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MILITARY STANDARD

CALORIMETRY FOR THERMAL BATTERY HEAT SOURCES TEST AND CALIBRATION PROCEDURES

FSC MISC

DEPARTMENT OF DEFENSE
Washington, D.C. 20310

Test method and procedure for calorimetry MIL-STD-1454

1. This military standard was developed by the Department of Defense.

2. This military standard is mandatory for use by all Departments and Agencies of the Department of Defense.

3. Recommended corrections, additions, or deletions should be addressed to Harry Diamond Laboratories (AMXDO), Washington, D.C. 20438.

CONTENTS

FIGURES

APPENDIX

1. SCOPE

1.1 Purpose. This standard prescribes a method for testing thermal battery heat source powder samples for calorific values. It includes the method for calibrating the calorimeter. Since thermal batteries must operate over a wide operating temperature range, the heating value expressed as calories per gram of the material must be closely controlled. The methods contained herein specify suitable conditions obtainable in the laboratory which give results equivalent to actual use. They also assure the required reliability of the equipment, and provide for reproducibility of test results. National Bureau of Standards (NBS) certified standard reference material will be used for this purpose. The following standard samples are available:

SRM-1651 -- Powder with Nominal Value of 350 Calories/gram
SRM-1652 -- Powder with Nominal Value of 390 Calories/gram
SRM-1653 -- Powder with Nominal Value of 425 Calories/gram.

Other standard samples will be provided as required.

2. REFERENCED DOCUMENTS

2.1 The following document of the issue in effect on date of invitation for bids or request for proposal form a part of this standard to the extent specified herein.

SPECIFICATIONS

ASTM #116C-68 -- Specification for ASTM Solid-stem Thermometers

3. DEFINITIONS

3.1 Definitions. For the purpose of this standard, the following definitions apply:

3.1.1 Calorific value. Calorific value in this procedure is defined as calories per gram. A gram-calorie is the heat required to raise one gram of water from 14.5°C to 15.5°C.

3.1.2 Certified standard reference material. Certified standard reference material is a powder, certified to have specific calorific value for use as a standard in testing powder samples from the production of thermal batteries. It is also used in calibrating the calorimeter.

3.1.3 Qualified test facility. A qualified test facility is a facility capable of calibrating the instrument for test and certifying by means of a certification report that the calibrated data is correct.

4. GENERAL STATEMENTS

(Not applicable)

5. DETAIL REQUIREMENTS

5.1 Handling precautions. The standard reference material prescribed in this document for calibration purposes is thermodynamically unstable, the components reacting to produce the desired heat. The material is a zirconium-barium chromate mixture which can be ignited readily by flame, spark, friction, or discharge of static electricity. This mixture burns rapidly generating intense heat and solid ash. Both the heat from the reaction and the hot slag which may be thrown from the reaction zone can cause severe burns. However, if proper precautions are taken, this heat powder is no more dangerous than solvents such as acetone and ether. The following instructions, therefore, are recommended for safe-handling of these materials:

a. No flame, smoking, or matches should be permitted in the heat powder area.

b. Working surfaces should be conductive and grounded to prevent accumulation of static charges; personnel should wear grounded wrist straps and avoid wearing silk or synthetic fabrics that can generate static electricity. Cotton clothing is preferred.

c. The area should be kept clean and the amount of heat powder stored in a container or exposed at one time should be limited to 50 grams. It is advisable to break 50 gram samples down to smaller quantities for laboratory use.

d. The heat powder should be kept in covered conductive rubber containers or small metal containers with rubber stoppers. Friction lids should not be used. Desiccators should be made of metal, contain only solid desiccants, and should be placed on a grounded surface before cover is removed to insert or remove a sample.

e. Since heat powder may generate an electrostatic charge when poured, it should be transferred with a grounded metal scoop.

f. Personnel should wear leather gloves and either safety glasses or a face shield while handling heat powder.

5.2 Preparation of the sample.

5.2.1 Since the weight and possibly the chemical composition of heat powder is affected by humidity, it should be dried for two hours at 71°C ± 3°C and ten millimeter (mm) pressure in a flat metal container in a vacuum oven, when cooled and when stored, placed in a desiccator until used. If heat powder is caked, the lumps should be carefully broken up by pressing through a 30 mesh grounded screen with a rubber spatula. Heat powder should never be dried in an oven with open heating coils. The standard samples should be stored in a desiccator over any adequate, dry desiccant such as indicating Drierite, etc.

