



**Laboratory Quality**

**Manual: *eriss* Analytical**

**Chemistry Laboratory:**

**Revised 1997**

Peter Cusbert

Chris leGras

Craig Hunt



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## **2.0 AMENDMENT RECORD**

To ensure that laboratory staff are kept informed of current laboratory methods and procedures, an amendment record system is incorporated into this quality manual. A copy of the superseded sheets from this manual will be retained on registry file, JR/02/018

The Laboratory Manager is responsible for the following protocol, which is implemented whenever an alteration or new entry is made in the quality manual:

- (i) the new material is inserted in its proper sequence in the manual;
- (ii) superseded sheets are removed and filed;
- (iii) an amendment record is completed on the sheet placed immediately after the Table of Contents.

A copy of an amendment record sheet is given at Appendix A.

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### 3.0 INTRODUCTION

The Director of the Environment Protection Group of the Commonwealth Environment Portfolio acts as the Supervising Scientist for the Alligator Rivers Region. This organisation has a mandate to assist in the protection of the region from the effects of mining. This Region, which includes Kakadu National Park, is an area of very high environmental value, national profile and political sensitivity.

The Supervising Scientist administers the Office of the Supervising Scientist in Canberra and Darwin and the Environmental Research Institute of the Supervising Scientist (ERISS) which is composed of a number of research groups (Sections) in varied scientific disciplines. Unique among the Sections is the Environmental Chemistry Section which has a dual role:

- (i) to research the chemical pathways and fates of species of interest to the environment; in surface and groundwaters, atmosphere, biota, sediments and soils; and
- (ii) to provide an analytical chemistry service to the other Sections.

For these reasons it is necessary for the ERISS to have an analytical chemistry laboratory which can consistently provide analyses which are reliable and precise. A comprehensive Quality Assurance program is necessary to assess the performance of the laboratory and thereby assure the above objectives.

The Laboratory Manager is responsible for the preparation and maintenance of the laboratory's Quality Manual, which is essential to its quality assurance. The Quality Manual provides the following documentation:

- (i) the policies and operating procedures of the laboratory;



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- (ii) the duties and responsibilities of staff; and
- (iii) the location of equipment, consumables and documentation.

The basic objective of the Manual is to enable new staff to be able to perform their duties in accordance with the requirements of the laboratory, and as a reference resource.

Three copies of the manual are available:

- (i) in the office of the Laboratory Manager;
- (ii) on the shelves on the eastern wall of the laboratory; and
- (iii) in the Registry of the ERISS as file JH-02-043.

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#### **4.0 MANAGEMENT OF THE LABORATORY QUALITY SYSTEM**

##### **4.1 Quality System Review**

A formal review of the quality system of the laboratory is performed annually by the Laboratory Manager usually in June or July. This period also corresponds to a time when the research projects of the institute are reviewed and new projects are proposed. The equipment requirements and staff training needs are assessed with each potential new demand on analytical chemistry services.

The major tool used in assessing the effectiveness of the laboratory's quality system is a thorough review of the results obtained for certified reference materials for the previous year. These are grouped by analytical method, and a table for all analyte determinations is placed at the beginning of each test method in the standard methods manual. From these tables, trends and biases in reference values (even those which conform to certified concentrations) may be evident and thereby, alterations in methods or other procedures may be indicated.

There are other sources of information on the performance of the quality system. These include: NATA assessments; proficiency testing and other round robin experiments; internal and external audits; the alteration of standard methods on advice from other workers in the field. The investigation of client complaints may reveal defects in the quality system. In addition, ineffectiveness in the system may become obvious by chance, without a formal review.

##### **4.2 Alteration of the Quality Manual**

The Laboratory Manager is responsible for alterations to and updating of the Laboratory Quality Manual. This is the only person authorised to make alterations to the manual. All alterations must be authorised.

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## **5.0 DESCRIPTION OF THE LABORATORY AND ITS FUNCTIONS**

### **5.1 Identification**

The Analytical Chemistry Laboratory is located at Jabiru, Northern Territory, about 260 km east from Darwin. The laboratory is a Section within the Environmental Research Institute of the Supervising Scientist (ERISS). The ERISS has a mandate to conduct research to protect the natural environment of the Alligator Rivers Region from the effects of mining.

### **5.2 Range of Testing**

The objectives of this laboratory are detailed in Section 1. It has the capacity to analyse: natural and artificial waters; biota; soils and sediments. The Analytical Chemistry laboratory staff are responsible for analysis and reporting results to clients and for the implementation of research projects. Sampling of the above components of the environment is shared with the client Sections.

Even though this laboratory is part of a research organisation rather than a routine testing facility, it maintains NATA registration so that it may offer authoritative advice when conflict exists between testing bodies. Currently, registration is held for Items 7.66 (Waters) and 7.81 (Constituents of the Environment) as listed below:

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The procedure and requirements for updating portions of this manual are as follows:

- (i) a complete page of the manual must be replaced;
- (ii) the entry must contain: the name of the laboratory; section, sheet and issue number; the title of the section; the issue date and an authorised signature;
- (iii) the new page must be inserted in the appropriate place;
- (iv) superseded pages must be removed and placed on the laboratory's quality manual file; and
- (v) an amendment record is to be completed as detailed in Section 2 of this Manual.

All copies of the Manual are so amended by the Laboratory Manager.

Every two years or at least after an assessment from NATA inspectors the laboratory quality manual is published in updated form as an internal report.

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Item 7.66 - Waters

**Section 71 Sampling**

Sampling of surface waters, bore waters and pond waters (APHA 105)

**Section 99 Other Waters (Analysis of Uranium mine/mill waters)**

APHA: methods total alkalinity; conductivity; pH value; temperature; turbidity; dissolved oxygen; calcium (flame aas); magnesium (flame aas); manganese (flame and graphite furnace aas); zinc (flame and graphite furnace aas); copper (graphite furnace aas); lead (graphite furnace aas); ammonium-N (ion chromatography); filtrable available phosphorus (spectrophotometric)

Modified APHA methods: chloride; nitrate-N; sulphate (all ion chromatographic).

Australian Standard method: uranium (spectrophotometric)

In-house methods: sodium; potassium; ammonium-N; calcium; magnesium (all ion chromatographic); uranium (scintrex fluorometric).

Item 7.81 Constituents of the Environment

**Section 11 Waters other than Saline.**

APHA methods: total alkalinity; conductivity; pH value; temperature; turbidity; dissolved oxygen; calcium (flame aas); magnesium (flame aas); manganese (flame and graphite furnace aas); zinc (flame and graphite furnace aas); copper (graphite furnace aas); lead (graphite furnace aas); filtrable available phosphorus (spectrophotometric).

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Modified APHA methods: chloride; nitrate-N; sulphate (all ion chromatographic).

Australian Standard method: uranium (spectrophotometric).

In-house methods: sodium; potassium; ammonium-N; calcium; magnesium(all ion chromatographic); uranium (Scintrex fluorometric).

Complete details of NATA registration are contained in volume 1 of the Standard Methods Manual, Section 3, and also in Appendix B of this manual.

Complete details of all tests undertaken by this laboratory are listed in Section 9 of this manual.

### **5.3 Organisational Structure**

An organisational chart of the research institute is presented as Appendix C. The research institute is divided into three branches:

- (i) Corporate Services.
- (ii) Wetland Management.
- (iii) Impact of Mining (including the Analytical Chemistry Laboratory).

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In addition there is an administrative support section and a mechanical workshop. Each research section is headed by a research scientist who supervises experimental officers (qualified scientists), technical officers and technical assistants. Each research scientist reports to the Director of the Institute. The Director reports to the Supervising Scientist, who is based at Canberra, ACT.

The Senior Research Scientist in Environmental Chemistry is responsible for the research projects of the Section. Currently, this function is completely separate from the Analytical Chemistry Laboratory.

The Laboratory Manager, a senior professional officer, directs the everyday operation of the laboratory, such as work flow, calibration, maintenance, ordering of supplies and administrative tasks. The Laboratory Manager is responsible for the issue of reports, some checking and analyses as required, and also directs method development.

The number and designation of other staff varies. Currently there is a technical officer who is concerned with chemical analysis, purchasing consumable, calibration, maintenance, sample preparation and sample dispatch for the Inductively-Coupled Plasma Mass Spectrometry facility in Sydney (Section 15).



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## **6.0 STAFF**

### **6.1 Staff Responsibilities**

Duty statements, job descriptions and selection criteria for all staff within the laboratory are held by the Senior Administration Officer of ERISS at Jabiru. These documents specify duties, responsibilities and the supervisory hierarchy.

In summary, all staff are expected to perform all analytical and associated tasks with due competence and professionalism.

All tasks are performed without undue pressure that might influence their technical judgement.

### **6.2 Training and Development**

#### **6.2.1 Training**

The objective of training within the ERISS and specifically within this laboratory is: to facilitate the efficient discharge of the responsibilities of the laboratory and to increase the skills of laboratory staff and thereby increase work satisfaction and morale. Training of Analytical Chemistry Laboratory staff takes three forms: (i) training of new and existing staff within the laboratory in the procedures of the laboratory and in analytical methods; (ii) in-house ERISS courses which are usually of a non-technical nature, such as in scientific writing, supervision, and administrative tasks; (iii) and external courses and conferences which may either be highly technical, non technical or administrative courses which are specifically vocational (such as those offered by NATA).

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External courses from an approved institution, if they lead to a formal qualification, may be eligible for assistance with compulsory fees and necessary leave, through the Commonwealth Government Studies Assistance Scheme.

The most important form of staff training is that conducted within the laboratory on how to perform test methods. The general training methods comprises six steps:

- (i) a general explanation of the method by the trainer;
- (ii) a full-speed demonstration of the method by the trainer;
- (iii) a step-by-step demonstration by the trainer, with a full explanation of each portion of the method, and encouragement of questions by the trainee;
- (iv) a step-by-step performance by the trainee, with immediate guidance by the trainer should difficulty be encountered. The method is repeated by the trainee for all possible variations of it;
- (v) a straight-through performance by the trainee, with the trainer supervising the execution; and
- (vi) an assessment of the training by the determination by the trainee of replicate samples of at least one certified reference material.

In cases where subsequent problems are encountered, the procedures described in Section 13 are initiated.

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### 6.2.2 Staff Appraisal

The annual appraisal of staff is the policy of the Public Service Commission. The ERISS therefore has an obligation to develop and maintain a Staff Appraisal Scheme.

The ERISS Staff Appraisal Scheme takes the form of a documented interview between each staff member and his/her immediate supervisor. The elements of this interview are:

- (i) a review of the year's work, with emphasis on the performance of the subordinate from both the staff member's and supervisor's perspective;
- (ii) a discussion of how the skills of the subordinate can best be deployed in the forthcoming year. A preview of the anticipated work would be appropriate here;
- (iii) an honest airing of any problems or grievances which may have become apparent during the year; and
- (iv) a dialogue concerning staff development options for the coming year: staff development may include formal or informal courses of a technical or non-technical nature; the development of expertise in different test methods and perhaps altered supervisory duties.

A copy of the record of interview is reviewed by the overall supervisor of the participants in the staff appraisal session and eventually is placed on the personal file of the staff member. Complete confidentiality is assured under Public Service regulations.

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The objective of the Staff Appraisal Scheme is to improve the overall efficiency of the laboratory by attention to the professional development and technical, and interpersonal requirements of staff in a formal but supportive interview context. It is considered imperative that the morale and self-esteem of all staff be preserved and enhanced by the staff appraisal process.

#### 6.2.3 Maintenance of Personnel Records

Personal files and Standard Personnel Records are maintained by the Department of Administrative Services Personnel Section in Darwin, on behalf of the ERISS. A copy of the personal file of each staff member is kept at ERISS headquarters at Jabiru. Staff can inspect their personal file at any time.

