Order Experiments

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PPG Industries

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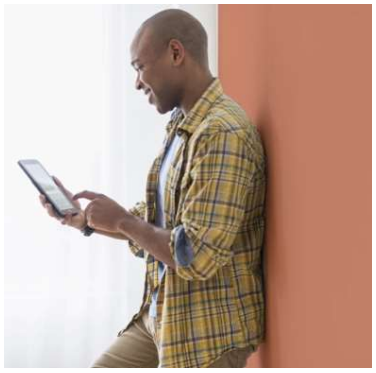
PPG: 46,600 employees protecting and beautifying our world

A global maker of paints, coatings, and specialty materials



A leader in all our markets: construction, consumer products, industrial and transportation markets and aftermarkets

Headquartered in Pittsburgh, Pennsylvania, with operations in more than 70 countries

Founded in 1883

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PPG Business Segments

Architectural Coatings

Homax

Automotive Coatings

Industrial Coatings

Packaging Coatings

Aerospace Coatings

Protective & Marine Coatings

PPG

PPG Coatings Innovation Center

- 250+ researchers
- synthesis chemists, formulators, analytical chemists, engineers

- 600+ patents in past 10 years

Ford 2016
Excellence Award

R&D 100
54 Years of Invention

Automotive News PACE AWARD
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A U.S. EPA Program

Fiat
Sustainability
Award

PPG is a coatings industry benchmark for innovation

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Today's Objectives:

- What is an Order Experiment?
- How do we design an Order Experiment?
- How should the experimental results be analyzed?
- What are the Factors and Factor Levels?

What is an Order Experiment?

An Order experiment is one in which there are multiple process steps and the order in which the steps are performed is studied.

Examples:

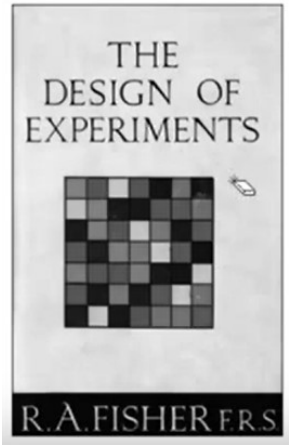
- Knee brace - The order in which the straps are tightened
- Survey - The order in which questions are asked
- Coatings - The order in which multiple coating layers are applied
- An important special case: Order-of-Addition - The order in which mixture ingredients are added
  - Paints
  - Cosmetics

  Resins/Polymers

  Pesticides

  Adhesives

  Foods



Lady Tasting Tea
$m = 2$ components,
4 replications

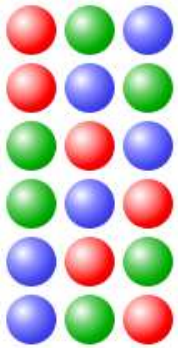
What would be the “Full Factorial” equivalent of an Order Experiment?

Full Factorial equivalent = all possible permutations

Lady tasting tea: m = 2 components:

Permutation 1: Milk ➔ Tea
Permutation 2: Tea ➔ Milk

Consider m = 3 components:



Each of the 6 rows is
a unique permutation of
the three colored balls.

What are the factors and levels in an Order Experiment?

Lady tasting tea: m = 2 components:

Permutation 1: Milk ➔ Tea
Permutation 2: Tea ➔ Milk

Milk = A
Tea = B

Run	Order	f M<T
1	MT	1
2	TM	-1

Pairwise ordering *factor*: M before T

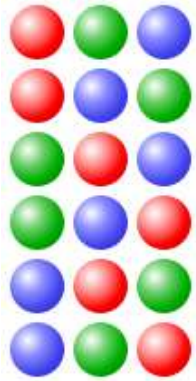
Factor Level: Does M enter before T? 1 = true, -1 = false

Just one *factor*

Consider m = 3 components:

Run

1
2
3
4
5
6



Red = R
Green = G
Blue = B

Run	Order	f R<G	f R<B	f G<B
1	RGB	1	1	?
2	RBG	1	1	?
3	GRB	-1	1	?
4	GBR	-1	-1	?
5	BRG	1	-1	?
6	BGR	-1	-1	-1

3 *factors*

Order Experiments with All Possible Permutations (Full Factorial)

number of components, m	number of pairwise factors, $\binom{m}{2}$	number of permutations, $m!$
1	-	1
2	1	2
3	3	6
4	6	24
5	10	120
6	15	720
7	21	5,040
8	28	40,320

