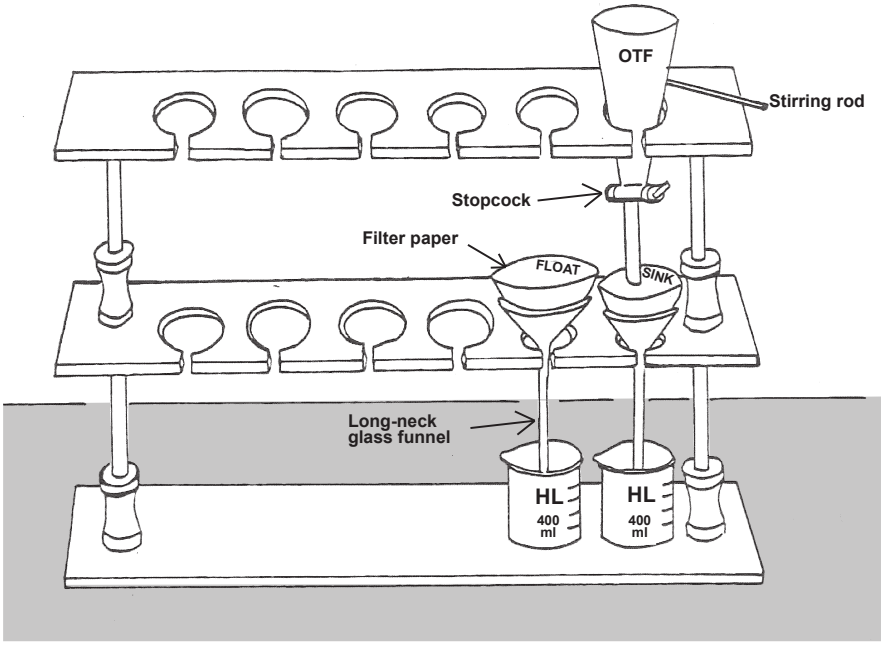
NEW JERSEY
GEOLOGICAL AND WATER SURVEY
Technical Memorandum 16-1

Protocol for Heavy-Liquid Separation of Heavy Minerals and Recovery of Non-Toxic Separation Liquids



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On the cover: Arrangement of the open-top flask with stopcock, filter papers, funnels, and beakers on the tiered rack for processing of one sample. Rack can hold set-ups to run as many as three samples concurrently. ("HL" on the 400-ml beakers is the NJGWS abbreviation for heavy liquids.) *Illustration by Jane Uptegrove.*

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Protocol for Heavy-Liquid Separation of Heavy Minerals and Recovery of Non-Toxic Separation Liquids

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2016

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Protocol for Heavy-Liquid Separation of Heavy Minerals and Recovery of Non-Toxic Separation Liquids

Introduction

This protocol was developed in the course of heavy-mineral analyses performed at the New Jersey Geological and Water Survey (NJGWS) in the 1980s and 1990s. NJGWS processed heavy-mineral samples strictly with non-toxic heavy liquid, sodium polytungstate, which was newly available at that time. This protocol details how to recover and recycle the non-toxic sodium polytungstate heavy liquid based on the full protocol detailed in Grosz and others (1990) and implemented in Uptegrove and others (1991). Some abbreviations have been changed from those in Grosz and others (1990) to reflect the change from tetrabromoethane heavy liquid to sodium polytungstate heavy liquid.

This protocol begins at the point where the heavy minerals in a sediment sample have already been concentrated in a spiral concentrator, and the "spiral heavies" have been separated from the remaining "spiral lights". A 200-gram(g) aliquot of the spiral lights is then collected and processed in the same manner as the spiral heavies are processed. This is detailed as follows.

The protocol includes a MS Excel™ (1997-2003) spreadsheet entitled, "Spreadsheet for weighing of heavy mineral separates". This protocol describes only the processes covered in Part I, "Heavy-liquid separation with sodium polytungstate".

Objectives

1. To perform a density separation of the spiral heavies to further isolate the heavy fraction of the overall sample.
2. To perform a density separation of the spiral lights to extrapolate the heavy fraction not recovered in the spiral process.
3. To recover as much of the heavy liquid as possible for future separation work.

