

Table 2. Bond lengths (Å) and angles (°) for [Ru(Cl)₂(C₁₀H₁₄)(C₇H₉N)]

Ru1—Cl1	2.418 (1)	C4—C5	1.393 (4)
Ru1—Cl2	2.412 (1)	C5—C6	1.412 (4)
Ru1—C1	2.161 (2)	C6—C7	1.498 (4)
Ru1—C2	2.151 (2)	C8—C10	1.493 (4)
Ru1—C3	2.180 (2)	C9—C10	1.508 (5)
Ru1—C4	2.122 (2)	N1—C16	1.430 (3)
Ru1—C5	2.143 (2)	C14—C15	1.366 (4)
Ru1—C6	2.184 (2)	C14—C19	1.379 (4)
Ru1—N1	2.118 (2)	C15—C16	1.374 (3)
Cl1—C2	1.412 (4)	C16—C17	1.366 (4)
Cl1—C6	1.394 (4)	C17—C18	1.369 (4)
C2—C3	1.397 (4)	C18—C19	1.378 (4)
C3—C4	1.417 (3)	C19—C20	1.479 (4)
C3—C10	1.511 (4)		
Cl1—Ru1—Cl2	89.04 (2)	Ru1—Cl1—C6	72.2 (2)
Cl1—Ru1—C1	162.10 (8)	C2—C1—C6	121.0 (3)
Cl1—Ru1—C2	123.99 (7)	Ru1—C2—C1	71.3 (1)
Cl1—Ru—C3	94.28 (7)	Ru1—C2—C3	72.3 (1)
Cl1—Ru1—C4	89.69 (8)	C1—C2—C3	121.9 (2)
Cl1—Ru1—C5	112.87 (8)	Ru1—C3—C2	70.1 (1)
Cl1—Ru1—C6	149.93 (7)	Ru1—C3—C4	68.6 (1)
Cl1—Ru1—N1	80.12 (6)	Ru1—C3—C10	131.5 (2)
Cl2—Ru1—C1	108.79 (7)	C2—C3—C4	116.5 (2)
Cl2—Ru1—C2	145.52 (7)	C2—C3—C10	123.2 (2)
Cl2—Ru1—C3	165.36 (7)	C4—C3—C10	120.3 (2)
Cl2—Ru1—C4	127.50 (7)	Ru1—C4—C3	73.0 (1)
Cl2—Ru1—C5	96.27 (7)	Ru1—C4—C5	71.8 (1)
Cl2—Ru1—C6	87.84 (7)	C3—C4—C5	122.0 (3)
Cl2—Ru1—N1	83.15 (7)	Ru1—C5—C4	70.1 (1)
Cl—Ru1—C2	38.2 (1)	Ru1—C5—C6	72.5 (1)
Cl—Ru1—C3	68.9 (1)	C4—C5—C6	120.8 (2)
Cl—Ru1—C4	80.8 (1)	Ru1—C6—C1	70.4 (1)
Cl—Ru1—C5	67.9 (1)	Ru1—C6—C5	69.4 (1)
Cl—Ru1—C6	37.4 (1)	Ru1—C6—C7	129.7 (2)
Cl—Ru1—N1	99.9 (1)	C1—C6—C5	117.8 (2)
C2—Ru1—C3	37.6 (1)	C1—C6—C7	121.8 (3)
C2—Ru1—C4	68.1 (1)	C5—C6—C7	120.3 (3)
C2—Ru1—C5	80.7 (1)	C3—C10—C8	113.9 (3)
C2—Ru1—C6	68.6 (1)	C3—C10—C9	108.3 (3)
C2—Ru1—N1	92.4 (1)	C8—C10—C9	109.6 (3)
C3—Ru1—C4	38.5 (1)	Ru1—N1—C16	121.5 (2)
C3—Ru1—C5	69.31 (9)	C15—C14—C19	120.7 (3)
C3—Ru1—C6	82.2 (1)	C14—C15—C16	120.4 (3)
C3—Ru1—N1	111.46 (9)	N1—C16—C15	120.4 (2)
C4—Ru1—C5	38.1 (1)	N1—C16—C17	119.6 (2)
C4—Ru1—C6	69.0 (1)	C15—C16—C17	120.0 (2)
C4—Ru1—N1	147.8 (1)	C16—C17—C18	119.1 (3)
C4—Ru1—C6	38.1 (1)	C17—C18—C19	121.9 (3)
C5—Ru1—N1	167.0 (1)	C14—C19—C18	117.9 (3)
C6—Ru1—N1	129.04 (9)	C14—C19—C20	120.0 (3)
Ru1—Cl1—C2	70.5 (1)	C18—C19—C20	122.0 (3)

