

Formula development

**A methodical formula development increases
technological and economic advantages**

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Formula and recipe design

- ***A formula does not yield absolutely a good product .***
- ***But no good product without formula***

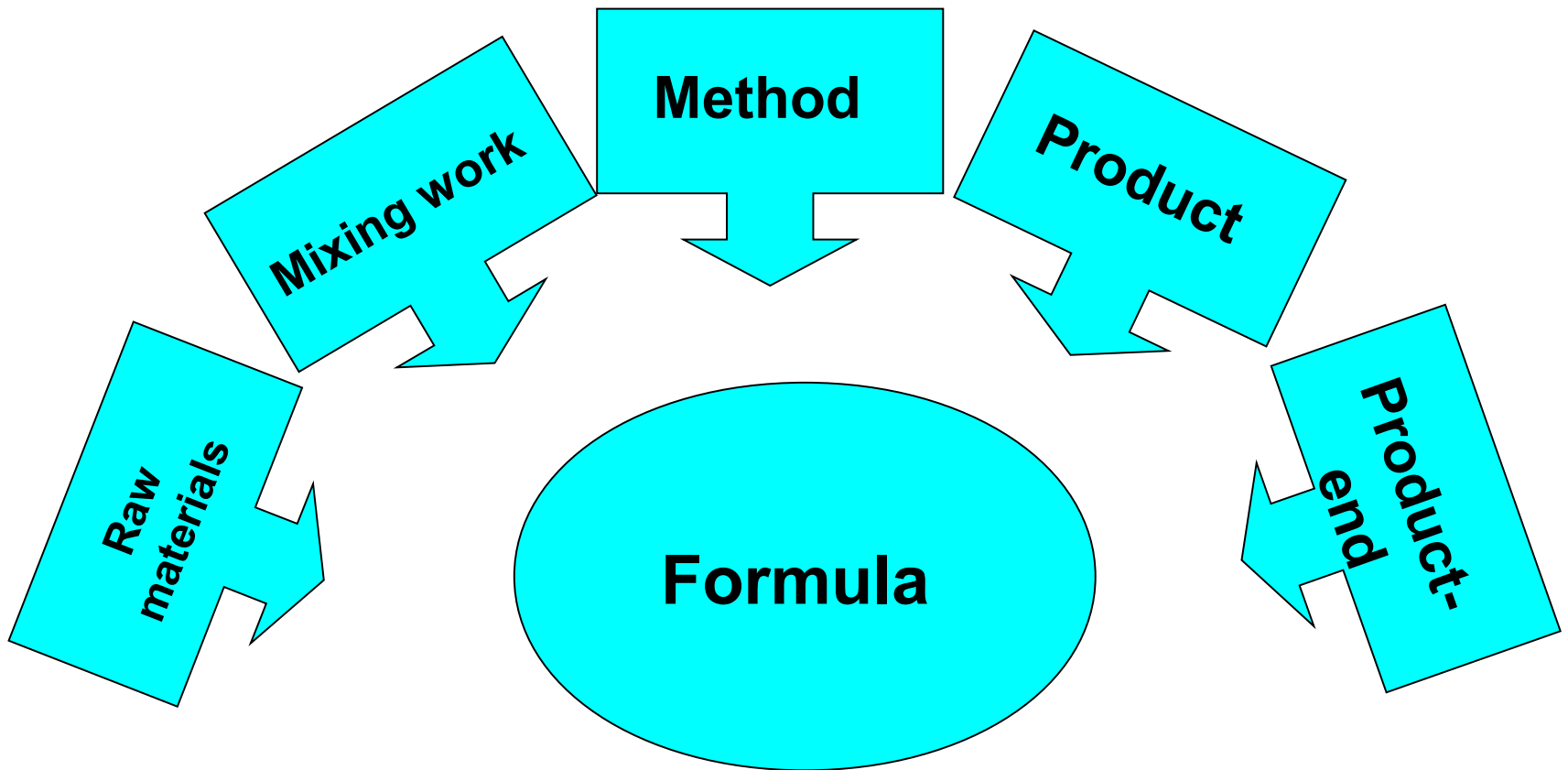


Aim of a recipe development

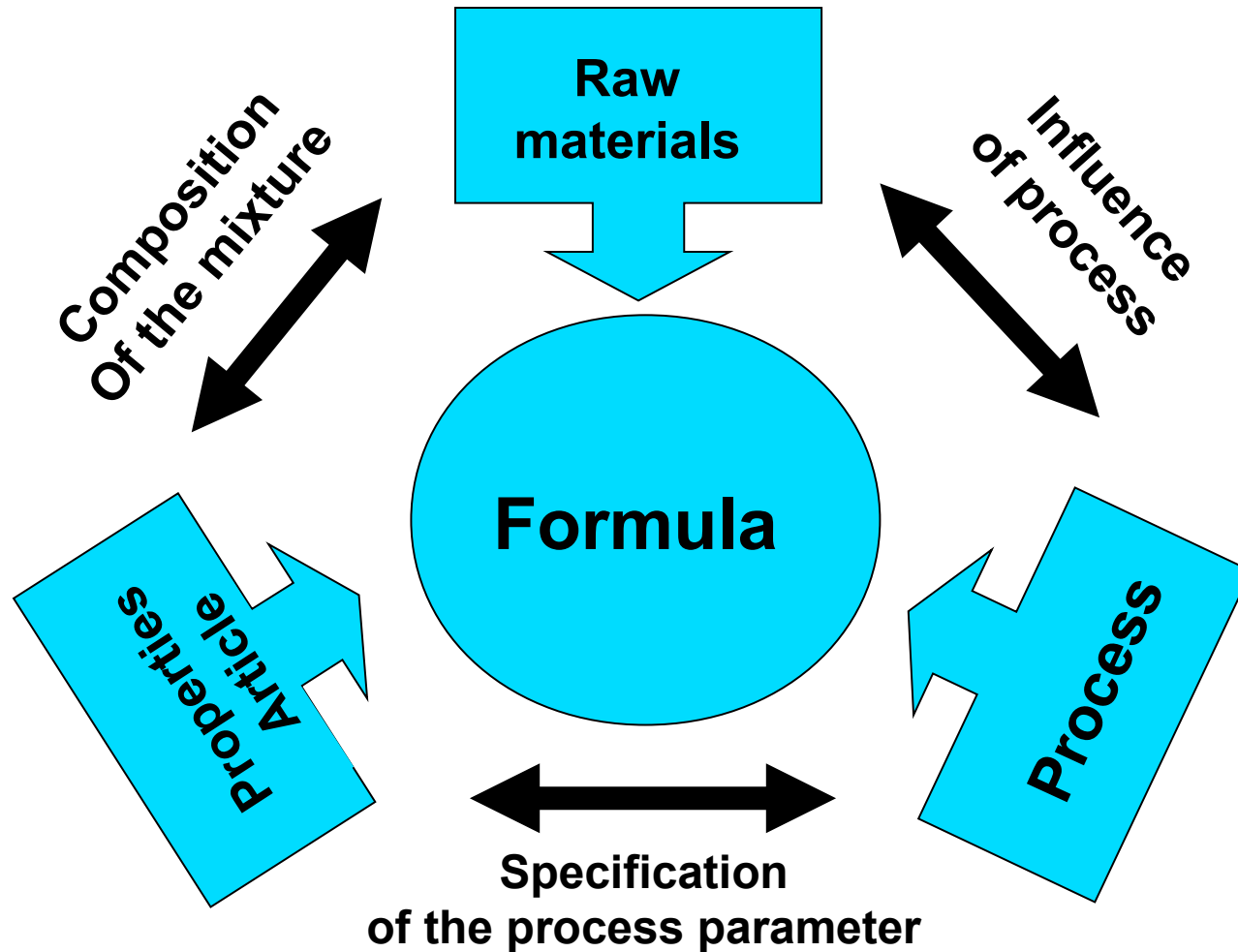
