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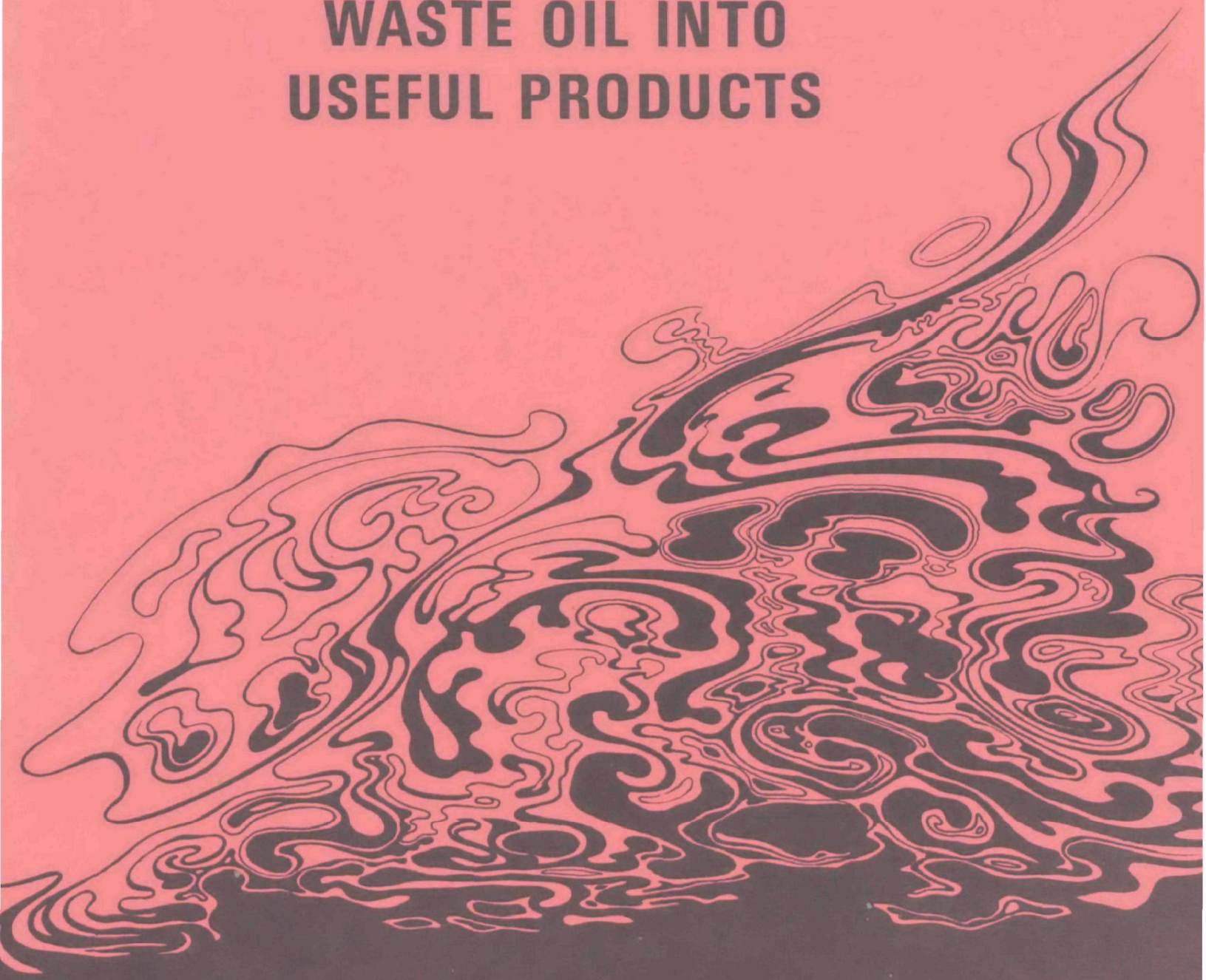
CONVERSION OF CRANKCASE WASTE OIL INTO USEFUL PRODUCTS



ENVIRONMENTAL PROTECTION AGENCY • WATER QUALITY OFFICE

WATER POLLUTION CONTROL RESEARCH SERIES

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CONVERSION OF CRANKCASE WASTE OIL INTO USEFUL PRODUCTS

by

National Oil Recovery Corporation
Hook Road and Commerce Street
Bayonne, New Jersey 07002

for the

WATER QUALITY OFFICE

ENVIRONMENTAL PROTECTION AGENCY

Project #15080 DBO

March 1971

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ABSTRACT

The project goal was to demonstrate a simplified technique for reprocessing spent automotive crankcase oils into useful petroleum products other than lube oils, without producing residues which cause water pollution.

To achieve the foregoing objectives, National Oil Recovery Corporation modified its entire plant system with special equipment and conducted laboratory and plant runs.

The objectives were substantially attained in that all the products from the vacuum distillation were sold as low sulfur heating fuels and as potential diesel fuel. Only the water in the fuel is not recovered.

Some technical work was done to upgrade the refinery products to obtain a higher product realization.

This report was submitted in fulfillment of Project Number 15080 DBO, under the (partial) sponsorship of the Water Ouality Office, Environmental Protection Agency.

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SECTION I

CONCLUSIONS

1. The composition of waste crankcase oil has significantly changed during recent years, thereby presenting greater problems to the present re-refining industry.

2. It is evident from the operational experience of existing acid/clay treatment plants that current techniques for reprocessing are uneconomical and the by-products of that process are environmental pollutants.

3. National Oil Recovery Corporation's experience with its modified plant and equipment proved that its process was a simplified technique for reprocessing waste crankcase oil into useful petroleum products without producing residues which cause water pollution.

4. National Oil Recovery Corporation's tests indicated disastrous results to burning equipment and to the environment if waste crankcase oils were burned directly or in combination with virgin oil.

5. National Oil Recovery Corporation's technical work to date has been directed toward obtaining a higher product realization and has shown that this goal is attainable.

6. The collection of waste crankcase oil should be systematic and should be monitored to assure that the collected waste oil is dispensed to a non-polluting refining facility.

SECTION II

RECOMMENDATIONS

It is recommended that National Oil Recovery Corporation demonstrate a revised flow sheet operation that will yield the following results:

1. Improve the quality of side-stream products.
   a. Modify distillation temperatures
   b. Conduct field tests with products
   c. Upgrade products for use as chemical feed stocks
   d. Simplify flow sheets

2. Modify unit processes to minimize effluent volumes.
   a. Replace steam jets with mechanical equipment
   b. Minimize bottoms volume
   c. Incinerate bottoms

3. Show optimum flow sheet conditions to be used for projected packaged refinery units.

4. Develop new products with greater returns. This will encourage re-refiners to consider necessary process modifications. Products should be use-tested by an accepted petroleum oriented testing laboratory to accumulate testimonial data for market acceptance of a "special fuel" and "special lube."

SECTION III

INTRODUCTION

Our country is faced with the enormous problem of cleaning the environment. One of the most destructive pollutants is automotive crankcase oil, of which one billion gallons are dumped annually into rivers, harbors, sewers, land dumps, etc.

According to the American Petroleum Institute,(1) approximately 2.5 billion gallons each of industrial and automotive lubricating oils are used annually in the United States. Approximately 1.25 billion gallons are drained and replaced with new or reprocessed oil. A significant quantity of industrial lubricating oils are collected and reprocessed and sold back to the original industrial or automotive users. However, automotive crankcase drainings must be collected from several hundred thousand service stations throughout the country.(2) The drainings are collected by thousands of relatively small independent collectors who traverse cities in a haphazard manner, competing with each other for the waste oil and for the cheapest way to get rid of their 'load.' If a receiver, such as a re-refiner or road oil user, is located within an economically geographical range, and if the re-refiner is willing to pay the collector more for his load of waste oil than it costs him to deliver it, the collector may deliver his load for some subsequent reprocessing and reuse. However, if any of these facets of the system are not favorable, the collector may dispose of his charge in sewers, onto empty fields, directly into rivers and streams, or he may sell it as a substitute for #6 fuel oil to an unsuspecting user who will pollute the air and ruin his burning equipment.

To help remedy that condition, the Environmental Protection Agency awarded National Oil Recovery Corporation a research and development grant to demonstrate a simplified technique for recycling crankcase waste oil into useful petroleum products without causing pollution.

The grant application was submitted in March 1968, but a grant was not received until January,1969. The original grant request was developed on the basis of the quality of automotive crankcase waste oils received during 1965-67. Crankcase waste oil is a difficult charge stock. It varies in that it contains different proportions of water, solids, additives, decomposed additives, road dust, gasoline, gasoline additives, some spent automotive transmission oil with additives, spent and unspent products of fuel and lube combustion, etc., etc. In the industrial New Jersey area, great care must be exercised to avoid taking in spent cutting oils, synthetic excessive phosphorous, etc. Each feed tank is a challenge.

The P&I of the previous plant, with proposed modifications to the original process, apparatus and instrumentation, were detailed in accordance with crankcase waste oil composition prevalent during 1965-67, and pollution controls enforced during that time. Meanwhile, the composition of crankcase waste oil has significantly changed. Automobile manufacturers have greatly increased the mileage and time intervals between recommended oil changes. The additive content in motor oil has been considerably increased and markedly changed; multi-grade motor oils with much higher additive contents are in much wider usage. Increasingly, present motor oil is made up of relatively low viscosity neutral oils into which are compounded ever higher percentages of additives containing more metals with dispersants, detergents and solids. Present waste crankcase oils delivered to National Oil Recovery Corporation contain more and more spent and broken down additives, sludge, and tar-forming materials from piston 'blow-by,' generating problems in the processing equipment and the products produced under the Process and Instrumentation Flow Diagram presented in the grant application. Greater pyrolytic polymerization was experienced during processing. All this, in addition to the special emphasis given to pollution control requirements, caused a substantially greater percentage of down time with the available equipment and necessitated numerous modifications to the piping and process equipment -- as the problems appeared -- plus a decrease in operating temperatures.

The project goal was to demonstrate a simplified technique for reprocessing spent automotive crankcase oils into useful petroleum products, without producing residues which cause water polltuion. Products produced would be suitable either as heating fuels, or as diesel fuels.

The foregoing objectives have been substantially attained in that all of the products resulting from the vacuum distillation of the waste crankcase oils are being sold as lube stocks to compounders, as well as for a low sulphur heating fuel market. Only the water in the feed stock is not recovered.

Some technical work has also been done to upgrade the sidestream and bottoms products to obtain a higher realization than the dark lube stocks and low sulphur fuels, and there is reason to believe this can be done. However, additional work is required to confirm the results obtained thus far.

In order that the general public avail themselves of the design criteria and operating data, the flow plan attached hereto presents the significant operating conditions, a yield balance and information on operating controls needed by anyone wishing to operate a similar process. The type of equipment used is designated schematically. (See following page for flow plan)

A detailed operating manual presenting start-up, shut-down and emergency procedures has also been prepared and is included in the appendices of this publication.

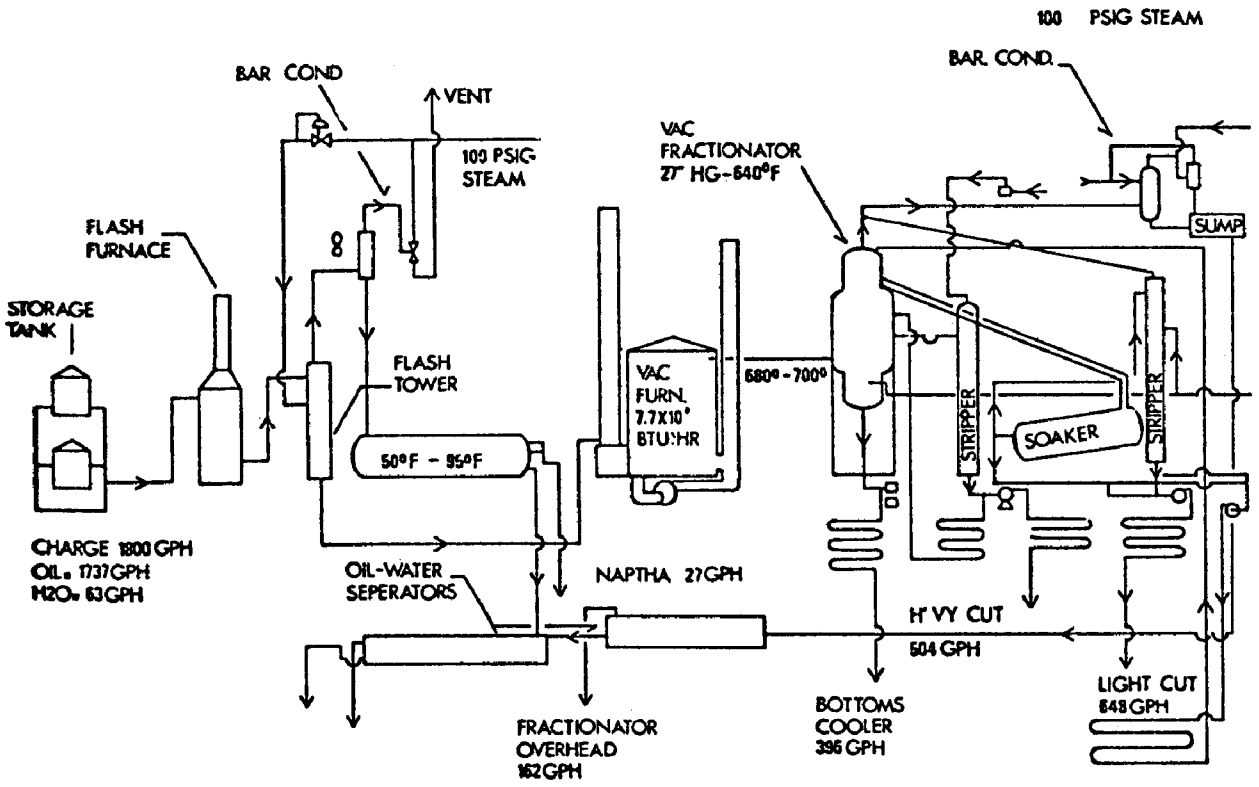

Figure 1.

SECTION IV

SUMMARY OF REFINERY RUNS #1-11 AND THEIR SIGNIFICANCE

Run #1 - January 15-20, 1970

This run, with the rather complete testing of products by E. W. Saybolt Co., showed that the results of operations in 1966-67 could be obtained in January, 1970, with some product improvement. The bottoms did not significantly change. Ash content and the color of both light and heavy sidestreams did improve slightly, in spite of increased additive content in the used crankcase oil charge in 1970, compared with 1966-67 (see Table I, page). The run demonstrated that operation could be maintained during very cold weather, despite malfunction of instruments, and difficulties with auxiliaries: boilers, oil heater burners, pumps, and electrical curcuits and apparatus. We stopped the run because of a sudden and unexplained increase in pressure at the inlet of the Fractionating Furnace. After shutting down and dismantling the equipment, a large amount of coke was observed in the furnace tubes. From the rapid rate of pressure buildup, it was deduced that a slug of oil containing undesirable polymers that are pyrolytically resynthesized to high molecular polymers produced heavy deposits inside furnace tubes, causing scaling on all heating surfaces.

An attempt at a run was made on February 28, 1970, however, hard porcelain Raschig ring fragments fell from the top of the flash tower and jammed the pump and control valves several times. The run was abandoned, but the experience later resulted in removal of the Raschig rings and installation of a cyclone separator in the top of the flash tower, with improved operation.

Run #2 - February 28 - March 1, 1970

NORCO placed a screen in the suction of the Fractionator feed pump and made a run of three days. The screen accumulated more porcelain fragments and considerable semi-solid material from the oil which was subsequently examined and proved to be organic metallic compounds that precipitated or coated the screen. We had difficulty controlling the Fractionator top temperature because of inadequate controls. Excessive amounts of Fractionator overhead oil went into the Kill van Kull. The Army Corps of Engineers, Coast Guard and Federal Water Quality Administration forced a shutdown until adequate oil-water separators could be designed, fabricated and installed.

Run #3 - April 21-22, 1970

NORCO installed oil-water separators, revised instrumentation to

control the top of the Fractionator and intermediate temperatures, and a new pump with piping connected to bypass the Vacuum Flasher, located at the South Feed Tank suction lines to feed unheated oil directly to the unit. NORCO started and operated the unit satisfactorily for about 36 hours. The oil-water separators, pumps, piping and revised instrumentation for control of Fractionator Top Temperature operated satisfactorily. The run was stopped because of plugging of the light product reflux cooling coil lines and control valve. The material fouling and plugging the cooling coil line was a dark resinous sticky and viscous material with a sharp acrid odor.

A material of this nature had been observed at times in various runs during the period 1965-67, in the light product section of the Fractionator, but it had not then plugged the cooler and lines and forced a shutdown, such as occurred during run #3. It was observed to separate out of the light and heavy products; and when bottled, a discrete floc was noted that gradually settled and accumulated as a sticky material that adhered firmly to the bottom of the bottles. After settlement of the floc, the oil became noticeably lighter in color. The floc was analyzed to be a metallic phosphate.

