



ASSOCIATION CONNECTING
ELECTRONICS INDUSTRIES®

IPC-1065

Material Declaration Handbook

(For Users and Manufacturers of Printed Circuit Boards)

IPC-1065

January 2005

A standard developed by IPC

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Material Declaration Handbook

This handbook has been developed by the Materials Declaration Handbook Task Group (4-34a) of the Materials Identification Subcommittee (4-34) of IPC to aid Printed Circuit Board manufacturers and users of Printed Circuit Boards in completing Material Declarations that follow the format and guidance of the Joint Industry Material Composition Declaration Guide.

Users of this publication are encouraged to participate in the development of future revisions.

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Acknowledgment

Any document involving a complex technology draws material from a vast number of sources. While the principal members of the Materials Declaration Handbook Task Group (4-34a) of the Materials Identification Subcommittee (4-34) are shown below, it is not possible to include all of those who assisted in the evolution of this standard. To each of them, the members of the IPC extend their gratitude.

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The following people helped by reviewing this handbook and giving feedback:

Mary Betsch, Sanmina SCI
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Rick Charbonneau, Storage
Technology Corporation
Ofer Cohen, Seabridge, Ltd
Vivek Gupta, Intel

Mark Newton, Dell Incorporated
Alan Rae, Cookson Electronics
Dongkai Shangguan, Flextronics
International
Seigmund Zweigart, Solelectron GmbH

Joan Girard, Teradyne Connection
Systems
Bret Bruhn, Merix Corporation
Jeff Instone, Sanmina-SCI Phoenix
Inc.

*Special thanks to Mitch Ostrowski, EMT Laboratories, who answered
many questions regarding analysis of electronic products.*



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Table of Contents

1 SCOPE.....	1	5.1 Joint Industry Material Composition Declaration Guide Substances	6
1.1 Purpose	1	5.2 Analysis Methodology	6
1.2 Introduction	1	5.3 Sample Preparation	7
1.3 Definitions, Acronyms, and Abbreviations.....	1	5.3.1 Substance Analysis Methods	7
2 WHAT TO DO WHEN YOU RECEIVE A MATERIALS DECLARATION REQUEST FROM A CUSTOMER	1	Appendix A1 Table of Chemical Lists and Laboratory Analytical Methods	15
2.1 Review and Assess Request.....	2	Appendix A2 Detailed Calculation of a Material Declaration of a Printed Circuit Board	35
2.2 Secure Product Expert(s) Support	2	Appendix A3 Specific Material Data	46
2.3 Gather Necessary Data.....	2	Appendix A4 Uncertainty of Calculated Data Values	49
3 CHEMICAL SUBSTANCES LIKELY TO BE FOUND IN A PRINTED CIRCUIT BOARD	3	Appendix A5 List of Analytical Laboratories	51
4 SIMPLIFIED SAMPLE CALCULATIONS FOR A MATERIAL DECLARATION OF A PRINTED CIRCUIT BOARD	4	Appendix A6 List of Relevant Documents	52
4.1 Example of Materials Calculation for a Printed Circuit Board	4	Appendix A7 Analytical Techniques for Substances Removed from Final Version of Joint Industry Guide	55
5 GUIDELINES FOR DETERMINING THE COMPOSITION OF AN ELECTRONIC PRODUCT USING ANALYTICAL TECHNIQUES.....	5		



Material Declaration Handbook

1 SCOPE

This handbook has been developed to aid Printed Circuit Board manufacturers and users of Printed Circuit Boards in completing Material Declarations that follow the format and guidance of the Joint Industry Material Composition Declaration Guide for Electronic Products.

1.1 Purpose This handbook's purpose is to demonstrate methods for calculating the information required for a Material Declaration. The data and calculations provided are intended as examples. They might or might not apply directly to your product. You will have to research your raw materials and processes to determine the exact parameters that go into the calculations provided.

1.2 Introduction Due to various regulations around the world, it has become increasingly important for electronics manufacturers to know the composition of their products. Electronic manufacturers have begun asking their suppliers for "material declarations (MDs)" that tell them the concentrations and locations of various substances in their products. The supply chain for many electronic products contains a wide range of organizational sizes - from global Original Equipment Manufacturers (OEMs) to small (<10 employees) companies that make a limited range of products. The information and abilities required to accurately complete a material declaration may exceed the capabilities of these smaller organizations.

In order to adequately perform a material declaration that is useful for a customer, suppliers must know:

- What substances should be included in the material declarations.
- What format should be used to inform customers about the substances.
- Where are the substances likely to be located in electronic products. If a supplier does not know if or where any substances may be in the part, how do they find out. If they have to send a sample to an analytical laboratory, what tests should be run.

To aid electronic manufacturers in meeting legal and regulatory requirements, accurate data and calculations must be used to generate MDs. Also, the information in MDs should be easily portable up and down the supply chains. To the extent possible, MDs should be standardized on the format and substance list of the Joint Industry Material Composition Declaration Guide for Electronic Products.

1.3 Definitions, Acronyms, and Abbreviations The following definitions are used in this handbook:

- **BIPM** – International Bureau of Weights and Measures
- **Chemical Abstract Services (CAS) Numbers** – a unique numeric identifier for chemical compounds and elements
- **FR-4** – a general grade printed circuit board laminate material composed of woven fiberglass fabric saturated with epoxy resin
- **HASL** – abbreviation for Hot Air Solder Levelling – which is the process of putting solder on a printed circuit board and levelling it with hot air
- **Immersion Silver** – abbreviated as Imm. Ag – the process of applying silver as a surface finish on a printed circuit board
- **Immersion Tin** – abbreviated as Imm. Sn – the process of applying tin as a surface finish on a printed circuit board
- **ISO – International Organization for Standardization**
- **Material** – a material is made up of one or more substances (e.g., copper alloy is a material, which in turn is made up of a number of defined substances, copper, nickel, silver, etc.) – (Joint Industry Material Composition Declaration Guide for Electronic Products)
- **MD** – abbreviation for Material Declaration – a document that lists the composition of an electronic product
- **Measurand** – the quantity of interest, such as the mass of copper in a printed circuit board which may be calculated from other quantities (e.g., plated area, etched area, thickness and density of copper, etc.)
- **OSP** – abbreviation for Organic Solderability Preservative – an organic coating applied as a surface finish to a printed circuit board that prevents oxidation of the copper on the surface of the printed circuit board
- **PCB** – abbreviation for Printed Circuit Board
- **Substances** – Substances are chemical elements and their compounds. Chemical Abstract Services (CAS) numbers are provided for these substances where known. – (Joint Industry Material Composition Declaration Guide for Electronic Products)

2 WHAT TO DO WHEN YOU RECEIVE A MATERIALS DECLARATION REQUEST FROM A CUSTOMER

This section assumes that your facility has received a request for a Material Declaration (MD) and gives some guidelines on the process that you should go through to adequately address the MD request.

There are several steps in completing an MD:

- Review and assess request
- Secure product expert(s) support
- Gather necessary data



- Perform calculations
- Convert raw material volumes to constituent contents
- Address other product constituents
- Compute concentrations
- Complete Material Declaration form

2.1 Review and Assess Request Be sure to review the MD request fully. Look for the following information:

- What part numbers are MDs being requested for? What particular “rev” of the part number is concerned? Sometimes this is relevant if you make/buy the same part number with different surface finishes (HASL vs. OSP vs. Immersion Tin, etc.) or different dielectric materials (tetra-functional FR-4 vs. halogen-free vs. Teflon, etc.).
- Do you have any prior MDs for this part number? If you do, have you made any updates to the system that calculates your MDs that would change the MD? For example, you have better data on your copper foil composition (e.g., you had an analysis done and found that it contains arsenic), or you have changed the thickness of copper that is plated.
- What information is the customer asking for? Does this MD request have any particular information that is specific to this customer only? For example, some customers ask for only a limited list of substances to be declared. Other customers ask that you account for 100% of the PCB’s weight. Some have a list of substances, and then ask for anything else that is more than 1% of the PCB’s weight. If the MD request does not match with the Joint Industry Material Composition Declaration Guide for Electronic Products substance list or format, consider asking your customer if they are aware they are not conforming with industry standards and if they would accept a response that conforms with the Joint Industry Material Composition Declaration Guide for Electronic Products. Inquire whether a conforming declaration would be deemed adequate. This type of feedback may encourage your customer to more closely align their request with the agreed-upon industry standard. Should your customer persist in requesting information beyond the standard, you will need to make a business decision as to how you will respond. You may wish to identify to your customer the additional effort associated with this request. In particular, if materials testing is to be conducted, we recommend that you identify whether you or your customer will be paying for the analysis.
- Is there a certification statement in the MD request? What are you agreeing to if you sign the customer’s certification statement? These statements can range from asking you to “do your best” at completing the MD to indemnifying the customer for any damages relating to a mistake in the MD. If you are not legally comfortable with the

certification statement, don’t sign it! An example of a generic certification that you might be able to convince your customer to accept is:

The results given are based on the best available information using customer-supplied tooling data, known manufacturing capabilities, applicable MSDS and other vendor-supplied information, and best engineering judgment. The concentrations are generally calculated and are not determined from laboratory analysis, although finished product weights are used if available.

