GrafCompounder

Software Application
in
Rubber Compounding

Dr. Hans-Joachim Graf

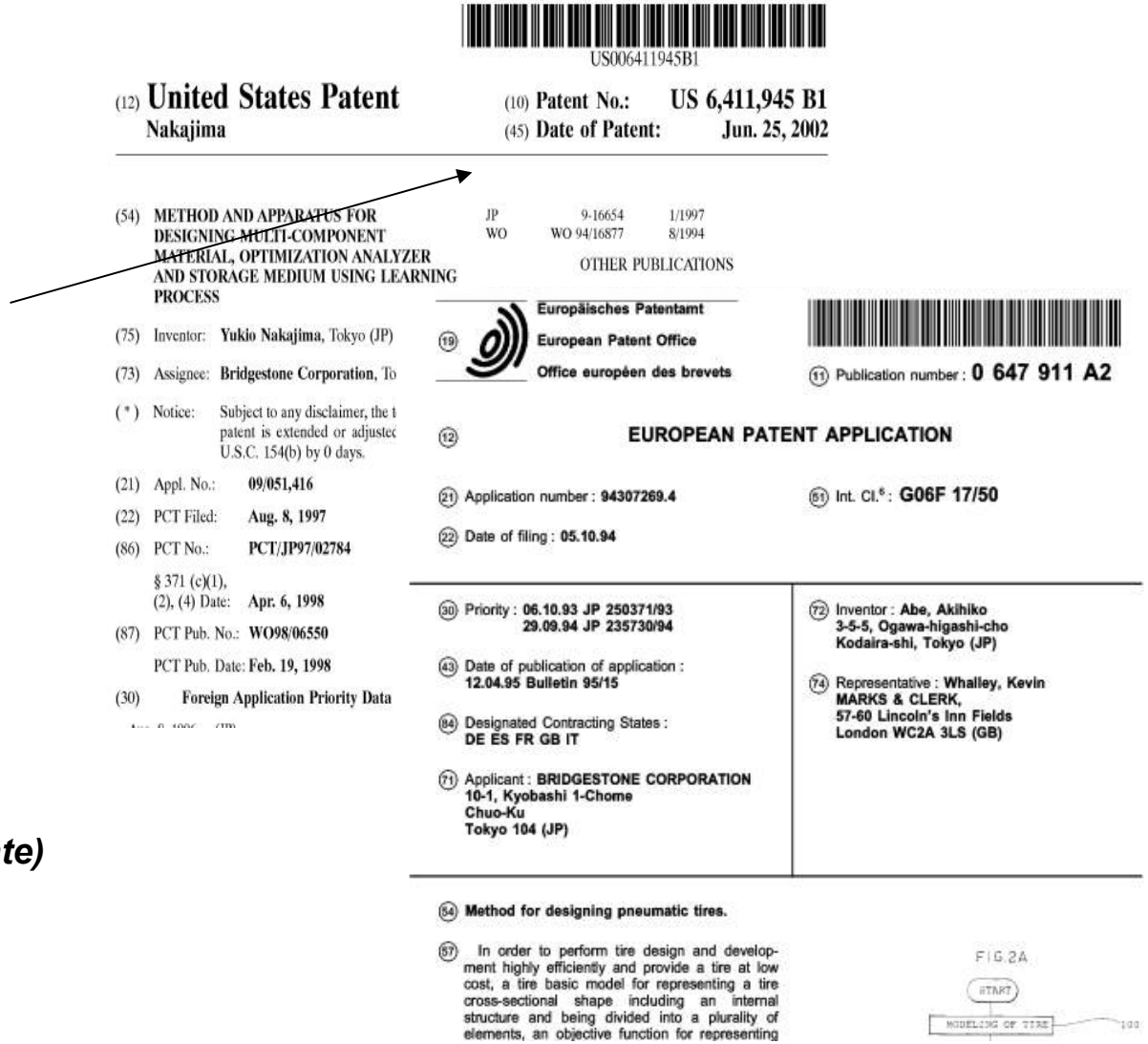
HJG

Content of this presentation

1. Introduction
2. Program idea
3. Justification of calculation method
4. Comparison with Statistic Experimental Design (DoE)
   - Filler / Oil Design
   - Accelerator Design
   - DoE Simulation
5. Conclusion

Computer Aided Compound Development

- **Bridgestone Patent 1994**
  Inventor: Akihiko Abe
- **Bridgestone Patent 2002**
  Inventor: Yukio Nakajima
- ***Colour Matching***
  Patents from
  BASF, CyanAmid, DuPONT
- ***Empirical DoE Patent:***
  Honeywell
- ***Recipe Libary Search and***
  Comparison
  CombiChem, GE, Hunt (Private)



US006411945B1

(12) **United States Patent**
Nakajima

(10) **Patent No.: US 6,411,945 B1**
(45) **Date of Patent: Jun. 25, 2002**

(54) METHOD AND APPARATUS FOR DESIGNING MULTI-COMPONENT MATERIAL, OPTIMIZATION ANALYZER AND STORAGE MEDIUM USING LEARNING PROCESS

(75) Inventor: Yukio Nakajima, Tokyo (JP)

(73) Assignee: Bridgestone Corporation, To

(*) Notice: Subject to any disclaimer, the t patent is extended or adjustec U.S.C. 154(b) by 0 days.

(21) Appl. No.: 09/051,416

(22) PCT Filed: Aug. 8, 1997

(86) PCT No.: PCT/JP97/02784

§ 371 (c)(1),
(2), (4) Date: Apr. 6, 1998

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PCT Pub. Date: Feb. 19, 1998

(30) Foreign Application Priority Data

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WO	WO 94/16877	8/1994

OTHER PUBLICATIONS

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European Patent Office
Office européen des brevets

(11) Publication number : **0 647 911 A2**

(12) **EUROPEAN PATENT APPLICATION**

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(84) Designated Contracting States :
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(54) **Method for designing pneumatic tires.**

(57) 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 cross-sectional shape including an internal structure and being divided into a plurality of elements, an objective function for representing

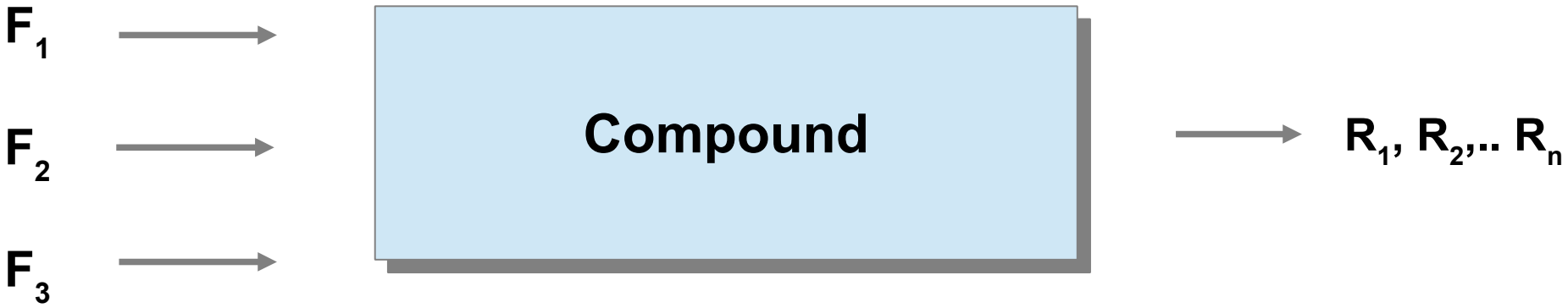
FIG.2A

START

MODELING OF TIRE

Influences:
Factors: Ingredients

Effects:
Responses: Properties



Objective of an DoE should be the identification of the most important factors ($F_{1,}..F_n$) on measurable effects (Responses $R_{1,}...R_n$) and to describe there dependency in a mathematical equation:

