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1 **Carbonate and silicate intercomparison materials for cosmogenic ^{36}Cl measurements**

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26

27 **Keywords**

- 28 1. Accelerator mass spectrometry
- 29 2. Terrestrial cosmogenic nuclides (TCN)
- 30 3. Round robin
- 31 4. Intercomparison material (ICM)
- 32 5. Consensus values

33

34 **Highlights**

- 35 1. Round-robin for ^{36}Cl and Cl_{nat} of calcite intercomparison material CoCal-N
- 36 2. First round-robin for ^{36}Cl of feldspar intercomparison material CoFsp-N
- 37 3. ^{36}Cl and Cl_{nat} concentrations indicate intra- and inter-laboratory consistency
- 38 4. Initial consensus values show coefficients of variation below 1.3%

39 **Abstract**

40 Two natural mineral separates, labeled CoCal-N and CoFsp-N, have been prepared to serve as
41 intercomparison material (ICM) for *in situ*-produced cosmogenic ^{36}Cl and natural chlorine (Cl_{nat})
42 analysis. The sample CoCal-N is derived from calcite crystals in a Namibian lag deposit, while the
43 sample CoFsp-N is derived from a single crystal of alkali-feldspar from a Namibian pegmatite. The
44 sample preparation took place at the University of Cologne and a rotating splitter was used to
45 obtain homogeneous splits of both ICMs. Forty-five measurements of CoCal-N (between 1 and 16
46 per facility) and forty-four measurements of CoFsp-N (between 2 and 20 per facility) have been
47 undertaken by ten target preparation laboratories measured by seven different AMS facilities. The
48 internal laboratory scatter of the ^{36}Cl concentrations indicate no overdispersion for half of the
49 laboratories and 3.9 to 7.3% (1σ) overdispersion for the others. We show that the CoCal-N and
50 CoFsp-N splits are homogeneous regarding their ^{36}Cl and Cl_{nat} concentrations. The grand average
51 (average calculated from the average of each laboratory) yields initial consensus ^{36}Cl concentrations
52 of $(3.74 \pm 0.10) \times 10^6$ at $^{36}\text{Cl}/\text{g}$ (CoCal-N) and $(2.93 \pm 0.07) \times 10^6$ at $^{36}\text{Cl}/\text{g}$ (CoFsp-N) at 95%
53 confidence intervals. The coefficient of variation is 5.1% and 4.2% for CoCal-N and CoFsp-N,
54 respectively. The Cl_{nat} concentration corresponds to the lower and intermediate range of typical
55 rock samples with $(0.73 \pm 0.18) \mu\text{g}/\text{g}$ in CoCal-N and $(73.9 \pm 6.8) \mu\text{g}/\text{g}$ in CoFsp-N. We discuss the
56 most relevant points of the sample preparation and measurement and the chlorine concentration
57 calculation to further approach inter-laboratory comparability. We propose to use continuous
58 measurements of the ICMs to provide a valuable quality control for future determination of ^{36}Cl
59 and Cl_{nat} concentrations.

60

61 **1 Introduction**

62 The number of studies using the cosmogenic nuclide ^{36}Cl has increased significantly during the last
63 two decades, and most of them are related to quantifications of Earth surface processes in non-
64 quartz-bearing lithologies. Since cosmogenic ^{36}Cl is produced and retained in Ca-, K-, Fe-, and Ti-
65 bearing minerals it can be applied for most carbonatic and basaltic rocks [1]. Applications of *in situ*-
66 produced ^{36}Cl cover a wide range of exposure dating applications allowing the age constraint of
67 depositional surfaces, of exhumation events (tectonic for example); and of volcanic eruptions [e. g.,
68 2-9]. Furthermore, ^{36}Cl denudation rate determinations allow insights into weathering rates and
69 sediment transport [10, 11].

70 Among other factors, the age constraints and calculated surface process rates depend on the
71 reliability of the ^{36}Cl analyses. Sample preparation and Accelerator Mass Spectrometry (AMS)
72 measurement techniques vary between facilities as they are adapted to their particular needs and
73 capabilities. It is desirable to have community-accepted and well-characterized materials that can
74 be prepared as targets and measured in the same way as samples of unknown cosmogenic nuclide
75 concentrations (“double-standardization”) [12]. This is pertinent when testing new target
76 preparation techniques or setting up new laboratories, but also to assure long-term measurement
77 accuracy for established laboratories. So far, inter-laboratory calibrations for ^{36}Cl on a larger scale
78 are limited to two studies. The first study used three silver chloride materials of different $^{36}\text{Cl}/\text{Cl}$
79 ratios ready to be pressed as targets, removing bias introduced during the sample preparation [13].

80 In the second study, three different laboratories prepared ^{36}Cl targets from seven whole-rock
81 samples of the Tabernacle Hill basalt [14]. One of the three laboratories reported 25-30% higher
82 ^{36}Cl concentrations, a difference that most likely arose during sample preparation [14, 15]. The
83 results of this study [14] highlight the need for readily available ICMs to identify and evaluate
84 differences between ^{36}Cl preparations/measurements at different laboratories.

85 Here, we present first results for carbonate and silicate materials that verify their homogeneity and
86 suitability for ^{36}Cl intercomparison studies. Ten target preparation laboratories and seven AMS
87 facilities participated in this evaluation exercise, yielding initial consensus concentrations for ^{36}Cl
88 and natural Cl (Cl_{nat}) for both the carbonate ICM (intercomparison material) 'CoCal-N' and the
89 silicate ICM 'CoFsp-N'.

90

91 **2 The ^{36}Cl intercomparison materials (ICMs)**

92 The samples used to prepare the ICMs were collected in the Namib Desert, ca. 8 km ESE from
93 Rössing mountain, Swakopmund district (Fig. 1a). About 20 kg of calcite (herein termed CoCal-N)
94 were collected as individual 5-15 cm tall crystals from a natural lag deposit in the vicinity of a
95 prospecting pit for Iceland spar (optical grade calcite) [16]. The crystals were weathered only
96 externally (Fig. 1b) and show optically clean interiors. The similar depth of the surface etching on
97 the crystals suggests a similar degree of weathering and comparable exposure duration.
98 Additionally, about 15 kg of feldspar (herein termed CoFsp-N) were collected as fragments from the
99 topmost 10 cm of a single large feldspar crystal from the surface outcrop of a pegmatite. The
100 pegmatite is physically weathered, but stands ~5 m above the gneisses of the surrounding areas
101 (Fig. 1c).

102 The preparation of the CoCal-N and CoFsp-N ICMs took place at the University of Cologne
103 (Germany). For both ICMs, all collected material was processed in one batch. Based on the different
104 type of materials the following pre-treatments were used.

105 Treatment of the calcite for the CoCal-N material:

- 106 1. Fracturing of the crystals into 2-3 cm-sized fragments to enable visual inspection of their
107 interior, removal of the impurities (rare dark inclusions, zones with fluid inclusions) with a die
108 grinder. Fragments with too widespread fluid inclusion zones for a complete removal
109 were discarded.
- 110 2. Etching of the remaining fragments in 10% HNO_3 to remove the weathered outer layer.
- 111 3. Crushing (Fritsch Disk Mill PULVERISETTE 13). Due to the fully transparent and colorless
112 interior of the calcite crystals and the use of a clean crusher, further etching was not
113 necessary.
- 114 4. Sieving with cleaned sieves to 250-500 μm .
- 115 5. Mixing and splitting using a clean rotating splitter (Fritsch Laborette 27 Rotary Cone Sample
116 Divider; division accuracy 99.9 %).

117 Half of the CoCal-N material (3.04 kg, separated from the whole using the rotating splitter) remains
118 as stock in Cologne; the other half was split into 32 vials with 95 g each for distribution.

