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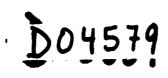
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Expert Group Meeting on Transfer of Know-how in Production and Use of Catalysts

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# PACTORS APPECTING THE DECISION TO MANUFACTURE CATALISTE

#### W

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We regret that some of the pages in the microfiche copy of this report may not be up to the proper legibility standards, even though the best possible copy was used for preparing the master fiche.

In order to provide background for the remarks which follow, I would first of all like to identify the organization which I represent and explain its functions and associations.

Catalyst Development Corporation is a manufacturer of catalysts. The company is associated with Scientific Design Co. which is designer and builder of chemical plants throughout the world, and Halcon International Inc., a libensor of technology and owners of large chemical manufacturing operations. Catalyst Development Corporation's primary activities are concerned with petrochemical catalysts which are part of the total technology package developed and licensed by Halcon for plants designed, engineered and constructed by Scientific Design Company.

To provide a framework for the discussion which follows, it will be useful to have a classification of various catalysts of potential interest as shown in the tabulation you see on the screen. Neither large volume petroleum catalysts such as the fluid catalytic cracking type or platinum-alumina used for reforming, nor catalysts used in large scale petrochemical operations, such as nickel catalyst for hydrogenation, or the several catalysts employed in ammonia plants are within the realm of our experience. Rather, I willdiscuss more specifically the area of specialty petrochemical catalysts such as those we manufacture for processes making intermediate chemicals such as ethylene oxide and maleic anhydride. These intermediates are used as "building blocks", the majority of which eventually become polymer products or additives.

## SINGNIFICANT CATALYST MARKETS

#### Fetroleum refining

Alkylation Cracking-fluid Cracking-moving beds Desulphurization Hydrocracking Hydrotreating Isomerization Folymerization Reforming (Pt)

#### Petrochemical

Major markets (1000 tons or more annual catalyst production)

Alkylation AlCl<sub>3</sub> Ammonia Reforming Shift conversion Kethanation Synthesis -2-

## Petrochemical (cont'd)

## Major markets (cont'd)

Butadiene Desulphurization Ethylene dichloride Hydrogenation Polymerization Metal organics Peroxides Styrene Sulphuric acid

## Other markets

Ethylene oxide Formaldehyde Ag or mixed oxides Maleic anhydride Methanol Nitric acid Pharmaceuticals Phthalic anhydride Folymerization Titanium trichloride

It may be an over-simplification but it is vital to recognise that catalyst performance is always an important factor and in many cases the critical one in determining the economics and successful practice of a chemical process. There are a tremendous variety of commercially available catalysts which may find usage in relatively broad chemical process areas such as nickel catalysts in hydrogenation reactions: while others have been designed for animay only be used successfully for *e* very specific reaction. Just as there are a myriad fo products bearing the name catalyst, there is a wide variety in the methods used to make them. Testing methods and requirements also vary widely and may sometimes be limited to relatively simple chemical and physical tests as opposed to frequently employed, very sophisticated testing in pilot plant prototype units. In some cases there is a real need for on-site preparation of a catalyst such as a situation where the catalyst is consumed or its physical form is altered beyond the point of continued utility in the reaction itself.

With the foregoing general remarks as a very brief background, let us explore in more detail the specific technical and economic factors which must be carefully considered when examining the possibility of catalyst manufacture. These factors are raw materials; material, equipment and personnel aspects of production- product testing; technical service.

#### Raw materials

Availability of key raw materials and and adequacy of supply, preferably from multiple sources, must be established in terms of suitable chemical and physical properties. This might be interpreted as a statement of the obvious but let me digress for a moment and describe one particular, familiar instance that points up the difficulties in what seemingly is a relatively simple situation. In our manufacture of catalyst for maleic anhydride, one of the principal constituents is a vanadium bearing compound. During the laboratory development of this catalyst, a certain commercial grade of this compound was used in the variety of catalyst formulations tested - As the process evolved to commercial status, and the need for commercial quantities of catalyst came closer to reality, efforts were undertaken to qualify less expensive and more readily available commercial grades of this vanadium compound but all of the efforts proved unsuccessful, primarily due, it was discovered, to different combinations of impurity levels. It was necessary, therefore, to manufacture the initial commercial charges of catalyst using the laboratory qualified product and the fact of the matter is that the catalyst is still prepared today using this same quality vanadium compound in significant quantities. During the time that this cutalyst has been manufactured, numerous sources of this compound have been evaluated and only one, the original, allows the preparation of catalyst of superior quality. In many cases, one cannot over emphasize the possible modifications in catalyst activity or selectivity caused by seemingly trace amounts of impurities or a different set of the same impurities. Naturally occurring or semi-refined raw materials such as clays or zeolites, whose chemical analysis may vary over a fairly broad spectrum, can be the source of major problems in the performance of finished catalysts - It is vital that raw material. manufacturing procedures and quality control are not only known to the catalyst manufacturer but also monitored by him on a regular basis. The establishment of specifications for catalyst raw materials is many times in itself a lengthy, tedious development programme wherein the effect of the impurities on catalyst performance must be determined independently. Where precious metals are used as catalysts, frequent accurate assays are essential, metal recovery systems must be devised, contamination prevention procedures constantly monitored and security programmes implemented and continuously maintained. As one example, in the area of raw material testing let us look at the critical properties of a catalyst carrier. The slide you now see on the soreen is a tabul tion of such physical and chemical properties that are measured to provide a definition of a catalyst carrier as a suitable substrate or co-catalyst. The list is rather a lengthy one; some of the tests required are rather elaborate and the understanding of the inter-relationship of all these factors with respect to catalyst performance is an essential element in the control of catalyst quality.

