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Abstract: The main goal of TA3 (Distributed Sample Analysis Facility-DSAF) is to provide European and international scientist access to four world-leading geochemical laboratory facilities to analyse planetary materials. The urgent requirement for access to these facilities is that Europe has a unique suite of meteoritic samples and samples returned from missions. European scientists have cooperated in, or are preparing further sample return missions. European scientists are also involved in the analysis of lunar samples collected in Russian and US missions. Moreover, high precision analyses are required of samples from terrestrial sites that are the location of the development of early life on Earth and analogues of environments on the moon and Mars. The materials of interest have highly diverse compositions and mineralogy and require careful sample handling by experienced researchers. The expanding planetary exploration programme is generating an increasing demand for the analysis of rare sample material with the aim of obtaining a better understanding of the physical-geological processes that formed specific planetary environments and the biogeochemical processes that control the likelihood that life could evolve or survive. These data will provide important constraints in developing future mission goals.

To address this demand the DSAF comprises four leading national laboratories that are recognized as world leaders in their field, all with specific expertise ranging from in situ secondary ionization methods of solids at sub- μ m scale, isotopic analysis of sub ng amounts of individual elements and isotopic analyses of rare gases. In this reporting period projects ranged from determining the metabolic processes used by microbial communities in the early Archaean (~3.2 Ga) on Earth to determining the timing of processes in the early accretion of planetesimals.

1. Explanation of the work carried out by the beneficiaries and Overview of the progress

1.1 Objectives

TA 3 (WP4) is designed to provide European and a limited number of international users, with access to four internationally renowned centres of excellence to enable state-of-the-art isotope geochemical analysis. "The Distributed Sample Analysis Facility (DSAF)" comprises national facilities in France (CRPG), Germany (Münster), United Kingdom (OU) and the Netherlands (VUA) and offers the broad range of instrumentation required to quantify the complex (bio)geochemical cycles that control the formation and evolution of planetary bodies. Isotopic and geochemical analyses require sophisticated infrastructure and extensive academic and technical support. The combined infrastructure available within DSAF includes a past investment of ~ ϵ 40 M (excluding buildings) mainly sourced from national funding agencies. The entire distributed facility was assembled to provide the comprehensive capability to determine:

- isotopic and elemental analyses at high spatial resolution, down to ~3 nm,
- high precision (down to 5 ppm)
- high sensitivity (sub ng sample sizes).

The broad range of scientific disciplines included in Planetary Science requires that DSAF has the capability to support diverse research. DASAF therefore contains large national facilities with a reputation for multi-disciplinary research and hence the capability to support a wide range of research topics related to fundamental processes that led to the formation of the planetary bodies in the Solar System (e.g., accretion & differentiation processes), to atmospheric and hydrological process at planetary surfaces. The understanding of the latter is required to support researchers studying planetary analogues in field studies in their quest to understand habitability on Earth and potentially on other planets.

JRA 3 introduced new capabilities for improved sample handling techniques (minimally destructive) and the analysis of smaller sample size. These new capabilities became fully available for access in late 2017. This resulted in a major increase in the number of TA applications in Call 3 and 4, which continued in Call 5.

The main goal of the facilities that comprises WP4 is to ensure that the visits by TA users are organised and implemented efficiently and that results obtained ultimately appear in peer reviewed journals so that the wider implications of the research are disseminated widely to policy makers. Brief details of the scientific teams, the topics covered and output resulting from these visits are listed below.

Description of the Trans-national Access activities during the third annual reporting period (1st September 2018 – 31st August 2019):

Task 4.1- Geology and Geochemistry Isotope Facility (GGIF)), Department of Earth Sciences Vrije Universiteit, Amsterdam, the Netherlands

There were 2 visits to the Geology and Geochemistry Isotope Facility during RP3, with 1 female/1 male lead applicants, from two countries (Ireland, Israel).

