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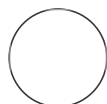
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Protocol status: Working
 We use this protocol and it's working

🌐 Preparation of a reduced Cr²⁺ solution for sulfide distillation using a Jones Reductor

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ABSTRACT

This protocol uses a Jones Reductor to prepare reduced Cr²⁺ for the distillation of H₂S from marine sediments. It is an adaptation of a protocol described by Backlund et al (DOI:<https://doi.org/10.2137/1459606054224147>)

GUIDELINES

IM1 0 Mass Percent

This procedure takes approximately 4-6 hours to complete, from setup to clean-up.

MATERIALS

12 N HCl
 1N HCl
 0.5 N HCl
 0.25 N HCl
 Granular Zinc (20-30 mesh)
 2% Mercury (II) Chloride (HgCl₂)
 1M Chromium (III) Chloride Hexahydrate (CrCl₃*6H₂O) acidified in 0.5N HCl
 60 ml plastic (polypropylene) syringes
 250 ml beakers
 Glass rod or spatula
 Small plastic funnel
 plastic tubing
 Support Stand with clamp
 glass column (ca. 40 cm x 1.5 cm internal diameter with integral glass sinter at bottom)
 stopcock (3-way) for glass column
 Citranox detergent



Many of these solutions can be toxic (i.e. HgCl_2). Set up the Jones Reductor in a fume hood and use caution and PPE for all steps.

Keywords: Jones Reductor,
Chromium reduction, Sulfide
Distillation

Jones Reductor Setup and Use

- 1 Once set up, the Jones reductor for the reduction of chromium will appear as follows.

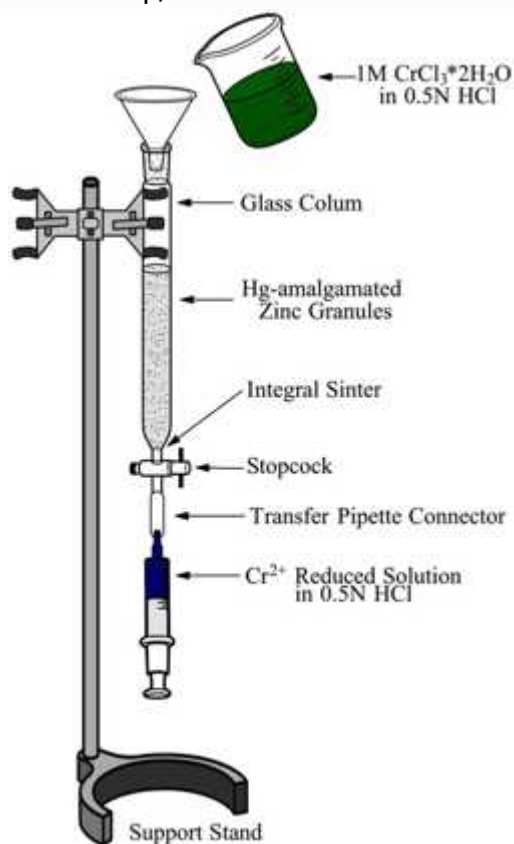


Figure 1 - Jones Reductor

- 2 Set up in a support stand a glass-column (~40-cm long, 1.5 cm interior diameter) with an integral sinter at the bottom. Secure a stopcock at the bottom of the column and turn the handle to the closed position.

- 3 Measure 40-50 g of granular zinc in a 250 mL beaker and wash for 1 minute with ~50 mL 1N HCl by stirring with a glass stir rod. Carefully decant the HCl solution into a waste beaker. Repeat the washing with 1N HCl and decantation two more times.
- 4 Wash the granular zinc from the previous step with ~50 mL DI water for 1 minute by stirring with a glass stir rod. Carefully decant the water. Repeat the washing with DI water and decantation one more time.
- 5 Cover the washed zinc granules with ~50 mL of 2% HgCl₂ solution. Stir the contents for 5 to 10 min with the aid of a stirring bar and/or glass rod or spatula.
- 6 Decant the HgCl₂ solution from the zinc granules into a waste beaker, and wash them with ~50 mL of DI water as before. Decant and repeat the washing two more times. The resulting amalgamated zinc should have a bright silvery luster.
- 7 Transfer the amalgamated zinc granules to the glass column using a plastic funnel and fill the column without going over the shoulder of the column. This completes the set-up of the Jones Reductor (See Figure No. 1).
- 8 Run 50 mL of DI water down the column into the waste beaker to wash the amalgamated zinc granules. Once done, close the stopcock and make sure the zinc granules are covered with water. Note: if the reductor is not to be used immediately, it must be left full of water in order to prevent atmospheric oxidation of the Zn granules.
- 9 Activate the amalgamated zinc granules by running ~75 mL of 0.5N HCl down the column into the waste beaker. Once done, close the stopcock and leave the zinc column submerged in HCl solution.
- 10 Begin percolating the 1M CrCl₃*6H₂O solution through the Jones reductor by opening the stopcock and letting the solution run down the column. An efficient reduction of the chromium ions is verified by a color change from dark green (Cr³⁺) solution to a bright blue (Cr²⁺) solution (See Figure No. 1).
- 11 10. As soon as the drops exiting the column turn bright blue, close the stopcock.

- 12** 11. Obtain an 8-cm long piece of narrow (5 mm OD) plastic tubing (the stems of transfer pipets will work). Attach one end to the stopcock and the other end to the tip of a plastic (polypropylene) syringe.
- 13** 12. Open the column stopcock and slowly start drawing the reduced Cr^{2+} solution from the column into the syringe. Prevent accumulation of air pockets inside the syringe as they may re-oxidize the solution.
- 14** 13. Collect 50 mL of the reduced Cr^{2+} solution into one plastic syringe. Make sure to keep adding the $\text{CrCl}_3 \cdot 6\text{H}_2\text{O}$ (green) solution to the zinc column, leaving it always covered.
- 15** 14. Once the collection is done, close the stopcock and quickly cap the syringe. If necessary, gently push out any air bubbles from the syringe before capping. Repeat 50 mL collections as necessary.
- 16** 15. Store Cr^{2+} solution at room temperature for subsequent sulfide distillation procedures.

Clean Up

- 17** Clean the zinc column by passing ~25 mL of 0.25N HCl, followed by ~25 mL of DI water. Leave the zinc column full of water for future use (2-3 weeks maximum).
- 18** Rinse each piece of glassware with DI water to dispose any solid or liquid into a waste bottle.
- 19** Wash glassware in warm, 1% Citranox solution. Rinse with Distilled H_2O (3x), followed by DI H_2O

(1x). Dry in oven for 2 hours at 100°C or air dry.

Waste Management

- 20 All liquid waste must be disposed in properly labelled waste bottles. Make sure to list every chemical in the waste label in the bottle.
- 21 All solid waste must be disposed of in a solid waste bucket.
- 22 **IMPORTANT:** any residues of the Hg-amalgamated zinc granules, including the reducing column, **MUST BE DISPOSED** into a metallic mercury waste bag.