Tableau 5. *Données géométriques* (Å) *des interactions intermoléculaires* (*molecules* I *et* II *associées autour d'un centre de symétrie*)

	(1)	(3)	(4)
$O(2)^{I}\cdots O(2)^{II}$	3,291 (5)	3,513 (7)	3,194 (3)
$O(3)^{I}\cdots O(3)^{II}$	4,456 (5)	4,276 (7)	4,406 (3)
$O(2)^{I}\cdots O(3)^{II}$	2,799 (5)	2,773 (7)	2,699 (3)
$O(2)^{I}\cdots H(3)^{II}$	1,867 (7)	1,845 (8)	1,939 (5)

Tableau 6. *Données géométriques* (Å) *des interactions intermoléculaires pour le composé* (5)

O(2*A*′)···O(3*B*)	3,141 (6)	O(2*A*′)···O(2*C*)	3,580 (6)
O(3*A*′)···O(3*B*)	2,786 (6)	H(3*B*)···O(2*C*)	1,748 (7)
H(3*A*′)···O(3*B*)	1,839 (7)	H(3*C*)···O(2*B*)	1,774 (7)
O(2*B*)···O(2*C*)	3,079 (7)		
O(2*B*)···O(2*C*)	2,759 (7)		
O(3*B*)···O(2*C*)	2,707 (8)		

Tous les atomes d'hydrogène du composé (1) sauf H(2), H(3), H(7) et H(11), atomes H(4) et H(5) du (2) et les hydrogènes cyclopropyls du (5*B*) ont étés introduits en positions idéalisées.

Programmes utilisés: *MULTAN*80 (Main *et al.*, 1980), *ORTEP*II (Johnson, 1976), *SHELXS*86 (Sheldrick, 1985) [composé (1)] et *SHELX*76 (Sheldrick, 1976) [composés (2)–(5)].

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Les listes des facteurs de structure, des facteurs d'agitation thermique anisotrope, des coordonnées des atomes d'hydrogène, des distances et angles des atomes d'hydrogène, des plans moyens et des distances intermoléculaires ont été déposées au dépôt d'archives de l'UICr (Référence: PA1059). On peut en obtenir des copies en s'adressant à: The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, Angleterre.

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4,5-Bis(2,4-dinitrophenylthio)-1,3-dithiol-2-one Nitromethane Solvate (BNPT-DTO.CH_3NO_2)

Wen-Tao Yu, Fang Qi, Xu Dong and
Min-Hua Jiang

Institute of Crystal Materials, Shandong University, Jinan 250100, People's Republic of China

Lei Hong

Department of Electronic Engineering, Shandong University, Jinan 250100, People's Republic of China

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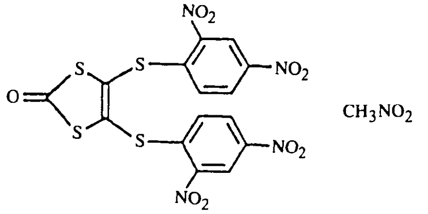
Abstract

The unit cell of the title crystal, $C_{15}H_6N_4O_9S_4.CH_3NO_2$, contains two BNPT-DTO molecules and two co-

crystallized nitromethane solvent molecules. A BNPT-DTO molecule comprises one conjugated 4,5-dimercapto-1,3-dithiol-2-one plane (analogous to 4,5-dimercapto-1,3-dithiole-2-thione, DMIT) and two phenyl planes which are almost perpendicular to the 'DMIT' plane. The dihedral angle between the phenyl planes is 45.8°. The crystal packing is stabilized by a number of intermolecular interactions in addition to normal van der Waals interactions.

Comment

Although DMIT (4,5-dimercapto-1,3-dithiole-2-thione) is one of the most important building blocks in crystals of organic salt-type or metal–DMIT complex-type electrical conductors or superconductors (Williams *et al.*, 1987; Cassoux & Valade, 1991), the secondary non-linear optical (NLO) properties of DMIT derivatives have not been reported until recently. It has been observed that BNPT-DTO, a DMIT derivative which crystallizes in the orthorhombic space group $P2_12_12_1$, exhibits an appreciable secondary NLO effect (Fang, Zheng, Yao & Xiao-Zeng, 1992; Fang *et al.*, 1994). In the course of crystal growth, we found that BNPT-DTO can crystallize with different crystal symmetry depending on the conditions, such as solvent and temperature. Here, we report the structure of a new triclinic crystal of BNPT-DTO with a CH_3NO_2 solvent molecule co-crystallized. The title compound, (I), is denoted as α-BNPT-DTO or BNPT-DTO.CH_3NO_2; accordingly, the aforementioned orthorhombic crystal of BNPT-DTO is denoted β-BNPT-DTO.



