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PROCESS ENGINEERING (PETROCHEMICALS AND FERTILIZERS)

DP/IND/71/047

INDIA

TURMINAL REPORT

Proposed for the Government of India by the United Nations Industrial Development Organization executing agency for the United Nations Development Programme



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United Nations Development Programme

PROCESS INGINERING

(PETROCHEMICALS AND FERTILIZERS)

DP/IND/71/047

INDIA

Project findings and recommendations

Prepared for the Government of India by the United Nations Industrial Development Organisation, executing agency for the United Nations Development Programme

Inced on the work of D. H. J. Prochligh. emert in process design

United Nations Industrial Development Organisation Vienna, 1975

Explanatory notes

A slash (/) between dates representing years indicates a crop year or financial year, e.g. 1971/72.

Reference to "tons" indicates metric tons, unless otherwise stated.

Reference to "dollars" (\$) indicates United States dollars, unless otherwise stated.

The following abbreviations are used in this report:

EIL Engineers India Limited

R.H. relative humidity

cP centipoise

cSt centistokes

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SUNMARY

A short background of the project "Process Engineering (Petrochemicals and Fertilizers)" (DP/IND/71/047) given in the introduction explains the changes in the situation for the production of synthetic fibres owing to the oil crisis. The introduction also contains lists of the main producers of synthetics in India and the companies to which letters of intent have been issued.

The work of Engineers India Limited (EIL), the main engineering company in India, is described. The steps taken by EIL in the direction of basic engineering, especially for Nylon 6, during the last 12 months are reviewed and recommendations are made for continuing this work and for involving EIL in a pilotplant project for SASMIRA in Bombay. The annexes indicate the scope of the engineering work to be done for a nylon plant.

INTRODUCTION

The purpose of the project "Process Engineering (Petrochemicals and Fertilizers)" (DP/IND/71/04/) was to strengthen the process-engineering manpower of India and to help in the designing of planned petrochemical, petroleum refining, synthetic fibre and fertilizer plants. The project was requested by the Government of India in July 1971 and approved by the United Nations Development Programme (UNDP) with the United Nations Industrial Development Organization (UNIDO) as executing agency. The project received a budget allocation of \$80,000.

The project experts were to assist the Engineers India Limited (EIL), a Government of India undertaking under the Ministry of Petroleum and Chemicals, in training Indian engineers in process engineering. At the time the project was approved, EIL was expected to be involved in engineering work for more than 8 synthetic fibre plants of the planned 13 projects for which letters of intent had been issued. Most of the projects were intended for the production of polyamides (Nylon 6). However, there was a sharp and unanticipated increase in oil prices in 1973/74. As a result, the Government of India did not license any new synthetic fibre plants because of the tremendous shortage in both raw materials and foreign exchange. Accordingly, the project was revised and the total UNDP contribution was reduced to \$33,500 in July 1975.

The situation varies for the three main areas of production of man-made fibres - polyamides, polyesters and acrylics. Polyamides are produced by seven established factories with a total licensed capacity of 25,000 tons/year for 1972, although the production in that year was only 11,000 tons. A production target for 1978/79 of 32,000 tons/year is set to meet the projected requirements.

The main producers and yearly production of nylon filament yarn are:

Producer	Thousand tons
J. K. Synthetics Ltd, Kota	3.84
Garware Nylon Limited	2
Nirlon Synthetics, Fibres and Chemicals Ltd (Bombay)	252
Modipon Limited (Modinagar)	2.2
Century Enka Ltd (Poona)	0.72
Shree Synthetics Ltd (Ujjain)	1.10
Stretch Fibres Ltd (Nagpur)	0.54

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Letters of intent for a production of 2,100 tons/year each have been issued to:

Industrial Development Corporation Orissa Ltd (Bhubaneshwar, Orissa) Assam Industrial Development Corporation Limited (Gauhati, Assam) M.P. Audigik Vikas Nigam Ltd (Bhopal, M.P.)

Haryana State Industrial Development Corporation Ltd (Chandigarh, Haryana) Mysore State Industrial Investment and Development Corporation Ltd (Bangalore, Mysore)

Gujarat Industrial Investment Corporation Limited (Baroda, Gujarat) Tamil Nadu Industrial Development Corporation Ltd (Tamil Nadu) Andhra Pradesh Industrial Development Corporation Ltd (Hyderabad, A.P.) Kerala State Industrial Development Corporation Ltd (Trivandrum, Kerala) Bihar State Industrial Development Corporation Ltd (Patna, Bihar) Punjab State Industrial Development Corporation Ltd (Chandigarh Punjab) West Bengal State Industrial Development Corporation Ltd (Calcutta) U. P. State Industrial Development Corporation Ltd (Kanpur)

The only producer of nylon staple fibre is J. K. Synthetics Limited (Kota, Rajasthan) with a capacity of four tons/day.

It is easy for a producing company to increase its capacity simply by copying the existing machinery, whereas a new company needs a licence either bought abroad for foreign currency or from an indigenous licensor and engineering company. To bridge this gap, EIL was selected by the Government of India to establish a synthetic-fibre engineering group. EIL, a design and consultancy organization, was established on 15 March 1965 as a joint venture of the Government of India and Bechtel Corporation of the United States of America to provide process design and detailed engineering services, procurement assistance, construction supervision, commissioning assistance and related services for setting up petroleum refineries, petrochemical and chemical plants and fertilizer plants. In 1967 the Government of India bought out the shares of Bechtel so that EIL is now fully government-owned.

EIL has a total of 1,900 employees of whom approximately 500 are engineers; about 12 engineers are currently working in the synthetic fibre section.

I. FINDINGS

The main activities of the project were to strengthen process-engineering know-how in the design of synthetic fibre plants and to train counterpart personnel in process design of synthetic fibre plants. To achieve these objectives, work covered the typical tasks and fields of engineering, e.g. preparation of basic data book, process flow sheets for several types of plants, process description for the several sections, process diagrams, and calculation of the process such as material balances and heat balances.

In addition, consideration was given to other activities that arise during the planning period, such as preparation of a general bid inquiry check-list and factors related to the selection of licensors.

As the complete engineering work for a Nylon-6 plant contains many more items than could be included in the training course (see annex I), areas of particular importance were identified and treated more intensively. Roughly 80 to 90 per cent of the total engineering work can be done by EIL directly without any outside help; it has an especially strong capability for detailed engineering if it is supported by the basic engineering work of a well-established company.

