A. PURPOSE AND SCOPE

The NIOSH Manual of Analytical Methods (NMAM®) is a collection of air, biological, bioaerosol, and bulk analytical methods that NIOSH has evaluated. NMAM will be updated as methods are developed, evaluated or revised by NIOSH. NMAM is available in looseleaf hard copy form, without binders, from the U.S. Government Printing Office, Washington, DC 20402 (telephone 202/512-1800), and in electronic form from various software companies that have entered into publishing agreements with NIOSH (as of the time of this printing):

ChemSW, Inc.
(Formerly WindowChem)
420-F Executive Court North
Fairfield, CA 94585
Telephone:800/536-0404 or
707/864-0845
FAX: 707/864-2815
[Diskettes]

Canadian Centre for Occupational Health and Safety (CCOHS)
250 Main Street East
Hamilton, Ontario
Canada L8N 1H6
Telephone:800/668-4284 or
905/570-8094
FAX: 905/572-2206
[Diskettes]

American Conference of Governmental Industrial Hygienists (ACGIH)
1330 Kemper Meadow Drive
Cincinnati, OH 45240
Telephone: 513/742-2020
FAX: 513/742-3355
[CD-ROM with TLVs® & BEIs®]

John Wiley & Sons, Inc.
Distribution Center
1 Wiley Drive
Somerset, NJ 08876-1272
Telephone:800/CALL-WILEY
FAX: 908/302-2300
[CD-ROM with Patty's Industrial Hygiene and Toxicology, Version 2.0]

These methods are intended to promote accuracy, sensitivity, and specificity in industrial hygiene analyses. There will be situations where users of the NIOSH methods will need to modify them (e.g., to accommodate interfering compounds from the workplace, to take advantage of special capabilities of the laboratory, to make possible the analysis of a single sample for several contaminants). When method modifications are made, quality control data showing the reliability of the modified method should be obtained. For example:

- The volume of air sampled on solid sorbents should be reduced in cases of high vapor concentration or high humidity and, in some cases, may be increased if such concentrations are relatively low.
- The automation of sample work-up and measurement procedures usually requires some modification of the nonautomated procedure.
- Chromatographic conditions, including choice of column and detector, can be modified to eliminate interferences during measurement.

B. HOW TO USE NMAM

The methods are arranged **alphabetically** by method name, and some method names may refer to a group of related substances. It is possible for you to rearrange the methods **in order of method number** if you find this arrangement more convenient.

1. METHOD FINDER

The easiest and fastest way to locate a method is to refer to the **Method Finder** inside the front cover of the Manual. The Method Finder is an alphabetical listing of common chemical names and their associated methods with information on Compound, Method Number, Method Name, Sampling Rate, Minimum Volume, Maximum Volume, Extraction Solvent, Analytical Technique, and Sampler for quick reference.

2. METHOD NUMBERING SYSTEM

Substances having the same sampler, sample preparation and measurement technique may be grouped together in one method (note that this numbering system is unchanged from the Third Edition):

Method #	Substances
0001-0799	General air samples
0800-0999	Bioaerosols
1000-1999	Organic vapors on charcoal
2000-3499	Organic vapors on other solid sorbents
3500-3999	Organic vapors on other samplers (e.g., liquids, direct-reading)
4000-4999	Organic vapors on diffusive samplers
5000-5999	Organic aerosols
6000-6999	Inorganic gases
7000-7999	Inorganic aerosols
8000-8999	Biological samples
9000-9999	Bulk samples

3. INDEXES

There are three indexes in the back of the Manual which can be used in locating methods:

a. Fourth Edition Methods - An index of current NIOSH methods in order of Method number (pp. A-11 through A-16). [**NOTE:** The fourth edition uses the same numbering system as the third edition. This Index also lists the disposition of all third edition methods.]

b. First and Second Edition Method Numbers - An index of the first and second edition "P&CAM" and "S" methods, from which many of the current methods were derived. This index shows the disposition of all of these methods, whether or not they were revised into current methods (pp. A-17 through A-30).

c. Names and Synonyms - An alphabetical listing of all chemical names and synonyms used in the current methods, including Chemical Abstracts Service numbers ("yellow pages," pp. A-31 through A-62).

4. METHOD FORMAT

The methods consist of three major parts:

a. Front page The first page of each method concisely summarizes sampling and measurement parameters, and gives estimates of limit of detection, working range, overall and measurement precision, and interferences. References to other methods are given. New to the fourth edition are: Method Classification, NIOSH Registry of Toxic Effects of Chemical Substances (RTECS) number, and an estimate of method Accuracy. (See Figure 1)

b. Instructions The second page of each method begins with lists of required REAGENTS and EQUIPMENT. Please note that these reflect the conditions under which the methods were evaluated, and that there may be some latitude for variation. Commercial versions of the samplers may differ slightly from the methods. The user of the methods is responsible for assuring accuracy of the results (e.g., to determine that breakthrough and recovery are acceptable for each lot of samplers used). Typical tolerances are:

- Glass tubing used to contain solid sorbents: inside diameter is usually not critical within the range 4 to 6 mm; length should be sufficient to contain the specified mass of sorbent.

- Contents of sorbent tubes: mass of sorbent within ±10% of specification; separators of either glass wool or cleaned polyurethane foam unless otherwise indicated; sorbent mesh size of 20/40 unless sampling efficiency dictates otherwise. Filled sorbent tubes should be sealed to protect from contamination.