During this run, we pumped feed from the large cold feed tank, directly to the Flash Furnace and then into the Fractionating Furnace and directly into the Fractionator. The pressure at the inlet of the Fractionator Furnace was rather high, as expected, 50 psig instead of 10 psig. However, the Vacuum Fractionator functioned well and produced satisfactory results. The operation demonstrated clearly the feasibility of simplifying the plant in accordance with flow sheet #3, submitted with the original proposal. The run was terminated because of stoppage of flow of light reflux resulting from an accumulation of heavy viscous material at the control and bypass valves. However, no trouble was experienced because of Raschig rings or semi-solid material derived from the charge stock. The high pressure drop did limit the feed rate and, of course, increased the unit cost of processing.

Run #4 - May 7-11, 1970

After accumulating and analyzing data collected during Run #3, the Raschig rings, along with liquid distributing and collection apparatus, and supporting structure were removed and replaced with a cyclone arrangement positioned at the top of the Vacuum Flasher. A large screen was installed in the bottom of the Vacuum Flasher above the suction line to the Fractionator Feed Pump to avoid possible trouble with gummy or leathery semi-solid material that might foul the positive displacement gear pump, which is used to transfer the feed from the Vacuum Flasher to the Fractionator

Furnace and Fractionator. We started the unit, but stopped the run after four days of improved operation because of jamming of the gears in the Fractionator Furnace and a boiler. The two pumps stopped almost simultaneously.

The fuel being pumped was largely made up of recently distilled light product. The material jamming the gears of the pumps was a very tenacious, tough, thick layer of film of dark resinous material with a dark red, almost black color and a sharp acrid odor, which characterizes the light product distilled from crankcase oil.

With Run #4, practical commercial operation is believed to have started. Yield data, etc. for run #4 is reported at the end of this summary. After run #4, certain conclusions were drawn on the basis of observations as noted below.

During 1965-67, the overhead, light and heavy distilled products were darker than expected from experience with similar materials distilled in ordinary crude petroleum refineries. This was thought to be caused by excessive entertainment of liquid and solid particles in the vapor passing through the Fractionator. A demister blanket was installed, with a wash oil distributor, but the blanket could not be kept clear. It quickly fouled up. Improvements in the flash zone were made, with some improvement in color. The improvement in color was not as great as expected from prior petroleum refining experience.

Under the present program, acting in agreement with the FWQA thinking, improved transfer line equipment, flash zone entry arrangements and secondary cyclones with steam spray nozzle injection of wash oil with arrangements for recycling the wash oil were installed to provide for stoppage of entrainment and improvement of color and reduction of floc in the distilled products.

The color and floc settlement from samples from runs #1 through #4 inclusive did <u>not</u> improve as expected.

The color of gasoline distilled over the top of the Vacuum Flasher <u>after</u> installation of the cyclone arrangement was good, about what might be expected from petroleum refinery experience.

The color of the Fractionator overhead caught in the new oil-water separators was much darker than would be expected from petroleum refinery experience. Also this material darkened considerably and quickly in the oil-water separator.

The above observations led to the conclusion that the dark color noted in distilled products probably was <u>not</u> a result of entrainment and carry-over of liquid or solid matter in the vapor stream.

It was concluded that the dark color noted in the distilled products and the dark, resinous, sticky semi-solid material might be derived from some material passing in the vapor phase through the Fractionator, but polymerizing and condensing as polymer, particularly in the light product section of the Vacuum Fractionator.

After conferences with the Federal Water Quality Administration Project Officer, relative to the observations and conclusions, the Project Officer arranged a consultation with Esso Research and Engineering Co. scientists concerning the problems of dark color of distilled products. Also discussed was the problem of drop out of dark, resinous semi-solid material from the light product as well as the heavy product and the fouling of the light product reflux cooler and control valves. Furthermore, the relatively quick fouling of the Fractionator Furnace tubes was discussed with the Esso scientists. The Esso personnel told us that the material causing dark color, etc. probably did pass up the Vacuum Fractionator in the vapor phase. They suggested trial usage of an inhibitor product which they sell.

Preparation of Run #5

We promptly began injection of the inhibitor according to their recommendations, with hastily assembled blow cases, into the crankcase oil charge and the two sidestreams. Control of the injection rate and steady application is difficult to achieve with makeshift injectors. Orders for positive displacement, adjustable flow injectors were placed. Results so far, in spite of primitive injection apparatus, are encouraging. The unit operated 7 days with an adequate flow of light product without significant indication of light reflux cooling and coil plugging.

The deposition of dark resinous materials from the samples of light product has been sharply reduced, and in some samples it stopped. In the samples were deposition is found, the material is softer and more fluid than before additive injection. Apparently, fouling of furnace tubes is significantly slowed by inhibitor injection.

Run #5 - May 21-28, 1970 (166 hours)

Recirculation of feed during startup and after upsets and stoppages was reduced to a minimum during this run. This procedure should reduce the formation of heavy insoluble polymers from V.I. additives, and the accompanying very rapid coking of furnace tubes and hot oil lines as noted during run #1. This procedure was also followed during run #4. During run #5 the cold feed was charged directly from the large south feed tank.

Several times during the run, the charge pump motor-heater "kicked out," as if the motor was suddenly overloaded. This may have been caused by the passage of slugs of viscous, insoluble in oil, polymer passing into the pump and greatly increasing, for a short time, the power required to run the pump. Minimum recirculation was practiced during run #4 and this accounts for the high percentage of bottoms made during that run. During run #5, the furnace outlet temperatures were quickly restored after each upset and recirculation of feed after an upset was reduced to a minimum. This procedure also markedly reduced the percentage of bottoms made.

During run #5 no tendency towards plugging of the light and heavy reflux and product coolers was noted. Some stream samples taken during this run produced no dark bottom settlings in the sample bottles. While others produced dark fluid bottom settlings with apparent viscosity not much greater than that of the oil, no hard sticky bottom settlings, usually observed during previous runs, were to be found. However, the coke deposit on the tubes in the Fractionator Furnace was found to be rather thick, as up to 5/8" was observed. The variation in the above findings might be caused by the known uneven injection of inhibitors or defective mixing of the feed in the feed tank.

The fouling of the Fractionator Furnace tubes did seem to proceed at an accelerated rate as the feed tank level dropped and the feed pump motor-heater began to "kick out" more often, thus stopping the pump. However, suspension of flow through the furnace probably also accelerated furnace fouling. Installation of a recording ammeter on the circuit was completed to provide information.

While the furnaces now in service have served to provide design and operating data and experience, product quality and characteristics, yield data, maintenance information and experience, it is clearly apparent that heat distribution in the tubes in the Fractionator heater is very uneven and leaves much to be desired. High metal temperatures on about eight tubes out of 84, forced reduced heater temperatures. Velocity through the furnace is less than optimum, particularly for a charge stock with strong fouling tendencies.

A new spiral 3" pipe coil must soon be bought for the Vacuum Flasher heater or a more suitable heater supplied. The bottom spirals of the coil are excessively burned and scaled on the outside. The inside of the coil would be very difficult to clean as the heavy deposit is not ordinary petroleum coke, readily steam-air decoked, but is largely composed of metallic oxides and sulfides derived from dispersants and detergent additives present in motor oils.

The existing heaters have provided the necessary information to set

up specifications for better suited, simplified standard equipment for slower fouling, longer runs, and improved products to obtain an economically viable processing scheme.

Run #6 - June 11-18, 1970

More than 200,000 gallons were processed during this run. The entire production was sold as high grade fuels. The 'bottoms' in particular, were especially attractive to our customer -- who markets this product as a low-sulphur, minus 15°F, pour fuel oil additive -- with excellent heat producing characteristics when mixed with either #4 or #6 fuel oil. The use of improved inhibiting additives resulted in improved operating equipment performance. The replacement and rearrangement of the baffle in the boiler to accomodate pressure surges steadied boiler performance. In general, the unit operated without difficulty, and with tube deposits at a minimum. The technique of blowing down light reflux lines helped eliminate blockage. Yields and product specifications were slightly better than in previous runs.

After run #6 was completed, Esso/Enjay engineers observed on-site conditions and recommended better inhibitor injection techniques at certain points in the operation to achieve optimum equipment performance.

Run #7 - July 25-31, 1970

On July 24, 1970, we started up the boilers and unit and circulated it until shut down on Friday, July 25, which was due to holes, one in an air line, and one in a super-heater. After making repairs, we once again started up the unit and ran it until July 31, 1970.

A refinery run of five days was aborted by a fire which occurred in the vertical primary heater (replacement of the heating coil was anticipated). This run pointed up the recurring problem of coking material accumulating in the various small lines around hot oil pumps feeding the lantern glands of the pump stuffing boxes, on the spray decks of the light product section of the Vacuum Fractionator, at the draw-off piping seal loop which leads from the Fractionator to the light draw-off stripper, and it also accumulated at the bottom light product stripper and light product run down tank.

The entire production was sold to our customer, who marketed the products as a high grade fuel blend and low sulphur, low pour fuel blend with #6 fuel oil. Our marketing concept was altered to accomodate

this potential -- which aided consumers unable to meet low sulphur requirements -- now being enforced throughout the nation. Our consultant engineer was assigned the task of upgrading our distillates (4 cuts) to a specification permitting use as #2, #4 and #6 fuels.

Run #8 - August 22 - September 2, 1970

During the month of August there was a concentrated effort to repair the damages to the vertical furnace caused by fire. The entire inner surface of the heater was insulated, a new burner was installed, and the electrical and control wiring and apparatus was replaced or repaired.

A refinery run was commenced on August 22, 1970, and terminated on September 2, 1970. The results of this run matched the previous results, with a marked increase in the coking material on the tubes of the horizontal furnace, in addition to the accumulation of gummy-like coke in the light reflux lines. Approximately 300,000 gallons of feed stock were processed during this run.

All the production of this run was again sold to our customer (a fuel oil dealer) who marketed products produced as fuel oil blends, straight fuel without blend, and diesel fuel.

Run #9 - September 26 - October 15, 1970

This run commenced on September 26 and was characterized by running at a higher charge rate than previous runs, in addition to which the charge stock was of a lower viscosity. While there was some accumulation of tarry meterial in the usual critical areas mentioned in previous runs, this run was practically free of tube coking. More than 250,000 gallons of feed stock were re-refined and sold as useful petroleum products.

However, the demise of an acid/clay treating re-refiner in this area, brought pleas by local 'compounders' to do 'something' for their critical need of a re-refined product, free of odor and tarry material, and with a minimum of #5 color.

Run #10 - October, 16-19, 1970

To meet the product demand of 'compounders' who were now faced with the problem of a new source for re-refined lube oils, piping and pump changes were made to permit re-distillation of distillates #3 and #4. Eighty thousand gallons of sidestreams were re-distilled, with resulting good color (35) and improvement of odor. Color was stable. All products were sold to 'compounders' as lube stock.

Yield, however, was poor, as hook-up was of experimental nature and feed stock had a narrow boiling range making it difficult to control. However, results were impressive enough to include re-distillation of distillates as an added feature of the future process unit, if economical.

Run #11 - November 21-26, 1970

The results of this run were interesting in that one combined distillate was produced instead of the usual light and heavy sidestreams. While this was attributed to blockage in the light product draw-off line, it is interesting to note the marketing advantage of a single combined sidestream product when needed. This phenomenon also resulted in a higher gravity 'bottoms' product which eliminated the necessity of additional blending for our customer. Entire production was sold to customers at equal or increased price

This run demonstrated ability to produce one combined sidestream with a higher quality bottoms product, which may be desirable as a marketing advantage, when occasion arises; such as low-sulphur fuel shortage, and high viscosity lube and fuel distillate demands.

PROCESSING COSTS AND YIELDS

A Breakdown of Runs #4 and #5 Including Input, Yields, and Costs of Products Produced

Run #	Feed (Gal.)	Bottoms (Gal.)	Hvy. (Gal.)	Lt. (Gal.)	Baro. (Gal.)	Gas (Gal.)	Fuel Water Loss
4	176,288	67,850	46,700	44,475	-	863	16,400
5	228,324	47,360	102,300	39,591	22,000	913	16,160
Total	404,612	115,210	149,000	84,066	22,000	1,776	32,560

Product	Total Gal. #4 and #5	Combined Yield % of #4 and #5	Yield % #4	Yield % #5
Bottoms	115,210	28.4	38.5	20.7
Heavy	149,000	36.8	26.5	44.9
Light	84,066	20.8	25.2	17.3
Barometric	22,000	5.5	-	9.6
Gasoline	1,776	0.5	0.5	0.4
Fuel, Loss (H_2O)	32,560	8.5 Fuel	9.3	5 Fuel / 4.3 Water 7.1	5 Fuel / 2.1 Water

Total Direct Costs (Runs #4 and #5) per gallon

Direct Costs	$16,240.77	= .0401/gal. processing cost)
Feed Charge	404,612	)
Total Fixed Costs	$1,226.58	= .003/gal. processing cost)
	404,612	)

Add Feed Charge Cost = .03/gal.

Total Cost = .0731/gal.

Schedule of Sale of Products
Runs #4 and #5

Items Produced	% Yield	Total Gal.	Selling Price/Gal.
Bottoms	28.4	115,210	.05
Heavy	36.8	149,000	.07
Light	20.8	84,066	.08
Barometric	5.5	22,000	.08
Gasoline	0.5	1,776	.10
Fuel (Plant Use)	5.0	20,230	.10
Water (Loss)	3.0	12,330	-
TOTAL	100.0	404,612	(avg. of .07/gal.)***

Table 1.

*** With improved equipment, the selling price per gallon will double.
(See specific recommendations - page - 55)

SECTION V

PLANT OBSOLESCENCE AND DEPRECIATION

Any analysis of processing costs and yields should make reference to the dramatic obsolescence and depreciation factors of the plant and equipment. These factors barred the way to a confirmation of results obtained thus far, as they were not anticipated in the P&I as presented in the grant application.

1. The rapidly changing composition of the waste crankcase oil raised havoc with plant and equipment.

2. Environmental codes being enforced by regulatory agencies required modifications to plant and equipment to solve problems as they appeared.

3. Change in the presently accepted marketing concept of re-refined products due to environmental restrictions and charge stock changes.

As delineated in the refinery runs (1-11), the present plant was rendered obsolete and ineffective due to the numerous and costly modifications to plant and equipment to solve problems caused by changes in the demonstration 'ground rules' (factors 1, 2 and 3 above) in order to acquire engineering data for the optimum design processing plant of the future. No single piece of equipment escaped the operating difficulties caused by the great percentage of fouling and tar formation resulting from pyrolytic polymerization during the processing. A plant designed for a 1965-67 feed stock was found difficult to operate and unsuitable for present feed stock loaded with tar-forming substances and metallic solids generated by decomposed additives. Moreover, environmental protection regulations practically force a small plant to avoid use of water as a cooling medium. Consequently, the costly installation of our A.P.I. oil-water separators designed to meet criteria formerly applied to refinery operations, are now obsolete according to New Jersey water quality standards.

Accordingly, any new plant design will benefit from the subject matter in this report, but additional work is required to demonstrate our ability to meet effluent standards for this type of operation, while at the same time indicating that this process, with additional equipment, will produce economically marketable products, i.e., total recycling of waste crankcase oils without polluting effluents.

SECTION VI

REVIEW OF ENGINEERING ACTIVITIES FROM 1/69 - 12/31/70

The following is a summary of the engineering activities during the grant period, including the results of laboratory and field investigations (pursuant to P&I ground rules) with comments on considerations for future investigation.

1. We installed separation equipment to definitely stop entrainment of bottoms into distilled products. The equipment was effective.

2. We found that entrainment, when crankcase oil was distilled under vacuum, did not account for poor color and odor.

3. We have established that waste crankcase oil can be diluted and centrifuged, removing substantially all solids from the waste crankcase oil. Centrifuging of feed effectively reduces solids in bottoms in laboratory vacuum distillation, however, plant operation economics has not been established.

4. We have identified the troublesome materials in the feed which produce tar, poor color and probably odor. These materials act in the same way in the plant and in laboratory vacuum distillations. They have been generally identified by scientists working in laboratories, and the results are available in technical literature on the formation of sludge in lubricating oils, and deposits in gasoline engines. The materials are:

a) Oxygenated hydrocarbons generated in quite small percentages, mainly from fuel. They enter engine crankcases as blowby, past the pistons.

b) The catalysts which induce formation of tar are blowby gases, mainly nitrogen oxide.

c) Additives which inhibit formation of deposits are compounded into motor oil. They are almost completely broken up by heating crankcase oil to 700°F. The presence of inhibitors and their breakdown at plant operating temperatures may explain the relative absence of deposits in engines, and the prominence of tar deposits when distilling in a crankcase oil plant.

d) The recovered tar, as expected, contains considerable ash, probably derived from deccomposing additives, dust and lead.

5. In the plant, we have demonstrated removal of tar and odorous products, by soaking and settling at temperatures, and by redistilling, which also includes considerable soaking and settling

at temperature, and by stripping. We found that even redistilled oil darkens and precipitates tar after soaking two hours at temperatures as low as 340°F.

6. We plan to conduct laboratory investigations concerning the use of catalysts, chemicals, separating aids, soaking and other procedures, either singly or in combination, for economically and completely (or almost so) reacting to, and rejecting tar, color and odor forming materials. Procedures such as caustic treating, acid/clay treating, etc., produce pollutants, and are not satisfactory economically.

