Problem 11-1 (as stated in *RSM Simplified*)

You will be glad we left this problem for last because it's a lollapalooza! A flexible part manufactured for medical use is made from four primary components:

- *Resin A.*
- *Cross-linker B.*
- *Polymer X.*
- *Polymer Y.*

The recipe for making this part is laid out as follows with acceptable ranges listed:

1. *Resin: 35 to 50 weight percent (wt. %) of copolymers (X and Y).*
2. *Cross-linker: 10 to 15 wt. % of copolymers.*
3. *Polymer ratio: 60/40 to 80/20 X-to-Y.*

The response is elongation–the higher the better. A Box-Behnken design (BBD) will be done to optimize the formulation on the basis of this response. But first some work must be done to set up proper ratios and translate them back to an actual composition.

The ratios can be defined as follows:

- $R_1 = A / (X + Y)$.
- $R_2 = B / (X + Y)$.
- $R_3 = X/Y$.

Table 11-3 shows the ranges of the ratios to be studied via the BBD. For reasons described in the sidebar titled "Dealing with Nonlinear Behavior of Ratios", it will be laid out in terms of natural logarithms (shown in parentheses).

Table 11-3: Ratio constraints

Ratio	Description	Ratio Range	Low – (ln)	High + (ln)
R_1	Resin A as percent of copolymer	35 to 50%	0.35 (-1.050)	0.5 (-0.693)
R_2	Crosslinker B as percent of copolymer	10 to 15%	0.10 (-2.303)	0.15 (-1.897)
R_3	Polymer X to polymer Y	60/40 to 80/20	1.5 (0.405)	4.0 (1.386)

The resulting BBD is shown in Table 11-4. We translated back from the log-scale to the original ratios by taking anti-logs. These will then be converted to compositions for experimental purposes. However, given the responses for elongation listed in Table 11-4, along with the layout of inputs in log-scale, you can develop a predictive model and perform the optimization (maximize).

Table 11-4: Box-Behnken design on formulation for medical part

	Coded			Actuals (done in natural log scale)			Elong.
#	R_1	R_2	R_3	R_1	R_2	R_3	%
1	-1	-1	0	-1.050	-2.303	0.896	150
2	1	-1	0	-0.693	-2.303	0.896	164
3	-1	1	0	-1.050	-1.897	0.896	93.7
4	1	1	0	-0.693	-1.897	0.896	129
5	-1	0	-1	-1.050	-2.100	0.405	147
6	1	0	-1	-0.693	-2.100	0.405	175
7	-1	0	1	-1.050	-2.100	1.386	181
8	1	0	1	-0.693	-2.100	1.386	220
9	0	-1	-1	-0.872	-2.303	0.405	145
10	0	1	-1	-0.872	-1.897	0.405	128
11	0	-1	1	-0.872	-2.303	1.386	206
12	0	1	1	-0.872	-1.897	1.386	154
13	0	0	0	-0.872	-2.100	0.896	149
14	0	0	0	-0.872	-2.100	0.896	155
15	0	0	0	-0.872	-2.100	0.896	149
16	0	0	0	-0.872	-2.100	0.896	152
17	0	0	0	-0.872	-2.100	0.896	148

The mixture constraint is:

- $A + B + X + Y = 1.$

From this and the ratio equations the actual composition of the polymer is derived as follows:

- $A = R_1/(R_1 + R_2 + 1)$.
- $B = R_2/(R_1 + R_2 + 1)$.
- $X = R_3/(R_3 + 1)(R_1 + R_2 + 1)$.
- $Y = 1/(R_3 + 1)(R_1 + R_2 + 1)$.

Table 11-5 shows the compositions based on the ratios from Table 11-4 for the BBD. This is necessary for carrying out the experiment.

