

I. C. 7370,
July 1946.

INFORMATION CIRCULAR

UNITED STATES DEPARTMENT OF THE INTERIOR - BUREAU OF MINES

REPORT ON THE INVESTIGATION BY FUELS AND LUBRICANTS TEAMS AT THE
I. G. FARBENINDUSTRIE A. G. LEUNA WORKS, MERSEBURG, GERMANY

Edited by R. Holroyd^{1/}

CONTENTS

	<u>Page</u>
Foreword	1
Introduction, by R. Holroyd	ii
Personnel of visiting teams	iii
I. Gas production, by G. U. Hopton, R. J. Morley, L. L. Newman, and W. W. Odell	1
Gas production	1
Water-gas generators	1
Winkler generators	2
Slagging producers ("Abstichgeneratoren")	5
Pattenhausen generator	6
Oxygen plant	6
Documents in connection with gas production	7
H ₂ S removal	7
Alkacid process	7
Active carbon process	8
Organic sulphur	9
CO conversion	9
II. Ammonia synthesis, by R. J. Morley and W. W. Odell	10
General	10
Staff interviewed	10
Compression	10
CO ₂ removal	11
CO removal	12
Final NH ₃ make-up gas purification	12
NH ₃ synthesis	12
Circulator	13
Catalyst	13
Converters	14
III. Methanol and higher alcohol synthesis, by R. J. Morley	15
Methanol	15
Higher alcohols	15
IV. Hydrogenation, by R. Holroyd and W. F. Faragher	18
A. General	18
B. Brown-coal hydrogenation	18
(i) Outline of present process	18
(ii) Consideration of process improvements made since the war	20
(iii) Details of plant equipment	22

^{1/} British Ministry of Fuel and Power.

CONTENTS (Continued)

	<u>Page</u>
IV. Hydrogenation (Continued)	
C. Brown-coal tar hydrogenation	24
D. Vapour-phase hydrogenation of middle oils	25
(i) Process conditions and yields	25
(ii) Details of plant	27
E. Vapour-phase splitting hydrogenation at 700 atmospheres pressure	28
F. Hydrogenation costs	29
V. D. H. D. (Dehydrierung - Hoch - Druck) process	38
(i) Description of process and Leuna operating results	38
(ii) Costs	40
VI. Hydroforming process, by E. Cotton and V. Haensel	43
VII. Arobin process, by D. A. Howes, J. G. Allen, and H. Schindler ...	46
Charge to Arobin plant	46
Operating conditions	46
Yield and quality of the product	47
Preparation of the Arobin catalyst	47
VIII. Catalytic cracking, by E. Cotton and V. Haensel	49
Introduction	49
Reaction conditions	49
Catalyst	49
Products	50
Catalyst life	51
Hydrogenation of product	51
Conclusions	51
IX. Dehydrogenation of butane - alkylation, by D. A. Howes, J. G. Allen, and H. Schindler	52
Introduction	52
Summary of operations	52
Dehydrogenation	52
Catalyst regeneration	54
Catalyst manufacture	54
Preparation of alkylation feed stock	55
Alkylation	55
Alkylate quality	56
Alkylate production in Germany	56
Economics of Leuna operation	57
X. Isomerization of normal butane, by E. Cotton and V. Haensel	59
XI. Synthetic lubricating-oil manufacture - Leuna, by D. A. Howes, J. G. Allen, H. Schindler, C. C. Chaffee, and R. J. Ozol	60
Summary of process	60
Process details	60
(1) Thermal cracking of ethane	60
(2) Acetylene removal	61
(3) Oil absorption	61
(4) Activated-charcoal absorption	61
(5) Alkazid washer	61
(6) Caustic washer	61

CONTENTS (Continued)

	Page
XI. Synthetic lubricating-oil manufacture - Leuna (Continued)	
Process details (Continued)	
(7) Linde low-temperature fractionation	62
(8) Polymerization of ethylene to lubricating oil	62
(9) Catalyst separation and disposal	63
(10) Oil fractionation	64
Operating requirements	64
Tests on products	65
Production data	66
Notes on catalysts and reaction conditions	67
(1) Acetylene removal catalyst	67
(2) Ethylene purity required	67
(3) Polymerization catalyst	68
Theory of lubrication	68
Sources of olefines	71
Hydrocarbon synthesis	71
Lubricating-oil additives and special products	72
I. Additives	72
II. Special lubricants	75
Conclusions and recommendation	77
XII. Synthetic lubricating oil - Schkopau, by P. K. Kuhne	78
Introduction	78
Description of the process	78
Miscellaneous	80
Conclusions	80
XIIA. Synthetic lubricating-oil manufacture at Stettin-Politz, by	
D. A. Howes, J. G. Allen, and H. Schindler	81
Source of information	81
Summary	81
Description of process	81
Wax cracking	81
Polymerization	82
Refining of polymerized oil	82
Tests on products	83
Yields	83
XIII. Aviation fuel manufacture and engine testing, by D. A. Howes,	
J. G. Allen, and H. Schindler	84
Introduction	84
Octane number determination and specification	84
B. M. W. 3-litre single-cylinder aero engine	85
Aviation gasoline	85
Nomenclature of aviation fuels	86
XIV. OXO process, by V. Haensel and J. P. Jones	87
Introduction	87
Chemistry of the OXO process	87
Process conditions	88
Leuna operation	88
Catalyst preparation	91
OXO processing of cracked middle oil	92

