Development method in bidimensional gas chromatography of a known blend.

Tiffany MARRE

Introduction

The team "Raffinage" at IRCELYON (Institut de Recherches sur la Catalyse et l'Environnement de Lyon) performs researches on the hydrocraking of petroleum products. Bi-dimensional gas chromatography is becoming essential to characterize hydrocracking of petroleum products as it is a multidimensional comprehensive chromatography therefore it is a powerful tools to analyze complex real samples.

Experimental methods

The principle of GCxGC is based on the hyphenation of two capillary GC columns of different selectivity connected through a modulation device, here, micro fluidics device. This interface enables sampling focusing by trapping successive fractions of the effluent coming from the first column in narrow bands onto the second column. They are re-injected in a continuous way of sharp fractions of the first column's effluent into the second column. The entire sample is then analyzed and reaches the detector.
In our case, the sample is submitted to two dimensions according to polarity (first dimension) and to volatility (second dimension).

GCxGC material

All experiments were carried out using a GC Agilent Technology and an FID detector (350°C, 20 Hz).
The first dimension column was a 15x100x0,1 (ZB-35).The temperature program of 50°C, ramped 2,1°C/min to 320°C an H_2 flow of 0,1 ml/min.
The second dimension column was a 5x250x0,25 (ZB-01).The temperature program of 50°C, ramped 2,25°C/min to 340°C an H_2 flow of 28 ml/min.
The modulator was a micro-fluidics device with a modulation delay of 0,02 minutes. The injection was performed with a MMI.
1µL of sample was injected at 500°C with a split of 1:100.
Other parameters were identical during the whole experiment.

Results and discussion

The first set of chromatograms showed the second dimension was too quick and compounds were impossible to separate (Fig.1) Optimization of the injection and the modulation device needs to be resolved as there are issues with tailing peaks and the flow rate.
A study of modulation time has been realized so the best modulation delay was 0,14s and the injection has been made at 5µL for a split of 1:250 (Fig.2).

The first and the ending Chromatogram

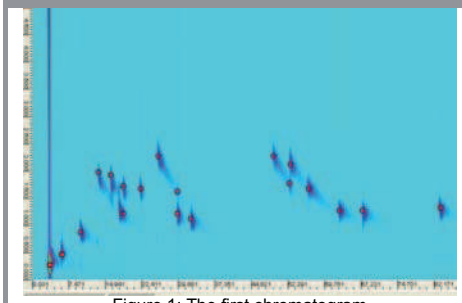

Figure 1: The first chromatogram

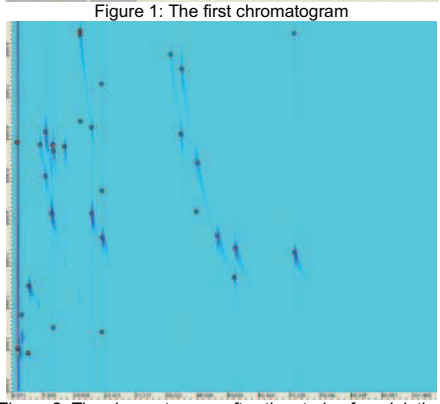

Figure 2: The chromatogram after the study of modulation time and injection

IRCELYON
2 avenue Albert Einstein
9626 Villeurbanne cedex