Z. Kristallogr. NCS 220 (2005) 135-136

Crystal structure of 4-chlorobenzylammonium dihydrogenmonophosphate, $(ClC_7H_6NH_3)H_2PO_4$

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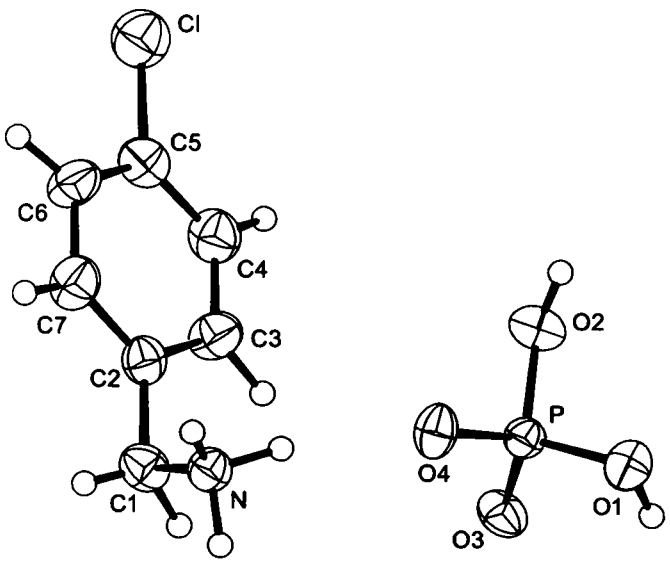
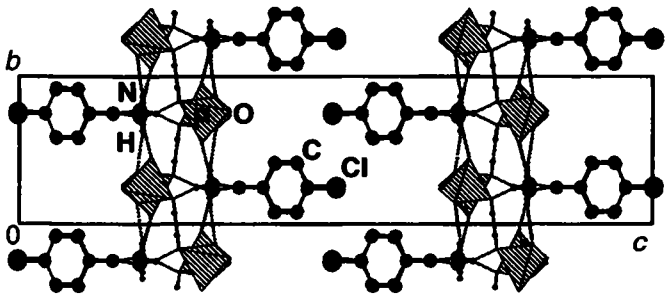
Received December 6, 2004, accepted and available on-line February 27, 2005; CCDC no. 1267/1440





Abstract

$C_7H_{11}ClNO_4P$, orthorhombic, *Pbcn* (no. 60), $a = 7.246(2)$ Å, $b = 9.134(2)$ Å, $c = 30.518(3)$ Å, $V = 2019.9$ Å^3, $Z = 8$, $R_{gt}(F) = 0.059$, $wR_{ref}(F) = 0.078$, $T = 296$ K.

Source of material

The title compound was prepared from an aqueous solution containing a stoichiometric mixture of 4-chlorobenzylamine and monophosphoric acid (molar ratio 1/1). The solution obtained after reaction was slowly evaporated at room temperature for several days until formation of transparent prismatic single crystals of good quality and suitable dimensions for crystallographic study. Chemical synthesis is reproducible and crystals obtained in this way are stable for a long time under normal temperature and humidity.

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Discussion

The title compound is composed of the two basic components, the $H_2PO_4^-$ anion and the $(p\text{-}ClC_6H_4CH_2NH_3)^+$ cation (figure, top). The crystal structure consists of inorganic layers of $H_2PO_4^-$ parallel to (001) planes at $z = ¼$ and $z = ¾$ (figure, bottom). Between these layers, organic groups $(p\text{-}ClC_6H_4CH_2NH_3)^+$ are located. The aromatic ring of the protonated organic amine is planar, with a mean plane deviation of 0.003 Å, the bond lengths of C2—C3, C3—C4, C4—C5, C5—C6, C6—C7 and C7—C2 are 1.390(7) Å, 1.392(7) Å, 1.367(7) Å, 1.368(7) Å, 1.379(7) Å, 1.328(7) Å, respectively, which are between single bond and double bond and agree with that in benzene [1]. Multiple hydrogen bonds connect the different entities of the crystal structure. Regarding the organic cations arrangement, these groups are in opposition, by creating thus a local inversion center. It is worth noting that the O···O distances involved in the hydrogen bonds (2.491(7) Å – 2.589(4) Å) are of the same order of magnitude as the O···O distances in the H_2PO_4 tetrahedron (2.442(4) Å – 2.536(4) Å). These distances and the short P···P separation of 4.567(1) Å allow to consider the $H_2PO_4{}_n^{n-}$ sub-network as a polyanion. The association of the $ClC_7H_6NH_3^+$ cations and $H_2PO_4^-$ anions is different to that of the ions in $[ClC_7H_6NH_3Cl][LiBeF_4]$ [2] or $[C_7H_7NH_3][H_2PO_4]$ [3].

