Table 2. *Debye–Scherrer diagram of* $BaFeO_{2.67}$, *hexagonal phase, at* 20°C

h k l	d_{obs}	d_{calc}	I_{obs}	I_{calc}
0 0 2		6·958	< 1	< 1
1 0 1	4·635	4·640	3	2
1 0 2	4·023	4·018	15	10
0 0 4		3·479	< 1	1
1 0 3	3·378	3·376	33	40
1 1 0	2·841	2·842	170	100
1 0 4		2·841		70
1 1 2		2·631	< 1	< 1
2 0 1	2·420	2·423	5	3
1 0 5		2·422		4
2 0 2	2·319	2·320	21	10
0 0 6		2·319		6
1 1 4	2·199	2·201	2	3
2 0 3	2·175	2·174	23	22
2 0 4	2·007	2·009	36	33
2 1 1		1·844	< 1	1
2 0 5	1·843	1·8435	13	4
1 0 7		1·843		11
2 1 2	1·798	1·797	11	3
1 1 6		1·796		6
0 0 8		1·739	< 1	1
2 1 3	1·727	1·727	7	9
3 0 0	1·641	1·641	43	17
2 1 4		1·6404		24
1 0 8		1·640		2
2 1 5	1·548	1·547	8	2
2 0 7		1·546		7
1 1 8	1·485	1·487	12	5
1 0 9	1·478	1·479		4
2 0 8	1·420	1·422	19	3
2 2 0		1·419		14

The significant interatomic distances are:

Ba(1)–Ba(2)	3·913 Å	Ba(2)–O(1)	2·824 Å
Ba(1)–Fe(1)	3·479	Ba(2)–O(2)	2·854
Ba(1)–Fe(2)	3·539	Fe(1)–Fe(2)	3·928
Ba(1)–O(1)	2·850	Fe(2)–O(1)	1·945
Ba(2)–Fe(1)	3·549	O(1) –O(2)	2·880

The best final data for the observed and calculated intensities and interplanar spacings are reported in Table 2.

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The Crystal Structure of 1*H*,3*H*-Naphtho[1,8]thiopyrane

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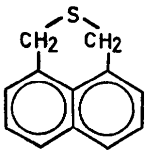
Crystals of 1*H*,3*H*-naphtho[1,8]thiopyrane, $C_{12}H_{10}S$, are monoclinic ($P2_1/c$) with $a=15\cdot2305$, $b=9\cdot0518$, $c=14\cdot6491$ Å and $\beta=108\cdot37°$. There are two molecules in the asymmetric unit. No individual bond lengths deviate by more than three standard deviations from the corresponding average values. The lengths of the differently located bonds in the aromatic system agree well with those reported earlier for the naphthalene nucleus. The hetero-atom is 0·94 Å out of the plane of the rest of the molecule.

Introduction

In connexion with studies on ring systems containing sulphur and selenium atoms Biezais-Zirnis & Fredga (1971) prepared some compounds with the hetero-atoms attached to a 1,8-dimethylnaphthalene nucleus. The crystal structure of 1*H*,4*H*-naphtho[1,8]diselenepine was earlier determined at this research unit (Aleby, 1972). In this connexion it was also considered desirable to perform an X-ray analysis of 1*H*,3*H*-naphtho-[1,8]thiopyrane (NTP, I).



(I)

Experimental

Recrystallization of NTP from petroleum spirit gave colourless crystals with a rhombic shape. The melting point was 96–97°C. Preliminary cell dimensions were obtained from Weissenberg photographs, which also showed the space group to be $P2_1/c$. The data collection was performed on a Picker FACS-I automatic four-circle diffractometer with graphite monochromated Cu $K\alpha$ radiation with a crystal mounted along the b axis. The $\theta/2\theta$ scanning mode was used with a scan speed of 1° per min. The background level was determined by 10 sec counts on each side of the reflexion. A total of 3425 reflexions were measured $2\theta \leq 125°$. 2699 of these had an intensity $I \geq 2\sigma(I)$ with and were used in the structure analysis. The Lorentz and polarization factors were applied but no absorption ($\mu = 24{\cdot}3$ cm^{-1}) or extinction corrections were made.

