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SYNTHETIC LUBRICATING OIL PRODUCTION IN FRANCE

Reported By

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PERSONNEL OF INSPECTION TEAM

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Synthetic Lubricating Oil Production in France

Introduction

The manufacture of synthetic lube oils is a subject of vital interest at this time to the U.S.Navy. Not only are the processes themselves important, but the quality and usefulness of the products obtained are subjects which call for careful study. It has been known for some time that the Germans have been using synthetic lubricants for all types of service since the outbreak of war. Some details of their work are now available here in France. In order to equip the Navy with a background on this subject, two field trips were made to study French plants producing synthetic lubricating oils; the first to the Kuhlmann Co. at Harnes (Lille) and the second to the Standard Kuhlmann Plant at Lestaque (Marseille). The former is only a pilot plant but operates on a process developed in Germany. The latter produces 25 tons of products per day and uses a process developed especially by the French which is probable now well known at this time outside of their country. Both processes are also interesting in that they use as a basic raw material the gas oil made by the Fischer-Tropsch process. Should a plant using this process be built in the U.S., the offtake could be used to make lubricants, if these products ever come into demand.

Description of Process

(a) Standard Kuhlmann - Lestaque.

This process uses Fischer-Tropsch gas oil, benzol, and dichlorethane, as raw materials. To make one ton of finished lubes, 600 kg of gas oil are required, 600 kg of benzol and 160 kg of dichlorethane. The gas oil should be just as paraffinic as possible - i.e. the Fischer-Tropsch process should be carried out with a H_2/CO ratio of 2/1 and the temperature of the reactors held down to 190°C. in order to produce a few olefines and as many paraffines as possible. The end point of its distillation should not be above 350°C. The benzol used is not a pure compound but contains some toluene and a little Xylens, its end point should not be over 150°C.

The gas oil is first chlorinated in 9 lead lined tanks arranged in series. The flow is countercurrent, that is the gas oil enters the first tank and is contacted by chlorine which has already passed through the other 8 tanks. Raw chlorine enters the last tank into which the gas oil flows. This reaction is slightly exothermic and the heat of reaction is removed by water cooled coils. No mechanical agitation is needed, as the chlorine supplies sufficient agitation in bubbling through the gas oil. The temperature of the reaction is approximately 90-100°C. Chlorine and hydrochloric acid leaving the last tank are passed through a conventional gas oil absorption unit to remove any valuable gas oil entrainment. The hydrochloric acid is used at this plant for other chemical processes.

The benzol and dichlorethane are mixed in the presence of Aluminum chloride in a special, mechanically agitated tank. The benzol enters at one end of the tank while the dichlorethane is injected through the sides. The temperature is maintained at 70°C. The tank is divided into three sections, each having an agitator and dichlorethane injection line.

In a similar tank, having 6 instead of 3 compartments, the chlorinated gas oil and benzol-dichlorethane are mixed also in the presence of aluminum chloride, the benzol-dichlorethane enters the end of the drum, while the gas oil is injecte through the side. The temperature is held at 70°C.

The reaction is terminated in a third such tank. No side injection occurs but the temperature is raised to 110°C. Following this, no chemical changes or condensation are thought to take place. The total time in passing through the three tanks is 6 hours. The total quantity of aluminum chloride used is 10% by weight.

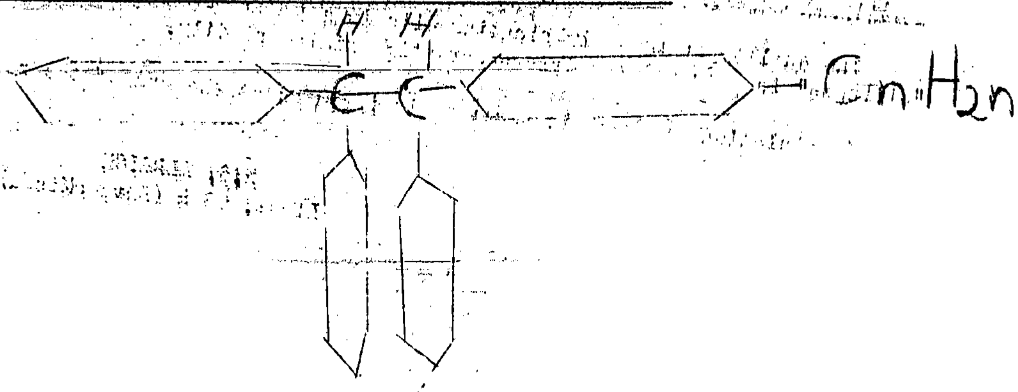
The liquid product is next put through a stripping tower with open steam where the uncombined benzol is removed. This is used over again. The stripped oil is now sent to a horizontal settling drum where the products settle into two layers. The upper is decanted as the high grade product, while the lower containing sludge, is of inferior quality. The aluminate is washed from the sludge by water. In order to lower its viscosity the washed polymer is blended with gas oil. No further treatment is required. This oil is used for general machine lubrication where a high grade quality is not needed.

The upper layer, after decantation, is given a conventional clay treatment. It is then sent to a pipe still with 2 side streams. The overhead product is gas oil which is sent back for reprocessing. The upper side stream is transformer oil. The bottoms product is very high grade steam cylinder oil which can be used with superheat temperatures up to 350°C. The present volumetric break down of these products is:- recycle gas oil 20%, transformer oil 30%, turbine oil 30%, and steam cylinder oil 20%.

