

Refinement

Refinement on F^2 $R(F) = 0.038$ $wR(F^2) = 0.045$ $S = 1.05$

854 reflections

145 parameters

H-atom parameters not refined

 $w = 4F_o^2/\sigma^2(F_o^2)$ $(\Delta/\sigma)_{\max} = 0.03$ $\Delta\rho_{\max} = 0.30 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\min} = -0.18 \text{ e } \text{\AA}^{-3}$

Extinction correction: none

Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C) and Cromer & Waber (1974)Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

$$B_{\text{eq}} = (8\pi^2/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	B_{eq}
O1	0.0771 (5)	0.7336 (4)	0.6421 (4)	5.6 (1)
O2	-0.1659 (5)	0.6273 (4)	0.6164 (4)	8.1 (1)
O3	0.1017 (5)	0.3274 (4)	0.7436 (4)	6.5 (1)
N4	0.1837 (6)	0.5943 (4)	0.7247 (4)	4.8 (1)
C5	0.3430 (7)	0.5178 (5)	0.6458 (5)	4.8 (1)
C6	0.4302 (7)	0.3612 (5)	0.7135 (5)	4.4 (1)
C7	0.6375 (7)	0.2981 (6)	0.7311 (6)	5.3 (1)
C8	0.7174 (8)	0.1511 (7)	0.7901 (6)	6.6 (2)
C9	0.5916 (8)	0.0667 (6)	0.8344 (6)	6.2 (2)
C10	0.3881 (8)	0.1302 (6)	0.8177 (6)	6.1 (2)
C11	0.3051 (6)	0.2728 (5)	0.7589 (5)	4.2 (1)
C12	0.2545 (8)	0.6280 (6)	0.8500 (6)	5.8 (2)
C13	0.070 (1)	0.6945 (8)	0.9249 (7)	9.2 (2)
C14	0.3934 (9)	0.7248 (7)	0.8298 (7)	8.8 (2)
C15	-0.1060 (8)	0.7334 (6)	0.5986 (6)	6.2 (2)
C16	-0.216 (1)	0.8836 (8)	0.5188 (7)	9.0 (2)

Table 2. Selected geometric parameters (\AA , $^\circ$)

O1—N4	1.443 (3)	C6—C11	1.405 (7)
O1—C15	1.345 (7)	C7—C8	1.384 (7)
O2—C15	1.193 (8)	C10—C9	1.363 (7)
O3—C11	1.354 (5)	C10—C11	1.351 (6)
N4—C5	1.461 (6)	C8—C9	1.378 (9)
N4—C12	1.484 (8)	C15—C16	1.511 (8)
C5—C6	1.489 (6)	C12—C13	1.521 (8)
C6—C7	1.387 (6)	C12—C14	1.511 (9)
N4—O1—C15	111.7 (4)	C9—C10—C11	122.4 (5)
O1—N4—C5	107.1 (3)	O3—C11—C6	121.2 (4)
O1—N4—C12	107.3 (4)	O3—C11—C10	119.0 (5)
C5—N4—C12	115.2 (4)	C6—C11—C10	119.8 (4)
N4—C5—C6	109.7 (4)	N4—C12—C13	108.1 (5)
C5—C6—C7	120.8 (5)	N4—C12—C14	114.4 (5)
C5—C6—C11	121.0 (4)	C13—C12—C14	111.9 (5)
C7—C6—C11	118.2 (4)	O1—C15—O2	124.1 (4)
C6—C7—C8	120.4 (5)	O1—C15—C16	109.9 (5)
C7—C8—C9	120.3 (5)	O2—C15—C16	126.0 (5)
C8—C9—C10	118.8 (5)		

Table 3. Contact distances (\AA)

O2...N4	2.574 (4)	O2...O3	3.006 (4)
O3...N4	2.756 (5)		

A small colourless crystal was mounted on a glass fibre in a random orientation for data collection. A linear decay correction was applied but no absorption correction was made. The structure was solved using direct methods and refined by full-matrix least-squares methods. The weights were further modified using a sharpening factor of 20.0 (Dunitz & Seiler, 1973). Anomalous dispersion effects were included in F_c (Ibers & Hamilton, 1964); the values for $\Delta f'$ and $\Delta f''$ were those of Cromer (1974). The standard deviation of an observation of unit weight was 1.05. H atoms were placed in idealized positions (C—H = 0.96 \AA) with isotropic displacement parameters, $U = 0.05 \text{\AA}^2$.

