INTERNAL REPORT

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INVESTIGATION OF METHODS FOR THE DETERMINATION OF SUSPENDED SOLIDS IN WATER

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The Supervising Scientist for the Alligator Rivers Region has research, supervisory and co-ordination responsibilities related to effects on the environment and uranium mining in the Alligator Rivers Region and research and supervisory responsibilities related to effects on the environment of non-uranium mining in a conservation zone declared within the Region.

Supervising Scientist for
the Alligator Rivers Region

INVESTIGATION OF METHODS FOR THE DETERMINATION OF SUSPENDED SOLIDS IN WATER

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ABSTRACT

A method for the determination of suspended solids in billabong water and Ranger Retention Pond 1 water was evaluated. The study was extended to evaluate methods for the determination of the organic and inorganic portions of suspended solids.

Some of the factors which influence the precision and accuracy of the methods were examined: filter selection; volume of test sample; drying time and ashing time. Error analysis of weighings showed that precision was strongly affected by absorption of moisture onto the glass surface of petri dishes which were used as containers for the filters.

When a test sample volume of 1000 millilitres was taken it was found that the detection limits for the methods were: suspended solids(1.2 mg/L); inorganic residue (0.4 mg/L) and organic residue (1.3 mg/L).

iii) characteristics of suspended material eg. particle size, bacteria, algae;
iv) variations in weight of dried petri dish and filter at equilibrium with the desiccator and laboratory atmosphere.

The variation in factor (iv), these variations can lead to the greatest source of error. The petri dish and filter is transferred from three separate environments:

(a) drying oven/or plasma asher;
(b) desiccator cooling to laboratory temperature;
(c) laboratory balance pan in laboratory atmosphere.

There are changes in moisture absorbed on the surface of the petri dish, membrane filter and suspended solids in the transfer from environments (a) through to (c).

The minimum time of heating in an oven at 105°C to attain constant weight for the membrane filter and suspended solids has to be established. In this investigation, the petri dish and filter were assumed to have attained "constant weight" when no further losses in weight occurred after drying at 105°C for prolonged periods.

A number of trials were investigated covering different aspects of the method for the determination of suspended solids, inorganic residue and organic residue. These are described in the following sections:

3.1 Precision and accuracy of balances used for weighing;
3.2 Type of filter used;
3.3 Determination of total suspended solids using Retention Pond 1 water;
3.4 Determination of organic residue/inorganic residue using Retention Pond 1 water;
3.5 Comparison of weight changes in petri dishes under conditions of drying at 105°C and plasma ashing; and
3.6 Evaluation of blank value in the determination of total suspended solids, inorganic residue and organic residue.

2. EXPERIMENTAL PROCEDURES

2.1 Apparatus

The following apparatus was used: (i) Glass petri dishes, 50 mm diameter with glass lids; (ii) Sartorius model 2434 electronic analytical balance, scale division 0.01 mg used before 24.4.87; (iii) Sartorius model R200D electronic analytical balance, scale division 0.01 mg used after 24.4.87; (iv) Vacuum filtration apparatus, all glass comprising 1 litre Buchner flask, 300 mL funnel, fritted glass filter holder; (v) Mini oven (Labmaster) maintained at a temperature of 105°C; (vi) Low temperature plasma asher, L.F.E. Model LTA-504 with vacuum pump, Dynavac model D330; (vii) Membrane filter, cellulose acetate type, 47 mm diameter, pore size 0.45 micron. Sartorius SM 11106 was found to be suitable; (viii) Measuring cylinder, glass, capacity 500 mL; and (ix) Desiccator with silica gel desiccant.

2.2 Reagents

The following reagents were used: (i) High purity water from Millipore reverse osmosis RO60 unit and Milli-Q ion exchange units were used throughout the procedure to wash filterware and membrane filters; (ii) Extran solution 0.1% W/V, prepared by diluting 10 mL of Extran (phosphate free detergent concentrate) with 10 L of high purity water; and (iii) Nitric Acid 10% V/V, prepared by mixing one volume of BDH Analar grade nitric acid with sufficient high purity water to make ten volumes of the solution.

2.3 Precision and Accuracy of Balances used for Weighing

Two electronic analytical balances (Surtorius) were used for the determination of suspended solids: (i) Model 2434; and (ii) Model R200D. The Model 2434 balance was calibrated using standard masses at the 80g level on 24 April 1987. The Model R200D balance was only available for use after early April 1988.

Ten replicate weighings at the 80g level were measured and the precision and accuracy compared with the balance performance data and the certified mass value. Similarly, the Sartorius model R200D balance was calibrated using 20g and 40g standard masses on 20 April 1988.

The precision in weighing a petri dish on the two balances was compared in the following manner: A petri dish was placed in the air oven at 105°C for 24 hours. The petri dish was then transferred to a desiccator and allowed to cool for 30 minutes. The petri dish was then weighed on the Model 2434 balance. The petri dish was placed in an oven for one hour, cooled and weighed. The test was repeated ten times.

The experiment was repeated using the Model R200D balance. The results are reported in Section 3.1.

2.4 Filter Type

2.4.1 *Blank Test*

The following filters were assessed: (i) Sartorius 11106 cellulose acetate membrane filter (soaked in 10% hydrochloric acid solution) 0.45 μm pore size; (ii) Sartorius 11106 cellulose acetate membrane filter (water washed) 0.45 μm pore size; (iii) Nuclepore 11107 polycarbonate (water washed) 0.4 μm pore size; and (iv) Whatman GF/C glass fibre (water washed).

All filters were rinsed by filtering three portions of 20 mL high purity water in an all glass vacuum filtration apparatus followed by 250 mL of high purity water filtered through each filter type. A replicate determination of the blank was made for sets of membrane filters. In the case of Whatman GF/C glass fibre filters the blank was determined ten times. The time taken to filter 250 mL of water was noted. After filtration, each filter was transferred to a glass petri dish and then placed in an oven to dry at 105°C for two hours. After storing in a desiccator for two hours the petri dish and membrane filter were then weighed. Four more weighings, following reheating and recooling in the desiccator were taken over a 48 hour period.

2.4.2 *Sample Test*

Water from Buffalo Billabong, known to have relatively low suspended solids, was used for the determination of suspended solids using Sartorius and Nuclepore membrane filters. Five replicate determinations were made using a test sample volume of 250 mL. The results for assessment of filters are discussed in Section 5.2.

2.5 Determination of Suspended Solids Using Water of Low Suspended Solids

2.5.1 *Outline*

The method for the determination of suspended solids in water was investigated more thoroughly using another water sample; in this case water from Ranger Retention Pond 1 (RP1). In this experiment six replicate test water samples and two blanks were used. The method outline is as follows: (i) Wash eight Sartorius SM 11106 filters with 500 mL of high purity water; (ii) Transfer filters to separate petri dishes; (iii) Transfer to oven to dry at 105°C; (iv) Dry each filter for successive drying periods to establish constant weight; (v) After each drying period, transfer petri dish to desiccator, cool 30 minutes, then weigh; (vi) Record change in weight in each drying period; (vii) Assess weight changes, then calculate mean weight of each dried filter and petri dish; (viii) Filter 2 x 500 mL deionised water and 6 x 500 mL RP1 through separate filters; (ix) Wash each filter with 50 mL of high purity water; (x) Transfer to oven to dry at 105°C; (xi) Dry for 2 hours, 3 hours, 6 hours, 24 hours, 48 hours; (xii) After each drying period, transfer petri dish to desiccator, cool 30 minutes, then weigh; (xiii) Assess weight changes, then calculate mean weight of dried filter and suspended solids and petri dish; and (xiv) Calculate suspended solids on each filter.

