

Creating real connections, virtually

Conferences aren't the only way to stay connected throughout the year now. CIC VIRTUAL is a series of events and webinars that you can participate in from the comfort of your home.





# Artificial Intelligence [AI] versus Design of Experiments in Rubber Compounding



Dr. Hans-Joachim Graf

www.hans-joachim-graf.com

www.grafcompounder.com



Creating real connections, virtually

Conferences aren't the only way to stay connected throughout the year now. CIC VIRTUAL is a series of events and webinars that you can participate in from the comfort of your home.





# Tomorrow's machines will "think" for us – they may even tell us what to do!

Walter A. Rapetski, European Rubber Journal, March 1983

#### You can bet that tomorrow's thinking machines will be very similar to today's - old algorithms running on faster computers.

Bart Kosko, in John Brockman, Editor; What to think about machines that think; 2015





#### **Content of this presentation**

- 1. Introduction
- 2. Tools in Compounding
  - Design of Experiment
  - Compounding and Artificial Intelligence
- 3. Advantage and Limits
- 4. Comparison
- 5. Conclusion



Creating real connections, virtually

Conferences aren't the only way to stay connected throughout the year now. CIC VIRTUAL is a series of events and webinars that you can participate in from the comfort of your home,



- Computer Aided Compound Development in Patent Literature
  - Bridgestone Patent 1994 Inventor: Akihiko Abe
  - Bridgestone Patent 2002 Inventor: Yukio Nakajima
  - Colour Matching Patents from BASF, CyanAmid, DuPONT
  - Empirical DoE Patent: Honeywell
  - Recipe Library Search and Comparison CombiChem, GE, Hunt (Private)

0-	iited States Patent <sup>ajima</sup>	US006411945B1 (10) Patent No.: US 6,411,945 B (45) Date of Patent: Jun. 25, 200	i
MATE	HOD AND APPARATUS FOR INING MULTI-COMPONENT RIAL, OPTIMIZATION ANALYZER TORAGE MEDIUM USING LEARNING ISS	JP 9.16651	47 911 A2
(75) Inventor	Yukio Nakajima, Tokyo (10)	Goldberg, David E., "Genetic Algorithm in Sunday	
(73) Assignce	Bridgestone Corporation, Tokyo (JP)	zation and Machine Learning", Addison-Wesley, 1980. Oda, Juhachi, Okada, Hinyasu, "Design method of mate- rials composed of some ingredients by acient	
and the second sec	Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0.4	work"; Optimization Symposium '94; pp. 57–63, 1994. A formulation tool, Alan H. Bohl, Chemosch Mu.	50
(21) Appl. No.:	09/051,416	lation ISA To-	
<ol><li>PCT Filed:</li></ol>	Aug. 8, 1997	Liton, ISA Transactions, 1992, vol. 31, No. 2, pp. 151–157. Design Method of Materials Composed of Some Ingredients by Using Neural Network, Juliachi Oci.	hiko
86) PCT No.:	PCT/JP97/02784	Okada Ostani Network, Juhachi Oda and merchents	ushi-cho yo (JP)
\$ 371 (c)(1), (2), (4) Date:	Apr. 6. 1998	Network, Hideki Toyota, Asalura Back S.	Whalley, Kevin tK, Inn Fields
7) PCT Pub. No.	WO98/06550	Design and Analysis in Mixed Experiments, Manahu Iwasaki, Scientist Co.	3LS (GB)
PCT Pub. Data Foreign A	pplication Priority Data /	cited by examiner rimary Examiner—George B. Davis (4) Autorney, Agent, or Firm—Sughnie Mice, m.c.o.	

- 64 Method for designing pneumatic tires.
- (b) In order to perform tire design and development highly efficiently and provide a tire at low cost, a tire basic model for representing a tire structure and being divided into a plurality of elements, an objective function for representing







### Definition: Statistic Experimental Design

- The (statistical) design of experiments (DOE) is an efficient procedure for planning experiments so that the data obtained can be analyzed to yield valid and objective conclusions. ... An Experimental Design is the laying out of a detailed experimental plan in advance of doing the experiment.
- Statistic is a formal science, whose methods are applied to a wide variety of natural and engineering sciences. The interpretation of the results only make sense if they have been verified for their plausibility in the context of the intrinsic sciences.
- In engineering, it is often necessary to work with small samples, so the treatment of small samples or series of experiments and the presentation of distribution-independent test procedures is of particular importance.





An investigation consists of the following steps applied in a **sequential**, **iterative** manner:

# HYPOTHESIZE

# DESIGN

# ANALYZE

The statistic analysis answers the question whether test results are to be considered as random phenomena or may be treated as characteristic.









- The theory and development of computer systems able to perform tasks normally requiring human intelligence, such as visual perception, speech recognition, decision-making, and translation between languages. (google 15.10.21)
- Deep learning, meanwhile, is a subset of machine learning that enables computers to solve more complex problems.
- Neural networks, also known as artificial neural networks (ANNs) or simulated neural networks (SNNs), are a subset of machine learning and are at the heart of deep learning algorithms. Their name and structure are inspired by the human brain, mimicking the way that biological neurons signal to one another.





