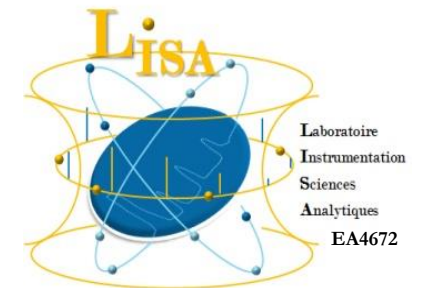
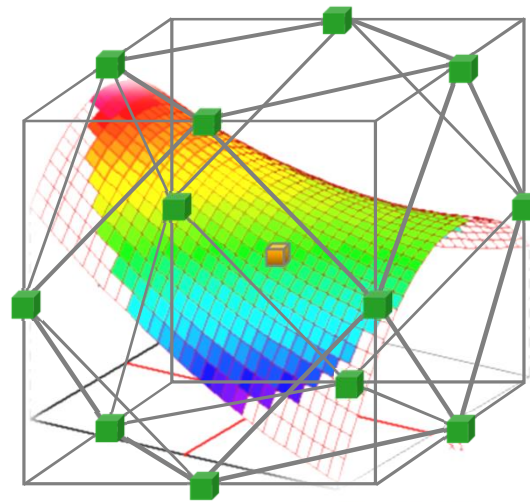


# Designs of experiments

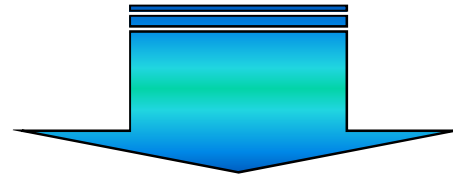


**Michelle Sergent**

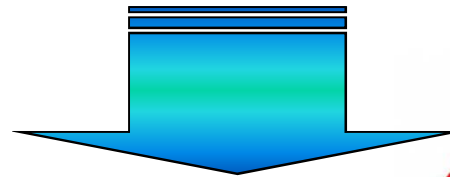
Laboratoire d'Instrumentation et Sciences Analytiques. EA 4672  
Aix Marseille Université (France)



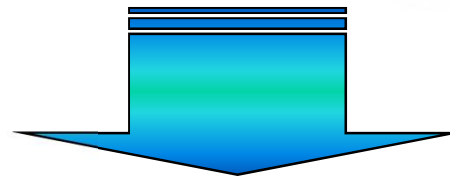
# Experiments



# Information



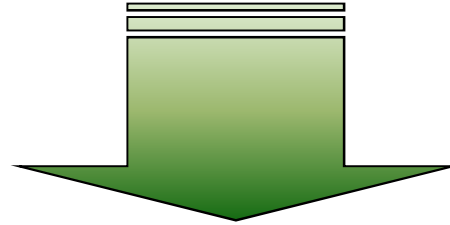
# Decision



# Risk



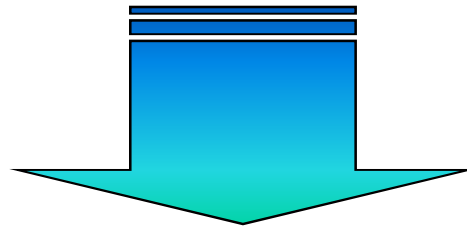
***The risk depends on the quality of the information***



***The information must be of the best quality or, of acceptable quality !!!***

***The quality of the information does not depend on the number of experiments !***

*The quality of the information depends on the  
position of the experimental points*



# *Experimental Planning*

Experimental Design can be defined as the strategy for setting up experiments in such a manner that the required information is obtained as efficiently and precisely as possible

# Description of the problem



## The targets

The list of the **input variables**

The list of the **output variables**

Domain of variation

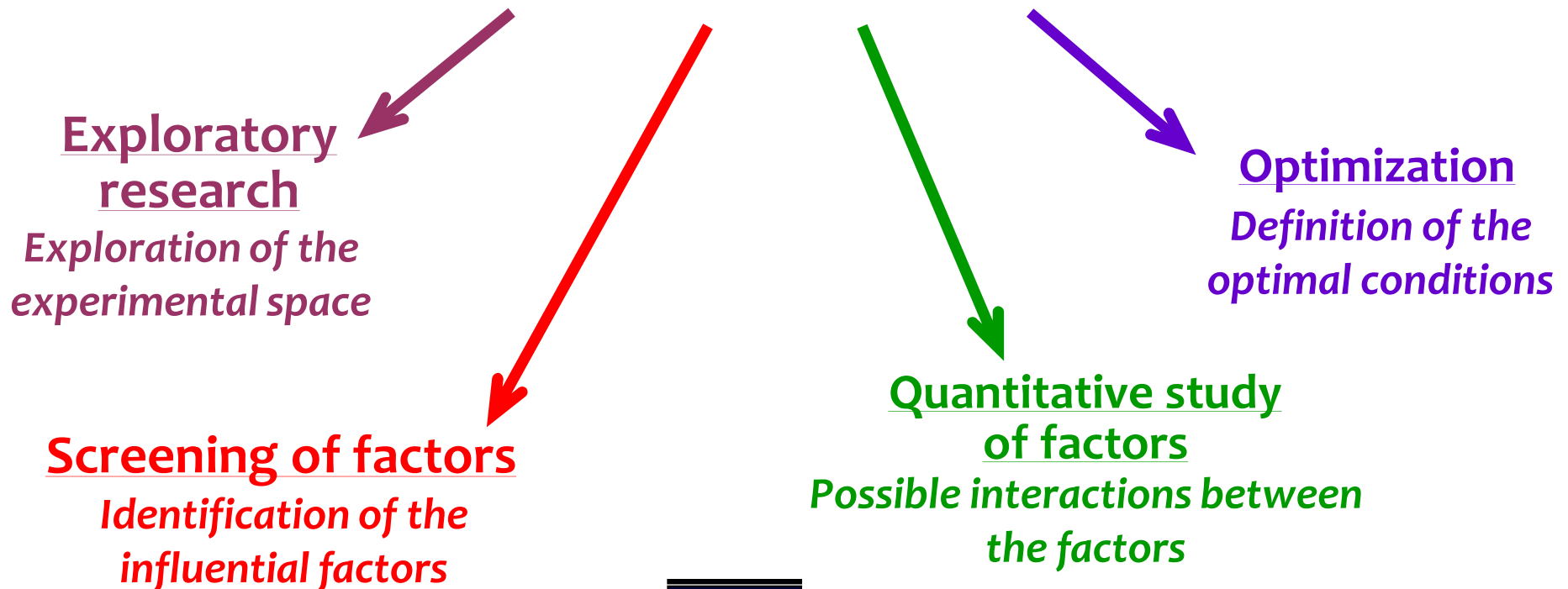
Elaboration of a strategy

*Design of experiments*

To elaborate the experimental strategy

➔ *to choose an appropriate design of experiments  
in accordance with*

# THE OBJECTIVES



Design of experiments

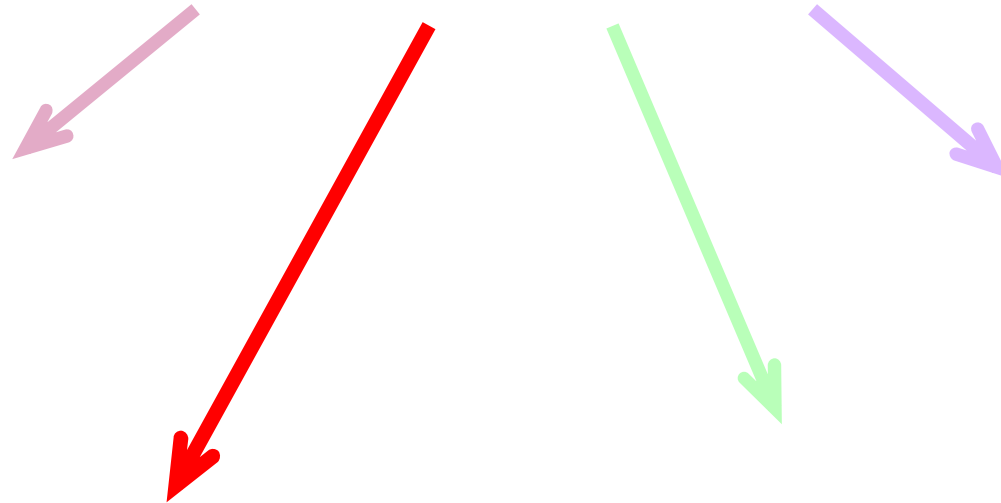


To elaborate the experimental strategy



*to choose an appropriate design of experiments  
in accordance with*

# THE OBJECTIVES



## Screening of factors

*Identification of the  
influential factors*

# Screening of factors





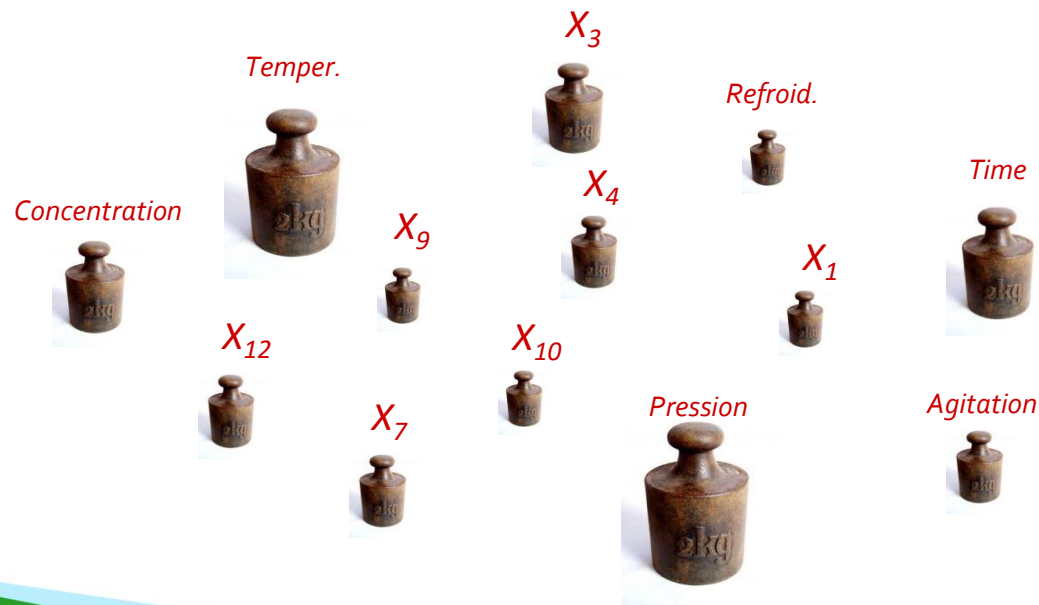
# Screening of factors

- ➔ Strategy allowing to quickly identify the few **really important factors** (**h**) among a lot of **potentially influential factors** (**k**)

$$h \ll k$$



## Estimation of the "weight" of the factors



# Screening of factors

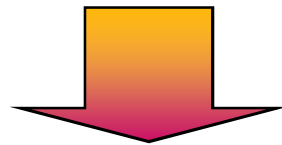
→  → Example:

Study of a tightness "test bed"

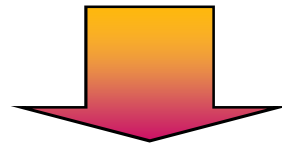


Objective :

The "test bed" are carefully considered. Every "bed" is tested and calibrated to evaluate their measure uncertainty. Before calculating this uncertainty, we must know the variation sources that are influential on the measure of the friction coefficient.



