Design of optimal experiments for improved modeling food processes

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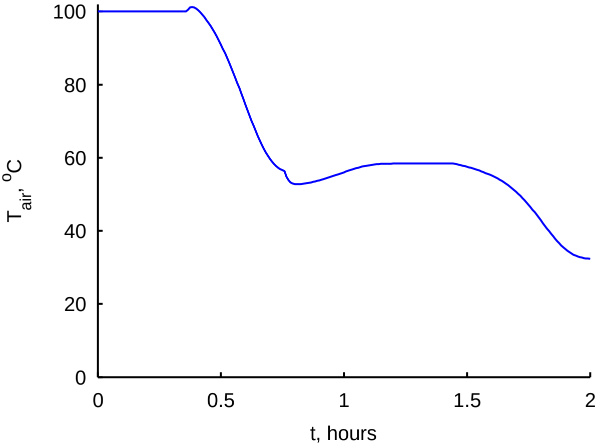
Context: Simulating a pilot plant for another product

- We have a simulation model for corn drying
  - → Often in food process: nonlinear ordinary differential equations (ODEs) and is nonlinear in parameters: nonclassic design of experiments (DoE) is difficult.
- Objective: use this model for **rice** drying
- Method: update **product**-related model parameters
  - → Diffusion coefficient $D_w(X, T)$
  - → Heat transfer coefficient at surface $h(T_{air})$
  - → Inner structure

Successive estimations of $h(100^\circ C)$ will follow, based on experiments with varying temperature.

Efficient "A-optimal" strategy

One experiment (2 hours):

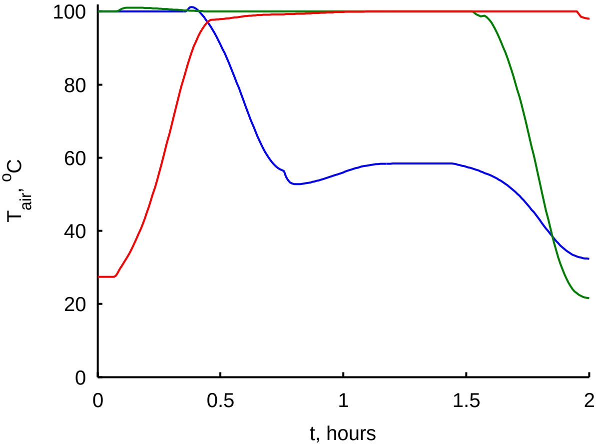


Note: HR_{air} is optimized similarly, other parameters are also estimated.
**"predicted quality of estimation" is the inverse of A-optimal criterion.*

$h(100^\circ C) = 15 \pm 9$ W.K^{-1}.m^{-2} has the right order of magnitude.   Predicted global **quality*** of estimation: q=0.27

Efficient ”A-optimal” strategy

Three experiments (2 hours each):



*Note: *”predicted quality of estimation” is the inverse of A-optimal criterion..*

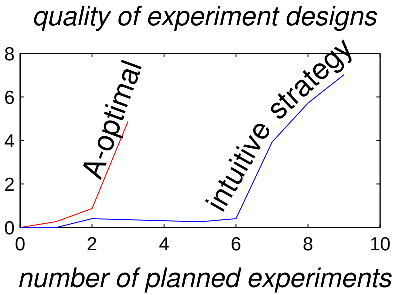
$h(100^\circ C) = 11.5 \pm 0.8$ W.K^{-1}.m^{-2}

Predicted global **quality*** of estimation: q=4.87

Comparing intuitive and optimized strategies

Two strategies were tested to choose T_{air} and HR_{air}.

- Model-based optimal strategy with **varying** optimized drying conditions.
  - $\rightarrow$ $h(100^\circ C) = 11.5 \pm 0.8$ W.K^{-1}.m^{-2}
- Intuitive strategy: central design
  - $\rightarrow$ Two-factor three-level grid of 9 experiments: T_{air} =50°C, 70°C, 90°C
  - $\rightarrow$ $h(100^\circ C) = 13.0 \pm 0.5$ W.K^{-1}.m^{-2}



Workload reduction factor 2.6*. **Journal of Process Control 2012, pp. 95-107*

Illustrating seven maximization pitfalls

Problem from van Impe's team:

- Identifying kinetics of Escherichia coli growth by van Derlinden et al's mOdeL (FOODSIM 2008, pp. 102-109).
- Only temperature is optimized.

