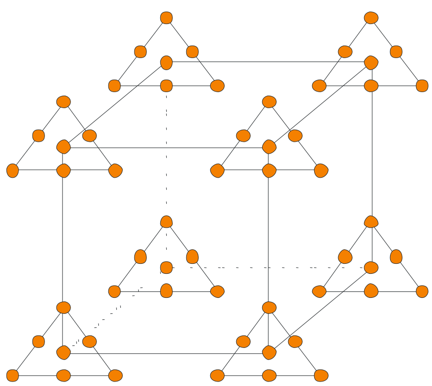
Combined Mixture-Process Tutorial

Introduction

Design-Expert® software offers designs that combine mixture components and process factors (numeric and/or categoric). Before embarking on this tutorial, you should first complete the previous tutorials on mixture design: Part 1 – The Basics, and Part 2 – Optimization. For details about software features and their statistical aspects, use the on-screen program Help. Also, Stat-Ease provides in-depth training in its advanced workshop for mixture design. Call for information on content and schedules, or better yet, visit our web site at www.statease.com.

In this tutorial, you will apply Design-Expert's unique features for mixture-process design and analysis by investigating John Cornell's famous fish-patty experiment from his textbook *Experiments with Mixtures*, 3rd edition, published by John Wiley and Sons, New York.

The food formulators hope to make something delicious from a blend of three tasty-sounding (?) fish: mullet, sheepshead, and croaker. Yum! The seven blends in the design include one each of the three fishes, plus three binary blends, and a blend of one-third each of the individual types of fish. The fish patties can be cooked at varying deep-frying time, oven temperature, and oven time. Combining each of these process factors at two levels for all seven blends creates a total of 56 experimental runs. The diagram below shows the blends as points on triangles, repeated at each of the eight corners of a cube representing the process.

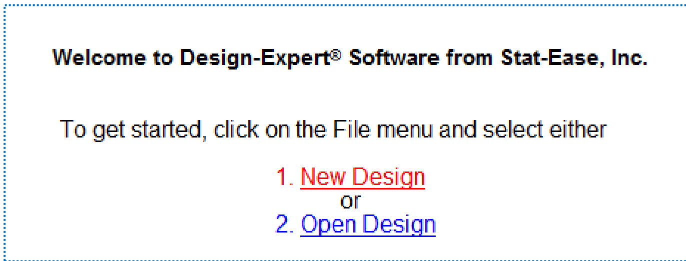


Fish-patty design: Seven blends (on triangles) at eight process combinations (cube)

The response measure is patty texture. (Never mind about taste!)

The Experiment as Originally Run

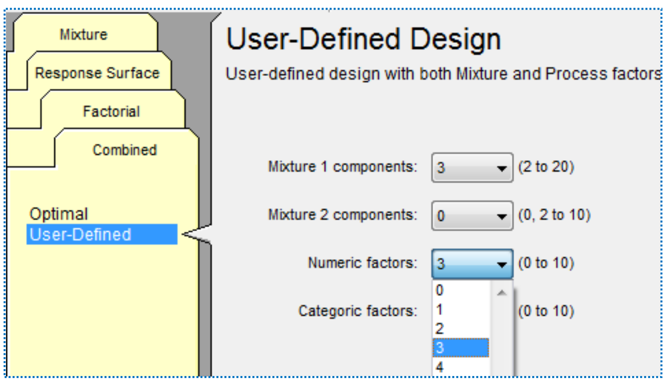
Start the program by finding and double clicking the Design-Expert software icon. Take the quickest route to initiating a new design by clicking the **New Design** option on our opening screen as shown below. Another route is via File, New Design (or associated Alt keys).



Opening screen – New Design option highlighted in red

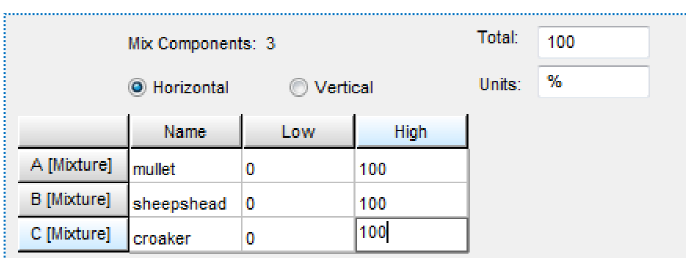
Click the **Combined** folder tab and select the **User Defined** option. The user-defined option allows you to reproduce Cornell's design with all points chosen. Normally, to save on runs, you want to use the first choice on the list, Optimal, which selects an ideal subset of design points from a candidate set for a specified model. We'll explore this option later.

For **Mixture 1 Components,** select **3** from the droplist. (Notice that Design-Expert offers the option for adding a second mixture, for example in a two-layer cake, film, or coating. For the number of **Numeric Factors** choose **3**. Leave Categoric Factors at zero, but keep in mind for future experimentation that you can add discrete variables – such as who supplies a given component.



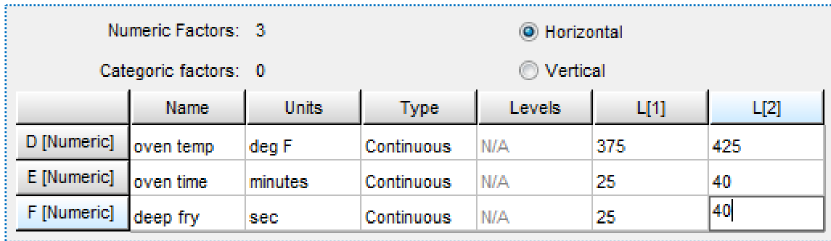
Specifying the combined user-defined design

Press **Continue** and enter the **Total** for mixture components (fish types) at **100** and **Units** as **%**. Then enter fish names as shown below. Low limits remain zero. Set all high limits to **100**.



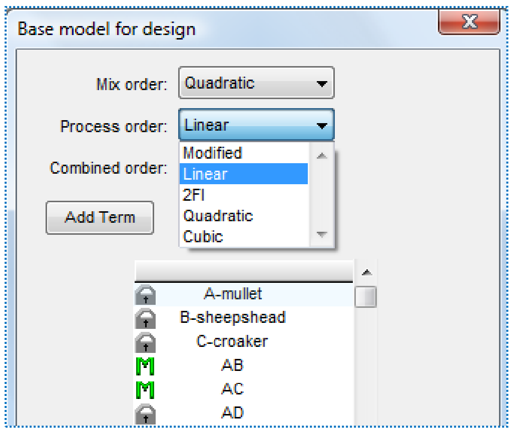
Entering mixture details

Press **Continue** to move on to the process design factors. Enter factor names, units, and ranges (low – L[1] and high – L[2]) as shown below.



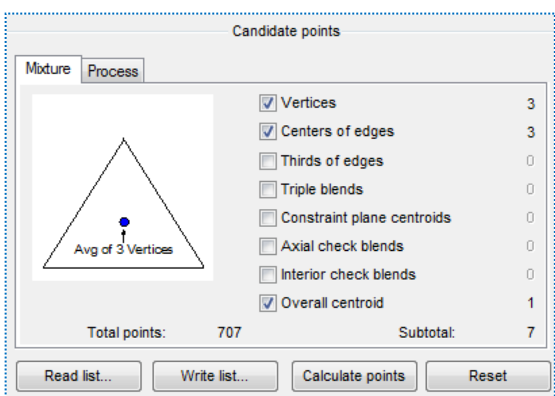
Entering process details

Press **Continue** to define the model you want to design for the combination of mixture and process variables in this experiment. The default model is quadratic by quadratic. Click the **Edit model** button and change the default for **Process Order** to **Linear**.



