

Latex Compatibility Studies for Zemea® Propanediol and Propylene Glycol



Performance is in our nature.

March 23, 2016



Study Goals

- Generate latex compatibility data comparing exposure to 1,3-propanediol (PDO) and propylene glycol (PG) under conditions set by ASTM D7661-10 (Tensile Strength of Male Condoms) and ASTM D638-14 (Tensile Properties of Plastics).
- Simplify test solution composition to directly test glycol compatibility for latex using a 2 hour exposure of latex to various glycol: water mixtures.
- Testing design and equipment:
 - Collect: tensile strength at break, elongation at break, peak load, and physical data for sample (thickness and width).
 - Instrument: Instron 5967 with 1 kN load cell and Mitutoyo micrometer and calipers. Crosshead speed = 5 in./min.
 - Die-cut latex rubber (0.01 inches thick x 0.13 inches wide; measured for each test).
 - 2 hour exposure to a glycol:water mixture at 40.0°C.

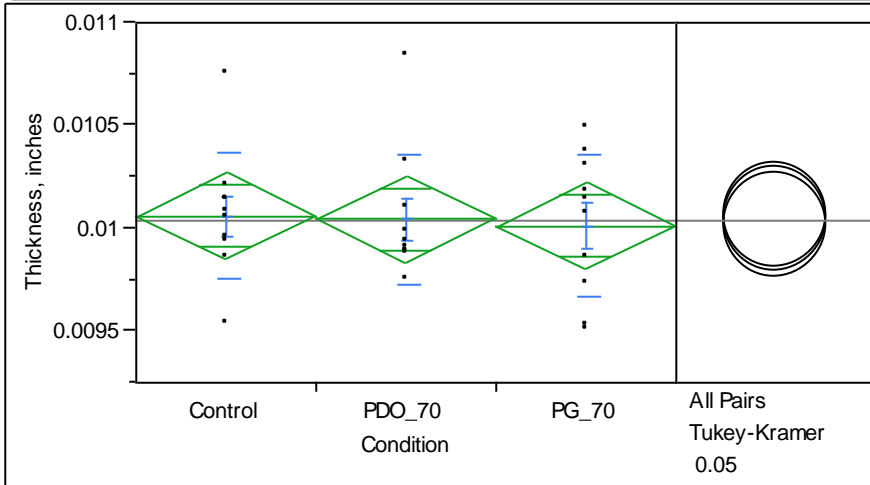
Study #1

Design: Direct comparison of PDO and PG at a high concentration. A control is used to test whether glycol exposure impacts physical properties of the latex.

- Design justification: Personal warming gels are reported to contain high levels of PG, up to 90%. This is the same lot of PDO used by the customer.
- **Condition 1 (control)**: No glycol exposure (dry latex). Incubated dry for 2 hours at 40°C.
- **Condition 2 (PDO_70)**: A 2 hour exposure to Zemea® propanediol + HPLC-grade water, 70:30 (v:v).
Lot# 80562653 Labware # 10020938
- **Condition 3 (PG_70)**: A 2 hour exposure to Propylene Glycol USP/FCC + HPLC-grade water, 70:30.
Fisher Scientific, Product# P355-4
Lot# 152503

Study #1 Data – Latex Sample Quality

Oneway Analysis of Thickness, inches By Condition



Means and Std Deviations

Level	Number	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
Control	10	0.010060	0.000309	0.0001	0.00984	0.01028
PDO_70	10	0.010044	0.000317	0.00010	0.00982	0.01027
PG_70	10	0.010012	0.000347	0.00011	0.00976	0.01026

Means Comparisons

Comparisons for all pairs using Tukey-Kramer HSD

	q*	Alpha	
	2.47942	0.05	
Abs(Dif)-LSD			
	Control	PDO_70	PG_70
Control	-0.00036	-0.00034	-0.00031
PDO_70	-0.00034	-0.00036	-0.00033
PG_70	-0.00031	-0.00033	-0.00036

Positive values show pairs of means that are significantly different.

