

Improved, high-throughput approach for phosphorus speciation in natural sediments via the SEDEX sequential extraction method

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Appendix 3: Preparation of SEDEX reagents

1 M MgCl₂ (1 L)

Measure out 1 L milli-Q water in a graduated cylinder.

Pour 1 L milli-Q water into a beaker.

Weigh out 203.3 g MgCl₂·6H₂O (FW 203.3) and add it to the milli-Q while stirring with a stir bar.

Stir for half an hour.

Filter the solution through a 0.40-µm PC or GF/F filter.

Adjust to pH 8.0 using NaOH.*

(Total volume required per 0.1-g sample: 55 mL)

Citrate-bicarbonate solution (1 L)

Measure out 1 L milli-Q water in a graduated cylinder.

Pour 1 L milli-Q into a glass beaker.

While stirring, add 88.41 g trisodium citrate (Na₃C₆H₅O₇·2H₂O; FW 294.10) and 84.32 g sodium bicarbonate (NaHCO₃; FW 84.01).

Slowly (to minimize effervescence) add 20 mL concentrated HCl (salts will not dissolve until the pH is brought down).

Check pH and add HCl or NaOH to bring the solution to pH 7.6.*

Recheck pH before beginning step II and adjust again if necessary.*

(Total volume required per 0.1-g sample: 15 mL)

1.0 M NaAc-Hac Buffer (1 L)

Measure out 1 L milli-Q water in a graduated cylinder.

Pour 1 L milli-Q into a glass beaker.

Weigh out 82.03 g sodium acetate (NaC₂H₃O₂; FW 82.03).

Slowly add sodium acetate to the milli-Q while stirring with a stir bar.

Slowly add 235 mL glacial acetic acid.

Adjust to pH 4.0 with glacial acetic acid.*

Recheck the pH before beginning step III and adjust again if necessary.*

(Total volume required per 0.1-g sample: 10 mL)

1 M HCl (1.2 L)

Pour 1100 mL milli-Q water into a beaker.

Add 100 mL concentrated HCl; stir.

(Total volume required per 0.1-g sample: 20 mL)

*Because the pH 7 buffer used to standardize the electrode is a phosphate buffer, it is important to presoak the electrode in a small split of the SEDEX reagent and rinse it well with milli-Q water, after standardization, before immersing in the SEDEX reagent, which requires pH adjustment. This should prevent contamination of your SEDEX reagent with phosphate.)

1 M FeCl₃ (in 0.1 M HCl)

Add milli-Q water to a 250-mL volumetric flask (do not fill completely).

Add 2.08 mL concentrated HCl to the flask.

Fill the flask to 250 mL with milli-Q.

Dissolve 40.55 g ferric chloride (FeCl₃; FW 162.21) in the 250 mL acid.

Filter the solution through a 0.40- μ m PC filter.

100% wt/vol Mg(NO₃)₂

Weigh out 86.30 g magnesium nitrate [Mg(NO₃)₂·6H₂O; FW 256.41]

Dissolve in 50 mL milli-Q water.

Reagent is good as long as it remains clear.

Note: When calculating the volumes of each reagent required, be sure to include enough volume for making blanks and standards for phosphate colorimetric (or other) analysis, which should be made up in the same solutions used for your sediment extraction.