119

120 Treatment of the feldspar for the CoFsp-N material:

- 121 1. Removal of the rare impurities (mostly mica) with a die grinder.
- 122 2. Crushing (Fritsch Disk Mill PULVERISETTE 13).
- 123 3. Sieving to 250-500 μm .
- 124 4. Etching in 1% HNO_3 /1% HF until 20% by weight dissolved.
- 125 5. Mixing and splitting using a clean rotating splitter (see above).

126 The entire stock of CoFsp-N material was split into 32 vials with 151 g each for distribution.

127 The chemical composition of the CoCal-N and the CoFsp-N material was determined at "Activation
128 Laboratories" (Canada) using four aliquots of 5 g from each of the ICM. Major and relevant trace
129 element concentrations for cosmogenic ^{36}Cl production are listed in [Table 1](#).

130 The calcite grains of CoCal-N are transparent rhombic fragments ([Fig. 1d](#)), whereas the shape of the
131 feldspar grains in CoFsp-N are irregular ([Fig. 1e](#)). The differential etching of the perthitic exsolution
132 lamellae of the feldspars (sodic feldspar lamellae in potassium feldspar) results in thin edges that
133 can easily break off. Consequently, the fine grained fraction of CoFsp-N has a different composition
134 than the bulk of the material. A rigorous homogenization is therefore important before taking
135 aliquots from the CoFsp-N stock, ideally utilizing a splitter. Using a spatula might yield an erroneous
136 result, which would not only bias an individual aliquot but also the remaining material. In addition,
137 any further etching of CoFsp-N renders comparison to the original material meaningless because
138 the potassium concentration, i. e. the concentration of the main target element, will no longer be
139 the same. For this reason, no additional rinsing or etching should be performed if the ICM is to be
140 used for intercomparison purposes. Considering the grain-size fractions and the expected range of
141 cosmogenic nuclide concentrations of individual grains, aliquots of 1 g of CoCal-N and 2 g of CoFsp-
142 N are considered to be homogenous ($< \pm 0.5\%$) with respect to their cosmogenic nuclide
143 concentration [17].

144

145 **3 Methods**

146 *3.1 ICM target preparation by participating labs*

147 The ICM vials with 95 g and 151 g of CoCal-N and CoFsp-N material, respectively, were distributed
148 to laboratories between 2011 and 2017. All preparation laboratories were informed that the
149 measurements are to be part of an inter-laboratory comparison. It was emphasized that the ICM
150 needs to be split appropriately and that it should not be rinsed or etched. It was left to each
151 laboratory how to treat the ICMs in detail, so that each laboratory used their own protocols to
152 process the carbonate and silicate materials to obtain the AgCl, required for AMS targets. The main
153 steps of the chemical preparation at the respective laboratories are listed in [Table 2](#) and references
154 are given for further details.

155 *3.2 AMS measurements*

156 The settings for the AMS measurements as well as the used standard and carrier chlorine isotope

157 ratios of the respective facilities are listed in Table 3.

158 3.3 Calculations of Cl_{nat} and ^{36}Cl concentrations

159 Each lab applies their own in-house procedure to calculate blank-corrected chlorine concentrations
160 from the AMS data, mostly using unpublished Excel spreadsheets. The calculation considers all
161 relevant weights, concentrations and ratios of the ICMs, the blanks and the carrier during the
162 preparation and AMS measurement. The calculation is adapted to the output of the respective AMS
163 facilities, e. g. it differs since some AMS facilities report $^{36}Cl/^{35}Cl$ ratios while others report ratios
164 $^{36}Cl/^{35+37}Cl$.

165

166 3.4 Statistical Methods

167 Forty-five aliquots of CoCal-N and forty-four aliquots of CoFsp-N were prepared and measured by
168 ten different laboratories and seven AMS facilities using their respective in-house methods.
169 Additionally, two large aliquots of CoCal-N (~24 g CoCal-N and 15 mg $^{35}Cl_{enriched\ carrier}$) were prepared
170 to obtain AgCl at the University of Cologne and split in 10 different targets just at the pressing
171 stage. These aliquots were measured at the AMS facilities ASTER (n=10) and CologneAMS (n=9).
172 This approach allows comparing the measurement performance at these two AMS facilities by
173 ruling out deviations due to preparation techniques. The results of each laboratory were tested for
174 outliers according to Dixon's criterion [37].

175 To quantify how well the sample statistics estimate the range of the likely ICM concentrations, we
176 calculated the standard deviation and the Coefficient of Variation

$$177 \quad CoV = \frac{1\sigma_{statistical\ uncertainty}}{X_{lab\ average}}$$

178 The statistical uncertainty of the weighted mean

$$179 \quad 1\sigma_{statistical\ uncertainty} = \sqrt{\frac{1}{\sum_{i=1}^N \frac{1}{\sigma_i^2}}}$$

180 [38] and the 95% confidence intervals of the ^{36}Cl and Cl_{nat} concentrations are assumed to be
181 significant for laboratories that measured at least three aliquots. The confidence intervals are used
182 to calculate the laboratory overdispersion, which describes the excess scatter (variance) that
183 cannot be explained by the analytical uncertainty alone. Therefore the following formula is
184 transformed and solved:

$$185 \quad 1\sigma_{confidence\ interval} = \sqrt{(1\sigma_{statistical\ uncertainty})^2 + (overdispersion)^2}.$$

186 For each laboratory with $n \geq 3$, the Mean Square of the Weighted Deviates (MSWD, a.k.a. "reduced
187 Chi-square", [39]) is reported based on

$$188 \quad MSWD = \frac{1}{N-1} \sum_{i=1}^N \frac{(x_i - \bar{x})^2}{\sigma_{x_i}^2}$$

189 A MSWD close to 1 indicates that the data dispersion reflects the analytical uncertainties. If the
190 MSWD is larger than 1, data are overdispersed, and if the MSWD is lower than 1, it is an indication

191 that the analytical uncertainties are probably overestimated [40].

192 Initial consensus values of the ^{36}Cl and Cl_{nat} concentrations are calculated using (i) the weighted
193 average of the single measurements, and (ii) the grand average (a weighted average of the
194 individual laboratory means, where the weights are the inverse of the variance of the mean [38]).
195 Furthermore, a linear regression fit between the amount of ICM dissolved and the total number of
196 ^{36}Cl and Cl_{nat} atoms measured allows an independent estimate of the precision of the data. Based
197 on the grand average concentrations, we calculated z-scores to evaluate possible trends of
198 individual laboratories

$$199 \quad z - score = X_{lab\ average} - \frac{X_{consensus\ value}}{1\sigma_{consensus\ value}}$$

200

201 **4 Results**

202 All measurement results and reported ^{36}Cl and Cl_{nat} concentrations are provided in [Table S1](#) and
203 [Figs. 2-4](#).

204 *4.1 ^{36}Cl concentrations*

205 CoCal-N results were reported for aliquots prepared at nine target preparation laboratories and
206 measurements at seven different AMS facilities ([Fig. 2a](#)). No intra-laboratory outliers were
207 observed among these 45 aliquots according to Dixon's method at the 95% confidence level. Two
208 laboratories performed only one measurement resulting in ^{36}Cl concentrations of
209 $(3.82 \pm 0.10) \times 10^6$ at/g (NMT/PRIME Lab) and $(4.404 \pm 0.094) \times 10^6$ at/g (DREAMS/DREAMS). For
210 the laboratories that measured at least two aliquots, the weighted mean average ranges from 3.46
211 to (3.98×10^6) at ^{36}Cl /g ([Table 4](#)). The standard deviations, coefficients of variation, statistical
212 uncertainties and 95% confidence intervals are given in [Fig. 2a](#) and [Table 4](#).

