

CAL³k

NEXT GENERATION CALORIMETERS

DDS CALORIMETERS OPERATIONS MANUAL

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DDS CALORIMETERS

Scientific Analytical Calorimeter Solutions



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DDS CALORIMETERS

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CAL3K Advanced Bomb Calorimeters
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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 V6 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 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.

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 loose inside the crucible. They can explode 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 in to 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

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. If the vessel fails to deflate use the pre-scribed 'emergency deflate' procedure as outlined later on 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 defiller 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 MUST 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.



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) |
| 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 of these commands as you like. That is to say without reading the rest of this manual.

The **CAL3K-A and CAL3K-AP** can operate in 4 different modes. All other units operate in one mode and the following command is not required. Without explaining the modes set the mode to ZERO, which is the DYNAMIC MODE. For details on mode changes see further on in this manual MODE CHANGE.

The keyboard commands below will be written in a different font, italic and indented.

82 SET OPS MODE 1

The CAL3K has four operational modes:

| | |
|---|--------------|
| 0 | Dynamic |
| 1 | Adiabatic |
| 2 | Isothermal |
| 3 | Fast Dynamic |

To reset the CAL3K to a working condition you need to install the default parameters by:



10 *PARA DEFAULTLOAD* 0

This command loads ALL default values in to 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 to operate the unit on the default parameters unless a change is absolutely necessary.

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:

- | | |
|--------------|--|
| ESC | Terminate, start fresh |
| | Enter a NO, YES, Number, text |
| ENTER | Make the change permanent: The NO, YES, Number, text 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.

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-F**. This command puts the FAN on.

120 PUT FAN ON 1

5.4 TESTING FOR OXYGEN LEAKS

CAL3K-AP only : Applies to 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 (30bar 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 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. 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.
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 **CAL3KA/AP** have four operating modes, all other units work in mode=0.

| Mode | | Initial time, Minutes | Main Time, Minutes |
|--------|--------------|-----------------------|--------------------|
| Mode=0 | DYNAMIC | 3 | 4 |
| Mode=1 | ADIABATIC | 3 | 5 |
| Mode=2 | ISOTHERMAL | 4 | 5 |
| Mode=3 | FAST DYNAMIC | 2 | 3 |

Each mode (and Vessel) must be calibrated if you intend using this mode. Once calibrated the mode can be changed without re-calibration. Note that a max. of 4 vessels are allowed per mode.

The **CAL3K-S/ST** have one vessel or two respectively.

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 mode=3 FAST DYNAMIC can be used without any decrease in accuracy and repeatability if you use four vessels and operate in airconditioned ambient temperature.

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.

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



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. This App allows the display of data, the setting of parameters, monitoring of conditions and the retrieval of results for storage on the PC. In general terms a PC is not needed, but it is nice. **USB only available on CAL3k-AP/A/F.**

8.2 PORTD1

This is a RS232 port and good for everything as seen later on. Of course, it can be connected to a PC but it is called the printer port.

8.3 PORTD2

This port is a RS232 port **only available on the CAL3K-AP/A/F.** Its speed is set at 115.2KB. Its main purpose is to connect to a BLUETOOTH bridge.

72 **BLUETOOTH NAME** **1**

This entry is important if you are using a BLUETOOTH bridge and if you have more than one BLUETOOTH device. The name identifies the link to your PC.

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 is: Data=8bit, Parity=none, Stop bit=1 (Standard).

The port speed is settable by:

54 **D1 PORT SPEED** **0**

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

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 the DDS Windows Software App must run such as TERA TERM or any virtual port emulator. 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 App 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)

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

If a BLUETOOTH BRIDGE is used then the D1-CAL3K port speed must set to 115200 by CMD=54.

The BLUETOOTH BRIDGE has a special application: It converts the RS232 (CAL3K) to a USB (PC). In other words, if your PC (laptop) hasn't got a RS232 port then the bridge will work.

8.8 CONNECTING TO A BALANCE

This step is not absolutely 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 being connect up. 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.



9 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 | (and BLUETOOTH) |
| D2 RS232 | BLUETOOTH | |
| USB | PC APP | |

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

54 D1 PORT SPEED 0

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)

9.1 OUTPUT PRINTING RESULTS TO PAPER, KEYBOARD INITIATED

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

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.

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.

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 PRT A-H HEADER 0

The unit has 8 print (Output formats) named A-H. The format can be set by

77 SIMULATE RESULT 1



This command simulates a result (junk data). If the print output is enabled (CMD=63, SET OUTFORM A-H) and a printer is connected then the junk data appear on the output.

