Discussion. The *ORTEPB* drawing of the molecular structure is given in Fig. 1. The positional and isotropic thermal parameters are given in Table 1.* Bond lengths and angles are given in Table 2. Three bonds, C1—C2, C3—C5 and C8—C9, are double bonds, and all the other C—C bonds are single. The geometry of the methyl ester group is normal. The result confirmed the structure proposed by Arya, Erdtman & Kubota (1961) and Thomas (1966).

* Lists of anisotropic thermal parameters, structure factors and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43803 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 2-*tert*-Butyl-6-methyl-1,6aλ^4-dithia-6-azapentalene

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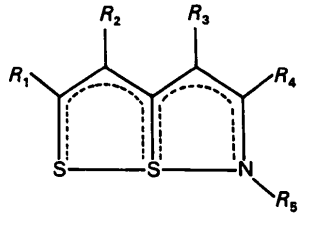
Abstract. $C_{10}H_{15}NS_2$, $M_r = 213{\cdot}4$, triclinic, $P\bar{1}$, $a = 5{\cdot}891\ (5)$, $b = 8{\cdot}857\ (4)$, $c = 11{\cdot}449\ (3)$ Å, $\alpha = 105{\cdot}96\ (3)$, $\beta = 95{\cdot}82\ (4)$, $\gamma = 74{\cdot}15\ (5)°$, $V = 552{\cdot}23\ (6)$ Å^3, $Z = 2$, $D_x = 1{\cdot}28$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 4{\cdot}2$ cm^{-1}, $F(000) = 228$, $T = 293$ K, final $R = 0{\cdot}0413$ for 2089 observed unique reflections. A crystal-structure determination of the title compound showed the molecule to be bicyclic, almost planar, and to possess elongated S—S and compressed S—N bond lengths of 2·496 (1) and 1·791 (2) Å, respectively, and an N—S—S bond angle of 173·4 (1)°.

Introduction. A number of crystal-structure analyses of heterocyclic compounds of the type (1) have been carried out.



(1)

The compounds studied so far have all possessed *N*-aryl groups. A summary of the bond lengths of the S—S and S—N bonds is given:

	R_1	R_2	R_3	R_4	R_5	S–S(Å)	S–N(Å)	Reference
1(*a*)	Ph	H	Ph	H	Ph	2·396 (12)	1·871 (10)	Leung & Nyburg (1972)
1(*b*)	Ph	H	Ph	H	3-Quinolinyl	2·364 (7)	1·887 (2)	Leung & Nyburg (1971)
1(*c*)	Ph	H	H	Ph	Ph	2·440 (2)	1·860 (4)	Borel, Leclaire, Le Coustumer & Mollier (1978).

It can be seen that there are no large differences in the bond lengths under consideration. A structural feature also of interest in these compounds concerns the internal S—S—N bond angles, which for the three compounds listed are 174·5, 174·2, and 173·9°, respectively.

We have undertaken the crystal-structure analysis of the title compound, which is the first of this class of compounds in which all the substituents are alkyl, to determine what effect an alkyl group bonded to N has on the structural parameters of interest.

Experimental. The title compound was prepared by treatment of 5-*tert*-butyl-3-(2-dimethylaminovinyl)-1,2-dithiolium perchlorate (Vilsmeir salt) in dimethylformamide with aqueous methylamine (Dingwall, Ingram, Reid & Symon, 1973) at room temperature. The compound was recrystallized from hexane, to give

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greenish-yellow needles (m.p. 395–396 K). A single crystal of dimensions 0·2 × 0·4 × 0·6 mm was mounted in a Lindemann capillary tube and sealed. Weissenberg photographs of the crystal showed it to be triclinic. The same crystal was used for data collection: Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation at room temperature, ω–2θ scan. Lattice parameters from least-squares refinement of 25 reflections, $16 < \theta < 18°$. Three standard reflections, $\bar{1}\bar{6}7$, $1\bar{5}7$, and $2\bar{5}5$, were monitored after every 77 reflections and showed no more than 0·7% intensity variation throughout the data collection. All (3351) reflections in the hemisphere h: −8 to 8, k: −12 to 12, l: 0 to 16 and the range $3 < \theta < 30°$ were measured. Max. $\sin\theta/\lambda = 0{\cdot}70$ Å^{-1}. 2089 unique reflections with $I > 4\sigma(I)$ were used in the analysis ($R = 0{\cdot}0113$). Empirical absorption corrections (North, Phillips & Mathews, 1968) (correction factors, max. and min. values 0·9998, 0·9856) and LPF corrections were applied.

