Invited paper presented at the National Science Foundation Workshop on Aerosol Research, Santa Monica, CA, February 20, 1982

NEW TECHNIQUES FOR AEROSOL COMPOSITION MEASUREMENT: NEEDS AND OPPORTUNITIES IN AEROSOL SCIENCE AND ENGINEERING

Billy W. Loo

February 1982

TWO-WEEK LOAN COPY

This is a Library Circulating Copy which may be borrowed for two weeks. For a personal retention copy, call Tech. Info. Division, Ext. 6782

Prepared for the U.S. Department of Energy under Contract DE-AC03-76SF00098
DISCLAIMER

This document was prepared as an account of work sponsored by the United States Government. While this document is believed to contain correct information, neither the United States Government nor any agency thereof, nor the Regents of the University of California, nor any of their employees, makes any warranty, express or implied, or assumes any legal responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by its trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof, or the Regents of the University of California. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof or the Regents of the University of California.
NEW TECHNIQUES FOR AEROSOL COMPOSITION MEASUREMENT: 
NEEDS AND OPPORTUNITIES IN AEROSOL SCIENCE AND ENGINEERING

Billy W. Loo
Lawrence Berkeley Laboratory
University of California
Berkeley, California 94720 U.S.A.

February 1982

This work was supported by the Assistant Secretary for Environment, Office of Health and Environmental Research, Pollutant Characterization and Safety Research Division of the U.S. Department of Energy under Contract No. DE-AC03-76SF00098; and by the U.S. Environmental Protection Agency under interagency agreement number IAG AD-89-F-0-013-0.
NEW TECHNIQUES FOR AEROSOL COMPOSITION MEASUREMENT: NEEDS AND OPPORTUNITIES IN AEROSOL SCIENCE AND ENGINEERING

Billy W. Loo
Lawrence Berkeley Laboratory
University of California
Berkeley, California 94720 U.S.A.

Over the past decade, a growing number of techniques have been applied to the measurement of aerosol composition. These include the conventional wet chemical methods of titrimetry and colorimetry, electrochemical techniques using specific ion electrode and anodic stripping voltammetry. While atomic absorption, atomic emission, mass spectrometry and a variety of chromatographic techniques represent physical methods of the destructive type, non-destructive methods of analysis include neutron activation, charged particle activation and x-ray fluorescence analysis.

A number of techniques are especially suited for surface measurements. These include the electron spectroscopy for chemical analysis (ESCA), scanning Auger microprobe, secondary ion mass spectroscopy, and scanning electron/XRF microprobe. In particular, Rutherford backscattering techniques may be further exploited by using heavier particle beams to yield elemental depth profiles up to several thousand angstroms deep. A prerequisite for quantitative composition measurement is the determination of total aerosol mass. The limitations of traditional gravimetric analysis are gradually being overcome by the development of automated beta gauge, piezoelectric microbalance and various resonant frequency mechanical oscillators. There is, however, still room for refinement.

The relative merit of the aforementioned techniques may be viewed in terms of how likely they will be to fulfill modern requirements. For example, specific chemical speciation, fast time resolution and morphological information are important for aerosol dynamic studies. On the other hand, large-scale measurement methods are necessary to provide statistically meaningful data for studies such as source apportionment and receptor modeling. In addition, fast and non-destructive techniques are useful for screening of pertinent samples for detailed or more costly analysis.
The need for future development may therefore be identified when a given technique is considered under the following categories:

<table>
<thead>
<tr>
<th>ANALYTICAL COMPLETENESS</th>
<th>single element</th>
<th>multielement</th>
<th>chem. speciation</th>
</tr>
</thead>
<tbody>
<tr>
<td>TIME RESOLUTION</td>
<td>off-line</td>
<td>intermittent</td>
<td>continuous</td>
</tr>
<tr>
<td>SPEED AND RELIABILITY</td>
<td>sample preparation</td>
<td>manual</td>
<td>automated</td>
</tr>
<tr>
<td>SAMPLE INTEGRITY</td>
<td>artifact formation</td>
<td>destructive</td>
<td>non-destructive</td>
</tr>
<tr>
<td>SIZE SEGREGATION</td>
<td>mixed</td>
<td>sized</td>
<td>morphology</td>
</tr>
</tbody>
</table>

It would be generally desirable to improve or upgrade the positions of a technique towards the right hand column as much as it is possible. In the area of x-ray analysis for example, chemical speciation is becoming practical with the powder diffraction technique where speed is improved by combining a bent crystal with a position-sensitive detector. Similarly, the development of laboratory-scale intense x-ray source should free the extended x-ray absorption fine structure (EXAFS) analysis from being tied to a synchrotron radiation facility.

It is likely that more demanding future requirements are to be met by combining two complementary techniques in the tradition of GC/MS. These include some newer developments such as achieving sulfate and carbon speciations by combining regular elemental sulfur and carbon determinations with thermal volatility analysis. The combination of titrimetry with radio-nuclei labelling is another example. In the interest of making fast and inexpensive large-scale measurement, one or more indirect measurement(s) may be used such as the determination of graphitic carbon by laser light absorption and photo-acoustic techniques.

There has been a recent interest in developing capability for real time chemical composition and size analysis on individual aerosol particles. For example, the CAART instrument by Allen and Gould of AeroChem Research Labs, Inc. utilizes vacuum acceleration, flash vaporization and a quadrupole mass
filter tunable over mass fragments in the range from $2^\text{-} 500$ amu. A scheme proposed by R. Otto of LBL would employ electrostatic acceleration, impact ionization and time-of-flight mass spectrometry. This approach holds promise for real time single particle elemental and structural analysis with ppm sensitivity on particles in the range of $0.01$ to $1 \, \mu m$ and a throughput of up to $10^3$ particles per second.

Undoubtedly the identification of organic compounds will be a continuing challenge. Instrumentation related to mine safety must be perfected as coal utilization is expected to continue to increase. As cases of special nature, the old problem of asbestos measurement must be improved and the new problem of carbon fiber monitoring needs to be addressed as we face the coming decades of material revolution led by the success of carbon fiber composites.

As modern aerosol instrumentation reaches higher degrees of sophistication and complexity, the mounting barrier of technological transfer from research institutions to engineering enterprises requires special recognition and support. Proven methods which have reached a certain level of refinement—such as the automated sulfur monitor, beta gauge, and photon-excited XRF analyzers—are providing opportunities for commercialization, enabling wider utilization by the research and monitoring communities at large.

Acknowledgements


This work was supported by the U.S. Environmental Protection Agency under interagency agreement, number IAG AD-89-F-0-013-0, with the Assistant Secretary for Environment, Office of Health and Environmental Research, Pollutant Characterization and Safety Research Division of the U.S. Department of Energy under Contract No. DE-AC03-76SF00098.
This report was done with support from the Department of Energy. Any conclusions or opinions expressed in this report represent solely those of the author(s) and not necessarily those of The Regents of the University of California, the Lawrence Berkeley Laboratory or the Department of Energy.

Reference to a company or product name does not imply approval or recommendation of the product by the University of California or the U.S. Department of Energy to the exclusion of others that may be suitable.