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

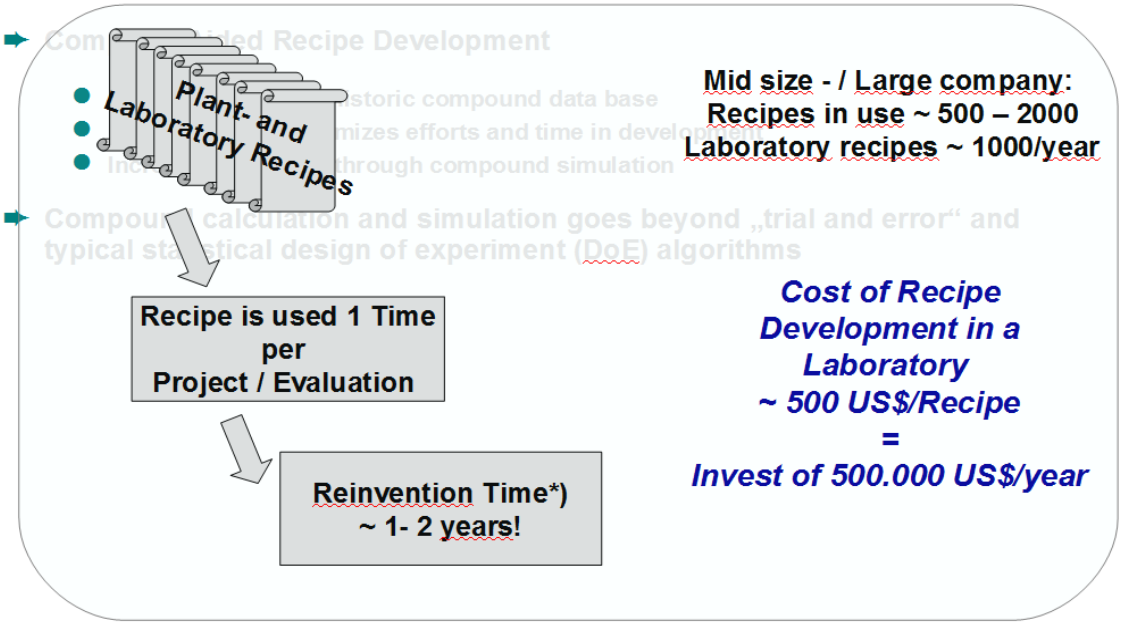
Why Computer Aided Recipe Development ?

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

No algorithm describing the relation between ingredients and properties

It gives a better start for a typical statistical "*design of experiment*" (DoE) approach.

Compound calculation and simulation should utilize Compound history, but not in a „trial and error" fashion.



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

Database created with Statistic Experimental Design (DoE)

- Organized / limited size
- Variation of few factors according DoE
- Optimization, numerical and graphical / prediction Tool available in the software

CARD [Computer Aided Recipe Development] with GrafCompounder - Historically created Database

- **Unorganized / Unlimited**
- **Multiple factor variation**
- **Prediction according specification**

Justification of calculation method with linear dependencies:

- **If the majority of factor / response relations are linear the MLI – method gives sufficient accurate results inside 95% confidence interval !**

Line call out:

- SEA J200: AA/BA/CA – NR, SBR, EPDM...and other Material
- SAE J200 M4 AA621 A13 B13 F17

AA 610 Suffix 2	Rubber Hardness Tensile Elongation	NR 60°ShA 21 Mpa 350%
A13	Heat Aging Hardness Change Change Tensile Chande Elongation	70h / 70°C + 15 °ShA ∓ 30 % - 50%
B13	C-Set (22h/70)	< 25%
F17	Low Temperature Res. Non Brittle (3Min)	- 40 °C pass

Properties of MB is determined by Polymer, CB and Oil content and the ratio or CB and Oil.

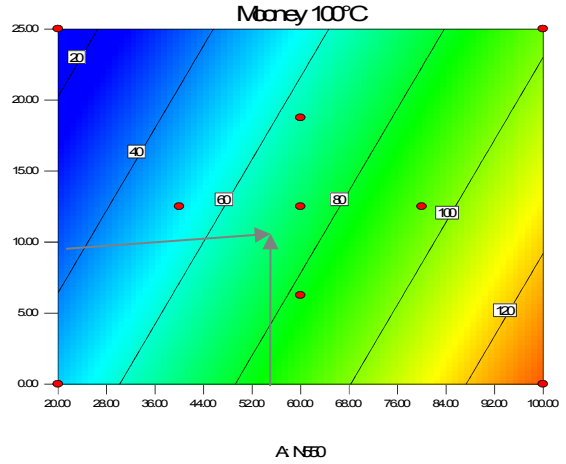
- Unit 2 as a reference (based on Cabot TG RG-135
- …

- CB 550: 55 phr
- Oil: 10 phr

- Mooney Viscosity: 71 M-Units
- Hardness: 60 °ShA
- Tensile: 21 MPa
- Elongation: 460 %
- C-Set: 28%

