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The destearination of Menhaden fish oil by use of a votator and an auxiliary plate and frame filter press.

Douglass White 1920-2013

University of Louisville

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UNIVERSITY OF LOUISVILLE

**THE DESTEARINATION OF MENHADEN FISH OIL BY USE OF A
VOTATOR AND AN AUXILIARY PLATE AND FRAME FILTER PRESS**

A Thesis

**Submitted to the Faculty
of the Graduate School
of the University of Louisville
in Partial Fulfillment
of the Requirements
for the Degree of**

MASTER OF CHEMICAL ENGINEERING

Department of Chemical Engineering

Douglass White

June 1947

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**THE DESTEARINATION OF KETCHIKAN FISH OIL BY USE OF A
VOTATOR AND AN AUXILIARY PLATE AND FRAME FILTER PRESS**

Douglass White

Approved by the Examining Committee

Director R. C. Ernst

G. C. Williams

Guy Stevenson

June 1947

The author wishes to acknowledge
the kind assistance and helpful guidance
of Dr. R.C. Ernst
who directed this Research

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ABSTRACT

The separation of stearine from the oil of the Menhaden fish was attempted in a continuous operation by the utilization of a Votator. The Votator consisted of a horizontal tube 3 inches in diameter and 12 inches in length, within a larger tube. The annular space served as the expansion area for an ammonia refrigeration system. Within the 3 inch tube, a $2\frac{1}{2}$ inch shaft equipped with two $12 \times 1 \times 1/8$ inch scrapers rotated, removing crystallized stearine from the inner walls of the tube during passage of the oil.

Two methods of preparing the oil were presented. In the first, the oil was chilled from an original temperature of 190°F to 70°F in one pass through the Votator, allowed to stand 24 hours for crystal growth, passed through the Votator again for cooling to 50°F , and filtered in a plate and frame press. Sufficient stearine was removed by this procedure to yield an oil having iodine numbers from 160 to 195 and a cloud point as low as 28°F .

In the second method, two passes through the Votator were made, but a filter aid, Celite 502 was added to the oil. Also, the filter cloths were precoated with a layer of Celite 502. This increased capacity of filtration and made removal of the stearine from the filter cloths easier. Oil cleared of stearine by this method stood a cold test at 28°F for 5 hours without clouding.

The Votator quickly produces fine crystals which are difficult to filter in present standard equipment such as a

plate and frame filter press. If some method of mechanically separating the fine crystals can be devised, the use of the Votator should prove satisfactory for destearinizing Menhaden fish oil.

INTRODUCTION

The research described in this thesis was that of developing a rapid method for the separation of stearine from the oil of the Menhaden fish. The present procedure of separation by filtration was not to be changed. However, the method of preparing the hot oil for the filtration was to be shortened.

At present, large batches of the hot oil are slowly cooled in big wooden tanks for minimum periods of two to three weeks. This slow cooling allows the stearine to crystallize into a filterable form. During this time the oil occupies considerable storage space. If it were possible to effect the separation of the saturated stearine from the unsaturated oil immediately after it is extracted from the fish, the holding time would be shortened and the storage space occupied by the oil could be minimized.

It was believed that continuous cooling and crystallization of the stearine into a filterable state could be effected successfully by means of a Vetator, product of the Girdler Corporation, Louisville, Kentucky. A laboratory model was provided for the investigation. In order to duplicate present filtration facilities, a small 12 x 12 x 1 inch plate and frame filter press was used for the separation of the Vetator treated stearine.

HISTORICAL

At present, stearine is removed from Menhaden oil by "cold clearing" (1). "Cold clearing" is a physical process whereby a fatty oil may be separated into solid and liquid components by cooling. The saturated stearine has a much higher freezing point than the unsaturated oil, so that it crystallizes and tends to settle out with gradual reduction in temperature. To effect the complete separation on a commercial scale, two pieces of equipment are used, a cooling tank and a device for separating the stearine. The oil may be cooled by several methods. Brine may be circulated in pipes which are inside an insulated tank or against its outer surface. Either method has the disadvantage of causing immediate solidification of the oil in contact with the cold surface, regardless of its melting point, with the loss of the more unsaturated oil. It is preferable to circulate brine, the temperature of which is continually decreasing from the temperature of the liquid oil to the final low temperature desired, at a rate such that there is never a large difference in temperature between it and the oil. Another arrangement is to have the tank in a cold room. Slow uniform cooling will then result because the heat loss of the oil is governed by heat transfer through the air. The temperature of the cooling coils and the size of the room and tank will govern the rate of cooling.