- No differences in latex thickness were observed between the groups (α 0.05).
- Variance for thickness was minimal for 10 independent measurements:

$$\text{COV, \%} = (\text{Std Dev}/\text{Mean}) * 100$$

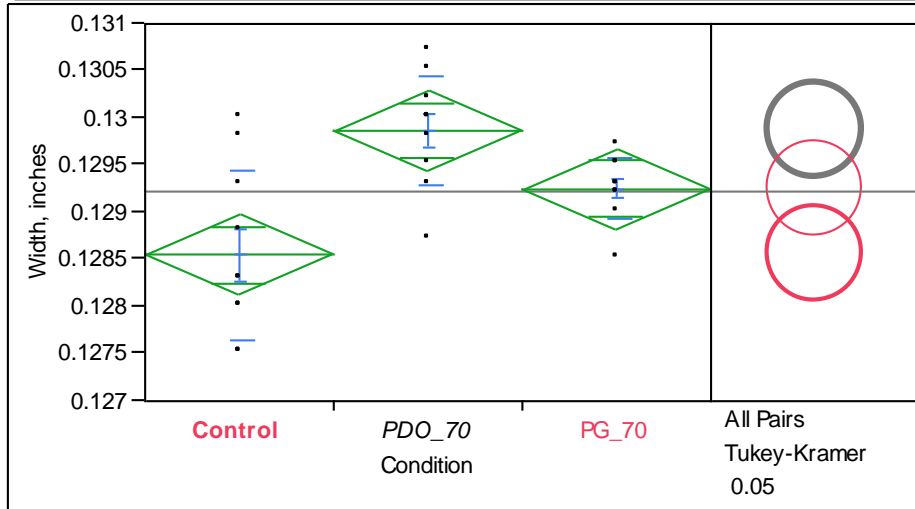
$$\text{COV PDO} = 3.2\%$$

$$\text{COV Control} = 3.07\%$$

$$\text{COV PG} = 3.47\%$$

Study #1 Data – Latex Sample Quality

Oneway Analysis of Width, inches By Condition



- Differences were observed in latex width for the die-cut samples (α 0.05).
- These differences did NOT have a significant impact on tensile strength ($r^2 < 10\%$).
- Variance in width was minimal for 10 independent measurements:

$$\text{COV, \%} = (\text{Std Dev}/\text{Mean}) * 100$$

$$\text{COV PDO} = \mathbf{0.45\%}$$

$$\text{COV Control} = \mathbf{0.70\%}$$

$$\text{COV PG} = \mathbf{0.25\%}$$

Means and Std Deviations

Level	Number	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
Control	10	0.128550	0.000896	0.00028	0.12791	0.12919
PDO_70	10	0.129870	0.000585	0.00019	0.12945	0.13029
PG_70	10	0.129250	0.000327	0.00010	0.12902	0.12948

Means Comparisons

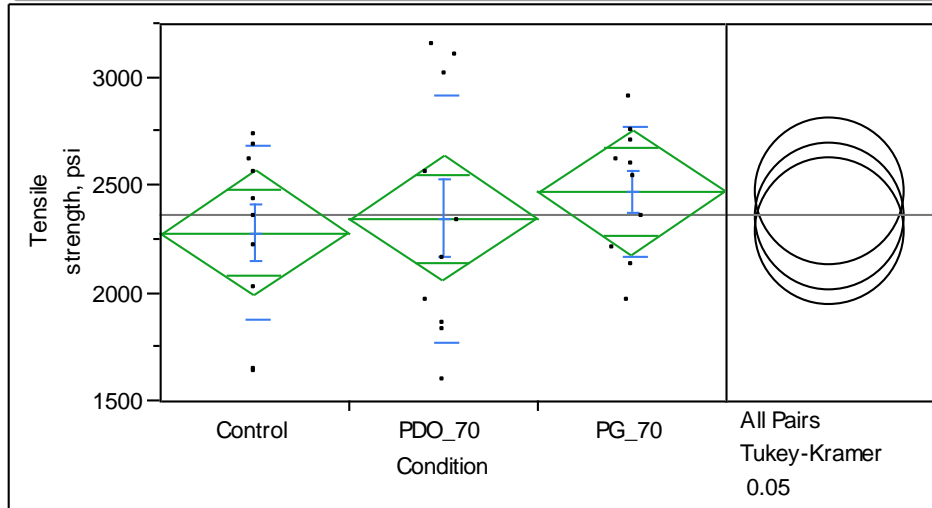
Comparisons for all pairs using Tukey-Kramer HSD

