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4 July 1972

United Nations Industrial Development Organization

Original : ENGLISH

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

Bucharest, Romania, 26 - 30 June 1972

CATALYST TESTING^{1/}

presented by

R. W. Bedford

Imperial Chemical Industries Limited
Billingham, Teesside, United Kingdom

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ID/WG.123/20/S-1972
19 June 1972

United Nations Industrial Development Organization

Original : ENGLISH

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

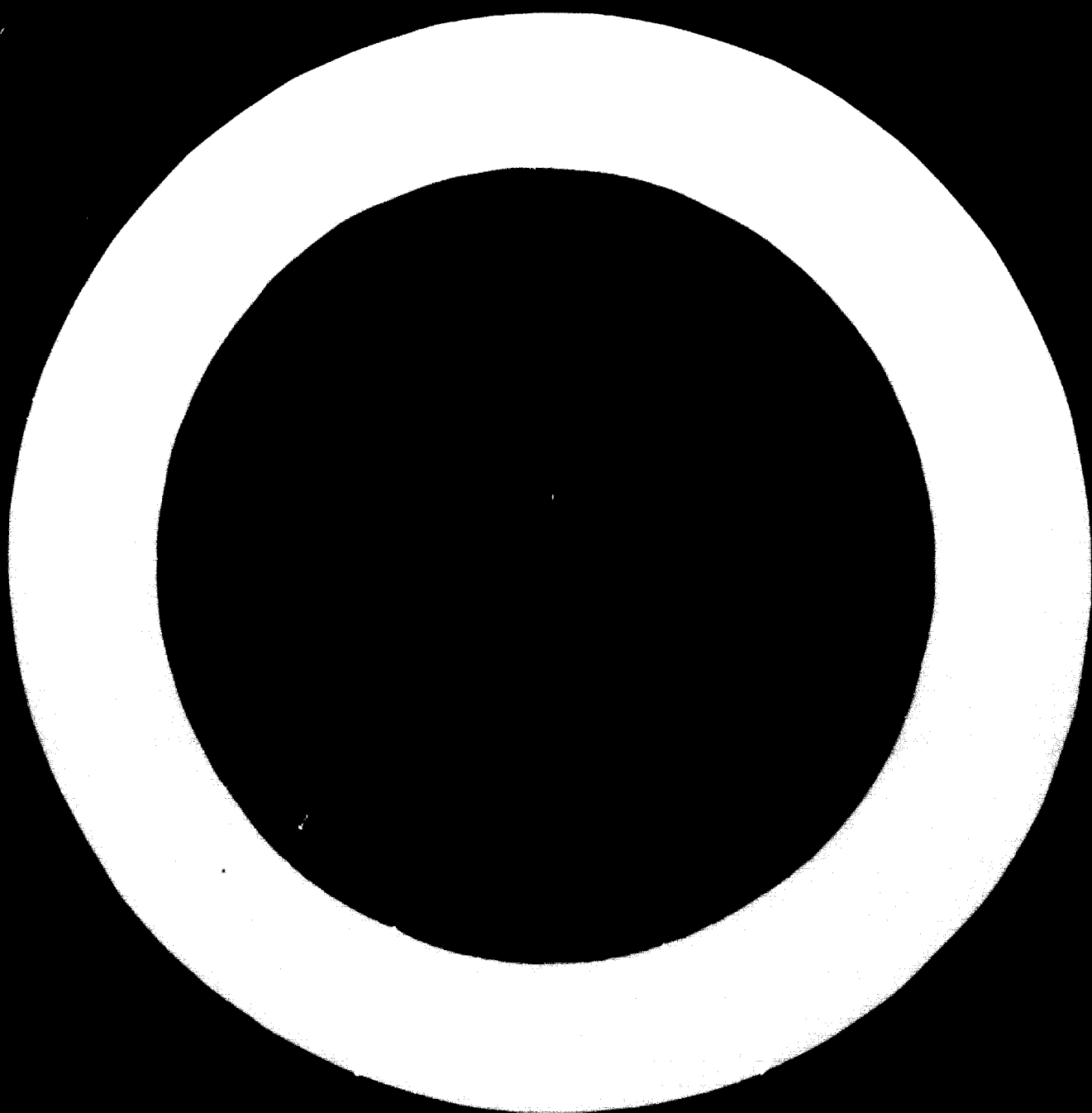
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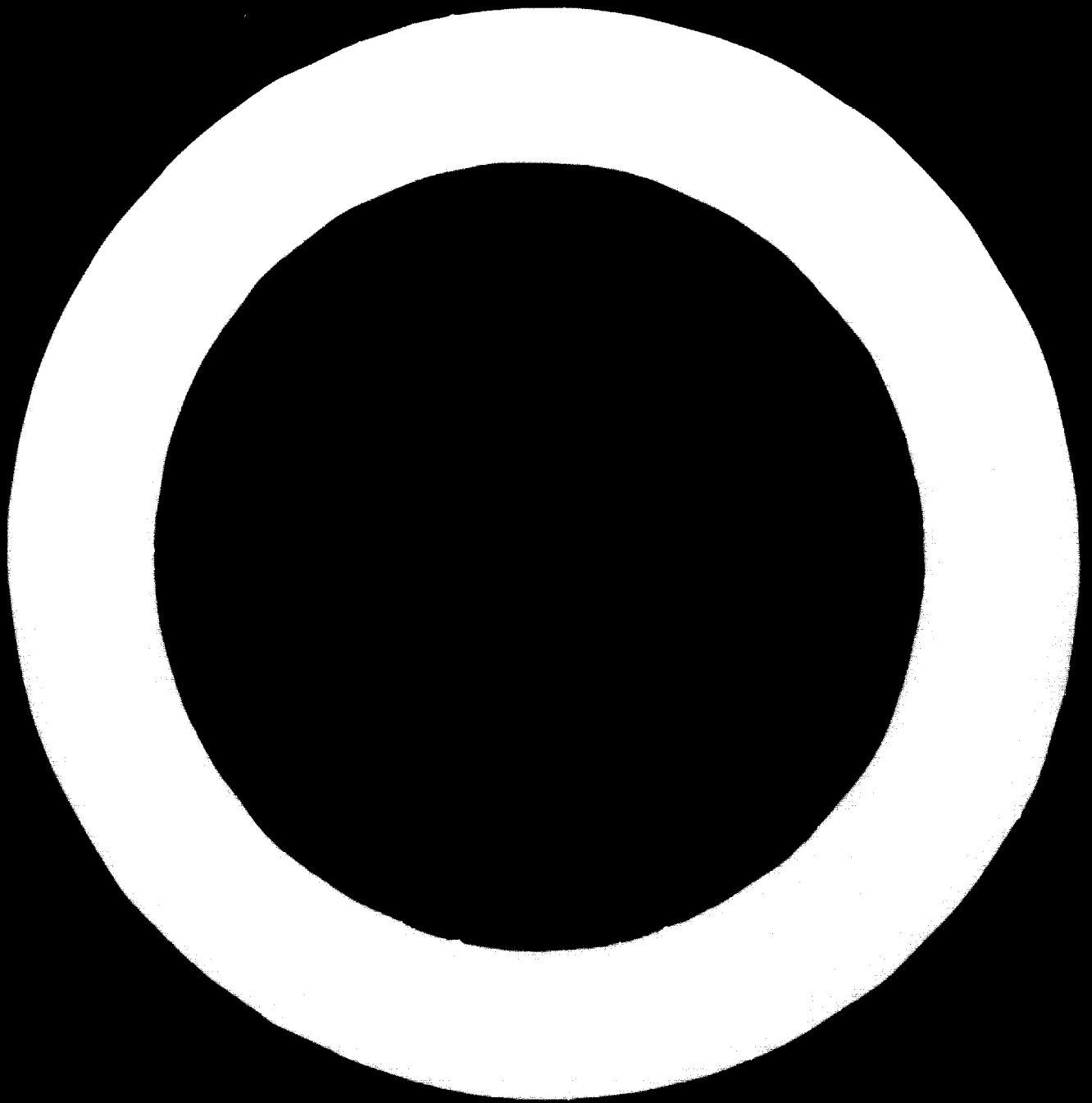
The evaluation of a catalyst's activity and other properties is essential in the development of new catalysts and in the control of catalyst manufacture. It is also regarded by some catalyst users as a useful aid to catalyst selection. It is, however, an extremely complex problem and there are many pitfalls into which unsuspecting catalyst tester may fall. In designing test methods, great care must be taken to ensure that the test will actually measure the desired property and that the equipment used is able reliably to fulfil all the requirements of the test.

