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1,2,3,4-Tetrahydroquinolinium hydrogen chloranilate

Hiroyuki Ishida

Department of Chemistry, Faculty of Science, Okayama University, Okayama 700-8530, Japan

Correspondence e-mail:
ishidah@cc.okayama-u.ac.jp

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Key indicators

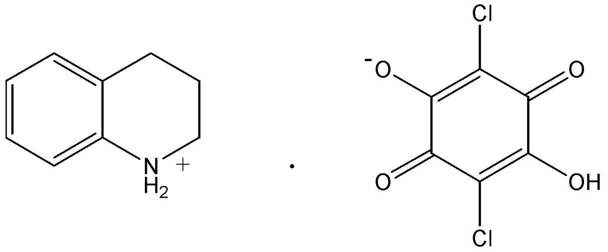
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.042
wR factor = 0.121
Data-to-parameter ratio = 16.1

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

In the title compound, $C_9H_{12}N^+\cdot C_6HCl_2O_4^-$, two chloranilate ions are connected by O—H···O hydrogen bonds to form a dimeric unit. The 1,2,3,4-tetrahydroquinolinium ions are linked on both sides of the dimer *via* an N—H···O hydrogen bond to give a 2:2 complex of chloranilic acid and tetrahydroquinoline. The 2:2 complexes are held together by an N—H···O hydrogen bond and a π–π stacking interaction to give a molecular tape.

Comment

Crystal structures in the chloranilic acid (2,5-dichloro-3,6-dihydroxy-1,4-benzoquinone)–secondary amine system have been analysed as 1:1 salts of morpholine (Ishida & Kashino, 1999), diethylamine (Ishida & Kashino, 2000) and piperidine (Fukunaga & Ishida, 2003), and as a 1:2 salt of pyrrolidine (Ishida, 2004).



(I)

In the present study, we have prepared the 1:1 salt, (I), of tetrahydroquinoline and determined its crystal structure in order to extend the previous examination. Two chloranilate ions related by a twofold rotation axis are connected by O—H···O hydrogen bonds (Table 1) and form a dimeric unit as observed in the diethylammonium and lutidinium salts (Ishida & Kashino, 2002). Atom H1 is also involved in an intramolecular hydrogen bond with O1. The 1,2,3,4-tetrahydro-

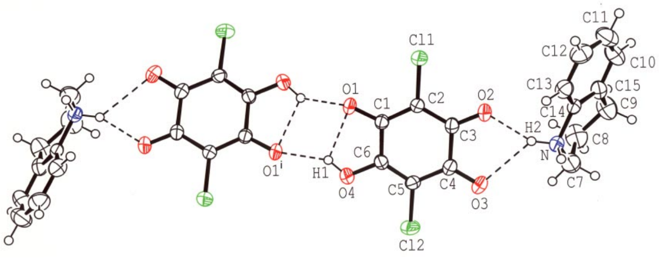


Figure 1
ORTEP-3 (Farrugia, 1997) drawing of the 2:2 complex of (I) with the atom-labeling. Displacement ellipsoids of non-H atoms are drawn at the 50% probability level. O—H···O and N—H···O hydrogen bonds are indicated by dashed lines [symmetry code: (i) $1 - x, y, \frac{3}{2} - z$].

