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A LOW COST ENVIRONMENTALLY BENIGN WASTE LUBRICANTS RECYCLING/RE-REFINING TECHNOLOGY

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Over two million gallons of spent aviation turbine lubricants are generated in the US by the Department of Defense and the commercial airlines. Media and Process Technology Inc. has developed the membrane-based process which can re-refine the spent turbine oil to near virgin state. This report summarized the results obtained from a pilot demonstration unit. About 300 gallons of finished product were produced and was sent to the customers for initial market testing. Based upon the positive response from the process and market testing conducted in this project, a full scale production facility will be pursued as a next step for commercialization.

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1. INTRODUCTION

High thermal stability lubricants, based upon synthetic polyol esters, are used throughout the military and industry in applications requiring high performance materials such as turbine oils, fire resistant hydraulic oils, heavy duty truck transmission oils, etc. Over 15 million gallons of spent ester based lubricants are disposed of yearly in the U.S. and Canada today. Of this total approximately 2 to 2.5 million gallons of spent MIL-L-7808 and -23699 (or equivalent) turbine oils are generated by the DoD and commercial airlines. In general, most of these oils are disposed of as low value fuel with a market value of $0.20 to $0.40 per gallon. However, virgin ester based synthetic lubricant basestocks command prices in the range of $7 to >$10 per gallon. Burning of spent synthetic lubricants, as is done with mineral oils, not only wastes a valuable resource but also raises environmental concerns, so that it is sensible to consider re-refining options.

Current commercial oil re-refining technology is practiced to a very limited extent and, more importantly, is not capable of producing a re-refined synthetic lubricant suitable for sale as a high value basestock. The limitations can be broken down into both economic and technological barriers. Three principle economic barriers to re-refining spent synthetic lubricants using conventional technology include (i) cost, (ii) complexity, and (iii) physical plant size required to achieve economy of scale (>30 million gallons per year). At present there are also at least two technological barriers to the re-refining of spent ester based synthetic lubricants. First, metal contaminants will catalyze degradation of the synthetic oil when fuel contaminants are removed during high temperature evaporation/distillation of the spent material. Second, the tricresylphosphate additive in the spent turbine oil is not removed thereby limiting the finished product applications. In summary, no technically viable conventional solution is currently available to re-refine spent ester based synthetic lubricants.

In response to these problems Media and Process Technology Inc. (M&P) has developed a modified version of its mineral oil re-refining LubriClear Process which overcomes the barriers associated with conventional technology. This modified LubriClear Process delivers high quality synthetic base oils from spent material at low cost on scales as small as 500,000 gallons per year.

Previously, M&P has demonstrated the technical feasibility of re-refining spent turbine oils into synthetic lubricant basestocks. Experimental products were produced using bench scale equipment and were well characterized. Based upon the physical characteristic/properties, several aftermarket packagers/blenders of synthetic ester based lubricants had expressed an interest in purchasing these materials. However, they required samples on the order of 5 to 10 gallons for in-house blending and performance testing to (i) verify our results and (ii) conduct applications testing.

In this effort, our primary objective was to generate test quantities of re-refined ester based synthetic lubricant basestock from spent turbine oils using the

LubriClear Process. To achieve this objective, a pilot test system was established, tested, and then used to produce finished quantities of re-refined polyol esters from several commercial and military sources. Excellent duplication of the previous bench results was achieved. The test samples produced using the pilot equipment were sent to several aftermarket synthetic lubricant packagers/blenders for in-house blending and performance testing. Feedback from these endusers was enthusiastic and customers for >250,000 gallons per year of finished product were identified.

Based upon the success of this phase of the research effort, M&P has established a small scale re-refining facility at our Schenley PA field demonstration facility to produce ca. 5,000 to 10,000 gallons per month of finished re-refined turbine oil. Over the next year, this facility will be used as a showpiece to attract additional financing for capacity expansion and development of an additional 2 to 3 sites in the US.

2. EXPERIMENTAL

2.1. Ceramic Membrane Ultrafiltration Pilot Unit

A photograph of the pilot scale ceramic membrane system used in this work is shown in Figure 1a. Samples of spent turbine oil were charged to the feed tank and heated to 150°C under nitrogen purge and recirculated on bypass for 0.5 to 4.0 hours to remove low levels of fuel contamination. This pretreatment was found to be adequate to improve the used oil flash point and viscosity to the virgin quality specifications. Following pretreatment, the used oil was recirculated through the membrane. Pilot scale membrane testing was conducted at temperatures of 130 to 160°C at pressures of 30 to 80 psig. Membranes used in this test program were full scale commercial M&P elements at 29.5" long and 1.4" in diameter. The nominal pore size of the elements was 0.10μm (1,000Å). The surface area was ca. $0.55m^2$. A photomicrograph of M&P's commercial ceramic membrane element is shown in Figure 1b.

The permeance used throughout this report is the overall membrane throughput (liters per hour) normalized by the membrane area and average driving pressure and is measured in liters per hour per m^2 of surface area per bar of driving pressure (lmhb).

2.2. Polishing/Finishing Pilot Unit

A photograph of the pilot scale polishing/finishing apparatus used in this work is shown in Figure 2. Used oil, pretreated in the ceramic membrane ultrafiltration pilot unit, was fed to the polishing/finishing unit using an HPLC pump. Finished lubricant samples were collected in one liter plastic containers for characterization of the color and then combined into larger 5-gallon samples. The unit can be operated at temperatures to 200°C and pressures in excess of 150 psi. About 1 gallon per day can be produced using this unit.

2.3. Feed Samples

M&P received a total of 20 samples of spent polyol ester based turbine lubricants with varying levels of contamination from a variety of military and commercial sources. Characteristics/properties measured for each of these samples is shown in Table 1. The data in Table 1 is broken down into two sections to show characterization results for the as-received sample and the sample following heat treatment at 200°C under inert purge. This was necessary because a significant number of the samples received were contaminated with jet fuel. Fifty-five gallon drum samples were recieved from Dallas Airmotive (Dallas, Texas, 5 drums), Robins AFB (Georgia, 1 drum), Tinker AFB (Oklahoma, 3 drums), and NAS Jacksonville (Florida, 4 drums). These samples were used during this phase of the program in the pilot systems to generate finished samples of re-refined lubricant. The remaining samples were received in 1-quart to 1-gallon sizes and were used to assess used oil quality from additional sources.

2.4. Sample Characterization Testing

Media and Process Technology Inc. (M&P) has a variety of waste oil characterization equipment in-house that was used throughout this project. Included in this list are a Cleveland Open Cup Flash Point Tester (Koehler Instrument Company, ASTM D-92), 40 and 100°C isothermal baths for viscosity determinations (Cannon Instrument Company, State College, PA, ASTM D-2270), a Beckman Spectraspan Direct Current Plasma Spectrophometer for metals analysis, a Brinkman (Metrohm) Autotitrator for Total Acid Number determinations (New York, ASTM D-664), and a colorimeter for oil color determinations (Precision Scientific, Bellwood, IL, ASTM D-1500). In addition, CTC Analytical Services, Inc. (Cleveland, OH), a nationally recognized full service lubricant characterization facility, handled sample analyses including water in oil by Karl Fisher titration (ASTM D-1744), fuel in oil by GC (ASTM D-3524), glycol in oil (ASTM D-2982), pour point (ASTM D-97), and metals analysis via Inductive Coupled Plasma.

3. RESULTS AND DISCUSSION

3.1. Ceramic UF Pilot Tests

The ceramic membrane subsystem has been established as a cost effective method for removing particulate, ash, and coke and improving the color and particularly the clarity of spent turbine oils. Without membrane pretreatment, significant reduction in the color removal capacity is observed in the finishing subsystem. In this phase of the research effort, pilot level ceramic membrane permeation tests were conducted to compare the membrane performance and product quality with results obtained in the previous bench study [Ref. 1]. Used oil samples obtained from a number of sources identified in Sec. 2.3. were used. The results are discussed below.

Figure 3 shows the permeance of a commercial M&P ceramic membrane element with a nominal pore size of ca. 1,000Å. The feed is from Dallas Airmotive (DAM 1 and DAM 5). The pilot test was conducted at 130 to 140°C at an average membrane pressure of 35 to 40psi. For the first nine hours, the test was conducted in a recycle mode in which all of the permeate was returned to the feed tank. During the final two hours, the test was conducted in a concentration mode in which the permeate was collected in separate storage drums. In this mode, contaminants in the feed oil are "concentrated". The permeance is compared with data obtained in the original bench experiment conducted using a single channel ceramic membrane element. The bench tests were conducted at lower temperatures (70 to 85°C) due to limitations of the equipment.

