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September 1987
ENGLISH

DEVELOPMENT OF NOVEL SHAPE SELECTIVE ZEOLITE CATALYSTS

DP/IND/87/007

INDIA

Report*

Prepared for the Government of the Republic of Indi
by the United Nations Industrial Development Organization,
acting as executing agency for the United Nations Development Programme

Based on the work of Dr. Sigmund M. Csicsery
CTA

Backstopping officer: M. Derrough, Industrial Operations Technology Division

United Nations Industrial Development Organization
Vienna

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Summary

This Report describes my visit to NCL in Pune, between November 24 and December 4, 1987. The Report contains the following documents.

1. Detailed Work Plan (Quarterly details for 1988, yearly details for subsequent years)
2. Detailed Work Schedule
3. Schedule of EXPERTS
4. List of Equipment to be Purchased in 1988
5. List of All Equipment to be Purchased between 1987 and 1992

5a. Justification for Quantitative Powder XRD software purchase

5b. Justification for upgrading of the Esca-3-MK Unit to handle high pressure reactions

6. Job Descriptions (with Recommendations for Experts, Background Information and Notes)
7. A short note on 'Catalysis Research at NCL - The place and role of the UNDP Project in it.
8. Copy of ICMA Award Document
9. Copy of the letter of the Prime Minister, 12.2.87.
10. Revised specifications for item No. 19 in project document.

NCL is a very good Institute. The Catalyst Group is particularly efficient and well organised. Notable accomplishments of this group are two inventions which eventually became full scale industrial processes. These are Xylene Isomerization and Benzene Alkylation with Ethylalcohol. The former has lower xylene loss than competing technologies. The latter uses ethylalcohol (available in India) instead of scarce ethylene. It is a new process. The attached letter of acknowledgement by the Prime Minister Rajiv Gandhi, and the 1985 ICMA Award show the significance of these two accomplishments.

It is my feeling that this Project is a good one, and that NCL is a competent Institution to carry it to successful completion. NCL has the necessary manpower and facilities. I recommend full UNDP support.

Next week, I will visit the plants of catalyst manufacturers. A separate report will describe my observations.

Dr. Sigmund M. Csicsery

December, 4, 1987.

Dr. Sigmund Csicsery

WORK PLAN

The major objectives of the present UNDP project may be stated as :

1. Synthesis and characterization of molecular sieves (zeolites) of novel chemical composition containing elements other than aluminium/silicon in the lattice framework (for example Fe, B, etc.)

2. Synthesis and characterization of zeolites with new structures, and

3. Discovery and development of industrial applications for these new zeolites in shape selective catalysis.

Even though there is likely to be certain overlaps, it is proposed that the first three years of this five year project be devoted mainly to achieving objectives (1) and (2); synthesis and characterization of new zeolites, while the development of applications be carried out mainly in the last two years.

A detailed work plan for 1988, and a more general plans for the following years are given below. A Work Schedule summarizes all these activities.

DETAILED WORK PLAN FOR 1988

The major emphasis during this period will be in the following two areas :

1. Synthesis and characterization of zeolites with faujasite-type structure and containing iron in the lattice framework.
2. Characterization of the structural, physicochemical and catalytic properties of a new zeolitic material of unknown structure.

These two areas of activities are discussed below :

(A): Faujasites containing Fe in lattice framework

Even though natural zeolates usually contain ppm quantities of iron in the lattice framework, the systematic synthesis of zeolites (ferrisilicates) containing controlled quantities of lattice iron has been reported only during the last 5-6 years. Groups, including NCL, have reported the synthesis of ZSM-5 type zeolites containing Fe in the lattice. The presence of Fe in lattice sites has been unequivocally confirmed by ESR, Mossbauer and IR spectroscopic techniques. The isomorphous substitution of Al by Fe in the ZSM-5 lattice leads to some interesting differences in the catalytic properties of the zeolites in reactions like xylene isomerisation, dewaxing and production of light olefins from methanol.

Since the feasibility of substituting Al by Fe in ZSM-5 frameworks has been established, it would be interesting to attempt this isomorphous substitution in other zeolitic structures. During the first year of this project, it is proposed to synthesise faujasites containing Fe in the lattice. Such a zeolite would be interesting for the following reasons : It is known from our earlier work on pentasil ferrisilicates that Fe insertion in tetrahedral sites is difficult and that not more than 1-2 iron ions per unit cell (of the pentasil zeolite) can be isomorphously introduced. In other words, zeolites with SiO_2/Fe_2O_3 lower than about 60 are unstable while those with higher values an be synthesised with relative ease.If a similar situation prevails in the case of faujasites also, one should be able to synthesise faujasites with high SiO_2/Fe_2O_3 values. Now, it is difficult to synthesise faujasites with SiO_2/Al_2O_3 values greater than bout 5 and post-synthesis methods which partially destroy the lattice framework (like steeming-extraction, $SiCl_4$ treatment, etc.) have to be resorted to achieve the objective. Highly crystalline faujasites, with a low concentration of acid sites (due to the high SiO_2/Fe_2O_3 values) would have catalytic properties different from those of conventional aluminosilicate analogs. Initial, exploratory studies have shown promise.

During the initial 3-4 months, the zeolite syntheses will be carried out at various conditions of temperature, pressure, pH, OH/SiO_2, templates etc.to achieve insertion of Fe in the lattice. The latter can be confirmed using ESR, IR and Mossbauer spectroscopies which are already available at NCL. In addition, NCL has

recently procurred a solid state NMR spectrometer (Bruker MSL-300) which is expected to be commissioned shortly. This is a very powerful tool for the characterization of zeolites including isomorphous substitution by Fe.

