UNCONTROLLED

WEST VALLEY NUCLEAR SERVICES CO., INC.

ANALYTICAL CHEMISTRY METHOD
ANALYTICAL AND PROCESS CHEMISTRY

ACM-DILU-4301, Rev. 1
Effective Date: 08/02/89

IN-CELL DILUTER/DISPENSER

Approved by:

C. W. McVay, Manager
Analytical and Process Chemistry

Part I

1.0 PURPOSE

To remotely dilute highly radioactive samples in order to transfer from analytical cells to hot laboratory for additional analyses.

2.0 APPLICATION

When highly radioactive supernatant samples need analyses which cannot be performed in-cell, they must be diluted so that the Radiochemistry Laboratory radiation exposure limit of <10 mR/hr is not exceeded.

3.0 DISCUSSION

This method will discuss radiation levels of samples, dilution water preparation, and dilutions using the Microlab Diluter/Dispenser.

4.0 REFERENCES

4.1 MicroLab M Manual, the Hamilton Company
4.2 Density methods ACM-DEN-1802 and ACM-DEN-1801
4.3 Glovebox method ACM-GLOVE-4501

Part II

5.0 EQUIPMENT

5.1 Microlab diluter/dispenser with Treadlite controller S/N MM20660479

5.2 Manipulators

8910260306 891025
PDR PROJ
M-32 PDC

5.3 Paar Models DMA-60 Digital Thermometer and DMA-512 Cell

5.4 Calibrated Thermometer

5.5 Calibrated analytical balance

5.6 Vial bottles

5.7 60 mL bottles

5.8 5 mL plastic beakers

5.9 Smart monitor radiation monitor and probe

6.0 REAGENTS AND STANDARDS

6.1 Nitric Acid, 0.5 M

7.0 SAFETY PRECAUTIONS

7.1 ACP 7.2, Laboratory Safety, must be complied with

7.2 ACP 7.4, Handling Radioactive Materials, must be complied with

8.0 RECORDS

8.1 All measurement data and sample identification shall be recorded on the work sheet (attachment A), and any work sheet needing the proper dilution for calculations.

8.2 Density of dilution water shall be run as per ACM-DEN-1801 and work sheets kept in a QC file.

9.0 CALIBRATION AND CONTROL

9.1 The analytical balance must be calibrated daily when in use, using the internal calibration and an external 50 gram weight. This is recorded in a QC control book.

9.2 The density meter shall be calibrated as per ACM-DEN-1802.

9.3 The Microlab pipetter shall be checked daily when in use, using NaNO-pure water weights and cell temperature as per the calibrated thermometer. See attachment B, table 1, and step 10.2.

10.0 PROCEDURE

10.1 To program the diluter/dispenser:

10.1.1 Enter syringe capacity: 1000 E

10.1.2 Clear all programs: P CE/C ^ E

10.1.3 Name your program #2: P 2 E

10.1.4 1000 1000)3
200 100 200 475 25 E S 10

10.2 Steps to check Microlab diluter/dispenser:

10.2.1 Record cell temperature and density of water at that temperature on attachment B as per table 1. Record density in g/mL. To convert from kg/m^3 to g/mL, multiply kg/m^3 value, from table 1, by 0.001. Example: water at 20.0°C has a density of 998.20 kg/m^3 or 0.9982 g/mL.

10.2.2 Tare a 5 mL plastic beaker on the analytical balance.

10.2.3 Using Microlab diluter/dispenser, program 2, and the Treadlite controller, draw up a 200 λ air gap. Open a vial bottle of NANO-pure water and place under Microlab needle, draw up 100 λ of water. Draw up another 200 λ air gap.

10.2.4 Place tared beaker under needle and aspirate 475 λ (375 λ air and 100 λ sample) into beaker. Weigh this on analytical balance and record weight on attachment B. Expel 25 λ of additional air gap and go through automatic rinse cycle.

10.2.5 Repeat step 10.2.2 through 10.2.4 two more times, for a total of three weight values. Calculate the average and record on attachment B.

10.2.6 To find the volume, the Microlab is delivering, divide the average weight by the density of water at cell temperature, equation is included on attachment B. This is only a check on the Microlab diluter/dispenser.

10.3 To prepare dilution water for in-cell use (see attachment A):

10.3.1 Prepare 0.5 M HNO_3 (32 mL concentrated HNO_3 per litre). Analyze for density as per ACM-DEN-1801. Record density on litre bottle. Record this also on dilution work sheet (attachment A) for density of diluent. Density work sheet for 0.5 M HNO_3 is placed in QC book.

10.3.2 Uniquely label vial bottles (for 10 mL dilution water) and 60 mL plastic bottles (for 50 mL dilution water) with letters or numbers. Correspondingly label dilution work sheets (attachment A) in the space for diluent identifier.

10.3.3 On a calibrated analytical balance, weigh empty capped bottle and record weight on dilution work sheet under weight of container.

10.3.4 Measure 0.5 M HNO_3 with graduated cylinders, 10 mL in vial bottles, 50 mL in 60 mL bottles, record volume on work sheet. Weigh filled capped bottle and record weight in weight of container and diluent on dilution work sheet.