Excellent membrane permeance and permeate quality was obtained using the membrane pilot unit. The product oil was clear and bright (no turbidity) and the color index was ca. 4.5. The higher permeance compared with the bench unit (ca. 30 vs. 3 lmhb) is due to the higher operating temperature. Roughly 100 gallons of used turbine oil permeate was generated during this phase of the test program. This oil was used in the pilot polishing/finishing subsystem tests as discussed in Sec. 3.2.

In an additional test, the overall percent volume of recovery of oil that could be achieved in the concentration mode was determined. This study was necessary to determine the yield in the membrane subsystem. Samples of used oil from Dallas Airmotive (DAM 2, 3, and 4) and NAS Jacksonville (Jack 1, 2, and 4) were used in this study. Figure 4 shows the permeance and system temperature plotted as a function of time during this concentration run. Overall, a total of 209 gallons of feed was charged to the ceramic membrane system of which approximately 201 gallons was recovered as permeate. The run was stopped with 8 gallons of used oil in the feed tank because this is the minimum required to charge the system. Based upon these results a minimum membrane system yield of 96.2% can be expected in the full scale system. Because the permeance was still relatively high at the end of the concentration run, it is not unreasonable to expect membrane system yields in excess of 99%.

Overall, the membrane pilot tests demonstrated that (i) the permeance was superior to the bench system tests, likely the result of the higher operating temperature, (ii) the product quality was comparable to that obtained in the bench tests, and (iii) very high yields of permeate oil can be expected in the full scale membrane subsystem.

3.2. Polishing/Finishing Pilot Tests

Permeate oil from the pilot scale membrane subsystem (labled DAM 1.5 representing a combination of drums 1 and 5 from Dallas Airmotive) was tested in both bench and pilot level finishing subsystems to verify the results from the previous study. Figure 5 shows the finished oil color plotted as a function of cumulative polishing/finishing capacity for both the original bench tests and the bench and pilot tests conducted as part of this research effort. The notable difference in the shape of the curves for the previous and current results is due to the higher color of the feed in the original bench tests. DAM1.5 permeate from the pilot membrane system was used in the recent bench polishing/finishing tests, since the color and viscosity grade of the original used oil sample was different than this current DAM1.5 sample. The polishing/finishing capacity for color removal at a cumulative polishing/finishing capacity of 1.5 gallons per pound is 3.5 and 3.8 gallons of oil treated per pound of finishing agent for the recent bench and pilot tests, respectively. This agrees well with the original bench results of 3.2 gallons per pound.

The polishing/polishing capacity is measured as the area of the graph swept out between the feed oil color line and the effluent color curve for cumulative adsorbent loadings less than 1.5 gal/lb (the shaded region in Figure 5 is given as an example of the area determination for the polishing/finishing capacity for the original bench test).

Overall, the pilot scale tests were very successful, demonstrating the ease of scaling the process by a factor of ca. 100 from the bench treatability tests. Because a commercial scale membrane element was used in the membrane pilot tests, there should be no problem with scaling to much larger production sizes. Similarly, commercial grade polishing/finishing agent in the bench system was used in the pilot polishing/finishing subsystem, scaling to much larger production volumes is straightforward engineering design.

3.3. Overall Treatability Status

Table 3 is a compilation of the status of the current state of the art of the LubriClear Process for the removal of various contaminants from spent turbine oils. No significant changes to the technology have resulted from the work conducted in this study.

3.4 Process Description

The overall process flow diagram is shown in Figure 6. No significant changes have been made to the PFD as a result of this research program. For information purposes, a description of the PFD follows. The numbers shown in the figure indicate the percent distribution of the feed to the various intermediate and product streams.

Waste lubricant is initially heat pretreated in a boiling kettle at temperatures of ca. 150°C in an inert purge to achieve via evaporation complete removal of trace contaminants of water, glycols and other lights ends. At times in excess of 4 hours, excellent removal of fuel contamination is also achieved. The overhead from the flash is condensed and phase separates. The composition of the overhead condensate is unknown. However, it is assumed that the top phase is organics which can be burned as fuel while the bottom phase is water/glycol (antifreeze components) which can be further treated with a number of waste water treatment technologies and then discharged.

Bottoms from the flash/evaporation, which represent about 99% by volume of the feed, is sent to the membrane subsystem. Here, proprietary ceramic membrane technology is used to remove various metals and other particulate matter contamination. It is assumed that 99.5% of the feed to the membrane subsystem flows through the membranes as product or permeate. This is a reasonable concentration level given that ca. 96.2% was achieved in the pilot test as described in Sec. 3.1. with little or no loss in membrane permeability. The rejected concentrated from the membrane system, containing >95% of the synthetic oil particulate contaminants (coke, metals, etc.), is sent to fuel the process. The permeate is sent to the polishing/finishing subsystem.

In the polishing/finishing subsystem, the color of the sample is adjusted to that of the virgin material and most of the remaining components are removed. Once the polishing/finishing capacity is utilized, the material can be regenerated. Because of the relatively high capacity of the agents compared with our motor oil recycling systems, it may be more cost effective to simply dispose of the agent and buy fresh material.

Finally, the finished sample is post-treated in an evaporator at ca. 300 to 320°C under a vacuum to remove final traces of fuel from the sample. At this point, the product can be sold as a re-refined polyol ester basestock.

3.5 Overall Finished Product Quality

Table 2 shows the characteristics of the finished oil from the pilot and bench systems. Good agreement is obtained between the current bench and pilot results and the previous bench results. It should be noted that a lower viscosity grade of turbine oil was used in the previous bench tests (MIL-L-7808) versus the current tests (MIL-L-23699). Hence, slight differences in the finished sample properties, such as the viscosity and flash point, can be attributed to differences in the source used oil. The quality of the finished oil produced in the pilot tests is comparable to that of the virgin turbine oil from Exxon (ETO 2380). The slightly darker color in comparison to the virgin polyol ester basestock from Henkel (Emery 2940) is due to the presence of the oxidation inhibitors in the re-refined and virgin turbine oils. Overall, the quality of the finished re-refined lubricant is comparable to that of the virgin products.

3.6 End Users' Feedback

Because M&P lacks the expertise and more importantly the contacts necessary to establish a turbine oil sales network, Kimes Trading International, KTI, was enlisted as a sales agent to identify potential large scale buyers of M&P's re-refined polyol esters. M&P provided ca. 15 gallons of re-refined polyol ester produced using our pilot systems as detailed in Secs. 3.1. and 3.2. In addition to the samples, M&P also provided a product MSDS for our finished lubricant as shown in Appendix I. Re-refined lubricant was sent to current clients of KTI who expressed interest in the product. Additionally, several other end users were identified using an advertisement placed by KTI in Lubes-n-Greases which is shown reproduced in Appendix II. All of the endusers that received samples from M&P through KTI were satisfied with the quality. They are eager to receive quantities ranging from 20 to 80+ drums per month. Overall, customers for over 250,000 gallons of re-refined polyol ester have been identified and confirmed by KTI.

3.7 Process Economics

<u>3.7.1 Process Capital and Operating Costs:</u>

Table 4 shows a comprehensive breakdown of the process operating assumptions and capital and operating costs of a re-refining facility that will produce 500,000 gallons per year of finished re-refined polyol ester from spent material in a plant operating 24 hours per day, 260 days per year. Two Base Cases are considered. Case I uses the cost estimates based upon our original bench results. Case II uses the improved membrane permeance results obtained from the pilot unit tests conducting in this effort.

In Case I and Case II, the economics are based upon an overall process yield of 89.2% and assumes conservative membrane and polishing/finishing subsystem yields of 97 and 92%, respectively. The yield loss in the membrane subsystem reflects segregation of particulate and other debris. The yield loss in the polishing/finishing subsystem reflects loss of tricresyl phosphate (~3% of the used oil volume) and loss of oil that can not be recovered during blowdown of the spent agents (~4 to 5%). The blowdown oil loss is due to oil trapped in the internal void volume of the polishing/finishing bed that is essentially unrecoverable. This loss increases as the capacity of the agent decreases.