CHEMICAL NAME **METHOD #**

FORMULA Molecular Weight Chemical Abstracts Service # RTECS #

Method numbers are the same as those in the 3rd edition. **Evaluation** (Full, Partial, Unrated, N/A) is assigned as described in Method Classification of these "blue pages." **Issue date** reflects current version (e.g., August 15, 1994) and previous 3rd edition versions, if any.

OSHA : These exposure limit values are
NIOSH: those in effect at the time of
ACGIH: printing of the method.

PROPERTIES: Boiling/melting points, equilibrium vapor pressure, and density help determine the sample aerosol/vapor composition.

SYNONYMS: Common synonyms for the substance. These are all listed alphabetically in the **Index of Names and Synonyms** ("yellow pages" in this Manual).

SAMPLING

SAMPLER: Brief description of sampling EQUIPMENT

FLOW RATE: Acceptable sampling range, L/min

VOL-MIN: Minimum sample volume (L); corresponds to Limit of Quantitation (LOQ) at OSHA PEL

-MAX: Maximum sample volume (L) to avoid analyte breakthrough or overloading

BLANKS: Each set should have at least 2 field blanks, up to 10% of samples, plus 6 or more media blanks in the case of coated sorbents, impinger solutions, or other special samplers.

ACCURACY

Data are for experiments in which known atmospheres of the substance were generated and analyzed according to the method. Target accuracy is less than 25% difference from actual concentration over the range of the method.

MEASUREMENT

TECHNIQUE: The measurement technique used

ANALYTE: The chemical species actually measured

A summary of the measurement EQUIPMENT, SAMPLE PREPARATION, and MEASUREMENT steps appearing on the second page of the method is given here.

CALIBRATION: Summary of type of standards used

RANGE: Range of calibration standards to be used; from **LOQ** to **upper limit of measurement** (Note: More concentrated samples may be diluted in most cases to fall within this calibration range.)

ESTIMATED LOD: Limit of detection (background + 3)

PRECISION ($_r$): Experimental precision of spiked samplers

APPLICABILITY: The conditions under which the method is useful, including the working range in mg/m^3 (from the LOQ to the maximum sampler loading) for a stated air volume are given here.

INTERFERENCES: Compounds or conditions which are known to interfere in either sampling or measurement are listed.

OTHER METHODS: Methods from the 2nd edition ("P&CAM" and "S" methods) and current methods which are related to this one, as well as similar OSHA and literature methods are keyed to REFERENCES.

Figure 1. Layout of front page of methods

The SPECIAL PRECAUTIONS section gives safe practices to be observed during sampling and measurement. Next are the step-by-step instructions for

SAMPLING, SAMPLE PREPARATION, CALIBRATION AND QUALITY CONTROL, MEASUREMENT, and CALCULATIONS. Any lengthy instructions for sampler preparation or standardization of stock solutions appear in appendixes to the method. Nomenclature is consistent with the Glossary of Abbreviations, Definitions and Symbols (page A-3).

[NOTE: Additional general information relating to sampling and measurement is contained in the chapters on quality assurance and sampling (pp. 7 through 16 and 17 through 35 of these "Blue Pages").]

c. Supporting information Laboratory and field data relating to the method are summarized in the EVALUATION OF METHOD section and on the summary page, along with pertinent references.

5. METHOD CLASSIFICATION

Methods in the fourth edition of the NIOSH Manual of Analytical Methods are classified into Evaluation categories: Full, Partial, Unrated, and Not Applicable. Classification is based on the results of laboratory testing and evaluation criteria as described in Chapter E, Development and Evaluation of Methods.

The data from these evaluations are summarized in the EVALUATION OF METHOD section in each method. This section may also contain other corroborating data, e.g., collaborative testing, Proficiency Analytical Testing (PAT) data, or field data from NIOSH Sequences. For Partially Evaluated methods, this section will state which evaluation points were not tested, thus providing the user with information on which to make a reasonable judgment on the quality of the data obtained.

Evaluation: Full

Fully evaluated methods are methods that have been tested and have met all the factors of the NIOSH evaluation protocol as presented in Chapter E.

Evaluation: Partial

Partially evaluated methods are methods that have been subjected to some of the evaluation experiments but have not received a full evaluation (i.e., Short-term Method Development). These may also include methods that were fully tested but did not meet one or two of the evaluation criteria, e.g., some of the earlier-developed methods that do not meet the current ±25% accuracy criterion.

Evaluation: Unrated

Unrated methods have not been tested by NIOSH, but have been developed by a recognized independent source such as the Occupational Safety and Health Administration (OSHA).

Evaluation: N/A

The designation, Not Applicable (N/A), is applied to methods where no quantitative data are collected:

Procedures for sample collection only. The collected samples are analyzed subsequently by an appropriate analytical method, e.g., the Lead Wipe procedure (NMAM 9100) where the samples are analyzed by one of the three lead analysis methods.

Qualitative methods that indicate results as positive or negative, e.g., Lead spot test (NMAM 7700), or *Mycobacterium Tuberculosis* by PCR (NMAM 0900).

6. USER EXPERIENCE WITH THE METHODS

NIOSH seeks to make these methods useful in industrial hygiene analyses. Therefore, the experience of people using the methods is important to us. Suggestions for improvement and questions relating to this Manual are welcome and should be directed to the Manual Coordinator. A comment form has been included in the back of this Manual for this use; it can be mailed or FAXed.