

INDUSTRIAL HYGIENE SAMPLING GUIDE
for
COMPREHENSIVE INDUSTRIAL HYGIENE LABORATORIES
(CIHLs)

NAVY AND MARINE CORPS PUBLIC HEALTH CENTER
620 JOHN PAUL JONES CIRCLE
SUITE 1100
PORTSMOUTH, VIRGINIA 23708-2103

JANUARY 2014

INDUSTRIAL HYGIENE SAMPLING GUIDE
for
NAVY COMPREHENSIVE INDUSTRIAL HYGIENE LABORATORIES (CIHLs)

TABLE OF CONTENTS

SECTION	PAGE	SECTION	PAGE
Introduction	1	Special Sampling & Analysis	14
Laboratory Organization	2	Bulk Sample Submissions	14
General Policy	3	Chromium & Chromates	14
Specific Policies	3	Environmental Lead Samples	15
Policy on SOPs & LOPs	3	Fiber Counts & Asbestos ID	15
Policy on Sample Acceptance	3	Polychlorinatedbiphenyls (PCB)	16
Policy on Methods	4	Silica (Crystalline Silica)	16
Policy on COV	4	Conversion Factors	16
Policy on Analytical Results	4	Zn to Zinc Oxide	16
Policy on Limit of Quantitation	5	V to Vanadium Pentoxide	16
Policy on Spiked Samples	5	Fe to Iron Oxide	17
Policy on Blank Media	6	Cr(VI) to Chromates (as CrO ₃)	17
Policy on Use of Disclaimers	7	Sources for Analytical Supplies	17
Quality Assurance	8	Manuals	17
Lab Analytical Equipment	8	Filters and Sorbent Tubes	18
Submission Requirements	8	Passive Monitors	19
Sample Submission Form	8	Printed Sample Number Labels	19
Biological Samples	8	Sample Bottles, Vials, Supplies	19
Sampling Requirements	10	Dust Wipe Media	19
Sample Packaging & Shipping	10	Abbreviations	20
Sample Turnaround Times	11		
Sample Compatibility	12		
Laboratory Specific Samples	13		
Routine Biological Samples	13		
Blood Lead & ZPP	13		
Urine Mercury	13		

INDUSTRIAL HYGIENE SAMPLING GUIDE
for
NAVY COMPREHENSIVE INDUSTRIAL HYGIENE LABORATORIES (CIHLs)

INTRODUCTION

This guide contains a compilation of sampling and analytical method recommendations for specific chemicals which the Navy has in-house analytical capability through its two Comprehensive Industrial Hygiene Laboratories (CIHLs) located in Norfolk, VA and San Diego, CA. The CIHLs are detachments of the Navy and Marine Corps Public Health Center (NAVMCPUBHLHCEN). This guide is a concise reference for the industrial hygienist in the proper submission of industrial hygiene, environmental, bulk and biological samples. This guide lists the analyte or substance, the Chemical Abstracts Service Registry Number (CAS #) for the substance, analytical method used by the laboratory in performing the analysis, method's coefficient of variation (CV), reporting limit (RL), sampling media, recommended air volume, sampling rate, special instructions for the industrial hygienist submitting the sample, and location of the CIHL which can analyze the sample. Customers should submit samples to the CIHL located nearest them or most convenient to them. If that CIHL does not have the desired analytical capability, call the CIHL to verify that the capability does not exist there. The CIHL will work with the requestor to obtain the required service by confirming that the other CIHL can do the analysis, by sending the sample out for contract analysis, or by helping the customer identify a laboratory that the customer can contract with directly for the analysis. Since both CIHLs are constantly updating their analytical services, always check with the closest CIHL first.

Each CIHL welcomes comments and suggestions regarding its services, additional method development requirements, alternate sampling techniques, and any other input. All questions regarding laboratory service/capability should be addressed to the CIHL which provides the service. Working hours are generally 0730 to 1600 hours Monday through Friday. If the CIHL can't be reached or additional information is required, please contact the CIHL Program Manager at NAVMCPUBHLHCEN, to which both laboratories report, using the contact information below. All comments concerning CIHL program management and additions, corrections and changes to this guide, should be addressed to:

Commanding Officer
Attn: CIHL Program Manager
Navy and Marine Corps Public Health Center
620 John Paul Jones Circle, STE 1100
Portsmouth, VA 23708-2103

Tel: (757) 953-0757
DSN: 377-0757
FAX: (757) 953-0689

<http://www.med.navy.mil/sites/nmcphc/Pages/Home.aspx>

LABORATORY ORGANIZATION

The mission of the NAVMCPUBHLTHCEN is to be the navy and Marine Corps center for public health services, and to provide leadership and expertise to ensure mission readiness through disease prevention and health promotion in the support of the National Military Strategy. The CIHLs' specialized qualitative and quantitative analyses of samples support that mission by providing objective data for occupational health and industrial hygiene investigations, assessments, recommendations, and risk management.

On 1 October 1989, all Navy medical department industrial hygiene laboratories, then named Consolidated Industrial Hygiene Laboratories, became part of the Navy Environmental Health Center. The CIHLs are now renamed Comprehensive Industrial Hygiene Laboratories and the parent command name has changed to Navy and Marine Corps Public Health Center. The following information for the two CIHLs is provided:

1 - Navy and Marine Corps Public Health Center
Comprehensive Industrial Hygiene Laboratory (CIHL) Detachment West
3235 Albacore Alley
San Diego, CA 92136-5199

Laboratory Director
Phone: (619) 556-1427 DSN: 526-1427
FAX: (619) 556-1492

CIHL Det West Phone: (619) 556-7070
DSN: 526-7070 FAX: (619) 556-1492

2 - Navy and Marine Corps Public Health Center
Comprehensive Industrial Hygiene Laboratory (CIHL) Detachment East
1285 West D Street, Bldg U-238
Norfolk, VA 23511-3394

Laboratory Director
Phone: (757) 953-6562; DSN: 377-6562
FAX: (757) 953-7213

CIHL Det East Phone: (757) 953-6622
DSN: 377-6622 FAX: (757) 953-7213

GENERAL POLICY

The CIHLs provide analytical support services for samples submitted through the BUMED Industrial Hygiene Program Offices. The analytical services available at the CIHLs are primarily designed for quantitative analyses of occupational health samples and selected environmental samples.

