Anton Paar PSA 1090 L/D Quick Start Guide

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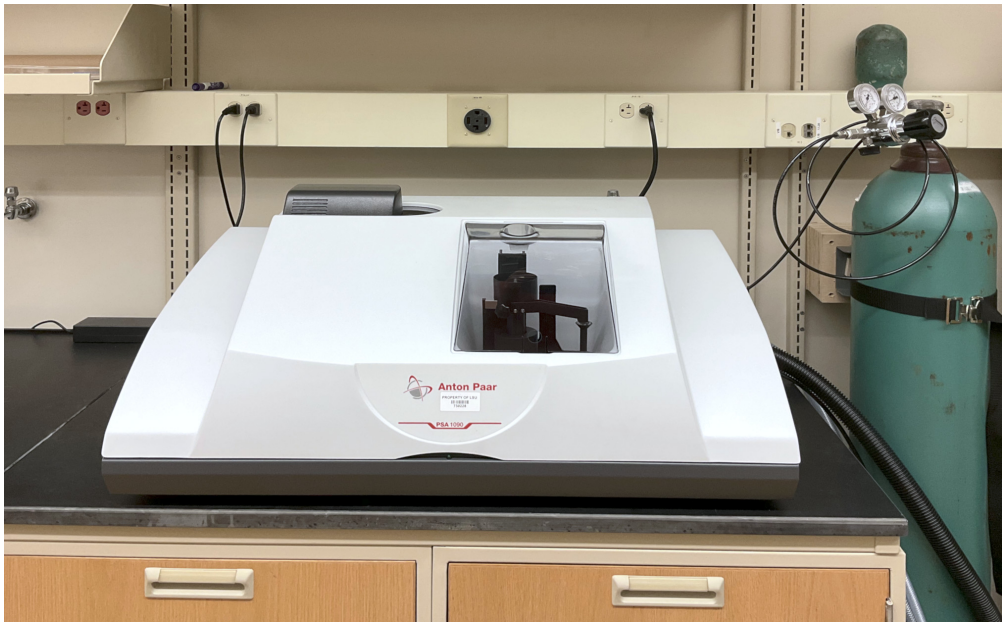

Figure 1: Particle Size Analyzer PSA 1090 L/D setup at the LSU SIF.

The Particle Size Analyzer PSA 1090 L/D instrument is designed for measuring the size of particles ranging from 0.04 µm to 500 µm, either dispersed in liquid or in powder/granulate form, using laser diffraction spectroscopy (LDS).

1 Particle size analysis in dry mode

*In dry mode, **compressed air** is used to transport the sample and generate a jet stream passing through a laser beam. A **vacuum cleaner** with HEPA filter system is used to safely collect the powder sample after the measurement.*

1.1 Before the measurement

1. Switch on the instrument at the left hand side.
2. Start up the PC; pw: `anton paar`
3. Vacuum cleaner system is required and is usually already powered on (switches located at the back of the instrument and on vacuum cleaner itself both in the "ON" position).
4. Open compressed air supply at gas cylinder (Figure 2a). Don't change the pressure at the regulator! Left pressure gauge should indicate 6 bar to 8 bar.
5. Open the **Kalliope** software
6. Click the ≡ Ka icon in the top left to open the menu.
7. Under **My Settings** click **Instrument family** dropdown and select *PSA*.
8. In the submenu "PSA" select **Model:** *PSA 1090*, **Configuration:** *LD*, **Accessories:** *None*.
9. Click the ≡ Ka icon again to close the menu.
10. Make sure the instrument is clean, otherwise clean using compressed air, bottle brush.

1.2 Performing the measurement

1. On the Kalliope start-up screen, click on the ⊕ icon to select a new measurement.
2. Select *Particle size (Dry)* or *Particle size series (Dry)* under "Measurement modes".

