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Compound Development

Advantage of Compound Development with the PC-Program “GrafCompounder”

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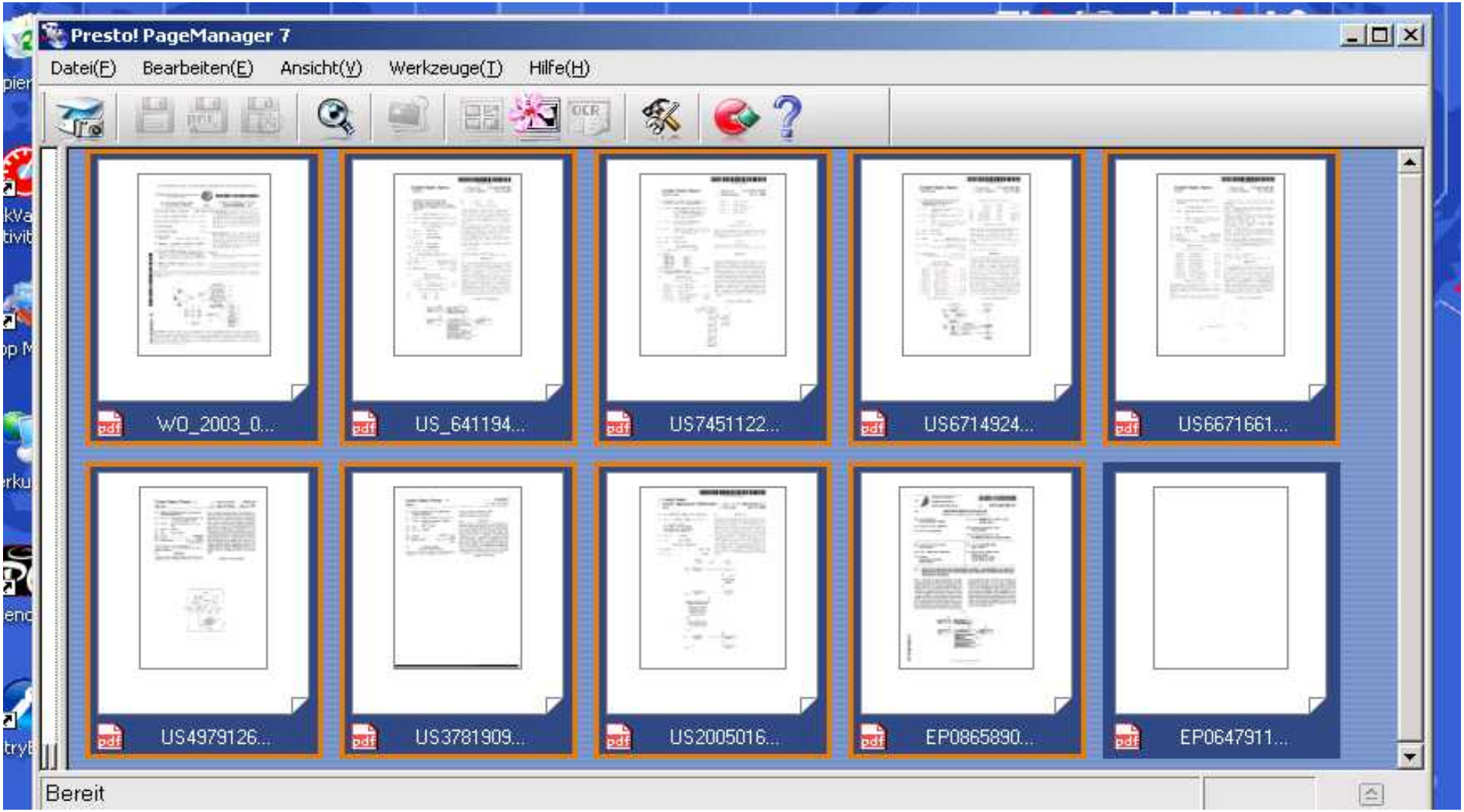
Educational & Knowledge-based Organization

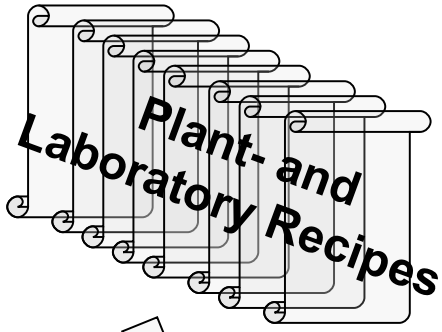
Compound Development

- **Advantage of a PC-Program**
  - **Motivation for Program Development**
  - **Description of the GrafCompounder?**
  - **Comparison with Statistic Experimental Design (DoE)**
  - **Combination of Grafcompounder with DoE**
  - **Advantages / Summary**

Motivation for Program Development



Motivation for Program Development



Recipe is used 1 Time per Project / Evaluation

**Reinvention Time*)
~ 1- 2 Jahre!**

) personal Estimation

**Mid size - / Large company:
Recipes in use ~ 500 – 2000
Laboratory recipes ~ 1000/year**

***Cost of Recipe Development in a Laboratory
~ 500 US$/Recipe
=
Invest of 500.000 US$/year***

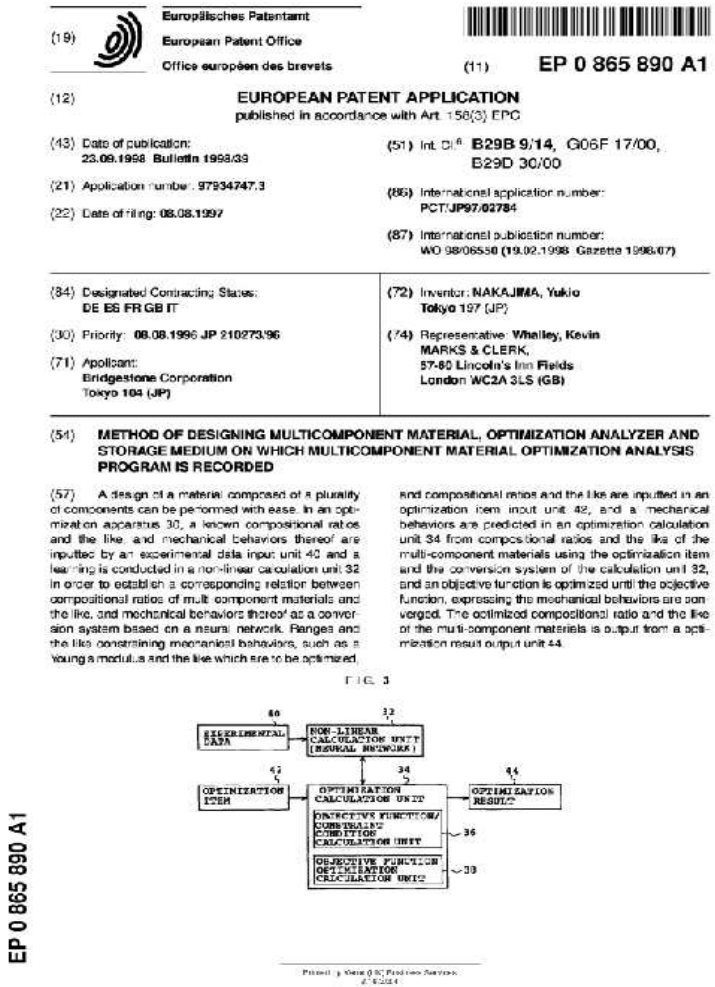
Motivation for Program Development

➡ **Question:**

- **Why we can hardly take Compound Databases as working capital,**
  Saving time and effort in our daily work?
  - **Avoiding reinvention**
  - **Increase our compounding knowledge.**
  - **Gaining room for really new ideas in compound development**

