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A simple gas chromatographic method for normalizing ^{18}O enriched water

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The enrichment of water in ^{18}O leads to the enrichment in deuterium as well. However, there are quite a number of experiments for which $H_2{}^{18}O$ with natural deuterium abundance is needed since the isotope effect of deuterium exceeding that of ^{18}O by an order of magnitude may present considerable inconvenience. To eliminate this disturbing effect the water must be "normalized".

Normalization by the techniques reported so far, such as isotopic exchange with H_2, H_2S, NH_3 by catalysis; electrolysis, requires operations of more than one step and an expensive apparatus. Thus these techniques are hardly practicable for small samples, except the method which involves an alternate oxidation and reduction on iron catalyst [1].

Considering that a gas-liquid chromatographic column has already been successfully applied for isotopic exchange by several authors [2, 3, 4, 5], this method was chosen for varying the deuterium concentration of ^{18}O enriched water samples while the ^{18}O concentration remains unchanged.

The experiments were performed on U-shaped glass columns, 2 m in length and 20 mm in diameter. The column was packed with 25% diglycerol (B. D. H. for gas chromatography) on celite. ^{18}O enriched water samples of 0.3 ml were injected into the column heated to 95° C. The flow rate of the purified nitrogen carrier gas was set to get a 30-40 min. retention time. It was found that up to the exchange of 80% of the exchangeable hydrogen atoms of diglycerol with deuterium, the deuterium content of the water samples did not exceed the natural isotopic abundance. The concentrations of D and ^{18}O were determined in the normalized samples with the use of mass spectrometer, type MI-1305. The column was regenerated and stabilized as soon as a sample with deuterium content was observed at its outlet. An example of the isotopic analysis of a typical normalization process is the following. Before the treatment the D-, and ^{18}O-content of a water sample was 7.0 ± 0.1 at%, and 70 ± 1.3 at%, respectively; after the treatment 0.02 ± 0.001 at%, and 70 ± 1.3 at%, respectively. There was neither ^{18}O isotopic dilution, nor any "memory" in the column due to the exchange.

The advantages of this method over the earlier techniques can be summarized as follows. Normalized $H_2{}^{18}O$ samples of high chemical purity can be obtained from ^{18}O enriched water at any concentration of deuterium.

The method can be applied to very small samples without any loss of water. It does not require any special apparatus since a gas chromatograph is a standard equipment in almost any chemical laboratory.

Alternatively, the method can be applied for enriching the D content of ^{18}O enriched water samples. In this case the column packed with diglycerol has to be deuterated with D_2O before the injection of the samples.

Considering that the cost of normalization of the commercially available ^{18}O enriched waters can be estimated from the price lists (e.g. [6]) as about an additional 10 % of the cost of the water, the present method seems to be more economic for any number or volume of samples.

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