The determination of suspended solids is shown schematically in Figure 1; the weights of the petri dish, filter and suspended solids have been assigned the symbols p, f and ss respectively.

Figure 1.

Schematic outline for determination of total suspended solids:

1. p = weight of petri dish

2. p+f = weight of petri dish and filter

3. p+f+ss = weight of petri dish, filter and total suspended solids.

4. (3) - (2): (p+f+ss) - (p+f) = ss
   Calculate weight of total suspended solids (ss) in grams.

5. Then calculate the concentration of total suspended solids when the volume of sample water filtered is V mL.

$$\underset{(\mathrm{mg/L})}{\text{Total suspended solids}} = T = \frac{ss \times 10^6}{V}$$

The optimum drying times were determined by weighing the filter and petri dish after drying for 2, 3, 4, 23 and 24 hours. At the end of each drying period the results were assessed to determine whether the filter was still losing weight. Constant weight was assumed when the weight varied randomly about a mean value which was accepted as the equilibrium constant weight. The Model R200D balance was used for all these weighing operations.

The experiment was designed to determine suspended solids in a sample and to estimate the contributions to error from weighings, volume measurement and other characteristics of the water sample. The petri dishes used in the experiment were prepared using the following procedure:

2.5.2 *Preparation of Petri Dishes*

1. Mark a paired set of petri dishes and corresponding lids with the same identification number using a diamond tipped pen.

2. Soak the petri dishes in Extran detergent solution, rinse with high purity water.
3. Place petri dishes and lids in a 10% nitric acid bath and soak for 24 hours.
4. Rinse free of acid with high purity water. Place the petri dish and lid in the oven at 105°C. Dry for 24 hours at 105°C.
5. Transfer the petri dishes and lids to a desiccator and cool for 30 minutes. Weigh the petri dishes and lids.

2.5.3 *Calculation of Suspended Solids Concentration*

The weight of total suspended solids on the membrane filter was calculated by firstly deducting the means of the weight of petri dish and filter (M_1) from the corresponding means of the weight of petri dish plus filter plus suspended solids (M_2).

Weight of total suspended solids, (g) = $M_2 - M_1$

Since 500 mL of water was filtered for the blanks and RP1 test water samples the total suspended solids concentration was calculated as follows:

$$\text{Concentration of total suspended solids (mg/L)} = \frac{(M_2 - M_1) \times 10^6}{500}$$

The results for determination of suspended solids in RP1 test water were corrected by deducting the mean of the two blanks.

2.5.4 *Error Analysis in Weighing*

The standard error in weighing a petri dish containing the filter and suspended solids was compared with error in weighing calibrated standard masses. The error was calculated using the following formula:

$$E = \frac{|s|}{\sqrt{n}}$$

where:

s = standard deviation of the mean of a number of mass readings;

n = number of readings; and
E = standard error.

In the determination of suspended solids, group weighings are made at different stages of the procedure. These are as follows:

(i) petri dish;
(ii) petri dish and filter; and
(iii) petri dish and filter and suspended solids.

The errors, due to weighings, may be calculated from the equilibrium dried weights at each stage and compared with one another to show which one is the greatest contributor to the total error. For example, the standard error in weighing the petri dish and filter, designated E_2, may be calculated from:

$$E_2 = \frac{|s_2|}{\surd n_2}$$

where: s_2 = standard deviation; and
n_2 = number of readings

Similarly the standard error in weighing the petri dish and filter and suspended solids is designated E_3 and calculated from:

$$E_3 = \frac{|s_3|}{\surd n_3}$$

where: s_3 = standard deviation; and
n_3 = number of readings

The total error in determining the weight of suspended solids E_t is given by:

$$E_t = \left\{ \left[\frac{s_2}{(n_2)^{1/2}} \right]^2 + \left[\frac{s_3}{(n_3)^{1/2}} \right]^2 \right\}^{1/2}$$

2.6 Determination of Organic and Inorganic Residue from Suspended Solids

2.6.1 *Outline*

The method for determination of organic and inorganic residue was investigated as a continuation of the experiment for the determination of suspended solids in Retention Pond 1 water (Section 2.5). The suspended solids on a cellulose acetate membrane filter were ignited in the petri dish using an RF plasma asher. The samples were ashed for varying periods of time - 1, 3, 6, 8 and 30 hours. The samples were ashed with the glass lid removed from the petri dish. The positions of samples in the ashing chamber were varied after each ashing period.

After each ashing period the petri dish and ashed residue were placed in a desiccator for 30 minutes. The petri dish was weighed using the Model R200D balance. Plasma ashing was discontinued when no further weight changes occurred. The petri dish was then assumed to contain only the inorganic residue.

The method outline is as follows:

(i) Transfer petri dish containing filter and suspended solids (see Section 2.5) to RF Plasma Asher;

(ii) Ash for periods of 1 hour, 3 hours, 6 hours, 8 hours and 30 hours;

(iii) At the end of each ashing period, transfer petri dish to desiccator, cool 30 minutes, then weigh;

(iv) Record weight and weight change, assess, then calculate mean weight of ashed petri dish; and

(v) Remove the ash from each petri dish using a clean lint-free tissue. Weigh the petri dishes again. The difference in weight is equal to the weight of inorganic residue.

A schematic outline of the steps involved in the calculation of inorganic and organic residue is shown in Figure 2. The weights of the petri dish, suspended solids, inorganic residue and organic residue have been assigned the symbols p, ss, i and o respectively.

Figure 2.

Schematic outline of the determination of inorganic residue and organic residue in suspended solids.

After ashing:

1. p + i = Weight of petri dish and inorganic residue (organic residue and filter lost in ashing process) in grams.
2. p = Weight of petri dish (ash removed) in grams.
3. i = Weight of inorganic residue in grams.
4. (1) - (2): (p + i) - (p) = i

   ∴Calculate weight of inorganic residue then calculate inorganic residue concentration (mg/L) designated as I.

   $$\text{Inorganic Residue} = I = \frac{i \times 10^6}{V}$$ where V = volume, in millilitres of test sample
5. T = I + O
   Suspended solids = inorganic residue plus organic residue.
   Rearrange O = T - I
   then calculate concentration of organic residue (mg/L).

2.7 Comparison of Weight Changes in Petri Dishes under Conditions of Drying at 105°C and following Plasma Ashing

2.7.1 *Outline*

The aim of this experiment was to determine if there were significant differences in the weight of petri dishes under conditions of ashing and drying. In the method for determination of inorganic residue (Section 2.6) the petri dish and ashed residue are transferred directly from the plasma asher to a desiccator to cool for thirty minutes before weighing. This procedure is in contrast to that used for the determination of suspended solids (Section 2.5) where the petri dish and filter are transferred from a drying oven at 105°C to a desiccator to cool for thirty minutes before weighing. It has to be clearly established whether it is necessary to transfer the petri dish and ashed residue from the plasma asher to the drying oven at 105°C, followed by cooling in a desiccator, before weighing.

