



ON-SITE CALIBRATION MANUAL

For CS-APC22M Particle Counter

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TABLE OF CONTENTS

| | |
|---|----|
| Introduction | 3 |
| Brief | 3 |
| Required Equipment & Standards | 3 |
| calibration procedure | 5 |
| Flow Chart | 5 |
| 1. Sensor Preparation for calibration. | 5 |
| 2. Entry of Equipment and Standards Information for the Calibration Record. | 6 |
| 3. Counter Start Level Adjustment | 7 |
| 4. Sensor Noise Determination & Signal to Noise Ratio Verification | 9 |
| 5. System Setup for Measuring Calibration Fluids. | 11 |
| 6. Determination of PSL Mono-Sphere Calibration Settings | 13 |
| 7. Determination of 2806 Calibration Fluid Settings | 19 |
| APPENDIX A | 23 |
| Figure 1 Equipment & Standards Spreadsheet | 6 |
| Figure 2 RM2806 Data Spreadsheet | 7 |
| Figure 3 Sensor Start Level Form- CINRG Software | 8 |
| Figure 4 Sensor Start Level Adjustment Form - CINRG Software | 8 |
| Figure 5 Count Standard Fluids - CINRG Software | 9 |
| Figure 6 CINRG Spiral Stirrer - Installed | 11 |
| Figure 7 Beaker 'up' position | 13 |
| Figure 8 Calibration Fluid Cycling Setup | 15 |
| Figure 9 Data file folder path | 16 |
| Figure 10 PSL 40 Graph - Calibration Spreadsheet | 17 |
| Figure 11 PSL Microsphere Calibration Graph - Spreadsheet | 18 |
| Figure 12 2806 Data Import Spreadsheet | 24 |
| Figure 13 Data import through Legacy Wizard | 25 |
| Figure 14 Data import through Legacy Wizard (Cont.) | 26 |
| Figure 15 Data import through Legacy Wizard (Cont.) | 26 |
| Figure 16 Data import through Legacy Wizard (Cont.) | 27 |
| Table 1 Maximum Allowed Dq Values for calibration data | 21 |

INTRODUCTION

Brief

For the purposes of this calibration procedure, it is assumed the software version running on the particle counter is later than 2023_03_31a and a CINRG accessories Kit containing a spiral stirrer for stirring calibration fluids has been purchased and the modified motor mounting block installed on the particle counter. A magnetic stirrer can be used for mixing the calibration fluids if the CINRG Kit components are not available, but this procedure does not provide any details for the use of a magnetic stirrer. The procedure covers the calibration of the particle counters for particle sizes between 4µm and 100µm in size.

Required Equipment & Standards

1. CINRG accessories kit for running calibration fluids.
 - Two lengths of PTFE delivery tubing approximately 40cm in length and having an outside diameter of 3.2mm (0.125”) and an inside diameter of 2.0mm (0.08”).
 - Two 3/32” stainless steel tube guides.
 - One Acetyl spiral stirrer attachment.
 - Stirrer Motor mounting with holes for tube guides. (Must be installed).
 - Cylindrical screw jack.
 - 10 x 100ml plastic beakers.
 - One 500ml glass beaker for collecting solvent waste when cleaning the spiral stirrer or magnetic follower.
3. Wash bottle with clean Heptane or Hexane to clean the spiral stirrer.
4. EXCEL Calibration Template workbook supplied by CINRG for the importation and processing of calibration data.
5. Microsoft EXCEL program or similar spreadsheet program compatible with the template file format.
6. Super Clean Fluid - 450ml bottle with certification certificate.

7. 2806 Calibration fluid traceable to NIST 2806 - 450ml bottle with certification certificate.
8. A nominal 40µm PSL mono-spheres suspension in super clean fluid. (200ml bottle).
9. A nominal 70µm PSL mono-spheres suspension in super clean fluid (200ml bottle).
10. A nominal 100µm PSL mono-sphere suspension in super clean fluid (200ml bottle).
11. Certification certificates for the aqueous concentrates for the 40µm, 70µm and 100µm PSL mono-sphere standards.
12. Clean, lint free laboratory tissue.

CALIBRATION PROCEDURE

Flow Chart

Procedure steps

Step 1:
Sensor Preparation for Calibration

Step 2:
Entry of Equipment and Standards information for the calibration Record

Step 3:
Counter Start level Adjustment

Step 4:
Sensor Noise Determination and Signal-Noise Ratio Verification

Step 5:
System Setup for measuring calibration fluids

Step 6:
Determination of PSL Mono-Sphere calibration points

Step 7:
Determination of calibration points using Conostan 2806 Calibration Fluid

1. Sensor Preparation for calibration.

- 1.1 Remove the waste line from the sensor outlet and the delivery tube to the left-hand port of the valve on the sample syringe. Replace them with the 40cm lengths of delivery tube provided with the instrument or in the CINRG accessories kit.
- 1.2 The laser sensor should be cleaned with Citrajet solution prior to calibration to ensure that the windows of the sample cell are clean. To clean the sensor, place the open ends of both the delivery tubes into a small beaker containing a warm dilute solution of Citrajet (50% V/V). Use the syringe control features available in the testing panel to program the sample syringe to aspirate and dispense the warm Citrajet solution back and forth across the sample cell in the sensor for several minutes. As an alternative a manual syringe can be connected to the delivery tube connected to the sensor inlet to implement the same cleaning process without the use of any

software. After the initial cleaning action, the Citrajel solution must be left in the sensor cell for 15 minutes before it is flushed out with warm water. After flushing with water, the cell should be flushed with Isopropanol to remove the water and finally with either heptane or hexane depending on availability.

2. Entry of Equipment and Standards Information for the Calibration Record.

2.1 All the relevant data for the instrument hardware and the calibration standards should be entered into the first spreadsheet (EQUIP&STDS) in the Calibration template workbook. The cells with the yellow background are to indicate where data input is required. Input data must be entered accurately as it is linked to the summary spreadsheet which is an important part of the calibration record. The summary spreadsheet is printed and signed once the calibration is complete to provide a hardcopy of the calibration certificate for the instrument.

| A | B | C | D |
|---------------------------------|-------------------|---|--|
| Equipment Information | | | |
| | Calibration Date | : | 26-01-2023 |
| | Service Engineer | : | |
| | Certificate ## | : | |
| | | : | |
| | Laser Sensor | : | Klotz LDS 45/50 |
| | Serial # | : | 11613 |
| | Flow Rate | : | 30ml/min |
| | | : | |
| | Counter | : | Klotz USB 4096 Channel |
| | Serial # | : | 673 |
| | | : | |
| | Syringe Pump | : | Gilson 4000 series. |
| | Serial # | : | RF40869 |
| Calibration Standard and Fluids | | | |
| | | : | |
| Std 1 | Standard | : | Conostan PartiStan 2806 secondary Standard |
| | Lot Number | : | D27 |
| | Expiry date | : | Sept, 2024 |
| | In certificate as | : | Conostan PartiStan 2806 secondary Standard Lot D27 - Expiry Sept, 2024 |
| | | : | |
| Std 2 | Description | : | Conostan Partistan Super Clean Fluid. |
| | Lot Number | : | 128 |
| | Expiry date | : | July, 2024 |
| | In certificate as | : | Conostan Partistan Super Clean Fluid. Lot 128 - Expiry July, 2024 |
| | | : | |
| Std 3 | Standard | : | Duke Standard 4240A |
| | Lot Number | : | 259664 |
| | Expiry date | : | Oct, 2025 |
| | Particle Size | : | 39.33µm ± 0.35µm |
| | Coef of Variation | : | 1.1% |
| EQUIP&STDS | | | |
| | | | STD PLOTS Noise RM2806_DATA Calibration Data |

Figure 1 Equipment & Standards Spreadsheet

2.2 The count data from the certificate provided with the 2806 secondary calibration fluid must be entered into the input cells with the yellow background in the spreadsheet label "RM2806 DATA" in the workbook.

