TOTAL SOLUTIONS POPs LABORATORIES OF THE FUTURE

Addink, R.*, Hall, T.
Toxic Report, 580 Pleasant St, 2nd Fl, Watertown, MA 02472, USA

Introduction

Laboratories all over the world are becoming interested in accreditation under the ISO 17025 International Laboratory Standard. Accreditation is especially becoming relevant for POPs laboratories in developing countries. The current Standard has been in place since 2005 and offers a Quality System for a variety of laboratory work. If a laboratory wants to become accredited the Quality System has to be put in place before the recognition can be awarded. The focus of the Quality System is on the improvement of the competence of the laboratory to produce valid results in a consistent way. Accreditation is decided by local (national) accrediting bodies in the country where the laboratory is located. A commercial system offering turn-key solutions in obtaining ISO 17025 accreditation is described below. The system is comprised of a number of Standard Operating Procedures (both management and technical ones; listed in Table 1); automated sample processing/clean up equipment; analytical equipment for high resolution gas chromatography and mass spectrometry analysis; and assistance in setting up the laboratory anywhere in the world via training and visits. Our own laboratory went through the entire accreditation process in about 7 months (from start of SOP writing to audit for accreditation).

Material and methods

Sample preparation and analysis are an important part of the ISO 17025 accreditation process. The Standard requires that laboratories seeking accreditation validate their methods. This is typically a time consuming process involving a large amount of sample preparation and analysis. Automating the sample preparation process can greatly help in getting the accreditation as fast as possible. Automation will, among others, result in faster turn around time of samples, lower and control the cost of analysis, and improve the quality of the data generated as part of method validation resulting in faster accreditation. Automated sample preparation includes Pressurized Liquid Extraction (PLE) with typical extraction times of 20-40 min at 100 atm; automated column chromatography using our PowerPrep system which is modular and allows for simultaneous clean of up to 6-8 samples using between 1 and 4 different pre-packaged chromatographic columns; automated Solid Phase Extraction for processing of aqueous samples using pre-packaged cartridges; and a line of automated evaporators for reduction of sample volume in between sample prep steps and for final blow down prior to analysis. The combined TotalPrep integrates PLE, PowerPrep and evaporators. [1]

High resolution GC/MS analysis at a resolution of 10,000 provides reliable analysis, identification and quantification of the analytes. The methods of analysis described in our SOPs are modeled after standard US Environmental Protection Agency (EPA) methods such as method 1613 for PCDD/F analysis and method 1668 for PCB analysis.

Results and discussion

Persistent Organic Pollutants such as polychlorinated dibenzo-p-dioxins and furans (PCDD/Fs), polychlorinated biphenyls (PCBs), brominated flame retardants (PBDEs), pharmaceuticals, and personal care products are analyzed by a wide variety of laboratories. Governments all over the world are increasingly requiring that foods and products are analyzed for POPs. This has especially been the case since the entry into force of the Stockholm Convention in 2004. Trace level analysis of these compounds often requires serious investment in very sensitive analytical instrumentation that is capable of detecting analytes at the nano-, pico-, and femtogram level. In addition a highly educated and skilled staff is often required for the analysis of these compound classes.

Table 1. List of Standard Operation Procedures created for ISO 17025 accreditation.

Document No.	Document Name
M-001.1	Toxic Report LLC Quality Manual
SOP 0.1	SOP for SOP writing
SOP 4.1.1	Confidentiality
SOP 4.1.2	Data Integrity
SOP 4.3	Document Control
SOP 4.4	Review of Requests, Tenders or Contracts
SOP 4.6.1	Purchasing Services and Supplies
SOP 4.6.2	Inventory of Standards
SOP 4.7	Customer Service
SOP 4.8	Complaints
SOP 4.9	Control of Non Conforming Work
SOP 4.11	Corrective and Preventive Action
SOP 4.13	Records
SOP 4.13.1	Chain of Custody Form
SOP 4.13.2	Data Security, Archival and Backup
SOP 4.14	Internal Audits
SOP 4.15	Management Review
SOP 5.2	Training of Laboratory Personnel
SOP 5.4.1	Method Validation
SOP 5.4.2	Measurement Uncertainty
SOP 5.4.3	HR GCMS PCDD-F Analytical Method
SOP 5.4.4	HRGCMS PCB Analytical Method
SOP 5.4.5	Procedure for PLE
SOP 5.4.6	Procedure for Power-Prep
SOP 5.4.7	Procedure for SPE
SOP 5.4.9	Analysis of OCP
SOP 5.4.10	Analysis of PBDE
SOP 5.4.11	Analysis of PAH
SOP 5.5.1.1	Inventory of Laboratory Equipment
SOP 5.5.1.2	Maintenance of Equipment
SOP 5.5.2	Qualification of Equipment
SOP 5.5.2.1	Procedure for Support Equipment Calibration
SOP 5.6	Measurement Traceability
SOP 5.8	Handling of Samples
SOP 5.9	Quality of Test Results
SOP 5.9.1	Data Review
SOP 5.10.1	Data Reporting