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## **7.0 EQUIPMENT AND CONSUMABLES**

### **7.1 Relevant Documentation**

In this Section the following files and registers will be cited. They are all located on the eastern wall of the General Chemistry Laboratory and are contained in heavy-duty, loose-leaf ring binders.

- (i) Standard methods manual volume 1;
- (ii) consumable items register and procurement schedule;
- (iii) order form books;
- (iv) register of orders placed;
- (v) chemical stocks register;
- (vi) calibration and maintenance records file; and
- (vii) calibration procedures files.

### **7.2 Procurement of Goods and Services**

No distinction is made between durable equipment, consumable goods and services for the purposes of procurement. Ordering is governed by the relevant policy and procedures regulations of the ERISS, a copy of which are held at the front of the consumable items register and procurement schedule. Brief details of these regulations are as follows:

- (i) for a total value less than \$500, one oral or written quote;
- (ii) for a total value of \$500-\$2000, at least three oral or written quotes;
- (iii) for a total value of \$2000-\$30000, at least three written quotes; and
- (iv) for a total value greater than \$30000, special tendering arrangements through the Department of Administrative Services.

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#### 7.2.1 The Ordering Procedure

A delegated staff member is issued with a credit card which is to be used for all purchasing, except where:

- the purchase is greater than \$2000 or
- the vendor does not have the relevant processing facilities. In this case a dummy order is printed (see below). Transactions are reconciled monthly by the cardholder.

All orders are made using the DSV (dummy sales voucher) program in Paradox for Windows. Registered credit card holders have access to this program. When the relevant quotes and details have been obtained, the information is electronically recorded in the program and printed out. This paper copy is signed by the cardholder and countersigned by the laboratory manager, then faxed or posted to the vendor. When the goods arrive, the date is recorded on the electronic copy and on the delivery docket. When the invoice arrives, it is matched and filed with a copy of the order. A reconciliation statement is issued monthly by the bank and this is balanced and returned to the administrative section with the original invoice for each order. A copy of the reconciliation, including a copy of each invoice, is made and filed for future reference in the laboratory. The original order is also filed with any relevant delivery docket.

Where the order exceeds \$2000, the approval of the Senior Administrative Officer is required and a credit card from Administration, with a higher transaction limit, is used.

Where credit card facilities are not available from the vendor, the 'purchase order' option in the DSV program is used and the DSV number becomes the order number. The order is printed out and sent to the vendor, with a copy given to the ERISS purchasing officer and another filed for laboratory records.

All consumable items used on a regular basis are specified in a consumable items register and procurement schedule. Once a year, usually in August, a stocktake of all such items is undertaken and recorded in this register, and on the basis of the previous year's consumption, ordering action is commenced and recorded in the register. The schedule is reviewed six months later to account for the possibility of abnormally high consumption. If the consumable item is a chemical, details of quantity, location within the Laboratory and hazard assessment are recorded in the chemical stocks register. These tasks are currently the duty of the technical officer.

All purchases of durable equipment are listed on a register which comprises section 7.3 of the Laboratory Quality Manual.

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### **7.3 Equipment Inventory**

#### **7.3.1 Graphite Furnace Atomic Absorption Spectrometry**

- (a) Perkin-Elmer model HGA 700 with power and gas service unit  
This module is used as an accessory with the Perkin Elmer model 4100 atomic absorption spectrometer.

Serial number: 8137.

- (b) Perkin-Elmer model AS-70 autosampler

Serial number: 3310.

- (c) Grants water cooler for use with graphite furnace.

Serial number:

These components are collectively used to determine trace metal concentrations in natural and artificial waters, and in biota digest solutions in the nanogram and microgram per litre ranges.

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This equipment is serviced on a contract basis by Perkin-Elmer Pty Ltd., 301 Coronation Drive, Milton. Qld 4064.

### 7.3.2 Flame Atomic Absorption Spectrometry

- (a) Perkin Elmer model 4100 microprocessor-controlled spectrophotometer with simultaneous background correction and double-beam optics.

Serial number:

- (b) Perkin Elmer model AS90 autosampler.

Serial number: 2011185.

- (c) Total Peripherals 85 computer model 386 with disc drive and keyboard.

Serial number: TP1476594.

- (d) Total Peripheral Model TP 250P VGA colour monitor.

Serial number: A30 - 21901382.

- (e) Atlas Copco Automan air compressor

Serial number: 52791374.

These components are collectively used to determine trace metal and major cation concentrations in natural and artificial waters, and in biota in the microgram and milligram per litre ranges. All results are retained on a floppy disc.

This equipment is serviced on a contract basis by Perkin-Elmer Pty Ltd., 301 Coronation Drive, Milton, Qld 4064.



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### 7.3.3 High Performance Liquid Chromatography

- (a) IBM Computer with Waters Maxima software.

Serial number: A160316.

- (b) 14 " VGA colour monitor.

Serial number: A30-21901382

- (c) Digital printer model LA50

Serial number: TC 24292B.

- (d) Waters Autosampler model 717 Plus

Serial number: not known

- (e) Waters programmable solvent delivery module model 590.

Serial number: 590-003281.

- (f) Waters HPLC pump model 510 (2 units).

Serial numbers: 510-123505; 510-123540.

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(g) Waters conductivity detector model 430 (2 units).

Serial numbers: 103; 607.

(h) Waters differential refractometer model R401.

Serial number: 401-007135.

(i) Waters LC spectrophotometer model 481.

Serial number: 481-003345.

(j) Waters Data Module model M730.

Serial number: 730-007056.

(k) Waters System Controller model 721.

Serial number: 721-003785.

(l) Waters automated switching valve.

Serial number: 1108.

(m) Waters system interface module (2 units).

Serial numbers: SIM 1173; SIM 7939.

This equipment is used as an ion chromatograph to determine the concentrations of  $\text{Na}^+$ ,  $\text{NH}_4^+$ ,  $\text{K}^+$ ,  $\text{Cl}^-$ ,  $\text{NO}_3^-$  and  $\text{SO}_4^{2-}$  in natural and artificial waters in the microgram and milligram per litre ranges.

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The equipment is serviced by Millipore-Waters Pty Ltd, 8 Durong Street, Newstead, Qld 4006 on an annual basis.

#### 7.3.4 Time-decay Fluorescence Spectrometry

- (a) Scintrex UA-3 uranium analyser.

Serial number: 8001193.

- (b) Hewlett-packard programmable calculator model HP41CV.

Serial number: 2511542300.

- (c) Hewlett-Packard data acquisition control unit model HP3421A.

Serial number: 2247A00803.

- (d) Hewlett-Packard thermal printer model 821262A.

Serial number: 2306587394.

This equipment is used for the analysis of uranium in natural and artificial waters in the nanogram and microgram per litre concentration ranges.

This equipment is serviced by an on-site electrical technician.

#### 7.3.5 Electronic Spectrometry

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- (a) Stansens visible spectrophotometer model 690 with single-beam optics.

Serial number: 011210.

- (b) Perkin Elmer Lambda 2 uv-visible spectrophotometer

Serial number: 2847

These spectrophotometers are used for the colorimetric determination of; U, Al, Fe,  $\text{PO}_4^{3-}$ ,  $\text{SO}_4^{2-}$ ,  $\text{Cl}^-$ , and  $\text{NH}_4^+$  in the microgram and milligram per litre concentration ranges.

These instruments are serviced by an on-site electronics technician.

#### 7.3.6 Electrometric Methods

- (a) Radiometer ion-scanning system comprising TTA80-IS titration assembly and REC80 Servograph.

Serial number: 48R27NO73 and 5912N12 respectively.

This equipment is used for the determination by potentiometric stripping analysis of Cu, Pb and Zn in the nanogram and microgram per litre concentration ranges.

- (b) EG&G Princeton Applied Research polarographic analyser model 384 with digital plotter and single mercury drop electrode model 303.

Serial number: 05119 (analyser) and 09162 (plotter).

This equipment is used for the polarographic determination of electroactive metals in the nanogram per litre concentration range.

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- (c) Metrohm titroprocessor model 682.

Serial number: 1g6/194.

- (d) Metrohm dosimat model 665 with stirrer model E649.

Serial number: 461405.

This equipment is used for microprocessor-controlled automatic titrations in the milligram per litre concentration range. It is chiefly used for the determination of alkalinity.

- (e) Metrohm sample changer model 673.

Serial number: OM7/128.

- (f) Metrohm control unit model 664.

Serial number: OM8/105.

- (g) Orion research digital analyser model 720A.

Serial number: 006064.

This equipment is used to determine the concentration of ions in the microgram and milligram per litre concentration ranges. It is chiefly used with a glass combination electrode to determine pH.

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- (h) Metrohm Herisau conductometer model E518.

Serial number: 008/00677.

This equipment is used to determine the conductivity of natural and artificial waters in the microsiemen and millisiemen per centimeter ranges.

All electrometric equipment is serviced by an on-site electronics technician.

#### 7.3.7 Carbon Analysis

- (a) O.I. Corporation carbon analyser model 700.

Serial number: not known

- (b) O.I. Corporation Autosampling module.

Serial number: not known.

- (c) O.I. Corporation Purging and sealing unit model 104554

Serial number: a420777196

- (d) NEC Powermate SX/vi desktop computer with 14" colour monitor.

Serial number: 12000352

- (e) OKI Microline 184 Turbo printer

Serial number: 211a0002867

- (f) Nitrogen Generator, Peak Scientific model No. NG1000A

Serial No. AT -01 -13

This equipment is used to determine total carbon and inorganic carbon by direct measurement and organic carbon by difference (T-I) in the milligram per litre concentration range.

This equipment is serviced by a contract electronics technician - Jeff Klein of Darwin.

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### 7.3.8 Nephelometry

- (a) Hach Ratio/XR turbidimeter model 43900.

Serial number: 881201147.

This equipment is used to measure the turbidity of natural and artificial waters in the range 0-2000 nephelometric turbidity units. It is serviced a contract electronics technician - Jeff Klein of Darwin.

### 7.3.9 Gravimetry

- (a) Mettler electronic balance model AT261 with Mettler model GA45 printer.

Serial number: M87696

This balance measures mass in the 0.1 microgram to 200 gram range. It is serviced by FSE Scientific Unit 3, 151 Granite St., Queensland 4014.

- (b) LFE Corporation low temperature plasma asher model LTA-504 with timer.

Serial number: 93592.

This equipment is used to oxidise organic matter in samples at low temperature to leave inorganic residue.

- (c) Labmaster air oven, small.

Serial number: 118068.

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- (d) Napco vacuum oven model 5831.

Serial number: 11-87-M05484-7.

These ovens are used to dry samples to constant weight prior to weighing.

The equipment for gravimetric determinations is chiefly used to determine the concentration of suspended solids in natural and artificial waters; total and inorganic solids directly and organic solids by difference (T-I), in the milligram per litre concentration range. Except for the Sartorius balance the equipment is serviced by an on-site electronics technician.

#### 7.3.10 *Microwave sample preparation system*

- (a) Questron model Q wave-1000 microwave  
Serial number: 01279

- (b) Epson quiet colour printer model LX-300  
Serial number: IQEE010734

- (c) Ipex Notebook Computer model NB8620  
Serial number: not known

#### 7.3.11 Other Major Items of Equipment

- (a) Milli-Q/Milli RO-60 water purification system with water softener.

Serial number: not known.

A source of high purity water.

- (b) Gilson electronic dilutor model 401.

Serial number: 72286.

- (c) Standardised Protection laminar-flow work station.

Serial number: DFB 424/12.

Used for sample handling under Class 100 conditions.



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- (e) Mettler electronic top-loading balance model BB2400.

Serial number: not known.

- (f) Hewlett-Packard "quiet jet plus" dot matrix printer model 2227A.

Serial number: 2910S25251.

- (g) Mettler top-loading electronic balance model PE 360.

Serial number: C59148.

- (h) Mettler infra-red dryer model LP-15.

