

Kappa Geometry

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Abstract

The investment in modern equipment and the development of highly automated beamline control software on the public MX-beamlines at the ESRF now allows standard X-ray diffraction experiments, even multiple anomalous diffraction (MAD), to be easily and robustly performed. But in general the success of these more routine experiments is still dependent on the quality of the crystals obtained. Modern structural biology projects have however become ever more complex and their success is often dependent on a few poorly diffracting crystals that are radiation sensitive. Such projects require a much more careful approach in data collection. One possibility, often used in small molecule crystallography, is the reorientation of a crystal using a multi-axis goniometer for data collection. Unfortunately most multi axis goniometers are very large and therefore difficult to integrate into modern MX-beamlines. In Grenoble we have developed an alternative system, a compact mini-kappa (MK3) goniometer head that is mounted on most of our MD2/MD2M diffractometers and fully compatible with high throughput crystal screening. In parallel, a user friendly software package "Strategy for Aligned Crystals" (STAC) has been developed. This software provides an accurate and simple access to new crystal reorientations. The MK3 and STAC allow a crystal to be reoriented in the X-ray beam for optimal data collection strategies or "smart data collection schemes". Here we will present the advantages of using STAC and an introduction on how to use this software for your future experiments.

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Kappa goniometers

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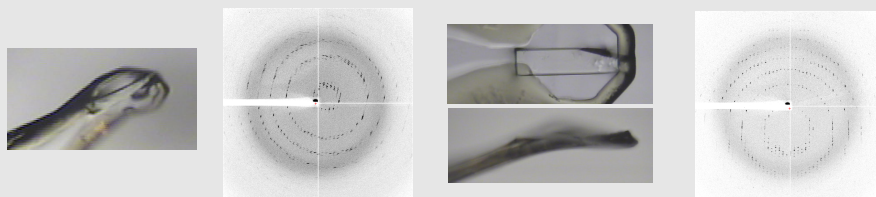


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Potential for challenging projects

- Radiation damage
 - Minimise the oscillation range
 - Measure the intensities of the Bijvoet pair reflections simultaneously
- Align a crystal to a previous orientation
- Realign a crystal to avoid defects



Crystal courtesy of J. Timmins (ESRF)

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Improving experimental phases

Exploiting the anisotropy of anomalous scattering boosts the phasing power of SAD and MAD experiments

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We would like to dedicate this article to Lucio and David Tompkins, whose pioneering studies of ASD provided much inspiration through the course of this work.

The X-ray polarization anisotropy of anomalous scattering in crystals of brominated nucleic acids and ribonucleic proteins is shown to have significant effects on the diffraction data collected at an absorption edge. For conventionally collected single- or multi-wavelength anomalous diffraction data, the main manifestation of the anisotropy of anomalous scattering is the leakage of the equivalence between symmetry-related reflections, inducing intensity differences between them that can be exploited to yield extra phase information in the structure solution process. A new formula for describing the anisotropy of anomalous scattering, which allows these effects to be incorporated into the general scheme of experimental phasing methods using an extended Harker construction is introduced. This requires a paradigm shift in the data processing strategy, since the usual separation of the data merging and phasing steps is abandoned. The data are kept unmerged down to the Harker construction, where the symmetry breaking is explicitly modelled and reduced and becomes a source of supplementary phase information. These ideas have been implemented in the phasing program SHARP. Experiments using actual data show that exploitation of the anisotropy of anomalous scattering can deliver substantial extra phasing power compared with conventional approaches using the same raw data. Examples are given that show improvements in the phases which are typically of the same order of magnitude as those obtained in a conventional approach by adding a second-wavelength data set to a SAD experiment. It is argued that such gains, which come essentially for free, i.e. without the collection of new data, are highly significant, since radiation damage can frequently preclude the collection of a second-wavelength data set. Finally, further developments in synchrotron instrumentation and in the design of data-collection strategies that could help to maximize these gains are outlined.

Keywords: anisotropy of anomalous scattering; phasing; SAD; MAD; polarized resonant diffraction.