The Ru—ring distance of 1.637 (2) Å is slightly shorter than that observed for [Ru(cymene)Cl(μ -N₃)₂] (Bates, Begley & Wright, 1990) (1.66 Å) and [Ru(cymene)Cl(pyz)₂]PF₆ (1.68 Å). Since the major metal—ligand interactions between the arene and the metal involve both σ and π donation from the arene, this shorter distance must reflect the stronger metal—arene interaction which in turn is a consequence of the relatively poor donating capacity of the amine ligand. The Ru—Cl bond lengths, however, do not differ significantly from those in the other determinations already mentioned and there is no detectable lengthening of the Ru—N bond.

The relatively small coordination site occupied by the amine ligand makes it seem likely that there are no great steric demands on the ligand that might lead to the observed lability. Rather, the lability must be attributed to the weak Ru—N bond. However, the detection of this weakness is not possible in terms of the bond length itself, and can only be inferred from the relatively short Ru—arene distance.

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Structure of *N,N*-Dimethyl-1-[2-(phenylthio)phenyl]-2-propylammonium Hydrogen Maleate

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Abstract. C₂₁H₂₅NO₄S, *M*_r = 387.49, monoclinic, *P*2₁/*c*, *a* = 10.092 (2), *b* = 32.095 (7), *c* = 6.498 (1) Å, β = 100.97 (2)°, *V* = 2066.4 (8) Å³, *Z* = 4, *D*_x =

1.246 g cm⁻³, Cu *K* α radiation, λ = 1.5418 Å, μ = 15.58 cm⁻¹, *F*(000) = 824, *T* = 295 K, *R* = 0.045, *wR* = 0.066 for 2411 observed reflections. The crystal

structure contains discrete pairs of hydrogen maleate anions and RMe_2NH^+ cations, where R is 1-[2-(phenylthio)phenyl]-2-propyl. The ion pairs are held together by van der Waals forces. The hydrogen maleate anion contains an intramolecular hydrogen bond. The angle between mean planes of the two phenyl rings of the diphenyl sulfide is $78.76(9)^\circ$.

Introduction. In recent years, research on antidepressants has focused on compounds with specific mechanisms of action, particularly on those which affect the potentiation of 5-hydroxytryptamine (serotonin) transmission in nerves. These compounds – ‘the second generation antidepressants’ (Enna & Eison, 1987) – have been developed to eliminate some of the many troublesome side-effects of classical tricyclic antidepressants (Blackwell, 1981). Most second generation antidepressants influence serotonergic nerve functions whereas noradrenaline functions are not affected.

Potentially antidepressive properties were recently found in a group of aminoalkyldiphenyl sulfides (Jilek *et al.*, 1989). They show a high affinity for both imipramine and desipramine binding sites in the rat brain and inhibit the re-uptake of 5-hydroxytryptamine as well as of noradrenaline in rat brain structures. A few, however, are specific for the uptake inhibition of just one amine.

In order to gain a better understanding of the relationship between structure and biological activity in these compounds, we have focused on an analysis of the structural behaviour of diphenyl sulfides using their known three-dimensional structures (Schneider, Rejholec & Kuchař, 1990) and X-ray structure determinations of some important new derivatives. The title compound is a relatively weak inhibitor of serotonin as well as of noradrenaline re-uptake.

Experimental. Colorless crystals were grown from ethyl alcohol–heptane solution (9:1) by slow evaporation at room temperature. Crystal dimensions $0.30 \times 0.30 \times 0.18$ mm, Syntex $P2_1$ diffractometer, graphite-monochromated $Cu K\alpha$ radiation. Cell constants by least squares using 15 reflections with $9 \leq 2\theta \leq 30^\circ$. Cell dimensions and space group independently determined by Weissenberg and oscillation techniques. Only 2805 unique reflections collected using $\theta/2\theta$ scans, 2411 reflections with $F > 3.92\sigma(F)$, $0 \leq 2\theta \leq 116^\circ$, $-10 \leq h \leq 10$, $0 \leq k \leq 34$, $0 \leq l \leq 7$. No absorption corrections applied ($\mu r = 0.41$). No significant variation observed for three standard reflections (230, 080, 002) measured every 47 reflections.

