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The Structure of N-(*tert*-Butyldithio)succinimide

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Abstract

$C_8H_{13}NO_2S_2$ is monoclinic, $a = 9.232$ (4), $b = 22.518$ (2), $c = 10.744$ (2) Å, $\beta = 93.81$ (2)°, space group $P2_1/c$, $Z = 8$. The structure was solved by the heavy-atom method and refined to $R = 0.041$. The angles between the planes C—S—S and S—S—N are 91.1 (86.0)°, and the S—S lengths are both 2.009 Å.

Introduction

The structures of N,N'-dithiodisuccinimide and N-(*tert*-butyldithio)phthalimide (Mazhar-ul-Haque & Behforouz, 1974, 1979) are known. The structure of this compound was undertaken to see the effect of the *tert*-butyl group on the S—S length and the angle between the planes C—S—S and N—S—S in comparison with those in N,N'-dithiodisuccinimide and N-(*tert*-butyldithio)phthalimide.

Experimental

Crystal data

$C_8H_{13}NO_2S_2$, $M_r = 219.3$, m.p. $= 387$–388 K, monoclinic, $a = 9.232$ (4), $b = 22.518$ (2), $c = 10.744$ (2) Å, $\beta = 93.81$ (2)°, $U = 2228.6$ Å³, D_m (by flotation) $= 1.31$, $Z = 8$, $D_c = 1.306$ Mg m⁻³. Space group $P2_1/c$; Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å, $\mu = 0.436$ mm⁻¹, $F(000) = 928$.

A crystal $0.25 \times 0.30 \times 0.6$ mm was used for data collection. The program *SEARCH* was used to obtain 25 reflections, which were then used in *INDEX* to obtain approximate cell dimensions. Accurate cell dimensions with their e.s.d.'s were obtained by least squares from these 25 accurately centred reflections.

The intensities of 3550 independent reflections [θ(Mo $K\alpha$) ≤ 25°] were measured on an Enraf–Nonius CAD-4 diffractometer controlled by a PDP 8/A computer at a take-off angle of 5° with Mo $K\alpha$

radiation from a square-graphite-crystal monochromator. Data were measured by the ω–$(\frac{1}{6})2\theta$ scan technique with a *ZIGZAG* routine; background counts were taken on each side of the peak. The intensities of three standard reflections were measured every 90 reflections and showed no significant change. 2237 reflections having $I > 3\sigma(I)$ were considered observed. Form factors for non-hydrogen atoms were from Cromer & Waber (1965), those of H from Stewart, Davidson & Simpson (1965). Anomalous terms for S were included.

All calculations were carried out on the University of Petroleum and Minerals IBM370/158 computer with the NRC crystallographic programs (Ahmed, Hall & Pippy, 1966).

The Patterson map revealed four independent S atoms, and refinement of these positions gave $R = 0.48$. A Fourier map revealed all non-hydrogen atoms of the two independent molecules. Block-diagonal isotropic refinement gave $R = 0.143$, and anisotropic refinement reduced R to 0.066. All H atoms were located from a difference map and further refinement of non-hydrogen atoms anisotropically and H atoms isotropically gave $R = 0.041$.* A weighting scheme (Hughes, 1941) of the form $w = 1$ for $|F_o| \leq 18$, $\sqrt{w} = 18/|F_o|$ for $|F_o| > 18$ was used. A final difference map was flat.

Results and discussion of the structure

Fig. 1 is an *ORTEP* drawing (Johnson, 1976) of two molecules illustrating 30% probability ellipsoids for the