Proposal	Access	Date of visit	Name of visitors	Project Title
number	site			
18-EPN4-055	GGIF-	17.06. –	Mr Brendan Hoare	The making of stable continents: Testing
	VUA	28.06.19		an exsolution origin for the Archaean
				cratonic lithosphere
18-EPN5-002	GGIF-	19/05/2019-	Dr Yaakov Weiss & Oded Elazar	Micro-Windows To Deep Carbon- and
	VUA	01/06/2019 &		Water-Rich Planetary Fluids And
		21/07/2019-		Associated Mantle Processes
		02/08/2019		

Project 18-EPN4-055:

PhD candidate Brendan Hoare from University College Dublin undertook a TA visit to address the fundamental processes that led to the formation and stabilisation of Earth's earliest continental crust and its underlying lithospheric mantle. Understanding the processes responsible is considered vital in determining how the Earth, and by analogue the terrestrial rocky planets, differentiated into core-mantle and crustal physio-chemical reservoirs. The visit analysed garnet bearing xenoliths from the Kaapvaal Craton in South Africa (Fig. 1). The analyses were a success and high quality Lu-Hf isotope date were obtained that confirm the antiquity of the samples.



Fig. 1: Orthopyroxene with garnet exsolution lamellae juxtaposed with granular garnet harzburgite. The Lu-Hf isotope analyses were conducted on the exsolved garnet grains that had been painstakingly separated from the orthopyroxenes.

A publication is in preparation: Hoare, Tomlinson et al., that will include the Lu-Hf data along with major and trace element data previously acquired. The publication will argue that the presence of garnet is intimately linked to craton stabilisation and will draw parallels between exsolved and 'normal' garnet found in. Both Tomlinson and Hoare made presentations about Kaapvaal Craton xenoliths at the Goldschmidt conference in Barcelona in August 2019.

Project 18-EPN5-002:

Volatiles, along with heat, are the fundamental parameters that control the melting and differentiation of planetary bodies. Moreover, they ultimately form any atmosphere. Determining how volatiles are re-distributed within the Earth is therefore vital information in understanding past and on-going evolution of the terrestrial planets and potentially provides first order information in understanding complex geochemical cycles, such as the C-cycle. With this background in mind a project was undertaken to validate that fluids trapped in diamond could be sampled in sufficient quantity to allow coupled major element, trace element and combined Sr-Nd-Pb isotope ratios to be determined. The project was designed as a method validation exercise but the data obtained are of such high quality that a publication is expected and further work will be undertaken.

The study undertaken by PhD student Oded Elazar and his supervisor Yaakov Weiss and focussed on fibrous diamonds, a fast-growing form of diamonds that often encapsulate carbon- and water-rich (C-O-H) high density fluids (HDF) as micro-inclusions. These micro-inclusions are a primary target for studies of C-O-H mantle fluids and how these fluids influence deep Earth processes. However, generally only a small amount of diamond (a fraction of a mg) and even smaller amounts of C-O-H micro-inclusions can be sampled and analysed by conventional approaches, yielding elements contents in the sub-ng range, typically too small for precise analyses using conventional mass spectrometry measurements.

To overcome the sampling hurdle, C-O-H fluids were collected from 11 micro-inclusions-bearing fibrous diamonds from Canadian using a new laser ablation of diamond-in-liquid technique. Analyses of this material coupled with low blank column chromatography and 10¹³ Ohm resistor TIMS analyses at Amsterdam provided, for the first time, high precision Sr-Nd-Pb isotope analyses of C-O-H mantle fluids encapsulated in micro-inclusions-bearing diamonds.

Previous studies have established that HDFs vary in major-element compositions between four major types: silicic, saline, and high-Mg and low-Mg carbonatitic. Moreover, they display two main traceelement patterns, regardless of their major element composition or origin: one with high field strength element (HFSE) depletions and large ion lithophile element (LILE) enrichments similar to calc-alkaline magmas and continental rocks, the other with lower LILE abundances and 'smoother' overall traceelement patterns similar to oceanic basalts e.g. [1, 2]. Laser ablation was performed on representative samples of known different composition and different sized samples were obtained to evaluate the extent of elemental contamination during sampling.

In total 24 samples, including blanks and standards, were processed in the VU ultra-low-blank clean laboratory and Sr, Nd, Sm and Pb were separated by column chromatography. The isotopic compositions of Sr, Nd, Sm and Pb were determined for each sample using 10¹³ Ohm resistor TIMS analysis at VU.