Table 11-5: Compositions for experiment on medical part

#	Actuals (anti-logged)			Composition				Total
	R_1	R_2	R_3	A	B	C	D	
1	0.35	0.10	2.45	0.241	0.069	0.490	0.200	1.0
2	0.50	0.10	2.45	0.313	0.063	0.444	0.181	1.0
3	0.35	0.15	2.45	0.233	0.100	0.473	0.193	1.0
4	0.50	0.15	2.45	0.303	0.091	0.430	0.176	1.0
5	0.35	0.122	1.50	0.238	0.083	0.408	0.272	1.0
6	0.50	0.122	1.50	0.308	0.075	0.370	0.247	1.0
7	0.35	0.122	4.00	0.238	0.083	0.543	0.136	1.0
8	0.50	0.122	4.00	0.308	0.075	0.493	0.123	1.0
9	0.418	0.10	1.50	0.275	0.066	0.395	0.264	1.0
10	0.418	0.15	1.50	0.267	0.096	0.383	0.255	1.0
11	0.418	0.10	4.00	0.275	0.066	0.527	0.132	1.0
12	0.418	0.15	4.00	0.267	0.096	0.510	0.128	1.0
13	0.418	0.122	2.45	0.271	0.079	0.461	0.188	1.0
14	0.418	0.122	2.45	0.271	0.079	0.461	0.188	1.0
15	0.418	0.122	2.45	0.271	0.079	0.461	0.188	1.0
16	0.418	0.122	2.45	0.271	0.079	0.461	0.188	1.0
17	0.418	0.122	2.45	0.271	0.079	0.461	0.188	1.0

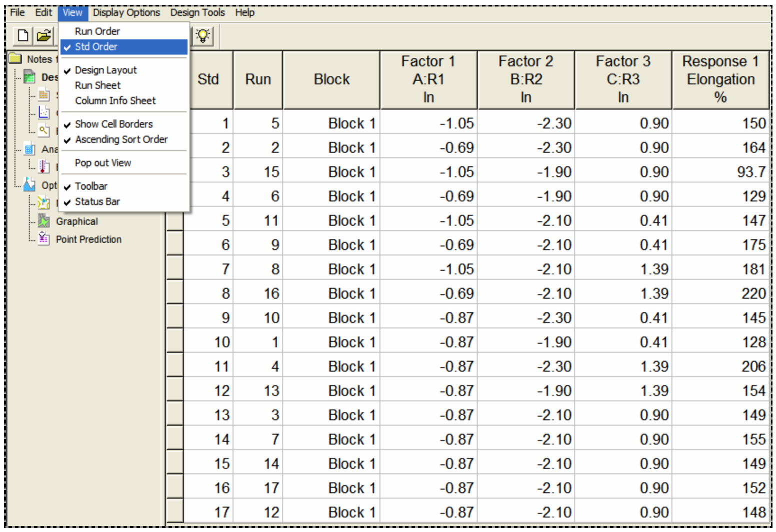
Ultimately you must translate the optimum point predicted from your model back to a composition by going through the same process detailed in Table 11-5:

1. *Antilog each of the factors levels to translate into actual ratios.*
2. *Plug-and-chug these through the ratio equations to solve for A, B, X, and Y, the resin, cross-linker, and two polymers, respectively.*

We never said this problem would be easy!

Solution to Problem 11-1

We will assume that by now you know how to set up a Box-Behnken design (BBD) from scratch using Design-Expert® software, so go ahead, run the program, go to **File** and **Open Design** and then **Open** the file named "**11-1 Prob - Medical polymer.dx7**" that we posted to the *RSM Simplified* website. To more easily compare what you see in this file with what's listed in Table 11-4, select **View** from the main menu and switch to **Std Order**.



Std	Run	Block	Factor 1 A:R1 ln	Factor 2 B:R2 ln	Factor 3 C:R3 ln	Response 1 Elongation %
1	5	Block 1	-1.05	-2.30	0.90	150
2	2	Block 1	-0.69	-2.30	0.90	164
3	15	Block 1	-1.05	-1.90	0.90	93.7
4	6	Block 1	-0.69	-1.90	0.90	129
5	11	Block 1	-1.05	-2.10	0.41	147
6	9	Block 1	-0.69	-2.10	0.41	175
7	8	Block 1	-1.05	-2.10	1.39	181
8	16	Block 1	-0.69	-2.10	1.39	220
9	10	Block 1	-0.87	-2.30	0.41	145
10	1	Block 1	-0.87	-1.90	0.41	128
11	4	Block 1	-0.87	-2.30	1.39	206
12	13	Block 1	-0.87	-1.90	1.39	154
13	3	Block 1	-0.87	-2.10	0.90	149
14	7	Block 1	-0.87	-2.10	0.90	155
15	14	Block 1	-0.87	-2.10	0.90	149
16	17	Block 1	-0.87	-2.10	0.90	152
17	12	Block 1	-0.87	-2.10	0.90	148