Table 1. *Fractional atomic coordinates and hydrogen atom isotropic thermal parameters*

The estimated standard deviations are multiplied by 10^4 for C and S, and for H by 10^3. For the hydrogen atoms the first appended number refers to that of the parent atom.

	x	y	z
C(1)	0·4394 (2)	0·2712 (4)	−0·0574 (2)
S(2)	0·3369 (1)	0·2719 (1)	−0·0217 (1)
C(3)	0·3587 (2)	0·0922 (4)	0·0335 (2)
C(3a)	0·4491 (2)	0·0841 (3)	0·1117 (2)
C(4)	0·4567 (3)	−0·0033 (4)	0·1922 (3)
C(5)	0·5412 (3)	−0·0169 (4)	0·2657 (2)
C(6)	0·6165 (3)	0·0559 (4)	0·2617 (2)
C(6a)	0·6132 (2)	0·1477 (3)	0·1827 (2)
C(7)	0·6923 (2)	0·2270 (4)	0·1772 (3)
C(8)	0·6864 (2)	0·3126 (4)	0·1003 (3)
C(9)	0·6039 (2)	0·3259 (4)	0·0256 (3)
C(9a)	0·5262 (2)	0·2535 (3)	0·0264 (2)
C(9b)	0·5278 (2)	0·1607 (3)	0·1060 (2)
C(1′)	−0·0105 (2)	0·2896 (3)	−0·0456 (2)
S(2′)	−0·1256 (1)	0·3300 (1)	−0·0433 (1)
C(3′)	−0·1470 (2)	0·1466 (3)	−0·0079 (2)
C(3′a)	−0·0770 (2)	0·0998 (3)	0·0858 (2)
C(4′)	−0·1062 (2)	0·0148 (3)	0·1482 (2)
C(5′)	−0·0441 (3)	−0·0338 (3)	0·2353 (2)
C(6′)	0·0463 (2)	0·0011 (3)	0·2595 (2)
C(6′a)	0·0806 (2)	0·0909 (3)	0·1982 (2)
C(7′)	0·1737 (2)	0·1320 (4)	0·2239 (2)
C(8′)	0·2046 (2)	0·2202 (4)	0·1654 (3)
C(9′)	0·1441 (2)	0·2706 (3)	0·0785 (2)
C(9′a)	0·0519 (2)	0·2329 (3)	0·0489 (2)
C(9′b)	0·0175 (2)	0·1408 (3)	0·1089 (2)

Table 1 (*cont.*)

	x	y	z	B
H(11)	0·432 (2)	0·182 (4)	−0·104 (3)	5·5
H(12)	0·438 (2)	0·362 (4)	−0·090 (2)	5·5
H(31)	0·359 (2)	0·011 (4)	−0·015 (2)	4·5
H(32)	0·303 (2)	0·071 (4)	0·061 (2)	4·5
H(4)	0·399 (2)	−0·054 (4)	0·191 (2)	5·5
H(5)	0·550 (2)	−0·080 (4)	0·324 (3)	6·2
H(6)	0·678 (2)	0·051 (4)	0·315 (3)	6·1
H(7)	0·749 (2)	0·214 (4)	0·233 (3)	5·6
H(8)	0·746 (2)	0·367 (4)	0·096 (3)	6·3
H(9)	0·602 (2)	0·393 (4)	−0·032 (2)	4·9
H(11′)	0·017 (2)	0·380 (4)	−0·062 (2)	4·5
H(12′)	−0·013 (2)	0·219 (4)	−0·097 (2)	4·5
H(31′)	−0·146 (2)	0·075 (4)	−0·062 (2)	4·2
H(32′)	−0·210 (2)	0·152 (4)	0·001 (2)	4·2
H(4′)	−0·179 (2)	−0·008 (4)	0·129 (2)	4·7
H(5′)	−0·069 (2)	−0·092 (4)	0·281 (2)	5·2
H(6′)	0·091 (2)	−0·034 (4)	0·321 (2)	4·8
H(7′)	0·217 (2)	0·092 (4)	0·287 (2)	4·9
H(8′)	0·270 (2)	0·247 (4)	0·188 (2)	5·3
H(9′)	0·165 (2)	0·332 (4)	0·035 (2)	4·7

Table 2. *Anisotropic thermal parameters in the form*
$\exp\,[-2\pi^2(h^2a^{*2}U_{11}+k^2b^{*2}U_{22}+l^2c^{*2}U_{33}+2klb^*c^*U_{23}+2lha^*c^*U_{31}+2hka^*b^*U_{12})]$

Standard deviations are given in parentheses. All values have been multiplied by 10^4.