The following print outputs are possible:

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.

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.

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.

57 PRT TODAY FORM: 1

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

55 SET FILTER C,F: 1

A filter can be applied to the print output

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



9.2 OUTPUT PRINTING, KEYBOARD INITIATED, OTHER

The CAL3K can supply the following information:

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.

107 PRT KEY CMD LIST 1

The command prints all installed keyboard commands as follows:

CMD NUMBER
KEYBOARD ENTRY
PRIORITY

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.

112 PRT HELP HISTORY 0

This feature applies to CAL3K_AP, A, F

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

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.



9.3 OUTPUT PRINTING, INTERNAL INITIATED

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 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 is printed as well.

9.4 OUTPUT, NON-PRINTABLE

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

84 DUMP CALIBR. S/N: 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.

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 a 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.

9.5 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 to run on the PC; it could be:

- A) The DDS Windows Software App (supplied)
- B) A PC terminal emulator or other communications App

A PC with the DDS CAL3K App can be connected via:

- 1) RS232 cable
- 2) USB cable
- 3) BLUETOOTH BRIDGE

A Terminal emulator requires a RS232 cable connection.

9.6 OUTPUT TO A SPEADSHEET

An output to a spreadsheet can be facilitated in a round-about fashion by using a standard terminal emulator like TERATERM VT. Then the data can be transferred to a spreadsheet via its own file saving method or transferring it to the Windows Notepad. Important is that the data are saved on the PC in file NAME.CSV, which is readable by the spreadsheet.

9.7 OUTPUT TO AN EXTERNAL PC

This is not for the faint hearted. You need to be a software nerd to understand the following command structure which request data from the CAL3K.

Each CAL3K has a two-letter type identification:

| | |
|----------|----|
| CAL3k-AP | CP |
| CAL3K-A | CA |
| CAL3K-F | CF |
| CAL3K-ST | CT |
| CAL3K-S | CS |

Then a command must be transmitted to the CAL3K, which starts with the two-letter type ID followed by the actual command:

| | |
|----------|--|
| __X | Reset the unit (Do not use! It will terminate the present operation) |
| __HU;a;l | Upload results form 'U' from record 'a', length 'l' |
| __HN;a;l | Upload results form 'N' from record 'a', length 'l' |
| __HI;a;l | Upload results form 'A-H' from record 'a', length 'l' |
| __HH | Upload form 'U' header |
| __HK | Upload form 'N' header |
| __HJ | Upload form 'A-H' header |
| __HS;n | Upload results day 'n' form 'U' |



__Hm;n Upload results day 'n' form 'A-H'
__HC Clear all results
__HZ Request results stats: Amount per day
__HF;x Set output form 'A-H' to x

The above commands must be transmitted without a time gap and without C/R, L/F

Example: CAL3K-AP:
CPHU;0;5 Uploads 5 results starting at record 0, form 'U'

For more PC commands consult the factory.

The above commands are used by the DDS Windows Software App.

10 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.

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 un-equalized temperatures are the result of filling and handling of the vessel.

| | |
|-------------------------------|------|
| Vessel communication | M210 |
| Initial cycle interruption | M216 |
| CAL3K-AP: 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 |
| CAL3K-AP: Pressure Abort | M382 |

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

10.5 CYCLE: END

| | |
|-------------------------------------|------------------------|
| CAL3K-AP: 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) is 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, 524

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). The password is '**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 0.9 grams. 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.

11.4 MASS ERASING

Once the mass has been entered, it is 'LOCKED' in to 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



11.5 SAMPLE IDENTIFICATION

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

The Group ID is a 16-character (or shorter) string of characters. The GID is right orientated. If the date is used as GID then only 8 characters are used: YY/MM/DD

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

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. Then '**ENTER**' must be pressed to LOCK the mass in to the calorimeter.

11.8 OPEN LID

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

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

17 **FIRE MANUALLY** 1



11.10 READ RESULT

This command is required to read the previous stored results. The 'last one first' result can be retrieved by:

50 READ RESULT MEM 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

11.11 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**)

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.

11.12 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 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:

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

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.

We know that the KBTU/lb is not conventional, but they fit nicely in to 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 calculates the compensation factors. These 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.

After about 8 calibrations the calibration management starts. Each calibration is compared to all the stored ones and is rejected if it does not fit or it gives no benefit. If a calibration must be deleted because the memory is full then the oldest calibration is deleted to make space for the new one.