The density indicated two molecules per unit cell, and it was initially assumed that the space group was $P\bar{1}$. The structure was solved by direct methods using *SHELX*86 (Sheldrick, 1986). The computed E map revealed all non-H atoms of the molecule.

The structure was subsequently refined using *SHELX*76 (Sheldrick, 1978). Full-matrix least-squares refinement based on F of positional and thermal parameters, and a scale factor {all H atoms were located from a difference Fourier map and were allowed to refine freely except H(4), H(10), and H(12) which were fixed, d[C(7)–H(4)] = 1·08, d[C(9)–H(10)] = 1·08, and d[C(9)–H(12)] = 1·08 Å}, with a common isotropic temperature factor for the H atoms, gave $R = 0{\cdot}0413$, $wR = 0{\cdot}0378$ [$w = 1/\sigma^2(F)$]. 149 parameters were refined, including anisotropic thermal parameters for non-H atoms and the common isotropic thermal parameters for the H atoms. $(\Delta/\sigma)_{max}$ for non-H atoms 0·094, for H atoms 0·519. Max. and min. residual electron density on final difference Fourier synthesis $\Delta\rho_{max} = 0{\cdot}29$, $\Delta\rho_{min} = -0{\cdot}355$ e Å^{-3}. Atomic scattering factors for non-H atoms from Cromer & Mann (1968), anomalous-dispersion correction factors from Cromer & Liberman (1970); H-atom scattering factors from Stewart, Davidson & Simpson (1965).

Discussion. Final atomic coordinates and bond distances and angles are presented in Tables 1 and 2.* The structure of 2-*tert*-butyl-6-methyl-1,6aλ^4-dithia-6-azapentalene is shown in Fig. 1. The frame of the molecule is approximately planar with maximum deviations from planarity not exceeding 0·05 Å. The methyl C atom [C(10)] and the central C atom of the *tert*-butyl group [C(6)] are not significantly displaced from the calculated mean plane of the molecule. The S–S and S–N bond lengths, and the S–S–N bond angle, may be compared with those of 1(*a*), 1(*b*), and 1(*c*). The S–S bond length, 2·496 (1) Å, is significantly

Table 1. *Fractional coordinates* (×10^4) *and equivalent isotropic temperature factors* (Å^2, ×10^3) *for non*-H *atoms*

	x	y	z	U_{eq}*
S(1)	3830 (1)	2774 (1)	2104 (1)	43 (1)
S(2)	3033 (1)	5279 (1)	1370 (1)	37 (1)
N	2778 (4)	7106 (3)	925 (2)	38 (1)
C(1)	6128 (4)	3267 (3)	3035 (2)	31 (1)
C(2)	6686 (5)	4651 (3)	3016 (2)	33 (1)
C(3)	5444 (5)	5687 (3)	2293 (2)	33 (1)
C(4)	5916 (5)	7107 (3)	2213 (3)	41 (2)
C(5)	4391 (6)	7876 (4)	1433 (3)	44 (2)
C(6)	7515 (4)	2115 (3)	3795 (2)	32 (1)
C(7)	9143 (5)	667 (4)	2937 (3)	44 (2)
C(8)	5852 (5)	1507 (4)	4387 (3)	43 (2)
C(9)	9035 (6)	2961 (4)	4794 (3)	43 (2)
C(10)	982 (5)	7571 (4)	17 (3)	47 (2)

* $U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

Table 2. *Bond lengths* (Å) *and angles* (°)

S(1)–S(2)	2·496 (1)	S(1)–C(1)	1·713 (2)
S(2)–N	1·791 (2)	S(2)–C(3)	1·741 (2)
N–C(5)	1·315 (3)	N–C(10)	1·464 (3)
C(1)–C(2)	1·359 (3)	C(1)–C(6)	1·535 (3)
C(2)–C(3)	1·411 (3)	C(3)–C(4)	1·388 (3)
C(4)–C(5)	1·381 (4)	C(6)–C(7)	1·535 (4)
C(6)–C(8)	1·527 (4)	C(6)–C(9)	1·535 (4)