Particularity: analytic solution of these differential equations

- Better illustrations.
- Not needed for optimal experiments design.

Sparing computer resources for better models.

Simulation model of Escherichia coli

Simulation model with 5 parameters:

$$\begin{cases} \frac{dN}{dt}(t) = Q(t)\mu\big(T(t)\big)N(t)(1 - \frac{N(t)}{N_{\max}}) \\ \frac{dQ}{dt}(t) = Q(t)\mu\big(T(t)\big)(1 - Q(t)) \\ \mu(T) = \frac{\mu_o}{T_o - T_-} \times \frac{(T-T_-)^2(T-T_+)}{(T_o-T_-)(T-T_o)-(T_o-T_+)(T_o+T_- - 2T)} \end{cases}$$

5 unknown parameters to identify by experiments

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N_{max} Upper bound of Colony Forming Units per ml.

μ_o Maximal growth rate of ln(N), 1/h.

$[T_-, T_+]$ Temperature range for growth.

T_o Temperature for fastest growth.

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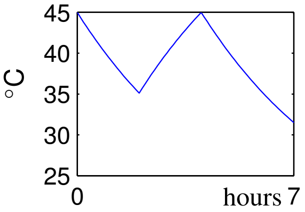
Parameterizations of the temperature profile

Temperature profile tuned by only* two numbers:

-
-

Example of temperature profile



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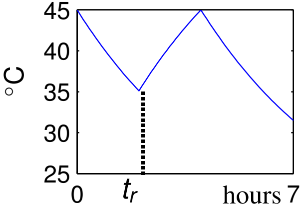


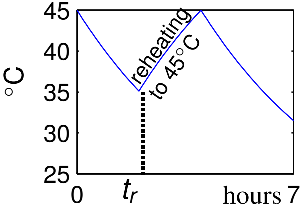
Parameterizations of the temperature profile

Temperature profile tuned by only* two numbers:

- t_r: cooling time before reheating, h
- s, heating power, $^\circ$C/h

Innoculation done at t=0; permanent cooling as exp(-t/5) to 15°C.

Example of temperature profile



Predicting the quality of estimation

- Notation required to compute quality of design: the Jacobian J of predictions is a 8x5 matrix:
  - $\rightarrow$ 8 predictions of sampled concentrations* (with standard error σ_{measure})
  - $\rightarrow$ 5 parameters.
- Definition of the quality of experimental design (inverse of A-optimal criterion)

$$q = \frac{\left(trace(J^T \times J)^{-1}\right)^{-1/2}}{\sigma_{\text{measure}}} \approx \frac{1}{\sigma_{\text{parameters}}}$$

  Different temperature profile leads to different qualities.

**7 hours of experiments, one hour between samples*

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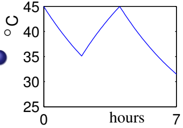
- 