II. RECONDENDATIONS

To meet the anticipated future demand in synthetic fibres, appropriate steps should be taken as early as possible to further strengthen EIL capability in this field. More man-hours should be devoted to some of the synthetic fibre projects which although not yet fixed may still be considered likely to be realized in future years.

Man-hours should be spent in preparing a pilot-plant project for polyester and nylon, including the preparation of test rows which could be run in the pilot plant and the process description of all the various conditions that might materialize in order to measure the influence of several parameters.

To strengthen process-engineering know-how in the design of synthetic fibre plants it would be useful to involve EIL's engineering capacity during the planning period for the UNDP/Federal Republic of Germany pilot plant for SASNIRA. Training courses abroad and in India in connexion with that project should be implemented, if possible within the resources available for the project.

Annex I

SCOPE OF ENGINEERING WORK FOR A NYLON-6 PLANT

A. General engineering

- 1. Preliminary specifications of equipment and services for erection and operation of the plant.
- 2. Schedules, giving dates for customer's and supplier's deliveries of equipment and services.
- 3. Supervision of erection and start-up of the plant by supplier's personnel (after the conclusion of a separate contract for erection and start-up).
- 4. Erection programme, including details on erection personnel, tools and erection materials.
- 5. Plan for the start-up of the plant.
- 6. Details on personnel required for the operation of the plant.
- 7. Details on type and quantity of spare parts to be kept continuously on stock.
- 8. General instructions for installation of equipment and machinery, for piping and insulation of plant parts as well as for all tightness checks.
- 9. If required, recommendations for plant organization with hints for keeping an operation diary.

B. Process engineering

- 1. General description of the plant with explanation of the process, limits of capacity and subdivision into different plant stages.
- 2. Process flowsheet for production plant.
- 3. Diagram showing quantities of raw material input and product output, with by-products and losses.
- 4. Raw and auxiliary material requirements specifications for the operation of the plant.
- 5. Detailed process flowsheet with indication of all measuring and control points and schematic piping system for product utilities as well as raw and auxiliary materials.
- 6. Process schedules for plants or plant sections operated batchwise.
- 7. Detailed description of the process.
- 8. Insofar as required, detailed spin and twist tables as well as flowsheet giving quantities of intermediate and final products for the individual textile processing stages.
- 9. Details of transportation and transportation means within the plant, with transportation schedule.
- 10. Plant parts lists for the complete scope of delivery.
- 11. Instructions for start-up, shut-down, operation with reduced capacity, maintenance and periodical overhauls, as well as for steps to be taken in case of plant failure, including specification of operation data.

- 12. Methods and instructions for quality control of raw materials as well as intermediate and final products.
- 13. Requirement and specification of power and utilities required for the operation of the plant.
- 14. Peak values of power and utility supply system.
- 15. Recommendations for provision of emergency power supply.
- 16. Information on inlet and outlet of power and utilities in the different production stages.
- 17. Details and specification of effluents.
- 18. Recommendations for packaging and storage of final product as well as for required auxiliary means.
- 19. Safety instructions based on local safety regulations.

C. Equipment layout

- 1. Preliminary sketches of items of equipment and/or data sheets with leading discussions, materials of construction, operating pressure and temperature, fare, weight, normal operating weight and overload weight.
- 2. Preliminary equipment layout within the preliminary building plans.
- 3. Final installation drawings, including all necessary ground plans and elevations.
- 4. Detail drawings for framing and supports for equipment items.
- 5. Details of openings required for the provision of flooring grids for the individual steel stagings.
- 6. Documentation drawings for the equipment of the suppliers delivery to facilitate spare parts identification and overhauls.
- 7. Individual operating instructions for equipment and machinery to be delivered, including characteristic curves, descriptions of functions, special instructions for assembly, operation, maintenance and lubrication, sectional views for spare parts lists.

Annex II

BASIC DESIGN DATA FOR NYLON-6 FILAMENT YARN PLANT

A. Unit capacity

1. The plant capacity is considered as 6 tons of Nylon-6 filament yarn per day.

Based on an onstream period of 8,000 hours per annum, the plant capacity would be 2,000 tons per annum.

2. The production programme would be according to the following product mix:

Monofilament:

300 tons per annum of 15 denier

700 tons per annum of 20 denier

Multifilament:

600 tons per annum of 40 denier

400 tons per annum of 76 denier

3. Monomer recovery is considered both from solid waste as well as extraction water. The capacity of monomer recovery section is based on 300 tons per annum of recovered caprolactam.

B. Yields

- 1. Design of the plant is based on the production of semi-dull chips and yarn.
- 2. The polymerization reactor outlet stream shall have the following composition based on the conversion of monomer to polymer:

Polymer	87.5 🖇
Monomer	12.0 %
Other components,	
e.g. Ti0 ₂ , H ₂ 0, etc.	0.5 %

- 3. Production losses:
 - (a) Solid waste:

Solid waste from a	pinning	2 🛸
Solid waste from t	ake-up	1 🕺
Solid waste from d		1 % 3 %
Monomer recovery f	from solid waste	60 🛸

(b) Extraction water:

Monomer concentration in extraction water 6.5 \$ 80.0 \$ Monomer recovery from extraction water

Annex III

SPECIFICATIONS OF RAW AND AUXILIARY NATERIALS AND PRODUCTS

A. Raw and auxiliary materials

1. Caprolactam

Specification	Unit	
Molscular formula		C6H ₁₁ NO
Nolecular weight		113.16
Purity	۶.	99.8 (Min.)
Bulk density	kg/litre	0.6 to 0.7
Nelting point	°C	68.8 (Min.)
Appearance		Solid: white crystalline Nolten: clear liquid
Colour of aqueous solution	АРНА	10 (Nax.)
Permanganate number ^b	Second	5000 (Min.)
Volatile bases	ppm	8 (Max.)
Free bases	om ³ /kg	5 (Nax.)
Water content	۶	0.1 (Naz.)
Iron content	ppm	1 (Naz.)
Water insolubles	ppm	10 (Max.)
Filtrate ignition residue	ppm	20 (Max.)

Visual comparison of 40 % solution, calculated on a 100 % basis. 3 g of caprolactam in 100 ml water with 1 ml N/10 KDmO₄.