5.2.2 During the test, the sample should not be removed from the desiccator until immediately prior to weighing.

5.2.3 To keep data for samples of various calorific values in the same order of magnitude as that used to establish the water equivalent, adjust sample weight so that the total calories will be approximately 1200 calories.

5.3 Apparatus.

5.3.1 Test room. Since an appreciable change in room temperature during a test will affect the uniformity of the measurements, the apparatus should be operated in a room or area free from drafts which can be kept at a reasonably uniform temperature or in enivronmental boxes. The apparatus should be shielded from sunlight and radiation from other sources. Since the formula for radiation correction (5.5.4) is based on the premise that during a test the water temperature never exceeds the room temperature, the room temperature for calorimetry should be within 24°C ± 1°C (75°F ± 2°F).

5.3.2 Calorimeter. The calorimeter shall be capable of testing slow-burning thermite type, pyrotechnic mixtures which liberate about 200 to 1000 calories per gram (Parr calorimeter, Model 1411, or equal). It shall be calibrated periodically. See 5.6.3 and 5.6.4.

5.3.3 Thermometer. The thermometer shall be an ASTM #116C, range 19-25C, having an NBS or other qualified test facility calibration report stating corrections to .001C at each 0.5°C interval from 19°C. A magnifying eye piece must be used which will minimize parallax and magnify the scale so the thermometer can be read to the closest 0.001C. An equivalent quartz thermometer may be used.

5.3.4 Argon system. An argon cylinder shall be fitted with a regulator to control the pressure between 25 and 35 pounds per square inch gage (psig). This, with two needle valves and a tee connection is a satisfactory method for allowing the bomb to be alternately filled with argon and exhausted. In this type of arrangement, fasten one needle valve between the pressure regulating valve and the tee connection. Connect the second needle valve to one leg of the tee, and connect the bomb inlet to the other leg, using a minimum length of pressure tubing.

5.3.5 Balances. Two balances may be used for calorimetry. One balance shall be sensitive to .02 milligrams (mg) and shall be accurate to at least ±0.1mg. It shall be checked periodically for accuracy, according to the manufacturer's recommendations, but no less often than every six months. The other balance shall be sensitive to .05 gram for a load consisting of the vacuum flask plus 450 grams of water.

5.3.6 Water. Water containing less than 100 parts per million of impurity shall be used for all determinations including those for calibration.

5.4 Test procedure. The following procedure is prescribed for instrument calibration and highest precision measurements. When the calorimeter is used for production tests, with substantial tolerances, some of the steps may not be needed.

5.4.1 Preparation and loading of the bomb.

a. First, remove any ash that may be left in the gas inlet tube of the bomb from the previous run. This may be done by pushing a piece of wire through the inlet tube or applying a short blast of argon to clear the tube.

b. Attach a seven centimeter (cm) length of fuze wire (Parr #45C7 or equal) to the electrodes of the bomb head assembly as shown in Fig. 1, forming a loop between the two electrodes as shown in the final assembly of Fig. 1.

c. Clean the bomb cup of any residue with a non-abrasive material, then wash cup with warm water and mild soap. Rinse and wipe dry.

d. Weigh the empty bomb cup to 0.1mg, then weigh the sample in the cup to 0.1mg observing all necessary safety practices.

e. Assemble the bomb and tighten the collar with a wrench.

5.4.2 Changing the atmosphere over the sample.

a. Look at the gas inlet tube to be sure it is open.

b. Set the argon regulator to control the pressure between 25 and 35psig, and open both needle valves long enough to flush out the lines with argon.

c. Close both needle valves and connect the pressure tubing to the gas inlet tube of the bomb.

d. With the exhaust valve closed, open the inlet valve slowly then close; open the exhaust valve slowly and close. The bomb has now been flushed once.

e. Repeat step d. an additional five times.

CAUTION: DO NOT OPEN EITHER THE INLET OR EXHAUST VALVES RAPIDLY AS THIS DISTURBS AND SCATTERS THE SAMPLE.

f. Close the gas inlet tube of the bomb and remove pressure tubing.

5.4.3 Preparing the calorimeter water.

a. Fill a clean 500 milliliter (ml) container with water and adjust the water temperature to 18.5°C to 19.0°C.