Figure 1 shows a comparison between the Sr and Nd isotope ratios of 4 duplicates of diamond no. 509 (sample weights of 0.1, 0.3, 2.8 and 3.4 mg; Fig. 2). The \sim 3 mg diamond samples are within error for Sr and Nd isotope ratios, whereas measurement of <1 mg diamond with higher blank/sample ratios show larger variability and uncertainties. These results indicate that the new laser ablation of diamond method, combined with the analytical protocol for isotopic measurements established at VU is viable. However, the work recognised that that improvement of the blank during the laser ablation will allow smaller sample sizes to be analysed (Fig. 2). Methods to improve this blank have been recognised and work is already underway to do so.



Figure 2. Sr and Nd isotope data obtained on different sized fibrous diamond. These data establish that the Nd blank is almost insignificant but that the current Sr isotope blank requires improvement to allow sub-mg size sample analysis. The Sr isotope data can be used to calculate the isotopic data of the blank and hence allow blank correction of the data from the larger samples, leading to even more precise isotopic data. Typically these correction are less than 1%.



Figure 3: Nd and Sr isotope compositions of HDF-bearing diamonds (green diamonds >2.5 mg sample; blue triangles <1 mg). Also shown are the values for bulk Earth and the mantle [3,4], group I and II kimberlites [4], marine sediments [5] and river suspended material [6].

The analysed HDF-bearing diamonds record large variation in Sr-Nd-Pb isotope ratios (e.g. Fig 3), between bulk Earth values to more radiogenic Sr and unradiogenic Nd ratios that characterize marine sediments and rivers suspended material (Figure 2). This isotopic variation supports the association between subduction-derived fluids, mantle metasomatism and fluid-rich diamond formation, and suggests that C-O-H fluids are cycled between Earths' surface and interior. In addition, the similarity in Sr and Nd isotopic composition between HDFs and kimberlites may indicate genetic relation or reflect a similar trace-element enrichment and depletion processes experienced by their mantle sources.

The results established that the combined methodology is viable but requires improved blanks to work on smaller samples. The success of the project will lead to a joint PhD project between The Hebrew University and VUA. These data will be the basis of 2 papers, one technical and the second focussed on data interpretation. Dr Weiss made a presentation on the isotopic results of HDF-bearing diamonds at Goldschmidt in August 2019.

- 1. Tomlinson, E.L., A.P. Jones, and J.W. Harris, Co-existing fluid and silicate inclusions in mantle diamond. Earth and Planetary Science Letters, 2006. 250(3–4): p. 581-595.
- 2. Weiss, Y., W.L. Griffin, and O. Navon, Diamond-forming fluids in fibrous diamonds: The trace-element perspective. Earth and Planetary Science Letters, 2013. 376(Supplement C): p. 110-125.
- 3. Zindler, A., Hart, S., 1986. CHEMICAL GEODYNAMICS. Annual Review of Earth and Planetary Sciences 14, 493-571.
- 4 . Becker, M., Le Roex, A.P., 2006. Geochemistry of South African on- and off-craton, Group I and Group II kimberlites: Petrogenesis and source region evolution. Journal of Petrology 47, 673-703.
- 5. Ben Othman, D., White, W.M., Patchett, J., 1989. The geochemistry of marine sediments, island arc magma genesis, and crust-mantle recycling. Earth and Planetary Science Letters 94, 1-21.
- 6. Goldstein, S.J., Jacobsen, S.B., 1987. The Nd and Sr isotopic systematics of river-water dissolved material: Implications for the sources of Nd and Sr in seawater. Chemical Geology: Isotope Geoscience section 66, 245-272.

Task 4.2: Radiogenic, non-traditional stable & rare gas isotopes. Le Centre de Recherches Pétrographiques et Géochimiques (CRPG-CNRS), Nancy, France:

[Ion probe facility (IPF) - Helium and Nitrogen isotope facility (HNIF)- Stable and Radiogenic Isotope Facility (SRIF)]

There were 9 visits to the TA facilities at CRPG during RP3, with 1 female/10 male lead or coapplicants, from five countries (Belgium, Germany, Netherlands, Norway UK,).

