

Navigating the Complexities of Rubber Development:

Development:

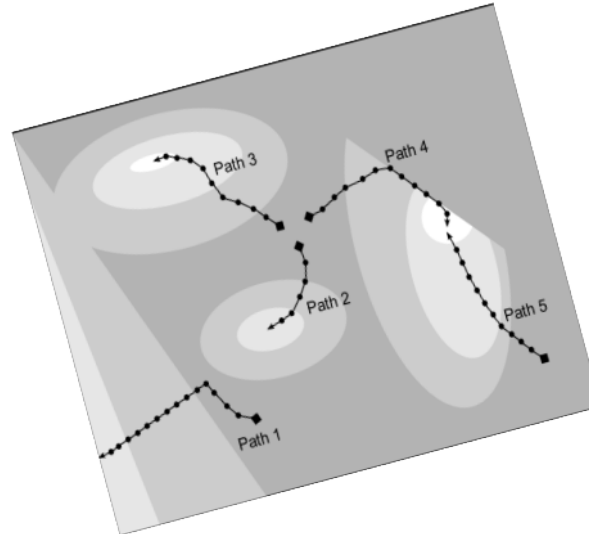
Bridging AI and DoE



Dr. Hans-Joachim Graf

Navigating the Complexities of Rubber Development

- **Content**
 - **Introduction**
 - **Development Tools**
 - Design of Experiments
 - Artificial Intelligence
 - **Datastructure**
 - Data Cloud
 - Data Quality
 - Managing Data
 - **Conclusion**



Development Tools

◆ DoE – New Materials / Processes

- **Model** / Ingredient Selection & Limits / *Perform Experiments* / Select Result / Confirmation Experiment

◆ AI – Historic Compound / Process Data

- **Data** / Criteria / *Prediction* / Select Result / Confirmation Experiment

Navigating the Complexities of Rubber Development



- Prediction based on AI

Code	Cost	Density	Ingredients	S0ALS11	S0ALS12	S0ALS13	S0ALS14	S0ALS15	S0ALS16
A001	200.00	0.82	NR (SMR - 10)	100.00	100.00	100.00	100.00	100.00	100.00
B003	115.00	1.80	N2O5	10.00	30.00	50.00	25.00	40.00	75.00
C010	24.00	2.71	CaCO3	20.00	20.00	20.00	20.00	20.00	20.00
D002	116.00	0.89	Naphtenic Oil	5.00	25.00	40.00	5.00	25.00	40.00
E001	385.00	5.80	ZnO	5.00	5.00	5.00	5.00	5.00	5.00
F001	165.00	0.82	Stearic Acid	2.00	2.00	2.00	2.00	2.00	2.00
G001	824.00	1.15	PPD	2.00	2.00	2.00	2.00	2.00	2.00
H001	158.00	1.80	S	1.50	1.50	1.50	1.50	1.50	1.50
I001	306.00	1.11	TMTD - 80	0.65	0.65	0.65	0.65	0.65	0.65
K005	708.00	1.28	CBS - 80	0.65	0.65	0.65	0.65	0.65	0.65

- Prediction based on DoE

Factor	Name	Level	Low Level	High Level	Std. Dev.	Coding
A	5-80	2.00	1.00000	3.00	0.00000	Actual
B	MBTS-80	1.30	0.00000	2.00	0.00000	Actual
C	ZDT/r	4.00	2.00	6.00	0.00000	Actual

GrafCompounder 5.0 (Screen shot)

Point Prediction Tool in Design Expert®12 Software (Screen shot)

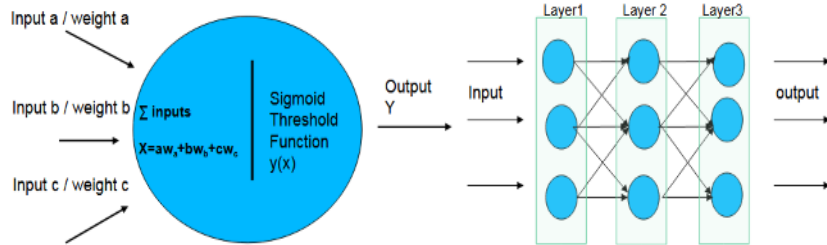


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- **Data Calculation AI**

- **Data Calculation DoE**

Artificial Intelligence: Neuronal Network and Algorithm

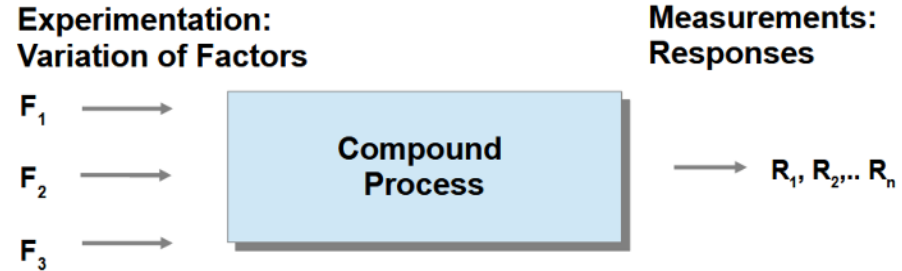


Extended model of an artificial neuron with moderated input weights

A model with multiple layers of neurons, each connected to the preceding and succeeding layer.

