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

SERVICE	FUNDING	AGREEMENT	ESTABLISHED as S&F	TERM
Life Sciences Mass Spectrometry Facility (LSMSF)	Block	EK: R8/H10/09 L: R8/H10/20 B: R8/12/15	East Kilbride 1994 Lancaster 1984 Bristol 1992	5 years (extended by 1 year)

TYPE OF SERVICE PROVIDED:

Facility: LSMSF comprises three nodes for the provision of organic and light element stable isotope mass spectrometry to the UK life sciences community, located at SUERC, East Kilbride, CEH, Lancaster and Bristol, University of Bristol. A more integrated approach improves accessibility to the Facility nodes and promotes efficient operation of the Facility overall. Whilst located at geographically distinct locations the Facility operates as a 'one-stop-shop' providing users with a single point of contact, the synergistic benefit of this mode of operation makes optimal use of the current resources. LSMSF provides 'free-at-point-of-delivery' support, each node according to their respective service level agreements (SLAs) or contracts, and is overseen by the NERC LSMSF steering committee. Each node offers a different portfolio of analytical techniques which UK based researchers may apply to use *via* NERC Services and Facilities (S&F) by standard peer review procedure. East Kilbride has strong associations with migration, agro-ecology and conservation studies. The primary remit of Lancaster is terrestrial and fresh-water studies (including those deriving from NERC programmes). Bristol fields a wide range of projects to which a compound specific approach is essential, e.g. biomarker analysis, isotopic PLFA and FFA profiling.

Analytical portfolio of the LSMSF:

- Isotopically enriched water ($D_2{}^{18}O$ to energy expenditure studies)
- Natural abundance e.g. $^{13}C/^{12}C$, $^{15}N/^{14}N$, $^{2}H/^{1}H$ and $^{34}S/^{32}S$ analyses of bulk animal organic matter to study food webs & element cycling
- Enriched & natural abundance analyses of organic and inorganic matter to study carbon and nitrogen fluxes within soil ecosystems e.g. $^{13}C/^{12}C$, $^{15}N/^{14}N$, $^{18}O/^{16}O$
- Enriched & natural abundance analyses of gases: e.g. $^{13}C/^{12}C$, $^{15}N/^{14}N$, $^{18}O/^{16}O$ of CO_2, CH_4, N_2O & N_2
- Natural & near natural abundance compound specific $^{13}C/^{12}C$, $^{15}N/^{14}N$, D/H analyses of biochemical extracts
- Organic mass spectrometric analyses of complex mixtures of compounds e.g. volatiles, functionalised molecules etc

ANNUAL TARGETS AND PROGRESS TOWARDS THEM

Bristol:

East Kilbride: The node has continued its high profile program of collaborative support for animal ecology projects; we can now offer routine simultaneous N-C-S isotope measurements for our users.

Lancaster: The node has continued with its high profile program of collaborative support for LSMSF approved projects, twenty this reporting period.

Bristol: 2014-1015 was a period of upheaval (staff changes and instrument installation and infrastructure upgrade), 46% of capacity (58% adjusted for understaffing) was utilised due to several mitigating factors. 2015-2016 will see this balanced.

SCORES AT LAST REVIEW (each out of 5)			Date of Last Review:	2008
Need	**Uniqueness**	**Quality of Service**	**Quality of Science & Training**	**Average**
5.0	4.5	5.0	5.0	4.9

CAPACITY of HOST ENTITY FUNDED by S&F	Staff & Status	Next Review (March)	Contract Ends (31 March)
BRIS 50% **EK** - **LANC** 58%	Dr. ID Bull (URF Grade K; 70% University of Bristol funded), Mrs. A Kuhl (Grade H), Mr JM Williams (Grade H) Dr J Newton (SRF-Level 9), Dr R McGill (RA-Level 7) Dr AW Stott (B5), Miss H Grant (B7), 0.3 FTE Dr. G Pereira (B4)	2016	2017