7. Laboratory work has shown that tar and color-forming material in distilled products can be extracted with small enough quantities of suitable solvents to be properly economic. Plant economics has not yet been demonstrated.

8. We have identified problems connected with distilling and processing crankcase oil, and have developed plant apparatus, and procedures for dealing with some problems.

9. As pollution problems continue to manifest and become noticeable with time, investigation and increase of knowledge, it becomes increasingly apparent that satisfactory complete disposal of crankcase oil to avoid all forms of environmental pollution is more of a problem than was at first supposed.

10. General layout of a crankcase oil plant for avoidance of pollution from the operation of the plant calls for very detailed design, as in the case of an oil refinery. Consideration of the topography of the land helps. The study of geology finds oil, and the study of topography helps hold it.

11. The major crankcase oil processing cost is labor. Plant processing must reckon with this fact. The labor force in a crankcase oil processing plant must be small. The application of relatively simple equipment and particularly simple instrumentation is an economic must.

SECTION VII

SUMMARY OF PLANT MODIFICATIONS RELATED TO PRODUCT QUALITY DEVELOPMENT - 1/69 - 12/31/70

Conditions Prior to Grant Award

Mist Blankets

Prior to the grant award, the Fractionator had been equipped with a mist blanket positioned between the flash zone of the Vacuum Fractionator and the bottom bubble tray, to prevent liquid particles entrained by vapors in the transfer line from passing through the Vacuum Fractionator flash zone into the bottom bubble tray, and into the heavy product sidestream drawoff below the bottom tray. The mist blanket, in spite of a wash oil stream distributed over it, fouled rapidly and repeatedly. The sidestream drawn off the bottom tray ran dark; always darker than ASTM 8 color. The upper sidestream was also darker than ASTM 8 color. The mist blanket was removed and the transfer line was connected to an eductor drawing in flash zone vapors and discharging tangentially around the cylindrical shell of the Fractionator to induce a high circular velocity and produce a pronounced cyclonic separating force. The superheated stripping steam then used was also introduced into an eductor drawing in stripping steam and stripped vapors and discharging at high velocity to set up a cyclonic separating force.

The color of the distilled heavy product drawn off the bottom tray improved slightly, but remained darker than ASTM 8 color. The vapor uptakes on the bottom bubble tray continued to foul, but at a considerably slower rate. The light product was also usually darker than ASTM 8 color.

Conditions After Grant Award

Cyclones

After the grant award, the 4' diameter bottom stripping section shell of the Vacuum Fractionator was extended up into the 6" diameter main shell of the Fractionator. A cyclone top was placed on top of the extended 4' diameter section. A new larger 8" diameter transfer line was run from the Fractionator Furnace through the outer shell of the 6" diameter section of the Fractionator, into a tangential connection with the extended stripping section shell forming a completed cyclone with the cyclone top. The stripping steam connections were also installed tangentially to provide a cyclonic effect.

Above the cyclone, at the top of the stripping section, a cyclone supporting deck was welded to the Fractionator shell. Six "high

efficiency" cyclones, each 17 1/2" in diameter, with proportions on which considerable performance data was available, were installed on the deck. Each of the 6 cyclones were provided with a steam atomizing spray nozzle which sprayed wash oil into the vapor inlets of each cyclone. Several unit runs were made with a noted slight improvement in bottom sidestream, heavy and light product color. Heavy product separators averaged about ASTM 8 color and light product averaged about 7-7 1/2 ASTM color.

To reduce the barometric oil loss to cooling water, two 10' x 22' x 4' API-type oil-water separators were installed. Operation showed conclusively that a water soluble coloring material was going over the top of the Fractionator into the barometric condenser and oil-water separators. The bottom bubble tray vapor uptakes accumulated very few deposits, however, dark tar deposits continued to accumulate, particularly in the pans of the side-to-side spray decks of the Fractionator and in the light reflux accumulator. The barometric oil collecting on the surface of the API-type oil-water separator, markedly darkened on standing.

The wash oil spraying was stopped, however, no definite difference with or without wash oil when considering the variable feed stock, could be noted.

Tar Formation and 'Blowby'

A dark viscous tar continued to cause difficulties by plugging the 2" pipe coil reflux coolers in the cooling tub. The evidence was rather conclusive that tar-forming material was going over in the vapor phase. Entrained solids carryover had been reduced to a practical level, while deposit buildup in vapor uptakes on the bottom tray above the flash zone of the fractionator was not significant. More tar was formed in the light sidestream product than in the heavy sidestream product. The color material was giving the overhead barometric condenser cooling water a yellowish tint that darkened on standing with an oil layer. The oil layer also darkened.

A paper by C.J. Comke, D.J. Lindley and C.N. Sechrist, "How to Study Effect of Blowby Gases, Hydrocarbon Processing," Vol. 45, No. 9, p. 303, Sept. 1966, reported various oxygenated compounds in the crankcase, derived from blowby past the pistons and rings of the engine. Through Mr. Richard Keppler, the Project Officer, representatives of an additive and chemical concern came to the plant and recommended injection of a furnace tube antifoulant, to be applied to the feed at 100 ppm, and to the heavy and light products, at the rate of 50 ppm. They also told us of a paper, "The Mechanism of Deposit Formation and Control in Gasoline Engines,"

by Dr. Jerome Geyer, presented before the Division of Petroleum Chemistry, Inc., American Chemical Society, New York City Meeting, September 7-12, 1969, wherein it was indicated that in tests "running a labeled benzene fuel," the carbon in the fuel contributed 95% of the total carbon found in the sludge formed in the crankcase oil of a gasoline engine operating in the steady state. The crankcase oil is stated to be the presumed source of the other 5% of the carbon in the sludge. The oxygenated hydrocarbons derived from gasoline, described as "including all oxys to C_{12}," would have a boiling range which would cause them to be found most abundantly in the light product. This is the product in which most tar forms. The paper also indicates the particular importance of blowby nitrogen oxides, and other blowby gases in catalyzing the formation of sludge from the oxygenated hydrocarbons, of which probably 95% are derived from gasoline. In any event, the total amount of active material, oxygenated hydrocarbons and nitrogen oxides, is surprisingly small.

The injection of inhibitors seemed to hold down the formation of coke within the furnace and the hardness of the tar in the distilled products, but the color of the products remained dark, and tarry material could be observed settling in streaks to the bottom of sample bottles left undisturbed.

Re-Run (Odor and Color Improvement)

Both light and heavy product were accumulated and then re-run through the Fractionator Furnace and Vacuum Fractionator. The heavy product color improved from ASTM 8, to ASTM 7 or 6 1/2. Odor was considerably better. Light product color improved from ASTM 7 1/2 or 7, to ASTM 6 1/2 or 6. Despite the fact that it had a considerably higher boiling range than the light product charge stock, the odor on re-run light product was noticeably better than that of the light product charged to the re-run operation. No fouling inhibitor was injected into the unit during re-running.

Inhibiting Additives

The additives compounded into automotive oils are chosen for their demonstrated ability to inhibit the formation of sludges and tarry material in crankcase oils. However, talks with suppliers of additives, plant experience and some data in the literature confirm the fact that these particular additives are substantially destroyed when the compounded oil is heated to 700°F. When waste crankcase oil is charged to the unit, the transfer line temperature has run between 640°F and 700°F, depending on the vacuum available on the Vacuum Fractionator and the API gravity required on the bottom product. During the re-run operations,

the transfer line temperature ran at 680-690°F. The amount of inhibitor additive left in the heavy product when distilling crankcase oil must be low. The inhibitor content in the re-run heavy product after a second heating and distillation must have been very slight. Light and heavy products were re-run separately.

Tar Settling

The bottoms of the light and heavy product rundown tanks were cleaned out before the re-run operation in order to observe deposition of tar from re-running. Tar settles to the bottom of the tanks when product is left standing in the tank. The vertical shell of the tank is coated with a thin dark layer of tar which apparently does not increase in thickness, and no attempt has been made to remove this thin coating.

After the re-run operation, the light and heavy product produced was permitted to stand for 15 days before removal and shipment. The accumulation of settled tar in the bottom of the tanks was observed to be approximately 1/4" in thickness. The tar in the light product tank was harder and more dense than the tar in the heavy product tank, as is also the case when waste crankcase oil is charged to the unit. The percentage of tar that settled was about three times as great as the percentage of tar observed to settle from light and heavy products produced directly from waste crankcase oil and permitted to stand for approximately the same length of time in the rundown tanks.

Clay Contact

Prior to the grant award, we contacted light and heavy products of ASTM color 8-plus in both cases, with about 0.3 lb./gal. of activated clay, at 550°F for 30 minutes, cooled to 180-195°F, and filtered in series through a plate and frame filter press and a plate and frame "polish" press. The color on light product dropped to 2-3 ASTM color, while on heavy product, the color dropped to ASTM 3-4. Some odor persisted in spite of clay treating. The treated oil lost the characteristic haze of untreated light and heavy product, but color was stable. No sediment fell to the bottom of undisturbed bottles of clay treated oil. Oxygenated hydrocarbons, and tar in the oils were effectively removed by clay treatment.

Labor and other costs, as well as environmental requirements, render clay treating as formerly practiced uneconomical and undesirable. The clay is costly and constitutes a relatively large volume of material which is costly to dispose of without generating pollution.

Soaking and Settling (Color and Odor Improvement)

The tarry material accumulated in the rundown tanks after settling was approximately .09 - .10% of the oil volume. This probably represents some 60-70% of the total tar-forming constituents that were originally present in the waste crankcase oil. Redistilling, on the very first plant attempt, did show a very significant improvement in color and odor. Further improvement, if only slight, would definitely open the door to acceptance in a higher-priced market.

However, it seems probable that the same improvement may be obtainable without redistilling, and the accompanying tying up of plant, with marked reduction in plant capacity, and increase in labor and fuel cost per gallon of product. Soaking and settling may be more economical and provide the same result.

With the existing plant arrangement, the light and heavy products, after distillation, are little exposed to standing or soaking at distillation temperatures, separation, and trapping of tar in vessels, from which tar can be removed inexpensively.

Therefore, it is planned to install a "soaker & settler" tank, possibly containing a tilted plate separator, for soaking and settling the light product as it comes hot off the light product section of the Fractionator.

In essence, the soaker-settler would closely approximate the appearance of an old-style thermal cracking plant reaction chamber with inlet, outlet and some other piping. The soaker tank would be tilted slightly to aid peeling the tar off the bottom of the horizontally positioned soaker tank during plant shutdown and cleanup operations. If soaking and settling works well with the light product, it will be applied to the heavy product.

Elimination of Tar Traps

The top of the Vacuum Fractionator has already been altered to provide minimum settling time for tar deposition. We had hoped that we could eliminate pockets and tar traps in the upper section of the Vacuum Fractionator and keep tar moving until it and the light product got to the product rundown tank. However, the tar settled particularly in cooler outlet lines where it must be cooled to a minimum of 190°F to avoid darkening by oxidation in rundown tanks. The lines now are about as short and direct as practical. We plan to give it a chance to form completely and settle where we can easily remove it. We may be able to find some catalyzing material that can be injected into the light product as it enters the line into the soaker. See Flow Diagram No.

Need for Laboratory Back-Up

Through organic chemistry investigations, we propose to seek catalysts and chemicals which may accelerate tar formation and deposition, and provide more information about the characteristics of color removal reaction or reactions, optimum temperature, pressure reaction velocity and equilibrium factors.

In the meantime, we continue to accumulate and collate everything in the way of data economically available that might be significant in developing basic information for finding an economic way to achieve better color, odor, and carbon residue on the distilled products; and less sediment and ash in the bottoms.

Accumulation of Engineering Data for Needed Equipment

We have accumulated and will continue to accumulate and collate experience and data relative to the equipment described in our annual report of June 21, 1970, pages 32 and 51, namely: Furnace; air-cooled coolers and condensers; vacuum pump; bottoms pump; and solids removal. Further investigation indicates that dilution of either the crankcase oil feed or bottoms, with low viscosity naptha and separation, is indicated to remove the solids from the bottoms. In view of data recently available, relative to metals pollution in the air, separation may become a requirement for use of bottoms for fuel; most other usages for bottoms also require solids removal.

Laboratory Distillation Investigation

Results of recent laboratory investigations of processing methods are worth noting. We diluted representative crankcase oil feed 1:1 with 46° API naptha distilled from the Vacuum Flasher and had it centrifuged at 10,000 x gravity, removing 2-3 wt. per cent of solids.

Then the centrifuged sample was distilled at 760 MM in the laboratory to remove the 46° API naptha. Distillation was stopped when the "head" temperature or temperature of the vapor distilled off, reached 400°F. The temperature of the "pot" or liquid in the flask at this time was recorded at better than 600°F. The remaining sample was cooled and later distilled under 5 MM Hg absolute pressure in a glass flask and column. The column was equipped with a refluxing condenser and an adjustable controlled valving arrangement that provided a cycle of refluxing 5 seconds and production for 5 seconds. With crankcase oil, it tended to stick. Cuts were made according to a schedule to produce material of the boiling point at 760 MM ranges as stated under the heading "Vapor Temperature of Condensing Product or Cut." See tabulation of

data from Laboratory Vacuum Distillation, attached (following page). The distillation required about 12 hours with one day's interruption between the first and second days of distillation. The actual flask or pot and head temperatures are recorded for the finish of the distillation of products No. 3 and No. 4. The peak temperature in the flask or pot (683°F) was actually about the same as the usual transfer line of the Fractionator Furnace. The cuts produced were roughly similar to average plant products, however, the fractionation was much closer. The light and heavy products were quite similar in gravity, flash and viscosity, but laboratory cuts were distinctly different in color as they condensed and were accumulated in the receiver. They were much lighter in color with the exception of the naptha cut, but all cuts quickly darkened on standing, as the condensed liquid accumulated in the receiving vessel. The final colors, after prolonged standing, were still lighter than plant products. The dark material began settling towards the bottom. The odor of all cuts was much more acid or sour than plant cuts, without the acrid or burnt smell of plant products.

The vacuum distillation, as witnessed in glass, emphasezed the advantages of lower vacuum, 5MM distillation, using motor-driven vacuum pumps without stripping steam.

Observations of laboratory vacuum distillation of waste crankcase oil, confirm the findings reported in technical literature and the observations of actual plant operations.

The testing of inflowing salt-cooling water, and the outflowing salt-cooling water also indicate the presence of acidic materials released to the water in the barometric condensers.

Table 2.

DISTILLATION OF CRANKCASE OIL AT 760 MM Hg. AFTER DILUTION 1:1 WITH NORCO NAPTHA AND CENTRIFUGATION

PRODUCT	NORCO Naptha Diluent Distilled at 760	CONTINUING DISTILLATION OF CRANKCASE OIL AT 5 MM AFTER DISTILLATION AT 760 MM				
		Remainder of Diluent	Barometric Oil	Light Product	Heavy Product	Bottoms
NORCO	Number 1, Plus 1:1 of Diluent		Number 2	Number 3	Number 4	Number 5
Gravity ° API	47.1	39.1	33.8	31.6	29.8	---
Vapor Temperature of Condensing Product or Cut 760 MM	140-352°F	352-400°F	400-680°F	680-792°F	792-900°F	900°F Plus
Viscosity SSU at 100°F	---	---	---	130	239	(Est.) 200 Furol at 122°F
Flash	---	---	---	405	455	---
Color, ASTM 15 Days Standing	Light 1	3 1/2	Light 5	4	5	Dark Brown
As Condensed	---	Approximately 2 1/2 Hazy Brown	Approximately 2 1/2 Hazy Brown	Approximately 2 1/2 Hazy Yellow	Approximately 3 Hazy Yellow	Dark Brown
Color of Bottom of Settled Bottle	Very Slight Deposit of Light Tan Sediment	Heavy Black Fixed Coating	Heavy Black Fixed Clusters	Soft, Dark Brown Semi Fluid	Dark Brown, Slightly Fluid	Dark Brown, Greasy Fine Solid Particles
Odor of Settled Cut	Sour, Somewhat Sharp, Slightly Oily	Sour	Sour and Oily	Very Slight Sour	Slight Sourish	Very Slight Oily
Approximate Volume M.L.	1,000 Plus	130	93.5	254	375	57.5 910 ML
Approximate Percent	---	14.3	10.3	27.9	41.2	6.3 100.0
End of One Distillation Temperature Pot	600 Plus °F	---	---	622°F	683°F	---
Head	---	---	---	550°F	567°F	---

SECTION VIII

REVIEW OF PLANT OPERATIONS WITH MODIFICATIONS TO IMPROVE EFFICIENCY FROM 1/69 - 12/31/70

The following is a summary of plant operation techniques including feed stock control, mixing temperature controls and plant equipment to achieve simplified processing with the use of antifoulants.