Note: With the ambient temperature approximately 24°C, the water temperature will rise about 0.5°C from the time the water is drawn and the calorimeter is completely assembled.

b. Pouring slowly to minimize splashing, transfer 450.0 grams ± 0.05 gram of water into the calorimeter flask. Wipe off any water that may have splashed onto the upper portion of the flask. A medicine dropper will facilitate the final weight adjustment.

5.4.4 Assembling the calorimeter.

a. Connect the firing circuit to the bomb and lower the bomb into the water checking to see that the bomb is air tight.

b. Place the cover on the calorimeter having the thermometer extend into the water. For uniformity, the thermometer should always be submerged the same amount. A minimum immersion of 4-5/8 inch is required and may be obtained in the Parr 1411 calorimeter by having the thermometer extend seven inches below the underside of the cover. When the parts are properly positioned, the thermometer should not touch the bomb, the stirrer, nor the wall of the flask.

5.4.5 The test run.

a. Operate the stirrer for at least three minutes before starting temperature readings. The same equilibrium period should be used for all tests to maintain uniformity.

b. Arrange a timer or a clock with a sweep second hand so that readings can be made at prescribed intervals during the test.

c. Record the room temperature within one minute before T_0 and within one minute after T_{18} for verification of ambient conditions.

d. If a glass thermometer is used, tap it solidly with a pencil for a few seconds to insure that the mercury in the thermometer has all settled downin the column before each reading.

e. Although it is possible to determine the calorific value of a sample with only four measurements (water temperature at T_0, T_6, T_{12} and T_{18}), it is recommended that a check be made on the uniformity of the system and the burning rate of the sample by recording the water temperature at the following intervals in minutes starting from T_0:

T_0	T_8	T_{11}	T_{12}
T_3	T_9	$T_{11.25}$	T_{15}
T_6	T_{10}	$T_{11.50}$	T_{18}
T_7		$T_{11.75}$	

f. During the initial rating period, T_6 minus T_3 should be within 0.003°C of T_3 minus T_0. If the difference is greater than 0.003°C, do not fire the bomb. Begin timing again with a new T_0 until the system is stable.

g. Within ten seconds after T_6, the sample shall be fired by completing the circuit.

h. Readings at T_7, T_8, T_9 and T_{10} may be recorded to the nearest 0.01°C. All others should be recorded to the nearest 0.001°C.

i. The 1/4 minute readings between T_{11} and T_{12} are taken to examine for equilibrium before the final rating period begins. If the average increase in temperature during each 1/4 minute interval is not greater than 0.003°C, it may be assumed that the sample burned completely during the interval between T_6 and T_{12}, thereby validating the use of the formula in 5.5.4 for radiation correction.

j. As a check on the uniformity of the final rating period, T_{18} minus T_{15} should be within 0.002°C of T_{15} minus T_{12}.

k. At the completion of the run, exhaust the residual gases, empty the bomb, clean all parts and wipe dry in preparation for the next test. Also run a wire through the gas inlet tube to remove any foreign matter that could clog the gas passage.

5.5 Calculations.

5.5.1 General requirements Subtracting T_6 and T_{12} will give the temperature rise produced by combustion of the sample and fuse, but this includes several other thermal effects for which corrections must be made. In order to calculate the heat of combustion of the sample, it is necessary to determine the true temperature rise due only to combustion of the sample, excluding the heat from the fuse wire, the heat of stirring, and any heat gained or lost by interchange with the surroundings. The steps for calculating the true temperature rise and the calorific value of the sample are given in the following paragraphs (5.5.2, 5.5.3, 5.5.4 and 5.5.5):

5.5.2 Thermometer scale correction. A correction must be applied to readings taken at T_6 and T_{12} to compensate for variations in the thermometer bore and in the graduated scale. These are indicated by the thermometer's calibration. After applying these corrections, subtract T_6 from T_{12} to determine the net corrected temperature rise.

5.5.3 Emergent stem correction. The thermometer is calibrated with the bulb and stem at the same temperature, but the calorimeter is operated with the bulb and a portion of the stem inside the calorimeter while the remainder of the stem is at a different temperature outside the calorimeter. This requires the application of a stem correction (SC) calculated as follows:

$$SC = 0.00016(T_{12} - T_6)(T_{12} + T_6 - I^\circ - t^\circ)$$

where I° is the temperature at which the mercury level in the thermometer is exactly the same as the water level in the calorimeter and t° is the mean temperature of the emergent stem during the calorimetric test. It often can be assumed that $t^\circ = T_{12}$ and if the thermometer extends seven inches below the underside of the cover in the Parr 1411 calorimeter, it can be assumed that $I^\circ = 17.0$, then

$$SC = 0.00016(T_{12} - T_6)(T_6 - I^\circ)$$

Example: Room temperature = 25°C

T_{12} = 23.012

T_6 = 19.528

I° = 17.0

SC = 0.00016(23 - 19.5) (19.5 - 17.0)

SC = 0.00016(3.5) (2.5)

SC = 0.0014

Add the stem correction to the net corrected temperature rise.

