PROCESS DEVELOPMENT AND SCALE UP: 1. PROCESS DEVELOPMENT STRATEGY AND METHODOLOGY

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ABSTRACT

This paper reviews research and development activities that are generally required to develop a catalytic process from a laboratory concept into an industrial scale plant, analyzes the nature of the development activities, their tasks and objectives. The course to be taken and the methodology to be adopted are discussed, and the role of process development tools such as mathematical models, reactor units of various sizes, and cold flow engineering mock ups are reviewed. Some general guiding principles are mentioned to maximize the effectiveness of the development program in terms of time and costs.

INTRODUCTION

In contrast to pure scientific (fundamental) research, which is an activity that primarily aims at furtherance of scientific knowledge, so-called applied research and development (R & D) has a more practical objective, which may be the improvement of the technology in a certain field or the introduction of new technology. In the field of industrial chemistry, the improvements or innovations may pertain to products as well as to processes which are applied to manufacture these products.

For an industrial enterprise which carries out R & D in a part of its organization or financially supports R & D extra-murally, the results of this R & D are not the goals in themselves, but are just means to ultimately achieve a competitive advantage. Therefore, it is important that R & D is carried out in a cost effective way to maximize the chances of success, not only in a technical sense, but in a commercial sense as well. In this context, selection of the appropriate topics for R & D and formulation of suitable targets are as important as carrying out the R & D in the best possible way so as to minimize expenditure of money and time, ensure the best utilization of resources, both material and talent, and maximize the chances of success.

A discussion of R & D strategy in terms of defining the optimal volume and mix of R & D topics against business objective is outside the scope of this paper. Instead, the R & D activities themselves, and in particular those that constitute the development part, are the subject of discussion. The discussion will focus on development activities related to chemical manufacturing processes and leave out developments aimed at innovations in the sphere of products.

A number of papers and monographs have been published on the subject of process development, e.g. by Blasz (1985) and by Euzen *et al.* (1993). Scale up problems have been treated by Hoften *et al.* (1990), by Palluzi (1990) and in a recent textbook by Thoenes (1994). Laboratory reactors have been discussed by Difford and Spencer (1974), Weekman (1974), Pratt (1987), Worstell and Ginestra (1993) and Palluzi (1994). These papers deal with

catalytic reactor types and aspects such as sizing, applications, limitations and laboratory safety.

In the present paper, we will discuss the role of the various elements in the overall context of the development program, rather than presenting a detailed discussion of a specific element.

PLACE AND ROLE OF PROCESS DEVELOPMENT

Process development is commonly seen as a segment of the chain that links a new process concept with its commercial exploitation. This new concept may concern a new reaction, a new manufacturing route for a desired chemical, a new reactor principle, etc., and may arise out of theoretical considerations or experimental investigations. Following a phase of exploratory investigations aimed at further defining the feasibility and merits of this new concept, a stage may be reached where the technical and economic viability has been broadly established and where the attractiveness of the new idea is considered to be sufficiently great to warrant its further development to commercial reality. In this context, the task of the process development activities is to define further the process conditions and boundaries, to find and remove possible practical obstacles, to demonstrate the technical and economic soundness of the process, and to gather the necessary data to allow designing and implementing the commercial process without taking unduly large risks. In this development phase, not only purely technical factors, but also economic, operational, safety, and environmental factors have to be considered.

The successive stages that make up the links of the chain between a new concept and its commercial application are shown in Figure 1. The primary input can be a new element from science or an identified business need. Each next stage from a preceding stage yields an output that feeds into the next stage. The ultimate output is the commercial application. The figure also shows the dividing line between "Research" and "Development",

following a common notion about the differences between these two categories of activities.

A shortcoming of the model of process R & D as depicted in Figure 1 is that each phase is clearly delineated and isolated from the others, whereas in actual practice the total activity is much more a continuous one with no or only vague borders. The absence of clear borders also pertains to the distinction between "R" and "D". Furthermore, all stages carry equal weight and no distinction is made in volumes of money and effort, nor in the differences in the nature of the efforts.

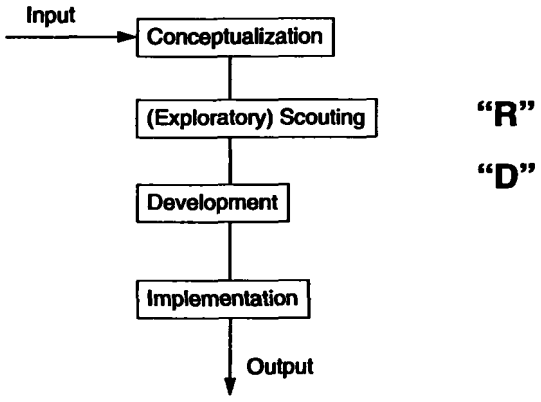


Fig. 1: Common model of process R & D showing the sequence of stages from concept to commercial implementation.

A model of Process R & D which is a better illustration of the continuity and the differences of the various stages is shown in Figure 2 (a) and (b). Figure 2 (a) shows a *diverging* activity, which is appropriate if one considers the total R & D process from the point of view of money to be spent or at risk. In the conceptual and exploratory phases, small amounts of money are generally involved, whereas in the development and implementation phase large sums of money are generally needed. The same applies to the number of people: in many cases a lone researcher or a small team carries out the scouting research whereas large development and design teams carry the

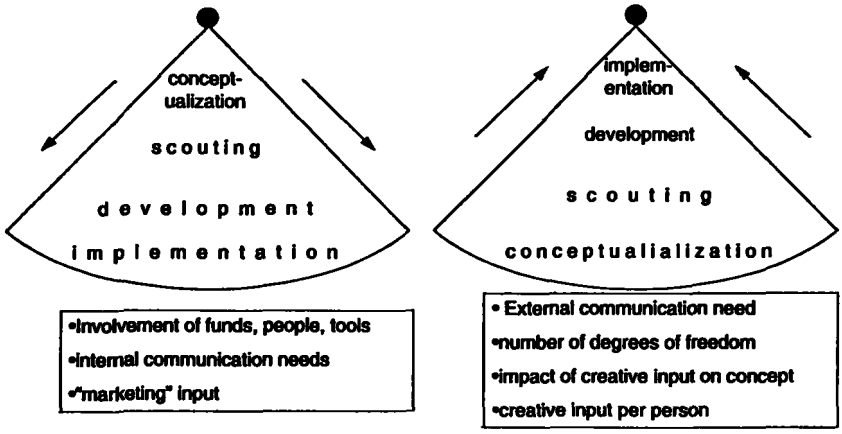


Fig. 2(a) and (b): Alternative models for process R & D.

project through the next stages. The larger number of people involved in later stages of the project has to do with the need to involve more disciplines: whereas the discipline of chemistry may be the only one involved in an initial stage, involvement of specialists in disciplines such as reactor engineering, separation science, corrosion research, mechanical engineering, process control, safety, process economics, etc., may be needed in a later development stage. Additionally, in the design and implementation phase civil engineering and utility engineering are generally also involved. This growing number of people places a greater emphasis on effective communication within the team and on project management. There is also growing need for communication with other groups within the company, in particular with the business sector, to ascertain that the objectives of the R & D work match with business objectives, but also with patent officers for protecting know-how and ascertaining freedom of operation, and with health and safety officers for making environmental impact statements and for obtaining working permits.

Figure 2 (b), on the other hand, depicts the R & D activity as a *converging* one. This way of representation is the proper one if one considers the R & D project from a scientific point of view. In the scouting phase, targets are seldom clearly defined and there is a wide field in which

to seek for potentially interesting concepts. There is a wide choice of problems for which solutions may be sought, and novel solutions may be found in other than the established areas. The process of idea generation is generally promoted by extensive contacts with scientists in other fields, including fields outside the company's expertise. As the project progresses, the number of choices and the degrees of freedom diminish, targets become more clearly defined and the technical and economic boundaries in general become more strict. Thus, as the project progresses, there is an increased focus on the process to be commercialized.

The different nature of the activities in the various phases, as discussed above, makes it clear why in principle different people are needed in the exploratory research and in the development phases. In the exploratory research phase researchers should be able to overview a wide field of science to make appropriate choices, unhindered by conventional wisdom. The need for and the impact of individual creativity is very great. In the development phase, the players should be target-oriented and operate effectively as a team. There is in general less scope for individual creativity within the boundaries of the project.

The above sketch of a R & D project pertains to the commercialization of a novel concept leading to a first-of-a-kind process. Although such a revolutionary innovation does occur from time to time in the chemical industry, most innovations are more of an evolutionary type, i.e., they represent improvements of existing technology without necessarily greatly departing from it. For these latter type of innovations, the basic ideas which have to be developed further do not generally arise in isolation from new science, but are born out of the need to amend the shortcomings of an established process which have been recognized by practical experience. In these cases, the task of process development is also to provide a link between the work of commercial operation and that of science and technology. In this latter task, which is less generally recognized but at least as important as the one described above, process development is not so much concerned with scaling-up and integrating of the different disciplines involved in the commercialization of the new process, but in reducing the difficulty in the

complex practical world into a problem in the relevant technical area for which solutions can be sought with the appropriate scientific approach in a laboratory. Hence, in this case process development is a differentiating and scaling-down exercise, rather than a scaling-up and integrating one. In this view, innovations in process technology can be seen as a cyclic process, as shown in Figure 3. This figure illustrates that scaling-down is also an important objective in process development; this aspect has been discussed in some detail by Sie (1991) for scaling down of trickle bed processes.