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* Lists of structure factors and anisotropic thermal parameters of the non-hydrogen atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35099 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
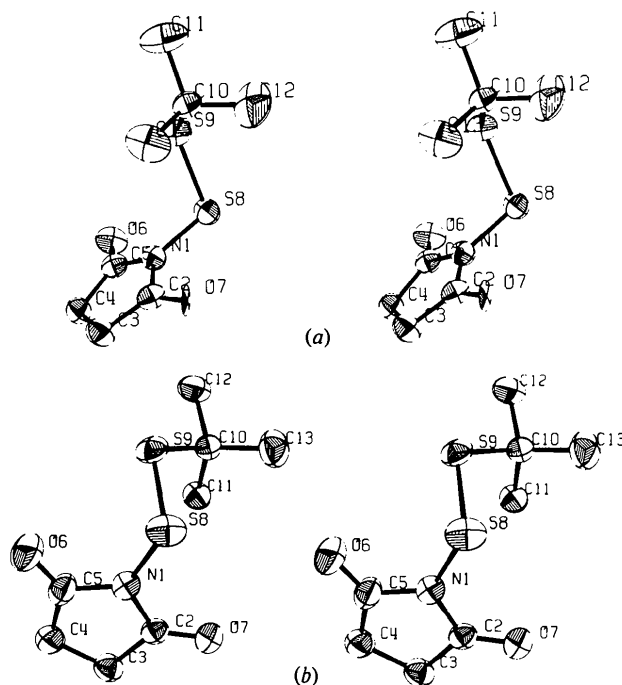

Fig. 1. Stereopairs for molecules 1 and 2 of *N*-(*tert*-butyldithio)-succinimide, showing the atom numbering.

non-hydrogen atoms. Fig. 2 shows the packing of molecules. The atomic coordinates of non-hydrogen and H atoms are listed in Tables 1 and 2 respectively. Bond lengths and angles are given in Table 3. Torsion angles and intermolecular contacts < 3.6 Å are given in Tables 4 and 5 respectively.

It has been shown (Hordvik, 1966) that the bond length between two divalent S atoms varies with the angle between the X–S–S and S–S–Y planes for molecules containing a disulphide group X–S^{II}–S^{II}–Y. The smallest bond lengths correspond to a dihedral angle of *ca* 90°, while the longest correspond to

Table 1. *Fractional coordinates* ($\times 10^4$) *for non-hydrogen atoms with e.s.d.'s in parentheses*

	Molecule 1			Molecule 2		
	x	y	z	x	y	z
N(1)	3706 (3)	1898 (1)	218 (3)	−1075 (3)	889 (1)	1616 (3)
C(2)	2586 (4)	2311 (2)	17 (4)	−2197 (4)	633 (2)	2242 (3)
C(3)	1722 (5)	2138 (2)	−1150 (4)	−3161 (5)	309 (2)	1309 (4)
C(4)	2404 (5)	1579 (2)	−1601 (4)	−2469 (5)	366 (2)	91 (4)
C(5)	3642 (4)	1447 (2)	−678 (4)	−1185 (5)	753 (2)	347 (4)
O(6)	4474 (3)	1039 (1)	−667 (3)	−329 (4)	932 (2)	−357 (3)
O(7)	2397 (4)	2722 (1)	697 (3)	−2331 (3)	685 (1)	3340 (3)
S(8)	4915 (1)	1899 (1)	1498 (1)	201 (1)	1345 (1)	2323 (1)
S(9)	6842 (1)	2134 (1)	884 (1)	2093 (1)	907 (1)	2281 (1)
C(10)	7032 (4)	2949 (2)	996 (4)	2280 (4)	415 (2)	3659 (3)
C(11)	8566 (6)	3052 (2)	599 (7)	1138 (4)	−66 (2)	3571 (4)
C(12)	6912 (8)	3139 (3)	2296 (6)	3798 (5)	137 (3)	3551 (5)
C(13)	5958 (6)	3234 (2)	85 (6)	2198 (6)	758 (2)	4867 (5)

Table 2. *Fractional coordinates* $(\times 10^3)$ *and isotropic temperature factors for* H *atoms with e.s.d.'s in parentheses*

