

## **Experimental design (KKR031, KBT120)**

**Tuesday 11/1 2011 - 8:30-13:30 V**

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**Jan Rodmar will be available at ext 3024 and will visit the examination room ca 10:30.**

**The examination results will be available for review at the earliest Friday 28/1 2011.**

**Time for examination = 5 h**

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### **Examination aids:**

**Textbook (Douglas C. Montgomery: Design and Analysis of Experiments) with notes. No calculation examples (in book or on paper) are allowed as aid. All type of calculators are allowed. Standard Math. Tables, TEFYMA table, Beta Mathematics Handbook or Handbook of Chemistry and Physics are accepted.**

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### Problem 1 (5 credits)

An article in the Journal of Materials Engineering (1989), reported the results of an experiment to determine failure mechanisms for plasma sprayed thermal barrier coatings. The failure stress ( $\times 10^6$  Pa) for one particular coating (NiCrAlZr) under two different test conditions is as follows:

<b>Test condition 1</b>	19.8	18.5	17.6	16.7	16.7	14.8	15.4	14.1	13.6
<b>Test condition 2</b>	14.9	12.7	11.9	11.4	10.1	7.9			

**A: Construct a 95% confidence interval on the ratio of the standard deviations,  $s_1/s_2$ , of failure stress under the two different test conditions.**  
Use your answer to determine whether there is a significant difference in the variances.

**B: Find a 95% confidence interval on the difference in mean failure stress under the two different test conditions. Does the result support the claim that the first test conditions yield higher results, on the average, than the second?**

### Problem 2 (7 credits)

The surface finish of metal parts made on three machines is being studied (high value is good). An experiment is conducted in which each machine is run by three different operators and two specimens from each operator are collected and tested. Because of the location of the machines, different operators are used on each machine, and the operators are chosen at random. The data are shown in the following table:

<b>Machine</b>	<b>I</b>			<b>II</b>			<b>III</b>		
<b>Operator</b>	1	2	3	1	2	3	1	2	3
<b>Specimens</b>	79	94	46	92	85	76	88	53	46
	62	74	57	99	79	68	75	56	57

**A: Analyze the data and draw conclusions.**

**B: Estimate if there is any significant difference between the three machines. Is any machine to prefer?**

Use the significance level:  $\alpha = 0.05$

### Problem 3 (8 credits)

An engineer has performed experiments in order to develop a nitride etch process on a single-wafer plasma etcher. The four factors are, the gap between the electrodes (A), the pressure in the reactor chamber (B), the flow rate of  $C_2F_6$  (C) and the power applied to the cathode (D). The response variable is the etch rate for silicon nitride. He decided to use a  $2^{4-1}$  design with D confounded with ABC. The resulting etch rates are shown in the table:

Treatment combination	(1)	ad	bd	ab	cd	ac	bc	abcd
Etch rate (y)	550	749	1052	650	1075	642	601	729

**A: Calculate the main and interaction effects and examine their significance ( $\alpha = 0.05$ ).**

**B: Decide which of the effects in confounded alias that are important.**

**C: Calculate the residuals and plot them versus the fitted values. Comment on the plot.**

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### Problem 4 (6 credits)

The production rate (y) of a chemical process depends on temperature (T) and pressure (P). The current operating conditions are  $T = 90^\circ C$  and  $P = 20$  bar. The company wants to increase the production with the use of the method of steepest ascent.

The search starts with a  $2^2$  – factorial experiment around the operating point with the result:

T	80	100	80	100	90
P	10	10	30	30	20
y	11	0	29	6	12

**A: Fit a first-order model  $y = \beta_0 + \beta_1x_1 + \beta_2x_2 + \varepsilon$ , to these data and determine the gradient direction.**

**$x_1$  och  $x_2$  are coded T resp. P,**

Six experiments are then performed with the following result:

T	64.5	47.5	39	30.5	43.25	34.75
P	38	50	56	62	53	69
y	43	58	72	62	65	68

**B: Which of these experiments lie on the path of steepest ascent?  
How should one continue with the optimization?**

### Problem 5 (8 credits)

Extraction of nisin in aqueous two-phase systems has been investigated. The nisin recovery (y) depends on the two regressor variables concentration (%) of PEG 4000 ( $x_1$ ) and concentration (%) of  $\text{Na}_2\text{SO}_4$  ( $x_2$ ). Experiments have been run according to a  $2^2$ -design with center points and the result obtained is as follow:

$x_1$	-1	1	-1	1	0	0	0	0	0
$x_2$	-1	-1	1	1	0	0	0	0	0
y	62.9	76.1	87.5	92.3	76.2	77.5	76.8	77.4	78.7

**A: Fit the model  $y = \beta_0 + \beta_1x_1 + \beta_2x_2 + \beta_{12}x_1x_2 + \varepsilon$  to the data and examine parameter significance.**

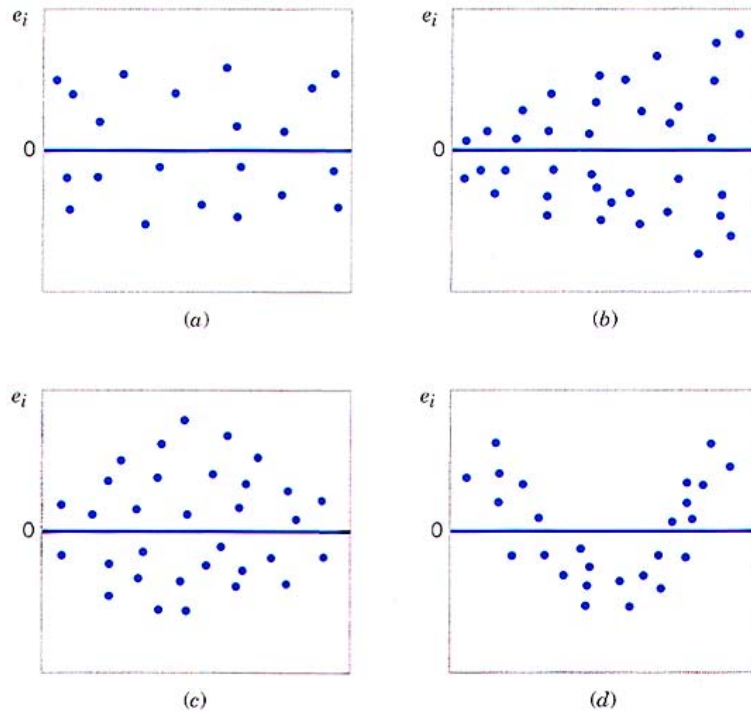
**B: Examine model significance and lack-of- fit.**

**C: Estimate the confidence interval on the mean response at the point  $x=[0.5 \ 1]$**

Use  $\alpha = 0.05$

### Problem 6 (6 credits)

The figure below shows four general patterns for residual graphs, where the residuals  $e$  are plotted against the predicted response  $\hat{Y}$  or the independent variable  $X$ .



**A: Some of the patterns are unsatisfactory. Explain what the plots indicate and suggest proper remedies to eliminate these anomalies.**

**B: Answer the following statements with true or false:**

1. The vector of fitted values  $\hat{Y}$  is always orthogonal to the vector of residuals  $e$ .
2. The  $R^2$  statistic is not the square of the correlation between the  $Y$  and  $\hat{Y}$  columns.
3. If we fit a straight line  $y = \beta_0 + \beta_1 x + \varepsilon$ , and we find that  $b_0 = 0$  exactly, then the residuals will still add to zero
4. An observation cannot be both influential and an outlier.