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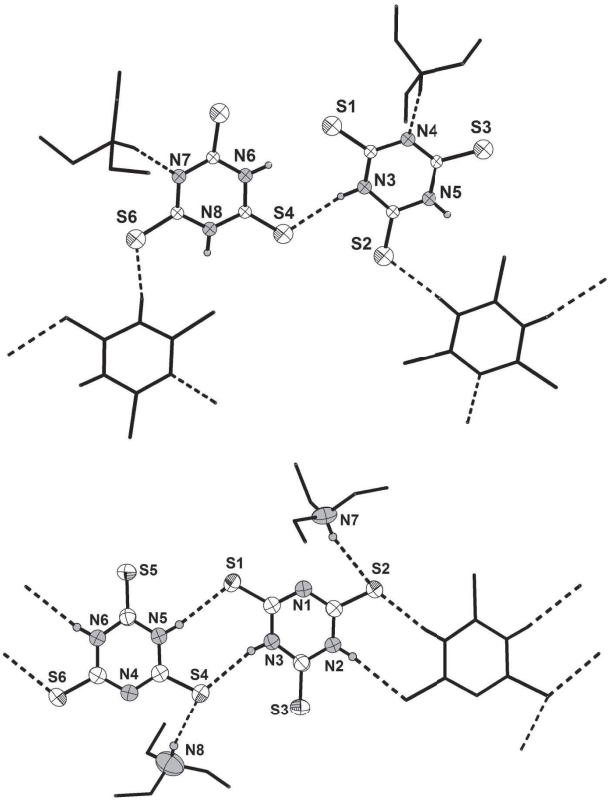
The second polymorph of triethylammonium 2,4,6-trisulfanylidene-1,3,5-triazinan-1-ide, $C_9H_{18}N_4S_3$



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Abstract

$C_9H_{18}N_4S_3$, monoclinic, $P2_1/c$ (no. 14), $a = 12.8444(3)$ Å, $b = 15.7980(5)$ Å, $c = 14.1803(4)$, $\beta = 98.293(2)°$, $V = 2847.32(14)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0510$, $wR_{ref}(F^2) = 0.1466$, $T = 296$ K.

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Table 1: Data collection and handling.

Crystal:	block, colorless, size 0.15×0.17×0.37 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	5.03 cm^{-1}
Diffractometer, scan mode:	CCD area detector, φ and ω scans
$2\theta_{max}$:	55.16°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	13621, 6579
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 4735
$N(param)_{refined}$:	307
Programs:	BRUKER Software [4], SHELX [5]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	Site	x	y	z	U_{iso}
H(2)	4e	1.056(1)	0.033(2)	0.164(2)	0.064
H(3)	4e	0.760(2)	0.029(2)	0.148(2)	0.066
H(5)	4e	0.548(2)	0.076(2)	0.098(2)	0.069
H(6)	4e	0.255(1)	0.070(2)	0.105(2)	0.067
H(7A)	4e	0.92520	0.16270	0.7876	0.086
H(7B)	4e	0.82540	0.17180	0.8386	0.086
H(7)	4e	0.965(3)	0.229(2)	0.922(2)	0.095
H(8)	4e	0.428(4)	0.095(2)	0.639(3)	0.137
H(8A)	4e	0.7837	0.2044	0.6786	0.156
H(8B)	4e	0.8685	0.2765	0.6893	0.156
H(8C)	4e	0.7682	0.2848	0.7399	0.156
H(9A)	4e	0.9856	0.3539	0.7951	0.101
H(9B)	4e	1.0563	0.3459	0.8942	0.101
H(10A)	4e	1.1474	0.3015	0.7751	0.147
H(10B)	4e	1.0616	0.2326	0.7436	0.147
H(10C)	4e	1.1330	0.2252	0.8427	0.147
H(11A)	4e	0.8058	0.2830	0.9424	0.095
H(11B)	4e	0.8295	0.3585	0.8770	0.095
H(12A)	4e	0.8628	0.3996	1.0349	0.171
H(12B)	4e	0.9686	0.4006	0.9919	0.171
H(12C)	4e	0.9438	0.3255	1.0574	0.171
H(13A)	4e	0.5856	0.1683	0.6811	0.186
H(13B)	4e	0.5270	0.2497	0.6384	0.186
H(14A)	4e	0.5450	0.2525	0.8018	0.294
H(14B)	4e	0.4826	0.2525	0.8018	0.294
H(14C)	4e	0.4254	0.2494	0.7594	0.294
H(15A)	4e	0.3352	0.2396	0.5902	0.208
H(15B)	4e	0.3142	0.1809	0.6752	0.208
H(16A)	4e	0.1827	0.1481	0.5357	0.282
H(16B)	4e	0.2528	0.0676	0.5607	0.282
H(16C)	4e	0.2731	0.1265	0.4763	0.282
H(17A)	4e	0.4331	0.0890	0.4894	0.289
H(17B)	4e	0.4516	0.1860	0.4871	0.289
H(18A)	4e	0.5740	0.1187	0.4412	0.290
H(18B)	4e	0.5956	0.0695	0.5381	0.290
H(18C)	4e	0.6143	0.1675	0.5358	0.290

