SERVICES & FACILITIES ANNUAL REPORT - FY April 2012 to March 2013

SERVICE	FUNDING	AGREEMENT	ESTABLISHED as S&F	TERM
FENAC	Block	PR120021	2009	2 years

TYPE OF SERVICE PROVIDED:

The Facility for Environmental Nanoscience Analysis and Characterisation (FENAC) provides a unique service meeting the needs of the 'environmental nanoscience' community. Originally envisaged as a service supporting the (eco)toxicological community investigating the biological impact of manufactured nanomaterials*, FENAC also underpins the wider environmental community looking at manufactured nanoparticle chemistry and transport, along with incidental (combustion, industry etc) and natural (microbial, weathering etc.) nanomaterials and research work into potential applications of nanomaterials in, for example, environmental remediation or alternative energy. FENAC provides access and analysis for relevant samples, helping FENAC users through the whole process from experimental design to data analysis, in a fully collaborative manner. For doctoral and postdoctoral researchers carrying out measurement and data analysis, the discussions during training have provided a sound basis for future work. In some years, FENAC has also trained researchers more formally through 2 day summer schools, also leveraging NERC Knowledge Exchange programmes and University of Birmingham support.

Professor Jamie Lead and Professor Eugenia Valsami-Jones are co-directors of FENAC. Dr. Bjorn Stolpe was the facility manager throughout most of the reporting period, with Dr. Christine Elgy taking over the role from January 2013, and Dr Gillian Kingston (5%) provides additional technical support within FENAC. The facility offers a unique combination of experimental, analytical and metrological methodologies and the expertise to deploy such methods appropriately; there is competitive access to FENAC, with submissions due every 6 months.

*Nanomaterials, defined as having at least one dimension between 1 and 100 nm, are of three types: manufactured (deliberately produced), incidental (accidentally produced) and natural (produced by natural sources). FENAC offers a unique, proven ability to characterise and interpret the physico-chemical properties of nanoparticles from all sources, including complex environmental matrices (e.g. organisms), for properties including size, aggregation properties, surface behaviour, dissolution and morphology. Using a multi-method approach, FENAC incorporates a number of methods grouped as:

- microscopy (atomic force microscopy (AFM), confocal laser scanning microscopy (CLSM) and electron microscopy, including scanning, environmental scanning, scanning tunnelling and transmission electron microscopy (SEM, ESEM, STEM and TEM)),
- spectroscopy (electron energy loss spectroscopy (EELS), x-ray energy dispersive spectroscopy (X-EDS), x-ray photoelectron spectroscopy (XPS), fluorescence correlation spectroscopy (FCS), and inductively-coupled plasma – mass spectrometry (ICP-MS)),
- separation (including field–flow fractionation (FFF), ultrafiltration (UF), analytical ultracentrifugation, (AUC), disc ultracentrifugation and dialysis),
- other (including dynamic light scattering (DLS), nanoparticle tracking analysis (NTA), differential centrifugal sedimentation (DCS), x-ray diffraction for crystal structure and surface area measurements by BET).

ANNUAL TARGETS AND PROGRESS TOWARDS THEM

FENAC has made significant progress on all approved projects. Of the 10 projects active during the 2012-2013 year, 6 are completed, and 4 on-going. There are a further 3 at the stage of preliminary discussions. The new facility manager is in place, and Dr Bjorn Stolpe has continued to provide support during the transition period. Method development was carried out on dialysis for separating dissolved ions from nanoparticles, and further method development continues to be undertaken in other research projects led by the FENAC Directors, ensuring the continuation of FENAC's leading role internationally. FENAC has been widely marketed and availability disseminated at conferences, workshops and other venues. Demand continues to be healthy, with increased numbers of new applicants from a wider pool of institutions. Ten research papers were published in 2012, and eight conference papers presented.

