



Data Analysis for the TAM-Air

For the McCoy Group

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Part A: Constructing the Data File

1. Export the TAM-Air experiment data as a '.txt' file.
2. Open the Read-Me file for the experiment.
Information about the empty and full vial weights, start-of-reaction time ("Time Stir"), and start-of-experiment time ("TAM-Air start at...") should be recorded here.

KEEP IN MIND...

This manual assumes some level of familiarity with navigating KaleidaGraph. If you would like to learn the basics of this software, go to: <https://nmt.edu/academics/mtls/faculty/mccoy/links.php>
Expand the "Overall Lab Manual" tab, and look through the "KaleidaGraph Plotting and Data Analysis Manual".

Vial	Empty Weight (g)	Full Weight (g)	828DEA Weight (g)	828 (g)	DEA (g)	Phenol (g)	Actual Phenol / "Free"828 ratio	Time Stir:	Baseline Shift: (TAMAIR Start at 11:36AM)
1	7.076	19.71	12.634	25.0	0.276	0	0.000	11:38 AM	-2 mins
2	7.154	15.784	8.63						
3	6.792	19.376	12.584						
4	6.847	15.096	8.249	25.0	0.278	2.69	0.200	11:53 AM	-17 mins
5	7.12	19.482	12.362						
6	7.036	18.463	11.427	25.0	0.287	4.06	0.301		
7	6.892	19.539	12.647						
8	6.905	17.863	10.958						

3. Calculate the sample weights by subtracting the empty vial mass from the full vial mass.
4. Calculate the time shifts ("Baseline Shift") by subtracting the start-of-reaction times from the start-of-experiment time.
5. Open the Master-datafile in KaleidaGraph.

KEEP IN MIND...

If many mixtures were batched for the experiment, you may need to calculate multiple time shift values. Keep track of which time-shifts apply to which samples.

Text File Input Format

Delimiter: Tab Space Other Special

Number: = 1 >= 1 >= 2 >= 3

Lines Skipped: << >>

Options: Read Titles

Help... View Text...

Time•Heat flow [Signal, 517:1]•Heat flow [Sign
-5.60628414154053•2.14292919549888E-05•
-4.88865876197815•2.14288476022114E-05•
-4.17103290557861•2.14284032491386E-05•

Control Char: • = Tab ↵ = Return ⌘ = Other

OK Cancel

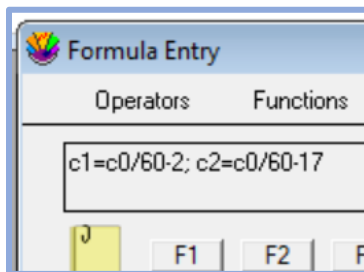
6. Make all entries Float values (through Ctrl+A. Ctrl+K).
7. Save this data file to your computer with some name indicating the experiment, and the date of when it was exported.
 - a. If this is the first export of this experiment's data, duplicate this data file. The name of the duplicate should indicate that it is the Master-File for the experiment. **Do not analyze the data of this Master-File duplicate.**
 - b. If this is not the first export of this experiment's data, refer to Part E.
8. Add four columns to the left of each "Heat flow [Signal]" column. Name the columns as such for each sample:

Heat Data 1 (W)	Heat Flow 1 (W/g)	Corr. Heat Flow 1 (W/g)	Int. Heat 1 (W min/g)	Final Heat 1 (J/g)
C2	C3	C4	C5	C6
2.1429e-05				
2.1429e-05				

9. Add more columns to the left of the "Time (s)" column. You will need as many columns as there are time shifts for your sample set. Name the columns as shown below (but edit the "#-#" values to reflect which samples the shifted data corresponds to).

Time (s)	Time 1-4 (min)	Time 5-8 (min)
C0	C1	C2
-5.6063		
-4.8887		

10. For each new time column, divide the Time(s) column by 60 to convert to minutes, and subtract/add the shift value.



11. Highlight any initial, negative entries within each “Time (min)” column. Clear them by right-clicking and selecting “Clear Data”. Do not delete any entries in the “Time (s)” column.

	Time (s)	Time 1-4 (min)	Time 5-8 (min)	Heat Data
	c0	c1	c2	
5	-2.3770	-2.0396	-17.040	2.14
6	-2.0183	-2.0336	-17.034	2.14
7	-1.6594	-2.0277	-17.028	2.14
8	66.598	-0.89004	-15.890	2.13
9	119.71	-0.0049028	-15.005	2.13
10	182.00	1.03		
11	244.30	2.07		
12	309.93	3.16		
13	383.57	4.39		
14				

COMMON ERROR...

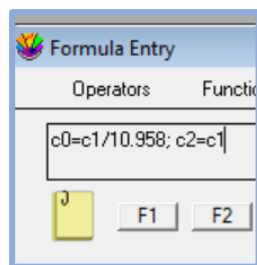
When clearing this data, DO NOT click any other operation (Cut, Clear, backspace, etc.). These operations shift the remaining data up to row 0, and we want to preserve the cell locations of our values.

12. Navigate to the very last rows of the dataset and delete the zeroes at the tail-end of the data. These zeroes are a hindrance to our long-time corrections (in Part B).

