Sediment samples have to go through a long purification process before clean diatom samples can be obtained. This is particularly difficult in the <10 μm fraction containing clay particles. As the structure of contaminants e.g. organic material, carbonate, mineral particles contains oxygen, it is essential to assess the degree of contamination. If high impurities exist a correction factor has to be implemented (Fig.1). We developed an updated protocol for purifying the <10 μm fraction and compared various methods of assessing the degree of purity to apply the fastest and most appropriate method for future analyses.

Preparation steps
To analyse the 18O of diatom material, it is essential to purify the original sediment samples in various physical and chemical preparation steps. Figure 2 shows the purification process. The sample is freeze-dried to remove water. Then, organic material and carbonate are removed by adding H2O2 and HCl for more than 20h on a heating plate at 50°C. The sample is sieved to gain different size fractions (>10 μm, <10 μm). The heavy liquid separation (HLS) using sodium-polytungstate (SPT) was repeated 4 times with different solutions of decreasing density (2.4 - 2.3 g/cm3). A final acid cleaning is applied to remove micro organics. Several rinsing procedures ensured a neutral pH value as well as the complete removal of the SPT solution from the sample.

Effect of preparation steps
The effect of the different cleaning stages was assessed by using energy dispersive x-ray spectroscopy (see Box, contamination tool kit) operated under the SEM. The samples were sputtered with Carbon. This is why a quality of the sample by removing organic material cannot be observed. The original sediment samples have a SiO2 content of app. 72 %. As more contaminants are (high clay content) is left in the <10 μm fractions, the purity of the >10 μm fraction increases already by sieving. A final purity of >97 % (value shifts below the instrument’s error) can be achieved in both fractions. The >10 μm fraction has a purity degree of >97 % already after the first heavy liquid separation, whereas for the <10 μm fraction the four repetitions of this step are essential.

Conclusion
Purification
For sediment cores from Lake Eirgoygyn, NE Russia, we found a purification protocol to decrease the non SiO2 fraction to <3 % for the <10 μm as well as for the >10 μm fraction. The major improvement was made by introducing a multiple heavy liquid separation with varying densities. The final acid cleaning showed no further cleaning effect and can be disregarded in the future.

Method for Contamination assessment
If there is more time it is recommended to analyze the sample with ICP-OES taking longer in the preparation phase but giving results with a higher precision. The optical methods don’t provide exact quantified results for the <10 μm fraction but SEM pictures should be used in addition to verify the degree of purity optically by providing detailed view. The EDX is the most recommended analysis after this comparison as it needs less than 0.5 mg. In addition it is the quickest technique (30/day, n=5) and its precision is good enough for the purpose of assessing degree of purity. Further tests on more biogenic standard material are carried out at the moment to underline the usefulness of EDX for contamination assessment.