When the oil has been cooled sufficiently, it is transferred by pumping or by forcing with compressed air to a filter press which should be at the same temperature as the oil to

prevent any loss in filtering ability of the stearine either by softening through warming or by solidifying in the press through further cooling.

In the Menhaden operations, since the extraction of the oil is a seasonal operation, the oil is collected over an extended period of time in a large storage tank. In this tank it is allowed to cool to the temperature of the outside winter air. The oil is then pumped to a cold room where the stearine is crystallized by slowly cooling for 10 to 20 days, followed by filtering in a plate and frame filter press.

THEORETICAL

Menhaden fish oil is composed primarily of saturated and unsaturated straight chain fatty acids of glycerol. (2) The chains vary in length from 16 to 24 carbon atoms with as many as 4 double bonds occurring in some of the more unsaturated straight chains. The completely saturated oils are called stearine and solidify in the pure state at temperatures ranging from $130^{\circ} F$ to $150^{\circ} F$. In the presence of the other constituents of the oil, they solidify lower, from $50^{\circ} F$ to $90^{\circ} F$. The unsaturates do not become solid until cooled below $30^{\circ} F$, in pure state or in the oil. It is possible to separate the stearine using this one physical property.

Crystallization of the stearine from the Menhaden oil is a slow process if filterable crystals are to be attained. (1) The size of the stearine crystals and the amount of stearine crystallized vary with the speed of cooling. Fast cooling produces fine and hard to filter crystals. For example, if a sample of oil is cooled rapidly from $100^{\circ} F$ to $40^{\circ} F$, crystallization will occur suddenly and completely throughout the sample. At approximately $70^{\circ} F$, a mass of tiny crystals will appear. Near $50^{\circ} F$ the oil will thicken, and although it is kept at $40^{\circ} F$ for days, the tiny crystals will grow so slowly that no more stearine will appear to come out of solution. No crystals will settle to the bottom of the sample. On the other hand, if the oil is cooled 4° to $7^{\circ} F$ per day from $100^{\circ} F$, by the time $40^{\circ} F$ is reached, large flake-like crystals of stearine will have drifted to the bottom of the sample leaving clear

fluid oil above it.

The fine crystals formed by rapid cooling would be difficult to filter. The semi-solid oil and stearine mix would plug the pores of any filtering medium with the tiny mushy crystals of stearine. The flake-like crystals produced by slow cooling would filter easily. However, many days would be required with slow cooling, waiting for ideal conditions to be reached.

If these two methods of cooling could be combined to utilize less time and still obtain a filterable product, the problem of continuous cooling would be solved. It can be assumed that the slow cooling gradient is unnecessary in the higher temperature range because no stearine crystals would form here under any circumstances. It should be possible to cool the oil quickly from 190° F as it comes from the extractor, to 70° to 80° F. From this moderate temperature to filtration temperature at 50° F, some substitution for slow cooling must be provided to allow crystal growth without the interference caused by the oil thickening.

Because the crystals formed in the Vatater would not grow as large as those described above which were slow-cooled, the smaller mushy crystals would be expected to clog the pores of a filter cloth. In an attempt to minimize this effect, a filter aid, Celite 502, was incorporated in one of the final runs.

EXPERIMENTAL

EQUIPMENT

The main equipment used in this research consisted of an ammonia type cooling device called a Votator (3), and a plate and frame filter press; the latter was kept cool in an insulated cold room, also refrigerated by the ammonia system. The Votator was to cool the oil, crystallizing the saturated stearine out of the Menhaden oil. This stearine was then filtered from the clear unsaturated oil in the press. It was necessary to maintain the press at the same temperature as the cooled oil to prevent the crystals from melting.

Referring to Figure 1, the oil was pumped from a heating barrel, lower left, through the Votator to the filter press in the cold room. Both the Votator and the cold room were refrigerated by the same ammonia system, shown in the top part of the diagram.