SPECIFIC POLICIES

POLICY ON STANDARD OPERATING PROCEDURES AND LOCAL OPERATING PROCEDURES

Standardization among the laboratories is an essential part of the CIHL program. The written CIHL Standard Operating Procedures (SOPs) document the quality assurance guidance for operation and standardization between the CIHLs. Based on these SOPs, each CIHL develops its own Local Operating Procedures (LOPs). The LOP (which implements instructions and any laboratory procedural changes) contains current procedures in use at each laboratory. Historical records are kept of the dates when procedures are implemented and taken out of service.

POLICY ON SAMPLE ACCEPTANCE/REJECTION

Sample submissions must be accompanied by a completed Form NMCPHC 5100/13 or 5100/14 (Note: Every information category must be completed.) when submitted by the industrial hygienist. Samples must be properly preserved, as appropriate, packaged and shipped by the proper method. Refer to the Sample Packaging and Shipping Requirements Section. Properly documented, preserved, packaged, and shipped samples will be accepted by the CIHL and analyzed as routine unless the submission is marked "URGENT". Urgent samples must arrive by a one- or two-day express shipping service.

If samples are taken incorrectly and/or incompletely documented, every effort will be made by the CIHL to obtain the necessary information to convert the invalid sample into a valid sample. Samples will only be returned to the customer when requested by the customer. Documentation may be returned for correct completion; however, the samples will remain at the laboratory.

In order to assure a quick laboratory turnaround time, please ensure samples are taken according to this guide, shipped appropriately, and the submission Forms are correct and complete.

When samples are received and are not able to be corrected for validity (e.g., fiber counts submitted on PVC filters), the customer will be notified by phone, fax, e-mail or letter in order to determine the disposition of the sample(s). Such samples will be returned to the customer upon the customer's request.

POLICY ON ANALYTICAL METHODS

Rarely are analytical methods either complete or fully comprehensive to preclude some interpretation, change or modification of the method. (NOTE: This is the reason for the CIHL requirement that a LOP manual be available at each CIHL.) Most methods are single analyte methods while most samples contain multiple contaminants. Most analytical methods used by the CIHLs are taken from the analytical methods published by the National Institute for Occupational Safety and Health (NIOSH) or the Occupational Safety and Health Administration (OSHA). Since OSHA does not require specific analytical methods, unless stated in a stressor-specific standard, any method (e.g., ASTM, scientific literature, journal articles, etc.) can be used as long as it meets NIOSH criteria of accuracy within $\pm 25\%$ at the 95% confidence level. All NIOSH and OSHA methods in this document are potentially "modified methods". The modification is necessary because of the variance in: analytical columns (types, sizes); desorbing agents; digesting acids/bases; analytical equipment conditions (temperatures, pressures, flow rates). All these modified methods are evaluated and validated for the NIOSH accuracy of $\pm 25\%$ at the 95% confidence level by each CIHL, and the method changes are documented as modifications.

POLICY ON COEFFICIENTS OF VARIATION

Randomly distributed errors occurring in industrial hygiene sampling are normal and are commonly included in analytical reports as the coefficient of variation (CV). The CV is a useful index for differentiating the true mean of known data points and laboratory reported data. The total CV (CV_T) of the sampling and analytical method is based on a statistical standard normal deviation for 95% two-sided confidence limits. The statistical decision techniques developed by NIOSH and OSHA are implemented in the CIHLs' use of the CVs. Therefore, since industrial hygienists will seldom receive true exposure results from the labs due to sampling and analytical variations, the CVs for each analyte are reported in the tables so 95% confidence levels may be calculated by the industrial hygienist. Our CIHLs are capable of reporting the same or lower CVs annotated in the official analytical method, the values of which are noted in the Laboratory Sampling Guide Table.

For Time-Weighted Average (TWA) sampling, the CV criteria originally adopted by NIOSH of $\pm 25\%$ accuracy, with 95% confidence limits, is usually cited, but accuracy specifications may vary from one standard to the next. Substances which have Permissible Exposure Limits (PELs), but for which no specific standard has been promulgated, do not have specific accuracy requirements. For these substances, the CIHLs consider the method acceptable (e.g., OSHA, NIOSH, literature cited methods) if it can meet the $\pm 25\%$ accuracy requirement with 95% confidence.

POLICY ON REPORTING ANALYTICAL RESULTS

The CIHLs are reporting air samples results in "total mass of contaminant per sample" because of the confusion in the interpretation when samples are reported in mg/M^3 . Some customers

were erroneously using the reported value as the TWA when the sample did not represent an eight-hour exposure. Blanks submitted with the samples are also reported in "total mass of contaminant per sample." The CIHL will notify the customer when the blank values are elevated more than normal. It is now the responsibility of the customer to take the analytical results and compute TWAs as necessary. If you need assistance, please contact your local industrial hygienist or the CIHL.

POLICY ON LIMIT OF QUANTIFICATION

It is not unusual for the Limit of Quantification (LOQ) of an analyte to vary from day to day. Instrumental conditions and environments vary day to day and this variation often affects the LOQ. If you envision detection levels (e.g., a short duration sample) to be a problem, please contact the CIHL performing the analyses, preferably before collecting the sample. Often the laboratory can modify a method to increase the sensitivity and selectivity; however, the analyst must know your requirements before the analyses are performed using the standard analytical method.

