Experiments on method for washing drill cuttings: evaluation of soaking, stirring, and milling effects

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Effects of soaking, stirring, and milling were investigated to standardize the washing method used for drill cutting samples. Drill cuttings are fragments of solid material ground by a drill bit at the bottom of a hole. They are recovered on the ship in riser drilling as a component of the circulating drilling mud. Removal of drilling mud from the surface of cutting samples must be done before cutting samples are analyzed. Cutting samples from IODP Expedition 337 were used in test experiments. The samples were analyzed before and after soaking, stirring, and milling with XRD, XRF, an elemental analyzer and a carbonate analyzer. Results show that Na and K contents are reduced by the removal of drilling mud. However, changes that occur after 6 h of soaking are rather minor. These results suggest that stirring of cuttings in heated seawater at 40°C for 6 h after quick washing is effective for XRF and TOC analyses.

Keywords: drill cuttings, ocean drilling, contamination, drilling mud

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1. Introduction

Drill cuttings are fragments of solid material that are produced when a drill bit penetrates into a formation at the bottom of a hole. In riser drilling, cuttings are removed from the borehole, after which they are transported to the surface together with drilling mud. Cutting samples are useful for lithology description, and are particularly important when core samples are unavailable or only partially available.

Cuttings have been utilized in previous deep drilling projects, mainly for lithology identification and studies on rock physical properties. In the German Continental Deep Drilling Program (KTB), a suite of petrophysical properties were obtained from cuttings (see review by Berckhemer et al., 1997; Emmermann and Lauterjung, 1997). In the San Andreas Fault Observatory at Depth (SAFOD), intensive analyses were conducted on cuttings sample from fracture and fault zone (e.g., Carpenter et al., 2011; Morrow et al., 2007). In addition to physical properties, mineralogical and geochemical analyses provided insights into in-situ faulting process and fluid-rock interactions (Schleicher et al., 2006; 2009). These studies show the value of cuttings from a deep hole where core samples are available only sparsely.

In scientific ocean drilling, the scientific use of cutting samples started when Chikyu began riser drilling in 2009. Some of the pioneer studies were carried out successfully using cuttings samples from Nankai Trough Seismogenic Zone Experiment (NanTroSEIZE) expeditions (Noguchi and Okamura, 2014; Takeshita et al., 2014). On the other hand, it was pointed out that the data from core samples and cuttings samples show differences; e.g., inorganic carbon content of cuttings was higher than those of core samples of IODP Expedition 337 (Expedition 337 Scientists, 2013a). The cause of the difference was, however, not fully discussed and standard protocol for the scientific use of cutting samples is not yet fully developed.

One difficulty of using cuttings samples for scientific analyses is that cuttings are more prone to contamination by drilling mud than core samples are. Small cuttings are fully coated by drilling mud during their transport from the borehole to the surface. It is not easy to remove the drilling mud from the cuttings, even after repeated washing. Removal of drilling mud is a crucially important problem when using cuttings for chemical analyses.

This report describes our attempt at different methods of washing cuttings, with a view to improve the routine protocol for washing a large amount of cuttings samples. Three treatments were attempted: (1) soak cuttings in seawater, (2) soak and stir cuttings in heated seawater, and (3) mill cuttings and soak the powdered cuttings in seawater. To evaluate the treatment methods, we examined the variations of chemical compositions of the washed cuttings.

2. Cutting, seawater, and drilling mud

Two cutting samples taken from Hole C0020A during Expedition 337 were used for this study. Sample 352SMW was taken from 2296.5–2306.5 m mud depth below the seafloor (mMSF). Sample 385SMW was taken from 2436.5–2446.5 mMSF (Table 1). Both intervals correspond to Unit IV in the hole (Expedition 337 scientists, 2013a). After the expedition, each sample was stored