Water and other light end loading of the used oil which needs to be evaporated is assumed to represent approximately 5% of the total in-coming feed. This figure is not included in the oil yield calculations but is taken into the cost calculation. Other non-obvious process variables/assumptions are described as follows:

Spent Lubricant Feed Rate: Represents the total amount of waste lubricant necessary to yield the finished lubricant production rate as defined by the process yield.

Membrane Permeance: Assumed to be 6 lmhb at 160°C for Case I and 30 lmhb for Cases II. The permeance used in Case I was based upon a previous assumption used in the original economic analysis, specifically, that the permeance would be double that of the bench results obtained at 80°C (2 to 3 lmhb [Ref. 1]). As was found in the pilot tests, much higher permeances are obtained (30 lmhb).

Membrane Area: Total membrane filtration area required.

Main Process Pump Power: Power required to recirculate used oil feed through the membranes. Significantly lower power consumption is required in Case II due to the much lower membrane area requirements (higher membrane permeances).

Polishing/Finishing Capacity: As determined experimentally. A conservative estimate of 2.9 gal/lb is assumed.

Polishing/Finishing Agent Life: Number of times agent can be regenerated and re-used. Worst case of only one use and no regeneration is assumed for this analysis.

Membrane Capital Requirements: Includes purchase of membranes, membrane housings (modules), and all system components including pumps, piping, valves, gauges, etc. Does not include purchase of land, storage tank facility, etc.

Membrane Operating Costs: All relevant costs are given. Depreciation is assumed to include all of the capital equipment and only the membrane housings (i.e.,: 1/3 membrane). The other 2/3 of the total membrane cost represents the membrane elements themselves which are assumed to be replaced every 3 years as shown.

Polishing/Finishing Capital Requirements: Includes agents and other system components. No regeneration is assumed in this case as mentioned previously, so that no capital equipment is required. Three polishing beds will be required operating at 40°C.

Polishing/Finishing Operating Costs: Similar to Membrane Operating Costs.

An important point should be highlighted about the differences in the capital and operating costs for the two cases given in Table 4. The membrane subsystem capital and operating costs are significantly reduced in comparison to the original Case I analysis. The membrane subsystem capital cost drops from ca. $210,000 to $42,000 to produce 500,000 gallons per year. Similarly, the operating costs drop from $0.40 to $0.23 per gallon. The lower cost reflects the significantly higher membrane permeance obtained with the pilot unit operating at temperatures of 130 to 160°C in comparison to the original bench data obtained at 80°C. Hence, membrane surface area requirements are reduced from $27m^2$ to $5.4m^2$. The lower operating cost is also a direct function of the lower membrane surface area requirement which impacts the recirculation rate and hence pump energy cost, as well as depreciation, membrane replacement and maintenance costs.

Overall, based upon the results obtained during the Phase I research program, significant reduction in the capital and operating costs are observed. In Sec. 3.7.3., the impact on system profitability is examined in light of these results.

3.7.2. Raw Material Costs:

Another cost that is expected to be significant in the production of re-refined polyol esters is the cost of collection since the used oil sources tend to be small (<15,000 gallons per year) and scattered throughout the country. Based upon discussions with KTI personnel, who have significant experience in distribution networks, we have been able to develop an estimated average cost of used oil collection. The cost has been developed using a collection strategy that focusses on the use of a distributed system of regional warehouses that act as storage facilities to accumulate spent polyol ester prior to long haul shipment to a re-refining facility. The overall cost is broken down into two categories:

First, warehouses and terminals for "toll" storage are available throughout the country so that no capital investment is required. KTI currently uses "toll" warehouses to store finished petrochemical products at a cost of ca. $0.10 to $0.12 per gallon per month.

Second, trucking costs in general are dependent upon distance. For long hauls over 500 to 1,000 miles, the cost can reach as high as ca. $1,200 per truckload independent of the size of the load. Hence, for long hauls, full capacity shipments of 80 drums (4,400 gallons) at a cost of $0.27 per gallon are ideal. Less than truckload costs for long hauls rise dramatically. Since a used oil generator will not store 80 drums of oil at his site, in general, less than truckload quantities will be collected from the generator and then accumulated at a localized warehouse prior to shipment to Pittsburgh.

In the economics section below, the worst case shipping costs are used, which assumes (i) long distance hauling from California to Pittsburgh (PA) and (ii) toll warehousing for one month. Even under these circumstances polyol ester rerefining can be very profitable as is demonstrated.

3.7.3 Revenue, Profitability, and Capital Payback:

In Table 5 two profitability models are developed. The first two profitability models (Models I and II) use the operating and capital costs developed in Case I and II (Sec. 3.7.) to show the improvement in the profitability following the use of higher membrane permeance values (30 vs. 6 lmhb). The last profitability model (Model III) shows the improvement in profitability using slightly higher finished oil market values and larger production volumes, respectively.

In all three models, several basic assumptions are made. First, an average payment of $1.00 per gallon is made to the generator as a segregation cost. This is offered as an incentive to minimize used oil contamination during collection at the generators site. It should be noted that no generator we have contacted has requested more than $0.50 as a segregation credit. Second, worst case total spent oil transfer and collection costs of $0.64 per gallon are used. This cost consists of (i) a worst case shipping cost of $0.27 per gallon of spent oil (trucking cost for 80 drums of used oil from California to Pittsburgh), (ii) a $0.12 per gallon warehousing cost, and (iii) a sample characterization cost of $0.50/gallon (this high cost reflects the fact that most of the used oil received will be in drum size quantities). Specific assumptions of each model are discussed below.

In Models I and II the re-refined polyol ester estimated market value is assumed to be $4.50 per gallon. This represents a minimum market penetration price based upon discussions with KTI. In Model III we have assumed an estimated market value of the finished polyol ester at $7.00 per gallon. At both $4.50 and $7 per gallon, the assumed estimated market price is well below that of virgin material which ranges from $10.61 to $14.70 per gallon based upon vendor quotes (see Table 6).

The net revenue and capital payback for all of these models is very attractive. Based upon our original bench data (Model I), net revenues of $785,000 per year could be obtained with a capital investment of ca. $499,000 yielding a 7.6 month capital payback. Using current pilot data and conservative estimates of the market value of the finished oil (Model II), net revenues improve to $1.02 million per year while the capital investment decreases to $197,000 yielding a capital payback of only 2.3 months. Hence, dramatic improvement in the overall profitability is demonstrated following the pilot testing program.

By increasing the estimated market value of the finished re-refined oil to $7.00 per gallon (Model III), net revenues increase by an additional 110% to $2,300,000 per year while the capital payback drops to <1 month. For market penetration purposes, we expect to initially sell the finished oil for ca. $4.50 per gallon initially. However, KTI discussions with potential customers indicate that a finished oil price of $7.00 per gallon is acceptable. At this price, we still offer a product that is 50 to 75% less than the virgin oil price (see Table 6).

Overall, the profitability analysis shows that excellent revenue and very short capital payback can be achieved using the proposed process for the rerefining of spent polyol ester based synthetic lubricants. It is estimated that up to 40% of the 17MM gallons of spent polyol ester available in North America can potentially be collected for rerefining into a synthetic lubricant basestock. Using this Model III analysis, this translates into a total market in excess of $35MM per year in net profit. The surprisingly good economics reflects the fact that the finished product, a synthetic polyol ester based lubricant, is extremely valuable, especially when compared with rerefined mineral oils ($7.00 versus $0.80 per gallon, respectively). Hence, although the total processing costs are higher for the synthetic oil, primarily because of the collection/segregation costs, the much higher market value of the finished product more than compensates.

4. CONCLUSIONS

1. **Completed pilot scale performance tests to verify previous bench results.** Pilot scale membrane throughput was as much as 10-fold greater than that obtained in the previous bench tests. The higher operating temperature is the likely source. Polishing/finishing results from the finishing subsystem were comparable to those obtained in the bench study. A total of 100 gallons of deashed oil and 35 gallons of finished oil was produced from spent turbine oil during the pilot work for initial market testing.

2. **The quality of the pilot scale re-refined product is comparable to virgin synthetic material.** Characterization results of samples obtained from the pilot scale test program were in good agreement with virgin polyol ester basestocks. Because the oxidation inhibitors are not completely removed during LubriClear processing, the oxidative stability is actually superior to that of virgin basestocks.

