NIWA
Taihoro Nukurangi

Assessing New Zealand's Stable Isotope Research Capabilities

National Workshop

Hosted by NIWA Wellington
6–7 November 2008

Summary Report

Compiled by Dr Sarah Bury

NIWA Information Series No.73

Assessing New Zealand's Stable Isotope Research Capabilities

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Hosted by NIWA, Wellington

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Summary report compiled by
Sarah Bury

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Introduction

Dr Sarah Bury (NIWA) introduced the workshop, and provided the following rationale and aims:

- At NIWA we have reached a point where we need to replace some of our aging stable isotope (SI) analytical instrumentation
- We wanted an integrative and collaborative approach involving the whole New Zealand SI community as means of identifying NIWA & New Zealand's technical requirements for SI research rather than making decisions about the way forward in isolation
- We organised this meeting to identify gaps in equipment and research, markets, & potential for collaboration so expensive purchases can be made wisely
- The aim is to encourage a free and open dialogue between NZ labs, organisations and end users: we look forward to enhancing and strengthening collaborations
- We need to be mindful and respectful of existing expertise and collaborations
- This is the 1st meeting of its kind. We hope this will be the stimulus for annual meetings of the SI community.

Welcome

Dr Clive Howard-Williams (Chief Scientist – Freshwater & Coasts, NIWA) welcomed participants to the workshop.

Dr Howard-Williams emphasised the need for such a meeting, stressing that direct interactions and face to face or phone conversations can forge much closer, more beneficial collaborations, than communications by email. He also stressed the importance of collaborative science, including sharing lab/sample analysis costs to build cases for high-end & new innovative technical purchases.

Original meeting information

The original meeting information and programme are provided in Appendix I.
Appendix II summarises New Zealand's analytical capabilities and complementary facilities.
A list of workshop participants with full contact details is provided in Appendix III.

PowerPoint presentations

Presenters have kindly agreed to make their PowerPoint presentations available to all registrants of the meeting. These are available in pdf format on CD and accompany this report. Some unpublished data have been removed at the request of the presenters. Please contact them directly if you require further information. Post-meeting updates of activities, actions and notes are provided in the report where relevant in italics within square brackets [xxx].

1. New Zealand's Stable Isotope Analytical Capabilities and Applications

Day 1 – Thursday 6 November

Presentations of current and future New Zealand stable isotope analytical capabilities from each laboratory including details of:

1) Current analytical capabilities
2) Current innovative applications
3) Key future applications
4) Limitations of existing equipment
5) Current gaps in technology
6) Wish list for new technology

1.1 GNS Science National Isotope Centre

Valerie Claymore **GNS Stable Isotope Laboratory capabilities and applications**

[P1_GNS_Claymore et al.pdf. All GNS presentations are compiled into this file]

Expertise & Analytical Capabilities

- Valerie provided a history of the GNS Stable Isotope Laboratory
- Technical staff in the lab: Andy Philips (mass spectrometer engineer), Jannine Cooper, Pam Rogers, Lan Hui
- GNS SI scientific staff: Kevin Faure, Karyne Rogers, Nancy Beavan, Troy Baisden, Nancy Bertler, Bruce Christensen, Uwe Morgenstern, Cornel de Ronde, Chris Hollis, Martin Crundwell, Perry Davy
- Laboratory currently measures all 5 light isotopes $\delta^{13}C$, $\delta^{15}N$, $\delta^{18}O$, $\delta^{34}S$ and δD in most phases
- Summary of isotope ratio mass spectrometer instrumentation: 5 isotope ratio mass spectrometers (2 Europa Geo 20-20, 3 GVI Isoprime), 8 automated preparation systems, offline combustion facilities and a sample preparation laboratory. Mass spectrometers comprise:
    - GVI Isoprime (Frodo) dual inlet & continuous flow with hydrogen collectors linked to GVI Aquaprep (Galadriel) Gilson 60 position autosampler: measures $\delta^{13}C$, $\delta^{18}O$ of small CO_2 gas samples & water
    - GVI Isoprime (Sam) continuous flow with hydrogen collectors, dual reference box, diluter and hydrogen collectors linked to Eurovector PyrOH (Eowyn) elemental analyser with LAS300 100 position liquid autosampler. Reduction by direct injection of water over hot chromium: measures δD in water
    - GVI Isoprime (Rosie) continuous flow with hydrogen collectors, dual reference box & diluter box linked to:
        - HEKAtech High Temperature Elemental Analyser (HTEA) (Borimier): high temperature furnace with 80 position solid autosampler: measures $\delta^{18}O$, δD in minerals, sulphates & organic samples
        - CSIA – Agilent Gas Chromatograph (GC) with combustion furnace (Aragorn) GC combustion system with 100 position autosampler for liquid injection and gas inlet: measures $\delta^{13}C$ in gases and hydrocarbons
    - Europa Geo 20-20 (Mari) dual inlet & continuous flow linked to:
        - automated 24 port manifold: measures $\delta^{13}C$, $\delta^{18}O$ in offline combustions of samples from radiocarbon laboratory or from CO_2 produced on laser line
        - ANCA elemental analyser (Legolas) with 130 position solid sample autosampler: measures $\delta^{13}C$, $\delta^{15}N$ in solid organic samples
    - Europa Geo 20-20 (Pippin) dual inlet & continuous flow, linked to:
        - Carbonate automated preparation system (CAPS) (Celeborn), 24 vial carousel: measures $\delta^{13}C$, $\delta^{18}O$ in calcium carbonate, calcite, aragonite

- Sercon sulphur elemental analyser (SEA) (Theodin), single combustion furnace with 50 position autosampler: measures $\delta^{34}S$ in mineral deposits, solid organic samples.

Offline sample preparation systems
Facilities available for preparation of samples for analysis on Europa 20-20 (Mari) dual inlet & continuous flow mass spectrometer include:
- Br5F laser fluorination line, for extracting CO_2 from Silicates
- DIC vacuum line, for extracting CO_2 from dissolved inorganic carbon (DIC) in water
- Combustion line, for extracting CO_2 from low carbon organic samples.

Additional analytical capabilities at GNS
- Radiocarbon Lab (^{14}C)
- Water Dating Lab (^{3}He, CFC, SF6, ^{32}Si)
- Water & Gas Laboratory (fluid and gas chemistry analysis)
- Mineral Lab (x-ray diffraction (XRD), rock & mineral preparation)
- Ion Beam Analysis (identification of most elements on the periodic table)
- Cosmogenic Isotope and Radiochemistry Lab (^{10}Be, ^{137}Cs, ^{210}Pb, ^{226}Ra and other U series isotopes)
- Terrestrial Isotope Biogeochemistry Lab (pyrolysis gas chromatography mass spectrometry (GCMS): Biomarker tracers in soils, sediments and dissolved phases
- New Zealand Ice Core Research Laboratory: established and funded by GNS Science and Victoria University under the umbrella of Joint Antarctic Research Institute, with subsequent support & collaboration with NIWA. Contact: Nancy Bertler. Housed in a purpose-built building at GNS Gracefield site comprising:
  - office space
  - ultra-clean lab
  - working freezer to -18°C
  - storage freezer to -35 °C with capacity to store 2000m of ice cores.

GNS Research and Applications

Valerie Claymore & Nancy Bertler **Ice core research**

- Nancy Bertler has been carrying out research on Victoria Lower Glacier, Antarctica (near McMurdo Station) using deuterium isotopes to generate palaeoclimate temperature records
- Ice cores are excellent recorders for regional temperatures
- A large annual temperature gradient (+10°C to -60°C) has been observed
- Ice core proxy isotope temperatures correlate well with regional temperature variations
- Nancy has reconstructed 900 years of temperatures from δD isotope ice core data.

Kevin Faure **Oxygen isotopes of silicates**

- GNS have developed a method to measure oxygen isotopes in silicates
- A CO_2 laser line is used to break the very strong O_2-silicon bond, whereby silicon is vapourised & oxygen can be extracted from the minerals enabling O_2 isotope signatures to be measured
- GNS can measure O_2 isotope signature of kaolinites (palaeoclimate interpretations), ore deposits/minerals from geothermal and volcanic environments (origin & formation of ore-bearing fluids & minerals)
- The method has facilitated mineral exploration: the analysis of minerals associated with gold has provided information on fluid origins and has led to the discovery of other gold deposits.

Wish List/Future work

- Extraction of O_2 from biogenic silica (palaeoenvironmental applications) – setting up collaborations with scientists working with diatoms& biogenic silica.

Nancy Beavan **Bioarchaeology and ecology**

15^{th}-17^{th}C Jar Burials in the Cardamon Mountains, Kingdom of Cambodia

- Nancy has investigated dietary offsets in radiocarbon dates of cooking pots found in Cambodian mountains using $\delta^{13}C$, $\delta^{15}N$ and $\delta^{34}S$. She used stable isotopic signatures to identify if there were marine offsets to the radiocarbon dates
- To do this you need region-specific stable isotope databases, as global data-sets are often not appropriate. The offsets depend on type of tissues used in analyses (e.g., skin, bone, muscle)
- C, N & S isotope analysis determined that populations in 15^{th}-17^{th}C were not eating much in way of marine foods
- Future plans are to use Sr and O isotopes to determine origin of populations.

Isotopic Analysis of Watom Island, Papua New Guinea inhumations to assess the influence of marine sources on ^{14}C ages

- In this study Nancy used C, N and S isotopes to establish if Watom Island human populations were marine foragers or agriculturalists
- Marine food in their diet was offsetting radiocarbon ages
- Found strong marine sulphur isotope signature in pigs and in the island's vegetation (due to sea-spray effect in vegetation) – possible that pigs of island were marine foragers – population could have gained their marine signature from pigs and vegetation rather than from directly eating seafood
- Seaweeds collected yesterday can give 2000 yr old ^{14}C signal!

Perry Davy **Stable isotopes and air particulate matter (APM)**

- Perry uses stable isotope ratios as an indicator of air particulate matter (APM) sources and investigates air particulate matter sources in urban environments
- Some of sources do not have unique SI signature, e.g. SI fossil fuel values are similar to wood burner emission values; wood burner emission values are similar to Wainouimata ambient values
- There are problems associated with teasing out the combination of sources, i.e. working out source apportionment from fossil fuels, biomass combustion, marine aerosols, crustal carbonates etc.
- High carbonaceous content in APM at high PM_{10} concentrations are mainly due to combustion products. There is great interest in PM_{10} concentrations due to detrimental effects on health
- The determination of total carbon in APM samples is important for mass closure in source apportionment studies - traditionally this was done by Thermal Optical Reflectance (TOR), however there is no analytical capability for this in NZ. Samples for TOR analysis have to be sent offshore to US & Australia
- Determining total carbon now by isotope ratio mass spectrometry (IRMS) and finding this correlates well to total carbon measured by TOR
- More work is required to accurately establish point source signatures (source apportionment studies) - $\delta^{13}C$ signatures are most effective at separating out sources. Lab is currently focussing on establishing difference between source and ambient samples
- N, O and S isotopes are also proving useful in source apportionment studies.

Karyne Rogers **From food to fuel: an overview of some current isotope research topics at GNS Science**

- Food authentication using $\delta^{15}N$ signals enables discrimination between synthetic fertilisers and organic manure. These $\delta^{15}N$ signals can also detect hot house effects, i.e. vegetables which are grown at a faster rate than using conventional gardening techniques
- Environmental tracers – tracing of nitrates in drinking water in Reunion. Measurement of nitrate concentrations alone gives no indication of source of nitrates. Using a dual isotope approach, measuring $\delta^{15}N$ and $\delta^{18}O$, facilitates discrimination between different nitrate sources, i.e. atmospheric nitrate deposition, nitrate in fertilisers, ammonium in fertiliser and rain, soil nitrate, and nitrate in sewerage and manure
- Coastal & estuarine food web studies: feeding ecology and ontogenetic diet shifts of Yellowstripe Goatfish in Reunion using $\delta^{15}N$ and $\delta^{13}C$
- Determining effects of urbanization and agriculture in Porirua Harbour and Pauatahanui Inlet using $\delta^{13}C$. How is changing land use affecting biodiversity in the inlet?
- Compound specific isotope (CSI) analysis of NZ oils in different oil wells: $\delta^{13}C$ and δD study of source oil rocks enables differentiation between marine and terrestrial oils ($\delta^{13}C$) and oils of different ages (δD).

Troy Baisden **The isotopic indicators of "Land-to-Water Nitrogen Transfers" programme**

- Troy runs the Terrestrial Isotope Biogeochemistry Lab at GNS
- Land-to-water nitrogen transfers programme is a $1M FRST-funded programme running over 3 years: GNS is collaborating with Lincoln & Waikato Universities, AgResearch and Environment Canada
- Developing isotopic indicators: determining $\delta^{15}N$ of soil organic matter; $\delta^{15}N$ and $\delta^{18}O$ changes during denitrification; and catchment applications
- Developing an isotope system/database for NZ
- Project is focussed on pastoral environment
- $\delta^{18}O$ in nitrates determines nitrate sources, whilst $\delta^{15}N$ values indicate degree of nitrification
- Changes in $\delta^{15}N$ soil values observed over time (1950-2007), with an increase in $\delta^{15}N$ reflecting land use changes from developing to highly developed farming practices
- Project has involved development of methods to analyse $\delta^{15}N$ in nitrates. Method used by GNS is to measure isotopic signature of N_2O after nitrifying sample using bacteria
- Currently developing protocols and standardisation across labs and ensuring linearity corrections are being carried out.

Andy Phillips **Future developments at GNS NIC**

- Include compound specific stable isotope analysis (CSIA) for D/H and N (organics) with separation and identifications of compounds on line
- $\delta^{15}N$ and $\delta^{18}O$ analysis of nitrates (see Troy's presentation)
- Automated simultaneous $\delta^{13}C$ measurements of CO_2 collected during ^{14}C analysis
- Zero blank autosampler
- High temperature combustion of inorganic minerals for O and D/H
- Pyrolysis of organics with high N content affects the shape & magnitude of the CO_2 isotope peak, therefore samples require dilution prior to mass spectrometer (MS) analysis. It is important to eliminate moisture and exchangeable H for organic samples. Len Wassenaar is advising on system development.

1.2 Victoria University, School of Geography, Environment & Earth Sciences

Joel Baker **Analysis and applications of non-traditional stable isotopes**

[No electronic presentation]
(Matthew Leybourne presented on behalf of Joel)

Expertise & Analytical Capabilities

- Joel's research interests include early solar system chronology and cosmochemistry, mantle and volcanic geochemistry, environmental chemistry, palaeoceanography and climatology
- The analytical facility has a Nu plasma multiple-collector inductively coupled mass spectrometer (ICPMS) and New Wave deep UV laser facility for analysis of trace elements in solutions (ICPMS) and in situ in solid samples (UV laser). There is also a wet chemistry lab and an ultra-clean chemical separation lab, comprising 3 clean rooms & four pico-trace high quality Class 10 laminar flow workstations (e.g., for Pb isotopes). Richard Wysoczanski is the laboratory manager.
- ICPMS and UV laser are capable of high-precision isotopic analysis of most elements in the Periodic Table. The instruments are very high resolution, have no problems with interference and can easily resolve different peaks
- Quadrupole ICPMS has a collision cell which allows for the measurement of a wider range of compounds at very low detection limits (e.g. can measure the 4 iron isotopes in seawater at parts per thousand (ppt) concentrations)
- Example measurements include: Mg isotopes in mantle rocks; Cu, Ni, Zn in meteorites; Sr, Nd, Pb, Hf, V, Si, Li, B in mantle rocks, seawater & forams
- Other measurements: CO_2 isotopes for palaeoclimate work; low level Ca and Na measurements in ice-cores; isotopologues of CO_2.

1.3 Canterbury University, Department of Geological Sciences

Travis Horton **University of Canterbury's new stable isotope analytical facility**

[P2_University of Canterbury_Horton.pdf]

Expertise & Analytical Capabilities

- Travis is an environmental chemist and has a background in geochemistry, river water chemistry, and whole rock weathering rates. He did a Masters in crustal fluid flow where he analysed $\delta^{18}O$ in calcite and quartz veins to investigate the migration of fluids, then went on to do a PhD at Stanford on plateau uplift history. Although based in a geological department he has more recently moved into studying biological systems, e.g. looking at the fate and transport of mercury in polar bears in high latitude ecosystems (looking at Hg concentrations versus $\delta^{15}N$ and $\delta^{13}C$ values) and investigating palaeotopography using isotopes
- The analytical laboratory was set up and equipment commissioned in June 2008, comprising:

  Thermo Delta V plus – continuous flow (CF) mode only, with 3 peripherals:

  - Thermo gas bench II (with carbonate option) – analysing carbonates, waters, O_2 in air (O_2 isotope discrimination in photosynthesis)
  - Thermo TC/EA (with zeroblank autosampler (AS)) + conflo III. This system has a pyrolysis unit and can autosample for solids & liquids – analysing O_2 and H in hydrous minerals
  - Costech ECS 4010 (with zeroblank AS) + conflo III – an elemental combustion system. It is possible to put a S kit into this, but it is not currently set up for S.

Applications

- Palaeoclimate studies - analysis of carbonates, currently analysing hydrothermal calcite from Marlborough fault system and middle Miocene lacrustrine laminated carbonates
- Biomineralogy research - palaeostratigraphy and topography of southern alps
- Palaeoenvironmental studies using kiwi egg shells (Operation Nest Egg) to look at climate variability. There is a need to first establish NZ proxies. Can we use egg shells as a proxy for palaeoprecipitation? Chironomid head capsules have been successfully used by Matt Wooller, John Gray and others in palaeoenvironmental research: $\delta^{18}O$ of chitin is a good proxy of lake surface water temperatures and can be linked to palaeoprecipitation. Would this approach work in NZ?
- Engineering geology: using vein calcites as a means of investigating meteoric/surface/ground waters Travis is looking at ground water recharge history. Within these ground water studies the lab has been investigating altitude effects on isotopic composition of precipitation using δD and $\delta^{18}O$, in particular investigating isotopic relationships under different atmospheric circulation patterns and lapse rates. Travis has arc mapped 40,000 δD relationships from

North American systems, which he plans to compare to NZ systems, asking the question: how do these isotopic precipitation maps change with elevation?
- Current Marsden-funded project investigating plant respiration using $\delta^{18}O$ discrimination.