POLICY ON SPIKED SAMPLES OR FIELD-SUBMITTED QC SAMPLES

The CIHLs are required by their accreditation through the American Industrial Hygiene Association (AIHA) to have a comprehensive quality assurance/quality control (QA/QC) program which involves, at a minimum:

- A written QA/QC plan,
- A designated Quality Assurance/Quality Control Coordinator (QA/QCC) responsible for the QA/QC program,
- Participation in the Proficiency in Analytical Testing (PAT) program for all categories of analytes performed for the customer,
- Records which demonstrate the routine introduction of control samples of known content along with samples for analysis,
- Records which demonstrate routine checks, calibrations, maintenance of equipment and instruments are performed to ensure adequate performance,
- Quality control data stored in an accessible manner,
- Routine checks made of procedures and reagents, and
- Inter-laboratory, as well as intra-laboratory, QC.

Occasionally the customer may feel uncomfortable with laboratory results and therefore "challenge" the QA/QC program of the laboratory by submitting blind QA/QC samples to the laboratory.

The only recommended method of challenging the laboratory is purchasing past PAT rounds from the AIHA and submitting these as controlled spikes. Literature articles have proven that side by side duplicate monitoring very rarely produces duplicate samples. The use of a duplicate sampling manifold will not produce duplicate samples; however this method of sampling is

superior to the use of two independent sampling systems side by side. Contact the AIHA (phone number (703) 849-8888 or FAX (703) 207-3561) for the purchase of PAT metals, solvents, fibers and crystalline free silica samples. PLEASE NOTIFY THE LABORATORY ONCE YOU RECEIVE THE RESULTS OF YOUR QC SAMPLE SO THE LABORATORY MAY DOCUMENT ITS QA/QC PROGRAM TO INCLUDE THIS BLIND QC SAMPLE. THIS SAMPLE THEN WILL BE IDENTIFIED IN THE CIHL DATABASE AS A TRUE QC BLIND. Also the laboratory will recharacterize the results for this sample in the Laboratory Information Management System (LIMS) database if you have identified the sample as a field sample (e.g., assigned fictitious breathing zone sample information).

Please realize that if there is a quality problem with the CIHLs, the labs want to be the first to know so they can identify and resolve the problem. The labs welcome and expect feedback from the customers.

POLICY ON BLANK MEDIA

The CIHLs follow NIOSH policy for submitting blanks and ask the customer to submit two (2) blanks with each batch of samples. One should be a "Field Blank". A "Field Blank" is an unopened cassette/tube/etc. taken to the work site where the sampling will be performed. The blank cassette/tube/ etc. is then opened on site and immediately closed, sealed and labeled. NO air is pumped through the "Field Blank". This "Field Blank" is used to check for contamination due to sampling process and the background contamination due to the work site. The second blank submitted should be a "Media Blank". A "Media Blank" is an unopened cassette/tube/etc. from the same lot number as the sampling cassettes that is NEVER opened. This blank is sealed, labeled and sent off to the laboratory with the "Field Blank" and the samples. The "Media Blank" is used to check the media analyte background levels and also as a check for laboratory reagents and methodology. Note that these blanks should be labeled with field sample ID numbers and listed on the sample request form as the type of blank that they are along with the samples.

If you are sampling for different types of analytes in the same operation please submit a complete set of blanks for each type of analyte. For example if you are sampling for cellosolves and toluene, even though both are collected on charcoal tubes the samples are processed differently so you should submit two sets of charcoal tube blanks with your request. Please consult the CIHL if you are uncertain whether more than one set of blanks may be needed.

Blanks are treated and analyzed the same way as samples. If the total amount of analyte found exceeds the Limit of Quantification (LOQ) for that analyte, then the results are reported as the total amount of analyte per blank (e.g., 0.7 µg of Cd). If the amount found is less than the LOQ then the result is reported as less than the LOQ (e.g., < 0.5 µg of Cd). Note that it is not unusual for the LOQs to vary depending on the instrument used for the analysis, the methodology and other experimental factors. If you are concerned about potential problems with the LOQ because of short sampling times, please contact the CIHLs. There are ways they can modify the procedure which will increase sensitivity and lower the LOQ but they must know your requirements IN ADVANCE.

The CIHLs also follow NIOSH policy concerning the blank correction. In short, blank value(s) are NOT SUBTRACTED from sample values unless stated otherwise on the Laboratory Report. If the client is concerned about high blank values (i.e., possible contamination) they should contact the laboratory for assistance in determining the correct course of action. However it is ultimately the responsibility of the client to decide if the sample values should be blank corrected.

POLICY ON USE OF DISCLAIMERS

The CIHLs recognize that there are field situations when samples cannot be taken according to required sampling methods (e.g., "a once in a lifetime opportunity sample"). In such cases, the laboratory will usually analyze the sample if taken on appropriate sampling media, and report a result possibly accompanied by one of the disclaimer statement listed below:

- 1- **INSUFFICIENT AIR VOLUME** - The air volume is less than the amount recommended for this method. Consequently the coefficient of variation (CV) published for the method may not apply. Professional judgment should be used in the interpretation of results.
- 2- **QUESTIONABLE FLOW RATE** - The flow rate differs from the recommended method's rate. Therefore, professional judgment should be used in the interpretation of results.
- 3- **INCORRECT SAMPLING MEDIUM** - The sample media is not one currently recommended by NIOSH, OSHA or the latest edition of NAVMCPUBHLTHCEN's Industrial Hygiene Sampling Guide for CIHLs. Therefore, professional judgment must be used in the interpretation of results.
- 4- **NON-NIOSH/NON-OSHA METHOD** - The analytical method is not one currently recommended by NIOSH, OSHA or the latest edition of NAVMCPUBHLTHCEN's Industrial Hygiene Sampling Guide for CIHLs. Therefore, professional judgment must be used in the interpretation of results.
- 5- **SHIPPING ERROR** - Bulk samples were received in the same shipping package as air samples for the same contaminant. Samples were not preserved or did not arrive at the laboratory within the recommended shipping time. Therefore, professional judgment must be used in the interpretation of results.
- 6- **BLANK(s) NOT SUBMITTED** – No field blank was submitted as required by the sampling and analytical method. Therefore, professional judgment must be used in the interpretation of results.
- 7- **OTHER** - Other laboratory specific comments requiring a disclaimer.