$\Rightarrow$ Quality q=0.0111$\cdots$

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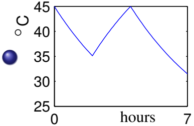
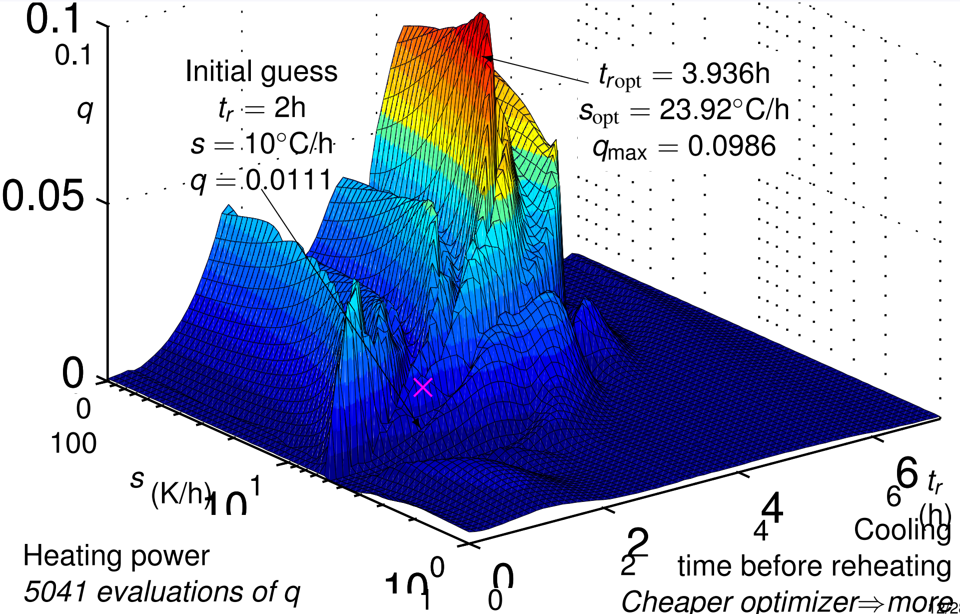
- 

$\Rightarrow$ Quality q=0.0111$\cdots$
- Other temperature profiles $\Rightarrow$ other qualities.