   A **series** allows repeated measurements. This can be useful for better statistics, for using different Vibrator frequencies/power, or air pressure.
3. Assign a **Name** to the experiment (click on "Untitled 1", top left)
4. Review and adjust the **Input parameters**:

   - **General:** Name your sample/batch
   - **Freefall/Freefall:** *Freefall mode not available with this model.*
   - **Sample measurement/Measurement time:**
     - For a standard measurement 20 s are recommended.
     - Time can vary a lot to analyze a sufficient number of particles, depending on particle flow and size.
   - **Dispersion parameters/Vibrator duty cycle:** (*Can be re-adjusted later.*)
     - Defines the vibrator "power" for initial dispersion parameters between 0 – 100 %.
     - The feeding rate increases with increasing duty cycle.
   - **Dispersion parameters/Vibrator frequency:** (*Can be re-adjusted later.*)

  - Defines the vibration frequency between 5 Hz to 80 Hz.
  - The maximum feed rate is reached at the resonance frequency (around 43 Hz).
- **Dispersion parameters/Air pressure:** (*Can be re-adjusted later*.)
  - Can be set between 0 mbar to 6000 mbar (default: 500 mbar).
- **Obscuration/Obscuration target:**
  - The obscuration is the percentage of laser light loss through the sample and represents the "optical concentration".
  - A typical obscuration should be 1 % to 10 %. A higher obscuration level leads to multiple scattering events and decreasing apparent particle sizes.
- **Obscuration/Limits:** If obscuration is below minimum, too little sample is being mobilized. Add more sample to the vibratory hopper or adjust the dispersion parameters. If obscuration is above maximum, too much sample is being mobilized. Adjust the dispersion parameters to reduce the powder flow.
- **Cleaning/Clean sample cell:** The instrument will clean the sample cell using compressed air after measurement.
- **Cleaning/Clean sample cell time:** Duration of the cleaning process.
- **Cleaning/Clean venturi:** The instrument will clean the venturi using compressed air after the measurement.
- **Cleaning/Clean venturi time:** Duration of the cleaning process.
- **Analysis/Reconstruction mode:**
  - **Mie:** for small and/or transparent particles.
    Requires prior knowledge of particle material's refractive index and absorbance (at 635 nm / 830 nm, 25 °C). Selection of the particle material in the material's database is compulsory for this option.
    New materials can be entered in the database, using the "Materials" submenu in Kalliope.
  - **Fraunhofer:** use for particles > 50 µm and/or opaque, when the sample contains a mixture of different particles, or when the refractive index is unknown.
- **Analysis/Analysis mode:**
  - **General:** if sample is not well known, or if single (broad) peak is expected
  - **Narrow:** if one or more narrow peaks expected
  - **Custom:** enables access to "Advanced Parameters"
- **Analysis/Material:** Only if **Reconstruction mode** "Mie" was selected. Select sample material from database.
  New materials can be entered in the database, using the "Materials" submenu in Kalliope.
- **Analysis/Specific surface:** When checked, specific surface area will be calculated. Requires **Shape factor** and **Material density** to be filled in.
- **Analysis/Shape factor:** Deviation from perfectly spherical shape (1.0).
- **Analysis/Material density:** Density of material in $\mathrm{g\,cm^{-3}}$
- **User-defined D-values:** D_p of x µm indicates that p % of the particles in the distribution have a size $\leq$ x µm. D_{10}, D_{50} and D_{90} are given by default.

- **User-defined Size Classes:** Define your own size classes to retrieve the percentage of particles contained within defined size limits.

5. Click the **Start** ▷ button.
6. A window will open suggesting to fill the system with water:
   (also for dry measurements, the liquid cell should be filled with clean water)
   - Fill (DI) water into the liquid tank (Figure 2c), either manually by directly adding water to the tank, or by using the connected supply (DI water tap in Figure 2b needs to be open).
   - Open the hood of the PSA
   - In the **Manual** section of the parameter window set "Fill" to **On**.
   - Observe the liquid cell; once no air bubbles are visible set "Fill" to **Off**.
   - Close the hood and press **Continue**. The background measurement will start.
7. A new window for adjusting the dispersion parameters will open.
   - Load your powder sample into the vibratory hopper (Figure 2d).
   - Adjust 3 parameters **vibratory duty cycle, vibrator frequency, and air pressure** to reach a satisfactory obscuration level (must be below 30 %).
   - If all sample is used click **Cancel measurement**, refill sample and repeat procedure.
   - After determining the best parameters, load the vibratory hopper with your sample and click **Continue** to launch the measurement.
8. When finished, the results will be displayed and the data saved in the current workbook.
9. Use the action buttons at the top right to export raw data or recalculate results, if needed.

