



**NEXT GENERATION CALORIMETERS**

# **DDS CALORIMETERS OPERATIONS MANUAL**

**Firmware Version 8**

**VERSION NO: 8.0  
DATE: October 2023**

**DIGITAL DATA SYSTEMS PTY LTD**



# DDS CALORIMETERS

Scientific Analytical Calorimeter Solutions

## Table of Contents:

<b>1</b>	<b>INTRODUCTION</b> .....	<b>1</b>
<b>2</b>	<b>PURPOSE OF THIS MANUAL</b> .....	<b>1</b>
<b>3</b>	<b>SAFETY</b> .....	<b>1</b>
3.1	SAFE BURNING PROCESS.....	1
3.2	SAFE ELECTRICAL INSTALLATION.....	2
3.3	SAFE HIGH-PRESSURE OXYGEN .....	2
3.4	OVER-PRESSURE ( <i>CAL3K-AP ONLY</i> ) .....	3
3.5	SAFE OXYGEN CONNECTIONS .....	3
3.6	SAFE EXHAUST GAS CONNECTION .....	3
3.7	SAFE VESSEL HANDLING .....	4
<b>4</b>	<b>BASIC KEYBOARD OPERATIONS</b> .....	<b>5</b>
4.1	GENERAL KEYBOARD ACCESS .....	5
4.2	COMMAND EXECUTION .....	6
4.3	COMMAND NUMBER EXECUTION.....	6
4.4	INSTALLED COMMANDS.....	7
<b>5</b>	<b>TESTING</b> .....	<b>7</b>
5.1	TEST CAL3K HARDWARE.....	7
5.2	TEST VESSEL HARDWARE .....	7
5.3	TEST THE FAN .....	7
5.4	TESTING FOR OXYGEN LEAKS ( <i>CAL3K-AP ONLY</i> ).....	7
5.4.1	TEST OXYGEN SUPPLY.....	8
5.4.2	TEST OXYGEN INTERNAL PLUMBING.....	8
5.4.3	TEST VESSEL LEAK.....	8
<b>6</b>	<b>PRINCIPLE OF COMBUSTION CALORIMETER</b> .....	<b>9</b>
<b>7</b>	<b>MODE CHANGES</b> .....	<b>10</b>
<b>8</b>	<b>CONNECTING</b> .....	<b>11</b>
8.1	USB PORT .....	11
8.2	PORT D1 .....	11
8.3	PORT D2 .....	11
8.4	BALANCE PORT .....	11
8.5	USE OF PORT D1 .....	11
8.6	CONNECTING TO A PC WITH A RS232 PORT .....	12
8.7	CONNECTING TO A PC FOR DATA COLLECTION.....	12



8.8	CONNECTING TO A BALANCE .....	12
8.9	CONNECTING FOR REMOTE OPERATION .....	13
<b>9</b>	<b>PRINTING AND OUTPUT .....</b>	<b>13</b>
9.1	OUTPUT PRINTING TO D1 PORT, KEYBOARD INITIATED .....	14
9.2	OUTPUT, NON-PRINTABLE .....	17
9.3	OUTPUT, EXTERNAL INITIATED.....	17
9.4	OUTPUT TO A SPEADSHEET.....	17
9.4.1	TERMINAL EMULATOR .....	17
9.4.2	DDS REMOTE APP.....	18
9.4.3	DDS CAL3K APP.....	18
<b>10</b>	<b>OPERATING CYCLES .....</b>	<b>18</b>
10.1	CYCLE: PREPARATION.....	18
10.2	CYCLE: FILLING ( <i>CAL3K-AP ONLY</i> ).....	18
10.3	CYCLE: INITIAL .....	19
10.4	CYCLE: MAIN.....	19
10.5	CYCLE: END .....	19
10.6	CYCLE: COOL.....	19
10.7	HELP DISPLAY .....	19
<b>11</b>	<b>OPERATIONS .....</b>	<b>20</b>
11.1	LOADING DEFAULT PARAMETERS .....	20
11.2	ENTERING PASSWORD .....	20
11.3	MASS ENTRY.....	20
11.4	MASS ERASING .....	21
11.5	SAMPLE IDENTIFICATION (SID).....	21
11.6	GROUP IDENTIFICATION (GID) .....	21
11.7	BALANCE MASS .....	21
11.8	OPEN LID .....	21
11.9	FIRE MANUALLY .....	22
11.10	ABORT .....	22
11.10	UNITS OF MEASURE .....	22
<b>12</b>	<b>CALIBRATION .....</b>	<b>23</b>
12.1	CALIBRATION CHECK SAMPLE .....	23
12.2	CALIBRATION COMMANDS .....	23
12.3	CALIBRATION CONDITIONS .....	24
12.4	CALIBRATION IN DIFFERENT MODES.....	25
12.5	FACTORY CALIBRATION .....	25
12.6	CALIBRATION FRQUENCY .....	25
12.7	ROUTINE CALIBRATION .....	26
<b>13</b>	<b>SPIKING.....</b>	<b>26</b>
13.1	SPIKE VALUE .....	27
13.2	SPIKE MASS .....	27
13.3	STEP BY STEP ENTERING THE SPIKE MASS.....	27
13.4	EASY CAPSULE SPIKING .....	28
<b>14</b>	<b>CORRECTIONS .....</b>	<b>29</b>
14.1	COTTON CORRECTION.....	29
14.2	SULPHUR CORRECTION .....	29



14.3	MOISTURE CORRECTION .....	29
14.4	GELATINE CAPSULE CORRECTION .....	30
<b>15</b>	<b>FIRING .....</b>	<b>30</b>
<b>16</b>	<b>MISS FIRE .....</b>	<b>31</b>
<b>17</b>	<b>COMPENSATION.....</b>	<b>32</b>
17.1	MOISTURE COMPENSATION .....	32
17.2	FOOD COMPENSATION .....	32
<b>18</b>	<b>LIMITS .....</b>	<b>33</b>
<b>19</b>	<b>TIME AND DATE .....</b>	<b>34</b>
<b>20</b>	<b>CAL3K-AP PRESSURE COMMANDS .....</b>	<b>35</b>
<b>21</b>	<b>PRINTING REAL TIME.....</b>	<b>38</b>
<b>22</b>	<b>CLEARING .....</b>	<b>39</b>
22.1	CLEAR RESULT MEMORY .....	39
22.2	CLEAR HELP EVENTS .....	39
22.3	CLEAR VESSEL CAL HISTORY .....	39
22.4	CLEAR CAL3K MAXIMUM .....	39
22.5	CLEAR VESSEL MAXIMUM .....	40
22.6	CLEAR PRESSURE RECORD.....	40
<b>23</b>	<b>READING .....</b>	<b>40</b>
<b>24</b>	<b>BLUETOOTH .....</b>	<b>42</b>
<b>25</b>	<b>SETTING A STATE.....</b>	<b>42</b>
<b>26</b>	<b>ALL CAL3K COMMANDS.....</b>	<b>44</b>
<b>27</b>	<b>TEMPERATURE RISE.....</b>	<b>55</b>
27.1	VERY SMALL SAMPLE MASS .....	56
27.2	HOW TO MEASURE THE WIRE+COTTON RISE .....	56
27.3	HOW TO MEASURE THE GELATIN CAPSULE RISE .....	57
<b>28</b>	<b>MAINTENANCE.....</b>	<b>58</b>
28.1	DAILY USE .....	58
28.2	MATERIALS TESTED .....	58
28.3	ENVIRONMENT.....	58
28.4	MAINTENANCE SCHEDULE (GUIDELINE) .....	58
<b>29</b>	<b>TROUBLESHOOTING .....</b>	<b>60</b>
29.1	ERROR – CALORIMETER DISPLAYS ##### INSTEAD OF THE RESULT.....	60
<b>30</b>	<b>WARRANTY – FOR NEW PRODUCTS.....</b>	<b>61</b>
30.1	PURPOSE OF PRODUCT WARRANTY.....	61
30.2	OWNER’S WARRANTY .....	61
30.3	EXCLUDED (NON-WARRANTY) PARTS .....	61
30.4	WARRANTY – REPAIRED PRODUCTS .....	62
30.5	GENERAL – WARRANTY VOID.....	62
<b>31</b>	<b>RETURNS / REPLACEMENTS POLICY .....</b>	<b>62</b>
<b>32</b>	<b>WARRANTY REGISTRATION FORM.....</b>	<b>64</b>



## 1 INTRODUCTION

Digital Data Systems (Pty) Ltd (**dds**) specializes in the design and manufacture of scientific bomb calorimeter systems for various types of industries and applications. **dds** bomb calorimeter systems are the most advanced combustion calorimeter systems available today with the inclusion of Artificial Intelligence (AI). The system is used to measure the calorific value of both solids and liquids. **dds** has a wide range of systems to suit different applications and sample requirements.

This manual applies to all systems Firmware V8 and higher:

CAL3K-AP	Automatic Oxygen Filling, 2-4 vessels, 4 modes
CAL3K-A	Manual Oxygen Filling, 2-4 vessels, 4 modes
CAL3K-F	Manual Oxygen Filling, 2-4 vessels
CAL3K-ST	Manual Oxygen Filling, Internal Cooling, 2 vessels
CAL3K-S	Manual Oxygen Filling, Internal cooling, 1 vessel

Please visit our website: [www.ddscalorimeters.com](http://www.ddscalorimeters.com) to find out more.

## 2 PURPOSE OF THIS MANUAL

It serves as a short introduction to the CAL3K system. It serves as a reference for all keyboard and interface operations. It explains the very basics of operation and guides you to perform hardware tests. If a command section applies to a particular system only (like the CAL3K-AP, which has internal oxygen filling) it is clearly marked.

Do not read this manual all at once. It appears to be complicated, but it isn't. Read the section you need and then perform the operation. This is a reference manual.

The difference to the Version 7 manual is small and refer to:

- Operator interface and command structure.
- PC interface, data capturing and remote operations.
- Printing
- Calibration management

## 3 SAFETY

A 'BOMB CALORIMETER' uses a vessel (the bomb) which is filled with high pressure oxygen and the sample material, and then the sample is ignited. The sample burns and the resultant temperature increase of the vessel is measured and converted to a calorific value.

### 3.1 SAFE BURNING PROCESS

During the burning the oxygen temperature rises and the pressure increases. The pressure increase depends on the speed of the burning process and to a lesser extent on the sample volume. The safety is therefore mainly concerned with the burning process.



If the sample explodes, then some sample material is settling unburned on the vessel wall (which makes the result useless) and the pressure rises too high which is un-safe. Therefore:

1. **DO NOT** burn powdery substances by itself loose inside the crucible. They can explode on ignition or will blow out of the crucible during the oxygen filling process. It is preferable to use a gelatin capsule 3K-4-068 or press the sample material into a tablet or pellet form using the DDS Pellet Press Die Set 3K-7-041 and the Arbor Press 3K-7-042.
2. Choose a sample volume according to the following guidelines:

Low calorific value (sugar):	~0.75g
Medium calorific value (Benzoic Acid):	~0.5g
High calorific value (Oil, Fat)	~0.3g
3. Examine the vessel inner wall after a sample burn for any sample residue. If residue is found, then the sample has exploded inside the vessel.
4. **DO NOT** use oil on any part of the calorimeter. It may ignite under pressure and elevate the temperature and may cause destruction of the apparatus.

### 3.2 SAFE ELECTRICAL INSTALLATION

- Connect the safety earth to the EARTH STUD on the back of the instrument. This is required when the external power supply fails.
- Do not open the CAL3K for any reason whatsoever. There are no serviceable parts inside the unit.



**DO NOT TAMPER WITH THE EARTHING - CONSULT A QUALIFIED ELECTRICIAN SO THAT THE EARTHING CAN BE CHECKED.**

### 3.3 SAFE HIGH-PRESSURE OXYGEN

- The type of Oxygen to be used is the standard Industrial Oxygen used for welding. A purity of 99.5% will be suitable.
- Conform to the industry standard of handling procedures for oxygen bottles.
- Secure the bottle against a wall.
- Should the Oxygen supply be in another room, make sure there is a high pressure shut off valve near the CAL3K calorimeter.

Start the oxygen regulator at a very low (Adjustor turned OUT) reading and then adjust it until 30bar = 3000KPA is indicated on the gauge.



**DO NOT USE ANY OTHER PIPE OTHER THAN THE HIGH-PRESSURE PIPE SUPPLIED FOR OXYGEN FILLING.**



**DO NOT USE THE HIGH-PRESSURE PIPE IF IT HAS A SHARP BEND OR KINK, DISCARD IMMEDIATELY**

- FILL THE VESSEL WITH OXYGEN TO A MAXIMUM PRESSURE OF 3000KPA (30 bar=default).**



**NOTE:** With some applications you may need to fill the Vessel to less than 3000KPA e.g., 1500KPA (15 bar), however this is only applicable when you have a sample which is highly combustible, for example powdery samples.



**REMEMBER:** You may fill the Vessel LESS than 3000KPA but NEVER more than 3000KPA.

### **3.4 OVER-PRESSURE (CAL3K-AP ONLY)**

Over-pressure can exist in:

- a) The oxygen feed line by incorrect oxygen regulator setting, or a faulty regulator. The max pressure is 35bar (3500KPa). The filling operation will fill to the “fill target setting” unless the time option is selected. We do not recommend “time filling”.

The CAL3K-AP has a high pressure ‘CUT-OUT’: It deflates the vessel pressure when it reaches 80-90bar. This cut-out is hard wired and is in addition to the settable pressure peak limit (Command=87), which can be turned off.

### **3.5 SAFE OXYGEN CONNECTIONS**

Install the high-pressure oxygen pipe without any sharp bends. A 4mm pipe connection is provided. The oxygen source connection depends on the installation (oxygen regulator or piped in oxygen). It is important that the oxygen connection is secured to the table to prevent ‘swiping’ in case it is cut accidentally.

### **3.6 SAFE EXHAUST GAS CONNECTION**

The exhaust gases should be routed to the outside. The exhaust smells and may contain corrosive gases depending on the sample material. A deflating cap 3K-3-22 (manual deflation) and suitable low pressure plastic hose is provided. The CAL3K-AP has a special exhaust port, and the exhaust plumbing parts are provided as well.



**Note: There is no pressure in the exhaust pipe.**

### 3.7 SAFE VESSEL HANDLING

- Handle the vessel over a table or solid surface.
- DO NOT** drop a vessel.
- DO NOT use any tools** to open or close the vessel. AP: If the vessel fails to deflate use the pre-scribed 'emergency deflate' procedure as outlined later in this manual, which uses the 3K-3-18 EMERGENCY DEFLATE tool.
- DO NOT** use any oil or grease on any of the oxygen fittings or the O-rings. A very small amount of high-pressure (vacuum) grease is allowed on the O-ring to make them pliable. Wipe them clean afterwards.
- Store the vessel with the lid open. Never store a vessel under pressure, always deflate using the de-filler cap / deflating cap.
- Clean the inside of the vessel with paper towel after every use.
- Weigh the crucible every week and discard it when it has lost 10% of its original mass.
- Discard the crucible when the bottom is distorted and extremely discoloured.
- Remove the deflector plate every week and clean the top surface. It does accumulate residue and must be clean regularly.