The structure was solved by direct methods using *SHELXS86* (Sheldrick, 1986), in the space group $P2_1/c$ and refined with *SHELX76* (Sheldrick, 1976). The atomic scattering factors were taken from this

Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors for non-H atoms (\AA^2) with their e.s.d.'s in parentheses*

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	$x \times 10^4$	$y \times 10^5$	$z \times 10^4$	$U_{eq} \times 10^4$
S1	4384.8 (7)	4755 (2)	1469.7 (12)	724 (3)
C2	2722 (2)	4678 (7)	2002 (4)	574 (10)
C3	2211 (3)	821 (8)	2446 (5)	713 (10)
C4	891 (3)	457 (10)	2694 (5)	812 (12)
C5	87 (3)	3916 (10)	2544 (5)	783 (12)
C6	589 (3)	7750 (9)	2131 (4)	657 (10)
C7	1910 (2)	8232 (7)	1850 (3)	534 (8)
C8	2391 (3)	12552 (8)	1431 (4)	601 (9)
C9	2404 (3)	13425 (8)	-853 (4)	615 (10)
C10	1108 (4)	12434 (12)	-2359 (5)	896 (16)
N11	2808 (2)	17980 (6)	-1064 (3)	543 (6)
C12	1736 (4)	21141 (10)	-1015 (6)	771 (13)
C13	3405 (3)	18641 (10)	-2967 (5)	701 (11)
C14	5394 (2)	7136 (7)	3673 (4)	551 (8)
C15	5006 (3)	7770 (8)	5578 (4)	648 (10)
C16	5884 (3)	9666 (9)	7194 (5)	696 (10)
C17	7152 (3)	10839 (9)	6970 (5)	714 (11)
C18	7550 (3)	10130 (10)	5092 (5)	743 (11)
C19	6684 (3)	8344 (9)	3459 (5)	678 (10)
O20	4519 (2)	20668 (6)	2539 (4)	828 (8)
C21	5695 (3)	19873 (8)	2396 (5)	677 (9)
C22	6782 (3)	21424 (10)	4090 (5)	820 (13)
C23	8121 (3)	21295 (10)	4289 (5)	809 (12)
C24	8991 (3)	19441 (8)	2943 (5)	650 (10)
O25	10210 (2)	19716 (7)	3492 (4)	878 (9)
O26	8442 (2)	17537 (7)	1247 (4)	887 (9)
O27	6007 (2)	17847 (7)	881 (3)	871 (9)

program without any changes. An isotropic refinement based on $|F|$ with all non-H atoms gave $R = 0.14$. H atoms were included at geometrically idealized positions but their parameters were refined without any constraints to $R = 0.12$. Finally, anisotropic refinement of non-H atoms with isotropic refinement of H atoms (344 variables) converged at $R = 0.045$, $wR = 0.066$, $S = 1.8$ using 2411 F 's with $F > 3.92\sigma(F)$ ($R = 0.052$, $wR = 0.069$ for all unique reflections), $w = [\sigma(F)^2 + 0.0009F^2]^{-1}$. $(\Delta/\sigma)_{\max} = 0.17$. Final difference Fourier map did not contain any peak higher than 0.32 or lower than $-0.22 e \text{\AA}^{-3}$.

Discussion. The final fractional coordinates and equivalent isotropic temperature factors are listed in Table 1 along with their estimated standard deviations.* A *PLUTO* (Motherwell, 1978) drawing of a single molecule of the title compound is depicted in Fig. 1 and a packing diagram is shown in Fig. 2. Intra- and intermolecular geometrical parameters were computed by the program *PARST* (Nardelli, 1983).

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53099 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The most pivotal crystal-packing forces are bifurcated hydrogen bonds N11—H111...O20 [N11...O20 2.769 (3) Å, N11—H111...O20 167 (2)°] and N11—H111...O27 [N11...O27 3.236 (3) Å, N11—H111...O27 132 (2)°]. Hydrogen-bonded ionic pairs do not form any other hydrogen bonds with their neighbours and other crystal-packing forces are of the van der Waals type. The small number of hydrogen bonds and the lack of any polymeric structure in the crystal are consistent with its relatively low melting point, 357–359 K.