FINANCIAL DETAILS: CURRENT FY							
Total Resource Allocation £k		**Unit Cost £k**			**Capital Expend £k**	**Income £k**	**Full Cash Cost £k**
		Unit 1	**Unit 2**	**Unit 3**			
BRIS	197.00	0.71					302.2
EK	199.00	0.500				0	213.18
LANC	216.00	0.526				0	233.69

FINANCIAL COMMITMENT (by year until end of current agreement) £k							
2014-15		**2015-16**					
BRIS	197.00	**BRIS**	197.00				
EK	199.00	**EK**	205.00				
LANC	218.00	**LANC**	216.00				

STEERING COMMITTEE	Independent Members	Meetings per annum	Other S&F Overseen
LSMSF	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*	0	0	4.5	1	0	0	1	0	0	0	0	2	0
Other academic	0	1	4	3	1	1	0	0	0	0	1	2	1
Students	0	0	2.5	2	5	2	0	0	0	0	0	4	1
TOTAL	0	1	11	6	6	3	1	0	0	0	1	8	2

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	0	4	2	0	0	0	0	0	0	0	0
Other Academic	0	1	4	2	1	0	0	0	0	0	0	1
Students	0	0	2	3	4	0	0	0	0	0	0	1

Project Funding Type (current FY – 2014/15) (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
55	**12**	**11.5**	**9.5**	**3.5**	**18.5**	**0**	**0**	**0**	**0**	**0**

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	PhD Students Other	NERC Centre	Other	NERC Grant*	PhD Student NERC	PhD Student Other	NERC Centre	Other
51	**11**	**11.32**	**9.66**	**3.16**	**15.83**	**0**	**0**	**0**	**0**	**0**

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

Academic	NERC Centre	NERC Fellows	PhD Students	Commercial
88.5	**19.5**	**2**	**26**	**3**

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

Academic	NERC Centre	NERC Fellows	PhD Students	Commercial
58.49	**11.83**	**1.63**	**25.03**	**1.33**

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
6	**1**	**17**	**0**	**18**	**0**	**2**	**44**	**26**	**9**	**9**

Distribution of Projects (by science areas) (FY 2014/15)

Grand Total	SBA	ES	MS	AS	TFS	EO	Polar
58	**6.333**	**3**	**15.333**	**0**	**29.333**	**0**	**4**

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
6	**3.89**	**15.04**	**1.5**	**21.17**	**0**	**2.05**	**49.66**	**33.33**	**10.3**	**5.99**

Distribution of Projects (by science areas) (FY 2011/2012, 2012/2013 & 2013/2014)

Grand Total	SBA	ES	MS	AS	TFS	EO	Polar
50.82	**5.33**	**1.5**	15.5	0.16	24.33	**0**	**4**

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
55	**3.83**	**22.86**	**8.53**	9.53	0.2	9.03	**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 (2013/14):

Bristol

The node received a total of 7 new applications over the 2014-2015 period of which 4 were supported; 3 were rejected on the basis of the subject area not being within NERC remit or being scientifically flawed. During this time the node serviced a total of 11 projects of which 1 has been fully completed whilst work on the remainder continues with many near completion, this work constitutes a capacity usage of 46% (58% adjusted for understaffing). Whilst disappointing, this lower usage (and project completion) was unavoidable due to changes in facility staffing (loss of ~0.5 FTE and training of new staff) combined with difficulties with obtaining samples from some successful applicants and instrument downtime. One of the Thermo TraceMS GC-MS instruments has now been decommissioned and, following a lengthy tender process, has been replaced with a Thermo Scientific ISQ GC-MS (S&F Capital Call). With a proven ability to deliver HT-GC/MS analyses combined with several innovative time-saving features this instrument is the ideal replacement to provide routine GC-MS analyses. Following a successful bid, led by IDB, to the NERC Strategic Environmental Science Capital Call UoB was awarded £252,000 for the purchase of a GC-MS (with accurate mass and MS/MS capability). Following a relatively short mini-competition the tender was been awarded to Agilent Technologies to supply an Agilent 7200B GC/Q-TOF with robot autos ampler and Mass Profiler Pro (MPP) software suite. As well as providing additional GC-MS capacity this new acquisition will, for the first time, enable routine GC-MS analyses to be made at accurate mass precision (enabling determination of elemental composition). Furthermore, the instrument will also be used for lipidomics/metabolomics based studies and (*via* NBAF) help support metabolomics studies where a GC, rather than LC, based approach is preferable. Together, the ISQ and 7200 GC/Q-TOF further expand the diversity of the facility portfolio of techniques available to UK based life scientists through NERC National Capability. LSMSF-B still maintains an active link with the Bristol Radiocarbon Accelerator Mass Spectrometer (BRAMS) project although what nature any formal relationship between the two shall take is still under discussion. April 2014 saw the departure of Mr James Williams (Grade H) and a new technical position was created and, in September 2014, filled by Miss Stephanie Rankin (Grade F). Whilst this year has not been without its difficulties and setbacks LSMSF-B is now entering the 2015-2016 period extremely well equipped with an enviable range of high-end instrumentation and infrastructural support combined with a full complement of well-trained and capable staff. The expectation is that the ensuing 2015-2016 period shall be a very productive and rewarding.

