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Crystal structures of three (trichloromethyl)(carbamoyl)disulfanes

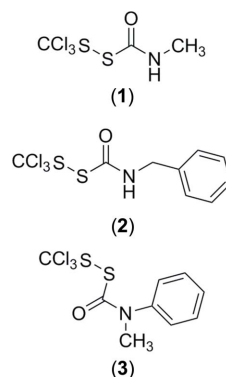
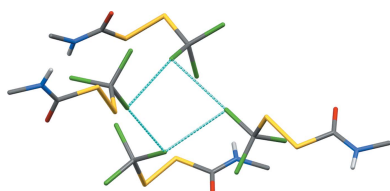
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The present paper reports crystallographic studies on three related compounds that were of interest as precursors for synthetic and mechanistic work in organosulfur chemistry, as well as to model nitrogen-protecting groups: (*N*-methylcarbamoyl)(trichloromethyl)disulfane, $C_3H_4Cl_3NOS_2$, (**1**), (*N*-benzylcarbamoyl)(trichloromethyl)disulfane, $C_9H_8Cl_3NOS_2$, (**2**), and (*N*-methyl-*N*-phenylcarbamoyl)(trichloromethyl)disulfane, $C_9H_8Cl_3NOS_2$, (**3**). Their molecular structures, with similar bond lengths and angles for the $CCl_3SS(C=O)N$ moieties, are confirmed. Compounds (**1**) and (**3**) both crystallized with two independent molecules in the asymmetric unit. Classical hydrogen bonding, as well as chlorine-dense regions, are evident in the crystal packing for (**1**) and (**2**). In the crystal of (**1**), molecules are linked *via* $N-H\cdots O$ hydrogen bonds forming chains along [110], which are linked by short $Cl\cdots Cl$ and $S\cdots O$ contacts forming sheets parallel to (001). In the crystal of (**2**), molecules are linked *via* $N-H\cdots O$ hydrogen bonds forming chains along [001], which in turn are linked by pairs of short $O\cdots Cl$ contacts forming ribbons along the *c*-axis direction. In the crystal of (**3**), there are no classical hydrogen bonds present and the chlorine-dense regions observed in (**1**) and (**2**) are lacking.

1. Chemical context

Carbamoyl disulfanes were first reported by Harris (1960). This family of compounds has served as useful model compounds for synthetic and mechanistic work in organosulfur chemistry and nitrogen-protecting-group development (Barany & Merrifield, 1977; Barany *et al.*, 1983; Schroll & Barany, 1986; Barany *et al.*, 2005; Schrader *et al.*, 2011). The trichloromethyl derivatives reported here, (trichloromethyl)(*N*-methylcarbamoyl)disulfane, (**1**) (Fig. 1), (trichloromethyl)(*N*-benzylcarbamoyl)disulfane, (**2**) (Fig. 2), and (trichloromethyl)(*N*-methyl-*N*-phenylcarbamoyl)disulfane, (**3**) (Fig. 3), are particularly stable. All three compounds have been stored under ambient conditions for periods in the range of two to four decades, with no evidence of decomposition based on unchanged 1H NMR spectra and melting points.



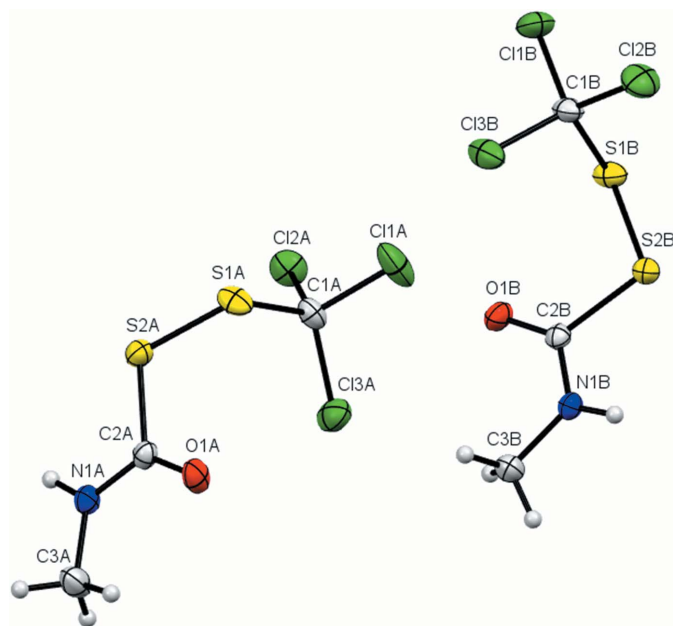


Figure 1
The molecular structure of compound (1) showing the atom-labelling scheme, with two molecules ($Z' = 2$) per asymmetric unit. Displacement ellipsoids are drawn at the 50% probability level.

2. Structural commentary

The three (trichloromethyl)(carbamoyl)disulfanes differ in the substituents on the carbamoyl nitrogen, but the bond lengths and angles of the common $\text{CCl}_3\text{SS}(\text{C}=\text{O})\text{N}$ moieties of each are markedly similar for the two molecules in the asymmetric units of (1) and (3), as well as for the single conformation of (2) (Tables 1 and 2). The corresponding structural features of (3) are also similar to the bond lengths and torsion angles of other carbamoyl disulfanes that include an $\text{SS}(\text{C}=\text{O})\text{N}(\text{Me})\text{Ph}$ chain, including, for example, bis(*N*-methyl-*N*-phenylcarbamoyl)disulfane (ZAQWUL, formula $[\text{Ph}(\text{Me})\text{N}(\text{C}=\text{O})\text{S}]_2$) (Schroll *et al.*, 2012) and (*N*-methyl-*N*-phenylcarbamoyl)(*N*-methyl-*N*-phenylamino)disulfane [formula $\text{Ph}(\text{Me})\text{N}(\text{C}=\text{O})\text{SSN}(\text{Me})\text{Ph}$] (Henley *et al.*, 2015).

Table 1
Selected bond lengths (Å) and angles (°) for $\text{CCl}_3\text{SS}(\text{C}=\text{O})\text{N}$ moieties.

	(1a)	(1b)	(2)	(3a)	(3b)
S1–C1	1.8242 (18)	1.8261 (18)	1.826 (3)	1.824 (2)	1.822 (2)
S1–S2	2.0100 (7)	2.0126 (6)	2.0099 (11)	2.0202 (7)	2.0160 (7)
S2–C2	1.8367 (17)	1.8426 (17)	1.842 (3)	1.856 (2)	1.842 (2)
O1–C2	1.214 (2)	1.212 (2)	1.213 (4)	1.208 (2)	1.211 (2)
N1–C2	1.322 (2)	1.324 (2)	1.319 (4)	1.345 (3)	1.346 (3)
N1–C3	1.458 (2)	1.460 (2)	1.475 (4)	1.467 (3)	1.460 (3)
N1–C4	–	–	–	1.440 (3)	1.447 (3)
C1–S1–S2	103.09 (6)	103.10 (6)	103.68 (11)	102.38 (7)	104.40 (7)
C2–S2–S1	102.20 (6)	101.43 (6)	101.40 (10)	99.96 (7)	101.59 (7)
C2–N1–C3	121.71 (15)	120.35 (14)	121.8 (3)	118.95 (18)	119.49 (18)
O1–C2–N1	126.31 (16)	126.23 (16)	126.4 (3)	125.9 (2)	126.4 (2)
O1–C2–S2	123.02 (13)	122.17 (13)	122.4 (2)	122.09 (16)	122.96 (16)
N1–C2–S2	110.67 (12)	111.58 (12)	111.2 (2)	111.99 (15)	110.65 (14)

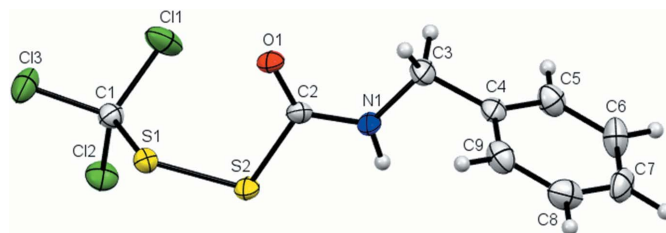


Figure 2
The molecular structure of compound (2) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

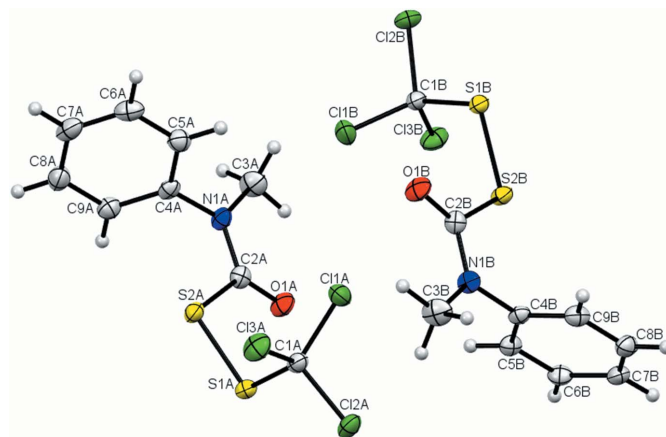


Figure 3
The molecular structure of compound (3) showing the atom-labelling scheme, with two molecules ($Z' = 2$) per asymmetric unit. Displacement ellipsoids are drawn at the 50% probability level.

