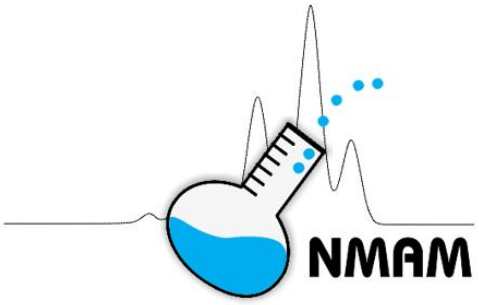


NIOSH Manual of Analytical Methods (NMAM), 5th Edition

Purpose, Scope and Use of the NIOSH Manual of Analytical Methods

by Kevin Ashley, Ph.D. and Paula Fey O'Connor, NIOSH

DEPARTMENT OF HEALTH AND HUMAN SERVICES
Centers for Disease Control and Prevention
National Institute for Occupational Safety and Health

1 Purpose and scope

The health of working people in myriad industries and occupations is potentially at risk through workplace exposure to airborne chemical and biological agents [Hathaway and Proctor 2004; Rose and Cohrssen 2011; Eduard et al. 2012; Jakubowski 2012]. Commonly it is the responsibility of occupational hygienists and often other public health professionals to determine the effectiveness of measures taken to minimize and control worker exposures to airborne toxins and toxicants, and this is normally achieved by monitoring workplace air quality [DiNardi 2003; Vincent 2007, 2012; Kulkarni et al. 2011]. Air monitoring is vital because inhalation is ordinarily the most likely route of exposure in occupational settings. Frequently other routes of workplace exposure, notably dermal contact with chemical and biological agents, must also be considered [Semple and Cherrie 2003; Brisson and Ashley 2011; Behroozy 2013]. Complementary biomonitoring methods are also often used to assess occupational exposures to toxic chemical compounds through measurement of specific analytes, e.g., metabolites and/or biomarkers, in body fluids (normally blood and urine) and tissues [Angerer and Greim 2006].

The *NIOSH Manual of Analytical Methods* (NMAM) is a compilation of analytical methods for air, biological, surface (including dermal) and bulk samples that have been evaluated and validated in consideration of their fitness for purpose for workplace exposure monitoring [NIOSH 1995]. NIOSH sampling and analytical methods are intended to promote accuracy, sensitivity, and specificity in industrial hygiene analyses and related applications. NMAM, which is published online (available at: www.cdc.gov/niosh/nmam), is constantly updated as new methods are developed and validated and as revised methods are evaluated and their performance verified. The methods published in NMAM are relied upon by authoritative bodies such as accrediting organizations and regulatory agencies. Besides sampling and analytical methods, NMAM also includes chapters on quality assurance, portable instrumentation, measurement of fibers, aerosol sampler design, and other guidance on specific areas of interest.

Often there are situations during use where certain NIOSH methods may require modification, for instance, to accommodate interfering compounds from a particular workplace, to take advantage of unique laboratory capabilities, to make use of equivalent sample preparation or analysis techniques, or to make possible the analysis of a single sample for multiple contaminants. When method modifications are made, quality control data demonstrating the reliability of the modified method must be obtained, recorded and reported. Examples where method modifications might be required include the following:

- 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.

- Automation of sample preparation and measurement procedures usually requires modification of the manual procedure on which the modified method is based.

- Chromatographic conditions, including choice of column and detector, can be modified to eliminate interferences or increase sensitivity during measurement.

- Acid mixtures used for sample dissolutions for elemental analysis may require modification for certain sample matrices that are difficult to dissolve.

For the measurement of each analyte or group of analytes of concern in workplace environmental samples or in biological specimens obtained from workers, it is desired to produce sampling and analytical methods that will meet the needs of field investigators (e.g., industrial hygienists, control engineers or occupational physicians) as well as laboratory personnel (e.g., analytical chemists, biochemists, epidemiologists or toxicologists). Many NIOSH methods are developed in parallel with related voluntary consensus standards [Ashley 2015]. The ultimate goal of the formalized NIOSH method development, evaluation and validation protocol is to make available sampling and analytical methods for applications in the occupational hygiene arena that are fit for purpose, analytically rigorous, and adequately ruggedized.

2 How to use NMAM

NIOSH methods are grouped alphabetically by method name, and some method names may refer to a group of related substances. It is also possible to locate methods through their arrangement by method number. Methods for particular analytes or groups of analytes can additionally be accessed by searching their Chemical Abstracts Service (CAS) number(s) through the online link.

a. Locating a NIOSH method

Often the easiest and fastest way to locate a method is to refer to the online method index, which contains an alphabetical listing of analytes and listing by method number. Each method's cover page contains information on alternate chemical names and information on: Compound(s), Method Number, Method Name, Sampling Rate, Minimum Volume, Maximum Volume, Reagents, Analytical Technique and Sampler (for a quick reference). It is also possible to search electronically by method number and/or CAS number (if known).

b. Method numbering system

The general NMAM method numbering system is outlined in the table below. Substances having the same sampling device, sample preparation procedure and measurement technique are often grouped together in one method (e.g., organic vapors; metals).