Uniform Feed Stock

To maintain uniform composition of feed to the process, and thereby maintain control over product streams specifications, two 40' diameter by 30' high feed tanks are equipped with four mixing eductors directing the flow at an angle of 30° upward and tangentially around the tank in a counter-clockwise direction. The eductors are fed with a rotary positive displacement pump at a rate of about 100 gpm at 30-40 psig. During the warmer half of the year, the tanks are well mixed by 3-10 hours pumping. Strainers are necessary to avoid damaging pumps and plugging eductors. The tanks are not equipped with heaters. During the cold winter months, mixing is difficult. The most economic answer would probably be the installation of adequate propeller type mixers. Keeping tanks hot all year is very costly. High energy input mixers, eductor or propeller types, operating a short time, are more efficient and economic than low energy input mixers consuming actually more KW hours of electric power.

Feeding cold, directly from tanks during the winter months has proven practical. Suction and discharge lines must be sized for laminar flow and practical pressure drop. A long suction line is not desirable because of pressure drop and lowered pump efficiency. Considerable savings can be realized by not maintaining feed tanks at some set temperature. Water content of crankcase oil as received has been low. Some water has been drawn off the bottoms of feed tanks. When feeding from cold feed tanks, adequate surface should be provided in the convection tube section of the flash furnace to accomodate the high viscosity and low heat transfer rate characteristic of cold oil. This provision prevents excessive skin temperatures and resultant coking of heater tubes, especially during the winter months. Heat exchangers might be provided to preheat the cold feed, but would have to be proven economic.

A motor-driven rotary positive displacement feed pump, equipped with reduction gear drive, installed and used since early April 1970, has proven satisfactory for both summer and winter. A strainer is required, primarily for the relief valve on the pump. as it showed a tendency to jam open on relatively small particles of trash in the feed.

After passing about 3,000,000 gallons of crankcase oil through two feed tanks, 40' diameter by 30' high, the tanks were opened and inspected. Very little water and sediment was noted and there was no apparent corrosion.

Antifoulant Injector

We installed anitfoulant injectors, and now inject antifoulant material into the feed entering the Flash Furnace at the rate of 100 ppm. The fouling of Fractionator Furnace tubes may have been reduced slightly, but performance is difficult to evaluate because of feed stock variations, troubles with instrument control, etc. The tube diameters in our furnaces are large, velocities in the tubes are low, and pressure drops through the furnace tubes are also low. Fouling normally is considerably greater in black oil heater tubes at lower velocities. We have increased the flow velocities in a few furnace tubes by installing cores. These tubes had a tendency to overheat, but cores increased cleaning time and cost. A re-refining oil heater, from our experience, should have fairly high velocities in the tubes, coinciding with a higher pressure drop.

We also injected antifoulant into the light and heavy products reflux to decrease the tendency to foul the Vacuum Fractionator internals, reflux lines, pipe coil coolers and control valves. We did notice a decrease in fouling, but color was darkened slightly. We have both injected and not injected, attempting to arrive at definite conclusions. The situation is now clouded because we wish to remove the color forming material from the product and are exploring re-running, soaking, polymerizing catalyst, etc. Antifoulants, we think, are troubleshooting items that can be helpful.

Modifications to Equipment to Improve Operation

The top of the flash tower was originally provided with Raschig ring packed sections, which tended to generate Raschig ring chips which caused trouble with the positive displacement rotary Fractionator Furnace charge pump. The Raschig ring sections were removed. The top of the Vacuum Flasher was converted into cyclone structure, and the Flash Furnace transfer line was enlarged and arranged to discharge tangentially into the cyclone top of the Vacuum Flasher. The results have been satisfactory. The color of the overhead product dropped from ASTM 2 to 1-1 1/2. Difficulty with Raschig ring chips has been avoided.

There is less accumulation of loose deposits on the cylindrical shell, and less dirt collects around the large screen, installed at the bottom of the Vacuum Flasher to keep particles of tower

deposit from getting into the rotary positive displacement pump feeding the Fractionator Furnace. The feed stock constantly changes, and exact comparison is difficult.

The Fractionator Furnace is of classical design. Originally, the tube surface exposed to direct radiation of the flame was quite small, requiring excessive air flow to avoid overheating of the tubes exposed to direct radiation. Before starting up, after the grant award, the furnace blower discharge was piped to provide flue gas recirculation into the furnace. This decreased the excessive radiation rate to relatively few tubes and also reduced fuel consumption.

Even so, difficulty was experienced with local overheating of radiant tubes nearest the flame in the Fractionator Furnace. The 1 1/2 HP motor was replaced with a 3 HP motor. New sheaves and vee belt were installed on the motor, increasing blower impeller speed 26%. This increased flue gas recirculation and relieved the tendency of radiantly heated tubes to become overheated.

Wear and Tear

During several runs, the wear rate at the end of the 8" Fractionator Furnace transfer line, where it discharges tangentially against the shell of the 4' diameter stripping section, has been excessive. We have installed a wear plate at this location and will install a wear 'plate' with a welded-on, hard-faced coating of a suitable super-hard metal. We now control the outlet pressure at the furnace by adjusting the amount of steam injected into the furnace inlet, the furnace transfer line temperature, and the dehydrated oil charge rate to the furnace. The wear rate may also be a function of the particulate hard solid matter which may be in the charge from time to time. Centrifuging feed stock would probably be helpful.

Superheated Steam

We originally used a steam superheater for superheating stripping steam. The superheater included controls, blower, fuel pump, extended surface heating tube, expansion joint, etc. The maintenance and attention required, and the upset operation of the Vacuum Fractionator, frequently caused by the superheater, resulted in the decision to abandon it. We now use throttled saturated steam for stripping in the bottoms stripping section, in the Vacuum Flasher, and in the two sidestream strippers. The Fractionator Furnace transfer line is run at slightly higher temperature to offset the lower heat content of the stripping steam. A furnace specifically designed for waste crankcase oil processing at 30-75 MM Hg would

include a conventional superheater in the convection section. A plant equipped with motor vacuum pumps operating at 5-10 MM would not require superheated steam.

Jamming and Screening

The 4" suction line for the Fractionator bottoms had no screen. Occasionally, we experienced evidence of the jamming of the side pot disc valves in the steam reciprocating simplex pump. A quite coarse screen, covering the 4" suction line, was installed at the bottom of the Fractionator. Now a cubic foot, or more, of solid deposit accumulates in the bottom of the Fractionator during a run, but the pump operates smoothly without evidence of valves jamming.

Reduce Fouling and Contact Loss

The top light product section of the Fractionator is provided with three spray decks and a bottom liquid catch pan. Tar has collected in the spray decks and catch pan. Tar is always heavier than oil and settles in low spots and traps, tending to polymerize and become more viscous and of higher specific gravity on standing. Tar accumulations build up and distort the flow patterns of descending light product reflux and the ascending vapors, reducing the effectiveness of contact between vapors and liquid.

To reduce fouling and loss of contact between liquid and vapor, the pans of the two spray decks have been filled with sloped concrete and provided with a low-volume flow channel at the weirs of the spray deck. This greatly reduces the volume available for catching tar and for "soaking" the reflux liquid and any formed tar.

The time and labor required for cleaning the spray decks has been very sharply reduced. Contact between vapors and liquid, judging from operating and product data, is good. The third spray deck will be modified to minimize trapping of tar and "soaking" of light reflux liquid. The catch pan, under the bottom of the three spray decks, will also be modified to reduce tar trapping and cleaning time.

Steam Blowing to Remove Tar

From observations of plant operations, examination of tar accumulation, the available subject literature, etc., it was decided to install adequate steam blowing facilities for blowing all four side-stream cooling coils. The four coils, installed in 1966, are all located together with the bottoms cooling coil, in a common steel shell tub, supplied at the bottom of the tub with harbor salt water.

The coils are made up of 2" steel pipe "U" bends, connecting 20-21' lengths of steel pipe. The steam blowing lines are 2" pipe, with 2" valves, 2" check valves, and 2" connections and fittings, supplied with 100-110 psig saturated steam. Outlet lines for blowing coils and sections of piping and controls, etc., are all 2" pipe.

One cooling coil or section of piping is blown at a time. Tar, which is quite solid at 0-95°F, is plastic or fluid at temperatures ranging between 212-344°F, which are the saturation temperatures at atmospheric to 110 psig pressure. So far, the blowing arrangements have served to clear lines and coils enough to permit maintenance of operation. Until now, no dismantling of cooling coils has been necessary. Practically no opening of 2" pipe lines has been necessary. Suction lines to pumps have been broken to permit pump repairs, and inspection of suction lines, pump impellers, wearing rings, etc.

The tar is extremely adhesive and positive displacement rotary pumps quickly jam and lock when tar gets to moving parts. Centrifugal pumps are satisfactory for moving hot oil containing the tar, which coats impellers, but the film does not become thick. On cooling the pump casing, the wearing rings may stick to the hub of the impeller, but on forcing rotation of the shaft with a wrench and breaking loose, they should run freely; once warm or hot oil reaches the pumps.

Simplified Gland-Oil Piping on Hot Oil Pumps to Reduce Maintenance Caused by Tar Formation

We have removed check valves, pipe fittings and other gears which too easily plugged with tar in the 1/2" and 1/4" lines supplying gland oil to the light and heavy product and Reflux Centrifugal pump packing boxes. Check valves were not replaced. Pipe and fittings were replaced with steel tubing and tube fittings. Tubing was bent as required, with tube fittings permitting rapid assembly, opening, inspection and replacement. Flexible wire cable provided with a cutting tip and a rotary drive arrangement, with benzol solvent, provide practical cleaning arrangements.

We have also provided an elevated tank, for receiving cold settled heavy product, with provisions for maintaining a level in the tank. A line from the side of the tank provides cold heavy product under slight pressure to all lantern glands. This one line is provided with a single check valve to avoid any back flow from the pumps into the tank. The connections from the pump dischargers to the lantern glands have been retained, but they are normally left closed, and used only in emergency, or in case of difficulty with the supply of cold heavy product oil.

SECTION IX

INVESTIGATION AND FEASIBILITY REPORT TO UPGRADE NORCO FRACTIONS 1/69 - 12/31/70

The initial program definition focused primarily on a feasibility study to upgrade the possible uses to which the various NORCO fractions could be applied.

From this product engineering phase, we soon learned that not much could be done with the distillation bottoms without prior removal of solids. This conclusion was reached after extensive and time consuming investigation showed that none of the largest potential users, namely the paint-sealant-plastics-lubrication- and rubber industries, could tolerate metal-organic contaminants in the oils used in compounding. Moreover, some of these are even sensitive as to moisture, odor and color.

For these reasons, we fell back on the old standby use as a fuel oil which, regrettably, is advocated by the American Petroleum Institute. In the "Final Report of the API Task Force on Used Oil," the Committee recommended that up to 25% of spent lubricating oil (untreated) may be blended with fuel oil, and that this mixture may be used in existing commercial and industrial combustion equipment without deleterious effects.

This recommendation is neither wholly supported by the burning tests noted in the report, nor by the evidence from our study, which follows:

Re: Progress report to Mr. Richard Keppler, Project Officer, Federal Water Quality Administration (FWQA), Project No. 15080 DBO

Subject: NORCO Comments on Final Report of the API Task Force on Used Oil Disposal

Date: October 16, 1970

Dear Sir:

We carefully studied (with subsequent laboratory and field work) the FINAL REPORT OF THE API TASK FORCE ON USED OIL DISPOSAL. A highlight of the report was their recommendation that up to 25% of used lubricating oil may be added to other grades of fuel oil, and that this blend can then be successfully used without deleterious effect in existing commercial or industrial combustion equipment. Subsequent laboratory and field work led us to conclude that the API recommendation is erroneous and unsupportable -- and may, if followed, result in a combination of serious health problems and costly maintenance problems to most oil-burning equipment.

The NORCO project has placed a high priority on the cleaning up and purification of the feed stock and/or distillation bottoms without resorting to conventional acid/clay treatment. (This is in order to avoid a large buildup of process residue which would have to be disposed of, and which would constitute an ecological violation.) Our tests and investigations have shown that the major roadblocks in most applications to which the reclaimed oil may be put are the additives, high molecular organic metal compounds which are not readily released from solution. Unfortunately, they will decompose and constitute a serious hazard even in low-grade applications such as when used in industrial combustion equipment. This is substantiated by samples taken from the combustion passages and flues of several boilers. In these instances, reclaimed crankcase oil and deposits ranging from fly ash to hard rock clinkers resulted. Spectro-analysis of these clearly shows the presence of lead, magnesium, copper, aluminum, vanadium, silicone, iron, barium and various compounds with calcium and phosphorous which are deposited as phosphates, carbonates, and silicates; and as such are intolerable in boilers. We append a number of documents which will support this contention.

Exhibit A reports the particulate matter contained in the distillation bottoms from the NORCO refinery. It consists largely of carbon ranging into the colloidal state. The metallic constituents derive from automotive additives and to a smaller extent from engine wear. This analysis does not reflect on the metal-organic compounds still in solution.

Exhibit B deals with the above subject matter far more exhaustively.

Specimen #8U is a bottoms sample similar to the one analyzed in Exhibit A. All major, minor and intermediate metallic contaminants are present in the amount of approximately 5.6% of the total bottoms. The balance to be found in colloidal and particulate carbon, asphaltines, olephines, and of course, aromatic, naphthenic and paraphenic hydrocarbons.

Specimen #9U is a boiler tube deposit. This analysis clearly shows the presence of all inorganic compounds found in #8U. These deposits are formed from the organic state during the short transit of the fuel droplet through the burner flame. A Commentary (page 5) is appended and discusses some of the findings, conclusions and recommendations of this effort.

Exhibit C is a further analysis of boiler tube deposits and was conducted by the Research and Development Department of Texaco. As may be seen, these findings are very much in agreement with Exhibit B in that the spectro-graphic analysis shows lead and iron to be present in major amounts and barium, phosphorous, magnesium, aluminum, silicone and vanadium are minors. Further, an x-ray analysis evidences the presence of rust, sand, lead sulphate and magnesium phosphate.

Exhibit D is an analysis of the #6 (low sulfur) fuel oil which was burned in the boilers from which the deposits in Exhibit A and Exhibit B (Specimen #9U) were collected.

This analysis confirms the absence of any contaminants in the other fuel used. The presence of vanadium in a concentration of 46 parts per million is insignificant.

Exhibit E shows a graphic presentation of a Cleaver-Brooks fire-tube boiler which is similar to the equipment used.

In addition, samples were also collected from residential boilers in apartment buildings. In these, the deposits were not hard rocks, but rather dense, one-quarter to one inch thick layers of fly ash which substantially covered all cast iron heat exchanger surfaces, thus very really preventing any heat transfer to the building.

It appears that, given the proper flame temperature and transit time of the atomized fuel/additive droplets, there results a coating which is very much akin to that achieved by flame spraying of metal parts with inorganic metal oxides, a process often resorted to to prevent heat transfer, thus causing exactly the opposite of the results desired in combustion equipment.

Further reference is made to the Final Progress Report on Water Pollution Control Demonstration by Villanova University (Project Grant WPD 174-01-67). On page 3, (6) recites as follows: "The vacuum bottoms contain solid metallic compounds (probably as organo metallics) that render it unsuitable as a residual fuel."

The comments of Exhibit B attempt to go a step further, showing that the high metal concentrations found in used lubricating oils is not only a hindrance in its use for most commercial purposes, but also constitutes a valuable by-product which may easily be recovered and thus yields additional revenue for the re-refiner.

To restate in more dramatic form what is said in the foregoing:

The combustion (or decomposition) of one gallon of distillation bottoms causes the formation of 7 grams of heavy metals (based on 2 grams per liter). This is equal to a quantity of 5 gallons of collected oil, since the bottoms account for approximately 20% of the feed stock. However, 7 grams of metal in turn yield roughly 21 grams of deposits in the oxide form.

According to API recommendations, a boiler operating on 60,000 gallons of fuel per day would consume 25% of this amount in waste oil, resulting in 120 pounds of rock clinkers similar to those analyzed in #9U and the samples furnished herewith.

That this quantity is not supported by actual boiler operation, is entirely due to the fact that some of these substances are lost as volatiles and fly ash in the form of oxides, carbonates, sulphides, sulphates, phosphides and silicates, and as such, constitute an intolerable air pollution hazard.

In view of the above supporting evidence, the recommendation in the API report appears irresponsible since it has led to the disabling of a number of boilers which had to be cleaned and repaired. Moreover, the presence of motor fuel additives in fuel oil constitutes a very real health hazard to the entire community.

Very truly yours,

/s/

Solfred Maizus, Project Director
NATIONAL OIL RECOVERY CORPORATION

From these studies, we conclude that burning with untreated crankcase oils can cause serious maintenance problems with many burners and can potentially produce serious health problems associated with the discharge of heavy metals in exhaust gases. The spectro and particulate analyses also clearly indicate that the ensuing metallic oxide and carbonate clinkers, as well as any fly ash, are direct derivatives of the metal phosphates in the oil. Consequently, an extensive development program was initiated to develop techniques to remove these suspended solids and dissolved organo-metallics to not only make all the streams useful as products, but to also maximize the duration of plant runs.

Methods of Separation

Our efforts to find suitable separation methods included the following:

A) Centrifugation

Up to 50,000 g's and 1/2 hour residence time in a SORVAL machine proved commercially impractical.

Further work in a PFAUDLER centrifuge by varying temperatures and flow rates proved unsatisfactory. Similar results were obtained with a SHARPLES machine.