**7 hours of experiments, one hour between samples*

Quality of all possible experiments

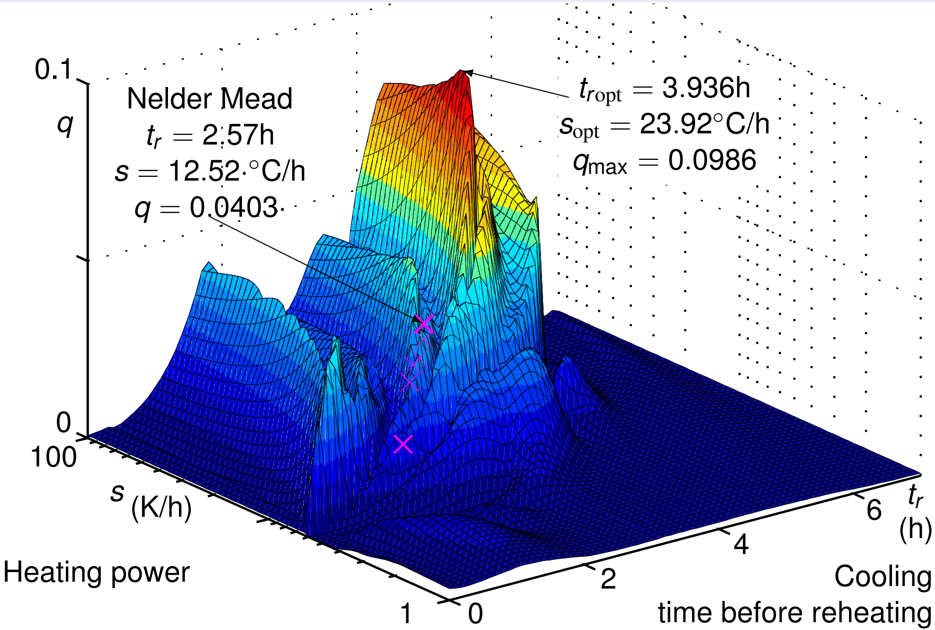


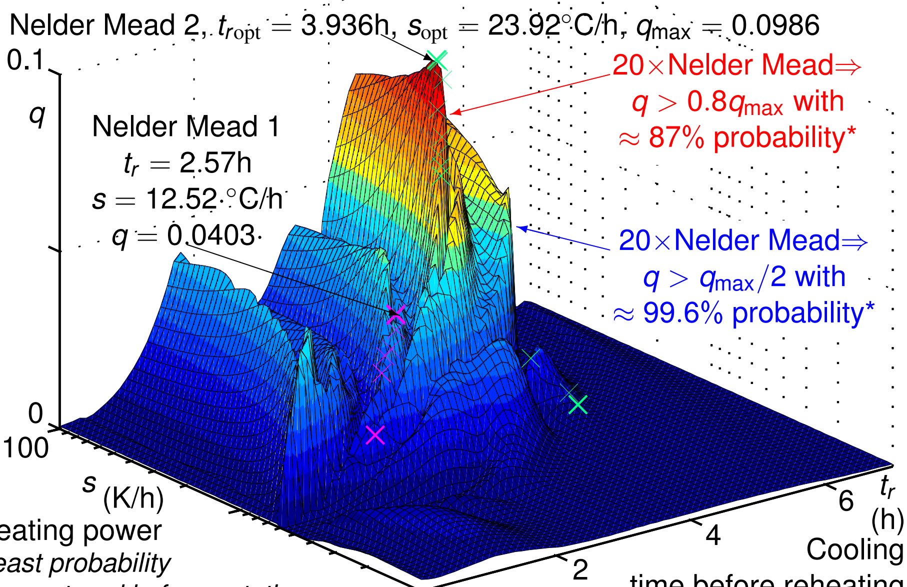
Cooling time before reheating

Heating power

5041 evaluations of q

Cheaper optimizer⇒more

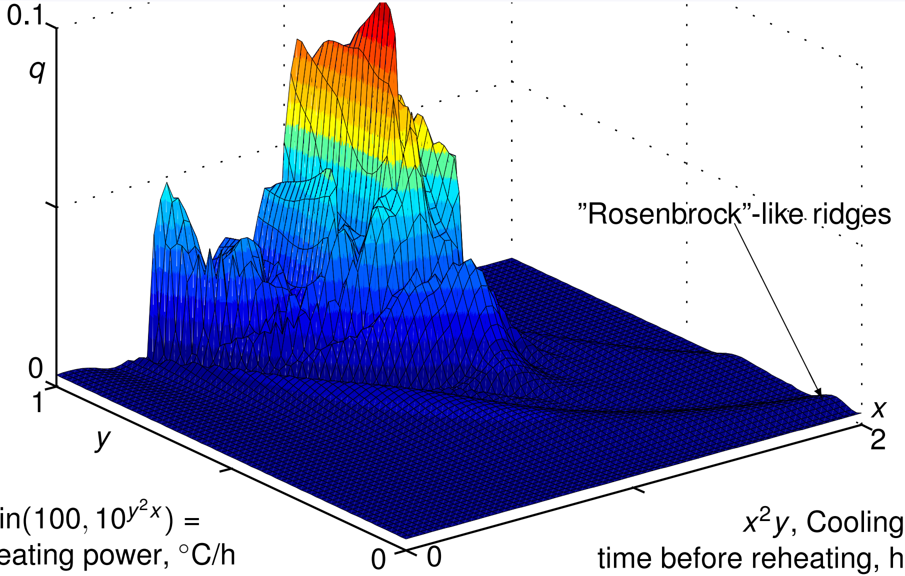

0.1
q
0
Nelder Mead
$t_r = 2.57$h
$s = 12.52$°C/h
$q = 0.0403$
$t_{ropt} = 3.936$h
$s_{opt} = 23.92$°C/h
$q_{max} = 0.0986$
100
s (K/h)
Heating power
1
0
2
4
6
t_r (h)
Cooling
time before reheating



**least probability encountered in fermentation, drying, reactions, pharmacokinetics.*

7 pitfalls should be avoided.

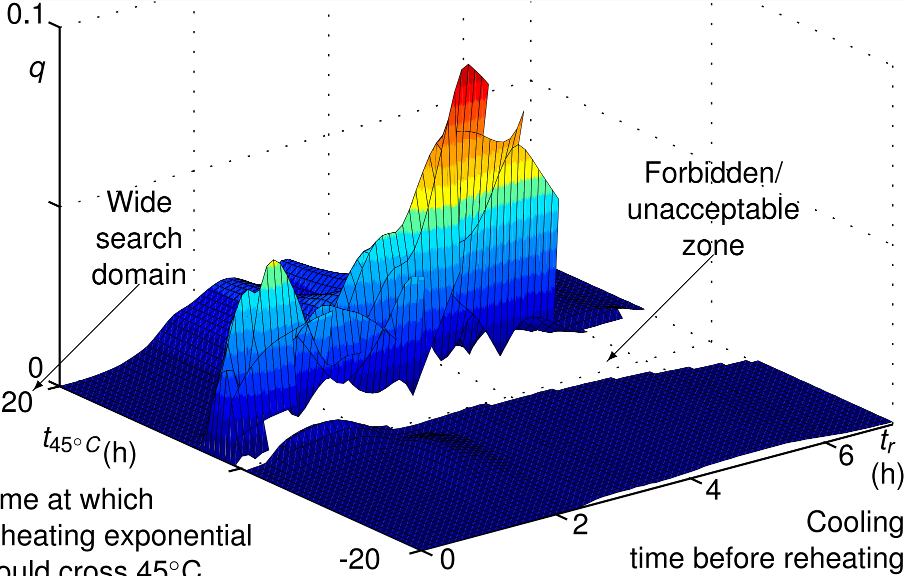
Pitfall 1: correlations ⇒ axis direction quite useless