This paper examines the whole field of catalyst testing. Three levels of catalyst testing are defined. These are:

1. Simple comparative testing
2. Major catalyst evaluation, including simulated plant operation
3. Fundamental testing in order to completely characterize the catalyst and the reactions it catalyses.

The various types of tests used at each level of catalyst testing are described and their usefulness to the catalyst researcher, catalyst manufacturer and catalyst user assessed.

The testing of the various types of catalysts used in ammonia manufacture is used as illustration with emphasis on both those testing methods which are common to all catalysts and those which are particular to an individual catalyst. The measurement of a catalyst's catalytic activity, chemical constituents, and physical properties such as surface area, pore structure, shape and strength and catalytic activity is discussed together with an evaluation to give good, long term performance in a full scale plant.



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Preface

It may seem to be stating the obvious to say that a catalyst tester must be sure he is measuring the property he desires and that the property he is measuring is relevant to the performance of the catalyst in a full scale plant. Testing catalyst is, however, a complex science requiring skill and experience in the catalyst tester and even then it is all too easy for an experienced catalyst tester to make mistakes if he does not maintain a rigid discipline of a clear objective approach to each and every test.

ICI Agricultural Division has been a major centre for research and development of commercial catalysts for over 40 years. During that time, testing techniques have increased in complexity and sophistication in parallel with the development of the chemical plants using the catalyst being developed. Nowadays it is possible to virtually completely characterise new catalyst and predict with reasonable confidence the way it will perform in a commercial plant. It is generally true, however, that the cost and sophistication of the experimental techniques is fairly closely paralleled by their usefulness in predicting plant performance.

This paper will discuss the testing of solid catalysts used in promoting gaseous reactions such as those used in ammonia plants and similar plants based on hydrogen production.

I. INTRODUCTION

1. Developing a new catalyst requires a lengthy, complex testing programme. There are four groups of characteristics which must be evaluated.

- 1 Catalytic activity
- 2 Physical characteristics
- 3 Mechanical strength
- 4 Chemical composition

2. Of these groups, the fourth one that of analysing a catalyst chemical composition is not particular to catalyst testing and will not be discussed in this paper. In each of the other three, a wide variety of tests may be carried out of varying complexity. These tests may be sorted into three groups which represent three different levels of testing.

- 1 Simple comparative testing
- 2 Major catalyst evaluation, including simulated plant operation
- 3 Fundamental testing in order to completely characterise the catalyst and the reaction it catalyses

II. CATALYST ACTIVITY

3. The measurement of catalyst activity is, of course, the whole basis of a catalyst research programme. The steps involved in developing a successful catalyst are set out diagrammatically in Figure 1. The procedure is designed to permit the maximum number of variables to be examined in a way which results in all potentially suitable catalysts receiving much more detailed investigation. By this technique, full scale testing is reserved for those catalysts which have passed the other tests.

