Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# 4-Ammonio-5-chloro-*N*-[2-(*N*,*N*-diethylammonio)ethyl]-2-methoxybenzamide sulfate

**C. R. Girija and Noor Shahina Begum***

Department of Studies in Chemistry, Central College Campus, Bangalore University, Bangalore 560 001, Karnataka, India

Correspondence e-mail: noorsb@rediffmail.com

Received 9 December 2003
Accepted 22 December 2003
Online 23 January 2004

**Key indicators**

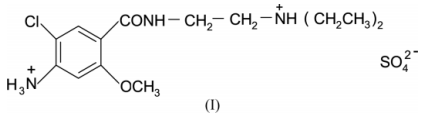
Single-crystal X-ray study
*T* = 293 K
Mean σ(C–C) = 0.006 Å
*R* factor = 0.057
*wR* factor = 0.178
Data-to-parameter ratio = 12.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_{14}H_{24}ClN_3O_2^{2+}\cdot SO_4^{2-}$, also known as metoclopramide sulfate, is non-planar, both the molecular and crystal structures being stabilized by N—H···O and C—H···O hydrogen bonds.

## Comment

The title compound, (I), a substituted benzamide, is a dopamine antagonist clinically used as neuroleptic drug (Pitre & Stradi, 1987). It is also a potential ligand, owing to its significant tendency to coordinate to metals. Its crystal structure is reported here.




(I)

In the title molecule (Fig. 1), the C=O group is almost *cis* with respect to the C1—C6 bond, the torsion angle C6—C1—C11—O12 being −5.5 (5)°. The methoxy group is coplanar with the attached ring. A *gauche* conformation is observed in an amino ethyl side chain. The geometrical parameters of the molecule are comparable with those in a similar structure (Blaton & Peeters, 1980). An intramolecular hydrogen bond is formed between methoxy O atom O7 and amide H atom H13 (Table 2). This hydrogen bond creates a virtual ringe which may be a key feature for the binding of neuroleptic benzamides to the dopamine receptor (van de Waterbeemd & Testa, 1981). The molecular structure is further stabilized by N16—H16···O12 and C6—H6···O12 intramolecular hydrogen bonds. In the crystal structure, the cation and anions

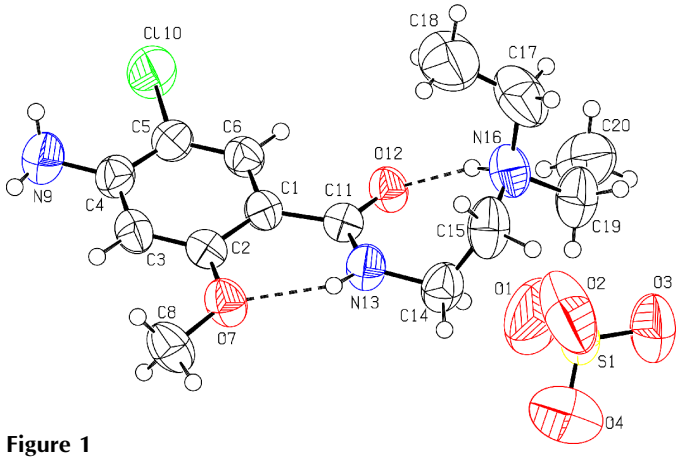



**Figure 1**
Molecular structure of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme.

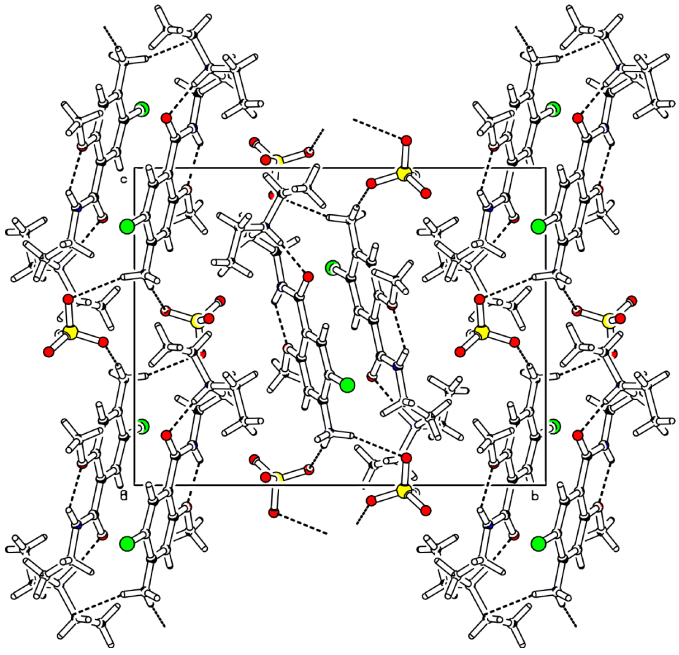


**Figure 2**
Packing of the title molecules, viewed down the *a* axis.

are involved in N—H···O and C—H···O hydrogen bond (Table 2). Further, Cl10···O12($1-x$, $1-y$, $1-z$) short contacts [3.262 (3) Å] link inversion-related molecules into a dimer (Fig. 2). A short H9*C*···Cl10 (2.90 Å) interaction is also observed.

