Fig. 1. Stereoscopic view of (3) with crystallographic atomic labelling.

group is rotated about the C(7)—C(8) bond by about 100° relative to (1), (3) has a conformation similar to (1), with rotation only 30°. The angle between the ketone group [plane of C(6), C(7), C(8) and O(1)] and the aromatic ring is 11·8 (1)° in (3), compared to 21·1 (1) in (2) and 2·7 (1)–13·3 (2)° in (1). The equatorial γ-hydrogen, H_e [H(101)], in (3) is closer to O(1) (2·70 Å) than the axial γ-hydrogen, H_a [H(102)] (3·90 Å), the 2·70 Å distance being close to the suggested upper limit of 2·72 Å (van der Waals radii sum) for hydrogen abstraction (Appel, Jiang, Scheffer & Walsh, 1983). The angle τ (Ariel & Trotter, 1985) is 63° for H_e [45° in (1), 65° in (2)], and the Δ angle is 76° [90° in (1), 73° in (2)]. As a result of the differing torsion angles about the C(7)—C(8) bond, abstraction of H_e would involve a boat-shaped six-membered transition state, as in (1), and in contrast to the

chair-shaped geometry in (2). It has not proved possible to establish the photochemical behaviour of (3).

The angle formed between the two aromatic rings in the molecule is 64·5 (1)°. Intermolecular distances correspond to van der Waals interactions.

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Structural Characterization and Synthesis of 7-Thia-1,5-diazatricyclo[7.4.1.0⁵,¹⁴]tetradec-9(14)-ene-6,8-dithione

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Abstract. $C_{11}H_{14}N_2S_3$, $M_r = 270·42$, triclinic, $P\bar{1}$, $a = 9·514$ (2), $b = 15·411$ (3), $c = 8·780$ (2) Å, $\alpha = 101·22$ (2), $\beta = 101·60$ (2), $\gamma = 83·45$ (2)°, $V = 1233·1$ Å³, $D_m = 1·433$ (5), $D_x = 1·456$ Mg m⁻³, $Z = 4$, $F(000) = 568$, $\lambda(Mo\,K\alpha) = 0·7107$ Å, $\mu = 0·617$ mm⁻¹, $T = 295$ K, $R = 0·037$ for 3728 observed reflections. The two crystallographically independent tricyclic molecules are found to be in an almost identical conformation. The terminal C—S bond distance has a mean value of 1·677 (2) Å for the two independent molecules; the mean ring C—S bond length is 1·739 (2) Å. There is a short non-bonded intramolecular contact between the terminal and the ring S atoms [mean 2·862 Å].

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Introduction. 1,8-Diazabicyclo[5.4.0]undec-7-ene (DBU) is a bicyclic amine which has a strong basicity and is useful as an organic reagent for a variety of organic syntheses (Ulrich, Tucker, Stuber & Sayigh, 1968).
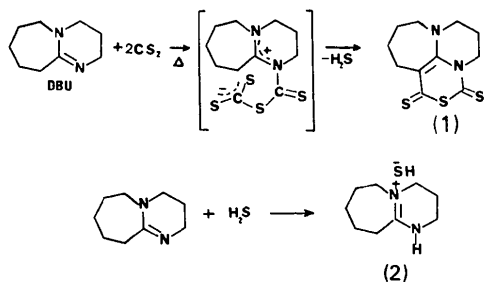
We found that DBU readily reacts with CS_2 to yield the title compound (1) and DBU hydrogen sulfide salt (2).

Our investigations of similar new reaction pathways to sulfur complexes and their related intermediates (Okamoto & Wojciechowski, 1982; Rice, Wojciechowski & Okamoto, 1982) have led us to the synthesis of this organosulfur compound and to its structural analysis, which is presented here. Of particular interest is the novel ring system, and the interactions that involve the terminal S atoms.