As the number of components increases:
- pairwise ordering factors increase
- permeations increase

A new JMP Addin is available:
- All possible permutations
- Pairwise ordering factors

Addin by Bradley Jones and Joseph Morgan

Fractional Experiments?
- JMP Custom Design
- Pairwise ordering factors
- Covariate Factors

Case Study: Automotive Clearcoat

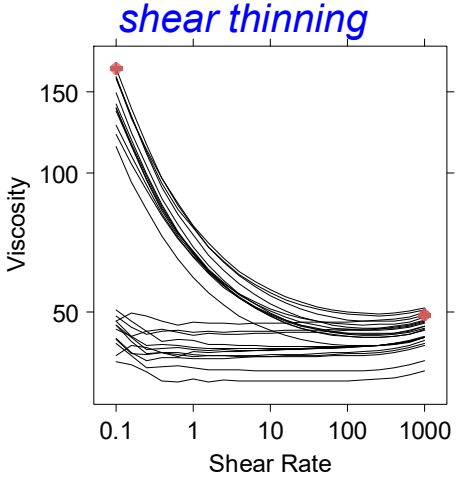
The design and analysis of order-of-addition experiments: An introduction and case study

Joseph G. Voelkel & Kevin P. Gallagher

Published online: 06 Aug 2019

Component	code	(4, 24)	(5, 15)	(6, 24)
primary binder resin	A	✓	✓	✓
secondary binder resin	B	✓	✓	✓
flow and leveling additive	C	✓	✓	✓
rheology modifier #1	D	✓	✓	✓
crosslinking resin	E		✓	✓
rheology modifier #2	F			✓

shear thinning



Four Component Experiment

24 total permutations, 6 pairwise factors

Experimental notation: Order (4, 24)

➔ Components (*m*) = 4,
➔ Runs (N) = 24

In general: Order (*m*, N)

The **order** column provides the instructions to how to run the experiment:

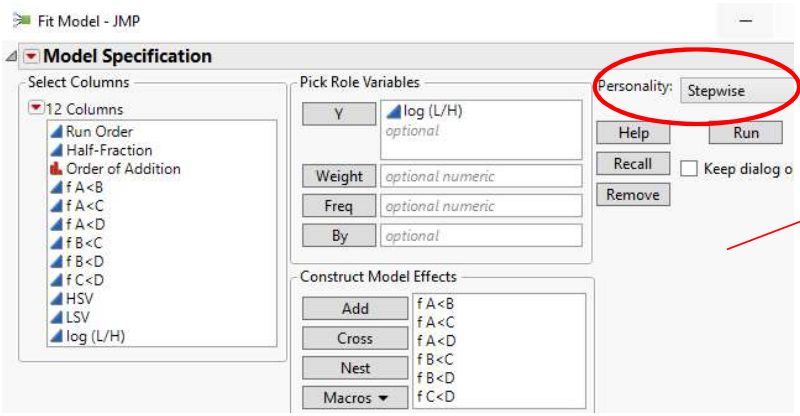
The **factor** columns used to analyze
- Forward 2-stage stepwise regression
  - Main effects first
  - 2-factor interactions with heredity