Materials

For each sample:

1 open-top flask, with stopcock closure in the neck (OTF)
3 #4 filter papers
1 glass or plastic stirring rod
four 400-ml beakers
2 long, narrow-neck glass funnels
sodium polytungstate heavy liquid (HL)
one 4000-ml flask
paper towels
distilled water
3-tiered rack
washing pan (1" x 12" plastic or glass pan)
vial for the heavy mineral sink
weighing paper
digital scale (for weights exceeding 200g)
Mettler balance (for use if scale cannot record weights below 50g and two decimal points)
pencil
permanent marker
label stickers or masking tape
drying oven
small electric burners
rectangular stainless steel pan (approx. 8" x 12" x 2") for double-boiling WASH beakers containing diluted HL
digital spreadsheet entitled, "Spreadsheet for weighing of heavy-mineral separates"

Preparation

Locate the spiral heavies and spiral lights of each core or original sample. You will be processing the entire spiral heavy sample. With respect to the spiral lights material, take a 200-g aliquot of well-mixed sample.

1. Weigh the spiral heavies concentrate and the spiral lights aliquot. Record these weights on the worksheet (fig. 3) for spiral-heavies concentrate (SH) and spiral-lights aliquot (SL), respectively. Wet samples must be dried in an oven at 100°C overnight before weighing.

For the purposes of this protocol, the total "spiral heavies" is one sample, and the spiral lights 200-g aliquot is one sample. Exactly the same set-up is required to process the spiral lights aliquot sample. A much smaller but not negligible amount of heavy minerals will be recovered from the spiral lights aliquot sample. Set aside the spiral lights aliquot sample and proceed with the following instructions. Having processed the spiral heavies sample completely, return to #2 in the protocol and process the spiral lights aliquot sample in exactly the same manner.

2. For each sample, label two #4 filter papers with permanent marker for identification and tracking. One will be labeled for the SINK separate, and one will be for the FLOAT separate (for example, for Raritan Bay #8 Spiral Heavies, label as follows: "RB#8SH SINK" and "RB#8SH FLOAT"). Fold filter paper twice to fit into funnel. Open filter in funnel so that labels are visible.

3. For separation of each sample, the following equipment is needed:
    a. 2 tall, narrow-necked glass funnels (approx. 10" long)
    b. two 400-ml beakers marked HEAVY LIQUID (HL)
    c. 1 open-top flask (OTF) with stopcock in closed position
    d. 2 labeled filters (see Fig. 1)