Identification of **the influential factors** on the uncertainty of measure



To know the "**weight**" of each possible source of variation

 **Example:**

**FACTORS :**

- Threading
- Nut quality
- Degreasing
- Boring
- Length
- Control
- Nut preparation
- Tightening speed
- Amplitude setting
- Programming
- Operator

**RESPONSES :**

- Friction coefficient



To know the "**weight**" of each factor

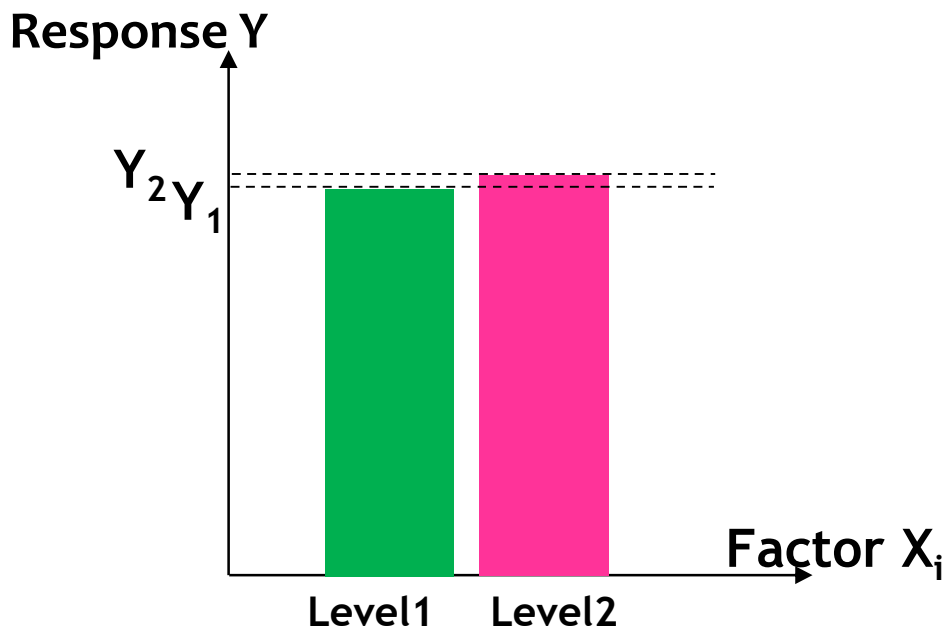
## Experimental domain :

	<b>FACTORS</b>	<b>Level 1 (-)</b>	<b>Level 2 (+)</b>
<b>U<sub>1</sub></b>	Threading	before	after
<b>U<sub>2</sub></b>	Nut quality	10	8
<b>U<sub>3</sub></b>	Degreasing	Ac.Et	Et.pet
<b>U<sub>4</sub></b>	Boring	No	Yes
<b>U<sub>5</sub></b>	Length	30	50
<b>U<sub>6</sub></b>	Control	Yes	No
<b>U<sub>7</sub></b>	Nut preparation	untreated	scrubbed
<b>U<sub>8</sub></b>	Tightening speed	5	12
<b>U<sub>9</sub></b>	Amplitude setting	Regular	Shift
<b>U<sub>10</sub></b>	Programming	1	2
<b>U<sub>11</sub></b>	Operator	A	B

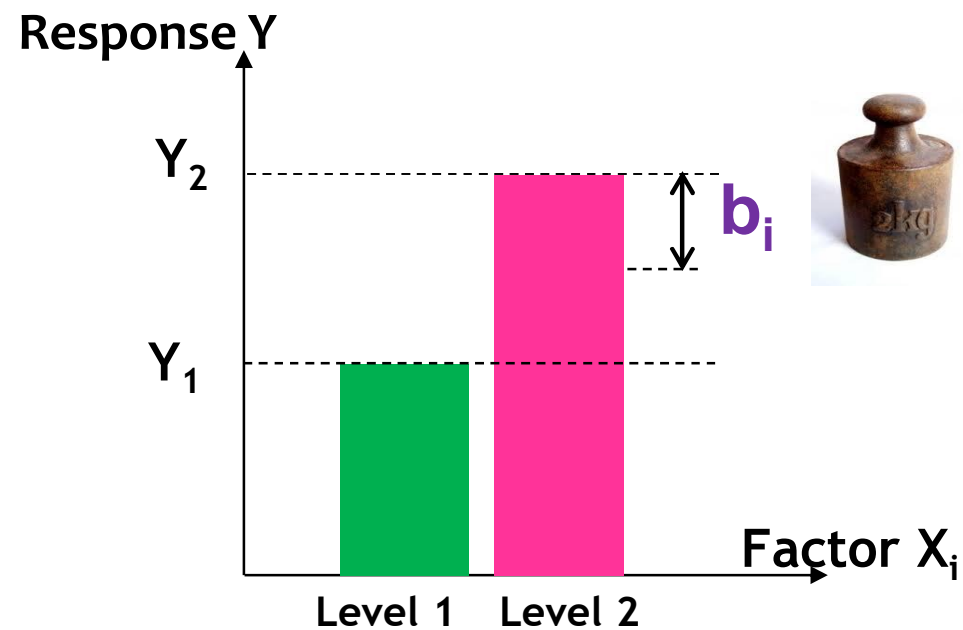


To know the "**weight**" of each factor, for these levels

The experimental strategy aims **to link** the variation of the **Responses** ( $Y_i$ ) to the variation of the **Factors** ( $X_i$ )



$$b_i \approx 0$$

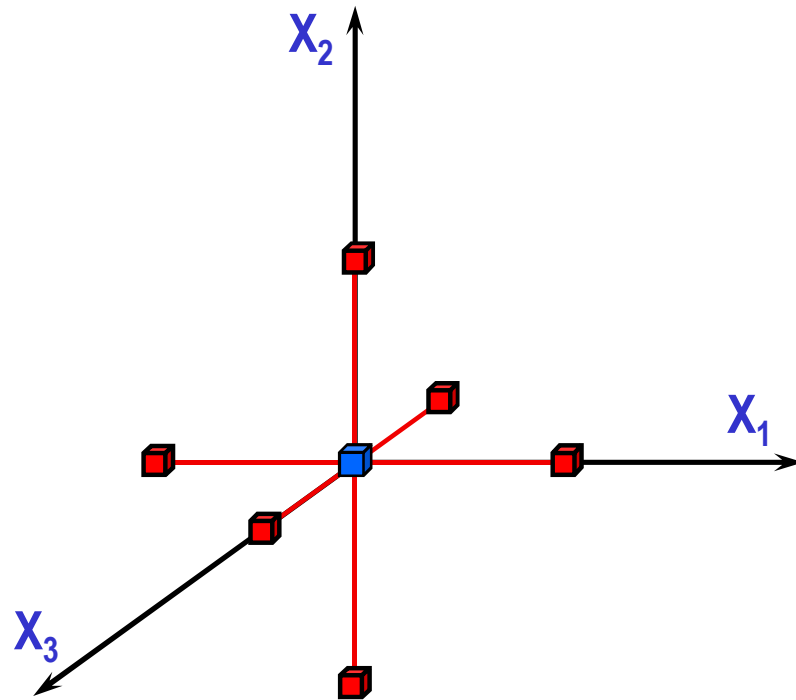


$$b_i \neq 0$$

$b_i$ : "weight" of the factor  $X_i$

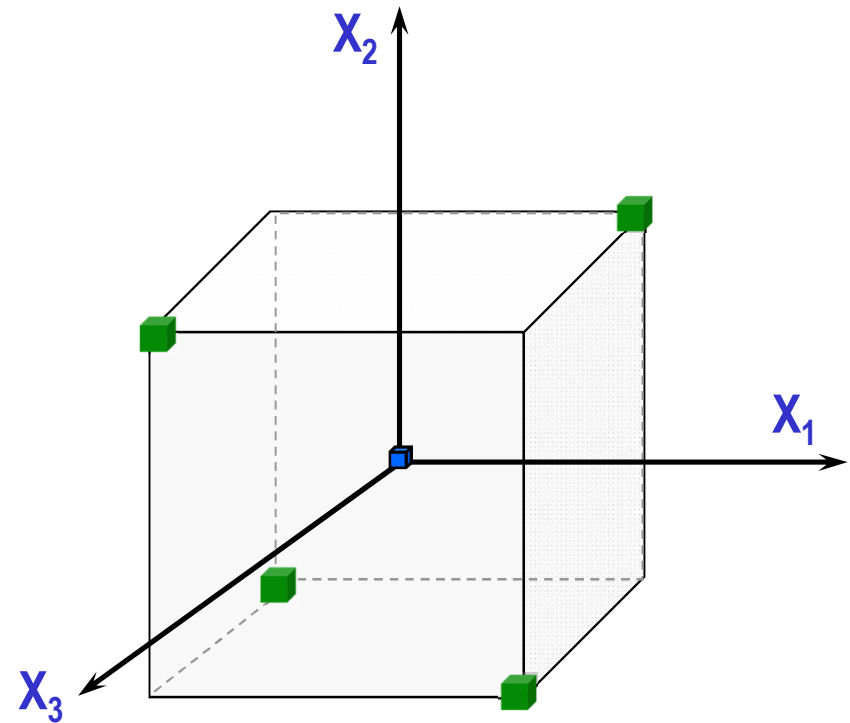
**Example** : 3 factors ( $X_1, X_2, X_3$ )

"1 Factor At a Time"



6 experiments

"Experimental design"



