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

**H-JG Consulting**

Dr. Hans-Joachim Graf

[www.hans-joachim-graf.com](http://www.hans-joachim-graf.com)

[www.grafcompounder.com](http://www.grafcompounder.com)



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**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*



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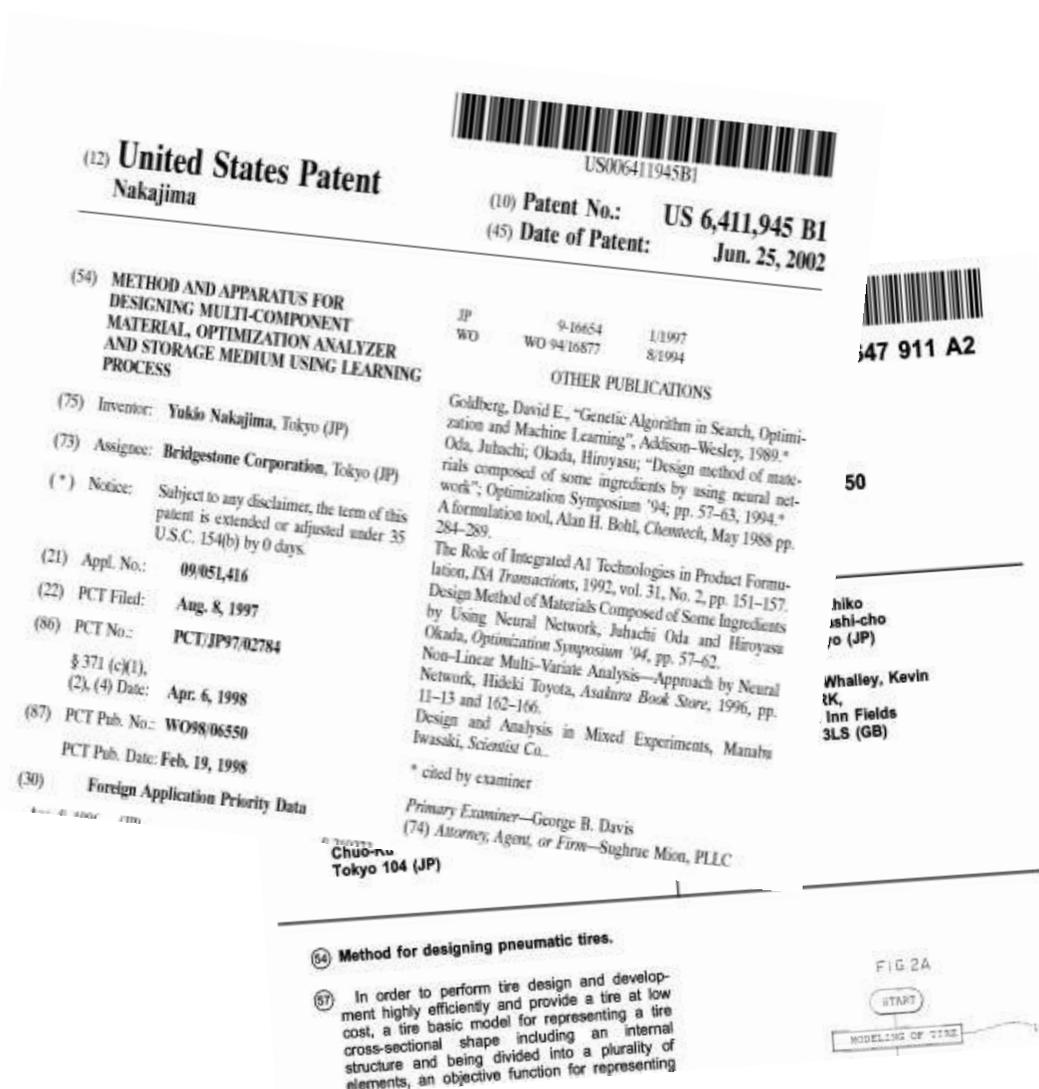


## Content of this presentation

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

## → 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)



## → 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.

## → Definition: Artificial Intelligence

- 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

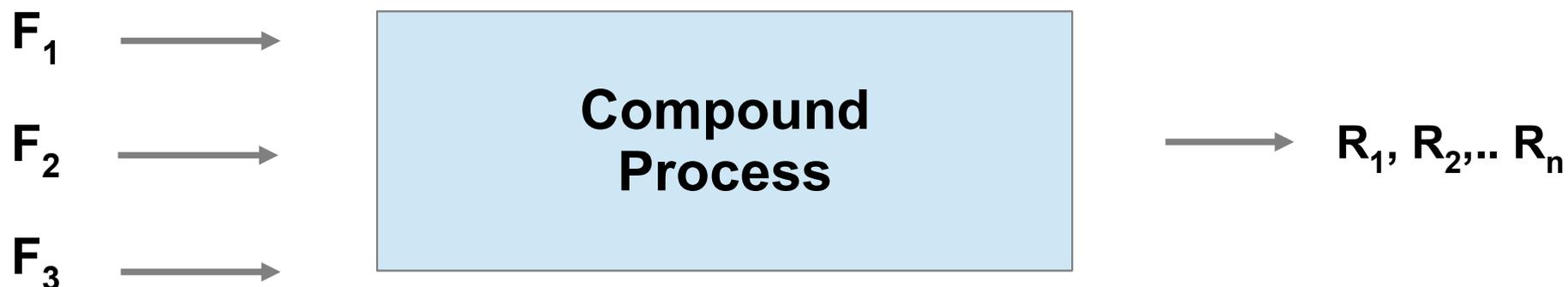
### → Experimentation oriented [DoE]

### → 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,

## Experimentation: Variation of Factors



## Measurements: Responses

Objective of the Experiment is the identification of the factors ( $F_1, \dots, F_n$ ) type of influence on the responses ( $R_1, \dots, R_n$ ) and description with mathematical equations for further processing. ANOVA is used for statistical evaluation.

$$R_{i(1\dots n)} = f(A_0 + A_1 F_1 + \dots + A_n F_n + \dots)$$

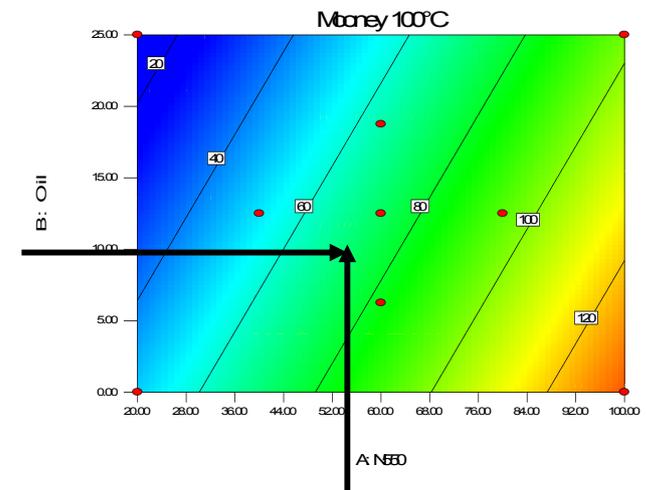
# Properties of Compound is determined by Polymer, CB and Oil content and the ratio of CB and Oil.

