

Special Tools Included with all new E-Series Flow Chemistry Systems



The robust E-Series system from Vapourtec was specifically developed to be easy to use.

A large contributor to the ease of use is the straightforward touch panel interface, which allows a user to simply set flow rates and temperatures and start work immediately.

But the interface also includes some advanced tools to make it easier to figure out the right settings in the first place, and to run experiments unattended.

These include tools to

- Determine when the output stream has reached steady concentration (and therefore how to collect the maximum quantity of the desired product for the minimum reagents used)
- Understand the mixing characteristics of the chosen reactor, obtaining clear graphical representations of dispersion and residence time distribution (RTD) data.
- Find the pressure required to prevent boiling for a given solvent at a particular temperature
- Determine the reduction in residence time that might be expected from running a reaction at a higher temperature.

This article explains some of these tools, and shows how to apply them to gain greater understanding of what goes on in the system.

Intuitive User Interface



Important Features

- Icons for accessing the productivity tools positioned across the top of the screen.
- Controls for starting and stopping reactions arranged on the right hand side.
- Actual temperature of each reactor position including, reactor power and set point temperature.
- Pump flow rate control including, set point, pump pressure and “push button” for pump priming.
- Clear display of reactor pressure
- Large fonts visible from “across the lab”

How to Access the Tools

Along the top of the main control interface screen on all E-Series systems is a series of buttons which each lead to another screen:



These are

- **Timer Tool** (for running a whole reaction experiment automatically)
- **Tubing Selector** (to determine which of the two fluoro-elastomeric pump tubing types is appropriate for pumping the chosen solvent/reagent)
- **Arrhenius Calculator** (for determining the reduction in residence time that can be expected from running a reaction at higher temperatures)
- **Stoichiometry Calculator** (for determining reactor residence times from combined flow rates, determining axial dispersion in reactors and obtaining residence time distribution data)
- **Boiling Point Calculator** (for working out how hot a volatile solvent can be taken, for example)
- **Settings Screen** (for general system configuration).

In this article we are excited to give a brief overview of the advanced tools supplied with all versions of the powerful E-Series system.



The Timer Tool

Allows programming of the E-Series to run a fully automated reaction.

Timer - Reaction Automation

1. Pump solvent at transition flow rate until reactors reach target temperature, then start reaction timer.

Transition Flow Rate
250 $\mu\text{L}/\text{min}$

	2	3	4	5	6	7
	Switch to target flow rates	Switch to pumping reagent	Switch to pumping solvent	Start Collecting	Stop Collecting	Switch to transition flow rate
A		0:00	0:00			
B	0:00	0:00	0:00	0:00	0:00	0:00
C		0:00	0:00			

8. Pump at transition flow rate until reactors all return to safe handling temperature.

← Back Status: Not Running

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W→C Waste

Collection Valve

Timers

Stop All

Stop Reaction

Run

An automated reaction can be specified to include the following steps:

- Heat the reactor(s) to target temperature
- Once reactors reach temperature change to the reaction flow rate
- Switch each pump from solvent to reagent
- Switch the collection valve on/off at the appropriate time to capture the desired part of the reaction output
- Switch back to pumping solvent after the reaction
- Switch back to low flow rate until reactors return to a safe temperature

To follow all these steps manually would require the user to be present for most of the experiment and to use some sort of timer.

The Timer Tool eliminates that need. It allows a single experiment to be run from start to finish, in an automated fashion, with the instrument shutting off safely at the end.



The Tube Selector Tool

To determine which of the two fluoro-elastomeric pump tubing types is appropriate for pumping the chosen solvent/reagent

Tubing - Pump Tubing Chemical Compatibility

Compatibility	Chemical
Do not use	Bromo tritloride
	Bromobenzene
	Butadiene
	Butane
Reduced life	Butyl acetate
	Butyl alcohol
Reduced life	Butyl ether
Reduced life	Butylamine
	Butylene
	Butylene glycol
	Butyllithium solution 1.6M in hexanes (BuLi)
	Butyric acid
	Calcium acetate
	Calcium carbonate
	Calcium chlorate
	Calcium cyanide
	Calcium hydrogen sulfite

← Back

Caution Contact between the BLUE tube and the following reagents will cause severe degradation and leakage:
 - Butyllithium
 - Bromine
 - Nitric Acid