4. A rapid initial screening is carried out at atmospheric pressure, so that those formulations showing promise can then be tested more rigorously under semi-technical and sidestream conditions to obtain basic kinetic and life data. More detailed kinetic and poisoning information can then be obtained under differential operating conditions. Formulations which pass sidestream and

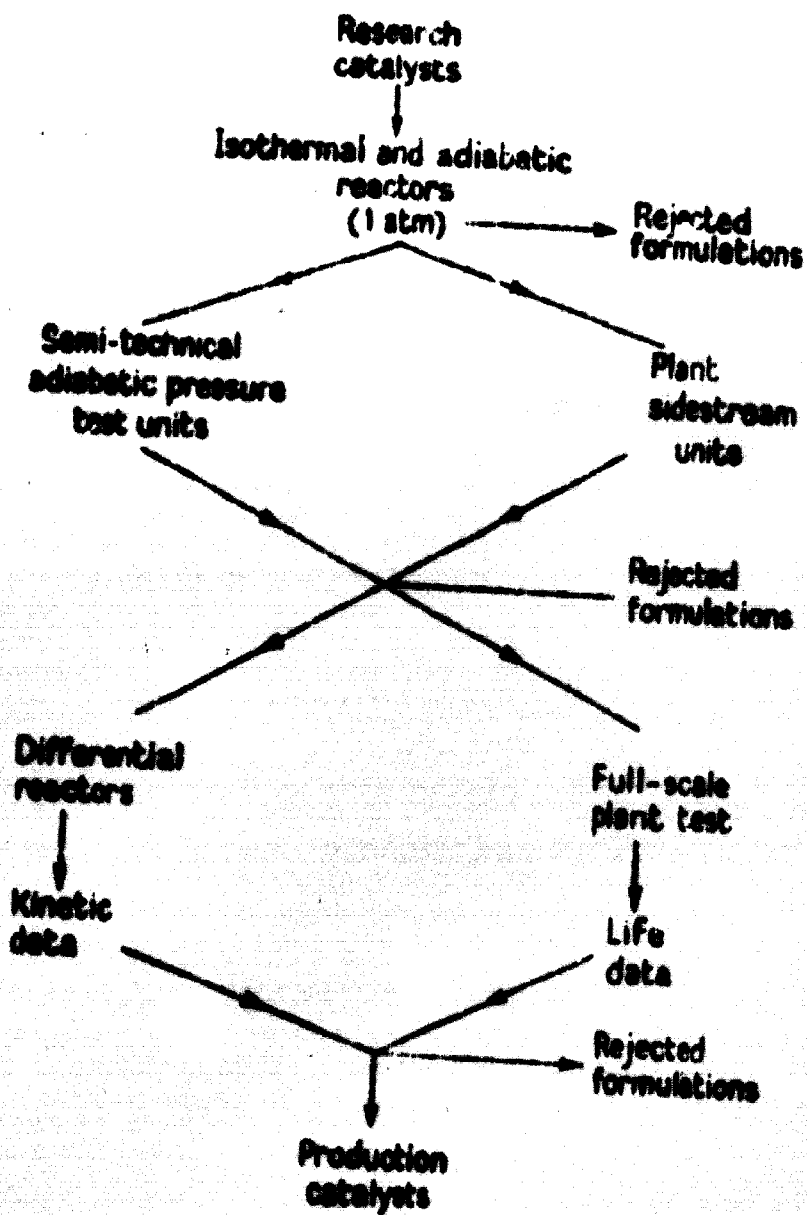


Fig 1 Steps involved in developing a successful catalyst.

semi-technical tests satisfactorily, over an extended period of time, are subsequently submitted to full scale plant tests.

5. The rapid screening tests represent the first level of testing defined above, the semi-technical and plant sidestream tests represent the second level while operation in differential reactors represents the third and highest level.
6. The performance of a catalyst is generally assessed in terms of the rate at which it promotes a desired reaction. It is generally defined by the following equation

$$\text{reaction rate} = \text{activity} \times e^{-E/RT} f(P, V, X)$$

Under equivalent reaction conditions, i.e. equal temperatures, pressures, reaction volumes, and mole fractions, reaction rate is proportional to catalyst activity.

7. Before considering how catalyst activity is measured, the properties controlling mass transfer to and from the catalyst surface must be considered. The flow of reactants to the surface of the catalyst can, and usually does, affect the measured activity of the catalyst. Specific activity is the measure of the reaction rate available when there are no external limitations. The observed activity of the catalyst may not be equal to the specific activity because of external limitations.
8. Unless the mechanism by which reactants are transported to the active surface of the catalyst and the products transported away is fully understood and the rate limiting step or steps are recognized serious errors in catalyst testing may be made. The commonly accepted route by which the reactants reach the active surface of the catalyst in pellet form is shown diagrammatically in Figure 2.