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%

NR Compound

- ◆ SMR 5CV – 100 phr
- ◆ CB – Var
- ◆ Oil – Var
- ◆ ZnO – 5 phr
- ◆ StAc – 1 phr
- ◆ AO – 1 phr
- ◆ NR 100 phr
- ◆ MBTS – 0.6
- ◆ S – 2.5 phr

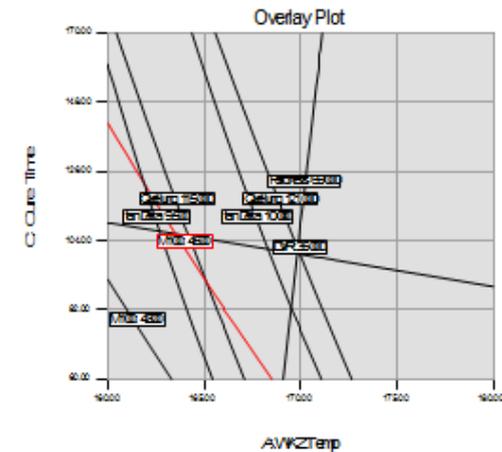
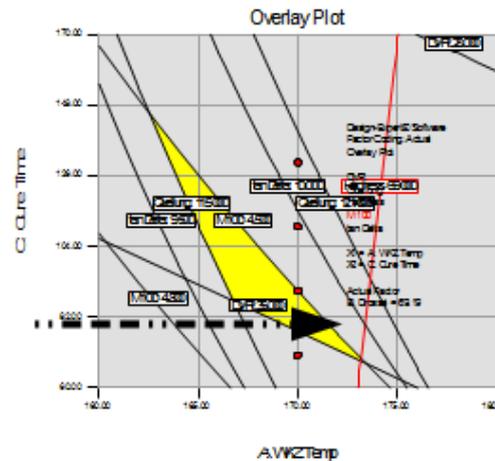
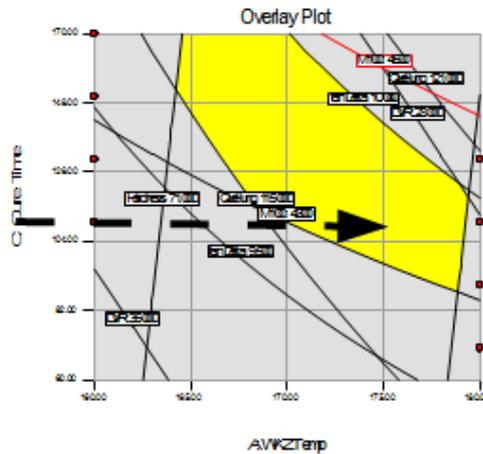




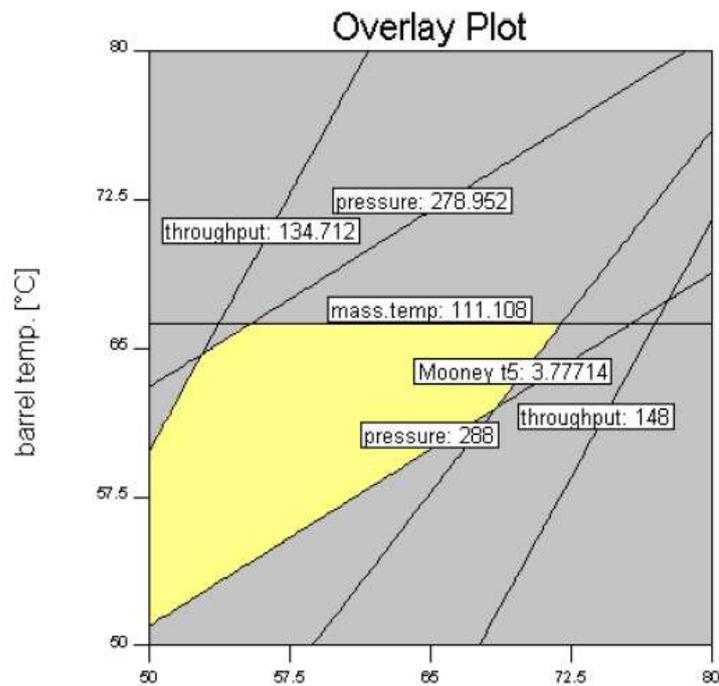
## DoE in Processing

- ➔ **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

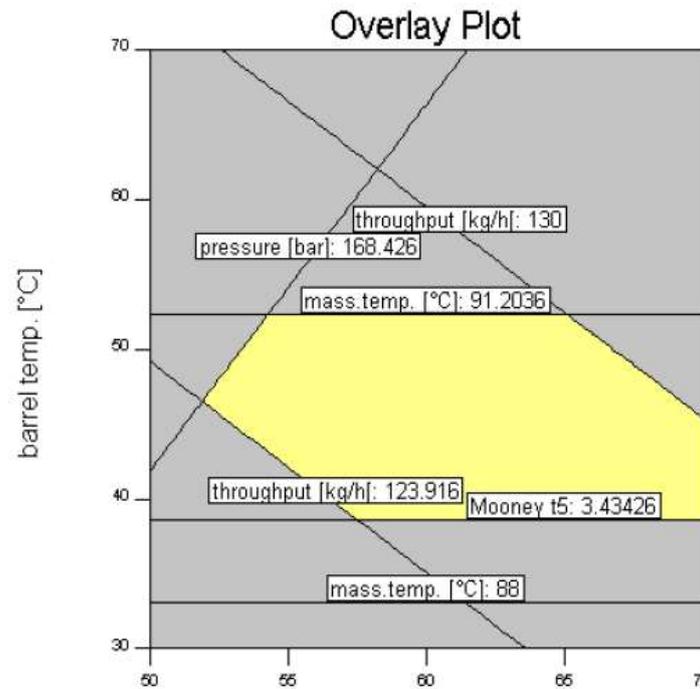
Design Space Software  
Ratio Control Actual  
Cure Time  
DUR  
Curing  
Thickness  
MUD  
In Cells  
Design Points  
X = A: WZTemp  
Y = C: Cure Time  
Actual Ratio  
B: Dose = 0.00



# DoE in Processing



screw speed: 45 rpm  
depth of thread: 9 mm  
screw temp. [°C]  
dense compound  
large die



