The Goal:

Predict how long it takes an epoxy to cure at a given temperature.

The Problem:

Can’t test it directly.

The Problem:

In the process of heating the sample to the target temperature,

The cure advances at intermediate temperatures.

The Solution:

"Isoconversional" techniques.

Isoconversional:

The rate that the cure progresses depends only on:

(a) The current temperature and
(b) The current degree of cure (0% - 100%)

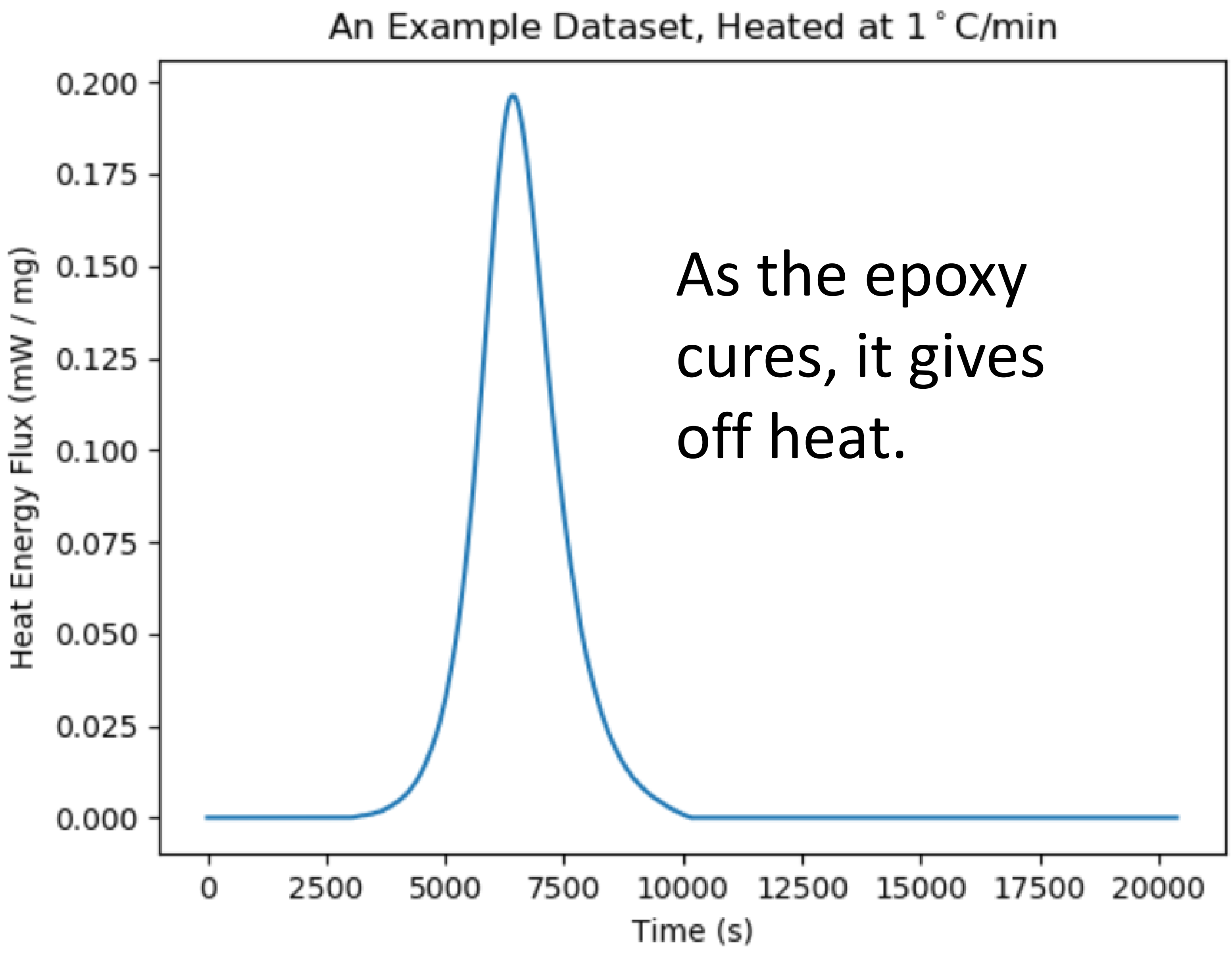
The Plan:

Heat the epoxy at a controlled temperature ramp.