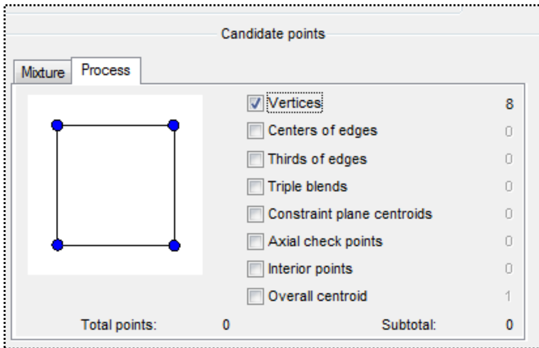
Specifying the design model for the mixture and process variables

Click the **OK** button to implement the change. Now, consider your "candidate" points. Normally only some of these points would actually make it to the design, but by choosing the User Defined option, you get all of them. However, there's a problem – the **Mixture** candidate set of points (Mixture tab at upper-left screen box) contains more points than those chosen by Cornell. Fix this by clicking off **Thirds of edges**, **Triple blends**, **Axial check blends,** and **Interior check blends**. Only vertices, centers of edges, and overall centroid should now be checked. Press **Calculate points** to get a count – this subtotals to 7 for the mixture candidate points.



Candidate points for mixture

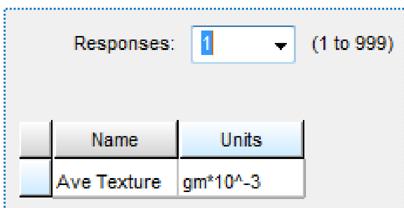
Now click the **Process** candidate set of points tab (right of Mixture tab). This design also contains more points than those chosen by Cornell, so click <u>off</u> every checkmark <u>except</u> **Vertices**. Press **Calculate points** to update total run count.



Candidate points for the process

You should now see a subtotal of 8 process combinations, which combined with the 7 mixture candidates generates 56 total runs (points).

Press **Continue** to see the response specification form. Enter **Ave Texture** with units of **gm*10^-3**.

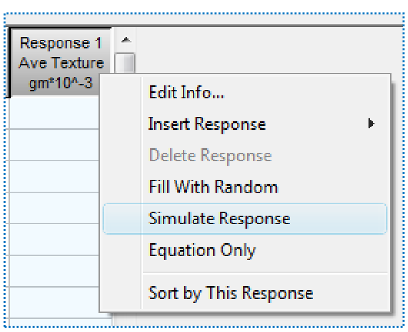


Entering response name and units

Press **Continue** to complete the design. Design-Expert now displays the 56 experiments in random run order.

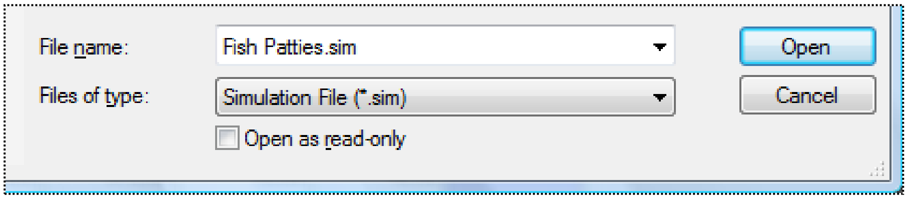
<u>Analyze the Results</u>

To view response data, <u>right-click</u> the **Response** column header and select **Simulate Response**.



Simulating the response

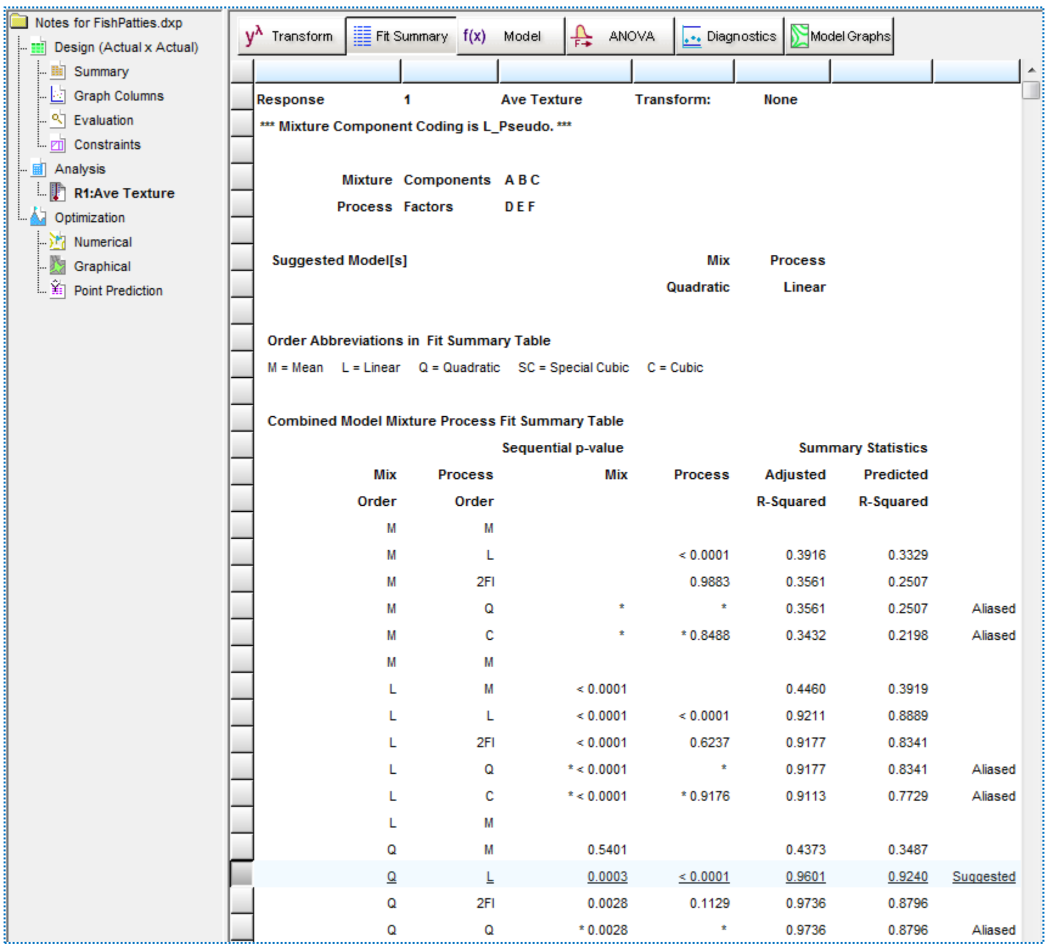
Click file **Fish Patties.sim** and **Open** it.



Opening the fish patty simulation

The simulation displays its processing rate at a readable speed so you can observe the responses, so be patient – it may take a minute to generate all the data. Results within the range of 2 to 3.5 are desirable. To preserve the responses, go to **File** and select **Save As**. Change the file name to **FishPatties.dxp** or some other unique name of your choice.

