

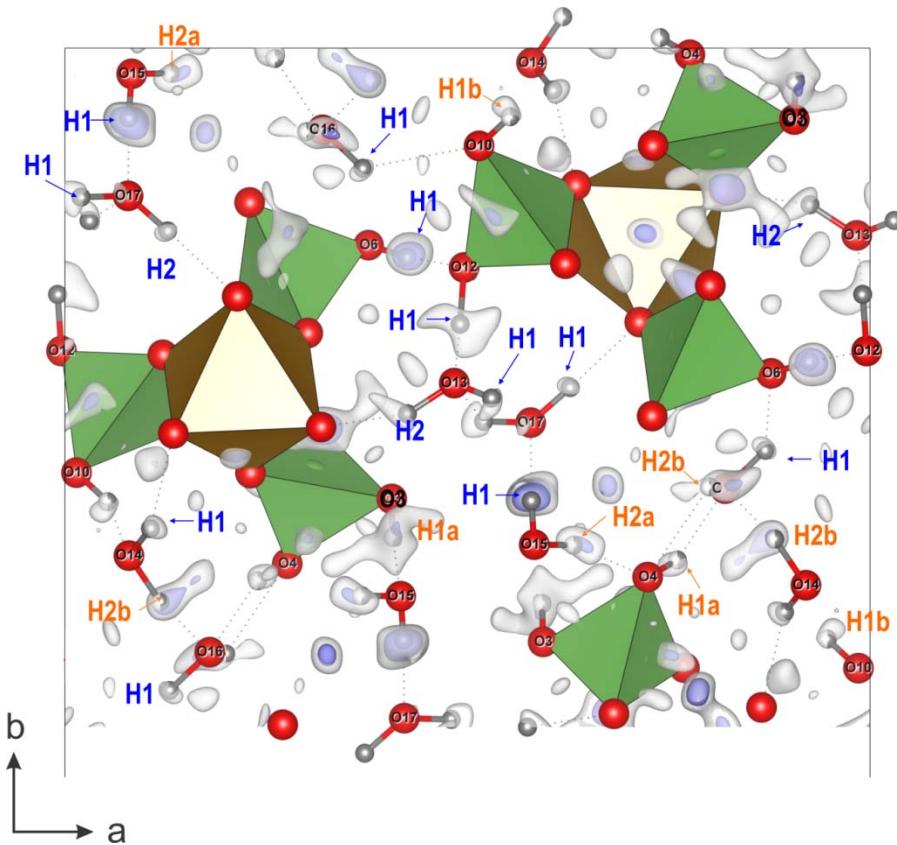
# IUCrJ

**Volume 8 (2021)**

**Supporting information for article:**

**Hydrogen disorder in kaatialaite  $\text{Fe}[\text{AsO}_2(\text{OH})_2]5\text{H}_2\text{O}$  from Jáchymov, Czech Republic: determination from low-temperature 3D electron diffraction**

**Gwladys Steciuk, Juraj Majzlan and Jakub Plášil**



**Figure S1** Crystal structure viewed along axis  $c$  with the superimposed difference-Fourier map generated from the second data set only and represented as isosurfaces  $2\sigma[\Delta V(r)]$  (white) and  $3\sigma[\Delta V(r)]$  (blue) with visible hydrogen positions after the dynamical refinement of kaatialaite structure without hydrogen.

**Table S1** Summary of the hydrogen positions revealed from the dynamical and the kinematical refinements. The significance of the maxima in the difference potential map  $\Delta V(r)$  is expressed with the isosurface level  $\sigma$ .