- Weights could be assigned to the connections between nodes

Source: T. Rashid, Neuronal Nets, 2017



Objective of the Experiment is the identification of the factors ($F_1...F_n$) type of influence on the responses ($R_1...R_n$) and description with mathematical equations for further processing.

ANOVA is used for statistical evaluation.

$$R_{i(1...n)} = f(A_0 + A_1F_1 + \dots + A_nF_n + \dots)$$

- **Algorithm AI**

Matrix multiplication with the terminology of neuronal nets

(w – weight, I = Input, O – X-Matrix multiplied sigmoid function)

$$\begin{bmatrix} w_{1,2} & w_{2,1} \\ w_{1,2} & w_{2,2} \end{bmatrix} = \begin{bmatrix} I_1 * w_{1,1} & I_2 * w_{2,1} \\ I_2 * w_{1,2} & I_2 * w_{2,2} \end{bmatrix}$$

The input values for the subsequent layer is in each case:

$$\mathbf{X} = \mathbf{w} * \mathbf{I}$$

Each x must be treated with the sigmoid, respective activation function:

$$Y =$$

Values for subsequent layers change to:

$$\mathbf{O} = \text{sigmoid}(\mathbf{X})$$

- **Algorithm DoE**

The Prediction is calculated with the

- **Intercepts and**
- **Regression Factors:**

(Table shows case for linear regression)

Response	Intercept	F1	F2	Fn
R1	A ₁	A _{F1.1}	A _{F2.1}	A _{Fn.1}
R2	A ₂	A _{F1.2}	X _{F2.2}	A _{Fn.2}
.....
Rn	A _n	A _{Fn}	A _{Fn}	A _{Fn}

Mathematic basics of Development Tools

◆ DoE

- Linear and 2nd, 3rd order regression: *Experimental effort.*

◆ AI

- Feed Forward
 - Depending on data accuracy: Experimental effort
- Machine Learning ?
 - **Limited resources, time constraints a hint for machine learning models**
 - Dependency on property**

Navigating the Complexities of Rubber Development



Ingredients

Properties

Data Structure Rubber Compounds

- Ingredients
 - ◆ Normalized to 100 parts polymer = phr
- Properties
 - ◆ Rheological properties
 - ◆ Physicals
 - ◆ Other
 - ◆ Appearance
 - ◆ Dynamics

Ingredient window

Ingredient	1111.A	111.F	111.D	107.A	107.L	107.H	107.C	107.S
PHR-001	100.00	100.00	100.00					
PHR-002				100.00	100.00	100.00	100.00	100.00
PHR-003								
PHR-004								
PHR-005								
PHR-006								
PHR-007								
PHR-008								
PHR-009								
PHR-010								
PHR-011								
PHR-012								
PHR-013								
PHR-014								
PHR-015								
PHR-016								
PHR-017								
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PHR-099								
PHR-100								

Property window

Property	1111.A	111.F	111.D	107.A	107.L	107.H	107.C	107.S
PHR-001	100.00	100.00	100.00					
PHR-002				100.00	100.00	100.00	100.00	100.00
PHR-003								
PHR-004								
PHR-005								
PHR-006								
PHR-007								
PHR-008								
PHR-009								
PHR-010								
PHR-011								
PHR-012								
PHR-013								
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PHR-100								

- **AI Data Structure**

- ◆ Trial&Error Data Collections
- ◆ OFAT Data Blocks
- ◆ DoE Data blocks
 - Property Data
According Customer / Market
Specification
 - Data Diversity

- **DoE Data Structure**

- ◆ Data Sets
 - Blocks of 9 – 20 Sets
 - Property Data
Project related
 - Data Diversity: blockwise