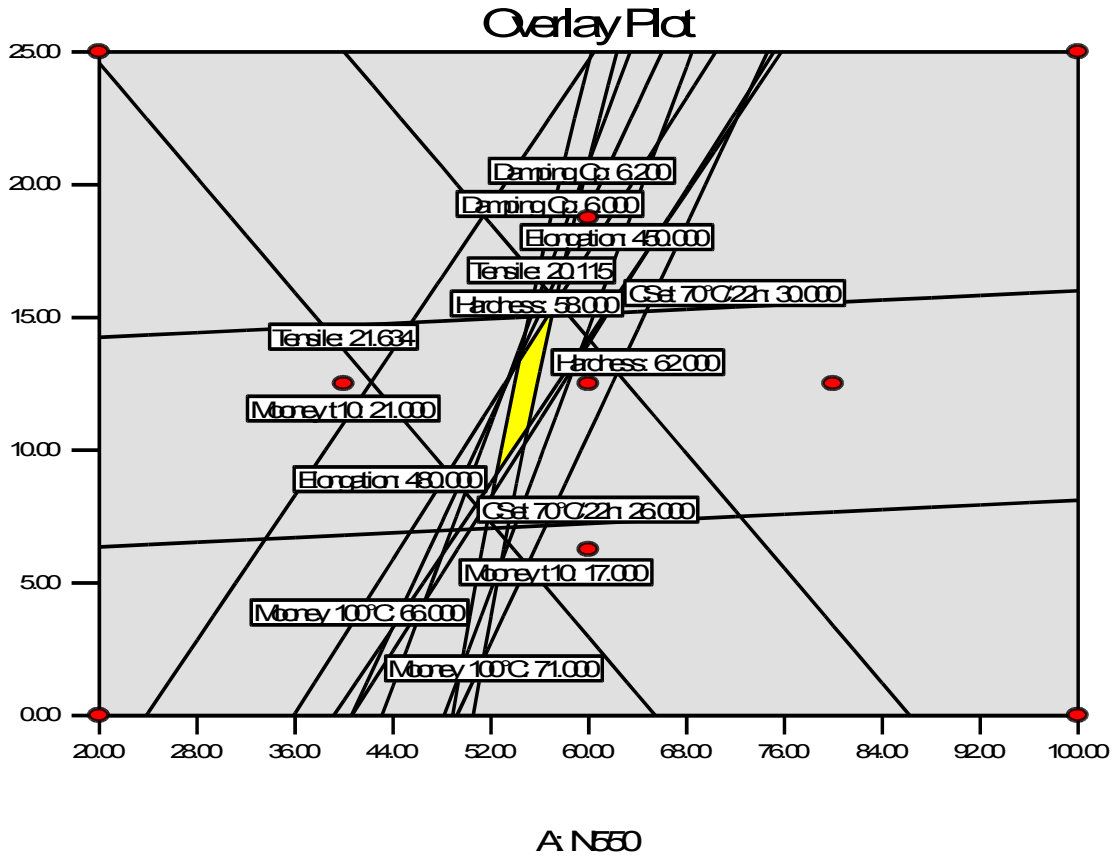
NR Compound

- *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*

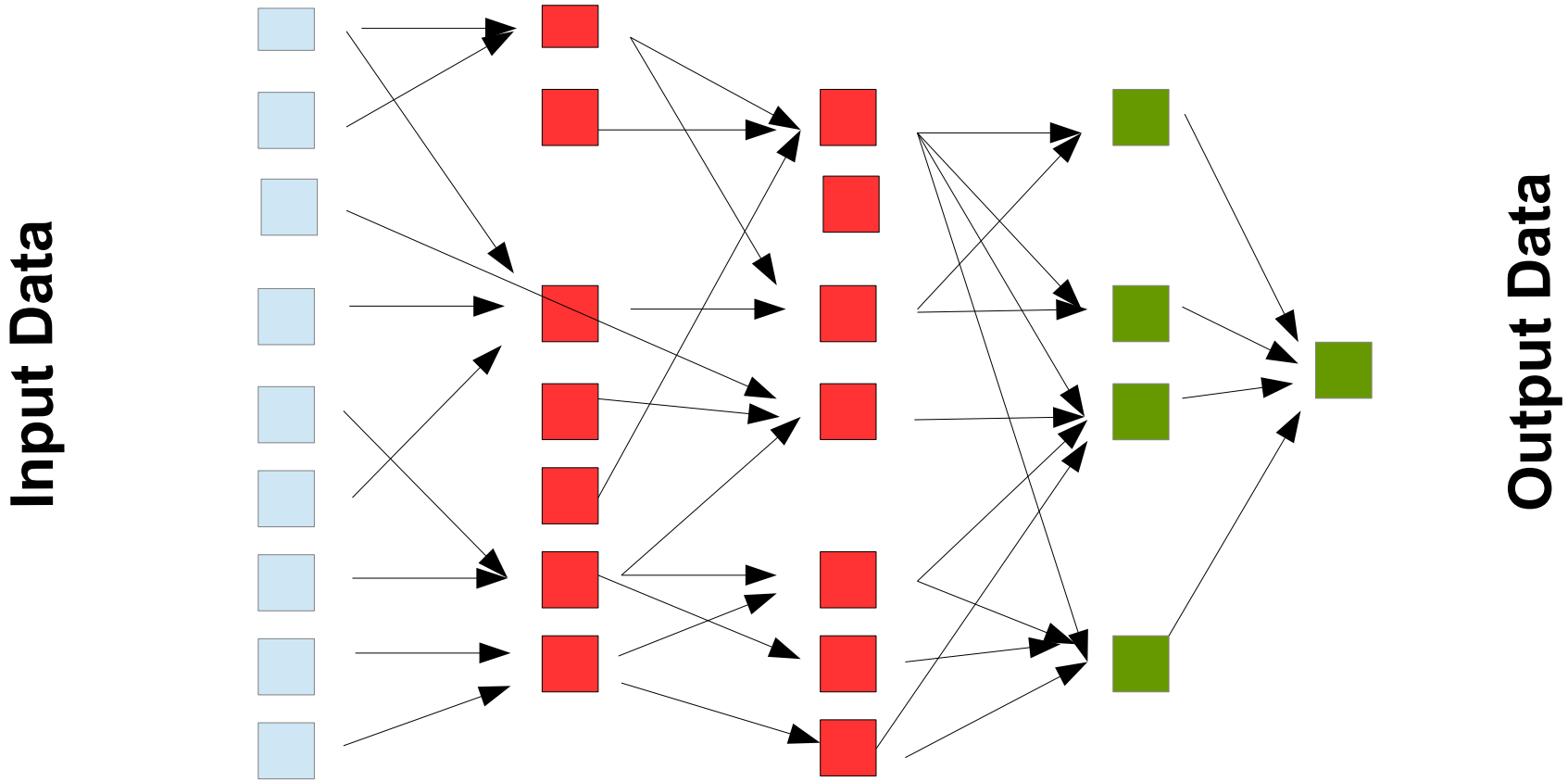


Properties of MB is determined by Polymer, CB and Oil content and the ratio or CB and Oil.

- **Unit 2 as a reference (based on Cabot TG RG-135)**
- **…**
- **CB 550: 55 phr**
- **Oil: 10 phr**
- **Mooney Viscosity: 66 – 71 M-Units**
- **Hardness: 58 – 62 °ShA**
- **Tensile: 20 – 22 MPa**
- **Elongation: 450 – 480 %**
- **C-Set: 26 – 30%**

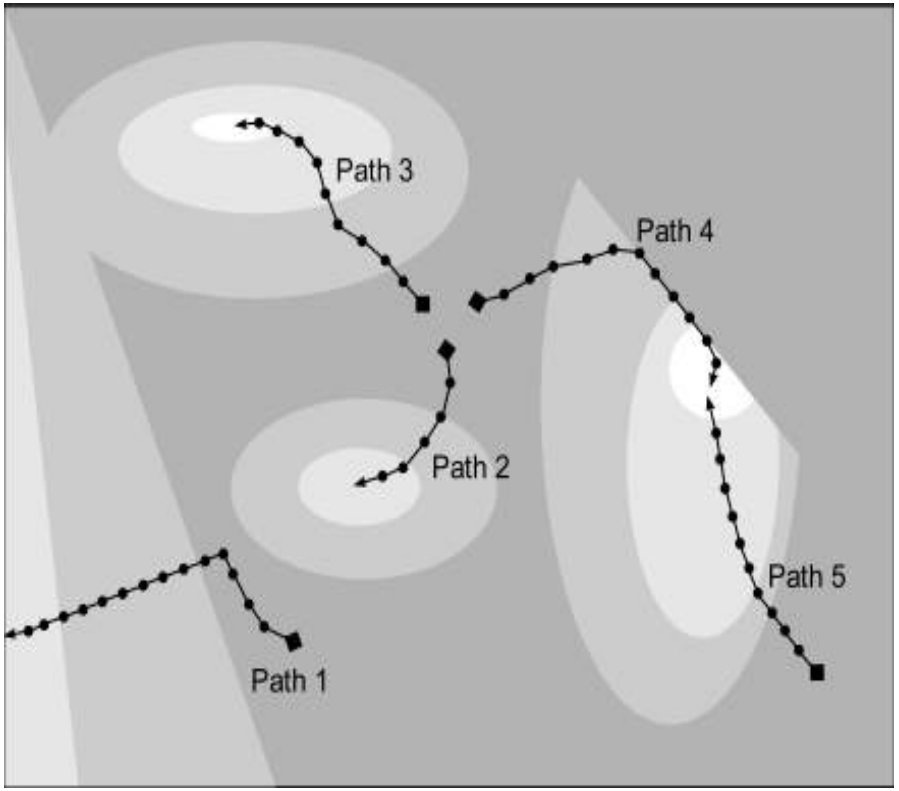


Original Data **1. Iteration Level, n^{th} - Iteration Level,** **Result**



Linear Relation between: Ingredient – Property.
Calculation of compounds with linear algorithm.
Approximisation to target(s) via multiple iteration

- **The GrafCompounder uses the Multiple Linear Iteration method [MLI] to calculate a new recipe according to properties targeted**
- **The GrafCompounder enables the user to analyze and improve their compound database via identification of faulty data sets**
- **Each compound taken into account for the calculation and its influence of each on the final result is visualized.**
  - **Its contribution is given as a ratio**
- **The GrafCompounder is a fast and easy to use tool without utilizing a complex "hidden" mathematical and analytical method**
- **The GrafCompounder works with smaller and larger Databases**



Calculation method confirmation

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

Example 1.

DoE with 4 Factors

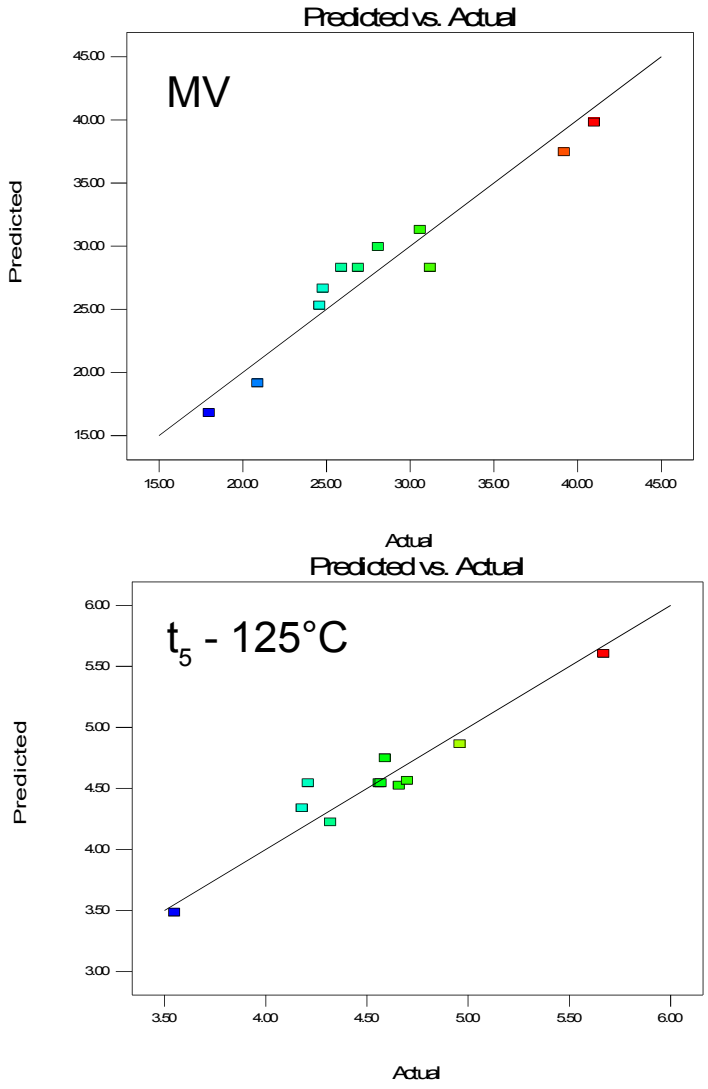
Polymer used: EPDM (Vistalon 8600)