4 experiments

$b_i$  : "Effect" of  $X_1, X_2$  and  $X_3$



 **Case study**

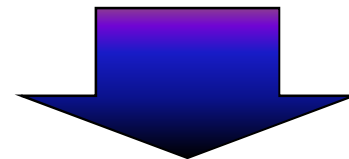
**Optimal experimental strategy**



*Experimental design to "weigh"  
11 factors with 2 levels,  
with an optimal quality and a minimum number of  
experiments*

**Optimal design of experiments**

**➔ 12 experiments**



**$b_i$  : "weight" of the factors**

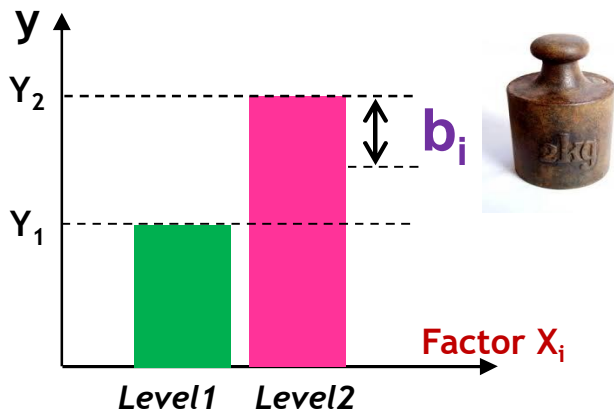
*( $b_1, b_2, b_3, b_4, b_5, b_6, b_7, b_8, b_9, b_{10}$  and  $b_{11}$ )*

# Screening design $2^{11}/12$

$N^{\circ}$	$X_1$	$X_2$	$X_3$	$X_4$	$X_5$	$X_6$	$X_7$	$X_8$	$X_9$	$X_{10}$	$X_{11}$
1	+	+	-	+	+	+	-	-	-	+	-
2	-	+	+	-	+	+	+	-	-	-	+
3	+	-	+	+	-	+	+	+	-	-	-
4	-	+	-	+	+	-	+	+	+	-	-
5	-	-	+	-	+	+	-	+	+	+	-
6	-	-	-	+	-	+	+	-	+	+	+
7	+	-	-	-	+	-	+	+	-	+	+
8	+	+	-	-	-	+	-	+	+	-	+
9	+	+	+	-	-	-	+	-	+	+	-
10	-	+	+	+	-	-	-	+	-	+	+
11	+	-	+	+	+	-	-	-	+	-	+
12	-	-	-	-	-	-	-	-	-	-	-

# Experimentation

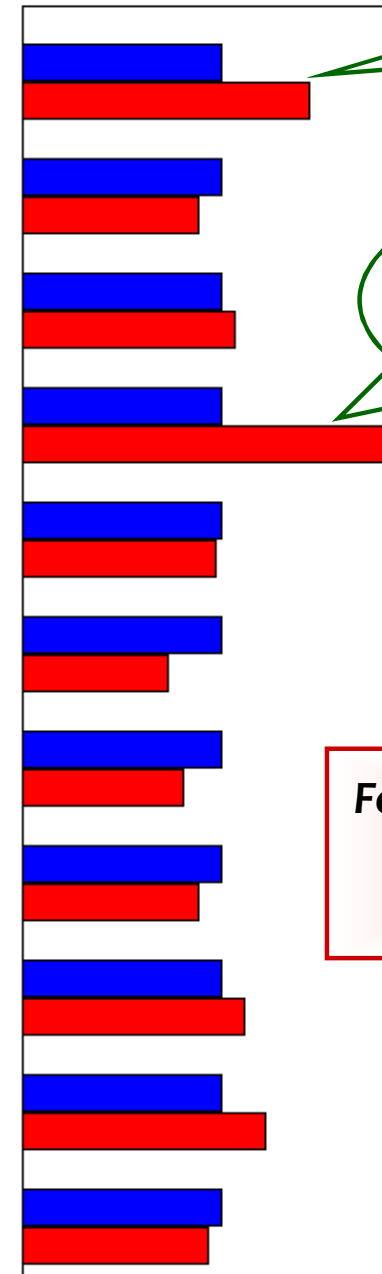
<i>N</i>	<i>U</i> <sub>1</sub>	<i>U</i> <sub>2</sub>	<i>U</i> <sub>3</sub>	<i>U</i> <sub>4</sub>	<i>U</i> <sub>5</sub>	<i>U</i> <sub>6</sub>	<i>U</i> <sub>7</sub>	<i>U</i> <sub>8</sub>	<i>U</i> <sub>9</sub>	<i>U</i> <sub>10</sub>	<i>U</i> <sub>11</sub>	<i>Coeff.</i>
1	after	10	Ac.Et	yes	50	no	untreated	5	regular	2	A	1.178
2	before	10	Et.pet	no	50	no	scrubbed	5	regular	1	B	1.139
3	after	8	Et.pe	yes	30	no	scrubbed	12	regular	1	A	1.171
4	before	10	Ac.Et	yes	50	yes	scrubbed	12	shift	1	A	1.165
5	before	8	Et.pet	no	50	no	untreated	5	shift	2	A	1.152
6	before	8	Ac.Et	yes	30	no	scrubbed	12	shift	2	B	1.168
7	after	8	Ac.Et	no	50	yes	scrubbed	12	regular	2	B	1.158
8	after	10	Ac.Et	no	30	no	untreated	12	shift	1	B	1.152
9	after	10	Et.pet	no	30	yes	scrubbed	5	shift	2	A	1.164
10	before	10	Et.pet	yes	30	yes	untreated	12	regular	2	B	1.172
11	after	8	Et.pet	yes	50	yes	untreated	5	shift	1	B	1.184
12	before	10	Ac.Et	no	30	yes	untreated	5	regular	1	A	1.152



## Estimation of the "weight" $b_i$

Factor	<u>Coefficient</u>
<b>Cst =</b>	1.1629
<b><math>b_1 =</math></b>	0.0049
<b><math>b_2 =</math></b>	-0.0013
<b><math>b_3 =</math></b>	0.0007
<b><math>b_4 =</math></b>	0.0101
<b><math>b_5 =</math></b>	-0.0002
<b><math>b_6 =</math></b>	-0.0029
<b><math>b_7 =</math></b>	-0.0021
<b><math>b_8 =</math></b>	-0.0013
<b><math>b_9 =</math></b>	0.0012
<b><math>b_{10} =</math></b>	0.0024
<b><math>b_{11} =</math></b>	-0.0008

Threading  
before  
after  
Nut quality  
10  
8  
Degreasing  
Ac.Et  
Et.pet  
Boring  
No  
Yes  
Length  
30  
50  
Control  
Yes  
No  
Nut preparation  
untreated  
scrubbed  
Tightening speed  
5  
12  
Amplitude setting  
regular  
shift  
Programming  
1  
2  
Operator  
A  
B



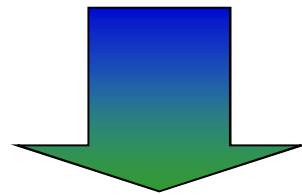
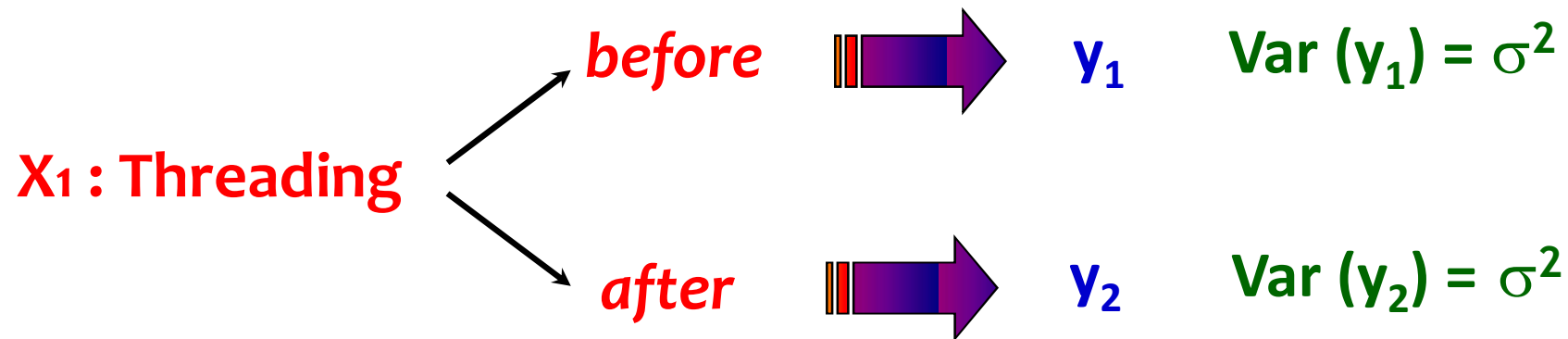
Threading is influential

"boring" induce an important variation of the friction coefficient

For the other factors, the 2 levels are equivalent

The quality of the information is better !!!

"1 factor at a time"



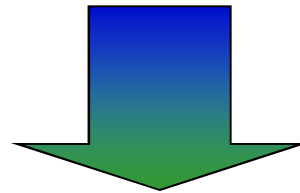
**22 experiments**

$$b_{(\text{threading})} = (y_2 - y_1)/2$$

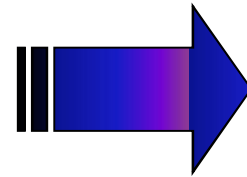
$$\text{Var}[b_{(\text{threading})}] = (\sigma^2 + \sigma^2)/4 = \sigma^2/2$$

# Design of experiments 2<sup>11</sup>//12

**"all the factors together"**



$b_j$



12 experiments

$$\text{Var}[b_j] = \sigma^2 / 12$$

***12 experiments***

The quality of the information is better !!!