Serial number: C89755.

- (i) Analite stainless steel analytical weights (calibrated 0.01g-2kg).

These items of equipment are used in support of the analytical program of the Laboratory. They are serviced as required by a contractor Klein Electronics , Darwin

#### 7.3.12 Minor Items of Equipment

All items of equipment which are valued at less than \$2000.00 are kept on an assets register system which may be accessed through the Corporate Services Group. A copy is currently held in the Consumable Items Folder which is located in the Laboratory Managers office.

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#### **7.4 Laboratory Equipment Assurance Program**

##### **7.4.1 Commission of New Equipment**

The first step in commissioning new equipment is for the on-site electronics technician to examine the operation and electrical safety of the device.

After this check is complete the equipment is tested by using it to determine analytes in Standard Reference Materials which have similar composition and matrices to samples. Where possible samples are analysed using at least one other independent method (e.g. aas and ion chromatography; alkalinity and inorganic carbon; potentiometric stripping analysis and aas). Typically, NBS, EPA and IAEA reference materials are used.

##### **7.4.2 Calibration**

A calibration schedule is included as Section 7 of the Laboratory standard methods manual volume 1. This schedule includes details of frequency of calibration, periodic checks and maintenance of the equipment.

Completed schedules and worksheets for calibration and maintenance for the past three years are stored in a folder in the Analytical Chemistry Laboratory foyer. Records of older calibration and maintenance checklists are stored in Registry file No. JR-06-091.

The frequency requirements for calibration and the nature of the calibration required is contained in the booklet "Supplementary Requirements for Registration for Chemical Testing" published by NATA in 1993. Specific calibration procedures are contained in the files "Calibration Procedures" in the Analytical Chemistry Laboratory. These files contain the relevant Australian and British Standards or other authorised methods.

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#### 7.4.3 Operational Checks

The performance of an instrument is assessed each time it is used to analyse samples. Three performance criteria are routinely applied.

- (i) Conformity of the results for reference materials to their accepted values.
- (ii) Repeatability checks on at least one sample in ten.
- (iii) Conformity of standard solutions to previously observed values (e.g. absorbance, integrated area, etc).

In addition, where data are available, analytical results using one method are compared with those using other methods.

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| <b>THE TESTING ENVIRONMENT</b>               | <b>Authorised by:</b>       |

## **8.0 THE TESTING ENVIRONMENT**

### **8.1 Specifications and Design**

The Analytical Chemistry Laboratory occupies four buildings. Building (i) is a demountable structure obtained from the Australian Army in 1978. These buildings are used as follows:

- (i) the main chemistry laboratory occupies a large demountable building; the building covers approximately 270 square metres and is divided into seven sections: an office reception area; a general laboratory; a chemical store; a general instrument laboratory; a room which houses the HPLC equipment; a clean preparation room where samples are treated and solutions are prepared, and a clean room (clean instrument room); and
- (ii) the sample preparation laboratory is a permanent building shared by all sections within the Institute. The Analytical Chemistry Laboratory occupies approximately 20 square metres of this building, comprising one room: the Digestion Room contains equipment for the digestion of biological samples.
- (iii) and (iv) Water samples and biological tissue are stored in a Cooler Room at 4°C and a Freezer Room at -12°C in separate units within the compound..

All these buildings are airconditioned, individual room units for building (i) and large ducted units for the other building. This minimises temperature-related drift of instruments and provides a comfortable working environment.

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Plans of the buildings and provision of utilities to them are contained in a special cabinet in the Administrative Building. Plans and other details for the main chemistry laboratory (i above) are contained in File JB-03-10, stored in the Institute Registry.

## **8.2 Special Environment Considerations**

The main laboratory building is equipped to provide an increasingly contaminant-free environment as one progresses from the general laboratory to the clean preparation room to the clean instrument room. The main door to the laboratory is protected by two tacky mats: a cloth-based strip (Takmat) one outside the door and a plastic strip ("First-Step") one inside the door. The door to the clean preparation laboratory has the same system: a Takmat outside and a "First-Step" mat inside. The entrance to the instrument analysis room contains another "First-Step" mat. Therefore, to proceed from outside the laboratory to the clean instrument room, a total of five tacky mats must be traversed.

The clean preparation room and clean instrument room may only be entered when wearing plastic overshoes. The clean instrument room is further protected by the mandatory wearing of a dacron laboratory coat, the provision of an independent air conditioner equipped with a 0.4µm filter, and special specifications for cleaning the room.

For those analyses where contamination may be most critical, a laminar-flow work station, detailed in Section 7 of this manual, is used. Clean-room gloves are used in conjunction with this work station.

The graphite furnace spectrometry, Scintrex fluorimetry and electrochemistry equipment is housed in the clean instrument room.

## **8.3 Access**

The access to the laboratory by other staff or visitors is restricted at all times. Permission to work in the laboratory requires prior clearance from the Laboratory Manager.

Chemicals may be borrowed from the laboratory provided that the details are recorded in a book at the chemical store. The chemicals are issued by the Technical Officer.

Poisonous chemicals can only be issued with the permission of the Laboratory Manager or his supervisor.

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| <b>TEST METHODS</b>                          | <b>Authorised by:</b>       |

## 9.0 TEST METHODS

### 9.1 Current Test Methods

#### 9.1.1 Determination of Analytes in Water

##### (a) General parameters

| Method Number | Method   |
|---------------|--|
| 1             | <b>Alkalinity; APHA 2320B</b>  |
| 2             | Acidity, grams titrimetric   |
| 3             | Acidity/alkalinity of rainwater; WMO #491  |
| 4             | Carbon, total and dissolved; APHA 5310D  |
| 5             | <b>Conductivity; APHA 2510D</b>  |
| 6             | Dissolved oxygen; APHA 4500-OG   |
| 7             | Dissolved oxygen; APHA 4500-OG   |
| 8             | <b>pH; APHA 4500-H<sup>+</sup></b>   |
| 9             | pH of rainwater; WMO #491  |
| 10            | <b>Temperature; APHA 2550B</b>   |
| 11            | Total hardness; APHA 2340C   |
| 12            | Total suspended solids; APHA 2540D   |
| 13            | Total dissolved solids; APHA A2540C  |
| 14            | Turbidity; APHA 2130B  |
| 15            | Reduction/Oxidation potential (Section 1.7.7. Redox Potential in Water Analysis, W. Fresenius, K.E. Quentin and W. Schneider (Eds.) Springer-Verlag pp 39-42 (1988). |
| 16            | Inorganic and Organic Residue in waters.   |
| 17            | Free cyanide, preparation of test sample by isothermal diffusion.  |

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(b) Spectrophotometric

| Method Number | Method  |
|---------------|---|
| 20            | Aluminium; catechol violet  |
| 21            | Ammonia in rainwater; indophenol blue   |
| 22            | Ammonia; APHA 4500NH3D  |
| 23            | Chloride; modified thiocyanate  |
| 24            | Chloride in rainwater; thiocyanate  |
| 26            | Chlorophyll and Pheophytin; APHA 10300C   |
| 27            | Iron; APHA 3500FeD  |
| 28            | Nitrate in rainwater; hydrazine reduction   |
| 29            | Filterable available phosphorus; APHA 4500PE  |
| 30            | Silica; APHA 4500SD   |
| 31            | Sulphate in rainwater; PAN method   |
| 32            | Sulphate; APHA 4500SO4E   |
| 33            | Sulphate; modified turbidimetric  |
| 34            | Sulphate; 2-aminoperimidine   |
| 35            | Uranium; AS 2723-1984   |
| 36            | Free cyanide, barbituric acid; APHA 4500 CNE  |
| 37            | Total Phosphorus, Acid Digestion and Molybdenum Blue Spectrophotometric method APHA 4500PE. |
| 38            | Total Kjeldahl nitrogen, acid digestion and alkaline distillation method.                   |

(c) Flame Atomic Absorption Spectrometric

| Method Number | Method                |
|---------------|-----------------------|
| 50            | Aluminium; APHA 3111D |
| 51            | Calcium; APHA 3111B   |
| 52            | Copper; APHA 3111B    |
| 53            | Iron; APHA 3111B      |
| 54            | Lithium; APHA 3111B   |
| 55            | Lead; APHA 3111B      |

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|-----------|------------------------------|
| <b>56</b> | <b>Magnesium; APHA 3111B</b> |
| <b>57</b> | <b>Manganese; APHA 3111B</b> |
| <b>58</b> | <b>Potassium; APHA 3111B</b> |
| <b>59</b> | <b>Sodium; APHA 3111B</b>    |
| <b>60</b> | <b>Zinc; APHA 3111B</b>      |
| <b>61</b> | <b>Cadmium APHA 3111B</b>    |
| <b>62</b> | <b>Cobalt APHA 3111B</b>     |

(d) **Graphite Furnace Atomic Absorption Spectrometric**

| <b>Method Number</b> | <b>Method</b>   |
|----------------------|---|
| <b>80</b>            | <b>Operation of Perkin-Elmer 5000 AAS/HGA500/AS40</b> |
| <b>81</b>            | <b>Pre-sample run trials; graphite furnace aas</b>    |
| <b>82</b>            | <b>Absorption spectrometry</b>                        |
| <b>83</b>            | <b>Aluminium; US EPA 202.2</b>                        |
| <b>84</b>            | <b>Barium; US EPA 208.1</b>                           |
| <b>85</b>            | <b>Cadmium; US EPA 213.2</b>                          |
| <b>86</b>            | <b>Chromium; US EPA 218.2</b>                         |
| <b>87</b>            | <b>Copper; APHA 3113B</b>                             |
| <b>88</b>            | <b>Iron; US EPA 236.2</b>                             |
| <b>89</b>            | <b>Lead; APHA 3113B</b>                               |
| <b>90</b>            | <b>Manganese; APHA 3113B</b>                          |
| <b>91</b>            | <b>Nickel; US EPA 249.2</b>                           |
| <b>92</b>            | <b>Strontium</b>                                      |
| <b>93</b>            | <b>Silver; US EPA 272.1</b>                           |
| <b>94</b>            | <b>Zinc; APHA A3113B</b>                              |

(e) **Time-decay Fluorimetric**

| <b>Method Number</b> | <b>Method</b>                         |
|----------------------|---------------------------------------|
| <b>100</b>           | <b>Uranium; Scintrex fluorimetric</b> |



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(f) High Performance Liquid Chromatography

| Method Number | Method                               |
|---------------|--------------------------------------|
| 105           | Ammonia                              |
| 106           | Cations                              |
| 107           | Anions; APHA 4110B                   |
| 108           | Anions, Cations; EDTA solvent system |

(g) Ion Selective Electrode Potentiometry

| Method Number | Method                |
|---------------|-----------------------|
| 120           | Chloride              |
| 121           | Copper                |
| 122           | Fluoride; APHA 4500FC |

(h) Electrochemical Techniques

| Method Number | Method   |
|---------------|--|
| 130           | Evaluation of test solution; potentiometric stripping analysis |
| 131           | Copper, total and labile; potentiometric stripping analysis    |
| 132           | Lead, total and labile; potentiometric stripping analysis      |
| 133           | Zinc, total and labile; potentiometric stripping analysis      |
| 134           | Manganese, labile; anodic stripping voltammetry                |

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(i) **Sample Preparation**

| <b>Method Number</b> | <b>Method</b> |
|----------------------|---------------|
|----------------------|---------------|

|     |  |
|-----|--|
| 190 | Field sampling methods                           |
| 191 | Preparation of plastic bottles                   |
| 192 | <b>Sampling and filtration of natural waters</b> |