All computations were carried out on a Compaq-PC using a combination of the PC-DOS versions of *SHELXS86* (Sheldrick, 1990) and *SDP* software (Frenz, 1988, 1991).

Data collection: *CAD-4 Software* (Enraf–Nonius, 1977). Cell refinement: *CAD-4 Software*. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SDP* (Frenz, 1988, 1991). Molecular graphics: *ORTEP* (Johnson, 1965).

We are thankful to KFUPM for supporting this work. Thanks are also due to Professor Sk. A. Ali for supplying the crystals and useful discussions.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BM1059). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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3-Hydroxyanthranilic Acid Hydrochloride

NOBUO OKABE,* AKIKO SHIMOSAKI AND JUNKO MIURA

Faculty of Pharmaceutical Sciences, Kinki University, Kowakae 3-4-1, Higashiosaka, Osaka 577, Japan

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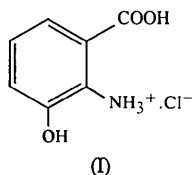
Abstract

In the structure of the title compound, 2-carboxy-6-hydroxyanilinium chloride, $\text{C}_7\text{H}_8\text{NO}_3^+\text{Cl}^-$, the amino group is protonated and the plane of the carboxyl group

is nearly coplanar with that of the phenol ring. The molecules are held together by direct hydrogen bonds between protonated amino and carboxyl groups, as well as by indirect ones *via* Cl atoms using all available hydrogen bonds.

Comment

3-Hydroxyanthranilic acid, a 2-amino-3-hydroxybenzoic acid, is an intermediate metabolite of tryptophan metabolism by the kynurenine pathway, which is derived from 3-hydroxykynurenine by kynureninase, a pyridoxal phosphate enzyme (Martin, Mayes & Rodwell, 1983). It is a carcinogen and induces DNA double-strand breaks in cultured human cells in the presence of Mn ions (Hiraku *et al.*, 1995), and also acts as an antioxidant for low-density lipoprotein (Bowry, Mohr, Cleary & Stocker, 1995). It is important to clarify the detailed structure of 3-hydroxyanthranilic acid in order to study its physiological properties. In this respect, we report here the crystal structure of its hydrochloride form, (I).



The molecular structure of the title compound is illustrated in Fig. 1. The plane of the carboxyl group is nearly coplanar with that of the phenol ring [O(1)—C(7)—C(1)—C(6) 9.1(8)°]. There is an intramolecular hydrogen bond between the protonated amino group and the adjacent carboxyl group [N(1)—H(22)···O(3) 2.673(6) Å]. The C(2)—C(3)—O(3) angle [117.0(5)°] is less than the C(4)—C(3)—O(3) angle [124.0(6)°]. This deviation may be caused by the formation of an intramolecular hydrogen bond. In the crystal packing shown in Fig. 2, the molecules are held together by four kinds of hydrogen bond, *i.e.* between carboxyl groups and anionic Cl atoms [O(1)—H(1)···Cl(1) 3.049(4) Å], between protonated amino groups and carboxyl groups [N(1)—H(23)···O(2)(-x, -y, 1-z) 2.872(6) Å], between protonated amino groups and Cl atoms [N(1)—

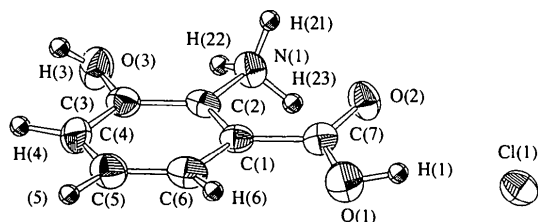


Fig. 1. An ORTEPII (Johnson, 1976) drawing of the title compound with the atomic numbering scheme. Ellipsoids correspond to 50% probability and H atoms are drawn as spheres of arbitrary radii.