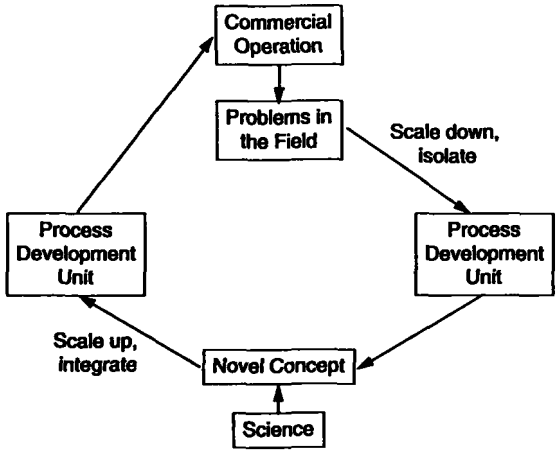


Fig. 3: The cycle in the development of new or improved process technology.

TASK AND OBJECTIVE OF PROCESS DEVELOPMENT

Although there is no sharp boundary between the later stages of exploratory research (focused exploratory research) and the onset of process development, it will be assumed that the decision to embark on a development project for a new process is taken when the technical and economic contours of the process have been defined in the exploratory phase, i.e., the underlying basic chemistry in terms of reaction mechanism, kinetics, and thermodynamics have been established and there is also

preliminary information on parameters which establish the technical and economic feasibility of the process. These parameters include reaction conditions, feedstock and product, type of catalyst, its activity, selectivity, life and regenerability, feedstock and other material requirements and product yields which are known in broad terms from the outcome of experiments in the exploratory stage or from intelligent guesses. There is also some preliminary assessment of the economic attractiveness of the process based on feedstock requirements and costs and product yields and prices. While this may seem a rather complete set of information it is seldom sufficient to form the basis for a decision to embark on a commercial venture at acceptable risk and to design a commercial plant. Table 1 lists some differences between how a reaction is studied in a preliminary stage in the laboratory and how a commercial process is carried out in practice.

The task of process development, then, is to make the design and implementation of the commercial process possible by firming up the data and by providing more precise information on optimal choices of process para-

Table 1

Some differences in process characteristics as studied in the laboratory in a scouting stage and in commercial operation.

Scouting experiments	Commercial process
batch or semi-batch	continuous preferred usually except in multi-product manufacture
isothermal	adiabatic or with internal heat exchange
well-mixed or plug flow	complex flow pattern
simple feed inlet	special distributors or injectors
once-through	recycle of streams
no heat integration	process flows or solids as heat carriers
limited duration	runs lasting up to a year or longer
defined conditions	operational upsets possible

meters for actual feedstocks, catalysts, reactors. An important difference from investigations in the previous exploratory stage is not only that real feedstocks and catalysts have to be investigated in a reactor which is representative of the commercial reactor envisaged, but that the various elements of the process have to be studied in an *integrated* manner, at least in the ideal case. In addition, aspects such as feed pretreatment, heat management, product work-up, process stability, start-up and shutdown, process control schemes, etc., have to be investigated or at least considered. The various aspects which need attention in the process development phase are listed in Table 2.

Table 2

Aspects to be considered in the development of a novel catalytic process. Economics are not listed, but represent a factor of overriding importance throughout.

- Reaction
  - Kinetics
  - Thermodynamics
- Catalyst
  - Formulation
  - Preparation technique
  - Base material choice
- Feedstock
  - Composition, impurity levels
  - Physical properties
  - Feed pre-treatment
- Reactor
  - Reactor type
  - Hydrodynamics and mass transfer
  - Heat transfer and temperature control
  - Materials of construction
- Process conditions
  - Optimal temperature, pressure, space velocity
- Product work-up
  - Definition of separation steps
- Process line-up
  - Recycles
  - Heat management
  - Instrumentation
- Catalyst regeneration and disposal
- Waste management and treatment of effluent streams

COURSE AND METHODOLOGY IN PROCESS DEVELOPMENT

Before embarking on a major development project for a new process, generally a plan should be made which defines the objectives and means to reach them, with estimates of the manpower and money needed as well as dates of major milestones and final completion. Such a plan serves to weigh the costs against potential future benefits, and allows the necessary preparations to be made in time. However, in actual practice it is seldom possible to follow the projected course in a steady, straightforward way. Since new technology is aimed at, which in many ways may involve step-outs from established practice, there are usually unforeseen findings. These surprises, both unpleasant and pleasant ones, necessitate readjustment of the course to be taken or can even raise the question whether continuation of the work is at all warranted. Hence, rather than a straightforward exercise, process development is generally an iterative process, as can be seen in Figure 4, which shows the course of an R & D project from laboratory finding to commercial plant design.

The necessity for regular check points during the course of a project with

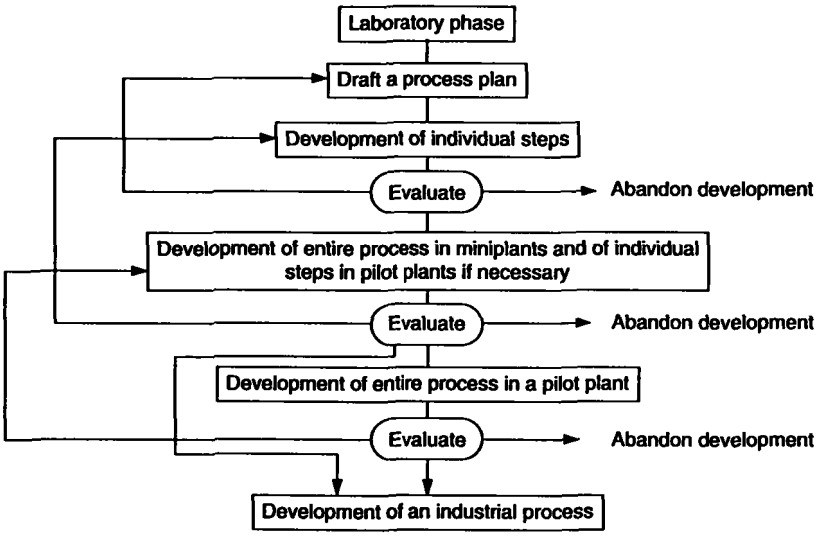


Fig. 4: The iterative nature of process R & D according to Vogel (1992).

re-evaluations whether to continue or to stop has to do with the escalating costs during the lifetime of the project, as has been mentioned before. The increase in R & D costs is illustrated in Figure 5, which shows an increase by an order of magnitude for each successive step. This makes it important to reduce uncertainties as much as possible in earlier stages where experiments are done on a small scale in relatively cheap flexible equipment, rather than in later stages with less flexible and more expensive equipment. This strategy is in line with the adage "make your mistakes on a small scale and reap your profits on a large scale".

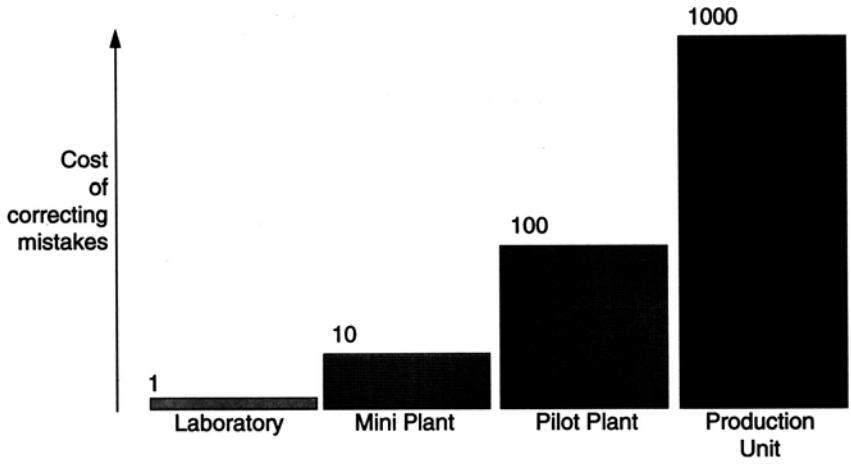


Fig. 5: Increase in costs in successive stages of process R & D (after Vogel, 1992).

The increase in size of the equipment which is generally used in the successive stages of process R & D is illustrated by Figure 6.

The increase of the capacity of equipment used in successive stages of process R & D shown in Figure 6 pertains to a typical case, but the scale factors are by no means the same for different types of equipment. Maximum factors for reliable upscaling as advocated in the literature are listed in Table 3.

Aside from the increase in size of the equipment in successive stages of process R & D, there is also an increase in the complexity, as mentioned

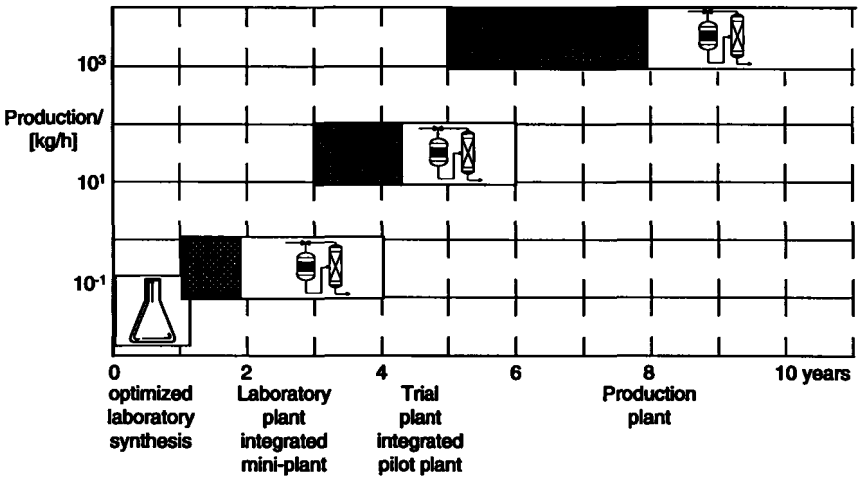


Fig. 6: Increase in the capacity of typical equipment used in successive stages of process R & D according to Vogel (1992).