To analyze the data, click the **Analysis** node labeled **Ave Texture**. Then click the ***Fit Summary*** button. You now see a unique matrix of probabilities that helps you determine the best crossed model for mixture and process. As shown below, the software provides suggestion(s) on which combination is best – in this case, a quadratic ("Q") mixture model crossed with a linear ("L") process model.



Response 1 Ave Texture Transform: None

*** Mixture Component Coding is L_Pseudo. ***

Mixture Components A B C

Process Factors D E F

Suggested Model[s] Mix: Quadratic, Process: Linear

Order Abbreviations in Fit Summary Table

M = Mean L = Linear Q = Quadratic SC = Special Cubic C = Cubic

Combined Model Mixture Process Fit Summary Table

Mix Order	Process Order	Sequential p-value Mix	Sequential p-value Process	Adjusted R-Squared	Predicted R-Squared	
M	M					
M	L		< 0.0001	0.3916	0.3329	
M	2FI		0.9883	0.3561	0.2507	
M	Q	*	*	0.3561	0.2507	Aliased
M	C	*	* 0.8488	0.3432	0.2198	Aliased
M	M					
L	M	< 0.0001		0.4460	0.3919	
L	L	< 0.0001	< 0.0001	0.9211	0.8889	
L	2FI	< 0.0001	0.6237	0.9177	0.8341	
L	Q	* < 0.0001	*	0.9177	0.8341	Aliased
L	C	* < 0.0001	* 0.9176	0.9113	0.7729	Aliased
L	M					
Q	M	0.5401		0.4373	0.3487	
Q	L	0.0003	< 0.0001	0.9601	0.9240	Suggested
Q	2FI	0.0028	0.1129	0.9736	0.8796	
Q	Q	* 0.0028	*	0.9736	0.8796	Aliased

Suggested model – quadratic (mixture) by linear (process)

For a detailed explanation about how to interpret sequential p-values in this Fit Summary table for combined designs, see Stat-Ease's *Handbook for Experimenters* (provided free of charge to all registered users of Design-Expert software) section titled "Combined Mixture/Process Analysis Guide." This section is available upon request to trial users.

Now click the ***Model*** button atop your screen. Design-Expert uses the suggested model as its default. Accept this for the moment by clicking ahead to the ***ANOVA*** button. Notice that many of the model terms, particularly ones of third-order (for example, ABF) exhibit high Prob>F values. This is a byproduct of crossing the two mixture and process models, which creates many superfluous higher-order terms. When analyzing designs like this, you will often find it beneficial to reduce your model to only the significant terms, subject to requirements for maintaining hierarchy (no interactions without their parent terms).

Transform | Fit Summary | f(x) Model | ANOVA | Diagnostics | Model Graphs

Response 1 Ave Texture

ANOVA for Combined Quadratic x Linear Model

*** Mixture Component Coding is L_Pseudo. ***

Mixture Components A B C

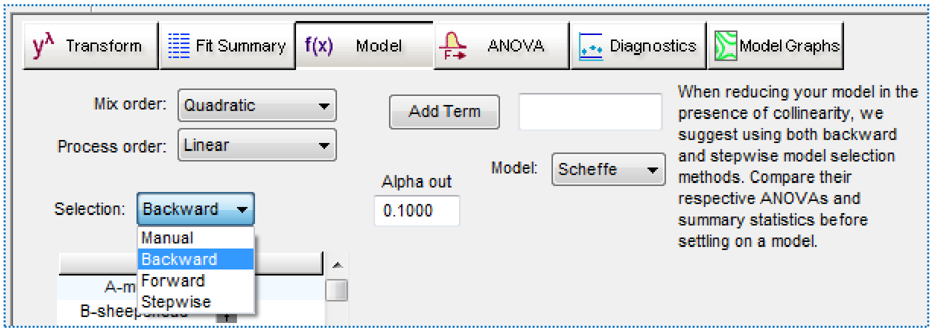
Process Factors D E F

Analysis of variance table [Partial sum of squares - Type III]

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F
Model	29.41	23	1.28	58.62	< 0.0001
Linear Mixture	*14.04*	*2*	*7.02*	*321.66*	*< 0.0001*
AB	*0.36*	*1*	*0.36*	*16.60*	*0.0003*
AC	*0.27*	*1*	*0.27*	*12.17*	*0.0014*
AD	*1.91*	*1*	*1.91*	*87.74*	*< 0.0001*
AE	*4.05*	*1*	*4.05*	*185.50*	*< 0.0001*
AF	*0.062*	*1*	*0.062*	*2.85*	*0.1013*
BC	*0.048*	*1*	*0.048*	*2.21*	*0.1466*
BD	*0.25*	*1*	*0.25*	*11.62*	*0.0018*
BE	*0.53*	*1*	*0.53*	*24.23*	*< 0.0001*
BF	*0.052*	*1*	*0.052*	*2.38*	*0.1327*
CD	*0.50*	*1*	*0.50*	*23.06*	*< 0.0001*
CE	*1.31*	*1*	*1.31*	*60.17*	*< 0.0001*
CF	*7.624E-004*	*1*	*7.624E-004*	*0.035*	*0.8529*
ABD	*0.25*	*1*	*0.25*	*11.23*	*0.0021*
ABE	*0.17*	*1*	*0.17*	*7.65*	*0.0094*
ABF	*4.246E-003*	*1*	*4.246E-003*	*0.19*	*0.6621*

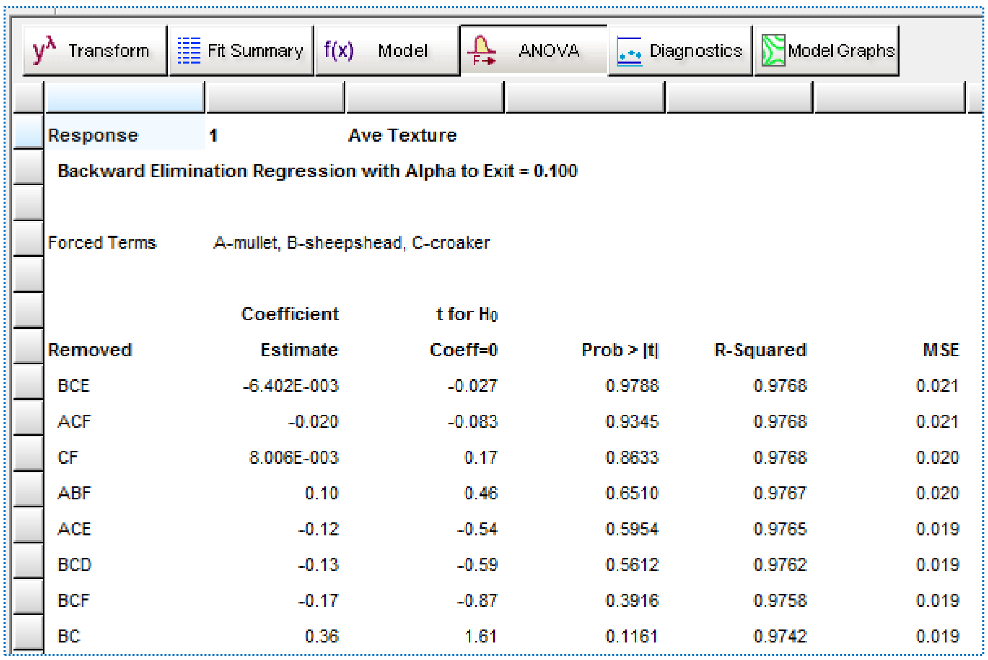
Initial ANOVA (no model reduction)

To reduce the model, go back and click the ***Model*** button. You could manually deselect the insignificant terms observed from the ANOVA, but it is quicker to let Design-Expert do this for you. Change selection mode to **Backward**.