	q*	Alpha	
	2.47942	0.05	
Abs(Dif)-LSD			
	PDO_70	PG_70	Control
PDO_70	-0.00072	-0.00010	0.00060
PG_70	-0.00010	-0.00072	-0.00002
Control	0.00060	-0.00002	-0.00072

Positive values show pairs of means that are significantly different.

Study #1 Data

Oneway Analysis of Tensile strength, psi By Condition



Means and Std Deviations

Level	Number	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
Control	10	2281.20	405.164	128.12	1991.4	2571.0
PDO_70	10	2348.30	573.963	181.50	1937.7	2758.9
PG_70	10	2470.80	301.700	95.41	2255.0	2686.6

Means Comparisons

Comparisons for all pairs using Tukey-Kramer HSD

q*	Alpha			
2.47942	0.05			
Abs(Dif)-LSD				
	PG_70	PDO_70	Control	
PG_70	-489.48	-366.98	-299.88	
PDO_70	-366.98	-489.48	-422.38	
Control	-299.88	-422.38	-489.48	

Positive values show pairs of means that are significantly different.

- No differences in tensile strength were observed between conditions (α 0.05). Latex tensile strength is marginally higher than the control for latex exposed to 70% PDO, and 70% PG, respectively.
- Significant** variance in tensile strength was observed for 10 independent measurements:

$$\text{COV, \%} = (\text{Std Dev}/\text{Mean}) * 100$$

$$\text{COV PDO} = \mathbf{24.4\%}$$

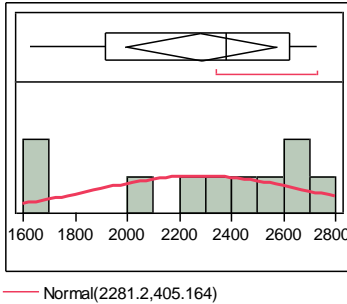
$$\text{COV Control} = \mathbf{17.8\%}$$

$$\text{COV PG} = \mathbf{12.2\%}$$

Study #1 Data

Distributions Condition=Control

Tensile strength, psi



Quantiles

100.0%	maximum	2729.0
99.5%		2729.0
97.5%		2729.0
90.0%		2723.8
75.0%	quartile	2624.5
50.0%	median	2382.0
25.0%	quartile	1918.0
10.0%		1630.6
2.5%		1630.0
0.5%		1630.0
0.0%	minimum	1630.0

Moments

Mean	2281.2
Std Dev	405.16411
Std Err Mean	128.12414
upper 95% Mean	2571.0369
low er 95% Mean	1991.3631
N	10

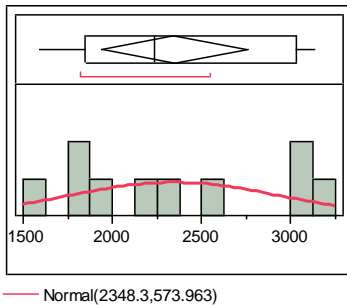
Fitted Normal

Parameter Estimates

Type	Parameter	Estimate	Lower 95%	Upper 95%
Location	μ	2281.2	1991.3631	2571.0369
Dispersion	σ	405.16411	278.68614	739.67172

Distributions Condition=PDO_70

Tensile strength, psi



Quantiles

100.0%	maximum	3144.0
99.5%		3144.0
97.5%		3144.0
90.0%		3139.5
75.0%	quartile	3027.0
50.0%	median	2236.5
25.0%	quartile	1844.3
10.0%		1607.1
2.5%		1583.0
0.5%		1583.0
0.0%	minimum	1583.0

Moments

Mean	2348.3
Std Dev	573.96304
Std Err Mean	181.50305
upper 95% Mean	2758.8884
low er 95% Mean	1937.7116
N	10