Motivation for Program Development

- **Patent EP 0865 890 A1 (Bridgestone) is dealing with compounds used in tire manufacturing**
  - **Dependency of factor – response relationship with none linear regression equation.**
  - **Usage of a function to determine boundary conditions.**
  - **Identification of a compound with targeted properties.**



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(54) METHOD OF DESIGNING MULTICOMPONENT MATERIAL, OPTIMIZATION ANALYZER AND STORAGE MEDIUM ON WHICH MULTICOMPONENT MATERIAL OPTIMIZATION ANALYSIS PROGRAM IS RECORDED

(57) A design of a material composed of a plurality of components can be performed with ease. In an optimization apparatus 30, a known compositional ratios and the like, and mechanical behaviors thereof are inputted by an experimental data input unit 40 and a learning is conducted in a non-linear calculation unit 32 in order to establish a corresponding relation between compositional ratios of multi-component materials and the like, and mechanical behaviors thereof as a conversion system based on a neural network. Ranges and the like constraining mechanical behaviors, such as a Young's modulus and the like which are to be optimized, and compositional ratios and the like are inputted in an optimization item input unit 42, and a mechanical behaviors are predicted in an optimization calculation unit 34 from compositional ratios and the like of the multi-component materials using the optimization item and the conversion system of the calculation unit 32, and an objective function is optimized until the objective function, expressing the mechanical behaviors are converged. The optimized compositional ratio and the like of the multi-component materials is output from a optimization result output unit 44.

FIG. 3

EP 0 865 890 A1

Motivation for Program Development

- **The patent US 7541122B2 (Fa. Honeywell) deal with „empirical“ DoE with the help of neuronal network algorithm**
  - **Datenbase from historical compound data**
  - **Elimination of foulty data sets out of the data base**
  - **Calculation of a compound with the help of none linear neuronal network algorithm**
  - **Building of a equation for the simulation of the correlation between factors (compound ingredients) and responses (properties).**

US007451122B2

(12) **United States Patent**
Dietrich et al.

(10) **Patent No.:** **US 7,451,122 B2**
(45) **Date of Patent:** **Nov. 11, 2008**

(54) EMPIRICAL DESIGN OF EXPERIMENTS USING NEURAL NETWORK MODELS

(75) Inventors: **Paul F. Dietrich**, Brooklyn Park, MN (US); **Sunil K. Menon**, Golden Valley, MN (US); **Dinkar Mylaraswamy**, Fridley, MN (US); **Lewis P. Olson**, Apple Valley, MN (US)

(73) Assignee: **Honeywell International Inc.**, Morristown, NJ (US)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 280 days.

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G06N 3/02 (2006.01)

(52) **U.S. Cl.** **706/15**

(58) **Field of Classification Search** .... None

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OTHER PUBLICATIONS

(Continued)

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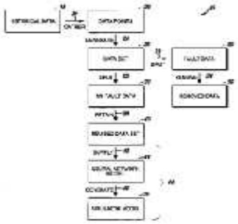
(57) **ABSTRACT**

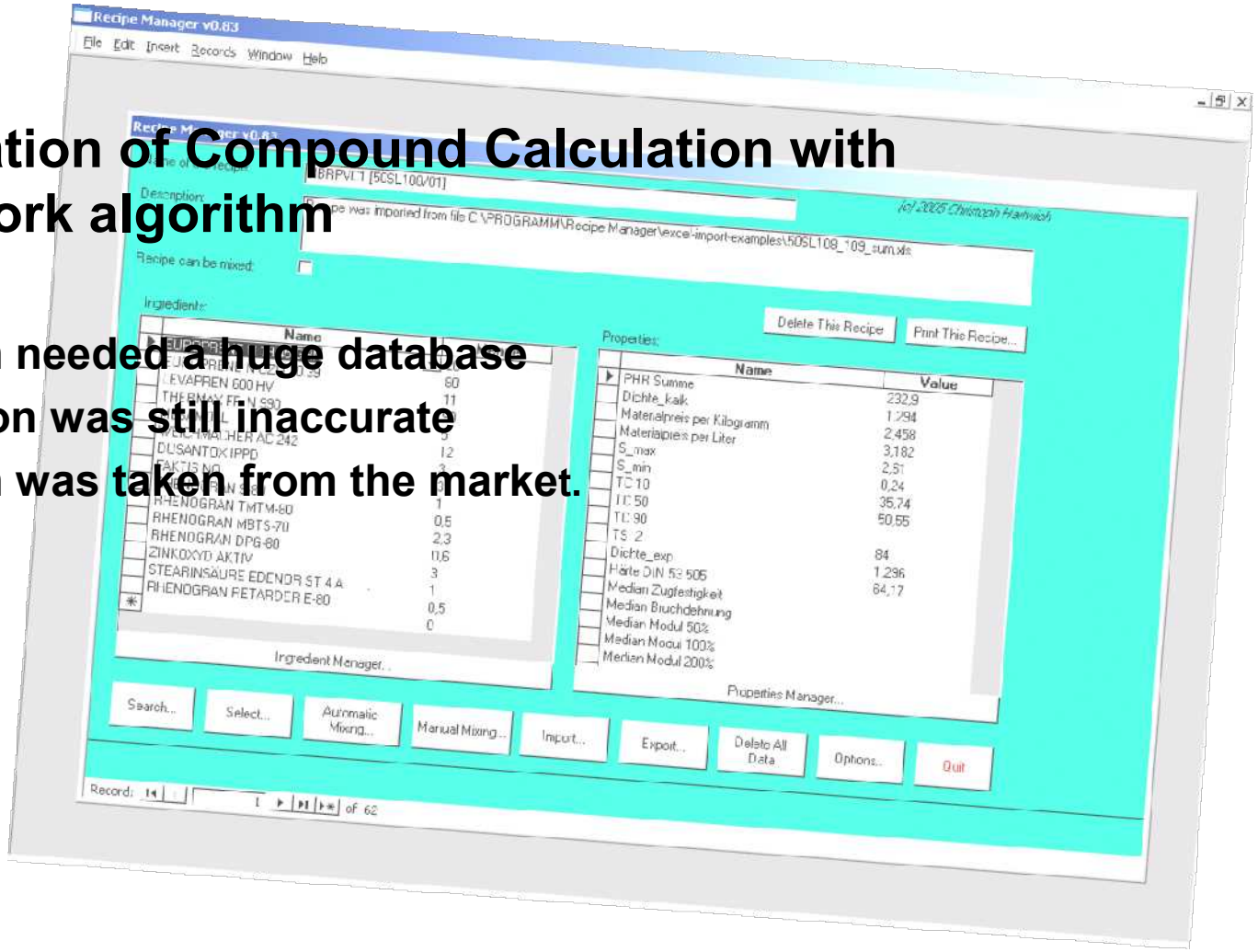
Methods and apparatus are provided pertaining to a design of experiments. The method comprises generating a data set from historical data, identifying and removing any fault data points in the data set so as to create a revised data set; supplying the data points from the revised data set into a nonlinear neural network model; and deriving a simulator model characterizing a relationship between the input variables and the output variables. The apparatus comprises means for generating a data set from historical data; means for identifying and removing any fault data points in the data set so as to create a revised data set; means for supplying the data points from the revised data set into a nonlinear neural network model; and means for deriving a simulator model characterizing a relationship between the input variables and the output variables.