Selecting backward reduction

This enables automatic model reduction by a backward (stepwise) algorithm. The criteria for elimination is a probability value of 0.1000, as specified in the Alpha Out field. Click the ***ANOVA*** button. As shown below, the software shows you a report on eliminated terms.



Response 1 Ave Texture

Backward Elimination Regression with Alpha to Exit = 0.100

Forced Terms A-mullet, B-sheepshead, C-croaker

Removed	Coefficient Estimate	t for H_0 Coeff=0	Prob > |t|	R-Squared	MSE
BCE	-6.402E-003	-0.027	0.9788	0.9768	0.021
ACF	-0.020	-0.083	0.9345	0.9768	0.021
CF	8.006E-003	0.17	0.8633	0.9768	0.020
ABF	0.10	0.46	0.6510	0.9767	0.020
ACE	-0.12	-0.54	0.5954	0.9765	0.019
BCD	-0.13	-0.59	0.5612	0.9762	0.019
BCF	-0.17	-0.87	0.3916	0.9758	0.019
BC	0.36	1.61	0.1161	0.9742	0.019

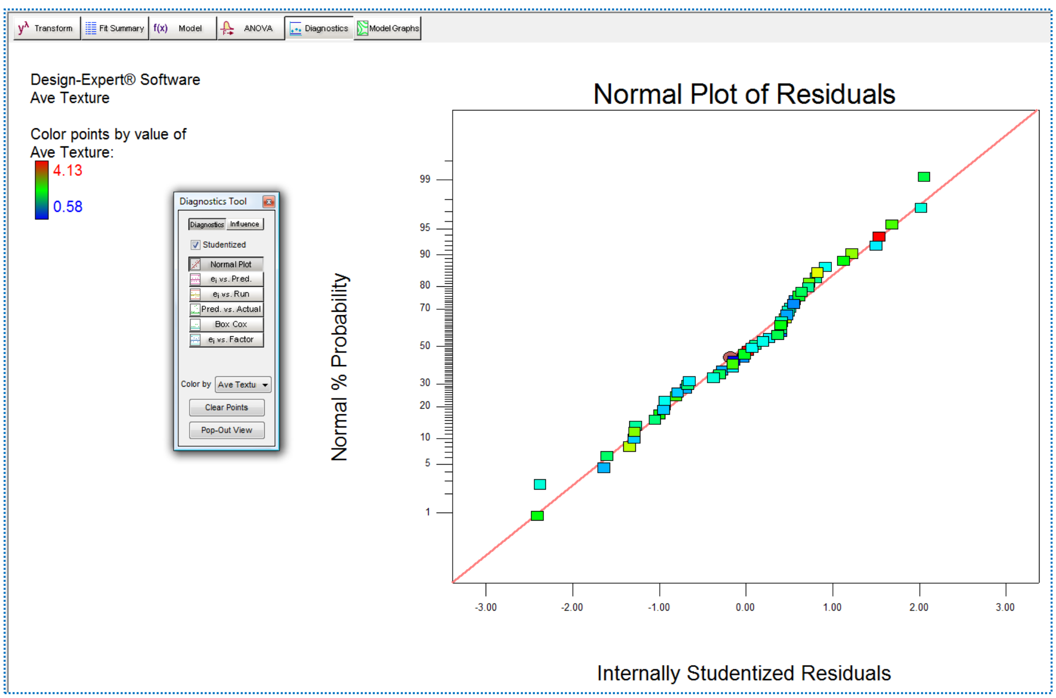
Report showing removed terms via backward model reduction

The ANOVA table shows that the reduced model fits very well.

Analysis of variance table [Partial sum of squares - Type III]					
	Sum of		**Mean**	**F**	**p-value**
Source	**Squares**	**df**	**Square**	**Value**	**Prob > F**
Model	29.33	15	1.96	100.58	< 0.0001
Linear Mixture	*14.04*	*2*	*7.02*	*360.95*	*< 0.0001*
AB	*0.38*	*1*	*0.38*	*19.30*	*< 0.0001*
AC	*0.28*	*1*	*0.28*	*14.23*	*0.0005*
AD	*1.92*	*1*	*1.92*	*98.95*	*< 0.0001*
AE	*4.71*	*1*	*4.71*	*242.37*	*< 0.0001*
AF	*0.077*	*1*	*0.077*	*3.97*	*0.0533*
BD	*0.26*	*1*	*0.26*	*13.63*	*0.0007*
BE	*0.64*	*1*	*0.64*	*32.84*	*< 0.0001*
BF	*0.096*	*1*	*0.096*	*4.92*	*0.0323*
CD	*0.55*	*1*	*0.55*	*28.15*	*< 0.0001*
CE	*1.81*	*1*	*1.81*	*93.23*	*< 0.0001*
ABD	*0.24*	*1*	*0.24*	*12.44*	*0.0011*
ABE	*0.16*	*1*	*0.16*	*8.47*	*0.0059*
ACD	*0.11*	*1*	*0.11*	*5.43*	*0.0250*
Residual	0.78	40	0.019		
Cor Total	30.11	55			
Std. Dev.	0.14		R-Squared	0.9742	
Mean	1.91		Adj R-Squared	0.9645	
C.V. %	7.32		Pred R-Squared	0.9477	
PRESS	1.57		Adeq Precision	47.396	

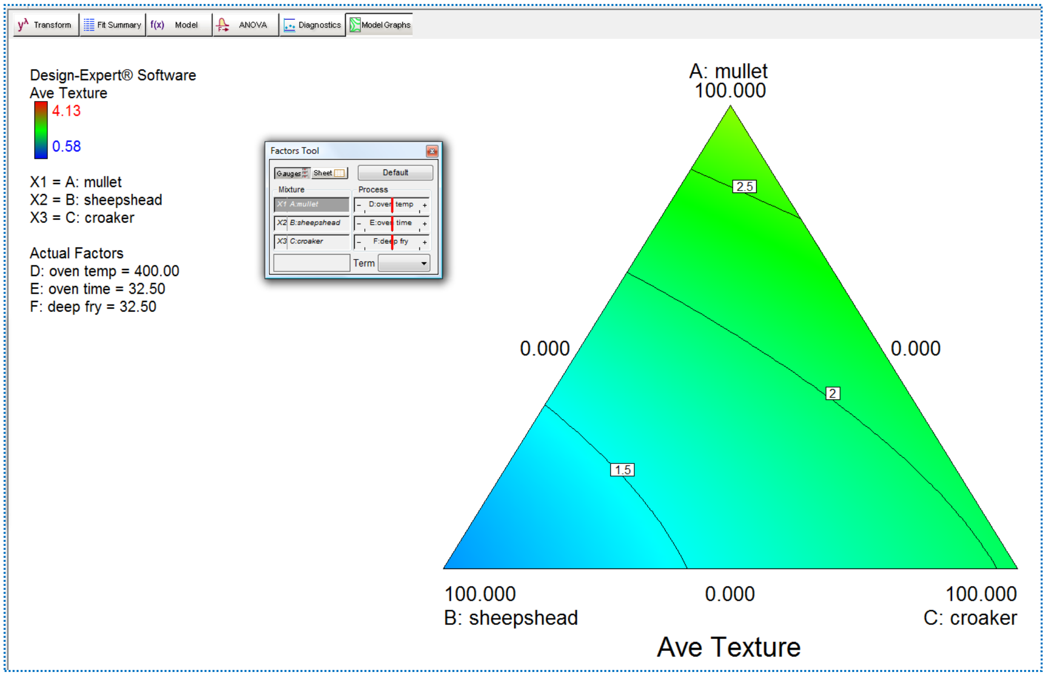
Analysis of Variance (partially shown) for Crossed Model

Click the ***Diagnostics*** button and examine the diagnostic graphs.