- **Data Quality**

- ◆ **Faulty Data in**

- ◆ **No useful Solution out**

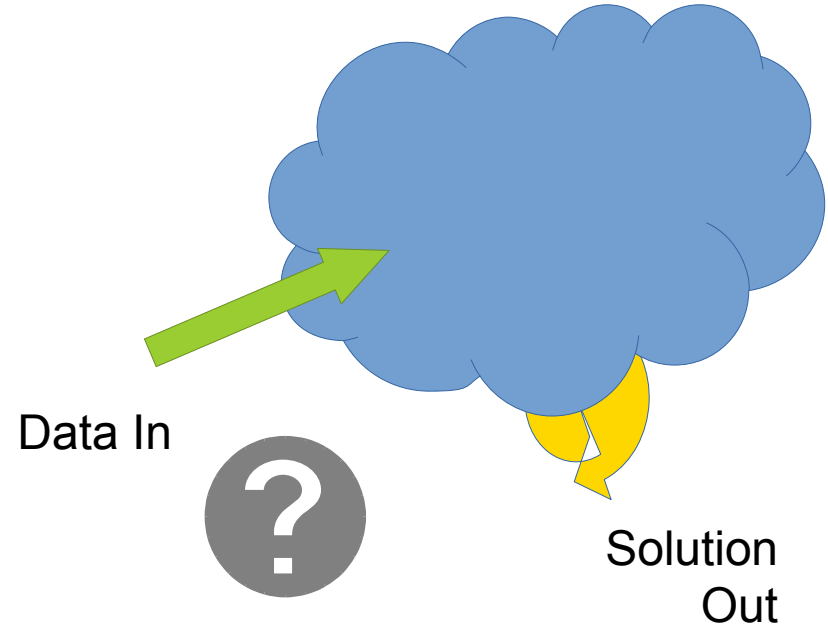
- **Sources of Error**

- ◆ Lack of Standardization

- Variation Mixing

- Variation Testing

- **Data Cloud?**



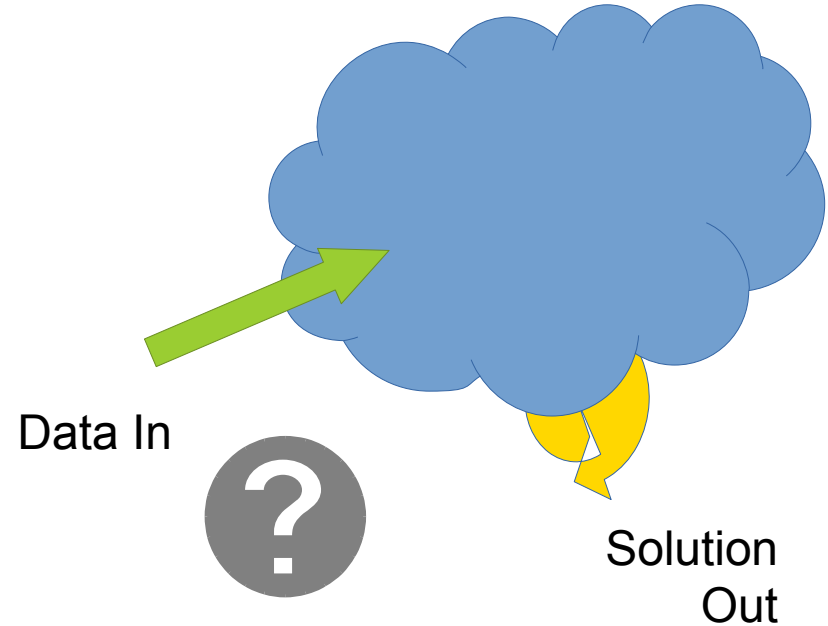
- **Standardization**

- ◆ Eliminate Redundant Ingredients
(Neutralization of Names)

- **Separation**

- ◆ Polymer Compatibility
- ◆ Ingredient Solubility
- ◆ Crosslinking Systems

- **Data Cloud?**



Navigating the Complexities of Rubber Development

- **Decrease of Variation, Increase Precision**

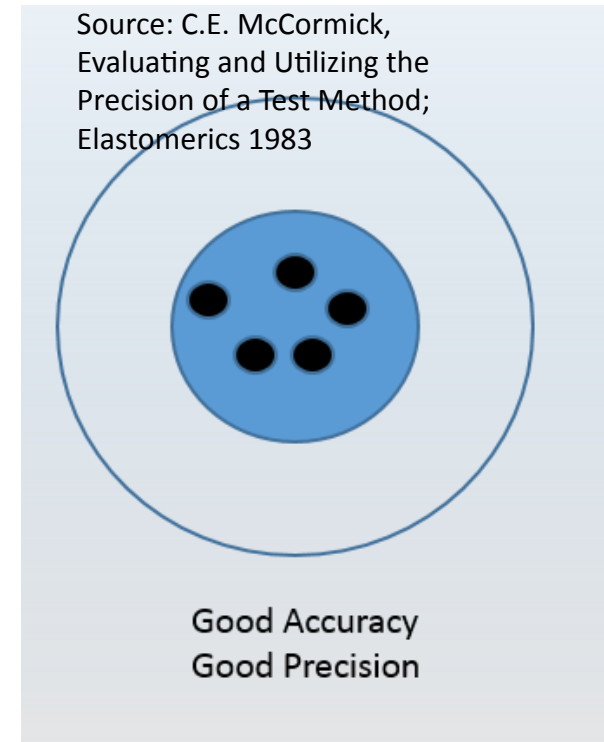
- ◆ **Laboratory Mixing Machine: Variation**

- Raw Material / Process Protocol Influences
- Testing

- ◆ **Test Protocol: 5 instead of 3 samples per Test**

- Evaluation of Measurement Error
(Man – Machine – Material)
- Design of Experiments: Number of Repeats