- **The process ability of a compound according to a formula under consideration of modern quality concepts.**
 - **The end product must fulfill all requirements under operating conditions.**
 - **The desired product and quality properties shall be got below the lowest costs.**

 - **The solution is the best relationship in the preparation from mixture components and qualities.**
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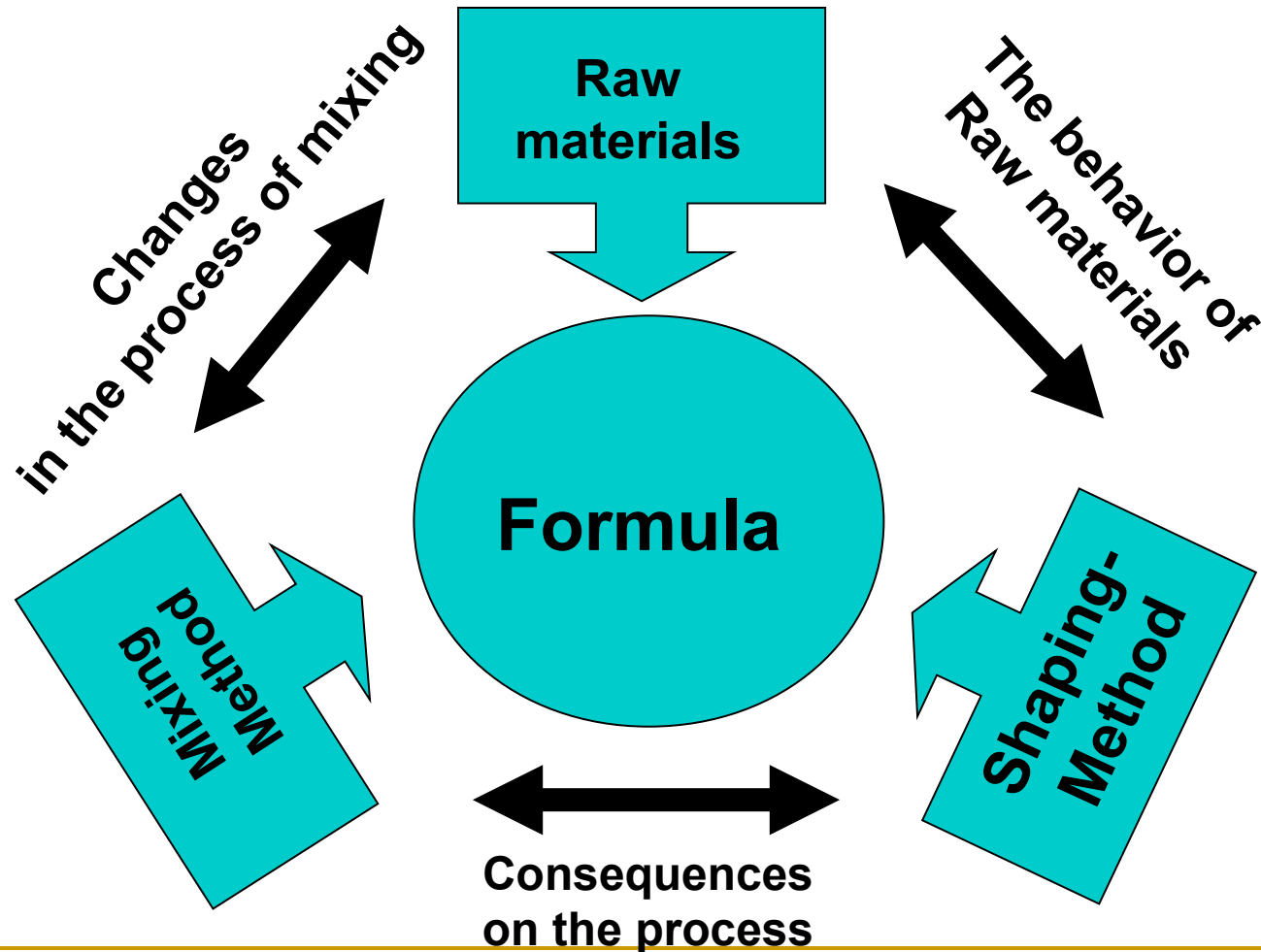
Formula development