- What is the deadline for completion of the MD? If you haven’t done any MDs before, it’s probably going to take you two weeks to a month to get the data that is required to complete the MD. If you need to do laboratory analyses or query your suppliers for data, it will take longer. Refer to Section 5. Guidelines for Determining the Composition of an Electronic Product Using Analytical Techniques if you are contemplating doing analyses. Be sure you communicate your expected completion date with your customer.
- If this is the first MD request you have received from the customer, be sure to communicate with your customer to ensure that you are both on the same page as to the requested information. You may also want to contact your company’s sales account representative or contract administrator as appropriate.

2.2 Secure Product Expert(s) Support You are going to need to know a lot about your manufacturing processes or those of your suppliers in order to complete the MD. It’s almost certain that no one person in your company will have all the required data. Be sure to involve the following experts:

- Front-end engineering
- Process engineering (especially plating, drilling, layup and lamination engineers)
- External experts such as laminate, prepreg, foil, solder-mask, and ink suppliers

2.3 Gather Necessary Data Here is the data that you are going to need to complete the MD:

Product Data:

- Dimension of final product
 - Length
 - Width
 - Thickness (and variation allowances)
 - Where on the PCB the thickness will be measured (e.g., over copper?)
 - It is best if you use data based on the finished PCB as shipped to your customer. If you use the entire panel size, you will have to remove the border or frame materials from the calculation.



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- Weight of final product (if possible)
- If the product is Foil or Conventional Build.

Composition Data:

- Copper foil
- Dielectric material
- Fiberglass in laminate and prepreg
- All plated materials that remain on the product, such as copper (electrolytic and electroless¹, solder, “tab” or “finger” plating, tin (if it remains on the product such as the Durabond™ process for lamination)
- Soldermask and Nomenclature/Legend Inks
- Surface finishes such as HASL, reflowed solder, OSP, Immersion Tin, Immersion Silver, Electroless Nickel/Immersion Gold, etc.
- If the composition of some of your raw materials is unknown, you should request a Material Declaration from your suppliers.

Material Characteristic Data:

- Copper foil
 - weight (0.5 oz, 1 oz, etc) for all layers
 - density of electrolytic foil (see Table A3-1)
- Dielectric material
 - Percent of resin in Dielectric material
 - Percent of fiberglass in Dielectric material
 - Construction of the laminate cores and prepreg
- Other plated substances
 - Densities (see Table A3-1)

Processing Data:

- Depth of microetch processes throughout facility.
- Area of copper retained on the layers after etching. The best source for this data is the Front End Engineering or PCB Design team. This type of data has to be gathered in order to plate at the appropriate current density in the plating process for the outer layers. It is highly likely that the Front End Engineering team can write a computer “script” that can calculate the area of copper retained on each layer after the etching processes.
- If there is a material (such as electroless tin) added to the layers prior to lamination, the thickness and density of this material must be known.
- Amount and style of prepreg fabrics added during the layup of the multilayer PCB.

- Drilled holes:
 - Number of drilled holes
 - Drilled diameters of holes
 - Finished diameters of holes (plated and unplated)
- Thickness of copper plated (electroless² and electrolytic)
 - On surface of PCB
 - In drilled holes
- Thickness of solder plate (if you use it and if it remains on the PCB)
 - On surface of PCB
 - In drilled holes
- Soldermask
 - Area covered³
 - Thickness (cured). This is usually described as an application rate such as grams per square foot.
- Nomenclature/Legend
 - Area covered
 - Thickness (cured)
- Surface Finishes (HASL, OSP, Immersion Silver, Immersion Tin, Electroless Nickel/Immersion Gold, etc.)
 - Area covered⁴
 - Thickness

Sources or References for Data:

- Customer supplied data and design specifications
- Batch Card (or Traveler)
- Drill Data Information
- Layup or Kitting Documentation
- Material Safety Data Sheets (MSDSs)
- Internet Website searches

3 CHEMICAL SUBSTANCES LIKELY TO BE FOUND IN A PRINTED CIRCUIT BOARD

A printed circuit board (PCB) has three main components - the metal conductors (plus any associated surface finishes), the resin in the dielectric material and coatings (e.g., soldermask or nomenclature), and the reinforcement in the dielectric material. Roughly speaking, the mass breakdown of the PCB will be about one-third for each of these components.

Although this list is not exhaustive, it is meant to provide a list of the chemical substances commonly found in each of these components. Your particular product might (and probably will) contain different substances.

1. If an alternative to electroless copper is used (e.g., graphite, Black Hole, Organic Palladium, etc.) you will need to determine if any of the substances included in the alternative are required to be reported by the customer.
 2. See Footnote 1.
 3. This area also needs to take into consideration the area of the drilled and plated holes (inside the barrels) that will be coated by Soldermask.
 4. See Footnote 3.

Component	Chemical Substance	Purpose	Substance on Joint Industry Guide List?
Metal Conductors	Copper	Conductor	No
	Phosphorus	In copper plating anodes	No
	Chromium	Conversion coating on laminate and foil	Yes, Level A if present as Cr (VI)
	Oxygen	Part of chromate conversion coating	No
	Arsenic	Use as a stain blocker on foil	Yes, Level B
	Lead	Surface Finish (might also be found in electroless Ni plating in low concentrations)	Yes, Level A
	Tin	Surface Finish	No
	Silver	Surface Finish	No
	Nickel	Surface Finish	Not in reportable form
	Gold	Surface Finish	No
Resin	Bromine	Fire Retardant	Yes, Level B
	Chlorine	Residual Contaminant	No
	Fluorine	In Teflon if used	No
	Epoxy	Most of Resin	No
Reinforcement	Silicon	Constituents of fiberglass	No
	Oxygen		No
	Calcium		No
	Aluminum		No
	Boron		No
	Magnesium		No
	Sodium		No
	Potassium		No
	Titanium		No
	Iron		No
	Fluorine		No

4 SIMPLIFIED SAMPLE CALCULATIONS FOR A MATERIAL DECLARATION OF A PRINTED CIRCUIT BOARD

This section assumes that your facility has received a request for a Material Declaration (MD) from a customer for a bare Printed Circuit Board (PCB). The calculations can be done in a similar fashion to the processes involved in manufacturing a PCB. Start with a certain quantity of material and subtract and add materials as the PCB proceeds through the processes. The process as described below may seem tedious. However, it is easily amenable to calculation by a spreadsheet program. A more rigorous example of a printed circuit board calculation can be found in Appendix A2.

4.1 Example of Materials Calculation for a Printed Circuit Board A printed circuit board (PCB) is constructed of multiple layers of reinforced dielectric layers alternating with layers of copper conductors. Each layer may have several vias, microvias, and drill holes.

For this discussion, imagine a simple PCB of area A_{PCB} consisting of one layer of dielectric sandwiched between two layers of copper.

To determine the amount of each material present, concentrate on one layer at a time.

For the dielectric layer, the **total area** is A_{PCB} and the **thickness** is t_{die} .

For the first calculation, the resin-to-reinforcement relationship for the dielectric is needed. For each type of commercial laminate layer available, the supplier may provide the weight percent of reinforcement. In addition, the unit weight of reinforcement is needed in order to calculate the corresponding resin content, and may be supplied as a weight per unit area for a supplied thickness, i.e., R in mass/area for a given thickness.