**NEVER OPERATE ANY UNSAFE VESSEL.**



**NEVER IMMERSE THE VESSEL INTO WATER WHEN COOLING. USE THE 3K-2 AIR COOLER OR ALLOW THE VESSEL TO COOL DOWN NATURALLY.**

- A SUITABLE INSPECTION WARNING PERIOD OF 2000 FIRINGS, AND A STOP OPERATING LIMIT OF 2500 FIRINGS ARE PRE-PROGRAMMED FROM THE FACTORY. THE EXACT WARNING LIMIT DEPENDS ON THE TYPE OF VESSEL.**



**THE VESSEL SHOULD BE INSPECTED AT REGULAR INTERVALS DEPENDING ON THE USE. A SUITABLE INSPECTION PERIOD CAN BE SET (By your agent!) AND THE UNIT WILL ISSUE AN INSPECTION WARNING AND SOON AFTER THIS IT WILL SHUT DOWN THE OPERATION. THE DEFAULT SETTING IS 2500 FIRINGS.**



**NOTE: THE MANUFACTURER (DDS) AND ITS AGENTS CANNOT BE HELD RESPONSIBLE FOR ANY LOSS OR DAMAGE RESULTING FROM NEGLIGENCE OR INCORRECT USE OF THEIR EQUIPMENT. IT IS YOUR DUTY AND RESPONSIBILITY TO CHECK ALL SAFETY ASPECTS OF THE CAL3K SYSTEM REGULARLY.**





## 4 BASIC KEYBOARD OPERATIONS

The **ESC** key serves as a: CLEAR/TERMINATE/ESCAPE

The **ESC** Key is safe to use at any time.

The **ENTER** key is: ACCEPT/ENTER

All other keys function normally!

The FUNCTION KEYS are assigned to re-occurring operations:

F1	Mass Entry
F2	Sample Identification (SID)
F3	
F4	Spike Mass
F5	Balance mass
F6	Open the LID (Electronic locks only)
F7	Test Vessel hardware
F8	Terminate user test, print displayed result.
F9	Test CAL3K hardware
F10	Function or Fault explanation
F11	Saved Fault event explanation (AP, A, F, ST)
F12	

The CAL3K has many keyboard commands of which you need only a few. The most popular and frequent used are readily accessible, the others require a password to get access to. You are welcome to experiment with all un-protected commands as you like. Commands are explained in this manual as follows:

### **6    *PASSWORD ENTRY*    0**

6	=	Command Number Index
Password Entry	=	Command text
0	=	Priority, Open, no password required

All commands with a priority = 1 are password protected (not accessible). Enter the password '**DDS3K**' to make priority = 1 commands visible. The password de-activation is active for 5 minutes.

### 4.1 GENERAL KEYBOARD ACCESS

All keyboard commands are in alphabetic order and the **UP/DOWN** keys will scroll through the commands visible at this password level.

There are several ways to execute a command:



- A) If you know the command then type it. After the first couple of letters and if no ambiguity exists the command is auto-completed. Go to Command Execution.
  - B) Type the first letter of the command and then use the **UP (Down)** keys to show the full command. Once you have the full command on the screen go to Command execution
  - C) Use a function key (when available). Then go to Command execution
  - D) Use the command index (if you know it). Press '**XX**' once, then the command index and the complete command appears on the screen. Then go to Command execution
- NOTE:** The '**XX**' numerical command access switches the priority off and renders the password superfluous.

## 4.2 COMMAND EXECUTION

At this point the full command is shown on your screen. Press **ENTER** to accept this command. Most of the time the command is not executed except in special circumstances which have no consequence.

After acceptance one of the following happens:

- A) The command is executed if it is of no consequence (e.g. Display firing voltage)
- B) A **NO/YES** dialogue for operations with a consequence (e.g. ABORT)
- C) A **YES** or **NO** display indicating the present state (e.g. CALIBRATION ON or OFF)
- D) A number display indicating the PRESENT value

At this point you can either:

- |              |  |
|--------------|--|
| <b>ESC</b>   | Terminate, start fresh   |
|              | Enter a <b>NO, YES, Number, text</b>                                     |
| <b>ENTER</b> | Make the change permanent: The <b>NO, YES, Number, text</b> takes affect |

## 4.3 COMMAND NUMBER EXECUTION

All the keyboard commands are indexed with a number, which is indicated in this manual with each key sequence. This number is used to report a problem to the factory (Software bug) when a unit is used with a foreign keyboard. It bridges any possible language barriers. The command index, Command text and command priority is indicated. Example: CMD=54

**54    D1 PORT SPEED    0**

- |               |   |                                      |
|---------------|---|--------------------------------------|
| 54            | = | Command Number Index                 |
| D1 PORT SPEED | = | Command text                         |
| 0             | = | Priority, Open, No password required |

To execute a command by the number you need to know its Index first. Type '**XX**' once to enable command number execution. No password entry is needed for command number execution. The '**XX**' entry is cleared after the standard password timeout.



#### 4.4 INSTALLED COMMANDS

The different units share the same command structure (and numbering). However, some commands are not installed in your unit. To find out which commands are installed in your unit you have two choices:

- A) Print them. This requires a printer
- B) If you have no printer then type the letter 'A' and scroll up or down to see the commands.

Then type the password (**DDS3K**, see password section) and repeat the above process. You will see many commands (for all possible operations).

### 5 TESTING

#### 5.1 TEST CAL3K HARDWARE

The test is activated by F9 or by:

*13 CAL3K-TEST: 0*

This command tests the CAL3K hardware. Once started the unit will cycle through all tests and will 'hang' on any fault. Individual test can be executed (without cycling through all preceding test) by replacing the 'YES' entry by the test number.

This test should be repeated once a day. The vessel must not be in the well.

#### 5.2 TEST VESSEL HARDWARE

This test is activated by F7 or:

*14 VESSEL-TEST: 0*

The vessel must be in the well.

**CAL3K-AP ONLY** - The oxygen pressure must be connected. All vessel functions are tested, and a summary is displayed. The test must be repeated for each vessel.

#### 5.3 TEST THE FAN

This test **does NOT** apply to the **CAL3K-A/AP/F**. This command puts the FAN on.

*120 PUT FAN ON 1*

#### 5.4 TESTING FOR OXYGEN LEAKS (CAL3K-AP ONLY)

Leaks are easily tested with soapy water, or dishwashing liquid (Bubble water) and a cotton bud. The oxygen plumbing can be separated in to 3 sections:



### 5.4.1 TEST OXYGEN SUPPLY

This CAL3K requires a regulated oxygen supply with approx. 33bar max (30 bar nominal) as indicated on the oxygen regulator. A leak on this line is depleting the oxygen bottle, but doesn't influence the unit's operation:

Check:

- Oxygen bottle, regulator with pressure gauge
- Optional shutoff valve
- Flexible nylon pipe to CAL3K-AP bulkhead or filling station
- Flexible nylon pipe from bulkhead to lid or filling-head
- Filter and throttle jet (not visible)

Open the oxygen bottle and observe that the regulated pressure increases to the prescribed setting. Then close the bottle and observe that the pressure holds. If it decreases even slightly then you need to find the leak by putting some 'bubble water' on each joint.

### 5.4.2 TEST OXYGEN INTERNAL PLUMBING

Replace the filling head nut (grey plastic) with the mushroom look alike blocking device. This blocks the oxygen feed to the vessel. Insert the vessel WITHOUT the lid in to the well and close the lid. Then activate command:

**94    SYSTEM LEAK TEST    1**

This is a service command to check if the system is leaking. The oxygen must be connected, and a vessel must be in the well, lid closed. Activate the test (YES) and the CAL3K will pressurise the system to the PRESS LEAK TEST, CMD=99 value. Then it monitors the pressure and displays it. During the test one can try to find any leak if the pressure is declining. Stop the test with ESC.

Note that the filling is very fast because the filling volume (no Vessel) is very small.

Replace the filling nut again. The filling nut is plastic and must be tighten by hand. Do not use tools and do not 'cross thread' it.

### 5.4.3 TEST VESSEL LEAK

This test INCLUDES the vessel. Depending on the type of vessel used there are O-rings in the vessel lid which could leak and require replacement. Use the same CMD=94 command:

**94    SYSTEM LEAK TEST    1**

Note that the filling is much slower because the vessel is filled. Monitor the pressure and if it is steadily declining than a leak is present. It is normal that the pressure declines rapidly in the beginning and then settles down. This is because a flow restriction is present in the vessel lid.



## 6 PRINCIPLE OF COMBUSTION CALORIMETER

In short, a known substance is burned in a contained environment (Vessel) and the resultant temperature rise is recorded as a calibration. 4 to 16 calibrations make up a full calibration. See calibration management for more details. Then un-known samples are burned and the temperature rise is compared to the calibration and the unknown calorific value is calculated.

The following sequence applies to a determination and a calibration. The broad difference is that the calibration saves more data for further reference.

Here is a more detailed, but short sequence of events, assuming default settings.

1. Approx. 0.5g (0.2-0.8g) is precisely weighed and entered in to the CAL3K (electronic or manual transfer)
2. The vessel is prepared (cotton, crucible, firing wire) and the CAP is closed
3. Manual filling: Place the vessel in the filling station and fill it with oxygen.
4. Place the prepared vessel in to the well and close the lid
5. If this vessel was never calibrated, then the unit will ask you to do so.
  - 5.1 Type **CALIBRATION**
6. **CAL3K-AP automatic filling ONLY:** If everything is fine the unit will go into the filling period, open the fill oxygen solenoid and monitor the filling progress. At the end of the filling process the unit will switch to the INITIAL PERIOD.
7. **ALL manual filling units:** the CAL3K will switch to the INITIAL PERIOD
8. It waits approx. 3 minutes for the vessel to stabilise.
9. After 3 minutes the sample is fired: 25 volts are connected to the firing wire and a very large current passes through it, the wire glows, the cotton ignites and the sample burns.
10. The unit enters the MAIN state (Final Period) and monitors the temperature rise of the vessel.
11. The temperature increases rapidly, peaks at approx. 1 minute and decreases thereafter.
12. At the end of the main period the Result is displayed on the screen and the End period starts.
13. **CAL3K-AP:** the vessel deflates automatically to the exhaust port and the lid opens.
14. **CAL3K-A/F:** The lid opens automatically.
15. **CAL3K-S/ST:** The lid must be opened manually.
16. Remove the vessel for cooling and subsequent new preparation.

### **Notes (all units):**

The Initial period is required to stabilise the vessel temperature. The main (Final, measuring) period measures the temperature rise from firing to the end. During both periods data is captured which are used when the result is calculated.



## 7 MODE CHANGES

The operating mode influences the operating parameters of a model such as:

- Initial timing            Time spend to equalise the vessel before firing.
- Main timing             Time after firing to wait for the temperature to stabilise.
- Compensations         The accumulated temperature loss compensations.
- Others                    Cooling time, filling times, parameters.

All models are parameter (settings) controlled. Some of the parameters can be changed by the user, others are set by the default settings and no change is possible. The operating modes control the temperature behaviour:

### Mode 0            **DYNAMIC**

This mode is a mixture of ISOTHERMAL and ADIABATIC (or ISOPERIBOL). The jacket temperature follows the vessel temperature during the initial period (adiabatic) and remains constant during the main period (Isothermal). Best overall performance!

### Mode 1            **ADIABATIC**

In this mode the jacket temperature follows the vessel temperature. Not popular, but traditional!

### Mode 2            **ISOTHERMAL**

The jacket temperature is static (sort of). Not popular, too slow,

### Mode 3            **FAST DYNAMIC**

Same as mode 0, but faster. Requires better environmental control!

Model	Mode=0		Mode=1		Mode=2		Mode=3	
	Initial	Main	Initial	Main	Initial	Main	Initial	Main
CAL3K_A	4	3	3	5	4	5	4.5	2.6
CAL3K_AP	4	3	3	5	4	5	4.5	2.6
CAL3K_F	4	3						
CAL3K_S	4	3						
CAL3K_ST	4	3						

Each mode (and Vessel) must be calibrated if you intend using this mode. Once calibrated, the mode can be changed without re-calibration.

There is a trade-off between operating speed and accuracy. The decrease in accuracy is dependent on the laboratory conditions and in the number of vessels you use.

The operating mode can be set by:

**82    SET OPS MODE            1**

Each of these modes has its own calibration, which means the vessel(s) must be calibrated in the new mode.



## 8 CONNECTING

Different units have the following ports and default speeds:

Unit	USB	PORTD1	PORTD2	BALANCE
CAL3K-AP	yes	19200	115200	2400
CAL3L-A	yes	19200	115200	2400
CAL3K-F	yes	19200	115200	2400
CAL3K-ST	-	19200	-	2400
CAL3K-S	-	19200	-	2400

### 8.1 USB PORT

It connects with a PC and the  
DDS Windows Software App  
DDS Windows REMOTE App.

Note: An App is required! It will not connect to a USB printer!

### 8.2 PORT D1

This is a RS232 port and good for everything as seen later. Of course, it can be connected to a PC and the DDS Windows App. It is called the printer port because all print commands work on this port, and all units have this port.

### 8.3 PORT D2

This port is a RS232 port **only available on the CAL3K-AP/A/F systems**. Its speed is set at 115.2KB. Its main purpose is to connect to a BLUETOOTH bridge, but it can connect to a PC. Printing is not possible.

### 8.4 BALANCE PORT

Again, this is a RS232 port. Its purpose is to receive the sample mass from the balance.

### 8.5 USE OF PORT D1

The RS232 D1 port must be configured to the same setting as the connecting port.

The CAL3k setting: Data=8bit, Parity=none, Stop bit=1 (Standard).

The port speed is settable by:

**54 D1 PORT SPEED 1**

This entry sets the D1 RS232 port speed in baud. The default is 19200baud.

The speed can be set from: 1200 to 115200baud.



This port can connect to:

- Printer
- PC
- BLUETOOTH bridge or RS232/USB bridge

## 8.6 CONNECTING TO A PC WITH A RS232 PORT

The PC must have a RS232 port configured to the same speed and a receiving App such as TERA TERM or any virtual port emulator. If the PC has no RS232 port, then a RS232/USB bridge can be used. The data output is initiated from the CAL3K keyboard or internally.

## 8.7 CONNECTING TO A PC FOR DATA COLLECTION

This method initiates data transfer on the PC. The DDS Windows Software will upload data and save them to the PC memory in a .CSV format. The physical connection can be:

1. RS232 cable
2. USB Cable
3. BLUETOOTH BRIDGE (if no RS232 port, or if USB cable to short)
4. RS232/USB bridge

Configure the DDS Windows Software App to suit any of the above methods.

