

Part 2 – Custom DOE Constraints

Using JMP's Custom DOE platform to create an appropriate experimental design

JMP's DOE Custom Designer has many features that are helpful in creating a customized experimental design. In this case, we wanted to employ a mixture design. However, for this experimental design we were dealing with a special requirement: One of the mixture components could consist of one of eight different materials, each of which had its own range of fractions that it comprised in the mixture. In addition, initially we wanted to study an additional 3 mixture factors in the experiment.

The solution involved using "Disallowed Combinations Script" to define constraints, but also involved some special scripting. The process is described below:

1. Define Factors
 - a. The first factor describes the eight material types of the special mixture component. This is a categorical factor which we will call Additive, which has eight levels (Additive 1, Additive 2, Additive 3, Additive 4, Additive 5, Additive 6, Additive 7, Additive 8.)
 - b. The second factor (M1) is defined as a continuous variable, representing the fractional level of the material defined by Additive. When defining this factor, we let the min and max range from 0 to 1. Individual levels for each level of Additive will be specified in Constraints.
 - c. M2, M3, and M4 are the remaining three mixture components, all defined as Continuous, and each with its own range of possible mixture fractions.
 - d. Other factors which are independent of the mixture can be added as needed, but are not shown here.
2. Set Constraint for overall mixture:
 - a. Mixture must sum to 100%. One way to define this would be to disallow any combination of $M1+M2+M3+M4$ that does not add exactly to 1.0. However, the algorithm that JMP uses to find an optimal design needs a little "flexibility" to operate efficiently. We do this by specifying that $M1+M2+M3+M4$ needs to fall in the range of 0.99 to 1.01. But since this is a "Disallowed Combinations" script, we want to define the constraint as the mixture sum cannot be less than 0.99, nor greater than 1.01. To do this we employ inequalities and an OR statement as follows:
$$M1+M2+M3+M4 > 1.01 \mid M1+M2+M3+M4 < 0.99$$
3. Set constraint on M1:
 - a. The range of M1 depends on the material selected by Additive type. For material Additive 1, let's say that M1 needs to fall between 0.5 and 0.6 of the mixture. Since we are again dealing with "disallowed combinations", we have to state what the range "can't be."
$$\text{Additive} == \text{"Additive 1"} \ \& \ (M1 < 0.5 \mid M1 > 0.6)$$
 - b. Similarly, for the other values of Additive:
$$\text{Additive} == \text{"Additive 2"} \ \& \ (M1 < 0.2 \mid M1 > 0.6)$$

$$\text{Additive} == \text{"Additive 3"} \ \& \ (M1 < 0.2 \mid M1 > 0.5)$$

$$\text{Additive} == \text{"Additive 4"} \ \& \ (M1 < 0.1 \mid M1 > 0.3)$$

$$\text{Additive} == \text{"Additive 5"} \ \& \ (M1 < 0.0 \mid M1 > 0.2)$$

$$\text{Additive} == \text{"Additive 6"} \ \& \ (M1 < 0.2 \mid M1 > 0.4)$$

$$\text{Additive} == \text{"Additive 7"} \ \& \ (M1 < 0.3 \mid M1 > 0.5)$$

$$\text{Additive} == \text{"Additive 8"} \ \& \ (M1 < 0.1 \mid M1 > 0.4)$$
 - c. Note that the ranges shown above are merely examples, and do not represent actual material ranges.

4. Pull the above constraints together into the Disallowed Combinations Script window. Each constraint is an “OR” condition, so the final code looks like this:

```
( M1+M2+M3+M4 > 1.01 | M1+M2+M3+M4 < 0.99 ) |  
( Additive == “Additive 1” & (M1<0.5 | M1>0.6) ) |  
( Additive == “Additive 2” & (M1<0.2 | M1>0.6) ) |  
( Additive == “Additive 3” & (M1<0.2 | M1>0.5) ) |  
( Additive == “Additive 4” & (M1<0.1 | M1>0.3) ) |  
( Additive == “Additive 5” & (M1<0.0 | M1>0.2) ) |  
( Additive == “Additive 6” & (M1<0.2 | M1>0.4) ) |  
( Additive == “Additive 7” & (M1<0.3 | M1>0.5) ) |  
( Additive == “Additive 8” & (M1<0.1 | M1>0.4) )
```

5. Create the model. We wanted to include curvature and interactions in the model. Since this is a mixture model, we chose a Scheffe Cubic model. (A future blog post will talk about why these terms result from a Scheffe Cubic model for mixtures.) This model includes the following terms:

- a. Five main effects terms (one for each factor)
 - Additive
 - M1
 - M2
 - M3
 - M4
- b. Four two-way interactions for the Additive type vs. each mixture component. These support :
 - Additive*M1
 - Additive*M2
 - Additive*M3
 - Additive*M4
- c. Six two-way interactions for each pairing of mixture terms
 - M1*M2
 - M1*M3
 - M1*M4
 - M2*M3
 - M2*M4
 - M3*M4
- d. Four 3-way interactions between mixture components:
 - M1*M2*M3
 - M1*M2*M4
 - M1*M3*M4
 - M2*M3*M4
- e. Six terms that handled squared mixture components in an interaction form:
 - M1^2*M2
 - M1^2*M3
 - M1^2*M4
 - M2^2*M3
 - M2^2*M4
 - M3^2*M4

Other Design Parameters

JMP's Custom Designer allows input of several more parameters to customize the designer's behavior. We chose the following:

Set Random Seed(0)

This allows the routine to use different "starting points" when finding a design. Things like run order and even details about factor settings can change slightly from design generation to generation. (Set this to any integer to have JMP always reproduce the same design.)

Number of Starts(100)

JMP uses an iterative solution based on a random starting point to design a test, then starts over with a different starting point to see if it can find an improved design. Increasing this parameter can have the effect of finding a better design, at the expense of longer computation times.

Set Sample Size(65),

This is the total number of trials in the test. It must be at least as large as the total number of parameters needed for a solution to the model. Larger number of trials can increase computation time, but more importantly require more experimental trials to be made.

Optimality Criterion(Name("Make D-Optimal Design")),

D-Optimal designs give the best estimates of the parameters in the resulting model. I-Optimal designs give the lowest average prediction variance resulting from the model.

Simulate Responses(0),

JMP can create simulated responses if you want to test the design. These have nothing to do with real world results. We chose not to simulate responses.

Save X Matrix(0),

JMP can save the Moments Matrix and Model Matrix if desired. We left this option turned OFF.