So, the mass of reinforcement, $m_{reinforcement}$ in the PCB under consideration can be determined from:

$$m_{reinforcement} = R * A_{PCB}$$

To calculate the corresponding weight of resin, the resin-to-reinforcement weight percent can be used, i.e.,:

$$X_{reinforcement} = 100 - X_{resin}$$



Thus,

$$mass_{resin} = \frac{X_{resin}}{(100 - X_{resin})} (mass_{reinforcement})$$

This PCB under consideration is double-sided with copper, of area $A_{Cu,1}$ on side 1, and $A_{Cu,2}$ on side 2. Each side has equal thickness t_{Cu} . Both sides are also equally overplated with more copper, of area A_{plated} and thickness t_{plated} . To determine the mass of copper on side 1, the density of copper, ρ_{Cu} is used.

For Side 1:

$$m_{Cu,1} = \rho_{Cu} * t_{Cu} * A_{Cu,1} + \rho_{Cu} * t_{plated} * A_{plated}$$

Similarly, for Side 2:

$$m_{Cu,2} = \rho_{Cu} * t_{Cu} * A_{Cu,2} + \rho_{Cu} * t_{plated} * A_{plated}$$

Therefore, the total copper content can be found by adding these results.

Sometimes the outer layers of copper on a PCB may be plated with a finish such as tin, silver, electroless nickel/immersion gold. The weight of the finish can be found in a similar manner knowing the area of the plated region, the finish thickness, and the density of the metal(s).

Often, the total area of patterned copper or of a dielectric layer can be determined from layout software, and would include any drill holes, vias, etc. However, if it does not include these, then the true areas can be found by subtracting the area of the drill holes from the layers. For example, if the PCB discussed previously had plated through-holes of diameter d , then the amount of drilled material needs to be considered. Each drill hole removes $\pi(d/2)^2$ worth of area, where d is the diameter of the drill hole.

From the supplier data, the weight of reinforcement per unit area is known. For each hole the amount of reinforcement removed is therefore;

$$m_{reinforcement,removed} = R * \pi(d/2)^2$$

And then the mass of resin removed for each hole of diameter d is:

$$mass_{resin\ removed} = \frac{X_{resin}}{(100 - X_{resin})} (mass_{reinforcement\ removed})$$

If the hole is drilled through a region of copper, then that weight must also be accounted for. The absence of copper from side 1 per drill hole of diameter d can be expressed as:

$$m_{Cu,removed,1} = \rho_{Cu} * t_{Cu} * \pi(d/2)^2 + \rho_{Cu} * t_{plated} * \pi(d/2)^2$$

Similarly, the absence of the plated copper on side 2 is expressed as:

$$m_{Cu,removed,2} = \rho_{Cu} * t_{Cu} * \pi(d/2)^2 + \rho_{Cu} * t_{plated} * \pi(d/2)^2$$

However, for plated through-holes, the walls get plated to form a barrel shape of plated copper. This addition of copper metal can be accounted for by using the thickness of the plated copper, t_{plated} , the drill hole diameter, d , and the panel thickness, t_{panel} :

$$m_{barrel} = [\pi(d/2)^2 - \pi(d/2 - t_{plated})^2] * t_{panel} * \rho_{Cu}$$

Therefore, the total mass of copper is:

$$m_{Cu,total} = m_{Cu,1} + m_{Cu,2} - (m_{Cu,removed,1} * N_d) - (m_{Cu,removed,2} * N_d) + (m_{barrel} * N_d)$$

Where N_d is the number of holes of diameter d .

The total mass of resin is:

$$m_{resin,total} = m_{resin} - (m_{resin,removed} * N_d)$$

The total mass of reinforcement is:

$$m_{reinforcement,total} = m_{reinforcement} - (m_{reinforcement,removed} * N_d)$$

The steps outlined here were for a simplified construction, but would be equally applied to each layer of a multilayer board with multiple holes of various diameters. As with any calculation, it will only be as accurate as the information on the raw materials, including tolerances on reinforcement content, foil thicknesses, diameters and plating thicknesses. Although many layout software tools may provide final copper layout areas, they may not take into account the plated barrel walls of a hole. In addition to the sources of metal addition and removal, there may be pre-clean and pre-etch stages that remove some thickness and must be accounted for.

5 GUIDELINES FOR DETERMINING THE COMPOSITION OF AN ELECTRONIC PRODUCT USING ANALYTICAL TECHNIQUES

The purpose of this section is not to define the sampling rate and quantity needed. It is the responsibilities of both the customer and supplier to agree upon the best arrangement to verify that the product complies with any composition restrictions. In general, there are four levels of verification:

1. Supplier self-verification
2. Third party verification
3. Customer internal verification
4. End-user verification

The needs for each level must be addressed to determine the best approaches (e.g., frequency, limits, declared substances, quantity, acceptable level, cost, etc). It is imperative to keep in mind that sampling is only considered as a safety net for controlling material contents. It is imperative to have a sound supply chain management system in place.

to control the use of restricted material contents effectively. In some cases, sampling frequencies can be optimized or reduced as confidence levels increase by using sound statistical principles.

5.1 Joint Industry Material Composition Declaration Guide Substances The Joint Industry Material Composition Declaration Guide lists 24 categories of substances that must be declared. The first fifteen categories represent substances that, when used in electronics or electrical products, are subject to currently enacted legislation somewhere in the world that either:

- Prohibits their use;
- Restricts their use; or
- Requires reporting or results in other regulatory effect.

These are referred to as “Level A” substances:

1. Asbestos
2. Azo Colorants
3. Cadmium/Cadmium Compounds
4. Hexavalent Chromium/Hexavalent Chromium Compounds
5. Lead/Lead Compounds/Lead Alloys
6. Mercury/Mercury Compounds/Mercury Alloys
7. Ozone Depleting Substances (CFCs, HCFCs, HBFCs, carbon tetrachloride, etc.)
8. Polybrominated Biphenyls (PBBs)
9. Polybrominated Diphenyl ethers (PBDEs)
10. Polychlorinated Biphenyls (PCBs)
11. Polychlorinated Naphthalenes (more than 3 chlorine atoms)
12. Radioactive Substances
13. Shortchain Chlorinated Paraffins
14. Tributyl Tin (TBT) and Triphenyl Tin (TPT)
15. Tributyl Tin Oxide (TBTO)

The remaining nine categories are composed of materials and substances that the industry has determined are reportable because they meet one of the following criteria:

1. Materials/substances that are of significant environmental, health, or safety interest;
2. Materials/substances that would trigger hazardous waste management requirements; or
3. Materials/substances that could have a negative impact on end-of-life management.

These are referred to as “Level B” substances:

16. Antimony/Antimony Compounds
17. Arsenic/Arsenic Compounds

18. Beryllium/Beryllium Compounds/Beryllium Alloys
19. Bismuth/Bismuth Compounds/Bismuth Alloys
20. Brominated Flame Retardants (other than PBBs or PBDEs)
21. Nickel/Nickel Compounds/Nickel Alloys
22. Certain Phthalates (defined in the “Substance Analysis Methods” section)
23. Selenium/Selenium Compounds/Selenium Alloys
24. Vinyl Chloride Polymer (PVC)

5.2 Analysis Methodology

SW-846

Since most electronic and electrical equipment are “solid” materials, it was determined that the EPA publication SW-846, entitled Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, should be the primary source of the analytical methods. SW-846 is EPA’s Office of Solid Waste’s (OSW) official compilation of analytical and sampling methods that have been evaluated and approved for use in complying with the RCRA regulations. SW-846 is a “living” document - it is constantly changing. The information given in this guideline may have been superseded by the time you read this Handbook. Please refer to the most current version of SW-846 on EPA’s website at: <http://www.epa.gov/epaoswer/hazwaste/test/main.htm>.

When you access this webpage, you will see that SW-846 is broken into several sections. There is some good information in the section labeled “Chapters” that will help a great deal. If you are going to send some materials to a laboratory for quantitative analysis, it is recommended that you read “Chapter 2 - Choosing the Right Procedure.” Although it gets technical in places, it will give you a basis to hold a conversation with your analytical laboratory. It also tells you what substances the various methods will determine.

