

# Reaction Monitoring with Alltesta TM Autosampler



# Introduction

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#### **Video Links**

**Injection with 48 Vial Plate** 

**Needle Wash with 96 Well Plate** 

#### REACTION SAMPLING

Monitoring of changes during chemical or bioprocess is important task. It is often required to monitor chemical or bio-reaction periodically to obtain information of reaction progress, study reaction kinetics, or monitor completion or state of the process. A manual sampling is not always convenient and accurate and sometimes impossible if this process dealing with aggressive or chemically unstable substances.

Alltesta offers an automated device capable to collect samples from a closed or open vessel periodically with high accuracy and safety.

The system include an autosampler equipped with a special electromechanical probe which immersed in the reaction liquid during the entire process.

Periodically, based on a required time interval, the probe is open and specified amount of sample transferred to a vial or a 96 wells plate. These vials or plate are stored on the Alltesta autosampler. Based on a proprietary alpha/beta mechanical configuration, this Miniature Autosampler offers reliable automation of sample collection.

Samples can be later transferred to an analytical instrument for analysis.

Direct and comprehensive control of the Autosampler's features, as well as high-level commands for succinct automation, provide quick and easy integration.

Users can customize the Autosampler with various valve configurations, tray designs, syringe volumes and pressures. A built-in shaking feature allows for sample-mixing before injection and/or simple sample extraction inside the vial. The device can be controlled directly via our custom OEM software, serial commands or even remotely through the cloud.

The Mini Autosampler can be used for many other liquid-handling applications that require the precision transfer of small liquid volumes under both high or low pressures.

Drivers for Serial Communication as well as our OEM software can be found on our <u>website</u>. For the list of serial commands, please contact us at <u>support@sielc.com</u>



# Introduction

Based on a proprietary alpha/beta mechanical configuration, this Miniature Autosampler offers affordable and reliable automation for many analytical- and liquid-handling settings, including HPLC. Direct and comprehensive control of the Mini

Syringe can be filled up with reactive chemicals such as quench reagent to stop reaction, or pH change pН buffer to for termination of fermentation. The accurate amount of the reagent can be placed in each vial automatically sampling before began.



# **Diagram of Probe**

Metal cap for heat dissipation. Caution: May become hot during intensive use.

Power cable.

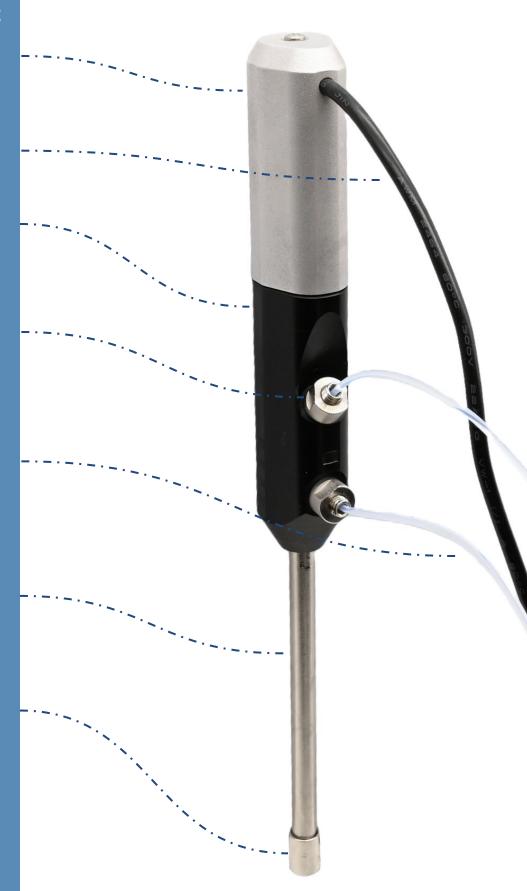
Main body adapted to a comfortable handle.