# Screening of factors

→ Example: numerical experiments

## Calculation code of Infrared signature\*

### Objective :

*Ranking the factors according to their influence on the dispersion of the signature, considering different scenarios.*

*The aim of this step is the selection of the most important factors for a later step.*



To know the "**weight**" of each factor

\*results from ONERA

## FACTORS :

- Flight altitude  
*2 levels*
- Flight path  
*2 levels*
- Atmosphere model  
*2 levels*
- Day time  
*2 levels*
- Flight attitude  
*3 levels*
- Paint emissivity  
*3 levels*
- Meteo visibility  
*3 levels*

## RESPONSES :

- spectral band II
- spectral band III



To know the "**weight**" of each factor, for these 2 or 3 levels

 **Example**

Optimal experimental strategy



Experimental design to "weigh"

4 factors with 2 levels and 3 factors with 3 levels,  
with an optimal quality and a minimum number of  
experiments

*Optimal asymmetrical screening design of  
experiments*

➔ 16 experiments



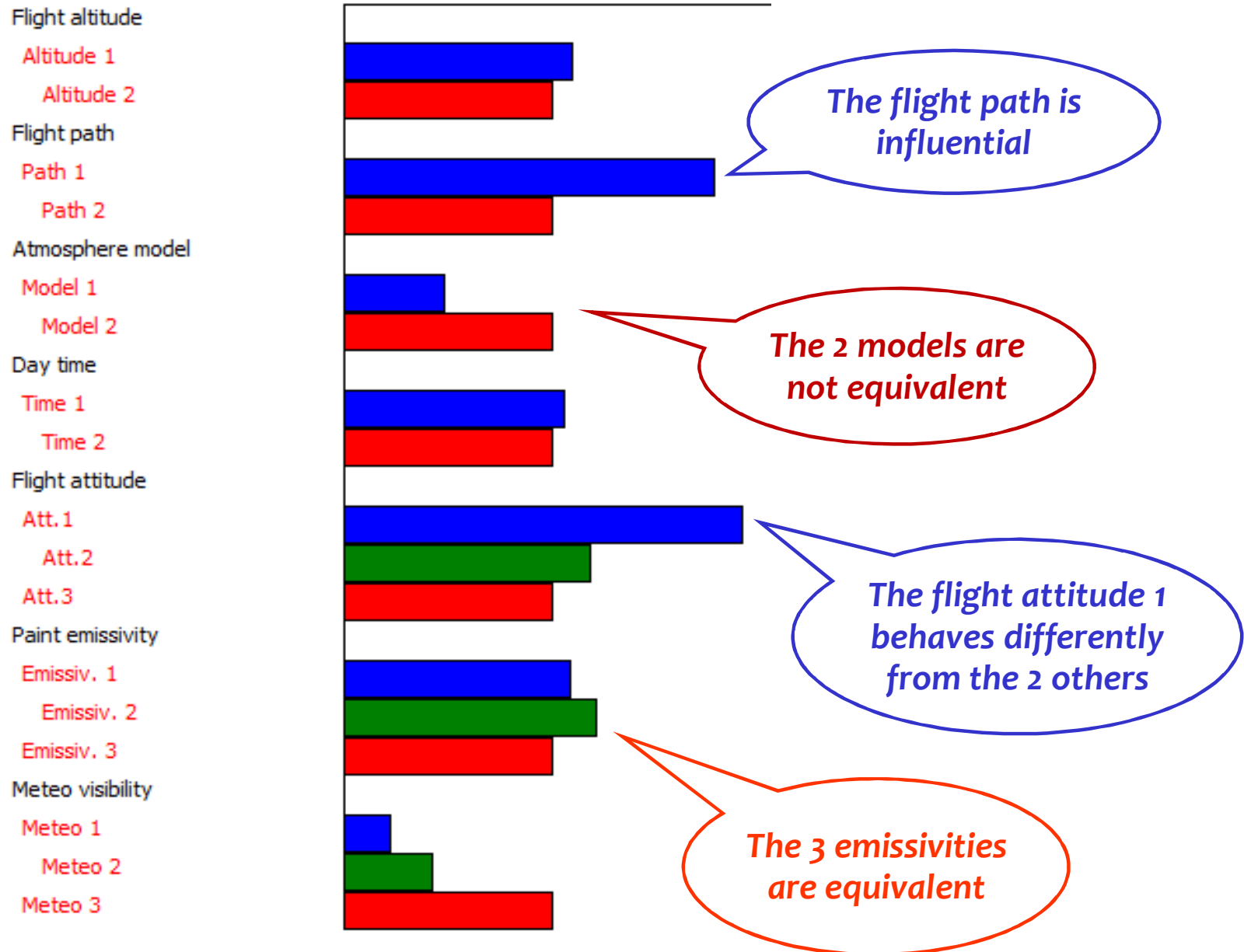
# Design $2^4 3^3 // 16$

$N^\circ$	$X_1$	$X_2$	$X_3$	$X_4$	$X_5$	$X_6$	$X_7$
1	1	1	1	1	1	1	1
2	1	1	1	2	2	2	2
3	1	1	1	1	3	3	3
4	1	1	1	2	1	1	1
5	2	1	2	1	2	3	1
6	2	1	2	2	1	1	3
7	2	1	2	1	1	1	2
8	2	1	2	2	3	2	1
9	1	2	2	1	3	1	2
10	1	2	2	2	1	3	1
11	1	2	2	1	1	2	1
12	1	2	2	2	2	1	3
13	2	2	1	1	1	2	3
14	2	2	1	2	3	1	1
15	2	2	1	1	2	1	1
16	2	2	1	2	1	3	2

# Numerical Experimentation

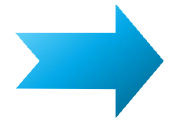
N°	Flight Altitude	Flight path	Atmosphere model	Time	Attitude	Emissiv. peintures	Meteo. Visibility	Log (Bande II)
1	Alt 1	Path 1	Model 1	Time 1	Att. 1	Emissiv. 1	Meteo 1	-8.260
2	Alt 1	Path 1	Model 1	Time 2	Att. 2	Emissiv. 2	Meteo 2	-8.780
3	Alt 1	Path 1	Model 1	Time 1	Att. 3	Emissiv. 3	Meteo 3	-8.600
4	Alt 1	Path 1	Model 1	Time 2	Att. 1	Emissiv. 1	Meteo 1	-8.460
5	Alt 2	Path 1	Model 2	Time 1	Att. 2	Emissiv. 3	Meteo 1	-8.720
6	Alt 2	Path 1	Model 2	Time 2	Att. 1	Emissiv. 1	Meteo 3	-7.200
7	Alt 2	Path 1	Model 2	Time 1	Att. 1	Emissiv. 1	Meteo 2	-7.760
8	Alt 2	Path 1	Model 2	Time 2	Att. 3	Emissiv. 2	Meteo 1	-8.760
9	Alt 1	Path 2	Model 2	Time 1	Att. 3	Emissiv. 1	Meteo 2	-9.288
10	Alt 1	Path 2	Model 2	Time 2	Att. 1	Emissiv. 3	Meteo 1	-8.740
11	Alt 1	Path 2	Model 2	Time 1	Att. 1	Emissiv. 2	Meteo 1	-8.500
12	Alt 1	Path 2	Model 2	Time 2	Att. 2	Emissiv. 1	Meteo 3	-8.600
13	Alt 2	Path 2	Model 1	Time 1	Att. 1	Emissiv. 2	Meteo 3	-8.300
14	Alt 2	Path 2	Model 1	Time 2	Att. 3	Emissiv. 1	Meteo 1	-10.150
15	Alt 2	Path 2	Model 1	Time 1	Att. 2	Emissiv. 1	Meteo 1	-9.960
16	Alt 2	Path 2	Model 1	Time 2	Att. 1	Emissiv. 3	Meteo 2	-9.140