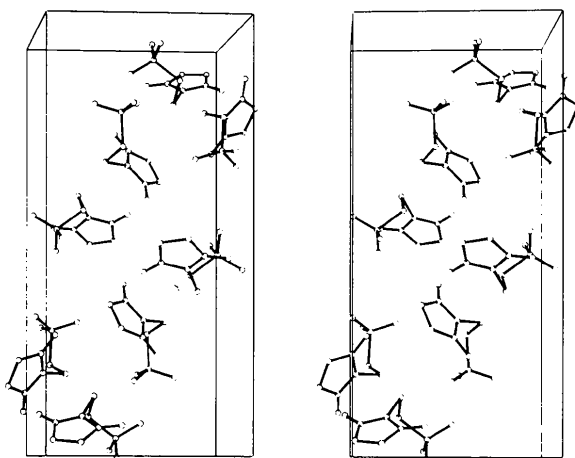|	Molecule 1				Molecule 2			
	x	y	z	B (Å²)	x	y	z	B (Å²)
H(1)[C(3)]	178 (4)	246 (2)	−172 (1)	6·4 (8)	−413 (4)	50 (2)	125 (4)	6·6 (7)
H(2)[C(3)]	76 (5)	208 (2)	−97 (4)	6·5 (9)	−323 (5)	−11 (2)	159 (4)	6·6 (6)
H(3)[C(4)]	272 (4)	161 (2)	−243 (4)	6·2 (8)	−310 (5)	58 (2)	−55 (4)	6·7 (8)
H(4)[C(4)]	174 (4)	123 (2)	−168 (4)	6·7 (7)	−207 (5)	0 (2)	−22 (4)	6·9 (8)
H(5)[C(11)]	916 (5)	284 (2)	114 (4)	7·6 (9)	117 (5)	−25 (2)	282 (4)	7·5 (7)
H(6)[C(11)]	878 (5)	349 (2)	71 (4)	7·8 (8)	18 (4)	9 (2)	367 (4)	7·5 (7)
H(7)[C(11)]	857 (5)	294 (2)	−33 (5)	7·6 (9)	139 (4)	−36 (2)	422 (4)	7·4 (7)
H(8)[C(12)]	699 (5)	349 (2)	234 (4)	7·3 (8)	397 (5)	−20 (2)	418 (4)	7·1 (8)
H(9)[C(12)]	763 (4)	294 (2)	286 (4)	7·8 (9)	438 (5)	44 (2)	357 (4)	7·8 (7)
H(10)[C(12)]	583 (5)	302 (2)	254 (5)	7·5 (5)	375 (5)	−5 (2)	275 (4)	7·6 (7)
H(11)[C(13)]	610 (5)	362 (2)	8 (4)	7·4 (7)	190 (5)	51 (2)	564 (5)	7·3 (8)
H(12)[C(13)]	497 (5)	316 (2)	24 (5)	7·9 (9)	169 (5)	108 (2)	480 (5)	7·8 (9)
H(13)[C(13)]	603 (6)	307 (2)	−76 (5)	7·6 (9)	302 (6)	946 (2)	505 (5)	7·7 (8)

Fig. 2. Stereopair showing the packing of molecules.

smaller dihedral angles. Therefore, one would expect the most favourable dihedral angle for the π bonding in these systems to be 90°. The dihedral angles of 91·1 (86·0)° and the S—S lengths of 2·009 Å fit well with Hordvik's (1966) curve as observed previously for *N*,*N'*-dithiodisuccinimide and *N*-(*tert*-butyldithio)-phthalimide (Mazhar-ul-Haque & Behforouz, 1974, 1979). If the lengths of single and double S—S bonds are taken as 2·08 and 1·88 Å respectively (Pauling, 1960) and it is assumed that a linear relationship exists between bond length and bond order, a π-bond order of 0·355 fits well with Hordvik's (1966) proposed bond length/bond order curve.