The Votator unit, Figure 2, consisted of two main pieces of equipment, the Votator and a 1/2 inch gear pump. The Votator, a heat transfer apparatus, consisted of a 3 inch, horizontal, stainless steel tube, 12 inches long. Outside of the tube was a 7/8 inch annular space for the ammonia cooling agent. Surrounding this concentrically was 1 1/4 inches of cork insulation protected by a thin sheet of stainless steel sheeting. Inside the tube revolved a 2 1/4 inch shaft called the mutator, see Figure 3. Floating on this were two 1 x 12 x 1/8 inch stainless steel scrapers which were thrown against the side of the chamber by the centrifugal force of revolution.

FIG. 1 ARRANGEMENT OF EQUIPMENT

Figure 2. A Vetator (A larger commercial model)

FIG 3. DETAIL OF MUTATOR SHAFT SHOWING BLADES

SECTION A-A ENLARGED

FIG 4. DETAIL OF MUTATOR SCRAPER BLADE

see Figure 4. These blades removed the stearine crystals as they froze out on the chamber wall.

The mutator shaft was supported in a stainless steel, fish-oil lubricated hub at one end and by a ball bearing isolated with a neoprene seal near the other end. The outside end of the shaft was keyed to a pulley driven by a 3 HP Howell induction motor. The motor shaft was equipped with a Reeves variable speed drive for adjusting the RPM of the mutator.

The ammonia float valve chamber was above the Mutator. It was connected to the annular space by a liquid inlet line equipped with a valve, a gas return line equipped with a valve, and also by an equalizing line. The ammonia liquor level in the upper chamber was controlled by a float which activated a valve in the inlet liquid ammonia line and kept the storage container constantly half full of liquid ammonia. To indicate the liquor level, three tail pipes extended horizontally at different heights from one end of the chamber. These frosted up to the level of the ammonia and thus indicated the level. Pressure in the chamber and consequently in the annular space was controlled by a manual valve on the outlet gas return line to the compressor. The chamber was equipped with a pressure relief valve set at 125 psi. and a 30 inch vacuum to 150 psi. pressure gage.

The gear pump was a 1/2 inch Northern pump, No. 2264, driven by a Howell induction motor. This pump served to remove the oil from the barrel and send it through the Mutator.

Oil flow rates could be adjusted by means of a Reeves variable speed drive on the motor shaft.

In connection with the Votator, several pieces of auxiliary equipment were employed. The oil for the particular run was temporarily stored in an open end, 55 gallon barrel. This barrel was equipped with a "Lightnin" agitator and a 1/2 inch copper steam coil used for heating the oil. A 110° centigrade thermometer was inserted in the inlet line to the Votator.

On the outlet line from the Votator was a tee containing a thermowell, a vent, and the line to the filter press in the cold room. The thermowell contained a Fahrenheit, industrial thermometer for measuring the outlet temperature of the cooled oil. Under the vent was a tared bucket on 50 pound scales for measuring the oil rate of flow. The vent contained a gate valve. There was a gate valve in the line to the filter press. With these two valves, oil flow could be directed to the scale bucket or to the press. A 3/8 inch gear pump was stationed in the cold room to pump oil to the filter press. This pump served to prevent the filtering pressure from building up in the Votator tube as preliminary runs gave evidence that stearine crystals formed under pressure would occlude oil and be more difficult to filter. This was a Viking pump driven by a 1/4 HP Master motor. The pump was equipped with a by-pass.

The pump fed a plate and frame filter press. Each

frame was 12 by 12 by 1 inches, with 5 frames being employed in the experiments. 15 ounce oxford filter cloths from Smith Fish Products, Lewes, Delaware, were employed in the press. These cloths were the same as those used in the Menhaden industry.

The filter press and the small gear pump were operated in a 6 by 6 by 6 foot cold box which was insulated with 6 inches of powdered cork. The box was cooled by ammonia circulated through finned coils, located in the ceiling for natural convection. Ammonia was provided by a compressor for both the Votator and the cold box. It was a water cooled, Model DC-4 upright, creamery compressor, No. 3641.

A Sparkler filter was also piped to the small gear pump in the cold room and in parallel with the plate and frame press. It contained four 8 inch screens for the filtering plates. Filter cloths were used on these. Celite 502 was used as a filter aid in the oil and as a precoat on the filter cloths in one of the runs.