Position of Formula in the process chain



Formula Changes through processing



Meaning of the formula

- Under the assumption that the processing methods run in the optimum, the formula, resp. the articles produced, determines, the economic of the production process, at least to the predominant part.
 - Mixing Plant: Material amount is between 80-90 %
 - Extrusion: Material amount is 75 -85 %
 - Injection molding: Material amount (without consideration inserts) may be between 50 and 70%
 - The importance of the recipe development must be carried out methodically and systematically.
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Methods for recipe development

- The recipe developer is into of use some few methods selected by him subjectively for most cases the intensity seeming suitable in this one in one! It seems more sensible, in
 - Dependence of the problem definition,
 - The acting persons and
 - The situation
 - selecting consciously of the existing methods and adapting in addition one.
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Methods for recipe development

- **Which methods are there?**
 - Trial and Error
 - Repetition
 - **Change of a known compound through:**
 - Gradual change of a factor
 - Relative change two factors to each other
 - **Analysis with the help of correlation and regression**
 - Pareto analysis
 - Cause effect diagrams
 - **Statistic Experimental Design**
 - Latin Square test approaches
 - Fractional Factorial Design / Respons Surface Desing
 - **Variance analysis**
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Tools for the recipe design

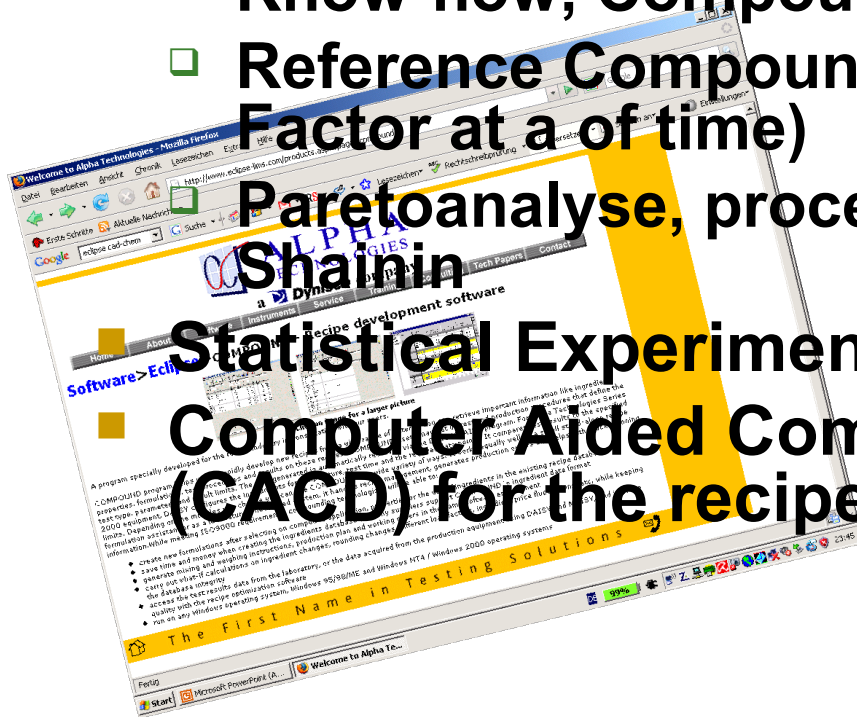
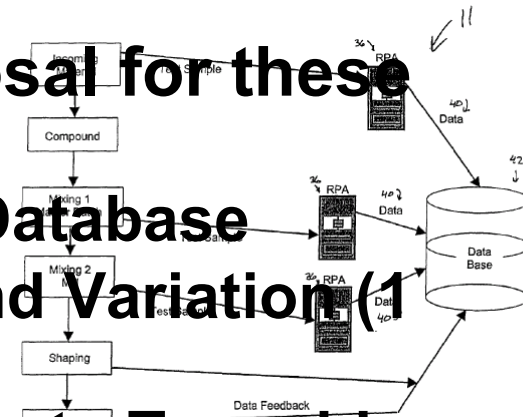
- Which tools are at disposal for these methods?

