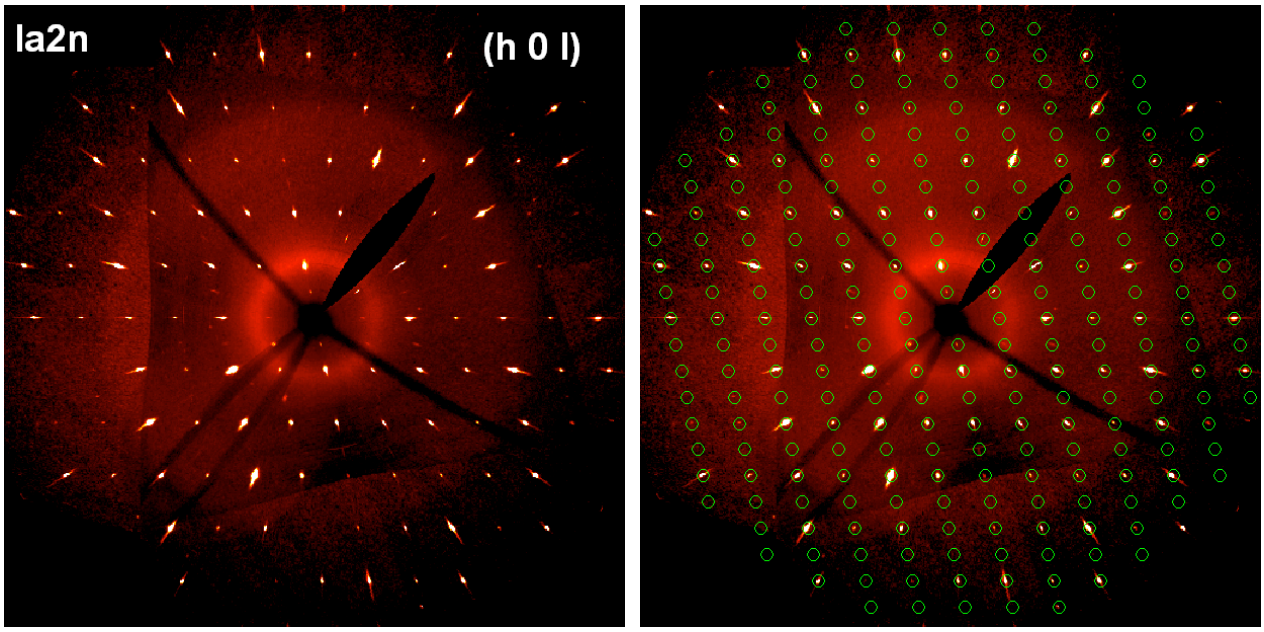
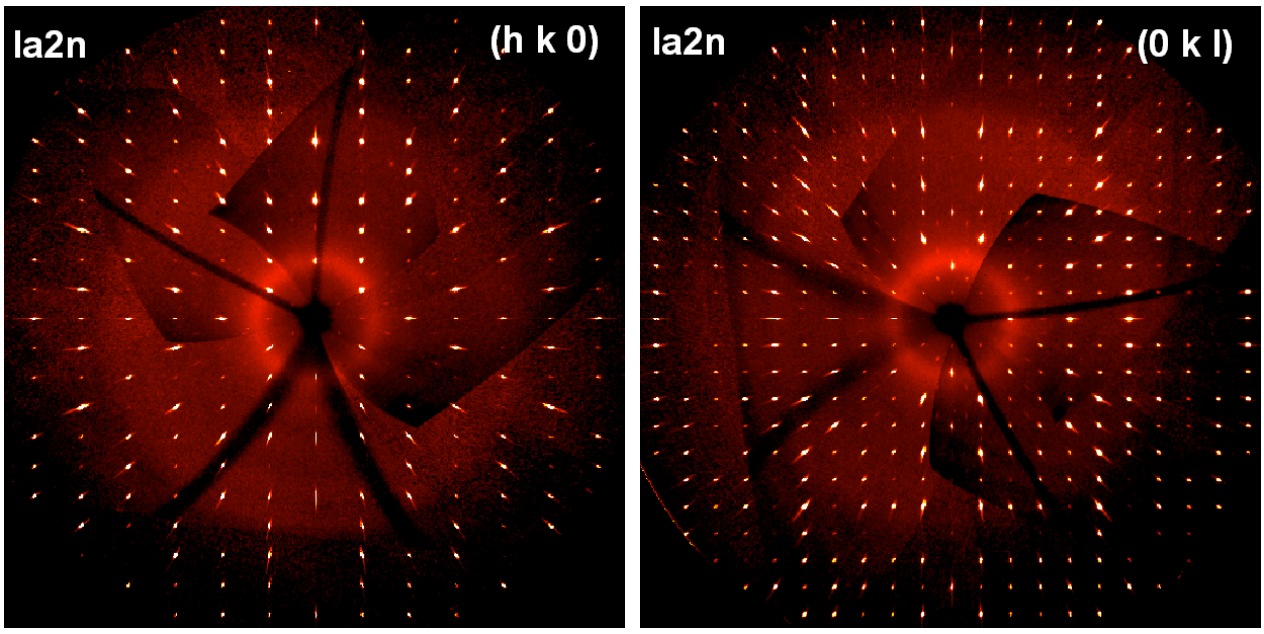
Test for possible twinning