Figure 11-1.1: The design in standard order

Note that the input factors A, B and C are actually ratios of mixture ingredients transformed to natural logarithm scale ("ln"). This would be very inconvenient for experimentation purposes, so that's why we generated the 'recipe' sheet shown in Table 11-5 of *RSM Simplified*. At this stage, all the responses have been entered, so under the **Analysis** branch of the program, click **Elongation**.

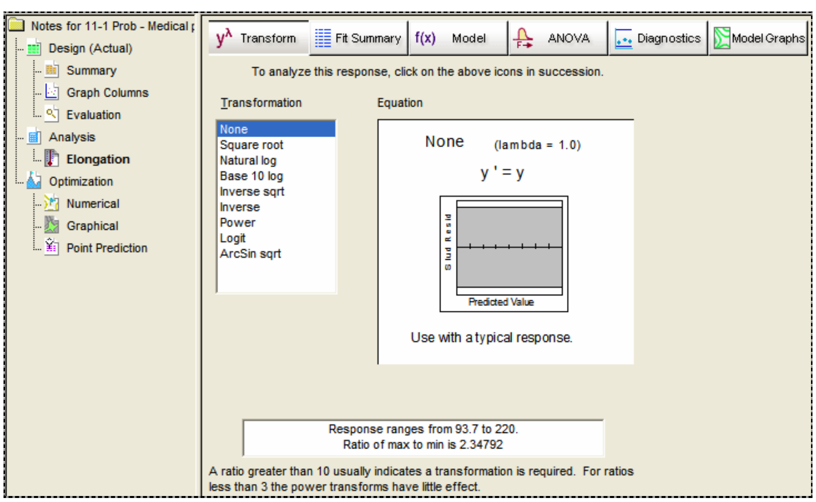


Figure 11-1.2: Performing the analysis

Press ahead to ***Fit Summary*** and note that the quadratic model is suggested.

Transform | Fit Summary | Model | ANOVA | Diagnostics | Model Graphs

Response 1 Elongation **Transform:** None

*** WARNING: The Cubic Model is Aliased! ***

Sequential Model Sum of Squares [Type I]

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Mean vs Total	4.117E+005	1	4.117E+005			
Linear vs Mean	8347.22	3	2782.41	7.08	0.0046	
2FI vs Linear	449.92	3	149.97	0.32	0.8097	
Quadratic vs 2FI	4519.29	3	1506.43	73.99	< 0.0001	Suggested
Cubic vs Quadratic	109.32	3	36.44	4.39	0.0935	Aliased
Residual	33.20	4	8.30			
Total	4.252E+005	17	25012.22			

"Sequential Model Sum of Squares [Type I]": Select the highest order polynomial where the additional terms are significant and the model is not aliased.

Lack of Fit Tests

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Linear	5078.53	9	564.28	67.99	0.0005	
2FI	4628.61	6	771.44	92.94	0.0003	
Quadratic	109.32	3	36.44	4.39	0.0935	Suggested
Cubic	0.000	0				Aliased
Pure Error	33.20	4	8.30			

"Lack of Fit Tests": Want the selected model to have insignificant lack-of-fit.

Figure 11-1.3: Fit summary

The lack of fit for this suggested model exhibits a p-value a bit lower than desired (<0.1), but the summary statistics look very good as you can see by scrolling down.