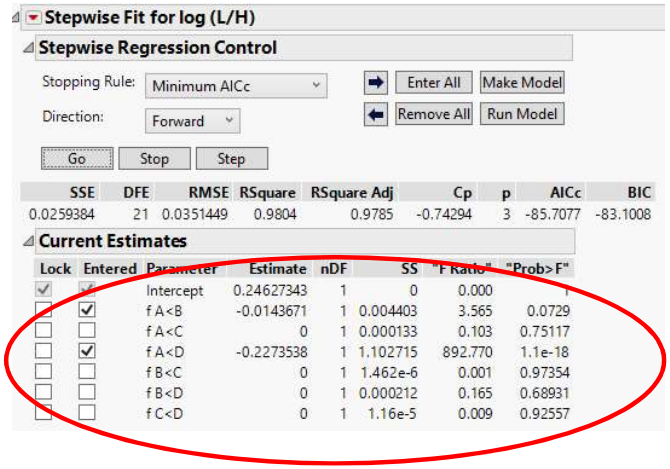
Run Order	Order	f A<B	f A<C	f A<D	f B<C	f B<D	f C<D	Y
1	BCAD	-1	-1	1	1	1	1	0.037
2	ACBD	1	1	1	-1	1	1	0.044
3	CDAB	1	-1	-1	-1	-1	1	0.414
4	BCDA	-1	-1	-1	1	1	1	0.510
5	CBAD	-1	-1	1	-1	1	1	0.004
6	ABDC	1	1	1	1	1	-1	0.009
7	DCBA	-1	-1	-1	-1	-1	-1	0.491
8	DBAC	-1	1	-1	1	-1	-1	0.436
9	BDAC	-1	1	-1	1	1	-1	0.482
10	ADBC	1	1	1	1	-1	-1	0.027
11	ACDB	1	1	1	-1	-1	1	0.033
12	DCAB	1	-1	-1	-1	-1	-1	0.476
13	BACD	-1	1	1	1	1	1	0.020
14	CADB	1	-1	1	-1	-1	1	-0.019
15	DACB	1	1	-1	-1	-1	-1	0.398
16	CBDA	-1	-1	-1	-1	1	1	0.537
17	ABCD	1	1	1	1	1	1	0.009
18	ADCB	1	1	1	-1	-1	-1	0.059
19	BDCA	-1	-1	-1	1	1	-1	0.534
20	DABC	1	1	-1	1	-1	-1	0.461
21	DBCA	-1	-1	-1	1	-1	-1	0.500
22	CDBA	-1	-1	-1	-1	-1	1	0.502
23	CABD	1	-1	1	-1	1	1	-0.038
24	BADC	-1	1	1	1	1	-1	-0.016

Order (4, 24).jmp

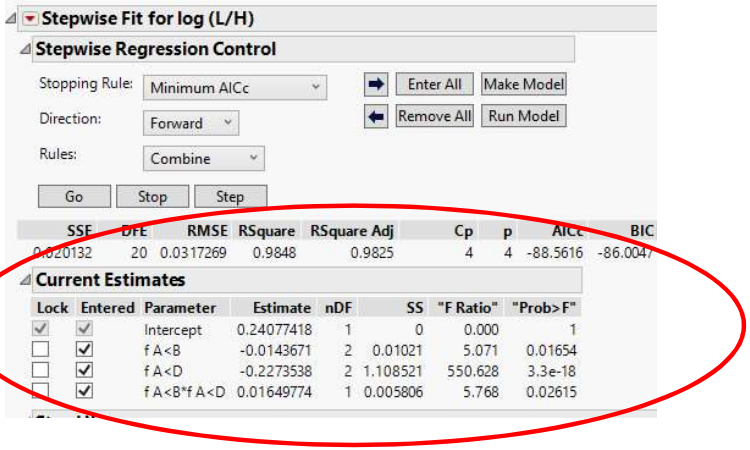
Case Study Example: Order (4, 24)

1) Stage 1: Use forward stepwise regression with only the “main effect” pairwise ordering factors

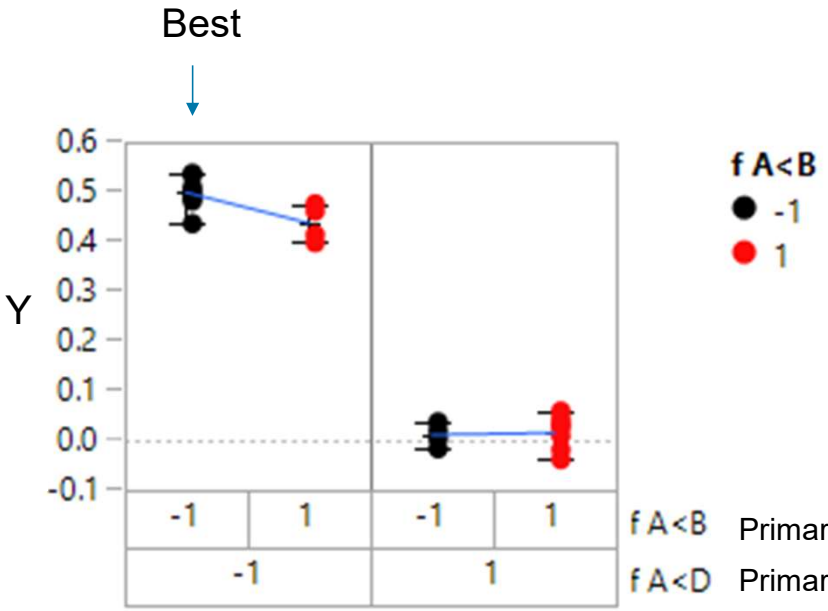




2) Stage 2: Use forward stepwise regression to add significant <u>interactions</u> between pairwise ordering factors involving only the important main effect factors (employing the strong heredity assumption)