12.1 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 to four. However, sample determinations are possible with less than 4 calibrations.

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 the 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.

12.2 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. 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 | 4 | 64 |
| CAL3K-ST | 1 | 2 | 2 | 32 |
| CAL3K-S | 1 | 1 | 1 | 16 |

12.3 PERFORMING A CALIBRATION

Prepare the vessel with Benzoic Acid (BA) and set the calibration on (YES).

19 CALIBRATION 0

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.

The first calibration after a cleared memory is a true calibration. The second calibration is a 'CALIBRATION and check sample combined.

12.4 CALIBRATION AND RESULT GRADING

The result (and calibration) quality is judged by the compensation amount in Degree C. See: COMPENSATIONS. The compensations are judged by:

- A) Total compensation. The sum of all compensations: Total Compensation
- B) The ABSOLUTE sum of all compensations: Maximum Compensation



A Max. Compensation of 0.05C results in approx. 0.1KJ/g which is just about acceptable.

| Grade Number | Grade Letter | Meaning |
|--------------|--------------|------------------------------|
| 0 | an | Excellent |
| 1 | b | Good |
| 2 | c | Acceptable |
| 3 | d | Poor, Calibrate |
| 4 | e | Bad! Calibrate |
| 5 | f | Disaster? Nearly impossible! |

12.5 AVERAGE CALIBRATION

DO NOT USE IT!

The averaging of calibrations is a historical requirement. It was left in operation so that we can claim we have it. The AI calibration process dynamically performs a 'sort off' average by looking at ALL calibrations.

The unit select the 'BEST FIT' calibration for use with a sample. If average calibration is enabled by setting it to a number higher than ZERO, then the next 'BEST FIT' calibrations are averaged and used for the determination. The Bomb Factors (Heat Capacity) are averaged and used for this operation.

32 AVERAGE CALIBR. 1

The average calibration makes sense if you have more than 4 calibrations stored, because the AI (Artificial Intelligence) selection process needs to check that the calibrations are acceptable. The acceptance window is specified with:

26 AVERAGE ACCEPT. 1

The entered value should follow the following grading guide:

| Value | Means |
|-------|----------------|
| 0.012 | Excellent |
| 0.024 | Good |
| 0.048 | Acceptable |
| 0.096 | Poor |
| 0.192 | Bad! Calibrate |

A 'ZERO' entry (Default) disables the acceptance check. A practical example:

Average calibration: 2

Average acceptance: 0.024

Then the average will be performed with THREE calibrations if all are excellent to go.



12.6 CALIBRATION SUMMARY

The preceding pages explain calibration. This section is the summary of all and the factory suggestions:

- A) **Do not use average calibration ever.**
- B) In the first week of use do three calibrations per day at various times, then one every second day
- C) Calibrate after you changed any vessel component

12.7 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.

12.8 CALIBRATION FREQUENCY

Starting with a CLEARED calibration memory you need calibrate the unit 4 times during the day. 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 experiment with.

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

After about 8 calibrations the AI will analyse your calibration performance and may even reject a calibration because it is not good or it contains no new information.

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 16th 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. For Details see: **COMPENSATION**

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 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). If a gelatine capsule is used to contain powdery substances, then the gelatine capsule compensation can be enabled. The gelatine capsule has a standard weight, which is tarred and results in a repeatable temperature rise. The temperature rise of the capsule is deducted from the total temperature rise. This method is reasonable accurate and very convenient.

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 GELATINE CAPSULE 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 as a result of:

- 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 contents. 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 SO₂ 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 SO₂ formation must be entered:

**8 *SULPHUR CORRECT.* 1**

The sulphur correction deducts energy which is released by the formation of SO₂. 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:

28 *GELATINE CAPSULE* 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 a pre-set time. The sample can be fired manually:

17 FIRE MANUAL 1

The firing wire can be heated at any time as long as the vessel is in the well and the firing wire is present.

The manual firing should be avoided during routine operations.

16 COMPENSATION

Compensations are a set of factors which are calculated after each calibration. They describe how a result should be treated if the present conditions differ from the calibration conditions.

In simple practical terms: The ambient condition has changed by 1C down then the result is 0.001C up. Of course, it is more complex. Mainly because there are 5 conditions with various effects and various magnitude. And, there are many (max 15) calibrations to choose from.

The compensation factors are calculated by an AI (Artificial Intelligence) module after each calibration when there are more than four already saved.