East Kilbride

The number of applications we receive increases year-on-year, with a record 24 applications last year, of which 13 were supported and most of the remainder invited for resubmission. The large number of applications we receive and then support (24 successful projects underwent measurement/interpretation at some point last year is very challenging for a laboratory with only two staff; it is thus quite challenging to pursue any method developments to keep the node at the forefront of technology and consequently provide a state-of-the-art service to the user community. Notwithstanding our administrative workload, we purchased a dedicated sulphur isotope system (shared with the NERC ICSF) thanks to a successful joint capital bid (£195K) in early 2014, and a year later we have working procedures to run simultaneous N, C and S isotope measurements on our new Pyrocube/VisION combination. The development of NCS has not been without teething problems and has had to take place alongside project analyses, and we have four current projects that require sulphur isotope measurements. Currently we are developing laboratory isotope standards for triple NCS isotope measurements, though it is a challenge to match the concentrations of all three elements with those of samples we are likely to measure. Both members of staff have been on Isoprime's training course for the VisION IRMS and Pyrocube EA in March. Both staff attended the 9th ISOECOL meeting in Perth in August where three talks and two posters demonstrated the strengths of the node, winning both the best student talk and poster prizes. We have produced nine peer-reviewed publications in 2014, and five PhD theses.

Lancaster

All available NERC capacity under the agreed SLA has been utilised by 20 different LSMSFSC approved projects. 65 % of the Lancaster application originated from NERC responsive mode, directed research programmes or NERC studentships. 25% were non NERC direct access and the remaining 10 % originating from NERC Centres, namely the National Oceanography Centre. The nodes diverse analytical portfolio has been used to support four main areas of NERC Science this year: Terrestrial & Freshwater (70 %), Polar (15 %) Marine Sciences (10 %) and Earth Systems (5%). Eight new applications to the node were received this reporting year, of which 6 were from new facility users. 87.5 % of the nodes applications were supported by the LSMSFSC this year with 12.5 % unsupported. Of the 8 applications, 3 were of merit 8, 2 of merit 7, 2 of merit 6 and 1 graded as 5 and unsupported. Instrument downtime has been minimal during the year with little disruption to hamper the efficient operation of the node. Peer reviewed publications have dropped this year, only amounting to one (refereed). However there are 6 that have recently been submitted or under review so manuscripts are forthcoming for 2015-2016 reporting year. We have completed more training in Sulphur isotopes using the LEC dual inlet Pyrocube coupled to an Isoprime 100 and samples can now be routinely analysed.