Model Summary Statistics

Source	Std. Dev.	R-Squared	Adjusted R-Squared	Predicted R-Squared	PRESS	
Linear	19.83	0.6202	0.5326	0.2164	10546.18	
2FI	21.59	0.6536	0.4458	-0.7320	23311.23	
Quadratic	4.51	0.9894	0.9758	0.8662	1801.04	Suggested
Cubic	2.88	0.9975	0.9901		+	Aliased

+ Case(s) with leverage of 1.0000: PRESS statistic not defined

"Model Summary Statistics": Focus on the model maximizing the "Adjusted R-Squared" and the "Predicted R-Squared".

Figure 11-1.4: Model summary statistics

Press ahead through the ***Model*** (accept full quadratic as suggested) and ***ANOVA*** (look this over) to the ***Diagnostics***. Notice that the normal plot of residuals looks good. On the **Diagnostics Tool** press **Predicted vs Actual**.

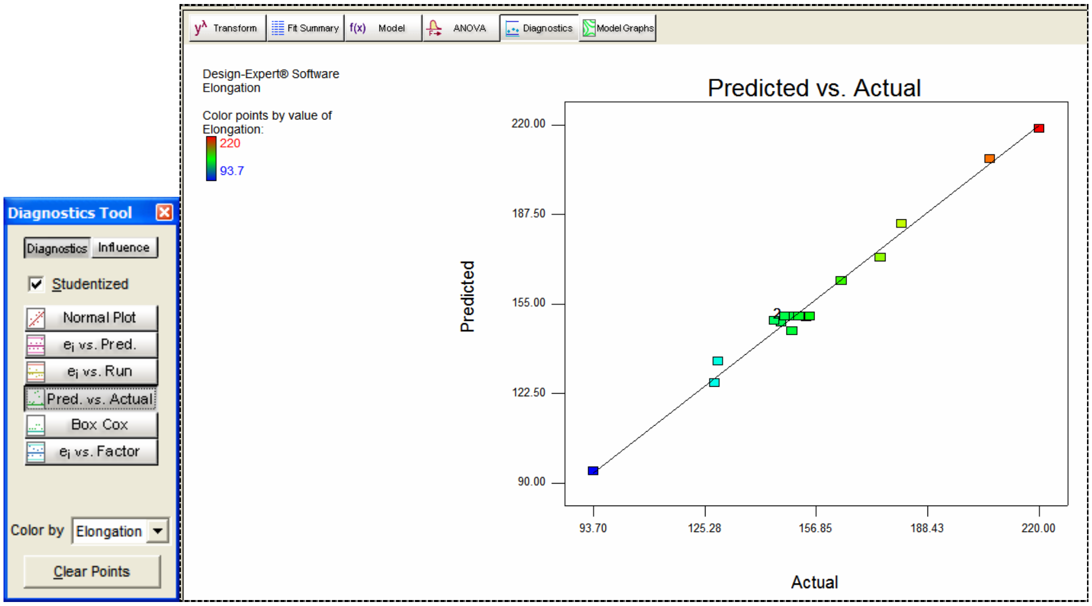


Figure 11-1.4: Predicted versus actual responses

Given the close agreement in predicted responses versus those actually observed, it seems safe to overlook the possible lack of fit. In any case, it appears to be of little importance as a practical matter.

Move on to ***Model Graphs*** and select **View**, **Perturbation**.

