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4-Amino-2-chloro-6,7-dimethoxyquinazoline Methanol Solvate

CHUNQIU P. LAI,[a] LIU BO[b] AND SONGPING D. HUANG[a]*

[a]*Department of Chemistry, University of Puerto Rico, San Juan, PR 00931, USA, and* [b]*Department of Chemistry, East China Normal University, Shanghai 200062, China. E-mail: huang@zintl.chem.uprr.pr*

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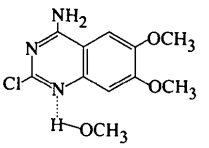
Abstract

Molecules of 4-amino-2-chloro-6,7-dimethoxyquinazoline, $C_{10}H_{10}ClN_3O_2.CH_4O$, form base-paired N—H···N hydrogen-bonded dimers in the solid state with N···N = 3.088 (2) Å. The quinazoline moieties are each flanked by a methanol molecule *via* N···H—O hydrogen bonding [N···O = 2.887 (2) Å].

Comment

4-Amino-2-chloro-6,7-dimethoxyquinazoline, (I), has been widely used in medicinal chemistry, particularly in the synthesis of cardiovascular agents such as telazosin (Winn, Kyncl, Dunnigan & Jones, 1977) and doxazosin (Campbell, Davey, Hardstone, Lewis & Palmer, 1987), which are members of a new class of antihypertensive agents.



(I).CH_4O

Single-crystal X-ray structure analysis shows that the unit cell contains molecules of (I) and the solvent methanol. Fig. 1 is an *ORTEP*II (Johnson, 1976) representation of (I).CH_4O. The molecules form base-paired N—H···N hydrogen-bonded dimers in the solid state. Furthermore, the quinazoline moieties are each flanked by a methanol molecule *via* N···H—O hydrogen bonding [N···O2.887(2) Å] as shown in Fig. 2.

The self-base-paired dimer is very similar to that of 6-amino-4-methoxy-2-methylthiopyrimidine, (II) (Low *et al.*, 1996). The bond length of the intermolecular

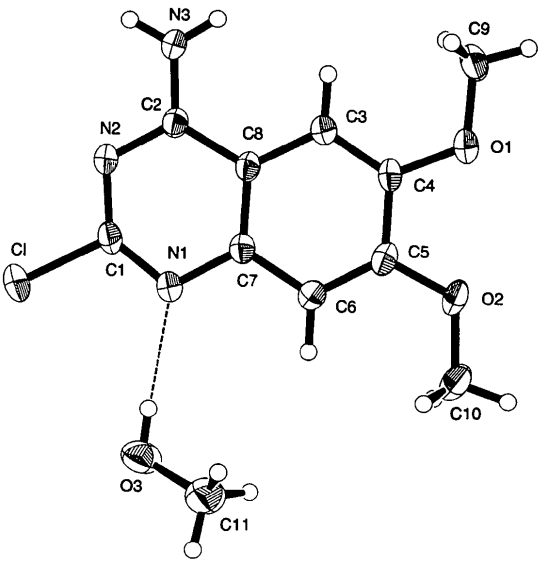


Fig. 1. *ORTEP*II (Johnson, 1976) representation of compound (I); displacement ellipsoids are drawn at the 50% probability level.

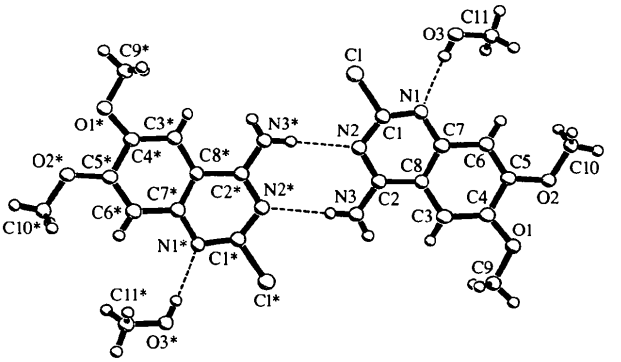


Fig. 2. A view of the hydrogen bonding in the unit cell with the atom-numbering scheme.

N···N hydrogen bond is 3.088 (2) Å in the present structure (symmetry operator: $1-x$, $1-y$, $1-z$) and 3.060 (3) Å in (II). In addition, there is only a single N—H···N hydrogen bond; the other amino H atom does not participate in hydrogen bonding because of steric hindrance in (I) and (II). Therefore, this structural feature indicates that self-base-pairing to a dimer occurs readily not only in the crystal structure of nucleobase compounds but also in that of nucleobase-like compounds, provided that an amine group of a ring C atom is adjacent to an unsubstituted ring N atom where neither group is sterically hindered.