2. Glacial acetic acid

Specification	Unit	
Nolecular formula		СН3СООН
Nolecular weight		60
Appearance		Colourless, suspension free liquid
Purity	\$	99.5 (Min.)
Concentration	\$	99
Specific gravity		1.055 to 1.058
Nelting point	°c	16.7 (Min.)
Iron as Fe	pya	1 (Nax.)
Sulphate as SO4	ppm	2 (Max.)
Chloride as Cl	ppm	1 (Max.)

3. <u>Titanium dioxide</u>

Specification	Unit	
Molecular formula	Ti0 ₂	
Molecular weight	80	
Туре	Anatase grade	
TiO ₂ content	\$ 95 (Nin.)	
Specific gravity	3.8	
Refractive index	2.5	
Brightness	good	

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4. <u>Diphyl</u>

Specification	Unit	
Content of diphenyl	×,	26.5
Content of diphenyl oxide	*	73.5
Colour of the solid product		Yellowish-white
Colour of the liquid		Colourless to yellowish (clear)
Application in permanent operation	°C	Up to 400
Boiling point at 1 atm	°C	256-258
Boiling range	°c	1.0
Solidification point	°C	12.3
Water content at 20°C	wt %	0.02
Density of the liquid at 20 ⁰ C	kg/m ³	1062

3. Preducts

1. Brion-6 chine

Specification	Unit	
Appearance		Celourless
Dimension		3mm dia.x 3mm (aylindrical)
Solidification range	°c	210 to 217
Bulk density at 20°C	kg/m ³	680
Nater content	\$	0.05 (Nex.)
Titanium dioxide content	week	0 to 3 according to application
Dust content	\$	0.02 (Nms.)
Hot water extractables	\$	0.50 (Nmx.)
Noisture pickup at 65 \$ R.H.	\$	4.5
Number of black and/or colewred spots per 10g of chips		2 (Has.)

2. Brion-6 filement rare

Specification	Unit	Nono filanent	Multifilement
Titre	denier	15/1, 20/1	40/10, 76/30
Denier variation	CVE	3.0	2.5
Uster Unevenness	U	1.5	1.5
Tenacity at break	e/ten	5.0	4.5
Elongation at break	\$	35 ± 5	35 ± 5
Boiling chrinkage	\$	15 10 17	15 10 17
Betract content	\$	2.0	2.0
Pull peckage weight	C	1890	1890
Pall pirm yield	\$	80	80
Piret quality yarm	\$	85	85

The first quality yarm is defined as full pirms without break, mosting the above specifications.

Annex IV

UTILITY SPECIFICATIONS

1. Steen. N.P. steam (saturated), kg/cm²g 12.5 + 10 \$ 2. Nater (a) <u>Cooling water</u> Type of system: Recirculating with raw water makeup Battery limit pressure at grade-level: Supply, kg/cm²g 5.0 Return, kg/cm²g 2.5 Temperatures Supply, ^oC Return, ^oC 32 42 Total alkalinity, ppm as CaCO3 100 to 120 300 (Max.) Calcium hardness, ppm as CaCO₂ Total hardness, ppm as CaCO₁ 700 (Max.) Chloride content, ppm as NaCl 750 (Max.) Silica content, ppm as SiO₂ 125 (Max.) Total dissolved solids, ppm 1500 (Nax.) Suspended solids, ppm 60 to 90 pH value 7.0 to 7.5 (b) **Demineralized water** Battery limit pressure at grade-level, ke/cm2"e 4.0 Temperature Ambient Total hardness, ppm as CaCO₃ 0.25 (Naz.) Total dissolved solids, ppm 3 (Max.) Silica content, ppm as SiO₂ 0.20 (Max.) Chloride content, ppm as Cl 3 (Max.) Iron content, ppm 0.05 (Nax.) ъH 7.0 to 7.5 Specific resistance at 25°C, ohm-om 500,000 (c) Chilled water Type of system: recirculating Battery limit pressure at grade-levels Supply, kg/om_g 4.0 Return, kg/om^cg 2.0 Temperatures Supply, ^oc 10 Return, °C 16

	Total dissolved solids, ppm	10 (Max.)
	Total hardness, ppm as CaCO3	0.25 (Haz.)
	Chloride content, ppm as Cl	3
	Silica content, ppm as SiO ₂	0.6
	Turbidity	0
	рН	7 to 7.5
(4)	Service water	

Battery limit pressure at grade-level, kg/om⁷g 4.0 Temperature Ambient Calcium hardness, ppm as CaCO3 120 160 Nagnesium hardness, ppm as CaCO₃ 280 Total hardness, ppm as CaOO3 400 Alkalinity, ppm as CaCO3 Total dissolved solids, ppm 750 Silica content, ppm as SiO₂ 50 300 Chloride content, ppm as NaCl pH value 7 to 8

(•) <u>Plant air and instrument air</u>

Battery limit pressure, kg/om ² g	7
Dew point, ^o C	-20 (Nag.)
Oil content	Oil free
Suspended particles	None; dust free

10

-20 (Nax.)

10,000

80 (Nax.)

66 (Min.)

3.5 (Naz.) 1.0 (Naz.) 0.1 (Naz.)

(f) <u>Inert me (Mitrogen)</u> Pressure, kg/om²g

Oxygen content:

for polymerisation section, ppm 10 (Haz.) for spinning section, ppm 3 (Haz.) Hydrogen content Traces

Dew point, ^oC

(e) <u>Puel oil</u>

Calorific value, koal/kg Kinematic viscosity at 50°C, off Flash point (Pensky-Martin closed),°C Bulphur content, wt.\$

Water content, wt.S Ash, wt.S

(h) Perer

Voltage

Prequency, Hs

420/2201 10 \$ 50 ± 3 \$

PHYSICAL PROPERTIES OF RAW AND AUXILIARY MATERIALS AND PRODUCTS

A. Caprolactam

Physical property	Unit	At		Refer- ence
Molecular formula			8	
^C 6 ^H 11 ^{NO}			H ₂ C CH ₂	
(c5 ^H 10 ^{CONH})			H ² C CH2	
Nolecular weight			H 2 113.16	
Appearance (salt)			Solid: white crystalline Molten: clear liquid (<10 APHA)	1 1
Density of solid lactam	g/om ³	20 [°] C	1.124	2
Bulk density	g/om ³	20° C	0.5 to 0.7	2
Density of molten lactam, P _L	g/om ³		$\rho_L = 1.075 - 7.7 \times 10^{-4}$, (where 0 is temperature in °C	2)
		80°c 77°c	1.013 1.03	2
Melting point, A	°c		69.2 for pure lactam	2
6 2			$\theta_{m} = 69.2 - 2.94 \text{ cw}$ (where cw is water content in %	2
Solidification point,	θ <mark>.</mark> °C		$\theta_{\rm g} = 69.2 - 3.57$ cw (where cw is water content in $\%$	2
Boiling point	°c	1 atm	268.5	2
		100 mm	200.0	3
Flash point	°c		125	1