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!**

Rheology Data are examined

- **MV and t_5 - 125°C can be measured quite accurate.**
- **Both are significant with a linear model equation**

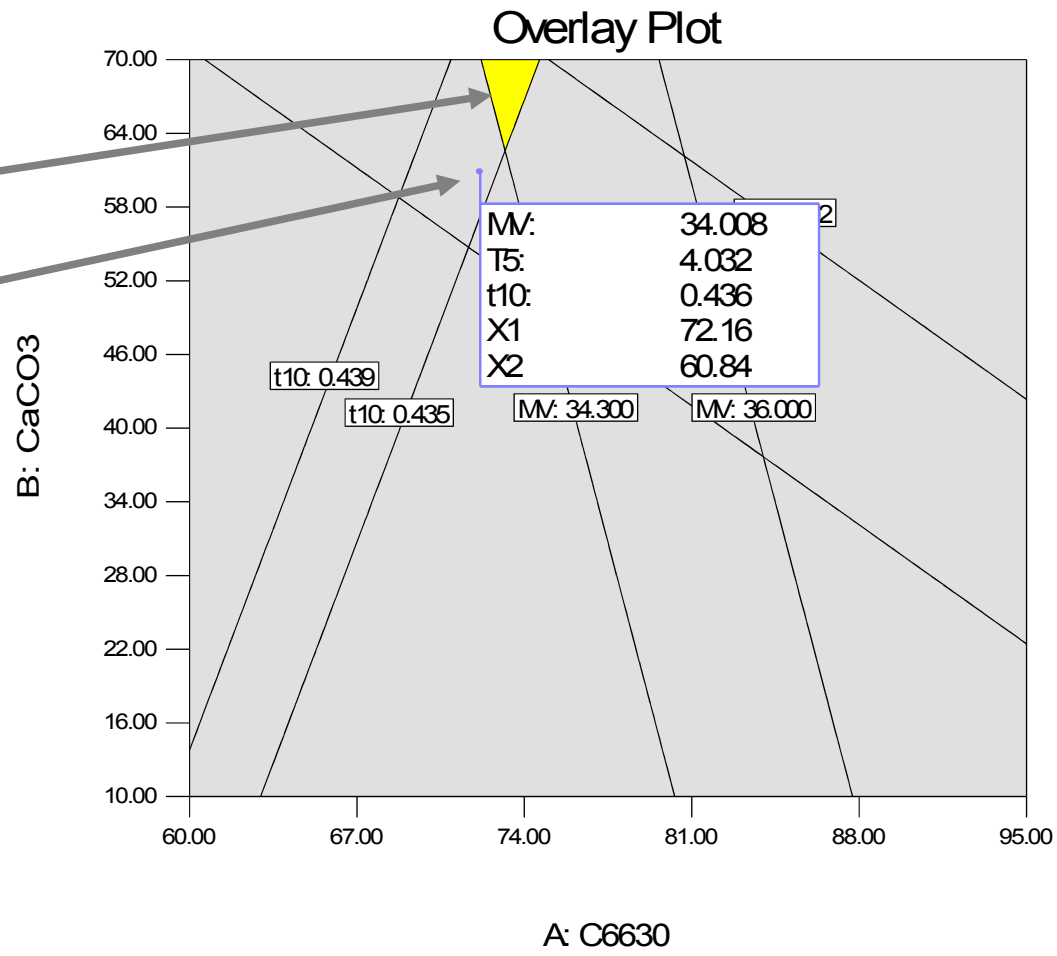


Optimization area

calculated with DoE Software: Design Expert®

calculated with GrafCompounder

- ***boundary condition (CC 6630 – 73 phr)***



Example 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

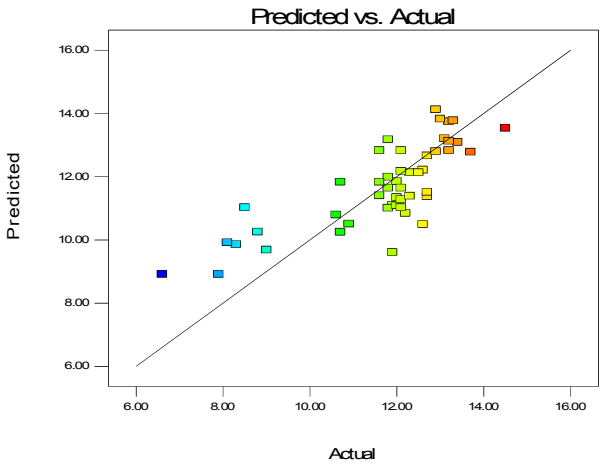
- **Sulfur has larger influence followed by DTDC and TiBTD, but negative**

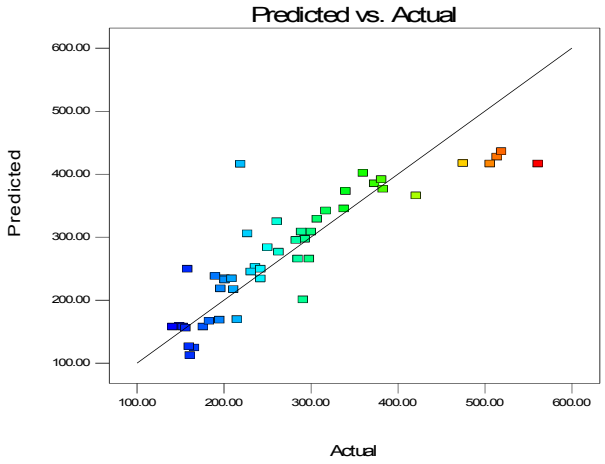
Elongation is significant with quadratic model, but linear model is a more than sufficient fit

- **Sulfur has the largest influence followed by DTDC**

Hardness is sufficient significant with linear model as well

- **Main influence Sulfur, DTDC**



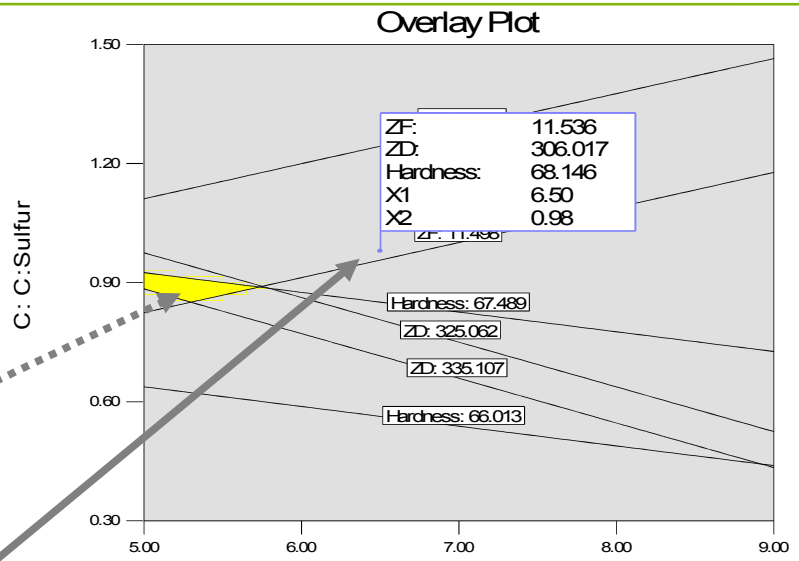


Boundary Conditions

- **Select boundaries**
- **TB-MPa: 11.5 - 12.0**
- **EB-% : 325 - 335**
- **H-°ShA: 65 - 67**

The Design Expert® optimization graph shows the location of the result as a yellow area.

GrafCompounder result is tagged with a flag.