1.3 Finishing up

1. Save the workbook in your folder and indicate the date in the name of a subfolder.
2. Create report (select a template beginning with *LSU* to include the LSU logo).
3. Close the valve of the compressed gas cylinder (Figure 2a).
4. Close the DI water tap, if used (Figure 2b).
5. Drain the liquid tank of the PSA, if used:
   Click the ≡ Ka icon, select **PSA control** and set "Drain" to **On**. Switch to **Off** when done.
6. Empty the bucket.
7. Close the Kalliope software and shutdown the PC.
8. Switch off the PSA.
9. Clean affected parts of the PSA from any sample residues using compressed air and the small cleaning brush.
10. Put the fabric protective cover back on the instrument.

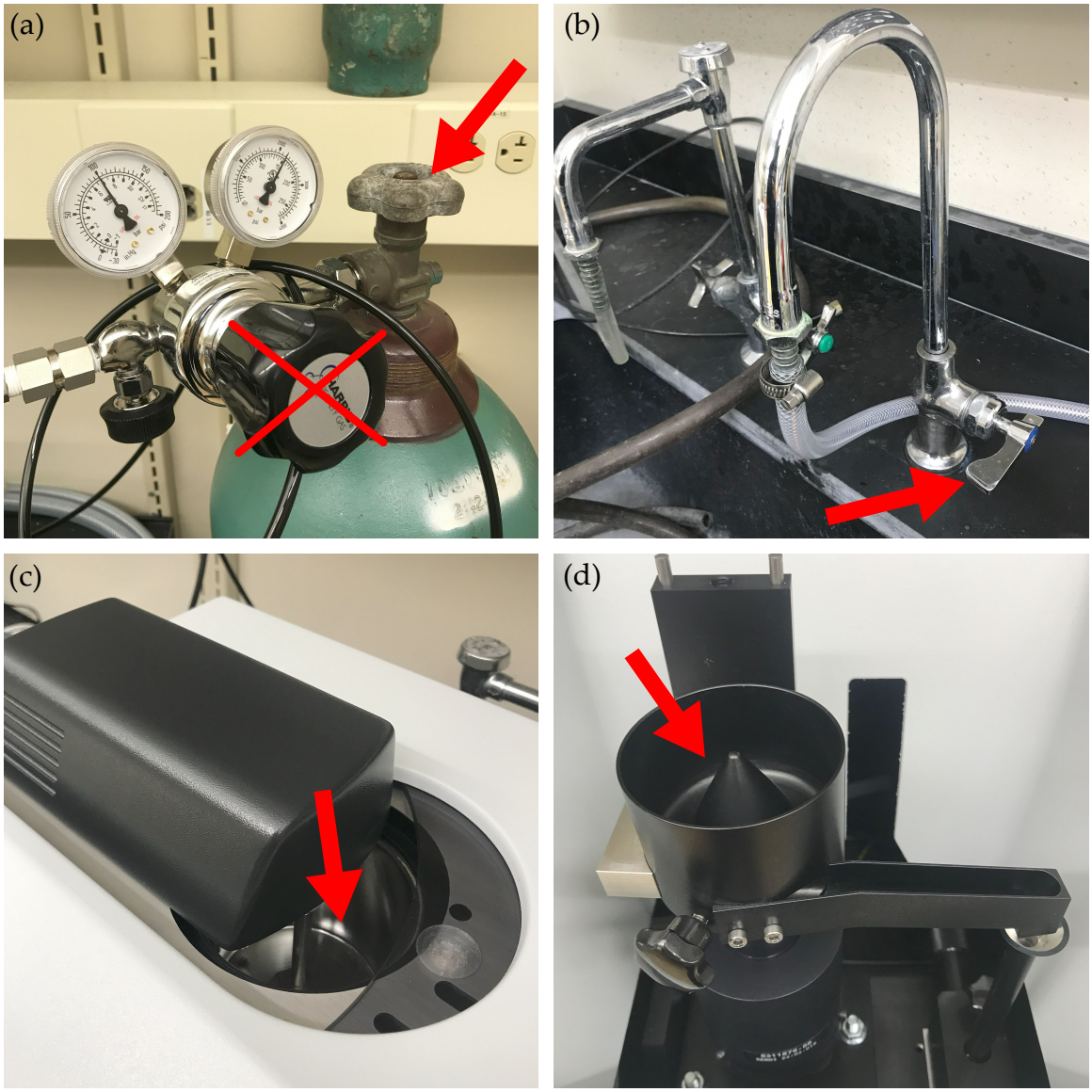

Figure 2: (a) open/close gas cylinder valve, (b) open/close water DI supply, (c) liquid tank for dispersed sample (or just water), and (d) vibratory hopper to fill in dry powder sample.

2 Particle size analysis in liquid mode

In liquid mode, either deionized or purified water is used to disperse the sample in. Peristaltic circulation pumps transport the dispersion through the measurement cell and then drain through a hose into an external bucket.

2.1 Before the measurement

1. Switch on the instrument at the left hand side.
2. Start up the PC; pw: `anton paar`
3. Open DI water tap if needed (Figure 2a).
   Water can be supplied either manually, by filling the liquid tank with clean water, or through the central DI water supply via water tap.
4. Open the **Kalliope** software
5. Click the ≡ Ka icon in the top left to open the menu.
