

2-(3-Hydroxy-4-methoxyphenyl)-*N,N,N*-tri-
methylethanaminium chlorideMai Efdi,^a Mamoru Koketsu,^{b*}
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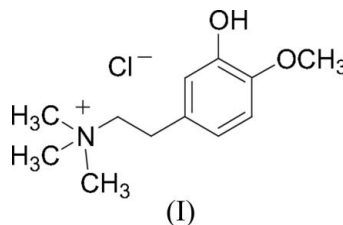
Key indicators

Single-crystal X-ray study
T = 296 K
Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$
R factor = 0.073
wR factor = 0.119
Data-to-parameter ratio = 19.3For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.The title compound, also known as salicifoline chloride, $\text{C}_{12}\text{H}_{20}\text{NO}_2^+\cdot\text{Cl}^-$, was isolated from *Enicosanthe membranifolium* J. Sinclair for X-ray analysis. Salicifoline is hydrogen bonded to a chloride ion *via* the OH group.

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Comment

Salicifoline [systematic name: 2-(3-hydroxy-4-methoxyphenyl)-*N,N,N*-trimethylethanamine] shows curare-like actions on the extirpated rectus abdominis muscles of frogs and on the sciatic skeletal muscle of rats *in situ* (Inoue, 1957; Shimamoto *et al.*, 1958). Salicifoline chloride was isolated from *Magnolia salicifolia* grown in Japan (Tomita & Nakano, 1952), but the crystal structure has not previously been determined. In order to study structure–biological activity relationships, it is essential to determine the conformation of salicifoline chloride. Salicifoline chloride has been isolated from an *n*-butanol extract of the *Enicosanthe membranifolium* J. Sinclair. This is the first report of the isolation of salicifoline chloride, (I), from this plant.The structure of (I) is shown in Fig. 1. The geometric parameters of the salicifoline cation are very similar to those observed in salicifoline picrate (Wang *et al.*, 1991). The salicifoline cation in (I) is hydrogen bonded to a chloride ion *via* the OH group (Table 1). In salicifoline picrate, salicifoline forms a hydrogen bond between the OH group and the O^- of the picrate. The difference between the salicifoline cations in (I) and in salicifoline picrate is the conformation of the OH group. The H atom in (I) points away from the OCH_3 group whereas in salicifoline picrate the H atom of the OH group points towards the methoxy O atom.

Experimental

A methanol extract (369 g) of *Enicosanthe membranifolium* J. Sinclair branches was partitioned successively with *n*-hexane, ethyl acetate and *n*-butyl alcohol. The *n*-BuOH extract (79 g) was dissolved in MeOH (500 ml) and then it was acidified with 5% acetic acid (500 ml) and extracted with EtOAc. The acidic aqueous solution was made alkaline with NaHCO_3 and extracted with *n*-BuOH (5 ×

500 ml) to yield a crude alkaloid mixture (40 g) which was fractionated by a silica gel 60 (Cica-reagent, 40–50 μm) column (in diameter 6 \times 50 cm, 500 g) and eluted with acetone–methanol (stepwise). This fractionation processing gave six fractions (A to F). Salicifoline chloride (800 mg) was crystallized spontaneously from fraction C (1.6 g) as colorless prismatic crystals.

Crystal data

$\text{C}_{12}\text{H}_{20}\text{NO}_2^+\cdot\text{Cl}^-$
 $M_r = 245.74$
 Monoclinic, $P2_1/c$
 $a = 9.758$ (5) \AA
 $b = 11.686$ (6) \AA
 $c = 11.253$ (6) \AA
 $\beta = 90.180$ (7) $^\circ$
 $V = 1283.2$ (12) \AA^3

$Z = 4$
 $D_x = 1.272$ Mg m^{-3}
 Mo $K\alpha$ radiation
 $\mu = 0.28$ mm^{-1}
 $T = 296$ (2) K
 Prism, colorless
 $0.46 \times 0.17 \times 0.11$ mm

Data collection

Rigaku/MSC Mercury CCD diffractometer
 ω scans
 Absorption correction: integration (NUMABS; Higashi, 1999)
 $T_{\text{min}} = 0.919$, $T_{\text{max}} = 0.975$

10211 measured reflections
 2928 independent reflections
 2472 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.043$
 $\theta_{\text{max}} = 27.5^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.073$
 $wR(F^2) = 0.119$
 $S = 1.25$
 2928 reflections
 152 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0217P)^2 + 0.8281P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.17$ e \AA^{-3}
 $\Delta\rho_{\text{min}} = -0.19$ e \AA^{-3}

Table 1

Hydrogen-bond geometry (\AA , $^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O2}-\text{H7}\cdots\text{Cl1}$	0.88 (4)	2.15 (4)	3.017 (2)	166 (3)

C-bound H atoms were placed in idealized positions and treated as riding atoms with C–H distances in the range 0.93–0.97 \AA and with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl H atoms or $1.2U_{\text{eq}}(\text{C})$ for the remaining H atoms. The methyl groups were allowed to rotate but not to tip. The positional parameters of the H atom on the O atom were refined with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$.

Data collection: *CrystalClear* (Molecular Structure Corporation & Rigaku, 2001); cell refinement: *CrystalClear*; data reduction: *TEXSAN* (Rigaku/MSC, 2004); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII*

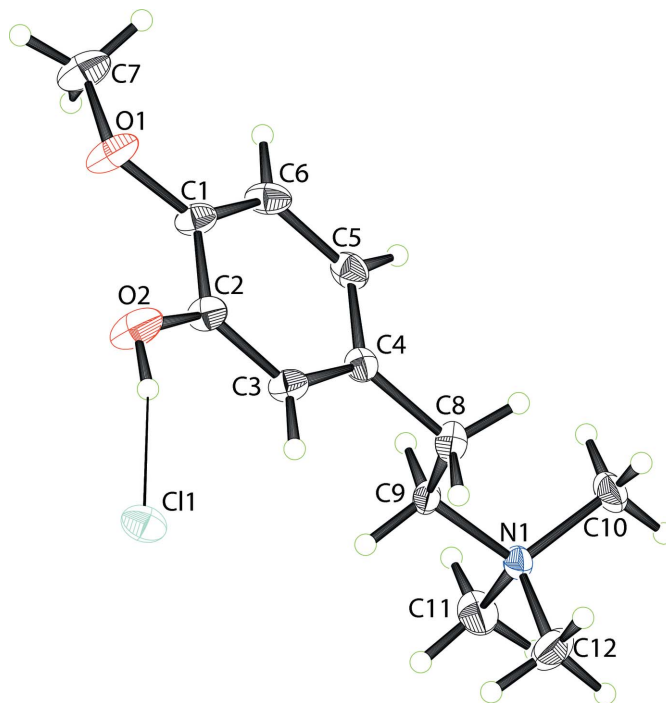


Figure 1

The molecular structure of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii. The hydrogen bond is drawn as a thin line.

(Johnson, 1976); software used to prepare material for publication: *SHELXL97* and *TEXSAN*.

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