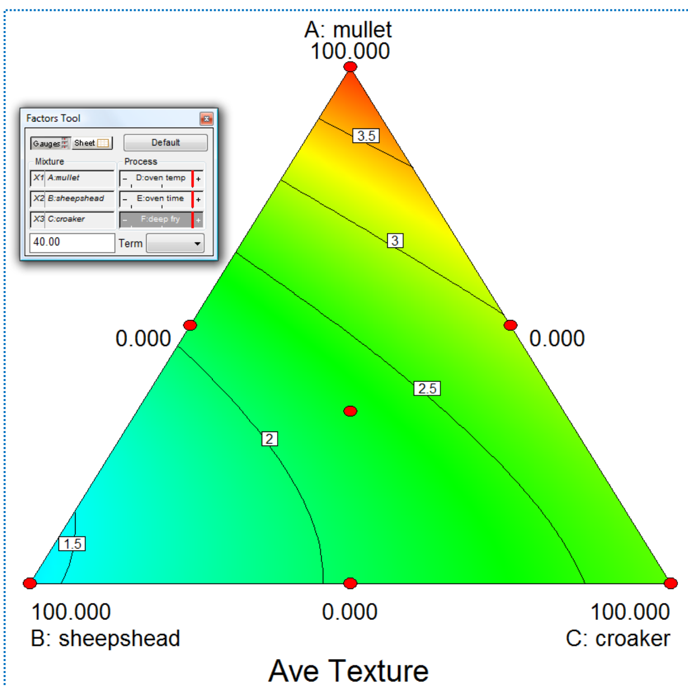
Normal plot of residuals – looks good

The normal plot of residuals looks good, so move on to the ***Model Graphs*** to view the response in mixture (triangular) space.



Model Graph with view of contour plot for mixture portion of combined design

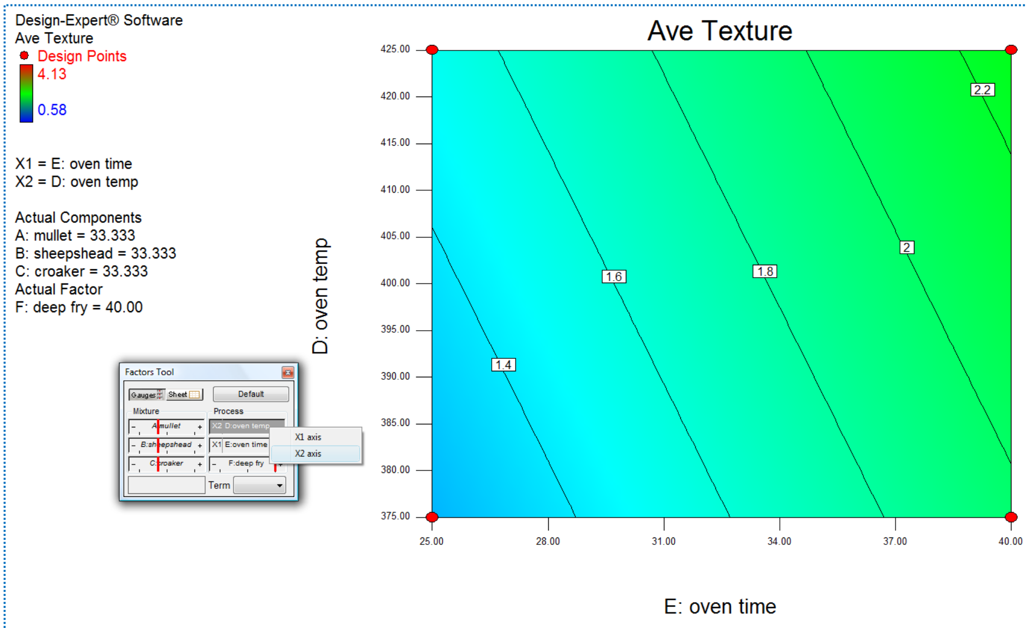
Click or grab various process factors on the floating tool bar and adjust them to see how response changes. For example, push all three bars to the right as shown below.



Process slide-bars pushed to their maximum levels

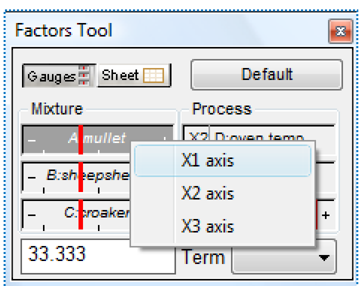
Notice by the hotter colors that texture increases at the 100 percent mullet (top of triangle).

Now right-click the **Factors Tool** where it shows **D:oven temp** and select **X2 axis**. As shown below, the view changes to process (rectangular) space.



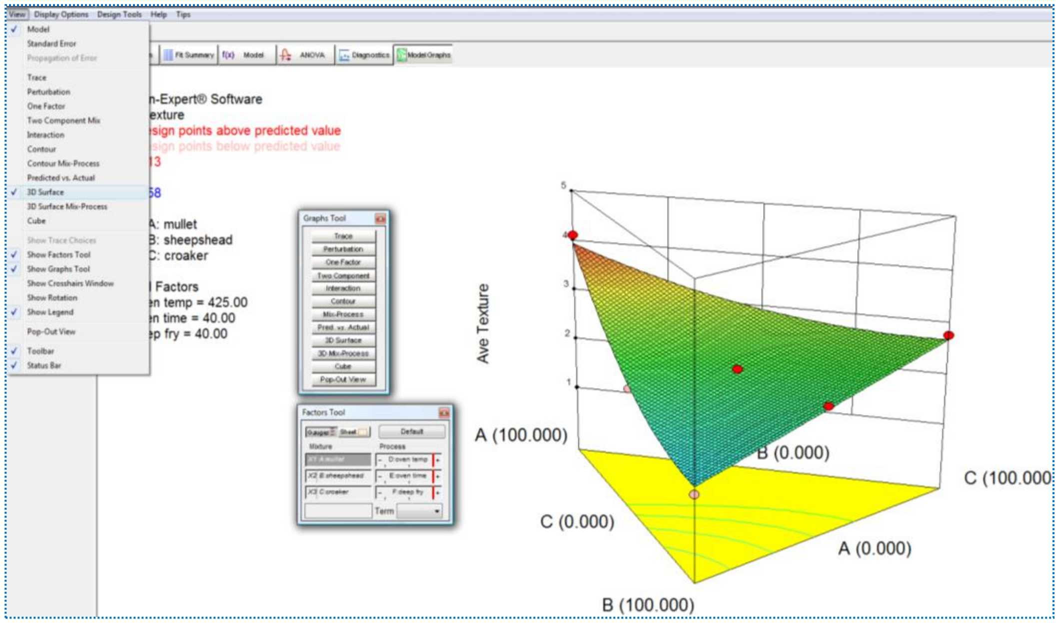
Changing to process view

Now shift back to mixture coordinates by right-clicking the **Factors Tool** where it lists **A:mullet.** Select **X1 axis**.



