

INITIAL WATER HANDLING (WH)**Version 1.1 (updated Nov 2001)**

Aim *To measure the conductivity and pH of samples of precipitation, soil solution and river water and to filter these samples prior to chemical analysis*

Rationale From the moment water samples are gathered they begin to deteriorate as a result of chemical and microbiological processes. Three methods of slowing this deterioration are to be used in ECN - filtration, cold storage, and (for Al and Fe determinations only) acidification. Some of the activities detailed in this Protocol require laboratory training, experience, specialised equipment, and compliance with health and safety requirements (eg diluting concentrated acids); guidance or help from the chemists who will perform the water analyses may also be required. Further background and quality control procedures are given in the documents referred to below.

Method **Initial storage**

Samples must be placed in cold storage, at a temperature between 1°C and 4°C, if the interval between collection and the measurement of conductivity and pH is more than seven hours. They should be returned to cold storage if filtering is not completed on the same day as these measurements. If samples are to be sent to another site for initial handling, they should be sent on the day of collection by the speediest possible method. Samples in transit must be placed in a cool box with pre-frozen cool blocks.

Conductivity measurement

Conductivity must be measured within 36 hours of collection on an unfiltered subsample at a temperature of 25°C according to the method given by HMSO (1978). Results should be expressed to one decimal place (0.1 $\mu\text{S cm}^{-1}$).

pH measurement

pH must be measured within 36 hours of collection on an unfiltered subsample. The same subsample can be used both for conductivity and for pH measurement, but conductivity should be measured first and the subsample should not be returned to the main sample after measurement. The method to be used is given in HMSO (1988) which implies the use of separate glass and reference electrodes. Results should be expressed to two decimal places (0.01 pH unit).

Filtering

Filtering must take place within 60 hours of collection. The method to be used is given in Appendix J. Filtering details should be recorded, using the unique sample codes described in the Protocols (also described in Appendix I of this Protocol). After filtration the samples are to be analysed for dissolved Na^+ , K^+ , Ca^{2+} , Mg^{2+} , Fe^{2+} , Al^{3+} , $\text{NH}_4^+\text{-N}$, Cl^- , $\text{NO}_3^-\text{-N}$, $\text{SO}_4^{2-}\text{-S}$, $\text{PO}_4^{3-}\text{-P}$, alkalinity and dissolved organic carbon (DOC). DOC is not mandatory for precipitation water and can be omitted. Separate documentation is provided for chemical analysis (see AG Protocol) in which the determinands are placed in an order of priority for those occasions on which sample volume is insufficient to allow the measurement of all determinands.

Storage prior to chemical analysis

Storage temperature should be between 1°C and 4°C. Analysis should be completed preferably within 16 days of collection but definitely within 28 days. If samples have to be sent to another location for analysis, the period when

samples are out of cold storage should be minimised, for example by specifying next day delivery and not despatching on a Friday.

Washing bottles

The method for washing bottles is given in Appendix II.

Author

J.K. Adamson

References

HMSO. 1978. *The measurement of electrical conductivity and the laboratory determination of the pH value of natural, treated and waste waters.* London: HMSO.

HMSO. 1988. *The determination of pH in low ionic strength waters.* London: HMSO.

Appendix I. Filtering

Materials

- Filter funnel assembly - most modern filter assemblies are suitable, eg the Millipore Sterifil which holds 47 mm diameter filters (Millipore catalogue no. XX11 047 00)
- Membrane filters (**only**) - Whatman, type WCN, pore size 0.45 μm , obtainable, for example, from BDH, catalogue no. (to fit the above filtering system) 402/0723/04
- Glass fibre filters - Whatman, type GF/C, obtainable, for example, from BDH, catalogue no. (to fit the above filtering system) 234/0856/11
- Vacuum pump - a pump capable of maintaining a vacuum of approximately 0.5 bar

Procedure

All parts of the filter funnel assembly must be thoroughly rinsed with deionised or distilled water before the first sample and between subsequent samples. The filters and the surfaces of the filter assembly which come into contact with the water sample must not be touched by hand. Filters should be moved with forceps and each filter should be used for only one sample. Waters originating from soil solution (SS) and rivers (WQ) may need pre-filtering through a glass fibre filter but these filters require washing with 250 ml of distilled water before use. The filtered water should be poured from the filter assembly into a clean, dry polypropylene bottle and not returned to the bottle used for the unfiltered water. A bottle of 250 ml volume is likely to be appropriate, but the laboratory performing the analyses should be consulted. If the volume of a sample exceeds 100 ml, a subsample of 20 ml should be transferred from each sample to a vial for determination of Al and Fe, and acidified with 20 μl HCl.

All containers should be labelled as follows:

- ECN Site Identification Code (eg T06).

- ECN Measurement Code (eg PC).

- Location Code (eg 01).

- Sampling Date

along with any additional instrument/sampler code necessary to uniquely identify the sample (eg SS sampler code). This unique reference should appear with the final data submitted for inclusion in the ECN database.

Suppliers

BDH (Head Office)

Merck House

Poole

Dorset BH15 1TD, UK

Tel: 01202 664778

Fax: 01202 666541

Millipore (UK) Ltd

The Boulevard

Blackmoor Lane

Watford

Hertfordshire WD1 8YW, UK

Tel: 01923 816375

Fax: 01923 818297

Appendix II. Bottle and vial washing

Procedure

Containers must be washed in a laboratory cleaning agent before being used for the first time, and subsequently at approximately six-monthly intervals, or if subjected to high levels of soiling (eg contamination of rainfall by bird droppings). The cleaning agent should be free of phosphate and hypochlorite. If a laboratory washing machine is available, Decomatic (not Dri-Decon) is suitable, and, if no machine is available, over-night soaking in Decon-90 is suitable. Subsequently, containers will be rinsed four times in tap water and three times in deionised or distilled water. At other times, after use, containers will be rinsed three times in distilled water and retained for use with the same sampler on subsequent occasions.

After washing, containers must be shaken to remove drops of distilled water, dried in warm air in a dust-free environment, and re-capped immediately.

Suppliers

Decon products can be obtained from a number of suppliers, including BDH (see Appendix I).