## 8.8 CONNECTING TO A BALANCE

This step is not necessary. The weight (mass) of the sample can be entered on the keyboard. The balance connection cable is included in the kit. If the calorimeter is installed near the balance, then we recommend it be connected. The default balance speed is 2400baud. Set your balance to this speed. Test the connection by pressing (F5). Then the stable balance weight should show on the display. If you press (Enter) then the mass is locked. See MASS ENTRY to unlock it.

**24 BALANCE SPEED 0**

The balance must have the same setup as the CAL3K. The setting is:

Parity	None
Stop bits	1
Data	8 bit
Default Speed	2400

The speed in the CAL3K can be adjusted from 1200 to 38400 baud.





## 8.9 CONNECTING FOR REMOTE OPERATION

The CAL3K range of calorimeters can be operated remotely via the Internet. For this to work the following connections must be made:

- A) The CAL3K must connect to a local PC. The connection can be:
  - A1) USB cable
  - A2) RS232 cable
  - A3) BLUETOOTH BRIDGE: RS232 to USB
  - A4) USB to RS232 converter (Cable)
- B) The local PC must be connected to the internet.
- C) It must run the supplied REMOTE3K software.
- D) The PC must use a desktop sharing APP such as: TEAM or ANYDESK
- E) The remote PC must use the same desktop sharing application.

If the CAL3K has only one RS232 port such as the CAL3K\_S, ST then all print operation is terminated but the print message (output of CAL3K) is displayed on the PC.

The remote operation copies the CAL3K screen display to the Local PC and via the internet to the far PC. Any keyboard entry on the far PC or the local PC or unit keyboard are visible on the CAL3K and all other PC's will be executed as if operated from the CAL3K keyboard.

The purpose of this remote operation is to teach, train, trouble shoot and operate the CAL3K from an offsite location.

## 9 PRINTING AND OUTPUT

The CAL3K has a D1 RS232 port which is its connection to the outside world. Some units have a D2 RS232 port and a USB port. These ports are designated as follows:

D1 RS232	PRINTING
D2 RS232	BLUETOOTH, OTHER
USB	PC APP

The D1 RS232 port must be setup to match the speed of the connecting port.

### ***54 D1 PORT SPEED 1***

This entry sets the D1 RS232 port speed in baud. The default is 19200baud.

The D1 Port operates with: Parity=none, Data=8, Stop bits=1.

The speed can be set from: 1200 to 115200baud.

The output on the D1 port can be triggered by:

- A CAL3K operator (keyboard) initiated.
- A CAL3K internal initiated
- A PC DDS Windows Software App (external)
- A PC DDS Remote Software App (external)



Outputs are designated as:

PRINT	{PRT....}	max 80 column output, readable
DUMP	(DUMP....)	More than 80 column output

## 9.1 OUTPUT PRINTING TO D1 PORT, KEYBOARD INITIATED

An Output appears when one of the PRT... commands are executed. The following commands require that an output format (for an 80-column printer) is connected.

Before printing can commence the printing must be enabled

**86 SET RESULT ENABL 1**

This command enables (YES) or disables (NO) the spontaneous printing of results after a determination. The results are entered in memory and can be printed later by any of the print commands.

**50 READ RESULT MEM 0**

This command displays the result in the display. By pressing F8 the result is printed in the selected format A-H.

**63 SET OUTFORM A-H: 1**

This command specifies one of the eight output formats A-H. The outputs can be disabled by specifying anything but A-H or by using CMD=86, SET RESULT ENABL=NO.

The formats are not set in stone, so it is best to print the HEADER of all formats. The text descriptions give you a faint idea of what the number columns(data) are.

**56 PRINT LIMS HEADER 0**

This outputs a single text line describing (sort off) the data contents of the A-H output formats.

**52 PRT RESULT DAY: 1**

Enter the day number 1-31 and all results recorded at this day are printed. The result memory should be cleared every month, CMD=39.

**57 PRT TODAY FORM: 1**

Enter an output format A-H and all results for today are printed.

**58 PRT CALORI INFO 1**

The command prints the relevant CAL3K information, including parameters and customer setting. This printout is included in the shipment.

**59 PRT VESSEL INFO 1**

This command prints the vessel information. The vessel must be in the well. The printout is included in the shipment.

**62 PRT MEM RSLT CR 1**

Enter C/R/YES to print data from memory:

C Calibrations only  
R Results only  
YES Results and calibrations

**64 PRT RESULT GID: 0**

Each result in memory has a GID (Group Identification) of 16 characters. The GID can be an entered text (CMD=3) or the date (CMD=122). Enter the GID (Up to 16 characters) and results stored with this GID are printed.

**67 PRT CV'S VESSEL: 0**

The entry is a 4-character vessel serial number, printed on the vessel lid. The complete result memory is searched for this serial number and all results are printed.

**69 PRT RESLTS FORM: 0**

Enter the output form descriptor A-H and all results in the memory are printed in the specified A-H format.

**84 PRT ALL CALIBRAT 1**

Enter the vessel number or place the vessel in to the well and all calibrations accumulated for this vessel are printed.

**85 PRT MAX DATA 1**

The accumulated maximum data are printed.

**107 PRT KEY CMD LIST 1**

The keyboard command list is printed for the present password priority. Enter the password to print the FULL list.

**112 PRT HELP HISTORY 0**

This feature applies to CAL3K-AP, A, F, ST



The CAL3K records all major operational or setup faults. This command prints all recorded events. After 64 events the unit overwrites the oldest one. The printout consists of:

Message Number                    Mxxx (M Number)  
Date and Time  
Short display text

### ***116 PRT COMPENS+MAX***

During calibration the unit calculates the compensations applicable to the vessel and mode. This command prints the compensations and associated max limits.

### ***55 SET FILTER C, F: 1***

A filter can be applied to exclude items from the print output.

C      Calibrations only  
F      Faults only  
Blank All, no exclusions

The CAL3K can supply the following information:

### ***101 PRT PRESS. RECRD 1***

An CAL3K-AP command. This command prints all (Max 16) pressure recordings which are caused by either:

Exceeding the PRESS PEAK LIMIT  
Exceeding the internal max pressure setting 90bar

The pressure recordings can be reset by CLEAR PRESS RCRD, CMD=38. The records can be read out on the display with CMD=115 and a junk record can be simulated with CMD=126.

The internal initiated (spontaneous) printing happens from two sources:

A) When a result is available. An output form A-H must be selected and:

### ***86 SET RESULT ENABL 1***

B) When the REAL TIME output is specified.

### ***61 SET RT FORM WXYZ 1***

This command enables and sets the REAL TIME output format to WXYZ. Selecting anything but WXYZ disables the real time output. The WXYZ format can be tested by printing the header with CMD=53.

### ***60 SET RTHEAD EN CP 1***

The REAL TIME output (from 30 seconds before firing) can be preceded by Customer (C) and Parameters (P). If none is desired enter anything but CP.



The header can be printed with

**53 PRT RTIME HEADER 1**

The CAL3K has a REAL TIME (RT) printout facility. It must be enabled by selecting one of the 4 output formats WXYZ, CMD=61. Then the header (Data descriptor) is printed. If the SET RTHEAD EN CP, CMD=60 has a selection (C=Customer, P=Parameter) then this info is printed as well.

**9.2 OUTPUT, NON-PRINTABLE**

These commands cause a string of information, which requires a PC to capture.

**79 DUMP ALL RESULTS 1**

All results from memory are printed to the D1 port. The output is in the N or U format with about 300 characters per line. Enter:

- YES N (Normal) format, a little shorter than:
- U U format, long

**9.3 OUTPUT, EXTERNAL INITIATED**

In principle the external PC must send a request for data to the CAL3K, and the unit replies with the requested data. This requires that an App runs on the PC; it could be:

- A) The DDS Windows Software App (supplied)
- B) The DDS remote software App (Supplied)

A PC with the DDS App can be connected via:

- 1) RS232 cable
- 2) USB cable
- 3) BLUETOOTH BRIDGE
- 4) RS232/USB bridge cable

**9.4 OUTPUT TO A SPEADSHEET**

An output to a spreadsheet can be facilitated in a round-about fashion by an App running on the PC. The App facilitates the capture of the data and designating a place and file for the data to go to.

**9.4.1 TERMINAL EMULATOR**

This is an open-source App which is freely available from the NET such as TERATERM VT. Start the App and configure the baud rate to 19200 or the same as the port you use.

Then type the command and the data appears on the screen. Select all the data, open NOTEPAD and paste the data into Notepad. Then save the data to a file as NAME.CSV. A spreadsheet understands this format. Note that all CAL3K outputs are available, excluding PC triggered outputs.



### 9.4.2 DDS REMOTE APP

This App is included in the shipment. It can be used to:

- A) Capture the keyboard-initiated output.
- B) Trigger the keyboard command which causes the output.

Once captured specify the NAME.CSV. Note that all CAL3K outputs are available, excluding PC triggered outputs.

### 9.4.3 DDS CAL3K APP

This App is included in the shipment. Configure it to the port and speed and it will connect to the CAL3K. It uses special commands to request outputs from the CAL3K. This allows you to upload:

- Short version results (Format N, Normal)
- Long Version results (Format U)

Note that only results are available! Keyboard commands are not serviced!

## 10 OPERATING CYCLES

The CAL3K units go through the following cycles, sometimes referred to as periods. Each of the cycles is indicated in the upper left corner of the LCD display. During the operating cycles the unit checks on the conditions and reports. Note that not all cycles and reports are installed on your unit!

### 10.1 CYCLE: PREPARATION

The unit checks and reports messages as follows:

Vessel calibration:	M101, M103
System setup	M378
Mass and Vessel data	M110, M106, M108, M111, M112, M109
Vessel Inspection and faults	M116, M118, M180
Lid closed	M184
Firing wire	M186

If everything is OK, it will go to the next cycle.

### 10.2 CYCLE: FILLING (*CAL3K-AP ONLY*)

The CAL3K-AP will operate the filling valve and monitor the filling progress. It will try to fill the vessel up to the target pressure (CMD=88) or until a plateau is reached above the minimum fill pressure (CMD=96). It monitors the filling time (CMD=97).

Manual Lid open	M214
Filling in progress	M230
Slow filling, No filling	M197, M206
Filling fault	M204

If all is in order it starts the initial cycle.



**10.3 CYCLE: INITIAL**

This is a very important cycle: it allows the vessel temperature to settle down and equalise. The unequalized temperatures are the result of filling and handling of the vessel.

Vessel communication	M210
Initial cycle interruption	M216
<b>CAL3K-AP:</b> Filling Leak	M304
Vessel temperature	M258
Firing voltage low	M270

If everything is OK, it fires the sample and switches to the MAIN cycle.

**10.4 CYCLE: MAIN**

The main (measuring) cycle monitors the vessel temperature and calculates an interim result every 6 seconds 2.5 minutes before the end.

Termination, blocked	M374
Vessel fault	M300
Abort	M302
Miss Fire	M376, M310
<b>CAL3K-AP:</b> Pressure Abort	M382

At the end of the main cycle all variables are saved, and the end cycle is started.

**10.5 CYCLE: END**

<b>CAL3K-AP:</b> Deflate the vessel	M360, M307, M305, M309
Remove vessel	M370
Calibration average check	M372

**10.6 CYCLE: COOL**

**CAL3K-S, ST only:**

The vessel is cooled down	M524, M525, M514
---------------------------	------------------

**10.7 HELP DISPLAY**

The messages (any message) are identified by the Mxxx number. The help text can be read out by:

**114 READ HELP TEXT 0**

Each fault or operation has a message number attach, the number starting with a 'M' followed by 3 numerical digits. If it is required to read the help text type the command and number.

Example: 114, enter, 320, enter

Display: **M320: The temperature Rise is lower than the set limit. Proceed with reduced accuracy or reduce the mass by 30%**



## 11 OPERATIONS

### 11.1 LOADING DEFAULT PARAMETERS

The default parameters are loaded before shipment. It is strongly recommended that the default parameters are used and are regularly loaded. At power-up the vital parameters are checked during operation, and if they are out of specification then the DEFAULT PARAMETERS must be loaded.

**10    *PARA DEFAULT LOAD*        0**

The above command loads the default parameters for the present mode. Each mode (**CAL3k-A/AP**) has its own default parameters.

The mode in a **CAL3K-A/AP** can be changed with:

**82    *SET OPS MODE*                1**

### 11.2 ENTERING PASSWORD

The Unit is delivered with a password activated. In this mode NOT all commands are accessible. The password is: **DDS3K**.

The available commands can be checked by entering the letter '**A**' and then pressing the **UP** or **DOWN** arrow repeatedly.

You will see the command (Line#3) and a short explanation (Line#2). If you wish to operate the indicated command, press: **ENTER**

Enter the password:

**6    *PASSWORD ENTRY*                0**

All commands with a priority = 1 are password protected (not accessible). Enter the password '**DDS3K**' to make priority = 1 commands visible. The password is active for 5 minutes.

If you hear a loud audible beep after pressing enter, you have entered the wrong password. Try again, only once the password has been correctly entered will the rest of the password protected menu options become available.

### 11.3 MASS ENTRY

The sample mass is a weight in the range 0.2 to 1.5 grams (Default). Once the mass has been entered, the calorimeter will display the mass in the first row of the display. At this point the mass is LOCKED and will only be deleted by the calorimeter once it has been ignited.





The default mass range can be changed to suit your operations:

<i>30</i>	<i>MIN MASS LIMIT</i>	<i>1</i>
<i>31</i>	<i>MAX MASS LIMIT</i>	<i>1</i>

#### 11.4 MASS ERASING

Once the mass has been entered, it is 'LOCKED' into the present operation until it is ignited. The only way to 'ERASE' the mass or 'UNLOCK' it is as follows:

1. Type the word '**MASS**', **Enter**, a beep can be heard, and the entered mass is displayed.
2. Press the '**DELETE**' button on the keyboard.
3. **RESET** (CMD=124) or **ABORT** (CMD=12)

#### 11.5 SAMPLE IDENTIFICATION (SID)

The sample ID is a 16-character (or shorter) string of characters. The last character will be automatically incremented at each firing. That is to say that a 5 is incremented to 6, and 'C' increments to 'D'. If a short SID is used, followed by spaces, then the last non-space character is incremented. The SID is right orientated. The SID can be letters and numbers.

If the time is used as SID (CMD=121, YES) then only 8 characters are used:           HH: MM: SS  
Note that not all print formats print all SID characters.

#### 11.6 GROUP IDENTIFICATION (GID)

The Group ID is a 16-character (or shorter) string of characters.

If the date is used as a GID (CMD=122, YES) then only 8 characters are used: YY/MM/DD

Note that a printout command: **64 PRT RESULT GID** is available to print all results store with this group ID.