3. Supramolecular features

The three compounds arrange in three distinct packing configurations. The two nearly superimposable molecular structures of (1) are alternately hydrogen-bonded ($\text{NH}\cdots\text{O}=\text{C}$) in chains along [110] (Table 3). Successive molecules of each of two chains are linked by 3.162 (1) Å $\text{S1A}\cdots\text{O1B}$ contacts, 0.157 Å less than their van der Waals radii sum (Fig. 4). Additional packing features result in a $Z = 16$ unit cell. A chlorine from each of four molecules – in separate hydrogen-bonded chains – form a short-contact skew quadrilateral with intermolecular contact distances of

Table 2

 Comparison of selected torsion angles ($^{\circ}$).

	(1a)	(1b)	(2)	(3a)	(3b)
C1–S1–S2–C2	93.63 (8)	93.49 (8)	96.54 (14)	92.91 (10)	–95.23 (10)
C3–N1–C2–O1	3.3 (3)	1.6 (3)	–1.3 (5)	0.3 (3)	–0.8 (3)
C3–N1–C2–S2	–176.22 (14)	–176.67 (12)	–178.2 (3)	–179.98 (15)	179.73 (16)
S1–S2–C2–O1	2.87 (16)	–0.66 (15)	–2.5 (3)	10.32 (19)	6.32 (19)
S1–S2–C2–N1	–177.64 (11)	177.64 (11)	174.6 (2)	–169.40 (14)	–174.23 (13)
C2–N1–C4–C9	–	–	–	–72.9 (3)	93.8 (2)
C2–N1–C4–C5	–	–	–	109.7 (2)	–86.4 (3)
C3–N1–C4–C9	–	–	–	104.1 (2)	–78.0 (3)
C3–N1–C4–C5	–	–	–	–73.3 (3)	101.8 (2)

Table 3

 Hydrogen-bond geometry (\AA , $^{\circ}$) for (1).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1A-H1AA\cdots O1B^i$	0.86 (1)	1.94 (1)	2.7825 (18)	164 (2)
$N1B-H1BA\cdots O1A^{ii}$	0.86 (1)	1.97 (1)	2.8231 (18)	175 (2)

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

Table 4

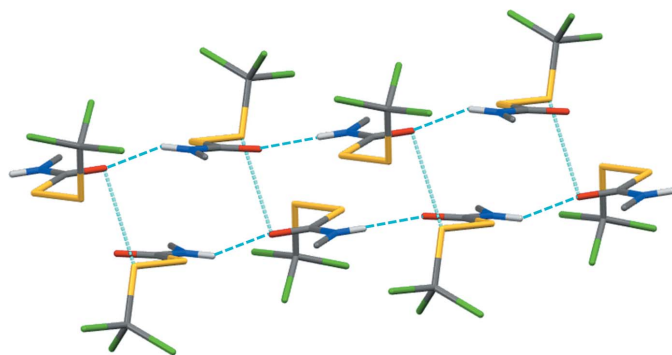
 Hydrogen-bond geometry (\AA , $^{\circ}$) for (2).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1-H1A\cdots O1^i$	0.87 (1)	2.02 (1)	2.887 (3)	174 (3)

 Symmetry code: (i) $x, -y + \frac{3}{2}, z + \frac{1}{2}$.

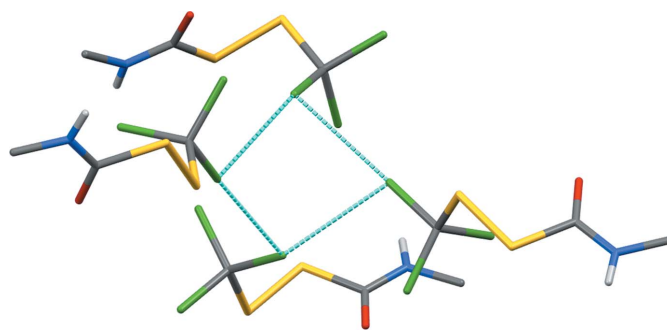
3.4304 (8) \AA (–0.070 \AA less than their van der Waals radii sum) and 3.3463 (8) \AA (–0.154 \AA less than their van der Waals radii sum), $Cl3B\cdots Cl1A\cdots Cl3B$ and $Cl1A\cdots Cl3B\cdots Cl1A$ angles 73.40 (2) and 82.01 (2) $^{\circ}$, and $Cl3B\cdots Cl1A\cdots Cl3B\cdots Cl1A$ and $Cl1A\cdots Cl3B\cdots Cl1A\cdots Cl3B$ torsion angles –50.45 (2) and 48.78 (2) $^{\circ}$. These result in chlorine-dense regions of the crystal structure (Fig. 5), and the formation of sheets parallel to (001). Halogen bonding involving trichloromethyl groups in supramolecular structures was described by Rybarczyk-Pirek *et al.* (2013).

The unit cell of (2) consists of pairs of hydrogen-bonded dimers about an inversion center. The molecules in each dimer are linked by $NH\cdots O=C$ hydrogen bonds (Table 4), which extend into hydrogen-bonded molecular chains along [001]. A network of linked chains is formed by $O1\cdots Cl3$ contacts. Two

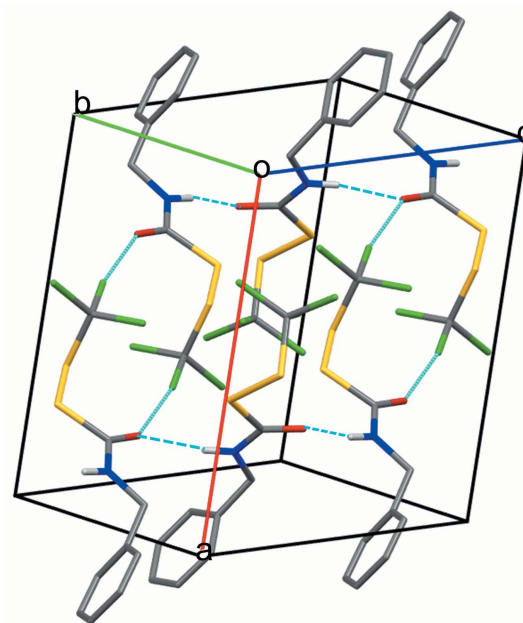

Figure 4

Hydrogen-bonded chains of (1) are linked by $S1A\cdots O1B$ contacts. Only H atoms involved in $N-H\cdots O=C$ bonds are shown.

$O1\cdots Cl3$ contacts [3.028 (2) \AA , 0.242 \AA less than their van der Waals radii sum] form between each pair of molecules in separate hydrogen-bonded chains, and the links extend throughout the chains in alternate molecules. In this way, each


Figure 5

A chlorine from each of four molecules of (1), in separate chains, form a short-contact skew quadrilateral. Only H atoms involved in $N-H\cdots O=C$ bonds are shown.


Figure 6

Packing structure of (2). Hydrogen-bonded chains are linked by pairs of $O1\cdots Cl3$ contacts. H atoms are not shown unless they participate in hydrogen bonding.