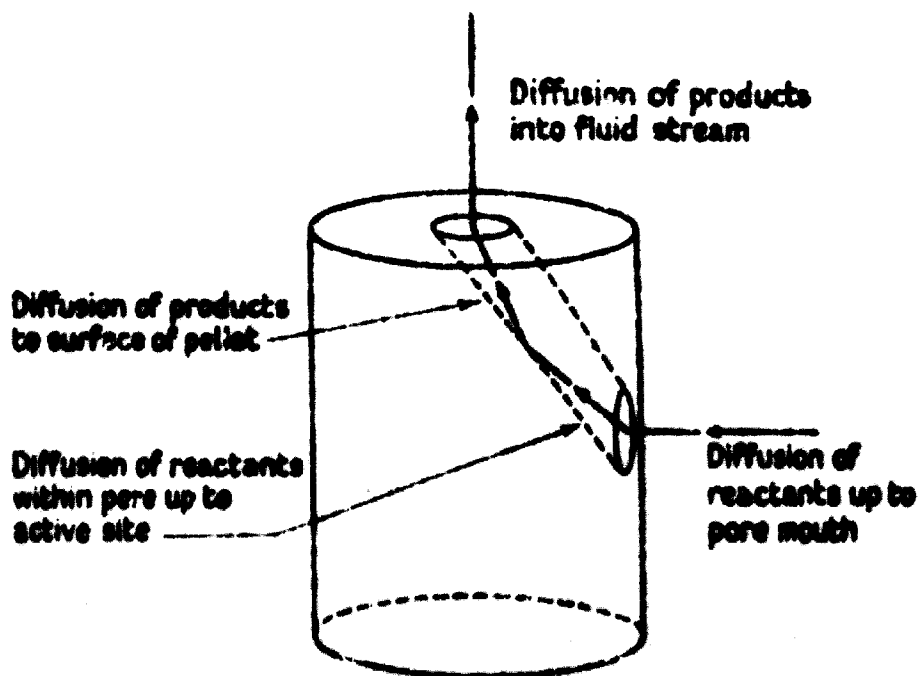


Fig 2 Diffusion of reactants and products within catalyst pellets

The first stage is normally regarded as the diffusion of reactants to the surface of the pellet through the gas film which surrounds it. Having reached the pellet, the reactants pass through the pellet's internal structure consisting of macro, micro and sub micro pores, before they reach the active surface. The reaction products desorb from the catalyst surface and return to the bulk of the gas stream by the reverse procedure. The behaviour of a catalyst in a commercial plant, depends on the overall effect and this is often very different from the effect which would be postulated from the catalyst's specific activity. Care must be taken, therefore, in catalyst testing to make sure that the catalyst test accurately reflects the limiting process or processes as found in a commercial unit.

9. With most catalysts the overall reaction rate can be considered to depend on three separate processes

- 1 The diffusion of one of the reactants from the gas phase to the surface of the catalyst particle

2 The diffusion of one of the reactants in the pores of the catalyst particle

3 The rate of reaction at the active surface of the catalyst

Process 1 is called gas film diffusion and process 2 pore diffusion.

Normally the difficulty of mass transfer inside the pellets outweighs that of transfer from the gas stream to the pellet surface. At high reaction rates, and with low gas velocity, however, gas film diffusion may become limiting.

It is worth remembering that this type of limitation is more likely to occur at gas velocities used for catalyst testing than under plant conditions, where reactor height usually ensures that velocities are also high. Care must be taken therefore, to find out if testing is being carried out in this region and, if so, steps must be taken to avoid it.

10. If pore diffusion is a contributing limitation, the rate of reaction is dependent on the properties of the catalyst particles, for example, size, voidage, and surface area. It is therefore important, that catalyst testing should, wherever possible, be carried out on the full size catalyst pellets. Serious errors may be made in determining the catalyst activity if different sizes of catalyst particle are used during the test. The best experimental method for assessing the importance of pore diffusion is to measure the rate of reaction with varying particle size. In the absence of pore diffusion limitations, the reaction rate will remain unchanged. It should also be borne in mind that pore diffusion limitations increase with temperature and pressure and catalyst activity measured at moderate temperatures and at atmospheric pressure will not usually be applicable for full scale plant operation at higher pressures.

11. While considering possible physical limitations in testing methods, it is worthwhile to point out the danger of so called "edge effects" in small scale reactors. Where catalyst particles are in contact with the walls of a tubular reactor, the voidage is much higher than that in the centre of the catalyst bed. Because of this, there is a preferential flow route for the

gas along the edge of the catalyst bed. In a full scale reactor this unevenness of flow is negligible in relation to the total flow through the catalyst bed, but in small scale reactors the amount of catalyst in contact with the reactor wall is much greater in proportion and can result in errors in the measurement of catalyst activity. This is especially true for reactions where high conversions are obtained, such as during methanation. If slip is occurring under these circumstances, not only will the catalyst activity be underestimated, but the difference between differing catalysts will be minimized. A poor catalyst of smaller particle size may well compare favourably with a better catalyst of larger particle size, not only because of the smaller pore diffusion limitation but also because of the smaller edge effect which will be obtained.

III. REACTOR TYPES

12. The simplest type of reactor is one which consists of a tube, made of glass or metal, containing the catalyst through which reactant gas is passed. This basic design can be developed for use in either of the three levels of catalyst testing. It is the type of reactor most widely used in ICI's catalyst testing programme.
13. There are two fundamental types of continuous test reactor, the integral reactor and the differential reactor. The simple tubular reactor is the type used for integral reactor systems. With an integral reactor a reactant concentration profile will develop along the length of the catalyst bed, and the rate of reaction along the length of the converter will change. With a differential reactor the reactant concentration in contact with all parts of the catalyst bed will ideally be the same, and the rate of reaction will therefore be the same at all points in the converter. This is why the differential reactor is potentially the more suitable for the derivation of kinetic data, because the reaction rate can be calculated directly from the flow rate and the inlet and exit reactant concentrations. In the case of an integral reactor, an integration, which may be impossible to carry out algebraically, is often required. Integral reactors are, however, much

simpler to operate, and this is the type normally used for the bulk of catalyst testing.