	Calculation Method	
Ingredients	GrafCompounder	Design Expert®
A: 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
E: DTP	1.45	1.44
Tensile @Break	11.5	11.5
Elongation @Break	306	330
Hardness	68	67

- **Simulation of a DoE**
- **Experiments made in the Laboratory**
  - **NR based Compound** — **LL** — **UL**
    - **Filler 1: CB 336** — **30 phr** — **70 phr**
    - **Filler 2: CB 550** — **0 phr** — **20 phr**
    - **Oil: Naphtenic Oil** — **5 phr** — **45 phr**
  - **Type of DoE: fractoinal factorial**
  - **Software: Design Expert®**
- **Calculation made with GrafCompounder**
  - **NR Formula index from MRPRA**
  - **Formula data adjusted, but responses taken as is.**

Example 3

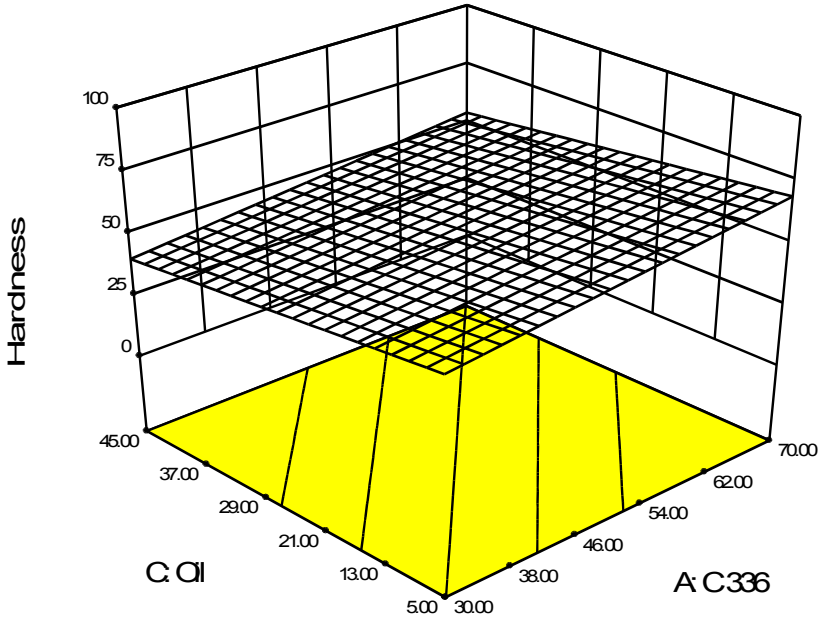
★ **For comparison: Hardness, Tensile - / Elongation at break**

Hardness:

X1 – A: CB 336

X2 = C: Naphtenic oil

B: CB 550 = 10.00 phr

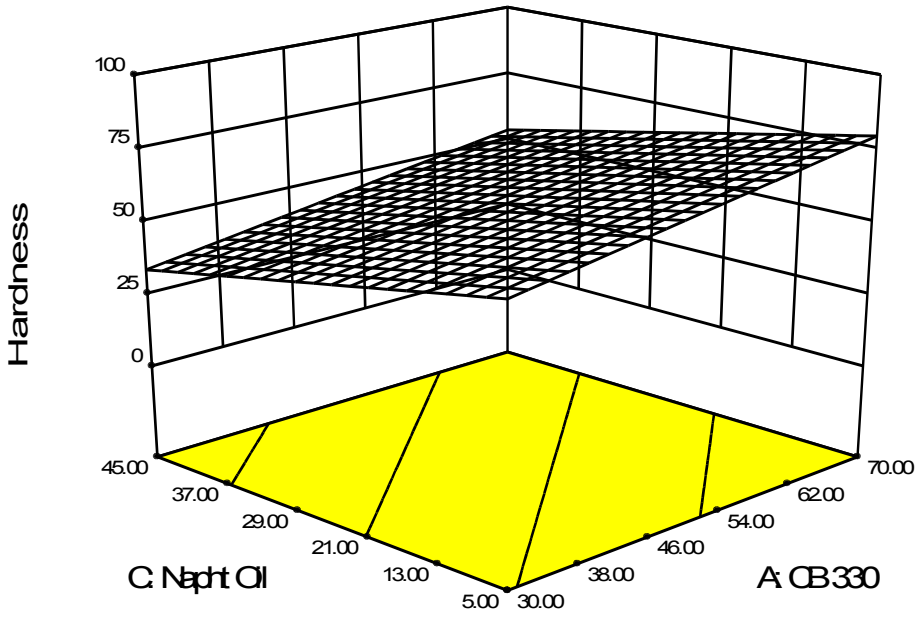


Hardness Simulation

X1 – A: CB 330

X2 = C: Naphtenic oil

B: CB 550 = 10.00 phr

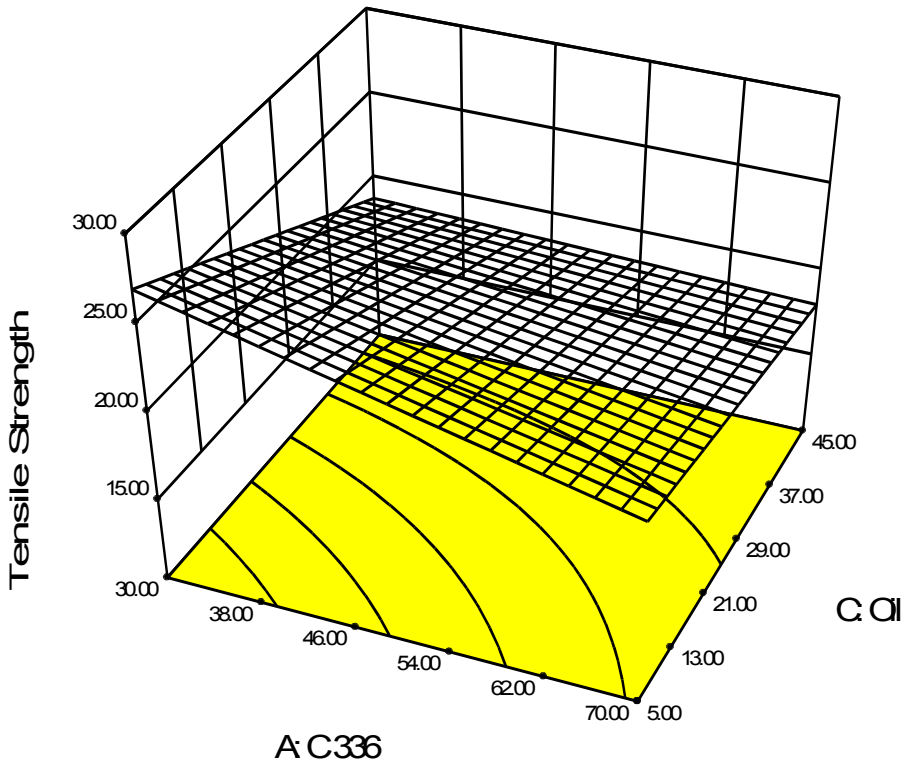


Tensile at break:

X1 – A: CB 336

X2 = C: Naphtenic oil

B: CB 550 = 10.00 phr

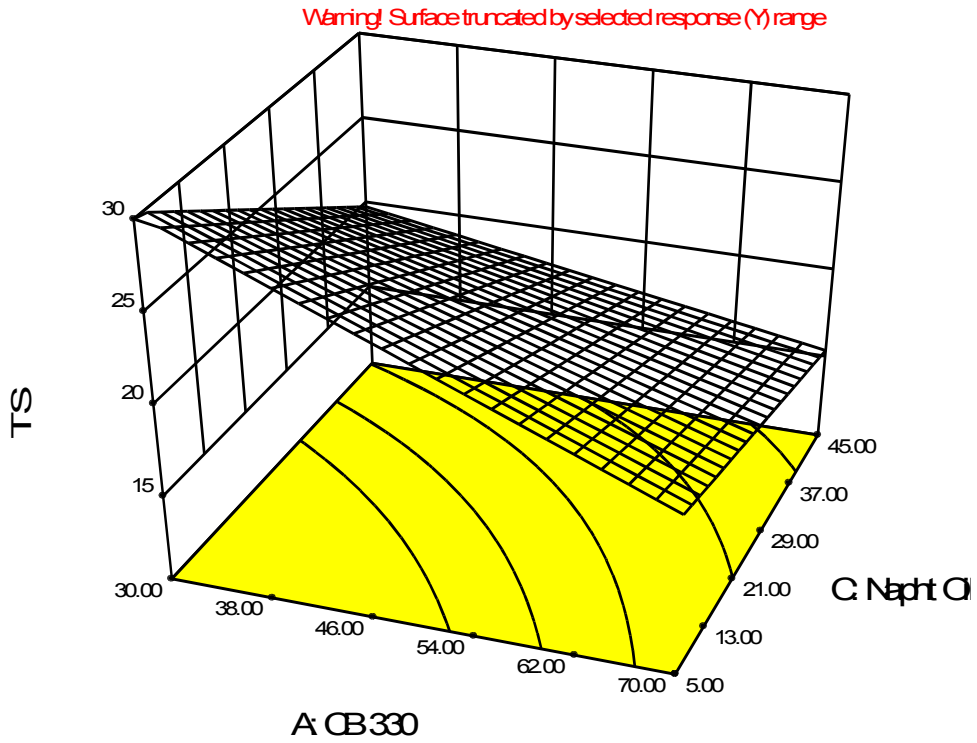


Tensile at break Simulation

X1 – A: CB 330

X2 = C: Naphtenic oil

B: CB 550 = 10.00 phr

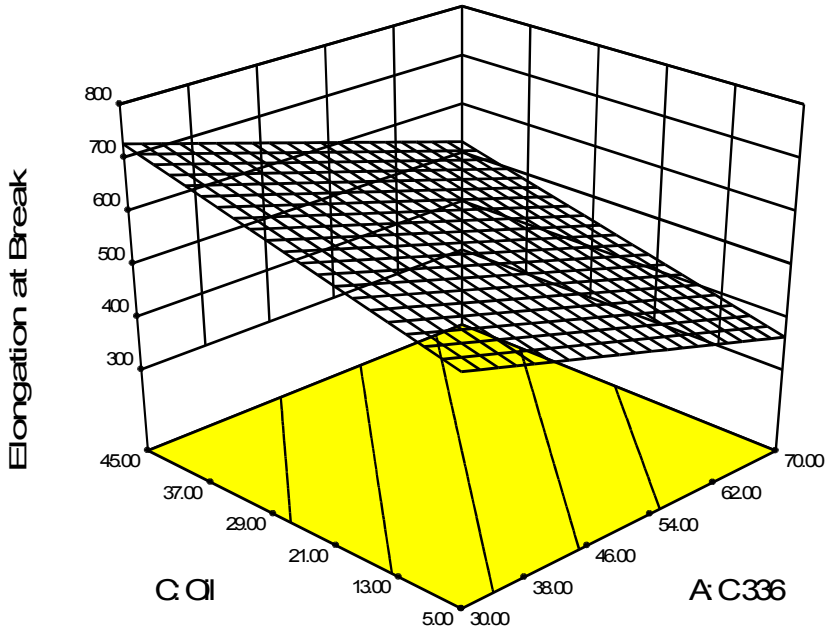


Elongation at break:

X1 – A: CB 336

X2 = C: Naphtenic oil

B: CB 550 = 10.00 phr

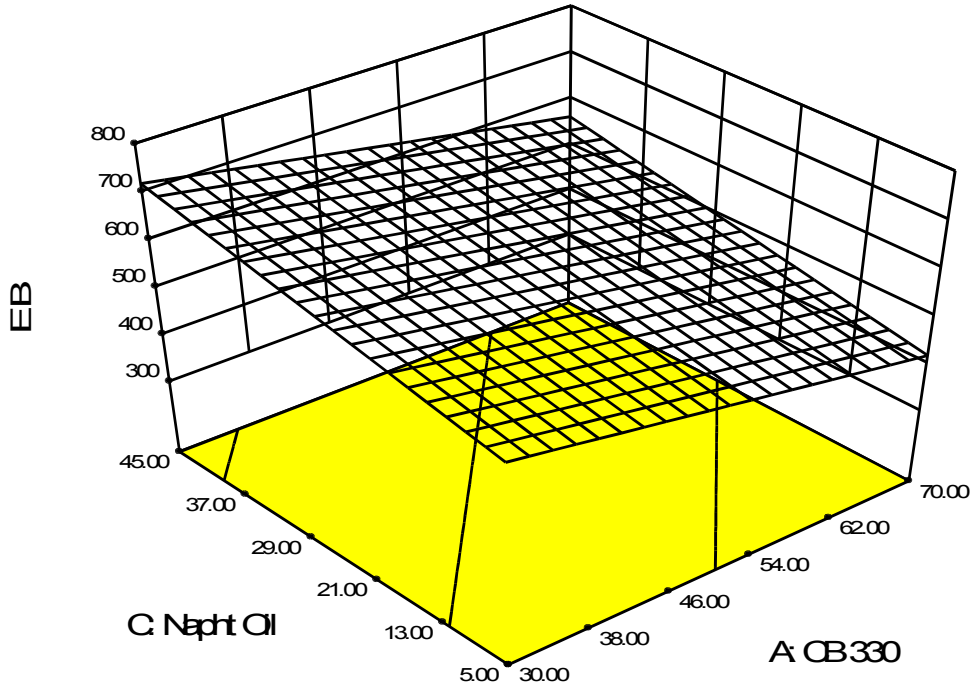


Elongation at break Simulation

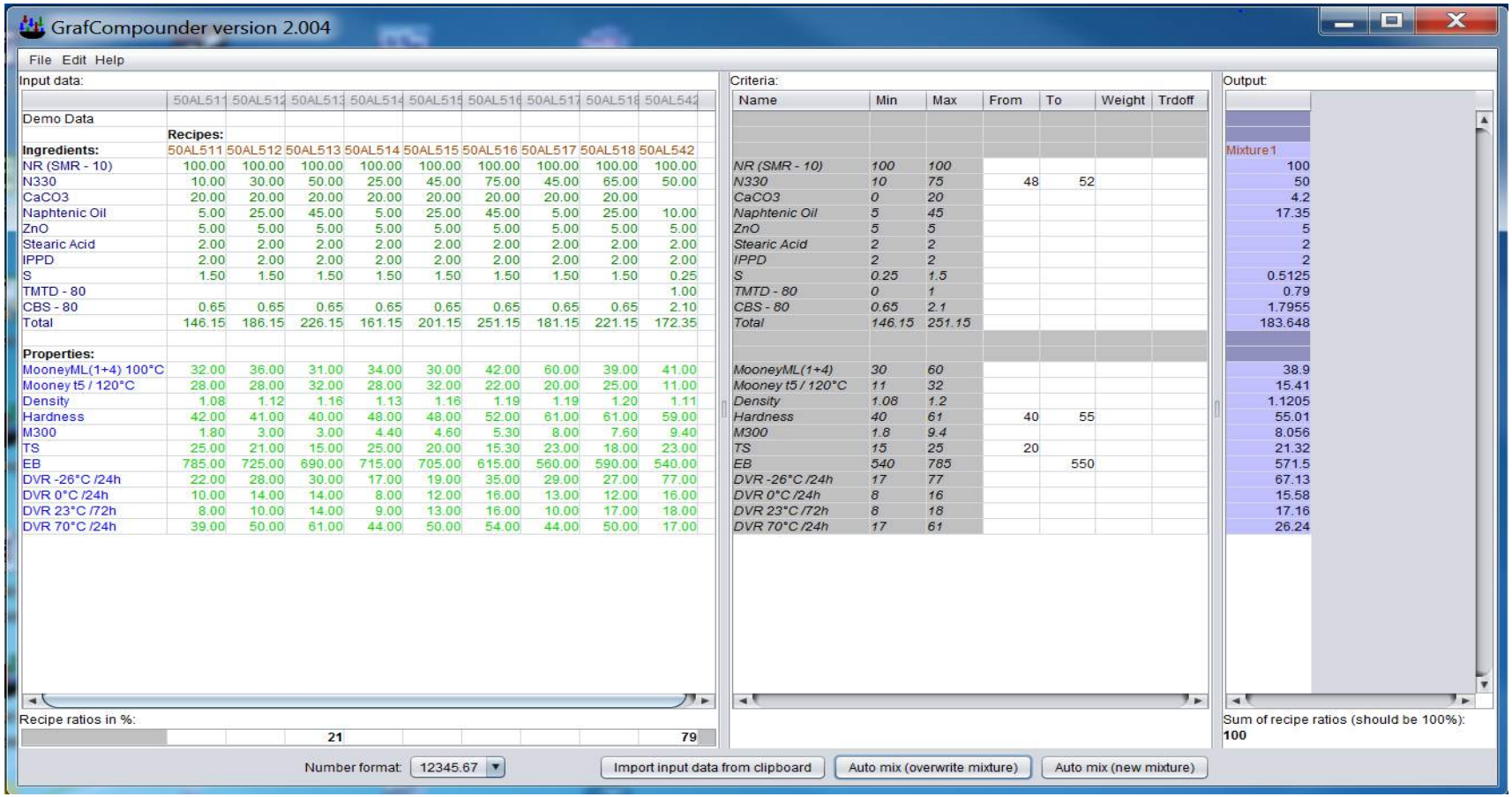
X1 – A: CB 330

X2 = C: Naphtenic oil

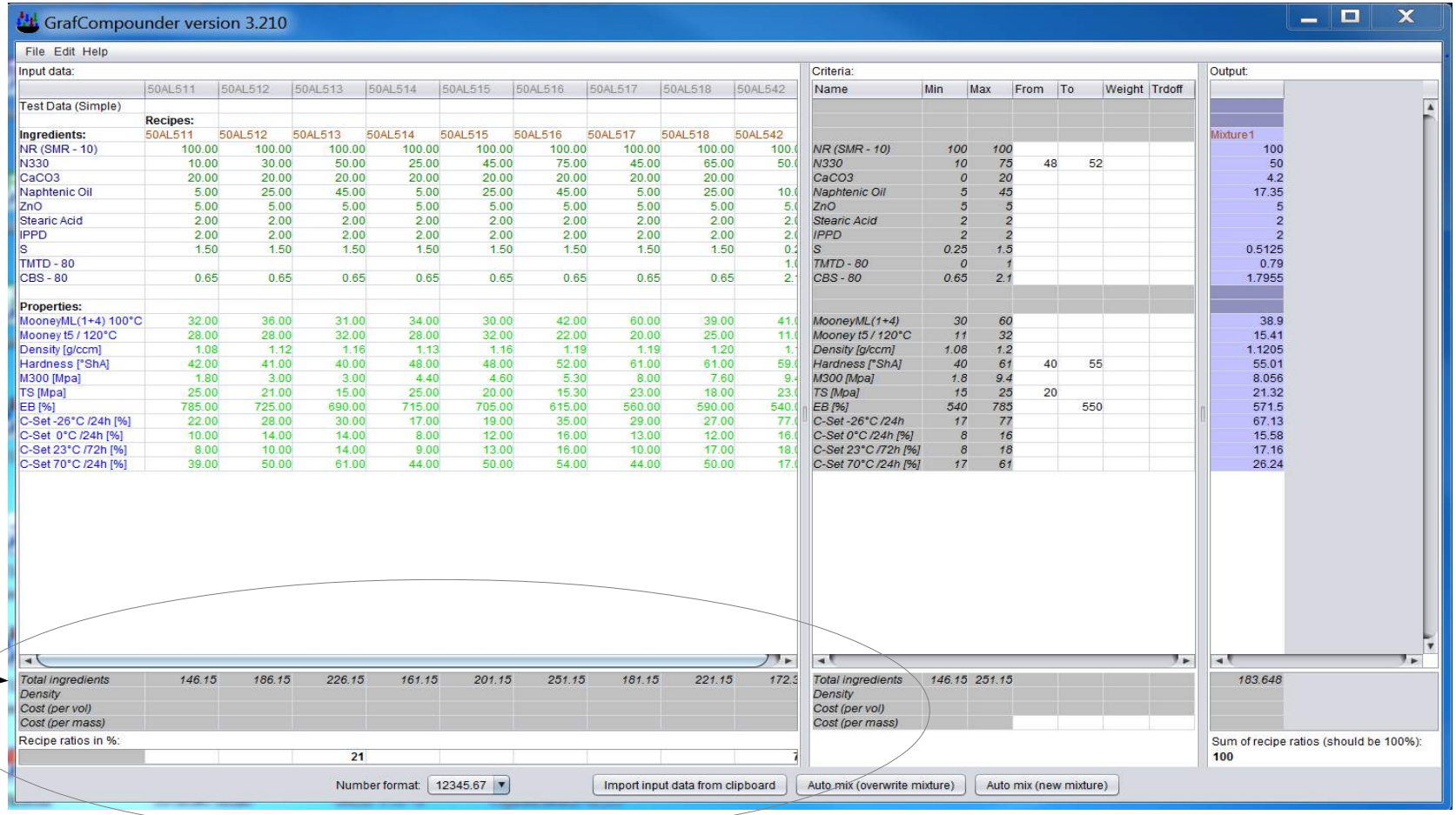
B: CB 550 = 10.00 phr



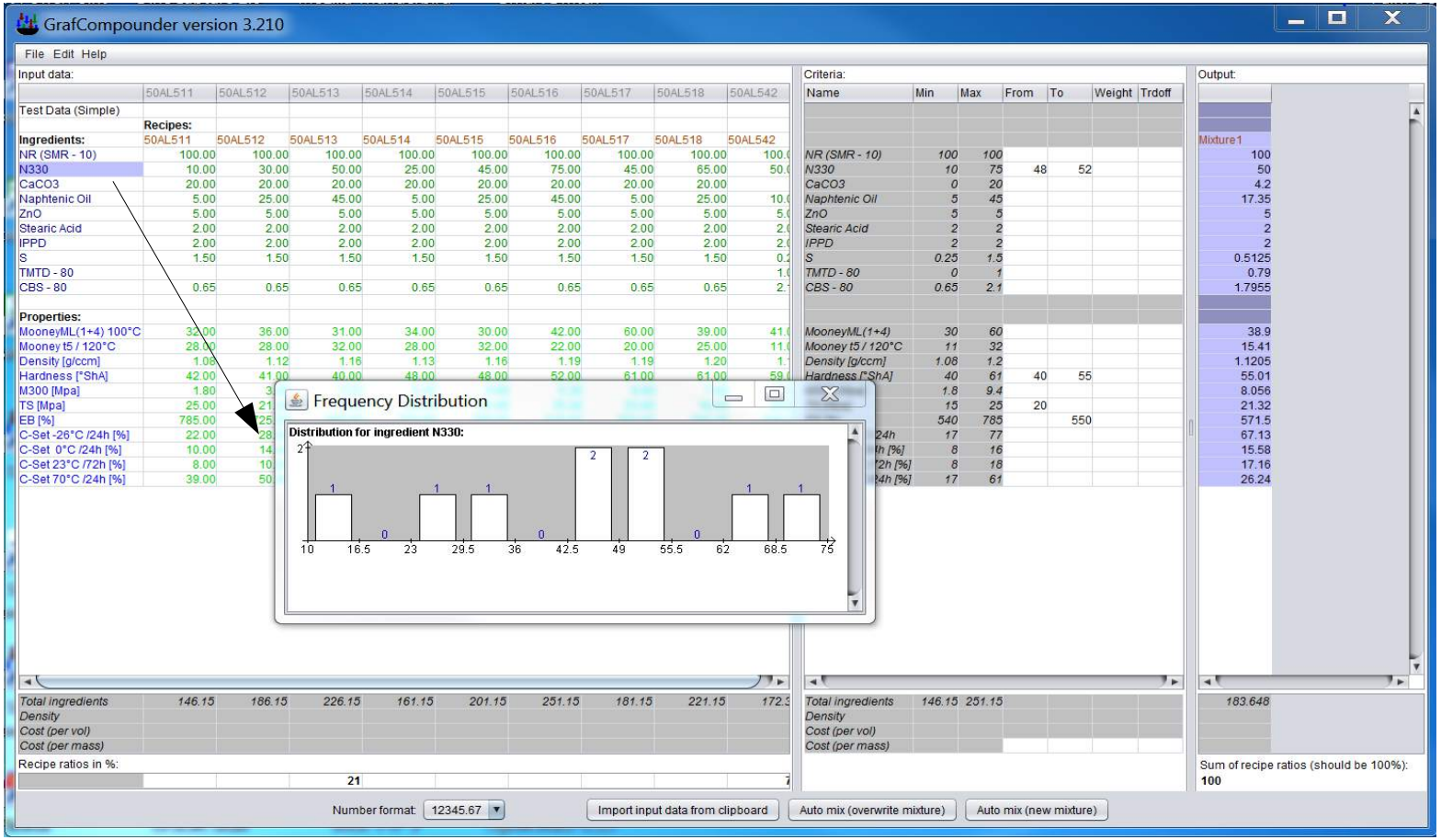
Screenshot of GrafCompounder V 2.004 with demo data, targets and a calculated compound