Two tubing couplers which hermetically sealed Probe with tubing

Two tubes with inner diameter 0,7 mm and 0,5 mm<sup>2</sup> cross-section

Immersion tube into a solution holding the opening end

Opening tip for taking a sample from a solution



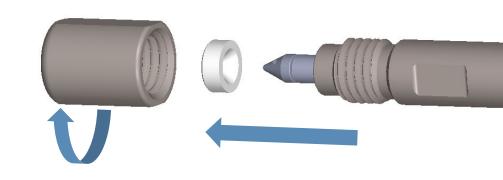


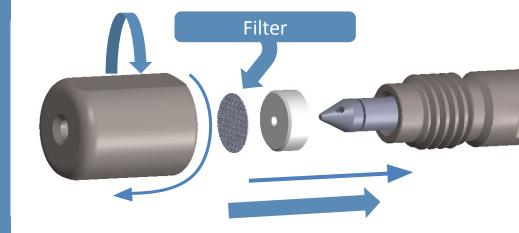
# **Diagram of Probe: Opening Tip**

- 1) The sample probe equipped with an opening on the bottom end of the probe. This opening can be opened or closed depends on the operation state. While it is opened the sample can be aspirate in the probe inner channel.
- 2) When it is closed the probe's internal volume became isolated from the reaction liquid and aspirated sample can be transferred to the vial by inert solvent supplied by the metering syringe.
- 3) The inert solvent can be also used to clean the probe from a previous operations or samples residue.
- 1) The sample probe equipped with an
- 2) When it is closed the probe's internal volume became isolated from the
- 3) The inert solvent can be also used to clean the probe from a previous operations or samples residue.



# Tip maintenance (adding filter):

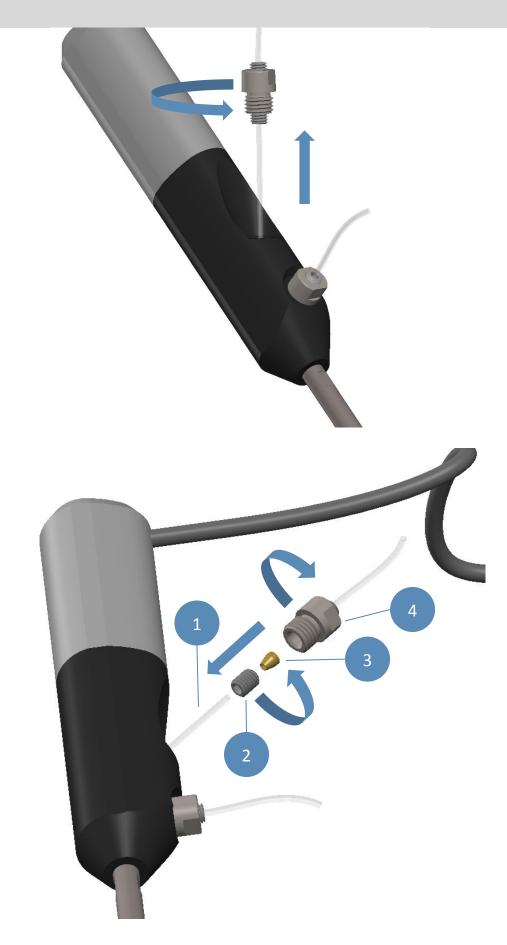






# Diagram of Probe: Tube maintenance

- 1) Replace the PTFE tube with a new
- 2) Reassemble the parts in reverse order

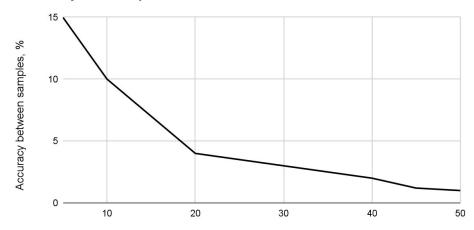




# **Graphs of characteristics**

There are some important dependencies which important to know before set values of sampling parameters:

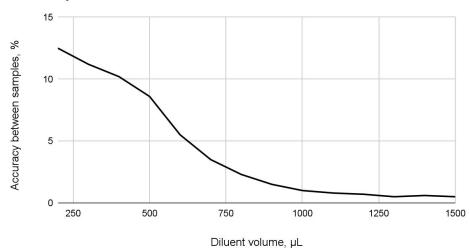
Accuracy vs. Sample volume\*



Volume of Sample, µL

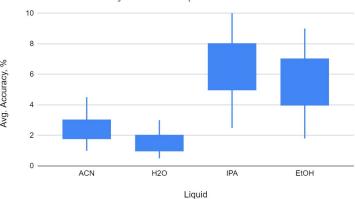
\* — Test was made with Caffeine diluted in water, diluent volume 1mL