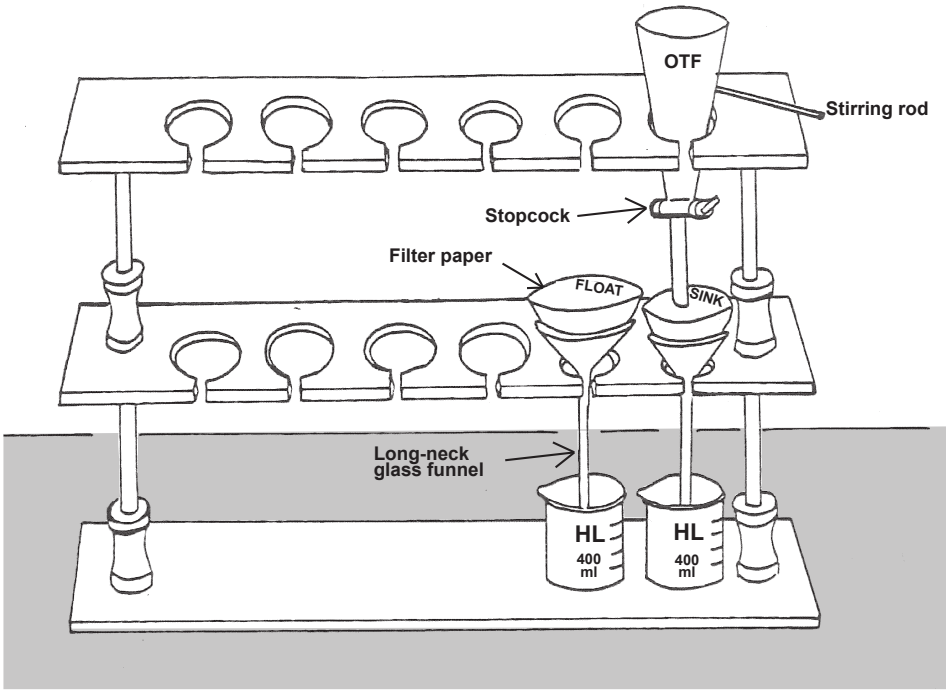

Figure 1. Arrangement of the open-top flask (OTF) with stopcock, filter papers, funnels, and beakers on the 3-tiered rack for processing of one sample. Rack can hold set-ups to run as many as three samples concurrently. ("HL" on the 400-ml beakers is the NJGWS abbreviation for heavy liquids.)

4. Arrange the OTF, the funnels with filters, and the beakers on the 3-tiered rack as shown in figure 1. This rack can hold the equipment needed for a maximum of three samples at one time.

Heavy mineral separation

(This section details the steps required for one sample.)

5. Pour the heavy liquid (HL) into the open-top flask (OTF), up to 1 inch below the rim.

6. With glass or plastic stirring rod (metal stirring rods react with the HL) in hand, slowly pour the sample into the OTF and stir into the HL. Let stand for 20-30 minutes to achieve separation. Stir occasionally to free entrained grains from both the sink and the float. At this time, the 4000-ml flask should be set up with a large plastic funnel lined with paper towel filter for recovering the diluted heavy liquids (DHL). Set oven temperature to 100° C for subsequent drying of the samples.

7. After 20-30 minutes, a significant amount of sample will have floated to the top of the HL in the OTF and will have become semi-hardened. This is the FLOAT. At the bottom of the OTF, just above the closed stopcock, the higher density heavy-mineral grains will have accumulated to form the SINK. The separation is complete when the HL in the OTF between the semi-hardened FLOAT and the accumulated SINK is clear.

8. The OTF is positioned above the funnel and beaker with the paper filter labeled "SINK". At this time, open the stopcock just long enough to clear the accumulated SINK heavy minerals from the base of the OTF. Close stopcock immediately. The heavy minerals are captured on the filter. The heavy liquid (HL) drains into the beaker below. Open the stopcock again very briefly to enable a small amount of the clear heavy liquid to flush any entrained grains out of the neck of the OTF onto the filter paper. Close quickly.

9. Remove the closed OTF from its position over the SINK beaker. Reposition it above the funnel and beaker with the filter paper labeled FLOAT. Open stopcock. Allow the remaining liquid and float to drain. This contains most of the contents of the OTF. A large amount of sediment grains remain in the OTF after the liquid has drained.

10. At this point, undiluted HL has drained through the filter paper in both funnels and is in both 400-ml beakers. It should be transferred from the beakers to a closed container (preferably the original plastic bottle). To avoid loss of any HL, rinse the used HL beakers with a squeeze bottle of distilled water over the paper-towel filter and funnel set in the 4000-ml flask.

Recovering the heavy liquid

Washing the "SINK" apparatus

11. Label a #4 filter paper with the sample # and the Word "SINK" (for example, "RB#8SH SINK"). Fold and place in a short narrow-neck funnel (approx. 5-6" long) over a 400-ml beaker marked "WASH". This differentiates the beaker contents as DHL. A filter paper is used due to the relatively small size of this fraction.

12. Return to the original sink filter and funnel. Place in 12" plastic or glass pan. Wash with a squeeze bottle of distilled water and pour into the short funnel with the beaker marked "WASH". Make sure no sample remains in pan.

Washing the "FLOAT" apparatus

13. Set up a plastic funnel with a wider neck (approx. ½ inch) and paper-towel filter clearly marked "FLOAT WASH" and the name of the sample (for example, "RB#8SH FLOAT WASH") and place over the 400-ml beaker marked "WASH". Place the OTF, glass funnel, filter paper and stirring rod in the plastic or glass pan. Rinse all with a squeeze bottle of distilled water until clean. Pour contents of pan, including the diluted HL, onto the paper towel filter and wider neck funnel. Again, be sure not to lose any of the sample. Limit the amount of distilled water used to rinse the OTF, glass funnel, and stirring rod. This reduces the amount of time needed to condense the diluted HL back to the required density (2.89 gm/cm^3). And, watch that the filtered diluted HL "WASH" does not overflow the 400-ml beaker.