6. Under **My Settings** click **Instrument family** dropdown and select *PSA*.
7. In the submenu "PSA" select **Model:** *PSA 1090*, **Configuration:** *LD*, **Accessories:** *None*.
8. Click the ≡ Ka icon again to close the menu.
9. Make sure instrument is clean, otherwise clean liquid tank and and rinse system.

2.2 Performing the measurement

1. On the Kalliope start-up screen, click on the ⊕ icon to select a new measurement.
2. Select *Particle size (Liquid)* or *Particle size series (Liquid)* under "Measurement modes".

   A **series** allows repeated measurements. This can be useful for better statistics, or for using different pump and stirrer speeds.
3. Assign a **Name** to the experiment (click on "Untitled 1", top left)
4. Review and adjust the **Input parameters**:

   - **General:** Name your sample/batch.
   - **Small Volume Unit:** *leave unticked, as not available at SIF.*
   - **Sample Dispersion/Ultrasound:** Activates the ultrasound generator during the sample dispersion step, which runs automatically before each measurement.
   - **Sample Dispersion/Pump during ultrasound:** Switch pump during ultrasonication.
   - **Sample Dispersion/Ultrasound duration:** Sets the duration of the ultrasound treatment during the sample dispersion step.
   - **Sample Dispersion/Debubbling:** not explained in manual
   - **Sample Dispersion/Debubbling duration:** Sets the duration of the debubbling process during the sample dispersion step.
   - **Sample Dispersion/Equilibriation:** Ensuring the resulting particle dispersion is uniformly distributed in the instrument's tubing and measurement cell.