#### Accuracy vs. Diluent volume



\* — Test was made with Caffeine diluted in water, sample volume 30

#### Difference in accuracy between liquids



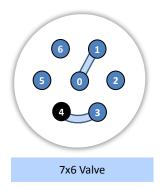


# **Before starting Sampling**

#### **Operation:**

Sampling system performs several operations to provide accurate free of cross-contamination sampling

Based on a proprietary alpha/beta mechanical configuration, this Miniature Autosampler offers affordable and reliable automation for many analytical- and liquid-handling settings, including HPLC. Direct and comprehensive control of the Mini



Quer clean samp

Syringe can be filled up with reactive chemicals such as quench reagent to stop reaction, or pH buffer to change pH for termination of fermentation. The accurate amount of the reagent can be placed in each vial automatically before sampling began.





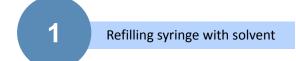
# Flow Diagram: Probe Cleaning

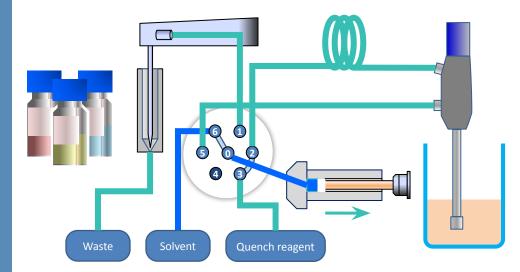
# **Operation:**

- 1) Refilling the syringe with solvent for subsequent cleaning of the probe. It is recommended to select a bigger Probe cleaning volume, since the internal dead volume of the probe is 767 µL.
- 2) Running the solvent through the probe to the waste for cleaning. Depending on the viscosity of the liquid, it is not recommended to select an excessively high Solvent cleaning rate. However, higher Solvent cleaning rate could create pressure to dissolve air bubbles within the probe. While running the solvent through the probe, you can also shake the probe lightly and turn it upside down to help the accumulated air draw with solvent.

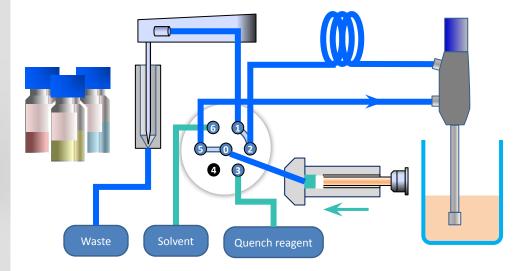


#### **Schematics**





2 Cleaning probe with solvent





Repeat the required number of times

# Flow Diagram: Probe Cleaning with opening

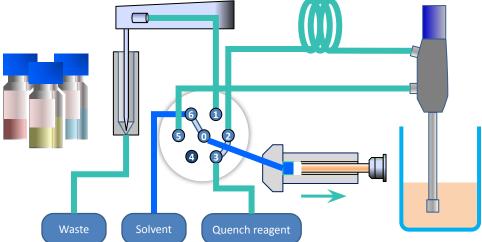
#### **Operation:**

- 1) Refilling the syringe with solvent for subsequent cleaning of the probe. It is recommended to select a bigger Probe cleaning volume, since the internal dead volume of the probe is  $767 \, \mu L$ .
- 2) Running half of the solvent volume through the probe while it is open to ensure the most effective cleaning. In this case, the solvent will preferentially flow out through the open probe tip and wash away the remaining solution along the shortest path, which will reduce the required number of washing cycles.