2.7.2 *Procedure*

Five petri dishes were prepared and dried for two hours in an oven at 105°C. The petri dishes were transferred to a desiccator and cooled for thirty minutes. The petri dishes were weighed on the Model R200D balance. The petri dishes were transferred back to the oven and dried at 105°C for a further 24 hours. The petri dishes were cooled in a desiccator as before for thirty minutes then weighed again. The mean weight of the petri dishes was calculated.

The petri dishes were then transferred to the RF plasma asher and ashed for 1, 3, 6, 8 and 30 hours. The weight losses after each successive ashing operation were assessed to see whether an equilibrium weight was achieved.

The mean of the equilibrium weight was calculated. An outline of all steps in the procedure is described as follows: (i) Dry five petri dishes at 105°C in the oven for 2 hours and 24 hours; (ii) At the end of each heating period, transfer to desiccator, cool thirty minutes; (iii) Transfer to desiccator, cool thirty minutes; (iii) Record weight, assess weight change, determine mean weight of dried petri dish; (iv) Transfer to plasma asher; (v) Ash for periods of 1 hour, 3 hours, 6 hours, 8 hours and 30 hours; (vi) At the end of each ashing period, transfer petri dish to desiccator, cool thirty minutes, then weigh; and (vii) Record weight and weight change, assess, then calculate mean weight of 'ashed' petri dish.

A schematic outline of the procedure is shown in Figure 3.

Figure 3. Schematic outline of trial on plasma ashing of petri dishes containing no membrane filter.

Drying Oven at 105°C		
1.	w_1	Clean petri dish. Dry for 2 hours at 105°C, transfer to desiccator 30 minutes, weigh.
2.	w_2	Dry for 24 hours at 105°C, transfer to desiccator for 30 minutes, weigh.
3.		Transfer petri dish to plasma asher.
4.	w_3	Ash for 1 hour, transfer to desiccator 30 minutes then weigh.
Plasma Asher		
5.	w_4	Ash for 3 hours, transfer to desiccator 30 minutes, then weigh.
6.	w_5	Ash for 6 hours, transfer to desiccator 30 minutes, then weigh.
7.	w_6	Ash for 8 hours, transfer to desiccator 30 minutes, then weigh.
8.	w_7	Ash for 30 hours, transfer to desiccator 30 minutes, then weigh.
		Ashed weight of petri dish = $w_8 = (w_3 + w_4 + w_5 + w_6 + w_7) \div 5$
		Compare w_3 and w_8.

2.8 Derivation of Blank Value in the Determination of Total Suspended Solids, Inorganic Residue and Organic Residue

The aim of this experiment was to determine the detection limits for the determination of total suspended solids, inorganic residue and organic residue.

Ten blank determinations of total suspended solids were performed using one litre of high purity water. The method is described in Section 2.5. Sartorius type SM 11106 cellulose acetate filters were used and all weighings were made using the Model R200D balance.

Initially the filters were washed with 500 mL of high purity water; dried for 16 hours; cooled in a desiccator for 30 minutes; and weighed. After filtration of the sample (1000 mL high purity water), the filters were dried for 4 hours; cooled for 30 minutes in a desiccator; and weighed.

After the determination of total suspended solids the filters were ashed using the RF plasma asher to determine inorganic residue. Organic residue was determined by calculating the difference between suspended solids and inorganic residue.

The method was the same as that described in Section 2.7. The petri dishes containing the filters were ashed for 17 hours.

3. RESULTS AND DISCUSSIONS

3.1 Precision and Accuracy of Balances used for Weighing

The results of balance calibrations are shown in Table 1.

Table 1.

Balance	Model 2434	Model R200D	
Date of calibration	24 April 1987	20 April 1988	
Standard mass (g)	80g	20g	40g
Certificate reading mass (g)	80.00047	20.00009	40.00024
Balance reading (g) (mean of 10 weighings)	80.00039	20.00011	40.00029
Standard deviation (g)	±0.00007	±0.00001	±0.00002
Correction to reading (g)	+0.00008	-0.00002	-0.00005

The performance of the Model R200D balance was superior to that of the Model 2434 in precision and accuracy. The standard deviation of the mean of ten readings at the 80g level was ±0.00007g which is higher than standard deviations at 20g level (±0.00001g) and the 40g level (±0.00002g) for the Model R200D balance.

Table 2: Precision in weighing petri dishes using two different balances.

Balance	Weight of Petri Dish (g) Model 2434 Balance	Model R200D Balance
Weighing		
1	30.67937	30.67905
2	30.67887	30.67923
3	30.67904	39.67930
4	30.67915	30.67922
5	30.67894	30.67913
6	30.67907	30.67915
7	30.67915	30.67908
8	30.67921	30.67900
9	30.67900	30.67923
10	30.67941	30.67927
11	30.67944	30.67935
Mean weight (g)	30.67915	30.67918
Standard deviation (g)	±0.000192	±0.000109
Standard error (g)	0.000058	0.000033
Total error (g) (2 weighings)	0.000082	0.000047

The precision in weighing a glass petri dish after successive heating is shown in Table 2; results for the precision are shown using the Model 2434 and Model R200D balances. The same petri dish and cover were used for both sets of tests. These results showed that the standard error using Model R200D balance was lower (0.000033 g) than that for the Model 2434 balance (0.000058 g). The corresponding standard errors for two weighings which represent the errors from the weight of petri dish and filter before and after filtration, were 0.000047 g for the Model R200D balance and 0.000082 g for the Model 2434 balance.

3.2 Filter Type

3.2.1 *Blank*

The results from the blank trial using various filters are shown in Table 3. The weight difference, calculated as mean and standard deviation, is shown in column (3) for each filter type. The results show that Sartorius SM 11107 (cellulose acetate type) and Nuclepore 11107 (polycarbonate), which had previously been washed with high purity water, gave the lowest weight differences. These membrane filters would be preferred to Sartorius SM 11106 which had previously been acid soaked and Whatman GF/C filters.

These latter filters had large negative weight differences after filtration; this would suggest that soluble material had been leached from the membrane filter during the filtration of 1000 mL of high purity water. The blank suspended solids determination for each filter type was calculated and the results are given in column (4).

Table 3: Blank trial for filtration of 250 mL high purity water through different types of membrane filters.

Filter Type	Volume filtered (mL)	Weight difference mean ± SD (g)	Suspended solids mean ± SD (mg/L)
Sartorius SM 11106 cellulose acetate (acid soaked)	250	-0.00021 ±0.00010 (n = 2)	-0.81 ±0.41 (n = 2)
Sartorius SM 11106 cellulose acetate (water washed)	250	0.00007 ±0.00003 (n = 2)	0.26 ±0.14 (n = 2)
Nuclepore 111107 polycarbonate (water washed)	250	0.00012 ±0.00003 (n = 2)	0.46 ±0.14 (n = 2)
Whatman GFC glass fibre (water washed)	1000	-0.00053 ±0.00016 (n = 10)	-0.53 ±0.16 (n=10)

Note: n = number of determinations.

3.2.2 *Sample*

The results for suspended solids determination using Buffalo Billabong water are shown in Table 4; the Model 2434 balance was used.

Table 4: Determination of suspended solids in Buffalo Billabong water using different types of filters.