	U_{11}	U_{22}	U_{33}	U_{23}	U_{31}	U_{12}
C(1)	810 (21)	739 (21)	485 (16)	74 (15)	196 (15)	168 (17)
S(2)	545 (4)	810 (6)	644 (5)	−75 (4)	23 (4)	190 (4)
C(3)	509 (16)	663 (20)	869 (22)	−237 (17)	224 (15)	−104 (15)
C(3a)	613 (16)	406 (14)	567 (15)	−70 (12)	278 (13)	−9 (12)
C(4)	1114 (27)	542 (19)	842 (23)	−25 (17)	589 (21)	−74 (18)
C(5)	1496 (36)	686 (22)	537 (18)	128 (17)	362 (21)	215 (23)
C(6)	1184 (29)	791 (24)	448 (16)	−28 (16)	24 (17)	345 (22)
C(6a)	601 (17)	615 (18)	503 (15)	−178 (14)	87 (13)	106 (14)
C(7)	478 (17)	940 (26)	897 (24)	−431 (21)	48 (16)	15 (17)
C(8)	657 (20)	923 (27)	1155 (30)	−392 (23)	427 (21)	−271 (19)
C(9)	885 (23)	563 (19)	872 (23)	−99 (17)	497 (19)	−113 (17)
C(9a)	578 (16)	455 (15)	511 (15)	−63 (12)	255 (13)	0 (13)
C(9b)	468 (14)	403 (13)	395 (12)	−75 (11)	137 (11)	18 (11)
C(1′)	880 (22)	580 (18)	463 (15)	87 (13)	255 (15)	15 (16)
S(2′)	727 (5)	507 (4)	540 (4)	39 (3)	0 (4)	130 (4)
C(3′)	497 (15)	539 (17)	624 (17)	−101 (14)	72 (13)	−21 (13)
C(3′a)	534 (15)	355 (13)	493 (14)	−72 (11)	185 (12)	−18 (12)
C(4′)	744 (19)	481 (16)	774 (20)	−27 (15)	382 (16)	−113 (15)
C(5′)	1093 (26)	537 (18)	686 (19)	91 (15)	513 (19)	28 (18)
C(6′)	1024 (23)	514 (17)	402 (14)	62 (13)	228 (15)	174 (17)
C(6′a)	663 (17)	414 (14)	420 (13)	−75 (12)	110 (12)	68 (13)
C(7′)	590 (17)	650 (20)	630 (18)	−140 (15)	−13 (14)	93 (15)
C(8′)	456 (16)	695 (21)	983 (24)	−258 (19)	118 (16)	−47 (15)
C(9′)	647 (18)	494 (17)	855 (21)	−106 (15)	387 (16)	−102 (14)
C(9′a)	556 (15)	369 (13)	476 (14)	−45 (11)	239 (12)	−19 (12)
C(9′b)	503 (13)	332 (12)	351 (12)	−52 (10)	145 (10)	11 (11)

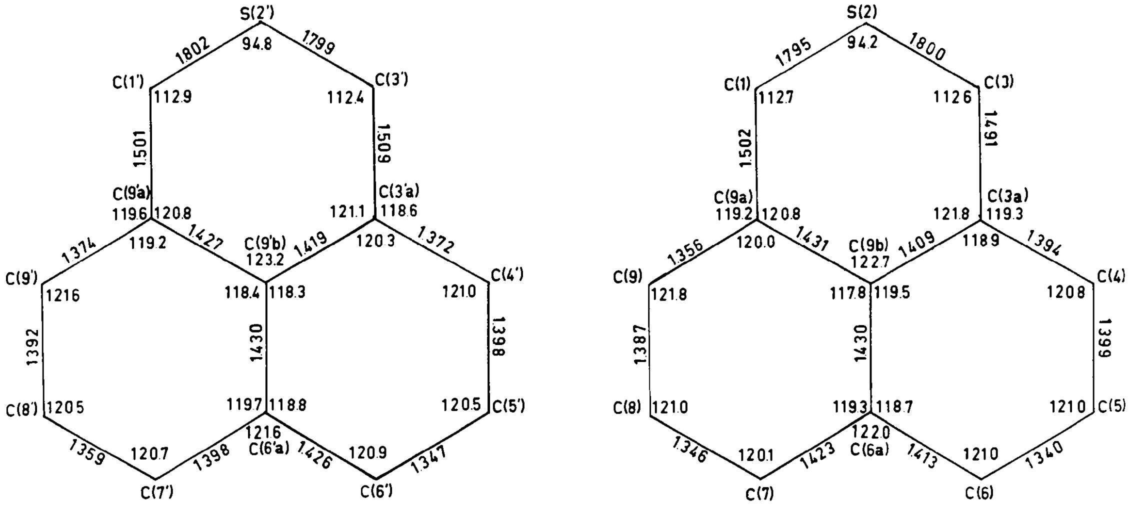


Fig. 1. Distances and angles of NTP.

Crystal data

$C_{12}H_{10}S$ 1*H*,3*H*-naphtho[1,8]thiopyrane.
Monoclinic: $a = 15{\cdot}2305\ (16)$, $b = 9{\cdot}0518\ (14)$, $c = 14{\cdot}6491\ (22)$ Å, $\beta = 108{\cdot}37\ (1)°$
$U = 1912{\cdot}7$ Å^3
$Z = 8$
$\varrho_c = 1{\cdot}3$ g cm^{-3}
$\lambda = 1{\cdot}54051$ Å (Cu $K\alpha_1$).