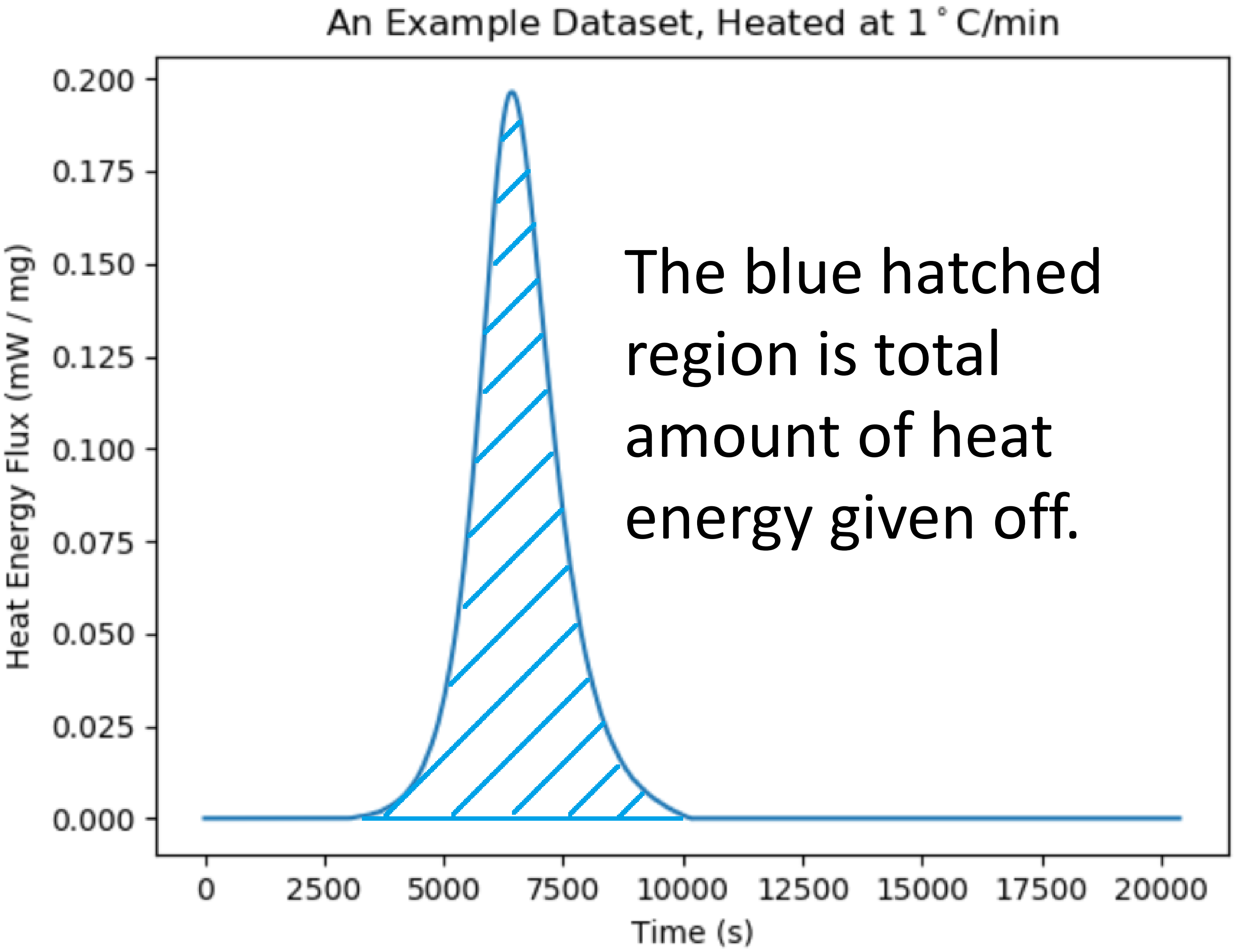
Record the temperature and information about cure.

The Data:

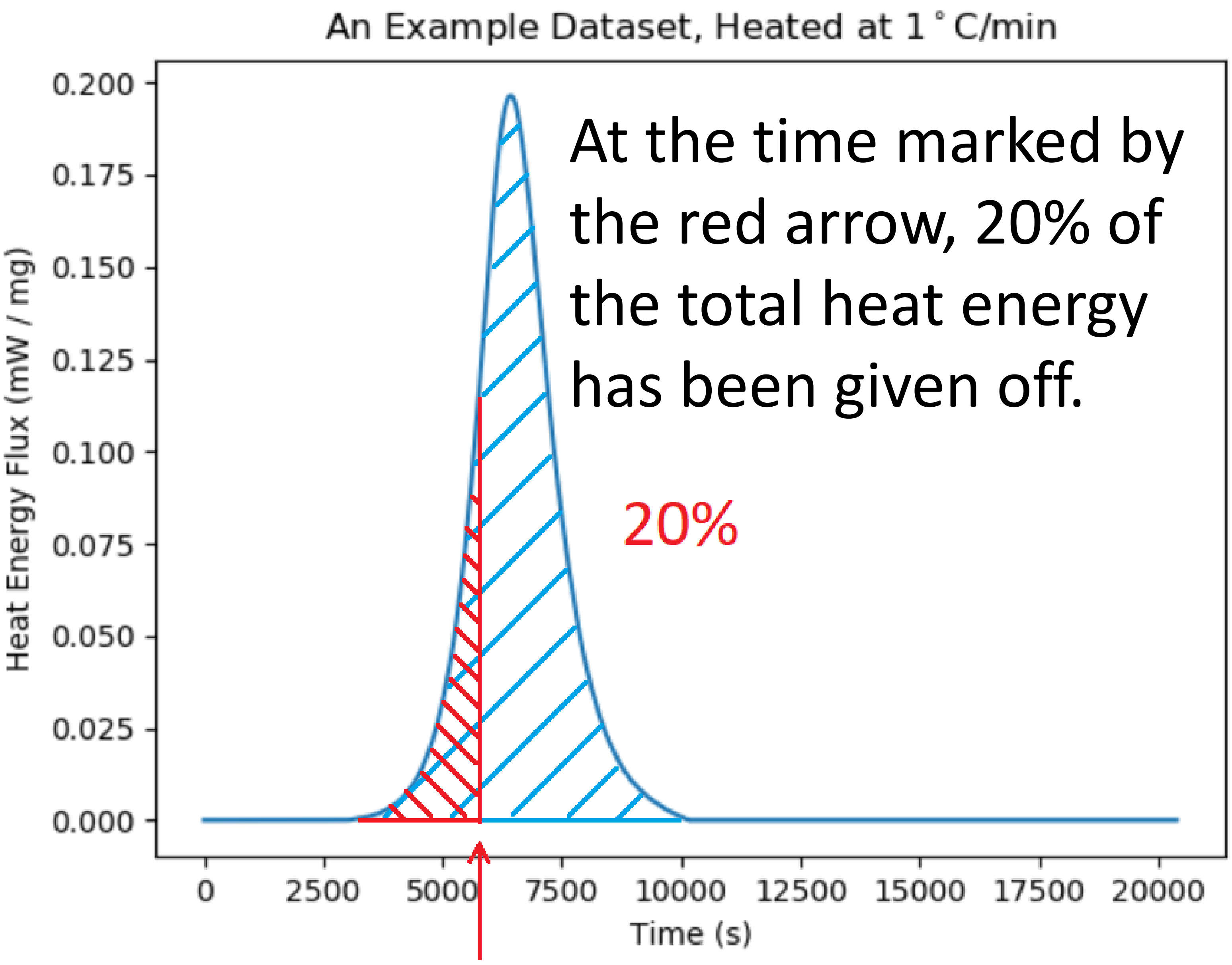
The following slides show pre-processing of the data.

