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Laboratory 101: A Guide to Understanding your Testing Laboratory

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Abstract:
Laboratory 101: A Guide to Understanding your Testing Laboratory

Within today's Consumer Electronics Industry, a laboratory report listing elemental content is standard protocol. Understanding the information listed within a lab report can be difficult and understanding how that information was obtained is not common knowledge.

- Do you know what the Laboratory sample preparation techniques are?
- Is the Laboratory using the correct test methods?
- Does the Laboratory have the proper certifications?
- How interactive are you with the Laboratory?

Understanding the answers to these questions is imperative to showing compliance to the various global eco-compliance directives and OEM "Green Programs" such as EU RoHS, China RoHS, REACH, Halogen Free, etc.

This paper will provide insight into laboratory protocols and practices. It will provide information on the appropriate certifications a testing laboratory should have. It will also try to make clear how to interpret the information on a "lab report" and explain terminology such as:

- MDLs
- PQLs
- Units of measure (mg/kg, ppm, etc…)
- LCS and LCS recoveries
- QC protocols
- Flags
- N.D. vs <

The paper will also discuss test methods specific to the various global eco-compliance directives as well as the different instrumentation used for these types of analyses.

Lastly, this paper will discuss the importance of building a "partnership" between the laboratory and the Client. Due to the diverse array of sample matrices as well as the various manufacturing procedures within the consumer electronics industry, the need of a synergy between the Laboratory and the Client is a very important component of a company's compliance strategy. It will also explain why sample preparation is more important than the actual testing of a sample and provide some examples of "issues" that are inherent to material testing.

The premise of this paper is to give a brief overview of laboratory protocols and practices, provide some answers to questions that you have and that we have been hearing, provide terminology/acronyms and their definitions, try to explain how to interpret the information on a "lab report", and try to increase your knowledge of what a Laboratory can provide. The topics we will cover are Accreditations, Acronyms/Terminology/Definitions, Lab Reports, Methods/Instrumentation, Sample Preparation, and criteria to consider when choosing a lab.

Accreditations:
Every Laboratory should have some type of accreditation. Types of accreditations can be:

•Regional – State by State
•National – NELAC, CPSC
•International – ISO 17025
•Company standard – Sony Green Partner, Motorola W-18, Apple, Intel etc…
It is important to know what appropriate accreditation is applicable to the compliance directive and that the lab you are considering has that accreditation and when it expires. Lastly it would be important to know what analytes and/or processes

the accreditation applies to. Accreditations are important because they validate that the laboratory is following protocols and practices that help to ensure that Good Laboratory Practices (GLP) are standard operating procedures.

Acronyms/Terminology/Definitions**N.D. or <** means Non Detect to the reporting limit
RL is reporting limit - is the lowest concentration that can be reliably achieved within the specified limits of precision and accuracy during routine laboratory operating conditions. The RL is generally 5 to 10 times the MDL or based on the lowest calibration point. The RL is the limit where one can quantify the analyte reliably (quantitative).
MDL (method detection limit) is the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix type containing the analyte. The MDL measures if the analyte is present or not present (qualitative).
PQL stands for "practical quantitation limits". The PQL is defined as the lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions.
Batch is a group of similar samples that are analyzed together with one set of QA. A batch usually contains up to 20 samples. Certain methods will dictate a smaller batch size.

CCV (continuing calibration verification) standard consists of a clean matrix (organic-free water or reagent sand), spiked with a known amount of analyte(s) and analyzed on the instrument. A CCV does not go through the preparation procedures.

MB (method blank) consists of a clean matrix (organic-free water or reagent sand), prepared and analyzed similarly to the samples in the batch.
LCS (laboratory control sample) consists of clean matrix (organic-free water or reagent sand), spiked with a known amount of analyte(s) from a different source than the CCV, prepared and analyzed similarly to the samples in the batch.
MS/MSD (matrix spike/matrix spike duplicate) consist of two separate aliquots of the sample spiked with a known amount of analyte(s) prepared and analyzed similarly to the samples in the batch. MS/MSD results are primarily designed to assess data quality in a given matrix, and not laboratory performance.
Surrogates are exclusively used in organic analyses and are added to the samples to monitor overall system performance within each individual sample. Surrogate compounds are chosen based on their analytical similarity to target compounds.
ppm – parts per million - unit of measure
mg/Kg – milligram per kilogram - unit of measure – the metric equivalent to ppm
IS (Internal Standards) are added to organic samples or sample extracts at a known concentration just prior to instrument analysis to permit correction for system inefficiencies. They are not extracted. The internal standard must be an analyte that is not a sample component.
Case Narrative - is written to qualify the data within the laboratory report. They indicate to the client items of concern that occurred from the time of receipt of their samples.