$\min(100, 10^{y^2 x})$ = Heating power, °C/h

$x^2 y$, Cooling time before reheating, h

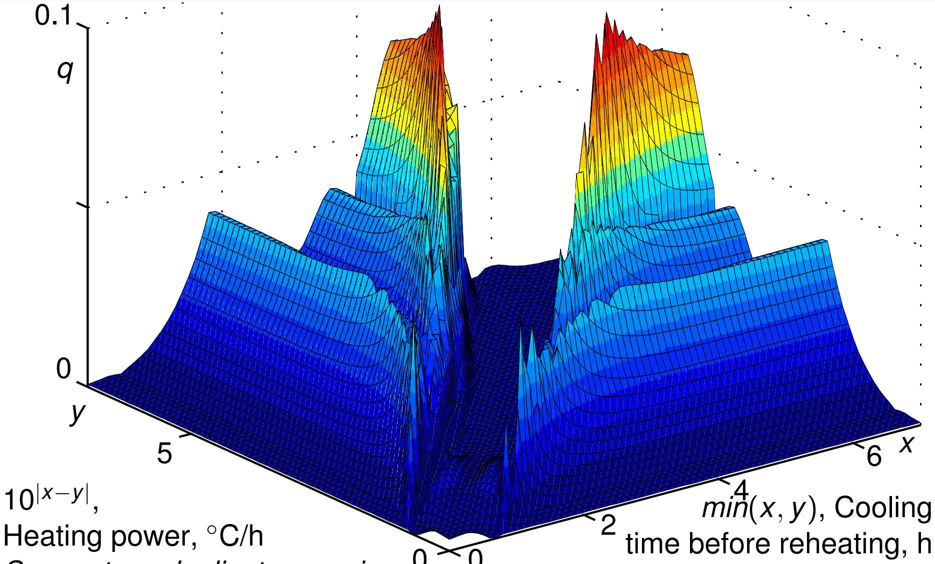
Pitfall 2: constrains, wide ⇒ complex boundaries



Time at which reheating exponential would cross 45°C

Cooling time before reheating

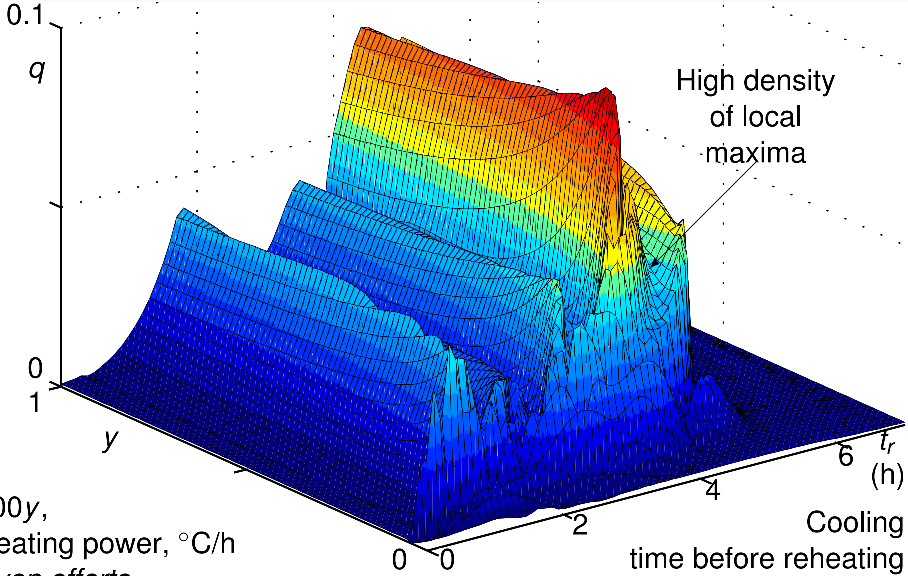
Pitfall 3: repetitions ⇒ higher density of local extrema



