SERVICES & FACILITIES ANNUAL REPORT - FY April 2015 to March 2016

SERVICE	FUNDING	AGREEMENT	ESTABLISHED as S&F	TERM
FENAC	Block	PR120021	2009	N/A

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.

Professor Eugenia Valsami-Jones was director of FENAC during 2015-2016, Dr. Iseult Lynch was Deputy Facility Director, Dr. Christine Elgy was facility manager and Dr. Maria Thompson provided technical support (5% FTE) 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.

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 techniques, 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.

*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).

ANNUAL TARGETS AND PROGRESS TOWARDS THEM

FENAC has provided support for 13 projects during the 2015-2016 year. Of these, six are completed and seven are ongoing. The measurement of nanoparticles from the air and rainwater, in the sea, their fate in in lakes and rivers, and the composition and interaction of nanoparticles in soils with organisms through the pore water have all been covered in FENAC projects this year. Nanoparticles in sewage and effluent from treatment plants have also been examined. In addition, there has been project work on mine wastes and method development for characterising surface hydrophobicity and hydrophilicity of individual nanoparticles.

Presentations have been made at conferences and meetings, to promote the work of FENAC. Demand for work at FENAC has remained strong throughout 2015, with 13 applications to FENAC and 7 projects funded.

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 Eugenia Valsami-Jones (3hrs/week) Dr. Christine Elgy (100% FTE) Dr. Maria Thompson (5% FTE)	2016	2017

FINANCIAL DETAILS: CURRENT FY						
Total Resource Allocation	Unit Cost £k			Capital Expend £k	Income £k	Full Cash Cost
£ 127.00k	£0.533k per day	£0.04k per day electron microscopy	£0.06k per 10 icp-ms samples			£135.89k

FINANCIAL COMMITMENT (by year until end of current agreement) £k				
2015-16 127	2016-17 127			

STEERING COMMITTEE	Independent Members	Meetings per annum	Other S&F Overseen
FENAC	7	2	0

APPLICATIONS: DISTRIBUTION OF GRADES (current FY — 2015/16)													
	10	**9**	**8**	**7**	**6**	**5**	**4**	**3**	**2**	**1**	**0**	**R***	**Pilot**
NERC Grant projects*			1										
Other academic			1				1				1	2	
Students			2	2				1				1	1
TOTAL 13			4	2			1	1			1	3	1

APPLICATIONS: DISTRIBUTION OF GRADES (per annum average previous 3 financial years —2012/2013, 2013/2014 & 2014/2015)													
	10	**9**	**8**	**7**	**6**	**5**	**4**	**3**	**2**	**1**	**0**	**R***	**Pilot**
NERC Grant projects*				0.67									
Other academic			1.33	1		0.67	0.67					0.33	0.67
Students			2	1		1.33	0.67		0.33		0.67	0.33	1.33
TOTAL 13			3.33	2.67		2	1.33		0.33		0.67	0.67	2

PROJECTS COMPLETED 2015-16 (current FY – 2015/16)												
	10 (α5)	**9**	**8 (α4)**	**7**	**6 (α3)**	**5 (α2)**	**4**	**3 (α1)**	**2**	**1 (β)**	**0 (Reject)**	**Pilot**
NERC Grant projects*				**2**								
Other Academic			**1**									**1**
Students												**2**

For projects worked on

Project Funding Type (current FY – 2015/16) (select one category for each project)										
Grand Total	**Infrastructure**					**PAYG**				
	Supplement to NERC Grant *	**PhD Students NERC**	**PhD Students Other**	**NERC Centre**	**Other**	**NERC Grant***	**PhD Students NERC**	**PhD Students Other**	**NERC Centre**	**Other**
13	1	2	4	1	5					

Project Funding Type (per annum average previous 3 financial years - 2012/2013, 2013/2014 & 2014/2015)										
Grand Total	**Infrastructure**					**PAYG**				
	Supplement to NERC Grant *	**PhD Students NERC**	**PhD Students Other**	**NERC Centre**	**Other**	**NERC Grant***	**PhD Student NERC**	**PhD Student Other**	**NERC Centre**	**Other**
12.67	**1.33**	**1.67**	**4.67**	**1.33**	**3.67**					

User type (current FY – 2015/16) (include each person named on application form)				
Academic	**NERC Centre**	**NERC Fellows**	**PhD Students**	**Commercial**
27	**3**	**1**	**6**	**2**

User type (per annum average previous 3 financial years - 2012/2013, 2013/2014 & 2014/2015)				
Academic	**NERC Centre**	**NERC Fellows**	**PhD Students**	**Commercial**
21.67	**2.67**	**1.67**	**8.00**	

OUTPUT & PERFORMANCE MEASURES (current year)