Filter Type	Volume filtered (mL)	Filtration time (min)	Weight difference mean ± SD (g)	Suspended solids mean mean ± SD (mg/L)
Sartorius SM 11106 cellulose acetate (acid soaked)	250	20	-0.00176 ±0.00075 (n = 4)	-7.1 ±3.0 (n = 4)
Sartorius SM 11106 cellulose acetate (water washed)	250	5	-0.00018 ±0.00030 (n = 5)	-0.7 ±1.2 (n = 5)
Nuclepore 111107 polycarbonate (water washed)	250	30	0.00018 ±0.00095 (n = 5)	0.76 ±0.38 (n = 5)

Note: n = number of determinations.

Negative results for determination of suspended solids were obtained using Sartorius SM 11106 acid and water washed filters. It would appear that soaking of these filter types in 10% HCl affected the long term integrity of filter material.

Acid soaked Sartorius SM 11106 Filters were found to be unsuitable because they caused slow filtration and became very brittle after the drying step. There was also considerable variation in the results for suspended solids concentration (-7.1±3.0 mg/L).

Filtration of water through Sartorius SM 11106 filters, as received was much faster. The coefficient of variation was high and some negative results for suspended solids were recorded. This indicated that the method for suspended solids determination adapted from APHA 209C should be modified so that the initial rinsing of the filters should be changed from 20 mL portions of high purity water to a volume of water equivalent to the volume of sample water to be filtered; in any case this volume should be not less than 250 mL. Nuclepore 111107 filters (30 minutes) were slower than Sartorius filters (twenty minutes).

Results for suspended solids determination were poor using the procedure (Section 2.5.1) but when the errors in weighing using Sartorius Model 2434 analytical balance are considered a much larger volume of sample eg. 1000 mL would need to be filtered to obtain satisfactory results for suspended solids at the 1 mg/L concentration level.

On the basis of the above results it was concluded that Sartorius SM 11106 cellulose acetate membrane filters as received were suitable because of adequate perfomance, lower cost and shorter filtration time. Each membrane filter should be washed thoroughly with high purity water before filtration using a minimum volume of 250 mL water; the filter should be dried to constant weight in an oven at 105°C. To determine the suspended solids in a water sample containing less than 10 mg/L, a test sample volume of 1000 mL is required.

3.3 Determination of Suspended Solids Using Water of Low Suspended Solids from Retention Pond 1

The petri dish and washed membrane filter were initially dried for 2 hours at 105°C. After 3 hours drying further weight losses occurred ranging from 0.00050 g to 0.00130 g. It was apparent that the petri dish and membrane filter were not completely dried. After successive drying periods of 3, 4, 23 and 24 hours the weight of petri dish and filters showed positive and negative variations about a mean. It was assumed that after 3 hours drying, a constant, equilibrium weight was attained. The equilibrium weight of each petri dish and filter was calculated as the mean of the weights obtained after drying at 3,4, 23 and 24 hours. The results are shown in Table 5. There was considerable variation in standard deviations from the mean weights of petri dishes; the standard deviation ranged from ± 0.00004 to ± 0.00037 grams. For the initial drying of petri dish and membrane filter, a drying period of 4 hours is recommended.

After the samples had been filtered, the petri dish and membrane filter plus suspended solids were subjected to similar drying periods of 2, 3, 6, 24 and 48 hours.

Table 5: Variation in weight of petri dish and washed membrane filter at different drying times (before filtration).

Container marked	B1	5	6	7	14	16	17B2	
Sample	Blank	RP1	RP1	RP1	RP1	RP1	RP1	Blank
	Change in weight (g) from dried weight after 2 hours drying.							
Drying time - hours:								
2	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
3	-0.00130	-0.00072	-0.00060	-0.00074	-0.00103	-0.00050	-0.00108	-0.00091
4	-0.00097	-0.00111	-0.00088	-0.00109	-0.00109	-0.00013	-0.00110	-0.00153
23	-0.00084	-0.00075	-0.00065	+0.00092	-0.00035	-0.00039	-0.00113	-0.00078
24	-0.00059	-0.00098	-0.00054	-0.00108	-0.00104	-0.00089	-0.00118	-0.00141
	Equilibrium weight (g) of dried petri dish and filter n = 4							
mean	26.63666	28.74530	27.87809	25.54353	25.62864	31.96146	21.42072	24.27513
standard deviation	±0.00030	±0.00019	±0.00016	±0.00020	±0.00035	±0.00032	±0.00004	±0.00037

The results for weight change of petri dish and membrane filter plus suspended solids at different drying times are shown in Table 6. It was found that there were no further weight losses after 3 hours drying. There were large variations about the mean for the weight of the petri dish plus filter plus suspended solids. The standard deviations from the means varied between ± 0.00027 to ± 0.00059 grams.

Table 6: Variation in weight of petri dish and membrane filter plus suspended solids at different drying times (after filtration). Test water was from Retention Pond 1 (RP1).

Container marked	B1	5	6	7	14	16	17	B2
Sample	Blank	RP1	RP1	RP1	RP1	RP1	RP1	Blank
	Change in weight (g) from dried weight after 2 hours drying.							
Drying time - hours:								
2	0.00000	+0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
3	+0.00006	-0.00007	-0.00025	-0.00073	+0.00001	-0.00023	-0.00035	+0.00144
6	-0.00034	+0.00020	-0.00052	-0.00139	-0.00125	+0.00023	+0.00029	-0.00095
24	+0.00037	-0.00099	-0.00112	-0.00125	-0.00107	-0.00064	-0.00037	-0.00093
48	+0.00103	+0.00058	-0.00063	-0.00124	-0.00043	+0.00092	-0.00017	-0.00072
	Equilibrium weight (g) of dried petri dish and filter (n = 4)							
mean	26.63619	28.75257	27.88296	25.54908	28.63309	31.96799	21.42847	24.27548
standard deviation	±0.00051	±0.00057	±0.00042	±0.00057	±0.00059	±0.00058	±0.00027	±0.00101
number of determinations	4	4	4	3	4	4	4	4

Note: The volume of sample filtered was 500 mL in each case.

The results for suspended solids concentrations in Retention Pond 1 water are shown in Table 7.

The mean suspended solids concentration was found to be 12 ± 2.7 mg/L (n = 6). The range of the results was 9.0 to 15.6 mg/L. All of the factors mentioned in Section 1 would have contributed to this variation.

The largest contribution to variation is caused by the change in mass of the petri dish and lid changing from one environment to another. The large surface area of the petri dish and lid allow for the opportunity of moisture absorption. However the petri dish is needed to protect the fragile membrane filter during handling.

Table 7: Calculation of suspended solids in Retention Pond 1 water.

Containter marked	B1	5	6	7	14	16	17	B2
Sample	Blank	RP1	RP1	RP1	RP1	RP1	RP1	Blank
M_1 (g)	26.63666	28.74530	27.87809	25.54353	28.62864	31.96146	21.42072	24.27513
M_2 (g)	26.63619	28.75257	27.88296	25.54908	28.63309	31.96799	21.42847	24.27548
$(M_2 - M_1)$(g)	-0.00047	+0.00727	+0.00487	+0.00555	+0.00445	+0.00653	+0.00775	+0.00035
Calculated suspended solids (mg/L)	-0.94	14.54	9.74	11.10	8.90	13.06	15.50	+0.70
mean blank (mg/L)	-0.12	-0.12	-0.12	-0.12	-0.12	-0.12	-0.12	-0.12
Blank corrected suspended solids mg/L	-	14.66	9.86	11.22	9.02	13.18	15.62	-

Note: The volume of sample filtered was 500 mL in each case.