Once the feasibility of synthesising ferrisilicate faujasites is established during the initial 3-4 months, a two-pronged strategy will be adapted for the next phase, 4-12 months. On the one hand,the kinetics of synthesis will be investigated to enable at a later date, a scale-up of the synthesis process to a larger batch size; second,a comprehensive program to characterize the physicochemical and catalytic properties of this new zeolite will be undertaken. Correlations between synthesis conditions and physicochemical properties like SiO_2/Fe_2O_3, porosity, crystal size, acidity, thermal and hydrothermal stability and adsorption capacity for various adsorbates will be explored. Such data are crucial prerequisites in developing commercial applications for any new zeolites. In addition, the catalytic activity of the new ferrisilicate in various model reactions will be studied in the second half of 1988 when the twin-reactors are expected to be installed and in operation at NCL. Some of the model reactions that are envisaged are :

1. Alcohol dehydration
2. o-Xylene isomerisation.
3. Toluene disproportionation.
4. Cracking of n-paraffins (hexadecane).

In addition, we may also test hydrogenation and dehydrocyclization activities of Pt-loaded zeolite. Fe in zeolites

affects Pt-distribution in a favourable way.

(B) : Characterization of new zeolite

A new crystalline zeolitic material of unknown structure has recently been synthesised at NCL. Since any new zeolite is of fundamental and potential technical interest, it is proposed to initiate a major program on a comprehensive study of its structure, physicochemical and catalytic properties. Testing facilities will be expanded to more reaction types to ensure that no potential application is overlooked. Two types of new tests will be implemented :

a : Model compound test (e.g. speciousness index). Here we will need experts in zeolite reactions.

b : Commercial reactions (e.g., like the ones listed above).

In the first 3-4 months, further exploratory synthetic studies will be carried out to delineate and standardise conditions to get good quality zeolite crystals in high, reproducible yields. In the remaining months of the year, the physicochemical and catalytic properties of this new zeolite will be studies in a systematic and thorough manner.

The equipment that are proposed to be procurred in the first year are given in Table 1.

WORK PLAN FOR YEARS 1989-92

(SECOND TO FIFTH YEARS OF THE PROJECT

(GENERAL SUMMARY)

SECOND YEAR

A: Synthesis of zeolites with novel framework structures

At present, there is a need for synthsising aluminosilicate zeolites with a 3 dimensional channel structure without cavities and with a pore diameter in the range 6-8$\mathring{A}$. The absence of cavities will minimise coking, a shape selective reaction, and the pore diameter will ensure ingress of substituted aromatics and branched hydrocarbons. It is proposed to synthesise this class of zeolite using bulky long chain quaternary ammonium salts.

B : Modification of zeolites to enable them to exhibit novel catalytic properties

Modifications of known and novel zeolites by steaming, silylation, and acid treatment to change the Al and hence acid site distribution and accessibility will be carried out and the performance of the zeolite in typical acid catalysed transformations will be evaluated.

C : Synthesis of zeolites with novel chemical composition and lattice framework structure

In the zeolites with novel framework structure discussed, attempts will be made to introduce elements like B or Fe in the lattice. Various physicochemical techniques (MAS, NMR, ESR, XRD, etc.) will be used to confirm the incorporation of heteroatom(s) in the lattice.

THIRD YEAR

The bench scale evaluation, over long periods of time, of the novel zeolites prepared during the first two years will be carried out. Chemical reactions of importance in the petroleum and chemical industries like xylene isomerisation, naphtha reforming, hydrocracking, hydrodewaxing, etc. will be chosen for such evaluation.

FOURTH AND FIFTH YEARS

During the fourth and fifth years, those novel zeolites which show promise in specific chemical applications will be identified and further work will be focussed on two areas :

1 : Scale-up of the synthesis of the novel zeolites to pilot plant and commercial batch levels. This will be done in collaboration with catalyst manufacturing companies.

2 : Process development leading to preliminary process design packages for selected chemical processes of industrial importance.

A final report containing all publications and internal reports from NCL in the area of zeolite catalysts prepared under this project will also be prepared and submitted during this period.

WORK SCHEDULE

The schedule of work alon gwith UNDP inputs are summarised below.

WORK SCHEDULE

Period	Activity	Person In-charge	Man-power utilised man months	Item No (Ann. V of proj. document	Equipment to be provided by UNDP (Cost, S)	Fellow-ships m/m person	Study tour m/m person
1988							
1 Qr.	1. Synthesis of Fe Faujasite	- A.N. Kotasthane	50	2	XRF Spectrometer (1987) (200,000)	-	-
	2. Characterisation	- S.G. Hegde	50	11	Twin Reactors (120,000)		
	3. Activity testing	- S. Sivasanker	120	7	Porosimeter (80,000)		
2 Qr.	1. Synthesis of Fe containing zeolites	- V.P. Shiralkar	50	8	Gas chromatographs (70,000)	6 2*	3 **
	2. Characterisation	- S.G. Hegde	50	1	Reactors for synthesis (50,000)		
	3. Activity testing	- S. Sivasanker	120				

* A.N. Kotasthane, A.J. Chandwadkar.
** P. Ratnasamy, S. Sivasanker, B.S. Rao.

