Design of Experiments

Design of experiments

Lecture 4

- Introduction to DoE and factorial design at two levels (Chapter 4)

Workshop 4

- Full factorial design at two levels

Lecture 5

- Block design and fractional factorial design (Chapter 5)

Workshop 5

- Fractional design

Lecture 6

- Optimisation (Chapter 6)

Laboration on optimization (or? Suggestions?)

After this part of the course, you will be able to

“Use basic techniques in the design of experiments for screening, and empirical or mechanistic model building”

“Use experimental design to maximize experimental output and for optimization”

What is experimental design?

“Scientific phenomena are commonly investigated through experiments where we change *one variable at a time (OVAT)”*

- *Change one variable*
- *Keep everything else constant (to the best of our ability)*
- *See how the results change*

Advantages… Disadvatages…

- OVAT is favored by non-experts, especially in situations where the data is cheap and abundant.
- There exist cases where the **mental effort** required to conduct a complex multi-factor analysis **exceeds the effort** required to acquire extra data, in which case OVAT might make sense.
- Furthermore, some researchers have shown that OVAT can be more effective than fractional factorials under certain conditions (number of runs is limited, primary goal is to attain improvements in the system, and experimental error is not large compared to factor effects, which must be additive and independent of each other)

- OVAT requires more runs for the same precision in effect estimation
- OVAT cannot estimate interactions
- OVAT can **miss optimal settings of factors**

Example:

Optimize the yield of a chemical reaction… what possible factors will influence the yield?

- Concentration of Reagent 1
- Concentration of Catalyst
- Concentration of Catalyst Ligand
- Solvent
- Temperature
- Reaction Time

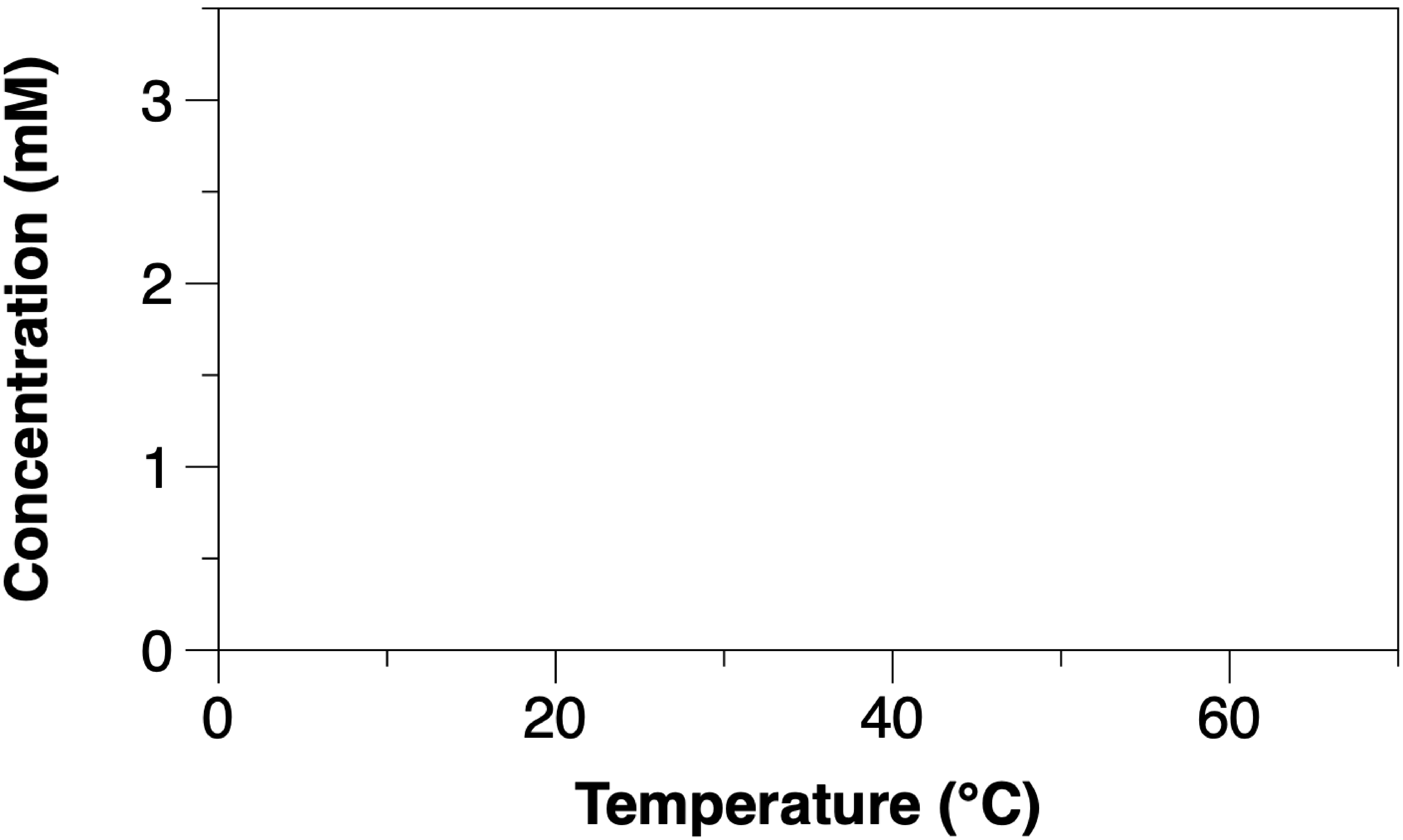
Any more?

Example:

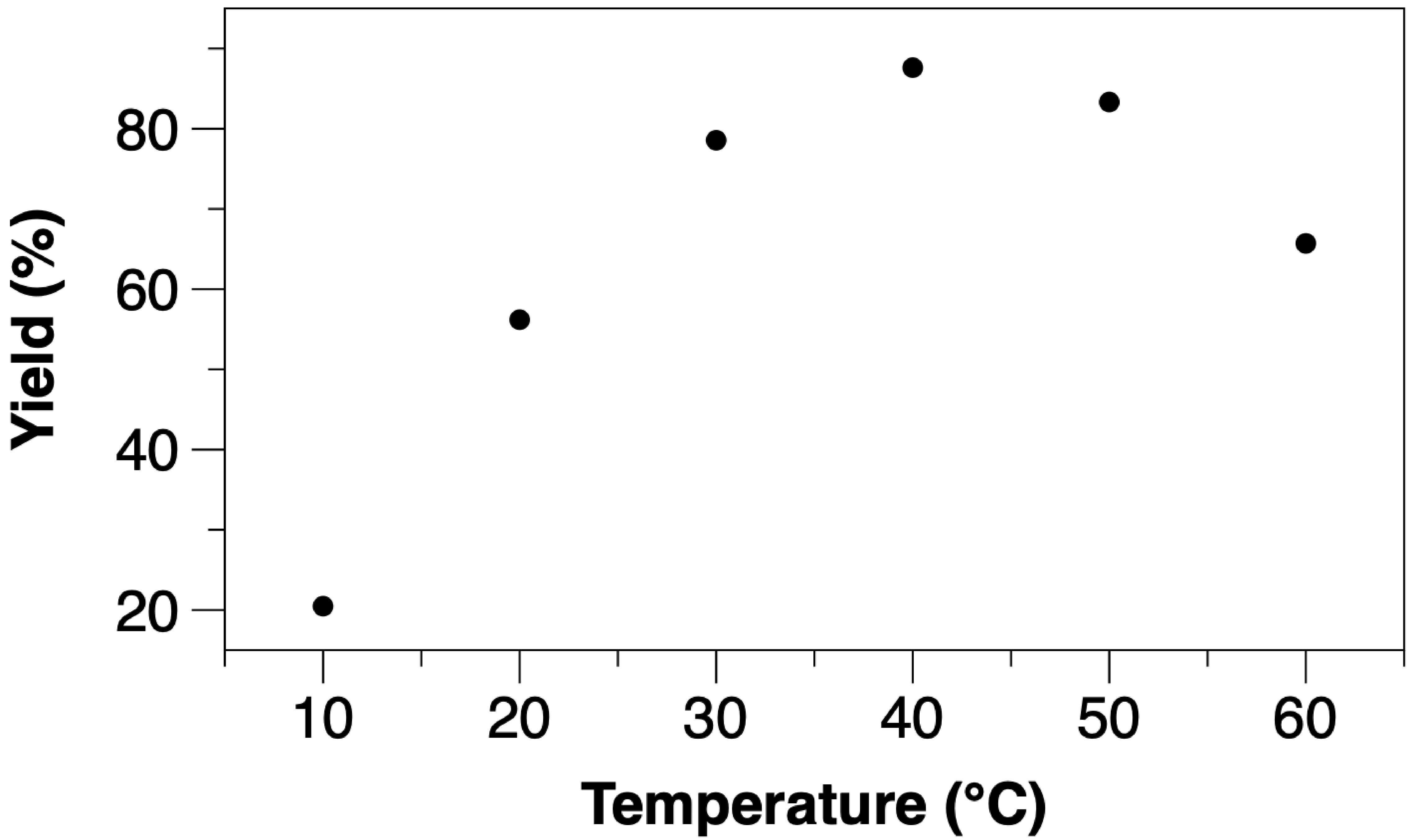
Optimize the yield of a chemical reaction… what possible factors will influence the yield?

- Concentration of Reagent 1
- **Concentration of Catalyst**
- Concentration of Catalyst Ligand
- Solvent
- **Temperature**
- Reaction Time

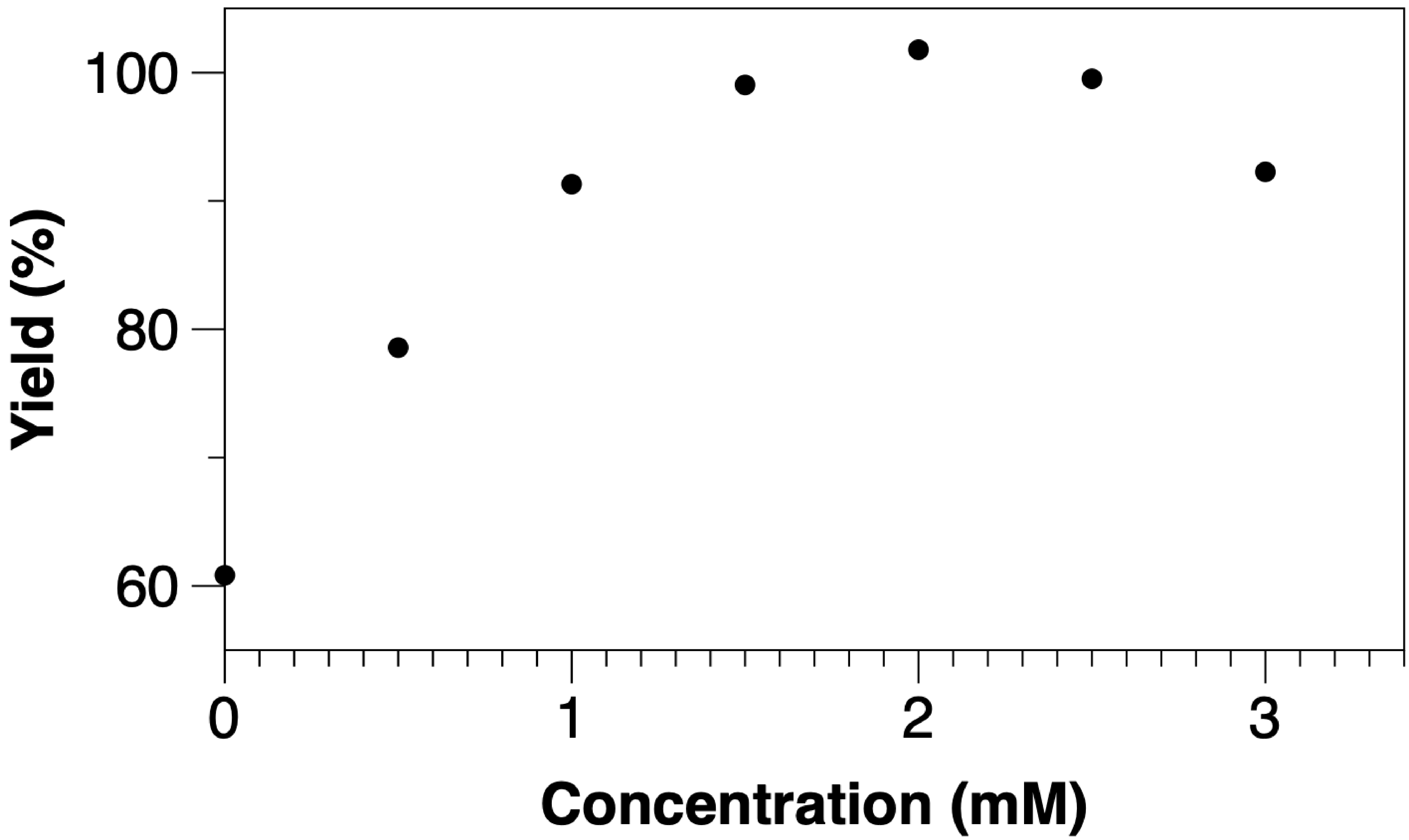
Any more?