You will notice that the online SW-846 has several “series” of analyses. These are:

0010 - 0100 Series	Stack emissions or indoor air quality methods
1000 Series	Characterization methods
3000 Series	Extraction, Dissolution, and Digestion methods
4000 Series	Immunoassay methods
5000 Series	Volatile Organic Compound sampling and analysis from other matrices
6000 Series	Inductively Coupled Plasma methods
7000 Series	Atomic Absorption Spectrophotometry (Flame and Furnace), and Anodic Stripping Voltametry methods

8000 Series	Gas Chromatography, Gas Chromatography/Mass Spectrophotometry, High-Performance Liquid Chromatography, Fourier Transform-Infrared Spectrometry methods
9000 Series	Miscellaneous methods (Radioactivity, Anions, Cyanides, coliform, etc.)

For our purposes, we are not concerned with the 0010-0100, 1000 and 4000 series methods. For most of the substances requiring analytical testing, follow these steps:

1. *Sample Preparation* – the electronic product will need to be reduced to a fine talc-like powder.
2. *Dissolution* – the powder will need to be dissolved in a solvent for analysis. For organic substances, this might be an extraction. For inorganic substances, this will probably be a digestion method. These methods are in the 3000 Series of SW-846 and are dependant upon which substance you are analyzing for.
3. *Analysis* – the solution resulting from the dissolution step must be analyzed. For inorganic substances, this will probably be either an ICP method from the 6000 series or an AAS method from the 7000 series. For organics, you will probably use an 8000 series method.

5.3 Sample Preparation In all of the methods listed below, there is serious potential for contamination. This is true for the sample preparation step also. Method blanks should always be run alongside samples. This includes the sample preparation step, to ensure that nothing is contaminating the sample from either previous sample preps, or from the grinding apparatus itself.

As mentioned above, the first step is the reduction of the electronic product to a fine powder (to approximately 0.25 mm or Mesh size 60). The size reduction is normally made in several steps. If you have a large product, it will need to be cut into smaller pieces (on the order of 1 inch x 4 inches x 4 inches). The actual size will depend on your analytical lab's equipment.

The first reduction step might take the particle size down to approximately 4 mm (Mesh size 5). The second reduction step might go to 1 mm (Mesh size 18 or 16). Prior to the final size reduction, the sample should be frozen by placing it in a test tube, and immersing the test tube in liquid nitrogen. After 15 minutes, the sample should then be ground so that it will pass through the 60 Mesh screen.

The sample is now ready for the extraction steps. For metals analysis, this may be an acid digestion. For organic analysis, there may be a solvent extraction or even a dissolution step.

5.3.1 Substance Analysis Methods

1. Asbestos

Asbestos can be identified only under a microscope by either Polarized Light Microscopy (PLM) or Transmission Electron Microscopy (TEM). There are no currently published methods for bulk analysis by TEM. The National Institute for Standards and Technology maintains a list of laboratories accredited for asbestos analysis for both techniques under the National Voluntary Laboratory Accreditation Program (NVLAP). The list of labs accredited under NVLAP can be accessed at:

PLM –

<http://ts.nist.gov/ts/htdocs/210/214/scopes/plmtm.htm>

TEM –

<http://ts.nist.gov/ts/htdocs/210/214/scopes/temtm.htm>

2. Azo Colorants

Azo compounds are organic chemicals used to dye or “color” many products such as plastics, textiles, paints, inks, etc. The Azo Colorants category of substances is composed of the following substances only:

Substance	CAS Number
4-amino azobenzene	60-09-3
O-anisidine or 2-methoxyaniline	90-04-0
beta-Naphthylamine	91-59-8
3,3'-Dichlorobenzidine	91-94-1
4-Aminobiphenyl	92-67-1
Benzidine	92-87-5
o-Toluidine	95-53-4
p-Chloro-o-toluidine	95-69-2
2,4-Diaminotoluene	95-80-7
C.I. Solvent Yellow 3 OR o-Aminoazotoluene	97-56-3
5-Nitro-o-toluidine OR 2-Amino-4-nitrotoluene	99-55-8
4,4'-Methylenebis (2-chloroaniline) OR MBOCA	101-14-4
4,4'-Methylenedianiline OR 4,4'-Diaminodiphenylmethane	101-77-9
4,4'-Diaminodiphenyl ether (4,4'-Oxydianiline)	101-80-4
p-Chloroaniline	106-47-8
3,3'-Dimethoxybenzidine	119-90-4
3,3'-Dimethylbenzidine (ortho-Tolidine)	119-93-7
p-Cresidine OR 5-Methyl-o-Anisidine	120-71-8
2,4,5-Trimethylaniline	137-17-7
4,4'-Thiodianiline	139-65-1
2,4-Diaminoanisole	615-05-4
3,3'-Dimethyl-4,4'-diaminodiphenylmethane OR 4,4'-Methylene bis (2-methylaniline)	838-88-0

SW-846 contains methods for the following:

Substance	CAS #	Extraction/ Cleanup Method	Analytical Method(s)
4-amino azobenzene	60-09-3	Not Given	8270C
O-anisidine or 2-methoxyaniline	90-04-0	Not Given	8270C
2-Naphthylamine	91-59-8	3640A	8270C
3,3'-Dichlorobenzidine	91-94-1	3640A	8270C, 8325
4-Aminobiphenyl	92-67-1	3640A	8270C
Benzidine	92-87-5	3640A	8270C, 8325
2-Toluidine (o-Toluidine)	95-53-4	3640A	5031, 8015B, 8260B, 8270C
2,4-Diaminotoluene (Toluene-2,4-diamine)	95-80-7	3640A	8270C
5-Nitro-o-toluidine	99-55-8	3640A	8270C
4,4'-Methylenebis (2-chloroaniline)	101-14-4	3640A	8270C
4,4'-Oxydianiline	101-80-4	3640A	8270C
4-Chloroaniline	106-47-8	3640A	8131, 8270C, 8410
3,3'-Dimethoxybenzidine	119-90-4	3640A	8270C, 8325
3,3'-Dimethylbenzidine	119-93-7	3640A	8270C, 8325
p-Cresidine	120-71-8	3640A	8270C
2,4,5-Trimethylaniline	137-17-7	3640A	8270C

As you can see in the table above, using the 3640A Extraction/Cleanup method followed by the 8270C Gas Chromatograph/Mass Spectrophotometry method is the preferred method for most of the Azo Colorants. There are six Azo Colorants that SW-846 does not address:

Substance	CAS Number
p-Chloro-o-toluidine	95-69-2
C.I. Solvent Yellow 3 OR o-Aminoazotoluene	97-56-3
4,4'-Methylenedianiline OR 4,4'-Diaminodiphenylmethane	101-77-9
4,4'-Thiodianiline	139-65-1
2,4-Diaminoanisole	615-05-4
3,3'-Dimethyl-4,4'-diaminodiphenylmethane OR 4,4'-Methylene bis (2-methylaniline)	838-88-0

It is unknown if the methods 3640A, followed by 8270C will analyze these six substances.

3. Cadmium/Cadmium Compounds

For cadmium, we are interested in the total cadmium content of the product. We are not looking for speciation. Therefore, a digestion method for solid materials and an analysis technique are needed.

Digestion methods:

- 3050B is an acid digestion method.
- 3051 is a microwave-assisted acid digestion method (faster than 3050B)
- 3052 is a microwave-assisted acid digestion method developed specifically for siliceous and organic matrices. This is a much more aggressive digestion method that is designed for **total dissolution** of the material. For

our purposes, this would be the preferred digestion method.

ICP Analytical methods:

- 6010B uses ICP-AES (Inductively Coupled Plasma - Atomic Emission Spectrometry)
- 6020 uses ICP-MS (Inductively Coupled Plasma - Mass Spectrometry)

Atomic Absorption Analytical methods:

- 7130 uses FAAS - Flame, or Direct Aspiration Atomic Absorption Spectrometry
- 7131A uses GFAAS - Graphite Furnace Atomic Absorption Spectrometry

Either analytical method (ICP or AAS) is sufficient for our purposes. The choice of analytical technique will depend on your laboratory's equipment and the concentration of the substance in the product.

4. Hexavalent Chromium/Hexavalent Chromium Compounds

For hexavalent chromium, we are looking for speciation. The techniques that are used to digest the sample should extract only hexavalent chromium. They should not result in the following:

- reduction of hexavalent chromium to trivalent chromium;
- oxidation of trivalent chromium to hexavalent chromium; or
- oxidation of base metal chromium to hexavalent chromium.

