Determination of Na^+ Cation Locations in Nanozeolite ECR-1 Through 3D ED Method

Taylan Örs [1,2], Irena Deroche [1,2], Corentin Chatelard [3,4], Mathias Dodin [3], Raquel Martinez-Franco [3], Alain Tuel [4] and Jean-Louis Paillaud [1,2,*]

[1] Université de Haute-Alsace, Institut de Science des Matériaux de Mulhouse, UMR 7361, 15 rue Jean Starcky, 68100 Mulhouse, France
[2] Université de Strasbourg, 67000 Strasbourg, France
[3] IFP Energies Nouvelles-Etablissement de Lyon, Rond-point de l'échangeur de Solaize, BP 3, 69360, Solaize, France
[4] Université de Lyon, Institut de Recherches sur la Catalyse et l'Environnement de Lyon, IRCELYON, UMR CNRS 5256, Université Lyon 1, 2 av. Einstein, 69626, Villeurbanne Cedex, France
* Correspondence: jean-louis.paillaud@uha.fr (J.-L.P.)

Supplementary Materials

Table of contents

Figure S1: TEM image of the measured ECR-1 crystal.

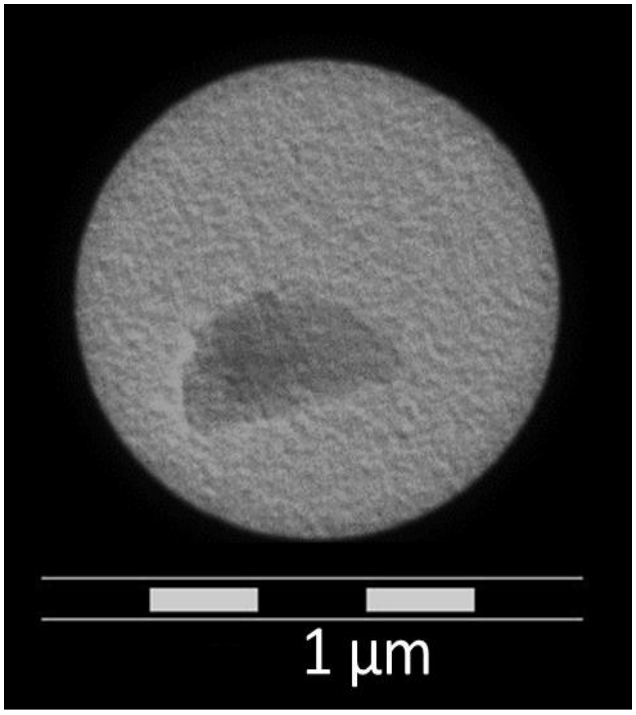

Figure S2: Energy dispersive X-ray spectroscopy (EDX) analyses of ECR-1 zeolite.

Analysis method: Phi-Rho-Z

Calibrated elements: Si, Al, Na, O on albite (Si/Al=3.03)

Atomic composition (%)								Formula $Na_xAl_xSi_{60-x}O_{120}$
Spectra	O	Na	Al	Si		Si/Al	Na/Al	x
Spectrum 1	65.82	5.1	4.87	24.21		4.97	1.05	10.05
Spectrum 2	65.23	5.15	5.11	24.5		4.79	1.01	10.35
Spectrum 3	65.23	5.1	5.09	24.58		4.83	1.00	10.29
Spectrum 4	64.98	5.19	5.2	24.64		4.74	1.00	10.46
Spectrum 5	65.01	5.21	5.18	24.6		4.75	1.01	10.44
Spectrum 6	65.75	5.12	5.13	24		4.68	1.00	10.57
Spectrum 7	65.35	5.14	5.19	24.32		4.69	0.99	10.55
Spectrum 8	65.31	5.08	5.13	24.48		4.77	0.99	10.40
Spectrum 9	65.1	5.14	5.2	24.56		4.72	0.99	10.48
Spectrum 10	65.13	5.39	5.12	24.35		4.76	1.05	10.42
Mean values	**65.29**	**5.16**	**5.12**	**24.42**		Mean values		
Standard deviation	**0.29**	**0.09**	**0.10**	**0.20**		4.77	1.01	10.40