# Interpretation



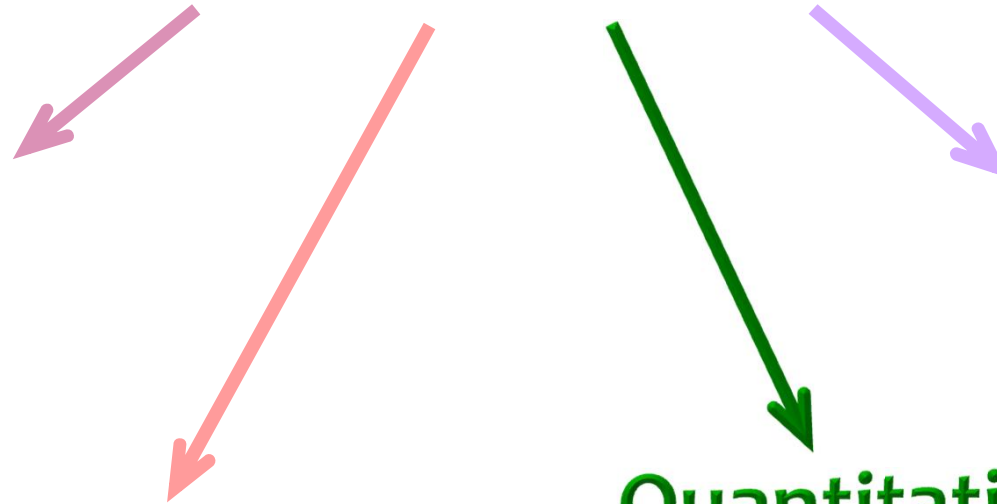


To elaborate the experimental strategy



*to choose an appropriate design of experiments  
in accordance with*

# THE OBJECTIVES

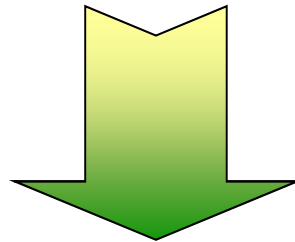


Quantitative  
study of factors

*Possible interactions between  
the factors*

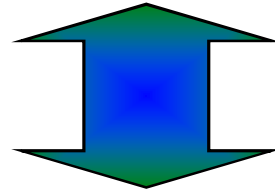
# Quantitative study of factors

We take into account the possibility of interaction effects between the factors

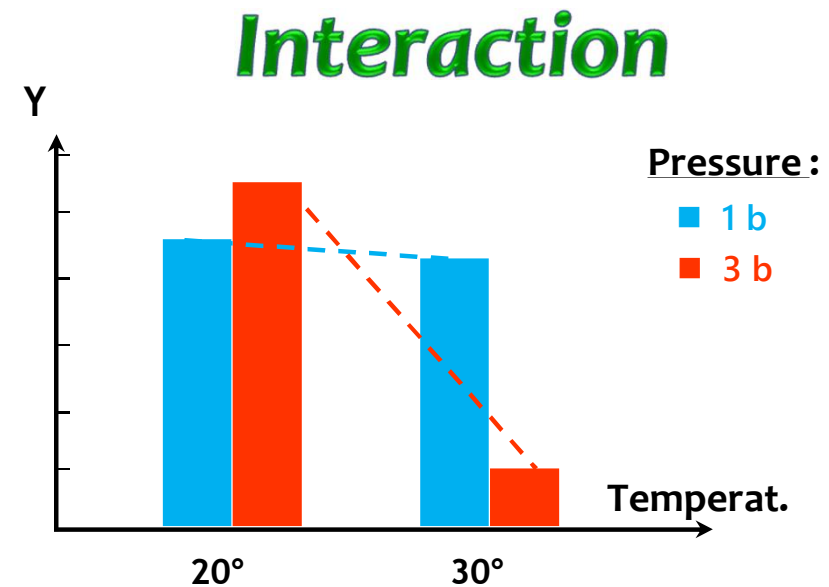
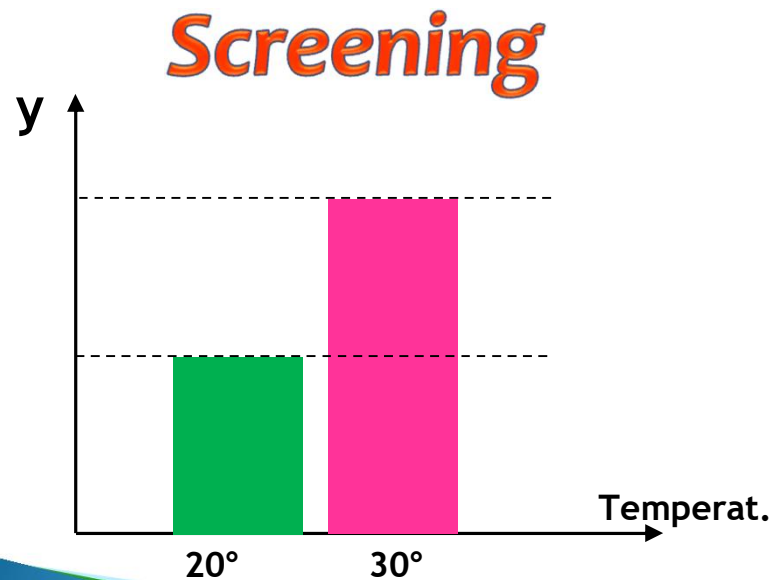


*The effect of a factor can be different according to the value of another factor*

The effects of the factors  $X_i$  et  $X_j$  are **independant** if the effect of the factor  $X_i$  **doesn't depend on the value** of the factor  $X_j$



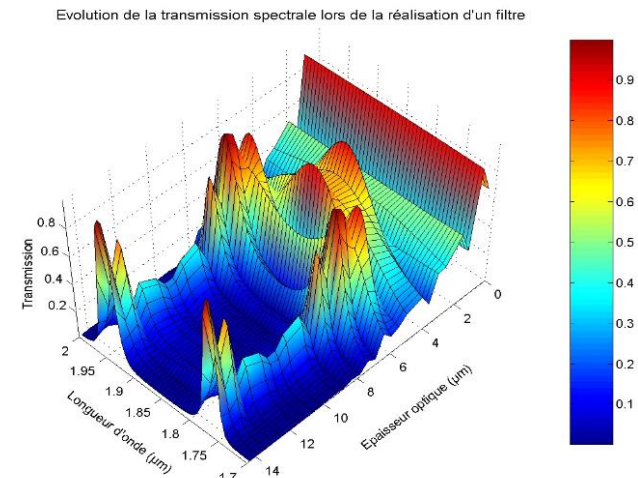
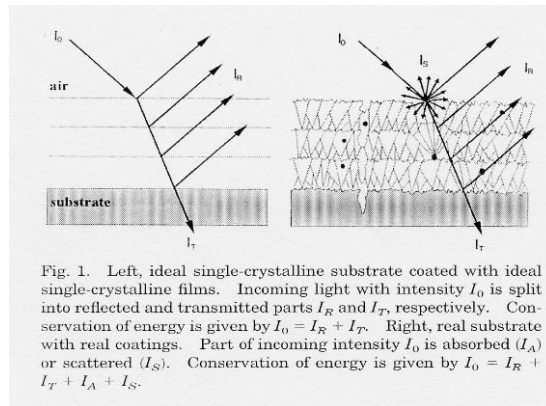
The effects of the factors  $X_i$  et  $X_j$  are **dependant** if the effect of the factor  $X_i$  **depends on the value of the** factor  $X_j$



# Quantitative study of factors



## Sensitivity analysis of interference optical filters\*:



**29-layers optical filter:**

Substrate/HLHL4HLHLH L HLHL4HLHLH L HLHL4HLHLH/air

2 materials → 2 refractive index values

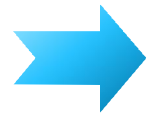
H : high refractive index value  $n_H$ , L : low refractive index  $n_L$

Desired optical properties → Perfect filter

**29 parameters:**

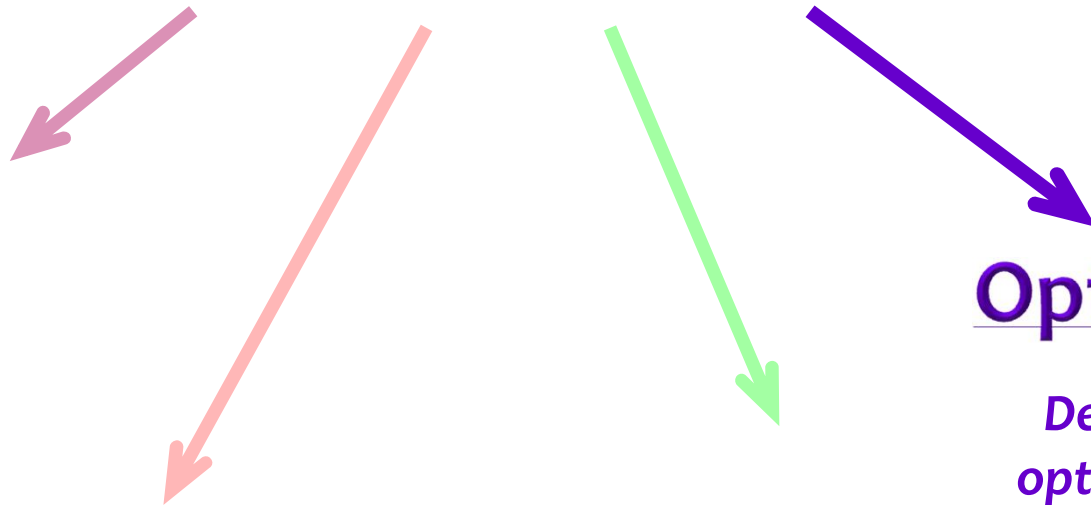
Variation of refractive index values: +/- x%

To elaborate the experimental strategy



*to choose an appropriate design of experiments  
in accordance with*

# THE OBJECTIVES



Optimization

*Definition of the  
optimal conditions*

*Quantitative study of the responses :*

# *Optimization*

To search for the optimum of one or several responses in the domain of interest



*To know, in the whole experimental domain, the value of the experimental response(s)*

## What is our purpose ?

To be able to estimate the value of experimental response *in any point within the experimental domain of interest*



To find, *if it exists*, the **domain** where all the experimental responses respect constraints imposed by specifications



**Region of acceptable compromise**

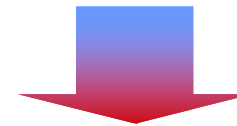
# We must do experiments



To predict the response value *in any point within the experimental domain of interest*

