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Some Reflections on My Zürich Years

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1. Education up to the Diploma

The main challenge from 1968 to 1970 was to create a course in Chemical Engineering, which was a blend of Chemistry and Chemical Engineering appropriate to the needs of Swiss chemical industry. Long discussions took place with many people in Basle as well as with colleagues at ETH in Chemistry and Verfahrenstechnik. The final result, leading to the dipl. Chem.-Ing. ETH, was introduced in October 1970 [1] and with small modifications is still in operation. Of the many persons who gave advice and guidance in the planning and early implementation of the new curriculum, I remember with gratitude Profs. E. Plattner and W. Regenass in Basel and Profs. H. Günthard, G. Gut, N. Ibl, P. Pino, W. Richarz, and H. Zollinger in Zürich.

Some guiding principles for the new Diploma course were [2]

1. science-based, not technology-based,
2. basics are Chemistry, Mathematics, and Physics,
3. Chemical Engineering must derive logically from the basics,
4. students should learn how specific answers to industrially oriented questions can be deduced from a set of broad, general principles (mass and energy balances, kinetics, thermodynamics, heat, and mass transfer, reaction engineering, etc.),
5. the intellectual development of students should be promoted,

6. students should be prepared for industrial careers in applied research, process development, and production.

Opportunities to criticize the ETH curriculum have come through internal discussions, in meeting colleagues from other countries and through sabbatical leave in other universities.

What are my conclusions?

1. Students tend to keep their knowledge in compartments, e.g. what is learned in Mathematics about differential equations is not always recognized when studying reactor performance.
2. Compared to many other courses, the substantial amount of practical work in Zürich, together with homework exercises and case studies in years 3 and 4 make the learning experience more active and less passive. The 'Diplomarbeit' reinforces learning through doing.
3. ETH is moving in the direction of 'Semesterprüfungen', not unlike the American examination system. For Chemical Engineers this could usefully reduce the large volume of material from Semesters 5, 6, and 7 currently in the final Diploma examinations.
4. Professors should collaborate more to produce well-balanced lectures and exercises. Earlier, Norbert Ibl, Werner Richarz, and I made some progress in the introductory courses of Year 2 and in Reaction Engineering in Years 3 and 4. Sharing the teaching by rotation, exchange, etc. could build up better material for students, but also be more interesting for professors. A person can not teach all subjects, but more collaboration is possible.
5. The student, who has just gained a Diploma, will have some overview of

Chemical Engineering. Many, however, lack confidence in their own abilities to apply their knowledge in industry. They need to remain for a doctorate, which helps the process of maturing to continue. The increasing ability to judge situations and the developing scientific and personal independence are worthwhile. These changes can not be so readily achieved if the necessary period of time (3½–4 years) were spent in industry, especially not under today's increased pressure and tempo. An educational establishment is generally better, although one must admit that these vary greatly in quality, do not always make efficient use of time and can dissipate effort in local disputes between rival academics.

6. Education takes place on a long timescale and is an on-going process, particularly needed today in a fast changing world. Its various phases – home, school, university, and professional life – should not be regarded separately. Swiss university education benefits from the broad and thorough preparation in the 'Gymnasium'. This is also of great benefit in later life and helps in understanding and dealing with our complex and ever changing modern world. I have been impressed by the all-round qualities of many ETH students.

2. Research and Postgraduate Education

Research was started early in a number of directions, some of which will be sketched in this section. The dates given here refer to the year in which a doctoral

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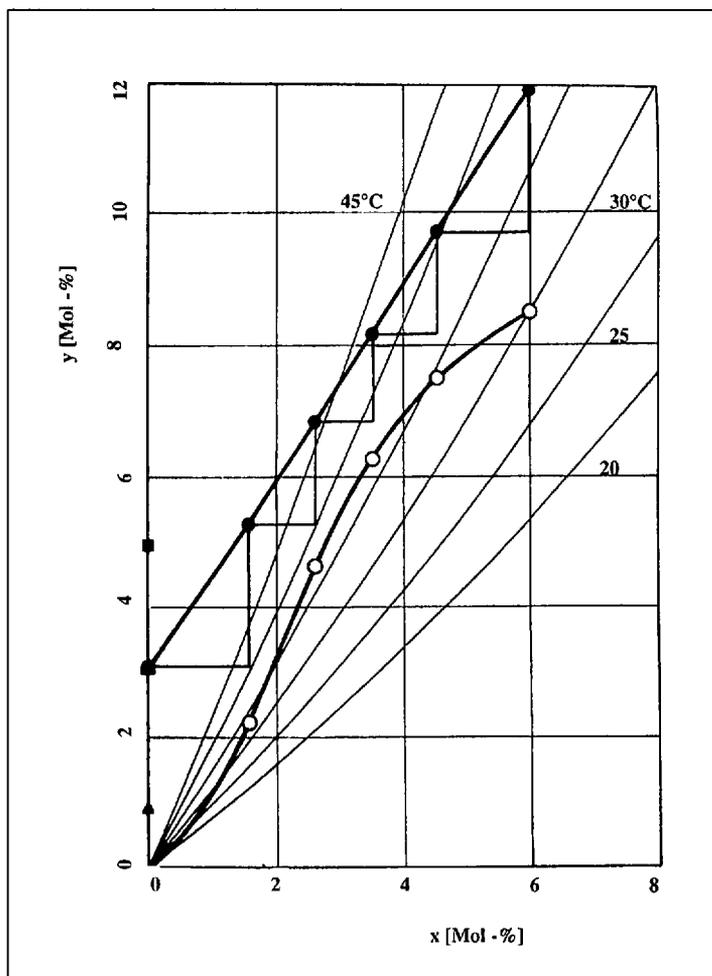


Fig. 1. McCabe-Thiele diagram for non-isothermal absorption of NH_3 in a five-stage absorption tower

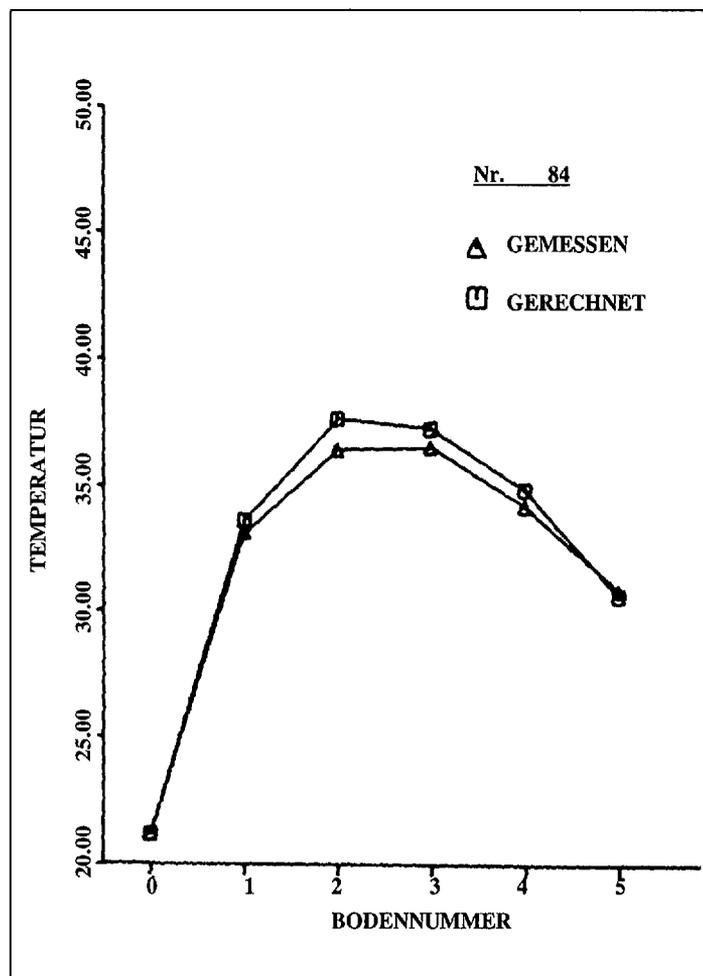


Fig. 2. Measured and computed liquid-phase temperatures for the experimental conditions of Fig. 1

student completed his or her dissertation. Before outlining research topics, some general characteristics of our approach will be summarized as follows:

1. include as much chemical and physical information as possible,
2. aim where possible for quantitative results,
3. reach conclusions which are reasonably general,
4. balance theoretical and experimental methods.

