

*On the Preparation of Isotopically
Pure Light Hydrogen*

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Natural abundance of heavy hydrogen isotope in the water is known to vary depending on the geochemical circumstances in which the water is found¹⁾. The standardization of the value of D-content in the water or the preparation of isotopically pure light hydrogen was thus needed and became the object of careful investigation particularly by Titani and his co-workers^{2,3)}. Electrolysis of water is known to produce hydrogen of less D-content. Combustion of the hydrogen followed by re-electrolysis leads to much less concentration in the heavy component. Repeating such procedure, these authors were able to obtain 125 g. light water, the atomic D% of which being $0.0002 \pm 20\%$, i.e., about one-hundredth of natural abundance.

Current investigation on the chromatography, on the other hand, showed that this technique is powerful for the separation of hydrogen isotopes. Glueckauf and Kitt⁴⁾ were able to isolate pure deuterium (99.5%) by a displacement technique using palladium as column packing. The work⁵⁾ of this laboratory proved that hydrogen isotopes are separated in the elution chromatogram when synthetic zeolite (Linde molecular sieve) is used as column packing and kept at -195°C .

Deuterium was found to adsorb strongly compared with ordinary hydrogen on this packing. As a result, self-displacement of a mixture of hydrogen isotopes through such a cooled column yielded pure hydrogen in the frontal band and then HD and D₂ in the succeeding band⁵⁾. The detection of hydrogen isotopes were made in this case by the thermal conductivity gauge equipped in Shimadzu Gas Chromatograph GC 1A.

1) M. Harada and T. Titani, This Bulletin, 10, 206, 263 (1935); G. R. Clarke, *Nature*, 174, 469 (1954).

2) T. Titani, S. Horibe and M. Kobayakawa, *Mass Spectroscopy*, 9, 32 (1957).

3) T. Titani, S. Horibe, T. Komori and M. Kobayakawa, *ibid.*, 9, 40 (1957).

4) E. Glueckauf and G. P. Kitt, "Vapour Phase Chromatography", Butterworths Scientific Publications, London, (1957), p. 422.

5) S. Ohkoshi, Y. Fujita and T. Kwan, This Bulletin, 31, 771 (1958).

It is obvious that the latter technique is promising to obtain isotopically pure light hydrogen. An attempt was made therefore to investigate the isolation of light component from ordinary hydrogen by means of such chromatographic technique. A spiral Pyrex column 400 cm. long and 0.5 diameter was filled with molecular sieve 5A and degassed overnight at 300°C by mercury diffusion pump. Two liters hydrogen sample from commercial electrolytic hydrogen* was introduced into the one side of the column immersed in liquid nitrogen. About ten minutes later hydrogen was found to appear on the other side of the column. The initial portion of the hydrogen (100~200 cc. N. T. P.) was then collected in a flask (sample I). The remaining hydrogen, together with those adsorbed on the packing, were collected back to the original flask (sample II). The deuterium content of the two samples were analyzed by Hitachi Mass Spectrometer RMD-3. The result is shown in Table I.

TABLE I

Run	Hydrogen sample	Volume cc. N.T.P.	Atomic D%	Error
1	I	100	0.0000	± 0.0005
	II	1900	0.0068	
2	I	200	0.0004	± 0.0005
	II	1800	0.0068	

As shown in this table, the sample I was found almost perfectly free from heavy component within the accuracy of the mass spectrometer. Thus, it is quite evident that the chromatographic process is simple and efficient to prepare isotopically pure light hydrogen. At the same time, the process is of practical importance to enrich deuterium or tritium at least in laboratory scale⁵.

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