24 Claims, 7 Drawing Sheets



Motivation for Program Development

- **Commerzialisation of Compound Calculation with neuronal network algorithm**
  - **CAD-CHEM**
    - **Program needed a huge database**
    - **Prediction was still inaccurate**
    - **Program was taken from the market.**

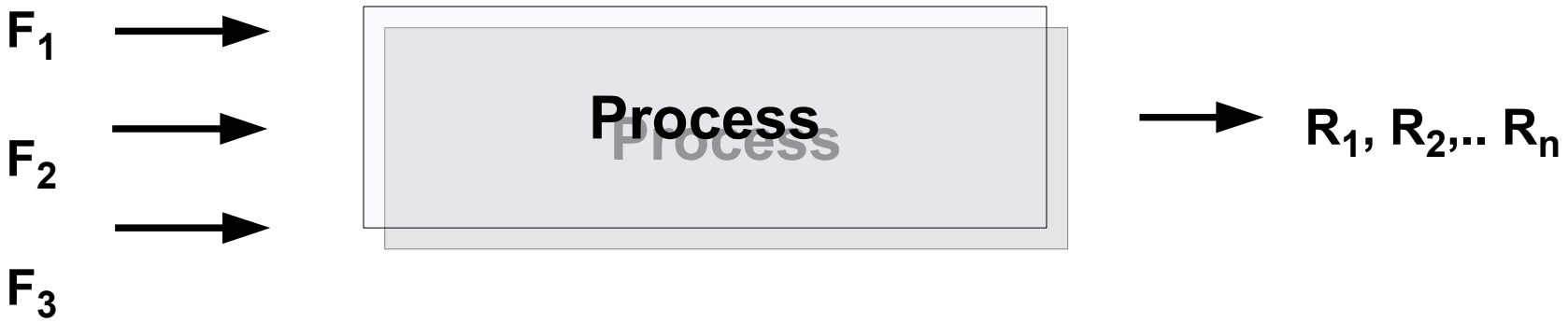


Motivation for Program Development

Statistic Experimental Design (DoE) allows a factor – response calculation with regression equations

Influences:
Factors are varied

Effects:
Responses are measured

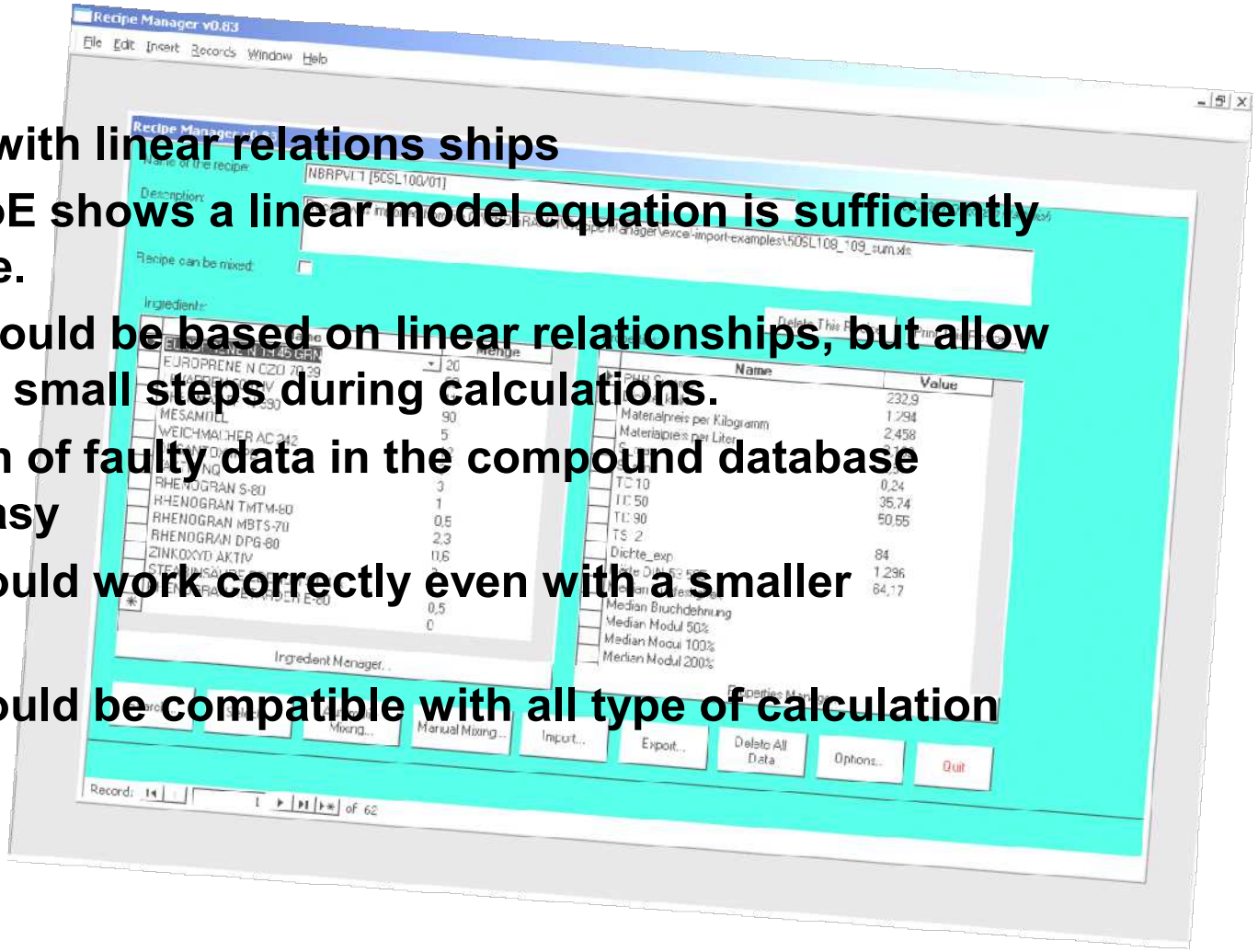


- **Objective of the Experiment should be the identification of the most important factors (F_1,..F_n), to be able to measure Effects (Responses R_1,...R_n) and to describe there dependency in a mathematical equation:**