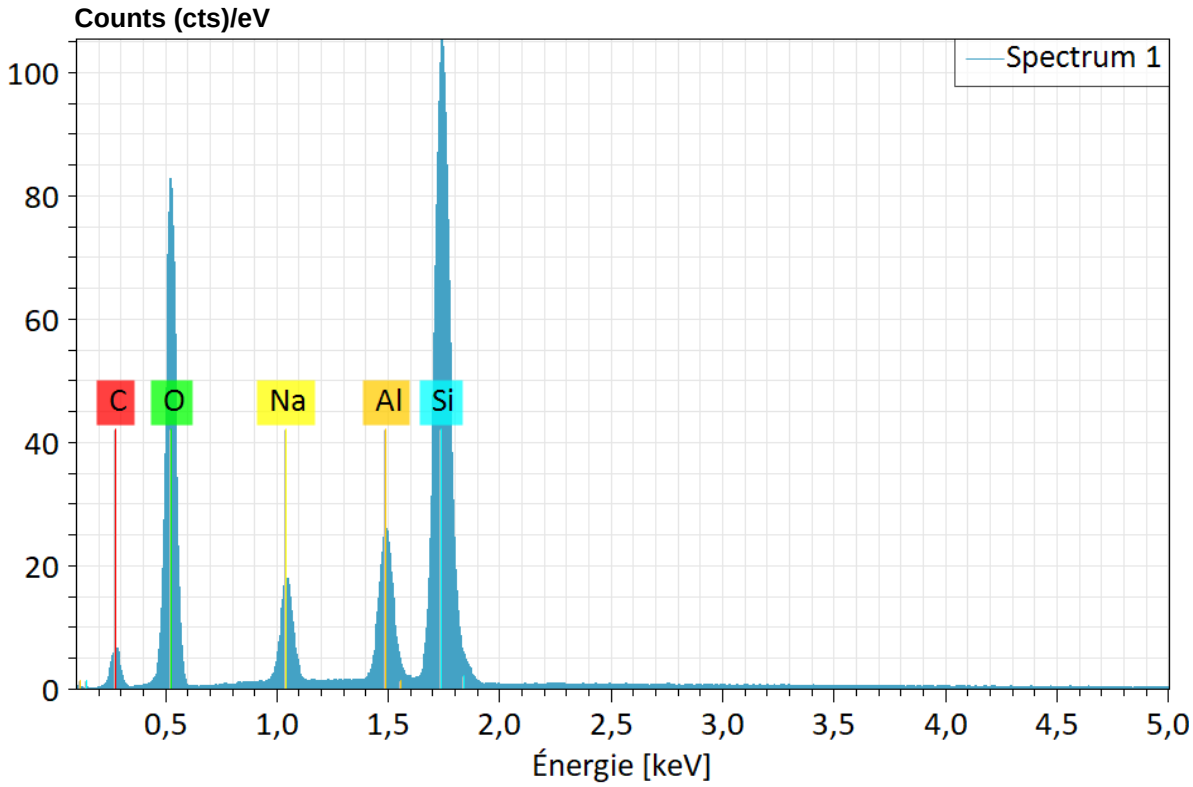
Counts (cts)/eV
Spectrum 1
100
80
60
40
20
0
C
O
Na
Al
Si
0,5
1,0
1,5
2,0
2,5
3,0
3,5
4,0
4,5
5,0
Énergie [keV]

Figure S3: Projections along [100] of the ECR-1 zeolite. In (a), the structure obtained earlier by Gualtieri et al. (Ref. [11] in the text) through a Rietveld refinement from PXRD data is shown, where Na^+ cations are labeled as C1 to C4. In (b), the resulting structure after the dynamical refinement of 3D ED data collected during the current study is presented.