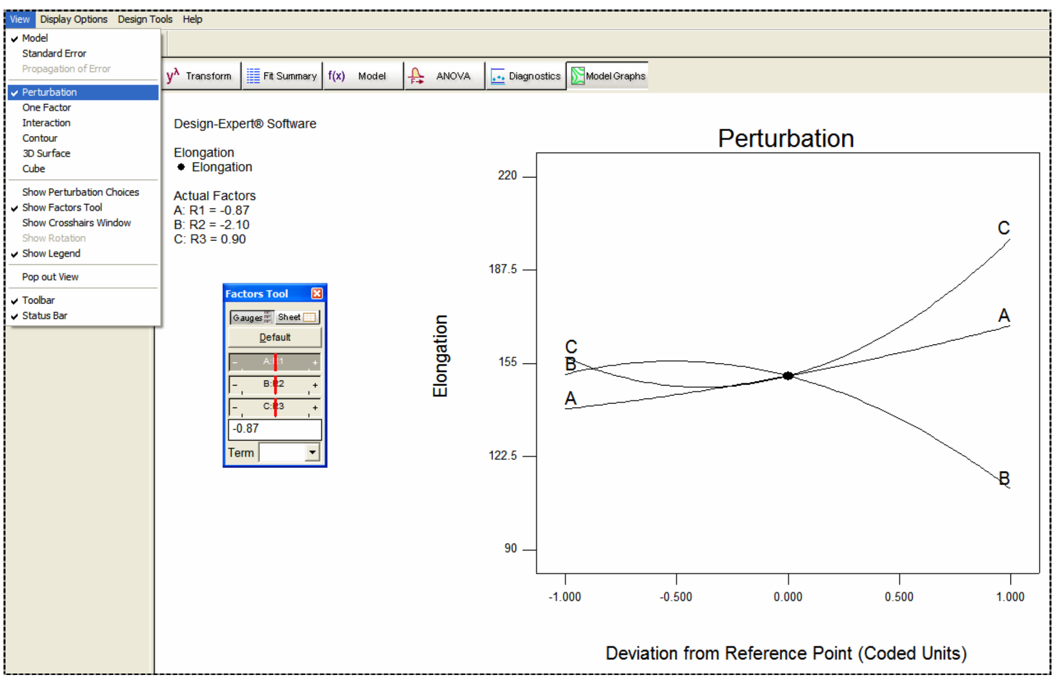


Figure 11-1.5: Perturbation plot

Notice that ratios B and C make a more dramatic impact on elongation than ratio A. Back on the **View** menu, go to the **Contour** plot. Then on the **Factors Tool**, right-click over the bar labeled **C:R3** and choose it for the **X1 axis**.

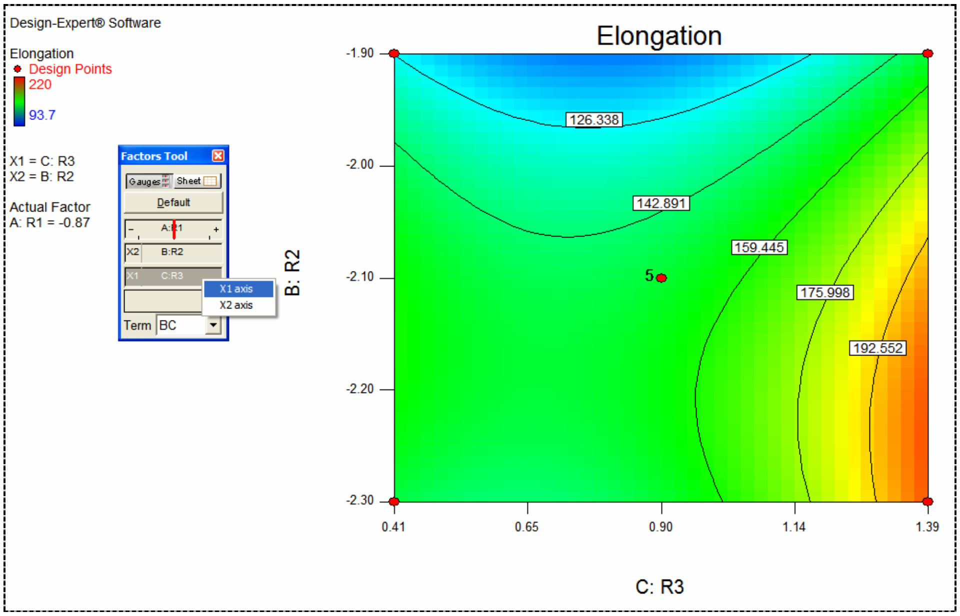


Figure 11-1.5: Contour plot with C specified for the X1 axis

Now select **View**, **3D Surface** and on the **Factors Tool** <u>slide the bar</u> for **A:R1** to the <u>right</u> (recall from the perturbation plot that higher is better for this factor).