SCIENCE HIGHLIGHTS. ***To focus on economic and societal impacts and benefits where possible*:**

Bristol: The LSMSF-B involvement with this large research effort came about via a successful application from Professor David Minnikan (University of Birmingham, LSMSFBRIS014). The overall aim of this project was, through the investigation of ancient human remains of subjects infected with leprosy (through the analysis of aDNA and *Mycobacterium leprae* cell wall lipid biomarkers), to obtain information about the migration of people from Central Asia in the first millennium and how this affected the spread of different strains of *M. leprae*. Analyses performed at Bristol used negative ion chemical ionisation (NICI) GC-MS to determine the concentration of mycocerosic acid pentafluorobenzyl ester derivatives derived from remains identified (by visual evidence of paleopathological lesions indicative of leprosy) to have played host to *M. leprae*. The mycocerosates showed an excellent profile of C_{30} to C_{34} mycocerosates for one specimen, corresponding closely with the *M. leprae* standard (Figure 1). The same mycocerosates, typical of *M. leprae*, were also observed, but with much reduced intensity in two other samples. Through the combined assessment of all analyses, it was shown that that historical *M. leprae* from Byzantine Anatolia, Eastern and Central Europe resembles modern strains in Asia Minor rather than the recently characterized historical strains from North West Europe. The westward migration of peoples from Central Asia in the first millennium may have introduced different *M. leprae* strains into medieval Europe and certainly would have facilitated the spread of any existing leprosy.

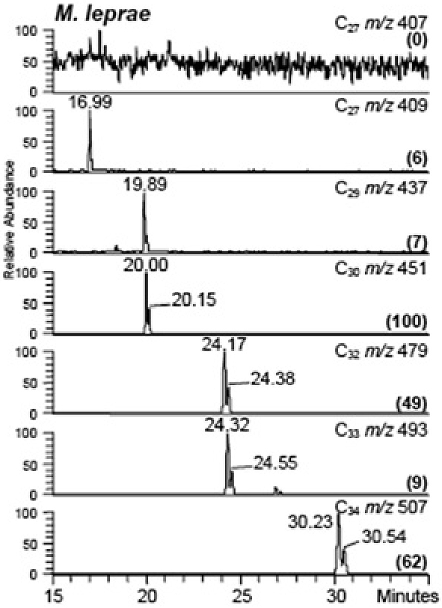

Figure 1 – NICI GC-MS mass chromatograms determined for mycocerosate derived from a *M. leprae* standard.

The subsequent decline of *M. leprae* in Europe may be due to increased host resistance. However, molecular evidence of historical leprosy and tuberculosis co-infections suggests that death from tuberculosis in leprosy patients was also a factor.

Donoghue, H.D., Taylor, G.M., Marcsik, A., Molnár, E., Pálfi, G., Pap, I., Teschler-Nicola, M., Pinhasi, R., Erdal, Y.S., Velemínsky, P., Likovsky, J., Belcastro, M.G., Mariotti, V., Riga, A., Rubini, M., Zaio, P., Besra, G.S., Lee, O.Y.-C., Wu, H.H.T., Minnikan, D.E., Bull., I.D., O'Grady, J. and Spigelman, M. (2015) A migration-driven model for the historical spread of leprosy in medieval Eastern and Central Europe. *Infection, Genetics and Evolution* 31, 250-256.

This, second, piece of in-house research provides the earliest direct chemical evidence for the production of the alcoholic beverage pulque in Mesoamerica, based on organic residues recovered from pottery vessels from Teotihuacan. A novel bacterial lipid biomarker approach that considers detection of bacteriohopanoids derived from the ethanol-producing bacterium *Zymomonas mobilis* for identifying pulque production/consumption in pottery vessels is reported, providing a new means of documenting the consumption of bacterially fermented alcoholic beverages in antiquity worldwide. At Teotihuacan, the authors discovered evidence that pulque was stored in distinctive amphorae vessels sealed with pine resin, as well as in other, less specialized vessels. GC-MS selected ion monitoring (*m/z* 191) of lipid extracts of >300 potsherds revealed characteristic bacteriohopanoid distributions in a subset of 14 potsherds. This direct evidence of pulque production provides new insights into how the nutritional requirements of Teotihuacanos were sustained in a region in which the diet was largely based on plants and crop failures, due to drought and frost damage, which resulted in frequent shortfalls in staples. Moreover, This hopanoid biomarker approach offers a new means of identifying commonly occurring bacterially fermented alcoholic beverages worldwide, including palm wine, beer, cider, perry, and other plant sap- or fruit-derived beverages [Swings J, De Ley J (1977) *Bacteriol Rev* 41(1):1–46].

