IPC Material Declaration Guidelines Project

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The European Union has recently passed two Directives – the Waste Electrical and Electronic Equipment (WEEE) and the Restriction on the use of certain Hazardous Substances (RoHS). These directives have resulted in the current push for lead-free electronic equipment worldwide, and the requirements for suppliers to OEMs to supply "Material Declarations". IPC has joined together with members of EIA to develop guidelines for printed circuit board manufacturers and assemblers for completing Material Declarations. The guidelines will cover the substances to be reported, methods of calculation and analysis, and the format of the Material Declaration. The status of these guidelines will be presented.

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Background

The European Union (EU) has recently passed two pieces of legislation that will change the way electronic products are designed, manufactured, and disposed of. Directive 2002/96/EC on Waste Electrical and Electronic Equipment (or WEEE) directs EU member states to set up systems for managing the "takeback" and recycling of electronic products. It also directs manufacturers of electronic equipment to design products so that 65% of the mass of the products is reuseable or recyclable. An additional 10% of the product's mass needs to be "recoverable". An example of recovery would be burning for energy content capture.[1]

Directive 2002/95/EC on the Restriction of the use of certain Hazardous Substances in electrical and electronic equipment (or RoHS) bans the presence of lead, cadmium, mercury, hexavalent chromium, polybrominated biphenyls (PBBs), and polybrominated diphenyl ethers (PBDEs) in electronic products. This ban will take effect on July 1, 2006. Accompanying these two Directives are numerous regulations from countries located mainly in the EU that ban or restrict the presence of other substances.

The reaction from electronic product manufacturers has been to push their supply chains to start the transition of products made without these restricted substances. Since the list of restricted substances is expected to increase in the future, manufacturers are also requesting information on what substances their products are made of. Their requests have become known as "material declaration" requests. These requests usually contain a list of substances (which can be broken down into the categories of banned, restricted to certain uses, and others) with threshold values over which the substance is reportable. There is also usually a form to be completed for each unique product.

Material Declaration Standardization

In general, manufacturers have each developed their own list of substances and their own reporting forms. Some have requested that essentially 100% of a product's mass be declared, while others have a more selective substance list. The proliferation of substance lists and reporting forms has made it difficult for the suppliers to respond adequately. In February 2001, the Electronic Industries Alliance (EIA) issued a list of substances that its members agreed to, in order to bring some level of standardization to substance lists. However, it didn't cover enough companies to become a standard. EIA began a collaborative effort with European Industry Association (EICTA) and the Japanese Green Procurement Survey Standardization Initiative (JGPSSI) to standardize the list of substances globally. This effort has resulted in the "Joint Industry Guide" (available through Holly Evans of the EIA – hevans@eia.org).

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[1] Acceptable recovery operations are defined in Annex IIB in EU Directive 75/442/EEC.

The Joint Industry Guide lists 29 categories of substances that must be declared. The first fifteen categories (Level A Substances) represent substances that when used in electronics or electrical products, are subject to currently enacted legislation somewhere in the world that either:

a) Prohibits their use and/or marketing;
b) Restricts their use and/or marketing; or
c) Requires reporting or results in other regulatory effect.

The Level A Substances are:

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 14 categories (Level B Substances) are composed of materials and substances that the industry has determined are reportable because they meet one of the following criteria:

a) Precious materials/substances that provide economic value for end-of-life management purposes;
b) Materials/substances that are of significant environmental, health, or safety interest;
c) Materials/substances that would trigger hazardous waste management requirements; and
d) Materials/substances that could have a negative impact on end-of-life management.

The Level B substances are:

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. Copper/Copper Compounds/Copper Alloys
22. Gold/Gold Compounds/Gold Alloys

23. Magnesium
24. Nickel/Nickel Compounds/Nickel Alloys
25. Palladium/Palladium Compounds/Palladium Alloys
26. Certain Phthalates
27. Selenium/Selenium Compounds/Selenium Alloys
28. Silver/Silver Compounds/Silver Alloys
29. Vinyl Chloride Polymer (PVC)

The Joint Industry Guide also provides chemical lists to show what chemicals may contain Level A and/or Level B substances. For the substances which are metallic in nature (copper, beryllium, silver, etc.), it is not the mass of the metallic compound that is reportable, just the mass of the metal.