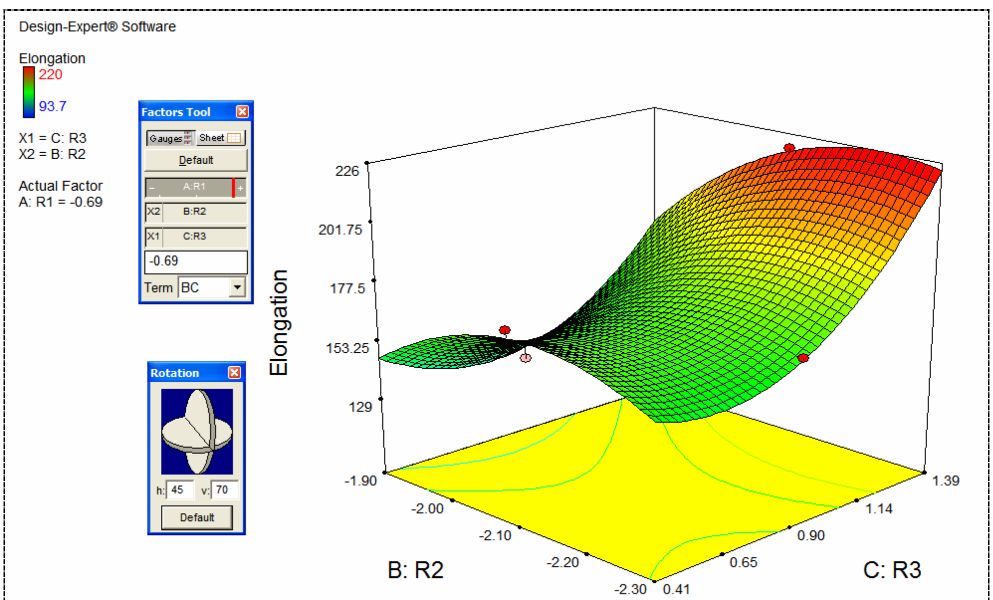


Figure 11-1.6: 3D plot with factor A 'sliced' at highest level

Now under the **Optimization** branch, choose the **Numerical** node and for the **Elongation** choose the **Goal** of **maximize** and change the **Limit** for the **Upper** side to **250**. This puts a 'stretch' on the goal to make the optimization try for more than just the observed maximum from the experiment.