3.3.2 *Error Analysis in Weighing*

The calibration of the Model R200D balance on 20 April 1988 (Table 8) using 0.1 g, 20 g and 40 g standard masses showed that the error in weighing was several orders of magnitude lower than the weight of a petri dish and membrane filter; all less than 0.01 mg.

Table 8: Precision data and error analysis for Model R200D balance.

Reference Mass Nominal Reading (g)	Mean reading ± SD (g) (n = 10)	Relative error (g)
0.1	0.10010 ± 0.0001	0.000003
20	20.00011 ± 0.00005	0.0000016
40	40.00029 ± 0.00002	0.000006

Note: n = number of determinations = 10; s = standard deviation; E = relative error; and $E = s/\sqrt{n}$

The relative error in weighing each petri dish before and after filtration was calculated using the data from Tables 5 and 6. The total error for weight of suspended solids was then calculated. These results are shown in Table 9. Calculation of the relative error (E_1) for weighing each petri dish plus filter ranged from 0.02 to 0.19 mg.

The relative error in weighing the petri dish plus filter plus suspended solids (E_2) was much higher (0.14 to 0.51 mg). There was no significant difference between the total error for the two blanks and six Retention Pond 1 replicates. This indicates that there was no significant difference in the moisture re-absorptive characteristics in the suspended solids of Retention Pond 1 water.

The main contributor to variation in suspended solids determination was the mass changes of the petri dish and lid and not the filter and suspended solids. It is highly probable that variations in homogeneity of test samples would also have contributed to variation as the water contained filamentous algae.

Table 9: Calculation of relative errors and total error in the determination of suspended solids in Retention Pond 1 water

Container marked	Sample	Relative error (g) E_1	Relative error (g) E_2	Total error (g) E_t
B1	Blank	0.00015	0.00026	0.00030
5	RP1	0.00010	0.00029	0.00031
6	RP1	0.00004	0.00021	0.00021
7	RP1	0.00012	0.00029	0.00031
14	RP1	0.00018	0.00030	0.00035
16	RP1	0.00016	0.00029	0.00033
17	RP1	0.00002	0.00014	0.00014
B2	Blank	0.00019	0.00051	0.00054

Mean total error for blanks = 0.00042 ± 0.00017 (n = 2)

Mean total error for RP1 water = 0.00028 ± 0.00008 (n = 6)

Note: E_1 is the relative error in weighing petri dish + filter.

E_2 is the relative error in weighing petri dish + filter + suspended solids.

3.4 Determination of Organic Residue/Inorganic Residue using Retention Pond 1 Water

The organic and inorganic residues were determined in suspended solids as described in Appendix A2. The results for successive weighing of petri dish and residue are shown in Table 10. After one hour ashing there was considerable variation in the amount of organic matter destroyed in each petri dish. In some cases, for example, dishes marked 6 and B2, ashing was almost completed. In dishes marked B1 and 17 ashing was far from completion. After six hours ashing there were no further weight losses. The weight of the petri dish and residue was calculated from the mean of weights obtained after 6 hours, 8 hours and 30 hours ashing (see Table 10).

After 30 hours ashing time and the final weighing of petri dish and residue, the ash was removed using a clean tissue and the petri dish was weighed again. The weight of ash (inorganic residue) in each petri dish was calculated by weight difference before and after ash removal (see Table 11).

The weight of inorganic residue was blank corrected. The mean of the blank residue was used in the calculation. When a test sample of 500 mL was taken the inorganic residue concentration was calculated as follows:

$$\text{Inorganic residue (mg/L)} = (\text{Weight of residue}) \times \frac{1000}{0.5}$$

The results for the concentration of organic residue, calculated as the difference between suspended solids and inorganic residue are shown in Table 12.

Table 10: Variation in weight of petri dish and residue at different ashing times.

Container marked	B1	5	6	7	14	16	17	B2
Sample	Blank	RP1	RP1	RP1	RP1	RP1	RP1	Blank
	A. Change in weight (g) from ashed weight after one hour ashing (g).							
Ashing time - hours:								
1	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
3	-0.05240	-0.01922	-0.00373	-0.01653	-0.05264	-0.02684	-0.05662	-0.00677
6	-0.05290	-0.02063	-0.00403	-0.01722	-0.05323	-0.02777	-0.05885	-0.00668
8	-0.05300	-0.02228	-0.00447	-0.01914	-0.05444	-0.02971	-0.05841	-0.00737
30	-0.05211	-0.02098	-0.00344	-0.01717	-0.05307	-0.02807	-0.05871	-0.00651
	B. Equilibrium weight (g) of ashed petri dish and filter (n = 3).							
mean	26.55854	28.66696	27.79949	25.46401	28.54886	31.88214	21.34443	24.19767
standard deviation	±0.00049	±0.00087	±0.00052	±0.00112	±0.00075	±0.00104	±0.00022	±0.00046
number of determinations	3	3	3	3	3	3	3	3

Table 11:: Calculation of inorganic residue in Retention Pond I water

Container marked	Sample	Weight of petri dish and residue	Weight of petri dish (ash removed)	Weight of residue	Mean of blank	Blank corrected	Inorganic residue
		(g)	(g)	(g)	(g)	(g)	(mg/L)
B1	Blank	26.55854	26.55907	-0.00053	-0.00001	-	
5	RP1	28.66696	28.66622	+0.00074	±0.00074	+0.00075	1.50
6	RP1	27.79949	27.79720	+0.00219		+0.00220	4.40
7	RP1	25.46401	25.46288	+0.00113		+0.00114	2.28
14	RP1	28.54866	28.54828	+0.00058		+0.00059	1.18
16	RP1	31.88214	31.88062	+0.00152		+0.00153	3.06
17	RP1	21.34443	21.34251	+0.00192		+0.00193	3.86
B2	Blank	24.19767	24.19716	+0.00051		-	

Note: Inorganic residue (mg/L) 2.71 ± 1.29 (n = 6)

The results for inorganic residue (Table 11) showed a wide variation 2.7 ± 1.3 mg/L with a range of 1.2 to 4.4 mg/L for the six tests. There was also wide variation in the results for organic residue (Table 12), calculated by difference from the corresponding suspended solids determination: 9.7 ± 2.7 mg/L, range 5.6 to 13.3 mg/L. The results for all three determinations: suspended solids, inorganic residue and organic residue did not show proportionate correspondence. Although high results for suspended solids concentration were found in petri dish 5 (14.7 mg/L) and petri dish 17 (15.6 mg/L), the corresponding concentrations of inorganic residue were not high.

The variation in results show that weight changes in the petri dish are consistently high.

Table 12: Summary of results for suspended solids, inorganic and organic residue in Retention Pond 1 water:

Container marked	Suspended solids (mg/L)	Inorganic residue (mg/L)	Organic residue (mg/L)
5	14.7	1.5	13.2
6	9.9	4.4	5.5
7	11.2	2.3	8.9
14	9.0	1.2	8.9
16	13.2	3.1	10.1
17	15.6	3.9	11.7
Mean	12.3	2.7	9.7
S.D.	±2.7	±1.3	±2.7
%RSD	22	48	28
n	6	6	6

3.5 Comparison of Weight Changes in Petri Dishes under Conditions of Drying at 105°C and Plasma Ashing

The mean weight of the petri dishes after 24 hours of drying in an air oven at 105°C is shown in Table 13A. Further variations in weight after successively different periods of ashing the petri dishes were recorded in Table 13B. The equilibrium weight of the petri dishes was attained after 6 hours of ashing. The mean equilibrium weight was calculated on the weights at 6 hours, 8 hours and 30 hours of ashing.