All of the substance categories have threshold levels, above which the substance is reportable. For some substances, a numerical value isn't given. Instead, the words "intentionally added" are shown. This is due to the fact that "lead-free", "cadmium-free", etc. have not been defined yet. There is speculation that they may be defined as less than 1000 ppm (by weight) AND not intentionally added. This will create the curious situation where a product with a low level of lead will not qualify as "lead-free" while a product with a greater lead concentration will. For example, if a company plates a small amount of eutectic solder onto a part to achieve certain properties, due to the intentional addition of lead, the part is not "lead-free". The lead concentration may be 50 ppm by weight, but it isn't "lead-free". Another manufacturer uses a certain alloy, of which lead is a contaminant. The manufacturer doesn't require the lead to be present – it is just a contaminant in the raw ore. As long as the lead concentration is less than 1000 ppm by weight, the part qualifies as "lead-free" even though it has more lead than the previous part.

IPC Material Declaration Guide

The Environment, Health, and Safety Committee of the IPC (the trade organization for the printed circuit board industry) began a team in April 2003 to assist its member companies in completing material declaration requests. The team is focused on developing guidelines on how to calculate the composition of a printed circuit board, how to analyze raw materials or products for composition data, and how to request material declaration data from members' supply chains. There is a Steering Team that oversees the work, which is occurring in two subteams – an Analysis subteam and a Calculation subteam.

Analysis Subteam

The Analysis Subteam's tasks are to identify as completely as possible the individual chemicals that make up the substance categories in the Joint Industry Guide, and identify analytical techniques for those chemicals. The Analytical Subteam has decided to focus on the analytical techniques that are published in EPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, which is the EPA Office of Solid Waste's official compendium of analytical and sampling methods that have been evaluated and approved for use in complying with the RCRA regulations[2]. These analytical techniques seem the most suited to the analysis of solid materials.

The Analysis Subteam has evaluated the list of available analyses in SW-846 and has included all relevant analytical techniques. However, there are no analytical techniques in SW-846 for many of the substance categories in the Joint Industry Guide. This is especially true of many organic substances. As an example, there are 22 listed Azo colorants in the Joint Industry Guide. There are analytical methods listed in SW-846 for 16 of these chemicals (see Table 1 for a list of Azo Colorants with methods listed in SW-846). There are no methods listed for the remaining 6 Azo Colorants (see Table 2).

This is a common situation throughout the IPC Material Declaration Guideline. However, this guideline is being developed as a voluntary standard, and will be updated on a regular basis. Any new analytical techniques will be incorporated into subsequent versions of the guidelines.

Where possible, non-SW-846 methods are being listed in the guidelines. The Analytical Subteam is actively pursuing other "approved" methods (e.g., DIN methods from Germany, ANSI methods, Japanese methods, etc.). As an example of alternative methods (and SW-846 methods), refer to Appendix 1.

The Analysis Subteam is also working to identify laboratories that can perform the required analyses. The Subteam is not qualifying or approving laboratories, just gathering potential laboratory contact information for use by members. As the guideline is updated laboratories can be added and removed from the list.

[2] EPA SW-846 is available online at: http://www.epa.gov/epaoswer/hazwaste/test/sw846.htm

Table 1. Azo Colorants with Methods listed in SW-846

Substance	CAS #	Extraction/ Cleanup Method	SW-846 Analytical Method(s)
4-amino azobenzene	60-09-3	Not Given, but 3640A should work	8270C
O-anisidine OR 2-methoxyaniline	90-04-0	Not Given, but 3640A should work	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

Table 2. Azo Colorants with no methods listed in SW-846.

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

Calculation Subteam

The Calculation Subteam is developing a method for calculating the material composition of a printed circuit board. The calculation method essentially follows the production process, using material composition data, process knowledge, and data files used to manufacture the product in order to add and subtract materials. An example of the Develop, Etch, and Strip process is provided in Appendix 2.

The Calculation Subteam is also including a section on the propagation of uncertainty and errors throughout the calculations for a material declaration.