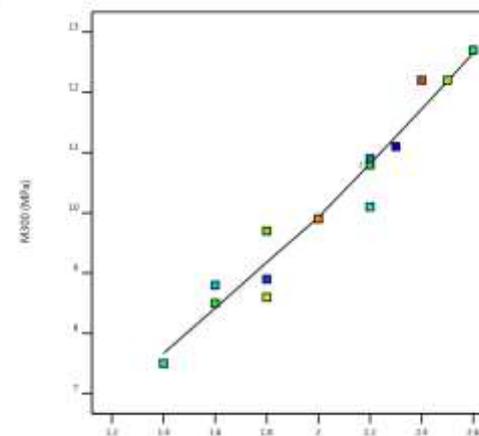
screw speed: 45 rpm  
depth of thread: 9 mm  
screw temp. [°C]  
sponge compound  
large die

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

## Statistic experimental design software tools in Design Expert®

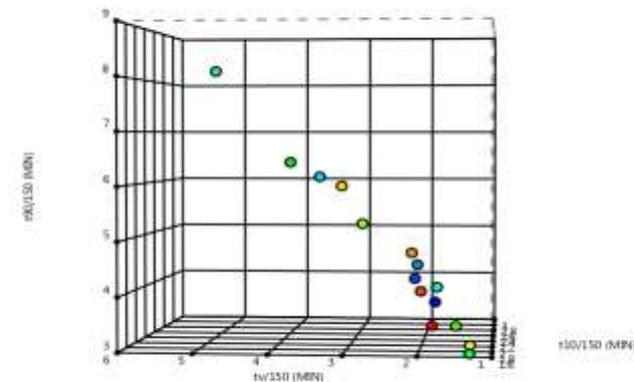
- Correlation diagrams allowing a first evaluation of the data:
  - Following inherent logic of rubber principles
- Histograms
- Scatter plots
  
- Further tools are
  - Contour plots
  - Desirability plots, whether target is met / or failed
  - Prediction

Correlation: 0.971  
 Color points by Run  
 1 21



Correlation Diagram:  
M100 over M300

Correlation: 0.888  
 Color points by Run  
 1 15

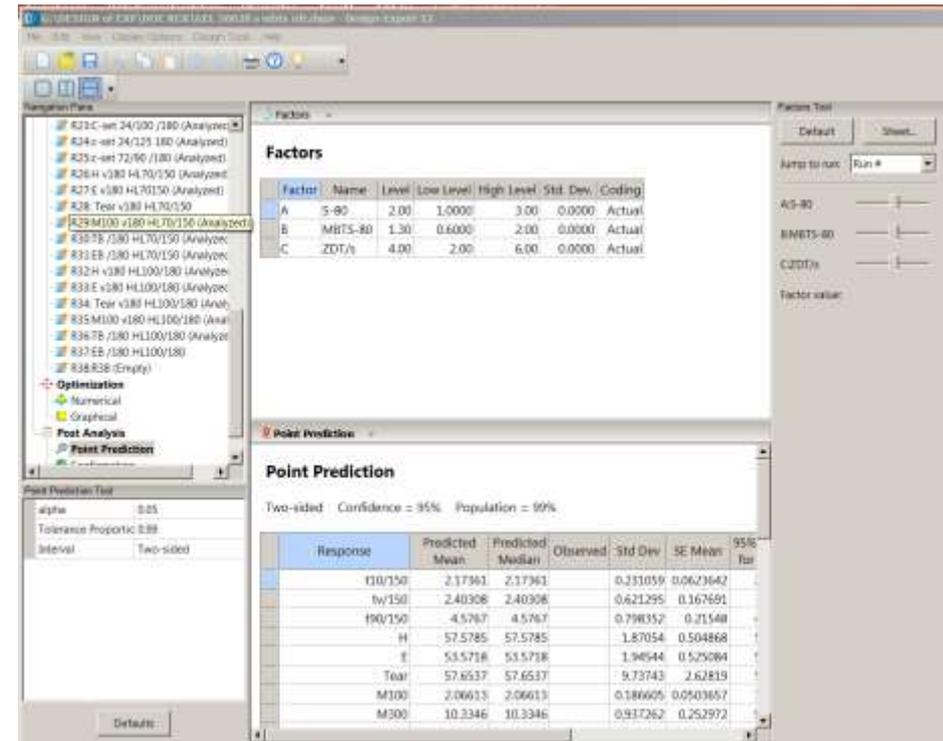


Correlation Diagram:  
t<sub>10</sub>/150C, T<sub>v</sub> 150C, and t<sub>90</sub>/150C

## 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	$A_n$	$A_{F.n}$	$A_{Fn.n}$	$A_{Fn.n}$

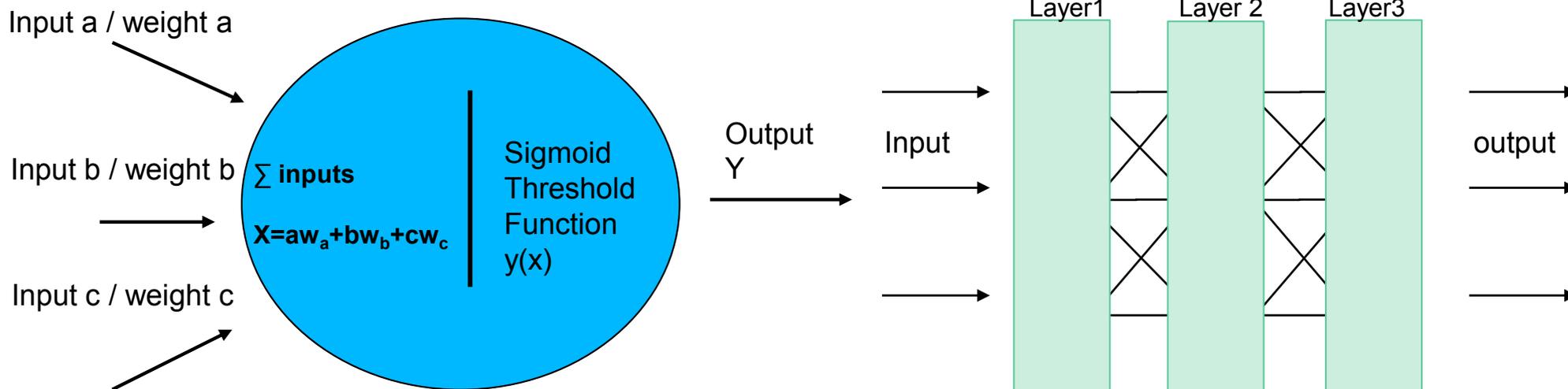


Point Prediction Tool in Design Expert®12 Software (Screen shot)