9.1.2 **Determination of Analytes in Biological Tissues**

| <b>Method Number</b> | <b>Method</b> |
|----------------------|---------------|
|----------------------|---------------|

|     |  |
|-----|--|
| 200 | Insects; Uranium; scintrex fluorometric  |
| 201 | Insects; manganese; flame aas            |
| 202 | Insects; Copper; flame aas               |
| 203 | Insects; Zinc; flame aas                 |
| 204 | Insects; Lead; graphite furnace aas      |
| 205 | Insects; Cadmium; graphite furnace aas   |
| 206 | Mussels; Sodium; flame aas               |
| 207 | Mussels; Potassium; flame aas            |
| 208 | Mussels; Calcium; flame aas              |
| 209 | Mussels; Magnesium; flame aas            |
| 210 | Mussels; Barium; flame aas               |
| 211 | Mussels; Strontium; flame aas            |
| 212 | Mussels; Copper; flame aas               |
| 213 | Mussels; Zinc; flame aas                 |
| 214 | Mussels; Manganese; flame aas            |
| 215 | Mussels; Lead; graphite furnace aas      |
| 216 | Mussels; Cadmium; graphite furnace aas   |
| 217 | Mussels; Aluminium; graphite furnace aas |
| 218 | Bird Feathers; Manganese; flame aas      |

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9.1.2 Determination of Analytes in Biological Tissues

| <b>Method Number</b> | <b>Method</b>                                  |
|----------------------|--|
| 219                  | Bird Feathers; Zinc; flame aas                 |
| 220                  | Bird Feathers; Copper; graphite furnace aas    |
| 221                  | Bird Feathers; Lead; graphite furnace aas      |
| 222                  | Bird Feathers; Cadmium; graphite furnace aas   |
| 223                  | Bird Feathers; Chromium; graphite furnace aas  |
| 224                  | Bird Feathers; Uranium; scintrex fluorimetric  |
| 225                  | Bird Feathers; Mercury; flameless aas          |
| 226                  | Bird Feathers; Arsenic; hydride evolution aas  |
| 227                  | Bird Feathers; Selenium; hydride evolution aas |
| 228                  | Bird Liver; Manganese; flame aas               |
| 229                  | Bird Liver; Zinc; flame aas                    |
| 230                  | Bird Liver; Copper; flame aas                  |
| 231                  | Bird Liver; Lead; graphite furnace aas         |
| 232                  | Bird Liver; Cadmium; graphite furnace aas      |
| 233                  | Bird Liver; Nickel; graphite furnace aas       |
| 234                  | Bird Liver; Chromium; graphite furnace aas     |
| 235                  | Bird Liver; Uranium; scintrex fluorometric     |
| 236                  | Bird Liver; Mercury; flameless aas             |
| 237                  | Bird Liver; Arsenic; hydride generation aas    |
| 238                  | Bird Liver; Selenium; hydride generation aas   |
| 239                  | Bird Muscle; Manganese; flame aas              |
| 240                  | Bird Muscle; Zinc; flame aas                   |
| 241                  | Bird Muscle; Copper; graphite furnace aas      |
| 242                  | Bird Muscle; Lead; graphite furnace aas        |
| 243                  | Bird Muscle; Cadmium; graphite furnace aas     |
| 244                  | Bird Muscle; Nickel; graphite furnace aas      |
| 245                  | Bird Muscle; Chromium graphite furnace aas     |
| 246                  | Bird Muscle; Uranium; graphite furnace aas     |
| 247                  | Bird Muscle; Mercury; flameless aas            |
| 248                  | Bird Muscle; Arsenic; hydride generation aas   |
| 249                  | Bird Muscle; Selenium; hydride generation aas  |
| 301                  | Plant tissue; Sodium; flame aas                |
| 302                  | Plant tissue; Calcium and Magnesium; flame aas |
| 303                  | Plant tissue; Iron and Manganese; flame aas    |

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**9.1.2 Determination of Analytes in Biological Tissues**

| <b>Method Number</b> | <b>Method</b>   |
|----------------------|---|
| 304                  |   |
| 305                  | Plant tissue; Potassium; flame aas  |
| 306                  | Plant tissue: Total Kjeldahl Nitrogen; acid digestion, alkaline distillation method and HPLC method   |
| 307                  | Plant tissue; Total Phosphorus; Kjeldahl acid digestion and molybdenum blue spectrophotometric method |
| 308                  | Plant tissue; Phosphorus; acid digestion and molybdenum blue spectrophotometric method                |
| 309                  | Plant tissue; Boron, Aluminium, Magnesium, Calcium, Manganese, Iron; preparation for ICP-AES          |
| 310                  | Plant tissue; Cobalt, Zinc, Copper, Selenium, Molybdenum, Lead, Uranium; preparation for ICP-MS       |

**9.1.3 Determination of Analytes in Soils, Interstitial Water and Sediment**

| <b>Method Number</b> | <b>Method</b>  |
|----------------------|--|
| 600                  | Soil; total sulphate; gravimetric  |
| 601                  | Soil; water-soluble sulphate; gravimetric  |
| 602                  | Soil; sodium; flame aas  |
| 603                  | Soil; potassium; flame aas   |
| 604                  | Soil; calcium; flame aas   |
| 605                  | Soil; magnesium; flame aas   |
| 606                  | Soil; aluminium; flame aas   |
| 607                  | Soil; iron; flame aas  |
| 608                  | Soil; manganese; flame aas   |
| 609                  | Soil; barium; flame aas  |
| 610                  | Soil; free moisture, gravimetric   |
| 611                  | Soil; total Kjeldahl nitrogen; acid digestion and alkaline distillation method       |
| 612                  | Soil; total phosphorus; acid digestion and molybdenum blue spectrophotometric method |
| 613                  | Soil; available phosphorus; sodium bicarbonate extraction method                     |
| 614                  | Soil; water soluble ions; extraction method  |
| 615                  | Soil; exchangeable cations; ammonium acetate extraction method                       |

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9.1.3      **Determination of Analytes in Soils, Interstitial Water and Sediment**

| <b>Method Number</b> | <b>Method</b>  |
|----------------------|--|
| 616                  | Soil; boron; azomethine-H spectrophotometric method  |
| 617                  | Soil; available metals; DTPA extraction method   |
| 618                  | Soil; organic carbon; reflux distillation and spectrophotometric method                              |
| 619                  | Soil; available potassium, extraction and flame atomic absorption spectrophotometric method.         |
| 620                  | Soil; extraction of iron, aluminium and manganese oxides; buffered sodium citrate dithionite reagent |
| 621                  | Soil; calcium phosphate extractable sulphur; icp-aes   |
| 624                  | Soil; aluminium in dithionite soil extract; acid digestion catechol violet method                    |
| 625                  | Soil; extraction of iron, aluminium and manganese oxides; acidic ammonium oxalate reagent            |
| 640                  | Interstitial Water; solvent displacement from sediment   |
| 650                  | Interstitial Water; pH; electrometric  |
| 651                  | Interstitial Water; Fluoride; ion selective electrode  |
| 652                  | Interstitial Water; Aluminium; spectrometric   |
| 653                  | Interstitial Water; Aluminium; graphite furnace aas  |
| 654                  | Interstitial Water; Lithium; flame aas   |
| 660                  | Sediment; Sodium; flame aas  |
| 661                  | Sediment; Potassium; flame aas   |
| 662                  | Sediment; Calcium; flame aas   |
| 663                  | Sediment; Magnesium; flame aas   |
| 664                  | Sediment; Barium; flame aas  |
| 665                  | Sediment; Strontium; flame aas   |
| 666                  | Sediment; Lead; graphite furnace aas   |

9.1.4      **Determination of Analytes in Dust.**

| <b>Method Number</b> | <b>Method</b>  |
|----------------------|--|
| 800                  | Dust Fallout. Method of Preparation of Test Sample for Laboratory Analysis. Field and Laboratory Procedures. |
| 801                  | Dust fallout, Preparation of Test Sample for Total Kjeldahl Nitrogen, Indophenol Spectrophotometric Method.  |

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## 9.2 Method Authorisation

New methods are developed when there is a perceived shortcoming in an existing method, or where no method exists for an analyte previously not determined. The Laboratory Manager does a thorough assessment of equipment and staff resources before undertaking any new projects. A schematic outline of the steps that are taken is shown in Appendix D. Where a shortcoming exists in a current method it is usually due to one or more of the following:

- (i) poor sensitivity;
- (ii) poor reproducibility;
- (iii) poor conformity to accepted values of reference materials;
- (iv) extreme or unpredictable matrix effects; and
- (v) excessive labour requirements or analytical time.

In all cases existing equipment is assessed to determine whether a new method can be developed to meet the new analytical requirements. If this is not feasible, steps to procure the necessary equipment/consumables, as well as relevant standards and technical documentation, is initiated by the procedure detailed in Section 7.

The principal criteria for the successful development of a method are:

- (i) the elimination or substantial diminution of matrix effects or the demonstration that no interference effects exist for the combinations of matrices and analytes that will be encountered. In most cases this is accomplished by comparing the results of direct calibration determinations with those from a standard additions method;

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- (ii) the establishment of the accuracy of the analyses yielded by the method. This is accomplished by the determination of at least two certified reference materials for all analytes for which the method is used, and by comparison of results with those from at least one other method; and
- (iii) elucidation of the repeatability of the method by replicate determinations of certified reference materials and other authorised control materials; the number of replicates is at least 10. The precision of the method is then expressed as the  $\pm$  % relative standard deviation.

Before a method is used routinely, its effective working range is established. The operational definition of this is usually from the detection limit to that point where a plot of instrument reading against concentration deviates from linearity.

A new or amended test method must be personally authorised by the Laboratory Manager.

Descriptions of test methods used in this laboratory have a standard format. This includes:

- (i) a method number and reference number of the method from which it is derived, if applicable.
- (ii) the date of original approval of method, and of any updates.
- (iii) a title for the method.

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- (iv) the application of the method, including analytes, concentration range, interferences and the types of samples analysed.
- (v) a list of interferences to the method, and their severity, if measured.
- (vi) a brief description of the principle of the method.
- (vii) a description of any preservation or storage requirements for samples or calibration stock solutions.
- (viii) a list of all apparatus required for the method.
- (ix) a list of all reagents used for the method, including concentrations of stock and working solutions and instructions for their preparation.
- (x) a detailed description of the operation of the method and any apparatus used. This includes specific instructions when difficulties or ambiguities may be encountered, where adherence to the method is particularly critical or where safety is a factor.
- (xi) a description of how calculations are made and results are reported.
- (xii) a statement of the precision and accuracy of the method; and
- (xiii) a bibliography which includes reference to methods from which the method was derived.



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## **10.0 OPERATIONAL PROCEDURES**

### **10.1 The Origin of Samples**

Samples may originate from two sources:

- (i) those generated from projects within the Environmental Chemistry Section; and
- (ii) those constituting service work for other sections.

In either case, at the beginning of a specific project, the work is given a job number of the form X/YZ where YZ is the last two digits of the year and X is a sequential number beginning at 1 for that year. At the same time a file is raised onto which is placed all documentation pertaining to that job. In addition, details of the job, including the anticipated number of samples are recorded in a green-covered book located in the bottom drawer of a filing cabinet near the door of the laboratory. This book is titled, "Work Record Book".

### **10.2 Sampling, Sample Receipt and Registration**

When surface waters are sampled by staff of this laboratory, they are collected in accordance with the NATA registered ERISS Method Number 192. However, samples are also received which were collected or prepared by other Sections within the ERISS. These are accepted for analysis on an as-received basis and documented as such.

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Upon receipt all samples are recorded in the laboratory sample register. This registers samples in sequential order, and records the following details: job number; sampling location; date collected; all determinations required; whether ICPMS analysis is required and if so, the batch number (see Section 15). On receipt, a diagonal stroke is placed in the box of each required analyte; completion of the determination is signified by a diagonal stroke in the opposite direction. Under current laboratory practice, all blanks receive a sample number and a separate sample number is assigned to filtered aliquots of a sample. If samples are to be analysed by ICPMS, reference and control waters, as well as replicates and dilutions of samples are assigned separate sample numbers. If analyses are performed in the laboratory, references, controls, replicates and dilutions do not receive separate sample numbers. The sample register is kept in the foyer of the Analytical Chemistry Laboratory, along with completed registers. They are not to be removed from the laboratory without express authorisation from the Laboratory Manager.