Symmetry⇒duplicates maxima

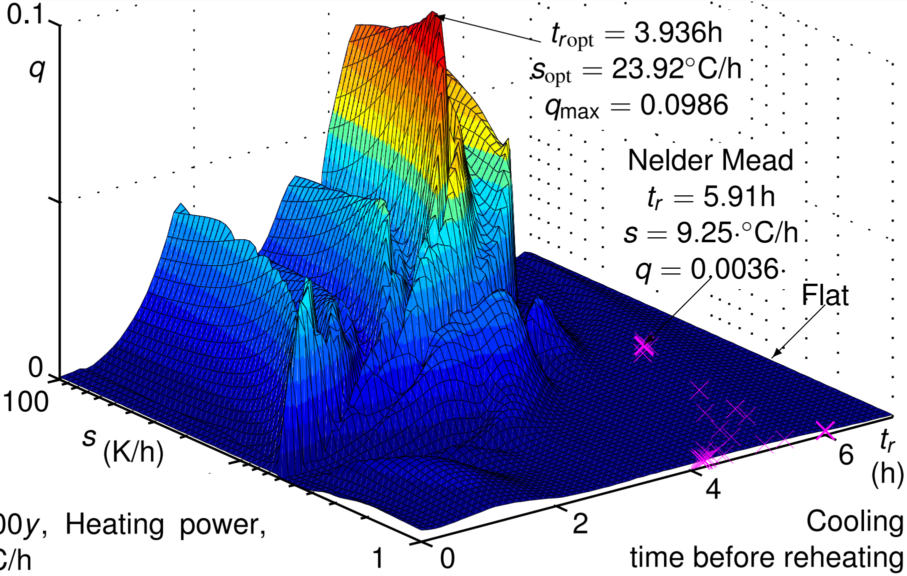
⇒*slows search*

Pitfall 4: sensitivity peaks ⇒ extrema overlooked



100y,
Heating power, °C/h
Even efforts

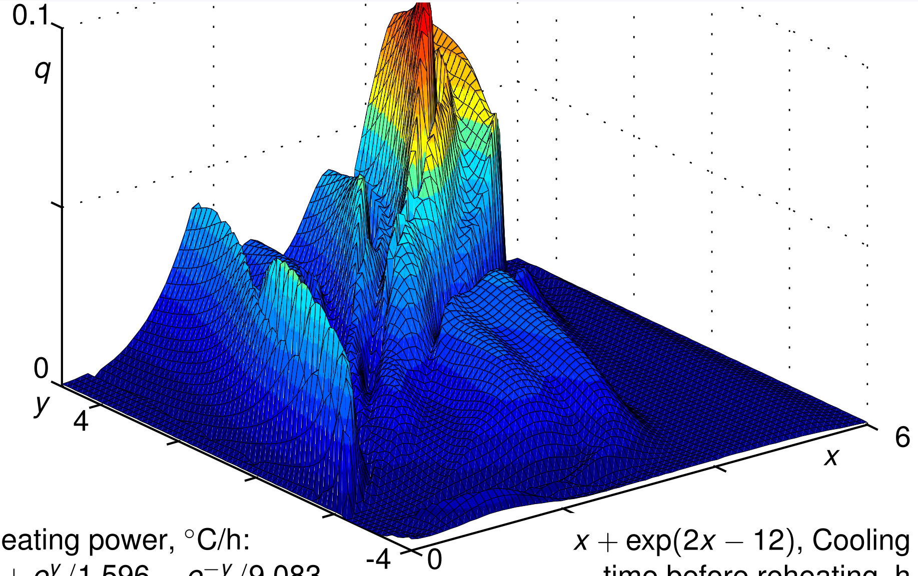
Pitfall 5: flat zones ⇒ no local identifiability