10.3.5 Put dilution bottles in cell either through cell #1 tube or can transfer (see ACM-GLOVE-4501).

10.4 To dilute a highly radioactive sample in-cell:

10.4.1 With aid of Smart Monitor radiation monitor and probe, net radiation levels and dilution size should follow these guidelines:

<40 mR/hr require no dilution
40 to 1000 mR/hr require a 1/100 dilution
>1000 mR/hr require a 1/500 dilution

10.4.2 Whether a 1/100 or 1/500 dilution is used, the same instructions are followed.

10.4.3 Weigh capped dilution bottle, record weight in space for in-cell weight on dilution work sheet for that bottle.

10.4.4 Weight of diluent on work sheet is determined by subtracting the weight of container from the lesser of weight of container and diluent and in-cell weight. This is because 0.5 M HNO_3 could evaporate or spill from bottle or bottles could get solids on them in-cell.

10.4.5 Using Microlab diluter/dispenser and Treadlite controller, draw up an air gap of 200 λ.

10.4.6 Decap sample and place under needle of Microlab, draw up 100 λ of sample. Cap sample bottle with plastic stopper. Draw up 200 λ air gap. Record sample name and log number on dilution work sheet.

10.4.7 Remove cap from dilution (without spilling) and tare uncapped bottle (balance reads 0.0000 g) with bottle on it.

10.4.8 Carefully expel 475 λ into dilution water (375 air gap + 100 λ sample) and place on balance again. Balance will read the weight of aliquot added. Record on work sheet.

10.4.9 Cap dilution bottle tightly and shake well for homogeneity.

10.4.10 If vial bottle of dilution water is used, this may be taken out of cell as is per ACM-GLOVE-4501. If 60 mL dilution bottle is used, empty vial bottles with sample name must be put into cell using Cell #1 tube. Pour diluted sample into vial bottle(s). Save remaining dilution in case more sample is needed in Radiochemistry Laboratory for analysis. Diluted samples may now be taken out of cell as per ACM-GLOVE-4501.

10.4.11 Analyze sample for density as per ACM-DEN-1802 and record result on dilution work sheet in density of sample space.

11.0 CALCULATIONS

$$\text{Dilution} = \frac{(D_d)\ F}{D_s\ (C + F)}$$

where: D_d - density of diluent (g/mL)
D_s - density of sample (g/mL)
F - weight of aliquot added (g)
C - weight of diluent (g)

12.0 ATTACHMENTS

Attachment A - Dilution Work Sheet
Attachment B - Microlab Check
Table 1 - Density of Water

ATTACHMENT A

Page ___ of ___

DILUTION WORK SHEET

DILUENT IDENTIFIER		
SAMPLE NAME		SAMPLE LOG NUMBER
	OUT-OF-CELL	
DILUENT VOLUME (mL)		
WEIGHT OF CONTAINER + DILUENT (g)	B	
WEIGHT OF CONTAINER (g)	A	
WEIGHT OF DILUENT (g/) (B or E) - A = C		
DENSITY OF DILUENT (g/mL)	D_d	
	IN-CELL	
IN-CELL WEIGHT (g)	E	
VOLUME OF ALIQUOT ADDED (mL)		
WEIGHT OF ALIQUOT ADDED (g)	F	
DENSITY OF SAMPLE (g/mL)	D_s	
DILUTION: $\frac{(D_d)\ F}{D_s\ (C + F)}$		

INSTRUMENTS USED: _____

ANALYST _____ DATE _____

APPROVED _____ DATE _____

ATTACHMENT B

MICROLAB CHECK

$$\frac{\text{AVG. WT. (g)}}{\text{DENSITY (g/mL)}} = \text{mL}$$

DATE	WT. 1	WT. 2	WT. 3	AVG. WT.	ANAL. CELL TEMP.	DENSITY H_2O AT TEMP OF CELL	VOLUME DELIVERED	TECHNICIAN	COMMENTS	REVIEWED
	g	g	g	g	°C	g/mL	mL			

Table nsity of water (kg/m³)

Tabelle 1: Dichte von Wasser in kg/m³ in Abhängigkeit von der Celsius-Temperatur in der Internationalen Praktischen Temperaturskala von 1968