S(2)–S(1)–C(1)	92·4 (1)	S(1)–S(2)–N	173·4 (1)
S(1)–S(2)–C(3)	85·7 (1)	N–S(2)–C(3)	87·9 (1)
S(2)–N–C(5)	113·8 (2)	S(2)–N–C(10)	120·2 (2)
C(5)–N–C(10)	125·9 (2)	S(1)–C(1)–C(2)	117·1 (2)
S(1)–C(1)–C(6)	119·5 (2)	C(2)–C(1)–C(6)	123·3 (2)
C(1)–C(2)–C(3)	123·2 (2)	S(2)–C(3)–C(2)	121·5 (2)
S(2)–C(3)–C(4)	111·7 (2)	C(2)–C(3)–C(4)	126·7 (2)
C(3)–C(4)–C(5)	113·8 (3)	N–C(5)–C(4)	112·8 (3)
C(1)–C(6)–C(7)	107·6 (2)	C(1)–C(6)–C(8)	111·0 (2)
C(7)–C(6)–C(8)	109·5 (2)	C(1)–C(6)–C(9)	111·0 (2)
C(7)–C(6)–C(9)	108·8 (2)	C(8)–C(6)–C(9)	108·8 (2)

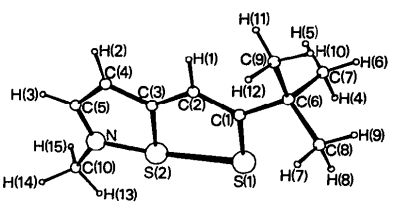

Fig. 1. A perspective view of the title compound showing atom numbering.

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond lengths involving H have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43759 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

longer than the S—S bond length in compounds having an *N*-aryl grouping, and the S—N bond length of 1·791 (2) Å is significantly shorter. The S—S bond in the title compound is 0·4 Å longer than that of an accepted single-bond length of 2·10 Å (Hordvik, 1970), but 0·70 Å shorter than the shortest distance found in a crystal between two non-bonded S atoms of 3·20 Å (Lozac'h, 1971). The sum of the S—S and S—N bond lengths is 4·287 (3) Å, approximately 10% greater than the sum of typical two-centre, two-electron covalent S—S and S—N bond lengths (3·85 Å), and is thus similar to that found in analogous molecules (Hansen, 1977).

The N—S—S bond angle, 173·4 (1)°, is very close to that found in analogous structures [see 1(*a*), 1(*b*), and 1(*c*)]. The length of the bond from the central S atom to the central C atom, S(2)—C(3), is 1·741 (2) Å. This is in the range found for the other three analogues, 1·740 (6)–1·752 (7) Å (Leung & Nyburg, 1971, 1972; Borel *et al.*, 1978). The S(1)—C(1) bond length is 1·713 (2) Å, which is significantly shorter than that of S(2)—C(3), indicating multiple-bond character for the S(1)—C(1) bond in contrast to the single bond of S(2)—C(3). Again this is typical of these types of molecule.

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Structure of Benzyldimethyl(phenyl)ammonium Chloride Monohydrate

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Abstract. $C_{15}H_{18}N^+.Cl^-.H_2O$, $M_r = 265·7$, monoclinic, $C2/c$, $a = 14·115$ (8), $b = 12·704$ (8), $c = 16·866$ (8) Å, $\beta = 108·83$ (3)°, $V = 2862$ (3) Å^3, $Z = 8$, $D_m = 1·24$, $D_x = 1·23$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 2·13$ mm^{-1}, $F(000) = 1136$, $T = 298$ K, final $R = 0·054$ for 1947 'observed' reflections. The conformation of the N^+—C—C—C part of the molecule is similar to the conformation of the active part of acetylcholine and its structural analogues. The Cl^- ions and the water molecules form hydrogen-bonded chains. The packing of the cations is determined mainly by Coulombic and van der Waals forces.

Introduction. Benzyldimethyl(phenyl)ammonium chloride was synthesized by the method of Michler & Gradmann (1877) and characterized by UV, IR and

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