1. Introduction

Experimental phasing methods based on anomalous scattering (AS), as currently implemented, describe the phenomenon of AS in terms of the wavelength-dependent anomalous scattering factors f' and f'' for certain heavier atoms. These quantities are scalar-valued and hence describe an isotropic AS behaviour without reference to any notion of directionality. It is known, however, that the phenomenon of AS at or very near absorption edges involves an extra degree of complexity, namely anisotropic behaviour through its dependence on the polarization of the X-ray beam, that has not so far been exploited for phase determination.

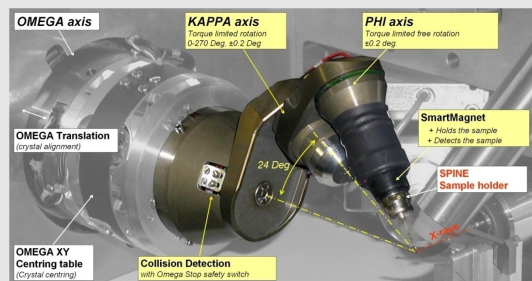
Potential to use the anisotropic behaviour of anomalous scattering atoms with respect to the polarization of the X-ray beam.

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The Grenoble MiniKappa goniometer head or MK3



- MiniKappa integrated into MD2/MD2M diffractometers
- Dedicated software: Strategy for Aligned Crystals, (STAC)
 - Also integrated into DNA for multi-axis goniometers on synchrotron beamlines.

Brockhauser, S., Ravelli, R.G.B. *et al.* (2009)
Reorienting Macromolecular Crystals,
J. Appl. Cryst. (in preparation)



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MK tab in mxCuBE

☒ Minikappa

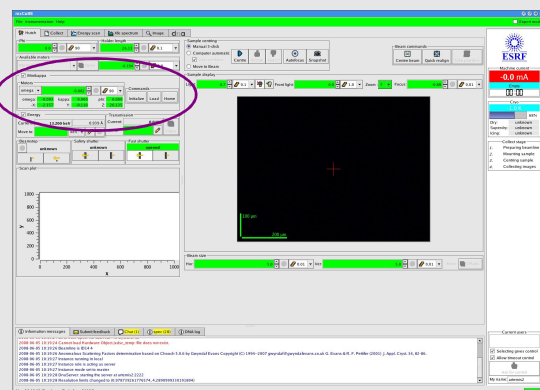
Motors

omega: -0.002

omega: -0.003 kappa: 0.000 phi: 0.000

-X: -2.357 Y: -0.110 Z: 26.139

Commands: Initialize Load Home



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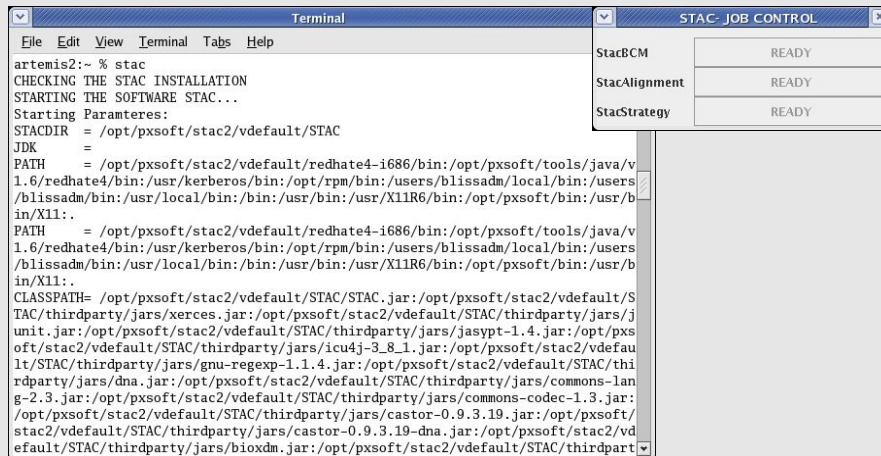
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STAC Startup – via ‘desktop icon’ or shell