PROCEDURE

To accomplish a relatively rapid separation of the stearine from the unsaturated Menhaden oil and still provide crystals large enough to be filtered, two methods were devised.

The first method employed the Vetator with two passes of oil, the second pass coming after a waiting period of 24 hours at a temperature conducive to crystal growth. The first pass cooled the oil quickly from 190° F, the initial temperature, to 70° F where the growth of stearine crystals could take place without interference caused by the oil thickening. During the 24 hour period thousands of "mother" crystals formed. These were transformed with cooling and agitation by a second Vetator pass, into filterable state.

Because the filtering rates of these crystals were so low and the pressure on the press rose so rapidly, it was decided to incorporate a filter aid to attain greater filtering capacity through a longer period of filtration at constant rate. Celite 502 was incorporated, using similar operating conditions of the first method.

In both runs, fresh Menhaden oil was transferred from an air tight container to the open end barrel equipped with steam coil and "Lightnin" agitator. The steam was turned on the coil and the oil agitated until the temperature reached 190° F. The compressor was then started and the inlet ammonia valve was opened to allow the float chamber to fill to the half way mark. The 1/2 inch gear pump was started at maximum rate, 315 pounds

per hour, to pump the oil through the Votator and out the vent to the bucket on the scales. The mutator agitation was started at maximum rate, 870 RPM. The inlet and outlet ammonia valves to the annular cooling section were opened wide. The valve on the gas return to the compressor was opened until the pressure in the Votator ammonia system was about 50 psi. This pressure was then adjusted to give an outlet oil temperature of 70° F. When this temperature became constant the oil was charged into the empty container, to be kept air tight for 24 hours at 70° F, the room temperature.

The next day, the compressor was again started and ammonia was allowed to flow into the cooling coils of the cold room at such a rate that it vaporized to maintain 15 psi. gage pressure at the compressor suction. This was controlled by the expansion valve on the liquid ammonia line to the cold box. While the box was cooling down, the fresh oil at room temperature which had stood for 24 hours was transferred from an air tight container to the open end barrel. The inlet valve to the Votator was again opened to allow the ammonia float chamber to fill to the half way mark. When the cold room had reached a temperature of 40° F, cooling was slowed by partially closing the inlet valve, thus reducing the amount of ammonia circulating through the box. Temperature was maintained at 40° F for 30 minutes to allow the equipment to cool.

The oil was then agitated and the 1/2 inch gear pump started to pump the oil through the Votator and out the vent to the

bucket on the scales. The line to the filter press was cut off. The rate was measured at minimum pump rate, 115 pounds per hour, as this was the maximum capacity of the 3/8 inch pump in the cold room.

The mutator agitation was started at maximum rate, 870 RPM. The inlet and outlet ammonia valves to the annular cooling space were opened wide. The valve on the gas return to the compressor was opened until the pressure in the Votator ammonia system was about 65 psi. When the oil reached constant temperature, the pressure was adjusted at this valve to give an outlet oil temperature of 55° F. When this temperature became constant and the oil rate had been measured, the valve on the oil line to the cold room was opened, the 3/8 inch gear pump was started, and the vent at the scale closed. This was the start of the timing. 3½ minutes later, clear oil began dripping at the filter plate drains.

The pressure on the inlet line to the filter was watched by the pump operator in the cold room. Whenever it rose above 75 psi., it was necessary to open the bypass around the pump to avoid damage to the filter press and cloths. As this bypass became opened further, it was necessary for the Votator operator to open the vent to the scale bucket to prevent pressure from building up on the Votator. When the small pump bypass was open wide and the pressure on the filter press still exceeded 75 psi., the run was concluded.

During the run a sample of the effluent of the Votator

and samples of the clear oil at filtering pressures of 5 psi., 15 psi., and 30 psi. were taken. Above 30 psi. the pressure approached 100 psi. so rapidly that no more samples were taken. The rate of filtration dropped off as the pressure mounted. The clear oil was collected in a tared container and weighed at the end of the run.

When the run was over, the inlet and outlet ammonia valves to the annular space were closed. As soon as the temperature of the outlet oil from the Votator warmed to 65° F the oil pump and the mutator were shut down. The filter press was opened, the oil collected and weighed, and the press was closed again. Gasoline was pumped through the Votator to the press and out the drains. This extracted the remaining oil from the cake and cleaned the equipment and lines. The cloths with the cake glued to them were removed, allowed to dry, and weighed. The stearine could only have been removed from the cloths by melting.