| AA6 | | =INDEX(\$S\$2:\$S\$4097,MATCH(MIN(ABS(T\$2:T\$4097-Z6)),ABS(T\$2:T\$4097-Z6),0)) | | | | | | | | | | | | | | | | | | | | | | | | | | | |
|-----|-------|--|-------|-------|-------|-------|--------|--------|--------|--------|--------|---------|----------|--------|--------|------|------|------|----|---------|---|------|----|---|---|---|----|----|----|
| # | Chan# | Run 1 | Run 2 | Run 3 | Run 4 | Run 5 | G | H | I | J | K | L | M | N | O | P | Q | R | S | T | U | V | W | X | Y | Z | AA | AB | AC |
| 1 | 0 | 0 | 0 | 0 | 0 | 0 | 411530 | 410671 | 410295 | 411810 | 411142 | 2055448 | 411089.6 | 410295 | 411810 | 0.37 | 0.15 | 2.4 | 1 | 20554.5 | | 1.2 | mV | | | | | | |
| 2 | 1 | 0 | 0 | 0 | 0 | 0 | 411530 | 410671 | 410295 | 411810 | 411142 | 2055448 | 411089.6 | 410295 | 411810 | 0.37 | 0.15 | 4.9 | 2 | 20554.5 | | 3.7 | mV | | | | | | |
| 3 | 2 | 0 | 0 | 0 | 0 | 0 | 411530 | 410671 | 410295 | 411810 | 411142 | 2055448 | 411089.6 | 410295 | 411810 | 0.37 | 0.15 | 7.3 | 3 | 20554.5 | | 6.1 | mV | | | | | | |
| 4 | 3 | 0 | 0 | 0 | 0 | 0 | 411530 | 410671 | 410295 | 411810 | 411142 | 2055448 | 411089.6 | 410295 | 411810 | 0.37 | 0.15 | 9.8 | 4 | 20554.5 | | 8.5 | mV | | | | | | |
| 5 | 4 | 0 | 0 | 0 | 0 | 0 | 411530 | 410671 | 410295 | 411810 | 411142 | 2055448 | 411089.6 | 410295 | 411810 | 0.37 | 0.15 | 12.2 | 5 | 20554.5 | | 11.0 | mV | | | | | | |
| 6 | 5 | 0 | 0 | 0 | 0 | 0 | 411530 | 410671 | 410295 | 411810 | 411142 | 2055448 | 411089.6 | 410295 | 411810 | 0.37 | 0.15 | 14.6 | 6 | 20554.5 | | 13.4 | mV | | | | | | |
| 7 | 6 | 0 | 0 | 0 | 0 | 0 | 411530 | 410671 | 410295 | 411810 | 411142 | 2055448 | 411089.6 | 410295 | 411810 | 0.37 | 0.15 | 17.1 | 7 | 20554.5 | | 15.9 | mV | | | | | | |
| 8 | 7 | 0 | 0 | 0 | 0 | 0 | 411530 | 410671 | 410295 | 411810 | 411142 | 2055448 | 411089.6 | 410295 | 411810 | 0.37 | 0.15 | 19.5 | 8 | 20554.5 | | 18.3 | mV | | | | | | |
| 9 | 8 | 0 | 0 | 0 | 0 | 0 | 411530 | 410671 | 410295 | 411810 | 411142 | 2055448 | 411089.6 | 410295 | 411810 | 0.37 | 0.15 | 22.0 | 9 | 20554.5 | | 20.8 | mV | | | | | | |
| 10 | 9 | 0 | 0 | 0 | 0 | 0 | 411530 | 410671 | 410295 | 411810 | 411142 | 2055448 | 411089.6 | 410295 | 411810 | 0.37 | 0.15 | 24.4 | 10 | 20554.5 | | 23.2 | mV | | | | | | |
| 11 | 10 | 0 | 0 | 0 | 0 | 0 | 411530 | 410671 | 410295 | 411810 | 411142 | 2055448 | 411089.6 | 410295 | 411810 | 0.37 | 0.15 | 26.9 | 11 | 20554.5 | | 25.6 | mV | | | | | | |
| 12 | 11 | 0 | 0 | 0 | 0 | 0 | 411530 | 410671 | 410295 | 411810 | 411142 | 2055448 | 411089.6 | 410295 | 411810 | 0.37 | 0.15 | 29.3 | 12 | 20554.5 | | 28.1 | mV | | | | | | |
| 13 | 12 | 0 | 0 | 0 | 0 | 0 | 411530 | 410671 | 410295 | 411810 | 411142 | 2055448 | 411089.6 | 410295 | 411810 | 0.37 | 0.15 | 31.7 | 13 | 20554.5 | | 30.5 | mV | | | | | | |
| 14 | 13 | 0 | 0 | 0 | 0 | 0 | 411530 | 410671 | 410295 | 411810 | 411142 | 2055448 | 411089.6 | 410295 | 411810 | 0.37 | 0.15 | 34.2 | 14 | 20554.5 | | 33.0 | mV | | | | | | |
| 15 | 14 | 0 | 0 | 0 | 0 | 0 | 411530 | 410671 | 410295 | 411810 | 411142 | 2055448 | 411089.6 | 410295 | 411810 | 0.37 | 0.15 | 36.6 | 15 | 20554.5 | | 35.4 | mV | | | | | | |
| 16 | 15 | 0 | 0 | 0 | 0 | 0 | 411530 | 410671 | 410295 | 411810 | 411142 | 2055448 | 411089.6 | 410295 | 411810 | 0.37 | 0.15 | 39.1 | 16 | 20554.5 | | 37.8 | mV | | | | | | |
| 17 | 16 | 0 | 0 | 0 | 0 | 0 | 411530 | 410671 | 410295 | 411810 | 411142 | 2055448 | 411089.6 | 410295 | 411810 | 0.37 | 0.15 | 41.5 | 17 | 20554.5 | | 40.3 | mV | | | | | | |
| 18 | 17 | 0 | 0 | 0 | 0 | 0 | 411530 | 410671 | 410295 | 411810 | 411142 | 2055448 | 411089.6 | 410295 | 411810 | 0.37 | 0.15 | 43.9 | 18 | 20554.5 | | 42.7 | mV | | | | | | |
| 19 | 18 | 0 | 0 | 0 | 0 | 0 | 411530 | 410671 | 410295 | 411810 | 411142 | 2055448 | 411089.6 | 410295 | 411810 | 0.37 | 0.15 | 46.4 | 19 | 20554.5 | | 45.2 | mV | | | | | | |
| 20 | 19 | 0 | 0 | 0 | 0 | 0 | 411530 | 410671 | 410295 | 411810 | 411142 | 2055448 | 411089.6 | 410295 | 411810 | 0.37 | 0.15 | 48.8 | 20 | 20554.5 | | 47.6 | mV | | | | | | |
| 21 | 20 | 0 | 0 | 0 | 0 | 0 | 411530 | 410671 | 410295 | 411810 | 411142 | 2055448 | 411089.6 | 410295 | 411810 | 0.37 | 0.15 | 51.3 | 21 | 20554.5 | | 50.0 | mV | | | | | | |
| 22 | 21 | 0 | 0 | 0 | 0 | 0 | 411530 | 410671 | 410295 | 411810 | 411142 | 2055448 | 411089.6 | 410295 | 411810 | 0.37 | 0.15 | 53.7 | 22 | 20554.5 | | 52.5 | mV | | | | | | |
| 23 | 22 | 0 | 0 | 0 | 0 | 0 | 411530 | 410671 | 410295 | 411810 | 411142 | 2055448 | 411089.6 | 410295 | 411810 | 0.37 | 0.15 | 56.2 | 23 | 20554.5 | | 54.9 | mV | | | | | | |
| 24 | 23 | 0 | 0 | 0 | 0 | 0 | 411530 | 410671 | 410295 | 411810 | 411142 | 2055448 | 411089.6 | 410295 | 411810 | 0.37 | 0.15 | 58.6 | 24 | 20554.5 | | 57.4 | mV | | | | | | |
| 25 | 24 | 0 | 0 | 0 | 0 | 0 | 411530 | 410671 | 410295 | 411810 | 411142 | 2055448 | 411089.6 | 410295 | 411810 | 0.37 | 0.15 | 61.0 | 25 | 20554.5 | | 59.8 | mV | | | | | | |
| 26 | 25 | 0 | 0 | 0 | 0 | 0 | 411530 | 410671 | 410295 | 411810 | 411142 | 2055448 | 411089.6 | 410295 | 411810 | 0.37 | 0.15 | 63.5 | 26 | 20554.5 | | 62.3 | mV | | | | | | |
| 27 | 26 | 0 | 0 | 0 | 0 | 0 | 411530 | 410671 | 410295 | 411810 | 411142 | 2055448 | 411089.6 | 410295 | 411810 | 0.37 | 0.15 | 65.9 | 27 | 20554.5 | | 64.7 | mV | | | | | | |
| 28 | 27 | 0 | 0 | 0 | 0 | 0 | 411530 | 410671 | 410295 | 411810 | 411142 | 2055448 | 411089.6 | 410295 | 411810 | 0.37 | 0.15 | 68.4 | 28 | 20554.5 | | 67.1 | mV | | | | | | |
| 29 | 28 | 0 | 0 | 0 | 0 | 0 | 411530 | 410671 | 410295 | 411810 | 411142 | 2055448 | 411089.6 | 410295 | 411810 | 0.37 | 0.15 | 70.8 | 29 | 20554.5 | | 69.6 | mV | | | | | | |
| 30 | 29 | 0 | 0 | 0 | 0 | 0 | 411530 | 410671 | 410295 | 411810 | 411142 | 2055448 | 411089.6 | 410295 | 411810 | 0.37 | 0.15 | 73.2 | 30 | 20554.5 | | 72.0 | mV | | | | | | |
| 31 | 30 | 0 | 0 | 0 | 0 | 0 | 411530 | 410671 | 410295 | 411810 | 411142 | 2055448 | 411089.6 | 410295 | 411810 | 0.37 | 0.15 | 75.7 | 31 | 20554.5 | | 74.5 | mV | | | | | | |
| 32 | 31 | 0 | 0 | 0 | 0 | 0 | 411530 | 410671 | 410295 | 411810 | 411142 | 2055448 | 411089.6 | 410295 | 411810 | 0.37 | 0.15 | 75.7 | 31 | 20554.5 | | 74.5 | mV | | | | | | |