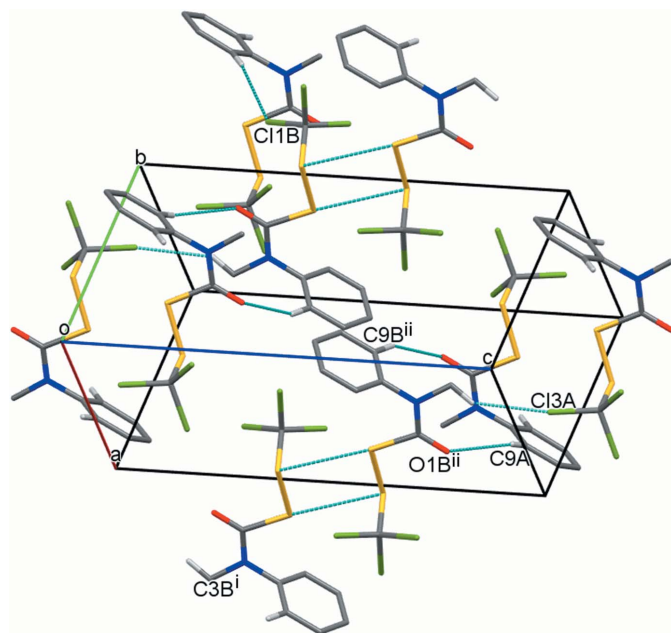


Figure 7
Packing diagram for (3). H atoms are not shown unless they participate in hydrogen bonding. [Symmetry codes: (i) $-x + 1, -y, -z$; (ii) $x, y + 1, z$.]

hydrogen-bonded chain has extensive links to two other chains. The resulting structure features alternating layers of trichloromethyl and benzyl groups (Fig. 6).

Compound (3) has no available classical hydrogen bonding and lacks the chlorine-dense regions of (1) and (2). Of the two conformations available for (3), it is noteworthy that the four sulfurs of two adjacent molecules of (3b) are positioned in a parallelogram [angles 80.65 (2) and 99.35 (2)°, torsion angle 0.00 (2)°] with intermolecular contact distances of 3.5969 (8) Å, slightly less than the sum of their van der Waals radii; no such configuration is evident for molecules of (3a). Fig. 7 shows a schematic view of the intermolecular interactions. A pair of non-classical hydrogen bonds [C9A—H9AA···O1B and C9B—H9BA···O1A, with H···C contact distances 2.55 and 2.54 Å, C···O distances of 3.360 (3) and 3.432 (3) Å, and C—H···O angles of 143 and 157°] connect (3a) and (3b) molecules. Two additional non-classical hydrogen bonds [C5A—H5AA···Cl1B and C3B—H3BA···Cl3A, with H···Cl contact distances 2.82 and 2.81 Å, C···Cl distances of 3.732 (2) and 3.649 (2) Å, and C—H···Cl angles of 161 and 144°] are shown.

4. Database survey

Crystal structures for two additional carbamoyl disulfanes have been reported: bis(indolylcarbamoyl)disulfane (BOWGAV, formula $[C_8H_6N(C=O)S]_2$) (Bereman *et al.*, 1983) and bis(*N,N*-dicyclohexylcarbamoyl)disulfane (UDALER, $[C_{12}H_{22}N(C=O)S]_2$) (Li *et al.*, 2006). Their molecular structures are consistent with those of the three compounds reported here. Neither of these comparison structures contains halogen atoms or supramolecular

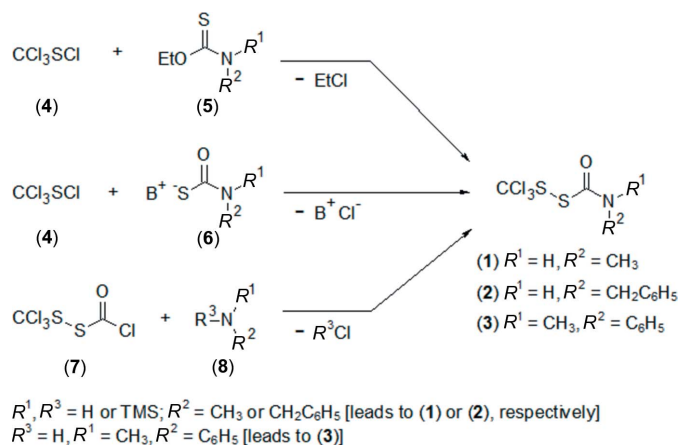


Figure 8
Synthetic routes to (trichloromethyl)(carbamoyl)disulfanes, (1), (2) and (3). See text for further details.

hydrogen bonds. The crystal structure of 1,7-bis(trichloromethyl)heptasulfane contains both short Cl···Cl contacts and a parallelogram (four sulfurs) formed from the trichloromethyl-adjacent S—S bonds of two molecules (REHKUK; Steudel *et al.*, 1995).

5. Synthesis and crystallization

Compounds (1) (Harris, 1960; Barany *et al.*, 2005), (2) (Barany *et al.*, 2005), and (3) (Barany *et al.*, 1983; Schroll & Barany, 1986) were synthesized and crystallized as outlined in Fig. 8 and described in the referenced publications. The reaction of (4) plus (5), shown in the top pathway of Fig. 8, is termed the Harris reaction (Harris, 1960). For the alternative Harris pathway shown in the middle of Fig. 8, compound (6), a thiocarbamate salt, is typically made by reaction of carbonyl sulfide (COS) with a primary or secondary amine HNR^1R^2 . Therefore B^+ is usually the appropriate ammonium counterion $H_2N^+R^1R^2$. Finally, several variations of acylation chemistry are summarized in the bottom pathway of Fig. 8, as originally worked out by Barany *et al.* (2005). When $R^3 = H$, starting amine HNR^1R^2 is present in sufficient excess so that a second equivalent of amine can absorb the HCl co-product. When R^1 and/or $R^3 = TMS$, stoichiometric ratios can be used, since co-product TMS-Cl is neutral. Note that for some reactions, a TMS group attached to N becomes an H after aqueous workup.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 5. N—H hydrogen atoms were refined positionally, with restrained $d(N-H) = 0.85$ (1) Å. H atoms attached to C were idealized (C—H: 0.95 Å, C—H₂: 0.99 Å, C—H₃: 0.98 Å). In all cases, $U_{iso}(H) = x \times U_{eq}(Host)$, $x = 1.2$ except for methyl groups, where $x = 1.5$.

Table 5
Experimental details.

	(1)	(2)	(3)
Crystal data			
Chemical formula	C ₃ H ₄ Cl ₃ NOS ₂	C ₉ H ₈ Cl ₃ NOS ₂	C ₉ H ₈ Cl ₃ NOS ₂
<i>M_r</i>	240.54	316.63	316.63
Crystal system, space group	Monoclinic, <i>C2/c</i>	Monoclinic, <i>P2₁/c</i>	Triclinic, <i>P$\bar{1}$</i>
Temperature (K)	123	173	123
<i>a</i> , <i>b</i> , <i>c</i> (Å)	13.1141 (16), 13.9234 (17), 20.172 (3)	11.4247 (17), 13.548 (2), 8.5675 (12)	8.9231 (12), 10.1724 (13), 15.364 (2)
α , β , γ (°)	90, 98.969 (2), 90	90, 103.176 (2), 90	81.964 (2), 81.806 (2), 68.851 (2)
<i>V</i> (Å ³)	3638.3 (8)	1291.2 (3)	1281.5 (3)
<i>Z</i>	16	4	4
Radiation type	Mo <i>K</i> α	Mo <i>K</i> α	Mo <i>K</i> α
μ (mm ⁻¹)	1.40	1.01	1.02
Crystal size (mm)	0.40 × 0.30 × 0.11	0.30 × 0.15 × 0.10	0.25 × 0.20 × 0.09
Data collection			
Diffractometer	Bruker SMART CCD area detector	Bruker SMART CCD area detector	Bruker SMART CCD area detector
Absorption correction	Multi-scan (<i>SADABS</i> ; Sheldrick, 2008)	Multi-scan <i>SADABS</i> , (Sheldrick, 2008)	Multi-scan (<i>SADABS</i> ; Sheldrick, 2008)
<i>T_{min}</i> , <i>T_{max}</i>	0.646, 0.746	0.752, 0.906	0.676, 0.746
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	21324, 4168, 3556	12180, 2284, 2056	15282, 5790, 4557
<i>R_{int}</i>	0.030	0.041	0.034
(sin θ/λ) _{max} (Å ⁻¹)	0.650	0.596	0.649
Refinement			
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.025, 0.061, 1.03	0.042, 0.080, 1.00	0.030, 0.073, 0.97
No. of reflections	4168	2284	5790
No. of parameters	189	148	291
No. of restraints	2	1	0
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
Δρ _{max} , Δρ _{min} (e Å ⁻³)	0.80, -0.63	0.33, -0.27	0.39, -0.27

Computer programs: *SMART* and *SAINT* (Bruker, 2007), *SHELXL2014* (Sheldrick, 2015), *SHELXTL* (Sheldrick, 2008), *Mercury* (Macrae *et al.*, 2008), *PLATON* (Spek, 2009) and *ACD/ChemBioDraw* (ACD/Labs, 2014).