Iodine numbers and cloud points were run on the samples. Iodine numbers were run according to the standard Sij's method as directed by A.S.T.M. Standards (4). Comparative cloud point tests were conducted so that the oil cooled a maximum of 1° C per minute. The oil was placed in a test tube at 22° C and agitated slowly with a -30° to 60° C thermometer down to -2° C. Cooling was controlled by the addition of ice chips at intervals to a saturated salt solution surrounding the test tube.

Sample calculations and data sheets of these two methods appear in Appendix V. and VI.

For the second method final run, the oil was heated to 190° F as already described. While being heated, 3% by weight of Celite 502 was mixed into the hot oil with the "Lightnin" agitator. The oil was cooled through the Votator to 60° F and collected in an open end barrel. This oil was then held at 60° F in the cold room for two hours.

During this time, 2 gallons of 10% Celite 502 in mineral spirits were pumped through the clean filter press. This pre-coated 1/4 inch of pure filter aid cake on the cloths. The mineral spirits drained at the filter press outlets. After the two hour waiting period elapsed, 3% by weight of Celite 502 was again agitated into the oil. The oil was then passed through the Votator and cooled to 28° F and collected at the scale vent in the air tight container. The oil was held at 28° F in the cold room for 10 hours.

After 10 hours had elapsed, the oil was pumped through the precoated filter press with the small gear pump in the cold room. Filtering pressure was maintained at 40 to 60 psi. The rate of clear oil leaving the press was constant for the entire run. A sample was taken of the effluent of the press. The press was flushed with mineral spirits, after which the cake was easily removed from the cloths, leaving them clean. The oil was tested by being held at 28° F for 5 hours.

In connection with the study of preparing the oil for

filtration, observations were made of the overall heat transfer coefficients on the Votator at various operating conditions. These were calculated in Appendix I. Sample calculations of the overall heat transfer coefficients also appear in the Appendix. During these runs, operating experience was gained by collecting the cooled oil at the scale vent and centrifuging the samples to observe the amount of stearine which could be separated at various operating conditions. Preliminary runs using a Sparkler filter also are recorded in the Appendix.

RESULTS

The object of destearinizing Menhaden oil is to increase the percentage of unsaturation in the oil. This is measured by the Iodine number and is indicated comparatively by the cloud point test. These two tests were used in checking what had been accomplished in the process.

The properties of the untreated oil were an Iodine number of 159 and a cloud point of 62° F. Samples of the treated oil showed an increase in Iodine numbers ranging up to 183 to 195 and cloud point improvement to from 35° F down to 28° F.

When Celite 502 was incorporated as a filter aid, more finely divided crystals were removed so that the oil could be chilled to 28° F and held there for 5 hours with no clouding. Filtering rates were more constant when Celite 502 was used.

The overall heat transfer coefficients which were calculated in Appendix I were arranged according to similar operating conditions in Appendix II. A good correlation of coefficients was obtained for each of the four maximum and minimum conditions. These were averaged to provide workable overall coefficients for the design of larger units.

The values of U at minimum oil rate and minimum and maximum mutator RPM were 80 and 117 respectively. At maximum oil rate and minimum and maximum mutator RPM, the values of U were 127 and 160.

DISCUSSION

The selection of the two pass method for the separation of the stearine was made on the basis of two trial runs. Trial Run A was made with clear oil at 86° F which was cooled by a single passage through the Votator to 52° F where no stearine crystals appeared. Recycling the oil also produced no crystals. Previous experience indicated that clear oil which had been cooled to 50° F and then was allowed to stand for 24 hours would yield stearine crystals after a second pass through the Votator. Therefore, Trial Run B was made with clear oil at 90° F being cooled to 50° F and then allowed to stand at room temperature for more than 24 hours to insure crystal formation. It was then cooled to 50° F with the Votator to yield stearine crystals which were filtered in a Sparkler filter. Although the waiting period was more than 24 hours in Trial Run B, the same results had occurred in 24 hours. Also, the first pass cooled to 50° F, but since it warmed to room temperature, 70° F, where satisfactory crystal growth occurred, cooling to 70° F yields more Votator capacity than cooling to 50° F.

