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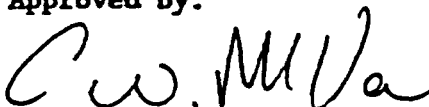
WEST VALLEY NUCLEAR SERVICES CO., INC.

ANALYTICAL CHEMISTRY METHOD
ANALYTICAL AND PROCESS CHEMISTRY

ACM-TS/TDS-2502, Rev. 2
Effective Date: 06/28/89

TOTAL SOLIDS AND TOTAL DISSOLVED SOLIDS BY MICROWAVE OVEN

Approved by:



C. W. McVay, Manager
Analytical and Process Chemistry

Part I

1.0 PURPOSE

This method will be used to quantitatively test an aqueous sample for total solids or a filtered sample for total dissolved solids.

2.0 APPLICATION

This method is designed to accurately analyze a small sample (<5mL) for total solids in a short period of time (<10 minutes).

3.0 DISCUSSION

The AVC-80 is a combination of microwave oven and balance that is a gravimetric moisture/solids measurement system in which a direct determination of weight loss on drying is used to compute the final result. The only conditions necessary to produce accurate, reproducible results are that drying time and power settings be established which ensure that the sample is completely dried to a constant weight, without burning, charring or splattering. The use of this system greatly reduces the time required for analysis of total solids and total dissolved solids.

4.0 REFERENCES

CEM Manual for the Automatic Volatility Computer, Model AVC-80.

Part II

5.0 EQUIPMENT

- 5.1 AVC-80, Automatic Volatility Computer
- 5.2 Various Syringes
- 5.3 Glass Fibre Pads

6.0 REAGENTS AND STANDARDS

- > 6.1 A ten percent Sodium Nitrate (NaNO_3) solution is used for the QC. The Sodium Nitrate used to make the QC solution is to be dried in an oven at 103°C to 105°C for one hour and placed in a desiccator prior to use.

7.0 SAFETY PRECAUTIONS

- 7.1 Do not operate or allow microwave drying oven to be operated with the door open.
- 7.2 Under no circumstances should metal or metallic materials be used for balance pan, stem or sample holder as irreparable damage to the instrument may result.
- 7.3 The AVC-80 should be checked for microwave leakage on a routine basis.
- 7.4 Standard lab safety precautions to be taken (ACP 7.2).

8.0 RECORDS

- > 8.1 All measurement data and sample identification shall be recorded on the worksheet that is printed out automatically by the AVC-80. The final results shall be recorded on the work sheet with the average recorded on the analytical request sheet.

9.0 CALIBRATION AND CONTROL

- > 9.1 A ten gram weight (NBS traceable) shall be used on a daily basis when the AVC-80 is in use. The ten gram used to check the balance in the microwave must be in the range of 9.9950 g to 10.0050 g. When not in this range, take out, rezero and let balance restabilize longer. If weight is still not in range, refer to CEM Manual for the Automatic Volatility Computer, Model AVC-80 and calibrate as necessary (record this change in the QC book with date). The weight shall be recorded in the quality control book to assure that the balance maintains the proper calibration.

- 9.2 A QC standard solution of known concentration will be analyzed before the start of sample analysis. The result shall be recorded on a Quality Control Chart. A result greater than three sigma variation to the known concentration shall be repeated. If a result of more than three sigma variation is noted on the next two repeats, the method is out of control, analysis should be halted, the manager of A&PC notified and the cause of the discrepancy identified and corrected.
- 9.3 The QC standard solution of known concentration shall be checked for accuracy by analyzing the solution using an alternate method (ACM-TS-2501).

10.0 PROCEDURE

- 10.1 Press the orange POWER switch upwards to turn on the unit. The switch button will illuminate, several beep tones will be sounded and the display will show: (SELECT MODE M-)

Press 2 followed by READY. The display will show: (DATE .././..)

The operator may enter the date numerically in any form desired keeping in mind that the numerical equivalents for days, months and years less than 10 must be preceded by a zero. For example, January 9, 1984 would be entered by pressing 0 1 0 9 8 4 READY to enter 1/9/84 in computer memory.

Not entering a date will not affect the operation of the AVC-80. After pressing READY to enter the date the display will show:

(%MOISTURE) will be displayed.

* Solids is desired as the final result, press CHANGE, the display will show:

(%SOLIDS) will be displayed.

Press READY, the display will show: (SET POWER P- %.)

Press 3 0, followed by READY to enter 30% power in computer memory. (Thirty percent power is the standard setting for this method.) The display will show:

(SET TIME-Interval DT- sec.)

This request is for one of the drying curve slope parameters used to define the desired end point of the analysis as mentioned before. It is the time interval in which the sample's weight loss (to be selected in the next step), is equal to or less than the selected amount which will cause the test to be terminated and the

result to be computed. Any time from 0 to 59 seconds may be entered, but press 1, 5, READY to enter 15 seconds in computer memory. The display will show:

(SET WEIGHT-Differential DW= mg.)

This request is for the value of weight loss occurring in the previously selected time interval which will terminate the test.

Press 2, READY. The display will show:

(WEIGHT 0.4064 GMS P-30% T-00.00)

(Weight Value displayed is approximate)

The instrument is now ready to operate in the CWM mode with an end point of 0.2 mg or less weight loss per 15 seconds.

Open the AVC-80 door, place 2 sample pads on the balance pan, close the door and press TARE. This display will show the previous weight plus the weight of the pads and the message: (*....WAIT....*.)

After a few seconds, the beep tone will be heard and the display will show:

(WEIGHT. 0.0000 GMS P-100% T-00.00.)

[If weight does not come out 0.0000 GMS; retare by pressing TARE again until weight does not come out to 0.0000 GMS.]

Open the door, remove the sample pads, transfer a few milliliters sample onto the pads, replace the pads on the balance pan, close the door and immediately press RUN. The display will show an increasing WEIGHT reading along with the *...WAIT...* message indicating the unit is seeking the maximum value of the initial sample weight before starting sample drying.

In a few seconds, the beep tone will be sounded, the LED indicator in the RUN key will illuminate and the time reading will start to increment indicating the start of sample drying.

When the preselected end point is reached, the beep tone will sound, the LED run indicator will go out, the result will be displayed and timer will read the elapsed time of the test.

Press READY to begin the next test.

> 10.2 Samples are done in duplicate; the average to be reported is calculated on the work sheet.

> Deleted

11.0 CALCULATIONS

$$> \quad \% \text{ Solids (wt/wt avg.)} = \frac{\text{Run 1} + \text{Run 2}}{2}$$

12.0 ATTACHMENTS

Attachment A - TS/TDS Work Sheet

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ATTACHMENT A

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TS AND TDS BY MICROWAVE OVEN

Sample Name _____ Log Number _____

Sample Instructions _____

Instrument (Model: S/N) _____

Sample ID					
Run 1 (%)					
Run 2 (%)					
Average (%)					

CALCULATIONS: % Solids Avg. = $\frac{\text{Run 1} + \text{Run 2}}{2}$

Analyst _____ Date _____

Approved _____ Date _____