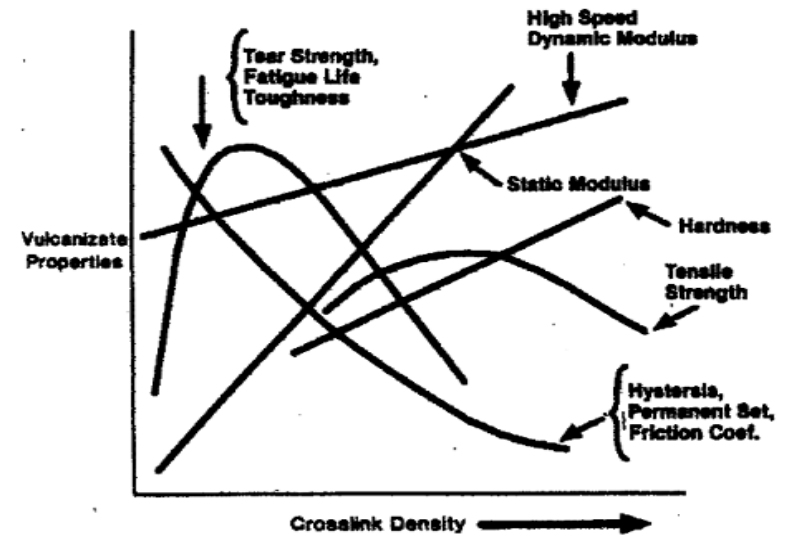
- ◆ **Round Robin Testing with external Laboratories**



Navigating the Complexities of Rubber Development

- **How to check / improve quality of a historic data pool?**
- **AI Prediction of compound inside 95% confidence interval**
 - ◆ Elimination of outliers possible, if ingredient – property relation can be demonstrated
 - ◆ Properties correlated to crosslink density (according Coran)
 - ◆ Correlation analysis of properties
 - ◆ Properties inter-correlated

Correlation between crosslink density and physical properties

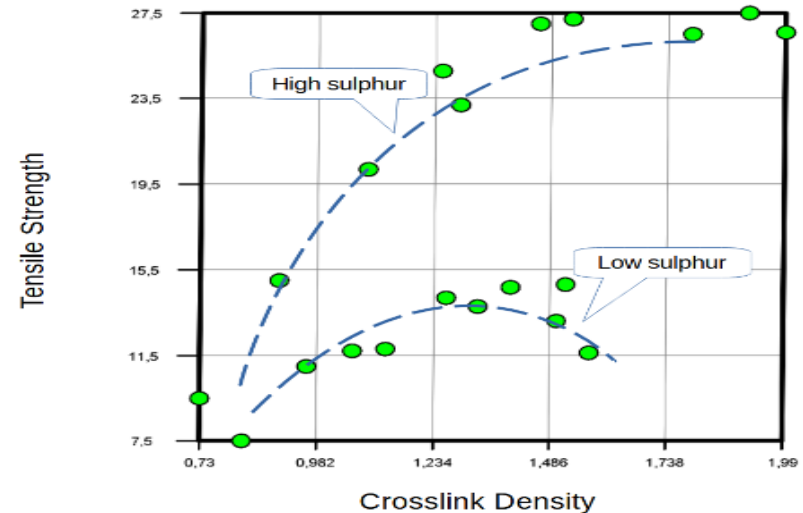


Source: D. Hertz, *Elastomerics* 1984,
 A.Y. Coran, in *Science Technology of Rubber, § Vulcanization*,
 Academic Press 1994
 M.L. Studebaker, *RCT* 39, 1966

• Properties over Crosslink Density

- ◆ Variation of ingredients cause a shift of properties
 - Hardness / Modulus
 - Static- / Dynamic Force
 - Crosslink Density / CLD Concentration
 -

- Variation of Ingredients: Example Sulphur
Source: L. Gonzales, et al., KGK 2005



• Checking Data

◆ Correlation of Related Properties

- Hardness / Modulus
- Static- / Dynamic Force



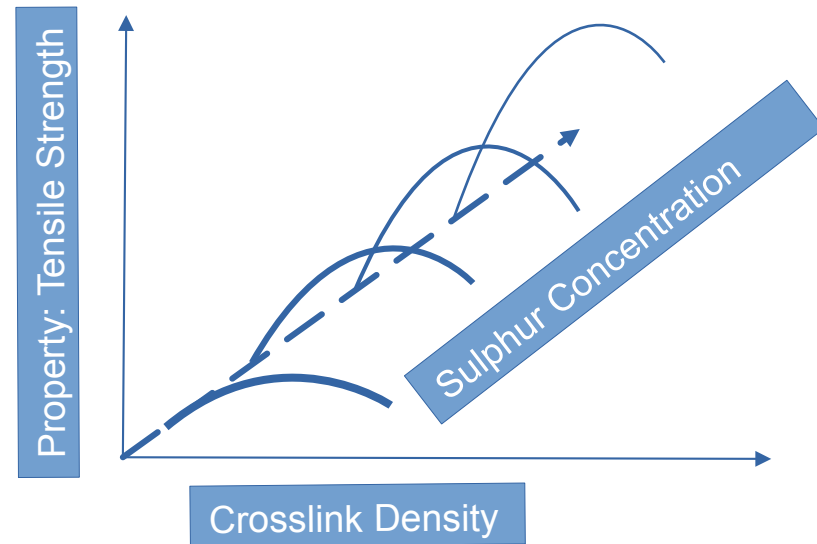
Crosslink Density / Cross Linker Concentration