Physical property	Unit	At		Refer- ence
Ignition point	°c		139	2
Vapour pressure, P	mnlig (abs)		$\log P = 8.90 - \frac{3.200}{T}$ (where T is temperature in °K)	2
	mnHg (abs)	20 [°] C	<0 .1	
Heat of vaporization, ΔH _v	cal/g		AH _v = 172.6 - 0.215 0 (where 0 is temperature in C)	2
		268.5°C	115	2
Heat of solidification	cal/g		30 to 35	2
Heat of fusion	cal/g		29	1
Heat of combustion	cal/g		7,616 ± 20	2
Heat of polymeri- sation	oal/g		33.6	2
Heat capacity	oal/g°C		0.3299	1
		35°C	0.3394	1
		70 ° C	0.5050	1
Viscosity of molten laotam, n L	oP		$\log n = \frac{1.282}{T} - 2.70$ (where T is temperature in ^o K)	2
		160°C	1.8	2
		80°C	8.7	2
.		78°c	9.0	1
Refractive index, n D		40 [°] с 30 [°] с	n D	1
			n _D = 1.4965	ĩ
	g/100 g polution	0°c	72	2
		20°C	82	2
		25°C	84	3
		40°C	90	2

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Physical property	Unit	At	· · · · ·	Refer- ence
Solubility in	g/100 g	0 ° C	25	2
bensene	solution	20°C	43	2
		40 [°] C	60.5	2
		60⁰ С	78	2
Density of solu- tion in water, ^p w	g/om ³	20⁰0	P _W = 0.99 + 8.8 x 10 ⁻⁴ C (where C is concentration of lactam)	2
Refractive index of solution in water, ⁿ D		25 ° C	$n_{\rm D}^{25}$ = 1.333 + 1.66 x 10 ⁻³ C (where C is concentration of lactam)	2
Viscosity of water	oP	20⁹ C	1.43 (10%)	2
			2.87 (30 %)	2
			4.16 (40%)	2

Sources:

1. Marl D. Oliver, "Mylon-6", <u>Report No. 41</u> (Palo Alto, Process Economics Program, Stanford Research Institute), p. 158, 159, 160 and 163.

2. Kunststoff-Handbuch, Band VI (Polyamide), p. 677.

3. <u>Chemicals and Plastics Physical Properties</u>, CNC Mition, Catalogue No. U-10 of Union Carbide.

B. <u>Hylon-6 chine</u>

Physical property	Unit	At		Refer- ence
Nolecular formula			$H - H - (CH_2)_5 - C - OH$	
Nolecular weight			10,000 to 20,000	
Density	kg/m ³	20 ⁹ 0	1,130	
Bulk density	kg/m ³	20⁹ C	68 0	
Nelting point	°c		215	

Physical property	Unit	At		Refer- ence
Softening point	°c		170	
Melt viscosity	Ρ	250 ⁰ C	2,200	
Relative solution viscosity $\underline{a}/, n_{rel}$			2.2 - 2.6	1
Heat capacity	cal/g	° c	0.485 + 3.36 x 10^{-4} T (where T is temperature in °C	1)
		0 ° C	0.33	
		100 [°] C	0.472	
		150 ⁰ C	0 .51 0	
		250 [°] C	0 .58 0	
Heat of fusion	cal/g	215°C	34.5	1
Heat of vaporisation	cal/g		116	

Source: Earl D. Oliver, "Nylon-6", <u>Report No. 41</u> (Palo Alto, Process Economics Program, Stanford Research Institute), p. 157 and 161.

 \underline{a} / The relative solution viscosity is determined as solution in sulphuric acid and is related to the average relevalar weight by the equations

 $Mr = 11,300 (n_{rel}-1)$

C. Diphyl

Diphyl is an eutectic mixture of 73.5% by weight of diphenyloxide and 26.5% by weight of diphenyl. It has virtually one single boiling point and one single solidification point. Other properties of diphyl are listed below.

Physical property	Unit	At .
Chemical formula of diphenyl		° 6^H5- °6 ^H 5
Chemical formula of diphonyl oxide		°6 ^N 5 ^{-O-C} 6N5
Nolecular weight of d	iphonyl	154

Physical property	Unit	At	
Nolecular weight of diphenyl oxide			170
Mean molecular weight of diphyl			1 65 . 76
Colour of solid product			Yellowish-white
Colour of liquid			Colourless to yellowish (clear)
Odour			Geranium
Miscibility with water			Practically nil
Application in permanent operation		°c	Up to 400
Boiling point	°c	1 atm	256 to 25 8
Boiling range	°c		1.0
Solidification point	• _C		12.3
Water content	wt%	20° c	0.02
Flash point	°c		115
Fire point	°c		138
Ignition temperature in air	°c		615
Ignition temperature in oxygen	°c		550
Ignition group			a ₁
Lower explosion limit of vapour in	n air vol\$		101
Upper explosion limit of vapour is			3.47
Dens by of liquid	kg/m ³	20° C	1,062
Density of vapour	kg/m ³	1 atm	3.8
Vapour pressure	kg/om ²	40 0°c	11.3
Heat of vaporisation	koal /kg	1 atm	69
Specific heat of liquid	kcal/kg °C	20 [°] C	0 .38
Thermal conductivity of liquid	koal/m h ^o C	20 [°] C	0.12
Dynamic viscosity of liquid	oP	20 [°] C	4.34
Kinematio viscosity of liquid	cSt	20 [°] C	4.04
Dielectric resistance		20 [°] C	3.30
Electric resistance	o han-on	20 [°] C	1.35 x 10 ¹¹
Groes calorific value	koal /kg		8 ,9 70
Net calorific value	koal /kg		8,650

Annex VI

PROCESS DESCRIPTION

A. Monomer melting

Flaked lactam is charged into a melting tank through the feeding device fitted with a built-in lump crusher to break any lumps of lactam if formed during storage. A fan in the feeding device removes any dust formed. The melting tank is jacketed for hot water heating and provided with an inner steam heating coil. Lactam is melted in an inert atmosphere of nitrogen to prevent oxidation.