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supporting information

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Crystal structures of three (trichloromethyl)(carbamoyl)disulfanes

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Computing details

For all compounds, data collection: *SMART* (Bruker, 2007); cell refinement: *SMART* (Bruker, 2007); data reduction: *SAINTE* (Bruker, 2007); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *SHELXTL* (Sheldrick, 2008) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008), *PLATON* (Spek, 2009), *ACD/ChemBioDraw* (ACD/Labs, 2014).

(1) (*N*-Methylcarbamoyl)(trichloromethyl)disulfane

Crystal data

$C_3H_4Cl_3NOS_2$

$M_r = 240.54$

Monoclinic, *C2/c*

$a = 13.1141$ (16) Å

$b = 13.9234$ (17) Å

$c = 20.172$ (3) Å

$\beta = 98.969$ (2)°

$V = 3638.3$ (8) Å³

$Z = 16$

$F(000) = 1920$

$D_x = 1.757$ Mg m⁻³

Melting point = 352–353 K

Mo *K*α radiation, $\lambda = 0.71073$ Å

Cell parameters from 2920 reflections

$\theta = 2.5$ – 27.5 °

$\mu = 1.40$ mm⁻¹

$T = 123$ K

Plate, colorless

$0.40 \times 0.30 \times 0.11$ mm

Data collection

Bruker SMART CCD area detector
diffractometer

Radiation source: sealed tube

phi and ω scans

Absorption correction: multi-scan
(*SADABS*; Sheldrick, 2008)

$T_{\min} = 0.646$, $T_{\max} = 0.746$

21324 measured reflections

4168 independent reflections

3556 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.030$

$\theta_{\max} = 27.5$ °, $\theta_{\min} = 2.0$ °

$h = -16 \rightarrow 17$

$k = -18 \rightarrow 17$

$l = -26 \rightarrow 26$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.025$

$wR(F^2) = 0.061$

$S = 1.03$

4168 reflections

189 parameters

2 restraints

Hydrogen site location: mixed

H atoms treated by a mixture of independent
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0241P)^2 + 4.8271P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.002$

$\Delta\rho_{\max} = 0.80$ e Å⁻³

$\Delta\rho_{\min} = -0.63$ e Å⁻³

Special details

Experimental. Compound (1) (Harris, 1960; Barany *et al.*, 2005) was synthesized and crystallized as outlined in the Scheme and described in the referenced publications.

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C11A	−0.00541 (4)	0.37732 (4)	0.35962 (3)	0.04712 (15)
C12A	0.19373 (3)	0.28755 (4)	0.39682 (2)	0.03390 (11)
C13A	0.08631 (4)	0.35934 (3)	0.49989 (3)	0.03684 (12)
S1A	−0.00492 (3)	0.19396 (3)	0.41765 (2)	0.02805 (11)
S2A	0.08261 (4)	0.10871 (3)	0.48401 (2)	0.02537 (10)
O1A	−0.04393 (10)	0.18404 (9)	0.56382 (7)	0.0302 (3)
N1A	0.08133 (11)	0.08323 (10)	0.61229 (7)	0.0239 (3)
H1AA	0.1296 (12)	0.0437 (12)	0.6058 (10)	0.029*
C1A	0.07085 (13)	0.30414 (13)	0.42009 (9)	0.0255 (4)
C2A	0.02947 (13)	0.13163 (11)	0.56154 (9)	0.0211 (3)
C3A	0.05085 (16)	0.08425 (15)	0.67878 (9)	0.0334 (4)
H3AA	0.0441	0.0181	0.6942	0.050*
H3AB	−0.0155	0.1174	0.6767	0.050*
H3AC	0.1034	0.1179	0.7102	0.050*
C11B	0.27558 (4)	0.53778 (4)	0.16143 (2)	0.03787 (12)
C12B	0.08921 (4)	0.64264 (4)	0.17392 (3)	0.04065 (13)
C13B	0.15634 (4)	0.48946 (4)	0.26583 (2)	0.03589 (12)
S1B	0.28444 (4)	0.66744 (3)	0.27052 (2)	0.02689 (10)
S2B	0.19567 (4)	0.71821 (3)	0.33553 (2)	0.02639 (10)
O1B	0.29056 (10)	0.57063 (9)	0.40606 (6)	0.0273 (3)
N1B	0.18133 (11)	0.65897 (10)	0.45778 (7)	0.0222 (3)
H1BA	0.1388 (12)	0.7061 (11)	0.4536 (10)	0.027*
C1B	0.19848 (14)	0.58421 (13)	0.21883 (8)	0.0254 (4)
C2B	0.22943 (12)	0.63574 (11)	0.40702 (8)	0.0195 (3)
C3B	0.20171 (14)	0.60575 (13)	0.52084 (9)	0.0262 (4)
H3BA	0.1735	0.6412	0.5558	0.039*
H3BB	0.2764	0.5979	0.5341	0.039*
H3BC	0.1690	0.5424	0.5149	0.039*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11A	0.0370 (3)	0.0591 (3)	0.0472 (3)	0.0132 (2)	0.0127 (2)	0.0337 (3)
C12A	0.0227 (2)	0.0413 (3)	0.0390 (3)	−0.00235 (18)	0.00915 (18)	0.0030 (2)
C13A	0.0477 (3)	0.0276 (2)	0.0371 (3)	−0.0091 (2)	0.0123 (2)	−0.00656 (19)
S1A	0.0242 (2)	0.0362 (3)	0.0220 (2)	−0.00899 (18)	−0.00211 (17)	0.00193 (18)
S2A	0.0311 (2)	0.0216 (2)	0.0251 (2)	0.00138 (17)	0.00954 (17)	−0.00147 (16)

O1A	0.0276 (7)	0.0306 (7)	0.0346 (7)	0.0138 (5)	0.0115 (5)	0.0094 (6)
N1A	0.0233 (7)	0.0235 (7)	0.0258 (7)	0.0083 (6)	0.0063 (6)	0.0027 (6)
C1A	0.0236 (8)	0.0281 (9)	0.0250 (9)	-0.0008 (7)	0.0044 (7)	0.0071 (7)
C2A	0.0216 (8)	0.0180 (8)	0.0250 (8)	-0.0005 (6)	0.0072 (7)	0.0001 (6)
C3A	0.0377 (11)	0.0366 (10)	0.0276 (10)	0.0080 (9)	0.0101 (8)	0.0080 (8)
Cl1B	0.0494 (3)	0.0412 (3)	0.0250 (2)	0.0009 (2)	0.0121 (2)	-0.00839 (19)
Cl2B	0.0380 (3)	0.0494 (3)	0.0303 (2)	0.0055 (2)	-0.0078 (2)	-0.0003 (2)
Cl3B	0.0457 (3)	0.0362 (3)	0.0246 (2)	-0.0184 (2)	0.00170 (19)	0.00053 (18)
S1B	0.0291 (2)	0.0312 (2)	0.0211 (2)	-0.00846 (18)	0.00602 (17)	-0.00414 (17)
S2B	0.0358 (2)	0.0232 (2)	0.0198 (2)	0.00546 (18)	0.00314 (17)	-0.00014 (16)
O1B	0.0289 (6)	0.0262 (6)	0.0270 (6)	0.0102 (5)	0.0051 (5)	-0.0037 (5)
N1B	0.0237 (7)	0.0207 (7)	0.0227 (7)	0.0074 (6)	0.0047 (6)	-0.0002 (6)
C1B	0.0307 (9)	0.0288 (9)	0.0163 (8)	-0.0028 (7)	0.0023 (7)	-0.0016 (7)
C2B	0.0197 (8)	0.0184 (8)	0.0193 (8)	0.0001 (6)	-0.0003 (6)	-0.0023 (6)
C3B	0.0308 (9)	0.0258 (9)	0.0228 (9)	0.0012 (7)	0.0065 (7)	0.0021 (7)