The screenshot shows two windows. The 'Terminal' window displays the following output:

```

artemis2:~ % stac
CHECKING THE STAC INSTALLATION
STARTING THE SOFTWARE STAC...
Starting Parameters:
STACDIR = /opt/pxsoft/stac2/vdefault/STAC
JDK =
PATH = /opt/pxsoft/stac2/vdefault/redhate4-i686/bin:/opt/pxsoft/tools/java/v
1.6/redhate4/bin:/usr/kerberos/bin:/opt/rpm/bin:/users/blissadm/local/bin:/users
/blissadm/bin:/usr/local/bin:/bin:/usr/bin:/usr/X11R6/bin:/opt/pxsoft/bin:/usr/b
in/X11:
PATH = /opt/pxsoft/stac2/vdefault/redhate4-i686/bin:/opt/pxsoft/tools/java/v
1.6/redhate4/bin:/usr/kerberos/bin:/opt/rpm/bin:/users/blissadm/local/bin:/users
/blissadm/bin:/usr/local/bin:/bin:/usr/bin:/usr/X11R6/bin:/opt/pxsoft/bin:/usr/b
in/X11:
CLASSPATH= /opt/pxsoft/stac2/vdefault/STAC/STAC.jar:/opt/pxsoft/stac2/vdefault/S
TAC/thirdparty/jars/xerces.jar:/opt/pxsoft/stac2/vdefault/STAC/thirdparty/jars/j
unit.jar:/opt/pxsoft/stac2/vdefault/STAC/thirdparty/jars/jasypt-1.4.jar:/opt/pxs
oft/stac2/vdefault/STAC/thirdparty/jars/icu4j-3.8.1.jar:/opt/pxsoft/stac2/vdefau
lt/STAC/thirdparty/jars/gnu-regexp-1.1.4.jar:/opt/pxsoft/stac2/vdefault/STAC/thi
rdparty/jars/dna.jar:/opt/pxsoft/stac2/vdefault/STAC/thirdparty/jars/commons-lan
g-2.3.jar:/opt/pxsoft/stac2/vdefault/STAC/thirdparty/jars/commons-codec-1.3.jar:/
opt/pxsoft/stac2/vdefault/STAC/thirdparty/jars/castor-0.9.3.19.jar:/opt/pxsoft/
stac2/vdefault/STAC/thirdparty/jars/castor-0.9.3.19-dna.jar:/opt/pxsoft/stac2/vd
efault/STAC/thirdparty/jars/bioxdm.jar:/opt/pxsoft/stac2/vdefault/STAC/thirdpart

```

The 'STAC-JOB CONTROL' window shows the status of three components:

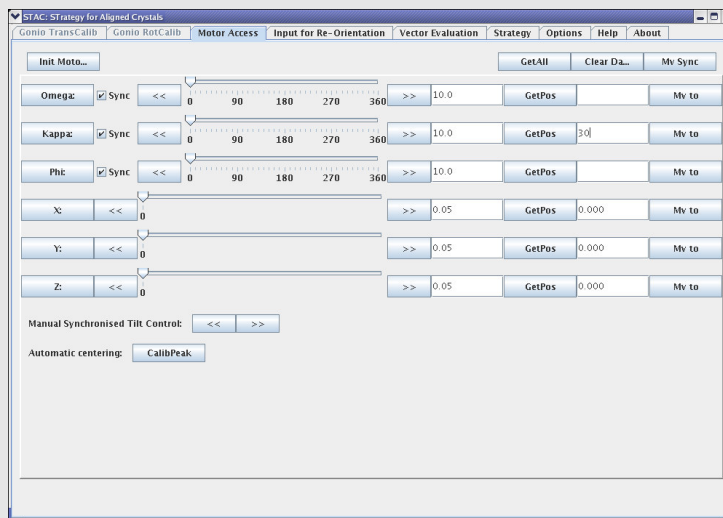
Component	Status
StacBCM	READY
StacAlignment	READY
StacStrategy	READY

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Synchronised movements



The screenshot shows the 'STAC: Strategy for Aligned Crystals' window. The 'Motor Access' tab is selected. The interface includes controls for synchronized movements of Omega, Kappa, Phi, X, Y, and Z. Each axis has a 'Sync' checkbox, a range slider (0 to 360 degrees for Omega, Kappa, and Phi), and a 'GetPos' button. The 'Manual Synchronised Tilt Control' section includes a 'CalibPeak' button.

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Input descriptor – or motor positions

STAC: Strategy for Aligned Crystals

Go to: TransCalib | Go to: RotCalib | Motor Access | **Input for Re-Orientation** | Vector Evaluation | Strategy | Options | Help | About

NEW XTAL Descriptor: pmo/20080524_id14eh4/mx/faq/process/xds_postref-fae_1.1_run2_1/xds_descr Load Rewrite

Current Angles: Get Pos.