Molten lactam is pumped into the mixing tank through a basked type filter to remove any foreign matter. Catalyst and stabilizer are charged to the mixing tank where the agitator provides for intimate mixing. The stabilized caprolactam is discharged to the rundown tank through a filter. As the lactam has to be kept under a nitrogen blanket, the lactam melter, mixing tank and rundown tank are all connected to a compensation vessel, maintained under a specified nitrogen pressure to compensate for level changes in the tanks. The run down tank serves as a surge vessel for subsequent continuous feed to the V.K. tube reactor.

All the tanks and lines handling molten lactam are jacketed for hot water heating. The hot water circulation system consists of an expansion ver el, circulation pumps and heat exchanger for steam heating of the hot water. Return water from jackets of tanks and lines is pumped through the heat exchanger to control temperature of the circulating water which goes to the jackets of the tanks and lines.

B. Delustrant preparation

Titanium dioxide, used to control lustre in the yarn is prepared in a separate system. It consists of a TiO_2 preparation tank and a colloid mill for microgrinding of TiO_2 . TiO_2 suspension is prepared with distilled water and some quantity of lactam and chemicals to impart stability to the emulsion. After suspension has achieved the desired particle size of TiO_2 , it is transferred to TiO_2 surge tank through mixing and settling tank which removes oversized TiO_2 particles. The surge tank is kept under agitation. Predetermined quantities of the stabilized lactam and the TiO_2 suspension are pumped to the mixing chamber of the V.K. tube reactor by two component metering pumps from where it is continuously fed to the V.K. tube polymerization reactor.

C. Polymerization

The process for polymerization is a continuous one. The polymerization takes place under atmospheric pressure. The V.K. tube is a jacketed stainless steel column divided into four parts and connected by flanges. The upper two sections are heated by diphyl in the jackets by electric immersion heaters located directly at the reactor jacket. The lower parts of the V.K. tube reactor are heated by diphyl circulation system consisting of diphyl pumps and heaters. All the diphyl jackets are connected to the expansion vessels which are blanketed with nitrogen.

A small distillation column and a reflux condenser are provided at the top of the V.K. tube reactor. Water vapours leaving at the top of the reactor carry some lactam vapours. The lactam content gets fractionated in the distillation column. A portion of the vapours is condensed in the reflux condenser and sent back as reflux. The remaining water vapours are condensed in a separate condenser. The condensed water is discharged through to a receiver which acts as a seal against the atmosphere.

The melt flows through the tube continuously, running down on baffles. The residence time in the reactor is roughly twenty hours. The polymer outlet at the lower part of the reaction is fitted with metering pumps. The pumps deliver the polymer to the spinning head through a diphyl jacketed pipe. The pumps are coupled to the adjustable drives which provides for a control of polymer discharge from the V.K. tube. The polymer gets filtered in the spin packs and is extruded in the form of spaghettis through spinnerets. The spaghettis pass through a cooling pan containing demineralized water. The temperature of cooling pan water is controlled by means of a recirculation system consisting of a pump and heat exchanger.

The strings are drawn off via a squeezing (take off) device and fed to the chip cutter. A roller pulls the strings in the cutter and they are out by the blades into chips of about 2 to 3 mm. The chips from the cutter are led to chip silo from where they are fed to the extractor.

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D. Chip washing and drying

At polymerization equilibrium about 90 % of monomer is converted to polymer. The rest of 10 % consists, mainly, of the monomer and 3 me cyclic dimers and trimers. The unreacted monomer and other low more ular components should be extracted in order to avoid difficulties in spinning and yarn processing operations. This is done by boiling water extraction. The extraction is carried out in three stages by a counter current cycle.

The water for extraction is supplied from three tanks by a free flow pump and the hot water temperature is maintained by the heat exchanger. The washing is started with the maximum monomer enriched water from previous batch and ends with fresh demineralized water. In this way every batch of chips is washed thrice. After each washing, water is transferred to the next tank. Monomer enriched water, from the final tank, is sent for recovery of monomer.

The chips, after washing, are pneumatically conveyed to the batch centrifuge where their moisture content is reduced to less than 1 %. Centrifuged chips are transferred to intermediate storage silos from where they are charged into jacketed vacuum drier. The chips must be completely free of moisture (0.1% max. allowable moisture) as the presence of moisture causes breakages during filament spinning. The jacket of the drier is supplied with hot water from a circulation system consisting of a surge tank, a heat exchanger and a pump, and it maintains drier temperature at 90° to 95° C. Vacuum to the drier is applied by low and high vacuum pumps and the final vacuum is of the order of 1.0 torr.

An oil scrubber in the vacuum system prevents chips powder/dust from getting into the vacuum pumps. After drying, chips are pneumatically conveyed to the chips silos. Pure nitrogen is used as the medium for conveying. The conveying system consists of the lift gas blower, dust catcher, filters and coolers. The chips from the storage silos are pneumatically conveyed to the extruder feed silos for spinning the filament yarn.

E. Spinning

The dried chips are fed to horizontal extruders via ohips feed silos, kept free of oxygen by purging with pure nitrogen. The screw casing in the extruder is electrically heated in independent zones and kept at uniformly

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constant temperatures. The molten, homogeneously mixed polymer, is supplied to the spinning boxes by distribution pipes. The spinning box houses the metering pumps which supply the polymer melt to the spinnerets through spin packs for filtering the melt. The melt comes out of the spinnerets in the form of filaments. The holdup time in the distribution pipes, spinning boxes etc. is short and constant from one spinning position to the other which is important for similar characteristics of all the filaments. The melt solidifies as the filaments come into contact with the nitrogen flowing co-currently in the blow ducts. The nitrogen in the ducts is blown at definite velocity, temperature and humidity. Crystallinity in the yarn is dependent on the rate of cooling in the blow ducts. The uniform treatment is very essential for good denier consistency.

The filaments are drawn off, after passing through spinning tubes, by the take-up machine. Before being wound up on take-up bobbins, spin finishes are applied by finish rolls to the yarn to give the filaments antistatic properties, smoothness, adhesiveness and adequate moisture pick up. The finish solutions are made in a separate system and are supplied to the finish rolls by a circulating system.