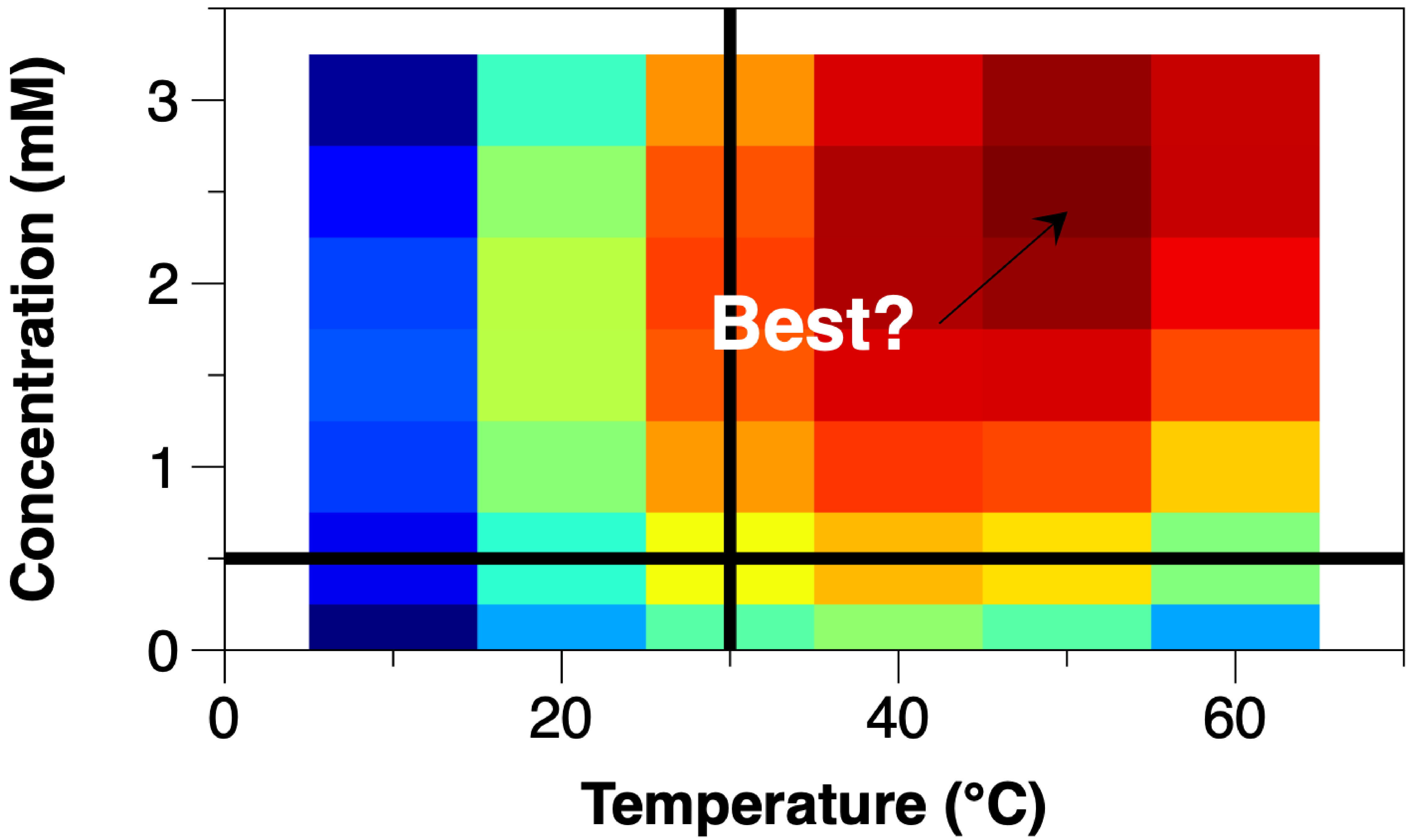


Concentration (mM)
3
2
1
0
0
20
40
60
Temperature (°C)



20
40
60
80
Yield (%)
10
20
30
40
50
60
Temperature (°C)


Yield (%)
100
80
60
0
1
2
3
Concentration (mM)

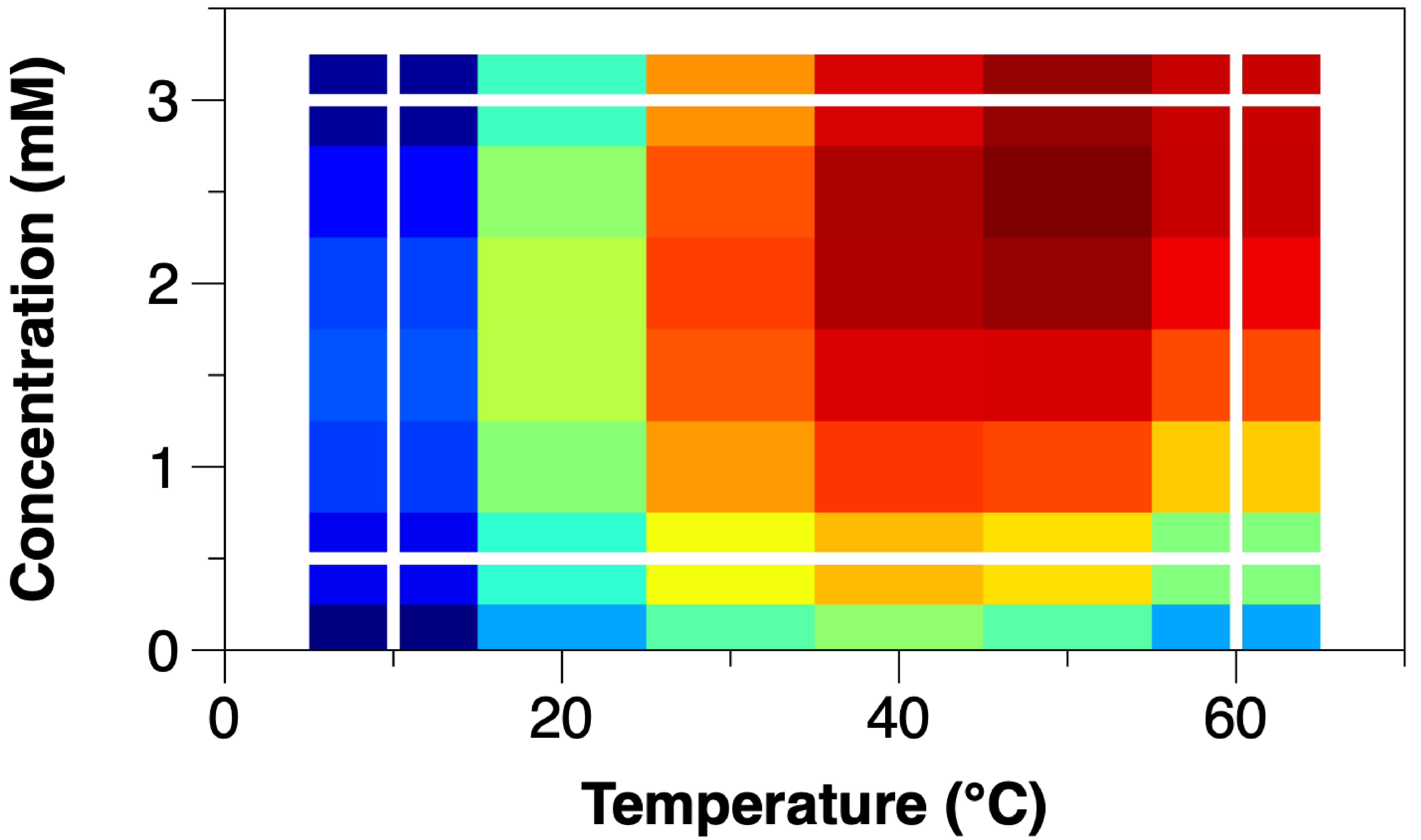


Concentration (mM)
3
2
1
0
Best?
0
20
40
60
Temperature (°C)

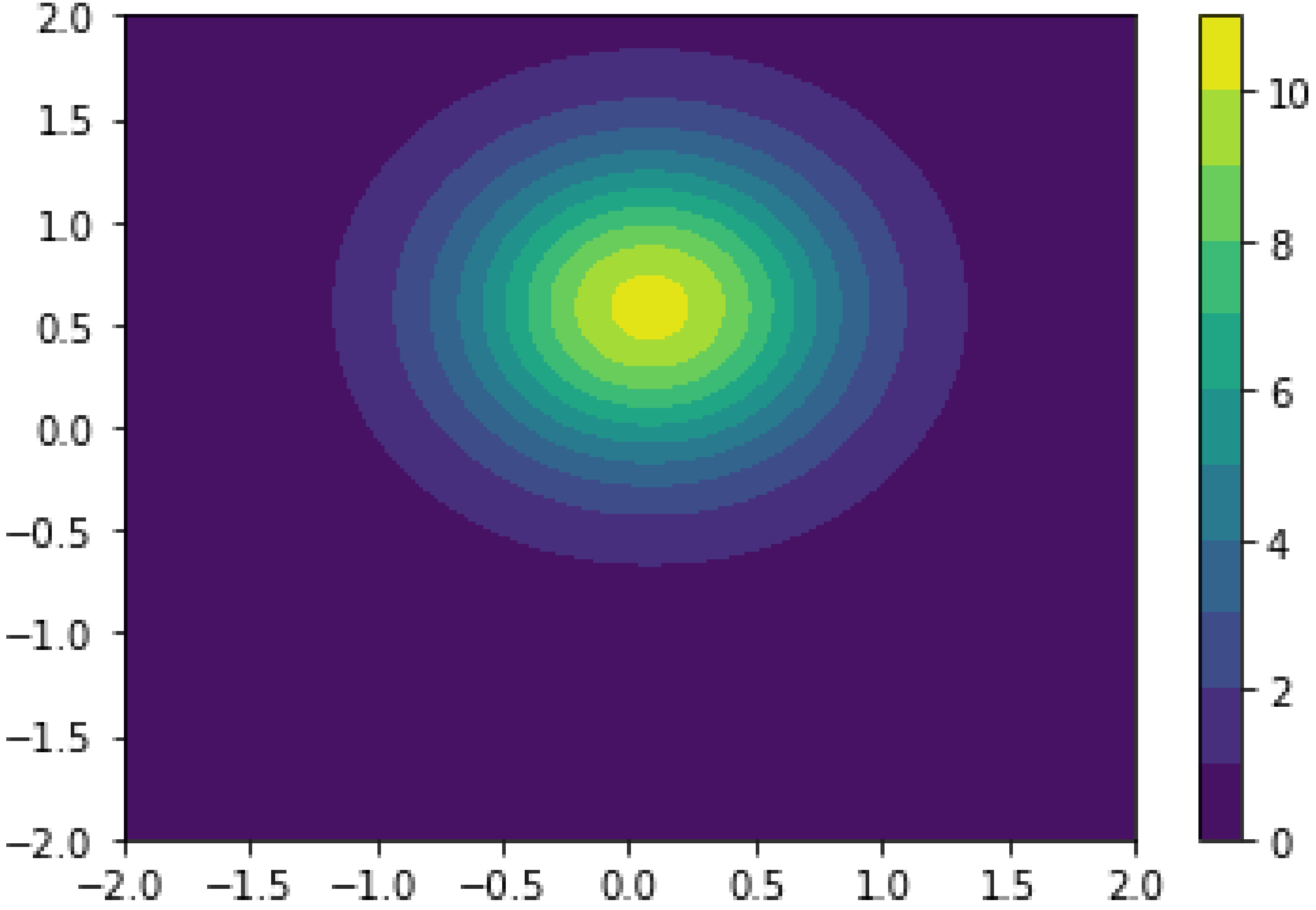
Why did we miss the optimal condition?