## Algorithm used in AI 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**

# 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

## Matrix multiplication with the terminology of neuronal nets

( $w$  – weight,  $I$  = Input,  $O$  – 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:

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

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

$$Y = 1/(1+e^{-x})$$

Values for subsequent layers change to:

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

## 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

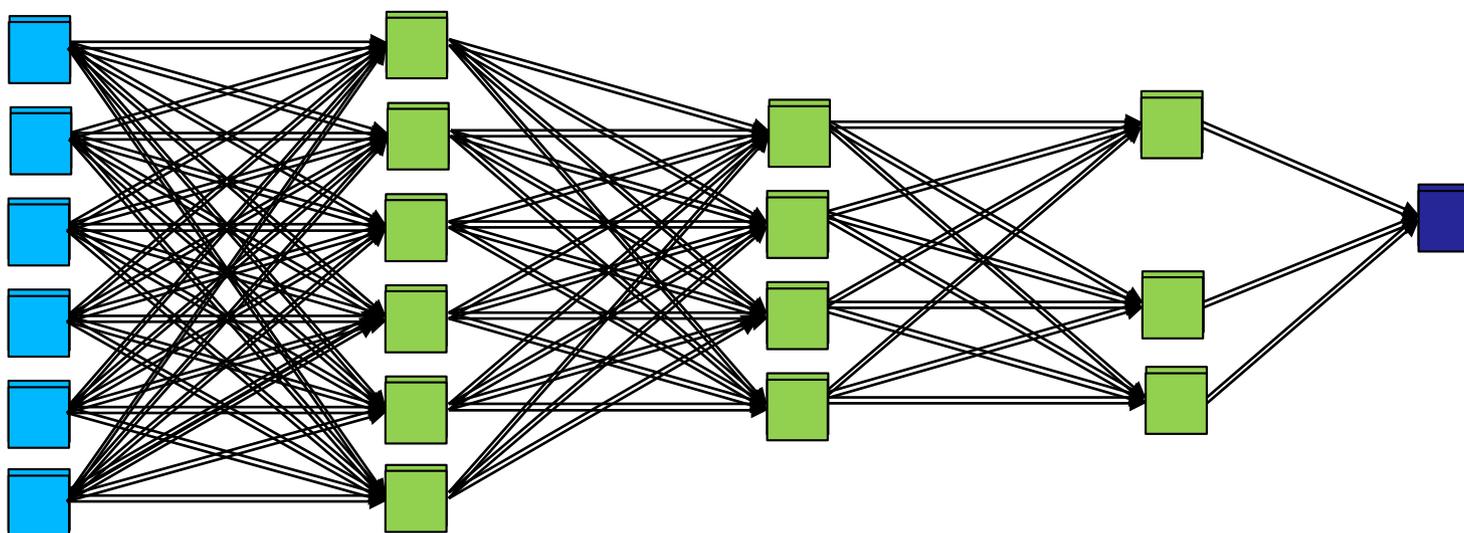
# Rubber Recipe Data Stag

- Ingredients
  - Normalized to 100 parts  
polymer = phr

## Properties

- 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

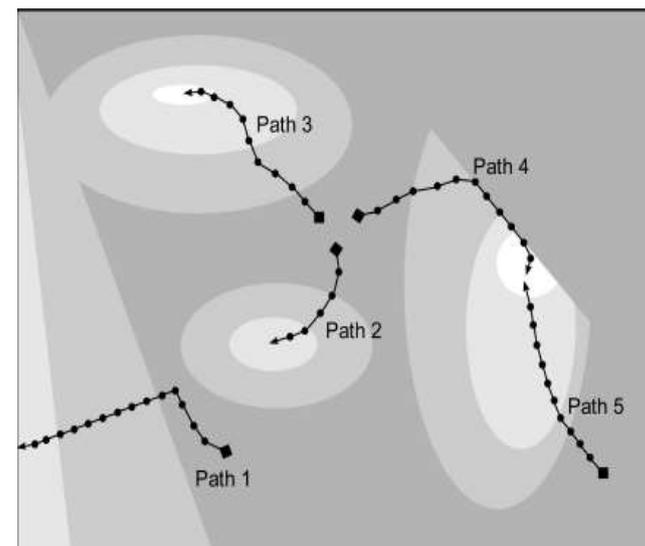
Each square represents recipe and property information

- ❑ 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.

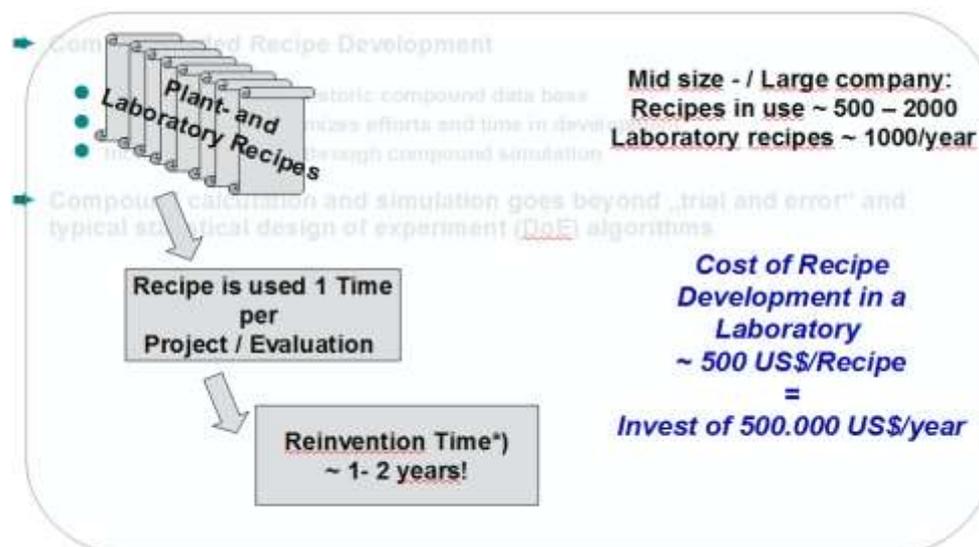


➤ **Output:**

**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 AI Programming*)

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

The screenshot displays the GrafCompounder version 4.0.4 interface. The main window is titled "GrafCompounder version 4.0.4 - G:\Seminare\01 - Seminars\Course 2021\Forum Vienna 21\Presentation HJG\NR Data-Code Cost Dichte test.Lgc".