Future Research Plans

- Geothermal/magmatic fluids: analysis of $\delta^{13}C$ in CO_2 from geothermal gas vents and hydrous minerals associated with N island magnetism
- Fluid inclusions & mineralization research: development of fluid inclusion isotopic analyses through utilization of an *Amsterdam Device* (crushing device comprising a Swagelok valve, a specialised base plate and additional hardened plates into which you put calcite crystals and squeeze out gases in inclusions). The device can also be utilised for crushing speleothems. This system facilitates the analysis of $\delta^{18}O$ and $\delta^{13}C$ in carbonates and δD and $\delta^{18}O$ in fluids (by employing a magnetic jump during TCA analysis it is possible to also get the O signal as well).
- Developing the methodology for carrying out $\delta^{15}N$ analysis of nitrates for nitrate tracing research. Lab is working on the development of both chemical and bacterial (i.e. denitrifier) methods required for oxygen isotope analysis of nitrate and has planned collaboration with Troy Baisden and Tim Clough
- Development and establishment of NZ-specific proxies.

Limitations of facility

- No dual inlet MS capability
- No offline preparation systems for gas samples
- No S capability on EA
- No GC peripheral
- Time!

Gaps in New Zealand

- No facilities in NZ for studying multiply substituted isotopologues (MSI) – also known as “clumped” isotope analysis. No laboratory in New Zealand is capable of MSI analysis (at present, only a couple of labs globally are set-up to do this: Caltech and Harvard in USA). Need a top-of-the-line instrument (e.g. Thermo Finnigan MAT253) with additional cups in the detector/collector (e.g. two universal triple collectors), providing extreme sensitivity across a range of masses. This has huge potential for palaeothermometer applications. Joel Baker is also interested in this methodology.
- Tuneable Diode Laser (TDL) facilities – Los Gatos Research makes these for rapid and inexpensive hydrogen and oxygen isotope analyses on water samples (there are other applications as well). New Zealand is currently lacking a detailed high spatial and temporal resolution isotopic map for meteoric water (à la Irving Friedman’s map of the western U.S.; U.S.G.S. OFR-2000-388). This new technology makes construction of such a map affordable and easier to achieve – although data needs to be collected regularly over decades to capture the effects of El Niño-Southern Oscillation (ENSO) & Pacific Decadal Oscillation (PDO) etc. Costs would easily be two orders of magnitude lower than current GNS rates over the life time of the device (i.e. thousands of water analyses for both oxygen and hydrogen). *[Post meeting note: TDL equipment is currently available at NIWA for*

N_2O flux measurements, and at Landcare is set up to measure gas concentrations of CO_2 as isotopologues $^{12}C^{16}O^{16}O$, $^{13}C^{16}O^{16}O$, $^{12}C^{16}O^{18}O$ (not isotope ratios) in ambient air].

- Mass Independent Fraction (MIF): no lab in NZ is pursuing mass independent fractionation processes. To do this you need a high-end instrument. None of the isotope ratio mass spectrometers in NZ satisfy this requirement for MIF
- Under-utilisation of compound specific isotope (CSI) techniques. *[Post-meeting note: Russell Frew's lab is set up to do CSI analysis of fatty and amino acids].*
- Meteoric water database (as per Friedman, USGS), in particular using new field-based MS techniques.

[Post-meeting note: GNS have been collecting data and compiling a δD and $\delta^{18}O$ precipitation and hydrology database for tens of years, but there are existing spatial and temporal gaps in the database and issues with data matching. A MOU has now been drawn up between GNS (Troy Baisden) and NIWA (Max Gibbs) to work together towards generating a meteoric water database for NZ. NIWA has just purchased a Picarro Wavelength-Scanned Cavity Ring Down Spectroscopy (WS-CRDS), a real-time portable field instrument, which will facilitate this process.]

Wish List

- TDL
- Compound specific GC
- Thermo Finnigan MAT 253 for clumped isotope work
- Support for more technical staff

Issues/Questions/Discussion

- There are issues with global diminishing supply of ultra-pure He supply as a carrier for MS. Is it possible to re-cycle He gas? Are there any other options, e.g. Ar? What will happen to continuous flow systems when He finally runs out? Community thought that there would have to be rapid developments by manufacturers to modify instrumentation to resolve this issue.
- There is a big discrepancy in cost of He carrier gas from different gas suppliers: Southern Gas on the South Island charges $150 per gas bottle of ultra high purity helium, compared to $800-900 for similar quality He provided by BOC. Should we as a community ship He gas up from the South Island from Southern Gas suppliers to save on running costs for carrier gas?
- Nitrate methods need to be discussed within the SI community. Karyne Rogers suggested having a dissolved nitrate $\delta^{15}N$ methods meeting
- Heinrich asked how much sample volume would be needed to analyse O_2 isotopes in air? Answer: a 20 µl sample loop is used for the analysis.
- Karyne pointed out that GNS has a crude Meteoric water database collated by Mike Stewart.

1.4 Isotrace & Otago University, Chemistry Department

Robert Van Hale **(1) Isotrace Analytical Capability**

[P3_Isotrace_VanHale 1.pdf]

Expertise & Analytical Capabilities

- Director of Isotrace: Russell Frew
- Lab Manager of Isotrace: Robert Van Hale
- Isotrace has 7 mass spectrometers comprising 5 continuous flow (CF) IRMS & 2 dual inlet:
  - Thermo Delta V Advantage CF (with H collectors) + thermal conversion elemental analyser (TCEA): analyses $\delta^{18}O$, δD in solids, liquids & water
  - Thermo Delta plus XP CF (with H collectors) + gas chromatograph-capillary-isotope ratio mass spectrometer (GC-C-IRMS): analyses δD, $\delta^{13}C$ ($\delta^{18}O$, $\delta^{15}N$) of all compounds. (Isotrace have the capability to analyse $\delta^{18}O$, $\delta^{15}N$, but this service is not yet up and running). The system will analyse compound specific isotopes (CSI) in hydrocarbons, lipids, fatty acids as methyl esters (FAMES) & resin acids. It also has the potential to analyse amino acids & polyaromatic hydrocarbons (PAH), although Isotrace has no experience in these analyses yet
  - Thermo Delta V Advantage CF + Thermo Gas Bench: analyses $\delta^{18}O$, $\delta^{13}C$, $\delta^{15}N$ in gas, liquids & carbonate solids
  - Micromass IsoPrime + EuroVector Elemental Analyser (EA): analyses $\delta^{13}C$, $\delta^{15}N$, $\delta^{34}S$ in bulk solids & liquids
  - Europa Scientific 20/20 CF + Carlo Erba EA: analyses $\delta^{13}C$, $\delta^{15}N$ in bulk solids, liquids & gases
  - VG Sira 10, dual inlet, 10 port + CO_2 equilibrium manifold: analyses $\delta^{13}C$ in CO_2
  - VG Sira 12, twin analysers, dual inlet, 20 port: in storage.

Auxiliaries

- Thermo Precon
- Thermo automated cold/chemical traps
- Zeroblank autosampler for solid combustions
- Gas handling rig for giggenbach bottles/gas bags etc. enabling repeated injections of sample
- Clean sampling containers – (Bruce Christensen development) with injections into autosampler
- CTC autosamplers (robotic)
- Water CO_2 equilibration manifold for water analysis, which has the capacity for handling 48 sample at a time
- Hydrogen equilibration rig for analysis of bound H in polymers.

Applications

- High-precision water isotope measurements: hydrology, oceanography
- $\delta^{18}O$ of bone phosphate: archaeology
- Doubly-labelled water experiments: energy expenditure, body water volume
- Breath CO_2: human metabolism, sports performance
- Compound-specific isotope analysis of C & H: food web analysis, pollution tracking, sedimentary inputs. N and O analysis not yet up and running
- Dissolved gases, e.g. dissolved N_2 for denitrification studies

Gaps

- Analysis of low concentrations of C and N in mineral phases – geological N (10 ppm limit of detection)
- Dissolved trace gases
- Isotopic signatures of reactive biogenic gases of CH_4, CO, O_2, NH_3, which are all (with the exception of O_2) present in seawater at very low concentrations. The system required is a membrane inlet IRMS (MIMS).

[*Post-meeting note: NIWA has recently purchased a MIMS system, which is configured to analyse gas concentrations, but has not yet been developed or utilised to measure isotopic ratios.*]

The Future

- Miniaturisation of C and N analysis – static vacuum IRMS? – laser ablation?
- Infra-red analysers
- Post-helium economy – viscous flow under vacuum.

Robert Van Hale **(2) H isotopes of organic material**

[P4_Isotrace_VanHale 2.pdf]

Problems

- 5-20% of H atoms in organic material are exchangeable
- High fractionation effects during analysis are an issue, with up to 80 ‰ during exchange processes
- Fast exchange with water vapour
- 25‰ range in raw δD for same samples.

Solution: Measure non-exchangeable hydrogen

- Add water of known δD content in excess, combust organic materials over Cr metal powder and convert R-H to H_2 (g) with expected yields and isotopic separation as postulated
- Derive plot to determine fraction of exchangeable H: the amount of exchangeable H in the sample is indicated by the slope of the relation between δ_{Water} and δ_{Total}
- Once you know the fraction of exchangeable hydrogen, then it is possible to solve the mass balance equation to obtain δD_n – the isotope ratio of the non-exchangeable hydrogen
- The raw dD then needs to be corrected using standardised materials. This calibration step is based on a large number of standards run at known

concentrations using standards that are chemically identical to the materials being measured.

Russell Frew **Isotrace Research: current applications**

[P5_Isotrace_Frew.pdf]

Isotopic Precipitation Database

- Isotrace have initiated a programme in collaboration with GNS to set up a NZ precipitation database
- GNS started work on this in 1970's, but there is an obvious need to infill spatial and temporal gaps in the existing database
- The aim of the programme is to provide detailed isotopic composition of NZ rainfall varying in time and space
- The ultimate goal is to produce a predictive model
- An agreement is in place with CSIRO to utilise Australian data
- Sampling commenced in Dec 2006 at 49 sites across NZ
- Data from rainwater collection stations are sent to IAEA and will be in the public domain.

Analysis of Organic Compounds

- Understanding sources & processes are key elements when interpreting stable isotope analyses of organic compounds
- Organic compounds in an aqueous matrix are traditionally prepared by solvent extraction
- Solid phase extraction (SPE) reduces preparation time, eliminates solvents and increases extraction efficiency and reproducibility. It results in good separation, and is cheaper and easier to use
- BUT there is an issue of isotopic fractionation with SPE method.

Methamphetamine Profiling

- **Issue:** Differentiation of batches of illicitly manufactured methamphetamine or "P"
- **Background:** Each "cook" will have a different "kitchen" and "ingredients" leading to different chemical fingerprints
- **Approach:** Isolate methamphetamine from as many samples as possible and compare the isotope profiles of C. N, H across batches. This can then be combined with synthetic approach to determine the causes of fractionation.

Wine Adulteration

- **Issue:** Suspected adulteration of Rosé wines with glycerol (artificial sweetener)
- **Background:** There are isotopic difference in plant sugar carbon signals compared to synthetic glycerol carbon signals
- **Approach:** Attempt to differentiate the isotopic signatures of alcohol and glycerol components of wine (should be very similar in genuine produce). Analysing trace element concentrations in wines also assists in differentiating variety and source of grapes used.

Forensics: Physical Evidence Comparison

- **Issue:** Linking physical evidence at scene of crime (twine) to material at suspects address
- **Background:** Manufacturing process of batches of the same product will impart measurable isotopic differences
- **Approach:** Profile a variety of twines to establish the likelihood that twine from both locations are dissimilar from randomly selected materials.

Point source contamination in aquatic environments (Jessica North)

- **2 general applications:**
  - Determining sources of contaminants in aquatic system using δD, $\delta^{18}O$, and $\delta^{13}C$ in meteoric water and leachates. The process of methanogenesis produces enriched δD and $\delta^{18}O$ signals. It is possible to use the relationship of $\delta^{13}C$-DIC versus 1/DIC concentration to identify leachate-contaminated waters
  - Determining contaminant assimilation in an aquatic system. This can be achieved using an environmental indicator species.

Compound-Specific IRMS

- Fatty acids - food web applications. Rebecca McLeod has a 3yr FRST postdoc studying energy sources and food webs associated with Hagfish in Doubtful Sound. Hagfish exhibit a large isotopic range in their bulk C, N and S isotopes indicating terrestrial to marine signatures. S isotopes indicate that chemoautotrophy is an important energy pathway. CSI data further support the theory that terrestrial food sources are an important dietary component. The fiord system is fuelled by terrestrial decaying organic matter entering the fjords. The depleted C isotope signature in Hagfish fatty acids (determined using CSI analysis) also reflects those found in small clams, implying a marine dietary component of some Hagfish.
- Amino acids: applications in nitrogen fixation studies
- Aliphatic and Aromatic hydrocarbons: applications in oil spill fingerprinting.

Point of Origin of Food and Fibre

- Isotrace is developing tools for the verification of authenticity of food products. They combine isotope signatures with heavy and trace elements concentrations to identify points of origin: light isotopes provide information on regional area, whereas trace elements pinpoint specific areas within that region. They use the inductively couple plasma mass spectrometer (ICP-MS) multi-collector, located in Centre for Trace Element Analysis at Otago University as the tracer tool.
- The Global traceability forum www.gotrace.com will promote awareness in this field at the meeting to be held in Wellington in June 2009. [*Post meeting note: the Gotrace Conference was held at Te Papa on Thursday 2nd July, 2009*].

1.5 Lincoln University, Bioprotection Research Centre & Ecology Division

Peter Holder **Stable isotopes and trace elements as New Zealand geo-location markers for biosecurity**

[P6_Lincoln_Holder.pdf]

Expertise

- Peter Holder worked as an entomologist for MAF before taking up a PhD with Lincoln University, collaborating with Otago University (Centre for Trace Element Analysis) for trace element analysis and Victoria University (Joel Baker) for heavy element stable isotope ratios.

Research aims

- To critically examine the validity of using hydrogen isotope ratios (and $\delta^{13}C$) to determine the point of origin of insect pests of biosecurity importance to New Zealand.
- To test the feasibility and benefits of using heavy element stable isotope (Pb, Sr, Ir, Nd) and trace element analyses for insect geo-location.

Background

- NZ$ 6.7m was spent on the eradication of fall webworm and NZ$ 62 m on the painted apple moth campaign. Despite these huge sums of money being spent on eradication programmes there have been post-eradication detections
- Were these detected individuals new independent incursions or residual individuals of the targeted populations? Isotopic signatures, δD and $\delta^{13}C$ may be able to provide the answers.

Methodology Issues

- Terrestrial δD and $\delta^{13}C$ isoscapes in USA have been able to provide a mapped isotopic terrain which has been utilized to track the migration pathways of monarch butterflies (Wassenaar & Hobson, 1998). Is this method applicable outside of a continental situation? The accuracy and applicability of this method in New Zealand is unknown. We have a long thin maritime country. Will natural δD variations here be too great to be able to use this method?
- Other issues are that biosecurity officials often only get hold of 1-2 samples of invasive species, so sufficient sample analyses are often problematic
- Insects may feed on multiple sources of plants, which is especially true for introduced insects, and often we don't know what the primary host plant is. There is often large host-to-insect isotopic variation (+/- 5‰ in $\delta^{13}C$), ecological complexity complicates distributions, and isotopic signatures are distorted by the nutritional quality of food. All this suggests that δD may be a better discriminant.
- However, there are also numerous problems with establishing δD isoscapes, mainly as a result of environmental water variation and photosynthetic variation producing lots of noise in environmental data. In δD precipitation maps, for example, in Auckland's late summer 2007 there was a +7 to -52 ‰ seasonal

rainwater range. Furthermore, the large and variable δD fractionations during photosynthesis, are hypothesised to add lots of variability from plant to insect.

- There is an untested assumption in plants that the δD signal is averaged out
- There is also an assumption that insect isotopic signatures will reflect the carbohydrate in host plant, however, insects more closely resemble the isotopic signature of the leaf water in the plant, which can have a large range in δD. Both assumptions are currently being tested.

- Using other elements in conjunction with δD and $\delta^{13}C$ values increases the chances of establishing point of origin discrimination, which is why Peter has been using trace element concentrations (e.g. Cd and Se), and 'heavy' element isotopes Pb, Sr; Ir, Nd. The advantage of using these elements is that fractionation of heavy elements is small.

1.6 AgResearch, Grasslands Research Centre, Palmerston North

Mike Tavendale **AgResearch Stable Isotope Analytical Facilities**

[No electronic presentation]

Expertise & Analytical Capabilities

- Mike is an organic chemist specialising in nutrition
- AgResearch Lab has a ThermoFinnigan Delta V plus with a GC linked to high temperature pyrolysis combustion system. Can analyse δD, $\delta^{18}O$, $\delta^{13}C$, $\delta^{15}N$ in volatile organic materials.

Applications

- Nutritional studies – what happens to fibre when digested in hind gut of animals, including humans?
- AgResearch has been investigating the dominant processes in the hind gut of people with irritable bowel syndrome. They have established that the microbial digestion of fibre components is different from that of a normal digestive system
- Manipulation experiments on rats using biomarkers are also providing insight into hind-gut disease. ^{13}C-enriched fibre is fed to rats to quantify the degradation of fibre in the hind gut. Breath $^{13}CO_2$ (a biomarker of hind gut fermentation processes) & the presence of labelled volatile fatty acids in the hind gut of the rat (i.e. the amount of ^{13}C associated with acetate) gives an indication of how much fibre has been degraded in the hind gut. These two measurements are proving to be very useful biomarkers of hind gut disease.
- A collaboration between AgReseach and Massey vet school is studying protein turnover in animals. They feed animals ^{13}C-labelled protein and trace this through to the animal's milk. Essential and non-essential labelled amino acids in the milk are analysed to establish if there is a net loss of protein in milk, representing a deficiency in the diet. This enables determination of whether an animal would be successful for milk production or not.
- Using CSI to study fermentation processes in rumen (first four chambers of fore-stomach) of ruminants (e.g. cattle, sheep, deer). Several processes lead to the formation of acetic acid, but it is not known where it forms. AgResearch is trying to establish how important certain microbial processes are in the rumen.

1.7 Isolytix Ltd., Dunedin

Ken Neal **Isolytix Analytical Services**

[P7_Isolytix Ltd_Neal.pdf]
(Sarah Bury presented on behalf of Ken)

Expertise & Analytical Capabilities

- Ken has set up a company in Dunedin (Isolytix Ltd.) operating as the sole NZ and Australian agent for Iso Analytical, based in Australia
- The Australian Iso Analytical laboratory has sample preparation laboratories (vacuum, distillation lines etc.) and 5 continuous flow Europa Scientific mass spectrometers linked to:
    - Europa Scientific Roboprep and Sercon elemental analysers: analysing $\delta^{13}C$, $\delta^{15}N$ & $\delta^{34}S$ in bulk solid organics
    - Europa Scientific ANCA GSL EA: analysing $\delta^{18}O$ & δD in organic solids & liquids
    - Europa Scientific ANCA-G EA + Gilson 222 Autosampler: analysing $\delta^{13}C$ & ^{8}O in carbonates
    - Europa Scientific ANCA-GSL Gilson 222 Autosampler: analysing $\delta^{18}O$ & δD in liquids by equilibration
    - HP5890 + proprietary GC-C combustion interface: analysing C1-C4 hydrocarbons and CO_2

Next year Iso Analytical plan to commission a hybrid compound specific IRMS system linked to a scanning mass spectrometer for simultaneous IRMS identification work.