# **Software Tools in Compound Development**

# → Database Oriented: Artificial Intelligence [AI]

- Better utilization of historic compound data base
- Faster results minimizes efforts and time in development
- Increases creativity through compound simulation

# Experimentation Oriented: Design of Experiments [DoE]

- Evaluation of New Material without History
- Correlation between effect of factors on responses
- Creates statistically sound knowledge about ingredients and processes





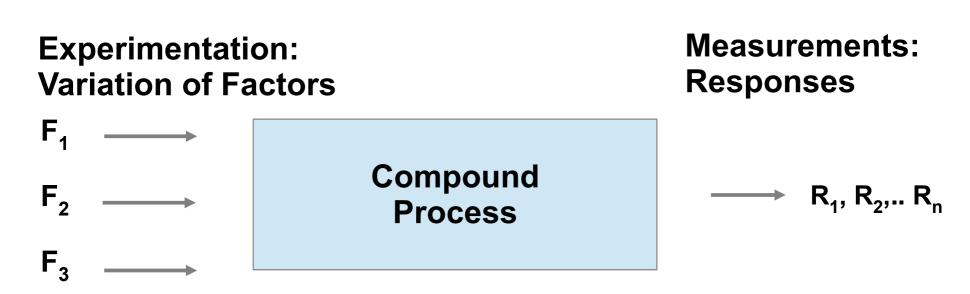
# Software Tools in Compound Development

# Experimentation oriented [DoE]

# →Input: Factor Variation

- Experiments according DoE systematic
- Testing
- Data treatment: ANOVA, Regression, Correlation between factor variation and response
- Output: Prediction of Recipe, Overlay Contour Plots, Correlation Maps,





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.

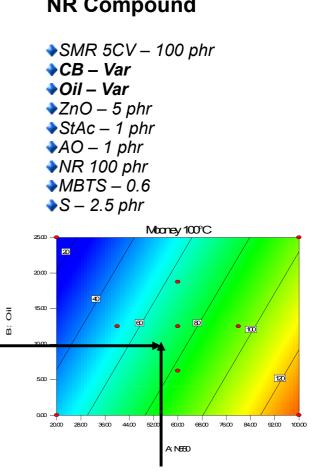
$$R_{i(1...n)} = f(A_0 + A_1F_1 + ....A_nF_n + ....)$$





Properties of Compound is determined by Polymer, CB and Oil content and the ratio of CB and Oil. NR Compound Data of DoE converted into a contour plot

- CB 550: 55 phr
- Oil: 10 phr
- Mooney Viscosity: 71 M-Units
- Hardness: 60 ° ShA
- Tensile: 21 MPa
- Elongation: 460 %
- C-Set: 28%







### **DoE in Processing**

- Example: Operating Window in Injection Molding Statistic Experimental Design (DoE) procedure
  - Factor 1 = T<sub>M</sub>
  - Factor 2 = T<sub>mold</sub>
  - Factor 3 = v<sub>inf</sub>
  - Factor 4 = t<sub>vulc</sub>

**Mass Temperature** 

Mold Temperature

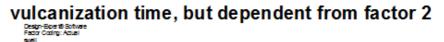
**Injection Speed** 

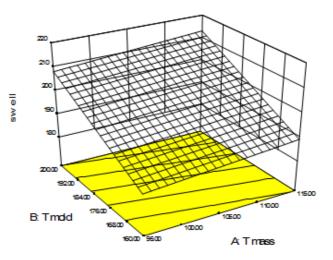
XI - A T ness X2 - B T nobi Actual Factors C: Virj - 20.00

D: tvulc = 75.00

(little influence EPDM, but big with NBR)

Name	Units	Low	High
Tmass	°C	95	115
Tmold	°C	160	200
V inj	mm/sec	15	25
t vulc	sec	30	120



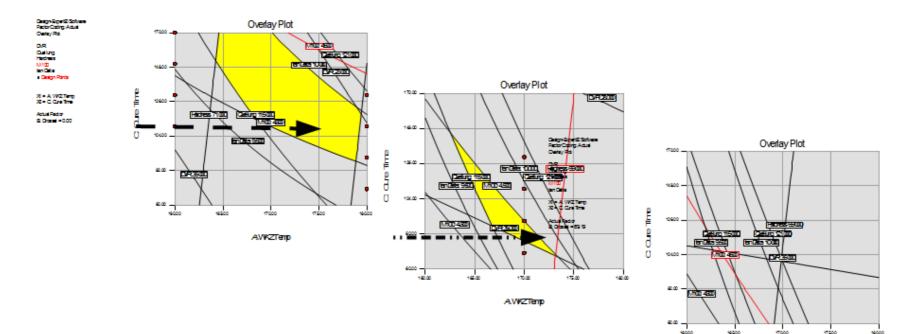






#### **DoE in Processing**

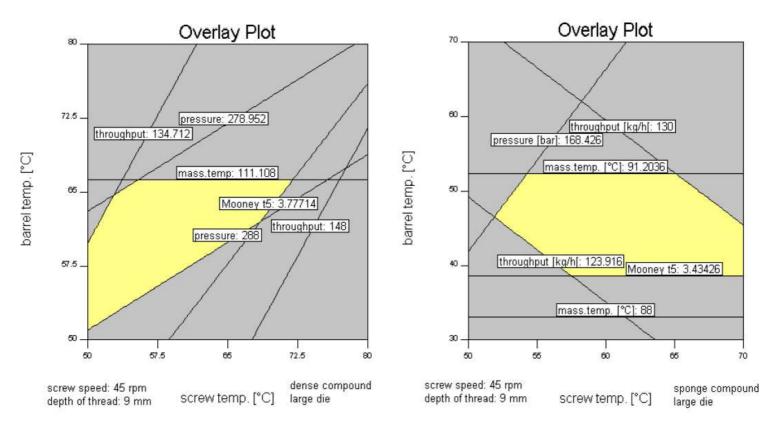
- Example: Operating Window in Injection Molding Resulting operating Window for a NR-72°ShA Compound: Dependency of Mass Temperatures (= Nozzle Valve Positions with LWB-EFE)
  - Nozzle position 0 open / 40 half closed / 70 almost closed







#### **DoE in Processing**



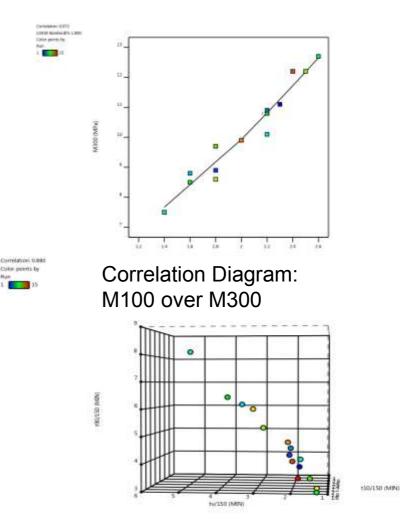
#### Dependency of Operating window in extrusion On screw [x-axis] and barrel [y-axis] temperature.