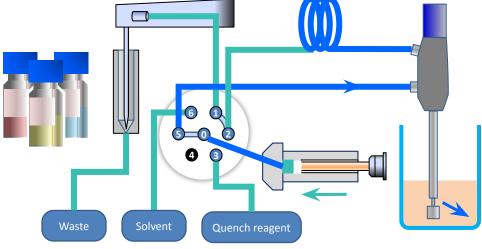


#### **Schematics**

Refilling syringe with solvent



Cleaning open probe with half of the solvent



# Flow Diagram: Probe Cleaning with opening

#### **Operation:**

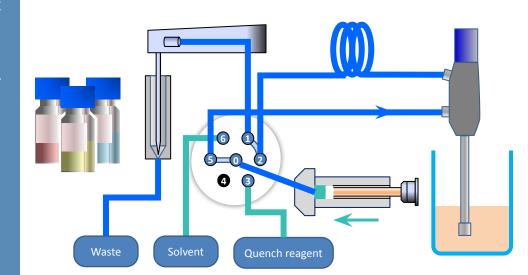
3) Running the last half of the solvent volume through the closed probe to finish cleaning.

Depending on the viscosity of the liquid, it is not recommended to select an excessively high Solvent cleaning rate.

However, higher Solvent cleaning rate could create pressure to dissolve air bubbles within the probe.

#### **Schematics**

Finishing cleaning probe with last solvent





Big picture

# Flow Diagram: Quenching (preparing)

# **Operation:**

1) Tubes are filled with quench from the exact container with quench reagent to the syringe for preparing them for the next step.

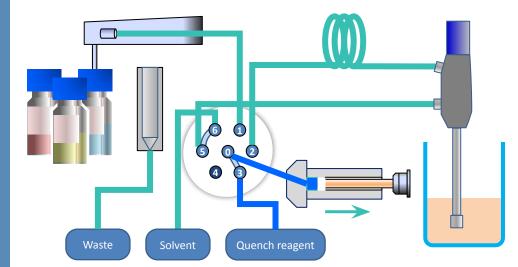
This is important because during operation of the device, some amount of solvent could infiltrate to the tube from the quench container.

This occurs when the syringe draw rate is set to high, which leads to excessively high pressure.

2) Cleaning the Syringe from the liquids that were previously in the tubes.

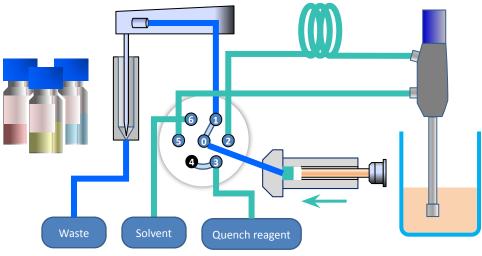


Filling tubes with quench





2 Emptying syringe to waste



# Flow Diagram: Quenching (cycle)

# **Operation:**

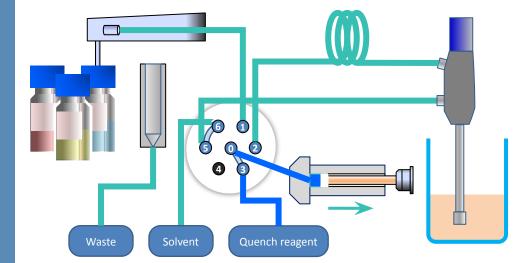
3) Syringe is filled with the required amount of quench, which is calculated as multiplication of the number of vials to be filled by the volume of quench required in one vial.

If the total volume of the quench exceeds the volume of the syringe - this cycle will be repeated.

4) Filling the vials with quench one by one: the needle lowers into the vile, the required amount of quench is flows out of the syringe, then a pause is required to normalize the pressure and the process is repeated with the next vial.

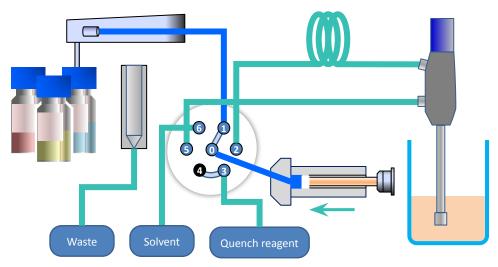


Filling syringe with quench





Filling vial with quench





Step 4 is repeated for each Vial (or steps 3 and 4)

# Flow Diagram: Syringe Cleaning

# **Operation:**

1) Refilling the syringe with solvent to wash it from the previous solution.