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

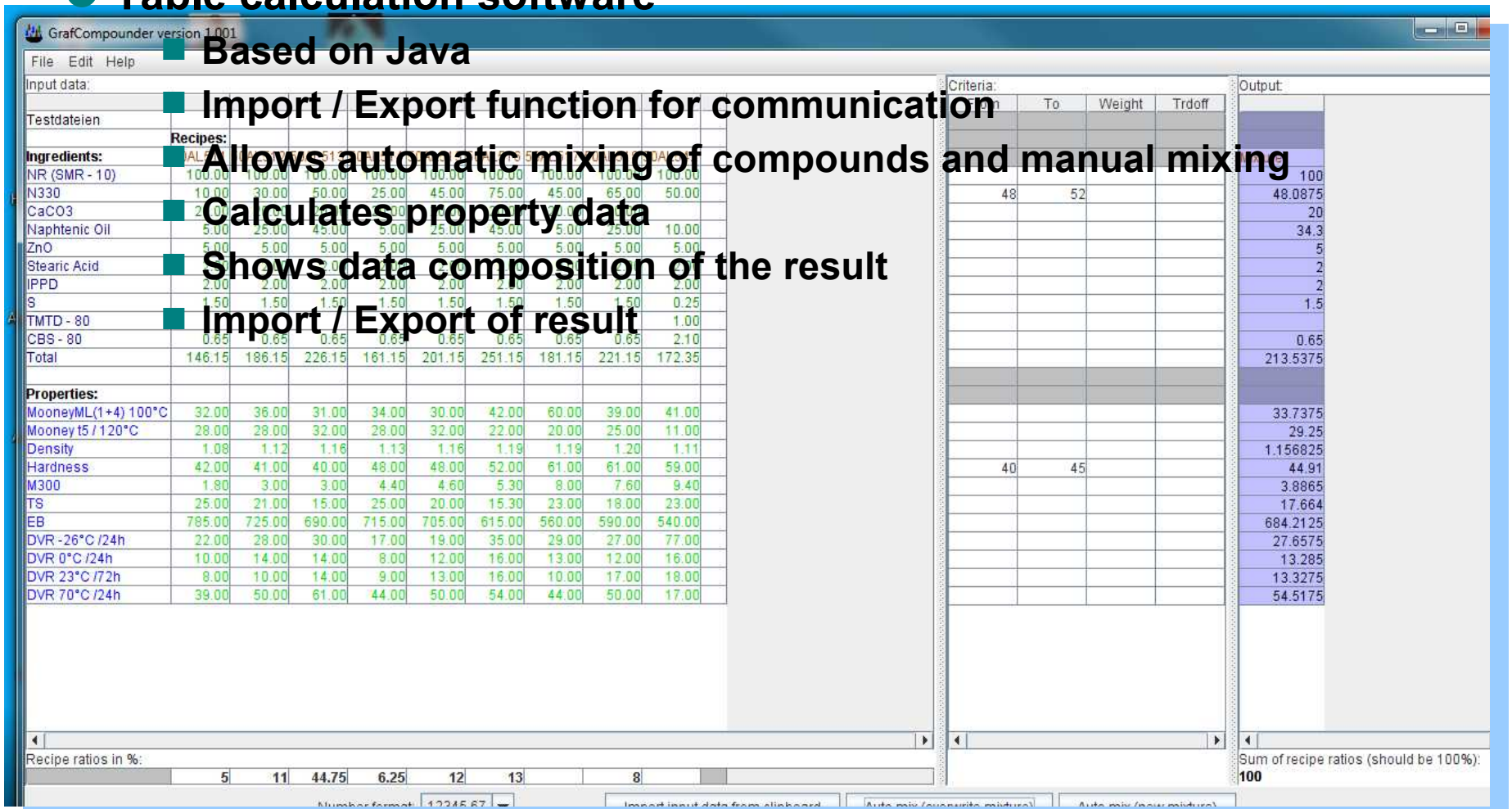
Design Guide
for GrafCompounder

- Calculation with linear relations ships
  - Most DoE shows a linear model equation is sufficiently accurate.
  - Math should be based on linear relationships, but allow multiple small steps during calculations.
- Identification of faulty data in the compound database should be easy
- Program should work correctly even with a smaller database
- Program should be compatible with all type of calculation programs



Description of GrafCompounder

- **GrafCompounder**
  - **Table calculation software**
    - **Based on Java**
    - **Import / Export function for communication**
    - **Allows automatic mixing of compounds and manual mixing**
    - **Calculates property data**
    - **Shows data composition of the result**
    - **Import / Export of result**

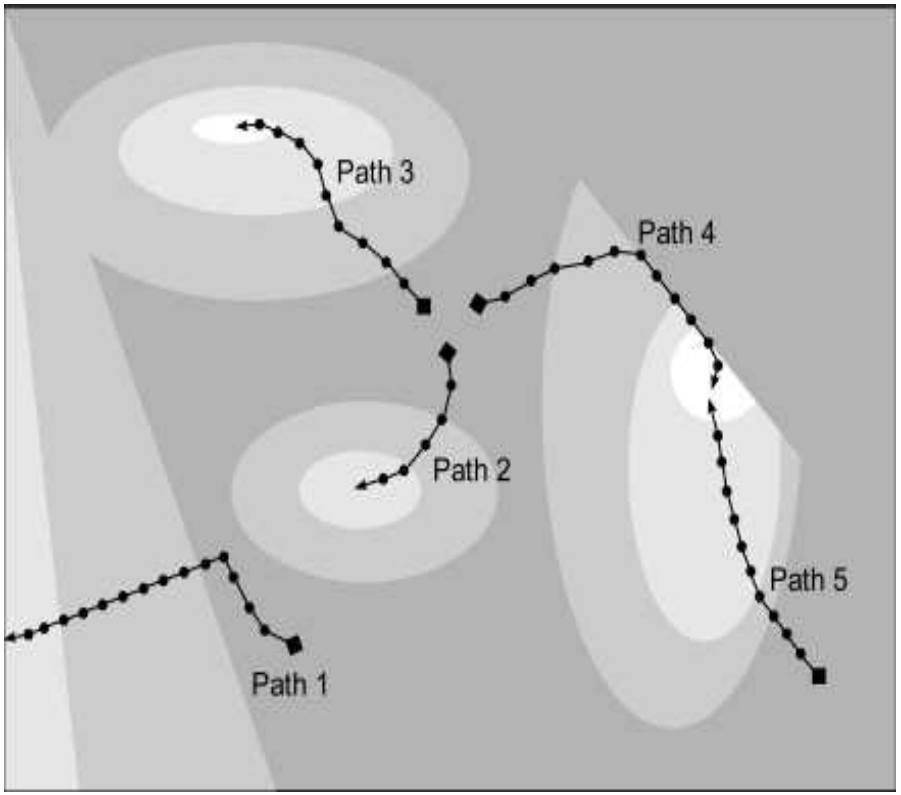


Description of GrafCompounder

- **Analysis of a recipe database with Multiple Linear Iteration (MLI)**
  - **Search criteria manageable with different weights!**
  - **Recipe Selection (Exclusion of unwanted recipes during analysis)**
    - **Avoid Analysis of none compatible Polymers**
  - **Automatic an Manual Mode**
    - **Simulation of Blends of Compounds**
  - **Property Data should be from a trustworthy source, if not your own**

Description of GrafCompounder

- **Analysis based on**
  - Measurables
  - Targets
  - Weights
  - Rating functions shows the distance between values and target
  - Iteration in small steps from different starting points
  - Check of maximum agreement with the target
- **Report of Results**
  - Recipe
  - All calculable physical properties
    - Missing data left out
  - Show all Recipes with their percentage used in an analysis



Description of GrafCompounder

- **Working with the GrafCompounder**
  - **Create a table by copy/paste from Design Expert®**
  - **Assign titles to the rows and columns with:**
    - **Recipes:**
    - **Ingredients:**
    - **Properties:**

	Recipes:		
Ingredients:	CMPD1	CMPD2	CMPD3
XXX	XXX	XXX	XXX
Properties:			
XXX	XXX	XXX	XXX

Comparison
DoE versus GrafCompounder

- **Testing the MLI-method a database is needed, which can be analyzed in different ways.**
  - **1. Example**
    - **Oil / Filler DoE (with own Experiments)**
    - **Factors: Filler 1, Filler 2, Filler3 and Oil**
  - **2. Example**
    DoE published by DuPont Dow in 1998
    - **Factors: ENB, DTDC, S, MBT, TiTBD, ZdiBC, DTP**
  - **Same Optimization criteria will be used in DoE Software (Design Expert®) and in GrafCompounder.**

Comparison
DoE versus GrafCompounder

- 1. Example
  - Oil / Filler DoE (based on own experiments)
  - Factors: Filler 1, Filler 2, Filler 3 and Oil