The extraction method should solubilize all forms of hexavalent chromium in the sample. The digestion technique recommended by EPA is the Method 100.1 (Alkaline

Digestion using a manganese phosphate buffer to suppress oxidation of the trivalent chromium.

The potential analytical techniques are:

- 7195: Co-precipitation with lead chromate, followed by analysis with either Flame (7190) or Furnace (7191) Atomic Absorption Spectrophotometry
- 7196: Colorimetric reaction with diphenylcarbazide, followed by UV-VIS measurement
- 7197: Chelation with APDC, extraction with MIBK, and Flame Atomic Absorption Spectrophotometry (7190)
- 7198: Differential Pulse Polarography
- 7199: Ion Chromatography

The choice of analytical technique will depend on your lab's equipment, and the concentration of hexavalent chromium in your sample (or the desired detection limit).

5. Lead/Lead Compounds/Lead Alloys

For lead, we are interested in the total lead content of the product. We are not looking for speciation. Therefore, a digestion method for solid materials and an analysis technique is needed.

Digestion methods:

- 3050B is an acid digestion method.
- 3051 is a microwave-assisted acid digestion method (faster than 3050B)
- 3052 is a microwave-assisted acid digestion method developed specifically for siliceous and organic matrices. This is a much more aggressive digestion method that is designed for **total dissolution** of the material. For our purposes, this would be the preferred digestion method.

ICP Analytical methods:

- 6010B uses ICP-AES (Inductively Coupled Plasma - Atomic Emission Spectrometry)
- 6020 uses ICP-MS (Inductively Coupled Plasma - Mass Spectrometry)

Atomic Absorption Analytical methods:

- 7420 uses FAAS - Flame, or Direct Aspiration Atomic Absorption Spectrometry
- 7421 uses GFAAS - Graphite Furnace Atomic Absorption Spectrometry

Either analytical method (ICP or AAS) is sufficient for our purposes. The choice of analytical technique will depend on your laboratory's equipment and the concentration of the substance in the product.

6. Mercury/Mercury Compounds/Mercury Alloys

For mercury, we are interested in the total mercury content of the product. We are not looking for speciation. Therefore, a digestion method for solid materials and an analysis technique is needed.

Digestion methods:

- 3051 is a microwave-assisted acid digestion method (faster than 3050B)
- 3052 is a microwave-assisted acid digestion method developed specifically for siliceous and organic matrices. This is a much more aggressive digestion method that is designed for **total dissolution** of the material. For our purposes, this would be the preferred digestion method.

ICP Analytical methods:

- 6010B uses ICP-AES (Inductively Coupled Plasma - Atomic Emission Spectrometry)

Atomic Absorption Analytical methods:

- 7471A uses GFAAS - Atomic Absorption Spectrometry with absorption cell

Either analytical method (ICP or AAS) is sufficient for our purposes. The choice of analytical technique will depend on your laboratory's equipment and the concentration of the substance in the product.

7. Ozone Depleting Substances (CFCs, HCFCs, HBFCs, carbon tetrachloride, etc.)

There are very few analytical techniques for Ozone Depleting Substances. In general, these are not substances that are expected to be "in" electronic products. In the past, some electronic products were manufactured using some ODSs, but that practice has declined greatly. ODSs are the only substance group where it is important to know if the product was manufactured using ODSs (with direct contact between the ODS and the product).

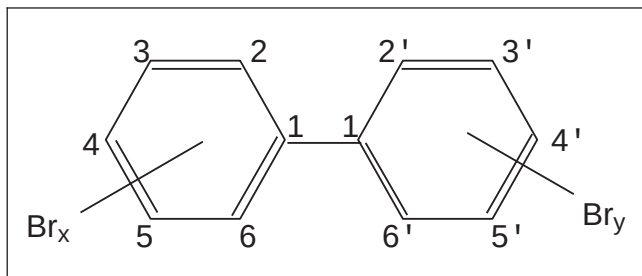
The ODSs where extraction or analytical methods were found are:

ODS	CAS Number	Extraction Method	Analytical Method
Trichlorofluoromethane (CFC-11)	75-69-4	Not found	8021B, 8261
Dichlorodifluoromethane (CFC-12)	75-71-8	5021, 5030	8021B, 8260, 8261
1,1,2-trichloro-1,2,2-trifluoroethane	76-13-1	5030	8260
Carbon tetrachloride	56-23-5	5021	Not found
1,1,1-Trichloroethane	71-55-6	Not found	8021B
Bromomethane	74-83-9	5021	8260B, 8261
Dibromofluoromethane	No CAS # found	Not found	8260B

8. Polybrominated Biphenyls (PBBs)

Polybrominated Biphenyls are a class of chemical compounds in which 1-10 bromine atoms are attached to the biphenyl molecule. The use of the term "poly" is a misnomer. If a biphenyl has only one bromine atom, it is a monobrominated biphenyl.

be classified as a PBB. This definition is the same as is used in the Toxic Release Inventory Reporting program for the US EPA. See the following figure:



It can be seen from the figure that the generic formula for PBBs is:

$$C_{12}H_{(10-x-y)}Br_{(x+y)}, \text{ where } x + y \text{ ranges from 1 to 10}$$

There are 209 possible PBB compounds (known as “cogeners”), all of which are required to be declared. Only 101 of these compounds have CAS numbers. There are no analytical methods for PBBs in SW-846. However, there are two reports available on the internet with analytical methods described:

1. <http://www.atsdr.cdc.gov/toxprofiles/tp68.html>
2. <http://www.inchem.org/documents/ehc/ehc/ehc152.htm>

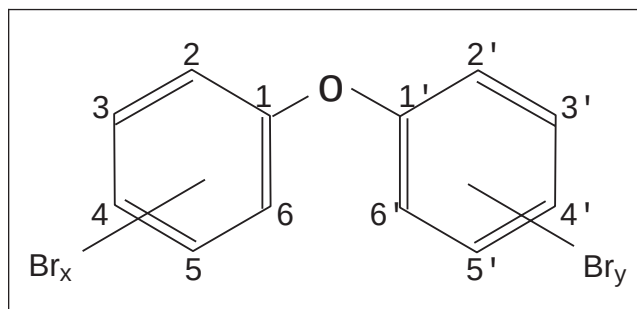
In general, PBBs are typically extracted with organic solvents, then cleaned up using Florisil (or other means such as gel permeation, silica gel, activated carbon, etc.) to remove other matrix interferences and to fractionate the samples. The identification and quantitation is usually by Gas chromatography. Capillary or high resolution gas chromatography columns capable of separating a substantial portion of the cogeners are necessary, and GC detectors possessing high selectivity and sensitivity for PBBs are required. One of the problems with the analysis of PBBs is the small number of certified reference standards that are available for the various cogeners. The CDC report gives some analytical information and references in Chapter 7.⁵

9. Polybrominated Diphenyl ethers (PBDEs)

Polybrominated Diphenyl Ethers are very similar to PBBs with the addition of an ether bridge between the two aromatic rings:

It can be seen from the figure that the generic formula for PBDEs is:

$$C_{12}H_{(10-x-y)}Br_{(x+y)}O, \text{ where } x + y \text{ ranges from 1 to 10}$$



There are also 209 possible PBDE compounds (known as “cogeners”), all of which are required to be declared. As with PBBs, there are no analytical methods for PBDEs in SW-846. However, the same reports that have methods for PBBs also have PBDE methods. There is also an Inchem report on PBDEs with analytical methods. Inchem is web-site operated by the International Programme on Chemical Safety (www.inchem.org).