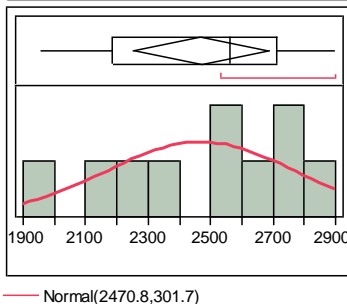
Fitted Normal

Parameter Estimates

Type	Parameter	Estimate	Lower 95%	Upper 95%
Location	μ	2348.3	1937.7116	2758.8884
Dispersion	σ	573.96304	394.79198	1047.8328

Distributions Condition=PG_70

Tensile strength, psi



Quantiles

100.0%	maximum	2898.0
99.5%		2898.0
97.5%		2898.0
90.0%		2882.3
75.0%	quartile	2710.3
50.0%	median	2562.0
25.0%	quartile	2183.3
10.0%		1971.9
2.5%		1955.0
0.5%		1955.0
0.0%	minimum	1955.0

Moments

Mean	2470.8
Std Dev	301.70029
Std Err Mean	95.40601
upper 95% Mean	2686.6234
low er 95% Mean	2254.9766
N	10

Fitted Normal

Parameter Estimates

Type	Parameter	Estimate	Lower 95%	Upper 95%
Location	μ	2470.8	2254.9766	2686.6234
Dispersion	σ	301.70029	207.52008	550.78713

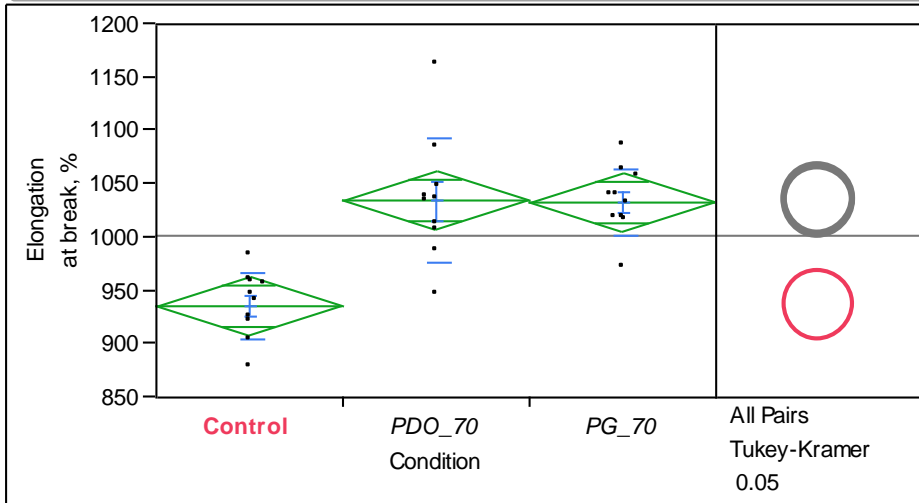
- Distribution of the 10 individual tensile strength measurements appeared random, which indicated that the tensile strength measurement were naturally variable.

- DT&L would suggest using a target COV of less than **10%** for the tensile strength measurement.

- The sample size required is estimated at **28 independent observations**.

Study #1 Data

Oneway Analysis of Elongation at break, % By Condition



Means and Std Deviations

Level	Number	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
Control	10	936.30	31.0145	9.808	914.1	958.5
PDO_70	10	1034.39	57.9119	18.313	993.0	1075.8
PG_70	10	1033.18	31.3236	9.905	1010.8	1055.6

Means Comparisons

Comparisons for all pairs using Tukey-Kramer HSD

	q*	Alpha	
	2.47942	0.05	
Abs(Dif)-LSD			
	PDO_70	PG_70	Control
PDO_70	-46.592	-45.382	51.498
PG_70	-45.382	-46.592	50.288
Control	51.498	50.288	-46.592

Positive values show pairs of means that are significantly different.

