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Key indicators

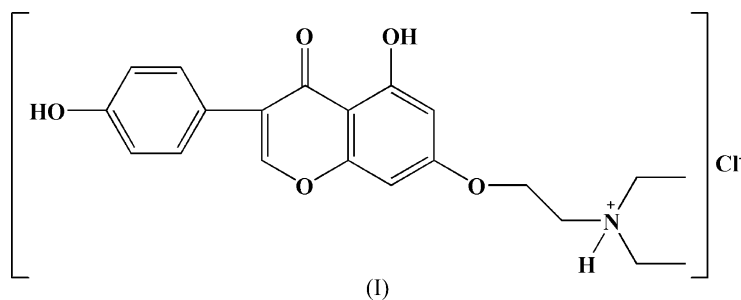
Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.007$ Å
 R factor = 0.075
 wR factor = 0.225
Data-to-parameter ratio = 13.8For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.Diethyl[2-[5-hydroxy-3-(4-hydroxyphenyl)-4-oxo-4*H*-chromen-7-yloxy]ethyl]ammonium chloride

The asymmetric unit of the title compound, $\text{C}_{21}\text{H}_{24}\text{NO}_5^+\cdot\text{Cl}^-$, contains two crystallographically independent cations and two chloride ions. The crystal structure is stabilized by intramolecular $\text{O}-\text{H}\cdots\text{O}$, and intermolecular $\text{N}-\text{H}\cdots\text{Cl}$ and $\text{O}-\text{H}\cdots\text{Cl}$ hydrogen bonds.

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Comment

Genistein [5,7-dihydroxy-3-(4-hydroxyphenyl)chromen-4-one] is reported to have many biological activities (Wang *et al.*, 2006; Chacko *et al.*, 2005; Mayr *et al.*, 2005; Record *et al.*, 1995; Ralph *et al.*, 1995; Mastuda *et al.*, 2002; Ulanowska *et al.*, 2006; Barnes *et al.*, 1995). The simplicity of genistein, along with its interesting anticancer activity (Barnes *et al.*, 1995), offers promise for the rational design of new chemotherapeutic agents. As a part of our studies on pharmacologically active genistein derivatives, we have synthesized the title compound, (I), and determined its structure in order to establish its conformation (Fig. 1).



The title compound contains two crystallographically independent cations and two chloride ions (labeled cat1 for the cation containing N1 and cat2 for the cation containing N2). All bond lengths and angles are in normal ranges. The dihedral angles between the benzene and chromone rings in cat1 is $41.96(16)^\circ$, which is essentially the same as in cat2 [$41.71(17)^\circ$]. The diethylammonium arms in both cations are in a *gauche* conformation. There are intramolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds, as well as intermolecular $\text{N}-\text{H}\cdots\text{Cl}$ and $\text{O}-\text{H}\cdots\text{Cl}$ hydrogen bonds in the packing network (Table 1 and Fig. 2).

Experimental

Diethylamine (0.37 g, 5 mmol) was added to a solution of 4',5-dihydroxy-7-(2-bromoethoxy)isoflavone (0.37 g, 1 mmol) in anhydrous DMF (5 ml) and heated at 353 K for 1 h until the starting material disappeared, after which ice-water was added dropwise. The mixture was then filtered, washed with water, dried over Na_2SO_4 and concentrated. The residue was purified with a silica-gel column and eluted with $\text{CH}_2\text{Cl}_2/\text{CH}_3\text{OH}$ (9:1) to afford the genistein derivative

(yield 0.30 g, 81%; m.p. 489–491 K). The purified product was dissolved in HCl–water (1:20) and single crystals were obtained after 6 d. Analysis found: C 68.40, H 6.19, N 3.90%; calculated for $C_{21}H_{24}ClNO_5$: C 68.28, H 6.28, N 3.79%.

Crystal data

$C_{21}H_{24}NO_5 \cdot Cl^-$ $V = 2041.2 (7) \text{ \AA}^3$
 $M_r = 405.86$ $Z = 4$
 Triclinic, $P\bar{1}$ $D_x = 1.321 \text{ Mg m}^{-3}$
 $a = 7.4420 (15) \text{ \AA}$ Mo $K\alpha$ radiation
 $b = 15.092 (3) \text{ \AA}$ $\mu = 0.22 \text{ mm}^{-1}$
 $c = 18.353 (4) \text{ \AA}$ $T = 293 (2) \text{ K}$
 $\alpha = 89.48 (3)^\circ$ Plate, colourless
 $\beta = 82.20 (3)^\circ$ $0.40 \times 0.30 \times 0.10 \text{ mm}$
 $\gamma = 88.21 (3)^\circ$

Data collection

Bruker SMART CCD area-detector 8658 measured reflections
 diffractometer 8003 independent reflections
 φ and ω scans 4500 reflections with $I > 2\sigma(I)$
 Absorption correction: multi-scan $R_{int} = 0.027$
 (SADABS; Bruker, 1998) $\theta_{max} = 26.0^\circ$
 $T_{min} = 0.918, T_{max} = 0.979$