| Sample | Source | Lithology | Size fraction | Processing | Initial wash | mill | Soak in RT | Stir+warm |
|--------|--------|-----------|---------------|------------|--------------|------|------------|-----------|
| A-1    | 352SMW | Silty shale | 1-4 mm        | yes        | -             | -    | -          | -         |
| A-2    | 352SMW | Silty shale | > 4 mm        | -          | 6 h          | -    | -          | -         |
| A-3    | 352SMW | Silty shale | > 4 mm        | yes        | -             | -    | 6 h        | -         |
| A-4    | 2296.5-2306.5 mMSF | Silty shale | > 4 mm        | yes        | -             | -    | 24 h       | -         |
| B-1    | 385SMW | Sandstone | 1-4 mm        | yes        | -             | -    | -          | -         |
| B-2    | 385SMW | Sandstone | > 4 mm        | -          | -             | -    | -          | -         |
| B-3    | 385SMW | Sandstone | > 4 mm        | yes        | -             | -    | 24 h       | -         |
| B-4    | 385SMW | Sandstone | > 4 mm        | -          | -             | -    | 24 h       | -         |

Table 1. List of cutting samples and processing methods used for this study. RT, room temperature.
unwashed in a 1 L-polyethylene container. Each was then kept at 4°C in a refrigerator for about 8 months.

Seawater used for this study was taken on May 22, 2013 when the Chikyu was located off Sado Island in the Sea of Japan. The seawater was put into a 20 L polyethylene container and was kept at room temperature in the Chikyu laboratory, which is air-conditioned at 20°C.

Riser drilling mud used in Exp. 337 was composed of KCl, NaCl, bentonite, barite (BaSO₄), polymer derived from cellulose, starch and polypropylene glycol, and sulfonated asphalt as hole stabilizer. Other minor components such as remains of lost circulation materials include mica, calcium carbonate, and grains of organic origin. These were mixed to prepare drilling mud with density maintained at 1.06–1.11 sg. The components and their concentrations were variable through drilling at Hole C0020A. In addition to reports from a mud engineer, several drilling mud samples were analyzed during Exp. 337. Table 2 shows results obtained for 373LMW, the most relevant drilling mud sample, taken between the sampling of 352SMW and 385SMW.

Experiments described in the following section were conducted during May–June, 2013 in the Chikyu laboratory.

### 3. Sample preparation and instruments

The sample preparation flow is presented in Fig. 1. A batch of 120 cc of cutting samples was quickly washed at once in seawater and was sieved with 0.25 mm mesh. The samples of >0.25 mm were sieved again in seawater for separation of 1–4 mm and >4 mm size fractions. The second sieving duration was 30 s. Iron grains, which were regarded as broken pieces of drill bit and drilling equipment, were removed using a hand magnet. Next, the sample batches were washed and sieved until the required volume of samples was obtained. To minimize variation in lithology, fragments of similar lithology were picked from the >4 mm size fraction. When the collected volume was insufficient, the 1–4 mm fraction was also used. In all, 160 cc of dark grayish-brown silty shale was selected from 352SMW to prepare eight 20 cc samples: Samples A1–A4 and B1–B4 in Table 1. From 385SMW, 40 cc of grayish brown to dark grayish brown silty shale, and another 40 cc of bluish gray to dark bluish gray sandstone were selected. Those were divided into four 10 cc samples respectively and were mixed to prepare four 20 cc samples: Samples C1–C4 in Table 1.

Each sample set A–C was divided into four subsamples. Subsample 1 was vacuum-dried immediately without further washing. Subsample 2 was soaked in 200 cc of seawater for fixed periods; then they were vacuum dried. Subsample 3 was soaked in 200 cc of warm (approx. 40°C) seawater with a stirring bar, and were stirred gently for the same period as Subsample 2. Subsample 4 was milled in an agate mortar. Then the milled powder was soaked in 200 cc of seawater for the same period as that used for Subsample 2.

