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Sodium perchlorate in the space group *Pnma*

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The first example of $Na^+ClO_4^-$ in the orthorhombic system in space group *Pnma* at room temperature is reported. The Na, Cl and two O atoms are located on positions of site symmetry *m*.

Key indicators

Single-crystal X-ray study
T = 571 K
Mean σ(l–O) = 0.006 Å
R factor = 0.075
wR factor = 0.200
Data-to-parameter ratio = 11.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

It has been reported that $LiClO_4$ (Henderson & Brooks, 2003), $KClO_4$, $RbClO_4$ and $CsClO_4$ (Bats & Fuess, 1982; Granzin, 1988) crystallize in the same orthorhombic space group *Pnma* at room temperature, while interestingly $NaClO_4$ has *Cmcm* (Liu, *et al.*, 2002; Wartchow & Berthold, 1978) or *Bbmm* orthorhombic symmetry (Zachariasen, 1930) and $F\bar{4}3m$ cubic symmetry (Herrmann & Ilge, 1930). It has a cubic NaCl structure with space group *Fm3m* at high temperature (Berthold *et al.*, 1979 and Berthold, *et al.*, 1983). To the best of our knowledge, no structure of sodium perchlorate in space group *Pnma* has been reported. We obtained it, by chance, from a solution of cucurbit[5]uril and present the structure here.

The asymmetric unit of the title compound consists of one Na, one Cl and three O atoms. Na1, Cl1, O2 and O3 are located on positions of site symmetry *m*. Figs. 1 and 2 show the

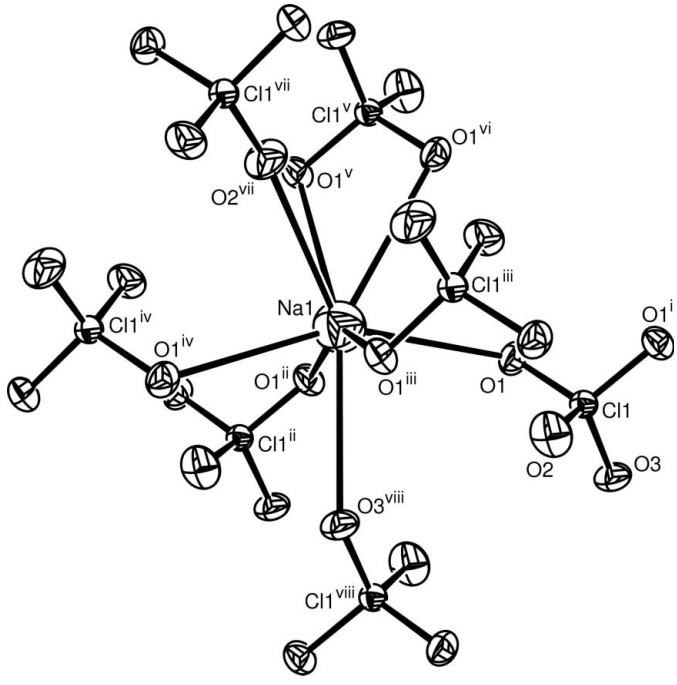

Figure 1
Na^+ cation coordination. Displacement ellipsoids are drawn at the 30% probability level. [Symmetry codes: (i) $x, \frac{1}{2}-y, z$; (ii) $x, \frac{3}{2}-y, z$; (iii) $x-\frac{1}{2}, y, \frac{1}{2}-z$; (iv) $x-\frac{1}{2}, \frac{3}{2}-y, \frac{1}{2}-z$; (v) $1-x, y+\frac{1}{2}, 1-z$; (vi) $1-x, 1-y, 1-z$; (vii) $\frac{1}{2}-x, 1-y, \frac{1}{2}+z$; (viii) $1-x, 1-y, -z$.]

coordination of the Na^+ cations and ClO_4^- anions, respectively. Each Na^+ cation is coordinated by seven different ClO_4^- anions with Na—O distances of 2.977 (7)–3.093 (7) Å (Table 1).

Experimental

Although the initial intention of our work was to prepare a polynuclear complex of cucurbit[5]uril with rare earth ions, sodium ions were added to enhance the solubility of cucurbit[5]uril. During the synthetic process crystals of sodium perchlorate were obtained as follows. To a 0.2 *M* sodium chloride aqueous solution (10 ml), cucurbit[5]uril (0.166 g) and a methanol (5 ml) solution of $Y(ClO_4)_3{\cdot}6H_2O$ (0.198 g) were added in turn with continuous stirring for 4 h at 373 K. The solution was filtered to remove a small amount of an insoluble material, and left to evaporate slowly at room temperature Colorless single crystals were obtained after a month.