1. <http://www.atsdr.cdc.gov/toxprofiles/tp68.html>
2. <http://www.inchem.org/documents/ehc/ehc/ehc152.htm> (PBBs and PBDEs)
3. <http://www.inchem.org/documents/ehc/ehc/ehc162.htm> (PBDEs only)

As with PBBs, the PBDEs are typically extracted with organic solvents, then cleaned up using Florisil (or other means such as gel permeation, silica gel, activated carbon, etc) to remove other matrix interferences and to fractionate the samples. The identification and quantitation is usually by Gas chromatography. Capillary or high resolution gas chromatography columns capable of separating a substantial portion of the cogeners are necessary, and GC detectors possessing high selectivity and sensitivity for PBBs are required. One of the problems with the analysis of PBDEs is the small number of certified reference standards that are available for the various cogeners. The CDC report gives some analytical information and references in Chapter 7.⁶

10. Polychlorinated Biphenyls (PCBs)

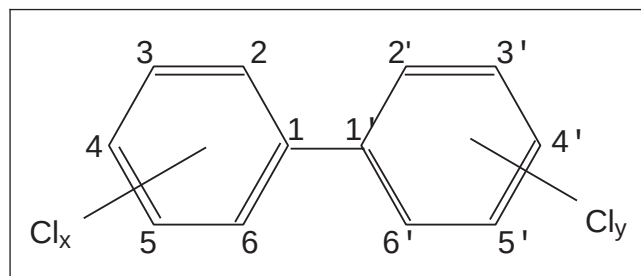
Polychlorinated Biphenyls are a class of chemical compounds in which 2-10 chlorine atoms are attached to the biphenyl molecule. See the following figure:

It can be seen from the figure that the generic formula for PCBs is:

$$C_{12}H_{(10-x-y)}Cl_{(x+y)}, \text{ where } x \text{ and } y \text{ range from 1 to 5}$$

5. “ASTDR Toxicological Profile for Polybrominated Biphenyls and Polybrominated Diphenyl Ethers (PBBs and PBDEs)”, Draft for Public Comment, Chapter 7, pp 367-368 September 2002, <http://www.atsdr.cdc.gov/toxprofiles/tp68.html>

6. “ASTDR Toxicological Profile for Polybrominated Biphenyls and Polybrominated Diphenyl Ethers (PBBs and PBDEs)”, Draft for Public Comment, Chapter 7, pp 367-368 September 2002, <http://www.atsdr.cdc.gov/toxprofiles/tp68.html>



There are 209 possible PCB compounds (known as “cogeners”), all of which are required to be declared. All of these compounds have CAS numbers. There are analytical methods for PCBs in SW-846, plus there are two reports available on the internet with analytical methods described:

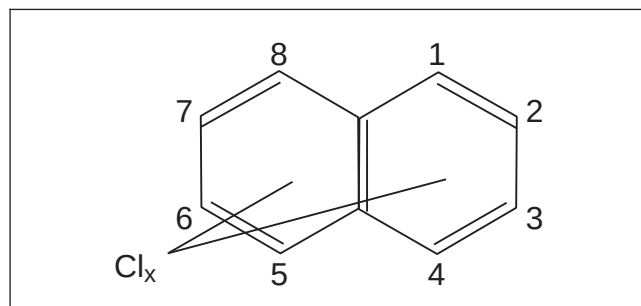
1. <http://www.atsdr.cdc.gov/toxprofiles/tp17.html>
2. <http://www.inchem.org/documents/ehc/ehc/ehc002.htm>

In general, PCBs are typically extracted with organic solvents, then cleaned up using Florisil (or other means such as gel permeation, silica gel, activated carbon, etc) to remove other matrix interferences and to fractionate the samples. The identification and quantitation is usually by Gas chromatography. Capillary or high resolution gas chromatography columns capable of separating a substantial portion of the congeners are necessary, and GC detectors possessing high selectivity and sensitivity for PCBs are required. The CDC report gives some analytical information and references in Chapter 7.⁷

SW-846 lists two methods (8082 and 8275A) for PCB analysis.

11. Polychlorinated Naphthalenes (PCNs) (with more than three chlorine atoms)

Polychlorinated Naphthalenes are a class of chemical compounds in which 2-8 chlorine atoms are attached to the naphthalene molecule. See the following figure:



It can be seen from the figure that the generic formula for PCNs is:

$$C_{12}H_{(8-x)}Cl_x, \text{ where } x \text{ ranges from 1 to 8}$$

There are 75 possible PCN compounds (known as “cogeners”). Only those containing more than three chlorine atoms are required to be declared. There are 63 individual compounds, plus many mixtures. Halowaxes, Nibren Waxes, Seekay Waxes, and Clonacire Waxes are proprietary mixes of PCNs.

There is an analytical method (8081) for only a few PCNs in SW-846, plus there is a report available on the internet with analytical methods described (see Section 3 - Analytical Methods):

<http://www.inchem.org/documents/cicads/cicads/cicad34.htm>

12. Radioactive Substances

There are three common types of nuclear radiation - alpha, beta, and gamma radiation. Alpha radiation is when a particle equivalent to the nucleus of a helium atom (two neutrons and two protons) is emitted. Alpha radiation can be stopped by a sheet of paper. Beta radiation is the emission of high-speed electrons. It takes 6 mm of aluminium to stop beta radiation. Gamma radiation is the emission of high-energy photons, and it takes several millimetres of lead or some other dense material to stop these photons.

There are other types of radiation mechanisms. In Electron Capture, the nucleus of a radioactive atom captures one of its own electrons and converts a neutron to a proton, while emitting a neutrino. Elements that decay by this mechanism are Cobalt-57; Strontium-80, -81, -82, -83, and -85.

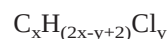
The radioactive elements all decay differently, and some isotopes of the same element decay by different radiation mechanisms. Thus, an analysis of the particular element is not necessary to determine if radioactive compounds exist. The best method to determine the general level of radioactivity would be a Geiger Counter, which will recognize the three common types of radiation.

There are a few analytical tests for radioactive substances listed in SW-846:

Gross Alpha and Beta Emissions	9310
Alpha-Emitting Radium Isotopes	9315
Radium-228	9320

13. Shortchain Chlorinated Paraffins

Chlorinated Paraffins can be divided into multiple categories. The category required to be “declared” is known as “Shortchain Chlorinated Paraffins,” and is defined by:



Where: $x = 10 \text{ to } 13$,
 $y = 3 \text{ to } 12$, and

7. “ASTDR Toxicological Profile for Polychlorinated Biphenyls (PCBs)”, Chapter 7, pp 595-613, November 2000, <http://www.atsdr.cdc.gov/toxprofiles/tp17.html>

the average chlorine content ranges from 40 to 70 percent with the limiting molecular formulas set at $C_{10}H_{19}Cl_{13}$ and $C_{13}H_{16}Cl_{12}$.

There are no analytical methods listed in SW-846 for Shortchain Chlorinated Paraffins. However, a report is available on the internet with analytical methods described (see Section 2.3.2 - Analytical Methods, Table 5):

<http://www.inchem.org/documents/ehc/ehc/ehc181.htm>

There are enormous difficulties with the analysis of Short-chain Chlorinated Paraffins, due to interferences. Also, it is almost impossible at this time to quantify for individual compounds.

14. Tributyl Tin (TBT) and Triphenyl Tin (TPT)

There are no analytical methods listed in SW-846 for Tributyl Tin or Triphenyl Tin. However, there are some reports available on the internet with analytical methods described:

- <http://www.shimadzu.com.br/analitica/aplicacoes/GC/G190.pdf> – This is a method paper published by Shimadzu, using Capillary GC-FPD.
- <http://www.chem.agilent.com/scripts/peakprint.asp?lPage=1186> – This is a method paper published by Agilent, using GC/ICP-MS.
- <http://www.restekcorp.com/restek/images/external/59550.pdf> – This is a method paper published by Restek Corporation.
- <http://www.ecy.wa.gov/pubs/0309043.pdf>. This document is the Sediment Sampling and Analysis Plan Appendix from the Washington State Department of Ecology. On page 23 of the document, the recommended method for organotin complexes is listed as Krone (1989). The full citation is Krone, C. A., D. W. Brown, D. G. Burrows, R. G. Bogar, S. L. Chan and U. Varanasi, 1989, "A Method for the Analysis of Butyltin Species and the Measurement of Butyltins in Sediment and English Sole Livers from Puget Sound," *Marine Environmental Research*, 27:1-18.

15. Tributyl Tin Oxide (TBTO)

There are no analytical methods listed in SW-846 for Tributyl Tin Oxide. Also, no other methods have been found on the internet. It is likely that the methods listed for TBT above will also quantify the TBTO. This will need to be checked with your analytical lab experts.

