Final Report

Project # DE-FC07-99CH11008 : Soy-Based, Water-Cooled, TC W-III Two Cycle Engine Oil

I. Project Objective

The objective of Project #DE-FC07-99CH11008 was to achieve technical approval and commercial launch for a biodegradable, soy oil-based, environmentally safe, TC W-III performance, water-cooled, two cycle engine oil. To do so would:

A. Develop a new use for RBD soybean oil

B. Increase soybean utilization in North America in the range of 500 K – 3.0 MM bushels.

C. Open up supply opportunities of 1.5 – 5.0 MM bushels worldwide.

These goals have been successfully obtained. An updated project Gantt Chart is seen in Table 1.

II. Background

Terresolve Technologies Ltd. began investigating the use of RBD Soybean oil in two cycle oil applications under a United Soybean Board grant in 1998. A commercial product, EnviroLogic® 440 ISO GD, API TC biodegradable, soy-based, air-cooled, two cycle engine oil was developed and is currently marketed into North America. This formulation for this product contains biodegradable soy oil mixed base oil system (SO-XBO), which delivers the requisite performance for this application.

Project #DE-FC07-99CH11008's focus is the water-cooled, TC W-III two cycle engine lubricant market. Marine outboard engine oil applications for these lubricants are dominated by Mercury Marine, OMC, and Yamaha (Table 2). The total volume of TC W-III oil used world-wide is in the range of 38 million gallons.

The forecast potential for North America and world-wide for a low cost biodegradable TC W-III oil can be seen in Table 3.

III. Major Issues and Targets

A. Environmental

The biodegradability of SO-XBO two cycle engine formulas should be >60% biodegradable as measured by the ASTM D5864 biodegradability test. Petroleum formulas are 10% - 30% biodegradable. Higher priced synthetic ester biodegradable products are >60%.

Reduced emissions volatility in production / handling / use of the SO-XBO should be lower than petroleum oil equivalents and formulated products should result in lower direct VOC emissions in production and handling as well as lower exhaust emissions of particulates in two cycle applications.

Condensation of unburned oil into bodies of water commonly seen as oil sheen, will be reduced, and non-toxic and biodegradable versus petroleum oils.

Vegetable oils from U.S. agriculture plant resources are inherently low in toxicity and have a very high flash point (average 570° F) for increased fire safety. Utilizing appropriate additive technology and SB-XBO produce products with low toxicity and low flammability.

B. Economics

The relative pricing of petroleum based, SO-XBO biodegradable, and synthetic biodegradable water-cooled, two cycle engine oils can be seen in Table 4. The SO-XBO fluid is targeted to deliver top tier biodegradable synthetic oil performance at ~ ½ of the cost.

Commercially, several major oil marketers and OEM's have entered the synthetic biodegradable market. Initial estimates of developed formulas project a selling price of a formulated two cycle product as approximately 50% less than the current price of competitive biodegradable, synthetic formulas with equivalent performance.

A qualified and approved NMMA TC W-III two cycle engine lubricant using a soy oil-base oil system will comply with the most stringent regulatory requirements for use in environmentally sensitive areas.

C. Technical Performance

The use of RBD soybean oil in lubricant applications has been limited due to its inherent performance deficiency regarding oxidative / thermal stability characteristics. The use of SO-XBO and performance additives in formulations should fully address oxidative and thermal stability concerns surrounding the use of RBD soybean oil in water-cooled TC W-III two cycle performance applications. The TC W-III performance requirements are seen in Table 5.

Previous base oil evaluations showed that mixtures of biodegradable hydrocarbons and RBD soybean oils (XBO) gave performance required for TC application at lower cost than synthetic ester biodegradable systems. The use of this SB-XBO was the starting point of the formulation / testing strategy developed in this work.

Commercially, ashless type of performance additives are required for TC W-III performance. Ash containing additives cause spark plug fouling / pre-ignition. The optimization of detergent / oxidation inhibitor levels in combination with an ashless performance additive and the use of high oleic soybean oil were target variables for study.