Figure 2 RM2806 Data Spreadsheet

2.3 When the information has been entered and checked for accuracy, use the “Save As” option to save the template as a new file with a suitable name to create what will become the working copy of the calibration workbook and the electronic record of the instrument calibration. This new file will differentiate the working copy of the workbook from the template workbook which must be preserved for use in future calibrations.

3. Counter Start Level Adjustment

- 3.1 The start level of the counter must be set to a level that is below that of the expected noise level of the sensor otherwise it will not be possible to measure the noise threshold of the sensor as defined by the ISO 11171 calibration standard.
- 3.2 Power up the particle counter and system computer. Load the application software and initialize the hardware. Enter the admin password and click on the Edit Parameters button.
- 3.3 In the Systems Parameter Configuration window select the “Sensor Start Level” Tab and then click on the “Alter Sensor Start Level” button.

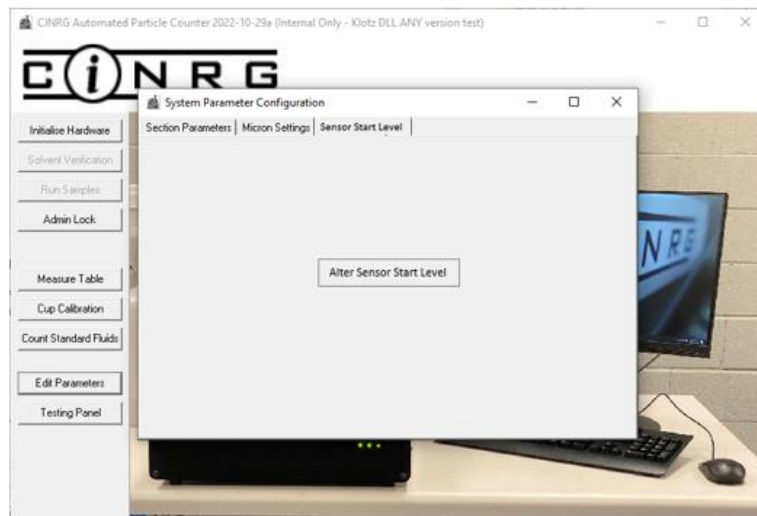


Figure 3 Sensor Start Level Form- CINRG Software

3.4 A window will appear that shows the value of current start level read from the counters NVRAM. Record this value for future reference and then change the value to 150mV and click on the set now button to change the NVRAM parameter to 150mV.

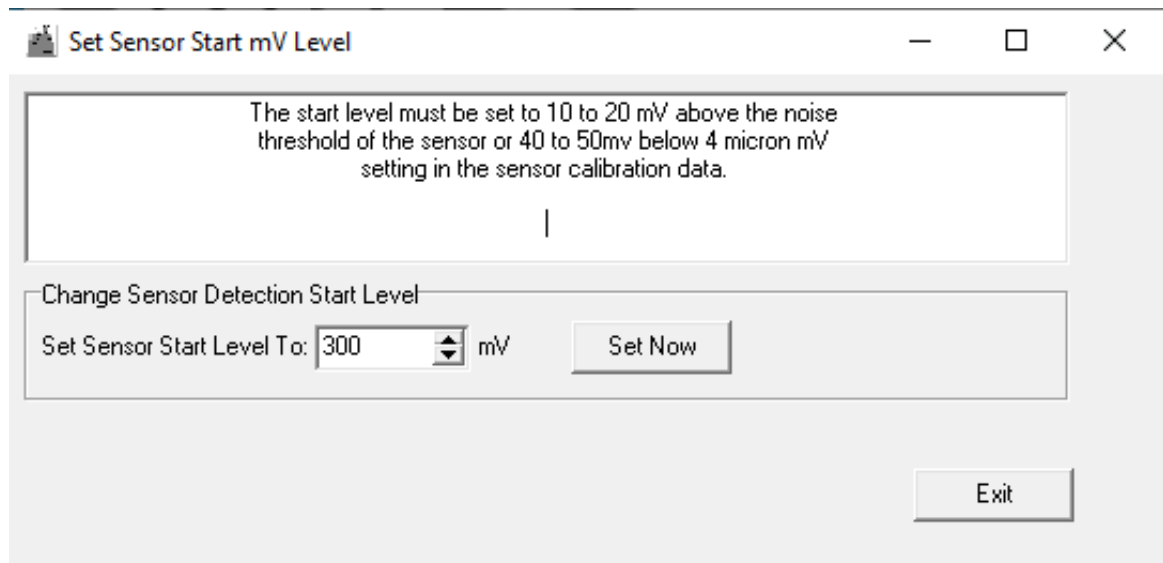


Figure 4 Sensor Start Level Adjustment Form - CINRG Software

3.5 Click on the exit button to exit the “Set Sensor Start mV Level and then close the “System Parameters Configuration window.”

4. Sensor Noise Determination & Signal to Noise Ratio Verification

- 4.1 Click on the “Count Standard Fluids” button to access the “Count Standard Fluids” window.