"OVAT generally misses interactions between otherwise independent factors in our experiments"

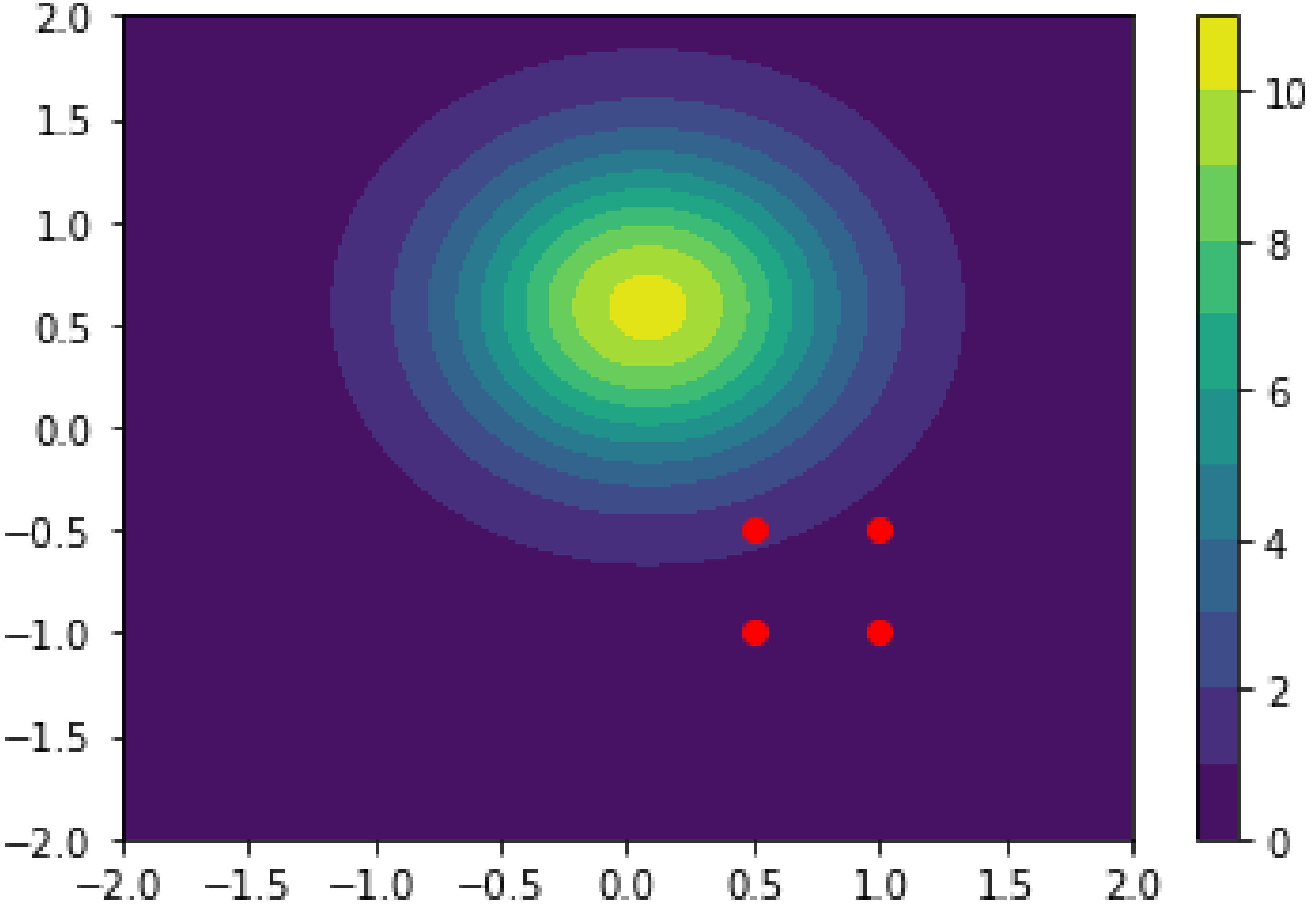
Instead of OVAT, we should have used "design of experiments"


Concentration (mM)
0
1
2
3
0
20
40
60
Temperature (°C)