QUALITY ASSURANCE (QA)

The CIHLs are accredited by the American Industrial Hygiene Association (AIHA) which requires participation in all applicable round robin testing programs. The AIHA accreditation program specifies operational guidelines for maintaining satisfactory performance, including qualified personnel, proficiency in analytical testing, adequate facilities, quality controls, equipment maintenance, documentation and site audits. In addition to this accreditation program, CIHLs participate in several quality control programs for monitoring daily performance. Both internal and external quality control samples are analyzed to assure accuracy and precision of results. Some of the QA techniques used include replicate analyses, recycles, spiked controls, commercial reference controls, daily instrument calibration, control charts, regression analyses, data review, reagent and media blanks. Each CIHL maintains its own quality control manual, which gives extensive description of the quality assurance program. Please address specific QA questions to the CIHL performing the analytical work.

LAB ANALYTICAL EQUIPMENT

The primary analytical instrumentation in each laboratory consists of gas chromatographs, atomic absorption spectrophotometers (graphite furnace), ultraviolet/visible spectrophotometers, high performance liquid chromatographs, ion specific electrode meters, ion chromatographs, microbalances and microscopes (both phase contrast and polarizing light), inductively coupled plasma (ICP) spectrometers, gas chromatograph/mass detectors, and an X-ray diffractometer are located in the CIHL laboratories. A few instruments, such as the X-ray diffractometer, are only present in one of the CIHLs.

SUBMISSION REQUIREMENTS

SAMPLE SUBMISSION FORM

Air samples must be submitted on Navy and Marine Corps Public Health Center Forms NMCPHC 5100/13 and 5100/14. Bulk samples must be submitted on Navy and Marine Corps Public Health Center Form NMCPHC 5100/16. Copies of these forms and instructions for completion are provided in the Industrial Hygiene Field Operations Manual (available on the NMCPHC website) or may be requested from the CIHLs.

BIOLOGICAL SAMPLES

Biological samples must be submitted with sample submission documentation containing at least the following:

- 1- Name of medical treatment facility submitting samples
- 2- Name of person submitting samples

- 3- Date of submission
- 4- Name of person sampled (i.e., patient first and last name)
- 5- Sample number [Complete Social Security Number (SSN) of the patient and the CHCS number].
- 6- Age of person sampled (required for blood lead samples only)
- 7- Date sample was collected
- 8- Name of test requested.
- 9- Occupational code of patient
- 10- Patient's command UIC.

Because most medical treatment facilities use a computerized system for medical records, biological samples submitted for blood lead/ZPP and urine mercury may be submitted with a computerized transmittal list. Please refer to section below entitled "ROUTINE BIOLOGICAL SAMPLES" for specific guidance on this transmittal list.

Biological samples for blood lead/ZPP and urine mercury may be submitted on Standard Form 557 (Miscellaneous Chemistry Request). The request must be signed and dated by the submitting MD, RN, PA, or Hospital Corps person. All biological samples must be properly packaged and labeled in accordance with Navy, Federal, State and local regulations. It is recommended that a commercial express package delivery service be used to transport samples to the CIHL (i.e. FedEx). Please contact the carrier for their shipping and labeling requirements. In general, the samples must be placed in a sealed, waterproof primary container that contains absorbent material sufficient to absorb all possible leakage. The primary container must then be placed in a sealed, secondary container. The secondary container can then be placed in an outer container for shipment. All containers should be adequately cushioned so the samples do not become loose and move during shipment. Freezer ice packs should be used to keep the samples cold. Do not use ice or dry ice, and do not freeze the samples. An Etiological Agent/Biomedical Material label must be affixed to the outside of the outer shipping container.

When samples are sent by U.S. Postal Service (USPS), Express Mail Delivery is required. Each package of samples using USPS cannot contain more than a total of 50 milliliters (1.7 ounces) of sample. If more than 50 milliliters of samples (e.g., approximately 7 blood lead samples) are sent to the lab, consider using a commercial express package delivery service. For more information on the shipment of samples, consult U.S. Postal Service Publication 52 entitled "Hazardous, Restricted, or Perishable Mail" dated July 1999 and NAVSUPINST 4610.31A entitled "Preparation of Medical Material Requiring Freeze or Chill Environment for Shipment."

SAMPLING REQUIREMENTS

Always review the preferred method of sampling given in this guide and amplified by the appropriate analytical method (e.g., NIOSH or OSHA analytical method manuals, etc.). If the recommendation cannot be followed, contact the laboratory prior to sampling for additional guidance.

The recommended air volumes provided in this guide are usually a range of volumes, with the higher value recommended for the majority of sampling. The lower air volume should only be used when: 1) the exposure may be at an unsafe/unhealthful exposure level such as an exposure exceeding the Time-Weighted Average (TWA) value given in the Occupational Safety and Health Administration's Final Rule Limits, 2) the application of a Short-Term Exposure Limit (STEL) or a Ceiling value is applicable to the substance, and 3) the operation limits the amount of sampling time. In the last two cases the maximum recommended sampling rate should be used to obtain as much sample volume as possible. As a general rule, the recommended sampling volumes will allow a detection limit of 10-50% of the TWA.