Comparison
DoE versus GrafCompounder

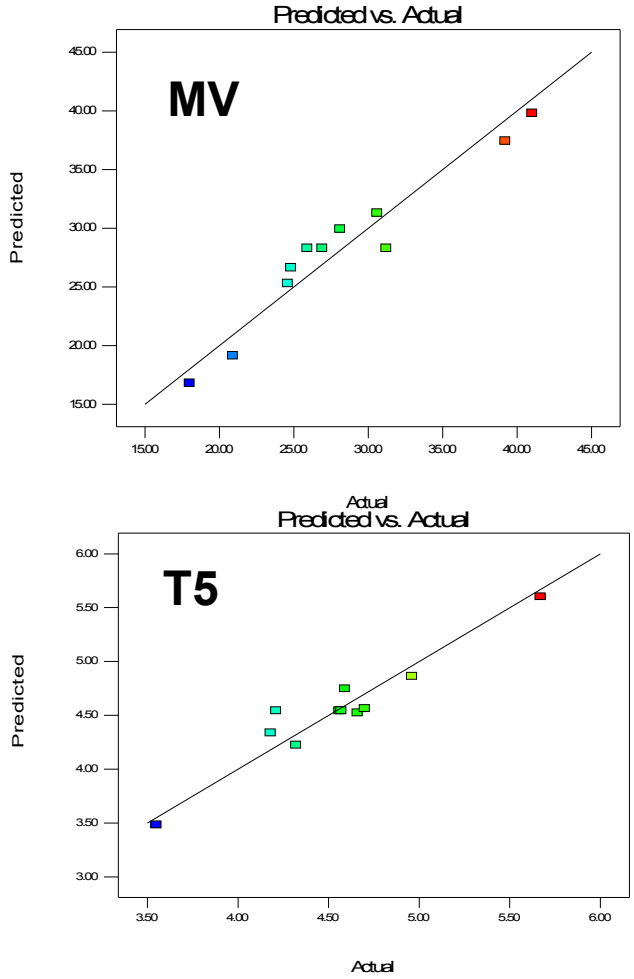
- DoE with 4 Factors
  Polymer used was Vistalon 8600
- Factor Name Units Minimum Maximum
  - A C6630 phr 60.00 95.00
    B CaCO3 phr 10.00 70.00
    C Clay phr 10.00 50.00
    D Oil phr 70.00 95.00

  - A fractional factorial DoE with 11 compounds only!

Comparison
DoE versus GrafCompounder

- **Rheological Data are examined**
  - **MV and T5 can be measured quite accurate.**

    Both are significant with a linear model equation

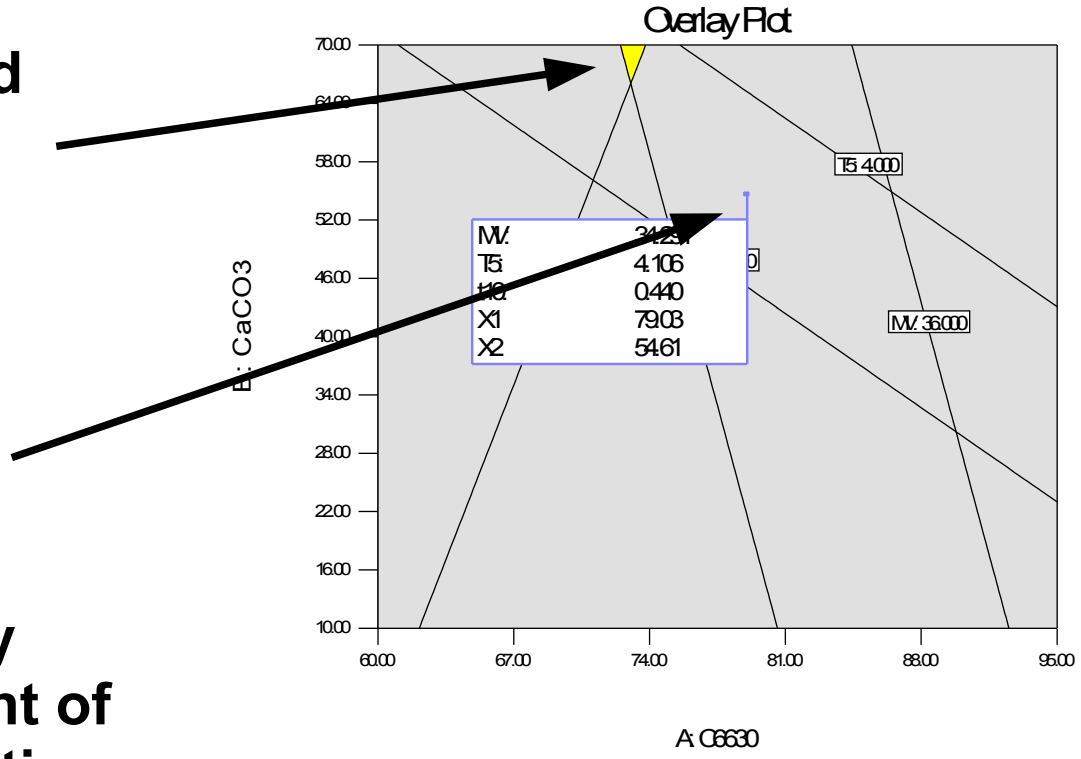


Comparison
DoE versus GrafCompounder

Ingredients	Unit	DoE Optimization	GrafCompounder
CB 6630	phr	73	79
CaCO3	phr	68	55
Clay	phr	39	39.5
Paraffinic Oil	phr	72	73
MV 120	MU	34	34.9
T5 (120°C)	min	4.04	4.2
t10 (170°C)	min	0.45	0.44

Comparison
DoE versus GrafCompounder

- Optimization area calculated with Design Expert
- Solution given by GrafCompounder



- With an additional boundary condition: take same amount of CB 6630 similar to Optimization Value in Design Expert