The principal bond lengths, angles and torsion angles are listed in Table 2. The H atom H261 of the maleate anion forms an intramolecular hydrogen bridge between O26 and O27 [O26...O27 2.426 (3) Å, O26—H261...O27 177 (3)°]. The non-H atoms of this anion have relatively small displacements from the mean plane [$\chi^2 = 931$ for five degrees of freedom with a greatest deviation for C22 of -0.055 (3) Å]. The C—C—C valence angles on the double bond are greater than normal (Table 2). The deformation

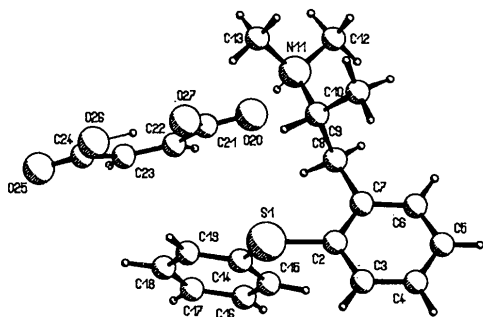


Fig. 1. Perspective view of *N,N*-dimethyl-1-[2-(phenylthio)phenyl]-2-propylammonium hydrogen maleate with the numbering scheme.

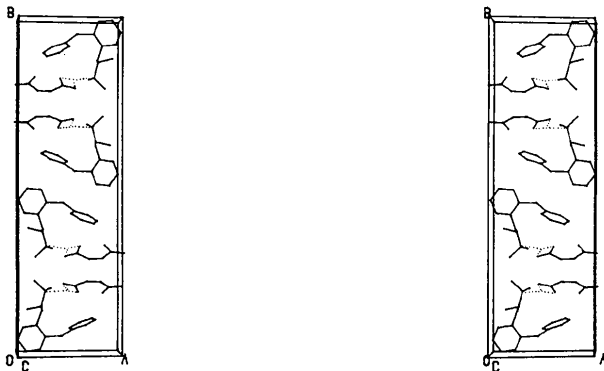


Fig. 2. Stereoscopic packing diagram of *N,N*-dimethyl-1-[2-(phenylthio)phenyl]-2-propylammonium hydrogen maleate. View down the shortest, *c*, axis shows isolated ion pairs. The pairs mutually interact only through nonspecific van der Waals forces. H atoms are omitted. Dotted lines are hydrogen-bonded contacts.

Table 2. Selected bond angles (Å), bond angles (°) and torsion angles (°)

S1—C2	1.777 (3)	N11—H111	0.961 (28)
S1—C14	1.765 (2)	O20—C21	1.235 (3)
C7—C8	1.511 (3)	O27—C21	1.268 (4)
C8—C9	1.513 (4)	O25—C24	1.217 (3)
C9—C10	1.511 (4)	O26—C24	1.290 (3)
C9—N11	1.531 (3)	C21—C22	1.484 (4)
N11—C12	1.488 (4)	C23—C24	1.477 (5)
N11—C13	1.490 (4)	C22—C23	1.333 (4)
C2—S1—C14	105.6 (1)	C8—C9—N11	108.5 (2)
S1—C2—C3	117.0 (2)	C9—N11—C12	116.2 (2)
S1—C2—C7	122.6 (2)	C9—N11—C13	111.5 (2)
S1—C14—C15	124.9 (2)	O20—C21—O27	123.2 (3)
S1—C14—C19	116.5 (2)	O25—C24—O26	122.1 (3)
C3—C2—C7	120.3 (2)	O20—C21—C22	117.4 (3)
C15—C14—C19	118.6 (2)	O25—C24—C23	118.6 (3)
C2—C7—C6	117.7 (2)	C21—C22—C23	130.8 (3)
C2—C7—C8	123.9 (2)	C22—C23—C24	131.4 (3)
C7—C8—C9	114.2 (2)	C22—C23—H231	118 (2)
C8—C9—C10	115.3 (3)	C23—C22—H221	117 (2)
C14—S1—C2—C3	-108.6 (2)	C8—C9—N11—H111	38 (2)
C14—S1—C2—C7	75.7 (2)	C10—C9—N11—C12	49.6 (3)
C2—S1—C14—C15	14.6 (3)	O20—C21—C22—C23	-175.0 (3)
C2—S1—C14—C19	-167.1 (2)	O27—C21—C22—C23	4.5 (5)
S1—C14—C15—C16	179.9 (2)	O25—C24—C23—C22	-179.3 (3)
S1—C2—C7—C8	-6.1 (3)	O26—C24—C23—C22	-0.3 (5)
C2—C7—C8—C9	80.7 (3)	C21—C22—C23—C24	-2.6 (6)
C7—C8—C9—C10	50.9 (3)	C23—C24—O26—H261	1 (2)
C7—C8—C9—N11	175.8 (2)	C21—C22—C23—H231	174 (2)
C8—C9—N11—C12	-78.0 (3)	C24—C23—C22—H221	179 (2)
C8—C9—N11—C13	-155.8 (2)		

relieves steric repulsion between the carboxy groups and helps to keep the molecule planar. The planarity is probably energetically advantageous owing to π -electron delocalization.