#### 11.7 BALANCE MASS

The supplied balance cable (Part No: 3K-1-117) can be connected to a suitable balance and then the weight is transferred automatically to the CAL3K display. The '**F5**' key connects the unit to the balance. When the mass weight is displayed press '**ENTER**' to LOCK the mass in to the calorimeter.

#### 11.8 OPEN LID

*The CAL3KS/ST* the operation is manual. All other instruments open the lid by a command or automatically.



**73 OPEN LID 1**

The lid can be open at any given time. The present operation is terminated, the vessel pressure. (**CAL3K-AP ONLY**) is deflated, and the lid is opened.

**Note: The Lid is opened automatically if no vessel is inserted, and the lid is closed.**

**11.9 FIRE MANUALLY**

This can be used to shorten the initial period. The initial cycle timing is circumvented, and the Vessel charge is ignited. It is useful to test the firing/ignition procedure. It is not recommended for use during normal operation, as this will affect the accuracy of your sample being determined.

**12 FIRE MANUAL 1**

This command triggers the firing circuit!

**11.10 ABORT**

The abort (Reset) commands terminate the present operation. The exact sequence depends on the type of unit and safety measures.

It will deflate the vessel (**CAL3K-AP ONLY**)

It will write a terminated result in to the memory and print it if enabled.

It will not save a calibration.

It will open the lid (**CAL3KAP/A/F**)

The ABORT is the same as a RESET. It can be performed at any time.

**12 ABORT/RESET 0**

This command clears everything. It terminates any of the operational cycles such as INITIAL or MAIN. Data is lost. However, if a MAIN cycle was terminated then a record is stored with the appropriate status.

**124 RESET 0**

The same as ABORT. Any process is terminated. A Sample in progress is lost, Balance mass is lost.

**11.10 UNITS OF MEASURE**

The unit works in KJ/g and Centigrade. However, the CV value can be expressed in other units of measure by:

**22 UNIT: KJ, KCA, KBU 1**

This command sets the measuring units. Note that the internal working is in KJ/g, but any output is converted to the units. Enter the letters:



KJ for KJ/g  
KCA for KCAL/g  
KBU for KBTU/lb

The following conversion applies:

$$1\text{KJ/g} = 429.9226 \text{ BTU/lb} = 0.4299226 \text{ KBTU/lb}$$
$$1\text{KJ/g} = 0.2388459 \text{ KCAL/g}$$

$$\text{Therefore: The } 26.454\text{KJ/g} = 11.373 \text{ KBTU/lb} = 6.3184 \text{ KCAL/g}$$

**Note: The unit conversion is only used in communicating with the CAL3K and the display.**

We know that the KBTU/lb and KCAL/g is not conventional, but they fit nicely into the display format.

## 12 CALIBRATION

Calibrations are needed to calculate the CV of an unknown sample. The calibration process is the same as with a sample, except that many more variables (conditions) are saved to a special calibration memory. All units keep 16 calibrations per vessel and operating mode.

After a calibration (and if more than 4 calibrations are stored) the **AI (Artificial Intelligence) module** starts and analyses the calibrations for consistency, quality, and compensations factors. The compensation factors are used with a regular sample to fine tune the result. The AI process applies every calibration to all other calibration and computes the compensations it needs to apply for best result consistency (RSD).

The CAL3K performs two functions during a calibration:

- 1) It saves the calibration to the respective vessel calibration memory.
- 2) It calculates the result with the already done calibration and displays it. Of course, this doesn't work with the first calibration and the standard value is displayed.
- 3)

### 12.1 CALIBRATION CHECK SAMPLE

Traditional a calibration check was performed to test the calibration. The CAL3K has no need for it because every calibration is a check sample performed with the previous calibration average when more than the MIN CALIBRATIONS are stored. The unit needs approx. 8 calibrations to perform to the specifications. If you make more, it automatically blocks one if it doesn't conform.

### 12.2 CALIBRATION COMMANDS

#### **19 CALIBRATION 0**

This command initiates a calibration cycle and saves this result and conditions in the calibration memory for this vessel and mode. This command must be used with every calibration.

**26 MIN CALIBRATIONS 1**

This command enables the AI module to calculate the compensations when more than the minimum calibrations are stored. The default is 4. The compensation calculations use the UN-BLOCKED calibrations.

**117 BLOCK CALI ABOVE 1**

This allows the calibration management module to block a calibration if more than the specified number are available. A zero entry disables the blocking. The default number is 8.

**27 BLOCK WORST CALI 1**

This command allows to the AI module to BLOCK a calibration if it is outside the set limit in KJ/g. The default is 0.04KJ. The blocking takes place when a NEW calibration was done when:

- A) When **BLOCK CALI ABOVE** is > 0 and the number of calibrations is > then the entry
- B) When a calibration deviates by more than the **BLOCK WORST CALI** from the average.

A blocked calibration is not used in the averaging process. If for any reason (disabled, all calibrations within limits) the calibration memory get filled then the OLDEST calibration is blocked.

**113 COMPENSATE BOMB 1**

This command analyses the un-blocked calibrations and calculates a new set of compensations. It displays the average CV, the average deviation (AD) and the peak deviation from the average (PK). This command is automatically executed after each calibration.

**12.3 CALIBRATION CONDITIONS**

With each calibration all the variables such as:

- Initial conditions
- Main conditions

are saved. It is therefore imperative that the calibrations are performed at different operating conditions like:

- First/last time in a day
- During routine operation
- Whenever the operating conditions have changed
- Whenever parts (consumables) of the vessel have changed

The AI module has one drawback: It requires more than one calibration to start working. The minimum number of calibrations is set by:

**26 MIN CALIBRATION 1**

Note that this number restricts the calibration management only. Sample determinations are possible with less than 4 calibrations if the present compensations or the DEFAULT (CMD=125) compensations are used.



The CAL3K uses AI (Artificial Intelligence) to improve accuracy and repeatability. The results are computed by comparing them to a calibration. With AI more calibrations are required. With each calibration the instrument learns the characteristics of the temperature and vessel behavior and the reaction to the environment.

In practical terms it means that accuracy improves with the number of calibrations performed. Calibration should be performed in all operating conditions at least once so that the CAL3K can learn the influences of the changed conditions. The conditions depend very much on the laboratory operation.

#### 12.4 CALIBRATION IN DIFFERENT MODES

A calibration is saved for a particular vessel and a particular mode. Note that not all CAL3K products have a mode parameter, but all units have 16 calibrations per vessel. The following table explains this.

Product	Modes	Vessel	Calibrations	Total calibrations
CAL3K-AP	4	4	16	256
CAL3K-A	4	4	16	256
CAL3K-F	1	4	16	64
CAL3K-ST	1	2	16	32
CAL3K-S	1	1	16	16

#### 12.5 FACTORY CALIBRATION

All units leave the factory with 6-10 calibrations per vessel (mode ZERO). We use the data to monitor the performance, accuracy, and repeatability. You can use the factory calibrations and add yours to it, or you can delete all and start fresh.

A calibration is deleted by:

**42 CLEAR B\_CAL S/N 1**

Specify the vessel number or the place in memory (CMD=106) from 1-4 as applicable.

#### 12.6 CALIBRATION FREQUENCY

Starting with a CLEARED calibration memory you need calibrate the unit once. It is advisable to load the default compensations at this point in time or rely on the present ones. If the compensation is destroyed, then it loads the default ones automatically to the bomb and mode.

**125 LOAD DEFAULT COMP 2**

If not loaded then the unit would use the present compensations, whatever they are!

Theoretical you can run a sample on one calibration, but it is good practice to run a second (and third?) calibration before starting to run samples.

Do not use the calibration on the first samples, rather perfect your preparation first. The unit is shipped with a calibration set installed and this is good for you to run with.



Once you mastered this preparation then try a calibration at different times of the day and whenever something changes.

After '**BLOCK MIN NUMBER**' calibration the AI will analyse your calibration performance and may reject a calibration because it falls outside the '**BLOCK WORST LIM**' limit.

A calibration should be performed once a day or week depending on the quantity and operating conditions. Note that the vessel temperature changes during an overnight break or during lunch hour. A calibration is advised once after these breaks and during routine operation. If large changes in ambient temperatures are encountered, then a calibration is also advised.

Once 15 calibrations are stored a new one is entered in the 16<sup>th</sup> place. Then AI decides if the new calibration is good. If it is good, then it deletes the OLDEST to make space for the next calibration. The AI process calculates the best compensations with every calibration. If 16 calibrations are stored, then a new calibration will override one (oldest or worst) after the next calibration.

## 12.7 ROUTINE CALIBRATION

All the above commands and procedures explain the internal handling of calibration. If you feel to perform a calibration, then do so with:

**19 CALIBRATION 0**

The details are automatically taken care off. No further input is required.

## 13 SPIKING

The spiking method is called for when a sample:  
has a very low CV  
or burns reluctantly.

At this point a more efficient burning material is added to the sample to help the process. This material sample is the spike.

The 'Spike' must be of a known calorific value, usually benzoic acid of approx. 26.454MJ/Kg. On some occasions a Gelatine Capsule (3k-4-068) or Crucible Cover Disc (CAL2K-FLDS) needs to be used as a Spike.

A Gelatine Capsule is used when burning:

- Fine powdery substances, which 'scatter' easily
- Benzoic acid powder

A crucible cover disk is used when burning:

- Volatile samples (fuels)

When using these items, you will first need to determine the energy value of the Spike.

GELATINE CAPSULE FOR POWDERY SUBSTANCES: The gelatine capsule is the Spike.

CRUCIBLE COVER DISK FOR VOLATILE SUBSTANCES: The cover disk is the Spike.



BAD/RELUCTANT BURNING SUBSTANCES: The BA is the Spike.

BA POWDER IN A CAPSULE: The gelatine capsule is the spike.

The following method is the traditional method. Later a 'EASY CAPSULE' spiking method is explained.

### 13.1 SPIKE VALUE

You need to burn the spike material and determine its calorific value. If a BA tablet is the spike, then the value is known. The CV of the gelatine capsule is also known, but the cover disk needs to be determined. This is best done by burning let say 10 cover disk and dividing the result by 10 to obtain the CV of a single disk. This obtained CV value must be entered as the SPIKE VALUE in the CAL3K. The spike value is entered in the specified Unit of measure.

**15**    **SPIKE VALUE**            **1**

The Spike Value is the CV of the spike material. If Benzoic Acid is used for spiking, then the CV=26.454KJ/g. The default setting is 26.454KJ/g

### 13.2 SPIKE MASS

This is the mass of the substance to be used for spiking. The procedure to follow is to place the crucible on the balance and press TARE. Then add the substance to be used for spiking to the crucible. This Mass value is the "Spike Mass" and must be entered **manually** into the Calorimeter. **Tare the balance again.** Then add the sample. This mass is the sample mass and must be entered in as the Mass (Balance Mass)

**7**    **SPIKE MASS**                    **0**

This is the spike mass. If the value is >0 the spiking is assumed, and the result will be reduced by:  
Spike mass x Spike value.

The spike mass is cleared after a determination. A 'ZERO' spike mass disables the spiking process.

### 13.3 STEP BY STEP ENTERING THE SPIKE MASS

- Place the crucible on the Balance.
- Press "Tare" to clear the weight of the crucible and get to 0.0000g
- Place one Benzoic Acid tablet (as a spike) and **note** the Mass = Spike Mass
- Enter the Spike mass by 'Spike Mass' enter, mass value.
- Tare the balance!
- Add the sample to the crucible!
- Enter the sample mass electronically (F5) or manual (F1)
- Place the prepared crucible (benzoic acid tablet with sample) inside the Vessel and close the Vessel.
- If you have a **CAL3K-AP**: Place the prepared vessel in to the well. All other units: fill the vessel in the filling station with oxygen and place it in to the well.



- Optional: Enter the SID, e.g. 001
- Close the Lid of the Calorimeter
- The CAL3K Calorimeter will now perform some standard checks and if all is good, it will begin with the “Initial Phase”, “Fire”, “Main” phase and then indicate that the determination has been completed.

Once the determination is complete, the result is the calculated calorific value of the sample. The calorimeter will automatically take the end result, minus the spike energy and present the final result of your sample.

### 13.4 EASY CAPSULE SPIKING

The Spike Mass must be zero!

**EASY SPIKING:** It uses a gelatine capsule and assumes that all capsules have the same CV value (temperature rise). The temperature rise of the capsule is deducted from the total temperature rise. This method is reasonable accurate and very convenient. You need to know the temperature rise of a capsule or you can use the default:

**29    CAPSULE RISE C    1**

The temperature rise caused by a gelatine capsule is entered here (Default=2.594). This entry is associated with the capsule (Manufacturer, weight, size) and is constant unless changed. The entry is in degree Celsius.

Of course, you can determine the average CV of let say 10 capsules. The **Temperature Rise** of each determination must be read out via the keyboard (CMD=36) and then averaged and entered via CMD=29.

The EASY SPIKING is enabled via:

**28    SET CAPSULE EN    0**

A YES/NO entry enables/disables the use of gelatine capsule temperature deduction.

Note that the ‘**GELATINE CAPSULE**’ enable setting is retained after each sample. If a sample without a capsule is burned then the capsule compensation must be disabled (CMD=28, NO)

In the ‘EASY SPIKE’ method the Capsule Rise value (CMD=29) is deducted from the measured temperature rise (Sample + Capsule) before the result is calculated.

The operation is very easy:

1. Enable the easy spike process (CMD=28)
2. Tare the empty capsule.
3. Put the sample in to the capsule.
4. Weigh the filled capsule and enter the weight as MASS.





5. Perform the determination.

## 14 CORRECTIONS

The corrections are applied because they should not appear in the final result, or some value was added during the combustion process.

### 14.1 COTTON CORRECTION

Some energy is added to the burning process because of the following:

- Firing energy
- Wire oxidation
- Electronic heating
- Cotton thread

**9 COTTON+WIRE CORR 1**

This is the cotton and wire correction. The cotton has a definable energy and the electrical current which heats the firing wire has a constant energy content. Both these (and others which produce an offset) are combined in this entry. The default value is 0.0593C. The entry is in CENTI GRADE. This correction is ALWAYS applied: To samples and calibrations alike. If the cotton or firing wire are changed then the value should be re-adjusted to suit.

The 'Cotton correction' is constant. It is therefore treated as temperature rise compensation. The cotton correction is subtracted from the raw temperature rise to yield the NETT RISE.

### 14.2 SULPHUR CORRECTION

If the sample contains sulphur, then energy is released during the formation of SO<sub>2</sub> from oxygen and sulphur. This energy is deducted from the nett CV. The sulphur content of the sample must be measured before and the CV (KJ/g) of SO<sub>2</sub> formation must be entered:

**8 SULPHUR CORRECT. 1**

The sulphur correction deducts energy which is released by the formation of SO<sub>2</sub>. The entry is in KJ/g. The default setting is zero.