SCORES AT LAST REVIEW (each out of 5)			Date of Last Review:	
Need	**Uniqueness**	**Quality of Service**	**Quality of Science & Training**	**Average**
5	4.5	4.5	5	4.75

CAPACITY of HOST ENTITY FUNDED by S&F	Staff & Status	Next Review (March)	Contract Ends (31 March)
20 %	Professor Jamie Lead and Professor Eugenia Valsami-Jones (3 hrs/wk combined); Dr. Bjorn Stolpe (62.5% FTE); Dr. Christine Elgy (20% FTE); Dr. Kenton Arkill (10% FTE); Ms. Fatima Nasser (16.5% FTE); Dr Gillian Kingston (5% FTE)	2015	2015

FINANCIAL DETAILS: CURRENT FY

Allocation £k	Unit Cost £k: Time on project per day	Unit Cost £k: Support and consumables per day	Unit Cost £k: Instrumental analysis per day	Capital Expend £k	Income £k	Full Cash Cost £k
125.02	0.45	0.25	0.32	25.00		**£133.80**

FINANCIAL COMMITMENT (by year until end of current agreement) £k

2012-13	2013-14	2014-2015	2015-2016	2016-2017
125.02	**128,605**	**128,605**		

STEERING COMMITTEE	Independent Members	Meetings per annum	Other S&F Overseen
	8	2	None

APPLICATIONS: DISTRIBUTION OF GRADES (current FY — 2012/13)

	10	9	8	7	6	5	4	3	2	1	0	R*	Pilot
NERC Grant projects*				1									
Other academic				2		2							1
Students			1	2									1
TOTAL 10			1	5		2							2

PROJECTS COMPLETED (current FY – 2012/13)

	10 (α5)	9	8 (α4)	7	6 (α3)	5 (α2)	4	3 (α1)	2	1 (β)	0 (Reject)	Pilot
NERC Grant projects				4	1							
Other				2								
Students			1	2								1

Project Funding Type (current FY – 2012/13) (select one category for each project)

Grand Total	Infrastructure					PAYG				
	Supplement to NERC Grant *	PhD Students		NERC Centre	Other	NERC Grant	PhD Students		NERC Centre	Other
		NERC	Other				NERC	Other		
10		3	4		3					

Project Funding Type (per annum average previous 3 financial years - 2009/2010, 2010/2011 & 2011/2012)

Grand Total	Infrastructure					PAYG				
	Supplement to NERC Grant *	PhD Students		NERC Centre	Other	NERC Grant	PhD Students		NERC Centre	Other
		NERC	Other				NERC	Other		
8		1.33	4	0.33	2.33					

User type (current FY – 2012/13) (include each person named on application form)

Academic	NERC Centre	NERC Fellows	PhD Students	Commercial
18		1	10	

User type (per annum average previous 3 financial years - 2009/2010, 2010/2011 & 2011/2012)

Academic	NERC Centre	NERC Fellows	PhD Students	Commercial
8	0.33		9.33	

OUTPUT & PERFORMANCE MEASURES (current year)

Publications (by science area & type) (calendar year 2012)

SBA	ES	MS	AS	TFS	EO	Polar	Grand Total	Refereed	Non-Ref/ Conf Proc	PhD Theses
		5		13			18	10	8	

Distribution of Projects (by science areas) (FY 2012/13)

Grand Total	SBA	ES	MS	AS	TFS	EO	Polar
10			4	1	5		

OUTPUT & PERFORMANCE MEASURES (per annum average previous 3 years)

Publications (by science area & type) (Calendar years 2009, 2010 &2011)

SBA	ES	MS	AS	TFS	EO	Polar	Grand Total	Refereed	Non-Ref/ Conf Proc	PhD Theses
		2.67		6			8.67	4	4.3	0.3

Distribution of Projects (by science areas) (FY 2009/2010 2010/2011& 2011/2012)

Grand Total	SBA	ES	MS	AS	TFS	EO	Polar
8		0.66	3.33	1	3		

Distribution of Projects by NERC strategic priority (current FY 2012/13)

Grand Total	Climate System	Biodiversity	Earth System Science	Sustainable Use of Natural Resources	Natural Hazards	Environment, Pollution & Human Health	Technologies
10			1			8	1

**Either Responsive Mode or Directed Programme grants*

NOTE: All metrics should be presented as whole or part of whole number NOT as a %

OVERVIEW & ACTIVITIES IN FINANCIAL YEAR (2012/13):