3. **Established marketing agreement with KTI.** A marketing agreement was established with Kimes Trading International, KTI. KTI will be responsible for marketing and sales of the re-refined lubricant produced by M&P.

4. **Excellent feedback from prospective end-users.** One and five gallon samples of re-refined oil were delivered by KTI to several potential blenders for characterization and performance testing. Based upon the response generated from these samples, KTI has verified that over 250,000 gallons per year of re-refined lubricant can be sold to these initial customers. This is more than adequate for the next phase field demonstration testing currently underway at M&P's Schenley PA facility.

5. **Updated the economic projections.** The process economics improved significantly because of the higher membrane throughputs observed in the pilot test program versus the original bench results. Net revenues increased from $785,000 to $1.02MM per year while capital payback declined from 7.6 to 2.3 months.

6. **Began construction of the full scale process facility.** Based upon the excellent pilot test and economic results and the enthusiastic feedback from the various end users identified by KTI, M&P has begun construction of a small scale turbine oil re-refining facility at its Schenley PA demonstration facility. Current plans are to produce ca. 5,000 gallons per month of finished re-refined polyol ester and expand the capacity in steps to 25,000 gallons per month as capital becomes available.

4. RECOMMENDATIONS

Based upon the results of this research program, several key recommendations can be made, specifically,

1. **Continue to optimize the re-refining process conditions.** Work up until this point has focussed on developing the technology and generating samples for endusers. To minimize capital and operating costs, it is appropriate to optimize the processing conditions in the major subsystems. Particular emphasis should be placed on the post-evaporation subsystem for jet fuel removal, since little work has been conducted in this area.

2. **Scale up the process to the production level.** The feedback from potential end users of the re-refined lubricant has been enthusiastic. Additionally, the process has been demonstrated using full scale system components. Hence, scale up to the field demonstration size of ca. 100,000 to 250,000 gallons per year is the recommended next step.

3. **Deliver samples in large volumes (100's of gallons) for market penetration studies.** To complete the market acceptance study, it is necessary to deliver multiple drum load quantities to the various end users identified in this work.

4. **Identify additional synthetic oils that may be amenable to re-refining.** The military and private sector uses a wide array of synthetic lubricants. For instance, polyalphaolefin based and phosphate ester based fire resistant hydraulic fluids are used extensively in all services of the military. The value of these materials ranges from $4 to >$10 per gallon, in the range of the polyol esters and significantly higher than mineral oil basestocks. It is believed that the modified LubriClear Process developed in this project is applicable to the re-refining of these oils. Pursuing the recycling of these oils in conjunction with turbine oils can eventually achieve the total lubricant recycling objectives of the Air Force.

6. REFERENCES

Media and Process Technology Inc., “A Low Cost Environmentally Benign Waste Lubricant Recycling/Rerefining Technology”, FINAL REPORT, Contract No. F33615-96-C-2654

Table 1: Sources and characterization data for spent polyol ester obtained from various locations throughout the US.

Sample	ID	As-Received: Color [-]	Visual Quality [-]	BS&W [%]	Flash Point [°C]	Viscosity @40°C [cSt]	Viscosity @100°C [cSt]	VI [-]	TAN [-]	API Gravity [@40°C]	Metals Si [ppm]	Metals P [ppm]	Metals Zn [ppm]	Post Evaporation at 200°C + 20µm filtration: Visual Quality [-]	Color [-]	Flash Point [°C]	Metals Si [ppm]	Metals P [ppm]	Metals Zn [ppm]	API Gravity [@40°C]	Viscosity @40°C [cSt]	Viscosity @100°C [cSt]	Index [-]	Mineral Oil [%]
DAM	1	5.5	S.Turbid	<2.0	254	27.3	5.16				32	3077		Clear		258					24.7	5.15	143	<5.0
	2	3.0	S.Turbid	<2.0	175	18.4	4.20				12	3711		Clear		260					24.4	4.94	131	<5.0
	3	2.25	Clear	2.5	208	24.6	4.85				4	3249		Clear		268					23.8	4.88	132	<5.0
	4	4.5	S.Turbid	5.0	234	24.4	4.96				10	3299		Clear		278					24.6	4.90	125	<5.0
	5	3.0	Clear	3.0	250	23.2	4.78				24	3355		Clear		DND					23.2	4.78	129	<5.0
Tinker	1	6.5	S.Turbid	<2.0	198	13.4	3.36			19.9		2062	2	Clear		234					13.1	3.34	130	<5.0
	2	8.0	S.Turbid	<2.0	94	6.0	1.96			28.9	7	1705	1	Clear		148					17.8	3.93	116	DND
	3	7.75	S.Turbid	<2.0	148	10.2	2.70			21.9	3	2324	1	Clear		228					12.8	3.26	125	DND
Jack	1	~2	V.Turbid	50.0	252									Clear		260					26.3	5.06	122	DND
	2	~2	V.Turbid	66.0	254									Clear		242					25.6	5.06	133	DND
	3	~2	V.Turbid	>95.0										Sample was all water.										
	4	~2	No Oil	15.0	236									Clear		246					25.7	4.96		DND
Robins	1	>8	S.Turbid	<2.0	125	11.7	2.89			21.5	1	2241	5	Clear		215					13.3	3.38	132	<5.0
Creek	1	3.5	Clear	<2.0	258	25.8	4.95		0.734	13.3	36	2707	1	Not necessary to conduct.										
	2	2.5	Clear	<2.0	250	25.5	5.46		0.504	15.7	7	2555	2	Not necessary to conduct.										
UA-SF	1	3.5	Clear	<2.0	254					16.9	1	1154	1	Did not conduct high temperature evap.										
GEdls	Love	>8	Opaque	<2.0	254	29.1	5.56		0.350	<20				Did not conduct high temperature evap.										
	Carter	3.5	Clear	<2.0	76	4.3	1.61		0.102	30.5				Significant fuel contamination.										
GEark	TestCl	3.5	Clear	<2.0		9.7	2.7							Significant fuel contamination.										
	Shop	>8	Opaque	<2.0		68.9	12.6							Significant mineral oil contamination.										

NOTES:

DAM: Dallas Airmotive Inc. (Texas)
Tinker: Tinker Air Force Base (Oklahoma)
Jack: Jacksonville Naval Air Station (Florida)
Robins: Robins Air Force Base (Georgia)

Table 2. Chracteristics and properties of various polyol ester based lubricant samples including virgin, spent and M&P re-refined samples.

Sample ID			Original Bench Feed	Original Bench Permeate	M&P Bench Rerefined Finished Lubricant	Pilot Feed (DAM1.5)	Pilot Permeate	M&P Pilot Rerefined Finished Lubricant	Virgin Turbine Oil (Exxon ETO 2380)	Virgin Polyol Ester (Henkel Emery 2940)
Color,	ASTM D-1500	[-]:	7.5	6.5	1.75	5.5	4.25	1.5	1.5	0.5
Water by KF	ASTM D-1744	[wt%]:	0.03	-	0.01	0.04	1.31	0.01	≤0.05	0.01
Fuel by GC	ASTM D-3524	[wt%]:	2.69	-	-	1.1	-	-	-	-
Glycol	ASTM D-2982	[wt%]:	Neg	-	-	Neg	-	-	-	-
TAN	ASTM D-664	[mgKOH/g]:	0.43	0.40	0.07	0.30	0.21	<0.05	0.37	0.02
Ash	ASTM D-482	[wt%]:	0.083	0.01	0.0	-	-	-	-	-
Flash Point	ASTM D-92	[°C]:	206	-	222	258	250	250	249	258
Fire Point	ASTM D-92	[°C]:	234	-	242	-	-	-	-	294
Kinematic Viscosity										
@40°C	ASTM D-2270	[cSt]:	14.6	-	17.0	27.3	26.1	24.7	26.3	24.5
@100°C	ASTM D-2270	[cSt]:	3.5	-	4.0	5.28	5.16	5.2	5.0	5.0
Viscosity Index		[-]:	119	-	135	128	131	134	121	134
Pour Point	ASTM D-97	[°F]:	-	-	-	<-50	-	<-50	-76	-80
Metals Analysis	via ICP									
	Fe	[ppm]:	13	1	0	8	3	0	0	0
	Pb		9	0	0	7	3	0	0	0
	Cu		10	0	0	1	2	0	0	0
	Al		1	0	0	0	0	0	0	0
	Si		10	1	6	32	13	0	1	0
	B		3	1	1	2	2	2	0	0
	Na		18	1	0	0	0	0	0	0
	Mg		28	1	0	1	0	0	0	0
	Ca		69	4	0	2	1	0	0	0
	Ba		27	4	0	0	0	0	0	0
	P		1579	1147	0	3007	3112	0	2480	0
	Zn		276	5	0	9	1	0	0	0
Total Metals Content			2043	1165	7	3069	3137	2	2481	0
Total Metals Content excluding Phosphorous			464	18	7	62	25	2	1	0

Table 3: Status of M&P's synthetic oil rerefining process for the removal of various contaminants from spent polyol esters.