For $LiAlGe_2O_6$ crystals, used in this study, Pseudo-precession image simulation was used to exclude possible twinning of crystals. Images were calculated using the APEX2 Software (Bruker - Nonius 2007), for a maximum resolution of 0.7 Å and layer thicknesses of 0.05 to 0.3, with no difference between the different thicknesses.

Crystal LA2n

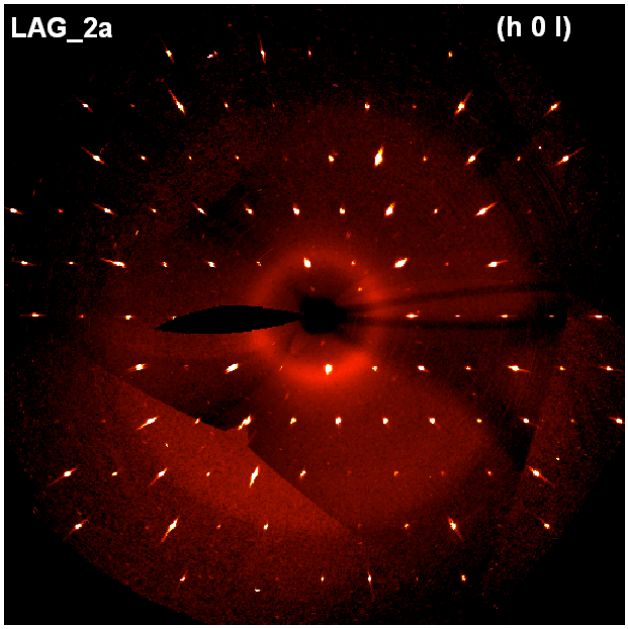


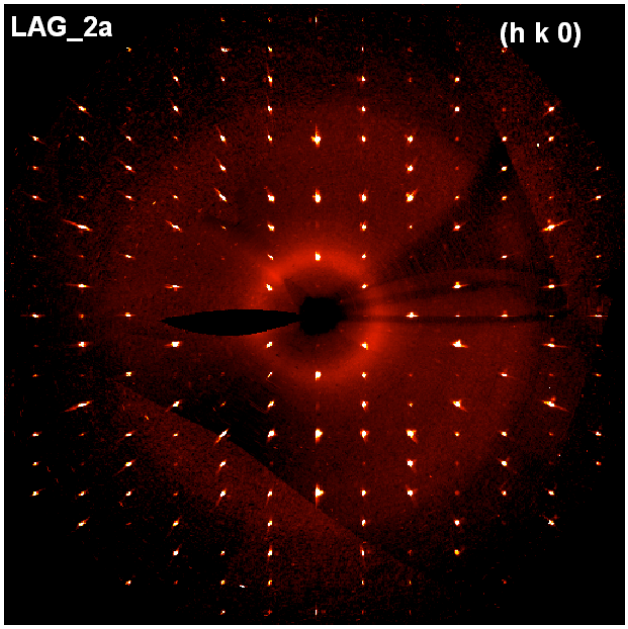
These images display the (h 0 l) plane, without and with an overlay of possible Bragg reflections in the monoclinic unit cell. No suspicious doubling of reflections is visible, thus crystals appear to be untwinned as expected also from optical inspection under the polarization mikroskope.