Period	Activity	Person In-charge	Man-power utilised man months	Item No (Ann. V of proj. document	Equipment to be provided by UNDP (Cost, $)	Fellow-ships m/m person	Study tour m/m person
3 Qr.	1. Synthesis of zeolites with new structure	- V.P. Shiralkar	50	10	High Pr. valves (10,000)	9 3 *	-
	2. Characterisation	- S.G. Hegde	70	12	Gas Regulators (10,000)		
	3. Activity testing	- S. Sivasanker	120				
4 Qr.	1. Synthesis of zeolites with new structure	- V.P. Shiralkar	50	New in place of 5,6 & 9	Accessory for XRD (60,000)	3 1**	
	2. Characterisation	- S.G. Hegde	70				
	3. Activity testing	- S. Sivasanker	120				
				Total :	200,000 (1987) 400,000 (1988)		

* A.N. Kotasthane, A.J. Chandwadkar, J. Kuruvilla.
** J. Kuruvilla

Period	Activity	Person In-charge	Man-power utilised man months	Item No (Ann. V of proj. document	Equipment to be provided by UNDP (Cost, $)	Fellow-ships m/m person	Study tour m/m person
1989							
	1. (a) Synthesis of B, Cr Fe containing zeolites	- V.P. Shiralkar	200	3	Adsorptomat (140,000)	18.0	6.0
	(b) Synthesis of zeolites with new structures	-		4	Catatest (200,000)		
	(c) Synthesis of zeolites with new structures and composition	-		13	Gas compressors (50,000)		
	2. Characterisation	- S.G. Hegde	220	14	Back Press. Reg. (50,000)		
	3. Activity evaluation	- S. Sivasanker	480	(19, see attached note)	High Press. Acc. (45,000) ESCA		
				Total : 485,000			

Period	Activity	Person In-charge	Man-power utilised man months	Item No (Ann. V of proj. document	Equipment to be provided by UNDP (Cost, $)	Fellow-ships m/m person	Study tour m/m person
1990							
	1. Synthesis of zeolites containing Ni, Pd, Pt	- V.P. Shiralkar	200	15	Pr. Transd. (20,000)	18.0	6.0
	2. Characterisation	- S.G. Hegde	200	16	H_2O Analyser (10,000)		
	3. Bench scale evaluation	- S. Sivasanker	500	17	O_2 Analyser (10,000)		
				18	Mass flow controllers (10,000)		
				20	Chromatographic data processor (15,000)		
					Total : 65,000		

Period	Activity	Person In-charge	Man-power utilised man months	Equipment to be provided by UNDP (Cost, $)	Item No. (Ann. V of Proj. Document	Fellow-ships m/m	Study tour m/m
1991							
	1. Scale-up of zeolite synthesis to 1 kg. level at NCL	- G.R.V. Krishnan	200	-		18.0	6.0
	2. Characterisation	- S.G. Hegde	120				
	3. Process development	- G.R.V. Krishnan	580				
1992							
	1. Scale-up of zeolite synthesis to 50 kg. level at catalyst manufacturer's premises	- G.R.V. Krishnan	100	-		-	1.5
	2. Characterisation	- S.G. Hegde	50				
	3. Process design and semi-pilot plant tests	- G.R.V. Krishnan	300				
	Final evaluation :						
	GOI						
	UNDP						
	UNIDO						

OUTPUTS : Schedule of Project Review, Judgement and Evaluation

It is proposed that the project be reviewed annually during the visit of the CTA and UNDP Expert. The proposed dates are :

Review 1	-	Nov. 1988
Mid-term Review		Nov. 1989
Review 3	-	Nov. 1990
Review 4	-	Nov. 1991
End of project Review	-	Sep. 1992

An annual progress report will be submitted to the Review Committee members at least one month prior to the review meeting to facilitate the review process.

SCHEDULE OF EXPERTS

Year	Zeolite Synthesis	Zeolite Physicochemical Characterization				Reactions, Chemistry and Engineering		Chemical Process Technology	Process Control	M/M	Number of Experts**
		General	Quant. Powder XRD	TEM	ESCA, EPR NMR		CTA				
1987							1			1	1
1988	3*	2					1			6	6
1989	2	2	1	1	1	1	1			7.5	9
1990	2	1		1	1	1	1	1	1	7.0	9
1991		1				1	1	2	2	5.5	7
Total	7	6	1	2	2	3	5	3	3	27	32

* Includes Dr. Lovink and Y. Ono

** Each for 3 or 4 weeks

In the first year, it is proposed to procure the following items listed in Annexure V of the project document :

	Equipment	Data generated	Price($)	Expected month of procurement
1.	X-ray Fluorescence spectrometer (item 2 in Proj. Doc.,Ann. V)	Analysis of chemical composition	200,000	Jan. 88
2.	Porosimeter (item 7)	Pore structure of catalysts	80,000	Feb. 88
3.	Twin Reactors (Item 11)	Catalytic activity of zeolites	120,000	Mar.88
4.	Gas chromato -graphs (item 8)	Analysis of reaction products	70,000	May 88
5.	Reactors for synthesis of zeolaites (item, 9)	Synthesis of zeolites	50,000	May 88
6.	High pressure valves (item 10)	Catalytic activity of zeolites	10,000	July 88
7.	High pressure high purity gas regulators (item 12)	Catalytic activity of zeolites	10,000	July 88
8.	Half shell furnaces (expendable)	Catalytic activity of zeolites	30,000	July 88
9.	Inconel reactors (expendable)	Catalytic activity of zeolites	10,000	July 88
10.	Accessory for XRD unit (instead of items 5,6 and 9)	Structure of zeolites	60,000	Nov 88

