Application of 'Klimisch' criteria to evaluate the quality of data used in surfactant monitoring studies

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Abstract

CEFIC-LRI have developed a user-friendly database (MonitoringBase)[1] to store metadata and actual measurements of chemicals in the environment. A system for appraising the quality of data arising from surfactant monitoring studies for inputting into this database has been developed based on application of Klimisch criteria [2].

Introduction

The Klimisch codes of reliability (1-4) are widely used by industry and regulators as the basis for assessing the quality of toxicological and ecotoxicological data on products. However, monitoring studies do not have any accepted test guidelines, nor are generated to GLP standards, so different selection criteria for monitoring studies need to be developed

Scope

Key features of a monitoring study are shown in Table 1. A number of detailed criteria for evaluating each key feature are also included to assist in the evaluation of the quality of the monitoring study.

Assignment of Klimisch score to monitoring studies

- Once a qualified judgment as to which key features have been satisfactorily addressed, it is then possible to assign the quality of the monitoring study and its data to the standard Klimisch codes (1-4).
- A study must have the three essential key features (i.e. 3, 5 and 6) to be considered to be 'reliable', i.e. Klimisch codes 1 ('reliable without restriction') and 2 ('reliable with restriction').
- Monitoring studies that cover at least five and preferably six of these key features would be assigned to Klimisch code 1, whereas those covering only 3 – 4 key features would be assigned Klimisch 2
- If any of the three key features is not sufficiently addressed then the study is assigned Klimisch code 3 ('not reliable').
- A study with insufficient experimental details is assigned Klimisch 4 (*not assignable')

References

(1) Klimisch, HJ, Andreae, M and Tillmann U (1997). "A systematic approach for evaluating the quality of experimental toxicological and ecotoxicological data". Regulatory Toxicology and Pharmacology, 25, 1-6.

(2) Schowanek et al., "Development of "MonitoringBase-Surfactants": a (meta)database for measured concentrations of surfactants in environmental matrices in Europe", Poster presentation, SETAC Gothenburg, June 2009.

Table 1. Six key features of a satisfactory monitoring study

No	Feature	Further criteria for evaluating whether a key feature has been properly covered in the monitoring study
1	Design and overall quality of study	*Desirable to show that sufficient forethought has gone into the design of the monitoring study as well as data collection and retention.* • Protocol with clearly defined objectives of the study, accurate locations for sampling (e.g. GIS coordinates), sampling/storage details and a validated analytical methodology to be applied to samples. • Raw data are archived and could be accessed by authorized person to check on the accuracy of data and calculations, if required. • Study has been carried out by an experienced group of workers with monitoring expertise. • Study has been audited internally (within company or group) and/or externally (Journal review).
2	Sample collection	*Desirable to collect samples of suitable volume and to minimise the possibility of contamination. Additional samples (blank/spiked) will enable further checks to be made in the laboratory, if needed.* • Use of appropriate containers for the study/analyte of interest. • Method of sampling and type of sample to be taken (composite or grab sample) is detailed. • Inclusion of 'blank' and 'spiked field' samples. • Care is taken to minimise the possibility of contamination. during sampling (e.g. prewashing of sample containers) • Sufficient sample is taken for analysis requirements and to avoid any sub-sampling.
3	Sample storage, transportation and receipt	*Essential to prove that the test substance has not degraded during the period between sampling and the start of sample preparation in the laboratory.* • Previous information on the stability of the analyte(s) of interest. • Use of appropriate stabilising agent to minimise sample deterioration. • Storage conditions in field/lab at suitable temperature to minimise sample deterioration. • Check on efficiency of preservation made (e.g. by analysis of 'spiked field' samples at laboratory). • Details of shipment and receipt ('chain of custody') are provided where appropriate.
4	Sample preparation	*Desirable to minimise interference from other compounds in the analysis and thereby achieve a sufficiently low limit of determination for the analyte of interest.* • Validated method for isolation of analyte of interest. • Isolation removes compounds likely to interfere in method. • Isolation achieves low limit of determination required.
5	Analytical detection	*Essential that the analytical method is sufficiently sensitive and specific enough to measure the test substance of interest, without interference and to the required limit of determination.* • Published/industry accepted and validated analytical method has been employed. • Preferably specific method (e.g. GC/MS, LC/MS). Non-specific methods can give rise to an overestimation of the level of the surfactant of interest due to the presence of structurally similar substances. • Allows quantification of all analytes of interest. • Little or no interference observed in the region of interest, confirmed by analysis of reagent blanks and field blanks. • Sufficiently low limit of determination with details of such parameters (e.g. LoD, LoQ, MDL).
6	Performance of the method	*Essential that there is satisfactory recovery of the test substance to give confidence that the monitoring data are valid.* • A set of recoveries for the analytes of interest have been carried out at different spiking levels to cover the likely monitoring concentrations. • Recovery data are >70% and with acceptable standard deviation. • Appropriate external standard has been used for recovery. • Internal standard, if appropriate, has been used in method.

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