Crystal data

$Na^+ClO_4^-$	$Z = 4$
$M_r = 122.44$	$D_x = 2.036$ Mg m^{-3}
Orthorhombic, *Pnma*	Mo $K\alpha$ radiation
$a = 9.236$ (7) Å	$\mu = 0.93$ mm^{-1}
$b = 5.809$ (4) Å	$T = 571$ (2) K
$c = 7.444$ (6) Å	Block, colorless
$V = 399.4$ (5) Å^3	0.28 × 0.23 × 0.19 mm

Data collection

Bruker APEX area-detector diffractometer	1882 measured reflections
	387 independent reflections
φ and ω scans	342 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)	$R_{int} = 0.032$
	$\theta_{max} = 25.0°$
$T_{min} = 0.782$, $T_{max} = 0.844$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.1006P)^2 + 2.6942P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.075$	
$wR(F^2) = 0.200$	
$S = 1.08$	$(\Delta/\sigma)_{max} < 0.001$
387 reflections	$\Delta\rho_{max} = 0.53$ e Å^{-3}
34 parameters	$\Delta\rho_{min} = -0.92$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl1—O1	1.438 (4)	Na1—O1^{v}	3.093 (7)
Cl1—O2	1.423 (6)	Na1—O2^{vii}	3.070 (9)
Na1—O1	2.977 (7)	Na1—O3^{viii}	2.962 (8)
Na1—O1^{iii}	2.989 (7)		
O3^{viii}—Na1—O1	86.6 (2)	O1—Na1—O1^{iii}	103.55 (15)
O3^{viii}—Na1—O1^{iii}	69.57 (18)	O1—Na1—O1^{iv}	156.0 (2)
O1^{ii}—Na1—O1	71.5 (2)	O1^{iii}—Na1—O1^{iv}	71.1 (2)

Symmetry codes: (i) $x, -y+\frac{1}{2}, z$; (ii) $x, -y+\frac{3}{2}, z$; (iii) $x-\frac{1}{2}, y, -z+\frac{1}{2}$; (iv) $x-\frac{1}{2}, -y+\frac{3}{2}, -z+\frac{1}{2}$; (v) $-x+1, y+\frac{1}{2}, -z+1$; (vi) $-x+1, -y+1, -z+1$; (vii) $-x+\frac{1}{2}, -y+1, z+\frac{1}{2}$; (viii) $-x+1, -y+1, -z$.

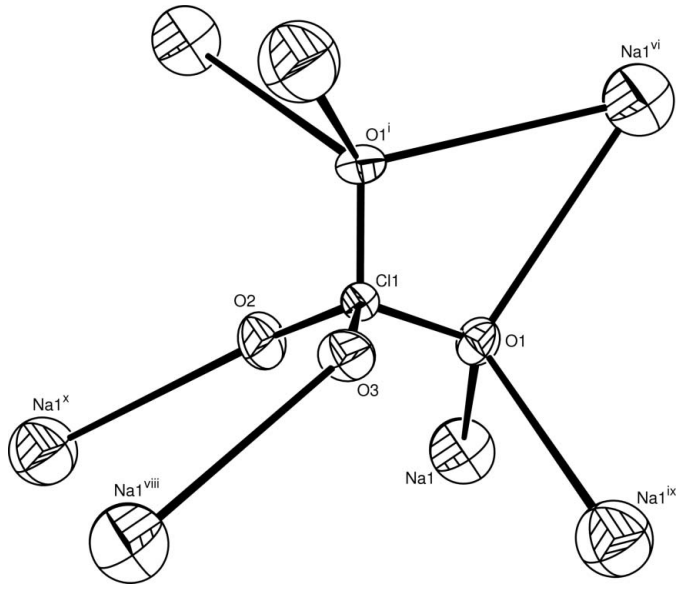

Figure 2
ClO_4^- anion coordination. Displacement ellipsoids are drawn at the 30% probability level. [Symmetry codes: (viii) $1-x, 1-y, -z$; (ix) $\frac{1}{2}+x, y, \frac{1}{2}-z$; (*x*) $\frac{1}{2}-x, 1-y, z-\frac{1}{2}$.]

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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