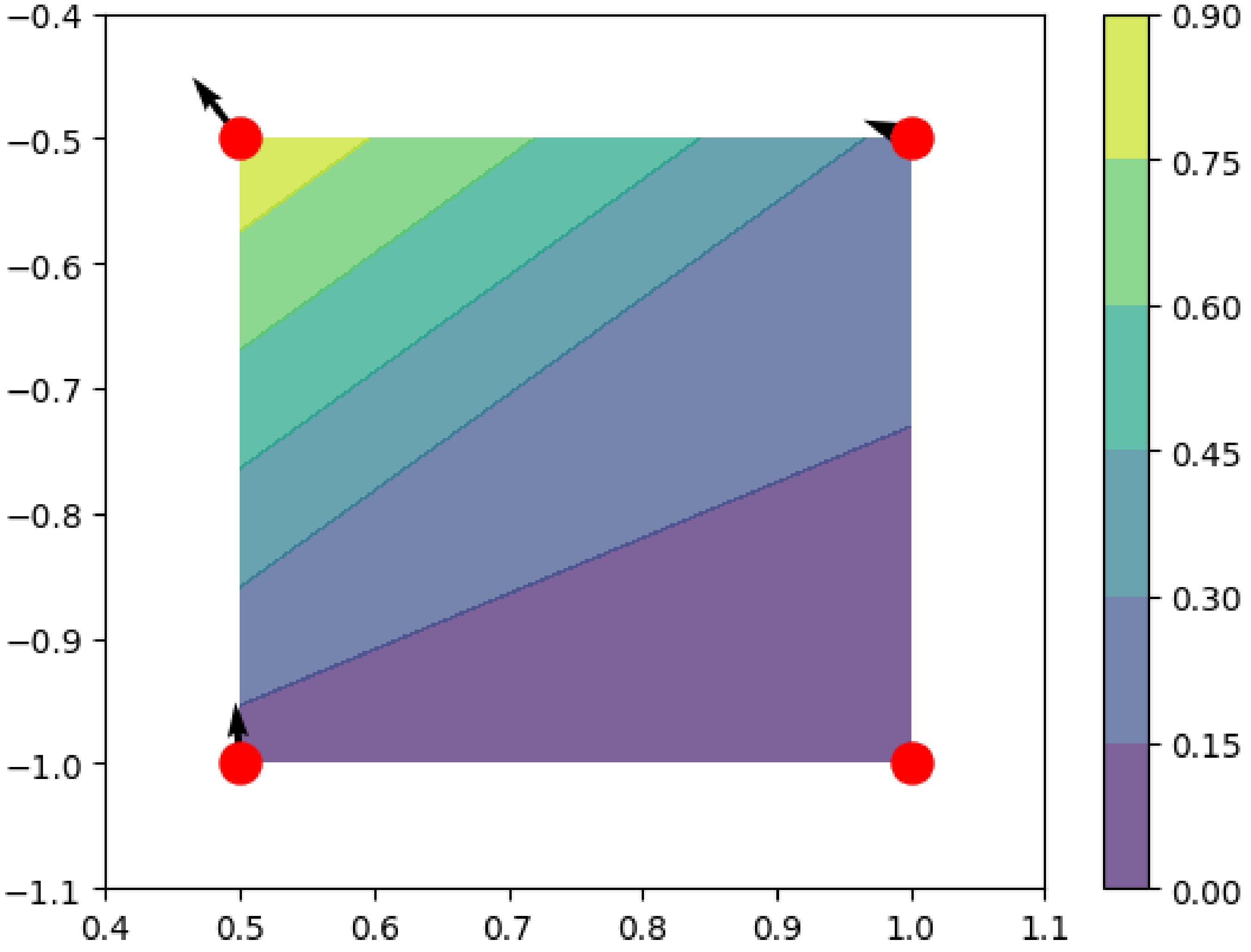
Iterative DoE



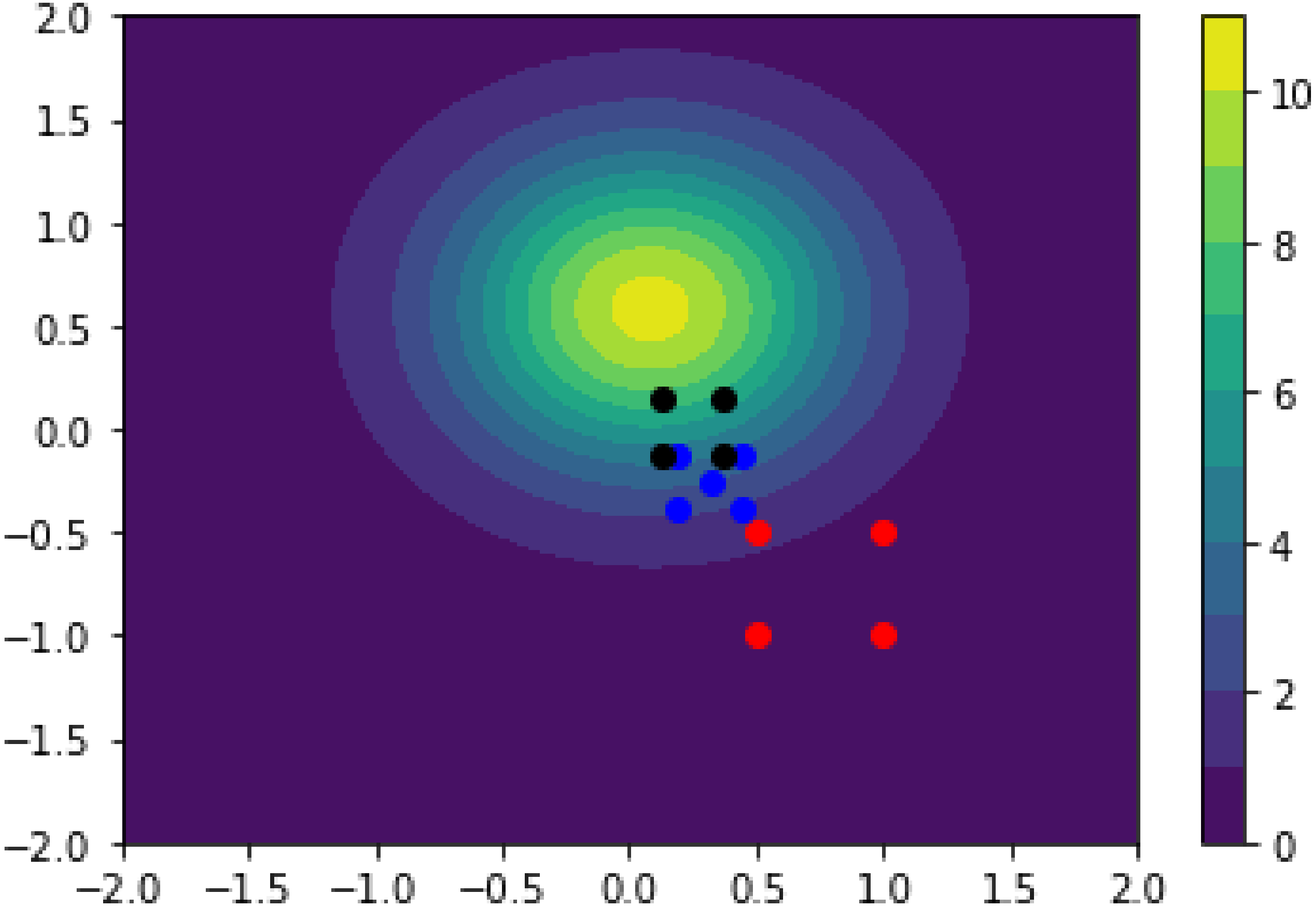
Iterative DoE



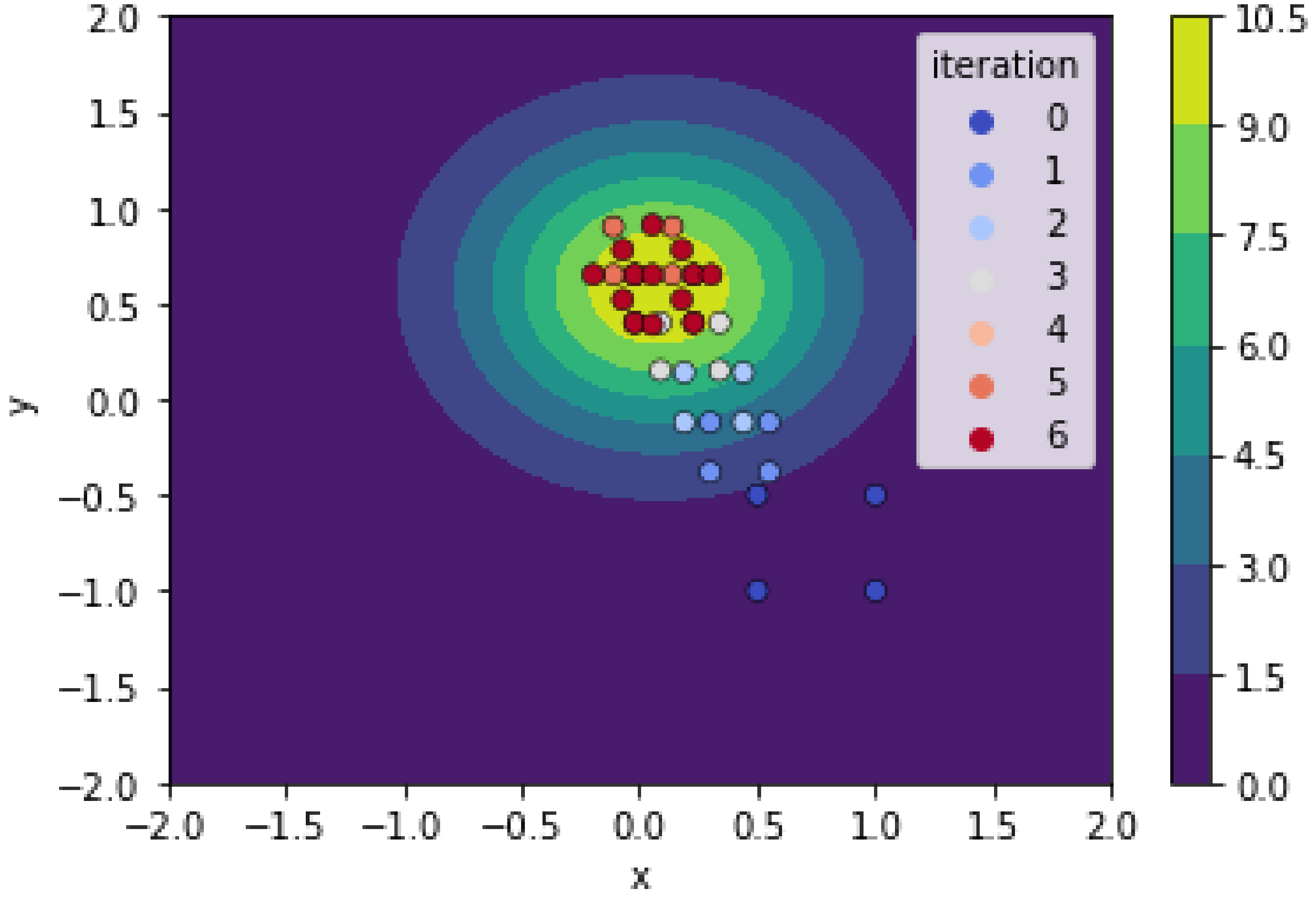
“Steepest ascent”



Iterative DoE



Finding the optimal factors



Different classes (stages) of experiments

- Screening (WHICH Stage)
  - aiming to determine the subset of important parameters from a given larger set of potentially important parameters
- Empirical Model Building (HOW Stage)
  - determine empirically the effects of the known input parameters
  - determine the approximate function for local interpolation
- Mechanistic Model Building (WHY Stage)
  - build a function for extrapolation

TABLE 1.1. **Some scientific problems**

Supposed unknown	Objective	Descriptive name	Stage
f, $\boldsymbol{\xi}$, $\boldsymbol{\theta}$	Determine the subset $\boldsymbol{\xi}$ of important variables from a given larger set $\boldsymbol{\Xi}$ of potentially important variables	Screening variables	Which
f, $\boldsymbol{\theta}$	Determine empirically the effects of the known input variables $\boldsymbol{\xi}$	Empirical model building	How
f, $\boldsymbol{\theta}$	Determine a local interpolation approximation $g(\boldsymbol{\xi}, \boldsymbol{\beta})$ to $f(\boldsymbol{\xi}, \boldsymbol{\theta})$	(Response surface methodology)	How
f, $\boldsymbol{\theta}$	Determine f	Mechanistic model building	Why
$\boldsymbol{\theta}$	Determine θ	Mechanistic model fitting	Why

What can we learn?

- Screening studies: vary a few things to determine which factors are important (e.g., in combination with ANOVA)
    - Consider the efficiency of a rechargable battery. The redox levels of the anode and cathode matter (voltage). But you also care about the mass, volume, speed of recharging … etc.
- Modeling a process: similar - get a better understanding of a system
    - Maybe a process has interactions or nonlinear effects?
- Optimization: finding the best yield, best coffee, etc.

How is it done?

- Mathematically, we usually treat this as an example of **multiple regression**:

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_{12} X_1 X_2 + \text{ experimental error}$$ Linear

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_{11} X_1^2 + \beta_{22} X_2^2 + \text{ experimental error}$$ Non-Linear

- In general, we might have a *lot* of factors and interactions.
- Second-order interactions are pretty common. Maybe a catalyst doesn't work as well at higher temperature (e.g., it decomposes). Or light roast coffee requires longer brew times?
- In general, third-order interactions and higher are much less common. This is good because we essentially get replication "for free."

Factorial Designs at Two Levels

Chapter 4 in “Empirical Model-building and Response Surfaces”

by George E. P. Box and Norman R. Draper, Wiley Series in probability and mathematical statistics (ISBN 0-471-81033-9)

Example: Hardening of steel

- Depends on temperature before hardening (S), Oil temperature (T) and carbon level (C):

	S	T	C
Low level	830	70	0.5
High level	910	120	0.7

Which factors are more important?

Full factorial design 2^k

2^3 gives 8 different experiments for a full factorial design

Trial #		Factor	
	S (°C)	T (°C)	C (%)
1	830	70	0.5
2	910	70	0.5
3	830	120	0.5
4	910	120	0.5
5	830	70	0.7
6	910	70	0.7
7	830	120	0.7
8	910	120	0.7

Full factorial design 2^k

The real data can be coded so that we end up between -1 and 1 → **design matrix**

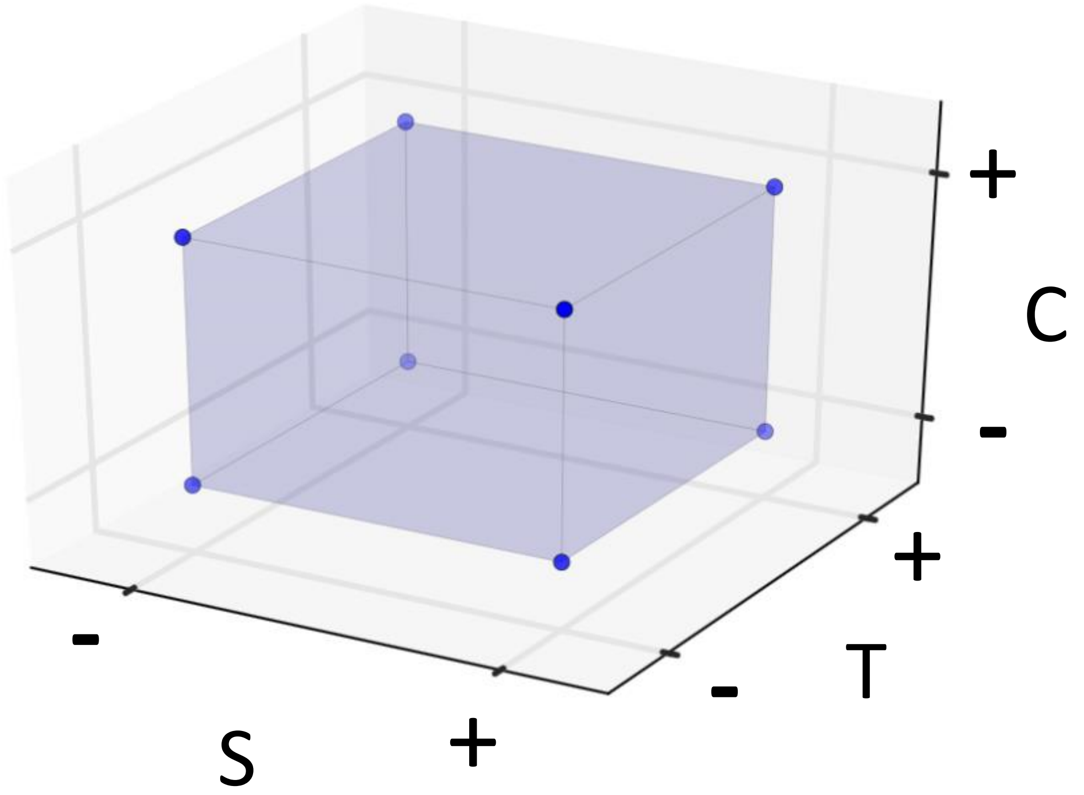
A full 2^k factorial design consists of all 2^k trial points:

$$(x_1, x_2, \ldots, x_k) = (\pm 1, \pm 1, \ldots, \pm 1),$$

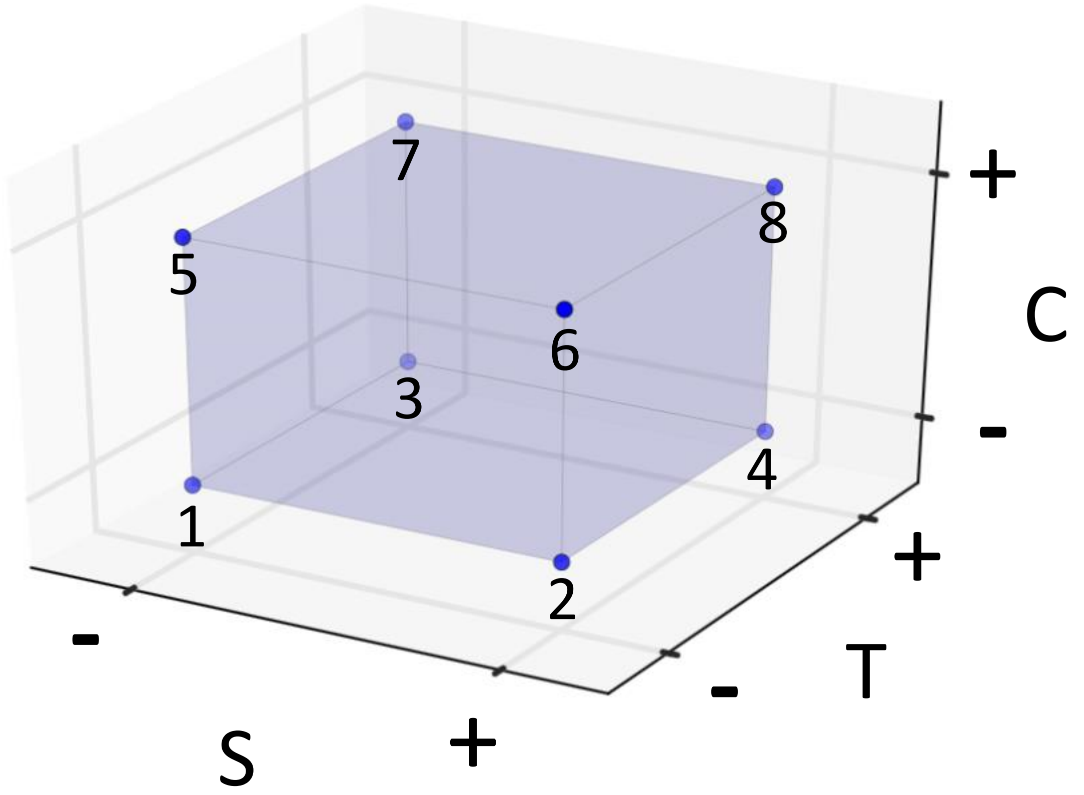
where every possible combination of +1/-1 is selected in turn

Trial #		Factor	
	S (°C)	T (°C)	C (%)
1	-1	-1	-1
2	+1	-1	-1
3	-1	+1	-1
4	+1	+1	-1
5	-1	-1	+1
6	+1	-1	+1
7	-1	+1	+1
8	+1	+1	+1

Graphical form



Graphical form



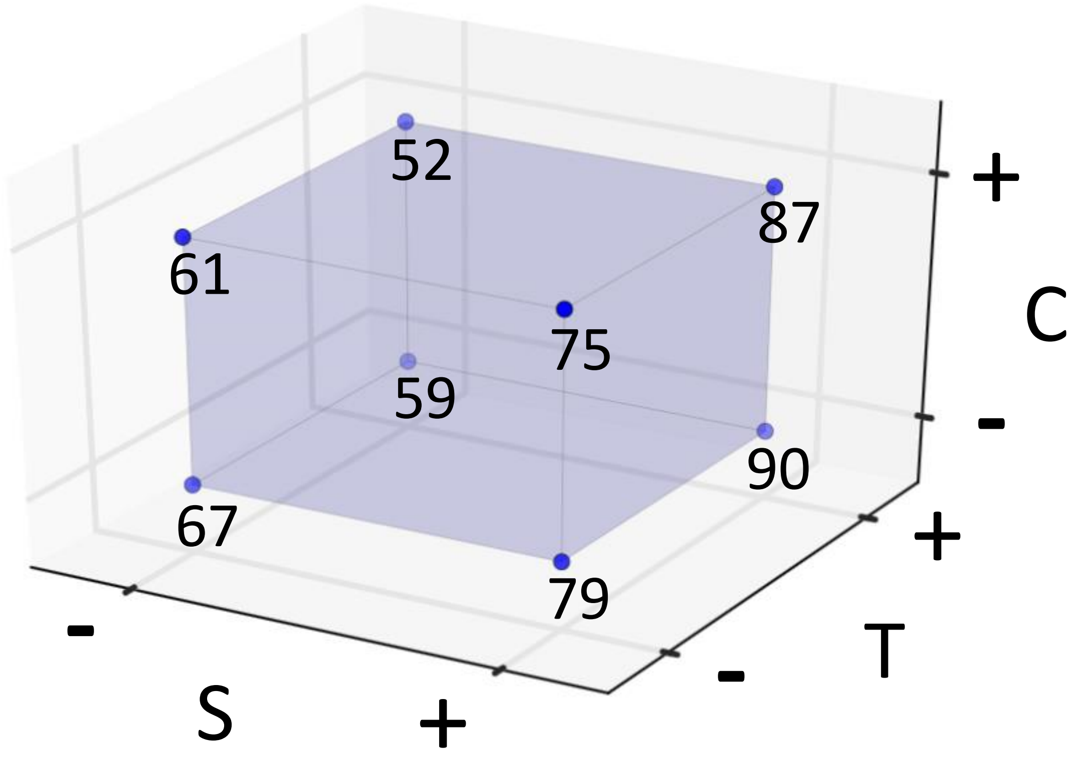
Should be randomized!

Full factorial design 2^k

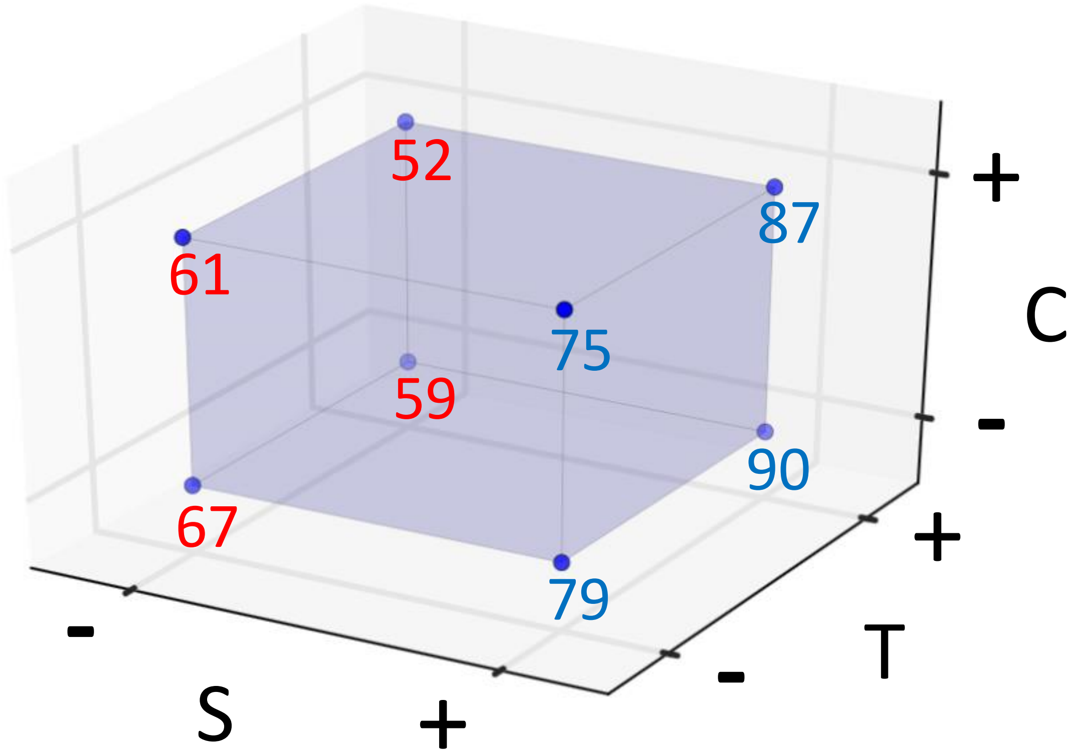
Each trial will give an outcome, in this case, a measure of the hardening of the steel in terms of % of defect-free springs.

Trial #		Factor		outcome
	S (°C)	T (°C)	C (%)	
1	-	-	-	67
2	+	-	-	79
3	-	+	-	59
4	+	+	-	90
5	-	-	+	61
6	+	-	+	75
7	-	+	+	52
8	+	+	+	87