Modeling has been important throughout and consisted of:

1. analyzing the actual situation by application of chemical and physical laws to obtain the defining differential equations,
2. solving these equations on a computer to find the behavior of the model,
3. comparing this behavior with experimental information to decide upon the applicability of the model,
4. using an accepted model to predict the behavior of a system under new conditions.

It is worth emphasizing the constant exchange of information between model and experiment in step 3.

2.1. Heat Effects during Gas Absorption and Reaction (1968–1978)

My first doctoral student was *Urs von Stockar* (1972), now professor, EPF, Lausanne. When a gas dissolves, its heat of solution tends to raise the temperature of the solvent whereas any evaporation of the solvent tends to lower its temperature. This is an important parameter determining the solubility of the gas and the partial pressure of the solvent. It is determined by a set of heat and mass balance equations, which together with all phase equilibria were incorporated in a dynamic model. When numerically integrated, the model predicted the steady-state behavior of a multistage gas absorption tower, which compared well with experiments on the absorption of NH_3 into H_2O .

Fig. 1 contains an essentially straight operating line, representing the mass balance on NH_3 between gas and liquid phases, and a strongly curved equilibrium curve, which would have been nearly linear if the temperature in the absorber had been constant. With an inlet gas concentration of 12 mol% NH_3 and 5 non-equilibrium stages (*i.e.* 5 imperfect plates) in the absorber, three predicted exit NH_3 concentrations

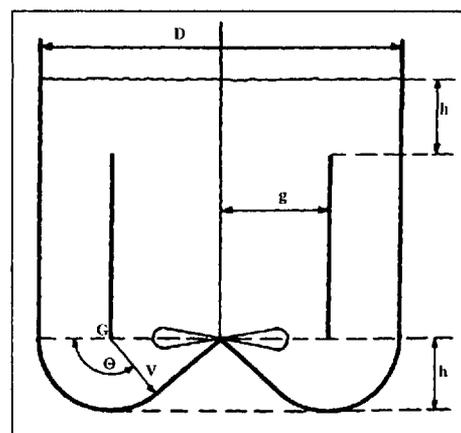


Fig. 3. Stirred tank for suspension of crystals having contoured base and constant cross-sectional area for the flow

are shown on the ordinate. Adiabatic absorption with no evaporation of H_2O would have resulted in 5% NH_3 in the exit gas stream. On the other hand, isothermal absorption would have produced 0.8% NH_3 . The measured value was 3% NH_3 which agreed very well with the model for adiabatic absorption allowing for evaporation of H_2O . Fig. 2 compares the measured and computed liquid-phase temperatures for the case shown in Fig. 1. The feed temper-

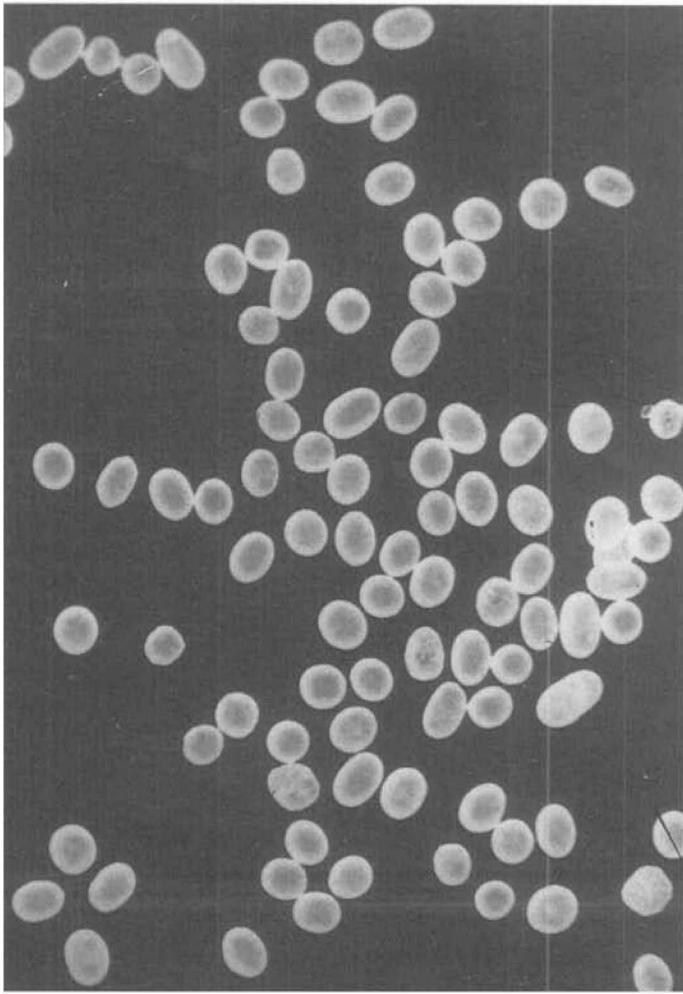


Fig. 4. Crystals of HMT after suspension in a conventional stirred tank showing damaged edges and corners

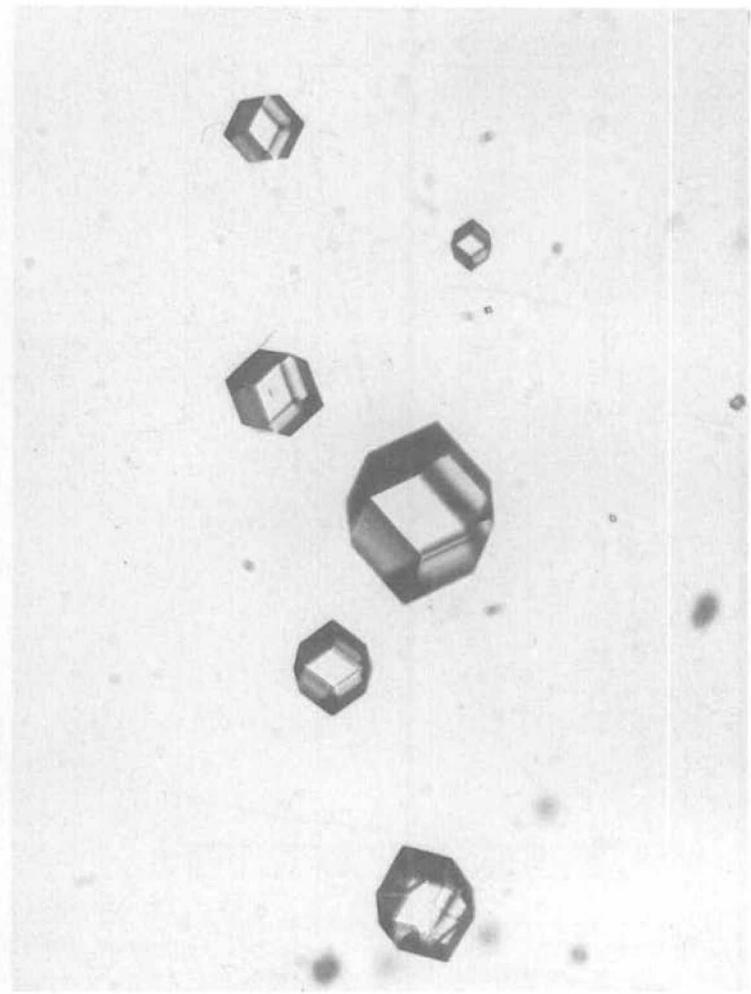


Fig. 5. Crystals of HMT after suspension in contoured base tank

ature of H₂O was 21°, but the maximum solvent temperature attained within the absorber was 36°. The fall of temperature to 31° at the bottom of the tower, despite heat generation due to NH₃ absorption, was due to evaporation of H₂O.