- Data for elongation at break indicated that glycol exposure alters physical properties of the latex.
- Both PDO and PG improved latex elongation by 10.5%
- Variance in elongation was minimal for 10 independent measurements:

$$\text{COV, \%} = (\text{Std Dev}/\text{Mean}) * 100$$

$$\text{COV PDO} = 5.6\%$$

$$\text{COV Control} = 3.3\%$$

$$\text{COV PG} = 3.03\%$$

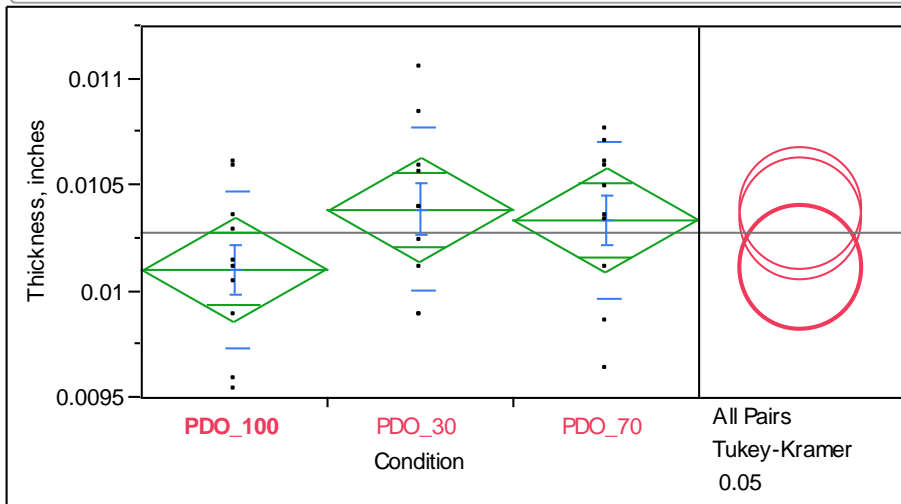
Study #2

Design: Dose-response study evaluating the effect of 1,3-propanediol concentration on physical properties of latex.

- Design justification: The impact of PDO on physical properties of latex may differ at high or low PDO concentrations. A block design for two different lots of 70% PDO was completed to compare different lots.
- **Condition 1 (PDO_30)**: 2 hour exposure to Zemea® propanediol + HPLC-grade water, **30:70** (v:v)
Lot# PT15K00007
- **Condition 2 (PDO_70)**: 2 hour exposure to Zemea® propanediol + HPLC-grade water, **70:30** (v:v)
Lot# PT15K00007
- **Condition 3 (PDO_100)**: 2 hour exposure to Zemea® propanediol + HPLC-grade water, **100:0** (v:v)
Lot# PT15K00007

Study #2 Data – Latex Sample Quality

Oneway Analysis of Thickness, inches By Condition



- No differences in latex thickness were observed between the groups (α 0.05).
- Variance for thickness was minimal for 10 independent measurements:

$$\text{COV, \%} = (\text{Std Dev}/\text{Mean}) * 100$$

$$\text{COV PDO}_{30} = 3.7\%$$

$$\text{COV PDO}_{70} = 3.6\%$$

$$\text{COV PDO}_{100} = 3.7\%$$

Means and Std Deviations

Level	Number	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
PDO_100	10	0.010106	0.000369	0.00012	0.00984	0.01037
PDO_30	10	0.010386	0.000383	0.00012	0.01011	0.01066
PDO_70	10	0.010337	0.000372	0.00012	0.01007	0.01060