100y, Heating power, °C/h

Cooling time before reheating

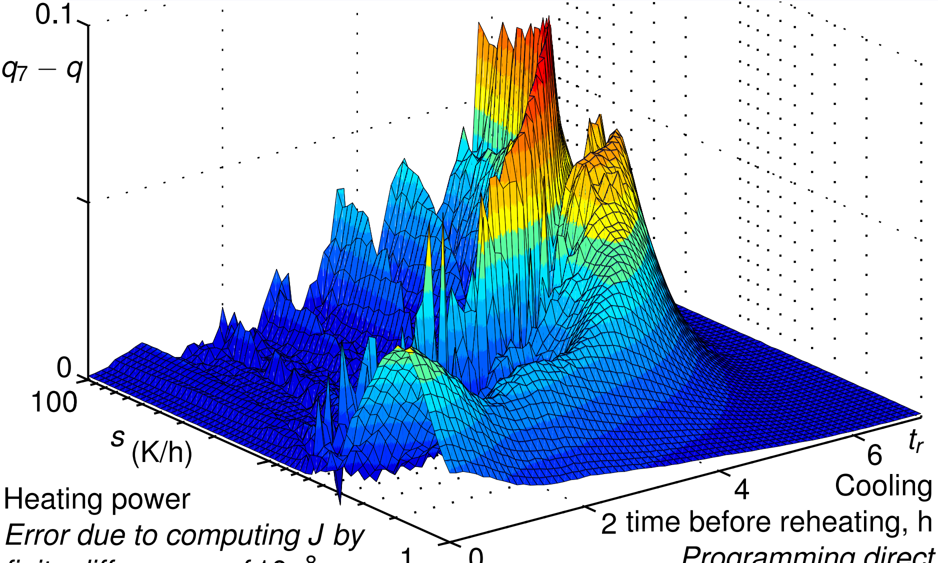
Pitfall 6: odd parameters ⇒ nonphysical parameters