5.5.4 Radiation correction. Since the calorimeter always is operated below room temperature, a correction must be applied to account for the heat which enters the calorimeter during the test. This can be determined in several ways, but for simplicity an empirical formula is suggested. This is derived from experimental data and will be accurate for all mixtures which burn as fast or faster than the standard zirconium-barium chromate mixtures. The user is warned that this method may not be accurate for mixtures with extremely slow burning rates which produce time-temperature curves appreciably different from those obtained with the zirconium-barium chromate mixture.

Compute the radiation correction (RC) from the formula:

$$RC = \frac{5(T_{18} - T_{12}) + (T_6 - T_0)}{6}$$

Subtract RC from the net corrected temperature rise.

5.5.5 Calculation of thermal value. The steps to be followed in computing the thermal value of the sample are summarized below. See Fig. 2 for a sample data sheet and Fig. 3 for an example of a completed data sheet.

a. Apply scale corrections to T_6 and T_{12}

b. Subtract T_6 from T_{12}

c. Add the stem correction

d. Subtract the radiation correction

e. Multiply the resulting true temperature rise by the water equivalent of the calorimeter to determine the total heat released in calories.

f. Subtract 1.13 calories (for 7cm Parr fuse wire) to account for the heat from the fuse wire.

g. Divide the weight of the sample in grams to determine the calorific value of the sample in calories per gram.

5.6 **Calibration.**

5.6.1 The calorimeter shall be calibrated (water equivalent established) with a standard sample of heat powder made expressly for calibration purposes and available from NBS. The address is: Office of Standard Reference Materials, National Bureau of Standards, Washington, D.C. 20234. The standard samples come in units containing 50 grams each. The following stocks have been made available: "one unit of SRM 1651, Heat-Source Calorimetry Standard Reference Material" for powder having a nominal heating value of 350 calories per gram; SRM 1652 for a nominal heating value of 390 calories per gram; or SRM 1653 for a nominal value of 425 calories per gram.

5.6.2 Each calorimeter and its component parts will be uniquely identified and used only as a unit. Each calorimeter shall be associated only with the calibration constant (water equivalent) determined for it as a unit. Not only is no modification of the calorimeter required for calibration, but no modification shall be made after the calorimeter is calibrated.

5.6.3 A minimum of ten calibration determinations shall be run to establish the water equivalent; the standard deviation of the result shall be no more than 0.5. A standard deviation greater than 0.5 would indicate a lack of uniformity in the equipment, ambient temperature, or the operator's procedure.

5.6.4 Each calorimeter shall be recalibrated either with ten determinations at six month intervals, or with two determinations at two week intervals, using control charts to monitor the data. The shorter interval is preferable if the calorimeter is in constant use. Whichever time-cycle is observed, the water equivalent shall be established or changed on no less than ten determinations. If water equivalent changes more than two sigma from the previous calibration, stop and make ten runs with standard powder to re-establish the water equivalent.

Custodians:
Army - MU

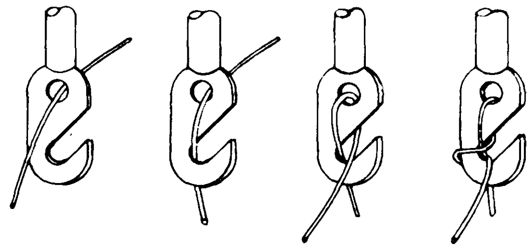
Reviewer:
Army - MU

User:
Army - MU
Navy - NM

Preparing Activity:
Army - MU

Project No.-
Misc - 0741

Steps in binding Fuse
Wire to an Electrode.



1 2 3 4

Final Assembly to
Both Electrodes.