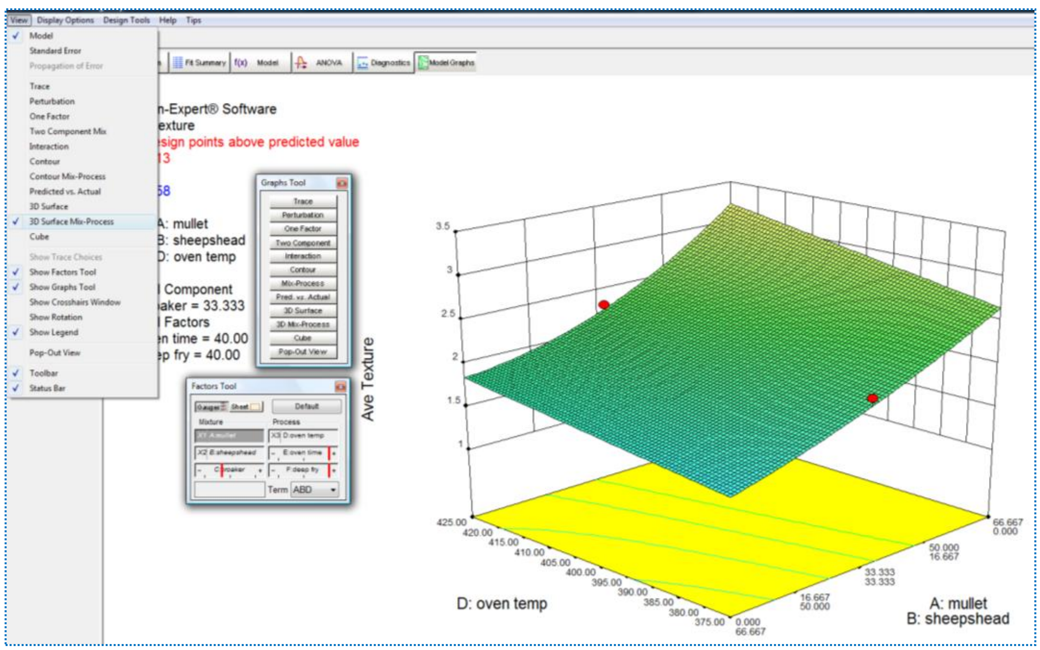
Back to mixture space

Select **View**, **3D Surface** (or click 3D Surface on the Graphs Tool) and notice the strong tilt in the A-B axis.



3D mixture view

Now select **View** for the **3D Surface Mix-Process** (or click 3D Mix-Process on the Graphs Tool). It's very likely you have never seen a plot like this!



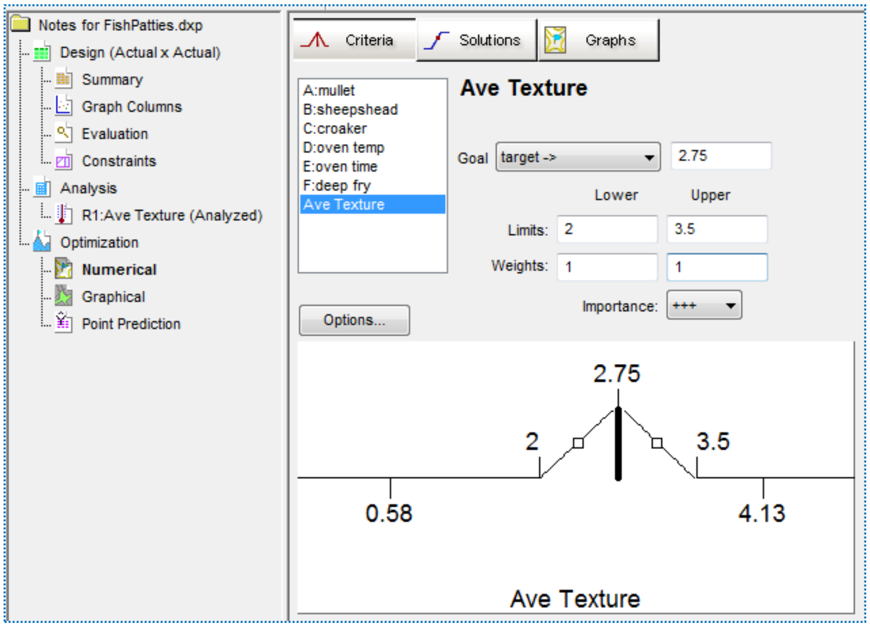
3D plot of two mixture components versus one process factor!

Now you see the impact of shifting component A (mullet) in direct substitution for B (sheepshead) on one axis versus the process factor D (oven temp) on the other axis. Isn't that something!

Continue if you like and explore different combinations of the mixture components and process factors in 2D contour view and 3D plots. Keep in mind that any textures outside of 2 to 3.5 are unacceptable.

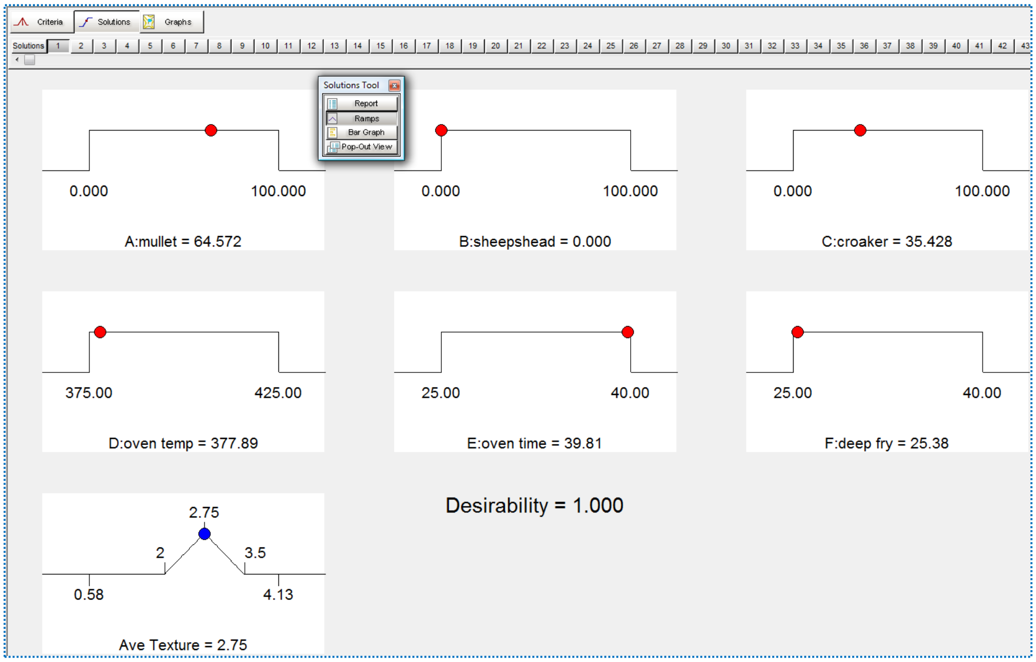
Find the Optimal Solution

The goal of the experimental program is to learn how to produce fish patties with a texture in the range of 2.00 to 3.50 x 10^3 grams. The target is 2.75. To find optimal combinations of formulas and processing, click the optimization node labeled **Numerical**. Then select **Ave Texture**. Select a **Goal** of **target->** and enter **2.75**. For **Limits** enter a **Lower** of **2** and **Upper** of **3.5**. Leave the weights and importance settings at their default levels. Your screen should now look like that below.



