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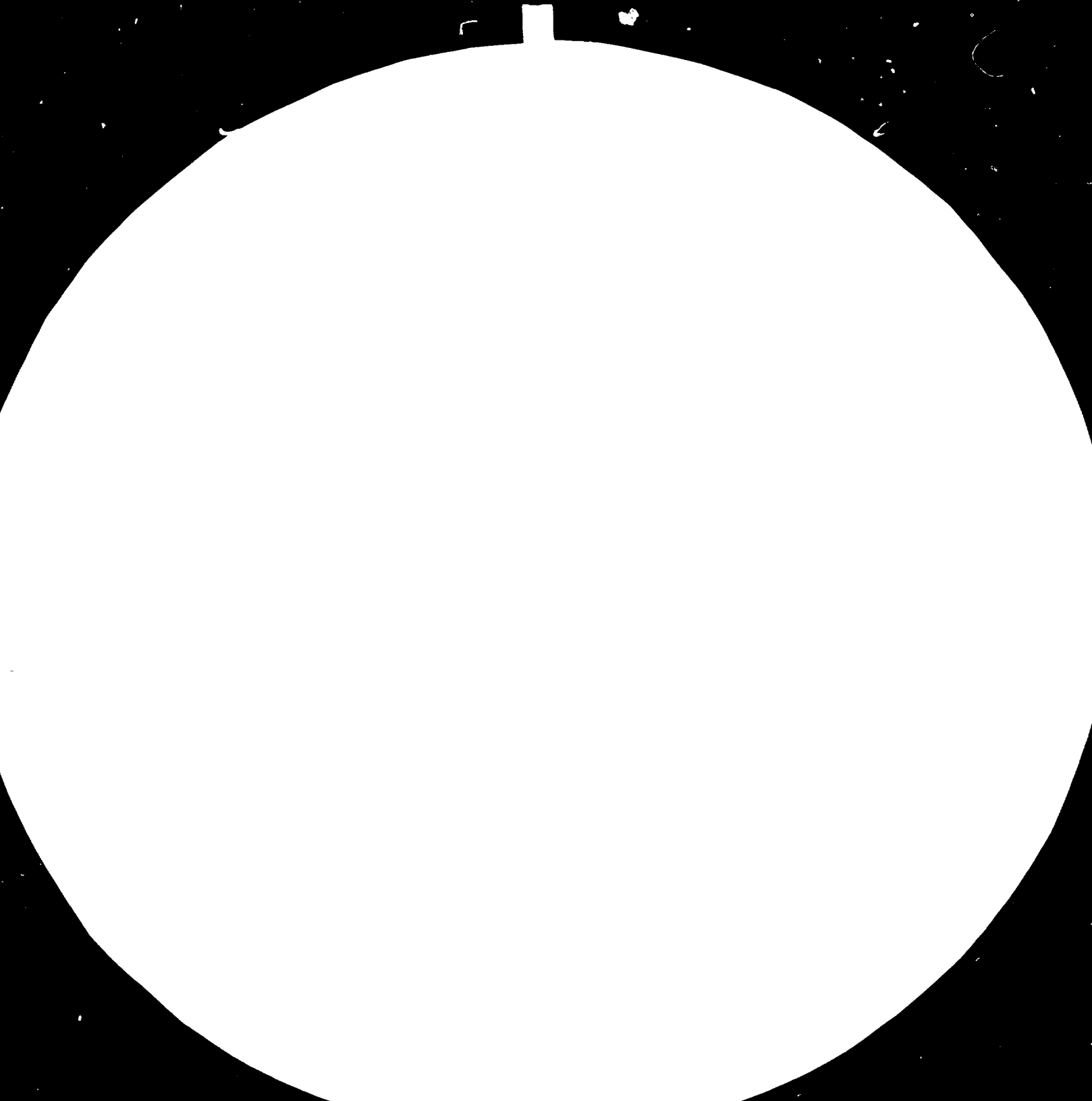
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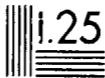
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Resolution test charts are available from the following sources:

1. National Bureau of Standards, Gaithersburg, MD 20899

2. National Institute of Standards and Technology, Gaithersburg, MD 20899

3. National Institute of Standards and Technology, Boulder, CO 80506

4. National Institute of Standards and Technology, Washington, DC 20585

14017



1984

FINAL REPORT

India.

on the commissioning of the polymerization plant for Polyester, Polyamide 6.6 and Polyamide 6 at SASMIRA, India

Contract No. 84/01
Project No. DP/IND /83/015
Activity Code DP/02 /32.1

between

THE UNITED NATIONS INDUSTRIAL DEVELOPMENT ORGANIZATION (UNIDO)

and

KARL FISCHER INDUSTRIEANLAGEN GmbH (Berlin-West)

This FINAL REPORT comprises

- 13 pages of text
- and six (6) Annexes (A through F)



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ANNEXES

The Annexes (A through F) of this FINAL REPORT mentioned hereunder have already been sent to you directly by SASMIRA soon after the start-up was finished.