The S—N lengths of 1·713 and 1·703 Å in the two independent molecules are shorter than the sum of the Pauling radii (1·74 Å), but agree fairly well with the single-bond lengths 1·688 and 1·709 Å in *N*,*N'*-dithiodisuccinimide and 1·702 Å in *N*-(*tert*-butyldithio)-phthalimide (Mazhar-ul-Haque & Behforouz, 1974,

1979) but even shorter S—N single-bond lengths (1·60–1·67 Å) have been reported (*Molecular Structures and Dimensions*, 1972). The S—C lengths in the two independent molecules, 1·846 and 1·848 Å, are in good agreement with the 1·853 Å in *N*-(*tert*-butyl-dithio)phthalimide, $C(sp^3)$—S values of 1·823 and 1·843 Å (Mellor & Nyburg, 1971) and with the accepted value of 1·812 Å (Pauling, 1960). The mean C—N distances (1·397, 1·396 Å) in the two independent molecules are in good agreement with those in *N*,*N'*-dithiodisuccinimide (1·406 and 1·399 Å) (Mazhar-ul-Haque & Behforouz, 1974), *N*-(*tert*-butyl-dithio)phthalimide (1·414 Å) (Mazhar-ul-Haque & Behforouz, 1979), *N*-chlorosuccinimide (1·39 Å) (Brown, 1961) and succinimide (1·385 Å) (Mason,
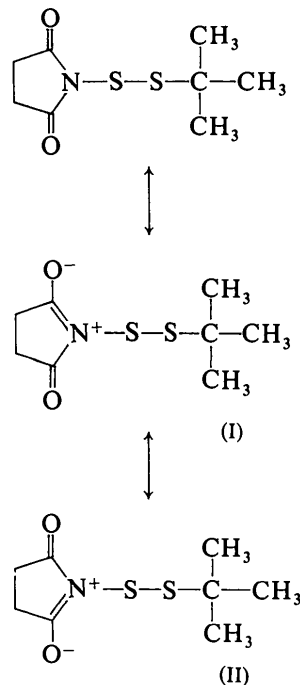
Table 3. *Bond lengths and angles with e.s.d.'s in parentheses*

	Molecule 1	Molecule 2
(a) Distances (Å)		
N(1)–C(2)	1·397 (5)	1·397 (5)
N(1)–C(5)	1·397 (5)	1·395 (5)
N(1)–S(8)	1·713 (3)	1·703 (3)
C(2)–C(3)	1·492 (6)	1·486 (6)
C(2)–O(7)	1·200 (5)	1·200 (5)
C(3)–C(4)	1·501 (6)	1·499 (6)
C(3)–H(1)	0·95 (4)	0·99 (4)
C(3)–H(2)	0·93 (5)	0·99 (4)
C(4)–C(5)	1·492 (6)	1·483 (7)
C(4)–H(3)	0·97 (4)	1·00 (4)
C(4)–H(4)	1·00 (4)	0·97 (5)
C(5)–O(6)	1·199 (5)	1·200 (4)
S(8)–S(9)	2·009 (2)	2·009 (2)
S(9)–C(10)	1·846 (4)	1·848 (4)
C(10)–C(11)	1·524 (7)	1·511 (6)
C(10)–C(12)	1·472 (7)	1·547 (6)
C(10)–C(13)	1·492 (7)	1·517 (6)
C(11)–H(5)	0·91 (5)	0·91 (4)
C(11)–H(6)	1·01 (5)	0·97 (4)
C(11)–H(7)	1·03 (5)	0·98 (4)
C(12)–H(8)	0·82 (5)	1·01 (5)
C(12)–H(9)	0·99 (5)	0·87 (4)
C(12)–H(10)	1.08 (5)	0·96 (4)
C(13)–H(11)	0·89 (5)	1·06 (5)
C(13)–H(12)	0·95 (5)	0·86 (5)
C(13)–H(13)	0·99 (5)	0·87 (5)
(b) Angles (°)		
C(2)–N(1)–C(5)	112·3 (3)	111·8 (2)
C(2)–N(1)–S(8)	124·0 (3)	123·2 (2)
C(5)–N(1)–S(8)	123·4 (2)	124·8 (3)
N(1)–C(2)–C(3)	107·8 (3)	107·9 (3)
N(1)–C(2)–O(7)	123·8 (4)	124·1 (3)
C(3)–C(2)–O(7)	128·5 (4)	128·0 (4)
C(2)–C(3)–C(4)	106·1 (4)	106·1 (3)
C(2)–C(3)–H(1)	107 (2)	109 (2)
C(2)–C(3)–H(2)	110 (3)	108 (3)
C(4)–C(3)–H(1)	113 (2)	110 (2)
C(4)–C(3)–H(2)	112 (3)	113 (3)
H(1)–C(3)–H(2)	110 (4)	111 (4)
C(3)–C(4)–C(5)	105·8 (3)	105·6 (4)
C(3)–C(4)–H(3)	113 (3)	112 (3)
C(3)–C(4)–H(4)	115 (2)	115 (3)
C(5)–C(4)–H(3)	111 (2)	105 (3)
C(5)–C(4)–H(4)	110 (2)	104 (3)
H(3)–C(4)–H(4)	102 (3)	113 (4)
N(1)–C(5)–C(4)	108·0 (3)	108·4 (4)
N(1)–C(5)–O(6)	123·6 (4)	122·3 (4)
C(4)–C(5)–O(6)	128·4 (4)	129·2 (4)
N(1)–S(8)–S(9)	106·3 (1)	105·7 (1)
S(8)–S(9)–C(10)	108·8 (1)	108·1 (1)
S(9)–C(10)–C(11)	102·7 (3)	110·6 (3)
S(9)–C(10)–C(12)	109·8 (3)	102·7 (3)
S(9)–C(10)–C(13)	109·2 (3)	111·9 (3)
C(11)–C(10)–C(12)	110·6 (4)	109·7 (3)
C(11)–C(10)–C(13)	109·9 (4)	110·2 (3)
C(12)–C(10)–C(13)	114·1 (4)	111·6 (4)
C(10)–C(11)–H(5)	106 (3)	108 (3)
C(10)–C(11)–H(6)	107 (3)	112 (2)
C(10)–C(11)–H(7)	107 (4)	108 (2)
H(5)–C(11)–H(6)	110 (4)	110 (4)
H(5)–C(11)–H(7)	117 (4)	108 (4)
H(6)–C(11)–H(7)	110 (4)	110 (3)
C(10)–C(12)–H(8)	109 (3)	111 (3)