- ◆ Using a consistent sulfur content when cutting out a curve enables the assessment of accuracy by observing the dispersion or clustering of points along the curve.

• Coran's Diagram

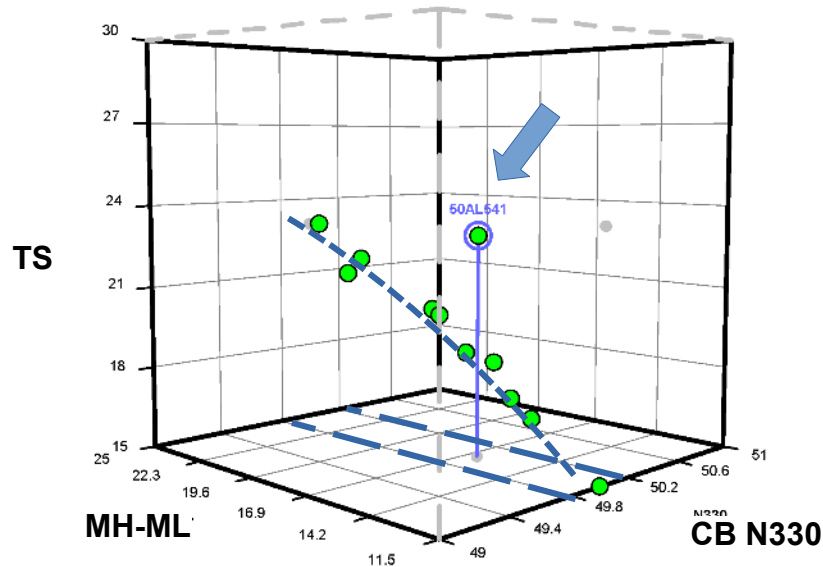
Variation of Ingredients: Example Sulphur