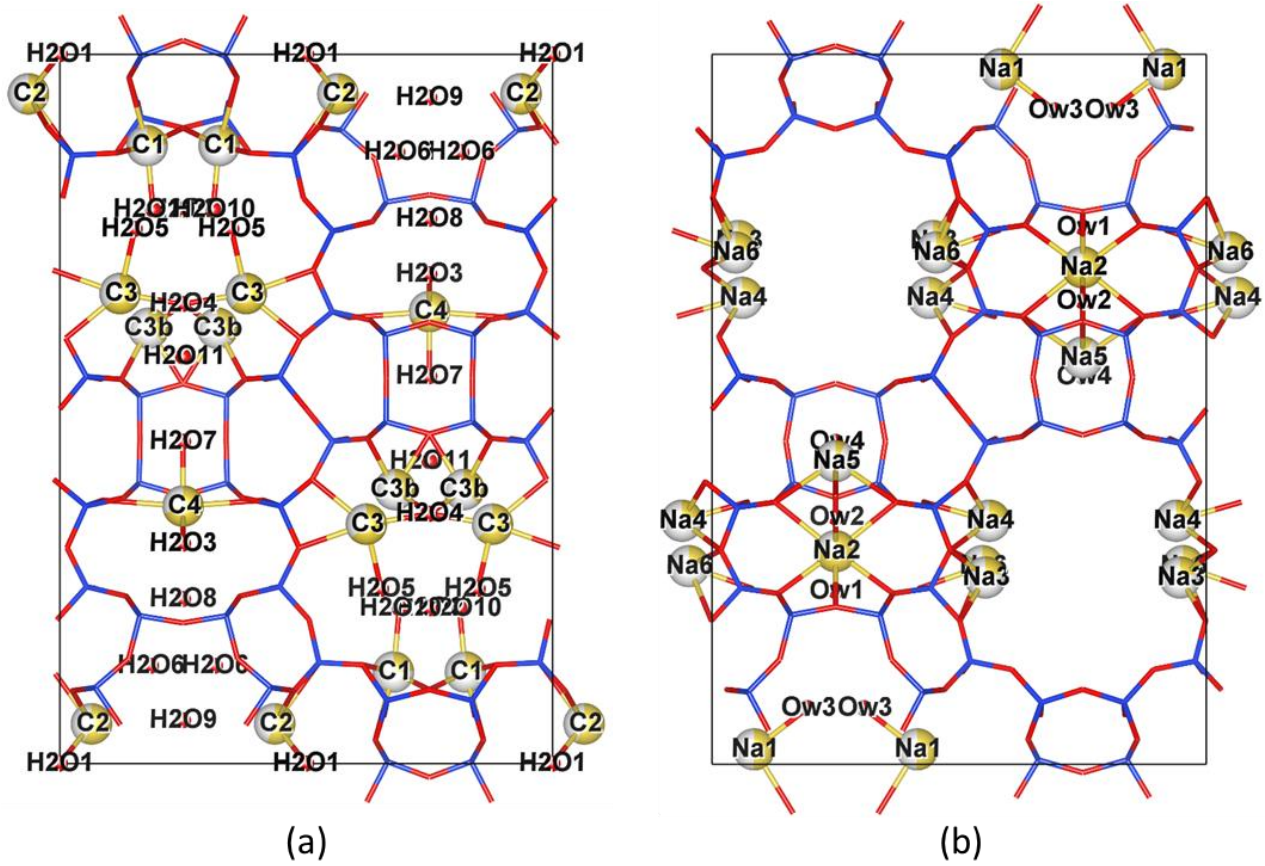

Table S1: Fractional atomic coordinates and isotropic displacement parameters (Å^2) of ECR-1(3D ED, kinematical refinement).