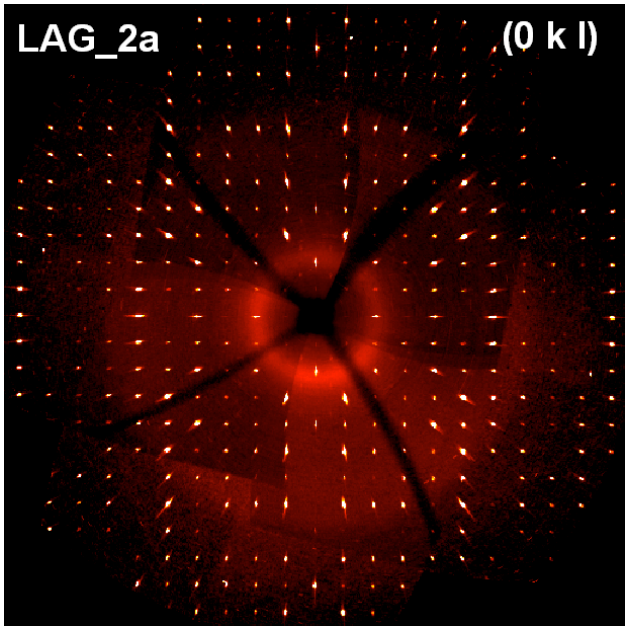


Crystal LAG_2A

(data not shown in Tables 1 -3)

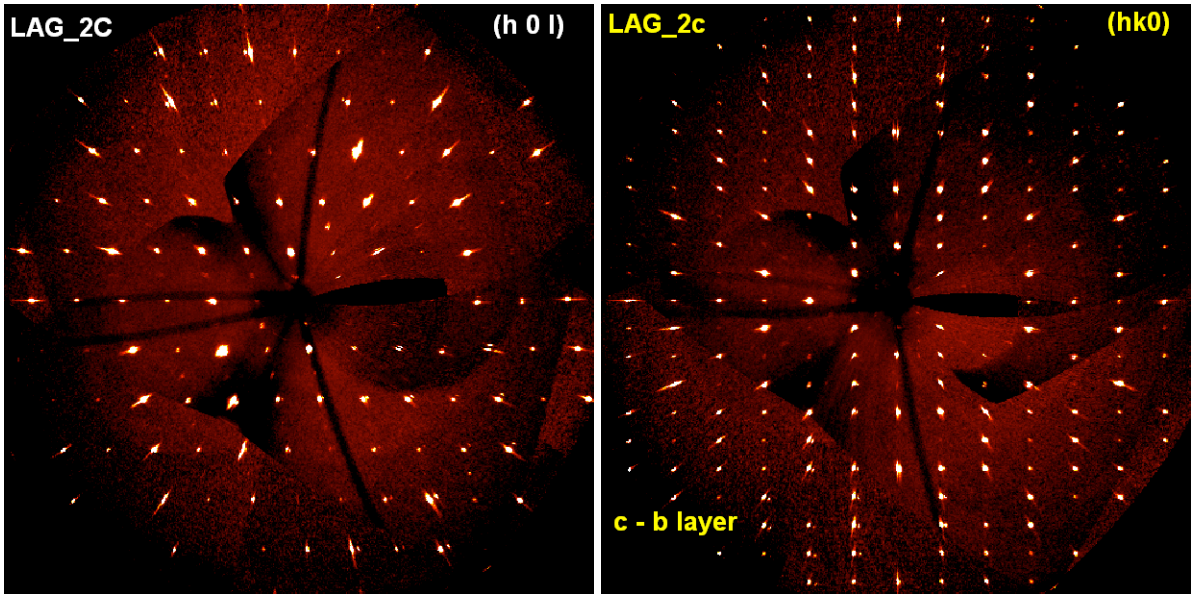






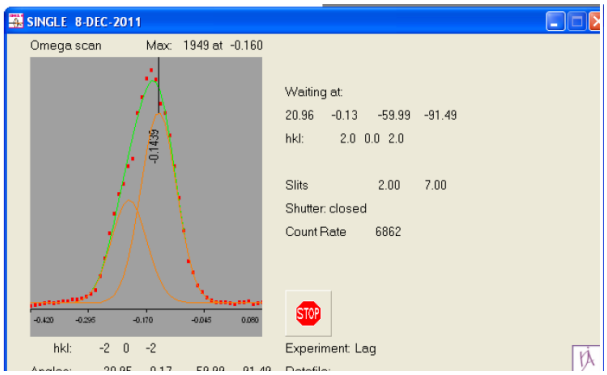
Also this crystals shows no twinning and has sharp reflections in the simulations of precession photographs (limit d = 0.8 Å. layer thickness 0.05)