Current Transl.: Get Pos.

OM file: ☒ Mosflm ☐ XDS ☐ Denzo

V1	V2	Close	Comment
a*	b*	<input checked="" type="checkbox"/>	Standard alignment
a*	c*	<input checked="" type="checkbox"/>	Standard alignment
b*	a*	<input checked="" type="checkbox"/>	Standard alignment
b*	c*	<input checked="" type="checkbox"/>	Standard alignment
c*	a*	<input checked="" type="checkbox"/>	Standard alignment
c*	b*	<input checked="" type="checkbox"/>	Standard alignment

Reorientations:

Add New Del Selected

Smallest Oscillation

Get a new from file:

Next >

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Input OM file – or “crystal orientation matrix”

STAC: Strategy for Aligned Crystals

Go to: TransCalib | Go to: RotCalib | Motor Access | **Input for Re-Orientation** | Vector Evaluation | Strategy | Options | Help | About

NEW XTAL Descriptor: pmo/20080524_id14eh4/mx/faq/process/xds_postref-fae_1.1_run2_1/xds_descr Load Rewrite

Current Angles: 178.0 0.0 0.0 Get Pos.

Current Transl.: 2.1307 -0.0058 22.6165 Get Pos.

OM file: ☐ Mosflm ☒ XDS ☐ Denzo

V1	V2	Close	Comment
a*	b*	<input checked="" type="checkbox"/>	Standard alignment
a*	c*	<input checked="" type="checkbox"/>	Standard alignment
b*	a*	<input checked="" type="checkbox"/>	Standard alignment
b*	c*	<input checked="" type="checkbox"/>	Standard alignment
c*	a*	<input checked="" type="checkbox"/>	Standard alignment
c*	b*	<input checked="" type="checkbox"/>	Standard alignment

Reorientations:

Add New Del Selected

Smallest Oscillation

Get a new from file:

Next >

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Options - Move close to a standard alignment

STAC: Strategy for Aligned Crystals

Genio TransCalib Genio RotCalib Motor Access **Input for Re-Orient** Vector Evaluation Strategy Options Help About

NEW XTAL Descriptor: pmo/20080524_id14eh4/ms/rae/process/sds.postref-fae_1.1_run2.1/sds.descr Load Rewrite

Current Angles: 178.0 0.0 0.0 Get Pos.

Current Transl.: 2.1307 -0.0058 22.6165

OM file: ☐ Mosflm ☒ XDS ☐ Denzo

Reorientations:

Add New Del Selected

Smallest Oscillation

Get a new from file:

V1	V2	Close	Comment
a*	b*	<input checked="" type="checkbox"/>	Standard alignment
b*	a*	<input type="checkbox"/>	Standard alignment
b*	a*	<input type="checkbox"/>	Standard alignment
c*	a*	<input type="checkbox"/>	Standard alignment
c*	b*	<input type="checkbox"/>	Standard alignment

Next >

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Alignment options – Result with close

STAC: Strategy for Aligned Crystals

Genio TransCalib Genio RotCalib Motor Access **Input for Re-Orient** Vector Evaluation Strategy Options Help About

Alignment Vectors:

Clear Table

ID	V1	V2	Omega	Kappa	Phi	Trans	Rank
5	(55.0;-26.079;-23.343)	(0.0;0.0;1.0)	211.245	147.366	334.721	(-8.2802;14.651	2529.69973962...
6	(55.0;-26.079;-23.343)	(0.0;0.0;1.0)	172.232	219.18	295.63	(-15.6955;6.079	2529.70364988...
7	(55.0;-26.079;-23.343)	(0.0;0.0;1.0)	31.245	147.366	334.721	(-8.2802;14.651	2529.06527719...
8	(55.0;-26.079;-23.343)	(0.0;0.0;1.0)	352.232	219.18	295.63	(-15.6955;6.079	2529.06915811...

Omega Kappa Phi X Y Z

Clear: My Sel. My Edit

Next >

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Other options – Add new, smallest oscillation

STAC: Strategy for Aligned Crystals

Go to: TransCalib | GoniometerCalib | Motor Access | **Input for Re-Orientation** | Vector Evaluation | Strategy | Options | Help | About

NEW XTAL Descriptor: pmo/20080524_id14eh4/mis/ae/process/ids_postref-fae_1_1_run2_1/ids_descr Load Rewrite

Current Angles: 178.0 0.0 0.0 Get Pos.

