

Experimental report for MX-1565 (ID29 – 25 november 2013/26 november 2013)

The aim of our experiment was twofold: obtain further insight into the structure of giant calix[4]arene tetrasulfonate supramolecular complexes and a screening of their stability and formation domains. We are grateful for the beamtime allocated at ID-29, as the facility offered extremely brilliant optics and fast data gathering (ideal for our weakly diffracting crystals) and a high throughput system, of which we were able to take full advantage. This was the main task to be done, since the experiments are clearly impossible using laboratory X-ray sources. Crystals can however be screened in the laboratory, since it is possible to determine the unit cells and crystal quality. The feasibility was checked during test runs at the ESRF.

To this extent we have prepared 90 samples of various compositions to take full advantage of the experimental facilities at ID29. We managed to scan around 60/90 samples prepared during our 24 hour shift.

Up until now the structure solution of giant calixarene/guanidinium or diaminoguanidinium architectures with unit cell volumes up to 150000 \AA^3 has not yet succeeded. However, diffraction resolution up to 0.9 \AA resolution with good indexation were obtained and we are continuing with the data processing and structure solution. It should be noted that such data quality proved illusive by using other X-ray sources, such as our laboratory setup (2.5 \AA) or the SOLEIL synchrotron ($1.5\text{-}2 \text{ \AA}$).

Furthermore, we observed similar superstructures for aminoguanidinium and diaminoguanidinium. In Table 1 (see below) the cells in green represent good data sets for the giant supramolecular calix[4]arene:guanidinium:amine 1:4:1 architectures.

In 24h of beamtime we managed to obtain 2TB of data (~50 datasets). Currently, we are still processing the data obtained and aligning our IT facilities to match the standards of ID29 ESRF. Several supramolecular architectures with smaller unit cells were routinely solved.

Table 1. Samples prepared for the 24h beamtime. Green cells indicate the formation of a giant supramolecular architectures (cell volumes of approx.. 137000 Å³).

CRY541		A	C[4] B	C
		Gua	GuaNH2	Gua(NH2)2
1	C3NH2	Green		Green
2	C4NH2	Green		
3	C5NH2	Green		
4	C6NH2	Green		
5	C7NH2	Green		
6	1-phenyl-1-amino-ethane rac	Green		
7	1-phenyl-1-amino-ethane L	Green		
8	1-phenyl-1-amino-ethane D	Green		
9	1-amino-2,3-propanediol rac	Green		
10	1-amino-2,3-propanediol S	Green		
11	1-amino-2,3-propanediol R	Green		
12	dopamine			
13	histidine	Green		
14	3-amino-1,2,4-triazole	Green		
15	galactose amine	Green		

CRY542		A	C[4] B	C
		Gua	GuaNH2	Gua(NH2)2
1	mannose amine	Green	Green	Green
2	beta-alanine	Green	Green	Green
3	6-aminohexanoic acid	Green	Green	
4	11-aminoundecanoic acid	Green	Green	
5	benzylamine	Green	Green	Green
6	2-picoline	Green	Green	
7	3-picoline	Green	Green	
8	4-picoline	Green	Green	
9	4-amino-benzylamine	Green	Green	
10	tyramine	Green	Green	
11	4-methoxy-benzylamine	Green	Green	
12	3-amino-1-propanol	Green	Green	
13	cyclobutylamine	Green	Green	
	1,2-trans-	Green	Green	
14	diaminocyclohexane	Green	Green	
15	1,4-diaminocyclohexane	Green	Green	