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#### CATALYST CARRIER

#### Physical properties

Size and shape Attrition loss Crushing strength Total porosity Pore volume Pore size distribution Specific gravity Packing density Surface area Crystal form Water absorption Absorption isotherms

## Chemical properties

Spectrographic analysis Reactivity Leachable impurities Resistance to acid or alkali

#### Production

Process steps or conditions for the manufacture of catalyst either on a batch or continuous basis cover a wide range of possibilities. The tabulation on the screen is intended only as a partial listing of the varied operations one might encounter in commeroial catalyst manufacture. The size of equipment in the plant depends, of course, on the annual market for the oatalyst. This is determined by the amount of catalyst used in the chemical process, and the frequency with which catalyst is replaced In the manufacture of intermediate chemicals where catalysts life may be from one to ten years, it is not unusual to find that a minimum sized catalyst manufacturing operation may need to operate over a period of only one, two or three weeks in order to manufacture a complete plant charge of catalyst. Such a circumstance of course raises sericus questions regarding the advisability of an investment in the catalyst manufacturing facility and the maintenance of an adequate level of training among operating personnel. During the long period the plant is not operated, people can and will lose the art. In our experience we must admit that there remains a measure of art and a specialized know-how needed in the continuing successful production of catalysts. Inprocess controls are a vital factor not only of such variables as weight, volume, temperature, viscosity, or dryness but also in the frequent evaluation of finished catalyst quality to ensure that current production is meeting pre-set standards during every step of the manufacturing process. In cases where a catalyst plant is charged

with the manufacture of more than one type of catalyst the dangers of contamination of one catalyst by the constituents of another must be carefully considered during design stage of the catalyst plant. Fumes from surrounding chemical plant operations must also be considered in this category for example the presence in the atmosphere of even small amounts of halogen compounds near an ethylene oxide catalyst manufacturing plant could be intolerable since these could be expected to poison freshly manufactured catalyst to the point of inutility. Some catalysts used commercially are relatively weak from a physical standpoint and must be handled during the manufacturing operation with a great deal of care to prevent attrition, orushing or loss of active ingredients. In the catalyst manufacturing business it is probable that a manufacturer or potential manufacturer will find that the specialized type of equipment required to make one particular catalyst is not at all suitable for another type because of the process steps involved or potential contamination. In many cases it is necessary to acquire, build and operate separate and distinct facilities.

# MANUFACTURE OF HETEROGENEOUS CATALYSTS - PROCESS STEPS AND EQUIPMENT

#### Preparation

Solution preparation heating/cooling viscosity control Formula control Solids - solids mixing Solids - liquids mixing Evaporation/crystallisation Drying Grinding & sizing Ball millin; Aging Pollution control

#### Forming

Extrusion Pelletising Spherodising Coating Impregnation Spray drying (fluid catalyst) Pollution control

#### Final steps

Activation heating/controlled atmosphere oxidation/reduction (not in situ) pollution control Elending/screening Protective packaging

#### Testing

To a potential catalyst manufacturer the area of catalyst testing and evaluation is as great a concern as the care necessary in the raw material control and the production process steps for that catalyst. It is possible to conduct a variety of chemical tests and in some cases necrossary to conduct numerous chemical tests in those areas where manufacturing conditions are not easily controlled. The definition of the precise amount of each of the key catalyst components and the levels of known critical impurities is frequently imperative. Similarily, depending upon the particular catalyst, physical tests may be warranted such as attrition loss, surface area, or volume and size, or sime of the others which were pointed out earlier as being critical for incoming quality control of catalyst carriers. In addition to all the chemical and physical tests that may be indicated as necessary controls, what interests both the catalyst manufacturer and the chemical plant operator is the question of catalyst performance. Does the charge of catalyst conform to the performance standards which will allow the chemical plant to operate as it was designed ? The key factor is that performance testing carried out by the catalyst manufacturer must be closely related to the chemical plant reaction system and a close correlation developed between the operation of the plant reactor and the laboratory or pilot plant test reactor. Perhaps a relatively simple example, which is pertinent to many chemical reactions, is the use of a tubular reactor. Such a test reactor can be designed to simulate the plant reactor by using one or only a small number of tubes of the same dimension as the tubes used in the plant reactor. The test reactor shouldhave the same catalyst bed height so that heat transfer, flow and recycle conditions are simulated as closely as possible. The catalyst manufacturer must determine the frequency with which he proposes to test the manufactured catalyst and I might interject here that waiting until a complete plant charge has been completed is not recommended. It is a very expensive crisis when one finds 10,000 or even perhaps 50,000 kilograms of catalyst have been made and none of several representative samples tested have been able to pass the performance test.