Structure determination

The sulphur atom coordinates were determined from a sharpened Patterson series. The remaining non-hydrogen atoms were then located from a sulphur-phased Fourier synthesis. After three cycles of block-diagonal refinement the *R* value was 0·20. Another two cycles with anisotropic refinement reduced *R* to 0·10. A difference series calculated at this stage defined very well all hydrogen-atom positions. In the following calculations the hydrogen atoms were given isotropic temperature factors corresponding to those of the parent atoms. The *B* values were not varied. Owing to the relatively large number of reflexions and parameters, the hydrogen atoms and other atoms were refined separately in alternating cycles. When none of the shifts for the non-hydrogen atoms were greater than 0·002 and 0·03 Å for the hydrogen atoms the refinement was terminated. The final *R* value was 0·050.

The form factors applied were those given in *International Tables for X-ray Crystallography* (1962) except for hydrogen where the values of Stewart, Davidson & Simpson (1965) were used. All calculations were performed on a Datasaab D21-PDP15 dual computer system with a program system developed at this laboratory. The weight assigned to each observation in the least-squares refinement was (Mills & Rollett, 1961)

$$\omega = \frac{1}{1 + [(|F_o| - 5{\cdot}1F_{\min})/11{\cdot}6F_{\min}]^2}.$$

Table 3. *Bond distances and angles*

(*a*) Involving non-hydrogen atoms
The estimated standard deviations for the distances are multiplied by 10^4, for the angles by 10.

C(1)—S(2)	1·795 (38) Å	C(1)—S(2)—C(3)	94·2 (2)°
C(1)—C(9a)	1·502 (35)	C(1)—C(9a)–C(9)	119·2 (3)
S(2)—C(3)	1·800 (34)	C(1)—C(9a)–C(9b)	120·8 (3)
C(3)—C(3a)	1·491 (35)	S(2)—C(1)—C(9a)	112·7 (2)
C(3a)–C(4)	1·394 (45)	S(2)—C(3)—C(3a)	112·6 (2)
C(3a)–C(9b)	1·409 (39)	C(3)—C(3a)–C(4)	119·3 (3)
C(4)—C(5)	1·399 (48)	C(3)—C(3a)–C(9b)	121·8 (3)
C(5)—C(6)	1·340 (62)	C(3a)–C(4)—C(5)	120·8 (4)
C(6)—C(6a)	1·413 (46)	C(3a)–C(9b)–C(6a)	119·5 (2)
C(6a)–C(7)	1·427 (47)	C(3a)–C(9b)–C(9a)	122·7 (2)
C(6a)–C(9b)	1·430 (32)	C(4)—C(3a)–C(9b)	118·9 (2)
C(7)—C(8)	1·346 (58)	C(4)—C(5)—C(6)	121·0 (3)
C(8)—C(9)	1·387 (44)	C(5)—C(6)—C(6a)	121·0 (3)
C(9)—C(9a)	1·356 (47)	C(6)—C(6a)–C(7)	122·0 (3)
C(9a)–C(9b)	1·431 (37)	C(6)—C(6a)–C(9b)	118·7 (3)
		C(6a)–C(7)—C(8)	120·1 (3)
C(1′)—S(2′)	1·802 (36)	C(6a)–C(9b)–C(9a)	117·8 (2)
C(1′)—C(9′a)	1·501 (34)	C(7)—C(6a)–C(9b)	119·3 (3)
S(2′)—C(3′)	1·799 (31)	C(7)—C(8)—C(9)	121·0 (4)
C(3′)—C(3′a)	1·509 (34)	C(8)—C(9)—C(9a)	121·8 (3)
C(3′a)–C(4′)	1·372 (45)	C(9)—C(9a)–C(9b)	120·0 (2)
C(3′a)–C(9′b)	1·419 (35)		
C(4′)—C(5′)	1·398 (41)	C(1′)—S(2′)—C(3′)	94·8 (1)
C(5′)—C(6′)	1·347 (50)	C(1′)—C(9′a)–C(9′)	119·6 (3)
C(6′)—C(6′a)	1·426 (44)	C(1′)—C(9′a)–C(9′b)	121·3 (3)
C(6′a)–C(7′)	1·398 (41)	S(2′)—C(1′)—C(9′a)	112·9 (2)
C(6′a)–C(9′b)	1·430 (30)	S(2′)—C(3′)—C(3′a)	112·4 (2)
C(7′)—C(8′)	1·359 (52)	C(3′)—C(3′a)–C(4′)	118·6 (2)
C(8′)—C(9′)	1·392 (42)	C(3′)—C(3′a)–C(9′b)	121·1 (2)
C(9′)—C(9′a)	1·374 (39)	C(3′a)–C(4′)—C(5′)	121·0 (3)
C(9′a)–C(9′b)	1·427 (38)	C(3′a)–C(9′b)–C(6′a)	118·3 (2)
		C(3′a)–C(9′b)–C(9′a)	123·2 (2)
		C(4′)—C(3′a)–C(9′b)	120·3 (2)
		C(4′)—C(5′)—C(6′)	120·5 (3)
		C(5′)—C(6′)—C(6′a)	120·9 (3)
		C(6′)—C(6′a)–C(7′)	121·6 (2)
		C(6′)—C(6′a)–C(9′b)	118·8 (3)
		C(6′a)–C(7′)—C(8′)	120·7 (3)
		C(6′a)–C(9′b)–C(9′a)	118·4 (2)
		C(7′)—C(6′a)–C(9′b)	119·7 (3)
		C(7′)—C(8′)—C(9′)	120·5 (3)
		C(8′)—C(9′)—C(9′a)	121·6 (3)
		C(9′)—C(9′a)–C(9′b)	119·2 (2)

