



Project Summary

Identification of Compounds in Particulate Pollution by X-Ray Diffraction

J. V. Gilfrich and L. S. Birks

The ability of x-ray diffraction techniques (Seeman-Bohlin and Bragg-Brentano) to identify small amounts of material deposited on low-mass substrates was examined. The Seeman-Bohlin focusing camera had somewhat better resolution than the Bragg-Brentano diffractometer, but the Bragg-Brentano diffractometer was commercially available and was equipped with sophisticated computer programs for identifying the crystalline compounds.

Four commercial diffractometers with automated "search-match" capabilities were used to measure simulated and actual pollution particulate samples deposited on filter substrates and in bulk. These instruments are shown to be moderately efficient for identifying mixtures of reasonably-well-crystallized compounds in the simulated samples (average of 60% successful identification). While the actual samples produced poor results, the software that might improve the results is still being developed. X-ray diffraction shows potential as a method to characterize pollution particulate samples.

This Project Summary was developed by EPA's Environmental Sciences Research Laboratory, Research Triangle Park, NC, to announce key findings of the research project that is fully documented in a separate report of the same title (see Project Report ordering information at back).

Introduction

The identification of chemical species in air pollution particles is important to

health studies, air chemistry research, emission control strategies, and source apportionment modeling. Techniques are needed for the identification and measurement of these chemical species. X-ray diffraction (XRD) has been used routinely for many years in qualitative and, in some cases, quantitative analysis. Recently, XRD has been used to measure free silica in the work environment. However, XRD has not been used as a tool in routinely measure compounds in source emissions.

The purpose of the present study was to evaluate several XRD methods for use in source emissions measurements. In the first phase, we conducted laboratory experiments to compare two types of x-ray optics, Seeman-Bohlin (S-B) and Bragg-Brentano (B-B), used for determining the compounds present in particulate pollution samples collected on thin substrates. In the second phase, we evaluated commercial automated B-B XRD instruments equipped with "search-match" facilities to provide computer identification of the compounds present. To test the success of identification in both phases of this work, we used simulated samples in which mixtures of known compounds were deposited on thin substrates. In the second phase, we also examined bulk samples and actual pollution samples collected from source emissions

Results

Samples of $(\text{NH}_4)_2\text{SO}_4$, CaCO_3 and a mixture of the two were run on both the B-B diffractometer and the S-B camera. The S-B film was traced with a densitometer. In all cases, the

compounds were easily identified. However, the loadings were 1 mg/cm² higher than most actual collected samples. The XRD patterns matched the Joint Committee for Powder Diffraction Standards (JCPDS) cards, but line intensity differences were noted. For example, reversal of the strongest and second strongest lines occurred with some samples, which may be due to preferred orientation developed in the sample grinding. This problem may not exist in "real world" aerosol samples.

The samples used in Phase II were first x-rayed at the Naval Research Laboratory, then sent to four companies who make XRD equipment. The single compound samples with loading of about 200 µg/cm² caused no problems. However, low loaded samples and redeposited bulk fly ash samples produced poor patterns and, therefore, poor results. Simulated bulk samples made up from reagent chemicals produced fairly good patterns, and the four components were identified. None of the actual samples collected in the field produced patterns which could be identified satisfactorily.

Recommendations Use of Automated Diffraction

The modern automated diffractometer shows promise as a method to characterize particulate pollutants. The "search-match" program used with the diffractometer was only moderately successful in identifying crystallized compounds. An off-line computer program through which the data might be run for redundancy (assuming that the raw data can be retrieved from the automated instrument) might be desirable. One such program is available

from Interactive Sciences Corporation, which implements the NIH/EPA Chemical Information System (CIS). When the "search-match" software is further developed, these off-line programs will not be necessary.

Sample Holder

The sample holders on the commercial instruments were adaptations of the standard bulk sample holder, which had the filter mounted on the surface of the flat plate and the cavity containing the bulk powder behind the filter. The adopted sample holders were not ideally suited for filler samples. The most appropriate sample holder was used at Siemens, where a United States Steel Corporation design was available. This holder was designed: 1) to contain a 25 mm silver filter for the determination of free silica in airborne dust and 2) to be handled by an automatic sample changer.

J. V. Gilfrich and L. S. Birks are with the Naval Research Laboratory, Washington, DC 20375.

Kenneth T. Knapp is the EPA Project Officer (see below).

The complete report, entitled "Identification of Compounds in Particulate Pollution by X-Ray Diffraction," (Order No. PB 84-122 829; Cost: \$8.50, subject to change) will be available only from:

*National Technical Information Service
5285 Port Royal Road
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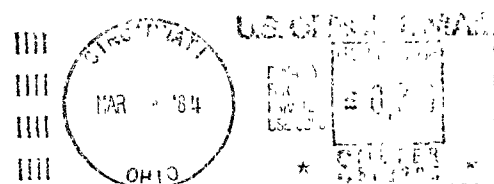
*The EPA Project Officer can be contacted at:
Environmental Sciences Research Laboratory
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