Crystal structure of caesium tetraphenylborate, (C₂₄H₂₀B)Cs

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Abstract

C₂₄H₂₀BCs, tetragonal, $\bar{4}2m$ (No. 121), $a = 11.1647(5)$ Å, $c = 8.3352(4)$ Å, $V = 1039.0$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.013$, $wR_{\text{ref}}(F^2) = 0.033$, $T = 100$ K.

Source of material

The title compound was prepared by mixing aqueous solutions of CsCl (0.24 M) and NaBPh₄ (0.34 M). The resulting precipitate was washed with water and air dried. Crystals were grown from slow evaporation of a acetone/1,2-dichloroethane (2:1) solution.

Discussion

The separation of radioactive caesium from nuclear wastes, through precipitation of its tetraphenylborate salt, is being considered for large scale application [1]. Despite the present importance of this solid, its crystal structure has not been determined, even though the lattice parameters of this, and several other tetraphenylborate salts, were determined more than 40 years ago [2]. As part of our continuing investigations into separations of large cations and cation- π interactions, we decided to determine this structure.

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

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
Cs	2 <i>a</i>	0	0	0	0.0183(1)	<i>U</i> ₁₁	0.0087(1)	0	0	0
C(1)	8 <i>i</i>	0.0821(1)	− <i>x</i>	−0.3787(2)	0.0122(4)	<i>U</i> ₁₁	0.0105(7)	0.0012(5)	0.0003(4)	− <i>U</i> ₁₃
C(2)	16 <i>j</i>	0.0409(1)	−0.1918(1)	−0.3155(2)	0.0172(5)	0.0137(5)	0.0139(5)	0.0000(4)	−0.0003(4)	−0.0007(4)
C(3)	16 <i>j</i>	0.1028(1)	−0.2546(1)	−0.1972(2)	0.0237(6)	0.0147(6)	0.0172(6)	0.0020(4)	−0.0005(5)	0.0038(5)
C(4)	8 <i>i</i>	0.2103(1)	− <i>x</i>	−0.1367(3)	0.0217(5)	<i>U</i> ₁₁	0.0180(9)	0.0066(7)	−0.0037(5)	− <i>U</i> ₁₃
B	2 <i>b</i>	0	0	1/2	0.0113(7)	<i>U</i> ₁₁	0.009(1)	0	0	0

The structure of the title compound bears close resemblance to the previously reported tetraphenylborate salts of rubidium [3] and potassium [4]. All three crystallize in $\bar{4}2m$ and exhibit the same overall structure. The metal cation rests in one of the clefts between two phenyl rings. Symmetry generates another tetraphenylborate anion which occupies the remainder of the cation's coordination sphere, thus forming a one-dimensional linear chain along the *c*-axis. The cation is therefore coordinated by tetrahedral arrangement of phenyl rings, with the cation-centroid distance decreasing significantly only in the potassium structure (Cs 3.16 Å, Rb 3.17 Å, K 2.99 Å). The caesium-centroid distance reported here is among the shortest published [5], and is considerably shorter than the 3.41 Å recently calculated for caesium-benzene [6].

Table 1. Data collection and handling.

Crystal:	colourless, platelet, size 0.07 × 0.23 × 0.63 mm
Wavelength:	Mo <i>K</i> _α radiation (0.71073 Å)
μ:	17.85 cm ^{−1}
Diffractometer, scan mode:	Nonius CAD4, ω
2θ _{max} :	58°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	5114, 745
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 745
<i>N</i> (<i>param</i>) _{refined} :	35
Programs:	SHELXS-97 [7], SHELXL-97 [8], SHELXTL [9], PLATON [10]

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

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(2)	16 <i>j</i>	−0.0319	−0.2242	−0.3553	0.018
H(3)	16 <i>j</i>	0.0715	−0.328	−0.1576	0.022
H(4)	8 <i>i</i>	0.2526	− <i>x</i>	−0.0557	0.025

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