213 The result from DREAMS/DREAMS was not included in the calculation of the consensus value due
214 to the chemical sample pre-treatment, which likely changed the composition of the sample. In
215 comparison to the distribution of the individual measurements, the reported ^{36}Cl concentration of
216 DREAMS/DREAMS is an outlier according to Dixon's criteria at 90% confidence level, but not at 95%
217 confidence level. For the remaining CoCal-N measurements, the weighted average and 95%
218 confidence intervals of the individual ^{36}Cl concentrations yields $(3.72 \pm 0.07) \times 10^6$ at ^{36}Cl /g with
219 6.4% CoV and the grand average yields $(3.74 \pm 0.10) \times 10^6$ at ^{36}Cl /g with 5.1% CoV ([Table 4](#)).

220 Results of the CoFsp-N material were reported from seven different target preparation laboratories
221 (each used 2 to 20 aliquots) measuring at five different AMS facilities ([Fig. 2b](#); [Table S1](#)). The 44
222 aliquots indicate no intra-laboratory outliers according to the Dixon test at 95% confidence level.
223 The ^{36}Cl weighted mean averages of the individual laboratories range between 2.72 to 3.04×10^6
224 at ^{36}Cl /g ([Fig. 2b](#), [Table 4](#)). The results of all measurements lead to weighted averages and 95%
225 confidence intervals of the ^{36}Cl concentration of $(2.91 \pm 0.05) \times 10^6$ at ^{36}Cl /g with 5.3% CoV
226 considering all individual measurements, and $(2.93 \pm 0.07) \times 10^6$ at ^{36}Cl /g with 4.2% CoV
227 considering the grand average. Hence, for both ICMs the differently calculated averages agree
228 within uncertainties.

229 The ^{36}Cl concentrations of jointly prepared CoCal-N aliquots measured at ASTER and CologneAMS,
230 agree within their 1σ uncertainties, except of one outlier (Dixon test, 95% confidence level), which
231 had a significantly lower current during its measurement at CologneAMS (Fig. 3). The weighted
232 average of the 18 individual measurements and the grand average both lead to a ^{36}Cl concentration
233 of $(3.79 \pm 0.06) \times 10^6$ at $^{36}\text{Cl}/\text{g}$ (95% confidence intervals). The concentrations determined by this
234 test agree with the inter-laboratory averages obtained from the in-house preparation and
235 measurement procedures (Fig. 3).

236 The amount of dissolved ICM and the total ^{36}Cl content of the dissolved aliquots is, as anticipated,
237 linearly correlated (Fig. 4). This correlation allows another kind of measurement of the mean ^{36}Cl
238 concentrations leading to $(3.79 \pm 0.04) \times 10^6$ at $^{36}\text{Cl}/\text{g}$ for CoCal-N (correlation coefficient
239 $R^2 = 0.997$) and $(2.86 \pm 0.08) \times 10^6$ at $^{36}\text{Cl}/\text{g}$ for CoFsp-N ($R^2 = 0.992$, 2σ uncertainties). These values
240 are identical with the previously obtained two averages.

241 4.2 Initial ^{36}Cl consensus values

242 For both ICMs, the agreement in the concentrations calculated using three different approaches
243 highlights the reliability of the results. Since each lab has its own preparation method, AMS
244 measurement and calculation, we feel that it is most appropriate to treat the average result of each
245 lab as one value, and use the grand average of these values for an initial consensus. We obtain
246 ^{36}Cl concentrations of $(3.74 \pm 0.10) \times 10^6$ at $^{36}\text{Cl}/\text{g}$ (CoV = 5.1%) for CoCal-N, and
247 $(2.93 \pm 0.07) \times 10^6$ at $^{36}\text{Cl}/\text{g}$ (CoV = 4.2%) for CoFsp-N (95% confidence interval). The z-scores of
248 both ICM's range from -1.6 to +1.2, suggesting a good measurement performance for all
249 laboratories (Table 4).

250

251 4.3 Cl_{nat} concentrations derived by AMS

252 The measurement of the stable chlorine concentrations by AMS is done simultaneously to the ^{36}Cl
253 measurement on exactly the same target. This is done by isotope dilution, i. e. by addition of a Cl
254 carrier with unnatural $^{35}\text{Cl}/^{37}\text{Cl}$ ratios [1].

255 For CoCal-N, most measurements yield very low Cl_{nat} concentrations of $\sim 1 \mu\text{g}/\text{g}$ (Fig. 2c, Table S1),
256 with analytical uncertainties that are consistent with the scatter of the data (MSWD values). The
257 grand average of the CoCal-N aliquots prepared with in-house procedures is $(0.73 \pm 0.18) \mu\text{g}/\text{g} \text{Cl}_{\text{nat}}$
258 (95% confidence intervals). This Cl_{nat} concentration is in agreement with the other averaging
259 methods (Table 4, Fig. S1a) and with the Cl_{nat} concentrations obtained from the large CoCal-N
260 aliquots split with measurements at ASTER and Cologne AMS ($(0.55 \pm 0.45) \mu\text{g}/\text{g}$ and
261 $(0.72 \pm 0.71) \mu\text{g}/\text{g} \text{Cl}_{\text{nat}}$; Table S1). Z-scores of the laboratories range between -0.5 and +0.7,
262 indicating a good measurement performance despite the very low Cl_{nat} concentration (Table 4).

263 Measurements of CoFsp-N result in intermediate concentrations of Cl_{nat} with reported values
264 between 53 and 96 $\mu\text{g}/\text{g}$ (Fig. 2c). The range of reported analytical uncertainties is highly variable
265 between and within the laboratories. According to the MSWD they are partly overestimated, partly
266 underestimated and partly fitting (Table 4). The grand average for the Cl_{nat} concentration of CoFsp-
267 N leads to $(73.9 \pm 6.8) \mu\text{g}/\text{g}$ and agrees well with the weighted average of the individual
268 measurements ($(70.4 \pm 1.8) \mu\text{g}/\text{g}$) and the weighted least square regression between the amount of
269 igCoFsp-N dissolved and the Cl_{nat} content ($(76.9 \pm 5.4) \mu\text{g}/\text{g}$, $R^2 = 0.953$; Fig. S1b). Z-scores based on

270 the grand average range between -1.2 and +0.6, indicating a good measurement performance
271 (Table 4).

272

273 **5 Interpretation and Discussions**

274 *5.1 Homogeneity of the ICMs*

275 It is important to know that the material is homogeneous, otherwise its use as intercomparison
276 material would not be appropriate. The CoCal-N material has a simple composition as a pure calcite
277 and the similar shape and size of the grains make any fractionation with different ^{36}Cl
278 concentrations unlikely. This is in large contrast to the composition of the CoFsp-N material, whose
279 sodic feldspar laminae tend to split away easily, producing fine grained material of a different
280 composition compared to the coarse grained fraction. Hence, an appropriate splitting of the CoFsp-
281 N is essential and is best accomplished by placing the entire contents provided in the vial through a
282 rotating splitter.

283 The coefficient of variation of the ^{36}Cl concentrations (5.1% for CoCal-N and 4.2% for CoFsp-N for
284 the grand averages) is a first analytical indicator of the homogeneity of both ICMs. They are in a
285 reasonable range of the analytical capabilities and more precisely than the results of the previous
286 study that obtained a CoV of 6-8% from whole-rock basalt samples [15]. This indicates a good
287 reproducibility of the ^{36}Cl concentrations between the participating laboratories, which is only
288 possible for homogeneous samples.

