Crystal structure of 4-bromomethyl-acridinium bromide—chloroform (1:1), $C_{15}H_{12}Br_2Cl_3N$

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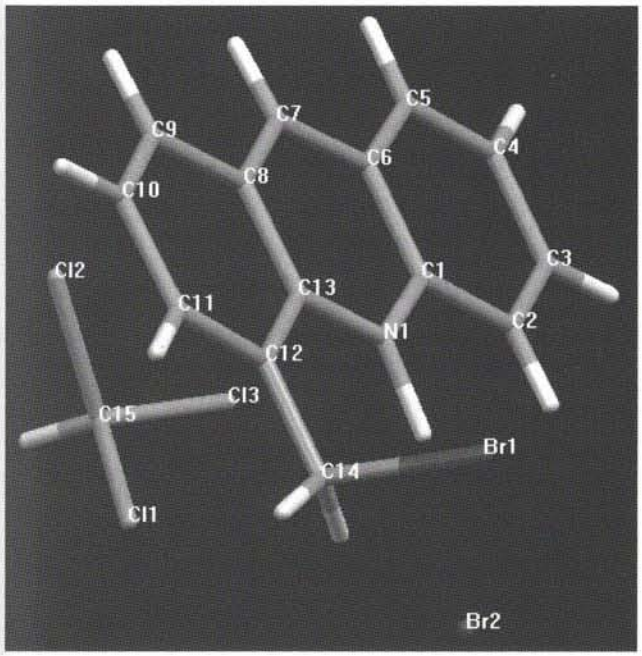

Abstract

$C_{15}H_{12}Br_2Cl_3N$, monoclinic, $P12_1/c1$ (No. 14), $a = 10.7811(4)$ Å, $b = 9.1048(4)$ Å, $c = 17.7633(8)$ Å, $\beta = 102.875(3)°$, $V = 1699.8$ Å^3, $Z = 4$, $R_{gt}(F) = 0.044$, $wR_{ref}(F^2) = 0.132$, $T = 293$ K.

Source of material

The compound was crystallised from chloroform.

Discussion

Bromomethyl-acridines are key compounds in the synthesis of new potent antitumors agents studied in our laboratory [1,2]. 4-bromomethyl-acridine was obtained according to a described synthetic route [1]. It is well known that benzylic and bromobenzylic positions are sensitive toward oxygen oxidation, leading to the corresponding cetone/aldehyde/acid [3,4].

According to these studies, we suppose that a photochemical oxydation of the benzylic position of one molecule of 4-bromomethyl-acridine occured, freeding one molecule of bromhydrid, which led to the formation of 4-bromomethyl-acridinium bromide.

In the crystal structure, three chlorines of the included chloroform molecule are orientated toward the acridine protonated nitrogen atom. These chlorine atoms stabilize, with their external electrons, the positive charge induced by the protonation. The distances between the nitrogen and the three chlorines are 3.850 Å to 4.424 Å and the distance between Br2 and the nitrogen is 3.352 Å. The C14—Br1 bond has a standard length of 1.97 Å.

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.15 × 0.2 × 0.2 mm
Wavelength:	Mo K_α radiation (0.71070 Å)
μ:	52.32 cm^{-1}
Diffractometer, scan mode:	Kappa CCD, 90 frames, $\Delta\varphi = 2°$
$2\theta_{max}$:	52.74°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3463, 3463
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2668
$N(param)_{refined}$:	194
Programs:	SHELXS-97 [5], SHELXL-97 [6], Mercury [7]

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

Atom	Site	x	y	z	U_{iso}
H(1)	4e	0.1608	0.3936	0.1992	0.10(3)
H(2)	4e	0.3174	0.5697	0.2214	0.05
H(3)	4e	0.4676	0.712	0.1806	0.05
H(4)	4e	0.4902	0.7068	0.0529	0.05
H(5)	4e	0.3511	0.5713	–0.0404	0.05
H(7)	4e	0.1735	0.3899	–0.0704	0.05
H(9)	4e	–0.0110	0.2214	–0.1025	0.05
H(10)	4e	–0.1646	0.0884	–0.0601	0.05
H(11)	4e	–0.1789	0.0960	0.0702	0.05
H(14A)	4e	0.0396	0.2337	0.2306	0.06(2)
H(14B)	4e	–0.1182	0.1606	0.1966	0.07(2)