Method No.	Substances
0001-0799	General air samples
0800-0999	Bioaerosols
1000-1999	Organic vapors on charcoal sorbents
2000-3499	Organic vapors on other solid sorbents
3500-3999	Organic vapors on other samplers (e.g., liquids; direct-reading instruments)
4000-4999	Organic vapors on diffusive samplers
5000-5999	Organic aerosols
6000-6999	Inorganic gases and vapors
7000-7999	Inorganic aerosols
8000-8999	Biological samples
9000-9999	Bulk samples; wipe samples

c. Indexes and Appendixes

Within the NMAM website there is an online link to indexes that can be used to locate methods published in previous editions of the Manual:

1) Fourth Edition Methods

An index of fourth edition methods in order of method number. Note that the same method numbering system is used for third, fourth and fifth edition NIOSH methods. Also denoted is the current disposition of historical or discontinued methods.

2) First and Second Edition Method Numbers

An index of the first and second edition "P&CAM" and "S" methods, from which many of the subsequent methods were derived. This index shows the disposition of all of these earlier methods, whether they were later revised / updated or not.

3) Names and Synonyms

An alphabetical listing of chemical names and synonyms used in current (and many previous edition) methods, including CAS numbers.

An online "Appendixes" link is also available for obtaining unit equivalents or for carrying out air concentration calculations for comparisons to Occupational Safety and Health Administration (OSHA) standards.

d. Method format

NIOSH methods consist of three major parts:

1) 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 relevant methods are given. Also provided are Method Classification, NIOSH Registry of Toxic Effects of Chemical Substances (RTECS) number, and an estimate of method accuracy (see Figure 1).

2) 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 still be some latitude for variation. The user of the methods is responsible for assuring the accuracy of the results (e.g., to determine that breakthrough and recovery are acceptable for each lot of samplers used). For example, typical tolerances for sorbent tubes are illustrated:

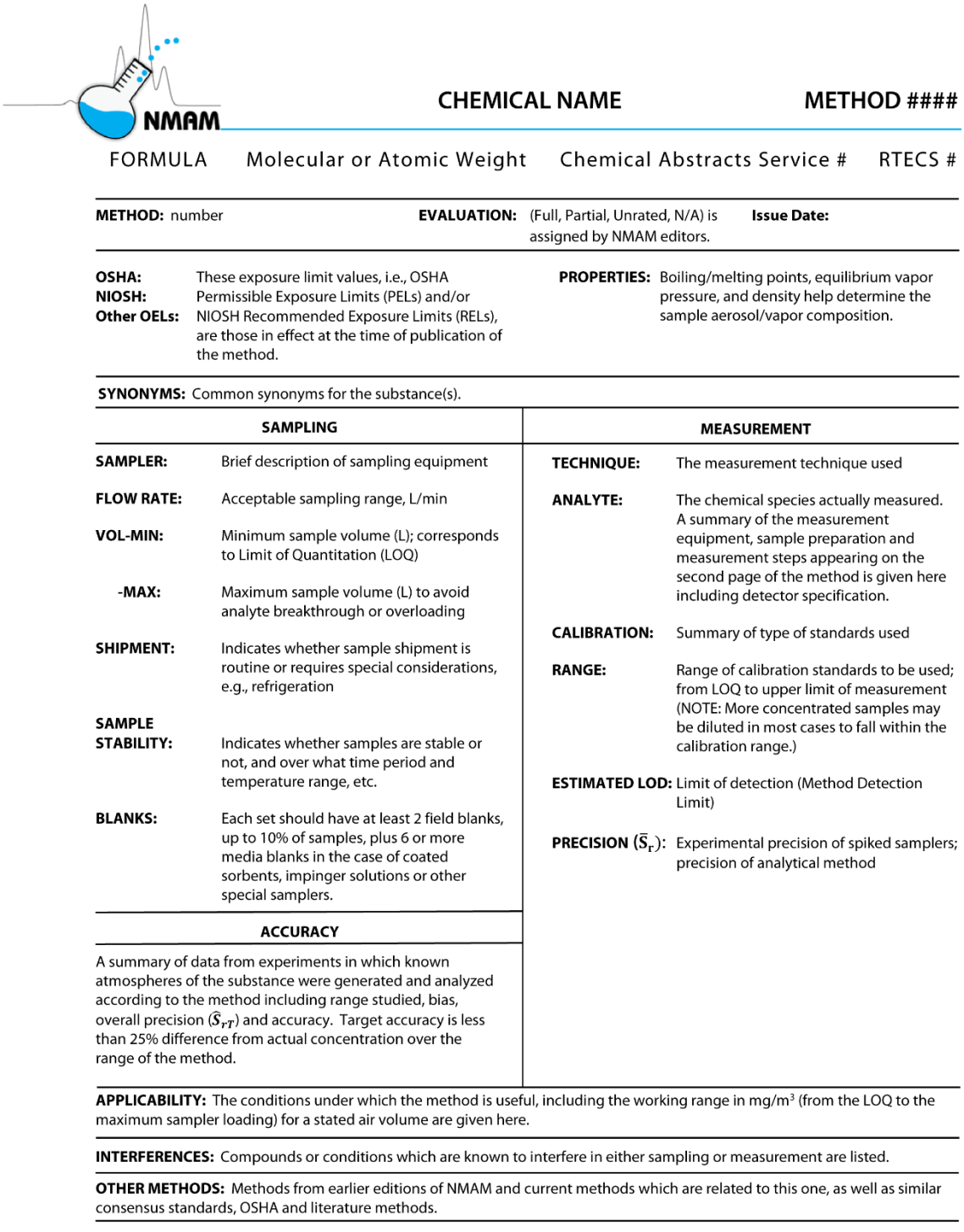
- Glass tubing used to contain solid sorbents: Inside diameter is usually not critical within the range of 4 to 6 mm; length should be sufficient to contain the specified mass of sorbent.