Current Transl.: 2.1307 -0.0058 22.6165

OM file: ☐ Mosflm ☒ XDS ☐ Denzo

Reorientations:

Add New Del Selected

Smallest Oscillation

Get a new from file:

V1	V2	Close	Comment
a*	b*	<input checked="" type="checkbox"/>	Standard alignment
a*	c*	<input type="checkbox"/>	Standard alignment
b*	a*	<input type="checkbox"/>	Standard alignment
b*	c*	<input type="checkbox"/>	Standard alignment
c*	a*	<input checked="" type="checkbox"/>	Standard alignment
c*	b*	<input type="checkbox"/>	Standard alignment
(0 0 1 0 0)	(1 0 0 0 0)	<input type="checkbox"/>	Optimal align for comp=100.0 ,specific,
(2 0 1 0 0)	(0 0 0 0 1)	<input type="checkbox"/>	Optimal align for comp=99.0 ,specific,
(1 0 1 0 0)	(0 0 0 0 1)	<input type="checkbox"/>	Optimal align for comp=98.0 ,specific,

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Result – Add new or smallest oscillation

STAC: Strategy for Aligned Crystals

Go to: TransCalib | GoniometerCalib | Motor Access | **Input for Re-Orientation** | Vector Evaluation | Strategy | Options | Help | About

Alignment Vectors:

Clear Table

ID	V1	V2	Omega	Kappa	Phi	Trans	Rank
5	(55.0 -26.079 -...)	(0.0 0.0 1.0)	211.245	147.366	334.721	-8.2802,14.651	2529.69979862
6	(55.0 -26.079 -...)	(0.0 0.0 1.0)	172.232	219.18	295.63	-15.6955,6.079	2529.7034988
7	(55.0 -26.079 -...)	(0.0 0.0 1.0)	31.245	147.366	334.721	-8.2802,14.651	2529.06527719
8	(55.0 -26.079 -...)	(0.0 0.0 1.0)	352.232	219.18	295.63	-15.6955,6.079	2529.06915811
9	(0.0 1.0 0.0)	(1.0 0.0 0.0)	227.33	129.719	95.395	-6.2496,12.379	2183.55622539
10	(0.0 1.0 0.0)	(1.0 0.0 0.0)	169.566	236.827	37.511	-13.7632,1.155	2183.55712274
11	(0.0 1.0 0.0)	(1.0 0.0 0.0)	47.33	129.719	95.395	-6.2496,12.379	2183.56270782
12	(0.0 1.0 0.0)	(1.0 0.0 0.0)	349.566	236.827	37.511	-13.7632,1.155	2183.55874419
13	(0.0 10.0 80.0)	(1.0 0.0 0.0)	89.644	152.487	219.61	-5.3224,14.008	2573.09759516
14	(0.0 10.0 80.0)	(1.0 0.0 0.0)	60.14	214.058	186.04	-12.6906,8.325	2573.10353231
15	(0.0 10.0 80.0)	(1.0 0.0 0.0)	273.644	152.487	219.61	-5.3224,14.008	2573.09922357
16	(0.0 10.0 80.0)	(1.0 0.0 0.0)	240.14	214.058	186.04	-12.6906,8.325	2573.10731487

Omega Kappa Phi X Y Z

My Sel

Clear: My Edit

Next >

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Strategies

STAC: Strategy for Aligned Crystals

Genio TransCalib Genio RotCalib Motor Access Input for Re-Orientation Vector Evaluation Strategy Options Help About

Multiple-Sweep Strategies:

ID	OmegaStart	Incr	Time	Images	1st img	Resolution	Kappa	Phi	Completeness	Rank
1	26.0	1.0	1.0	95	1	2.0	147.366	334.721	100.0	95.0
2	138.0	1.0	1.0	89	1	2.0	129.719	95.395	97.899	105.0
3	98.0	1.0	1.0	16	1	2.0	0.0	0.0	99.899	105.0
3	94.0	1.0	1.0	89	1	2.0	152.487	219.61	99.651	97.0
3	89.0	1.0	1.0	8	1	2.0	0.0	0.0	99.899	97.0