The soaking time was 6 h for sample series A, 24 h for sample series B, and 3 h for sample series C. After

| Sample | Sample | Unwashed sample | >0.25 mm | >4 mm | 1-4 mm | Mill | Seawater | Subsample of seawater from beaker | Soak in seawater | Stir in seawater | Dry | Mill to powder |
|--------|--------|-----------------|----------|-------|--------|------|---------|----------------------------------|----------------|----------------|-----|---------------|
| 373LMW | 373LMW | Unwashed sample | >0.25 mm | >4 mm | 1-4 mm | Mill | Seawater | Subsample of seawater from beaker | Soak in seawater | Stir in seawater | Dry | Mill to powder |
|        |        |                |          |       |        |      |         |                                  |                |                |     |               |
|        |        |                |          |       |        |      |         |                                  |                |                |     |               |
|        |        |                |          |       |        |      |         |                                  |                |                |     |               |
|        |        |                |          |       |        |      |         |                                  |                |                |     |               |

**Table 2. Analysis of drilling mud sample 373-LMW (from Table T12, Expedition 337 Scientists, 2013a)***

| Sample | Na⁺ (mM) | K⁺ (mM) | Mg²⁺ (mM) | Ca²⁺ (mM) | Cl⁻ (mM) | SO₄²⁻ (mM) |
|--------|----------|---------|-----------|-----------|----------|------------|
| 373LMW | 1019.8   | 668.84  | 2.23      | 4.05      | 1394.39  | 50.99      |

Fig. 1. Four washing methods were used for this study: washing during sieving, soaking in seawater, soaking milled sample in seawater, and stirring in seawater. All samples were dried and milled for measurements. EA, elemental analyzer; CA, carbonate analyzer.
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soaking, samples were filtered and vacuum-dried for 24 h. Then they were milled using a ball mill at 200 rpm for 6 min.

After the cuttings were removed, 2 ml of the soaking seawater was sampled to examine changes in the chemical composition. The subsampled seawater was divided into two portions. Then 6 M HCl was added to one for cation analysis.

The cutting samples were measured using XRF, XRD, an elemental analyzer (EA), and a carbonate analyzer (CA) to ascertain the major element concentrations, mineral composition, total carbon, organic carbon, nitrogen and sulfur contents. We also used ion chromatography to ascertain the concentrations of cation and anion in seawater samples. The measurement instruments and protocol, unless otherwise noted, are the same with those used in Exp. 337 (Expedition 337 Scientists, 2013b).

4. Results

4.1 Major elements

Major element concentrations from XRF data are presented in Table 3. The concentrations of the 10 major elements (Na2O, MgO, Al2O3, SiO2, P2O5, K2O, CaO, TiO2, MnO, and Fe2O3) are corrected by multiplying (100-LOI)/100 to include volatile components, where LOI is the loss on ignition. Results show that Sample C, containing sandstone, has higher concentrations of SiO2 and Na2O than either Sample A or Sample B.

Table 3. XRF data shows the concentrations of the 10 major elements (Na2O, MgO, Al2O3, SiO2, P2O5, K2O, CaO, TiO2, MnO, and Fe2O3). The values are corrected by multiplying (100-LOI)/100 to include volatile components, where LOI is the loss on ignition.