ANNEXURE II-A

LIST OF NON-EXPENDABLE EQUIPMENT TO BE SUPPLIED BY UNDP

(Price in US $)

Sr.No.	Item	1987	1988	1989	1990
1.	Reactor for synthesis of zeolites		50,000		
2.	X-ray fluorescence spectrometer	200,000			
3.	Adsorptomat with MS detector			140,000	
4.	Catatest unit			200,000	
5.	Deleted				
6.	Deleted				
7.	Porosimeter		80,000		
8.	Gas chromatograph		70,000		
9.	Deleted				
10.	High pressure valves		10,000		
11.	Twin reactor units		120,000		
12.	High pressure high purity gas regulators with adaptors		10,000		
13.	Gas compressors			50,000	
14.	Back pressure regulators			50,000	
15.	Pressure transducers				20,000
16.	Moisture analyser				10,000
17.	Oxygen analyser				10,000
18.	Mass flow controllers				10,000
19.	High pressure accessory			45,000	
20.	Chromatographic data processor				15,000
	Quantitative powder XRD software and hardware		60,000		
		200,000	400,000	485,000	65,000

Total : 1160,000

JUSTIFICATION FOR QUANTITATIVE POWDER XRD SOFTWARE PURCHASE

Recently, NCL scientists made a new aluminosilicate material. Catalytic activity tests with model compounds suggest that it is a medium-pore zeolite. Physical measurements (XRD, etc.) suggest that it is different from any known zeolite. We believe, that the NCL scientists succeeded making a new zeolite. Quantitative Powder X-Ray Diffraction is needed for the determination of zeolite structure. Neither the necessary computer software, nor the proper instrumentation, or the expertise needed is available at the present at NCL.

As structural determination is essential for full exploitation of this potentially very significant invention, I believe that UNDP should assist here.

I propose four actions :

1. Buy the software and the necessary instrumentation. A software program is "TREOR" (P.E.Werner), J.App.Cryst., 18 367(1985), or equivalent.

2. Invite Expert(s) to NCL to train quantitative powder XRD techniques. Suggested experts :

   : Dr. A. K. Cheetham, Department of Chemistry, University of Oxford, UK.

   : Dr. P. K. Werner, Arrhenius Laboratory, University of Stockholm, Stockholm, Sweden.

3. Send one or two NCL scientists to a Institution where quantitative powder XRD is practiced for 3-6 months training.

4. Send the sample(s) to the expert's home laboratory for analysis. In this case the expert may not need to come to NCL in June.

NCL has a Phillips 1730 XRD powder instrument.

UPGRADING OF THE ESCA-3-MK II UNIT TO HANDLE HIGH PRESSURE REACTIONS

Presently, at National Chemical Laboratory, we have ESCA-3-MK II Electron Spectrometer, manufactured by M/s V.G. Scientific Ltd (The Birches Industrial Estate, Imberhorne Lane, East Grinstead, West Sussex RH 197 UB), with the following capabilities :

1. X-ray Photoelectron Spectroscopy (Al and Mg anodes)
2. Ultraviolet Photoelectron Spectroscopy.
3. Auger Electron Spectroscopy.
4. Scanning Auger Microanalysis.
5. Depth Profiling facilities.
6. Secondary Ion Mass Spectrometry.
7. Residual Gas Analysis
8. Heating (600°C) and Cooling facilities. (Liquid Nitrogen temperature)

In addition, the present unit has got two chambers, preparation and analysis chambers. The preparation chamber is also provided with gas handling facilities. However, the pressure in the preparation chamber cannot be raised to more than one or two atmospheres. In order to study, in-situ, reactions of metal loaded zeolites, under high pressure, for example, in hydrocracking reactions, we need a high pressure reaction attachment to the ESCA Spectrometer. There is no facility at present to carry out reactions at high pressures. The present instrument can be upgraded by adding a high pressure accessory. M/s V.G. Scientific offers such an accessory, in which the sample can be exposed upto a pressure of 20 bar and can be heated upto 600°C. The sample may be subsequently analysed without exposure to atmosphere. The approximate cost of this accessory would be nearly $ 45,000.

There is already a provision of $ 45,000 in the project document for a data analyser accessory for ESCA (Item No.19 of Annexure V). This data analyser accessory has been procured by NCL (from NCL funds) recently and is hence not needed.

We recommend to replace Item No.19 of Annexure V (i.e, the Data Analyser) with the High Pressure Accessory. The prices of these two items are about the same. Therefore, UNDP will have no additional expense. NCL should procure this High Pressure Accessory from M/s V.G. Scientific.

NOTES FOR "JOB DESCRIPTIONS"

1. The following sections are the same for each "Job Description"

   Duty Section
   Purpose of Project
   Reporting
   Language
   Background Information.

The "Background Information" section is attached only to the first Job Description.

2. The "Experts in Physicochemical Characterization of Zeolite and Catalysts" classification is broken up to four sub-categories.

3. The "Dates Required" is changed on the Job Descriptions form for experts in Physicochemical Characterization of Zeolites and Catalysts from that of the original proposal.

Old "Dates Required"	:	30, 35, 40, 45 months
New "Dates Required".	:	Between Sept. and March of 1988, 1989, 1990 and 1991.

The reason of this change is that NCL has already discovered a new zeolite which needs characterization.

Names of experts who already indicated willingness to go to NCL, if invited, and names of those who should be contacted by UNIDO, are listed on the following pages.