The complete JMP script to handle this design

As we developed the design, we found it easier to adjust the parameters via editing a script and running the script. The script was first created by creating a manual design, and then using "Save Script to Script Window" from the red triangle. The resulting script (after some reformatting) is shown below, and attached to this video:

```
DOE(
  Custom Design,
  {
    Add Response( Maximize, "Y", ., ., . ),
    Add Factor( Categorical, {"Additive 1", "Additive 2", "Additive 3", "Additive 4",
"Additive 5", "Additive 6", "Additive 7", "Additive 8" }, "Additive", 0 ),
    Add Factor( Continuous, 0, 1, "M1", 0 ),
    Add Factor( Continuous, 0, 1, "M2", 0 ),
    Add Factor( Continuous, 0, 1, "M3", 0 ),
    Add Factor( Continuous, 0, 1, "M4", 0 ),

    // Manually enter all main effects
    Add Term( {1, 1} ), // Additive main effect
    Add Term( {2, 1} ), // M1 main effect
    Add Term( {3, 1} ), // M2 main effect
    Add Term( {4, 1} ), // M3 main effect
    Add Term( {5, 1} ), // M4 main effect

    // Manually enter all 2-way interactions between mixture components
    Add Term( {2, 1}, {3, 1} ), // M1*M2 interaction
    Add Term( {2, 1}, {4, 1} ), // M1*M3 interaction
    Add Term( {2, 1}, {5, 1} ), // M1*M4 interaction
    Add Term( {3, 1}, {4, 1} ), // M2*M3 interaction
    Add Term( {3, 1}, {5, 1} ), // M2*M4 interaction
    Add Term( {4, 1}, {5, 1} ), // M3*M4 interaction

    // Manually enter all interactions between Additive and mixture components
    Add Term( {1, 1}, {2, 1} ), // Additive*M1 interaction
    Add Term( {1, 1}, {3, 1} ), // Additive*M2 interaction
    Add Term( {1, 1}, {4, 1} ), // Additive*M3 interaction
    Add Term( {1, 1}, {5, 1} ), // Additive*M4 interaction

    // Manually enter all 3-way interactions
    Add Term( {2, 1}, {3, 1}, {4, 1} ), // M1*M2*M3 3-way interaction
    Add Term( {2, 1}, {3, 1}, {5, 1} ), // M1*M2*M4 3-way interaction
    Add Term( {2, 1}, {4, 1}, {5, 1} ), // M1*M3*M4 3-way interaction
    Add Term( {3, 1}, {4, 1}, {5, 1} ), // M2*M3*M4 3-way interaction

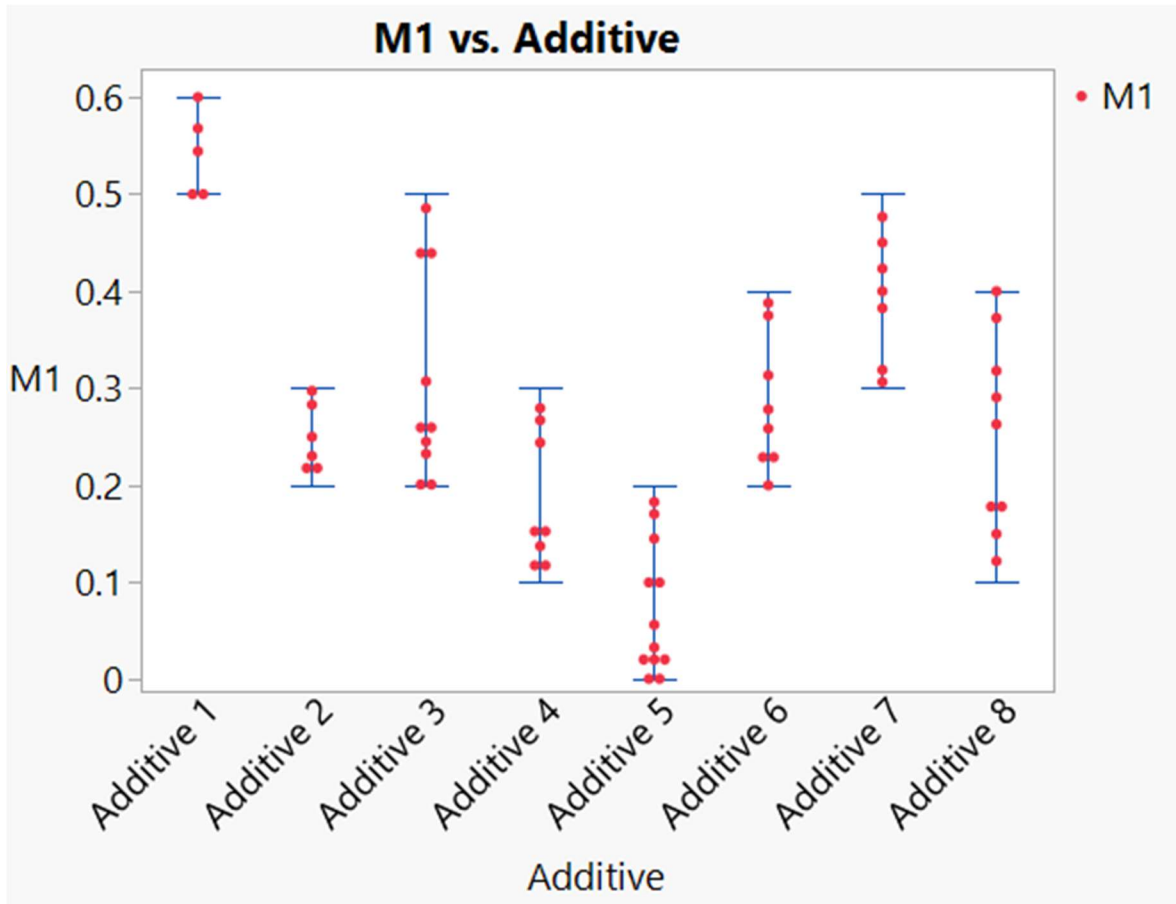
    // Manually enter appropriate Scheffe Cubic interactions
    Add Term( {2, 2}, {3, 1} ), // M1^2*M2 interaction
    Add Term( {2, 2}, {4, 1} ), // M1^2*M3 interaction
    Add Term( {2, 2}, {5, 1} ), // M1^2*M4 interaction
    Add Term( {3, 2}, {4, 1} ), // M2^2*M3 interaction
    Add Term( {3, 2}, {5, 1} ), // M2^2*M4 interaction
    Add Term( {4, 2}, {5, 1} ), // M3^2*M4 interaction

    // Now add the disallowed combinations:
    Disallowed Combinations
      (
        (M1 + M2 + M3 + M4 > 1.01 | M1 + M2 + M3 + M4 < 0.99) |
        (Additive == "Additive 1" & (M1 < 0.5 | M1 > 0.6)) |
        (Additive == "Additive 2" & (M1 < 0.2 | M1 > 0.3)) |
        (Additive == "Additive 3" & (M1 < 0.2 | M1 > 0.5)) |
        (Additive == "Additive 4" & (M1 < 0.1 | M1 > 0.3)) |
      )
  }
)
```

```
        (Additive == "Additive 5" & (M1 < 0.0 | M1 > 0.2)) |
        (Additive == "Additive 6" & (M1 < 0.2 | M1 > 0.4)) |
        (Additive == "Additive 7" & (M1 < 0.3 | M1 > 0.5)) |
        (Additive == "Additive 8" & (M1 < 0.1 | M1 > 0.4))
    ),
    Set Random Seed( 0 ),
    Number of Starts(100 ),
    Set Sample Size(65),
    Optimality Criterion( Name( "Make D-Optimal Design" ) ),
    Simulate Responses( 0 ),
    Save X Matrix( 0 ),
    Make Design}
);
```