Proposal	Access	Date of visit	Name of visitors	Project Title
number	site			
16-EPN2-066	IPF	10-18 Dec 2018	George Jacobs, Open University, UK	Investigating the chronology of clast formation and disruption in martian meteorite Northwest Africa 7034
18-EPN3-059	IPF	17-21 Sept 2018	Olivier Namur & Bernard Charlier, KU Leuven Belgium	Carbon solubility in reduced silicate melts: Implications for the differentiation of Mercury.
18-EPN4-050	SRIF	14-18 Jan & 18- 21 Jan 2019	Stein Holly & Judith Hannah, University Oslo, Norway	Planetary Degassing Of Mercury: Impact On Life and Death
18-EPN4-071	IPF	29 July – 2 August 2019	Timothy Gregory and Tu-Han Luu Bristol UK	New Constraints On The Chronology Of Chondrule Formation
18-EPN5-005	IPF	17-21 Sept 2018	Olivier Namur, KU Leuven Belgium	Carbon solubility in reduced silicate melts: Implications for the differentiation of Mercury
18-EPN5-025	IPF	20-27 July 2019	Timothy Gregory, Bristol UK	Constraining the Variability and Chronology of Chondrule Precursors
18-EPN5-042	IPF	20-25 May 2019	Maxim Portnyagin & Nikita Mironov GEOMAR Germany	Estimation Of Initial CO2 and S Contents And Their Sources In Parental Arc Earth Magmas Of Kamchatka Volcanoes: Insight From Local Sims Analyses Of C, S And 34S/32S In Glasses Of Homogenized And Non-Homogenized Olivine-Hosted Melt Inclusions
18-EPN5-043	SRIF	1-5 July 2019	Pieter Vroon, VUA Netherlands	Germanium Isotope Variations In The 3.5 Ga Old Buck Reef Chert Deposit
18-EPN5-053	SRIF	8-12 July 2019	Stepan Chernonozhkin, Ghent Belgium	Germanium Isotopic Composition Of Ureilite Meteorites And Their Components - Traces Of Accretion And Early Planetary Differentiation

Table 2. Visits to DAFS facility at CRPG, Nancy, France

Project 18-EPN4-050

To understand the past effects of major climate change, Hg concentrations and isotopic compositions were determined for samples from famously organic- and sulphide-rich intervals in deep time. Notably, (1) pristine oil-rich shale and copper sulphide samples (underground samples) from the upper Permian Kupferschiefer in Germany-Poland, (2) organic-rich shales embracing the Permo-Triassic boundary in the Perth Basin, Australia (Hovea), and (3) organic-rich latest Triassic shales and carbonate rocks from Sicily associated with the incipient opening of the Atlantic Ocean. All three localities are associated with biotic crises, and all three later developed mobile hydrocarbon from the organic cargo deposited during those biotic crises.

Studied samples show a wide range of Hg contents, from 1 ppb in some oil fractions to 23 ppm in bitumen-infiltrated shale from Sicily. Typical organic-rich shales range from 30-300 ppb Hg, with systematic stratigraphic variations revealed in the more densely sampled Hovea and Sicily locations. Acquiring data for a densely sampled Kupferschiefer section is planned for our continued collaboration (Stein, ERC-AdG to be submitted, August 2019). Further, our study is the first to explore the distribution of Hg between different oil and bitumen fractions, and kerogen.



Fig. 4. Mass-dependent (x-axis) and mass independent (y-axis) Hg isotopic variations in studied samples.

The data was presented at the recent Goldschmidt conference in Barcelona and a manuscript is in preparation.

Georgiev, S.V., Stein, H.J., Yang, G., Hannah, J.L., Grice, K., Cloquet, C. (in preparation). Raucous Os-Hg signals in a non-isochronous near-shore setting, Perth Basin, Western Australia.

There have been regular publications from previous TA visits ; e.g., 16-EPN2-014 (Kinetic isotope fractionation by diffusion in laboratory and natural zoned olivines).website Some papers have had major impact, for example 16-EPN2-047 (Analysis of Stable Isotopes in the Driest Locations of Earth, with Applications to Mars led to a publication in PNAS (Schulze-Makuch et al. 2018) and a paper is in submission to Nat. Geoscience from the work conducted in 17-EPN3-074 (Tracing metabolic pathways of Archean microbial community's; S. Nabhan). TA visitors have regularly presented their work at international conferences such as LPSC, EGU, EPSC and Goldschmidt.

Dirk Schulze-Makuch & 45 (2018). Transitory microbial habitat in the hyperarid Atacama Desert Others. PNAS, 115 2670–2675.

Task 4.3- Radiogenic & stable isotopes. Open University, Milton Keynes, United Kingdom

NanoSIMS facility (HS50L) Laser Flourination stable isotope laboratory (LFS).

