Crystal structure of anhydrous tetraethylammonium chloride, $[(\text{CH}_3\text{CH}_2)_4\text{N}]\text{Cl}$

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Abstract

$\text{C}_8\text{H}_{20}\text{ClN}$, monoclinic, $P12_1/n1$ (No. 14), $a = 11.8207(2)$ Å, $b = 20.4092(4)$ Å, $c = 12.4290(1)$ Å, $\beta = 108.173(3)^\circ$, $V = 2849.0$ Å³, $Z = 12$, $R_{\text{gt}}(F) = 0.053$, $R_w(F^2) = 0.116$, $T = 213$ K.

Source of material

The compound was isolated from acetonitrile as a side product. The crystals were grown in anhydrous acetonitrile in a dry atmosphere.

Discussion

The structure is similar to those reported previously, although there are three molecules per asymmetric cell and there are no waters of hydration. The structure reported by Loechlin and Kvik [1] is the monohydrate and Mak, Bruins, Slot and Beurskens [2] reported the tetrahydrate structure.

Table 1. Data collection and handling.

Crystal:	colorless plate, size 0.05 x 0.10 x 0.15 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	3.38 cm ⁻¹
Diffractometer, scan mode:	SMART CCD, ω , $\delta\omega = 0.3^\circ$
$2\theta_{\text{max}}$:	56.76°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	18492, 6893
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 5145
$N(\text{param})_{\text{refined}}$:	266
Programs:	SHELXS-97 [3], SHELXL-97 [4]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U_{iso}
N(3)	4e	0.4582(1)	-0.26034(7)	0.2334(1)	0.0225(3)
H(1A)	4e	0.3901	0.1511	0.1940	0.034
H(1B)	4e	0.4251	0.1485	0.3276	0.034
H(2A)	4e	0.4965	0.2445	0.2720	0.061
H(2B)	4e	0.5703	0.2036	0.2086	0.061
H(2C)	4e	0.6054	0.2009	0.3423	0.061
H(3A)	4e	0.6519	0.0271	0.3684	0.037
H(3B)	4e	0.6838	0.1020	0.3635	0.037
H(4A)	4e	0.6708	0.0766	0.5415	0.059
H(4B)	4e	0.5378	0.0525	0.4868	0.059
H(4C)	4e	0.5697	0.1276	0.4819	0.059
H(5A)	4e	0.5978	0.0302	0.1673	0.037
H(5B)	4e	0.6209	0.1065	0.1688	0.037
H(6A)	4e	0.5030	0.0742	-0.0109	0.056
H(6B)	4e	0.4290	0.1223	0.0415	0.056
H(6C)	4e	0.4058	0.0458	0.0401	0.056
H(7A)	4e	0.3919	0.0371	0.3263	0.035
H(7B)	4e	0.3515	0.0377	0.1925	0.035
H(8A)	4e	0.3919	-0.0689	0.2583	0.059
H(8B)	4e	0.5237	-0.0489	0.3286	0.059
H(8C)	4e	0.4833	-0.0483	0.1945	0.059
H(9A)	4e	-0.0090	-0.1565	0.3357	0.041
H(9B)	4e	0.1081	-0.1215	0.3294	0.041
H(10A)	4e	0.0916	-0.1102	0.5095	0.065
H(10B)	4e	-0.0334	-0.0752	0.4591	0.065
H(10C)	4e	0.0840	-0.0402	0.4529	0.065
H(11A)	4e	-0.1685	-0.0404	0.3030	0.041
H(11B)	4e	-0.2131	-0.0388	0.1691	0.041
H(12A)	4e	-0.3195	-0.1166	0.2340	0.074
H(12B)	4e	-0.2001	-0.1537	0.2991	0.074
H(12C)	4e	-0.2448	-0.1520	0.1650	0.074
H(13A)	4e	-0.0423	0.0160	0.1651	0.036
H(13B)	4e	0.0023	0.0169	0.2990	0.036
H(14A)	4e	0.1599	0.0394	0.2307	0.065
H(14B)	4e	0.1403	-0.0282	0.1649	0.065
H(14C)	4e	0.1850	-0.0273	0.2990	0.065
H(15A)	4e	0.0422	-0.1201	0.1341	0.039
H(15B)	4e	-0.0743	-0.1577	0.1351	0.039
H(16A)	4e	-0.1004	-0.1130	-0.0420	0.059