Statistic experimental design software tools in Design Expert®

- Correlation diagrams allowing a first evaluation of the data:
  - Following inherent logic of rubber principles
- > Histograms
- Scatter plots
- Further tools are
  - Contour plots
  - Desirability plots, whether target is met / or failed
  - > Prediction



Correlation Diagram:  $t_{10}/150C,\,T_{\nu}$  150C, and  $t_{90}/150C$ 





#### The Prediction is calculated with the

- o Intercepts and
- Regression Factors:

(Table shows case for linear regression)

Response	Intercept	F1	F2	Fn
R1	A <sub>1</sub>	A <sub>F1.1</sub>	A <sub>F2.1</sub>	A <sub>Fn.1</sub>
R2	A <sub>2</sub>	A <sub>F1.2</sub>	X <sub>F2.2</sub>	A <sub>Fn.2</sub>
Rn	A <sub>n</sub>	A <sub>F.n</sub>	A <sub>Fn.n</sub>	A <sub>Fn.n</sub>

		-00	•										
langeton (Tara -	n 24/100 /100 (Analyzer )*	- Factors	1.1								Partner Tell		
# 434z-0	t 34/125 180 (Analysed) t 34/125 180 (Analysed) t 72/10 /180 (Analysed) 180 HL70/150 (Analysed)	Factors									Detaut Aanu tu rus	Steel Fuin #	•
	80 HL70150 (Analyzed)	Facto	Factor Name Level Low Level High Level Std. Dev. Coding										
	N V380 HE702/350	A	5-80	2.00	1.0000	3.00	0.0000	Actual			AS-80	-3	_
	00 v180 HL70/156 (Analyzed)	8	M875-80	1.30	0.6000	2:00	0.0000	Actual			INSTS-60		
	180 HL70/150 (Analyzes 180 HL70/150 (Analyzes	c	ZDT/s	4.00	2.00	6.00	0.0000	Actual			enteries.		
	180 HL100/180 LAnivger										C3DD1/M		-
- # 833 E vi	80 HL100/180 Ukralyzek e v380 HL100/180 Ukrah										Factor value:		
Optierizat     Optierizat	al al ana	2 Point P	volition :							- 2			
al Mr antes		Point	Point Prediction										
Point Prediction Tex			, realisting										
alphe Tolerance Pros	025	Two-sid	ed Confide	nnce =	35% Papul	lation = 99	16						
Interval	Two sided		Response		Prodicted Mwan	Predicted Median	Observed	d Std Dev	SE Mean	95%**** Tax			
			13	0/150	2.37361	Z17961		0.231059	0.0623642				
				w150	2,40308			0.621295	0.167691				
			194	0/150	4.5767	4.5767	1	0.794352	0.21548				
				H.	57.5785	57.5785	-	1.87054	0.504868	1			
				£	53.5718				0.525084				
				Tear	57.6537	\$7.6537	5	9.73743		1.12			
				M100	2.06613				0.0503657				
	Details			M300 M300	2.06613				0.0503657 0.252972				

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





#### Algorithm used in Al Programming

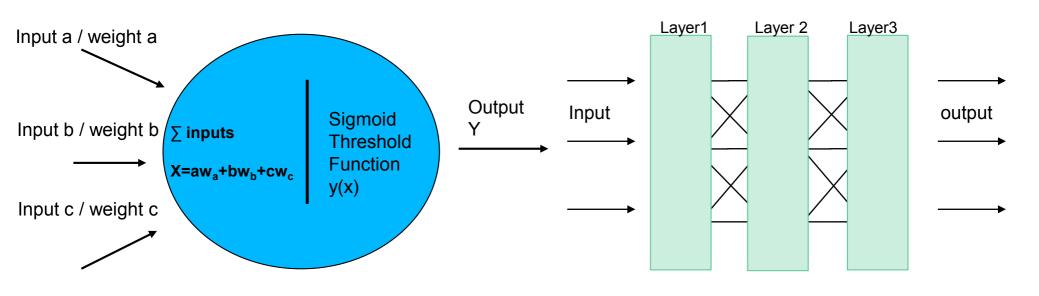
- k-Means-Algorithm
  - Creates cluster, calculates mean (Centroid) reorganization, Iteration
- Genetic Algorithm
  - Proposed Solution, iteration and mutation towards optimum, selection of results with "fitness" function
- Neuronal Net
  - Complex algorithm (Neuron): Calculation of a weighted sum, normalization to emphasize or dampen the summands. "Input – Hidden – Output" layer. Feed Forward Networks / Recurrent Neuronal Networks





Source: T. Rashid, Neuronal Nets, 2017

# 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

# Matrix multiplication with the terminology of neuronal nets (w - weight, I = Input, 0 - X-Matrix multiplied sigmoid function)

$$\begin{bmatrix} I & 1 \\ I & 2 \end{bmatrix} \begin{bmatrix} w & 1,2 & w & 2,1 \\ w & 1,2 & w & 2,2 \end{bmatrix} = \begin{bmatrix} I & 1 & *w & 1,1 & I & 2 & *w & 2,1 \\ I & 2 & *w & 1,2 & I & 2 & *w & 2,2 \end{bmatrix}$$

The input values for the subsequent layer is in each case: X = w \* I

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

Y = 
$$1/(1+e^{-x})$$
  
Values for subsequent layers change to:  
**O** = sigmoid (**X**)





Source: T. Rashid, Neuronal Nets, 2017

#### **Basic Idea of Neuronal Network Calculation:**

- □ The calculation of signals passing through a neural network can be modeled as a **matrix calculation**.
- The links can be specified more precisely independent of the size of the network.
- Programming languages designed for matrix calculations can perform such calculations efficiently and quickly.