CONTENTS (Continued)

<u>Page</u>		<u>Page</u>
62	IV. Synol process, by V. Haensel, J. P. Jones, and W. A. Horne	93
62	Synthesis gas	93
63	Scale of operations	93
64	Process conditions	93
64	Yields	94
65	Design of reactors	95
66	Catalyst preparation	95
67	Work-up of crude Synol product	96
67	V. Mersol process, by A. J. V. Underwood	98
67	Scale of operation	98
68	Process conditions	98
68	VI. Methylamine, by R. J. Morley	100
71	Isobutylamine manufacture	100
71	Schiff's base	101
72	VII. Manufacture of carboxylic acids, by D. A. Howes, J. G. Allen, and H. Schindler	102
72	VIII. Polymerization of C ₆ and C ₇ olefines, by D. A. Howes, J. G. Allen, and H. Schindler	103
75	IX. Fertilizers and nitric acid, by R. J. Morley	104
77	Ammonium sulphate	104
78	Nitric acid	105
78	Concentrated nitric acid	105
80	Ammonium nitrate	105
80	Calcium nitrate (Kalksalpeter)	106
81	Phosphate fertilizers	106
81	Leuna salt peter (ammonium sulphate and nitrate)	106
81	Ammonium chloride fertilizers	106
81	X. Synthesis of toluene from benzene and methanol, by R. Holroyd and C. C. Chaffee	107
81	Introduction	107
82	General process data	107
82	Plant operation	108
83	Development work on related processes	109
83	XI. Catalyst preparation, by W. A. Horne and W. F. Faragher	110
81	Catalyst 616	110
81	Catalyst 1132	111
81	Catalyst 1750	111
81	Catalyst 2493	111
85	Catalyst 2730	112
85	Catalyst 3076	112
86	Catalyst 3390	112
87	Catalyst 3510	113
87	Catalyst 4577	113
87	Catalyst 4788	114
88	Catalyst 4821	114
88	Catalyst 5058	114
91	Catalyst 5436	115
92	Catalyst 5623	115

CONTENTS (Continued)

	<u>Page</u>
XXII. Catalyst preparation (Continued)	
Catalyst 5633	116
Catalyst 5780	116
Catalyst 6067	116
Catalyst 6069	117
Catalyst 6448	117
Catalyst 6523	117
Catalyst 6853	117
Catalyst 7187	118
Catalyst 10927	118
Ammonia-catalyst (no number)	118
XXIII. Metallurgy, by J. F. Ellis	119
I. G. experience with hydrogen-resistant steels	119
Enamelling of preheater bends	120
Chromium-plated injector rams	120
Material for butane dehydrogenation reactor tubes	121
Material for catalytic cracking regenerators	121
XXIV. Blechhammer, by R. Holroyd and W. F. Faragher	122
Introduction	122
Details of coal hydrogenation to give excess heavy oil	124
Phenol extraction	126
Treatment of hydrocarbon bases	127
Costs	127
XXV. Brux, by R. Holroyd and W. F. Faragher	128
Introduction	128
General outline of the Brux factory	128
(a) Brown-coal carbonization	128
(b) Hydrogen production	128
(c) Hydrogenation plant	129
(d) Phenol extractions	129
Operation of the hydrogenation units	129
Costs	130
Present condition of Brux plant	130
Underground hydrogenation plant	130
XXVI. German oil production, by R. Holroyd	133
XXVII. Use of bottled propane and butane for road vehicles, by R. Holroyd and J. F. Ellis	135

ILLUSTRATIONS

<u>Fig.</u>		<u>Following page</u>
I.	Separation of crude higher alcohols	16
II.	Hydrogenation of brown coal, 1944	18
III.	Hydrogenation of brown coal; 1939-40	20
IV.	Hot catchpots	22
V.	Flow sheet for petrol from brown-coal tar	24
VI.	Flow sheet for production of motor or aviation petrol from brown-coal middle oil	26