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W→C Waste

Collection Valve

Timers

Stop All

Stop Reaction

Run

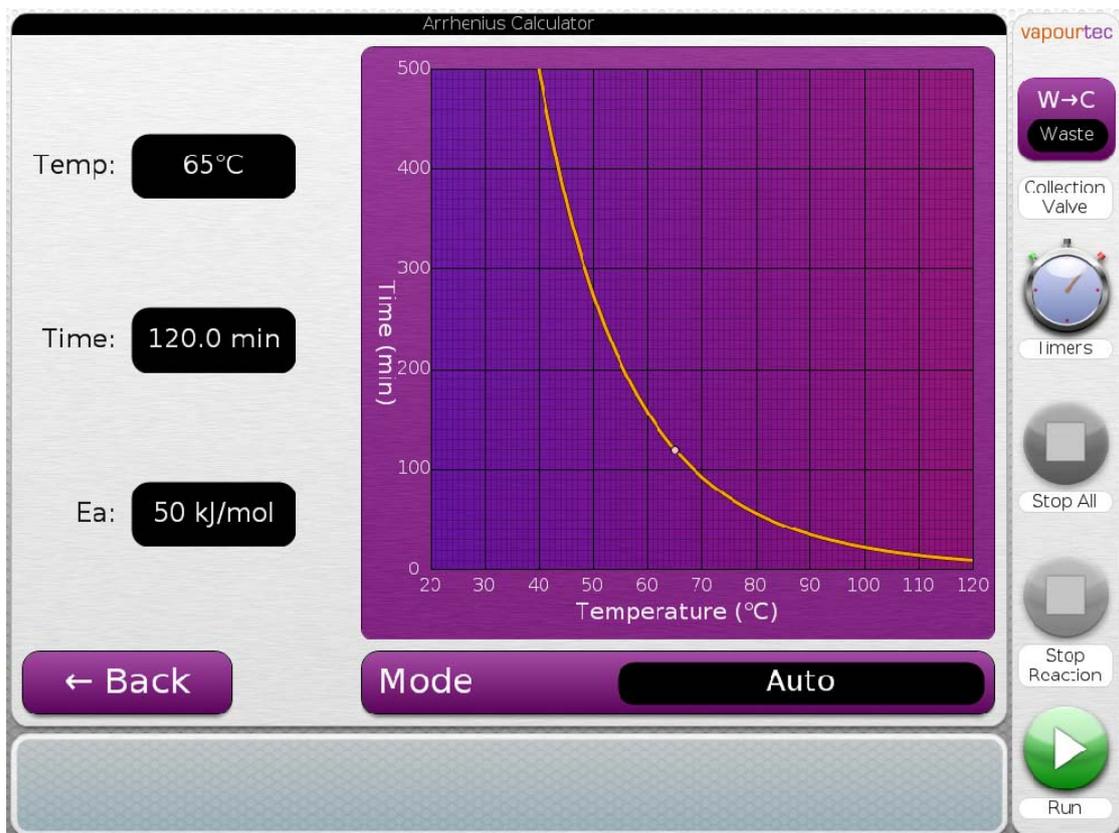
Each E-Series system is supplied with two different peristaltic pump tube materials, RED and BLUE. Both pump tubes are manufactured from a fluoro-elastomer but the RED and BLUE tubes have complementary chemical compatibilities

- Simply scroll through the list of 300 of solvents / reagents to select the liquid to be pumped
- Determine the correct coloured tube for each pump channel
- Ensure the correct tubes are fitted, then plumb each liquid to the chosen pump and start the reaction.



Arrhenius Calculator

For determining the reduction in residence time that can be expected from running a reaction at higher temperatures