This analysis of heat effects features today in textbooks, e.g. [3][4].

Heinz Winzeler (1975), now professor, HTL, Winterthur, studied the liquid distribution in the *Sulzer* packing, widely used in absorption towers, particularly the residence-time distribution (RTD) of the liquid film.

Henri Gros (1977), now *Sulzer*, included chemical reactions. Modeling and experimentation were carried out for the absorption and reactions of oxirane in H₂O to form ethylene glycol in a two-stage bubble column.

2.2. Crystallization of Organic Solids from Solution (1970–1985)

Initially basic quantities like diffusivities and solid-liquid mass transfer coefficients as well as the quality of crystal suspension in stirred tank and fluidized bed crystallizers were studied. *Pietro Bomio* (1973), *Stefan Aeschbach* (1976),

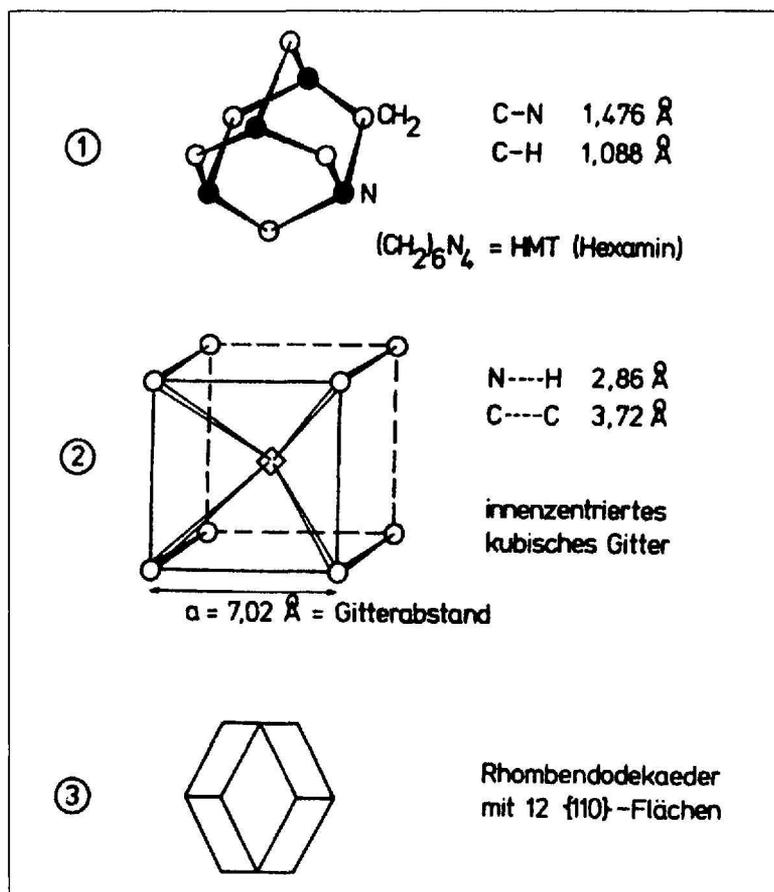


Fig. 6. Some properties of HMT crystals

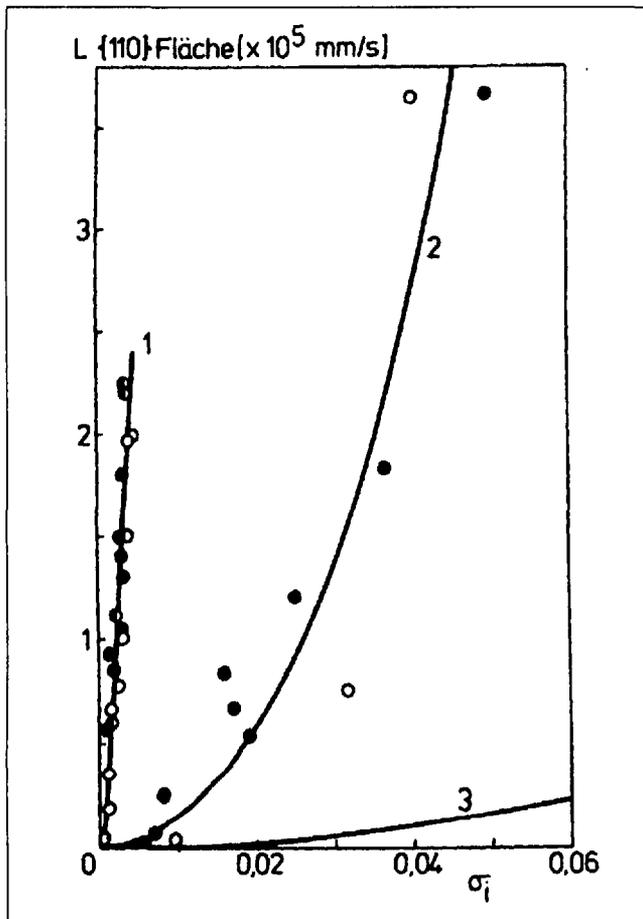


Fig. 7. Comparison of growth rates of HMT from 1) aqueous solution, 2) ethanolic solution, and 3) vapor phase