The following have told Dr. Ratnasamy, the Director of the NCL's and Coordinator of the UNDP Project, that if invited, they will come to NCL as experts of zeolite synthesis.

1. Professor Y. Ono (Nov 1988) : 2-3 weeks
   Tokyo Institute of Technology,
   o-Okayama, Meguro-ku, Tokyo, Japan.

2. Professor E. G. Derouane (1989) : 2-3 weeks
   Director, Department of Chemistry,
   Facultes Universitaires Notre Dame de la Paix,
   Rue de Bruxelles, B-5000 Namur, Belgium.

3. Dr. Clarance D. Chang,
   Mobil research and Development Corporation,
   Central Research Laboratory, P.O. Box 1025,
   Princeton, New Jersey 08540, USA.

4. Professor J. C. Vedrine,
   Institute de Recherches sur la Catalyse
   2, Avenue Albert Einstein,
   69626 Villerbanne Cedex, France.

I would also recommend the following :

1. Dr. Jule A. Rabo, Union Carbide Technical Center,
   Tarrytown, NY 10591,Old Saw Mill, River Rd at State Route 100C, USA : (Phone 914-789-2353)

2. Dr. E. Flannigen, Union Carbide Technical Center,
   Tarrytown, NY 10591, Old Saw Mill, River Rd at State Route 100C, USA.

In the area of zeolite characterization, the following have indicated that, if invited, they will come to NCL as experts.

1. Dr.T. R.Hughes,
   4 Keith, Orinda, California 94563, USA
   (Phone : 415-254-1711)

2. Dr. P. A. Jacobs,
   Centre for Surface and Colloid Sciences,
   Katholieke University at Leuven de Croylaan 42,
   B-3030, Leuven, Belgium.

3. Professor H.G. Karge,
   Fritz Haber Institute der Max-Planck-Gasellschaft
   Faradayweg 4-6, D-1000, Berlin 33, FRG.

4. Dr. Nagy,
   Department of Chemistry,
   Facultes Universitaires Notre Dame de la Paix
   Rue de Bruxelles, B-5000 Namur, Belgium.

5. Professor J. Weitkamp,
Department of Chemistry,
Chemical Technology, University of Oldenburg,
Postfach : 2503, D-2900 Oldenburg, FRG.

I would also recommend the following :

1. Dr. John Q. Adams,
c/o Dr. T.R. Hughes,
4 Keith, Orinda, California, 94563, USA.

2. Dr. Hermann Beyer
Budapest II, Martirok utja 8-10 Hungary

JOB DESCRIPTION

DP/IND/87/007

Post title : Experts in Zeolite Synthesis

Duration : 3 to 4 weeks

Date required : Several zeolite synthesis experts are needed. They should come in three consecutive seasons, between September and March, in 1988, 1989 and 1990.

Duty station : Pune (India).

Purpose of project :

: The long term objective of the project is to develop new shape selective zeolites and their application in the petroleum and organochemical industries. The immediate objectives are (a) to support NCL research schemes for the development of synthesis, and characterisation of new shape selective zeolites and catalysts, (b) demonstrate the preparation of suitable catalysts for refinery and petrochemical industries, (c) establish a competent research and development team of scientists and engineers at NCL capable of synthesising, characterizing and developing new shape zeolite catalysts and of developing their applications.

Duties : The expert will be assigned to the National Chemical Laboratory, Pune, and in consultation with appropriate Government authorities UNDP and UNIDO, he will be expected to carry out the following tasks :

- Review NCL's zeolite synthesis program & suggest improvements.
- Advise on establishing short and long-term research objectives - Indicate priorities.
- Give directions for laboratory work on zeolite synthesis.
- Review the equipment available and equipment proposed for further work.

Reporting : The expert will submit a report on his findings and recommendations.

Qualifications : High level scientist or engineer with extensive experience in zeolite synthesis.

Language : English

Background information :

The Indian Government is keen to use the facilities of the National Chemical Laboratory (NCL) to develop SHAPE SELECTIVE CATALYSTS, e.g. zeolites. These relatively new catalysts have already brought dramatic improvements in a number of refinery and petrochemical processes. The Indian National Chemical Laboratory has a proven record of invention, research and development in this area.

The Project should help India to develop new SHAPE SELECTIVE CATALYSTS and those processes which will use these catalysts. Its main and foremost task is to evolve and demonstrate a procedure for the production of highly efficient zeolite catalysts. It is expected that many more novel applications will be discovered for these catalysts and they will facilitate many chemical reactions which presently cannot be practiced on an economic scale.

SHAPE SELECTIVE CATALYSTS are used for the production of gasoline from methanol, production of olefins, isomerization of aromatics, alkylation of olefins, alkylation of benzene and toluene and dewaxing of middle distillates and lube stocks.

NCL has been engaged in the synthesis, characterization and development of applications in the area of shape selective zeolite catalysts for the past 6 years. More than 60 publications in research journals, 25 patents and 15 presentations in international research conferences have been achieved in this area during this period. NCL is a recognized institution where graduate work leading to Ph.D's in chemistry and chemical engineering are carried out. Currently, about 60 students work there for their Ph.Ds in these two fields.

NCL's most important accomplishments are two catalytic processes used commercially in India. These are xylene isomerization and ethylbenzene production. The former has better yield than competing processes. The latter uses a completely new and unique route based on dilute ethanol instead of ethylene. NCL invented the catalysts, worked out their large-scale synthesis and developed the processes.