Experimental. When DBU (1·5 g, 10 mmol) was dissolved in CS_2 (30 ml, 500 mmol) at room temperature, the solution immediately became dark red. The reaction mixture was heated under reflux for 6 h with stirring under a nitrogen atmosphere. CS_2 was distilled from the heterogeneous reaction mixture to leave a dark-red viscous material. Methanol (20 ml) was added with stirring to obtain an orange precipitate which was filtered and dried under vacuum (1·3 g, 48% yield) (m.p. 478–481 K). Crystallization from acetonitrile gave orange crystals (1·2 g, 44% yield) (m.p. 483 K). The crystals were identifield as (1). Mass spectroscopy: 271($M+1$), chemical ionization (isobutane). IR(KBr): 2920(w), 1540(s), 1440(s), 1420(s), 1310(m), 1290(s), 1250(s), 1200(m), 1170(w), 990(w), 940(m), 690(w) cm^{-1}.

^{1}H NMR (CF$_3$COOH): $\delta = 4·35$ (t, $J = 6$ Hz, 4H, CH$_2$–NH–CH$_2$), 3·75 (d, $J = 6$ Hz, 2H, –CH$_2$– NH–R), 2·76 [broad S, 2H, –CH$_2$–C(–)=C], 2·3 (m, 2H), 1·92 (broad S, 4H, –CH$_2$–). (This spectrum corresponds to the protonated form.)

Analysis for $C_{11}H_{14}N_2S_3$: found: C 48·38, N10·92, S 35·77, H 4·93; calculated: C 48·70, N 10·93, S 35·42, H 5·16%.

The filtrate, on evaporation of the solvent under vacuum overnight, yielded a red viscous oil (1·0 g, 53% yield). IR(neat): 3350(s), 3210(s), 2900(s), 2800(s), 1630(s), 1570(w), 1440(s), 1200(m), 1170(w), 1110(s), 980(s), 820(w), 690(w) cm^{-1}.

^{1}H NMR (D$_2$O): $\delta = 3·6$ (m, 4H, –CH$_2$–N(=C)–CH$_2$–), 2·8 (m, 2H, –NH–CH$_2$–C), 2·2 (m, 2H, C–CH$_2$–C(=N)–NH–), 1·8 (m, 8H, –CH$_2$–). This compound corresponds to (2).

The red oil [(2); 0·90 g, 5 mmol] was dissolved in benzene (20 ml). KOH (0·56 g, 20 mmol in 20 ml H$_2$O) was added dropwise at 278 K with stirring. The organic layer was then separated, washed with water, followed by brine, dried over Na$_2$SO$_4$, and the solvent evaporated to yield a light-brownish oil (0·60 g, 78% yield). The IR and NMR analysis showed it to be identical to DBU.
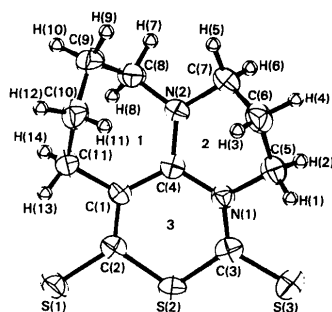
Orange parallelepiped crystal of (1) with dimensions 0·40 × 0·16 × 0·44 mm. D_m by pycnometry at 298 K. Examined by Weissenberg and precession techniques. Cell dimensions based on 38 high-θ values. CAD-4 diffractometer, ω–2θ scan of width (1·0 + 0·347 tanθ)°. Three standard reflections, no significant variations. 7827 reflections, $-11 \le h \le 9$, $-19 \le k \le 19$, $0 \le l \le 10$, $2 \le \theta \le 27°$; averaged to 4273 independent reflections; $R_{int} = 0·016$. 545 with $I < 2\sigma(I)$ excluded from the refinement. Correction for Lorentz–polarization effects and absorption (Coppens, Leiserowitz & Rabinovich, 1965); transmission factors: 0·832–0·922. Structure solved by direct methods ($MULTAN$78; Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). Refinement at first by difference synthesis ($DIFSYN$; McMullan, 1978) and then by least squares minimizing $\sum w(|F_o| - |F_c|)^2$ ($ORFLS$; Busing, Martin & Levy, 1962) with $w = 1/\sigma^2(F_o)$ and $\sigma^2(F_o) = [\sigma^2_{count}(I) + (0·02F_o^2)^2]/(4F_o^2)$. H atoms located from Fourier difference map and refined isotropically. $R = 0·037$, $wR = 0·036$, $S = 1·46$ for 391 parameters. In final cycle $(\Delta/\sigma)_{max} = 0·13$ (C, N, S) and 0·27 (H). $\Delta\rho = -0·18$ (5) to 0·28 (5) e Å^{-3}. Scattering factors from $International\ Tables\ for\ X$-ray $Crystallography$ (1974). Other programs used were $ORTEP$II (Johnson, 1976) and locally written programs.*