1.8 Lincoln University, Agriculture & Life Sciences Division

Tim Clough **Lincoln University Stable Isotope Analytical Facilities**

[P8_Lincoln_Clough.pdf]
(Sarah Bury presented on behalf of Tim)

Expertise & Analytical Capabilities

- Staff: Tim Clough, Roger Cresswell
- PDZ Europa (Sercon) continuous flow mass spectrometer with triple collector, plus collectors for H isotopes with the following peripherals:
  - PDZ Europa (Sercon) elemental analyser: analysing $\delta^{15}N$ & $\delta^{13}C$ of solid organic materials, e.g. soil, plants etc.
  - Trace gas interface with cryogenic trapping and focusing and a pyrolysis furnace: analysing $\delta^{13}C$ in CH_4
  - Gilson autosampler (standard liquid autosampler adapted) & PDZ Europa (Sercon) trace gas unit. The introduction of gas samples is usually via 12 ml exetainers using the Gilson autosampler, which has had the liquid sampler removed and replaced with a double concentric needle which can take 125 and 250 ml bottles: analysing $\delta^{15}N$, $\delta^{13}C$, & $\delta^{18}O$ of N_2O, N_2, CO_2 and CH_4
  - Software is versatile and allows for customization of the method e.g. enables isotopomer analysis of N_2O.

Applications

- Climate change, i.e. effects of pasture soil warming on net greenhouse gas fluxes (^{15}N, ^{13}C)
- Fate of plant litter in pastoral soils (^{15}N)
- Effects of biochar (charcoal created by biomass pyrolysis) on the N cycle (^{15}N, ^{13}C) *
- Fluxes of N_2O from river systems (^{15}N, ^{18}O) *
- Fate of nitrate in the vadose zone (unsaturated zone above area of groundwater flow, i.e. between ground surface and water table) (^{15}N, ^{18}O) *
- Identifying nitrate sources and fractions in a grazed catchment (^{15}N, ^{18}O).

* collaboration with Troy Baisden, GNS

Future/Wish List

- Replace current machine which is now 10 years old
- GC-MS capacity for organic compounds
- A multiple collector instrument which would make isotopomer analyses easier.

1.9 Cawthron Institute

Chris Cornelisen & Reid Forrest **Applications and future directions of stable isotope research at Cawthron Institute**

[P9_Cawthron_Cornelisne&Forrest.pdf]

Organisation & Expertise

- Cawthron is a not-for-profit science organization owned by the Cawthron Trust Board. The Institute is based in Nelson and Marlborough and employs 180 people
- Provides research, advice and analytical services to support the development of New Zealand's seafood industry and sustainable management of the coastal and freshwater environment (e.g. aquaculture, biosecurity, business sustainability, food testing)
- There are no stable isotope facilities at Cawthron. Analyses are contracted out to other laboratories
- Cawthron scientists using stable isotopes in their research:
    - Chris Cornelisen (coastal ecosystems, river plumes, macro algae physiology)
    - Reid Forrest (coastal ecosystems)
    - Paul Gillespie (aquatic microbial ecology, river plumes)
    - Barrie Forrest (marine ecology and biosecurity)
    - Roger Young (fish otoliths)
    - Joanne Clapcott (freshwater ecology, nutrient enrichment indicators in streams, trophic level interactions in streams).

Applications

- Contaminant tracking (using molecular markers, trace elements, stable isotopes) of:
    - Sediments
    - Metals
    - Nutrients
    - Pollutants (molecular markers, faecal contamination)
    - Freshwater discharge – study in Doubtful Sound
    - Invasive species
- Food web studies
- Palaeoecology applications – Tasman Bay
- Fish movement.

Research

- Integrated Catchment Management Programme (collaboration between Landcare, NIWA, Cawthron, Tasman District Council) in Motueka River & Tasman Bay. Cawthron are studying the pathway of tracers, following the impact of river plumes & fate of contaminants through to the food web. Forrest et al. (2007) tracked a contaminant plume into Tasman Bay using isotopic terrestrial vs. marine system signals. They also studied macro algae physiology and isotopic signatures in primary producers focussing on the food web ecology of the region.

Issues:

- How do river plumes impact on aquaculture management areas? The plume of tracers comes out into path of the managed area
- High metal contents exist in the hinterland geology. Soils are naturally high in Cr and Ni and as a result, wines from the plains have very high metal content.

- Nutrient transport and fate in benthic systems, in particular studying the effect of hydrodynamics on benthic processes. The main focus is a study of the effects of turbulence on the uptake of ammonium and nitrate in sea grasses using ^{15}N-enriched experiments. Ammonium uptake was found to be greatest under low flow regimes and leaves covered in epiphytes had lower ammonium uptake than those without epiphytes. These patterns were not observed for nitrate, since nitrate uptake is an active process not a passive diffusion process like ammonium uptake.
- Algal physiology studies in Fiordland collaborating with Otago University to determine what drives food web variability in Fiordland? This area is a very physically dynamic environment with great variability at base of food web. Cawthron have been studying the effect of physical processes (light & turbulence) on the uptake of $\delta^{13}C$ and $\delta^{15}N$ in macro algae.

1.10 Landcare Research Ltd., Lincoln

John Hunt & Margaret Barbour **Stable isotope tuneable diode laser facility at Landcare Research**

[P10_Landcare_Hunt&Barbour.pdf]

Expertise & Analytical Capabilities

- Campbell Scientific Instruments Trace Gas Analyser TGA100A Tuneable diode laser (TDL) and absorption spectroscopy
- The TDL has 16 inlets (35 sec/inlet), is field portable and provides instant online delta ratios
- It measures gas concentrations of CO_2 as isotopologues $^{12}C^{16}O^{16}O$, $^{13}C^{16}O^{16}O$, $^{12}C^{16}O^{18}O$ (not isotope ratios) in ambient air

Applications

- Greenhouse gas based research: determination of sources, sinks, & controlling processes, such as eddy covariance and gradient flux measurements
- Plant ecophysiology: below ground and above ground processes
- Investigation and measurement of processes ranging between sub-cellular, leaf and ecosystem levels
- Biological and environmental interactions: following isotopes through ecosystems using natural or imposed perturbations (enrichment studies).

Tuneable diode laser for SI analysis

- The TDL used at Landcare is the only one in the southern hemisphere measuring isotopologues of CO_2, i.e. $^{12}C^{16}O^{16}O$, $^{13}C^{16}O^{16}O$, $^{12}C^{16}O^{18}O$. In air, ambient concentrations are 380, 3 and 1ppm respectively
- The system is portable and is used for eddy co-variance and gradient flux measurements
- Measurements are concentrations of isotopologues, not isotope ratios. It is necessary to use on-line equations to calculate isotope ratios based on these concentrations.

Calibration and precision

- A small offset error is caused by optical interference
- This is not important for gradient measurements, but it is necessary to carry out this offset error correction for isotope measurements
- The offset error changes slowly over time, so accuracy depends on the calibration interval
- Landcare carry out a 2 point calibration using NIWA analysed standards (350 and 500 ppm CO_2 in N, O & Ar).
- Gordon Brailsford measures $\delta^{13}C$ and $\delta^{18}O$ ratio plus [CO_2] on the mass spectrometer and Landcare then convert this to molar concentrations.
- Precision for 15 x 1s measurements for $\delta^{13}C = 0.1$ ‰ & for $\delta^{18}O = 0.2$ ‰.

Advantages of TDL

- On-line instant delta ratios
- Can measure water vapour isotopes as well as gas CO_2 isotopes
- Can measure 16 inlets (35s/inlet) or sample bags (but not vials)
- High temporal resolution
- Liquid N_2 + calibration cylinders are the only consumables required
- Insulated and temperature controlled
- Doesn't need a dedicated technician
- Can be linked with other measurement systems
- Field portable, inlets can be 200 m away.

Disadvantages of TDL

- Sensitive to vibration
- Non-linear response over wide concentrations - need to calibrate over range of interest
- Short interval between standards – depends on application
- High cost $NZ150,000.

John Hunt **Understanding below-ground carbon dynamics using stable C isotopes**

- Marsden funded project with David Whitehead, Pete Millard and Andy Midwood, Macaulay Institute, Aberdeen
- A PhD student (Yoshi Uchida) with Lincoln University is studying the effects of soil temperature on below-ground partitioning of soil respiration: how does soil temp affect soil respiration? Yoshi is asking the following questions:
  - Where is the C coming from?
  - How is old and new soil C partitioned during soil respiration?
  - What is proportion of old and new respired C?
  - What proportion of respiration comes from roots and soil?
  - What is the respiration response to rain?

Margaret Barbour **Applications of stable C and O isotopes in plant ecophysiology**

- Margaret is a plant physiologist with a research focus on carbon and water dynamics in plants
- She uses leaf chambers to modify key photosynthetic factors (e.g. humidity, light, temperature) and measures the $\delta^{13}C$ and $\delta^{18}O$ discrimination during water exchange between the plants and atmosphere
- These water exchange processes are related to the degree of stomatal opening and to make these measurements you need to measure the rate of CO_2 movement from inside the stomata cavity into the chloroplasts
- TDL is attached to leaf chamber and sampling takes place at the stomatal inlet and outlet enabling CO_2 concentrations across the plant to be sampled.

1.11 Waikato University, Department of Biological Sciences

Brendan Hicks **(1) Waikato University Stable Isotope Unit (WSIU)**

[P11_Waikato_Hicks1.pdf]
(Max Gibbs presented on behalf of Brendan)

Expertise & Analytical Capabilities

Waikato stable isotope unit (WSIU)

- WSIU was established in 1991 by Warwick Silvester
- Facility Director: Brendan Hicks; Manager: Anjana Rajendram
- Services SI research within university and offers commercial analytical services
- Accredited in 2006 by the International Atomic Energy Agency for analyses of C and N content and $\delta^{13}C$ and $\delta^{15}N$ in plant materials
- 2 continuous-flow stable isotope ratio mass spectrometers:
    - Europa Scientific (Sercon) 20-20 Blue Box IRMS linked to an ANCA-NT GSL (Gas Solids Liquids) preparation unit – analyses $\delta^{13}C$ & $\delta^{15}N$ in solid organic and liquid samples
    - PDZ Europa (Sercon) 20-20 linked to an ANCA-NT GSL (gas solids liquids) preparation unit: analyses $\delta^{13}C$ & $\delta^{15}N$ in gas samples.

University of Waikato, Department of Chemistry and Department of Earth & Ocean Sciences

- Equipment run and operated by Chris Hendy, Steve Cooke and Steve Cameron
- PDZ Europa (Sercon) 20-20 linked to a DI Carbonate Acid Preparation System (CAPS): analyses $\delta^{13}C$ & $\delta^{18}O$ in carbonates (Contact: Chris Hendy; Facility manager: Steve Cooke)
- Perkin Elmer SCIEX Elan DDRC II, Inductively Coupled Mass Spectrometer (ICP-MS) with automatic liquid sampler. The system has Dynamic Reaction Cell Technology (DRCT) to eliminate isobaric interferences, an autosampler for solutions, and High Pressure Liquid Chromatography (HPLC) separation for speciation determination. Analyses major & trace element concentrations and chemical speciation as well as strontium, lead and uranium isotope abundances in water & solid samples (after acid digestion) (Contact: Chris Hendy; Facility manager: Steve Cameron)
- Perkin Elmer SCIEX Elan DDRC II, ICP-MS with automatic liquid sampler New Wave Laser UP 213-A/F Laser Ablation System. The laser ablation enables fine sectioning or sub-sampling of solid samples down to 10nm size with the subsequent analysis of major& trace element concentrations, as well as strontium, lead, uranium isotope abundances (Contact: Chris Hendy; Facility manager: Steve Cameron)
- Helium mass spectrometry analysis comprising home-built helium extraction bench with quadrupole mass spectrometer. This system measures Helium (^4He) abundance in single crystal apatite or zircon by laser heating or multiple apatite furnace heating (Contact: Peter J.J. Kamp; Facility manager: Steve Cooke).

Preparation/Additional Equipment

WSIU

- Retsch MM 2000 ball mill for sample grinding and homogenisation in preparation for stable isotope sample analysis ($\delta^{13}C$, $\delta^{15}N$, C & N concentrations)
- LECO TruSpec CN determinator with coupled autosampler for the analysis of C and N concentrations in solid and liquid samples.

University of Waikato, Department of Chemistry and Department of Earth & Ocean Sciences

- Radiocarbon dating laboratory using Quantulus spectrometers with Accelerator Mass Spectrometry (AMS) graphitisation for analysis of ^{14}C in solid samples (Contact: Alan Hogg).

Applications

- Archaeology
- Geochronology & thermochronology
- Geology
- Biology
- Biogeochemistry
- Environmental studies
- Terrestrial systems
- Riparian studies
- Forestry
- Freshwater ecology
- Food webs
- N Cycling
- Forensics
- Medical (breath-testing) studies

Future

- Vario EL Elemental Analyser to facilitate analysis of Antarctic soils with very low C & N content: 0.08 % C , 0.03% N
- Isoprime stable isotope mass spectrometer.

Wish List

- Perkin Elmer ICP-MS for dedicated laser ablation use for trace metal analysis of otoliths
- Upgrade of aging MS instruments.

Brendan Hicks **(2) Use of laser ablation and ICP-MS to analyse otolith microchemistry: determining the migrations of freshwater fish.**

[P12_Waikato_Hicks2.pdf]
(Max Gibbs presented on behalf of Brendan)

Research

- Does marine-derived N and C from adult salmon subsidise the nutrient needs of juveniles in Alaskan floodplain beaver ponds?

  Coho spawners were shown to enrich the ponds. All food web components in ponds with spawners showed elevated ^{13}C and ^{15}N derived from the growth of adults in the sea (Hicks, B.J., et al. 2005. *Oecologia 144*: 558-569)

- Laser ablation and ICP-MS are being used to analyse otolith microchemistry to determine the migrations of freshwater fish

  Of the 36 species of NZ native fish, 50% migrate to sea at some stage in their life-cycle. They usually spawn in freshwater, spend 3-5 months as juveniles or "whitebait" at sea, then migrate back upstream as adults between 1-10 yrs old. Using otolith banding it is possible to distinguish between periods that fish have spent in salt and freshwater: $^{88}Sr/^{43}Ca$ ratio is high in salt water and low in fresh water. Analysing $^{88}Sr/^{43}Ca$ ratio in Banded Kokopu otoliths enables determination of the water bodies that the fish were in at different stages of their lives.

- Study of the effect that the Lake Rotoiti diversion wall will have on the migration of trout and smelt (trout prey)

  Will the diversion wall affect the rainbow trout production/migration and their food source (smelt) abundance between Lake Rotoiti and Lake Rotorua? Waikato is using a combination of elements (Mn, Zn, Rb, Sr and Ba) to determine the stream origin of the juveniles. The rainbow trout can be identified to their spawning stream with a 70-100% accuracy.

1.12 NIWA Ltd., Wellington

Sarah Bury **(1) Introduction to NIWA's stable isotope analytical facilities**

[P13_NIWA_Bury.pdf]

Expertise & Analytical Capabilities
The recruitment and retention of good technical staff to support and run mass spectrometers is as important as having good analytical equipment. It is important for mangers to acknowledge this when supporting new equipment purchases. Current stable isotope analytical facilities at NIWA include:

ThermoFinnigan Delta^{Plus} Facility
Contact/Manager: Sarah Bury
Additional Staff: Julie Brown, Jan Vorster

- Continuous flow Fisons NA 1500 Elemental Analyser linked to continuous flow Delta^{plus} IRMS, with Conflo III reference gas and diluter open split for analysis of $\delta^{13}C$ & $\delta^{15}N$ in solid organic materials: e.g. flora, fauna, sediment, atmospheric particles.
- We have the capability & expertise to analyse $\delta^{34}S$ in organic material, but due to the current high capacity of $\delta^{13}C$ & $\delta^{15}N$ analyses, restricted machine availability precludes $\delta^{34}S$ analysis. Sulphur analysis is extremely time consuming and results in degradation of carrier lines and equipment resulting in a reluctance to run sulphur as a routine analysis. If we obtain our planned mass spectrometer upgrades we will look at reinstating $\delta^{34}S$ analysis on a dedicated system if demand is sufficient. We need to collaborate and co-ordinate with Isotrace and Isolytix when this issue arises.
- **Supporting Equipment:**
    - DIONEX 200 accelerated solvent extraction system (ASE) (NIWA Hamilton, Organic Chemistry Laboratory) for lipid extraction of solid organic samples for lipid-free SI analysis
- **Applications:** Environmental, oceanic (including sub-tropical, sub-Antarctic, Antarctic), coastal, lacrustrine, riverine, riparian & terrestrial ecological studies, trophic studies, pollution tracking & management, sedimentary pathways & inputs, source apportioning (e.g. aquatic & atmospheric contaminants, dietary studies), marine, freshwater and lacrustrine carbon and nitrogen cycles/fluxes (e.g. process measurements of nitrogen fixation, denitrification, primary production etc.), nutrient supply on offshore islands, terrestrial & marine conservation, aquaculture, animal and plant physiology, animal migrations, fisheries management, tracking of invasive species.

ThermoFinnigan MAT252
Contact/Manager: Gordon Brailsford
Additional Staff: Bill Alan, Keith Lassey, Katja Riedel, Hinrich Schaefer, Peter Franz, Ross Martin, Rowena Moss, Jan Vorster, Tony Bromley

- Thermo Finnigan MAT 252 dual inlet mass spectrometer measuring $\delta^{13}C$ in atmospheric CH_4 as well as $\delta^{18}O$ and $\delta^{13}C$ in atmospheric CO
- GC-IRMS continuous flow linked to MAT 252 measuring $\delta^{13}C$ in atmospheric carbon dioxide.
- **Supporting Equipment:**
    - Gas preparation laboratory with offline vacuum preparation systems for the extraction of CO_2 and CH_4 from low concentration atmospheric samples
    - DIC vacuum line for the extraction of CO_2 from dissolved inorganic carbon in fresh water & seawater
    - Automated air & aeolian dust sampling equipment, including vertical air sampling & meterological profiling with tethersondes and radiosondes
- **Applications:** Agricultural emissions, atmospheric monitoring, climate change, gas emissions.