SAMPLE PACKAGING AND SHIPPING REQUIREMENTS

(See Biological Samples Section for requirements on shipping Biological Samples.)

1. Small sample media such as sorbent tubes and filter cassettes should be bound together (i.e., rubber band) or placed in plastic bags to reduce the possibility of being overlooked or discarded. Sample cassettes and sorbent tubes should NOT be wrapped in tape. Simply affix a legible sample submission number (preferably a preprinted label) to each sample and blank and neatly package it to avoid shipping damage. Never ship air samples and bulk solvent (i.e. fuels, naphthas) samples in the same shipping package.
2. Submit separate request forms for each type of analyses as follows: Segregate and ship your samples in individual categories of air, bulk, wipe, and biological samples subdivided by metals and organics.
3. Many solvents can be analyzed simultaneously unless they are incompatible. Always check compatibility information to ascertain the organic contaminants collected on charcoal tube media are compatible with each other and with the analytical procedure. Call the laboratory regarding compatibility when in doubt. MCEF samples submitted for metal analyses must be collected on a 37-millimeter diameter membrane filter not a 25-millimeter MCEF. Please note that hexavalent chromium, mercury, and organic tin are analyzed separately and may not be combined with other metal analyses. Individual metals may be ordered or an ICP metal scan may be requested. Unusual situations requiring additional analyses should be coordinated with the laboratory prior to sample collection.
4. When necessary, small quantities of bulk organic solvents may be shipped in small screw cap glass containers (5-15 ml size) with a tight fitting Teflon-lined cap. One, but not the only, suitable container is the Fisher Scientific 12 ml glass sample vial with PTFE-lined cap (Cat. #

03-340-60C). Prior to shipment place a permanent ink mark at the level to which the vial is filled. This allows the chemist to determine potential leakage during shipment. Rarely will more than 5 milliliters of sample be required. Place each vial in a zip-lock bag and then place that bag in another zip-lock bag to provide double bag security against leakage. Never ship the bulk and air samples in the same shipping package. Provide information telling the chemist which bulk sample corresponds to the air samples. The CIHL only needs bulk samples for non-standard organic mixtures such as Stoddard's Solvent, Petroleum Naphtha, mineral spirits, etc. The CIHL has standards for Gasoline, Kerosene, JP-4, JP-5 and JP-8. Do not send bulk samples of paints as they cannot be analyzed successfully. When in doubt, call the lab and ask for guidance.

5. Most determinations require a minimum of two blanks or one blank for every ten samples submitted, whichever is larger. Remember to always provide 20-30 ml of unexposed impinging solution to be used by the laboratory as media blanks and for quality control. The blanks are analyzed by the CIHLs and reported as micrograms (ug) of "contaminant" per sample (e.g., per filter, per tube, etc.).

6. All references to water in this guide mean deionized or double-distilled water.

7. When submitting a sample for elemental analysis of hard metal alloys, the bulk sample must be in the form of fine filings, powder or very thin wire to facilitate digestion prior to analysis.

8. The preferred refrigerant for samples that require refrigeration is freezer packs or frozen gel blocks. Ice may be used for hand-carried lab samples, however the ice must be doubly wrapped in plastic zip-lok bags to avoid leakage. Never use ice or dry ice when shipping by U.S. Postal Services or commercial delivery services.

9. Shipping containers should be appropriately labeled such as "Fragile", "Refrigerated Material", "Liquid Samples", "Etiologic Agent/Biomedical Material", etc.

10. All samples and materials being packaged, labeled and shipped are governed by Federal, State and local regulations. Compliance with these regulations is the responsibility of the person submitting the samples.

11. In the case of unusually large shipments or high priority samples, please contact the laboratory prior to submission.

SAMPLE TURNAROUND TIMES

Samples will be analyzed on a "first come, first served" basis. Urgent samples will be given special priority and analyzed in one to three working days when the laboratory has been notified in advance of the shipment, sufficient production capacity is available and the samples have arrived by expedited shipment, e.g., hand delivery within 24 hours, FedEx overnight or USPS express mail. More than 90% of routine samples are analyzed within 10 working days after receipt of the sample. If you have not received your analytical report after 20 working days, please notify the laboratory to check on the status of the samples.

SAMPLE COMPATIBILITY

Since sampling and analytical methods are normally evaluated for a single analyte, care should be taken in the interpretation of a method's CV. When in doubt concerning multi-component samples, take individual samples. The following compounds require special processing for analysis and consequently the lab cannot analyze for other compounds in the same sample:

Acetic Acid

Acetonitrile

Acrolein

All cellosolves can be analyzed from the same tube, e.g., butyl-, methyl-, etc., **HOWEVER** cellosolves and common organics cannot be analyzed from the same solid sorbent tube.