Summary

In summary, the IPC Material Declaration team is creating a guideline document that will help its members complete material declaration requests from customers. The guideline will include methods of calculating the composition of printed circuit boards and information on analytical techniques that can be used to determine the composition of most electronic products. The draft of the guideline is expected to be completed in the spring of 2004, and begin circulation for approval.

<table>
<tr><th rowspan="2">Substance Category</th><th rowspan="2">Substance Name</th><th rowspan="2">Substance CAS #</th><th colspan="2">SW-846 Methods</th><th rowspan="2">Other Methods</th><th rowspan="2">Comments</th></tr>
<tr><th>Extraction/ Digestion</th><th>Analysis</th></tr>
<tr><td rowspan="3">Organo Tin Complexes</td><td>Tributyl Tin (TBT)</td><td>Not Found</td><td>Not Found</td><td>Not Found</td><td colspan="2" rowspan="3">Please see either of the following:

1. http://www.shimadzu.com.br/analitica/aplicacoes/GC/G190.pdf . This is a method paper published by Shimadzu, using Capillary GC-FPD.
2. http://www.chem.agilent.com/scripts/peakprint.asp?lPage=1186. This is a method paper published by Agilent, using GC/ICP-MS.
3. http://www.restekcorp.com/restek/images/external/59550.pdf . This is a method paper published by Restek Corporation.
4. 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.</td></tr>
<tr><td>Triphenyl Tin (TPT)</td><td>Not Found</td><td>Not Found</td><td>Not Found</td></tr>
<tr><td>Tributyl Tin Oxide (TBTO)</td><td>56-35-9</td><td>Not Found</td><td>Not Found</td></tr>
<tr><td colspan="2">Cadmium & Cadmium Compounds</td><td>7440-43-9</td><td>3050B, 3051, or 3052</td><td>ICP – 6010B or 6020
AAS – 7130 or 7131A</td><td></td><td></td></tr>
<tr><td colspan="2">Hexavalent Chromium & Hexavalent Chromium Compounds</td><td>18540-29-9</td><td>3060A</td><td>7195, 7196A, 7197, 7198, 7199</td><td></td><td>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).</td></tr>
</table>

APPENDIX 1. Examples of Analytical Method Information Presented in Tabular Format

APPENDIX 2. Example Calculations for Develop, Etch, and Strip Process

In the Develop, Etch, and Strip (DES) process, the inner layer circuits are etched. The data that is needed for the MD calculation is the area of foil remaining on each layer after etching. This data should be available from the original PCB design data. The Develop and Strip processes do not add or subtract anything from the layers that will remain on the final product. No MD-pertinent calculations are needed for these processes.

Assume that we have gotten the following data from our Front End Engineering group:

Table A-1 Foil Remaining after Etch[3]

Layer	Foil Area Remaining (in^2)	Foil Thickness (after microetch) (in)
1	186.34	Not etched at DES
2	37.45	0.00136
3	181.41	0.00136
4	27.07	0.00136
5	26.14	0.00136
6	181.41	0.00136
7	181.41	0.00136
8	28.35	0.00136
9	27.72	0.00136
10	181.41	0.00136
11	104.39	0.00136
12	183.61	Not etched at DES

From this, it is simple to calculate the remaining foil weight. For example, for Layer 2:

Eq A-1
$$\left(37.45in^2\right)\left(0.00136in\right)\left(\frac{2.54cm}{in}\right)^3\left(\frac{8.92grams}{cm^3}\right) = 7.44grams$$

The remaining layers are calculated the same way. Layers 1 and 12 are etched later in the Outer Layer Etching process. The overall calculation is:

Eq A-2
$$\sum_{Layer2}^{Layer11}\left(Layer \cdot remaining \cdot area\right)\left(Foil \cdot thickness\right)\left(\frac{2.54cm}{in}\right)^3\left(\frac{8.92grams}{cm^3}\right) = Mass \cdot Foil \cdot remaining$$

[3] This area is the nominal area. The actual profile of the traces and features on the PCB is different from ideal 90º angles, due to underetching. The top of the trace is smaller and the bottom of the trace is larger than ideality.