**Input data table:**

Code:	NR Testdaten	Ingredients:	Cost:	Density:	50ALS11	50ALS12	50ALS13	50ALS14	50ALS15	50ALS16	50ALS17	50ALS18
A001	SMR 10		290.00	0.92	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
A003	SMR CV60		290.00	0.92								
A004	SMR L		310.00	0.92								
B003	N330		115.00	1.80	10.00	30.00	50.00	25.00	45.00	75.00	45.00	
B004	N330		115.00	1.80								
B005	N550		115.00	1.80								
B006	N762		115.00	1.80								
C010	CaCO3		24.00	2.71								
D002	Naphthenic Oil		116.00	0.89	5.00	25.00	45.00	5.00	25.00	45.00	5.00	
D001	Paraffinic Oil		120.00	0.90								
E001	ZnO		385.00	5.60	5.00	5.00	5.00	5.00	5.00	5.00	5.00	
E021	Zn-2EH		150.00	1.80								
F001	Stearic Acid		165.00	0.92	2.00	2.00	2.00	2.00	2.00	2.00	2.00	
F101	Paraffin Wax		130.00	0.90								
G001	TMQ		924.00	1.15								
G001	IPPD		924.00	1.15	2.00	2.00	2.00	2.00	2.00	2.00	2.00	
H001	S-80		158.00	1.80	1.88	1.88	1.88	1.88	1.88	1.88	1.88	
K004	DPG-80		420.00	1.28								
H802	DTDM-80		360.00	1.28								
K001	TMTD-80		396.00	1.11								
K005	CBS-80		708.00	1.28	0.63	0.63	0.63	0.63	0.63	0.63	0.63	

**Properties table:**

Code:	Properties:	50ALS11	50ALS12	50ALS13	50ALS14	50ALS15	50ALS16	50ALS17	50ALS18
PR001	MooneyML(1+4) 100°C	32.00	36.00	31.00	34.00	30.00	42.00	60.00	
PR002	MooneyTS 120°C	28.00	28.00	32.00	28.00	32.00	22.00	20.00	
PR003	Density	1.08	1.12	1.16	1.13	1.16	1.19	1.19	
PR004	Hardness	42.00	41.00	40.00	48.00	48.00	52.00	61.00	
PR007	M300	1.80	3.00	3.00	4.40	4.60	5.30	6.00	
PR008	TS	25.00	21.00	15.00	25.00	20.00	15.30	23.00	
PR009	EB	785.00	725.00	690.00	715.00	705.00	615.00	560.00	
PR020	C-Set -26°C (24h [%])	22.00	28.00	30.00	17.00	19.00	35.00	20.00	
PR021	C-Set 0°C (24h [%])	10.00	14.00	14.00	8.00	12.00	16.00	13.00	

**Criteria table:**

Name	Min	Max	From	To	Wei	Trndoff
SMR 10	0	100				
SMR CV60	0	100				
SMR L	0	100				
N330	0	75				
N336	0	40				
N550	0	60				
N762	0	65				
CaCO3	0	20				
Naphthenic Oil	0	45	8	10	10	
Paraffinic Oil	0	10				
ZnO	5	10				
Zn-2EH	0	1				
Stearic Acid	0	2				
Paraffin Wax	0	2				
TMQ	0	2				
IPPD	2	4				
S-80	0.31	4.08				
DPG-80	0	0.25				
DTDM-80	0	1.25				
TMTD-80	0	1.5				
CBS-80	0	2.63				

**Output table:**

Mixture!	Mobari!
2.75	1.5
97.25	98.5
7.045	0.125
19.45	20.2
0.55	0.05
7.9875	2.0825
0.005	0.125
5	5
2	2
1.945	1.97
0.025	0.025
2.9725	2.985
2.35685	3.56525
	0.003125
	0.015625
1.452825	1.161575
30.7738	29.402
32.0873	32.907
1.0508	1.040925
50.01	52.711
5.08455	6.2179
25.69705	26.107
634.6817	598.36
22.4705	15.389
8.525	6.4265

**Summary table:**

Total	148.51	196.51	226.51	161.51	201.51	251.51	181.51
Density	1.097	1.176	1.128	1.138	1.145	1.172	1.186
Cost (per	262.484	237.496	270.591	259.16	235.861	219.811	255.359
Cost (per	239.274	212.729	195.559	227.733	205.454	187.562	215.311

**Criteria Summary:**

Total	132.8	251.5
Density (calc.)	1.027	1.214
Cost (per vol)	219.8	326.3
Cost (per	187.5	301.9

**Output Summary:**

Total	150.766875	139.35065
Density	1.055	1.045
Cost (per vol)	277.212	287.094
Cost (per	262.76	274.731