Discussion. The atomic positional and thermal parameters for the two independent molecules of the asymmetric unit are given in Table 1, and Table 2 includes the molecular bond lengths and angles. One of the two molecules is represented in Fig. 1 and the molecular-packing diagram is given in Fig. 2. There is excellent metric and angular agreement between the two molecules. The only important deviation seems to be between the N(2)–C(7), N(2)–C(8) and N(2')–C(7'), N(2')–C(8') distances where the variation is 4σ; this

Table 1. *Atomic positional parameters* ($\times 10^5$, $\times 10^3$ *for* H) *and* U_{eq} ($\mathring{A}^2 \times 10^4$) *values* [$B_{iso}$ ($\mathring{A}^2 \times 10^3$) *for* H]

$$U_{eq} = \tfrac{1}{3}\Sigma_i \Sigma_j U_{ij} a^*_i a^*_j \mathbf{a}_i . \mathbf{a}_j .$$

	x	y	z	U_{eq} or B_{iso}
C(1)	65255 (21)	39143 (14)	−9585 (23)	326 (7)
C(2)	65045 (22)	41919 (14)	6403 (25)	372 (7)
C(3)	80696 (22)	55464 (15)	3633 (26)	394 (8)
C(4)	73930 (21)	42973 (14)	−17469 (29)	325 (7)
C(5)	83124 (30)	56588 (17)	−22724 (29)	475 (9)
C(6)	77494 (31)	52671 (18)	−39637 (29)	511 (9)
C(7)	82568 (30)	43038 (17)	−42314 (29)	492 (9)
C(8)	77206 (28)	28986 (16)	−35495 (29)	462 (9)
C(9)	62476 (31)	26436 (19)	−44797 (30)	549 (10)
C(10)	50912 (28)	31593 (19)	−36307 (28)	498 (9)
C(11)	55015 (26)	32123 (17)	−18380 (27)	410 (8)
C(1')	96026 (22)	90760 (14)	13078 (24)	357 (7)
C(2')	9410 (23)	88879 (14)	8810 (27)	406 (8)
C(3')	94292 (25)	80629 (15)	−19587 (26)	429 (8)
C(4')	83703 (22)	86799 (13)	3695 (24)	346 (7)
C(5')	68276 (27)	82839 (19)	−23248 (29)	491 (9)
C(6')	56510 (27)	87480 (19)	−14927 (32)	519 (9)
C(7')	58068 (28)	84240 (20)	409 (31)	542 (10)
C(8')	73649 (30)	86557 (18)	27062 (29)	497 (9)
C(9')	71257 (31)	95995 (19)	35467 (32)	556 (10)
C(10')	80159 (27)	1914 (17)	29954 (32)	490 (9)
C(11')	95109 (26)	97554 (17)	28027 (30)	463 (8)
N(1)	78972 (18)	51482 (11)	−11753 (19)	347 (6)
N(2)	77627 (19)	38671 (12)	−31094 (19)	371 (6)
N(1')	82635 (18)	83596 (12)	−12660 (19)	366 (6)
N(2')	72221 (19)	86065 (12)	9823 (20)	385 (6)
S(1)	55941 (7)	37861 (5)	17777 (7)	503 (2)
S(2)	75107 (7)	50598 (4)	17300 (6)	449 (2)
S(3)	88251 (7)	64989 (4)	10851 (8)	534 (2)
S(3')	93119 (8)	75810 (5)	−38442 (8)	614 (2)
S(2')	11544 (6)	81578 (4)	−8712 (8)	485 (2)
S(1')	25128 (7)	92687 (5)	19198 (9)	564 (2)
H(1)	206 (2)	375 (2)	196 (3)	48 (5)
H(2)	59 (3)	436 (2)	207 (3)	47 (5)
H(3)	330 (3)	463 (1)	409 (3)	47 (5)
H(4)	185 (2)	440 (1)	464 (3)	43 (5)
H(5)	785 (2)	398 (2)	464 (3)	50 (5)
H(6)	64 (3)	579 (2)	408 (3)	55 (6)
H(7)	150 (2)	730 (2)	420 (3)	46 (5)
H(8)	204 (2)	737 (1)	254 (2)	35 (4)
H(9)	613 (2)	275 (1)	440 (3)	46 (5)
H(10)	377 (2)	801 (2)	451 (3)	49 (6)
H(11)	508 (2)	622 (2)	380 (3)	48 (5)
H(12)	586 (3)	711 (2)	404 (3)	49 (5)
H(13)	539 (2)	663 (1)	136 (2)	31 (4)
H(14)	408 (2)	738 (1)	157 (2)	36 (5)
H(1')	306 (2)	146 (2)	318 (3)	49 (6)
H(2')	330 (3)	236 (2)	270 (3)	52 (6)
H(3')	420 (2)	63 (2)	128 (3)	45 (5)
H(4')	531 (3)	139 (2)	216 (3)	52 (6)
H(5')	510 (3)	875 (2)	65 (3)	56 (6)
H(6')	428 (2)	223 (2)	19 (2)	44 (5)
H(7')	665 (3)	824 (2)	281 (3)	49 (5)
H(8')	837 (3)	835 (2)	310 (3)	45 (5)
H(9')	606 (3)	980 (2)	331 (3)	47 (5)
H(10')	745 (2)	961 (2)	475 (3)	53 (6)
H(11')	753 (2)	38 (1)	196 (3)	40 (5)
H(12')	813 (2)	76 (2)	371 (3)	45 (5)
H(13')	14 (2)	23 (2)	278 (2)	39 (5)
H(14')	−4 (2)	951 (2)	372 (3)	44 (5)