Correa-Ascencio, M., Robertson, I.G., Cabrera-Cortes, O., Cabrera-Castro, R. and Evershed, R.P. (2014) Pulque production from fermented agave sap as a dietary supplement in Prehistoric Mesoamerica. *Proceedings of the National Academy of Sciences of the United States of America* 111, 14223-14228.

East Kilbride: Few of the many studies which use feathers for determination of $\delta^{15}N$ and $\delta^{13}C$ isotope ratios, consider the consistency of these values within and between feathers from the same individual. The isotopic composition of feathers reflects the diet of the individual during tissue formation, and this moulting period occurs primarily during the nonbreeding season. Knowledge of the sequence and timing of moult in adult birds is essential for robust interpretation of stable isotope data from feathers and this is further complicated in species in which moult may be prolonged or the timing poorly defined, including those that move between several nonbreeding areas.

Grecian et al (2015) looked at feathers from broad-billed and Antarctic prions (*Pachyptila vittata* and *P. desolata*) from the Southern Ocean. Broad-billed prions specialise on large copepods, whereas Antarctic prions feed on a wider range of small zooplankton. Prions undergo a complete moult of primary feathers during the nonbreeding period.

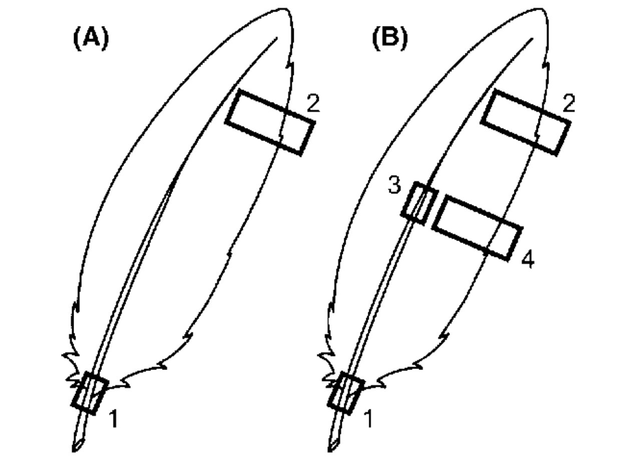

Fig. 1 Feather sampling protocols for a broad-billed prion and b Antarctic prion, illustrating the sampling location of (*1*) rachis, (*2*) vane, (*3*) mid-rachis and (*4*) mid-vane

In broad billed prions – $\delta^{13}C$ varied between different primary feathers, but there was little variation within each feather. Movement

during the moulting period is likely to account for these variations but may reflect changes in distribution or diet during feather regrowth. In Antarctic prions only one feather was available for each individual, but significant differences were found between rachis and vane samples within the same feather. The mid-rachis and mid-vane sections of a feather are presumably grown at the same time reflecting the same diet. The significant difference in $\delta^{13}C$ between these two samples therefore suggests a consistent structural difference between rachis and vane, and could be the result of different diet–tissue discrimination factors.
Within-feather differences were detected in $\delta^{15}N$ for both species. In broad-billed prions feather vane differed from rachis material but in Antarctic prions a longitudinal variation was observed i.e. rachis differed from mid-rachis. This could reflect changes in distribution or diet during feather growth. Overall, however, bird identity explained a larger proportion of variation in $\delta^{15}N$ that samples type in both species and individual dietary specialization may account for persistent differences between individual birds. The study concludes that between-feather differences in $\delta^{13}C$ and between individual differences in $\delta^{15}N$ may be more important than within-feather differences. Nevertheless, it is important to note that the magnitude of within-feather differences was up to ~0.5 ‰ in $\delta^{15}N$ and up to ~0.8 ‰ in $\delta^{13}C$ after accounting for individual and feather differences, and so caution is advised when interpreting isotope data from partial feathers sampled in isolation.
W. James Grecian, Rona A.R. McGill, Richard A. Phillips, Peter G. Ryan, Robert W. Furness (2015) Quantifying variation in $\delta^{13}C$ and $\delta^{15}N$ isotopes within and between feathers and individuals: Is one sample enough? Marine Biol 162, 733–741.