14. After steps 10-13 are completed, pour the DHL from the wash beakers through a paper-towel filter into the 4000-ml flask.

15. Place the SINK wash and FLOAT wash samples (material captured on the filter papers during the washing process) in oven at 100° C. The relatively larger FLOAT wash samples on paper-towel filters require more time to dry than the smaller SINK wash samples on #4 filter paper.

16. Pour DHL from large flask into 400-ml beakers and place in rectangular stainless steel pan half-filled with water. Place pan on the electric burners. Evaporate the DHL by double-boiling in the large pan, weighing periodically to monitor the density. A 100-ml sample of HL at the correct density weighs 289.0 g. If beakers in bath develop sediment at the bottom, filter through a small funnel with #4 filter paper. After liquid is at the correct density, place it in one of the original containers. Mark the container "Recycled", with the measured density and the current date.

17. Starting at #2, repeat this process for the Spiral Lights aliquot of the same sample.

Weighing, recording, labeling, and archiving the SINK separates

18. After drying, very carefully remove the SINK separate from filter paper and place on a tared weighing paper on the scale and weigh. Measure the weight to two decimal points, (for example, xx.xx g). Record on spreadsheet. Spiral heavies SINK is recorded on line labeled **SHSPTS**, for Spiral Heavies sodium polytungstate SINK. (Spiral lights aliquot SINK is recorded on the line labeled **SLSPTS**, for Spiral Lights sodium polytungstate SINK). Transfer to vial and label clearly with sample name and weight (for example, RB#8 SHSPTS, _____g, or RB#8 SLSPTS, ____ g).

Combining and storing the FLOAT separates

19. Combine the FLOAT wash separates of the spiral heavies and spiral lights aliquot. Store the combined float wash in the original sample jars and label as float wash (for example, RB#8 FLOAT WASH). The weight of the float is not required in the assessment. However, we recommend archiving it along with the vial of heavy minerals for each sample.

Proceeding to next step in Grosz and others (1990) protocol

20. The heavy-liquid separation of the sample and the recovery of the non-toxic heavy liquid is complete and the weights listed in Part I of the spreadsheet have been recorded. Proceed to the Magnetic Mineral Separation section of the Grosz and others (1990) protocol.

Acknowledgements

Amanda Santangelo and Kathleen Vandegrift formatted the manuscript and figures for publication.