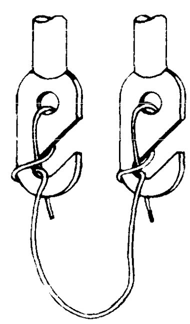


Figure 1

CALORIMETER DATA SHEET

TOTAL WEIGHT ______________ SAMPLE ______________

CUP WEIGHT ______________ DATE ______________

SAMPLE WEIGHT ______________ ROOM TEMP ______ °C ______ °C

TEMPERATURE READINGS

T_0 ________ Δ Δ

T_3 ________

Fire Sample at T_6 ________
C* ________
= ________

T_7 ________
T_8 ________
T_9 ________
T_{10} ________
T_{11} ________ Δ

11.25 ________

11.50 ________

11.75 ________

T_{12} ________
C* ________
= ________
________ Δ

T_{15} ________

T_{18} ________

CALCULATIONS

T_{12} corrected	________
Minus T_6 corrected	________
Temp. Rise, DT	________
Plus SC	+ ________
Equals DT + SC	= ________
Minus RC	- ________
True Temp. Rise	= ________
Times Water Equiv.	x ________
Total Calories	= ________
Minus Fuse Correction	- 1.13
Equals Correct Total	= ________
Divide by Sample Weight	÷ ________
Equals Calorific Value	= ________ cal/g

STEM CORRECTION = SC

**T_6	________
Minus	- 17.000
Equals	= ________
Times	x .00016
Equals	= ________
Times DT	x ________
Equals SC	= ________

RADIATION CORRECTION = RC

**T_6	________
Minus T_0	- ________
Equals A	= ________
T_{18}	________
Minus** T_{12}	- ________
Equals	= ________
Times	x 5
Equals	= ________
Add A	+ ________
Equals	= ________
Divide by	÷ 6
Equals RC	= ________

*Correction from Thermometer Chart.
Add or subtract for corrected reading.
**Use uncorrected reading.

Figure 2

CALORIMETER DATA SHEET

TOTAL WEIGHT 60.9948
CUP WEIGHT 58.0625
SAMPLE WEIGHT 2.9323

SAMPLE STD H425-46
DATE 31 October 1969
ROOM TEMP 23.2°C 23.3°C

TEMPERATURE READINGS

		Δ	Δ
T_0	19.609		
		.023	
T_3	19.632		.001
		.022	

Fire Sample at (.652 - .658): T_6 19.654; C* + .007; = 19.661

		Δ	Δ
T_7	21.85		
T_8	22.02		
T_9	22.05		
T_{10}	22.057		
T_{11}	22.061		
		.002	
11.25	22.063		
		.000	
11.50	22.063		
		.001	
11.75	22.064		
		.001	

T_{12} 22.065; C* +.005; =22.070

		Δ	Δ
		.011	
T_{15}	22.076		0
		.011	
T_{18}	22.087		

CALCULATIONS

T_{12} corrected	22.070
Minus T_6 corrected	19.661
Temp. Rise, DT	2.409
Plus SC	+ .0010
Equals DT + SC	= 2.4100
Minus RC	- .0258
True Temp. Rise	= 2.3842
Times Water Equiv.	x518.33
Total Calories	=1235.8024
Minus Fuse Correction	- 1.13
Equals Correct Total	=1234.6724
Divide by Sample Weight	: 2.9323
Equals Calorific Value	=421.06 cal/g

STEM CORRECTION = SC

**T_6	19.654
Minus	- 17.000
Equals	= 2.654
Times	x .00016
Equals	= .00042
Times DT	x 2.409
Equals SC	= .0010

RADIATION CORRECTION = RC

**T_6	19.654
Minus T_0	- 19.609
Equals A	= .045
T_{18}	22.087
Minus **T_{12}	- 22.065
Equals	= .022
Times	x 5
Equals	= .110
Add A	+ .045
Equals	= .155
Divide by	: 6
Equals RC	= .0258

*Correction from Thermometer Chart.
Add or subtract for corrected reading.
**Use uncorrected reading.

Figure 3

CALORIMETER DATA SHEET

TOTAL WEIGHT (2)
CUP WEIGHT (1)
SAMPLE WEIGHT (3)

SAMPLE ______
DATE ______
ROOM TEMP (4)°C (5)°C

TEMPERATURE READINGS

T_0 ______ Δ (6) Δ

T_3 ______ (10)

(9)

(7) Fire Sample at T_6 ______
(8) C* (15)
= (16)

T_7 ______
T_8 ______
T_9 ______
T_{10} ______
T_{11} ______ Δ (11)
11.25 ______ ______
11.50 ______ ______
11.75 ______ ______
______

T_{12} ______
C* (17)
= (18) Δ

(12)
T_{15} ______ (14)
(13)
T_{18} ______

*Correction from Thermometer Chart.
Add or subract for corrected reading.
**Use uncorrected reading.