All isocyanates

Ammonia

Aniline

2-Butanone

Butyl Cellosolve

Camphor

Cellosolve

Chlordane

Chromic Acid or Chromium (VI)

Coal Tar Pitch Volatiles

Cresols

Ethylene glycol

Ethylene oxide

Ethyl ether

Formaldehyde

Hydrazine

Methanol

Methyl Cellosolve

Methyl methacrylate

2-Nitropropane

PCBs

PGDN (Otto Fuel II)

Phenol

Pyridine

Tungsten

The following groups of compounds require special processing for analysis. More than one compound within each group can be analyzed in the same sample, but compounds outside the group are incompatible and cannot be analyzed from the same sample:

Group I – Ethyl Alcohol, Isopropyl Alcohol, and t-Butyl Alcohol

Group II – n-Butyl Alcohol, sec-Butyl Alcohol, iso-Butyl Alcohol and n-Propyl Alcohol

Group III – Iso-Amyl Alcohol, Diacetone Alcohol, and Cyclohexanol

Group IV – 2-Methoxyethanol, 2-Ethoxyethanol, and 2-Butoxyethanol

LAB SPECIFIC SAMPLES

Both of the CIHLs have specific areas of unique expertise and only those laboratories should be used in those specialty areas. The areas and labs are:

<u>Area</u>	<u>Laboratory</u>
Multi-element analysis using Inductively Coupled Plasma (ICP) Spectrometry	Both
Advanced High Performance Liquid Chromatography	Both
Gas Chromatography/Mass Spectrometry	Both
X-Ray Diffraction	San Diego
Volatile and Semi-Volatile Organic Compounds by SUMMA Canister	San Diego
Volatile Organic Compounds by Thermal Desorption of Solid Sorbent Tubes	Norfolk

ROUTINE BIOLOGICAL SAMPLES

Consult the section on Submission Requirements for Biological Samples for general policies of sampling, packaging, labeling and shipping biological samples.

BLOOD LEAD AND ZINC PROTOPORPHYRIN

Collect in one of the following Becton Dickinson (BD) Vacutainer Systems listed below:

BD Number	Top Color	Description
6527	Dark Blue	Sodium heparin tube for whole blood (Specifically for trace element studies)
6450	Lavender	15% EDTA tube for whole blood & Zinc Protoporphyrin

Samples must be thoroughly mixed with the heparin or EDTA immediately following collection. Keep samples refrigerated (do not freeze) and hand deliver or ship to the nearest laboratory using priority shipping methods. Use an insulated shipping container, such as a styrofoam shipper. For shipping long distances, freezer packs and express delivery are required.

URINE MERCURY

See the section on Submission Requirements for Biological Samples for general policies of sampling, packaging, labeling and shipping biologicals.

Per BUMED INSTRUCTION 6260.2, dated 7 November 1988, biological monitoring for mercury is no longer required. The potential for personnel exposure to elemental mercury vapor has been greatly reduced by the use of pre-encapsulated amalgams. Industrial hygiene surveys have shown routine use of pre-encapsulated amalgams does not result in overexposure of dental

personnel to elemental mercury vapor. Therefore, per this BUMED instruction, neither biological sampling nor air sampling is specifically required. Occasionally mercury urine may be prescribed by an occupational health professional as circumstances warrant.

If urine mercury analysis is necessary, collect the sample (first morning void, if possible) in the standard drug screening plastic bottle (NSN 6640-00-165-5778) and add 100 milligrams of potassium persulfate, a preservative. Please do not send more than 20 milliliters of urine per sample. Hand tighten the lid, and place each bottle in a zip-lock bag to contain any leakage during transit to the laboratory. Refrigerate during storage and ship, as soon as possible, in an insulated shipping container, using freezer packs (gel blocks) and express delivery.

SPECIAL SAMPLING & ANALYSES

BULK SAMPLE SUBMISSIONS

The primary function of the CIHLs is the analysis of breathing zone air samples to document occupational exposure levels. The CIHLs do not analyze samples for facility inventory purposes (e.g., materials suspected to contain asbestos) or as an aid to production planning (e.g., heavy metals in paint chips, materials suspected to contain asbestos) or analysis of bulk samples to determine whether a product meets manufacturer's specifications. Information for the latter is available by writing the manufacturer and requesting product literature and Material Safety Data Sheets. Products for which this information is not available should not be used in the Navy system. Bulk samples should be submitted to the laboratories only under the following conditions:

(1) When the laboratory requests a bulk, as is required in the analytical method (e.g., PCBs, Naphthas, etc.).

(2) When all other means of obtaining information on the chemical composition of the material have been exhausted and prior approval has been given by the CIHL Director.

CHROMIUM AND CHROMATES

Chromium metal, (or total Chromium) Cr (II) and Cr (III) compounds are collected on mixed cellulose ester filters (MCEF) and analyzed using ICP. Cr (VI) compounds cannot be determined if sampled on a MCEF.

Chromium in the +6 oxidation state (i.e., Cr (VI) or Hexavalent Chromium, chromic acid, chromium trioxide, all chromates and dichromates must be collected on PVC filters, with backup pads. If other filter materials are used, the Cr (VI) may be reduced to the Cr (II) or Cr (III) states and thus give a diminished value for Cr (VI). Note: You no longer need to separate the filter from the backup pad prior to shipping the sample. Simply ship the PVC filters in their sampling cassettes.

OSHA has issued a revised version of the OSHA ID-215 method for hexavalent chromium sampling. Method Number ID-215 (version 2), Control Number T-ID215-FV-02-0604-M. The significant modification (related to sample collection) in the method is that when using the 37 or 25 mm PVC filter with cellulose back-up pad for welding operations, or chromium plating operations, special handling requirements have been added.

A summary of the new special handling requirements for samples collected from welding or plating operations follows:

1. Samples collected on PVC filters from welding or plating operations must be shipped overnight to the laboratory within 24 hours of sampling.
2. Samples collected on PVC filters from welding operations must be analyzed within 8 days of sampling.
3. Samples collected on PVC filters from chromium plating operations must be analyzed within 6 days of sampling or be stabilized at the laboratory upon receipt.
4. Please make sure that the shop operation is plainly stated on your IH Air Sample Survey Form, e.g., welding, plating, painting, abrasive blasting. This allows the CIHL to verify that expedited analysis is not required but the examples of painting and abrasive blasting do NOT imply that samples arising from those operations require special handling or expedited analysis.