- Know-how, Compound Database
- Reference Compound and Variation Factor at a of time

Paretoanalyse, procedure to Taguchi, Shainin

Statistical Experiment Design (DOE)

- Computer Aided Compound Design (CACD) for the recipe development

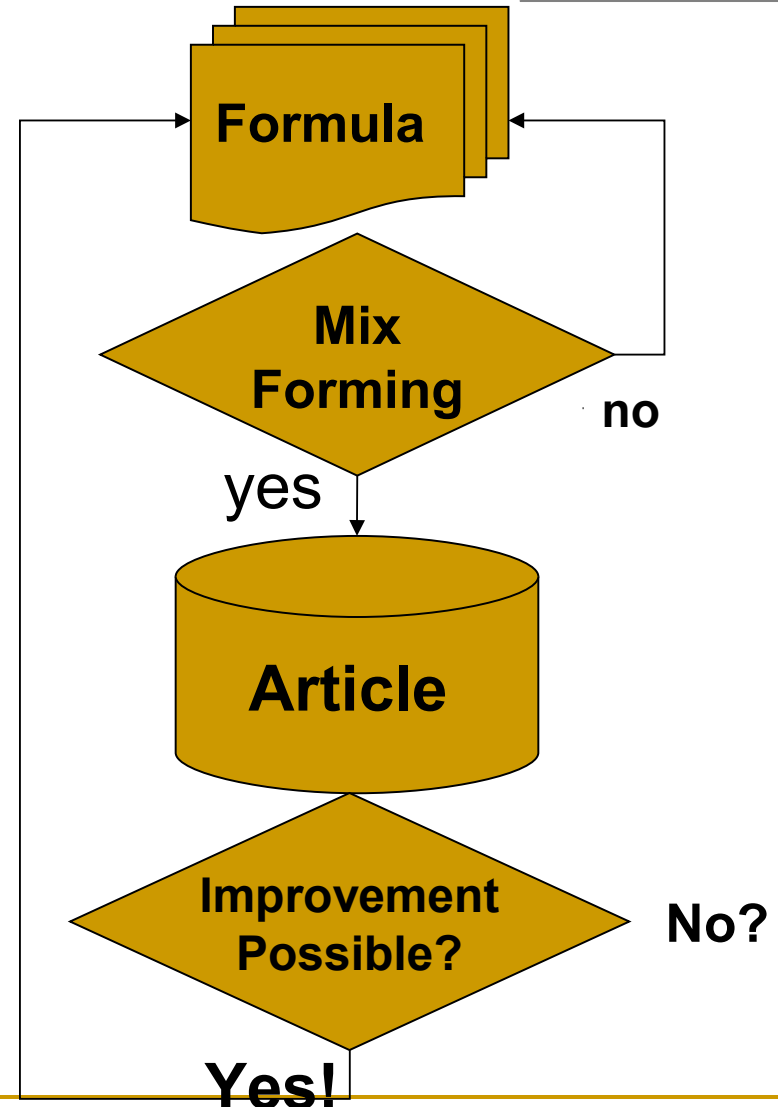


■ Recipe database

- Useful, if the following information exists and can be analyzed.
 - Historical knowledge around materials in the accompanying mixtures
 - Database about conspicuous features of the mixture in production
 - Database about the product and its complaints
 - Design guide for the recipe construction
-