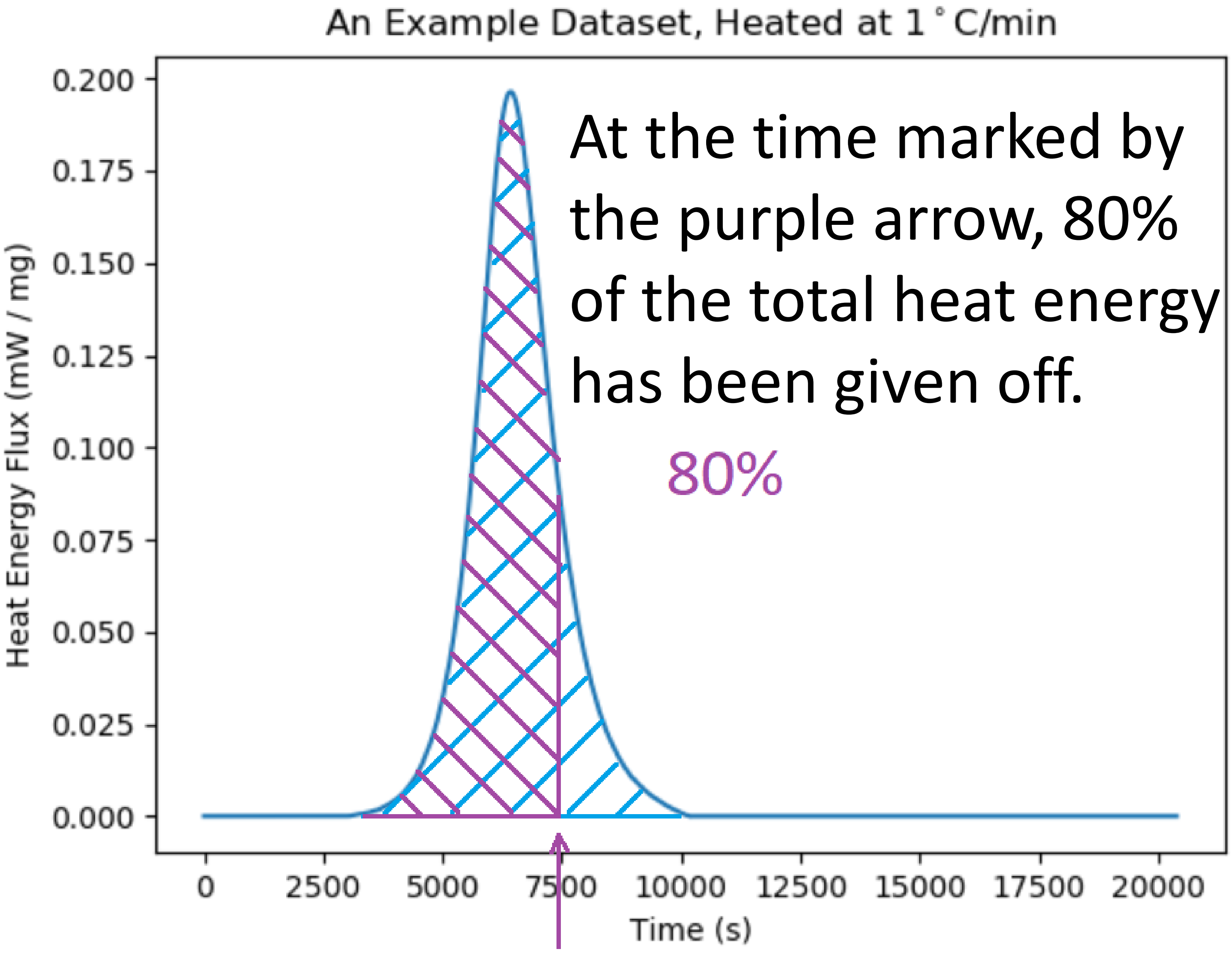


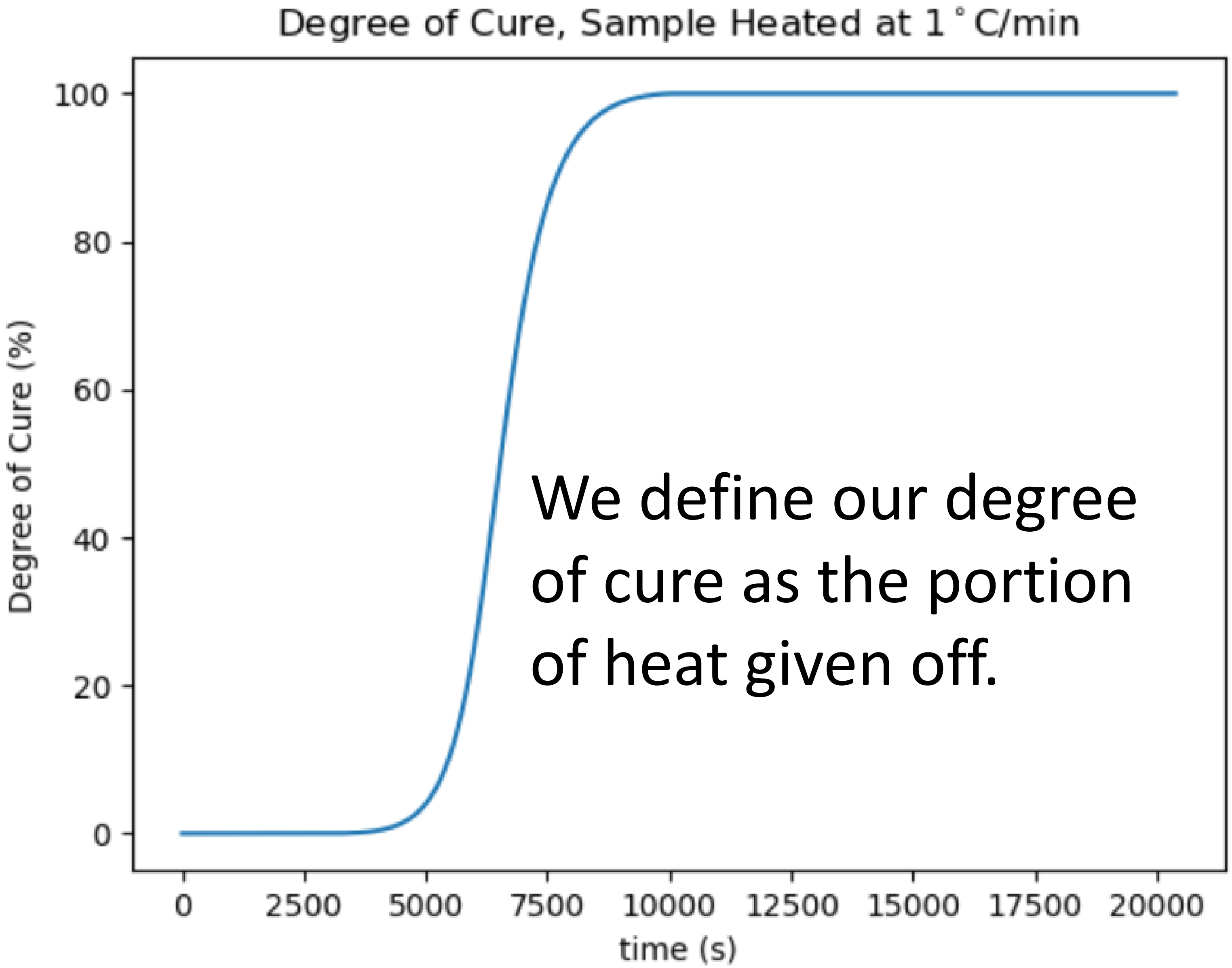
As the epoxy cures, it gives off heat.