14. Integral tubular reactors can be operated in three different ways: isothermally, adiabatically or pseudo-adiabatically (that is neither isothermally nor adiabatically). In practice isothermal operation is ~~usually~~^{rarely} possible, particularly with reactions having a high heat of reaction, because of heat transfer limitations. These limitations are important because poor heat flow control, leading to small temperature differences in the bed, can have a very severe effect, since the rate of reaction is exponentially dependent on temperature.
15. With exothermic reactions, tubular adiabatic reactors are commonly employed in order to overcome this problem. The temperature control system is arranged in such a way as to prevent heat flowing either in or out of the reactor through the walls. Consequently a temperature profile similar to that found in operating plants develops along the length of the reactor, the extent and shape of the latter depending on the heat of reaction, the specific heat of the gas and the reaction kinetics. Small scale adiabatic converters are therefore useful for life tests or for simulation of full scale plant operation where adiabatic operation is normal.
16. In the first level of testing, perhaps 100-1,000 formulations are tested in a catalyst research programme. It is often convenient to do a quick screening operation using even simpler reactors operating pseudo-adiabatically. In this case, the reactor is surrounded by a heating jacket as in the small scale adiabatic reactor, but no attempt is made to balance the heat flow in or out of the reactor. In this case it is essential that the test conditions for each catalyst be as near identical as possible, so that a true comparative test is obtained. The test is essentially qualitative and can sort good catalyst from moderate catalyst from bad catalyst but it cannot sort out good catalyst into very good and not so good.

17. In all comparative testing it is essential that a standard catalyst size is used, together with a standard catalyst volume, standard temperature conditions and standard gas composition. The simpler the test the more standard the conditions must be if meaningful results are to be obtained.
18. In addition in choosing the operating conditions it should be borne in mind that it is much more difficult to differentiate between catalysts under conditions of high conversion or conditions which result in a product gas being close to equilibrium.
19. When more advanced testing is required it is necessary to have much more complicated apparatus. Whether the testing unit is a semi technical laboratory unit or a plant sidestream unit, great care must be taken in its design. Whereas fairly simple control and measurement systems may be used at the first level of catalyst activity testing, much more precise methods must be used at this level. These units are essentially miniature versions of full scale plant units and so require miniature versions of all the control and measurement devices which are used in a full scale plant. In addition, equipment is required to maintain adiabatic operation and to provide further information, such as temperature profiles, on the behaviour of the catalyst which is not normally required in a full scale plant. In order to obtain as much precise information as possible, attention must be paid to the detail design. The edge effect caused by miniaturisation of equipment has been mentioned earlier. A similar effect is that of conduction of heat from the exit of the catalyst bed to the inlet along the reactor itself. The reactor material must therefore be chosen so as to minimize this effect. The location of measuring devices for the control circuits is also important and the position of them must be chosen so as to give quick response to changes in operating conditions. This is particularly true of those thermocouples controlling the adiabatic operation of the converter. They must be located so as to minimize the fluctuations of heat inflow to heat outflow if the system controlling the adiabatic function is sluggish in its response.

20. Under these conditions, extremely good results can be obtained and catalysts can be sorted with great accuracy. Life testing over several months can be carried out and the effects of poison measured on sidestream units. The optimum catalyst may then be developed by balancing the requirements of a good life, high initial activity and poison resistance.
21. Although it is generally more difficult to derive a kinetic equation from integral data than from differential data it is nevertheless possible to obtain a large amount of information from a relatively small number of experiments and often it is possible to derive design equations predicting full scale plant operations from adiabatic integral experiments. In any case, a design equation obtained from differential data must be cross checked against integral data obtained both on the small scale and the full scale before it can be used with any degree of confidence.
22. In a normal research programme one might think in terms of testing 20-30 catalysts on semi technical units and about 10-20 on plant sidestream units before choosing the two or three formulations to be investigated more fully in differential reactors and in full scale plant tests.

IV. DIFFERENTIAL REACTORS

23. In differential reactors the reaction rate is the same for all the catalyst being tested and changes in reaction rate caused by changes in each variable can be studied separately. The procedure can be very time consuming because a very large number of experimental results over a wide range of conditions may be needed before a design equation can be produced. The exercise is therefore very expensive, but is worthwhile for a catalyst researcher because the very accurate results obtained are extremely useful in plant designs.
24. Single pass tubular reactors may be used under differential conditions. Here the conditions are chosen so that the amount of reaction over the catalyst is very small and thus the change of concentration of reactants in the gas phase and the change in temperature due to the reaction may be ignored. The use of

single pass tubular reactors, however, suffers from the disadvantage that very accurate composition analyses are required if an accurate reaction rate is to be calculated from the small differences between inlet and exit concentration. With modern methods of analysis this can be done for many reactions, but it is often better to use other methods if very accurate results are required.

25. This analytical requirement is removed when a recirculating system is used. With this system, part of the exit gas is recycled and mixed with a small amount of fresh gas. After some initial changes, the system will reach a steady state and the composition of the make up gas and exit gas are then measured together with the exit flow rate. Since the pass conversion depends on the recycle rate, which can be very large, the system can be made to approach very closely to true differential operation.
26. The overall concentration difference between the feed gas and the exit gas can be very different, even though the converter is operating differentially, which decreases the importance of analytical error. A major disadvantage of this system, however, is the potential for build up of by products or impurity concentrations. For this reason it cannot be used for reactions in which there are likely to be by product formations but it has been used very successfully to study the kinetics of ammonia synthesis from nitrogen and hydrogen (see later).
27. A type of differential reactor which is being widely used within ICI, is the continuous stirred tank reactor. In this system, the reactants are fed continuously into a tank where they are perfectly mixed and brought into contact with the catalyst by a stirrer. In order to balance the inflow of reactants, gas which contains both the reactants and the products is continuously removed from the system. Within ICI it has been found convenient to contain the catalyst in baskets which form the blades of the stirrer and it has been found particularly suitable for the differential study of poisoning reactants since all the catalyst is exposed simultaneously to the same concentration of poison.