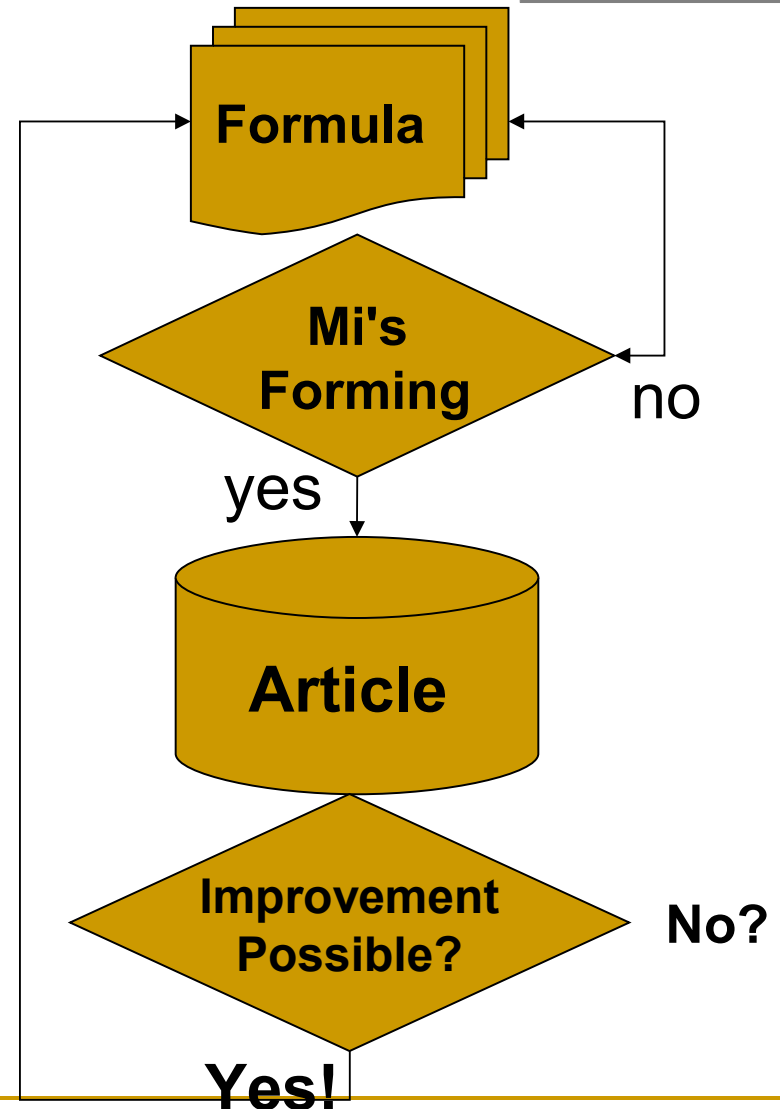
Tools for the recipe design

- **Historical knowledge around materials and mixtures**
 - The experienced compound developer can create mixture recipes from this accumulated experience with a good feeling in a more "artistic" process
 - A processing method is then established based on this creation
 - What fails to appear is the iterative optimization process like 6sigma® or alternative like "Kaizen"



Tools for the recipe design

- "Creative recipe design" does not permit any incremental customization strategy
- Being needed
 - Simultaneous Engineering
 - Statistical methods
 - Statistic Design of Experiments



Tools for the recipe design

- "Creative design" permit an incremental custom strategy
- Being
- Simultaneous Enwickem
- Statistical methods
- Statistical test plan technology

Remarks:

Formulae are improved: so-called tuning, but the correspondent processes (tools/machines) are left open

if processes improvements worked out and established, however mostly without inclusion of the mixture development.

Improvement
Possibly?

No?

Yes!

- **Reference compound and variation (1 Factor at a time)**
 - **Ignores interactions**
 - **Neglect statistical noise**
 - **Causes high effort, because many iteration steps do not lead – possibly but not for certain - to the goal envisioned, even over a great time period.**
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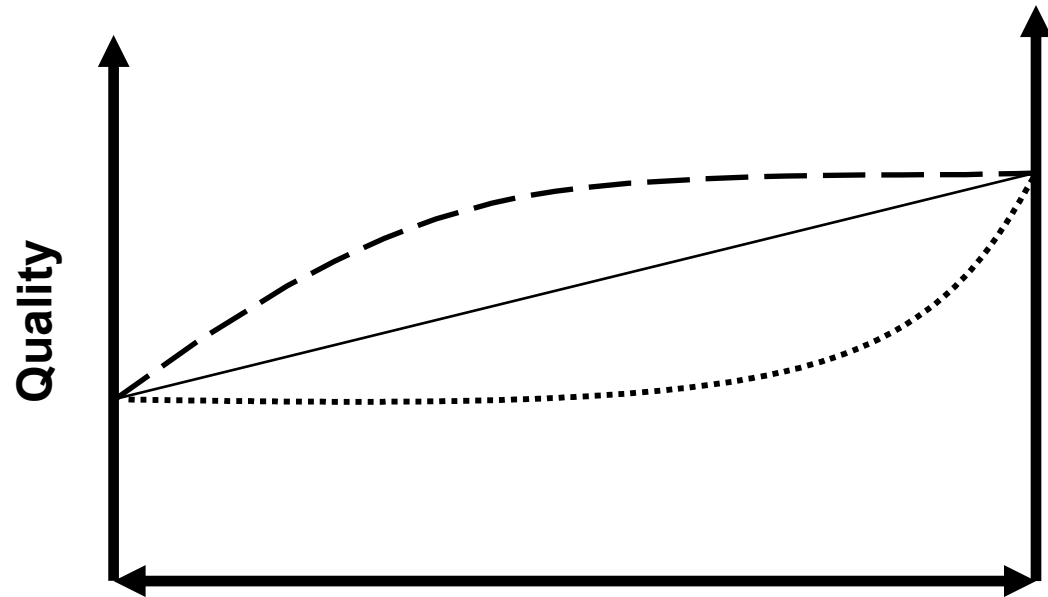
Tools for the recipe design

- **Pareto Analysis, procedure acc. to Taguchi, Shainin**
 - **Statistical Design of Experiment (DOE)**
 - **Allows to work out relations between cause and effect statistically secured**
 - **Effect can be caused and eliminated again**
 - **Knowledge keeps its validity!**
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Tools for the recipe design

Statistical Experimental Design: Example

- Analysis of a filler-/ softener pair
- Exchange of fillers/softeners



Filler 1	%	0	25	50	75	100
Filler 2	%	100	75	50	25	0

Tools for the recipe design

Statistical Experimental Design: Example

- For the assessment of polymer a filler/softener with the help of a square test plan:
 - Polymeri - categorical factor
 - Filler - numeric factor
 - Plastiziser - numeric Factor
- This investigation results the exact behaviour of the Polymer with the filler/platiziser interaction.
- It can as well be used for the choice of fillers (softeners). If qualities differ, the suitable correction can be read from the results of experiments.

