

SERVICES & FACILITIES ANNUAL REPORT - FY April 2014 to March 2015

SERVICE IMF	FUNDING Contract	AGREEMENT R8/H10/51	ESTABLISHED as S&F 1987	TERM 5 yrs
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TYPE OF SERVICE PROVIDED:

The **Ion Microprobe Facility (IMF)** (see <http://www.geos.ed.ac.uk/facilities/ionprobe/>) is the only UK facility enabling **SIMS (secondary ion mass spectrometry)** analysis of a wide range of natural and synthetic materials. It allows in-situ, high spatial resolution (5–20 µm) analysis of elements and isotopes across the whole Periodic Table, and is ideal for studies of fine-grained and zoned Environmental and Earth Sciences materials. The IMF has a very strong international reputation and has pioneered the development and application of SIMS isotope and trace element analysis in the Earth Sciences.

Two ion microprobes (Cameca ims-1270 and Cameca ims-4f) provide the analysis of a wide range of **stable isotope ratios** (H, Li, B, C, N, O, S, Si), **trace elements** (e.g. rare-earth elements, large ion lithophile elements, high field strength elements) and **light elements** (H, Li, Be, B, C, N). The high transmission of the ims-1270 permits U-Th-Pb isotopic **geochronology** of zircon and other U- and Th-bearing minerals. In general most heavy (>C) stable isotope work is done on the ims-1270 since its analytical precisions are some four to five times higher and analysis times (with multi-collector) significantly shorter than on the ims-4f. The ims-4f is mainly used for multi-element and light element isotope analysis. A number of projects have combined access to both instruments. The in-house developed multi-sample airlocks for both the ims-4f and ims-1270 offer significant advantages for the analysis of volatile elements and isotope analysis.

The IMF is located in the Grant Institute of Earth Science, School of GeoSciences, University of Edinburgh, where the ion microprobe (SIMS) instruments form the centre-piece of a unique and complementary suite of micro-beam instruments. The IMF also provides access to the Cameca SX100 electron microprobe and analytical SEM facilities (a newly installed Zeiss Sigma HD VP SEM with FEG electron source and including detectors for Energy Dispersive Analysis, Secondary Electron Imaging, Cathodoluminescence and Electron Backscatter Diffraction); these may be used for full characterisation of samples on site. A range of state-of-the-art transmitted and reflective microscopes (with digital cameras) are available for on-site material characterisation. Sample preparation is critical to the success of this surface analysis technique. Help on sample preparation is available to users (both on the website and by staff members) and includes, if necessary, access to a sample preparation equipment and specialist support.

ANNUAL TARGETS AND PROGRESS TOWARDS THEM

The total number of S&F instrument user hours of 4265.5 was significantly up on the average of the previous 3 years following a high number of successful applications in the previous year. This usage was equivalent to 94% of the total 4545 hours provided on both instruments. Despite client postponements/cancellations, that are at times difficult to accommodate, all users received time within deadlines set.

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

CAPACITY of HOST ENTITY FUNDED by S&F	Staff & Status	Next Review (March)	Contract Ends (31 March)
60%	Dr J. Craven (65%), Dr R. Hinton (60%), Dr C-J De Hoog (35%), Prof S. Harley (5%), Prof R. Wood (5%)	2017	2018

FINANCIAL DETAILS: CURRENT FY						
Total Resource Allocation £235k	Unit Cost £k			Capital Expend £k	Income £k	Full Cash Cost £k 493.3
	Unit 1 0.125	Unit 2	Unit 3			
FINANCIAL COMMITMENT (by year until end of current agreement) £k						
2014-15	293	2015-16	286			
STEERING COMMITTEE		Independent Members	Meetings per annum	Other S&F Overseen		
		6	2	0		

APPLICATIONS: DISTRIBUTION OF GRADES (current FY — 2014/15)

	10	9	8	7	6	5	4	3	2	1	0	R*	Pilot
NERC Grant projects*			3	2								1	
Other academic		3	6	4								5	
Students			5	3	2		2		1			1	
TOTAL		3	14	9	2		2		1			7	

APPLICATIONS: DISTRIBUTION OF GRADES (per annum average previous 3 financial years —2011/2012, 2012/2013 & 2013/2014)

	10	9	8	7	6	5	4	3	2	1	0	R*	Pilot
NERC Grant projects*		1.33	2	1.33	.33							2.66	
Other academic		2	1.66	2	.33	.66	1.33		.33		1	2.33	
Students		.66	2	2.33	2		.33				.33	1	
TOTAL		4	5.66	5.66	2.66	.66	2		.33		1.33	6	