These compensation factors change with:

- Vessel construction: Deflector plate, Crucible holder, firing wire length, cotton length
- CAL3K construction: Ambient temperature, ATC temperature (if installed), Ventilation
- Vessel age and surface condition



The total amount of the compensation is typically less than 0.05C, which results approx. in a 0.1KJ/g result adjustment if the mass was 0.5g.

To have a balanced compensation it is prudent to calibrate the unit at various times of the day and operating conditions, such as:

- First time after the equipment was not used for 8 hours
- At high/low ambient temperatures
- At high/low vessel temperature
- During routine determinations
- Whenever something was changed

Note that after each calibration a new set of compensations is calculated from the sum of all available calibrations.

The above may sound that you continually calibrating the unit. If the calibration memory was cleared you would do that, but after about 8-10 calibrations you need to calibrate once a day to once a week. The unit was calibrated about 8-12 times before shipment. You can use these calibrations or you can delete them and start fresh.

For more details see: CALIBRATION

16.1 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.

16.2 FOOD COMPENSATION

Traditional food was burned in a combustion calorimeter, although a body uses a different process. This is best explained by burning 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 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.

17 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 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.



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.

18 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). 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.

The time can be used as a Sample Identification (SID). This is not available on the **CAL3K-S**. 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'.

19 3K-AP PRESSURE COMMANDS

This section applies to the **CAL3K-AP only** which automatic oxygen filling. The 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 thru 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. Faster burning material have 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.

At the end of the main period the pressure is compared to the filling pressure and if this loss exceeds a settable limit a warning is issued.

98 PRESS FILL LOSS 1

The pressure filling loss is measured from just before firing to the end of the main period. It should be less than 2bar. If it is more than the vessel leaked when the pressure. This check can be disabled by entering zero. If the vessel leaks than this would show up in this measurement. A leaking vessel produces a LOW result.

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

92 PRESS DEFLAT. SEC

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. 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 vessel can be flushed with oxygen after deflating. 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 (Cooling) is applied 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, 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 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.

20 PRINTING

The unit has a 'PRINTING' port: RS232 PORT D1.

To operate the RS232 port the speed of the CAL3K output must match that of the receiving input. The receiving input can be:

1. PRINTER, with RS232 input
2. PC, with RS232 input
3. RS232 to USB converter

Whatever you connect to, the RS232 output speed must match the receiving input speed. The other settings are:

| | |
|-----------|-------|
| Data | 8 bit |
| Parity | NONE |
| Stop bits | 1 |

The following section refers to a print output initiated from the keyboard.

For other operations see: PRINTING REAL TIME
PRINTING SOLICITED

20.1 PRINTING: SETTING THE PORT SPEED

The D1 port is a SERIAL RS232 port. The port D1 speed can be set with:

54 D1 PORT SPEED 0

20.2 PRINTING: SETTING A PRINTOUT FORM

The printer output comes in 8 forms, labelled A-H. Choose a printer output form by:

63 SET OUTFORM A-H 0

It is possible that some output forms change in future. It is therefore advisable that you check the output forms A-H for suitable data in a printable form. Use:

57 PRT TODAY FORM: 0



Enter any output form with the letters A-H and see the printing of the header and today's data.
Or print result from memory (any day) with:

69 PRT RESLTS FORM: 0

Results are printed. If no results are in memory, then put one in:

77 SIMULATE RESULT 0

20.3 PRINTING A FORM HEADER

A header is a text string, which identifies each data column. The identifications are 'cryptic' but you get the idea:

56 PRT A-H HEADER 0

20.4 PRINT FILTER

If required a filter can be applied before printing. Results (memory entries), which are:

Calibrations

Faulty determinations

can be suppressed with the following command:

55 SET FILTER C, F: 1

20.5 PRINTING RESULTS FROM MEMORY

All results in memory can be printed with the provision:

C Calibrations

R Results

YES, Calibrations and Results

CR Calibrations and results

62 SET PRT MEMO. CR: 1

20.6 PRINTING RESULTS BY GID

If required all results with the specified GID can be printed.

64 PRT RESULT GID 0

Note that the FULL GID is required. If GID = Date is set then all results of a particular date are printed. The print format may not show the GID or not all of it.



20.7 PRINTING ENABLE

This command enables/disables the spontaneous printing.

86 SET RESULT ENABL 1

20.8 PRINTING TODAY'S RESULTS PER VESSEL

All the results, which have today's date stamp and are done on the specified vessel can be printed by:

67 PRT CV' S VESSEL: 0

Enter the vessel number (4 characters).