Example:

Polymeric	phr	Categorical	C1	C2
Filler	phr	Numeric	20	80
Plasticizer	phr	Numeric	10	50

Tools for the recipe design

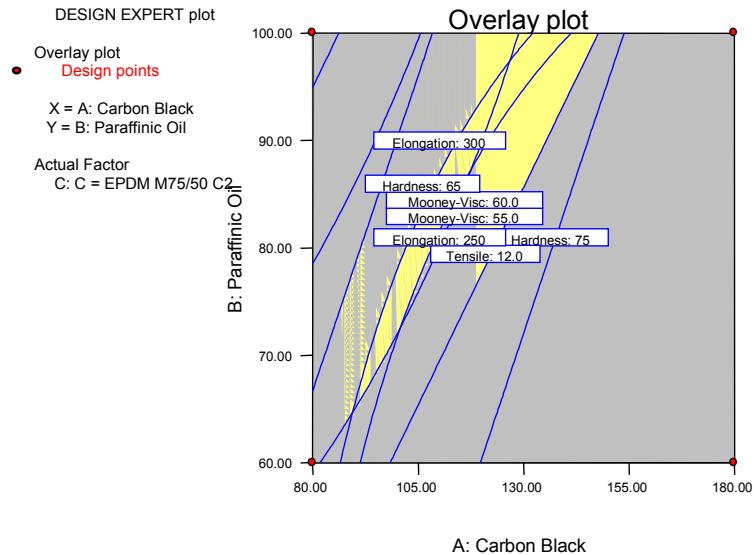
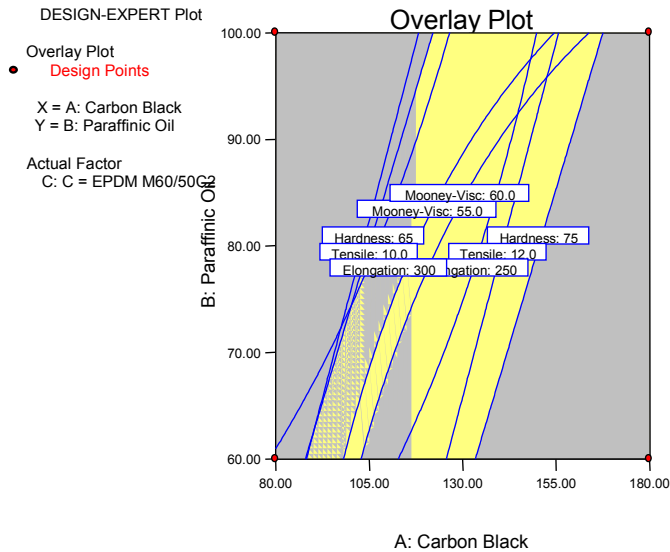
Statistical Experimental Design: Example

- Test Design for the choice of polymer
- the square test plan corresponds to $\frac{1}{2}$ Fractional Factorial design with of 2 numeric factors and 1 categorical factor:

StOrd		Run			
1	4	Block 1	10.00	20.00	C1
2	2	Block 1	50.00	20.00	C1
3	8	Block 1	10.00	80.00	C1
4	1	Block 1	50.00	80.00	C1
5	3	Block 1	10.00	20.00	C2
6	6	Block 1	50.00	20.00	C2
7	5	Block 1	10.00	80.00	C2
8	7	Block 1	50.00	80.00	C2

Tools for the recipe design

Statistical Experimental Design: Example



■ Result of a polymer comparison:

- To receive the same compound viscosity, hardness, tensile strength and elongation at break
- Does the correction of the carbon black amount become necessary.
- Retention of the carbon black amount and correction of the quantity of oil yields deviations at properties

Tools for the recipe design

Statistical Experimental Design: Example Accelerator System

- **Accelerator systems can be examined for two ways:**
 - **"Response Surface" test plan**
 - Sulfur/accelerator relationship is not constant!
 - The influence of the sulfur bridge structure on the qualities is ignored!
 - **"Mixture" test plan**
 - Sulfur/accelerator relationship remains constant
 - The interactions of the accelerators with each other are recognized.

 - ***Recommendation: Both types of the test plans behind each other carry out with the respective best result from the former one.***
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Tools for the recipe design

