JMP Academic Case Study 050

Polymerization at Lohmann – Part 1

Design of Experiments, Stepwise Regression

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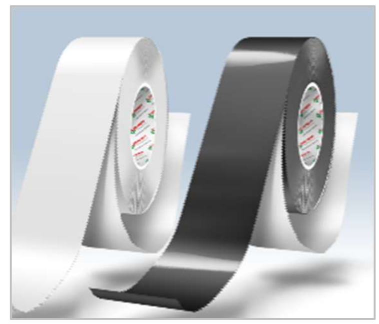
Design of Experiments, Custom Design, Stepwise Regression

Key Ideas

This case study requires the application of statistical methods to understand the process and enhance its performance. Design of experiments and Regression techniques were applied to understand and analyze the different factors impacting the process responses.

Background



Lohmann is a manufacturer of adhesive tape systems and adhesive solutions for industrial applications. The company was founded in Frankfurt am Main by Julius Lüscher in 1851. The adhesive business of the company includes a wide choice of services ranging from formula development to customized adhesive solutions. Lohmann has been serving her customers in the home appliances, electronics, transportation, graphics, building, renewables, textile, paper, hygiene, and medical markets for 170 years. The company's claim "The Bonding Engineers" is lived out by the employees in the 29 subsidiaries and distribution partners in more than 50 countries daily. The adhesive tape group is headquartered in Neuwied, Germany.

The company's focus is to develop cutting-edge and innovative products. Always bearing the customer benefit in mind, Lohmann works every day on making processes even more effective and efficient by means of adhesive solutions. The Research & Development team deals with the question of how innovative ideas can be "translated" into new applications and how existing products can be improved.

Lohmann's Research and Development experts focus on three areas that will shape the company in the future also concerning market potential and environmental issues. These are: Faster solutions for market and customer, data driven product and process development and leveraging new technologies.

Pressure Sensitive Adhesives

One of the key products of the company are Pressure-Sensitive Adhesives (PSA). It is a non-reactive type of adhesive which forms a bond when pressure is applied with a surface. No solvent, water, or heat is needed to activate the adhesive. Besides pressure, the viscoelastic behavior of the adhesive plays a major role in the degree of bonding.

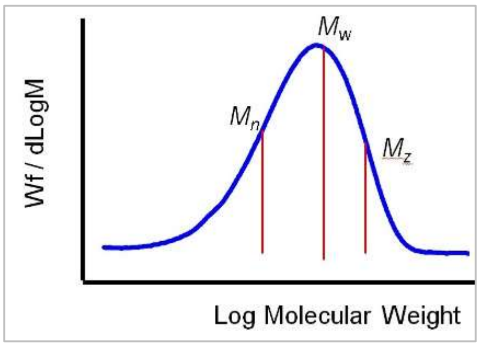
Pressure-sensitive polymer compositions have been used for over 50 years and many types of polymers can be made pressure sensitive via various formulation methods. Thus, polymers represent a vital ingredient in pressure sensitive adhesives.

For a solvent-based PSA, one class of polymers is based on the polymerization of acrylates. The basic polymer is normally formulated with further ingredients, like (optionally) resins or cross-linkers to further tune the properties of the adhesive. The performance of a PSA can be improved by tuning the properties of the basic polymer, which is applied in the adhesive formulation.

Fine-tuning of the polymer can be achieved by changing the composition of the monomers, changing the molecular weight and distribution of the molecular weight, changing the structure of the polymer and finally the type of polymerization.

Molecular Weight Distribution (MWD) or Molar Mass Distribution is a key item of information in tailoring polymer structures for different end uses. It is basically the amounts of component polymers that make up a polymer. A polymer's molecular weight (MW) is related to that of the monomer, and the number of monomers forming the polymer molecule (repeating units). Therefore, measuring MW requires measuring the MW of individual chains and the number of chains of any specific weight.

Exhibit 1 Moments related to Molecular Weight Distribution



The general distribution of polymer MW is seen in Exhibit 1. The X axis shows the log value of the Molecular weight and the Y axis shows the differential weight fraction of the log value of molecular weight.

Using statistics, three different moments can be defined for this distribution.