16. Antimony/Antimony Compounds

For antimony, we are interested in the total antimony content of the product. We are not looking for speciation. Therefore, a digestion method for solid materials and an analysis technique is needed.

Digestion methods:

- 3050B is an acid digestion method.

- 3051 is a microwave-assisted acid digestion method (faster than 3050B)
- 3052 is a microwave-assisted acid digestion method developed specifically for siliceous and organic matrices. This is a much more aggressive digestion method that is designed for **total dissolution** of the material. For our purposes, this would be the preferred digestion method.

ICP Analytical methods:

- 6010B uses ICP-AES (Inductively Coupled Plasma - Atomic Emission Spectrometry)
- 6020 uses ICP-MS (Inductively Coupled Plasma - Mass Spectrometry)

Atomic Absorption Analytical methods:

- 7040 uses FAAS - Flame, or Direct Aspiration Atomic Absorption Spectrometry
- 7041 uses GFAAS - Graphite Furnace Atomic Absorption Spectrometry
- 7062 also uses FAAS, but adds borohydride reduction. This method is useful when the product contains high concentrations of copper, cobalt, iron, mercury, and nickel. This method is also suitable for arsenic analysis. In electronic products, this may be the preferred method.

Either analytical method (ICP or AAS) is sufficient for our purposes. The choice of analytical technique will depend on your laboratory's equipment and the concentration of the substance in the product.

17. Arsenic/Arsenic Compounds

For arsenic, we are interested in the total arsenic content of the product. We are not looking for speciation. Therefore, a digestion method for solid materials and an analysis technique is needed.

Digestion methods:

- 3050B is an acid digestion method.
- 3051 is a microwave assisted acid digestion method (faster than 3050B)
- 3052 is a microwave assisted acid digestion method developed specifically for siliceous and organic matrices. This is a much more aggressive digestion method that is designed for **total dissolution** of the material. For our purposes, this would be the preferred digestion method.

ICP Analytical methods:

- 6010B uses ICP-AES (Inductively Coupled Plasma - Atomic Emission Spectrometry)
- 6020 uses ICP-MS (Inductively Coupled Plasma - Mass Spectrometry)

Atomic Absorption Analytical methods:

- 7060A uses GFAAS - Graphite Furnace Atomic Absorption Spectrometry



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- 7061A uses FAAS, but adds gaseous hydride generation. This method is useful only when the sample does not contain high concentrations of chromium, copper, mercury, nickel, silver, cobalt, or molybdenum.
- 7062 also uses FAAS, but adds borohydride reduction. This method is useful when the product contains high concentrations of copper, cobalt, iron, mercury, and nickel. This method is also suitable for antimony analysis. In electronic products, this may be the preferred method.

Either analytical method (ICP or AAS) is sufficient for our purposes. The choice of analytical technique will depend on your laboratory's equipment and the concentration of the substance in the product.

18. Beryllium/Beryllium Compounds/Beryllium Alloys

For beryllium, we are interested in the total beryllium content of the product. We are not looking for speciation. Therefore, a digestion method for solid materials and an analysis technique is needed.

Digestion methods:

- 3050B is an acid digestion method.
- 3051 is a microwave-assisted acid digestion method (faster than 3050B)
- 3052 is a microwave-assisted acid digestion method developed specifically for siliceous and organic matrices. This is a much more aggressive digestion method that is designed for **total dissolution** of the material. For our purposes, this would be the preferred digestion method.

ICP Analytical methods:

- 6010B uses ICP-AES (Inductively Coupled Plasma - Atomic Emission Spectrometry)
- 6020 uses ICP-MS (Inductively Coupled Plasma - Mass Spectrometry)

Atomic Absorption Analytical methods:

- 7090 uses FAAS - Flame, or Direct Aspiration Atomic Absorption Spectrometry
- 7091 uses GFAAS - Graphite Furnace Atomic Absorption Spectrometry

Either analytical method (ICP or AAS) is sufficient for our purposes. The choice of analytical technique will depend on your laboratory's equipment and the concentration of the substance in the product.

19. Bismuth/Bismuth Compounds/Bismuth Alloys

There are no SW-846 test methods for bismuth. However, internet searches show that possible methods are:

EPA 200.7
EPA 200.8
EPA 6010B
EPA 6020
SM 3500Bi

20. Brominated Flame Retardants (other than PBBs or PBDEs)

Of the list of 66 Brominated Flame Retardants (BFRs) provided in the Joint Industry Guide, only 2,4,6-Tribromophenol has an analytical method (8270C) listed in SW-846. Although a cleanup/extraction step isn't given, it is likely that the 3640A Gel Permeation Cleanup step would work, since that is the extraction step listed for many other substances analyzed by 8270C.

For the remainder of the BFRs, it is unlikely that there are any "direct" methods of analyzing for their content in electronic products. However, there are some methods that can be used to determine the bromine content of a plastic.

The generally accepted test method is IEC-61189-2C12, which determines both Chlorine and Bromine in printed circuit board materials.

Discussions with some BFR manufacturers revealed that several other methods can also be used to determine the bromine content of a plastic.

For PVC, the plastic can be dissolved in tetrahydrofuran, which is then filtered and evaporated. The remaining material is then analyzed by GC or GC/MS.

For most types of plastic, the Schöniger Combustion Method can be used. This is an older (developed in the 1950's by Wolfgang Schöniger) method in which a small sample is combusted in an oxygen atmosphere. The resulting ash is dissolved (in either sodium hydroxide or water), and analyzed for inorganic ion content. Some researchers have used Ion Selective Electrodes (ISEs) for bromine and chlorine determinations, while others may use a titration method.

Check with your analytical laboratory to see if they can do the Schöniger Combustion Method. If not, microanalytical labs in universities often use this method. For example, the University of Massachusetts at Amherst (USA) and the University of Manchester (UK) list this analysis technique on their Chemistry department web pages. Check with your local university to see if they are able to provide this analysis.

21. Nickel/Nickel Compounds/Nickel Alloys

For nickel, we are interested in the total nickel content on the external surfaces of the product. We are not interested in nickel platings that are covered by other surface finishes - only the nickel that has the potential for being in significant dermal contact. It is considered extremely unlikely that any nickel species other than plated nickel metal on the outside of an electronic product's case will have a potential for significant dermal contact. Therefore, we are not looking for speciation. Therefore, a digestion method for solid materials and an analysis technique is needed.

Digestion methods:

- 3050B is an acid digestion method.
- 3051 is a microwave-assisted acid digestion method (faster than 3050B)
- 3052 is a microwave-assisted acid digestion method developed specifically for siliceous and organic matrices. This is a much more aggressive digestion method that is designed for **total dissolution** of the material. For our purposes, this would be the preferred digestion method.

ICP Analytical methods:

- 6010B uses ICP-AES (Inductively Coupled Plasma - Atomic Emission Spectrometry)
- 6020 uses ICP-MS (Inductively Coupled Plasma - Mass Spectrometry)

Atomic Absorption Analytical methods:

- 7520 uses FAAS - Flame, or Direct Aspiration Atomic Absorption Spectrometry
- 7521 uses GFAAS - Graphite Furnace Atomic Absorption Spectrometry

Either analytical method (ICP or AAS) is sufficient for our purposes. The choice of analytical technique will depend on your laboratory's equipment and the concentration of the substance in the product.

22. Certain Phthalates

The category of "Certain Phthalates" indeed refers to a few certain phthalates. The substances covered in this category are:

Substance Name	CAS Number
Bis (2-ethylhexyl) phthalate (DEHP)	117-81-7
Dibutyl phthalate (DBP)	84-74-2
Bis (2-methoxyethyl) phthalate (DBP)	117-82-8

SW-846 lists analytical techniques for all of these phthalates. Acceptable analytical methods are 3640A (Gel Permeation Cleanup) followed by either 8061A (GC with Electron Capture Detection), 8270C (Semivolatiles by GC/MS), or 8410 (GC/Fourier Transform-Infrared Spectrometry).

23. Selenium/Selenium Compounds/Selenium Alloys

For selenium, we are interested in the total selenium content of the product. We are not looking for speciation. Therefore, a digestion method for solid materials and an analysis technique is needed.