28. The simplifying assumption of perfect gas mixing is necessary if continuous stirred tank reactors are to be used for the derivation of kinetic data, so the occurrence of such mixing must be checked before carrying out experimental work. If any deviations from ideal behaviour are discovered their causes should be traced and the situation corrected, because the experimental data resulting from a combination of the effects of both imperfect mixing and the chemical reaction are usually too complex for accurate interpretation.

V. STRENGTH TESTING

29. Catalyst particles must be strong enough to withstand four different forms of stress.

- 1 Abrasion during transit
- 2 Impact when loaded into the converter
- 3 Internal stresses, especially those occurring during reduction or being brought on line
- 4 External stressing caused by pressure drop, catalyst weight and possibly thermal cycling

30. It is generally difficult to subject catalysts to a simulation of the conditions which affect their strength during life while measuring the reaction of the catalyst to those conditions. ICI uses two types of general tests which may be applied to both new catalyst and to catalyst subjected to operating conditions. One test is to measure the crushing strength of the catalyst and the other test involves tumbling the catalyst.

31. The equipment used for the crushing strength is manually operated and consists of two flat polished platens one set above the other. The pellet is placed on the lower platen and the platen raised manually until it comes into contact with the upper platen. The pressure generated by compressing the pellet between the platens actuates a piston which hydraulically activates a pressure gauge. The piston has a cross section of exactly one square inch so the pressure gauge measures the load on the catalyst particle in pounds

weight. The load at which the catalyst particle breaks is then noted. This test is carried out on at least 20 particles and the mean taken. For solid pellets it is usual to measure the crushing strength with the catalyst pellet set vertically so the platens come in contact with the flat ends of the pellet. For extrusions and rings the particles have to be laid horizontal, and in the case of rings, it is normal to replace the bottom platen with a bar shaped one. Some care must be taken in the operation of the machine as the crushing strength obtained does vary with the speed of compression, but it has been found better to use a simple manually operated machine than to complicate it with a mechanical drive.

32. In the tumbling test a fixed volume of catalyst (normally 25 mls) is placed in a steel tube which is rotated end over end at a speed of 40 rpm for 45 minutes (3,600 falls). After the test catalyst is sieved to remove dust and catalyst chips and the percent weight loss is measured.
33. In establishing these tests as standards, ICI carried out extensive investigations into the parameters measured in these tests and their relationship to the strength requirements outlined above. Most of this work was a once and for all project relating the pressures involved in a catalyst bed with the bed size and shape and involved analysing the way catalyst forming methods affected the final strength. For each new catalyst, however, the relationship between strength as new and strength under operating conditions has to be determined so as to establish parameters for production control.
34. One interesting outcome of ICI's work on catalyst strength, was that the most important requirement for high strength in a catalyst was resistance to the forms of stress outlined under 1 and 2 above and that the actual strength required during operation was much less than that required for handling the catalyst before operation.

VI. PHYSICAL CHARACTERISTICS

35. When deciding on the required strength of a new catalyst, it is often necessary to arrive at a compromise between the strength of the catalyst and other factors. The other major factors which are affected are the density of the catalyst and thus its pore structure and surface area. Generally speaking, because of the pore diffusion limitation which exists for most catalysts, it would be advantageous to make lower density, more porous catalyst with higher surface areas. The requirement of sufficient strength to withstand handling of the catalyst, however, often results in a catalyst being denser than otherwise would be required.
36. Density, pore structure, and surface area measurements are usually made at both the first and second level of catalyst testing and are done by the normal techniques of mercury density, helium density, and BET surface area measurement. In ICI we now use Krypton as the absorbent gas in our BET experiments. At the highest level of testing information can be obtained by measuring the pore size distribution using a mercury porosimeter or adsorption-desorption techniques.
37. ICI has also developed a method for measuring the surface area of the active species in the catalyst rather than the total surface area. This is done by using a gas which is chemisorbed only on the active species and the diagram below (Figure 3) shows a correlation between the surface area of the active species in low temperature CO shift catalyst with the catalyst activity.

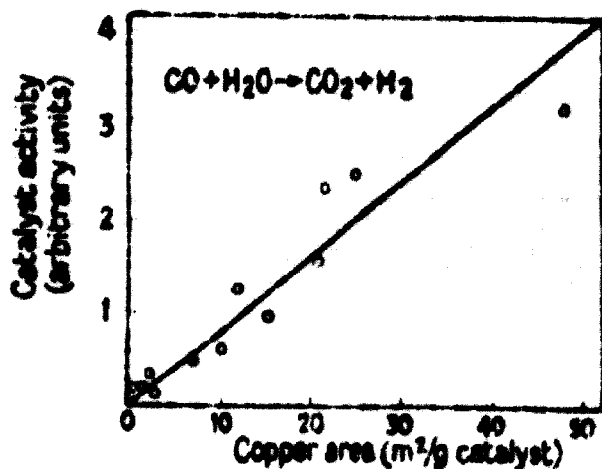


Fig 3 Relationship of catalyst geometry to catalytic activity. Copper crystal sizes and exposed surface areas measured by X-ray diffraction and oxygen chemisorption.

This was very useful in defining the mode of operation of the catalyst. At the fundamental level of testing it is possible to engage the whole range of physical measurement and testing techniques now available to research chemists to assist in understanding the mechanism of the reaction, the way the catalyst entered into the reaction, and the way the active species are produced during the preparation of the catalyst. ICI has found X-ray diffraction particularly useful in this respect and has used it both to follow the various steps in catalyst manufacture and also to measure the crystallite sizes existing in the final catalyst product.

VII. APPLICATION TO PARTICULAR CATALYSTS

38. The above discussion is relevant to almost all catalyst testing but each catalyst used in a modern ammonia plant has certain points which need to be borne in mind when testing is carried out. These points are outlined in the following paragraphs.