A comparison of the means of the equilibrium weights of the petri dishes under drying and ashing conditions can be made by comparing corresponding weights from the results in the last columns of Table 13A and Table 13B. These results show that the mean weights of all the petri dishes was higher under drying conditions. The standard deviations under drying conditions was higher than those under ashing conditions. Weight differences were apparent but they were not considered significant. It was concluded that the transfer of petri dishes to a drying oven, after the ashing step was not necessary.

Table 13:

Container marked	Weight of petri dish, g, 2 hours	Weight of petri dish, g, 24 hours	Weight of petri dish mean ± SD, g
A. Weight of petri dish after drying in oven at 105°C for 2 hours and 24 hours.			
2	29.23514	29.23446	29.23450 ± 0.00048
5	30.75877	30.74981	30.75929 ± 0.00074
11	31.23340	31.23227	31.23284 ± 0.00080
13	29.23454	29.23388	29.23421 ± 0.00047
18	25.41775	25.41701	25.41738 ± 0.00052

B. Change in weight of petri dish after ashing in plasma asher for different periods.

Ashing Time (h)	0	1	3	6	8	30	Change in weight from dried weight in oven at 105°C for 24 hours mean ± SD g (n = 3)
Container Marker							
2	0.00000	-0.00093	-0.00148	-0.00113	-0.00105	-0.00077	29.23382 ± 0.00019
5	0.00000	-0.00037	-0.00058	-0.00068	-0.00058	-0.00034	30.75876 ± 0.00017
11	0.00000	-0.00116	-0.00149	-0.00096	-0.00092	-0.00066	31.23199 ± 0.00016
13	0.00000	-0.00091	-0.00126	-0.00102	-0.00096	-0.00060	29.23335 ± 0.00022
18	0.00000	-0.00057	-0.00076	-0.00085	-0.00077	-0.00048	25.41668 ± 0.00019

3.6 Evaluation of blank value in the determination of total suspended solids, inorganic residue and organic residue

The results for determination of total suspended solids in high purity water used as a blank are shown in Table 14. The mean blank concentration was 0.76 mg/L with a standard deviation of ± 0.34 mg/L. The detection limit for total suspended solids was calculated as 1.2 mg/L. In a similar manner, the detection limit for inorganic residue was calculated as 0.22 mg/L and for organic residue as 1.3 mg/L (Table 14). These results do not take into account the nature of the sample and the error in test sample volume measurement.

Table 14: Summary of results for blank determination of total suspended solids, inorganic residue and organic residue in high purity water.

Container marked	Suspended solids (mg/L)	Inorganic residue (mg/L)	Organic residue (mg/L)
A	-0.61	0.68	-1.09
B	0.46	0.31	0.15
L	0.41	0.28	0.13
4	0.45	0.26	0.19
8	1.32	0.54	0.58
19	0.72	0.15	0.57
21	1.0	0.27	0.73
22	1.11	0.37	0.74
23	-0.37	0.57	-0.94
24	0.59	0.36	0.13
Mean	0.51	0.32	0.12
Number of determinations	8	8	8
Standard deviation (SD)	±0.61	±0.11	±0.64
Detection limit (=2SD)	1.22	0.22	1.28

4. OVERALL SUMMARY AND CONCLUSIONS

On the basis of the tests which have been carried out, methods for the determination of suspended solids and inorganic/organic residue have been prepared and set out in the Appendix as follows:

Appendix A1: Water, Determination of Total Suspended Solids, Gravimetric Method.

Appendix A2: Water, Determination of Inorganic and Organic Residue in Total Suspended Solids, Gravimetric Method.

The methods have been written to describe how precision can be improved. This can be achieved by using the following key steps in the procedures:

1. Use Sartorius SM 11106 membrane filters directly from the box;
2. Wash the filters with a minimum volume of 250 mL of high purity water;
3. Dry the petri dish and filter for a minimum period of 3 hours to attain constant weight;
4. Cool the petri dish and filter for 30 minutes in desiccator;
5. Ash the petri dish and filter for a minimum period of 6 hours ensuring that the lid is off the petri dish; and
6. Transfer the petri dish and residue after ashing is complete to a desiccator and cool for 30 minutes before weighing.

It was quite apparent however, that the detection limit for suspended solids, inorganic residue and organic residue was approximately 0.5 mg/L when 1000 mL of sample water was filtered. When the sample contained less than 2 mg/L suspended solids, further differentiation of solid concentrations into inorganic and organic residue concentrations is extremely difficult due to the poor precision. It has been shown that the loss of precision was caused by weight change in petri dishes due to absorption of atmospheric moisture onto the large glass surface.

A comparison of the standard deviations in weight of petri dishes and contents in all steps of the procedures showed that weight differences can be up to ± 1.0 mg approximately. This is shown in Table 15.

Table 15: Standard deviation of all weighings which involve the use of glass petri dishes.

Weighing Operation	Standard Deviation Range (mg)	Refer to
petri dish + filter	0.04 to 0.37	(Table 5)
petri dish + filter + suspended solids	0.27 to 1.01	(Table 6)
petri dish + inorganic residue	0.22 to 1.12	(Table 10)
petri dish (dried at 105°C)	0.47 to 0.80	(Table 13A)
petri dish (ashed)	0.16 to 0.22	(Table 13 B)

Note: Model R200D balance was used.

The calibration of the Model R200D balance on 20 April 1988 using standard reference masses showed that: (i) at 20 g the standard deviation was ± 0.00001 g (n = 10); and (ii) at 40 g the standard deviation was ± 0.00002 g (n = 10).

The standard deviations using reference masses were considerably lower than for weighings of petri dishes under the conditions of the tests. The high variation in weighing glass petri dishes in all phases of the test leads to loss of precision. Precision in test results could be improved by filtering a larger volume of sample. However, in the case of Retention Pond 1 water under test, 500 mL of sample took four hours to pass through the membrane filter; filtration of 1000 mL would have taken considerably longer.

Precision may be improved by avoiding the use of glass petri dishes in the weighing operations. It is recommended that the petri dish should be used simply as a transport container for the filter and suspended solids/ash from the balance to the oven/plasma asher. Ashed residue could also be removed from the petri dish using a balance brush.

5. REFERENCES

APHA 209C (1985). Standard Methods for the Examination of Water and Waste Water, A.P.H.A., 16th Edition, Washington D.C.

APPENDIX A1

WATER

DETERMINATION OF TOTAL SUSPENDED SOLIDS

GRAVIMETRIC METHOD

OFFICE OF THE SUPERVISING SCIENTIST

ALLIGATOR RIVERS REGION RESEARCH INSTITUTE

ANALYSTICAL CHEMISTRY SECTION: METHODS MANUAL

WATER, DETERMINATION OF TOTAL SUSPENDED SOLIDS, GRAVIMETRIC METHOD

METHOD: ARRRI 12	APPROVED: 16.10.88
REFERENCE: A.P.H.A. 209D	REVISED: 25.11.88
REGISTRY FILE NO: JP/07/19	AUTHOR: P. CUSBERT

SCOPE AND APPLICATION

1.1 The method is suitable for all types of water. The amount and type of particulate matter in the sample will influence the volume of test sample to be filtered.