> **M_n** : Number average molar mass (Number Average Molecular Weight) $M_N = \frac{\sum N_x M_x}{\sum N_x}$, which defines the total weight of all molecules in a polymer sample divided by the total number of molecules present
>
> **M_w** : Mass average molar mass is the weight averaged Molecular Weight $M_W = \frac{\sum N_x {M_x}^2}{\sum N_x M_x}$, which correlates with mechanical properties, processing or viscosity
>
> **M_z** : Z average molar mass or (Z is for centrifugation) $M_W = \frac{\sum N_x {M_x}^3}{\sum N_x {M_x}^2}$, which correlates to melt elasticity

To a large extent, the polymers' molar mass and its distribution determine the final mechanical properties.

The Task

On one hand, the R&D team has to enhance the process and product performance by understanding the relationship between the input and output variables. On the other hand, they also want to identify those key variables influencing the molecular weight distribution.

Therefore, one task is to identify the key factors influencing the molecular weight distribution. There were multiple process variables influencing the molar mass. An internal discussion with subject matter experts resulted in three continuous process variables (X1, X2 and X3) and a categorical parameter called type of the ingredient (X4). The goal is to understand the effect of these variables on the moments of molecular weight.

Design of Experiments

With the aim of identifying the key parameters affecting the molar mass, the team leveraged a designed experiment to collect the data. There are various classical designs available which can be chosen. However, the team used an optimal design tailored to their problem to size the experiment since they had limitations in the number of experimental runs

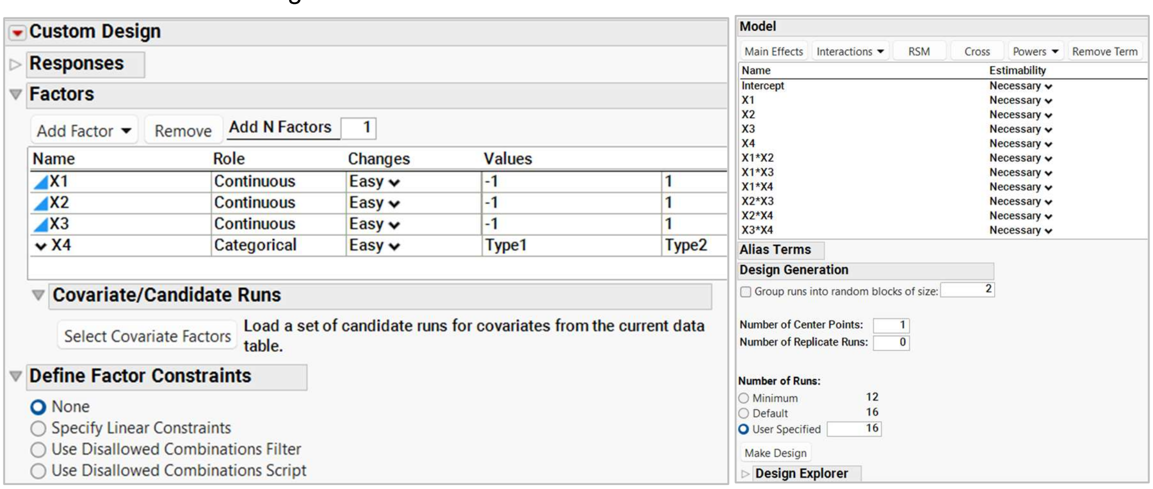
In JMP, this can be achieved by a Custom Design: If a predefined standard design doesn't fit all the conditions of the problem, one can construct such cost-effective, optimal designs that are custom-built for the specific experimental situation. The Custom Design platform creates a wide array of design types capable of addressing a wide range of experimental goals. A Custom Design has an unbeaten flexibility to manipulate factors, constraints on the design space, the model effects to estimate and other experimental conditions like the number of runs. Let us construct an optimal design using the JMP Custom Designer.

