GEOANALYSIS

International Association of Geoanalysts — IAG

2018

10th International Conference on the Analysis of Geological and Environmental Materials

Macquarie University
Sydney, Australia

GEOANALYSIS 2018
8-13 July
SYDNEY

MACQUARIE University

CCFS

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Speaker Ready Room

Meeting room, Lvl 1, 12 Wally's Walk 127 (E7A)

PC terminals will be available for delegates to review presentations and check emails.

A4 colour printing available.

Contact Information

Dorrit Jacob: 0459900499 (organiser)

Tim Murphy: 0401137008 (Company exhibits)

WIFI ACCESS

Network Name: Macquarie Events Browse to **www.mq.edu.au** where the guest will be redirected to insert the passcode: **gc2018**

Registration and Help Desk

11 Wally's Walk 210 (E5A)

Sunday: 15:30–17:00

Graduation Hall, 12 Wally's Walk (E7B)

Monday: 08:15–17:00

Tuesday: 08:15–17:00

Wednesday: 09:00–14:00

Thursday: 08:00–17:00

Company Exhibition

Graduation Hall, 12 Wally's Walk (E7B)

Tuesday: 09:00–19:30

Wednesday: 09:00–17:00

Thursday: 09:00–17:00

Poster Presenters in attendance

Graduation Hall, 12 Wally's Walk (E7B)

Monday: 15:30–17:00

Thursday: 15:20–17:00

Friday: 14:30–15:30

Posters on Display

Graduation Hall, 12 Wally's Walk (E7B)

Monday: 12:00–19:30

Tuesday: 09:00–19:30

Wednesday: 09:00–17:00

Thursday: 09:00–17:00

Friday: 09:00–15:30

000 EMERGENCY

Macquarie Lighthouse, built in 1816 at the entrance of Sydney Harbour, was the first lighthouse in Australia and features in the arms of Macquarie University.

Dear Delegate,

Welcome to the 10th International Conference on the Analysis of Geological and Environmental Materials, *Geoanalysis 2018*. We are honoured and delighted to have you here at Macquarie University in beautiful Sydney for a week of exciting science and discussions.

Macquarie University, named after Major General Lachlan Macquarie, Governor of New South Wales between 1810 and 1821, is only 50 years young and prides itself in being dynamic, innovative and close to industry. The Department of Earth and Planetary Sciences reflects this spirit through its strength in research in the fields of geochemistry, geophysics and marine science, powered by 23 staff, a third of which are women.

The Department hosts Macquarie GeoAnalytical (MQGA), the major geoanalytical laboratory in the Sydney Basin, one of the leading laboratories in Australia and renowned worldwide. MQGA is firmly based on the ground-breaking work carried out under its former name, the Geochemical Analysis Unit (GAU), in the National Key Centre GEMOC and the ARC Centre of Excellence for Core to Crust Fluid Systems (CCFS), both directed by Sue O'Reilly. This enabled the work of analytical pioneers such as Norman Pearson, Bill Griffin and Simon Jackson.

Major breakthroughs in method development, for example the *in-situ* analysis of Os isotopes in sulfides (*Pearson et al., 2002; Alard et al., 2002*), *in-situ* analysis of Hf isotopes (*Griffin et al., 2000*) and *in-situ* analysis of U-Pb in zircon (*Jackson et al., 2004*) came from this laboratory. While driven by great minds in academia, none of the success would have been possible without the support of and close collaboration with instrument manufacturers: MQGA is very proud of its traditionally strong links with NU, Cameca and Agilent as well as its newest collaboration with ThermoFisher Scientific.

Pushing the boundaries of analytical methodology, both in spatial resolution and in precision, would be impossible without dedicated specialists and appropriate, matrix-matched high-quality reference materials. The International Association of Geoanalysts (IAG) provides an umbrella for the vibrant community of these specialists from academia and industry as well as for students striving to become our colleagues in the future.

Geoanalysis 2018 is proud to provide this year's forum to learn, to teach and to discuss the newest developments in geoanalytical research in a collegial and inclusive atmosphere on the Macquarie University campus.

This conference was made possible by the generous support of the companies that manufacture many of the excellent analytical instruments that geoanalysts use in their work.

Welcome to Macquarie University, and enjoy the conference!

Professor Dorrit Jacob
Director Macquarie GeoAnalytical
Chair Organising Committee Geoanalysis 2018

Geoanalysis 2018 Organising Committee

Dorrit Jacob	*Chair, Scientific program*	dorrit.jacob@mq.edu.au
Damian Gore	*Deputy Chair, Scientific program, Sponsorship coordinator*	damian.gore@mq.edu.au
Elena Belousova	*Scientific program*	elena.belousova@mq.edu.au
Thomas Meisel	*Scientific program*	thomas.meisel@unileoben.ac.at
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Yolima Perez	*Logistics, Event manager*	yolima.perezmoyana@mq.edu.au

1   11 Wally's Walk 210 (E5A)
2   Graduation Hall, 12 Wally's Walk (E7B)
3   Mason Theatre, 12 Wally's Walk (E7B)
4   Atrium, 12 Wally's Walk (E7A)
5   Macquarie Campus Commons
6   Train Station
7. Australian Hearing Hub
8. Robert Menzies College
9. Travelodge
10. MGSM Hotel
11. Macquarie Shopping Centre
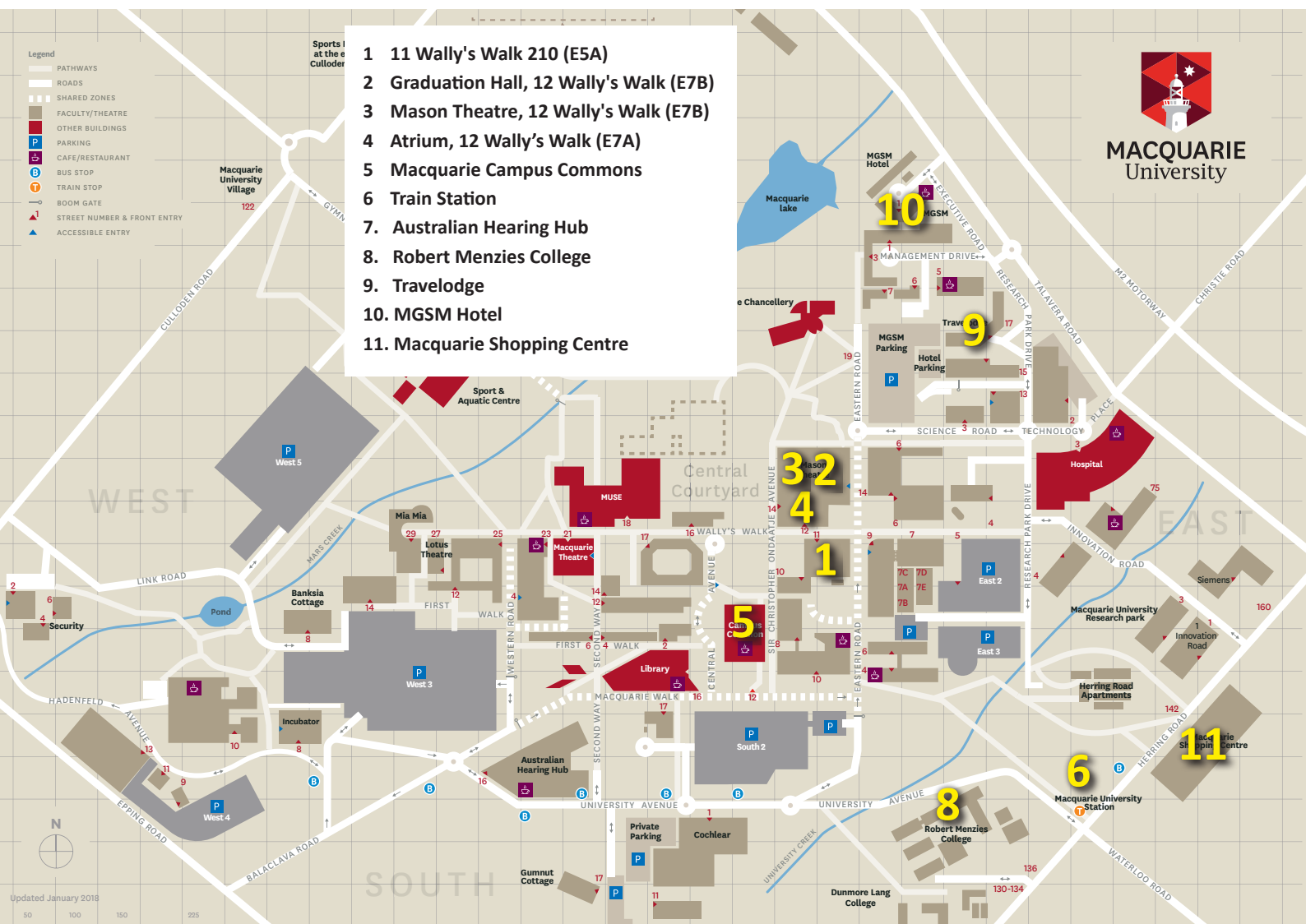
Our Sponsors

We would like to thank all of our generous Sponsors for their contributions:

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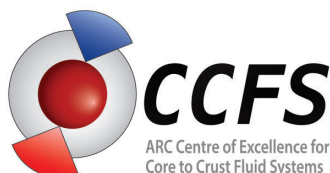
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GEO ANALYSIS 2021

1st to 6th AUGUST │ FREIBERG

Will be hosted by

- **Helmholtz Institute Freiberg for Resource Technology**
- **TU Bergakademie Freiberg**
- **Eurofins Umwelt Ost GmbH**

Firmly grounded in the long tradition of mining combined with the newest scientific results starting with Agricola's *De re metallica* and the foundation of the Bergakademie in 1765, today Freiberg is a centre of vivid and future-oriented research in all fields of natural resources. This is being supported by research institutions, university and industry represented by our trio of proud organisers.

Don't miss an interesting program of scientific, cultural and geological activities!

More information on **geoanalysis2021.de**

Helmholtz Institute Freiberg for Resource Technology

PROGRAM

Workshops

11 Wally's Walk 210 (E5A)

09:00   Registration for Workshops open

10:00   **Workshop I - Transportable Analytical Instruments and Material Analysis**

Christabel Brand (Portable XRF Services), Andrew Somers (SciAps), Maurice Wicks (AXT)

Theory and application of transportable analytical instruments for elemental and mineralogical applications for industry and research

Transportable analytical instruments are now mainstream tools for diverse applications within industry and research. This workshop will provide a theoretical and practical background with applied examples for using "*off the shelf*" transportable analytical instruments for elemental and mineralogical compositions.

Topics to be covered include sample preparation, instrument configurations and settings, safe operation, understanding qualitative and quantitative data and QA/QC. Case studies will be presented and discussed. Participants are encouraged to bring 3-5 slides illustrating some of their own applications, and challenges, pertaining to transportable elemental and mineralogical analyses.

11:30   *Morning Tea Break*

13:00   *Lunch Break*

14:00   **Workshop II - Application of LA-ICP-MS/MC-ICP-MS to exploration needs**

William Powell (Rio Tinto), Elena Belousova, Romain Tilhac, Stephen Craven (CCFS, Macquarie University)

Theory and application of Laser Ablation ICP-MS and MC-ICP-MS for elemental analysis and isotopic dating - applications for industry and research

This workshop will provide a theoretical and practical background with applied examples for the application of Laser Ablation ICP-MS methods to exploration geology and related fields in industry and research.

Topics to be covered include sample preparation, targeted sampling strategy, introduction to the *TerraneChron®* method (integrated U/Pb, Hf and trace element analysis of zircon), U/Pb dating of accessory minerals. Case studies will be presented and discussed. Participants are encouraged to bring 3-5 slides illustrating some of their own applications, and challenges, pertaining to the topics of this workshop.

15:30   *Rio Tinto Afternoon Tea Break*
         Registration for Conference opens

17:00   End of Workshop II
         Registration for Conference closes
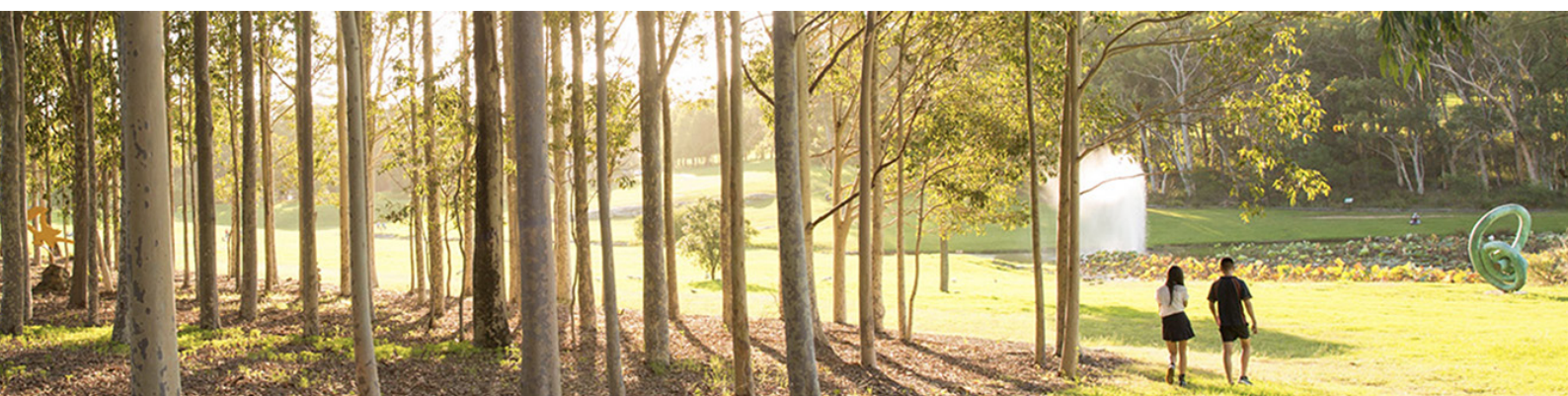
Scientific Sessions

08:15 **Graduation Hall, 12 Wally's Walk (E7B)**
Registration for Conference
Posters set up

Mason Theatre, 12 Wally's Walk (E7B)

09:00 **Conference Opening**
Professor Sakkie Pretorius, *Macquarie University Deputy Vice Chancellor*
Associate Professor Thomas Meisel, *President International Association of Geoanalysts (IAG)*
Professor Dorrit Jacob, *Chair Organising Committee Geoanalysis 2018*

Session 1 - Reference Material Production and Applications
Mason Theatre, 12 Wally's Walk (E7B)
Chairperson: Thomas Meisel

09:30 **Keynote Presentation**
Development of Geochemical reference materials new directions new challenges
Stephen Wilson
U.S. Geological Survey, Crustal Geophysics & Geochemistry Science Center, Denver, CO, USA

10:00 **Homogeneous pressed powder pellets as new MRMs for *in-situ* microanalytical techniques**
Dieter Garbe-Schönberg[1], S. Müller[1], S. Nordstad[1], L. Schönberg[1], M. Wiedenbeck[2] and A.D. Renno[3]
[1]CAU Kiel University, Geosciences, Kiel, Germany
[2]GFZ Helmholtz Zentrum Potsdam, Potsdam, Germany
[3]Helmholtz Zentrum Dresden-Rossendorf, Dresden, Germany

10:20 **Using robust plasma conditions to examine preferred values for BCR-2G and ATHO-1G relative to NIST glasses**
Ivan Belousov[1], Leonid Danyushevsky[2], Jay Thompson[2], Michael Shelley[3] and Paul Olin[2]
[1]TMVC Research Hub, University of Tasmania, Private Bag 79, Hobart, TAS 7001, Australia
[2]CODES, University of Tasmania, Australia
[3]Laurin Technic, Australia

10:40 *Morning Tea Break sponsored by Bruker and Cameca/Nu*

11:10 **Trace elements concentration and distribution in microanalytical reference materials suitable for sulfide and FeNi alloy analyses**
Olivier Alard[1], Rosanna Murphy[1], Yoann Gréau[1], Timothy D. Murphy[1] and Claude Merlet[2]
[1]Dept. of Earth and Planetary Sciences, Macquarie University, Sydney and ARC Centre of Excellence Core to Crust Fluid Systems, Macquarie University
[2]CNRS, Université Montpellier, France

11:30 **Development and characterisation of five new USGS shale reference materials**
Stephen A. Wilson[1] and Justin E. Birdwell[2]
[1]U.S. Geological Survey, Mineral Resources Program, Crustal Geophysics & Geochemistry Science Center, Denver, CO, USA
[2]U.S. Geological Survey, Energy Resources Program, Central Energy Resources Science Center, Denver, CO, USA

11:50   **Apatite reference materials for oxygen and chlorine isotope analysis**
Alicja Wudarska[1,2], Michael Wiedenbeck[2], Frédéric Couffignal[2] and Ewa Słaby[1]
[1]Institute of Geological Sciences of the Polish Academy of Sciences, Warsaw, Poland
[2]GFZ German Research Centre for Geosciences, Potsdam, Germany

12:10   **Study of mineral component in technogenic soils of Irkutsk Region by X-ray electron probe microanalysis**
Olga Yu. Belozerova and G.A. Belogolova
Vinogradov Institute of Geochemistry, Siberian Branch of Russian Academy of Sciences, 664033 Irkutsk, Russia

12:30   **Geo-analysis - Dealing with the analytical challenges with ICP-MS**
Naoki Sugiyama
ICP-MS Product Manager, Agilent Technologies International, Japan

13:00   *Agilent Lunch Break - Atrium, 12 Wally's Walk (E7A)*

Session 2 - Geoanalysis in mining, exploration and remediation
Mason Theatre, 12 Wally's Walk (E7B)
Chairperson: Sarah Gilbert

13:50   **A Super-SIMS for resource technology**
Alex D. Renno[1], Shavkat Akhmadaliev[2], Georgii Belokonov[1], Roman Böttger[2], Johannes von Borany[2], Jens Gutzmer[2], Peter Kaever[3], Markus Meyer[3], Pavol Noga[4], Georg Rugel[1], Collin J. Tiessen[1,5], Jörg Voigtländer[3], Nicole Wagner[3], Michael Wiedenbeck[6], Armin Winter[3], Hao Sheng Wu[1] and René Ziegenrücker[1]
[1]Helmholtz-Zentrum Dresden-Rossendorf, Helmholtz Institute Freiberg for Resource Technology, 09599 Freiberg, Germany
[2]Helmholtz -Zentrum Dresden-Rossendorf, Institute of Ion Beam Physics and Materials Research, 01328 Dresden, Germany
[3]Helmholtz-Zentrum Dresden-Rossendorf, Department of Research Technology, 01328 Dresden, Germany
[4]Slovak University of Technology in Bratislava, Faculty of Materials Science and Technology in Trnava, Advanced Technologies Research Institute, Slovakia
[5]André E. Lalonde AMS Laboratory, University of Ottawa, Canada
[6]Helmholtz-Zentrum Potsdam - Deutsches GeoForschungsZentrum GFZ, Germany

14:10   **Li abundances of magmatic zircons in Eocene-Oligocene porphyry Cu mineral systems of Yunnan, China**
Mei-Fei Chu[1], Yu-ya Gao[2], Qiu-li Li[3,4], Yong-Jun Lu[3,4], Xian-Hua Li[2] and Suzanne Y. O'Reilly[5]
[1]Institute of Oceanography, National Taiwan University, Taiwan
[2]Institute of Geology and Geophysics, Chinese Academy of Sciences, Beijing, P.R. China
[3]Geological Survey of Western Australia, Perth, Australia
[4]CET and CCFS, School of Earth Sciences, the University of Western Australia, Perth, Australia
[5]CCFS and GEMOC, Macquarie University, Sydney, Australia

14:30   **Microanalysis of Cu Slag: Understanding the role of mining waste in ongoing environmental contamination**
Mike Ashelford and Damian Gore
Department of Environmental Sciences, Macquarie University, Sydney

14:50   Group Photo

15:10   *Afternoon Tea Break sponsored by Bruker and Cameca/Nu*

15:30   Poster Session/Lab tour - Graduation Hall, 12 Wally's Walk (E7B)

17:00   **Icebreaker Reception - Atrium, 12 Wally's Walk (E7A)**

Scientific Sessions

Session 2 - Geoanalysis in mining, exploration and remediation
Mason Theatre, 12 Wally's Walk (E7B)
Chairperson: Timothy Murphy

09:00 **Keynote Presentation**
Horizon scanning: An industry perspective on PFAS, an emerging contaminant of concern
Danielle Toase, Jason Lagowski and Ian Ross
Arcadis Australia Pacific

09:30 **Geoanalysis in industry: Rio Tinto**
William Powell and Alan Kobussen
Rio Tinto. 1 Research Avenue, Bundoora 3083, Victoria, Australia

09:50 **A machine-learning interpretation of regional geochemical patterns in northern Australia**
Evgeniy Bastrakov[1], John Wilford[1], Patrice de Caritat[1], Karol Czarnota[1], P. Main[1], Andrzej Wygralak[2] and Sean Chua[1]
[1]Geoscience Australia
[2]Northern Territory Geological Survey

10:10 **Tracing Hg mobility and distribution in a historical mercury mine ecosystem**
Michael Pribil[1], Danny Rutherford[1], JoAnn Holloway[1], Ben McGee[1] and Johanna Kraus[2]
[1]USGS, Denver Federal Center, Denver, CO, 80225
[2]USGS, Fort Collins Science Center, Fort Collins, CO, 80526

10:30 **Comminution and sizing of gold particles in geological samples and reference materials**
Lou Daniel and Drew Parsons
Murdoch University, Perth, Western Australia

10:50 *Morning Tea Break sponsored by Bruker and Cameca/Nu*

11:20 Company Presentations

12:30 *BBQ Lunch Break - Lawn, 12 Wally's Walk (E7A)*

Session 3 - Dating, geochemical mapping and imaging with elements and isotopes
Mason Theatre, 12 Wally's Walk (E7B)
Chairperson: Jon Woodhead

14:00 **Exploring the range of U-containing minerals for geochronological applications**
Elena Belousova[1,2]
[1]Dept. of Earth and Planetary Sciences, Macquarie University, Sydney, Australia
[2]ARC Centre of Excellence Core to Crust Fluid Systems, Macquarie University, Australia

14:20 **U-Pb geochronology of grandite by femtosecond laser ablation high resolution inductively coupled plasma mass spectrometry**
Lei Chen, Hou Kejun, Qiaoqiao Zhu, Yanhe Li and Qian Wang
MLR Key Laboratory of Metallogeny and Mineral Assessment, Institute of Mineral Resources, Chinese Academy of Geological Sciences, Beijing 100037, P.R. China

14:40   **Impact of atmospheric air and laser fluence on accuracy of zircon U-Pb dating**
Jay M. Thompson, Sebastian Meffre, and Leonid V. Danyushevsky
CODES, University of Tasmania

15:00   **G-Chron - IAG's proficiency testing programme for zircon geochronology**
Michael Wiedenbeck
GFZ-Potsdam, Telegrafenberg, 14473 Potsdam, Germany

15:20   *Afternoon Tea Break sponsored by Bruker and Cameca/Nu*

15:45   *Mason Theatre, 12 Wally's Walk (E7B)*
IAG General Meeting

16:15   Poster Session/Lab tour - Graduation Hall, 12 Wally's Walk (E7B)

17:30   *Atrium, 12 Wally's Walk (E7A)*
Thermo Fisher Wine Tasting Evening
Hosted by Macquarie University Deputy Vice Chancellor Prof Sakkie Pretorius

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Wednesday 11th July 2018

Field Trip / Laboratory Demos

08:30   **Field Trip to Boolaroo and the Hunter Valley**
Bus Departure from Macquarie University Campus, return at approx. 19:00

Led by: Mike Ashelford, Tony Morrison, Peter Johnston, Dept. Env. Sci, MQ Uni.

Pasminco lead smelter
1. Boolaroo, the former Pasminco Lead smelter in the North Lake Macquarie area has created extensive environmental contamination through the dissemination of metal-rich slag to public places such as parks, and to local households in the form of garden soil amendments. Dr Tony Morrison (Macquarie University) has spent a lot of effort understanding the mineralogy and chemistry of the slags around Boolaroo. The contamination includes lead, with secondary amounts of copper, zinc, selenium, cadmium and acidic leachate. Tony has found that the distributed slags are leaching metals into the environment. Professor Mark Taylor, Associate Professor Heather Handley and Dr Paul Harvey (all from Macquarie University) have assessed the present-day contamination of the site and found a clear and present environmental risk. The conference field trip will visit the site, and Dr Tony Morrison will give an on-site talk while we have morning tea in the area.

Wine country at Pokolbin
2. After Boolaroo, we will drive to the vineyards of the delightful Hunter Valley, where we will have the opportunity to walk in the wonderful Hunter Valley Gardens (entry fee payable), visit one of a number of vineyards and winemakers (Brokenwood, McGuigan or Tempus Two), and have lunch in one of a number of local cafes. We will briefly discuss some of the geology and geochemical challenges of the area en route to the vineyards. Included in the fee are morning and afternoon tea/refreshments and transport. Lunch and incidentals (entry fee Hunter Valley Gardens, wine tasting or purchases) are not included.

10:00   **Thermo Fisher Laboratory tour**
Led by: Bruce Schaefer, John Creech, Paul Gorjan

14:00   **Agilent Laboratory tour**
Led by: Olivier Alard

Scientific Sessions

Session 3 - Dating, geochemical mapping and imaging with elements and isotopes
Mason Theatre, 12 Wally's Walk (E7B)
Chairperson: Elena Belousova

09:00   *Award Ceremony - Award of an IAG Honorary Fellowship to Prof Jon Woodhead, University of Melbourne*

09:30   **Keynote Presentation**
LA-ICP-MS as an imaging tool: past, present, and future
Jon Woodhead
School of Earth Sciences, The University of Melbourne, VIC 3010, Australia

10:10   **Dating $^{87}Rb/^{86}Sr$ in minerals using LA-ICP-MS, achievements and problems**
Charlotte M. Allen, Karine Harumi Moromizato and Mitchell DeBruyn
CARF Institute for Future Environments, Queensland University of Technology, Brisbane, QLD 4000

10:30   **Testing the limits of *in-situ* Rb-Sr dating of igneous minerals by LA-ICP-QQQ**
Ahmad Redaa[1], Juraj Farkas[1], Sarah Gilbert[1], Thomas Zack[2], Fred Fryer[3], Benjamin Wade[1] and Alan Collins[1]
[1]*The University of Adelaide, SA, Australia*
[2]*University of Gothenburg, Sweden*
[3]*Agilent Technologies Ltd., Australia*

10:50   *Morning Tea Break sponsored by Bruker and Cameca/Nu*

11:10   ***In-situ* Rb-Sr dating of authigenic clays from soils and sediments: Potential and limitations**
Juraj Farkas[1], Grant Cox[1], Morgan Blades[1], Sarah Gilbert[1], Benjamin Wade[1], Alan Collins[1], Thomas Zack[2], Stefan Loehr[3], David Chittleborough[1], Hamed Al Sarakhi[1], Ahmad Reeda[1], Lise Jensen[1] and Fred Fryer[4]
[1]*University of Adelaide, Department of Earth Sciences, Adelaide*
[2]*University of Gothenburg, Department of Earth Sciences, Sweden*
[3]*Macquarie University, Department of Earth and Planetary Sciences, Sydney*
[4]*Agilent Technologies Ltd., Australia*

11:30   **Laser Induced Breakdown Spectroscopy: Mineralogical mapping of drill core and thin sections**
Gary Thompson[1] and Derek H.C. Wilton[2]
[1]*Office of Applied Research and Innovation, College of the North Atlantic, St John's, NL, Canada*
[2]*Department of Earth Sciences, Memorial University, St. John's, NL A1B 3X5*

Session 4 - Instrumentation and new developments
Mason Theatre, 12 Wally's Walk (E7B)
Chairperson: Olivier Alard

11:50   **Atom probe microscopy as a tool for geoanalysis**
David W. Saxey[1], Steven M. Reddy[1,2], Denis Fougerouse[1,2] and William D.A. Rickard[1]
[1]*Geoscience Atom Probe, Advanced Resource Characterisation Facility, John de Laeter Centre, Curtin University, Perth, Australia*
[2]*School of Earth and Planetary Sciences, Curtin University, Perth, Australia*

12:10   **SHRIMP analysis of water abundances and oxygen isotopes**
Trevor Ireland[1], Janaina Ávila[1], Peter Holden[1], Simon Turner[2], Michael Turner[2] and Joerg Hermann[3]
[1]Research School of Earth Sciences, Australian National University, Canberra
[2]Department of Earth and Planetary Sciences, Macquarie University, Sydney;
[3]Institute of Geological Sciences, University of Bern, Bern

12:30   *Lunch Break*

13:50   **High precision, high accuracy silicon isotope determination in quartz using SIMS, by reducing the topography effect**
Yu Liu[1], Xian-Hua Li[1], Qiu-Li Li[1], Xiao-Chi Liu[1], Guo-Qiang Tang[1] and Fang Huang[2]
[1]State Key Laboratory of Lithospheric Evolution, Institute of Geology and Geophysics, Chinese Academy of Sciences, Beijing 100029, P.R. China
[2]CAS Key Laboratory of Crust-Mantle Materials and Environments, School of Earth and Space Sciences, University of Science and Technology of China, Hefei 230026, P.R. China

14:10   **SIMS analyses employing a flood gas tracer**
Michael Wiedenbeck[1], and Ziliang Jin[1,2]
[1]GFZ-Potsdam, Telegrafenberg, 14473 Potsdam, Germany
[2]Arizona State University, School of Earth and Space Exploration, Tempe, AZ 85287, USA

14:30   **2008-2018 – "*The Portable XRF Decade*"**
Aaron Baensch
International Mining Group, Olympus Scientific Solutions, Waltham, MA, USA

15:00   *Afternoon Tea Break sponsored by Bruker and Cameca/Nu*

15:20   Poster Session - Graduation Hall, 12 Wally's Walk (E7B)

16:30   Lab tours

19:00   **Geoanalysis Conference Dinner Cruise, Sydney Harbour** - Cruise end 22:00

Geoanalysis Conference Dinner Cruise, Sydney Harbour - Blue Room Glass Boat Charter
32 The Promenade | King Street Wharf 5

Train
https://transportnsw.info/routes/train

T1 Macquarie Station to Wynyard
(32 min train + 7 min walk)
Walk via York St / Erskine St / The Promenade

T1 Macquarie Station to Town Hall
(34 min train + 14 min walk)
Walk via Druitt St / Kent St / The Promenade

Drive ($ after 5pm)
25-55 min from MQ Uni (trafic dependent)
P1 King street wharf car park - $42, 3 min walk
P2 132 Sussex street car park - $20, 5 min walk
P3 Allianz centre car park - $12, 7 min walk

Bus
https://transportnsw.info/routes/bus

Scientific Sessions

Session 4 - Instrumentation and new developments
Mason Theatre, 12 Wally's Walk (E7B)
Chairperson: Yongsheng Liu

09:00 **Keynote Presentation**
***In-situ* carbon isotope analysis by laser ablation MC-ICP-MS**
Wei Chen, Jue Lu, Shao-Yong Jiang, Kui-Dong Zhao and Deng-Fei Duan
State Key Laboratory of Geological Processes and Mineral Resources, Collaborative Innovation Center for Exploration of Strategic Mineral Resources, Faculty of Earth Resources, China University of Geosciences, Wuhan, 430074, P.R. China

09:30 **Portable heavy metal analyzer based on hydride generation-glow discharge micro-plasma sources**
Chun Yang, Dong He and Zhenli Zhu
State Key Laboratory of Biogeology and Environmental Geology, School of Earth Sciences, China University of Geosciences, Wuhan, P.R. China

09:50 **The limitation of Os isotopic analysis for geological samples using Faraday cups equipped with 10^{13} Ω amplifiers by NTIMS**
Guiqin Wang[1] and Jifeng Xu[2]
[1]*State Key Laboratory of Isotope Geochemistry, GIG, CAS, 510640 Guangzhou, P.R. China*
[2]*China University of Geosciences, 100083 Beijing, P.R. China*

10:10 **Zirconium stable isotope analysis by double-spike MC-ICP-MS**
John B. Creech[1,2], Edward Inglis[1], Zhengbin Deng[1] and Frédéric Moynier[1]
[1]*Institut de Physique du Globe de Paris, 1 rue Jussieu, 75328 Paris cedex 05, France*
[2]*Dept. of Earth and Planetary Sciences, Macquarie University, Sydney, Australia*

10:30 *Morning Tea Break sponsored by Bruker and Cameca/Nu*

11:10 **Determination of Zr and Nb concentrations in chromium matrices using Agilent 8900 ICP-QQQ and Elan ICP-DRC-MS**
Yi-Jen Lai[1,2] and Maria Schönbächler[2]
[1]*Macquarie University GeoAnalytical, Department of Earth and Planetary Sciences, Macquarie University, Sydney, Australia*
[2]*ETH Zurich, Institute of Geochemistry and Petrology, 8092 Zürich, Switzerland*

11:30 **Simultaneous sensitive determination of selenium, silver, antimony, bismuth and lead in geological materials by liquid spray dielectric barrier discharge induced plasma-chemical vapor generation**
Xing Liu, Zhenli Zhu, Huasheng Shan and Zhengyu Bao
State Key Laboratory of Biogeology and Environmental Geology, School of Earth Sciences, China University of Geosciences (Wuhan), Wuhan, Hubei 430074, P.R. China

11:50 **Rapid determination of elements and isotopes in NH_4F-modified geological sample powders by LA-(MC)-ICP-MS**
Wen Zhang, Zhaochu Hu, Yongsheng Liu and Junyi Xie
State Key Laboratory of Geological Processes and Mineral Resources, China University of Geosciences, Wuhan 430074, P.R. China

12:10   **Success and pitfalls during the dissolution of silicate rocks in a microwave oven with a solution of NH_4HF and HNO_3**
Thais Takayassu Magaldi, Margareth Sugano Navarro and Jacinta Enzweiler
Institute of Geosciences, UNICAMP Campinas, SP, Brazil

12:30   *Lunch Break*

13:30   **The effect of laserprobe optical path design on laser ablation of minerals with low melting points**
Leonid Danyushevsky[1,2], Sarah Gilbert[3], Jeremy Thompson[2], Paul Olin[1] and Olivier Alard[4]
[1]TMVC Research Hub, University of Tasmania, PB 79, Hobart, TAS 7001, Australia
[2]CODES, University of Tasmania, Australia
[3]Adelaide Microscopy University of Adelaide, Australia
[4]CCFS/GEMOC, Dept. EPS, Macquarie University, Sydney, Australia

13:50   **High-resolution fs-LA-ICP-MS single shot measurements of Mg/Ca in foraminifers**
Klaus P. Jochum, Ralf Schiebel, Brigitte Stoll, Ulrike Weis, Anna Jentzen and Gerald H. Haug
Climate Geochemistry Department, Max Planck Institute for Chemistry, 55020 Mainz, Germany

14:10   **Geoanalysis 2021 in Freiberg, Germany**
Axel Renno

14:20   Closing Remarks

14:30   Poster Session - Graduation Hall, 12 Wally's Walk (E7B)

15:30   **Closing Drinks Reception Port Botany Café and UBar**
Macquarie Campus Commons

Post-Conference Field Trip

Saturday 14th - Monday 16th July 2018

Post-Conference Field Trip - South Coast, NSW

This three-day field trip travels along the beautiful coast of southern New South Wales starting at Sydney's Macquarie University Campus on the morning of Saturday 14th July and returning late on Monday 16th July. You will get a good impression of the geology of the Permo-Triassic Sydney Basin sequence and the underlying deformed early Palaeozoic Wagonga Beds. The sedimentary rocks in this part of the Sydney Basin include glacio-marine conglomerate, sandstone, and mudstone that were deposited when this margin of Australia was at high southern latitudes. The coastal scenery between Sydney and Batemans Bay is spectacular. The vegetation includes spotted gums, casuarina or sheoaks, and burrawang palms. Eastern grey kangaroos are common near the beaches and campgrounds. There is abundant birdlife.