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The use of soybean oil in lubricant formulations had not been seen to any significant commercial degree until the introduction of EnviroLogic® 440 two cycle lubricant, due to the low content of oleate and the relatively high content of linoleate and linolenate ester components in the oil. This composition deficiency affects primarily the thermal and oxidative stability of the oil in application. The thermal and oxidative weaknesses of a base oil manifest themselves in several ways in a finished lubricant:

- dirt / deposits in an engine
- short fluid use life
- abnormal fluid viscosity increases leading to deterioration of lubricant performance

A. Testing: Refer to Table 7

1. Phase I: Laboratory Physical / Bench Testing (10-99 - 3-00)

Formulations 124-02, 124-03 were developed using the optimized SB-XBO base oil previously developed (EnviroLogic® 440) and Ashless TC W-III performance additive technology.

These fluids were tested against several key tests required for TC W-III approval. As is seen from Table 7, fluid # 124-03 gave promising performance:

a. Viscometric / physical testing – Pass

b. Rush protection – Pass

c. Filter plugging – Pass

d. Mercury 15 HP – Pass

At this junction, due to this performance, fluid # 124-03 was selected for field testing.

2. Phase 2: Field Testing of # 124-03 (4-00 - 12-00)

A summary of the field test plan is seen in Figure 1. This plan was implemented in April, 2000 working directly with the Mercury Marine Corporation using their state of the art Optimax 150 HP engine model. The test engine running on # 124-03 developed cylinder compression loss in a single piston at ~200 hours. The engine trial was halted and the piston replaced. The engine was put back into service (3rd quarter, 2000). The engine suffered additional compression loss at 230 hours. The trial was halted.

The engine running on # 124-03 was torn down for inspection. As shown in Table 7, the pistons showed heavy deposits, there were multiple stuck rings, piston scoring, cylinder galling, but good bearing condition.

Failure analysis indicated that a carbonaceous build-up on the exhaust side of the pistons occurred leading to piston / cylinder scoring, compression loss and engine failure. These results show a requirement for increased oxidative stability and detergency in the # 124-03 lubricant formula.

Candidate formula # 124-214 was developed and put into field trial in a new Mercury Marine 150 HP engine. This formula was # 124-03 plus supplemental detergent and lubricity additive. At 200 hours the engine was torn down and inspected. No engine compression loss was observed. As is seen in Table 7, the engine trial was rated a fail, but significant improvement in formulation performance was observed. Less piston deposit buildup was observed with no scoring or stuck rings.
Higher levels of detergent / lubricity additive (# 124-214) gave directional engine performance improvement.

Formulation # 124-273 was then developed. This product contained further detergent and oxidative inhibitor supplements onto # 124-214 core formulation in an effort to optimize overall lubricant performance.

Formulation # 124-273 @ 200 hours testing in a Mercury Marine 150 HP Optimax was performed. Upon engine tear down inspection this formulation indicated a fail rating due to moderate piston deposit formation. Formula # 124-273 contained a 1.0% detergent supplement. Other than the deposit formation, the engine operated flawlessly (See Table 7).

3. <u>Phase 3: (1-01 - 6-01)</u>

At this juncture our field testing procedure was reviewed. The following conclusions were reached:

1. Engine field testing of candidate formulations was effective in predicting performance improvements in the areas of engine deposits using SBO base oils. Detergent and lubricity improvements were obtained.

2. Field testing, though effective, was slow.

3. The use of screen testing of fluid candidates / formulation adjustments was deemed prudent in terms of project time restrictions.

4. Screen test evaluation was scheduled using the following tests:

    a. <u>West Bend two cycle engine</u>

This is a relatively inexpensive procedure used in petroleum two cycle oil development to screen for a fluids tendency for engine deposits.

Use of the West Bend vs. field trials to screen for formulation effects was faster than field trial approach. Results of candidates # 124-273, # 125-06, # 125-167, and # 125-195 are shown in Table 7.

This evaluation determined that the West Bend test could not be used as a screen test.

b. TGA / PDSC testing (Table 7)

Thermal / Gravimetric Analysis (TGA) measures % wt. oil combusted at a given temperature. Pressure Differential Scanning Calorimetry (PDSC) measures the onset temperature to oxidation of a fluid. These are relatively simple, bench test techniques which can be used to screen oil performance. The approach is to generate a TGA / PDSC profile of a known "pass" or good performing baseline fluid, and try to match that with a development candidate.