Refinement:	Dynamical		Kinematical	
Configuration:	$a$	$b$	$a$	$b$
<b><i>OH: H1O<i>i</i></i></b>				
H1O3	$3.02\sigma$	$2.81\sigma^*$	$\geq 2\sigma$	$\geq 2\sigma$
H1O4	$2.50\sigma$	$2.15\sigma^*$	$\geq 2\sigma$	$\geq 3\sigma$
H1O6	-----	$3.55\sigma$ -----	-----	$\geq 3\sigma$ -----
H1O8	-----	$2.85\sigma$ -----	-----	not visible -----
H1O10	$3\sigma^*$	$2\sigma$	not visible	$\geq 2\sigma$
H1O12	-----	$3.02\sigma$ -----	-----	$\geq 3\sigma$ -----
<b><i>H<sub>2</sub>O: H1O<i>i</i>/H2O<i>i</i></i></b>				
H1O13/H2O13	-----	$3.38\sigma / 2.31\sigma$ -----	-----	$\geq 3\sigma / \geq 3\sigma$ -----
H1O14/H2O14	$3.55\sigma / 2\sigma^*$	$3.55\sigma / 2.50\sigma$	$\geq 2\sigma / \text{not visible}$	$\geq 2\sigma / \geq 2\sigma$
H1O15/H2O15	$3.55\sigma / 2.50\sigma$	$3.55\sigma / 2.80\sigma^*$	$\geq 2\sigma / \text{not visible}$	$\geq 2\sigma / \text{not visible}$
H1O16/H2O16	$3.38\sigma / \text{not visible}$	$3.38\sigma / 3.20\sigma$	$\geq 3\sigma / \text{not visible}$	$\geq 3\sigma / \geq 2\sigma$
H1O17/H2O17	-----	$3.20\sigma / 3.20\sigma$ -----	$3\sigma / \text{not visible}$	

\* the heights of the maxima corresponding to the hydrogen atoms obtained after a second step of dynamical refinement involving the first visible hydrogen.

**Table S2** Atom positions, displacement parameters ( $\text{\AA}^2$ ) and bond-valence sums (BVS, in valence-units) for kaatialaite as obtained from dynamical refinement.

atom	occu	x/a	y/b	z/c	Uiso	BVS
As1	1	0.19667(7)	0.08330(8)	0.1282(2)	0.0121(3)	4.84(3)
As2	1	0.22229(7)	0.28286(8)	0.1154(2)	0.0103(3)	5.12(3)
As3	1	0.42806(7)	0.16204(8)	0.1411(2)	0.0127(3)	4.83(3)
Fe1	1	0.28056(7)	0.17728(9)	0.6325(2)	0.0072(3)	2.752(11)
O1	1	0.18162(18)	0.1392(2)	0.3758(6)	0.0218(9)	1.922(16)
O2	1	0.2682(2)	0.0941(2)	-0.1187(6)	0.0203(9)	1.841(13)
O3	1	0.0937(2)	0.0687(2)	-0.0095(7)	0.0304(11)	2.10(6)
O4	1	0.22467(19)	0.0046(2)	0.2706(6)	0.0201(10)	1.98(5)
O5	1	0.1974(2)	0.2272(3)	-0.1229(6)	0.0258(10)	1.97(2)
O6	1	0.1247(2)	0.3134(2)	0.2395(6)	0.0314(10)	2.04(3)
O7	1	0.28702(19)	0.2637(2)	0.3902(6)	0.0208(9)	1.79(1)
O8	1	0.2721(2)	0.3499(3)	-0.0098(7)	0.0399(12)	2.22(4)
O9	1	0.3661(2)	0.1287(2)	0.3878(6)	0.0219(9)	1.88(1)
O10	1	0.4848(2)	0.0954(3)	0.0130(8)	0.0438(13)	2.15(7)
O11	1	0.37989(18)	0.2074(2)	-0.1103(6)	0.0178(9)	1.84(1)
O12	1	0.5051(2)	0.2118(2)	0.3087(6)	0.0297(10)	2.11(3)
O13	1	0.51566(19)	0.3215(2)	0.0500(5)	0.0391(12)	2.04(3)
O14	1	0.9241(2)	0.4860(2)	0.1268(7)	0.0326(11)	1.86(5)
O15	1	0.9193(3)	0.0246(2)	0.3880(6)	0.0429(12)	1.88(8)
O16	1	0.6806(2)	0.0784(3)	0.0903(7)	0.0420(12)	1.98(4)
O17	1	0.9232(2)	0.1404(2)	0.0636(5)	0.0402(12)	1.97(4)
H1o3a	0.5	0.093(2)	0.0317(12)	-0.144(6)	0.036456	1.07(7)
H1o3b	0.5	0.0434(13)	0.0937(19)	0.055(5)	0.036456	0.97(5)
H1o4a	0.5	0.261(2)	0.9698(11)	0.189(3)	0.024118	0.98(5)
H1o4b	0.5	0.8258(14)	0.0121(13)	0.631(5)	0.024118	0.99(5)
H1o6	1	0.0808(8)	0.3096(11)	0.089(2)	0.037707	1.01(3)
H1o8	1	0.2378(11)	0.3758(11)	-0.147(3)	0.047882	1.01(4)
H1o10a	0.5	0.534(2)	0.124(2)	0.965(4)	0.052573	0.97(6)
H1o10b	0.5	0.4470(14)	0.066(2)	0.901(6)	0.052573	1.04(6)
H1o12	1	0.5091(11)	0.2542(5)	0.207(4)	0.035683	1.10(3)
H1o13	1	0.4754(6)	0.3315(12)	-0.107(2)	0.046971	0.99(2)
H2o13	1	0.5745(4)	0.3345(12)	0.001(3)	0.046971	0.99(2)
H1o14	1	0.8938(11)	0.4520(4)	0.013(3)	0.039105	1.03(3)
H2o14a	0.5	0.9426(16)	0.4664(6)	0.3061(19)	0.039105	1.06(5)
H2o14b	0.5	0.6183(11)	0.0169(12)	0.297(6)	0.039105	0.99(5)
H1o15	1	0.9207(11)	0.0667(5)	0.283(3)	0.051441	0.99(2)
H2o15a	0.5	0.8685(14)	0.0242(9)	0.506(6)	0.051441	0.97(8)
H2o15b	0.5	0.086(3)	0.0130(6)	-0.258(3)	0.051441	1.05(3)
H1o16	1	0.6428(8)	0.1064(7)	0.971(3)	0.050432	0.97(3)