1.2 Suspended solids is defined as the particulate matter retained on a membrane filter (with 0.45 micron pore size) after filtration of a water sample. The solids are dried to constant weight at 105°C.

1.3 The method is subject to the following interferences:

1.3.1 Suspended solids dried at 105°C may be expected to retain not only water of crystallisation but also some mechanically occluded water. Because the expulsion of occluded water is slow at 105°C attainment of constant weight is very slow.

1.3.2 Some dried suspended solids readily absorb moisture. Rapid weighing is essential to this method.

1.4 The range of the method is 1 - 200 mg of suspended solids retained on the filter. For samples which containing low suspended solids 1 to 10 mg/L , a test sample volume of 1000 mL is recommended. For samples which contain greater than 10 mg/L a smaller test sample volume may be chosen.

2. PRINCIPLE

2.1 The sample is passed through a weighed filter and the filter with its residue is oven dried at 105°C. The increase in weight of the filter represents the suspended solids.

3. REAGENTS AND MATERIALS

3.1 General Requirements

3.1.1 High purity water should be used at all times throughout the procedure.

3.1.2 Extran detergent solution (0.1% V/V) for cleaning filter equipment.

3.1.3 Nitric acid (10% V/V) for cleaning filter equipment.

4. APPARATUS

4.1 Glass petri dishes, 50 mm diameter with glass lids.

4.1.1 Mark each dish and lid with a number using a diamond tipped pen.

4.2 Analytical balance, capable of weighing to 0.01 mg.

4.2.1 Sartorius Model R200D research balance is suitable.

4.3 Vacuum filtration apparatus, all glass comprising Buchner flask, graduated 300 mL capacity funnel, fritted glass filter holder, and spring action holding clamp.

4.4 Drying oven, maintained at 105°C.

4.4.1 A Labmaster mini oven is suitable.

4.5 Membrane filter, 0.45 micron pore size, 47 mm diameter.

4.5.1 Sartorius SM 11106 filter type made of cellulose acetate is suitable.

4.6 Measuring cylinder, graduated, capacity 1000 mL made of glass.

4.7 Desiccator, glass.

4.7.1 The desiccator must be provided with a desiccant containing a colour indicator of moisture content, eg. silica gel.

5. SAMPLING

5.1 The sample may be collected in polyethylene containers prepared in accordance with Australian Standard AS 2031: Part 1 - 1977, Clause 3.2.2.

5.2 The sample volume collected, should be adequate for satisfactory determination of total suspended solids. The weight of total suspended solids to be retained by the filter should preferably be in the range 1 to 200 mg.

5.3 The sample should be filtered in the laboratory as soon as possible after collection.

6. PROCEDURE

6.1 Preparation of the filter:

6.1.1 Place a membrane filter in the filtration apparatus. Moisten the filter with 10 mL of high purity water.

6.1.2 Fill the funnel with 250 mL of high purity water.

6.1.3 Connect the vacuum line to the Buchner flask and turn on the vacuum pump.

6.1.4 When all the water has passed through the filter, turn off the vacuum pump and disconnect the vacuum line.

6.1.5 Add further volumes of 250 mL of high purity water to the funnel and commence filtering again.

NOTE: The volume of water used for preparing the filter should be equivalent to the volume of test sample or not less than 250 mL.

6.1.6 Transfer the filter to a clean glass petri dish and dry in an oven maintained at 105°C for 4 hours.

6.1.7 Transfer the petri dish and filter to a desiccator and cool for 30 minutes.

6.1.8 Weigh the petri dish and filter using the Sartorius model R200D balance.

6.2 Filtration of the Sample:

6.2.1 Select a suitable sample volume to yield the maximum practical amount of suspended solids.

6.2.2 Transfer the weighed filter prepared in Clause 6.1 to the filtration apparatus.

6.2.3 Moisten the filter with approximately 10 mL of high purity water before clamping the funnel in place.

6.2.4 Shake the sample thoroughly, then pour the well mixed sample into the graduated funnel and commence to filter.

6.2.5 Connect the vacuum line, then turn on the vacuum pump.

6.2.6 When all of the sample has been filtered, rinse the inside of the funnel with approximately 10 mL of high purity water using a wash bottle. Repeat this step.

6.2.7 Turn off the vacuum pump, disconnect the vacuum line, then transfer the filter to the glass petri dish.

6.2.8 Transfer the petri dish containing the filter to an oven maintained at 105°C.

6.2.9 Dry for a minimum period of 3 hours.

6.2.10 Transfer the petri dish containing the filter and suspended solids to a desiccator.

6.2 11 Allow to cool for 30 minutes, then weigh the petri dish containing the filter and suspended solids.

6.3 Blank Test

6.3.1 Carry out all steps in the procedure described in Sections 6.1 and 6.2 using an equivalent volume of high purity water instead of the test sample.

7. CALCULATION

7.1 Calculate the suspended solids concentration in the test sample and blank using the following formula

$$T = \text{Total suspended solids (mg/L)} = \frac{(M_2 - M_1) \times 1{,}000{,}000}{V}$$

where

$M_1 = p + f$ = mass, in grams of membrane filter before filtration
$M_2 = p + f + ss$ = mass, in grams or membrane filter after filtration
V = volume in millilitres of test sample.

7.2 Blank correct the total suspended solids concentration of the test sample using the formula:

Total suspended solids (mg/L) = $T_1 - T_2$
where
T_1 = total suspended solids found in test sample (Section 6.2);
T_2 = total suspended solids found in blank test (Section 6.3).

7.3 Report result to the nearest 1 mg/L. The detection limit of the method when 1000 mL of sample is taken is 0.5 mg/L.

8. PRECISION

8.1 A planned trial of the method was carried out in this laboratory using water from Ranger Uranium Mine retention pond No. 1. The precision of the method in terms of repeatability is shown in Table 1.

Table 1: Precision of method for total suspended solids in water.

Number of Determinations	Total Suspended Solids (mg/L)	Range mg/L	Standard Deviation (mg/L)
1 operator n = 6	12	9 - 16	± 2.7

9. BIBLIOGRAPHICAL REFERENCES

9.1 Standard Methods for the Examination of Water and Wastewater, American Public Health Association, 16th Edition, Washington D.C., 1985.

9.2 AS2031: Part 1: 1986: Recommendations for the selection of containers and preservation of water samples for chemical and microbiological analysis. Part 1 - Chemical (Standards Association of Australia).

APPENDIX A2

WATER

DETERMINATION OF INORGANIC AND ORGANIC RESIDUE IN TOTAL SUSPENDED SOLIDS

GRAVIMETRIC METHOD

OFFICE OF THE SUPERVISING SCIENTIST

ALLIGATOR RIVERS REGION RESEARCH INSTITUTE

ANALYSTICAL CHEMISTRY SECTION: METHODS MANUAL

WATER DETERMINATION OF INORGANIC RESIDUE AND ORGANIC RESIDUE IN TOTAL SUSPENDED SOLIDS

GRAVIMETRIC METHOD

METHOD: ARRRI	APPROVED: 25.11.88
REFERENCE:	REVISED:
REGISTRY FILE NO: JP/07/19	AUTHOR: P. CUSBERT

1. SCOPE AND APPLICATION

1.1 The method is suitable for all types of water and is applied to a water sample after total suspended solids has been measured.