289 For both ICMs, the MSWDs of the ^{36}Cl concentrations are distributed rather close to unity (Fig. 2a,b,
290 Table 4). The low MSWD for ^{36}Cl derived from UEdin/CologneAMS (0.2 for both samples, $n=7$ and
291 $n=9$) and from NMT/PRIME (MSWD=0.1 for CoFsp-N, $n=3$) are beyond the 95% confidence interval
292 of unity [41], indicating that the analytical uncertainties are overestimated. On the other hand,
293 some laboratories suggest a significantly high MSWD at the 95% confidence level, i. e.,
294 ANSTO/ANSTO and ULeeds/SUERC for CoCal-N, and CEREGE/ASTER and ITU/ASTER for CoFsp-N.
295 This might indicate that the scatter of ^{36}Cl concentrations is larger than expected based on the
296 given analytical uncertainties. However, since their MSWDs are based on only 4-5 measurements
297 and the ICMs were in some cases not appropriately split, this impression might change with further
298 measurements.

299 The best indicator of homogeneity is given by the very good correlation of the dissolved amount of
300 ICM versus the total ^{36}Cl -content in the dissolved ICM ($R^2 = 0.997$ for CoCal-N and $R^2 = 0.992$ for
301 CoFsp-N, Fig. 4). This correlation shows that preparing different ICM amounts results in the same
302 ^{36}Cl concentrations, which would not be expected in the case of inhomogeneous material. For both
303 ICMs all low-mass ^{36}Cl results lie within the 2σ regression range. From the 64 CoCal-N and 44 CoFsp-
304 N results, an expected amount of four aliquots occur outside the 2σ regression range (within 2σ ^{36}Cl
305 concentration uncertainties) and they are related to intermediate and large dissolved aliquots
306 (2×5 g CoFsp-N, 1×10 g CoCal-N, 1×15 g CoFsp-N, Fig. 4).

307

308 *5.2 Uncertainties of Cl_{nat} concentrations*

309 Even though the effect of Cl_{nat} on the ^{36}Cl concentration is minor, it should be accounted for
310 correctly to approach the best accuracy and precision of ^{36}Cl concentrations. While the obtained
311 Cl_{nat} concentrations indicate a well-defined value for both ICMs, their uncertainties are highly
312 variable regarding the individual laboratory measurements (Fig. 2c,d; Table 4). They depend on
313 several factors like (i) the estimation of uncertainties of the carrier's isotopic $^{35}Cl/^{37}Cl$ ratio, (ii) the
314 ratio of ICM to carrier amount (Fig. S2), (iii) the AMS performance regarding $^{35}Cl/^{37}Cl$ of the ICM and
315 the blank, and (v) the consideration of uncertainties during the calculation of the AMS ratios (e. g.,
316 blank correction).

317 So far, the AMS facilities of ETH and SUERC tested the precision and uncertainties of the Cl_{nat}
318 concentrations in rocks: SUERC highlights a clear correlation between the uncertainties of Cl_{nat} and
319 the concentration of Cl_{nat} , which corresponds to expected uncertainties in the range of >50% for
320 CoCal-N and 2-4 % for CoFsp-N [42]. This is in agreement with the reported high uncertainty of
321 ~90% for the CoCal-N aliquots obtained from ULeeds/SUERC. Measurements from ETH indicate that
322 their Cl_{nat} precision is below 1% for Cl_{nat} concentrations of >5 $\mu g/g$ Cl_{nat} [23], which is better than the
323 reported uncertainties of the other laboratories. Hence, it appears that the calculation of the Cl_{nat}
324 uncertainties is somewhat inconsistent for the different laboratories and could be modified in
325 future for an improved inter-laboratory comparability.

326

327 *5.3 Implications for ICM target preparation and measurement*

328 The ^{36}Cl and Cl_{nat} data from different laboratories agree and suggest that all laboratories produce
329 comparable results despite the differences in the target preparation techniques, AMS
330 configurations and concentration calculations. The CoCal-N aliquots that were split at the pressing
331 stage and measured at ASTER and CologneAMS revealed ^{36}Cl concentrations which agree within
332 uncertainties (Fig. 3), highlighting the identical performance of both AMS facilities. In terms of the
333 chemical sample preparation steps, no clear trends can be observed between method differences
334 and resulting ^{36}Cl concentrations. This includes the implication that degassing of ^{36}Cl during the
335 CoCal-N dissolution is negligible. For instance, laboratories that performed a slower addition of
336 cooled 2 M HNO_3 yielded intermediate ^{36}Cl concentrations, while the aliquots that were dissolved
337 at room temperature (ASTER and ANSTO) show both the highest and lowest tendencies of ^{36}Cl
338 concentrations. Furthermore, testing the addition of $AgNO_3$ before the sample dissolution on two
339 CoCal-N aliquots at UoC/CologneAMS indicated no difference to the aliquots where $AgNO_3$ was
340 added after the dissolution (Table S1).

341 The linear relationship between the amount of dissolved ICM and the total ^{36}Cl atoms (Fig. 4)
342 indicates that small aliquots down to ~1 g reveal representative concentrations for both ICMs. This
343 agrees with the calculation of a <0.5% deviation of the cosmogenic nuclide concentration in 1 g of
344 CoCal-N and 2 g of CoFsp-N material. Slightly lower amounts can be used since the precision of the
345 measured ^{36}Cl concentrations is >2% even in optimal circumstances, thus it is sufficient to use 1 –
346 3 g of ICM per aliquot. After an adequate homogenization and splitting, the use of low sample
347 amounts is desirable since it will extend the life of the ICMs as long as possible. If feasible, the
348 amount of sample and carrier could be adjusted to result in similar $^{36}Cl/^{35}Cl$ ratio as the expected
349 $^{36}Cl/^{35}Cl$ ratio of the unknown samples to enlarge the degree of analytical reproducibility [12]. In
350 this study, a relation of dissolved ICM weight to carrier weight of 0.5 - 11 g/mg resulted in

351 successfully measured $^{36}\text{Cl}/^{35}\text{Cl}$ AMS ratios in the range of 8.5×10^{-14} to 5.2×10^{-12} (Table S1). Since
352 the adaptations regarding the sample size and carrier amount are limited, further ICMs of different
353 ^{36}Cl and Cl_{nat} concentrations are required to extend the inter-laboratory comparability
354 measurements to the range of typically measured samples.

355

356 6 Recommendations and Conclusions

357 Initial ^{36}Cl results of CoCal-N and CoFsp-N show that both ICMs are suitable as in-house quality
358 assurance material and for inter-laboratory comparisons, provided they are split appropriately
359 (ideally utilizing a splitter). The initial consensus values are $(3.74 \pm 0.10) \times 10^9$ at/g (95% confidence
360 interval) with an inter-laboratory 1σ -overdispersion of 1.3% for CoCal-N, and $(2.93 \pm 0.07) \times 10^9$ at/g
361 (95% confidence interval) with an inter-laboratory 1σ -overdispersion of 1.1% for CoFsp-N. As
362 suggested by Phillips et al. [12], we recommend routine measurements of the ICMs along with
363 unknown samples for quality assurance. This will allow an appreciation of realistic inter-laboratory
364 uncertainties for in-situ produced cosmogenic nuclides, instead of internal uncertainties only. We
365 recommend the use of 1-3 g of ICM per aliquot, while the preparation of aliquots - particularly in
366 the case of CoFsp-N - must be performed by appropriate splitting of the stock. At present, the
367 remaining stock of CoCal-N and CoFsp-N in Cologne is 3.9 kg and 2.1 kg, respectively. Those
368 interested in obtaining CoCal-N or CoFsp-N may contact T. Dunai (tdunai@uni-koeln.de).

369

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376 comments of two anonymous reviewers.

377

378 Supplementary Information

379 **Figure S1.** Correlation between the amount of dissolved ICM and the content of Cl_{nat} (1σ
380 uncertainties). The slope of the weighted least square linear regression represents the Cl_{nat}
381 concentrations of the ICMs. The color of the symbols refers to the respective laboratories (for the
382 legend see Fig. S2). For CoCal-N the measurement at DREAMS/DREAMS and the outlier of
383 UoC/CologneAMS were excluded from the regression. The given values and the gray envelopes of
384 the regression line correspond to 2σ uncertainties. The y-axis intercept is around zero, which is an
385 important criterion of data quality since the intercept represents the extrapolated amount of atoms
386 in a hypothetical zero gram aliquot.