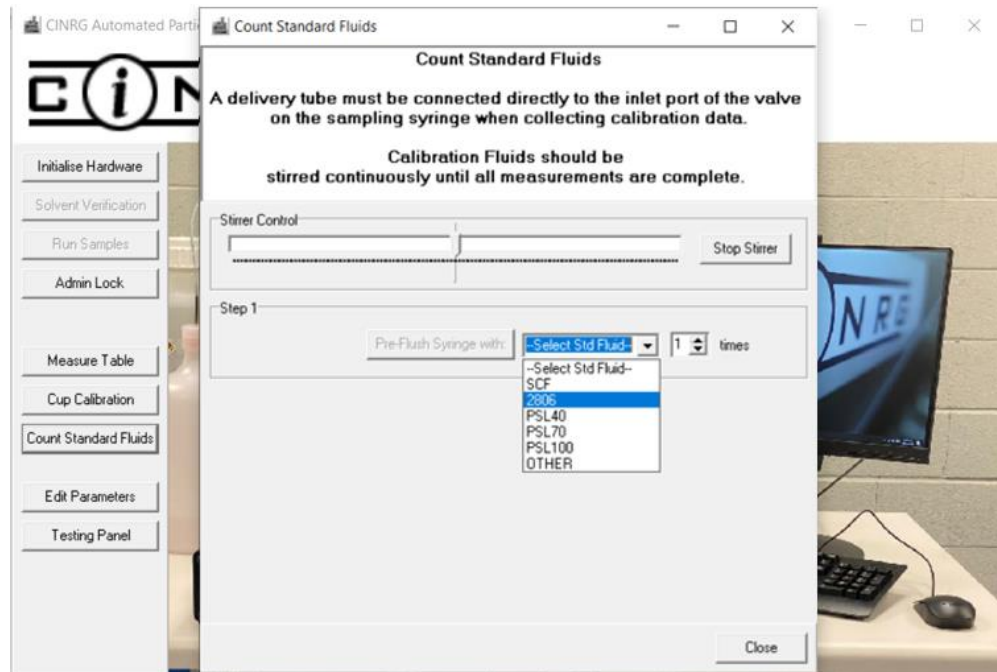


Figure 5 Count Standard Fluids - CINRG Software

- 4.2 Pour about 100ml of super clean fluid into a plastic beaker and place the open end of the delivery tube connected to the inlet port of sample syringe into the fluid in the beaker.
- 4.3 Place the open end of the delivery tube connected to the outlet port of the laser sensor into the waste container.
- 4.4 In the “Step 1” section of the “Count Standard Fluids” window select “SCF” from the dropdown list next to the “Pre-Flush Syringe with” button and enter “5” for the number of times the syringe should be pre-flushed.
- 4.5 Click on the “Pre-Flush Syringe with” button to start the process and make sure the delivery tube remains below the surface of the super clean fluid in the plastic beaker until the final flush is completed to prevent air from entering the fluid delivery system.
- 4.6 On completion of the fifth flush a “Step 2” section will appear in the lower portion of the window and an additional button will appear in the top section labelled “Measure Sensor Noise Threshold”.

- 4.7 Wait for about 15 to 30 seconds and then select the “Measure Sensor Noise Threshold Button”, This will initiate a 60 second measurement of the super clean fluid that is stationary in the sample cell of the laser sensor.
- 4.8 When the measurement is complete the noise threshold channel and its equivalent millivolt value will be displayed adjacent to the button. Record the two values and then repeat the measurement a total of five times.
- 4.9 Each time a noise threshold measurement is completed a data file is stored in a newly created “Sensor Noise” folder. Open this folder and import the five data files from the folder into columns C to G of the “Noise” spreadsheet within the working copy of the calibration workbook. See Appendix A for details on how to import a text file into an EXCEL worksheet.
- 4.10 Look down column A of this spreadsheet after the data has been imported to find the cell with the letters “NT” displayed in it. If you look across this row, columns S and T will contain the average channel number for the noise threshold and the millivolt value that corresponds to it. The mV value is the noise threshold of the sensor as defined by the ISO 11171 calibration standard.
- 4.11 Record the noise channel number in cell M33 of the “PSL40GRAPH” spreadsheet as this value is needed in the moving window or half count method when determining the threshold settings for mono-sphere calibrations.
- 4.12 The millivolt value for the noise threshold must be transferred manually to cell G16 of the “Summary” spreadsheet of the calibration workbook.
- 4.13 Before proceeding with the new calibration determine the signal to noise ratio (SN ratio) by dividing the current $>4\mu\text{m(c)}$ mV setting by the noise threshold value. Ideally this value should be ≥ 1.5 but can be as low as 1.2 and still yield good calibration data.
- 4.14 If the SN ratio is less than 1.2 the calibration procedure should be aborted, and the sensor returned to the manufacturer for repair.
- 4.15 If the SN ratio is satisfactory continue with the calibration procedure but before doing so set the sensor start level to 40mV below the current $>4\mu\text{m(c)}$ mV setting. This is important as the start level inhibits the counting of voltage peaks that occur below the start level and reserves the processing power of the hardware to voltage peaks that are above the start level. The hardware can accurately process voltage peaks up to a maximum of 40 peaks per millisecond. Above this value counts will be underestimated owing to the limitations of the hardware.

- 4.16 Standard fluids are measured in the following order - PSL40, PSL70, PSL 100 and finally the 2806 calibration fluid.

5. System Setup for Measuring Calibration Fluids.

- 5.1 A set of hex wrenches will be needed to install and adjust the system hardware to prepare it to measure calibration fluids.
- 5.2 Remove the sample tray from system if there is one in place and clean the glass surface with some solvent (IPA).
- 5.3 Use a 7/64" hex wrench to loosen the socket head caps screws on either side of the black Delrin motor mounting and then rotate the mounting through a sufficient angle to allow the stirrer motor sit vertically in the mounting.
- 5.4 Remove the aluminum paddle stirrer from the stirrer motor shaft and replace it with the black Delrin spiral stirrer from the CINRG kit. A 0.050" and 1/16" hex wrench will be needed to loosen and tighten set screws that secure the stirrers to the motor shaft.

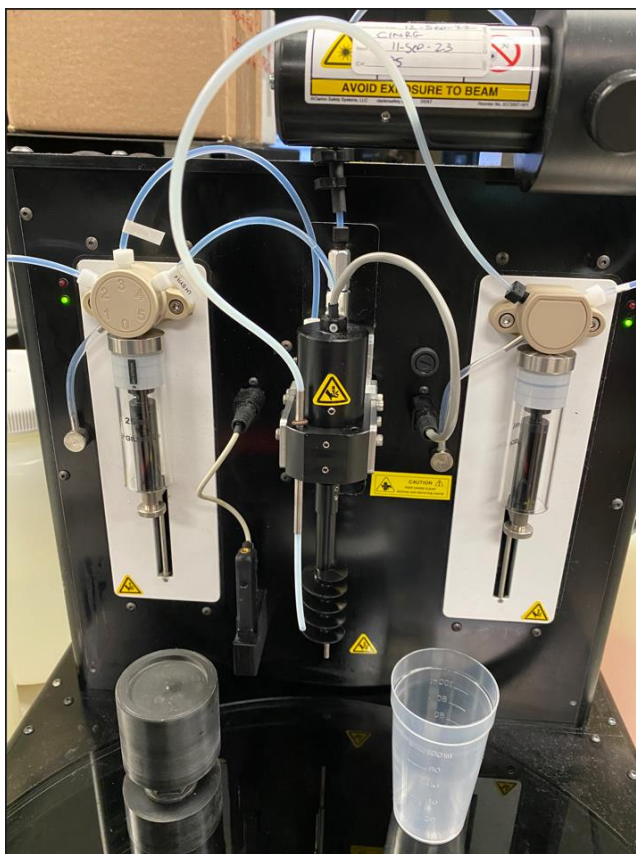


Figure 6 CINRG Spiral Stirrer - Installed

- 5.5 Place the cylindrical screw jack directly under the spiral stirrer and adjust its height until the top surface of the jack is approximately 5mm from the base of the spiral stirrer.
- 5.6 Remove the jack and place an empty beaker directly under the stirrer then use your left hand to raise the beaker up over the spiral stirrer while using your below the right hand to slide the cylindrical screw jack under the beaker and then let the beaker down gently onto the screw jack's top surface.
- 5.7 Check that the stirrer can turn freely without touching the bottom or sides of the plastic beaker. It may be necessary to make some adjustments to the height and/or position of the screw jack to ensure no contact is made.
- 5.8 The position of the stirrer is not changed when running calibration fluids and beakers containing the fluids must be placed directly below the stirrer and then lifted to submerge the stirrer in the fluid. The beaker is then supported in its "up" position by sliding the cylindrical screw jack beneath it.