1.2 The method is used for preliminary characterisation of suspended solids in water. The particulate matter in water may contain inorganic and organic components. After removing the organic component using a low temperature plasma ashing technique, the organic residue is calculated from the difference between the total suspended solids and inorganic residue.

1.3 The method is free from interferences.

1.4 The range of the method is 1 - 200 mg of inorganic/organic residue. For samples which contain less than 10 mg/L of inorganic residue a sample volume of 1000 mL is recommended.

2. PRINCIPLE

2.1 The glass petri dish containing a membrane filter and suspended solids (retained from WATER, DETERMINATION OF TOTAL SUSPENDED SOLIDS, GRAVIMETRIC METHOD) is placed in a low temperature plasma asher. After all organic matter has been removed the petri dish and inorganic residue are weighed. Inorganic residue is removed using a tissue and the petri dish is weighed again. The difference in weight represents the inorganic residue. Organic residue is calculated from the difference between total suspended solids and inorganic residue.

3. REAGENTS AND MATERIALS

3.1 General Requirements

3.1.1 High purity water should be used at all times throughout the procedure.

3.1.2 Extran detergent solution (0.1% V/V) for cleaning of petri dishes.

3.1.3 Nitric acid (10% V/V) for cleaning of petri dishes.

3.1.4 Oxygen gas, industrial grade is suitable.

4. APPARATUS

4.1 Low temperature plasma asher:

4.1.1 L.F.E. model LTA-504 is suitable.

4.2 Vacuum pump:

4.2.1 Dynavac model D330 is suitable.

4.3 Analytical balance, capable of weighing to 0.01 mg.

4.3.1 Sartorius Model R200D research balance is suitable.

4.4 Glass petri dishes, 50 mm diameter with glass lids:

4.4.1 Mark each dish and lid with a number using a diamond tipped pen.

4.5 Membrane filter, 0.45 micron pore size, 47 mm diameter:

4.5.1 Sartorius SM 11106 filter type made of cellulose acetate is suitable.

4.6 Desiccator, glass:

4.6.1 The desiccator must be provided with a desiccant containing a colour indicator of moisture content eg. silica gel.

5. SAMPLING

5.1 The sample may be collected in polyethylene containers prepared in accordance with Australian Standard AS2031: Part 1 - 1986, Clause 3.2.2.

5.2 The sample volume collected should be adequate for satisfactory determination of inorganic residue ie. the weight of inorganic residue in the petri dish should preferably be in the range 1 to 200 mg..

6. PROCEDURE

6.1 Test Sample

6.1.1 Connect the oxygen line from the rear of the plasma asher to the laboratory bench outlet.

6.1.2 Connect the vacuum line at the rear of the instrument to the laboratory wall outlet.

6.1.3 Turn on the Dynavac D330 vacuum pump and allow to run for 15 minutes before loading the samples in the cell chambers.

6.1.4 Load the petri dish (containing the filter and suspended solids) into the cell chamber. Ensure that the petri dish lid is off.

6.1.5 Replace the cell chamber lid with metal clamp in place. Ensure that the cell chamber is sealed.

6.1.6 Turn power on (Press WHITE button on). The cells will be evacuated slowly. When the vacuum pressure is 1.0 - 1.5 mm Hg, press RF button (yellow) on.

6.1.7 Turn up RF (forward) power to approximately 200 watts.

6.1.8 Adjust oxygen flow rate to 200 cc/minute and readjust power to 200 watt. Check that REFLECTED POWER is much less than FORWARD POWER. There will be a colour change to blue as oxidation beings.

6.1.9 After one hour of ashing, vent the RF power exhaust gases from vacuum pump to atmosphere, then turn off RF power.

6.1.10 Press the RF button (yellow) off.

6.1.11 Release the vacuum by pressing the green button on the front of the panel.

6.1.12 Remove the petri dishes.

6.1.13 Change the positions of the sample in the chambers in a random fashion. NOTE: This precaution is necessary to ensure that oxidation has occurred in a uniform manner for all samples.

6.1.14 Repeat the procedure in Sections 6.1.5 to 6.1.8 to commence ashing for a further minimum period of five hours.

6.1.15 After a minimum total period of six hours ashing vent the exhaust gases from the vacuum pump to atmosphere.

6.1.16 Turn off RF power.

6.1.17 Press RF button (yellow) off.

6.1.18 Release the vacuum.

6.1.19 Press the green button off.

6.1.20 Transfer the samples to a desiccator. Allow to cool for 30 minutes.

6.1.21 Turn off vacuum pump.

6.1.22 Weigh the petri dish and residue using the analytical balance. Record the weight.

6.1.23 Remove all ash (inorganic residue) from the petri dish using a tissue. Take care not to touch the dish with fingers.

6.1.24 Weigh the petri dish once more using the analytical balance.

6.2 Blank Test

6.2.1 Carry out all steps in the procedure for the test sample (Section 6.1) using the petri dish containing a filter which had previously been used for the blank test in the determination of total suspended solids.

7. CALCULATION

7.1 Inorganic Residue

7.1.1 Calculate the inorganic residue concentration in the test sample and blank using the following formula:

$$I = \text{Inorganic residue (mg/L)} = \frac{(M_2 - M_1)}{V} \times 10^6$$

where

$M_1 = p =$ mass, in grams of petri dish after ash has been removed;

$M_2 = p + i =$ mass, in grams of petri dish plus ash;

V = volume, in millilitres of test sample.

7.1.2 Blank correct the inorganic residue concentration of the test sample using the formula:

Inorganic residue (mg/L) = $I_1 - I_2$

where

I_1 = inorganic residue found in test sample (Section 6.1)

I_2 = inorganic residue found in blank test (Section 6.2).

7.1.3 Report result to the nearest 1 mg/L. The detection limit of the method when 1000 mL of sample is taken is 1 mg/L.

7.2 Organic Residue

7.2.1 Calculate the organic residue of the test sample using the following formula:

O = organic residue (mg/L) = $(T_1 - T_2) - (I_1 - I_2)$

where

T_1 = concentration of suspended solids in test sample (mg/L);

I_1 = concentration of inorganic residue in test sample (mg/L);

T_2 = concentration of suspended solids in blank (mg/L);

I_2 = concentration of inorganic residue in blank (mg/L).

7.3 Report results for inorganic residue and organic residue and organic residue to the nearest 1 mg/L. The detection limit of the method when 1000 mL of test sample has been taken as 0.5 mg/L.

8. PRECISION

8.1 A planned trial of the method was carried out in this laboratory using water from Ranger Uranium Mines Retention Pond No. 1. The precision of the method in this terms of repeatability is shown in Table 1.

Table 1: Precision of method for determination of inorganic residue and organic residue in water.

Number of Determinations		Range (mg/L)	Standard Deviation (mg/L)
	Inorganic residue (mg/L)		
6	3	1-4	±1.3
	Organic residue (mg/L)		
6	10	6-13	±2.7

9. BIBLIOGRAPHICAL REFERENCES

9.1 Standard Methods for the Examination of Water and Wastewater, American Public Health Association, 16th Edition, Washington D.C., 1985.

9.2 AS2031: Part 1; 1986: Recommendations for the selection of containers and preservation of water samples for chemical and microbiological analysis Part 1 - Chemical (Standards Association of Australia).