Exhibit 1 Responses and Factors of the Design Experiment

Responses
M_n
M_w
M_z

Factors	Nature	Lower Limit	Upper Limit
X1 = Process Variable 1	Continuous	-1	+1
X2 = Process Variable 2	Continuous	-1	+1
X3 = Process Variable 3	Continuous	-1	+1
X4 = Type of Ingredient	Categorical	Type 1	Type 2

Exhibit 2 Custom Design



(DOE > Custom Design > add three responses, Mn, Mw and Mz. Add 3 continuous factors and one categorical factor as shown and populate the lower and upper values. Under the Model options, choose X1, X2 and X3 from the factor list and select 2nd under the Interactions dropdown. This will add the two factor interactions of all the continuous variables selected. To add the quadratic effects, please select the 2nd option from the dropdown. Enter 1 for number of center points. Choose the default Number of Runs (16) > Make Design > Make Table) (you can set Random seed as 2024498060 with starting number as 5000, if you want to replicate the same output)

The above process will create a data table along with the details of the 16 runs with their factor settings, the team used the template to conduct all 16 experiments and saved the measurements in the response columns.

The Data **custom-design-results.jmp**

Exhibit 3 Custom Design Results

	X1	X2	X3	X4	Mn	Mw	Mz
1	1	-1	-1	Type 1	132000	928000	2250000
2	-1	0	0	Type 2	143000	992000	2250000
3	0	0	-1	Type 2	132000	1040000	2250000
4	1	1	0	Type 1	132000	1040000	2250000
5	1	-1	1	Type 2	121000	768000	1800000
6	-1	-0.428	-0.333	Type 1	143000	976000	2100000
7	1	1	-1	Type 2	143000	1136000	2400000
8	1	0	1	Type 1	121000	832000	1800000
9	-1	-1	1	Type 1	121000	816000	1800000
10	0	1	1	Type 1	132000	928000	1950000
11	0	0	0	Type 1	132000	976000	2100000
12	-1	-1	-1	Type 2	132000	960000	2250000
13	1	1	1	Type 2	132000	928000	2100000
14	-1	1	1	Type 2	154000	1024000	2100000
15	-1	-1	-1	Type 1	132000	960000	2250000
16	0.1	-1	0.02	Type 2	143000	848000	1950000

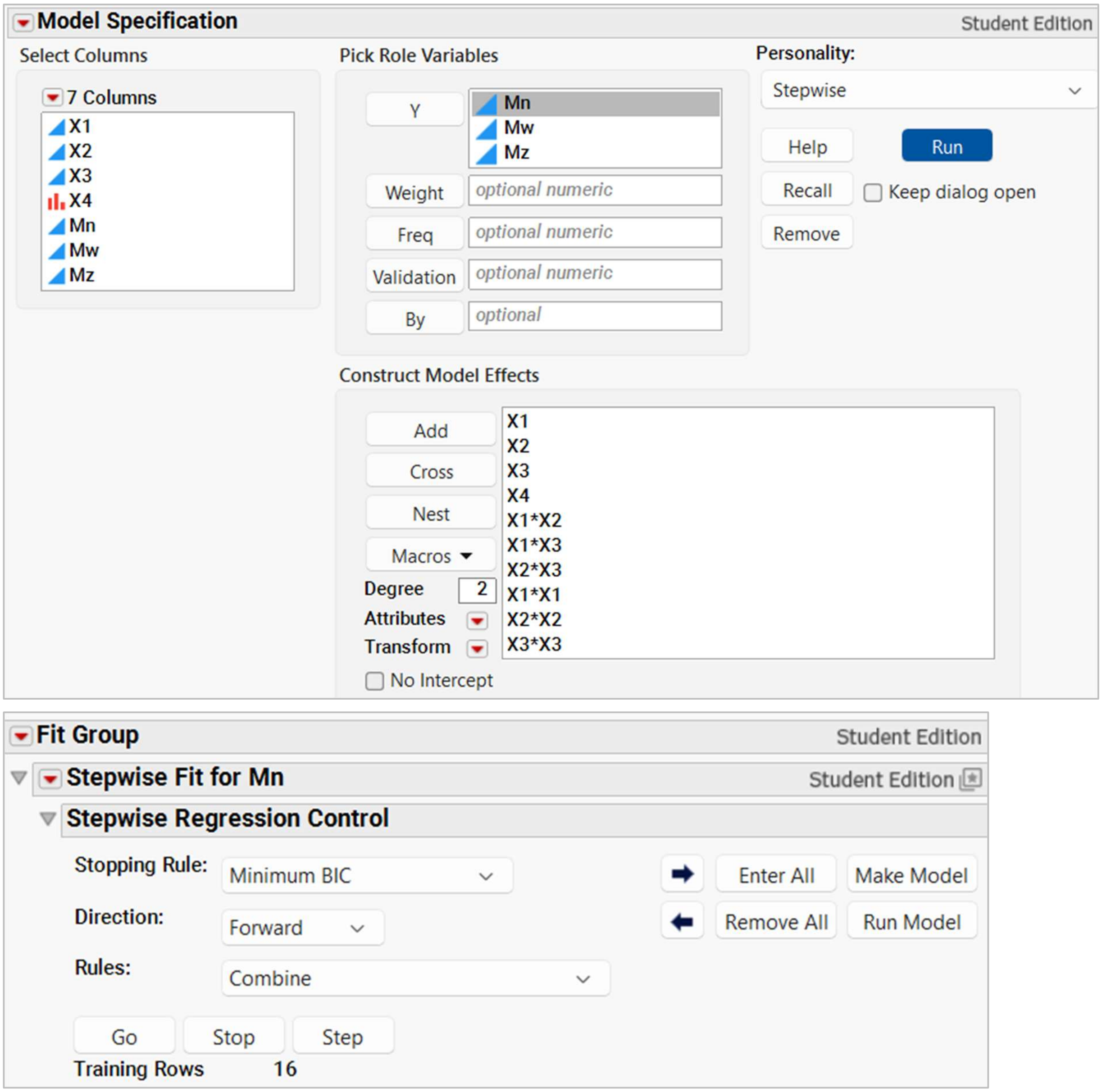
All the The results from the 16-run experiment (at randomized order) are shown in the Exhibit 3. All the variables are continuous except X4.