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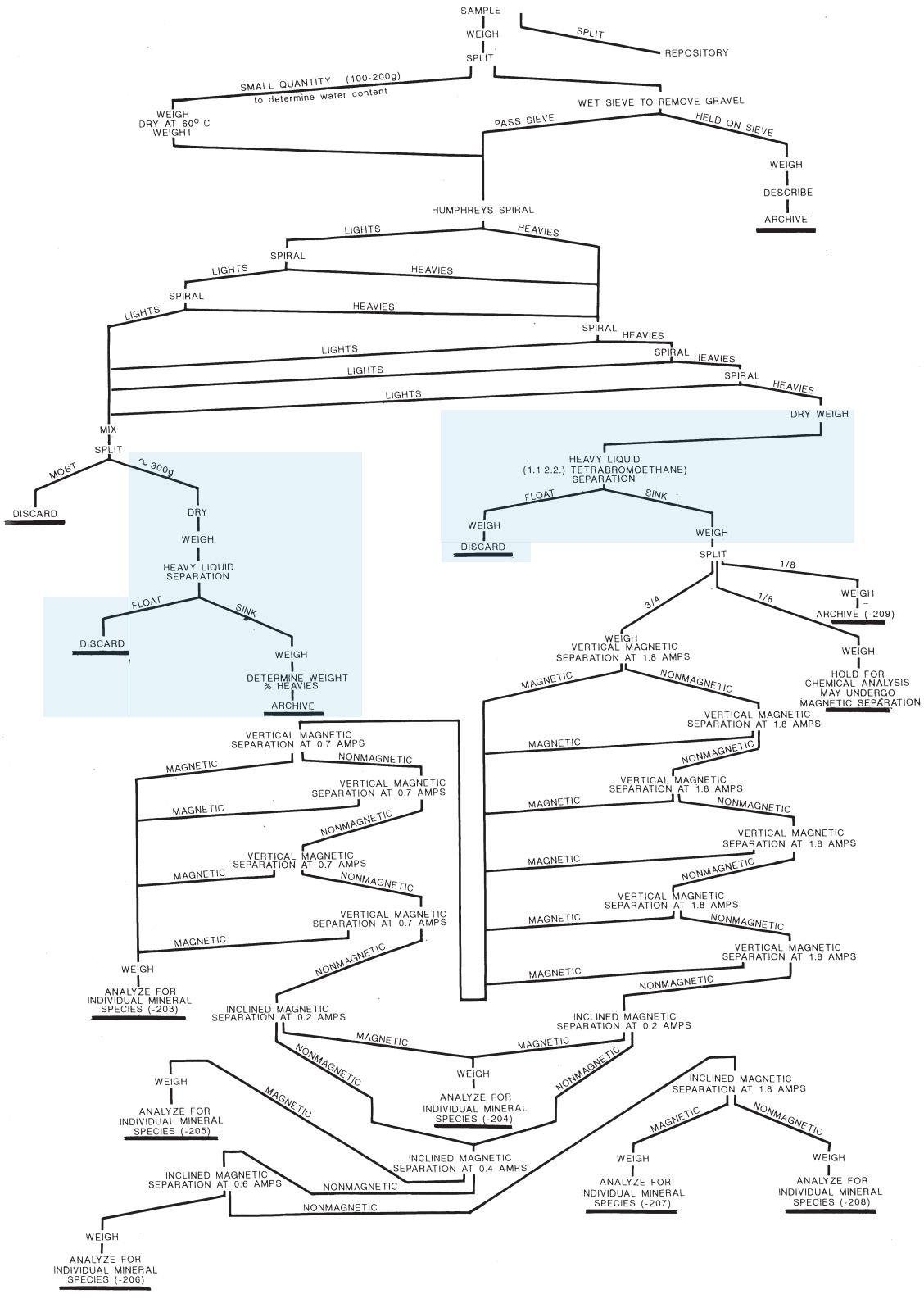

Figure 2. Flowchart from Grosz and others (1990), showing the scheme of sample processing and analysis. Blue shaded area is the section of the process covered by this protocol.

A	B	C	D	E	F
New Jersey Geological and Water Survey, 2015 **Spreadsheet for weighing of heavy-mineral separates** all measurements are listed in grams (g) unless otherwise noted					
		Part I: Heavy liquid separation with sodium polytungstate			
		SPIRAL HEAVIES		Spiral Lights approx. 200g ALIQUOT	
Sample name/#	**Date**	Weight of Spiral Heavies Concentrate, aka **SH**	Weight of Heavy Mineral Sink from the Spiral Heavies Concentrate, aka **SHSPTS**	Weight of Spiral Lights 200-g Aliquot, aka **SL**	Weight of Heavy Mineral Sink from the Spiral Lights Aliquot, aka **SLSPTS**
RB#8 (e.g. only)	10/10/12	330.90	115.20	373.10	1.81

G	H	I	J	K	L	M	N	O
New Jersey Geological and Water Survey, 2015 **Spreadsheet for weighing of heavy-mineral separates** all measurements are listed in grams (g) unless otherwise noted								
Part II: Magnetic separation								
WEIGHT OF MAGNETIC FRACTIONS						Total (203...208) ([G] + [H] + [I] + [J] + [K] + [L])	**SHSPTS** (from col. D) ([D])	Loss of sample during magnetic separating (**SHSPTS** minus Total of 203....208, ([N] - [M])
203 (HAND MAGNETIC)	204	205	206	207	208			
5.25	16.95	53.67	14.32	10.32	7.21	107.72	115.20	7.48

P
New Jersey Geological and Water Survey, 2015 **Spreadsheet for weighing of heavy-mineral separates** all measurements are listed in grams (g) unless otherwise noted
Comments/Notes values are listed only as examples

Figure 3. Sample copy of MS Excel™ spreadsheet, broken up into three sections due to size. Blue cells indicate heavy liquid separation process. For easy comprehension, the top row of each section indicates the column letter, as found in any MS Excel™ spreadsheet.