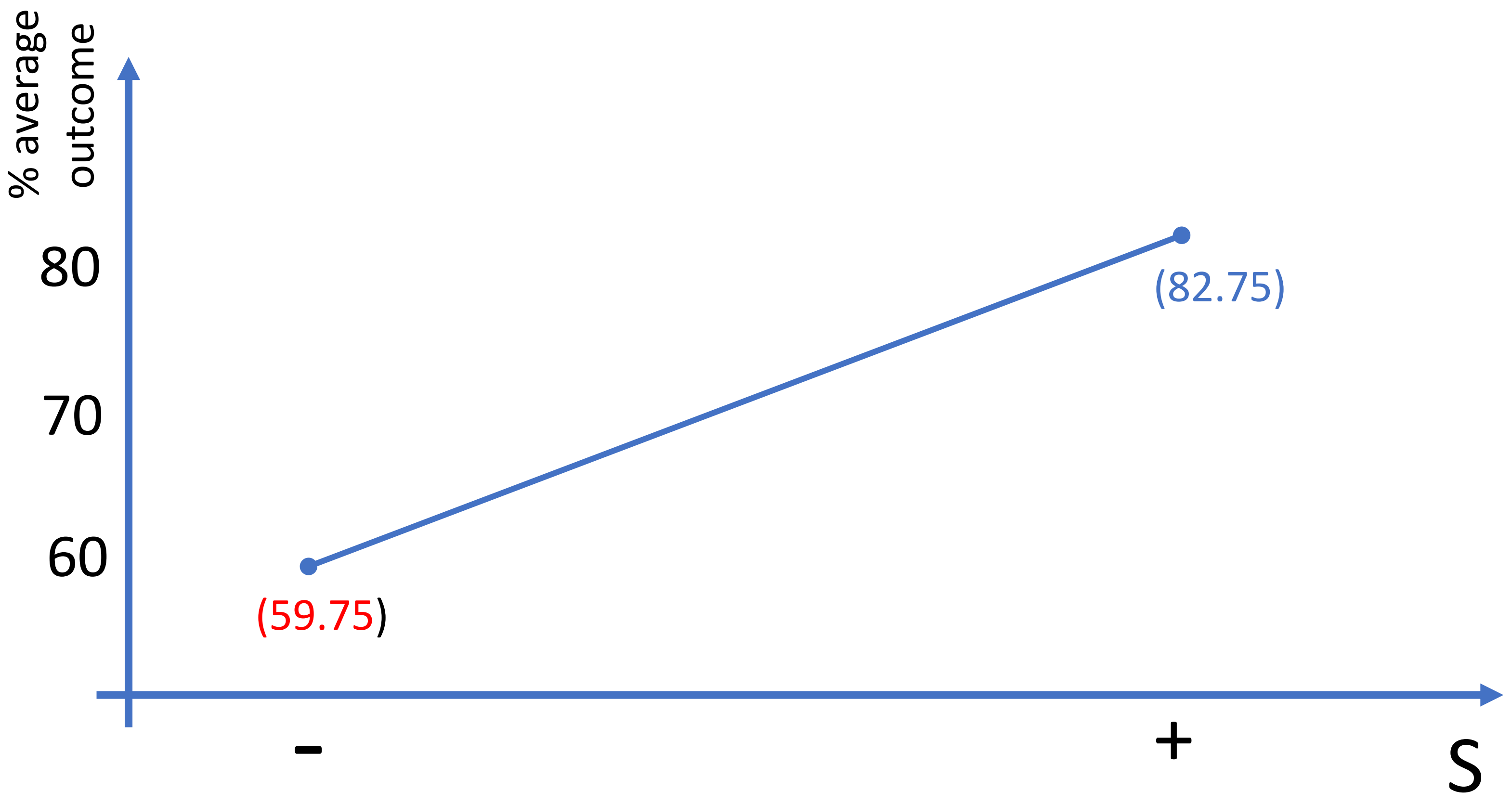
Graphical form



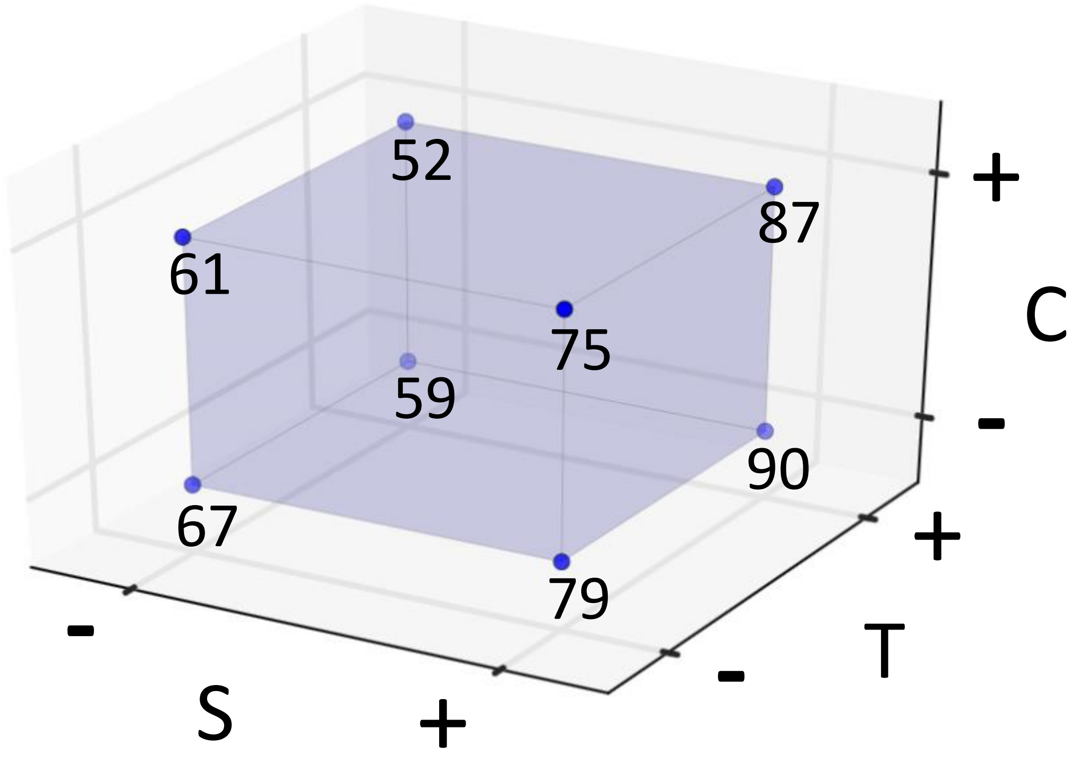
Graphical form



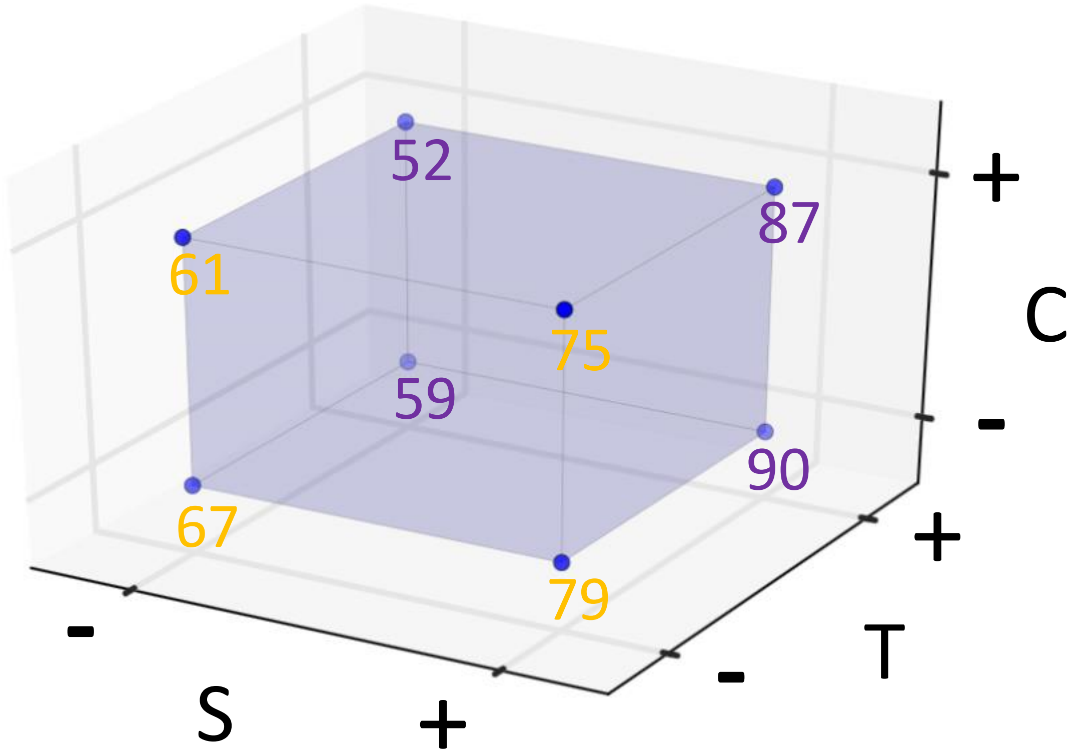
Graphical illustration



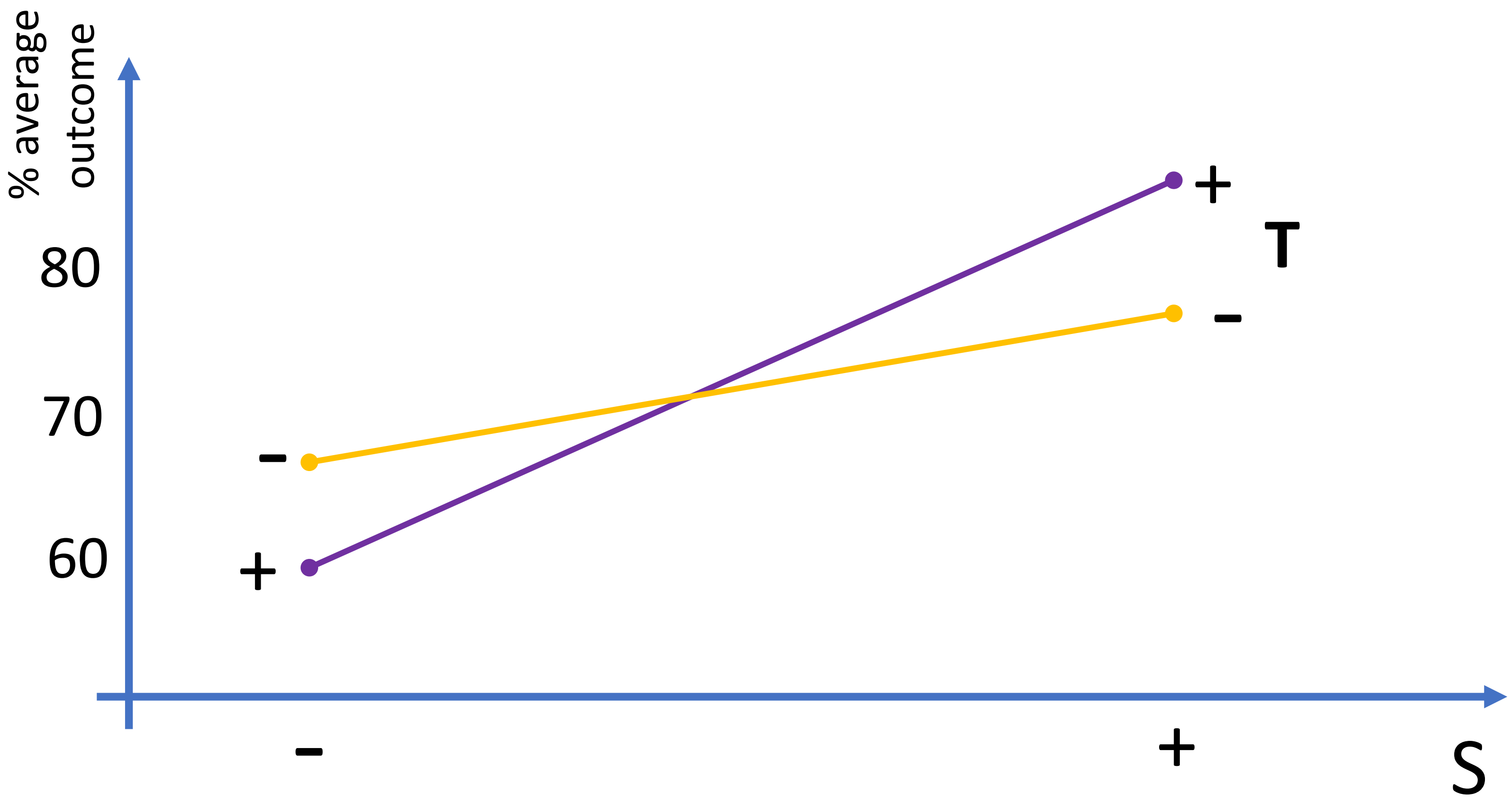
Graphical form



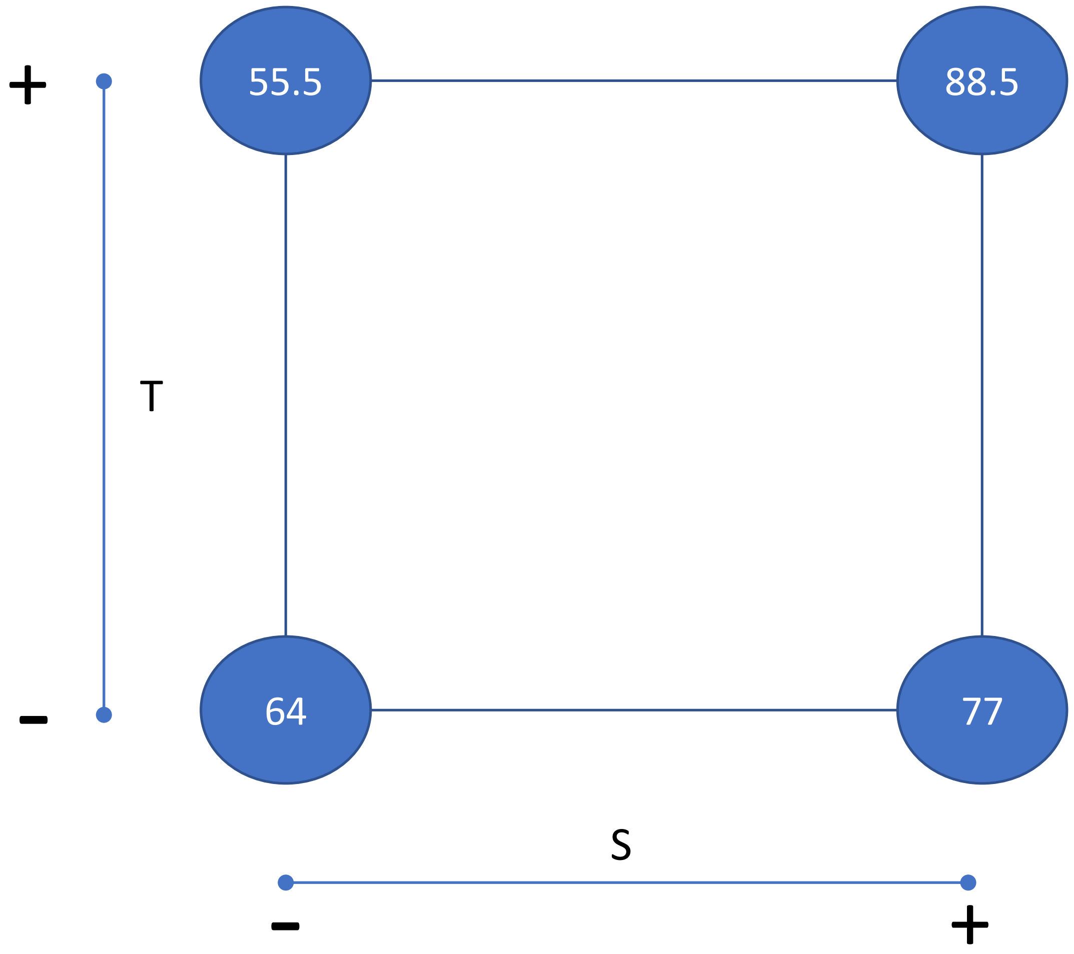
Graphical form



Graphical illustration



Graphical illustration



Full factorial design 2^k

- Graphical view of the result is difficult for $k > 3$
- Also, it is difficult to distinguish "real" effects from random variation

→ we need a quantitative way of determining the "effects" of the +/-

Example: what is the effect of increasing S? **solution:** study differences…

Trial #		Factor		outcome
	S (°C)	T (°C)	C (%)	
1	-	-	-	67
2	+	-	-	79
3	-	+	-	59
4	+	+	-	90
5	-	-	+	61
6	+	-	+	75
7	-	+	+	52
8	+	+	+	87

—

Full factorial design 2^k

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→ we need a quantitative way of determining the "effects" of the +/-

Example: what is the effect of increasing S?
solution: study differences… we have four different differences per variable…

$$1 \leftarrow \frac{12+31+14+35}{4} = 23$$

Trial #		Factor		outcome
	S (ºC)	T (ºC)	C (%)	
1	-	-	-	67
2	+	-	-	79
3	-	+	-	59
4	+	+	-	90
5	-	-	+	61
6	+	-	+	75
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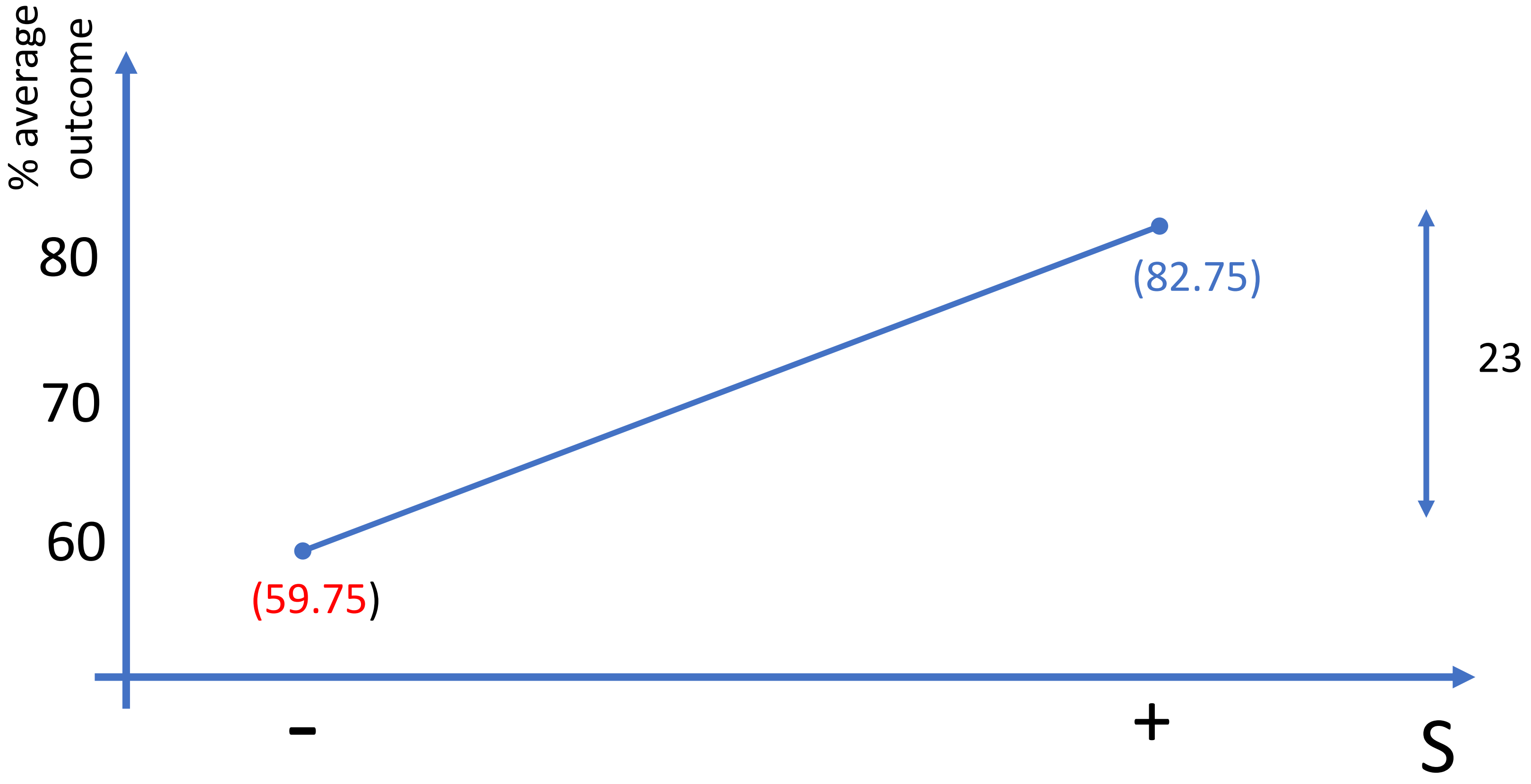
Graphical illustration



The effect of S is the difference between the arithmetic means at the low and high values of S

Full factorial design 2^k

- Graphical view of the result is difficult for k > 3
- Also, it is difficult to distinguish "real" effects from random variation

→ we need a quantitative way of determining the "effects" of the +/-

Example: what is the effect of increasing S?
solution: study differences… we have four different differences per variable…

$$S \leftarrow \frac{12+31+14+35}{4} = 23$$

$$C \leftarrow \frac{61+75+52+87-67-79-59-90}{4} = -5$$