Means Comparisons

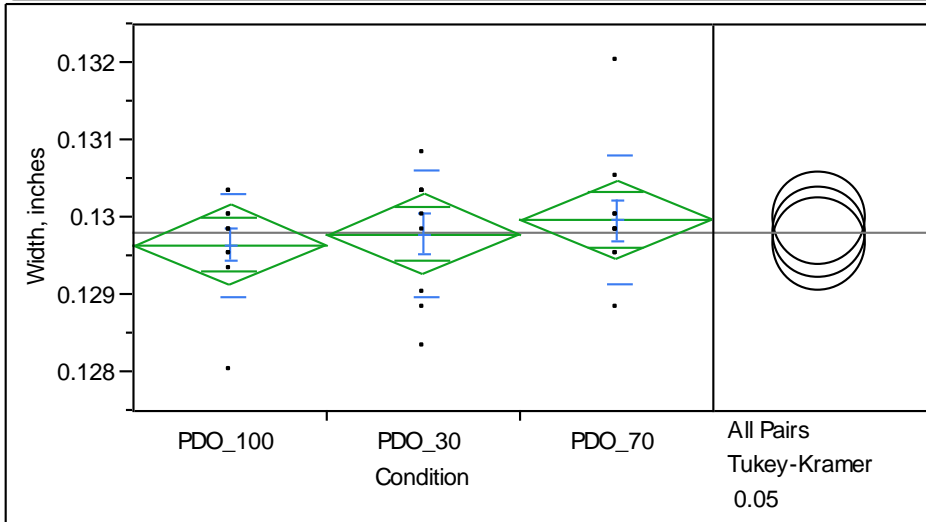
Comparisons for all pairs using Tukey-Kramer HSD

	q*	Alpha	Abs(Dif)-LSD		
	2.47942	0.05			
			PDO_30	PDO_70	PDO_100
PDO_30			-0.00042	-0.00037	-0.00014
PDO_70			-0.00037	-0.00042	-0.00018
PDO_100			-0.00014	-0.00018	-0.00042

Positive values show pairs of means that are significantly different.

Study #2 Data – Latex Sample Quality

Oneway Analysis of Width, inches By Condition



Means and Std Deviations

Level	Number	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
PDO_100	10	0.129650	0.000669	0.00021	0.12917	0.13013
PDO_30	10	0.129790	0.000812	0.00026	0.12921	0.13037
PDO_70	10	0.129970	0.000837	0.00026	0.12937	0.13057

Means Comparisons

Comparisons for all pairs using Tukey-Kramer HSD

	q*	Alpha	
	2.47942	0.05	
Abs(Dif)-LSD			
	PDO_70	PDO_30	PDO_100
PDO_70	-0.00086	-0.00068	-0.00054
PDO_30	-0.00068	-0.00086	-0.00072
PDO_100	-0.00054	-0.00072	-0.00086

Positive values show pairs of means that are significantly different.

- No differences were observed in latex width for the die-cut samples (α 0.05).
- Variance in width was minimal for 10 independent measurements:

$$\text{COV, \%} = (\text{Std Dev}/\text{Mean}) * 100$$

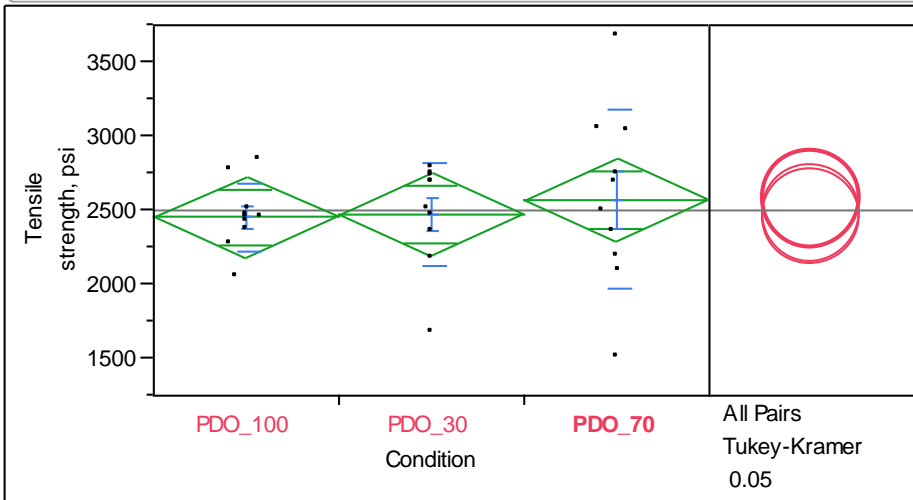
$$\text{COV PDO}_{30} = \mathbf{0.63\%}$$

$$\text{COV PDO}_{70} = \mathbf{0.64\%}$$

$$\text{COV PDO}_{100} = \mathbf{0.52\%}$$

Study #2 Data

Oneway Analysis of Tensile strength, psi By Condition



Means and Std Deviations

Level	Number	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
PDO_100	10	2452.30	227.775	72.03	2289.4	2615.2
PDO_30	10	2473.90	344.527	108.95	2227.4	2720.4
PDO_70	10	2571.70	602.596	190.56	2140.6	3002.8