H2o16a	0.5	0.6450(8)	0.0514(9)	0.213(3)	0.050432	0.98(3)
H2o16b	0.5	0.2750(8)	0.9425(9)	0.023(3)	0.050432	0.96(3)
H1o17	1	0.9642(8)	0.1427(9)	-0.088(2)	0.048273	0.96(2)
H2o17	1	0.8724(8)	0.1681(8)	0.017(3)	0.048273	0.97(3)

**Table S3** Selected interatomic distances (in Å) and angles (degree) in the structure of kaatialaite.

As-O	M-(H <sub>2</sub> O)			M-OH	
As1-O1	1.653(4)	O13-H1O13	0.981(11)	O3-H1O3a	0.98(3)
As1-O2	1.658(3)	O13-H2O13	0.980(10)	O3-H1O3b	0.98(3)
As1-O3	1.728(4)	O14-H1O14	0.982(12)	O4-H1O4a	0.98(3)
As1-O4	1.766(5)	O14-H2O14a	0.981(12)	O4-H1O4b	0.98(2)
As2-O5	1.634(4)	O14-H2O14b	0.98(2)	O6-H1O3	0.981(13)
As2-O6	1.746(4)	O15-H1O15	0.981(12)	O8-H1O4	0.982(18)
As2-O7	1.675(3)	O15-H2O15a	0.98(2)	O10-H1O10a	0.98(4)
As2-O8	1.666(5)	O15-H2O15b	0.981(14)	O10-H1O10b	0.98(3)
As3-O9	1.683(3)	O16-H1O16	0.981(14)	O12-H1O4	0.984(13)
As3-O10	1.719(5)	O16-H2O16a	0.981(16)	angle H1O <sub>i</sub> -O <sub>i</sub> -H2O <sub>i(a,b)</sub> [°]	
As3-O11	1.666(4)	O16-H2O16b	0.981(15)	O13	109.5(11)
As3-O12	1.730(4)	O17-H1O17	0.981(12)	O14 conf. a	109.5(11)
Fe1-O1	2.078(3)	O17-H2O17	0.981(14)	O14 conf. b	109.5(15)
Fe1-O2	2.060(4)			O15 conf. a	109.5(15)
Fe1-O5	2.028(4)			O15 conf. b	109.5(12)
Fe1-O7	2.088(4)			O16 conf. a	109.5(12)
Fe1-O9	2.040(3)			O16 conf. b	109.5(13)
Fe1-O11	2.030(3)			O17	109.5(12)
Strong hydrogen bonds O---H					
O1-H2O13	1.853(11)	O10-H2O14a	1.703(14)	O15-H1O3a	1.63(3)
O3-H2O15b	1.633(14)	O12-H1O6	1.805(13)	O16-H1O8	1.771(18)
O4-H2O16b	1.902(17)	O13-H1O12	1.546(13)	O16-H2O14b	1.87(2)
O4-H2O15a	1.91(2)	O13-H1O17	2.060(13)	O16-H1O4a	1.90(2)
O6-H1O16	1.966(15)	O14-H1O10b	1.70(3)	O17-H1O13	1.843(13)
O7-H2O17	1.980(15)	O14-H2O16a	1.868(16)	O17-H1O15	1.812(12)
O9-H1O14	1.770(10)	O15-H1O4b	1.90(2)	O17-H1O3b	2.08(3)