20.9 PRINT TODAY'S RESULT IN FORM A-H

All results of today can be printed in the specified form by:

57 PRT TODAY FORM: 0

Enter a form A-H. The above command does not change the output form selected by the 'CMD=63, SET OUTFORM A-H' command.

The day number is the calendar day number 1-31.

52 PRT RESULT DAY: 0

If more than one month of results are in memory then the results with the same day-number but different month are printed. The results can be cleared via 'CLEAR RESULTS'.

20.9.1 PRINT CAL INFO

A printout of the CAL3K configuration and setup can be obtained by:

58 PRT CALORI INFO 0

This printout shows all the relevant information of the CAL3K.

20.9.2 PRINT VESSEL INFO

This printout shows all information of the inserted vessel:

59 PRT VESSEL INFO 0



20.9.3 PRINT VESSEL CALIBRATION

The calibrations of the specified vessel serial number are printed. Places without a calibration are left blank. The printout shows the date and time, the temperatures and other relevant information which is important when performing a determination.

111 PRT E_CALIBRA. SN 1

Enter the vessel serial number and all available calibrations are printed. The vessel must be out of the well.

The printout does not require any format information and the output format is fixed.

20.10 PRINT ALL KEY COMMANDS

All AVAILABLE keyboard command can be printed. The commands are preceded with the Index number, followed by the keyboard command text and by the accessibility:

- 0 No password required, always visible
- 1 Password (DDS3K) required to operate
- 2 Not accessible, reserved

107 PRT KEY CMD LIST 0

21 PRINTING TO A PC

Sometimes it is required to have the CAL3K data on a spreadsheet as opposed to a paper copy. The CAL3K can provide spreadsheet data activated from the keyboard. The operation is the same as activating a printable form output.

A spreadsheet requires the data in a form it can understand. A common input form to all spreadsheets is the 'Comma Separated Variable', in short .CSV.

To get the CAL3K output in to your PC (in a .csv form) the following steps are required:

1. Download a free RS232 service app such as TERATERM or any other Virtual Terminal App
2. Connect the CAL3K to your PC's RS232 port
3. Start the App and configure it to the same speed as the CAL3K port
4. If you have no results in memory then simulate a result:

77 SIMULATE RESULT 1

5. Type the print command on the CAL3K keyboard:

57 PRT TODAY FORM: 1



6. A line of characters should appear on the VT terminal screen
7. This line must be saved in a file: name.csv. This can be done by transferring the data to the Notepad.
8. Select the data, then open Notepad and past the data
9. Save the pasted data in Notepad to a file: NAME.CSV
10. Open your spreadsheet with NAME.CSV and the data appear!

There are many ways to achieve this but the principal is the same:

- A) You need a RS232 receiving App
- B) The received data must be stored in a NAME.CSV file in your PC
- C) Open your spreadsheet with NAME (the spreadsheet will recognise .CSV)

The above example is just a teaser. Try the dump:

```
79 DUMP RESULT DATA 1
```

The dump data are preceded by a header to explain what the columns are.

22 PRINTING REAL TIME

These commands apply to the **CAL3K-AP/A/F only**. 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)

23 PRINT RESULT DUMPS

The following outputs are not printable because the record length exceeds the paper width. These outputs are good for saving to a spreadsheet for analysis.

79 DUMP RESULT DATA 1

This command results in an un-printable output. All the result data are dumped to the port preceded by a header. This is useful if a PC is connected to the port with a port monitor such as 'Tera Term VT' or any other port monitor. The data are dumped in a 'CSV' format and it is easy to transfer the data from the port monitor to a file for further processing or storage.

The data are preceded by a header which requires an interpreter to understand. If you struggle to master this, please email us for assistance.

23.1 PRINT CALIBRATION DUMP

84 DUMP CALIBR. S/N 1

Enter the vessel serial number and all (max=15) calibrations are output.

This command results an un-printable output. All stored calibrations for the specified vessel serial number (S/N) are dumped to the port preceded by a header in a CSV format. This command has no real value except it shows the calibrations data for checking the 'AI' performance. The data are preceded by a header. It may not make much sense to you but it provides vital information to us.

24 REAL TIME PRINTING

This section is applicable to **CAL3K-AP/A/F only**.

The 'Real Time' (RT) operation refers to the printing of a protocol of the sample determination as it happens. This output is used by universities where the data are required in the raw form and the computations are performed outside the CAL3K.