Table 3

Maximum factors for reliable upscaling (Krekel and Siegmann, 1985)

Reactors		
	Multi-tubular fixed bed reactor	> 10000
	Homogeneous tubular reactor	> 10000
	Homogeneous stirred tank	> 10000
	Bubble column	< 10000
	Gas-solid fluidized bed	50 - 100
Separation processes		
	Distillation and rectification	1000 - 50000
	Absorption	1000 - 50000
	Extraction	500 - 1000
	Drying	20 - 50
	Crystallization	20 - 50

earlier. The problems to be studied are no longer confined to the field of chemistry as may be the case in an initial exploratory stage of process R & D, but may involve physics and mechanics as well in the development stage. In the ideal situation these different aspects should be studied in a closely integrated manner as occurs in practice. However, since physical and mechanical aspects can often only be studied in a representative way at scales not too different from that of commercial plants, a study in a fully integrated manner would imply using a very large pilot plant or demonstration unit for these studies. The very high costs and limited flexibility of such a unit is generally a deterrent to this approach, except perhaps at a final stage of the development program, when all or most of the process parameters have been fixed and when proving the integrated system in a demonstration unit is deemed essential.

A way out of this dilemma is to separate the more chemistry oriented studies (which can be done on a substantially reduced scale compared to practice) from the study of the physical and mechanical aspects of the process, which often needs to be done on a large scale to be representative for practice. Thus, conversions of actual feedstocks with real catalysts under representative conditions of temperature, pressure and space velocity are studied in relatively small reactor systems (e.g., bench-scale units) that are considerably scaled-down versions of the (envisaged) commercial plant, whereas hydrodynamics, mass and heat transfer, residence time distributions, attrition of solid particles, etc., are studied on a representative, not necessarily small scale in so-called cold models using fluids and solids that are more easily available and more convenient to use. These cold models, together with the hot process units and computational models, are the main tools in process development. Their main role is specified in Table 4.

This separation between the more chemistry oriented "process research" and the more physics/mechanics oriented "engineering research" and their interaction is shown in Figure 7. This figure shows how the information generated in the separate areas can be integrated by means of mathematical models. These models, for instance, describe physical aspects (hydrodynamics, mass transfer, heat transfer, etc.) or chemical aspects (kinetics,

Table 4

Roles of the three major tools in process development

Tools	Main field of study
Reactor units, pilot plants	Chemical kinetics
	Catalyst development
	Product yields
	Operational procedures
	Process demonstration
	Problem recognition
Cold flow models, engineering test rigs	Hydrodynamics
	Multi-phase behaviour
	Physical transport
	Geometric effects
Computational models	Correlation and prediction
	Translation and integration
	Sensitivity analysis

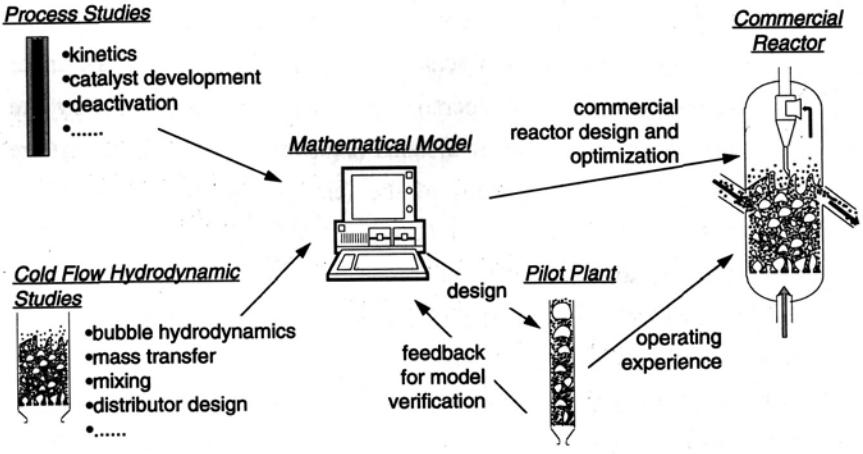


Fig. 7: Interaction between process and engineering research in process development.

yields, catalyst deactivation, etc.). when based on pertinent fundamental principles, such models allow extrapolations outside the area where the experimental data can be generated and thus they can be used to link the engineering information obtained in cold flow models that are reasonably

scale representative but different in temperature, pressure and nature of the materials, with the process data from reactor data systems that are representative in the latter respects but which deviate considerably in scale. This way of integrating can in many cases form a sufficient basis for a definition of the desired process. Further confirmation of the integrated operation of the process can, if deemed necessary, be obtained in studies using a larger and/or more complex pilot plant, which can be designed on the basis of the information obtained in the above studies. Depending upon whether flexibility in varying process parameters or true simulation of the integrated process is considered more important, this pilot plant can be either a relatively small, flexible pilot plant or a near-commercial size demonstration plant which is generally designed on the basis of a rather narrow process specification.

The guiding principle which determines the approach to be followed and the size and complexity of the process development tools to be used is that the information obtained should allow commercialization of the process without unduly large risks and that this information should be generated in the most cost effective manner. The degree of optimization and precision in defining process parameters that is required and the time and effort needed for the development phase will differ from process to process and no generally valid recipe for process development can be given.

COMPUTATIONAL TOOLS

Computational tools, which can be considered as software tools, are relatively cheap means to obtain information, as compared to the Process Development Units that represent the hardware tools to be discussed later. Computational tools include databases on properties of products, thermodynamics, ecological and toxicological information, etc. They generally provide fast access to the required information and can be used to determine data where these are lacking by using established correlations as a substitute for experimental determinations. Thus, maximum use can be made of existing knowledge and unnecessary experiments are avoided.

As already mentioned above, dedicated models which describe certain aspects of the process are profitably used in process R & D. They can be quite specific to the process under study (e.g., kinetic data pertaining to certain types of feed molecules and catalysts) or have a wider range of applicability (e.g., pressure drop correlations, heat transfer models, etc.).

Generic models that are capable of describing various processes include process simulation models or so called flow-sheeting models. These models generally contain the above data bases and general correlations and allow simulation of individual units (reactor units, separation units, etc.), as well as the integration of these units in a process line-up with internally consistent material and heat balances. These models are particularly useful in the process design stage, where the effects of changes in process line-up can be studied. Examples of available models or computer programs belonging to this category are listed in Table 5.

Table 5

Examples of process simulation models

Steady-state simulations	
Aspen Plus	Aspen Technology, Inc
Chemasim	BASF AG
Chemcad	Chemstation Eng
Design II	Chemshare
Flowpack	ICI
Hysim	Hydrotech Ltd
Pro II	Simulation Sciences Inc.
VT Plan (Conti)	Bayer AG
Dynamics simulations	
Chemadyn	BASF AG
Diva	University of Stuttgart
Satu	Hoecht AG
Simusolv	Dow
Speed-Up	Aspen Technology, Inc

In optimizing the process, particularly in the design stage, various aspects other than purely technical ones deserve proper attention. Special

attention should be given to the following important E's:

Economy (costs)
Efficiency
Energy
Environmental
Excellence (quality)

With respect to the environmental aspects, one may keep in mind that "Waste Reduction Always Pays" (the WRAP principle as advocated by Dow). In striking a proper balance between quality and costs, one may also remember the adage that "The bitterness of low quality remains when the sweetness of low costs has long been forgotten".

The advent of more powerful computers has increased the importance of computational tools. This does not only apply to mechanistic models and hydrodynamic models where the increased computer capacity has led to a gradual displacement of empiricism and conventional chemical engineering correlations by more fundamentally based, *ab initio*-type calculations, but also to the process simulation models which are continually refined and include both technical and economic aspects of processes.

PROCESS DEVELOPMENT UNITS

Purpose of Process Development Units

Process developments in a wider sense than usual comprise all major dedicated hardware tools in the laboratory that play an essential role in the development phase of a process. Different units may serve different objectives which can be distinguished as follows:

- Study of an isolated specific problem. This problem can be either a chemical one, viz., catalysis and kinetics, or a physical one, viz., reactor physics.
- Study of the integrated process by simulating the commercial plant on a laboratory scale.
- Producing representative material.

In the study of a catalytic process, the catalysis and kinetic studies generally aim at obtaining an optimal catalyst for the process in terms of activity, selectivity and stability, at finding an optimal set of process conditions, at defining suitable feedstocks and their specifications, and at establishing catalyst pretreatment and regeneration procedures (if required). These studies may usually be carried out in rather simple reactors of relatively small size, e.g., micro- or bench-scale reactors. The reactant stream may flow through the reactor in a single pass. The characteristics of the conversion reaction determine the preferred type of reactor (Krishna and Sie, 1994) and the type of laboratory scale reactor may be chosen accordingly (e.g., a stirred, fixed-bed or fluidized bed reactor).