Your analytical results could potentially be jeopardized if the above requirements are not adhered to. We are **not** requiring that the NaOHqz filters be used for the chromium plating operations at this time. We will preserve the samples with the appropriate buffers when received in our laboratory if the samples cannot be analyzed within the days required by the new method changes.

ENVIRONMENTAL LEAD SAMPLES

Both CIHLs are accredited by AIHA under the Environmental Lead Laboratory Accreditation Program (ELLAP) and accept paint chips and dust wipes for lead analyses.

FIBER COUNTS AND ASBESTOS IDENTIFICATION

These determinations are to be made in the field or at the local activity level. The CIHLs will assist on a case-by-case basis, however, prior approval for accepting these samples must be received from the CIHL before submitting fiber count or asbestos identification samples to the CIHLs.

Laboratories performing these analyses must be proficient in the appropriate quality assurance (QA) programs. For fiber counts, the appropriate QA program is the American Industrial Hygiene Association (AIHA) Proficiency Analytical Testing (PAT) program. For bulk asbestos identification the appropriate QA programs are NMCPHC's contractor (i.e., Research Triangle Institute) -operated quality assurance program, the AIHA Bulk Asbestos PAT program, or the National Voluntary Laboratory Accreditation Program for Asbestos Identification.

POLYCHLORINATEDBIPHENYLS (PCBs)

Both CIHLs routinely determine the PCB content in bulk samples as it relates to occupational health, with a lower reporting level of 0.1% or 1,000 ppm. The laboratories do not routinely analyze to the EPA standard of 50 ppm for waste disposal purposes.

SILICA (CRYSTALLINE SILICA) ANALYSIS

This method determines silica in respirable and total dust by the OSHA method. The sample filter used is a 5 um PVC filter. SKC Cat No. 225-8-01 (low silica homopolymer PVC), the Omega SILICAL PVC filters, or equivalent low silica homopolymer PVC filter should be used. The respirable dust sample is collected at 1.7 LPM to obtain 800 to 1,000 liters of air. A smaller air volume may be used if filter loading greater than 2.0 milligrams is expected.

Bulk samples can be semi-quantitatively analyzed for quartz and cristobalite.

CONVERSION FACTORS

In a metal scan, Iron (Fe), Zinc (Zn) and Vanadium (V) concentrations (in mg/m³) are reported instead of the metal oxide concentrations (i.e., Fe₂O₃, ZnO, and V₂O₅) for which one is actually sampling. Therefore, a conversion factor must be used to "convert" the reported result for the metal to the equivalent concentration of the metal oxide for comparison with the PEL/TLV listed for the oxide. The following are examples of how to calculate a conversion factor and use it to calculate the concentration of metal oxide:

1. Conversion of zinc to zinc oxide:

Calculate the conversion factor - MW of ZnO / MW of Zn = 81.4 / 65.4 = **1.245**

Multiply the conversion factor times the result reported as Zn to obtain the amount of ZNO since the PEL/TLV is for ZnO.

2. Conversion of vanadium to vanadium pentoxide:

Calculate the conversion factor - MW of V₂O₅ / MW of V₂ = 181.9 / 101.9 = **1.785**

If sampling for V₂O₅ as a dust, then the 1989 OSHA PEL is as a "respirable" dust. Technically, that means sampling with a cyclone. However, if the "total" dust result (typical sampling method) is below the "respirable" PEL then there shouldn't be a problem since the sampling method would overestimate the respirable fraction. But, if the "total" dust sample result exceeds the PEL, then the IH will have to decide if respirable fraction sampling is needed to accurately assess PEL compliance. This "twist" does not affect V₂O₅ "fume" results. The correction factor is applied to results reported by the lab.

3. Conversion of iron to iron oxide:

Calculate the conversion factor - MW of Fe₂O₃/MW of Fe₂ = 159.7 / 111.7 = **1.43**

The 1989 OSHA PEL (what the Navy generally uses) and ACGIH TLV for Fe₂O₃ (iron oxide) dust and fume is "as Fe" (the element) and not "Fe₂O₃" (the oxide). Therefore, normally the correction factor would NOT be used to convert the "Fe" results reported by the lab to the "oxide". However, because the 1989 PELs were stayed by the court, the true current (old 1970) OSHA PEL is as "iron oxide fume" (Fe₂O₃), therefore, the conversion factor is used. The conversion factor is used to convert lab results reported as Fe for both "iron oxide" fume and dust samples.

4. Conversion of Cr(VI) to Chromates (as CrO₃):

Calculate the conversion factor - MW of CrO₃/atomic weight of Cr(VI) = 99.9 / 51.9 = **1.92**

a. **Chromic Acid (CAS # 7738-94-5):** The 1989 OSHA PEL (0.1 mg/ m³) for chromates is expressed "as CrO₃" and is a Ceiling Limit. The results reported by the laboratory when using a PVC filter are "as total Cr(VI)". Therefore, the conversion factor must be used to "convert" the sampling result to the OSHA PEL. **Note:** Laboratory results reported for an MCEF filter are for "total Cr", that is, all forms of chromium, including Cr(VI). Therefore, you may overestimate "chromate" exposure if there are other forms of chromium generated by the process being evaluated.

b. **Hexavalent Chromium Cr(VI) (CAS # 18540-29-9):** However, **NO** conversion factor is used if sampling for chromates (as an eight-hour TWA) to compare to the **NEW** OSHA PEL of 5 µg/m³ (0.005 mg/m³). This is because the NEW OSHA TWA-PEL is "as hexavalent chromium (Cr(VI))" and not "as CrO₃." This also holds true when comparing a Cr(VI) result reported from the lab to an ACGIH TWA-TLV for lead chromate, barium chromate, strontium chromate, or insoluble Cr(VI) compounds (NOC). **Note:** Although the ACGIH TWA-TLV listings for lead chromate, barium chromate, strontium chromate, etc. indicate "as Cr", it is actually "as Cr(VI)" per discussions with the ACGIH Technical Affairs Office on 01 October 1997.