- A Interim notice to the start-up of Polyester production
(of 17-7-84)
- B Interim notice to the start-up of Polyamide 6.6 production
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- C Final notice to the start-up of the polymerization plant
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- F Analytical Results and Conclusion
(by Dr. P. Hirt, Polymer expert of Institut für Chemiefasern, Stuttgart)



1. Modification of the plant

In the period of 15-5-84 to 2-7-84 the following modification work was carried out by

Mr. U. Lazik, Mechanical Engineer
Mr. N. Wagner, Electrical "
Mr. N. Nietsch, Electronical "

according to KARL FISCHER - Report of 20-5-83 paragraph 4
(Annex G of UNIDO-Contract No. 84 / 01):

- Modification of pressure measurement piping (autoclave)
- Enlarging of casting vat
- Displacing of vacuum control valve (autoclave)
- Fitting of suitable pressure reduction stations (nitrogen feeding system)
- Modification of piping (delusterant preparation plant)
- Modification of the stored process computer and supply of new software
- Furthermore the modification work is described in Annex E of this FINAL REPORT



2. Production of Polyester chips

The preparation for and the operation of the production of Polyester chips was carried out in the period of 3-7-84 to 18-7-84 by the KARL FISCHER-Team, consisting of:

Mr. G. Panke	Chief of Process
Mr. U. Lazik	Mechanical Engineer
Mr. N. Wagner	Electrical "
Mr. N. Nietsch	Electronical "
Mr. R. Hagen	Chemical "
Mr. T. Daebel	Process "

Of 21 batches in total, No. 10-18 represent the warranty performance test, which was made with constant process parameters (listed below).

2.1 Preparations

- Tightness test of autoclave and tumble dryer
- Gauging level indicator of glycol dosing tank, as well as methanol and glycol collecting tanks.
- Preparing of delusterant suspension
- Preparing solutions of stabilizer and catalysts in the chemical laboratory
- Cleaning of main reactors and connecting pipes (inside) with ethylene glycol
- Testing of process units
- Programming computer for Polyester process
- Process running simulation



2.2 Operations

2.2.1 Melting of DMT (Dimethyl-terephthalate)

Quantity	132	kg/batch
Temperature	164	°C
Melting time	appr. 3	hrs

2.2.2 Transesterification

Feeding of EG (ethylene glycol)	80,4	kg/batch
Temperature:	at start 165 at end 272	°C °C
Catalysts:	NaAc 8,84 MN(Ac) ₂ 49,39	g g
Methanol-phase	3,5	hrs
Delusterant (Titaniumdioxide)	550	g/batch
Polycondensation catalyst:	Sb ₂ O ₃ 58,74	g
Polycondensation stabilizer:	TPP 92,78	g
Glycol phase	1,5	hrs
Process-glycol	appr. 25,2	kg

2.2.3 Polycondensation

Process time	4,5	hrs
Melt temperature	275	°C
Vacuum	1..2	mbar
Process glycol	appr. 25	kg



2.2.4 Outcasting and chips cutting

Duration		30..35	min
Drawing speed	appr.	42	m/min
chips size:			
- diameter	appr.	2,5	mm
- length	appr.	2,0	mm

2.2.5 Chips drying

Duration:	appr.	24	hrs
Chips temperature	appr.	148	°C
Vacuum:	appr.	0,3	mbar

2.3 Polymer properties

The Fibre Research Laboratory of SASMIRA obtained the following analytical results (given as average values of batches No. 10-18):

Relative viscosity (25 °C, Phenol- Tetrachlorethane 1 : 1, C = 0,5 %)	1,343	
Intrinsic viscosity	0,647	
Carboxyl end groups	20,2	mval/kg
Melting point (by Differential Scanning Calorimeter)	253,7	°C
Ash content	0,443	% b.w.
Residual moisture less than	0,008	% b.w.

compare with Annex F, paragraph (1)



3. Production of Polyamide (Nylon) 6.6 chips

The preparation for and the operation of Polyamide (Nylon) 6.6 chips was carried out by the KARL FISCHER-Team in the period of 19-7-84 to 3-8-84. Of 21 batches in total, batches No. 9-16 represent the warranty performance test which was made with constant process parameters (listed below).