Ingredients

roperties

Δ

# **Rubber Recipe Data Stag**

Ingredients
Normalized to 100 parts polymer = phr

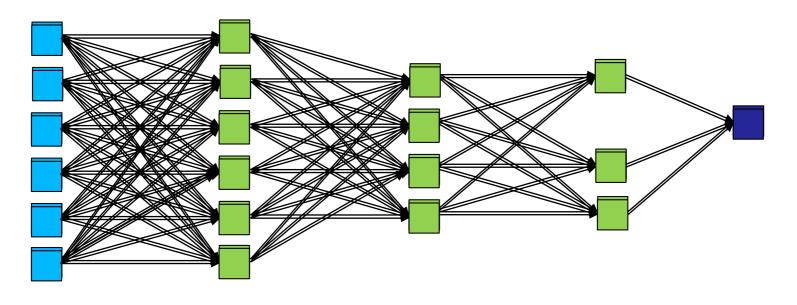
Properties

- Rheological properties
- Physicals
- Other
  - □ Appearance
  - Dynamics





#### A square represents one dataset = data stag consist of n data



Input 1. Layer n. Layer n + 1 Layer Output

Each square represents recipe and property information

- □ Solutions closer to targets survive
  - □ Walking gradient method





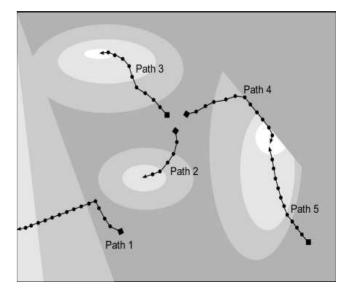
Database (historic, probably incomplete)

# Input: Data + Multi target query

- Search for the best compromise with K-Mean / Multi Objective Evolutionary Algorithm (MOEA)
- Numerical Solution with a Gradient Walking Method. Data treatment simultaneously.
- Approximation / Desirability Function calculates distance from target.

#### > Output:

Recipe with Ingredients and its Properties

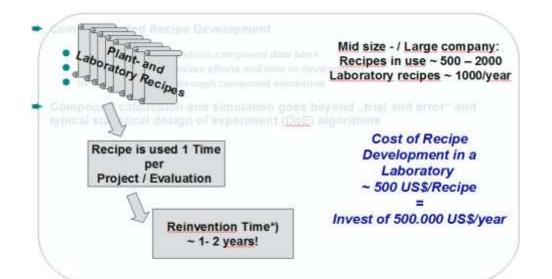






# **Compound Database Characterization**

- Unorganized
- Incomplete
- Inhomogeneous



# **Solution of Database Quality**

Inclusion / Exclusion of recipes from query.

Analytical tools for transparency:

Backtracking to data set used for Result (Opposite to Al Programing)