Geometric parameters (Å, °)

Cl1A—C1A	1.7736 (18)	Cl1B—C1B	1.7736 (18)
Cl2A—C1A	1.7625 (18)	Cl2B—C1B	1.7696 (19)
Cl3A—C1A	1.7667 (19)	Cl3B—C1B	1.7629 (18)
S1A—C1A	1.8242 (18)	S1B—C1B	1.8261 (18)
S1A—S2A	2.0100 (7)	S1B—S2B	2.0126 (6)
S2A—C2A	1.8367 (17)	S2B—C2B	1.8426 (17)
O1A—C2A	1.214 (2)	O1B—C2B	1.212 (2)
N1A—C2A	1.322 (2)	N1B—C2B	1.324 (2)
N1A—C3A	1.458 (2)	N1B—C3B	1.460 (2)
N1A—H1AA	0.864 (9)	N1B—H1BA	0.857 (9)
C3A—H3AA	0.9800	C3B—H3BA	0.9800
C3A—H3AB	0.9800	C3B—H3BB	0.9800
C3A—H3AC	0.9800	C3B—H3BC	0.9800
C1A—S1A—S2A	103.09 (6)	C1B—S1B—S2B	103.10 (6)
C2A—S2A—S1A	102.20 (6)	C2B—S2B—S1B	101.43 (6)
C2A—N1A—C3A	121.71 (15)	C2B—N1B—C3B	120.35 (14)
C2A—N1A—H1AA	120.5 (14)	C2B—N1B—H1BA	119.5 (14)
C3A—N1A—H1AA	117.3 (14)	C3B—N1B—H1BA	120.2 (14)
Cl2A—C1A—Cl3A	108.65 (10)	Cl3B—C1B—Cl2B	108.82 (10)
Cl2A—C1A—Cl1A	109.45 (9)	Cl3B—C1B—Cl1B	109.68 (10)
Cl3A—C1A—Cl1A	110.43 (10)	Cl2B—C1B—Cl1B	109.38 (9)
Cl2A—C1A—S1A	113.54 (10)	Cl3B—C1B—S1B	112.65 (9)
Cl3A—C1A—S1A	112.03 (9)	Cl2B—C1B—S1B	112.28 (10)
Cl1A—C1A—S1A	102.60 (9)	Cl1B—C1B—S1B	103.90 (9)
O1A—C2A—N1A	126.31 (16)	O1B—C2B—N1B	126.23 (16)
O1A—C2A—S2A	123.02 (13)	O1B—C2B—S2B	122.17 (13)
N1A—C2A—S2A	110.67 (12)	N1B—C2B—S2B	111.58 (12)
N1A—C3A—H3AA	109.5	N1B—C3B—H3BA	109.5
N1A—C3A—H3AB	109.5	N1B—C3B—H3BB	109.5

H3AA—C3A—H3AB	109.5	H3BA—C3B—H3BB	109.5
N1A—C3A—H3AC	109.5	N1B—C3B—H3BC	109.5
H3AA—C3A—H3AC	109.5	H3BA—C3B—H3BC	109.5
H3AB—C3A—H3AC	109.5	H3BB—C3B—H3BC	109.5
C1A—S1A—S2A—C2A	93.63 (8)	C1B—S1B—S2B—C2B	93.49 (8)
S2A—S1A—C1A—C12A	60.37 (10)	S2B—S1B—C1B—C13B	-60.94 (10)
S2A—S1A—C1A—C13A	-63.18 (10)	S2B—S1B—C1B—C12B	62.34 (9)
S2A—S1A—C1A—C11A	178.38 (6)	S2B—S1B—C1B—C11B	-179.58 (6)
C3A—N1A—C2A—O1A	3.3 (3)	C3B—N1B—C2B—O1B	1.6 (3)
C3A—N1A—C2A—S2A	-176.22 (14)	C3B—N1B—C2B—S2B	-176.67 (12)
S1A—S2A—C2A—O1A	2.87 (16)	S1B—S2B—C2B—O1B	-0.66 (15)
S1A—S2A—C2A—N1A	-177.64 (11)	S1B—S2B—C2B—N1B	177.64 (11)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1A—H1AA...O1B ⁱ	0.86 (1)	1.94 (1)	2.7825 (18)	164 (2)
N1B—H1BA...O1A ⁱⁱ	0.86 (1)	1.97 (1)	2.8231 (18)	175 (2)

Symmetry codes: (i) $-x+1/2, -y+1/2, -z+1$; (ii) $-x, -y+1, -z+1$.**(2) (N-Benzylcarbamoyl)(trichloromethyl)disulfane***Crystal data* $C_9H_8Cl_3NOS_2$ $M_r = 316.63$ Monoclinic, $P2_1/c$ $a = 11.4247$ (17) Å $b = 13.548$ (2) Å $c = 8.5675$ (12) Å $\beta = 103.176$ (2)° $V = 1291.2$ (3) Å³ $Z = 4$ $F(000) = 640$ $D_x = 1.629$ Mg m⁻³

Melting point = 357–359 K

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2312 reflections

 $\theta = 2.4$ – 24.9° $\mu = 1.01$ mm⁻¹ $T = 173$ K

Rod, white

 $0.30 \times 0.15 \times 0.10$ mm*Data collection*Bruker SMART CCD area detector
diffractometer

Radiation source: sealed tube

phi and ω scans

Absorption correction: multi-scan

SADABS, (Sheldrick, 2008)

 $T_{\min} = 0.752, T_{\max} = 0.906$

12180 measured reflections

2284 independent reflections

2056 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.041$ $\theta_{\max} = 25.1^\circ, \theta_{\min} = 1.8^\circ$ $h = -13 \rightarrow 13$ $k = -16 \rightarrow 16$ $l = -10 \rightarrow 10$ *Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.042$ $wR(F^2) = 0.080$ $S = 1.00$

2284 reflections

148 parameters

1 restraint

Hydrogen site location: mixed

H atoms treated by a mixture of independent
and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0157P)^2 + 3.520P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$

$$\Delta\rho_{\max} = 0.33 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.27 \text{ e } \text{\AA}^{-3}$$

Special details

Experimental. Compound (2) (Barany *et al.*, 2005) was synthesized and crystallized as outlined in the Scheme and described in the referenced publication.

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.57223 (8)	0.68834 (7)	0.12990 (11)	0.0399 (2)
Cl2	0.69283 (7)	0.60896 (7)	0.43579 (10)	0.0320 (2)
Cl3	0.64826 (8)	0.48462 (7)	0.15317 (11)	0.0385 (2)
S1	0.44925 (7)	0.53104 (6)	0.27940 (9)	0.02193 (18)
S2	0.37502 (7)	0.64387 (6)	0.37478 (9)	0.0249 (2)
O1	0.26774 (19)	0.66377 (16)	0.0620 (2)	0.0250 (5)
N1	0.2069 (2)	0.7676 (2)	0.2344 (3)	0.0247 (6)
H1A	0.222 (3)	0.785 (2)	0.3348 (16)	0.030*
C1	0.5911 (3)	0.5817 (2)	0.2514 (4)	0.0249 (7)
C2	0.2734 (3)	0.6962 (2)	0.1955 (3)	0.0207 (7)
C3	0.1156 (3)	0.8196 (3)	0.1127 (4)	0.0327 (8)
H2A	0.0778	0.7728	0.0272	0.039*
H2B	0.1544	0.8728	0.0632	0.039*
C4	0.0210 (3)	0.8631 (2)	0.1889 (4)	0.0258 (7)
C5	0.0178 (3)	0.9637 (3)	0.2167 (4)	0.0319 (8)
H5A	0.0737	1.0060	0.1833	0.038*
C6	-0.0662 (3)	1.0032 (3)	0.2928 (5)	0.0374 (9)
H6A	-0.0681	1.0724	0.3102	0.045*
C7	-0.1466 (3)	0.9429 (3)	0.3430 (4)	0.0347 (9)
H7A	-0.2029	0.9701	0.3974	0.042*
C8	-0.1457 (3)	0.8434 (3)	0.3145 (4)	0.0377 (9)
H8A	-0.2023	0.8019	0.3481	0.045*
C9	-0.0627 (3)	0.8028 (3)	0.2371 (4)	0.0333 (8)
H9A	-0.0632	0.7338	0.2171	0.040*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0348 (5)	0.0426 (5)	0.0407 (5)	-0.0039 (4)	0.0055 (4)	0.0208 (4)
Cl2	0.0283 (4)	0.0376 (5)	0.0262 (4)	-0.0033 (4)	-0.0021 (3)	-0.0023 (4)
Cl3	0.0296 (5)	0.0506 (6)	0.0360 (5)	0.0088 (4)	0.0088 (4)	-0.0132 (4)
S1	0.0225 (4)	0.0209 (4)	0.0225 (4)	0.0011 (3)	0.0054 (3)	0.0005 (3)
S2	0.0263 (4)	0.0321 (5)	0.0159 (4)	0.0072 (4)	0.0036 (3)	-0.0006 (3)
O1	0.0283 (12)	0.0321 (13)	0.0143 (11)	0.0036 (10)	0.0043 (9)	-0.0007 (9)

