INTRODUCTION

The determination of water in silicate glasses of different compositions, using confocal micro-Raman spectroscopy, is now a well-established routine method (Chabiron et al. 2004; Di Muro et al. 2006; Thomas 2000, 2002; Thomas et al. 2000, 2003, 2005). This technique can be used for an accurate and fast analysis of total dissolved water (H$_2$O$_T$) even in small volume (<10 µm$^3$) melt inclusions (MI). The same technique can determine D$_2$O, provided the D$_2$O-OD spectra are taken in the frequency range 2250–2900 cm$^{-1}$ with the band maxima at 2608 and 2656 cm$^{-1}$. Previous methods involved focusing the laser beam on MI exposed by polishing; however, Chabiron et al. (2004) have shown that better results can be achieved if the inclusions are at a depth of ~15 µm below the surface. According to Thomas (2000) and Di Muro et al. (2006) the optimal depth depends primarily on the confocal performance, and is about 2 µm for the Dilor XY spectrometer used in this study. Besides the quantification of water, Di Muro et al. (2006) have shown that the water speciation (H$_2$O$_m$/OH) can also be determined successfully by confocal micro-Raman spectroscopic analysis.

During our analytical work on MI from different rocks we observed apparent water-loss during secondary ion mass spectrometry (SIMS), or electron microprobe analyses (EMPA) taken prior to Raman analyses, a feature also observed by Di Muro et al. (2005), Leschik et al. (2004) and Humphreys (personal communication). At high water concentrations in the glass (>8–10 wt%) we also observed H$_2$O$_m$-loss with time, possibly caused by changes in the glass related to polishing, and also by diffusion effects during local heating by the laser. Leschik et al. (2004) have shown that H$_2$O is released under vacuum, at room temperature, from glasses containing >7 wt% H$_2$O. Determination of water content in unexposed glass inclusions would eliminate these sources of error. Such a technique also would make it possible to determine high water concentrations in MI (>20 wt%), which is at the present time impossible with FTIR, EMPA, or SIMS. A solution to this problem, outlined briefly in Thomas (2000), is described in detail in the present study. Being a non-destructive method, once H$_2$O analysis has been performed the inclusions are still available for further analysis. For example, the MI can be exposed to allow analysis of major and trace element compositions.

ANALYTICAL TECHNIQUE AND SAMPLE CHARACTERISTICS

Total water concentration (H$_2$O$_T$) was measured using a Raman probe consisting of a Dilor XY Laser Raman Triple 800 mm spectrometer (1800 G/mm gratings), equipped with an Olympus optical microscope, and a long working distance 80 × objective (required for imaging deep inclusions). The spectral resolution of the system is less than 1 cm$^{-1}$ in the high resolution mode. Spectra of the MI glasses were collected with a Peltier cooled CCD detector. The 488-nm line of a Coherent Ar$^+$ Laser Model Innova 70-3 at 450 mW was used for sample excitation (corresponding to 36 mW on sample). For all measurements a confocal pinhole of 150 µm was used (see Thomas 2002). All spectra were measured in the high-frequency range between 2800 and 3980 cm$^{-1}$ (Fig. 1). For simplicity we have adopted a linear background correction in the integration limits between 3100 and 3750 cm$^{-1}$ (see Fig. 1).

**FIGURE 1.** Unpolarized Raman spectra at the high frequency region taken from a water-rich MI-glass in pegmatite quartz from Ehrenfriedersdorf, and the reference albite glass (AB83) with a total water concentration H$_2$O$_T$ of 11.71 wt% for comparison.