Mathematical model

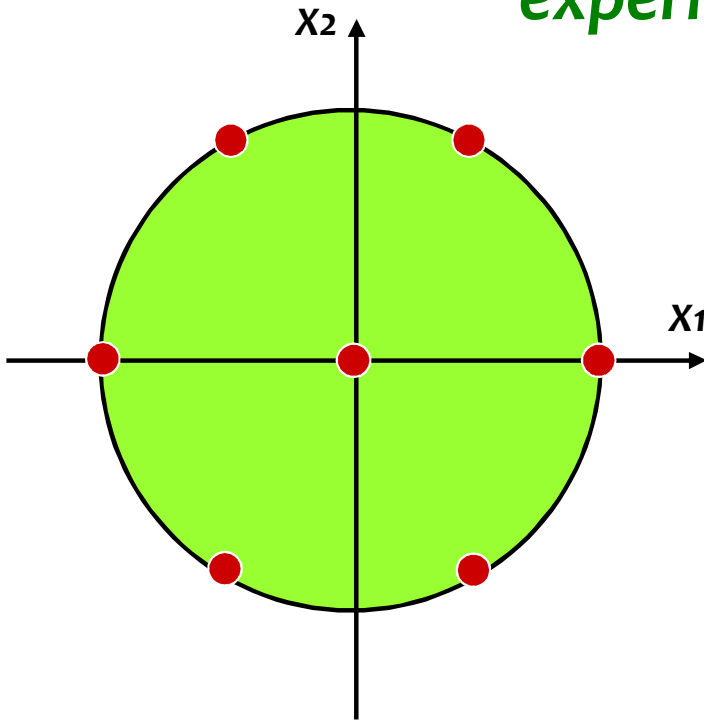
$$Y = f(X_i)$$



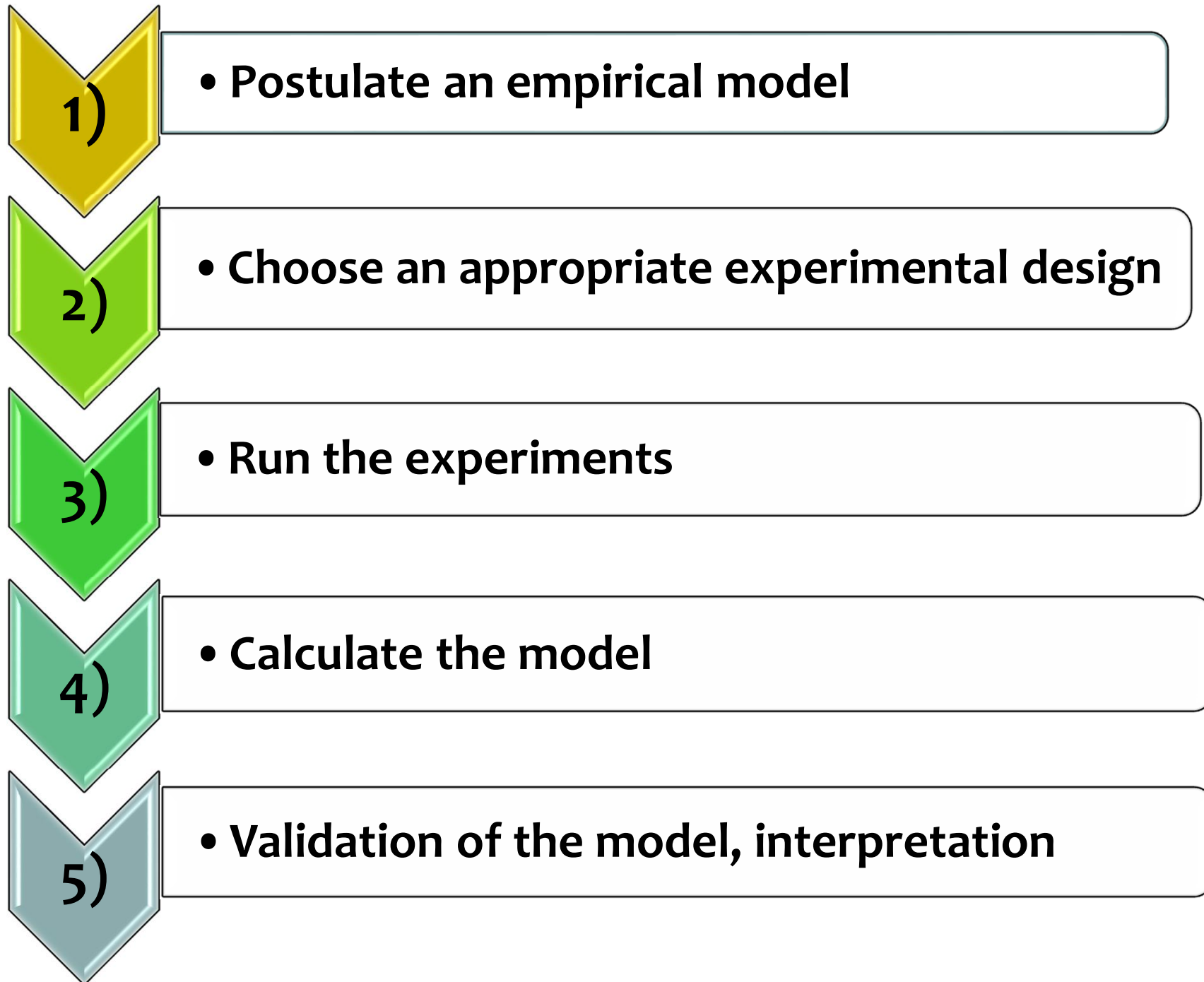
Experimental design



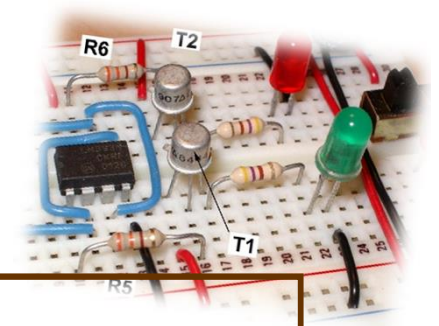
If the phenomenon is well represented by the model, *it will be possible to accurately predict, in any point of the experimental domain, the value of the studied response.*







→ Example : optimization of a process LPVD



FACTORS :

-Temperature TEOS:

62° → 78°C

- Flow TEOS:

55 → 105 ml

- Pressure:

180 → 320 mT

RESPONSES :

- Run uniformity

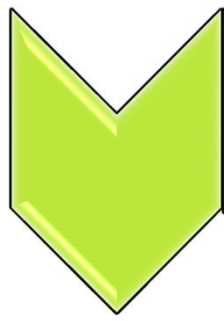
- Speed

- Thickness

- Wafer uniformity



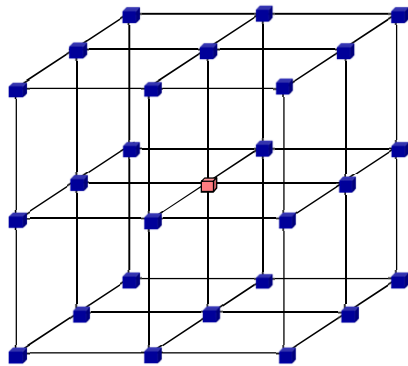
To determine the optimal conditions to have the **best compromise** between the different responses



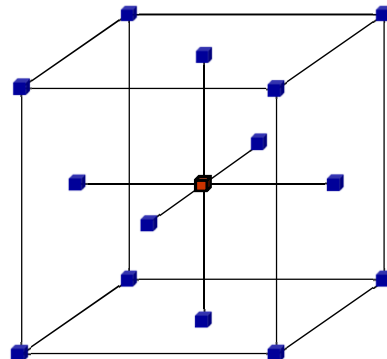
**The appropriate experimental design depends on the postulated model**

for example,

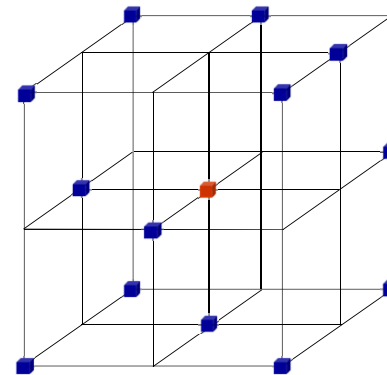
Optimal designs of experiments for a 2<sup>nd</sup> order polynomial model (3 factors)



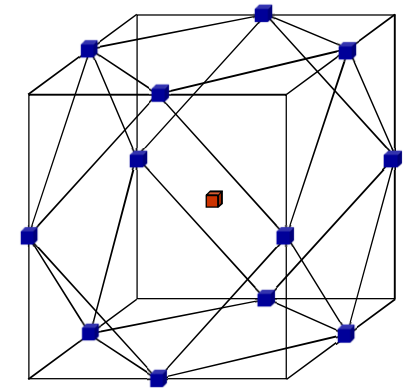
FACTORIAL 3<sup>k</sup>



FFC



HOKE D6



BOX-BEHNKEN

1)

- Postulate a model

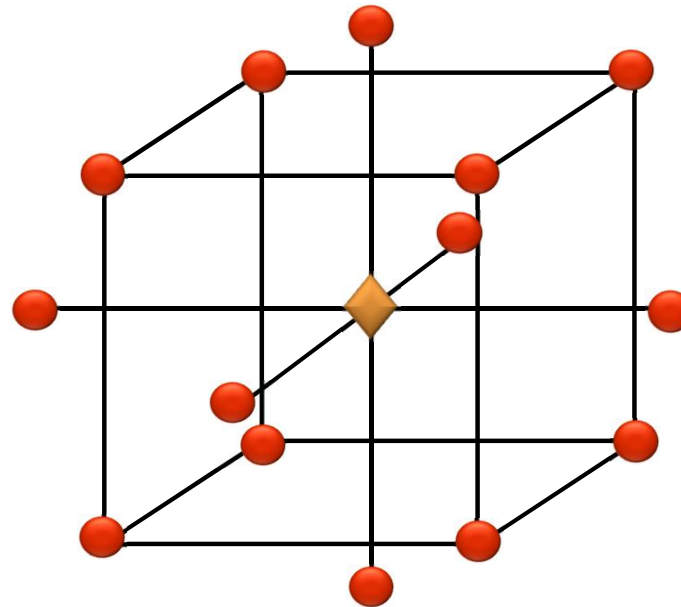
2<sup>nd</sup> order polynomial model :

$$Y = b_0 + b_1 X_1 + b_2 X_2 + b_3 X_3 + b_{11} X_1^2 + b_{22} X_2^2 + b_{33} X_3^2 \\ + b_{12} X_1 X_2 + b_{13} X_1 X_3 + b_{23} X_2 X_3$$

2)

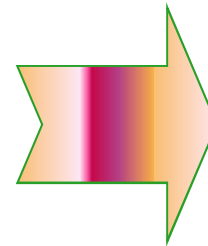
- an appropriate experimental design

15 experiments



## Experimental design

N°	X1	X2	X3
1	-1,00	-1,00	-1,00
2	1,00	-1,00	-1,00
3	-1,00	1,00	-1,00
4	1,00	1,00	-1,00
5	-1,00	-1,00	1,00
6	1,00	-1,00	1,00
7	-1,00	1,00	1,00
8	1,00	1,00	1,00
9	-1,41	0,00	0,00
10	1,41	0,00	0,00
11	0,00	-1,41	0,00
12	0,00	1,41	0,00
13	0,00	0,00	-1,41
14	0,00	0,00	1,41
15	0,00	0,00	0,00



## Experimentation

U1 °C	U2 ml	U3 m T
65	64	200
75	64	200
65	96	200
75	96	200
65	64	300
75	64	300
65	96	300
75	96	300
63	80	250
77	80	250
70	58	250
70	102	250
70	80	179
70	80	320
70	80	250

3)

- Run the experiments

## Experimental Responses

N°	Y1	Y2	Y3	Y4
1	3.30	66.90	1120.7	1.64
2	2.89	65.70	1100.9	1.43
3	6.04	59.70	1000.0	2.13
4	6.69	60.23	1008.9	2.23
5	3.64	58.38	977.8	2.29
6	3.65	59.55	997.0	1.55
7	5.36	52.30	876.0	2.52
8	4.80	53.40	894.0	2.09
9	3.93	64.50	1080.0	2.03
10	2.98	54.10	905.0	1.93
11	2.42	63.90	1070.0	1.55
12	6.88	55.10	922.0	2.52
13	3.25	60.58	1015.0	2.10
14	4.72	60.50	1013.0	1.70
15	3.94	60.60	1015.0	1.86

4)