387 **Figure S2.** Correlation of the ICM to carrier amount versus the uncertainty of the Cl_{nat}
388 concentration. This highlights the different methods of the uncertainty calculation at each
389 laboratory.

390 **Table S1.** Preparation and measurement details of all CoCal-N and CoFsp-N aliquots.

391

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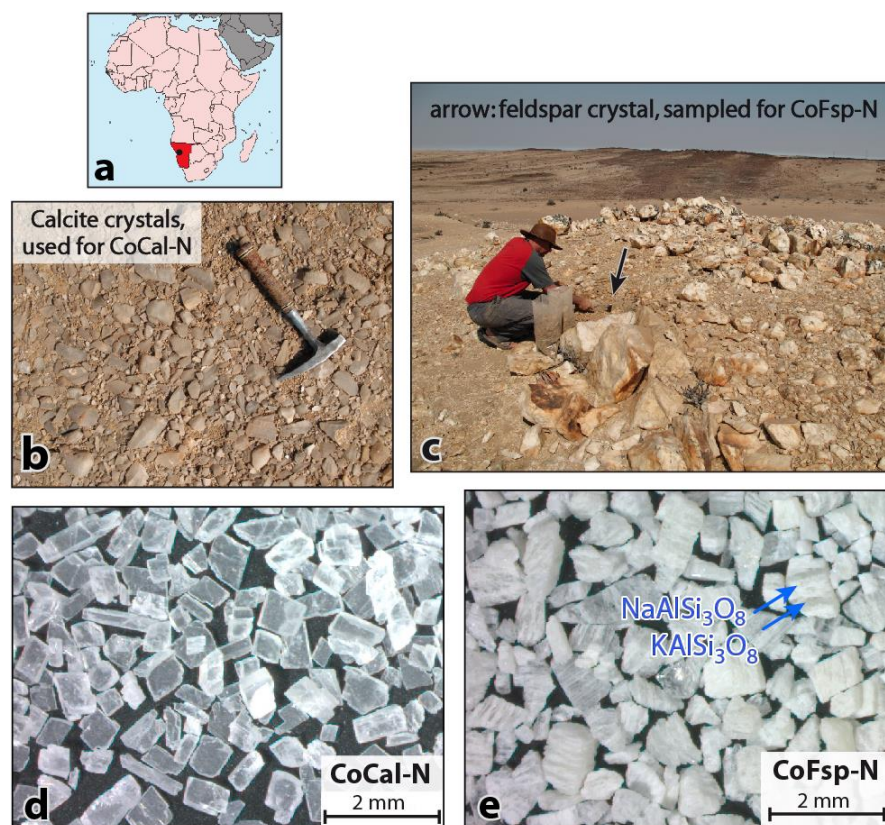
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479 **Figures and Tables**

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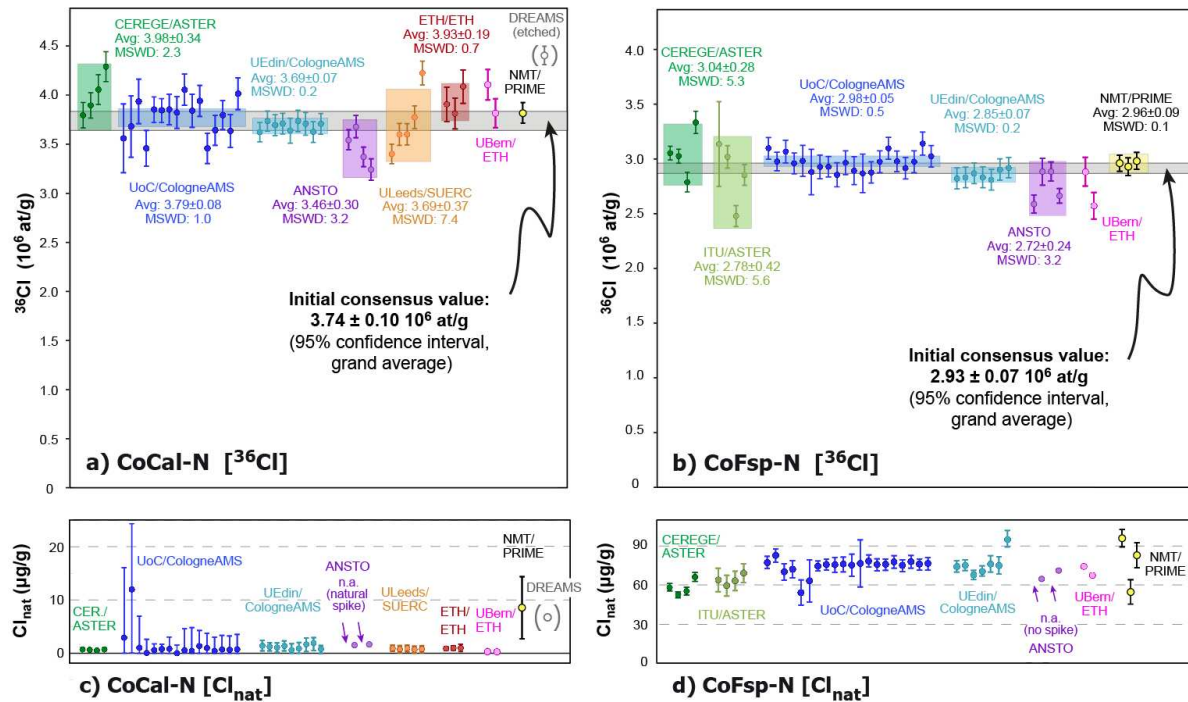


482

483 **Fig. 1:** (a) Sampling location in the Namib Desert. (b) A part of the calcite lag deposit sampled for CoCal-N. (c)
484 View of the landscape in the background and pegmatite in the foreground with the marked location of the
485 feldspar crystal sampled for CoFsp-N. (d) Light microscope view of the prepared sample CoCal-N, showing
486 rhombic cleavage fragments of calcite. (e) Light microscope view of the prepared sample CoFsp-N, showing
487 the texture of the etched material. Perthitic exolutions (sodic feldspar lamellae in potassium feldspar,
488 illustrated by arrows on the image) are visible in most grains.

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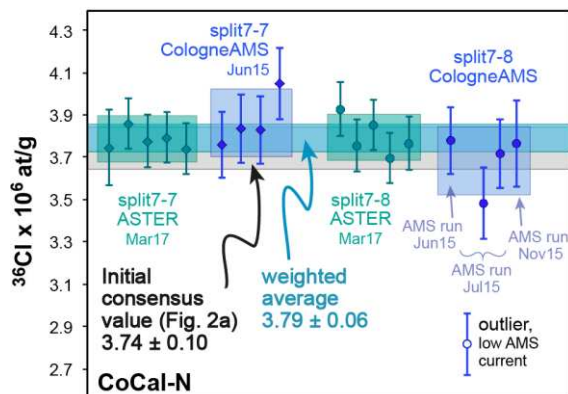
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492 **Fig. 2:** Reported chlorine concentrations (1σ uncertainties). The codes of the sample preparation laboratories
 493 and AMS measurement facilities are indicated. **(a)** ^{36}Cl concentrations for CoCal-N. **(b)** ^{36}Cl concentrations for
 494 CoFsp-N. Colored rectangles in (a,b) mark the weighted averages with their 95% confidence intervals. Mean
 495 Squares of Weighted Deviates (MSWD) are provided for laboratories with at least three measurements. **(c)**
 496 Natural chlorine (Cl_{nat}) concentrations of each CoCal-N measurement. **(d)** Cl_{nat} concentrations of each CoFsp-N
 497 measurement.