The output data are therefore devoid of any computation, adjustments or compensation. In addition, the output protocol can be preceded by a:

USER HEADER: Name of institution, faculty, division

PARAMETER HEADER: The list of parameters under which this experiment was performed



The actual data output starts 30 seconds before firing in the initial period and ends after the main period. A single line of data are printed every 6 seconds as the data are gathered (in Real Time).

Four data output formats are available: WXYZ.

The RT output is enabled by:

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.

53 PRT RTIME HEADER 0

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.

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.

If the RT printout is enabled it may be advisable to disable the normal result print.

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. Enter 'NO' to disable the result print.

25 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 month depending on usage.

25.1 CLEAR RESULT MEMORY

39 CLEAR RESULTS 1

This command clears all results. Depending on the unit the CAL3K keeps from 300 to 700 results. Enter 'YES' to clear the result memory.



25.2 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 particularly 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'.

25.3 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.

25.4 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

25.5 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.

25.6 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.

26 READING

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

49 READ AMB TEMP. 0

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

78 READ ATC TEMP. 0

Only applicable to **CAL3K-AP**.

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

106 READ BOMB CALI 1

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

47 READ CAL3K MAX 1

The CAL3K maximum data are displayed.

45 READ CARD ID 0

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

34 READ CHA. FIRINGS 1

The number of firings in this chassis are displayed.

44 READ DRIFT 1

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

51 READ FIRING VOLT 1

The actual firing voltage is displayed until ESC is pressed.



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.

46 READ MAX VESSEL 0

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

89 READ PRESSURE 0

The actual vessel pressure is displayed until ESC. This applies to the CAL3K_AP only.

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.

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.

65 READ RESULT DAY: 0

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

36 READ RISE VESSEL 0

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

68 READ STABILITY 1

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

43 READ TEMP VESSEL 1

The vessel temperature is displayed until ESC.

83 READ VESS. FIRING 1

The number of firings of the inserted vessel are displayed.



48 READ VESSEL INFO 0

The information of the inserted vessel is displayed.

27 BLUETOOTH

The BLUETOOTH operation requires optional hardware. These 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. If more than one BLUETOOTH connection is used then it requires a name in order to distinguish between them.

72 BLUETOOTH NAME 1

This entry is important if you are using a BLUETOOTH bridge to connect from the CAL3K RS232 port to a PC USB port, and if you have more than one BLUETOOTH device. The name identifies the link to your PC.

The BLUETOOTH link can't be used for a connection to a computer because a virtual driver is loaded on to your PC.

28 SIMULATIONS

Simulations are a nice tool for testing performance, printing, storage and display. This is especially true when connecting to an external device such as a computer or printer. The simulated data is useless, but this does not matter because the data flow, presentation, capture and output are important.

28.1 SIMULATING CV PROCESS

The CAL3K operates normal, but 6 times faster. It requires no oxygen (**CAL3K-AP: No pressure**), no sample, no mass entry, but a firing wire. It will produce a real time (RT) output if enabled and available and print a result line if enabled (CMD=86). A vessel must be in the well.

20 SIMULATION 0

This is a service command. It allows checking out the complete calorific determination but 6 times faster without oxygen or balance. See: DEMO (CMD=108) for the slowed down version.

Note: The simulated result is entered in to memory, but is suitable flagged.



28.2 SIMULATING CALIBRATION PROCESS

Enable the simulation (CMD=20) and calibration and calibration (CMD=19)

NOTE: A calibration record is entered in the calibration memory of this bomb (and mode). The calibration memory must be cleared before proper use.

28.3 SIMULATING A RESULT RECORD

To test the printing and other keyboard commands it may be required to put some results in to the memory. The data in the simulated result are junk and are easy to recognise. They are also flagged. This command puts a result record in to memory. If the printing is enabled then the simulated result is printed.

77 SIMULATE RESULT 1

This command simulates a result (junk data). If the print output is enabled (CMD=63, SET OUTFORM A-H) and a printer is connected then the junk data appear on the output.

All results in memory can be cleared with:

39 CLEAR RESULTS 1

This command clears all results. Depending on the unit the CAL3K keeps from 300 to 700 results. Type 'YES' to clear the result memory.

28.4 SIMULATING A CALIBRATION RECORD

This command puts a simulated calibration record into the specified vessel serial number. This command doesn't PERFORM a calibration, it just puts a calibration record in to memory. The mode is active! The data is useless, but printable.