Case Study Example: Order (4, 24)



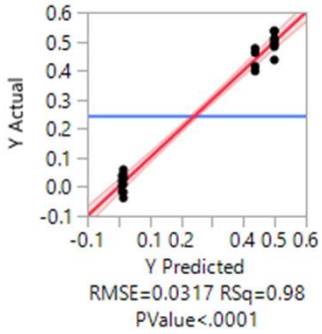
Primary before Secondary Binder

Primary before Rheology Modifier

Summary of Fit	
RSquare	0.984791
RSquare Adj	0.98251
Root Mean Square Error	0.031727
Mean of Response	0.246273
Observations (or Sum Wgts)	24

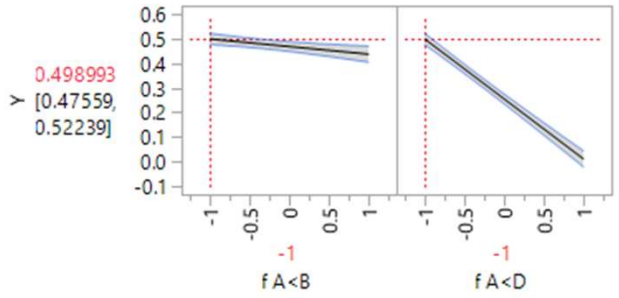
Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	3	1.3035773	0.434526	431.6775
Error	20	0.0201320	0.001007	Prob > F
C. Total	23	1.3237093		<.0001*



Source	LogWorth	PValue
f A<D	18.215	0.00000
f A<D*f A<B	1.582	0.02615
f A<B	1.306	0.04944 ^

Prediction Profiler

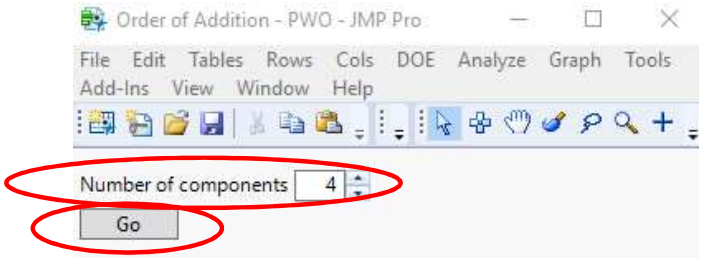


To maximize the efficacy of the rheology modifier:

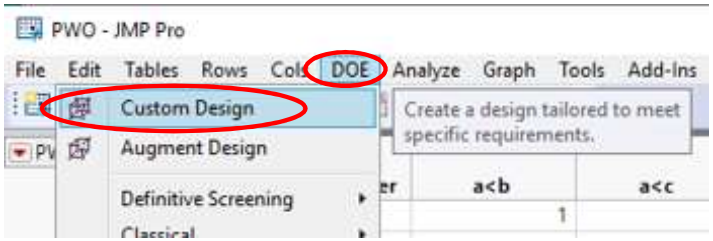
- f A<D = false and f A<B = false
- Thus, primary binder should be added **after** both the rheology modifier and secondary binder