	Time 5-8 (min)	Heat Data 1 (W)	Heat Flow 1 (W/g)	Corr. Heat Flow 1 (W/g)	Int. F
	c2	c3	c4	c5	c6
16251	16242	0.00037861	2.9968e-05		
16252	16242	0.0000	0.0000		
16253	16242	0.0000	0.0000		
16254	16242	0.0000	0.0000		
16255	16242	0.0000	0.0000		
16256	16242	0.0000	0.0000		
16257	16242	0.0000	0.0000		
16258	16242	0.0000	0.0000		
16259					
16260					

13. Highlight a “Heat Data (W)” column so that it is designated as C0. Calculate “Heat Flow (W/g)” for that sample by dividing the “Heat Data” column by the sample weight. Make “Corr. Heat Flow (W/g)” equal to “Heat Flow (W)”.

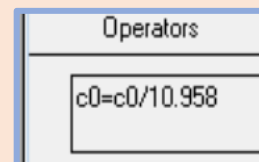
	Heat Data 1 (W) c0	Heat Flow 1 (W/g) c1	Corr. Heat Flow 1 (W/g) c2
0	2.1429e-05		



Repeat these operations for each sample

COMMON ERROR...

It can be tempting to try and “save space” in your data file, and perform operations within a column like so:



Don't self-reference a column like this, not for any step in this procedure. It will only cause trouble in the event that your calculations need revising.

KEEP IN MIND...

You will not need to retype any “C#” column markers in the formula window if you make the “Heat Data # (W)” for each sample C0 in turn.

KEEP IN MIND...

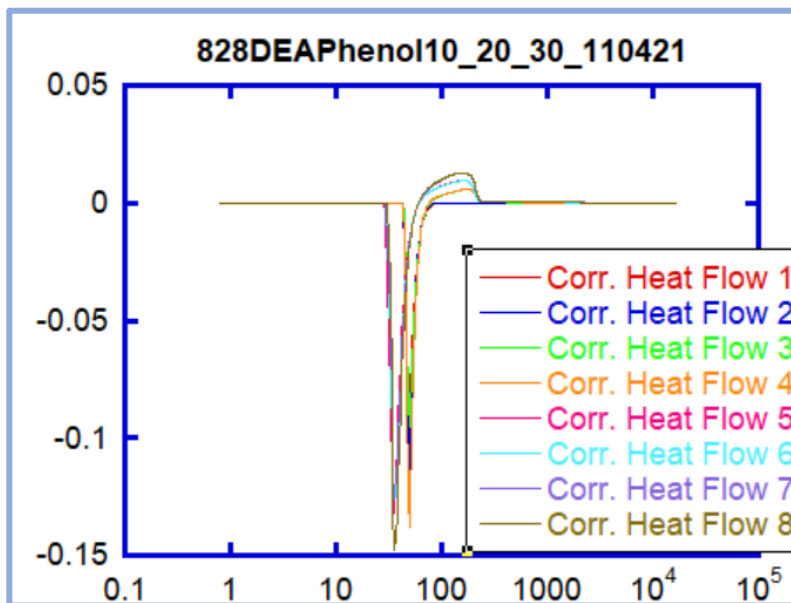
Generally, you will be dividing “Heat Data” by the sample's overall weight to generate “Heat Flow”. However, it may be more appropriate for some systems – specifically those with non-828DEA additives- to divide by parameters like 828DEA weight, additive weight, etc. Consult with Dr. McCoy on this.

Part B: Long-Time Corrections

1. Plot “Time (min)” vs “Corr. Heat Flow (W/g)” for all samples.

X	Y	Name	Position
<input type="checkbox"/>	<input type="checkbox"/>	Time (s)	0
<input checked="" type="checkbox"/>	<input type="checkbox"/>	Time 1-4 (min)	1
<input type="checkbox"/>	<input type="checkbox"/>	Time 5-8 (min)	2
<input type="checkbox"/>	<input type="checkbox"/>	Heat Data 1 (W)	3
<input type="checkbox"/>	<input type="checkbox"/>	Heat Flow 1 (W/g)	4
<input type="checkbox"/>	<input checked="" type="checkbox"/>	Corr. Heat Flow 1 (W/g)	5

A plot like the one below will appear:



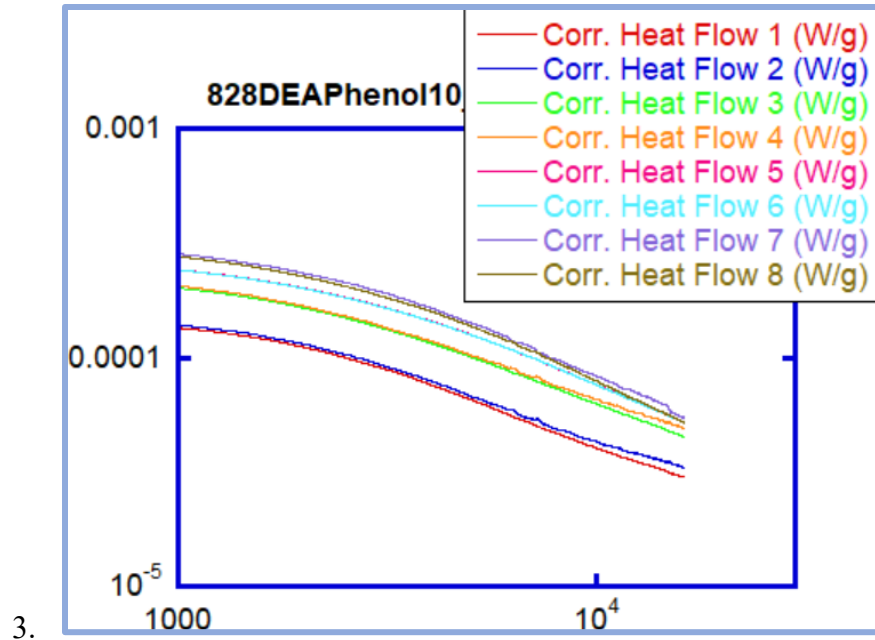
2. Make the axes Log-Log, and zoom in on the long-time portion of the heat curves (as pictured below). Under the “Plot” tab, enable “Auto-Link”. You may find it helpful to make the heat curves Hairline width.