The Sulphur correction is NOT applied during calibration.

### 14.3 MOISTURE CORRECTION

If a sample has a moisture content, then the nett result can be compensated for. The moisture entry is in percent.

**33 MOISTURE PERCENT 1**

This entry is important when the moisture content of a sample is known, and the nett calorific value must be calculated. The CAL3K calculates the nett mass and displays the nett calorific value. The default setting is ZERO=Disabled.

The moisture content reduces the mass to a nett mass and increases the CV.

**14.4 GELATINE CAPSULE CORRECTION**

If a gelatine capsule is used during the process (to contain a powdery sample) then this energy can be compensated for by enabling the capsule correction (EASY SPIKING):

**28 SET CAPSUKE EN 0**

A YES/NO entry enables/disables the use of gelatine capsule temperature deduction.

If enabled the capsule temperature rise will be deducted from the raw temperature rise before further calculations. See **EASY SPIKING**

**29 CAPSULE RISE C 1**

This refers to the EASY SPIKING method and assumes that all gelatine capsules have the same mass and consistency. Then the temperature rise caused by a gelatine capsule is entered here (Default=2.594). This entry is associated with the capsule (Manufacturer, weight, size) and is constant unless changed. If a capsule is used (for powdery samples) then it must be enabled with CMD=28.

**15 FIRING**

The CAL3K automatically fires the sample by sending a very high current through the firing wire, which glows and ignites the cotton, which in turn ignites the sample. The amount of current to send through the wire can be programmed.

**16 FIRING VOLTAGE 1**

The firing voltage is default set at 25.0 volts. The setting depends on the sample and the oxygen pressure.

The firing voltage must be decreased if the 'semi-permanent' firing wire melts too often. You should get 20 to 50 firings out of the wire depending on how corrosive the sample is.

The actual firing voltage is produced by a charging circuit, which takes about 2 minutes to charge. The charged voltage can be monitored by:

**51 READ FIRING VOLT 1**

Type **ESC** to end the firing voltage display. The firing takes place automatically after the INIRTIAL period. The sample can be fired manually:

**17 FIRE MANUAL 1**

The manual firing can be activated at any time if the vessel is in the well and the firing wire is present.

The manual firing should be avoided during routine operations.

**16 MISS FIRE**

The CAL3K measures, in broad terms, the temperature rise in the vessel when a sample is burned. The sample is ignited (fired), and when it doesn't ignite then we call it a 'miss fire'.

This can have many reasons:

1. Technical: There is no or not enough current flowing through the firing wire
2. Bad preparation: No oxygen, no cotton, or the cotton thread does not touch the sample.
3. Bad Sample: It just doesn't like to ignite!

The CAL3K informs the operator of a miss fire condition and terminates the MAIN period. The result is marked accordingly.

**21 MISSFIRE LIMIT 1**

The 'MISS FIRE LIMIT' checking can be disabled by entering ZERO.

**M310 MISSFIRE: TEMPERATURE RISE: xx.xxxx**

This error comes up 30 seconds after firing when the temperature rise is below the set limit. The actual temperature rise of the vessel is indicated as xx.xxxx. The best way to determine what was the cause is to open the vessel and check the content.

- A) The sample has burned, but the temperature rise is lower than the limit. The temperature rise is indicated as xx.xxxx. Two possible ways to solve this:
  - A1) Increase the sample mass.
  - A2) Decrease the 'LOW RISE LIMIT'
- B) The sample has not burned, and Cotton thread is visible but burned off the firing wire: No oxygen.
- C) Cotton thread is burned (gone!), but sample is not: The cotton didn't ignite the sample.



C1) It could be that the oxygen filling process is too fast, or the bomb handling (bump) has moved the cotton thread away from the sample material. The filling should take 30-60 seconds.  
C2) The sample doesn't ignite easily. If the sample is moist then try drying it. Otherwise use the spiking method: The cotton ignites the spike material and this in turn ignites the sample.

Following is a list of other useful commands when one of the above errors was indicated:

F11	Display last error message first. Press F11 for next, 'Enter' for full explanations.
112	'PRT HELP HISTORY' Print on D1 the complete event errors
104	'LOW RISE LIMIT' Set the low-rise limit
81	'READ LAST N_RISE' Displays the last NETT temperature rise
114	'READ HELP TEXT' Enter 'M' number and display the explanations
51	'READ FIRING VOLT' Display the firing voltage until 'Esc'.
39	'CLEAR RESULTS' Enter "HELP" instead of 'YES' and the help event store is cleared
17	'FIRE MANUAL' Needs vessel and lid closed. Useful for testing the firing circuit. DO NOT USE DURING OPERATION!

## 17 COMPENSATION

### 17.1 MOISTURE COMPENSATION

If the sample has known moisture content, then the result can be compensated for the moisture. The moisture entry is in percent, and the sample mass is reduced to a NETT MASS. The compensation is enabled by:

**33**    *MOISTURE PERCENT*    **1**

The 'Moisture Percent' entry will be retained until set to zero. The moisture compensation is not used during calibration.

### 17.2 FOOD COMPENSATION

Food is consumed by a body through quite a complex process, involving time, enzymes, and a lot more. This is a 'wet' process, and nothing is burned. Because it is a complex process to repeat in a laboratory and it takes time (days) it is seldom used but a 'COMBUSTION CALORIMETER' is used instead. The calorimeter has a lot of advantages, **BUT** it requires some special considerations. This is best explained by analysing sawdust: It burns nicely and produces energy but consuming it yields no benefit and probably makes one sick. However, a calorimeter is a nice, fast, and convenient tool to determine the calories.



Food is measured in CALORIES or KJ. The ATWATER specification reads:

Protein	4Kcal	16.736KJ
Carbohydrates	4Kcal	16.736KJ
Fat	9Kcal	37.656KJ
Alcohol	7Kcal	29.288KJ
Fibre	4Kcal	16.736KJ

The last item (Fibre) is the interference factor: It burns nicely like the sawdust but...

To compensate for fibres:

**25 FIBRE CV VALUE 1**

The fibre CV value is entered in the unit of measurement specified by:

**22 UNIT: KJ, KCA, KBU 1**

The actual percentage of fibres is entered by:

**23 FIBRE IN FOOD % 1**

Entering a fibre percentage REDUCES the calorific value of the result.

The 'FIBRE IN FOOD' entry is retained. But not used during calibration or BA checks.

## 18 LIMITS

Limits can be applied to warn the operator of possible bad operations. If a limit is violated the result is flagged accordingly.

**103 HIGH AMBIENT LIM 1**

The unit measures the Ambient temperature, which has an influence on the result and the operation. The influence is mitigated by calibrating the unit at relatively high ambient temperatures. However, this limit can be set up to warn the operator. The default setting is 36C.

**102 LOW AMBIENT LIMT 1**

The ambient temperature affects the unit's operation and result. The result influence can be eliminated by calibrating the unit at low ambient temperatures. The default setting is 8C. The lowest possible temperature measurement is 2C.

**105 HIGH RISE LIMIT 1**



The result is affected by the temperature rise after firing. This affect is minimised by calibrating the unit for high rise (e.g., 0.75g Benzoic Acid). However, the sample mass should be adjusted so that the temperature rise is near to the optimal 12C +-2C. The default setting is 16C.

**104    LOW RISE LIMIT    1**

The temperature rise after firing should be 12C+-2C. This can be managed by adjusting the sample mass. A limit can be set up to warn the operator that the temperature rise was too low. The default setting is 8C. A lower rise can be achieved by calibrating the unit to an 8C+-2C.

**31    MAX MASS LIMIT    0**

This limit applies to all possible mass entries. Exceeding this limit prevents a mass from entry. The default limit is 1.5g

**30    MIN MASS LIMIT    0**

This limit applies to all possible mass entries. An entry below this limit will be rejected, the default is 0.2g.

**21    MISFIRE LIMIT    1**

If the sample is not igniting, then it is called a misfire. The unit checks the temperature rise 30 seconds after firing, and if the rise is below the limit a misfire is declared and the process is terminated. The default value is 0.44C. The misfire limit checking can be disabled by entering zero.

## 19 TIME AND DATE

The CAL3K units have a date and time circuit which keeps the time. The data and time are attached to every calibration, result, and event. The following command set the time and date:

**4    TIMESET HH:MM:SS    1**

This command sets the time in HH:MM:SS. The internal clock runs for about 1 week without power and then the time (and date) must be set.

**5    DATESET YY:MM:DD    1**

This sets the date of the CAL3K internal real time clock. Note that the clock remains life up to a week without power. The entry is the form:    YY/MM/DD

The date can be used as a Group Identification (GID).

**122    DATE IS GID    1**



This YES/NO entry allows for replacing the GID with the DATE in the form YY/MM/DD. The GID (Group Identification) is attached to every sample recording. Note that the first 8 characters of the GID are left blank.

The time can be used as a Sample Identification (SID). When this feature is enabled then the auto-increment of the SID is disabled.

**121    TIME IS SID            1**

The Sample Identification (SID) is a 16-character text string. It can be replaced with the time in HH:MM:SS by entering 'YES'.

## 20 CAL3K-AP PRESSURE COMMANDS

This section applies to the **CAL3K-AP ONLY** which has automatic oxygen filling. It performs the following:

1. It fills the vessel from the connected oxygen supply.
2. It monitors the filling process to the specified pressure.
3. It monitors the pressure during the initial period.
4. It monitors the pressure during the main period.
5. It records all pressure variables.
6. It deflates the vessel.
7. It optionally flushes the vessel to remove harmful gases.

The vessel is filled to about 30bar (3000KPA) and this pressure is monitored with a pressure transducer. This device needs a 'zero' adjustment after prolonged use and storage. The adjustment must be done with the lid open.

**90    PRESS ZERO ADJST    1**

The pressure is measured with a transducer which has a zero offset of +-1bar. This error can be eliminated by using this command. The offset can be checked when the lid is open and the 'READ PRESSURE', CMD=89., it should read ZERO. +-0.2bar. If not use the 'PRESS ZERO ADJ' to set it to zero.

**89    READ PRESSURE        0**

The actual vessel pressure is displayed until ESC.

The filling is done up to the filling target pressure. During filling the oxygen flows through a filter and restrictor to slow the filling down. The actual vessel pressure may be a little lower than the target pressure as a combination of the vessel lid flow restrictions and the fill restrictor.

**97    PRESS FILL SECND    1**

The filling time is monitored and when it exceeds the limit the operator is warned. The reason for slow filling could be: Empty oxygen bottle or blocked inlet filter. The filter (and filling restrictor) is a very unlikely reason for slow filling and the oxygen pressure should be investigated first.

**88 PRESS FILL TRGET 1**

An CAL3K\_AP command. The filling target is the nominal filling pressure when the filling valve is shut down. The actual pressure may be 1bar down because of pressure settling. The target should be 30bar. Lower filling pressures are indicated for powdery substances to reduce scattering of sample material.

The default is 30bar.

The entry range is from 0 to 35bar.

The filling is monitored and if it slows down to 0.05bar per second it is terminated. This eliminates any problem with the pressure regulator setting being a fraction lower than the target pressure. However, when the filling pressure doesn't reach the pressure fill minimum then filling process is terminated. The minimum limit can be adjusted by:

**96 PRESS FILL MIN 1**

The minimum filling pressure specifies the limit at which the unit stops working. It allows for slow filling and near empty oxygen bottles. The default is 20bar, which is sufficient for avoiding oxygen starvation at 26KJ/g Samples.

Once the filling operation is complete the unit goes to the INITIAL PERIOD and monitors the pressure. If the pressure drops by more than the 'FILL LEAK' then a warning is displayed. Note that the pressure will drop about 1 bar depending on the restrictor and the flow constrictions in the lid.

**95 PRESS FILL LEAK 1**

After filling the vessel with oxygen, the pressure is monitored and if it declines (leaks) by more than the limit a warning is issued. Theoretical the pressure should be constant, but it requires approx. 1 bar to equalise. The default setting is 1.5bar.

After the INITIAL PERIOD the sample is ignited, and the MAIN PERIOD starts. The burning sample caused a very large gas temperature rise. This pressure rise is monitored. The pressure rise depends on the burning speed of the sample. A faster burning material has a higher peak pressure. The peak pressure and time in milliseconds are recorded with the sample. A very high pressure can also be the result of a large sample mass or energy content. For safety concerns the unit has two pressure limits:

- A) A fixed limit of 90bar. This limit is always in effect.
- B) A variable user setting limit:

**87 PRESS PEAK LIMIT 1**

The pressure peak limit (Default=70bar) is the max. pressure allowed shortly after firing when the pressure increases proportional to the burning speed. When the pressure exceeds this limit, the event is recorded, and the operator is warned. At this point the unit is NOT shutdown. The pressure peak checking can be disabled by entering zero.





If a limit is exceeded the vessel is deflated to a safe pressure and the main period is terminated after a short time. The CAL3K-AP records:

- The absolute pressure
- The time from firing
- Peak Temperature, approx. 30 seconds after firing

### ***115 READ PRESSURE REC 0***

The unit records any vessel pressure excess above the PEAK LIMIT (CMD=87) and above the absolute maximum of 90bar. This command reads the recordings from the memory. Press {enter} for the next and ESC to terminate reading. The pressure recordings can be cleared by CMD=38 and printed by CMD=101. A pressure record can be simulated with CMD=126, but the data are junk.

After the MAIN PERIOD the vessel is deflated. This process is monitored in time:

### ***92 PRESS DEFLAT. SEC***

The deflate time is monitored and if the limit is exceeded a warning is displayed. It could be that the filter or the exhaust valve is blocked. The default setting is 20 seconds. Again, this check can be disabled by entering zero.

Once the vessel is deflated it can be (optionally) flushed with oxygen to remove any harmful gases, or at least dilute them.

### ***93 PRESS FLUSH SEC 1***

The flush cycle is time based. A zero entry eliminates the flush cycle, otherwise the vessel is filled for the specified time in seconds and then deflated. Default value is 3 seconds.

**SPECIAL:** In the **CAL3K-S/ST** models this command is used to actuate the FAN for flush (Cooling) before a determination.

If a pressure leak is suspected, then the system can be tested. The complete pressure test is described in the TESTING section.

The test pressure can be set by:

### ***99 PRESS LEAK TEST 1***

This command sets the leak test pressure applied during SYSTEM LEAK TEST, CMD=94. The leak test is useful when a leak is suspected. The default value is 25bar.

**94 SYSTEM LEAK TEST 1**

This is a service command to check if the system is leaking. The oxygen must be connected, and a vessel must be in the well without the lid. Then the supplied 'MUSHROOM' filling nut must be screwed on to the filling head by hand. Then close the lid. The mushroom fits in to the vessel.

