Dimethylglyoxime reagent for cobalt analyses

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Abstract

This protocol describes how to recrystallize dimethylglyoxime in order to purify it for use in measuring total dissolved and labile cobalt using cathodic stripping voltammetry. This procedure makes a 0.1 mol L^{-1} DMG solution.

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Materials

- Methanol PA-33900HPLCCS4L by P212121
- MilliQ water by Contributed by users
- EDTA, disodium salt, dihydrate S312-500 by Fisher Scientific
- dimethylglyoxime by Contributed by users

Protocol

Dissolved cobalt analyses

Step 1.
Dissolve 0.2232 g of EDTA into 600 mL of Milli-Q for a final concentration of 10^{-3} mol L^{-1} EDTA.

Dissolved cobalt analyses

Step 2.
Weigh out approximately 1.2 g of DMG (dimethylglyoxime) onto a weigh boat, and combine it into the EDTA solution. Weigh what is left on the weigh boat (DMG is very sticky so not all of it will pour into the EDTA solution) and record the final weight that was placed into the EDTA solution.

Dissolved cobalt analyses

Step 3.
Heat this solution in a microwave at 50% power until the DMG is fully dissolved, stirring it carefully in between microwaving, and trying to prevent it from boiling. This takes up to 30 minutes, so be patient and heat it slowly and carefully.

Dissolved cobalt analyses

Step 4.
Put the solution on ice and let it recrystallize overnight in a refrigerator.

Dissolved cobalt analyses

Step 5.
After it has recrystallized, pour off (or slowly pipette off) the supernatant until there is as little liquid as possible. Then pour the remaining crystals into a clean, dry weigh boat (that you have weighed and noted the weight for later use) and let the remaining liquid evaporate overnight inside a laminar flow

hood.

Dissolved cobalt analyses

Step 6.

After all the liquid has evaporated, measure the weight boat again to get an approximate measurement of the amount of DMG you have recrystallized. Calculate how much methanol is needed to add to the DMG in order to get a final concentration of 0.1 mol L^{-1} DMG, and obtain a trace metal clean bottle of the appropriate size.

Dissolved cobalt analyses

Step 7.

Weigh the clean bottle, and then carefully transfer the recrystallized DMG into this clean bottle and re-weigh (not all the DMG is easily transferred into the clean bottle, so the re-weigh is necessary). Now recalculate the amount of optima methanol needed to get the desired concentration of 0.1 mol L-1, and add that amount of optima methanol. Make sure to test the reagent before use.