(Note: one of the factor combinations or the experimental run had operational challenges and the values were changed manually.(row 6 in the dataset))

Analysis

JMP's powerful Regression platform that enables the researchers or engineers to explore cause and effect relationships between the factors and responses.

Exhibit 4 Regression Analysis



(Analyze > Fit Model > Y = Mn, Mw and Mz, choose X1, X2, X3 and X4 from the columns list as model effects. Choose X1, X2 and X3 from the column list > select Factorial to Degree from the Macros dropdown. This will add the two-way interactions of all the continuous variables selected. To add the quadratic effects, please select the Polynomial to Degree to add quadratic effects of all the continuous factors > select Stepwise as the personality > Run. This will open a launch window to fit stepwise regression for each of the response variable. From the Stepwise regression Control option, Select Minimum BIC as stopping rule > Go > Once the stepwise regression is run, click Run Model. This launches the Fit Model platform for the model parametrized using the stepwise procedure. Repeat the same process to build stepwise regression models for response variables Mw and Mz.

Exhibit 5 shows the regression output of each of the response variable.

Exhibit 5   Model output

Response Mn

Effect Summary

Summary of Fit

RSquare	0.825418
RSquare Adj	0.672658
Root Mean Square Error	5249.841
Mean of Response	134062.5
Observations (or Sum Wgts)	16

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	7	1042450897	148921557	5.4034
Error	8	220486603	27560825	Prob > F
C. Total	15	1262937500		0.0150*

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|	VIF
Intercept	137100.62	2823.604	48.56	<.0001*	.
X1	-5616.619	1650.652	-3.40	0.0093*	1.1872416
X2	7149.0329	1818.051	3.93	0.0043*	1.3258901
X3	-4676.022	1758.391	-2.66	0.0288*	1.2432221
X4[Type 1]	-2262.843	1373.298	-1.65	0.1380	1.0948574
X1*X2	-5225.488	2016.179	-2.59	0.0320*	1.2538161
X2*X2	6153.0231	3291.686	1.87	0.0985	1.2647555
X3*X3	-7744.11	3171.621	-2.44	0.0405*	1.2029778

Response Mw

Effect Summary

Summary of Fit

RSquare	0.987354
RSquare Adj	0.978924
Root Mean Square Error	13839.09
Mean of Response	947000
Observations (or Sum Wgts)	16