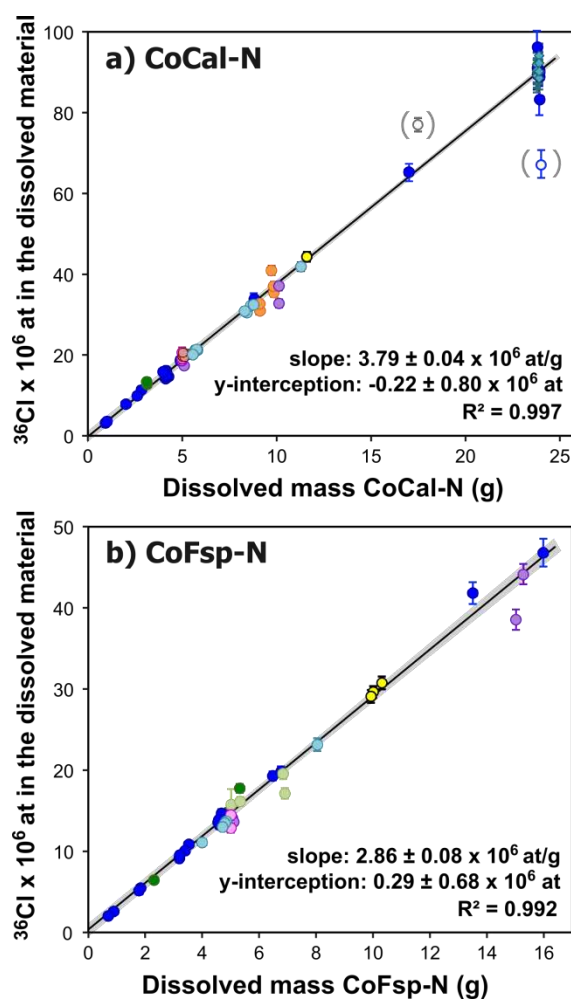
498



499 **Fig. 3:** ^{36}Cl concentrations of the two large CoCal-N aliquots, which were prepared at the University of
500 Cologne (UoC) and split in 10 different targets just at the pressing stage. The measurement results of the AMS
501 runs at ASTER and CologneAMS are shown by green and blue signatures, respectively. The month and year of
502 the measurement is indicated. Surrounding boxes highlight the weighted averages of the respective aliquots
503 (excluding the outlier), and the average resulting from the aliquots shown in Fig. 2a.

504

505



506

507 **Fig. 4:** Correlation between the amount of dissolved ICM and the ^{36}Cl content of the aliquots (1σ data
508 uncertainties). The slope of the weighted least square linear regression represents the ^{36}Cl concentrations of
509 the ICMs. The color of the symbols refers to the respective laboratories with the same coding as in Figs. 2 and
510 3. All measured aliquots shown in Figs. 2 and 3 are included. The DREAMS/DREAMS measurement and the
511 outlier of UoC/CologneAMS (both in brackets) were excluded from the regression. The given values and the
512 gray envelopes of the regression line correspond to 2σ uncertainties.

513 **Table 1:** Relevant chemical composition, based on 4 aliquots (5 g each) measured at Activation Laboratories
 514 (Canada) and by AMS (accelerator mass spectrometry) measurements in this study. Sample uncertainties
 515 represent the absolute standard deviation of the means of the four aliquots. FUS-ICP: fusion inductively
 516 coupled plasma. LOI: loss on ignition.

Element	CoCal-N	CoFsp-N	Element	CoCal-N	CoFsp-N
<i>FUS-ICP AES (atomic emission spectrometry)</i>			<i>FUS-ICP MS (mass spectrometry)</i>		
SiO ₂	0.10 ± 0.10 %	65.20 ± 0.69 %	Rb	< 2 µg/g	568 ± 17 µg/g
Al ₂ O ₃	0.03 ± 0.01 %	18.68 ± 0.24 %	Sm	< 0.1 µg/g	< 0.1 µg/g
Fe ₂ O ₃	0.01 ± 0.01 %	0.03 ± 0.01 %	Gd	< 0.1 µg/g	< 0.1 µg/g
MgO	0.11 ± 0.01 %	0.04 ± 0.03 %	Th	< 0.1 µg/g	< 0.1 µg/g
CaO	56.43 ± 0.78 %	0.14 ± 0.06 %	U	< 0.1 µg/g	< 0.1 µg/g
Na ₂ O	< 0.01 %	3.14 ± 0.08 %	<i>FUS-ICP AES (atomic emission spectrometry)</i>		
K ₂ O	0.01 ± 0.01 %	12.03 ± 0.28 %	Ba	3 ± 2 µg/g	122 ± 3 µg/g
TiO ₂	0.001 ± 0.001 %	0.004 ± 0.00 %	<i>TD-ICP (Total digestion inductively coupled plasma)</i>		
LOI	42.20 ± 0.54 %	0.35 ± 0.10 %	Li	< 1 µg/g	2 ± 0.1 µg/g
Total	98.89 ± 0.28 %	100.01 ± 0.79 %	<i>PGNAA (Prompt gamma neutron activation analysis)</i>		
<i>Gravimetric</i>			B	1.6 ± 1.2 µg/g	6.7 ± 2.6 µg/g
H ₂ O	< 0.1 %	< 0.1 %	<i>AMS (Accelerator Mass Spectrometry, Table S1)</i>		
			Cl	0.73 ± 0.18 µg/g	73.9 ± 6.8 µg/g

517 **Table 2:** Preparation procedures of the samples in the respective laboratories.