- Estimation of the coefficients

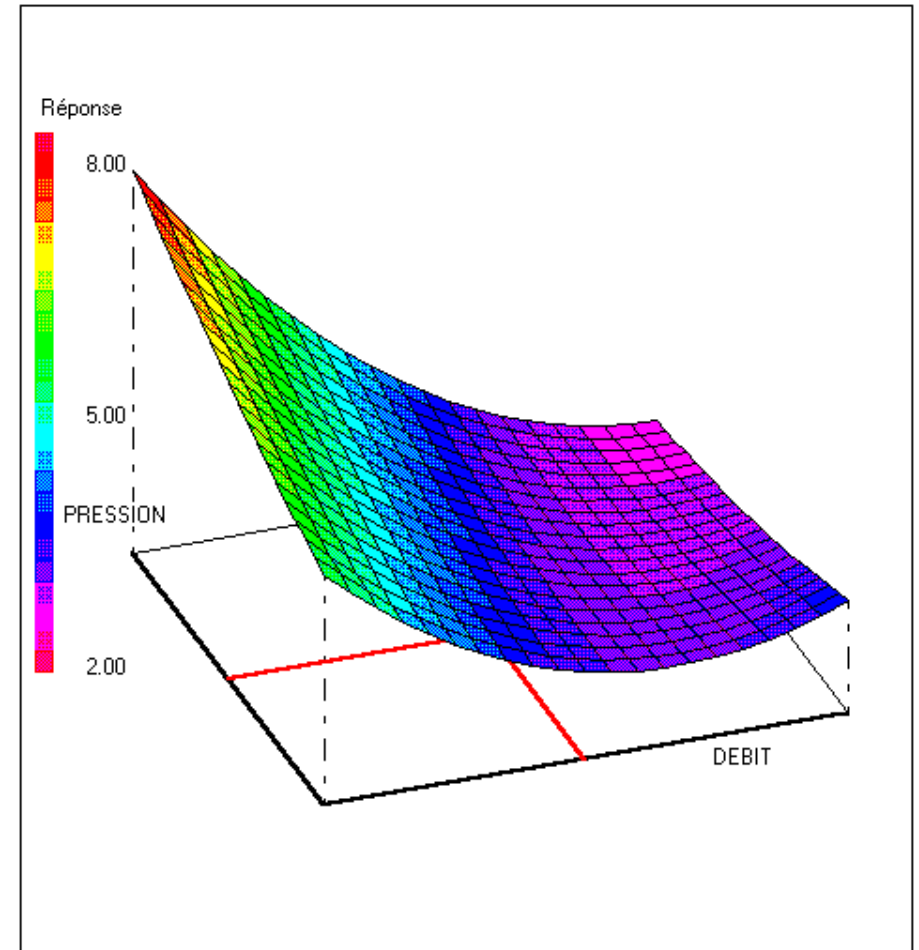
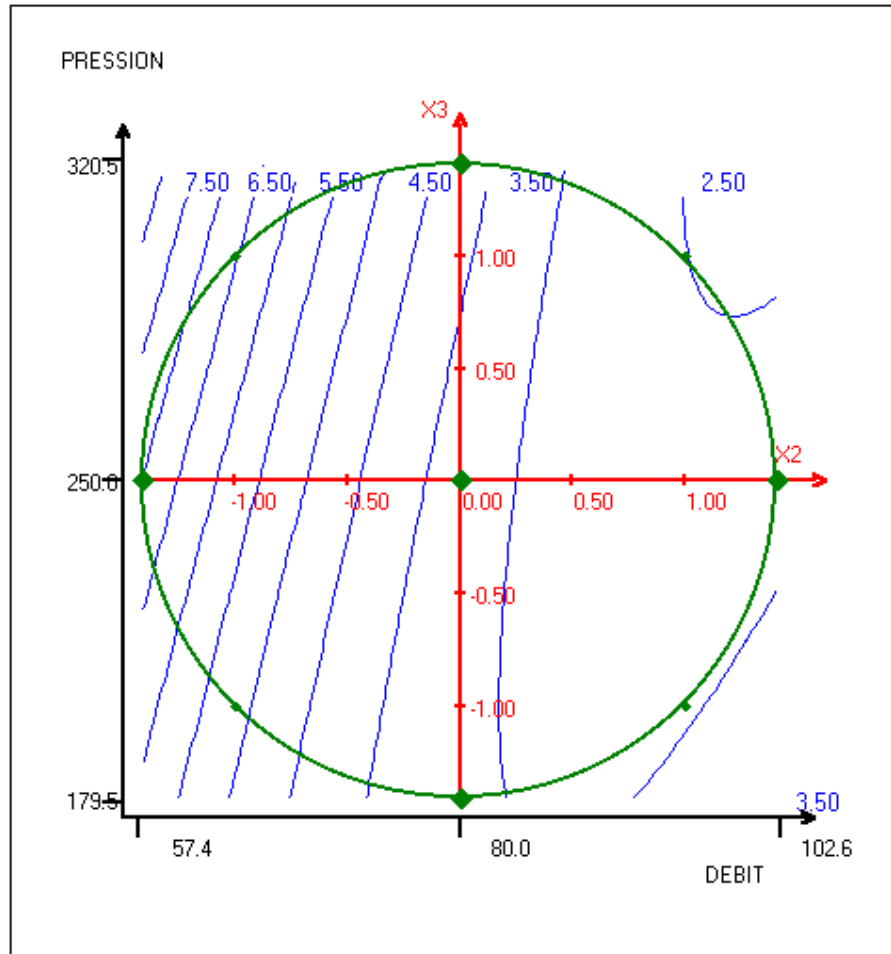
### RESPONSE : Run Uniformity ( $Y_1$ )

$$Y_1 = 3,28 + 0,12 X_1 - 1,31 X_2 + 0,24 X_3 + 0,51 X_1^2 + 0,68 X_2^2 + 0,08 X_3^2 + 0,06 X_1 X_2 - 0,10 X_1 X_3 - 0,46 X_2 X_3$$

5)

# • Interpretation

## RESPONSE : Run Uniformity ( $Y_1$ )



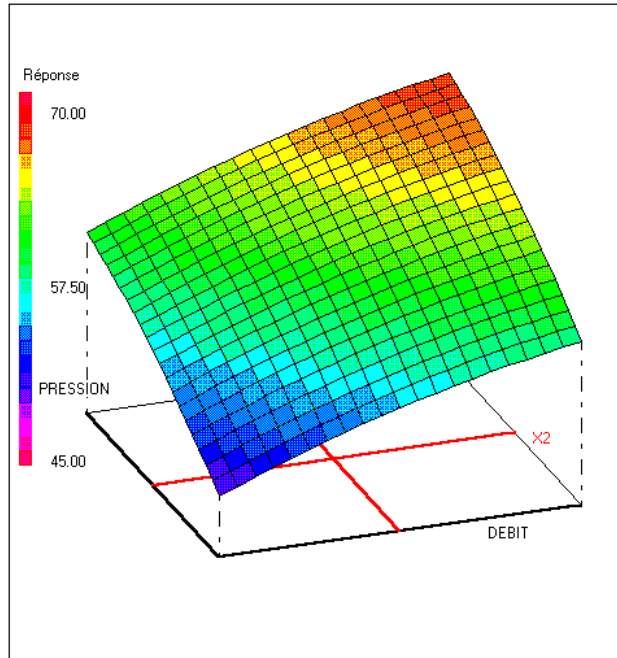
Variation of **Uniformity Run** in the plane: Flow, Pressure

$T^{\circ}\text{TEOS} = 70.0^{\circ}\text{C}$

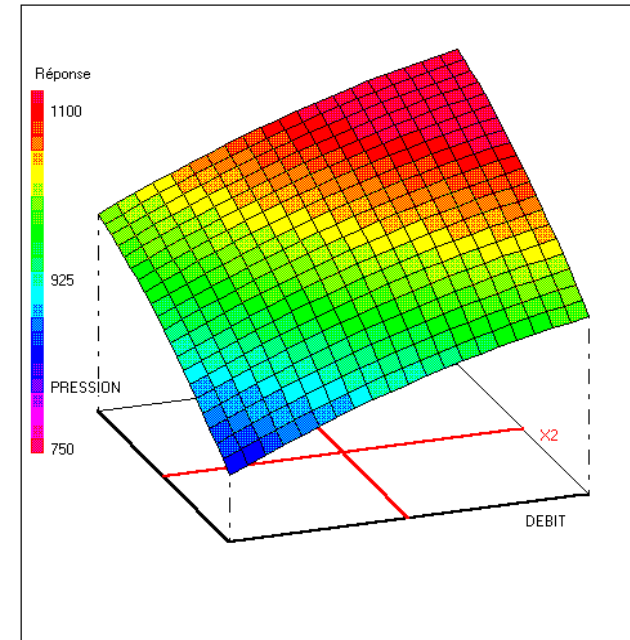


5)

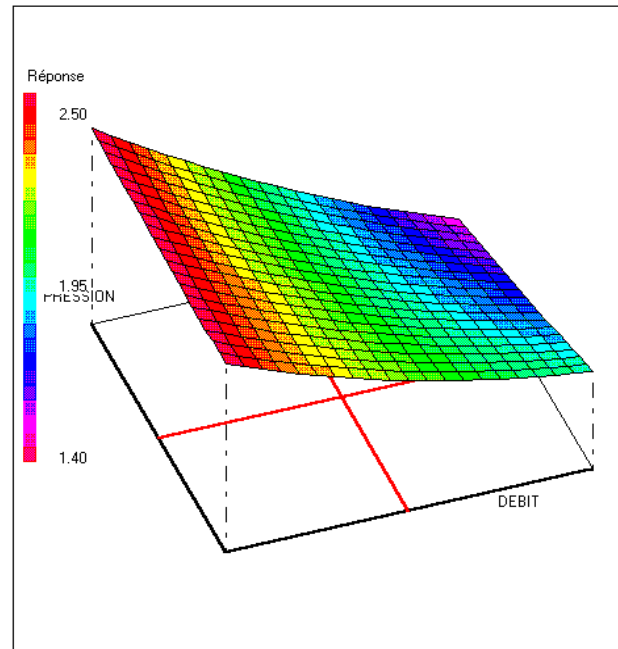
# • Interpretation



Speed (Y<sub>2</sub>)



Thickness (Y<sub>3</sub>)



Wafer Uniformity (Y<sub>4</sub>)

# The objectives :

## Experimental Responses

$\eta_1$  : Run uniformity  $\leq 4$

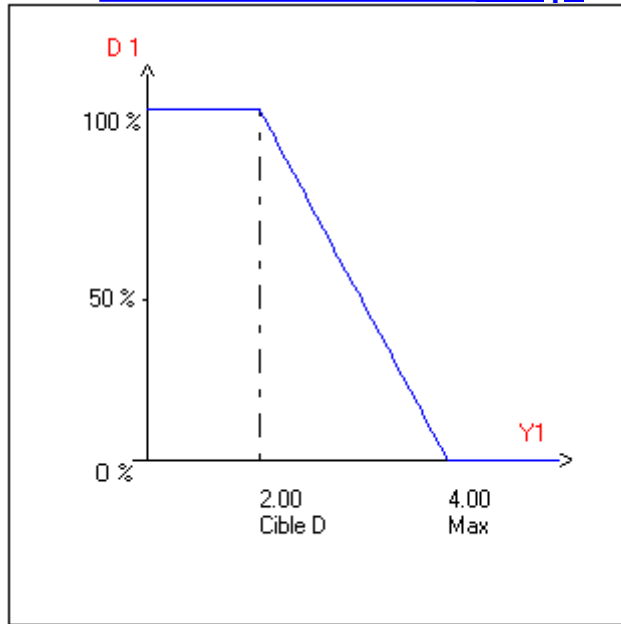
$\eta_2$  :  $62 \leq \text{Speed} \leq 72$

