§4. Improvement of Infinitesimal Concentration Hydrogen Analyzer

Kawano, T., Uda, T.,
Tsuboi, N., Tsujii, H. (GTR TEC)

An analyzer able to detect extremely small concentrations of dideuterium in diprotium gas was developed. A previous study concluded that the analyzer needed improvement before it could be applied to measuring dideuterium concentration of a gas sample with diprotium as the major component and dideuterium as a minor component at very low concentration. Because peaks corresponding to deuterium molecules in a chromatogram obtained would appear in the tail of the main peak corresponding to diprotium within the same chromatogram. Consequently, in the present study developing an analyzer able to detect extremely small concentrations of dideuterium in a diprotium gas sample, distinguishing a peak corresponding to dideuterium from a peak corresponding to diprotium in actual measurement is a significant problem. To overcome this limitation, the original analyzer was improved by employing a pre-cut method. The improved analyzer consisted of two components, a gas chromatograph and an atomic absorption spectrophotometer. The gas chromatograph is distinctive in the use of two gas line switches.

Gas line switch 1 is employed especially for removing impurities that may disturb the measurement of infinitesimal concentration hydrogen. Gas line switch 2 is used essentially to remove all of the protium molecules. Figure 1 illustrates the function of gas line switches 1 and 2.

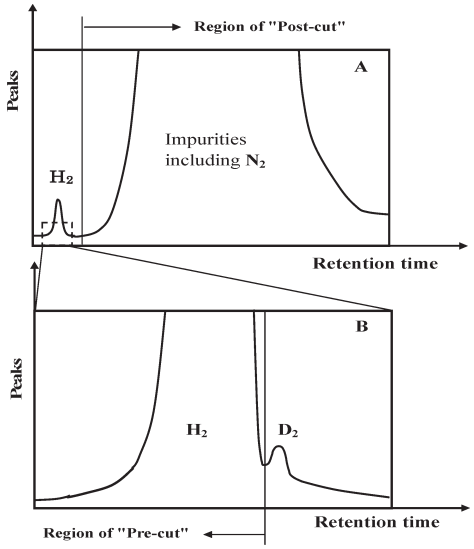

Fig. 1. Gas chromatograms obtained for: (A) a gas sample containing hydrogen molecules and large amounts of impurities including nitrogen, and (B) a gas sample containing dideuterium in a large amount of diprotium.

When the gas sample undergoes chemical separation, dihydrogen appears first at the outlet of the separation column; other elements, including nitrogen, follow as shown in Fig. 1A. Gas line switch 1 is operated so that impurities in the later part of a chromatogram (Fig. 1A) are eliminated. This gas element separation technique is termed the post-cut.

Figure 2B illustrates separation of the dideuterium using gas line switch 2. After the gas element separation using gas line switch 1, carrier gas containing only dihydrogen is supplied to a hydrogen isotope separation column. At the outlet of the column, diprotium appears first, followed by dideuterium as shown in Fig. 1B. When a gas sample contains a very small amount of dideuterium and a large amount of diprotium, a large peak and a very small peak corresponding to the diprotium and dideuterium, respectively, appear in the chromatogram (Fig. 1B). The column cannot practically separate the dideuterium from diprotium, because the small peak corresponding to dideuterium is hidden in the tail of the large diprotium peak. In the improved analyzer, the major diprotium component in the carrier gas is released to the outside of the analyzer with proper operation of gas line switch 2. This procedure eliminates almost all of the diprotium from the analysis, reducing the area of the diprotium peak and increasing isolation of the dideuterium peak in the chromatogram, being called the pre-cut.

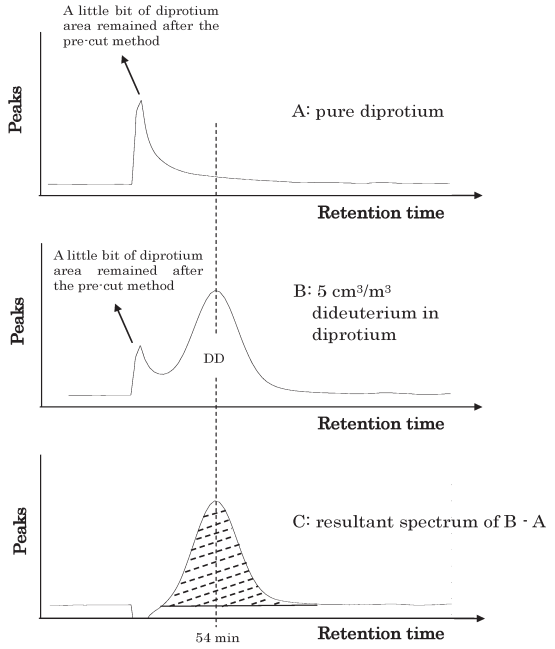

Fig. 2. Chromatograms measured by the improved analyzer and B-A

Figure 2 shows three typical chromatograms of diprotium gas (A), the 5 cm^3/m^3 dideuterium sample (B), and the resultant chromatogram (C = B-A) obtained by the improved analyzer. The peak with rapid rising and slow tailing found in the early part of the chromatogram (A) corresponded to remained diprotium. The peak had a distorted shape because the major part of peak area was eliminated by applying the pre-cut method and just a little bit of tailing part was remained. Examination of (B) indicated that the retention time of the dideuterium peak was 54 min. The resultant chromatogram (C) shows that the improved analyzer analyzes minor part of dideuterium well separating from major part of other hydrogen molecules.