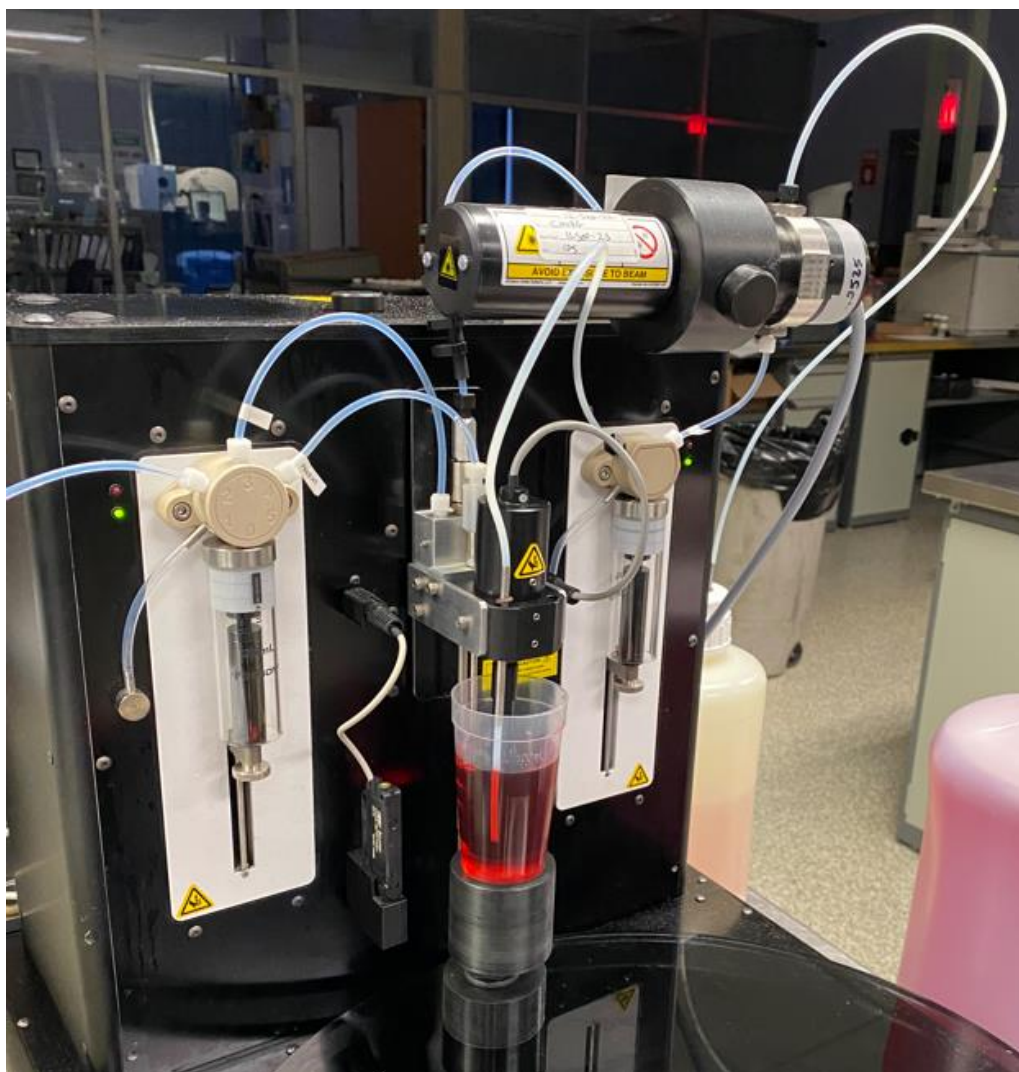


Figure 7 Beaker 'up' position

6. Determination of PSL Mono-Sphere Calibration Settings

- 6.1 Shake the bottle containing the PSL40 fluid standard vigorously by hand for 1 or 2 minutes and then place it in the ultrasonic bath briefly to degas the liquid. The bottle should only be treated in the ultrasonic bath until the bubble layer has diminished in size and travelled upwards to reach the liquid surface.
- 6.2 Remove the cap from the bottle and pour about 100ml of the fluid into a clean plastic beaker. Place the beaker centrally below the spiral stirrer and then raise it upwards and slide the cylindrical lab jack under the beaker to support it.
- 6.3 Adjust the speed of the spiral stirrer to create a small vortex at the liquid surface. For the APC22M the speed of the spiral stirrer is adjusted by clicking on the indicator of the Speed Control slider displayed in the software and dragging it to

the left or right of its central position to adjust the stirring speed in a clockwise or anti-clockwise direction. The APC-2 requires the speed to be set to 1 in the testing panel beforehand so the manual speed control knob on the MCI unit can be used to adjust the stirring speed.

- 6.4 Transfer the delivery tube that connects to the sampling syringe from the super clean fluid beaker to the beaker with the PSL40 fluid standard. The stainless-steel guide tube on the left side of the motor mounting should be used to guide the delivery tubing into the fluid in the beaker.
- 6.5 Position the open end of the delivery tube close to the side of the plastic beaker 2 to 3 cm from the bottom of the container.
- 6.6 Click on the drop-down arrow to the right of the “Pre-Flush Syringe with” button to display the list of standards and select PSL40 from the list. Set the number of pre-flushes to one and then click on the “Pre-Flush Syringe with” button to start flushing the system with PSL40 standard.
- 6.7 When the pre-flush completes, rinse the outside of the waste line with clean solvent and wipe it with a lint free tissue and then use the stainless-steel guide tube on the right of the motor mounting to direct the tubing into the beaker containing the PSL40 standard. The end of the tubing should be positioned about 2cm below the liquid surface.



Figure 8 Calibration Fluid Cycling Setup

- 6.8 In the lower section of the “Count Standard Fluids” window (Step 2), set the sample volume to 20ml and the repeat measurements option to 5 times and then start the measurement sequence.
- 6.9 The syringe will aspirate fluid from the beaker and dispense it through the sensor at a flow rate of 30ml/min a total of five times. The system will collect count data on 20ml of the fluid during each cycle and save the count data in a text file. The name of the txt file indicates what the volume of fluid measures and the date and time of measurement. The data is stored in a folder called “PSL40” and the path to this folder is shown below. The software automatically creates a current date folder whenever standard fluids are measured and then creates various sub-folders within this folder to store the data for the fluids that are measured that day.

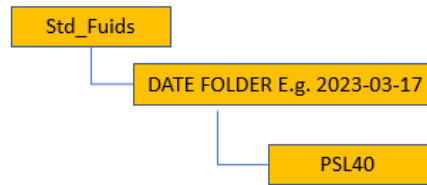


Figure 9 Data file folder path

- 6.10 Import these data files from the PSL40 folder into columns C to G of the “PSL40DATA” spreadsheet within the calibration workbook provided.
- 6.11 Select the PSL40GRAPH spreadsheet and from the Counts vs Channel Number plot, estimate the channel number that corresponds with the mid-point of the peak displayed. Ideally this peak should exhibit a normal distribution (bell shaped curve) but in practice this is seldom the case owing to a variety of sensor artifacts.
- 6.12 Enter this estimate into Cell F29 (40 μ m Threshold Channel) and note the value displayed in cell E39.
- 6.13 Adjust the estimate in cell F29 with small increments or decrements until the value of E39 is minimized. Ideally the value of E39 should be zero but in practice it will have a small positive or negative value as its minimum value. The value of F29 that yields the smallest value for E39 is the threshold setting for the PSL40 standard. This method is called the half count or moving window method.