Qualifiers – sometimes are called flags and they identify criteria that are pertinent to the testing of that material. Some examples are:

B – analyte detected in the associated method blank
C – Laboratory not accredited for this parameter
S – Spike recovery outside accepted recovery limits

LIMS – Laboratory Information Management System is the software used to manage and report laboratory data.**Lab Reports**

Compliance Directives have made a lab report an important part of the manufacturing process. Because lab reports are provided in different formats, understanding the information listed can be confusing. A Lab Report should contain the following information:
1.Lab ID # (work order #) – an identifier, assigned by the lab, that will follow the sample through the testing process
2.Sample name w/ description – so the Client has an identifier, such as a part name, type of plastic, etc…
3.Case narrative - is written to qualify the data within the laboratory report. They indicate to the client items of concern that occurred from the time of receipt of their samples.
4.The testing method
5.Element(s) of interest
6.The result(s)
7.The reporting limit (RL)
8.Method Detection Limit (MDL)
9. Directive limits – maximum concentration limits for the elements of concern set by the compliance directive
10.Unit of measure – typically ppm or mg/Kg
11.Qualifiers/flags - identify criteria that are pertinent to the testing of that material
12.Date analyzed

13.Sample weight
14.Lab's accreditation
15.Picture – this is important because sample deconstruction and/or grinding will cause the original sample to be unrecognizable after testing is completed.

A lab report may look like:

Sample Description : Widget

Test Item(s):	Unit	Method	Result	MDL	RoHS Limit
Cadmium (Cd)	mg/kg	With reference to US EPA	n.d.	2	100
Lead (Pb)	mg/kg	Method 3052. Analysis was	n.d.	2	1000
Mercury (Hg)	mg/kg	performed by ICP/AES	n.d.	2	1000

We see that the report lists the analytes, unit of measure, method, results, MDL and compliance directive limits. We will first concentrate on the "Result" listed. What does a result of N.D. mean?

N.D. = non detect. The N.D. result means that the analyte was not present at a concentration level greater than the reporting limit (RL). Sometimes non detect will also be reported as <. The < result will always be followed by the reporting limit (i.e.) < 2 ppm or < 2 mg/Kg.

Note – with a non-detect result, the analyte of interest could be present below the reporting limit. Sometimes the reporting limit will be listed as the PQL

Secondly, we will concentrate on the "MDL" column and what it means. Listing the MDL after the result can be a little misleading because the MDL is the "method detection limit" and may not be the same value as the reporting limit. Typically, the MDL is determined under ideal laboratory conditions by spiking a sample that has no matrix interferences. Also, the MDL is determined by using a specific sample amount, hence, any subsequent sample analyzed that has a different sample volume than the amount used in determining the MDL, affects the MDL. Therefore, different sample amounts, matrix interferences, and dilutions are some of the items that change the MDL for that specific sample. Thus, one would expect the MDL to have different values for different samples. It can be misleading to report the MDL without these adjustments.

Facts:

- MDL is always $\leq$ RL (reporting limit)
- RL is not always = to the MDL
- N.D. is not always = unadjusted MDL

Most important fact:

- N.D. can be > than the unadjusted MDL

The more appropriate way to provide results might be:
Example 1

Sample Description : Widget

Test Item(s):	Unit	Method	Result	MDL	RoHS Limit
Cadmium (Cd)	mg/Kg	With reference to US EPA	< 3.32	2	100
Lead (Pb)	mg/Kg	Method 3052. Analysis was	< 10.45	2	1000
Mercury (Hg)	mg/Kg	performed by ICP/AES	< 32.80	2	1000

The result column lists the results as < with the reporting limit (RL) and still reporting the method detection limit (MDL).

Example 2

Sample Description : Widget

Test Item(s):	Unit	Method	Result	RL	RoHS Limit
Cadmium (Cd)	mg/Kg	With reference to US EPA	n.d.	2	100
Lead (Pb)	mg/Kg	Method 3052. Analysis was	n.d.	2	1000
Mercury (Hg)	mg/Kg	performed by ICP/AES	n.d.	2	1000

The result column lists the results as N.D. with the reporting limit (RL) listed instead of the method detection limit (MDL).

Lastly, we will concentrate on why is it important that the applicable method is listed on the lab report. The method should match the compliance directive so that the instrumentation and sample preparation follow all of the compliance directive's protocols. Listed under Method are US EPA Method 3052 (microwave digestion) and analysis by ICP/AES, which are excellent tried and true applications for metals analysis. However, the RoHS compliance directive requires IEC 62321 methods which also apply the same microwave digestion and analysis by ICP/AES, however, IEC 62321 chapter 5 requires mechanical sample preparation. Mechanical sample preparation incorporates manual cutting, coarse grinding/milling, homogenizing, fine grinding/milling and very fine grinding of polymers and organic materials. US EPA Method 3052 does not incorporate any sample preparation as listed in IEC 62321, therefore isn't the acceptable test method for RoHS compliance.