### **10.3 Storage of Samples**

Prior to analysis and following acidification, if appropriate, all samples are refrigerated, preferably in the refrigerator in the General Instrument room, or if space is insufficient, in the refrigerators in the foyer of the Analytical Chemistry Laboratory. Samples for nutrient analyses are frozen, (nitrate, phosphate, ammonia). Other samples, reference and control waters (except nutrients) are stored at 4°C.

When all analyses have been completed and results checked, samples are stored in the cool room (4°C) or the freezer room (-18°C), both located in the Sample Preparation Laboratory, until assessment of the results has finished. This is usually reviewed annually, although occasionally it may be in three months.

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When samples are transferred to long-term storage, details are entered in a Sample Storage Register, located on the east wall of the laboratory. Samples are stored in "hobby boxes" and the storage register records details of specific storage location within the refrigerated rooms. Records of sample transfer and disposal are immediately updated when the action is undertaken in a paradox database in two files : 'job.list' and 'box.list'.

#### **10.4 Analysis of Samples and Quality Control**

All analyses of samples and their related blanks, reference solutions, control samples and replicates must be performed according to the methods detailed in the Standard Methods Manual, and listed in Section 9 of this manual.

All methods provide for a results sheet. Results and calculations are shown clearly on this sheet, which must be signed by the analyst and an authorised checking officer. Both the date of analysis and checking must be affixed. Liquid paper is not permitted to change errors. These must be neatly crossed out, initialled and the correct value placed adjacent.

All analytical runs must contain at least one certified reference water. Control samples prepared by this laboratory and having in-house accepted values are sometimes also included in runs. In the case of certified reference waters, unless the values obtained are within two standard deviations of the accepted value, the analytical run must be repeated. Values for these waters are written in the appropriate register by the analyst.

The duties of the checking officer are:

- (i) to ensure conformity of reference values to their accepted value;
- (ii) to check calculations and the possibility of transcription errors; and
- (iii) to compare results with those for similar samples. If a marked deviation from expected values is encountered, the cause is investigated.

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Apart from poor results for certified reference waters, results may be invalidated if there is an obvious large drift in instrumental readings, particularly if this is not uniform, or if blank values are large and variable.

### **10.5 Processing of Results**

Once the validity of results has been established beyond reasonable doubt, they are placed on the relevant job file. Reports are prepared by abstraction of results from these files. Occasionally, results are entered into a computer data base to permit statistical manipulation. In that case, the veracity of data entry is ensured by a checking officer. As explained in Section 15, all ICPMS results are entered into a computer data base. The person responsible for the production of test reports is the Laboratory Manager.

### **10.6 Housekeeping**

The housekeeping policy of the laboratory is designed to ensure the efficiency, safety and morale of the working environment.

A key element of housekeeping policy is the maintenance of standards of cleanliness proper to a laboratory performing trace and ultratrace analyses. Special cleaning procedures have been instituted for the clean preparation and clean analysis rooms. These are documented in the Standard Method Manual, Volume 1, especially in Sections 5 and 7.

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All unnecessary clutter is avoided to conserve limited bench and desk space and filing and storage systems have been instituted to implement this objective.

In all areas in which samples are prepared for analysis, special places are reserved for analytes particularly prone to contamination, for example, nutrients and solutions of high dilution. Details of these provisions are contained in Section 5 of Volume 1 of the Standards Methods Manual.

A flowsheet of the operational procedures of this laboratory is included as Appendix D of this manual.

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## **11.0 CONTROL OF TEST ITEMS**

### **11.1 Receipt, Storage and Disposal**

The receipt, storage and disposal of samples is discussed in Sections 10.2 and 10.3 of this manual. The protocol can be summarised as follows:

- (i) all samples received into this laboratory for analysis, whether collected or prepared by laboratory staff or by the staff of other sections, are presented as aqueous solutions (including digests). Samples received may require further treatment, for example filtration or acidification;
- (ii) upon receipt, sample details are entered into the laboratory sample register. Filtered samples receive a different sample number from an unfiltered portion of the same sample. Blanks also receive a sample number;
- (iii) after sample registration, the samples are treated as required and stored; either at  $-12^{\circ}\text{C}$  (for nutrient analyses) or  $4^{\circ}\text{C}$  (for other analyses);
- (iv) after all analyses are complete, samples are placed in long-term storage (at  $-12^{\circ}\text{C}$  or  $4^{\circ}\text{C}$ ) in freezer and cold rooms in the Sample Preparation Laboratory. These facilities are shared with other sections. Storage details are registered and samples are retained until the analytical report is written and assessment of it is complete. This is at least for 3 months, when a review is made by the Laboratory Manager. Further reviews are made after 1 year, thence annually; and

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- (v) all samples are dilute aqueous solutions; therefore eventual disposal is via the sewerage system.

### **11.2 Protection and Security of Test Items**

Security of test items from interference is provided by their storage behind locked doors in a locked compound which is patrolled by a security guard. The freezer and cold rooms are secured with independent padlocks.

Integrity of samples with regard to losses and contamination is ensured by a three-step process:

- (i) attention to the cleanliness and orderliness of the laboratory so that contamination from the laboratory environment is minimised;
- (ii) rigid adherence to the sample-collection, handling and storage instructions in the authorised test methods. In addition, precepts of good laboratory practice should be adhered to; such as recapping sample and reagent containers as soon as possible and with the correct top, and efficient use of space in the work area; and
- (iii) use of only specified reagents: Suprapur or Aristar grades for acidification, digestion and matrix modification (aas) and Analar or equivalent for other purposes.

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| <b>TEST RECORDS</b>                          | <b>Authorised by:</b>       |

## **12.0 TEST RECORDS**

### **12.1 Records System**

The central repository for all completed documentation from all sections of the Institute is the Registry, located in the administrative building. Completed worksheets, test reports and method development documentation are placed on the relevant job file. Other laboratory documentation, such as correspondence, ordering details for supplies and administrative material are also retained on appropriate files. Blank worksheets, report forms, calibration and maintenance schedules are stored in a filing cabinet near the door of the general laboratory.

No records are ever destroyed. Current files are held in the Registry Compactus, as are some frequently-consulted non-current files. Files which are closed and not used are stored temporarily in an internal archive before eventual transfer to the Australian Archive at Millner, Darwin.

### **12.2 Verification of Data**

Calculations and data transfer are verified in the first instance by the checking officer. This is done by proof reading all transcriptions and by actually performing a random selection of calculations. Special verification of calculations and transcription is made if the value obtained is unusual, based on experience.

### **12.3 Confidentiality and Security**

Laboratory reports are only issued to clients who have made the analysis request. A copy of the test report is retained in the ERISS internal registry filing system and is therefore subject to this organisations file security policy.



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### **13.0 DIAGNOSTIC AND CORRECTIVE ACTION**

#### **13.1 Internal Feedback**

If unusual results are encountered, these are typically noticed by the analyst if he/she has had experience with these type of analyses before, for example with the concentration ranges normally expected. The first course would be to ensure that the values for certified reference waters conformed to their rejection criterion, i.e. within two standard deviations of the certified mean values. If no problems are discerned with reference materials, and no obvious drift or malfunction of equipment is observed then the correct course is as follows:

- (i) write a minute describing the deviation of the results from those expected and stating that no problems with equipment or the results of certified reference materials have been detected; and
- (ii) if possible suggest a cause for the deviation - this may be in terms of seasonal variation, matrix effects or errors in sample collection or preparation.

All documentation is passed onto the checking officer who either initiates action on the basis of statements of deviation, or when in the course of checking detects anomalous results either in reference materials or samples.

It is the duty of the Laboratory Manager in consultation with other laboratory staff to diagnose the cause of the anomalies and, if necessary to determine and to implement corrective action. The resolution of the unexpected situation involves the following:

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- (i) diagnosis: this involves a definitive judgement of whether a problem exists with results, and where possible the precise nature of the problem, and its origin;
- (ii) corrective action determination: this involves deciding what is to be done to overcome the problem; it may include evaluation of equipment performance, assessment of training requirements, modification of laboratory or test procedures or counselling of staff;
- (iii) implementation: this involves putting the plan developed above into action. It may include calibration, maintenance or repair of equipment; training or counselling of staff or amendment of laboratory procedures or documentation; and
- (iv) review: this involves the assessment of the program implemented above. It requires an honest appraisal of the efficacy of any remedial action deemed necessary.
- (v) All details of corrective action are recorded in the appropriate method development file for the test.

### **13.2 External Feedback**

Generally, feedback from clients would involve questioning results which do not conform to what was expected. In many cases this could have been anticipated above (13.1) and an explanation already formulated. If a genuine error has escaped, the investigative procedure, which is the responsibility of the Laboratory Manager, is similar to that in Section 13.1

### **13.3 Proficiency-testing Programs**

It is laboratory policy to participate in proficiency-testing programs as they give an independent assessment of the laboratory's performance and management of quality control.

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The main proficiency-testing programs which involve this laboratory are those organised by NATA. NATA endorsed reports are issued to NATA on completion of the exercise. In the past, round-robin programs have been organised by this laboratory and Northern Territory government departments; other round-robin exercises have been undertaken occasionally.

Upon receipt of results from a proficiency-testing program, a critical appraisal is made of the laboratory's performance. Any deficiencies discovered are investigated using the procedure detailed in Section 13.1. The results of proficiency-testing results are shown to each staff member. A copy of the proficiency-testing results is filed in the appropriate method development file for the test.

#### **13.4 Verification Procedures**

The procedures used in this laboratory to ensure the quality of results are:

- (i) **Certified Reference Materials.** These are analysed routinely with every batch of samples determined. If possible two such materials are included in each run and all must satisfy the rejection criterion of being within two standard deviations of the certified mean value. A list of all certified reference materials held by this laboratory is provided at Appendix E. Depleted stocks of such materials are recorded and reordered by the Laboratory Manager;
- (ii) **replicate Testing.** At least one sample in ten is repeated to monitor the stability of the equipment; and
- (iii) **other Verification Procedures.** The principal other verification procedure used in this laboratory is the determination of the same analytes using a different method, for example aas and potentiometric stripping analysis or ion chromatography and aas. This usually involves a different analyst. In many cases reanalysis occurs after a substantial time delay. This serves to verify both the method(s) and the storage procedures. Where the analytes are major cations/anions, verification can take the form of an equivalent ion balance.

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## **14.0 TEST REPORTS**

### **14.1 Format of Reports**

#### **14.1.1 NATA-Endorsed Reports**

Test documents must present results clearly and unambiguously. The actual test report should contain the following information:

- (i) the full name and address of the testing laboratory;
- (ii) the name of the client;
- (iii) a unique identification of the test document which can clearly relate it to the client;
- (iv) the total number of pages in the report, including coversheet;
- (v) the date of the test report;
- (vi) the NATA logo;
- (vii) the signature of an approved signatory; and
- (viii) the date that samples were received.

The actual test report must contain the following information:

- (i) a full and unambiguous identification of the test sample, which may include details of sampling techniques and sample history;
- (ii) which test method(s) were used including details of any modifications;
- (iii) results reported with a statement of their uncertainty, or as required by the test method(s);
- (iv) a unique identification of the test document;

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- (v) page numbers which are sequential and which state the total number of pages in the report;
- (vi) the signature of an approved signatory; and
- (vii) a statement of compliance with nominated specifications or procedures, where appropriate.

NATA endorsed reports must contain test results only; no comment or interpretation of data is permitted. Such additional information, where required, must be contained in a clearly identified separate document. No NATA endorsed test report may contain any alterations or erasures.