PROJECTS COMPLETED (current FY – 2014/15)

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

Project Funding Type (current FY – 2014/15) (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		
33	7			11	4		11					

Project Funding Type (per annum average previous 3 financial years - 2011/2012, 2012/2013 & 2013/2014)

Grand Total	Infrastructure						PAYG					
	Supplement to NERC Grant *			PhD Students		NERC Centre	Other	NERC Grant*	PhD Student		NERC Centre	Other
				NERC	Other				NERC	Other		
33.7	8.67			8	5.67		11	0.33				

User type (current FY – 2014/15) (include each person named on application form)

Academic	NERC Centre	NERC Fellows	PhD Students	Commercial
23			11	

User type (per annum average previous 3 financial years - 2011/2012, 2012/2013 & 2013/2014)

Academic	NERC Centre	NERC Fellows	PhD Students	Commercial
20		0.33	13.3	

OUTPUT & PERFORMANCE MEASURES (current year)

Publications (by science area & type) (calendar year 2014)											
SBA	ES	MS	AS	TFS	EO	Polar	Grand Total	Refereed	Non-Ref/ Conf Proc	PhD Theses	
	48	4					52	29	17	6	

Distribution of Projects (by science areas) (FY 2014/15)							
Grand Total	SBA	ES	MS	AS	TFS	EO	Polar
33		29.9	3			0.1	

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

Publications (by science area & type) (Calendar years 2011, 2012 & 2013)											
SBA	ES	MS	AS	TFS	EO	Polar	Grand Total	Refereed	Non-Ref/ Conf Proc	PhD Theses	
	41.66	3.67		1.3			46.33	19.3	20.33	6.67	

Distribution of Projects (by science areas) (FY 2011/2012, 2012/2013 & 2013/2014)							
Grand Total	SBA	ES	MS	AS	TFS	EO	Polar
33.67		28.1	4.33		0.67	0.067	0.5

Distribution of Projects by NERC strategic priority (current FY 2014/15)

Grand Total	Climate System	Biodiversity	Earth System Science	Sustainable Use of Natural Resources	Natural Hazards	Environment, Pollution & Human Health	Technologies
33	1.8		23.1	0.8	7.3		

*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 (2014/15):

The range of science tuned to the NERC strategic priority themes and addressed through ion probe microanalysis in EIMF continues to be both diverse and innovative. Thirty two reports from IMF users are included in this year's Annual Science Report, 31 of which are available on the Facility web site. The Climate System, biodiversity and palaeoclimates in deep time have been considered through several trace element (Sr, Mg, Nd) and isotopic ($\delta^{11}\text{B}$, $\delta^{30}\text{Si}$, $\delta^{18}\text{O}$) studies of organic materials (corals, sponges, conodonts, fish). Natural Hazards research is strong, with a key focus on volatiles, volcanism and volcanic degassing, which is also relevant to Earth System Science through projects on the related near-surface magmatic processes in arcs, flood basalts and Iceland. Earth System Science research in deeper-level magmatic and mantle processes includes new isotopic and trace element constraints on melt contributions to arc magmas, the mobility of H in mantle materials and isotopic evidence for near-surface sources within superdeep diamonds. Projects on trace element fingerprinting of mineral compositions to infer fluid compositions and hence changes in mineralisation and diagenesis illustrate the value of ion probe microanalysis in understanding ore and reservoir processes of relevance to natural resource management. As in previous years, the reports serve to highlight different approaches, both in terms of the elements and isotopes analysed and materials investigated, to the studies of how magmas form, how volcanoes de-gas and how biological materials may record environmental change. They also highlight the importance of controlled experiments, whether at high pressures and temperatures for magmatic processes or at ambient conditions with fixed environmental conditions, integrated with ion probe microanalysis in order to define and correctly calibrate the key signatures of Earth and environmental processes and conditions.