Activate the test (YES) and the CAL3K will pressurise the system to the PRESS LEAK TEST, CMD=99 value. Then it monitors the pressure and displays it. During the test one can try to find any leak if the pressure is declining. Stop the test with ESC.

**Note** that the filling is very fast because the volume (The Vessel is not filled!) is very small. Replace the filling nut again. The filling nut is plastic and must be tightened by hand. Do not use tools and do not 'cross thread' it.

**21 PRINTING REAL TIME**

It refers to a print output while the determination is in progress. The data are gathered every 6 seconds and then output to the D1 Port. The real time (RT) printing is for customers who like to compute their own results like universities. It reveals the data the unit accumulates without any adjustments or compensations.

Select/enable a real time output by:

**61 SET RT DATA FORM WXYZ 1**

Enter one of the letters (WXYZ) to enable an output format or any other letter to disable real time printing.

Optional select a customer (C) and/or Parameter (P) header.

**60 SET RTHEAD EN CP 1**

The letter 'C' enables the printing of the customer header information and the letter 'P' the printing of the relevant parameters before the real time printing starts 30 seconds before firing.

Optional disable result printing:

**86 PRT ENABLE RESULT 1**

Enter 'NO'. This switches the result printing off.

To see a preliminary output, you can trigger this by:

**53 PRINT RT HEADER 1**

This prints the header; the data will appear during a determination. You may like to try the other Real Time output formats: WXYZ (CMD=61)



## 22 CLEARING

It is possible that the CAL3K memory becomes useless because too many items are clogging up the effective search and storage. It is therefore advisable to clear the memory once every 1 to 6 months depending on usage. The units store from approx. 400 to 800 results which are un-manageable. It is advisable to clear the result store after a week.

### 22.1 CLEAR RESULT MEMORY

**39 CLEAR RESULTS 1**

This command clears all results. Depending on the unit the CAL3K keeps from 400 to 800 results. Enter 'YES' to clear the result memory.

### 22.2 CLEAR HELP EVENTS

The unit keeps 64 events. These are recordings of severe operational faults. Recorded is the Date and time and the actual 'Mxxx' message. These 'HELP EVENTS' are important when things happen fast. These recordings can be read out via the F11 key, and they can be printed with CMD=112. The 'HELP EVENT' recordings can be cleared by the 'CLEAR RESULTS' command by entering the letters 'HELP' instead of the 'YES'.

### 22.3 CLEAR VESSEL CAL HISTORY

**42 CLEAR B\_CAL S/N 1**

This command CLEARS all calibrations for the specified vessel Serial Number. The vessel must be removed from the CAL3K. The deletion is permanent. This command should be used when memory space is needed for a new vessel. CMD=106 displays all used vessel serial numbers. Once a vessel calibration is cleared it must be calibrated, See CMD=19.

### 22.4 CLEAR CAL3K MAXIMUM

**40 CLEAR CAL3K MAX 1**

This command clears the CAL3K maximum recordings. The recordings consist of:

- Maximum Start Temperature and date and time it happened.
- Maximum Vessel Temperature and date and time.
- Maximum Ambient temperature and Date and Time.
- Max Vessel pressure and date and time (**For CAL3K-AP only**)

Note that the CAL3K Maximums can be printed with CMD=85



## 22.5 CLEAR VESSEL MAXIMUM

**41 CLEAR VESSEL MAX 1**

The maximum recordings in the vessel consist of:

- Maximum Temperature
- Firing count at max Temperature
- Date and time of max Temperature

The vessel must be inserted for the clearing.

The command should be repeated with each vessel.

## 22.6 CLEAR PRESSURE RECORD

**38 CLEAR PRESS RCRD 1**

This command clears the pressure recordings in the **CAL3K-AP**. The unit keeps 16 recordings of:

- Date and Time
- Mass
- Temperature Rise
- Pressure
- Peak pressure time (Milliseconds)

**Note** that the pressure recordings are triggered by exceeding the 'PRESSURE PEAK LIMIT' CMD=87. The recordings are in a circular fashion and the oldest one is over-written.

## 23 READING

This section contains all reading and display operations. Reading refers to retrieving information and displaying it on the LCD display.

**34 READ CHA. FIRINGS 1**

The number of firings in this chassis are displayed.

**36 READ RISE VESSEL 0**

The temperature rise of the vessel is displayed.

**43 READ TEMP VESSEL 1**

The vessel temperature is displayed until ESC.

**44 READ DRIFT 1**

The drift (Temperature difference per 6 seconds) is displayed until ESC.

**45 READ CARD ID 0**



The card identification is displayed. The Card ID is used by the factory.

**46 READ MAX VESSEL 0**

The vessel maximum data are displayed. The vessel must be in the well.

**47 READ CAL3K MAX 1**

The CAL3K maximum data are displayed.

**48 READ VESSEL INFO 0**

The information of the inserted vessel is displayed.

**49 READ AMB TEMP. 0**

This causes the display of the present ambient temperature until ESC is pressed.

**50 READ MEM RESULT 0**

This command reads results from the memory to the display starting with the last one first. Press ENTER to display the next result. The result in the display can be printed by pressing F8.

**51 READ FIRING VOLT 1**

The actual firing voltage is displayed until ESC is pressed.

**65 READ RESULT DAY: 0**

This command displays the number of results for the entered day number 1-31.

**68 READ STABILITY 1**

The vessel stability (2<sup>nd</sup> derivative of temperature) can be displayed until ESC.

**78 READ ATC TEMP. 0**

Only applicable to **CAL3K-AP**.

The ATC temperatures (Bottom and Top) are displayed until ESC is pressed.

**81 READ LAST N\_RISE 1**

The last Nett Temperature rise is displayed.

**83 READ VESS. FIRING 1**

The number of firings of the inserted vessel are displayed.

**89 READ PRESSURE 0**

The actual vessel pressure is displayed until ESC. This applies to the CAL3K\_AP only.

**106 READ BOMB CALI 1**

The number of calibrations (Max=15) per vessel are displayed for each vessel.

**114 READ HELP TEXT 0**

Each fault or operation has a message number attach, the number starting with a 'M' followed by 3 numerical digits. Type the number with an 'M' or without it and the full help text is displayed.

**115 READ PRESSURE REC 0**

The unit records any vessel pressure excess above the PEAK LIMIT (CMD=87) and above the absolute maximum of 90bar. This command reads the recordings from the memory. Press {enter} for the next and ESC to terminate reading. The pressure recordings can be cleared by CMD=38 and printed by CMD=101. A pressure record can be simulated with CMD=126, of course the data are junk.

**24 BLUETOOTH**

The BLUETOOTH operation requires optional hardware. These BLUETOOTH devices are used to connect a PC to the CAL3K. A good reason to use the extra hardware is:

- A) The USB cable is too short for a computer connection.
- B) The PC has no RS232 port.
- C) It is too difficult to run a RS232 cable from the CAL3K to the PC.

The BLUETOOTH uses wireless communication between the CAL3K (with a RS232 port) and the PC (with a USB port).

The BLUETOOTH communication speed is 115.2KB.

**25 SETTING A STATE**

This section groups all commands which set a state:

ON	OFF
HIGH	LOW
ENABLE	DISABLE

The entry to these commands is indicated below.

**28 SET CAPSULE EN. 0**

Enter Yes if EASY SPIKING is used. See: SPIKING for details

**55 SET FILTER C, F 1**

Enter 'C' to filter calibrations and 'F' to filter faulty results from printing. See: Printing for details.

**60 SET RTHEAD EN CP 1**

This command applies to the **CAL3K-AP/A/F** units with real time (RT) printing. Enter 'C' for customer header and 'P' for parameter header. See: REAL TIME OUTPUT for details.

**61 SET RT FORM WXYZ 1**



This command applies to the **CAL3K-AP/A/F** units with real time (RT) printing. Enter any of the letters 'WXYZ' to enable RT printing or any other letter to disable it.

**62 SET PRT MEMO CR 1**

A 'YES' entry enables the printing of Calibrations and Results. A 'C' entry enables calibrations only and a 'R' entry Results. See: PRINTING for details.

**63 SET OUTFORM A-H 1**

Each letter A-H selects a print format with different data. See: PRINTING FORM details.

**70 SET HIGH RESOLUT 1**

A 'YES' entry selects the high resolution display.

**82 SET OPS MODE 1**

This command applies to the **CAL3K-A/AP only**. Enter the numbers 0-3 to select an operating mode. See: OPERATIONS for details.

**86 SET RESULT ENABL 1**

Enter 'YES' to enable the printing of spontaneous result messages. See: PRINTING for details.



## 26 ALL CAL3K COMMANDS

This command list is universal to all DDS CAL3K products. Some commands may behave a little different to the explanations below, but the intensions are the same.

Each command is preceded by the Index (the reference number under which it is stored), the command text and followed by the priority.

A Zero priority means it is open (no password required) and a ONE means the password must be entered before accessing it.

### **1 MASS 0**

The sample mass is normally entered manual from the F1 key or electronical via the F5 (Balance mass) key or typing the command. Once a mass is entered it is LOCKED into the unit. To UN-LOCK (delete an entered mass) the command must be typed followed by ENTER, DELETE.

### **2 Sample IDENTIFY 1**

This command is the same as the F2 key. The sample identification are 8-16 characters. They are auto incremented if the 'Time is SID' command is not selected.

### **3 GROUP IDENTIFIER**

The Group Identification (GID) is an 8-16character text string. It can be replaced by the 'Date is DID' command.

### **4 TIMESET HH:MM:SS 1**

This command sets the time in HH:MM:SS. The internal clock runs for about 1 week without power and then the time (and date) must be set. The time can be used as a SID. This is not available on the **CAL3K-S**.

### **5 DATESET YY:MM:DD 1**

This sets the date of the CAL3K internal real time clock. Note that the clock remains life up to a week without power. The entry is the form: YY/MM/DD

The date can be used as a Group Identification (GID). This is not available on the **CAL3K-S**.

### **6 PASSWORD ENTRY 0**

All commands with a priority = 1 are password protected (not accessible). The password is 'DDS3K' to make priority=1 commands visible. The password is active for 5 minutes.

### **7 SPIKE MASS 0**

This is the spike mass. If the value is >0 the spiking is assumed, and the result will be reduced by:  
Spike mass x Spike value.

The spike mass is cleared after a determination.



**8    *SULPHUR CORRECT.*    1**

The sulphur correction deducts energy which is released by the formation of SO<sub>2</sub>. The entry is in KJ/g. The default setting is zero. This correction is switched off during calibration.

**9    *COTTON+WIRE CORR*    1**

This is the cotton and wire correction. The cotton has a definable energy and the electrical current which heats the firing wire has a constant energy content. Both these (and others which produce an offset) are combined in this entry. The default value is 0.0593C. The entry is in CENTI GRADE. This correction is ALWAYS applied: To samples and calibrations alike. If the cotton or firing wire are changed then the value should be re-adjusted to suit.

The 'Cotton correction' is constant. It is therefore treated as temperature rise compensation. The cotton correction is subtracted from the raw temperature rise to yield the NETT RISE.

**10    *PARA DEFAULTLOAD*    0**

This command loads ALL default values into the operating parameters. Any changes done to the parameters are over-written (lost). This command should be used whenever the parameters were changed, and the outcome is not desirable. We strongly suggest operating the unit on the default parameters unless a change is necessary.

**11    *BALANCE MASS***

This is the same as the F5 key. It displays the balance mass if connected. Press Enter to lock the mass in. To delete a locked mass type:

MASS, Enter, Delete

**12    *ABORT/RESET*    0**

It terminates any of the operational cycles such as INITIAL or MAIN. Data is lost. However, if a MAIN cycle was terminated then a record is stored with the appropriate status.

**13    *CAL3K-TEST*    0**

This is the same as the F9 key. It initiates the CAL3K (Chassis) test.

**14    *VESSEL-TEST:*    0**

This test is activated by F7 or typing.

The vessel must be in the well. **CAL3K-AP only:** The oxygen pressure must be connected. All vessel functions are tested, and a summary is displayed. The test must be repeated for each vessel.

**15    *SPIKE VALUE*    1**

The Spike Value is the CV of the spike material. If Benzoic Acid is used for spiking, then the CV=26.454KJ/g. The default setting is 26.454KJ/g

**16    *FIRING VOLTAGE*    1**

The firing voltage is default set at 25.0 volts. The setting depends on the sample and the oxygen pressure.

The firing voltage must be decreased if the 'semi-permanent' firing wire melts too often. You should get 20 to 50 firings out of the wire depending on how corrosive the sample is.



**17 FIRE MANUALY 1**

This can be used to shorten the initial period. The firing conditions are circumvented, and the Vessel charge is ignited. It is useful to test the firing/ignition procedure. It is not recommended for use during normal operation, as this will affect the accuracy of your sample being determined.

**18 BENZOIC ACID VAL 1**

This is the Benzoic Acid value in the specified units! See CMD=22 for specifying the units. The default entry is 26.454KJ/g.

**19 CALIBRATION 0**

Prepare the vessel with Benzoic Acid (BA) and set the calibration on (YES). Then close the LID and wait. You will notice that the calibration operation is indicated in the top left corner. Once done, remove the vessel and repeat the procedure with the other vessels (**Except CAL3K-S**) which only has one vessel.

**21 MISFIRE LIMIT 1**

If the sample is not igniting, then it is called a misfire. The unit checks the temperature rise 30 seconds after firing, and if the rise is below the limit a misfire is declared and the process is terminated. The default value is 0.44C. The misfire limit checking can be disabled by entering zero.

**22 UNIT: KJ, KCA, KBU 1**

This command sets the measuring units. Note that the internal working are in KJ/g, but any output is converted to the units. Enter:

KJ	KJ/g, MJ/Kg
KCA	KCAL/g
KBU	KBTU/lb

Enter the letters: KJ for KJ/g  
KCA for KCAL/g  
KBU for KBTU/lb

The following conversion applies:

1KJ/g	=	429.9226	BTU/lb	=	0.4299226	KBTU/lb
1KJ/g	=	0.2388459	KCAL/g			

Therefore: The 26.454KJ/g = 11.373 KBTU/lb = 6.3184 KCAL/g

**Note: The unit conversion is only used in communicating with the CAL3K and the display.**

We know that the KBTU/lb is not conventional, but it fits nicely into the display format.

**23 FIBRE IN FOOD % 1**

This percent (of mass) entry is used to reduce the result by the percentage multiplied with the FIBRE CV VALUE, CMD=25. This fibre adjustment is NOT used during a calibration.

**24 BALANCE SPEED 0**

The balance must have the same setup as the CAL3K. The setting is:



Parity            None  
Stop bits        1  
Data             8 bit  
Default Speed 2400

The speed in the CAL3K can be adjusted from 1200 to 38400 baud.

**25    FIBRE CV VALUE    1**

The Fibre CV value is used during food analysis the default is 16.736KJ/g. Unit conversion is active.

**26    MIN CALIBRATION   1**

These are the minimum calibrations in memory before the AI calibration management start.