Atom	Wyck.	Site	s.o.f.	x/a	y/b	z/c	U [Å^2]
Si1	8g	1		0.0459(14)	0.0529(8)	0.1390(4)	0.0470
Si2	8g	1		0.5453(13)	0.5549(8)	0.0451(4)	0.0400
Si3	4e	m..		1/4	0.0713(13)	0.3989(7)	0.0620
Si4	8g	1		0.5440(13)	0.0578(8)	0.2512(4)	0.0440
Si5	4e	m..		1/4	0.6585(13)	0.8956(5)	0.0560
Si6	8g	1		0.0472(14)	0.0263(9)	0.6467(4)	0.0510
Si7	4e	m..		1/4	0.6612(13)	0.0201(5)	0.0440
Si8	8g	1		0.0437(13)	0.6646(8)	0.4120(4)	0.0490
Si9	4e	m..		1/4	0.1630(14)	0.2913(7)	0.0680
Si10	4e	m..		1/4	0.1553(13)	0.5044(7)	0.0630
O1	8g	1		0.514(3)	0.5214(18)	0.1015(8)	0.0980
O2	8g	1		0.066(3)	0.1285(16)	0.2595(8)	0.0870
O3	8g	1		0.076(3)	0.628(2)	0.0434(9)	0.1130
O4	4e	m..		1/4	0.111(2)	0.4536(13)	0.0920
O5	4e	m..		1/4	0.079(2)	0.9581(10)	0.0770
O6	4e	m..		1/4	0.073(2)	0.1301(9)	0.0690
O7	4e	m..		1/4	0.576(2)	0.7488(9)	0.0620
O8	2b	mm2		1/4	3/4	0.0339(16)	0.0880
O9	2a	mm2		1/4	1/4	0.2849(16)	0.0870
O10	8g	1		0.066(3)	0.1432(15)	0.5391(7)	0.0740
O11	8g	1		0.004(2)	0.0128(14)	0.7053(7)	0.0570
O12	2a	mm2		1/4	1/4	0.4908(17)	0.1040
O13	2b	mm2		1/4	3/4	0.8790(16)	0.0930
O14	8g	1		0.017(2)	0.0332(15)	0.1954(7)	0.0740
O15	8g	1		0.075(3)	0.0254(17)	0.3921(7)	0.0860
O16	8g	1		0.559(3)	0.122(2)	0.1309(9)	0.1000
O17	4f	.m.		0.505(3)	1/4	0.6008(9)	0.0700
O18	4e	m..		1/4	0.648(3)	0.9558(13)	0.1100
O19	8g	1		0.001(2)	0.6094(16)	0.3613(7)	0.0690
O20	4e	m..		1/4	0.141(3)	0.3526(13)	0.1110
O21	4e	m..		1/4	0.007(2)	0.6303(9)	0.0630
O22	4e	m..		1/4	0.657(2)	0.4296(10)	0.0750
O23	4c	-1		0	0	0	0.1900
Na1	4e	m..	0.4	1/4	0.573(8)	0.521(4)	0.1600
Na2	4f	.m.	0.46	0.537(10)	1/4	0.211(3)	0.1600
Na3	4e	m..	0.23	1/4	0.596(15)	0.233(7)	0.1600
Na4	2b	mm2	0.27	1/4	3/4	0.738(8)	0.1600
Na5	2a	mm2	0.53	1/4	1/4	0.159(4)	0.1600
Na6	2a	mm2	0.39	1/4	1/4	0.059(5)	0.1600
Na7	4f	.m.	0.2	0.70(3)	1/4	0.420(6)	0.1600
Na8	4e	m..	0.33	1/4	0.080(9)	0.820(4)	0.1600

Table S2: Fractional atomic coordinates and isotropic displacement parameters (Å^2) of ECR-1 (3D ED, dynamical refinement).