SCIENCE HIGHLIGHTS:

Coupled C and O isotopes of superdeep diamonds and their inclusions. (Burnham et al.) Superdeep diamonds have been shown in previous studies to often have isotopically light carbon, interpreted to reflect either their precipitation at depth in subducted slabs with crustal carbon or in mantle preserving primordial C-isotope heterogeneity. To resolve this controversy this study has examined the O isotope signatures of high-pressure silicate inclusions in such diamonds (coesite, garnet, CaSiO_3 - enabled by significant standard development work in collaboration with SUERC) on the basis that if they preserve high $\delta^{18}\text{O}$ this would confirm a crustal origin for their protoliths and support deep subduction to the transition zone of the Earth's mantle. The silicate inclusions in the lightest diamonds record elevated $\delta^{18}\text{O}$ (up to 13 ‰) consistent with crustal sources for the oxygen. The data further show a strong anti-correlation between C and O isotopes, modelled by these workers to reflect reaction between an oxidised carbonate melt evolved from the subducted slab (the source of the high $\delta^{18}\text{O}$ and low $\delta^{13}\text{C}$ signature) and a reduced ambient deep mantle that contains a carbide phase as well as silicates.

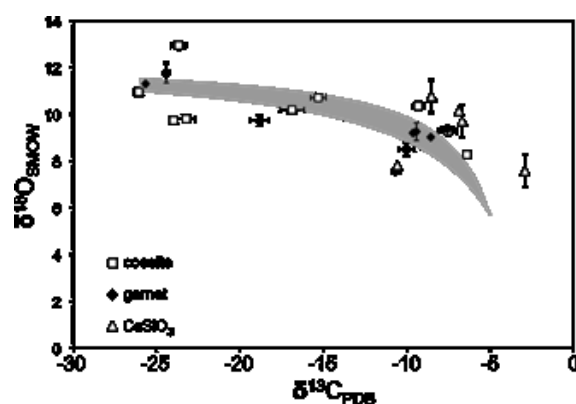


Figure 1: Covariation of $\delta^{18}\text{O}$ (silicate inclusions) and $\delta^{13}\text{C}$ (host diamond) in superdeep diamond samples. The shaded field indicates fits to the data dependent on proportions of mantle carbon phase interacting with carbonate melt.



Figure 2: Example of a black shale melting experiment performed at 850°C. The product consists of about 50% melt (top part of capsule) and 50% residual crystals (lower part of the capsule)

Distinguishing between sediment melting and crustal assimilation in the Lesser Antilles using Molybdenum. (Freythuth et al.) The extent to which sediments can undergo melting during slab subduction, and whether and how such melting contributes to the genesis of arc magmas, is a controversial issue with implications for the origins of arc signatures, the recycling of crust, and the fate of water into the mantle. Fingerprinting unique chemical signatures of sediment melting requires information on the partitioning of trace elements between melts and their residual crystals, determined through high-P-T experiments. This study has determined the partitioning of Mo and Nd between melts and crystals produced from high-Mo black shales, a chemically distinctive sediment found in the Lesser Antilles arc, in order to assess the potential contribution from their melting to the eventual arc magmas which show a range in Nd/Mo correlated with $\delta^{98}\text{Mo}$. The surprising result that the black shale melts have very low Nd/Mo, coupled with their high $\delta^{98}\text{Mo}$, has led to a new model for arc magma genesis in this region. The Mo is derived from a mixture of three sources: melts from the black shales, slab-derived fluids and sub-arc mantle with high Nd/Mo.

Oxygen isotopes in vertebrates: correlating between calcium phosphate and calcium carbonate. (Sansom and Wheeley) Marine vertebrates are increasingly being used for their chemical and isotopic (e.g. $\delta^{18}\text{O}$) archives of climate change, using either calcium phosphate (bone, teeth or scales) or calcium carbonate (ear stones or otoliths). In the case of fish it is possible to examine the $\delta^{18}\text{O}$ in both types of biomineralisation and compare the two. This project has assessed the accuracy of seawater temperature values as determined from otoliths (CaCO_3) versus those from bone (CaPO_4) in the same fish. Crucially, the fish, a barracuda, was from a controlled environment (a tropical tank at 24.5°C) so the results could be compared objectively. SIMS microanalysis of the jawbone phosphate yielded a $\delta^{18}\text{O}$ of 25.3 ‰, giving an average temperature of 23.7°C. The otolith carbonate yielded a $\delta^{18}\text{O}$ of 31.6 ‰, giving an average temperature of 24.6°C. Both estimates are within error of the real tank temperature. This, and the excellent agreement between the two methods, demonstrates the utility of teleost fish in reconstructing sea water temperature records.



Figure 3. Jawbone dentition (left hand images) and otolith (right hand images) from the barracuda studied for phosphate and carbonate $\delta^{18}\text{O}$.