Generating Optimal Fractions with JMP - Order Experiment

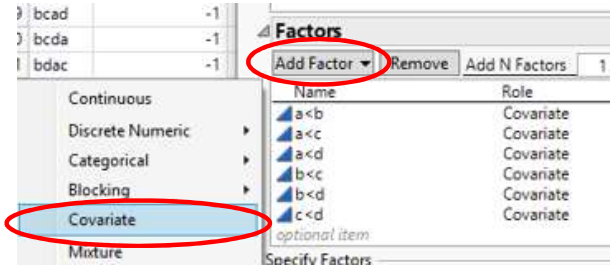
1) Use JMP Order of Addition addin



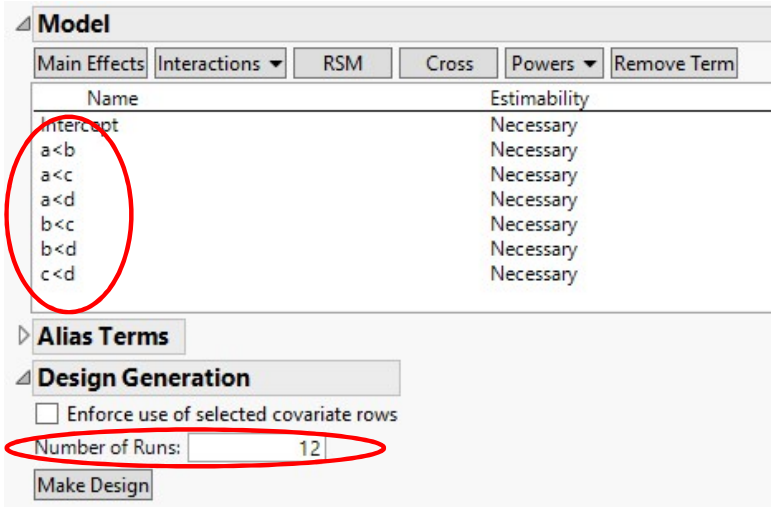
2) Custom Design



3) Add Covariate factors = pairwise ordering factors



4) Define model and number of runs



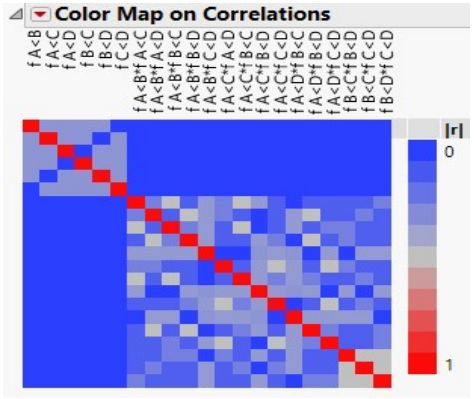
Evaluating Designs

Order (4, 24)

Power Analysis

Significance Level	0.05
Anticipated RMSE	1

Term	Anticipated Coefficient	Power
Intercept	1	0.996
f A<B	1	0.93
f A<C	1	0.93
f A<D	1	0.93
f B<C	1	0.93
f B<D	1	0.93
f C<D	1	0.93

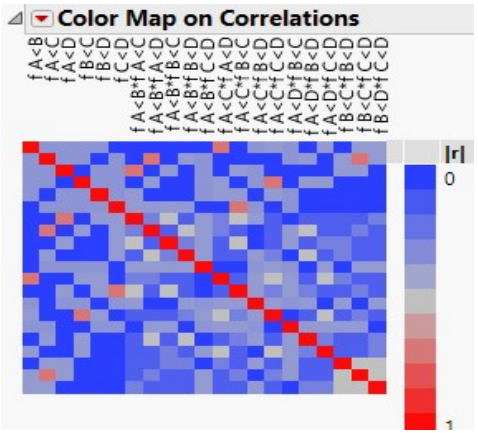


Order (4, 12)

Power Analysis

Significance Level	0.05
Anticipated RMSE	1

Term	Anticipated Coefficient	Power
Intercept	1	0.789
f A<B	1	0.548
f A<C	1	0.548
f A<D	1	0.548
f B<C	1	0.548
f B<D	1	0.548
f C<D	1	0.548



The 12-run experiment has:

- Half the number of runs
- Lower power to detect effects (increased chance to miss an effect – type II error)
- More correlation of main effects with 2-factor interactions

Summary

An Order experiment is one in which there are multiple process steps and the order in which the steps are performed is studied.

- Order-of-Additions experiments are an important class of order experiments.

- Pairwise order factors (e.g. B enters before C: B<C) are used to:
  - Analyze the experiment – treated as you would any other process variable
  - Find optimal subsets of the full permutation experiment to create manageable sized experiments
  - The factor levels are (1 = true; -1 = false)

- The recommended analysis method is 2-stage forward stepwise regression:
  - Stage 1 – main effects; Stage 2 – interactions (limited to those with strong heredity)

- Fractional subsets can be created by using the pairwise ordering factors as “covariate” variables with the custom design platform in JMP

Forward thinking:
- Mixture-Order experiments – ingredient amounts and order
- Process-Order experiments – e.g. change process step order and reaction temperature.

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