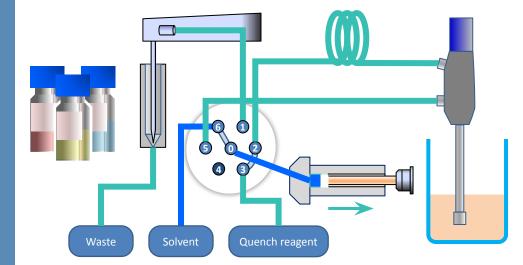
It is recommended to fill the syringe at high refill rate to provide better mixing.

It is also recommended to set a smaller Syringe cleaning volume to ensure efficient solvent consumption and save time.

2) Drawing the Syringe to waste and repeat the cycle. The process should be repeated several times depending on the solubility characteristics of the liquids.

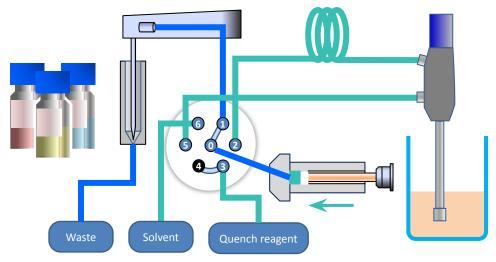
#### **Schematics**

Refilling syringe with solvent





2 Emptying syringe to waste





Repeat the required number of times

# Flow Diagram: Reactor Sampling with Probe

# **Operation:**

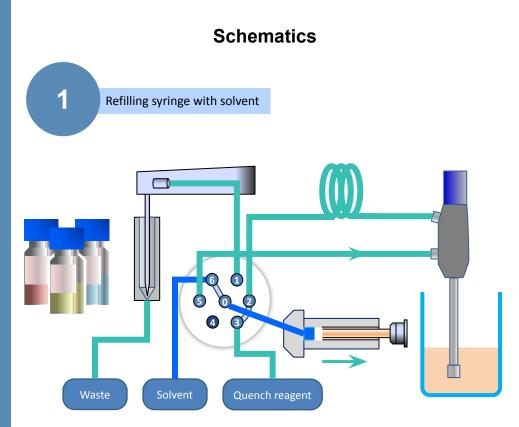
1) Refilling the syringe with a solvent volume greater than the Diluent volume, this solvent will subsequently be used to dilute the sample and go to a vial.

After filling, there is a pause to normalize the pressure to ensure better sampling accuracy.

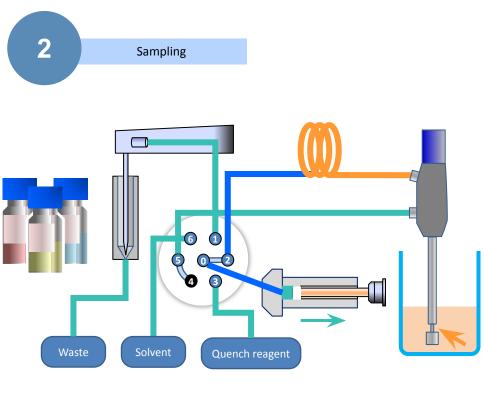
2) The main step. Probe opens and the sample is refilled. Delay auxiliary after the refilling and Sample refill rate depend on the viscosity and density of the liquids: solution and solvent.

A lower Sample refill rate and a properly chosen Delay auxiliary allow to achieve greater accuracy.

If the density of the solution is less than the density of the solvent, it is recommended to select a higher Sample refill rate and reduce Delay auxiliary.







# Flow Diagram: Reactor Sampling with Probe

# **Operation:**

3) Drawing an excess volume of solvent through the tubes to the waste, this volume is less than the internal volume of the tubes.

This is necessary to bring the sample as close as possible to the needle for subsequent filling the vial. Then the volume of solvent for dilution will most effectively wash the sample from the tubes directly into the vial.

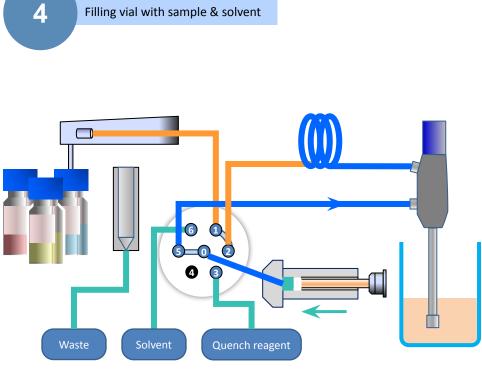
4) Aspirating the remaining volume of the syringe into the vial, while simultaneously lifting the needle so as not to wet it.