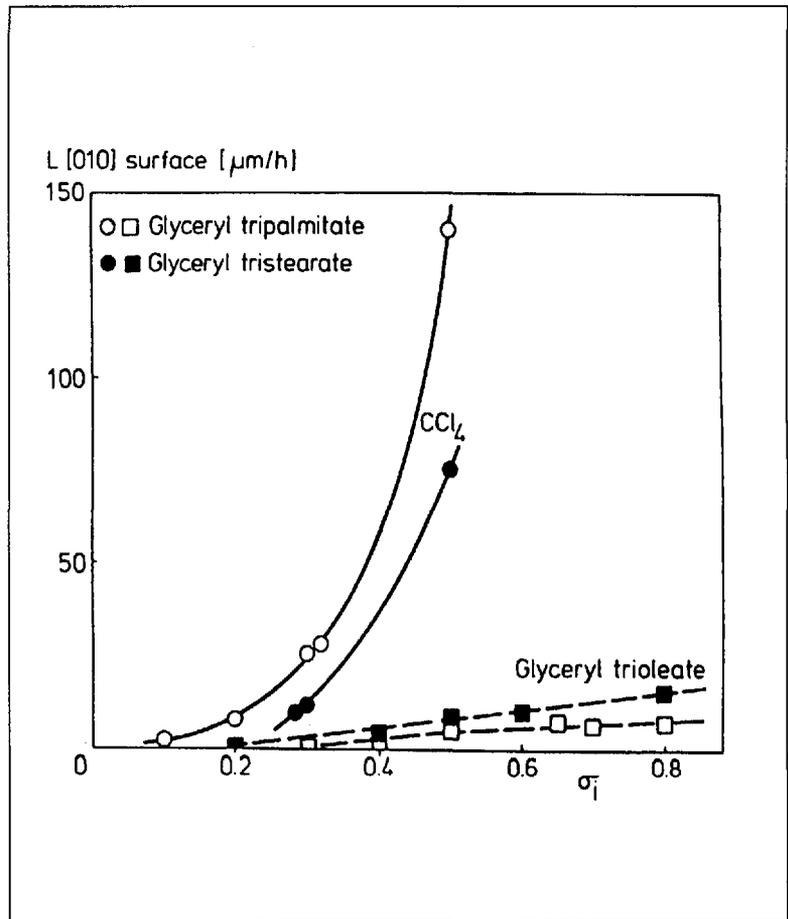


Fig. 8. Comparison of growth rates of two triglycerides from two solvents

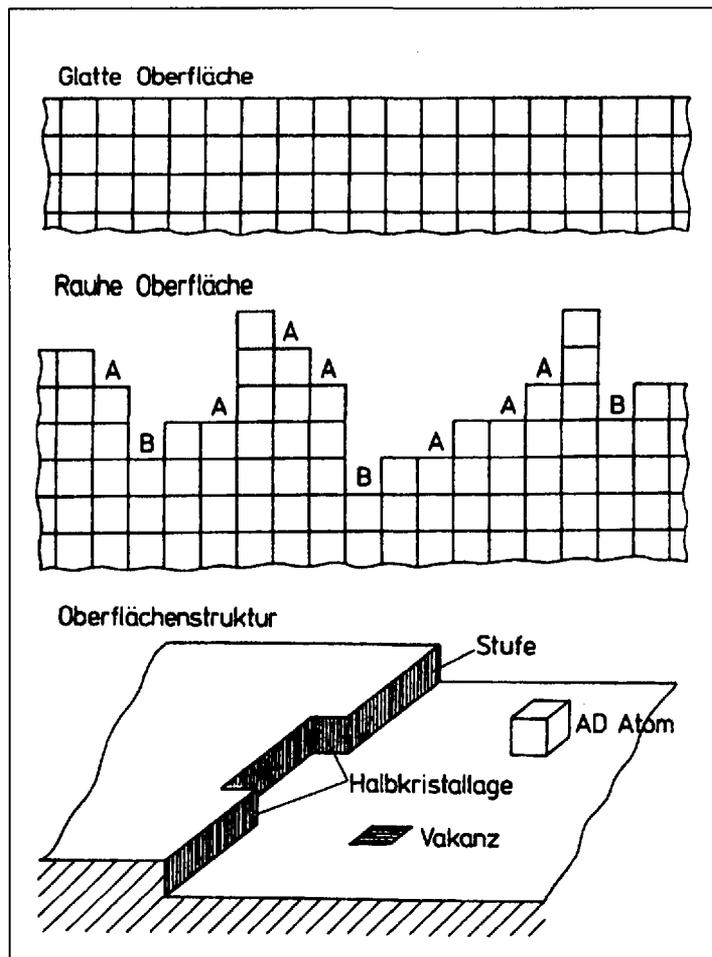


Fig. 9. Rough and smooth crystal surfaces showing steps and kink sites

Armin Rütli (1976) – all later with Sulzer –, and Marcel Olbrecht (1974), now Lonza, USA, worked on these problems. Development then occurred in two directions: applied and fundamental.

In the *applied* direction the contoured base tank of Aeschbach and Rütli (Fig. 3) was used for continuous crystallization at various scales by Konrad Hungerbühler (1978), subsequently Ciba and now professor, ETH-Zürich. The principle of this tank is a constant cross-sectional area, which minimizes hydraulic losses and increases the circulation velocity, so aiding crystal suspension and simplifying scale-up. We are all familiar with the CSTR (continuous stirred tank reactor) where it is easy to achieve perfect bulk-scale mixing. Sedimentation, due to a density difference, makes it much more difficult to homogenize a suspension, particularly during slurry removal in continuous operation. Larger crystals are preferentially retained in the crystallizer, whilst smaller ones are preferentially washed out. This makes Reaction Engineering calculations for nucleation and crystal growth inaccurate or even useless in continuous operation.

Fig. 4 shows crystals of an organic solid grown in a conventional stirred tank.

Sedimentation of larger crystals caused higher concentration and larger residence times than when mixing is perfect. This promoted mechanical damage (abrasion) and the natural edges and corners were worn away. Fig. 5 refers to the same solid (hexamethylenetetramine, HMT) but these crystals were grown in a contoured base tank. Well developed edges and corners in crystals up to 1–2 mm size are evident and resulted from the lower agitator speed needed in the special tank.

Classification – as opposed to perfect mixing – was deliberately induced by Michel Zabelka (1978), later Sulzer, and a theory for predicting the particle-size distribution during nucleation and growth was developed which compared well with experiments.

Marius Leimer (1985), now Ciba, applied cycles of growth and dissolution to cause recrystallization and hence to modify the purity and particle-size distribution of the crystals. Once again this investigation involved considerable effort in modeling and computing as well as substantial experimentation.

In the fundamental direction a molecular-scale interpretation was found for the influence of the solvent on the growth kinetics of organic crystals. Experimental work concentrated on

- a) hexamethylenetetramine (Fig. 6), a molecular solid having only one crystal face,
- b) 1,3-dihydroxybenzene (resorcin) which has both polar and non-polar faces. It was studied experimentally by Bogdan Milisavljevic (1982).

A post-doctoral guest, Dr. Roger Davy, now ICI, played an important role in these investigations. Fig. 7 shows the linear growth rate of HMT as a function of interfacial supersaturation from 1) aqueous solution, 2) ethanolic solution, and 3) condensation from the vapor phase. Figs. 7 and 8 show a large influence of the solvent on the crystal growth rate. Fig. 9 refers to the structure of the solid-liquid interface, which when it is rough exhibits a higher density of step and kink sites than when it is smooth. Fig. 10 shows the energy balance for a roughening transition and the surface entropy factor α . The bond energies in this model, Φ , refer to solid-solid (SS), liquid-liquid (LL), and solid-liquid interactions (SL). The latter depend strongly on hydrogen bonding, e.g., as it occurs between HMT and water and, to a smaller extent, HMT and ethanol. It proved possible to establish a quantitative link between these thermodynamic aspects and the kinetic ones shown by the examples in Figs. 7 and 8.

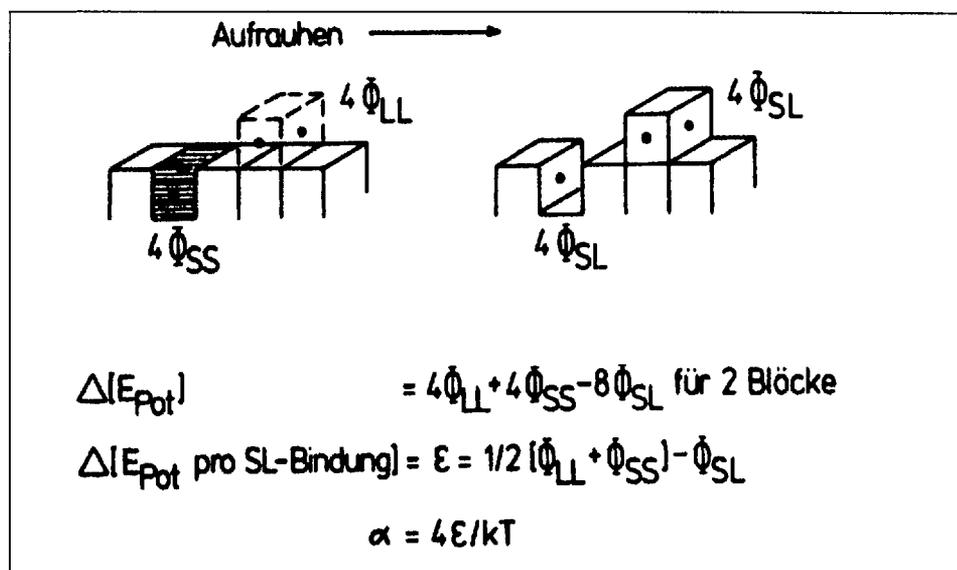


Fig. 10. Energy balance for a roughening transition and the surface entropy factor