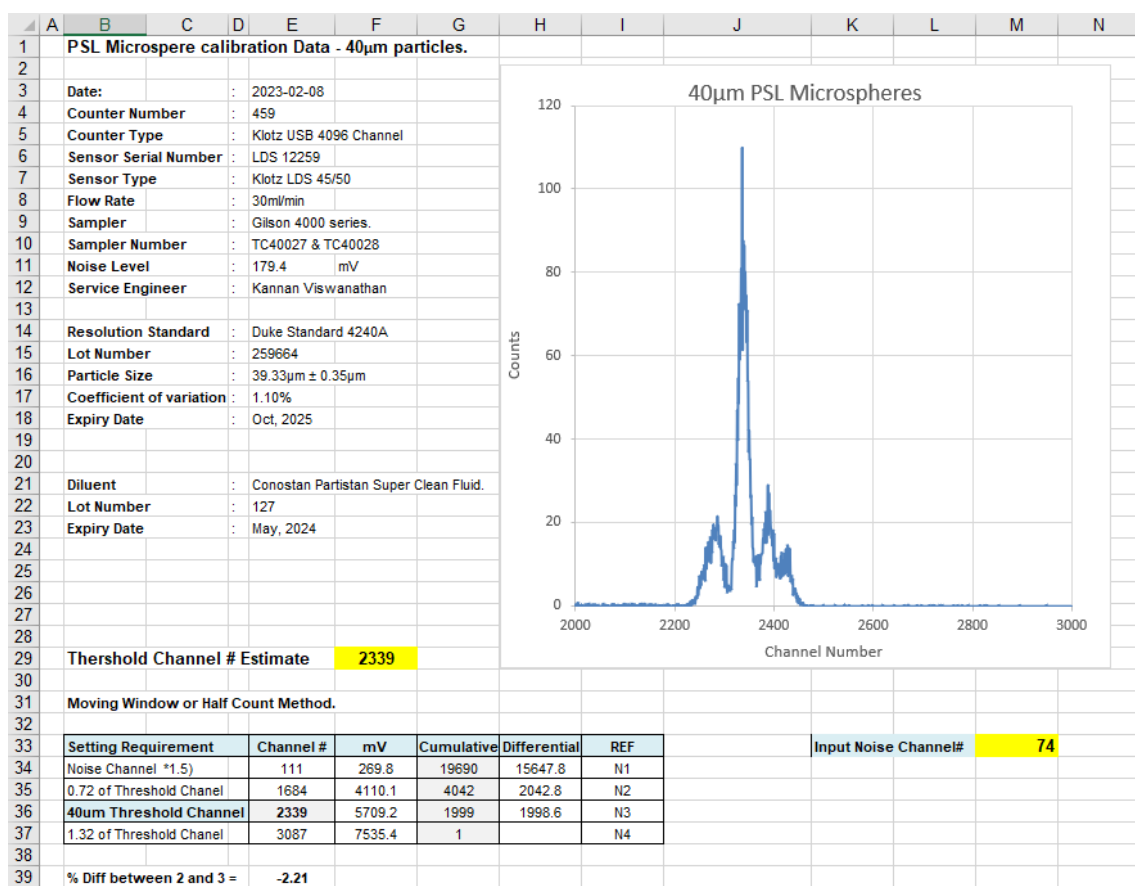


Figure 10 PSL 40 Graph - Calibration Spreadsheet

- 6.14 Turn off or reduce the speed of the stirrer to its slowest speed and then remove the screw jack from below the beaker and lower the beaker to the glass surface. Allow the fluid to drip from the spiral stirrer into the beaker for about half a minute and then remove the plastic beaker and replace it with the 500ml beaker.
- 6.15 Adjust the stirrer speed to a slow to medium speed and then spray the spiral stirrer with a jet of solvent (heptane or hexane) from a laboratory squeeze bottle. Direct the solvent stream up and down the stirrer shaft and spiral blade to flush the red fluid from its surface. Once the red fluid has been completely removed, increase the speed of the stirrer to its maximum to displace and evaporate solvent rapidly from the stirrer.
- 6.16 When the stirrer is dry, turn off or reduce the speed of the stirrer to its minimum value and remove the beaker with the waste solvent and discard the solvent into the appropriate laboratory waste stream.
- 6.17 Measure the PSL70 and PSL100 standard fluids in the same way as the PSL40 fluid but using the spreadsheets in the calibration workbook dedicated to these two standards.

- 6.18 When the three standards have been measured open spreadsheet “38-70-100” and adjust the values in cells B9, B10 and B11 to the actual sizes of the monospheres as indicated on the individual certificates of the three PSL calibration standards.

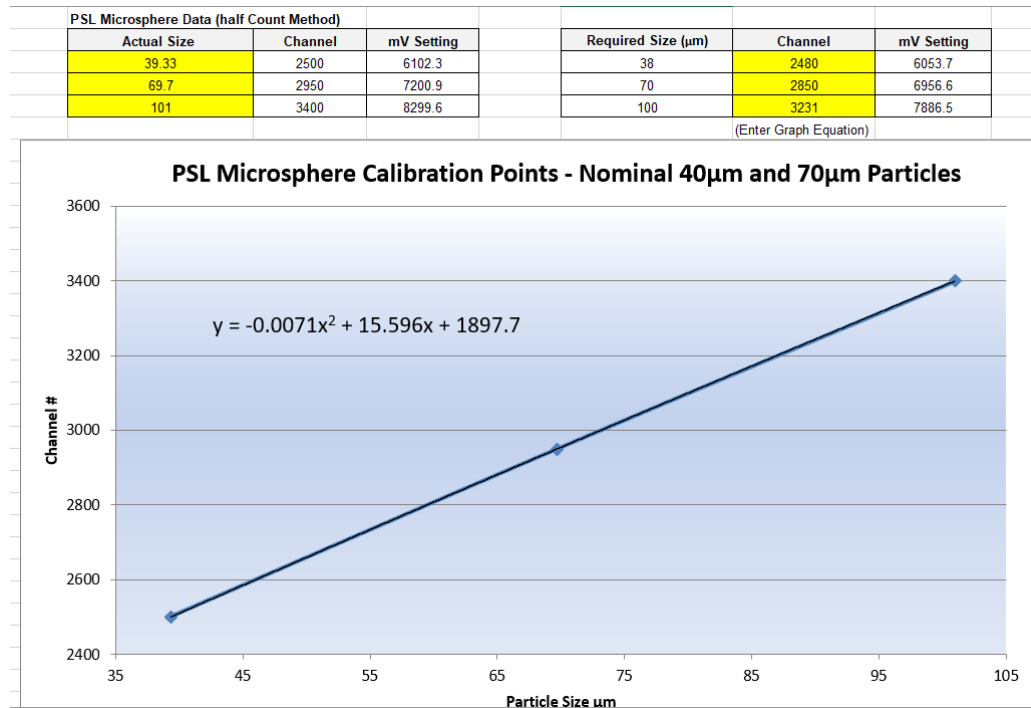


Figure 11 PSL Microsphere Calibration Graph - Spreadsheet

- 6.19 Finally edit the equation in cell G9 so that it is the same as the equation displayed on the chart of Channel # vs. Particle Size μm plot and then copy the equation to cells G10 and G11. When editing the equation pay particular attention to the sign of each term as the first term of the equation is normally negative.
- 6.20 The threshold settings for 38μm, 70μm and 100μm are displayed in cells H9 to H11 and these values are automatically linked to the “Summary” Spreadsheet that can be printed as a calibration record when the entire calibration procedure has been completed.
- 6.21 The 100μm setting is not normally required but 100μm data is collected to allow a PSL curve to be plotted. If only a nominal 40μm and nominal 70μm standard are measured the threshold setting for the actual 38μm and 70μm particle can only be determined from a linear plot between two points and this is obviously less accurate.

7. Determination of 2806 Calibration Fluid Settings

- 7.1 Remove the fluid return line from the right-hand guide tube of the motor mounting and place it directly into the waste container so fluid flowing from the sensor's outlet port flows to waste and is no longer returned to the plastic beaker.
- 7.2 Flush the sensor several times with super clean fluid and then implement one last flush with the delivery tube removed from the super clean fluid to displace most of the fluid in the delivery system with air.
- 7.3 Clean and dry the spiral stirrer as previously described in section 6 and rinse the outside of the last 50mm of the delivery tube that is connected to the sample syringe with clean solvent and wipe it dry with a lint free tissue. This is the section of the delivery tube that protrudes from the end of the stainless-steel guide tube.
- 7.4 Shake the calibration fluid vigorously by hand for at least two minutes and then place it upright in an ultrasonic bath briefly to degas the fluid. The sample will clarify from the base upwards and should remain in the bath only until the clarified layer reaches the surface of the fluid. Repeat this process a second time.
- 7.5 Hold the bottle on its side and roll it for two to three minutes to help breakdown the large bubbles that remain on the liquid surface after removal from the ultrasonic bath.
- 7.6 Immediately after rolling the fluid, pour about 20ml of fluid into a clean plastic beaker and swirl the container to run the fluid up the side of the container and then pour out the fluid to waste while rotating the beaker so the fluid washes over the beaker walls. This is done as a precaution to remove particulate contamination that may have been in the beaker and eliminate its possible influence on the calibration data.
- 7.7 Pour at least 50ml of calibration fluid into the plastic beaker and place the beaker centrally below the spiral stirrer and then raise it upwards and slide the cylindrical lab jack under the beaker to support it.
- 7.8 Adjust the speed of the spiral stirrer to create a small vortex at the liquid surface. For the APC22M the speed of the spiral stirrer is adjusted by clicking on the indicator of the Speed Control slider displayed in the software and dragging it to the left or right of its central position to adjust the stirring speed in a clockwise or anti-clockwise direction. The APC-2 requires the speed to be set to 1 in the testing panel beforehand so the manual speed control knob on the MCI unit can be used to adjust the stirring speed.