Broad-billed prion


Antarctic prion

Lancaster: Climate change is affecting the amount and complexity of plant inputs to tropical forest soils. This is likely to influence the carbon (C) balance of these ecosystems by altering decomposition processes e.g., “positive priming effects” that accelerate soil organic matter mineralization. However, the mechanisms determining the magnitude of priming effects are poorly understood. We investigated potential mechanisms by adding ^{13}C labelled substrates, as surrogates of plant inputs, to soils from an elevation gradient of tropical lowland and montane forests. We hypothesized that priming effects would increase with elevation due to increasing microbial nitrogen limitation, and that microbial community composition would strongly influence the magnitude of priming effects. Quantifying the respired C sources (substrate or soil organic matter) in response to substrate addition revealed no consistent patterns in priming effects with elevation. Instead we found that substrate quality (complexity and nitrogen content) was the dominant factor controlling priming effects. For example a nitrogenous substrate induced a large increase in soil organic matter mineralization whilst a complex C substrate caused negligible change. Differences in the functional capacity of specific microbial groups, rather than microbial community composition per se, were responsible for these substrate-driven differences in priming effects. Our findings suggest that the microbial pathways by which plant inputs and soil organic matter are mineralized are determined primarily by the quality of plant inputs and the functional capacity of microbial taxa, rather than the abiotic properties of the soil.

Changes in the complexity and stoichiometry of plant inputs to soil in response to climate change may therefore be important in regulating soil C dynamics in tropical forest soils. In conclusion, our findings demonstrate that in these tropical forest soils the microbial pathways by which C inputs are processed and the magnitude and direction of priming effects are driven by substrate-specific properties. Substrate quality (i.e., complexity and N content) determined the source of respired CO_2 (i.e., substrate: SOM) and that partitioning was regulated by the functional capacity of specific microbial groups–in particular K-strategists that access labile C inputs and accelerate SOM mineralization. Changes in the complexity and stoichiometry of plant inputs to soil in response to climate change could therefore be important in regulating soil C dynamics and climate-C cycle feedbacks in these C-rich tropical forest soils.

FUTURE DEVELOPMENTS/STRATEGIC FORWARD LOOK

Bristol

Following the disruption caused by staff changes and upgrades/replacement of instrumentation and laboratory infrastructure over the 2014-2015 period, the next year will see the node concentrating on fully exploiting these improvements to maximise the throughput of new and outstanding applications. Whilst there are no, critical instrument requirements in the immediate future the node is looking at ways for current, more peripheral, techniques (e.g. pyrolysis, thermal desorption, solid phase micro extraction) to be combined be combined in one single, automated, platform to improve efficiency and reliability whilst lowering the overall resource demands associated with providing all of these techniques separately.

East Kilbride

Given the success of the Pyrocube replacement elemental analyser in terms of its throughput and versatility, we intend to replace our old Costech EA with a Pyrocube in the near future. That instrument is becoming difficult to maintain, and is used only for large or “dirty” samples because of its high blank and low throughput. In addition, the ageing microbalance we share with the ICSF (and others) is now seriously oversubscribed to the point whereby our sample throughput is affected: already we are unable to offer the use of the microbalance to all visitors. Thus we also need a replacement microbalance. We anticipated some time ago (in fact it is already evident) that N,C,S isotope measurements will become more popular with our users, and thus we will be putting most of our development effort into ensuring that this is routine for all sample types. Given the VisION has such a large dynamic range, we are looking into developing methods to run biological samples with very low sulphur (or nitrogen) concentrations. JN has been invited to prepare a special topic on Stable Isotopes in Foodwebs for Frontiers in Ecology and Evolution; publication should be due by the end of 2015.

Lancaster

The node will continue to support a wide variety of projects training and augment new methods where applicable. Looking further ahead, we eventually intend to replace at least one of our older IRMS’s with a new Isoprime 100 coupled to a new trace gas pre-concentrator or elemental analyser. This will initially replace ageing pieces of instrumentation as well as enhancing the nodes trace gas and bulk isotopes portfolio.

Non-Mandatory Facility-specific OPMs: utilisation, allocation of capacity etc