QAM-I-107

Operation and Calibration of the Block Digestor

Revision 11

Approval:

Laboratory Manager — Date: 8-25-20

Concurrence — Date: 8/24/2020

Effective date: 8-31-20

Renewed 8-31-21 for 8-31-22

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1. Applicability and Purpose

i. This procedure applies to the operation and calibration of the Tecator block digestion systems. It is applicable to any procedure which requires sample digestion temperatures within a range of 50 to 440° C. A uniform operation and calibration method eliminates sample anomalies resulting from digestion error.

2. Definitions

i. Tecator 2040 block digestion system - a set of equipment for digestion, which includes a Tecator 2000 series digestor control pad, a 2040 Tecator block digestor or Tecator Digester 40 Auto, 40 - 100mL Pyrex digestion tubes, and a digestion tube rack with metal safety covers on each end. Newer models may be acquired before the next revision of this QAM, but general temperatures and ramping requirements will still apply.

ii. Standard QA/QC definitions are found in QAM-Q-101 "Laboratory Quality Control". Any changes in method must be accompanied by a standard DOP.

3. Equipment, Reagents, and Standards

i. Equipment
   a. Tecator™ Digestor Auto block digestor
   b. Lachat BD40 digestor and control pad
   c. Digestion tube rack to fit the digestor
   d. Digestion tubes- 100mL, up to 40
   e. Thermometer or thermocouple capable of measuring temperatures within the 50 to 440° C digestor range
   f. Hengar Plain Granules™ or other boiling chips which are capable of enduring the digestion reaction parameters. Mesh size 4 is preferred.

ii. Reagents
   a. Deionized (DI) water
   b. All reagents listed in the standard operation procedure for chemical analysis of the species being digested will be needed.
   c. All standards listed in the standard operating procedure for chemical analysis for the species being digested will be needed.

4. Procedure

i. Calibration

a. Adjust the digestion block temperature to 160ºC.
b. Place the tube rack with safety covers over the digestion block.
c. Place the thermometer into a digestion tube with enough digestion solution or sand to cover the bulb well. Place the digestion tube in one quadrant of the block in a randomly selected well.
d. Allow the digestion block to reach the desired temperature and remain there for 15 minutes.
e. Place the tube in another quadrant and repeat the measurement at the same temperature.
f. Read and record the temperatures measured by the thermometer and average the results.
g. Temperature average is within 5% of the readout on the digital controller with an RPD of 20% between the two readings. Initiate corrective action if it is not.
h. Record the temperature in the Equipment Temperature Logbook (QAM-Q-103-2) and record the results of the calibration in the Maintenance Logbook.
i. Make the proper adjustments in the preprogrammed digestion parameters stored in the controller as part of corrective action to account for any discrepancies in temperature compared to the thermometer.
j. Repeat sections 5.1.2 through 5.1.9 for 98ºC and 380ºC. Sand or high temperature oil may need to be used at 380ºC.
k. Perform this procedure annually.

ii. Operation

a. Place the digestion tubes into the tube rack (up to 40).
b. Place two or more boiling chips into each tube.
c. Prepare a template for the rack and label each tube as a sample, dupe, spike, standard, or blank.
d. In the designated tube add the correct quantity of the sample, DI water, or standard, as indicated by the instructions listed in the standard operating procedure for chemical analysis of the specified analyte(s).
e. Add all of the specified reagents and digestion solutions to each tube.

f. Rinse the reagents, samples, and standards which may be clinging to the side of the digestion tube, into the mixture with DI water.
g. Swirl each tube to mix the digestion reagents with the sample, standard, etc.
h. Place the digestion tubes into the digestor and allow the digestion tube rack to slide down into place.
i. Place the digestion tube rack end covers in their designated slots.
j. Set at the desired temperature, or use a preprogrammed method.
k. Press the "START" key on the controller to begin the digestion reaction program.
l. Allow the digestion to proceed until the digestion procedure is complete. It may be necessary to perform the final dilution phase during the final temperature cycle of the program.
m. Use the “STOP” key to stop the digestion cycle, or turn the controller off.
n. Remove the digestion rack and tubes. Turn off the digestion block.
o. Proceed with the analysis indicated in the standard operating procedure for chemical analysis for the designated species.

5. Quality Control and Safety Aspects

i. All aspects of this procedure comply with QAM-Q-101 "Laboratory Quality Control" and QAM-S-101, "Laboratory Safety".
ii. The analyst treats all samples, reagents, standards, and blanks with equal care and accuracy to ensure consistency in analysis.
iii. The analyst is trained by authorized personnel prior to operating the digestion system.
iv. Prior to performing the digestion, the analyst carefully reads the standard operating procedure for the analyte being digested.
v. The digestion tubes and surface of the block digestor are very hot during operation, do not touch these.
vi. The digestion solutions are always be handled with care.
vii. Prior to performing digestion procedures, the analyst reviews the SDS file for each chemical species utilized.

viii. Newer models of this instrument may have slightly different operating key functions as long as the temperature and times are met.

6. References

i. Tecator™ Digestor User Manual, 1001 3846 / Rev. 4, Foss Analytical AB, Sweden.
ii. Tecator™ Digestor Software Manual, 1001 2957 / Rev. 4, Foss Analytical AB, Sweden.
iii. Lachat BD40^{HT} User Manual, 04/2014, Edition 2, Hach Company.
iv. Methods for Chemical Analysis of Water and Wastes, U. S. Environmental Protection Agency, 1983, Cincinnati, OH, (Method 365.4).
v. Standard Methods for the Examination of Water and Wastewater, ed. A.E. Greenberg, latest online edition, methods 4500 N-org B and 4500 NH3 B (approved 2017).
vi. The National Environmental Laboratory Accreditation Conference Institute (TNI) standard, 2016.
vii. Code of Federal Regulations, 40 CFR 136.

7. Attachments

None