Kiel III preparation device
Contact/Manager: Helen Neil
Additional Staff: Jan Vorster

- Thermo Finnigan MAT 252 dual inlet mass spectrometer linked to Kiel III preparation device measuring $\delta^{13}C$ & $\delta^{18}O$ in carbonate materials: e.g. foraminifera, bivalves, corals, speleothems, otoliths
- **Supporting Equipment:**
    - Micromill hi-resolution sampling laboratory for fine sampling of carbonate materials, e.g. otoliths, bivalves, corals, speleothems
- **Applications:** Palaeoceanography, palaeoclimate, fish migration, fish age at maturity, fisheries management, bivalve aging & growth studies, terrestrial climate, precipitation, climate change hazard indices.

GVI Isoprime
Staff: Peter Franz, Katja Riedel, Hinrich Schaefer

- GVI Isoprime continuous flow mass spectrometer linked to a methane-to-CO_2-converter peripheral, measuring $\delta^{13}C$ of CH_4 in ice cores & gas seeps
- **Supporting Equipment:**
    - Ice core gas extraction system (ice grater)
- **Applications:** Climate change, methane fluxes.

[*Post-Conference Information*:
Picarro Wavelength-Scanned Cavity Ring Down Spectroscopy *(WS-CRDS)*
Contact/Manager: Max Gibbs (NIWA Hamilton)

- *Funding was approved for the purchase of a WS-CRDS real-time portable field instrument (purchased June 2009) which will measure δD & $\delta^{18}O$ in water & water vapour*
- *Collaboration set up between GNS & NIWA to monitor and analyse δD & $\delta^{18}O$ in water sampled from a network of hydrology stations throughout NZ*
- ***Applications:*** *Generation of water isoscapes (meteoric water line), hydrology, biosecurity*]

Additional equipment

- Membrane Inlet Mass Spectrometry (MIMS) for high resolution simultaneous continuous measurements of major (CO_2, O_2, N_2, Ar) and trace (CH_4, DMS, N_2O, H_2S) gas concentrations in seawater, freshwater and sediment pore waters (Cliff Law)
- Campbell Scientific Instruments Trace Gas Analyser TGA100A TDL (Tuneable diode laser) and absorption spectroscopy for N_2O flux measurements using GC micrometeorological eddy co-variance techniques (Gordon Brailsford, Ross Martin, Andrew Mc.Millan)
- Hewlett Packard 5972 mass spectrometer linked to a 5890 GC for analysis of fatty acid and sterol concentrations in solids and liquids. This system only measures concentrations, not isotope ratios (Matt Nelson & Michael Bruce, NIWA Auckland)
- Series of gas chromatographs (GC's) set up for specific gas concentration measurements:
    - CO_2, N_2O, CH_4, CO, SF_6, O_3, non-methane hydrocarbons (C1-C6), acetylene, H_2, O_2/N_2 ratios, & moisture content (Rowena Moss)
    - seawater concentration measurements of SF_6, N_2O & CH_4 (Cliff Law)
    - seawater and air concentration measurements of dimethylsulphide (DMS) & dimethylsulphoniopropionate (DMSP) (Mike Harvey)
- Condensation nuclei counter (CNC) (Mike Harvey)
- GC-MS, Liquid Chromatography Mass Spectrometry (LC-MS), HPLC, Nuclear Magnetic Resonance (NMR) for measuring organic contaminant concentrations of dichlorodiphenyl-trichloroethane (DDT), PAH & polychlorinated biphenyls (PCB's) (Greg Olsen, Organic Chemistry Laboratory, NIWA Hamilton).

Sarah Bury **(2) Ecological applications of bulk C & N analysis**

[P13_NIWA_Bury.pdf]
(Also presented data on behalf of Fleur Matheson and Cliff Law)

Enrichment Applications

- Nitrogen assimilation by sea anemones (Morar, Bury & Davy et al., submitted)
- Nitrogen transformation studies in wetlands (Matheson et al., 2002). Fleur used ^{15}N labelling experiments to study the influence of vegetation on nitrogen removal in wetlands. Denitrification (i.e. permanent N removal) was enhanced in oxidised wetland plant rhizospheres. Rhizosphere processes are key to sustainable N removal in wetlands
- Algal N fixation (Cliff Law): Cliff uses ^{15}N-N_2 enrichment manipulation experiments to measure the degree of algal N fixation in marine systems. The Tasman Front represents the southern boundary for N fixation, as temperatures south of the front drop below 20ºC. A 9-fold increase in N fixation was measured over a 12 day period. The study revealed N-fixation represented ~35% of new nitrogen, with dust deposition representing about 5% and diapycnal mixing about 60%. This has led to a new nitrogen "layer cake" theory with distinct layers being supplied by different sources of new nitrogen (Law, Boyd, Bury et al.)
- C (photosynthesis) & N (NO_3, NH_4 and urea) phytoplankton uptake rates, measuring new versus regenerated production in Hauraki Gulf (Bury, Zeldis, Gall, Nodder) & Tasman Sea (Bury, Law & Boyd). Studies in Hauraki Gulf have shown this system to be primarily driven by regenerated nutrients despite frequent seasonal upwelling events.

Natural Abundance Applications

Trophic Studies

- Chatham Rise open ocean food web study (Nodder, Thompson & Bury)
- Coastal Antarctic food web study (Norkko, Cummings, Gibbs et al., 2007); IPY Ross Sea voyage (Pinkerton, Cummings, Bury)
- Ross Sea Antarctic Toothfish prey apportionment study & modelling (Bury, Pinkerton, Thompson et al., 2008)
- Ross Sea squid trophic studies (Thompson, Pinkerton, Bury et al., 2008)
- Microbial invertebrate food webs in groundwater ecosystems (Hartland, Fenwick, Bury, submitted)
- C flow and food web structure in rocky reef ecosystems (MacDiarmid, Bury and Beaumont)
- C flow and food web structure in sea grass and mangrove ecosystems (Morrison & Bury)
- Historic dietary changes in Hector's dolphins (Hutchinson, MacDiarmid)
- Historic dietary changes in Little Blue Penguins (Geurts, Brunton, Bury)
- Shortfin eel trophic changes due to macrophyte loss in Lake Ellesmere (Kelly & Jellyman, 2007).

Other Ecological Applications

- Pollution & sediment tracking studies (Gibbs)
- Habitat migration markers of juvenile plaice in Firth of Forth (Augley, Bury et al., 2007)
- Migratory patterns: marine vs. freshwater residence in eels (Jellyman)
- Linkages between stream & terrestrial food webs (Collier, Bury & Gibbs, 2002)
- Survival & dispersal of translocated Stitchbirds to mainland site (Richardson, Castro, Brunton, Armstrong, Bury)
- Foraging ecology of Cook's Petrel (Rayner, Phillips, Bury et al., 2008)
- Causes of decline of Rock Hopper Penguins (Hilton, Thompson, Bury et al., 2006).

David Thompson **Stable isotopes and apex predators**

[P14_NIWA_Thompson.pdf]

- David applies nitrogen & carbon SIA to marine consumers with special emphasis on seabirds & cephalopods.

Rock Hopper Penguins (RHP) case study:

- A global decline has been observed in RHPs. David obtained skins and feathers from museums from 1860 to present and analysed them for N & C isotopes. He observed an overall steady decline in $\delta^{13}C$ of RHP signals over time (1860 to 2000). The $\delta^{13}C$ signature is a valuable proxy for marine productivity which is also mediated by temperature.
- The main question is whether RHP diet has changed over the time period of the decline in population?
- Project sampling strategy: RHP's return to the same breeding site each year. When the chicks have fledged and left the colony at the end of the breeding season the adult birds also leave and feed at sea for a month, returning back to the same breeding colony to moult. They shed their feathers and re-grow new ones without feeding during this time period. Feathers sampled from moulting birds therefore reflect the bird's diet mainly during that premoult exodus from the breeding colony prior to moult. The isotopic signature in these feathers is therefore an excellent indicator of productivity around the breeding grounds where the birds have foraged.
- An isotopic correction needs to be applied to the data for the oceanic Suess effect: anthropogenic CO_2 from fossil fuel burning has a more negative $\delta^{13}C$ value than background atmospheric CO_2. Consequently, there has been an exponential decrease in oceanic $CO_{2[aq]}$ $\delta^{13}C$ over the last 150 yrs. To interpret changes in $\delta^{13}C$ due to productivity changes, you need to correct for the oceanic Suess effect. Isotopic C signals in contemporary RHP feathers will be more negative than feathers grown 150 yrs ago. When these values are corrected for the Suess effect we still see a decline in $\delta^{13}C$ values, confirming a genuine decline in productivity.
- Combing $\delta^{15}N$ data with these corrected $\delta^{13}C$ values, there was no isotopic evidence that RHP's diet had changed, although overall oceanic production seemed to have declined
- RHP are a useful marine indicator (or sentinel) of productivity because they forage relatively close to their breeding sites.

- Sampling RHP's from all breeding sites around the Southern Ocean is providing NIWA with marine productivity isoscapes around those islands with breeding colonies and enabling a Southern Ocean marine isoscape to be derived
- How do birds exploit and use marine resources when not constrained to their breeding site? NIWA is now supporting these SIA with foraging trip tracking data using satellite telemetry tags on the birds and attempting to address this question.

Neill Barr & Bruce Dudley **The use and application of nitrogen stable isotopes in marine macro algae for tracing anthropogenic nitrogen**

[P15_NIWA_Barr&Dudley.pdf]

- Neill and Bruce have been tracing anthropogenic input to the marine environment using the seaweed *Ulva*
- The study site was at the sewage outflow in Titahi Bay, where moorings were put out attaching *Ulva* at the surface and a few metres deep
- The treatment plant removes N from the sewage, which leaves the remaining dissolved N in seawater isotopically heavier. It is therefore possible to use N isotopes to trace the outflow from the sewage plant
- Total inorganic nitrogen (TIN), phosphate and $\delta^{15}N$ in *Ulva* tissues were found to be higher at the surface than at a few metres depth
- Key Questions:
    - Is *Ulva* (compared to other seaweeds) an appropriate indicator of seawater nitrogen sources across depth gradients? Previous studies show large variation of isotopic values can be observed within the same plant
    - How much does light affect $^{14}N/^{15}N$ fractionation in *Ulva* during N-uptake and assimilation?
    - Is $^{14}N/^{15}N$ fractionation in *Ulva* dependant on the nitrogen source?
- Examination of *Ulva* as an indicator of nitrogen loading around NZ coastline: two surveys were carried out in summer & winter 2002, in sites classified as sheltered or exposed rural, sheltered/exposed/or enriched urban sites
- Neill found a very narrow range of $\delta^{15}N$ values (most *Ulva* samples were between 6-8 ‰ reflecting clean coastal water values) regardless of site classification, season (summer/winter) and temp/light regimes, with only a very few contaminated urban sites having enriched $\delta^{15}N$ values of 10-13 ‰. The average of all sites was: summer 7.8 ‰ (± 0.6 s.d.), winter 7.6 ‰ (± 0.6 s.d.)
- These findings led to the question: How well do plants reflect the ambient source pool of N? Enrichment-manipulation ^{15}N studies were then set up to investigate the effect of light on $^{14}N/^{15}N$ fractionation in *Ulva* during N-uptake and assimilation.
- Experimental setup:
    - Removed all ambient N from seawater using a biological scrubber (*Ulva*)
    - Experimental *Ulva* tissue was turned over on a known N source beforehand
    - Water was dosed with known enrichments of labeled nitrate (3.95 ‰) and ammonium (5.5 ‰) & equal molar N concentrations
    - 2 light levels were used: full light, 20% light.
    -

- Results:
  - Under contrasting light conditions the fractionation in *Ulva* ranged from +1.2 to -2.5‰
  - These light conditions are likely to encapsulate most of the light environments in which *Ulva* grow
  - The added nutrient concentration (~10μM) is higher than most coastal environments
  - *Ulva* appears to give a good representation of water column $\delta^{15}N$ signatures.

Gordon Brailsford **Atmospheric stable isotope analytical facility and applications**

[P16_NIWA_Brailsford.pdf]

Thermo Finnigan MAT 252 dual inlet mass spectrometer: measurement of concentrations and isotopic ratios of methane (CH_4) and carbon monoxide (CO)

- Trace gases are oxidised to CO_2 offline and isotopic ratios of CH_4 and CO are determined versus a reference gas
- The lab has been measuring seasonal cycles of methane from 1987 to 2007 (Brailsford, Martin, Moss, Allen)
- A seasonal amplitude of ~25-30 ppb, and 0.2 ‰ has been measured
- There are processing and measurement uncertainties of 3.4 ppb and 0.01 ‰
- The growth rate was high during 1990's when methane concentrations increased from 1987 then levelled off in 1999
- There was no growth from 2000 to 2007
- There was variability observed in the methane $^{13}C/^{12}C$ signal when methane concentrations stabilized. The data record picked up the Mt. Pinatubu eruption
- If methane concentrations versus change in isotopic signature are compared this gives an indication of the complexity of the sources and the changes in the major sources of methane
- Data also detected when biomass burning was reduced and there was a decrease in the number of methane sources.

GC-IRMS continuous flow linked to MAT 252 for measurement of atmospheric $\delta^{13}CO_2$ (Ferretti, Brailsford, Martin, Moss)

- Method:
  - Separate CO_2 from air using gas chromatography
  - Inject 1ml air ~ 20 nmol CO_2 into MS
  - CO_2 and N_2O are separated
  - Determine $^{13}C/^{12}C$ ratio versus the reference in air
- The lab has measured a decrease in $\delta^{13}C$ values in CO_2 at Baring Head. The depleted CO_2 in the atmosphere reflects an increase in fossil fuel burning. A similar pattern has also been seen in the atmospheric CH_4 record from Law Dome.

GV Isoprime continuous flow mass spectrometer: $\delta^{13}CH_4$ of ancient air in ice cores (Ferretti, Riedel, Schaefer, Franz)

- Method:
    - Extract small bubbles of air from ice cores
    - Determine $\delta^{13}CH_4$ for ancient air trapped in ice cores using GC-IRMS techniques
- How have the source contributions changed with changes in anthropogenic activity?
- Atmospheric methane concentrations have increased recently, but the 2000 yr Law Dome ice core record of methane concentrations and $\delta^{13}CH_4$ show unexpectedly ^{13}C-rich CH_4 prior to 1600 AD, which indicates more natural CH_4 emissions from biomass burning and/or thermogenic sources in the past than previously thought.

Hinrich Schaefer **Stable carbon isotope constraints on greenhouse gas budgets in the past**

[P17_NIWA_Schaefer.pdf]

Ice core greenhouse gas work (Hinrich Schaefer, Peter Franz, Katja Riedel, Gordon Brailsford, Ross Martin, John McGregor).

- A correlation has been observed between atmospheric methane and temperature ($\delta^{18}O_{ice}$) from the GISP2 ice core (Brook et al., 1996). CH_4 follows general and episodic variations in temperature (as indicated from $\delta^{18}O$ record), including Younger Dryas and Heinrich events.
- Air bubbles in polar ice are an ideal archive for past atmospheres
- The NIWA Ice Grater System (NIGS) was designed to avoid artefacts for both CH_4 (from internal moving parts) and CO_2 (from melting) during gas extraction
- NIWA has the capability to extract gases for both CO_2 and CH_4 analysis
- NIWA is also carrying out continuous-flow IRMS analysis of $\delta^{13}CH_4$ on samples of ~2.5 mmol of carbon (40 ml of air at ambient CH_4 concentration or equivalent)
- Rapid warming from 11.2 to 12.2 thousand yrs BP (Younger Dryas transition to Early Holocene), as indicated by $\delta^{15}N$ patterns, are associated with a rise in CH_4 concentration profiles, but no similar pattern is observed in $\delta^{13}C$-CH_4 (Schaefer et al., 2006). Hinrich used a mass balance model to establish the equilibrium point for all expected sources & sinks of CH_4 at the YD cold period (~12,000 yrs ago). The model considered methane fluxes from termites, temperate and boreal wetlands, animals, oceans, clathrates, aerobic methane production (AMP), geological methane & biomass burning (isotopic signatures ranging from -70 to -26 ‰)
- Transition source $\delta^{13}CH_4$ is close to tropical wetlands and AMP
- Outlook:
    - Develop gas extraction methods at NIWA
    - Analysis of $\delta^{13}CH_4$, δD of CH_4 and $\delta^{13}CO_2$ in conjunction with concentration measurements of CH_4, CO_2 and N_2O
    - Develop a facility for stable isotope analysis of nobel gases.

Helen Neil **Carbonate analytical facility: C & O environmental applications**

[P18_NIWA_Neil.pdf]

Expertise & Analytical Capabilities

- Research and technical staff: Helen Neil, Jan Vorster, Peter Marriot, Rowena Moss
- Carbonate analytical facility comprises Thermo Finnigan MAT252 linked to a Kiel III device
- Other analytical facilities that can also perform C and O SI analysis of carbonates are: GNS and Universities of Canterbury and Waikato
- NIWA's system is optimised for analysis at very high resolution for very low volume carbonates
- NIWA uses a micromill for sample preparation, rather than a laser ablation system
- Micromill - complex structures can be sampled with submicron stage resolution, positional accuracy and precise depth control with real-time video observation
    - Milling resolution: 10 μm
    - Sample size: 20 μg (ideal size is 30-50 μg)
    - Reported error: ± 0.04 ‰ $\delta^{13}C$: ± 0.08 ‰ $\delta^{18}O$ from extraction to measurement.

Some Applications

- **Establishing the environmental influence on otolith structure of deep sea fish** (e.g. blue nose ruby, oreos & black cardinal fish). Helen uses structural and $\delta^{18}O$ isotopic variations in otoliths in conjunction with oceanic water temperature profiles to reconstruct vertical water migrations over the life history of the fish. Deep dwelling adults are shown to live in warm surface waters as juveniles, they then migrate as adolescents, exhibiting feeding movements in midwaters, to eventually reside in deep waters as adults living and spawning in stable deep water temperatures (Paul et al., 2003; Neil et al., 2008).
- **Using $\delta^{13}C$ signal in otoliths to establish size and age of fish at maturity** As fish grow and reach maturity their metabolism rapidly decreases, trophic level increases and $\delta^{13}C$ signatures decrease from around -5‰ to 0‰. At maturity they reach a stable metabolic rate and trophic level and their $\delta^{13}C$ signal plateaus. By simultaneously analysing the otolith growth rings, age at maturity can be established.
- **Establishing age and growth rates of Paua** Paua do not have regular growth rings like otoliths and, as such, are very difficult to age. Annual seawater temperature cyclicity of Paua's habitat along with shell $\delta^{18}O$ signals can be used to define age and longevity of tagged Paua (Naylor, Neil et al., 2006).
- **Palaeoceanography- long-term spatial and temporal changes in SST constructed from planktonic foraminifera** Analysing $\delta^{18}O$ in forams collected from sediment cores from Campbell Plateau (dated to 100-200 Kyr) has enabled reconstruction of paleo water mass development (Neil et al., 2004)
- **Palaeoceanography- definition of source waters** Changes in water masses flowing over sites can be derived from the isotopic signature recorded in foraminifera retrieved from the sediments. Combined $\delta^{18}O$ and $\delta^{13}C$ values from Campbell Plateau sediment forams enabled subantarctic (SA), subtropical front

(STF) and subtropical (ST) source waters to be categorised, with ST waters exhibiting depleted $\delta^{18}O$ and $\delta^{13}C$ values compared to SA waters (Neil et al., 2004).