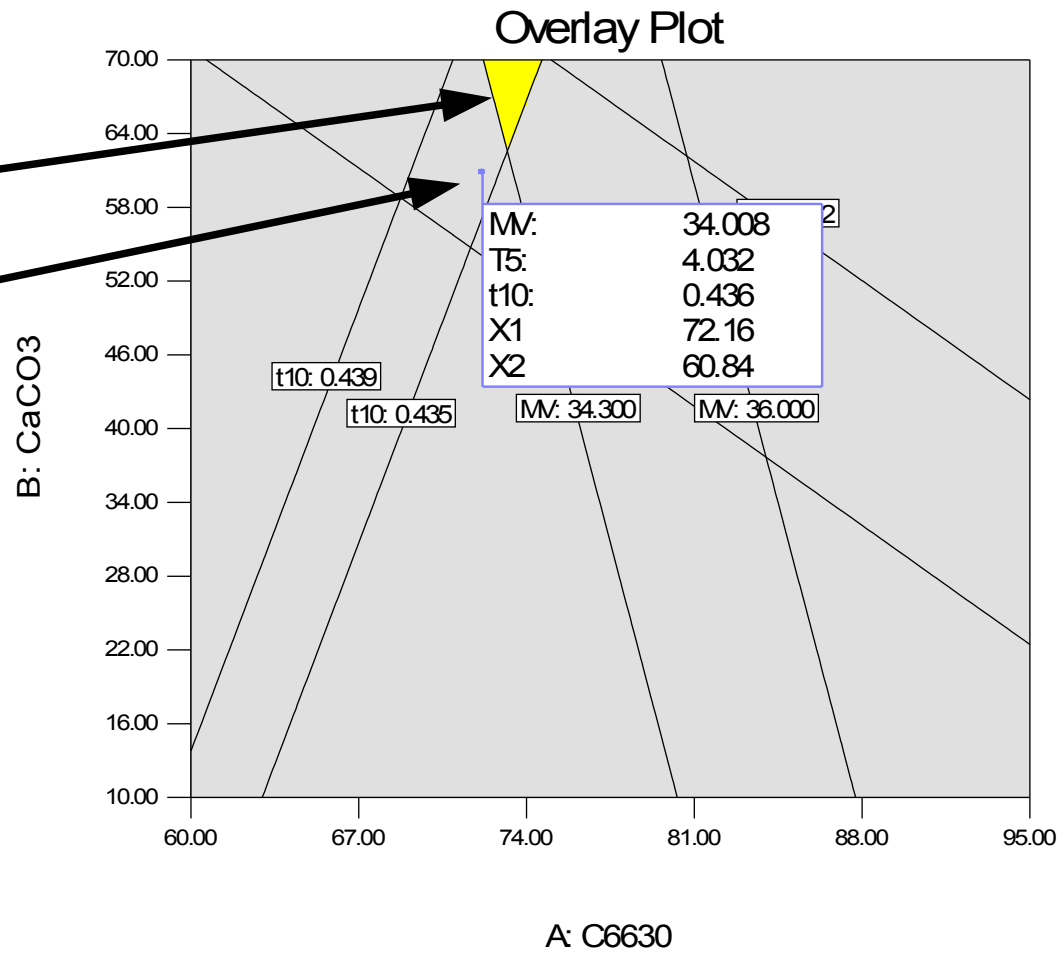
Comparison
DoE versus GrafCompounder

Ingredients	Unit	DoE Optimization	GrafCompounder
CB 6630	phr	73	73
CaCO3	phr	68	61
Clay	phr	39	32
Paraffinic Oil	phr	72	70
MV 120	MU	34	34.1
T5 (120°C)	min	4.04	4.1
t10 (170°C)	min	0.45	0.45

Comparison
DoE versus GrafCompounder

- **Optimization area calculated with Design Expert**
- **Solution given by GrafCompounder**

  with the additional condition (CC 6630 – 73 phr)



Comparison
DoE versus GrafCompounder

Ingredients	Unit	DoE Optimization	GrafCompounder	DoE Point Prediction
CB 6630	phr	73	73	73
CaCO3	phr	68	61	61
Clay	phr	39	32	32
Paraffinic Oil	phr	72	70	70
MV 120	MU	34	34.1	34.2 ± 3
T5 (120°C)	min	4.04	4.1	4.01 ± 0.25
t10 (170°C)	min	0.45	0.45	0.43 ± 0.07

Comparison
DoE versus GrafCompounder

- What we have learned

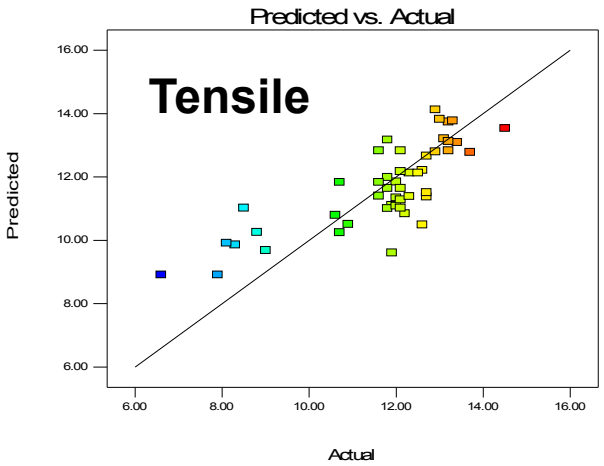
  - Calculation with GrafCompounder and optimization result with Design Expert has some characteristic differences
    - GrafCompounder gives always one solution
    - Design Expert provides an area, where you can identify a solution
    - With an additional boundary condition both solutions can be narrowed, that they fit into 95% confidence interval and measurement error of test methods for the repsonses.

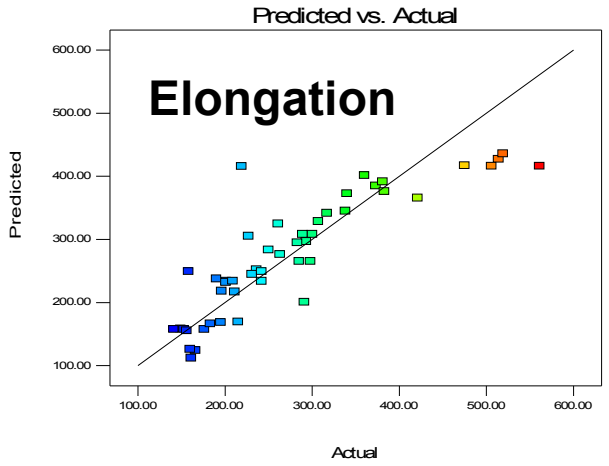
Comparison
DoE versus GrafCompounder

- 2. Example
- DoE published by DuPont Dow in 1998
  - Factors: ENB, DTDC, S, MBT, TiTBD, ZdiBC, DTP
  - DoE with 41 Experiments

DoE Analysis and Result

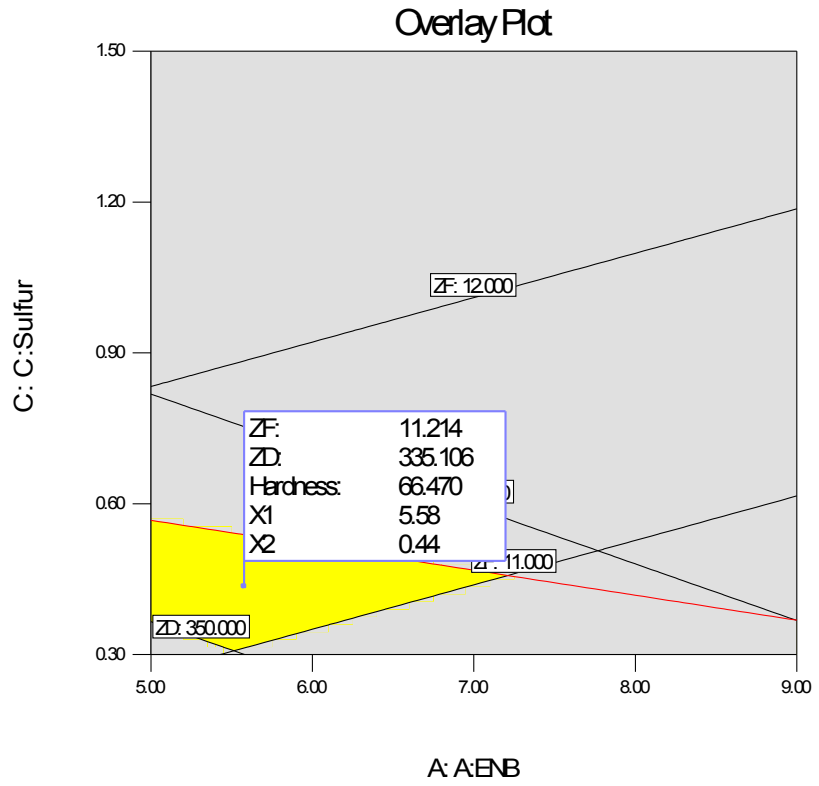
- **Tensile at break is significant with linear model**
  - **Sulfur has larger influence followed by DTDC and TiBTD, but negative**
- **Elongation is significant with quadratic model, but linear model is a sufficient fit**
  - **Sulfur has the largest influence followed by DTDC**
- **Hardness is sufficient significant with linear model as well**
  - **Main influence Sulfur, DTDC**