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Analytical facilities associated with the catalyst test equipment must be designed as accurately and dependably as those which are used to monitor chemical plant reactor operations on a continuing basis. Even after the catalyst manufacturing plant is built and operating and the quality of the catalyst has been confirmed by frequent testing of representative samples from reasonably sized production lots, there remains one troublesome question which is frequently left unanswered. That question is: What is the life expenctancy of a given charge of catalyst if operated, under normal plant design conditions? The shutting down of a plant reactor and the consequent loss of production can very seriously affect the economic success of a process if it occurs earlier or more frequently than expected. Insuring that such shut-downs do not occur because of catalyst quality is a factor that the catalyst manufacturer must consider and be in a position to evaluate above and beyond the initial catalyst performance characteristics used as a quality control procedure. It is apparent that the expertise of the process designer, chemical plant operator and catalyst manufacturer must be carefully co-ordinated to avoid problems and optimize plant performance.

In the manufacture of various catalysts, it is not unusual to find that the investment in testing facilities and the training of people to operate those facilities is more expensive and time consuming than the catalyst manufacturing operation itself. If no other point is made in the course of these remarks today, I do want to stress the very important and critical step that catalyst performance testing represents.

#### Technical considerations

In the discussion of catalyst manufacturing and catalyst testing, we see that a corollary is the need for high quality technical manpower to support the catalyst manufacturing operation. Various experts are needed beginning with the design phase of the catalyst manufacturing plant and including those responsible for the day-to-day operation of it, maintenance engineers, a diversity of analytical talent and detailed knowledge and understanding of the commercial process or processes in which the catalyst or catalysts may be used. Never is the availablity of this talent more necessary or vital to success than those times when problems occur. I refer not so much to those problems which might be associated with the breakdown of catalyst manufacturing plant equipment but to those situations where the catalyst has been manufactured in accordance with well delineated procedures using all the specified precautionaFy controls and then the performance tests show repeatedly that the catalyst is of inadequate quality to afford economic operation using the plant charge. At a point in time such asthis the catalyst manufacturer must have a group of individuals, each with unique skills, who can be called upon to investigate and solve the catalyst manufacturing problem in a very short period of time. I think the other catalyst manufacturers prosent would agree with me when I say that there is a great deal of art involved in the manufacture of many catalysts and it is difficult if not impossible to define specifically all of the variables critical to the preparation of good quality catalyst. The economic considerations at stake are great. The loss of two or three yield points in the initial performance of a catalyst charge or a greater than expected rate of decline in activity can be extrapolated to millions of dollars in product losses and a completely unacceptable, uneconomical plant situation.

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During the remarks made thus fer, the basic assumption has been made that wellpracticed technology would be available to any organization desiring to establish a new catalyst manufacturing facility. When this technology is translated into a new plant facility it is essential that the fewest possible number of changes in equipment size and type be made. One of the major problems in the connercial manufacture of a new catalyst is the necessity for scaling up to economical size the successful catalyst preparation technique that has been developed in the laboratory. I think you will find in the case of many catalysts, that production operations may appear very simple or even backward in the light of known readily available equipment that might have been chosen. Often the reason for this is the fact that such relatively primitive techniques have been shown to produce catalyst of superior quality, whereas seemingly more complex, automated techniques could produce inferior quality catalyst.

It should be recognized that two processes for making the same chemical commodity may very likely employ two entirely different catalysts each one of which is entirely consistent with the unique process conditions for which it was deisgned. They will not, however, be found to be inter-changeable and a catalyst manufacturing plant for the preparation of one may very well be completely unsulted for the preparation of the other. This is another of the market factors that must be considered in deciding whether or not to manufacture catalyst. A similar point might be made with regard to catalysts which may have the same basic constituent but are used for entirely different processes. A good example of this is vanadium pentoxide catalyst used for sulphuric acid or the vanadium pentoxide based catalyst used for phthalic anhydride or maleic anhydride. The catalyst manufacturing processes in all three of these cases are significantly different and I doubt that very little equipment would be interchangeable. A universal catalyst plant is a desirable objective, but I fear, an unreal one from a practical standpoint.

The decision to manufacture catalyst is a complex one us the foregoing remarks have indicated. It is a decision which requires the careful consideration of: the availability of appropriate technical know-how and manpower the obtaining and evaluation of raw materials; the design and building of a facility which may be under-utilized causing technical and personnel problems due to infrequent use: extensive and in some cases elaborate testing facilities and a thorough understanding of the use of the catalyst in a chemical plant.

The decisions which you may be contemplating relate to specific catalysts and specific plant situations. The problems involved and the approach to their solution will undoubtedly be different in each case. However, the factors which will affect the solutions of these problems must universally take into account all of those I

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have been discussing with you today. One must always be prepared to accept those risks that involve answers to operating plant management explaining unsatisfactory catelyst performance.

I trust I have been able to give you some ideas in the brief time available which will at least be of help in avoiding difficult problems should the manufacture of catalyst be a prime consideration in the planning that is being done for chemical industry expansion.