The study of the physical aspects of the reactor type chosen, e.g., hydrodynamics, dimensions, geometry of internals, attrition of solids, etc., may usually be carried out with model fluids and solids at a representative scale in cold-flow models or engineering mock-ups.

Study of the integrated process generally aims at obtaining information on the interaction of process elements such as reaction and separation, on the effect of recycles, build-up of impurities, etc. With process streams having representative compositions, information may be obtained on aspects such as corrosion, fouling, etc.

Production of relatively large quantities of representative materials may be required for developing a market for new products, which may involve field trials on the application. In such a case a large, but not necessarily very complex reactor unit may be required.

Types of Process Development Units

Process development units serving as tools to accomplish the above objectives may be distinguished as follows:

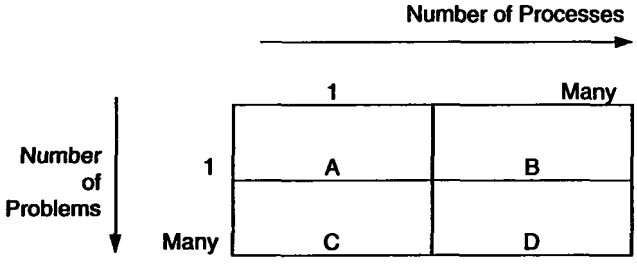
- According to the problems to be studied one may distinguish between specific test units in which one or a limited number of related aspects are investigated (a reactor unit or an engineering test rig), and an integrated pilot plant for simultaneous study of various process aspects and the

interdependence of various elements of the process.

- According to the number of processes for which the unit can be applied, one may distinguish between a dedicated unit which is only suited for the study of one specific process or a set of very similar processes, and a multipurpose unit which can be used to investigate a variety of processes which can be quite dissimilar in their chemistry and line-up.
- According to the continuity of application a distinction can be made between "one-shot" pilot plants which are only used during the development of a specific process and serve no purpose thereafter, and (semi-) permanent installations which are used to study recurring problems over a large number of years.

The above differences in the role of process development units will determine how these units are best constructed, e.g., the temporary nature of a one-shot pilot plant makes it less important to design for long-term durability. To study dynamic aspects of an integrated process in a pilot plant it is important that dead volumes in vessels and tubes do not give rise to unduly long response times. A multi-purpose pilot plant will be designed to be flexible in order to cope with the needs of different processes.

Figure 8 presents a matrix in which process development units can be



Examples:
A: MAT unit for FCC catalyst testing
B: Bench-scale trickle flow reactor
C: Integrated pilot plant for a novel process

Fig. 8: Classification of process development units according to the number of processes and the number of problems to be investigated.

classified according to the first two criteria, and mentions a few typical representatives of the classes that can be thus distinguished.

Size of Process Development Units: General Considerations

A general principle for choosing the size of the development tools in the different R & D stages is that they should be as small as possible without detracting from their essential purpose. This is because Small Scale generally has many advantages, including Savings, Safety and Speed. Hence it can be said that Small is Beautiful.

The money savings permitted by installing smaller scale units are not only due to their lower construction and installation costs, but also because in their operation less materials are consumed, see Figure 9. Lower material requirements are particularly important when unconventional feedstocks, catalysts, etc., have to be used since special preparation of these materials can be both money and time consuming when carried out on a large scale. Small equipment is also more economic because it places lesser demands on laboratory infrastructure: utility requirements (electricity, water, cooling water, steam, pressurized air, special gases, etc.) are lower while demands for building space, cooling and ventilation, storage of feedstocks and

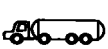
	Pilot Plant	Bench Scale	Micro Flow
Catalyst volume/[m^3]	0.01	0.00015	0.000008
Liquid rate/[m^3/h]	0.020	0.0003	0.000016
Gas rate/[Nm^3/h]	20	0.3	0.016
Monthly consumption of liquid/[m^3]	 14.4	 0.2	 0.012
Monthly consumption of gas/[Nm^3]	 14400	 216	 12

Fig. 9: Effect of scale of reactor units on consumables for typical hydro-processing studies.

products and disposal of waste streams are also less.

Decreasing the size of equipment generally enhances safety, which is particularly true for process studies involving high pressures, combustible or toxic reactants. This is illustrated by the data in Table 6, showing the intrinsic safety of very small scale process studies, viz., in a so-called nanoflow reactor.

Table 6

Potential hazards of a nanoflow reactor (200 mg of catalyst)

Type of hazard	Typical experiment	Effect
Fire		
by reactant stream	Paraffin isomerization	3 % of burning candle
Toxic gas emission		
CO from reactant stream	Fischer-Tropsch synthesis	20 % of burning cigarette
H_2S from inventory	Hydrotreating	5 ppm H_2S in 20 m^3 unventilated room
Explosion		
gas release	Fischer-Tropsch synthesis	20 % of punctured bicycle tyre
Detonation power	Fischer-Tropsch synthesis	less than small firecracker

An argument which is often put forward to advocate experiments on a relatively large scale is that a large scale is a prerequisite to obtain accurate material balances. This is not always true however. Modern techniques of weighing and mass flow determination can give very accurate results even with very minute amounts of material. Another argument is that the samples taken from the process stream for analysis significantly disturb the process when carried out on a small scale. However, with modern on-line measuring instruments this argument may not be valid since material is not necessarily withdrawn from process streams (e.g., in the case of on-line spectrometric analysis) or only very tiny samples are needed for the analysis, e.g., in case

of on-line gas chromatographic or mass spectrometric analysis. In fact, the latter techniques are demonstrations that one can handle minute amounts of material in a quantitative way.

Carrying out experiments on a small scale thus does not have to lead to less accurate results, provided adequate measures are taken and accurate working practices are adhered to. In fact, the need to adhere to accurate working practices can be considered to be an advantage of small-scale experimentation.

Factors Determining the Minimum Size of Process Development Reactors

The minimum size of process development reactors is determined by various factors. In the investigations of chemically oriented problems it is in principle possible to work on an extremely small scale since the number of molecules in very small samples is sufficiently great to comply with statistical laws. For example, a microgram of a compound with a molecular weight of 600 contains of the order of 10^{15} molecules. Catalysis studies involving determination of the activity, selectivity and stability of a catalyst, determination of the effects of catalyst formulation variables, determination of reaction kinetics, optimization of reaction conditions, feedstock evaluation, etc., can in principle be carried out on a very small scale. Indeed, fundamental catalysis studies are generally carried out with very small amounts of material: in studying a conversion over a single crystal face the catalyst surface may correspond to that in a microgram of a practical catalyst.

In more applied catalysis and process studies, however, a larger scale than in the above fundamental studies is required. This is because laboratory reactors generally have to be integral reactors achieving realistic conversions in order to obtain relevant selectivity data for the practical process. Hence, the laboratory reactor has to be representative of a practical reactor in terms of backmixing, catalyst contacting, etc. The limits to size reduction in these respects are discussed by Sie (1991) for the case of trickle-flow reactors as used in hydroprocessing studies.

A minimum scale of experimentation may also be dictated by the sample sizes required for analysis. Determination of the chemical composition or basic physical properties of samples by modern analytical techniques generally requires only small samples, i.e., of the order of 1 gram or even considerably less, as can be seen from Table 7. However, determination of performance characteristics by more traditional methods may require considerably larger samples, e.g., determination of octane and cetane numbers of oil products in engines, determination of pour points, viscometric characteristics, and thermal stability of oils, etc. When such determinations are crucial and have to be actually carried out, relatively large process developments are needed. However, in many cases it is possible to develop correlations for predicting these performance characteristics of products from more basic chemical and physical properties which can be determined from sensitive analytical methods so that large samples are no longer required.

The minimum size of the reactor in a process development unit is often determined by the requirement that the hydrodynamics in the laboratory reactor should not be radically different from that in a practical reactor, or that the effects of deviating hydrodynamics at least do not give rise to deviating results on the process aspect under study. For example, in the study of a fluidized-bed process fluid velocities should be at least equal to minimum

Table 7

Some analytical techniques requiring less than 1 g of sample

- Gas chromatography, including true boiling point simulation
- Liquid chromatography
- Mass spectrometry
- Infrared, ultraviolet and visible spectroscopy
- Nuclear magnetic resonance
- Microcoulometry
- Combustion mass elemental analysis
- X-ray fluorescence
- Atomic absorption spectrometry
- Refractometry
- Melting point determination
- Osmometric molecular weight determination

fluidization velocities for the catalyst under study. In the case of fixed-bed reactors the requirement of representative fluid velocities can often be relaxed by special measures (Sie, 1990).

The minimum size of the reactor in a process development unit may also be influenced by temperature profiles in the reactor. In fixed beds heat conduction through the bed is rather poor. Consequently, large fixed-bed reactors for endothermic or exothermic processes are most easily constructed as adiabatic ones, as in actual practice, whereas small reactors are conveniently designed and operated as isothermal reactors, see Table 8.

Table 8

Effect of fixed-bed reactor size on accuracy of temperature definition

	Small reactor	Large reactor
Isothermicity in radial direction	Good	Poor
Longitudinal isothermicity	Good*	Poor
Adiabaticity	Poor	Good
Representative dynamic response, e.g. runaway behaviour	Poor	Good

*) Provided there are sufficient individually controlled heating zones of proper design

Figure 10 is a drawing of an isothermal fixed-bed microreactor suitable for catalyst studies at high pressure and temperature. The reactor which can accommodate a bed volume of about 10 ml is heated by three independently controlled heaters so as to allow an even temperature in axial direction within about 2°C. The axial temperature profile can be measured by a sliding thermocouple in a central thermowell. Figure 11 shows a scheme of a typical microflow system for once-through reactions in the gas phase.