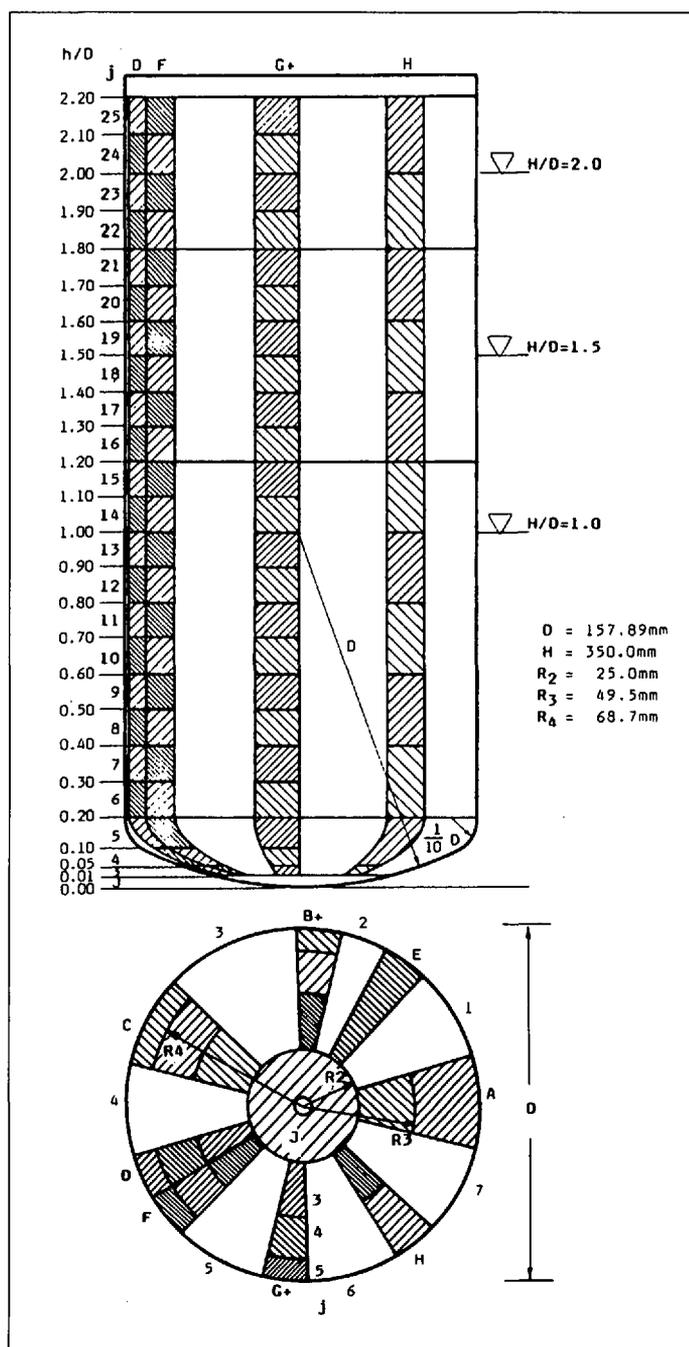


Fig. 11. Stirred tank with electrodes in its wall and base for local electrochemical mass transfer measurements

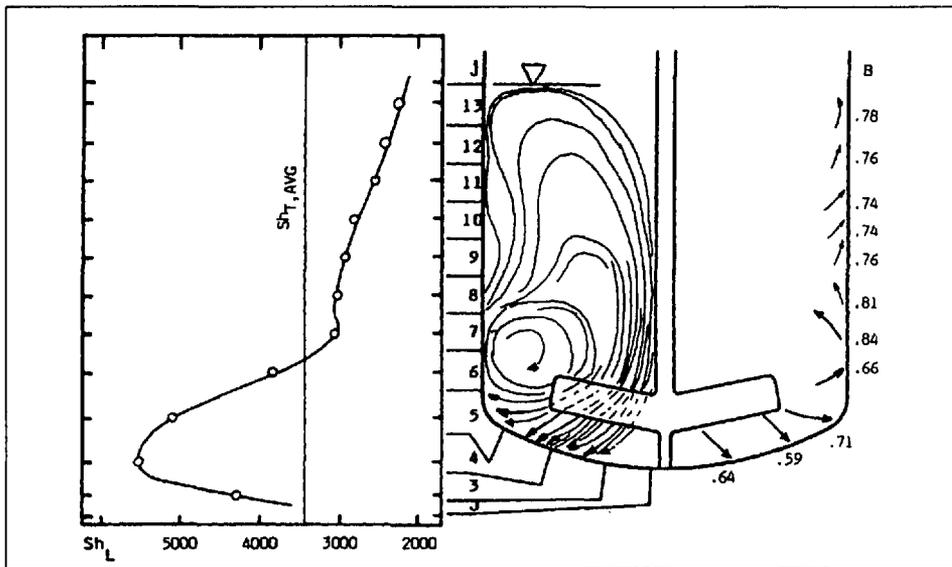


Fig. 12. Local and average mass transfer rates, represented by the Sherwood number, as a function of position (left); tank with Pfaudler impeller showing flow pattern and positions for k_L determinations (right)

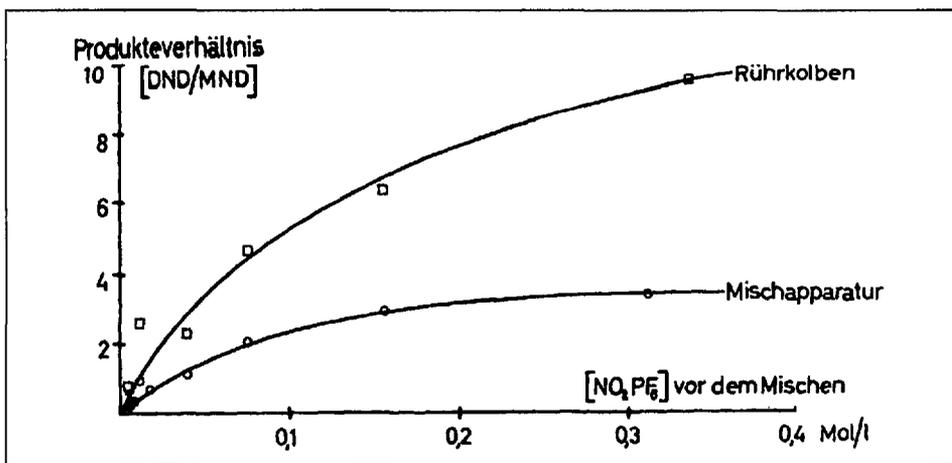
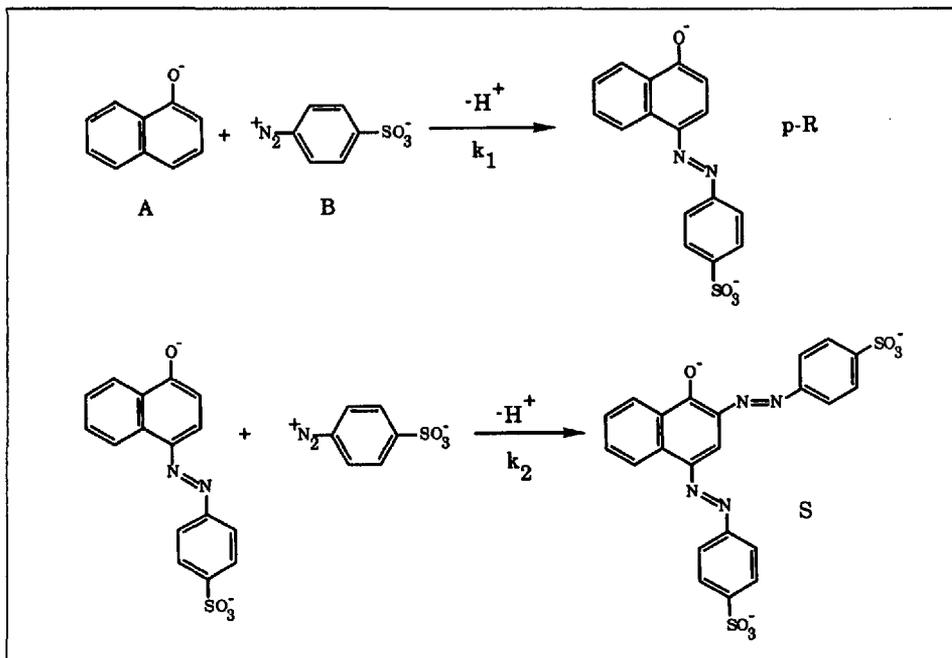


