

# MEETINGS

## Final Recommendations for Reference Materials in Black Carbon Analysis

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Last summer, a symposium was held to discuss aspects of global biogeochemical cycles, including organic matter cycling in soils, rivers, and marine environments; black carbon particle fluxes and the biological pump; dissolved organic matter; and organic matter preservation. Seventy scientists from various disciplines, including oceanography, soil science, geology, and chemistry, attended the 3-day meeting at the Friday Harbor Laboratories, a research station of the University of Washington.

"New Approaches in Marine Organic Biogeochemistry" commemorated the life and science of a colleague and friend, John I. Hedges, who was also involved in several groups developing chemical reference materials. Part of this symposium included a workshop on chemical reference materials, where final recommendations of the Steering Committee for Black Carbon Reference Materials were presented.

It is well known that biomass burning is an important source of CO<sub>2</sub> to the atmosphere. However, along with CO<sub>2</sub>, biomass burning also produces small amounts of solid, combustion-altered material, called black carbon (BC). BC has received increasing attention due to its importance in a wide range of biogeochemical processes. For example, BC storage in ocean sediments represents a long-term sink in the global carbon cycle, and BC aerosols in the atmosphere affect Earth's radiative heat balance. BC can be a useful tracer for Earth's fire history; it is a significant fraction of the carbon buried in soils and sediments; and it is an important carrier of organic pollutants. A compilation of recent review articles can be found at the Web site mentioned below.

BC is not one chemical compound; instead, it is best understood as a suite of compounds occurring along a "combustion continuum," ranging from partially charred biomass, through charcoal, to sub-micron soot spheres. The chemical properties of BC vary along this continuum. For example, charcoal retains some plant chemistry and morphology, but since soot is created by the secondary condensation of hot combustion gases, its geochemistry reflects combustion conditions and not plant precursors.

Variations in BC chemistry along this combustion continuum create serious methodological problems, as every BC measurement method detects a unique window of the BC spectrum. For example, methods which rely on optical microscopes to detect charcoal particles fail to detect sub-micron soot particles, while methods which rely on the refractory nature of BC fail to detect the partially charred material which can be easily degraded. Effective

atmospheric methods, which measure the absorptivity of a sample, cannot be used when BC occurs within an absorptive matrix, like soils or sediments.

The gravity of methodological problems was made clear in a recent inter-comparison study on a set of soils which showed a factor of 500 difference between BC concentrations measured over a range of techniques [Schmidt *et al.*, 2001]. Variation in BC measurement techniques has caused serious problems in a number of fields which measure BC. A few of these problems are the following:

(1) BC is a radiatively important aerosol, and its atmospheric concentration has undoubtedly varied over the last 1000 years. Because atmospheric BC measurements are made using techniques quite different from sedimentary BC techniques, we cannot accurately compare the two and assess historical changes in BC aerosols. Similarly, air quality concerns have stimulated the development of large data bases of atmospheric BC concentrations. (Los Angeles, for example, has >30,000 BC aerosol measurements). These data cannot be placed in a historic context, because they cannot be compared with pre-industrial BC concentrations measured in sediment cores.

(2) On geologic time scales, biomass burning is believed to be a significant source of atmospheric O<sub>2</sub> due to the creation and long-term storage of BC. Burial of BC in sediments prevents its decomposition, and without decomposition, an equivalent amount of O<sub>2</sub> accumulates in the atmosphere. Although individual research groups have reported that BC is between 5% and 50% of soil and sedimentary organic carbon, most of these measurements are made using methods unique to each laboratory, precluding the development of an accurate BC budget. Until we can assess how much BC is stored globally in soils and sediments, we cannot assess its carbon cycle significance.

(3) Biomass burning can be traced through sediment core BC concentrations, but method variation prevents comparison of data from differing sites. For example, we cannot determine differences between biomass burning histories measured in sediment cores in Central America and those measured in Asian loess deposits, if measurements at these two sites were made in separate laboratories. This has serious implications for our understanding of the history of human use of fire.

(4) Some parts of the BC spectrum are highly sorptive of contaminants like polycyclic aromatic hydrocarbons (PAH). Accurate measurements of BC would allow a better understanding of when PAH will sorb to sedimentary organic carbon.