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	6	1.3458e+11	2.243e+10	117.1157
Error	9	1723683699	191520411	Prob > F
C. Total	15	1.363e+11		<.0001*

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|	VIF
Intercept	952950.25	7055.05	135.07	<.0001*	.
X1	-32056.32	4460.826	-7.19	<.0001*	1.2477738
X2	104588.24	4905.361	21.32	<.0001*	1.3890393
X3	-92879.21	4670.663	-19.89	<.0001*	1.2622692
X1*X2	-13096.94	5323.842	-2.46	0.0362*	1.2580652
X2*X3	-16588.34	5169.116	-3.21	0.0107*	1.1734974
X1*X1	18030.456	8242.287	2.19	0.0565	1.0588596

Response Mz

Effect Summary

Summary of Fit

RSquare	0.956712
RSquare Adj	0.918836
Root Mean Square Error	54054.75
Mean of Response	2100000
Observations (or Sum Wgts)	16

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	7	5.1662e+11	7.38e+10	25.2586
Error	8	2.3375e+10	2.9219e+9	Prob > F
C. Total	15	5.4e+11		<.0001*

Parameter Estimates

Term	Estimate	Std Error	t Ratio	Prob>|t|	VIF
Intercept	2078691.6	31315.79	66.38	<.0001*	.
X1	-12905.22	17339.53	-0.74	0.4780	1.2357404
X2	111729.24	18622.71	6.00	0.0003*	1.3122171
X3	-200898.4	17471.49	-11.50	<.0001*	1.1577165
X4[Type 1]	-24480.5	14012.46	-1.75	0.1188	1.0751803
X2*X3	33871.522	20720.15	1.63	0.1407	1.2358979
X1*X1	87553.022	32505.99	2.69	0.0274*	1.0794862
X3*X3	-46572.95	32585.09	-1.43	0.1908	1.1977256

(From red triangle next to Response > Save Columns > Save Prediction Formula. This will save the Prediction Formula as a new column to the data table)

From the ANOVA tables, it can be seen that all three individual regression models are statistically significant overall. The R-square values suggest that a major portion of the response variation can be explained by the factors. Looking at the parameter estimates and their significance (P values), Exhibit 5 shows the significant effects influencing the response variables.

Exhibit 6 Statistically Significant Effects

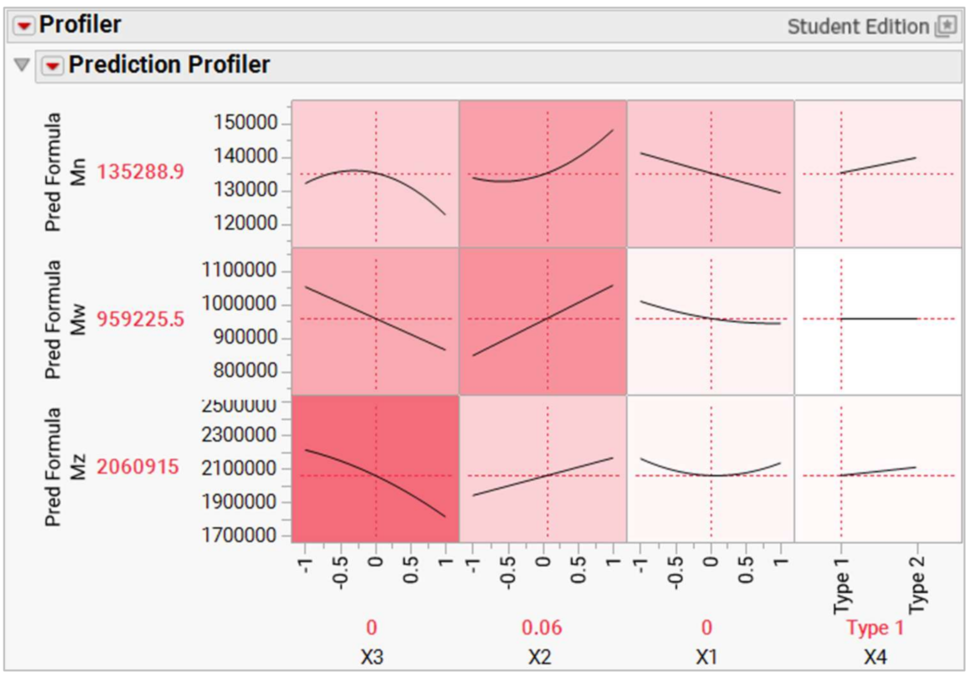
Response Variable	Statistically Significant Parameters or Factor Effects		
	Main Effects	Interaction Effects	Quadratic Effects
Mn	X1, X2, X3	X1*X2	X3*X3
Mw	X1, X2, X3	X1*X2, X2*X3	
Mz	X2, X3		X1*X1