WHY THIS IS IMPORTANT...

Logically, we know that the heat curves of every channel should approach zero (or at least some constant rate) as time approaches infinity. However, small baseline errors in the TAM-Air can prevent the heat curves from converging as they should. Shifting the data by some low-power constant (the effects of which should be only perceptible in the curve’s “tail” on a log-log plot) can correct these baseline errors.

COMMON ERROR...

When plotting your heat curves, make sure you are matching each heat column with the correct time column for that sample.



4. In Variable Settings, blank out all the heat curves except the line you are currently adjusting and your chosen reference line.
5. Click into the “Heat Flow (W/g)” column of the sample you are currently adjusting, so that it is designated as C0. Adjust “Corr. Heat Flow (W/g)” as shown below, varying the low-power constant you are adding or subtracting until the desired effect is achieved. If Auto-Link is on, the curve you are correcting will automatically shift as you adjust this constant.

	Heat ... 2 (W/g) C0	Corr. ...2 (W/g) C1	Int. H... min/g C2
0	0.0000	0.0000	
1	2.3970e-06	2.3970e-06	
2	2.3969e-06	2.3969e-06	
3	2.3967e-06		
4	2.3966e-06		
5	2.3965e-06		
6	2.3964e-06		
7	2.3964e-06		
8	2.3830e-06		

Formula Entry

Operators Functions

$c1=c0+1e^{-c}$

J F1 F2 F3

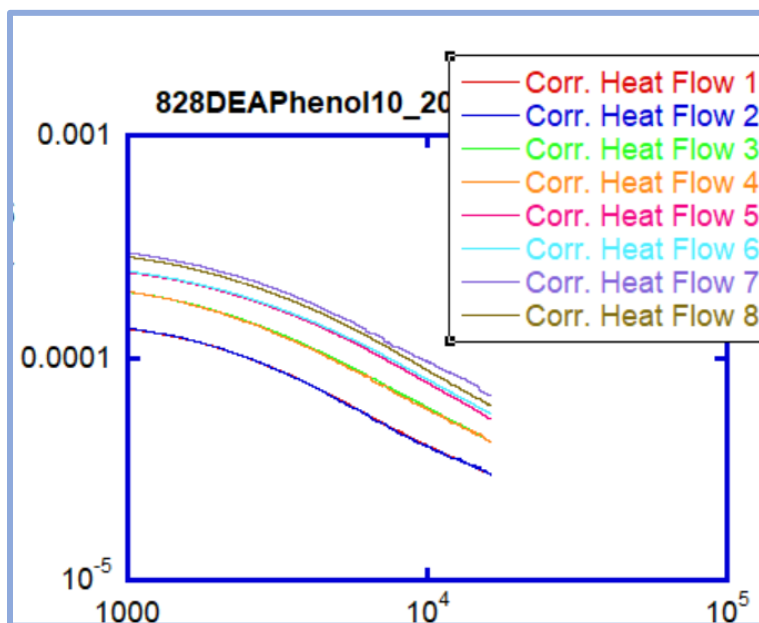
KEEP IN MIND...

The goal of your long-time correction can vary. You may be trying to make the slopes of each curve parallel, or you may be trying to make the endpoints of the curves converge. Whatever the case, you will need to choose a reference curve that all other curves are adjusted to match. Here our reference is Sample 1, whose long-time slope will be approximately replicated across all samples

(You may want to record the long-time correction constants for each sample in the Read-Me file, so that analysis of data-splices is expedited (see Part E).)

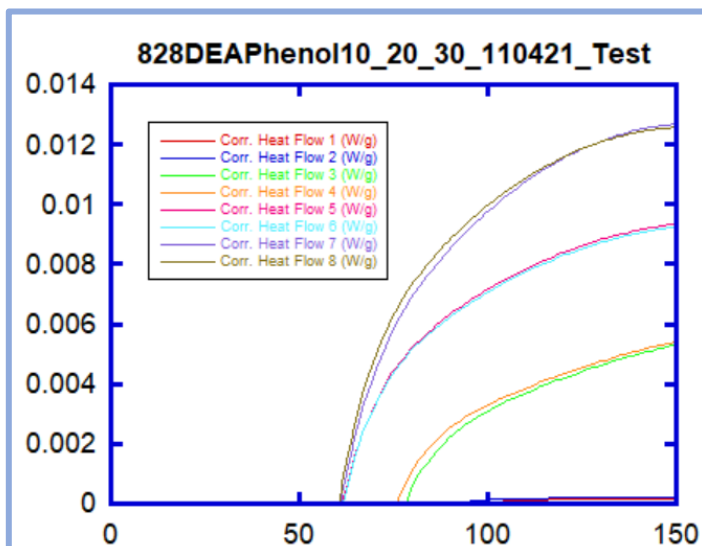
6. After one curve has been adjusted, blank it out in Variable Settings and move onto the next curve.