- **Climate change: use of brachiopods to determine changes in mixing/upwelling** Brachiopods live in the mixed layer of the ocean (below 200m water depth). The $\delta^{18}O$ values recorded in their shells has provided a master chronology of Antipodes mixed layer marine temperatures and given a direct indication of mixing/upwelling events (related to westerly winds). The brachiopod mixed layer temperature record correlates strongly with westerly wind patterns over the Subantarctic area. Of note, is the significant decrease in temperature during the 1960's. Subantarctic penguin populations are generally in decline, however a small recovery occurred during the 1960's; a time of greater westerly winds, cooler temperatures, increased upwelling and potential increase in food supply (Thompson & Neil, unpublished).
- **Climate change: use of corals to log deep water mass changes** $\delta^{18}O$ deep water bamboo coral signatures, in conjunction with Mg/Ca ratios, represent an ideal temperature logger of deep water mass changes and hence oceanic gyre circulation. Banded coral from Snares Platform in1000m water lay down growth rings: measuring $\delta^{18}O$ signatures in the coral bands provides a proxy for deep sea water temperatures as far back as 1600 (Neil & Thresher, in prep; Thresher & Neil, in prep.). Tasman Sea deep ocean temperature time series (Sutton, unpublished) indicate significant temperature anomalies are occurring on decadal cycles. These have been attributed to variability in the Subtropical Gyre. The long-term deep sea temperature records derived from bamboo coral indicate that this decadal variability in the Subtropical Gyre has occurred over the past 150 years, with significant long-term spin-up of circulation occurring prior to this time. Such gyre changes may result in a warmer sea, lower nutrient availability, decrease in phytoplankton production & decreased food supply.
- **Climate change: using the isotopic record in speleothems as hazard indices** Speleothems, which are calcium carbonate cave deposits (stalactites and stalagmites being examples) can provide historical records of tropical cyclones via recorded $\delta^{18}O$ carbonate signatures. Depleted $\delta^{18}O$ rainfall signatures are associated with tropical cyclones and tracking these depleted signatures by finely sampling speleothems has enabled a historical 780 yr climate record to be recreated in tropical Australia. The tropical cyclone events identified by $\delta^{18}O$ signatures can be translated into hazard indices (depleted $\delta^{18}O$ = high hazard index value) enabling the return periods of tropical cyclones to be easily identified (Nott, Neil et al, 2007).

Wish list

- Equipment capable of measuring isotopologues in high resolution in carbonate molecules.

Scott Nodder **Compound Specific Stable Isotopes (CSI): Oceanographic applications**

[P19_NIWA_Nodder.pdf]

Examples of some NZ CSI research projects

- Hauraki Gulf (Uhle, Nodder et al., 2006; Sikes, Nodder et al., 2009). Use of amino acids and N-alkanes stable isotope signatures to trace sources of organic matter in marine sediments of the Gulf
- Fiordland (Mcleod, Frew, Wing) Use of fatty acids isotope signatures to investigate trophic links and carbon source pool use, including tracking of terrestrial carbon into marine ecosystems
- Chatham Rise (Nodder, Pinkerton, Bury, Thompson, submitted). Future plans to incorporate CSI analyses to supplement C and N SI food web studies to provide further insight into trophic linkages and organic matter sources.

Other work

- Sources of dissolved methane in sediments (Law, Nodder). Sampled sediments close to methane seeps sites and analysed $^{13}C\text{-}CH_4$ signature of sediment gases and organic matter to determine methane source. Isotope values were close to -60 ‰ (indicating a shallow organic/biogenic source rather than a clathrate methane source (-70 to -80 ‰).

Hauraki Gulf: Establishing sources of organic matter in marine sediments using CSI

- Study area: the Hauraki Gulf is an upwelling coast affected by wind-driven processes and by sporadic subtropical surface intrusions from East Auckland Current (EAUC). In summer the system becomes predominantly heterotrophic, shifting from spring autotrophic conditions
- Methods
    - Sediment samples ground & lyophilised
    - Amino acids extracted using acid hydrolysis & esterification (HP 6890 gas chromatograph/HP 5973 GC/MSD (mass selective detector) with He carrier gas)
    - Analysed on ThermoFinnigan Delta^{plus} XL isotope ratio mass spectrometer (combustion furnace/nafion water trap) in USA
    - Used a two end member source apportionment model (marine and terrestrial end member values taken from Keil and Fogel, 2001) to derive relative proportions of organic matter sources to sediments. These data are now published: amino acid model (Uhle, Nodder et al., 2006); N-alkanes model (Sikes, Nodder et al., 2009).

Max Gibbs **Using stable isotopes for tracking and tracing**

[P20_NIWA_Gibbs.pdf]

Tracking and tracing applications

Use of laser ablation to read shellfish life history records

- Max used laser ablation to subsample 50 µm diameter, 5 µm deep portions of scallop shells across shell. The nacre in scallop shell was analysed using an innovative low level N analysis technique to obtain N isotopic signals across the shell as the scallop ages. Results for various life stages revealed:
    - Spat: $\delta^{15}N$ 0.9 ‰ indicating a phytoplankton signature and pelagic habitat
    - Juveniles: $\delta^{15}N$ 2.6 ‰ indicating an ice algal signature and under-ice attached feeding regime
    - Adults: $\delta^{15}N$ 10.88 ‰ indicating a bottom habitat, feeding on benthic diatoms.

Soil and land use contributions to estuarine sediments: 2 field study sites

- This study was tracking the possible sources of carbon to an estuary. Likely sources were: marine/estuarine/salt marsh, terrestrial (pasture, forest, scrub, bare soil etc.) and anthropogenic (industrial, fertiliser, urban runoff, sewage etc.).
- **Whitford Estuary**: $\delta^{13}C$ isoscapes were used to pick up sources of clay and anthropogenic pollution signals.
- **Mahurangi Harbour**: Max used a combination of $\delta^{13}C$ bulk and CSI fatty acid signatures (oleic, palmitic, stearic acids) in sediments to determine the relative proportions of terrigenous, marine and diatom C sources in the estuary. The study mapped CSI isoscapes in the estuary and using the IsoSource mixing model worked out proportional contributions of different land uses to sediment in Mahurangi: e.g. the relative contributions from pine, pasture, & native forest. Although 70% of the catchment area is pasture, with pine occupying only 8% of the area, the contribution of pine soils to sediment in Mahurangi Harbour was 50 %. This was largely due to the poor management of logging operations where logs were dragged through riverbeds (Gibbs, 2008).

- **Water isotopes**: The heavier isotope of oxygen (^{18}O) preferentially forms rain, such that clouds become isotopically lighter and rain becomes isotopically heavier as clouds move across mountain ranges from their formation over the oceans. This produces variability in precipitation values across landmasses, which can be mapped into isoscapes. Bowen & Revenaugh (2003) have produced global isoscapes of δD and $\delta^{18}O$ precipitation values. Similar isoscapes can be mapped and drawn up for groundwater isotopic signals. Processes of precipitation, water uptake by vegetation, evapotranspiration, infiltration etc. all cause isotopic fractionation of H and O in water, leading to isotopic variability in groundwaters. Mapping such variability gives us the opportunity of tracing water sources and will have important applications in New Zealand in areas such as Raglan Harbour, Rotorua Lakes, Lake Taupo and the Canterbury/Otago region. For more information on water isotopes go to the website "waterisotopes.org".

2. Discussion

1) **Discussion and synthesis of presentations**
2) **SI annual forum – rotated through host institutes?**
3) **Standards – inter-calibrations?**

1) Discussion and synthesis of presentations

Due to a shortage of time at the end of the first day the group decided that discussion and synthesis of presentations should take place the following morning. The following topics were suggested for discussion as break-out groups:

- Database discussion – how do we collate a database of analytical facilities?
- Standards intercalibration exercise
- Collaboration between institutes and science sectors – development of joint projects
- Joint mapping of waters – for D/H isoscapes – future exemplar for SI community working together (Troy/Max). This would be a good use of capability funding to get such projects off the ground
- Nitrates – development of technologies and techniques (Troy/Russell&Robert/Travis)
- How we are educating students? – annual get together of SI community – upskilling introduction of SI to those new to subject (Karyne/Max/Troy)
- Technical group – swap meet – how can we share our resources? (Gordon/Robert)
- Costing of SI analyses? Cost vs. Profit models
- Consumables purchase. Are we getting the best deals? Should we select one provider: collective purchasing?
- TDL, MIMS – development of denitrification techniques & applications.

2) SI annual forum – rotated through host institutes?

There was resounding agreement from all participants for an Annual Forum to be held. It was agreed the best time to hold it was around November with more lead-in time. GNS scientists (Valerie, Karyne, Troy, Nancy) volunteered to host the next meeting. Participants agreed that a meeting lasting at least 2 days with presentations and possible break-out groups to focus on specific areas (Travis) would work well. Topics for discussion might include: new developments and applications, resourcing issues, fostering linkages (cross-linkages, joint projects between institutes - Max), technical tips & services (establishing contacts and realising expertise - Gordon, Robert). It was suggested that a SI introductory day course could be held prior to the 2-day workshop for students and those new to the subject area.

3) Standards – inter-calibrations?

Due to time constraints the discussion for this was delayed until the following day.

3. Stable Isotope Working Group Forum

Day 2 – Friday 7 November

Workshop to focus on:

- generating a database of NZ's SI analytical capabilities
- how new developments/applications/ideas can be applied to solve problems facing us in environmental research
- fundamental research questions we cannot currently address
- what new developments we might still need to make to address fundamental research questions.

Present: Troy Baisden, Nancy Beavan, Gordon Brailsford, Julie Brown, Sarah Bury, Valerie Claymore, Russell Frew, Max Gibbs, Hilary McMillan, Mandy Meriano, Rowena Moss, Helen Neil, Sophie Mormede, Scott Nodder, Andy Phillips, Karyne Rogers, Richard Storey, David Thompson, Robert Van Hale, Jan Vorster.

Discussion Items
The group decided not to have break out sessions as too many people wanted to make input to all discussion areas. These were distilled into 7 discussion topics.

1) Standards intercalibration
2) Joint projects - collaboration between institutes e.g. Water Isotopes NZ – Isoscapes Project
3) Nitrogen: development of technologies and techniques for nitrogen cycle measurements: nitrates, denitrification (TDL, MIMS)
4) Education/Next SI meeting
5) Technical group/swap meet: how can we share our resources? Collective purchasing?
6) Database of analytical facilities – SI webpage?
7) Workshop Outputs – report format.

The following discussions have been recorded in note form. Where noted the person making a comment/suggestion is given in brackets.

1) Standards intercalibration

- Need to establish a working group to drive intercalibration of standards for NZ labs
- Needs a more formal integrative approach than is currently used. Each lab at the moment has its own independent method of calibration, but there is no intercalibration between labs
- For NIWA's atmospheric stable isotope measurements there are world meteorological organisation (WMO) requirements that have to be adhered to. Gas cylinders are sent round world for intercalibrations. This is driven by the motivation of individual labs and is globally integrated. Fifty percent of sample analyses in the gas lab at NIWA are intercalibrations. For the dual inlet systems

intercalibrations are more frequently done than when running samples under CF-IRMS (GordonB, RossM)
- We need to state parameters for standardizing data-sets, especially for developing isoscapes and future-proofing data-bases (GordonB)
- Data comparisons and intercalibrations will be very important for establishing NZ water isoscapes
- We need to determine a set of NZ standards for ecological applications (TroyB)
- We should be careful to note the difference between primary IAEA or NIST standards and secondary laboratory standards used in each individual laboratory (Sarah B)
- Metrological organisations are encouraging physical and chemical standards for NZ, but this is an expensive process (GordonB)
- We should use ring-tests, e.g. IAEA standards for intercalibrations. We need to get more involvement with global intercalibration programmes (KaryneR)
- We need to encompass involvement of the whole NZ community
- Standards need to be compatible with the measurements that you are making, e.g. standards for calibration of deuterium. There are issues when no compatible standards are available, especially in new applications, e.g. CSI analysis (RussellF, MaxG)
- Should we try to organise a NZ Ring-test over the next year? Perhaps starting with internal laboratory standards as a comparative process, perhaps co-ordinated through lab managers? (ValerieC)
- A standards database would be useful (AndyP)
- After the last radiocarbon meeting they ran an intercalibrations exercise and presented the data at the following conference. There was no identification of laboratories (each lab is assigned a coded number and the co-ordinator is the only person who knows which lab presented what data (NancyB)
- Intercalibration tests should be carried out, disseminated and reported at future SI meetings. Try to complete a NZ ring-test by next year's meeting (NancyB)
- Need a volunteer to lead the intercalibrations exercise.

[*Post meeting note: Since the meeting Robert van Hale has offered to take this up and has emailed all the key laboratories in New Zealand and Australia inviting them to take part in an intercalibrations exercise. Please contact Robert if you wish to take part in this and have not received an email from him.*]

2) Joint projects: collaboration between institutes e.g. Water isotopes NZ – Isoscapes

- A good example of a current joint collaborative projects is "Land-to-Water Nitrogen Transfers" programme involving GNS, Lincoln & Waikato Universities, AgResearch and Environment Canada
- Water Isotopes NZ Isoscapes project would be an ideal candidate for a joint collaborative venture.

[*Post meeting note: A joint project has now been set up between NIWA & GNS with a memorandum of understanding drawn up: GNS will have access to a full suite of water samples from NIWA's sampling sites through the National Rivers Water Quality Network (77 sites). The agreement is for samples to be taken on a monthly* *basis over a year and to be analysed at GNS with joint ownership and use of data between NIWA & GNS.*]

- Some applications of NZ $\delta^{18}O$ and δD isoscapes would be: identifying & monitoring soil erosion problems, including sediment transport, water resource issues, water vapour mapping
- $\delta^{18}O$ and δD is presently measured by GNS (ValerieC & AndyP), Landcare (MargaretB, JohnH), IsoTrace (RussellF, RobertVH), Canterbury University (TravisH) [*Post meeting note: Since the meeting NIWA has purchased a Picarro Wavelength-Scanned Cavity Ring Down Spectroscopy (WS-CRDS) real-time portable field instrument for measurement of $\delta^{18}O$ and δD in water & water vapour*]
- There are 3 possible methods for measuring $\delta^{18}O$ and δD:
    - Landcare - cavity ring-down tuneable diode laser (TDL) using water vapour equilibration
    - GNS - pyrolysis (reduction over Cr for H/D, equilibration method for $\delta^{18}O$)
    - Isotrace - pyrolysis over glassy carbon for H/D and equilibration method for $\delta^{18}O$
- Intercalibrations between all laboratories are currently ongoing.

Current status of $\delta^{18}O$ and δD water sample database

- GNS have paper copies of $\delta^{18}O$ and δD values in precipitation, surface water and ground water at scattered locations around NZ from 1960's onwards. It would require funding to quality check and extract these historical data before they would be of use to contemporary studies. Most of the analyses were carried out over different spatial and temporal periods so the datasets are not continuous in time or space. In other words, they are local studies rather than co-ordinated NZ-wide measurements: e.g. measurements of D in rainwater (Kaitoke, Hutt river); surface water & ground water measurements since early 2000's from localised N island locations. Measurements have been more of a snapshot and not a thorough co-ordinated set of all measurements throughout NZ (TroyB, ValerieC, KaryneR)
- Isotrace have 52 stations where they have been measuring $\delta^{18}O$ and δD in rainwater. The stations are linked to the NIWA meteorological & climate database. Russell has an MSc student who is modelling data, which they may develop into a PhD. Cost is $40k to co-ordinate sample collection & for project management (RussellF, RobertvH)

- NZ-wide co-ordination of water sampling for isotope analysis: there is a possibility of using NIWA weather and river stations for monthly sample collection of precipitation, surface and groundwaters. Perhaps a joint project between NIWA and GNS could start with sample collections of water for analyses (MaxG) [*See post meeting note above*]
- For water samples run by GNS 2 mls minimum sample size is required. This is unfiltered & needs to be sealed & frozen (ValerieC, TroyB)
- Is it possible to infill any spatial gaps in Russell's sample locations with the NIWA weather station sites? (ValerieC)
- We should also use the NIWA river's field team for taking water samples (GordonB)

- NIWA has 170 river field sites which is sampled monthly. It also has over 1000 sites with manually accessed rain gauges (MaxG)
- Mike Stewart compiled a report of all δD (but not $\delta^{18}O$) river and rainfall measurements made by GNS. This report could be a useful template. We could possibly use student projects as a means for infilling existing spatial and temporal gaps in the data records (TroyB)
- We may want to co-ordinate these δD & $\delta^{18}O$ water sampling initiatives with the nitrate sampling programme as well (TroyB)
- Do we have enough spare capacity with current NZ mass spectrometer facilities to do these water isoscapes analyses? Does Travis need to invest his money in equipment?
- With GNS's new mass spectrometer in place [*Post-meeting note: this has now been purchased and installed – June 09*] there will be a spare capacity of 4000 samples per year for δD & $\delta^{18}O$ analyses (although this is a moveable feast with current unknowns of commissioned work coming in)
- We should take Carol Kendall's philosophy: take as many samples as possible and bank them if they can't be analysed immediately
- Funding of NZ water isoscapes project? We need to co-ordinate an integrated collaborative project between institutes
- We have agreed on the need for rainwater, ground water, river water isoscapes. What about leaf water isoscapes along the lines of Gabe Bowen's isoscapes? (MargaretB)
- Another missing isoscape is tap water and irrigation isoscapes for forensic applications (TroyB)
- Joint funding initiatives are required to launch the water isoscapes project - co-ordination between GNS & Isotrace with NIWA hydrology group (Hillary McMillan, Mandy Meriano, & Ross Woods) (MaxG, TroyB, RussellF).

[*Post-meeting note: Since the SI workshop a memorandum of understanding has been drawn up between GNS and NIWA (mediated by TroyB and MaxG). NIWA has purchased a Picarro WS-CRDS portable instrument for measuring δD & $\delta^{18}O$ in the field and the NZ water isoscapes project is underway*].