#### 14.1.2 Non-NATA-Endorsed Reports

Most reports issued by this laboratory are to clients within the Institute. These are not NATA endorsed. They take three forms:

- (i) Reports which comprise one page for each sample or submitted blank, together with a covering minute detailing the analyses reported therein. These reports contain details of: sample description; project; job and sample numbers; date of receipt, analysis and report; client; file number and test results. Reports must be signed by the Laboratory Manager. They have provision for remarks to be made but generally do not provide an estimate of uncertainty. It is now laboratory policy to provide details of the test method used;
- (ii) Reports in the form of a computer printout of ICPMS results, together with a covering minute nominating the samples analysed. The printout

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may either be the hard copy direct from Sydney, accompanied by the sample details printout or a derived computer printout which has been modified by laboratory staff, as detailed in Section 15.2.3; and

- (ii) a typed table of results when the number of samples is very large.

#### **14.2 Checking of Reports**

In all cases, whether NATA-endorsed or not, reports are signed by the Laboratory Manager after thorough checking of all details. Currently the junior experimental officer of the laboratory is also a NATA signatory, and may sign test reports which are urgent, in the absence of more senior staff.

#### **14.3 Revision of Reports**

If an error is detected in a report after dispatch, an amended report must be issued. In the case of NATA-endorsed reports the amended report must be a complete re-issue. For reports to Institute clients only the individual sheets, one for each sample, that are in error are re-issued. These are clearly annotated that they are amended result sheets.

#### **14.4 Preliminary Reports**

With approval from the Laboratory Manager a preliminary report may be issued to a client. A preliminary report is subject to the same conditions for production as a final laboratory report. A preliminary report is labelled as an "INTERIM LABORATORY REPORT", and is described as such in the "Comments" section in the report. When a final laboratory report is issued the reference to the preliminary report is made in the "Comments" section of this report.

#### **14.5 Retention of Reports**

A copy of all reports is retained on the appropriate job file. A copy of all the current year's reports are also retained in the laboratory. The files are stored in the Institute Registry while regularly accessed and otherwise for three to five years as space permits. They are finally transferred to the Australian Archives in Darwin.

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## **15.0 SUBCONTRACTING**

### **15.1 Background**

In response to a large anticipated increase in the requirement for chemical analyses within the ERISS, the decision was taken to join a consortium, with the Australian Nuclear Science and Technology Organisation (ANSTO) and the Commonwealth Scientific and Industrial Research Organisation (CSIRO), to purchase a VG Plasmaquad Inductively Coupled Plasma - Mass Spectrometer (ICPMS). The OSS is 25% owner of the equipment and has use of the equipment for one day per week. It is housed at the CSIRO Division of Fuel Technology at Lucas Heights in Sydney and is operated by CSIRO staff. The ERISS contributes to the cost of salaries, maintenance and consumables in proportion to its percentage ownership.

The ICPMS operates as follows. A radio frequency signal generates a plasma in a stream of argon gas. This plasma 'torch' has a temperature of about 8000 K. At this temperature, metal atoms (and a few other elements) which are drawn into the plasma using a nebuliser similar to that on aas, are ionised almost entirely to the 1+ state. These ions are directed to the inlet of a quadrupole mass spectrometer under vacuum and are separated on the basis of their mass. The ICPMS is therefore capable of rapid sequential determination of approximately 70 elements and can discriminate between isotopes.

The main attraction of ICPMS to the ERISS has been its ability to complete multi-element analyses on 200-250 samples per day under favourable conditions. For most elements sensitivity and accuracy is at least as good as for alternative methods.

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## **15.2 Operation of the System**

The procedure for sending samples and obtaining analytical results from the ICPMS comprises five components:

- (i) preparation of solutions for analysis, and packing these for dispatch;
- (ii) documentation of the solutions (including blanks, samples, reference and control waters) and checking of this documentation;
- (iii) dispatch of the samples to Sydney where they are analysed and the results returned to the laboratory as both hard copy and on floppy disk;
- (iv) processing the data received using the IBM personal computer; and
- (v) issue of reports to clients.

### **15.2.1 Preparation of samples**

Solutions for analysis consist of: blanks; samples; certified reference waters and internal control waters, as well as replicates of samples. All solutions are contained in 50 mL bottles and are acidified to 1%  $\text{HNO}_3$  with BDH Aristar acid (500  $\mu\text{L}$   $\text{HNO}_3$  to 50 mL). They are also spiked with 25  $\mu\text{L}$  of 50 mg/L indium solution (giving a final indium concentration of 25  $\mu\text{g/L}$ ). In many cases the solutions need to be diluted (1:10 or 1:100) to bring one or more analytes into the operational concentration range (0 to 1000  $\mu\text{g/L}$ ) of the ICPMS. In all such cases it is the final dilution which is acidified and spiked with indium. The solutions are placed in a heavy-duty cardboard box divided into compartments with cardboard spacers.



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#### 15.2.2 Sample documentation and storage of sample details

All sample details and documentation is managed by the ERISS LAN network via a personal computer work station and a Toshiba laser printer located in the office. The sample details are recorded as a batch file in a sub-directory with the pathway:

**u:\groups\mining\chem\data\db\icpms\despatch\'batch filename' e.g. b117des.prn**  
using word for windows software.

- (i) batch number and sequence number within a batch.
- (ii) sample number;
- (iii) sample number of the corresponding blank (or 'yes' if the current sample is a blank);
- (iv) whether the sample is a reference or control sample or not;
- (v) the sample number of replicates to the current sample;
- (vi) job number;
- (vii) date;
- (viii) information on source and location of sample;
- (ix) analytes to be determined;
- (x) filtration details;
- (xi) acidification details;
- (xii) dilution factor; and

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A labels file is written and labels printed (blank label sheets must be positioned in the Toshiba laser printer in the laboratory office before printing is commanded. Each batch is checked by the Laboratory Manager before dispatch. A copy of the documentation for checking and to provide analytical details to the analyst in Sydney is generated. Both of these documents are checked for errors in data entry and cross-checked against the samples in the dispatch box. This checking is properly annotated on the documents. The printout containing complete sample details is included in the box of samples. Copies of both printouts are placed in a file called 'ICPMS Batch Dispatch' in this laboratory. A copy of all documentation is also placed on each relevant job file. Only current 'ICPMS Batch Dispatch' files are stored in this laboratory. Older volumes are placed in the ERISS Registry under File JR-06-074.

#### 15.2.3 Dispatch of samples and receipt of results

The box of samples is sent to Sydney either by air transport or road transport, as the urgency of the consignment dictates. Analytical results are returned in hard copy by mail or facsimile as the urgency requires. The hard copy is filed in the most recent part of 'ICPMS Batch Results' file in the laboratory. Old results are filed in the ERISS Registry under File JR-06-075. A floppy disc containing the results is sent by mail. The data on the floppy disc is downloaded to the personal computer in the sub-directory - :

**u:\groups\mining\chem\data\icpms\rawresul\'batch filename' e.g. b117res.prn**

and converted to a quottro pro file. The data is then processed. Occasionally samples need to be returned to Jabiru for further analysis. Return is usually by road transport.

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#### 15.2.4 Processing data

Data are transferred from the collected floppy disc records to the results file and further to 'icpms' directory into the following sub- directories: 'aes', 'quant' and 'semiqual'. The data can then be manipulated using two choices:

- (i) generation of a worksheet which allows arithmetic manipulation such as blank and dilution corrections via option 5; and
- (ii) generation of a spreadsheet which allows modification of data and changes of format.

Instructions on how to use these options are contained in registry file "ICP-MS Waters Method Development" JR-07-041.

#### 15.2.5 Issue of Reports

Computer based data manipulation allows data to be presented in a form suitable for a final report, either as modified raw data via a spreadsheet or as blank- or dilution-corrected data via a worksheet. There also remains the alternative to abstract data from either the computer or hard copy to present as a conventional test report (Section 14).

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## **16.0 OCCUPATIONAL HEALTH AND SAFETY**

### **16.1 General**

The ERISS has an Occupational Health and Safety Committee of five members. The chair of this committee is the head of the Environmental Chemistry Section. It meets periodically to review safety for the whole Jabiru East site. First Aid facilities, including respiration equipment is available on-site. The Institute encourages all staff to have a basic knowledge of First Aid, and especially to complete an approved course, and three staff members are designated as First Aid officers.

In accordance with Commonwealth Government Workers' Compensation regulations, all accidents and incidents must be reported in writing to the Senior Administrative Officer, who informs the Occupational Health and Safety Committee, which must investigate and report on the circumstances. Occupational rehabilitation is also governed by Commonwealth Government regulations, details of which are available from the Administrative Support Section.

Appropriate protective clothing, such as safety glasses, hats, boots and face masks, are supplied to all ERISS staff whose job requires it.

All ERISS field vehicles are equipped with radio transceivers and winches. The use of other field equipment is subject to special competence training and assessment.

Within the Analytical Chemistry Laboratory, the following Occupational Health and Safety provisions have been instituted.

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### **16.2 Poisoning and Other Chemical Hazards**

All chemicals which are stored in the laboratory have their details entered in a Chemical Register located on the eastern wall of the laboratory. This Register contains details of quantity, location and hazard rating. All chemicals whose poison hazard is rated as high are stored in a locked Poisons Cabinet. The key to this cabinet is kept in the Laboratory Managers office. Chemicals stored in this cabinet must only be removed for immediate use and returned immediately afterwards. Eye-wash stations are located in all rooms of the laboratory. All rooms have emergency showers. There is also a absorbent kit for spills of hazardous chemicals, particularly acid solutions.

### **16.3 Fire and Explosion Hazards**

Bulk quantities of all substances which constitute a fire or explosion hazard are retained in a special solvent store which is located at the south-western corner of the ERISS compound. Laboratory-scale quantities are stored in a metal cabinet in the laboratory. All flammable or explosive materials must at all times be kept well separated from oxidising agents such as nitric and perchloric acids, or from flames and heating mantles.

All rooms within the laboratory have fire control and safety equipment, such as fire extinguishers (halon type and/or soda acid), fire blanket and water-jel burn blanket. All fire-control and fire-safety equipment is inspected annually by Fire Fighting Enterprises Pty Ltd.

Control of other fire hazards, such as the accumulation of flammable waste, is accomplished by daily disposal of rubbish from waste paper receptacles and attention to the general tidiness of the laboratory, under the direction of the Laboratory Manager.

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#### **16.4 Other Hazards**

The most important hazards in this category are physical obstructions. All staff members are responsible for ensuring that thoroughfares are not blocked by equipment or containers of samples, that sufficient room is available to move portable items and that material stored on shelves cannot fall. It is, however, the ultimate responsibility of the Laboratory Manager to ensure that physical safety hazards are absent.

When sampling in the field there must be two persons present at all times. The staff must be properly attired with protective clothing including shoes and a hat. UV resistant sun screen cream should be applied to exposed areas of the skin. Caution should be exercised in the presence of salt-water crocodiles around artificial and natural water bodies.

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## **17. ACCREDITATIONS HELD**

### **17.1 NATA**

The Analytical Chemistry Laboratory of the Environmental Research Institute of the Supervising Scientist is registered with the National Association of Testing Authorities (NATA) in the field of Chemical Testing.

Registration number: 1511.

The terms of registration are listed in Appendix E. There are currently three approved signatories denoted by ♦

|                          |  |
|--------------------------|--|
| D. Klessa                | Senior Research Scientist, (Head Chemistry Group)        |
| ♦ P. Cusbert<br>Manager) | Senior Professional Officer, MRACI, C. Chem. (Laboratory |
| ♦ C. le Gras             | Senior Professional Officer, MRACI, C. Chem.             |
| ♦ C. Hunt                | Senior Technical Officer                                 |
| Tho Anh Tien             | Technical Officer  |

NATA-endorsed reports must be issued in accordance with the laws and by-laws of the Association.