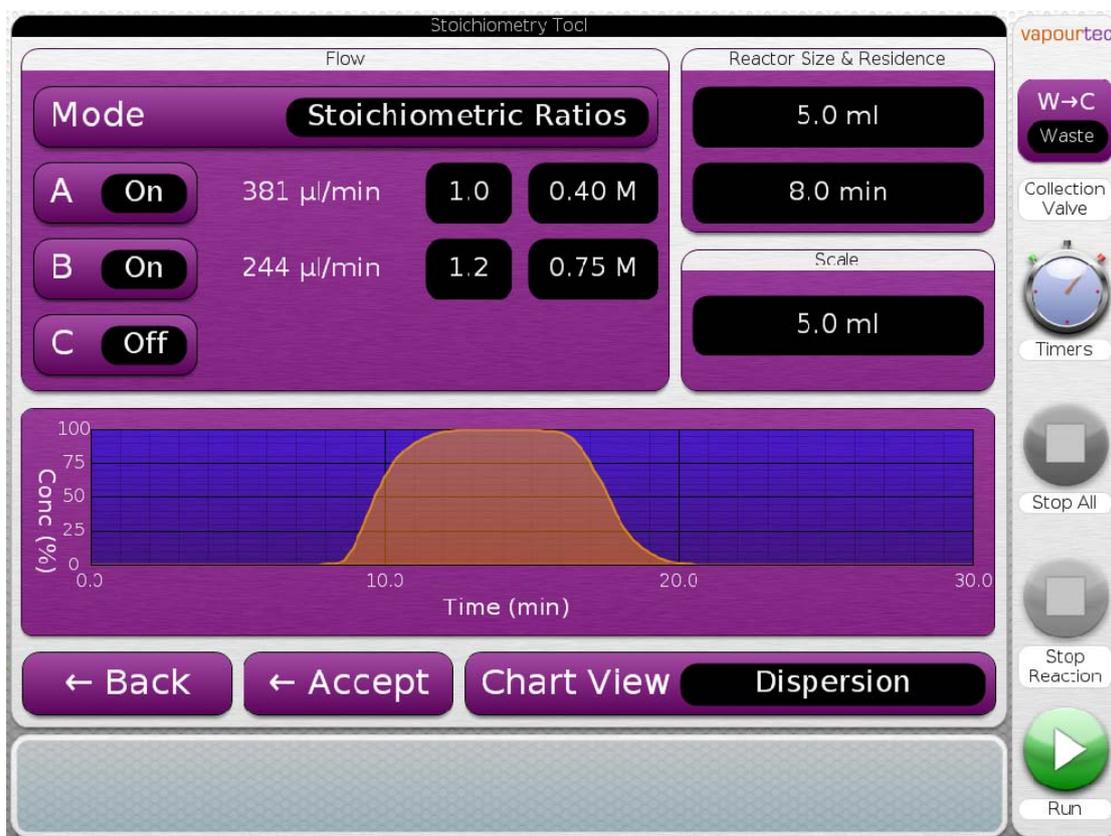
An estimate for the reaction time can be determined from one known reaction condition and knowledge of the approximate activation energy, as follows:

- Enter the value for the activation energy
- Enter the known reaction condition. In the above example the batch condition was a reflux (in Methanol) at 65°C for 2 hours.
- Use the graph to estimate the time for higher (or lower) temperatures
- The mode button can be used to change the scale of the graph between Low, Auto and High temperature zones.



Stoichiometry Calculator

For determining pump flow rates required to achieve residence times in a particular reactor from combined pump flows using either volumetric ratios or stoichiometric ratios. Also to calculate axial dispersion in reactors and obtain residence time distribution data – This is an excellent teaching tool.



By way of example using Stoichiometric Ratios, data is entered as follows:

- Enter the concentration of the reagents
- Enter the relative molar concentration of reagents required in the reaction
- Enter the reactor size (5 ml), the residence time (8 min), the amount of reagent A to be processed (scale, 5 ml)

The tool will then determine the required flow rates and will also calculate and display a graph showing the variation in concentration of reagents with time at the collection point. Zero on the time axis is the moment that reagent A is switched from solvent to reagent, so the dispersion graph can be used to help estimate collection timing when setting up the timer screen.

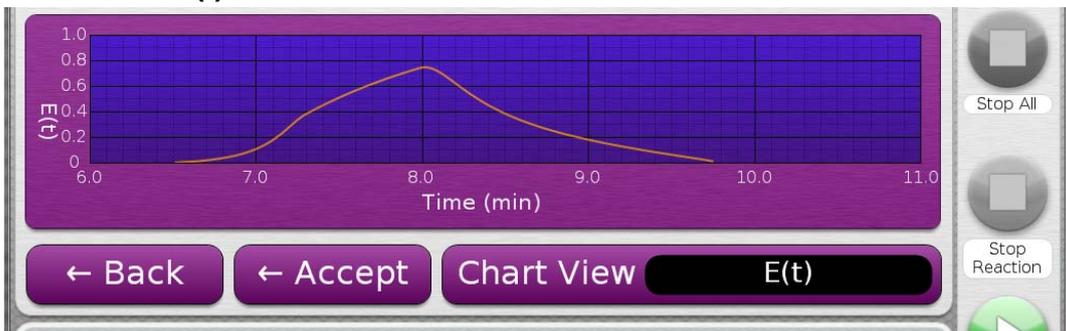
The chart view button allows the dispersion information to be viewed in a different way, showing reactor residence time distribution (RTD) instead. The RTD data can be used simply as a teaching tool or to compare the mixing performance of the E-Series reactors with reactors that may be used for pilot or production scale reactions:

Chart View – Histogram



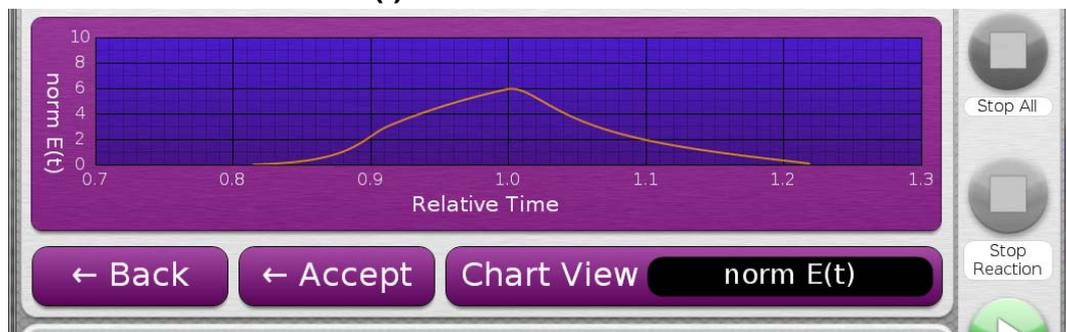
This view shows what fraction of the output falls into each residence time range.

