Investigation of BDH Zinc for Hydrogen Isotope Analysis by Mass Spectrometry

A. Tanweer

Isotope Hydrology Laboratory, International Atomic Energy Agency, P.O. Box 100, A-1400 Vienna, Austria

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Using metallic zinc from BDH Chemicals, the conversion of water into hydrogen was investigated by measuring the D/H ratio of the latter. It was found that 8 µl of water, 3.0 g of zinc, and a reaction time of 30 minutes at 480 °C are the optimal conditions for the conversion reaction.

Zinc is required by many laboratories in the world for the reduction of water to hydrogen for the determination of the deuterium concentration in natural waters. Most laboratories are using BDH Anal R Zinc shot 0.5–2.0 mm. The optimal conditions for the preparation of hydrogen from this particular zinc have been given in [1]. For reasons not fully known, however, BDH Chemicals Ltd., Poole, UK is no more able to supply this particular type of zinc. Instead, BDH now supplies BDH Anal R Zinc coarse powder 0.3–1.5 mm. Using this zinc, it was not possible to get complete reduction by applying the conditions mentioned in [1] for hydrogen sample preparation. Most of the laboratories which receive technical assistance from the IAEA were enquiring what to do as the zinc which was recommended is not available. Therefore it was decided to make some investigations on this new type of zinc.

The results are as follows:

(a) The cleaning procedure of zinc remains the same as mentioned in [2]. Though this new zinc is much finer and dustier, the use of concentrated HNO_3 led to an incomplete reduction of water.

(b) As before, the amount of water per analysis was taken to be 8 µl.

(c) Samples were prepared to find the optimal amount of zinc required per analysis. Starting with 0.25 g, the amount of zinc was increased gradually in steps of 0.25 g. Till 2.50 g the reaction was incomplete; one could see the water condense in the sample preparation tube as it cooled. With 3.0 g the reaction was complete; the amount was further increased to 3.25 g, but there was no change in the results. The older type of zinc showed an amount effect: By using more than the optimal amount of zinc, which was 0.25 g, an adsorption of hydrogen was observed [1]. In order to see this effect in the new type of zinc, one has to increase the amount of zinc to 6.0 g per analysis. The aim of this investigation was to find the optimal amount required per analysis. It has been observed that the zinc grains join and form kind of a small ball during the reduction of water, which is not the case with the older type of zinc. The reaction takes place on the surface of the zinc grains. This surface decreases due to the formation of the ball, thus a higher amount of zinc is required in order to obtain enough surface for the complete reduction of water. The small amount of zinc that volatilizes to form a ring on the colder part of the sample preparation tube is not as prominent with this new type.

(d) The temperature must be increased by 20 °C, i.e. from 460 °C to 480 °C. Working at a lower temperature gave poor reproducibility of the analysis. The zinc grains can withstand a higher temperature than the melting point of zinc because of the zinc oxide film formed over the zinc grains, which has a much higher melting point.

In order to show the validity of the procedure and the reproducibility of the analysis, samples were prepared from two water standards: (1) an international reference GISP (Greenland Ice Sheet Precipitation); and (2) VTW (Vienna Tap Water) an internal laboratory standard. The results are shown in Table 1. The deuterium contents are expressed in terms of δD, i.e.

Table 1. Measurements of δD (‰) of GISP and VTW relative to VSMOW.

	GISP	VTW
	−190.7	−82.8
	−190.5	−83.6
	−190.1	−82.9
	−190.8	−81.1
	−190.5	−83.9
	−190.4	–
Mean	−190.5	−82.9
$\sigma (n-1)$	0.24	1.09

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the per-mille (‰) deviation of the isotope ratio from that of VSMOW (Vienna Standard Mean Ocean Water) as defined by Craig [3] and Gonfiantini [4].

$$\delta(‰) = \frac{R_{\text{sample}} - R_{\text{vsmow}}}{R_{\text{vsmow}}} \cdot 10^3$$

where R is the D/H of the sample or VSMOW.

The results are in excellent agreement with the recommended value of GISP $\delta D = -189.8‰$ [5] and VTW $\delta D = -82.8‰$ (average of four laboratories).

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