Contaminant	Current Status	What Technology	Comments
H_2O/Glycol	Excellent	Flash/Evaporation	Effective removal has been demonstrated. Both components show low miscibility with polyol ester based lubricants (≤1wt%) and much lower boiling point.
Fuel	Excellent	Flash/Evaporation	100% removal of kerosene from virgin turbine oil has been demonstrated.
Mineral Oil	Can't Remove	None available	No attempt has been made to remove mineral oil contamination.
Total Acid Number	Excellent	Polishing	Total acid number is reduced to ≤0.1 mgKOH/g (which is the spec for virgin oils) following finishing via polishing in all cases tested.
Metals	Excellent	Membrane/Polishing	Overall removal is ≥99.9%. Membrane removal of wear and additive metals is ≥95%. Polishing/finishing removes remaining metals.
Additives	Excellent	Polishing/Finishing	Overall removal of tricresyl phosphate as measured by phosphorous content is ≥99.9%.
Particulate	Excellent	Membrane	Membrane technology removes all traces of particulate contamination.
Color Bodies	Excellent	Membrane/Polishing	Color of re-refined finished product is equivalent to virgin turbine oils (Exxon ETO 2380).

Table 4: Capital and operating costs to rerefine spent polyol esters into synthetic lubricant basestocks under various operating assumptions.

	CASE I	CASE II
System Parameters and Operating Assumptions	Bench	Pilot
Overall Finished Lubricant Production Rate [gal/yr]	500,000	500,000
Operating time [days/yr]	260	260
On-Stream Time [hr/day]	24	24
Overall Process Yield [%]	89.2	89.2
Oil Yield, Membrane [%]	97.0	97.0
Oil Yield, Hydrolysis/Decolorization [%]	92.0	92.0
Overall Lubricant Inlet Rate [gpy]	560,287	560,287
Water in Inlet Lubricant [%]	2.0	2.0
Water in Outlet Lubricant [%]	0.0	0.0
Membrane System Temperature [°C]	130	160
Membrane System Permeance [lmhb]	6.0	30.0
Membrane System Permeance [gpm/m2/psi]	0.00179	0.00896
Membrane System Pressure [psia]	30.0	30.0
Membrane Area [m2]	27.8	5.6
Membrane Main Process Pump Power Consumption [Hp]	154.7	30.9
Lubricant Heat Capacity [cal/g/°C]	0.70	0.70
Lubricant Density [kg/liter]	0.87	0.87
Membrane System Heat Requirements [kW]	30	37
Adsorbent Capacity [gal/lb]	2.90	2.90
Adsorbent Required, Minimum [lb/day]	663	663
Adsorbent Life [cycles]	1	1
Adsorbent Cost [$/lb]	1.20	1.20
Capital Requirements, Membrane and Pre-Treatment SubSystems		
Membranes [$]	69,614	13,923
Other Subsystem Components for Pre-treatment[$]	139,229	27,846
Total Membrane Subsystem Cost[$]	208,843	41,769
Operating Costs, Membrane and Pre-Treatment SubSystems		
Pump Energy [at $0.08/kWhr]	57,919	11,584
Heater Energy [$10/MM BTU]	6,372	7,924
Labor [Supervisor + 3 Operators]	86,870	86,870
Cleaning [$200/m2]	5,569	1,114
Maintenance [8% of System]	11,138	2,228
Depreciation [10yr System+1/3 Membrane]	16,243	3,249
Membrane Replacement [3yr at 2/3 membrane]	15,470	3,094
Operating Costs Total [$]	199,582	116,062
$/gal of finished lubricant	0.40	0.23

Table 4: .....continuation of capital and operating costs.

Capital Requirements, Decolorization SubSystem

Adsorber Subsystem [$]	100,444	100,444
Hydrolysis Subsystem [$]	0	0
Regeneration Subsystem [$]	0	0
Other Adsorber System Components [$]	25,111	25,111
Total Decolorization Subsystem Cost [$]	125,555	125,555

Operating Costs, Decolorization Subsystems

Blower Energy [$0.08/kWh]	5,616	5,616
Heater Energy [$10/MM BTU]	0	0
Labor [Supervisor + 3 operators]	86,895	86,895
Maintenance [8% of Total System]	10,044	10,044
Depreciation [10yr Total System]	12,556	12,556
Adsorbent Replacement	206,897	206,897
Operating Costs Total [$]	322,008	322,008
$/gal of finished lubricant	0.64	0.64

Summary of Process Capital Investment Requirements

Membrane Capital Cost [$]	208,843	41,769
Decolorization Capital Cost [$]	125,555	125,555
Site Preparation [$]	32,500	32,500
Assembly/Installation [$]	32,500	32,500
First Adsorbent Load Purchase [$]	99,870	99,870
Total Capital Cost [$]	499,268	332,194

Summary of Process Operating Costs

Membrane and Pre-treatment [$/gal]:	0.40	0.23
Decolorization [$/gal]:	0.64	0.64
Total Operating Costs [$/gal]:	1.04	0.88

Table 5: Profitability analysis and capital payback to rerefine spent polyol ester into synthetic lubricant basestock.

		Model 1 Using Case I Economics	**Model 2** Using Case II Economics	**Model 3** Model 2 + Higher Value
Production Volume	[gallons/year]	500,000	500,000	500,000
Segregation Cost*	[$/gallon]	1.00	1.00	1.00
Collection Cost**	[$/gallon]	0.89	0.89	0.89
Processing Cost	[$/gallon]	1.04	0.57	0.57
Total Cost	[$/gallon]	2.93	2.46	2.46
Estimated Market Value	[$/gallon]	4.50	4.50	7.00
Gross Revenue	[$/year]	2,250,000	2,250,000	3,500,000
Total Cost	[$/year]	1,465,000	1,230,000	1,230,000
Net Revenue	[$/year]	785,000	1,020,000	2,270,000
Capital Costs	[$]	499,000	197,000	160,000
Payback Period	[months]	7.63	2.32	0.85

* Segregation cost assumes a payment to generator to segregate spent oils.

** Collection Cost assumes:
$1,200/80 drum truckload from California + $0.12/gallon storage + $0.50/gallon characterization cost.

Table 6. Cost of virgin polyol ester based synthetic oils from vendor quotes.

Manufacturer [-]	Product Designation [-]	Viscosity @ 100°C [cSt]	Cost Drums <5,000 gal [$/gal]	Bulk >5,000 gal [$/gal]
ICI Americas	Emkarate 1550	4.4	13.75	10.61
HATCO	HATCOL 2970	4.95	14.70	13.00
Henkel	Emery 2931 and 2935	5.2	13.97	12.81