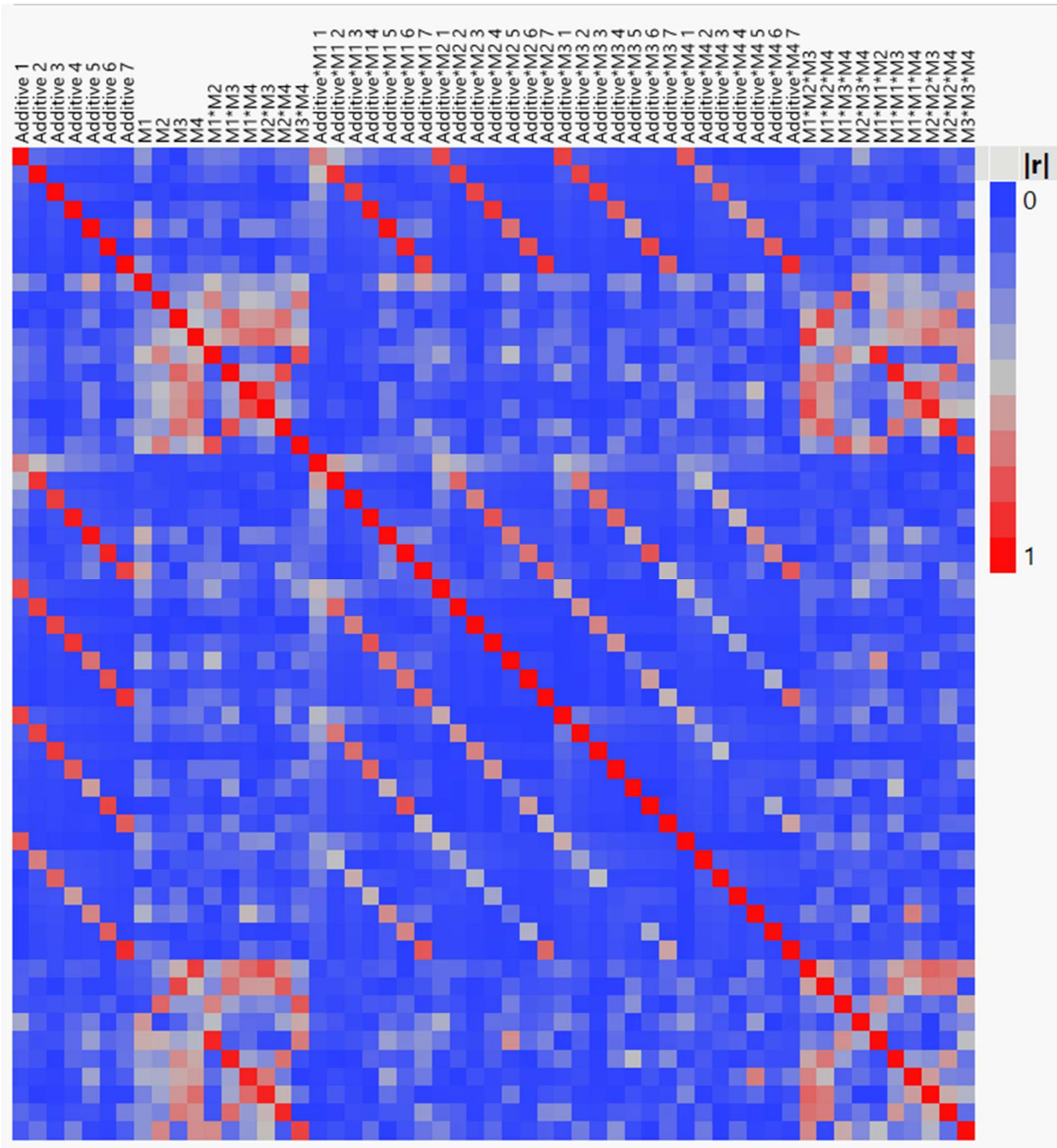
Design Diagnostics

The diagnostics on this design are interesting. First, let's look at how well we achieved the constraints of the test on the M1 additives:



In Figure 1, the blue “whiskers” indicate the desired ranges for each of the M1 additive types, as input in the script. The red dots indicate the actual design points. As can be seen, the Custom Designer tried to spread the points out across each range but didn't completely cover the desired ranges. Increasing the Number of Starts can improve the spread of the points, but it seemed as though the ranges were never completely covered. This has been reported to the JMP Development team, in hopes that it may be addressed in future software releases.

The color map on correlations produced the following plot:



Other Observations

Design Convergence

Sometimes custom designer didn't converge. Increasing NStarts and/or rerunning generally resolved this problem.

Minimum Trials when running a Script

Normally JMP's Custom Designer will provide the minimum number of independent experimental trials required to solve for the unknown coefficients in the designated model. We don't know a priori what this value is when running the script. Fortunately, if Sample Size is set to some small value (e.g. 2), the script defaults to the minimum number of trials required to solve for the coefficients. So our suggestion is to run the design once to determine the minimum number of

trials, then increase the Sample Size to some number greater than the min trials (in order to estimate pure error and goodness of fit) and rerun the design. In the case studied in this paper, the minimum number of trials was 56, and the chosen Sample Size was 65.

Savings vs. a Full Factorial Design

Since we are looking for curvature in this design, a full factorial would have required:

$$8 \text{ additive types} * 3 \text{ M1 levels} * 3 \text{ M2} * 3 \text{ M3} * 3 \text{ M4} = 648 \text{ trials}$$

Therefore the Custom Design saved nearly 90% of the trials that would have been required in a full factorial test.

Analysis Notes

Prediction Profiler

Since the input factors were entered as Continuous (as opposed to Mixture) factors, we need to now tell JMP of the mixture relationship that all mixture factors must sum to 1. The easiest way to do this is to change the Design Role property for each of the Mixture columns to "Mixture," and activate the Mixture property and ensure that the sum adds up to 1.0.

Intercept

When running Fit Model to estimate the model and produce the Prediction Profiler, we need to turn OFF the intercept. This is due to the Scheffe Cubic model for mixtures, which does not require an intercept. (This will be addressed in an upcoming JMP Blog post.)

Limit of Detection

The measurement of Separation Index in this experiment had a Limit of Detection of 0.001 units. JMP 16 Pro can handle this by setting the "Detection Limit" property for that particular response column, and then using Generalized Regression for the analysis. This gives significantly different results than if Least Squares Regression is used.

Extrapolation Control

We want to use the Prediction Profiler to find an optimum setting of Additive type, M1 (Additive Level), M2, M3, and M4. The Prediction Profiler can do this, but we want to restrict the ranges of the additive levels to be appropriate for those seen in the test. In JMP Pro, the Extrapolation Control feature in the Prediction Profiler is intended to cover this functionality. Extrapolation Control can be found under the Prediction Profiler red drop-down triangle menu.

Unfortunately, it appears that Extrapolation Control does not work with all types of analyses and would not work in our case. This is being reported to the JMP development team.

Testing Notes

Requirements at Fuchs Lubricants changed as the test was being designed. In the end, the actual experiment did not correspond to the script shown above. In fact, it was greatly simplified to involve only seven additive types, and no other mixture variables. It was also simplified to reduce the model complexity by only looking at main effects terms (as in a screening test).

This changed the actual test from a mixture design to a simple two-factor design, with one categorical factor (Additive, with 7 levels) and different levels for each additive type. This can be handled in one of two ways:

- A series of seven independent experiments, one for each Additive type, with two trials each (a high and low level). This results in 14 trials, not including replicates.

- A combined 14-trial test with two factors (categorical Additive and continuous M1). The design includes the constraints on the levels of M1, but no mixture sum since there is only one component. The experimental trials are the same 14 trials as mentioned above, but the analysis changes somewhat. Model terms include the two main effects (Additive and M1) and the Additive*M1 interaction. In this case the Intercept term is left On for the analysis.

Repeated trials were also desired in this test, but were not available at the time of this writing. Therefore, results were “exact,” with no uncertainty estimates.