Godets, drive rolls and traverse motion for making yarn packages in the take-up machines are driven at very uniform speeds with the help of synchronous motors. The speeds are controlled by frequency converter sets.

The filament take-up bobbins are retained in a conditioned room for 12-16 hours for yarn conditioning, before sending to the draw twister for stretching of the yarn.

F. Draw twisting

The take-up bobbins carrying undrawn yarn are loaded onto the draw twisting machine. The filaments are cold stretched and are drawn approximately four times to obtain longitudinal orientation of the molecular chains. The stretching is carried out at controlled temperature and humidity. During this process the yarn gets its specific strength and elongation properties.

After being drawn off the bobbin, the yarn runs through a thread guide and tension bars for adjustments of tension in the yarn. The yarn then passes through the feed roller and goes to the chromium plated godet. This region between the feed roller and the godet is the drawing zone. A mechanical pull is exerted because of the difference in speeds of the feed roller and the godet. The yarn is then taken-up on a rotating cop. The build up of the cop is regulated by a traverse mechanism of the ring rails. A traveller, through which the yarn passes before being wound up on the cop, runs circumferentially on the ring and gives a uniform package build up.

G. Caprolactam recovery section

The polymerization waste from spinning, take-up and draw twisting sections together with a non-volatile acid, like phosphoric is charged in lots in a depolymerization reactor where it is treated with superheated steam. The steam promotes depolymerization, vaporizes reconstituted caprolactam and acts as a carrier for the vapour. The vapour is condensed, and stored in a tank. If necessary, it is treated with potassium permanganate $(KMnO_4)$ which helps in improvement of permanganate number in the recovered lactam. The treated solution is filtered and collected in an intermediate tank and then sent to a three stage evaporator.

The wash water from the extraction section is received in a separate tank and pumped to the three-stage evaporator. The wash water and depolymerized solution are evaporated either together or separately, depending upon the quality of the depolymerized solution and the product specifications desired. It is concentrated to a 70 % lactam solution in the evaporator and then treated with NaOH solution which acts as a stabilizer during distillation. The treated solution is transferred to the distillation still which is operated batch-wise and under vacuum and is heated by steam coils.

The distillation is carried out in four cuts. The first cut is collected up to 110° C. It is mainly water and is drained out. The second cut is collected between 110° C and 115° C. It is mainly water along with some quantity of lactam. When the temperature becomes steady at 115° C, pure laotam starts coming out and is transferred to the jacketed lactam tank under nitrogen blanketing. When the temperature starts rising again the fourth cut is collected which is lactam with some oligomers. This operation is carried up to 165° C after which the portion remaining in the still, which is mainly unconverted polymer, is drained out.

The second and fourth cuts are mixed with the next batch. Pure lactam from the lactam tank is passed to the powdering machine where it solidifies on a cooled roller and is powdered. The powdered lactam is filled up in bags, weighed and transferred to the polymerization section.

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Annex VII

EQUIPMENT	LIST	FOR	NYLON-6	PLANT
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lumber required	Description	Material
	A. <u>Monomer melting</u>	
1	Feeding device with built-in lump crusher	SS
1	Lactam melter jacket for hot water heating, inner steam heating coil	SS with CS jacket SS coil
2	Lactam pumps centrifugal type, jacket for hot water heating	SS with CS jacket
2	Lactam filters basket type, filter jacket for hot water heating	SS with CS jacket SS wire mesh
1	Nixing tank jacket for hot water heating, with agitator	SS with CS jacket SS agitator
1	Compensation vessel jacket for steam heating	SS with CS jacket
1	Sealing tank	CS
1	TiO ₂ preparation tank, with agitator	SS
1	Colloid mill for micro grinding, with sealing liquid system and built-in drive	SS
1	TiO ₂ mixing and settling tank, with agitator	SS
1	TiO ₂ surge tank, with agitator	SS
2	Two-component metering pumps for proportioning of lactam and TiO ₂ suspension, jacket for steam heating, with DC-drive and electrical control for the DC motors	SS
1	Run down tank, jacket for hot water heating	SS with CS jacket
1	Metering tube jacket for hot water heating	SS with CS jacket
1	Mixing chamber jacket for hot water heating	SS with CS jacket SS internals
2	Hot water circulation pumps (1 (stand by)	CS
1	Heat exchanger for hot water system	CS
1	Expansion vessel for hot water system	CS
1	Dust exhaust fan	SS
1	Lactam filter, with jacket for hot water heating	SS with CS jacked SS wire mesh
	B. Polymerization	
1	V.K. tube with internals to guide the melt flow, diphyl heated by heat exchangers flanged between the V.K. tube sections and by heating jackets	SS with CS jacket SS internals
1	Overhead distillation column filled with rasching rings	SS

umber equired	Description	Natorial
1	Vapour condenser	Shell-side SS, tube-side CS
1	Reflux condenser	58
1	Seal pot	58, glass
3	Netering pumps precision gear pumps, jacket for diphyl heating	CB, special steel
3	Slide valves for the spinning pumps	55
3	Pump shafts (1 spare) for the spinning pumps, complete with shear pin and coupling	CS
2	Adjustable drives with DC motors, including electrical control for the DC motors	
1	Diphyl storage tank with internal coil for steam heating	CE
1	Diphyl condenser	CE
1	Diphyl feed pump, centrifugal type	CS
3	Diphyl heaters for electrical heating	CS
3	Expansion vessels for diphyl cycles	CS
1	Spaghetti spinning head, for 2 spinneret packs, jacket for Dowtherm heating	SS with CS jacket
4	Spinneret packs with distribution plates and spinnerets	86
1	Cooling pan, for cooling strands from 2 spaghetti spin packs	86, C 8 frame
1	Take-off device, complete with drive and infinitely variable gear	98, CS
2	Chip cutters, complete with drive and infinitely variable gear	88, C 8
1	Quench water cooler	Shell-side SS, tube-side CS
2	Quench water circulation pumps, centrifugal type	55
5	Diphyl circulation pumps, canned motor pumps	C S
	C. <u>Discontinuous extraction</u>	
2	Chip silos	88
1	Extractor, with internals for guiding the chip flow and a steam heating jacket at the vessel bottom	58
2	Free flow pumps	88
1	Centrifuge	55
2	Chip silos	58
4	Extraction water tanks	SS with CS jacket

