

## PREPARATION STEPS FOR ACCREDITATION IN THE FIELD OF PCDD/F EMISSION SAMPLING IN A RESEARCH FACILITY

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### Introduction

An accreditation according to standard DIN EN ISO/IEC 17025 (1) provides a laboratory with a guarantee of the quality of its measurements; for a customer (e.g. an external client from private companies, the German authorities or within the framework of co-operations), it demonstrates the competence of this testing laboratory to carry out specific tests.

To obtain an accreditation, a Quality System has to be established which meets the requirements of the Standard EN ISO/IEC 17025. In the field of emission sampling, the compliance to the requirements §5.7 of ISO 17025 is not sufficient to get the accreditation in Germany. Additional specific standards have to be satisfied, such as VDI 4220 (2). VDI 4220, a technical rule from Kommission Reinhaltung der Luft (Germany), views on technical requirements for emissions and immissions testing laboratories and concerns the criteria competence/personnel, premises/equipment and working procedures. An accreditation body (e.g. DAR, DACH or DAP in Germany) is charged to control this Quality System of the lab and delivers a certificate (accreditation) if the requirements are satisfied.

In the following paper, the activity in our research facilities in the field emission sampling will be first explained. Secondly the implementation of our quality system, also our experienced difficulties will be exposed.

### Main Subject

The dioxin laboratory belongs to a research facility: GSF (Neuherberg, Germany), the permanent staff consists of a group of four employees. More than a year was needed to install a quality system to meet the standard ISO/IEC 17025 and to obtain accreditation for the dioxins and furans analysis (September 2001).

As it would be useful in the scope of European projects, it was decided to extend the quality system to the emissions sampling. In this field, the complying with the requirements of ISO/IEC 17025 is not sufficient to obtain the accreditation in Germany. Additional specific standards have to be satisfied, such as VDI 4220, a technical rule established by KRdL (Kommission Reinhaltung der Luft), the German Standards Committee. The preparation for accreditation of dioxins and furans emission sampling for our research group needed one year (January 2004).

Those who implement a quality system need to:

- be continually aware of the current specific standards, European and national ones. (Note: in the field of emission sampling, a new European standard has been published: DIN EN 13284-1 (3)),

- write down internal standard operating procedures (SOP). These SOP have to be verified against the above-mentioned standards or validated (in case where a published method does not exist and an in house method has to be developed). The SOP will be identified, approved and continually updated. The work has to be carried out according to this SOP,

- produce a quality manual,

- specify the responsible person and their deputy for key functions in a laboratory and for equipment,
- identify the equipment, establish a timetable and a procedure to check and calibrate regularly the equipment, calculate the measuring uncertainty, define acceptance limits and the corrective measures to take in case of "out of control" situations,
- have a technical and a quality manager and deputies,
- organise and document a schedule for continuous training of all personal,
- involve the employees in the QS-system,
- establish and update regularly records (e.g. registration of sample number in internal sample books, registration of the equipment, produced data, list and results of inter-laboratory studies) for traceability,
- collect and validate data (for example with inter-laboratory studies, in control charts, against limit values),
- carry out internal and external audits and quality management reviews.

To satisfy the Standards regarding emission sampling every step of the following quality controls has to be taken into account.

The operating conditions for the sampling equipment and the environmental parameters are checked and documented before sampling. The system is adjusted to the optimal gas flow rate 1.5-2 m<sup>3</sup>/h for PCDD/F and PCB (4).

The sampling equipment is regularly maintained and calibrated. An external calibration of devices measuring temperature, gas flow, dynamic and static pressure and velocity is performed by competent institutions, which grant a certificate. These calibrated devices will be used for internal calibration of the sampling devices used for emission sampling of PCDD/F. The results of each calibration and parameters such as uncertainty, repeatability and reproducibility are documented in a protocol sheet.

The following parameters must be controlled before sampling: the sampling system must be leak tight (the flow rate must be lower than 2% of the usual flow rate under standard conditions), the temperature in the condensate flask and in the chimney, should be lower than 20°C, the velocity and the nozzle diameter, the environmental parameters and the operation ability of the measuring devices and software.