The introduction of another variable - namely dilution ratios - finally proved successful in causing significant separations and precipitations of additives as a dirty white to gray sediment. We noted approximately 3% of detention.

These preliminary results initiated a more intensive program, using solvents for pretreatment. These experiments were carried out by Centrico using WESTPHALIA equipment. Practical feedrates (resulting in approximately 20 seconds residence time) were obtained for the following minimum dilutions:

0.5 parts Naptha : 1 part waste oil, and
0.3 parts Naptha : 1 part distillation bottoms

B) Considerations relating to centrifuge cost and maintenance, plus a limit of roughly 1.5 on the specific gravity of the solid particles which can be safely processed, led to an investigation of Cyclone separators (DEMCO, - DORR OLIVER). It was not possible to properly evaluate this equipment due to budget restrictions, but indications are very promising, and this approach is definitely to be pursued further.

C) Settling Tanks

From our studies with centrifugation, it is also possible to use settling tanks in place of, or preceding centrifuges. Budget restrictions prevented a parametric study of this concept. However, it is safe to say that dilutions must be at least doubled in order to achieve fair results, and for cold temperature operation these ratios may have to be substantially increased.

D) Filtration

Organic and inorganic filter cartridges made of metal, ceramics, hair, fiber, felt, paper and plastic were investigated. Also

studied were the various filter shapes; namely round, flat and accordian zig-zag. Filters which were both throw-away and cleanable (wash or burn-off) were also investigated (CUNO, FRAM, PALL, CARBORUNDUM, etc.). Of these, edge-type filtration (VOKES) proved promising by providing relatively long filter runs. Further extensive efforts in this area should be beneficial. Also, the use of filter aids in combination with the above method is recommended for further study. (The use of clay and filter aids was not permitted to be included in this study by the Water Quality Office.)

E) Tests with Ultrasonic treatment in order to achieve coagulation, separation and emulsification are inconclusive. The response from FIBRSONICS and BRANSON SONIC PRIORY CO., as to performance, experience and available electro acoustic equipment, ranged from doubtful to negative. However, HOMO-REX, a mechanical ultrasonic emulsifying machine, yielded very stable suspensions. This should be pursued further.

F) Ion Exchange Resins

All literature investigations reveal this method as effective, but too costly for our plant-scale operation. D) and F) may be used without diluents, while E), on the other hand, should be combined with solvent pretesting in order to reduce the required force field power levels.

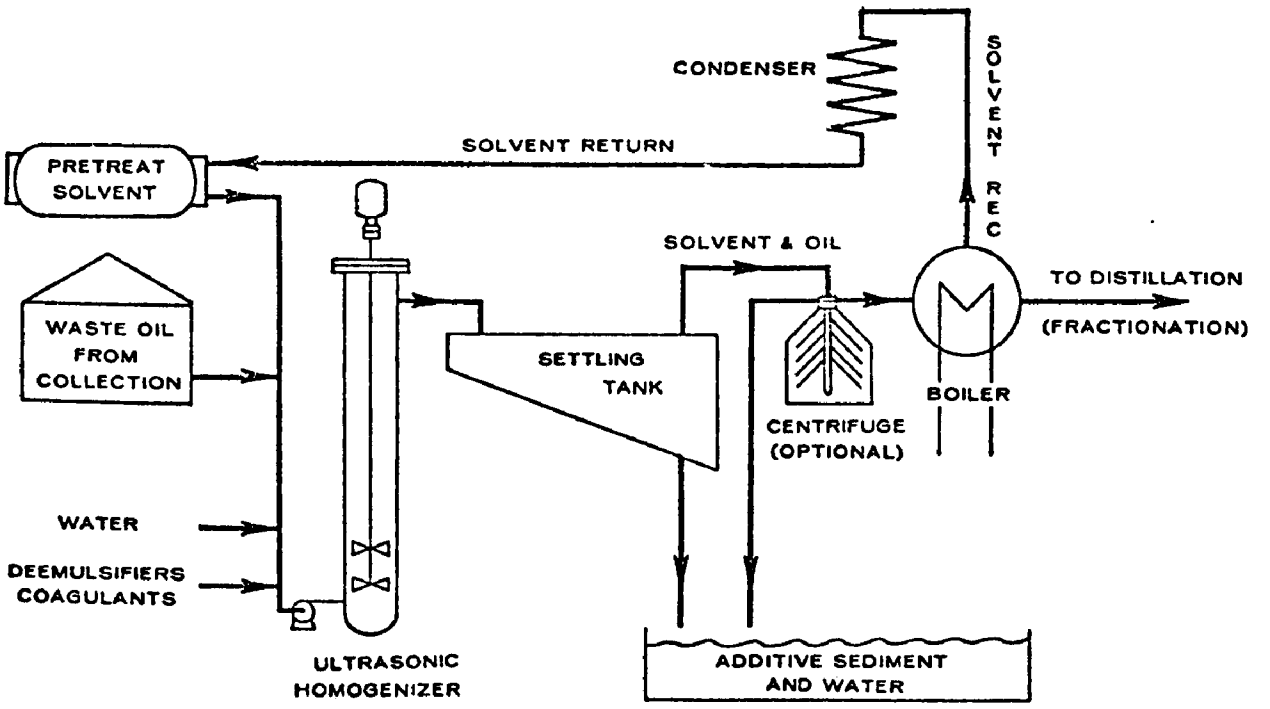

Figure 2.

Pretreating System

From the above studies, it is obvious that the existing or previously proposed flow sheets should be revised. NORCO proposes a process (see flow sheet above) which pretreats the incoming lube stock with organic solvents or aqueous diluents, including coagulants and de-emulsifiers to neutralize surfactant action. Centrifugation or settling tanks are used to eliminate particulate contaminants as well as precipitated metal-organic additives. Aqueous diluents are removed by decanting, separation and evaporation. Organic solvents are returned for use with fresh lube stock via a solvent recovery system. In this cycle, the solvent function is entirely mechanical and not chemical in nature.

A precise laboratory vacuum distillation of representative NORCO feed stock, pretreated as indicated above, provided the tabulated information that follows: (Note: Some overlap in boiling ranges of NORCO plant products, with less precise fractionation, would be expected.)

NORCO Fractions - Physical Properties

Fraction	Name	Amount % of Feed Stock	Color	Viscosity Saybolt Seconds Univ. (SSU)	Boiling Range of Init./Final	Specific Gravity (g/cm3)	Flash Pt. of Open Cup
1	Naptha	1 1/2			145 - 446	.7972 46° API	
2	Barometric	9	4 1/2	33-34@ 100°F	400 - 680	.8602 33° API	
3	Light Sidestream	36	8	100 @ 100°F 39.1 @ 210°F	680 - 792	.8735 30.5° API	350
4	Heavy Sidestream	28	8	222 @ 100°F 47.6 @ 210°F	792 - 900	.8789 29.5° API	430
5	Bottoms	22	Dark	284 Sayb. Furol @ 122°F	900 - up	.9937 10.9° API	
Residue	Water Loss	3.5				1.0 10° API	
		Total 100%				Av. S.G. Feed Stock .9035 25.1° API	

Table 3.

The naptha and barometric fractions may be used as solvents, thinners or in the blending of fuel oil and diesel fuel. The light and heavy sidestreams can be considered to be for various products such as lubricants, diesel fuel, rubber extenders or as vehicles in paints, coatings and sealants, and as plasticizers in the plastics industry. The goal is to provide extensive pretreatment so the bottoms will contain negligible residues of additives as well as colloidal matter (mostly carbon). In this way, it can be used as a fuel oil without deleterious effect or need for mixing or monitoring. Alternately, the bottoms may be further cleaned for use as lubricants or any of the above-mentioned compounding applications. Their higher viscosity and flash point make them well suited as heavy blending components.

Analysis of the first four fractions shows the complete absence of particulate matter and metal ions. However, residual odor and color are present. These properties appear generic to olephines and asphaltines. These contaminants are presently removed by an adsorption process known as "clay treating." However, in order to avoid the ensuing clay waste, we propose to explore the use of solvent extractions and column chromatography.

The ecological error in clay treating is best illustrated by the fact that upward of 1/8 lb. (approx. 50 grams) of clay are used per gallon of oil in order to remove less than 1 gram of odor and color-carrying organic material, which could be easily incinerated were it not for the inorganic adsorbent.

Further investigation relating to odor and color in the distillation fractions led to the development of solvent extraction as a non-polluting, non-residue producing method in place of clay treatment. It was found that selective solvents for olephines and asphaltines, are capable of pulling these contaminants, as well as water, out of the oil. The dirty solvent is then separated by decanting and recovered by distillation (solvent recovery).

Hydrogenation presents an alternate approach to the above-mentioned methods, and its ability to change the offending organics into aromatics is not seriously questiond. However, the economic viability of hydrofining vs. extraction is open to question. Also, the equipment needed to carry out this process is of necessity expensive, complex and not generally available in small refineries.

A Discussion of Results and Recommendations

Producing several petroleum fractions having discrete boiling point and viscosity ranges may prove to be quite unnecessary for a successful product development program.

In this connection, one may question the need for distillation, except as an established economical fractionating means. Since it appears quite feasible to remove the additives, particulate matter, as well as color and odor carriers by emulsification, filtration, solvent extraction, electrophoresis, membrane dialysis, coagulation and centrifugation or settling, the case for distillation is not clearly established. In fact, there is a tempting notion that it should be possible to tailormake a process for the disposal of a variety of industrial waste oils by combining several of the above methods.

A further process alternative is suggested for consideration. The distillation of incoming lube stock, without pretreatment, results in an accumulation of additive and particulate matter in the bottoms.

These could be ignored altogether, as a potential source of revenue, and incinerated. Such a notion becomes even more attractive if the quantity of this fraction is reduced from 22% to 10% by suitable changes in the distillation temperature ranges.

Proposals solicited from THE ENGINEER CO. and THERMAL RESEARCH AND ENGINEERING CORPORATION evidence the availability of commercial equipment at reasonable cost where an incineration method is indicated.

If products can be found that do not need to be deodorized or decolorized after distillation, there emerges an extremely simple reclamation scheme requiring no pretreating. For the conversion to carbon black, oils need not be odor or color-free, but the admission of metallics from additives seems out of the question.

In order to make reclamation possible, all carbon and hydrocarbon matter must be separated from organic as well as inorganic contaminating particulate and additive material. This could be difficult. It may prove economical and perhaps even very profitable to produce industrial grades of carbon black from tank bottom residues. (See Product Creation below)

Finally, the exploitation of additives promises a source of revenue to be investigated. Our research has shown approximately 2-3% by weight of "dry mud" can be collected from the incoming waste oil. This is equal to roughly 9 lbs. per barrel (approximately 42 gal. or 360 lbs.). This material represents metals primarily in carbonate and phosphate form which can be extracted.

We have no knowledge about the economics of such a process. However, at worst, incineration can yield a clean landfill, abrasives and anti-skid coatings. From the organic state, fertilizers and salts for ice and snow removal may be precipitated.

From the foregoing, it is clear that regardless of this last reclamation phase, it is necessary to complete the clean-up and separation of the incoming waste oil into compounds which may logically lead to further exploitation. We hope to be able to show that this can be accomplished profitably and without violence to our environment. At any rate, incineration is generally an exothermic process from which heat can be realized for the refinery operation.

In summary, the NORCO process is sufficiently effective and flexible that with new and added equipment, it should be able to recover over 90% of the collected waste oil, without residual waste and at a lower cost than existing methods allow. Moreover, it utilizes existing process equipment and is, therefore, not dependent on technological breakthroughs.

Product Creation

If this project is continued, it is probable that the NORCO refinery products will be upgraded to a market potential to be sold competitively as petrochemicals to the paint-sealant, plastics and rubber industries. As explained elsewhere in this report, extensive investigation of the foregoing possibilities reveals that these potential markets have never been tapped by the re-refining industry. The added advantage of the use of re-refined products in these potential market areas is that the re-refined lubes will not return to haunt the environment as a waste lube oil, as it will be totally consumed in these petro-chemical uses. The present re-refined lubes which are primarily for automotive uses do come back to pollute the environment as waste lube oils.

The rubber, paint-sealant-coatings, plastics, carbon black and special fuel industries can absorb more than the entire annual crankcase waste oil accumulation in the United States and, if the know-how to produce petro-chemicals from waste crankcase oils can be demonstrated, these industries would be the most logical market for the re-refiner to consider.

Operational Analysis of the Projected Refinery

The proposed process may be carried out in a typical re-refining facility capable of processing 1,000 barrels/day (42,000 gallons).

Collection Costs This is a maximum figure and does not include allowances for BS&W (10-15% of the collected waste).	3¢/gallon
Wages Based on 3 shifts, 3 men per shift @ $50/day, including workman's compensation, social security and fringe benefits.	1¢/gallon
Utilities, Maintenance and Repairs	1¢/gallon
Overhead, Management, Insurance & Sales Costs	1¢/gallon
Depreciation, Contingencies, etc.	1¢/gallon
Total Operating Costs (not including pre-treating or special extraction steps	7¢/gallon

Oil Collection Data

The independent collector, probably the main supplier of most

re-refiners, can gather about 3,000 gal/day. He is paid 2-3¢/gal. by the service stations for removal, plus 3¢/gal. at the refinery. His daily receipts, therefore, amount to approximately $180.

The proprietary truck fleet operator can expect to collect about 3,000 gal/day/truck at a cost of approximately $100-125/day/vehicle.

Availability and geographic limitations indicate a practical collection radius of 50 miles (7,500 square mile area) for direct truck pickup and delivery to the refinery ('A' below), over 100 miles (37,500 sq. mi.) if trailers are used to pick up the oil from collection storage depots ('B' below).

Under 'C' below, we attempt to show how a truck can successfully cover a collection route of up to 150 miles (60,000 sq. mi.) radius over several consecutive days of operation, or involving the use of a fleet of several trucks concurrently.

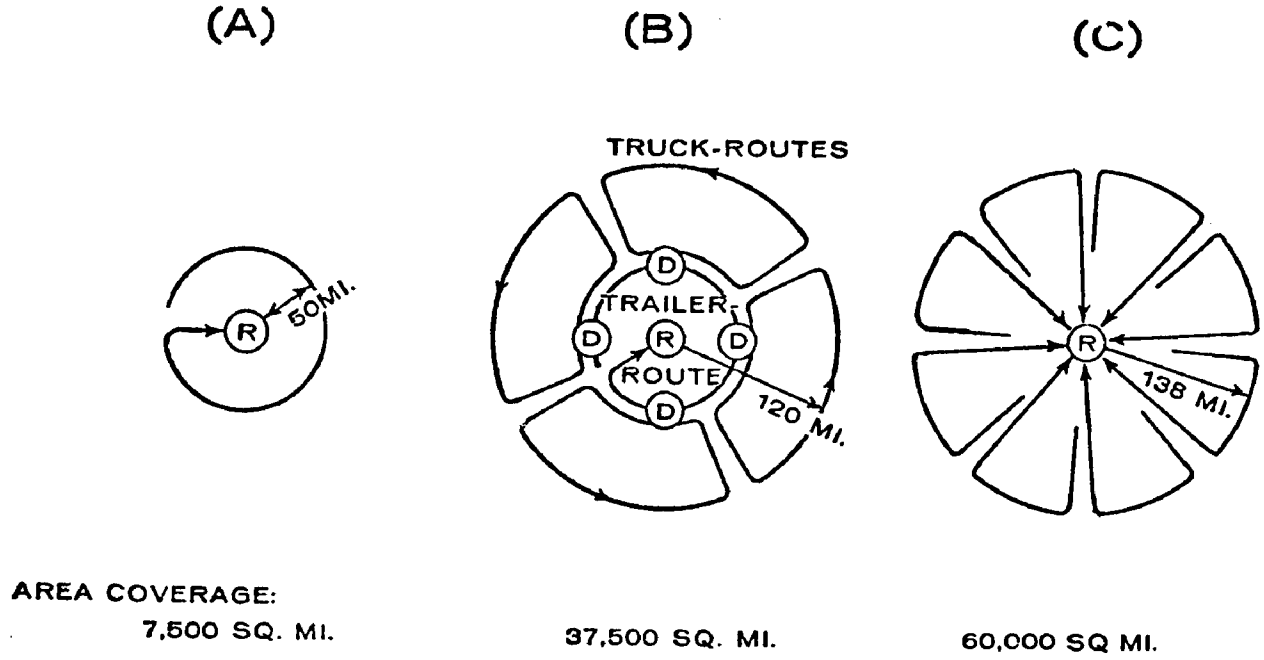

Figure 3.

LABORATORY

OF

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October 16, 1970

INTERIM REPORT - ANALYSIS OF SAMPLES #8U and #9U:

The following report is a partial one, pertaining only to the principal metallic constituents of these two samples.

#8U - marked "Bottom no additive" is one of a NORCO distillation series.

Analysis:

A) 1) microscopic - by polarizing interference microscopy
2) michrochemical
3) microphysical (magnetic, hardness, density, etc.)