$$T \leftarrow \frac{59+90+52+87-67-79-61-75}{4} = 1.5$$

Trial #		Factor		outcome
	S (°C)	T (°C)	C (%)	
1	-	-	-	67
2	+	-	-	79
3	-	+	-	59
4	+	+	-	90
5	-	-	+	61
6	+	-	+	75
7	-	+	+	52
8	+	+	+	87

Full factorial design 2^k

- Graphical view of the result is difficult for k > 3
- Also, it is difficult to distinguish "real" effects from random variation

→ we need a quantitative way of determining the "effects" of the +/-

Example: what is the effect of increasing S?
solution: study differences… we have four different differences per variable…

$$S \leftarrow \frac{12+31+14+35}{4} = 23$$

$$C \leftarrow \frac{61+75+52+87-67-79-59-90}{4} = -5$$

$$T \leftarrow \frac{59+90+52+87-67-79-61-75}{4} = 1.5$$

Trial #		Factor		outcome
	S (°C)	T (°C)	C (%)	
1	-	-	-	67
2	+	-	-	79
3	-	+	-	59
4	+	+	-	90
5	-	-	+	61
6	+	-	+	75
7	-	+	+	52
8	+	+	+	87
Estimated effect	**23**	**-5**	**1.5**	

What have we achieved so far?

- We have now estimates of the effect of each parameter in a minimum number of trials.
- In fact, using an OVAT would need 16 experiments to get the same information.
- But, what about cooperative effects? Can we get these as well?

Full factorial design 2^k

Trial #		Factor				Cooperative effects		# outcome
	S	**T**	**C**	**S x T**	**S x C**	**T x C**	**S x T x C**	
1	-	-	-	+	+	+	-	67
2	+	-	-	-	-	+	+	79
3	-	+	-	-	+	-	+	59
4	+	+	-	+	-	-	-	90
5	-	-	+	+	-	-	+	61
6	+	-	+	-	+	-	-	75
7	-	+	+	-	-	+	-	52
8	+	+	+	+	+	+	+	87
Estimated effect	**23**	**-5**	**1.5**					

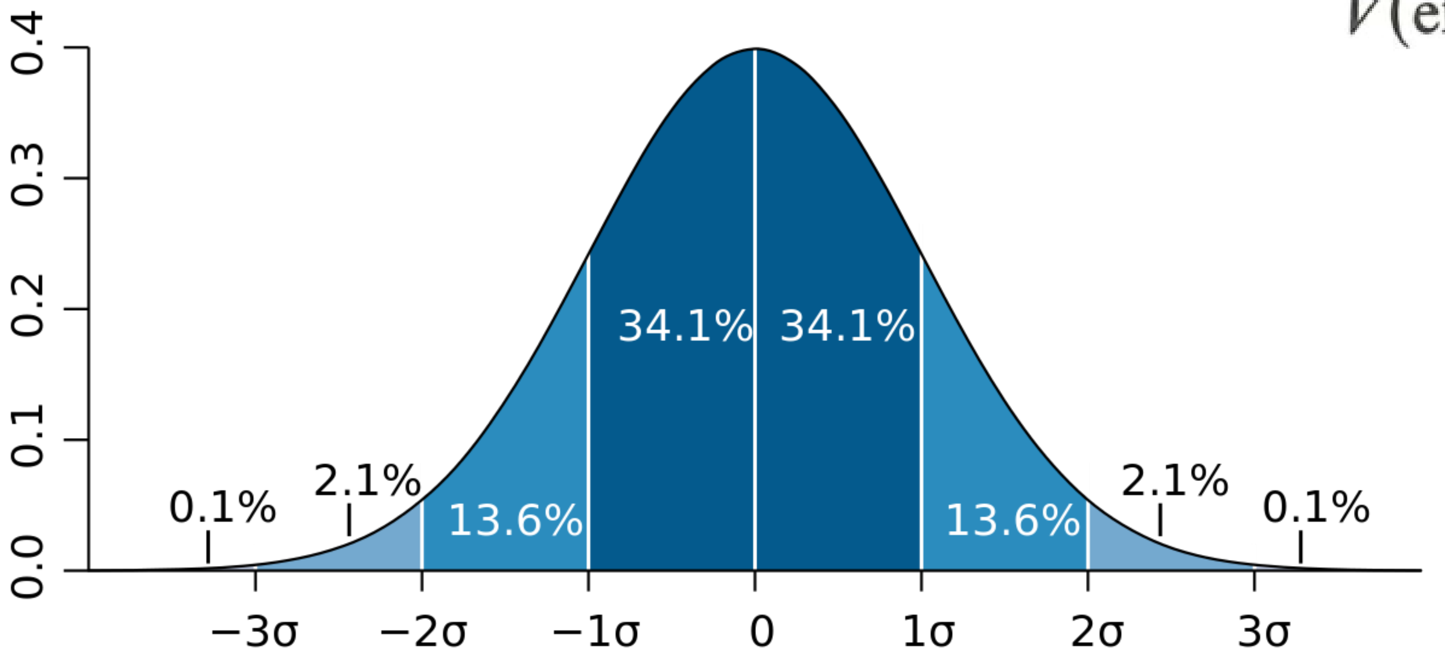
Full factorial design 2^k

Trial #		Factor				Cooperative effects		# outcome
	S	T	C	S x T	S x C	T x C	S x T x C	
1	-	-	-	+	+	+	-	67
2	+	-	-	-	-	+	+	79
3	-	+	-	-	+	-	+	59
4	+	+	-	+	-	-	-	90
5	-	-	+	+	-	-	+	61
6	+	-	+	-	+	-	-	75
7	-	+	+	-	-	+	-	52
8	+	+	+	+	+	+	+	87
Estimated effect	**23**	**-5**	**1.5**	**10**	**1.5**	**0**	**0.5**	**71.25**