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Table 3: Atomic displacement parameters (Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S(1)	4*e*	0.68466(5)	0.12886(5)	0.00357(5)	0.0382(3)	0.0625(4)	0.0580(4)	0.0032(3)	0.0017(3)	0.0149(3)
C(1)	4*e*	0.8058(2)	0.0977(2)	0.0515(2)	0.041(1)	0.043(1)	0.041(1)	0.001(1)	0.006(1)	0.002(1)
N(1)	4*e*	0.8923(2)	0.1257(1)	0.0182(2)	0.040(1)	0.052(1)	0.044(1)	0.0030(9)	0.0071(9)	0.010(1)
S(2)	4*e*	1.09725(5)	0.13404(5)	0.02222(6)	0.0397(3)	0.0814(5)	0.0583(4)	0.0012(3)	0.0111(3)	0.0261(4)
C(2)	4*e*	0.9102(2)	0.0176(2)	0.1758(2)	0.042(1)	0.038(1)	0.044(1)	0.003(1)	0.010(1)	0.002(1)
N(2)	4*e*	0.9945(2)	0.0469(1)	0.1380(2)	0.037(1)	0.050(1)	0.041(1)	0.0041(9)	0.0064(9)	0.0093(9)
S(3)	4*e*	0.92109(6)	−0.03944(5)	0.27461(6)	0.0564(4)	0.0634(5)	0.0569(4)	0.0062(3)	0.0120(3)	0.0249(4)
C(3)	4*e*	0.9866(2)	0.1002(2)	0.0612(2)	0.041(1)	0.046(1)	0.039(1)	0.002(1)	0.009(1)	0.002(1)
N(3)	4*e*	0.8165(2)	0.0428(1)	0.1273(2)	0.036(1)	0.048(1)	0.048(1)	−0.0002(9)	0.0073(9)	0.010(1)
S(4)	4*e*	0.63874(6)	0.02075(6)	0.26842(6)	0.0398(4)	0.1015(6)	0.0544(4)	0.0031(4)	0.0025(3)	0.0219(4)
C(4)	4*e*	0.5128(2)	0.0332(2)	0.2176(2)	0.043(1)	0.055(2)	0.046(1)	0.003(1)	0.007(1)	0.007(1)
N(4)	4*e*	0.4333(2)	0.0139(2)	0.2645(2)	0.042(1)	0.069(2)	0.043(1)	0.001(1)	0.007(1)	0.014(1)
S(5)	4*e*	0.37445(6)	0.11787(5)	−0.03062(5)	0.0508(4)	0.0663(5)	0.0431(4)	−0.0014(3)	0.0053(3)	0.0133(3)
C(5)	4*e*	0.3964(2)	0.0799(2)	0.0793(2)	0.039(1)	0.038(1)	0.044(1)	0.001(1)	0.007(1)	0.001(1)
N(5)	4*e*	0.4941(2)	0.0644(1)	0.1259(2)	0.037(1)	0.057(1)	0.044(1)	0.0018(9)	0.0084(9)	0.009(1)
S(6)	4*e*	0.22892(6)	0.00516(6)	0.27428(6)	0.0458(4)	0.0870(6)	0.0561(4)	−0.0009(4)	0.0158(3)	0.0199(4)
C(6)	4*e*	0.3346(2)	0.0276(2)	0.2217(2)	0.045(1)	0.048(1)	0.044(1)	0.001(1)	0.009(1)	0.005(1)
N(6)	4*e*	0.3189(2)	0.0626(1)	0.1317(2)	0.036(1)	0.055(1)	0.043(1)	0.0008(9)	0.0047(9)	0.009(1)
C(7)	4*e*	0.8759(3)	0.2031(2)	0.8076(2)	0.093(2)	0.063(2)	0.057(2)	−0.007(2)	0.005(2)	0.007(2)
N(7)	4*e*	0.9346(2)	0.2628(2)	0.8783(2)	0.086(2)	0.049(1)	0.052(2)	0.001(1)	−0.003(1)	0.012(1)
N(8)	4*e*	0.4374(3)	0.1429(2)	0.6133(3)	0.122(3)	0.066(2)	0.092(2)	−0.005(2)	0.036(2)	0.007(2)
C(8)	4*e*	0.8190(4)	0.2461(3)	0.7211(3)	0.123(4)	0.113(3)	0.068(2)	−0.004(3)	−0.014(2)	0.010(2)
C(9)	4*e*	1.0190(3)	0.3137(2)	0.8416(3)	0.092(3)	0.064(2)	0.096(3)	−0.007(2)	0.012(2)	0.016(2)
C(10)	4*e*	1.0972(4)	0.2638(3)	0.7968(3)	0.122(4)	0.084(3)	0.096(3)	−0.008(3)	0.041(3)	0.014(2)
C(11)	4*e*	0.8616(3)	0.3179(2)	0.9237(3)	0.097(3)	0.066(2)	0.077(2)	0.005(2)	0.018(2)	0.004(2)
C(12)	4*e*	0.9140(4)	0.3652(3)	1.0098(3)	0.146(5)	0.111(4)	0.087(3)	−0.010(3)	0.026(3)	−0.025(3)
C(13)	4*e*	0.5185(6)	0.1976(4)	0.6726(5)	0.176(6)	0.127(5)	0.164(6)	−0.025(4)	0.035(5)	−0.019(4)
C(14)	4*e*	0.4904(7)	0.2185(5)	0.7672(4)	0.33(1)	0.195(7)	0.077(4)	−0.048(7)	0.069(5)	−0.014(4)
C(15)	4*e*	0.3344(5)	0.1814(4)	0.6119(6)	0.137(5)	0.137(5)	0.254(9)	0.038(4)	0.058(6)	0.085(6)
C(16)	4*e*	0.2523(6)	0.1251(5)	0.5386(5)	0.159(6)	0.231(9)	0.156(6)	−0.001(6)	−0.038(5)	0.088(6)
C(17)	4*e*	0.4738(7)	0.1347(7)	0.5220(7)	0.22(1)	0.31(1)	0.22(1)	0.058(8)	0.144(9)	0.002(8)
C(18)	4*e*	0.5709(7)	0.1218(5)	0.5084(6)	0.213(8)	0.197(8)	0.190(8)	−0.019(6)	0.094(7)	−0.080(6)