### Committee Formed for Development of BC Reference Materials

The need for a BC methods intercomparison is critical. To address this need, a committee was formed during the 1999 Geochemical Society Meeting Goldschmidt Conference to develop representative and accessible BC reference materials for the entire environmental sciences Community. Chairs are Michael W.I. Schmidt (co-chair Europe; University of Zurich), and Caroline A. Masiello (co-chair North America; University of California-Santa Barbara and California Institute of Technology). Other members are William P. Ball (Johns Hopkins University, Maryland), Lloyd Currie (NIST, Maryland), Jan O. Skjemstad (CSIRO, Adelaide, Australia), and Dwight M. Smith (University of Denver, Colorado).

As a result of a first workshop hosted by the Max-Planck-Institute for Biogeochemistry in Jena, Germany, in March 2000, this committee issued preliminary recommendations for BC materials spanning the combustion continuum and appropriate to the needs of atmospheric scientists, soil scientists, oceanographers, and ecologists. This collection of standards was chosen carefully, keeping in mind the sometimes competing demands of many disciplines. Materials selected for this purpose are: (i) generally available, (ii) homogeneous, (iii) stable over a longer period of time, (iv) inexpensive to obtain, and (v) represent natural samples.

After issuing preliminary recommendations via a Web site, the BC steering committee actively solicited input from scientists and used this information to make a final set of recommendations of BC benchmark materials. This initiative was advertised through presentations on conferences and letters to interdisciplinary journals, such as *Environmental Science & Technology* in January 2001.

The final recommendations of the steering committee for Black Carbon Reference Materials were presented during the chemical reference materials workshop at the Friday Harbor Laboratories. Recommendations included (i) five matrices containing BC (soot, charcoal, aerosol, soil, and sediment); and (ii) five materials potentially creating BC during analysis, for use in detecting methodological artifacts (shale, melanoidin, natural organic matter, and coal). Recommended materials are mostly available from commercial sample distributors. As examples, coal samples can be obtained from the Argonne National Laboratory (Argonne, Illinois) or aerosols and sediments from the U.S. National Institute of Standards and Technology (NIST, Gaithersburg, Maryland). Others, such as the soil reference materials, were prepared and are distributed by individual members of the committee.

The complete list of recommendations is available at the Web site listed below. The development of this set of reference materials ensures long-term intra- and inter-laboratory data quality allowing comparative analyses between different analytical techniques and scientific approaches.

*Other Methodological Problems Remain*

Development of common reference materials resolves only some of the BC methodological uncertainties, however. Next, we must work together to analyze these materials. To achieve this goal, we propose a comparative analysis project using these reference materials to gauge how different methods can be used to interpret BC components in aerosol, soils, and sediments. The intention of the study is not to advocate a single technique; rather, such a comparative analysis will help immensely to better understand what is actually being determined by the different methods and how these results relate to one another. Researchers

of all disciplines are being actively solicited for this intercomparison. We are particularly interested in connecting with researchers who measure BC in the atmosphere with those who measure it in soils and sediments.

Participating research groups will be expected to make available all results and details of their methodology for eventual group publication. Distribution of standards will commence immediately, and all samples must be requested by January 2004. Data will be posted on a secure Web site by December 2004 for general discussion.

Further details can be found at the Web site: <http://www.geo.unizh.ch/phys/bc>.

The symposium, "New Approaches in Marine Organic Biogeochemistry," and the associated workshop were held 28–30 August 2003.

*Reference*

Schmidt, M. W. I., J. O. Skjemstad, C. I. Czimczik, B. Glaser, K. M. Prentice, Y. Gelin, and T. A. J. Kuhlbusch, Comparative analysis of black carbon in soils, *Global Biogeochemical Cycles*, 15, 163–167, 2001.