The difficulty of maintaining isothermicity in a large fixed bed reactor is that reaction heat has to be removed or supplied via the wall, and due to the poor heat conductivity in a fixed-bed this may give rise to unacceptably large

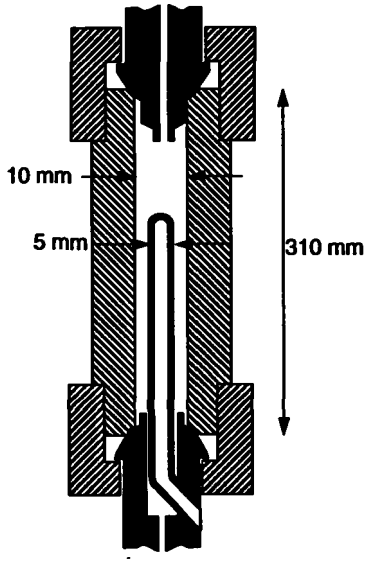


Fig. 10: Fixed-bed microreactor suitable for catalytic process studies at high pressure and temperature featuring a central thermowell which accommodates a movable thermocouple.

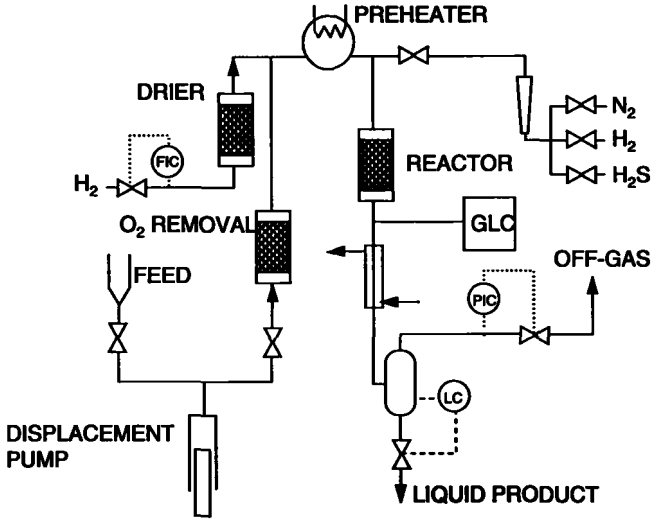


Fig. 11: Flow scheme of fixed bed microflow unit used for catalytic conversions with gaseous reactants (catalytic reforming) featuring on-line GLC analysis of the reactor effluent (Sie and Blauwhoff, 1991).

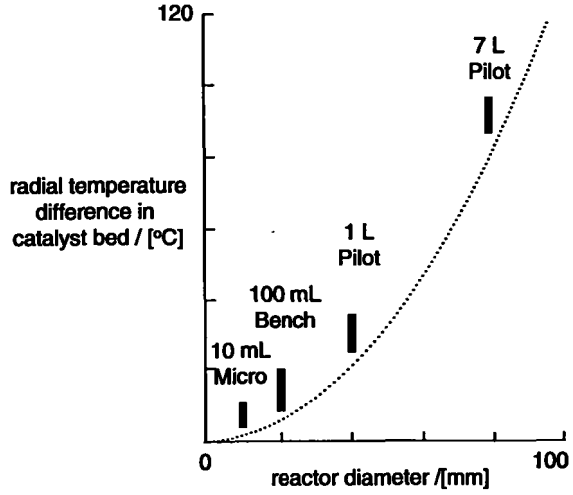


Fig. 12: Effect of reactor diameter on radial temperature profiles in fixed-bed reactors for hydrotreating.

radial temperature differences. This is illustrated in Figure 12 for a typical hydrotreating process.

The relatively easy heat flow in radial direction in narrow fixed-bed reactors, on the other hand, makes adiabatic operation much more difficult than in large reactors. In a true adiabatic reactor, there should be no radial heat flow and all heat generated or consumed by the reaction should be taken up or supplied by the process stream, resulting in an axial temperature profile. Although this situation is more difficult to obtain in small reactors, it is by no means impossible. An adiabatic minireactor can be constructed by mounting several independent heating sections along the reactor axis and controlling each section in such a way that at each axial position the temperature difference between the heater and the reactor wall is zero. Thus, there is no radial heat flux as in a true adiabatic reactor. Figure 13 shows a minireactor with a catalyst volume of only 10 ml, designed for adiabatic operation at a temperature level of 500°C with heat effects of the order of 5 W. Figure 14 demonstrates that this small reactor is indeed capable of simulating adiabatic operation with acceptable accuracy.

Although small adiabatic reactors can be constructed, they require more

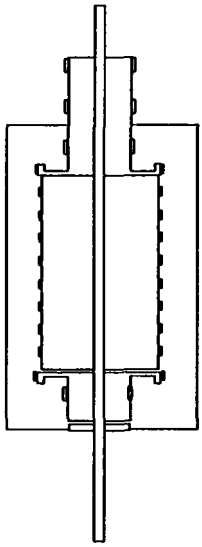


Fig. 13: Adiabatic minireactor with a catalyst volume of 10 ml developed by Gierman. After Sie and Blauwhoff (1991).

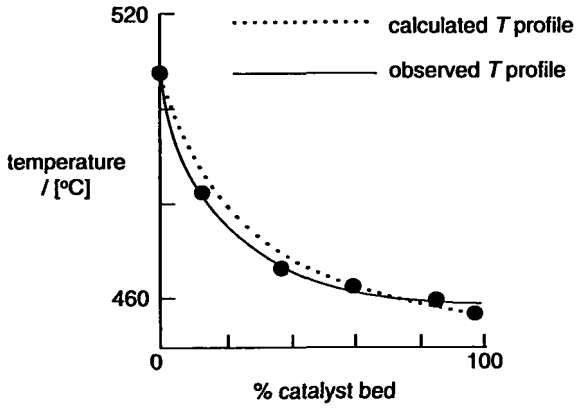


Fig. 14: Temperature profiles in an adiabatic minireactor used as first reactor in a catalytic reformer. After Sie and Blauwhoff (1991).

instrumentation for operatior than isothermal ones. This is particularly true for processes where the total adiabatic temperature rise or temperature drop necessitates the use of several reactors in series, with interstage cooling or heating. To simulate a commercial process such that similar conversions are

achieved, several reactors are also needed in the process development unit. Thus, even with relatively small adiabatic reactors, the process development unit is a rather complex miniplant instead of a simple one reactor system as may be the case with an isothermal reactor. An example of an adiabatic miniplant for catalytic reforming studies is depicted in Figure 15.

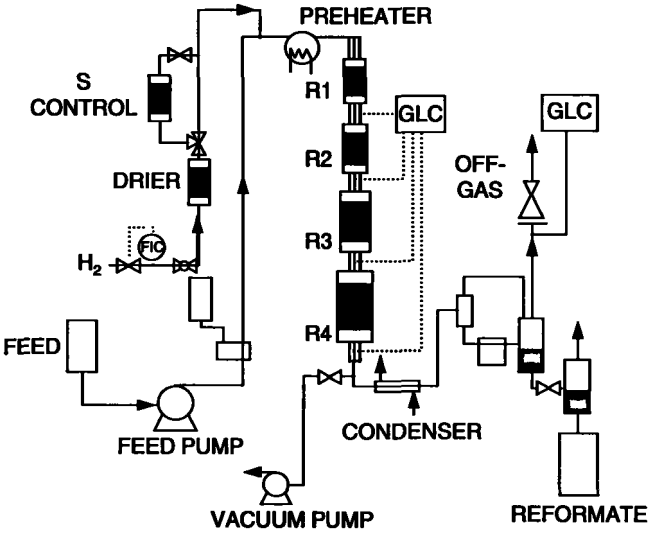


Fig. 15: Flow scheme of an adiabatic miniplant for catalytic reforming studies. After Sie and Blauwhoff (1991).

Minimum Size of Other Process Equipment

Especially in the case of integrated pilot plants the minimum feasible size may be dictated by other factors than those applicable to the reactor itself. In the case of a plant with recycles the need for representative response times of the system may dictate the size of reactor if certain dead volumes in the system are unavoidable. In addition, other pieces of equipment that cannot be sufficiently scaled down may dictate the minimum feasible size of the integrated pilot plant. Minimum sizes of some pieces of equipment are listed in Table 9.