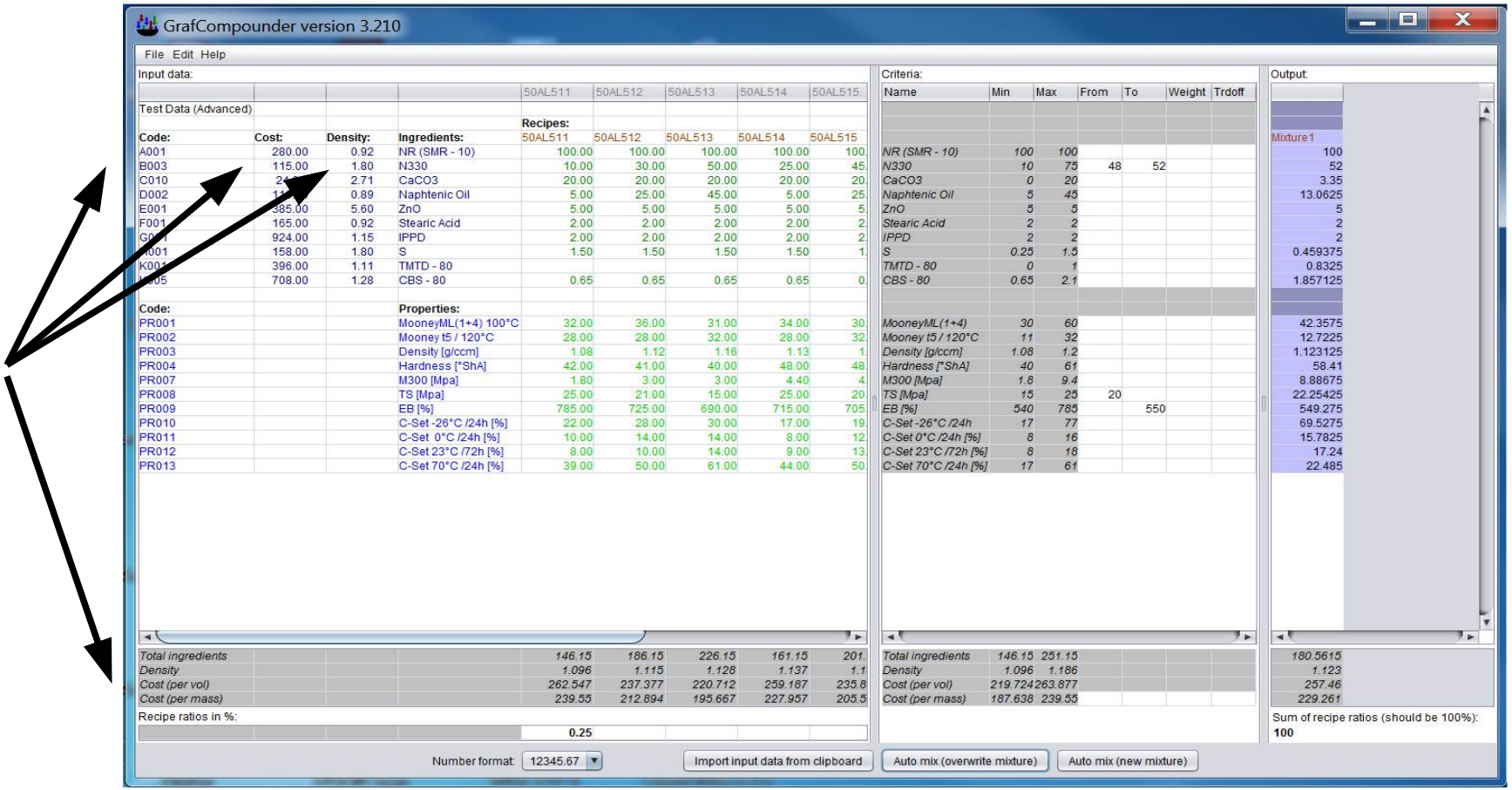
Screenshot of GrafCompounder V 3.210 with demo data, targets and a calculated compound



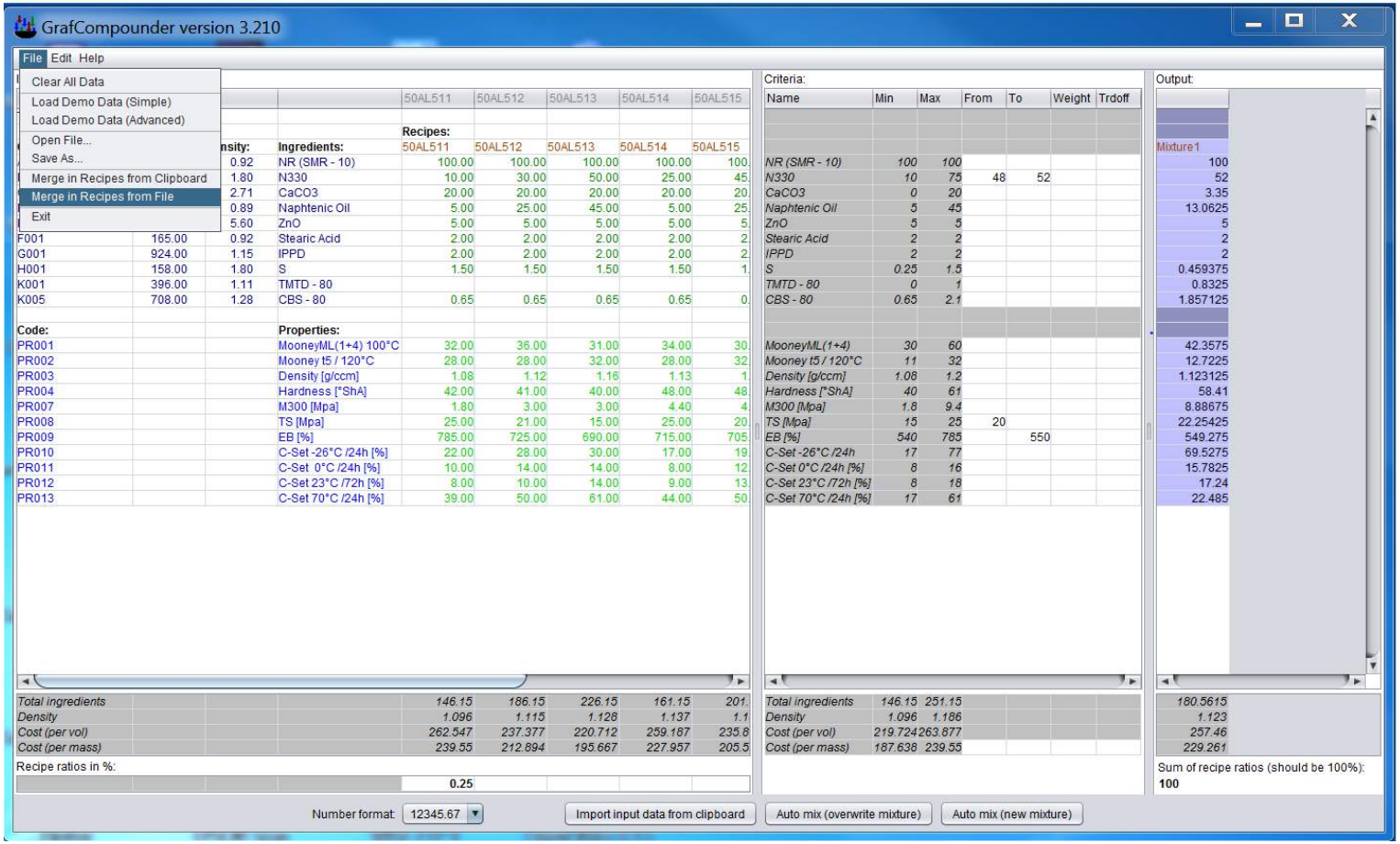
Screenshot of GrafCompounder V 3.210 with demo data, targets and a calculated compound



Screenshot of GrafCompounder V 3.210 with demo data, targets and a calculated compound



Screenshot of GrafCompounder V 3.210 with demo data, targets and a calculated compound



Recipe manager

- **Creation of a formula according predefined criteria**
  - **Ingredients**
  - **Properties**
  - **Cost**
- **Trace ability back to the starting formulas**
  - **Analysis of outliers and their correction or elimination in the database is possible.**
  - **Integration of results from statistical experimental designs with merge function.**
  - **Integration of databases of different origin, provided that an export of the data is possible with table calculation programs.**

Result of the calculations MUST be confirmed by an experiment.

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

- **Examples show:**
- **The resulting formulas calculated correspond to the general rules of compounding**
  - **Differences with calculations based on regression obtained with DoE is marginal**
- **The formulas will show property scores larger than the 90 % – 95 % confidence interval in confirmation experiment**

- **Only one confirmation experiment would be needed as opposed to multiple trials in case of development targets.**
  - **Starting formula calculated with GrafCompounder**
  - **Optimized formula with Optimization Tool in DoE Software**

More information under: **www.grafcompounder.com**

- **Release of the „GrafCompounder“ Version 3.210**

Thank you for joining this presentation.

- **Any question, comment?**

More information under: **www.grafcompounder.com**