Fig. 13. Product distribution (di- and monosubstituted durenes) in the single phase nitration of durene with a nitronium salt

Scheme 1. Diazo Coupling between 1-Naphthol and Diazotized Sulfanilic Acid Represented by a Two-Reaction Scheme



2.3. Bioengineering

In 1972, Prof. Armin Fiechter, Institute of Microbiology, proposed some collaborative research and four doctoral students worked partly in the TCL and partly with biotechnologists. This stimulated an interest in applying Chemical Engineering methods to fermentation, enzymatic catalysis, cell production, etc. PD Dr. I.J. Dunn, PD Dr. E. Heinzle, and Dr. J.E. Prenosil have continued to work on selected biotechnological problems and have described much of the progress achieved in two recent books [5][6].

2.4. Mixing (1968–1996)

2.4.1. Viscous Mixing and Residence-Time Distribution

Willy Knöpfli (1973), subsequently Escher-Wyss, made a detailed study of flow patterns in Newtonian and pseudo-plastic (shear-thinning) fluids stirred in the laminar and transitional regimes by a helical ribbon impeller. To study the continuous mixing of viscous liquids, a suitable tracer was needed for RTD measurements. A reusable photochromic dye – a spiropyrane – was developed and successfully employed by Gerhard Giger, now Ciba, working with Prof. Richarz, and by Rudolf Riesen (1978), now Mettler.

2.4.2. Heat and Mass Transfer Rates in Tanks

Gianni Ruchti (1979) worked on the sulfite oxidation method to measure k_{1a} , the volumetric gas-liquid mass transfer coefficient.

Martin Bürli (1979), working with Prof. Regenass and his colleagues in Ciba, applied the heat-flow calorimeter to measure the power consumption of a number of impellers as well as their process-side film heat transfer coefficients.

Daniel Meister (1980), now Ciba, designed and characterized a multistage, mechanically agitated column for gas absorption and reaction.

Tom Post (1983), now Lightnin Mixers, USA, employed the electrochemical method of Prof. Norbert Ibl and Dr. Othmar Dossenbach to measure local mass transfer coefficients on the wall of a stirred tank. Fig. 11 shows the tank, with 144 wall electrodes and 28 electrodes in its base. The limiting current for the reduction of iron-cyanide ions at a cathode was transmitted to a PC to evaluate the local liquid-film mass transfer coefficient at any electrode. Fig. 12 shows on the right the tank with a Pfaudler impeller and various electrode positions. On the left local and average mass transfer rates, represented in

dimensionless form by the *Sherwood* number, are shown as function of position. The flow patterns deduced from the local mass transfer rates are given on the right of *Fig. 12*. When averaged over the whole inner surface, the mass transfer correlation could be re-expressed as a heat transfer correlation. The same result was then obtained as in *Bürli's* work with the *Ciba* heat-flow calorimeter.

Bruno Peterer (1986), now *Ciba*, extended this study to pseudoplastic fluids, where even more variability than in *Fig. 12* arises from the non-*Newtonian* property. The electrochemical method to measure mass transfer coefficients also performed well in this case.

Felix Hoch (1986), now *Ciba*, measured local temperature distributions in a tank in the absence of mechanical agitation when free convection heat transfer took place. This work was undertaken on the initiative of Prof. *Regenass* and his colleagues in the wake of the *Seveso* accident (1976). A thin thermal boundary layer was formed and free convection heat transfer correlations in various regimes and at various scales were established.

2.4.3. Mixing and the Selectivity of Fast Organic Reactions

Fig. 13 refers to the nitration of durenene (1,2,4,5-tetramethylbenzene) using a nitronium salt in nitromethane. The ratio of the two products mononitro- and dinitrodurenenes (MND and DND) is represented on the ordinate [7]. Despite the very much smaller rate constant for the second nitration relative to mononitration, more DND than MND was found in the product mixture after the addition of one mole of nitronium salt per mole of aromatic hydrocarbon. Two further anomalies are evident in *Fig. 13*. As the concentration level increases, whilst the stoichiometric ratio is held constant, more DND and correspondingly less MND are formed. When the mixing intensity is decreased in changing from a mixing head (a three-way valve and a capillary) to a laboratory magnetic stirrer in a small flask, more DND was again formed.

These results of *Zollinger* and coworkers (1969) coincided with my 'Antrittsvorlesung' at the *ETH* (1969) where I discussed such mixing problems. Two years later, similar findings from *Merck*, USA, on the iodination of the amino acid *L-tyrosine* [8] emphasized the role of mixing as a relevant factor in determining the product distribution of some rapid multiple reactions. One problem at that time was to find a set of sufficiently simple test reactions to enable safe technical scale investigations.