127 SIM CALI BOMB SN 1

Enter a 4-digit (or less) serial number and a calibration record is entered in to the specified memory (and mode). This calibration is useless together with valid calibrations and must be cleared before use. The vessel doesn't have to be inserted, but the serial number must be entered.

The calibration entry can be displayed with:

106 READ BOMB CALI 1

The bomb calibration field (all entries) are cleared with:

42 CLEAR B_CAL S/N 1



The bomb calibration can be printed with:

111 PRT E_CALIBR. S/N 1

28.5 SIMULATING A HELP EVENT

This command simulates a help event entry.

143 SIM HELP EVENT # 0

It enters a message number (#) in to the stored help memory. Other commands:

CMD=112 PRINT HELP HISTORY

F11 Read last help history, Enter=more, F11=next

The complete help event memory can be cleared with the:

39 CLEAR RESULTS 1

Enter the letters 'HELP' instead of 'YES'.

28.6 DEMO

The Demonstration (DEMO) feature prepares the unit for demonstration to a customer. This demo mode requires:

A vessel with firing wire attached

Power supply for CAL3K

A calibration: If not available then perform a DEMO CALIBRATION (see below)

It does NOT require:

Oxygen to fill the vessel

A filling station

A cooler

For details see: SIMULATING CV PROCESS

The demo process works at the same speed as an ACTUAL determination. In other words it is a simulation at the normal speed. A mass must be entered electronically or manual.

108 DEMO 1

This command is for demonstration only. It allows to demonstrate the complete operating cycle without:

Oxygen pressure

Sample insertion

Mass entry

It is possible to perform a DEMO CALIBRATION.

If a demo calibration needs to be done then set calibration active:

**19 CALIBRATION 0**

A DEMO calibration results in a calibration record, which is added to the vessel (and mode) calibration memory.

NOTE: Before this vessel can be used for real operations the calibration memory must be cleared!

The demo results may vary significantly depending of what calibrations are in memory. The calibration memory can hold 15 calibrations. The type can be:

- Real calibrations (performed before shipment)
- Simulated calibration (By CMD=20, + CMD=19)
- Demo calibrations (CMD=108)

The unit doesn't differentiate between them, but the data are different and flagged.

28.7 SIMULATING AN OVER-PRESSURE RECORD

This command applies to the **CAL3K-AP only**. It simulates a pressure event record in memory. See 'PRESSURE' section for details.

126 SIM PRESS RECORD 1**29 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/A 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.



30 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 in to 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 ' is not selected.

3 *GROUP IDENTIFIER*

The Group Identification(GID) is an 8-16 character 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 SO₂. 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 contents. 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 in to 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 to operate the unit on the default parameters unless a change is absolutely 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

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.

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 MANUAL 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.

20 SIMULATION 0

This is a service command. It allows checking out the complete calorific determination but 6 times faster without oxygen or balance. See: DEMO (CMD=108) for the slowed down version.

Note: The simulated result is entered in to memory, but is suitable flagged.

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.

We know that the KBTU/lb is not conventional, but they fit nicely in to 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 AVERAGE ACCEPT. 1

The entered value should follow the following grading guide:

| Value | Means |
|-------|----------------|
| 0.012 | Excellent |
| 0.024 | Good |
| 0.048 | Acceptable |
| 0.096 | Poor |
| 0.192 | Bad! Calibrate |

A 'ZERO' entry (Default) disables the acceptance check.

27 DELETE CAL ABOVE 1

The is a test command. It deletes calibrations above the set limit.

28 GELATINE CAPSULE 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

32 AVERAGE CALIBR. 1

Do not use this command!

The average calibration makes sense if you have more than 4 calibrations stored, because the AI (Artificial Intelligence) selection process needs to check that the calibrations are acceptable. It averages the bomb factors (heat capacity) for the best fitting calibrations.

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.

35 TEMP SENSOR CALBR 2

This command is no longer needed.

36 READ RISE VESSEL 0

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

37 READ VS. TOT. FIR. 1

The total vessel firings are displayed. The vessel must be in the well.

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 is 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.

66 COOLER OVERRIDE 2

The command applies to the CAL3k-S and ST only.

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 affect 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.

72 BLUETOOTH NAME 1

This entry is important if you are using a BLUETOOTH bridge and if you have more than one BLUETOOTH device. The name identifies the link to your PC.

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.

74 COOL READY TEMP 2

The air cooler can be programmed with the ready temperature. The default is 32C.