Laboratory	preparation step	Aix-Marseille University (CEREGE)	Istanbul Technical University (ITU)	University of Cologne (UoC)	University of Edinburgh (UEdin)	ANSTO	University of Leeds (ULeeds)	ETH Zurich (ETH)	University of Bern (UB)	New Mexico Tech (NMT)	DREAMS
pre-treatment	sample splitting method, and chemical treatment if applied	no homogenization; except CoFsp4: shake and scoop	CoFsp1+2: no homogenization; CoFsp 3+4: rotating splitter	rotating splitter	rotating splitter	shake & scoop	rotating splitter	shake & scoop	shake & scoop	coned & quartered	shake & scoop; 2xH ₂ O shaker-table, 1x10%-dissolution in HNO ₃
Carrier	enriched material and laboratory preparation	Enriched ³⁵ Cl (C-Chem LTD, Israel), dissolved with MilliQ to ~1.5 mg _{Cl} /g _{sol} ^{a)}	Enriched ³⁵ Cl (Aldrich Chem Co.), dissolved with MilliQ to ~1.5 mg _{Cl} /g _{sol} ^{b)}	99.9% at ³⁵ Cl (ORNL, batch 150301); mixed with Fisher NaCl (natural ratio) to 20.1 ³⁵ Cl/ ³⁷ Cl, dissolved with MilliQ to (6.56±0.066) mg _{Cl} /g _{sol}	99.9% at ³⁵ Cl (ICON Isotopes, #IK 7425, Lot EY79), mixed with Fisher NaCl (natural ratio) to 19.96 ³⁵ Cl/ ³⁷ Cl, diluted with MilliQ to 5.457 mg _{Cl} /g _{sol}	Carrier1: natural ³⁵ Cl/ ³⁷ Cl ratio. Carrier2: 98.00% at ³⁷ Cl (Oak Ridge, batch 198590), dissolved with MilliQ to ~1.5 mg _{Cl} /g _{sol}	99.635% at ³⁵ Cl (ICON Isotopes), dissolved with natural NaCl to (19.9 ³⁵ Cl/ ³⁷ Cl), diluted with MilliQ to 6.2 mg _{Cl} /g _{sol}	99.65% at ³⁵ Cl (ICON Isotopes), dissolved with MilliQ to 5-6 mg _{Cl} /g _{sol}	99.65% at ³⁵ Cl (ICON Isotopes), dissolved with MilliQ to 5-6 mg _{Cl} /g _{sol}	99.9% at ³⁵ Cl (ICON Isotopes, #IK 7425, Lot EY79), mixed with Fisher NaCl (natural ratio) to 19.96 ³⁵ Cl/ ³⁷ Cl, diluted with MilliQ to 5.457 mg _{Cl} /g _{sol}	99.9% at ³⁵ Cl (Sigma Aldrich, certificate 04/06/2009), dissolved with MilliQ to (1.4981 ±0.0075) mg _{Cl} /g _{sol}
Carbonates	AgNO ₃ addition	after dissolution	--	16 aliquots after dissol. & 2 aliq. before dissol.	after dissolution	after dissolution	after dissolution	after dissolution	after dissolution	after dissolution	after dissolution
	HNO ₃ addition	2M HNO ₃ added in ~10ml steps at room temp.	--	2M HNO ₃ of 5-20°C added in 10ml steps at room temp.	2M HNO ₃ added in 5/10/20ml steps in ice bath	2M HNO ₃ added at room temp.	2M HNO ₃ added in 10/20ml steps in ice bath	2M HNO ₃ added in 10ml steps at room temp.	2M HNO ₃ added in 10ml steps at room temp.	~2M HNO ₃ added all at once at room temp. to sealed container, 50°C hot plate	2M HNO ₃ added in ice bath in 2.5ml steps
Silicates	AgNO ₃ addition	after dissolution	after dissolution	after dissolution	after dissolution	before dissolution	--	--	after dissolution	after dissolution	--
	HNO ₃ /HF addition	2M HNO ₃ & conc HF in ice bath, ≥ 24h on shaker table at room temp.	conc HF, 6 hours at 130°C in acid digestion vessels	2M HNO ₃ & conc HF, several days on shaker table at room temp.	2M HNO ₃ & conc HF, several days on shaker table at room temp.	2M HNO ₃ & conc HF, room temp. 24h, shaker table for 8h at 50°C, room temp. for weekend	--	--	conc HNO ₃ & conc HF, room temp. overnight, then heat 100°C	conc HNO ₃ & conc HF, several days on hot plate at 50°.	--

AgCl purification of all samples	removal of undissolved material	carbonates: filtration silicates: centrifuging	centrifuging	centrifuging	centrifuging	centrifuging	centrifuging	centrifuging	centrifuging	centrifuging	filtration
	sulfur removal	1xBa(NO ₃) ₂ overnight, centrifuging, 0.45μm filtered	2xBa(NO ₃) ₂ >24 hours, centrifuging	1xBa(NO ₃) ₂ >48 hours, centrifuging, 0.1μm filtered	1xBa(NO ₃) ₂ , >12 days, 0.2μm filtered	1xBa(NO ₃) ₂ , >48 hours, 0.22μm filtered	1xBa(NO ₃) ₂ , >48 hours, 0.2μm filtered	1xBa(NO ₃) ₂ >48 hours, centrifuging, 0.45μm filtered	1xBa(NO ₃) ₂ >48 hours, centrifuging, 0.45μm filtered	1xBa(NO ₃) ₂ , >7 days, 0.45μm filtered	1xBa(NO ₃) ₂ , overnight, 0.45μm filtration
References	--	[18, 19]	[20]	[21]	[22]	--	[6]	[23]	[23]	[24] ^{c)}	[25]

^{a)} Two different batches of carrier were used at CEREGE: CoFsp-N-1 to CoFsp-N-3: 99.88 % at ³⁵Cl diluted to 5.91 mgCl/g_{sol}, CoFsp-N-4 and all CoCal-N aliquots: 99.89 ³⁵Cl diluted to 6.92 mgCl/g_{sol}.

^{b)} Three different batches of carrier were used at ITU: CoFsp-N-1 and CoFsp-N-2: 99.847 ³⁵Cl/at diluted to 1.690 mgCl/g_{sol}, CoFsp-N-3: 99.652 ³⁵Cl/at diluted to 1.537 mgCl/g_{sol}, CoFsp-N-4: 99.850 ³⁵Cl/at diluted to 1.405 mgCl/g_{sol}.

^{c)} The preparation procedure has been modified to the use of a lower ³⁵Cl/³⁷Cl carrier ratio (see [Tables 3](#) and [S1](#)) and plastic/disposable test tubes. The carbonate processing procedure varied from that of Marrero (2012) in that the acid was dripped in slowly at room temperature rather than rapidly.

518 **Table 3:** Measurement conditions at the participating AMS facilities.

a)	Laboratory							Comment
	ASTER	CologneAMS	DREAMS	ANSTO	SUERC	PRIME Lab	ETH	
Accelerator	Tandetron			Pelletron		Pelletron-converted FN	Pelletron-converted EN	Mechanical (Pelletron) or electronic (Tandetron) accelerator high-voltage power supply
Stripping	Gas					Foil		Constant & high beam-brightness gas stripping or high charge-state (& ion energy) foil stripping
Ion energy	30 MeV	35 MeV			30 MeV	59.2 MeV	46.4 MeV	
Sulphur suppression technique	Foil			Detector			Detector & gas-filled magnet	Detector ³⁶ S suppression is time & sample efficient, whereas post-accelerator foil suppression is more ³⁶ S tolerant/suppressing facilitating different cathodes
Cathode	Ni	Ni/Cu	Cu with steel pin	Cu with AgBr insert			Cu with Ta inlet	
Primary ³⁶ Cl/ ³⁵⁺³⁷ Cl standard ^{b)}	KN (1.60 ± 0.02) × 10 ⁻¹²		SM-Cl-12 (1.082 ± 0.016) × 10 ⁻¹²	PRIME Lab Z93-0005 (1.2) × 10 ⁻¹²		KN (1.6 ± 0.02) × 10 ⁻¹²	KN (5.0 ± 0.1) × 10 ⁻¹³	Sample measurements are validated by secondary standards measurements of the same quality, all calibrated to primary standard analysis. The measured standard deviation of the secondary standards accounts for multiple AMS runs with multiple standard analysis. Standard uncertainties can be included or not included during the calculation of the Cl concentrations.
Secondary ³⁶ Cl/ ³⁵⁺³⁷ Cl standards ^{b)} & their measured standard deviation	SM-Cl-12 (1.082 ± 0.016) × 10 ⁻¹²	KN (5.0 ± 0.1) × 10 ⁻¹³	-	KN (1.6 ± 0.02) × 10 ⁻¹² KN (5.0 ± 0.1) × 10 ⁻¹³		KN (5.0 ± 0.1) × 10 ⁻¹³	ETH K382/4N (17.36 ± 0.35) × 10 ⁻¹²	
	1.5%, included	2%, included	std uncert. included	3%, included	3%, included	2%, included	2%, not included	
Carrier & stable Cl measurement	³⁵ Cl/ ³⁷ Cl 287-918; simultaneous	³⁵ Cl/ ³⁷ Cl 20.1; simultaneous	³⁵ Cl/ ³⁷ Cl 999; simultaneous	³⁵ Cl/ ³⁷ Cl 3.127 and 0.49; simultaneous	³⁵ Cl/ ³⁷ Cl 19.9; simultaneous	³⁵ Cl/ ³⁷ Cl 6.1; sequential	³⁵ Cl/ ³⁷ Cl 283; simultaneous	Addition of a chlorine carrier with unnatural ³⁵ Cl/ ³⁷ Cl ratio allows isotope dilution and increase of AgCl target size. Stable isotope analysis are done either simultaneously with ³⁶ Cl measurement, or sequentially on the same cathode after ³⁶ Cl/ ³⁵ Cl measurement.
Reference	[26, 27]	[28]	[29, 30]	[31]	[32, 33]	[34]	[35, 36]	

a) ASTER = HVE 5 MV Accélérateur pour les Sciences de la Terre, Environnement, Risques (ASTER), CEREGE, Aix-Marseille Université, Aix-en-Provence, France; CologneAMS = 6 MV Tandatron Accelerator, University of Cologne, Cologne, Germany; DREAMS = 6 MV Tandatron Accelerator, DREsdEN AMS, Helmholtz-Zentrum Dresden-Rossendorf (HZDR), Dresden, Germany; ANSTO = 6 MV SIRIUS Tandem Accelerator, Australian Nuclear Science and Technology Organization (ANSTO), Sydney, Australia; SUERC = 5 MV NEC Accelerator, Scottish Universities Environmental Research Centre (SUERC), East Kilbride, UK; PRIME Lab = 8 MV Tandem Accelerator, Purdue Rare Isotope Measurement Laboratory (PRIME Lab), Purdue University, IN 47906, USA; ETH = 6 MV HVEC EN-Tandem Accelerator, Laboratory of Ion Beam Physics, ETH Zurich, Zurich, Switzerland.