Statistical Experimental Design: Example Accelerator System

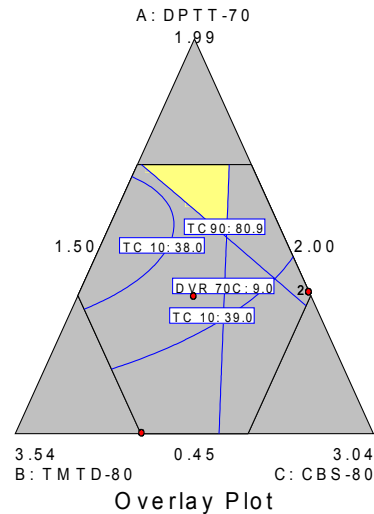
DESIGN-EXPERT Plot

Overlay Plot

- Design Points

X1 = A: DPTT-70
X2 = B: TMTD-80
X3 = C: CBS-80

Actual Component
D: Schwefel-80 = 0.81



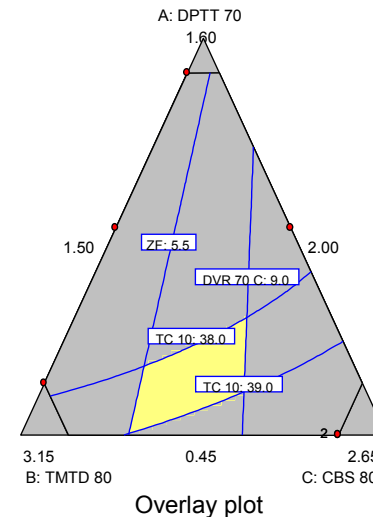
DESIGN EXPERT plot

Overlay plot

- Design points

X1 = A: DPTT 70
X2 = B: TMTD 80
X3 = C: CBS 80

Actual Component
D: Sulfur 80 = 1.20



- In the example the relationships of the accelerators at different accelerator/sulfur levels were examined.
- It can be shown, that
 - Sulfur can be replaced by an adequate sulfur donator amount
 - The properties looked at being not changed.

Tools for the recipe design

Statistical Experimental Design: Example

- **Killer argument**
 - **NO TIME FOR A SYSTEMATIC EXAMINATION! WE NEED A FAST SOLUTION!**
 - **IT IS SIMPLE AND FAST**
 - **TOO MANY EXPERIMENTS AND TOO HIGH COSTS**

 - **Counter-arguments**
 - **In the case of a complaint Resources and money are spent which was saved putatively.**
 - **Developments remain valid and do not lose her meaning even if the conditions are subject to changes.**
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Tools for the recipe design

CAD - calculation of formulae

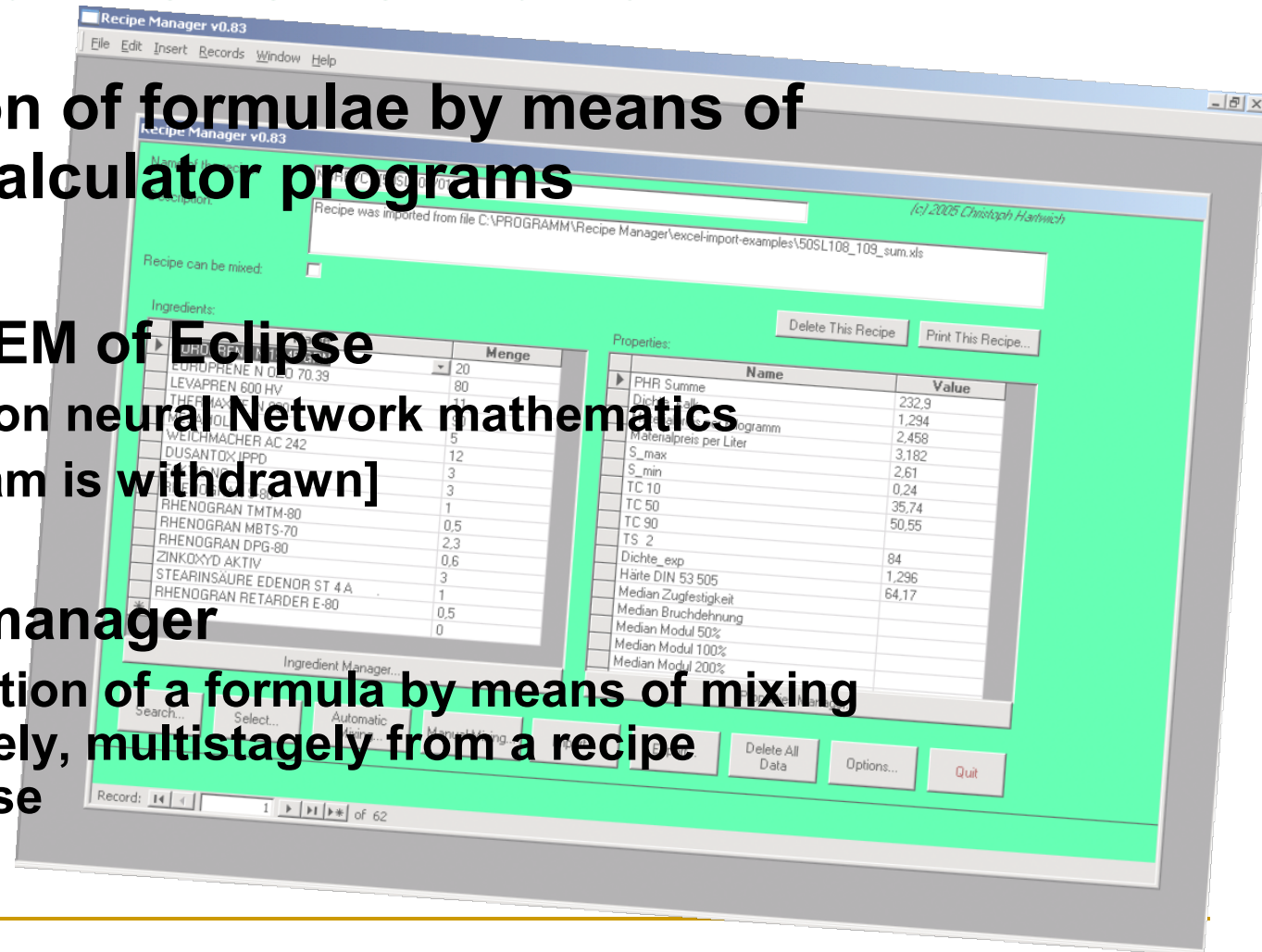
- Calculation of formulae by means of suitable calculator programs