- **Sample Dispersion/Equilibration duration:** Sets the equilibration time during the sample dispersion step.
- **Sample measurement/Measurement time:** Sets the total sample measurement time.
- **Sample measurement/Ultrasound:** Continuous ultrasound during measurement.
- **Sample measurement/Stirrer speed:** Sets the stirrer speed during the measurement.
- **Sample measurement/Pump speed:** Sets the pump speed during the measurement.
- **Obscuration/Obscuration target:** Defines the minimum and maximum obscuration values recommended to perform a meaningful measurement.
- **Obscuration/Limits:** If obscuration is below minimum, there is too little sample in the tank. If obscuration is above maximum, there is too much sample in the tank.
- **Obscuration/Obscuration limits mandatory:** When the obscuration limits are not between the target values (defined above), the measurement cannot be started.
- **Rinsing/Rinse after measurement:** Run rinsing cycle(s) after the measurement.
- **Rinsing/Rinsing cycles:** Defines number of rinsing cycles.
- **Analysis/Reconstruction mode:**
  - **Mie:** for small and/or transparent particles.
    Requires prior knowledge of particle material's refractive index and absorbance (at 635 nm / 830 nm, 25 °C). Selection of the particle material in the material's database is compulsory for this option.
    New materials can be entered in the database, using the "Materials" submenu in Kalliope.
  - **Fraunhofer:** use for particles > 50 µm and/or opaque, when the sample contains a mixture of different particles, or when the refractive index is unknown.
- **Analysis/Analysis mode:**
  - **General:** if sample is not well known, or if single (broad) peak is expected
  - **Narrow:** if one or more narrow peaks expected
  - **Custom:** enables access to "Advanced Parameters"
- **Analysis/Material:** Only if **Reconstruction mode** "Mie" was selected. Select sample material from database.
  New materials can be entered in the database, using the "Materials" submenu in Kalliope.
- **Analysis/Solvent:** Select the solvent the particles are dispersed in.
  New solvents can be entered in the database, using the "Solvents" submenu in Kalliope.
- **Analysis/Specific surface:** When checked, specific surface area will be calculated. Requires **Shape factor** and **Material density** to be filled in.
- **Analysis/Shape factor:** Deviation from perfectly spherical shape (1.0).
- **Analysis/Material density:** Density of material in $\mathrm{g\,cm^{-3}}$
- **User-defined D-values:** D_p of x µm indicates that p % of the particles in the distribution have a size $\leq$ x µm. D_{10}, D_{50} and D_{90} are given by default.
- **User-defined Size Classes:** Define your own size classes to retrieve the percentage of particles contained within defined size limits.

5. Click the **Start** ▷ button.
6. *When the input parameters have been defined, and before the actual measurement can take place, a filling and equilibration step must be completed to ensure the proper filling of the instrument.* The opened window will provide the required device controls.

   - If central DI water supply is used:
     - Set "Fill" to **On** to fill the system and tubing with water.
     - After approx. 20 s switch "Fill" to **Off** or click **Stop all**.
   - If solvent is delivered manually:
     - Slide tank cover to the side (Figure 2c).
     - Fill tank to capacity (450 mL) with solvent (make sure level sensor is covered by liquid).
     - To distribute the solvent in the system activate pump manually by setting the pump speed (**Slow, Medium, Fast** or **Full**).
     - After approx. 20 s set pump to **Off**.
   - Refill the liquid tank with solvent if necessary.
   - Press **Continue**. The background measurement will start taking ≈ 1 min.

7. For loading the sample, raise the stirrer and turn it CCW 90° to lock it in position.
8. Add sample to the solvent in the chamber, together with a dispersing agent (if required).
9. To lower the stirrer unit, raise it slightly and reposition it in the chamber.
10. Disperse the sample particles in the solvent using the stirrer and ultrasound, followed by distribution in the instrument's tubing and measurement cell using the control panel:

    - **Ultrasound:** Beneficial to disperse particles which tend to aggregate / form clumps.
    - **Stirrer:** The stirrer is recommended to properly disperse the particles in the solvent.
    - **Pump:** The pump distributes the sample in the tubing and measurement cell. Excessive pump speed can induce the formation of air bubbles in the tubing!
      Proper dispersion and equilibration is achieved when obscuration value becomes stable (or near-stable) for more than 10 s.
    - **If obscuration is $< 5\%$, too few particles are present.** Add sample in the tank and repeat the dispersion/equilibration step. **If obscuration is $> 30\%$, particle concentration is too high.** Drain a portion of the sample using the "Drain" sub-menu, then add solvent and repeat the dispersion/equilibration step.

11. Click **Continue** to start the actual measurement.
12. When finished, the results will be displayed and the data saved in the current workbook.
13. Use the action buttons at the top right to export raw data or recalculate results, if needed.

2.3 Finishing up

1. Save the workbook in your folder and indicate the date in the name of a subfolder.
2. Create report (select a template beginning with *LSU* to include the LSU logo).
3. Close the DI water tap, if used (Figure 2b).
4. Drain the liquid tank of the PSA:
   Click the ≡ Ka icon, select **PSA control** and set "Drain" to **On**. Switch to **Off** when done.
5. Empty the bucket.
6. Clean the liquid tank from sample residues using a lint-free cloth wetted with ethanol.
7. Switch off the PSA.
8. Close the Kalliope software and shutdown the PC.
9. Close the water tap, if used.
10. Put the fabric protective cover back on the instrument.