Refinement

Refinement on F^2 $w = 1/[\sigma^2(F_o^2) + (0.0959P)^2 + 2.9117P]$
 $R[F^2 > 2\sigma(F^2)] = 0.075$ where $P = (F_o^2 + 2F_c^2)/3$
 $wR(F^2) = 0.225$ $(\Delta/\sigma)_{max} < 0.001$
 $S = 1.02$ $\Delta\rho_{max} = 0.94 \text{ e \AA}^{-3}$
 7185 reflections $\Delta\rho_{min} = -0.75 \text{ e \AA}^{-3}$
 521 parameters
 H atoms treated by a mixture of independent and constrained refinement

Table 1

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

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$N2-H2B \cdots Cl1^i$	0.78 (5)	2.36 (6)	3.130 (5)	175 (6)
$N1-H1B \cdots Cl2^i$	0.96 (6)	2.14 (6)	3.101 (5)	172 (5)
$O4-H4 \cdots O3$	0.82	1.84	2.566 (4)	147
$O9-H9 \cdots O8$	0.82	1.86	2.591 (4)	148
$O6-H6A \cdots Cl2^{ii}$	0.82	2.25	3.064 (5)	174
$O1-H1 \cdots Cl1^{ii}$	0.82	2.28	3.092 (4)	174

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

All the H atoms, except for H1B and H2B attached to N1 and N2, respectively, were placed in idealized positions ($C-H = 0.96 \text{ \AA}$, $N-H = 0.90-0.98 \text{ \AA}$, and $O-H = 0.91$ and 1.01 \AA) and refined as riding, with $U_{iso}(H) = 1.2U_{eq}(C)$, $1.5U_{eq}(O)$ and $1.5U_{eq}(N)$. Atoms H1B and H2B were located in a difference Fourier map and their positional coordinates were refined. In the refinement, the instructions SIMU 0.005 0.01 4.0 \$C, and OMIT 0 -3 2, OMIT 0 3 0, OMIT 0 0 1, and OMIT -3.00 50.00 were used to improve the data quality.

Data collection: SMART (Bruker, 1998); cell refinement: SAINT (Bruker, 1998); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997a); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997a); molecular graphics: SHELXTL (Sheldrick, 1997b); software used to prepare material for publication: SHELXL97.

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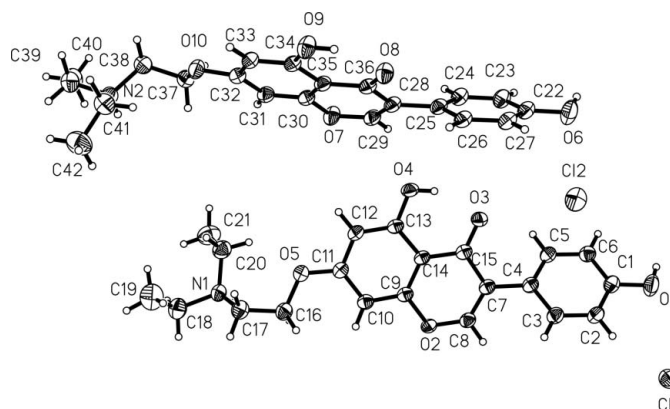


Figure 1 The asymmetric unit of (I), showing 35% probability displacement ellipsoids (arbitrary spheres for the H atoms).

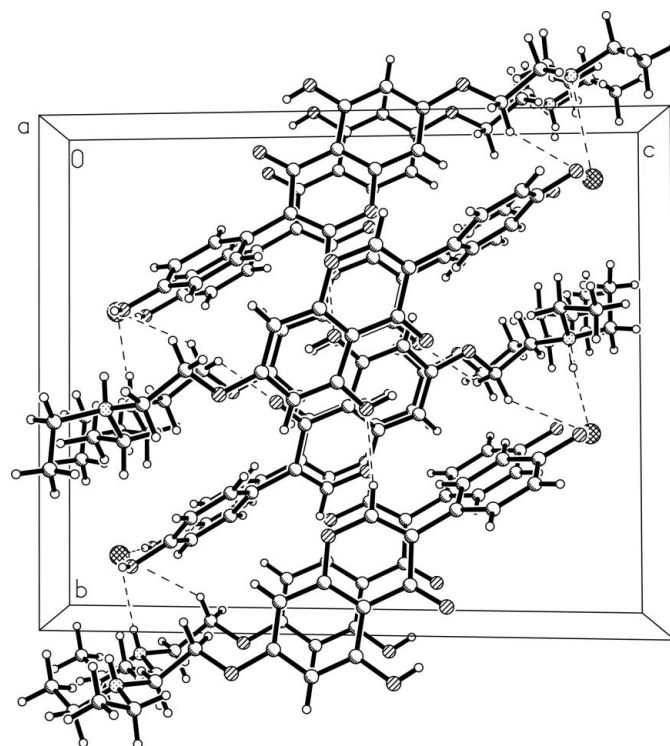


Figure 2 The crystal packing of (I), viewed along the a axis. Hydrogen bonds are shown as dashed lines.

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