Quality of Products

As already mentioned, the quality of the upper layer is extremely good. The viscosity index is approximately 100% for the lubricating oils. This is due to its unusual chemical makeup; as the oil consists of a polybenzene with a long saturated paraffine chain added on. The oil then is roughly half aromatic and half paraffinic giving it both a high viscosity index and a low pour point. A tabulation of these inspections is included at the end of the report.

A typical structure of this mollecular arrangement is:



Description of Process
(b) Kuhlmann Co. -Harnes.

This process starts with the gasoline product from the Fischer-Tropsch process. Under normal conditions, this product will contain 50% alphatic paraffines and 50% olefines. The olefine content on the gasoline can be increase in the Fischer-Tropsch reaction by lowering the normal H_2/CO ration from 2/1 to 1/1 and also raising the temperature.

The gasoline is mixed with 3% aluminum chloride and placed in a revolving cylindrical drum with steel balls in the bottom to increase contact surface. This reaction takes place at room temperature and requires 5 hours.

At the end of the reaction two layers are formed, the lower a heavy polymer chemically bound with aluminum chloride and an upper layer, of paraffinic gasoline and free polymer. These are separated in a decanter. The heavy tar is treated with N_aOH and washed with water, giving an aluminate, N_aCL and a lube oil produced.

The free polymer and gasoline are allowed to stand for 4-5 days in order to completely separate. The light or upper cut is steam stripped to remove the gasoline which is used as motor fuel. The gas oil and lube remaining after steam stripping are clay treated and filtered. The resulting oils are a gas oil, a light lube suitable for cold service and a heavy lube for ordinary lubrication.

At present, this heavy cut is being hydrogenated for special use. This is being done at 100 atmospheres pressure and 200°C with standard Fischer Tropsch Catalyst (Cobald-Thorium-Magnesium-Kieselguhr). This produces a substance similar to vaseline which is being used in the cosmetic industry.

This plant is only producing 1000 litres/day but if needed becomes urgent it can process 2000 litres/day.

Quality of Products

The quality of these lubricating oils is not exceptionally good. The "oiliness" is poor and the oils apparently oxidize readily.

A tabulation of these inspections is included at the end of the report.

24th March 1945

R.C. ALDRICH
Lieut. USNR (NavTecMisEu)

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Harnos

ANALYSIS - SYNTHETIC LUBRICATING OILS

KUHLMANN COMPANY

	Lubo F-2	Lube S-3	Lube 5/6	Lube 25/30
APL Grav.	38.0-39.0	37.0-38.0	35.0-37.0	33.0-35.0
Viscosity, SSU				
at 68°F	209-281	-	-	-
at 122°F	65-83	102-121	174-210	880-1050
at 212°F	35-39	39-43	50-54	103-121
V.I.	-70 (approx)	-70	85	82
Flash, °F (Luchaire)	275	293	320	-
Flash, °F (Open cup)	293	311	-	446
Fire, °F (Open cup)	347	356	374	518
*Neut.No.	0.2-0.4	0.1-0.3	0.1-0.2	0.1
*Pour Point, °F	-22	-13	-5	23
Carbon Residue, %	.01	.01	.02	.05
Ash, %	.02	.02	.05	.05
Use	Refrigerating machines	High speed & transmission	Engines & transformers	Engines & Compressors

*Methods possibly differ from A.S.T.M.

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INSPECTION OF SYNTHETIC LUBE OILS

KUHLMANN COMPANY

	Lube F-2	Lube S-3	Lube 5/6	Lube 25/30
Uses	Refrigerating machines	High speed transmission	Motors & transformers	Compressors & motors
Density 0 15° C	0.83/0.835	0.835/0.84	0,84/0.835	0.85/0.86
Engler Viscosity @ 20° C	6 to 8	11 to 13	-	[illegible]
50° C	2 to 2.5	3 to 3.5	5 to 6	25 to 30
100° C	1.2-1.3	1.3-1.4	1.6-1.7	3 to 3.5
Flash - Luchaire	135° C	145° C	160° C	[illegible]
Flash - Open cup	145° C	155° C	-	230° C
Fire Point	175° C	180° C	190° C	270° C
Acidity	0.2/0.4	0.1/0.3	0.1/0.2	0.1
Parer Point	-30/-35 C	-25/-20°C	-15/-10°C	-5° C
Consadson Carbon	0.01	0.01	0.02	0.05
Ash Content	0.02	0.02	0.05	0.05

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INSPECTION OF SYNTHETIC OILS - STANDARD KUHLMANN

DESIGNATION	API GRAVITY	VISCOSITY 122°F	VISCOSITY 212°F	VISCOSITY 392°	V.I.	FLASH °F	HARD ASPHALT c/δ METH. SNCF SPEC. 179	POUR POINT °F
Spindle	15.9	42 -73				284		27
Transformer A	17.4	84 max.				311		23
Transformer B	17.4	84 max.				311		-4
Turbine	15.9	95 - 110				356		-10
Motor 1	15.9	281 max.			>70	419		10
Motor 2	14.4	563 max.			>70	446		5
CYLINDER OILS								
Saturated Steam	12.1		96 - 121			392	<0.6	
Low Superheat	12.1	1370-2300			>60	518	<0.4	27
High Superheat	12.1		350 - 480		>80	572		43
Very High Superheat	12.1		350 - 480		>80	572		43
Heavy Machine Oil	—	84				271		

NOTE: THESE SPECIFICATIONS ARE THE MINIMUM.

IN GENERAL THE STANDARD KUHLMANN CAN PRODUCE BETTER THAN THE ABOVE