Variance and standard deviation

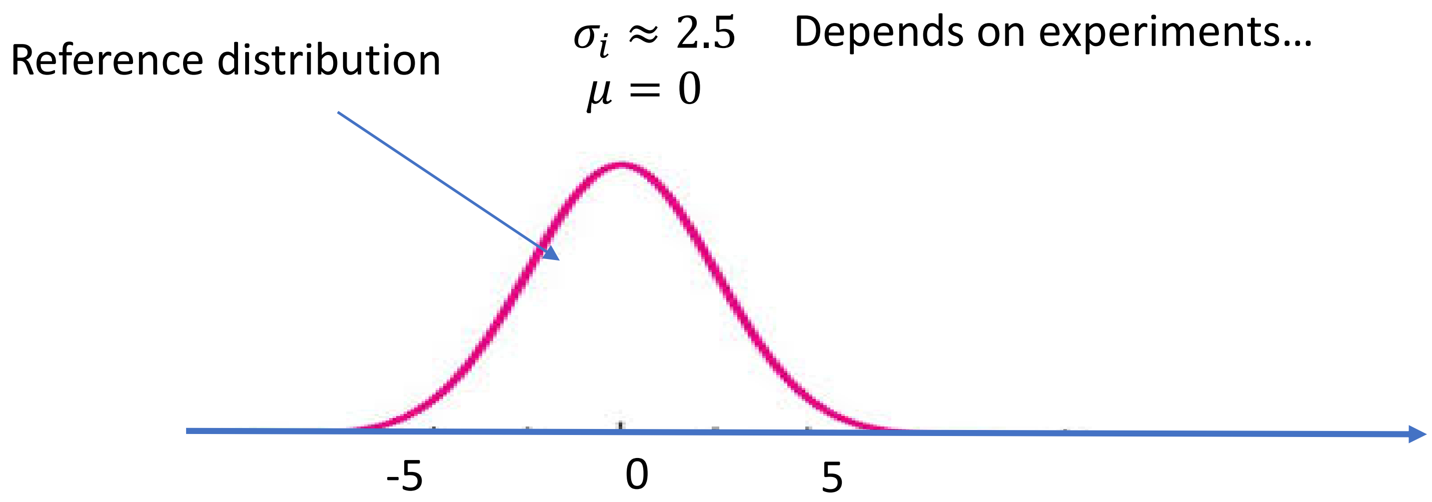
$$V(\text{grand mean}) = \frac{\sigma^2}{2^k},$$

$$V(\text{effect}) = \frac{4\sigma^2}{2^k}.$$

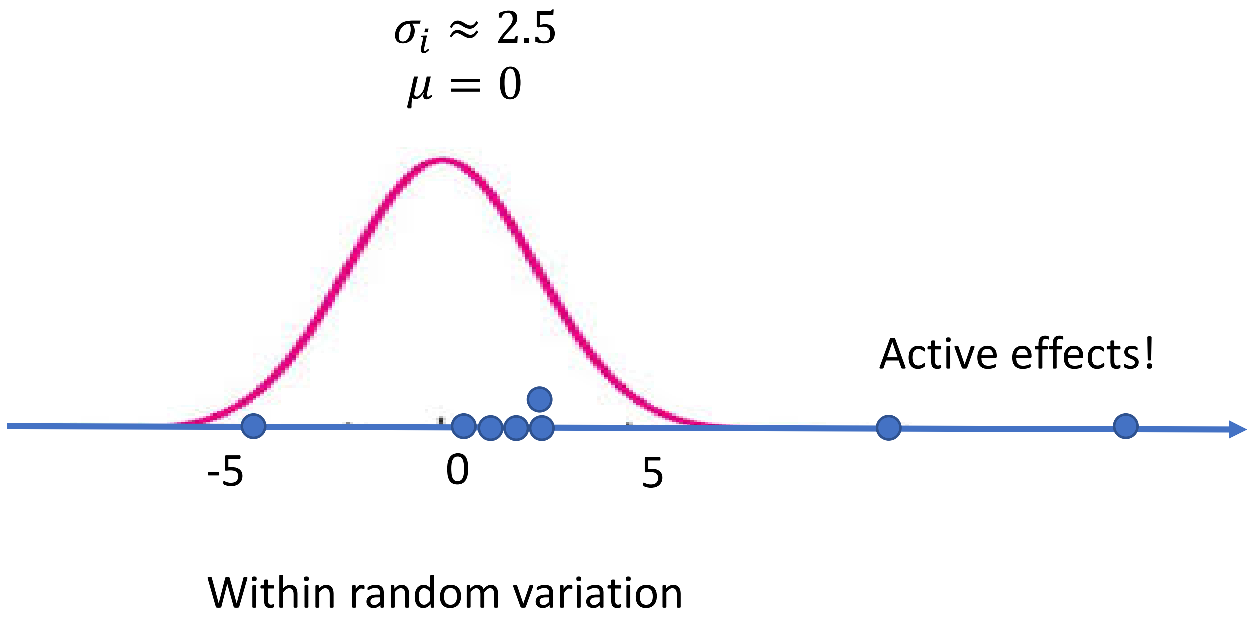


Sigmai < 3.6 →

Normal distribution



Normal distribution



Recap… new example

1.

Coded levels	x_i	-1	1
Length of test specimen (mm)	ξ_1	250	350
Amplitude of load cycle (mm)	ξ_2	8	10
Load (g)	ξ_3	40	50

$$x_1 = \frac{(\xi_1 - 300)}{50}, \quad x_2 = \frac{(\xi_2 - 9)}{1}, \quad \text{and} \quad x_3 = \frac{(\xi_3 - 45)}{5}.$$

2.

Uncoded			Coded			Cycles	
Specimen length (mm), ξ_1	Amplitude (mm), ξ_2	Load (g), ξ_3	Specimen length, x_1	Amplitude, x_2	Load, x_3	to failure, Y	$y = \log Y$
250	8	40	-1	-1	-1	674	2.83
350	8	40	1	-1	-1	3636	3.56
250	10	40	-1	1	-1	170	2.23
350	10	40	1	1	-1	1140	3.06
250	8	50	-1	-1	1	292	2.47
350	8	50	1	-1	1	2000	3.30
250	10	50	-1	1	1	90	1.95
350	10	50	1	1	1	360	2.56

3.

	I	1	2	3	12	13	23	123	y
	+	−	−	−	+	+	+	−	2.83
	+	+	−	−	−	−	+	+	3.56
	+	−	+	−	−	+	−	+	2.23
	+	+	+	−	+	−	−	−	3.06
	+	−	−	+	+	−	−	+	2.47
	+	+	−	+	−	+	−	−	3.30
	+	−	+	+	−	−	+	−	1.95
	+	+	+	+	+	+	+	+	2.56
Divisor	8	4	4	4	4	4	4	4	

4.

$$1 \leftarrow \tfrac{1}{4}(3.56 + 3.06 + 3.30 + 2.56) - \tfrac{1}{4}(2.83 + 2.23 + 2.47 + 1.95) = 0.75.$$

$$2 \leftarrow -0.59,$$

$$3 \leftarrow -0.35.$$

$$(1|x_3 = -1) \leftarrow \tfrac{1}{2}(3.56 + 3.06) - \tfrac{1}{2}(2.83 + 2.23) = 0.78.$$

$$(1|x_3 = 1) \leftarrow \tfrac{1}{2}(3.30 + 2.56) - \tfrac{1}{2}(2.47 + 1.95) = 0.72.$$

$$13 = \tfrac{1}{2}\{(1|x_3 = 1) - (1|x_3 = -1)\} \leftarrow \tfrac{1}{2}\{0.72 - 0.78\} = -0.03.$$

$$12 \leftarrow -0.03,$$

$$23 \leftarrow -0.04.$$

$$(12|x_3 = -1) \leftarrow \tfrac{1}{2}\{(3.06 - 2.23) - (3.56 - 2.83)\} = 0.05,$$

$$(12|x_3 = 1) \leftarrow \tfrac{1}{2}\{(2.56 - 1.95) - (3.30 - 2.47)\} = -0.11.$$

$$123 = \tfrac{1}{2}\{(12|x_3 = 1) - (12|x_3 = -1)\} \leftarrow -0.08.$$

3.

	I	1	2	3	12	13	23	123	y
	+	−	−	−	+	+	+	−	2.83
	+	+	−	−	−	−	+	+	3.56
	+	−	+	−	−	+	−	+	2.23
	+	+	+	−	+	−	−	−	3.06
	+	−	−	+	+	−	−	+	2.47
	+	+	−	+	−	+	−	−	3.30
	+	−	+	+	−	−	+	−	1.95
	+	+	+	+	+	+	+	+	2.56
Divisor	8	4	4	4	4	4	4	4	

4.

$$1 \leftarrow \tfrac{1}{4}(-y_1 + y_2 - y_3 + y_4 - y_5 + y_6 - y_7 + y_8).$$

$$2 \leftarrow \tfrac{1}{4}(-y_1 - y_2 + y_3 + y_4 - y_5 - y_6 + y_7 + y_8),$$

$$13 \leftarrow \tfrac{1}{4}(y_1 - y_2 + y_3 - y_4 - y_5 + y_6 - y_7 + y_8).$$

$$123 \leftarrow \tfrac{1}{4}(-y_1 + y_2 + y_3 - y_4 + y_5 - y_6 - y_7 + y_8).$$

5. suppose that an estimate $s^2 = 0.0050$ $\longrightarrow$ $\hat{V}(\bar{y}) = 0.000625$, $\hat{V}(\text{effect}) = 0.0025$, $\longrightarrow$ $s(\bar{y}) = 0.025$, $s(\text{effect}) = 0.05$.

$I \leftarrow \bar{y} = 2.745 \pm 0.025,$

$1 \leftarrow 0.75 \pm 0.05,$

$2 \leftarrow -0.59 \pm 0.05,$

$3 \leftarrow -0.35 \pm 0.05,$

$12 \leftarrow 0.03 \pm 0.05,$

$13 \leftarrow 0.03 \pm 0.05,$

$23 \leftarrow 0.04 \pm 0.05,$

$123 \leftarrow 0.08 \pm 0.05.$

Regression gives:

fit a first degree polynomial

$$\hat{y} = \underset{(0.025)}{2.745} + \underset{(0.025)}{0.375x_1} - \underset{(0.025)}{0.295x_2} - \underset{(0.025)}{0.175x_3} .$$

What have we learnt?

- How to create a design matrix for a 2-level factorial design
- How to analyse the data… we will look at more examples in the workshop…