There were 5 visits to the TA facilities at OU during RP3, with 1 female/7 male lead or co-applicants, from five countries (France, Germany, Italy).

Proposal	Access	Date of visit	Name of visitors	Project Title
number	site			
17-EPN3-063	HS50L	16-18 Oct 2018	Tomasso Di Rocco & Maurizio Gemelli, Uni Pisa, It	The fractionation of refractory lithophile elements (RLE) and the oxygen isotope systematics in enstatite chondrites (II).
18-EPN4-64	HS5OL	20-26 Jan 2019	Denis Harries, Jena, Germany	The origin of terrestrial water: A perspective of metamorphism and fluids within planetesimals of the inner Solar System.
18-EPN4-69	LFS	31 March- 4 April 2019	Jan Hellmann, Uni Munster Germany	Oxygen Isotopes And The Number And Thermal History of ordinary chondrite parent bodies
18-EPN4-80	HS50L	15-19 May 2019	Keyron Hickman-Lewis CNRS Fr and & Barbara Calvazzi, Uni Bologna It.	Determination Of Isotopic Signatures Of Carbonaceous Matter In Mars-Analogue Early Archaean Cherts
18-EPN5-044	HS50L	8-12 July 2019	Martin Stuttle, University Pisa, It	Oxygen Isotope Signatures Within Primitive Cometary Micrometeorites: Revealing The Mechanisms Of Early-Stage Aqueous Alteration In The Outer Solar System- Trace s Of Accretion And Early Planetary Differentiation

Table 3. Visits to DAFS facility at OU, UK

Project 18-EPN4-060:

Micrometeorites are millimetre-sized dust grains which originate from local solar system small bodies (Genge et al., 2008). Their analysis provides information on the composition of asteroids and comets and helps us better understand the origin and evolution of our solar system. Furthermore, data from micrometeorites provides a complementary perspective to the insights obtained from the study of larger meteorites.

Triple oxygen isotope data was collected on 6 giant (>500µm) unmelted micrometeorites from the TAM collection. A further 8-12 particles which will be measured in March/April (2019), without my attendance. Particles had been pre-characterised by high resolution µCT on the PSICHE beamline at the French synchrotron SOLEIL. This study therefore represents the first attempt to analyse the bulk O-isotopic signatures of unmelted micrometeorites. Through the use of µCT we were able to avoid contamination of samples by epoxy resin (which is otherwise necessary for the conventional microanalysis pathway of micrometeorites). This allowed us to examine in detail their particle textures and pair this with their isotopic data. Furthermore, because particles are unmelted, they experienced minimal mass-dependent fractionation and exchange with terrestrial oxygen, overprint artefacts that affect all previous O-isotopic measurements performed on bulk (melted) micrometeorites (referred to as cosmic spherules). Finally, the high precision of the laser fluorination line allowed us to more accurately constrain parent body sources. This demonstrated that all of the hydrated fine-grained micrometeorites we analysed originate from a CR chondrite source – an unexpected result as previous literature analyses suggest a CM source is dominate. Likewise, all of the coarse-grained micrometeorites so far studied originate from an L/LL source within the ordinary chondritic field.

The data were present at Metsoc 2019 conference in Japan and a manuscript is currently under review in PNAS.

Task 4.4- Radiogenic & non-traditional	table isotopes: Institute for Planetology; University of
Münster. Münster. Germany	

There were no applications for the WWM facility in the final TA call. There were 2 visits to the TA facilities at WWM during RP3, with 2 male leads from the UK.

Proposal number	Access site	Date of visit	Name of visitors	Project Title
18-EPN4-029	RNTSI	6-15 May 2019	Richard Windmill &Mahesh Anand Open University UK	Untangling The Earliest Stages Of Planetary Differentiation
18-EPN4-030	RNTSI	21 Jan to 1 Feb 2019	Mahesh Anand & Richard Windmill, Open University UK	Insights Into Differentiation Of Planetary Objects / Title on report: Chronological constraints on planetary differentiation and subsequent evolution

18-EPN4-030

Pallasites, meteorites characterised by a mixed silicate-metal assemblage (Figure 4) were once thought to originate at the core-mantle boundary of differentiated asteroids but recently it was proposed that they are impact-generated mixtures of core and mantle materials.