CALCULATIONS

T_{12} corrected		(19)
Minus T_6 corrected		(20)
Temp. Rise, DT		(21)
Plus SC	+	(27)
Equals DT + SC	=	(28)
Minus RC	-	(36)
True Temp. Rise	=	(37)
Times Water Equiv.	x	(38)
Total Calories	=	(39)
Minus Fuse Correction	-	1.13 (40)
Equals Correct Total	=	(41)
Divide by Sample Weight	:	(42)
Equals Calorific Value	=	(43) cal/g

STEM CORRECTION = SC

**T_6		(22)
Minus	-	17.000
Equals	=	(23)
Times	x	.00016
Equals	=	(24)
Times DT	x	(25)
Equals SC	=	(26)

RADIATION CORRECTION = RC

**T_6		(29)
Minus T_0	-	(30)
Equals A	=	(31)
T_{18}		(32)
Minus T_{12}	-	(33)
Equals	=	
Times	x	5
Equals	=	
Add A	+	(34)
Equals	=	
Divide by	÷	6
Equals RC	=	

NOTE: See corresponding item numbers in Appendix A for explanation of the use of this data sheet.

Figure 4

APPENDIX A

NOTES FOR USING DATA SHEET

In Fig. 4, steps for performing the calculations have been numbered in the suggested order of performance. The following corresponding numbers offer explanation of each item:

1. Weight of the empty bomb cup (to 0.1mg).

2. Weight of the cup with the sample in it.

3. The difference between items 1 and 2 is the weight of the sample. Record this figure also on line 42 in the Calculations column.

4. Room temperature within one minute before T_0.

5. Room temperature within one minute after T_{18}.

6. Immediately after T_3, calculate the difference in water temperature from T_0 to T_3.

7. Add the resultant figure from item (6) to T_3. The result is the ideal water temperature to expect for T_6.

8. Since a .003°C fluctuation is allowable, record the ideal temperature for T_6 minus .003 and plus .003. If the water temperature at T_6 does not exceed the limits, the bomb may be fired. No other calculations are required to be done during the test.

9. Calculate the difference between T_3 and T_6 uncorrected.

10. Calculate the difference between item 6 and item 9. This figure will not exceed .003°C. See 5.4.5(f) for purpose.

11. Calculate the difference in water temperature during each 1/4 minute from T_{11} and T_{12}. These four differences should average no more than .003°C (no more than .012°C change from T_{11} to T_{12}). See 5.4.5(i) for purpose.

12. Calculate the difference between T_{12} uncorrected and T_{15}.

13. Calculate the difference between T_{15} and T_{18}.

14. Calculate the difference between item 12 and item 13. This figure should not exceed .002°C. See 5.4.5(j).

15. Add or subtract the thermometer correction to T_6.

16. T_6 corrected. Record this figure also as item (20) in the Calculations column.

17. Add or subtract the thermometer correction to T_{12}.

18. T_{12} corrected. Record this figure also as item (19) in the Calculations column.

19. (In the Calculations column) the figure from item (18).

20. Figure from item (16).

21. DT (difference in water temperature). Record this figure also as item (25) in the Stem Correction column.

22. (In the Stem Correction column) The uncorrected temperature at T_6.

23. Perform the calculation, or eliminate this calculation and the next.

24. Perform the calculation or eliminate this calculation and the previous one.

25. Figure from item (21) in Calculations column.

26. Perform the calculation. Record this figure also as item (27) in Calculations column.

27. (In the Calculations column) the figure from item (26) of the Stem Correction column.

28. The difference in temperature plus stem correction.

29. (In the Radiation Correction column) the uncorrected temperature at T_6.

30. The temperature at T_0.

31. Calculate the difference and record this figure also as item (34).

32. The temperature at T_{18}.

33. The uncorrected temperature at T_{12}.

34. Place here the figure from item (31).

35. RC (radiation correction), record this figure also as item (36) in Calculations column.

36. (In Calculations column) the figure from item (35) of the Radiation Correction column.

37. The temperature rise with the two corrections made.

38. This is the water equivalent for this calorimeter.

39. The resultant total calories

40. This figure of 1.13 calories may be used if the sample was ignited with seven centimeters of Parr fuse wire #45C7 or equal.

41. Total calories.

42. Figure from item (3).

43. The calorific value in calories per gram.