**Table S4** Atom positions, displacement parameters (Å<sup>2</sup>) for kaatialaite as obtained from kinematical refinement. The value d(A<sup>kin</sup>-A<sup>dyn</sup>) (Å) is the distance between the atoms of this model and the one after dynamical refinement.

atom	occu	x/a	y/b	z/c	Uiso	d(A <sup>kin</sup> -A <sup>dyn</sup> )
As1	1	0.1965(2)	0.0845(3)	0.1255(7)	0.0186(11)	0.027(5)
As2	1	0.2215(2)	0.2827(3)	0.1162(7)	0.0204(11)	0.014(4)
As3	1	0.4275(2)	0.1613(3)	0.1438(6)	0.0181(11)	0.021(5)
Fe1	1	0.2811(2)	0.1778(3)	0.6340(7)	0.0170(11)	0.016(5)

O1	1	0.1793(6)	0.1383(8)	0.3832(19)	0.025(3)	0.054(11)
O2	1	0.2718(7)	0.0927(8)	-0.115(2)	0.028(3)	0.065(12)
O3	1	0.0897(10)	0.0703(11)	-0.009(3)	0.067(5)	0.069(17)
O4	1	0.2226(7)	0.0026(9)	0.256(2)	0.041(4)	0.084(13)
O5	1	0.1929(7)	0.2257(8)	-0.1240(19)	0.028(3)	0.076(12)
O6	1	0.1200(6)	0.3122(7)	0.2377(18)	0.020(3)	0.077(10)
O7	1	0.2926(6)	0.2648(7)	0.3889(18)	0.016(3)	0.090(10)
O8	1	0.2783(8)	0.3523(9)	-0.021(2)	0.048(4)	0.122(14)
O9	1	0.3637(7)	0.1300(7)	0.4017(19)	0.026(3)	0.082(11)
O10	1	0.4859(8)	0.0956(9)	0.009(2)	0.050(4)	0.026(13)
O11	1	0.3808(6)	0.2049(8)	-0.1145(18)	0.024(3)	0.055(15)
O12	1	0.5032(8)	0.2126(7)	0.310(2)	0.037(3)	0.035(13)
O13	1	0.5160(8)	0.3211(8)	0.044(2)	0.050(4)	0.030(10)
O14	1	0.9212(8)	0.4866(6)	0.124(2)	0.041(4)	0.048(12)
O15	1	0.9206(7)	0.0224(7)	0.388(2)	0.031(3)	0.048(14)
O16	1	0.6807(7)	0.0800(7)	0.095(2)	0.028(3)	0.037(14)
O17	1	0.9228(8)	0.1378(8)	0.063(2)	0.058(4)	0.051(17)
H1o3a	0.5	0.096(8)	0.033(5)	-0.14(2)	0.080268	0.06(12)
H1o3b	0.5	0.038(6)	0.094(9)	0.054(17)	0.080268	0.08(9)
H1o4a	0.5	0.242(6)	0.979(6)	0.091(10)	0.048875	0.58(7)
H1o4b	0.5	0.828(5)	0.002(6)	0.63(2)	0.048875	0.20(12)
H1o6	1	0.0663(14)	0.308(4)	0.124(6)	0.024503	0.29(3)
H1o8	1	0.235(4)	0.380(3)	-0.120(7)	0.057961	0.17(5)
H1o10a	0.5	0.535(5)	0.122(6)	0.94(2)	0.059886	0.12(11)
H1o10b	0.5	0.450(5)	0.073(5)	0.86(2)	0.059886	0.23(11)
H1o12	1	0.509(4)	0.2573(15)	0.226(11)	0.044726	0.11(5)
H1o13	1	0.478(3)	0.325(3)	-0.124(8)	0.059873	0.16(5)
H2o13	1	0.5699(16)	0.346(2)	0.018(8)	0.059873	0.26(5)
H1o14	1	0.895(3)	0.4521(12)	0.001(8)	0.049495	0.06(4)
H2o14a	0.5	0.945(7)	0.4655(19)	0.294(11)	0.049495	0.07(7)
H2o14b	0.5	0.624(4)	0.017(4)	0.315(17)	0.049495	0.12(7)
H1o15	1	0.916(4)	0.0655(14)	0.290(10)	0.036933	0.08(6)
H2o15a	0.5	0.867(3)	0.014(3)	0.487(13)	0.036933	0.22(7)
H2o15b	0.5	0.072(5)	0.014(2)	-0.251(10)	0.036933	0.22(9)
H1o16	1	0.647(3)	0.114(2)	0.988(10)	0.033437	0.18(4)
H2o16a	0.5	0.642(3)	0.056(2)	0.220(10)	0.033437	0.11(4)
H2o16b	0.5	0.302(3)	0.9559(19)	0.031(10)	0.033437	0.49(4)
H1o17	1	0.961(2)	0.145(4)	-0.094(8)	0.069999	0.07(6)
H2o17	1	0.8661(17)	0.158(2)	0.022(10)	0.069999	0.22(4)