Table 3 (cont.)

	Molecule 1	Molecule 2
C(10)–C(12)–H(9)	111 (3)	104 (3)
C(10)–C(12)–H(10)	107 (3)	105 (3)
H(8)–C(12)–H(9)	112 (4)	120 (4)
H(8)–C(12)–H(10)	108 (4)	105 (4)
H(9)–C(12)–H(10)	109 (4)	111 (3)
C(10)–C(13)–H(11)	109 (3)	116 (3)
C(10)–C(13)–H(12)	114 (3)	114 (3)
C(10)–C(13)–H(13)	111 (3)	110 (4)
H(11)–C(13)–H(12)	109 (4)	110 (4)
H(11)–C(13)–H(13)	110 (4)	109 (4)
H(12)–C(13)–H(13)	103 (4)	96 (5)

Table 4. *Torsion angles (°) with e.s.d.'s in parentheses*

	Molecule 1	Molecule 2
N(1)–C(2)–C(3)–C(4)	1·5 (3)	3·1 (3)
N(1)–C(5)–C(4)–C(3)	−1·4 (3)	2·9 (4)
N(1)–S(8)–S(9)–C(10)	91·1 (1)	−86·0 (1)
C(2)–N(1)–C(5)–C(4)	2·5 (3)	−1·0 (3)
C(2)–C(3)–C(4)–C(5)	0·0 (4)	−3·6 (3)
C(2)–N(1)–S(8)–S(9)	−110·5 (1)	114·5 (1)
C(5)–N(1)–C(2)–C(3)	−2·5 (3)	−1·4 (3)
C(5)–N(1)–S(8)–S(9)	76·2 (1)	−69·4 (1)

Table 5. *Intermolecular contacts <3·6 Å involving non-hydrogen atoms*

Atoms*	
C(3)···O(7ᴵ)	3·499 (6)
C(4)···O(7ᴵ)	3·302 (5)
O(6)···C(3ᴵᴵ)	3·369 (5)
O(6)···C(4′ᴵᴵ)	3·258 (6)
O(6)···C(3′ᴵᴵᴵ)	3·322 (5)
O(7)···C(13′ᴵ)	3·537 (6)
S(9)···N(1′ᴵᴵ)	3·461 (3)
C(13)···O(7′ᴵⱽ)	3·510 (6)

* Primed atoms are in molecule 2 and unprimed in molecule 1. Roman numeral superscripts denote the following equivalent positions, relative to the reference molecule at x,y,z.