- 7.9 Click on the drop-down arrow to the right of the “Pre-Flush Syringe with” button to display the list of standards and select “2806” from the list. Set the number of pre-flushes to five and then click on the “Pre-Flush Syringe with” button to start flushing the system with 2806 standard.
- 7.10 While the sensor is being flushed and during fluid measurements, the bottle of 2806 calibration fluid must be gently rocked and rolled to keep the dust particles suspended and uniformly distributed. This process is only suspended temporarily to top up the fluid level in the plastic beaker.
- 7.11 When the flushing cycles have completed, stop, or reduce the speed of the stirrer, remove the cylindrical screw jack and lower beaker. Discard the bulk of fluid that remains in the plastic beaker and refill it immediately to just above the 100ml mark, Reposition the beaker and cylindrical screw jack to re-submerge the spiral stirrer in the fresh calibration fluid.
- 7.12 Increase the stirrer speed to create a small vortex at the fluid surface and allow the fluid to mix for about three minutes while periodically stopping the stirrer or slowing it down for several seconds to allow any air bubbles to rise to the surface.
- 7.13 Use the “Pre- flush syringe with standard fluid” button to flush the sensor a final time with the calibration fluid.
- 7.14 Set the sample volume to 20ml measurements and the repeat option to 7 times and then start the measurements.
- 7.15 The remaining calibration fluid in the 400ml bottle should be rolled continuously during these measurements and used to top up the fluid in the plastic beaker as soon as the syringe has completed filling and prior to the start of the measurement.
- 7.16 When the last measurement is completed import the data for measurements 2 to 6 into columns B to F (Run 1 to Run5) in the” RM2806B_Data” spreadsheet. This data should be imported starting from the second row of each column as the first row of each column in this spreadsheet has a heading.
- 7.17 In column Z of the “RM2896_Data” spreadsheet, enter the certified count values for the various particle sizes from the certificate for the 2806 calibration fluid.
- 7.18 As each value is entered the channel number for which the cumulative counts are the closest match to the certified count entered will be displayed in column AA.

The channel numbers in column AA are linked (transferred) to the Calibration spreadsheet as well as the Summary spreadsheet.

- 7.19 The table below is used to validate the count data based on the Dq values calculated from the count data for each particle size. The calibration data for each particle size of interest is considered valid if the calculated Dq value is less than or equal to the Max Dq value allowed.

| Mean Number of Particles Counted per ml | | Maximum Dq Allowed |
|---|-----------|--------------------|
| Greater than or Equal to | Less than | |
| 500 | | 11.0 |
| 250 | 500 | 11.3 |
| 100 | 250 | 11.9 |
| 50 | 100 | 13.4 |
| 25 | 50 | 15.6 |
| 10 | 25 | 19.3 |
| 5 | 10 | 27.5 |

Table 1 Maximum Allowed Dq Values for calibration data

- 7.20 The channel numbers transferred to the summary spreadsheet are used to calculate the millivolt settings for each channel as calibration data is entered into the CINRG software as a set of millivolt settings.
- 7.21 Inspect the results of the calibration procedure in the summary spreadsheet and if satisfied with the data, print the spreadsheet to create a hardcopy certificate. The certificate should be signed and retained as a calibration record.
- 7.22 Enter the new calibration parameters into the CINRG software following the procedure described in the user manual for the instrument.
- 7.23 Remove and clean the spiral stirrer and the calibration tubing from the particle counter and re-install the standard stirrer and delivery tubing. The calibration equipment should be stored in a safe place until needed for the next calibration.
- 7.24 Exit the “Count standard Fluids” window in the software and implement a couple of cleaning cycles to flush the system with solvent.

- 7.25 The remaining fresh calibration fluid or a verification fluid can be run as an undiluted sample on the particle counter to verify the newly entered calibration data. A separate verification fluid can also be used for this purpose.

APPENDIX A

IMPORTING TEXT DATA FILES INTO An EXCEL CALIBRATION WORKBOOK WITH EMBEDDED IMPORTATION SCRIPTS (MACRO PROGRAMS).

The workbook with embedded macro programs contains active buttons that facilitate quick access to the data files and the seamless importation of the data file as soon as it is selected. The need to navigate through menu selections and specify the format of the file to be imported is eliminated as this is all handled by the embedded scripts.

In the example show below shows the buttons that are available when importing data file from the measurement of 2806 calibration fluid. Selecting the first button will display the “Open” file window where the end user can navigate to where the 2806 data files are stored and select the first file to be imported. Clicking on the file automatically imports the data to the appropriate column in the spreadsheet.

The selection of subsequent buttons will take the end user to the same folder where the first file was selected and allow the next data file in the sequence to be selected and imported. This efficient process allows the five files to be quickly and accurately imported.

Similar buttons appear in the Noise Level, PSL40 Data, PSL70 Data and PSL100 Data spreadsheets where data files are imported in the same manner.

| 28D | INPUT | CH# | mV | Notes: | | | |
|---------|---------|------|--------|--------|--|--|--|
| 4µm(c) | 11138.6 | 174 | 423.6 | | | | |
| 5µm(c) | 7927.8 | 275 | 670.2 | | | | |
| 6µm(c) | 4865.6 | 471 | 1148.7 | | | | |
| 7µm(c) | 3295.4 | 653 | 1593.0 | | | | |
| 8µm(c) | 2313.2 | 828 | 2020.3 | | | | |
| 9µm(c) | 1616.9 | 1007 | 2457.3 | | | | |
| 10µm(c) | 1125 | 1189 | 2901.6 | | | | |
| 11µm(c) | 806.1 | 1346 | 3284.9 | | | | |
| 12µm(c) | 609.1 | 1460 | 3563.2 | | | | |
| 13µm(c) | 482.3 | 1539 | 3756.1 | | | | |
| 14µm(c) | 389.6 | 1601 | 3907.5 | | | | |
| 15µm(c) | 308.5 | 1661 | 4054.0 | | | | |
| 16µm(c) | 248.8 | 1710 | 4173.6 | | | | |
| 17µm(c) | 207.8 | 1748 | 4266.4 | | | | |
| 18µm(c) | 177.5 | 1778 | 4339.6 | | | | |
| 19µm(c) | 150.9 | 1808 | 4412.8 | | | | |
| 20µm(c) | 125.2 | 1842 | 4495.9 | | | | |
| 21µm(c) | 101 | 1877 | 4581.3 | | | | |
| 22µm(c) | 84.9 | 1905 | 4649.7 | | | | |
| 23µm(c) | 74.3 | 1926 | 4700.9 | | | | |
| 24µm(c) | 64.3 | 1948 | 4754.6 | | | | |
| 25µm(c) | 51.7 | 1980 | 4832.8 | | | | |
| 26µm(c) | 40.2 | 2017 | 4923.1 | | | | |
| 27µm(c) | 31.2 | 2053 | 5011.0 | | | | |
| 28µm(c) | 24.9 | 2084 | 5086.7 | | | | |
| 29µm(c) | 21 | 2108 | 5145.3 | | | | |
| 30µm(c) | 18.6 | 2125 | 5186.8 | | | | |
| | | | | Notes: | | | |

Figure 12 2806 Data Import Spreadsheet

IMPORTING TEXT DATA FILES INTO An EXCEL CALIBRATION WORKBOOK WITHOUT EMBEDDED IMPORTATION SCRIPTS OR WHERE EMBEDDED MACRO

Some IT departments will not allow company personnel to receive, download or open EXCEL workbooks with embedded macros as computer viruses can be disguised as macros within an EXCEL workbook. If an end user is faced with these restrictions a calibration workbook without the embedded macros can be supplied as an alternative option.

To import data into a spreadsheet in a workbook without the use of macro programs the following steps are followed.

1. Select the “Data” option from the menu header to display the various options for importing data.
2. Select the first option (Get Data) from the choices displayed to show a drop-down menu with a subset of data sources.

