Routine application of DCO method to dissolve unnecessary iron oxides-hydroxides

Introduction

DCO method is a relatively fast and efficient application to remove unnecessary iron oxides-hydroxides (eg.: goethite, hematite). The method leaves ilmenite and magnetite grains unharmed so they can be used for further analysis (unless you use a stirring magnet, which removes them :-)). Although, the amount of sample and its iron content has a great influence on the end result. In the following table (Table I.) I present a guide about how many samples advised to choose to get about 90-100% removal from 63-125 µm sieved fraction (the numbers are not proved just estimated by experience).

Table I.: Guide to help sample selection for DCO method

Sample amount	Estimated FeO(OH) content	Amount of Na-dithionite	Time of reaction	Result
0.1 g	60%	1 g	30 min	~2000 “pure” grains
1 g	60%	2 g	30 min	75% of start FeO(OH) content remains, poor result

Chemicals and equipments to be prepared:

33.3 g $H_2C_2O_4.2H_2O$ (oxalic acid)
35.3 g Na_2CO_3
2 g $Na_2S_2O_4$ (Na-dithionite)
1l distilled water
1 mol NaCl solution
Heat plates (2 is recommended)
flasks: 500 ml (2 times) for buffer, smaller ones (80 ml, 100 ml) for complete solution
safety goggles

Process (for 0.1 -1 g sample):

1. Make a 1 mol NaCl solution in a 1 l flask.
2. Measure the oxalic acid, Na_2CO_3 and distilled water.
3. In small portions by constant stirring with a glass stick pure the oxalic acid and Na_2CO_3 into the distilled water. The end result should be a buffer solution (Na-oxalate), which maintains constant pH conditions during dissolution. The pH should be between 7.5 and 8 (7.8 is the best). This is highly important, you can add more acid or base component if necessary to reach this interval.
4. During the creation of buffer solution prepare and calibrate a pH meter for measurement.
5. Measure and calibrate the pH of the created buffer solution.
6. Set up a heat plate to 150 °C and place 90 ml buffer solution on in. Wait until you get a temperature about 70-80 °C (generally the higher the better, but the upper limit is 100 °C). Meantime measure sample amount and Na-dithionite.

7. After you have the right temperature pure the whole sample into the solution and add Na-dithionite as well constantly (eg. 1 g at the beginning and 0.5 g after every 10 minutes). Keep on stirring with glass stick.
8. Cleaning procedure: Wash out the solution with NaCl solution 5 times (to prevent clay formation) than with distilled water 3 times (to prevent salt formation).
9. After 1 hour in drying cabinet you have the purified sample(s).

Hints:

- Check temperature in every ten minutes to estimate reaction time (at 60-70 °C the method is still effective, but needs longer reaction time).
- At the beginning of the reaction the solution should be green-yellowish.
- Stirring makes the dissolution faster and more efficient, highly recommended.
- At the beginning you can create the buffer solution in several flasks to prevent the materials escape from the container due to the heavy reaction.
- For NaCl solution and buffer solution you can use heat plate (maximum 50 °C degree) to make each process faster.
- pH is not changing remarkably in 20 °C-100°C.
- Before pouring the sample into the solution take out the glass stick, otherwise samples will stuck on its surface, instead entering the solution (same goes for Na-dithionite).
- Working with solution about 7 pH or below will end up pyrite formation (I never experienced).
- Recalculate the start amount of chemicals for a better fit to your samples (you don't need a 1l buffer, only 90 ml for 0.1 g).
- Simultaneously you can treat 3-5 samples at the same time.
- $Na_2S_2O_4$ is irritative, work under fume hood and wear safety goggles, never inhale gases from the solution.

Further information in:

Goswami G., Varadachari C. és Ghosh K. 1995. Dissolution of iron oxides by a dithionite- carbonate - oxalate method. *Clays Controlling the Environment*, 317-322.

Varadachari C., Goswami G. és Kunal G. 2006. "Dissolution of Iron Oxides." *Clay Research* no. 25:1-19.