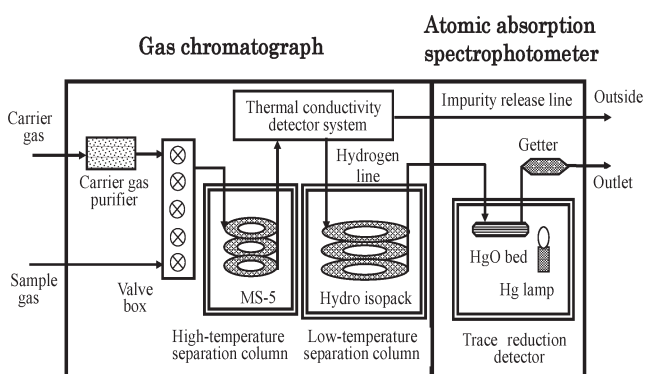


#### §4. Isotopic Separation Analysis Using Improved Hydrogen Analyzer

Kawano, T., Yamato, A., Uda, T.  
 Tuboi, N., Tsuji, H. (GTR Tec)

We previously developed an analyzer that used a trace reduction detector to measure infinitesimal concentrations of hydrogen in air. The detector detects extremely small amounts of hydrogen by using the reduction reaction of mercuric oxide with hydrogen. Experiments demonstrated that the analyzer can measure concentrations as low as  $5 \text{ cm}^3/1000 \text{ m}^3$ . The analyzer is equipped with a carrier gas purifier, high- and low-temperature separation columns, and an “after-cut method” is used to remove impurities,

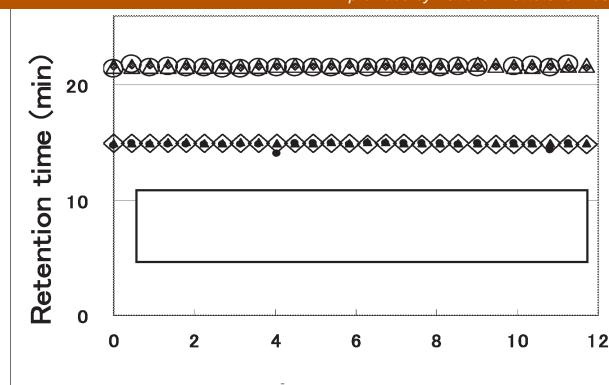


including nitrogen, contained in the sample gas. Experiments using protium and deuterium separately demonstrated that the analyzer works well regardless of differences in the hydrogen isotope. We have used the analyzer to distinguish hydrogen isotopes in three gas mixture test samples (protium and deuterium with almost equal concentrations in nitrogen). Nitrogen gas was used as the dilution gas to simulate an air sample.

We repeatedly measured the concentrations of protium and deuterium in each sample for about 12 hours and obtained more than 30 chromatograms for each. Then we examined these chromatograms from the viewpoints of retention time stability and peak area stability.

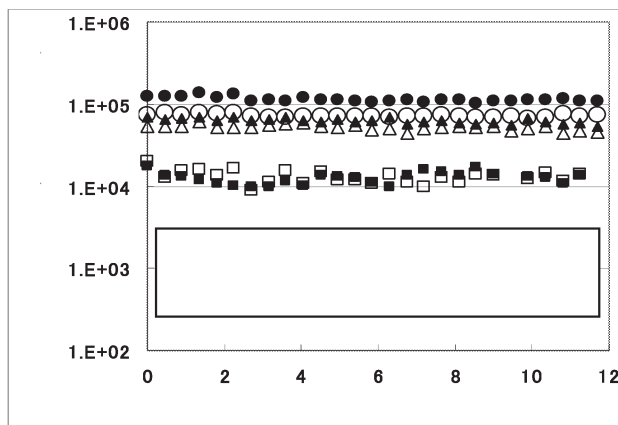
The retention times obtained for the three test sample gases are shown in Fig. 2. The elapsed time in hours, shown on the X axis, indirectly represents the repetition time of the measurements. Because each measurement took about 27 minutes, the total elapsed time divided by 27 equals the repetition time. The Y axis shows the retention time in minutes.

As shown in Fig. 2, all the data for the three sample gases are distributed closely along one of two horizontal lines, although the lines themselves are not actually drawn. The upper line corresponds to the retention time obtained for deuterium ( $D_2$ ), and the lower corresponds to that for protium ( $H_2$ ). The retention times clearly differ between the two. Moreover, the two times are constant and virtually



indistinguishable, independent of the concentration and repetition time. The average retention times (and relative standard deviations) were 14.8 min (1.45%), 15.0 min (0.26%), and 14.9 min (0.25%) for the protium in the 5-, 20-, and 50- $\text{cm}^3/1000 \text{ m}^3$  sample gases, respectively. For deuterium they were 21.6 min (0.47%), 21.7 min (0.17%), and 21.6 min (0.22%). The difference between the protium and deuterium was about 6.5 minutes. The retention times were thus distinguishable and very stable.

The changes in the peak areas in the chromatograms obtained for the three test sample gases are shown in Fig. 3. While they fluctuated from trial to trial, the lines connecting them are still horizontal. The relative standard deviations derived for the protium and deuterium in the 5-, 20-, and 50- $\text{cm}^3/1000 \text{ m}^3$  sample gases were about 17.3, 8.3, and 7.3%, and 18.3, 8.8, and 4.6%, respectively. It seems reasonable that the deviation increased as the concentration was reduced for both the protium and deuterium.



These results indicate that the peak areas obtained at the same time for both elements were stable, independent of the elapsed time and repetition time. This means that the analyzer can detect concentrations of protium and deuterium as low as about  $5 \text{ cm}^3/1000 \text{ m}^3$ .