The ten-membered bicyclic ring is essentially planar with a mean deviation of 0.028 (2) Å and a maximum deviation of 0.053 (2) Å. The methoxy groups are almost coplanar with the quinazoline ring [torsion angles C5—C4—O1—C9 179.6 (2) and C4—C5—O2—C10 −178.0 (2)°]. All the other bond distances and bond angles are in the normal range.

Experimental

4-Amino-2-chloro-6,7-dimethoxyquinazoline was synthesized from vanillin *via* multi-step reactions, including methylation, nitration, oxidation, hydrogenation, cyclization, chlorination and amination. The detailed synthetic procedure will be published elsewhere. Single crystals suitable for X-ray diffraction studies were obtained from methanol by slow evaporation of the solvent.

Crystal data

$C_{10}H_{10}ClN_3O_2.CH_4O$
$M_r = 271.70$
Monoclinic
$P2_1/n$
$a = 7.254\,(4)$ Å
$b = 11.914\,(2)$ Å
$c = 14.953\,(2)$ Å
$\beta = 100.34\,(3)°$
$V = 1271.3\,(6)$ Å^3
$Z = 4$
$D_x = 1.419$ Mg m^{-3}
D_m not measured
Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 12.0$–$14.0°$
$\mu = 0.305$ mm^{-1}
$T = 295.2$ K
Needle
$0.72 \times 0.26 \times 0.20$ mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2573 measured reflections
2402 independent reflections
1779 reflections with $I > 2.5\sigma(I)$
$R_{int} = 0.0117$
$\theta_{max} = 24.97°$
$h = 0 \rightarrow 8$
$k = 0 \rightarrow 14$
$l = -17 \rightarrow 17$
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

Refinement on F
$R = 0.042$
$wR = 0.055$
$S = 1.484$
1779 reflections
163 parameters
$w = 1/[\sigma^2(F_o)]$
$(\Delta/\sigma)_{max} = 0.0280$
$\Delta\rho_{max} = 0.50$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

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

Cl—C1	1.748 (2)	N1—C1	1.296 (3)
O1—C4	1.366 (2)	N1—C7	1.388 (2)
O1—C9	1.416 (3)	N2—C1	1.330 (3)
O2—C5	1.353 (2)	N2—C2	1.342 (2)
O2—C10	1.416 (3)	N3—C2	1.325 (2)
C4—O1—C9	116.3 (2)	N2—C2—N3	117.4 (2)
C5—O2—C10	118.2 (2)	N2—C2—C8	120.3 (2)
C1—N1—C7	113.9 (2)	N3—C2—C8	122.3 (2)
C1—N2—C2	115.4 (2)	O1—C4—C3	124.9 (2)
Cl—C1—N1	115.4 (2)	O1—C4—C5	114.9 (2)
Cl—C1—N2	113.0 (1)	O2—C5—C4	114.5 (2)
N1—C1—N2	131.6 (2)	O2—C5—C6	125.4 (2)

The hydroxyl H atom of the methanol was located from a difference map. All other H atoms were placed in calculated positions, with NH_2 assumed to be planar. H-atom parameters were not refined.

Data collection: *CAD-4-PC Software* (Enraf–Nonius, 1992). Cell refinement: *CAD-4-PC Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *SIR92* (Altomare, Cascarano, Giacovazzo & Guagliardi, 1993). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: JZ1177). Services for accessing these data are described at the back of the journal.

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(*E*,*Z*)-2-(2-Chloro-5-nitrostyryl)-1-(1-propenyl)benzimidazole

DANIEL E. BACELO, OSVALDO COX,* LUIS A. RIVERA, MARISOL CORDERO AND SONGPING D. HUANG*

Department of Chemistry, University of Puerto Rico, San Juan, PR 00931, USA. E-mail: huang@zintl.chem.uprr.pr

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Abstract

The title compound, $C_{18}H_{14}ClN_3O_2$, was synthesized by the condensation of 2-chloro-5-nitrobenzaldehyde with 2-methyl-1-propenylbenzimidazole, and the molecule comprises a 2-chloro-5-nitrobenzene and a 1-(*Z*)-propenylbenzimidazole. The two aromatic moieties are conjugated through the vinyl group. The dihedral angle between the two rings is 1.4 (6)°. The propenyl group lies out of the benzimidazole plane with a dihedral angle of 112.9 (9)°.