Means Comparisons

Comparisons for all pairs using Tukey-Kramer HSD

q*	Alpha			
2.47942	0.05			
Abs(Dif)-LSD				
		PDO_70	PDO_30	PDO_100
PDO_70		-467.69	-369.89	-348.29
PDO_30		-369.89	-467.69	-446.09
PDO_100		-348.29	-446.09	-467.69

Positive values show pairs of means that are significantly different.

- No differences in tensile strength were observed for 30%, 70%, or 100% PDO ($\alpha = 0.05$).
- Significant** variance in tensile strength was observed for 10 independent measurements:

$$\text{COV, \%} = (\text{Std Dev}/\text{Mean}) * 100$$

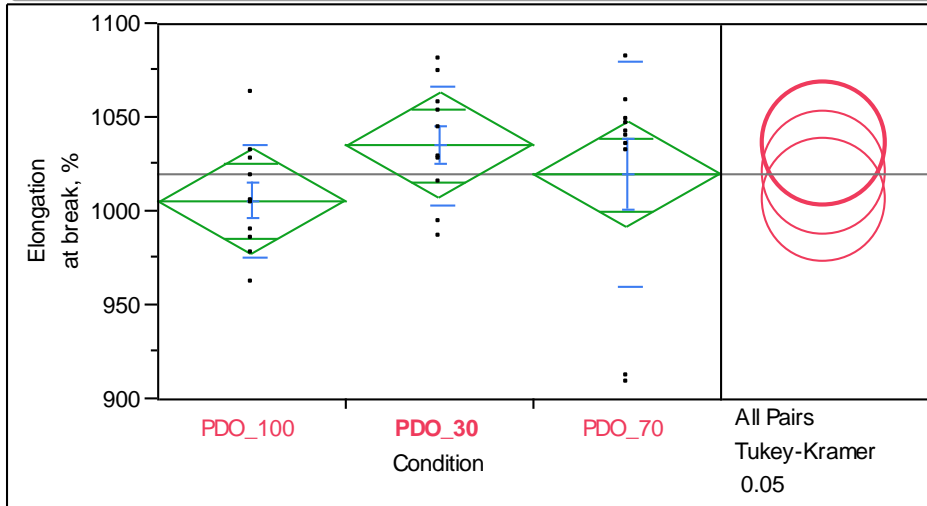
$$\text{COV PDO}_{30} = \mathbf{13.93\%}$$

$$\text{COV PDO}_{70} = \mathbf{23.43\%}$$

$$\text{COV PDO}_{100} = \mathbf{9.29\%}$$

Study #2 Data

Oneway Analysis of Elongation at break, % By Condition



Means and Std Deviations

Level	Number	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
PDO_100	10	1005.60	29.7690	9.414	984.3	1026.9
PDO_30	10	1035.38	31.6461	10.007	1012.7	1058.0
PDO_70	10	1019.67	59.8455	18.925	976.9	1062.5

Means Comparisons

Comparisons for all pairs using Tukey-Kramer HSD

	q*	Alpha	Abs(Dif)-LSD		
	2.47942	0.05	PDO_30	PDO_70	PDO_100
PDO_30			-47.344	-31.634	-17.564
PDO_70			-31.634	-47.344	-33.274
PDO_100			-17.564	-33.274	-47.344

Positive values show pairs of means that are significantly different.