(I) $x, \frac{1}{2} - y, z - \frac{1}{2}$
(II) $1 + x, y, z$
(III) $\bar{x}, \bar{y}, \bar{z}$
(IV) $1 + x, \frac{1}{2} - y, z - \frac{1}{2}$.

1961). If a $C(sp^3)$–$N(sp^3)$ single bond is taken as 1·47 and a $C(sp^2)$–$N(sp^2)$ double bond as 1·29 Å (Pauling, 1960), and a linear bond length/bond order relationship is assumed, the present C–N bonds possess 40% double-bond character. This is probably due to the contributions of resonance structures (I) and (II).

Mean C=O distances (1·200 and 1·200 Å) in the two independent molecules are identical and compare well with 1·195 and 1·202 Å in N,N'-dithiodisuccinimide, and 1·198 Å in N-(tert-butyldithio)-phthalimide (Mazhar-ul-Haque & Behforouz, 1974,

1979) but are a little shorter than the typical value of 1·22 Å in N-chlorosuccinimide (Brown, 1961) and 1·23 Å in succinimide (Mason, 1961). C(2)—C(3) (1·492, 1·486 Å) and C(4)—C(5) (1·492, 1·483 Å) compare well, and the values are between those for trigonally and tetrahedrally hybridized C atoms. They are in good agreement with 1·492 and 1·493 Å, 1·500 and 1·492 Å found in N,N'-dithiosuccinimide, and 1·479 and 1·476 Å in N-(tert-butyldithio)phthalimide (Mazhar-ul-Haque & Behforouz, 1974, 1979). Such short C—C lengths adjacent to carbonyl groups are quite common (Brown, 1961; Mason, 1961).

The chemically equivalent bond lengths and angles in these three compounds are almost identical as are the torsion angles.

There are no short intermolecular distances (Table 5) and normal van der Waals contacts are observed.

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Struktur von 1,1',2''-Tris(trimethylsilyl)thiophosphoryltrihydrazid

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Abstract

$C_9H_{33}N_6PSSi_3$, triclinic, $P\bar{1}$, $a = 8.867$ (7), $b = 11.456$ (8), $c = 12.486$ (8) Å, $\alpha = 92.51$ (8), $\beta = 109.55$ (9), $\gamma = 112.48$ (9)°, $Z = 2$, $D_c = 1.143$ Mg m^{-3}; $R = 0.061$, $R_w = 0.034$. The title compound is a reaction product of thiophosphoryl trichloride with 1,2-bis(trimethylsilyl)hydrazine. It crystallized from an n-pentane solution containing a mixture of isomers. The molecular structure determined shows that two of the three trimethylsilyl groups have migrated to α N atoms. One is found in the original β position. The two trimethylsilyl-substituted α N atoms have a planar configuration, whereas the third α N atom is pyramidal. The angle Si(3)—N(32)—N(31) of 119·4 (5)° indicates an almost planar configuration at the trimethylsilyl-substituted β N atom. The N—N distances in the hydrazido groups vary from 1·399 (12) to 1·475 (10) Å. The molecule reveals strong intramolecular steric interactions.

Einleitung

Präparative und NMR-spektroskopische Untersuchungen (Engelhardt, Metter & Steger, 1977) hatten vor einiger Zeit ergeben, dass bei der Umsetzung zwischen Thiophosphoryltrichlorid und 1,2-Bis-(trimethylsilyl)hydrazin das erwartete Endprodukt mit drei endständigen Trimethylsilylgruppen nicht in reiner Form entsteht, sondern dass durch Silylgruppen-wanderung bei Raumtemperatur in Lösung Isomere gebildet werden, in denen auch α-Stickstoffatome durch Trimethylsilylgruppen substituiert sind.