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Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(16B)	4e	−0.0738	−0.0420	0.0114	0.059
H(16C)	4e	−0.1907	−0.0797	0.0124	0.059
H(17A)	4e	0.4581	−0.3521	0.1675	0.033
H(17B)	4e	0.5045	−0.3514	0.3013	0.033
H(18A)	4e	0.6606	−0.3759	0.2314	0.060
H(18B)	4e	0.6871	−0.3084	0.2973	0.060
H(18C)	4e	0.6406	−0.3091	0.1632	0.060
H(19A)	4e	0.2886	−0.2962	0.1688	0.033
H(19B)	4e	0.3322	−0.2928	0.3026	0.033
H(20A)	4e	0.1832	−0.2157	0.2295	0.057
H(20B)	4e	0.2597	−0.1825	0.1600	0.057

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(20C)	4e	0.3034	−0.1790	0.2940	0.057
H(21A)	4e	0.4318	−0.1779	0.1341	0.036
H(21B)	4e	0.5458	−0.2178	0.1324	0.036
H(22A)	4e	0.4010	−0.2242	−0.0432	0.058
H(22B)	4e	0.3099	−0.2551	0.0129	0.058
H(22C)	4e	0.4242	−0.2951	0.0112	0.058
H(23A)	4e	0.6118	−0.2166	0.3318	0.035
H(23B)	4e	0.4963	−0.1776	0.3315	0.035
H(24A)	4e	0.5861	−0.2227	0.5093	0.056
H(24B)	4e	0.5746	−0.2938	0.4560	0.056
H(24C)	4e	0.4588	−0.2547	0.4558	0.056