The TGA / PDSC profile of the Mercury Quicksilver oil was used as the "pass" baseline reference (# 125-195). The results showed that fluid candidate # 126-26, which is core formula # 124-273 with higher level of detergent, matches the TGA / PDSC profile of the Mercury Quicksilver oil.

5. A higher stability, commercially available hydro brushed soybean oil was also secured and included in the testing matrix (# 126-88). As is seen, its TGA /PDSC profile also matched that of the Mercury Quicksilver reference.

4. Phase 4: Development of Candidate # 126-88

Fluid Sample # 126-88 became the focus of our development work in January 2003 (see Table 7). ISO detergency testing on this sample was completed at Southwest Research Institute and compared against sample candidate # 124-273. It shows excellent cleanliness character.

Sample # 124-273 had been established as a "poor" baseline formulation in our development work. This sample had previously been tested in both the lab and in the field showing deficient performance. As is seen in Table 7, the detergency performance on fluid Sample # 126-88 is superior to that of Sample # 124-273.

The engine field trial test location for Sample # 126-88 was changed in mid-2002. Previously, all field trials were being run at Pennekamp State Park in Key Largo, FL using as the field trial test engine on a Mercury

Marine Optimax 150 HP engine. Although Pennekamp had proved to be a successful field trial location, getting the required number of in-service hours on the field trial test engine was taking an inordinate amount of time. When using the Pennekamp field test venue, a 50 hr. engine use operation was taking 5-6 months. This obviously slowed down the completion of this project. Our subsequent plan of running the trial at the Ft. Lauderdale Marine Patrol Division did not work out due to the inordinate amount of time to get their city council approval.

As a result, the field trial testing on Sample # 126-88 was recently completed at Biscayne National Park in Florida in conjunction with the National Park Service. We again ran the Mercury Marine 150 HP Optimax as the test engine for the park boat. The use of this venue for testing allowed a significantly shorter field trial period. Also, we overcame difficulty in securing a Mercury Marine Optimax 150 HP engine through working with the folks at Biscayne and their local engine dealer.

After 100 hours of use by the National Park Service at Biscayne, engine teardown inspection indicated that fluid candidate # 126-88 meets our targeted engine performance (Table 7). The success will now allow Terresolve Technologies to implement our commercial launch of this product, and successfully ends this development project.

5. Technical Conclusions

The technical conclusions reached from the formulations evaluated are as follows:

a. Sample # 126-88 has successfully met the technical targets established for the objectives of this Project #DE-FC07-99CH11008. The fluid will be commercially marketed by Terresolve Technologies as AquaLogic® 460.

b. 100% RBD soybean oil formulations will not meet the technical performance needed for commercial TC W-III two cycle engine performance.

c. The use of SO-XBO formulation approach allows the performance deficiencies of RBD soy oil to be addressed in a way which allows soy oil to be used in commercial, biodegradable TC W-III marine two cycle applications.

d. Detergent substrate type and level are key additive variables in fluid application / performance.

V. Commercial Development

Another key objective for Project #DE-FC07-99CH11008 is the commercialization of the product technically developed, Sample # 126-88 (AquaLogic® 460). Terresolve's commercial strategy consists of the following elements:

A. The contacting of two cycle engine original equipment manufacturers (OEM's) for both private label sales opportunities as well as approval testing of AquaLogic® 460. OEM's identified as part of this initiative are Mercury Marine, Johnson, Evinrude, Yamaha, and others. These OEM partners offer a key potential distribution channel and field test resource. They are a major element of this commercial strategy.

B. Commercial launch of AquaLogic® 460 into the outboard marine market through the Terresolve sales and distribution networks. This will begin 4^{th} quarter 2003.