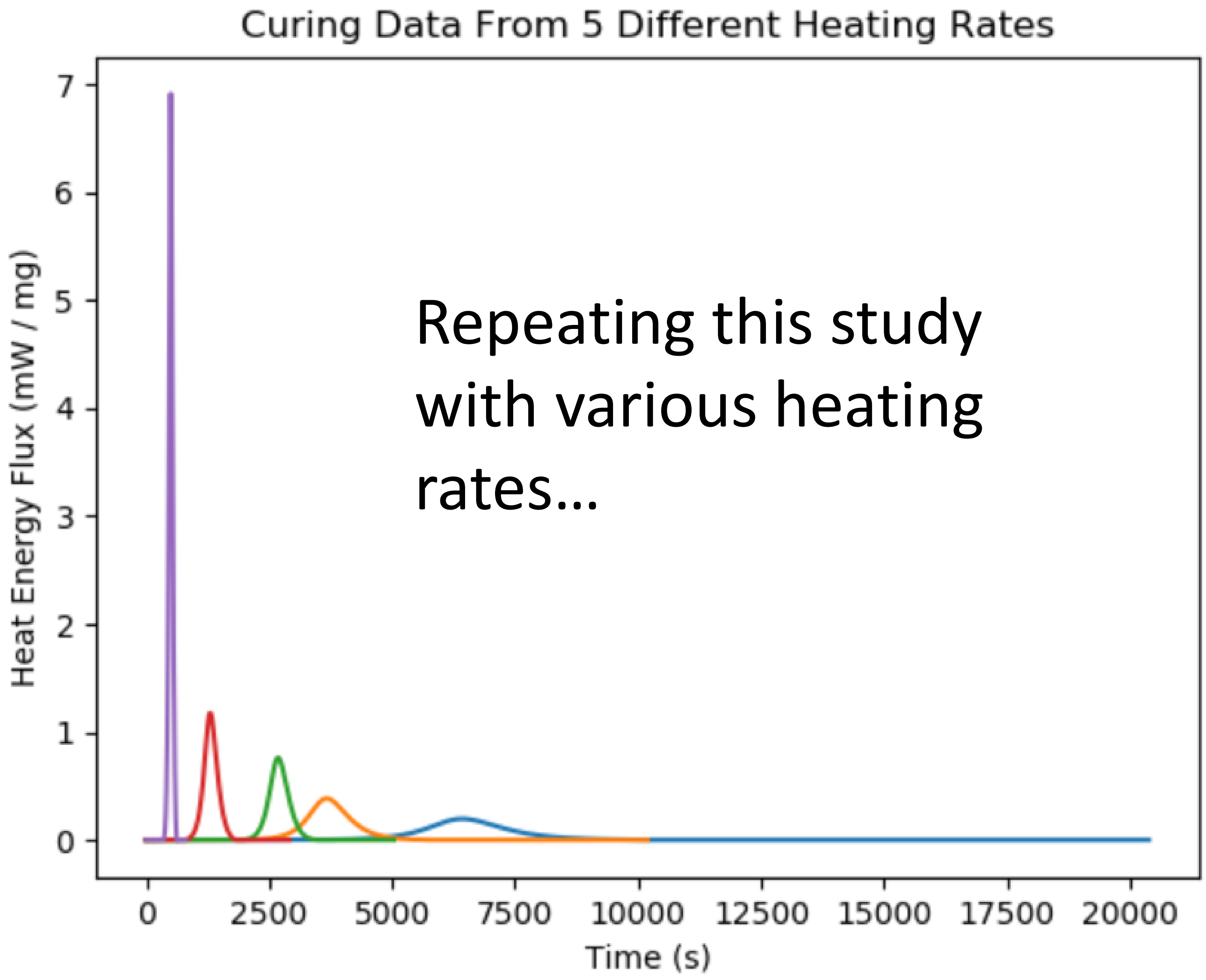
The blue hatched region is total amount of heat energy given off.