Combination of data sets with merger function

**Confirmation Experiment of Results** 

Verification prior to integration in database





# Screenshot of GrafCompounder with demo data, targets and a calculated compound

put data											Criteria						Output:		
				6044,515	the state	8041513	HUALERIA	604LSts	HEMEENI !!	KOALSTP HOAL	Name	Min	Max	From	To V	Wei. Trdi	a.		
	NR Testdateen			Recipes:														10111 mar 2	
Code:	Ingredients:	Cost	Density:	50AL511	50AL612	50AL613	D0AL514	50AL515	50AL916	50AL017 50A							Matture?	Monaroli	
4001	SMR 10	280.00	0.92	100.00	100.00	100.00	100.00	100.00	100.00	100.00	Star 10	0	100				2.75	15	
V003	SMR CV60	290.00	0.92								SMR CV60	0	100				97.25	99.5	
004	SMR L	310.00	0.92	1000	10000		42.00		100000	h manna	SMR L	D	100						
3003	N330	115.00	1.80	10.00	30.00	50.00	25.00	45.00	75.00	45 00	A4330	0	75				7.045	0.125	
3004	N336	115.00	1.90								A0336	0	40				-	1000	
3005	N550	115.00	1 80								N550	0	60				19.45	20.2	
1006	N762	115.00	1.80	1000 000	100.000		-		1000 1000	Contraction of the local distance of the loc	N762	0	85						
2010	CaCO3	24.00	2.71	20.00		20.00	20.00	20.00			CaCO3	0	20		- 10-	120	0.55	0.05	
2002	Naphtenic Oil	116.00	0.89	5.00	25.00	45.00	5.00	25.00	45.00	5.00	Maphtenic Oil	0	初	8	10	10	7.9875	2.0825	
1000	Parafinic Oil	120.00	0.90				1000	1			Paratinic Of	0	10				0.005	0.125	
EDD1	ZnO	385,00	5.00	5.00	5.00	5.00	5.00	5.00	5.00	8.00	200	9	10				5		
E021	Zn-2EH	150.00	1.80	2.06	2.00	2.00	2.00	2.00	-2.00	2.00	Zn-2EH						2	2	
-001	Stearic Acid Paraffin Wax		0.92	2.00	2.00	2.00	2.00	2.00	2.00	2.00	Stearic Acid	0	2				1.945	1.97	
3001	The second s	130.00	1,15								Parattio Wax TMQ	0					1.940	0.025	
5001	TMQ	924.00	1.15	2.00	2.00	2.00	2.00	2.00	2.00	2.00	IPPD		- 5				2.9725	2.985	
1001	S-80	158 00	1.80	1.88		1.88	1.88	1.88			5-80	0.31	4.08				2.35885	3 585825	
(804	5-00 DPG-80	420.00	1.28	1.00	0.00	1.00	0.00	1.00	1.00	1.00	DPG-80	0	100000				2.33000	0.003125	
4802	DTDM-80	360.00	1.28								DTDM-80	0						0.015625	
K001	TMTD-80	396.00	1.11								TMTD-80	0						0.010045	
K005	CBS-80	708.00	1.28	0.63	0.63	0.63	0.63	0.63	0.63	0.63	C85-60	ő					1.452825	1.183575	
1000	000-00	100.00	1.20	0.00	0.00	0.00	0.05	0.00	0.65	0.00	000-00		2.00	-	-		1.402.020	1.103010	
	Properties:																		
PR001	MooneyML(1+4) 100°C			32.00	36.00	31.00	34.00	30.00	42.00	60.00	MooneyML(1+	4 27	80	-	_		30.7739	29.402	
PR002	Mooney t5 / 120°C			28 00		32.00	28.00	32.00			Mooney 15/	T	39				32 0873	32.907	
PR003	Density			1.06		1.16	1.12	1.16			Density	1.02					1.0598	1.040825	
PR004	Hardness			42.00		40.00	48.00	48.00			Hardness	40		47	50	50 5	0 50 01	62.711	
PR007	M300			1 80		3.00	4 40	4.60	5 30		M300	1.8		7			5.08455	6,2179	
PR008	TS-			25.00		15.00	25.00	20.00	15.30		TS	15		26			25 69705	26.107	
PR000	EB			785.00		690 00	715 00	705.00	615.00		EB	445		-	800		634 6817	598.35	
PR020	C-Set -26°C /24h [%]			22.00		30.00	17.00	19.00	35.00		C-Set-20"C	10			10	10	22 4705	15.389	
PR021	C-Set 0°C /24h (%)			10.00	14.00	14.00	B.00	12.00	110.00		C-Set 0°C	4	1000		0		8.525	6.4265	
			5				2.00				4			-	-		- 14T		7.
Total				146.51	186.51	226.51	161.51	201.51	251.51	181.51	Total	122.6	251.5	8			150 786875	139.35065	
Densily				1.097		1.128	1.738	1.148	1.172		Density (calc.)						1.055	1.045	
Cost (per				262 484		220.591	259.16	235.661	219.811		Cost (per vog						277.212	287.094	
Cost (per				239.274		195.559	227.733	205.454			Cost (per		301.9	1		_	262.76	274.731	
and the same	a la Ru										Concert Male							CALL AND	1000 V
ecipe ratio	5.01%																	ratios (should be	10(0%)
						0.25											100		



Creating real connections, virtually

Conferences aren't the only way to stay connected throughout the year now. CIC VIRTUAL is a series of events and webinars that you can participate in from the comfort of your home,



#### 田\_(in)× **GrafCompounder Screen** File Edit Diagram Help input dates Output. Critera Nane West: Dist. CROWN **ICHOP** Distant. Max From To NR SBR Merger Buna Hole Recipes: Role Code Ingredients: Cost Density CMDPT CMETERS CAUTE Habiel Data field 1004 SMR 10 280.00 0.52 SMR 10 NR Sheets II 0.92 NR Sheets N 100 A002 240.00 18.5 0.92 2008 SMR CVER 201 290.00 SMR CVAR $\circ$ Code 0.82 ACO SWRL 310.00 SMRL 300 601 SBR 1500 170.00 0.94 5897 1500 100 81.5 0.94 SBR 1609 (5C#40CR) 170.00 A012 SBR 1609 (SCI440CB) 72.5 Ingredient name 176.00 1.12 A013 SBR 1618 (50450C8) 589 1610 (SO450CB) 355 Ο A014 SBR 1620 (-Oilt0CB) 178.00 1.13 BR 1020 FOMMOCED 721 0.94 A015 SBR 1711 (17.500-CB) 178.00 10 17 11 200 ○ Cost information Ad 16 SBR 1707 (37 5CM-CB) 176 80 0.94 2443300 +37.5 A017 58R 1779 (37.508-CB) 176.00 SBR 1779 30 SBR 1808 (47,50876CB A018 170.00 1.14 SBR NOT 223.5 SBR 1043 (1504100CB) A019 176.00 1.20 588 1843 24 o **Density** A021 Buna CB 10 200.00 0.92 Hata CE 10 0.91 TOR 520.00 TOR 25 105.00 1.25 10.5 Alter **Recycled** Tread Recycled Tread 20 Criteria field BOOK 1/ 220 115.00 1.80 N 220 -7,0375 45.35 N330 115 DO 1.80 8 3625 B003 70.00 70.00 N338 47,1625 11330 115 00 1.80 8001 N736 Min/Max Value 115.00 1.80 1.575 **NO**P 11550 NEED $\cap$ BOOB N 762 115.00 1.80 N 762 B901 Ground Rubber 55.00 1.25 10.00 Ground Rubbe 2.00 145 00 000 Soca VN3 Silica WU3 **Query column:** Ο CONT CaCO3 24.00 1.57 CeCO3 172.00 2.70 8.4 C021 Clay 36 Chiv Sugar la 125.00 2.00 C022 Kilder M ○ From 0.00 000 Parafine OI 120.00 Paraffinic OI DOW Nephtenic Oil 116.00 0.91 Naphdenic CN Acomatic OI 128.00 0.96 D003 18.00 18.00 Assentic O/ 12 6.21375 7,7225 o To ED0 ZnO 385.00 5.60 5.00 6.00 20 10 4,7375 EDIT TiO2 260.00 2.74 1102 4 4.87 • Weight 172 7595 203 853625 Total ingediests 201.9 217.9 1157 38 Total agredients Density (cetz.) 1.168 Density (calc.) 1.028 1.606 1158 1.189 Cost (per vol) 202 012 196 18 190 125 286 317 037 194.962 205 545 Cost (per vol) TrdOff 172-958 167.389 162 92 108 293 201 168-361 172.872 Cost (per mass Cost (per mas $\cap$ Recipe ratios in % Sum of recipe ratios (should be 100%) 100 Number format: 12,346.67 Import input data from clipboard Auto mix (overwrite maduze) Auto mix (rew matuze) ≻Output field

