A STANDARD LABORATORY PROCEDURE FOR SEPARATING CLAY-SIZED DETRITUS FROM UNCONSOLIDATED GLACIAL SEDIMENTS AND THEIR DERIVATIVES

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INTRODUCTION

The following procedure for separating clay-sized particles from glacial sediments has been employed in the Drift Prospecting Laboratory of the Terrain Sciences Division of the Geological Survey of Canada since 1973. It is important to recognize, when interpreting geochemical results from projects carried out by members or associates of this division, that the clay analyses yield significantly different results from those obtained from dry-sieved silt and clay (<250 mesh or <64μm) samples. Using this method sufficient clay-sized material for geochemical and X-ray diffraction analyses has been obtained from esker gravels (Shilts and Wyatt, 1989), till, glaciolacustrine sediments and glaciomarine sediments.

Equipment

- Centrifuge; International Centrifuge (IEC), Model DPR-6000, 6-place head with 1000-millilitre capacity and Nalgene bottles.
- Milkshake mixer; modified by replacing steel blade with Nalgene blades cut from heavy gauge labware.
- Stainless steel mixer buckets; baffles removed.
- Sodium hexametaphosphate; 5 grams per litre in distilled, deionized water.
- Nalgene cups; 100 millilitres (for drying clay).
- Stainless steel, long-handled spatula.
- Drying oven, low temperature.
- Beakers; 250-millilitre (for washing and drying sand and granule oversize).
- Agate pestle or mortar and pestle for disaggregating dried clay.

PROCEDURE

Three hundred to five hundred-gram samples are removed from plastic bags, preferably in fragments or chunks representing the sample as it occurred in outcrop, and placed in a stainless steel milkshake mixer vessel, wet. It is neither necessary nor desirable to dry the sample. Pebbles larger than 1 centimetre in diameter are removed if possible, and before adding about 200 millilitres of distilled, deionized water to which a small amount of dispersant has been added (5 g/l of reagent grade sodium hexametaphosphate is good unless phosphorus is a metal of interest; trace element "purity" of whatever dispersant is used must be confirmed). More dispersant can be added if problems with flocculation persist, but a minimum and constant amount is desirable as some dispersant is inevitably precipitated with the clay during the final drying step.

The normal stainless steel blade that is screwed on to the end of the milkshake mixing rod is replaced by a blade cut from discarded Nalgene labware. After considerable testing, it was found that no metal blade survived long when disaggregating till, with the result that considerable metal contamination could be found in sand-sized heavy minerals derived from the disaggregation process. The Nalgene blades wear rapidly, but are very cheap, and the easily recognized Nalgene residue is rarely found in the materials separated for analysis.

The sample and water slurry is mixed on the milkshake mixer machine for approximately 30 seconds and the slurry is allowed to sit for 5 to 10 seconds to allow sand and coarser grains and aggregates to settle. The slurry is then decanted into a 1000-millilitre centrifuge vessel. Another 200 millilitres of metaphosphate solution is added and the process repeated. After the second decantation, the process is repeated once more and the decanted, supernatant solution is, by this time, fairly clean. The granule-sand residue remaining in the milkshake mixer is removed and set aside to dry for further examination of pebble lithology (1 to 6 mm sizes), heavy and light mineral analysis of the sand fraction by petrographic, scanning electron microscope or geochemical techniques, and bulk magnetic susceptibility measurements of the sand fraction.

The slurry from the three decantations is now in the 1000-millilitre centrifuge vessel which is "topped up" with metaphosphate solution to 750 to 800 millilitres, depending on sediment concentration. Once six samples have been prepared this way, the vessels that are inserted on opposite sides of the centrifuge head are weighed, and metaphosphate solution is added until their weights are within 1 gram of each other, so that the six-place centrifuge will be balanced.