3) Nitrogen: development of technologies and techniques for nitrogen cycle measurements: nitrates, denitrification (TDL, MIMS)

Nitrates

- Laboratories currently analysing $\delta^{15}N$ in nitrates:
  - GNS (chemical method – use enzymes instead of Cd)
  - Isotrace (bacterial method, moving towards chemical method), Lincoln University (bacterial method)
  - University of Canterbury (future plans)
- We need to standardise laboratory methods of $\delta^{15}N$ in nitrates (both analysis of soil and water nitrates) and carry out a NZ-wide intercalibration. Currently both the bacterial and chemical methods are being used. How do we develop a standardisation method? Can we dissolve ^{15}N-labelled IAEA standards and measure those? (TroyB)

- There are problems associated with low level seawater $\delta^{15}NO_3$ analytical methods (RussellF)
- Should we think about setting up a national laboratory in one place focussing on $\delta^{15}NO_3$ analysis, requesting contributions from users? (RobertvH)
- Would there be funding for a National Nitrate Isotope Analysis Laboratory? (RobertvH)
- A good approach is to educate end-users, especially regional councils, to fund $\delta^{15}NO_3$ research (TroyB)
- Nitrate and N run-off are important issues, and $\delta^{15}NO_3$ measurements are a useful way of measuring and tracing run-off, therefore these measurements should be important to end-users. Despite nitrate runoff being a major problem with councils, funding from councils for this type of research is decreasing (RichardS)
- We would prefer the set up of 2-3 labs running $\delta^{15}N$ in nitrates as this expands the capacity (provides slack in system if there is machine downtime), & increases intellectual property, communication & collaboration on complex issues (TroyB)
- It is possible that GNS can lead the way here, focussing on terrestrial systems (in collaboration with Tim Clough at Lincoln), with Isotrace's expertise in oceanographic applications focussing on low level nitrate analysis. Len Wassenaar is coming out to GNS to assist in the set up for Troy's nitrogen transfers project. GNS is looking into the bacterial method as well as the chemical method (AndyP)
- NIWA has a national database (held by NIWA and regional councils) for NO_3 concentration in river waters. This could complement $\delta^{15}NO_3$ isotope data.
- Call for education as to what you can do with isotopes (RichardS)
- We do need to generate good results before we sell the $\delta^{15}NO_3$ method
- N isotopes on their own don't solve all problems.

Denitrification

- ^{15}N-nitrate enrichment studies can be used to measure denitrification rates and processes
- We can establish how much of an isotopic shift is due to denitrification. What is influencing fractionation factors? (ScottN)
- Fractionation effects will be determined from Land-Water project and we expect methods to be applicable to low level nitrates as well (TroyB)
- MIMS system is also used for measuring denitrification rates – this measures concentrations not isotopic ratios (ScottN).

4) Education/Next meeting

- For 6 yrs GNS has been running an annual Quaternary Techniques workshop (costed at $100 for students, $180 non-students). GNS bring in outside experts to present information on methods and applications, whilst KevinF, KaryneR, TroyB and NancyBertler present isotope related work. Maybe we could co-ordinate stable isotope issues & presentations with this course, or have a SI student introduction day prior to next year's SI meeting? (KaryneR)
- The question we have to address is, do we want to have a future meeting with focussed SI groupings or do we want a broader SI meeting like this one? (DavidT)
- Do we want a similar format meeting to this one, which has been focussed on delivering information to people on what the technical capabilities are within NZ

isotope laboratories, with highlights of applications and key research? Or do we need to change the format of the next meeting? (SarahB)

- The most important thing is that representatives from all labs attend
- The meeting needs to have a research-focus
- It also needs to incorporate students
- Maybe we just highlight new technologies & equipment which has come into laboratories over the past year so we can update the "Summary NZ analytical capabilities" table output from this meeting? (SarahB)
- Getting a wide range of people together covering different subject areas enables cross-fertilisation of ideas. Perhaps we could break-up future meetings with focus groups on specific applications, i.e. have a 3-day symposium with focus groups on the last day (ValarieC, SarahB)
- There is a preference to maintain this workshop model (i.e. exchange of technical information/new advances/opportunities for collaborative projects etc), with science input via posters & short talks. It needs to be a workshop format with plenty of opportunity for discussions & collaborations (RussellF)
- We could have extensions into particular research areas with specific sessions and encourage students to attend as well (TroyB, SarahB)
- We could also have a technical forum via exhibitions, perhaps even encouraging manufacturers to attend? (TroyB, AndyP)
- We should use ISOECOL conferences as a model where organisers and attendees made the decision that there would only be one session that everyone attends, with no concurrent sessions (MaxG)
- If we are to have an introductory SI course for students prior to the meeting we should develop some course material for education
- Who would take charge of the course? (No firm decisions made at meeting)
- Suggested format for next meeting:
  - Day 1 education
  - Day 2-3 talks
  - Day 4 Workshop and discussion topics
  - Focus on talks with 5 min presentations only to get across key points
  - End Oct/early Nov was noted to be the best time for the next SI meeting.

5) Technical group/swap meet: how can we share our resources? Collective purchasing?

- Do we need to have a swap meet within the next meeting just for a technical group? For example, GNS has an old manual dual inlet Micromass 1202 with no rotary pumps which might be good perhaps for student use? This is available to a good home
- Should we try to organise collective purchasing of mass spectrometer consumables?
- Would this include standards?
- If we could co-ordinate all NZ labs to use one supplier we should have some leverage to procure better prices (KaryneR)
- At the moment NIWA & Otago use Elemental Microanalysis, whilst GNS use IVA

- A discussion centred around gas suppliers: GNS use BOC, NIWA use Air Liquide, and Isotrace use Southern Gas (which is imported directly from Australia). Southern Gas is currently only available in the South Island and is sold at a fraction of the price (about 1/3) that BOC and Air Liquide sell their He at.
- Russell offered to look into perhaps doing a bulk import of He from Southern Gas (he offered to co-ordinate this with Ken Garnet) and then ship cylinders in bulk to labs in the North Island. (Should only cost around $50 per cylinder for shipment from South to North Island).
- What will the SI community do if He gas runs out? This appears to be a real possibility. Do we rely on new technologies coming on board that will provide alternatives to He carrier gas mechanisms? For the time being everyone seems to be carrying on with business as usual!
- Scrubbing lower grade He is a cheaper option than purchasing UHP He (NIWA has done this in the past) (SarahB, GordonB)
- Do we want to try and use our power as a collective of labs to lever lower prices for gases?
- Volunteers put their hands up to investigate getting bulk discount prices from suppliers
    - IVA: KaryneR
    - Elemental Microanalysis: JulieB
    - Southern Gas: RussellF.

[*Post-meeting note: Bulk discount price information has been obtained from IVA, however these prices are 25-30% higher than the normal undiscounted rates of Elemental Microanalysis. When Julie Brown approached Elemental Microanalysis requesting the possibility of discounts on large bulk order purchases the manager was willing to look into this for us, but he would need further information on the number of labs likely to participate and likely quantities of orders. This is something Julie could perhaps pursue by collating information from interested parties at the next SI meeting? Ken Neal has also recently (June 2009) proposed prices approximately 20% lower than current Elemental Microanalysis tariffs.*]

6) Database of analytical facilities/SI webpage

- How do we co-ordinate all the capability information that has been presented at this workshop?
- It was suggested to tabulate all details of SI analytical facilities at each organisation, detailing contact person, equipment, isotopes, sample types, applications, wish lists etc. Sarah is to co-ordinate this from all of the PowerPoint presentations and scribed notes and will mail out a summary table to all lab managers for them to edit prior to distribution. Sarah will also create a summary report from the workshop. (SarahB, ValerieC)
- The summary facilities table should also include a listing of international and secondary standards that lab managers use.

[*Post-meeting note: This has not been done as this is probably better to be co-ordinated by Robert van Hale through his laboratory intercalibrations exercise.*]

- Was also suggested that mass spectrometer spare capacity should be indicated on the summary table. [*Post-meeting note: This was also not done as it is an ever-changing moveable feast*].
- How will the workshop report & summary table be disseminated?
- General agreement that it would be good to have it on some kind of centralised webpage.
- Ideally we should have a NZ SI webpage where we can host information on NZ-wide analytical facilities and SI community issues
- Who would organise and maintain this? Who would be webmaster? Who would do updates?
- Would we post data on the website? There would be issues of commercial sensitivities and potential for duplication of effort.
- Would the website have links to each institute with SI facilities/research interests?
- Troy Baisden very kindly offered to take the responsibility for generating a central NZ SI webpage which would include a posting of NZ analytical facilities & capabilities (which could be continually updated as organisations procure new equipment). Sarah will post summary meeting notes on the NIWA website until this is available and then transfer the information across to the website once it is developed (TroyB, SarahB)
- GNS also confirmed that they will host the next meeting. This was accepted with thanks from all at workshop.

7) Workshop Outputs:

- Written summary report – including a summary table of NZ analytical facilities, with details of: institute/organisation, location, contact person, analytical equipment, isotopes, sample types, applications, wish lists, future plans etc.
- PPT presentations – presenters to update/edit their presentations into versions they are happy to be circulated, which will then be transferred into pdf format before distribution
- Report to be posted on NIWA webpage initially to enable open access to whole isotope community, then to be transferred to independent (non-affiliated) NZ SI webpage once this has been set up by Troy Baisden

[*Post-workshop decision: SI summary meeting report will also be prepared as a hard copy NIWA information report which will be circulated to all laboratory managers. A CD will also be produced, which will include the full report plus summary facilities table and the PowerPoint presentations as pdf files. Costs for producing this will be funded by NIWA.*]

- NZ isotope webpage to be developed – Troy Baisden has kindly offered to set this up and be the webmaster. This will become the location for posting summary reports from future SI meetings & the summary table of facilities which can be updated annually following SI meetings. It is anticipated that this webpage will become our forum for SI discussions and information dissemination.

Appendices

Appendix 1 Meeting Information and Programme

NIWA is hosting the workshop with complimentary tea, coffee and lunch, provided on the first day and coffee on the second day. There is no cost to register, although travel and accommodation costs will need to be met by attendees.

ACCOMMODATION: The closest accommodation to NIWA is at Bella Vista opposite Greta Point. Other accommodation can be found at http://www.wellingtonnz.com/. Close locations within 20-30 minutes walking distance are in Kilbirnie (try 747 Motel Studio Units) or along Oriental Parade.

REGISTRATION DESK: Please collect your name badge and updated workshop programme between 08.00 – 08.30 outside the Main Conference Room, Allen Building. There is no requirement to sign in at Reception for attendance of the workshop.

POSTERS: If you wish to display a poster please put this up during registration or coffee/lunch breaks. Posters will be displayed on the walls of the Conference Room. You can obtain Velcro sticky tabs from the registration desk.

ORAL PRESENTATIONS: We are requesting that **innovative science presentations** be presented as part of the time allocation slot for each analytical facility. Facilities and co-ordinators that are presenting are: GNS (Valerie Claymore), NIWA (Sarah Bury, Max Gibbs, Gordon Brailsford, Helen Neil), Victoria University (Joel Baker), Landcare (Margaret Barbour, John Hunt), Lincoln (Tim Clough), Otago University (Russell Frew, Robert Van Hale), Canterbury University (Travis Horton), Waikato University (Brendan Hicks), Environmental Isotopes (Ken Neal). Please contact your relevant coordinator if you wish to make a **succinct presentation focusing on innovative applications** (maximum 5-10 minutes time slot).

PRESENTATION FORMAT: Presentations should be in Microsoft PowerPoint, version 2003. If you are presenting in the first session before coffee break please make sure you load your presentation onto the laptop during the registration period. All other presentations can be loaded during the coffee/lunch breaks immediately before your session.

QUESTIONS: There will be an opportunity for you to submit requests to the workshop conveners of what you would like to get out of the workshop. If you have any particular questions or items you would like discussed please submit a request via a request slip which will be at the reception desk, or notify Sarah in advance at s.bury@niwa.co.nz

LABORATORY TOURS: We will be running four tours of the mass spectrometer laboratory at Greta Point during the lunch period on the first day (Thursday). Please sign up for a ¼ hr tour slot at the registration desk on arrival. Rowena Moss and Gordon Brailsford will be coordinating these tours. For Health & Safety reasons we can only show 8 people round at a time, so it is essential to sign up and book your tour time in advance!

THURSDAY EVENING SOCIAL PLANS: Most registrants indicated they would like to meet for dinner on Thursday night, however due to Christmas bookings we cannot make a large group booking at such short notice. We plan to suggest a central bar to rendezvous in before and after dinner and provide a list of varied restaurants close to the bar to facilitate social interactions after the meeting!

PROGRAMME

This is an informal meeting with the main objective of getting the stable isotope community together to interact in a fun and positive manner to enhance collaborations and strengthen New Zealand's Stable Isotope Analytical Capability.

Main aims of the Meeting

1) to assess current stable isotope research capabilities within NIWA and New Zealand
2) to identify key areas of innovative research and associated technologies, and
3) to recommend the necessary analytical infrastructure to support ongoing and new science programmes over the next 5-10 years.

Thursday 6th November: Presentation Forum "NZ's SI Analytical Capabilities and Applications"

08:00-08.30 Registration (Main Conference Room, Allen Building)

08:30 – 08:35 Opening of Meeting and Welcome by Clive Howard-Williams, NIWA Ltd., Chief Scientist Freshwater & Coasts

08:35 – 08:45 Introductions & Housekeeping Workshop aims and introduction of workshop co-ordinators (Sarah Bury)

Current and Future New Zealand Stable Isotope Analytical Capabilities

Summary of analytical facilities from laboratory managers, including innovative science application presentations. These presentations will include information from each organisation on:

1) Current analytical capabilities
2) Current innovative applications
3) Key future applications
4) Limitations of existing equipment
5) Current gaps in technology
6) Wish list for new technology

08:45 – 10.00 GNS Science, National Isotope Centre (NIC)

Valerie Claymore *GNS Stable Isotope Lab capabilities and applications*

Valerie Claymore/ Nancy Bertler *Ice core research*

Kevin Faure *Oxygen isotopes of silicates*

Nancy Beavan *Bioarchaeology and ecology*

Perry Davy *Stable isotopes and air particulate matter (APM)*

Karyne Rogers *From food to fuel: an overview of some current isotope research topics at GNS Science*

Troy Baisden *The isotopic indicators of "Land-to-Water Nitrogen Transfers" programme*

Andy Phillips *Future developments at GNS NIC*

10.00 – 10:30 COFFEE BREAK

10:30 – 10.50 Victoria University, School of Geography, Environment & Earth Sciences

Joel Baker (Matthew Leybourne presenting on behalf of Joel)
Analysis and applications of non-traditional stable isotopes

10:50 – 11.10 Canterbury University, Department of Geological Sciences

Travis Horton *University of Canterbury's new stable isotope analytical facility*

11:10 – 11:50 Isotrace & Otago University, Chemistry Department

Robert Van Hale *1) Isotrace Analytical Capability*
2) H isotopes of organic material

Russell Frew *Isotrace Research: current applications*

11:50 – 12:00 Lincoln University, Bioprotection Research Centre

Peter Holder *Stable isotopes and trace elements as New Zealand geo-location markers for biosecurity.*

12:00 – 13:30 LUNCH

12:20-13.20 Mass Spectrometer Laboratory Tours.

We will be running 4 tours, each lasting ¼ hr over the lunch period. Please sign up for a tour slot at the registration desk on arrival. See Rowena Moss or Gordon Brailsford who will be coordinating these tours. For Health & Safety reasons we can only show 8 people round at a time, so it is essential to sign up and book your tour time in advance!

13:30 – 13:35 AgResearch, Grasslands Research Centre, Palmerston North

Mike Tavendale *AgResearch Stable Isotope Analytical Facilities*

13:35 – 13:40 Isolytix Ltd., Dunedin

Ken Neal (Sarah Bury presenting on behalf of Ken)
Isolytix Analytical Services

13:40 – 13:45 Lincoln University, Agriculture & Life Sciences Division

Tim Clough (Sarah Bury presenting on behalf of Tim)
Lincoln University Stable Isotope Analytical Facilities

13.45 – 14.00 Cawthron Institute

Chris Cornelisen & Reid Forrest
Applications and future directions of stable isotope research at Cawthron Institute

14.00 – 14:30 Landcare Research Ltd., Lincoln

John Hunt & Margaret Barbour
SI tuneable diode laser facility at Landcare Research

John Hunt *Understanding below-ground carbon dynamics using stable C isotopes*

Margaret Barbour *Applications of stable C and O isotopes in plant ecophysiology*

14.30 – 15:00 Waikato University Stable Isotope Unit (WSIU), Department of Biological Sciences

Brendan Hicks (Max Gibbs presenting on behalf of Brendan)

1) Waikato University Stable Isotope Unit (WSIU)

2) Use of laser ablation and ICP-MS to analyse otolith microchemistry: determining the migrations of freshwater fish.

15:00 – 15:30 TEA BREAK

15:30 – 16:45 NIWA Ltd., Wellington

Sarah Bury 1) *Introduction to NIWA's stable isotope analytical facilities*
2) Ecological applications of bulk C & N analysis (Also presenting data on behalf of Fleur Matheson and Cliff Law)

David Thompson *Stable isotopes and apex predators*

Neill Barr & Bruce Dudley
The use and application of nitrogen stable isotopes in marine macro algae for tracing anthropogenic nitrogen

Gordon Brailsford *Atmospheric stable isotope analytical facility and applications*

Hinrich Schaefer
Stable carbon isotope constraints on greenhouse gas budgets in the past

Helen Neil *Carbonate analytical facility: C & O environmental applications*

Scott Nodder *Compound Specific Stable Isotopes (CSI): Oceanographic applications*

Max Gibbs *Using stable isotopes for tracking and tracing*

16.45 – 17.30 DISCUSSION

Discussion and synthesis of presentations.
SI annual forum – rotated through host institutes?
Standards – inter-calibrations?

There will be an opportunity for you to submit requests to the workshop conveners of what you would like to get out of the workshop. If you have any particular questions or items you would like discussed please submit a request via a request slip which will be at the reception desk. We will endeavour to address these requests during Thursday or Friday discussion sessions.

