C2—C1—H1D	109.5	N3—C9—H9B	108.6
H1B—C1—H1D	109.5	C8—C9—H9B	108.6
H1C—C1—H1D	109.5	H9A—C9—H9B	107.6
C5—C2—C1	107.8 (2)	O2—C10—N3	121.0 (3)
C5—C2—C4	111.8 (2)	O2—C10—C11	120.5 (3)
C1—C2—C4	109.5 (2)	N3—C10—C11	118.5 (3)
C5—C2—C3	108.1 (2)	C14—C11—C10	107.8 (3)
C1—C2—C3	109.3 (2)	C14—C11—C12	110.2 (3)
C4—C2—C3	110.3 (2)	C10—C11—C12	114.2 (2)
C2—C3—H3B	109.5	C14—C11—C13	109.9 (3)
C2—C3—H3C	109.5	C10—C11—C13	106.3 (3)
H3B—C3—H3C	109.5	C12—C11—C13	108.3 (3)
C2—C3—H3D	109.5	C11—C12—H12A	109.5
H3B—C3—H3D	109.5	C11—C12—H12B	109.5
H3C—C3—H3D	109.5	H12A—C12—H12B	109.5
C2—C4—H4A	109.5	C11—C12—H12C	109.5
C2—C4—H4B	109.5	H12A—C12—H12C	109.5
H4A—C4—H4B	109.5	H12B—C12—H12C	109.5
C2—C4—H4C	109.5	C11—C13—H13A	109.5
H4A—C4—H4C	109.5	C11—C13—H13B	109.5
H4B—C4—H4C	109.5	H13A—C13—H13B	109.5
O1—C5—N1	120.7 (2)	C11—C13—H13C	109.5

O1—C5—C2	121.9 (2)	H13A—C13—H13C	109.5
N1—C5—C2	117.4 (2)	H13B—C13—H13C	109.5
N2—C6—N1	117.2 (2)	C11—C14—H14A	109.5
N2—C6—S1	124.4 (2)	C11—C14—H14B	109.5
N1—C6—S1	118.4 (2)	H14A—C14—H14B	109.5
N2—C7—C8	112.2 (2)	C11—C14—H14C	109.5
N2—C7—H7A	109.2	H14A—C14—H14C	109.5
C8—C7—H7A	109.2	H14B—C14—H14C	109.5
C6—N1—C5—O1	−7.0 (5)	C6—N2—C7—C8	−90.0 (3)
C6—N1—C5—C2	173.5 (2)	N2—C7—C8—C9	−62.7 (3)
C1—C2—C5—O1	16.0 (4)	C10—N3—C9—C8	95.3 (4)
C4—C2—C5—O1	136.4 (3)	C7—C8—C9—N3	−62.9 (4)
C3—C2—C5—O1	−102.1 (3)	C9—N3—C10—O2	−3.7 (5)
C1—C2—C5—N1	−164.5 (3)	C9—N3—C10—C11	176.3 (3)
C4—C2—C5—N1	−44.1 (3)	O2—C10—C11—C14	−60.0 (4)
C3—C2—C5—N1	77.4 (3)	N3—C10—C11—C14	120.1 (3)
C7—N2—C6—N1	−176.2 (2)	O2—C10—C11—C12	177.1 (3)
C7—N2—C6—S1	1.7 (4)	N3—C10—C11—C12	−2.8 (4)
C5—N1—C6—N2	7.2 (4)	O2—C10—C11—C13	57.7 (4)
C5—N1—C6—S1	−170.8 (2)	N3—C10—C11—C13	−122.2 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2A···O1	0.86	1.96	2.631 (3)	134
N2—H2A···O2	0.86	2.38	3.025 (4)	132
C7—H7B···S1	0.97	2.71	3.082 (4)	103
N1—H1A···S1^{i}	0.86	2.75	3.581 (3)	163
N3—H3A···O1^{ii}	0.86	2.48	3.051 (4)	125
N3—H3A···O2^{ii}	0.86	2.48	3.161 (4)	137

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

Fig. 1

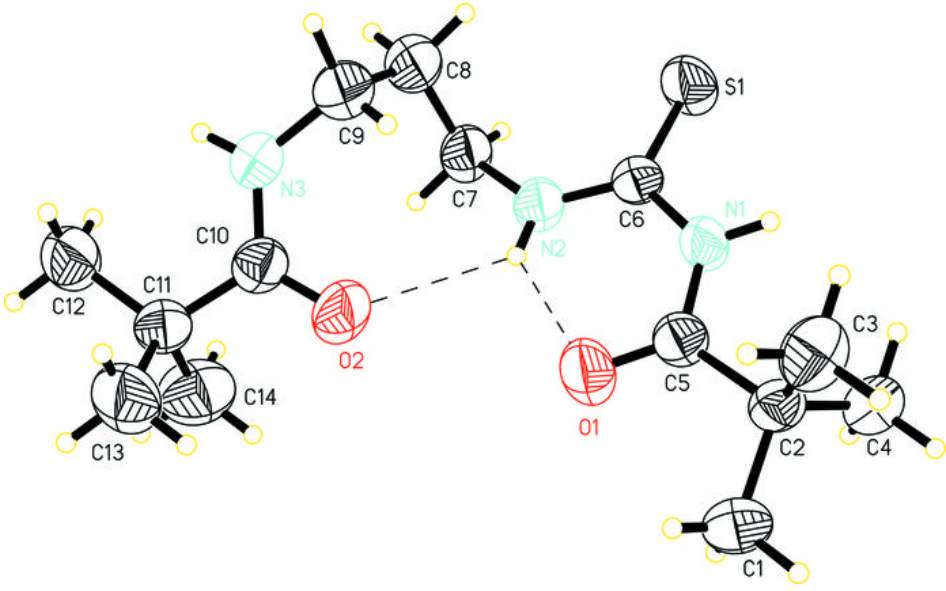

Fig. 2