Publications (by science area & type) (calendar year 2015)										
SBA	**ES**	**MS**	**AS**	**TFS**	**EO**	**Polar**	**Grand Total**	**Refereed**	**Non-Ref/ Conf Proc**	**PhD Theses**
	7	**4.5**	**0.5**	**16**			**28**	**12**	**14**	**2**

Distribution of Projects (by science areas) (FY 2015/16)							
Grand Total	**SBA**	**ES**	**MS**	**AS**	**TFS**	**EO**	**Polar**
13	**0**	**2.33**	**1.5**	**1.33**	**7.83**	**0**	**0**

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

Publications (by science area & type) (Calendar years 2012, 2013 & 2014)										
SBA	**ES**	**MS**	**AS**	**TFS**	**EO**	**Polar**	**Grand Total**	**Refereed**	**Non-Ref/ Conf Proc**	**PhD Theses**
0	**1.67**	**3.17**	**0.33**	**9.5**			**14.76**	**8**	**6.33**	**0.33**

Distribution of Projects (by science areas) (FY 2012/2013, 2013/2014 & 2014/2015)							
Grand Total	**SBA**	**ES**	**MS**	**AS**	**TFS**	**EO**	**Polar**
12.67		**2.22**	**3.56**	**0.55**	**6.33**		

Distribution of Projects by NERC strategic priority (current FY 2015/16)							
Grand Total	**Climate System**	**Biodiversity**	**Earth System Science**	**Sustainable Use of Natural Resources**	**Natural Hazards**	**Environment, Pollution & Human Health**	**Technologies**
13	**0**	**0**	**2.5**	**0**	**0.5**	**10**	**0**

**Either Discovery Science (Responsive Mode) or Strategic Science (Directed Programme) grants*

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

OVERVIEW & ACTIVITIES IN FINANCIAL YEAR (2015/16):

Projects: FENAC projects in 2015-2016 have encompassed a broad spectrum of environments, including the presence and composition of nanoparticles in the sea and in rainwater, the fate of nanoparticles in static and flowing water systems, and monitoring iron nanoparticles in the air where method development on novel procedures has commenced. Nanomaterials in soils, their mineralogy, and interactions with organisms and within the soil compartment have been examined; work has started on the mobility of uranium from mine wastes in water which could have important implications to nuclear decommissioning. As a means of characterising nanomaterials in a more fundamental way, and develop a better understanding of their interactions with biological and ecological systems, a method of measuring hydrophilicity and hydrophobicity of nanoparticles has been evaluated.
Equipment: A state-of-the-art Quartz Crystal Microbalance has been installed and commissioned in the laboratory. This will measure interactions between nanoparticles and layers of molecules, including proteins, allowing, for example, the strength of interactions with different proteins to be assessed for elucidation of protein corona formation.
Training of FENAC staff: The FENAC manager has received additional, hands-on training in Field-Flow Fractionation at the site of the instrument manufacturer (Germany), to enable better support of FENAC projects using this instrument (3 active projects this year). The FENAC manager also attended a user-group meeting for Atomic Force Microscopy, where she learned about methods to customise the settings to improve control of the tip speed, providing superior quality imaging when larger particles are present, and also showed the enhanced capability available with the latest software and camera updates.
FENAC has also been promised additional support to develop improved imaging in liquid mode.
Radiation Protection training has been undertaken in preparation for laboratory work on characterisation of uranium colloids which will commence in the coming year.
A broader understanding of the applications for Single Particle ICP-MS was gained through the Perkin Elmer user group.

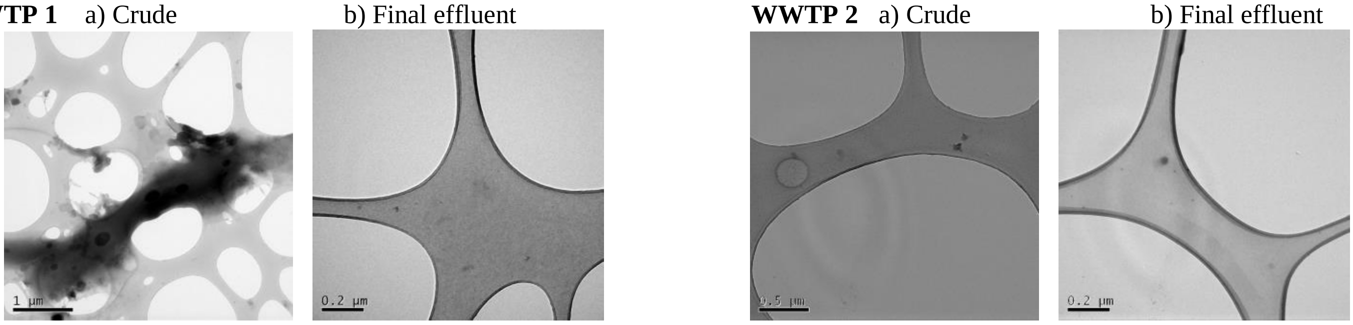
Figure 1. Professor Valsami-Jones: Royal Society of Chemistry Distinguished Guest Lecturer