SIMS microanalysis of the jawbone phosphate yielded a $\delta^{18}\text{O}$ of 25.3 ‰, giving an average temperature of 23.7°C. The otolith carbonate yielded a $\delta^{18}\text{O}$ of 31.6 ‰, giving an average temperature of 24.6°C. Both estimates are within error of the real tank temperature. This, and the excellent agreement between the two methods, demonstrates the utility of teleost fish in reconstructing sea water temperature records.

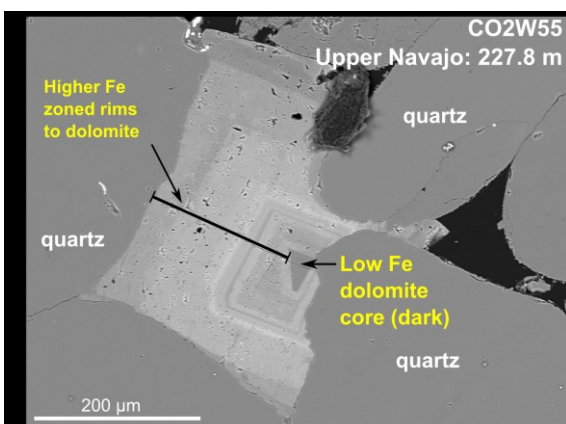


Figure 4. Backscatter image of representative dolomitic cements in the Green River locality, showing the distinct zoning and highlighting the amount of high-Fe dolomite precipitated, correlated with CO_2 -rich reducing conditions.

Trace element compositions of zoned dolomite cements in reservoir rocks from the naturally leaking Green River CO_2 accumulation. (Maskell et al.) An important issue for the storage of CO_2 in deep geological reservoirs is the behaviour of the CO_2 when injected into brines, how this reacts with reservoir minerals and whether this can enhance or limit long-term storage capacity. This project used a range of trace elements, including the REE and Sr, to trace the relationships between CO_2 -charged fluid-rock interactions and Fe-zoned dolomite cement precipitation in a well characterised example of a leaking natural CO_2 -charged reservoir. Pronounced depletions in Ce relative to the other LREE in Fe-poor dolomite indicates these cements were precipitated under oxidising conditions, whereas a lack of Ce depletion relative to the other LREE in Fe-rich dolomite supports the concept that these cements were precipitated under reducing conditions. The reducing conditions are correlated with the passage of H_2S -bearing, CO_2 charged fluids. This study demonstrates that CO_2 -charged fluids can react with host reservoir minerals and precipitate chemically distinctive dolomite, thereby providing a means of ‘fixing’ CO_2 in the reservoir rock mass.

ADDITIONAL SCIENCE HIGHLIGHT:

2014 saw the EIMF publish its 500th peer reviewed international publication, a major landmark for the Facility and SIMS-based geoscience in the UK. The 500th paper was Hartley et al. “Reconstructing the deep CO_2 degassing behaviour of large basaltic fissure eruptions” (EPSL 393, 120-131). This paper, appropriately highlighting the EIMF contribution to research on volatiles and volcanism, was but one of the 29 papers published from the facility during 2014, including 2 in Nature Geoscience and 5 in EPSL (Earth & Planetary Science Letters).

FUTURE DEVELOPMENTS/STRATEGIC FORWARD LOOK

Initial bids for facility time are beginning to be received from successful participants in the NERC Habitable Planet call, and it is expected that these will dominate EIMF usage over the next two years. This will require extensive use of both instruments for the microanalysis of melt inclusions and mineral-melt experimental run products, both for volatile / light element (H, C, Li, Be, B, halogens) and heavier trace element (REE, Nb, Mo, Fe etc) concentrations and for isotope ratios (boron, sulphur, oxygen). We expect several climate-related or biogeochemical projects to continue to require access (corals, conodonts, fish) and others in this area to emerge, whereas demand for accessory mineral U-Pb and trace element studies is likely to remain moderate or low from within the NS&F access route.

As advised in 2013-14, EIMF service provision is likely to be at the cross roads within the next two years as a replacement for the aging IMS 4f instrument will be required if EIMF is to maintain its position as a leading, internationally recognised and competitive facility providing the UK with excellence in ion beam microanalysis. Strong bids for a replacement instrument, requiring a total expenditure of ca. £2.2 million, have been placed with or submitted to NS&F, BGS and Geosciences in Edinburgh in 2014, in the event that funding becomes available.