(I)

The bond lengths and angles of α-BNPT-DTO in the title compound are in good agreement with those of β-BNPT-DTO. All the C—S bond lengths fall within the normal range for C—S single-bond lengths with some double-bond character. Two kinds of C—S bonds found in the five-membered ring of the dithiole have slightly different lengths: C1—S2 1.778 (4) and C3—S2 1.747 (4) Å. The 'DMIT' moiety is roughly planar; the deviations from its least-squares plane are 0.0521 for S4 and −0.0603 Å for C3. Thus, this moiety is highly conjugated. The dihedral angles between the plane of this moiety and the two phenyl planes are 90.7 and 86.3°; the angle between the two phenyl planes is 45.8°. By comparison, the corresponding dihedral angles of β-BNPT-DTO are 94.1, 95.9 and 36.6°, respectively (Fang *et al.*, 1994). The molecular configurations of α-BNPT-DTO and β-BNPT-DTO are quite similar.

The two aromatic rings are essentially planar (deviations of atoms are less than 0.023 Å). The bond angles within the two aromatic rings at the nitro-substituted positions are greater than 120°, as commonly found in nitroaromatic compounds. In the nitromethane molecule, the sum of the three angles around N1′ is 360.1° showing that N1′ has an sp^2-hybridized orbital. The two N═O bonds have slightly different lengths of 1.220 (6) and 1.206 (6) Å, respectively.

A number of intermolecular distances shorter than the sum of the van der Waals radii are observed: O7···S1(*x*, −1 + *y*, *z*) 3.230, O5···C9(1 + *x*, *y*, *z*) 3.013 Å, S4···S2 and S4···S4(2 − *x*, 1 − *y*, 1 − *z*) 3.655 and 3.692, respectively, O6···S3(3 − *x*, 1 − *y*, 1 − *z*) 3.043, O2···N3(2 − *x*, −*y*, 1 − *z*) 2.864 Å. These may stabilize the crystal packing.

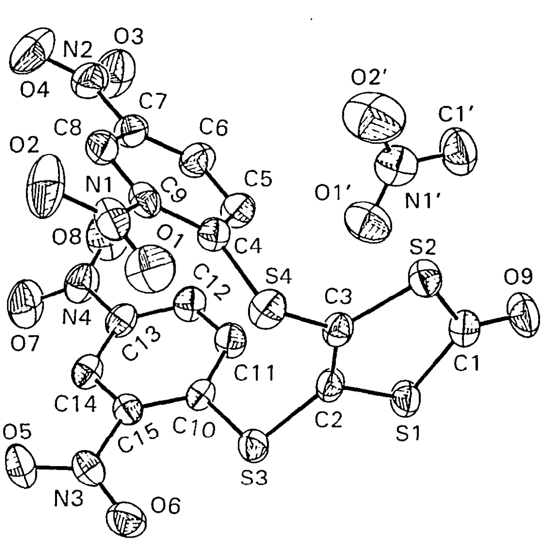


Fig. 1. An *ORTEP* (Johnson, 1965) drawing of BNPT-DTO.CH_3NO_2 with 50% probability displacement ellipsoids.

Experimental

The title compound was synthesized by Fang *et al.* (1994) and recrystallized in CH_3NO_2 below 322 K.

Crystal data

$C_{15}H_6N_4O_9S_4.CH_3NO_2$
$M_r = 575.51$
Triclinic
$P\overline{1}$
$a = 8.582\,(3)$ Å
$b = 9.702\,(2)$ Å
$c = 15.004\,(4)$ Å
$\alpha = 93.75\,(2)°$
$\beta = 98.00\,(3)°$
$\gamma = 114.38\,(2)°$
$V = 1116\,(1)$ Å^3
$Z = 2$
$D_x = 1.71$ Mg m^{-3}

Mo *Kα* radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 3–14°$
$\mu = 0.48$ mm^{-1}
$T = 293$ K
Parallelepiped
0.3 × 0.3 × 0.2 mm
Yellow

Data collection

Nicolet $R3m/E$ diffractometer
$\omega/2\theta$ scans
Absorption correction: none
4336 measured reflections
4246 independent reflections
3397 observed reflections [$F > 3\sigma(F)$]
$R_{\rm int} = 0.0150$
$\theta_{\rm max} = 25.0°$
$h = 0 \rightarrow 11$
$k = -12 \rightarrow 12$
$l = -18 \rightarrow 18$
2 standard reflections monitored every 100 reflections intensity decay: <5%