N1	0.0275 (15)	0.0317 (15)	0.0137 (13)	0.0101 (12)	0.0020 (11)	-0.0024 (11)
C1	0.0216 (16)	0.0311 (18)	0.0216 (16)	0.0023 (14)	0.0038 (13)	0.0012 (14)
C2	0.0210 (16)	0.0242 (17)	0.0176 (16)	-0.0015 (13)	0.0059 (12)	0.0018 (13)
C3	0.0319 (19)	0.041 (2)	0.0228 (17)	0.0146 (17)	0.0023 (14)	0.0023 (16)
C4	0.0239 (17)	0.0323 (19)	0.0188 (16)	0.0067 (14)	-0.0004 (13)	0.0013 (14)
C5	0.0242 (18)	0.0318 (19)	0.038 (2)	0.0007 (15)	0.0032 (15)	0.0032 (16)
C6	0.030 (2)	0.0291 (19)	0.052 (2)	0.0074 (16)	0.0059 (17)	-0.0070 (17)
C7	0.0199 (17)	0.048 (2)	0.035 (2)	0.0112 (16)	0.0039 (15)	-0.0053 (17)
C8	0.0246 (18)	0.051 (2)	0.037 (2)	-0.0034 (17)	0.0051 (16)	0.0099 (18)
C9	0.0334 (19)	0.0282 (19)	0.0344 (19)	0.0018 (16)	-0.0006 (16)	-0.0005 (15)

Geometric parameters (Å, °)

Cl1—C1	1.764 (3)	C3—H2B	0.9900
Cl2—C1	1.773 (3)	C4—C5	1.386 (5)
Cl3—C1	1.766 (3)	C4—C9	1.391 (5)
S1—C1	1.826 (3)	C5—C6	1.385 (5)
S1—S2	2.0099 (11)	C5—H5A	0.9500
S2—C2	1.842 (3)	C6—C7	1.369 (5)
O1—C2	1.213 (4)	C6—H6A	0.9500
N1—C2	1.319 (4)	C7—C8	1.370 (5)
N1—C3	1.475 (4)	C7—H7A	0.9500
N1—H1A	0.870 (10)	C8—C9	1.389 (5)
C3—C4	1.504 (4)	C8—H8A	0.9500
C3—H2A	0.9900	C9—H9A	0.9500
C1—S1—S2	103.68 (11)	H2A—C3—H2B	108.2
C2—S2—S1	101.40 (10)	C5—C4—C9	118.7 (3)
C2—N1—C3	121.8 (3)	C5—C4—C3	120.7 (3)
C2—N1—H1A	117 (2)	C9—C4—C3	120.6 (3)
C3—N1—H1A	121 (2)	C6—C5—C4	120.6 (3)
Cl1—C1—Cl3	109.72 (17)	C6—C5—H5A	119.7
Cl1—C1—Cl2	108.81 (18)	C4—C5—H5A	119.7
Cl3—C1—Cl2	109.94 (17)	C7—C6—C5	120.3 (3)
Cl1—C1—S1	113.13 (17)	C7—C6—H6A	119.9
Cl3—C1—S1	102.59 (17)	C5—C6—H6A	119.9
Cl2—C1—S1	112.48 (17)	C6—C7—C8	119.9 (3)
O1—C2—N1	126.4 (3)	C6—C7—H7A	120.1
O1—C2—S2	122.4 (2)	C8—C7—H7A	120.1
N1—C2—S2	111.2 (2)	C7—C8—C9	120.6 (3)
N1—C3—C4	110.0 (3)	C7—C8—H8A	119.7
N1—C3—H2A	109.7	C9—C8—H8A	119.7
C4—C3—H2A	109.7	C8—C9—C4	119.9 (3)
N1—C3—H2B	109.7	C8—C9—H9A	120.0
C4—C3—H2B	109.7	C4—C9—H9A	120.0
C1—S1—S2—C2	96.54 (14)	N1—C3—C4—C9	-72.6 (4)
S2—S1—C1—Cl1	-57.38 (18)	C9—C4—C5—C6	0.8 (5)

S2—S1—C1—C13	-175.51 (11)	C3—C4—C5—C6	-177.4 (3)
S2—S1—C1—C12	66.42 (17)	C4—C5—C6—C7	0.7 (5)
C3—N1—C2—O1	-1.3 (5)	C5—C6—C7—C8	-1.5 (5)
C3—N1—C2—S2	-178.2 (3)	C6—C7—C8—C9	0.9 (5)
S1—S2—C2—O1	-2.5 (3)	C7—C8—C9—C4	0.5 (5)
S1—S2—C2—N1	174.6 (2)	C5—C4—C9—C8	-1.4 (5)
C2—N1—C3—C4	155.8 (3)	C3—C4—C9—C8	176.8 (3)
N1—C3—C4—C5	105.5 (4)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1 <i>A</i> ...O1 ⁱ	0.87 (1)	2.02 (1)	2.887 (3)	174 (3)

Symmetry code: (i) *x*, $-\gamma+3/2$, *z*+1/2.**(3) (*N*-Methyl-*N*-phenylcarbamoyl)(trichloromethyl)disulfane***Crystal data*C₉H₈Cl₃NOS₂ $M_r = 316.63$ Triclinic, $P\bar{1}$ $a = 8.9231$ (12) Å $b = 10.1724$ (13) Å $c = 15.364$ (2) Å $\alpha = 81.964$ (2)° $\beta = 81.806$ (2)° $\gamma = 68.851$ (2)° $V = 1281.5$ (3) Å³ $Z = 4$ $F(000) = 640$ $D_x = 1.641$ Mg m⁻³

Melting point = 327–328 K

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2932 reflections

 $\theta = 2.5$ – 27.4 ° $\mu = 1.02$ mm⁻¹ $T = 123$ K

Plate, colourless

0.25 × 0.20 × 0.09 mm

*Data collection*Bruker SMART CCD area detector
diffractometer

Radiation source: sealed tube

phi and ω scansAbsorption correction: multi-scan
(*SADABS*; Sheldrick, 2008) $T_{\min} = 0.676$, $T_{\max} = 0.746$

15282 measured reflections

5790 independent reflections

4557 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.034$ $\theta_{\text{max}} = 27.5$ °, $\theta_{\text{min}} = 1.4$ ° $h = -11$ →11 $k = -13$ →12 $l = -19$ →19*Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.030$ $wR(F^2) = 0.073$ $S = 0.97$

5790 reflections

291 parameters

0 restraints

Hydrogen site location: inferred from
neighbouring sitesH atoms treated by a mixture of independent
and constrained refinement $w = 1/[\sigma^2(F_o^2) + (0.0294P)^2 + 0.677P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\text{max}} = 0.001$ $\Delta\rho_{\text{max}} = 0.39$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.27$ e Å⁻³

Special details

Experimental. Compound (3) (Barany *et al.*, 1983; Schroll & Barany, 1986) was synthesized and crystallized as outlined in the Scheme and described in the reference publications.