Chart View – E(t)



In this continuous chart, the area under this graph between any two time values equates to the fraction of the total product output which has a residence time through the reactor between those values. (The total area under the graph is 1).

Chart View – Normalised E(t)

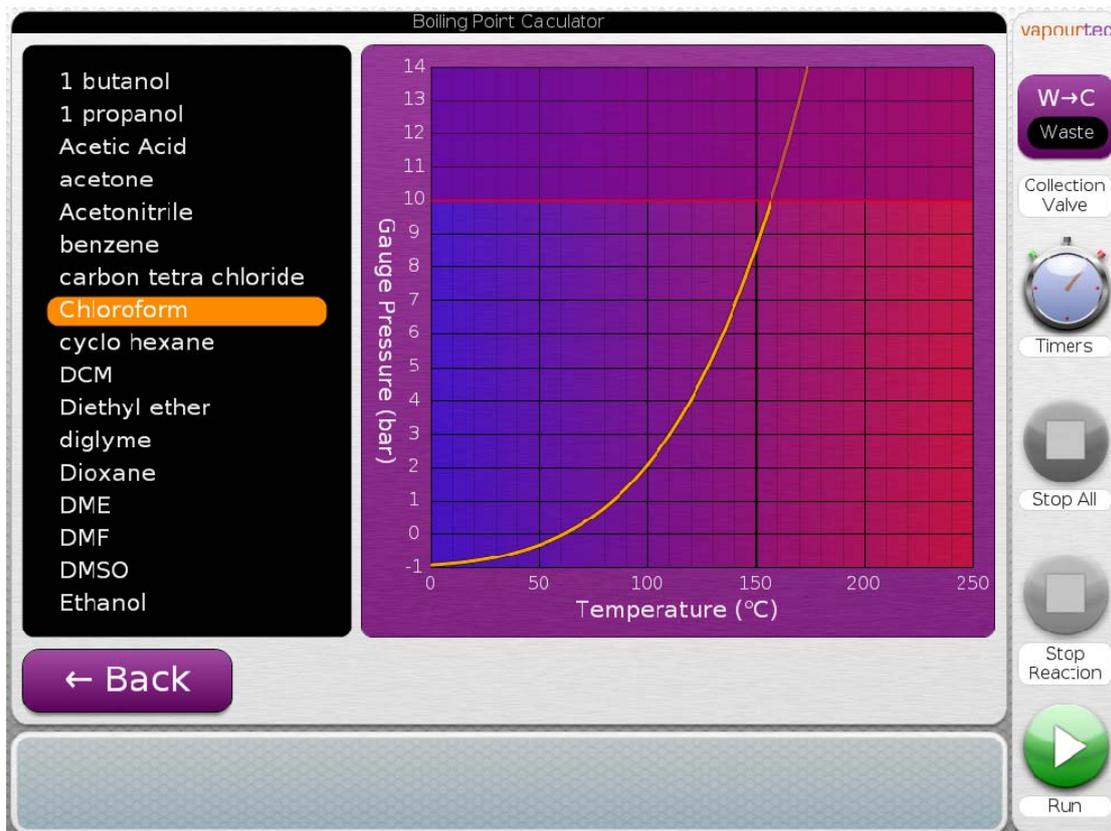


This is the normalised form of the E(t) curve, commonly used to compare the mixing performance of different reactors.



Boiling Point Calculator

This is used for working out how hot a reaction solvent can be taken (at a particular reactor pressure) without the creation of vapour bubbles.



The E-Series user interface has saturated vapour pressure (SVP) data stored for 17 common synthesis solvents. The screenshot above shows an example of this feature in use. Chloroform is the solvent, and the desired reactor temperature of 120°C:

- Select the solvent of interest (chloroform). The SVP for the highlighted solvent (chloroform) is presented graphically.
- The minimum pressure equating to a reactor temp. of 120°C is 4 bar.
- So for a reaction with chloroform at 120°C, reactor pressure would need to be set a little higher than 4 bar. 6 bar would be ideal.

The easy-MedChem user interface

The features described above are present on all E-Series systems. In the next newsletter article we'll look at the extra features included with the optional easy-Medchem software.