3.1 Preparations

- Cleaning main reactors and connecting pipes (inside) with ethylene glycol
- Fitting of pressure control valve (pos. 1104)
- Gauging level indicator of DM (demineralized) water tank
- Preparing of delusterant suspension
- Preparing solutions of stabilizer and oxidation inhibitors in the chemical laboratory
- Programming computer for Polyamide 6 process
- Testing of process units
- Process running simulation



3.2 Operations

3.2.1 Dissolving of AH-Salt

AH-Salt:	120,5	kg/batch
DM-water:	71	l/batch
Concentration:	63	% b.w.
Temperature:	80	°C
Dissolving time:	appr. 3	hrs
Stabilizer:	208	g Acetic acid
Oxidation inhibitors:		
41,6 mg	Hydraziniumhydroxide	
169,9 mg	Ammonia	

3.2.2 Preconcentration

Temperature:	140	°C
Pressure:	1,7	bar
Condensate:	35	l water/batch
Concentration:	appr. 77	% b.w.
Duration:	appr. 3	hrs

3.2.3 Polycondensation

Process pressure	19,0	bar
Melt temperature	280	°C
Process-water	appr. 52	kg
Process-time	4,5	hrs
Delusterant (Titaniumdioxide)	510	g/batch



3.2.4 Outcasting and chips cutting

Duration	appr.	30	min
Drawing speed	appr.	68	m/min
chips size:			
- diameter	appr.	2,2	mm
- length		1,8..2,0	mm

3.2.5 Chips drying

Duration	appr.	24	hrs
Chips temperature		100	°C
Vacuum	appr.	0,3	mbar

3.3 Polymer properties

The FIBRE RESEARCH LABORATORY of SASMIRA obtained the following analytical results (given as average values of batches No. 9-16):

Relative Viscosity (25 °C, Sulphuric acid, C = 1 %)		2,322	
Melting point (by DSC)		261,5	°C
Ash content		0,49	% b.w.
Residual moisture	less than	0,1	% b.w.

compare with Annex F, paragraph (2)



4. Production of Polyamide (Nylon) 6 chips

The preparation for and the operation of the production of Polyamide (Nylon) 6 chips was carried out in the period of 4-8-84 to 8-8-84 by the KARL FISCHER-Team. The computerized production has been demonstrated by running two batches.

4.1 Preparations

- Cleaning main reactors and connecting pipes (inside) with lactam
- Removing of pressure control valve (pos. 1104)
- Testing of process units
- Programming computer for Polyamide 6 process

4.2 Operations

4.2.1 Melting of caprolactam

Quantity:	145	kg/batch
Melting temperature:	85	°C
Duration:	appr. 2	hrs
Stabilizer:	210	g Acetic acid

4.2.2 Delusterant of monomer

Titanium dioxide	663	g/batch
Mixing time:	appr. 1	hrs



4.2.3 Polycondensation

Melt temperature		260	°C
Pressure phase	appr.	3	hrs
Vacuum phase	appr.	2	hrs

4.2.4 Outcasting and chips cutting

Duration	appr.	60	min
Drawing speed	appr.	81	m/min
chips size:			
- diameter	appr.	1,7	mm
- length		2,0..3,0	mm

4.3. Polymer properties

The FIBRE RESEARCH LABORATORY of SASMIRA obtained the following analytical results:

Batch No.	1	2	
Relative viscosity (25 °C, Sulphuric acid, C = 1 %)	2,62	2,62	
Melting point (by DSC)	215,7	218,6	°C
Ash content	1,13	0,42	%

compare with Annex F, paragraph (3)



5. Conclusion

The commissioning (modification and start up) of the autoclave polycondensation plant for Polyester, Polyamide (Nylon) 6.6 and Polyamide (Nylon) 6 chips at "The Silk and Art Silk Mill's Research Association" (SASMIRA) has been successfully accomplished by KARL FISCHER Industrieanlagen GmbH (Berlin-West) according to UNIDO-Contract No. 84/01.

KARL FISCHER agrees to supply the in Annex C (of this FINAL REPORT) mentioned parts to SASMIRA.

The KARL FISCHER-Team left the polycondensation plant on 8th of August '84 in a perfect technical condition, i.e. every mechanical, electrical and process units were fully ready for work.

Berlin, ²²⁻⁸⁻⁸⁴

[Signature]
.....
Panke

[Signature]
.....
Daebel