can be understood in terms of the pivotal function of the N(2) and N(2') atoms. There is also a small angular deviation in the angles involving the C(2)—S(1) and C(2')—S(1') bond: $\Delta\sigma = 1.2°$ between C(1)C(2)S(1) and C(1')C(2')S(1'), $\Delta\sigma = 1.1°$ between S(1)C(2)S(2) and S(1')C(2')S(3').

Table 2. *Interatomic distances* ($\mathring{A}$) *and angles* (°)

Molecule (1)		Molecule (2)	
C(1)—C(2)	1·389 (3)	C(1')—C(2')	1·383 (3)
C(1)—C(4)	1·417 (3)	C(1')—C(4')	1·416 (3)
C(1)—C(11)	1·526 (3)	C(1')—C(11')	1·522 (3)
C(5)—C(6)	1·504 (3)	C(5')—C(6')	1·497 (3)
C(6)—C(7)	1·496 (4)	C(6')—C(7')	1·500 (4)
C(8)—C(9)	1·525 (4)	C(8')—C(9')	1·512 (4)
C(9)—C(10)	1·516 (4)	C(9')—C(10')	1·516 (4)
C(10)—C(11)	1·532 (2)	C(10')—C(11')	1·529 (3)
N(1)—C(3)	1·356 (3)	N(1')—C(3')	1·362 (3)
N(1)—C(4)	1·411 (3)	N(1')—C(4')	1·412 (3)
N(1)—C(5)	1·491 (3)	N(1')—C(5')	1·493 (3)
N(2)—C(4)	1·342 (3)	N(2')—C(4')	1·336 (3)
N(2)—C(7)	1·470 (3)	N(2')—C(7')	1·457 (3)
N(2)—C(8)	1·470 (3)	N(2')—C(8')	1·480 (3)
S(1)—C(2)	1·689 (2)	S(1')—C(2')	1·690 (2)
S(2)—C(2)	1·746 (2)	S(2')—C(2')	1·753 (2)
S(2)—C(3)	1·727 (2)	S(2')—C(3')	1·731 (2)
S(3)—C(3)	1·664 (2)	S(3')—C(3')	1·665 (2)
C(2)—C(1)—C(11)	116·3 (2)	C(2')—C(1')—C(11')	116·7 (2)
C(2)—C(1)—C(4)	121·8 (2)	C(2')—C(1')—C(4')	121·6 (2)
C(4)—C(1)—C(11)	121·8 (2)	C(4')—C(1')—C(11')	121·7 (2)
C(1)—C(2)—S(1)	128·1 (2)	C(1')—C(2')—S(1')	126·9 (2)
C(1)—C(2)—S(2)	120·4 (4)	C(1')—C(2')—S(2')	120·5 (2)
S(1)—C(2)—S(2)	111·5 (1)	S(1')—C(2')—S(2')	112·6 (1)