Sum of recipe ratios (should be 100%)  
100

Number format: 12345.67

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

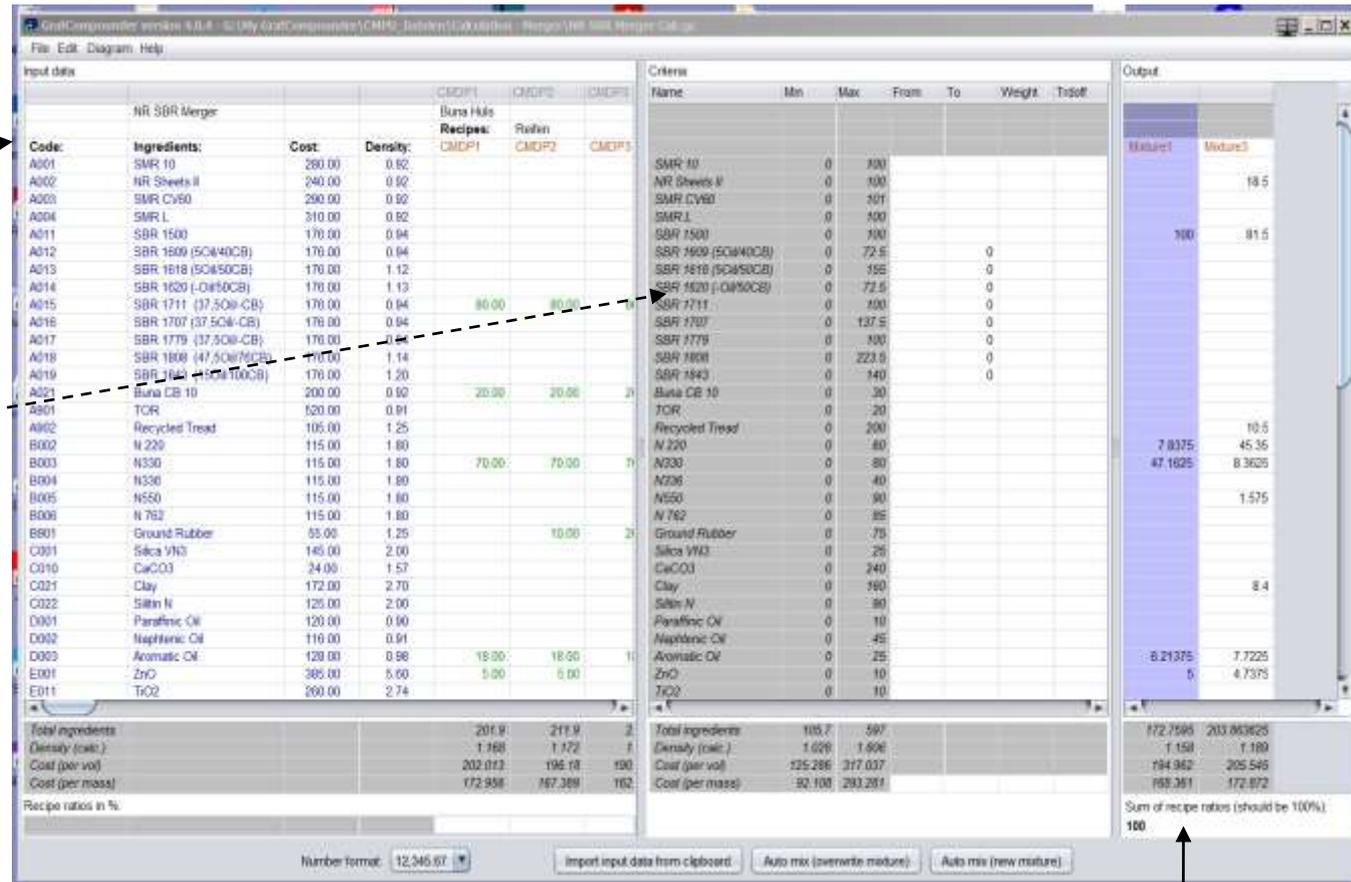
## GrafCompounder Screen

### ■ Data field

- Code
- Ingredient name
- Cost information
- Density

### ■ Criteria field

- Min/Max Value
- Query column:
  - From
  - To
  - Weight
  - TrdOff



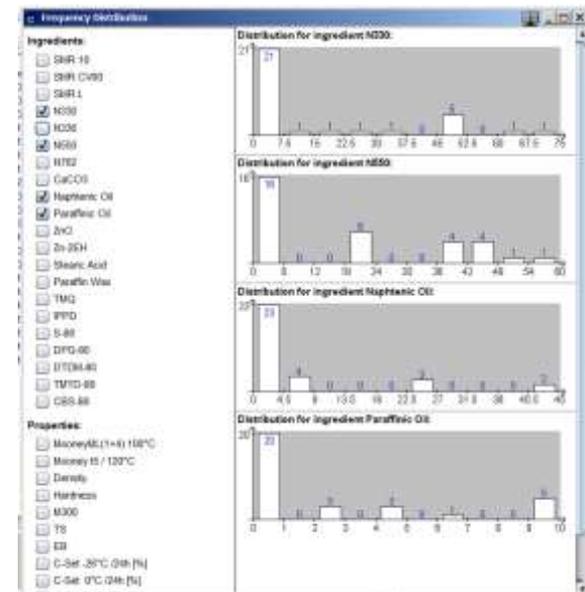
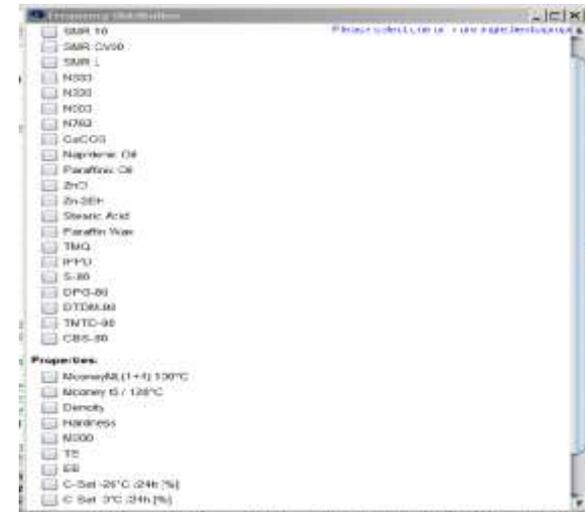
The screenshot displays the GrafCompounder software interface. It is divided into several sections:

- Input data:** A table listing ingredients with columns for Code, Ingredients, Cost, and Density. It includes items like SMR 10, NR Sheets II, and various SBR grades.
- Criteria:** A table with columns for Name, Min, Max, From, To, Weight, and TrdOff. It lists criteria such as SMR 10, NR Sheets II, and Bura CB 10.
- Output:** A table showing calculated values for ingredients, including a 'Sum of recipe ratios (should be 100%)' which is currently 100.
- Summary:** A section at the bottom providing totals for ingredients, density, cost, and recipe ratios.

### ➤ 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

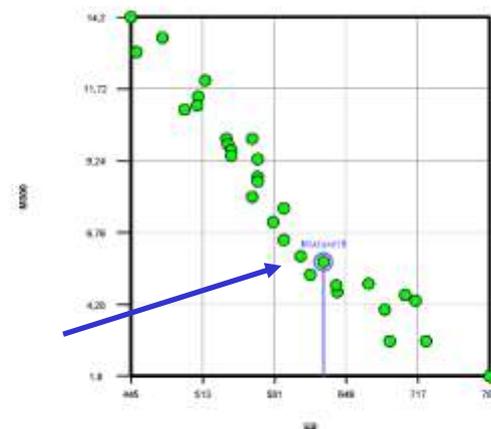
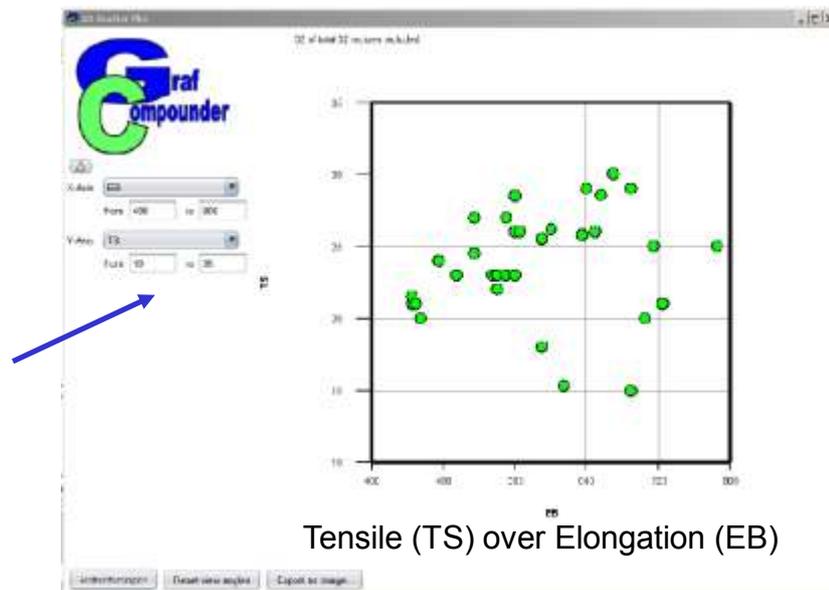
### ➤ Example: Tensile (TS) over Elongation at Break (EB)