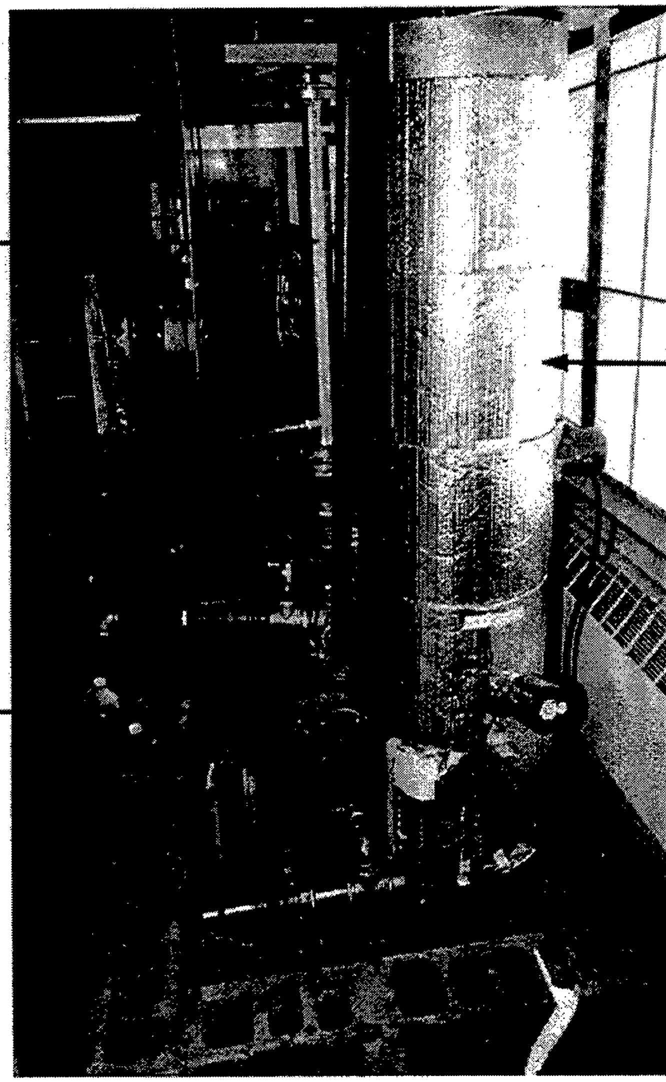


Figure 1a: Membrane pilot unit for high temperature oil applications.

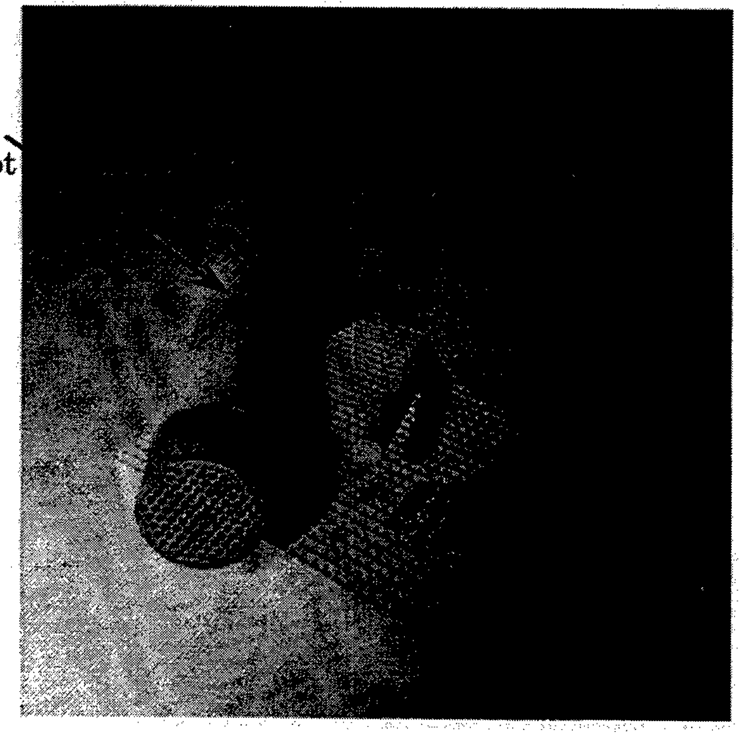


Figure 1b: Various M&P ceramic membrane elements.

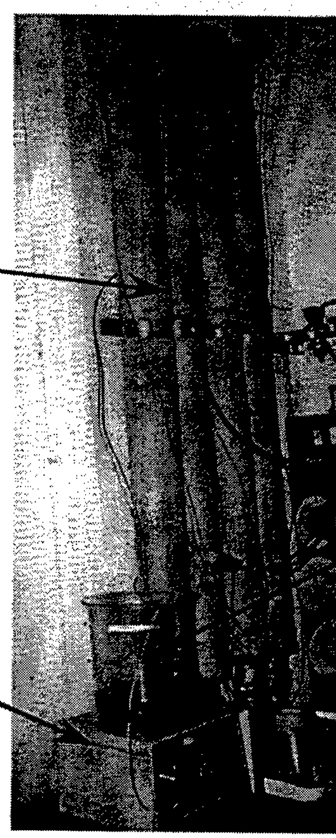
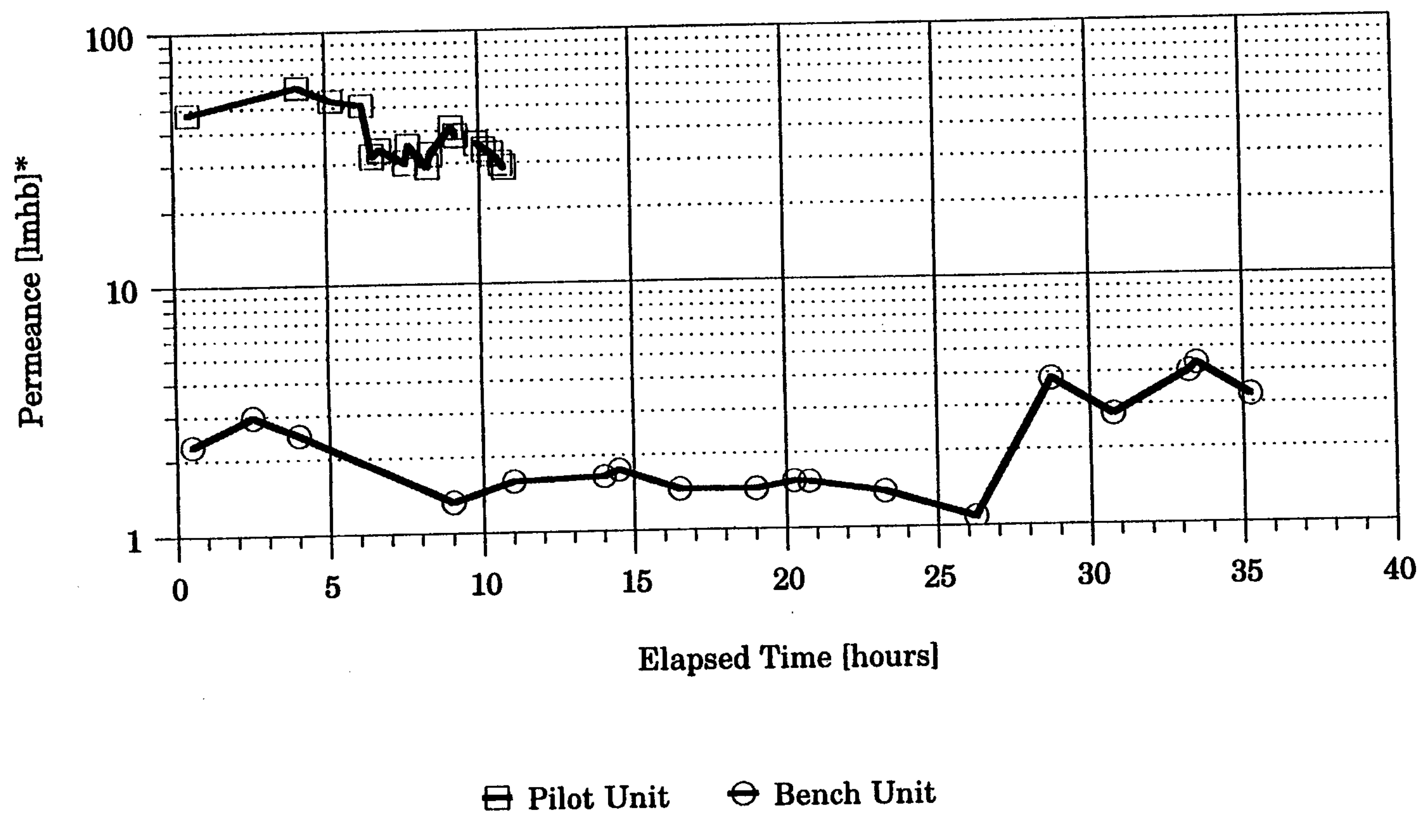


Figure 2: Pilot scale adsorbers for oil decolorization.

Figure 3: Comparison of bench and pilot scale membrane performance.



* liter/m2/hr/bar

Figure 4. Membrane permeance and operating temperature during the pilot scale concentration run.
Feed: Used turbine oil, various.