N(1)—C(3)—S(2)	120·3 (2)	N(1')—C(3')—S(2')	120·5 (2)
N(1)—C(3)—S(3)	124·4 (2)	N(1')—C(3')—S(3')	123·6 (2)
S(2)—C(3)—S(3)	115·3 (1)	S(2')—C(3')—S(3')	115·9 (1)
C(1)—C(4)—N(1)	122·7 (2)	C(1')—C(4')—N(1')	122·4 (2)
C(1)—C(4)—N(2)	121·1 (2)	C(1')—C(4')—N(2')	121·5 (2)
N(1)—C(4)—N(2)	116·1 (2)	N(1')—C(4')—N(2')	116·0 (2)
N(1)—C(5)—C(6)	110·8 (2)	N(1')—C(5')—C(6')	111·2 (2)
C(5)—C(6)—C(7)	108·3 (2)	C(5')—C(6')—C(7')	108·2 (2)
C(6)—C(7)—N(2)	109·1 (2)	C(6')—C(7')—N(2')	109·2 (2)
C(9)—C(8)—N(2)	111·4 (2)	C(9')—C(8')—N(2')	111·7 (2)
C(8)—C(9)—C(10)	109·1 (2)	C(8')—C(9')—C(10')	109·2 (2)
C(9)—C(10)—C(11)	112·0 (2)	C(9')—C(10')—C(11')	112·6 (2)
C(10)—C(11)—C(1)	116·1 (2)	C(10')—C(11')—C(1')	116·7 (2)
C(3)—N(1)—C(4)	123·9 (2)	C(3')—N(1')—C(4')	123·1 (2)
C(3)—N(1)—C(5)	115·8 (2)	C(3')—N(1')—C(5')	116·2 (2)
C(4)—N(1)—C(5)	120·3 (2)	C(4')—N(1')—C(5')	120·7 (2)
C(4)—N(2)—C(7)	123·9 (2)	C(4')—N(2')—C(7')	123·9 (2)
C(4)—N(2)—C(8)	120·5 (2)	C(4')—N(2')—C(8')	120·0 (2)
C(7)—N(2)—C(8)	115·6 (2)	C(7')—N(2')—C(8')	116·0 (2)
C(2)—S(2)—C(3)	106·0 (2)	C(2')—S(2')—C(3')	105·0 (2)

Fig. 1. One of the two independent $C_{11}H_{14}N_2S_3$ molecules with thermal ellipsoids drawn to enclose 50% probability. A constant $B = 1\ \mathring{A}^2$ was used for the H atoms.
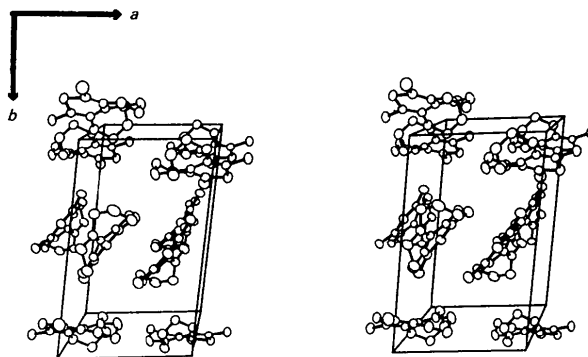
Fig. 2. Stereoscopic view of the crystal packing down the c^* axis with the a axis horizontal. The ellipsoids are drawn to enclose 50% probability.