### **17.2 ASPAC**

The laboratory holds accreditation from the Australian Soil and Plant Analysis Council (ASPAC) in the analysis of soils for the following tests:

pH, electrical conductivity, and organic carbon:

and in the analysis of plants for the following tests:

sodium ,potassium, magnesium, iron, aluminium, zinc. copper, total Kjeldahl nitrogen and total phosphorus





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**NATIONAL ASSOCIATION OF TESTING AUTHORITIES AUSTRALIA**

**Terms of Registration**

**Item 7.66 Waters**

**Section 71 Sampling**

Sampling of surface waters, bore waters, pond waters

Reference: APHA Part 1060

In house methods adopted from Batley, G.E. and Gardner, D., Water Research, 11, 745-756 (1977).

**Section 99 Other Waters (Analysis of uranium mine/mill waters)**

| Analyte          | Method                             | Reference                       |
|------------------|------------------------------------|---------------------------------|
| alkalinity       | acidimetric titration              | APHA Part 2320                  |
| conductivity     | electrometric                      | APHA Part 2510B                 |
| pH value         | electrometric                      | APHA Part 4500-H <sup>+</sup> B |
| temperature      | thermometer                        | APHA Part 2550B                 |
| turbidity        | nephelometric                      | APHA Part 2130B                 |
| dissolved oxygen | electrometric                      | APHA Part 4500-OG               |
| aluminium        | flame atomic absorption            | APHA Part 3111D                 |
| calcium          | flame atomic absorption            | APHA Part 3111B                 |
| magnesium        | flame atomic absorption            | APHA Part 3111B                 |
| manganese        | flame atomic absorption            | APHA Part 3111B                 |
| zinc             | flame atomic absorption            | APHA Part 3111B                 |
| iron             | flame atomic adsorption            | APHA 3111B                      |
| aluminium        | graphite furnace atomic absorption | APHA 3113B                      |
| cadmium          | graphite furnace atomic absorption | APHA 3113B                      |
| copper           | graphite furnace atomic absorption | APHA 3113B                      |
| lead             | graphite furnace atomic absorption | APHA 3113B                      |
| manganese        | graphite furnace atomic absorption | APHA 3113B                      |
| zinc             | graphite furnace atomic absorption | APHA 3113B                      |
| sodium           | ion chromatography                 | cation exchange ion             |
| potassium        | ion chromatography                 | chromatography HPLC             |
| ammonium-N       | ion chromatography                 | non standard (in house)         |
| chloride         | ion chromatography                 | modified APHA Part 4110B        |
| nitrate-N        | ion chromatography                 | modified APHA Part 4110B        |
| sulphate         | ion chromatography                 | modified APHA Part 4110B        |
| orthophosphate   | ascorbic acid spectrophotometric   | APHA Part 4500-PE               |
| uranium          | PAR spectrophotometric             | AS 2733 - 1984                  |
| uranium          | Scintrex fluorimetric              | (in house) non standard         |

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|  | <b>Issue Number: 6</b>      |
| <b>TITLE OF SECTION</b>                      | <b>Issue Date: 07/05/97</b> |
| <b>TERMS OF REGISTRATION</b>                 | <b>Authorised by:</b>       |

Item 7.81 Constituents of the Environment

Section 11 waters other than saline

Tests

| Analyte          | Method                             | Reference                       |
|------------------|------------------------------------|---------------------------------|
| alkalinity       | acidimetric titration              | APHA Part 2320                  |
| conductivity     | electrometric                      | APHA Part 2510B                 |
| fluoride         | electrometric                      | APHA Part 4500-F <sup>-</sup> C |
| pH value         | electrometric                      | APHA Part 4500-H <sup>+</sup> B |
| temperature      | thermometer                        | APHA Part 2550B                 |
| turbidity        | nephelometric                      | APHA Part 2130B                 |
| dissolved oxygen | electrometric                      | APHA Part 4500-O-G              |
| aluminium        | flame atomic absorption            | APHA Part 3111D                 |
| calcium          | flame atomic absorption            | APHA Part 3111B                 |
| iron             | flame atomic absorption            | APHA Part 3111B                 |
| magnesium        | flame atomic absorption            | APHA Part 3111B                 |
| manganese        | flame atomic absorption            | APHA Part 3111B                 |
| zinc             | flame atomic absorption            | APHA Part 3111B                 |
| aluminium        | graphite furnace atomic absorption | APHA Part 3113B                 |
| barium           | graphite furnace atomic absorption | APHA Part 3113B                 |
| cadmium          | graphite furnace atomic absorption | APHA Part 3113B                 |
| chromium         | graphite furnace atomic absorption | APHA Part 3113B                 |
| copper           | graphite furnace atomic absorption | APHA Part 3111B, 3113B          |
| lead             | graphite furnace atomic absorption | APHA Part 3111B, 3113B          |
| manganese        | graphite furnace atomic absorption | APHA Part 3111B, 3113B          |
| nickel           | graphite furnace atomic absorption | APHA Part 3113B                 |
| silver           | graphite furnace atomic absorption | APHA Part 3113B                 |
| strontium        | graphite furnace atomic absorption | APHA Part 3113B                 |
| zinc             | graphite furnace atomic absorption | APHA Part 3111B, 3113B          |
| sodium           | ion chromatography                 | cation exchange ion             |
| potassium        | ion chromatography                 | chromatography HPLC             |
| ammonium-N       | ion chromatography                 | non standard (in house)         |
| chloride         | ion chromatography                 | modified APHA Part 4110B        |
| nitrate-N        | ion chromatography                 | modified APHA Part 4110B        |
| sulphate         | ion chromatography                 | modified APHA Part 4110B        |
| orthophosphate   | ascorbic acid spectrophotometric   | APHA Part 4500-PE               |
| uranium          | PAR spectrophotometric             | AS 2723 - 1984                  |
| uranium          | Scintrex fluorimetric              | (in house) non standard         |

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| <p style="text-align: center;"><b>LABORATORY QUALITY MANUAL</b></p> <p style="text-align: center;"><b>ERISS ANALYTICAL CHEMISTRY LABORATORY</b></p> | <p><b>Section: Appendix B</b></p> <p><b>Sheet: 3</b></p> <p><b>Issue Number: 2</b></p> |
| <p style="text-align: center;"><b>TITLE OF SECTION</b></p> <p style="text-align: center;"><b>TERMS OF REGISTRATION</b></p>                          | <p><b>Issue Date: 24/07/95</b></p> <p><b>Authorised by:</b></p>                        |

Section .71  
Sampling of waters other than saline

Reference:           APHA Part 1060  
In house methods adopted from Batley, G.E. and Gardner, D., Water  
Research, 11, 745-756 (1977)

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| <p style="text-align: center;"><b>LABORATORY QUALITY MANUAL</b></p> <p style="text-align: center;"><b>ERISS ANALYTICAL CHEMISTRY LABORATORY</b></p> | <p style="text-align: right;"><b>Section: Appendix C</b></p> <p style="text-align: right;"><b>Sheet: 1</b></p> <p style="text-align: right;"><b>Issue Number: 4</b></p> |
| <p style="text-align: center;"><b>TITLE OF SECTION</b></p> <p style="text-align: center;"><b>ORGANISATION CHART</b></p>                             | <p style="text-align: right;"><b>Issue Date: 07/05/97</b></p> <p style="text-align: right;"><b>Authorised by:</b></p>   |

## Appendix C

### ORGANISATION CHART OF ERISS EMPHASISING ANALYTICAL CHEMISTRY

|   |   |
|---|---|
| <b>THE SUPERVISING SCIENTIST</b>                                      | <b>VACANT AWAITING APPOINTMENT</b>                  |
| <b>THE DIRECTOR (ERISS)</b>   | <b>ARTHUR JOHNSTON</b>                              |
| <b>CORPORATE SERVICES<br/>WETLAND MANAGEMENT<br/>IMPACT OF MINING</b> | <b>R. GRAHAM<br/>M. FINLAYSON<br/>B. PRENDEGAST</b> |
| <b>ENVIRONMENTAL RADIOACTIVITY<br/>GEOMORPHOLOGY<br/>CHEMISTRY</b>    | <b>P. MARTIN<br/>K. EVANS<br/>D. KLESSA</b>         |
| <b>ANALYTICAL CHEMISTRY</b>   | <b>P. CUSBERT<br/>THO ANH TIEN</b>                  |
| <b>ENVIRONMENTAL CHMISTRY</b>   | <b>C. Le GRAS<br/>C. HUNT</b>                       |

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|---|---|
| <p style="text-align: center;"><b>LABORATORY QUALITY MANUAL</b></p> <p style="text-align: center;"><b>ERISS ANALYTICAL CHEMISTRY LABORATORY</b></p> | <p><b>Section: Appendix E</b></p> <p><b>Sheet:1</b></p> <p><b>Issue Number: 4</b></p> |
| <p style="text-align: center;"><b>TITLE OF SECTION</b></p> <p style="text-align: center;"><b>LIST OF STANDARDS HELD BY THE LABORATORY</b></p>       | <p><b>Issue Date: 07/05/97</b></p> <p><b>Authorised by:</b></p>                       |

**US National Bureau of Standards**

|  |                  |
|--|------------------|
| Flint Clay                                     | NBS SRM 97a      |
| Sodium Feldspar                                | NBS SRM 99a      |
| Potassium Hydrogen Phthalate                   | NBS SRM 185f     |
| Potassium Dihydrogen Phosphate                 | NBS SRM 186-I-C  |
| Disodium Hydrogen Phosphate                    | NBS SRM 186-II-C |
| Borax  | NBS SRM 187      |
| Sodium Bicarbonate                             | NBS SRM 191a     |
| Sodium Carbonate                               | NBS SRM 192a     |
| Uranium Oxide (U <sub>3</sub> O <sub>8</sub> ) | NBS SRM 950 b    |
| Oyster Tissue                                  | NBS SRM 1566     |
| Oyster Tissue                                  | NBS SRM 1566a    |
| Wheat Flour                                    | NBS SRM 1567     |
| Rice Flour                                     | NBS SRM 1568     |
| Brewers Yeast                                  | NBS SRM 1569     |
| Trace Elements in Spinach                      | NBS SRM 1570     |
| Orchard Leaves                                 | NBS SRM 1571     |
| Tomato Leaves                                  | NBS SRM 1573     |
| Pine Needles                                   | NBS SRM 1575     |
| Bovine Liver                                   | NBS SRM 1577     |
| Trace Elements in Coal                         | NBS SRM 1632a    |
| Trace Elements in Coal Fly Ash                 | NBS SRM 1633a    |
| Mercury in Water µg/mL                         | NBS SRM 1641a    |
| Mercury in Water ng/mL                         | NBS SRM 1642a    |
| Trace elements in water                        | NBS SRM 1643c    |
| River Sediment                                 | NBS SRM 1645     |
| Estuarine Sediment                             | NBS SRM 1646     |
| Urban Particulate Matter                       | NBS SRM 1648     |
| Potassium Chloride                             | NBS SRM 2202     |
| Potassium Fluoride                             | NBS SRM 2203     |
| Gold   | NBS SRM 3121     |
| Palladium                                      | NBS SRM 3138     |
| Platinum                                       | NBS SRM 3140     |