DINNER **See suggestions of a meeting place and dinner options in INFORMATION section above**

Friday 7th November: MORNING WORKSHOP "SI Working Group Forum"

09:00 – 10:30 Discussion and Working Forum
10.30 – 11.00 Morning Tea
11:00 – 12:00 Discussion and Working Forum

Workshop, to focus on:

- generating a database of NZ's SI analytical capabilities
- how new developments/applications/ideas can be applied to solve problems facing us in environmental research
- fundamental research questions we cannot currently address
- what new developments we might still need to make to address fundamental research questions

Appendix II Summary Table of Stable Isotope Analytical Capabilities and Complementary Facilities in New Zealand

Summary Table of Stable Isotope (SI) Analytical Capabilities and Complementary Facilities in New Zealand

Current Facilities (May 2009)

Organisation facility listings appear in the following order:

Mass Spectrometer or Stable Isotope Analytical Instrument	Peripheral Equipment or Complementary Facility	Isotope or Type of Analysis	Sample Type	Applications	Contact Person & Details
NIWA, Wellington					
ThermoFinnigan Delta Plus Continuous flow	Fisons NA 1500 Elemental Analyser (EA) linked to continuous flow IRMS, with Conflo III reference gas and diluter open split	$\delta^{13}C$, $\delta^{15}N$	Solid organic materials: e.g. flora, fauna, sediment, atmospheric particles	Environmental, oceanic (including sub-tropical, sub-Antarctic, Antarctic), coastal, lacrustrine, riverine, riparian & terrestrial ecological studies, trophic studies, pollution tracking & management, sedimentary pathways & inputs, source apportioning (e.g. aquatic & atmospheric contaminants, dietary studies), marine, freshwater and lacrustrine carbon and nitrogen cycles/fluxes (e.g. process measurements of nitrogen fixation, denitrification, primary production etc.), nutrient supply on offshore islands, terrestrial & marine conservation, aquaculture, animal and plant physiology, animal migrations, fisheries management, tracking of invasive species	Sarah Bury s.bury@niwa.co.nz 04 386 0347
		$\delta^{34}S$ – NIWA has capability and expertise to analyse sulphur but current capacity of C & N analyses means insufficient time to re-configure system to switch to S analysis			
ThermoFinnigan MAT252 Dual inlet		$\delta^{13}C$ in CH_4	Atmospheric methane	Agricultural emissions, atmospheric monitoring, climate change	Gordon Brailsford g.brailsford@niwa.co.nz 04 386 0393 Ross Martin r.martin@niwa.co.nz 04 386 0384
		$\delta^{18}O$, $\delta^{13}C$ in CO	Atmospheric carbon monoxide	Climate change, gas emissions	Gordon Brailsford Rowena Moss r.moss@niwa.co.nz 04 386 0534
	GC-IRMS continuous flow linked to MAT 252	$\delta^{13}C$	Atmospheric carbon dioxide	Climate change, gas emissions	Gordon Brailsford
	Kiel III preparation device	$\delta^{13}C$, $\delta^{18}O$	Carbonate materials: e.g. foraminifera, bivalves, corals, speleothems, otoliths	Palaeoceanography, palaeoclimate, fish migration, fish age at maturity, fisheries management, paua aging & growth studies, terrestrial climate, precipitation, climate change hazard indices	Helen Neil h.neil@niwa.co.nz 04 386 0375

GVI Isoprime Continuous flow	Methane to CO_2 converter peripheral linked to Isoprime	$\delta^{13}C$ in CH_4	Methane in ice cores, gas seeps	Climate change, methane fluxes	Peter Franz p.franz@niwa.co.nz 04 386 0506 Hinrich Schaefer h.schaefer@niwa.co.nz 04 386 0399 Katja Riedel k.riedel@niwa.co.nz 04 386 0382
Picarro Wavelength-Scanned Cavity Ring Down Spectroscopy (WS-CRDS) Real-time portable field instrument		δD, $\delta^{18}O$	Water & water vapour	Generation of water isoscapes (meteoric water line), hydrology, biosecurity	Max Gibbs m.gibbs@niwa.co.nz 07 856 7026
NIWA Offline Sample Preparation Systems					
ThermoFinnigan Delta ^{Plus} Continuous flow	DIONEX 200 accelerated solvent extraction system (ASE) (NIWA Hamilton, Organic Chemistry Laboratory)	$\delta^{13}C$, $\delta^{15}N$	Lipid extraction of solid organic samples for lipid-free SI analysis	Environmental studies, ecological applications (see above for full details)	Greg Olsen g.olsen@niwa.co.nz 07 856 1781 Sarah Bury s.bury@niwa.co.nz 04 386 0347
ThermoFinnigan MAT252 Dual inlet & Kiel III carbonate preparation device	Micromill hi-resolution sampling lab	$\delta^{13}C$, $\delta^{18}O$	Carbonate materials: e.g. foraminifera, bivalves, corals, speleothems, otoliths	Palaeoceanography, palaeoclimate, fish migration, fish age at maturity, fisheries management, paua aging & growth studies, climate change hazard indices	Helen Neil h.neil@niwa.co.nz 04 386 0375
ThermoFinnigan MAT252 Dual inlet	Gas preparation laboratory - offline vacuum preparation systems	$\delta^{13}C$, $\delta^{18}O$	Extraction of CO_2 and CH_4 from low concentration atmospheric samples	Agricultural emissions, atmospheric monitoring, climate change	Rowena Moss r.moss@niwa.co.nz 04 386 0534
	Dissolved inorganic carbon (DIC) vacuum line	$\delta^{13}C$	CO_2 produced from dissolved inorganic carbon in water	Aquatic ecology, biological processes, palaeoceanography, carbon fluxes & budgets	Kim Currie k.currie@niwa.co.nz 03 479 5249 Gordon Brailsford g.brailsford@niwa.co.nz 04 386 0393

	Automated air & aeolian dust sampling equipment, including vertical air sampling & meterological profiling with tethersondes and radiosondes			Atmospheric chemistry, climate studies	Gordon Brailsford g.brailsford@niwa.co.nz 04 386 0393
GVI Isoprime and ThermoFinnigan MAT252 Dual inlet	Ice core gas extraction system (ice grater)	$\delta^{13}C$ in CH_4 and CO_2	Gas extraction from ice cores	Palaeoclimate, climate studies	Katja Riedel k.riedel@niwa.co.nz 04 386 0382 Gordon Brailsford g.brailsford@niwa.co.nz 04 386 0393
NIWA Complementary Facilities (Mass Spectrometry, Aerosol and Tracer Labaratories)					
Membrane Inlet Mass Spectrometry (MIMS)		Concentrations of major (CO_2, O_2, N_2, Ar) and trace, (CH_4, DMS, N_2O, H_2S) gas species Potential to use this system for isotope measurements	High resolution simultaneous continuous measurements of gas concentrations in seawater, freshwater and sediment pore waters	Marine and freshwater biogeochemistry, measurement of biogeochemical rates e.g. denitrification (from Ar/N_2 ratios) & community production/respiration (from O_2/Ar ratios), eutrophication studies, nitrogen budgets, aquaculture, atmospheric emissions & gas exchange rates	Cliff Law c.law@niwa.co.nz 04 386 0478
Campbell Scientific Instruments Trace Gas Analyser TGA100A TDL (Tuneable diode laser) and absorption spectroscopy Portable field instrument		N_2O flux measurements using GC micrometeorological eddy co-variance techniques	Air samples	Agricultural emissions	Gordon Brailsford g.brailsford@niwa.co.nz 04 386 0393 Ross Martin r.martin@niwa.co.nz 04 386 0384 Andrew McMillan a.mcmillan@niwa.co.nz 04 386 0399
Hewlett Packard 5972 Mass Spectrometer (NIWA Auckland)	Hewlett Packard 5890 GC (NIWA Auckland)	Fatty acid and sterol concentrations	Solids and liquids (samples prepared off-line, extracted using chloroform/ methanol solution)	Aquaculture – monitoring of nutrient incorporation into aquaculture species, food web biomarkers, identification of bacterial contamination	Matt Nelson (Auckland) m.nelson@niwa.co.nz 09 375 2073 Michael Bruce (Auckland) m.bruce@niwa.co.nz 09 375 4539

	Series of gas chromatographs (GC) set up for specific gas concentration measurements	Gas mixture preparation and measurement of concentrations of CO_2, N_2O, CH_4, CO, SF_6, O_3, non-methane hydrocarbons (C1-C6), acetylene, H_2, O_2/N_2 ratios, moisture content	Air samples	Atmospheric chemistry, gas flux measurements, agricultural emissions, climate change	Rowena Moss r.moss@niwa.co.nz 04 386 0534 Matt Evans m.evans@niwa.co.nz 04 386 0309
	Gas chromatographs (GC) for seawater concentration measurements	SF_6, N_2O, CH_4	Seawater	Oceanic gas concentration and flux measurements, methane seeps, climate change	Cliff Law c.law@niwa.co.nz 04 386 0478
	Gas chromatographs (GC) for seawater and air concentration measurements	DMS, DMSP	Seawater and air	Phytoplankton ecology, air-sea interactions and gas fluxes, climate change	Mike Harvey m.harvey@niwa.co.nz 04 386 0308
	Condensation nuclei counter (CNC)	Concentration of atmospheric air particles	Atmospheric particles	Atmospheric chemistry, climate change	Mike Harvey m.harvey@niwa.co.nz 04 386 0308
	Water Quality Laboratories (Hamilton & Christchurch)	Conductivity, pH, alkalinity, turbidity, nutrient concentrations, fluorometry, spectrophotometry (chl a, CDOM, PAB's), HPLC, grain size analysis, transmittance/reflectance particle absorption, GC analysis of CH_4, CO_2 and N_2O			Mike Crump (Hamilton) mike.crump@niwa.co.nz 07 856 1739 Chris Cunningham (Christchurch) c.cunningham@niwa.co.nz 03 348 8987
	Elementar Vario ELIII CHN Analyser (Hamilton)	%C and %N content (Total organic carbon (TOC), particulate organic carbon (POC), total organic nitrogen (TON), particulate organic nitrogen (PON))	Solid samples (2-10mg)	Environmental, pollution studies, sediments, ocean & riverine samples, river water quality	Mike Crump mike.crump@niwa.co.nz 07 856 1739
	Becton Dickinson FACSCalibur Flow Cytometer, with 488 nm laser (Microbiology Laboratory Hamilton)	Particle size, complexity and fluorescence, cell sorting	Phytoplankton & bacteria in fresh and seawater samples	Enumerating & characterising bacterial & phytoplankton populations, quantifying DNA content	Julie Hall j.hall@niwa.co.nz 04 386 0322

	GC-MS, LC-MS, HPLC, NMR (Organic Chemistry Laboratory, Hamilton)	Organic contaminant concentrations (DDT, PAH, PCB's)			Greg Olsen (Hamilton) g.olsen@niwa.co.nz 07 856 1781
GNS, National Isotope Centre, Wellington					
GVI Isoprime (Frodo) Dual inlet & continuous flow with hydrogen collectors	Performed manually directly on dual inlet	$\delta^{13}C$, $\delta^{18}O$	Small CO_2 gas samples	Small CO_2 samples from radiocarbon laboratory	Valerie Claymore v.claymore@gns.cri.nz 04 570 4645 Andy Phillips a.phillips@gns.cri.nz 04 570 4620 **Please note that Valerie Claymore no longer works at GNS**
	GVI Aquaprep (Galadriel) Gilson 60 position autosampler integrated with dual inlet	$\delta^{18}O$	Water	Ice core, palaeoclimate reconstruction, ground water studies, geothermal system studies	
GVI Isoprime (Sam) Continuous flow with hydrogen collectors, dual reference box, diluter and hydrogen collectors	Eurovector PyrOH (Eowyn) elemental analyser with LAS300 100 position liquid autosampler. Reduction by direct injection of water over hot chromium.	δD	Water	Ice core, palaeoclimate reconstruction, ground water studies, geothermal system studies	
GVI Isoprime (Rosie) Continuous flow with hydrogen collectors, dual reference box, diluter box	HEKAtech HTEA (Borimier) High temp. furnace with 80 position solid autosampler	$\delta^{18}O$, δD	Minerals, sulphates, organic samples	Geological, ecological, and forensic studies	
	CSIA – Agilent GC with combustion furnace (Aragorn) GC combustion system with 100 position autosampler for liquid injection and gas inlet	$\delta^{13}C$	Geothermal gases, hydrocarbons,	Geothermal studies	
Europa Geo 20-20 (Mari) Dual inlet & continuous flow	Automated 24 port manifold	$\delta^{13}C$, $\delta^{18}O$	Offline combustions of samples from radiocarbon laboratory or from CO_2 produced on laser line	$\delta^{13}C$ & $\delta^{18}O$ values in radiocarbon dated samples, $\delta^{18}O$ in silicates	
	ANCA elemental analyser (Legolas) with 130 position solid sample autosampler	$\delta^{13}C$, $\delta^{15}N$	Solid organic samples	Environmental contamination, sources of air particulate particles, food adulteration, ecological & food web studies, land-water nitrogen transfer, archaeology	

Europa Geo 20-20 (Pippin) Dual inlet & continuous flow	CAPS (Celeborn) Carbonate automated preparation system, 24 vial carousel	$\Delta^{13}C$, $\delta^{18}O$	Calcium carbonate, calcite, aragonite	Palaeoclimate reconstruction, ore deposit studies, forensic applications	
	SEA (Theodin) Sercon sulphur elemental analyser, single combustion furnace with 50 position autosampler	$\delta^{34}S$	Mineral deposits, solid organic samples	Environmental, geothermal systems including black smokers, hydrocarbons	
GNS Offline Sample Preparation Systems					
Europa Geo 20-20 (Mari) Dual inlet & continuous flow	Br5F Laser Fluorination Line	$\delta^{18}O$	CO_2 produced from Silicates	Geological studies	Valerie Claymore v.claymore@gns.cri.nz 04 570 4645 Andy Phillips a.phillips@gns.cri.nz 04 570 4620 **Please note that Valerie Claymore no longer works at GNS**
	DIC vacuum Line	$\delta^{13}C$	CO_2 produced from dissolved inorganic carbon in water	Groundwater and ecological studies	
	Combustion Line	$\delta^{13}C$	CO_2 produced from low carbon organic samples	Paleoecology, soil and sediment studies	
GNS Complementary Facilities					
	Radiocarbon laboratory	^{14}C dating	Organic material, water	Archaeology, oceanography, environmental studies, groundwater research, geology and antiquities	Christine Prior c.prior@gns.cri.nz 04 570 4644
	Terrestrial Isotope Biogeochemistry laboratory Agilent 7890/5975C GCMS	Biomarker tracers in soils, sediments and dissolved phases	Solid (pyrolysis or thermochemolysis), liquid or gas injection	Biogeochemistry and organic geochemistry	Troy Baisden t.baisden@gns.cri.nz 04 570 4653
	Water dating laboratory	Tritium ^{3}He, CFC, SF6 and ^{32}Si	Water	Radioactive dating of groundwater samples, identifying sources, pathways and resources of ground waters, land use impacts	Uwe Morgenstern u.morgenstern@gns.cri.nz 04 570 4652
	Cosmogenic Isotope and Radiochemistry Lab	^{10}Be, ^{137}Cs, ^{210}Pb, ^{226}Ra and other U series isotopes	Rainwater, snow, hydrothermal deposits, sediments	Dating of sediments, hydrothermal deposits, manganese nodules, surface exposure, sediment accumulation studies, study of atmospheric precipitation, loess formation.	Robert Ditchburn r.ditchburn@gns.cri.nz 04 570 4648
	Ion Beam Laboratory	Ion beam analysis – all elements in the periodic table	Air, water, soil, dust, rocks, sediment, minerals, fauna, flora	Source of air, water & soil pollution	Andreas Markwitz a.markwitz@gns.cri.nz 04 570 4785

	Water and gas laboratory	Chemical preparation of fluid and gas samples for stable isotope analysis	Fluids & gases	Geology, geochemistry, environmental chemistry	Bruce Mountain b.mountain@gns.cri.nz 07 376 0137
	Minerals laboratory	XRD, thin section & rock and mineral preparations	Minerals	Geology, environmental	John Simes j.simes@gns.cri.nz 04 570 4896
	Palynology laboratory	Pollen analysis	Terrestrial and marine sediments	Palaeoceanography, dating, stratigraphy	Dallas Mildenhall d.mildenhall@gns.cri.nz 04 570 4696
Victoria University of Wellington, School of Geography, Environment & Earth Sciences					
Nu Instruments Nu plasma HR multiple collector inductively coupled mass spectrometer (MC-ICPMS)		Isotopes of Li, B, O, Mg, Si, V, Cr, Fe, Ni, Cu, Zn, Mo, Ti, Sr, Nd, Pb, Hf, U and Th	Solutions and solid inorganic and organic samples	Environmental chemistry, geochemistry, cosmochemistry, palaeoceanography, climatology, biological sciences	Joel Baker joel.baker@vuw.ac.nz 04 463 5493
Victoria University of Wellington Preparation & Complementary Facilities					
Wet chemistry laboratory plus an ultraclean chemical separation laboratory with 4 pico-trace laminar flow workstations	Trace element sample preparation	Solutions and solid inorganic and organic samples		Joel Baker joel.baker@vuw.ac.nz 04 463 5493	
Agilent 75000CS inductively coupled mass spectrometer	Trace element concentrations	Solutions			
New Wave Deep ultraviolet laser ablation (193 nm solid state)	Trace element concentrations	In situ solid samples			
New Zealand Ice Core Research Laboratory: Joint GNS Science, Victoria University & NIWA Facility					
	Ice Core Research Lab, in purpose built building located at GNS Science, Gracefield site	Ultra clean lab, working freezer (-18°C), storage freezer (-35 °C) with capacity to store 2000m of ice cores	Ice cores	Climate change, atmospheric gases, trace gases	Nancy Bertler n.bertler@gns.cri.nz 04 570 4631

Isotrace, University of Otago, Dunedin					
Thermo Delta V Advantage Continuous flow	TCEA (Thermal Conversion – Elemental Analyser)	$\delta^{18}O$, δD	"Bulk" solids and liquids	Point of origin, migration studies,	Russell Frew rfrew@chemistry.otago.ac.nz 03 479 7913 Robert Van Hale robertv@chemistry.otago.ac.nz 03 479 7915
		$\delta^{18}O$, δD	Water	Hydrology, doubly labelled water experiments – energy expenditure, body water volume	
		$\delta^{18}O$	Bone phosphate	Archaeology, migration studies	
ThermoFinnigan Delta^{Plus} XP Continuous flow	GC-C-IRMS	Compound specific stable isotopes of any one of δD, $\delta^{18}O$, $\delta^{13}C$ or $\delta^{15}N$	Hydrocarbons, lipids, fatty acids (as methyl esters – FAMEs), resin acids, amino acids, polyaromatic hydrocarbons (PAHs)	Food web analysis, pollution tracking, sedimentary inputs	
Thermo Delta^{Plus} Advantage Continuous flow	Thermo "GasBench"	$\delta^{18}O$, $\delta^{13}C$, $\delta^{15}N$	Water, CO_2 (g or aq), nitrogen (g or aq), N_2O (g or aq), oxygen (g or aq)	Hydrology, nitrogen biogeochemistry, labelled denitrification, CO_2 in breath – human metabolism, sports performance	
		$\delta^{13}C$, $\delta^{18}O$	Carbonate solids		
Micromass IsoPrime Continuous flow	EuroVector Elemental Analyser	$\delta^{13}C$, $\delta^{15}N$, $\delta^{34}S$	"Bulk" solids and liquids		
Europa Scientific 20/20 Continuous flow	Carlo Erba Elemental Analyser	$\delta^{13}C$, $\delta^{15}N$	"Bulk" solids, liquids and gases		
VG Sira 10 Dual Inlet, 10 port manifold	CO_2 – Water Equilibration Manifold	$\delta^{13}C$	$CO_{2(g)}$	High precision water isotope measurements – hydrology, oceanography, meteorology	
VG Sira 12 Dual inlet, twin analysers, 20 port manifold	In storage	-	-	-	
Isotrace Offline Sample Preparation Systems					
Thermo Delta^{Plus} XP	Thermo Precon	$\delta^{18}O$, $\delta^{13}C$, $\delta^{15}N$	CO_2, N_2O and CH_4	Trace gas separations	Russell Frew rfrew@chemistry.otago.ac.nz 03 479 7913 Robert Van Hale robertv@chemistry.otago.ac.nz 03 479 7915
Thermo Delta^{Plus} XP	Thermo automated cold/chemical traps	Any	CO_2, N_2O and CH_4	Trace gas separations	
Thermo Delta^{Plus} XP	Gas handling rig for Giggenbach bottles/gas bags etc.	Any	CO_2, N_2O and CH_4	Clean transfer form field-sampling containers	