- CAD CHEM of Eclipse

- Based on neural Network mathematics
- [Program is withdrawn]

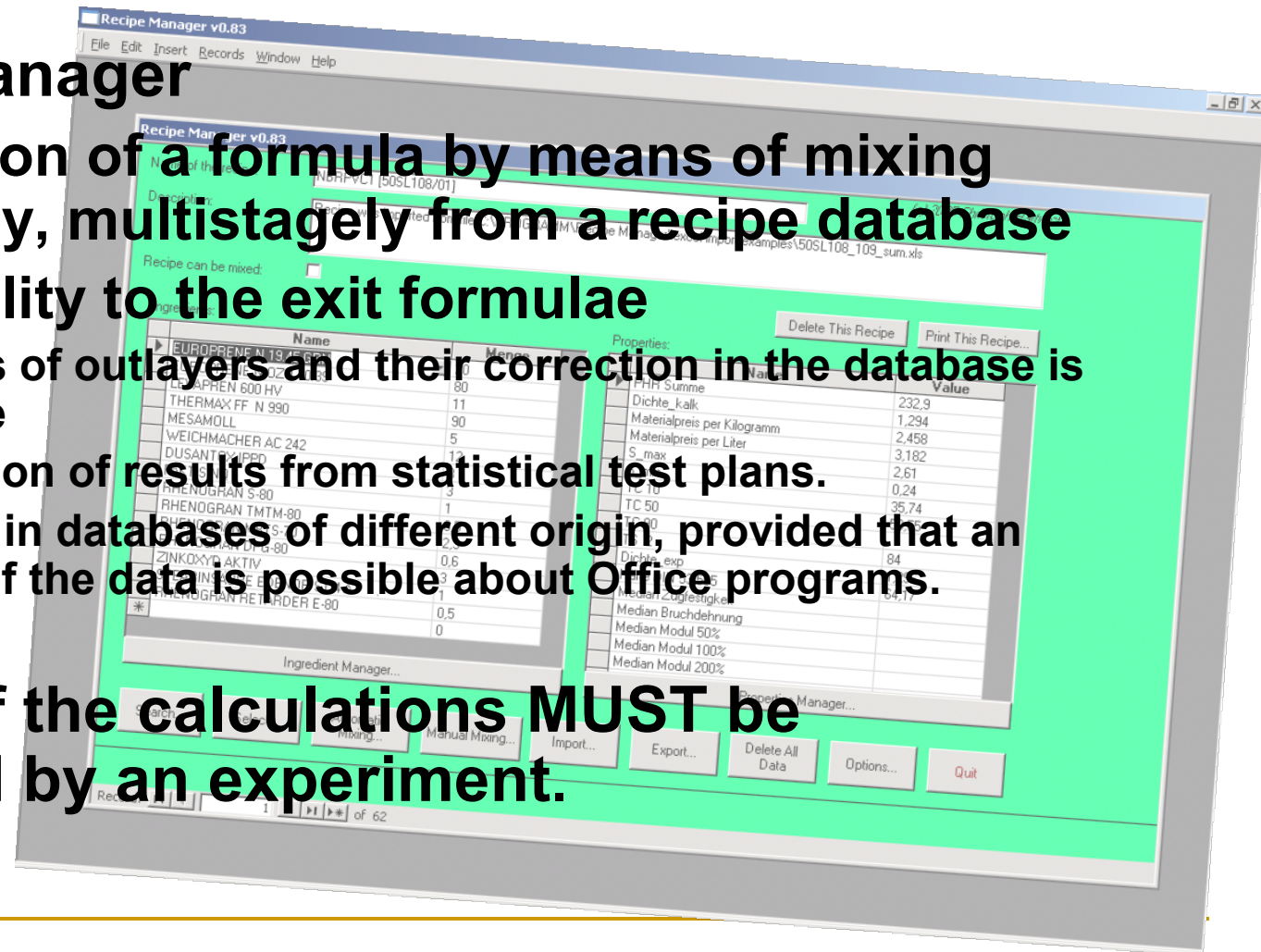
- Recipe manager

- Production of a formula by means of mixing iteratively, multistagely from a recipe database



Tools for the recipe design CAD - calculation of formulae

- **Recipe manager**
 - Production of a formula by means of mixing iteratively, multistagely from a recipe database
 - Traceability to the exit formulae
 - Analysis of outliers and their correction in the database is possible
 - Integration of results from statistical test plans.
 - Enquiry in databases of different origin, provided that an export of the data is possible about Office programs.
- Results of the calculations **MUST** be confirmed by an experiment.



Tools for the recipe design

Summary

- These tools described here, if problem-oriented used, permit:
 - Quick, planable development results even at complex problems
 - Results statistically sound
 - Cost advantages are gained if these instruments are used for the improvement in the production and a more efficient usage of materials.