# 3 Draw excess solvent from tubes

Quench reagent

Solvent





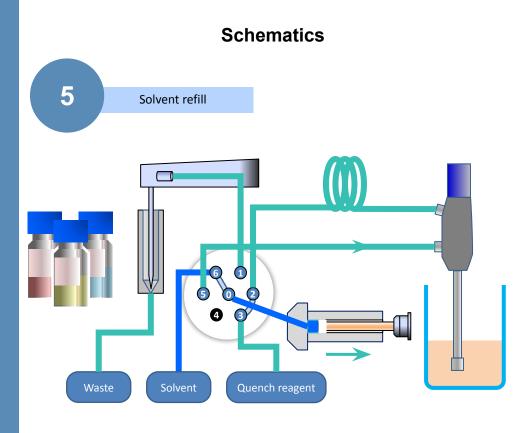
# Flow Diagram: Reactor Sampling with Probe

#### **Operation:**

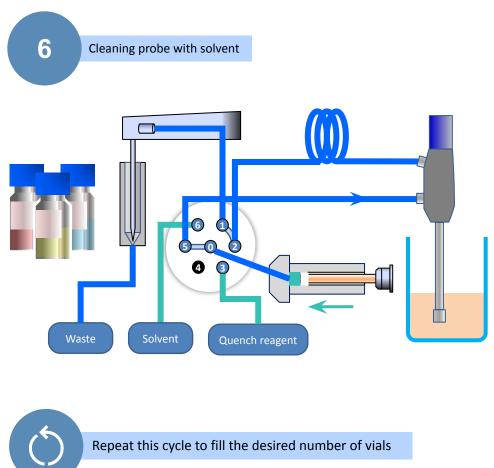
- 5) Refilling the syringe with solvent for subsequent cleaning of the probe. It is recommended to select a bigger Probe cleaning volume, since the internal dead volume of the probe is 767 µL.
- 6) Running the solvent through the probe to the waste for cleaning. Depending on the viscosity of the liquid, it is not recommended to select an excessively high Solvent cleaning rate.

However, higher Solvent cleaning rate could create pressure to dissolve air bubbles within the probe.

While running the solvent through the probe, you can also shake the probe lightly and turn it upside down to help the accumulated air draw with solvent.







# Software options

#### **Options:**

1) Serial communication allows customers to achieve maximum customization by giving them complete control over automated programs.

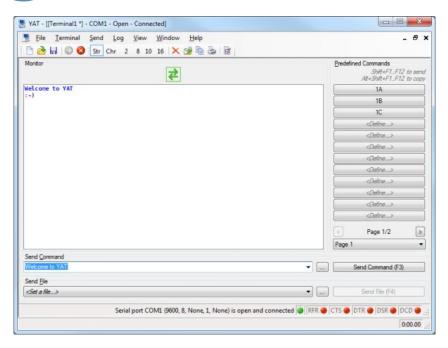
All Autosampler purchases will come with a copy of the Command Protocols so you can start building up your Automated Program as soon as possible.

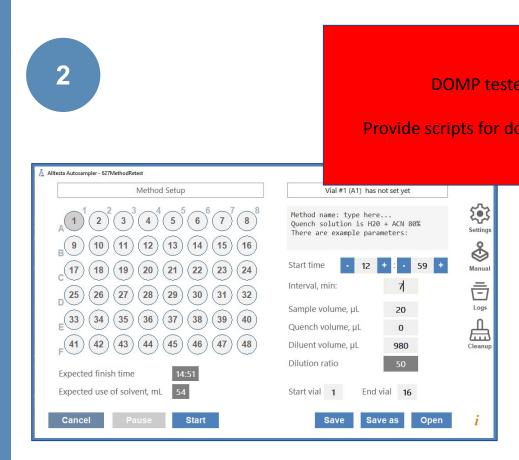
Serial communication also has the added benefit that it is compatible with any operating system as long as you have a Serial terminal installed, have downloaded the correct drivers, and the Autosampler is connected to your computer via the included USB A – USB B cable.