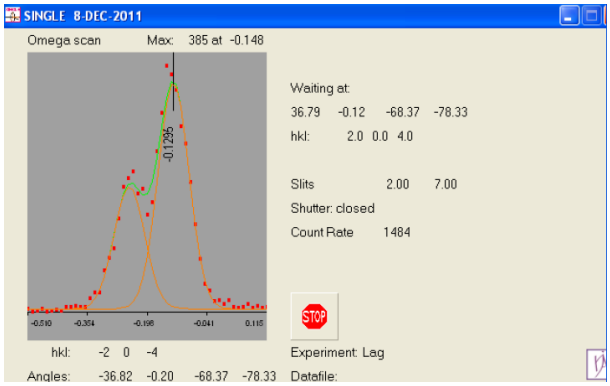
To see how a twinned crystal of $LiAlGe_2O6$ can look like, we here add two precession sections of a crystal which shows intermediate twinning.

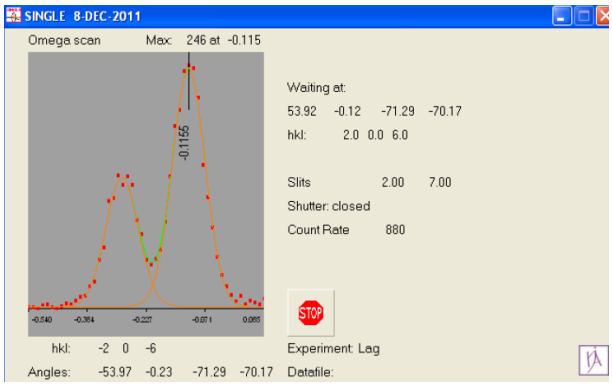


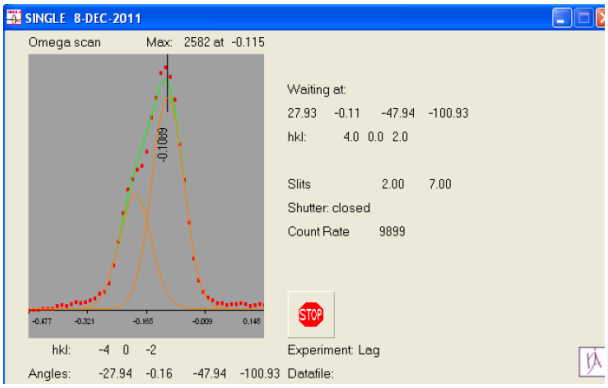
Especially within the a -c (h 0 l) section these is a distinct doubling of reflections, which is as prominent that it cannon be overseen. Also the (h k 0) section shows such doubling of reflections which is not seen in the images above. All in all it may be noted that data integration of this twinned crystal yield a primitive unit cell with symmetry $P2_1/n$.