Heating power, °C/h:
$7 + e^y/1.596 - e^{-y}/9.083$

$x + \exp(2x - 12)$, Cooling time before reheating, h

Pitfall 7: finite differentiation ⇒ noise and bias

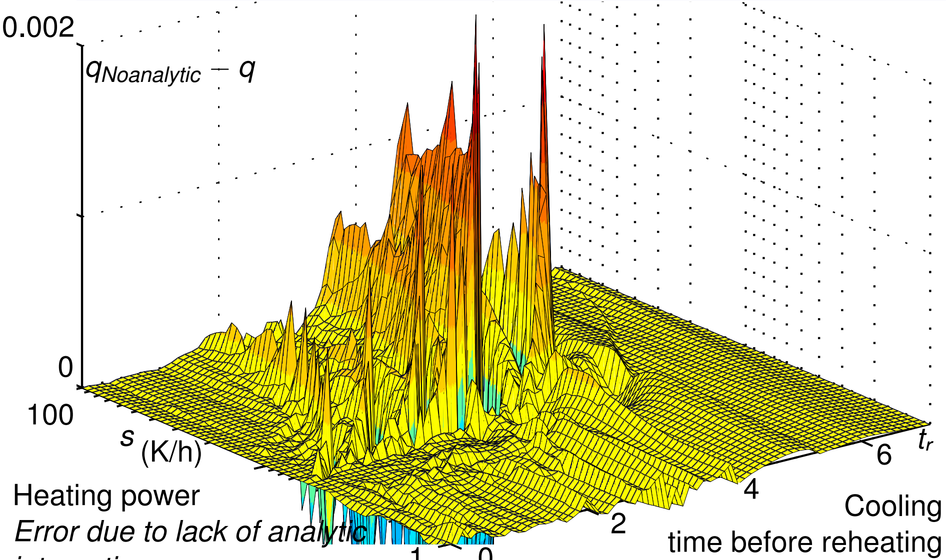


Heating power

Error due to computing J by finite differences of 10^{-8}

Programming direct differentiation avoids this pitfall

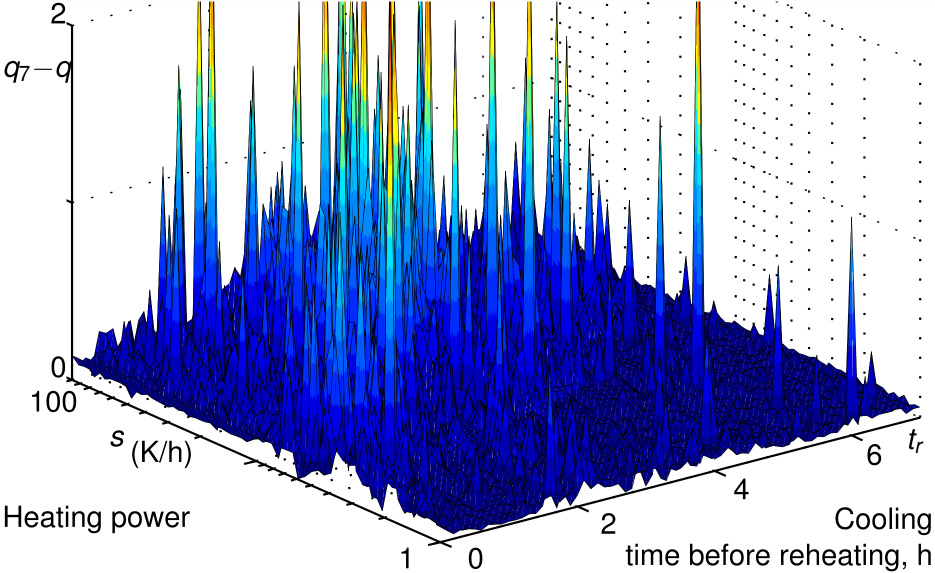
No analytic integration⇒workable ridges+ODE noise



Heating power
Error due to lack of analytic integration

Cooling
time before reheating
Error is small

Pitfall 7+no analytic integration=disaster (noise>signal)



Error for computing J by finite differences of 10^{-5} *& lack of analytic integration*

Pitfall 7: disaster without analytic

- Works without analytic, when solution is non-analytic:
  - → Real quality design: $q_{\mathrm{opt,NoAnalyTiC`3}} = 0.0986$ despite the ridges.
- Pitfall 7, finite differentiation method, with optimal difference=10^{-11}:
  - → $q_{7opt} = 0.0358$
  - → Bigger powers of 10 create bias, over-estimating q_{7opt}.
  - → Smaller powers of 10 create precision loss on q_{7opt}.
  - → Mean of all q_{7opt} is 0.0102.
- Pitfall 7 without analytic, with optimal difference=10^{-5}
  - → $q_{7\&8opt} = 0.0111$
  - → $q_{7\&8opt}$ For other powers of 10: mean=0.0052.
  - → Terrible noise ⇒
    - Progression limited to 9.5% of what is needed.
    - Hence 0.095^{-2} more random guesses are needed.

Pitfall 7 is terrible.

After experiment, estimation of model's unknowns:

- Sensitivity matrix J is available$\Rightarrow$ Gauss-Newton methods are allowed.
- Reparameterization of model's unknowns is mandatory, and:
  - $\rightarrow$ Changes the optimal experiment.
  - $\rightarrow$ Facilitates the use of A-optimality, which gives the better results than D-optimal strategy.

1. Utility and application of optimal design
2. Maximizing the predicted quality
3. Maximizing the predicted quality: seven numerical pitfalls
4. **Conclusions**

New toolbox for experiment design

- Requires simulation model, computer time, pilot plant.
- Implemented in MATLAB (saisir Toolbox)
- Uses fast optimization methods when all 7 pitfalls are avoided.
- Double-checked on two published DoE: van Impe (2008, E. Coli) and Pronzato (2008, pharmacology).
- Experimental validation on application of rice drying in Goujot (2012, J Proc Cont), pending on Ethylene Diacetate + 2 NaOH in reactor (LGC, Toulouse).

Thank you for your attention.