The next procedure is critical for adequate clay-silt separation, and lab staff should be trained to carry it out in a consistent and careful manner. The six vessels, each of which is closed with a screwtop, must be shaken briefly and vigorously so that all sediment is suspended. Then, as quickly as possible, the vessels should be placed in the centrifuge and the centrifugation process begun. To cause the silt (particles 2 to 64μm in diameter) to settle, the DPR-6000
centrifuge must be accelerated to 750 rpm as rapidly and smoothly as possible and must be held at 750 rpm for 3 minutes, after which it is decelerated rapidly and smoothly to a stop. The supernatant suspensions are poured carefully into six more 1000-millilitre centrifuge vessels, being careful not to resuspend the silt sedimented by the first centrifugation. Jackson (1956) estimates that about 75% of the -2-micron fraction is removed from the silt/clay suspension by this first centrifugation. If the original sample is clay-poor or too small to recover adequate clay, the settled silt and clay may be resuspended and the centrifugation separation repeated. In most cases this is not necessary, and in any case, after the second or third centrifugation, very little of the remaining approximately 10% of clay can be recovered. The silt with its included clay component is discarded in our procedure.

After the clay suspensions in the second set of vessels are topped up and the vessels carefully balanced, the suspensions are centrifuged at 2800 rpm for a further 14 minutes. After this time, some clay and colloids remain in suspension, but further centrifuging will cause little of this very fine sediment to settle. Electron microscope scans of clay particles separated using this procedure show that more than 99% of the particles range from 0.3 to 2 microns in true maximum dimension and, furthermore, that they consist predominantly of plate or disc-shaped aluminosilicates.

The final supernatant solution is discarded, again being careful not to resuspend any of the sedimented material on the bottom of the vessel during decantation. At this point the colour of the sediment surface and colours of any banding in the centrifuged sediment should be noted. These colours and bandings may have mineralogical and geochemical significance (Shilts, 1978). The sedimented clay is removed using a long-handled stainless steel spatula. Removal of the sticky, sedimented clay 'cake' may be facilitated by adding a very small amount of distilled, deionized water to the centrifuge vessel while it is vibrated at high frequency on the rubber pad of a vortex mixer. At this point, if clay mineralogical analysis is to be carried out in addition to geochemical analysis, the wet sample can be subsampled, and smear or other suitable mount(s) can be prepared for further chemical treatment and X-ray diffraction analysis. The portion of the sample to be used for geochemical analysis is placed in a small, disposable weigh boat and dried at least 75°C (<40°C if Hg analyses are to be done). The dried sample is then disaggregated using an agate mortar and pestle or any convenient, noncontaminating technique (the dried clay can be quite hard and difficult to pulverize). The powdered sample is submitted for geochemical analysis.

The procedure described above is used routinely at the Geological Survey of Canada to process till samples. It has been transferred to the private sector where a variety of centrifuge types are used. The centrifuge speeds and concentrating procedures have to be modified to obtain the appropriate size distribution, and this should be carefully monitored among laboratories. Also, if smaller centrifuges are used, the clay-silt fraction can be preconcentrated by dry sieving, a procedure we followed in our early application of this method. Tests of the wet and dry methods of precentrifuge disaggregation showed, however, that considerable disparity in trace element concentrations was evident in the same sample (e.g. for uranium; Klassen and Shilts, 1977). Thus, it is not recommended that preconcentration techniques involving drying be employed. Finally, although centrifuges have been used to increase the value of g and, therefore, decrease the time to settle a set distance from Stoke's Law of settling, similar results could be obtained in settling columns or calibrated beakers, but the time required for each sample is greater. In other words, the separations can be done: using various types of centrifuge to increase g (centrifuge heads capable of accepting 1000-millilitre vessels are the best); by a combination of settling (to remove silt) and centrifugation (to remove clay); or totally by settling, processing many samples in sequence, so that eventually those for which the clay has settled (in days) are being dried at the same rate as new samples are being suspended. This technique requires considerable space and organization.

If a procedure were followed where the large amount of liquid resulting from the high liquid/solid ratio in the clay suspensions could be evaporated in a reasonable length of time, the fine clay particles and colloids could also be added to the analyzed clay sample. However, practical and chemical problems arising from the concentration of deflocculants and adherence of colloidal materials to vessel walls probably obviate the need for the total evaporation method.

REFERENCES
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