| Sample Name | Na2O mass% | MgO mass% | Al2O3 mass% | SiO2 mass% | P2O5 mass% | K2O mass% | CaO mass% | TiO2 mass% | MnO mass% | Fe2O3 mass% | LOI % | Total mass% |
|-------------|------------|-----------|-------------|-------------|------------|-----------|-----------|------------|-----------|-------------|-------|-------------|
| A-1         | 2.155      | 2.138     | 15.268      | 56.785      | 0.154      | 4.040     | 0.876     | 0.731      | 0.124     | 6.909       | 9.914 | 99.094      |
| A-2         | 2.104      | 2.304     | 15.471      | 58.084      | 0.194      | 3.197     | 1.028     | 0.743      | 0.134     | 7.176       | 8.541 | 98.997      |
| A-3         | 2.312      | 2.289     | 15.415      | 57.938      | 0.169      | 3.154     | 1.023     | 0.743      | 0.142     | 7.198       | 8.545 | 98.992      |
| A-4         | 2.180      | 2.450     | 15.649      | 58.138      | 0.163      | 2.831     | 1.025     | 0.748      | 0.138     | 7.218       | 8.752 | 99.292      |
| B-1         | 2.186      | 2.120     | 15.060      | 56.578      | 0.163      | 4.031     | 0.933     | 0.721      | 0.135     | 7.084       | 10.157 | 99.168      |
| B-2         | 2.140      | 2.313     | 15.480      | 58.095      | 0.160      | 3.187     | 1.003     | 0.747      | 0.138     | 7.213       | 8.543 | 99.020      |
| B-3         | 2.364      | 2.326     | 15.611      | 57.857      | 0.158      | 3.073     | 0.978     | 0.744      | 0.129     | 7.042       | 8.827 | 99.108      |
| B-4         | 2.239      | 2.401     | 15.425      | 57.793      | 0.167      | 2.841     | 0.997     | 0.743      | 0.139     | 7.186       | 8.951 | 98.881      |
| C-1         | 2.377      | 1.774     | 14.284      | 61.674      | 0.110      | 4.004     | 0.869     | 0.594      | 0.057     | 5.424       | 8.134 | 99.301      |
| C-2         | 2.182      | 1.815     | 14.196      | 63.381      | 0.102      | 3.292     | 1.004     | 0.599      | 0.065     | 5.594       | 6.914 | 99.145      |
| C-3         | 2.505      | 1.903     | 13.955      | 63.382      | 0.116      | 3.073     | 1.131     | 0.584      | 0.063     | 5.517       | 6.815 | 99.047      |
| C-4         | 2.330      | 1.979     | 13.300      | 63.253      | 0.112      | 2.954     | 0.940     | 0.612      | 0.067     | 5.621       | 6.941 | 99.109      |

| Error (1σ)  | 0.037      | 0.062     | 0.010       | 0.037       | 0.002      | 0.019     | 0.017     | 0.010      | 0.002     | 0.013       | 0.283 | 0.296       |

4.2 Total C, N, S, and TOC

Table 4 presents the contents of total carbon (TC), total nitrogen (TN), total sulfur (TS), inorganic carbon (IC), and total organic carbon (TOC). TOC is calculated by subtracting the IC from the TC. With the exception of TS, all contents are higher in Samples A and B than in Sample C.

The TC and TOC contents decrease 0.6–0.8% during soaking with seawater. The TS contents also decrease by soaking, although changes in TN and IC contents are not significant. The milling effects show a further decrease in TC and TOC, but the effects of stirring are inconsistent.

4.3 Mineral composition

Table 5 presents X-ray counts for representative minerals. All samples were composed mainly of quartz (2θ = 26.7°), albite (2θ = 28.0°), orthoclase (2θ = 31.8°), chlorite-serpentine (2θ = 12.5°), muscovite (2θ = 8.9°), and pyrite (2θ = 56.5°). Sample C shows higher counts of quartz and albite than Sample A or B. Smectite group was not detected. X-ray counts of albite and chlorite-serpentine increase by soaking, whereas those of orthoclase decrease after soaking. The effects of milling and stirring were
inconsistent. After soaking, pyrite tends to be washed away to below the detection limit.

4.4 Seawater

Table 6 presents the Na\(^{+}\), K\(^{+}\), Mg\(^{2+}\), Ca\(^{2+}\), Cl\(^{-}\), and SO\(_4\)\(^{2-}\) concentrations in seawater sampled before and after soaking. Increased K\(^{+}\) concentration after soaking agrees with the decrease in K\(_2\)O in XRF data. The Na\(^{+}\), Cl\(^{-}\), and SO\(_4\)\(^{2-}\) concentrations also showed minor increases after soaking.

Relations between soaking time and the differences among Na\(^{+}\), K\(^{+}\), and Cl\(^{-}\) concentrations are presented in Fig. 2. Dissolution progressed with time, particularly in the stirred samples. However \(\Delta\)Cl\(^{-}\) decreased after 6 h.