$\eta_3$  :  $950 \leq \text{Thickness} \leq 1050$

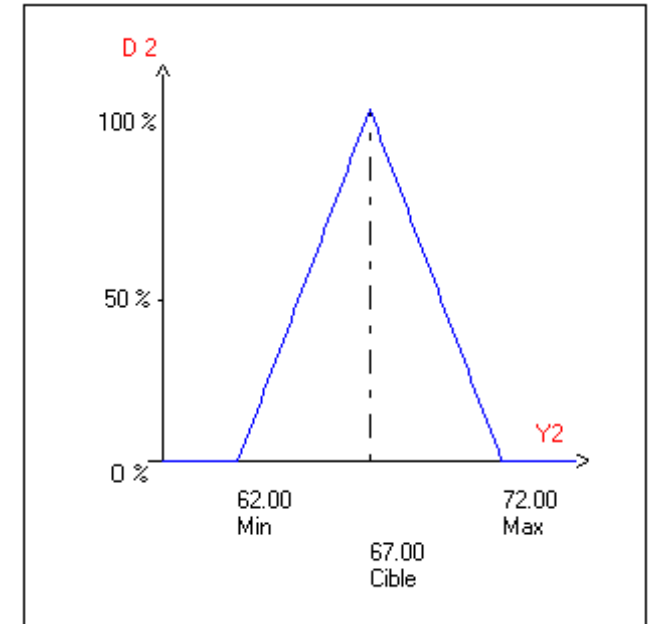
$\eta_4$  : Wafer Uniformity  $\leq 2$

# Partial desirability

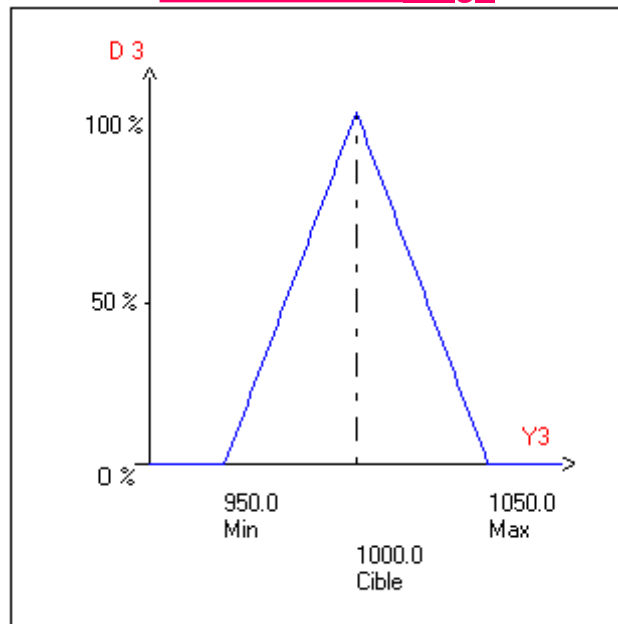
## Uniformity Run ( $Y_1$ )



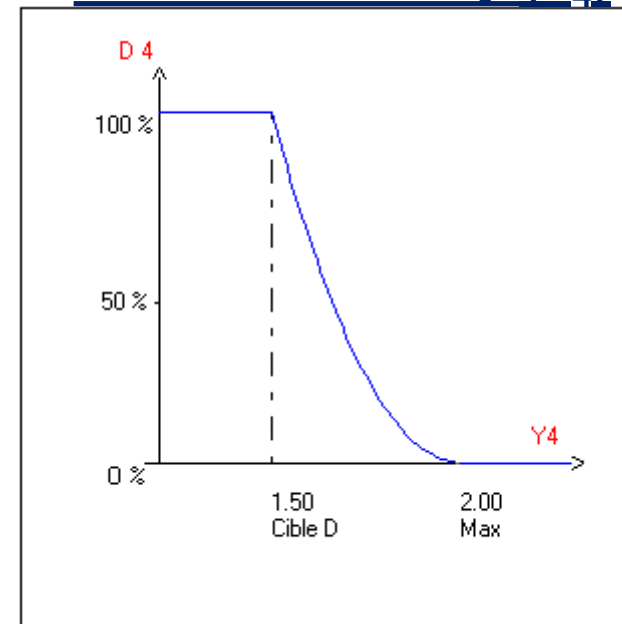
## Speed ( $Y_2$ )



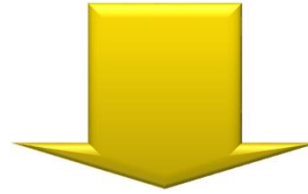
## Thickness ( $Y_3$ )



## Wafer Uniformity ( $Y_4$ )



# MULTICRITERIA OPTIMIZATION



## Global désirability

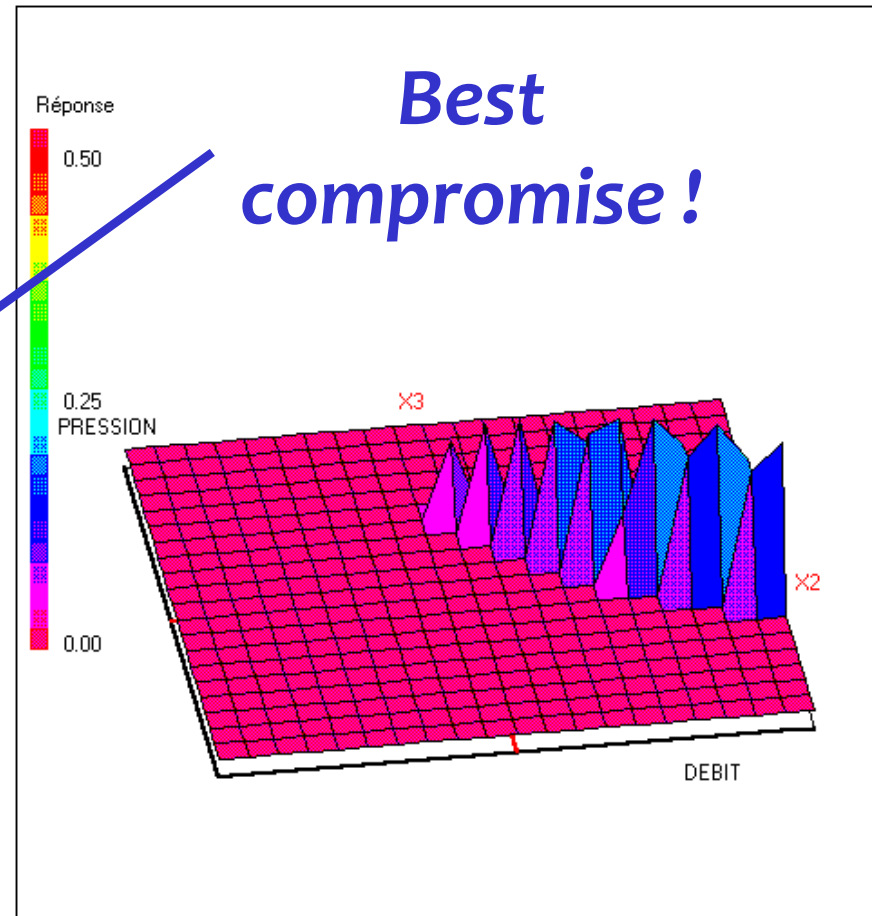
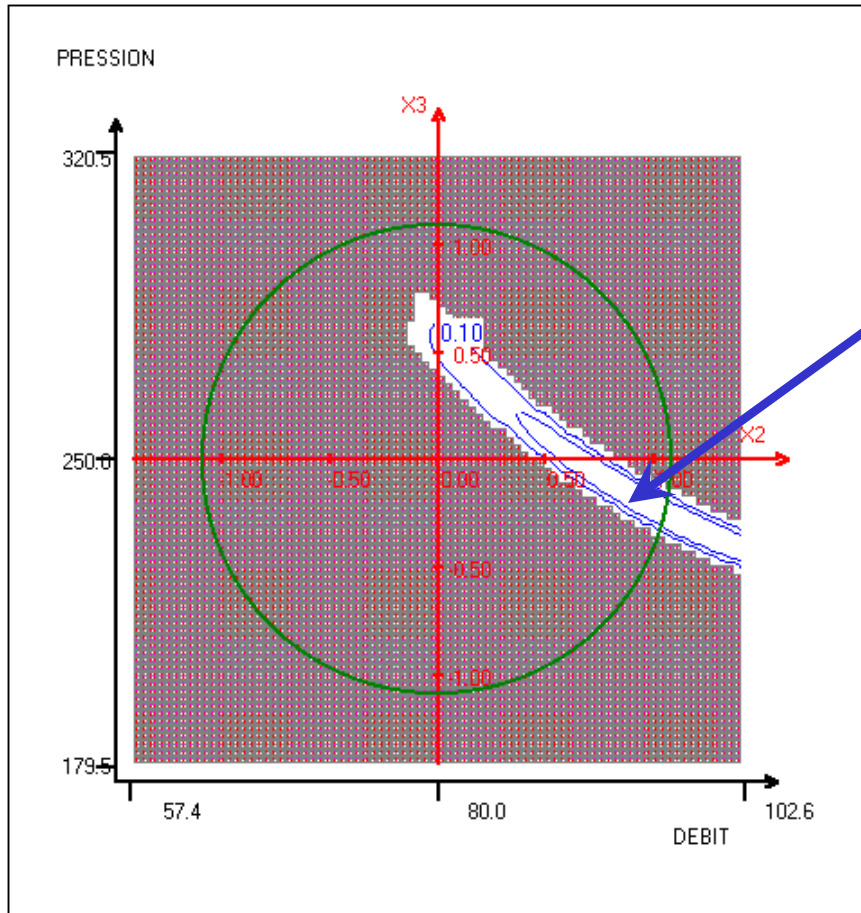
$$D = (d_1^{w_1} \times d_2^{w_2} \times \dots \times d_r^{w_r})^{1/W}$$

*$d_j$  : partial desirability*

*$w_j$  : reponse ponderation*

$$W = \sum w_j$$

# Desirability



**Best  
compromise !**

**Désirability** in the plane: Flow, Pressure

$T^{\circ}\text{TEOS} = 70.0^{\circ}\text{C}$

# Designs of experiments



**Just tools !!!**



**Thank you for your attention!**

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