VIII. HYDRODESULPHURISATION CATALYST

39. Generally speaking one uses the standard series of tests to test hydro-desulphurisation catalysts but it is better to use standardised feedstocks, whether gaseous or liquid hydrocarbons, because of the complication of the different reaction rates obtained with different types of sulphur compounds. When comparing different catalysts or different batches of the same catalyst it is essential that exactly similar feedstocks are used so that synthetic feedstocks which can be reproduced time and time again are most convenient. In ICI, a useful standard test involves the use of thiophene doped heptane.

IX. ZINC OXIDE

40. When comparing zinc oxide catalyst, the total sulphur pick up possible with the catalyst is meaningless, because this is merely a measure of the available zinc oxide in the catalyst. What is important is the measure of the amount of sulphur which may be picked up under normal operating conditions before significant quantities of sulphur break through the catalyst. Besides the standard series of tests, and again one must bear in mind the question of

the complication of different sulphur types, ICI use a standard test to measure the absorption capabilities of zinc oxide catalyst. The test uses 5% hydrogen sulphide in a hydrogen stream which is passed through the zinc oxide catalyst in a pseudo-isothermal reactor at 370°C. When the first minute trace of hydrogen sulphide breaks through, as measured in lead acetate solution, the test is stopped and the average sulphur content of the catalyst bed measured. This is only a simple, semi-quantitative test with an accuracy of plus or minus 5-10%, but is a useful accelerated life test for differentiating between good catalyst and those which are second rate.

41. Because ICI catalyst 32-4 is in the form of granules, the crushing strength tests are meaningless and the standard strength test is the tumbling test.
42. An interesting example of the use of sophisticated techniques is the use of electron beam scanning in the study of the absorption of hydrogen sulphide by ICI catalyst 32-4. In this case the sulphur profile across a cross section of a catalyst granule is measured after varying degrees of sulphur absorption. The granule cross section forms the target in an X-ray tube and the sulphur concentration is measured by the amplitude of the characteristic fluorescence given off by the sulphur atoms. This work shows that the controlling rate in the absorption is diffusion of the reactants through the sulphided layer.

X. REFORMING

43. Unfortunately there appears to be no short cut to accurate testing of reforming catalyst. A simple test had been found to be of very little use in evaluating primary reforming catalyst. For a long time full size tube tests were the only way in which reliable information could be found, and this proved very costly. ICI have, however, developed small tubular reactors with a complex heating system to simulate the heat input in a reformer tube. These units have proved very successful for tests in the first and second level of catalyst testing but have proved a little less than 100% in simulation of full scale plant operation, not least because the catalyst has to be in smaller granules than full size rings. The ultimate test remains,

therefore, a full scale trial in a full size tube in a semi technical reformer.

44. ICI have also carried out strength tests under operating conditions but it was generally found that tests on the strength of the catalyst discharged after operation in a full size tube were sufficient indication of the strength during operation.
45. Tests of a secondary reformer catalyst are similar to those for a primary reformer, except that the temperatures which the catalyst must withstand are much higher. In this context it may be pointed out that thermal shock tests on these types of catalyst are more or less meaningless. If the high temperature strength of the catalyst is to be determined, it must be taken using a standard strength test while under simulated operating conditions.

XI. CO CONVERSION CATALYSTS

46. Whereas the high temperature conversion catalyst has been manufactured for many years, low temperature conversion catalyst are a recent addition to the ammonia plant scene. In developing their present catalysts, ICI catalyst 52-1, ICI followed the process steps outlined in Figure 1. Special catalyst test units were built for the programme, both in the laboratory and on a semi technical scale. All the units were truly adiabatic and were designed for sorting tests both for initial activity and life and run continuously under full automatic control for periods of up to several months. The laboratory units consist of two reactors in series so that two different catalysts may be tested on each one at any one time. A continuous on line gas chromatograph measures the inlet and exit concentrations to each reactor and the dry inlet gas of the correct composition is obtained by mixing the purified constituent gases in a mixer/preheater/final purifier. The water is added by a metering pump via a vaporiser. The pressure units are exactly similar but are built to withstand the normal elevated pressures found in ammonia plants. Studies of the kinetics of the process and the effects of poisoning were carried out using a stirred tank reactor technique as outlined above. Under these conditions the normal adiabatic reactors were substituted by a stirred tank reactor. These units are also used for high

temperature conversion and methanation testing.

47. Methanation presents a specially difficult problem because of the high conversion to low carbon oxide levels normal in ammonia plants makes differentiation between catalysts difficult under simulation conditions. One, therefore, has to choose a different set of conditions that still measure the correct apparent activity, where differentiation may be made. In ICI use is mainly made of high space velocity tests for these sorting tests. Even so a special analytical technique and apparatus were developed to enable the accurate analysis required to be made.

XII. AMMONIA SYNTHESIS

48. For studies on ammonia synthesis catalysts very similar reactors to those used for CO conversion are used but, of course, only pressure units can be used because equilibrium ammonia concentrations at atmospheric pressure are negligible. Because of the large volume of gas required ICI uses an ammonia cracker to produce the feed gas to its small scale ammonia units. The differential reactor used for ammonia kinetics and poisoning studies was a recirculatory type similar to that developed by Professor G K Borekov and mentioned above. To maintain isothermal operation, the reactor is immersed in a heated, fluidised sand bath.

XII. THE CATALYST MANUFACTURER

49. Once the manufacturing method has been established, further use of the more sophisticated testing techniques are unnecessary. If the manufacturing technique is applied rigorously, good catalyst should always be produced but the catalyst manufacturer has to carry out enough tests to measure critical parameters which directly or indirectly identify the catalyst he is producing. He must determine that it is similar to that originally developed under laboratory conditions. These tests must, however, be simple so as to allow them to be carried out quickly on a regular basis to control production.