b) All standards have the natural $^{35}\text{Cl}/^{37}\text{Cl}$ ratio of 3.129. All $^{35}\text{Cl}/^{37}\text{Cl}$ ratios of the samples are normalized to the primary standard.

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Table 4: Statistical analysis of the obtained ^{36}Cl and Cl_{nat} concentrations. A full statistical interpretation is only obtained if at least 3 measurements were available. The data of the individual aliquot measurements are given in [Table S1](#).

Laboratory/AMS name	CEREGE/ ASTER	ITU/ ASTER	UoC/ Cologne AMS	UEdin/ Cologne AMS	ANSTO/ ANSTO	ULeeds/ SUERC	ETH/ ETH	UBern/ ETH	NMT/ PRIME Lab	DREAMS/ DREAMS	weighted average of all aliquots ^{c)}	grand average ^{d)}
CoCal-N: [^{36}Cl]												
number of aliquots ^{a)}	4	-	16	9	4 ^{e)}	5	3	2	1	1	40	6
weighted mean (10^6 at/g)	3.98	-	3.79	3.69	3.46	3.69	3.93	3.96	3.82	4.40	3.72	3.74
1 σ standard deviation (10^6 at/g)	0.22	-	0.18	0.05	0.19	0.31	0.14	0.21	-	-	0.24	0.19
1σ coefficient of variation (%)	5.4%	-	4.9%	1.2%	5.6%	8.4%	3.5%	5.2%	-	-	6.4%	5.1%
1 σ statistical uncertainty of wt. mean (10^6 at/g)	0.07	-	0.04	0.04	0.05	0.05	0.10	0.11	0.104 ^{f)}	0.094 ^{f)}	0.02	0.03
95% confidence interval (10^6 at/g) ^{b)}	0.34	-	0.08	0.07	0.30	0.37	0.19	-	-	-	0.07	0.10
MSWD (-)	2.3	-	1.0	0.2	3.2	7.4	0.7	-	-	-	2.8	2.6
1 σ overdispersion (%)	3.9%	-	<0.1%	<0.1%	4.1%	4.8%	<0.1%	-	-	-	0.7%	1.2%
z-score (-)	1.2	-	0.2	-0.3	-1.4	-0.2	1.0	-	-	-	-0.1	-
CoCal-N: [Cl_{nat}]												
weighted mean ($\mu\text{g/g}$)	0.54	-	0.54	0.98	1.65	0.72	0.86	0.18	8.4	6.6	0.79	0.73
1 σ standard deviation ($\mu\text{g/g}$)	0.11	-	0.66	0.43	0.98	0.09	0.05	0.02	-	-	1.57	0.19
1 σ coefficient of variation (%)	21%	-	122%	44%	60%	13%	6%	11%	-	-	199%	27%
1 σ statistical uncertainty of wt. mean ($\mu\text{g/g}$)	0.06	-	0.21	0.29	0.15	0.31	0.05	0.01	6.6 ^{f)}	0.5 ^{f)}	0.04	0.04
95% confidence interval ($\mu\text{g/g}$) ^{b)}	0.12	-	0.40	0.56	-	0.61	0.10	-	-	-	0.09	0.18
MSWD (-)	1.12	-	0.1	0.2	-	0.02	0.4	-	-	-	1.6	3.8
1 σ overdispersion (%)	<0.1%	-	<0.1%	<0.1%	-	<0.1%	<0.1%	-	-	-	4%	11%
z-score (-)	-0.5	-	-0.5	0.7	-	0.0	0.4	-	-	-	0.2	-
CoFsp-N: [^{36}Cl]												
number of aliquots	4	4	20	7	4	-	-	2	3	-	44	6
weighted mean (10^6 at/g)	3.04	2.78	2.96	2.85	2.72	-	-	2.72	2.96	-	2.91	2.93
1 σ standard deviation (10^6 at/g)	0.22	0.29	0.08	0.04	0.16	-	-	0.22	0.03	-	0.15	0.12
1σ coefficient of variation (%)	7.3%	10%	2.8%	1.5%	6.1%	-	-	8.1%	0.9%	-	5.3%	4.2%
1 σ statistical uncertainty of wt. mean (10^6 at/g)	0.04	0.06	0.03	0.04	0.04	-	-	-	0.04	-	0.01	0.02
95% confidence interval (10^6 at/g) ^{b)}	0.28	0.42	0.05	0.07	0.24	-	-	-	0.09	-	0.05	0.07

MSWD (-)	5.3	5.6	0.5	0.2	3.2	-	-	-	0.1	-	2.6	2.6
1 σ overdispersion (%)	4.4%	7.3%	<0.1%	<0.1%	4.1%	-	-	-	<0.1%	-	0.6%	1.0%
z-score (-)	0.8	-1.1	0.2	-0.6	-1.6	-	-	-	0.2	-	-0.2	-
CoFsp-N: [CI_{nat}]												
weighted mean (CI _{nat} $\mu\text{g/g}$)	57.2	64.2	75.5	74.3	70.6	-	-	70.5	82.2	-	70.4	73.9
1 σ standard deviation ($\mu\text{g/g}$)	5.9	4.0	6.1	8.9	3.1	-	-	4.9	21	-	9.5	9.91
1 σ coefficient of variation (%)	10%	6.2%	8.0%	12%	4.4%	-	-	7.0%	26%	-	13%	13%
1 σ statistical uncertainty of wt. mean ($\mu\text{g/g}$)	1.5	3.9	1.2	1.75	0.19	-	-	1.0	4.8	-	0.8	4.1
95% confidence interval ($\mu\text{g/g}$) ^{b)}	8.9	7.7	2.3	6.8	-	-	-	-	52	-	1.8	6.8
MSWD (-)	3.5	0.3	0.5	2.5	-	-	-	-	6.5	-	5.1	1.5
1 σ overdispersion (%)	7.3%	<0.1%	<0.1%	3.9%	-	-	-	-	31%	-	0.7%	<0.1%
z-score (-)	-1.2	-0.7	0.1	0.0	-	-	-	-	0.6	-	-0.3	-

- ^{a)} The identically prepared targets that were measured at ASTER and CologneAMS not included.
- ^{b)} 95% confidence interval includes statistical uncertainty and overdispersion.
- ^{c)} The measurement at DREAMS/DREAMS is excluded from the statistics since the sample was leached.
- ^{d)} Only labs with ≥ 3 aliquot measurements are used to calculate the grand average
- ^{e)} ANSTO/ANSTO used a natural spike for half of their samples and hence CI_{nat} concentrations results are limited to 2 aliquots per ICM.
- ^{f)} Analytical uncertainty since only one aliquot was measured.