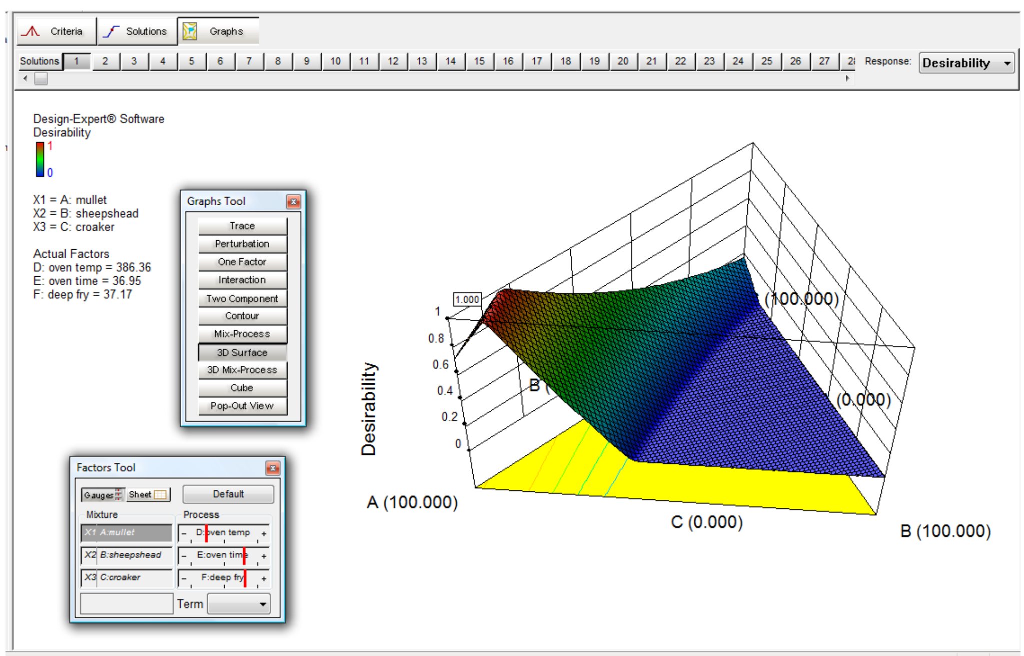
Optimization criteria for texture

Click the ***Solutions*** button. Then mouse to the **Solutions Tool** and select **Ramps**. Notice the many solutions listed atop your screen. (Due to the random nature of the optimization algorithm, your screen may show fewer or more solutions.) The software defaults to the most desirable solution.



One of many solutions for optimized texture <u>(your results may vary)</u>

Click solution number 2 and beyond: You will see many combinations that satisfy the texture requirements. Next press **Graphs** and look select from the **Graphs Tool** the **3D Surface**. Move your mouse cursor over the graph and when it turns into a hand (✋) grab it by depressing the left mouse button and rotate it to a vantage point like that shown below.

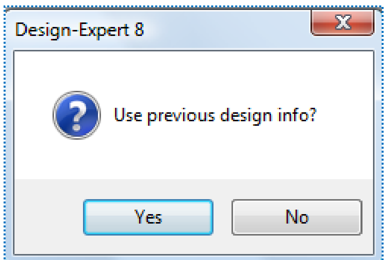


3D desirability view

Explore other desirability views if you like. Remember that by right-clicking any of the process factors on the Factors Tool, you can create a graph in process space. To preserve your modeling and optimization work, select **File**, **Save** or press the 🖫 icon.

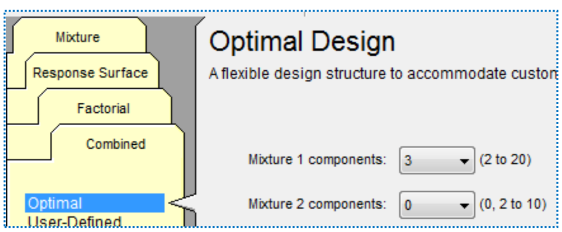
The Experiment via Optimal Design

As noted in the introduction, we want to reproduce the fish patty case exactly as reported by Cornell in his textbook *Experiments with Mixtures*. Now let's try an optimal design, which is much more efficient. We recommend you use this approach when designing your own mixture-process experiment. Assuming you are continuing from before, proceed by choosing **File**, **New Design**. The software asks whether to "**Use previous design info?**" Click **Yes**.



Re-using previous design information

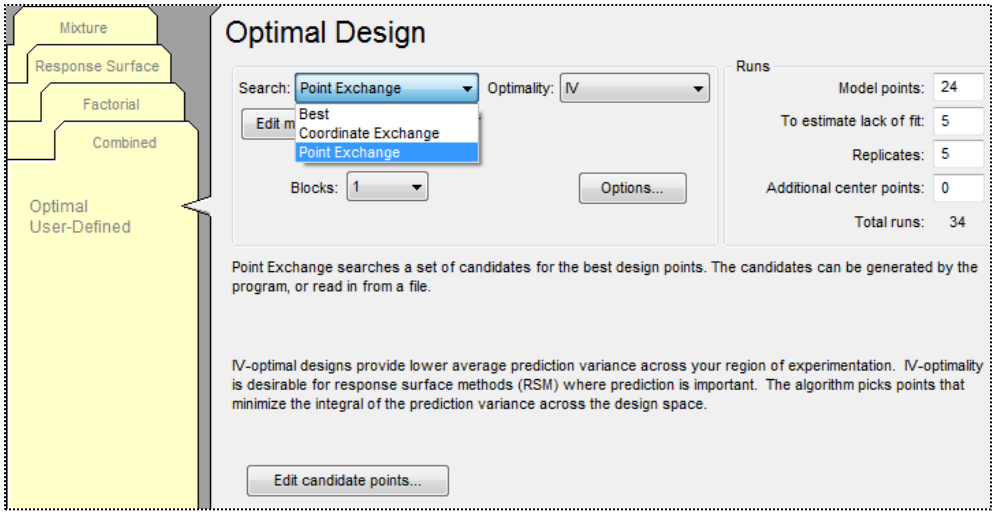
You see the builder from the previous user-defined design session. Select **Optimal**.