Today, these NCL catalysts are manufactured under license from NCL by M/s Associated Cement Companies Limited, Bombay and M/s United Catalysts India Limited, Bombay. Naphta reforming, production of methyl amines, toluene disproportionation, etc. are expected to be commercialised in the next few years.

NCL has well established facilities for the collection of kinetic data on catalytic processes using different types of micro-reactor systems. NCL's Instrumentation Centre has facilities such as ECSA, X-ray, SEM, etc. An internationally renowned experts team in chemical reaction engineering supports reactor and modelling simulation with special emphasis on the critical problem of dynamics and stability. However, NCL's current, more sophisticated objectives need UNDP assistance. New experimental and analytical facilities are needed and technical and scientific expertise should be built up in certain critical fields. Without this help NCL's work will not have the required and expected impact on the industrial development of India.

JOB DESCRIPTION

DP/IND/87/007

Post title : Experts in Physicochemical Characterization of Zeolites and Catalysts. I : General

Duration : Three to Four weeks, with possibility of extension in subsequent year(s).

Date required :

About three such experts are needed.They should come in four consecutive seasons, between September and March in 1988, 1989, 1990 and 1991. The first one or two expert should come before the end of March 1988, therefore recruitment is urgent.

Duty station : Pune (India).

Purpose of Project :

The long term objective of the project is to develop new shape selective zeolites and their application in the petroleum and organochemical industries. The immediate objectives are (a) to support NCL research schemes for the development of synthesis, and characterization of new shape selective zeolites and catalysts, (b) demonstrate the preparation of suitable catalysts for refinery and petrochemical industries, (c) establish a competent research and development team of scientists and engineers at NCL capable of synthesising, characterizing and developing new shape zeolite catalysts and of developing their applications.

Duties :

The expert will be assigned to the National Chemical Laboratory, Pune, and in consultation with appropriate government authorities UNDP and UNIDO, he will be expected to carry out the following tasks :

- Review NCL's zeolite and catalyst characterization programme, and suggest improvements and new methods.
- Review the instruments available and the instruments proposed for further work.
- Help determine the structure of the new zeolite NCL discovered recently.
- Advise on establishing short and long-term research objectives - indicate priorities.

Reporting : The expert will submit a report on his findings and recommendations.

Qualifications : High level scientist with extensive experience in physicochemical characterization of zeolites and catalysts.

Language : English.

JOB DESCRIPTION
DP/IND/87/007

Post title : Experts in Physicochemical Characterization of Zeolites and Catalysts : II : Quantitative Powder X-Ray Diffraction.

Duration : Three to Four weeks, with possibility of extension in subsequent year(s).

Date required :
One expert is needed. He should come between September and March of 1989.

Duty station : Pune (India), or the laboratory of the Expert.*

Purpose of Project :

The long term objective of the project is to develop new shape selective zeolites and their application in the petroleum and organochemical industries. The immediate objectives are (a) to support NCL research schemes for the development of synthesis, and characterization of new shape selective zeolites and catalysts, (b) demonstrate the preparation of suitable catalysts for refinery and petrochemical industries, (c) establish a competent research and development team of scientists and engineers at NCL capable of synthesising, characterizing and developing new shape zeolite catalysts and of developing their applications.

Duties :
The expert will be assigned to the National Chemical Laboratory, Pune, and in consultation with appropriate government authorities UNDP and UNIDO, he will be expected to carry out the following tasks :

- Help determine the structure of the new zeolites NCL discovered recently.
- Review NCL's zeolite and catalyst programme, and suggest improvements and new methods.
- Review the instruments available and the instruments proposed for further work.

Reporting : The expert will submit a report on his findings and recommendations.

Qualifications : High level scientist with extensive experience in quantitative powder X-ray diffraction structure determination.

Language : English.

* An Expert in quantitative powder X-ray diffraction needs his own instrument and computer in his own laboratory to accomplish his task. He need not come to NCL in Pune.

JOB DESCRIPTION
DP/IND/87/007

Post title : Experts in Physicochemical Characterization of Zeolites and Catalysts : III : Transmission Electron Microscopy.

Duration : Three to Four weeks, with possibility of extension in subsequent year(s).

Date required :
One or two such experts are needed. They should come between September and March in 1989, 1990 and 1991.

Duty station : Pune (India).

Purpose of Project :

The long term objective of the project is to develop new shape selective zeolites and their application in the petroleum and organochemical industries. The immediate objectives are (a) to support NCL research schemes for the development of synthesis, and characterization of new shape selective zeolites and catalysts, (b) demonstrate the preparation of suitable catalysts for refinery and petrochemical industries, (c) establish a competent research and development team of scientists and engineers at NCL capable of synthesising, characterizing and developing new shape zeolite catalysts and of developing their applications.

Duties :
The expert will be assigned to the National Chemical Laboratory, Pune, and in consultation with appropriate government authorities UNDP and UNIDO, he will be expected to carry out the following tasks :

- Review NCL's zeolite and catalyst programme, and suggest improvements and new methods.
- Review the instruments available and the instruments proposed for further work. Suggest training programme in transmission electron microscopy for NCL staff.
- Help determine crystal size morphology and agglomerisation of NCL's new zeolites.
- Advise on establishing short and long-term research objectives - indicate priorities.

Reporting : The expert will submit a report on his findings and recommendations.

Qualifications : High level scientist with extensive experience in Transmission Electron Microscopy.

Language : English.