- 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 them from contamination.

The Special Precautions section gives guidance on safe practices to be observed during sampling, sampler preparation 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 and standardization of stock solutions appear in method appendixes. Nomenclature is consistent with the NMAM Glossary (chapter) of Abbreviations, Definitions and Symbols. (Note that additional general information relating to sampling and measurement is contained in other NMAM chapters.)

3) 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.



NMAM

CHEMICAL NAME METHOD ####

FORMULA Molecular or Atomic Weight Chemical Abstracts Service # RTECS #

METHOD: number **EVALUATION:** (Full, Partial, Unrated, N/A) is assigned by NMAM editors. **Issue Date:**

OSHA: These exposure limit values, i.e., OSHA
NIOSH: Permissible Exposure Limits (PELs) and/or
Other OELs: NIOSH Recommended Exposure Limits (RELs), are those in effect at the time of publication 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(s).

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)

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

SHIPMENT: Indicates whether sample shipment is routine or requires special considerations, e.g., refrigeration

SAMPLE STABILITY: Indicates whether samples are stable or not, and over what time period and temperature range, etc.

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

A summary of data from experiments in which known atmospheres of the substance were generated and analyzed according to the method including range studied, bias, overall precision ($\hat{S}_{rT}$) and accuracy. 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 including detector specification.

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 the calibration range.)

ESTIMATED LOD: Limit of detection (Method Detection Limit)

PRECISION ($\bar{S}_r$): Experimental precision of spiked samplers; precision of analytical method

APPLICABILITY: The conditions under which the method is useful, including the working range in mg/m³ (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 earlier editions of NMAM and current methods which are related to this one, as well as similar consensus standards, OSHA and literature methods.

NIOSH Manual of Analytical Methods (NMAM), Fifth Edition

Figure 1. Layout of front page of NIOSH methods

e. Method classification

Methods in previous (fourth) edition of NMAM 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 a NIOSH guidelines document [NIOSH 1995] and in Chapter ME (Development and Evaluation of Methods). Most methods in the fifth edition are classified as 'fully validated.'

The performance 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., results from collaborative testing, Proficiency Analytical Testing (PAT) data, or field data from NIOSH studies. 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 those that have been tested and found to have met all of the factors of the NIOSH evaluation protocol [NIOSH, 1995].

Evaluation – Partial: Partially evaluated methods are those that have been subjected to some of the evaluation experiments but have not received a full evaluation (e.g., short-term method development). These may also include methods that were fully tested but did not meet one or two of the evaluation criteria specified in the NIOSH protocol [NIOSH, 1995]; for example, 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 may have been developed by a recognized independent source such as OSHA.

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

- Procedures for sample collection only. The collected samples are analyzed subsequently by an appropriate analytical method.

- Qualitative methods that indicate results as a positive or negative (or inconclusive).

f. User experience with NIOSH methods

NIOSH strives to make the methods published in NMAM useful and fit for purpose in industrial hygiene analyses. Therefore, feedback on the experiences of people using the methods is important to us. Suggestions for improvement and questions relating to

NMAM are welcome and should be directed to the editors of the Manual. Their contact information is provided on the NMAM webpage.

Disclaimer

Mention of any company or product does not constitute endorsement by NIOSH. In addition, citations to websites external to NIOSH do not constitute NIOSH endorsement of the sponsoring organizations or their programs or products. Furthermore, NIOSH is not responsible for the content of these websites. All web addresses referenced in this document were accessible as of the publication date.

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