An Example Dataset, Heated at 1 °C/min
Heat Energy Flux (mW / mg)
0.200
0.175
0.150
0.125
0.100
0.075
0.050
0.025
0.000
0
2500
5000
7500
10000
12500
15000
17500
20000
Time (s)
At the time marked by the red arrow, 20% of the total heat energy has been given off.
20%

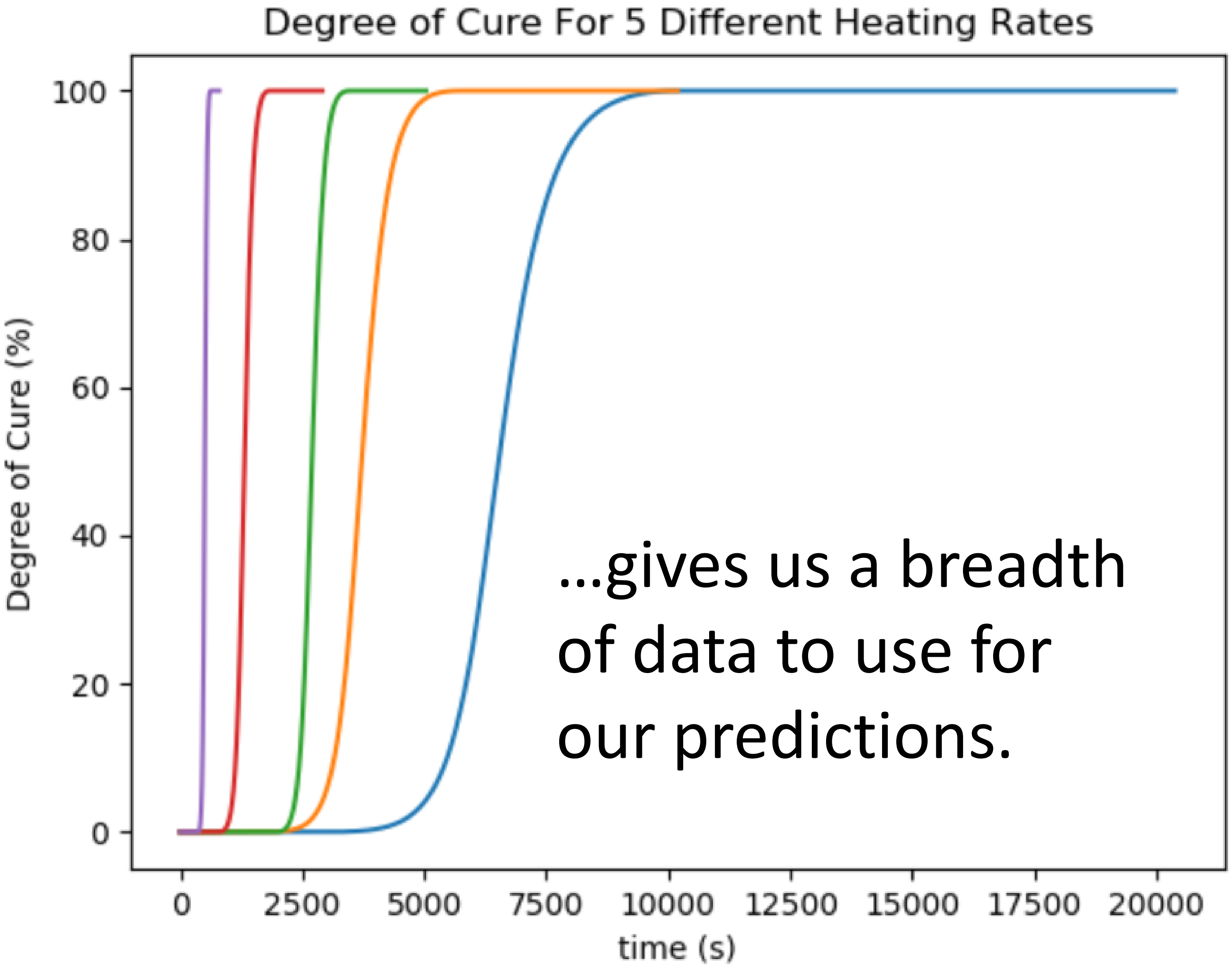

An Example Dataset, Heated at 1 ° C/min
At the time marked by the purple arrow, 80% of the total heat energy has been given off.
80%
Heat Energy Flux (mW / mg)
Time (s)
0.200
0.175
0.150
0.125
0.100
0.075
0.050
0.025
0.000
0
2500
5000
7500
10000
12500
15000
17500
20000



We define our degree of cure as the portion of heat given off.



Repeating this study with various heating rates…



…gives us a breadth of data to use for our predictions.

The Math:

At the end of the day, we want the time it takes to reach a degree of cure.