Promotion of FENAC: Congratulations are due to Professor Eva Valsami-Jones, the FENAC director, who was presented with a medal by the Royal Society of Chemistry as the 2015 Distinguished Guest Lecturer for the Environmental Chemistry Group. She was also the Mineralogical Society's Distinguished Lecturer for 2015. FENAC was widely promoted during the Distinguished Guest Lectures, which generated new FENAC applications from Earth Scientists.
A presentation promoting FENAC's work was given at a Royal Society of Chemistry, Analytical Division meeting by the FENAC manager.
The FENAC website was updated to make it more compatible with mobile phone applications. A video was added to provide a readily accessible introduction to FENAC characterisation techniques (https://youtu.be/eaUn1A01HRE). This has received favourable comments from users.
Workshop: A workshop was held by FENAC, primarily for academics, to provide an introduction to the facility by showcasing some of the FENAC projects, to provide guidance to applicants about how to structure the information in the applications, in support of both new and previous users and finally to gauge future needs of the FENAC community. This was attended by about 40 participants, with approximately 50% previous users, and 50% who had not been involved with FENAC before. Feedback forms were received from 43% of the people attending, and all of these were positive about both the workshop and the support that FENAC provides.

SCIENCE HIGHLIGHTS.

Behaviour and fate of nanoparticles in wastewater treatment works. J. Bridgeman and E. Carstea.
The first study of quantification and characterisation of nanoparticles through the main wastewater treatment processes, from crude to final effluent, with potential for commercial benefits.
Work to assess the removal efficiency of nanoparticles along the wastewater treatment process has been undertaken at six different waste water treatment plants (WWTP). This has provided a broad assessment of the influence of the various stages of the process on nanoparticle removal. The presence of nanoparticles was shown by dynamic light scattering measurements, and to assess concentration and size of nanoparticles, deposited nanoparticles were imaged by Atomic Force Microscopy and Transmission Electron Microscopy. Energy Dispersive Spectroscopy also allowed the composition of inorganic nanoparticles to be investigated. Figure 2 demonstrates the differences in the particulate level of the crude effluent and the similarity in the final effluent from two WWTPs.

Figure 2. Transmission Electron Microscopy images from water collected at different waste water treatment plants (WWTP)
WWTP 1 a) Crude b) Final effluent **WWTP 2** a) Crude b) Final effluent

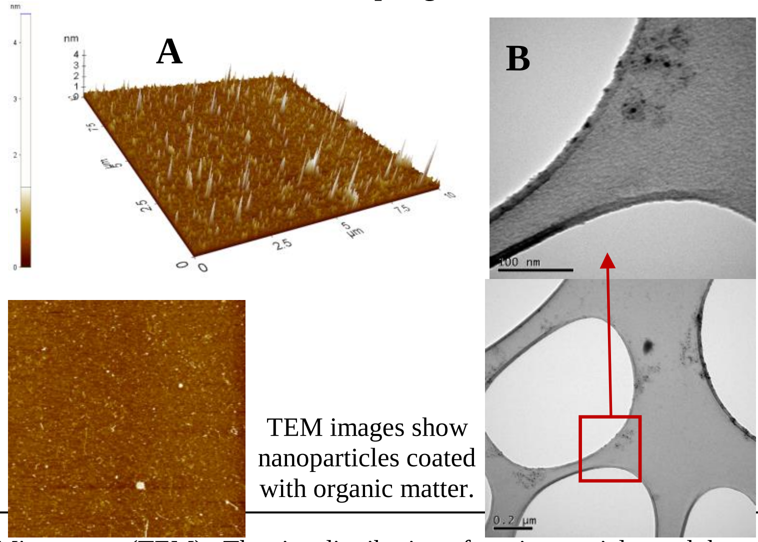


The knowledge arising from this research will be beneficial for the environment by identifying areas where the treatment could be adjusted, to reduce the ecological impact on the effluent receiving water bodies. It will also benefit the waste-water treatment industry as it will facilitate minimisation of energy usage during wastewater treatment while maintaining the quality of the effluent.

Characterisation of marine colloids and nano particles associated with metals. M. Gledhill and D. Rusiecka.

This work has produced the first direct visual investigation into the formation, water column size distribution and seasonal variation of NP in the Celtic Sea.