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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}
Br(1)	4*e*	–0.12990(6)	0.40959(8)	0.21387(4)	0.0606(4)	0.0839(5)	0.0573(4)	–0.0030(3)	0.0214(3)	–0.0158(3)
Br(2)	4*e*	0.24750(5)	0.35270(6)	0.32523(3)	0.0493(3)	0.0624(4)	0.0362(3)	–0.0024(2)	0.0066(2)	0.0026(2)
Cl(1)	4*e*	0.3569(2)	0.0513(3)	0.1825(1)	0.074(1)	0.120(2)	0.0552(9)	–0.028(1)	0.0230(8)	–0.0001(9)
Cl(2)	4*e*	0.3717(1)	0.0970(2)	0.02438(8)	0.0511(7)	0.0680(9)	0.0506(8)	–0.0044(7)	0.0070(6)	0.0026(7)
Cl(3)	4*e*	0.5442(2)	0.2475(2)	0.1468(1)	0.097(1)	0.077(1)	0.068(1)	–0.042(1)	–0.0061(9)	0.0131(9)
N(1)	4*e*	0.1498(4)	0.3978(4)	0.1339(2)	0.039(2)	0.039(2)	0.035(2)	–0.004(2)	0.008(2)	0.001(2)
C(1)	4*e*	0.2392(4)	0.4833(5)	0.1129(3)	0.038(2)	0.037(2)	0.038(2)	0.000(2)	0.011(2)	0.003(2)
C(2)	4*e*	0.3220(5)	0.5682(6)	0.1670(3)	0.051(3)	0.057(3)	0.044(3)	–0.013(3)	0.004(2)	0.002(2)
C(3)	4*e*	0.4117(5)	0.6507(6)	0.1441(4)	0.052(3)	0.053(3)	0.062(4)	–0.016(3)	0.008(3)	0.003(3)
C(4)	4*e*	0.4237(6)	0.6516(7)	0.0673(4)	0.051(3)	0.067(4)	0.066(4)	–0.012(3)	0.019(3)	0.017(3)
C(5)	4*e*	0.3440(6)	0.5707(7)	0.0136(4)	0.056(3)	0.063(4)	0.058(4)	–0.003(3)	0.024(3)	0.012(3)
C(6)	4*e*	0.2481(5)	0.4833(5)	0.0343(3)	0.046(3)	0.041(3)	0.043(3)	0.006(2)	0.016(2)	0.007(2)
C(7)	4*e*	0.1644(5)	0.3956(6)	–0.0176(3)	0.052(3)	0.050(3)	0.035(3)	0.009(2)	0.015(2)	0.002(2)
C(8)	4*e*	0.0713(4)	0.3134(5)	0.0052(3)	0.042(2)	0.042(3)	0.032(2)	0.007(2)	0.008(2)	–0.001(2)
C(9)	4*e*	–0.0153(5)	0.2259(6)	–0.0482(3)	0.054(3)	0.054(3)	0.041(3)	0.005(3)	0.007(2)	–0.006(2)
C(10)	4*e*	–0.1049(6)	0.1476(7)	–0.0246(3)	0.057(3)	0.057(3)	0.054(3)	–0.009(3)	0.004(3)	–0.020(3)
C(11)	4*e*	–0.1126(5)	0.1507(6)	0.0542(3)	0.046(3)	0.052(3)	0.058(3)	–0.011(3)	0.011(2)	–0.008(3)
C(12)	4*e*	–0.0310(5)	0.2311(5)	0.1085(3)	0.039(2)	0.040(3)	0.042(3)	–0.002(2)	0.008(2)	0.001(2)
C(13)	4*e*	0.0649(4)	0.3155(5)	0.0838(3)	0.038(2)	0.031(2)	0.038(2)	0.005(2)	0.008(2)	0.001(2)
C(14)	4*e*	–0.0436(5)	0.2300(6)	0.1900(3)	0.054(3)	0.048(3)	0.047(3)	–0.011(2)	0.014(2)	0.004(2)
C(15)	4*e*	0.4580(5)	0.0851(6)	0.1208(3)	0.041(3)	0.053(3)	0.051(3)	–0.001(2)	0.008(2)	0.009(2)

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