Table 3 (*cont.*)

(*b*) Involving hydrogen atoms.
The estimated standard deviations for the distances are multiplied by 10^2.

C(1)–H(11)	1·04 (4) Å	S(2)—C(1)–H(11)	105·0 (2)°
C(1)–H(12)	0·92 (4)	S(2)—C(1)–H(12)	102·2 (2)
C(3)–H(31)	1·01 (3)	C(9a)—C(1)–H(11)	109·7 (2)
C(3)–H(32)	1·04 (4)	C(9a)—C(1)–H(12)	112·6 (2)
		H(11)–C(1)–H(12)	114·2 (3)
C(4)–H(4)	0·96 (4)	S(2)—C(3)–H(31)	112·0 (2)
C(5)–H(5)	0·97 (4)	S(2)—C(3)–H(32)	105·7 (2)
C(6)–H(6)	1·01 (3)	C(3a)—C(3)–H(31)	106·0 (2)
C(7)–H(7)	0·97 (3)	C(3a)—C(3)–H(32)	110·8 (2)
C(8)–H(8)	1·02 (4)	H(31)–C(3)–H(32)	109·7 (3)
C(9)–H(9)	1·00 (4)	C(3a)—C(4)–H(4)	114·2 (2)
C(1′)–H(11′)	0·97 (3)	C(5)—C(4)–H(4)	124·9 (2)
C(1′)–H(12′)	0·98 (3)	C(4)—C(5)–H(5)	122·7 (2)
C(3′)–H(31′)	1·00 (3)	C(6)—C(5)–H(5)	116·3 (2)
C(3′)–H(32′)	0·97 (3)	C(5)—C(6)–H(6)	124·3 (2)
C(4′)–H(4′)	1·05 (3)	C(6a)—C(6)–H(6)	114·7 (2)
C(5′)–H(5′)	1·01 (4)	C(6a)—C(7)–H(7)	115·1 (2)
C(6′)–H(6′)	0·95 (3)	C(8)—C(7)–H(7)	124·8 (2)
C(7′)–H(7′)	1·00 (3)	C(7)—C(8)–H(8)	118·9 (2)
C(8′)–H(8′)	0·97 (3)	C(9)—C(8)–H(8)	120·0 (2)
C(9′)–H(9′)	0·96 (4)	C(8)—C(9)–H(9)	119·1 (2)
		C(9a)—C(9)–H(9)	119·0 (2)
		S(2′)—C(1′)–H(11′)	108·8 (2)
		S(2′)—C(1′)–H(12′)	110·0 (2)
		C(9′a)–C(1′)–H(11′)	108·4 (2)
		C(9′a)–C(1′)–H(12′)	110·3 (2)
		H(11′)–C(1′)–H(12′)	106·3 (3)
		S(2′)—C(3′)–H(31′)	108·4 (2)
		S(2′)—C(3′)–H(32′)	103·2 (2)
		C(3′a)–C(3′)–H(31′)	111·3 (2)
		C(3′a)–C(3′)–H(32′)	110·6 (2)
		H(31′)–C(3′)–H(32′)	110·6 (3)
		C(3′a)–C(4′)–H(4′)	116·4 (2)
		C(5′)—C(4′)–H(4′)	122·5 (2)
		C(4′)—C(5′)–H(5′)	119·1 (2)
		C(6′)—C(5′)–H(5′)	120·3 (2)
		C(5′)—C(6′)–H(6′)	121·9 (2)
		C(6′a)–C(6′)–H(6′)	117·1 (2)
		C(6′a)–C(7′)–H(7′)	118·1 (2)
		C(8′)—C(7′)–H(7′)	121·2 (2)
		C(7′)—C(8′)–H(8′)	117·8 (2)
		C(9′)—C(8′)–H(8′)	121·7 (2)
		C(8′)—C(9′)–H(9′)	121·9 (2)
		C(9′a)–C(9′)–H(9′)	116·4 (2)

Table 4. *Least-squares planes in the molecule*

The equations are expressed in terms of the crystal axes.