Because the pores of the filter cloth clogged in a comparatively short time with the crystals produced by this method, it was decided to incorporate a filter aid to promote steady rate filtration, while still using a basically similar two pass method. The presence of the filter aid, Celite 502, made possible a shorter overall time and increased filtration capacity. Pressures on the filter press averaged 40 to 60 psi. in a

press containing a 1/4 inch precoat of Celite 502 on the cloths. These cloths could be cleaned free of stearine much more readily than those collecting stearine which contained no filter aid.

The high iodine numbers on the filtered oil ranging from 183 to 195 and the cloud points down to 28° F as compared with the 159 iodine number and 59° F cloud point on the fresh oil indicate that the amount of stearine removed compares favorably with the Menhaden industry (5).

The overall heat transfer coefficients were calculated with the assumption that the heat of crystallization of stearine was almost negligible. This was confirmed by a cooling curve test which showed a constant rate of cooling from clear to solid product. This curve, Figure 5, appears in the Appendix. The overall coefficients were classified according to operating conditions. Since no definite trend of flow such as streamline flow or turbulent flow could be estimated, this was the best comparison available. These classifications appear in Appendix II, where the coefficients were placed according to similar maximum and minimum operating conditions. For instance, those calculated at minimum oil rate and minimum motor RPM were thus placed together. The coefficients of each of the four maximum and minimum operating conditions were averaged to give a mean coefficient for each set of conditions.

CONCLUSION

From the data presented it is concluded that a Vetator can be utilized in a two pass operation to prepare the stearine in Menhaden oil in filterable condition. This means that a semi-continuous operation requiring from 16 to 24 hours could replace the present system of cooling which requires two to three weeks.

The new procedure would accomplish two main improvements, either in whole or in part. They are: (1) reduce the large amount of storage space occupied by the fish oil, (2) reduce tremendously the holding time of the oil so that it is processed on the day of extraction from the fish instead of weeks later.

Because of the low filtering rates and rapid pressure build-ups occurring in the first method with no filter aid, it is felt that the second method incorporating a filter aid would offer the better means of filtration of the two.

However, the Vetator quickly produces fine crystals which are difficult to separate by either means or with any standard filtering equipment now available. The proposed method looks promising in that whenever a means for separating the fine crystals of stearine can be devised, the use of the Vetator should prove satisfactory for destearinizing Menhaden oil.

The average heat transfer coefficients at various operating conditions provide data for the design of larger Vetators in the Menhaden field.

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APPENDIX

SAMPLE CALCULATION OF HEAT TRANSFER COEFFICIENTS

- U** is the overall heat transfer coefficient
Btu per hour x square foot x degree Fahrenheit
- A** is the area of heat transfer surface, inside area with
oil film controlling, (constant) 3 /12 or 0.785 sq. ft.
- Δt_m** is log mean overall temperature difference between
bulk temperatures of warmer and colder fluids. In Run 1,
oil is 84° and 40° F, ammonia is 12° F
- w** is mass rate of flow of oil
In Run 1, 315 pounds per hour
- c** is specific heat of Menhaden oil (5) (a constant prop-
erty), 0.454 Btu per pound x degree Fahrenheit
- t** is temperature differential of oil
In Run 1, (84° - 40°) is 44° F

$$(w) \times (c) \times (\Delta t) = (U) \times (A) \times (\Delta t_m) \quad (7)$$

$$315 \times 0.454 \times 44 = U \times 0.785 \times \frac{(84-12) - (40-12)}{2.3 \log(84-12)/(40-12)}$$

$$\text{Overall } U = 162 \text{ Btu per hr. x sq. ft. x F.}^\circ \text{ in Run 1}$$