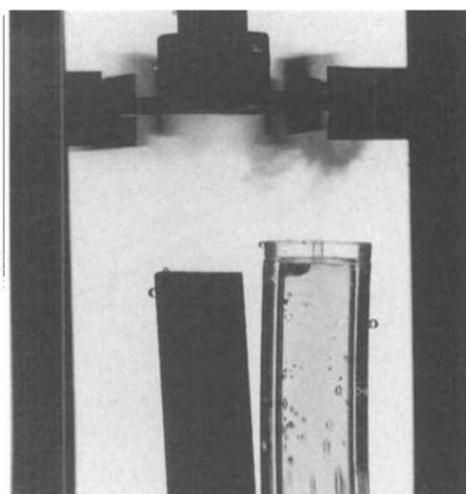


Fig. 14. Diazo coupling in the zone of high turbulence near the turbine impeller

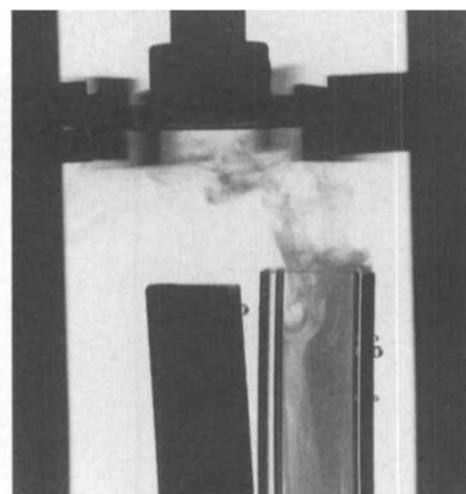
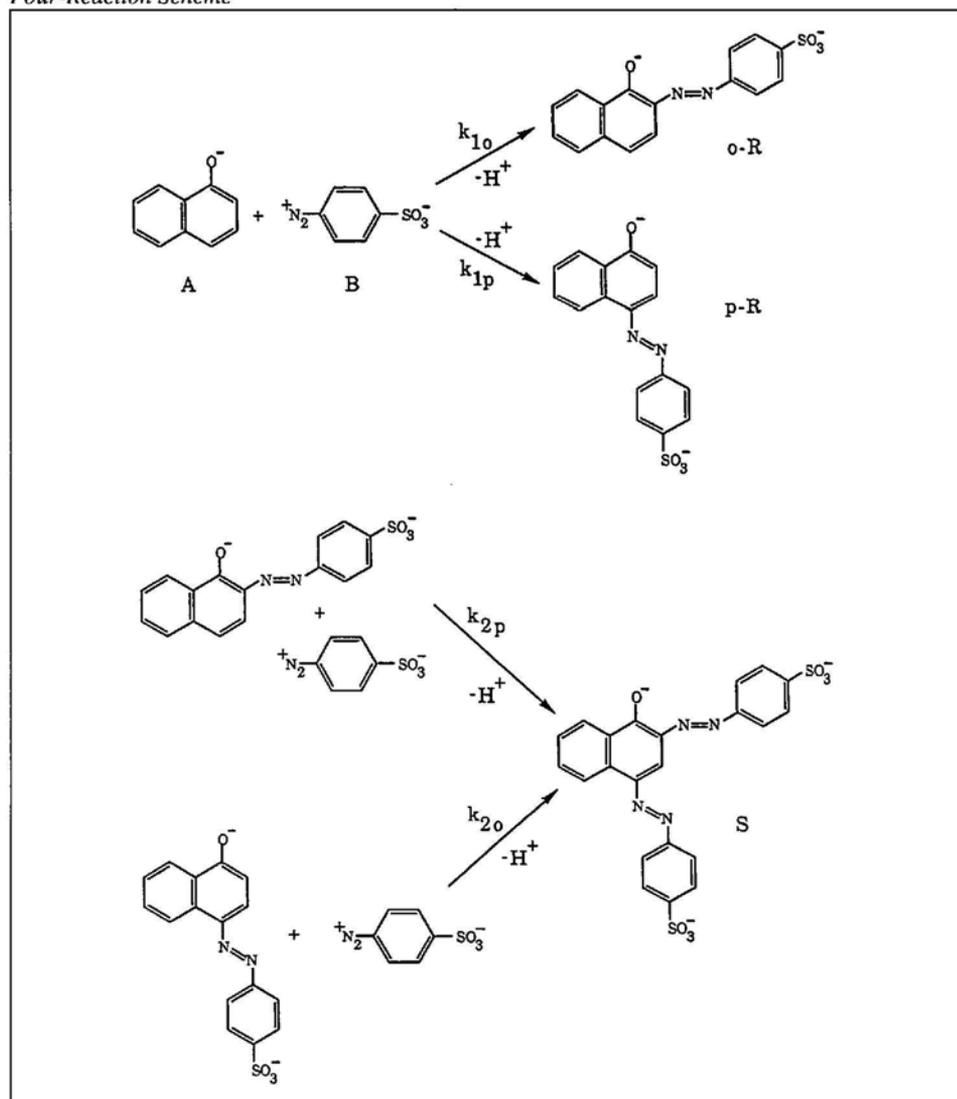


Fig. 15. Diazo coupling in the zone of weak turbulence within the feed pipe

Scheme 2. Diazo Coupling between 1-Naphthol and Diazotized Sulfanilic Acid Represented by a Four-Reaction Scheme



Kurt Suter (1974), now *Sandoz*, following *Peter Eugster* (1973) with Profs. *Rys* and *Zollinger*, worked with the bromination of resorcin, where the distribution between the 2,4- and 4,6-dibromoresor-

cins was particularly sensitive to several mixing parameters. The reactive substrate 1,3,5-trimethoxybenzene was also brominated and showed high sensitivity to mixing. For mechanistic and technical rea-

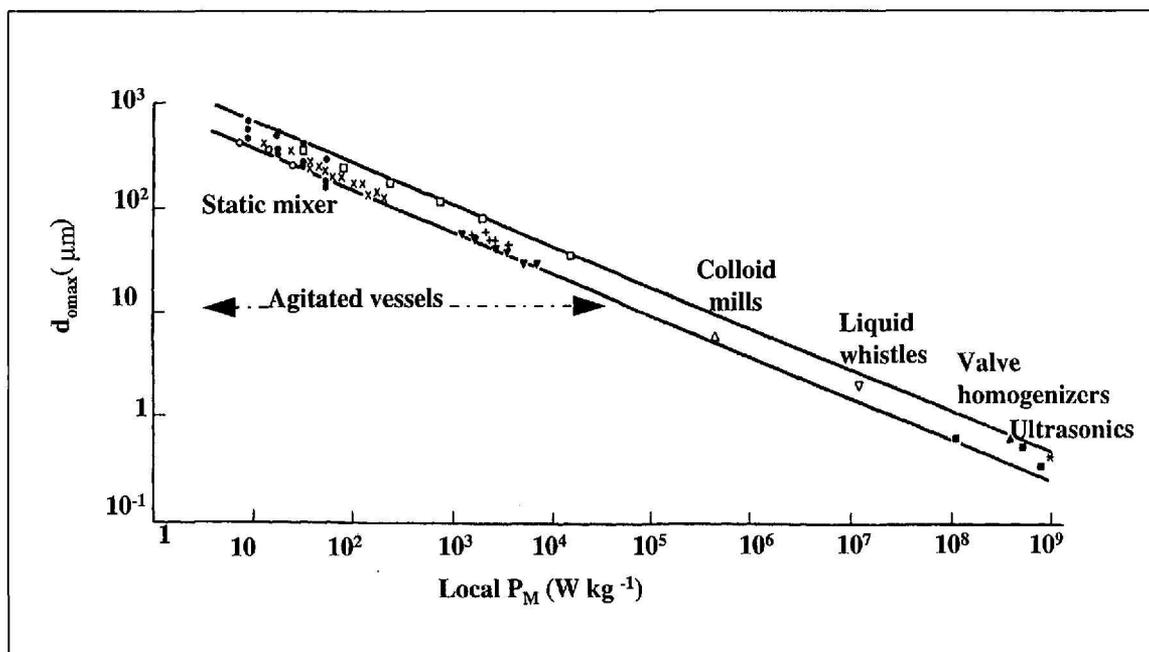


Fig. 16. Maximum stable drop sizes in dilute emulsions as a function of the turbulent energy dissipation rate

Table. Maximum Stable Drop Sizes (ca. 1 h; 0.2 vol% silicone oil (DC 100): 960 kg m⁻³; 0.142 Pa s; 0.045 N m⁻¹)

u_o [m s ⁻¹]	d_{max} [μm]	E_{max} [W kg ⁻¹] (dispersion)	E_{max} [W kg ⁻¹] (chemical)
4	240	900	640
6	160	3 100	2 200
8	120	7 400	5 100
10	100	13 500	10 000
12	85	22 000	17 000
14	80	27 000	27 000
16	70	42 000	41 000

sions further work on bromination was however stopped. An important reason was the discovery by *Urs Mörgeli* (1976) of the cleaner and safer diazo coupling between 1-naphthol and diazotized sulfanilic acid. *Scheme 1* gives a simplified representation (using two reactions) conforming to the scheme



Convenient reaction conditions were now attained – room temperature, pH=10, water as solvent, low reagent concentrations, fairly low toxicity. Technical studies were then undertaken, e.g. location of feed point, stirrer speed, rate of feed addition to a semi-batch reactor, comparison of continuous and semi-batch operations.