t °C	0,0	0,1	0,2	0,3	0,4	0,5	0,6	0,7	0,8	0,9
0	999.84	999.85	999.85	999.86	999.87	999.87	999.88	999.88	999.89	999.89
1	999.9o	999.9o	999.91	999.91	999.92	999.92	999.93	999.93	999.93	999.94
2	999.94	999.94	999.95	999.95	999.95	999.95	999.96	999.96	999.96	999.96
3	999.96	999.97	999.97	999.97	999.97	999.97	999.97	999.97	999.97	999.97
4	999.97	999.97	999.97	999.97	999.97	999.97	999.97	999.97	999.97	999.97
5	999.96	999.96	999.96	999.96	999.96	999.95	999.95	999.95	999.95	999.94
6	999.94	999.94	999.93	999.93	999.93	999.92	999.92	999.91	999.91	999.91
7	999.9o	999.9o	999.89	999.89	999.88	999.88	999.87	999.87	999.86	999.85
8	999.85	999.84	999.84	999.83	999.82	999.82	999.81	999.8o	999.79	999.79
9	999.78	999.77	999.76	999.76	999.75	999.74	999.73	999.72	999.72	999.71
10	999.7o	999.69	999.68	999.67	999.66	999.65	999.64	999.63	999.62	999.61
11	999.6o	999.59	999.58	999.57	999.56	999.55	999.54	999.53	999.52	999.51
12	999.5o	999.48	999.47	999.46	999.45	999.44	999.43	999.41	999.4o	999.39
13	999.38	999.36	999.35	999.34	999.32	999.31	999.3o	999.28	999.27	999.26
14	999.24	999.23	999.21	999.2o	999.19	999.17	999.16	999.14	999.13	999.11
15	999.1o	999.o8	999.o7	999.o5	999.o4	999.o2	999.o1	998.99	998.97	998.96
16	998.94	998.92	998.91	998.89	998.88	998.86	998.84	998.82	998.81	998.79
17	998.77	998.76	998.74	998.72	998.7o	998.68	998.67	998.65	998.63	998.61
18	998.59	998.57	998.56	998.54	998.52	998.5o	998.48	998.46	998.44	998.42
19	998.4o	998.38	998.36	998.34	998.32	998.3o	998.28	998.26	998.24	998.22
2o	998.2o	998.18	998.16	998.14	998.12	998.1o	998.o8	998.o5	998.o3	998.o1
21	997.99	997.97	997.95	997.92	997.9o	997.88	997.86	997.84	997.81	997.79
22	997.77	997.75	997.72	997.7o	997.68	997.65	997.63	997.61	997.58	997.56
23	997.54	997.51	997.49	997.46	997.44	997.42	997.39	997.37	997.34	997.32
24	997.29	997.27	997.24	997.22	997.19	997.17	997.14	997.12	997.o9	997.o7
25	997.o4	997.o2	996.99	996.97	996.94	996.91	996.89	996.86	996.83	996.81
26	996.78	996.76	996.73	996.7o	996.67	996.65	996.62	996.59	996.57	996.54
27	996.51	996.48	996.46	996.43	996.4o	996.37	996.34	996.32	996.29	996.26
28	996.23	996.2o	996.17	996.15	996.12	996.o9	996.o6	996.o3	996.oo	995.97
29	995.94	995.91	995.88	995.85	995.83	995.8o	995.77	995.74	995.71	995.68
30	995.65	995.62	995.58	995.55	995.52	995.49	995.46	995.43	995.4o	995.37
31	995.34	995.31	995.28	995.25	995.21	995.18	995.15	995.12	995.o9	995.o6
32	995.o2	994.99	994.96	994.93	994.9o	994.86	994.83	994.8o	994.77	994.73
33	994.7o	994.67	994.64	994.6o	994.57	994.54	994.5o	994.47	994.44	994.4o
34	994.37	994.34	994.3o	994.27	994.23	994.2o	994.17	994.13	994.1o	994.o6
35	994.o3	994.oo	993.96	993.93	993.89	993.86	993.82	993.79	993.75	993.72
36	993.68	993.65	993.61	993.58	993.54	993.51	993.47	993.43	993.4o	993.36
37	993.33	993.29	993.25	993.22	993.18	993.15	993.11	993.o7	993.o4	993.oo
38	992.96	992.93	992.89	992.85	992.82	992.78	992.74	992.7o	992.67	992.63
39	992.59	992.55	992.52	992.48	992.44	992.4o	992.37	992.33	992.29	992.25
40	992.21	992.17	992.13	992.1o	992.o6	992.o2	991.98	991.95	991.91	991.87
41	991.83	991.79	991.75	991.71	991.68	991.64	991.6o	991.56	991.52	991.48
42	991.44	991.4o	991.36	991.32	991.28	991.24	991.2o	991.16	991.12	991.o8
43	991.o4	991.oo	99o.96	99o.92	99o.88	99o.84	99o.79	99o.75	99o.71	99o.67
44	99o.63	99o.59	99o.55	99o.51	99o.47	99o.43	99o.39	99o.34	99o.3o	99o.26
45	99o.22	99o.18	99o.14	99o.1o	99o.o5	99o.o1	989.97	989.93	989.88	989.84
46	989.8o	989.76	989.71	989.67	989.63	989.59	989.54	989.5o	989.46	989.41
47	989.37	989.33	989.28	989.24	989.2o	989.16	989.12	989.o7	989.o3	988.98
48	988.94	988.9o	988.85	988.81	988.76	988.71	988.67	988.62	988.58	988.53
49	988.49	988.45	988.4o	988.36	988.31	988.27	988.23	988.19	988.14	988.1o
50	988.o5	988.oo	987.96	987.91	987.87	987.82	987.77	987.73	987.68	987.64