The diphenyl sulfide skeleton of the *N,N*-dimethyl-1-[2-(phenylthio)phenyl]-2-propylammonium cation has all the characteristic features of diphenyl sulfide molecules (Schneider, Rejholec & Kuchař, 1990) as found by statistical analysis of geometries retrieved from the Cambridge Structural Database (Allen *et al.*, 1979). The dihedral angle between the mean planes of the two phenyl rings is 78.76 (9)° and the torsion angles at the S—C bonds are C7—C2—S1—C14 75.7 (2) and C2—S1—C14—C15 14.6 (3)°.

Both phenyl rings are almost planar ($\chi^2 = 12.5$ and 39.0), but the atom S1 is displaced from the mean planes by distances of 0.1438 (8) and 0.0167 (8) Å respectively.

The atoms C7, C8, C9, N11 lie almost in a plane which is inclined to the mean plane of the first phenyl ring (C2—C7 inclusive) by an angle of 78.7 (2)°. The C12 and C13 atoms break an all-*trans* arrangement of the dimethylaminoisopropyl side chain presumably owing to the steric requirements of the N11—H111...O20, O27 hydrogen bond. Orientation of H111 in the direction of the O20 and O27 atoms determines the torsion angles of the atoms C12 and C13 (see Table 2).

The structure contains a racemic mixture of *R*- and *S*-forms of the ammonium cation (the chiral atom is C9).

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Structure of a Guaianolide Lactone Glucoside

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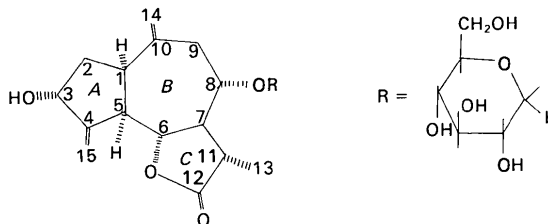
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Abstract. (3*S*)-(3 α ,3 $\alpha\alpha$,4 α ,6 $\alpha\alpha$,8 α ,9 $\alpha\alpha$,9 β)-4-(β -D-Glucopyranosyloxy)decahydro-8-hydroxy-3-methyl-6,9-bis(methylene)azuleno[4,5-*b*]furan-2(3*H*)-one, C₂₁H₃₀O₉, m.p. = 517–518 K, M_r = 426.47, orthorhombic, $P2_12_12_1$, a = 7.093 (1), b = 13.365 (1), c = 21.557 (6) Å, V = 2043.6 (7) Å³, Z = 4, D_x = 1.386 Mg m⁻³, λ (Cu $K\alpha$) = 1.5418, μ = 0.86 mm⁻¹, $F(000)$ = 912, T = 293 K, final R = 0.031 for 1501 observed reflections. The *AB* and *BC* junctions are *cis* and *trans* respectively. C(3)—OH is substituted in the α position. The molecules are linked by hydrogen bonds.

Introduction. *Saussurea involucrata* Kar. et. Kin (Chinese name, Xue Lian Hua) is a Chinese herb, which is distributed mainly in Xinjiang province of China. It has been used for the treatment of rheumatic arthritis and gynopathy. A sesquiterpene lactone glucoside, C₂₁H₃₀O₉, was isolated from this plant. A very similar guaianolide has been characterized previously by NMR studies (Das, Baruah, Sharma, Baruah, Kulanthaivel & Hert, 1983), but this natural compound differs from ours in the configuration at C(3). Other guaianolide structures and closely related structures have also been reported (Declercq, Ger-

main, Van Meerssche, Demuynek, De Clercq & Vandewalle, 1980; McPhail & Sim, 1973; Asskawa, Taira, Toyota, Takemoto, Hert & Sakai, 1981; Hert, Watanabe & Blount, 1982; Declercq, Germain, Van Meerssche, Kok, De Clercq & Vandewalle, 1980). Though these structures differ from ours in some ways, these structural descriptions and features are helpful to further understand the structure of our compound.



Experimental. A crystal of dimensions 0.19 × 0.15 × 0.11 mm was mounted on an Enraf–Nonius CAD-4 diffractometer, graphite-monochromatized Cu $K\alpha$ radiation. Cell dimensions were obtained by least squares from 25 accurately centered reflections in the range 20–35°. A total of 1781 unique reflections (R_{int} = 0.021) with $4 \leq 2\theta \leq 120^\circ$ were collected at room temperature (293 K). Experimental conditions were:

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