2) OEM software gives users simple manual control over each component within the Autosampler, ideal for simple tests that do not require automation.

The software can be downloaded directly from our website and is compatible with both Windows 7 and Windows 10. Once it is installed and your device is connected, you can begin controlling it immediately!

1





# Included software

#### **Options:**

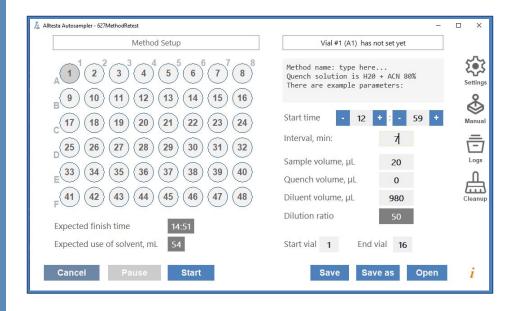
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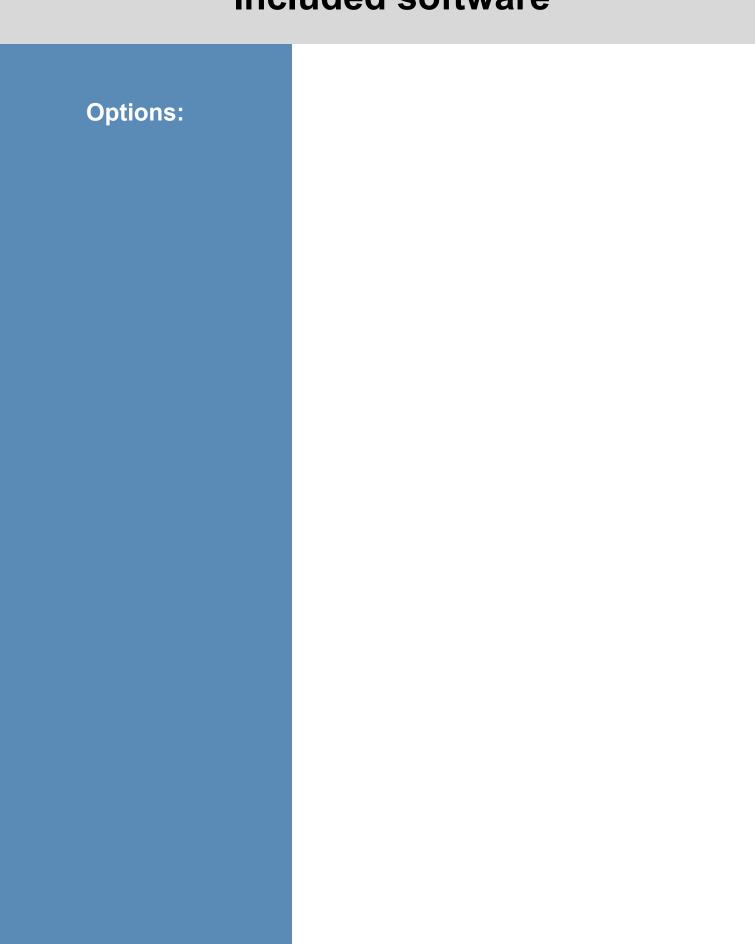
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first pause and wait until the done, then press stop if you need emergency stop immediately

# **Included software**



# **Specifications**

#### **Instrument Size:**

6 x 6.5 x 7 inch (15 x 16 x 17 cm)

Weight: 5 lb (2.3 kg)

**Vial Capacity Options:** 

48 vial plate 96 well plate

**Pressure Max:** 

250 psi/ 14 bar (4000 μL volume)

Syringe Capacity Options:

4000 μL

**Valve Options:** 

7x6

**Volume Accuracy:** 

1 μL (4000 μL volume)

Probe dead volume:

767 µL

Syringe draw/refill rates:

48,000 μL/min

Communication:

**USB (type B) COMM port** 

Power: 24 V

**Contact with liquid:** 

Stainless Steel 316 (316L)

PEEK PTFE Vespel



48 Vial Plate