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C11A	0.39735 (6)	0.18646 (6)	0.21843 (4)	0.02947 (13)
C12A	0.74662 (6)	0.06953 (6)	0.18508 (4)	0.02861 (13)
C13A	0.54721 (7)	0.15353 (7)	0.04057 (4)	0.03453 (14)
S1A	0.59372 (6)	0.36579 (6)	0.14069 (4)	0.02254 (12)
S2A	0.37780 (6)	0.50367 (6)	0.10688 (3)	0.02229 (12)
O1A	0.34261 (18)	0.50799 (16)	0.28397 (10)	0.0278 (3)
N1A	0.1137 (2)	0.61782 (18)	0.21527 (11)	0.0216 (4)
C1A	0.5668 (2)	0.1953 (2)	0.14591 (14)	0.0222 (4)
C2A	0.2724 (3)	0.5449 (2)	0.21823 (14)	0.0216 (4)
C3A	0.0124 (3)	0.6590 (3)	0.29789 (15)	0.0330 (5)
H3AA	0.0804	0.6312	0.3468	0.050*
H3AB	-0.0674	0.6115	0.3089	0.050*
H3AC	-0.0433	0.7618	0.2931	0.050*
C4A	0.0386 (2)	0.6638 (2)	0.13409 (13)	0.0202 (4)
C5A	-0.0647 (3)	0.6002 (2)	0.11406 (15)	0.0264 (5)
H5AA	-0.0818	0.5242	0.1525	0.032*
C6A	-0.1425 (3)	0.6478 (3)	0.03785 (16)	0.0300 (5)
H6AA	-0.2142	0.6052	0.0244	0.036*
C7A	-0.1160 (3)	0.7568 (3)	-0.01833 (15)	0.0307 (5)
H7AA	-0.1693	0.7889	-0.0707	0.037*
C8A	-0.0124 (3)	0.8198 (2)	0.00094 (15)	0.0301 (5)
H8AA	0.0066	0.8940	-0.0386	0.036*
C9A	0.0643 (3)	0.7748 (2)	0.07821 (14)	0.0255 (5)
H9AA	0.1332	0.8195	0.0924	0.031*
C11B	-0.02011 (7)	0.26039 (6)	0.25446 (4)	0.03331 (14)
C12B	-0.30856 (6)	0.26958 (5)	0.37202 (4)	0.02562 (12)
C13B	-0.02396 (7)	0.27511 (6)	0.43942 (4)	0.03100 (14)
S1B	-0.04426 (6)	0.01403 (5)	0.38013 (3)	0.02037 (12)
S2B	0.19477 (6)	-0.06161 (6)	0.39221 (3)	0.02144 (12)
O1B	0.19752 (18)	-0.07526 (17)	0.21777 (10)	0.0290 (4)
N1B	0.4406 (2)	-0.17006 (18)	0.27509 (11)	0.0220 (4)
C1B	-0.0950 (2)	0.2053 (2)	0.36108 (14)	0.0213 (4)
C2B	0.2795 (3)	-0.1046 (2)	0.27890 (13)	0.0205 (4)
C3B	0.5317 (3)	-0.2140 (3)	0.19095 (15)	0.0352 (6)
H3BA	0.4622	-0.1715	0.1431	0.053*
H3BB	0.5691	-0.3174	0.1926	0.053*
H3BC	0.6250	-0.1826	0.1807	0.053*

C4B	0.5250 (2)	-0.2155 (2)	0.35376 (13)	0.0191 (4)
C5B	0.5752 (2)	-0.1230 (2)	0.39013 (14)	0.0213 (4)
H5BA	0.5574	-0.0298	0.3626	0.026*
C6B	0.6515 (3)	-0.1679 (2)	0.46693 (14)	0.0241 (5)
H6BA	0.6852	-0.1048	0.4926	0.029*
C7B	0.6789 (2)	-0.3044 (2)	0.50636 (15)	0.0243 (5)
H7BA	0.7295	-0.3344	0.5596	0.029*
C8B	0.6322 (3)	-0.3973 (2)	0.46799 (15)	0.0269 (5)
H8BA	0.6535	-0.4917	0.4943	0.032*
C9B	0.5547 (2)	-0.3531 (2)	0.39146 (15)	0.0245 (5)
H9BA	0.5223	-0.4166	0.3652	0.029*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1A	0.0243 (3)	0.0276 (3)	0.0345 (3)	-0.0101 (2)	0.0015 (2)	0.0022 (2)
Cl2A	0.0235 (3)	0.0256 (3)	0.0300 (3)	0.0005 (2)	-0.0074 (2)	-0.0007 (2)
Cl3A	0.0382 (3)	0.0403 (3)	0.0264 (3)	-0.0111 (3)	-0.0104 (2)	-0.0075 (2)
S1A	0.0169 (3)	0.0241 (3)	0.0257 (3)	-0.0062 (2)	-0.0052 (2)	0.0014 (2)
S2A	0.0195 (3)	0.0243 (3)	0.0189 (3)	-0.0028 (2)	-0.0042 (2)	0.0009 (2)
O1A	0.0284 (8)	0.0337 (9)	0.0205 (8)	-0.0074 (7)	-0.0086 (7)	-0.0031 (7)
N1A	0.0222 (9)	0.0225 (9)	0.0173 (9)	-0.0035 (7)	-0.0029 (7)	-0.0030 (7)
C1A	0.0182 (10)	0.0252 (11)	0.0205 (11)	-0.0041 (9)	-0.0028 (8)	-0.0019 (9)
C2A	0.0244 (11)	0.0194 (10)	0.0215 (11)	-0.0072 (9)	-0.0038 (9)	-0.0026 (8)
C3A	0.0304 (13)	0.0390 (14)	0.0248 (12)	-0.0056 (11)	0.0022 (10)	-0.0100 (10)
C4A	0.0167 (10)	0.0203 (10)	0.0189 (10)	0.0003 (8)	-0.0013 (8)	-0.0054 (8)
C5A	0.0239 (11)	0.0248 (11)	0.0301 (12)	-0.0082 (9)	-0.0023 (9)	-0.0028 (9)
C6A	0.0214 (11)	0.0370 (13)	0.0329 (13)	-0.0081 (10)	-0.0035 (10)	-0.0122 (11)
C7A	0.0214 (11)	0.0432 (14)	0.0200 (11)	0.0001 (10)	-0.0034 (9)	-0.0080 (10)
C8A	0.0284 (12)	0.0304 (12)	0.0255 (12)	-0.0044 (10)	-0.0024 (10)	0.0014 (10)
C9A	0.0238 (11)	0.0241 (11)	0.0273 (12)	-0.0062 (9)	-0.0035 (9)	-0.0028 (9)
Cl1B	0.0299 (3)	0.0319 (3)	0.0323 (3)	-0.0101 (2)	0.0021 (2)	0.0089 (2)
Cl2B	0.0155 (2)	0.0262 (3)	0.0328 (3)	-0.0023 (2)	-0.0045 (2)	-0.0064 (2)
Cl3B	0.0288 (3)	0.0259 (3)	0.0428 (3)	-0.0104 (2)	-0.0143 (2)	-0.0046 (2)
S1B	0.0169 (2)	0.0196 (3)	0.0239 (3)	-0.0055 (2)	-0.0045 (2)	0.0002 (2)
S2B	0.0169 (2)	0.0262 (3)	0.0181 (3)	-0.0022 (2)	-0.0042 (2)	-0.0037 (2)
O1B	0.0285 (8)	0.0374 (9)	0.0212 (8)	-0.0080 (7)	-0.0092 (7)	-0.0065 (7)
N1B	0.0224 (9)	0.0251 (9)	0.0180 (9)	-0.0060 (7)	-0.0013 (7)	-0.0071 (7)
C1B	0.0169 (10)	0.0212 (11)	0.0251 (11)	-0.0058 (8)	-0.0046 (8)	0.0005 (9)
C2B	0.0253 (11)	0.0185 (10)	0.0185 (10)	-0.0073 (9)	-0.0020 (9)	-0.0052 (8)
C3B	0.0332 (13)	0.0463 (15)	0.0239 (12)	-0.0089 (11)	0.0030 (10)	-0.0158 (11)
C4B	0.0149 (10)	0.0209 (10)	0.0196 (10)	-0.0028 (8)	-0.0010 (8)	-0.0053 (8)
C5B	0.0205 (10)	0.0187 (10)	0.0242 (11)	-0.0057 (8)	-0.0015 (8)	-0.0045 (8)
C6B	0.0221 (11)	0.0272 (11)	0.0268 (12)	-0.0113 (9)	-0.0042 (9)	-0.0061 (9)
C7B	0.0163 (10)	0.0279 (11)	0.0270 (12)	-0.0056 (9)	-0.0045 (9)	-0.0005 (9)
C8B	0.0213 (11)	0.0202 (11)	0.0370 (13)	-0.0057 (9)	-0.0047 (10)	0.0017 (10)
C9B	0.0207 (11)	0.0217 (11)	0.0322 (12)	-0.0072 (9)	-0.0018 (9)	-0.0074 (9)