Number required	Description	Material	
2	Lactam water pumps, centrifugal type	55	
1	Heat exchanger	Shell-side CS, tube-side SS	
1	Chips silo	55	
1	Cyclone	58	
	D. Chip drying, transportation and storage		
2	Tumbling driers, 16 m ³ volume, with special gear motor	55, C 3	
1	Oil scrubber, with heating device and a bed of rasching rings	CS	
1	Oil pump of special design	C 8	
1	Low vacuum pump, roots type	C S	
1	Condenser, with condensate collecting vessel	CS	
1	High vacuum pump, rotary oil seal type	C S	
1	Heat exchanger	C S	
2	Hot water circulation pumps	C 5	
1	Expansion vessel	CS	
1	Gate valve for emptying the chips out of the drier, special vacuum-tight design	88	
2	Liftgas blowers, roots type, complete with motor, belt drive, silencers, compensator, safety valves for over and under pressure	C 8	
1	Dust catcher, with gear motor for change of the filter bags	3 5 C 5	
1	Liftgas filter before blower	C 8	
1	Liftgas filter after blower, candle filter type	58, A1, CS	
1	Liftgas cooler for cooling	C S	
3	Rotary gates below the chip silos, complete with gear motor	38	
7	Pipe switches, piston operated (pressurized air of 5 to 8 atm) with operating pistons and limit switches	88	
9	Cyclones at the inlets of 3 storage silos and 6 extruder feed silos	58	
3	Chip storage silos	5 8	
	E. Spinning		
6	Extruder feed silos	58	
6	Connection pieces between extruder feed siles and extruders	38	

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Number required	Description	Naterial
6	Extruders (melting capacity approx. 105 kg/hr) electrically heated, pressure controlled, cooled chips feeding system, including drive and motor, control center and measuring head	Barrel lined with X-alloy, CB
6	Nelt distribution pipes, for distribution of melt to 4 spinning boxes each	55 with CS jacket
24	Spinning boxes, each equipped with two spinning positions, provided for Dowtherm heating	58 with CS jacket
6	Spinning pump main drives with DC motor, gear, connection shaft and coupling, each to drive 8 spinning positions (16 quadruple spinning pumps)	
3	Dowtherm evaporators for electrical heating	C S
48	Blow ducts	A1, C5
48	Spinning tubes	A1, C S
3	Take-up machines for textile yarn, double-sided design, equipped with traverse motion system	
	Each machine has 8 spinning positions per side, 16 in total, separate drive for finishing and moistening, also separate for each side and includes:	
	Generator set for godets, drive rolls and travers motion system.	
	One additional generator set for machine 3 for different take-up speed on each side.	
	One additional set of change parts for the traverse motion system of one side for machine 3 to change the winding from multifilaments to monofilaments.	
144	Spinning accessories comprising quadruple spinning pumps 4 x 0.6 m^3 /rev (96 in operation, 48 stand by)
384	Spinnerets, 50 mm \emptyset , 10 holes (round) to den (max. 192 in operation, 192 stand by)	
384	Spinnerets, 50 mm Ø holes (round) for 15 and 20 dem (max. 192 in operation, 192 stand by)	1
64	Spinnerets, 70 mm Ø, 34 holes (round) for 100 den and 70 den (max. 32 in operation, 32 stand by)	
100	Spinneret packs for 2 spinnerets 70 mm ø (max. 16 in operation, 16 stand by)(alternatively 64 spinneret packs for 1 spinneret)	
1	Preheating oven for pumps and spinnerets	
3	Precision scales, 0-100 g	
1	Air velocity meter	
4	Stop watches	

Number required	Description	Naterial
2	Revolution counters, 0-1,000 rev./min.	
1	Communication system between spinning and take-	up
4	Table scales, 0 - 10 kg	
1	Set of operating tools (hooks, scrapers, etc.)	
4	Assembly fixtures for spinneret packs	
2	Assembly fixtures for spinning pumps	
1	Complete set of general and special tools for spinning plant	
1	Spinneret testing device	
,500	Sets of filters and gaskets	
,000	Take-up bobbins	
20	Spray-cans for spinnerets	
	Molykote paste, 4 kg	
1	Finishing and wetting system, complete with 3 preparation tanks for finish oil and 3 prepar tanks for moistening emulsion	ation
	F. Draw-twisting	
17	Draw-twisters for textile filaments, each with stretching positions, complete with machine fra drive with transmission gears, bobbin holders, filament guide, feeding mechanism, draw godets, hydraulic ring-rail motion	me, inlet
	Draw-twister accessories	
102	Sets of change gear wheels for draw ratio (6 sets/machine)	
85	Sets of change gear wheels for draw-twisting sp (5 sets/machine)	beed
51	Change pulleys for spindle speed (3/machine)	
	3,000 run-off covers	
1	Lubrication device for spindles	
20	Electrically heated waste cutting knives	
10	Traveller pixers	
1	Set special tools	
1	Spindle band sewing machine	
1	Waste cutting unit	

umber equired	Description	Naterial
	G. <u>Caprolactam recovery section</u>	
2	Steam superheaters	55
1	Depolymerization reactor	58
1	Condenser cum cooler	56
1	Raw monomer receiving tank	55
2	Transfer pumps	55
1	Mixing vessel with agitator	88
1	Treatment tank with agitator	88
2	Filter feed pumps	55
1	Filter	55
1	Intermediate tank	88
2	Transfer pumps	
1	Wash-water receiving tank	88
2	Wash-water feed pumps	55
1	Three stage evaporator	35
1	Concentrated solution storage tank	55
2	Concentrated solution transfer pumps	88
1	Shell and tube condenser	CB
1	Nixing vessel with agitator	CS
1	Treatment tank with agitator	58
1	Distillation still, with heating coil and jacket	SS with CS jacket and SS coil
1	Distillation overhead condenser	85
1	Protective condenser	35
2	Intermediate jacketed tanks	SS with CS jacket
1	Multi-stage steam jet ejector system	CB
1	Sealing tank	C S
2	Recovered lactam pumps	86
2	Jacketed lactam tanks	SS with CS jacket
1	Powdering machine	88
1	Flash drum	CS
1	Distilled-water tank	C 5
1	Distilled-water transfer pump	CB
1	Bagging and weighing machine	CS