Led by: Kelsie Dadd

Departure: 09:00 from the MGSM Hotel

Posters

Graduation Hall, 12 Wally's Walk (E7B)

08:15   Monday 8th - Posters set up

Posters will be on display for the entire duration of the conference.
Authors will be in attendance during the poster sessions.

#		Presenting Author
P1	**Certified reference material for determination of total cyanide in soil** Batzorig Lkhagvasuren[1], Urjin Oyuntungalag[1], E. Tegshjargal[1], Sebastian Recknagel[2], Holger Scharf[2] and Juergen Rausch[3] *[1]Central Geological Laboratory, Ulaanbaatar, Mongolia* *[2]Federal Institute for Materials Research and Testing, Berlin, Germany* *[3]Federal Institute for Geosciences and Natural Resources, Hannover, Germany*	Batzorig Lkhagvasuren
P2	**$\delta^{18}O$ measurements of gem-quality apatite crystals and their application as reference material** Łukasz Birski[1], Elżbieta Krzemińska[2], Ewa Słaby[1] and Zbigniew Czupyt[2] *[1]Institute of Geological Sciences, Polish Academy of Sciences, Warsaw, Poland* *[2]Polish Geological Institute – National Research Institute, Warsaw, Poland*	Łukasz Birski
P3	**Ga isotopic compositions of various reference materials** Lanping Feng, Lian Zhou, Zhaochu Hu and Yongsheng Liu *State Key Laboratory of Geological Processes and Mineral Resources, China University of Geosciences, Wuhan 430074, P.R. China*	Lanping Feng
P4	**A suite of nine soil reference materials for targeted environmental geochemical analysis** Charles J.B. Gowing[1], Michael J. Watts[1], Katherine V. Knights[2], John Wheeler[1], Charles R. Brettle[1], Andrea J. Mills[1], Mark K. Kalra[1], Mohmed Raja[1], Andrew L. Marriott[1], Judith Mather[2] and Peter Heath[2] *[1]British Geological Survey, Keyworth, Nottingham, NG12 5GG, UK* *[2]Geological Survey of Ireland, Beggars Bush, Dublin, D04 K7X4, Ireland*	Charles Gowing
P5	**MUSTAFAA-3 and -4: Two new reference glasses for *in-situ* analysis of phosphates** Joachim Krause[1], Stephan Klemme[2], Jasper Berndt[2], Gerhard Brügmann[3], Klaus Peter Jochum[4], Denis Scholz[5], Jan Schulz-Isenbeck[1], Brigitte Stoll[4], Andreas Stracke[2] and Klaus Mezger[6] *[1]Helmholtz-Zentrum Dresden-Rossendorf, Helmholtz Institute Freiberg for Resource Technology, Chemnitzer Strasse 40, 09599 Freiberg, Germany* *[2]Institute for Mineralogy, Westfälische Wilhelms-Universität Münster, Corrensstraße 24, 48149 Münster, Germany* *[3]Curt-Engelhorn-Zentrum Archäometrie gGmbH, D6 3, 68159 Mannheim, Germany* *[4]Max Planck Institute for Chemistry, 55128 Mainz, Germany* *[5]Institute of Geosciences, Johannes Gutenberg University Mainz, 55128 Mainz, Germany* *[6]Institut für Geologie, Universität Bern, 3012 Bern, Switzerland*	Joachim Krause
P6	**A new pyrrhotite reference material for micro-beam determination of sulfur isotopes** Lei Chen[1], Xianhua Li[2], Yu Liu[2], Qiuli Li[2], Bin Hu[1] and Changfu Fan[1] *[1]MLR Key Laboratory of Metallogeny and Mineral Assessment, Institute of Mineral Resources, Chinese Academy of Geological Sciences, Beijing 100037, P.R. China* *[2]State Key Laboratory of Lithospheric Evolution, Institute of Geology and Geophysics, Chinese Academy of Sciences, Beijing 100029, P.R. China*	Lei Chen

#	Presenting Author

P7   **Characterisation of three ultramafic PGE reference materials**   Thomas Meisel

Thomas Meisel[1] and O. Marcus Burnham[2]
[1]Montanuniversität, 8700 Leoben, Austria
[2]Geoscience Laboratories, Ontario Geological Survey, Sudbury, P3E 6B5, ON, Canada

P8   **The GeoPT Proficiency Testing Programme as a scheme for the certification of reference materials**   Charles Gowing

Phil J. Potts[1], Peter C. Webb, Michael Thompson[2], J.M. Cook[3] and Charles J.B. Gowing[3]
[1]Faculty of Science, Technology, Mathematics and Computing, The Open University, Milton Keynes, MK7 6AA, UK
[2]School of Biological and Chemical Sciences, Birkbeck University of London, London, WC1E 7HX, UK
[3]British Geological Survey, Keyworth, Nottingham, NN12 5GG, UK

P9   **The analysis of nothing and nearly nothing – concepts for reference materials for the Super-SIMS**   Axel Renno

Axel D. Renno
Helmholtz-Zentrum Dresden-Rossendorf, Helmholtz Institute Freiberg for Resource Technology, 09599 Freiberg, Germany

P10   **Box-profile implants as geochemical reference materials for EMP and SIMS**   Axel Renno

Hao Sheng Wu[1], Georgii Belokonov[1], Roman Böttger[2], Frédéric Couffignal[3], Frans Munnik[2], Axel D. Renno[1], Michael Wiedenbeck[3] and René Ziegenrücker[1]
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[3]Helmholtz Zentrum Potsdam, Deutsches GeoForschungsZentrum (GFZ), Germany

P11   **The nanogram-scale heterogeneity of IAEA-CO-1 calcite**   Michael Wiedenbeck

Alexander Rocholl[1], Z. Jin[1,2], Stanisław Hałas[3†] and Michael Wiedenbeck[1]
[1]GFZ-Potsdam, Telegrafenberg, 14473 Potsdam, Germany
[2]GFZ-Potsdam & Arizona State University, School of Earth and Space Exploration, Tempe, AZ 85287, USA
[3]Maria Curie-Skłodowska University, 20-031 Lublin, Poland [†]deceased

P12   **UQAC-FeS: A new series of base metal sulfide quality control reference material for LA-ICP-MS analysis**   Dany Savard

Dany Savard[1], B. Bouchard-Boivin[1], Sarah-Jane Barnes[1] and Dieter Garbe-Schönberg[2]
[1]LabMaTer, Université du Québec à Chicoutimi, 555 boul. Université, Chicoutimi, G7H 2B1, QC, Canada,
[2]CAU Kiel University, Institute of Geosciences, D-24118 Kiel, Germany

P13   **Determination of Uranium(U) and Thorium by inductively coupled plasma mass spectrometry in geological reference materials**   Hyung Seon Shin

Hyung Seon Shin, Hana Cho, Chansoo Park and Min Yeong Park
Korea Basic Science Institute, Ochang-eup, Cheongju-Si, 28119, South Korea

P14   **The Mud Tank zircon revisited: New ID-TIMS ages reveal complex crystallisation history**   Jay Thompson

Jay M. Thompson[1], Leonid V. Danyushevsky[1], Sebastien Meffre[1] and Fernando Corfu[2]
[1]CODES, University of Tasmania, Australia
[2]Department of Geosciences and CEED, University of Oslo

P15   **A simple method for preparing homogeneous and stable solid powder standards: applied to sulfide analysis by LA-ICP-MS**   Yongsheng Liu

Wenwu Yang[1], Zhaochu Hu[1], Yongsheng Liu[2], Wen Zhang[1] and Tao Luo[1]
[1]State Key Laboratory of Geological Processes and Mineral Resources, China University of Geosciences, Wuhan 430074, P.R. China
[2]School of Earth Science, China University of Geosciences, Wuhan 430074, P.R. China

#	Presenting Author

P16 **C, H and S content in 36 geological reference materials**
Oliver Alard, Lauren Gorojovsky, Michael Förster and Annanuer Halimulati
Department of Earth and Planetary Sciences, Macquarie University, Australia

Olivier Alard

P17 **The stability over an 18-year period of consensus values as composition location estimators in results from the GeoPT proficiency testing programme**
Peter Webb[1], Phil J. Potts[1], Michael Thompson[2], Stephen Wilson[3] and Charles J.B. Gowing[4]
[1]*Faculty of Science, Technology, Engineering and Mathematics, The Open University, Milton Keynes, MK7 6AA, UK*
[2]*School of Biological and Chemical Sciences, Birkbeck University of London, Malet Street, London, WC1E 7HX, UK*
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[4]*British Geological Survey, Environmental Science Centre, Keyworth, Nottingham, NG12 5GG, UK*

Charles Gowing

P18 **Certification of coal fly ash reference material**
Batzorig Lkhagvasuren, Urjin Oyuntungalag, D. Gantsetseg, Ts. Bolormaa and N. Bolormaa
Central Geological Laboratory, Ulaanbaatar, Mongolia

Batzorig Lkhagvasuren

P19 **U-Pb doped synthetic reference material for *in-situ* hematite geochronology**
Liam Courtney-Davies[1], Marcus Richardson[2], Sarah E. Gilbert[3], Cristiana L. Ciobanu[1], Max R. Verdugo-Ihl[1], Nigel J. Cook[1], Kathy Ehrig[4] and Benjamin P. Wade[3]
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[3]*Adelaide Microscopy, University of Adelaide, SA, Australia*
[4]*BHP Olympic Dam, Adelaide, 5000 SA, Australia*

Liam Courtney-Davies

P20 **Assessment of trace-element homogeneity in gem quality zircons from Mud Tank, NT**
Sarah E.M. Gain[1], Elena A. Belousova[1], Norman J. Pearson[1], Ivan Dainis[2] and William L. Griffin[1]
[1]*CCFS/GEMOC, Macquarie University, North Ryde, NSW 2109, Australia*
[2]*Henley Beach, South Australia*

Sarah Gain

P21 **Determination of sulfate sulfur in Au-bearing ores by wavelength-dispersive X-ray fluorescence analysis**
Victor Chubarov and Alena Amosova
Vinogradov Institute of Geochemistry, Siberian Branch of the Russian Academy of Sciences, 664033, Irkutsk, Russia

Alena Amosova

P22 **Determination of the elemental composition and manganese valence state in Co-bearing ferromanganese crusts by wavelength-dispersive X-ray fluorescence analysis**
Victor Chubarov and Alena Amosova
Vinogradov Institute of Geochemistry, Siberian Branch of the Russian Academy of Sciences, 664033, Irkutsk, Russia

Alena Amosova

P23 **Metal(loid)s in Agbogbloshie E-waste recycling site in Accra, Ghana: pollution distribution, bioaccessibility and human health risk assessment**
Michael Ackah
Department of Environmental Sciences, Macquarie University, North Ryde, NSW 2109, Australia

Michael Ackah

P24 **Investigation of Lake Baikal bottom sediments by XRF and EPMA methods**
Olga Yu. Belozerova, Tatyana S. Aisueva and Egor V. Ivanov
Vinogradov Institute of Geochemistry, Siberian Branch of Russian Academy of Sciences, 664033 Irkutsk, Russia

Olga Belozerova

#		Presenting Author
P25	***In-situ* Sr isotopic measurement of scheelite using fs-LA-MC-ICP-MS** Chao Li[1,2], Limin Zhou[1] and Zheng Zhao[1] *[1]National Research Center for Geoanalysis, Beijing 100037, P.R. China* *[2]Institute of Mineral Resources, Chinese Academy of Geological Sciences, Beijing 100037, P.R. China*	Chao Li
P26	**Quantitative approaches to determination of elements in lake sediments by total reflection X-ray fluorescence** Galina V. Pashkova[1,2], Maria M. Mukhamedova[1,2] and Tatiana S. Aisueva[3] *[1]Institute of the Earth's Crust SB RAS, Irkutsk, Russia* *[2]Irkutsk State University, Irkutsk, Russia* *[3]Vinogradov Institute of Geochemistry SB RAS, Irkutsk, Russia*	Galina Pashkova
P27	**Au speciation in humic acids and Fe(III) compounds after sorption from chloride solutions** Bagai-ool Saryg-ool[1,2], Irina N. Myagkaya[1] and Elena V. Lazareva[1] *[1]Sobolev Institute of Geology and Mineralogy SB RAS, Novosibirsk, Russia* *[2]Novosibirsk State University, Novosibirsk, Russia*	Bagai-ool Saryg-ool
P28	**Thermochromatic method application for express local prospecting of gold mineralisation productivity** Alexander Tatarinov[1], G. Yalovik[2], Lubov Yalovik[1] and Vasiliy Tatarinov[3] *[1]Geological Institute, Siberian Branch of Russian Academy of Sciences, Ulan-Ude, Russia* *[2]Territorial Fund of Geological Information, Siberian Federal District, Novosibirsk, Russia* *[3]Vinogradov Institute of Geochemistry, Siberian Branch of the Russian Academy of Sciences, Irkutsk, Russia*	Vasiliy Tatarinov
P29	**Method for estimating the basic composition of fine-dispersed inclusions of native gold in a matrix of a sulfide mineral by electron microprobe** Vasiliy Tatarinov and A. Finkelshtein *Vinogradov Institute of Geochemistry, Siberian Branch of the Russian Academy of Sciences, 664033, Irkutsk, Russia*	Vasiliy Tatarinov
P30	**Use of pXRD in the quantitative assessment of alteration in epithermal Au deposits** Dane Burkett[1,2], Ian Graham[2] and Giverny Chomiszak[2] *[1]Olympus Corporation of Asia-Pacific, Singapore* *[2]University of New South Wales, Australia*	Dane Burkett
P31	**REEs in UHP garnet, its inclusions and matrix minerals** Franziska D.H. Wilke[1], Patrick J. O'Brien[2] and Alexander Schmidt[2] *[1]German Research Centre for Geoscience GFZ, Potsdam, Germany* *[2]Institute for Earth and Environmental Sciences, University of Potsdam, Germany*	Franziska Wilke
P32	**U-Pb zircons dating by LA-SF-ICP-MS at Geological Institute GIN RAS (Moscow)** Victor S. Sheshukov[1], Alexander B. Kuzmichev[1], Alexander S. Dubenskiy[1], Olga I. Okina[1], Kirill E. Degtyarev[1], Nadezhda A. Kanygina[1], Nikolay B. Kuznetsov[1], Tatiana V. Romanjuk[2] and Sergey M. Lyapunov[1] *[1]Geological Institute, Russian Academy of Sciences (GIN RAS), 119017, Moscow, Russia* *[2]IPE RAS, 123242, , B. Gruzinskaya 10, Moscow, Russia*	Alexander Dubenskiy
P33	**Isotopic composition of lead in bivalve shells using LA-ICP-MS** Igor Pessoa and Mauro C. Geraldes *Universidade do Estado do Rio de Janeiro Rua, (RJ) - 20559-900, Maracana, Rio de Janeiro*	Igor Pessoa

#		Presenting Author
P34	***In-situ* U-Th-Pb dating and Sr-Nd isotope analysis of bastnaesite by LA-(MC)-ICP-MS** Yue-Heng Yang[1], Fu-Yuan Wu[1], Qiu-Li Li[1], Yamirka Rojas-Agramonte[2,3], Jin-Hui Yang[1], Yang Li[1], Qian Ma[1], Lie-Wen Xie[1], Chao Huang[1], Hong-Rui Fan[1], Zi-Fu Zhao[4], Cheng Xu[5], Yan Liu[6] and Ki-Yi Wang[1] *[1]Institute of Geology and Geophysics, Chinese Academy of Sciences, Beijing, 100029, P.R. China* *[2]Institut für Geowissenschaften, Johannes Gutenberg-Universität, 55099 Mainz, Germany* *[3]Universidad de los Andes, M1-307, Bogotá, Colombia* *[4]School of Earth and Space Science, University of Science and Technology of China, Hefei, 230026, P.R. China* *[5]Laboratory of Orogenic Belts and Crustal Evolution School of Earth and Space Sciences, Peking University, Beijing, 1000871, P.R. China* *[6]Institute of Geology, Chinese Academy of Geological Sciences, Beijing, 100037, P.R. China*	Yue-Heng Yang
P35	**High precision U-Pb dating of terrestrial and extraterrestrial materials at SPIDE²R lab, ANU** S. Zink, Y. Amelin and V. Bennett *Research School of Earth Sciences, The Australian National University, Australia*	Sonja Zink
P36	**High-speed 3D imaging of geological materials using rapid-response laser ablation cell technology** Cosmin Stremtan[1], Stijn M. van Malderen[2] and Gavril Săbău[3] *[1]Teledyne CETAC Technologies, Omaha, NE, USA* *[2]Dept. of Chemistry, Ghent University, Ghent, Belgium* *[3]Geological Institute of Romania, Bucharest, Romania*	Cosmin Stremtan
P37	**Provenance determination of cigars using $^{11/10}$B ratios by ICP-QMS with single pulse signal acquisition mode** Wei Guo[1], Zhiwei Wu[1], Qin Shuai[2] and Shenhong Hu[1] *[1]State Key Laboratory of Biogeology and Environmental Geology, School of Earth Sciences, China University of Geosciences, Wuhan, 430074, P.R. China* *[2]Faculty of Materials Science and Chemistry, China University of Geosciences, Wuhan, 430074, P.R. China*	Wei Guo
P38	**Multiple sulfur isotopes with SHRIMP-SI** Janaína N. Ávila, Trevor R. Ireland, Peter Holden, Peter Lanc and Andrew Latimore *Research School of Earth Sciences, The Australian National University, Australia*	Janaina Avila
P39	**"*Soft*" ablation cell for LA-ICP-MS investigations of large precious samples** Haihong Chen, Yongsheng Liu, Ming Li, Zhaochu Hu and Wen Zhang *State Key Laboratory of Geological Processes and Mineral Resources, China University of Geosciences, Wuhan 430074, P.R. China*	Haihong Chen
P40	**Development of CA-ID-TIMS zircon U-Pb dating technique at Institute of Geology and Geophysics, Chinese Academy of Sciences** Zhuyin Chu[1], Junjie Xu[1,2], Wei Wang[1,3], Chaofeng Li[1], Mengjie Wang[1,3] and Jinghui Guo[1] *[1]Institute of Geology and Geophysics, Chinese Academy of Sciences, Beijing 100029, P.R. China* *[2]School of Ocean Sciences, China University of Geosciences, Beijing 100083, P.R. China* *[3]College of Earth Sciences, University of Chinese Academy of Sciences, Beijing 100049, P.R. China*	Zhu-Yin Chu
P41	**An evaluation of total uncertainty in WDXRF Geoanalysis** Richard M. Conrey, Laureen C. Wagoner and David G. Bailey *Geosciences Dept., Hamilton College, 198 College Hill Rd Clinton, 13323, NY, USA*	Rick Conrey
P42	**Use of a single background per analyte in WDXRF geoanalysis: Minimisation of spectral interference** Richard M. Conrey, Laureen C. Wagoner and David G. Bailey *Geosciences Dept., Hamilton College, 198 College Hill Rd, Clinton, 13323, NY, USA*	Rick Conrey

#	Presenting Author
P43 **Determination of nitrogen in experimental and natural samples using EPMA and CHNS analyser**	Michael Förster

Michael W. Förster[1], Stephan Buhre[2], Olivier Alard[1] and Stephen F. Foley[1]
[1]*ARC Centre of Excellence of Core to Crust Fluid Systems (CCFS), Department of Earth and Planetary Sciences, Macquarie University, NSW 2109, Sydney, Australia*
[2]*Institute of Geosciences, Johannes Gutenberg University, 55099 Mainz, Germany*

#	Presenting Author
P44 **Removal of Hg interferences for common Pb correction when dating minerals by LA-QQQ-ICP-MS**	Sarah Gilbert

Sarah Gilbert[1] and Jack Gillespie[2]
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P45 Is the handheld XRF device a useful field tool for volcanologists? — Heather Handley

Heather Handley[1] and Paul Harvey[2]
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[2]*MQ Centre for Energy and Environmental Contaminants, Department of Environmental Sciences, Macquarie University, Australia*

P46 A practical method for measuring high precision calcium isotope ratios without chemical purification for calcium carbonate samples by multiple collector inductively coupled plasma mass spectrometry — Dong He

Dong He, Zhenli Zhu and Lingyun Zhao
State Key Laboratory of Biogeology and Environmental Geology, China University of Geosciences (Wuhan), Wuhan, 430074, P.R. China

P47 The system of simultaneous double laser ablation for *in-situ* micro-analysis — Chao Huang

Chao Huang[1,2], Lie-Wen Xie[1,2], Yue-Heng Yang[1,2] and Jin-Hui Yang[1,2]
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P48 UV-femtosecond laser ablation, fluorination and GC-IRMS analysis of four sulfur isotopes — Alexander Ignatsiev

Alexander Ignatiev, Tatiana Velivetskaya and Victoria Ykovenko
Far East Geological Institute FEB RAS, Vladivostok, Russia

P49 *In-situ* lithium isotope ratio in natural and synthetic glasses by LA-SF-ICP-MS — Margareth Sugano Navarro

Margareth Sugano Navarro, Gustavo Macedo de Paula Santos and Jacinta Enzweiler
Institute of Geosciences, UNICAMP Campinas, SP, Brazil

P50 New software for data reduction of LA-ICP-MS data sets — Ashley Norris

C. Ashley Norris[1] and Leonid Danyushevsky[2]
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P51 Nanoscale inhomogeneities in nacre: New analytical challenges for the Atom Probe — Laura Otter

Laura M. Otter[1], Katja Eder[2], Oscar Branson[3], Julie M Cairney[2] and Dorrit E. Jacob[1]
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#		Presenting Author
P52	**A Geoscience Atom Probe facility** David W. Saxey[1], Steven M. Reddy[1,2], Denis Fougerouse[1,2] and William D.A. Rickard[1] *[1]Geoscience Atom Probe, Advanced Resource Characterisation Facility, John de Laeter Centre, Curtin University, Perth, Australia* *[2]School of Earth and Planetary Sciences, Curtin University, Perth, Australia*	David Saxey
P53	**Analytical capabilities of the Cameca 1280 SIMS instrument** Michael Wiedenbeck, Alexander Rocholl and Frederic Couffignal *GFZ-Potsdam, Telegrafenberg, 14473 Potsdam, Germany*	Michael Wiedenbeck
P54	**Effect of signal acquisition mode on isotope ratio precision and accuracy in ICP-quadrupole-MS** Zhiwei Wu, Wei Guo and Shenhong Hu *State Key Laboratory of Biogeology and Environmental Geology, School of Earth Sciences, China University of Geosciences, Wuhan, 430074, P.R. China*	Zhiwei Wu
P55	**A novel sample cell for reducing the *"Position Effect"* in Laser Ablation MC-ICP-MS isotopic measurement** Lie-Wen Xie[1,2], Lei Xu[1,2], Qing-Zhu Yinc[3], Yue-Heng Yang[1,2], Chao Huang[1,2] and Jin-Hui Yang[1,2] *[1]State Key Laboratory of Lithospheric Evolution, Institute of Geology and Geophysics, Chinese Academy of Sciences, P.O. Box 9825, Beijing 100029, P.R. China* *[2]Institutions of Earth Science, Chinese Academy of Sciences, P. O. Box 9825, Beijing 100029, P.R. China* *[3]Department of Earth and Planetary Sciences, University of California at Davis, Davis, CA 95616, USA*	Li-Wen Xie
P56	**Advanced 193 nm Laser Ablation** Burkhard Fechner and Ralph Delmdahl *Coherent LaserSystems GmbH & Co. KG Hans-Boeckler-Str. 12, 37079 Goettingen, Germany*	Burkhard Fechner
P57	**Achieving clumped isotope efficiency to characterise palaeoenvironmental variation: the next generation of equipment and automation** Sean T. Murray and Martin J. Kennedy *Macquarie University, 2109, NSW, Australia*	Sean Murray
P58	**LA-ICP-MS analysis routine for trace elements in olivine** Marina Veter, Stephen F. Foley and Dorrit E. Jacob *CCFS/GEMOC, Department of Earth and Planetary Sciences, Macquarie University*	Marina Veter
P59	**The influence on PA factors by P/A correction under different signal response values of Agilent 7500Ce** Fang Ma *Key Laboratory of Orogenic Belts and Crustal Evolution, Ministry of Education, School of Earth and Space Sciences, 100871, Peking University, Beijing*	Fang Ma
P60	**Calibration systematics of the LA-ICP-MS at the Geological Survey of Brazil** Lynthener Bianca Takenaka[1,2], Sandra Andrade[2] Letícia Muniz[3] Joseneusa Brilhante Rodrigues[1] Carlos Eduardo Ganade de Araújo[1] and Maria Alice Ibañez Duarte[1] *[1]Geological Survey of Brazil-CPRM, Brazil* *[2]São Paulo University – USP, Brazil* *[3]Rio de Janeiro State University – UERJ, Brazil*	Lynthener Takenaka
P61	**Laboratory study of multiple sulfur isotope fractionation during SO_2 photolysis** Tatiana Velivetskaya, Alexander Ignatiev and Victoria Yakovenko *Far East Geological Institute FEB RAS, Russia*	Tatiana Velivetskaya
P62	**Chlorine analysis with XRF to determine salinity of pore water in the Baltic Sea sediments** Siim Pajusaar Toivo Kallaste and Tarmo Kiipli *Tallinn University of Technology, Department of Geology, 19086, Tallinn, Estonia*	Siim Pajusaar

#		Presenting Author
P63	**Analysis of organic materials by LA-ICP-MS using carbon as internal standard, an example grass study** Charlotte M. Allen and Karine Harumi Moromizato *CARF Institute for Future Environments, Queensland University of Technology, Brisbane, QLD 4000, Australia*	Karine Harumi-Morimozato
P64	**Wavelength dispersive X-ray fluorescence determination of major oxides in bottom and peat sediments for the paleoclimatic studies** Alena Amosova[1], Victor Chubarov[1] and Galina Pashkova[2] *[1]Vinogradov Institute of Geochemistry, Siberian Branch of the Russian Academy of Sciences, 664033, Irkutsk, Russia* *[2]Institute of the Earth Crust, Siberian Branch of the Russian Academy of Sciences, 664033, Irkutsk, Russia*	Alena Amosova
P65	**Struggle with inhomogeneously quenched melts** Zsanett Pintér[1,2,3], Stephen F. Foley[1,2], Gregory M. Yaxley[3] and Tracy Rushmer[1] *[1]Department of Earth and Planetary Sciences, Macquarie University* *[2]ARC Centre of Excellence for Core to Crust Fluid Systems (CCFS)* *[3]Research School of Earth Sciences, Australian National University*	Zsanett Pinter
P66	**Isotopic heterogeneity of the Pre-Ordovician Earth's crust of the western part of the Central Asian Fold Belt (CAFB) based on LA-ICP-MS study of detrital zircons** Nikolay B. Kuznetsov[1,2], Tatiana V. Romanjuk[2,3], Elena A. Belousova[2], Kirill E. Degtyarev[1], Victor S. Sheshukov[1], Alexander S. Dubenskiy[1], Nadezhda A. Kanygina[1] and Sergey M. Lyapunov[1] *[1]Geological Institute, Russian Academy of Sciences (GIN RAS), 119017, Moscow, Russia* *[2]Australian Research Council Centre of Excellence for Core to Crust Fluid Systems (CCFS) and GEMOC; Macquarie University, Sydney, NSW 2109, Australia* *[3]IPE RAS, 123242, B. Gruzinskaya 10, Moscow, Russia*	Alexander S. Dubenskiy
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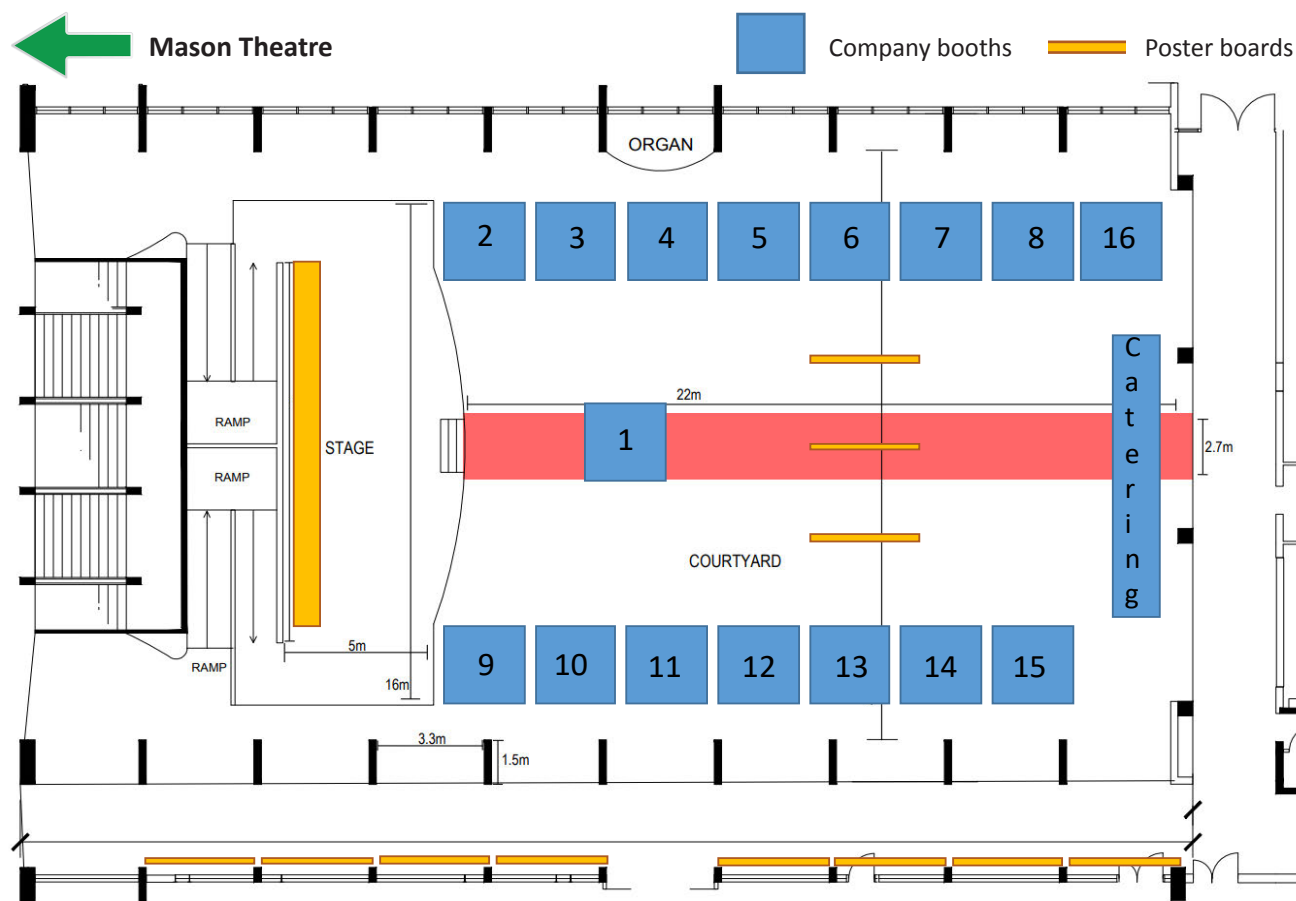11. CAMECA
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Abstracts

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Metal(loid)s in Agbogbloshie E-waste recycling site in Accra, Ghana: pollution distribution, bioaccessibility and human health risk assessment

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Agbogbloshie E-waste recycling site is the largest electronics waste recycling site in Ghana. I hypothesised that the soils, dust and waters are impacted as a result of extensive E-waste dismantling, disjointment, burning and recycling. The objective of my research was to assess bulk and bioavailable concentrations of metal(loid)s in different environmental compartments in the E-waste site and assess human health risks due to exposure to toxic metal(loid)s. Sediment, floor dust and water samples were collected in Agbogbloshie E-waste site and Odaw River/Korle Lagoon from December to January 2017. Sediment, dust and water samples were analysed using Energy Dispersive X-ray fluorescence (EDXRF) and Total Reflection X-ray Fluorescence (TXRF) spectrometry. Elevated concentrations of metal(loid)s were found in E-waste recycling soils and floor dust while Odaw River/Korle Lagoon water contained low dissolved metal concentrations. Topsoils of burn-area and dismantling areas contained the highest concentrations of metals. Copper, zinc and lead were highly bioavailable. Human health risk assessment showed that ingestion was the most important exposure pathway and children were more susceptible. Lead presents the greatest risk in soils of the E-waste site. Results from the study will help in management decisions on pollution at the Agbogbloshie E-waste recycling site.

C, H and S contents in 36 geological reference materials

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C, N, H and S have been determined in 36 reference materials spanning a large range of compositions and mineralogies (high and low temperature rocks and one soil). The reference materials investigated are from the USGS, CCRMP, SARM, IAG and BAM. C, N, H and S have been measured by elemental analyser from LECO (CNS2000) and ELEMENTAR (EL-cube). Nitrogen data are discussed in detail by Förster and co-workers (*this conference*). Analysed powder aliquots were between 50 mg and 1g, but typically between 100 and 200 mg. Between 3 and 30 replicates have been obtained for each reference material. For H, C and S so far, no statistically meaningful difference has been observed between oven- and vacuum-oven-dried samples. Similarly, the addition of WO_3 flux has so far no impact on the measured values.

Reproducibility of C measurements for high contents (>1000 ppm) is overall between 3 and 10%, the only exception being UB-N (C=1523±265 ppm, n=27). Reproducibility for low C-contents (<1000 ppm) varies between 3 and 45%. The lowest value obtained (C= 49±14 ppm, n=3) is for RGM-2. Although this low value is close to the theoretical detection limit for a thermal conductivity detector (TCD) we note that blank replicates are much lower and extremely reproducible. Agreement with literature values is rather good, while reducing significantly the uncertainties especially in the 200-3000 ppm range.

H contents range between 136 ppm and 2.21 wt.% and reproducibility is better than 20% for most reference materials. The data compare well with published values and with Karl Fischer titration from the Max Planck Institute (Mainz, Germany).

S contents range between 8 ppm and 4.33 wt.%. Reproducibility is highly variable between 6% and 65%. The relationship between TCD and IR detector remains linear from 50 to 43500 ppm, below 50 ppm TCD is much more variable and seems to slightly overestimate S-content. For the 5 to 500 ppm S range high temperature iodo-titration (Gros et al., 2005) and EL-cube measurements using the IR detector are in good agreement. S measurements by LECO and Elementar are within uncertainties for the 50 to 3000 ppm range (TCD).

The ≈1500 analyses obtained should greatly improve the determination of C H (N) S concentration for these 36 references materials.

Gros, M. et al (2005) *Geostandards and Geoanalytical Research* **29**, 123-130.

Trace elements concentrations and distributions in microanalytical reference materials suitable for sulfide and FeNi alloy analyses

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Using scanning electron microscope (SEM, Zeiss EVO MA15) and micro XRF mapping (µXRF, M4 Tornado Bruecker) we investigated the homogeneity and distribution of siderophile and chalcophile elements in sulfur-based and steel materials. Further work using electron probe (Cameca SX100) and laser-ablation ICP-MS (Agilent 7700 and 8900 and Element XR-HR) available at Macquarie GeoAnalytical (MQGA) and Géosciences Montpellier (GM) were carried out to assess the composition and homogenity of these sulfides and FeNi alloys. Sulfur materials investigated are PGE-A, Po62, Po726, Po727 and 5 *in-house* NiS quenched beads either doped with pure metal or using natural rocks (e.g., SARM-7). Steel materials are alloyed steel reference materials Jernkontoret JK27 and JK37 and NIST alloy steels N661, N1158 and N1262b. These materials were cross-calibrated with each other and obtained values are compared. The various calibration strategies were tested for assessing the composition of terrestrial sulfides and metal in iron meteorites.

Analysis of organic materials by LA-ICMPS using Carbon as internal standard, an example grass study

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Quantitative laser ablation ICP-MS analysis is usually achieved by using an internal standard element, and an external reference material in the method of Longerich et al. [1]. The concentration of that internal standard element is either measured by some other method or is assumed to be stoichiometric. We have shown that using ^{12}C or ^{13}C as the internal standard for a range of biological materials works well even though C is difficult to ionise to 1+ in an Ar plasma. Given there is a great abundance of C in organic compounds (~40 wt%), the moderately high C background (from CO_2 in air) is not problematic. In the case of a world-wide grass study by QUT workers in which the grass was dried and powdered and analysed for carbon isotopes, we had starting material ideal for making 6 mm "*pills*" of the grass in a hydraulic press, and then mounting them with glue-stick onto a marked plexiglass template for positioning in the excimer laser's cell. With the grasses we treated two NIST certified reference materials similarly: peach leaves and spinach. These materials are certified for a wide range of major and trace elements and one can be used for quantitation while the other can be used for monitoring the precision and accuracy of the results. We found that sample and standard homogeneity is the main difficulty with achieving great accuracy and precision, and that multiple spots or tracks (~80 microns wide) on each sample must be undertaken.

We developed this method for grass but have applied it to chick peas, hair and carbonates with reasonable results.

[1] Longerich, Jackson and Gunter (1996) *JAAS* **11**, 899-904.

Dating $^{87}Rb/^{86}Sr$ in minerals using LA-ICP-MS, achievements and problems

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Zack and co-workers [1,2] have introduced the use of reaction gases in "*triple quad*" ICP-MS instruments such as Agilent 8800 or 8900 in order to separate ^{87}Rb from ^{87}Sr by mass shifting Sr^+ to SrO^+ in an oxidizing gas reaction cell between the quadrupoles. They originally used O_2 but have found N_2O a much more efficient reagent. After exiting the plasma, Rb is 1+ and in a noble gas configuration and does not react, whereas Sr^+ would prefer to combine and thus Sr is moved away from Rb at mass 87. Further, the other complications of doubly charged REEs are mitigated in the setup in which quad1 filters for a single mass (87 in this example) and the second quad is used to check for the reactant (87+16=103). In a triple quad set up one can check mass 85+16=101 to prove that Rb does not move with Sr to the main mass of interest.

Although the method has great promise there are technical issues to overcome: biases in the dual electron multipliers and how to cope with them (the PA factor problem), down hole fractionation of Rb and Sr while laser drilling, and homogeneity of the reference material.

We present studies of biotite and muscovite dating to show what accuracies and precisions can be achieved. Further we explored ozone as a reaction gas and found N_2O to be more desirable.

[1] Zack and Hogmalm (2016) *Chemical Geology* **437**, 120-133. [2] Hogmalm et al., (2017) *JAAS* **32**, 305-313.

Wavelength dispersive X-ray fluorescence determination of major oxides in bottom and peat sediments for the paleoclimatic studies

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Wavelength dispersive X-ray fluorescence (WDXRF) method is widely used for quantitative analysis of main rock-forming elements [1]. Bottom and especially peat sediments may contain carbonate minerals and organic parts up to 70 %, therefore for quantitative determination of major rock-forming elements special selection of calibration set and additive comparison analysis are necessary. We employed certified reference materials (CRMs) of silts, sediments, dolomites, siltstone, limestone, clays, ooze and loess. 143 samples of bottom sediment core and 21 samples of peat sediment core were analysed. Cores were drilled in 2013 from sites located in high-mountain outlying from large cities areas, where undisturbed ecosystems prevail. All samples were calcined in a muffle furnace at temperature 950 °C and 110 mg of calcined sample were mixed with 1.1 g of lithium metaborate in platinum crucible. The mixture was fused in automatic electric furnace TheOX at 1100 °C [2]. The measurements were performed by a WDXRF spectrometer S8 Tiger. Selected set of CRMs is not completely corresponded in elemental and mineralogical composition to analyzing sample, so accuracy of XRF analysis was estimated by comparison with spectrophotometry, flame photometry and atomic absorption spectrometry results. Comparison using Z-criteria [3] shows that results of XRF analysis can be accepted as satisfactory. Proposed technique requires only 110 mg for analysis. It allows to analyse each centimetre of cores and to achieve unique high resolution of paleoclimatic reconstructions of regional climate and environment changes in Siberian region over the last 7000 years [4].

[1] Potts P.J. (1992) *A Handbook of Silicate Rocks Analysis,* *226-285.* [2] Amosova et al. (2016) *Spectrochimica Acta Part B: Atomic Spectroscopy* **122**, *62-68.* [3] Thompson et al. (2015) *Geostandards and Geoanalytical Research* **39**, *433-442.* [4] Bezrukova et al. (2017) *Contemporary problems of ecology* **10**, *431-440.*

Microanalysis of Cu Slag: Understanding the role of mining waste in ongoing environmental contamination

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Legacy base metal mines host a variety of waste materials, yet until recently, slag has been regarded as inert waste with little environmental consequences. This has lowered its remediation priority at former mines.

The environment downstream of Gulf Creek, a former copper mine, has been impacted by leachate and the stream there is lined with powder blue precipitate. The workings have been extensively rehabilitated, with Cu-Zn rich slag distributed across parts of the former mine to control soil erosion. These distributed slags potentially increase leaching of Cu and Zn into adjacent streams.

This study constrains the potential contribution that leaching of slag has on downstream metal contamination, via an understanding of the macro- and micro-scale elemental composition and mineralogy. Elemental mapping was undertaken with 20 μm resolution on polished mounts using a Bruker M4 Tornado μ-XRF, and then to sub-μm resolution using an FEI FEG-SEM. Mineralogy from the μ-XRF and FEG-SEM was then inferred from comparison of the sample XRF spectra with those from pure mineral standards. Bulk powder diffractograms, collected with a PANalytical X'Pert Pro diffractometer, provided further constraint on the minerals inferred to be present.

Metals are present in mineral grains and patches in glass to <1 μm diameter. Elements unresolved at 20 μm using the μ-XRF, appear uniformly dispersed at 1 μm resolution using the FEG-SEM. Elemental maps show that Cu and S co-occur, Zn is found in minerals independent of other metals, and Cd, Al and Ni tend to co-occur weakly in minerals. Micro-scale, metal-hosting mineral grains make up a large proportion of the slag's surface area and volume. Minerals in this slag potentially contribute substantial metal leachate to Gulf Creek. Slags rich in other metal(loid)s including As and Pb should also be investigated for the macro and micro-distribution of metal-bearing minerals, to help constrain their role in environmental contamination.

Multiple sulfur isotopes with SHRIMP-SI

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Sulfur isotopes have been used to address a wide variety of questions and processes about the evolution of Earth's atmosphere and hydrosphere. The four-isotope analysis of sulfur allows the distinction of different reaction pathways during geological and biological processing of sulfur. Of particular note in isotope geochemistry is the fractionation of S isotopes in the early Earth where correlated variations in $\Delta^{33}S$ and $\Delta^{36}S$ are apparent with a ratio of approximately -1. The variations of $\Delta^{33}S$ and $\Delta^{36}S$ measured in rocks older than ~2.4 Ga have been attributed to mass-independent fractionation (MIF) of sulfur isotopes in atmospheric source reactions. On the other hand, measurements of multiple sulfur isotopes in some post-Archean sulfides have found non-zero $\Delta^{33}S$ and $\Delta^{36}S$ produced by mass-dependent biogeochemical fractionation (MDF) that show a different $\Delta^{36}S/\Delta^{33}S$ relationship (~ -7) than that found for Archean rocks. Therefore, quantifying the relationships between $\Delta^{33}S$ and $\Delta^{36}S$ offers the possibility to decouple signatures produced from mass-independent processes from those produced from mass-dependent processes.

Over the past few years, there have been major advances on ion microprobe measurements of sulfur isotopes. One of our recent goals has been to optimise the SHRIMP-SI detection system for measurement of multiple sulfur isotopes (^{32}S, ^{33}S, ^{34}S, and ^{36}S). Due to the large abundance difference between the major (^{32}S and ^{34}S) and minor isotopes (^{33}S and ^{36}S) and the presence of sulfur in different proportions in minerals, different analytical protocols need to be adopted. For instance, the addition of each isotope requires a longer analytical time simply because of the lower abundances of ^{33}S and ^{36}S relative to ^{32}S and ^{34}S. Analysis of ^{33}S only requires an additional few minutes to get the required statistics (to around 0.1‰ in $\Delta^{33}S$). Four isotope analysis including ^{36}S requires approximately 20 minutes to achieve 0.2‰ precision in $\Delta^{36}S$ in sulfides. Charge-mode detection of the low abundance ^{36}S isotope has proven to be a major advantage on the determination of $\delta^{36}S$. The analysis of sulfate is more difficult because the proportion of S in this mineral is far lower than in sulfide, which results in ion signals a factor of two or more lower. Accuracy of measurements ($\delta^{34}S$, $\delta^{33}S$, $\delta^{36}S$, $\Delta^{33}S$, and $\Delta^{36}S$) is assessed by analysis of sulfur reference materials (e.g., Ruttan and Balmat pyrite, Canyon Diablo troilite, Norilsk chalcopyrite, Anderson pyrrhotite, Balmat galena). We present the analytical protocols for SHRIMP-SI multiple sulfur isotope analysis of a suite of sulfide reference materials as well as examples of high precision multiple sulfur data of MIF ($\Delta^{33}S/\Delta^{36}S$ ~ -1) and MDF ($\Delta^{33}S/\Delta^{36}S$ ~ -7) sulfur signatures. One of the goals of this work is to document the degree of homogeneity of the reference materials, and to what degree of confidence they are useful as reference materials.

2008-2018 – *"The Portable XRF Decade"*

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The last decade (2008-2018) has seen an unprecedented acceleration in the innovation and adoption of Hand-Held and Portable X-ray Fluorescence (pXRF) analysers within the mineral exploration and mining industries. A futuristic device that was once heavily criticised, has now become an industry standard tool and continues to evolve at an increasingly rapid pace, in line with the high performance and field ruggedisation expectations demanded by the modern geoscientist. This talk will aim to summarise the key advancements and achievements both in pXRF technology, as well as application development. Key highlights will include:

Technological Innovation (Hardware/Software):

- Move from active sources to tubes
- Evolution of form factor – computing power & ruggedisation
- The innovation to Silicon Drift Detectors (SDDs) from Silicon Pin (Si-PIN) detectors
- New generation High Count Rate (HCR) Digital Pulse Processing (DPP) that has sped up and de-bottlenecked performance
- The continual drive towards lower LODs and expansion of the element suite (light elements & REEs)
- The uptake and innovation in field based sample preparation equipment
- Real-time QA/QC, data management, visualisation and the move to cloud-based data delivery

Application Development & Highlights

- The change in culture and adoption of best practice guidelines (off the back of CAMIRO pXRF project…etc.)
- Key organisations, researchers and companies that are leading the pack in best practice application of pXRF (CAMIRO, DET CRC, CSIRO, MDRU, Reflex…etc.)
- Chemo-stratigraphy
- Gold pathfinders & alteration vectoring
- REE & Battery Metals
- GeoMet applications
- Key Journal Volumes, Papers & Publications
- JORC / 43-101 considerations

A machine-learning interpretation of regional geochemical patterns in northern Australia

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Machine-learning (ML) enhances the predictive capacity of geochemical surveys by finding relationships between the measured point values and a comprehensive suite of geospatial datasets (e.g. geophysical, terrain and satellite imagery). We applied ML to interpret the combined Mobile Metal Ions (MMI) results of the Northern Australia Geochemical Survey (NAGS) and the National Geochemical Survey of Australia (NGSA) in the Tennant Creek – Mt Isa area of northern Australia (Figure 1). The samples were taken exclusively from transported sediments; thus model training and predictions were restricted to depositional landscapes. ML modelling was completed with the uncoverML code (Geoscience Australia) using Random Forest, a supervised decision tree based algorithm for regression and classification.
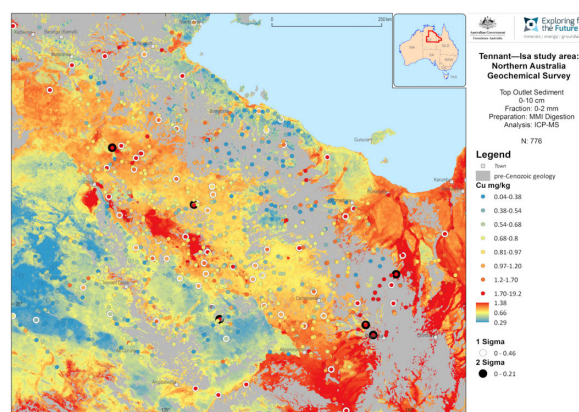

Figure 1. Predicted Cu concentrations for NAGS and NGSA samples over the Tennant Creek – Mt Isa study area. Cu values above mean + 1 or 2 σ values of the predictive model indicate "anomalous" sites where Cu concentrations are above the local background variations.

The ML algorithm generates several hundred predictions by varying the sites used for training and the covariates used for prediction. All calculations are based on log-normalised analytical values. The mean values are used for the model predictions and standard deviations are used as a measure of the model uncertainty. To emphasise potential anomalies above the local background variations, values that are 1 or 2 standard deviations (σ) above the mean of the model prediction are specifically highlighted.

The most important covariates include the gamma-ray map, Landsat imagery, climate surfaces (e.g. annual evaporation, annual mean radiation and annual mean temperature) and MODIS satellite imagery. The results highlight the importance of climatic factors in controlling the local distribution of labile forms of chemical elements extracted by MMI.

Future work will incorporate new covariate datasets including solid geology and hydrological features (e.g. flow accumulation and surface flow dispersion).

Using robust plasma conditions to examine preferred values for BCR-2G and ATHO-1G relative to NIST glasses

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Elemental fractionation during LA-ICP-MS analysis occurs as the result of processes happening at the ablation site and in the plasma. In this study we have focussed on the processes occurring in the plasma. By changing Ar carrier gas flows we observe the effect of different plasma conditions on element fractionation. It has been demonstrated that robust plasma conditions are an essential requirement for minimizing elemental fractionation. The U/Th [1] and Ar^+/Ar^{2+} [2] ratios are useful indicators of robust plasma conditions as the former is an indicator for particle digestion in the plasma. We used both of these ratios to investigate further the effect of aerosol formation during ablation on the processes occurring in the plasma, by comparing measurements on materials characterised by different ablation rates, and by comparing results from measurements at different laser beam sizes.

Measurements were performed using silicate glasses NIST610, NIST612, BCR-2G and ATHO-1G. With the instrumentation setup used in this study and utilizing a 70 μm beam, U/Th ratios remain unchanged at Ar flow rates < 0.95 l/min, which can be interpreted as robust plasma conditions. Comparison of concentration-normalised U/Th ratio between the four glasses reveals that at robust plasma conditions both BCR-2G and ATHO-1G glasses are characterised by different concentration-normalised U/Th ratios, compared to the NIST glasses. The differences in the concentration-normalised U/Th ratios are ~3% and ~4%, respectively. The observed differences can be explained by the inconsistency in the accepted concentration values for the 4 glasses studied, and our results suggest that this approach can be used to assess the consistency between accepted concentrations of reference materials.

This approach together with looking at differences in mass response curves reveals inconsistencies not only in U and Th, but also in preferred values of Se, Ag, Sn, Co, Ni, Zn, Pb and some other elements in BCR-2G and ATHO-1G reference materials relative to NIST glasses.

[1] Wang et al. (2006) *JASMS, 17, 641-651.* [2] Fietzke & Frische (2016) *JAAS, 31, 234-244.*

Exploring the range of U-containing minerals for geochronological applications

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The increasing use of LA-ICP-MS U-Pb dating derives from the fact that it is the cheapest, most widely available and fastest technique for *in-situ* U-Pb dating. Despite increasing usage, however, this technique is largely used for zircon dating (Fig. 1). The application of this method to more than a single accessory mineral (zircon) offers the advantage to study complex geologic problems, enabling dating of the late-crystallisation events as well as providing ages for rocks that do not crystallise zircon.
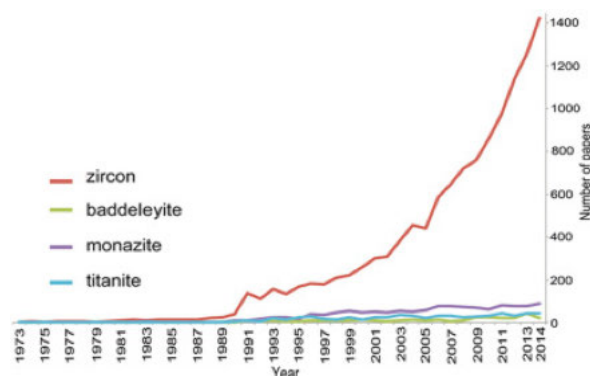

Figure 1. Growth in applications of U-Pb zircon geochronology in contrast to other U-containing minerals (Spencer et al., 2016).

The potential and limitations of several U-containing minerals (e.g. perovskite, baddeleyite, rutile and apatite) for geochronological applications using the LA-ICP-MS approach will be discussed.

Perovskite is one of the late-phase minerals to crystallise from the kimberlitic magma. The fact that perovskite is a main carrier of U and Th in the kimberlite makes possible the application of U-Pb dating on this mineral and thus allows dating the emplacement of the kimberlite.

Baddeleyite, a common trace mineral phase in silica under-saturated mafic, ultramafic and alkaline rocks and thus has been a prime target mineral for dating mafic rocks by the U-Pb method.

Rutile is a common accessory mineral that occurs in a wide spectrum of metamorphic rocks, such as in blueschists, eclogites, and granulites and as one of the most stable detrital heavy minerals in sedimentary rocks. Is is increasingly used for *in-situ* U-Pb geochronology.

Apatite is a common U-bearing accessory mineral with a U-Pb closure temperature of ~500°C, making U-Pb dating of apatite a potentially valuable thermo-chronometer. Challenges to reliable dating of apatite will be outlined.

Spencer, C.J., Kirkland, C.L. & Taylor, R.J. (2016) *Geoscience Frontiers, 7, 581-589.*

Study of mineral component in technogenic soils of Irkutsk Region by X-ray electron probe microanalysis

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Soils, as basic component of geochemical landscapes, are an informative indicator of environment pollution. Intensive activity of industrial centres leads to irreversible processes of soil cover pollution in considerable territories. In this regard, the research of processes of heavy metals biological absorption by microorganisms, directed on remediation of soils, is very relevant today. When studying technogenous formations it is necessary to know detailed information on the chemical composition, substance phase distribution at micron level, the sizes and shapes of phase separation and surface structure. The X-ray electron probe microanalysis (EPMA) is the perspective method for solving such tasks.

The results of EPMA investigation of technogenic soils cultivated by biologicals for the purpose to study the influence of rhizospheric bacteria (*Azotobacter and Bacillus*) on the distribution and migration of As and Pb elements in the "*soil-plant*" system are presented in this work.

Soil samples were collected around of Svirsk town in regions with various man-caused loading degrees from polluter (Angarsk enterprise on production of arsenic). Each soil from all regions was divided on the soil "test" and soil cultivated by biologicals "experiment", which are used for the cultivation of plants (wheat, oats). The biologicals developed at the Tomsk State University were applied as plant growth stimulant.

The EPMA technique is developed for soil mineral component applying JXA-8200 (JEOL Ltd, Japan) microprobe with energy-dispersive and wavelength-dispersive spectrometers. The phase and chemical composition of "*test*" and "*experiment*" soils were studied. The surface and shapes of phase separation, sizes and homogeneity of their distribution were estimated using scanning electron microscope. The differences of phase composition and distribution of As and Pb elements in mineral formations of "*test*" soils and "*experiment*" soils inoculated by rhizospheric bacteria were detected.

Results of EPMA research allowed to establish the signs of rhizospheric bacteria influence on the crystallisation of secondary mineral formations of As and Pb in soils of technogenic regions. The high geochemical activity of rhizospheric bacteria to influence on the transformation of As and Pb compounds in the soil, as well as on the processes of an immobilisation and mobilisation of these elements in "*the soil-plant*" system is shown.

Investigation of Lake Baikal bottom sediments by XRF and EPMA methods

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Bottom sediments of Lake Baikal represent unique archive for reconstructing the environment and climate changes in the Baikal Region. In this study it is important to acquire more information both on the chemical composition, ratios of elements and the substance phase distribution at micron level, mineral and granulometric composition and surface structure. The promising analytical methods for solving these tasks are nondestructive methods such as X-ray fluorescence analysis (XRF) and X-ray electron probe microanalysis (EPMA).

This paper reports the methodical approaches and results of Baikal bottom sediments study using XRF and EPMA. The samples of bottom sediments core were collected at the Posolsk Bank (shoal) through depth 0 to 150 cm with 6 cm spacing.

Contents of major and microelements in sediments were determined by XRF. The measurements were performed using wavelength-dispersive XRF spectrometer S8 TIGER (AXS Bruker, Germany).

The phase mineral composition of sediments, shapes of phase separation, the surface structure and size distribution were studied by EPMA through entire depth of core. The measurements were applying Superprobe JXA-8200 (JEOL Ltd, Japan) microprobe.

The XRF results allowed studying the regularity of element distribution through entire depth of bottom sediments core. The analyses identified the correlations of element ratios and drastic increase of some element contents in near-surface layers of core.

The EPMA results indicated that, bottom sediments consist of mineral phases presented as differently shaped and sized particles. The change of phase mineral composition of sediments through core depth was observed. In the analysed layers the sediments are basically composed of silicate mineral component contained quartz, albite, feldspar, pyroxene and amphibole. The upper surface layers of core are characterised by diverse compositions of mineral phases occurring as spinel, magnetite, ilmenite, apatite, sulfide, zircons, as well as metallic granules and spherules with high contents of Fe and Ti and low Mg, Al and Si.

Thus, the comprehensive study of the Baikal bottom sediments by XRF and EPMA provides complete information, which may be useful for assessment and reconstruction of changes occurring in the environment and climate of the Baikal Region.

$\delta^{18}O$ measurements of gem-quality apatite crystals and their application as reference material

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Oxygen isotope ratio ($\delta^{18}O$) has been widely used in archeology, geochemistry, paleoclimatology and paleoceanography (e.g. [1-2]). The use of *in-situ* methods such as secondary ion mass spectrometry (SIMS) allows to analyse very precisely even a single crystal or microfossil element. Unfortunately, the possibility of using SIMS is limited by imperfection of reference materials (RMs). Main requirements to be complied with by every RM are "similarity" to the sample and homogeneity of measured elements or isotopes. Therefore, the quality of the RM is of extreme importance. Commonly used as RM for $\delta^{18}O$ in apatite crystals measurements Durango apatite [3] is not free of imperfections.

Several gem-quality apatite crystals were analysed by us using SHRIMP IIe/MC and Thermo Flash EA 1112HT elemental analyzer connected to a Thermo Delta V Advantage isotope ratio mass spectrometer to determine their potential as RMs. Furthermore, bulk measurements of each apatite were performed twice. Firstly, all of the analysed apatite crystals were inspected with SEM-CL.

All analysed by us gem-quality apatite crystals have rather low $\delta^{18}O$ values (tab. 1), due to their magmatic origin. Values obtained by two different scientific methods are in good agreement and it can be used as an argument confirming correctness and accuracy of data obtained on ion microprobe. Furthermore, we have found large variation in $\delta^{18}O$ in range of single gem-quality apatite crystals. None of the examined apatite crystals does not satisfy the requirement of homogeneity. According to data collected by us, this commonly used as RM apatite crystal still remains the most suitable for this purpose, yet search for a better $\delta^{18}O$ in apatite RM remains unresolved.

Sample	$\delta^{18}O$ VSMOW (bulk)	$\delta^{18}O$ VSMOW (*in situ*)		
		average	σ	n
DURANGO	5.89	6.82	6.33	0.24	24
AFGHANISTAN	7.13	7.12	6.56	0.71	9
APATITI KOLA	3.03	2.79	2.23	0.24	11
KOLA	7.21	5.86	5.61	0.52	4
MAROCCO	12.57	12.69	11.98	0.44	31
MADAGASCAR	9.50	10.02	7.91	0.47	16
CHILE	5.06	5.02	6.09	0.24	16

Tab. 1 $\delta^{18}O$ values of gem-quality apatite in this study. σ – standard deviation, n – number of measurements

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[2] Blake et al. (2010) *Nature* **464**, 1029-1032. [3] Frei et al. (2005) *Geochim Cosmochim Acta* **69**, A794.

Use of pXRD in the quantitative assessment of alteration in epithermal Au deposits

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X-ray powder diffraction (XRD) is a well-established tool in the Earth Sciences, as it allows for the identification and quantification of mineral assemblages. For epithermal Au systems, the quantification of alteration mineral assemblages can allow geologists to characterise the geometry of a deposit and draw inferences regarding the fluid evolution and environmental conditions of deposition (e.g., pH, T). Traditionally, XRD devices have largely been restricted to laboratories; however, advances in XRD sample holders and X-ray sources have allowed for the development of portable XRD (pXRD) devices. This study assessed the validity of the Olympus Terra pXRD instrument for qualitative and quantitative studies of hydrothermal systems through comparisons with data from laboratory XRD and XRF techniques for both synthetic mixtures of natural minerals and a variety of samples from the Kulumadau epithermal gold deposit, Woodlark Island, Papua New Guinea and the Drake Goldfield of NE NSW, Australia.

Diffractograms of synthetic mineral mixtures with known concentrations of quartz, kaolinite, muscovite, albite, and pyrite were firstly analysed quantitatively using the Rietveld-based Siroquant technique and showed good overall agreement for both devices. Results from the pXRD unit for collection times of 5, 10, 20, and 40 minutes for representative samples spanning a range of rock types and alteration styles revealed that 5 minutes was sufficient for qualitative analysis, even of minor phases. Quantitative mineral estimates for 20- and 40-minute data sets were compared directly to estimates made using the Empyrean II data and showed excellent correlation with R2 values of >0.90 for all major mineral phases (i.e., >5%). Due to its portability, robustness, minimal sample preparation, relatively fast collection times, and excellent correlation with laboratory-based XRD devices, the pXRD has been shown to be of great use for rapid acquisition of quantitative mineralogical data by the exploration geologist, allowing for more informed and faster decisions during drilling programs.

"*Soft*" ablation cell for LA-ICP-MS investigations of large precious samples

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A novel "soft" ablation cell for laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS) has been developed. The cell includes a sample holder with a gas inlet, a local aerosol extraction device fixed and connected to the outer transfer tube, and a large soft amorphous outer chamber, which is not limited to samples of particular size or shape and is insensitive to sample surface. Compared to commonly applied standard ablation cell, the sensitivity was increased by a factor of 3.8–18.0 and 1.1–1.3 for single-pulse and continuous ablation respectively, the washout time was 94% faster and provides washout times within approx.1s for 99.9% of the signal. The signal intensities obtained in six different three-dimensional positions in the cell remained almost unchanged, and the RSD was less than 3.21%. The evaluation of the ablation cell demonstrates high resolution capabilities on large solid samples. It promises to provide direct, *in-situ*, and non-destructive analysis of large precious samples in micro-area geochemistry, archaeology, gem Identification, forensic science, and conservation science.

In-situ carbon isotope analysis by laser ablation MC-ICP-MS

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Carbon isotopes have been widely used in tracing a wide variety of geological and environmental processes. The carbon isotope composition of bulk rocks and minerals was conventionally analysed by isotope ratio mass spectrometry (IRMS), and more recently secondary ionisation mass spectrometry (SIMS) has been widely used to determine carbon isotope composition of carbon-bearing solid materials with good spatial resolution. Here we present a new method that couples a RESOlution S155 193 nm laser ablation system with a Nu Plasma II MC-ICP-MS, with the aim of measuring carbon isotopes *in situ* in carbonate minerals (i.e., calcite and aragonite). Under routine operating conditions for $\delta^{13}C$ analysis, instrumental bias generally drifts by 0.8 – 2.0‰ in a typical analytical session of 2-3 hours. Using a magmatic calcite as the standard, the carbon isotopic composition was determined for a suite of calcite samples with $\delta^{13}C$ values in the range of -6.94 to 1.48‰. The obtained $\delta^{13}C$ data are comparable to IRMS values. The combined standard uncertainty for magmatic calcite is <0.3‰ (1s). No significant matrix effects have been identified in calcite with the amplitude of chemical composition variation (i.e., MnO, SrO, MgO, or FeO) up to 2.5 wt%. Two modern corals were investigated using magmatic calcite as the calibration standard, and the average $\delta^{13}C$ values for both corals are similar to the bulk IRMS values. Moreover, coral exhibits significant heterogeneity in carbon isotope compositions, with differences up to 4.85‰ within an individual coral. This study indicates that LA-MC-ICP-MS can serve as an appropriate method to analyse carbon isotopes of carbonate minerals *in situ*.
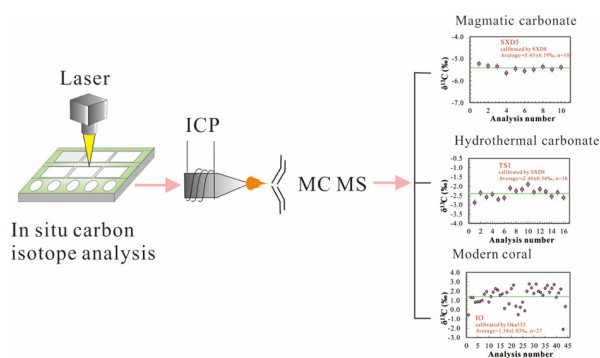
A new pyrrhotite reference material for micro-beam determination of sulfur isotope

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High spatial resolution sulfur isotope analyses of sulfide minerals by secondary ion mass spectrometry (SIMS) provide a powerful tool for understanding the geological and biological processes. However, the lack of isotopically well-characterised sulfide reference material, severely hinders the application of micro-beam analyses of sulfur isotope analyses to understand the past geological histories. A new pyrrhotite reference material (JC-Po) collected from the Jinchuan Ni-Cu-PGE sulfide deposit in Gansu province, northwest China were used for micro-beam (SIMS) analysis of sulfur isotope. The results demonstrate that this sulfide reference material is a compositionally and isotopically homogeneous standard for *in-situ* sulfur isotope analysis. The $\delta^{34}S$ value of JC-Po was determined by elemental analysis-isotope ratio mass spectrometry (EA-IRMS) and standard dual-inlet isotope ratio mass spectrometry (Dual-inlet IRMS), which yielding the mean $\delta^{34}S$ value is -0.11 ± 0.17 ‰ (1σ).

U-Pb geochronology of grandite by femtosecond laser ablation high resolution inductively coupled plasma mass spectrometry

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Grandite commonly occurs in alkaline and ultramafic intrusions and Fe, Cu, Mo, and W skarn mineralisation. This type of garnet often has high U and Th coupled with negligible common Pb. In this case, dating garnet may potentially provide direct constraints on the time and history of magma emplacement and hydrothermal processes.

We conducted U-Pb isotopic dating of grandite garnet from several skarn deposits in China. The results demonstrate *in-situ* U-Pb garnet dating provide a robust tool for determining the timing of hydrothermal ore forming processes by using femtosecond laser ablation high resolution inductively coupled plasma mass spectrometry (Fs-LA-HR-ICP-MS). The matrix effects between zircon and andradite have been investigated. Our observations suggest that the matrix effects can be neglected by using femtosecond laser ablation. We successfully obtained the U-Pb age of three garnets samples from: the Yixingzhai gold deposit, the Jinshandian iron deposit, and the Tongshankou copper-iron deposit. Three hydrothermal garnets yielded U-Pb dates of 140 ± 2 Ma, 127 ± 2 Ma, and 149 ± 5 Ma, which are consistent with the ages determined from U-Pb zircon ages of their respective ore-related intrusions, which supports the reliability of our analytical protocol. The observed results improve our understanding and application of garnet geochronology in future research.

Li abundances of magmatic zircons in Eocene-Oligocene Porphyry Cu Mineral systems of Yunnan, China

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Li abundances of magmatic zircons in Cu ± Mo ± Au ore-fertile and ore-barren Eocene-Oligocene potassic intrusions in western Yunnan, SW China [1,2], were measured by SIMS [3] and compared in this study to examine their potential in tracing metallogeny. Our preliminary results demonstrate that neither Li concentrations nor Li isotopic ratios (δ^7Li) of zircons show correlation with those of their host rocks and zircon $\delta^{18}O$ values.

In ore-barren potassic adakite-like quartz-monzonites and shoshonitic rocks (SiO_2: 62 ~ 65 wt.%) [1], Li contents of zircon are low, mostly <0.01 ppm, similar to those in oceanic crust [4,5]. Zircons in ore-barren potassic adakite-like granites (SiO_2: >70 wt.%), formed by partial melting of a thickened potassic and mafic lower crust with minor input from an older igneous felsic component [1], have relatively higher Li concentration. Their Li isotopic values are mainly negative, lower than those of their host rocks (δ^7Li: +1.0 ~ +5.1), and scattered (mainly -40 ~ 0) as a result of kinetic fractionation [5].

Zircons in the ore-fertile intrusions of the Machanqing porphyry Cu-Mo (SiO_2: 68.6 and 70.8 wt.%), Beiya skarn Au (SiO_2: 62.5 ~ 69.9 wt.%), and Yao'an Au porphyry deposits (SiO_2: 63.1 wt.%)[2] were also selected for Li analyses. In the Beiya skarn Au deposits, zircon has consistent characteristics in Li contents and isotopes with those in ore-barren rocks ([Li]: <0.05 ppm, mostly <0.01 ppm; δ^7Li: mainly -30 ~ 0). However, Li contents in zircons of the Machanqing porphyry Cu-Mo and Yao'an porphyry Au deposits, are elevated (0.01 ~ 0.5 ppm). Their Li isotopic values are heavier and show narrower clusters (δ^7Li: mainly -20 ~ 0). It is thus proposed that Li abundances and its isotopic composition of zircon are affected by porphyry mineralisation, which may be able to fingerprint such mineralisation process.

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Development of CA-ID-TIMS zircon U-Pb dating technique at Institute of Geology and Geophysics, Chinese Academy of Sciences

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Zircon U-Pb geochronology using isotope dilution-thermal ionisation mass spectrometry (ID-TIMS) is the most precise isotope dating technique up to now (Schmitz and Kuiper, 2013). Recently, a chemical abrasion (CA)-ID-TIMS zircon U-Pb dating technique (Mattinson, 2005) has been developed at Institute of Geology and Geophysics, Chinese Academy of Sciences.

First, a ^{205}Pb-^{235}U mixed spike was prepared and calibrated against solutions made from Pb NIST 981 and GBW04205 U_3O_8 standards. The isotopic compositions (mean ± 2SE, n = 6) of the spike were: $^{205}Pb/^{206}Pb$ = 1739 ± 122, $^{204}Pb/^{206}Pb$ = 0.111 ± 0.003, $^{207}Pb/^{206}Pb$ = 0.852 ± 0.013, $^{208}Pb/^{206}Pb$ = 1.998 ± 0.025 and $^{235}U/^{238}U$ = 1251 ± 5. The Pb and U concentrations (mean ± 2SE, n = 6) in the spike were 9.223 ± 0.015 pmol/g for ^{205}Pb and 476.38 ± 0.23 pmol/g for ^{235}U.

Second, the sources of the laboratory Pb blanks including exposure blanks, reagent blanks and beaker blanks were thoroughly investigated. It was likely that the Pb leached from the wall of the Teflon beakers which used for collection of Pb-U from the column chemistry was a main source of the Pb blanks. At current, the procedural blanks were about 2 ~ 3 pg for Pb and 0.2 ~ 0.6 pg for U. The Pb isotopic compositions for the total procedural blanks have also been precisely measured. They (mean ± 2SE, n = 9) were about 17.92 ± 0.16 for $^{206}Pb/^{204}Pb$ and 15.36 ± 0.11 for $^{207}Pb/^{204}Pb$. In the near future, we will try our best to further lower the Pb procedural blanks.

Finally, several standard zircons including Temora 2, Qinghu and Plešovice, were analysed with the method. The analytical results of Pb/U ages were in good agreement with previously reported values. The analytical precisions (2RSE) of the weighted mean $^{206}Pb/^{238}U$ ages for these standard zircons were better than 0.25% (n > 7).

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Determination of the elemental composition and manganese valence state in Co-bearing ferromanganese crusts by wavelength-dispersive X-ray fluorescence analysis

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A complex study of the composition and properties of Co-bearing ferromanganese crusts of the world's oceans is the basis of the industrial development of this valuable ore. A promising tool for a comprehensive study of the processes of ore genesis in Co-bearing ferromanganese crusts is the development of wavelength-dispersive X-ray fluorescence (WDXRF) techniques that allow to estimate not only the elemental composition with high productivity but also the valence state of the elements on the basis of the influence of the chemical bond on the relative intensities of the $MnK\beta_5$ line and the $MnK\beta'$ satellite. The measurements were carried out by a WDXRF spectrometer S4 Pioneer (Bruker AXS). Certified reference materials of ferromanganese nodules, cobalt-manganese crusts, manganese and iron-manganese ores were used. Contents of Na, Mg, Al, Si, P, K, Ca, Ti, Mn, Fe, Co, Ni, Cu, Zn, Ba, Sr, Zr, Mo, Cl, as well as MnO_2 content were certified, which allowed additionally to calculate the average valence state of manganese. The dependence of the manganese valence state on analytic parameters (ratios of line intensities $MnK\beta_5/MnK\beta_{1,3}$ and $MnK\beta'/MnK\beta_{1,3}$) was approximated by a polynomial equation into which a term proportional to the total iron content was included. Analysis of the control samples by the titrimetric method showed that this technique can be used to quantify the manganese valence state.

31 samples of Co-bearing ferromanganese crusts were collected by I.S. Peretyazhko on Govorov and Kotzebue guyots (Magellan seamount) during the voyage 06-16 under the agreement with JSC "*Yuzhmorgeologiya*" and analysed. The samples were dried at a temperature of 120 °C to remove hygroscopic moisture and pressed onto a substrate of boric acid. According to XRF data the average manganese valence state in analysed samples varies from 3.62 to 4.00 (standard deviation was 0.1). Obtained analytical data provide the studying of the relationship between the layer concentration of ore and impurity elements in Co-bearing ferromanganese crusts with oxidation-reduction conditions of their formation.

The research was performed using equipment of the SB RAS Joint Use Centers (Geodynamics and Geochronology, Isotope-geochemical Research).

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Determination of sulfate sulfur in Au-bearing ores by wavelength-dispersive X-ray fluorescence analysis

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The data on sulfur chemical state are often required for geochemical investigations in sulfide ore analysis. Distribution of sulfides and sulfates is important for assessing of deposits genesis and recognising of its commercial value. The labour-consuming gravimetric method is commonly employed to find sulfate sulfur in ores. The intensities of some diagram lines and satellites of sulfur X-ray fluorescence spectrum are affected by sulfur chemical state. Satellite $SK\beta'$ is preconditioned by transition from the molecular orbitals, which, in addition to 3p electrons of sulfur, is considerably contributed by 2s and 2p electrons of the element chemically bonded with sulfur [1, 2]. In sulfate spectra $SK\beta'$ satellite intensity reaches 40 % of that of $SK\beta_{1,3}$ line, but in sulfide spectra this satellite is practically absent. Satellite $SK\alpha_{3,4}$ is preconditioned by transition from orbitals contributed by 2p electrons and also affected by chemical bond [3,4].

A set of sulfide ores samples was collected to recognise the possibility to quantitative determine sulfur chemical state in real geological samples. On the XRD data sulfur occurs as sulfide minerals wherein sulfur is chemically bonded basically with iron (pyrite, marcasite, arsenopyrite, chalcopyrite, pyrrhotite). The concentrations of total and sulfate sulfur were determined by a WDXRF spectrometer S4 Pioneer. Ratios of lines intensities of $SK\beta'/SK\beta_{1,3}$ and $SK\alpha_{3,4}/SK\alpha_{1,2}$ were used as analytical parameters for determination of sulfate sulfur from pressed powder pellets of sulfide ore samples. Results of XRF determination of the sulfate and total sulfur ratios were compared with the data of gravimetric analysis. The presence of lead M-series lines in the field of measuring sulfur spectrum lines can significantly influence on the intensity of analytical lines, therefore selected analytical parameters can be used for analyzing samples not containing significant lead abundance. The technique proposed shows an evident advantage in terms of time and expenditures as compared with gravimetric analysis.

The research was performed using equipment of the SB RAS Joint Use Centers (Geodynamics and Geochronology, Isotope-geochemical Research).

[1] Uhlig et al. (2016) *X-ray Spectrometry 45, 133-137.* [2] Chubarov et al. (2016) *X-ray Spectrometry 45, 352-356.* [3] Kavčič et al. (2004) *Nuclear Instruments and Methods in Physics Research B: Beam Interactions with Materials and Atoms 222, 601-608.* [4] Sattar et al. (2007) *X-ray Spectrometry 36, 369-372.*

An evaluation of total uncertainty in WDXRF geoanalysis

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The compositional diversity of rocks, soils, and minerals, and their wide ranges in elemental concentrations, present difficulties in the evaluation of total uncertainty in WDXRF geoanalysis. For evaluation of total uncertainty in our laboratory we have adapted the Nordtest method [1] to encompass a range of concentrations. We derive power functions which express the total uncertainty for each analyte as a function of concentration. Our functions are similar to those proposed by Horwitz [2]. We determined the precision of our instrument by analyzing 11 fused pellets each of 16 diverse materials with wide ranges in analyte concentrations, and fit power functions to the precision of each analyte as a function of concentration. Similarly, we analysed approximately 150 reference materials (RMs) for validation of our calibration and fit power functions to the average difference between the RM certified values and our analytical results for each analyte as a function of concentration. We compiled uncertainty values for geologic RMs of diverse types and fit power functions to those uncertainties as functions of concentration. We evaluated instrument drift for all analytes over a period of several months spanning the precision and validation measurements and found it to be essentially nil. To estimate total uncertainty, we adapted the Nordtest procedure, and used the power functions fit to our precision, validation, and RM uncertainies to calculate the uncertainty contributed by each component over a range of concentrations appropriate to each analyte, and then summed the squares of those uncertainties at those concentrations. We then took the square root of the sum of those squares and multiplied by 2 to estimate the total uncertainty U (2-sigma) at those concentrations. The final step was to fit power functions to the total uncertainty as a function of concentration for each analyte.

These uncertainty estimations illustrate the superior precision of WDXRF compared with stated RM uncertainties at concentrations above approx. 200 ppm (analyte dependent), and conversely, the inferior precision below that concentration.

To ease the calculation of total uncertainty for our users we provide an on-line link to a workbook wherein they can paste their data set and output the total uncertainty for each analyte for each sample.

[1] http://www.nordtest.org/register/techn/tlibrary/tec537 .pdf. [2] Horwitz & Albert (1995) *Fresenius' Journal of Analytical Chemistry, 35, 507-513.*

Use of a single background per analyte in WDXRF geoanalysis: Minimisation of spectral interference

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Many background subtraction methods are employed in WDXRF geoanalysis. The most common is the use of two backgrounds, one on either side of the analyte peak. While effective at high 2-theta angles where background is nearly flat for all matrices, at low 2-theta WDXRF backgrounds are curved and the degree of curvature is a function of matrix composition, with lighter matrices having greater curvature. The two-background method thus always underestimates net peak minus background intensities at low 2-theta. In addition, the use of two background measurements increases the chance of spectral interference and hence the need for correction of the background intensity. Another commonly employed method is to fit a polynomial function to a curved background by measurement at multiple 2-theta angles. That method addresses the change in background curvature with matrix, but vastly increases the odds of spectral interference. If any spectral interference is not corrected prior to curve fitting, the background measurement will be in error. To overcome the curvature change with matrix and minimise the potential for spectral interference in background measurement, we measure only a single background for each analyte. To establish the relationship between a single background and the analyte-free peak position intensity for each analyte we counted intensities on 20 glass pellets doped with spec-pure silica and varied amounts of several elements, including Fe, Ca, Mn, Ti, K, and Mg. All of the major and minor elements, except Mn, and the minor and trace elements Cl, Cr, Cu, Zn, and Zr required extensive scanning instead of simple counting due to ubiquitous contamination even in spec-pure compounds. Scans over contaminated peak positions were fit by eye to estimate uncontaminated peak intensities. Compton scatter intensities were also collected at the same time and several means of background correction were examined. These included linear versus parabolic fitting of the peak and background intensity relationship, peak to background ratios (both linear and parabolic fits), and several expressions involving Compton scatter, for example peak minus background versus Compton scatter intensity. Simple linear fitting was superior in most cases, although some analyte relationships were best fit with parabolas, especially F and Mo. Most intercepts were near zero counts per second, but we include the intercepts in our corrections. Use of the Compton scatter intensity was the best choice only with Ni due to its presence in the tube spectrum. In general, the use of theoretical or Compton background correction introduces far more uncertainty than counting a background near the analyte peak.

U-Pb doped synthetic reference material for *in-situ* hematite geochronology

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Uranium-bearing hematite is widespread within iron-oxide-Cu-Au deposits and ores associated with banded iron formations of South Australia [1-4]. Valuable age data has been obtained via LA-ICP-MS applying GJ-1 Zircon and mixed U-Pb solutions as the primary reference materials [1,2,5], as well as high precision ID-TIMS U-Pb data, which is independent of the need for solid reference materials [6]. However, routine high-accuracy microbeam dating of hematite requires near-matrix-matched reference materials, of which suitable natural specimens have currently not been identified. We report a method to produce U-Pb doped hydrated ferric oxide, through slow introduction of ~150 ppm U/Pb solutions, calcinated at 700 °C to dehydrate and convert to hematite. Polycrystalline hematite fragments, up to 1 cm-long, are commonly produced through this method. The material was mounted in epoxy, polished, and imaged by SEM in backscatter electron mode (BSE) to check for potential heterogeneity or inclusions of U-Pb-bearing phases. Further characterisation by LA-ICP-MS showed that the precision of the $^{207}Pb/^{235}U$ and $^{206}Pb/^{238}U$ (50 spot analyses) was 0.2% (95% confidence interval) and with a MSWD of 0.61, compared to the zircons GJ-1 (0.5% *n*=36), Plesovice (0.6% *n*=18), and 91500 (0.6% *n*=9) analysed in the same analytical session. U concentrations are ~70 ppm, and Pb ~35 ppm. The material displays both dark and bright areas under BSE, with slightly varying U/Pb most likely formed during heating steps. Downhole U-Pb profiles for the synthetic material displayed a similar fractionation trend to natural hematite analysed within the same analytical session. To further evaluate homogeneity, the samples will be measured by ID-TIMS to define absolute U-Pb ratios, and subsequently by SHRIMP to examine U-Pb isotope homogeneity at the micron-scale. Our aspiration is that this material can be used routinely as a reference material for microbeam techniques.

[1] Ciobanu et al. (2013) *Precambr. Res.* **238**, *129-147.* [2] Courtney-Davies et al. (2016) *Minerals.* **6**, *3, 85.* [3] Verdugo-Ihl et al. (2017) *Ore Geol. Rev.* **91**, *173-195.* [4] Keyser et al. (2018) *Ore Geol. Rev.* **93**, *337-360.* [5] Apukhtina et al. (2017) *Econ. Geol.* **112**, *1531-1542.* [6] Courtney-Davies et al. (2018) *Chem. Geol. (in review).*

Zirconium stable isotope analysis by double-spike MC-ICP-MS

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Zirconium and other high-field-strength elements (HFSEs; Hf, Zr, Ti, Nb and Ta) have been extensively studied within the field of high-temperature geochemistry. These elements are not readily incorporated in the crystal lattice of most mantle minerals, and thus behave incompatibly during mantle melting events, leading to their relative enrichment in the crust (>100 µg g^{-1}) compared to bulk silicate Earth (~10 µg g^{-1}). In addition, they are relatively immobile in aqueous fluids, and so resistant to late metamorphic and alteration effects. Consequently, HFSE are excellent tools for tracing historic mantle depletion events and the petrogenesis of igneous rocks. However, despite their extensive use in geochemistry, the HFSEs have received relatively little attention in terms of their stable isotope geochemistry.

In addition to the properties of HFSE above, zirconium is among the most refractory trace elements found on Earth, and highly resistant to evaporation and condensation as well as metamorphism and late alteration. Furthermore, zirconium is the major cation in zircons, the oldest preserved minerals on Earth, and thus Zr stable isotopes could serve as powerful tracers of source evolution and growth of continental crust through geological time, with less potential disruption from the effects of metamorphism and alteration that can impact other stable isotope systems.

We have developed methods for purification of Zr from geological samples and ultra-precise Zr stable isotope analysis by double-spike MC-ICP-MS. In addition to these methods, we will present the first Zr stable isotope data from a range of geological reference materials, including individual Plešovice zircon grains, and from a suite of lavas from the Hekla volcano, Iceland. We will then discuss the use of Zr as a tracer in igneous processes, as well as potential future uses for this new geochemical tracer.

Comminution and sizing of gold particles in geological samples and reference materials

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We demonstrate, for the first time, a reliable technique for the comminution of gold particles in mineral matrices by ultrafine grinding using standard commercial laboratory reciprocating bowl type pulverisers of up to 2 kg capacity. Conditions for effective and accurate ultrafine grinding include optimising charge weight, using grinding aids to prevent caking, and managing smearing losses of gold to grinding media through incorporation of hard matrix diluents or washes in the analysis. We show that gold particles of up to nominally 1000 micron diameter can be readily comminuted to 95% < 75 micron in practically short grinding times of up to 5 minutes, and that 95% < 45 micron is achievable. To support this work we have developed technique for qualified interpolation of maximum gold particle size in samples (interpolated particle size or IPS) from sampling theory, using analytical replicate variance. IPS has been calibrated against sieve fraction analysis of samples and promises a useful means of characterising gold particle size distribution rapidly, and at particle sizes below the practical limitations of sieve analysis. Work to date has been directed to µg g^{-1} levels of gold applicable to gold resource definition, but has useful implications for the study of samples with ng g^{-1} concentrations of gold for grassroots exploration and fundamental geochemical application. Using IPS we review literature data pertaining to gold ore sample preparation and analysis, and the question of homogeneity in geological gold reference materials.

The effect of laserprobe optical path design on laser ablation of minerals with low melting points

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Parameters which have been demonstrated to control interaction of the laser beam with the sample include laser wavelength [1], laser pulse width [2] and laser beam fluence [3]. In this study we suggest that the optical path design, which affects the size and shape of the beam outside the focal plane, is the likely primary influence on the depth and size of ablation craters.

The shape of the laser beam is controlled by the working distance and numerical aperture of the objective lenses. Higher numerical aperture and shorter working distance result in a more focused beam which size and shape change rapidly outside the focal plane. The number of optical components in the beam path has an impact on the distribution of energy across the beam.

We present a comparison of ablation behaviour of observed on 6 different systems: two different laserprobes (ASI RESOlution SE and Cetac Analyte G2) which employ the same ATL excimer laser, 193 nm wavelength ~ 5 ns pulse width, but use different optical path design for focusing the beam onto the sample surface; two laserprobes which use a 213 nm Nd-YAG solid state laser and also have different optical path design (NWR UP213 and Cetac LSX-213 G2, both with ~ 4 ns pulse width); and two laserprobes which use CompexPro excimer laser from Coherent, 193 nm wavelength ~ 20 ns pulse width (ASI RESOlution LR and Cetac Analyter HE).

We show that when used with the same fluence; laserprobes with higher numerical aperture objective lenses produce shallower craters which are wider compared to the nominal spot size as determined by ablating materials with higher melting points.

LA-ICP-MS analysis of chalcopyrite, pentlandite, bornite and other sulfides with low melting points using laserprobes with low numerical aperture objective lenses leads to rapid decline in aerosol intensity (signal drop-off) during ablation at a fixed position, when using a laser beam size < 25 micron, cause by deeper craters being clogged by the melted material.

Higher laser beam fluence decreases signal drop-off on all systems, but also leads to increased crater size and significant dispersion over the sample surface of particles produced during ablation, thus leading to potential cross-contamination between different locations on the sample, and we do not recommend using higher fluence to minimise signal drop-off [4].

[1] Guillong & Gunther (2002) *JAAS* **17**, *831.* [2] Günther et al. (1997) *JAAS* **12**, *939.* [3] Eggins et al. (1998) *ASS*, **127-129**, *278.* [4] We acknowledge support of Teledyne CETAC Technologies by providing access to LSX-213-G2

In-situ Rb-Sr dating of authigenic clays from soils and sediments: Potential and limitations

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Reliable dating of igneous and sedimentary rocks is essential for the calibration of geological time scales and for the absolute age determination of major tectonic and depositional events throughout the Earth's history. Such geochronological constraints are particularly important for the studies and exploration of Proterozoic basins, which cannot rely on biostratigraphy due to a lack of reliable macrofossil record. The depositional ages of Proterozoic basins can, however, be constrained via dating of selected authigenic minerals, which readily form either during the sediment depositions (i.e., glauconite), and/or during the early stages of sediment diagenesis (i.e., authigenic illite).

This contribution presents analytical results of *in-situ* Rb-Sr dating of Proterozoic illites hosted in shales, as well as recent and biologically-mediated illites formed in modern soils, which were mineralogically mapped at micro-scale level (via QEMSCAN) and analysed by a laser ablation (LA) reaction cell ICP-MS, or so-called LA-QQQ approach [1, 2]. Here, we will discuss the analytical setup, research potential and limitations of this novel micro-analysis Rb-Sr dating technique for geochronological applications on illites from soil/regolith/sediment sytems, illustrated on selected case studies from both recent and Proterozoic low-temperature Earth's surface environments.

[1] Jensen L. (2017) *Honours Thesis, University of Adelaide.*
[2] Zack T. and Hogmalm K.J. (2016) *Chemical Geology,* **437,** 120-133.

Advanced 193 nm Laser Ablation

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Pulsed excimer lasers are the most powerful 193 nm laser processing tools on the market and have a history of success in demanding industrial laser markets such as refractive surgery, microelectronics manufacturing or laser engraving. High pulse energies of some 10 to several 100 mJ are available already from compact, stand-alone excimer lasers which are easily integrated into laser ablation systems for LA-ICP-MS solid sample analysis. This is because their 6,4 eV photons are generated directly without the need for frequency conversion. The high pulse energy is usually transformed into high fluence levels favourable in the case of optically transparent specimens or when analysing a variety of geological samples. The latest and most advanced 193 nm laser optical systems for LA-ICP-MS analysis such as the GeoLasHD provide nanosecond pulsed output for ablation with better than 1% rms shot-to-shot stability and on-sample fluence levels as high as 50 J/cm^2. LA-ICP-MS has come a long way since its introduction over 25 years ago. Whereas the first generation of laser systems started out with infrared lasers, today's most advanced solid sampling systems are predominantly using UV excimer lasers driving accuracy and precision achieved in analysing soils and sediments, gemstones, quartz samples, calcite, fluorite and other materials. Ultra-precise excimer lasers at 193 nm with extended communication features ensure optimum ablation pattern programming and get the most out of the resolution and signal processing capabilities of the attached ICP-MS instrumentation.

Ga isotopic compositions of various reference materials

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Recently, Ga isotopes have attracted increasing research interest in the geological community. High precision determination methods for Ga isotope in geological samples have been proposed by three laboratories, including the Institute de Physique du Globe de Paris (IPGP), the Institute of Geochemistry, Chinese Academy of Sciences (IGCAS) and the State Key Laboratory of Geological Processes and Mineral Resources (GPMR), however these use diverse relative reference materials (RM). High purity Ga metal NIST SRM 994 was served as relative RM at GPMR and IGCAS, while an ICP standard solution was used at IPGP. Furthermore, the Ga isotopic ratios differed by 0.80‰ between GPMR and IGCAS for the same geological standard (e.g. BHVO-2). Thus, inter-laboratory comparison is still unachievable for the Ga-isotope community. In this study, two approaches were used to help establish the intercalibration between different laboratories. Two commercially available Ga standard solutions purchased from Alfa Aesar (#Lot 626491D) and NIST (SRM 3119a) were measured repeatedly relative to SRM 994, aiming to establish the conversion relationship for data comparison. On the other hand, four commonly used geological RMs were measured to monitor bias induced by Ga purification procedures. Samples were purified by two-stage ion exchange chromatograph and measured by MC-ICP-MS. The instrumental mass bias was corrected by using a model of standard-sample bracketing with internal normalisation, and Cu was used as an internal standard which was added to both the sample and standard solutions. The comprehensive data set presented in this study serves as a reference for quality assurance and interlaboratory comparison of high-precision Ga isotopic compositions.

Determination of nitrogen in experimental and natural samples using EPMA and CHNS analyzer

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The determination of nitrogen in geological and experimental charges with electron probe microanalyzer (EPMA) has been a challenging task due to the element's high volatility, low X-ray count rates, and lack of matrix-matched reference materials.

We have calibrated a new EPMA routine for concurrent analysis of nitrogen in silicate minerals using the LDE-B crystal for wavelength dispersive spectrometers of a JEOL JXA 8200 Superprobe at the Johannes Gutenberg-University, Mainz. The acceleration voltage was set to 10 kV, a minimum value to excite K-alpha lines of heavy elements such as Fe. Peak counting times were 160 s for N, with a sample current of 40 nA with a defocussed beam 10-20 μm in diameter. As reference materials we used boron nitride (56.4 % m/m N) as well as synthesised NH_4-feldspar (5.25 % m/m N) and NH_4-bearing glass (2 % m/m N). Limits of detection for N were found to be 500 μg g^{-1}.

This routine was used to determine nitrogen contents in high pressure experiments that featured reaction between sediment and depleted peridotite, simulating nitrogen recycling within subduction zones. The sediment melted partially and reacted with the depleted peridotite to produce phlogopite. Nitrogen contents of glass and phlogopite yielded mass fractions of 0.5-1.5 % m/m N, and enabled the calculation of partition coefficients which were found to be ~0.5 for D_N (phlogopite/glass).

Besides experimental charges we measured minerals in various natural micaceous ultramafic rocks. Most samples fall below the detection limit of the EPMA, though those from the Finero Massif in Italy were found to contain up to 0.1 % m/m N. In addition, samples were measured using an Elementar VarioCube CHNS analyzer at Macquarie University, Sydney. Limits of detection were found to be 30 μg g-1 N due to the adhesion of air on the sample surface. We are working on lowering this detection limit by drying samples in a vacuum oven coupled with gas exchange of air by Ar.

Assessment of trace-element homogeneity in gem quality zircons from Mud Tank, NT

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The Mud Tank zircon is found in the Strangeways Range in NT, Australia. The fossicking area is famous for the abundance and unusually large size of the zircons, this makes them perfect for cutting as gem stones.

The Mud Tank zircon is currently used as a reference material for U/Pb [1] and Lu-Hf [2] isotopic analyses at Macquarie University and in other analytical laboratories worldwide. The aim of this study was to assess the suitability of gem quality Mud Tank zircons as reference material for Laser Ablation Inductively Coupled Plasma Mass Spectrometry (LA-ICP-MS) trace-element analysis. Off cuts from nine gem quality zircons (Fig. 1) were mounted and polished for imaging and chemical analysis.

Figure 1: Nine gem quality Mud Tank zircons

The nine crystals showed a range of internal structures and trace-element compositions. The trace-element compositions varied between the different crystals but there was also a range within certain crystals (Fig. 2). The trace-element concentrations were usually higher in the darker coloured crystals and had a larger variation in the more complexly zoned crystals revealed by cathodoluminescence (CL) studies. Therefore, we would recommend assessing individual crystals before they are used as trace-element reference materials.
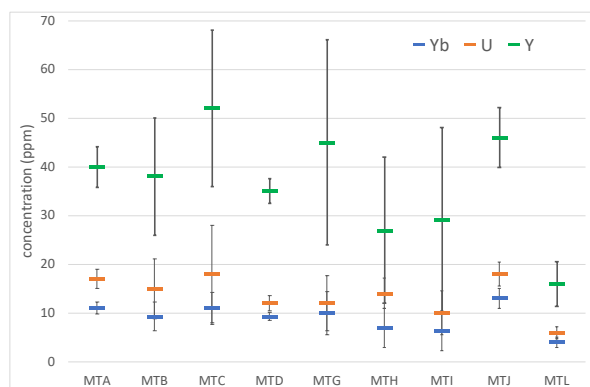

Figure 2: Variation of Yb, U and Y concentrations in the nine different crystals (symbol=mean, error bars=range, n=10 except MTL n=28)

[1] Jackson, S.E., Pearson, N.J. et al. (2004) *Chemical Geology, 211,* 47-69. [2] Woodhead, J.D. and Hergt, J.M. (2005) *Geostandards and Geoanalytical Research, 29,* 183-195.

Homogeneous pressed powder pellets as new MRMs for *in-situ* microanalytical techniques

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While *in-situ* analytical instrumentation for the direct elemental and isotopic analysis of solid materials has undergone continuous and significant improvement over recent years, development of well-characterised and homogeneous microanalytical reference materials (MRM) for calibration and validation of analytical data has been delayed. Ideally, MRM must be homogeneous down to the single micrometer scale for major, minor, trace, and ultra-trace elements and isotopes, withstand high-vacuum and impact of high-energy electron, ion, and photon beams, stable in its physical and chemical properties over time and under various environmental conditions, certified following ISO guidelines, and available for a wide variety of materials.

We developed a method for manufacturing undiluted, binder-free pressed powder pellets [1] with particle grain size down to the nanometer range (D50 <170 nm), extremely low roughness of pellet surface (RA <50 nm), and excellent within and between pellet homogeneity. This technique has been applied so far to a wide range of very different sample types: biogenic carbonates (foraminifera, clam shells, red algae, corals), speleothem, silicate rocks, iron ores and banded iron formation, manganese nodules, sulfides UQAC-FeS, refractory minerals, plutonic and volcanic rocks, fly ash, bone-apatite, minerals for Rb/Sr age-dating [2] etc. We successfully blended different materials opening new ways for producing e.g., series of elemental and isotopic calibration standards. These "*nanopellets*" have been successfully used with LA-ICP-MS, LIBS, µ-XRF, handheld-XRF instruments, and with EPMA, PIXE, SIMS. Hence, nanopellets proved to be a new and, possibly, universal matrix-matched MRM for many custom solid materials to be used with many *in-situ* analytical techniques.

In addition, this way of sample preparation bears the potential of completely replacing conventional tedious and time-consuming wet-chemistry procedures for bulk analysis, and this holds true in particular for refractory samples like ceramics, granites, ultramafic rocks, and samples with volatile or easy-to-contaminate components (e.g., B). Here we give an overview of the present state of development of new MRM [3] and their characterisation in terms of grain size distribution, surface topography, porosity, homogeneity, and accuracy of analytical results for both elemental (major, minor, trace and ultra-trace elements) and isotopic (Sr, Li, B, O) composition.

[1] Garbe-Schönberg D & Müller S, (2014) *JAAS, 29, 990*. [2] Hogmalm KJ et al (2017) *JAAS, 32, 305*. [3] www.my-standards.com; see also paper by D. Savard et al. this conference.

Removal of Hg interferences for common Pb correction when dating minerals by LA-QQQ-ICP-MS

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LA-ICP-MS is a commonly used technique for U-Pb dating of zircons and other accessory minerals such as apatites, monazite, and rutiles. The dating of these minerals is often complicated by the presence of non-radiogenic Pb.

This common Pb can be corrected using the non-radiogenic ^{204}Pb isotope, where the isobaric overlap of ^{204}Hg must be corrected via the analysis of ^{202}Hg. However, Hg is a common contaminant in many LA-ICP-MS systems where the measured 204 amu signal is predominantly ^{204}Hg. This can limit the applicability of the ^{204}Pb corrections. The recent development of QQQ-ICP-MS instruments provides a means of removing the ^{204}Hg interference and subsequently more accurate common Pb corrections.

A RESOlution 193nm excimer laser ablation system was coupled with an Agilent 8900 QQQ-ICP-MS, using NH_3 in the collision/reaction cell. Hg is highly reactive with NH_3 via the charge transfer reaction: $Hg^+ + NH_3 \rightarrow Hg^0 + (NH_3)^+$, whereas Pb does not react [1]. This method removes more than 99.98% of the Hg signal, enabling interference free common Pb correction with the equivalent of 10,000 cps ^{202}Hg as measured by conventional single quadrupole ICP-MS. In addition, the sensitivity of all Pb isotopes doubled in NH_3 mode, while the U sensitivity was comparable between NH_3 and standard conditions.

This method was tested with a range of common Pb bearing apatite, titanite, and rutile samples. Accurate correction was achievable using ^{204}Pb at much lower levels than previously possible with a single quadrupole ICP-MS.

[1] Woods, G. (2014) Agilent Technologies Application Note 5991-5270EN

A suite of nine soil reference materials for targeted environmental geochemical analysis

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M. RAJA[1], A.L. MARRIOTT[1], J. MATHER[2], P. HEATH[2]

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Accurate chemical analysis depends on well characterised Reference Materials with appropriate matrix. The need for the determination of as many elements as possible has grown as the number and variety of applications has developed. Existing sets of soil reference materials have proved popular and supplies have become depleted, especially in those that had a comprehensive range of certified elemental concentrations.

A suite of nine soil samples have been collected at rural sites across Ireland, selected to represent a diverse underlying geology and soil matrix (i.e. limestone, sandstone, shale, granite, serpentinite, Pb-Zn deposits). The suite comprises soils that have enhanced levels of trace elements that have environmental significance (e.g. As, Cd, Hg), impact upon human and animal nutrition (e.g. Se, I, Co, Mo, Cu) and are of particular interest in geochemical exploration (e.g. Cr, Ni, Zn, Pb, U) as well as those of complementary matrix with relatively low levels of such elements. Together they represent a comprehensive set of materials that cover a range of geology on a national scale.

The materials have been produced in 30-75 kg quantities, for which a bespoke mixing barrel has been designed to increase the efficiency of homogenisation. Representative sample splitting reduced portion sizes to 30-80 g suitable for dispatch to laboratories. The materials have been tested for homogeneity using XRFS for 54 elements and for loss on ignition. A cooperative analysis exercise is being undertaken to establish robust composition concentration values and their uncertainties.

Reference Materials preparation and characterisation has been carried out at BGS over the past 50 years. Following an interlaboratory investigation of accuracy in ore analysis carried out by the then Institute of Geological Sciences in 1968, further ores and mineral concentrates have helped establish accurate and consensual analysis in the mining sector and, more recently, soil and stream sediment samples are helping provide Quality Control for multielemental geochemical analysis programmes and contaminated land analysis.

Provenance determination of cigars using $^{11/10}$B ratios by ICP-QMS with single pulse signal acquisition mode

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Boron is absorbed by plants from the source soil and water, producing B isotope ratios (IRs) which could reflect those of the sources, thus indicating the provenance of products [1]. An accurate and precise method was established based on a single pulse signal acquisition mode ICP-MS for $^{11/10}$B ratio measurements, and was then used for the geographic origins determination of cigars.

The internal precision of B IRs which obtained by single pulse mode were gradually increased (from 0.27 to 0.08%) with the increasing intensities and agreed with the principle of lowest theoretical statistical error (TSE), and the poorer RSDs were obtained by dual mode (0.16–0.24%) and single analog mode (0.10–0.13%). Thus, the single pulse acquisition mode was conducted with the relative optimum B concentration range (80-100μg L-1).

The seventeen cigar samples from eight countries were digested and diluted to the above recommended Boron concentration range (the dilution factor>300). Without matrix separation [2], the matrix effect from major elements could be neglected. After mass bias correction by SSB method with NIST 951a isotopic standard solution, the results of cigar samples fall into different distinct categories based on the δ^{11}B values were shown in Figure. 1. The obtained δ^{11}B values of cigar samples were ranged from +2.29 to +27.19 ‰ with an average external precision (N=5) of 0.27-0.98‰. It has sufficient precision to distinguish the cigars originating from different geographic regions.

[1] C.M.R. Almeida et al. (2004) *Food Chem, 85, 7-12.*
[2] W. Guo et al. (2016) *Rsc Advances, 6, 47394-47401.*

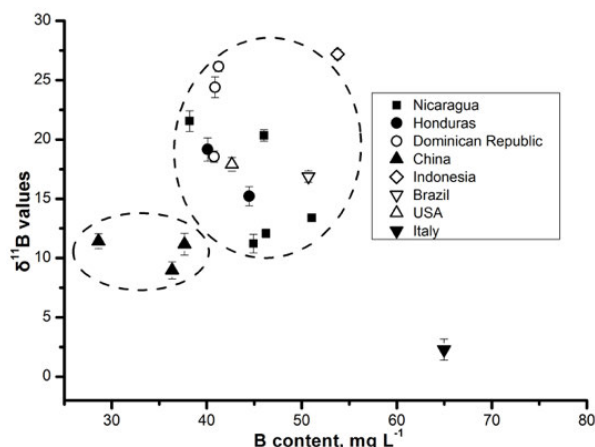Figure 1. Provenance determination of cigars using δ^{11}B (‰) values.

Is the handheld XRF devise a useful field tool for volcanologists?

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The handheld X-ray fluorescence (XRF) devise has great potential in the field to be able to produce high-resolution, stratigraphical geochemical profiles of volcanic deposits in little time. However, the accuracy of this technique requires testing for volcanic deposits which often have variable grainsize, homogeneity, vesicularity and moisture content. In this study, major element contents determined by conventional XRF on fused glass discs and trace element compositions determined by solution ICP-MS are compared with the results from handheld XRF analysis of the same samples as hand specimens and rock powders. We provide the limitations of the XRF devise in the field and discuss whether it can be used for quantitative data collection of volcanic deposits in addition to sample targeting.

A practical method for measuring high precision calcium isotope ratios without chemical purification for calcium carbonate samples by multiple collector inductively coupled plasma mass spectrometry

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As calcium isotope fractionation may occur during its diffusion, condensation, evaporation and some chemical reactions, it was widely used in geochemical investigation such as source tracing, paleoclimate, paleoenvironment studies and biology. Also, there are still increasing demands for measurements of Ca in carbonate samples to better understand paleoclimate change and help to unravel global matter cycling. Ca isotopes are commonly analysed by TIMS and alternatively measured by MC-ICP-MS. But the large ^{40}Ar and unignorably polyatomic interference generally precludes precise Ca isotope measurements by MC-ICP-MS. What's more, matrix elements which may affect Ca isotope measurement should also be removed before measurement. But for some samples with simple chemical matrices it is not cost-efficient to expend considerable effort in chemical purification when equivalent precision and accuracy could be achieved by a simpler one-step chemical purification process or for some high purity carbonate samples no chemical purification whatsoever.

We developed a practical method to measure Ca isotope ratios without column chemistry separation by MC-ICP-MS for calcium carbonate samples. The interference of ArH_2 and ion/electron scattering and sputtering from ^{40}Ar can be reduced by simultaneously tuning the ICP torch position, sweep gas flow rate of membrane desolvation and adjusting the collector mask slit position. The Sr^{2+} interference can be corrected when the Sr/Ca ratio in the sample is less than 0.1, which allows for the measurement of most calcium carbonate samples. By this method we obtained the $\delta^{44/42}Ca$ of an internal laboratory standard which made by stalagmite 0.29 ± 0.08 ‰ (2sd, n = 32). In addition, several random selected samples were analysed in the Oxford University for comparison. Our measurement results agreed well with the data obtained in Oxford University. These results demonstrate that our proposed method could be used for high precision Ca isotope analysis for carbonate samples without the need for column chemical separation procedures.

Rapid U-Pb geochronology screening of zircons by sector field ICP-MS using Dual Concentric Injector laser ablation technology

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The measurement of U-Pb ages in zircons by laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS) has become a widely used analytical tool in geoscience. Precision and accuracy of the reported LA-ICP-MS U-Pb ages is dependent on many factors, with sensitivity one of the most important. The high sensitivity of the Thermo Scientific™ Element XR™ sector field ICP-MS with the Jet Interface has been used to determine U-Pb ages for some of the youngest zircons [1]. Increased sensitivity also allows lower sample volumes to be ablated, vital for resolving complex zonation in zircons.

The development of rapid response, high efficiency laser ablation cells, such as the ESI Lasers™ Dual Concentric Injector (DCI)™ system [2], has resulted in increased sensitivity and an order of magnitude decrease in washout time available from commercial systems, spurred by applications in imaging of biological tissues [3].

Rapid response laser ablation cells have been demonstrated to improve analysis of geological material on a quadrupole-based ICP-MS [4]. From this we extrapolated combining the Dual Concentric Injector (DCI) system to the high sensitivity Element XR ICP-MS we can maximise sensitivity for LA-ICP-MS U-Pb of zircons. This combination allowed for high resolution chemical and chronological mapping of discrete zones within altered zircons. Here we report U-Pb results with this system for a variety of reference sample zircons and other matrices.

[1] Guillong (2014) *J. Anal. At. Spectrom.,* **29**, *963-970.* [2] Douglas (2015) Anal. Chem., **87**, *11285–11294.* [3] Breglio (2017) *Nature Communications, **8**, 1654-1662.* [4] Petrus (2017) *Chem. Geo., **463**, 76-93.*

The system of simultaneous double laser ablation for *in-situ* micro-analysis

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In-situ isotopic analysis is one of the huge advances in modern isotopic geochemistry. This method can reveal the micro-scale geological information that is commonly concealed by traditional bulk analysis, and avoid the complex mineral separation and chemical processes [1] [2] [3] [4]. However, the present laser ablation systems use single ablation mode and the internal standard cannot be ablated at the same time as the unknown sample. Therefore, the sensitivity variation of mass spectrometry and elemental fractionation cannot be effectively corrected, which induces the inaccuracy of the analytical results.

To resolve this issue, we are developing a new laser ablation system. In the system, a single laser beam is divided into two parts. The internal standard and unknown sample can be ablated at the same time, respectively, which ensures the fractionation of elements in the unknown sample and standard being same during their laser ablation. Adopting the new system, the internal standard can be mixed into the sample by the laser ablation for the analysis of trace elements and correction of elemental fractionation. The new technology also can improve the accuracy of *in-situ* trace elemental and isotopic analysis. Importantly, it can be applied to analyse the single or the stable isotope-absent isotopic systems, provide effective method to correct the elemental fractionation by internal standard, and improve the applicability of *in-situ* isotopic analysis.

[1] Cioni R et al.(1995) *J.P, 36, 739-76*. [2] Geist et al. (1988) *CMP, 99, 105-12*. [3] Sisson et al. (1993) *CMP, 113, 143-66*. [4] Davidson et al. (1997) *Science. 275 (5301) 826-829.*

UV-femtosecond laser ablation, fluorination and GC-IRMS analysis of four sulfur isotopes

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Modern laser techniques make it possible to analyse sulfur isotope ratio with good spatial resolution and allow to achieve high precision analysis of four stable isotopes of sulfur at ~ 100 nmol level [1]. The advantages of laser ablation method include direct estimation of sulfur isotope ratio in minerals, no chemical procedures, reduced risk of sample contamination. The spatial resolution of sulfur isotope ratios in sulfide provides a powerful insight into geological processes that cannot be obtained using traditional bulk analytical techniques. High precision $\delta^{34}S$ $\delta^{33}S$ and $\delta^{36}S$ data are required in studies of early life, to constrain the formation of ore deposits and atmospheric processes [2].

We present a new fs-laser ablation technique that accepted advantage of continuous He flow gas chromatography – isotope-ratio-mass spectrometry (LA-GC-IRMS) and to achieve precise analysis of stable sulfur isotopes (^{32}S, ^{33}S, ^{34}S and ^{36}S) at 2 - 3×10^{-9} mol level of SF$_6$. Our system consisted of laser ablation system (NWR Femtosecond UC, 256 nm and a pulse width of 270 fs) and includes ablation chamber, reactor, cryogenic traps, gas chromatograph, modified open split and mass spectrometer MAT-253. From the ablation chamber ablated nanoparticles are injected into the reactor in helium flow. SF$_6$ is produced inside nickel tube reactor at a temperature 350 ^{0}C with vapour of BrF$_5$. Exit of reactor is connected to cryogenic traps for SF$_6$ treatment. Final purification of the sulfur hexafluoride is carried out on chromatographic capillary column. Pure SF$_6$ are condensed in the loop at temperature of liquid nitrogen. After condensation of SF$_6$ on the loop, the open split is closed and the helium is pumped out to high vacuum. Loop are heated, and the released sulfur hexafluoride are injected into the ion source of the mass spectrometer MAT-253. Mass spectrometer is equipped with a multicollector system for simultaneous measurement of the ion beams at masses 127, 128, 129 and 131. The results obtained for reference materials Ag$_2$S (IAEA-S1, S2, S3), elemental sulfur (IAEA-S4), sphalerite (NBS-123), and natural sulfides (pyrite, chalcopyrite, sphalerite and galena) show excellent agreement with the accepted values. The isotope composition results for the standards and natural sulfides demonstrate the accuracy and reproducibility of our method $\delta^{34}S$ ± 0.15‰, $\Delta^{33}S$ ± 0.03‰ (2σ). and $\Delta^{36}S$ ± 0.5‰ (2σ) with spatial resolution of the laser ablation *in-situ* analysis up to 40 microns and considerably reduces the analysis time (~ 15 minutes).

[1] Ono et al. (2006) *Chemical Geology, 225, 30-39*. [2] Thiemens M. H. (2006) *Annu. Rev. Earth Planet. Sci, 34, 217-262.*

SHRIMP analysis of water abundances and oxygen isotopes

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The high abundance of water at Earth's surface makes it difficult to measure water concentrations in nominally anhydrous materials [1]. This includes all aspects of the analysis from sample preparation, cleaning, applying the conductive coating, and insertion to the source chamber.

Samples must be prepared free of epoxy. We have used metal mounts composed of In, where samples are prepared separately and pressed into the metal, and BiSn mounts, where samples can be cast and polished. The polished samples are coated with a conductive material. We have found that the material chosen has a significant impact on the H_2O background, with C and Ni being the worst, and Au, Cu, and Al being the best.

Nature hates a vacuum pump. In pumping down from atmospheric pressures, the main residual species in the vacuum is water, and it remains the principal gas to very low vacuum levels ($<10^{-8}$ mbar). SHRIMP SI was initially configured with an ion pump and Ti sublimation pump specifically to achieve low vacuum levels. However, the daily transfer of epoxy-based mounts into the vacuum system is not optimal for this type of pumping system. We have now replaced the ion pump system with a cryogenic pump that is better suited to pumping water.

Samples are placed in mount holders and are typically held in a vacuum oven for several days prior to placement in the SHRIMP vacuum lock.

SHRIMP is a large mass analyser mass spectrometer and so getting down to the background levels of smaller ion microscopes is proving difficult. This is further exacerbated by the multipurpose usage of SHRIMP for analysis of a wide variety of materials, typically mounted in epoxy, and inserted after variable curing times. Nevertheless, we have made significant progress towards an analysis protocol that does include a variety of other sample types. The water analysis sessions are well suited to being scheduled along with triple oxygen isotope analysis of planetary materials.

Measurement of samples with greater than 100 ppm H_2O is analytically straightforward, but issues remain regarding outgassing of porous rock fragments. Calibration at low levels is also somewhat problematic. We have used San Carlos olivine for both oxygen isotope and water background measurements but we have found SCO to be is heterogeneous on a grain by grain basis and we have found areas with up to 80 ppm H_2O.

[1] Turner et al. (2015) *Journal of Analytical Atomic Spectrometry* **30**, 8, 1706-1722.

High-resolution fs-LA-ICP-MS single shot measurements of Mg/Ca in foraminifers

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Foraminifers are microorganisms with a size of typically $20 - 500$ µm composed of chambers with a shell-wall about 5 to 50 µm thick. The chemical composition of their calcareous shells is sensitive to the marine environment and climate. In particular, the Mg/Ca signal is used as a proxy for ambient seawater temperature at the time of shell formation.

For precise and accurate microanalysis, we developed a new fs - LA-ICP-MS technique using a NWR-FEMTO-200 nm laser ablation system combined with a sector-field Element2 ICP mass spectrometer. To get highly resolved depths profiles of the shell, we performed the measurements with a low fluence of about $0.3 - 0.5$ J cm^{-2}. To measure Mg and Ca simultaneously, we used the Escan mode by choosing double charged $^{44}Ca^{++}$ and single charged $^{25}Mg^+$ ions for analysis. In addition, we performed single-shot measurements using a pulse repetition rate of 1 Hz, integrated the whole background-corrected pulses, and calibrated them with the microanalytical reference material MACS3. To evaluate accuracy, we also analysed international reference materials. Our data agree within uncertainty limits with certified and compiled reference values.

The technique was applied for specimens of planktic and benthic foraminifers from different locations. Published calibration curves were used to determine seawater temperatures for the different chambers.

MUSTAFAA-3 and -4: Two new reference glasses for *in-situ* analysis of phosphates

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The determination of trace element mass fractions (e.g. REE, Zn, Pb, U, Th) and isotope ratios (e.g. Sr, U, Th, Pb) in various phosphates, including minerals and biomaterials like teeth, and bones, has gained increasing importance over the last years. LA-ICP-MS is a powerful technique for such analyses, but it also requires adequate reference materials to obtain accurate results.

To facilitate phosphate analysis with LA-ICP-MS we synthesised and characterised two different Ca-phosphate glasses: MUSTAFAA-3 (**MU**enster **STA**ndard **F**or **A**patite **A**nalysis) a phosphate glass doped with Sr only (ca. 1500 µg/g), for *in-situ* Sr-Isotope analyses. MUSTAFAA-4 doped with ca. 1500 µg/g Sr, plus B, Cu, Zn, Ga, Y, Zr, Nb, Mo, Cs, Ba, La, Ce, Pr, Nd, Sm, Eu, Gd, Dy, Lu, Hf, Ta, Pb, U and Th (ca. 50µg/g) and designated as a reference material for elemental and isotopic analysis.

The concentrations of Sr, Zr, Ba, Nd, Sm, Gd, Dy, Hf, Pb, and U in MUSTAFAA-4 were determined with ICP-MS using an ID (isotope dilution) method. Cesium, La, Ce, Pr, Eu, Lu, Ta and Th were also measured by ICP-MS using standard solutions. Representative sections of both phosphate-glasses were analysed along profiles with LA-ICP-MS in two different laboratories. In addition to the elemental concentrations, Sr-, U- and Th- isotopes were characterised with MC-ICP-MS.

The results indicate that both phosphate glasses are homogeneous within the limits of the analytical methods for both elemental compositions and isotope ratios. The MUSTAFAA-3 and -4 glasses are therefore suitable reference materials for the determination of trace element abundances and isotope ratios in Ca-phosphates.

Isotopic heterogeneity of the pre-Ordovician Earth's crust of the western part of the Central Asian Fold Belt (CAFB) based on LA-ICP-MS study of detrital zircons

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U-Pb dating and Hf-isotope data are reported for detrital zircons (dZr) from a number of samples from the Ordovician rocks of Kazakhstan and Southern Urals (SU) representing all tectonic zones of the western part of CAFB. The main aim was to determine the nature and primary tectonic setting for some of the blocks located within the present-day structure of the SU, for which the (para)autochthonous relation to the Baltica or the Kazakhstan Epi-Caledonian composite continent is not obvious.

A typical provenance signal of the Kazakhstan continent was characterised by samples from the Kokchetau massif. Among dZr from the Kokchetau massif, the juvenile zircons (εHf>0) with Meso-Proterozoic (PR) ages are prevailed, suggesting that they are most probably sourced from oceanic arcs. The characteristic features of dZr sourced from the crystalline basement of Baltica were obtained using samples available from different levels of stratotypical sequences of Riphean Gr. (Baskir Uplift). They showed that during the Meso-PR time (until about 1 Ga), the only erosional products accumulated at the Uralian margin of Baltica were derived from the Archean domains and from Paleo-PR orogens that sutured the Archean domains into the supercontinent Columbia. No younger zircons have been identified there. The Baltica basement provenance signal is also characterised by occurrences of dZr with crustal Hf model ages T_{DM}^{c} > 3.5 Ga.

Obtained age spectra of dZr from all of the Uralian zones demonstrated no similarities neither to the Kazakhstan nor to the Baltica blocks. In the Eastern-Uralian zones and Sakmara allochtone, a strong predominance of the Late Neo-PR to Cambrian zircons implies that they could not been sourced from Baltica or Central Kazakhstan. It is known from the previous studies that the same dZr are typical for the Cadomian/Avalonian domains of Anatolia, Central and Western Europe. Thus, our new results are the first isotopic evidence for existing of Epi-Cadomian-age terranes in the Cis-Caspian basin and SU.

Determination of Zr and Nb concentrations in chromium matrices using Agilent 8900 ICP-QQQ and Elan ICP-DRC-MS

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The short-lived ^{92}Nb-^{92}Zr decay system is a novel tool to date early planetary evolution. To obtain a precise age from coeval mineral phases of meteorites using the isochron approach, it is crucial to determine precise Nb and Zr concentrations in high chromium matrices (e.g. chromite in eucrites). In ICP-MS mass spectrometry, Cr argides (CrAr$^+$) are formed from a high Cr sample matrix introduced in the Ar plasma. These argides have *m/z ratios* similar to those of our elements of interest (^{90}Zr+/^{40}Ar^{50}Cr$^+$, ^{92}Zr+/^{40}Ar^{52}Cr$^+$, and ^{93}Nb+/^{40}Ar^{53}Cr$^+$). A mass resolution of 24,000 and 27,800 are required to resolve ^{90}Zr$^+$ from ^{40}Ar^{50}Cr$^+$ and ^{93}Nb$^+$ from ^{40}Ar^{53}Cr$^+$, respectively. To eliminate these interferences, an ICP-MS with a dynamic reaction cell (DRC) was used to counteract the formation of Cr^{x}Ar$^+$ with both H$_2$ and NH$_3$ as a reaction gases to neutralise the Ar background species [1]. However, clustering reactions with transition metal ions when introducing NH$_3$ as reaction gas limited the general use of NH$_3$ in ICP-DRC-MS. Moreover, H$_2$ was tested as the reaction gas and this led to strong suppression of Nb and Zr signals in high Cr solution [2]. Therefore, Rh was introduced as internal standards to correct the drift and signal suppression due to Cr matrix effect [2]. We tested this setup on a Elan ICP-DRC-MS and obtained consistent results to [2].

Through new instrumental developments, it has recently become possible to eliminate the formation of polyatomic ions using ICP-tandem mass spectrometers (Agilent 8900 QQQ). These instruments feature an additional quadrupole located before the collision-reaction cell. To test the efficiency of this set-up, solutions with known Nb and Zr concentrations in Cr matrices up to 500 μg/g were measured using varieties of collision and reaction gases (H$_2$, He, O$_2$ and NH$_3$). When using O$_2$ and NH$_3$, ^{90}Zr and ^{93}Nb concentrations were analysed at M+16 and M+ 82,83, respectively. We obtained consistent Nb and Zr concentrations in high Cr solutions when using (i) ICP-DRC-MS with H$_2$ as a reaction gas and (ii) ICP-MS/MS with H$_2$, He, O$_2$ and NH$_3$. The measured basaltic reference material, BHVO-2, is in good agreement with previously published data for all measurement conditions. The benefits of ICP-MS/MS compared to ICP-DRC-MS identified in our preliminary study are that 1) very limited signal suppression of Zr, Nb, and Rh in Cr-matrix solution; 2) best intensity when using the Agilent 8900 QQQ for Nb and Zr with O$_2$ and NH$_3$ as reaction gases, which indicates that higher precision data for Nb and Zr concentrations can be acquired using this new method. This is important for improving the precision of Nb-Zr isochron ages in cosmochemical applications.

[1] Hattendorf & Günther (2000) *Anal. At. Spectrom.* **15**, 1125-1131. [2] Hattendorf et al. (2001) *Anal. Chem.* **73**, 5494-5498.

In-situ Sr isotopic measurement of scheelite using fs-LA-MC-ICP-MS

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Scheelite is one of the main ore minerals in tungsten deposits, and Sr isotopic compositions of scheelite can be used to examine the petrogenesis of igneous rocks and fluid metasomatism in mineralisation processes. Here we describe a femtosecond laser ablation multi-collector inductively coupled plasma mass spectrometer (fs-LA-MC-ICP-MS) technique which can obtain *in-situ*, accurate, high precision ^{87}Sr/^{86}Sr ratios from 20-50 μm scheelite in line mode without requiring time-consuming mineral separation and pre-chemical procedures. We found that Ca dimers and Ca argides do not detectably affect the obtained ^{87}Sr/^{86}Sr ratios, and the adopted protocol overcomes interferences from Kr$^+$, Rb$^+$, Er^{2+} and Yb^{2+}. We show that the values of ^{87}Sr/^{86}Sr are relatively homogeneous for two scheelites from quartz veins, and are comparable to the values determined by a traditional solution method. Hence, these two scheelite samples have the potential to be reference materials for Sr isotopic determination by LA-MC-ICP-MS. To illustrate the utility of the technique, Sr isotopes of scheelites from three different types of tungsten deposits of South China are documented. The ^{87}Sr/^{86}Sr of scheelite from granite-related veinlet-disseminated and porphyry tungsten deposits varies systematically, showing a positive correlation between Sr content and ^{87}Sr/^{86}Sr ratios. These micrometer scale inhomogeneities could be explained by mixing of two components, reflecting intense fluid metasomatism during mineralisation processes. High ^{87}Sr/^{86}Sr ratios were obtained for the scheelite samples from a quartz vein type tungsten deposit, indicating that the late stage ore-forming fluid was mainly derived from the surrounding strata. These examples show that *in-situ* Sr isotopic measurement of scheelite is a powerful tool to decipher the degree of fluid-rock interaction in ore-forming processes.
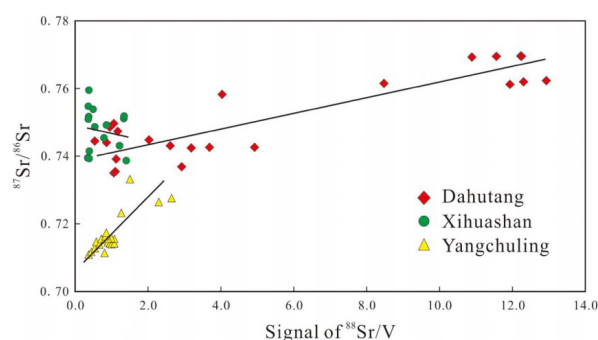

Figure 1. Correlations between the ^{87}Sr/^{86}Sr ratios of scheelites and ^{88}Sr signal intensities from three types of tungsten deposits from South China.

Simultaneous sensitive determination of selenium, silver, antimony, bismuth and lead in geological materials by liquid spray dielectric barrier discharge induced plasma-chemical vapour generation

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Recently, there has been a surge in the development of analytical methods to analyse ultratrace elements in very small amounts of samples [1]. Therefore, it is attractive to develop excellent sampling techniques for atomic spectrometry to analyse small amounts of samples with complex matrixes associated with biological, geological and toxico-logical study [2,3].

In the present study, five elements have been examined including Se, Ag, Sb, Bi and Pb by coupling the liquid spray dielectric barrier discharge induced plasma-chemical vapour generation technique (LSDBD-CVG) with the inductively coupled plasma mass spectrometry (ICP-MS). In this LSDBD approach (Fig. 1), the sample solution is nebulised into fine droplets and simultaneously reacted with the DBD plasma. The effects of the doping of organic substances, different discharge parameters such as discharge voltage and discharge gap, as well as the foreign ion interferences were investigated. Compared with conventional pneumatic nebulisation ICP-MS (PN-ICP-MS), the sensitivity of Se, Ag, Sb and Bi, increased by 27, 21, 16 and 27 times respectively when using LSDBD–CVG-ICP-MS (Fig. 1). These results demonstrate that, our proposed method is simple, sensitive, environmentally friendly, and cost-effective, being promising for determination of ultratrace elements in limited samples.
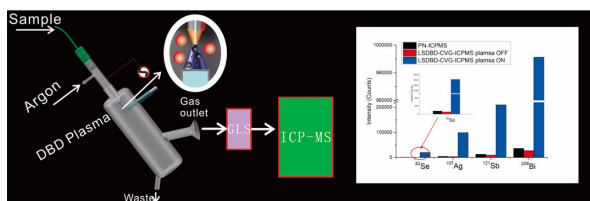

Figure 1. Schematics diagram of the LSDBD–CVG –ICP-MS, and comparison of the signals for three sample introduction modes.

[1] Zenobi, R. (2013) *Science, 342, 1243259.* [2] Li, Z., Tan, Q., Hou, X. et al. (2014) *Analytical Chemistry, 86, 12093-2099.* [3] Liu, Xing & Zhu, Zhenli & Li, Huilai et al. (2017) *Analytical Chemistry, 89, 6827-6833.*

High precision, high accuracy silicon isotope determination in quartz using SIMS, by reducing the topography effect

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Silicon isotope determination using SIMS was usually focused on low temperature process, mostly due to its relatively low precision, ±0.3 ‰ (2 SD) for instance. [1] With the aim of improving analytical precision and accuracy of SIMS (Cameca IMS-1280) Si isotopic analysis, attempts were made in this study, including increasing primary beam intensity and extra data processing. A primary beam of 10-14 nA with diameter of ~20 μm was used, a 25 μm raster was also employed to avoid deep crater.

Tang et al. [2] has reported the correlationship between $\delta^{18}O$ and DTCA-X (a secondary ion centring parameter, which was adjusted automatically before each acquisition) during SIMS oxygen isotope determination. The correlated trend is believed to be due to the topography effect on the sample surface. A similar linear relationship between $\delta^{30}Si$ and DTCA-X was also found in this study, as shown in Figure 1, which provided a potential data correction method for Si isotope and even all the stable isotope analysis using SIMS. By applying the DTCA-X topography correction, the external error was reduced from ~ ± 0.3 ‰ to < ± 0.15 ‰ (2SD) for NBS-28. Several unknown samples were determined using this method and also checked by the MC-ICP-MS method, the results from both method were identical within an error of ± 0.1 ‰.

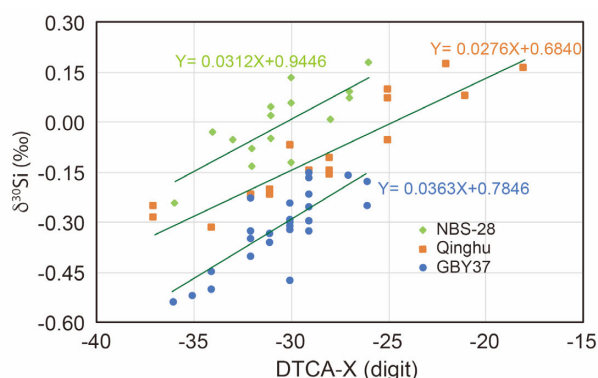*Figure 1. The relationship between DTCA-X and $\delta^{30}Si$ for different quartz samples in the same mount, all the fitted line have similar slope.*

[1] Heck, P.R. et al. (2011) *Geochimica et Cosmochimica Acta, 75, 5879-5891.* [2] Tang, G.-Q. et al. (2015) *Journal of Analytical Atomic Spectrometry, 30, 950-956.*

Certified reference material for determination of total cyanide in soil

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Due to their toxicity and widespread application for mining and industrial purposes, cyanides are ranking among the most important inorganic pollutants which should be tested and monitored not only in the aquatic environment, but also in soils and soil-like materials. Reference materials of soils with relevant contents of cyanide to ensure reliable test results of laboratories are rare today.

New certified reference material (CRM) BAM-U116/ CGL306 "*Cyanide in soil*" was produced within a framework of cooperation between Central Geological Laboratory (CGL) of Mongolia and Federal Institute for Materials Research and Testing (BAM) of Germany in 2013-2017.

The CRM BAM-U116/CGL306 represents a mixture of a sandy soil collected from a contaminated former gasworks area in the Berlin region (Germany) and an unpolluted sandy soil from Nalaikh region (Mongolia). The bulk candidate material for this reference material was prepared at CGL CRM Laboratory exclusively destined to the preparation of reference materials and equipped with modern technical equipment. Homogeneity, stability and shelf life were studied in full compliance with ISO Guide 35. The CRM was evaluated as sufficiently homogeneous. Statistical evaluation of certification analysis was performed using software packages *SoftCRM* and *ProLab* Plus. Certified value of total cyanide of the CRM is 12.0 mg/kg and expanded uncertainty was assigned as 0.8 mg/kg.

The intended purpose of this material is the verification of analytical results obtained for the mass fraction of total cyanide in soils and soil-like materials applying the standardised procedure ISO 11262:2011. As any reference material, it can also be used for routine performance checks (quality control charts) or validation studies.

Certification of coal fly ash reference material

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The rapid development of the world economy depends directly on the availability of energy. Today, a certain part of the world's total energy needs are generated from fossil energy resources like coal, oil and gas. Coal is the cheapest among them and has greater known resources than petroleum and natural gas. The low price and the overall availability recently lead to increasing rates of coal combustion year by year. Modern power plants usually filter fly ash from coal combustion gases in order to protect the environment. Therefore coal fly ash is a typical waste from power plants which either has to be dumped safely or recycled for special purposes. Different studies on recycling and re-using of coal fly ash are being conducted in many countries due to the development of science and technology. For experimental studies, the exact composition of the ash is of crucial importance. Certified reference material (CRM) of this type of material is therefore in high demand.

This CRM had been produced according to the ISO Guides 30-35.

The material was prepared (cleaning, sieving, splitting, bottling and labelling) at specialised CRM laboratory in Central Geological Laboratory of Mongolia. To evaluate data of certification analysis, "ProLab Plus" software for statistical evaluation was used. An inter-laboratory approach with 21 qualified participating laboratories was selected to obtain a reliable base of data for assignment of the certified values. A nested design was chosen for maximum information output. The Certified values and uncertainties of the CRM had been determined after exclusion of outliers according to Nested design, ISO 5725-3, ISO Guide 35.

Quantitative mineral mapping of complex fine grained materials

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Spatially resolved mineral identification and quantification are essential to many fields of scientific enquiry. Studies of coarse-grained materials routinely determine mineral distribution using simple optical petrography, mapping XRF or SEM-based techniques such as QEMSCAN. However, spatially resolved mineral identification has long proven a challenge in fine-grained materials, where individual mineral grains are commonly smaller than the resolving power or analytical volumes of the techniques used.

Here we present several case studies demonstrating the capabilities of a new SEM-based mineral mapping system now available at Macquarie University and specifically developed for rapid, automated analysis of fine-grained, complex mineral mixtures. The FEI Teneo field emission SEM platform incorporates dual high-throughput Bruker Series 6 EDS detectors, with automated data collection via FEI Maps Mineralogy. In combination with the new Nanomin data processing software, this system is able to resolve complex mineral mixtures that occur in the electron-beam excitation volume of a typical fine-grained material. Unlike previous SEM-based mineral mapping systems such as QEMSCAN, Nanomin can deconvolve mixed X-ray spectra into up to three component minerals. This opens up analysis of complex fine-grained materials in which quantitative mineral mapping has not previously been possible, resulting in greater accuracy in mineral identification and, especially, quantification.

We show several case studies where this new technology has permitted us to tackle previously intractable problems, including: 1) the role of clay minerals in sequestration of organic matter in black shales 2) the palaeoenvironmental significance of trace mineral phases 3) disentangling depositional and diagenetic mineral phases for paleoenvironmental analysis, 4) petrographic characterisation of glauconite and detrital contaminant phases prior to in-situ Rb/Sr dating, 5) verification of element host phases following sequential acid extraction.

The influence on PA factors by P/A correction under different signal response values of Agilent 7500Ce

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In the instrument debugging process of Agilent 7500Ce mass spectrometer, one of the steps is to do P/A factor tuning of the detector so that the signal measured by detector in different modes (Pulse mode and Analog mode) can be cross corrected and ensure the instrument can obtain good linearity in a wide range of concentrations. This step requires that the signal response value of the isotopes are among 40,000 to 400,000 cps (count per second) so that the tuning of the P/A factors can be carried out smoothly. We tuned P/A factors under different isotope signal response values. It was found that the P/A factors obtained by P/A factor tuning will vary with the change of the signal response value. With the increase of the signal response value of the isotopes, the P/A factors decrease. When the signal response value of isotope is close to 40,000 cps, the P/A factors are the largest, and they are significantly higher than the P/A factors calculated by higher signal response value (The deviation of maximum value versus minimum up to 7.5%). When the signal response value of the isotopes 100,000 - 400,000 cps, the P/A factors decreases slowly (The deviation are 1.2-2.2%). Unsuitable signal response values used for P/A factor correction will produce significant system deviation for the test. We suggest that when doing P/A factor tuning, control the signal response values of the isotopes among 100,000 to 400,000 cps, which the corresponding P/A factors change little.

U-series analyses at Macquarie University: Updates, observations and advances in U-Th-Ra-Pb techniques

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Radionuclides of the Uranium series decay chain are routinely analysed at Macquarie University. Previously we have focussed on volcanic and sedimentary samples for U-Th, but have recently analysed a suite of low concentration, chondritic samples. We continue to analyse volcanic samples for Ra ratios, and have recently re-established ^{210}Pb work for recent eruptive volcanic samples. The 22-year half-life of ^{210}Pb makes it of interest for future environmental (for contaminant research) and magmatic related problems (for volcanic degassing studies).

We present a compilation of solution and rock standards for U and Th isotopes and rock standards for Ra and Pb isotopes. We show that whilst the activity ratios of TML (table mountain latite – one of the most widely used U-series standards) are highly reproducible, both U and Th isotope dilution concentrations are far more variable suggesting heterogeneity. BCR-2 is presented as a far more reliable rock standard. Repeat digestions and analyses of the chondrite Allende (U = 0.016 ppm) are shown to be reproducible although interestingly the sample is heterogeneous in its (^{234}U/^{238}U) ratio.

^{210}Po is measured as a proxy for ^{210}Pb when samples are > 2 years old and ^{210}Po (half-life = 138 days) is in equilibrium with ^{210}Pb. We provide details of the chemical extraction and analysis by alpha spectroscopy and highlight our issues with spike instability.

Characterisation of three ultramafic PGE reference materials

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For the purposes of method validation, calibration, and verification of Os isotope ratios and platinum group element (PGE), Re, and Au contents, three ultramafic reference materials (RMs) with different matrices have been prepared and characterised through the International Association of Geoanalysts' (IAG) certification and GeoPT proficiency-testing programs. OKUM, an ultramafic komatiite (Ontario, Canada), and MUH-1, a depleted harzburgite (Kraubath, Austria), were prepared and certified for major and trace elements through an interlaboratory certification process led by the IAG. HARZ01, a highly depleted and unserpentinised harzburgite (Devolli, Albania), was characterised for major and trace elements through GeoPT round 38A. The three RMs (OKUM, MUH-1, and HARZ01) have since been included in an interlaboratory comparison (ILC) in which varying sample masses (0.5 – 15 g), diverse digestion (fire assay, acid digestions) and analyte matrix separation techniques were applied by up to 12 laboratories. In order to minimise the influence of outliers, the modes of the median/modes of the laboratory data were used as the best estimates for the consensus values. The experience with GeoPT data evaluation was the basis for this approach [1]. A major conclusion of this study is that, in most cases, the most commonly used test portion of 2g is insufficient to achieve reproducible results for the precious metals.

Through this ILC, it was possible to derive consensus values for all three RM, with fit for purpose uncertainties even in the sub ng/g range. The combination of well-defined consensus values (and associated uncertainties) for the precious metals and certified or GeoPT consensus values for the major and trace compounds/elements makes these three materials the best characterised ultramafic RMs currently available. All three can be purchased through IAGeo limited.

[1] Thompson (2017) *Geostand. Geoanal. Res.,* **42**, *91-96.*

Achieving clumped isotope efficiency to characterise palaeoenvironmental variation: the next generation of equipment and automation

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Carbonate clumped isotope paleothermometry focuses on the measurement of mass-47 of CO_2 in a sample relative to the stochastic distribution of all atoms in the sample (Δ_{47}) which varies based on the temperature of equilibration of the carbonate. This measurement has made waves in the geochemical community as it offers the opportunity to make a single measurement on a carbonate derived CO_2 gas to retrieve $\delta^{13}C$, $\delta^{18}O$, and the temperature of formation of the carbonate which can then be used to calculate the composition of the precipitating fluid. The techniques for making the measurement however have been dogged by difficult sample preparation, long run times, large sample sizes, and no shared carbonate standards. Measuring a single unknown can take up to a day. This has proved impractical for palaeoenvironmental studies and time series in which accurately characterizing spatial and temporal variability can require tens to hundreds of samples .

The Macquarie University Stable Isotope Lab (MUSIL) is addressing this problem by developing efficient techniques to measure clumped isotopes combining increased mass spectrometer sensitivity with automated sample preparation. MUSIL will be the first laboratory to combine the IBEX© automated clumped isotope preparation system with the analytical power of the Thermo 253-plus equipped with ultra-sensitive amplifiers utilizing $10^{13}\Omega$ resistors. By utilizing micro-volumes down stream from the IBEX and bypassing the bellows, samples can be measured in significantly reduced times with sample sizes minimised to the µg range, yet still produce the same statistical accuracy. This combination will increase the number of samples run per day to 10-15 while greatly reducing operator time and increasing consistency. With these advances, clumped isotope paleothermometry becomes more accessible to palaeoclimatologists focused on the implications rather than the acquisition of data.

This automated approach will also benefit from recent approaches in the clumped isotope community to identify a series of 6 different carbonate standards of known Δ_{47} and standardise these across all labs. MUSIL is a keen participant in this effort and will use this new series of standards to develop and test the methods we are developing.

New software for data reduction of LA-ICP-MS data sets

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The formula for computing concentrations from LA-ICP-MS data sets was first published by Longerich et al. in 1996[1]. Demand for computer software soon followed, and twelve years later the 40th MAC short course [2] identified nine software packages that were available for data reduction of LA-ICP-MS data. Many of those software packages are no longer available, yet despite this attrition no single title has become ubiquitous, which we suggest is due to the level of complexity inherent to many factors ancillary to the main calculation.

In addition to solving the basic Longerich equation, successful data reduction of LA-ICP-MS data sets requires consideration of: identification and removal of transient spikes; corrections for interferences; modelling of variations in background intensity; synchronisation with laser firing time; modelling and correction for down hole fractionation; modelling of calibration drift; assessment of secondary standards and correction for matrix effects; addition of unmeasured elements by stoichiometry; quantification to a total concentration; support for mixed quantification types within a data set, including changes to internal standard and calibration SRMs; and most importantly, correct and robust error propagation from all sources of uncertainty.

Here we present an overview of a new software package "LADR" designed to perform all of these functions. To assist the user's comprehension of the data it also displays graphics for visualisation of all time-dependent values such as backgrounds, calibration drift, and downhole fractionation curves. The software is able to load data files from most commercially available ICP-MS instruments and it can compute results for trace element analysis, ages for UPb dating, and quantify arbitrarily defined ratios of any two masses.

Recent workers [3] have hi-lighted the need for robust error propagation, which we can be met by the "bottom up" approach of the EURACHEM/CITAC-Guide [4] as applied to LA-ICP-MS [5][6]. Our LADR software accounts for all sources of uncertainty, which are combined and reported to the user in an expandable view. The software makes it possible to conveniently report uncertainty values at a level appropriate for the requirements, such as intra-run, intra-laboratory, or inter-laboratory comparison.

The software package LADR is available for download from the authors' website [7].

[1] Longerich et al. (1996) *JAAS, **11**, 9, 899-904.* [2] Longerich & Sylvester (2008) *MAC **40**.* [3] Horstwood et al. (2016) *Geostand. Geoanalytical Res. **40**, 3, 311-332.* [4] Ellison & Williams (2012) *Eurachem/CITAC guide, ISBN 978-0-948926-30-3.* [5] Luo et al. (2007) *JAAS, **22**, 2, 122-130.* [6] Gilbert et al. (2013) *Geostand. Geoanalytical Res,. **37**, 1, 51-64.* [7] *http://norris.org.au/ladr/ "LADR" (Mar 2018).*

New frontiers in atom probe tomography: Probing the nanoscale inhomogeneities in nacre

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Atom probe tomography (APT) is a powerful technique to probe 3D (geo)chemical information at nm length scales. Originally, used to study conductive materials (e.g. metal alloys) more recent applications include semiconductors, geological materials, and very recently also biominerals such as bone, teeth, and foraminifera [1-4].

Biomineralisation benefits largely from APT since it is the only technique available to probe its complex hierarchical arrangement of organic-inorganic interfaces down to the atomic-scale.

In this study we use APT to characterise the nano-scale inhomogeneities within the bio-composite material nacre, which consists of aragonite pseudo-hexagonal lamellae that are enveloped by organic membranes [5]. High resolution TEM investigations revealed 2-25 nm sized inclusions to occur within the aragonite lamellae that have been speculated to be organic phases [6-8] with the potential to bear information on nacre-formation processes.

We used nacre from the maximum growth axis of the black mussel (*Mytilus galloprovincialis*, Lamarck, 1819) that has been sharpened to a fine tip by means of focused ion beam milling. Laser assisted APT was carried out using a CAMECA LEAP 4000X Si at the Australian Centre for Microscopy and Microanalysis following the protocol of Branson et al. [4] This presentation will include spatial and compositional information of the organic inclusions as well as trace elements.

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Chlorine analysis with XRF to determine salinity of pore water in the Baltic Sea sediments

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Chlorine X-Ray fluorescence analysis of dry sediment can be used to determine salinity of pore water in clay sediments, if the water content of the original sample has been measured. Cl is one of the elements that is rarely analysed within bulk chemical composition, but can give an estimation of pore water salinity in sediments. In this study, samples that were analysed to test the method were gathered from the Gulf of Finland during the SedGoF project. Sediment profiles from western, middle and eastern regions of the gulf were analysed. Samples were taken from the sediment core with a step of 2 cm. The length of the cores varied between 34 and 54 cm. For sample preparation, pressed pellets were made after freeze-drying and grinding. Pellets were analysed with Bruker S4 Pioneer spectrometer and using precalibrated MultiRes method.

The calculated salinity values of pore water correlate well with salinity measurement results above sediments. Salinity of bottom water was measured during the same period the drill cores were collected. Salinity data gathered during a separate monitoring expedition were provided by TTU Department of Marine Systems.

Although Cl can be analysed well with the XRF method, the sample preparation needs special attention. Samples need to be analysed directly after drying, otherwise Cl could migrate to pellet surface because of moisture. Water-based binding materials cannot be used for the same reason. In this study two different pellet preparation methods were used. Pellets from sediment surface layers were pressed without the binding material, other samples were pressed after adding 20 % boric acid.

In this case, well-corresponding results with bottom water salinity measurements were achieved with the Bruker's precalibrated MultiRes method, but for more accurate analysis reference materials are needed. There are but a few different reference materials for Cl analysis. Some of these were analysed to test the accuracy of the Cl determination, but all of them had lower values than the analysed sample. Even though we got a good match between the measured and reference values, more reference materials with higher Cl concentrations are needed. Anyway, this method works well for estimating the salinity of sediment pore water.

Quantitative approaches to determination of elements in lake sediments by total reflection X-ray fluorescence

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One of the most popular geological applications of X-ray fluorescence spectrometry (XRF) is the analysis of lake or ocean core sediments to study environmental and climate change over geological time scales. Absolute changes in the element concentration with the depth of the core can be obtained by means of analyzing dry, powdered, homogenised sediment fragments. It is important to emphasise that complex high-resolution studies require analyzing large number of discrete samples, whose quantities are often limited (< 500 mg). Conventional WDXRF technique usually requires relatively large mass of a sediment sample. From this point of view, total reflection X-ray fluorescence (TXRF) seems to be well suited for multi-elemental analysis of small quantity (10-50 mg) of subsamples obtained by cutting the sediment core at different intervals.

The aim of this work is to estimate and minimise the error in quantification of Al, K, Ca, Ti, V, Cr, Mn, Fe, Ni, Cu, Zn, Br, Rb, Sr, and Ba. Preparation of the sample as slurry from 20 mg of powdered sediment and 2 ml of disperser agent was chosen as easy and rapid sample preparation strategy. Triton 1 % solution or metilcellulose 0.5 % solution in deionised water can be used as a disperser agent. It was demonstrated that normalisation of the net intensity signal of analyte to the incoherent scattered signal (Compton peak) could reduce the uncertainties associated with the sample preparation process. Similar repeatability (generally better than 7 %) was obtained using normalisation with Ga internal standard or the Compton peak.

Different quantitative approaches have been evaluated using certified reference materials of sediments: (i) internal standardisation, (ii) external calibration with correction by adding internal standard, (iii) external calibration with 'fluorescent-to-Compton' correction. We consider that the external calibration may be useful to improve the results of determinations of most elements determined.

The TXRF results obtained by proposed quantitative approaches were compared with those obtained by conventional WDXRF spectrometer from 300 mg of pressed sample. Distribution of elements along of the lake sediment cores obtained by both methods were comparable.

Isotopic composition of lead in bivalves shells using LA-ICP-MS

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Bivalve mollusks are able to fix and accumulate chemical elements inside their shells by filtering ambient water. Therefore, they are potential indicators of environmental changes and pollution events. To date, several studies have reported lead (Pb) accumulation inside bivalves shells however there is no study addressing isotopic composition of lead. In this way, this is a pioneer study; our goals were to determine lead isotope composition in bivalves shells from Ilha Grande Bay, Brazil, using LA-ICP-MS in order to investigate lead isotopic signatures and lead accumulation along shells layers. Eight samples shells were analysed using Neptune Mass Spectrometer with Coupled Plasma and Laser Photomachine 193 nm; NIST-612 was used as standard.

Data of isotopic composition of lead in shells were correlated with signatures reported in superficial sediments from the same geographic area [1] in the ^{206}Pb/^{207}Pb vs. ^{208}Pb/^{206}Pb diagram (Fig. 1).
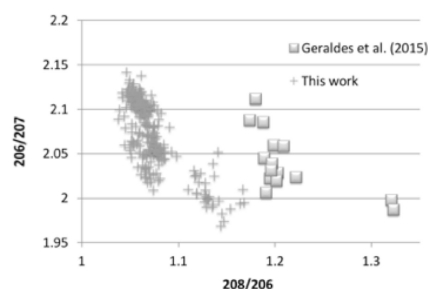

Figure 1. ^{206}Pb/^{207}Pb vs. ^{208}Pb/^{206}Pb diagram.

Results of lead accumulation showed high variability between shells (Fig. 2). These variations were associated with several factors: Biological processes, complex interplay of environmental conditions, input from many anthropogenic sources and distinct doses incorporated during the lifetime of each animal.
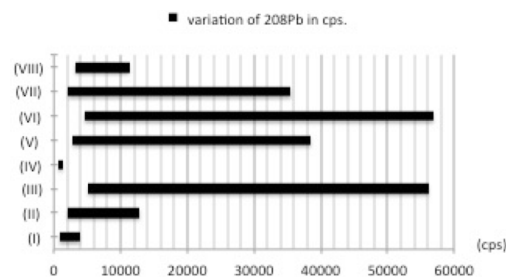

Figure 2. Variation of lead accumulation, maximum and minimum recorded signals intensities of isotope ^{208}Pb.

This study provides new evidence that is possible to conduct analyses of lead isotopic composition in bivalve shells using LA-ICP-MS to yield valuable insights about environmental changes. The efficiency of the method is able to detect small amounts of lead accumulation.

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Struggle with inhomogeneously quenched melts – an approach to standardise their major element measurement

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High pressure experiments are essential to provide better insights into the Earth's deep volatile cycle and its evolutions. However any volatile-bearing melts (containing H_2O or/and CO_2) show a strong quenching effect, usually with the presence of crystal overgrowths, films or spheres of modified glasses and even pores (Fig. 1).

During the experiment, at a given pressure and temperature, the melt exsists as one single homogeneous phase, which is quenching to a multiphase inhomogenous intersertal texture, therefore the determination of the initial bulk melt is challenging. This quenching effect could modify the major and trace element distribution in the melt pool as crystals growing from the initial melt cause strong partitioning and zoning inside the quenched texture.

In this presentation, we provide a standardised approach, using defocused microprobe and calibrated SEM, to the inhomogenously quenched melt pools and try to resolve subjective (user´s) errors during measurements.
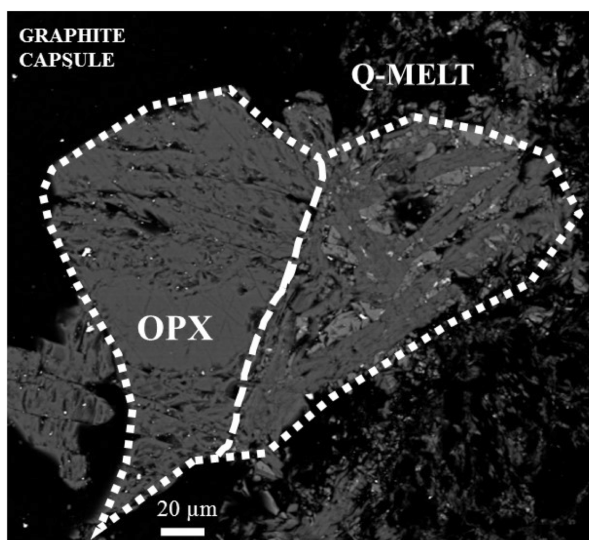

Figure 1: Back-scattered SEM image of a typical quenched hydrous, carbonate-bearing melt in intersertal texture adjacent to orthopyroxene crystal and graphite capsule. Quench phases include phlogopite, clinopyroxene, dolomitic carbonate and titanomagnetite.

The Geo*PT* Proficiency Testing Programme as a scheme for the certification of reference materials

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Traditionally, the role of proficiency testing has been limited to providing support for a laboratory's quality assurance activities, particularly in demonstrating a laboratory capability of meeting the ISO 17025 standard. Such schemes, involving the regular distribution of appropriate test materials, are designed to evaluate the routine analytical performance of laboratory procedures. Participating laboratories are provided with feedback on the accuracy of their individual measurements, which is one of the most effective ways of identifying measurement bias. In Geo*PT*, robust statistical analysis and expert judgement are used to derive consensus values from the results contributed by participants to provide best estimates of true values for the test material. These assigned values for Geo*PT* test materials have gained an exceptional reputation, exemplified by a comparison of values for 6 certified geochemical reference materials that were used as Geo*PT* test materials, where overall no significant difference was found between Geo*PT* assigned and certified values [1]. Confidence in the Geo*PT* scheme has also been engendered by the exceptional stability in results derived from the same test material that was distributed in Geo*PT* rounds 18 years apart [2].

Recently REMCO, the ISO committee that provides international guides on the development of reference materials has revised its 'guidance for assessment and characterisation of homogeneity and stability of reference materials' [3] to recognise the use of proficiency testing schemes for the certification of reference materials. Taking advantage of this opportunity, the International Association of Geoanalysts has developed a scheme based on the Geo*PT* programme for the certification of geological reference materials. This presentation will evaluate the performance of this scheme, describing the justification for having confidence in the results.

[1] Potts, Thompson and Webb (2015) *Geostandards and Geoanalytical Research, 39, 407-417.* [2] Thompson (2018) *Anal. Methods, (in press).* [3] ISO Guide 35 (2017).

Geoanalysis in industry: Rio Tinto

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Utilisation of geochemical data is an integral part of mining and the mineral exploration process. All large-scale mining and exploration operations depend on data derived from multiple techniques and materials to help locate and define resources, to help understand mineralogy and element deportment in relation to ore processing, and to understand the behaviour of the by-products of mineral processing.

The BTDC (Bundoora Technology and Development Centre) is a Rio Tinto facility located in Melbourne which houses some of the key analytical equipment routinely used to generate this data. Work undertaken on site includes development of flowsheets for processing and recovery from new ore types; optimisation of chemical and mechanical processes to increase efficiency and recovery from existing processing circuits; and remediation of sites undergoing closure. Analytical facilities related to this work are *in-house*, and include XRF, ICP-MS, ICP-OES and SEM/MLA/QEM.

Also located at BTDC is part of the exploration team, which uses geochemical data during the early stages of exploration to help define targets. The benefit of *in-situ* analysis to exploration was recognised more than a decade ago, and a laser-ablation ICP-MS laboratory was set up in house. Programs using resistate indicator minerals to explore for commodities such as copper, nickel, uranium and diamonds were introduced, and continue to be effective today.

By working with industry partners, academia and bodies such as AMIRA, advances have been made in areas such as hardware, software and applications that have provided benefits to the geoanalytical community as a whole. The adoption of new technologies and expertise is recognised as essential, to enable BTDC to adopt new geoanalytical tools for exploration as they are developed.

Tracing Hg mobility and distribution in a historical mercury mine ecosystem

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Persistent mercury (Hg) impact from legacy mines continues to present risk to the environment and human health globally. The Stibnite Mining District is a historical mining area located near the headwaters of the East Fork South Fork Salmon River in central Idaho. Past mining of antimony, gold, silver, mercury and tungsten from the early 1900s to the 1990s has resulted in elevated Hg as well as other trace elements in the surrounding ecosystem. The Cinnabar Hg mine, within the Stibnite Mining district is a primary source area for elevated Hg entering the ecosystem. The extraction of Hg from cinnabar was carried out via two methods over the 45 years of Hg production in this mining district: 1) Hg was initially retorted in rotary kilns (1921 to 1956), a process during which the ore is heated to 500-600°C volatizing the Hg to gas with recovery as elemental $Hg_{(l)}$ via condensation and 2) Hg was later produced by wet flotation and electro-separation (1956 to 1966). These distinct methods of extracting Hg from cinnabar resulted in wide variations of Hg concentration and isotopic composition being introduced into the environment during the active processing of the Hg ore, as well as long term legacy input of Hg from the weathering of calcine waste piles and other sources related to the original mining and processing of Hg ore. Mass dependent fractionation of Hg isotope ratios and variation in Hg concentrations resulting from the processing of cinnabar ore has been reported previously for produced Hg metal and residual waste fractions ($Hg_{(l)}$, $Hg_{(g)}$, and calcines). These Hg fractions possess the potential of releasing Hg into the local environment over sustained periods of time. In order to better understand mercury mobility in legacy mining areas, establish a priority of effort for remediation, and determine Hg mobility and pathway of in this ecosystem, we measured Hg concentrations and isotopic compositions in a wide range of samples: two distinct calcine produced waste piles, vegetation, lichen, periphyton, seston, stream sediment, soil, tadpoles, tadpole eggs, caddisflies larvae, mayflies larvae, spiders, tailed frog, sculpin, and bull trout. We will present Hg concentrations and isotopic compositions of these samples collected within the mining district and provide a post-mining baseline of Hg distribution. This baseline will help inform environmental assessments for future landuse activities, including recommencing of mining operations and onsite remediation in this ecosystem.

Testing the limits of *in-situ* Rb-Sr dating of igneous minerals by LA-ICP-QQQ

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The Rb-Sr dating system is a powerful tool to constrain the timing of igneous rock crystallisation. Coupling a laser ablation (LA) system with a "triple quad" inductively coupled plasma mass spectrometer (ICP-QQQ) makes the *in-situ* Rb-Sr dating of individual igneous minerals possible [1]. The precision and accuracy of this technique is sensitive to several analytical parameters, including the spot size of the laser beam, and related matrix and down hole fractionation effects. This study aims to investigate the impact of these issues on the Rb-Sr age of an igneous mica analysed by an Agilent 8900 ICP-QQQ filled with N_2O as a reaction gas. The mica (MDC), which has an expected age of 519.4 ± 6.5 Ma [1], was collected from the Ampandrandava phlogopite quarry, Madagascar, mounted vertically and ablated by two different models of laser. One is an ASI RESOlution ArF 193µm excimer system, the other an ESI NWR213 Nd-YAG system. All results were corrected to the primary Mica Mg nano-powder pellet standard [1]. The obtained age of MDC mica from the NWR213 laser is 521.2 ± 6.6 Ma, whereas the obtained age from the excimer laser is 528 ± 4 Ma. The ages are within the expected range but the raw signals from both lasers were varied and influenced by ablation characteristics of each laser including the ablation rate and aerosol formation processes. In addition, the differences between the Mica-Mg and MDC in physical properties have significant impact on the accuracy by adding 5 – 10% of uncertainty onto any unknown sample. The precision and the accuracy can be assessed by using the MDC mica as a secondary standard because it has well-known age and its RSD of $^{87}Rb/^{86}Sr$ did not exceed 1.75% with the excimer laser.

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The analysis of nothing and nearly nothing – concepts for reference materials for the Super-SIMS

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The Super-SIMS idea goes back to the year 1979 [1]. Since then several attempts have been made to install such instruments [2-5], although with varied success.

Most of the published data were either analysis of semiconductor materials or isotope ratios of natural materials. Having a strong focus on natural, metal, and mineral resources, the Helmholtz Institute Freiberg for Resource Technology installed such a system at the Ion Beam Centre at HZDR. This new Super-SIMS will be embedded into a system of consecutive micro-analytical methods devoted to the characterisation of minerals and ores. Therefore, our focus will lie on the analysis of ultra-trace elements in these natural matrices.

Despite the high precision, the accuracy of SIMS analysis can be problematic. The sensitivity factor as well as the instrumental mass fractionation vary with the chemical composition. This so-called matrix effect demands that the sample and the reference material (RM) should have exactly the same chemical composition and structure, this is difficult to achieve. Even trace elements and in the case of the Super-SIMS ultra-trace elements may affect the sensitivity factor. The compromise is the usage of matrix matched RMs.

The combination of good lateral and depth resolution of the SIMS instrument with the resulting small sample volumes / masses (sub ng-range) and the aspired detection limits in the pg/g range yield to the fact that the probability to meet one atom of the analyte in the sample volume will be < 1.

This contribution will stimulate the discussion about the concepts of detection limit, homogeneity and heterogeneity in RMs and present considerations about the design of future RMs for ultra-trace element analysis with the Super-SIMS.

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A Super-SIMS for resource technology

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The integration of an ion source with very high spatial resolution with a tandem accelerator is a long-standing concept for improving analytical selectivity and sensitivity by orders of magnitude [1-3]. Translating this design concept to reality has its challenges [e.g. 4-6]. Supporting a strong focus on natural, metallic and mineral resources the, Helmholtz Institute Freiberg for Resource Technology installed such a system at the Ion Beam Centre at HZDR. This so-called Super-SIMS will be at the core of a comprehensive pallet of micro-analytical methods devoted to the characterisation of minerals and ores. Secondary ion beam from a CAMECA IMS 7f-auto are injected into the pre-existing 6MV Dresden Accelerator Mass Spectrometry facility [7,8], which quantitatively eliminates isobaric molecular species from the ion beam. Our SIMS component can function as either a stand-alone device or can be used to inject the negatively charged secondary ions at energies of up to 40 keV (to match the acceptance conditions) into the accelerator. A dedicated ion optical unit has been constructed and installed to match the SIMS ion beam to the maximum acceptance of the accelerator.

We will present measurements of the performance parameters of the instrument as well as first results of halogen (F, Cl, Br, and I) determinations in galena, sphalerite and pyrrhotite.

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The nanogram-scale heterogeneity of IAEA-CO-1 calcite

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We used a Secondary Ion Mass Spectrometer to evaluate the $\delta^{13}C$ and $\delta^{18}O$ homogeneity of the IAEA-CO-1 calcite reference material, which was derived from Carrara marble sampled at Viaregio, Italy. According to the provider this material has assigned uncertainties of ± 0.030 and ± 0.1 ‰ (1s) for carbon and oxygen isotope ratios, respectively. Our isotope ratio determinations using a Cameca 1280-HR SIMS instrument consumed < 300 pg of material, which is orders of magnitude less than the mass consumed by gas source mass spectrometer analyses. The repeatability of our SIMS method as applied to calcite is better than ± 0.15 ‰.

CARBON: We conducted a total of N = 211 SIMS carbon isotope analyses on 26 fragments of this calcite material; the total range in these data is 40 ‰, with a corresponding 1s distribution of ± 4.5 ‰. These data show an anti-correlation between the measured $^{13}C^-/^{12}C^-$ and the $^{12}C^-$ count rate. This suggests the presence of two phases: an isotopically heavy, low-carbon material (e.g., calcite) and an isotopically light, high-carbon component (e.g., graphitised organic material). Considering only the 156 lowest $^{12}C^-$ count rate results we calculate a 1s value of ± 0.56 ‰; this implies that the supposed calcite component contains significant heterogeneity in its carbon isotope ratio at the picogram sampling scale.

OXYGEN: We also conducted a total of N = 195 oxygen isotope SIMS analyses on 26 fragments of IAEA-CO-1. This data set yields a 1s distribution of ± 0.17 ‰, which is similar to our method's repeatability. However, a fragment-by-fragment evaluation of our data from IAEA-CO-1 suggests that a modest level of variability in $^{18}O/^{16}O$ exists at the between-fragment scale.

Our data indicate that IAEA-CO-1 is not suitable for use as a microanalytical reference material for carbon isotope determinations. In the case of oxygen isotope determinations, IAEA-CO-1 should only be used for microanalytical purposes with caution. We will also present data from the next generation calcite reference material IAEA-603 (derived from the same marble material as used for preparation of IAEA-CO-1); we will discuss its limitations for use as a microanalytical reference material.

Au speciation in humic acids and Fe(III) compounds after sorption from chloride solutions

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There is a wide variation in the distribution of Au species over mineral fractions, including the existence of high contents of water-soluble/mobile species, in metastable systems such as weathering profiles and soils according to the data of sequential extraction [1]. The nature of such (mobile) gold is not finally established, as well as the behaviour of gold associated with various mineral fractions during sequential extraction. It is known about Au readsorption on a residual solid during leaching [2], which becomes significant if the sample contains carbonaceous/ graphitised matter.

Monomineral/single-component Au-containing compounds (quartz with coarse native gold; quartz with nano-sized native gold; sulfides with "invisible" gold; Au-containing Fe (III) compounds and humic acids obtained by both gold sorption and coprecipitation) were selected, in which the species of Au were studied using 7 step sequential extraction procedure: (1) water-soluble, (2) ion-exchange, (3) acid-soluble, (4) easily oxidisable, (5) reducible, (6) hardly oxidisable, (7) residual fractions. Au contents in the fractions were determined by the graphite furnace atomic absorption spectrometry (GFAAS) with preconcentration into the organic phase.

Results show that:
•	both coarse and nano-sized native gold are extracted in several stages (nano-sized – in 4, 5, 6 steps, coarse – in 4, 6, 7 steps);
•	"*invisible*" Au in sulfides leaches predominantly in hardly oxidisable fraction;
•	both sorbed and coprecipitated Au in Fe(III) compounds presents predominantly as a mobile species leached in 1-3 steps and less as a easily oxidisable species; extremely low amounts of Au are bound to iron compounds;
•	humic acids contain less mobile Au species than Fe(III) compounds but Au completely leaches in 4 step.

As a result it is established that:
•	joint extraction of several Au species in one step takes place (for example, joint extraction of Au in sulfides and nano-sized gold in 6 step);
•	Au readsorption processes are insignificant in the absence of a carbonaceous/ graphitised matter.
•	mobile Au species are associated with water-soluble Au complexes with organic ligands and desorbed Au (likely as a complexes similar to water complexes of Au before sorption and/or coprecipitation).

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UQAC-FeS: A new series of base metal sulfide quality control reference material for LA-ICP-MS analysis

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The ability of LA-ICP-MS to quantify trace elements at micron-scale has improved knowledge in almost all fields of science. *In-situ* analysis of base metal sulfide minerals (BMS) is of great interest in the geochemical community. Calibration and/or quality control should, like most material analysed by LA-ICP-MS, be processed with matrix-matched reference materials [1]. Analysis of BMS by LA-ICP-MS is particularly challenging because they contain volatile elements (e.g. S, Hg, Se, As, Te, Sn, Bi, Os,…) and because there are a very limited number of commercially available BMS reference materials e.g. MASS-1 [2]. We have developed a technique to produce "suitable" reference materials for the determination of a wide range of elements in sulfide minerals. "*Suitable*" refers to: 1) matrix-matching, 2) concentrations of selected elements at desired levels, 3) large batch production (>100g), 4) homogeneity (the level of tolerance can vary depending on the project requirement and instrumental capacities). Our sulfide-RM production requires: 1) preparation of a highly concentrated "*starting material*" from natural massive sulfide powder (10g) doped with native elements, solutions and/or other natural phases (e.g. arsenopyrite for As), 2) rapid and short (c.a. 1 minute) fusion at high temperature to avoid complete loss of the volatile elements, 3) rapid cooling to minimise segregation (typical for platinum-group minerals), 4) a pre-analysis of the doped material by LA-ICP-MS, 5) a first step of crushing (ceramic mortar), 6) mixing with non-processed powdered natural sulfide (e.g. pyrite, chalcopyrite or other desired matrix) 7) sieving (~75µm), 8) a second (and key) phase of crushing to produce sub-microns particle size in a high-power planetary ball mill [3], 9) homogenisation of the whole sub-micron powdered material, 10) production of pressed pellets, 11) homogeneity tests (LA-ICP-MS, SEM, micro-XRF), 12) round robin of analysis in which both *in-situ* and "whole rock" techniques are involved, 13) data compilation and, finally, 14) distribution of the sulfide QCRM. Results (concentrations and homogeneity) will be presented for UQAC-FeS-1 along with preliminary data for the new UQAC-FeS-2, and ongoing projects.

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Atom Probe Microscopy as a tool for geoanalysis

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In recent years, atom probe microscopy (APM) has made inroads into the chemical and isotopic analysis of geological samples. Significant progress has been made in applying atom probe techniques across a growing collection of minerals, with several research groups active in the field, addressing long-standing questions in planetary science, geochronology, and fundamental processes within geological materials [1-8].

Conventional geoanalytical techniques have remarkable chemical and isotopic sensitivity. However, their spatial resolution is typically at micron length-scales, with a few secondary ion mass spectrometry techniques capable of sub-micron resolution (e.g. NanoSIMS). The chemical and isotopic landscape of minerals at nm length scales is therefore largely unexplored – though it is expected to contain a wealth of information on past geological, biochemical and planetary processes. APM allows these variations to be studied at much smaller scales, providing access to new information of geological significance.

This presentation will showcase a selection of work across several geoscience applications, highlighting the significant role that APM is playing within the developing field of nanoscale geochemistry. Examples include the analysis of silicates, oxides, sulfides, and phosphates, with applications in mineral processing, extra-terrestrial materials, isotope geochemistry, and geochronology. Future prospects for geoscience applications will also be discussed [9].

[1] Valley, J.W. et al. (2015) *American Mineralogist, **100**, 1355-1377.* [2] Felmy, A.R. et al. (2015) *Chemical Geology, **395**, 119-125.* [3] Lewis, J.B. et al. (2015) *Ultramicroscopy, **159**, 248-254.* [4] Reddy, S.M. et al. (2016) *Geochimica et Cosmochimica Acta, **195**, 158-170.* [5] Piazolo, S. et al. (2016) *Nature Communications, **7**, 10490.* [6] Pérez-Huerta, A. et al. (2016) *Micron, **80**, 83-89.* [7] White, L.F. et al. (2017) *Nature Communications, **8**, 15597.* [8] Fougerouse, D. et al. (2018) *Chemical Geology, **479**, 251-258.* [9] Saxey, D.W. et al. (2018) *Scripta Materialia, **148**, 115-127.*

A Geoscience Atom Probe facility

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Atom probe microscopy (APM) is a powerful technique for the characterisation of three-dimensional structures at nanometre length scales. Using field-evaporation of atoms on to a two-dimensional detector, combined with time-of-flight mass spectrometry, it provides three-dimensional information down to 1 nm, including chemical and isotopic identification of individual atoms, with equal sensitivity across all elements. APM has been used to study a wide variety of material systems, from metal alloys to semiconductors, and more recently geological materials.

The Geoscience Atom Probe (GAP) facility was established at Curtin University in August 2015, with support from the Science and Industry Endowment Fund. Forming part of the Advanced Resource Characterisation Facility (ARCF) – a collaboration between Curtin, UWA and CSIRO – it is the first atom probe facility in the world aimed at establishing the technique within the geosciences. The GAP facility is housed within the John de Laeter Centre and hosts a Cameca LEAP 4000X HR atom probe microscope. This high-resolution ('HR') instrument is designed to maximise the mass resolving power (typ. >1000) of the time-of-flight mass spectrometer, thereby providing the best separation, and least convolution, between adjacent isotopic peaks for geological applications. The facility is open for researchers to access from academic institutions and industry.

This presentation will describe the workflow of the GAP facility and showcase some recent applications.

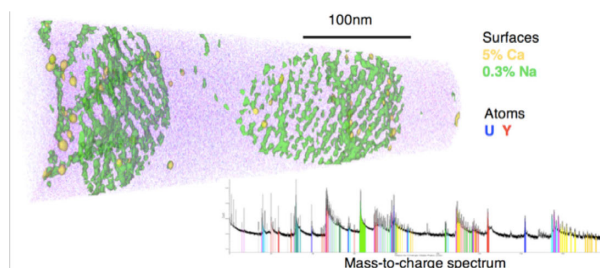*Figure 1. The Geoscience Atom Probe facility.*

Figure 2. Three-dimensional APM atom maps, iso-concentration surfaces, and time-of-flight spectrum, which reveal trace element distributions in a monazite sample.

Determination of Uranium(U) and Thorium by inductively coupled plasma mass spectrometry in geological reference materials

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Uranium-series dating can be used as tool for dating igneous rock. As a element with a long half-life, and often observed in the crust, it measures the age at which rocks were formed from the ratio of uranium (U) to thorium (Th). Accurate and precise analysis results are required to obtain geologically significant results. Inductively coupled plasma mass spectrometry (ICP-MS) is mainly used for the analysis of trace amounts of uranium (U) and thorium (Th) in geological samples.

We tested the sample digestion methods for some geological reference materials used in the ICPMS for the elemental analysis of uranium (U) and thorium (Th). The tested sample digestion methods are; normal acid digestion with Teflon vessel, alkali fusion, acid digestion with NH_4F and microwave digestion.