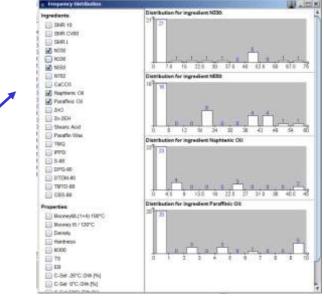




#### **Database Diagnose Tool**

- Frequency distribution of
  - ≻Ingredient≻Property
  - can be chosen
- Scale is automatically selected according Minimum and Maximum value in the database









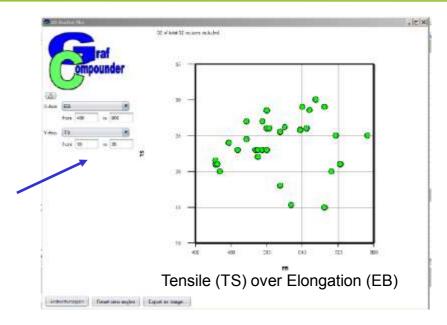
#### **Database Diagnose Tool**

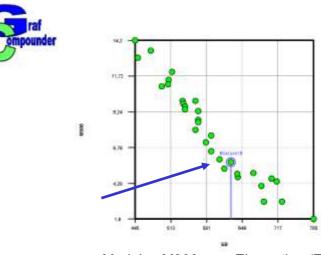
Correlation Diagram

- Example:
   Tensile (TS) over Elongation at
   Break (EB)
  - ≻Scale for axis

Selected

- automatically / manually
- Highlight recipe for evaluation or exclusion





Modulus M300 over Elongation (EB)



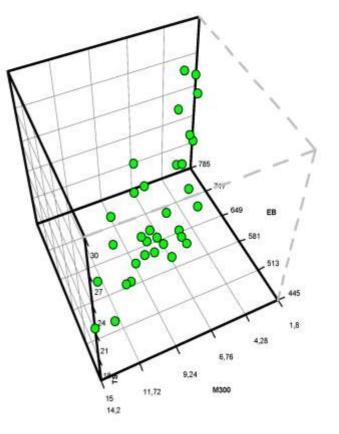


# **Database Diagnose Tool**

#### ≻3D Correlation Diagram

#### ≻Example:

- X-axis: Elongation at Break (EB)
- Y- axis: Modulus 300 (M300)
- Z-axis: Tensile at break (TS)
- Scale for axis:
   Selected automatically / manually







Mixture1

Mixture2

# How to do a query:

- 1. Ingredients Exclude with target = 0
- 2. Properties
  - ✓ Target from Min value to Max value
  - Put a weight on property target,

if more important than others

 In case of conflicting target: Give a preference with Trdoff: Back off a bit from the target in favour of others

FIC	,,,,,	10	weight	muon			02
2						0.025	
4					2.9725	2.985	
4.06					2.35885	3.585825	
0.25						0.003125	
1.25						0.015625	
1.5				1			
2.63					1.452825	1.183575	
80					30.7739	29.402	
39					32.0873	32.907	
1.21					1.0598	1.040925	
71	47	50	50	50	50.01	52.711	
14.2	7				5.08455	6.2179	N
30	25				25.69705	26.107	
785		600			634.6817	598.35	
83		10	10		22.4705	15.369	
16		6			8.525	6.4265	1
18		10			7.9618	4.838	
61		15	10		26.3231	22.0725	
	_						
51.51					150.766675	139.35065	
1.214					1.055	1.045	
26.37					277.212	287.094	
01.915					262.76	274.731	

Weight Trdoff

From

To



of your home.



#### Recipe for confirmation experiment

- Append selected recipe in "Input data" field
  - It is highlighted as "gcunconfirmed"