Table 2 PCDD/F concentrations analyzed by Toxic Report and reference values from Cerilliant EDF-2526 Fortified Fish Reference Material.

Analyte	Toxic Report value in ppt	Reference value in ppt
Analyte(s)		
Dioxins		
2,3,7,8-TCDD	20.40	19.8±4.18
1,2,3,7,8-PeCDD	38.02	39.9±10.6
1,2,3,4,7,8-HxCDD	47.42	54.9±7.80
1,2,3,6,7,8-HxCDD	50.13	51.1±19.3
1,2,3,7,8,9-HxCDD	50.48	52.9±18.1
1,2,3,4,6,7,8-HpCDD	65.61	70.7±23.2
OCDD	166.25	181±53.4
Difurans		
2,3,7,8-TCDF	19.79	18.7±5.58
1,2,3,7,8-PeCDF	36.34	39.0±7.36
2,3,4,7,8-PeCDF	37.77	37.8±10.2
1,2,3,4,7,8-HxCDF	74.62	83.3±23.0
1,2,3,6,7,8-HxCDF	56.72	62.8±19.6
1,2,3,7,8,9-HxCDF	51.78	57.3±10.9
2,3,4,6,7,8-HxCDF	54.72	58.6±14.2
1,2,3,4,6,7,8-HpCDF	76.24	81.6±13.7
1,2,3,4,7,8,9-HpCDF	70.84	76.7±26.6
OCDF	173.68	185±57.4

As an alternative a system has been developed to offer a relatively simple “turn-key” solution to the needs of laboratories to analyze POPs which can be implemented both in commercial laboratories that want to expand their activities into this field, and in industries that are looking to create an in-house laboratory for their specific analytical needs. 16 methods have been validated using the automated sample preparation equipment described. These include the analysis of PCDD/Fs, PCBs, PBDEs, PAHs, and pesticides in sediment, serum, soil/solids, tissue, and water. In addition development of analytical methods for perfluorinated compounds and pharmaceuticals is underway.

The accredited matrices offer a wide opportunity for analysis of food and beverage (meat, fish, milk, shell fish, feed and oil); biological matrices such as breast milk, whole blood and serum; and environmental samples such as air, bottled water, drinking water, waste water, soil, sediments, and solid waste. An example of the testing of a tissue reference material is shown in Table 2. As can be seen there is excellent agreement between the values found in our laboratory and certified values.

ISO accreditation will allow a laboratory to get more work such as contracts from government and industry. This will also result in attracting other customers. National and international recognition that comes from ISO accreditation can also greatly help in increasing customer loyalty. Streamlining laboratory operations through standardized processes and procedures will increase efficiency. The investment in accreditation has a long term return. Laboratories interested in obtaining accreditation may include: service labs, food testing labs, environmental testing labs, chemical labs and clinical diagnostics labs.

A complete package of SOPs, sample preparation equipment, high resolution GC/MS and training and facility visits has been set up by Toxic Report LLC. The concept of this "Total Solution" can greatly help companies or existing laboratories to branch out into the field of analysis of Persistent Organic Pollutants. If a laboratory is interested in starting up one aspect of POPs analysis (e.g. POPs in water) the package can be tailored to their needs. Specific kits available include food and feed; environmental; water; breast milk; and serum. Our kits include consumables, reagents and standards. It can also include basic lab start up supplies such as balances and glass ware. The main bottle neck in traditional POPs laboratory work – sample preparation – is eliminated via the newest automated sample prep techniques that are providing high throughput on the front end of the POPs analysis. Automated sample prep yields better (higher quality) data and allows for fast turn around.

Toxic Report makes it easier for companies and organizations to perform screening and confirmation of POPs. Our services include implementation and transfer of technology and SOPs. It allows for different levels of POPs testing in an accredited format. We are able to supply the entire accredited laboratory.

References:

1. Focant JF, Shirkhan H, Patterson Jr DG. (2009) *Organohalogen Cmpds* 71: 2438-2443.