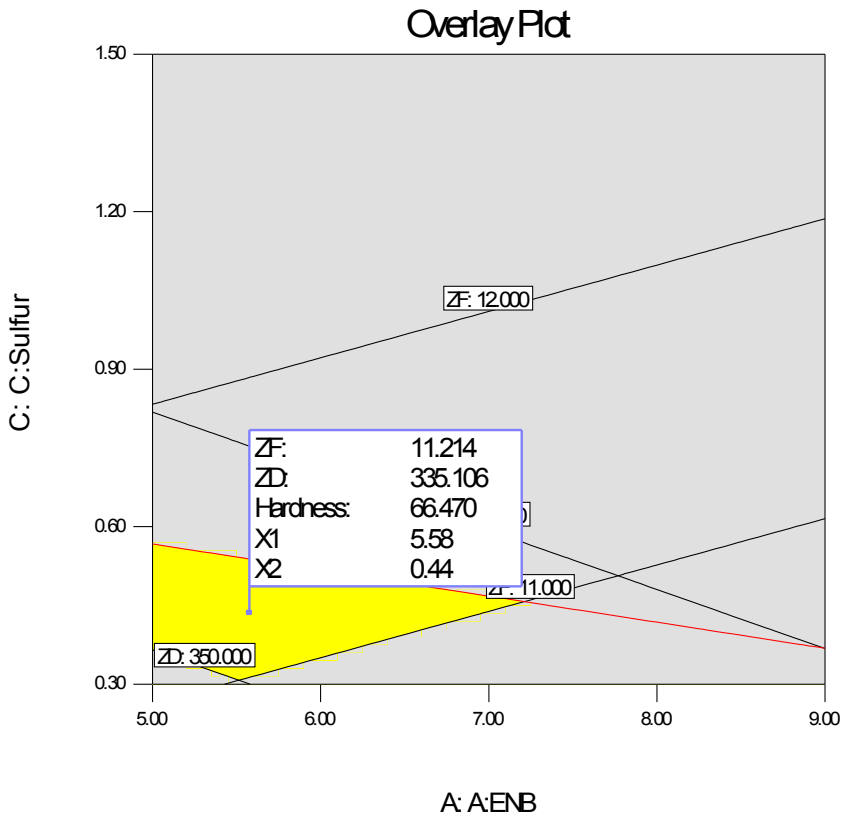
DoE Analysis and Result

- **Selection of responses for the test with graphical optimization:**
  - **Hardness**
    65°ShA - 70°ShA
  - **Tensile at break**
    11MPa – 12 MPa
  - **Elongation of Break**
    350 % - 400 %
- **Flag points to one solution**



DoE Analysis and Result

- **Factor values giving this result**
  - **ENB: 5,58%**
  - **Sulfur – 0.44 phr**
  - **DTDC – 2.11 phr**
  - **MBT – 1.00 phr**
  - **TiBTD – 1.50 phr**
  - **ZdiBC – 1.50 phr**
  - **DTP – 1.50 phr**



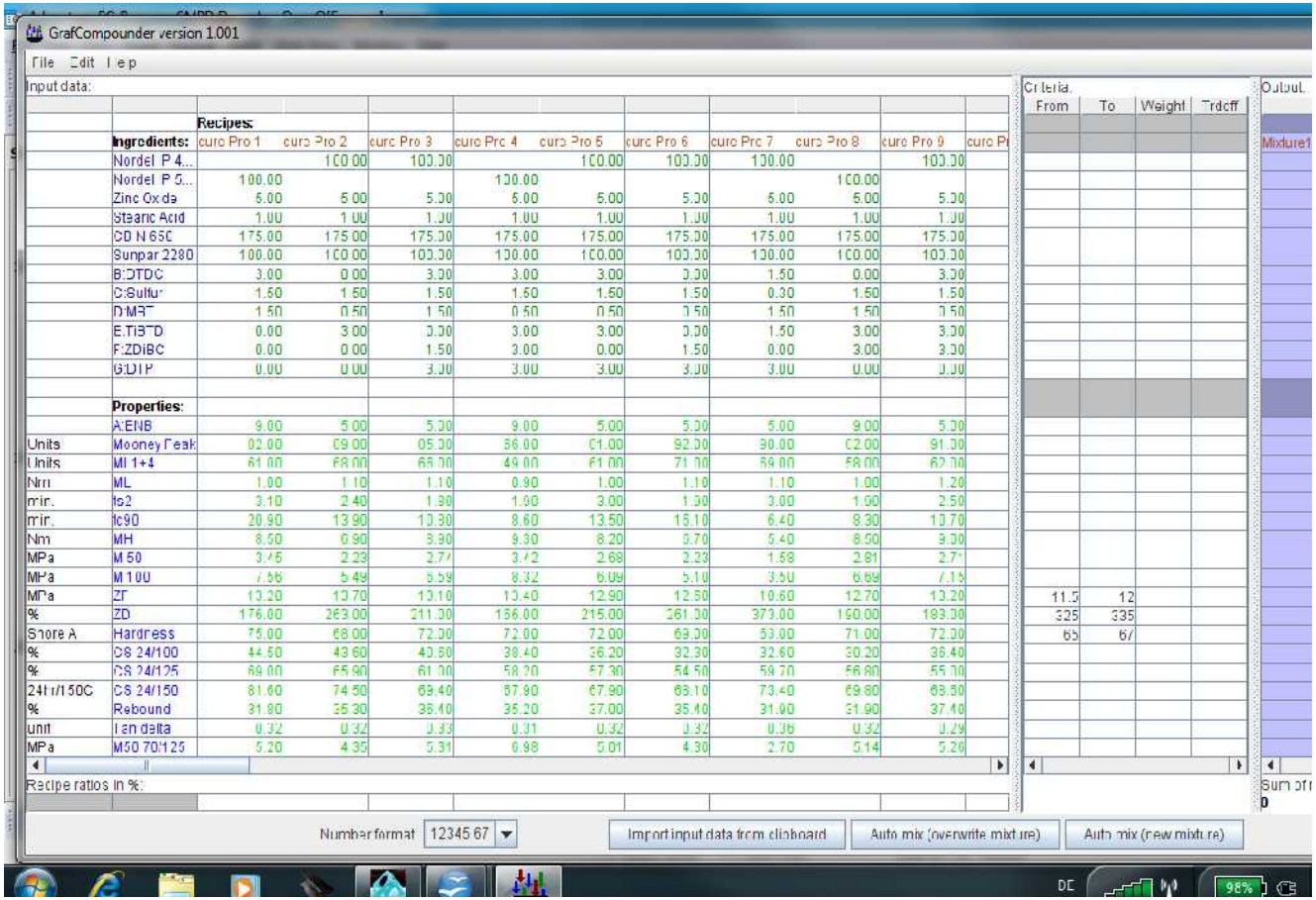
DoE Analysis and Result

- Analysis with point prediction results:
  - ZF 11.2 MPa
  - ZD 335 %
  - Hardness 66.5°ShA

Factor	Name	Level
A	ENB	5.58
B	DTDC	2.11
C	Sulfur	0.44
D	MBT	1.00
E	TiBTD	1.50
F	ZDiBC	1.50
G	DTP	1.50