**Table S5** Results of the bond-valence analysis for kaatialaite (values given in valence units).

	O1	O2	O3	O4	O5	O6	O7	O8	O9	O10	O11	O12	O13	O14	O15	O16	O17	$\Sigma$ BV
Fe1	0.43	0.45			0.49		0.42		0.47		0.49							2.75
As1	1.37	1.36	1.11	1.00														4.84
As2					1.45	1.06	1.29	1.32										5.12
As3									1.26	1.14	1.32	1.11						4.83
H1O3a		0.01	0.86	0.01											0.19			1.07
H1O3b		0.01		0.86									0.02	0.01		0.07		0.97
H1O4a		0.01		0.86				0.01								0.10		0.98
H1O4b			0.02	0.86				0.01							0.10			0.99
H1O6				0.02	0.86							0.13						1.01
H1O8					0.01		0.86								0.14			1.01
H1O10a					0.05				0.86	0.01	0.03				0.02			0.97
H1O10b		0.012					0.008		0.86					0.16				1.04
H1O12										0.01	0.86	0.23						1.10
H1O13										0.01		0.86				0.12		0.99
H2O13	0.12			0.01								0.86						0.99
H1O14		0.01		0.01				0.13	0.02					0.86				1.03
H2O14a		0.01						0.01	0.17					0.86	0.01			1.06
H2O14b		0.01						0.01						0.86	0.11			0.99
H1O15										0.01		0.86			0.12			0.99
H2O15a			0.10				0.01							0.86				0.97
H2O15b			0.19											0.86				1.05
H1O16				0.09				0.02							0.86			0.97
H2O16a								0.01					0.11		0.86			0.98
H2O16b			0.10												0.86			0.96
H1O17			0.03										0.07			0.86		0.96
H2O17					0.09	0.02										0.86		0.97
$\Sigma$ BV <sub>Conf.a</sub>	1.92	1.86	2	2.84	1.97	2.07	1.8	2.23	1.87	2.22	1.84	2.13	1.96	1.9	1.92	2.08	1.96	
$\Sigma$ BV <sub>Conf.b</sub>	1.93	1.842	2.21	1.97	1.97	2.02	1.8	2.22	1.86	2.05	1.83	2.1	1.98	1.95	1.94	1.86	2.03	