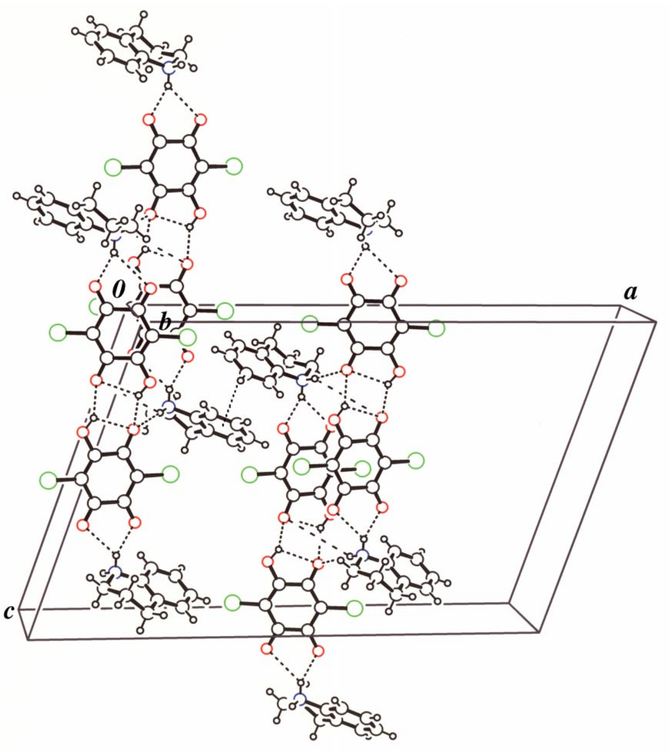


Figure 2
Packing diagram showing molecular tapes formed *via* O—H···O and N—H···O hydrogen bonds (shown as dashed lines). A C—H···π interaction between the molecular layers is also shown as a dashed line.

quinolinium ions are linked on both sides of the dimer *via* a bifurcated hydrogen bond of N—H2···O2 and N—H2···O3, forming a 2:2 complex of chloranilic acid and tetrahydroquinoline (Fig. 1). The 2:2 complexes are held together by an N—H3···O1^{ii} hydrogen bond (Table 1) and a π–π stacking interaction between the chloranilate ions related by an inversion center, the interplanar and centroid–centroid separations being 3.351 (2) and 3.6980 (17) Å, respectively, giving a molecular tape running along the *c* axis (Fig. 2). The molecular tapes are stacked along the *b* axis to form a layer extending parallel to the *bc* plane. Between neighboring layers, a C—H···π interaction [H12···Cg^{iii} = 2.85 Å, C12···Cg^{iii} = 3.646 (3) Å and C12—H12···Cg^{iii} = 144°, where *Cg* denotes the centroid of benzene ring C10–C15; symmetry code: (iii) $\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$] is observed.

Experimental

Crystal of (I) were obtained by slow evaporation of an acetonitrile solution of 1,2,3,4-tetrahydroquinoline and chloranilic acid in a 1:1 molar ratio.

Crystal data

$C_9H_{12}N^+\cdot C_6HCl_2O_4^-$
M_r = 342.16
Monoclinic, $C2/c$
a = 25.840 (8) Å
b = 7.0633 (10) Å
c = 17.107 (5) Å
β = 108.79 (3)°
V = 2955.9 (14) Å^3
Z = 8
D_x = 1.538 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 11.5–12.5°
μ = 0.46 mm^{-1}
T = 298 K
Prism, brown
0.40 × 0.30 × 0.30 mm

Data collection

Rigaku AFC-5*R* diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.783, T_{max} = 0.872
3771 measured reflections
3395 independent reflections
2498 reflections with $I > 2\sigma(I)$
R_{int} = 0.015
θ_{max} = 27.5°
$h = -2 \rightarrow 33$
$k = 0 \rightarrow 9$
$l = -22 \rightarrow 21$
3 standard reflections every 97 reflections intensity decay: 0.2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.121
S = 1.05
3395 reflections
211 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.067P)^2 + 1.1P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.61 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O4—H1···O1	0.88 (5)	2.18 (4)	2.630 (2)	111 (4)
O4—H1···O1^{i}	0.88 (5)	1.90 (5)	2.693 (2)	148 (4)
N—H2···O2	0.88 (3)	1.91 (3)	2.733 (2)	154 (3)
N—H2···O3	0.88 (3)	2.51 (3)	3.173 (3)	132 (2)
N—H3···O1^{ii}	0.90 (3)	2.01 (3)	2.883 (3)	166 (3)

Symmetry codes: (i) $1 - x, y, \frac{3}{2} - z$; (ii) $x, 1 - y, z - \frac{1}{2}$.

H atoms attached to the N and O atoms were refined isotropically. Other H atoms were treated as riding atoms, with C—H = 0.97 and 0.93 Å for methylene and aromatic H atoms, respectively. U_{iso}(H) values were set at $1.5U_{eq}$(C) for the methylene H atoms and at $1.2U_{eq}$(C) for the aromatic H atoms.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1990); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN for Windows* (Molecular Structure Corporation, 1997–1999); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 2003).

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