APPENDIX I - CALCULATION OF OVERALL HEAT TRANSFER COEFFICIENTS

Run No.	TEMP. °F		Ammonia Press.	Ammonia Temp. (°S)	T ₂ -T ₁	Δt_m	Rate #/hr.	D	Motor HPH	% Stear. Centrif.
	Inlet Oil T ₁	Outlet Oil T ₂								
1	84	40	32	19	44	39.0	242	158	870	-
2-A	82	57	68	46	26	21.1	106	72	320	-
2-B	82	50	57	39	32	23.6	106	82	320	10.8
2-C	82	54	66	44	28	21.1	106	81	320	10.0
2-D	82	46	37	23	37	37.4	106	60	320	13.2
2-E	82	51	57	39	31	24.2	106	78	320	12.0
2-F	82	52	63	43	30	20.4	106	90	320	10.2
2-G	82	51	63	43	31	19.6	106	97	320	11.2
3-A	78	60	66	46	18	23.0	327	148	320	11.4
3-B	78	58	52	35	20	22.0	327	118	320	12.6
3-C	78	56	48	33	22	23.0	327	126	320	12.0
3-D	78	54	43	29	24	26.0	327	126	320	12.2
4-A	73	57	67	46	16	18.6	316	156	870	12.6
4-B	73	55	61	42	18	20.6	316	152	870	14.0
4-C	73	52	56	38	21	22.8	316	168	870	14.4
4-D	73	49	50	34	24	25.8	316	170	870	14.4
4-E	66	46	46	31	21	22.8	316	168	870	13.2
4-F	57	40	36	21	17	26.4	316	117	870	16.0
5-A	80	54	56	37	26	28.2	316	168	870	8.4
5-B	80	48	46	30	32	31.2	316	187	870	10.4

Run No.	TEMP.		Ammonia Press.	Ammonia Temp.	T ₂ -T ₁	ΔT_m	Rate #/hr.	U	Mutator RPM	% Stear. Centrif.
	Inlet Oil T ₁	Outlet Oil T ₂								
5-C	80	45	40	26	36	33.6	315	190	870	10.4
5-D	80	42	35	20	36	37.8	315	183	870	11.6
5-E	80	40	25	12	40	45.0	315	162	870	11.0
6-A	84	62	54	37	22	34.8	315	115	320	6.0
6-B	82	60	52	35	22	34.8	315	115	320	7.6
6-C	82	58	50	34	24	34.6	315	126	320	7.0
6-D	82	56	45	30	26	37.6	315	126	320	8.0
6-E	82	54	42	28	28	38.2	315	134	320	8.4
6-F	82	52	38	24	30	41.0	315	134	320	8.0
6-G	82	50	30	17	32	47.1	315	124	320	7.8
7-A	172	58	56	40	114	57.3	115	133	870	2.8
7-B	173	60	64	44	113	54.2	115	139	870	4.2
7-C	172	55	40	36	113	58.3	115	129	870	4.6
7-D	170	53	35	31	117	76.0	115	102	870	3.2
7-E	170	49	31	18	121	76.0	115	106	870	2.4
8-A	113	46	30	17	67	56.0	125	121	870	0
8-B	111	55	38	24	56	54.0	125	105	870	0
8-C	109	53	35	22	56	54.0	125	105	870	0
8-D	109	49	33	20	60	53.5	125	113	870	0
9-A	77	58	40	36	19	30.4	315	114	320	0
9-B	95	49	25	11	46	58.0	315	144	320	2.0
9-C	90	51	25	11	39	57.4	315	124	870	3.6
9-D	104	64	40	26	40	55.5	315	131	870	-

APPENDIX II - AVERAGE OVERALL U AT VARIOUS OPERATING CONDITIONS

100-125 #/Hr.		300-325 #/Hr.		100-125 #/Hr.		300-325 #/Hr.	
Oil Rate		Oil Rate		Oil Rate		Oil Rate	
320		320		320		320	
Mutator RPM		Mutator RPM		Mutator RPM		Mutator RPM	
Run	U	Run	U	Run	U	Run	U
2-A	72	3-A	148	7-A	133	4-A	155
2-B	82	3-B	118	7-B	139	4-B	152
2-C	81	3-C	126	7-C	129	4-C	168
2-D	80	3-D	126	7-D	102	4-D	170
2-E	78	6-A	115	7-E	106	4-E	168
2-F	90	6-B	115	8-A	121	4-F	117
2-G	97	6-C	126	8-B	105	5-A	168
		6-D	126	8-C	105	5-B	187
		6-E	134	8-D	113	5-C	190
		6-F	134			5-D	183
		6-G	124			5-E	162
		9-A	114			9-C	124
		9-B	144			9-D	151
Ave.	80	Ave.	127	Ave.	117	Ave.	160