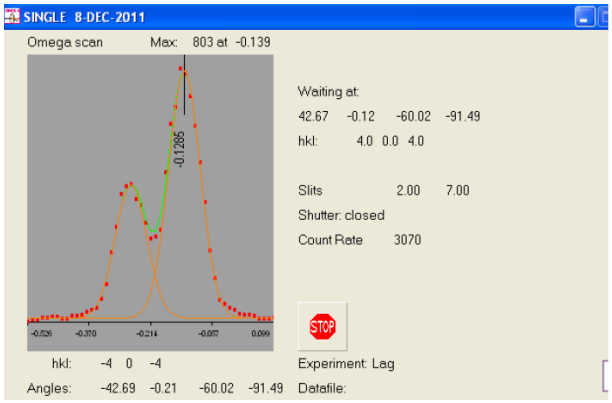
If our samples would have been twinned, this could have caused an erroneous determination of the space group from $P2_1/n$ to $C2/c$. A typical twinning law within the $C2/c$ clinopyroxenes in (1 0 0), which causes the presence of h + k = 2n+1 reflections, typical for a primitive Bravais lattice. Such a twinning law - however, should show split reflections with indices (h 0 l) for h + l = 2n reflections. Therefore we used a point detector with very long sample - detector distance to analyse such reflections up to high resolution (e.g. 60° in 2θ). Results are shown in the images below. We have measured, in detail, the (2 0 2), (2 0 4), (2 0 6), (4 0 2), (4 0 4) and the (4 0 6) reflections. The unit cell setting is the conventional for $P2_1/c$ or $C2/c$ with a ~ 9.8 Å, b ~ 8.4 Å, c ~ 5.3 Å and the monoclinic angle ~110°. As expected the reflections only show a clear $K\alpha_1$ - $K\alpha_2$ split, which is noticeable even at low angles as a distinct shoulder with the two peaks having equal peak broadening and the $K\alpha_2$ peak half intensity, but - most important - no indication of any split due to a twinning is evident. Moreover, the reflections all show fine peak profiles with a very small line width of 0.06° in omega.

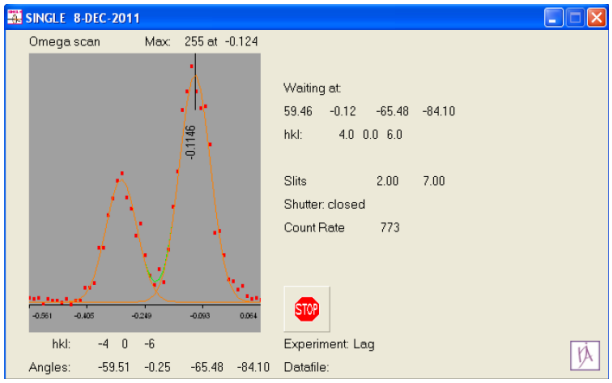

SINGLE 8-DEC-2011
Omega scan Max: 1949 at -0.160
-0.1439
Waiting at:
20.96 -0.13 -59.99 -91.49
hkl: 2.0 0.0 2.0
Slits 2.00 7.00
Shutter: closed
Count Rate 6862
STOP
hkl: -2 0 -2
Experiment: Lag


SINGLE 8-DEC-2011
Omega scan Max: 385 at -0.148
-0.1295
Waiting at:
36.79 -0.12 -68.37 -78.33
hkl: 2.0 0.0 4.0
Slits 2.00 7.00
Shutter: closed
Count Rate 1484
STOP
hkl: -2 0 -4
Experiment: Lag
Angles: -36.82 -0.20 -68.37 -78.33 Datafile:


SINGLE 8-DEC-2011
Omega scan Max: 246 at -0.115
-0.1155
Waiting at:
53.92 -0.12 -71.29 -70.17
hkl: 2.0 0.0 6.0
Slits 2.00 7.00
Shutter: closed
Count Rate 880
STOP
hkl: -2 0 -6
Experiment: Lag
Angles: -53.97 -0.23 -71.29 -70.17 Datafile:


SINGLE 8-DEC-2011
Omega scan Max: 2582 at -0.115
-0.1089
Waiting at:
27.93 -0.11 -47.94 -100.93
hkl: 4.0 0.0 2.0
Slits 2.00 7.00
Shutter: closed
Count Rate 9899
STOP
hkl: -4 0 -2
Experiment: Lag
Angles: -27.94 -0.16 -47.94 -100.93 Datafile:


SINGLE 8-DEC-2011
Omega scan Max: 803 at -0.139
-0.1286
Waiting at:
42.67 -0.12 -60.02 -91.49
hkl: 4.0 0.0 4.0
Slits 2.00 7.00
Shutter: closed
Count Rate 3070
STOP
hkl: -4 0 -4
Experiment: Lag
Angles: -42.69 -0.21 -60.02 -91.49 Datafile:


SINGLE 8-DEC-2011
Omega scan Max: 255 at -0.124
-0.1146
Waiting at:
59.46 -0.12 -65.48 -84.10
hkl: 4.0 0.0 6.0
Slits 2.00 7.00
Shutter: closed
Count Rate 773
STOP
hkl: -4 0 -6
Experiment: Lag
Angles: -59.51 -0.25 -65.48 -84.10 Datafile: