Co-ordinated approach for Aerosol Trace element Solubility and Bioavailability Research in Oceanography

(CoATS-BRO)

1. Summary

The availability of iron directly impacts marine ecosystem functioning, and indirectly influences global climate through its role in the carbon cycle. In large areas of the ocean, atmospheric deposition is the dominant source of iron and other elements (which, depending on the element, can be either beneficial or detrimental to the ecosystem). Therefore, it is vital that we fully understand the processes governing the solubility and bioavailability of aerosol-derived metals. In order to advance current understanding in this field, our vision is two-fold: (1) standardize aerosol solubility methods and (2) establish standard protocols for assessing which of these methods accurately reflects the biologically-available fraction to marine biota. The results of this SCOR working group will provide both the foundation and a framework for proposals to address questions of methodology, biological cycles, and modelling of trace element availability. In addition, two papers will be published in open-access journals (a review paper linking the results of the biological-availability of iron experiments to the solubility of aerosol elements; a paper linking previously published and unpublished aerosol TE leach data to the standardized recommended protocols). One of these papers will be adapted for youngers audiences (aged 8-15 years). A low-cost and accessible aerosol reference material will be distributed to the community for intercalibration, and a database of the intercalibration results will be made publicly-available. Dedicated sessions will be hosted at major international meetings, which will promote the findings and recommendations of the proposed Working Group and highlight the work of SCOR.

2. Scientific background and rationale for the working group

Atmospheric dry and wet deposition is a major input route of iron (Fe) and other trace elements (TEs) to the sunlit, surface ocean (e.g. Duce et al., 1991). The availability of Fe exerts a fundamental control on biological activity in the ocean, primary productivity and fisheries while indirectly influencing global climate through its role in the carbon cycle (e.g. Martin, 1990; Moore et al., 2013; Mahowald et al., 2014). At the pH of oceanic waters (~ pH 8.2), Fe is relatively insoluble and, despite being maintained in solution in excess of its solubility by Fe-binding ligands, primary productivity is limited by insufficient Fe availability in a significant proportion of the global ocean (Moore et al., 2013). A common assumption of biogeochemical and ecosystem models is that all soluble Fe is biologically available, but there is evidence that casts doubt on this assumption (von der Heyden et al., 2012; Fitzsimmons et al., 2015). The links between the biological availability of Fe and its physico-chemical speciation can be complex and indirect. Yet, Fe solubility, both a constraint and a proxy for bio-availability, is a critical parameter in global models as they evolve to explicitly include Fe biogeochemistry; thus, it is essential to progress this research area by bringing together a multidisciplinary team to review the current state of knowledge and identify priorities for future research.

Simultaneously, there is an urgent need within the marine aerosol community to undertake further intercalibration and standardisation of aerosol solubility experiments. Due to the short residence time of aerosols in the atmosphere (hours to days), there are no aerosol cross-over stations among different cruises, so aerosol intercalibration requires a different approach than those used for water column parameters. An initial aerosol intercalibration exercise was undertaken in 2008 (GEOTRACES aerosol intercalibration; Morton et al., 2013). In this intercalibration a set of replicate aerosol samples consisting of a mixture of marine, lithogenic and anthropogenic components was successfully analysed for many total element and soluble ion concentrations. This exercise resulted in a recommended protocol to follow to ensure total dissolution of aerosol material, but it revealed discrepancies in the measurement of soluble aerosol trace element and isotope (TEI) concentrations, most importantly for Fe, a key parameter in many observational and modelling studies. Therefore, we propose a timely follow-up (a decade has passed since the first aerosol intercalibration; IC) to standardise aerosol TEI solubility measurements to address the discrepancy highlighted by the 2008 IC. This second round of intercalibration will use a 'reference' material, Arizona Test Dust (ATD, Powder Technology Inc.), that has been found to be homogeneous at subsample masses of 10-20 mg (Shelley et al., 2015). A large quantity of this reference material has been purchased and work has already begun to characterize its TEI composition. Our goal is to avoid the cost and time delays needed to produce a true certified or standard reference material, and to use the ATD material to intercalibrate analyses of aerosol TEIs in much the same way that the SAFe and GEOTRACES seawater samples have been used to intercalibrate the analyses of TEIs in seawater (Baker et al., 2016).

In addition to investigations with the ATD material, a large number (e.g. 60) of aerosol samples will be collected simultaneously for distribution to WG participants and other interested parties. Most marine TE aerosol collections are done using high- or low-volume samplers that filter known volumes of air onto suitable filters, and therefore it is necessary to undertake a portion of the IC using the same sampling procedures currently in use by the marine aerosol community. As part of this second IC round, we propose to use the aerosol-laden filters as well as the ATD for the intercomparison of various aerosol solubilization schemes.

2.2. The importance of a standardized aerosol Fe solubility protocol

As aerosol Fe solubility is frequently used as a proxy for the bioavailable fraction in models, it is essential that we fully understand the links between the two and the controls on both. At present, several aerosol leaching protocols are used by different research groups (Morton et al., 2013), which hampers direct inter-lab comparison of aerosol fractional solubility data. This situation presents a challenge for modellers who want to use experimental data to validate and refine their models. Regardless of the leaching scheme used, an inverse trend between atmospheric loading and fractional solubility appears to be reasonably robust throughout the world ocean. Unfortunately, the value for fractional solubility is a function of the different leaching schemes to some extent (e.g. batch versus flow-through methods and/or differences in pH, ionic strength and contact time between the aerosols and leach medium). Therefore, it is a research priority to introduce some means of standardization into leaching protocols (Baker et al., 2016). An additional research priority that was recently identified was to link aerosol TE fractional solubility to bioavailability (Baker et al., 2016) which can be used to improve the predictive capabilities of the next generation of biogeochemical and ecosystem models. The focus of this WG will be on Fe, but a strength of bringing together this diverse group of Full and Associate members is that data for other TEs will be produced.

2.3. Why a SCOR Working Group?

Much of our current understanding of the ***relationship between the solubility*** of trace nutrients in aerosols ***and the availability*** of those nutrients to marine microbes is ***operationally defined***, but few of these methods have been properly compared (or intercalibrated; Morton et al. 2013) and even fewer accurately related to bioavailability in the field or lab cultures. Our vision is two-fold: (1) standardize solubility methods and (2) establish standard protocols for assessing if any of these methods represent an accurate bioavailable fraction in the marine environment. The results of this SCOR working group will provide both the foundation and a framework for proposals to address questions of methodology, biological cycles, and modelling of trace nutrient availability. A co-ordinated global effort of this type would not be funded through national funding channels.

This vision will be realized by bringing together experts in the fields of aerosol TE solubility, aerosol chemical composition, experimental biogeochemists focused on the bioavailability of TEs and modellers (biogeochemical, ecosystem and atmospheric) in order to:

i) Produce an environmentally relevant, consensus aerosol reference material (ATD).

ii) Link the results of aerosol leaching experiments to bioavailability of TEs in the ocean. Specifically, we need to bring together aerosol and bioavailability/uptake researchers to understand which aerosol leach provides the closest approximation to what the bioavailability experiments tell us. It is also important that it is understood and communicated which fraction of Fe is accessed by the various leaching schemes in use by the community.

iii) Use the information from (ii) to agree on a standardized aerosol leaching protocol.

iv) Link historical aerosol leach data with outputs from the recommended standardized protocol

3. Terms of reference (ToR)

ToR1. Establish a database of the composition and fractional solubility of elements in the aerosol reference material, Arizona Test Dust (ATD, nominal size 0-3 µm diameter).

ToR2. Identify gaps in current knowledge of the relationship between the physico-chemical speciation of elements in aerosols, aerosol trace element solubility and bioavailability.

ToR3. Undertake a community aerosol intercalibration exercise to standardize aerosol solubility experiments.

ToR4. Improve parameterization of aerosol trace metal solubility in combined atmosphere-ocean models.

The ToRs will be met by:

- Distributing sub-samples of the aerosol reference material, ATD, to the marine aerosol community. The ATD will be chemically characterized using e.g. mass spectrometry, acid digestion, Synchrotron X-ray fluorescence spectrometry (S-XRF), XANES and scanning electron microscopy (SEM) (**ToR1**).
- A review paper will be the outcome of **ToR2** and will inform the WG on the most environmentally-relevant leaching scheme(s), resulting in identification of standardized leach protocols.
- Collecting replicate sub-samples of authentic aerosols collected on filters and distributing them to the marine aerosol community. The sampling period will coincide with the maximum period of Saharan dust deposition. The equipment necessary for this sample collection is available from Florida State University. The equipment will be shipped to Miami in advance and forecasts monitored to ensure that the samplers are deployed during a Saharan dust deposition event. These samples will be characterized using the same techniques used to characterize ATD. (**ToR3**).
- **ToR4** will be realized through open and focused dialog with the biogeochemical modellers in the WG.

4. Work plan

Month 1:

First full WG meeting (Aquatic Sciences Meeting, 2019).

Planning meeting to address the following points:

- Which fraction do existing leach methods access?
- Which leach best replicates what the bioavailability experiments tell us?
- Which leach protocol will be used in the intercalibration?
- What do modellers need to know?

Month 2 – 12:

Project website and social media pages online.

Distribution of ATD to interested parties.

ATD must be digested using the recommended protocol (Morton et al., 2013) and data submitted before the end of month 12. This step is an absolute prerequisite for participation in the aerosol sample leach intercalibration.

Collection and physico-chemical characterization of replicate aerosol samples (min. 60) at RSMAS, Miami in July/August 2019.

Submit review paper: Linking the results of TE bioavailability of Fe experiments to the solubility of aerosol TEs.

Session on the bioavailability of TEs from aerosols at an international meeting.

Month 12-18:

Second full WG meeting (Ocean Sciences Meeting/EGU, 2020).

Parallel sub-group meetings to identify gaps in our knowledge.

- 1) Aerosol IC – results from ATD total digestion.
- 2) Bioavailability of TEs

Database of ATD total TEs online (submit to GeoREM).

Adaptation of the review paper for younger readers (aged 8-15).

Distribution of replicate aerosol samples from Miami.

Month 18-24:

Participants to complete leaching experiments by the end of this period and submit data.

Submission of proposals to funding bodies for bioavailability experiments necessary to address gaps in knowledge.

Months 24-36:

Third full WG meeting (Aquatic Sciences Meeting, 2021).

Meeting goal: A strategy for best practice in incorporation of the WG findings into the next generation of biogeochemical models.

Database of aerosol leach intercalibration online.

Submission of review paper linking historical leach data to the standardized leaching approach and physico-chemical composition of aerosols.

Funding for proposals to link historical leach data with the standardized leach data will be sought to address gaps in knowledge.

5. Deliverables

- A review paper will be published in an open-access journal (e.g. *Frontiers in Marine Science*) that summarizes the state of the art in linking the results of TE bioavailability of Fe (and other TEs) experiments to the solubility of aerosol TEs, with a focus on Fe. This paper will consider a number of key questions, e.g. i) is there a direct link between bioavailability and solubility, ii) what is nature of any link(s), iii) what experiments have been done to date (including experimental 'dead-ends'), iv) do existing leach protocols mimic natural processes and v) future directions and recommendations.

- The above review paper will be adapted for younger audiences for publication in *Frontiers for Young Minds*, or similar.
- A low cost and accessible aerosol reference material (ATD) analogous to the SAFe and GEOTRACES seawater TE reference materials.
- Database of an aerosol reference material (ATD) of total and soluble TE concentrations (GeoREM).
- Recommended procedures to follow when leaching aerosol material. This will be published on the GEOTRACES website and added to the GEOTRACES aerosol 'cookbook'.
- Publish a paper linking previously published and unpublished aerosol TE leach data to the standardized recommended protocol so that valuable historical data is not lost. This will be published in a special issue of an open-access journal (e.g. *Frontiers in Marine Science* or *Biogeosciences*) to reach a wide audience.
- Dedicated aerosol TE solubility and TE bioavailability sessions will be hosted at oceanographic research meetings (e.g. AGU Fall Meeting, Ocean/Aquatic Sciences, Goldschmidt).
- WG meetings and Town Halls will be held before, during or immediately after large international meetings (e.g. Ocean/Aquatic Sciences) to maximize opportunities for participation by Associate Members and others.
- A dedicated website and social media accounts (Twitter, Facebook) will be created so that any interested party can follow the progress of the WG and find out how to participate in the IC.

6. Capacity building

Our vision is that this WG will identify significant gaps in the current knowledge which will stimulate new research proposals and collaborations between established and early-career scientists globally.

Proposed membership of the WG is diverse in terms of gender and geographical representation, and it is interdisciplinary, promoting dialogue between chemical oceanographers, modellers and atmospheric scientists from around the world in a focused group. In addition to a number of the participants from the 2008 IC, early-career scientists will be members of this working group, which will aid their career development. There are two proposed Full Members from developing countries, Argentina and South Africa. The working group will seek opportunities to involve early-career scientists in sessions related to the group's topic and will provide mentoring opportunities (Urban and Boscolo, 2013).

The social media (Twitter, Facebook) accounts and the adaptation of the review paper for younger audiences will facilitate access to the work of this WG and the work of SCOR to a wider audience than the scientific community.

This outputs of this WG will be of particular interest to the GEOTRACES, SOLAS and IMBER communities, as these communities have an interest in processes that occur at the ocean-atmosphere boundary. We will seek opportunities for co-sponsorship from the aforementioned organisations (and other sources) as products of this WG will contribute to the realisation of their mission statements. In addition, this WG will complement current SCOR WGs: WG139. Organic Ligands – A Key Control on Trace Metal Biogeochemistry in the Ocean, WG141. Sea-Surface Microlayers, WG145. Chemical Speciation Modelling in Seawater to Meet 21st Century Needs (MARCHEMSPEC), WG151. Iron Model Intercomparison Project (FeMIP) and GESAMP WG38 (Changing Atmospheric Acidity and the Oceanic Solubility of Nutrients). Some of the Full and Associate members are members of the listed synergistic WGs which will help ensure that the work of this WG is complementary and does not repeat work already done.

The results of this group may be useful to the final cruises and processes studies of GEOTRACES, although most of the field work of GEOTRACES will be completed by the time this group is finished. Nonetheless, research that builds on the work of GEOTRACES will benefit from the work of the proposed working group.

7. Working group composition

This WG will have 10 Full Members and 10 Associate Members which have the expertise to address the terms of reference.

7.1 Full members

Name	Gender	Place of work	Country	Expertise relevant to proposal
Rachel Shelley (co-chair)	F	National High Magnetic Field Lab/Florida State University	USA	TEs in aerosols. GEOTRACES and CLIVAR aerosols. TE biogeochemistry
Simon Ussher (co-chair)	M	University of Plymouth	UK	Aerosol TE solubility and Fe biogeochemical cycling in the ocean
Peter Croot (co-chair)	M	National University of Ireland, Galway	Ireland	Aerosol Fe solubility and determination of metal binding ligands in seawater
Alakendra Roychoudhury	M	Stellenbosch University	South Africa	TE biogeochemistry in the ocean and particle dissolution kinetics
Cassandra Gaston	F	Rosenthiel School of Marine & Atmospheric Science/University of Miami	USA	Atmospheric chemistry. Composition of aerosols. Focus on soluble phosphorus and the single-particle mixing-state of nutrients in individual aerosol particles.
Karine Desboeufs	F	Laboratoire Interuniversitaire de	France	Aerosol TE solubility and composition

		Systemes Atmospheriques, Paris		
Diego Gaiero	M	National University of Cordoba	Argentina	High latitude dust and Fe biogeochemistry in Southern Ocean
Bikkina Srinivas	M	Stockholm University	Sweden	Marine aerosol Fe solubility
Christel Hassler	F	University of Geneva	Switzerland	Bioavailability of Fe
Akinori Ito	M	Japanese Agency for Marine-Earth Science and Technology	Japan	Atmospheric Fe modelling

7.2 Associate members

Peter Morton	M	National High Magnetic Field Lab/Florida State University	USA	TEs in aerosols. GEOTRACES and CLIVAR aerosols. TE biogeochemistry
Andrew Wozniak	M	University of Delaware	USA	Organic composition of aerosols
Alex Baker	M	University of East Anglia	UK	TEs in aerosols. GEOTRACES and CLIVAR aerosols. TE biogeochemistry
Alessandro Tagliabue	M	University of Liverpool	UK	Biogeochemical modeler
Yeala Shaked	F	Hebrew University of Jerusalem	Israel	Biogeochemistry of TEs in the ocean
Dolores Gelado-Cabellero	F	University of Las Palmas de Gran Canaria	Spain	Aerosol TEs
Jaw Cheun Yong	M	GEOMAR - Helmholtz Centre for Ocean Research, Kiel	Germany	Aerosol TEs and solubility
Tung-Yuan Ho	M	Academia Sinica	Taiwan	Marine biogeochemistry
Andrew Bowie	M	University of Tasmania	Australia	Fe biogeochemistry
Barak Herut	M	National Institute of Oceanography	Israel	Aerosol mineral dust; bioaerosols

8. Working group contributions

Rachel Shelley (co-chair)

Contributes expertise in the trace element composition of aerosols and trace element biogeochemistry in the ocean. Early-career scientist.

Simon Ussher (co-chair)

Expertise in trace element biogeochemistry, trace element air-sea interactions. Oversees aerosol collection of inorganic species at Penlee Atmospheric Observatory, UK.

Peter Croot (co-chair)

Marine biogeochemist whose research focuses on understanding the role of biogeochemical processes on the concentration and distribution of trace elements and chemical species in the ocean.

Alakendra Roychodhury

Marine biogeochemist. Expertise in dust and marine particle chemistry and biogeochemical dynamics in aquatic environments with a focus on reaction kinetics and other controls over elemental cycling.

Cassandra Gaston

Expertise in atmosphere-ocean interactions, atmosphere-biosphere interactions, atmospheric chemistry, heterogeneous chemistry, air pollution, climate change. Cassandra Gaston (RSMAS) chemically characterises Saharan dust by single-particle mass spectrometry, which provides vital data on the mixing-state of nutrients found in the aerosols. She also looks at the solubility of particulate phosphorus. Early-career faculty.

Karine Desboeufs

Extensive experience and expertise in determining atmospheric dust chemistry and making trace element deposition measurements.

Diego Gaiero

Expertise in understanding dust activity in southern South America. Diego Gaiero's main research foci are determining dust fluxes from southern South America, improving understanding of present-day and past dust provenance, and evaluating the relevance of iron-rich dust on the biogeochemistry of the Southern Ocean.

Bikkina Srinivas

Expertise in the chemistry of atmospheric aerosols. Fe speciation. Early-career scientist.

Christel Hassler

Expertise in the connection between iron chemistry with iron bioavailability and the role of microbes in the carbon pump.

Akinori Ito

Expertise in Earth System model development and application with a focus on Fe in the atmosphere.

9. References

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Appendix

Key Publications of Full Members of relevance to this Working Group.

Rachel Shelley

1. **Shelley, R.U.**, Landing, W.M., Ussher, S.J., Planquette, H., and Sarthou, G. (accepted). Regional trends in the fractional solubility of Fe and other metals in North Atlantic aerosols (GEOTRACES

cruises GA01 and GA03) following a two-stage leach. Submitted to Biogeosciences Discuss. DOI: 10.5194/bg-2017-415.

2. **Shelley, R.U.**, Wyatt, N.J., Tarran, G.A., Rees, A.P., Worsfold, P.J., and Lohan, M.C. 2017. A tale of two gyres: Contrasting distributions of dissolved cobalt and iron in the Atlantic Ocean during an Atlantic Meridional Transect (AMT-19). Progress in Oceanography. 158: 52-64. DOI: 10.1016/j.pocean.2016.10.013.
3. **Shelley, R.U.**, Roca-Marti, M., Castrillejo, M., Sanial, V., Masque, P., Landing, W.M., van Beek, P., Planquette, H. and Sarthou, G. 2017. Elemental composition and trace element fluxes from atmospheric deposition during the GEOVIDE campaign (Lisbon, Portugal – St John's, Canada; GEOTRACES GA01). Deep Sea Research I. 119: 34-39. DOI: 10.1016/j.dsr.2016.11.010.
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5. **Shelley, R.U.**, Morton, P.L. and Landing, W.M. 2015. Elemental ratios and enrichment factors in aerosols from the US-GEOTRACES North Atlantic Transects. Deep Sea Research II. 116: 262-272. DOI: 10.1016/j.dsr2.2014.12.005i.

Simon Ussher

1. Fishwick, M.P., Sedwick, P.N., Lohan, M.C., Worsfold, P.J., Buck, K.N., Church, T.M. and **Ussher, S.J.** 2014. The impact of changing surface ocean conditions on the dissolution of aerosol iron. Global Biogeochemical Cycles. 28: 1235-1250.
2. **Ussher, S.J.,** Achterberg, E.P., Powell, C., Baker, A.R., Jickells, T.D., Torres, R. and Worsfold, P.J. 2013. Impact of atmospheric deposition on the contrasting iron biogeochemistry of the North and South Atlantic Ocean. Global Biogeochemical Cycles. 27: 1096- 1107.
3. **Ussher, S.J.,** Achterberg, E.P., Sarthou, G., Laan, P., de Baar, H.J.W., Worsfold, P.J., 2010. Distribution of size fractionated dissolved iron in the Canary Basin. Marine Environmental Research, 70: 46-55.
4. Seguret, M.J.M., Kocak, M., Theodosi, C., **Ussher, S.J.**, Worsfold, P.J., Herut, B., Mihalopoulos, N., Kubilay, N. and Nimmo, M. 2011. Iron solubility in crustal and anthropogenic aerosols: The Eastern Mediterranean as a case study. Marine Chemistry, 126: 229-238.
5. **Ussher, S.J.**, Worsfold, P.J., Achterberg, E.P., Laes, A., Blain, S., Laan, P. and de Baar, H.J.W. 2007. Distribution and redox speciation of dissolved iron on the European continental margin. Limnology and Oceanography, 52: 2530-2539.

Peter Croot

1. Simonella, L.E., Palomeque, M.E., **Croot, P.L**., Stein, A., Kupczewski, M., Rosales, A., Montes, M.L., Colombo, F., García, M.G., Villarosa, G., Gaiero, D.M. 2015. Soluble iron inputs to the Southern Ocean through recent andesitic to rhyolitic volcanic ash eruptions from the Patagonian Andes. Global Biogeochemical Cycles, 29 : 1125-1144. DOI: 10.1002/2015GB005177.

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Alakendra Roychoudhury

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