

Use of micro-proton elastic scattering analysis to determine water content in geological powders

W.J. Trompetter^a, A.G. Reyes^b, J. Kennedy^a, A. Markwitz^{a,*}

^a Department of Ion Beam Technologies, National Isotope Centre, GNS Science, 30 Gracefield Road, Lower Hutt 5010, New Zealand

^b Marine Science Department, GNS Science, 1 Fairway Drive, Avalon, Lower Hutt, New Zealand

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ABSTRACT

Hydrogen in the form of water (H₂O), hydroxyl anion (OH⁻) and H₂ is a pervasive component in minerals, affecting geochemical processes from the surface of the earth to its deep interior and the geophysical and rheological properties of the rock. To investigate the application of ion beam techniques to the measurement of structural water (OH⁻ and H₂O), purified mineral separates, volcanic glass and synthetic borosilicate glass with known total structural water contents were selected. Mineral and glass particles <50 μm in diameter and thickness, were deposited onto Teflon filters and scanned with a focussed 2.5 MeV proton beam. Hydrogen was measured using proton elastic scattering analysis and converted to total H₂O. Heavier elements were detected via Proton-Induced X-ray Emission (PIXE). Measurements were conducted in high vacuum to detect bound water only. The water content measurements for the minerals measured showed very good agreement with bulk water measurements and literature values. This method can be used successfully to noninvasively determine the hydrogen content of individual mineral particles with >1% total water contents.

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1. Introduction

Hydrogen exists in the form of pore water (H₂O⁺), structurally bound water (H₂O⁻) in crystals and fluid/glass melt-inclusions, hydroxyl anions (OH⁻) and/or as H₂ in mineral defects. The presence of hydrogen, in whatever form, affects geochemical processes from the surface to the interior of the earth and changes the geophysical and rheological characteristics of the rock. Hydrogen is an essential and pervasive component of minerals, comprising less than a few tens of ppm in nominally anhydrous minerals such as pyroxenes [1] up to about 4% structurally bound in smectites (about 36% H₂O [2]). The determination of water loss at 110 °C (H₂O⁺) and 1000 °C (H₂O⁻) is a standard procedure in rock analysis, but the procedure requires relatively large amounts of rock powder (at least 5 g) and is affected by oxidation of multivalent cations such as Fe⁺² when heated to 1000 °C in a furnace. The water content of minerals analysed under an electron microprobe or scanning electron microscope can only be estimated by weight difference. However there are a variety of methods used to measure hydrogen, in its various forms, in geological samples e.g., infrared spectroscopy (e.g., FTIR or Fourier transform infrared spectroscopy); thermo-gravimetric methods; P₂O₅ cell coulometry; and secondary ion mass spectroscopy (SIMS) and nuclear magnetic resonance

[1]. Several ion beam analysis (IBA) methods have been developed to measure hydrogen in materials [3]. These methods have also been successfully applied to geological samples. For example, 3–4 MeV ⁴He⁺ ion beams were used to measure hydrogen via elastic recoil detection analysis (ERDA) at glancing angles [4–6]. Nuclear methods using ¹⁹F [7] or ¹⁵N reactions [8] have also been employed. Furuno et al. [9] successfully used a focussed high energy 20 MeV ¹H⁺ beam to measure water in fluid inclusions within samples up to 200 μm thick using proton–proton elastic recoil coincidence spectroscopy. A similar method at much lower energies (~2.5 MeV) commonly used in conjunction with IBA of air filters is known as particle elastic scattering analysis (PESA) which measures hydrogen in air particulate matter collected on Teflon filters [10–14]. While the limit of detection for PESA (~0.1% for H in this study) is not as low as coincidence methods (~10 ppm for H [14]), PESA is a much simpler and easier to setup method. Coincidence methods require specialised electronic units such as single channel analysers, coincidence and gating modules that can take considerable amounts of time to setup. In addition, coincidence measurements require two forward scattering particle detectors. These forward mounted detectors have a limited lifetime due to damage from the high fluxes of scattered particles in the forward directions (typically ~5 mC of accumulated measurement charge). Therefore, the simpler PESA method may be advantageous for some laboratories to use for investigating samples with >1% H₂O. This method allows simultaneous determination of both hydrogen (via PESA) and

* Corresponding author.

E-mail address: a.markwitz@gns.cri.nz (A. Markwitz).

Table 1
Proton scattering energies of various elements for a 2.5 MeV proton beam.

Element	Energy detected at 45° forward angle (MeV)
H	1.250
C	2.380
O	2.410
F	2.424
Fe	2.474

heavier elements (via (article-Induced X-ray Emission) PIXE). In this study we have used it for the first time to investigate structural water in geological samples, however, it also has the potential to be used to study other types of samples.