Figure 5; Image of the Esquel Pallasite from the British Museum Collection.

To address the origin of pallasites, nine samples from five Main Group pallasites (PMG), four samples of olivine and five of metal-hosted chromite, were prepared for Cr isotope analysis by TIMS. These samples included both olivine and chromite phases from Sericho, Seymchan, Hambleton and Fukang, as well as chromite from Brenham. The aim of these analyses was to establish whether a resolvable isotopic difference exists between olivine and metal-hosted chromite in PMGs in order to test some recent impact formation models for pallasite generation (Tarduno et al., 2012). The procedure followed was modified from that outlined in Yamakawa (2009) and involved dissolution of the phases and subsequent column chemistry steps aimed to separate chromium from the sample matrix. After Cr separation and purification the samples were mounted on to filaments and analysed using a Thermofisher TritonPlus thermal ionization mass spectrometer. Owing to their Mg-rich composition, cation removal from the olivine samples was unsuccessful initially and has had to be repeated three times. The results to date for Sericho chromite are ε^{53} Cr = 0.01 ± 0.07, ε^{54} Cr = -0.49 ± 0.29. For Seymchan chromite ε^{53} Cr = -0.03 ± 0.2 and ε^{54} Cr = -0.45 ± 0.3. For Hambleton chromite ϵ^{53} Cr = -0.14 ± 0.07 and ϵ^{54} Cr = -0.79 ± 0.1. For Brenham chromite ϵ^{53} Cr = -0.03 ± 0.07 and ϵ^{54} Cr = - 0.43 ± 0.18 . These results highlight a small difference in ε^{53} Cr and ε^{54} Cr between Hambleton and the other samples which is also seen in the cation composition of these samples. Brenham, Sericho, and Seymchan all contain end-member chromite with little or no Mg²⁺ and/or Al³⁺. By contrast, Hambleton contains a small amount of Mg²⁺. It may be that bonds between the Fe and Cr in Seymchan, Sericho, and Brenham, preferentially incorporate the heavier Cr isotopes when compared to the Mg-Cr bonds in Hambleton. Fukang, which contains a high amount of Al in the 3+ site, may show a similar difference. Following further analysis of Fukang chromite and the olivine samples from Hambleton, Sericho, Fukang, and Seymchan, it should be possible to identify whether there is any disequilibrium between the metal-borne chromite and the olivines. If this is the case, it will strongly support an impact mechanism for pallasite generation (e.g. Tarduno et al., 2012). Given that some pallasite groups are thought to have formed in the outer solar system (Warren, 2011), such a discovery would suggest that an impact accretion mechanism for pallasite formation was solar system-wide. It is likely that a conference talk and/or a paper will come as a result of this work.

Tarduno, J. T., Cottrell, R. D., Nimmo, F., Hopkins, J., Voronov, J., Erickson, A., Blackman, E., Scott, E. R. D., McKinley, R. 2012. Evidence for a Dynamo in the Main Group Pallasite Parent Body. *Science*, 338, pp 939-942.

Warren, P. H. Stable-isotopic anomalies and the accretionary assemblage of the Earth and Mars: A subordinate role for carbonaceous chondrites. *EPSL*, 311, pp 93-100.

Yamakawa, A., Yamashita, K., Makishima, A., Nakamura, E. 2009. Chemical Separation and Mass Spectrometry of Cr, Fe, Ni, Zn, and Cu in Terrestrial and Extraterrestrial Materials Using Thermal Ionization Mass Spectrometry. *Analytical Chemistry*, 81 (23), pp 9787-9794.

1.2 Impact

The international impact of TA3 has been substantial and will be discussed in detail in the final report. There were non EU applications (Ethiopia, Israel, India, South Africa and the US) but only one application from an Under Represented State (Greece). Visits have resulted in many presentations at workshops, conferences and seminars, including Metsoc, Lunar and Planetary Science Conference, EGU. The session LFI2 "The distributed planetary simulation and sample analysis facilities" at EPSC has showcased research enabled through the first three calls of TA3, including oral presentations and posters at EPSC 2017. Multiple presentations are scheduled for a dedicated session on laboratory applications in support of planetary missions at EPSC 2019 in Geneva.

Details of publications arising from the work are on the website and will be discussed and analysed in detail in the final report.