Table 4. The contents of total carbon (TC), total nitrogen (TN), total sulfur (TS), inorganic carbon (IC), and total organic carbon (TOC). TOC is calculated by subtracting the IC from the TC.

| Sample Name | Total carbon (wt %) | Total nitrogen (wt %) | Total sulfur (wt %) | Inorganic carbon (wt %) | Total organic carbon (wt %) |
|-------------|---------------------|-----------------------|--------------------|-------------------------|-----------------------------|
| A-1         | 3.187               | 0.080                 | 0.115              | 0.431                   | 2.756                       |
| A-2         | 2.372               | 0.077                 | 0.085              | 0.450                   | 1.922                       |
| A-3         | 2.268               | 0.076                 | 0.097              | 0.468                   | 1.800                       |
| A-4         | 2.356               | 0.076                 | 0.091              | 0.452                   | 1.903                       |
| B-1         | 3.193               | 0.076                 | 0.104              | 0.473                   | 2.719                       |
| B-2         | 2.353               | 0.076                 | 0.082              | 0.478                   | 1.875                       |
| B-3         | 2.290               | 0.074                 | 0.096              | 0.432                   | 1.858                       |
| B-4         | 2.501               | 0.075                 | 0.085              | 0.459                   | 2.042                       |
| C-1         | 2.460               | 0.056                 | 0.209              | 0.401                   | 2.065                       |
| C-2         | 1.890               | 0.054                 | 0.149              | 0.462                   | 1.428                       |
| C-3         | 1.615               | 0.054                 | 0.144              | 0.475                   | 1.141                       |
| C-4         | 1.832               | 0.057                 | 0.217              | 0.410                   | 1.422                       |
| Error (1σ)  | 0.020               | 0.004                 | 0.050              | 0.009                   | 0.022                       |

Table 5. X-ray counts for representative minerals from XRD data.

| Sample Name | Quartz Height (cps) | Albite Height (cps) | Orthoclase Height (cps) | Chlorite-Serpentine Height (cps) | Muscovite Height (cps) | Pyrite Height (cps) |
|-------------|---------------------|---------------------|-------------------------|----------------------------------|------------------------|--------------------|
| A-1         | 1688                | 280                 | 132                     | 33                               | 19                     | 15                 |
| A-2         | 1675                | 338                 | 97                      | 40                               | 17                     | n/d                |
| A-3         | 1697                | 244                 | 118                     | 36                               | 13                     | n/d                |
| A-4         | 1723                | 233                 | 94                      | 42                               | 12                     | n/d                |
| B-1         | 1625                | 225                 | 142                     | 31                               | 19                     | 20                 |
| B-2         | 1655                | 262                 | 114                     | 33                               | 13                     | n/d                |
| B-3         | 1644                | 289                 | 125                     | 41                               | 17                     | 33                 |
| B-4         | 1803                | 251                 | 124                     | 38                               | 19                     | n/d                |
| C-1         | 2453                | 332                 | 120                     | 24                               | 17                     | 25                 |
| C-2         | 2694                | 448                 | 76                      | 31                               | 25                     | n/d                |
| C-3         | 2633                | 475                 | 107                     | 27                               | 27                     | n/d                |
| C-4         | 2565                | 419                 | 76                      | 33                               | 22                     | 22                 |

Table 6. The Na\(^{+}\), K\(^{+}\), Mg\(^{2+}\), Ca\(^{2+}\), Cl\(^{-}\), and SO\(_4\)\(^{2-}\) concentrations in seawater sampled before and after soaking.