The plot of a long-time-corrected dataset is shown below:



Part C: Short-Time Corrections

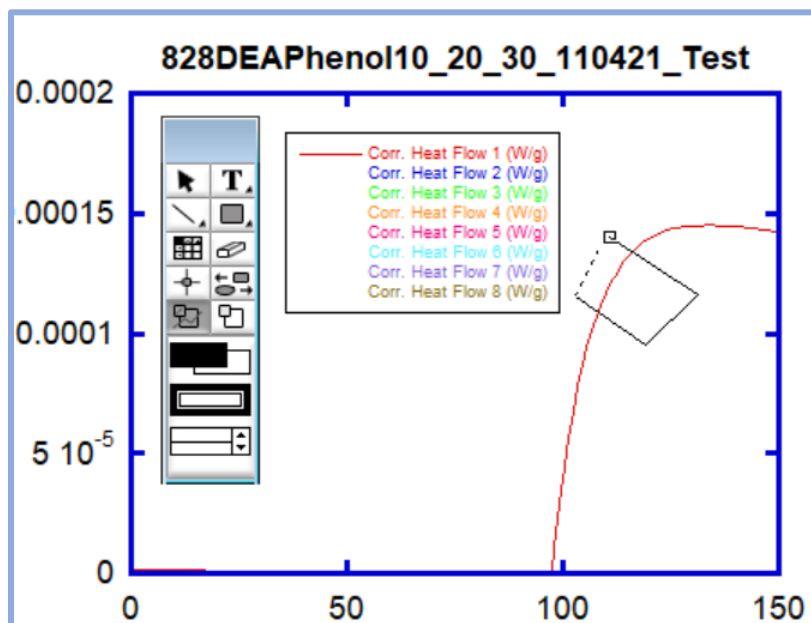
1. Plot "Time (min)" vs "Corr. Heat Flow (W/g)" for all samples. Make the axes linear-linear with a (0,0) minimum. Zoom into short-time, as shown below:



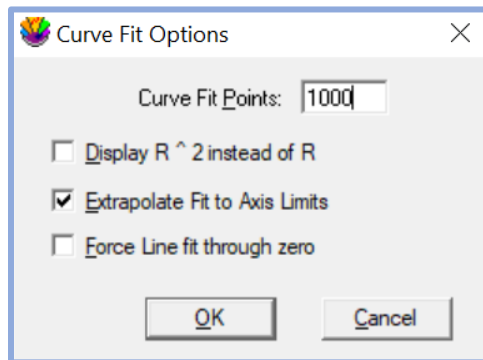
WHY THIS IS IMPORTANT...

Your time values have been adjusted so that $t=0$ is the start of the reaction. However, some amount of time passes between the start of reaction and the placement of samples into the TAM-Air. The heat profile of this interim is lost, and must be approximated. As such, we will graft a short-time curve that passes through (0,0) onto our data.

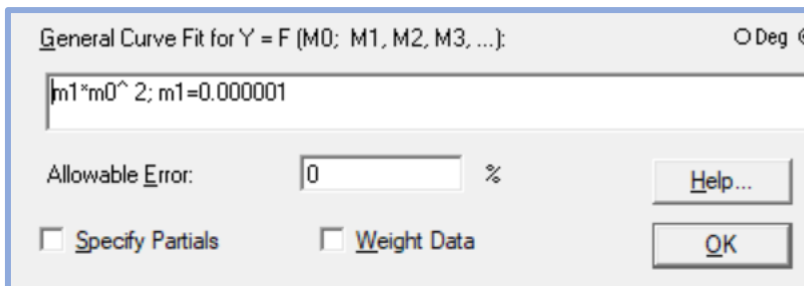
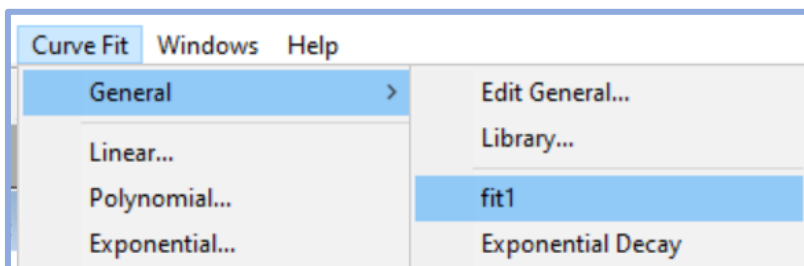
2. In Variable Settings, blank out all the heat curves except the line you are currently correcting.
3. Imagine a line extending seamlessly from (0,0) to some point on the curve. Using the Masking tool, isolate that "connection" section of the curve. Do not "unmask" the data until Step 6.



4. Under Curve Fit Options, enable “Extrapolate Fit to Axis Limits”.



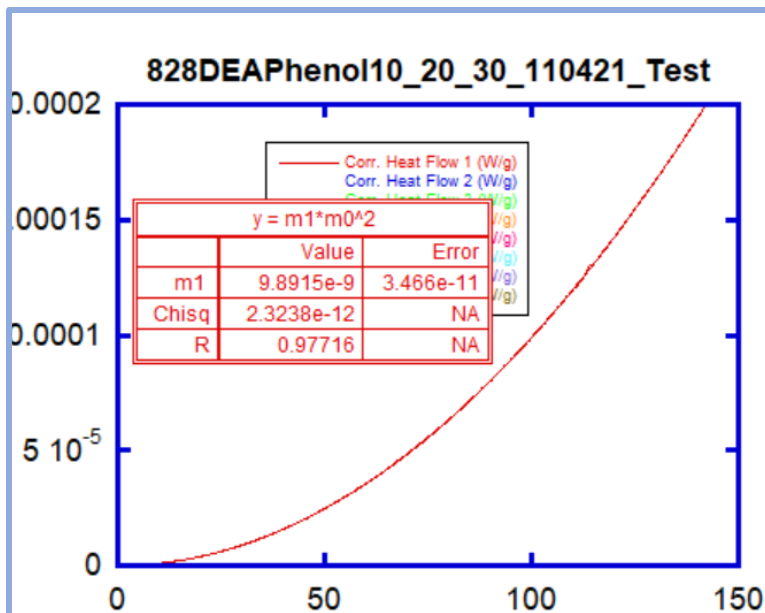
5. Use a customizable General curve-fit on whichever line you are working with. Input the appropriate formula for your short-time approximate heat curve.