<u>Fig.</u>	VII.	Comy
	VIII.	Flow
	IX.	Hyd
	X.	Flow
	XI.	Hyd
	XII.	Flow
	XIII.	Flow
	XIV.	Flow
	XV.	Cat
	XVI.	Cat
	XVII.	Mat
	XVIII.	But
	XIX.	Sul
	XX.	Top
	XXI.	Cor
	XXII.	All
	XXIII.	Flow
	XXIV.	Th
	XXV.	Sol
	XXVI.	Fl
	XXVII.	Fl
	XXVIII.	Fl
	XXIX.	Fl
	XXX.	Co
	XXXI.	Co
	XXXIA.	Da
	XXXIB.	Co
	XXXII.	S
	XXXIII.	S
	XXXIV.	P
	XXXV.	S

ILLUSTRATIONS (Continued)

Page	Fig.	Following page
116	VII.	Comparative flow sheets for petrol from bituminous-coal-tar middle oil 28
116	VIII.	Flow sheet for motor petrol from brown coal by hydrogenation 28
116	IX.	Hydrocarbon-gas flow sheet, motor petrol from brown coal 28
117	X.	Flow sheet for aviation petrol from brown coal by hydrogenation 28
117	XI.	Hydrocarbon-gas flow sheet, aviation petrol from brown coal 28
118	XII.	Flow sheet for D. H. D., based on achieved results, first quarter, 1944 38
118	XIII.	Flow diagram, D. H. D. process 38
119	XIV.	Flow diagram, hydroforming process 42
119	XV.	Catalytic cracking research unit, Deusen 48
120	XVI.	Catalyst valve - catalytic cracking unit 48
120	XVII.	Material balance - butane dehydrogenation and alkalation, Leuna works 52
121	XVIII.	Butane dehydrogenation, Leuna works Ammoniakwerke, Merseburg 52
122	XIX.	Sulfuric acid alkylation, Ammoniakwerke, Merseburg, Leuna works 56
124	XX.	Tops of two dehydrogenation reactors 52
126	XXI.	Control panel for one of regenerators 52
127	XXII.	Alkylation reactor 56
127	XXIII.	Flow diagram of isomerization process 58
128	XXIV.	Three isomerization reactors 58
128	XXV.	Schematic flow diagram, lubricating-oil manufacture from ethane cracking, Ammoniakwerke, Merseburg, Leuna works 60
128	XXVI.	Flow diagram, section 1, Leuna works, lubricating oil from ethylene, ethane cracking, design data for one unit, four units parallel 60
129	XXVII.	Flow diagram, section 2, Leuna works, lubricating oil from ethylene, ethylene purification 60
129	XXVIII.	Flow diagram, section 3, Leuna works, Linde low-temperature fractionating plant 60
130	XXIX.	Flow diagram, section 4, Leuna works, lubricating oil from ethylene, catalyst separation and final oil treating 60
130	XXX.	OXO plant 88
133	XXXI.	OXO plant batch filter (filling position) 90
135	XXXIA.	Details of OXO plant ceramic filter tubes 90
	XXXIB.	OXO converters 92
	XXXII.	Synol process 92
	XXXIII.	Synol process, alternative method 94
	XXXIV.	Proposed design for Synol reactor 94
	XXXV.	Synol process; catalyst reduction 96

ILLUSTRATIONS (Continued)

<u>Fig.</u>		<u>Following page</u>
XXXVI.	Synol process, boric acid plant	96
XXXVII.	Methylamine manufacture	100
XXXVIII.	Toluol synthesis, capacity 5,000 tne. per month, Waldenburg, Schlesien	108
XXXIX.	Production of aviation petrol and fuel oil from Upper Silesian coal; flow sheet of Blechhammer hydrogenation plant	124
XL.	Aviation gasoline production	132
XLI.	Aviation gasoline production, normal program	132
XLII.	Aviation gasoline production, maximum program	132
XLIII.	Achieved and planned oil production (subdivided according to origin)	132
XLIV.	Achieved and planned oil production (subdivided according to products)	132
XLV.	Achieved and planned oil production from hydrogenation plants (including hydro-forming DHD, Di 1,000, and S. S. oil)	132
XLVI.	Achieved oil production (subdivided according to origin)	132
XLVII.	Achieved oil production (subdivided according to products)	132
XLVIII.	Achieved oil production from hydrogenation plants..	132

Editor's Note: In preparing the following report, many of the sections have had to be written on the basis of notes supplied by two or three investigators. Although in these cases the names of the people supplying the basic notes have been put down as "authors" of the section, it has not, in every case, been possible for them to see and approve the final documents. Every effort has, however, been made to include all the information obtained and to keep as closely as possible to the tone of the individual notes.

I.C.
and D
Holl
infor
and M
Schro
1945,
(abst

Gas P.

made
ators
rest
slag
the B
the f

Type

"Pint
"Pint

Winkl

Slagg

Pinte

Water

built
of th
Gener
and c
as a
diffi
gener
each