Digestion methods:

- 3050B is an acid digestion method.
- 3051 is a microwave-assisted acid digestion method (faster than 3050B)

- 3052 is a microwave-assisted acid digestion method developed specifically for siliceous and organic matrices. This is a much more aggressive digestion method that is designed for **total dissolution** of the material. For our purposes, this would be the preferred digestion method.

ICP Analytical methods:

- 6010B uses ICP-AES (Inductively Coupled Plasma - Atomic Emission Spectrometry)
- 6020 uses ICP-MS (Inductively Coupled Plasma - Mass Spectrometry)

Atomic Absorption Analytical methods:

- 7740 uses FAAS - Graphite Furnace Atomic Absorption Spectrometry
- 7741A uses GFAAS - Flame, or Direct Aspiration Atomic Absorption Spectrometry with Gaseous Hydride
- 7742 - Flame, or Direct Aspiration Atomic Absorption Spectrometry with Borohydride Reduction

Either analytical method (ICP or AAS) is sufficient for our purposes. The choice of analytical technique will depend on your laboratory's equipment and the concentration of the substance in the product.

24. Vinyl Chloride Polymer (PVC)

The presence of PVC can be determined using the Beilstein Test. In general, a hot copper wire is touched to the plastic of concern to get some plastic melted onto the wire. The wire is then placed in a flame. If the plastic is PVC (or CPVC or another chlorinated plastic), the fire will glow green, due to the interaction between the chlorine atoms from the plastic and the copper wire.

This experiment is detailed at:

<http://www.hc-sc.gc.ca/hecs-sesc/cps/pdf/laboratory/c-28.pdf>

If a quantitative value for the PVC content is needed, a lab with experience in PVC analysis will need to be contacted. A method is described in:

http://europa.eu.int/comm/environment/waste/studies/pvc/landfill_annexes.pdf (see Annex III, "Chemical Analysis")

In this method, the plastic was dissolved in tetrahydrofuran, and then filtered. The tetrahydrofuran was then dissolved, leaving only tetrahydrofuran-soluble components (i.e., PVC-polymer, plasticizers, and organic additives). The chlorine content was then analyzed by X-ray-fluorescence analysis. The chlorine content was used to determine the PVC content of the plastic.

Analytical Method Summary

A summary of the analytical techniques discussed above is presented in Appendix A1.

Appendix A1 – Table of Chemical Lists and Laboratory Analytical Methods

Substance Category	Substance Name	Substance CAS #	SW-846 Methods		Other Methods	Comments
			Extraction/ Digestion	Analysis		
Asbestos	Asbestos	1332-21-4	Not Applicable	Polarized Light Microscopy		Analytical methods can only provide a "rough" concentration.
	Crocidolite	12001-28-4				
	Chrysotile	12001-29-5		Or		
	Amosite (Grunerite)	12172-73-5				
	Actinolite	77536-66-4		Transmission Electron Microscopy		
	Anthophyllite (or Anthophyllite)	77536-67-5				
	Tremolite	77536-68-6				
Azo Colorants	4-amino azobenzene	60-09-3	3640A or 3540B	8270C		The EU has listed these substances as Azo Colorants.
	O-anisidine or 2-methoxyaniline	90-04-0	3640A or 3540B	8270C		
	beta-Naphthylamine	91-59-8	3640A or 3540B	8270C		The EU has also directed that analytical methods be developed for the Azo Colorants (see 2002/61/EC).
	3,3'-Dichlorobenzidine	91-94-1	3640A or 3540B	8270C or 8325		
	4-Aminobiphenyl	92-67-1	3640A or 3540B	8270C		
	Benzidine	92-87-5	3640A or 3540B	8270C or 8325		
	o-Toluidine	95-53-4	3640A or 3540B	5031, 8015B, 8260B, or 8270C		
	p-Chloro-o-toluidine	95-69-2	Not Found	Not Found		
	2,4-Diaminotoluene	95-80-7	3640A or 3540B	8270C		
	C.I. Solvent Yellow 3 OR o-Aminoazotoluene	97-56-3	Not Found	Not Found		
	5-Nitro-o-toluidine OR 2-Amino-4-nitrotoluene	99-55-8	3640A or 3540B	8270C		
	4,4'-Methylenebis (2-chloroaniline) OR MBOCA	101-14-4	3640A or 3540B	8270C		
	4,4'-Methylenedianiline OR 4,4'-Diaminodiphenylmethane	101-77-9	Not Found	Not Found		
	4,4'-Diaminodiphenyl ether (4,4'-Oxydianiline)	101-80-4	3640A or 3540B	8270C		
	p-Chloroaniline	106-47-8	3640A or 3540B	8131, 8270C, or 8410		
	3,3'-Dimethoxybenzidine	119-90-4	3640A or 3540B	8270C or 8325		
	3,3'-Dimethylbenzidine (ortho-Tolidine)	119-93-7	3640A or 3540B	8270C or 8325		
	p-Cresidine OR 5-Methyl-o-Anisidine	120-71-8	3640A or 3540B	8270C		
	2,4,5-Trimethylaniline	137-17-7	3640A or 3540B	8270C		
	4,4'-Thiodianiline	139-65-1	Not Found	Not Found		
	2,4-Diaminoanisole	615-05-4	Not Found	Not Found		
	3,3'-Dimethyl-4,4'-diaminodiphenylmethane OR 4,4'-Methylene bis (2-methylaniline)	838-88-0	Not Found	Not Found		

Substance Category	Substance Name	Substance CAS #	SW-846 Methods		Other Methods	Comments
			Extraction/ Digestion	Analysis		
Cadmium & Cadmium Compounds		7440-43-9	3050B, 3051, or 3052	ICP - 6010B or 6020 AAS - 7130 or 7131A		
Hexavalent Chromium & Hexavalent Chromium Compounds		18540-29-9	3060A	7195, 7196A, 7197, 7198, 7199		The digestion method may possibly cause any trivalent or zero-valent chromium to oxidize to hexavalent chromium. Be sure to have your lab verify that the digestion method did not cause oxidation (this is explained in the 3060A method).
Lead, Lead Compounds, & Lead Alloys		7439-92-1	3050B, 3051, or 3052	ICP - 6010B or 6020 AAS - 7420 or 7421		
Mercury, Mercury Compounds, & Mercury Alloys		7439-97-6	3051 or 3052	ICP - 6010B, AAS - 7471A		
OZONE DEPLETING SUBSTANCES:						
Ozone Depleting Substances/ Isomers	Trichlorofluoromethane (CFC-11)	75-69-4	Not found	8021B, 8261		
	Dichlorodifluoromethane (CFC-12)	75-71-8	5021, 5030	8021B, 8260, 8261		
	1,1,2-trichloro-1,2,2-trifluoroethane	76-13-1	5030	8260		
	Dichlorotetrafluoroethane (CFC 114)	76-14-2	Not Found	Not Found		
	Monochloropentafluoroethane (CFC 115)	76-15-3	Not Found	Not Found		
	Bromotrifluoromethane (Halon 1301)	75-63-8	Not Found	Not Found		
	Dibromotetrafluoroethane (Halon 2402)	124-73-2	Not Found	Not Found		
	Bromochlorodifluoromethane (Halon 1211)	353-59-3	Not Found	Not Found		
	Chlorotrifluoromethane (CFC-13)	75-72-9	Not Found	Not Found		
	Tetrachlorodifluoroethane (CFC-112)	76-12-0	Not Found	Not Found		
	1,2,3-Trichloropentafluoropropane	76-17-5	Not Found	Not Found		
	Pentachlorofluoroethane (CFC-111)	354-56-3	Not Found	Not Found		
	Trichlorotrifluoroethane (CFC 113)	354-58-5	Not Found	Not Found		
	Heptachlorofluoropropane (CFC-211)	422-78-6	Not Found	Not Found		
	Chloroheptafluoropropane (CFC-217)	422-86-6	Not Found	Not Found		
	Dichlorohexafluoropropane (CFC-216)	661-97-2	Not Found	Not Found		
	Trichloropentafluoropropane (CFC-215)	1599-41-3	Not Found	Not Found		
	1,1,1,3-Tetrachlorotetrafluoropropane	2268-46-4	Not Found	Not Found		
	CFC-213	2354-06-5	Not Found	Not Found		
	Hexachlorodifluoropropane (CFC-212)	3182-26-1	Not Found	Not Found		