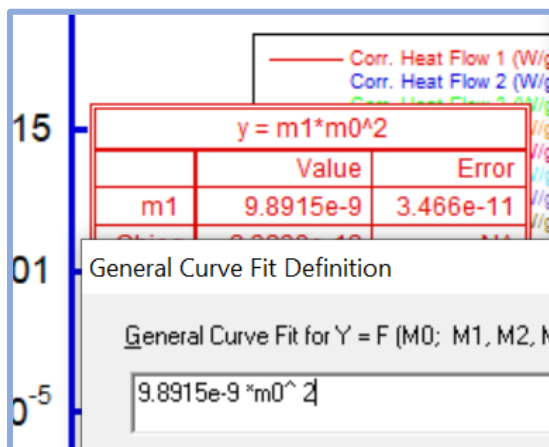
KEEP IN MIND...

Your short-time approximate heat curve may be linear, polynomial, or even exponential. In any case, the generated curve should be positive for all $t > 0$ and should pass through $(0,0)$. Here, we are using a 2nd degree polynomial fit.

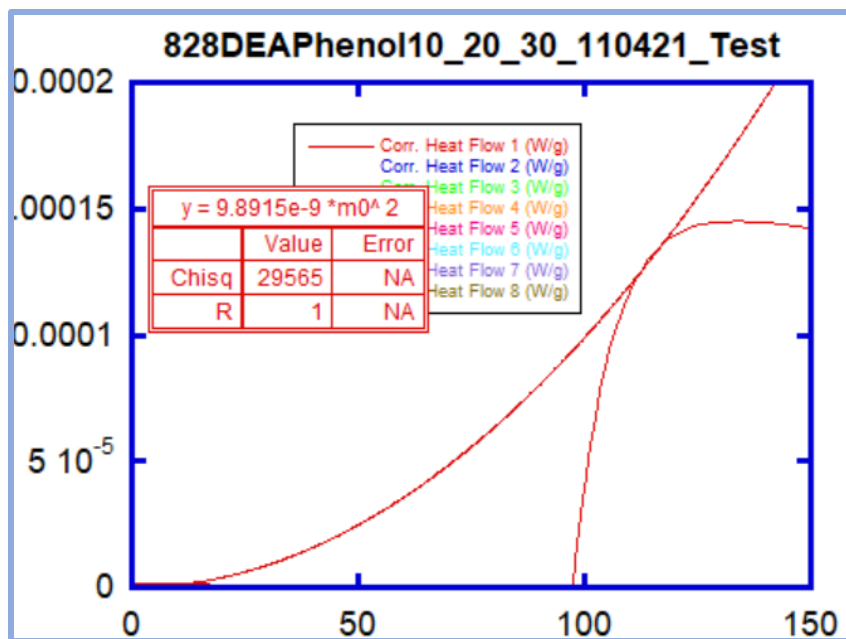
A curve like the one shown below should be generated:



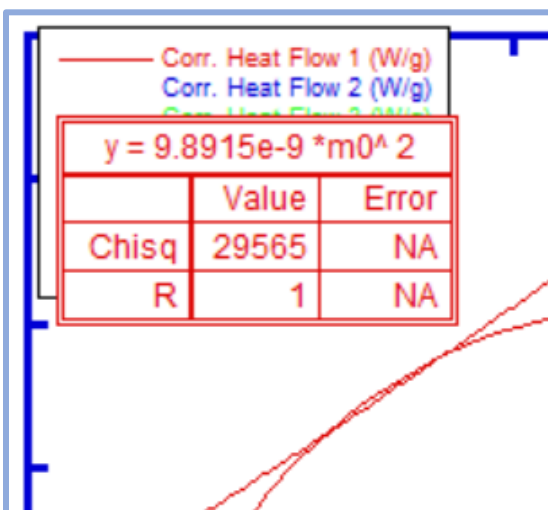
- Once again navigate to the Curve Fit Definition window. “Hardwire” the fit by replacing m1, m2, etc. with their decimal numbers (as determined by the fitting software in the previous step).



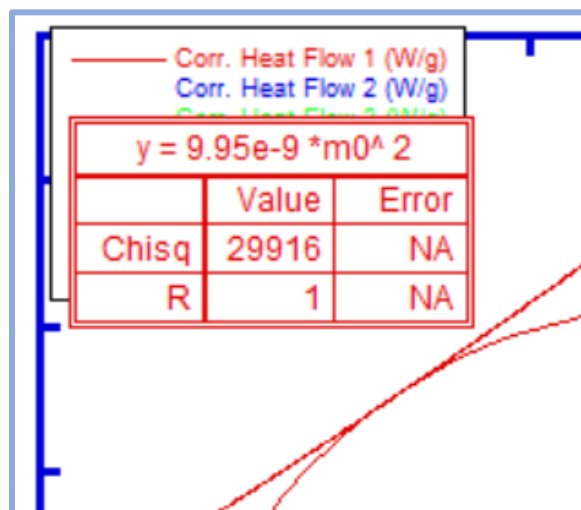
You may now unmask the data by double-clicking the Mask tool. The fit will no longer be affected by the data:



7. If needed, manually adjust the fit equation until it connects to the heat curve nicely.

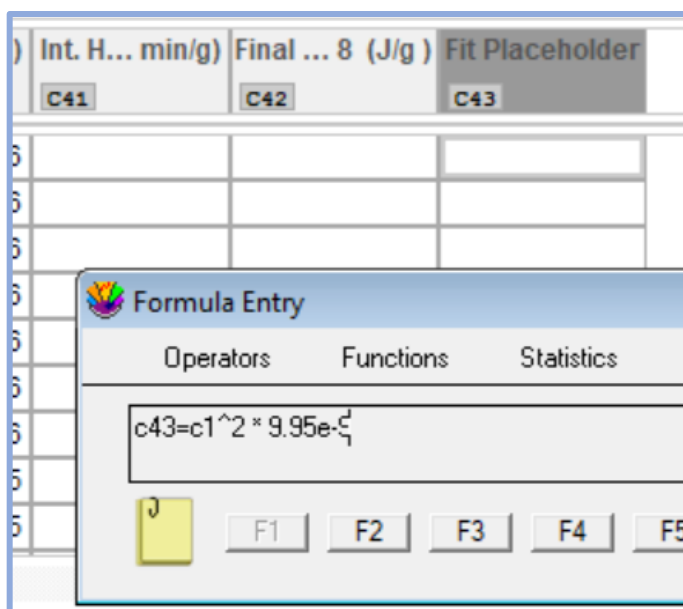


A bit off...



Just right!

8. Back in your data file, add a column to the far left. Generate the data for your short-time “graft” using the fit equation (as determined in the previous step).
- You may want to record the fit equation for each sample in the Read-Me file, so that analysis of data-splices is expedited (see Part E).

**COMMON ERROR...**

When generating your graft data, make sure you are using the correct time column for that sample.

KEEP IN MIND...

The first several entries of the graft data may be blank. This comes from when we removed those initial, negative time values.

9. Go back to your plot. Using the crosshair tool, approximate the time at which the short-time fit joins the heat curve. Here, they connect at around 114 minutes, or at row 115 in the data window.

	Time (s)	Time 1-4 (min)
	c0	c1
110	6650.5	108.84
111	6716.3	109.94
112	6778.9	110.98
113	6841.6	112.03
114	6915.0	113.25
115	6982.0	114.37
116	7049.0	115.48

10. In the data window, copy the short-time graft data from row 0 to row n (the row containing the time at which the graft and curve join). The initial rows may be blank; copy them anyways.

	Int. H... min/g)	Final ... 8 (J/g)	Fit Placeholder
	C41	C42	C43
110			0.00011787
111			0.00012026
112			0.00012255
113			0.00012487
114			0.00012761
115			0.00013014
116			0.00013270
117			0.00013531

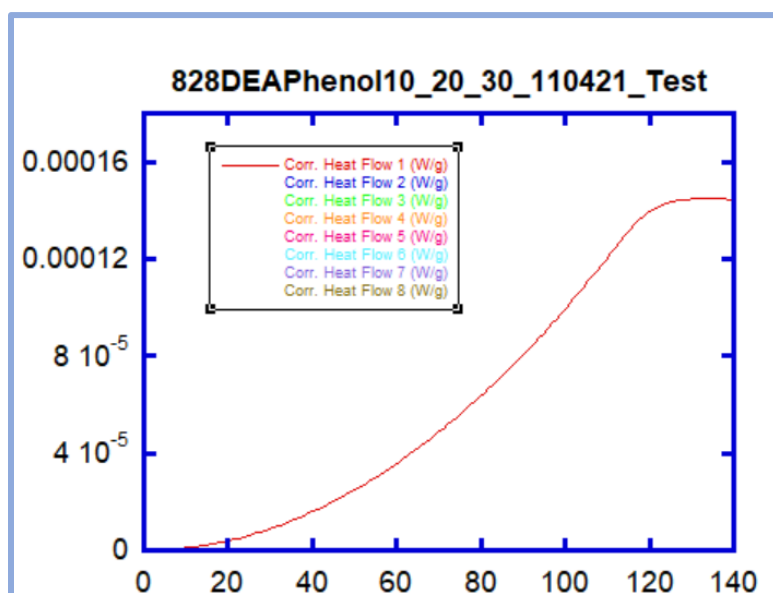
11. Highlight the same portion of the “Corr. Heat Flow (W/g)” data and paste the graft data.

	Heat Flow 1 (W/g)	Corr. Heat Flow 1 (W/g)	Int. Heat 1 (W mi
	co	ci	
112	0.00012072	0.00012255	
113	0.00012380	0.00012487	
114	0.00012741	0.00012761	
115	0.00013020		
116	0.00013247		
117	0.00013477		
118	0.00013648		
119	0.00013760		

COMMON ERROR...