Clear Table

Finish

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Help

STAC: Strategy for Aligned Crystals

Genio TransCalib Genio RotCalib Motor Access Input for Re-Orientation Vector Evaluation Strategy Options Help About

Input:

- + put a crystal + center it + get images with arbitrary motor settings + index them
- + optionally:
 - It should describe the OM file, its type (xosfls/xds/denzo), and the datum/translation used
 - + [Load this descriptor], or manually fill the fields below with the required info
 - + optionally: positions can help in getting the Datum/Translation values
- + optionally:
 - The information in the GUI is stored to the given descriptor
 - + Edit the v1/v2 table in the lower right corner:
 - one can edit the fields manually
 - V1: the crystal vector to be aligned parallel to the spindle-axis
 - V2: the crystal vector to lay on the plane of the spindle- and beam-axes
 - Valid crystal vector entries are:
 - a,b,c,a*,b*,c* - special vectors
 - (h,k,l) - reciprocal space vectors
 - [a,b,c] - real space vectors
 - [Add] a new row can be added
 - [DelSel] the selected rows can be removed
 - [GetFromFile]: the orientation of a previous crystal can be attached
 - to do so, one should specify the OM descriptor of the previous indexing
 - If a 0 file is specified, its naming must follow the standard conventions:
 - *.mat xosfls matrix file with a separate save file descriptor (xosfls.inp)
 - *.lp xds indexing in CORRECT.LP with a separate hkl descriptor (INTEGRATE.HKL)
 - *.x denzo indexing
 - optionally select only the lines of interests
- + [Next]: possible datums are calculated for the selected(l) reorientations.
- If no row has been selected, all the entries are processed.

Align:

- + select one or more offered reorientation alignments
- Be careful with the possible collisions!
- + [Move Sel]: The offered reorientation of the uppermost selection can be performed.
- If no selection has been made yet, the arbitrarily editable datum fields are taken into account.
- Note that the appropriate translations are automatically applied.
- + [Next]: appropriate strategies are calculated for the selected entries.

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BCM.dat

```

artemis2:~/STAC/config % more BCM.dat
# GONIO Calibration Settings
OmegaRot -8.15E-4 -3.3E-4 1.0
KappaRot -0.28613 -0.30305 0.909
PhiRot 0.00487 -0.00614 0.99997
KappaTrans 0.17545 -0.2136 -0.01185
PhiTrans 0.21699 -0.0848 -0.00128
KappaTransD -0.29172 -0.2957 0.90965
PhiTransD 0.00434 -0.00599 0.99997
# BCM plugin selection
BCM_Plugin BCMplugin_esrf_complex
# spec version:
SPECVERSION lid144:eh4
# motorDescriptor:
# eg:
# motorName motorName multiplication factor offset dialOffset
# (in STAC) (in spec) (specValue=StacValue*f+offset) (user=dial-dialOffset)
#
X sampx -1.0 -2.44637329701 -5.177111999987716E-5
Y sampy 1.0 -0.15339673024200005 -5.4495909999979775E-5
Z phiy 1.0 21.0822111111 -1.1111099997407337E-5
Omega phi 1.0 0.0 2.343749999990505E-4
Kappa kap1 1.0 0.0 0.0
Phi kap2 1.0 0.0 0.0
#
# remark:
# mulfac assumes that calibration has been done, and the
# motors are aligned to the lab axes, otherwise
# [XYZ] <-> [spec translation] would require a 3d transformation
#
INITSTRING minikappa_init
#
CENTERPREPARE centerprepare 0 0 0
TANGOURL_CENTERNEEDLE tango://deino:20000/id14/mxautocentring/4 0 0 0
#source:
# CACHE - 0
# CACHE_DEVICE - 1
# DEVICE - 2
SOURCE 1 0 0 0
CENTERNEEDLE CenterNeedle 0 0 0
CENTERNEEDLERADIUS R 0 0 0
CENTERSTATUSREQUEST State 0 0 0
CENTERSTATUSOK STANDBY 0 0 0
CENTERSTATUSMOVE RUNNING 0 0 0
artemis2:~/STAC/config %

```

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Joint Structural Biology Group

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BLOMs: D. Flot, D. Nuizzo,
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EMBL Instrumentation Group

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