Neutralization of Dilute Hydrochloric Acid Waste from Barite Separation Procedure
Ellen Gray, Lab Manager

**Purpose:** The objective of this procedure is to neutralize hydrochloric acid waste safely in order to minimize the amount of hazardous waste generated by the Paytan Lab.

**Chemicals:**

1. **Hydrochloric acid waste:** In the barite separation SOP, 6M hydrochloric acid is added to sediment samples to dissolve calcium carbonates that are present. The sediment cores are from the ocean floor and do not contain any inherently hazardous material such as significant presence of heavy metals. Approximately 50-250 ml of acid is added to a 20g sample until dissolution is complete (the reaction visibly stops) and then diluted to 250 ml. After 12 hours the acid is discarded into a hazardous waste container and the sample is rinsed in 250 ml of water three times, each rinse is also discarded in the hazardous waste container. The final concentration of hydrochloric acid discarded per sample assuming full 250ml of 6M hydrochloric acid is used is 1.5M.

   **Calculation:**
   
   \[
   \text{250 ml of 6M HCl is used in the procedure treatment.}
   \]
   
   \[
   \text{The sample is rinsed 3 times in 250 ml of water}
   \]
   
   \[
   \text{Total volume per sample} = 250ml \times 4 = 1L
   \]
   
   \[
   M_1 \times V_1 = M_2 \times V_2 \Rightarrow (6M)(250ml) = (x M)(1000ml)
   \]
   
   \[
   \text{Final M} = 1.5 \text{ M}
   \]
   
   There will be some variability of this concentration.

2. **Sodium bicarbonate:** aka. Baking soda, sodium bicarbonate was chosen as the cheapest chemical available for acid neutralization.

   \[
   \text{NaHCO}_3 \text{, baking soda, cost $2.40/kg (3kg purchase, reagent grade).}
   \]
   
   \[
   \text{Grocery store: $9.30/3kg including tax } \Rightarrow $3.10/kg
   \]
   
   \[
   \text{NaHCO}_3 + \text{HCl} \Rightarrow \text{NaCl} + \text{H}_2\text{CO}_3
   \]
   
   \[
   \text{H}_2\text{CO}_3 \Rightarrow \text{H}_2\text{O} + \text{CO}_2
   \]
   
   \[
   \text{Need:} \ 1.5 \text{M NaHCO}_3 \Rightarrow 126g/L \Rightarrow 2.52kg/20L(5gal) \Rightarrow $6.00/5gal or $7.80/5 gal
   \]

**Equipment:**

1. Fume hood
2. 18 L square bottom container of HDPE plastic that is notable for its height. The 5L mark is approximately \( \frac{1}{4} \) its height. Matching lid.
3. Plastic stirring rod
4. pH strips
5. accessible pH meter
6. small beaker for sampling an aliquot on the pH meter
7. accessible balance and weigh boats for weighing NaHCO3
8. Personal protective equipment: safety goggles/glasses, lab coat, pants, closed toe shoes, Blue-Grip LL301 gloves (the big rubber gloves we use for handling acid baths. These are made by MAPA Professional - http://www.toolfetch.com/p-457-301428-4.shtml)

**Personnel:** This procedure can be done by one person. It should only be conducted during regular hours when others are present in nearby lab areas.

**Process:** 5L of hydrochloric acid waste will be neutralized by the incremental addition of 450-625g of sodium bicarbonate until the pH of the solution is > 6.

**Process Completion:** Completion is determined by a pH of > 6 on the pH meter of an aliquot of the solution. Visually, when the sodium bicarbonate is added and bubbles no longer rise (it fizzes flat and quickly), the pH is likely in the 5.5-6.5 range. At this point, check the pH to ensure that it is in the 6-9 range.

**Process Waste Disposition:** Neutralized waste is primarily water and dissolved sodium chloride (see chemical equation above) and will be disposed of down the drain. It is no longer hazardous.
Precautions / Safety Equipment / Personal Protective Equipment: Standard precautions will be taken since this process involves a strong acid. Safety goggles must be worn when moving the acid from the waste accumulation area to the hood. Once the fume hood sash is down, you may switch to glasses. For all work, the blue-grip LL-301 gloves must be worn, as well as labcoat, pants, and closed-toed shoes to minimize skin exposure. The procedure will take place entirely in the fume hood.

Training (operational and safety):
1. All Paytan Lab Safety SOP guidelines outlined in the Chemical Hygiene Plan apply
2. Topics covered in training will include personal protective gear, the procedure, and emergency response in case of a spill
3. Training will be given to interested lab users who use the barite separation procedure and generate the waste. They will be trained by Ellen Gray, lab manager and developer of this procedure one-on-one in a series of sessions: 1 – observation and explanation session, 2 – supervised session. Once the trainee demonstrates that they can perform the whole procedure without verbal reminders and is visibly comfortable with the procedure then they may work unsupervised.

Documentation:
1. During training, a training checklist specific to the neutralization procedure will be completed and once training is complete, the trainer sign and put the completed and signed training checklist in the Paytan Lab IIPP notebook.
2. A dedicated Neutralization Log will be maintained containing the date of waste generation, the name of person neutralizing, the date and time of neutralization, quantity of waste neutralized, quantity of NaHCO3 used, final pH, waste disposition, and other notes. In addition the log will contain the training records of those trained and the date their training was completed.

Procedure: Waste must be neutralized within ten days of accumulation.
Location: C474 at sink next to hazardous waste accumulation area and fume hood.
1. Wear gloves, goggles, lab coat, long pants, close-toed shoes. Warn others in working space.
2. Record the date, time, and quantity of acid to be neutralized.
3. Start with 2.5 L (empty glass original bottle) or 20L waste container of ~1.5 M HCl. Over secondary containment, pour 5 L into the square bottomed neutralization container. Because of the wide top opening, spills are minimal.
4. Place lid on neutralization container and move to the fume hood. [You procedure calls for adding the acid waste to the neutralization container outside of the fume hood, using secondary containment and then moving the neutralization container into the fume hood. Are the logistics such that this process is safer / preferable to adding the acid waste to the neutralization container inside the fume hood, using secondary containment?] [Yes. If the waste is in a 2.5 L container then it can be transferred to the square container in the hood. If it’s in a 5 Gal carboy it is too heavy to lift and manipulate in the hood.]
5. In weigh boat, weigh out ~30g of sodium bicarbonate. (I do this in sets of three.)
6. In the hood, slowly pour the sodium bicarbonate into the acid and let react. Bubbles will likely rise 5-8 cm which is well below the lip of the container.
7. After an adding ~100g of sodium bicarbonate, stir the acid with the plastic stir rod to dissolve remaining NaHCO3.
8. Repeat steps 6 and 7 until there are no longer rising bubbles; the addition of sodium bicarbonate results in a flat fizz. Check the pH with a pH strip and verify that it is in the 6-9 range.
9. Take an aliquot of the acid to measure the pH on the pH meter to verify the strip.
10. If necessary, add sodium bicarbonate in 20g increments until the pH meter reads >6, stirring between each addition. Bubbles should be flat and delayed.
11. Once the bubbles are flat and the pH >6, the acid is neutralized and may be poured down the sink with water running.
12. Record time completed, amount of sodium bicarbonate used, and other notes.