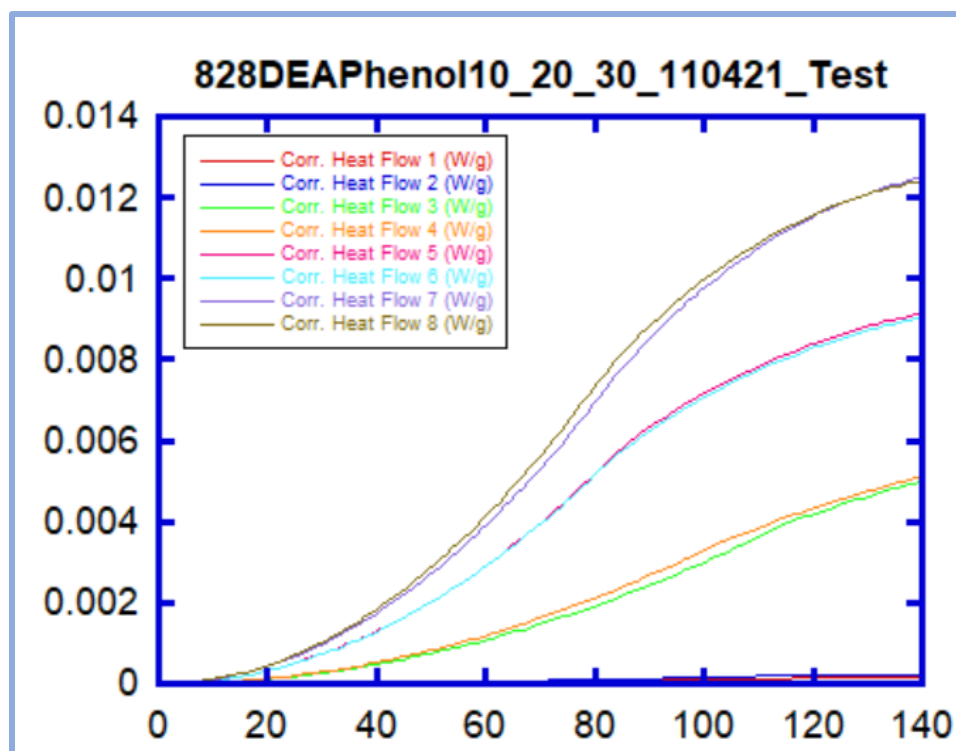
Do not simply paste the graft data here without highlighting the rows. This would merely shift down the data we are trying to overwrite.

12. Refresh the plot window. The heat curve should now slope to (0,0).



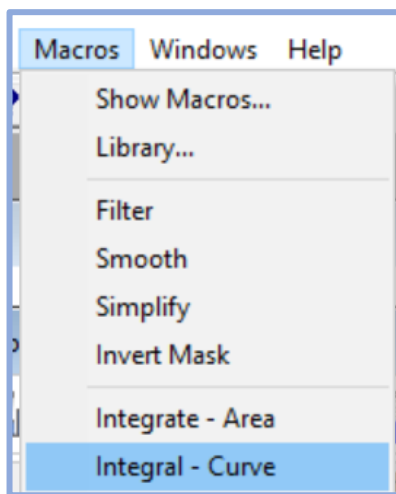
After one curve has been adjusted, blank it out in Variable Settings and move onto the next curve.

The plot of a short-time-corrected dataset is shown below:

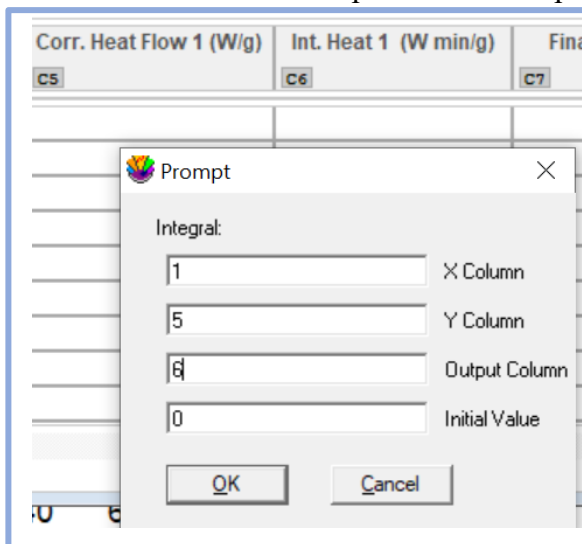


Part D: Heat Integration

1. Make sure you are clicked into the data file, and not the plot. Under Macros, pick “Integral-Curve”.



2. The window below will come up. For each sample input as follows:



- a. “X Column” will be the “Time #-# (min)” column number.
- b. “Y Column” will be the “Corr. Heat Flow” column number.
- c. “Output Column” will be the “Int. Heat” column number.
- d. “Initial Value” is 0.

WHY THIS IS IMPORTANT...

The integral of the Heat Flow is the Cumulative Heat of Reaction. We use the Cumulative Heat to determine the extent of the reaction at a given time.

COMMON ERROR...

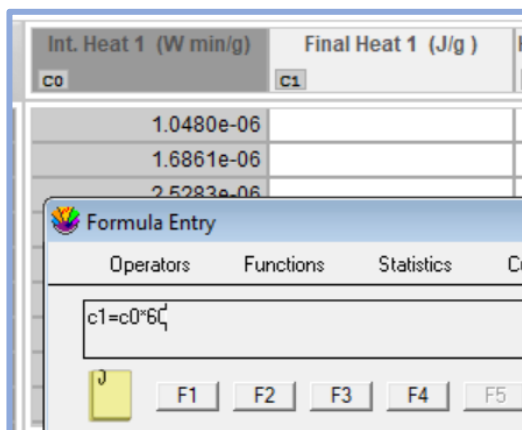
Make sure the **first column** of your data is C0 when integrating. If the C0 column is to the left of your time columns, you will not be able to locate them in the integration parameters.