The following are particulates in approximately decreasing order (as to estimated total particulate volume).

a) Abundant (accounting for over 99.9 percent of the particles and about 96 percent of the particulate volume - other than carbon)
Ca (1) as various phosphates
(2) as carbonates, rarely sulfates
Ba (1) as carbonates
(2) much less as phosphates, etc.
Pb (1) as the oxide
(2) much less as the sulfide, etc.
Fe (1) as hematite
(2) as magnetite; OH, CO_3, etc.
Zn (1) as the oxide
(2) much less as sulfide, carbonate, etc.

b) Intermediate
Mg (1) oxide, phosphate, carbonate, rarely sulfate
Al (1) oxide, silicate
Na (1) silicate, oxide
Cr (1) oxide, inchromate
Sn (1) oxide
Cu (1) oxide, sulfide, carbonate

c) Rare (less than 0.5 percent)
Nl (1) oxide, phosphide, sulfide
Mo (1) present both as a metal and in the Molybdate radical

Note: occasional particles of free metal - also present

B) Emission Spectrographic Analysis, etc.

Ion	mg percent		Calculated wt/wt percent (to total ash)	
Ba	892		16.0	
Ca	852		15.2	
Pb	380		6.8	
Zn	140		2.5	
Fe	140		2.5	
Mg	64		1.2	
Al	52		.9	
Na	42		.8	
Cr	21		.4	
Sn	16		.3	
Cu	13		.2	
Ni	9		.06	
Cd	3		.05	
B	2		.04	
Mn	2		.04	
Sb	1		.02	
Cu	1		.02	
K	1		.02	
In	0.2		.004	
Ag	0.1		.002	
	2,631.3	equals 2.6%	approx.	46%
P	279	(as the oxide and in phosphates)		5
Si	12	(as oxide and in silicates)		.2
Mo	8	(as methal and in molybdate)		.15
S	793	(as S, H_2S, oxide and in sulfides and sulfates)		14.0
	3,723.3	equals 3.7%		approx. 65%

Total ash (carbon free) equals 5.6 percent.

NOTE: 1) O, H and C in inorganic radicals equals about 1.9 percent.

2) Approximate ratio of metal to nonmetal probably about 2.8: 2.4 indicates phosphates and carbonates predominate over oxides.

3) It is likely that almost all (certainly over 90%) of the above metals are particulate. This should be investigated.

4) Dissolved metals are probably Na, Ca, and Ba.

#9U - This sample consists of hard, dense, coarse, irregular masses of a mineral-like conglomerate; predominately gray with reddish and brownish streaks; the surface powders readily with hardness appearing to range from about 2-4 (MOHS); the core and some streaks leave sizeable particles (up to about 2-3 mm) ranging from about 4-8 (MOHS).

This sample is the residue from the burning of a sample similar to #8U in an industrial boiler.

Reference A)-Microscopic, etc. (as for #8U above) - examination ground particles - reveals an extensive array of inorganic mineral compounds, and suggests formation in layers at highly variable temperatures and oxygen availability (outer layers, for example, show C_2O; inner layers, $CaCO_3$; $CaCO_3$ converts at 900°C.

Free carbon is negligible, consisting of less than .01 percent of the particulate volume; no free S is detected.

Oxides are considerably higher than in #8U; sulfides are much lower than in #8U.

Principal constituents: Ca - phosphate (various); $CaCO_3$, $BaCO_3$, Ba - PO_4 (various) PbO, PbO_2 (indicating low temperature areas). PbS, Pb in complexes, e.g. (spinels); Fe_2O_3 (hematite); Fe_3O_4, FeO; ZnO, ZnS.

Others: P, P_2O_5; MgO, $MgSO_4$; Al (as oxides, silicates, spinels and conjugates) Cr_2O_3; SnO; CuO; Cus: various Na salts; Si - O.

Rare: Sb, Ni, Mn, Cd, Co. - various

At least 65 different compounds have been identified.

Note that a given mineral array will depend not only on the initial sample, but also on the temperature and available oxygen.

Reference B) Emission Spectrography

	Percent wt/wt
Ba	13.8
Ca	26.3
Pb	6.8
Zn	3.9
Fe	5.7
Mg	1.6
Al	1.4
Na	.7
Cr	.5
Sn	.6
Cu	.3
Ni	.9
Cd	.2
B	.03
Mn	.13
Sb	.03
Co	.08
K	.17
In	.02
Ag	.02
Approximately	63 percent
P	9
Si	.6
Mo	.05
Approximately	73 percent

C, O, H and S are approximately 27 percent, indicating that oxides are higher by comparison with #8U, i.e. (mean molecular wt. of non-metals has dropped).

COMMENTS:

A) Apparent discrepancies between the percentages calculated for the two samples may be due to the following:

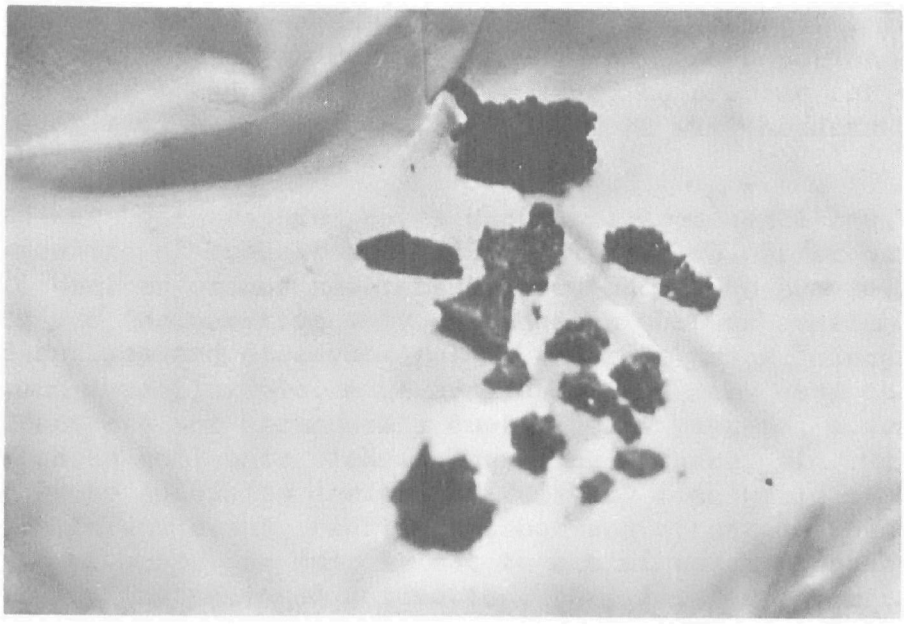
1) The samples are not from identical sources.
2) "Bottom" samples will vary with time of sampling, i.e. (temperature, oxygen, etc.) and sampling techniques i.e. (mixing). For example, particles of greater weight and/or density settle out faster, while total ash determination accounts for particulates and solutes both.
3) "Residue" samples will similarly vary with location of sample with respect to temperature, oxygen, updraft gradients, etc.
4) Total ash (for either sample) varies with technique, i.e. (temperature, oxygen, etc.).

5) For #9U, some substances may be lost as volatiles and fly-ash particles.
6) Higher percentages, in general, are expected for #9U because conditions allow for greater initial oxidation.

B) It is likely that high oxygen, high temperature incineration would yield a higher percentage of metals, but oxides would predominate.

C) It is recommended that dilution-centrifugation and/or dilution-filtration techniques be investigated and, as the initial data seems to indicate, if recovery of metal particulates exceeds 90 percent of that found by ashing, such techniques should be used in preference to recovery from burning of "bottoms" because:

1) High metal concentration in the feedstock boiled for distillation will act catalytically in a number of indefinite and uncontrolled reactions.
2) High metal concentration would tend to poison, perhaps irreversibly, most of the adsorbants that could be used at any step in recovery, i.e. (removal of odor or color by various "clays").
3) Metals obtained by dilution-centrifugation and washing would be in a form that would probably be more easily recoverable from their compounds, i.e. (CO_3, OH, S SO_4, etc.) than from the highly oxidized forms.



Hard Rock Clinkers
Figure 4.

SECTION X

SPECIFIC RECOMMENDATIONS

The following are detailed and specific recommendations based on information gained during NORCO plant operations in refinery runs 1-11, on conferences held with vendors and their engineers, and on a survey of the available subject literature.

Furnace

A new furnace design is required which will be more efficient and require less maintenance. Furthermore, the pipe still heater for waste crankcase oil processing should be designed to charge cold oil directly from a mixed feed tank to a large convection section at relatively low velocities, which can be increased with increases in temperature. Tubes should be equipped with headers for cleaning. Variation in charge feed and unknown future possibilities rule against closed furnace coils and steam-air decoking without considerable plant experimentation and development, both of which are costly.

Preheat, by heat exchange with product streams, at this stage of waste oil plant development, should stand on its own economic feet, and not be thrown into process development. If recovery of heat from products is thought to be economic, it might first be applied to generation of steam, usually required for treating sludges, emulsions, BS&W, and for firing furnaces, burners and incinerating wastes. Steam is often produced from waste heat in petroleum refineries where there are fewer unknowns.

The main furnace for vacuum distillation should provide for fairly high mass velocities in furnace tubes, and the reduction of mass velocity just ahead of the transfer line, to provide for considerable vaporization in tubes. Tubes equipped with headers and removable plugs are again advisable.

The radiant heat transfer rates should be kept quite low to avoid quick fouling of tubes, say at 5,500 BTU per sq. ft/hr on outside areas. Whether or not separate heaters should be provided for each heating and incinerating duty, depends on what is available. There are advantages and disadvantages in separate units. Separate furnaces are more flexible, more available, and many even cost less since they are not necessarily custom jobs, whereas, a combination two or three coil unit with incinerating burners, etc. will almost certainly be specially designed. Delivery time would probably be longer. In any event, handling, labor and attention required on the part of operators must be cut to a minimum, but not by attempting heavily instrumented automation; that is, for major refineries.

Special attention must be paid to collecting, transporting and firing the difficult waste materials which are incinerated. Also,

attention must be paid to removal of particulate metallic dust and solid matter from flue gasses, and transporting of incinerator dust and solid matter to avoid nasty, dirty, depressing surroundings, characteristic in the past of re-refining. Shoveling, scraping, moving with a wheel barrow, clearing of plugged lines, sweeping, thawing out lines and wiping, all have one common denominator--they all cost too much. They are to be avoided by installing sloped bottom and cone bottom tanks, proper drainage systems for tankage, vessels, pumping, slopping, shutdown and startup draining, and all the little nasty, time-consuming details required to handle the difficult end of re-refining procedures. In the past this housekeeping end of re-refining has been shoved into the future by piling up clay, dumping into lagoons, or letting acid and oil seep into the ground, but the future has a way of pushing the stuff right back into the present. Apparently, the future has its own plans. The best time to work with the future, as regards housekeeping details, is when the experience of plant operations is being detailed into drawings.

Incinerator

An incinerator for the NORCO processing plant would have to accept the following products:

1. Tar, which is a special material because of stickiness and tensile strength.
2. Tank bottoms containing solids or semi solids.
3. Emulsions from tanks; liquid or semi-liquid.
4. Acid water from crankcase dehydration, or neutral water containing oxygenated hydrocarbons.

It would have to have special burner, transportation and exposure arrangements for solids, and it would probably be similar to a cement kiln for handling solids and semi-solids, and be provided with special burners for emulsions and tars, plus be provided with spray-in devices for phenolic and acid water.

Conveying arrangements for getting solids into the incinerator and removing them from the incinerator would be required.

Satisfactory particulate matter removal equipment would be required for flue gases with suitable solids handling conveyors, and a bin or bins, plus associated equipment, required for transporting the solids into a tank truck.

Considerable shopping around and after-purchase debugging, or development, would probably be required to produce a satisfactory incinerator calling for an economic amount of attendance.

Air-Cooled Coolers and Condensers

Air-cooled coolers and condensers should certainly be tried out as the cooling required permits relatively high outlet cooler temperatures, which air coolers economically provide. The greater part of the cooling load would not require cooling below 140°F, while 160°F might be acceptable. Dehydration and removal of naptha, if diluted feed or bottoms are centrifuged, can readily be performed at 800 MM Hg down to 200 MM Hg at condenser coutlet temperatures of 120-130°F. If economic, a small refrigerated cooler might be installed before the suction of the motor-driven vacuum pump - if dehydration and naptha evaporation is not performed at 800 MM.

In the case of the Vacuum Fractionator, if operated at 5-10 MM, a small refrigerated cooler, installed before the inlet of the motor-driven vacuum pump, would be desirable. Minimum economic temperature in an air-cooled condenser outlet would be 105°F in hot weather.

A small refrigerated condenser would be practical for reducing the temperature further - to about 40°F. The heat quantity involved is small. The control specifications, as furnished by a large air-cooled cooler manufacturer, show clearly that the manufacturer is well aware of the need for simple rugged instrumentation for air coolers in duties where instrument maintenance service is not economically available.

This manufacturer has also produced skid mounted, modular prefabricated cyrogenic processing plants for natural gas processing, with one man in operating attendance in the gas fields.

Available in chemical and petroleum processing technical magazines, are reports on the economy of operation of air-cooled coolers, including variations in very hot areas with ambient temperatures of 100-110°F, where air is economically cooled by evaporation of a minimum amount of water to provide product outlet temperatures at 100°F.

Vacuum Pump

Steam actuated vacuum jet pumps are not desirable for a waste crankcase oil plant. A three-stage installation, with two intercondensers and a final after-condenser, would be required for a 5-10 MM vacuum. High pressure, 90-100 psig steam would be needed. Water treatment, licensed boiler attendance, blow-down disposal to avoid pollution, feed water testing, etc., would also be required.

Motor-driven vacuum pumps which will operate at 5 MM Hg are available. We recommend installation of such a pump to replace steam jet vacuum pumps.

Fractionation

As it is planned to provide some head-end treatment to remove the suspendid solids prior to processing, it is extremely difficutl to know exactly how to proceed with fractionation arrangements. Until the apparatus and process for dealing with the tar problem are more fully developed, the fractionation unit process will have to be somewhat flexible. At best, this unit process will require relatively extensive development and modifications. For example, at the moment, the best arrangement would be to go ahead with the transfer line from the Fractionator Furnace discharging into an efficient cyclone, at about 5-20 MM Hg. Vapor coming out of the cyclone goes to a series of air cooled partial condensers with horizontal tubes. If we find cases of severe trouble with fouling, the tubes might be pitched down or placed vertically. In case of no fouling, tubes could be pitched up or vertically, providing liquid refluxing and probably the equivalent of a fractionating tray. At low absolute pressure, and high differences in relative volatilities of compounds, the fractionating effect of a fractionating tray or partial condensation is greater than at higher pressures.

It should be noted that the existing Vacuum Fractionator heavy product section is provided with two trays located above the cyclone separators, both six feet in diameter: the bottom bubble cap tray, provided with a liquid drawoff pan; and a glitsch ballast top tray. Circulating reflux is fed to the top tray and reflux and products are drawn off the bottom tray. The total fractionating effect is probably about like that of a partial condenser.

The Vacuum Fractionator light product section is provided with three spray decks, each four feet in diameter, over which circulating reflux passes. Circulating reflux is fed to the top spray deck and circulating reflux, plus light product, are drawn off a catch pan below the bottom spray deck. The fractionating effect is obviously limited, probably not greater than that of a partial condenser.

Waste crankcase oil contains substantial amounts of solids. Additive suppliers say the additive materials break down to yield solids in the range between 400-700°F. At 700°F, breakdown is rapid and substantially completed within a few minutes. The effect of presence of NaOH with exposure to temperature is not fully known. It is known, however, that crankcase oil which has been diluted and centrifuged, and vacuum distilled at temperatures between 600-700°F does not yield a fair amount of additional solids. Some investigation indicates this is largely carbon. Further work would be required to predict whether future air quality standards require removal of this material to meet heavy fuel oil specifications.

Pumps

Rotary positive displacement pumps of several types are recommended for use with cold and warm waste crankcase oil, and also with the bottoms stream. Very stiff bottoms requires short suction lines.

Rotary pumps stick and jam on even small quantities of the tar produced in the Vacuum Fractionator or strippers.

Centrifugal pumps, with suction lines under the vacuum are recommended for use on hot light and heavy products, and also on reflux containing tarry material. The lantern gland in the hot oil stuffing box should be provided with a supply of cold oil under a slight positive pressure to avoid air leakage into the pump suction. Adequate piping for venting and discharging back to the suction vessel should be provided to get and maintain flow, particularly when starting up the operation.

Centrifugal pumps also do well on light and heavy products being pulled from storage tanks.

The present NORCO Fractionator bottoms pump is an all steel, side pot valve simplex steam reciprocating pump. There is very little suction head available. While the pump runs reasonably well, a variable speed motor-driven reciprocating hot oil pump, to save on steam, would be preferable.

Other Specific Recommendations

The results of the completed studies indicate that the quality of the products and bottoms can be increased if the suspended colloidal and dissolved organics and metallic compounds can be removed prior to processing. Accordingly, treatment and/or physical removal equipment is required at the charge stock end of the process system.

Present lube distillates are dark and emit a slight odor. Double distillate runs indicate that the recommended head-end handling will increase the quality of the product streams and the color and odor specifications of the products. Diesel runs should be included to unify the quality of the light product and its effect on diesel engines.