Source: L. Gonzales, et al., KGK 2005

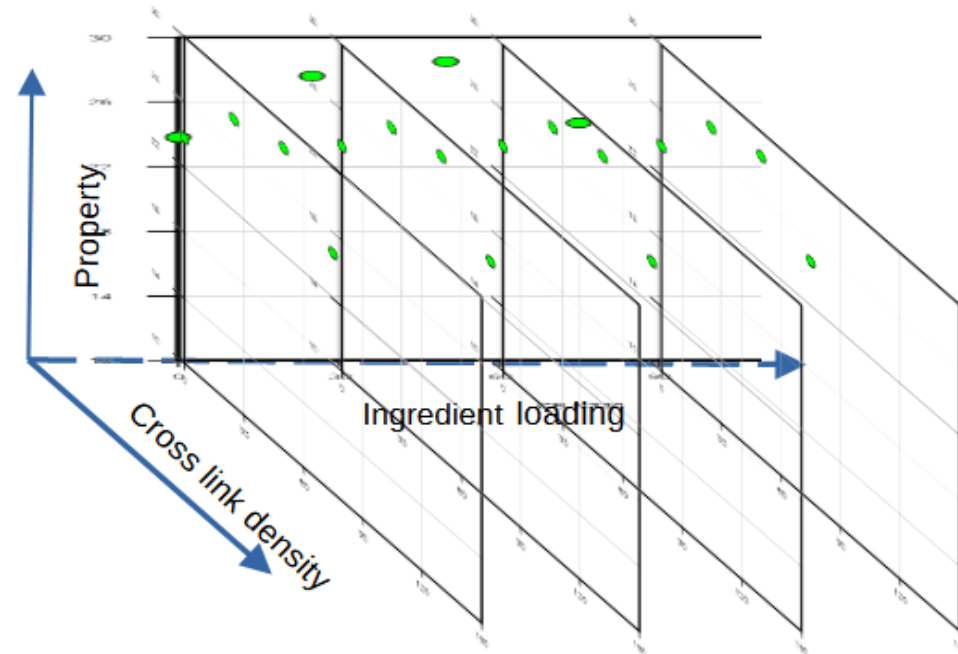


Navigating the Complexities of Rubber Development

- **Property Data Slice**
 - ◆ With outlier



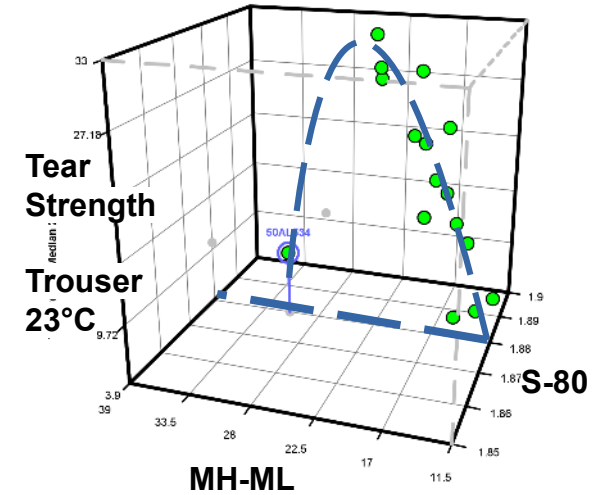
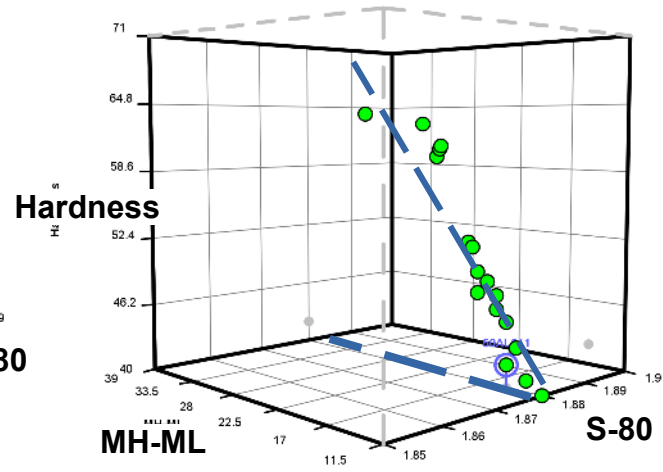
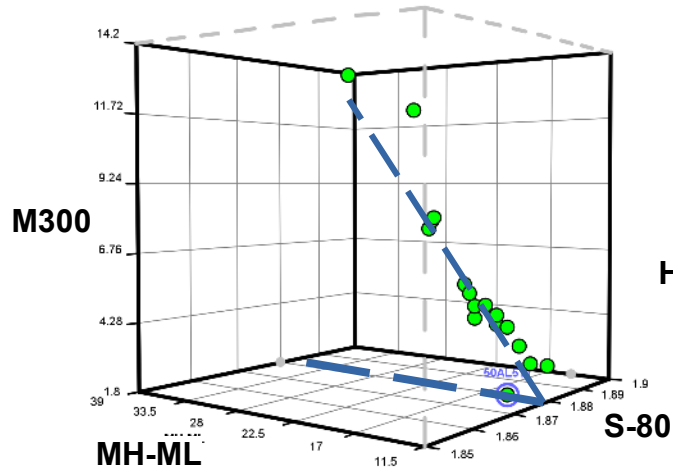
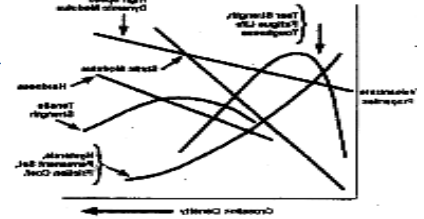
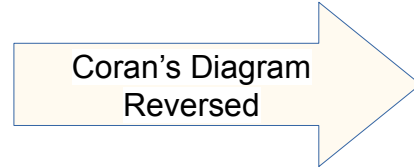
- **Property (TS) over XLD & Loading (CB N330)**
 - ◆ Cutting property-XLD slices



Navigating the Complexities of Rubber Development



- **Property over XLD & Sulfur**
 - ◆ Cutting Property-XLD Slices



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Navigating the Complexities of Rubber Development



Data Management

- ◆ Properties
- ✔ Copy / Paste property names from **master property file**
- ◆ Standardization
- ✔ Standard properties
- ◆ Custom properties.