75 COOL AMBIENT ADD 2

The air cooler ambient Addition temperature can be set. The default value is 9C.

76 COOL MAX TEMP 2

The air cooler maximum temperature is set. The default setting is 42C.

77 SIMULATE RESULT 1

This command simulates a result (junk data). If the print output is enabled (CMD=63, SET OUTFORM A-H) and a printer is connected then the junk data appear on the output.

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 a 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.

80 INITIAL TIME 1

The initial time can be set in the CAL3K-A/AP in the experimental mode.

81 READ LAST N_RISE 1

The last nett rise is displayed after the main period.

**82 SET OPS MODE 1**

The CAL3K 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 DUMP CALIBR. S/N: 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 before a determination.

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.

**98 PRESS FILL LOSS 1**

This command applies to the **CAL3KAP**. The pressure filling loss is measured from just before firing to the end of the main period. It should be less than 2bar. If it is more than the vessel leaked when the pressure peaked after firing to approx. 60bar. The filling loss checking is disabled by entering ZERO. The default is ZERO.

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.

100 PRT CALCUL. DATA 1

After a determination a complete data set is available for printing. These data are important for the engineer and have no use for the customer.

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 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.

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

108 DEMO 1

This command is for demonstration only. It allows to demonstrate the complete operating cycle without:

Oxygen pressure
Sample insertion
Mass entry

It is possible to perform a DEMO CALIBRATION.

If a demo calibration needs to be done then set calibration active:

109 BLOCK CALIBRAT. 2

The specified calibration 1-15 is blocked. A blocked calibration is used by the next calibration.

110 UNBLOCK CALI S/N 2

The first calibration which is blocked in the specified vessel is un-blocked.

111 PRT E_CALIBRA. SN 1

Enter the vessel serial number and all available calibrations are printed. The vessel must be out of the well.

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
Short display text

113 COMPENSATE BOMB 2

The calibrations of the specified bomb are compensated. The operation is automatically performed after each calibration.

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.

116 BLOCK CALI LIMIT

This command sets the block limit. The default value is 0.15KJ/g

117 BLOCK CALI ABOVE

All calibration which are above the block limit are blocked.

118 Spare**119 Spare****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.

126 SIM PRESS RECORD 1

This command applies to the **CAL3K-AP only**. It simulates a pressure event record in memory.

127 SIM CALI BOMB SN 1

Enter a 4-digit (or less) serial number and a calibration record is entered in to the specified memory (and mode). This calibration is useless together with valid calibrations and must be cleared before use. The vessel doesn't have to be inserted, but the serial number must be entered.

**143 SIM HELP EVENT # 0**

It enters a message number (#) in to the stored help memory. Other commands:

CMD=112 PRINT HELP HISTORY
F11 Read last help history, Enter=more, F11=next

The complete help event memory can be cleared with the:

147 DUMP RSLT VESSEL 1

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

31 TEMPERATURE RISE

The vessel is calibrated with 0.5g BA which rises the vessel temperature by approx. 12.5C. 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.

31.1 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 ~2C 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 4 calibrations must be performed with the low mass. If more than one vessel is used then all vessels 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!

31.2 HOW TO MEASURE THE WIRE+COTTON RISE

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

Disable the Miss-fire Limit with:

21 MISSFIRE LIMIT 1

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.

31.3 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 Miss-fire Limit with:

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 by:

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



32 MAINTENANCE

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

32.1 DAILY USE

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

32.2 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.

32.3 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.

32.4 MAINTENANCE SCHEDULE (GUIDELINE)

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

The following Vessel parts require maintenance:

- Large Lid O-ring (3K-4-016)
- Centre electrode (3K-4-036)
- Outside electrode (3K-4-037)
- Firing wire (3K-4-032)
- Top & Bottom O-ring (3K-4-022)
- Nozzle O-ring for CAL3k-AP (3K-1-080)
- Nozzle O-ring for CAL3k-AP (3K-4-107) (High quality)
- FAN (Both for Cooler and Calorimeter)
- Crucible (3K-4-047)
- Deflector Plate (3K-4-005)
- 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-4-016):
Worst conditions: 2-5 samples
Average conditions: 40-50 samples
Good conditions: 100+ samples

CENTRE ELECTRODE (3K-4-036): 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 (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-4-005): 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



33 WARRANTY – FOR NEW PRODUCTS

33.1 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.

33.2 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.

33.3 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.



33.4 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.

33.5 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.



33.6 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: _____