

ナトリウム溶融後ガスクロマトグラフィーによる フェナントレン中のジフェニールスルフィドの同定

ジフェニールスルフィドの保持時間はフェナントレンのものに近い¹⁾ため、そのままでは同定に困難することがある。そこでジフェニールスルフィドをナトリウム溶融してビフェニールに変化させるとガスクロマトグラフィーによる同定が容易になることを見出した。

Identification of Diphenylenesulfide in Phenanthrene by Gaschromatography after Na fusion

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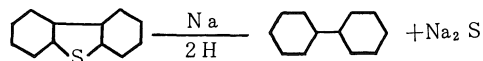
Outline

Diphenylenesulfide (DPS) has a retention time very close to that of phenanthrene. And so we find difficulties some times to identify the contaminant in phenanthrene on gaschromatography. The authors established a new method for this purpose.

This method consists of two processes of Na fusion and gaschromatography. DPS is decomposed to biphenyl which we can identify very easily instead of DPS on gaschromatography.

1 Introduction

G. Schroter and his coworkers gave an evidence of the DPS present in phenanthrene commercial by Na fusion. B. D. Blaustein and others²⁾ referred to the desulfurization of phenanthrene by the same method. It seems not so easy to find any other literatures concerned in this problem besides these two. Usually DPS appears at the foot of phenanthrene peak on gaschromatography (GLC). But it's somewhat difficult to discriminate the DPS peak, if contained so much as more than 1% or so. Owing to this reason Na fusion was used here to convert DPS into biphenyl and detect its sharp peak on the chart. The reaction occurs as follows.



But we should be very precarious for protecting the interference by the decomposition products of phenanthrene during Na fusion. The result of investigation of this problem made us sure that biphenyl was not generated from any one of phenanthrene, fluorene and pyrene by this process.

2 Experimental process

Various kinds of phenanthrenes were zone-refined for 3 days with an elevation speed of $4.4 \frac{\text{cm}}{\text{hr}}$ and cut off into eight or nine sections, which were numbered 1, 2, ..., 8 from the top of zone-refining. About 10 mg of the selected section described in the next paragraph was added to 0.5~0.8 g metallic Na in a small test tube and heated directly over a Bunsen burner under continuous shaking until the contents were colored black soon after. The benzene extract was dehydrated with CaCl_2 and evaporated. The residual powder was injected by the author's method³⁾ in the GLC.

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3 Identification of Sulfur compound in Phenanthrene

Only one pair of charts were selected from each of four companies' phenanthrenes for the sake of saving space. And the huge peak of major component, phenanthrene was also cut off in every chart, but written with figures responsible for the length and width in the middle of the peak.

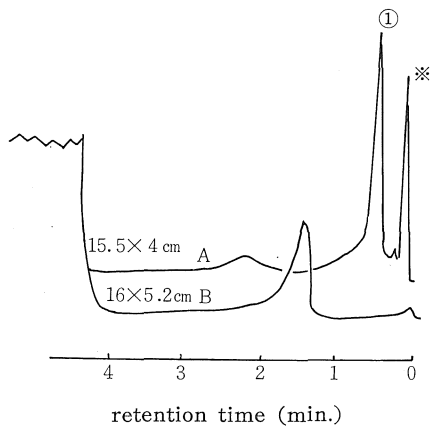


Fig. 1 GLC of the desulfurization product of TZ8

Yanagimoto 5DH FID
 column : stainless steel 0.75m, 0.3cm
 PEG 20M 2% on celite 545
 Ct 150°C N₂ 7.5m 1/min
 It 250 H₂ 30 "
 Et
 S 10⁹Ω Air 0.9 1/min
 Att. $\frac{1}{2}$

1 : Biphenyl * Injection shock
 T : compahy, Z8 : zone-refined section
 number from the top
 A : afte Na fusion
 B : before "

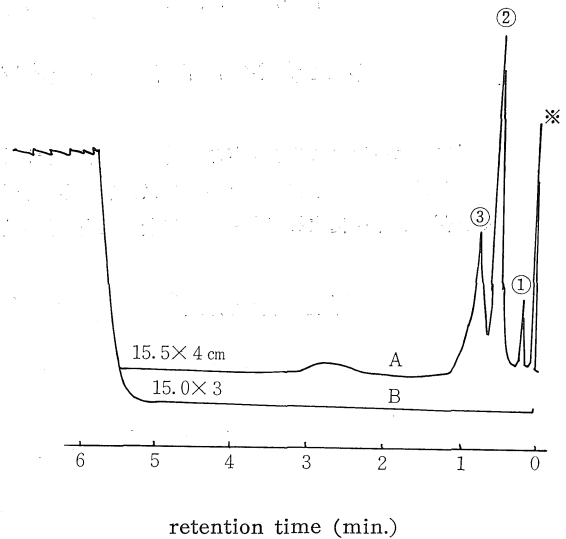


Fig. 2 GLC of the desulfurization product of KZ6

conditions : the same with Fig. 1
 1 : Biphenyl

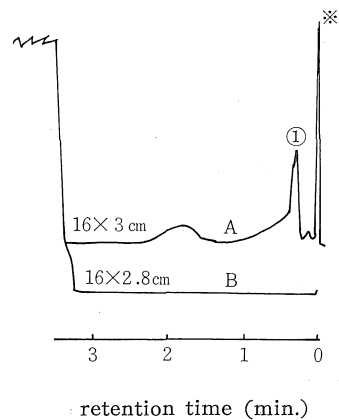
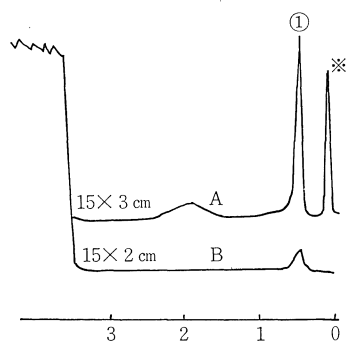


Fig. 3 GLC of the desulfurization product of NZ6

conditions : the same with Fig. 1
 except for Ct : 120°C

1 : Naphthalene
 2 : Biphenyl
 3 : Methylbiphenyl



retention time (min.)

Fig. 4 GLC of the desulfurization product of YZ6

conditions : the same with Fig. 3

1 : Biphenyl

4 Conclusion

Na fusion is a simple and convenient method for the determination of DPS, one of the contaminants in phenanthrene. But the application of this method is limited to phenanthrene, because of its stability against Na fusion. In every case a blank test should be tried along with the sample.

Literature

- 1) B. D. Blaustein and others:
Anal.Chem., 37 2295(1965)
- 2) G. Schroter and others :
Ber. 62 650(1929)
- 3) K. Kihara : Analytical Instrument,
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