**27    BLOCK WORST CALI  1**

Calibrations which are worse than the specified difference can be blocked.

**28    SET CAPSULE EN    0**

A YES/NO entry enables/disables the use of gelatine capsule temperature deduction.

Note that the 'GELATINE CAPSULE' enable setting is retained after each sample. If a sample without a capsule is burned then the capsule compensation must be disabled (CMD=28, NO)

In the 'EASY SPIKE' method the Capsule Rise value (CMD=29) is deducted from the measured temperature rise (Sample + Capsule) before the result is calculated.

**29    CAPSULE RISE C    1**

The temperature rise caused by a gelatine capsule is entered here (Default=2.594). This entry is associated with the capsule (Manufacturer, weight, size) and is constant unless changed. The entry is in degree Celsius.

This capsule rise is used ONLY for EASY SPIKING

**30    MIN MASS LIMIT    0**

This limit applies to all possible mass entries. An entry below this limit will be rejected, the default is 0.2g.

**31    MAX MASS LIMIT    0**

This limit applies to all possible mass entries. Exceeding this limit prevents a mass from entry. The default limit is 1.5g

**33    MOISTURE PERCENT  1**

This entry is important when the moisture content of a sample is known, and the nett calorific value must be calculated. The CAL3K calculates the nett mass and displays the nett calorific value. The default setting is ZERO.

**34    READ CHA. FIRINGS  1**

The number of firings in this chassis are displayed.

**36 READ RISE VESSEL 0**

The temperature rise of the vessel during the main period can be displayed until ESC.

**38 CLEAR PRESS RCRD 1**

This command clears the pressure recordings in the **CAL3K-AP**. The unit keeps 16 recordings of:

- Date and Time
- Mass
- Temperature Rise
- Pressure
- Peak pressure time (Milliseconds)

**Note** that the pressure recordings are triggered by exceeding the 'PRESSURE PEAK LIMIT' CMD=87. The recordings are in a circular fashion and the oldest one is over-written.

**39 CLEAR RESULTS 1**

This command clears all results. Enter 'YES' to clear the result memory. The HELP memory is cleared if the text 'HELP' is entered instead of 'YES'.

**40 CLEAR CAL3K MAX 1**

This command clears the CAL3K maximum recordings. The recordings consist of:

- Maximum Start Temperature and date and time it happened.
- Maximum Vessel Temperature and date and time
- Maximum Ambient temperature and Date and Time
- Max Vessel pressure and date and time (**For CAL3K-AP only**)

Note that the CAL3K Maximums can be printed with CMD=85

**41 CLEAR VESSEL MAX 1**

The maximum recordings in the vessel consist of:

- Maximum Temperature
- Firing count at max Temperature
- Date and time of max Temperature

The vessel must be inserted for the clearing.

The command should be repeated with each vessel.

**42 CLEAR B\_CAL S/N 1**

This command CLEARS all calibrations for the specified vessel Serial Number. The vessel must be removed from the CAL3K. The deletion is permanent. This command should be used when memory space is needed for a new vessel. CMD=106 displays all used vessel serial numbers. Once a vessel calibration is cleared it must be calibrated, See CMD=19.

**43 READ TEMP VESSEL 1**

The vessel temperature is displayed until ESC.

**44 READ DRIFT 1**

The drift (Temperature difference per 6 seconds) is displayed until ESC.

**45 READ CARD ID 0**

The card identification is displayed. The Card ID is used by the factory.

**46 READ MAX VESSEL 0**

The vessel maximum data are displayed. The vessel must be in the well.

**47 READ CAL3K MAX 1**

The CAL3K maximum data are displayed.

**48 READ VESSEL INFO 0**

The information of the inserted vessel is displayed.

**49 READ AMB TEMP. 0**

This causes the display of the present ambient temperature until ESC is pressed.

**50 READ MEMORY RESULT 0**

The unit displays the Last result number and the result details in line #4.

Press **ENTER** to display the result before the last

The result can be printed with the *F8* function key!

**51 READ FIRING VOLT 1**

Type **ESC** to end the firing voltage display. The firing takes place automatically after a pre-set time.

**52 PRT RESULT DAY: 0**

Enter the day number 1-31 and all results recorded at this day are printed. The result memory should be cleared every month, CMD=39.

**53 PRT RTIME HEADER 0**

The CAL3K has a REAL TIME (RT) printout facility. It must be enabled by selecting one of the 4 output formats WXYZ, CMD=61. Then the header (Data descriptor) is printed. If the SET RTHEAD EN CP, CMD=60 has a selection (C=Customer, P=Parameter) then this info id printed as well.

**54 D1 PORT SPEED 0**

This command sets the D1 RS232 port speed. The default is 19200Kb.

**55 SET FILTER C, F: 1**

The print outs can be filtered to exclude Calibrations (C) and Faults (F).

**56 PRT A-H HEADER 0**

The unit has 8 print (Output formats) named A-H. The format can be set by CMD=63, SET OUTFORM A-H.

This command prints the header (a short text description of each format position) so you can see what the data consist of.

**57 PRT TODAY FORM: 1**



Enter an output format A-H and all results for today are printed.

**58 PRT CALORI INFO 1**

The command prints the relevant CAL3K information, including parameters and customer setting. This printout is included in the shipment.

**59 PRT VESSEL INFO 1**

This command prints the vessel information. The vessel must be in the well.

**60 SET RTHEAD EN CP 1**

The REAL TIME output (from 30 seconds before firing) can be preceded by Customer (C) and Parameters (P). If none is desired enter anything but CP.

**61 SET RT FORM WXYZ 1**

This command enables and sets the REAL TIME output format to WXYZ. Selecting anything but WXYZ disables the real time output. The WXYZ format can be tested by printing the header with CMD=53.

**62 SET PRT MEMO. CR: 1**

This command prints results from memory. A 'YES' entry prints calibration and results, a 'C' entry prints Calibrations only and a 'R' entry print result only.

**63 SET OUTFORM A-H: 0**

This command specifies one of the eight output formats A-H. The outputs can be disabled by specifying anything but A-H or by using CMD=86, SET RESULT ENABL=NO.

**64 PRT RESULT GID: 0**

Each result in memory has a GID (Group Identification) of 16 characters. The GID can be an entered text (CMD=3) or the date (CMD=122). Enter the GID (Up to 16 characters) and results are printed.

**65 READ RESULT DAY: 0**

This command displays the number of results for the entered day number 1-31.

**67 PRT CV'S VESSEL: 0**

The Calorific Values (CV) of the specified vessel are printed to the D1 port.

**68 READ STABILITY 1**

The entry is a 4-character vessel serial number, printed on the vessel lid. The complete result memory is searched for this serial number and all results are printed.

**69 PRT RESLTS FORM: 0**

Enter the output form descriptor A-H and all results in the memory are printed. The selected (CMD=63) form remains as specified.

**70 HIGH RESOLUTION 1**

The unit can be switched to high display result resolution by a 'YES' entry. The resolution is:



HIGH	XX.XXXX
LOW	XX.XX

Note that the resolution switch affects the DISPLAY only. All digital outputs depend on output format and destination. An output to a PC has a resolution of: XX.XXXXX

### **71 CONTRAST ADJUST 1**

The LCD contrast is temperature dependant. It can be adjusted on some models. An entry of '0' results in a very dark display, an entry of '15' is very faint. Enter '8' to start.

### **73 OPEN LID 0**

This command applies to the **CAL3K-AP/A/F**. Its command opens the lid. The present process is terminated, and the lid will open when it is safe to do so. The command is the same as the F6 function key.

### **78 READ ATC TEMP. 0**

Only applicable to **CAL3K-AP**. The ATC temperatures (Bottom and Top) are displayed until ESC is pressed.

### **79 DUMP RESULT DATA 1**

This command dumps all results to the D1 port. The data are not printable but can be saved as a NAME.CSV file for a spreadsheet.

The capturing of the data can be done by any terminal emulator like TERA TERM VT. Connect to the PC with an RS232 cable, then configure it for the RS232 baud speed (CMD=54). Of course, you can capture the printable outputs as well as the non-printable. If the data should be useable in a spreadsheet see: OUTPUT TO A SPREADSHEET.

### **81 READ LAST N\_RISE 1**

The last nett rise is displayed after the main period.

### **82 SET OPS MODE 1**

The **CAL3K-A and AP** has four operational modes:

- Dynamic
- Adiabatic
- Isothermal
- FAST Dynamic

Each of these modes has its own calibration, which means the vessel(s) must be calibrated in the new mode.

Each of the modes has its own default parameters. The most preferred mode is 0=dynamic, followed by 3=Fast Dynamic.

### **83 READ VESS. FIRING 1**

The number of firings of the inserted vessel are displayed.

**84 PRT CALIBRA. DUMP 1**

This command dumps all calibrations to the D1 port. The data are NOT printable but are spreadsheet enabled if saved as a NAME.CSV file. Use this command for transmitting a calibration set to the factory for further analysis.

**85 PRT MAX DATA 0**

The CAL3K (Chassis) accumulates maximum data. This command prints all maximums. The maximum readings can be reset by CLEAR CAL3K MAX, CMD=40.

**86 SET RESULT ENABL 1**

This command enables (YES) or disables (NO) the spontaneous printing of results after a determination. The results are entered in memory and can be printed later by any of the print commands.

**87 PRESS PEAK LIMIT 1**

An **CAL3K-AP** Command. The pressure peak limit (Default=70bar) is the max. pressure allowed shortly after firing when the pressure increases proportional to the burning speed. When the pressure exceeds this limit, the event is recorded, and the operator is warned. At this point the unit is NOT shutdown. The pressure peak checking can be disabled by entering zero.

If the pressure increases to 90bar (Internal limit) then the vessel is partial deflated, and the process is terminated. This event is also recorded.

**88 PRESS FILL TRGET 1**

An **CAL3K-AP** command. The filling target is the nominal filling pressure when the filling valve is shut down. The actual pressure may be 1bar down because of pressure settling. The target should be 30bar. Lower filling pressures are indicated for powdery substances to reduce scattering of sample material. The default is 30bar.

**89 READ PRESSURE 0**

CAL3K-AP command. The actual vessel pressure is displayed until ESC.

**90 PRESS ZERO ADJST 1**

An **CAL3K-AP** command. The pressure is measured with a transducer which has a zero offset of +-1bar. This error can be eliminated by using this command. The offset can be checked when the lid is open and the 'READ PRESSURE', CMD=89., it should read ZERO. +-0.2bar. If not use the 'PRESS ZERO ADJ' to set it to zero.

**91 DEFLATE SYSTEM 1**

The pressure in the **CAL3K-AP** piping is deflated by opening all valves until ESC.

**92 PRESS DEFLAT. SEC 1**

The command applies to the **CAL3K-AP**. After the main period the vessel is deflated. The time is monitored and if the limit is exceeded a warning is displayed. It could be that the filter or the exhaust valve is blocked. The default setting is 20 seconds.

**93 PRESS FLUSH SEC 1**





The vessel can be flushed with oxygen after deflating. The flushing removes harmful gases from the pipes and vessel. The flush cycle is time based. A zero entry eliminates the flush cycle, otherwise the vessel is filled for the specified time in seconds and then deflated. Default value is 3 seconds. In the **CAL3K-S/ST** models the flushing is applied to the vessel well before a determination with the FAN.

#### **94 SYSTEM LEAK TEST 1**

Note that the filling is much slower because the vessel is filled. Monitor the pressure and if it is steadily declining than a leak is present. It is normal that the pressure declines rapidly in the beginning and then settles down. This is because a flow restriction is present in the vessel lid.

#### **95 PRESS FILL LEAK 1**

This command applies to the **CAL3K-AP**. After filling the vessel with oxygen, the pressure is monitored and if it declines (leaks) by more than the limit a warning is issued. Theoretical the pressure should be constant, but it requires approx. 1 bar to equalise. The default setting is 1.5bar.

#### **96 PRESS FILL MIN 1**

An **CAL3K-AP** command. The minimum filling pressure specifies the limit at which the unit stops working. It allows for slow filling and near empty oxygen bottles. The default is 20bar, which is sufficient for avoiding oxygen starvation at 26KJ/g Samples.

#### **97 PRESS FILL SECND 1**

An **CAL3K-AP** command. The filling time is monitored and when it exceeds the limit the operator is warned. The reason for slow filling could be: Empty oxygen bottle or blocked inlet filter. The filter (and filling restrictor) are a very unlikely reason for slow filling and the oxygen pressure should be investigated first.

#### **99 PRESS LEAK TEST 1**

An **CAL3K-AP** command. This command sets the leak test pressure applied during SYSTEM LEAK TEST, CMD=94. The leak test is useful when a leak is suspected. The default value is 25bar.

#### **101 PRT PRESS. RECRD 1**

An CAL3K-AP command. This command prints all (Max 16) pressure recordings which are caused by either:

- Exceeding the PRESS PEAK LIMIT
- Exceeding the internal max pressure setting 90bar

The pressure recordings can be reset by CLEAR PRESS RCRD, CMD=38. The records can be read out on the display with CMD=115 and a junk record can be simulated with CMD=126.

#### **102 LOW AMBIENT LIMIT 1**

The ambient temperature affects the unit's operation and result. The result influence can be eliminated by calibrating the unit at low ambient temperatures. The default setting is 8C. The lowest possible temperature measurement is 2C.

**103 HIGH AMBIENT LIM 1**

The unit measures the Ambient temperature, which has an influence on the result and the operation. The influence is mitigated by calibrating the unit at relatively high ambient temperatures. However, this limit can be set up to warn the operator. The default setting is 36C.

**104 LOW RISE LIMIT 1**

The temperature rise after firing should be 12C±2C. This can be managed by adjusting the sample mass. A limit can be set up to warn the operator that the sample mass was too low. The default setting is 8C. A lower rise can be achieved by calibrating the unit to an 8C±2C. It is just difficult to split a Benzoic Acid tablet to 2/3.

**105 HIGH RISE LIMIT 1**

The result is affected by the temperature rise after firing. This affect is minimised by calibrating the unit for high rise (e.g., 0.75g Benzoic Acid). However, the sample mass should be adjusted so that the temperature rise is near to the optimal 12C ±2C. The default setting is 16C.

**106 READ BOMB CALI 1**

The number of calibrations (Max=15) per vessel are displayed for each vessel.

**107 PRT KEY CMD LIST 1**

The command prints all installed keyboard commands as follows:

CMD NUMBER  
KEYBOARD ENTRY  
PRIORITY

**112 PRT HELP HISTORY 0**

The CAL3K records all major operational or setup faults. This command prints all recorded events. After 64 events the unit overwrites the oldest one. The printout consists of:

Message Number            Mxxx (M Number)  
Date and Time

**113 COMPENSATE BOMB 1**

It requires an inserted vessel or the vessel number. It will analyse the calibrations and computes the compensations for this vessel.