**LABORATORY QUALITY MANUAL**

**Section: Appendix E**

**ERISS ANALYTICAL CHEMISTRY LABORATORY**

**Sheet: 2**

**Issue Number: 3**

**TITLE OF SECTION**

**Issue Date: 24/07/95**

**LIST OF STANDARDS HELD BY THE LABORATORY**

**Authorised by:**

**US EPA ENVIRONMENTAL PROTECTION AGENCY: QUALITY CONTROL**

**Reference Waters**

|   |         |              |
|---|---------|--------------|
| US EPA QC Sample Nutrient Conc 1                | WP 486  |              |
| US EPA QC Sample Nutrient Conc 2                | WP 486  |              |
| US EPA QC Sample Nutrient Conc 1                | WP 481  |              |
| US EPA QC Sample Nutrient Conc 2                | WP 481  |              |
| US EPA QC Sample Nutrient Conc 3                | WP 481  |              |
| US EPA QC Sample Nutrient Conc 4                | WP 481  |              |
| US EPA QC Sample Nutrient Conc 5                | WP 481  |              |
| US EPA QC Sample Nutrient Conc 6                | WP 481  |              |
| US EPA QC Sample Nutrient Conc 7                | WP 481  |              |
| US EPA QC Sample Nutrient Conc 8                | WP 481  |              |
| US EPA QC Sample Nutrient Conc 3                | 1272    |              |
| US EPA QC Sample Nutrient Conc 1                | WP 486  | W333         |
| US EPA QC Sample Nutrient Conc 2                | WP 486  | W333         |
| US EPA QC Sample Nutrient Conc 1                | WP 987  |              |
| US EPA QC Sample Nutrient Conc 2                | WP 987  |              |
| US EPA WS QC Turbidity Conc 1                   | WS 278  |              |
| US EPA WS QC Turbidity Conc 2                   | WS 278  |              |
| US EPA Water Supply QC Nitrate/Fluoride Conc I  | WS 378  |              |
| US EPA Water Supply QC Nitrate/Fluoride Conc II | WS 378  |              |
| US EPA QC Demand Conc 2                         | 372     |              |
| US EPA QC Demand Conc 1                         | WP 678  |              |
| US EPA QC Demand Conc 4                         | WP 678  |              |
| US EPA QC Demand Conc 6                         | WP 678  |              |
| US EPA Water Supply Trace Metals II Conc 1      | WP 1183 |              |
| US EPA Water Supply Trace Metals II Conc 2      | WP 1183 |              |
| US EPA Water Sample Trace Metals III Conc 1     | WP 1183 |              |
| US EPA Water Sample Trace Metals III Conc 2     | WP 1183 |              |
| US EPA Water Supply Trace Metals Conc 1         | WS 378  | W 104        |
| US EPA Water Supply Trace Metals Conc 1         | WS 378  | W106         |
| US EPA Water Supply Trace Metals Conc 10        | WS 378  | W104         |
| US EPA Water Supply Trace Metals Conc 10        | WS 378  | W 106        |
| US EPA Water Supply Trace Metals Conc 10        | WS 378  |              |
| US EPA Water Supply Trace Metals Conc 13        | WS 378  | W194         |
| US EPA Water Supply Trace Metals Conc 2         | WS 378  | W194         |
| US EPA Trace Metals Analysis Conc 1             | WP 475  |              |
| US EPA Trace Metals Analysis Conc 2             | WP 475  |              |
| US EPA Trace Metals Analysis Conc 4             | WP 475  | W 67         |
| US EPA Trace Metals Analysis Conc 5             | WP 475  | W 67         |
| US EPA Trace Metals Analysis Conc 5             | WP 475  | W 65         |
| US EPA QC Sample Trace Metals Conc 1            | WP 284  |              |
| US EPA QC Sample Trace Metals TMA989 Conc 1     | WP 675  |              |
| US EPA SPEX QC Sample Minerals Conc A           | WP 1290 | (Batch 1320) |
| US EPA SPEX QC Sample Minerals Conc B           | WP 1290 | (Batch 1321) |
| US EPA SPEX QC Sample Minerals pH               | WP 1290 | (Batch 1324) |
| US EPA SPEX QC Sample A Trace Metals            | TMAA-1  |              |
| US EPA SPEX QC Sample A Trace Metals            | TMAA-2  |              |
| US EPA SPEX QC Sample A Trace Metals            | TMAA-3  |              |
| US EPA QC Sample pH Conc 1                      | WP384   |              |
| US EPA QC Sample pH Conc 2                      | WP384   |              |
| US EPA QC Sample pH                             | WP1185  |              |
| US EPA QC Sample pH                             | WP1188  | W592         |
| US EPA QC Sample Cyanide                        | WP 582  | (CYN 989)    |

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| <b>LIST OF STANDARDS HELD BY THE LABORATORY</b> | <b>Authorised by:</b>       |

**International Atomic Energy Agency**

|                          |           |              |
|--------------------------|-----------|--------------|
| Animal Muscle            | H-4       | One Bottle   |
| Animal Bone              | H-5       | One Bottle   |
| Horse Kidney             | H-8       | One Bottle   |
| Human Diet               | H-9       | Two Bottle   |
| Rye Flour                | V-8       | One Bottle   |
| Hay                      | V-10      | One Bottle   |
| Freeze Dried Fish Meal A |           | One Bottle   |
| Trace elements in soil   | Soil-5    | One Bottle   |
| Trace elements in soil   | Soil-7    | Five Bottles |
| Trace elements in soil   | SL-1      | One Bottle   |
| Copepod Homogenate       | MA-A-1/TM | Six Bottles  |
| Copepod Homogenate       | MA-A-1/OC | One Bottle   |
| Oyster Homogenate        | MA-M-1/OC | Six Bottles  |
| Fish Homogenate          | MA-A-2/OC | Five Bottles |
| Sediment                 | SD-N-1/2  | Two Bottles  |

**Commission of European Communities**

|                             |                 |
|-----------------------------|-----------------|
| Lagarosiphon Major          | CBR BCR No. 60  |
| Platihyphnidium Riparioides | CBR BCR No. 61  |
| Beech Leaves                | CBR BCR No. 100 |
| Mussel Tissue               | CBR BCR No. 278 |
| Light Sandy Soil            | CBR BCR No. 142 |
| Cod fish                    | CBR BCR No.     |
| Mussel                      | CBR BCR No.     |

**National Research Council Canada**

|                 |        |
|-----------------|--------|
| Marine sediment | PACS-1 |
|-----------------|--------|

**I.A.P.S.O.**

|   |         |
|---|---------|
| Standard seawater P92                   | 10.8.84 |
| Standard seawater P92 Diluted 1 in 1000 | 10.8.84 |

**National Research Council Canada**

|                           |            |
|---------------------------|------------|
| Marine Sediment           | PACS-1     |
| St. Lawrence River Sample | NRC SLRS-1 |
| St. Lawrence River Sample | NRC SLRS-2 |

**Canadian Certified Reference Materials**

|                         |        |            |
|-------------------------|--------|------------|
| Reference Lake Sediment | LKSD-1 | One Bottle |
| Reference Lake Sediment | LKSD-2 | One Bottle |
| Reference Lake Sediment | LKSD-3 | One Bottle |
| Reference Lake Sediment | LKSD-4 | One Bottle |

**Institute of Marine and Veterinary Science**

Bovine Liver

|   |                             |
|---|-----------------------------|
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| <b>LIST OF STANDARDS HELD BY THE LABORATORY</b> | <b>Authorised by:</b>       |

#### **AUSTRALIAN SOIL AND PLANT ANALYSIS COUNCIL**

|                                   |         |
|-----------------------------------|---------|
| Soil ASPAC                        | 11K     |
| Soil ASPAC                        | 12L     |
| Soil ASPAC                        | 13K     |
| Soil ASPAC                        | 14J     |
| Soil ASPAC                        | 15K     |
| Soil ASPAC                        | 17K     |
| Plant ASPAC Eucalypt Leaves       | 1A(2)   |
| Plant ASPAC Indian Mustard Leaves | 2H(3)   |
| Plant ASPAC Oats herbage          | 5C4     |
| Plant ASPAC Wheat grain (1)       | 8C4     |
| Plant ASPAC Pinus radiata leaves  | 9A(1)   |
| Plant ASPAC Mixed pasture         | 11H(1)  |
| Plant ASPAC Mango leaves          | 3D(3)   |
| Plant ASPAC Macadamia leaves      | 4D(3)   |
| Plant ASPAC Wheat grain (2)       | 10B(2)a |
| Plant ASPAC Lucerne leaves        | 12f(3)  |

#### **PEKIN ELMER STANDARDS FOR ICP-MS**

|  |              |
|--|--------------|
| Multielement calibration standard      | PE N930-0231 |
| Multielement verification standard I   | PE N930-0232 |
| Multielement verification standard II  | PE N930-0233 |
| Multielement verification standard III | PE N930-0234 |
| Multielement verification standard IV  | PE N930-0235 |

#### **GOODFELLOW METALS**

|            |               |
|------------|---------------|
| Arsenic    | Lumps         |
| Cadmium    | -53#          |
| Calcium    | -250#         |
| Chromi m   | -38#          |
| Copper     | -800#         |
| Iron       | -500#         |
| Lead       | 75-150#       |
| Magnesium  | -50#          |
| Manganese  | Fl ke         |
| Molybdenum | 2# (mean)     |
| Nickel     | -63#          |
| Selenium   | Pallets       |
| Silicon    | Broken Ingots |
| Tantalum   | 45-350#       |
| Tantalum   | 45-350#       |
| Tin        | -50#          |
| Titanium   | -250#         |
| Vanadium   | 45-75#        |
| Zinc       | 75-150#       |



|   |                             |
|---|-----------------------------|
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| <b>TITLE OF SECTION</b>   | <b>Issue Date: 24/07/95</b> |
| <b>Current list of staff qualifications and relative experience</b> | <b>Authorised by:</b>       |

**Dr. D. Klessa - *Senior Research Scientist***

**Qualifications:** B.Sc. (Aberdeen), Ph.D.(East Anglia

**Experience:** Lecturer - Scottish Agricultural College  
Lecturer - University of Sydney  
Senior Research Scientist, Environmental Research Institute  
of the Supervising Scientist, Jabiru.

**Mr. P. J. Cusbert - *Senior Professional Officer***

**Qualifications:** B.Sc. (U.N.E.), M. Eng. Sc. (Newcastle), A.Dip.E.S.  
(M.C.A.E., Bathurst, Dip. Nat. Res. (U.N.E.)

**Experience:** 1962-1982 Supervising Chemist - Pollution Control  
BHP Quality Control Labs., Newcastle Steel Works.

1982- Senior Professional Officer, Environmental Research Institute  
of the Supervising Scientist, Jabiru.

**Dr. C. le Gras - *Senior Professional Officer***

**Qualifications:** B.Sc.(Hons) and Ph.D. (Sydney).

**Experience:** 1977-1982 Post graduate student - University of Sydney; Laboratory  
demonstrator.

1984- Senior Professional Officer, Environmental Research Institute  
of the Supervising Scientist, Jabiru.

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| <b>TITLE OF SECTION</b>   | <b>Issue Date: 07/05/97</b> |
| <b>Current list of staff qualifications and relative experience</b> | <b>Authorised by:</b>       |

**Mr. C. W. Hunt**      *Technical Officer Grade 3*

**Qualifications:**      Certificate in Environmental Biology (Darwin).

**Experience:**      1980-1982      Technical Assistant (2), Northern Territory Department of Mines and Energy.

                                 1982-1987      Technical Assistant (2), Office of the Supervising Scientist, Jabiru.

                                 1987-1988      Technical Assistant (2), CSIRO Division of Soils, Canberra.

                                 1988-1991      Technical Officer (2), Environmental Research Institute of the Supervising Scientist, Jabiru.

                                 1991-      Technical Officer (3), Environmental Research Institute of the Supervising Scientist, Jabiru

**Ms. Anh Tho Tien**      *Technical Officer Grade 2*

**Qualifications:**      B. Sc. Physical Chemistry (University of Hochiminh City, Vietnam)  
Grad. Dip. Sc. (NTU, Darwin).

**Experience:**      1991-1992      Chemistry Technician, Centre of Analytical Services and Experimentation, Hochiminh City.

                                 1992-1994      Engineer Chemist, Centre of Analytical Services and Experimentation, Hochiminh City.

                                 1997-      Technical Officer (2), Environmental Research Institute of the Supervising Scientist, Jabiru.