C. A public relations strategy promoting AquaLogic® 460 has begun and articles about the product have been published in the following periodicals:

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For patentability reasons, this commercial strategy must be carefully managed. There is a twelve month window in which to file patent claims for a potential product invention, once that product has been commercialized. By years end, 2003, a patent claims set for AquaLogic® 460 should be filed with the Patent Office. Failure to do so could negatively effect the product's patentability. For this reason both the intellectual property and commercial development strategies must be executed in parallel.

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Table 6 documents itemized financial costs for the contract period of 3-30-00 to 6-30-03.

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Table 1: Gantt Chart Worksheet

Project	Prepared By	Date
# DE-FOO2-99CH 11008: Soy-Based, Water Cooled, TC W-III Two Cycle Engine Oil	Curtis R. Scharf	6-30-03

Task Name	Start Date	End Date	Status
Contract Start	10-1-99	—	—
Gather Raw Material	10-1-99	1-1-00	Complete
Blend Study	12-1-99		Complete
Physical / Chemical Testing	12-1-99		Complete
Engine Testing	12-1-99		Complete
Contact OEM Partners	2-1-00		Ongoing
OEM / Field Testing	4-1-00		Complete
OEM Visits	4-1-00		Ongoing
Formulation Optimization	6-1-00		Complete
Environmental Evaluation	10-1-00		Ongoing
Communications Plan	12-1-00		Ongoing
Distributor Strategy	1-1-01		Ongoing
Press Release	1-1-01		Ongoing
Production Start	8-1-01		Pending
Contract Completion	9-1-01	6-30-03	

Table 2

OEM Share

TC W-III Worldwide Market

Players	% Total of TC W-III (gallons)
• Mercury Marine	35 (13.3 MM)
• OMC (Johnson / Evinrude)	25 (9.5 MM)
• Yamaha	25 (9.5 MM)
• Others	15 (5.7 MM)
	100% (38.0 MM)

Table 3
TCW-III Performance Oil; Volume / Potential

	Current Technology	Proposed Technology
Petroleum TC W-III marine, two cycle engine oils	North America – 15 million gallons Worldwide – 38 million gallons	North America – 7.5 million gallons Worldwide – 19 million gallons
Biodegradable, TC W-III	North America – <1 million gallons Worldwide – <1 million gallons	North America – 7.5 million gallons Worldwide – 19 million gallons

Increase soybean utilization:

North America - 500 K – 3.0 MM bushels.

Worldwide - 1.5 – 5.0 MM bushels.