Atom	Wyck.	Site	s.o.f.	x/a	y/b	z/c	U [Å^2]
Si1	8g	1		0.5476(6)	0.4488(5)	0.36172(17)	0.0334(17)
Si2	8g	1		0.4520(6)	0.5545(4)	0.45250(15)	0.0218(14)
Si3	4e	m..		3/4	0.4270(7)	0.1018(3)	0.046(3)
Si4	8g	1		0.4528(6)	0.4422(5)	0.24804(16)	0.0241(15)
Si5	4e	m..		1/4	0.3366(7)	0.3955(2)	0.025(3)
Si6	8g	1		0.4550(7)	0.5228(5)	0.14502(17)	0.0301(17)
Si7	4e	m..		1/4	0.3344(7)	0.5186(2)	0.039(3)
Si8	8g	1		0.5429(6)	0.6665(6)	0.08679(16)	0.0337(18)
Si9	4e	m..		3/4	0.3398(7)	0.2102(3)	0.043(3)
Si10	4e	m..		3/4	0.3396(5)	-0.0060(2)	0.044(3)
O1	8g	1		0.4865(11)	0.5137(8)	0.3979(3)	0.0702(9)
O2	8g	1		0.5667(14)	0.3664(6)	0.2405(3)	0.0702
O3	8g	1		0.0668(11)	0.3731(6)	0.5385(4)	0.0702
O4	4e	m..		3/4	0.3853(10)	0.0471(4)	0.0702
O5	4e	m..		1/4	0.5738(15)	0.4587(5)	0.0702
O6	4e	m..		3/4	0.4285(14)	0.3694(5)	0.0702
O7	4e	m..		1/4	0.4195(16)	0.2478(5)	0.0702
O8	2a	mm2		1/4	1/4	0.5363(8)	0.0702
O9	2b	mm2		3/4	1/4	0.2218(9)	0.0702
O10	8g	1		0.4254(12)	0.6394(10)	0.0350(3)	0.0702
O11	8g	1		0.5023(11)	0.5013(7)	0.2025(3)	0.0702
O12	2b	mm2		3/4	1/4	0.0119(8)	0.0702
O13	2a	mm2		1/4	1/4	0.3758(8)	0.0702
O14	8g	1		0.5095(11)	0.4822(10)	0.3013(4)	0.0702
O15	8g	1		0.5631(13)	0.4701(10)	0.1051(3)	0.0702
O16	8g	1		0.4373(14)	0.3727(7)	0.3692(4)	0.0702
O17	4f	.m.		0.5021(18)	3/4	0.1054(6)	0.0702
O18	4e	m..		1/4	0.3508(14)	0.4549(5)	0.0702
O19	8g	1		0.5063(14)	0.6067(13)	0.1334(4)	0.0702
O20	4e	m..		3/4	0.3630(14)	0.1475(5)	0.0702
O21	4e	m..		1/4	0.5069(15)	0.1301(4)	0.0702
O22	4e	m..		3/4	0.6645(15)	0.0695(5)	0.0702
O23	4d	-1		1/2	1/2	1/2	0.0702
Na1	4e	m..	0.507(13)	1/4	0.415(3)	0.0209(9)	0.1200 [a]
Na2	4f	.m.	0.789(15)	0.492(2)	1/4	0.3001(7)	0.1200 [a]
Na3	4e	m..	0.212(14)	3/4	0.553(6)	0.263(2)	0.1200 [a]
Na4	4e	m..	0.209(15)	3/4	0.561(3)	0.343(3)	0.1200 [a]
Na5	4f	.m.	0.082(19)	0.93(2)	1/4	0.427(4)	0.1200 [a]
Na6	4e	m..	0.240(15)	1/4	0.547(3)	0.279(2)	0.1200 [a]
Ow1	2a	mm2	0.71(3)	1/4	1/4	0.2439(13)	0.1300 [a]
Ow2	2b	mm2	0.70(3)	3/4	1/4	0.3477(13)	0.1300 [a]
Ow3	8g	1	0.159(12)	0.313(9)	0.307(4)	0.079(2)	0.1300 [a]
Ow4	2b	mm2	0.36(4)	3/4	1/4	0.454(3)	0.1300 [a]

[a] These ADPs have been fixed to a reasonable values during the refinement.

Table S3: Si—O bond lengths and O—Si—O angles of ECR-1 (3D ED, dynamical refinement).