2. Methods

To determine the water content of particles such as geological powders, particles <50 μm in diameter were deposited onto 30 μm Teflon filters. Teflon is hydrogen-free. The particles were scanned with a 2.5 MeV proton beam focused to about 10 μm in diameter [15,16]. Both X-rays and forward scattered protons were monitored during the measurements using a Si(Li) detector (mounted at 135°, 155 eV resolution) and a silicon surface barrier detector (mounted at 45°), respectively. X-rays from particle induced X-ray emission were used to detect elements heavier than Neon. The forward scattered protons allowed hydrogen to be detected by PESA.

The detected elements are used to provide information about elemental distributions and their concentrations. When the beam was scanning over a sample, elemental distributions were mapped using an Oxford micro beams system [17]. To analyse an individual powder particle, the beam was located on the centre of a particle to allow X-rays and forward scattered proton spectra to be collected. PIXE spectra were analysed with Gupix Software [18,19]. Hydrogen was detected by measuring the elastically scattered protons in a forward direction for a proton beam also passing through the filter. At a forward scattering angle of 45°, the protons are elastically scattered or recoiled with 50% of the initial proton energy (i.e. for an incident beam of 2.50 MeV the energy of protons scattered of hydrogen is 1.25 MeV), which is much less energy than the energy

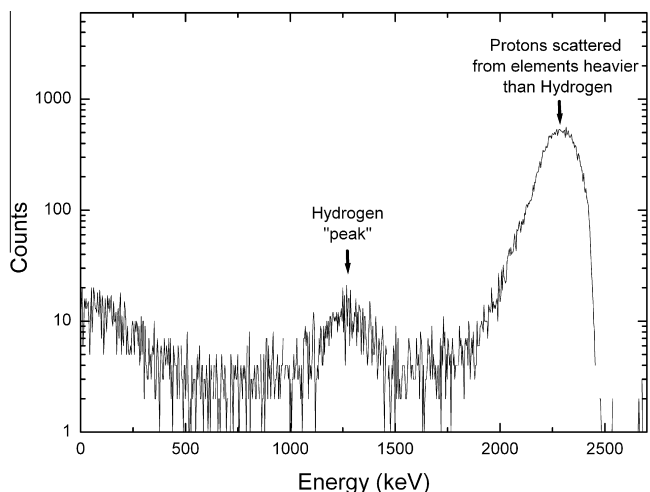


Fig. 1. Proton elastic scattering analysis spectrum of an analcite powder particle shown in Fig. 2A. The spectrum shows the location of the hydrogen peak at 1.250 MeV.

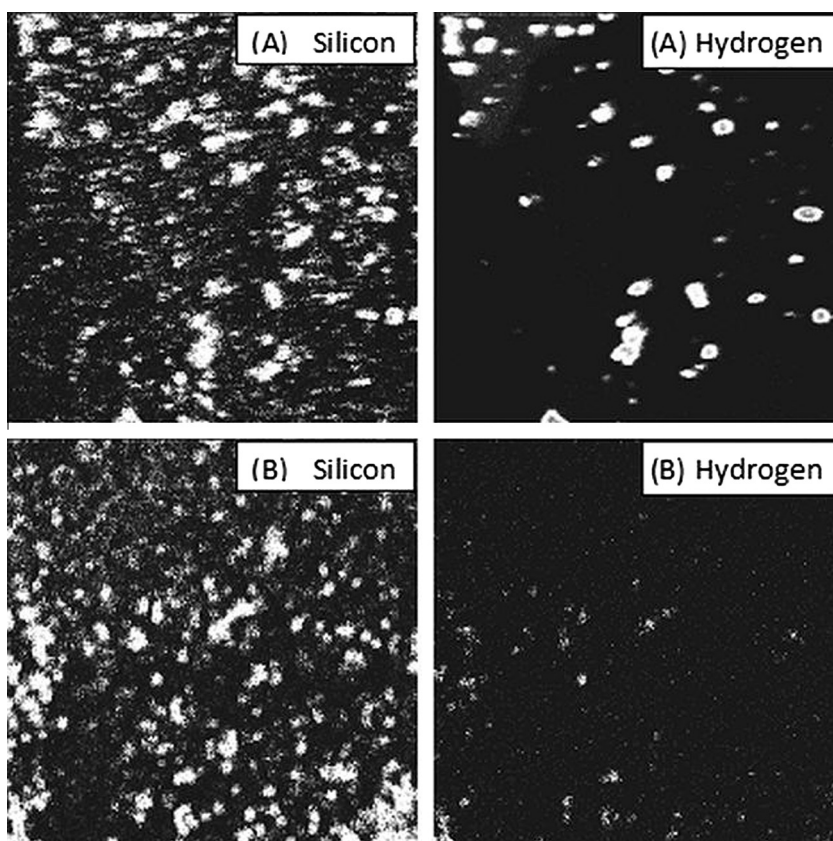


Fig. 2. (A) Silicon and hydrogen (H: $1.3 \pm 0.4\%$) elemental maps for serpentine from Mt. Arthur in South Island, New Zealand; (B) silicon and hydrogen (H: $1.2 \pm 0.7\%$) elemental maps for analcite (scan area 2 mm \times 2 mm).

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