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A novel method to measure isotopic labeled gas-phase nitrous acid (HO15NO) in biogeochemical studies

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We developed a new method (gas-phase stripping-derivatization coupled to LC-MS) to measure the 15N atom percent excess (APE) of HONO in the gas-phase. Gaseous HONO is quantitatively collected and transferred to an azo dye by the well-known Griess reaction in the Long Path Absorption Photometer (LOPAP). The reaction solutions containing the dye are collected at the outflow of the LOPAP, purified by solid-phase extraction and analyzed using high performance liquid chromatography coupled to mass spectrometry (HPLC-MS). The unlabeled azo dye ($C18H19O_2N5S$) with a monoisotopic molecular mass of 369.41 g mol-1 can be detected as its protonated molecular ion ([M+H+], M) by HPLC-MS at a retention time of 2.8 min. Due to the natural isotope distribution M + 0, M + 1, M + 2, and M + 3 ions were considered for the calculation of the 15N APE. The optimal working range was found to be between 20 and 50% for the 15N/14N ratio. The optimum pH and solvents for extraction by SPE and potential interferences are discussed. The method has been applied for the measurement of HO15NO emissions from soil in a dynamic chamber with and without spiking 15N labeled urea. Our results confirm biogenic HONO emissions from soil as HO15NO was measured after addition of 15N urea.