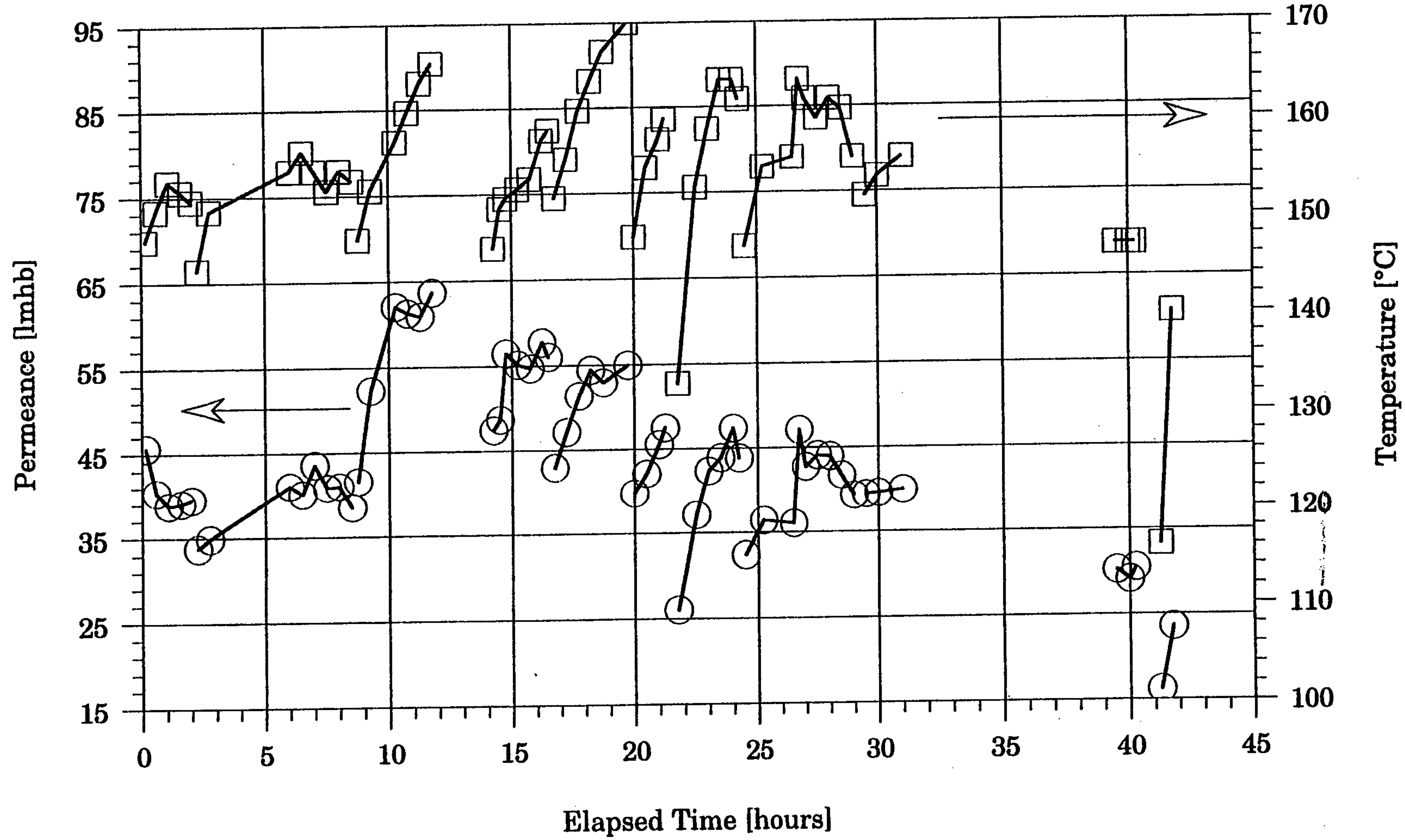
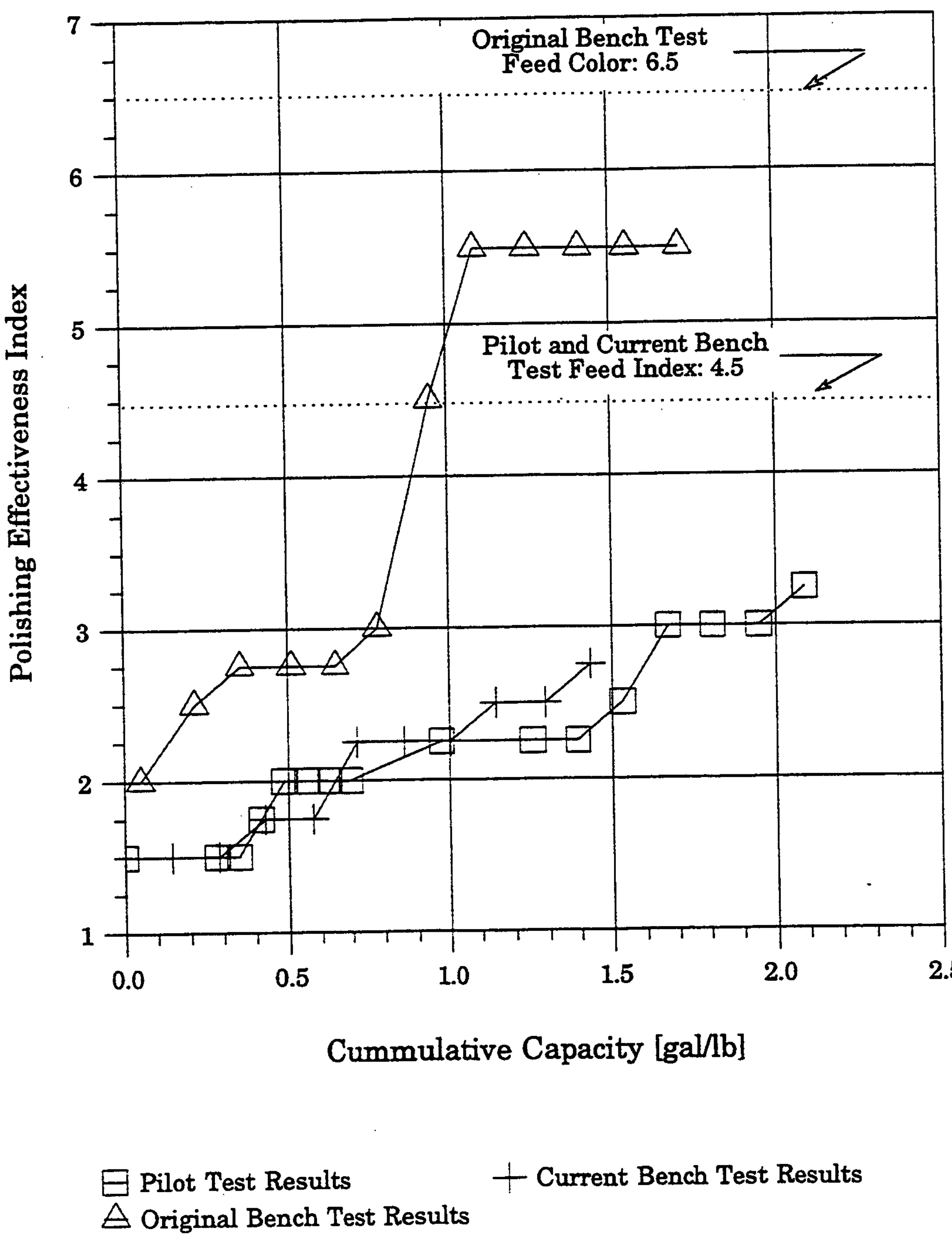


Figure 5: Comparison of thepolishing/finishing effectiveness for the original and current bench tests and the pilot test. Feed oil index is 6.5 for the original bench test and 4.5 for current bench and pilot tests.