#### ➤ Scale for axis

#### Selected

- automatically / - manually

#### ➤ Highlight recipe for evaluation or exclusion



Modulus M300 over Elongation (EB)

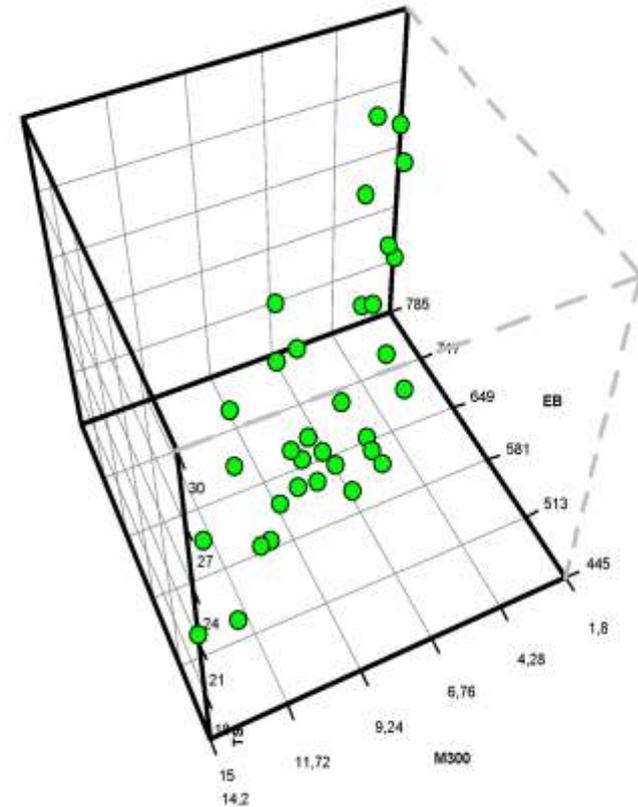
## Database Diagnose Tool

### ➤ 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



## How to do a query:

### 1. Ingredients

**Exclude with target = 0**

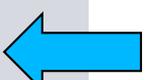
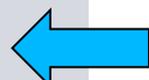
### 2. Properties

✓ 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

From	To	Weight	Trdoff	Mixture1	Mixture2
2					0.025
4				2.9725	2.985
4.06				2.35885	3.585825
0.25					0.003125
1.25					0.015625
1.5					
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	50.01	52.711
14.2	7			5.08455	6.2179
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
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





## 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)**
  - 2. 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

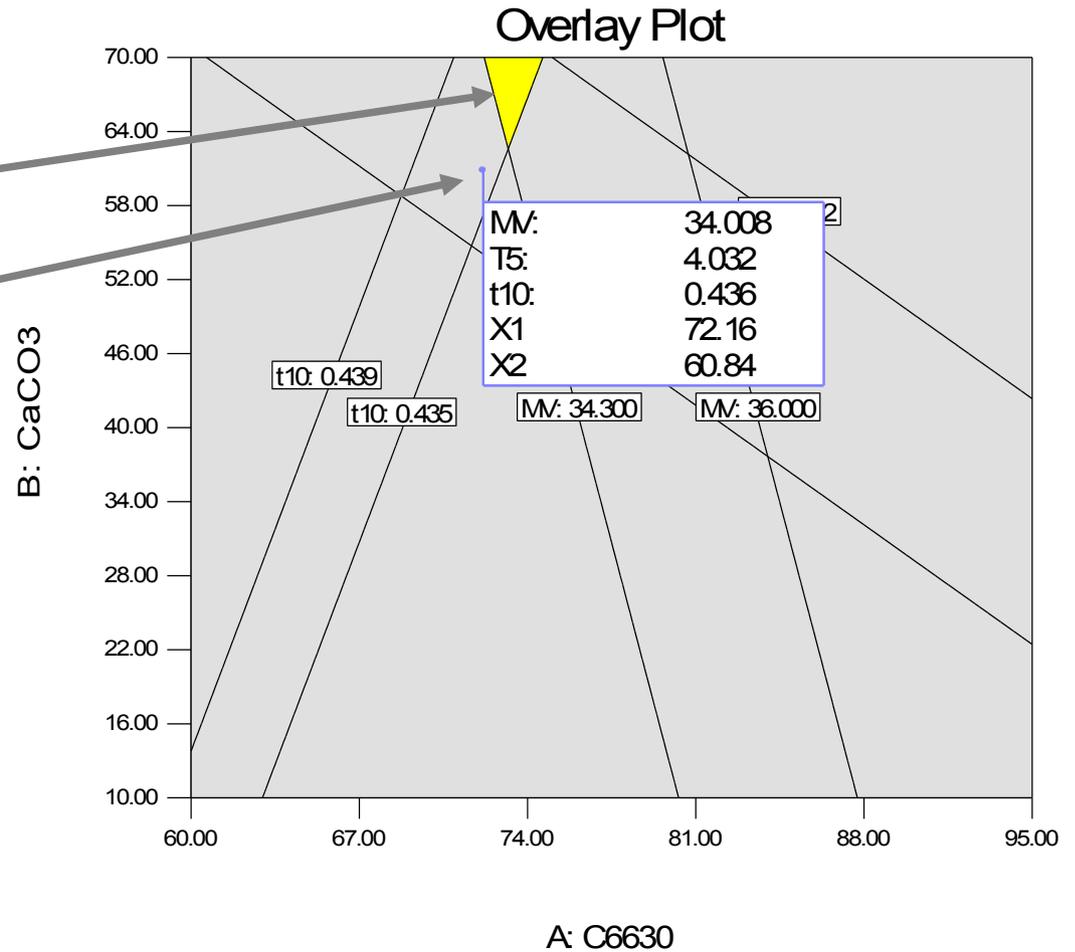
Polymer used: EPDM (Vistalon 8600)

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

1.

- A fractional factorial DoE with 11 compounds only!

**Optimization area calculated with Design Expert**  
**Solution given by GrafCompounder**  
**with the additional condition (CC 6630 – 73 phr)**



2.