JOB DESCRIPTION
DP/IND/87/007

Post title : Experts in Physicochemical Characterization of Zeolites and Catalysts : IV - ESCA & EPR

Duration : Three to Four weeks, with possibility of extension in subsequent year(s).

Date required :
One or two such experts are needed. They should come between September and March in 1989, and 1990.

Duty station : Pune (India).

Purpose of Project :

The long term objective of the project is to develop new shape selective zeolites and their application in the petroleum and organochemical industries. The immediate objectives are (a) to support NCL research schemes for the development of synthesis, and characterization of new shape selective zeolites and catalysts, (b) demonstrate the preparation of suitable catalysts for refinery and petrochemical industries, (c) establish a competent research and development team of scientists and engineers at NCL capable of synthesising, characterizing and developing new shape zeolite catalysts and of developing their applications.

Duties :
The expert will be assigned to the National Chemical Laboratory, Pune, and in consultation with appropriate government authorities UNDP and UNIDO, he will be expected to carry out the following tasks :

- Review NCL's zeolite and catalyst programme, and suggest improvements and new methods.
- Review the instruments available and the instruments proposed for further work.
- Help in characterizing Fe in Fe-faujasite and Fe-pentasil zeolite not just qualitatively but also quanatitatively.
- Advise on establishing short and long-term research objectives - indicate priorities.

Reporting : The expert will submit a report on his findings and recommendations.

Qualifications : High level scientist with extensive experience in EPR and ESCA techniques (Recommended Dr. J. Q. Adams, see list.

Language : English.

JOB DESCRIPTION
DP/IND/87/007

Post title : Experts in Catalytic Reactions, Chemistry and Engineering.

Duration : Three to Four weeks, with possibility of extension in subsequent year(s).

Date required :
About five such experts are needed. They should come in three consecutive seasons, between September and March in 1989, 1990 and 1991.

Duty station : Pune (India).

Purpose of Project :

The long term objective of the project is to develop new shape selective zeolites and their application in the petroleum and organochemical industries. The immediate objectives are (a) to support NCL research schemes for the development of synthesis, and characterization of new shape selective zeolites and catalysts, (b) demonstrate the preparation of suitable catalysts for refinery and petrochemical industries, (c) establish a competent research and development team of scientists and engineers at NCL capable of synthesising, characterizing and developing new shape zeolite catalysts and of developing their applications.

Duties :
The expert will be assigned to the National Chemical Laboratory, Pune, and in consultation with appropriate government authorities UNDP and UNIDO, he will be expected to carry out the following tasks :

- Review NCL's zeolite and catalyst programme, and suggest improvements and new methods.
- Review the test reactors available
- Advise on potential applications of NCL's new zeolites. Select the most apropriate test reactor. Help designing the appropriate differential and/or integral test reactor.
- Advise on establishing short and long-term research objectives - indicate priorities.
- In later stages of the project help in scaling up the reaction.

Reporting : The expert will submit a report on his findings and recommendations.

Qualifications : High level chemical enginer as chemist with extensive experience in catalysis over zeolites and reaction engineering.

Language : English.

JOB DESCRIPTION
DP/IND/87/007

Post title : Experts in Chemical Process Technology.

Duration : Three to Four weeks.

Date required :

About three such experts are needed. They should come between September and March of 1990 and 1991.

Duty station : Pune (India).

Purpose of Project :

The long term objective of the project is to develop new shape selective zeolites and their application in the petroleum and organochemical industries. The immediate objectives are (a) to support NCL research schemes for the development of synthesis, and characterization of new shape selective zeolites and catalysts, (b) demonstrate the preparation of suitable catalysts for refinery and petrochemical industries, (c) establish a competent research and development team of scientists and engineers at NCL capable of synthesising, characterizing and developing new shape zeolite catalysts and of developing their applications.

Duties :
The expert will be assigned to the National Chemical Laboratory, Pune, and in consultation with appropriate government authorities UNDP and UNIDO, he will be expected to carry out the following tasks :

- Advice on engineering problems associated with commercialisation
- Help in scaling up the process from pilot plant to commercial scale.
- Advice on establishing short and long-term research objectives - Indicate priorities.
- Give directions for laboratory and pilot plant work.
- Review the equipment available.

Reporting : The expert will submit a report on his findings and recommendations.

Qualifications : High level engineer with extensive experience in chemical process technology.

Language : English.

Recommendation : Dr. S. Khoobiar, the UNDP Expert of this project : 750 Ridge Rd., Kinnelon, N.J.07405, USA.

JOB DESCRIPTION
DP/IND/87/007

Post title : Experts in Process Control.

Duration : Three to Four weeks.

Date required :

Two or three such experts are needed. They should come between September and March of 1990 and 1991.

Duty station : Pune (India).

Purpose of Project :

The long term objective of the project is to develop new shape selective zeolites and their application in the petroleum and organochemical industries. The immediate objectives are (a) to support NCL research schemes for the development of synthesis, and characterization of new shape selective zeolites and catalysts, (b) demonstrate the preparation of suitable catalysts for refinery and petrochemical industries, (c) establish a competent research and development team of scientists and engineers at NCL capable of synthesising, characterizing and developing new shape zeolite catalysts and of developing their applications.

Duties :

The expert will be assigned to the National Chemical Laboratory, Pune, and in consultation with appropriate government authorities UNDP and UNIDO, he will be expected to carry out the following tasks :

- Advice on engineering problems.
- Help in scaling up the process from pilot plant to commercial scale.
- Advice on establishing short and long-term research objectives - Indicate priorities.
- Give directions for laboratory and pilot plant work.
- Review the equipment available.