Geometric parameters (Å, °)

C11A—C1A	1.768 (2)	C11B—C1B	1.774 (2)
C12A—C1A	1.776 (2)	C12B—C1B	1.768 (2)
C13A—C1A	1.777 (2)	C13B—C1B	1.771 (2)
S1A—C1A	1.824 (2)	S1B—C1B	1.822 (2)
S1A—S2A	2.0202 (7)	S1B—S2B	2.0160 (7)
S2A—C2A	1.856 (2)	S2B—C2B	1.842 (2)
O1A—C2A	1.208 (2)	O1B—C2B	1.211 (2)
N1A—C2A	1.345 (3)	N1B—C2B	1.346 (3)
N1A—C4A	1.440 (3)	N1B—C4B	1.447 (3)
N1A—C3A	1.467 (3)	N1B—C3B	1.460 (3)
C3A—H3AA	0.9800	C3B—H3BA	0.9800
C3A—H3AB	0.9800	C3B—H3BB	0.9800
C3A—H3AC	0.9800	C3B—H3BC	0.9800
C4A—C9A	1.387 (3)	C4B—C9B	1.384 (3)
C4A—C5A	1.389 (3)	C4B—C5B	1.386 (3)
C5A—C6A	1.385 (3)	C5B—C6B	1.385 (3)
C5A—H5AA	0.9500	C5B—H5BA	0.9500
C6A—C7A	1.376 (3)	C6B—C7B	1.385 (3)
C6A—H6AA	0.9500	C6B—H6BA	0.9500
C7A—C8A	1.383 (3)	C7B—C8B	1.387 (3)
C7A—H7AA	0.9500	C7B—H7BA	0.9500
C8A—C9A	1.393 (3)	C8B—C9B	1.387 (3)
C8A—H8AA	0.9500	C8B—H8BA	0.9500
C9A—H9AA	0.9500	C9B—H9BA	0.9500
C1A—S1A—S2A	102.38 (7)	C1B—S1B—S2B	104.40 (7)
C2A—S2A—S1A	99.96 (7)	C2B—S2B—S1B	101.59 (7)
C2A—N1A—C4A	123.13 (17)	C2B—N1B—C4B	122.00 (17)
C2A—N1A—C3A	118.95 (18)	C2B—N1B—C3B	119.49 (18)
C4A—N1A—C3A	117.85 (17)	C4B—N1B—C3B	118.00 (17)
C11A—C1A—C12A	110.05 (11)	C12B—C1B—C13B	109.89 (11)
C11A—C1A—C13A	108.33 (11)	C12B—C1B—C11B	110.25 (11)
C12A—C1A—C13A	108.62 (11)	C13B—C1B—C11B	107.66 (11)
C11A—C1A—S1A	112.54 (11)	C12B—C1B—S1B	103.07 (10)
C12A—C1A—S1A	104.78 (11)	C13B—C1B—S1B	113.09 (11)
C13A—C1A—S1A	112.43 (11)	C11B—C1B—S1B	112.83 (11)
O1A—C2A—N1A	125.9 (2)	O1B—C2B—N1B	126.4 (2)
O1A—C2A—S2A	122.09 (16)	O1B—C2B—S2B	122.96 (16)
N1A—C2A—S2A	111.99 (15)	N1B—C2B—S2B	110.65 (14)
N1A—C3A—H3AA	109.5	N1B—C3B—H3BA	109.5
N1A—C3A—H3AB	109.5	N1B—C3B—H3BB	109.5
H3AA—C3A—H3AB	109.5	H3BA—C3B—H3BB	109.5
N1A—C3A—H3AC	109.5	N1B—C3B—H3BC	109.5
H3AA—C3A—H3AC	109.5	H3BA—C3B—H3BC	109.5
H3AB—C3A—H3AC	109.5	H3BB—C3B—H3BC	109.5
C9A—C4A—C5A	120.4 (2)	C9B—C4B—C5B	120.95 (19)

C9A—C4A—N1A	120.05 (19)	C9B—C4B—N1B	118.54 (18)
C5A—C4A—N1A	119.51 (19)	C5B—C4B—N1B	120.51 (18)
C6A—C5A—C4A	119.8 (2)	C6B—C5B—C4B	119.38 (19)
C6A—C5A—H5AA	120.1	C6B—C5B—H5BA	120.3
C4A—C5A—H5AA	120.1	C4B—C5B—H5BA	120.3
C7A—C6A—C5A	120.0 (2)	C7B—C6B—C5B	120.23 (19)
C7A—C6A—H6AA	120.0	C7B—C6B—H6BA	119.9
C5A—C6A—H6AA	120.0	C5B—C6B—H6BA	119.9
C6A—C7A—C8A	120.4 (2)	C6B—C7B—C8B	119.9 (2)
C6A—C7A—H7AA	119.8	C6B—C7B—H7BA	120.1
C8A—C7A—H7AA	119.8	C8B—C7B—H7BA	120.1
C7A—C8A—C9A	120.2 (2)	C9B—C8B—C7B	120.3 (2)
C7A—C8A—H8AA	119.9	C9B—C8B—H8BA	119.8
C9A—C8A—H8AA	119.9	C7B—C8B—H8BA	119.8
C4A—C9A—C8A	119.2 (2)	C4B—C9B—C8B	119.2 (2)
C4A—C9A—H9AA	120.4	C4B—C9B—H9BA	120.4
C8A—C9A—H9AA	120.4	C8B—C9B—H9BA	120.4
C1A—S1A—S2A—C2A	92.91 (10)	C1B—S1B—S2B—C2B	-95.23 (10)
S2A—S1A—C1A—C11A	-55.40 (11)	S2B—S1B—C1B—C12B	-169.19 (7)
S2A—S1A—C1A—C12A	-174.96 (8)	S2B—S1B—C1B—C13B	-50.59 (12)
S2A—S1A—C1A—C13A	67.26 (11)	S2B—S1B—C1B—C11B	71.90 (11)
C4A—N1A—C2A—O1A	177.3 (2)	C4B—N1B—C2B—O1B	-172.5 (2)
C3A—N1A—C2A—O1A	0.3 (3)	C3B—N1B—C2B—O1B	-0.8 (3)
C4A—N1A—C2A—S2A	-3.0 (2)	C4B—N1B—C2B—S2B	8.1 (2)
C3A—N1A—C2A—S2A	-179.98 (15)	C3B—N1B—C2B—S2B	179.73 (16)
S1A—S2A—C2A—O1A	10.32 (19)	S1B—S2B—C2B—O1B	6.32 (19)
S1A—S2A—C2A—N1A	-169.40 (14)	S1B—S2B—C2B—N1B	-174.23 (13)
C2A—N1A—C4A—C9A	-72.9 (3)	C2B—N1B—C4B—C9B	93.8 (2)
C3A—N1A—C4A—C9A	104.1 (2)	C3B—N1B—C4B—C9B	-78.0 (3)
C2A—N1A—C4A—C5A	109.7 (2)	C2B—N1B—C4B—C5B	-86.4 (3)
C3A—N1A—C4A—C5A	-73.3 (3)	C3B—N1B—C4B—C5B	101.8 (2)
C9A—C4A—C5A—C6A	0.0 (3)	C9B—C4B—C5B—C6B	-2.1 (3)
N1A—C4A—C5A—C6A	177.36 (19)	N1B—C4B—C5B—C6B	178.12 (19)
C4A—C5A—C6A—C7A	0.8 (3)	C4B—C5B—C6B—C7B	0.7 (3)
C5A—C6A—C7A—C8A	-0.3 (3)	C5B—C6B—C7B—C8B	1.1 (3)
C6A—C7A—C8A—C9A	-1.0 (3)	C6B—C7B—C8B—C9B	-1.6 (3)
C5A—C4A—C9A—C8A	-1.2 (3)	C5B—C4B—C9B—C8B	1.6 (3)
N1A—C4A—C9A—C8A	-178.58 (19)	N1B—C4B—C9B—C8B	-178.57 (19)
C7A—C8A—C9A—C4A	1.7 (3)	C7B—C8B—C9B—C4B	0.2 (3)
