QUALITY CONTROL FOR CONTRACTED DIOXIN/FURAN ANALYSES

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ABSTRACT

When the analysis of environmental samples for dioxins/furans is performed by a contract laboratory, quality control measures can include blind replication, analysis of low-level spiked blanks, and inter-laboratory sample exchange. It is imperative that all GC-MS data and final sample extracts are stored until the raw/final data can be examined by a qualified analyst for consistency of congener and isomer distributions.

INTRODUCTION

Many government and industrial organizations require the analysis of environmental samples for the chlorinated dibenzo-p-dioxins (dioxins) and the chlorinated dibenzofurans (furans). The services of contract laboratories are often engaged for this work, because relatively few laboratories have the capability of performing this determination. Since the analysis of dioxins/furans is generally required for applications where significant environmental contamination is suspected, the samples taken tend to be among the most difficult to analyze. In addition, the detection levels required for dioxin/furan determinations are often in the parts-per-trillion (ppt; pg/g) or lower concentrations. Organizations that engage the services of contract laboratories must be aware of basic QA/QC principles, and should design quality checks into their contracted work. Several methods of ensuring quality control on contracted work are discussed in this paper.

OBJECTIVES OF CONTRACTED WORK

It is essential to clarify the objectives of a study before analytical work is commissioned. Different minimum degrees of analytical quality assurance are required for general surveys and routine monitoring compared to health studies or where results are used for litigation. For example, if analytical data are used to charge a company for illegal waste dumping, strict chain-of-custody

protocols must be followed. The study objectives will also affect the types and numbers of samples needed, and whether dioxin/furan toxic congener or congener group determinations will be performed.

SAMPLING

It is obvious that the sampling methods employed depend upon the specific matrix to be analyzed. However, the QA/QC measures taken also depend upon the sample. In some cases, it may not be possible to obtain true replicate samples. Splitting the sample equally after it is taken may also be very difficult (i.e. air particulate filters). Organizations that employ contract laboratories to analyze their samples for dioxins/furans should not underestimate the importance of ensuring the original samples were collected properly. Obtaining true replicate or split samples is an important QA/QC tool. For sample types that are not amenable to equal splitting or for which replicates cannot be obtained, a solvent extract of the sample may be obtained. A solvent extract may be easily split into many equal portions for the purpose of QA/QC studies.

QA/QC PRACTICES

It is difficult to ensure accurate and precise results are obtained for ultra-trace dioxin/furan determinations. When such analytical work is contracted from an external laboratory, this task is especially difficult. As there are no real-matrix reference materials that have been certified for the dioxins/furans, those who purchase analytical services for these compounds have to rely on the internal QA program of the contractor to ensure analytical results are of acceptable quality.

When contracting dioxin/furan work, however, there are a number of features that can be designed in the work program to check the quality of work performed. Some of these will increase the cost of the contract, but it is essential that the QA/QC component of contracted work not be reduced. Some of these features are:

1. include replicates in the samples submitted, but give them different sample identification (ID) numbers (blind replication)
2. employ a second laboratory, send them a small number of replicates of the samples submitted to the main laboratory
3. include a previously analyzed sample (same matrix) and give it a new ID number (be aware of sample storage effects)
4. purchase standards in solution - include a low-level spiked solvent sample for analysis (concentration within 3-5 times stated detection limit of contract laboratory)
5. confirm selected samples by a different instrumental technique (if low resolution mass

6. for ultra-trace work - especially for contentious samples such as drinking water/food - insist dedicated glassware and laboratory space be used. Include cost of new set of glassware to prepare samples if necessary.
7. for the best quantitative work, spiking samples with ^{13}C-labelled dioxin/furan analogues is essential.
8. do not accept a table of results as a final report. The methodology used should be described (at least generically), especially the QA/QC followed and how detection limits and concentrations were calculated
9. include travelling blanks and/or spiked blanks with the samples
10. consider contracting an expert (independent of laboratory) to review results, if in-house expertise does not exist
11. ensure all sample extracts, raw data stored on computer media, and related records are retained by the contract laboratory until their disposal is approved
12. ask for delivery of all raw GC-MS chromatograms in addition to analytical report. Dioxin/furan isomer/congener patterns in samples of the same matrix and which were taken from the same general location will generally be the same.

CONCLUSIONS

Implementation of a number of the above suggestions will allow an assessment of the overall quality of contracted dioxin/furan work, independently of the contractor. Many of the suggestions will result in increased cost of the work. However, cost savings should be obtained by carefully choosing the minimum number of samples required to supply the needed information, not by reducing the QA/QC.