Time 5-8 (min)	Heat Data 1 (W)
C0	
	2.1429e-05
	2.1429e-05

COMMON ERROR...

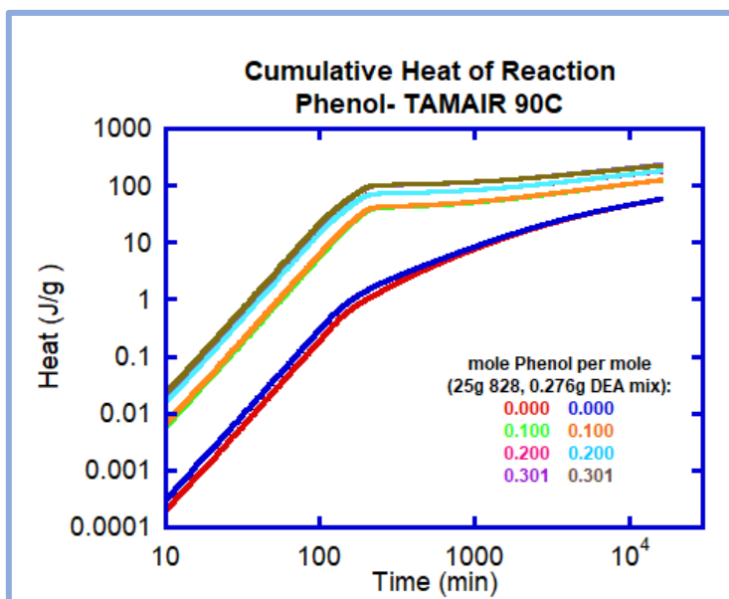
When generating your heat-integral data, make sure you are using the correct time column for that sample.

- For each sample, click into the “Int. Heat (W min/g)” column so that it is C0. Calculate “Final Heat (J/g)” by multiplying “Int. Heat” by 60 (this converts W*min to W*s, or J).

**KEEP IN MIND...**

The first several entries of the heat-integral data may be blank. This comes from when we removed those initial, negative time values.

- Plot “Final Heat (J/g)” vs “Time ## (min)” for each sample. Make the axes Log-Log, and format the plot according to the McCoy lab standards.

**KEEP IN MIND...**

Instead of a legend, a helpful text indication of the relevant properties of each sample may be better. Here, we indicate the Phenol fraction of each line.

- Send this plot to Dr. McCoy, along with the Read-Me document. Determine whether the samples should be left in the TAM-Air for longer, or if a new run should be planned.

Part E: Data Splicing

1. Open your experiment's Master-File.
2. Navigate to the very last rows of the dataset Master-File and delete the zeroes at the tail-end of the data. Make a "clean cut" (at the same row across all samples), even if you chop off some real data.

WHY THIS IS IMPORTANT...

As a general rule, an experiment's data should be exported and analyzed every two weeks. Data outside of that two-week window will be low-resolution, as it is "thinned out" to save bytes. This is why a new set of exported data will be analyzed and then "spliced" into a master-file every two weeks.

	Time	Heat ..., 517:1]	Heat ..., 517:2]	Heat ..., 517:3]
7	9.7554e+05	0.00037873	0.00028240	0.0005670
8	9.7554e+05	0.00037869	0.00028241	0.0005669
9	9.7555e+05	0.00037866	0.00028243	0.0005669
0	9.7555e+05	0.00037862	0.00028244	0.0005669
1	9.7555e+05	0.00037861	0.00028244	0.0005669
2	9.7555e+05	0.0000	0.00028244	0.0005669
3	9.7555e+05	0.0000	0.0000	0.0005669
4	9.7555e+05	0.0000	0.0000	0.0000
5	9.7555e+05			0.0000
6	9.7555e+05			0.0000

3. Locate the 'Time (s)' value where the data ends on the Master-file. (Here it is 9.7555e+5.)
4. Open your most recent export datafile. Copy all columns of the export's data from that Time(s) value (determined in the previous step) down to the end. (Here the nearest available value is 9.7559e+5)

COMMON ERROR...

Record the **Time value** and not the row number. The row locations of datapoints are not preserved between exports.

	Time (s)	Heat ...I, 517:1]	Heat ...I, 517:2]	Heat ...I, 517:3]
	C0	C1	C2	C3
6489	9.7517e+05	16253	16239	0.0003902
6490	9.7530e+05	16255	16241	0.0003902
6491	9.7543e+05	16257	16243	0.0003902
6492	9.7559e+05	16260	16246	0.0003903
6493	9.7575e+05	16262	16248	0.0003904
6494	9.7590e+05			0.0003904
6495	9.7606e+05			0.0003904
6496	9.7620e+05			0.0003904
6497	9.7634e+05			0.0003903
6498				

- Paste the copied export data into the Master-file.

	Time	Heat ...I, 517:1]	Heat ...I, 517:2]
16257	9.7555e+05	0.0000	0.0000
16258	9.7555e+05	0.0000	0.0000
16259			
16260			
16261			
16262			
16263			

You now have a block of continuous data that preserves the resolution of the short-time points, but extends for many weeks of long-time.

- Save the Master-file. Duplicate it, and indicate the export date in the name of the duplicate file. **You will perform data analysis on this duplicate export file, and not on the Master-file.**
- Proceed to step 8 of Part A. Perform all steps of the data analysis.

KEEP IN MIND...

You may have recorded long-time shifting constants for Part B from a past analysis. These will be good starting points, but are subject to change.

If the long-time shifts change, then any short-time fit equations may also need adjustment.