

DISSOLVED INORGANIC PHOSPHATE

New MAGIC 25 method

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Dissolved Inorganic Phosphate has been quantified according to the Rimmelin & Moutin procedure (2005), derived from the initial MAGIC method proposed by Karl & Tien (1992). About 1 L sample was filtered through a 0.6µm polycarbonate membrane using a Nalgene™ filtration apparatus. The sample was then divided into aliquots and poured into 250mL pre-gauged polycarbonate centrifuged bottles. After the addition of 1.75mL of reagent 1, two vigorous homogenizations were successively conducted for 5 min. The bottles were then centrifuged for 10 min at 1500×g with a smooth deceleration curve and the supernatant decanted. The precipitate was then solubilized in 6mL reagent 2 under vigorous homogenization. From the concentrates, two were used to quantify phosphate. After addition of 1mL reagent 3, the concentrates were maintained at room temperature for 15 min to allow reduction of arsenate into arsenite. The final volume of the MAGIC concentrate was 10 mL, which corresponds to a 25 times preconcentration.

The S&P colorimetric procedure was performed by adding 0.2mL of reagent 4 and 0.8mL reagent 5 to the MAGIC concentrate. After a 30 min duration, the absorbance was measured at 880 nm, with a 10 cm-path length cell. In order to fit the volume to 10 mL, 1.2mL DW and 0.8mL of reagent 5 were added. Reagents 3 and 4 were omitted to avoid the blue colour formation. The absorbance was also measured at 880 nm and subtracted from the mean phosphate absorbance. A synthetic reagent blank was determined and subtracted. It was prepared by mixing 1.75mL reagent 1, 7mL reagent 2, 1mL reagent 3, 0.25mL DW (to adjust the volume), 0.2mL reagent 4 and 0.8mL reagent 5. The increase in reagent 2 volume in comparison to the sample treatment allows for the same pH- and $[H^+]/[Mo]$ ratio values as in the MAGIC concentrate (~1.3 and 91, respectively). A $[H^+]/[Mo]$ ratio above 200 inhibits the phosphomolybdic reaction whereas below 60 a self-reduction of the molybdate ion occurs. Seawater was used to fit volume to 40 mL. The standards were then treated as previously described (see DIP according to S&P method).

All reagents were prepared with pro analysis Merck™ Reagent Grade chemicals and with Milli-Q™ high purity demineralised water (DW). All utensils were washed with 10% hydrochloric acid and rinsed three times with DW. All instruments used are commercially available. A SIGMA (4-15) centrifuge allowed centrifuging simultaneously four 300mL samples at 1500×g. A CECIL™ 1011 spectrophotometer (range of measurement from 0.001 to 2; S.D. = 0.001) equipped with a 8 mL-volume 10 cm-path length-cell was used to measure absorbance at room temperature (~20–25 °C). A hand-made support was adapted to position precisely the cylindrical cell.

. *Reagent 1*: 1M NaOH solution (ref. 1.06495.1000; PO₄ < 0.0001%). Storage of this solution is not recommended.

. *Reagent 2*: 0.25M HCl solution (ref. 1.00317.1000). The reagent can be stored at room temperature.

. *Reagent 3*: The sodium-disulfite reducing reagent was prepared daily by dissolving 1.4 g Na₂O₅S₂ (ref. 1.06528.0500) in 10mL DW followed by addition of 5mL of a H₂SO₄ 1.75M (ref. 1.00731.1000) and 10mL of Na₂S₂O₃ solution prepared by diluting 0.7 g Na₂S₂O₃ (ref. 1.06516.0500) in 50mL DW. The reagent can be stored at 4 .C for 24 h.

. *Reagent 4*: The ascorbic acid solution was prepared by dissolving 9 g l(+) C₆H₈O₆ (ref. 1.00127.0250) in 170mL DW. The reagent can be stored at 4 .C for several days.

. *Reagent 5*: The molybdic reagent was prepared by mixing 250mL H₂SO₄ (2.5 M) followed by addition of 75mL of (NH₄)₆Mo₇O₂₄·H₂O, 40 g/L (ref. 1.01182.1000) and 23mL of K(SbO)C₄H₄O₆ · 0.5H₂O, 3 g/L and 52mL of DW. The mixed reagent can be stored at 4 .C for several days.

References:

- Karl, D. M., & G. Tien, 1992: MAGIC: A sensitive and precise method for measuring dissolved phosphorus in aquatic environment. *Limnology and Oceanography*, **37**, 105-116.
- Rimmelin, P. & T. Moutin. 2005. Re-examination of the MAGIC method to determine low orthophosphate concentration in seawater. *Analytica Chimica Acta*, 548(1-2), 174-182.
- Additional references in Rimmelin & Moutin (2005)