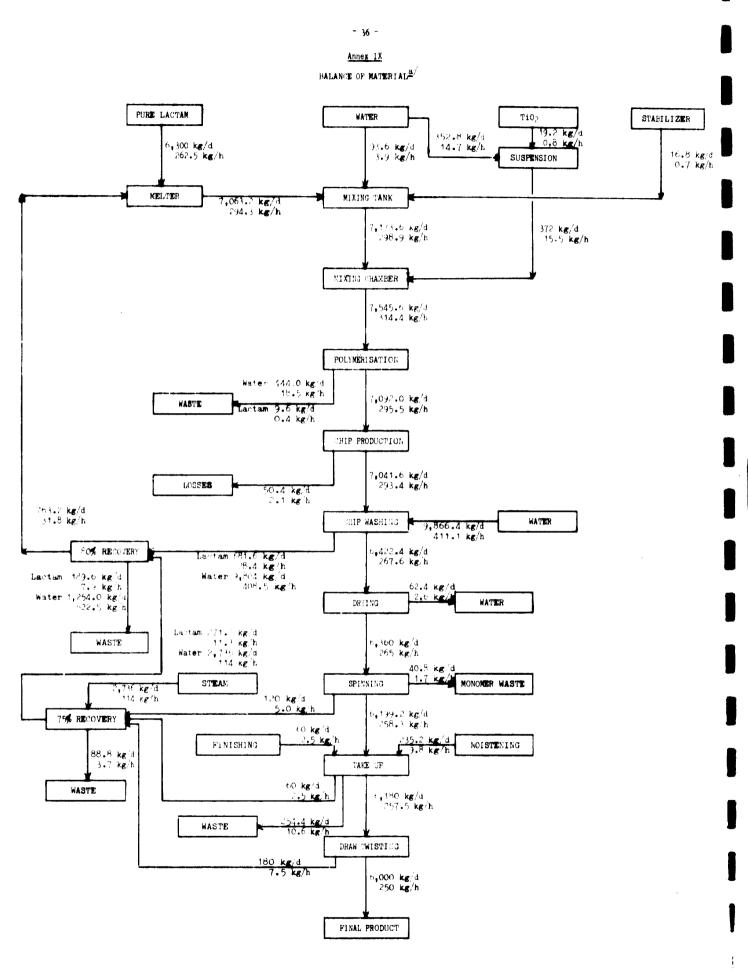
Number required	Description
	H. Auxiliary units
1	Nitrogen purification unit, complete with nitrogen storage tank purification system, pressure reducer, oxygen tracer, switchboard with hydrogen analyser
1	Complete cleaning unit for cleaning spinning pumps and spinneret packs
	I. Textile laboratory
1	Analytical balance, weighing range: 0-1000 g, accuracy: 10 mg
1	Microscope, binocular, with object micrometer, graduated dial and
	objective revolver objective 3.2/012 10/0.25 40/0.65 ocular 10 x ocular 6 x
1	Nicrotome
2	Precision yarn reels with meter control, reel circumference: 1 m, with 5 reeling positions
1	High-speed scale for table mounting, weighing range: 0-10 kg
3	Denier balances, weighing range: 0-100 den filament suspension: 90 m
2	Precision yarn reels, motor driven, with meter control, reel circumference: 1 m, with 5 reeling positions, draw-off device for pirns
1	Automatic strength tester, with multiple bobbin attachment
1	Yarn evenness tester (seriplane), motor driven, with winding boards 600 x 260 x 160 mm
5	Winding boards 600 x 260 x 160 mm
1	Electronic "USTER" evenness tester, model C, with rotafil and integrator
1	Research microscope, binocular with polarisator and analysator lenses, objective revolver and complete attachment for camera objective: 6.5/0.18 3.5/10 11/0.25 10/25 11/0.45 40/65
	ocular 6 x ocular 10 x
1	Torsion precision scales, weighing range: 0-50 mg
2	Nechanical filament tension meters, range: 1-12 g
2	Nechanical filament tension-meters, range: 5-50 g
1	Stroboscope for 600-14,000 rew/min.
1	Densimeter for 0-100° shore-hardness
1	Balance for table mounting, range 0-10 kg, with high speed

Number required	Description
4	Sets, preparation and langet needles
300	Specimen holders 76 x 26 mm
300	Coverslips 18 x 18 mm
300	Coverslips 18 x 36 mm
20	Bottom dies 80 x 25 mm, 6 holes 0.8 mm diameter
	J. Chemical laboratory
1	Nelting point apparatus, with test tubes
12	Colour comparison tubes with shadowless bottom, 10 ml volume (Nessler tubes)
1	Constant temperature bath for 20°C
5	Reitmeier tops of special design
5	Liebig condensers
5	Absorption receivers to Fresenius of special design
3	Nagnetic stirrers
15	Teflon stirring bars
3	Hydrometers, range 1,000-1,070 ° 36
2	Acid pumps
3	Stirrers with motor
1	pH meter with electrodes, with temperature compensator
3	Platinum dishes, 8-9 om diameter
1	Photometer, Elko II, complete
2	Filters 490 Nm
5	Bulbs, depth of bed 2 mm
1	Nuffle furnace
1	Analytical balance, Model 5B, Nettler
1	Karl Fischer titrator, complete, with one spare electrode unit
30	Titration cells
2	Pipet filling attachments
1	High speed incinerator
1	Hot-plate for Brienmayer flack
20	Suepension cylinders
1	Immersion refractometer, Zeiss, with Prism 1 (not temperable), range 1.325-1.366, complete, with a spare prism and 20 refracto- meter glasses

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Number required	Description	
1	Constant temperature bath for tempering the flasks	
1	Constant temperature bath for immersion, refractometer with thermometer, with a joint scale for 0-50 C, length of stem 10 cm, $1/10$ graduation	
1	Shakar	
10	Ubbelohde viscosimeters II, diameter of capillary approx. 1 mm	
10	Netal frames for viscosimeters	
1	Viscosimeter, constant temperature batch with 5 suspension	
. 1	Diagonal cutting pliers or mill to disintegrate the chips	
10	Extraction apparatuses, complete	
10	Attachments for the recovery of the petroleum ether	
25	Squibb funnels, 250 ml, with ground stopper	
3	Water baths, with 4 heating points	
12	Support rings, split type, 10 cm diameter	
2	Clocks, interval timers, for 60 minutes	
5	Desiccators for 5 flasks, approx. I.D. 300 mm	
3	Drying ovens	



🕈 Source: Engineers India Limited, New Delhi.

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