# Interaction with user

- Eliminate nonsense values and consolidate
- Round values to two decimal places

# Confirmation

 Experiment: Change property values to confirmed values

					Criteria:			10.0				Output	
04	Minturet	Midurel	Mixture2	Minture4	Name	Min	Max	From	To	Wei	Trdoff		
				gc-unconfirm									
104	Modure1	Mixture-	Midure2	Mixture4								Mixture1	Mottare4
					SMR 10	0	100	8				2.75	1.5
00.00	100 00	100.00	100.00	100.00	SMR CV60	0	100					97.25	98.5
					SMRL	0	100						
45.00		7.00			N330	0	75					7.045	0.125
					N336	0	40	n					
	20.00	20.00	20.00	21.00	N550	0	60					19.45	20.2
					N762	0	85						
					CaCO3	0	20					0.55	0.05
4,00	2.00	8.00	2.00	2.00	Naphteric Oil	0			10	10		7.9875	2.0825
					Paraffinic Oil	0						0.005	0.125
5.00	5.00	5.00	5.00	6.00	ZnO	5	10	1				5	1
					Zn-2EH	0							
2.00	2.00	2.00	2.00	2.00	Stearic Acid	0						2	1
2.00	2.00	2.00	2.00	2.00	Parallin Wax	0	2					1.945	1.97
					TMQ	Ø	2	č.					0.025
3.00	3.00	3.00	3.00	3.00	IPPD	2	4	8				2.9725	2.985
3.13	3.60	2.25	1.90	3.60	S-80	0.31	4.06	ę				2.35885	3.585825
					DPG-W	0	0.25	B					0.003125
					DTDM-80	0	1.25						0.015625
					TMTD-80	0	1.5						
1.00	1.20	1.50	1.75	1.20	CBS-80	0	2.63	-	-	÷	-	1.452825	1.183575
57.00	29 20	30.90	30.22	29.40	MooneyML(1+	27	80	_				30,7739	29.40
22.00	33.20	32.00		32.91	Mooney 15/	8	39					32.0873	12.907
1.10	1.04	1.08	1.04	1.04	Density	1.02	1.21	5				1.0598	1.040925
71.00	52.60	50.03		52.71	Hardness	40	71		50	50	50	50.01	52.71
10.80	6.14	5.06		6.22	M300	1.8	14.2			0	100	5.08455	6.2179
27.00	26.20	25.81	28.56	26.11	TS	15	30	S			1	25.69705	26.10
50.00	600.00	635.32	655.95	598.35	EB	445	785		600	1		634.6817	598.35
31.00	15.40	23.01	24.67	15.37	C-Set-26*C	10	83	e	10			22.4705	15.360
12.00	6.40	8.61		6.43	C-Set 0*C	4			é			8.525	6.4265
20100	C	and a second		7+	-	-	-	-	-	-	2.0	14	
65.13	138.8	150 75	137.65	139.0	Total	192.6	251.5	81		1		150.766675	139.35065





#### **Compound Simulation with GrafCompounder**

- Creation of a formula with multiple criteria query including
  - Ingredients
  - Properties
- Traceability back to each formula used in calculation
  - Analysis of outliers and their correction or elimination in the database is possible.
  - Integration of results from statistical experimental designs to improve database
  - Merger of databases of different origin
    - either with copy / import from table calculation program
    - Merger of data.gc files

#### **Result of simulation MUST be confirmed by an experiment.**

 Probability of a match between calculation and confirmation experiment result is about 90-5% according first experience





#### Experiments to evaluate accuracy of the method

- To prove calculation of recipe using a database or even historic data from literature three experiments were chosen:
  - 1. Recalculation of Filler / Oil DoE (EPDM Extrusion Compound)
  - EPDM ENB / Accelerator DoE recalculation (published by DuPont 1998) for optimum ENB content and None n-nitrosamine accelerator system
  - 3. Prediction and Confirmation Experiment Existing development project





# Calculation method confirmation

- > Prove with
  - 1. EPDM Filler / Oil DoE most of basic physicals are linear
  - 2. Filler / Oil DoE
  - **3. Accelerator DoE**

#### **DoE with 4 Factors**

Polymer used: EPDM (Vistalon 8600)

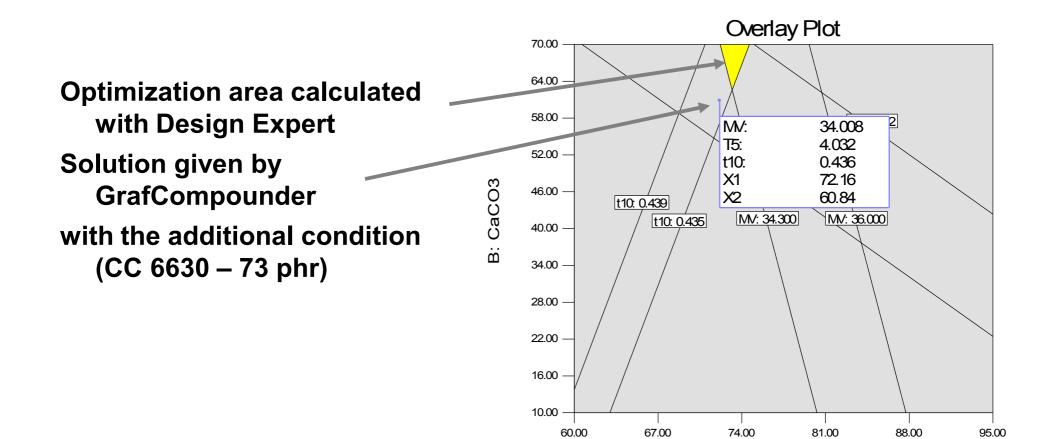
Factor Name	Units	Min	Max
_	A C6630	phr 60.00	95.00
	B CaCO3	phr 10.00	70.00
	C Clay	phr 10.00	50.00
	D Oil	phr 70.00	95.00

A fractional factorial DoE with 11 compounds only!

1.







A: C6630





**DoE published by DuPont Dow in 1998** 

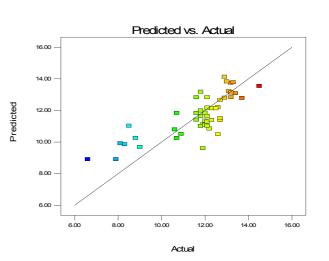
- Factors: ENB, DTDC, S, MBT, TiTBD, ZdiBC, DTP
- DoE with 41 Experiments

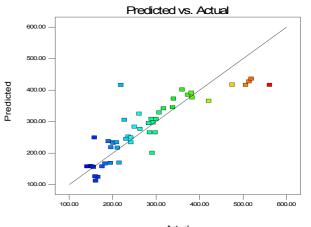
Tensile at break is significant with linear model

- Sulfur has larger influence followed by DTDC and TiBTD, but negative
- Elongation is significant with quadratic model, but linear model is a more than sufficient fit
  - Sulfur has the largest influence followed by DTDC

Hardness is sufficient significant with linear model as well

 Main influence Sulfur, DTDC





Actual

2.



of your home.