U-Pb zircon dating by LA-SF-ICP-MS at Geological Institute GIN RAS (Moscow)

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In order to put into operation the LA-ICP-MS facilities (Element2, and NWR-213) at Geological Institute of Russian Academy of Science (Moscow, Russia) a series of experiments by determination of optimal settings for zircon U-Th-Pb dating were carried out. When performing the ICP-MS tuning routine we tried not only to adjust the maximum sensitivity, but also to achieve an approximate conformity of the measured 206/238 ratio to that of the scanning standard, which was found to affect the accuracy. The Method used was based on the operating conditions proposed in [1].

We applied the following settings:

ICP-MS: measured masses – 206, 207, 208, 232 and 238; sampling time – 12, 16, 8, 8, 8 ms respectively; mass window – 4 %; samples per peak – 100; the number of scans – 800; background measurement time – 15 s; ablation time – 25 s; RF-generator power – 1200 W; sample gas (Ar) flow – 1,2 L min^{-1};

LA: spot diameter – 25 μm; frequency – 10 Hz; energy density – 5-10 J cm^{-2}; carrier gas (He) flow – 1.0 L min-1.

The data processing was carried out using software Glitter [2] and Iolite [3]. The dating results of some the widely used zircon standards with the manual integration during data reduction revealed a deviation from the TIMS ages not exceeding 2%. We will demonstrate examples of the dating detrital zircons (including samples previously dated in other laboratories) and zircons of igneous rocks. The probable reasons of instability of the analysis and possible ways to improve the results will be discussed.

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High-speed 3D imaging of geological materials using rapid-response laser ablation cell technology

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Laser ablation-inductively coupled plasma-mass spectrometry (LA ICP-MS) is known to be affected by the effects of aerosol dispersion in the sample transport system: seconds long pulse response duration, increased gravitational settling, etc. As a result, even though LA ICP-MS has become a routine analytical technique for elemental and isotopic analysis, the lateral resolution of images generated using continuous scanning is often sub-optimal. Furthermore, due to the complexities of experiment setup and data reduction, in-depth 3D LA-ICP-MS [1] has received even less attention. In this contribution we present new developments in ultra-rapid response laser ablation cell technology coupled to innovative state-of-the-art data reduction software. This enables the generation of high-resolution images assembled automatically to create 3D distribution maps of elemental, isotopic, and even geochronological information. The use of rapid-response technology enables the acquisition of full pulse signals of less than 3 ms (FW0.1M), which allows the increase of the laser's repetition rate and scanning speed, in order to decrease scanning time without compromising data quality. Due to the increased mass flux in this setup, sensitivity is also increased, allowing for a higher lateral image resolution to be achieved.

The zircons used for this study are from the Miaskitic alkaline suite of the Ditrău Massif in Romania, in which zircon occurs frequently throughout the magmatic, pegmatitic, and deuteric evolution stages of the intrusion. These zircons are associated with magnetite and subordinately biotite, which occasionally forms schlieren in the coarse-grained nepheline syenite. As interpretations vary on the relative role of orthomagmatic and metasomatic processes in the formation of especially the coarse-grained nepheline syenitic internal zone of the massif, the isotopic distribution maps allowed for additional discriminating criteria. As such, internal zoning consistent with magmatic crystallisation was identified.

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In-situ lithium isotope ratio in natural and synthetic glasses by LA-SF-ICP-MS

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The large relative mass difference between lithium isotopes (^{6}Li and ^{7}Li) results in high fractionation in natural systems. Therefore, lithium isotopes are becoming a useful tool to understand several processes such as crust–mantle recycling, silicate weathering and fluid–rock interaction. Over the last years, publications concerning applications and measurements of lithium isotope ratios are increasing in geological and environmental areas. In general, the data presented are obtained after the isolation of lithium isotopes from a matrix using equipment with multiple detectors (MC-ICP-MS, TIMS and SIMS), due to the high measurement precision these instruments are able to provide.

Here we present preliminary data obtained using a laser ablation (Excite 193, Photon Machines, USA) coupled to a single collector SF-ICP-MS (Element XR, Thermo, Germany). The aim of our work is to test precision, accuracy and limits using isotope ratio data obtained *in situ* in single collector equipment. We used MPI-DING, NIST SRM 612 and some USGS natural and synthetic reference glasses as unknowns to evaluate our method. Established measurement protocol consisted of five 80 µm spots, with laser firing at 10 Hz and 9 J cm^{-2} during 80 seconds (20 seconds for local baseline measurement). Iolite software was used for data reduction (baseline subtraction, mass fractionation and instrumental drift correction).

In a preliminary experiment GSD-1G and SRM 612 provided δ^7Li = 30.2 ± 5.3 ‰ and 32.1 ± 5.9 ‰ (2SD, n=5) respectively. These data and respective uncertainty, are in good agreement with literature (δ^7Li = 31.0 ± 2.3 ‰ and 31.2 ± 0.1 ‰)[1]. On the other hand, under the same conditions, BCR-2G yielded δ^7Li = 5.2 ± 15 ‰ (2SD, n=18), confirming significant inhomogeneity as previously reported (δ^7Li = 4.07 ± 4.70 ‰) [1], limiting its use as reference material for *in-situ* determinations.

After homogeneity tests, the material GSD-1 (synthetic glass with basaltic composition) was selected as reference material for bracketing and drift corrections in future experiments.

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Success and pitfalls during the dissolution of silicate rocks in a microwave oven with a solution of NH$_4$HF and HNO$_3$

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The complete dissolution of representative test-portions of powdered rock samples for the determination of the mass fractions of trace elements by solution ICP-MS requires aggressive and time-consuming acid digestions or fusion with appropriate fluxes. Concerning acid digestions; in recent years some authors have used ammonium bifluoride/fluoride as alternative sources of HF to solubilise the silicates phases. Here we describe a microwave oven dissolution method that employs 100 mg sample test-portions and a solution that contains ammonium bifluoride and nitric acid. The whole procedure takes up to 4 hours and is entirely performed in a closed vessel system, including the evaporation steps, therefore minimising sample contamination. In hundreds of digestions, we never observed precipitation of fluorides, which is frequent during the regular pressurised decomposition of acid rocks. Results obtained for replicates of twenty international reference materials of igneous rocks with a broad range of silica content, and also one of a shale, presented recovery factors of forty-one trace elements mostly better than 90%. The results obtained for Zr and Hf served as an indication of the completeness of the dissolution, which was effective for AC-E, OShBo, RGM-1, YG-1, STM-1 and remaining RM, except G-2 and GSP-2. The mean recoveries of Zr and Hf in these two RM attained only 70%, despite the satisfactory results obtained for most other trace elements, including the heavier REE, which in a rock sample can be predominantly associated with zircon. As general purpose applications require the quantitative dissolution of all mineral phases of a sample test-portion, we conclude that to advance in this field of geoanalysis, we need a better understanding on how characteristics such as crystallinity and composition of some minerals control their reactivity.

Calibration systematics of the LA-ICP-MS at the Geological Survey of Brazil

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In 2017 the Geological Survey of Brazil (GSB-CPRM) coupled a laser system to a mass spectrometer (LA-ICP-MS) to quantify key trace and minor elements in minerals such as garnets, zircons and others relevant to geological studies. During the implementation, it was calibrated in a two-stage process in order to optimise analytical conditions such as gas flows, radio frequency (RF) power, laser ablation settings and also to reduce common interferences.

The first stage investigated the signal intensities from Li^7, Ca^{42}, Sc^{45}, Y^{89}, La^{13}9, Pb^{208}, Th^{232}, U^{238}, Hg^{202}, background (220 amu) and ThO in NIST 610. After 31 analytical sessions a nebulizer Ar gas flow of 0,7L/min, He gas flow of 0,5L/min and RF power of 1250-1300W, were considered the best values for the analytical routines. The second stage focused on adjustment of laser ablation parameters by determining concentrations of more than 40 elements in NIST 612 (external standard) and KL2-G basalt glass (unknown; MPI-DING serie). Analytical runs of 120s (60s of background), in alternating sequences of standards and unknowns, used Ca^{42} as internal standard and Glitter® as data reduction software. For that purpose, the repetition rate, spot size, energy and raster velocity were varied.

The results obtained for both materials were evaluated by comparison with preferred means ([1] for NIST 612 and [2] for KL2-G), including the REE chondrite-normalised diagrams for the basalt glass. Most NIST 612 values obtained lay within ±15% of the reference, except for Be, Mg, P and Ti, with higher deviations. Relative standard deviation (RSD) for most analytical sessions were between 1% and 10%, other than for Li, Be, Mg, P, Ti and Zn. In KL2-G most elements also lay within ±15% of the reference, except for Li, Ti, Zn, Mo and Sb. The range in RSD values varied, in almost all analytical sessions, from 1% to 10% for Sc, Ti, V, Cr, Mn, Co, Ni, Cu, Zn, Ga, Rb, Sr, Y, Zr, Nb, Ba, La, Ce, Pr and Nd. The REE patterns also showed overall concordance relative to [2]. This results indicated spot sizes and repetition rates of 45µm to 20Hz, and 50 µm to 10Hz respectively, as ideal conditions for these materials.

In general, the instrument and this established routine can be considered already suitable for *in-situ* analysis of geological materials with individual laser settings, to a broad range of matrices.

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Thermochromatic method application for express local prospecting of gold mineralisation productivity

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When prospecting and exploring gold mineralisation within Badransky and Tokichansky ore fields (Eastern Siberia, Russia), an unconventional thermochromatic method was successfully tested that can replace expensive mineral analysis of grinding samples and, in combination with other methods, increase the accuracy of prospect-exploring evaluations. Using the thermochromatic method, the express qualitative mineralogical analysis of any rocks and ores is possible. It was found that in the process of calcination in a muffle furnace of gold concentrated samples swell up and crack, while non auriferous ones remains thermally intact [1].

Samples, weighing 50-75 g, selected from ore deposits and host rocks, were placed in a muffle furnace and were being calcined up to a temperature of 900°C for 2 hours. Different colouring is provided by compounds appearing as a result of ore minerals decomposition (oxides, sulfides, sulfosalts) from auriferous parageneses.

Host rocks, not containing other sulfides besides the usual pyrite and pyrrhotite, are coloured in brown of different shades due to the Fe oxidation. Ore zones with commercial Au and containing arsenopyrite, sphalerite, sulfosalts and arsenic pyrite, become off white and white-purple colour in various shades during the calcination process. The white colouring is caused by the sublimates layers of As_2O_3, Sb_2O_3 and ZnO during the arsenopyrite, arsenic pyrite, sphalerite, and antimonite decomposition. The purple-white ones with a bluish tinge appear in the antimony sulfosalts and partly dolomite decompositions (mixed with Sb) from the ore parageneses with the formation of Sb_2O_3 layer. Yellow, orange-yellow and greyish-yellow shades, sometimes diluting the white colour of calcined rocks, are formed due to released Bi_2O_3 and PbO layers during the decomposition of Bi- and Pb-containing ore minerals (sulfides and sulfosalts).

[1] Tatarinov, A.V., Yalovik, G.A. and Yalovik, L.I. (2011) *Ores and metals. №5.*

Method for estimating the basic composition of fine-dispersed inclusions of native gold in a matrix of a sulfide mineral by electron microprobe

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Many gold deposits have native gold in the form of fine-dispersed inclusions of micrometer- and submicrometer size. The existing methods of matrix correction in Electron Probe X-ray Microanalysis (EPMA) reliably define the contents of Au, Ag and other associated elements in particles larger than the area of X-rays generated, commonly over 3-5 μm. However, when smaller-sized inclusions are analysed, a part of the microprobe's electrons excite the elements of the mineral-surrounding matrix, which significantly reduces the reliability of determing microinclusion composition.

In the present work we propose a method for evaluating the basic composition of the fine-dispersed gold of micron and submicron size in a sulfide mineral matrix exemplified by pyrite and arsenopyrite for the case, when the area of X-ray generation is larger than the inclusion size. This technique is based on extrapolating content trends for the inclusion vs matrix elements into the region, where the content of matrix elements tends to zero. The technique is substantiated through calculations of X-ray radiation intensity based on modelling of electron trajectories in a sample with the Monte Carlo (MC) method applied. This approach is handy in assessing the native gold content in finely dispersed inclusions occurring in an homogeneous matrices, as well as the uncertainty of its determination. It is noteworthy, that extrapolation of content trends into the region, where the matrix element content tends to zero, makes it feasible to correctly evaluate the content.

Application of this technique was exemplified with the EPMA procedure intended to explore native gold in arsenopyrite. The approach was helpful in assessing gold and silver contents in finely dispersed inclusions sized 1 μm and smaller from the Natalka ore gold deposit (north-east of Russia).

[1] Love, G., Cox, M.G.C. and Scott, V.D. (1977) *Journal of Physics D: Applied Physics,* **10**, 1, 7.

Laser Induced Breakdown Spectroscopy: Mineralogical mapping of drill core and thin sections

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Laser-induced breakdown spectroscopy (LIBS) is capable of determining the chemical composition of a substance by focusing a laser beam on its surface and detecting the atomic emission from the resulting plasma cloud. Multiple spectral lines are produced for each element above detection limit in the sample. Two areas which LIBS has the advantage over XRF scanners is range of elements that can be detected and speed of analysis.

LIBS can detect most of the naturally occurring elements including those elements lighter than sodium. For comparison XRF is limited to elements with atomic numbers greater sodium or potassium for some configurations. Meaningful data can be collected with a few laser pulses significantly speeding up the analytical time required to scan a large surface area. The analytical area can be as small as 10 μm when detailed mineral mapping is required.

Matrix effects, plasma stability and complexity emitted spectra complicate quantification of LIBS data and "*calibration-free*" quantification is an area of on-going research. Classification is often a simpler problem than quantification.

Drill core was scanned using a customised Applied Spectra J200 instrument with a 266 Nd:YAG laser and enhanced CDD spectrometer. Analytical spectra phases analysed were compared with a "library" of mineral spectra to identify the particular phase. A similar approach was used to map polished thin-sections and the data compared with mineral maps produced by Scanning Electron Microscope Mineral Liberation Analysis.

Impact of atmospheric air and laser fluence on accuracy of zircon U-Pb dating

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LA-ICP-MS is common technique for determination of U-Pb ages in the mineral zircon. LA-ICP-MS offers higher spatial resolution and sample through put compared to bulk digestion ID-TIMS, however at a cost of inferior precision and accuracy. The accuracy of LA-ICP-MS U-Pb ages in zircon relative to their corresponding ID-TIMS ages has been shown to be a function of the degree of crystallinity which is controlled by the radiation dose from decay of U and Th [1]. Since the calibration of LA-ICP-MS U-Pb ages is typically done with one zircon reference material [2]. If there is a difference in the crystallinity of a zircon relative to the calibration material this will lead to an inaccurate result. Annealing zircon prior to ablation has been shown to decrease this effect [1], particularly for zircon with high radiation damage compared to the calibration zircon. Using a wide range of zircon reference materials, we present results on un-treated and annealed zircon (annealed at two different temperatures: 850° C and 1000° C) and demonstrate, with annealing, an improvement in accuracy for the high radiation dose zircons, but little to no effect on low radiation dose zircon. Additionally, we demonstrate that use of low (< 2 J/cm^2) laser fluence decreases the impact of the zircon crystallinity on the accuracy of the U-Pb age by LA-ICP-MS. Use of low laser fluence negates some of the advantages of annealing zircon grains. We also performed experiments that show that a small amount of moisture or air present in one area of the laser ablation cell can strongly affect the U-Pb results. This can be negated by making sure samples are placed under vacuum prior to analysis. Using optimised conditions we can achieve ~1% accuracy for high radiation dose zircon and ~2% accuracy for low radiation dose zircon.

[1] Allen, C.M. and Campbell, I.H. (2012) *Chemical Geology, 332-333, 157-165.* [2] S.E. Jackson, N.J. Pearson, W.L. Griffin and E.A. Belousova (2004) *Chemical Geology, 211, 47-69.*

The Mud Tank zircon revisited: New ID-TIMS ages reveal complex crystallisation history

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The Mud Tank zircon located in Strangways Range NT, Australia is a world famous locality for megacryst zircon up to several cm in size. Initial TIMS analysis by Black & Gulson [1] gave an age of 732 Ma +/- 5 Ma which has been confirmed recently by Horstwood et al. 2016 [2] using ID-TIMS with chemical abrasion. The Mud Tank zircon has been widely used as a reference material for U-Pb dating [3] as well as Hf isotopes [4], however LA-ICP-MS analyses done at CODES [5] suggest different crystals may yield anomalous ages compared to the TIMS analyses. Since matrix effects in zircon are well known [6], we confirm the LA-ICP-MS ages with new ID-TIMS analyses on two different Mud Tank megacrysts, one colourless and the other red in colour. The colourless megacryst shows simple CL sector zoning and a homogenous age of ~719 Ma while the red megacryst shows a range of ages between ~720 M and ~750 Ma and contains a mixture of CL sector zoning and anomalous '*wormy*' texture in CL. Trace element and Raman Spectroscopy results are presented to show a complex crystallisation history for at least some of the zircon megacrysts from the Mud Tank deposit with important implications for its use as a reference material for U-Pb dating.

[1] Black, L.P. and Gulson, B.L. (1978) *BMR Journal of Australian Geology & Geophysics, 3, 227-232.* [2] Horstwood, M.S.A. et al. (2016) *Geostandards and Geoanalytical Research, DOI: 10.1111/j.1751-908X.2016.00379.x.* [3] Jackson, S.E. et al. (2004) *Chemical Geology, 211, 47-69.* [4] Woodhead, J.D. and Hergt, J.M. (2005) *Geostandards and Geoanalytical Research, 29, 183-195.* [5] Thompson, J.M., Meffre S. and L. Danyushevsky, L. (2018) *Journal of Analytical Atomic Spectrometry, 33, 221-230.* [6] Allen, C.M. and Campbell, I.H. (2012) *Chemical Geology, 332–333, 157-165.*

Horizon scanning: An industry perspective on PFAS, an emerging contaminant of concern

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Per- and polyfuoroalkyl substances (PFAS) are synthetically produced highly-fluorinated organic compounds. PFAS are lipid and water repellent, highly stable and are effective surface-active agents in high temperature environments. These unique physio-chemical characteristics account for their widespread use in Aqueous Film Forming Foams (AFFF), household products, semiconductors, hydraulic fluids, photolithography and the textiles industry.

Legacy PFAS contamination has been found in soil, sediment, biota and surface and ground water. PFAS have also been detected in landfill leachate, wastewater and sewerage sludge. PFAS are highly mobile, resistant to environmental degradation and have been shown to bioaccumulate in the ecosystem. As PFAS are naturally resistant to degradation by hydrolysis, biotransformation and abiotic reduction, the management of waste and soil contaminated with PFAS is highly challenging.

The majority of PFAS destruction technologies remain in developmental stages, however a range of water and soil treatment technologies are being applied at sites across Australia and internationally.

This presentation describes the physio-chemical properties, fate and transport behaviour and analytical challenges associated with measuring PFAS in the environment. Remediation and destruction technologies applicable to PFAS contaminated soil and water will also be discussed.

Advances in techniques for analysis of H$_2$O with application to clinopyroxene in sub-arc mantle xenoliths, ocean island basalts and continental flood basalts

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Secondary ion mass spectrometry is routinely used to measure H$_2$O concentrations in glasses, melt inclusions and nominally anhydrous minerals, especially clinopyroxene. Until recently most of this work has been carried out on small geometry Cameaca ims 3f-7f instruments that had a small source chamber and surface volume compared to the more recent 1280 models. We have developed methods for H$_2$O analysis ($\geq$ 20-40 ppm) on the SHRIMP-SI, using cross calibration with FTIR and Cameca 6f measurements [1] and BiSn as an easily polishable mounting medium.

Using this methodology we have analysed the H$_2$O contents of clinopyroxenes from sub-arc mantle xenoliths, ocean island basalts and continental flood basalts and applied our H$_2$O partitioning data [2] to calculate original magmatic H$_2$O contents. Sub-arc mantle apparently can store between 88 and 3737 ppm H$_2$O, similar to some continental lithospheric mantle xenoliths [3]. Ocean island basalts from the Azores demonstrate that clinopyroxene can retain fidelity with regard primary H$_2$O contents (0.3-2.2 wt. %) which are elevated in the islands compared with the mid-Atlantic ridge [4]. Continental flood basalts are indistinguishable from island arc lavas (3-8 wt. % H$_2$O).

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Laboratory study of multiple sulfur isotope fractionation during SO$_2$ photolysis

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Multiple-sulfur isotope (^{32}S, ^{33}S, ^{34}S and ^{36}S) studies of ancient sedimentary rocks have led to the remarkable discovery of mass-independent fractionation of sulfur isotopes (MIF-S) in sulfides and sulfates older than 2.4 Ga [1]. Subsequent laboratory experiments have shown that MIF-S may result from photolysis of S-bearing molecules. There is a series of SO$_2$ photolysis experiments in which some effects of MIF-S were established by changing the values for temperature and pressure in photochemical processes (for a recent review see, e.g. [2]), but numerous questions still remain to be answered.

In the present research, the mass-independent fractionation of sulfur isotopes during SO$_2$ photolysis was examined in detail under a wide variety of conditions of SO$_2$ column density and temperature in experiments. These experiments were performed using a flow-through photochemical reactor irradiated with broadband ultraviolet light. Analyses of ^{34}S/^{32}S, ^{33}S/^{32}S and ^{36}S/^{32}S from the end products of SO$_2$ photolysis were performed with a new laser ablation method combining a femtosecond laser ablation system and a combustion reactor to convert sulfide aerosol into SF$_6$ gas. This approach provides an opportunity to determine sulfur isotope ratios in elemental sulfur with high precision (0.1‰ for δ^{34}S, 0.03‰ for Δ^{33}S, and better than 0.5‰ for Δ^{36}S) consuming much less sample material.

Results of our SO$_2$ photolysis experiments shown that the elemental sulfur products were enriched in ^{34}S (δ^{34}S $\approx$ 60‰ to 160 ‰). The magnitude of Δ^{33}S anomalies was a pressure function showing the drop in Δ^{33}S values from ~ +22‰ at lower pressure to ~ −4‰ at higher pressure. Our results, showing negative Δ^{33}S anomalies in the elemental sulfur products, are distinctly different from those observed in previous photolysis experiments [2] that report only positive values for photolytically derived elemental sulfur. The Δ36S anomalies in elemental sulfur were characterised by negative values (~ −38‰ to +3.3‰) with decreasing magnitude Δ^{36}S values at higher pressure. Our experiments also revealed that the Δ^{36}S/Δ^{33}S relationships of sulfur were depended on SO$_2$ partial pressure; the slope of Δ^{36}S/Δ^{33}S was found to be ranged -1.7 to +0.2 increasing as pressure was elevated. Results from the temperature experiments shown that the magnitude of Δ^{33}S anomalies was larger at lower temperatures than at higher temperatures for a given pressure. The experimental pressure-temperature data could help in interpreting the MIF-S sulfur isotope signatures in Archean rocks.

[1] Farquhar et al. (2000) *Science, 289, 756-758.* [2] Ono (2017) *Annu. Rev. Earth Planet. Sci., 45, 301-329.*

LA-ICP-MS analysis routine for trace elements in olivine

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The aim is to investigate the applicability of trace element incorporation in olivines to the identification of the types of rocks present in the source during generation of parental melts. Olivines are normally the first silicate minerals to crystallise from mafic and ultramafic magmas and hence bear great potential for new insights into our understanding of melting in the mantle and early magmatic processes. This application of olivine mineral chemistry was developed earlier for minor elements (Mn, Ni, Cr, Al) by electron microprobe; the extension to LA-ICP-MS permits access to a greater range of elements at lower concentrations.

In the last few decades, laser ablation ICP-MS became a powerful analytical technique for highly spatially resolved *in-situ* microanalyses in geochemical samples. This method is capable of rapid and precise simultaneous sampling of a broad range of masses with major to trace element concentration levels and/or isotope-ratio analyses. The ability to measure low concentration levels while maintaining a low detection limit enables the collection of more precise data that enhances the interpretation of processes recorded in the samples.

However, characterising trace element contents in phases with low concentration levels with LA-ICP-MS has challenges and limitations. Problems include interferences of doubly charged ions that overlap with element masses of interest, and interferences due to argide formation in the plasma (e.g. $^{23}Na^{40}Ar$ on ^{63}Cu). The latter requires astute choice of isotopes to be measured for some elements (e.g. ^{66}Zn, ^{67}Zn or ^{68}Zn). The overlap of $^{29}Si^{16}O$ on ^{45}Sc cannot be resolved by quadrupole-MS and must be corrected for. Therefore, careful handling of the data set before and after analysis is essential. Another challenge for natural samples is finding an appropriate and matrix-matched reference material, usually are synthetic glasses, with representative concentrations of a range of elements.

The limitation of Os isotopic analysis for geological samples using Faraday cups equipped with 10^{13} Ω amplifiers by NTIMS

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Introduction

The content of Os is typically 10 to 100 pg g-1 in crustal rocks and 1 to 5 ng g−1 in mantle peridotites. The measurement for these samples are badly limited by the signal/noise ratio via Faraday cups in previous studies. Fortunately, newly developed amplifiers equipped with 1013 Ω feedback resistors have increased the signal/noise ratios. This study measured Os isotopes in various sample sizes (5 pg - 15 ng of Os) using static Faraday cup collections with five newly designed 10^{13} Ω amplifiers. The purpose of this study is to determinate the limit of Os isotopes using Faraday cups with 10^{13} Ω amplifiers by NTIMS.

Standard materials and methods

Two solution standards (Merck Os and JMC Os) and five international rock reference (BIR-1a, BHVO-2, BCR-2, WGB-1 and WPR-1) were analysed. The rock standard have Os contents ranging from 21 pg/g to 15.2 ng/g. In this study, all data were measured using a TIMS (TRITON Plus) instrument at GIG, CAS. The TIMS was equipped with 10 Faraday cups and 10 amplifiers, one 10^{11} Ω, four 10^{12} Ω, and five 10^{13} Ω. The description of the chemical procedures, the filament loading techniques and details on the operation of the TIMS were given in Xu et al.[1] and Wang et al.[2-4].

Results

The Os content in these international rock reference samples was very low, it could only be measured via SEM before the 10^{13} Ω amplifiers were configured to the Faraday cup. In this study, the Os isotopic compositions of the five rock standard samples were measured using the 10^{13} Ω amplifiers, showing that the accuracy and precision were identical to the reported reference values, even if the Os amount was as low as 21 pg. The total Re blank was 1.4 pg, and the Os blank was 0.02 pg. The blank Os contribution to the total Os was less than 0.1% for all samples, and the blank correction was negligible.

Conclusions

Our study indicated that a small amount of Os, i.e., 10 - 500 pg, can be quickly and accurately determined using a static Faraday cup with 10^{13} Ω amplifiers at a NTIMS. The Os isotopic compositions of the international reference materials, the five rock samples, were also measured using the 10^{13} Ω amplifiers, which showed that the accuracy and precision were identical to the reference values even if the Os amount was as low as 21 pg.

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The stability over an 18-year period of consensus values as composition location estimators in results from the Geo*PT* proficiency testing programme

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The outcome of re-issuing the same geochemical test material to participants of the Geo*PT* proficiency testing programme without disclosing its identity, 18 years after its first appearance, has revealed remarkable stability of the independently derived consensus values for over 50 measurands. Not only does this demonstrate the robustness of the methods used to estimate the location of consensus values but also the stability of results from analytical systems over an extended period.

A comparison of datasets acquired in 1999 and 2017 proficiency tests for the same andesitic test material supplied by the US Geological Survey indicates that values for almost all measurands are indistinguishable according to an established comparability test [1, 2, 3], which takes account of associated uncertainties. In Geo*PT*, consensus values that are best estimates of true values of mass fractions for each measurand are derived by a combination of robust statistical evaluation of all contributed results and expert judgement. Although Geo*PT* credits '*assigned*' status to values when a number of rigorous conditions are met, and '*provisional*' when they are not met in full, both categories of result were included in this investigation.

Inferences that may be drawn from the exceptional agreement between the datasets include: (a) that the consensus estimation procedures were robust and gave consistent results; (b) that overall, the contributed analytical results have been unaffected by changes in: (i) the population of laboratories participating, (ii) the proportions of results produced by different techniques, (iii) the means by which systems are calibrated and validated, and (iv) the aspirations of laboratories progressively to attain higher quality results; and (c) that unsurprisingly, the test material has remained stable and unchanged during that time.

A further implication of these findings is that values obtained by the robust procedures employed in Geo*PT* generally have the stability that might be associated with values derived from a detailed reference material certification programme.

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Analytical capabilities of the Cameca 1280 SIMS instrument

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The Potsdam Cameca 1280-HR instrument has been in operation since November 2013. A major motivation for acquiring this technology was our desire to determine isotope ratios on sub-nanogram test portions masses while achieving analytical uncertainties comparable to those provided by "*bulk*" methods. Here we provide a brief overview of the performance of this instrument.

The 1280 already provides maximum transmission at mass resolution $M/\Delta M \approx 2000$, meaning that for isotope ratio determinations on many light elements it is possible to operate at or near full transmission. The resulting high count rates for major element analyses, such as $\delta^{11}B$ in tourmaline, $\delta^{12}C$ in diamond, $\delta^{18}O$ in silicates and $\delta^{34}S$ in pyrite, in conjunction with the multi-collection mode of data acquisition, frequently make it possible to achieve a data repeatability of ± 0.1 ‰ (1s) or better while needing only 80 seconds of data acquisition per analysis. Test portion masses for such analyses are generally in the range of 300 to 500 picograms. In order to achieve the best possible data quality for such applications, in most cases data are collected with the primary beam rastered over typically a 10 x 10 μm area, assuring a flat-bottomed crater, yielding a spatial resolution of better than 25 μm for such applications. Better spatial resolution, in some applications better than 5 μm, can be achieved at the cost of a somewhat reduced data quality.

A key limitation of SIMS technology is the need to employ matrix matched reference materials in order to quantify instrumental mass fractionation, test for time dependent IMF drift and also to establish the analytical repeatability of a method. Even when multiple RMs are available, it is often the characterisation of the RMs that is the limiting factor for the 1280's overall data quality. Here we highlight the example of determining $\delta^{11}B$ in tourmaline, where B_2O_3 contents are typically around 10 wt%. A suite of three such RMs was described by Dyar et al. (2001) [1], with which we routinely achieve $\delta^{11}B$ repeatabilities of ± 0.2 ‰ (1s) or better. For these same three natural materials the range in values for the wet chemical characterisations on N = 3 aliquots is ≤ 0.3 ‰ [2]. Nonetheless, we observe that the repeatability of the IMF, based on using all three of these materials and over the course of several days of data acquisition, is commonly no better than ± 0.8 ‰ (1s). It is unclear whether this broad distribution in results is solely attributable to the presence of a chemical matrix effect or if the characterisations of RMs' are also a contributing factor.

[1] Dyar et al. (2001) *Geostandards Newsletter, 25, 2-3, 441-463.* [2] Leeman and Tonarini, (2001) *Geostandards Newsletter, 25, 2-3, 399-403.*

SIMS analyses employing a Flood Gas Tracer

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We have identified a novel approach for calibrating a Secondary Ion Mass Spectrometer that is based on leaking an isotopically enriched gas into the SIMS vacuum chamber at the time of analysis. Using the Potsdam CAMECA 1280-HR ion probe, and injecting into the sample chamber high-purity oxygen with an isotopic enrichment of $^{18}O/^{16}O \approx 30$; we found that our secondary ion spectrum contained numerous molecular species incorporating the ^{18}O spike isotope. Based on the energy distributions of such ions we could conclude these form from gas adsorbed onto the sample surface at the point of sputtering. Furthermore, the process of surface saturation with flood gas derived oxygen is very rapid, effectively instantaneous, when operating at a chamber pressure of 1e-3 Pa.

Sputtering the 91500 reference zircon with a ~10 μA, $^{16}O_2^-$ primary beam and operating at a mass resolution of M/ΔM ≈ 4550, we observed a $^{238}U^{18}O^+$ count rate of ~8000 cps; the measurement of the $^{206}Pb^{18}O^+$ peak was not possible due to the $^{208}Pb^{16}O^+$ isobaric interference. The repeatability of the new method was found to be superior both to that obtained using a simple calibration of the relative sensitivity factor and to that using a power law fit (N = 34, quoted uncertainties 1s):

Calibration Method	Mean Age (Ma)
$^{206}Pb+/^{238}U^+$	1062.2 ± 9.6
$^{206}Pb+/^{238}U^{18}O^+$	1062.4 ± 3.0 Ma
Power law UO_2/UO	1066.6 ± 19.2 Ma
TIMS $^{206}Pb/^{238}U$ age	1062.4 ± 0.4 [1]

It thus appears that the new calibration strategy, at least in the case of U-Pb zircon age determination, may be an attractive alternative to established calibration methods. Whether the flood gas tracer is suitable for other SIMS applications and whether a similar method based on other flood gas species (such as halogens) offer advantages remain topics for future study.

[1] Wiedenbeck et al. (1995) *Geostandards Newsletter, 19/1,* 1- 23.

REEs in UHP garnet, its inclusions and matrix minerals

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One garnet of one fresh coesite-bearing eclogite from the Tso Morari massif, western Himalaya, was investigated by EPMA, LA-ICP-MS and Raman spectroscopy to reveal REE distribution processes, to monitor changing mineral paragenesis during subduction, peak metamorphism and exhumation and to constrain the p-T path.
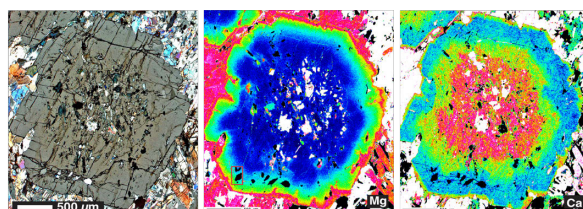

Figure 1. Element distribution map of garnet. [1]

Inclusions of garnet in the eclogite reveal a general but probably not entirely complete mineral growth succession of the protolith. Rare earth element concentrations (REE) were obtained for garnet, mineral inclusions in garnet and matrix minerals. The shape of the REE profile of the garnet reflects the growth and decomposition of minerals during subduction and, to a minority, during exhumation.
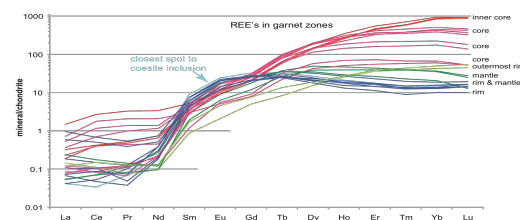

Figure 2. REE distribution in garnet zones. [1]

The inclusion-type zoning in the garnet together with their REE profiles shows that HREE were preferentially taken up as long as they are available by breakdown of matrix minerals. Judging from their REE pattern, these are mainly amphiboles but could also be epidote group minerals. The preference for HREEs has been proven for epidote group minerals, amphibole, but also for white mica. Hence, an opposed slope, negative for matrix paragonite but positive for paragonite included in the garnet mantle, will be presented.