Removal of the polymers and metallics at the head-end should produce a bottoms product with an acceptable solids level and with minimal sulphur to make it compatible with burning equipment. Controlled burning tests should be conducted to determine if any limitations on the use of the bottoms exist.

Centrifugation, solvent extraction, chemical treatment equipment or a combination of these concepts should be installed in the head-end of the plant.

The head-end treatment will permit greater furnace temperatures. This design will also mitigate coking, permit greater extractions of product and longer, more reliable runs.

Provide a furnace with optimum flow velocities through the tubes, thus avoiding coking and erosion and resulting in an increase in percentage of product stream recovery as well as increase 'on stream' time and reduce 'down time.'

Provide a plant waste converter to condition solids, emulsions and all plant wastes into materials meeting pollution standards.

Install a motor-driven mechanical high-vacuum pump to reliably maintain the vacuum required which will increase the quality of the sidestream products and improve the length of runs. This would also eliminate the high pressure boiler apparatus and a costly licensed engineer.

Replace existing water-cooled condensers and coolers (to eliminate water cooling from plant operations, as present water quality standards are difficult to meet) with new air-cooled condensers and coolers or circulating water cooling systems. Some would meet national refinery concept criteria--as inland refineries need not rely on hard to obtain water sources.

Conduct runs necessary to determine optimum flow sheet conditions.

Subject products produced to use testing so as to accumulate testimonial data for market acceptance of "special fuel" and "special lube" products. In this regard, a close affiliation with an accepted petroleum oriented testing laboratory is recommended.

SECTION XI

GENERAL RECOMMENDATIONS

Crankcase disposal plants have, in the past, been catch-as-catch-can, makeshift, haphazard affairs. In the design of future plants, attention should be paid to details, such as:

1. Topography and geology of the selected site, to avoid underground movement of liquids.

2. Grading for drainage, prevention of fire hazards, losses by leakage from tank or personnel failure.

3. Surface water drainage, collection, separation, treatment and final disposal. Loading and receiving arrangements for good housekeeping and low fire hazards.

4. Design of vessels and tanks for economic settlement and transportation of B.S.W. sediment and all other waste materials.

5. Collection and disposal of vapors, fumes, flue gases, etc.

6. Placement of drainage sewer lines to avoid freezing, permit economical cleaning, etc.

7. Fire protection criteria for this scale refinery should be developed for operation by one or two men, with provisions for access by fire department equipment. A guide list should also be developed such as used in petroleum refinery or chemical plant design which would serve as a practical designer's reminder list. API publications and Chemical Industry publications would also be helpful.

SECTION XII

APPENDICES

A. Preliminary to Startup	Done	When	Who Initial
Mix feed tank			
Complete all lubrication items			
Complete all instrument air items			
Clear all level glasses and connections			
Clear all stripping steam distributors			
Clear all suction lines			
Clear gland oil connections to pumps			
Fill gland oil tank			
Clean all burner fuel screens			
Clear all pump casing vents			
Steam blow & clear light & heavy reflux coolers, lines, and control valves. Repair, fix leaks			
Steam blow & clear light & heavy reflux coolers, lines, and control valves. Repair, fix leaks			
With top fractionator manway open, circulate light reflux. Make repairs or clean as needed to pass inspection			
With both fractionator heavy cut section manways open, circulate heavy reflux			
Make repairs or clean as needed to pass inspection.			
Clean all strainers on list			
Pressure test Flash Furnace at 30 psig with steam			
Pressure test Fractionator Furnace at 50 psig with steam			
Drain all items on drain list			
Check vacuum pumps as required			
Close unit			
Put boilers on H.P. steam			
Close cooling water system			
Fill gland oil tank on cooling water pump			
Start cooling water pump			

Preliminary to Startup (continued)	Done	When	Who Initial
Put steam test on unit & repair leaks as required .			
Drain all items on unit drain list			
Test Flash and Fractionator Vacuum pumps against shut suctions. Repair as required. Get 29.2" vacuum			
Open stripping steam line; hold 12 psig steam pressure against stripping steam distributor valves; crack steam drains at bottom of Fractionator on 3" line, and at end of 3" line at heavy cut stripper			
Put vacuum test on Flash Tower and Fractionator; repair as required. Get 28.9" Hg vacuum			

B. Startup Instructions	Done	When	Who Initial
Establish flow from Feed Tank through Flash Furnace and Tower			
Establish flow from Flash Tower through Fractionator			
Establish flow from Fractionator Bottom to pump, cooler and feed tank			
Check over system, note leaks and other items . .			
Stop feed flow as required, repair leaks and other items			
Re-establish feed flow through Flash Tower and Fractionator as required			
Fill light & heavy reflux tanks & light settler .			
Establish flow through light & heavy make coolers and note repair items			
Repair light and heavy reflux flow as needed . . .			
Check over light & heavy reflux systems and note items			
Stop light & heavy reflux flow; make repairs as needed			
Re-establish light & heavy reflux flows and leave flowing			
Start cooling water flow to pipe coils in box coolers			
Adjust Flash and Fractionator Furnace dampers; leave stack dampers wide open			
With feed flowing through unit, start Flash Furnace burner			
With feed flowing through unit, start Fractionator Furnace burner. After fire is established, start blower discharging up stack. Gradually get flue gas recirculating, and stop discharging to stack			

Startup Instructions (continued)	Done	When	Who Initial
When Flash & Fractionator Furnace transfer lines are up to 180°F, start stripping steam into bottom of Flash Tower, bottom of Fractionator, and into expansion joint in Fractionator Furnace transfer line. Keep raising both transfer line temperatures			
Adjust flow of feed to unit at rate specified in log book instructions			
Bring up Flash & Fractionator transfer line temperatures as fast as possible, maintaining light & heavy reflux flows to hold specified Fractionator top temperature			
Increase stripping steam flows as transfer line temperatures go up, unitl the specified stripping steam is flowing into Fractionator bottoms and Flash Tower			
When levels in Light & heavy cut strippers begin to build up, line up flow and discharge to slop tank			
When Fractionator bottoms reach specified API gravity, pump bottoms to bottoms tank			
Put stripping steam flow specified in log book into light & heavy cut strippers			
Keep close watch on Flash and Fractionator bottoms levels			
When Fractionator light & heavy cuts from strippers reach specified gravities and colors, pump to light & heavy cut make tanks			
Now hold operating temperatures, flows, pressure vacuum, etc. steady; vary only to maintain product streams as specified in log book			
Keep barometric oil accumulating in oil-water separator skimmed off and pumped into barometric oil tank			
Keep water drained off barometric oil tank			

Startup Instructions (continued)	Done	When	Who Initial
Every two hours, look into furnace fire boxes for hot spots (black spots) on furnace tubes or coil. If found, adjust burner, dampers, feed flow, steam into feed going through furnace tubes to maintain furnace inlet pressure, and reduce black area on tube; avoid operating conditions that result in large black areas on furnace tubes. Note situation in log book. Do not operate with large sections of tubes black and overheated			
Hold 200-210°F temperature at the bottom of the Flash Tower at 19.0-21.0" Hg vacuum			

C. Instructions on Controlling Product Specifications

Regulate Naptha, API gravity, end point and yield by adjusting the Flash Furnace transfer line and the Flash Tower stripping steam flow and vacuum. Usually, the Vacuum jet pump will be left with the steam flow to the pump set, and not changed except under unusual circumstances.

Regulate API gravities, viscosities, flash points and yields of bottoms, heavy cut, light cut and barometric oil by adjusting the Fractionator Furnace transfer line, the Fractionator vapor temperatures with light and heavy section vapor temperature controllers controlling flow of heavy reflux, light reflux and steam to light and heavy strippers.

Note: Hold stripping steam flow to light and heavy strippers steady by putting pressure controller on stripping steam in operation.

Change	Results, Assuming One Change at a Time
Flash Tower	
Raise flash furnace transfer line temperature.	API gravity of Naptha decreases. End point of Naptha increases. Flash point of barometric oil increases. Viscosity of Naptha increases. API gravity of barometric oil tends decrease. Viscosity of barometric oil tends to increase if fractionator conditions are steady. Initial boiling point of barometric oil increases. Flash point of barometric oil increases.
Increase stripping steam flow (the stripping steam flow is adjusted mainly to control the flash point of the barometric oil).	Flash and initial boiling points of barometric oil will increase. All other specifications will change as above, but only slightly, unless change in steam flow is large.
Increase vacuum on flash tower.	Same as for raising Flash Furnace transfer line temperature.
Fractionator	
Raise Fractionator transfer line temperature.	API gravity on all products: Bottoms, heavy cut, light cut, barometric oil, goes down. Viscosity on all above products goes up. Flash point on all above products goes up. End point on all products goes up. Initial boilpoint on all products goes up. The yield of bottoms decreases.

Increase vacuum on Fractionator.	Same as above. However, vacuum pumps are usually operated to produce maximum possible vacuum.
Increase stripping steam flow to bottom of Fractionator, mainly to reduce the yield of bottoms, but also to raise the flash point and incidentally lower the API gravity of the bottoms and raise the viscosity.	The bottoms specifications will be most affected. Gravity goes down. Yield goes down. Viscosity and flash point of bottoms go up. Heavy cut, yield, viscosity & flash point go up; API gravity goes down. Light cut yield goes up some; API gravity goes down; viscosity and flash go up. Barometric oil yield goes up some; gravity goes down. Viscosity and flash point go up slightly.
Raise vapor temperature above top heavy reflux bubble tray (hvy. reflux flow is reduced).	Bottoms API gravity goes down somewhat. Bottoms yield goes down somewhat, while bottoms viscosity and flash point tend to go up. Heavy cut yield and API gravity go down. Viscosity and flash point go up. Light cut yield, viscosity and flash point go up and API gravity goes down. Barometric oil yield, viscosity, flash point and API gravity remain unchanged.
Raise vapor temperature above top light reflux bubble tray (lt. reflux flow is reduced).	Bottoms are unaffected. Heavy cut yield and API gravity go down very slightly, while viscosity and flash point tend up. Light cut yield and API gravity go down; viscosity and flash point go up. Barometric oil yield, viscosity, flash point, initial boiling point and end point go up, while API gravity goes down.
Increases stripping steam flow to heavy cut stripper.	Bottoms are unaffected. Heavy cut flash point goes up. Heavy cut viscosity goes up very slightly, but heavy cut gravity and yield go down very slightly. Light cut flash point and viscosity tend to go up slightly, but light cut gravity and yield change very little. API gravity goes down very slightly. Barometric oil end point goes up slightly, while API gravity goes down very slightly.

• Increase stripping steam flow to light cut stripper.	Bottoms and heavy cut are not affected. Light cut flash point goes up. Light cut viscosity goes up very slightly, while light cut API gravity and yield go down very slightly. Barometric oil yield goes up very slightly.

Limitations

The flow of stripping steam that can be used is limited by the pressure drop of the vapors going through the Fractionator, and the tendency of excess steam to blow oil out of the stripper or out of the light cut section, over the top of the Fractionator. Steam cost is also a consideration.

The flash and Fractionator Furnaces transfer line temperatures are limited by the tendency of furnace tubes to overheat. It is very difficult to insure even tube metal temperatures where an oil burner flame radiates heat to tubes in a poorly designed furnace. The quantity of heat which can be absorbed by oil going through the tubes is limited by the coke depositing tendency of the oil going through the tubes. A build-up of coke inside the furnace tubes causes the tube metal to overheat. The ash dust, normally present on the outside of the tube, melts, causing the side of the tube which is exposed to the radiation from the burner flame to become black as the tube overheats. When black spots and areas show up on tubes, the transfer line temperature and/or the feed flow rate must be reduced to avoid having the tube split, permitting oil to leak into the fire box. Turbulence in the oil passing through furnace tubes is maintained when the oil flow is reduced by opening the steam valve to pass more steam into the tubes. Normally, as the oil flow is reduced, open the steam valve into the oil feed line, into the inlet pipe to the furnace tubes, just enough to maintain the same furnace inlet pressure as before reduction of the oil flow into the furnace tubes.

To repeat, black spots appear on tubes in the furnace. Reduce oil feed to the furnace 5-10%, and furnace inlet pressure will drop. Increase steam flow into the oil feed to bring the inlet pressure back up to where it was before reduction in the feed flow. If black spots do not fade, reduce feed rate another 10%. Add steam to oil feed to maintain furnace inlet pressure. If black spots do not fade, reduce furnace transfer line temperature by 10-15°F. Notify supervisors, upon completion of above reduction of transfer line temperature. In case of a sudden onset of evidences of overheating, with reductions in flow and transfer line temperatures not effective, shut down furnace burners and proceed to shut down the entire unit.

D. Shutdown Instructions	Done	When	Who Initial
Stop flash and vacuum furnace burners			
Open wide, all furnace, stack and burner dampers . .			
Slop bottoms to feed tank			
Shut off heavy and light reflux			
Pump out heavy and light strippers and soaker to product tanks			
When cyclone deck temperature in Fractionator drops to 425°F, shutdown cold charge pump and flash tower bottoms pump			
Shut off all stripping steam valves to steam distributors at flash tower, heavy and light strippers, and bottom of Fractionator			
Close stripping steam block valves at 100 psig main .			
Open all stripping steam piping drains			
Pump out bottom of flash tower to slop tank			
Close all flash and vacuum furnace dampers			
Open wide 100 psig steam valve to vacuum furnace oil inlet. Steam through tubes for 30 minutes			
Open wide 100 psig steam valve to flash furnace oil inlet. Steam through furnace coil for 15 minutes . .			
With vacuum furnace tubes wide open, steam blow again for 30 minutes			
With flash furnace coil wide open, steam blow again for 15 minutes			
With tubes and coil of vacuum and flash furnaces wide open, continue steam blowing alternately for 30 and 15 minutes respectively, until you have blown through both furnaces four times			
Close off block valves on steam lines for blowing furnace tubes and coil at 100 psig. Steam main and open drains on blowing steamlines			
Open wide all air and flue gas dampers on both furnaces			
Leave wide open to allow furnace interiors to cool .			
Remove brick from manways into interior of both vacuum and flash furnaces			

Shutdown Insturctions (continued)	Done	When	Who Initial
Reduce steam pressure to vacuum jet pumps on flash tower to 50 psig and allow steam to backflow and bring both flash tower and Fractionator back up to atmospheric pressure. Then close off steam to vacuum jet pumps			
Shut 100 psig steam block valves on lines to vacuum pumps. Open steam drains on these lines			
Pump Fractionator bottoms, heavy and light cut stripper and soaker as low as possible, through the pump and out the lines to the feed tanks			
Put steam boilers on low pressure operation			
Pump flash tower as low as possible to slop tank, through 1" direct line, pump slop tank to feed tank.			
Shut down instrument air compressor			
Open drains at instrument air tank, salt dryer, etc.			
Pump naptha from flash tower barometric oil-water separator tank to naptha tank			
Shut down salt cooling water pump, drain pump . . .			
Drain salt-cooling water strainers and piping . . .			
Leave drains open			
Drain water from cooling coil box			
Close block valve at main on steam line to bottoms pump and open drains on steam line			
Open steam cylinder drains on bottoms pump, rod through and leave clean and wide open			

E. Cleanup Instructions	Done	When	Who Initial
Open 3" caps on flash furnace coil and inspect. Clean as indicated by inspection			
Open 2 headers located on east side of vacuum furnace, in bottom row of tubes. When inspection of above indicates, open last two tubes before the transfer line and inspect. If these two tubes need cleaning, clean and proceed to open tubes from back of transfer line towards furnace inlet until tubes in upper section do not require cleaning			
In the lower tube section of the vacuum furnace, in all of the six rows, open the two tubes closest to the burner, inspect and clean as necessary . . .			
Open the last two tubes in each row, as the condition of the above two tubes closest to the burner indicates and clean as necessary			
Inspect vacuum furnace transfer line and clean as inspection dictates			
Open bottom manway of flash tower. Clean suction screen and bottom of flash tower			
Rod through flash tower level glass and float level instrument connections and blow them out			
Inspect stripping steam distributor, cleaning as required			
Inspect 4" suction line to vacuum furnace feed pump and clean if necessary			
Clean strainer before relief valve on vacuum furnace feed pump			
Plate-up bottom of flash tower			
Open all Fractionator manways, top to bottom, and clean as each are opened using dirty wiping cloths .			
Clean Fractionator shell, light reflux piping inside of Fractionator, light cut trays and light cut draw off pan and entry to 4" draw off line into soaker if necessary			
Inspect top 6' diameter tray, clean as needed as all valve discs must be clear and free moving . . .			
Inspect top 6' tray liquid downcomer, clean out the bottom of liquid downcomer and liquid entry onto next tray			