From this table it can be deduced that distillation columns and their hold-up can very often be the limiting factor in size reduction of an

Table 9

Minimum size of tried and tested equipment (Maier and Kaibel, 1990)

Columns	
• Packed	30 mm diam.
• Structured packing	35 mm diam.
• Bubble cap	50 mm. diam.
Pumps	
• Displacement	0.000001 m^3/h
• Piston	0.00001 m^3/h
• Centrifugal	0.00001 m^3/h
Belt filter	50 mm width
Centrifuge	50 m^3/h delivery
Measuring equipment	
• Gas flow, thermal	0.00002 - 0.0006 m^3/h
• Gas flow, volumetric	0.0002 - 0.2 m^3/h
• Liquid flow, thermal	0.0002 - 0.06 m^3/h
• Liquid flow, volumetric	0.0001 - 0.001 m^3/h
• Liquid flow, gravimetric	0.002 - 0.05 m^3/h

integrated pilot plant. However, in some cases a true distillation may be replaced by another separation technique that achieves a similar separation on a smaller scale. An example of this approach is the replacement of a distillation column by a stripping fractionator in a laboratory hydrocracking unit, which enables the construction of a recycle hydrocracker with catalyst volumes in the reactor as small as 10 mL (Van Dijk *et al.*, 1991). Figures 16

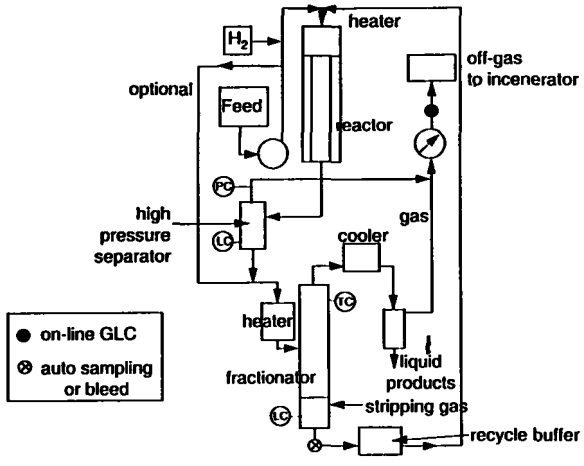


Fig. 16: Flow scheme of a small-scale recycle unit. After Van Dijk *et al.* (1991).

and 17 show a flow scheme and photograph of such a small recycle unit. Figure 18 compares the boiling point distribution of the laboratory-scale reactor effluent with that expected from an actual fractionator in the system, taking into account fractionator efficiency and product specifications.

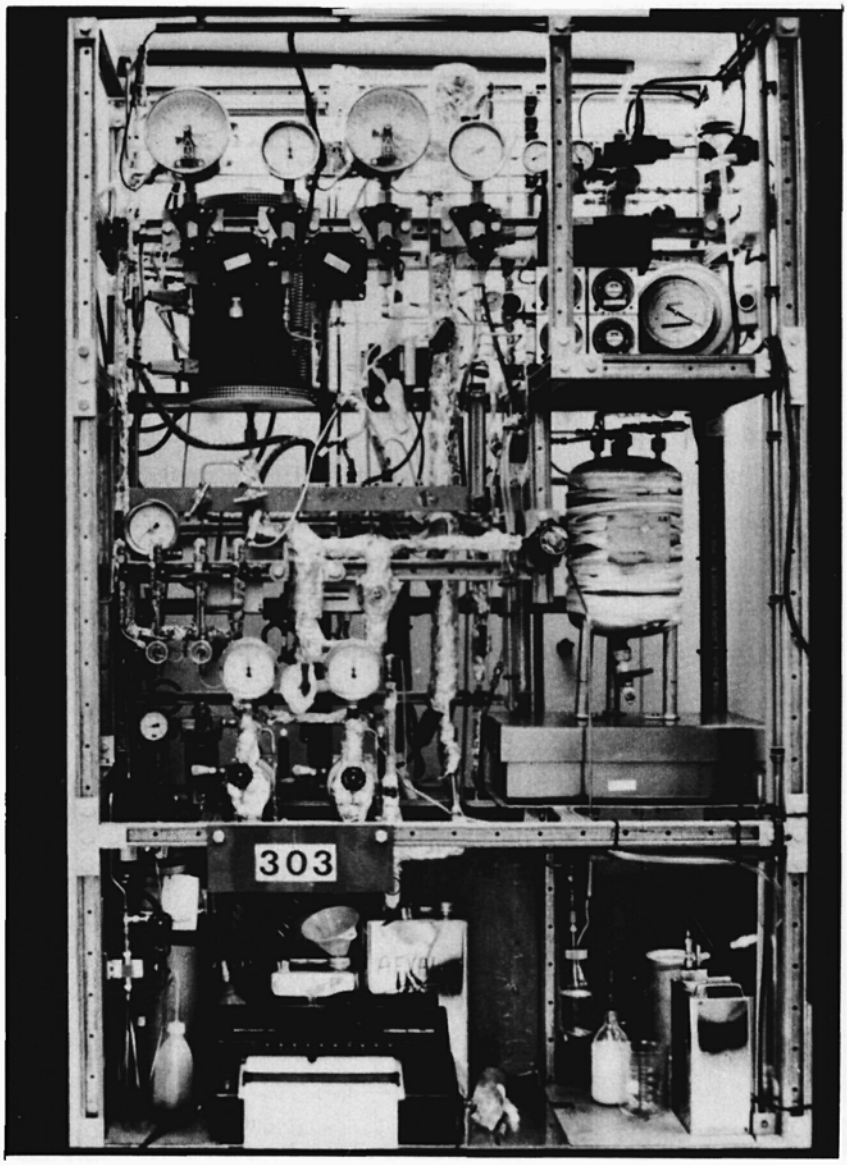


Fig. 17: Photograph of small-scale recycle unit. After Van Dijk *et al.* (1991).

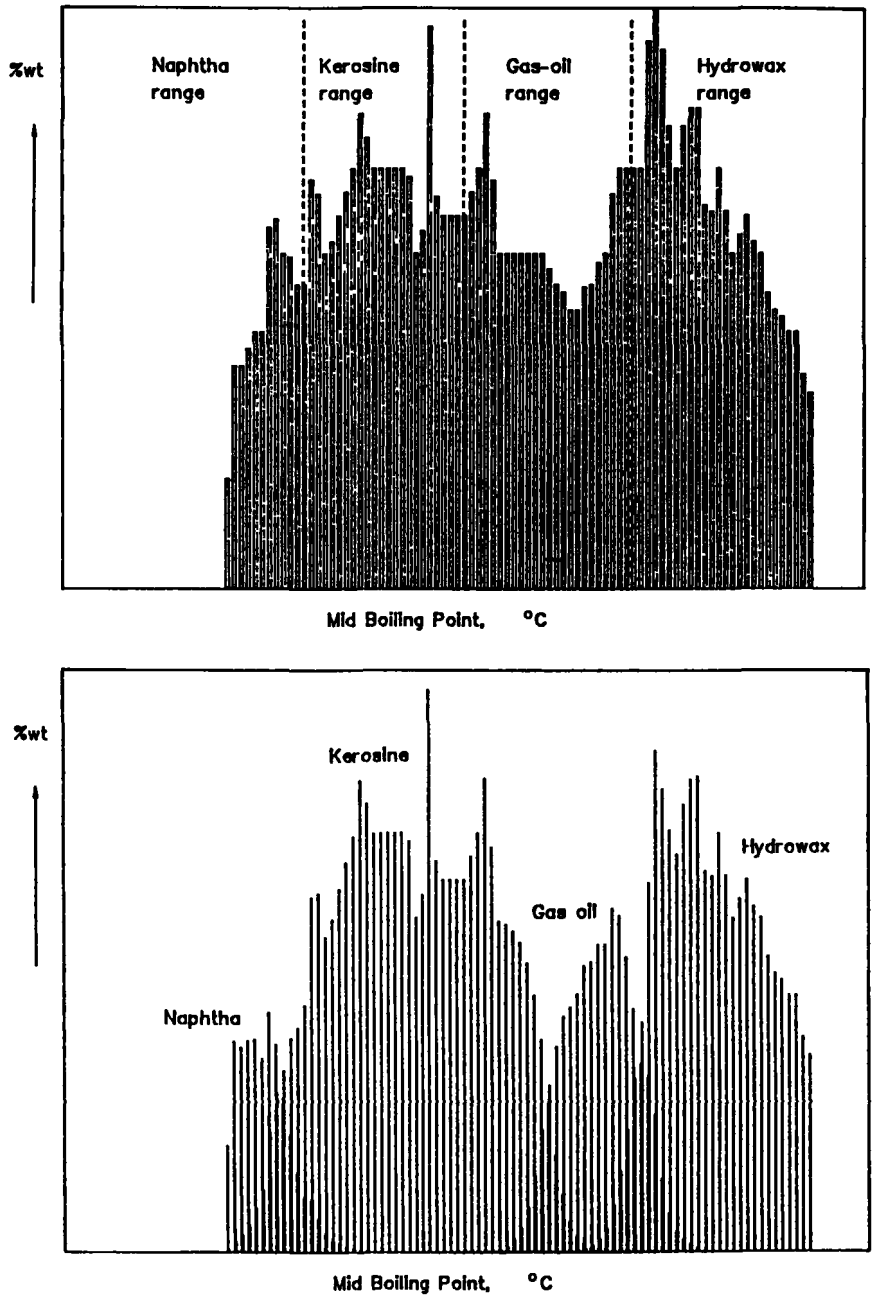


Fig. 18: Boiling point distributions of a reconstructed laboratory reactor effluent (above) and the corresponding simulated fractionator stream composition (below). After Van Dijk *et al.* (1991).

Installation Requirements and Costs of Process Development Units

Table 10 gives some typical data on tubing size, area requirement and cost of process development units. These are order of magnitude figures only and actual figures may differ for specific processes.