The cooled suction tube method is used to take samples and determine PCDD/PCDF according to DIN EN 1948-1 (5). Before sampling, the collection units (cartridges) are spiked with C<sub>13</sub>-marked sampling standards PCDD/PCDF. The samples are taken by an isokinetic extraction from the flow of waste gas. The PCDD/PCDF are collected in the sample cartridges. The sampling parameters are documented in sampling reports.

In later steps the samples are transported into the laboratory, registered in internal sample books according to a labelling system. Soxhlet extraction and clean-up procedure for PCDD/F and PCB are additionally carried out and documented. Internal controls for the new reagents, methods, equipment, employees are performed. The sample is analysed by HRGC/HRMS (6). PCDD/F and PCB data are collected and validated (control cards).

The sampling measurements are validated with the following controls: the blank value has to be lower than 10% of the limit value for PCDD/F for the flue gas (0.1 ng I-TEQ/Nm<sup>3</sup> (7)); the calculated value of a sample has to be higher than the blank value; the value of the back-up cartridge has to be lower than 10% of the other cartridges (resin + glass wool); the recovery rate of sampling standards must be more than 50%; the isokinetic ratio should be around 1.

Along the accreditation process, we experienced the following problems:

- Differences between research facility and industry

In industry, standard and routine methods are normally used. In research facilities, the quality system should be more flexible, in order to implement it into a new department, or to adapt it for a new method. A number of people are working for a limited period of time, like trainees, guest scientists, PhD or Master thesis. The work (procedures, results, samples) produced from these temporary engaged people with their own projects has to be rigorously checked before data is released. The languages, in which documents are written, should be chosen intelligently. English is an essential language and is preferable. It must be understood that a calibration certificate does not ensure that equipment has been well calibrated, according to the specific standards. Understanding the procedure and checking the validity of a calibration is critical.

- Additional requirements required to accredit field emission sampling in Germany  
In Germany, the LAI (Länderarbeitsgemeinschaft für Immissionsschutz = german Bundesländer Committee for immission protection) produce a guideline for notification. If a laboratory submits for an accreditation or/and for a public recognition (as an authorised testing laboratory) in the field "immission protection", it must satisfy the corresponding requirements of ISO/IEC 17025 and LAI-guideline (specific module "immission protection"). The LAI-specific module is used as basis to confirm the competence of an authorised testing laboratory or an accredited laboratory. In conformity to this LAI-guideline and the German standard VDI 4220, an authorised testing laboratory and an accredited laboratory must use and control the whole measuring process in the field "immission protection". The determination of this competence can be checked in the scope of the accreditation or by the public institution. The two above-mentioned institutions recognise each others confirmation of the competence. In this way, an accredited laboratory can submit for a notification and reciprocally a notified laboratory can submit for an accreditation. The process will therefore be simplified.

According to VDI 4220 (technical rule), the competence of the following will be controlled:

- the qualification of the personal, as well as the practical experience and the specific knowledge of measurement techniques (at least 3 years full-time work in the field of the determination of emissions/immissions),
- the technical equipment, required for emission measurements,
- the establishment of an extra LAI-sampling report,
- the establishment of a detailed measurement plan for emission, which defines the measurement objective and the strategy to reach this objective. The requirements for this measurement plan are set out in VDI 2448-1 (8).

A standard form of test report for the determination of emissions in accordance with §§ 26, 28 BImSchG (Bundes-Immissionsschutzgesetz = Federal Immissions Control Act) as well as checklists of equipment for emission measurements are enclosed in appendix of VDI 4220 and must be followed.

## Conclusions

The positive side of the parallel process (accreditation/notification):

- the submission of the accredited laboratory for a notification and the control by the public institution will be facilitated,
- a proof of real competence in the field of emission sampling is achieved.

Recently, the tendency to demonstrate this competence has become more and more important: from certification (test of conformity with a standard) to accreditation (formal recognition of the competence of a laboratory to carry out tests) and from accreditation to notification (formal act of announcement by a government body).

The negative side of the parallel process:

- do all laboratories (especially in case of research facilities), submitting for this accreditation of emission sampling, need this preparation and notification? This process takes time, more staff and more investment to satisfy the additional requirements,
- will all European accredited laboratory be accepted in Germany?

## References

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