

New procedure to Pb, Zn and Cu separation and isotopic analysis on aerosol samples

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The multi isotopic approach has been used to characterize pollutant sources in environmental complex systems such as the atmosphere. Aerosol samples usually contain few micrograms per filter and manipulation increases the blank values, which is critical in these samples. A new procedure was developed in order to analyze Pb, Cu and Zn isotopes in a single aerosol membrane sample, which is digested with a mixture of HF/HNO₃ 2:1; the solution is then dried and redissolved in HCl 7M. This solution is loaded through a column with AG MP-1 resin and the Pb fraction is eluted together with the matrix, and it is further purified with AG1-X8 a resin. The Cu fraction is collected and submitted to a second purification using AG MP-1 resin¹⁻². Zn is the last one eluted in this procedure and does not need further purification. To evaluate separation process and recovery elution curves were constructed for an aerosol sample and BCR-2 reference material. The metal concentrations were determined in a ICP-MS (Thermo). Cu and Zn isotopic compositions were determined using a MC-ICP-MS Neptune (Thermo) and reference materials (BCR-1, BCR-2, BHVO-2 and AGV-1) were used to validate the analytical procedures. The Pb purification was previously validated. Pb, Cu and Zn showed recovery around 100%. The Zn and Cu isotopic compositions values were corrected by m-SSB and en-SSB, and errors were reported for 2σ. The BCR-1 values were δ⁶⁶Zn_{JMC} = +0.24±0.01 ‰ (n=3) and δ⁶⁵Cu_{NIST976} = +0.13±0.12 ‰ (n=2), while for BCR-2 showed δ⁶⁶Zn_{JMC} = +0.27±0.05 ‰ (n=10) and δ⁶⁵Cu_{NIST976} = +0.27±0.06 ‰ (n=2). The BHVO-2 presented δ⁶⁶Zn_{JMC} = +0.36±0.08 ‰ (n=6), and δ⁶⁵Cu_{NIST976} = +0.03±0.02 ‰ (n=2), and the AGV-1 showed δ⁶⁶Zn_{JMC} = +0.35±0.06 ‰ (n=4) and δ⁶⁵Cu_{NIST976} = -0.02±0.06 ‰ (n=2). The aerosol samples collected in São Paulo presented a δ⁶⁶Zn_{JMC} values ranging between -0.99 and +0.16 ‰. These results agree with the previous values published in the literature, and therefore validates the accuracy of the proposed analytical procedure.

[1] Babinski et al. (2003) *Journal Physics IV France* **107**, 87-90. [2] Dong et al. (2013) *Talanta* **114**, 103-9.