The Math:

$$t_\alpha = [\exp(-E_\alpha / RT_{iso})]^{-1} \sum_{0}^{\alpha} \int_{t_{\alpha-\Delta\alpha}}^{t_\alpha} \exp(-E_\alpha / RT(t))dt$$

(Please forgive the calculus)

The Math:

$$t_\alpha = \sum_0^\alpha [\exp(-E_\alpha/RT_{iso})]^{-1} \int_{t_{\alpha-\Delta\alpha}}^{t_\alpha} \exp(-E_\alpha/RT(t))dt$$

Time to reach α

Gas constant

Isothermal temperature
For prediction

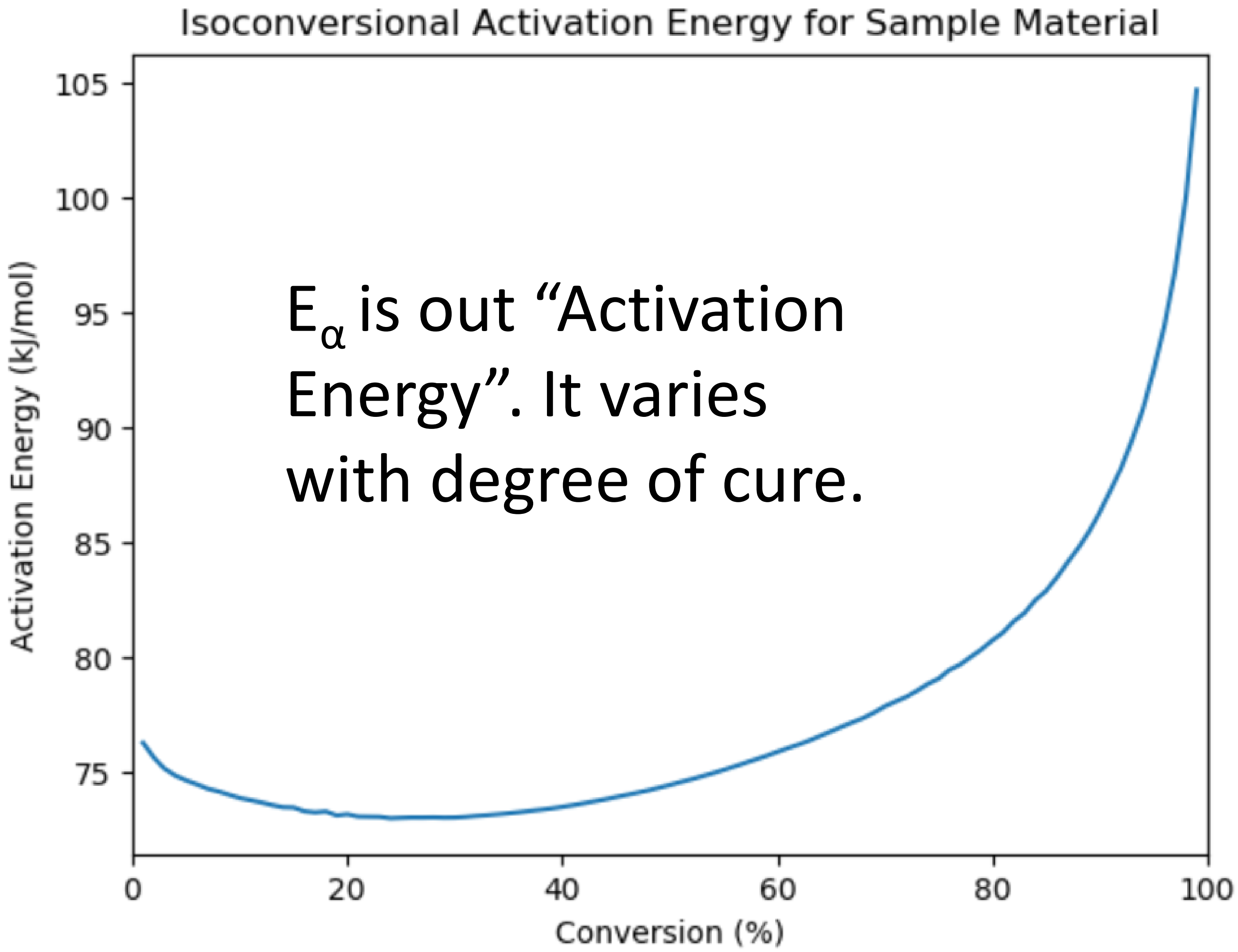
Temperature and
time from
experimental data

Something else altogether… we need to calculate this.