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cl(1)	4e	0.22881(5)	−0.08198(3)	−0.00897(4)	0.0451(3)	0.0399(3)	0.0360(3)	0.0085(2)	0.0100(2)	0.0026(2)
Cl(2)	4e	0.79225(5)	0.25494(3)	0.51659(4)	0.0406(3)	0.0457(3)	0.0322(3)	−0.0036(2)	0.0085(2)	−0.0009(2)
Cl(3)	4e	0.26751(5)	0.08822(3)	0.48435(5)	0.0478(3)	0.0429(3)	0.0398(3)	0.0120(3)	0.0087(2)	0.0010(2)
N(1)	4e	0.5144(1)	0.08033(7)	0.2638(1)	0.0211(7)	0.0229(8)	0.0228(7)	−0.0004(6)	0.0061(6)	0.0013(6)
N(2)	4e	−0.0439(1)	−0.07506(7)	0.2339(1)	0.0255(8)	0.0230(8)	0.0250(8)	0.0029(6)	0.0074(6)	0.0008(6)
C(1)	4e	0.4572(2)	0.14670(9)	0.2639(2)	0.030(1)	0.026(1)	0.029(1)	0.0042(8)	0.0071(8)	0.0008(8)
C(2)	4e	0.5397(2)	0.2040(1)	0.2724(2)	0.049(1)	0.027(1)	0.042(1)	−0.0049(9)	0.008(1)	0.0009(9)
C(3)	4e	0.6213(2)	0.0717(1)	0.3684(2)	0.0234(9)	0.037(1)	0.028(1)	0.0015(8)	0.0026(7)	0.0027(8)
C(4)	4e	0.5978(2)	0.0831(1)	0.4796(2)	0.042(1)	0.048(1)	0.024(1)	0.002(1)	0.0059(9)	0.0028(9)
C(5)	4e	0.5596(2)	0.0732(1)	0.1630(2)	0.031(1)	0.037(1)	0.028(1)	0.0023(8)	0.0144(8)	−0.0002(8)
C(6)	4e	0.4659(2)	0.0795(1)	0.0481(2)	0.041(1)	0.046(1)	0.025(1)	0.003(1)	0.0114(9)	−0.0004(9)
C(7)	4e	0.4194(2)	0.03013(9)	0.2605(2)	0.027(1)	0.028(1)	0.031(1)	−0.0047(8)	0.0081(8)	−0.0001(8)
C(8)	4e	0.4580(2)	−0.0403(1)	0.2605(2)	0.051(1)	0.027(1)	0.039(1)	−0.0033(9)	0.012(1)	−0.0001(9)
C(9)	4e	0.0289(2)	−0.1137(1)	0.3366(2)	0.041(1)	0.029(1)	0.029(1)	0.0082(9)	0.0070(9)	0.0055(8)
C(10)	4e	0.0441(2)	−0.0820(1)	0.4496(2)	0.055(1)	0.044(1)	0.027(1)	0.003(1)	0.009(1)	0.001(1)
C(11)	4e	−0.1705(2)	−0.0654(1)	0.2350(2)	0.027(1)	0.040(1)	0.036(1)	−0.0001(9)	0.0118(8)	−0.0048(9)
C(12)	4e	−0.2399(2)	−0.1274(1)	0.2331(2)	0.043(1)	0.058(2)	0.048(1)	−0.022(1)	0.017(1)	−0.006(1)
C(13)	4e	0.0073(2)	−0.00718(9)	0.2324(2)	0.031(1)	0.025(1)	0.032(1)	−0.0025(8)	0.0083(8)	0.0015(8)
C(14)	4e	0.1344(2)	−0.0057(1)	0.2317(2)	0.031(1)	0.060(2)	0.038(1)	−0.010(1)	0.0091(9)	0.005(1)
C(15)	4e	−0.0411(2)	−0.1142(1)	0.1307(2)	0.039(1)	0.030(1)	0.029(1)	0.0079(8)	0.0102(9)	−0.0033(8)
C(16)	4e	−0.1074(2)	−0.0846(1)	0.0182(2)	0.050(1)	0.039(1)	0.027(1)	0.004(1)	0.0088(9)	−0.0002(9)
C(17)	4e	0.5086(2)	−0.32834(9)	0.2335(2)	0.031(1)	0.0236(9)	0.0277(9)	0.0049(8)	0.0087(8)	0.0004(8)
C(18)	4e	0.6356(2)	−0.3307(1)	0.2311(2)	0.032(1)	0.052(1)	0.035(1)	0.010(1)	0.0094(9)	−0.002(1)
C(19)	4e	0.3312(2)	−0.26900(9)	0.2338(2)	0.0232(9)	0.031(1)	0.028(1)	−0.0013(7)	0.0081(7)	0.0014(8)
C(20)	4e	0.2633(2)	−0.2059(1)	0.2289(2)	0.034(1)	0.042(1)	0.041(1)	0.0107(9)	0.0139(9)	0.004(1)
C(21)	4e	0.4625(2)	−0.2221(1)	0.1298(2)	0.033(1)	0.030(1)	0.029(1)	−0.0035(8)	0.0105(8)	0.0063(8)
C(22)	4e	0.3932(2)	−0.2518(1)	0.0177(2)	0.046(1)	0.046(1)	0.025(1)	0.001(1)	0.0097(9)	0.0057(9)
C(23)	4e	0.5308(2)	−0.2215(1)	0.3358(2)	0.030(1)	0.028(1)	0.030(1)	−0.0055(8)	0.0066(8)	−0.0039(8)
C(24)	4e	0.5382(2)	−0.2508(1)	0.4493(2)	0.042(1)	0.043(1)	0.024(1)	−0.002(1)	0.0058(9)	−0.0033(9)

References

- Loehlin, J. H.; Kvick, A.: Tetraethylammonium Chloride Monohydrate. *Acta Crystallogr. B* **34** (1978) 3488–3490.
- Mak, T. C. W.; Slot, H. J. B.; Beurskens, P. T.: Tetraethylammonium Chloride Tetrahydrate. *J. Inclusion Phenom.* **4** (1986) 295–298.
- Sheldrick, G. M.: Phase Annealing in SHELX-90: Direct Methods for Large Structures. *Acta Crystallogr. A* **46** (1990) 467–473.
- Sheldrick, G. M.; SHELXL-97. Program for refining crystals structures. University of Göttingen, Germany 1997.