Table 10

Typical tubing size, area requirement on costs of process development units

	Tubing diameter/ [mm]	Floor area/ [m^2]	Cost/ Mfl
Microflow	2 - 5	0.5 - 1	0.1 - 0.3
Bench Scale	5 - 10	2 - 5	0.3 - 1
Pilot Plant	10 - 30	5 - 100	1 - 10
Demo Unit	> 25	> 1000	> 50

Depending upon the size of the units and the possibility of release of toxic gases, process development units may be housed in different ways, e.g.,

- In an air thermostat or ventilated cabinet
- In a fume hood
- In a containment cell within a building
- In a separate building
- In the open air

When dealing with flammable and/or toxic gases, suitable detectors are generally required to signal inadvertent releases of such gases to the surrounding atmosphere. As stated before, the consequences of such releases are far less serious when the scale of the experiments is very small.

Depending upon whether the units are expected to be in operation at a given location for a long time or whether they may have to be moved, one

may opt for a fixed-mounted or a skid-mounted unit. A unit that will have to be modified frequently to adapt it to the needs of specific processes is best constructed in a modular way.

Instrumentation and Control of Process Development Units

The degree of instrumentation of a process development unit depends very much on the information to be obtained from it, the expected duration of the investigations, and the complexity of the unit. In general a balance has to be obtained between accuracy and extensiveness of information generation, and cost of instrumentation. Several levels of instrumentation and control of process development units can be distinguished, viz.,

- *Level 1:* Individual measuring instruments giving local readings. Monitoring and control by operating personnel.
- *Level 2:* Individual measuring instruments linked to a monitoring computer via interfacing equipment. Data logging by computer, control by operating personnel. Set point adjustment manually on local individual instruments.
- *Level 3:* As above, but set points adjusted centrally by operating personnel on the basis of off-normal warnings and recorded data and trends given by the monitoring computer.
- *Level 4:* As above, but set points controlled by computer. Automatic actions safeguarding correct operation. Logical controller for safe start-up, shut down, and other changes in the process operation.

Because of the trends towards cheaper electronic measuring and control instruments and cheaper computing power, there has been an increase in the degree of instrumentation and control in time. This has led not only to more accurate experimental results but also to reduced manpower needs, as will be discussed below.

Evolution in Process Development

The drive towards small-scale experiments and the increased use of

computers to enhance the cost effectiveness of process R & D have caused changes in the way in which process development studies are carried out during the past decades. The increased knowledge of the role of scale factors on the performance of reactors has in many cases made it possible to reduce considerably the size of the equipment for a given duty. An example is the scale reduction of laboratory reactors for the study of trickle-flow processes (Sie, 1991).

Testing of practical catalysts with realistic feeds which 30 to 40 years ago had to be carried out in large pilot plants can now be done in bench-scale or even micro-scale reactors with comparable results. A typical pilot plant from that previous period is shown in Figure 19, while Figures 20 and

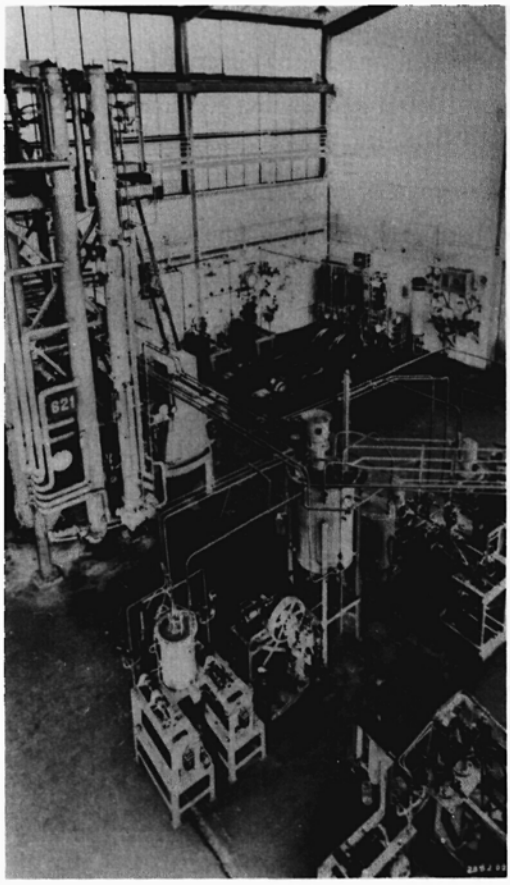


Fig. 19: **Pilot plant in the fifties for testing of hydroprocessing catalysts and feeds.**

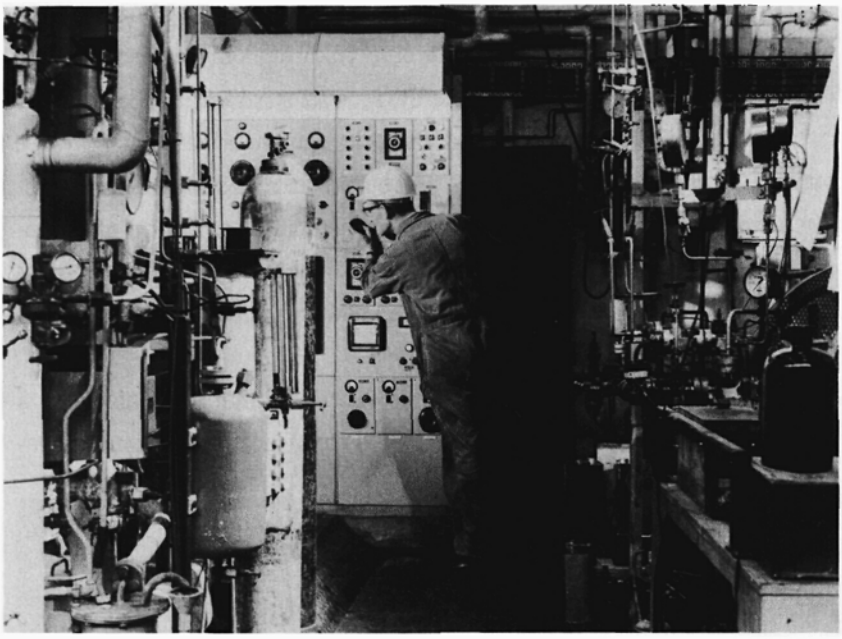


Fig. 20: Instrument panel of pilot plant of Figure 19.

21 show the measuring and control system of this pilot plant and the facilities for storage of liquid feed and products.

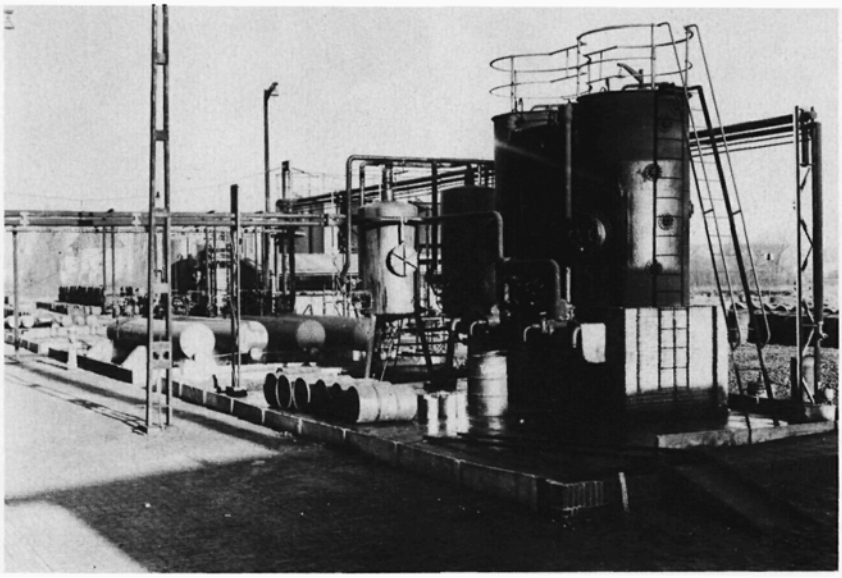


Fig. 21: Feed and product storage for pilot plant of Figure 19.

The pilot plant shown in Figure 19 is a large (about 0.02 m^3 catalyst volume), but otherwise rather simple one, featuring only a feed and gas dosing system, a single fixed-bed reactor, product separator and gas recycle system. Most of the data generated in such equipment can nowadays be obtained in much smaller equipment, i.e., bench-scale and microflow reactor units (Sie, 1995). A typical bench-scale unit suitable for testing of hydroprocessing feeds and catalysts, i.e., the same duty as the above older pilot plant, is shown in Figure 22. Such a unit is nowadays operated with computer control, see Figure 23.