General Given the scale of the nanotechnology industry and the importance that NERC, other research councils, the EU and end users attach to the environmental and human health hazards and risks of manufactured nanomaterials, FENAC's operation has proved to be of great interest to the relevant NERC research community and to a range of governmental and industrial bodies, both globally and nationally. There is also considerable interest in FENAC from researchers investigating incidental and natural nanoparticles, where a detailed understanding of processes in the nanoscale is also essential. FENAC pioneered the concept that in order to improve the quality of nanotoxicology research, it is essential to underpin studies with the highest quality detailed characterisation, including under relevant exposure conditions. This concept has also been adopted at European level by FPVII, and a Europe-wide infrastructure facility now exists (Quality Nano, where Birmingham are partners), equivalent to FENAC although the selection criteria and scope are substantially different. FENAC has produced a steady stream of high quality publications (24 to date, with 329 citations as of 20th May 2013) and, through the activities of its Directors, has raised the profile of UK nanoscience research globally, for instance coordinating a successful major EU FP7 bid (NanoMILE, worth 10M Euro). FENAC has also been involved in discussions with organisations such as OECD and standards organisations (ISO, BSI) and one of the Directors (JRL) is technical author on a BSI PAS document published in 2012. FENAC has also negotiated formal collaboration and is continually developing links with the STFC neutron (ISIS) and synchrotron (Diamond Light Source) Facilities: a successful current project using Diamond has been developed from a FENAC project. Highlights of selected projects are given below. There has been some initial commercial interest in the testing capability within FENAC for characterisation of manufactured nanoparticle samples. This demand is expected to increase as the EU's definition for nanomaterials for regulatory purposes means that companies will need to be able to demonstrate whether their product contains nanomaterials as defined on the basis of size and surface area. There is also significant scope here for method development, as the definition itself notes the lack of available methodologies for implementation. FENAC will play an important role when the definition is reviewed again in 2014.

Methods, Training and Staff Development.

In the last financial year, NERC provided support for the acquisition of an autotitrator complementing the DLS instrumentation, the value of which was £10,000. A similar titrator will be developed via the EU FP7 NanoMILE project, for the NanoSight NTA platform to enable tracking of nanoparticle agglomeration / dissolution as a function of pH or ionic strength; this will be subsequently available to FENAC users. More recently, a cryo-electron microscopy capability has been added and the use of cryo SEM for imaging of plant materials has been discussed for a pending project application. Method development has been carried out to study silver nanoparticle dissolution, in collaboration with scientists at the Natural History Museum. Dialysis with ultrafiltration, ultracentrifugation and centrifugation-ultrafiltration were compared and a publication is in preparation from this work. Additional method development has been carried out by researchers in both Lead's and Valsami-Jones's groups, which has continued to inform FENAC practices, maintaining FENAC as an internationally leading centre. Research workers on six separate projects, including four Ph.D. students, have carried out work at FENAC for training purposes in the last year, spending typically between one and three weeks at the facility. The FENAC manager has been given specific instrument training and been involved in training others. User surveys are routinely distributed and indicate high satisfaction with the FENAC facility but will be used critically to ensure this level is maintained.

SCIENCE HIGHLIGHTS

A project in collaboration with the University of Essex has been investigating the toxicity of methoxy-polyethylene glycol capped and non-capped silver nanoparticles to bacteria to understand the role of capping agents in nanotoxicity. In previous years the nanoparticles were characterized, and it was found that the capped nanoparticles are smaller (Fig. 1a-b), have greater specific surface area, are more monodisperse, and have a lower tendency to aggregate in natural estuarine water, compared with uncapped ones.

