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Matrix effects on secondary ion emissions for H and OH from natural spinel-structured oxides: Implications for H_2O quantification by secondary ion mass spectrometry

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Spinel-structured oxides (hereafter "spinels") are a group of nominally anhydrous minerals characterized by a wide range of solid solution. To allow the analysis of hydrogen in spinels by secondary ion mass spectrometry, Relative Sensitivity Factors (RSFs), which are typically matrix-dependent, need to be determined. For this work, we have selected 11 natural spinels from the Mg-Al-Fe-Ti-Cr-Mn-Zn compositional space, ranging in density from 3.7 to > 5.1 g cm^{-3}. This is the first attempt to determine ^{2}H and $^{16}O^{2}H$ RSF values in several crystals with different cation compositions. Samples were implanted with a fixed dose of deuterium (1×10^{15} atoms cm^{-2}) using an ion energy of 40 keV. Subsequently, we performed depth profiling using a Cs^{+} primary beam on a CAMECA IMS 1280-HR ion microprobe, monitoring secondary ions of $^{2}H^{-}$ and $^{16}O^{2}H^{-}$ until the implanted ion intensities dropped below background levels. From the reduced data, we calculated the RSFs for ^{2}H and $^{16}O^{2}H$ relative to ^{18}O as matrix element. RSF values vary with Al_2O_3 content. RSFs for ^{2}H yield 2.53 ± 0.99 (2SE) $\times 10^{22}$ atoms cm^{-3} at $Al_2O_3 < 2$ wt% and 1.49 ± 0.51 (2SE) $\times 10^{22}$ atoms cm^{-3} at $Al_2O_3 \geq 10$ wt%. RSFs for $^{2}H^{16}O$ relative to ^{18}O range from 2.59×10^{20} atoms cm^{-3} to 2.51×10^{21} atoms cm^{-3} and increase with increasing Al_2O_3 content. Preliminary RSF data for $^{2}H^{16}O$ relative to ^{18}O (Zellmer et al., 2025) closely matches the data presented here, and taken together, the RSF for $^{2}H^{16}O$ relative to ^{18}O follows a second order polynomial relationship with Al_2O_3 content: RSF = 1.516×10^{17} Al_2O_3 + 2.121×10^{19} Al_2O_3 + 2.758×10^{20}, where the RSF is in atoms cm^{-3} and Al_2O_3 is in wt%. Along this polynomial relationship, the relative standard error is about 23% (2SE). The overall lower RSF values of the dimer and the lower standard errors of the mean indicate that the analysis of hydrogen in spinels is best undertaken through the analysis of OH rather than H.