The two independent tricyclic molecules in the asymmetric unit adopt a butterfly conformation, which is probably dependent on the N···H interactions between N(2) or N(2′) and the H atoms [H(3), H(11),H(3′),H(11′)] of the rings [N(2)···H(3), N(2)···H(11) = 2·64 (2), 2·67 (2) Å and N(2′)··· H(3′), N(2′)···H(11′) = 2·73 (2), 2·57 (2) Å]. These interaction distances are all shorter than the van der Waals distance (N···H) of 2·75 Å (Bondi, 1964). The puckering of rings (1) and (2) to the same side may be due to the lone pair (partial) of N(2) and N(2′), which is directed to a point on the opposite side of the two H atoms [H(3) and H(11), or H(3′) and H(11′)]. Atoms N(2) and N(2′) do show some conjugation with C(4) and C(4′) of the rings, giving rise to the rather short N—C bond distances of 1·342(3) and 1·336(3)Å respectively. In fact, some double-bond character is evident when compared to the accepted N=C and N—C values of 1·259 and 1·475 Å (Ymén, 1983). Weak double-bond character is also shown by N(1) and N(1′) which belong to the conjugation systems of the S-containing rings.

The terminal C—S bond [⟨1·677 (2)⟩Å] is longer than the C=S distance of 1·595 (3) Å found in ClFCS (Hamm, Kohrmann, Günther & Zeil, 1976) and in CH_2SO [1·610 (2) Å] (Penn & Olsen, 1976), but significantly shorter than the accepted (*International Tables for X-ray Crystallography*, 1974; Ymén, 1983) single-bond C—S distance of 1·81 Å. The ring C—S bond distances are longer than the terminal C—S bonds [⟨C—S⟩$_R$ = 1·739 (2) Å] but again shorter than a C—S single bond of 1·81 Å. The average C—S bond length in the title compound compares well with that found in thiourea [1·714 (8) Å] (Mullen & Hellner, 1978) and dithiooxamide [1·665 (5), 1·633 (5) Å] (Wheatley, 1965) where some conjugation does take place with the chains, indicating some double-bond character in the terminal C—S bonds. In fact, all bond distances in the C(1),C(2),S(2),C(3),N(1),C(4) and C(1′),C(2′),S(2′), C(3′),N(1′),C(4′) rings are shorter than the corresponding single-bond values — evidence of some conjugation in this ring system. Both rings (3) and (3′), including the two terminal S atoms, are quite planar, with only the upper part of the ring, fused to the upper bicyclic system [(1) and (2)], showing any important dihedral deviation [C(1)C(4)N(1)C(3) 24·5, C(2)C(1)C(4)N(1) −22·0°] from planarity [C(1′)C(4′)N(1′)C(3′) 27·8, C(2′)C(1′)C(4′)N(1) −24·8°].

It is of interest to note the short non-bonded intramolecular contacts between the terminal S atoms and the ring S atoms (⟨S···S⟩) = 2·862 Å. Similar types of non-bonded interactions involving S atoms have recently been observed in $C_{12}H_{13}NOS_2$ (Kamath & Venkatesan, 1984) and $C_{12}H_{18}ClNO_2S$ (Sergeyeva, Struchkov, Kurkutova, Zemlyanukhina, Sadovaya & Zefirov, 1984), where short S···O distances have been observed. This S···S interaction may be in part the

result of intramolecular steric interactions between the terminal S atoms and the H atoms of the rings opposite them (Ymén, 1983). This is corroborated by the opening of the angles N(1)C(3)S(3) and C(1)C(2)S(1) and the closing of the angles S(3)C(3)S(2) and S(1)C(2)S(2). The shortest S···H non-bonded distances are: S(1)—H(13) 2·70 (2), S(3)—H(1) 2·60 (2), S(3′)— H(1′) 2·66 (2), S(1′)—H(13′) 2·71 (2), ⟨S$_T$—H⟩ 2·67 (2) Å. These distances may indicate a C—H···S steric interaction. Such C—H···S steric interactions have been observed in several dithiocarbamates (Oskarsson & Ymén, 1984; Ymén, 1983, 1984).

The C—H bond geometries are normal with distances between 0·95 (2) and 1·04 (2) Å and tetrahedral angles between 102·8 (15) and 113·3 (15)°.

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