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The crystal structure of dimethylammonium *catena*-[di(μ -aqua)-bis(μ_9 -benzene-1,3,5-tricarboxylato)pentalithium], $C_{20}H_{16}Li_5NO_{13}$

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.21 × 0.17 × 0.14 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.14 mm ⁻¹
Diffractometer, scan mode:	Bruker D8 quest, φ and ω
$\theta_{\max}$, completeness:	26.4°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	9719, 2086, 0.085
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1430
$N(\text{param})_{\text{refined}}$:	190
Programs:	Bruker [1], SHELX [2], Olex2 [3]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.3316(2)	0.61540(14)	0.98985(15)	0.0111(5)
C2	0.3527(2)	0.58610(14)	0.89394(15)	0.0119(5)
H2	0.277525	0.575826	0.852047	0.014*
C3	0.4824(2)	0.57152(14)	0.85810(15)	0.0113(5)
C4	0.5925(2)	0.58884(14)	0.91913(15)	0.0118(5)
H4	0.681195	0.577546	0.895762	0.014*
C5	0.5737(2)	0.62253(14)	1.01400(15)	0.0112(5)
C6	0.4426(2)	0.63586(14)	1.04877(16)	0.0123(5)
H6	0.429038	0.659136	1.113326	0.015*
C7	0.1928(2)	0.61438(15)	1.03547(15)	0.0121(5)
C8	0.5016(2)	0.52939(14)	0.75820(15)	0.0121(5)
C9	0.6919(2)	0.63341(14)	1.08296(15)	0.0126(5)
C10	1.0615(4)	0.750000	0.8549(3)	0.0276(9)
H10A	1.134857	0.750000	0.903212	0.041*
H10B	1.068137	0.804460	0.813482	0.041*
C11	0.8155(4)	0.750000	0.8361(3)	0.0290(9)
H11A	0.730414	0.750000	0.872753	0.044*
H11B	0.820204	0.695540	0.794513	0.044*
Li1	0.2258(4)	0.5644(3)	1.2248(3)	0.0177(8)
Li2	0.0494(4)	0.4660(3)	0.9122(3)	0.0190(9)
Li3	0.1297(6)	0.750000	1.2191(4)	0.0200(12)
N1	0.9301(3)	0.750000	0.9062(2)	0.0294(8)
H1	0.925(3)	0.7991(14)	0.9455(16)	0.035*
O1	0.18033(14)	0.64744(10)	1.12066(10)	0.0152(4)
O2	0.09736(14)	0.57719(10)	0.98906(10)	0.0160(4)
O3	0.40498(14)	0.53272(10)	0.69771(10)	0.0153(4)

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Abstract

$C_{20}H_{16}Li_5NO_{13}$, orthorhombic, $Pnma$ (no. 62), $a = 9.9218(8)$ Å, $b = 14.6941(11)$ Å, $c = 13.5697(10)$ Å, $V = 1978.4(3)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0439$, $wR_{\text{ref}}(F^2) = 0.0960$, $T = 173(2)$ K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

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Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
O4	0.61176(15)	0.49114(10)	0.73767(10)	0.0167(4)
O5	0.80962(14)	0.62671(11)	1.04832(10)	0.0175(4)
O6	0.66678(14)	0.64572(10)	1.17317(10)	0.0156(4)
O7	−0.0669(2)	0.750000	1.20898(17)	0.0235(6)
H7	−0.101(2)	0.7056(14)	1.1784(16)	0.028*

Source of material

A mixture of 1,3,5-benzenetricarboxylic acid (H₃BTC; 0.21 g, 1 mmol), LiOH·H₂O (0.125 g, 3 mmol) and *N,N'*-dimethylacetamide (10 mL) was sealed in a 25 mL Teflon-lined stainless steel vessel and heated at 120 °C for 4320 mins. Yield: 50% based on Li.

Experimental details

The hydrogen atoms from O and N atoms were positioned based on difference electron peaks and refined freely with O7–H7 = 0.844 Å (*U*_{iso}(H) = 1.2*U*_{eq}(O)) and N1–H1 = 0.899 Å (*U*_{iso}(H) = 1.2*U*_{eq}(N)).

Comment

Metal-organic frameworks (MOFs) are porous materials composed of metal ions/clusters and organic units, which have the characteristics of porosity, high specific surface area and are easy to modify. MOFs and CPs are widely used in gas storage and separation [4], fluorescent responses [5], proton conductivity [6], membrane separation [7, 8], environmental pollution control [9, 10] and energy conversion [11, 12]. However, most of the MOFs are constructed from the transition metals or rare earth, and the alkali-metal-based MOFs are rare examples, particularly the lithium-based MOFs [13, 14]. Compared to other metals, the lithium-based MOFs can enhance gravimetric energy storage capacity more effectively, so we are interested in embarking on the synthesis of the lightest lithium-based MOFs.

Single crystal X-ray analysis reveals that the title compound crystallizes orthorhombically in the space group *Pnma* and contains three crystallographically independent Li ions (two in general positions one on a mirror plane). The Li1 center adopts four-coordinated geometry with four O atoms from different BTC ligands (Li1–O: 1.887–1.920 Å). The Li2 center is five-coordinated with the square pyramidal geometry and the Li–O bond lengths are in the range of Li2–O: 1.996–2.289 Å. Although the Li3 center also adopts five-coordinated geometry, one coordinated O atom is from one water (Li3–O: 1.954–2.149 Å). Note, the oxygen atoms offered by BTC anions connect different Li ions to form a complex 3D framework, which

is completely different from other reported skeleton structures based on lithium and BTC [15–17].

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