Tables 1–3 contain details of the measurement and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

Trithiocyanuric acid (0.044 g, 0.25 mmol) was dissolved in a water-ethanol (1:2 v/v) mixture and triethylamine was added to neutralize the acid. Colorless block crystals formed after several weeks.

Experimental details

All hydrogen atoms bonded to carbon were introduced at idealized positions and allowed to ride on their parent atoms. Hydrogen atoms bonded to nitrogen were located in difference Fourier syntheses and refined with a N—H distance of 0.86 Å.

Discussion

1,3,5-Triazinane-2,4,6-trithione (thiocyanuric acid), which can be regarded as the polymer of three thiourea molecules, tends to form various hydrogen bonds [1]. In the aymmetric unit of the title structure, there are two thiocyanuric anions and two triethylammonium cations, in which the anions are both planar with the mean deviation from the least-square plane are 0.0352 Å and 0.0165 Å respectively. Two crystallographically independent triethylammonium cations form N—H···S hydrogen bonds to yield the stable title structure.

Previous articles report the crystal structures of tripropylammonium [2] and triethylammonium [3]. Interestingly, the crystal structure of the title compound has been reported before [3] which is different from the structure in this paper. The literature known structure (polymorph 1 [3]) is shown in the upper part of the figure, whereas the title structure (polymorph 2) is shown in the lower part. In both polymorphs, there are two thiocyanuric anions and two triethylammonium cations in the asymmetric unit. But the cell parameters are different: polymorph 1: $a = 13.1648$ (3) A, $b = 13.0636$ (3) A, $c = 16.9552$ (4) A, $\beta = 93.779$ (2)°. In both polymorphs, the thiocyanuric anions are linked to form hydrogen-bonded ribbons with N—H···S contacts and the cations both generate

N—H···S bonds with the anions, but the anionic ribbons show a completely different connectivity (see the figure).

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