Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Thiosemicarbazidium picrate monohydrate

Zhi-Yuan Xie

Department of Physics, Xiangfan University, Xiangfan 441053, People's Republic of China

Correspondence e-mail: zhiyuan_xie@126.com

Received 20 March 2007
Accepted 15 May 2007

In the title compound, $CH_6N_3S^+\cdot C_6H_2N_3O_7^-\cdot H_2O$, the components are linked by a number of N—H···O and O—H···O hydrogen bonds into a complex three-dimensional network.

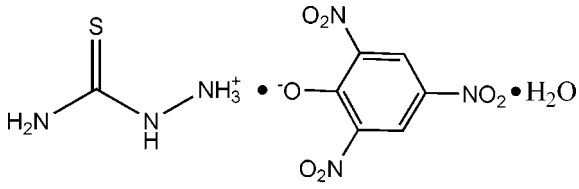
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.044
wR factor = 0.137
Data-to-parameter ratio = 10.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

Intermolecular or inter-ionic hydrogen-bonding interactions, which are not only the strongest of the non-covalent interactions but also highly directional, have played an important role in constructing supramolecular structures (Braga *et al.* 2004). Recently, we have obtained crystals of the title compound, (I), formed from thiosemicarbazide and picric acid, and report its crystal structure here.



(I)

In compound (I), the acidic H atom is released from the phenol hydroxyl group to atom N6, forming a 1:1 stoichiometric organic salt complex (Fig. 1) with one solvent water molecule. In (I), all H atoms except for H5 participate in hydrogen-bonding interactions.

In the supramolecular structure of (I), there are a number of intermolecular N—H···O, O—H···O, C—H···O, O—H···S and N—H···S hydrogen bonds, leading to a complex three-dimensional supramolecular network (Table 1 and Fig. 2).

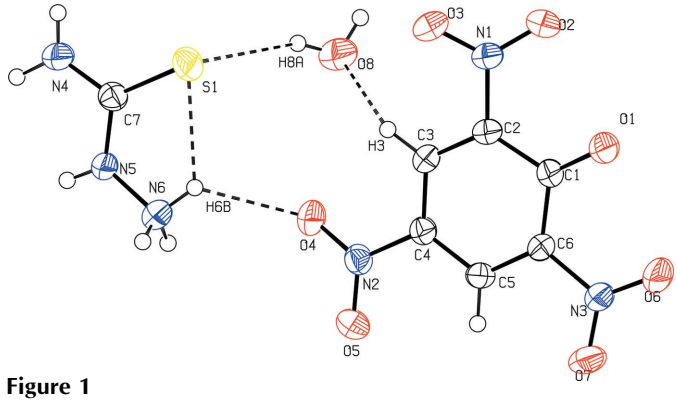


Figure 1
The asymmetric unit of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Dashed lines indicate hydrogen bonds.

Experimental

All reagents and solvents were used as obtained without further purification. Equimolar amounts of thiosemicarbazide (0.1 mmol, 23.0 mg) and picric acid (0.1 mmol, 9.1 mg) were dissolved in 95% methanol (20 ml). The mixture was stirred for half an hour at ambient temperature and then filtered. The resulting yellow solution was left to stand in air for several days. Yellow crystals of (I) suitable for a single-crystal X-ray diffraction analysis were grown on the bottom of the vessel by slow evaporation of the solution.