Optimal design option for combined mixture-process design

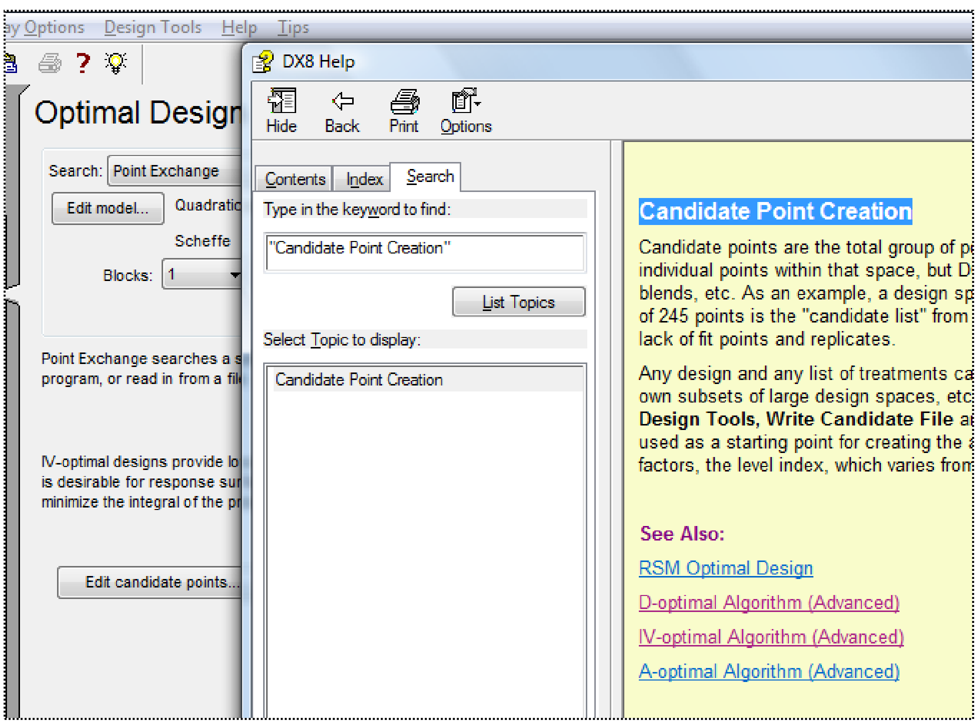
Press **Continue** to see that all component names and Low/High values remain filled in for you. Press **Continue** again to see your process factors, again at their previous ranges. Press **Continue** one more time to bring up the Optimal Design screen. Notice it's already set up for the quadratic-linear base model that you want for the fish-patties process-mixture design.

The objective of this exercise is to pick a subset of runs from Cornell's actual experiment so the search must be limited to specific combinations – not just any coordinate within the mixture-process space. Therefore you must change the **Search** droplist from its defaults or "Best" to **Point Exchange**, as shown below.



Optimal design search by Point Exchange

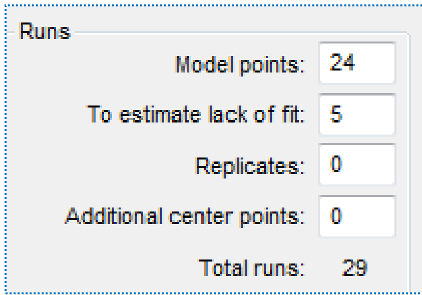
Leave the optimality criterion at its default of "IV" – a good choice in this case where continuous variables must be optimized. This criterion will dictate the selection of the minimal subset of model points from the candidate set, which in a moment you will specify as the original combinations run by Cornell. However before moving ahead, now would be a good time to view **Help** on the **Search** term **"Candidate Point Creation"**. From there you can link to details on the **IV-optimal** and alternative algorithms offered by Design-Expert.



Help on optimal design details

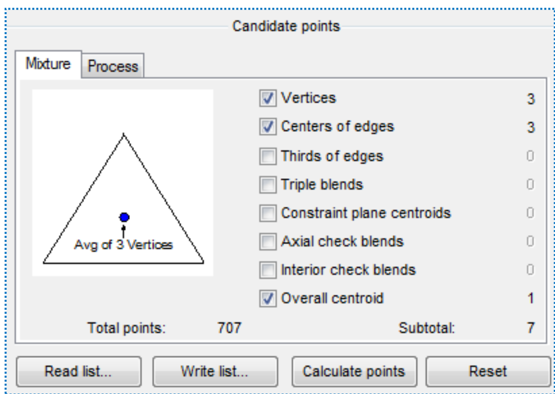
Exit out of Help (**X**) to get back to the work of building an optimal design for fish patty mixture and process development.

Because the original data had no replicates, set **Replicates** to **0**. Hit the tab key and you see a total of 29 runs. Leave "Model points" and "To estimate lack of fit" at their default levels.



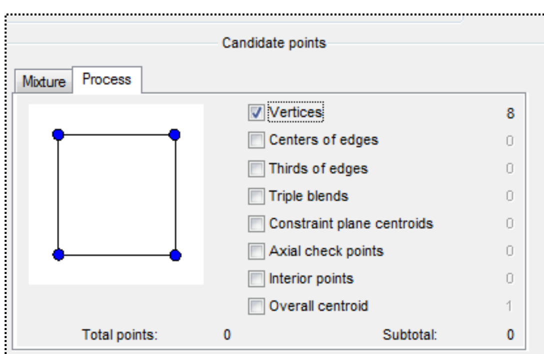
Default for replicates reduced to zero to match textbook case

Before continuing, you need to re-create the points originally selected by Cornell. (Recall doing this earlier when you set up the User Defined design.) Click **Edit candidate points.** In the **Mixture** tab, checkmark <u>off</u> Thirds of edges, Triple blends, **Axial check blends,** and **Interior check blends**. Press **Calculate points**. You now see a subtotal of 7 mixture points.



Mixture candidate points after deselecting some defaults

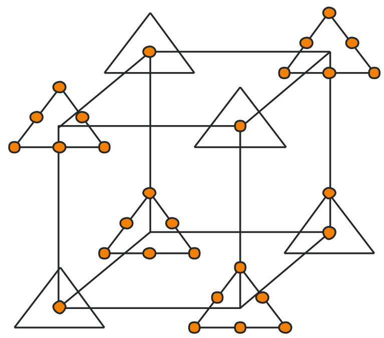
On this same screen, press the **Process** tab and checkmark <u>off all points except</u> for Vertices. Click **Calculate points**. This produces a subtotal of 8 process points. You now have 56 total candidate points, the same as earlier when you set up this case study as a User Defined design.



Candidate points total 56 after deselecting some defaults for process

Click **OK** to move on to the response specification. Accept the default response name and units (as in the user defined example) by pressing **Continue**.

Design-Expert now builds the optimal design. Due to random elements in the algorithm (configurable via Options), the 29 runs chosen may differ each time you do an Optimal design, but they will be essentially identical in their matrix attributes.



Fish patty design: one possible subset of 29 D-optimal combinations

Remember that the point of this follow up to the first part of the tutorial is to show how you can get by with only a fraction of all possible mixture-process combinations by using an optimal design – in this case one that includes combinations from within the original experiment. Thus, this is a "what-if" exercise. If you were to build a completely new design for this case, it would be advisable to include the 5 replicates that Design-Expert software suggests by default. This would provide 4 degrees of freedom to estimate pure error, which would allow a test for lack of fit, and generally improve the quality of the design.

Final comments

In all but the simplest cases, a User Defined design (all possible combinations of mixture and process points) is too large to be practical. Consider using an optimal design, which provides a reasonable subset of points to estimate the model you specify. For any combined design, but especially if done via optimal for a minimum-run experiment, it's advisable to over-ride any suggested model that falls short of what can be estimated without aliases. Then perform a backward elimination to rid the model of the large number of superfluous terms that are characteristic of these experiments that combine variables from the mixture and the process. If all goes well, you can blend your fish and bake it too. However, we have a suggestion: Include taste as one of your sensory attributes (not just texture!).