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.15 × 0.30 × 1.10 mm
Wavelength:	Ag K_α radiation (0.5608 Å)
μ:	2.73 cm^{-1}
Diffractometer, scan mode:	Enraf-Nonius Mach3, ω-2θ
$2\theta_{max}$:	49.92°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4118, 4118
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 3\,\sigma(I_{obs})$, 1660
$N(param)_{refined}$:	171
Programs:	SIR92 [4], teXsan [5], DIFABS [6]

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

Atom	Site	x	y	z	U_{iso}
H(1)	8*d*	0.073(5)	−0.097(4)	0.748(1)	0.10(2)
H(2)	8*d*	0.263(4)	−0.291(4)	0.668(1)	0.07(1)
H(3)	8*d*	0.748(3)	0.203(3)	0.7180(8)	0.032(7)
H(4)	8*d*	0.677(4)	0.063(3)	0.6966(7)	0.029(7)
H(5)	8*d*	0.869(5)	0.086(3)	0.6972(9)	0.049(9)
H(6)	8*d*	0.623(5)	0.269(4)	0.6553(9)	0.06(1)
H(7)	8*d*	0.838(5)	0.267(4)	0.650(1)	0.07(1)
H(8)	8*d*	0.465(4)	0.096(3)	0.6092(9)	0.038(8)
H(9)	8*d*	0.459(4)	−0.073(3)	0.5472(9)	0.040(8)
H(10)	8*d*	1.035(5)	−0.056(4)	0.547(1)	0.07(1)
H(11)	8*d*	1.017(4)	0.093(3)	0.6007(8)	0.038(8)

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	8*d*	0.7407(1)	–0.19752(9)	0.49723(2)	0.0619(5)	0.0570(4)	0.0507(4)	–0.0010(5)	–0.0039(5)	–0.0172(3)
P	8*d*	0.2472(1)	–0.08553(7)	0.70243(2)	0.0270(2)	0.0274(2)	0.0317(3)	0.0015(3)	–0.0066(3)	0.0000(2)
O(1)	8*d*	0.1708(3)	–0.1505(2)	0.74542(6)	0.0339(9)	0.0415(9)	0.0370(9)	0.0053(9)	0.0007(9)	0.0074(9)
O(2)	8*d*	0.2289(3)	–0.2008(2)	0.66556(6)	0.048(1)	0.0316(9)	0.0363(9)	0.0041(9)	–0.0106(9)	–0.0022(7)
O(3)	8*d*	0.4538(2)	–0.0573(2)	0.71036(6)	0.0251(8)	0.052(1)	0.0358(9)	–0.0053(8)	–0.0030(8)	0.0046(9)
O(4)	8*d*	0.1425(3)	0.0461(2)	0.68840(6)	0.036(1)	0.0261(8)	0.050(1)	0.0037(8)	–0.0059(9)	0.0050(8)
N	8*d*	0.7726(3)	0.1337(3)	0.69312(7)	0.032(1)	0.036(1)	0.031(1)	0.000(1)	–0.0008(9)	–0.0029(8)
C(1)	8*d*	0.7455(5)	0.2126(3)	0.65059(9)	0.045(1)	0.034(1)	0.038(1)	–0.001(2)	–0.004(2)	0.002(1)
C(2)	8*d*	0.7453(4)	0.1083(3)	0.61265(8)	0.038(1)	0.035(1)	0.032(1)	–0.002(1)	–0.001(2)	0.0052(9)
C(3)	8*d*	0.5793(4)	0.0571(3)	0.5958(1)	0.039(2)	0.049(2)	0.044(2)	0.004(1)	0.002(1)	–0.007(1)
C(4)	8*d*	0.5781(4)	–0.0381(3)	0.5601(1)	0.037(2)	0.048(2)	0.054(2)	–0.003(1)	–0.005(2)	–0.002(2)
C(5)	8*d*	0.7426(4)	–0.0824(3)	0.54264(8)	0.048(1)	0.039(1)	0.033(1)	–0.003(2)	–0.006(2)	0.001(1)
C(6)	8*d*	0.9082(4)	–0.0349(4)	0.55883(9)	0.044(2)	0.055(2)	0.036(2)	0.002(2)	0.005(1)	–0.011(1)
C(7)	8*d*	0.9080(4)	0.0603(4)	0.5939(1)	0.038(2)	0.055(2)	0.040(2)	–0.004(2)	–0.001(1)	–0.003(2)

Acknowledgment. The authors gratefully acknowledge the support by the Secretary of State for Scientific Research and Technology.

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