Developing and validating an analytical method to detect PCB

Vanessa BEL

Introduction

Residual muds of purification stations contain polycyclic aromatic hydrocarbons (HAP), polychlorinated biphenyls (PCB), etc... These muds can be incinerated or used to spread. However PCBs are dangerous in large quantities for the environment and people. This is why some administrations have asked LAEPS (Laboratoire d'Analyses Environnementales des Pays de Savoie) to control the level of PCBs in purification mud before their spreading and sometimes in sediment. In fact the maximum authorized limit of PCB in the purification mud is 800 µg.kg-1 of dry material for the addition of the seven principal PCB (PCB: 28, 52, 101, 118, 138, 156 and 180). Therefore, the laboratory would like to develop a more accurate method than the previous one which overestimated three of the seven principal PCBs.

Experimental Conditions

Development of the method
Methods of PCB analysis on mud are in norm XP X33-012 (2000 march). This norm includes several methods for each stage of the analysis. LAEPS choose these four stages for the extraction of the sample:

1. Extraction by acetone-hexane
2. Concentration
3. Purification by copper and sulphuric acid
4. Concentration

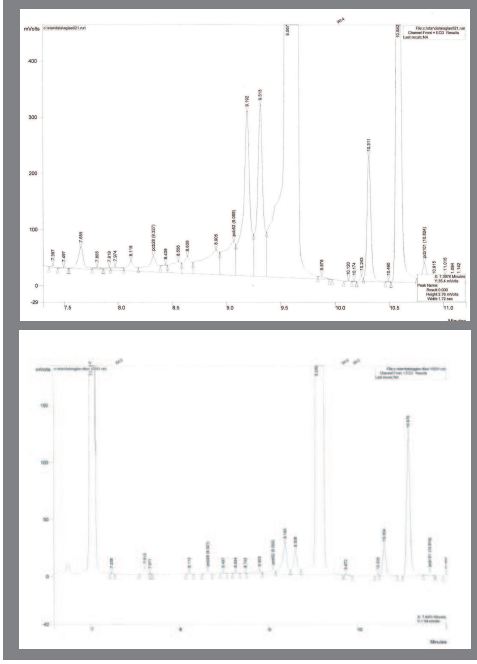
After that, the sample extract was diluted by 10 in the solvent (hexane) to minimize impurities which could interfere with PCB peaks. Then, the sample was analyzed with a gas chromatograph and an electron capture detector (GC-ECD). The following parameters were used for the analysis:
Temperature gradient:

Rate (°C/min)	Temperature (°C)	Time (min)
	130	2
10	250	0
30		4.67
Time of analysis		21

In comparison with this method and so as to adjust the methodology, retention time intervals of each PCB were reduced, and functions have been added to remove the impurities' peaks.

Validation of the method
In order to validate this new method of analysis, six standards from 10 µg.L-1 to 1000 µg.L-1 of each of the seven PCBs were prepared. The validation consisted of checking linearity, repeatability and accuracy of this new method.

Figure1: chromatogram of AGLAE (standard artificial mud) obtained with GC-ECD base method
Figure2: chromatogram of AGLAE obtained with GC-ECD new method.



Results and Conclusion

Different temperature gradients have been tried to test their effects on the accuracy of the method. A relation could be found between the gradient and the analysis time: a higher duration did not ensure a higher accuracy.
There is coelution between PCBs peaks and impurities peaks, and the obtained base line remains horizontal. Moreover limits of detection and quantification for PCBs are lower than before modifications. The new method is more accurate than the previous

53, rue du Busard des Roseaux
ZI des Bordets Est
74130 BONNEVILLE