| Sample Name | Na\(^{+}\) (mM) | K\(^{+}\) (mM) | Mg\(^{2+}\) (mM) | Ca\(^{2+}\) (mM) | Cl\(^{-}\) (mM) | SO\(_4\)\(^{2-}\) (mM) |
|-------------|----------------|----------------|-----------------|----------------|----------------|---------------------|
| A-Seawater  | 454.7          | 9.8            | 51.0            | 10.0           | 532.1          | 26.5                |
| A-2         | 461.9          | 18.3           | 50.9            | 9.8            | 541.2          | 27.0                |
| A-3         | 457.0          | 18.6           | 50.5            | 9.7            | 541.1          | 26.9                |
| A-4         | 465.6          | 23.5           | 50.5            | 9.7            | 543.5          | 27.0                |
| B-Seawater  | 446.7          | 9.7            | 50.2            | 9.8            | 540.0          | 26.8                |
| B-2         | 463.3          | 36.4           | 52.3            | 9.8            | 547.3          | 27.3                |
| B-3         | 460.4          | 32.0           | 51.0            | 9.6            | 547.4          | 27.3                |
| B-4         | 481.1          | 34.5           | 52.2            | 9.9            | 566.8          | 28.2                |
| C-Seawater  | 456.9          | 9.9            | 51.5            | 10.1           | 528.4          | 26.2                |
| C-2         | 466.4          | 19.0           | 51.3            | 9.9            | 544.7          | 27.0                |
| C-3         | 457.4          | 17.6           | 50.2            | 9.7            | 534.5          | 26.6                |
| C-4         | 460.2          | 26.8           | 52.6            | 10.1           | 552.0          | 27.4                |
| Error (1σ)  | 2.7            | 0.1            | 0.3             | 0.06           | 2.7            | 0.1                 |
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5. Discussion

Major element analyses reveal that Na$_2$O and K$_2$O contents of the cuttings decrease by soaking. Considering the order of magnitude difference between drilling mud and seawater, we infer that changes in K contents reflect the removal of drilling mud from cuttings. Changes in Na contents also provide supportive data. However, most other element concentrations, such as those of Mg, Al, Si, Ca, Ti, Mn, and Fe, showed slight increases, suggesting an inverse relation that is dominated by decreased Na and K.

5.1 Dissolution of KCl from cuttings

To minimize the effects of inter-elemental variations, K$_2$O content is divided by TiO$_2$ contents (Fig. 3). The K$_2$O/TiO$_2$ ratios in XRF data decrease and the K$^+$ concentration in seawater increased after the soaking, indicating that KCl is dissolved during soaking. All samples show a decrease of K$_2$O/TiO$_2$. Subsamples 4 (stirring) exhibited the greatest decrease.

Figure 3 shows that K$_2$O/TiO$_2$ ratios exhibited little change with soaking time between 6 h and 24 h. Although the differences between 3 h and 6 h might be attributable to different lithology of the samples, the data indicate that the soaking time of 6 h was sufficient to reach equilibrium.

5.2 Dissolution of NaCl from cuttings

Figure 4 shows that Na$_2$O/TiO$_2$ ratios decrease by the soaking, with or without stirring. When the cuttings are milled before soaking, the Na$_2$O/TiO$_2$ ratios increase after the soaking (Subsample 3). The Na$^+$ in seawater increased in all cases, but the rate of increase was minimal in the case of the milled samples. These results suggest that Na$^+$ was absorbed on the increased surface of the milled cuttings. The absorption is possible only if the inside of the cuttings have lower Na content than the original surface because, otherwise, the ratio of dissolution versus absorption would be the same whether or not the samples were milled. This fact supports the hypothesis that the dissolved Na$^+$ originates from drilling mud that remained on the cutting surfaces.

Similarly to the case of K$_2$O/TiO$_2$ presented in Fig. 3, Fig. 4 shows that Na$_2$O/TiO$_2$ ratios exhibited only slight differences between the soaking times of 6 h and 24 h. That fact confirms that the soaking time of 6 h was sufficient to reach equilibrium.

5.3 Contamination of organic material and removal from cuttings

The LOI and TOC contents of the cuttings decrease after soaking, suggesting that water removed organic materials (Figs. 5 and 6). Sample C3 showed a notable
Fig. 3. Content ratios of K$_2$O/TiO$_2$ in cuttings decreased after soaking, but showed little difference between 6h (sample group A, left) and 24h (sample group B, middle).

Fig. 4. Content ratios of Na$_2$O/TiO$_2$ in cuttings decreased after soaking, but showed little difference between 6h (sample group A, left) and 24h (sample group B, middle).
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Fig. 5. LOI in cuttings decreased by soaking, but showed slight effects from milling and stirring.