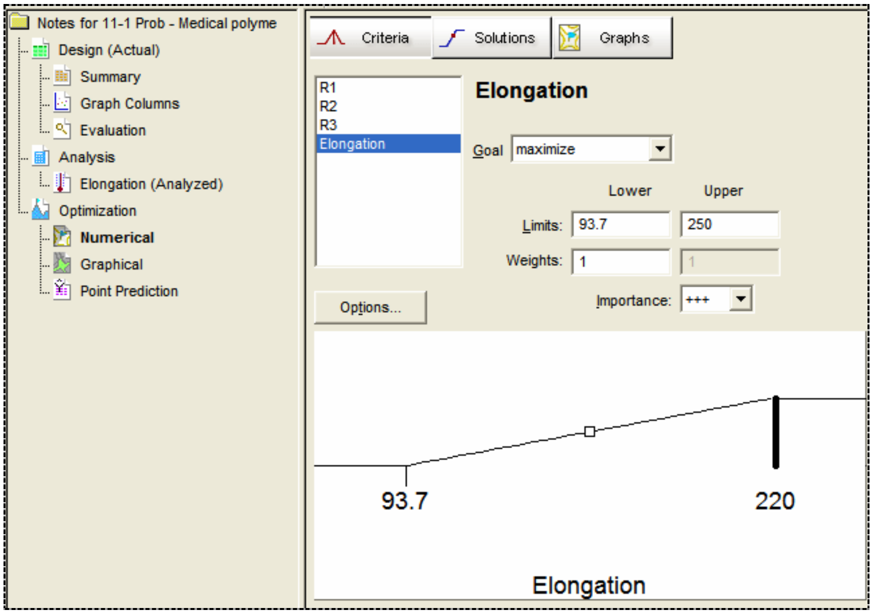
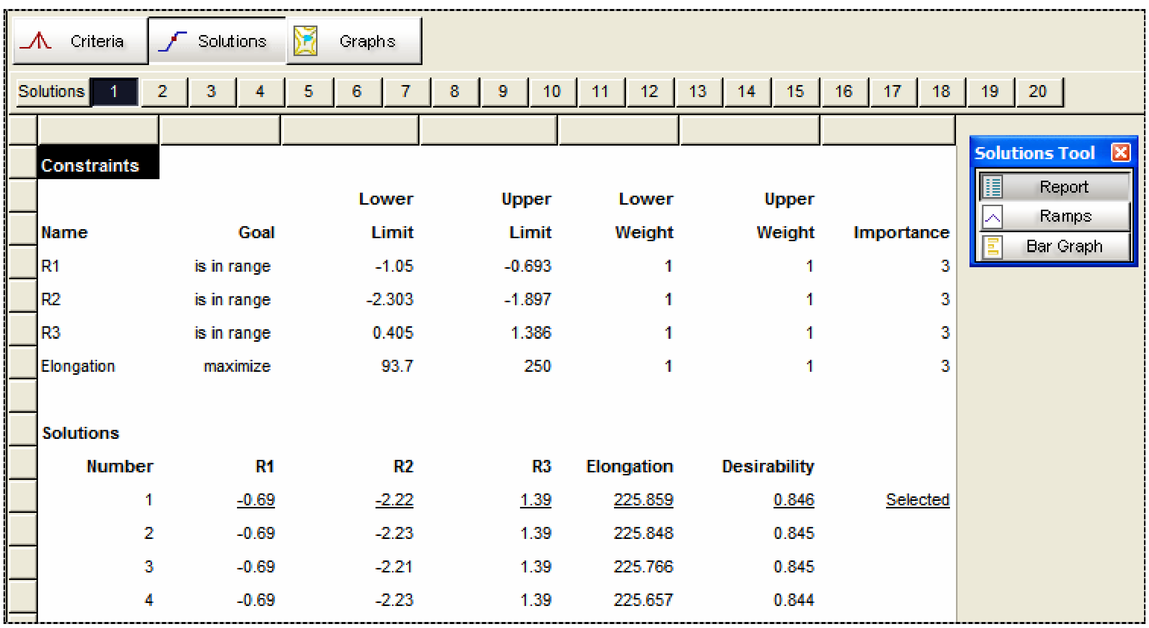


Figure 11-1.7: Setting the goal for elongation

Press ***Solutions*** to see what Design-Expert recommends for the ratios.



Constraints						
Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
R1	is in range	-1.05	-0.693	1	1	3
R2	is in range	-2.303	-1.897	1	1	3
R3	is in range	0.405	1.386	1	1	3
Elongation	maximize	93.7	250	1	1	3

Solutions						
Number	R1	R2	R3	Elongation	Desirability	
1	-0.69	-2.22	1.39	225.859	0.846	Selected
2	-0.69	-2.23	1.39	225.848	0.845	
3	-0.69	-2.21	1.39	225.766	0.845	
4	-0.69	-2.23	1.39	225.657	0.844	

Figure 11-1.8: Solutions report

Your results may vary a bit due to random elements in the numerical search, but you should find a maximum elongation of 225 or better at:

A. $\ln R1 \approx -0.693$ ($R_1 = e^{-0.693} = 0.500$)

B. $\ln R2 \approx -2.222$ ($R_2 = e^{-2.222} = 0.108$)

C. $\ln R3 \approx +1.386$ ($R_3 = e^{+1.386} = 3.999$)

Using the antilogs shown in parentheses above, the ideal composition is:

- $A = R_1/(R_1 + R_2 + 1) = 0.5/1.608 = 0.31$
- $B = R_2/(R_1 + R_2 + 1) = 0.108/1.608 \approx 0.07$
- $X = R_3/(R_3 + 1)(R_1 + R_2 + 1) = 3.999/(4.999)(1.608) \approx 4/8 = 0.50$
- $Y = 1/(R_3 + 1)(R_1 + R_2 + 1) = 1/8 \approx 0.12$

These fractions add up to a total of 1.00, so the recipe complies with the mixture constraint discussed in Chapter 11 of *RSM Simplified*.