Cleanup Instructions (continued)	Done	When	Who Initial
Inspect under side of bottom 6' tray, clean vapor uptakes under bubble caps as inspection indicates. Also go down through top 6' tray and clean bubble caps as inspection requires			
Clean cyclone deck, clear cyclone deck drain pipe as necessary			
Remove 6 cyclone caps, clean cyclones inside as required			
Clean 6 cyclone drain pipes, remove as necessary to clean below drain pipes			
Remove coke from outside of 6 cyclones and Fractionator shell as needed			
Clean out tower shell below drain pipes			
Clear 3" drain pipe from cyclone section of Fractionator down into bottoms stripping section . . .			
Remove dirt around strainer at bottom of Fractionator			
Remove suction strainer at bottom of Fractionator and clean below strainer			
Remove cyclones section 3" drainpipe, and clean as necessary			
Inspect inlet of 4" section to bottoms pump. Note condition of 4" suction line. Put plug in 4" inlet end of 4" bottoms pump suction after cleaning, to avoid dropping dirt inside			
Clean inside of bottom shell of Fractionator . . .			
Clean stripping steam distributors			
Clean top of cyclone			
Inspect cyclone entry wear plate, report condition of same and repair as needed			
Clean bottoms level instrument connections			
Rod through bottoms stripping vacuum connection to manometer			
Rod through bottoms level glass connections, including loop inside the vessel			

Cleanup Instructions (continued)	Done	When	Who Initial
Position stripping steam distributors in bottom of Fractionator			
Replace 3" drain pipe from cyclone section of Fractionator			
Remove 2" pipe plug in 4" bottoms pump suction line, and inspect. If inspection of 4" inlet into 4" suction line and appearance at 2" pipe plug, and operation of bottoms pump all indicate need for cleaning 4" suction line, unflange line, remove center section and clean the whole line from the pump suction flange to the bottom of the Fractionator. Replace 2" plug and flanged section of pipe			
When light cut soaker, light cut stripper, and heavy cut stripper are cooled down, pumped out, and drained as low as possible, remove the following: a) 8" cover plate at bottom of light cut soaker b) 8" cover plate at bottom of light cut stripper c) 8" cover plate at top of light cut stripper d) 3" pipe cap on oil inlet line. Light cut stripper e) 3" reducer at bottom of 3" drain pipe on seal loop on 4" oil line into top of heavy cut stripper			
Remove tar and clean out bottom of light cut soaker			
Remove tar and clean out bottom of light cut stripper			
Inspect, also clean top of light cut stripper as needed			
Inspect 3" oil inlet line to top of light cut stripper and clean as needed			
Blow with air hose through above item and check for volume of air coming out at 8" cover plates opening at bottom of light cut soaker. If low, repeat the cleaning out process			

Cleanup Instructions (continued)	Done	When	Who Initial
Also, blow with air hose through 3" pressure equalizer line, back into light cut soaker and check for volume of air coming out at 8" cover plate opening at bottom of light cut soaker. Clean 3" equalizer line as necessary			
Blow with air hose through 6" vapor outlet line at the top of the light cut stripper and check volume of air coming out of top manway of Fractionator . .			
Rod through level instrument and level glass connections on light cut stripper			
Rod through level glass connections on light cut soaker			
Open 2-3" suction pipe plugs, also 4" cover on pot in 3" suction line to light reflux pump. Clean out suction pipe as required			
Air blow the heavy cut pump 4" suction line back from pump discharge into heavy cut stripper. Check flow at 4" vapor outlet line in Fractionator at manway at the top 6' tray, cleaning as necessary . .			
Air blow into 4" heavy cut stripper vapor outlet line, inside Fractionator, at top 6' Fractionator tray, and observe volume of air coming out of the bottom 6' bubble tray vapor uptakes under the bubble caps. Clean as needed			
Remove 3" reducing coupling at end of 3" drain nipple on 4" heavy cut seal loop, in the 4" heavy cut draw line and clean out the 3" drain nipple as necessary			
Air blow into the 4" heavy cut stripper vapor outlet line and out of the 3" drain nipple on the bottom of the seal loop. Observe volume of air, clean again if necessary			
Rod through Light cut stripper level glass and level control instrument connections			
Rod through heavy cut stripper (2) level glasses connections and the level control instrument connections			

Cleanup Instructions (continued)	Done	When	Who Initial

As is necessary, fill level glasses with "Red Devil" paint remover, and clean out level glasses on the flash tower, Fractionator drain oil section, Fractionator bottom section, light cut soaker, light cut stripper and the two heavy cut strippers .

Steam blow, following coolers with 2" steam blow lines, the heavy cut to the 2" outlet at the line to the rundown tank, the heavy reflux to the 2" outlet at the base of the Fractionator, the light cut to the 2" outlet at the line to the rundown tank, and the light reflux to the 2" outlet at the base of the Fractionator . . . . . .

After steam blowing the cooling coils, blow out the cooling coils with an air hose through the heavy cut to the outlet at the line to the rundown tank, the heavy reflux to the outlet at the base of the Fractionator, the light cut to the outlet at the line to the rundown tank, and the light reflux to the outlet at the base of the Fractionator . . . . .

When time allows, do the following to thoroughly clean out the cooling coils and maintain the required cooling effectiveness:

a) Freezing weather: First steam blow the cooling coils with 2" steam blow lines wide open to clear the cooling coils. Then leave the 2" valve on the steam blowing lines cracked open, to avoid freezing the cooling coil, and permit water to dissolve tarry material.

b) Above freezing weather: Repeat procedure as in a) above, but shut off steam completely and let cooling coil sit full of water.

In both cases, a) and b), before startup, blow out coolers with 2" steam blow valve wide open, and finish up by air blowing all coolers to remove water and dirt as completely as possible . . . . . .

The above instructions apply to the following coolers: Heavy cut, heavy reflux, and light cut and light reflux.

The above blowing procedure is based upon blowing experience and the following observations: The material which forms and precipitates out of the distilled oil liquid and vapor, in flow lines, cooling coils, bubble trays, vessels, etc., is a material of a density of approximately 1.2 to 1.3 specific gravity at 60°F.

Cleanup Instructions (continued)

It is dark brown or black in color, and settles out quite slowly in the rundown tanks, but more quickly in hot oil. It forms mainly in the liquid or semi-liquid state at the usual atmospheric temperatures, but also is found as a solid, occurring in solid particles bound together by a viscous, extremely sticky, tarry binder. Both the solid particles and the tar binder phases apparently enter the liquid phase and are fluid at 338°F, the saturation temperature of 100 psig. The tar is brittle at low temperatures, showing a glossy surface upon fracture.

The tar or conglomerate of tar and solid material are both somewhat soluble in hot or cold water and the tar and solid particles slowly crumble into fine particles, readily entrained in a flowing stream of water. Both materials are soluble in other polar solvents, but relatively insoluble in various hydrocarbons. The tar or tar and conglomerated solids both form a brownish solution in water. The odor of the solution and dissolving material tends to change on standing, from a burnt, pungent acrid odor to a much more flat, earthy smell.

Cooling coils and piping tend to plug, but upon prolonged exposure to 100 psig steam, have gradually opened up. Once even a small flow is established, it tends to increase fairly rapidly.

	Done	When	Who Initial
With the air hose, back-blow the discharge of light cut pump into the light cut soaker; check air volume coming out of the 8" bottom opening. Clean the light reflux pump suction if needed			
With the air hose, back-blow from the discharge of light reflux pump into the light stripper; check air volume coming out of the 8" bottom opening. Clean the light reflux pump suction if needed			
Air blow gland oil lines to the light reflux, light cut and heavy cut pumps, cleaning as necessary . . .			
Clear cooling water lines to stuffing boxes of bottoms pump and heavy cut pump as needed			
Brush and blow dust off the flash furnace coil inside the firebox			
Brush and blow dust off the Fractionator Furnace tubes and header surfaces inside the five boxes. Brush and blow scale and dirt from all cooling coils in the cooling coil box; including, bottoms, heavy cut, heavy reflux, light cut and light reflux			

Cleanup Instructions (continued)	Done	When	Who Initial
Remove the 4" cap on instrument air dryer, check salt level in the dryer and refill as necessary. Brush thread compound into 4" pipe cap threads and replace			
Plate-up manways and 8" openings; including, bottom of Fractionator, drain oil section of Fractionator, bottom of light cut soaker, and bottom of light cut stripper			
Go over lines, pumps, etc., and close them, ready for pumping bottoms, light cut, light reflux, heavy cut and heavy reflux			
From the heavy cut make tank, fill to normal operating level, the heavy cut stripper, light cut soaker and light cut stripper			
Circulate light cut from soaker over the top of the Fractionator. Check flow over the light cut trays. Check the reflux control valve. Clean and repair as needed to get satisfactory flow and make the lines and cooler tight and the control valve operative			
Circulate heavy reflux from the heavy stripper over the 6' trays in the Fractionator. Check the flow over the 6' trays and the bottom tray for leakage, as well as make a check of the reflux control valve. Repair as needed to get satisfactory flow, bottom 6' tray tight, control valve operating, and lines and cooler tight, and with no leaks			

Warning: The bottoms cooler is a special case. Care must be taken to avoid getting any water into the bottoms in the three bottoms tanks. Water does not settle readily out of the bottoms because of the low API gravity and high viscosity. Customers complain of poor burner operation when even a very small amount of water gets into the bottoms tanks and product. It does not settle well.

The bottoms cooler does not foul quickly. It seldom needs to be blown with steam and air and does not require soaking with water to dissolve tarry material.

Avoid steam blowing the bottoms cooler during cold weather until shortly before startup. This prevents freezing-up and wasteage of steam. Careful! Never, under any circumstances, let any water get into any of the 3 bottoms tanks.

Cleanup Instructions (continued)	Done	When	Who Initial
Bottoms, Cooler, Steam, Air Blowing			
Blow bottoms cooler with steam valve wide open and cooler discharging to slopping tank line, until cooler is empty and oil and solid matter stop coming out			
Blow with air wide open, until all water stops coming out			
During startup or before, as required by other circumstances, establish feed flow through the plant into the bottom of the Fractionator, through the bottoms pump, cooler, and back to the feed tank. Hold feed at a maximum flow with bottoms pump keeping up, for 10 minutes to thoroughly flush water out of the system. The bottoms cooler is now ready for startup			
During startup, turn bottoms into bottoms tank when API gravity is down and cooler outlet temperatures are both within the range specified in the log book .			
Pump light cut from the light cut stripper, through the control valve, into the slopping tank. Clean as needed to get satisfactory flow, lines tight and control valve operating			
Pump heavy cut from the heavy cut stripper, through the control valve, and into the slopping tank. Clean as necessary to get satisfactory flow, lines tight, and control valve operating			
Start at top of Fractionator and plate-up three upper manways			
Plate-up the top of the light cut stripper and replace the 3" pipe cap on the oil inlet line			
Install a 3" blank in the suction to the Fractionator steam jet vacuum pumps			
Check over the whole unit and close up anything found open which should be closed			

F. Emergency Procedures

Drastic and immediate action which must be taken when there is little or no time for investigation or troubleshooting follows:

Electric Power Failure	Done	When	Who Initial
Open wide all furnace stack and burner dampers . . .			
Shut off power to both furnace burners			
Shut off both stripping steam valves at the main . .			
Open furnace doors into fire boxes, and push in the brick to let cold air inside			
Shut off steam flow to vacuum pumps			
Swing Fractionator bottoms product into feed tank .			
Pump Fractionator bottoms level low, with steam left in the boilers			
Pump Fractionator bottoms slowly. There is no flow of cooling water			
Blow down vacuum furnace tubes into Fractionator with steam left in the boilers			
Shut off the valves from the feed and the light and heavy cut tanks			
Go over electrical systems; including, circuit breakers, fuses, etc., and check for trouble. Shut off all starters until ready to start up again . . .			
Start up plant when ready, following the usual procedures			
Cooling Water Failure			
Stop both furnace burners			
Open wide all furnace stack and burner dampers . . .			
Shut off both stripping steam valves at steam main .			
Open furnace doors into fireboxes and push in brick to let cold air inside			
Shut off steam to vacuum pumps			

Cooling Water Failure (continued)	Done	When	Who Initial
Pump out bottom of Fractionator into feed tank . . .			
Reduce feed to flash furnace			
Keep feed going through furnaces until the vacuum furnace outlet temperature is 450°F			
Shut down cold feed and vacuum furnace feed pumps .			
When cooling water supply is normal, start up following the usual procedure			
Steam Failure			
Stop both furnace burners			
Open wide all furnace stack and burner dampers			
Shut steam to vacuum jet pumps			
Shut both stripping steam valves at steam main			
Open both furnace doors into fireboxes and push brick in to allow cooling			
Shut down cold feed and vacuum furnace feed pumps . .			
Shut down heavy and light cut pumps and light reflux pump			
Leave vacuum pumps running			
Leave cooling water running			
When vacuum furnace transfer line is down to 450°F, shut down cold feed and vacuum furnace feed pumps . .			
Bad Fire in Furnace			
Stop cold feed and vacuum furnace feed pumps			
Stop both furnace burners			
Shut off PROPANE to furnace burners			
Shut air dampers at furnace burners when not too hot .			
Spray water into firebox with fog nozzle			
Pump out bottom of Fractionator to feed tank			
Pump light and heavy cut strippers and light cut soaker to product tanks or slop, according to gravity.			

General Fire; Outside of Furnaces	Done	When	Who Initial
If possible, shut off material which is burning, i.e., oil, vapor or gas			
If that cannot be done safely, or the source cannot be identified, go back to tank valves and shut off all valves at all tanks, i.e., PROPANE supply, fuel oil to burners, naptha, Fractionator overhead, light cut, heavy cut and bottoms			
Shut down burners on both furnaces as well as the boilers, if practical			
Open all furnace stack and burner dampers, if the burners are stopped and it is possible to do so with safety			
Do not use water on areas traversed by electrical power lines			
Shut off main electric power switch as necessary .			
Shut down all pumps at individual starters, or at main electric power switch as necessary			
City water may be used as required for fire fighting			
The salt water pump is electric motor driven . . .			
When the fire is out or under control, do <u>not</u> attempt to start up again until careful inspection proves the repair or cause of the fire is completely adjusted, and there is <u>no</u> hazard in starting up again			

The foregoing instructions are action directed for maintanance of safety. They are, "Do it now, and we will talk about it later," directives. The objectives are given below in approximate order of importance:

1) Avoid injury to others and yourself.

2) Save the plant, or parts of the plant that can be saved.

3) Do not waste time on details. First get things under control and talk about it later, etc.

Small Fire in a Furnace

In case of smoking at one or several of the stacks, and the furnace outlet temperature goes up while burner flames go down, inspect the inside of the furnace carefully for leakage in a tube or header. If the flame leak is small and stays small, reduce feed flow through the furnace and shut down gradually as in a regular shutdown. Proceed according to what happens. Do not try to start up again if the fire goes out while shutting down. Shut down completely and examine the evidence. Make necessary repairs, evaluate the situation and then think about starting up again.

G. General Fire and Safety Instructions

Three things are necessary for a fire: fuel, air and ignition temperature. When a fire is going, shut off the fuel if at all possible, close all valves that might possibly be passing flammable material to the flame. Next, shut off the air supply to the flame by using dry powder and/or foam. Extinguishers should also direct cooling fluid into the flame area and cool the fuel supply to the flame. Water-borne foam, and water fog are particularly effective in reducing the temperature within the fuel vaporizing and flame space. If the temperatures can be dropped low enough, the fuel stops vaporizing, the flame temperature drops and the combustion reaction stops -- hence, the fire goes out.

Use red or blue dry powder extinguishers on oil, gas and electrical fires. The blue dry powder extinguishers are also effective on fires fueled by wood, paper, textiles and the usual solid combustibles. Do not use foam or water on electrical fires until electricity is shut off.

In case of a general fire, before the fire department or anyone else applies water, be sure to shut off all electric power in the plant by opening the main switch.

Use foam and water on fires inside of tanks. oil-water separators or any place else where the oil is confined and will not overflow and spread. Water spray nozzles are effective on crankcase oil, heavy and light cuts, barometric oil and bottoms. It is not effective on gasoline or naptha fires.

In general, apply the stream from an extinguisher at the base of a fire. Approach a fire from upwind and upslope if possible, but avoid going above fires where a release of gas liquid or a puff of air may cause an upsurge of flame that might surround you.

In case of a large general fire around the Fractionator and strippers, with a situation where the fuel feeding the fire cannot be shut off, call the fire department. Shut off the main electric power switch and request the fire department to blanket the Fractionator and stripper area with a fine water fog to keep the structure cooled down.

SECTION XIII

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1 Accession Number	2 Subject Field & Group	SELECTED WATER RESOURCES ABSTRACTS INPUT TRANSACTION FORM
	05D	

5 Organization

National Oil Recovery Corporation (NORCO)
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6 Title

CONVERSION OF CRANKCASE WASTE OIL INTO USEFUL PRODUCTS

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16 Project Designation

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21 Note

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*Crankcase Oil, *Reprocessing, *Vacuum Distillation

27 Abstract

The project goal was to demonstrate a simplified technique for reprocessing spent automotive crankcase oils into useful petroleum products other than lube oils, without producing residues which cause water pollution.

To achieve the foregoing objectives, National Oil Recovery Corporation modified its entire plant system with special equipment and conducted laboratory and plant runs.

The objectives were substantially attained in that all the products from the vacuum distillation were sold as low sulfur heating fuels and as potential diesel fuel. Only the water in the fuel is not recovered.

Some technical work was done to upgrade the refinery products to obtain a higher product realization.

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