Analysis with GrafCompounder

- **Paste table into Graf Compounder**
  - **Select boundaries**



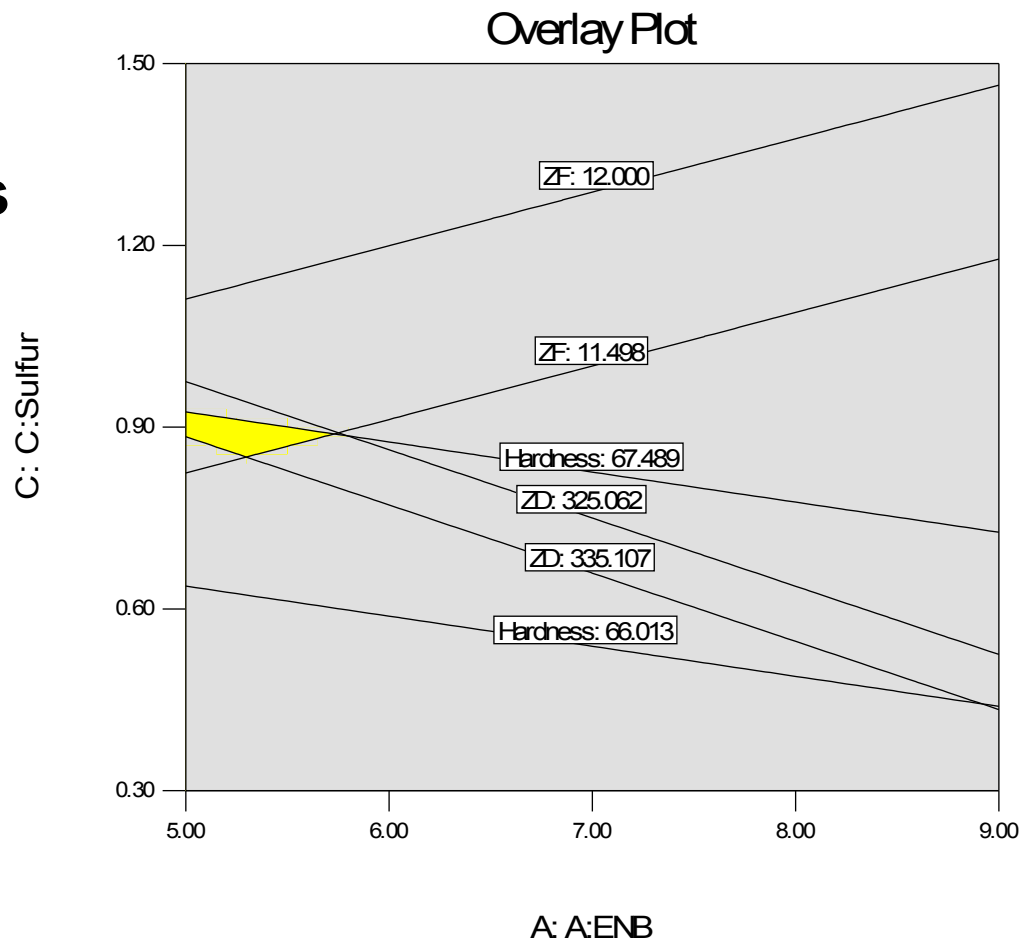
Analysis with GrafCompounder

- **Paste table into GrafCompounder**
  - **Select boundaries**
  - **ZF-MPa : 11.5-12.0**
  - **ZD-% : 325-335**
  - **H-°ShA : 65-67**

Ingredients	Result
A: ENB	**6.5**
B:DTDC	**0.98**
C:Sulfur	**0.93**
D:MBT	**1**
E:TiBTD	**1.51**
F:ZDiBC	**1.33**
G:DTP	**1.45**
ZF	**11.5**
ZD	**325**
Hardness	**67**

Analysis with Design Expert®

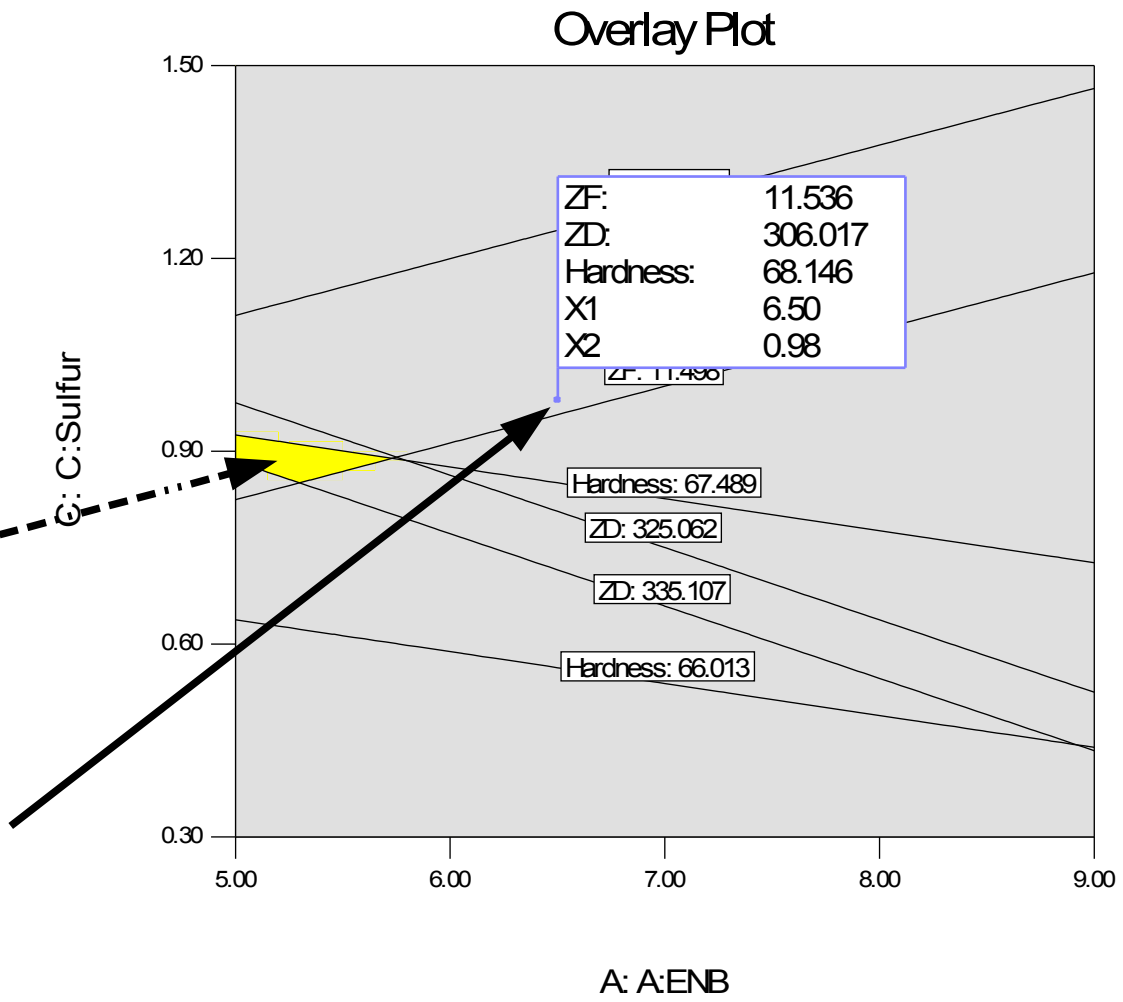
- **Run Optimization Graphical**
  - **Select same boundaries**
  - **ZF-MPa : 11.5-12.0**
  - **ZD-% : 325-335**
  - **H-°ShA : 65-67**



Compare Result
Design Expert® vs GrafCompounder

- **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.**



Analysis with GrafCompounder

- **Boundary Conditions**
  - **Select boundaries**
  - **ZF-MPa : 11.5-12.0**
  - **ZD-% : 325-335**
  - **H-°ShA : 65-67**

Ingredients	Result GrafCompounder	Result 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**

+) Note: Accelerators are preset!

Conclusion

- Compounds in databases are type of happen stance data
  - Which can not analyzed with a systematic approach today
  - DoE in each case needs data based on a planned experiment.
- GrafCompounder allows to search a database for a possible solution using targets
  - At minimum you get an very good idea about the center point in a DoE