Reporting : The expert will submit a report on his findings and recommendations.

Qualifications : High level chemical engineer with extensive experience in process control

Language : English.

Recommendation : Professor Ken. Bishop
University of Delaware
Dept. of Chem. Engg.
Newark, Delaware 19716.

CATALYSIS RESEARCH AT NCL. THE PLACE AND ROLE OF THE UNDP PROJECT

1. BACKGROUND:

Research in the area of catalysis and catalytic reaction engineering has been carried out at the NCL during the past 30 years. Zeolite chemistry and catalysis by zeolites are major areas of activity since 1980. Todate, NCL's achievements in the area of zeolites include more than 80 publications in the international journals, around 30 Indian patents and commercialisation of two major petrochemical catalysts/processes (xylene isomerisation at IPCL, Baroda and Production of ethylbenzene at HP, Vizag) with a few more on the anvil.

2. UNDP PROJECT

The UNDP funded project titled **DEVELOPMENT OF NOVEL SHAPE SELECTIVE CATALYSTS** was started in July 1987 with the objectives of augumenting the existing expertise and facilities in the area of zeolites to enable the development of novel shape selective catalysts. The project is **NOT** oriented towards the development of any particular process or application of zeolite catalysts. Rather, the emphasis is on the development of an integrated capability and expertise in the area of zeolite catalysis.

3. MIDDLE DISTILLATES

An earlier proposal was submitted (about 3 years back) by NCL for UNDP funding for a project for the conversion of natural gas to middle distillates. The proposal was not accepted by UNDP which suggested a revision to confine the project to basic studies on zeolites with emphasis on developing novel zeolites. Accordingly, the present proposal (which was later approved) was submitted by NCL. Under the present project, UNDP funds are to be used only for basic studies on zeolites. In the meantime, work on the middle distillates was continued at NCL, using NCL's funds. At present, NCL, Davy McKee and Bharat Petroleum Corpn Ltd. have entered into an

agreement for setting up a 1 ton/day pilot plant for producing middle distillates. BPCL is funding the project The PP is expected to be ready in 2 years.

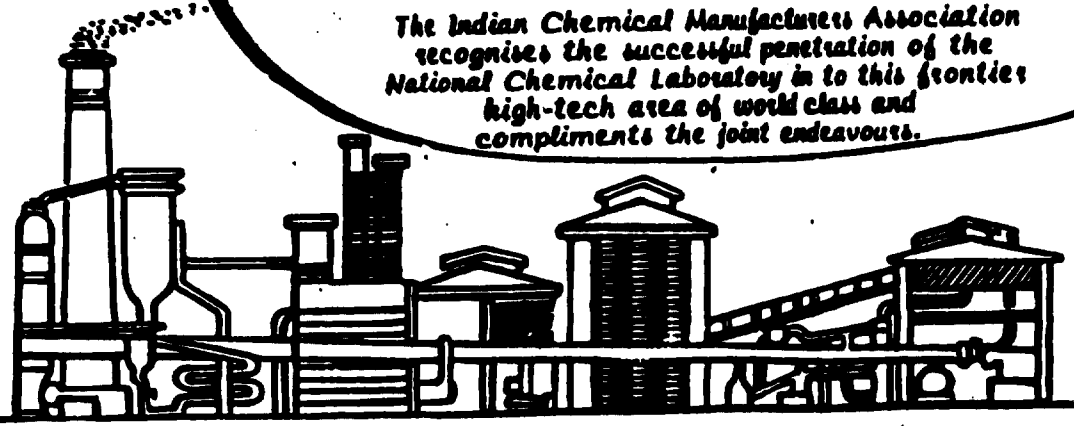
1985

THE I.C.M.A. AWARD

SPECIAL CITATION
to
NATIONAL CHEMICAL LABORATORY, PUNE

The National Chemical Laboratory, Pune, have secured an astounding success in inventing a highly original catalyst for isomerisation of xylene, containing ethyl benzene. This development calls for very high level scientific inputs from diverse areas. They have successfully interacted with The Associated Cement Companies Limited and the Indian Petrochemicals Corporation Limited in transferring this complex technology in a record period.

The Indian Chemical Manufacturers Association recognises the successful penetration of the National Chemical Laboratory in to this frontier high-tech area of world class and compliments the joint endeavours.



(K R V SUBRAHMANIAN)
President, I.C.M.A.

INDIAN CHEMICAL MANUFACTURERS ASSOCIATION

PRIME MINISTER

New Delhi
February 12, 1987

Dear Dr. Doraiswamy,

I have seen your brief report on the research activities of the National Chemical Laboratory. Although the scientists at NCL have made several contributions, the ones related to encilite, xylanase enzyme and tissue cultures are particularly important. The development of encilite and of the corresponding xylofining process will stand as an excellent example of collaborative work carried out by the Indian scientists and the industry.

Please do convey my sincere appreciation to the concerned scientists for their outstanding contributions.

Yours sincerely,

Dr. L.K. Doraiswamy,
Director,
National Chemical Laboratory,
Pune - 411 008

REVISED SPECIFICATIONS FOR ITEM 19 OF PROJECT DOCUMENT

Item No.	Item name	Specifications	Address of firm
19.	High pressure accessory for ESCA.	To operate at high pressure and temperature	M/s V.G. Scientific Ltd The Birches Industrial Estate Imberhorne Lane East Grinstead West Sussex RH 19 IUB U.K