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meso-1,2-(2-Hydroxyphenyl)-1,2-ethylenediammonium dichloride

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Key indicators

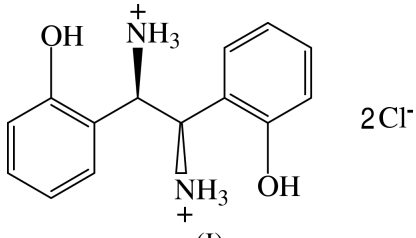
Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.002 Å
R factor = 0.026
wR factor = 0.068
Data-to-parameter ratio = 16.9

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

In the crystal structure of the title compound, $[(HOC_6H_4CH(NH_3)CH(NH_3)C_6H_4OH]Cl_2$ or $C_{14}H_{18}N_2O_2^{2+}\cdot 2Cl^-$, the dication lies about an inversion center at the midpoint of the central C—C bond. The asymmetric unit forms one intramolecular N—H···O hydrogen bond, with an N···O distance of 2.7759 (14) Å, three intermolecular N—H···Cl hydrogen bonds, with N···Cl distances ranging from 3.1343 (10) to 3.3343 (10) Å, and one intermolecular O—H···Cl hydrogen bond, with an O···Cl distance of 3.0142 (9) Å.

Comment

meso-1,2-(2-Hydroxyphenyl)-1,2-ethylenediamine (Vogtle & Goldschmidt, 1976), (I), was synthesized in order to prepare its complex with copper(II). This complex could be used as a sensor of chirality for amino acids and chiral diamines using circular dichroism. Complexation of the ligand to the metal, however, did not occur and the resulting clear and colourless crystals were of the diammonium dichloride salt.



(I)

Experimental

To a stirred solution of *meso*-diphenoxyethylenediamine (100 mg, 1.1 equivalents) in methanol (1 ml) was added copper dichloride dihydrate (63 mg, 1.0 equivalents). The mixture was stirred at room temperature overnight. The insoluble excess ligand was removed by vacuum filtration. The residue was dissolved in H_2O and was then set aside for 2 weeks for crystallization. During this period colourless crystals, suitable for X-ray diffraction, precipitated. ^{1}H NMR ($CDCl_3$): δ 7.21 (*m*, 2H, Ph), 7.14 (*m*, 2H, Ph), 6.93 (*m*, 2H, Ph), 6.88 (*m*, 2H, Ph), 4.29 (*s*, 2H, CH), 1.55 p.p.m. (*br*, 4H, NH_2).

Crystal data

$C_{14}H_{18}N_2O_2^{2+}\cdot 2Cl^-$
M_r = 317.20
Trigonal, $R\overline{3}$
a = 21.8715 (7) Å
c = 7.8297 (3) Å
V = 3243.64 (19) Å^3
Z = 9
D_x = 1.461 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1380 reflections
θ = 2.5–27.5°
μ = 0.45 mm^{-1}
T = 150 (1) K
Needle, colourless
0.25 × 0.12 × 0.10 mm

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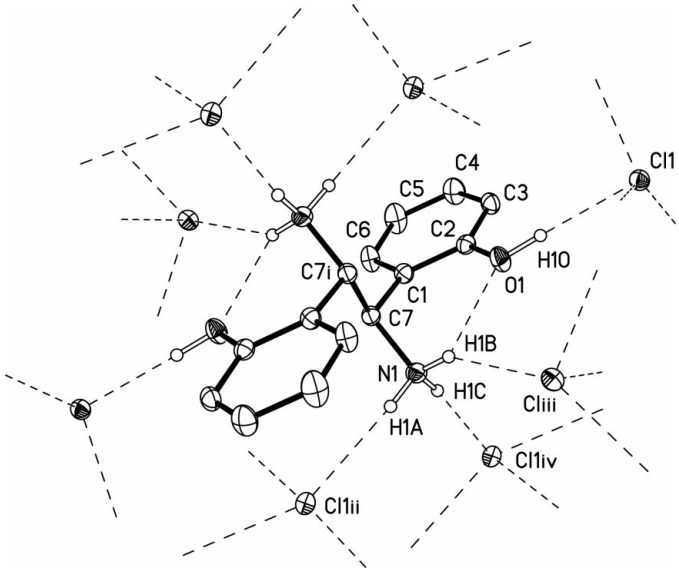

Figure 1
The structure of (I), with the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms bonded to C atoms have been removed for clarity. Hydrogen bonds are shown as dashed lines. See footnotes to Tables 1 and 2 for equivalent positions corresponding to the symmetry codes in the figure.

Data collection

Nonius KappaCCD diffractometer
φ and ω scans with κ offsets
Absorption correction: multi-scan (*DENZO-SMN*; Otwinowski & Minor, 1997)
T_{min} = 0.895, T_{max} = 0.956
4231 measured reflections
1640 independent reflections
1538 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 27.5°
$h = -28 \rightarrow 28$
$k = -28 \rightarrow 29$
$l = -7 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.068
S = 1.06
1640 reflections
97 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0097P)^2 + 3.9792P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0020 (6)

Table 1
Selected geometric parameters (Å, °).

O1—C2	1.3607 (14)	C7—N1	1.5070 (15)
C1—C7	1.5108 (15)	C7—C7^{i}	1.545 (2)
O1—C2—C3	123.05 (10)	N1—C7—C7^{i}	107.59 (11)
O1—C2—C1	116.72 (10)	C1—C7—C7^{i}	112.32 (12)

Symmetry code: (i) $1-x, 1-y, 1-z$.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···Cl1^{ii}	0.91	2.53	3.3344 (10)	148
N1—H1B···O1	0.91	2.28	2.7759 (14)	114
N1—H1B···Cl1^{iii}	0.91	2.55	3.3724 (10)	150
N1—H1C···Cl1^{iv}	0.91	2.25	3.1343 (10)	163
O1—H1O···Cl1	0.91 (2)	2.20 (2)	3.1042 (9)	177.2 (17)

Symmetry codes: (ii) $\frac{2}{3}+x-y, \frac{1}{3}+x, \frac{4}{3}-z$; (iii) $\frac{2}{3}-y, \frac{1}{3}+x-y, \frac{1}{3}+z$; (iv) $\frac{1}{3}-x+y, \frac{2}{3}-x, \frac{2}{3}+z$.

H atoms were placed in calculated positions, with C—H distances ranging from 0.95 to 1.00 Å and N—H distances of 0.91 Å, and then included in the refinement in a riding model, with $U_{iso} = 1.2U_{eq}$ of the carrier atom ($1.5U_{eq}$ for H atoms of the NH_3 group). The hydroxyl H atom was refined independently with an isotropic displacement parameter [O1—H1O 0.91 (2) Å].

Data collection: *COLLECT* (Nonius BV, 1997–2001); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1999); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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