

Analysis of Kepone by Solid Phase Microextraction coupled with GC/MS

Amélie BOBICHON

Introduction

Kepone (or Chlrorodécone) is a very persistent pesticide in nature. Currently, laboratories carry out liquid-liquid extractions, which are afterwards analysed with different techniques that are costly in both solvent as well as time.

Therefore, the utilisation of another extraction method, such as the microextraction on solid phase (SPME) has come up. This technique uses a fiber covered with a polymeric phase to extract the molecule.

In the first stage, the fiber is dipped directly into a solution, which sets up a balance in molecule sharing between the fiber and the solution. Then, it is desorbed thermally in the injector of a GC/MS. In order to optimize this extraction, the different factors based on the adsorption were varied, so the extraction of the compound was changed. These factors were as follows: nature and thickness of the polymeric film, as well as the presence of salt (NaCl)

Experimental Conditions

For this test of extraction, two types of fiber were used:

- 1 Red, composed of Polydimethylsiloxane, with a thickness of 100 μm and an injector temperature of 280°C
- 1 Blue, composed of Polydimethylsiloxane/Divinilbenzen, with a thickness of 65 μm and an injector temperature of 250°C

Afterwards, salt was inserted into the standards (3g NaCl in 19mL), and new analyses were carried out.

After extraction, compounds were separated by GC, and then detected by mass spectrometry.

First, Kepone concentrated solution was used in scan mode for identification. Then, a diluted solution (0.5 $\mu\text{g/L}$) was analysed in SIR mode, where the selected m/z ions were: 272, 274, 237.

In order to quantify, various internal standards were used: PCB 101, Kepone substituted by 13C, DDT op' and Lindane.

Results and discussion

A concentrated solution was employed in Scan Mode, in order to identify Kepone, with a comparison of its spectrum with the Nist database. Kepone was identified with the retention time of 16.26 min on the chromatogram, so this one was well extracted by the fiber.

Then, diluted solutions were used in SIR mode. This mode is more sensitive, and accordingly it was used for quantification. Consequently, this mode was also used to obtain the lowest detection limit.

The influential parameters were then varied, in order to determine the optimal conditions of adsorption.

In this way, as seen in figure 1, the various signals of noise were obtained. It appears the Blue fiber adsorbed the compound better than the Red one; however, the use of salt decreased the detection of the molecule. Therefore, the Blue fiber will be chosen. Furthermore, this one showed a detection limit of 0.05 $\mu\text{g/L}$.

This method is not highly repeatable; the use of an internal standard is essential. Out of the four tested internal standards, only one, the lindane, has given convincing results in a range of calibration 5x5 points. Indeed, the calibration curve (Figure d) obtained is acceptable.

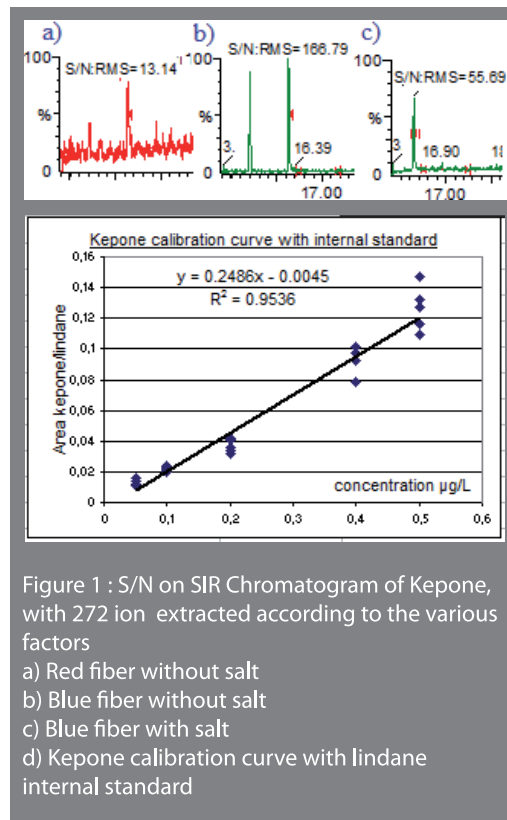


Figure 1 : S/N on SIR Chromatogram of Kepone, with 272 ion extracted according to the various factors

- a) Red fiber without salt
- b) Blue fiber without salt
- c) Blue fiber with salt
- d) Kepone calibration curve with lindane internal standard

Conclusion

This technique can be validated to quantify Kepone, with the Blue fiber and lindane internal standard, and ultimately used routinely.



La Drôme-Laboratoires
BP118, 37 avenue de Lautagne,
26904 Valence cedex 9