Crystal structure of acridinium chloride monohydrate, $[C_{13}H_{10}N]Cl \cdot H_2O$

Kwang Ha*

Chonnam National University, School of Applied Chemical Engineering, Research Institute of Catalysis, Gwangju 500-757, Republic of Korea

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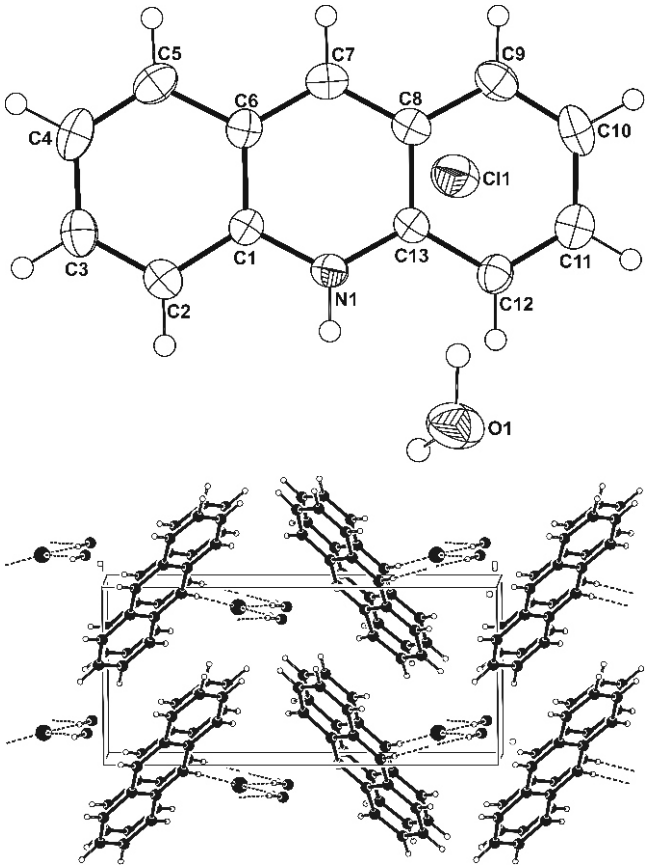

Abstract

$C_{13}H_{12}ClNO$, monoclinic, $P2_1$ (no. 4), a = 5.442(2) Å, b = 14.935(5) Å, c = 6.931(2) Å, β = 99.016(8)°, V = 556.4 Å^3, Z = 2, $R_{gt}(F)$ = 0.057, $wR_{ref}(F^2)$ = 0.131, T = 200 K.

Source of material

To a solution of acridine (0.201 g, 1.12 mmol) in acetone (20 ml) were added five drops of hydrochloric acid (37%) and stirred for 5 min at room temperature. The formed precipitate was separated by filtration, washed with ether and dried under vacuum, to give a yellow powder (0.232 g). Crystals suitable for X-ray diffraction analysis were obtained by slow evaporation from a CH_3CN solution.

Experimental details

Hydrogen atoms were positioned geometrically and allowed to ride on their parent atoms with d(C—H) = 0.95 Å and U_{iso}(H) = 1.2 U_{eq}(C). The H atoms at the N and O atoms were located from Fourier difference maps and refined isotropically: d(N—H) = 0.87(5) Å and d(O—H) = 0.91(5) Å and 0.94(7) Å. The highest peak (0.42 e·Å^{-3}) and the deepest hole (–0.47 e·Å^{-3}) in the difference Fourier map are located 1.03 Å and 0.80 Å from the Cl1 atom, respectively. The Flack parameter is 0.33(11) in the refinement. Because the compound is a weak anomalous scatterer, the parameter is meaningless and the absolute crystal structure cannot be determined reliably.

Discussion

The title compound consists of a protonated acridinium cation, a Cl^- anion and a solvent water molecule (figure, top). In the crystal structure, the component ions and the water molecule interact by means of intermolecular N–H···Cl and O–H···Cl hydrogen bonds with d(N···Cl) = 3.095(4) Å and d(O···Cl) = 3.234(4) Å and 3.233(4) Å, forming chains along [100] (figure, bottom). The nearly planar cations are arranged in a V-shaped packing pattern along [001] and stacked in columns along [100]. In the columns, numerous intermolecular π-π interactions between adjacent six-membered rings are present. The centroid-centroid distance between Cg1 (the centroid of ring C1-C6) and $Cg2^i$ (ring C8-C13, symmetry code i: x,y,z–1) is 3.937(3) Å, and the dihedral angle between the ring planes is 1.1(2)°.

Table 1. Data collection and handling.