H(21)···Cl(1)(1-x, -y, 1-z) 3.218(5) Å; N(1)—H(22)···Cl(1)(-x, -y, 1-z) 3.245(4) Å], and between hydroxyl groups and Cl atoms [O(3)—H(3)···Cl(1)(-x, 1/2+y, 3/2-z) 3.104(4) Å]. In these intra- and intermolecular hydrogen bonds, N(1)—H(22) participates as a bifurcated H-atom donor.

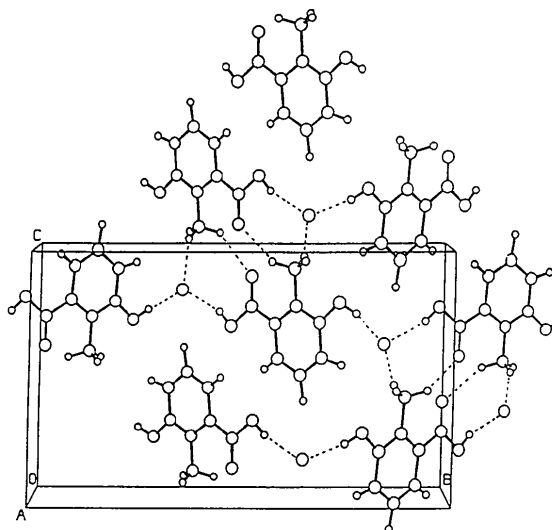


Fig. 2. The packing of the molecules viewed down the *a* axis. Hydrogen bonds are indicated by dashed lines.

Experimental

The light-brown needle crystal used for analysis were obtained by slow evaporation of a 0.5 N HCl/50% ethanol solution at room temperature.

Crystal data

C₇H₈NO₃⁺.Cl⁻
M_r = 189.60
 Monoclinic
*P*2₁/*c*
a = 4.470(2) Å
b = 17.568(2) Å
c = 10.183(2) Å
 β = 97.84(2)°
V = 792.1(4) Å³
Z = 4
D_x = 1.590 Mg m⁻³
D_m not measured

Mo *K*α radiation
 λ = 0.71069 Å
 Cell parameters from 25 reflections
 θ = 13.5–20.15°
 μ = 0.442 mm⁻¹
T = 296 K
 Needle
 0.3 × 0.1 × 0.1 mm
 Light brown

Data collection

Rigaku AFC-5R diffractometer
 ω -2 θ scans
 Absorption correction:
 ψ scans (North, Phillips & Mathews, 1968)
 T_{\min} = 0.94, T_{\max} = 1.00
 2118 measured reflections
 1889 independent reflections
 808 observed reflections
 $[I > 3\sigma(I)]$

R_{int} = 0.085
 θ_{max} = 27.5°
 h = 0 → 5
 k = 0 → 22
 l = -13 → 12
 3 standard reflections monitored every 150 reflections
 intensity decay: none

Refinement

Refinement on F
 $R = 0.054$
 $wR = 0.056$
 $S = 1.58$
 808 reflections
 109 parameters
 H atoms not refined
 $w = 4F_o^2/\sigma^2(F_o^2)$

