Supplementary Table 3. Evaluation of the observed powder diffraction pattern up to 40° 2θ of the monoclinic LT-modification of alunogen for Cu-$K\alpha_1$ radiation (T = -100 °C).

h	k	l	d_{hkl}	2θ	rel. Intensity, %
0	2	0	13.423	6.579	3.8
0	0	1	7.327	12.068	4.4
0	1	1	7.069	12.511	9.9
0	4	0	6.711	13.180	0.8
0	2	1	6.431	13.756	3.7
1	0	0	6.008	14.730	2.6
1	1	0	5.863	15.097	0.3
0	3	1	5.669	15.617	1.6
0	4	1	4.949	17.907	0.5
1	-1	-1	4.890	18.124	3.6
1	-2	-1	4.663	19.013	3.2
1	4	0	4.476	19.815	6.2
0	6	0	4.474	19.825	50
1	-3	-1	4.347	20.412	100
1	1	1	4.319	20.546	6.2
1	2	1	4.160	21.338	11.9
1	-4	-1	3.996	22.228	5.70
1	3	1	3.931	22.598	74.1
0	6	1	3.818	23.273	1.7
1	4	1	3.665	24.259	8.2
0	0	2	3.663	24.272	32.0
1	-5	-1	3.648	24.374	3.6
0	1	2	3.630	24.501	7.0
1	6	0	3.588	24.788	16.5
0	2	2	3.534	25.175	11.5
0	7	1	3.398	26.204	22.7
1	5	1	3.392	26.249	6.0
0	3	2	3.390	26.262	4.6
0	8	0	3.355	26.539	3.3
1	-6	-1	3.326	26.778	10.5
-1	0	2	3.325	26.782	6.5
1	-1	-2	3.300	26.991	5.3
1	-3	-2	3.117	28.609	9.6
0	8	1	3.051	29.246	27.2
1	-7	-1	3.037	29.383	15.7
0	5	2	3.026	29.490	9.2
2	0	0	3.004	29.712	24.1
1	-4	-2	2.980	29.959	32.9
1	0	2	2.962	30.147	5.8
1	1	2	2.944	30.334	17.1
1	2	2	2.892	30.890	8.1
1	7	1	2.884	30.978	6.8
2	-2	-1	2.849	31.369	24
1	-5	-2	2.827	31.618	9.4
2	-3	-1	2.772	32.263	13.3
2	4	0	2.742	32.628	17.9
1	4	2	2.709	33.028	9.1

2	-4	-1	2.674	33.480	5.3
2	5	0	2.621	34.170	31.9
2	3	1	2.550	35.153	8.2
2	6	0	2.494	35.976	75.3
2	4	1	2.473	36.284	12.6
1	6	2	2.469	36.344	7.1
1	9	1	2.464	36.421	9.3
1	10	0	2.451	36.631	7.8
0	1	3	2.432	36.922	6.1
2	7	0	2.365	38.014	19.5
1	-1	-3	2.363	38.041	15.3
1	-2	-3	2.336	38.498	20.8
2	-4	-2	2.331	38.577	8.6
2	-7	-1	2.321	38.762	9.1
0	9	2	2.313	38.900	7.3
0	4	3	2.295	39.217	5.8
1	-3	-3	2.293	39.249	12.5

Supplementary Figure. Result of a structure independent LeBail-fit of the X-ray powder diffraction pattern of the triclinic *RT*-form of alunogen collected at 20 °C. Observed step intensities are represented by a black line. Calculated step intensities (in red) have been modeled based on the crystal structure obtained from the single-crystal diffraction study. Tick marks for the Bragg peaks are given. The lower blue line represents the difference curve between observed and calculated step intensities.