Crystal:	yellow block, size 0.06 × 0.25 × 0.28 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	3.19 cm^{-1}
Diffractometer, scan mode:	Bruker SMART 1000 CCD, φ/ω
$2\theta_{max}$:	56.58°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4131, 2719
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1755
$N(param)_{refined}$:	157
Programs:	SHELXS-97, SHELXL-97 [1], ORTEP-3 [2], PLATON [3]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1)	2*a*	1.07(1)	0.261(3)	0.945(7)	0.07(2)
H(2)	2*a*	1.1718	0.3426	0.6715	0.038
H(3)	2*a*	1.0623	0.4593	0.4549	0.045
H(4)	2*a*	0.7275	0.5542	0.4878	0.048
H(5)	2*a*	0.5094	0.5367	0.7439	0.044
H(7)	2*a*	0.4421	0.4491	1.0412	0.034
H(9)	2*a*	0.3717	0.3575	1.3311	0.043
H(10)	2*a*	0.4891	0.2363	1.5336	0.046
H(11)	2*a*	0.8240	0.1462	1.4842	0.045
H(12)	2*a*	1.0397	0.1721	1.2303	0.037
H(1A)	2*a*	0.697(9)	0.077(3)	0.850(6)	0.05(1)
H(1B)	2*a*	0.99(1)	0.074(4)	0.842(7)	0.08(2)

* e-mail: hakwang@chonnam.ac.kr

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cl(1)	2*a*	0.3383(2)	0.15989(8)	0.8646(1)	0.0360(5)	0.0374(5)	0.0391(5)	0.0025(6)	0.0098(4)	–0.0031(6)
N(1)	2*a*	0.9580(7)	0.2989(2)	0.9686(5)	0.027(2)	0.030(2)	0.029(2)	0.003(2)	0.005(2)	–0.002(2)
C(1)	2*a*	0.8988(7)	0.3672(3)	0.8407(5)	0.023(2)	0.023(2)	0.025(2)	–0.004(2)	–0.000(2)	–0.001(2)
C(2)	2*a*	1.0365(8)	0.3808(3)	0.6865(6)	0.030(2)	0.036(2)	0.029(2)	–0.003(2)	0.005(2)	–0.003(2)
C(3)	2*a*	0.9711(9)	0.4499(3)	0.5593(6)	0.040(3)	0.040(3)	0.033(3)	–0.009(2)	0.006(2)	0.004(2)
C(4)	2*a*	0.7713(9)	0.5075(3)	0.5798(6)	0.049(3)	0.031(3)	0.035(3)	–0.009(2)	–0.005(2)	0.008(2)
C(5)	2*a*	0.6413(9)	0.4967(3)	0.7300(7)	0.040(3)	0.026(2)	0.042(3)	0.003(2)	0.000(2)	–0.001(2)
C(6)	2*a*	0.7013(8)	0.4257(3)	0.8674(6)	0.032(2)	0.024(2)	0.028(2)	–0.005(2)	0.003(2)	–0.002(2)
C(7)	2*a*	0.5744(7)	0.4102(3)	1.0224(6)	0.025(2)	0.025(2)	0.033(2)	–0.004(2)	–0.001(2)	–0.010(2)
C(8)	2*a*	0.6351(8)	0.3396(3)	1.1513(5)	0.027(2)	0.031(2)	0.025(2)	–0.003(2)	0.002(2)	–0.007(2)
C(9)	2*a*	0.5067(9)	0.3207(3)	1.3085(7)	0.035(3)	0.038(3)	0.035(3)	–0.004(2)	0.010(2)	–0.009(2)
C(10)	2*a*	0.5769(9)	0.2492(3)	1.4287(6)	0.047(3)	0.041(3)	0.030(2)	–0.018(2)	0.014(2)	–0.005(2)
C(11)	2*a*	0.7769(8)	0.1948(3)	1.3980(6)	0.041(3)	0.035(3)	0.034(3)	–0.004(2)	0.002(2)	0.001(2)
C(12)	2*a*	0.9049(8)	0.2098(3)	1.2488(6)	0.030(2)	0.029(2)	0.032(2)	–0.003(2)	0.003(2)	0.003(2)
C(13)	2*a*	0.8358(7)	0.2822(3)	1.1212(5)	0.025(2)	0.027(2)	0.023(2)	–0.002(2)	0.002(2)	–0.004(2)
O(1)	2*a*	0.8319(8)	0.0439(2)	0.8322(5)	0.037(2)	0.044(2)	0.072(3)	–0.003(2)	0.019(2)	–0.007(2)

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References

1. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112-122.
2. Farrugia, L. J.: ORTEP-3 for Windows - a version of ORTEP-III with a Graphical User Interface (GUI) J. Appl. Crystallogr. **30** (1997) 565.
3. Spek, A. L.: Single-crystal structure validation with the program PLATON. J. Appl. Crystallogr. **36** (2003) 7-13.