Select boundaries

ZF-MPa	: 11.5-12.0	
ZD-%	: 325-335	<b>AREFERENCE</b>
H-°ShA	: 65-67	******
	Export optimizati	

The Design Expert optimization graph shows the location of the result as a yellow area, but

GrafCompounder result is tagged with a flag.

1 50	Overlay Plot	
1.50 —		
1.20 — Jing	ZF: 11.536 ZD: 306.017 Hardness: 68.146 X1 6.50 X2 0.98 Zr. 11.490	
— 000 C: - 000 C: - 000 C:	Harchess: 67.489 ZD: 325.062	
0.60 —	ZD: 335.107 Hardness: 66.013	
0.30 —		
0.00		

Ingredients	GrafCompounder	Design Expert®
ENB	6.5	5.45
C:Sulfur	0.93	0.88
B:DTDC	0.98	0.98
D:MBT	1	1
E:TIBTD	1.51	1.51
F:ZDiBC	1.33	1.33
G:DTP	1.45	1.44
ZF	11.5	11.5
ZD	325	330
Hardness	67	67.5





3.

# **Prediction and Repeat in an Experiment in Laboratory**

	EPM Compound No1								
	Predicted	Test results	Deviation						
Hardness	69.99	70.00	0.01%						
Tensile at Break	19.56	18.75	2.16%						
Elongation at Break	338.61	339.00	0.06%						
M 100	3.87	4.75	9.26%						
C-Set 23°C / 22h	10.28	10.91	2.89%						

#### EPM Compound No2

Predicted	Test results	Deviation
60.28	61.00	0.59%
15.74	18.08	6.47%
513.48	476.00	3.94%
2.04	3.05	16.56%
10.00	9.97	0.15%

#### Data in good agreement except Modulus 100



of your home



- → Statistic Experimental Design (DoE)
  - Organized / Limited size Data set
    - DoE's with large amount of Factors difficult to handle and analyze
  - Variation of few factors according DoE scheme
    - Evaluation of a portion of compound
  - Measurement error statistically evaluated (ANOVA)
    - Experimentation controlled
      - Lack of fit test noise to signal ratio
      - Significance (F-value, p-value, Fit statistics)
  - Optimization, numerical and graphical / prediction Tool available in the software

#### Advantages of DoE

- Evaluation of New Raw Materials
  - Correction of Compound due to Raw Material Changes
- Process Optimization
  - Operating Window / Process in control according SPC
  - Process Design with new machines / raw materials





# Compounder

- Unorganized / Unlimited Database
  - Measurement error unknown
  - Incongruent values (due to different test methods)
  - Raw materials in Laboratory (Storage time?)
  - Missing values
- Multiple Factor Query
- Compound Simulation according Multi Targets

Calculation in multiple small steps excludes none linear effects.
 Accurate Property Data yield Output in 95% confidence interval.





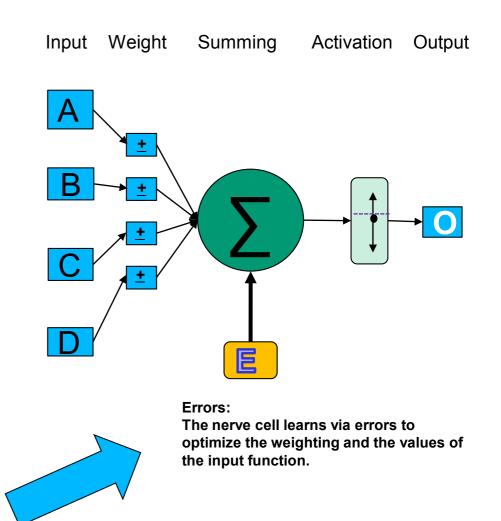
Source: S. Velasco in Spektrum 11/21

#### Challenges and Hints to machine learning with "Back Propagation" in rubber compounding:

- Time consuming testing specifically long term aging / dynamic
- Compound preparation to generate data with high effort.

(Tests with laboratory generated data has failed in the past)

- Database standardization
  - Basic data collective
  - Specific data collective
- Identification of data errors
  - Data transfer errors
  - Compound preparation / measurement errors







# **Conclusion:**

- <u>Compounder Program</u>
  - Compound Cost estimation
  - Starting Formula for Compound Development
  - Simulation of Recipe for Specification Adjustments
  - Historic Data usage and improved Data storage
- <u>Statistic Experimental Design</u>
  - Evaluation of New Materials
  - Cost optimization in Compounding
  - Design / Improvement of New Processes
  - Process Window to Control Quality according SPC
- Combination of Database / Compound Simulation with Statistic Experimental Design Experimentation Procedure

# Both methods have their justification.





# GRAFCOMPOUNDER

## **EXPERIMENTAL DESIGN**

- History
- > Analyze
- Simulate
- Select
- Confirm

- - \_\_\_\_

> Explore

- Evaluate
- Decide
- Confirm
- Conclude





#### Release of the "GrafCompounder" Version 4.0 was June 2021

Upgrades for Owners of Version 3.211 provided for a special price Free upgrade if purchased 2<sup>nd</sup> half 2020 / 1<sup>st</sup> half 2021

#### Thank you for joining this presentation.

→ Questions, Remarks, Discussion ?

More information under: www.grafcompounder.com