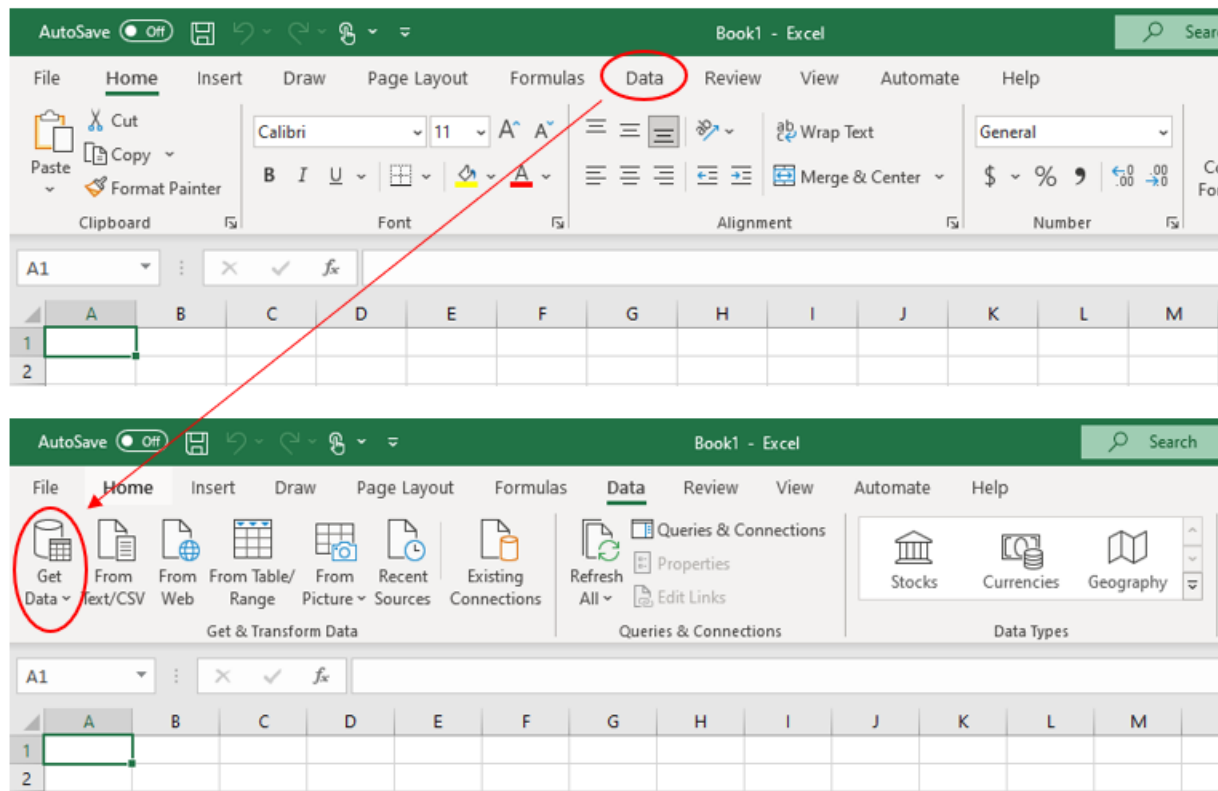


Figure 13 Data import through Legacy Wizard

3. Select the “Legacy Wizards” option from the list and click on “From Text (Legacy)” to display the “open” file window.

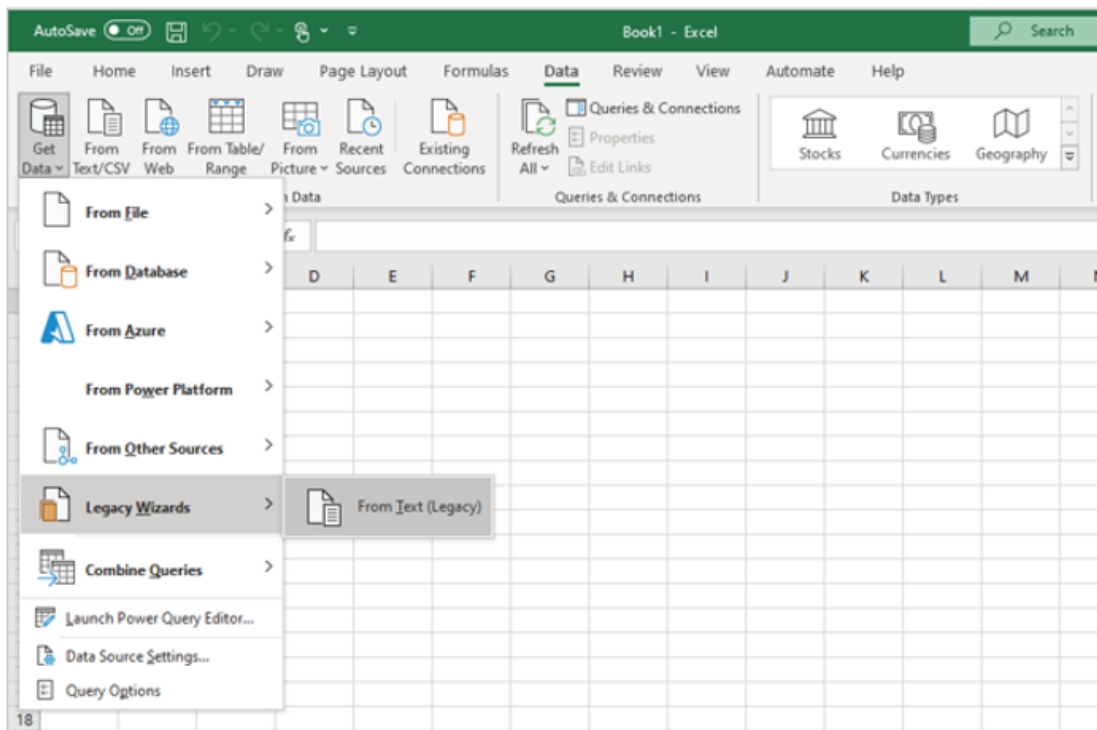


Figure 14 Data import through Legacy Wizard (Cont.,)

4. Navigate to the folder where the data file is stored and double click on the file to be imported.
5. In the "text Import Wizard" window that opens when the file is selected, click on the "Finish" button.

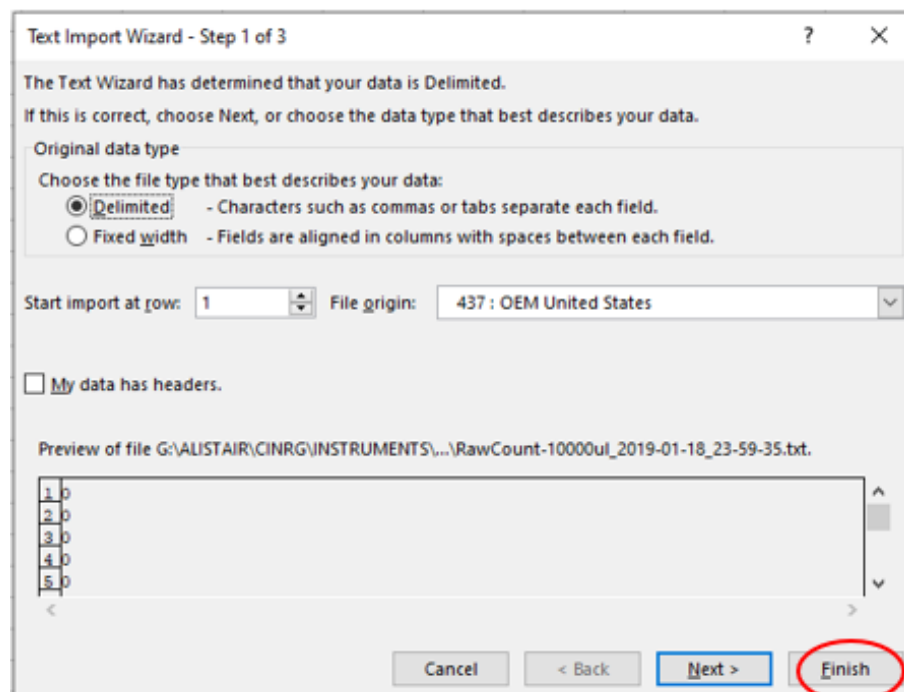


Figure 15 Data import through Legacy Wizard (Cont.,)

6. In the small “import Data” window that opens check that destination cell for the data import is correct and if so, click on the “OK” button.

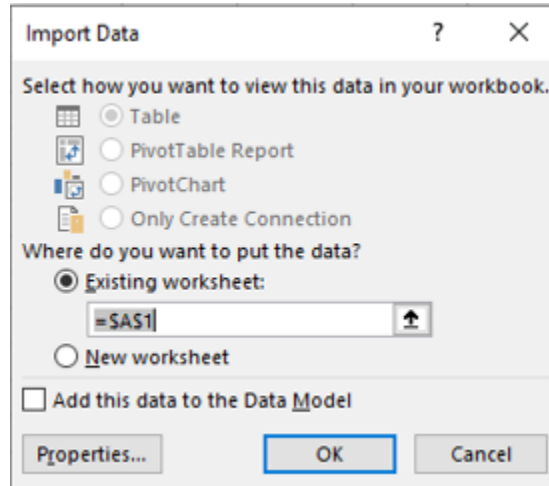


Figure 16 Data import through Legacy Wizard (Cont.,)

7. If the destination cell is incorrect click the mouse in the correct cell in the actual spreadsheet to update the data to the required cell and then click on the ok button.
8. The 4096 data points will be imported into a column starting with destination cell.