I $0{\cdot}50691X+0{\cdot}55706Y+0{\cdot}65782Z+0{\cdot}10822=0$

II $0{\cdot}44356X+0{\cdot}61128Y+0{\cdot}65543Z-0{\cdot}15132=0.$

Deviations

I		II	
C(1)	−0·014	C(1′)	0·006
C(3)	−0·003	C(3′)	−0·020
C(3a)	0·011	C(3′a)	0·001
C(4)	0·017	C(4′)	0·024
C(5)	−0·010	C(5′)	0·022
C(6)	−0·012	C(6′)	−0·013
C(6a)	−0·002	C(6′a)	−0·019
C(7)	0·003	C(7′)	−0·012
C(8)	0·004	C(8′)	0·012
C(9)	0·006	C(9′)	0·020
C(9a)	0·001	C(9′a)	0·000
C(9b)	−0·001	C(9′b)	−0·020
*S(2)	0·947	*S(2′)	0·942

* Atoms not included in the plane calculation.

Results

A table of the final structure factors can be obtained from this laboratory.* The atomic parameters are given in Tables 1 and 2 and distances and angles in Fig. 1, where also the atomic numbering is indicated, and with standard deviations in Table 3. From a comparison of the differences in individual bond lengths and angles of the two independent molecules of the asymmetric unit it appears likely that the standard deviations are

Table 5. *Intermolecular van der Waals contacts*

i, ii, iii and iiii refer to the equivalent positions x,y,z; $\bar{x},\bar{y},\bar{z}$; $\bar{x},\frac{1}{2}+y,\frac{1}{2}-z$; $x,\frac{1}{2}-y,\frac{1}{2}+z$.

A	*B*	Unit translation on atom *B*	
S(2)	C(7$'^{\rm iiii}$)	(0, 0, −1)	3·87 Å
S(2)	C(8$^{\rm ii}$)	(1, 1, 0)	3·92
S(2)	C(9$^{\rm ii}$)	(1, 1, 0)	3·76
S(2′)	C(9$'^{\rm ii}$)	(0, 1, 0)	3·65
S(2)	H(5$^{\rm iii}$)	(1, 0, 0)	3·20
S(2)	H(8$^{\rm ii}$)	(1, 1, 0)	3·57
S(2)	H(9$^{\rm ii}$)	(1, 1, 0)	3·20
S(2)	H(4$'^{\rm ii}$)	(0, 0, 0)	3·42
S(2)	H(7$'^{\rm iiii}$)	(0, 0, −1)	3·09
S(2′)	H(12$^{\rm ii}$)	(0, 1, 0)	3·23
S(2′)	H(6$^{\rm iiii}$)	(−1, 0, −1)	3·24
S(2′)	H(7$^{\rm iiii}$)	(−1, 0, −1)	3·28
S(2′)	H(8$^{\rm i}$)	(−1, 0, 0)	3·26
S(2′)	H(5$'^{\rm iiii}$)	(0, 0, −1)	3·80
S(2′)	H(9$'^{\rm ii}$)	(0, 1, 0)	3·10
C(3a)	H(11$^{\rm ii}$)	(1, 0, 0)	3·04
C(3a)	H(5$^{\rm iii}$)	(1, 0, 0)	3·21
C(6a)	H(11$^{\rm ii}$)	(1, 0, 0)	3·19
C(6a)	H(31$^{\rm ii}$)	(1, 0, 0)	3·00
C(6a)	H(5$^{\rm iii}$)	(1, 0, 0)	3·49
C(9a)	H(31$^{\rm ii}$)	(1, 0, 0)	3·00
C(9a)	H(5$^{\rm iii}$)	(1, 0, 0)	3·19
C(9b)	H(11$^{\rm ii}$)	(1, 0, 0)	3·16
C(9b)	H(31$^{\rm ii}$)	(1, 0, 0)	2·93
C(9b)	H(5$^{\rm iii}$)	(1, 0, 0)	2·98
C(3′a)	H(12$'^{\rm ii}$)	(0, 0, 0)	3·18
C(3′a)	H(5$'^{\rm iii}$)	(0, 0, 0)	3·71
C(6′a)	H(12$'^{\rm ii}$)	(0, 0, 0)	3·19
C(6′a)	H(31$'^{\rm ii}$)	(0, 0, 0)	2·94
C(6′a)	H(5$'^{\rm iii}$)	(0, 0, 0)	2·90
C(9′a)	H(31$'^{\rm ii}$)	(0, 0, 0)	3·14
C(9′a)	H(5$'^{\rm iii}$)	(0, 0, 0)	2·90
C(9′b)	H(12$'^{\rm ii}$)	(0, 0, 0)	3·26
C(9′b)	H(31$'^{\rm ii}$)	(0, 0, 0)	3·03
C(9′b)	H(5$'^{\rm iii}$)	(0, 0, 0)	2·90
H(12)	H(9$^{\rm ii}$)	(1, 1, 0)	2·97
H(9)	H(5$^{\rm iiii}$)	(0, 0, −1)	2·67
H(9)	H(6$^{\rm iiii}$)	(0, 0, −1)	2·94
H(32′)	H(8$^{\rm i}$)	(−1, 0, 0)	2·59
H(4′)	H(31$'^{\rm ii}$)	(0, 0, 0)	2·76
H(4′)	H(7$^{\rm i}$)	(−1, 0, 0)	2·97
H(5′)	H(11$'^{\rm iiii}$)	(0, 0, 0)	2·97
H(6′)	H(11$'^{\rm iiii}$)	(0, 0, 0)	2·73
H(6′)	H(8$^{\rm iii}$)	(1, −1, 0)	2·61
H(7′)	H(8$^{\rm iii}$)	(1, −1, 0)	2·65

