

ANNEX V

METHODS OF ANALYSIS TO CONTROL UNDESIRABLE SUBSTANCES IN FEED
[^{F1}B. DETERMINATION OF THE LEVELS OF DIOXINS (PCDD/PCDF) AND PCBs

CHAPTER II

Sample preparation and requirements for methods of analysis used in official control of the levels of dioxins (PCDD/Fs) and dioxin-like PCBs in feed**7. Specific requirements for bioanalytical methods**7.4. *Performance characteristics*

- 7.4.1. Since no internal standards can be used in bioanalytical methods, tests on the repeatability of bioanalytical methods shall be carried out to obtain information on the standard deviation within and between test series. Repeatability shall be below 20 % and intra-laboratory reproducibility shall be below 25 %. This shall be based on the calculated levels in BEQ after blank and recovery correction.
- 7.4.2. As part of the validation process, the test shall be shown to discriminate between a blank sample and a level at the cut-off value, allowing the identification of samples above the corresponding cut-off value (see point 7.1.2).
- 7.4.3. Target compounds, possible interferences and maximum tolerable blank levels shall be defined.
- 7.4.4. The percent standard deviation in the response or concentration calculated from the response (only possible in working range) of a triplicate determination of a sample extract may not be above 15 %.
- 7.4.5. The uncorrected results of the reference sample(s) expressed in BEQ (blank and at the maximum level or action threshold) shall be used for evaluation of the performance of the bioanalytical method over a constant time period.
- 7.4.6. Quality control charts for procedure blanks and each type of reference sample shall be recorded and checked to make sure the analytical performance is in accordance with the requirements, in particular for the procedure blanks with regard to the requested minimum difference to the lower end of the working range and for the reference samples with regard to within-laboratory reproducibility. Procedure blanks shall be controlled in a manner to avoid false-compliant results when subtracted.
- 7.4.7. The results from the confirmatory methods of suspected samples and 2 to 10 % of the compliant samples (minimum of 20 samples per matrix) shall be collected and used to evaluate the performance of the screening method and the relationship between BEQ and TEQ. This database may be used for the re-evaluation of cut-off values applicable to routine samples for the validated matrices.
- 7.4.8. Successful method performance may also be demonstrated by participation in ring trials. The results from samples analysed in ring trials, covering a concentration range up to e.g. 2 × maximum level, may be included in the evaluation of the false-compliant rate, if a laboratory is able to demonstrate its successful performance. The samples shall cover most frequent congener patterns, representing various sources.

Changes to legislation: There are currently no known outstanding effects for the
Commission Regulation (EC) No 152/2009, Division 7.4.. (See end of Document for details)

- 7.4.9. During incidents, the cut-off values may be re-evaluated, reflecting the specific matrix and congener patterns of this single incident.]

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