Refinement

Refinement on F
$R = 0.0466$
$wR = 0.0466$
$S = 0.973$
3397 reflections
325 parameters
H-atom parameters not refined
$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{\rm max} = 0.032$
$\Delta\rho_{\rm max} = 0.63$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.29$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	$U_{\rm eq}$
S1	1.2680 (1)	0.6734 (1)	0.2733 (1)	0.043 (1)
S2	0.9181 (1)	0.5441 (1)	0.3207 (1)	0.041 (1)
S3	1.3871 (1)	0.4694 (1)	0.3790 (1)	0.038 (1)
S4	0.9908 (1)	0.3274 (1)	0.4354 (1)	0.041 (1)
C1	1.0622 (5)	0.6797 (4)	0.2616 (3)	0.043 (2)
C2	1.2196 (4)	0.5230 (4)	0.3386 (2)	0.034 (1)
C3	1.0600 (4)	0.4650 (4)	0.3604 (2)	0.034 (1)
C4	0.8867 (4)	0.1567 (4)	0.3584 (2)	0.035 (1)
C5	0.8700 (5)	0.1600 (4)	0.2641 (2)	0.041 (1)
C6	0.7973 (5)	0.0267 (4)	0.2026 (3)	0.044 (2)
C7	0.7398 (4)	−0.1116 (4)	0.2364 (3)	0.041 (1)
C8	0.7450 (4)	−0.1236 (4)	0.3273 (3)	0.040 (1)
C9	0.8178 (4)	0.0123 (4)	0.3873 (2)	0.036 (1)
C10	1.3539 (4)	0.3219 (4)	0.2919 (2)	0.033 (1)
C11	1.2583 (5)	0.3057 (4)	0.2051 (2)	0.041 (2)
C12	1.2275 (5)	0.1844 (4)	0.1384 (2)	0.044 (2)
C13	1.2960 (5)	0.0823 (4)	0.1605 (2)	0.039 (1)
C14	1.3930 (5)	0.0932 (4)	0.2449 (2)	0.039 (1)
C15	1.4202 (4)	0.2147 (4)	0.3086 (2)	0.033 (1)
N1	0.8181 (4)	−0.0031 (4)	0.4839 (2)	0.044 (1)
N2	0.6684 (4)	−0.2525 (4)	0.1707 (3)	0.054 (1)
N3	1.5241 (4)	0.2237 (4)	0.3968 (2)	0.041 (1)
N4	1.2634 (5)	−0.0471 (4)	0.0919 (2)	0.054 (2)
O9	1.0252 (4)	0.7661 (3)	0.2191 (2)	0.059 (1)
O1	0.8801 (4)	0.1129 (3)	0.5381 (2)	0.059 (1)
O2	0.7596 (4)	−0.1309 (3)	0.5063 (2)	0.070 (2)
O3	0.6739 (5)	−0.2387 (4)	0.0910 (2)	0.075 (2)
O4	0.6090 (4)	−0.3751 (3)	0.2004 (3)	0.076 (2)
O5	1.5351 (4)	0.1077 (3)	0.4184 (2)	0.056 (1)
O6	1.5977 (4)	0.3498 (3)	0.4440 (2)	0.054 (1)
O7	1.3438 (5)	−0.1244 (4)	0.1083 (2)	0.074 (2)
O8	1.1591 (5)	−0.0694 (4)	0.0226 (2)	0.086 (2)
C1′	0.7423 (7)	0.4637 (6)	0.0102 (3)	0.073 (3)
N1′	0.7771 (5)	0.3696 (4)	0.0782 (2)	0.059 (2)
O1′	0.9283 (4)	0.3944 (4)	0.1051 (2)	0.074 (2)
O2′	0.6552 (5)	0.2721 (5)	0.1025 (3)	0.110 (2)

Table 2. *Selected geometric parameters* (Å, °)

S1—C1	1.777 (5)	S4—C4	1.766 (3)
S2—C3	1.747 (4)	C1′—N1′	1.492 (7)
S4—C3	1.770 (4)	S2—C1	1.778 (4)
C2—C3	1.346 (5)	S3—C10	1.776 (4)
N1′—O2′	1.206 (6)	C1—O9	1.201 (6)
S1—C2	1.749 (4)	N1′—O1′	1.220 (6)
S3—C2	1.767 (4)		
C1—S1—C2	96.9 (2)	C1—S2—C3	96.5 (2)
C2—S3—C10	101.6 (2)	C3—S4—C4	101.2 (2)
S1—C1—S2	112.1 (2)	S1—C1—O9	123.9 (3)
S2—C1—O9	124.0 (3)	S1—C2—S3	118.2 (2)
S1—C2—C3	116.8 (3)	S3—C2—C3	124.7 (3)
S2—C3—S4	117.4 (2)	S2—C3—C2	117.8 (3)
S4—C3—C2	124.6 (3)	S4—C4—C5	120.9 (3)
S4—C4—C9	122.4 (3)	S3—C10—C11	122.1 (3)
S3—C10—C15	120.6 (2)	C1′—N1′—O1′	117.7 (4)
C1′—N1′—O2′	118.7 (5)	O1′—N1′—O2′	123.7 (5)

Data collection: *P*3 *Data Collection Program* (Nicolet, 1985). Cell refinement: *P*3 *Data Collection Program*. Data reduction, structure solution, structure refinement, molecular graphics and preparation of material for publication: *SHELXTL* (Sheldrick, 1984).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AS1133). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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An Epoxyamide

William Clegg,* Massimo Ranelli and
Richard F. W. Jackson

Department of Chemistry, University of Newcastle, Newcastle upon Tyne NE1 7RU, England

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Abstract

The title compound, (2*S*,3*R*)-3-phenyl-*N*,*N*-bis[(*R*)-1-phenylethyl]oxirane-2-carboxamide, $C_{25}H_{25}NO_2$, con-