A more appropriate way to list the method for RoHS compliance might be:

CLIENT:	Company ABC123	Client Sample ID:	XX-XXXX
Lab Order:	000000001	Report Date:	12/5/2008
Project:	Widget	Collection Date:	11/30/2008
Lab ID:	000000001-01A		

Analyses	Result	Reporting Limit	RoHS Limit	Units	Date Analyzed
ICP-AES Metals, Total		**Method: IEC 62321**			
Cadmium	<1.67	1.67	100	mg/Kg	12/2/2008
Lead	<2.45	2.45	1000	mg/Kg	12/2/2008

Sample Weight as Received: 1.26 g

Methods and Instrumentation:
Typically, a specific method will indicate the appropriate instrumentation.

Some of the methods that the Electronics Industry may be dealing with are:

IEC – RoHS, JIG Annex A & B
MII – China RoHS
EPA – JIG Annex A & B, REACH, Low Halogen, CPSC, Company Green Programs
ASTM - JIG Annex A & B, REACH, Low Halogen, CPSC, Company Green Programs
BS EN - JIG Annex A & B, REACH, Low Halogen, CPSC, Company Green Programs

Some of the instrumentation needed for these methods are:

GC - Gas Chromatograph
ICP - Inductive Coupled Plasma
AAS - Atomic Absorption Spectrometry
IC - Ion Chromatograph
HPLC – High Pressure Liquid Chromatograph
MS – Mass Spectrometry
OES – Optical Emission Spectrometry
AES – Atomic Emission Spectrometry
XRF – X-Ray Fluorescence
FTIR - Fourier Transform Infrared Spectroscopy

Also, the method can indicate specific sample preparation protocols, which can be very important to the matrices being tested.

Sample Preparation:

Sample Preparation is as important as any aspect of testing and in some cases, sample preparation is the most important process. Sample preparation is the process the sample goes through to prepare it for the required testing. The Compliance Directive as well as the sample matrix will determine sample preparation. Another determining factor of what sample preparation is required is what needs to be tested.

Sample Preparation Case Study:

A company was conducting a standard compliance check of incoming material by XRF. XRF indicated that Lead (Pb) was present in the incoming material. Therefore, the company contacted their CM about the Pb hit in the sample, which was a plated or coated metal (figure 1).

(Figure 1)

The CM contracted a testing laboratory to test the material. Also, the company contracted a different lab to test the material. The testing criteria were:
- Material of interest – plating or coating only
- Compliance Directive – RoHS
- Specifically Cadmium (Cd) & Lead (Pb)

The company's Lab and the CM's Lab are each ISO 17025 certified, each followed IEC testing methods and each analyzed by ICP/AES. The laboratory's results were:

Company's Lab	CM's Lab
Lead (Pb) = 759 ppm	Lead (Pb) = 24 ppm
Cadmium (Cd) = 867 ppm	Cadmium (Cd) = 13 ppm

The company's contracted lab had results that indicated non-compliance and the CM's contracted lab had results that indicate compliance.

The question that needed to be answer was how was it possible that one Lab's results were 40 to 50 times higher than the other?

The answer came when the company requested pictures of the sample after sample prep was completed.
The picture of the Sample Prep (Figure 2) from the Company's lab consisted of scratching the coated/plated surface of the sample. Although the process was not very "high tech", it was the most accurate way to isolate the surface material

(Figure 2)

The picture of the Sample Prep (Figure 3) from the CM's lab consisted of drilling into the surface and substrate of the sample. This process diluted the coated/plated surface with substrate, which caused a low bias when it was tested.

(Figure 3)

The company determined the results from the CM's lab were not acceptable because the sample prep was incorrect. Again, by drilling into the sample, the coated/-plated material was diluted by the substrate, which caused a very low bias when tested.

This clearly shows that Sample Preparation is as important as any aspect of testing and in some cases sample preparation is the most important process.

Criteria to consider when choosing a lab:

Global Compliance Directives, Company Green Programs, Vendor requirements are only a few reasons why testing laboratory services are needed. Here are some strategies you can incorporate when choosing what testing laboratory to contract:

1. **Ask around –** Contact your colleagues and peers. They can provide excellent information and opinions on testing labs. An event such as IPC/Expo is an ideal time and place to get information on what lab may be the best match for your company.
2. **Accessibility -** How quickly can you get answers and what is the knowledge base of your contact are great indicators of what a lab can provide.

3. **Communication -** A lab doesn't know your processes as well as you do and you don't know the lab's processes as well as the lab. A sign of a good lab is one that is going to try to understand your processes in order to provide a compliance plan that is best suited to your needs.
4. **A Laboratory should be an advocate for their Client –** The primary function of a testing lab should be to provide their Client dependable, reliable, defensible and cost effective information. Therefore, it is important for the laboratory to build a relationship with the Client that will help inform both parties as to what is and isn't possible. Global compliance directives have provided revenue opportunities for testing labs; however, sustaining a relationship with Client is far more important than the cost of one testing opportunity.

Conclusion

Understanding a lab report and what a testing lab can provide is not common knowledge. Choosing an appropriate testing lab is a daunting task given all the different criteria's that can enter into your decision process. Hopefully the information offered in this paper will assist in expanding your knowledge on the capabilities of testing labs as well as provide insight into laboratory protocols and practices.