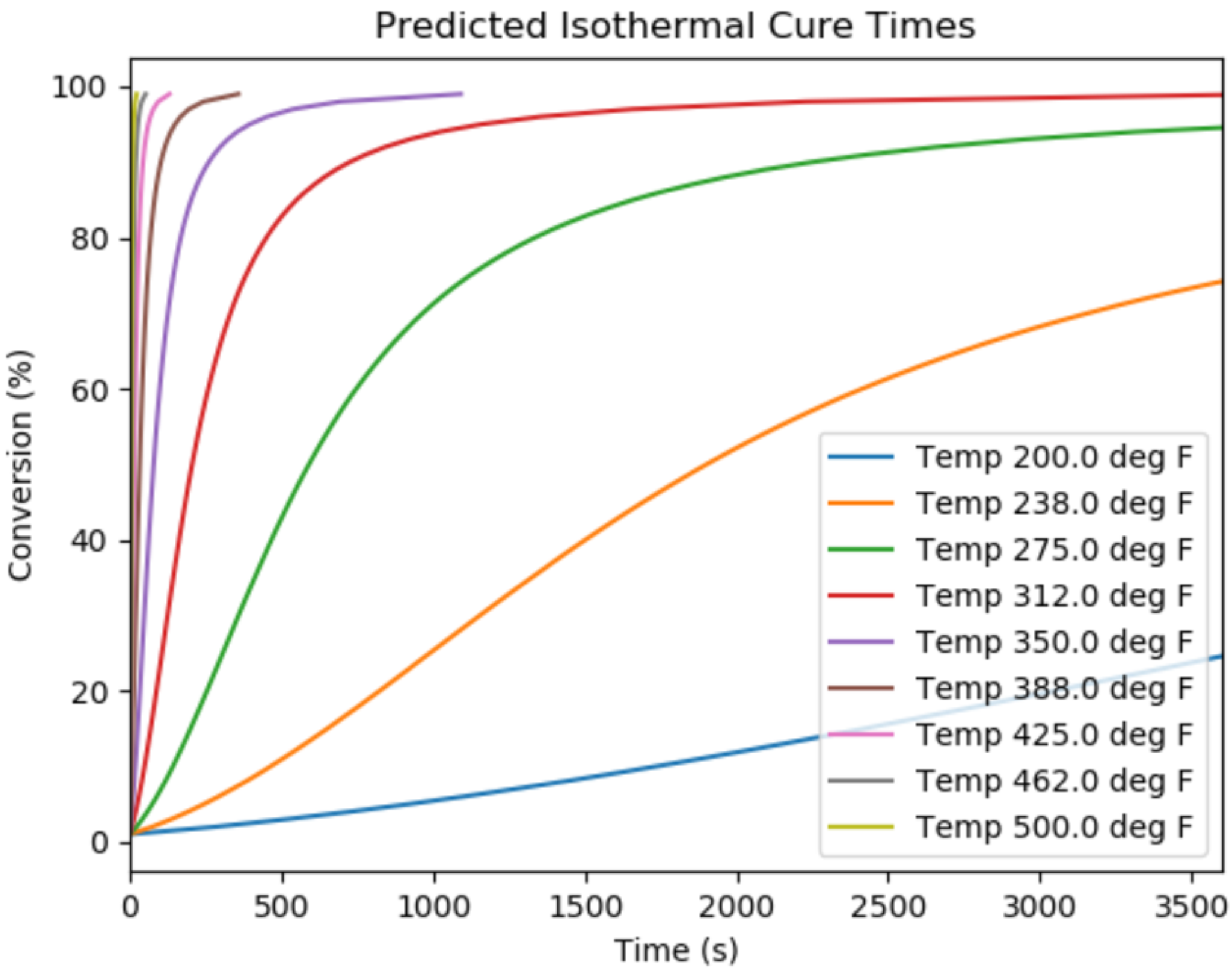
The Math:

$$\Phi(E_\alpha) = \sum_i \sum_{j \neq i} \frac{\int_{t_{\alpha-\Delta\alpha}}^{t_\alpha} \exp(-E_\alpha/RT_i(t_\alpha))dt}{\int_{t_{\alpha-\Delta\alpha}}^{t_\alpha} \exp(-E_\alpha/RT_j(t_\alpha))dt}$$

We'll calculate E_α by minimizing this cost function.
(Find the value of E_α for which ϕ has the lowest value.)



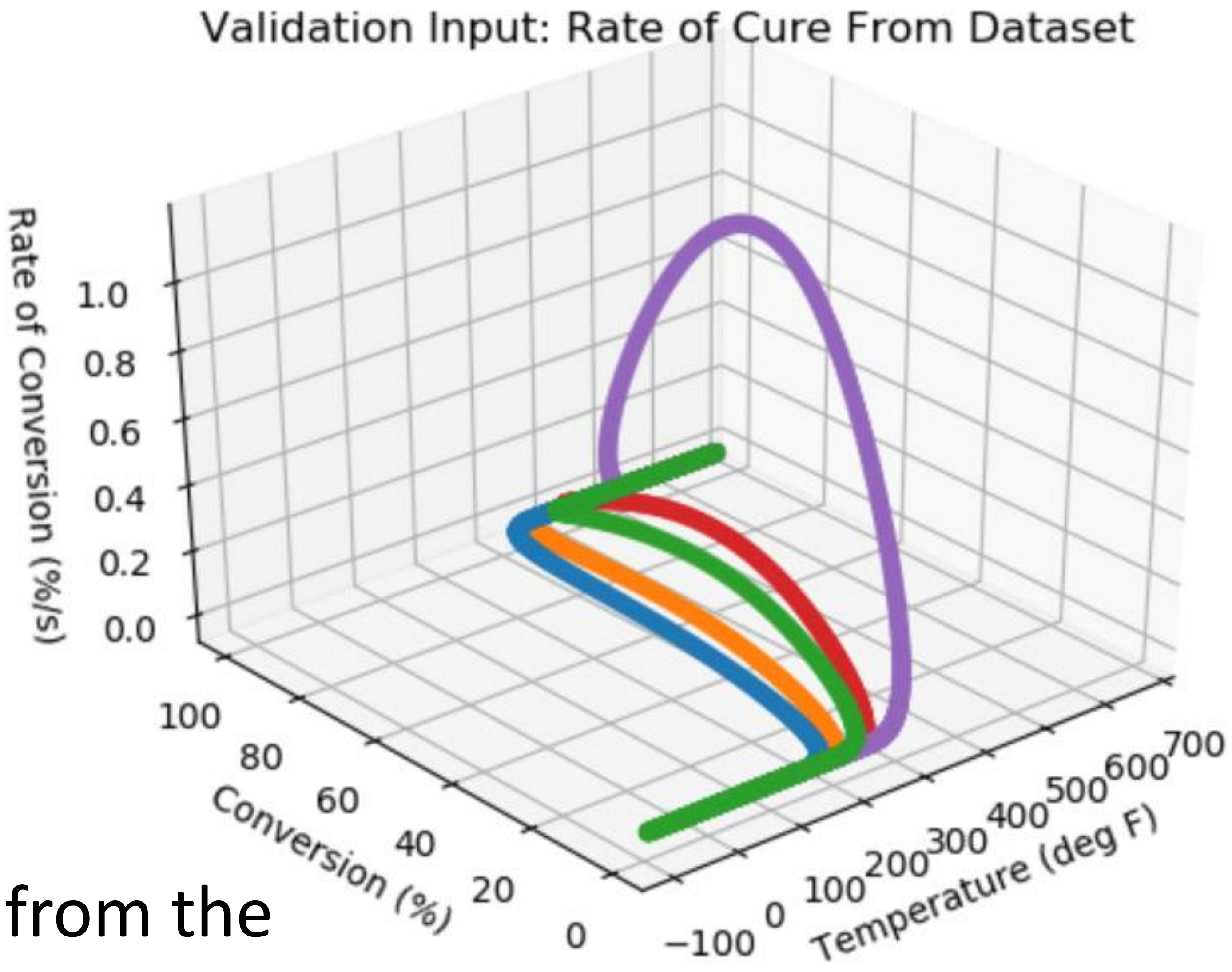
E_α is out “Activation Energy”. It varies with degree of cure.