This coupling was published in 1977 and was found to be in use in American

industry two years later as a technique to study how mixing influences selectivity. Since then it has been refined and work has proceeded in three broad fields a) reactors, b) modeling, and c) test reactions.

a) Reactors

Many mixers are on the market, but few have been characterized for getting a high product yield from fast multiple reactions. Studies in Zürich included.

Stirred tank reactors: *Felix Kozicki* (1980), *William Angst* (1982), *Peter Dell'Ava* (1985), *Clemens Hilber* (1987), *Suzanne Thoma-Hubis* (1989), *Serge De Boni* (1993), and *Shengyao Yu* (1993).

Tubular reactors: *Georg Schwarz* (1981), *George Tovstiga* (1986) – with simple tubes –, *Horst Maire* (1990), and *Joachim Lenzner* (1991) – with tubes containing mixing elements to enhance turbulence. Centrifugal pump: *Oliver Bolzern* (1984).

Rotor-stator mixer: *Martin Studer* (1989). Grid turbulence: *Marcel Lips* (1990). Turbulent jet: *Bertin Zimmermann* (1993).

It would be quite unrealistic to want to summarize the results of these investigations. Two photographs show however the value of simple flow visualization in locating a reaction zone: they are taken from *Kozicki*. *Fig. 14* refers to the start of the diazo coupling in a stirred tank. Two wall baffles, a turbine impeller, and the feed pipe for diazotized sulfanilic acid can be seen. The feed pipe had a diameter of 8 mm and was fitted with a plastic cap having a 1-mm hole. The formation of dyes in the immediate region of the turbine is evident. *Fig. 15* is identical to *Fig. 14* except that there is no cap on the feed pipe. Obviously, reaction occurred within this pipe where the turbulence level is low causing the formation of much more bisazo dye than when coupling takes place near the turbine. The relatively large opening of the feed pipe (8 mm) permitted backmixing of the alkaline naphthol from the tank and pre-reaction within the feed pipe. In some situations, e.g. nitration this effect could represent a hazard.

b) Modeling

An important idea in modeling is to compare characteristic times for mixing and reaction. When a mixing time is on the order of or shorter than a suitable reaction time, an influence of mixing on chemical reaction is to be expected. Several problems arise in implementing this idea. For multiple reactions there are many chemical time scales. Depending upon the most relevant mixing step, e.g. bulk blending or mixing on the molecular scale, there are different time scales, e.g. for macromix-

ing and for micromixing. Much is now known about such matters and has been well confirmed experimentally. Valuable contributions were made by a series of post-doctoral visitors, who often worked on problems whose outcome was too uncertain for a doctoral student. An intense collaboration since 1982 with Dr., now Prof. Jerzy Baldyga, Technical University, Warsaw, accelerated and improved the evolution and validation of models to represent the coupling between mixing and reaction.

c) Reactions

Helen Gablinger-Salzmann (1988), now von Roll, further investigated the fall in pH which occurs at the reaction site when the buffer capacity – a carbonate/bicarbonate mixture is employed – is insufficient. For example, it is now possible by computer simulation or experimentation to determine the buffer concentration needed to maintain a given pH at the molecular scale even when some reagents are partially segregated.

Hilber's kinetic and analytical studies showed that the two reaction scheme in Scheme 1 is too simple. Maire and Lenzner then determined the kinetics of the four reactions in Scheme 2 and worked out quantitative analytical methods.

By adding 2-naphthol to the reaction corresponding to Scheme 2, Lenzner created a competition for the diazonium ion between the two naphthols, which significantly accelerated the whole system. The faster such reactions become, the more intense is the turbulence which can be studied. 'Intense' can be expressed by the rate of dissipation of turbulent kinetic energy to heat [W/kg]. Some rough values are:

pipe flow and centrifugal pump	1,
stirred tank (depending on position)	0.1–10,
static mixer	100–500,
rotor-stator mixer;	
turbulent jet	1000–10000.

With the extended diazo couplings, whose product distribution is still sensitive to mixing up to 100 000 W/kg, mixing and fast reactions in jets could be studied.

Yu developed a system of two competitive reactions, which is slower than the couplings in Scheme 2 and suitable for rather weak turbulence, e.g. in some designs of stirred tank. For instance, concentrated sodium hydroxide was added to a solution of hydrochloric acid and an ester (ethyl monochloroacetate). In the absence of a mixing constraint only neutralization would occur. Ethanol is however detected in the product mixture and the hydrolysis

of the ester signals imperfect mixing of the reagents. De Boni worked with similar reaction systems.

New reaction systems continue to be developed. Each one offers a window where something can be learned about the coupling between mixing and chemical reaction. By comparing such measurements with theoretical models the opportunity to generalize such knowledge opens up.

One technologically relevant generalization is to link the reaction studies to the dispersion of immiscible liquids. Denoting the turbulent dissipation rate by E [W/kg], Kolmogorov [9] showed that the size of the largest drop d_{max} , which is stable in a turbulent flow, varies with E according to

$$d_{max} \sim E^{-0.4}$$

Fig. 16 illustrates this, using logarithmic scales, for an interfacial tension of 30 mN/m over an eight-decade variation in E [10]. (The symbol P_M in Fig. 16 is identical with E .) The solid lines have a slope of -0.4 .

Using an extension of the above theory to viscous drops, due to Davies [10], the simultaneous coupling of 1- and 2-naphthols was run in a turbulent jet using nozzle velocities in the range 4–16 m/s. From the measured product distributions, the corresponding maximum values of the turbulent energy dissipation rate in the jet (E) were calculated from the theory of micromixing. In an entirely separate set of experiments a small amount of viscous silicone oil was dispersed and from the measured maximum stable drop size and Davies' extension of Komogorov's theory, E was again calculated. The Table shows some of Zimmerman's results and a reasonable comparison of E values for well developed turbulence.

Although the main thrust has been to investigate how mixing influences the selectivity of fast multiple reactions, the understanding of turbulence thereby gained allows apparently unrelated matters to be better understood.

2.4.4. Conclusions

I should like to draw some personal conclusions from all this activity.

1. Both doctoral students and I have learned much from our joint investigations. It was a stimulating learning experience.
2. Most doctoral students felt that the approach, with its blend of theory and experiment (or modeling and practice), was about right.

3. As already implied in Sect. 1, the personal and professional development of doctoral students was, in my experience, significant and valuable.

What were the weakness and the strength of the above-mentioned doctoral students? Many were probably too pragmatic, in the sense that if something could not be directly applied to their own research, then they were often not interested in it. This remark tended to apply more to speculative, theoretical considerations. Of course a pragmatic attitude has also benefits, but a little less caution could sometimes have brought interesting developments. Among the strengths I would mention a) frequent, although not universal, collaboration and learning from each other in a small team, and b) fine practical abilities, where doctoral students were motivated by practical problems and worked hard and with real flair to solve them. They would, I thought, be confronted with complex situations and challenging problems in their industrial careers and I had, and have, confidence that they would deal well with them. For myself, it was a great privilege to work closely with such fine young people, who I shall not forget.

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