GrafCompounder version 5.0.1 - ABS0-0013-005 LPDM DLMO NEUTRAL V2.jc

File Edit Diagram Help

Input data

CMPD-55	CMPD-56	CMPD-57	CMPD-58	CMPD-59	CMPD-60	CMPD-61	CMPD-62	CMPD-63	CMPD-64	CMPD-65	CMPD-66
36,20	36,00	47,50	56,90	28,70	47,70	43,90	44,20	97,50	89,70	92,70	49,9
23,00	22,10	35,00	35,70	18,90	31,60	27,10	28,10	83,00	71,20	74,90	30,1
9,51	11,90	9,55	6,43	8,98	9,25	5,89	5,73	19,18	6,51	44,86	61,6
94,05		43,38	15,87	58,98	82,18	13,88	22,28				
0,70	0,70	1,15	1,10	0,49	0,94	0,79	0,78	3,55	3,31	3,08	0,8
0,48	0,48	0,32	0,35	0,41	0,42	0,41	0,42	0,34	0,34	0,42	0,8
0,60	0,61	0,55	0,53	0,59	0,60	0,57	0,57	0,57	0,55	0,59	0,9
1,28	1,30	1,22	1,15	1,27	1,25	1,17	1,18	1,08	1,07	1,20	2,1
7,67	7,56	15,26	11,85	9,89	9,30	9,25	8,63	15,10	16,99	10,91	5,2
8,97	8,85	14,11	10,75	9,4	8,38	8,48	7,85	11,55	13,89	7,02	4,3
0,71	0,75	1,12	1,15	0,51	1,01	0,83	0,83	3,88	3,67	3,38	1,0
1,58	1,54	0,86	0,99	1,48	1,39	1,32	1,38	0,63	0,74	1,11	6,1
3,27	3,26	3,00	2,87	3,56	3,18	2,99	3,03	2,34	2,62	2,82	6,5
7,38	7,38	7,25	7,00	6,23	7,47	7,13	7,35	5,48	6,22	6,87	12,6
10,48	10,48	10,23	10,33	11,61	10,68	10,37	10,62	7,98	9,00	9,90	15,6
8,47	8,54	17,15	12,19	9,78	9,69	9,44	9,03	16,37	16,20	11,89	5,2
7,78	7,78	16,03	11,03	8,26	8,67	8,61	8,2	12,49	14,53	8,5	4,7
51,09	59,09	63,00	66,00	50,90	64,00	64,00	54,00	60,00	70,00	54,00	45
9,80	11,41	10,13	7,37	22,36	450,44	11,49	18,66	14,65	13,19	19,27	11,0
531,88	540,22	187,42	811,92		649,56	557,43	589,54	793,08	911,32	70	
5,4	4,9		8,19	0,00	7,22	7,49	6,16	6,28	4,34	2,4	
28,61	33,14	31,39	24,37	49,26	0,00	34,04	45,32	41,31	36,97	44,48	35,1
1,04	1,04	1,08	1,05	1,04	1,06	1,06	1,06	1,07	1,06	1,04	1,04
5,40	16,60	40,80		0,70	-3,70	4,50	-16,30	-13,20	-16,60	-13	
0,86	6,30	5,40		-10,20	-20,30	-15,00	-43,30	-50,80	-63,30	-26	
2,00	1,00	3,00		3,00	5,00	5,00	9,00	7,00	10,00	4	
257,85	222	223,5	232,5	205	237	237	237	237	237	237	23

Recipe ratios in %

Criteria

Name	Min	Max	From	To	Weight	Tdoff
Mooney Viscosity						
MI-100 C	17,6	100				
Mooney Scorch						
MI-125 Crin	11,5	83,6				
T5-125	4,14	61,64				
T35-125	5,28	94,05				
Rheometer -						
ML190	0,34	3,55				
Tc5-190	0,25	0,89				
Tc50-190	0,28	1,03				
Tc90-190	0,35	2,22				
MH-190	3,11	19,07				
MI ML 190	4,38	17,22				
Rheometer -						
ML165	0,32	3,88				
Tc5-165	0,41	6,14				
Tc50-165	0,53	6,66				
Tc90-165	0,9	12,81				
Tc90-165	1,18	15,87				
MI 165	5,27	20,43				
MH-MI-165	4,24	18,47				
Physicals						
Hardness (ShA)	44	86				
Tensile (Mpa)	3,62	22,35	10	22	100	
Elongation (%)	135,33	911,32				
Modulus (Mpa)	0	8,27				
Tear (N/mm)	0	48,28				
Density	0,98	1,17				
Heat Aging 165						
ΔTensile (% 165)	-16,6	40,8	-2	0	50	
ΔElongation (%)	-56,9	6,3	-2	0	50	
ΔHardness (°)	7	10	0	2		
Total ingredients	163,95	372,83				
Density (calc.)						
Cost (per vol)						
Cost (per mass)						

Output:

30,08975	36,509
20,35175	25,67775
12,12085	8,87755
0,68925	0,89135
0,38265	0,37415
0,51305	0,53675
1,138775	1,162975
7,568075	9,769175
8,979825	8,687925
0,802675	0,85955
0,304825	1,0223
1,63985	2,501325
3,81438	6,09755
5,48375	8,8763
0,01515	10,1737
7,332475	9,31715
52,045	55,05
8,962125	9,98955
455,461225	457,59425
27,15255	28,4538
1,042675	1,02825
0,32975	0,01
0,0056	-2,00225
2	2,6625