Si1	O1	1.5531(139)	O6	1.5704(77)	112.410(805)
	O1	1.5531(139)	O16	1.6010(144)	114.146(796)
	O1	1.5531(139)	O14	1.6850(126)	103.656(548)
	O6	1.5704(77)	O16	1.6010(144)	106.675(801)
	O6	1.5704(77)	O14	1.6850(126)	111.122(821)
	O16	1.6010(144)	O14	1.6850(126)	108.846(672)
Si2	O5	1.5591(76)	O3	1.5689(119)	109.536(792)
	O5	1.5591(76)	O1	1.5997(106)	110.273(800)
	O5	1.5591(76)	O23	1.6000(53)	105.928(702)
	O3	1.5689(119)	O1	1.5997(106)	114.537(626)
	O3	1.5689(119)	O23	1.6000(53)	105.397(472)
	O1	1.5997(106)	O23	1.6000(53)	110.728(491)
Si3	O4	1.5884(153)	O15	1.5989(135)	105.792(789)
	O4	1.5884(153)	O15	1.5989(135)	105.792(789)
	O4	1.5884(153)	O20	1.6368(222)	107.811(862)
	O15	1.5989(135)	O15	1.5989(135)	122.112(829)
	O15	1.5989(135)	O20	1.6368(222)	107.323(871)
	O15	1.5989(135)	O20	1.6368(222)	107.323(871)
Si4	O7	1.5714(88)	O14	1.5990(132)	112.021(886)
	O7	1.5714(88)	O2	1.6107(132)	107.128(827)
	O7	1.5714(88)	O11	1.617(12)	112.753(832)
	O14	1.5990(132)	O2	1.6107(132)	109.695(660)
	O14	1.5990(132)	O11	1.617(12)	105.389(539)
	O2	1.6107(132)	O11	1.617(12)	109.859(662)
Si5	O18	1.5448(144)	O13	1.6260(136)	117.550(877)
	O18	1.5448(144)	O16	1.6842(120)	109.417(843)
	O18	1.5448(144)	O16	1.6842(120)	109.417(843)
	O13	1.6260(136)	O16	1.6842(120)	103.831(706)
	O13	1.6260(136)	O16	1.6842(120)	103.831(706)
	O16	1.6842(120)	O16	1.6842(120)	112.728(694)
Si6	O11	1.5643(94)	O19	1.5743(239)	110.890(666)
	O11	1.5643(94)	O21	1.6070(75)	113.416(793)
	O11	1.5643(94)	O15	1.6088(141)	110.048(486)
	O19	1.5743(239)	O21	1.6070(75)	110.844(910)
	O19	1.5743(239)	O15	1.6088(141)	108.225(938)
	O21	1.6070(75)	O15	1.6088(141)	103.036(824)
Si7	O8	1.5732(134)	O3	1.6182(105)	108.516(716)
	O8	1.5732(134)	O3	1.6182(105)	108.516(716)
	O8	1.5732(134)	O18	1.6602(145)	116.93(85)
	O3	1.6182(105)	O3	1.6182(105)	115.890(678)
	O3	1.6182(105)	O18	1.6602(145)	103.609(785)
	O3	1.6182(105)	O18	1.6602(145)	103.609(785)
Si8	O17	1.5943(114)	O22	1.6131(57)	106.689(847)
	O17	1.5943(114)	O19	1.6260(187)	111.159(816)

	O17	1.5943(114)	O10	1.6652(106)	114.153(733)
	O22	1.6131(57)	O19	1.6260(187)	110.468(924)
	O22	1.6131(57)	O10	1.6652(106)	106.375(843)
	O19	1.6260(187)	O10	1.6652(106)	107.885(526)
Si9	O9	1.6300(131)	O2	1.6470(112)	101.374(738)
	O9	1.6300(131)	O2	1.6470(112)	101.374(738)
	O9	1.6300(131)	O20	1.6610(161)	114.954(883)
	O2	1.6470(112)	O2	1.6470(112)	112.853(686)
	O2	1.6470(112)	O20	1.6610(161)	112.657(816)
	O2	1.6470(112)	O20	1.6610(161)	112.657(816)
Si10	O10	1.5551(100)	O10	1.5551(100)	115.218(743)
	O10	1.5551(100)	O4	1.5877(142)	106.659(716)
	O10	1.5551(100)	O12	1.6637(104)	111.327(705)
	O10	1.5551(100)	O4	1.5877(142)	106.659(716)
	O10	1.5551(100)	O12	1.6637(104)	111.327(705)
	O4	1.5877(142)	O12	1.6637(104)	104.886(679)