Conventional geothermobarometry show maximum p–T conditions of around 44–48 kbar, 560–760 °C. Matrix glaucophane points to initial cooling (ca. 550 °C/<17 kbar) during decompression, followed by a stage of reheating (660–750 °C/~ 12 kbar) before final exhumation.

Supplementary Gibbs free energy minimisation modelling using the bulk-rock and fractionated chemistry of the fresh UHP eclogite yielded a rather steep and deep prograde p-T path, well within the diamond stability field.

[1] Wilke, F.D.H. et al (2015) *Lithos, 231, 77-91.*

Development and characterisation of five new USGS shale reference materials

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In support of expanding domestic unconventional petroleum resource development, the U.S. Geological Survey has collected and is preparing five new shale geochemical reference materials (GRMs) from major shale resource plays in the United States. Materials were collected from the Eagle Ford-equivalent Boquillas (ShBOQ-1; Texas), Marcellus (ShMAR-1; New York), Woodford (ShWFD-1; Oklahoma), Mancos (ShMCO-1; Colorado) and Niobrara (ShNIO-1; Colorado) formations. Discussions will focus on specific shale properties relevant to the petroleum industry and results from interlaboratory testing studies. These properties include mineralogy, major/trace element chemistry, and organic carbon quality/content. Mineralogy and major element content can provide information on shale brittleness, an important factor to help determine the applicability of hydraulic fracturing. Trace element distributions can be used as a proxy for organic matter content (Mo, U), to assess characteristics of shale depositional environment (Co, Ni, V, Cr), and may be useful for relating produced waters back to source rocks. Total organic carbon (TOC) content and programmed pyrolysis (e.g., Rock-Eval) parameters (Hydrogen index, Tmax) are widely used to assess petroleum resource potential, anticipate product quality (e.g., oil vs. gas), and estimate thermal maturity. These GRMs cover the range of organic and mineral properties commonly encountered in source rocks within unconventional petroleum systems. Sufficient supplies (400-800 kg) of the GRMs, available in both powdered and cuttings-sized varieties, were prepared to support the needs of the fossil energy industry and research community for the next 10-to-20 years.

Development of geochemical reference materials new directions new challenges

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As the search for new geologic resources continues and the models designed to understand them expand, a key aspect is the availability of reliable geochemical data. An important factor in this effort is the use of well characterised matrix matched geologic reference materials to verify data accuracy. Complicating this effort is the fact that traditional interest in elements such as gold copper lead and zinc must now share the stage with trace determinations of elements such as tellurium, lithium, lanthanides and scandium. Added to this need for different elements comes the desire identify the location of these elements at the micron spot size and mg/kg concentrations. Both bulk and micro analysis have their own requirements but they have a similar need for significant supplies of well characterised materials whose composition is ascertained through a rigorous scientific evaluation. Making these materials available to a wide audience for an extended period of time, increases their utility to the scientific community. The ability to independently confirm suggested information, provide new more robust values or generate additional information beyond the original scope of the materials as to the value of these materials. Discussion will focus on new geologic reference materials with special interest in today's "*critical elements*". Information on the new generation of microanalytical reference materials (MRMs) will also be presented, looking at sample types ranging from sulfides to glasses to metal oxides.

LA- ICP-MS as an imaging tool: past, present, and future

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A wide variety of microbeam methods are now in use for the construction of compositional images. Until very recently laser ablation ICP-MS was poorly represented in this field despite many significant advantages such as relatively clean spectra, widely variable spatial resolution, low detection limits, and the ability to perform isotopic as well as elemental analyses, e.g. Figure 1. The reasons for limited adoption of this platform by the imaging community are multiple but the primary drivers likely relate to the complexities involved in synchronising two fundamentally different instruments (i.e. a laser ablation probe and a mass spectrometer), and a lack of suitable processing software. A number of solutions are now available addressing these issues, however, and LA-ICP-MS is rapidly gaining traction as a complementary imaging tool. This talk explores recent developments in this changing landscape, and exciting new opportunities for the future.
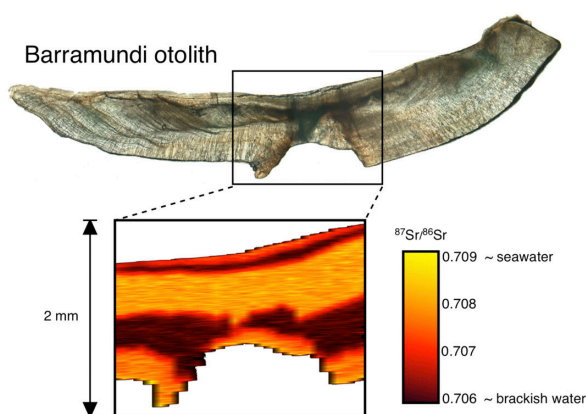

Figure 1. Laser ablation image of a fish otolith illustrating the variation in Sr isotope ratios between marine ($^{87}Sr/^{86}Sr$ ~0.709) and freshwater ($^{87}Sr/^{86}Sr$ ~0.706) values, revealing migration pathways.

Box-profile implants as geochemical reference materials for EMP and SIMS

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EMP (Electron Microprobe) and SIMS (Secondary Ion Mass Spectrometry) are widely used analytical techniques for geochemical and mineralogical applications. Nevertheless, metrologically rigorous quantification remains a major challenge for these methods. SIMS in particular is a matrix sensitive method; the use of matrix-matched reference materials (RMs) is essential in order to avoid significant analytical bias. A major problem is that the list of available reference materials for SIMS is vanishingly short compared to the needs of the analyst. A current evaluation of the GeoReM database [1] shows a strong focus on using the well-known NIST SRM 610-617 glasses for trace element analysis along with several zircons for isotope analysis, with few other matrices being readily available.

One approach for the production of matrix specific RMs is the use of ion implantation that introduces a known amount of a selected isotope into a material. This strategy is widely-used for SIMS applications in materials science, but rarely used for geochemical applications. Bumett et al. (2015) [2] demonstrated the appropriateness of this method. We choose the more elaborate way of implanting a so-called box profile to generate a homogeneous concentration of the selected isotope in three dimensions.

For proof of concept we used the mineralogically and chemically "simple" SiO_2 system which addresses many interesting scientific challenges, such as the Ti-in-quartz geothermometer [3]. We implanted either ^{47}Ti or ^{48}Ti into synthetic, ultra-high purity silica glass. Several box profiles with concentrations between 10 and 1000 µg/g Ti and a maximum depth of homogeneous Ti distribution between 200 and 3000 nm were produced at the Ion Beam Center in Dresden-Rossendorf. Multiple implantation steps using differing ion energies and ion doses were simulated with the SRIM (Stopping and Range of Ions in Matter) software [4], optimising for the target concentrations, implantation-depths and technical limits of the implanter. A thorough TEM assessment showed that the Ti is dispersed throughout the glass structure. We characterised several implanted test-samples by means of SIMS, EMP and other analytical techniques. The homogeneity of the Ti-concentration is within ± 5% uncertainty in all 3 dimensions.

[1] http://georem.mpch-mainz.gwdg.de, [2] Bumett, D.S. et al. (2015) *Geostandards and Geoanalytical Research, 39, 3, 265-276.* [3] Thomas, J.B. et al. (2010) *Contrib. Mineral. Petr., 160, 5, 743-759.* [4] Ziegler, J.F. (2004) *Nucl. Instrum. Meth. B, 219-220, 1027-1036.*

Effect of signal acquisition mode on isotope ratio precision and accuracy in ICP-quadrupole-MS

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An MS detector with the dual acquisition mode (analog and pulse-counting modes) has become the standard configuration in commercial ICP-QMS instruments since 1990s [1-2], which was used to extend the dynamic linear range for elemental analysis. However, the precision and accuracy of isotope ratios(IRs) that obtained on ICP-QMS was commonly limited. In this work $^{11/10}$B, $^{114/110}$Cd, and $^{208/206}$Pb were selected for measuring IRs in our experiment. As shown in Figure 1, the internal precision (RSD,%) obtained on single pulse mode (P) gradually improved with increased counting intensities, and the trend was in agreement with the principal of the counting statistical error (CSE). However, the RSDs suddenly deteriorated on dual (D) or single analog mode (A) with continuous increased counting intensities.
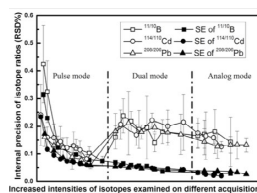

Figure 1. Internal precision (RSD,%) and statistical error (SE) of isotope ratios with increasing intensities by three acquisition modes (n=5).

Figure 2. Accuracy (RE%) of isotope ratios with increasing intensities, (a) raw data and (b) corrected data (b).

Obvious differences of the accuracy values using three acquisition modes (P, D, and A) were also observed. As shown in Figure 2a, the relative errors (RE, %) of raw data (uncorrected for mass bias) with P or A mode are better than that by D mode. Figure 2b shows the RE values after the correction by a commonly used SSB method. Although the RE with different acquisition modes seemed to be comparable; however, with an appropriate intensities range, good external precision (RESD) was only obtained by P mode (0.03%-0.09%, RESD) rather than D or A mode. In our experiment, we also found that the P-A crossover factor (CF, which applied to convert the analog to pulse signal outputs) was time dependent. However, the ICP-MS instrument can not dynamically monitor and calibrate it, so an initial CF value for each isotope was used in ICP-MS software. Therefore, this contradiction may be lead to the poor IRs precision and accuracy using D acquisition mode.

The optimal analytical performance for isotope ratio in ICP-QMS analysis should be conducted with the single pulse mode and the best signal intensity ranges from 0.7 to 1.2M, and the prefect precision (0.06% to 0.08%, RSD; 0.05% to 0.09%, RESD) and accuracy (<±0.07%, RE) were obtained.

[1] Onda, N. et al. (1996) *Tohoku Journal of Experimental Medicine, 178, 91-99.* [2] Hutton, R.C. et al. (1990) *Applied Spectroscopy, 44, 238-242.*

Apatite reference materials for oxygen and chlorine isotope analysis

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Calcium phosphates of the apatite supergroup can crystallise over a wide spectrum of geological environments, most often as solid solutions represented by chemical formula $Ca_{10}(PO_4)_6(F, Cl, OH)_2$. Complicated crystal chemistry of apatite makes it both very useful and challenging to apply in geochemical studies. Apatite is also the main component of bones and teeth and, therefore, it is widely used for reconstructing marine paleotemperatures based on investigation of microfossils and their oxygen isotope (δ^{18}O) compositions [1]. Chlorine isotopes (δ^{37}Cl) in apatite have recently gained attention and are being applied in studies of metamorphic fluids [2] as well as halogen reservoirs on Mars [3].

The determination of δ^{18}O is commonly conducted using gas source methods, but in contrast to secondary ion mass spectrometry (SIMS) they do not allow for targeted measurements at the microscale, which can be a key consideration when working with very small or heterogeneous samples. However, SIMS is hampered by the lack of homogeneous reference materials (RMs) that are required for quantitative measurements. Furthermore, matrix effect needs to be taken into consideration in chlorine and oxygen isotope analysis due to extensive substitution of Cl- by F- and/or OH- in apatite crystal structure. RMs for δ^{37}Cl are currently available in limited amounts and do not cover the full spectrum of Cl concentrations necessary to quantify the matrix effect.

We are developing a suite of well-characterised reference materials with ± 0.2‰ repeatability of δ^{18}O (1sd) and ± 0.1‰ repeatability of δ^{37}Cl (1sd) measurements. We have tested 58 apatite samples, provided by mineral collections, using both isotopic (SIMS) and chemical (EPMA) methods. We have identified a set of specimens representing various Cl-F-OH compositions and which are homogenous in our SIMS tests. With further determination of absolute δ^{18}O and δ^{37}Cl values by multiple GS-IRMS laboratories recruited to this research we plan to provide apatite RMs to all interested isotopic laboratories, making data they generate traceable between various laboratories.

[1]Joachimski et al. (2009) *Earth and Planetary Science Letters, 284, 599-609.* [2] Kusebauch et al. (2015) *Contributions to Mineralogy and Petrology, 170, 34.* [3] Bellucci et al. (2017) *Earth and Planetary Science Letters, 458, 192-202.*

A novel sample cell for reducing the "*Position Effect*" in Laser Ablation MC-ICP-MS isotopic measurement

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A novel laser ablation cell geometry significantly reduces the so-called position effect on high precision and accuracy isotopic measurement by Laser Ablation-Multiple Collector-Inductively Coupled Plasma-Mass Spectrometry (LA-MC-ICP-MS). Dependence of the peak shape, amplitude and pulse duration for a single laser shot on the carrier gas flow rate was studied with twelve analytical areas in the sample cell selected to assess the transport efficiency of particles ablated by a single laser shot under optimised gas flow condition. To further investigate whether position effects occur in the new cell, the Fe isotopic composition of Balmat pyrite was measured with extreme separation distances between the 'sample' and 'standard' in horizontal and vertical carrier gas flow directions in two gas flow rate scenarios. The results show that the position effect on Fe isotopic ratios is obvious when vortices formed at a flow rate of 0.85 L min^{-1}. However, ablated particle transportation is not influenced by vortices at a flow rate of 1.00 L min-1 and position effect is negligible. The Fe isotopic composition of *in-house* reference pyrite sample H1 is determined with δ^{56}Fe and δ^{57}Fe values of -0.11 ± 0.20 (2SD, n = 24) and -0.19 ± 0.40 (2SD, n = 24), respectively. These results are consistent with those measured by MC-ICP-MS solution methods (95% confidence interval). We conclude that the new sample cell can be extensively applied for high precision and accuracy LA-MC-ICP-MS stable isotope analysis.

Portable heavy metal analyser based on hydride generation-glow discharge micro-plasma sources

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In the present work, we describe a portable optical emission spectrometer (HG-APGD-OES) based on a new low power and low argon consumption (<8 W, 100 mL min-1) miniature direct current (dc) atmospheric pressure glow discharge (APGD) microplasma source [1]. In this method, the analytes were reduced to volatile species by hydride generation (HG), which were then transported to the APGD source for excitation. Different parameters affecting the APGD and the hydride generation reactions were investigated. The prototype miniaturised spectrometer is battery-operated with a total weight below 10 kg (battery included) and is compact measuring only 330 mm×240 mm×260 mm (Fig. 1). The instrument can run continuously for 9 h on a single battery charge (input of APGD is 500 V/15 mA). The total analysis time for one sample is no more than 1.5 min, and thus the instrument is capable of analyzing about 300 samples on a single battery charge. The proposed HG-APGD-OES coupled to hydride generation is also being tested in its capability for multi-element analysis. Preliminary experiments have also demonstrated the HG-APGD-OES could be used for determination of As, Sb, Hg, Cd, Pb, Bi, Se, and Ge with satisfied sensitivity.

Figure 1. The photo of the prototype instrument.

[1] Yang, C., He, D., Zhu, Z. et al (2017) *Analytical Chemistry, 89*, 3694-3701.

A simple method for preparing homogeneous and stable solid powder standards: applied to sulfide analysis by LA-ICP-MS

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A simple method of immobilisation powder samples was proposed to prepare the external calibration standard for the analyses of elemental compositions or isotopic ratios by LA-(MC)-ICP-MS. By mixing with epoxy resin, a multi-element doped powder mixture was transformed into a stable and mechanically resistant target, which is suitable for LA-ICP-MS analysis. Before solidification of the resin target, centrifugation was used to improve the uniformity of the target. This resin preserved silver sulfide standard (RPSS) developed in this study contains 47 kinds of elements including PGE, most chalcophile elements, siderophile elements, lithophile elements and REE. The multi-element doped powder mixture was also directly pressed into a pellet to carry on the contrast experiment. Homogeneity of most elements in the normal pressed powder pellet and RPSS from different batches were better than 10% (RSD, one sigma) on a micro-scale of bigger than 24 μm. Mass fraction of most elements of RPSS by LA-ICP-MS analysis calibrated against the pressed powder pellet agrees well with solution ICP-MS analysis. These results show that there is no obvious matrix effect between the RPSS target and the original powder sample. The proposed method is convenient to quickly prepare the homogenous and stable solid powder standards and can be adopted to prepare calibration standard for LA-(MC)-ICP-MS analysis. Because of the successful isolation of oxygen, this proposed method is particularly suitable for the preparation of standard samples such as sulfides, which are unstable in air and cannot be preserved for long periods.

In-situ U-Th-Pb dating and Sr-Nd isotope analysis of bastnaesite by LA-(MC)-ICP-MS

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Bastnaesite (Bastnäsite or Bastnasite) is the end member of a large group of minerals with the common formula (REE) $CO_3F \bullet CaCO_3$. This group is generally widespread, although it never occurs in large quantities, and represents the major economic light rare earth element (LREE) mineral in deposits related to carbonatite and alkaline intrusions. Taking into account that bastnaesite is easily altered and commonly contains inclusions of earlier-crystallised minerals, *in-situ* laser ablation is considered as the most suitable method to measure their U-Th-Pb and Sr-Nd isotopic compositions. Electron microprobe and LA-(MC)-ICP-MS analyses of forty seven bastnaesite samples from LREE deposits in China, Pakistan, Sweden, Mongolia, USA, Malawi and Madagascar indicate that this mineral typically has high Th, LREE and moderate U, Sr contents. Analysis of an *in-house* bastnaesite standard (K-9) demonstrates that precise and accurate U-Th-Pb ages can be obtained after common Pb correction [1,2]. Additionally, in our experiments we obtained a higher precision with the Th-Pb age, than with the U-Pb age because of the relatively higher Th than U content in most of our samples [3]. The moderate Sr and extremely high Nd contents in bastnaesites with generally lower Rb-Sr ratio also allow for the precise determination of *in-situ* Sr-Nd isotopic analyses by LA-MC-ICP-MS. In this contribution, we demonstrate the capability of bastnaesite as a geochronometer and tracer by using U-Th-Pb and Sr-Nd isotopic ratios. These results will have significant implications for understanding the genesis of endogenous ore deposits and formation processes related to metallogenic geochronology and ore deposits research in the near future.

[1] Sal'Nikova et al. (2010) *Doklady Earth Sciences, 430*, 134-136. [2] Yang et al. (2014) *J. Anal. Atom. Spectrom., 29*, 1017-1023. [3] Ling et al. (2016) *J. Anal. Atom. Spectrom., 31*, 1680-1687.

Rapid determination of Elements and Isotopes in NH$_4$F-modified Geological Sample Powders by LA-(MC)-ICP-MS

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We described a NH$_4$HF$_2$ digestion method as sample preparation for the rapid determination of elements and isotopes (Sr and Mg) in silicate rocks using LA-(MC)-ICP-MS. Sample powders digested by NH$_4$F at 250 °C for 2 hours form ultrafine powders, and various silicate rocks have a consistent grain morphology and size, allowing us to produce pressed powder pellets that have excellent cohesion and homogeneity suitable for laser ablation micro-analysis without the addition of a binder. The optimised NH$_4$F digestion method was applied to dissolve six silicate rock reference materials (BCR-2, BHVO-2, AGV-2, RGM-2, GSP-2, GSR-1) covering a wide range of rock types. Ten major elements and thirty-five trace elements were simultaneously analysed by LA-ICP-MS. The analytical results of the six reference materials generally agreed with the recommended values, with discrepancies of less than 10 % for most elements. The analytical precision is within 5 % for major elements and within 10 % for most trace elements. In addition, the isotopic compositions of ^{87}Sr/^{86}Sr and ^{26}Mg/^{24}Mg in basalts were measured by LA-MC-ICP-MS. Compared with previous methods of LA-(MC)-ICP-MS bulk analysis, our method enables the complete dissolution of refractory minerals, such as zircon, in intermediate-acidic intrusive rocks and limits contamination as well as the loss of volatile elements. Therefore, the NH$_4$HF$_2$ digestion method can be used as a new alternative in LA-(MC)-ICP-MS for bulk analysis of geological samples.

High precision U-Pb dating of terrestrial and extraterrestrial materials at SPIDE^{2}R lab, ANU

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Uranium-thorium-lead dating by isotope dilution thermal ionisation mass spectrometry (ID-TIMS) has been a major focus of the facility for Sensitive and Precise Isotopic Dating of Earth's and Extraterrestrial Rocks (SPIDE^{2}R) at the Australian National University since 2009. The range of materials we date include zircon (using chemical abrasion), apatite, baddeleyite, xenotime, rutile, cassiterite, and diverse meteorites and their components.

As the large range of sample types we analyse provides widely variable amounts of Pb (<10^{-12}g to >10^{-9}g), we use two different ion beam detection systems (multicollector Faraday arrays and secondary electron multipliers) in our two mass spectrometers (Triton Plus and *in-house* customised MAT 261). In addition, to be able to spike optimally across a wide range of ages (<1 Ma to >4560 Ma), we use ^{233}U-^{236}U-^{202}Pb-^{205}Pb spikes with different U-Pb ratios, followed by various methods of age calculations. To assure that the data acquired under these diverse conditions are accurate, precise and internally coherent, and consistent with the data produced in other labs worldwide, we designed a two-step system for cross-calibration and quality monitoring of analyses.

Step one is establishing consistency in spike calibration. The ^{202}Pb-^{205}Pb ratios are calibrated against the NIST standards SRM 981 and SRM 982 [1], and the ^{233}U-^{236}U spike IRMM-3636 [2] is used for uranium. The U/Pb ratios of the spikes are calibrated against the same set of gravimetric primary standards used for calibration of the EARTHTIME ^{233}U-^{235}U-^{202}Pb-^{205}Pb and ^{233}U-^{235}U-^{205}Pb spikes [3]. Within-run isotopic fractionation for both Pb and U is corrected using the measured ratio of two artificial isotopes (^{202}Pb/^{205}Pb and ^{233}U/^{236}U) and exponential fractionation law. The possible residual systematic uncertainties in Pb and U isotopic ratios caused by the differences in ion optics and ion detection systems of the mass spectrometers are monitored using NIST isotopic standards.

Step two is specific for the type of analyses. For dating of zircons and other terrestrial U-bearing minerals, we analyse the EARTHTIME synthetic isotopic mixtures that mimic the radiogenic U-Pb systems that evolved in the closed system for 100 Ma, 500 Ma, or 2 Ga. Additonally, single grains of Temora zircon standard treated the same way as samlpes are analysed. For meteorites, we analyse EARLYTIME synthetic isotopic mixtures that mimic the mixtures of 4560 Ma concordant radiogenic U-Pb system with primordial Pb of the Solar System.

Using these protocols, along with continuing method developments, the ANU SPIDE^{2}R lab is able to produce leading edge precision and accuracy U-Pb dates for a wide range of terrestrial and extraterrestrial materials.

[1] Amelin & Davis (2006) *JAAS, 21, 1053-1061.* [2] Richter et al. (2008) *IJMS, 269, 145-148.* [3] Condon et al. (2015) *GCA, 164, 464-480.*
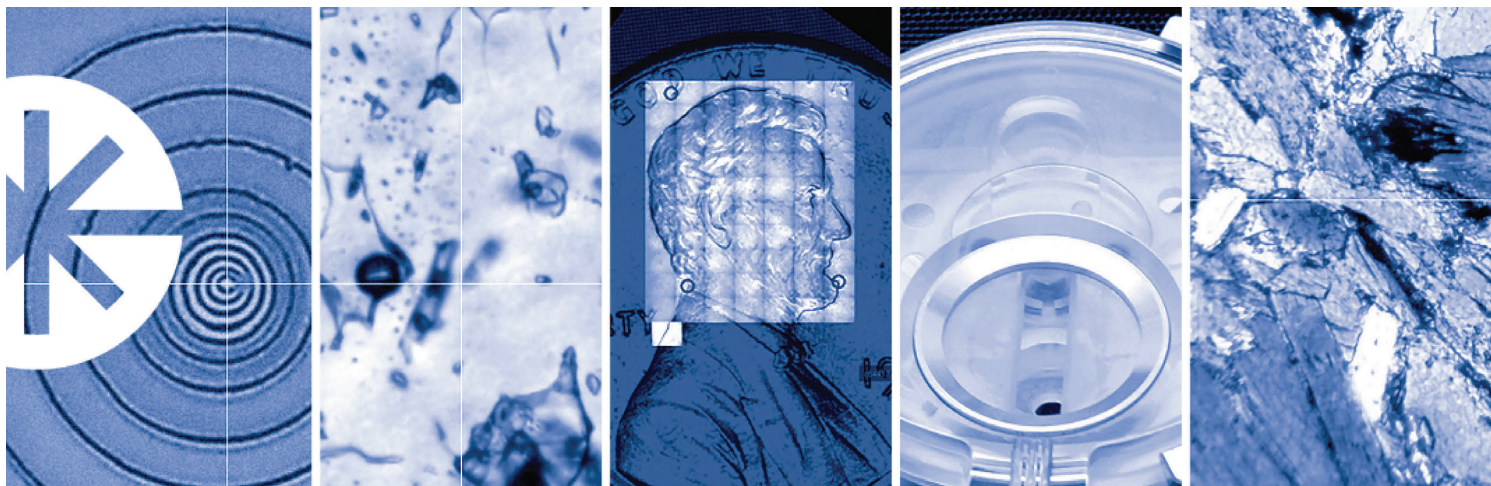
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- Sub-micron precision x-y-z stage and **bidirectional** trigger of mass spectrometer to provide the highest process control

APPLICATIONS

- Fluid Inclusion Research
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- Marine Sediment Analysis
- Multicollector-ICP-MS
- Gemstone Fingerprinting

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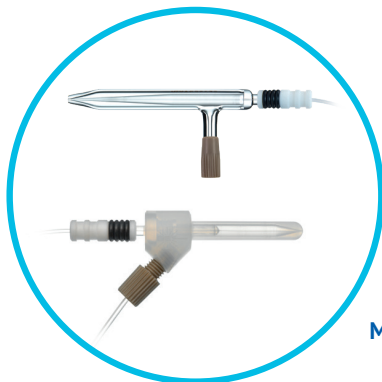
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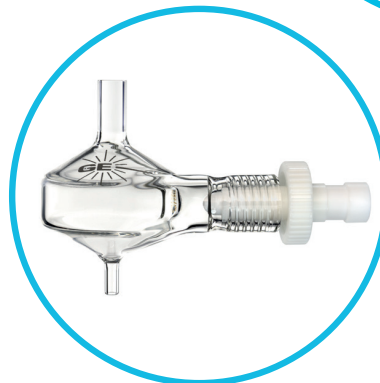
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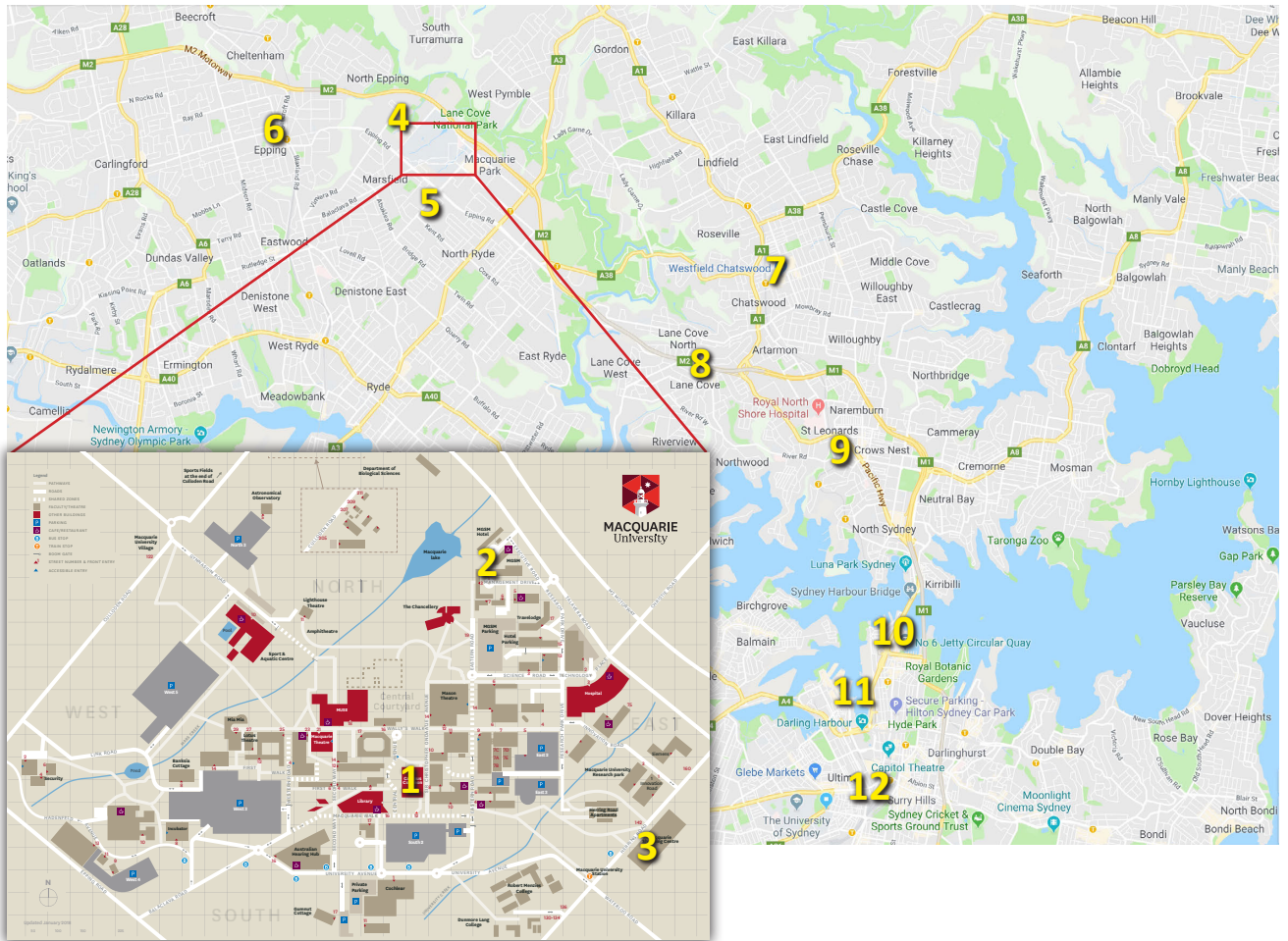
On or near campus:

1. Campus Common (food court and bar)
2. Lachlan's Restaurant
3. Macquarie Centre
    - Chefs Gallery - Chinese
    - Bondi Pizza - Italian
    - Grill'd - Burgers
    - TGI Fridays - American
    - and many more
4. Marsfield shops
    - Mediterranean
    - Basils Seafood
    - Red Ginger Thai
5. The Ranch - Pub
6. Epping (one stop by Train):
    - Borrellis Italian
    - Rawsons Restaurant@ the Epping Club
    - Plus a range of other restaurants
7. Chatswood (three stops by Train):
    - Bavarian Bier Cafe
    - 465 The Avenue - Bar
    - Din Tai Fung - Chinese
    - Hurricane's Express - BBQ
    - many more

Sydney area:

8. Lane Cove (20-30 minutes by Bus - 288,291)
    - Ullah - middle eastern
    - Via Napoli Pizzeria - Italian
    - Encasa - Spanish
    - Guzman Y Gomez - Mexican
    - Longeville Hotel - Pub
    - Plus a range of other restaurants
9. Crows Nest (40 minutes by Bus - 291 or 15 minutes by Train + short walk)
    - Garfish - seafood
    - Ha Na Ju Rin - sushi & teppanyaki
    - Raavi's Cumin - Indian
    - Thai Face - Thai
    - Plus a range of other restaurants
10. The Rocks (30 minutes by Train + short walk)
    - Aria - fine dining
    - Quay - fine dining
    - Altitude - fine dining
    - Range of bars and restaurants
11. Darling Harbour - key locations
    - The Star Casino
    - King Street Wharf
    - Harbourside shopping centre
12. Sydney Central
    - China Town - Haymarket
    - Surry Hills

11 Wally's Walk 210 (E5A)

Sunday 8th July 2018
Time	Event
09:00	Workshop Registration
10:00	Workshop I: Transportable methods
11:30	Morning Tea
13:00	Lunch
14:00	Workshop II: LA-ICP-MS
15:30	Rio Tinto Afternoon Tea
	Conference Registration opens
17:00	End Workshop 2 / Registration closes

Monday 9th July 2018
Time	Event
08:15	Registration
	Poster setup
09:00	Opening
	Reference materials
09:30	**Wilson**
10:00	Garbe-Schönberg
10:20	Belousov
10:40	*Morning Tea*
11:10	Alard
11:30	Wilson
11:50	Wudarska
12:10	Belozerova
12:30	Agilent / Sugiyama
13:00	*Agilent Lunch*
	Mining and remediation
13:50	Renno
14:10	Chu
14:30	Ashelford
14:50	*Group Photo*
15:10	*Afternoon Tea*
15:30	Poster session / Lab tour
17:00	Ice breaker
19:00	End Ice Breaker

Tuesday 10th July 2018
Time	Event
09:00	**Toase**
09:30	Powell
09:50	Bastrakov
10:10	Pribil
10:30	Daniel
10:50	*Morning Tea*
11:20	Company presentation
12:30	*BBQ Lunch*
	Dating and imaging
14:00	Belousova
14:20	Chen
14:40	J. Thompson
15:00	Wiedenbeck
15:20	*Afternoon Tea*
15:45	IAG General Meeting - non-members welcome
16:15	Poster session / Lab tour
17:15	End IAG Meeting
17:30	ThermoFisher / Wine Tasting
19:30	End Wine Tasting

Wednesday 11th July 2018
Company Exhibition

Posters on Display

Mason Theatre, 12 Wally's Walk (E7B)

Time	Event
08:30	Field Trip / *Meeting Point:* Australian Hearing Hub
10:00	**Thermo Fisher Laboratory tour** / *Meeting Point:* 12 Wally's Walk entrance
14:00	**Agilent Demo and Laboratory tour** / *Meeting Point:* 12 Wally's Walk entrance

Thursday 12th July 2018
Time	Event
09:00	Award Ceremony
09:30	**Woodhead**
10:10	Allen
10:30	Redaa
10:50	*Morning Tea*
11:10	Farkas
11:30	G. Thompson
	Instrumentation & new developments
11:50	Saxey
12:10	Ireland
12:30	*Lunch*
13:50	Y. Liu
14:10	Wiedenbeck
14:30	Baensch
15:00	*Afternoon Tea*
15:20	Poster Session
16:30	Lab tour
19:00	Conference Dinner Cruise

Friday 13th July 2018
Time	Event
09:00	**Chen**
09:30	Yang
09:50	Xu
10:10	Creech
10:30	*Morning Tea*
11:10	Lai
11:30	X Liu
11:50	Zhang
12:10	Enzweiler
12:30	*Lunch*
13:30	Danyushevsky
13:50	Jochum
14:10	Renno - Geoanalysis2021
14:20	Closing remarks
14:30	Poster session
15:30	Closing Reception
17:30	End of Event

Post Conference Field Trip
Sat 14th–Mon 16th July
South Coast NSW