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* The table has also been deposited with the National Lending Library, England, as Supplementary Publication No. 30069. Copies may be obtained through the Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

somewhat underestimated. However, no individual bond or angle differs more than three standard deviations from the corresponding average value for the two molecules. The average C–S distance is 1·799 Å (σ=0·004 Å) and the average C–S–C angle is 94·50° (σ=0·15°). Abrahamsson & Rehnberg (1972) report a value of 1·806 Å for the C–S bond in 1,5-dihydroxy-3-sulphinyl-7-thia-9-oxabicyclo[3,3,1]nonane. The $C(sp)^3$–$C(sp)^2$ bonds in NTP have an average value of 1·501 Å (σ=0·004 Å). The average values for the diferently located bonds in the naphthalene ring agree very well with those reported by Aleby (1972). The value for the bonds of type C(9a)–C(9b) is 1·419 Å (as compared to Aleby's figure of 1·430 Å), for C(9)–C(9a) 1·361 Å (1·359), for C(8)–C(9) 1·394 Å (1·389) and for C(6a)–C(9a) 1·430 Å (1·424). The average valence angle in the naphthalene ring system is 121·0° (σ=0·3°). The mean C–H bond distance is 0·990 Å (σ=0·035 Å) which is a normal X-ray analysis value.

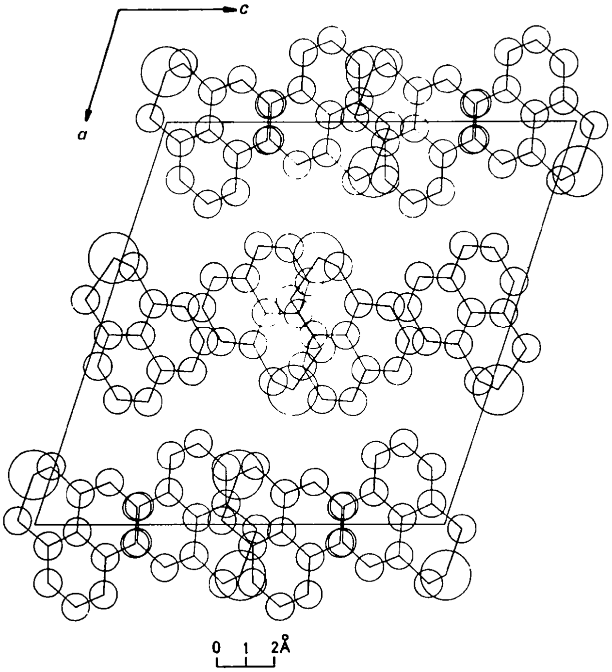

Fig. 2. Molecular packing of NTP as seen along the *b* axis.

The molecules are planar within 0·025 Å (Table 4), except for the sulphur atoms which are out of plane by 0·94 Å. The C–S bonds form an angle of 31° with the naphthalene plane.

The molecular packing is illustrated in Fig. 2 where the hydrogen atoms have been excluded for clarity and by a stereoscopic drawing in Fig. 3. The two molecules of the asymmetric unit are almost parallel, the angle between the plane normals being 4·5°. They pack with each other with the direct contacts only between hydrogen atoms and hydrogen free carbon atoms (Table 5). The angles between molecules related by the twofold screw axis are 77·4 and 70·3°. In this case there are close contacts of both H···H and C···H type. Table 5 also shows the packing environment of the sulphur atoms with the shortest S···H contact of 3·09 and 3·65 Å for S–C. Fig. 4 shows the thermal ellipsoids as viewed along the *b* axis.