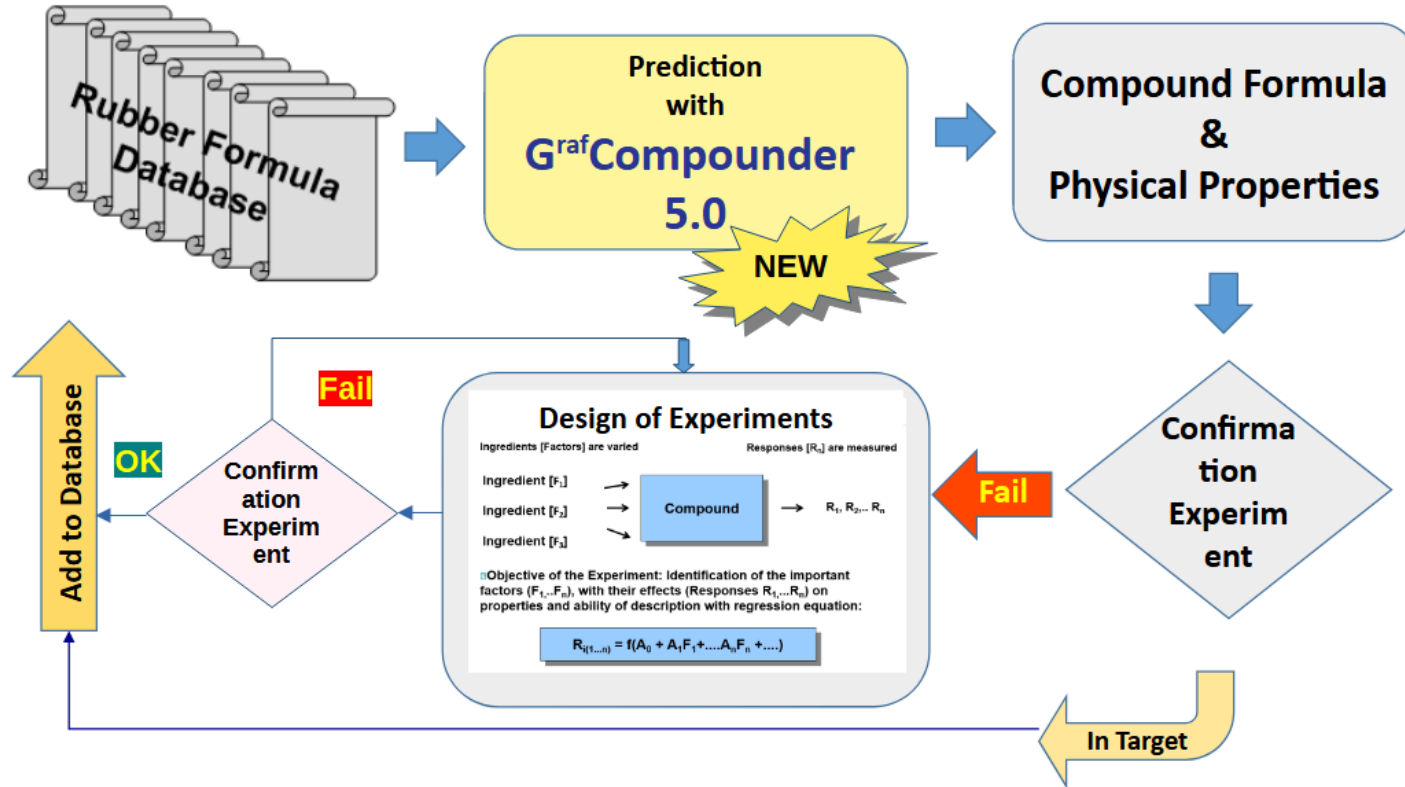
Sum of recipe ratios (should be 100%)
100

Number format: 12345,67

Import input data from clipboard Auto mix (overwrite mixture) Auto mix (new mixture)



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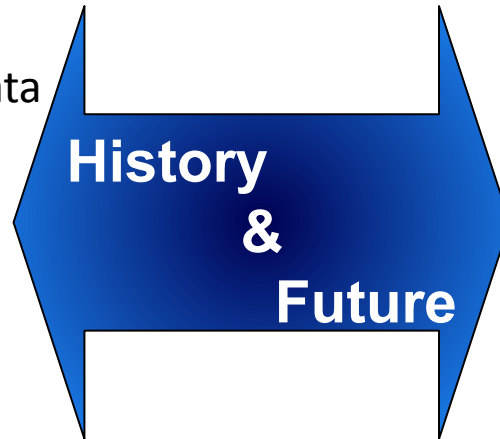


Navigating the Complexities of Rubber Development



Artificial Intelligence: G^{raf}Compounder

- Compound History
- Analyze / Eliminate Foulty Data
- Simulate
- Select Solution for Confirmation Trial
- Confirm



EXPERIMENTAL DESIGN

- Explore Materials
- Evaluate
- Decide
- Select Possible Solution for Confirmation Trial
- Confirm

- **Conclusion**

- ◆ DoE and AI complement each other perfectly.
- ◆ All Data in the cloud is not recommended.
- ◆ Quality of data remains an issue.
- ◆ Database can be improved with suitable tools.
- ◆ Management of data will enhance AI accuracy