Analysis of surface active agent in mineral oil by Heart-cut EGA-GC/MS

[Background] Mineral oils are widely used as raw materials in the production of lubricants and cosmetics. Depending on the application, a variety of additives such as surface active agents are added to modify the properties of the mineral oil. The additives in the oil are analyzed by GC/MS; however, mineral oils are complex hydrocarbons mixtures. Often the additive of interest is difficult to identify and quantitate due to the hydrocarbon interference. This report describes the analysis of a surface active agent in mineral oil by using the Heart-cut EGA-GC/MS technique. The heart cut is chosen so that only the portion of the sample containing the target compounds are transferred to the separation column. The portion of the sample eluting before the target compound(s) is vented. The portion of the sample eluting after the target compound(s) remains in the sample cup.

[Experimental] The sample was a mineral oil: Hygold 100 (Ergon Refining, USA). Hygold 100 contains about 500 ppm of sodium di(2-ethylhexyl)sulfosuccinate (DESS) as a surface active agent. The Double-Shot pyrolyzer included a selective sampler. The selective sampler enables the analyst to either vent or analyze individual EGA thermal zones.

[Results] The EGA thermograms of Hygold 100 and DESS are shown in Fig. 1. The hydrocarbons which make up the bulk of Hygold 100 evolve between 100-180ºC. DESS decomposes between 180-270ºC to give di(2-ethylhexyl)fumarate (DEF). Fig. 2 shows the assumed mechanism in which $NaSO_3$ is eliminated from DESS to form DEF. Based on this result and using the peak of DEF formed during the thermal desorption between100-300ºC, the quantitative analysis of DESS in Hygold 100 can be done. However, when a Hygold 100 sample containing about 500 ppm of DESS was analyzed by thermal desorption GC/MS without heart-cutting, the DEF peak co-eluted with the hydrocarbons present in Hygold 100 as shown in Fig. 3-a; consequently DEF could not be identified using the TIC. The only indication that DEF was present was the mass chromatogram (m/z 112), with a S/N of 11. The impact of the interfering compounds on the DEF determination was reduced by heart-cutting the temperature zone over which DEF is formed. The result obtained by heart-cutting the 160-300ºC zone (Fig. 1) is shown in Fig. 3-b. Interference with the DEF peak was almost eliminated. DFE could be clearly observed even on the TIC. On the mass chromatogram, the S/N ratio was improved by a factor of 10. Reproducibility was 4.7 %RSD (n=5) which was well within project's DQO (data quality objectives). The heart-cutting technique can easily be automated using the Multi-Shot Pyrolyzer with Auto-Shot sampler.

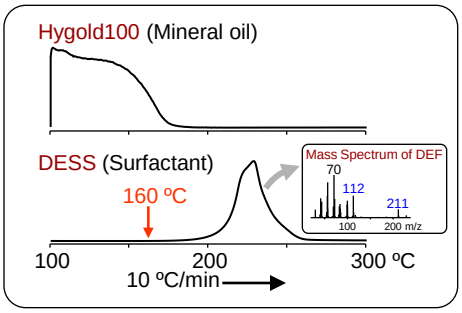

Fig. 1 EGA thermograms of DESS and Hygold100

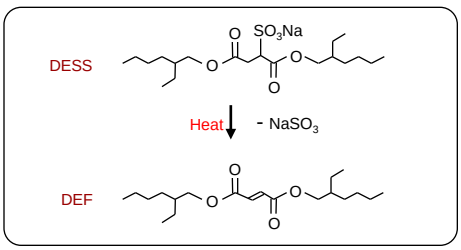

Fig. 2 Thermal decomposition of DESS

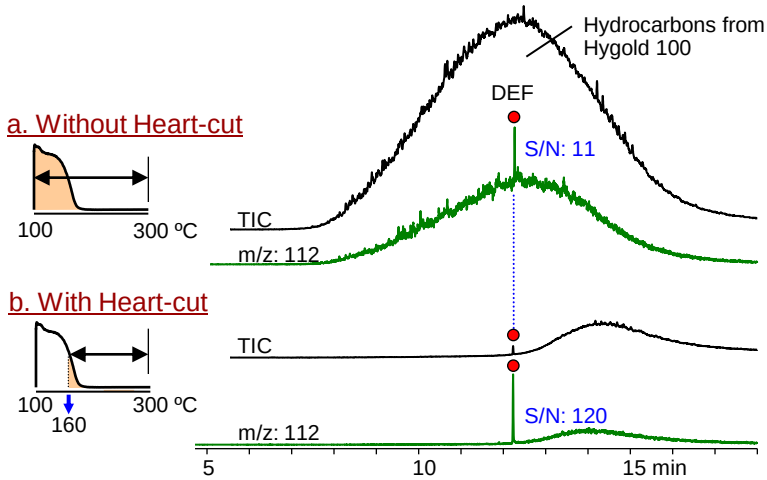

Fig. 3. Quantitative analysis of DESS (500 ppm) contained in Hygold100 using Heart cut–GC/MS

Pyrolysis temp.: 100 – 300 ºC (10 ºC/min), GC oven temp.: 40 – 320 ºC (20 ºC/min, 4 min)
Separation column: Ultra ALLOY+-5 (5 % diphenyl 95 % dimethylpolysiloxane, L = 30 m, i.d. = 0.25 mm, df = 0.25 µm), Column flow rate: 1 mL/min He, split ratio: 1/20, sample: 100 µg

Keywords : Mineral oil, surface active agent, Heart-cut, Thermal desorption, GC/MS

Products used : Multi-functional pyrolyzer, Selective Sampler, Vent-free GC/MS adapter, UA-5

Applications : Fats and oils, Cosmetics

Related technical notes : PYA1-012E, PYA1-031E

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