Crystal data

$CH_6N_3S^+\cdot C_6H_2N_3O_7^-\cdot H_2O$
M_r = 338.27
Triclinic, $P\overline{1}$
a = 7.1686 (5) Å
b = 9.7720 (6) Å
c = 10.3293 (7) Å
α = 94.904 (1)°
β = 104.613 (1)°
γ = 109.257 (1)°
V = 649.63 (7) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.31 mm^{-1}
T = 298 (2) K
0.26 × 0.20 × 0.18 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
T_{min} = 0.925, T_{max} = 0.941
3394 measured reflections
2377 independent reflections
2198 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.137
S = 1.08
2377 reflections
224 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.50 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O8	0.93	2.50	3.266 (3)	140
O8—H8A···S1	0.82 (3)	2.72 (2)	3.439 (2)	148 (4)
N6—H6B···O4	0.90 (3)	2.41 (3)	2.963 (3)	120 (2)
N6—H6B···S1	0.90 (3)	2.35 (3)	2.948 (2)	124 (2)
O8—H8B···O2^{i}	0.82 (3)	2.52 (3)	3.006 (3)	120 (3)
O8—H8B···O3^{i}	0.82 (3)	2.550 (17)	3.328 (3)	161 (4)
N6—H6C···O7^{ii}	0.85 (3)	2.21 (3)	2.791 (3)	125 (2)
N6—H6C···O3^{iii}	0.85 (3)	2.33 (3)	3.031 (3)	140 (3)
N6—H6A···O8^{iv}	0.89 (4)	1.82 (4)	2.708 (3)	176 (3)
N5—H5A···O6^{v}	0.79 (3)	2.46 (3)	3.072 (3)	134 (3)
N5—H5A···O1^{v}	0.79 (3)	1.92 (3)	2.647 (2)	153 (3)
N4—H4B···O2^{v}	0.92 (3)	2.29 (3)	3.056 (3)	140 (2)
N4—H4B···O1^{v}	0.92 (3)	1.99 (3)	2.781 (3)	143 (2)
N4—H4A···O5^{vi}	0.83 (3)	2.18 (3)	2.976 (3)	160 (3)

Symmetry codes: (i) $-x+1, -y+1, -z+2$; (ii) $-x+1, -y+2, -z+1$; (iii) $-x+1, -y+1, -z+1$; (iv) $-x, -y+1, -z+1$; (v) $x-1, y-1, z-1$; (vi) $x, y-1, z$.

H atoms bonded to atoms C3 and C5 were positioned geometrically, with C—H = 0.93 Å, and refined as riding with U_{iso}(H) = $1.2U_{eq}$(C). All H atoms bonded to N atoms were located in a difference map and refined without coordinate constraints but with U_{iso}(H) fixed at $1.2U_{eq}$(N). H atoms bonded to the water O atom were also located in a difference map, and were treated with O—H restrained to 0.82 (3) Å and with U_{iso}(H) = $1.5U_{eq}$(O).

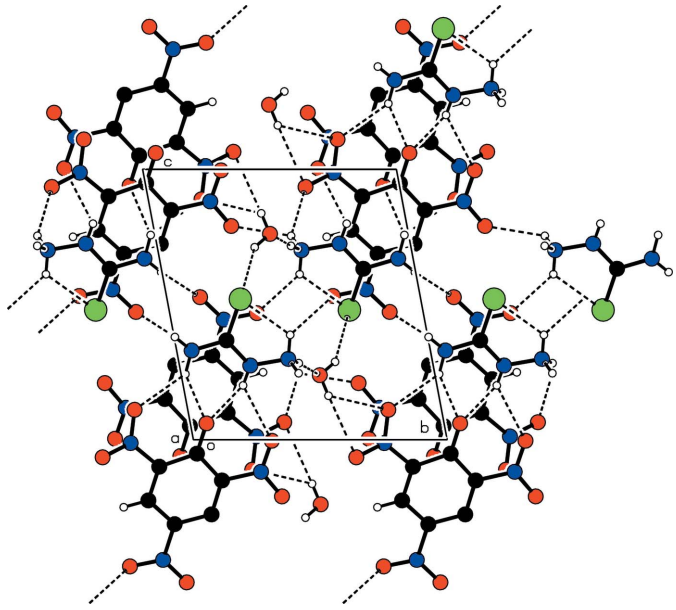


Figure 2
Part of the crystal structure of (I), showing the formation of the three-dimensional network. Hydrogen bonds are shown as dashed lines. For the sake of clarity, atoms H5 and the C—H hydrogen bonds have been omitted.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

This work received financial support mainly from the Key Fundamental Project (grant No. 2002CCA00500).

References

Braga, D., Maini, L., Polito, M. & Grepioni, F. (2004). *Struct. Bond.*, **111**, 1–32.
Bruker (2001). *SAINT-Plus* (Version 6.45) and *SMART* (Version 5.628). Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (2001). *SADABS*. Version 2.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.