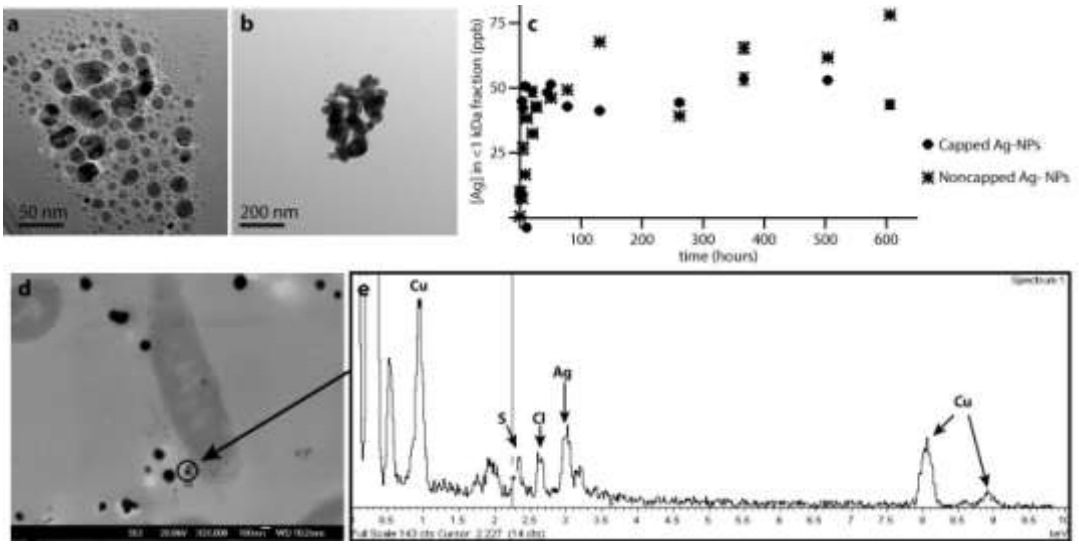

Fig. 1: TEM-images of methoxy-polyethylene glycol -capped (a) and non-capped (b) silver nanoparticles. Variations in 'soluble' (<1 kDa) silver with time after the dilution (to 5 ppm) of silver nanoparticle suspensions determined by dialysis (c). STEM-image of an *E. coli* bacterium cell exposed to capped silver nanoparticles (d) with associated EDX-spectrum (e).

The nanoparticles have been found to be considerably less efficiet in inhibiting *Escherichia coli* cell growth compared with ionic Ag^+, but high concentrations of the capped nanoparticles could also inhibit the cell growth. In 2012-2013, the production of soluble (<1 kDa) silver as a result of nanoparticle dissolution has been investigated by FENAC using dialysis. The method functions well in the presence of a soluble silver-complexing ligand such as Cl^- (in estuarine water) or organic ligands (in natural freshwater), while adsorption of soluble Ag^+ to the dialysis membrane made it difficult to study nanoparticle dissolution in Milli-Q water.
The solubility was similar for both nanoparticle types (Fig. 1c), but ten times higher in estuarine water than in natural freshwater. The attachment of silver nanoparticles to the surfaces of *Escherichia coli* cells has also been verified using scanning transmission electron microscopy (STEM) with energy-dispersive X-ray spectroscopy (EDX) detection (Fig. 1d-e). The study has resulted in a manuscript that has been submitted to Environmental Science & Technology.

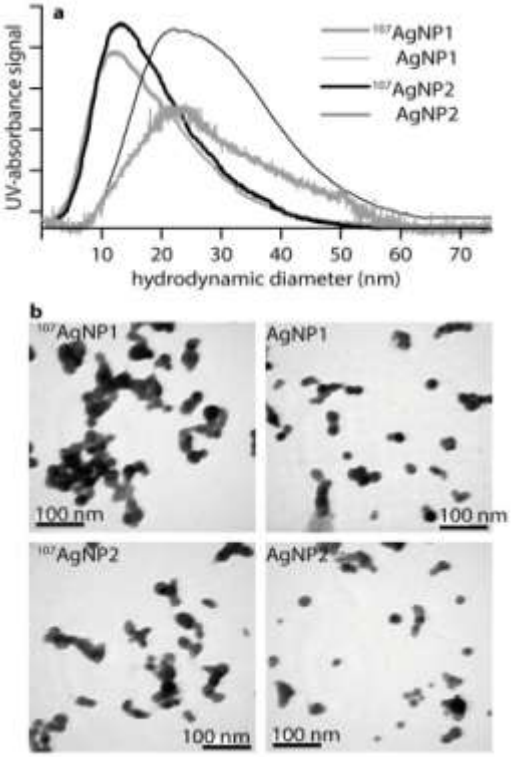

Fig. 2: ^{107}Ag-labelled and non-labelled citrate-capped silver nanoparticles with two different sizes (NP1 and NP2). Size (hydrodynamic diameter) distributions determined by FFF (a) and TEM- images (b).

