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Determination of chlorine isotope ratio with 0.01‰ accuracy

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The most common method used in the chlorine isotope ratio analysis employs the conversion of a chlorine sample to chloromethane (CH3Cl) prior to the mass spectrometric analysis.
For CH3Cl preparation the exchange reaction between the iodomethane (CH3I) and silver chloride (AgCl) is applied. We followed the procedure described by Eggenkamp [1] with the following modifications: (i) the aliquot of iodomethane is added to the preparation line by using the pipete connected to a container with pure liquid CH3I, thereby the injection of iodomethene throught a septum is eliminated, (ii) the conversion of AgCl to CH3Cl is performed in glass ampoules with Teflon stopcocks sealed with elastomer O-rings, thereby cracking tubes are eliminated, (iii) the obtained chloromethane is cryogenically separated from iodomethane using three traps (one with butyl acetate and two with trimethylpentane) connected in series.
The obtained chloromethane is then analysed by the isotope ratio mass spectrometry (IRMS). For this purpose we have devised a negative ion mass spectrometer which retains all the best features of IRMS, including dual inlet system with changeover valve, dual collector assembly and CH3Cl gas as analyte. In the modified ion source we have replaced the ionization chamber with electron beam by a metal tube with a hot metal filament inside. Within this tube the 35Cl- and 37Cl- ions are generated. No other ionic species were found in the mass spectrum except of traces of CN- and CO-. The method's precision is better than 0.01‰ (1σ) [2,3].

[1] H. Eggenkamp (2004) Ch. 28 in P. de Groot (ed.) Handbook of Stable Isotopes, Elsevier.
[2] A. Pelc, S. Hałas, (2008), Rapid Commun. Mass Spectrom., 22, 3977–3982.
[3] S. Hałas, A. Pelc (2009), Rapid Commun. Mass Spectrom. 23, 1061–1064.