Thermo Delta V Advantage	Zero-blank autosampler for solid combustions	Any	Air-sensitive materials, low nitrogen materials	Analysis of natural polymers or other hygroscopic substances, N in sediments	
Any	CTC autosamplers	Any	Fluids	Automated sample preparation	
VG Sira 10	Water-CO_2 equilibration manifold, 48 samples	$\delta^{13}C$, $\delta^{18}O$	Water, carbonates		
Any	Hydrogen equilibration rig	δD	Bound H of natural polymers		
Centre for Trace Element Analysis, University of Otago					
Nu Instruments Nu plasma HR multiple collector inductively coupled mass spectrometer (MC-ICPMS), housed in class 1000 clean laboratory		Isotopes of Li, B, O, Mg, Si, V, Cr, Fe, Ni, Cu, Zn, Mo, Cd, Ti, Sr, Nd, Pb, Hf, U and Th	Solutions and solid inorganic and organic samples, e.g. Cu, Zn, Cd, Pb, U isotopic analysis of waters & digested materials, high precision U-series isotopic analysis of digested samples – e.g. corals, speleothems, meteorites	Meteorites & solar system studies (U), Southern Ocean nutrient cycling & ocean-atmosphere CO_2 partitioning (Cd), heavy metal contamination in urban environments (Pb, Cd, Zn), riverine pollution & geochemical weathering cycles in catchment areas (Sr, U, Pb), marine & riverine fish migration (Sr in otoliths), anthropology - human migrations in Pacific (Sr in teeth), SL reconstruction from fossil coral reefs (U- series dating)	Claudine Stirling cstirling@chemistry.otago.ac.nz 03 479 7932
Centre for Trace Element Analysis, University of Otago Preparation & Complementary facilities					
	Class 10 clean chemistry laboratory				Claudine Stirling cstirling@chemistry.otago.ac.nz 03 479 7932
Quadrupole ICP-MS (Q-ICP-MS) Agilent 7500 cs/ce, housed in class 1000 clean laboratory		Trace element concentrations	Solutions, elemental quantification of waters and digested samples, e.g. rocks, soils, biological materials	Riverine pollution & geochemical weathering cycles in catchment areas, ultra-low concentrations of trace metals in mineral quartz – tracking sources & movement sediment from modern & ancient mountain belts, bio-accumulation pathways of Cd in oysters	
Quadrupole ICP-MS (Q-ICP-MS) Agilent 7500 cs/ce, housed in class 1000 clean laboratory	Laser ablation system	Trace element concentrations	In situ solid samples, e.g. Sr and U isotopic analysis of solid samples		

Isolytix Ltd., Dunedin					
Europa Scientific 20-20 Continuous flow	Europa Scientific Roboprep-CN Elemental analyser	$\delta^{13}C$, $\delta^{15}N$	Plants, soils, animal metabolites, marine filters, food stuffs, organic liquid, ethanol	Ecological, agricultural, forensic, medical, authentication of foodstuffs	Ken Neal ken@isolytix.com 03 478 1171 021 824 388 New Zealand and Australian agent for Iso-Analytical based in Crewe, UK.
	Europa Scientific Roboprep-CN Elemental analyser	$\delta^{13}C$	Kerogen, whole oil or oil fraction	Geochemical, geological	
	Sercon Elemental analyser	$\delta^{34}S$	Plants, soils, animal metabolites, marine filters, food stuffs, whole oil or oil fraction, sulphate, sulphide or sulphur-bearing minerals, sulphate in water samples	Ecological, agricultural, forensic, geochemical, geological	
	Europa Scientific ANCA-GSL Elemental analyser	$\delta^{18}O$, δD	Organic solids and liquids e.g. animal tissue, nitrate, cellulose, food stuffs, ethanol, whole oil or oil fraction	Ecological, agricultural, forensic, geochemical, geological, authentication of foodstuffs	
	Europa Scientific ANCA-G Elemental analyser + Gilson 22 autosampler	$\delta^{13}C$, $\delta^{18}O$	Carbonates (by acid digestion), bicarbonate in blood, CO_2 in breath	Geochemical, geological, medical	
Europa Scientific 20-20 Continuous flow	Europa Scientific ANCA-G Elemental analyser + Gilson 222 autosampler	$\delta^{13}C$	Amino acid by decarboxylation	Nutrition research	
Europa Scientific 20-20 Continuous flow	HP5890 + proprietary GC-C combustion interface	C1-C4 hydrocarbons and CO_2	Landfill, drilling gases	Geochemical, environmental	
Europa Scientific Geo 20-20	Europa Scientific ANCA-GSL + Gilson 222 autosampler	$\delta^{18}O$ and δD by equilibration	Fresh water and seawater, total body water, doubly-labelled water, water in foodstuffs and drinks	Ecological, agricultural, geochemical, geological, medical, authentication of foodstuffs	

University of Canterbury, Department of Geological Sciences					
Thermo Delta V plus Continuous flow	Thermo TC/EA with zero-blank autosampler	$\delta^{18}O$, δD	Solid and liquid inorganics	multiple	Travis Horton travis.horton@canterbury.ac.nz 03-364-2987 x 7734
	Thermo GasBench II, with carbonate option	$\delta^{18}O$, $\delta^{13}C$	Carbonates, air, DIC, water	multiple	
	Costech ECS 4010 with zero-blank autosampler	$\delta^{13}C$, $\delta^{15}N$	Solid organics	multiple	
Lincoln University, Agricultural and Life Sciences Division					
PDZ Europa (Sercon) Continuous flow mass spectrometer triple collector, plus collectors for H isotopes	PDZ Europa (Sercon) Elemental analyser	$\delta^{13}C$, $\delta^{15}N$	Solid samples, e.g. soil, plants	Agricultural, N and C cycle in soils and plants, greenhouse gas emissions	Tim Clough clought@lincoln.ac.nz 03-325-2811 x 8360
	Pyrolysis furnace as supplied at 1200°C	$\delta^{13}C$ in CH_4	Gas samples from pastures and wetlands	C cycle, CH_4 emissions	
	Gilson autosampler (standard liquid autosampler adapted) & PDZ Europa (Sercon) trace gas unit	$\delta^{13}C$, $\delta^{15}N$, $\delta^{18}O$	Gases: N_2O, N_2, CO_2 and CH_4	Greenhouse gas emissions, labelled uptake/emission incubations	
		$\delta^{15}N$	Inorganic N species by conversion to N_2O	N cycling, rivers and ground waters, agricultural streams & runoff, fate of fertilisers and excreta	
		N isotopomer analysis	N_2O	Climate change, gas emissions	
Landcare Research, Lincoln					
	Campbell Scientific Instruments Trace Gas Analyser TGA100A TDL (Tuneable diode laser) and absorption spectroscopy 16 inlets (35 sec/inlet), field portable and instant online delta ratios	$\delta^{13}C$, $\delta^{18}O$	Gas concentrations of CO_2 as isotopologues $^{12}C^{16}O^{16}O$, $^{13}C^{16}O^{16}O$, $^{12}C^{16}O^{18}O$ (not isotope ratios) in ambient air	Greenhouse gases – source, sinks & controlling processes, sub-cellular leaf and ecosystem processes, biological & environmental interactions, eddy covariance and gradient flux measurements	John Hunt HuntJ@landcareresearch.co.nz 03 321 9703 Margaret Barbour BarbourM@landcareresearch.co.nz 03 321 9608

AgResearch Ltd., Palmerston North					
ThermoFinnigan Delta V plus	GC linked to high temperature pyrolysis	δD, $\delta^{18}O$, $\delta^{13}C$, $\delta^{15}N$	CSI amino acids	Nutrition	Michael Tavendale michael.tavendale@agresearch.co.nz 06 351 8107
Waikato Stable Isotope Unit (WSIU), University of Waikato, Hamilton					
Europa Scientific (Sercon) 20-20 Blue Box IRMS	ANCA-NT GSL (Gas Solids Liquids) preparation unit	$\delta^{13}C$, $\delta^{15}N$	Solid and liquid samples	Biological and environmental for terrestrial and freshwater ecology, food webs	Facility Director: Brendan Hicks b.hicks@waikato.ac.nz 07 838 4661 Facility Manager: Anjana Rajendram a.rajendram@waikato.ac.nz or isotope@waikato.ac.nz 07 838 4361
PDZ Europa (Sercon) 20-20	ANCA-NT GSL (Gas Solids Liquids) preparation unit	$\delta^{13}C$, $\delta^{15}N$	Gas samples	Medical – breath-test analysis	
Waikato Stable Isotope Unit Preparation & Complementary Facilities					
Europa Scientific 20-20 Blue Box IRMS and PDZ Europa 20-20	Retsch MM 2000 ball mill for sample grinding	Homogenisation in preparation for sample analysis ($\delta^{13}C$, $\delta^{15}N$, C & N concentrations)	Solid samples	Biological and environmental	Facility Director: Brendan Hicks b.hicks@waikato.ac.nz 07 838 4661 Facility Manager: Anjana Rajendram a.rajendram@waikato.ac.nz or isotope@waikato.ac.nz 07 838 4361
	LECO TruSpec CN Determinator with coupled autosampler for liquids	C & N concentrations	Solid and liquid samples	Biological and environmental	
University of Waikato, Department of Chemistry and Department of Earth & Ocean Sciences					
PDZ Europa (Sercon) 20-20	DI Carbonate Acid Preparation System (CAPS)	$\delta^{13}C$, $\delta^{18}O$	Carbonate samples, CO_2 gas samples	Earth science, geochemistry	Chris Hendy c.hendy@waikato.ac.nz 07 838 4381 Facility Operator: Steve Cooke steve@waikato.ac.nz 07 858 5163

University of Waikato, School of Science & Engineering					
Perkin Elmer SCIEX Elan DDRC II, Inductively Coupled Mass Spectrometer (ICP-MS) with automatic liquid sampler	Dynamic Reaction Cell Technology to eliminate isobaric interferences, autosampler for solutions, HPLC separation for speciation determination	Major & trace element concentrations and chemical speciation, strontium, lead, uranium isotope abundances	Water & solid samples (after acid digestion)	Earth science, biological, chemical, environmental, forensic	Chris Hendy c.hendy@waikato.ac.nz 07 838 4381 Facility manager: Steve Cameron 07 838 4395 stevecam@waikato.ac.nz
Perkin Elmer SCIEX Elan DDRC II, Inductively Coupled Mass Spectrometer (ICP-MS) with automatic liquid sampler	New Wave Laser UP 213-A/F Laser Ablation System	Major & trace element concentrations, strontium, lead, uranium isotope abundances	Solid samples (laser ablation, down to 10nm size)	Earth science, biological, chemical, environmental, forensic	Chris Hendy c.hendy@waikato.ac.nz 07 838 4381 Facility manager: Steve Cameron 07 838 4395 stevecam@waikato.ac.nz
Helium mass spectrometry	Home built helium extraction bench with quadrupole mass spectrometer	Helium (^4He) abundance	Single crystal apatite or zircon by laser heating, also multiple apatite furnace heating	Geochronology and thermochronology	Peter J.J. Kamp pjjk@waikato.ac.nz 07 838 4876 Facility Operator: Steve Cooke steve@waikato.ac.nz 07 858 5163
Radiocarbon dating laboratory	Quantulus spectrometers, AMS graphitisation	^{14}C	solid samples	Carbon dating archaeological, geological samples	Alan Hogg alan.hogg@waikato.ac.nz 07 838 4707

New/Ongoing Developments

Institute	Ongoing/planned developments	Scientist
NIWA	Picarro Wavelength-Scanned Ring Down Spectroscopy (WS-CRDS) for measurement of $\delta^{18}O$ and δD for hydrological applications	Max Gibbs m.gibbs@niwa.co.nz 07 856 1773
GNS, NIL	Agilant GC with combustion furnace and pyrolysis system attached to combi-pal for pyrolysis of organics. Analysis of δD also planned on this system. Compound specific isotope analysis for D/H and N Zeroblank autosampler	Valerie Claymore v.claymore@gns.cri.nz 04 570 4645 Andy Phillips a.phillips@gns.cri.nz 04 570 4620
	Automated simultaneous ^{13}C measurement and CO_2 collection during ^{14}C radiocarbon analysis	Valerie Claymore, Andy Phillips & Nancy Beavan n.beavan@gns.cri.nz 04 570 4650
	Isotopic analysis of nitrates in water	Troy Baisden t.baisden@gns.cri.nz 04 570 4653
Isotrace	Miniaturisation for C and N analysis Equipment/developments to deal with post-helium economy	Russell Frew rfrew@chemistry.otago.ac.nz 03 479 7913 Robert Van Hale robertv@chemistry.otago.ac.nz 03 479 410
Isolytix Ltd.	Hybrid compound specific IRMS system linked to scanning mass spectrometer for simultaneous IRMS identification work	Ken Neal ken@isolytix.com 03 478 1171 021 824 388
Waikato Stable Isotope Unit	Vario EL Elemental Analyser Isoprime stable isotope mass spectrometer	Facility Director: Brendan Hicks b.hicks@waikato.ac.nz 07 838 4661
University of Canterbury	Development of fluid inclusion isotopic analyses through utilization of an *Amsterdam Device* (crushing device) and thermal decrepitation. Development of both chemical and bacterial (i.e. denitrifier) methods for oxygen isotope analysis of nitrate.	Travis Horton travis.horton@canterbury.ac.nz 03-364-2987 x 7734

Current Gaps in Technology (Wish lists)

Institute	Technology Gaps/Wish list	Scientist
NIWA	Upgrade aging instruments (ThermoFinnigan MAT 252 dual inlet mass spectrometer, including Kiel carbonate preparation device, and continuous flow Delta ^{Plus} mass spectrometer) New instruments will enable dedicated S isotope analytical capability to be set up to analyse $\delta^{34}S$ in organic materials (current facility requires switching single instrument between C/N and S which is usually impractical). Additional requirements of new facility: Capability to analyse $\delta^{13}C$ of DIC, dissolved CH_4, $\delta^{18}O$ and δD in waters ($\delta^{18}O$ and δD in waters most likely covered by collaboration with GNS and field use of Picarro system above) Peripheral GasBench GC facility for measurement of $\delta^{15}N$ in NO_2, N_2O and low level organic compounds Compound specific amino acids, fatty acids, alkanes, alkenes, polycyclic aromatics (PAH's) in organic materials? (collaborate and discuss with Isotrace)	Gordon Brailsford g.brailsford@niwa.co.nz 04 386 0393 Sarah Bury s.bury@niwa.co.nz 04 386 0347 Helen Neil h.neil@niwa.co.nz 04 386 0375 Max Gibbs m.gibbs@niwa.co.nz 07 856 1773
	$\delta^{34}S$ in sulphates	
GNS, NIL	Total carbon analysis in air particulate matter by thermal optical reflectance	Perry Davy p.davy@gns.cri.nz 04 570 4688
Victoria University	Analysis of isotopologues of CO_2 in carbonates enabling palaeotemperature reconstruction without any knowledge of the isotopic composition of the liquid the carbonate was in equilibrium with. (See work coming out of John Eiler's group, Caltech).	Joel Baker joel.baker@vuw.ac.nz 04-463-5493
Isotrace	Low concentrations in C and N mineral phases – geological nitrogen	Russell Frew rfrew@chemistry.otago.ac.nz 03 479 7913 Robert Van Hale robertv@chemistry.otago.ac.nz 03 479 410
	Dissolved trace gases – CH_4, CO, O_2, NH_3 – membrane inlet-IRMS	
University of Canterbury	Multiply substituted isotopologues (MSI) – aka – "clumped" isotope analysis. Travis is not aware of a laboratory in New Zealand that is capable of MSI analysis (at present, only a couple of labs globally are set-up to do this). You need a top-of-the-line instrument (e.g. MAT253) with additional cups in the detector/collector (e.g. two universal triple collectors) providing extreme sensitivity across a range of masses.	Travis Horton travis.horton@canterbury.ac.nz 03-364-2987 x 7734

	Tuneable Diode Laser (TDL) – Los Gatos Research makes these for rapid and inexpensive hydrogen and oxygen isotope analyses on water samples (there are other applications as well). New Zealand is currently lacking a detailed high spatial and temporal resolution isotopic map for meteoric water (à la Irving Friedman's map of the western U.S.; U.S.G.S. OFR-2000-388). This new technology makes construction of such a map much less costly and simpler – although data needs to be collected regularly over decades to capture effects of ENSO, PDO, etc. Costs would easily be two orders of magnitude lower than current GNS rates over the life time of the device (i.e. thousands of water analyses for both oxygen and hydrogen).	
	Gas Chromatography (GC) peripheral – UC does not have one, but other institutions in NZ do. You need one for compound specific analyses – an extremely powerful and novel analytical technique.	
	Mass Independent Fraction (MIF) – no lab in NZ is pursuing mass independent fractionation processes (to do this you need a high end instrument – none of the isotope ratio mass spec's in NZ satisfy this requirement for MIF).	
Lincoln University	GC-MS capacity for organic compounds	Tim Clough clought@lincoln.ac.nz 03-325-2811 x 8360
Waikato Stable Isotope Unit	Perkin Elmer ICP-MS for dedicated laser ablation use	Facility Director: Brendan Hicks b.hicks@waikato.ac.nz 07 838 4661
	Upgrade of aging MS instruments	

Appendix III Participants' Contact Information

Participants' Contact Information

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