APPENDIX III - TRIAL RUNS USING SPARKLER FILTER

Run No.	Inlet Oil Temp.	Outlet Oil Temp.	Oil Rate #/Hr.	Mutator Rate	Filter Inlet Press.	Time
A	86	50	325	870	-	0
	86	50	325	870	-	10
	72	49	325	870	-	20
B	68	53	125	320	0	2
	68	53	125	320	10	5
	68	52	125	320	20	15
	68	53	125	320	35	25
	68	53	125	320	60	35

APPENDIX IV - EXPERIMENTAL DATA RECORDED ON FINAL RUNS

Run No.	Time Min.	Inlet Oil Temp. °F	Outlet Oil Temp. °F	Ammonia Press.	Oil Rate #/Hr.	Motor RPM	Filter Inlet Pr. psi.	Samples
I	3.5	68	54	63	122	870	0	-
	5	68	54	63	122	870	10	-
	7	68	54	63	122	870	75	A
	10	68	54	63	122	870	95	B
	15	68	54	63	122	870	100	-
II	3.5	67	53	63	103	870	0	-
	5	67	53	63	103	870	5	5 psi.
	10	67	53	63	103	870	15	15 psi.
	11	67	53	63	103	870	30	30 psi.
	13	67	53	63	103	870	90	-
III	5	50	28	35	117	870	0	-
	10	50	28	35	117	870	40	-
	15	50	28	35	117	870	45	C
	20	51	28	35	117	870	49	-
	25	51	28	35	117	870	53	-
	30	51	28	35	117	870	59	-

APPENDIX V - SAMPLE CALCULATION OF IODINE NUMBER

Flask No.	2
Weight of oil	52.7345 gms.
	<u>52.6517 gms.</u>
Difference	0.0828 gms.
Time of Hance Sol'n	0 min.
Time of titration	30 min.
Titre	23.1 c.c.
Blank	36.6 c.c.
Normality $K_2S_2O_8$ Sol'n	0.095
Molecular weight of Iodine	126.9

Calculation

$$\frac{(36.6 - 23.1) \times (0.095) \times (126.9)}{(0.0828) \times (10)} = 197$$

APPENDIX VI - CLOUD POINT TEST SAMPLE DATA

Run II - 80 psi.

Time Min.	Temp. °C	Remarks
0	22.0	Oil Clear & Fluid
2	20.2	Oil Clear & Fluid
4	18.0	Oil Clear & Fluid
6	15.7	Oil Clear & Fluid
8	13.7	Oil Clear & Fluid
10	11.9	Oil Clear & Fluid
12	10.2	Oil Clear & Fluid
14	8.2	Oil Clear & Fluid
16	6.5	Oil Clear & Fluid
18	4.7	Oil Clear & Fluid
19	3.5	Oil Clear & Fluid
20	2.2	Oil Clear & Fluid
21	1.0	Oil Clear & Fluid
22	0.2	Oil Clear & Fluid
23	-0.7	Oil Clear & Fluid
24	-1.5	Oil Clear & Fluid
25	-2.0	Oil Clear, Less Fluid
26	-2.2	Oil Clouding
30	-2.2	(28° F) Oil Cloudy

APPENDIX VII - COOLING CURVE OF MEEHADEN OIL DATA

<u>Time</u> <u>Min.</u>	<u>Temp.</u> <u>°F</u>	<u>Time</u> <u>Min.</u>	<u>Temp.</u> <u>°F</u>
0	98.0	110	60.0
10	94.0	120	58.0
20	89.5	130	56.5
30	85.5	140	54.5
40	82.0	150	52.5
50	78.0	160	51.0
60	75.0	170	50.5
70	71.0	180	49.0
80	67.0	190	47.5
90	64.5	200	46.5
100	62.0

FIG 5. COOLING CURVE - MENHADEN OIL

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VITA

The Author, Douglass White, was born on December 21, 1920, in Fargo, North Dakota, the son of Joshua James and Laura Douglass White. He attended grade school, Junior High and Senior High School in Lower Merion Township, Ardmore, Pennsylvania.

He obtained a Bachelor of Science Degree in Chemical Engineering at Lafayette College, Easton, Pennsylvania, on May 15, 1942. Since graduation he has been employed by the E. I. duPont de Nemours Company in the Louisville Sceptrene Works.