## Experimental

The title compound (98% pure) was bought from IPCA laboratories. Single crystals were grown from a methanol solution by slow evaporation at room temperature.

*Crystal data*

$C_{14}H_{24}ClN_3O_2^{2+}\cdot SO_4^{2-}$
$M_r$ = 397.88
Monoclinic, $P2_1/c$
$a$ = 9.1514 (2) Å
$b$ = 16.5218 (3) Å
$c$ = 12.8524 (3) Å
$\beta$ = 97.186 (1)°
$V$ = 1927.99 (7) Å$^3$
$Z$ = 4
$D_x$ = 1.371 Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 7814 reflections
$\theta$ = 2.0–23.3°
$\mu$ = 0.34 mm$^{-1}$
$T$ = 293 (2) K
Prism, colourless
0.30 × 0.25 × 0.10 mm

*Data collection*

Bruker SMART CCD area-detector diffractometer
$\omega$ scans
Absorption correction: none
7814 measured reflections
2768 independent reflections
1932 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.034
$\theta_{max}$ = 23.3°
$h = -10 \rightarrow 10$
$k = -14 \rightarrow 18$
$l = -13 \rightarrow 14$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.179
$S$ = 1.03
2768 reflections
227 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0961P)^2 + 1.029P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.004
$\Delta\rho_{max}$ = 0.56 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.28 e Å$^{-3}$
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0074 (19)

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| S1—O1 | 1.360 (5) | O7—C8 | 1.428 (5) |
| S1—O2 | 1.372 (4) | C11—O12 | 1.251 (4) |
| S1—O4 | 1.389 (5) | C11—N13 | 1.325 (4) |
| S1—O3 | 1.390 (3) | N13—C14 | 1.456 (5) |
| C1—C11 | 1.485 (5) | C15—N16 | 1.504 (6) |
| C2—O7 | 1.350 (4) | N16—C17 | 1.486 (6) |
| C4—N9 | 1.376 (5) | N16—C19 | 1.532 (6) |
| C5—Cl10 | 1.748 (4) | | |
| C2—O7—C8 | 119.5 (3) | C17—N16—C19 | 111.6 (4) |
| C11—N13—C14 | 123.4 (3) | C15—N16—C19 | 109.5 (4) |
| C17—N16—C15 | 114.4 (4) | | |
| C3—C2—O7—C8 | 5.4 (5) | C14—C15—N16—C17 | 150.6 (4) |
| C6—C1—C11—O12 | −5.5 (5) | C14—C15—N16—C19 | −83.3 (4) |
| C1—C11—N13—C14 | 174.7 (4) | C15—N16—C17—C18 | −73.7 (6) |
| C11—N13—C14—C15 | 73.5 (5) | C15—N16—C19—C20 | 159.5 (5) |
| N13—C14—C15—N16 | −78.0 (5) | | |

**Table 2**
Hydrogen-bonding geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N13—H13···O7 | 0.86 | 2.00 | 2.658 (4) | 132 |
| N16—H16···O12 | 0.91 | 1.79 | 2.665 (4) | 160 |
| C6—H6···O12 | 0.93 | 2.39 | 2.735 (4) | 102 |
| N9—H9*A*···O2$^{i}$ | 0.89 | 2.45 | 3.325 (7) | 169 |
| N9—H9*B*···O3$^{ii}$ | 0.89 | 2.51 | 3.372 (6) | 164 |

Symmetry codes: (i) $2-x, 1-y, 1-z$; (ii) $x, y, 1+z$.

H atoms were placed at calculated positions (N—H = 0.86–0.91 Å and C—H = 0.93–0.97 Å) and allowed to ride on the atoms to which they are bonded; $U_{iso}$(H) values were set at 1.5$U_{eq}$(parent) for $CH_3$ and $NH_3$ H atoms and 1.2$U_{eq}$(parent) for the remaining H atoms. The highest peak and deepest hole in the final difference map were located at 1.39 and 1.09 Å, respectively, from atom O1. The large $U_{eq}$ values for atoms O1, O2 and O4 indicate a possible disorder in the sulfate anion.

Data collection: *SMART* (Siemens, 1993); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 1990); software used to prepare material for publication: *SHELXL*97.

CRG thanks UGC–FIP for a Teacher fellowship. The authors thank Dr G. U. Kulkarni, JNCASR, for data collection.

## References

Blaton, N. M., Peeters, O. M. & De Ranter. C. J. (1980). *Cryst. Struct. Commun.* **9**, 857–864.
Pitre, D. & Stradi, R. (1987). *Anal. Profiles Drug Subst.* **16**, 327–360.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Siemens (1993). *SMART*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Siemens (1995). *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.
Waterbeemd, H. van de & Testa, B. (1981). *Helv. Chim. Acta*, **64**, 2183–2185.