—MICHAEL W. I. SCHMIDT, University of Zurich, Switzerland; CAROLINE A. MASIELLO, University of California-Santa Barbara and California Institute of Technology, Pasadena; and JAN O. SKJEMSTAD, CSIRO Land and Water, Adelaide, Australia

# FORUM

## From A. D. Kirwan, Jr.: More on Anonymous Reviews

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Judging by the considerable and varied response it generated, the letter by Myrl Beck in the 1 July 2003 issue calling for signed reviews touched a sensitive point with many AGU members. As succinctly noted by Joseph Walder (*Eos*, 23 September 2003), all of the evidence cited for abuse of the review process is anecdotal. Yet, the sheer volume and variety of the responses was surprising, at least to me. This suggests there are some broad editorial issues that appropriate AGU oversight committees might address. To further this, I offer my perspective as a former editor of the *Journal of Geophysical Research* (Oceans and Atmospheres) and *Nonlinear Processes in Geophysics*, and as current editor of the *International Journal of Engineering Science*.

We, as scientists, are distinguished from the chattering professions by use of the scientific method and by peer review of our research to ensure compliance to this principle. Peer-reviewed literature is the primary vehicle for the transition of basic research to users. Because of this pivotal role, peer review must meet the Caesar's

wife criterion: it not only must be proper, it must appear proper. Signed reviews do not meet this standard.

Despite the honorable intentions of John Goff (*Eos*, 23 September 2003) and others, this practice is easily corrupted by the unscrupulous and self-serving. Moreover, signing reviews is hard to defend to some in the chattering professions who claim that peer review in science is just another scheme for cronies to protect their turf, propagate "the same old same old," and that every good review should not go unrewarded. There are ample avenues for reviewers and authors to communicate informally. AGU and other scientific society meetings are one example. I do not understand why an editor, journal, or AGU needs to officially sanction this through the review process. Moreover, it is not clear to me how signed reviews address the fundamental complaint that some reviews communicated to contributors are personally abusive and otherwise not constructive. Isn't it the editor's responsibility to review the reviews and communicate those parts that need to be addressed for further

consideration of any contribution? Perhaps the real message in the *Eos* dialogue is that some editors are not doing this and that the screening process for editors needs to be reviewed.

David Fisher's suggestion (*Eos*, 30 September 2003) that the review process should be double-blind was made to me by Bob Garrels many decades ago when I was whining about the trials and tribulations of being a *Journal of Geophysical Research* editor. This is worth considering, however. Fisher's last paragraph suggests that with a double-blind system that reviewers might be tempted to pierce the cloak of anonymity by speculating about author identities. Under the present system some authors speculate about reviewers identities, especially when they feel the reviews are abusive and unconstructive. Such speculation is counterproductive, as my anecdotal experience illustrates. I have been confronted and severely rebuked four times by senior investigators (two are members of the U.S. National Academy) for hatchet reviews of their submissions. In fact, the confrontations were the first I learned of each of these papers.

In these cases, the potential damage to the review process was limited. But if reviewers elect to speculate about author identities, similar mistakes might affect objectivity of their reviews and the review process would suffer.

## From D. J. Wesolowski: Preserving Anonymity in the Review Process

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To those scientists who haven't served as editors or associate editors of scientific journals, I can assure you that it's already too hard to find enough qualified reviewers willing to do the job without threatening them with exposure as well! So, if you want your papers to be published within a reasonable timeframe, you'll pretty much have to put up with anonymous reviews.

I've been an associate editor of *Geochimica et Cosmochimica Acta* for 11 years and an Editorial Board member of *Chemical Geology* for 4.

I think anonymous reviews are perfectly acceptable, but that the editor evaluating the reviews should always be identified, both to the authors and in the published manuscript. Reviewers should be permitted to request anonymity, but authors should always be instructed to specifically acknowledge in their manuscripts the contributions of those reviewers who do not request anonymity. This, in fact, might encourage more reviewers to identify themselves. I don't know about you, but it tickles me pink when I see my name in print!

The review process is intended to ensure that: the material is new or a useful summary

of previous work, the data and conclusions are correct or at least believable, proper credit is given to previous researchers, the subject matter and impact are appropriate for the target journal, and the presentation is readable and civil. How best to ensure these should be the only consideration in soliciting and evaluating reviews. Requiring reviewers to identify themselves to the authors is likely to force a more favorable review than is warranted or more likely result in the most suitable reviewers declining to comment. Anonymity certainly encourages vindictive or superficial reviews to be submitted, but it is the associate editor's job to weed these out. Furthermore, authors normally have the option of appealing a rejection to the editor-in-chief directly or options should always be made available.