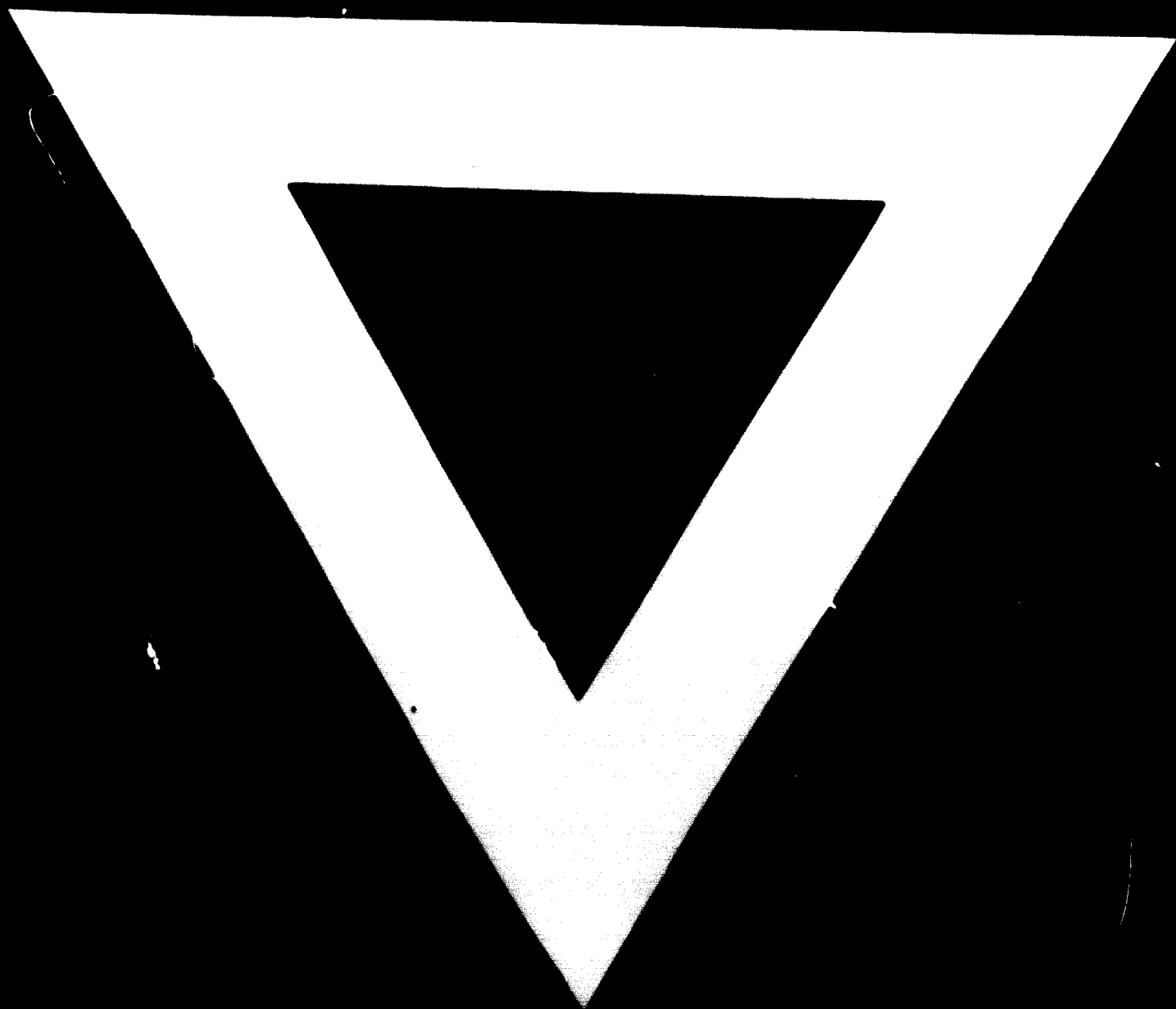
50. Besides chemical analysis, strength tests are also routine on all catalysts. Also routine are pellet density, bulk density and size measurements because these infer the catalyst has the requisite internal pore structure.
51. Catalyst activity measurement is avoided unless absolutely necessary because of its time consuming nature and this is especially true for those catalysts where activity measurement must be measured under elevated pressure. If initial activity must be measured then simple pseudo-adiabatic or pseudo-isothermal units are preferred to simplify the procedure. Activity measurements are, of course, required when other characteristics do not infer that a catalyst must have good or bad activity. Similarly surface area or pore volume measurements must be made on some catalysts if measurement of density and other characteristics do not enable one to infer that an adequate pore structure is present.
52. Besides tests on the finished product it is normal to carry out tests on intermediates and vital stages in the production strength.

XIV. THE CATALYST USER

53. It is unrealistic for a catalyst user to envisage becoming a full scale catalyst tester. He is only interested in testing his catalyst outside of his plant every 2-5 years as they require changing and this is hardly sufficient opportunity for him to build up the required expertise in his organisation. Nor is it likely that he would be in a position to spend the required amount of money. Most fertilizer producers who are not involved in catalyst manufacture do not test catalyst themselves but leave it to the catalyst suppliers. They rely on building up a relationship of mutual trust between them and their suppliers. Many fertilizer producers find they need to concentrate all their resources on the running of their plants and this leaves little opportunity for them to develop their technical abilities as catalyst testers.

54. Some simple tests can be carried out by a catalyst user, if he so desires. Tests which lend themselves to be done by a catalyst user are chemical analysis, density and strength measurement. There is, however, really no possibility of fully checking on your catalyst supplier. Because of this there is no substitute for the relationship of mutual trust mentioned above and the competence and reliability of the catalyst supplier should play a major role in the catalyst choice.





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