SOURCES FOR ANALYTICAL SUPPLIES

NOTE: The mention of specific company names and products does not constitute endorsement by the Navy and Marine Corps Public Health Center. Similarly, the omission of a specific company name or product does not imply that they or their product is not recommended for use it only means that this is not and cannot be an all inclusive listing.

MANUALS

The NIOSH analytical manuals may be obtained from:

<http://www.cdc.gov/niosh/nmam/nmampub.html>

Superintendent of Documents

PO Box 371954

Pittsburgh, PA 15250-7954

(202) 512-1800 or fax orders to (202) 512-2250.

[NIOSH Manual of Analytical Methods, fourth edition, 3 parts, Aug 94, DHHS Publication No. 94-113, price approximately \$64.]

The OSHA analytical manuals may be obtained from:

<http://www.osha.gov/dts/sltc/methods/index.html>

ACGIH Publications

1330 Kemper Meadow Drive

Cincinnati, OH 45240-1634

Phone: (513) 742-2020 FAX: (513) 742-3355

[Publications #4542, #4544 and #4545]

<http://www.acgih.org/>

FILTERS AND SORBENT TUBES

Filters and sorbent tubes may be obtained from a number of sources; however, this manual cites SKC order number for filters and tubes (listed in the SPECIAL INSTRUCTIONS column in the Laboratory Sampling Guide), simply because of convenience and uniformity.

Special attention should be given to SKC Guide to NIOSH/OSHA Air Sampling Standards which is in the SKC Comprehensive Catalog and Air Sampling Guide (Request free copy from SKC.)

SKC, Inc. World Headquarters

863 Valley View Road

Eight Four, PA 15330-9614

Phone: (800) 752-8472 FAX: (800) 752-8476

Website: <http://www.skcinc.com/>

SKC, Gulf Coast

9827 Whithorn Drive

Houston, TX 77095-5027

Phone: (800) 225-1309 FAX: (800)752-4853

SKC, West
P.O. Box 4133
Fullerton, CA 92634-4133
Phone: (800) 752-9378 FAX: (800) 752-1127

Supelco, Inc.
Supelco Park
Bellefonte, PA 16823-0048
Phone: (800) 247-6628 FAX: (800) 447-3044
Technical information only phone: (800) 359-3041
Website: <http://www.sigma-aldrich.com/>

PASSIVE MONITORS

3 M Company
Occupational & Environmental Safety Division
3 M Center, Bldg 224-5S-04
St. Paul, MN 55144-1000
Phone: (800) 752-3623 (Federal System Group orders)
Technical information only phone: (800) 243-4630
Website: <http://www.3m.com/>

PRINTED SAMPLE NUMBER LABELS

Shamrock Scientific
34 Davis DR, Bellwood, IL 60104
Phone: (800) 323-0249
Website: <http://www.shamrocklabels.com/>

SAMPLE COLLECTION BOTTLES, VIALS, AND SUPPLIES

Supelco, Inc.
Supelco Park, Bellefonte, PA 16823-0048
Phone: (800) 247-6628
Website: <http://www.sigma-aldrich.com/>

SKC, Inc.
863 Valley View RD
Eighty Four, PA 15330-9614
Phone: (800) 752-8472
Website: <http://www.skcinc.com/>

DUST WIPE MEDIA

Palintest Dust Wipe

Available from Palintest USA, 21 Kenton Lands RD, P O Box 18733, Erlanger KY 41018,
Phone: (800) 835-9629

Ghost Wipe

Available from Environmental Express, 490 Wando Park Blvd., Mt. Pleasant, CA 29464

Phone: (800) 343-5319

Website: <http://www.envexp.com/>

ABBREVIATIONS

C	Contract Laboratory
N	Norfolk Laboratory
S	San Diego Laboratory
@	at the concentration of
AMBERSORB	Special type of adsorption tube
aq	aqueous
CASRN	Chemical Abstract Service Registry Number
CAS#	Chemical Abstract Service Registry Number
CIHL	Comprehensive Industrial Hygiene Laboratory
CHROMOSORB	Special type of adsorption tube
CT	Charcoal tube (see special instructions for part number)
CV	Coefficient of Variation
FLORISIL	Special type of adsorption tube
FLT	Filter
GFF	Glass fiber filter
HOPCALITE	Special type of adsorption tube for Mercury vapor
ICP	Inductively Coupled Plasma (analyzes multiple metals per sample)
INHOUSE	laboratory method developed within the organization
L	liters
LPM	liters per minute
LOD	Limit of detection (an amount equal to three times the standard deviations of the analytical noise or three times that of a blank, whichever is more appropriate).
LOQ	Limit of Quantitation. The lowest concentration at which a contaminant can be reliably reported.
0.8 MCEF	Mixed cellulose ester filter, 0.8 micrometer pore size
mg/m ³	milligrams per cubic meter
ml	milliliters
mm	millimeter
MW	Molecular Weight
NIOSH	National Institute for Occupational Safety and Health
NOS	Not otherwise specified
ORBO	Adsorption tube trade marked by Supelco
OSHA	Occupational Safety and Health Administration
OVS	OSHA Versatile Sampler--Special collection device for pesticides, available from SKC # ST 226-30-16.
ppm	parts per million
PTFE	Polytetrafluoroethylene filter
PVC	Polyvinylchloride filter, 5 micrometer pore size
QCC	Quality Control Coordinator
SG	Silica gel sampling tube
ST	Sorbent tube
TENAX	Special type of adsorption tube
um	micrometer
XAD	Special type of adsorption tube