The above examples show that in the course of time there has been a reduction in scale accompanied by increased sophistication in instrumenta-

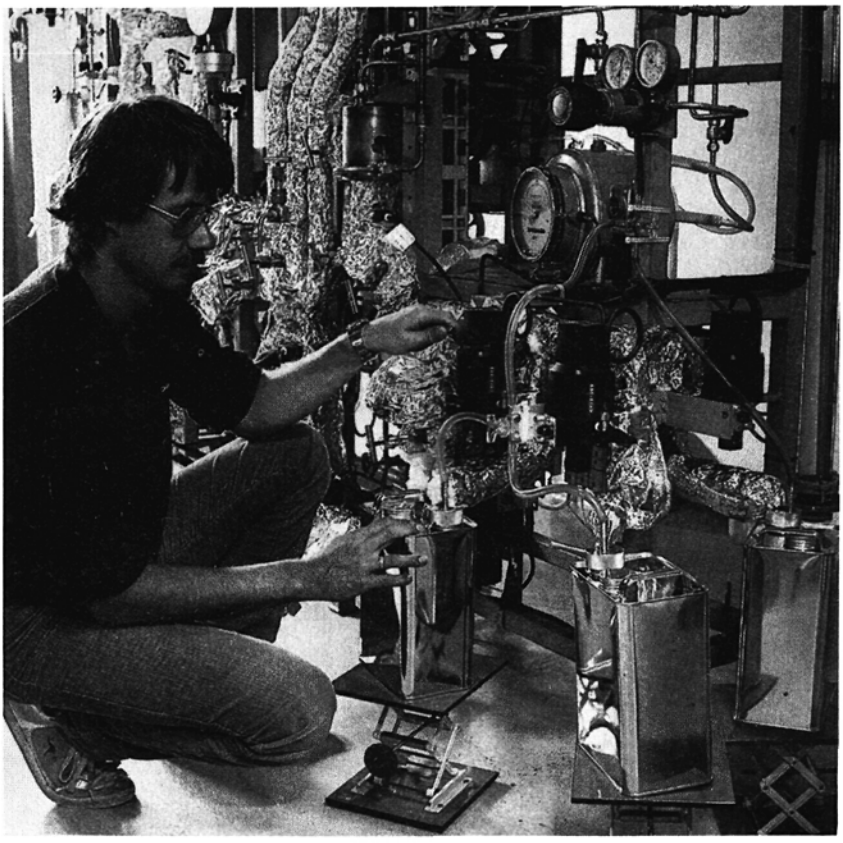


Fig. 22: Typical bench-scale fixed-bed reactor unit in the late eighties for study of trickle-flow processes.

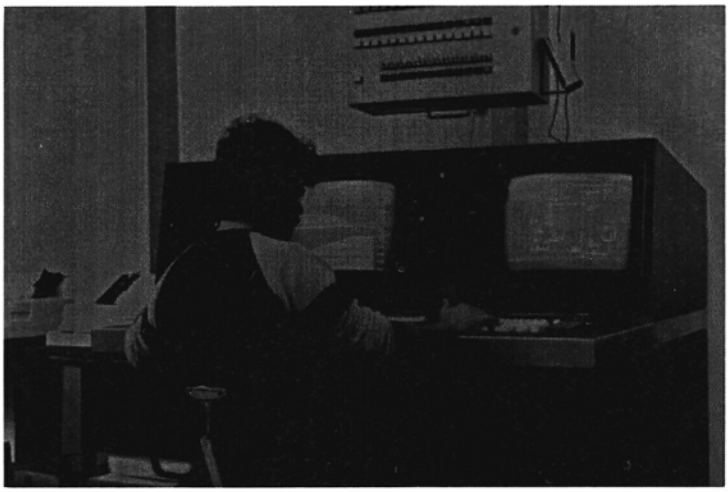


Fig. 23: Operator panel of bench-scale unit.

tion. As a consequence, current small-scale process development units are not only more flexible, but may in most cases yield at least as good results as the older large pilot plants. The trend towards smaller scale equipment is illustrated by the breakdown of reactor units in an oil process R & D laboratory shown in Figure 24.

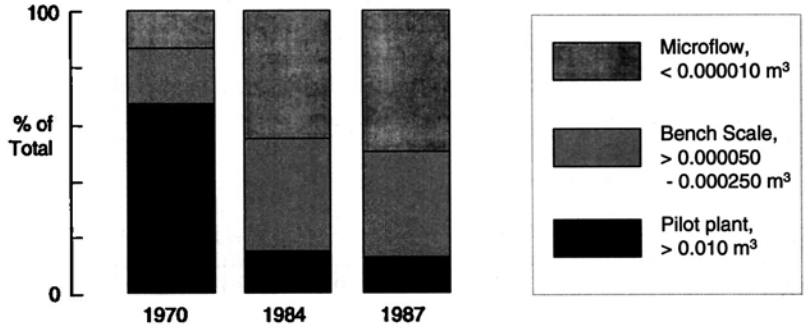


Fig. 24: Breakdown of reactor units in an oil process R & D laboratory, showing evolution in size reduction.

The size reduction accompanied by enhanced intrinsic safety and increased instrumentation has enabled automatic, unattended round-the-clock operation of units, even those operating at elevated temperature and

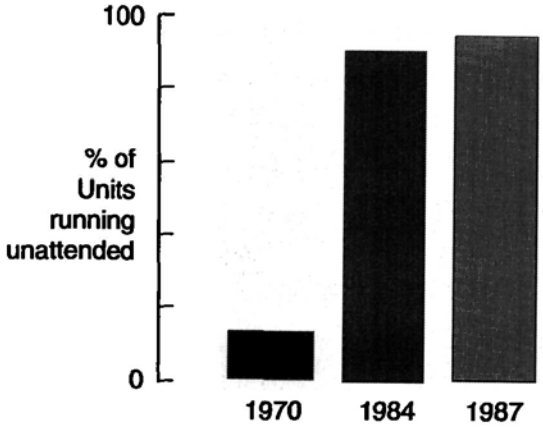


Fig. 25: Evolution in automation of reactor units in an oil process R & D laboratory.

pressure with flammable and toxic gases. Figure 25 shows the increase of the proportion of units running without attention over the past 25 years in the same laboratory. Whereas operating personnel in the past were mainly needed to ensure stable operation of the unit, this task is now in most cases taken over by computers. With the automated equipment, the task of the operating personnel has now shifted towards preparation of the experiment, setting of control values, corrections in case of malfunctioning, and interpreting and reporting of results. Table 11 compares the manpower needs for the operation of process development units of varying size and illustrates the gain in research effectiveness by size reduction.

Table 11

Operator requirement of reactor units in hydrotreating process development

	Manhour per reactor hour
Pilot plant, 24 h shift	1.2
Bench-scale, 24 hr shift	0.5
Bench-scale, unattended	0.15
Micro-flow, unattended	0.08

The increased degree of instrumentation of process development units has not been restricted to more extensive monitoring and control of flows, temperatures, pressures to ensure precise and stable operating conditions, but in addition there has been a growing use of on-line instruments to monitor the composition or properties of process streams. The growth in the application of on-line quality measuring instruments (QMI) is demonstrated by the figures in Table 12.

Table 12

Growth in the number of on-line quality measuring instruments (QMI) in an oil process research and development laboratory

Year	GLC	Other QMI	Total QMI
1970	6	0	6
1980	26	38	64
1984	34	100	134
1986	42	76	118
1988	49	66	115

The application of on-line QMI not only allows closer monitoring of product properties than with less frequent off-line analyses having a delay because of turnaround times, but also saves manpower in the analytical control laboratory. This manpower saving is in addition to the savings in operating personnel which has been realized by automation of the process development units. Figure 26 shows the increase in on-line gas chromato-

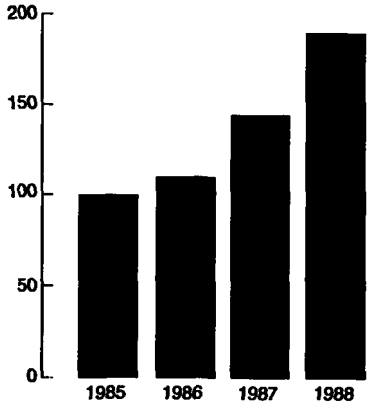


Fig. 26: Relative increase in on-line gas GLC analyses in an oil process R & D laboratory.

graphic analyses in the above oil process R & D laboratory during the last decade.

CONCLUDING REMARKS

Preparing for the commercial application of a new process or the improvement of an existing commercial process by introduction of new technological solutions requires a methodological effort which is generally termed process development. This effort should be aimed at providing the technical information required to design and implement the new technological concepts with an acceptably low risk and high chance of success. A crucial step in an early stage of process development is a timely identification of process imperatives and potential problem areas which to a large extent determine the course of process development.

The development of a novel commercial process from a concept that has been studied on a small scale in a laboratory implies a scaling-up exercise. This scaling-up is generally done in steps, and the maximum scale-up factor for each step will depend on the degree of understanding of underlying factors and upon experience available.

The generation of the necessary information in process development studies should be done in the most effective way, i.e., in the cheapest and speediest manner without detracting from the validity and accuracy of the data. Therefore, the main purpose of any process development unit should be well defined in advance and the choice and design of the unit should be made accordingly, bearing in mind cost minimization.

The advent of modern techniques in catalysis research (*in situ* characterization methods, high resolution imaging techniques, etc.) and in engineering research (laser diagnostics, sensitive transducers, etc.) and in data acquisition and handling (more powerful computers, parallel computers, etc.) have contributed to increased effectiveness of research.

Whereas the development of a novel process can be considered to be a scaling-up exercise, scaling-down of the process development tools is also an

important goal. Scale reduction of process development units and advanced instrumentation have considerably increased the cost effectiveness of process development, as illustrated by the examples given. Although reactor units of very small scale can now be used to gather relevant data on catalytic processes in many cases, there are still situation where a large pilot plant is indispensable. This is particularly true in the development of major novel processes representing appreciable step-outs from the fields of existing technology. Unpredictable interaction of process elements in a complex process line-up with many recycles, unknown behaviour of solid particles in the process, difficult to quantify fouling phenomena and need for sufficient quantities of a new product for field trials or for the development of new markets may all be reasons for carrying out process development on a relatively large scale. Even in these cases there is no merit in choosing a scale larger than strictly necessary. A good guiding principle should therefore be:

"SMALL IF YOU CAN, BIG IF YOU MUST"

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