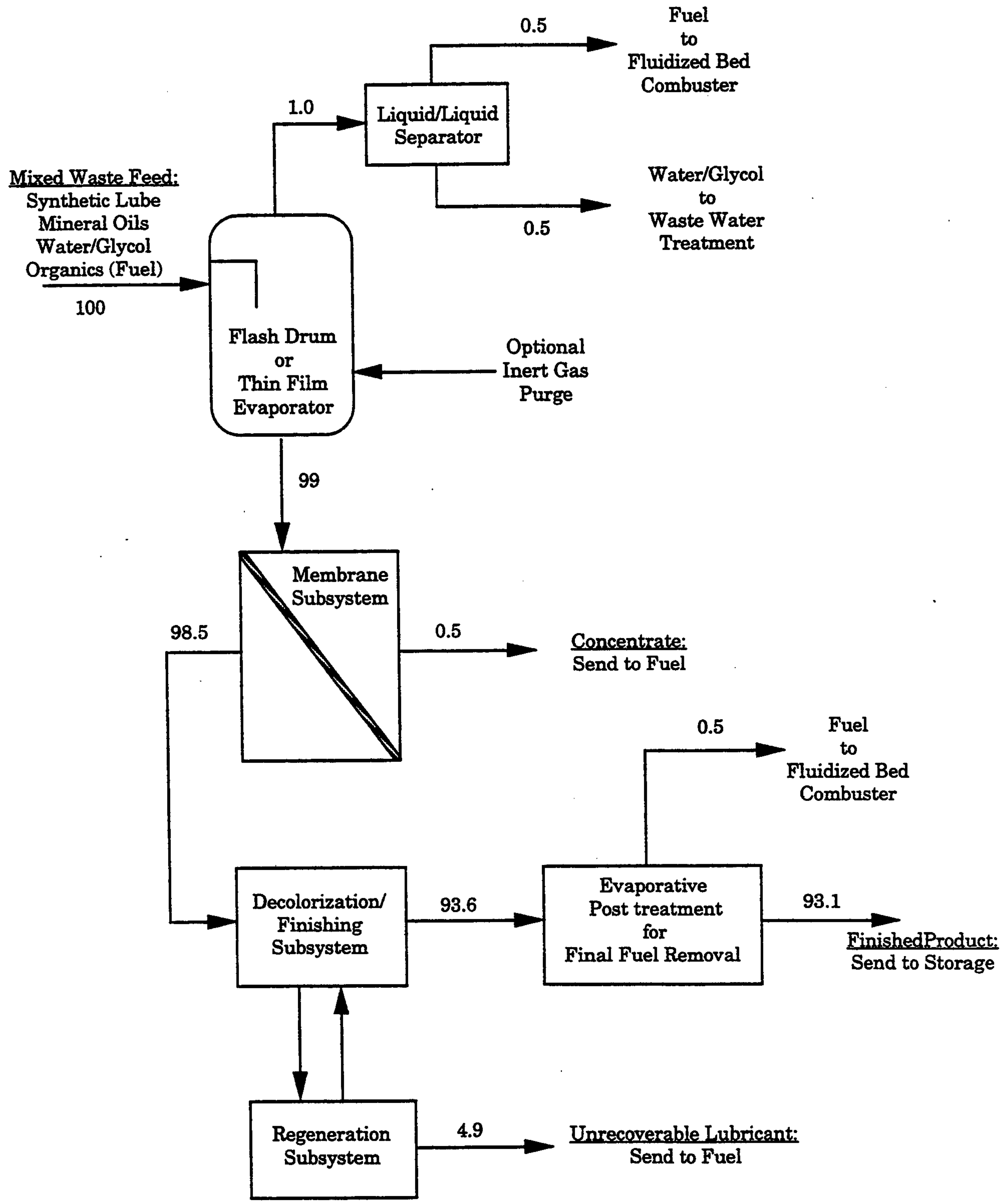


Figure 6: Proposed layout of LubriClear Process for the recovery of synthetic turbine lubricants from spent material. Numbers reflect approximate stream flowrates based upon feed = 100.

APPENDIX I

Material Safety Data Sheet
for
M&P's Re-refined Polyol Ester

MATERIAL SAFETY DATA SHEET

Identity: Re-Refined Polyol Ester 5

SECTION I - SUPPLIER AND OTHER INFORMATION

<u>*Supplier Information*</u>

Name:	Media and Process Technology Inc.
Address:	1155 William Pitt Way
City:	Pittsburgh
State:	Pennsylvania
Zip Code:	15238
Emergency Telephone Number:	(412) 826-3721
Telephone Number for Information:	(412) 826-3721

<u>*Other Information*</u>

Contact Person:	Richard J. Ciora Jr.
Date Prepared:	March 6, 1998
Product Use:	Lubricant Basestock

Identity: **Re-Refined Polyol Ester 5**

SECTION II - INGREDIENTS/IDENTITY INFORMATION

Listed below are the components required to be identified by Federal and/or Pennsylvania law. Other components may be present at less than 1%.

Components	OSHA PEL	ACGIH TLV
Pentaerythritol Ester of caprylic, capric, heptanoic and isopentanoic acids	None listed	None listed

Media and Process Technology Inc. Recommends a TLV of 5 mg/m^3 as a precautionary measure.

This product is listed on the U.S. Toxic Substance Control Act (TSCA) Chemical Substance Inventory.

SECTION III - PHYSICAL/CHEMICAL CHARACTERISTICS

Boiling Point: N/A

Specific Gravity (H2O = 1): 0.97 @ 68°F

Vapor Pressure (mm Hg): N/A

Melting Point: N/A

Vapor Density (Air = 1): N/A

Evaporation Rate (Butyl Acetate = 1): N/A

Solubility in Water @ 25°C: <0.1%

Appearance and Odor: Clear, oily liquid, low odor

Other Information: N/A

Identity: **Re-Refined Polyol Ester 5**

SECTION IV - FIRE AND EXPLOSION HAZARD DATA

Flash Point (Method Used): 450°F (C.O.C.)

Flammable Limits: LEL unknown UEL unknown

Extinguishing Media

Foam, CO_2, dry chemical. Use water spray to cool surface of container.

Special Fire fighting Procedure

Use self-contained breathing apparatus, avoid breathing fumes, vapors, or mists. Water may cause frothing.

Unusual Fire and Explosive Hazards: None

Other Information: Avoid contact with fire and sparks.

SECTION II - INGREDIENTS/IDENTITY INFORMATION

Stability: Stable

Conditions to Avoid: High temperatures

Incompatibility (Materials to Avoid): Strong acids or strong bases

Hazardous Decomposition or Byproducts: CO, CO_2

Conditions to Avoid: None

Other Information: None

Identity: **Re-Refined Polyol Ester 5**

SECTION VI - HEALTH HAZARD DATA

Routes of Entry: Inhalation, skin contact and/or ingestion

Health Hazards (Acute and Chronic): May be an eye or skin irritant. Low ro medium toxity.

Carcinogenicity: No

Signs and Symptoms of Exposure: None known

Medical Condition Generally Aggravated by Exposure: None known

Emergency and First Aid Procedure

Eye Contact: Flush with water for 15 minute. See physician.

Skin Contact: Waste with soap and water thoroughly. See physician if necessary. Remove contaminated clothing.

Other Information:

Ingestion: Do not induce vomiting. Drink plenty of Water. Do not give anything to an unconscious victim. See physician immediately.

Inhalation: Remove to fresh air. See physician if necessary.

SECTION VII - PRECAUTIONS FOR SAFE HANDLING AND USE

Steps to be taken in case material is released or spilled: Dike or contain spill. Apply absorbent. Put in container, close container. Prevent from going into sewer and waterways. Notify proper authorities.

Waste disposal method: Free liquids may not be landfilled. Recover and/or incinerate liquids where possible. Obey local, state and Federal regulations.

Identity: Re-Refined Polyol Ester 5

Precautions to be taken in handling or storage: For industrial use only. Store in cool dry place.

Other precautions: N/A

SECTION VIII - CONTROL MEASURES

Respiratory Protection (Specific Type): Use NIOSH approved organic vapor cartridge respirator.

Ventilation - local Exhaust: Yes *Mechanical (General Exhaust):* Yes

Protective Gloves: Neoprene

Eye Protection: Chemical splash goggles

Other Protective Clothing or Equipment: Neoprene apron. Long sleeved shirt and pants. Safety shoes, hard hat. Safety shower and eyewash station.

Work/Hygiene Practices: Wash with soap and water after contact. Avoid ingestion. Practice good personal hygiene.

Other Information: N/A

SECTION IX - ADDITIONAL INFORMATION

Media and Process Technology Inc. expressly disclaims liability for any injury or loss from the use of this information or the materials described. This data is believed to be reliable, but certain values may vary from source to source. This data is not to be construed as absolutely complete. It is the responsibility of the user to determine the best precautions necessary for his/her applications. This data only refers to the specific materials designated and not to any combinations.

APPENDIX II

Kimes Trading International
Advertisment for M&P Polyol Esters
Appeared in Lubes-n-Greases

HAVING TROUBLE WITH SUPPLY?
HAVE YOU CONSIDERED?