Novel peroxosolvates of tetraalkylammonium halides: first case of hydrogen bonded peroxide-containing layers.

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Experimental data sets were collected on a Bruker SMART APEX II diffractometer (for **1**, **3** and **4**) and Bruker D8 Venture machine for **2** using graphite monochromatized Mo-*K*α radiation (λ = 0.71073 Å). Absorption corrections based on measurements of equivalent reflections were applied.[1] The structures were solved by direct methods and refined by full matrix least-squares on F^2 with anisotropic thermal parameters for all non-hydrogen atoms.[2] All hydrogen atoms were found from difference Fourier synthesis and refined with isotropic thermal parameters. In all structures, partial substitutional disorder of hydrogen peroxide by water molecules[3-6] was not observed since no residual peaks with an intensity more than 0.17 $e \cdot A^{-3}$ were seen in the hydrogen peroxide molecule regions.

Table S1. Crystal data and details of X-ray analysis.

	1	**2**	**3**	**4**
Empirical formula	$C_8H_{24}Cl_1N_1O_4$	$C_8H_{24}Br_1N_1O_4$	$C_5H_{15}Cl_2N_1O_2$	$C_9H_{16}Cl_1N_1O_2$
*F*w	233.73	278.19	192.08	205.68
colour, habit	colourless, prism	colourless, prism	colourless, unshapen	colourless, prism
cryst size (mm)	0.30 × 0.20× 0.10	0.30 × 0.15× 0.05	0.40 × 0.30× 0.25	0.45 × 0.20× 0.10
tempreture (K)	150	150	150	120
crystal system	orthorhombic	orthorhombic	orthorhombic	monoclinic
space group	$Pna2_1$	$Pna2_1$	$P2_12_12_1$	$P2_1/c$
a (Å)	11.8741(5)	12.0887(6)	7.5628(2)	7.2420(3)
b (Å)	8.4164(3)	8.5504(5)	9.9033(3)	10.0690(4)
c (Å)	12.6736(6)	12.7308(7)	25.3929(7)	14.7992(6)
β (deg)	90	90	90	100.379(2)
V (Å^3)	1266.56(9)	1315.90(12)	1901.84(9)	1061.50(7)
Z	4	4	8	4
D_c (g·cm^{-3})	1.226	1.404	1.342	1.287
μ (mm^{-1})	0.295	3.118	0.634	0.330
F(000)	512	584	816	440
θ range (deg)	2.91 to 28.98	2.87 to 29.00	2.21 to 29.99	2.46 to 26.99
refl collcd	14169	22397	35364	10629
indep reflns / R_{int}	3342 / 0.0328	3496 / 0.0398	5462 / 0.0188	2287 / 0.0200
reflns $I>2\sigma(I)$	3271	3324	5422	2080
No of param	223	224	302	182
GooF on F^2	1.041	1.023	1.063	1.045
R_1 ($I>2\sigma(I)$)	0.0359	0.0198	0.0187	0.0270
wR_2(all data)	0.1013	0.0429	0.0493	0.0735
Flack parameter	0.09(5)	0.184(9)	0.01(3)	—
largest diff peak / hole (e·Å^{-3})	0.263 / -0.156	0.277 / -0.283	0.304 / -0.232	0.288 / -0.188

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5. G. Laus, V. Kahlenberg, K. Wurst, T. Lörting and H. Schottenberger, *CrystEngComm*, 2008, **10**, 1638.
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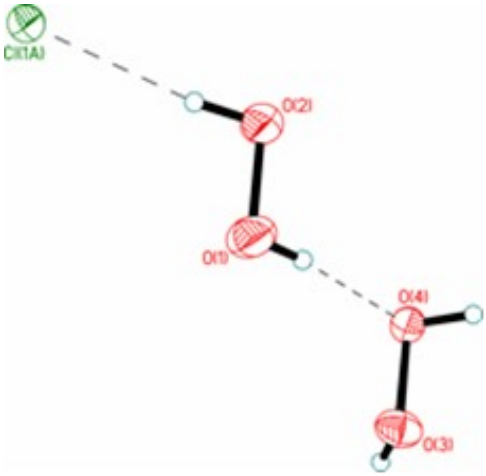

Fig. S1 H-bonds formed by H1–O1–O2–H2 molecule in the structure **1**. Symmetry operation: (A) x, -1+y, z.

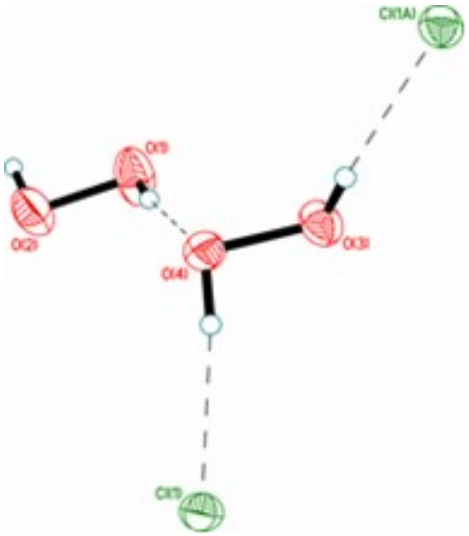

Fig. S2 H-bonds formed by H3–O3–O4–H4 molecule in the structure **1**. Symmetry operation: (A) 0.5-x, 1.5+y, 0.5+z.