It can be concluded that X4, our only categorical variable is not at all statistically significant when it comes to explaining the responses.

Prediction Profiler

The profilers in JMP provide highly interactive cross-sectional views of any response surface.

Exhibit 7 Prediction Profiler and Variable Importance



Variable Importance: Independent Uniform Inputs

Summary Report

Overall

Column	Main Effect	Total Effect
X3	0.438	0.46
X2	0.367	0.404
X1	0.088	0.119
X4	0.034	0.042

Pred Formula Mn

Column	Main Effect	Total Effect
X2	0.4	0.459
X1	0.203	0.262
X3	0.227	0.239
X4	0.086	0.097

Pred Formula Mw

Column	Main Effect	Total Effect
X2	0.505	0.53
X3	0.396	0.419
X1	0.039	0.058
X4	1e-17	2e-17

Pred Formula Mz

Column	Main Effect	Total Effect
X3	0.691	0.721
X2	0.195	0.225
X1	0.022	0.039
X4	0.017	0.028

(Graph > Profiler > Pred Formula Mn, Pred Formula Mw and Pred Formula Mz as Y Profiler > Ok. From the red triangle next to Prediction Profiler, select Assess Variable Importance > Independent Uniform Inputs. Click the red triangle next to Variable Importance: Independent Uniform Inputs and select Colorize Profiler.)

The report shows tables for each of the three responses. The Overall table averages the factor importance indices across responses. The factors in the Prediction Profiler have been reordered to match their order in the Overall table's Total Effect importance. Colors from red to white intensity scale are overlaid on profiler panels to reflect Total Effect importance. For example, you easily see that the most important effect is that of X3 on Mz.

Summary

This summary concludes the first part of this two-part case study series on polymerization.

Statistical Insights

This case introduced the concept of applying custom design to generate factor settings for conducting the experiments. It also involved the use of stepwise regression to analyze the experimental data to identify the key variables/factors and their effects (main effects, 2 factor interactions and quadratic effects)

Managerial Implications

Organisations always strive to improve the processes by predicting the most significant factors affecting the responses. The R&D team used structured experimentation to better understand the existing process. The unique custom design platform helped the team to extract maximum information from a small number of runs. Thus, the goal of predicting the key variables affecting the molecular weight distribution was achieved.

JMP Features and Hints

This case used the Analyze platform for data analysis and a DOE platform for creating a tailor made design accommodating all the requirements and specifications. It also leveraged Prediction Profiler to understand the interaction between response and effect.

Exercises

1. With the below information, create an experimental design using custom design platform.

Responses	Goal
Y1	Maximization
Y2	Maximization
Y3	Maximization

Factors	Nature	Lower Limit	Upper Limit
X1 = Process Variable 1	Continuous	-1	+1
X2 = Process Variable 2	Continuous	-1	+1
X3 = Process Variable 3	Continuous	-1	+1
X4 = Process Variable 4	Continuous	-1	+1
X5 = Ingredient 1	Categorical	A	B
X6 = Ingredient 2	Categorical	P	Q

   The team is interested in understanding all the main effects, two way interactions of all the continuous factors and quadratic effect of X1 and X2 only.What will be the minimum and default number of Runs without any additional center points and replicate points? (Use a random seed of 12345 to replicate the results)

2. Exercise.jmp has the experimental results for a 21 run custom design for exercise 1. Develop individual regression models for each of the responses and identify the statistically significant main effects, interaction effects and quadratic effects.