Measuring Hydrogen in Ground Water:

Introduction: Hydrogen can be added to groundwater as a terminal electron acceptor during remediation. It is important to monitor the level of hydrogen being added to ensure that it is being distributed throughout the aquifer and in order to maintain the appropriate concentration. A customer (an environmental laboratory) inquired if it would be possible to measure trace levels (p.p.b.) of hydrogen in ground water in order to provide analytical support to a remediation project.

Experimental: A Varian 3400 GC was configured with a gas sampling valve, a ShinCarbon ST micro-packed column and an A.I. C. dielectric barrier discharge (D.B.D.) helium ionization detector (H.I.D.) . Samples were collected in large volume gas sampling bulbs with no headspace and transported from the field to the laboratory. For analysis, a small volume of water was removed from the sampling bulb to create a headspace within the bulb and the sample was shaken briefly. A gas tight syringe was used to withdraw an aliquot of the gas in the headspace and loaded into the gas sampling valve. The G.C was maintained isothermally at 50^0 C while the detector was kept at 220^0C. In order to get down to p.p.b levels, it is necessary to use a large volume of water in the sampling bulb and use a large sampling loop (1 c.c.) The H.I.D. used the 3400 heater base and 3400 FID electrometer without modification. Standards and samples were analyzed on the range 12 setting of the electrometer. Helium reaction gas to the D.B.D. plasma was provided by the make-up gas flow controller from the removed F.I.D. detector

Results: Below is a chromatogram from the customer showing the analysis of a 1.75 p.p.b standard prepared in laboratory reagent water. Next to that is an expanded view of the hydrogen peak that shows the signal to noise at this level. The large peak at 1.5 minutes is the air peak from the 1 c.c. injection of bulb headspace. Even with a large volume of gas injected the detector will return to baseline in a matter of minutes.

What is not shown is the water. At 50 ^{0}C isothermal, water will eventually elute from the ShinCarbon column as a broad tailing peak. Operating in an isothermal mode at a moderate temperature it is possible to run several injections back to back for hydrogen measurement prior to the elution of water from the column. In this case, up to five injections were made prior to baking the column at 200^0C for about 15 minutes and then returning to the isothermal conditions for more analysis. Standards in reagent water were analyzed after every ten injections of customer samples.

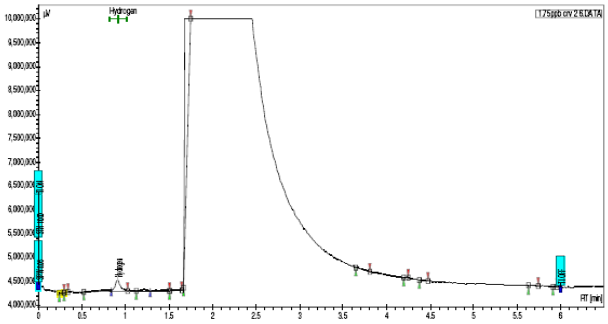

Figure 1: Chromatogram of hydrogen in water by by headspace extraction.

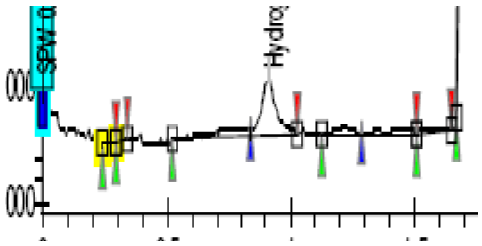

Figure 2: Close up of 1.75 p.p.b. hydrogen peak.

Conclusion:

Using the appropriate sample volume and a large volume gas injection loop along with a helium ionization detector it is possible to measure part-per-billion levels of hydrogen in groundwater samples. In order to efficiently accomplish this analysis it is essential that the detector used is able to quickly recover from large volume injections of air and the entrained water.

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