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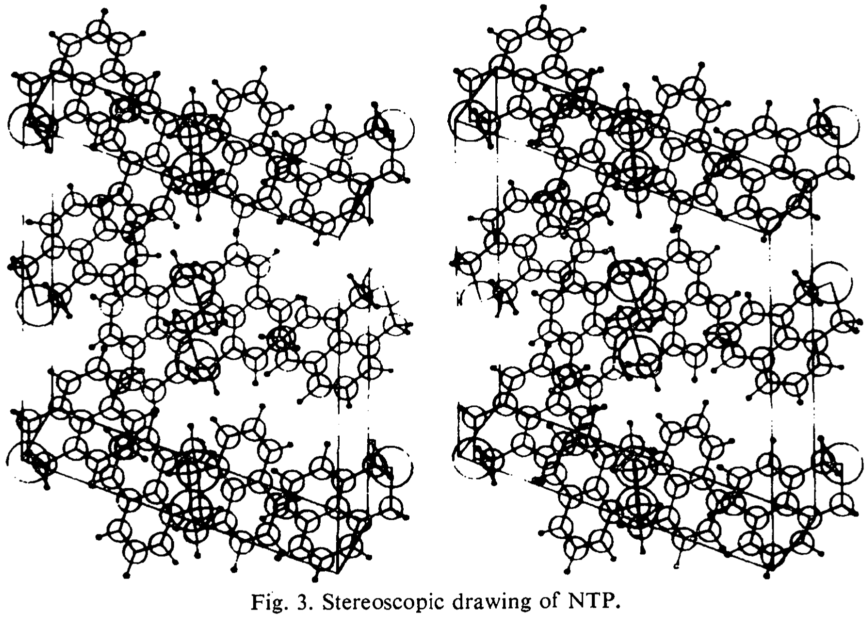

Fig. 3. Stereoscopic drawing of NTP.

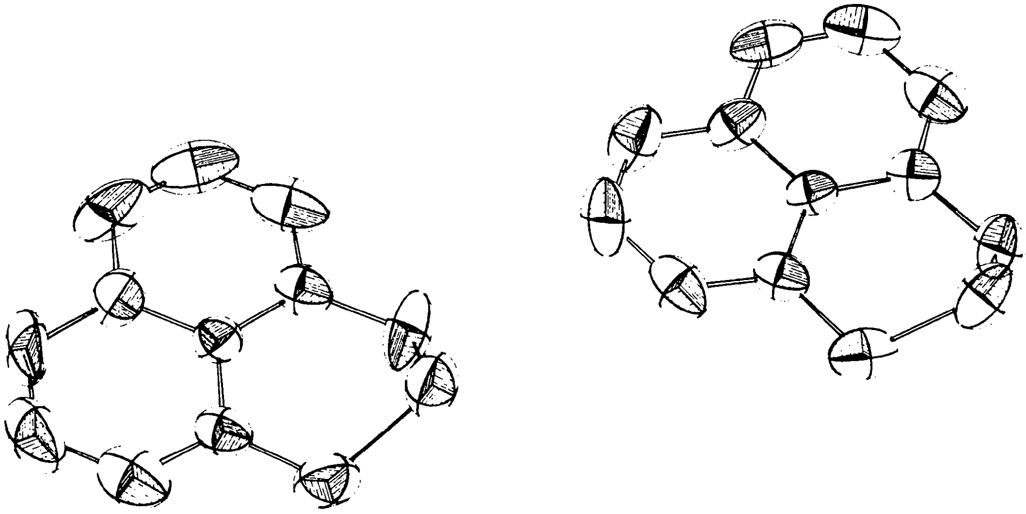

Fig. 4. Drawing of NTP showing the thermal ellipsoids as viewed along the *b* axis.

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The Crystal Structure of $NaMnCl_3$

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Crystals of $NaMnCl_3$ are rhombohedral, $R\bar{3}$, with hexagonal cell dimensions $a=6{\cdot}591$ (3) and $c=18{\cdot}627$ (9) Å and $Z=6$. $NaMnCl_3$ adopts the same structure as $FeTiO_3$ (ilmenite). A second isostructural halide is $NaCdCl_3$. Up to now no other halides isostructural with ilmenite have been reported.

Introduction

The crystal structure determination of $NaMnCl_3$ is part of an investigation of ABX_3 halides. B represents a first-row transition or an alkaline earth metal and $X=Cl$, Br or I. The choice of the monovalent metal A depends on the size of the anions. Since the authors are especially interested in ABX_3 compounds that do not have a perovskite-like structure, $A=Li$ or Na when $X=Cl$. The purpose of this investigation is to advance our understanding of the structures and the structural relations of these compounds. The first author is engaged in a systematic derivation of ABX_3 structures with both A and B octahedrally coordinated, the results of which will be published elsewhere. The phase diagram of the $NaCl$–$MnCl_2$ system was constructed by Ehrlich, Koknat & Seifert (1965). The crystal structure of Na_2MnCl_4 was determined by Goodyear, Ali & Steigmann (1971). This compound is isostructural with Sr_2PbO_4 (Trömel, 1965). Kestigian & Croft (1969) reported single crystals of $NaMnCl_3$ to have hexagonal symmetry with cell dimensions $a=26{\cdot}65$ (6) and $c=6{\cdot}19$ (8) Å.

Experimental

According to the phase diagram of $NaCl$–$MnCl_2$ the compound $NaMnCl_3$ melts congruently at 428 °C.