Figure 2. AFM (A) and TEM (B) images from surface waters in Spring



The growth of phytoplankton, and therefore the potential for the ocean to absorb carbon dioxide, in 30% of the open ocean is limited by low iron concentrations. Shelf sediments represent an important source of dissolved iron and other biologically essential dissolved trace metals to open ocean waters. The supply of iron from shelf sources involves long range transport of iron in seawater, and the physico – chemical form of iron within the water column is critical for its stabilisation. In a project to investigate the transport of iron from the shelf sediment, colloids and particles have been characterised at FENAC to understand their chemical behaviour and assess the importance of different iron fractions in offshore iron transport.

Water samples have been collected from a NERC research vessel from the surface waters, an intermediate depth and deep waters in December 2014, April 2015 and July 2015. The water samples were treated to remove the solution while concentrating the nanoparticles. The presence of nanoparticles in natural seawater was confirmed by Atomic Force Microscopy (AFM) and Transmission Electron Microscopy (TEM). The size distribution of marine particles and the abundance of the particles varied with the seasons and the depth of the water. The presence of dissolved organic matter was seen to increase the stabilisation of the iron ions in seawater and reduce precipitation. The nanoparticle size distribution in seawater may also be influenced by the concentration and nature of organic matter present. *Dagmara Rusiecka et al. Direct visual observations of nanoparticles in The Celtic Sea, Ocean Sciences, 22-26th of February 2016, New Orleans, USA*

Probing surface hydrophobicity of "soft" nanoparticles by AFM. M-C Jones.

The method under development in this work has applicability for interactions of natural, manufactured and anthropogenic nanoparticles with humans and environmental organisms, and has the potential to elucidate toxicity mechanisms.

The use of nanoparticles as carriers for drug delivery to targeted regions of the body is an area of current development. One of the factors determining interactions between nanoparticles and bio- and ecological systems is surface hydrophobicity although this is not routinely explored as part of standard nanoparticle characterisation. A pilot study at FENAC has investigated the feasibility of using atomic force microscopy (AFM) to measure adhesion forces between chemically modified AFM tips and nanoparticles and establish if this can be used as a measure of hydrophobicity. Lipid nanoparticles and partially hydrolysed polyvinyl alcohol (PVA) have been identified as suitable nanocarriers for biomedical applications. Two hydrolysis grades of PVA and two sizes of lipid nanoparticles have been assessed by AFM, along with polystyrene, which provided a hydrophobic control. A minimum of 1,000 adhesion measurements were performed in air, and in liquid for each nanoparticle/fluid combination, with unmodified and with coated AFM tips.

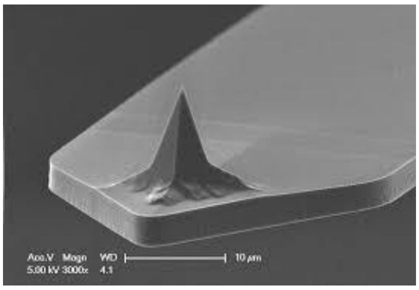


Figure 3. Image of silicon AFM tip
http://www.brukerafmprobes.com/t-new-rtesp-silicon-AFM-probes.aspx

From the pilot data, atomic force microscopy shows potential for the evaluation of nanoparticle surface hydrophobicity. The ability to study behaviour at single-nanoparticle level could prove an invaluable tool in understanding nanoparticle-cell interaction and the true impact of surface properties.

Selected publications

Valsami-Jones, E., Lynch, I. (2015) How safe are nanomaterials? Science 350 (6259), 388-389 Impact factor 33.6

McKenzie, Z., Kendall, M., Mackay, R-M., et al. (2015) Surfactant protein A (SP-A) inhibits agglomeration and macrophage uptake of toxic amine modified nanoparticles. Nanotoxicology (2015) 9(8) 952-62. Impact factor 6.4

Khan, F. R., et al. (2015) Accumulation dynamics and acute toxicity of silver nanoparticles to Daphnia magna and Lumbriculus variegatus: Implications for metal modelling approaches. Environ Sci Technol., 49(7), 4389-97. Impact factor 5.3

Echavarri-Bravo, V., et al. (2015). Shifts in the metabolic function of a benthic estuarine microbial community following a single pulse of silver nanoparticle exposure. Environmental Pollution 201, 91-99. Impact factor 4.1

FUTURE DEVELOPMENTS/STRATEGIC FORWARD LOOK

Instrumentation: An ICP centre within the FENAC laboratory has been set up and the final instrument will be commissioned in the coming year. It is intended that the developing relationships with several instrument manufacturers will be strengthened, to support work at FENAC; to that end, joint workshops are being planned (Postnova and Perkin Elmer), during which FENAC will be showcasing the capabilities of instrumentation to industry. This may generate new users of the facility from industry and will establish stronger links and collaborations with the instrument manufacturers.

Method development: is planned to develop a new methodology for imaging nanoparticles in tissue, currently used in medical work, combining optical reflectance imaging with transmission electron microscopy (TEM).