ESRF	**Experiment title:** **Block Allocation Group (Portugal)**	**Experiment number**: LS-1667
Beamline: BM-14	**Date of experiment**: from: 24-06-00 to: 26-06-00	**Date of report**: 21-08-2000
Shifts: 6	**Local contact(s)**: Dr. Gordon LEONARD	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Pedro MATIAS (ITQB-UNL) *

Francisco ENGUITA (ITQB-UNL) *

David ARAGÃO (ITQB-UNL) *

Report:

Calcium binding protein from *Desulfovibrio gigas* (Co-crystallised with $SmCl_3$):

This protein was isolated from the natural microorganism, and has a M.W. of about 45 kDa. Its N-terminal shows significant homology with the calcium binding 'finger' found in calmodulins. A previous data collection on ID14-1 on a similar crystal gave indications that Sm might have displaced Ca in the binding site, and therefore a MAD experiment was undertaken. However, since the full protein sequence is not known, the total number of possible calcium binding sites (and hence, the total number of possible Sm sites) is unknown at present. Crystals belong to hexagonal crystal system, space group $P6_1$ or $P6_5$ and the crystal used in the experiment had unit cell parameters a=50.36, c=247.60 Å.

Possibly due to the small crystal size (75 μm) an X-ray fluorescence scan failed to reveal a Sm absorption edge either at the L_I or L_{III} edges. Therefore, three suitable wavelengths were chosen from theoretical tables of f' and f" in order to maximise anomalous signal and dispersive differences:

Wavelength (Å)	f'	f"
λ_1, 1.4586	-3.83	11.19
λ_2, 1.5998	-8.23	12.85
λ_3, 0.9918	-0.24	6.07

The diffraction data were recorded on a MAR CCD detector (small diameter) and processed with Denzo, Scalepack and CCP4. The data statistics are summarised below (values in parentheses refer to the highest resolution shell):

Wavelength, Å	Resolution, Å	% R-merge	% R-anom	% complete	I/σ(I)	Redundancy
λ_1, 1.4586	25.0 - 3.05 (3.18 - 3.05)	5.2 (10.3)	3.6 (5.2)	99.9 (100.0)	10.5 (5.1)	5.5 (5.5)
λ_2, 1.5998	25.0 - 3.05 (3.18 - 3.05)	6.5 (12.6)	4.9 (6.5)	99.6 (100.0)	7.2 (4.3)	4.5 (4.4)
λ_3, 0.9918	25.0 - 2.60 (2.71 - 2.60)	3.9 (9.0)	2.2 (3.9)	99.8 (100.0)	14.1 (7.6)	5.5 (5.2)

While the anomalous Patterson maps from the λ_2 data showed features suggesting interatomic Sm vectors, attempts to interpret it with SOLVE, SnB and SHELXL have so far been unsuccessful.