$(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.36 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.32 \text{ e } \text{Å}^{-3}$
 Extinction correction: none
 Atomic scattering factors
 from *International Tables*
 for *X-ray Crystallography*
 (1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)

$$B_{\text{eq}} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i\cdot a_j$$

	x	y	z	B_{eq}
Cl(1)	0.7493 (3)	-0.1490 (1)	0.6718 (1)	3.18 (6)
O(1)	0.4008 (9)	-0.0280 (2)	0.8031 (4)	3.6 (2)
O(2)	0.2749 (8)	0.0190 (2)	0.5998 (4)	3.1 (2)
O(3)	-0.494 (1)	0.2209 (2)	0.6696 (4)	3.6 (2)
N(1)	-0.175 (1)	0.1182 (3)	0.5533 (4)	2.7 (2)
C(1)	0.067 (1)	0.0759 (3)	0.7776 (5)	2.2 (2)
C(2)	-0.129 (1)	0.1237 (3)	0.6994 (5)	2.3 (2)
C(3)	-0.302 (1)	0.1778 (3)	0.7552 (6)	2.6 (3)
C(4)	-0.274 (1)	0.1842 (4)	0.8927 (6)	3.1 (3)
C(5)	-0.080 (1)	0.1364 (4)	0.9714 (5)	3.2 (3)
C(6)	0.089 (1)	0.0834 (4)	0.9159 (6)	3.0 (3)
C(7)	0.252 (1)	0.0198 (4)	0.7179 (6)	2.5 (3)

Table 2. Selected geometric parameters (Å , $^\circ$)

O(1)—C(7)	1.320 (6)	C(1)—C(7)	1.469 (8)
O(2)—C(7)	1.221 (6)	C(2)—C(3)	1.394 (8)
O(3)—C(3)	1.366 (7)	C(3)—C(4)	1.394 (8)
N(1)—C(2)	1.477 (6)	C(4)—C(5)	1.382 (8)
C(1)—C(2)	1.386 (7)	C(5)—C(6)	1.370 (8)
C(1)—C(6)	1.405 (7)		
C(2)—C(1)—C(6)	118.1 (5)	C(2)—C(3)—C(4)	119.0 (6)
C(2)—C(1)—C(7)	121.1 (5)	C(3)—C(4)—C(5)	119.8 (6)
C(6)—C(1)—C(7)	120.8 (5)	C(4)—C(5)—C(6)	120.8 (5)
N(1)—C(2)—C(1)	122.0 (5)	C(1)—C(6)—C(5)	120.7 (5)
N(1)—C(2)—C(3)	116.4 (5)	O(1)—C(7)—O(2)	122.5 (6)
C(1)—C(2)—C(3)	121.5 (5)	O(1)—C(7)—C(1)	114.6 (5)
O(3)—C(3)—C(2)	117.0 (5)	O(2)—C(7)—C(1)	122.9 (6)
O(3)—C(3)—C(4)	124.0 (6)		

H atoms were located from difference Fourier maps, except for atoms H(22) and H(23), which were placed in calculated positions. All of these H atoms were included in subsequent least-squares refinement calculations with fixed isotropic displacement parameters.

Data collection: *MSCIAFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSCIAFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *SHELX86* (Sheldrick, 1985) and *DIRDIF* (Beurskens, 1984). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEPII* (Johnson, 1976).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: TA1084). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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9,10,12,14-Tetrahydro-12,12,14,14-tetra-phenyl-9,10[3',4']-furanoanthracene

TAI YU FU, GUNNAR OLOVSSON, JOHN R. SCHEFFER AND JAMES TROTTER

Department of Chemistry, University of British Columbia, Vancouver, BC, Canada V6T 1Z1. E-mail: jirt@xray4.chem.ubc.ca

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Abstract

The title molecule, $\text{C}_{42}\text{H}_{30}\text{O}$, is the product of solid-state photolysis of inclusion complexes of a dibenzobarrelene-bis(diphenylmethanol), and has normal geometry and dimensions.

Comment

The title compound, (I), was studied to assist in the interpretation of the photolysis of inclusion complexes of 9,10-dihydro-9,10-ethenoanthracene-11,12-bis(diphenyl-