Fig. 6. TOC in cuttings decreased by soaking but showed slight differences between soaking time of 6 h (sample group A, left) and 24 h (sample group B, middle).
decrease in TOC, but it remains unclear whether the removed organic carbon originated from the drilling mud or from the cuttings. Slight differences were found between 6 h and 24 h soaking times. Effects on milling or stirring were minor and inconsistent.

5.4 Variation of mineral compositions by soaking

Figure 7 shows X-ray count ratios of (top) orthoclase/quartz and (bottom) chlorite-serpentine/quartz. The rate of decrease of orthoclase/quartz by soaking with seawater might reflect the orthoclase hydrolysis during

Fig. 7. X-ray count ratios of orthoclase/quartz (top) and chlorite-serpentine/quartz (bottom).
soaking, but further analyses must elucidate the clay mineral behavior.

5.5 Proposed cutting treatment to reduce drilling mud effects

Our results demonstrate that K2O/TiO2 ratios decrease more than 30% by soaking in heated seawater at 40°C for 6 h. Furthermore, the Na2O/TiO2 ratios decrease by soaking with seawater, but the rate of decrease is less than 10% when the sample is not milled. Moreover, TOC and TS contents decrease by soaking with or without heating and stirring. These results suggest that stirring of cuttings in heated seawater at 40°C for 6 h after quick washing is effective for XRF and TOC analyses. Further experiments can assess the treatments and suggest the best protocol.

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References

Berckhemer, H., Rauen A., Winter H., Kern H., Kontny A., Lienert M., Nover G., Pohl J., Popp T., Schult A., Zinke J., Soffel H. C. (1997), Petrophysical properties of the 9-km-deep crustal section at KTB, J. Geophys. Res., 102(B8), 18337–18361, doi:10.1029/96JB03396.

Carpenter, B.M., Marone, C. and Saffer, D.M. (2011), Weakness of the San Andreas Fault revealed by samples from the active fault zone, Nature Geoscience, 4, 251–254, doi:10.1038/ngeo1089.

Emmermann, R. and J Lauterjung (1997), The German Continental Deep Drilling Program KTB: Overview and major results, J. Geophys. Res., 102(B8), 18179–18201, doi:10.1029/96JB03945.

Expedition 337 Scientists (2013a), Site C0020, In: Inagaki, F., K.-U. Hinrichs, Y. Kubo, and the Expedition 337 Scientists, Proc. IODP, 337, Tokyo (Integrated Ocean Drilling Program Management International, Inc.), doi:10.2204/iodp.proc.337.102.2013.

Morrow, C., J. Solum, S. Tembe, D. Lockner, and T.-F. Wong (2007), Using drill cutting separates to estimate the strength of narrow shear zones at SAFOD, Geophys. Res. Lett., 34, L11301, doi:10.1029/2007GL029665.

Noguchi, T. and K. Okamura (2014), Data report: long-term storage of cuttings for geochemical research, In: Saffer, D., L. McNeill, T. Byrne, E. Araki, S. Tozcko, N. Eguchi, K. Takahashi, and the Expedition 319 Scientists, Proc. IODP, 319, Tokyo (Integrated Ocean Drilling Program Management International, Inc.), doi:10.2204/iodp.proc.319.201.2014.

Schleicher, A. M., B. A. van der Pluijm, J. G. Solum, and L. N. Warr (2006), Origin and significance of clay-coated fractures in mudrock fragments of the SAFOD borehole (Parkfield, California), Geophys. Res. Lett., 33, L16313, doi:10.1029/2006GL026505.

Schleicher, A. M., S. N. Trouscher, B. A. van der Pluijm, and L. N. Warr (2009), Constraints on mineralization, fluid-rock interaction, and mass transfer during faulting at 2–3 km depth from the SAFOD drill hole, J. Geophys. Res., 114, B04202, doi:10.1029/2008JB006092.

Takeshita, T., A. Yamaguchi, and N. Shigematsu (2014), Stress reversal recorded in calcite vein cuttings from the Nankai accretionary prism, southwest Japan, Earth, Planets and Space, 66(144), doi:10.1186/s40623-014-0144-4.