Collaboration with another research group within the School of Geography Earth and Environmental Sciences at the University of Birmingham has continued to investigate the potential use of hydroxyapatite for radionuclide decontamination in the nuclear industry. Previous work has resulted in a published article in Environmental Science & Technology and an additional manuscript in preparation. It has been demonstrated that bacterial hydroxyapatite has a much higher metal-sorption capacity (up to 15 times) than synthetic hydroxyapatite. The specific structure of hydroxyapatite biominerals (such as amorphous content, large specific surface area and smaller crystallite size) is known to underlie these advantages. In 2012-2013, the mechanism by which the bacteria *Serratia sp.* induce biomineral hydroxyapatite formation was investigated, with the aim of developing new methods for manufacture of high-efficiency materials for radionuclide remediation.

The production of hydroxyapatite nanoparticles attached to organic material in the near vicinity of *Serratia sp.* cells has been shown using AFM (Fig. 3a-d). The hypothesis is that the growth and structure of the biomineral is controlled by the Serratia cells and their thick surrounding layer of extracellular polymeric substance (EPS). An application was submitted to the ISIS facility to test the hypothesis using small angle neutron scattering (SANS). The application was approved, and the experiments will be carried out in June 2013. This research has received interest from the ISIS media department (http://www.isis.stfc.ac.uk/science/energy/can-biological-minerals-made-by-bacteria-clean-up-contaminated-fukushima-soils14028.html).

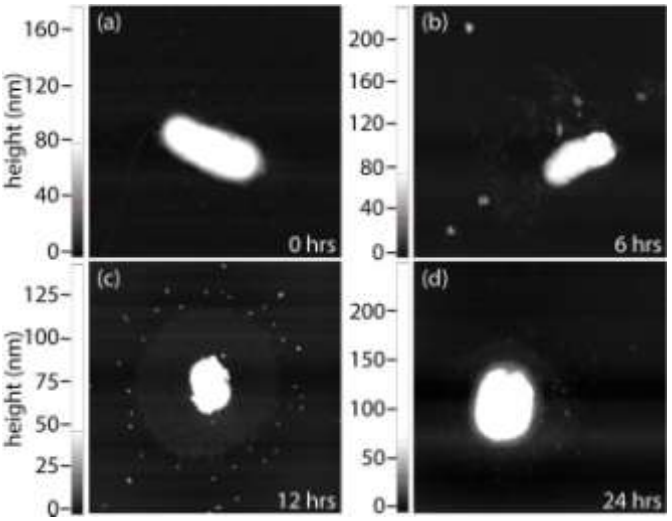

Fig. 3: AFM-images of bacteria cells (*Serratia sp.*) with associated organic material and hydroxyapatite before (a) and 6 (b), 12 (c) and 24 (d) hours after the addition of the calcium and glycerol-2-phosphate substrates.

FUTURE DEVELOPMENTS AND STRATEGIC VIEW

FENAC will continue with the progress made to date in supporting the 'nano' community by broadening access to the potential user community. Applications to FENAC are increasing as are collaborations on new national and international initiatives based on its remit. FENAC will continue to improve and widen access to essential training in regular one-to-one laboratory support and in workshops, either on site or at relevant conferences, which will act as valuable outreach mechanisms. It is proposed to run a hands-on training session jointly with the EU FP7 QualityNano research infrastructure, where FENAC will act as the host and provide access to key equipment. Instrument suppliers such as NanoSight and CPS will be invited to support the training by providing "clinics" for researchers to tackle their own complex samples and video tutorials will be recorded as part of the training event, funded via QualityNano, but using the skills within FENAC.

The facilities for analysis and characterisation will be extended in the coming year with the purchase a new Inductively Coupled Plasma – Mass Spectrometer with single particle analysis capability, allowing development of methods to determine the number and size of nanoparticles in the presence of the dissolved ions. FENAC will also gain access to an Inductively Coupled Plasma - Optical Emission Spectrometer at the University of Birmingham, and both of these instruments will enhance the FENAC capability in trace metal detection and quantification.

A dispensing unit will be installed to allow the automation of the DLS measurements of size and zeta potential as a function of pH, conductivity or additive concentration. This will facilitate the characterisation of nanoparticle properties in a range of media.