## 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

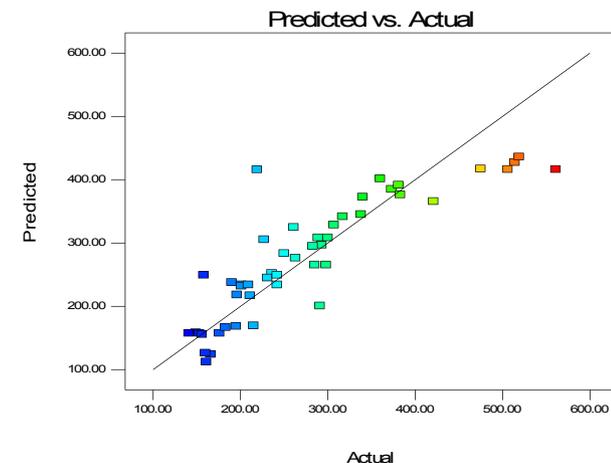
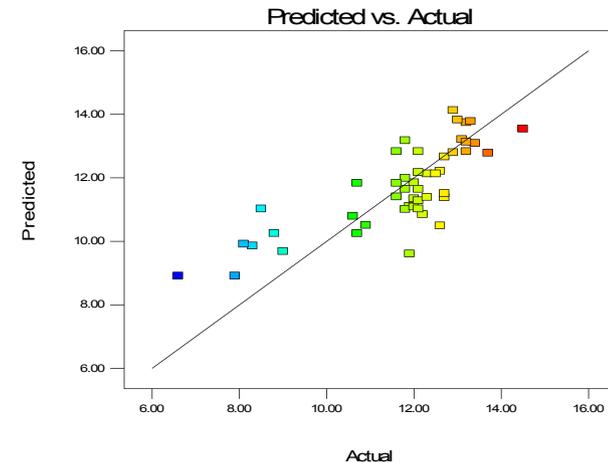
- Sulfur has larger influence followed by DTDC and TiTBD, but negative

## 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



- **Boundary Conditions**

- **Select boundaries**

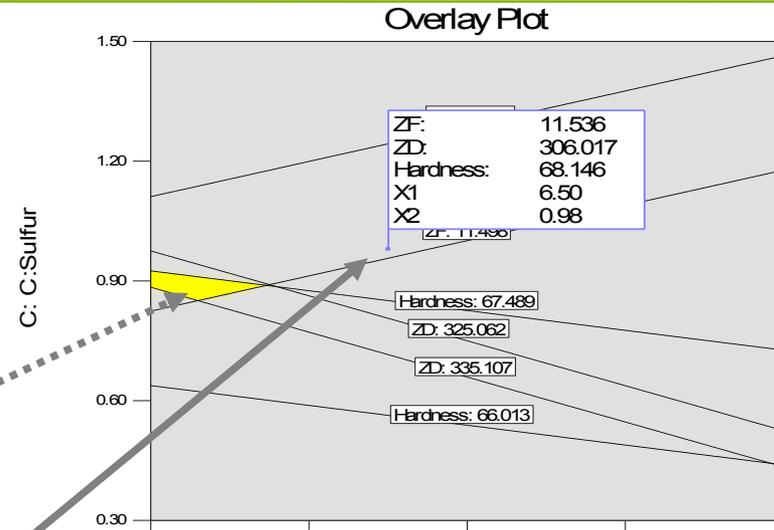
**ZF-MPa : 11.5-12.0**

**ZD-% : 325-335**

**H-° ShA : 65-67**

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

GrafCompounder result is tagged with a flag.



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
<b>Hardness</b>	69.99	70.00	0.01%
<b>Tensile at Break</b>	19.56	18.75	2.16%
<b>Elongation at Break</b>	338.61	339.00	0.06%
<b>M 100</b>	3.87	4.75	9.26%
<b>C-Set 23°C / 22h</b>	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**

## ➔ 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**

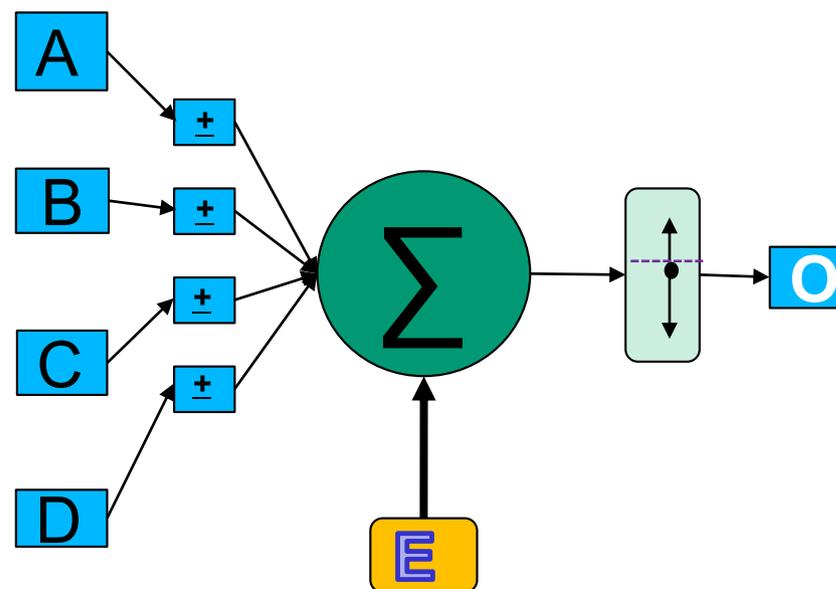
- **Unorganized / Unlimited Database**
  - **Measurement error unknown**
  - **Incongruent values (due to different test methods)**
  - **Raw materials in Laboratory (Storage time?)**
  - **Missing values**
- **Multiple Factor Query**
- **Compound Simulation according Multi Targets**

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

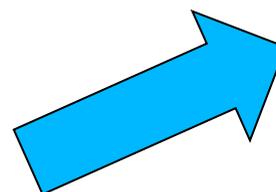
## Challenges and Hints to machine learning with “Back Propagation” in rubber compounding:

- 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

Input    Weight    Summing    Activation    Output



**Errors:**  
The nerve cell learns via errors to optimize the weighting and the values of the input function.



## Conclusion:

- Compounder Program

- Compound Cost estimation
- Starting Formula for Compound Development
- Simulation of Recipe for Specification Adjustments
- Historic Data usage and improved Data storage

- Statistic Experimental Design

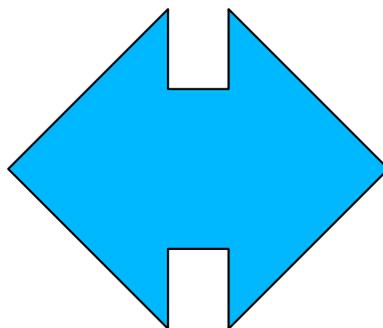
- Evaluation of New Materials
- Cost optimization in Compounding
- Design / Improvement of New Processes
- Process Window to Control Quality according SPC

- **Combination of Database / Compound Simulation with Statistic Experimental Design Experimentation Procedure**

**Both methods have their justification.**

## GRAFCOMPOUNDER

- **History**
- **Analyze**
- **Simulate**
- **Select**
- **Confirm**



## EXPERIMENTAL DESIGN

- **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](http://www.grafcompounder.com)***