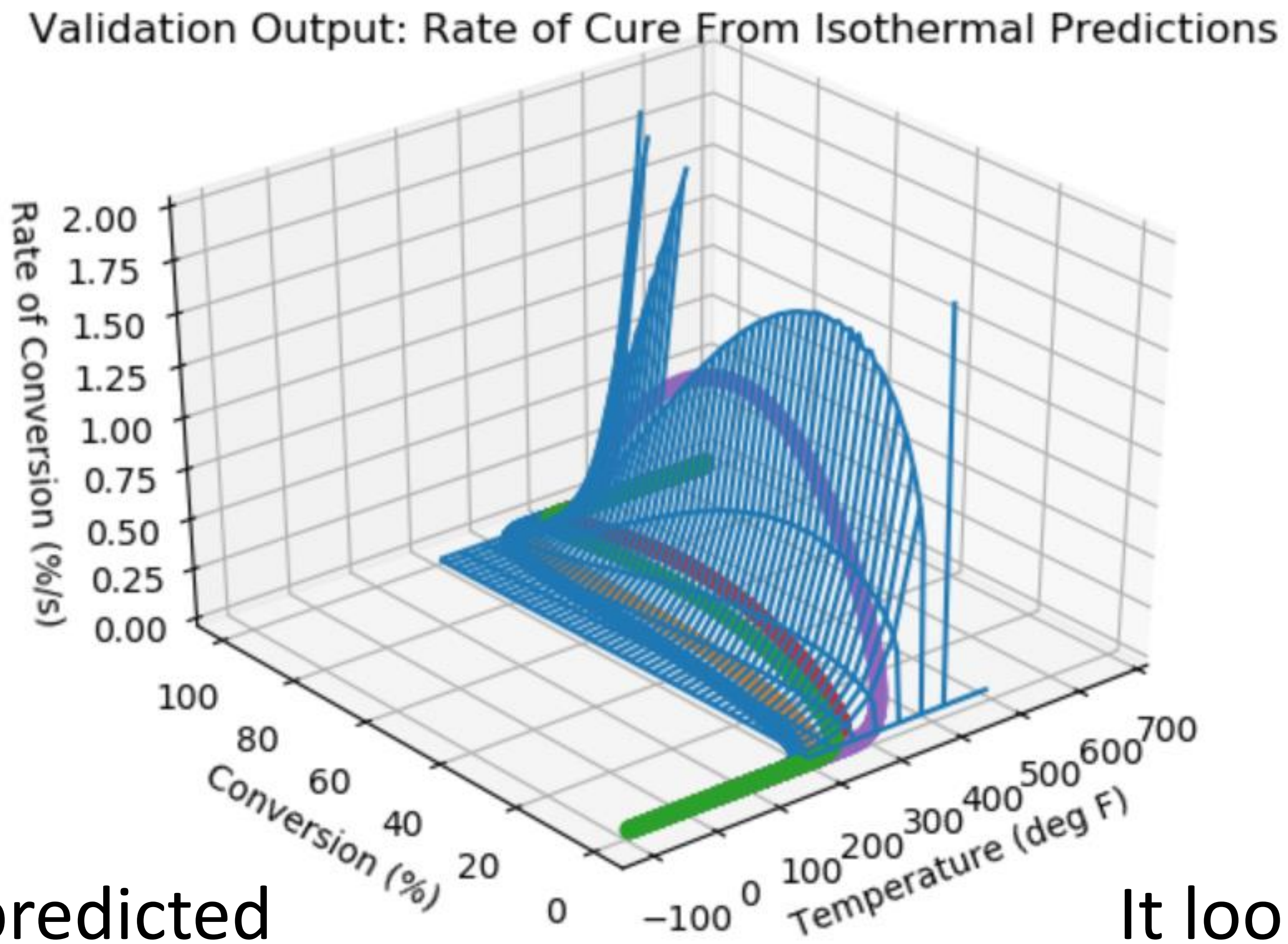
Now we can predict cure time for any isothermal temperature!

Validation:

…but is it right?



This is data from the experimental runs.



Now with predicted results on top.

It looks good!

Reference:

Sbirrazzuoli, Nicolas, et al. "Integral, differential and advanced isoconversional methods: complex mechanisms and isothermal predicted conversion–time curves." *Chemometrics and Intelligent Laboratory Systems* 96.2 (2009): 219-226.

Questions?

?

?

?

?

?