- No differences were observed for elongation at break for three concentrations of PDO, 30%, 70% and 100%.
- Elongation at break trended higher with increasing concentration of water (inversely correlated with PDO concentration).
- Variance in elongation was minimal for 10 independent measurements:

$$\text{COV, \%} = (\text{Std Dev}/\text{Mean}) * 100$$

$$\text{COV PDO}_{30} = 3.06\%$$

$$\text{COV PDO}_{70} = 5.87\%$$

$$\text{COV PDO}_{100} = 2.96\%$$

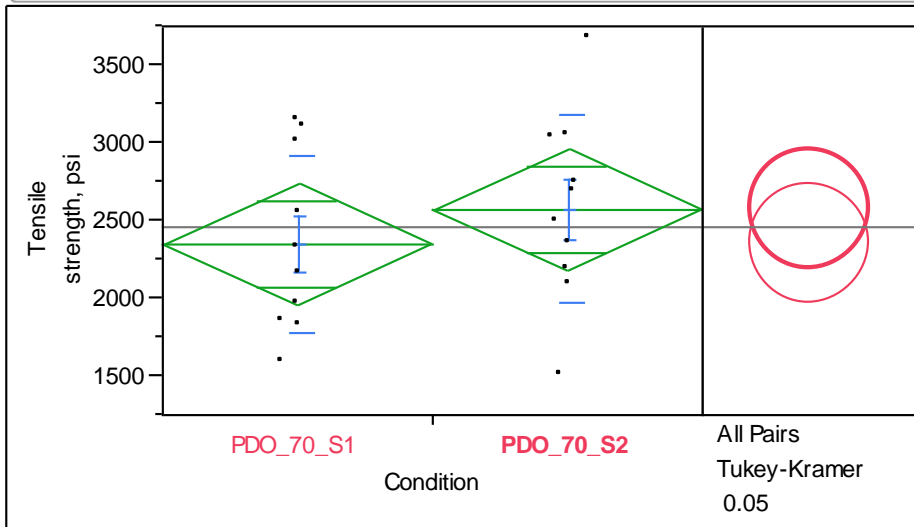
Study #1 & #2 Data – Lot Comparison

Design: Lot comparison via block design for two different lots of Zemea® propanediol at a concentration of 70%.

- **Condition S1 (PDO_70_S1)**: A 2 hour exposure to Zemea® propanediol + HPLC-grade water, 70:30 (v:v).
Lot# 80562653 Labware # 1002093
Retainer for the Zemea® propanediol used by the customer.
- **Condition S2 (PDO_70_S2)**: 2 hour exposure to Zemea® propanediol + HPLC-grade water, 70:30 (v:v)
Lot# PT15K00007
Zemea® USP propanediol (higher purity).

Study #1 & #2 Data – Lot Comparison

Oneway Analysis of Tensile strength, psi By Condition



- No differences in tensile strength were observed between two different lots of Zemea® propanediol, tested at a concentration of 70% (α 0.05).
- **Significant** variance in tensile strength was observed for 10 independent measurements:

$$\text{COV, \%} = (\text{Std Dev}/\text{Mean}) * 100$$

$$\text{COV PDO}_70_S1 = \mathbf{24.4\%}$$

$$\text{COV PDO}_70_S2 = \mathbf{23.4\%}$$

Means and Std Deviations

Level	Number	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
PDO_70_S1	10	2348.30	573.963	181.50	1937.7	2758.9
PDO_70_S2	10	2571.70	602.596	190.56	2140.6	3002.8

Means Comparisons

Comparisons for all pairs using Tukey-Kramer HSD

	q*	Alpha
	2.10092	0.05
Abs(Dif)-LSD		
	PDO_70_S2	PDO_70_S1
PDO_70_S2	-552.89	-329.49
PDO_70_S1	-329.49	-552.89

Positive values show pairs of means that are significantly different.

Conclusions

- No significant differences in tensile strength were observed for samples of latex rubber that were exposed to PG (70%) or PDO (70%) for a period of 2 hours at 40°C.
- Tensile strength of latex rubber was not impacted by concentration of PDO over a range of 30% to 100%.
- There were no significant differences detected for two lots of Zemea® propanediol, differing in purity (Zemea® propanediol *versus* Zemea® USP propanediol).
- Tensile strength measurements of latex were shown to have significant variance for a population of 10 independent measurements. This variance appears to be associated with the tensile strength measurement, since other measurements achieve significantly lower COVs.
- In order to acquire a COVs of less than 10%, the sample number for tensile strength measurements should be increased to a minimum of 28 independent observations.



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