Short display text

**114 READ HELP TEXT 0**

Each fault or operation has a message number attach, the number starting with a 'M' followed by 3 numerical digits. If it is required to read the help text type the command and number.

Example:     114, enter, 524

**115 READ PRESS. REC 1**

This command reads the recordings from the memory. Press {enter} for the next and ESC to terminate reading. The pressure recordings can be cleared by CMD=38 and printed by CMD=101. A pressure record can be simulated with CMD=126, but the data are junk.

**120 PUT FAN ON 1**

This test **does NOT** apply to the **CAL3K-F**. This command puts the FAN on.

**121 TIME IS SID 1**

The Sample Identification (SID) is a 16-character text string. It can be replaced with the time in HH:MM:SS by entering 'YES'. This is not available on the **CAL3K-S**.

**122 DATE IS GID 1**

This YES/NO entry allows for replacing the GID with the DATE in the form YY/MM/DD. The GID (Group Identification) is attached to every sample recording. Note that the first 8 characters of the GID are left blank. This is not available on the **CAL3K-S**.

**123 VERSION 0**

This command displays the CAL3K firmware version.

**124 RESET 0**

The same as ABORT. Any process is terminated! A Sample in progress is lost, Balance mass is lost. This command applies to the **CAL3K-AP ONLY**. It simulates a pressure event record in memory. See 'PRESSURE' section for details.

**125 LOAD DEFLT COMP 1**

The default compensation factors from default.

**147 DUMP RSLT VESSEL 1**

The results of the specified vessel are dumped to the D1 port.

**27 TEMPERATURE RISE**

The vessel is calibrated with 0.5g BA which raises the vessel temperature by approx. 10C. This rise should be achieved when a sample is burned. That it turns means that the sample mass should be adjusted so that it yields approx. the same rise. The CAL3K can 'tolerate' a range from 8 to 16C rise when calibrated with 0.5g of BA. Adjust the sample mass as follows:

Benzoic Acid	26.454KJ/g	0.5g
Food	~17KJ/g	0.75g
Other	<10KJ/g	1.0g
Oil, Fett, Fuel	~37KJ/g	0.3g

The following commands are valuable:

**81 READ LAST N\_RISE 0**

This reads the Net Rise of the last sample. The display is valid until a new vessel is fired.

**27.1 VERY SMALL SAMPLE MASS**

Sometimes a sufficient sample mass is not available. Then a vessel should be calibrated with less than 0.5g BA. It is a bit messy and not convenient to split a 0.5g tablet to ~0.2g or less, but it can be done.

BA Calibration Mass	Rise	Rise Range
~0.2g	5C	3-8C
~0.1g	2.5C	2-4C

Less than ~1C rise is not advised because the errors get out of hand.

If the calibration mass is changed then the calibration memory must be cleared, and some calibrations must be performed with the low mass. If more than one vessel is used, then all vessels' calibrations must be cleared and fresh calibrated.

The following parameters must be set to confirm with the changed calibration:

**104 LOW RISE LIMIT 1**

The default is 8C. Change to suit!

**105 HIGH RISE LIMIT 1**

The default is 16C. Change to suit!

**30 MIN MASS LIMIT 1**

The default is 0.2g. Change to suit!

**31 MAX MASS LIMIT 1**

The default is 1.5g. Change to suit!

**27.2 HOW TO MEASURE THE WIRE+COTTON RISE**

The default setting is ~0.06C. This is not very important because it is an offset to the raw temperature rise.

Disable the Miss-fire Limit with:

**21 MISSFIRE LIMIT 1**

The default setting is 0.44C. Enter '0', this disables the misfire limit!  
Then clear the Cotton correction.

**9 COTTON+WIRE CORR 1**

Enter '0.0'.

Prepare the vessel (without a sample) and run a determination. After the complete run read:

**81 READ LAST N\_RISE 0**

This reads the Net Rise of the last sample. Since the cotton correction=0 it is the RAW TEMPERATURE RISE.

Enter the obtained reading with CMD=9.



### 27.3 HOW TO MEASURE THE GELATIN CAPSULE RISE

The gelatin capsule rise is needed when EASY SPIKING is used. In this method the sample is packed inside the capsule and the capsule rise is deducted from the net-rise. Proceed as follows:

Disable the misfire limit:

**21 MISSFIRE LIMIT 1**

Default setting is 0.44C. Enter '0', this disables the misfire limit!

Prepare a vessel with a gelatin capsule but without a sample and run a determination. After the run obtain the temperature rise:

**81 READ LAST N\_RISE 0**

This reads the Net Rise of the last sample. This is the temperature rise produced by the gelatin capsule. Enter this value in:

**29 CAPSULE RISE C 1**



## 28 MAINTENANCE

The DDS Calorimeters are designed to work in a laboratory. However, all laboratories are different, and the maintenance depends largely on:

### 28.1 DAILY USE

This works out to samples per day. The maintenance of the system is fairly in line with the usage of the system.

### 28.2 MATERIALS TESTED

This is the biggest maintenance contributor. The materials differ from powdery substances (which may have explosive combustion) to environmental hazardous samples. They all affect the O-rings. The powdery substances are the worst: The material settles on the O-ring surface and 'cracks' the O-ring. Corrosive materials effect the crucible and the deflector plate. But the effects are highly visible and can be sustained.

### 28.3 ENVIRONMENT

Dust, temperature, installation conditions also affect the system and maintenance. Dust being the main influence on the calorimeter. It blocks the fan (in the cooler and calorimeter) and is electrically conductive if it is coal dust.

### 28.4 MAINTENANCE SCHEDULE (GUIDELINE)

The following maintenance schedule serves as a guideline only: Please adjust it to suit your conditions.

The following Vessel parts require maintenance:

- Large Lid O-ring (3K-4-094)
- Centre electrode (3K-4-122)
- Outside electrode (3K-4-037)
- Firing wire (3K-4-093)
- Top & Bottom O-ring (3K-4-022)
- Nozzle O-ring for CAL3k-AP (3K-1-080) (Black)
- Nozzle O-ring for CAL3k-AP ONLY (3K-4-107) (High quality)(Green)
- FAN (Both for Cooler and Calorimeter)
- Crucible (3K-4-047)
- Deflector Plate (3K-4-092)
- Nozzle O-Ring for filling station (3K-3-29)



**When to change consumables:**

VESSEL CLEANING: After each sample: wipe the vessel clean with paper towel

LARGE LID O-RING (3K-4-094):  
Worst conditions: 2-5 samples  
Average conditions: 40-50 samples  
Good conditions: 100+ samples

CENTRE ELECTRODE (3K-4-122): 20-50 samples

OUTSIDE ELECTRODE (3K-4-037): 30-120 samples

TOP & BOTTOM O-RINGS (3K-4-022):  
Worst conditions: 2-5 samples  
Average conditions: 40-50 Samples  
Good Conditions: 100+ samples

**CAL3K-AP O-rings:**

3K-AP NOZZEL O-RINGS (3K-1-080):  
Worst conditions: 10-20 samples  
Average conditions: 20-50 samples

3K-AP NOZZLE O-RING ONLY (3K-4-107): Worst conditions: 50-100 samples

FAN CLEANING: WEEKLY to MONTHLY depending residue build up

CRUCIBLE (3K-4-047):  
When weight loss >10%  
When bottom distorts and becomes completely discoloured

DEFLECTOR PLATE (3K-4-092): 30-150 samples  
Weekly to Monthly, or when centre electrode O-ring is changed. Wipe top of deflector plate clean weekly.

FILLING STATION NOZZLE O-RING  
(3K-3-29): 50-100 samples



## 29 TROUBLESHOOTING

### 29.1 ERROR – CALORIMETER DISPLAYS ##### INSTEAD OF THE RESULT

Remove the vessel from the calorimeter.

Type "xx" and Enter

Type "10", Enter and "yes" enter. This is to load the default factory values back into the calorimeter.

Type "42", Enter and "yes" Enter. This will clear all the calibrations on the calorimeter.

Place the vessel back into the calorimeter well.

Type "125", Enter and "yes" Enter. This is loading the default vessel compensation values into memory.

Now prepare the vessel for calibration and enable calibration on the calorimeter by typing "calibration", enter and "yes" Enter.

Let the vessel go through the entire cycle. Remove the vessel and prepare for another calibration.

Before placing the prepared vessel into the vessel well, delete the calibration that has just been performed. To do this, type in "xx" "42", Enter and "yes" Enter.

Now proceed to calibrate the vessel again. At least 5 calibrations will need to be performed before a normal sample can be analysed. Please always calibrate with benzoic acid supplied.





## 30 WARRANTY – FOR NEW PRODUCTS

### 30.1 PURPOSE OF PRODUCT WARRANTY

Any part of a product is subject to ageing and eventual failure. These failures occur during normal use. The purpose of a warranty is to protect the Owner against a part failure caused by manufacturing defects in workmanship, material, or design for a specific time.

### 30.2 OWNER'S WARRANTY

In the event of the Owner contracting directly with Digital Data Systems (Pty) Ltd “**dds**”, or in the event of the installation of a unit arising out of a contract between the Owner’s Dealer or any other party on his/her behalf and the said “**dds**”, the Owner records that notwithstanding same, this warranty is the only warranty applicable to “**dds**” units and is expressly in lieu of all other warranties, expressed or implied, including any implied warranty of merchantability or fitness for a particular purpose. No amendments or additions to this warranty shall be binding on “**dds**”, unless recorded in writing and signed by a duly authorised officer of the company. **“dds” agrees to repair and replace with a new or remanufactured part, any part, other than those excluded below, which after inspection, upon its return to the factory in Randburg, Johannesburg by the Owner, or the authorised Dealer to which he/she returns the unit, is proven to be defective.**

Replacement of a defective part will be made only within the applicable warranty period of **12 (twelve) months from date of shipment**. Notwithstanding anything to the contrary herein, any labour involved in repairing or replacing a defective part will be charged at “**dds**” rates applicable at the time.

**If upon inspection it is found that the pressure vessel was submerged in water the warranty will automatically be null and void and the owner will be responsible for the labour and parts charges.**

### 30.3 EXCLUDED (NON-WARRANTY) PARTS

“**dds**” shall be entitled to treat this warranty as void unless the relevant warranty form (attached) is sent by registered post to “**dds**” by the Owner within 10 (ten) days from date of original installation.

Regardless of warranty-status, the Owner shall be responsible for labour charges connected with periodic maintenance requirements involving visual and functional inspection of the systems.

This warranty expressly covers the unit(s) “**dds**” supplied only and does not cover any other part, which may, as a consequence of the installation and operation of the unit, become damaged in any way whatsoever.

**Special attention should be given to the safe operation of the high-pressure Vessels. The published rules are guidelines only and enclosed in this leaflet for the convenience of the User. The manufacturer is not responsible for any damages, consequential, indirect, or direct, or loss whatsoever, resulting out of electrical shock, Vessel explosion, or any other cause.**



### 30.4 WARRANTY – REPAIRED PRODUCTS

Any “dds” scientific product that has been repaired has a warranty period of **30 Days from date of shipment** for the Repair request only. If something else goes wrong that is not part of the original Repair, the warranty does not apply. Each product that is sent to “dds” for Repair is tested and checked and leaves “dds’s” Factory in 100% working order. It is the Owner’s responsibility to inspect any repaired product on receipt thereof and confirm in writing, any faults, errors, or problems with the said product. If a fault, error, or problem is reported after the 30-Day warranty period, and “dds” finds that the product has been damaged and requires another repair, the full cost of repair will be applicable and paid for by the Owner.

### 30.5 GENERAL – WARRANTY VOID

All serial numbers on any relevant “dds” Scientific product need to remain on the product at all times, or warranty will be void. Warranty will also be void if any of the branding stickers or decals have been removed and/or the colour of the Scientific product has been changed in any way.

## 31 RETURNS / REPLACEMENTS POLICY

When returning any of our bomb calorimeter products, please use either a reputable Courier Company or Air Freight Company. We suggest returning the good using the Door-to-Door Courier Service or Air Freight Shipping Service.

---

For returns via a Courier or Air Freight Shipping Company:

Digital Data Systems (Pty) Ltd.  
22 Arbeid Avenue  
Strydom Park  
Randburg  
2196  
Gauteng  
South Africa

---

**PLEASE ENSURE THAT YOU INCLUDE A ZERO VALUE INVOICE AND A DESCRIPTION OF THE "RETURN"/"REPAIR" FOR CUSTOMS PURPOSES. A LOW VALUE FOR CUSTOMS PURPOSES MAY BE INCLUDED.**

---

All returns must be sent pre-paid (customer account) and no collect shipments will be accepted. Please ensure the parcel for its full value.



Once we receive the returned products, Digital Data Systems will inspect and test the products for manufacturing faults. If we determine that there is a manufacturing fault, we will repair or exchange the product and pay for the shipping costs to send the repaired/replacement product back to the customer.

Replacement of a defective product will only be made within the applicable 12 (twelve) months warranty. Notwithstanding anything in the contrary herein, any labour involved in the repairing or replacing of a defective product will be charged at the "DDS" rates applicable at the time.

Repair or replacement of a defective product will only be done once the product is received and inspected at our DDS factory at the above address.

---

***No Refunds will be issued by DDS under any circumstances. Only Replacement products will be offered by DDS.***



**32 WARRANTY REGISTRATION FORM**

**SERIAL NUMBERS (S/N):** \_\_\_\_\_

**Calorimeter:** \_\_\_\_\_

**Air Cooler:** \_\_\_\_\_

**Vessel(s):** \_\_\_\_\_

**Other:** \_\_\_\_\_

**DEALER NAME:** \_\_\_\_\_

**OWNER'S/CUSTOMER'S COMPANY NAME:** \_\_\_\_\_

**AUTHORISED BY:** \_\_\_\_\_

**INSTALLED AT:** \_\_\_\_\_

**TEL NO:** \_\_\_\_\_ **FAX NO:** \_\_\_\_\_

**CONTACT PERSON:** \_\_\_\_\_

**INSTALLED BY:** \_\_\_\_\_

**DATE INSTALLED:** \_\_\_\_\_ (REPRESENTATIVE OF DEALER/AGENT)

**CUSTOMER ORDER NUMBER AND DATE:** \_\_\_\_\_

**INSTALLATION DETAILS (LOCATION, WATER SUPPLY, AIR-CONDITIONING ETC.):**

\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

**ACCEPTED IN GOOD CONDITION:**

**DATE:** \_\_\_\_\_ **SIGNATURE:** \_\_\_\_\_

**NAME (printed):** \_\_\_\_\_

**WITNESS:** \_\_\_\_\_

I have read, understood, and accepted the conditions of this warranty, the details of which are printed in this Manual.

**CUSTOMER'S SIGNATURE:** \_\_\_\_\_

**NAME (printed):** \_\_\_\_\_

**DATE:** \_\_\_\_\_