Table 4

Economics - Base Fluids

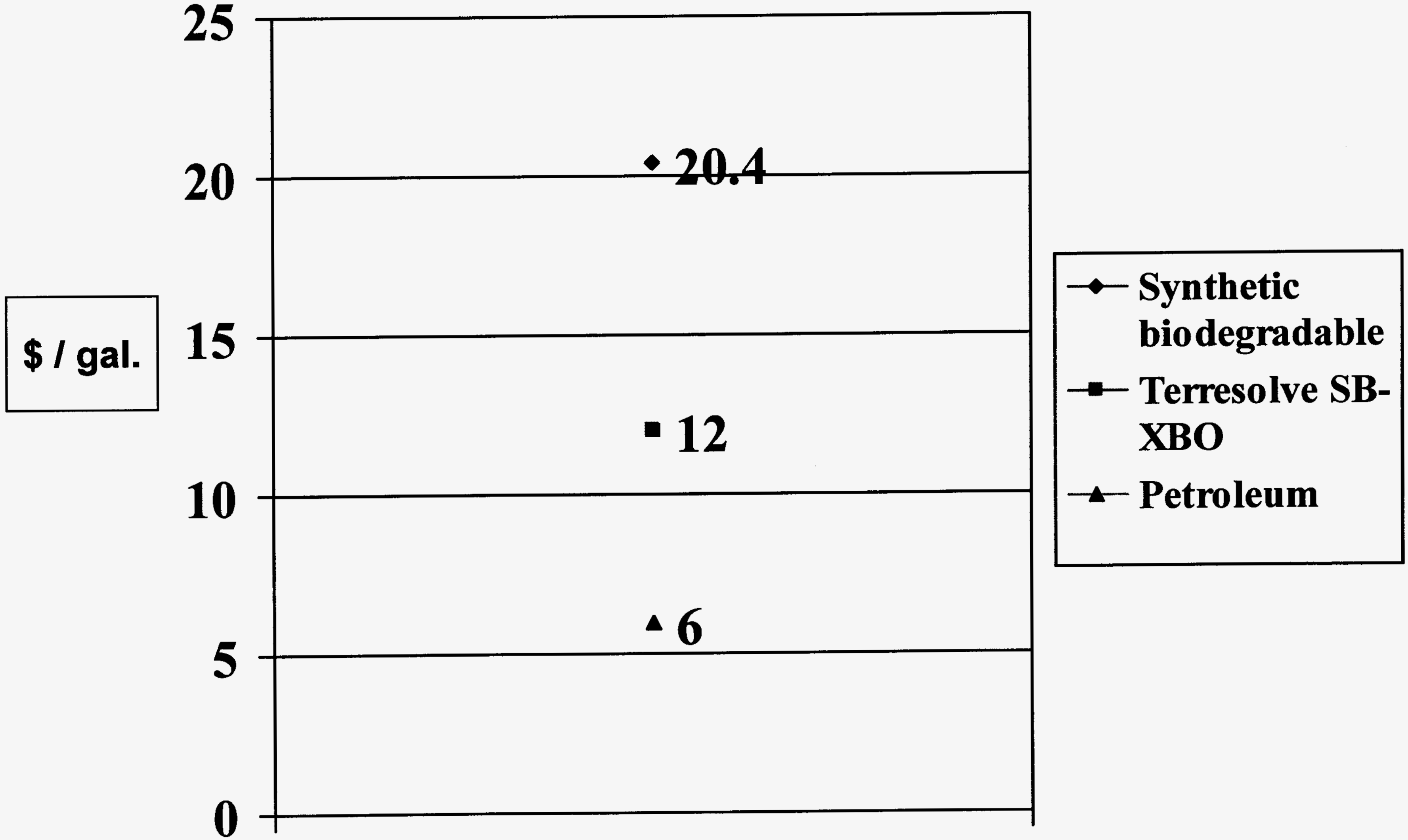

Table 5: TC W-III Performance Requirements

1. Physical/Chemical* - Viscosity
2. Lubricity
3. Mercury 15 HP*
4. OMC 40 HP
5. OMC 70 HP
6. Preignition
7. Compatibility
8. Filterability*
9. Fluidity
10. Miscibility
11. Rust*
12. Identification

*Tests used for initial formulation screening

Table 6

Summary of Expenses

9/1/99 – 6/30/03

	Personnel	Direct Expense	Testing	Total
DOE	$ 164,350	$ 39,667	$ 107,933	$ 311,950
TTL	$ 206,000	$ 44,255	$ 79,470	$ 329,725
USB	$ -	$ -	$ 45,548	$ 45,548
Total	$ 370,350	$ 83,922	$ 232,951	$ 687,223

Itemized Summary of Direct Expenses

9/1/99 – 6/30/03

Direct Cost	DOE	TTL	Total
Travel	$ 16,900	$ 25,350	$ 42,250
Raw Material	$ 13,200	$ 9,765	$ 22,965
Publication	$ 1,250	$ 300	$ 1,550
Telecommunication	$ 1,450	$ 2,850	$ 4,300
Freight	$ 6,867	$ 5,990	$ 12,857
Total	$ 39,667	$ 44,255	$ 83,922

Figure 1: Field Testing of # 126-88

a. Terresolve Technologies / Mercury Marine / Lubrizol partnership

b. Field Test Design

Equipment	Test Site	Type of Operation	Target Hours
Mercury Optimax 150 HP	Key Largo, FL; Biscayne National Park	Deep sea fishing, recreation boating, scuba diving, park patrol.	100 hrs. engine inspection

1. **Field Test Objectives**

   a. Proof of performance / justification for initiation of full TC W-III approval ($100-150K).

   b. Mercury Marine acceptance – Biodegradable “Quick Silver” credentials.

2. **Field Test Results** - Successful performance

Table 7: Soy oil based TC W-III Two Cycle Oil Development

Terresolve ID	124-02	124-03	124-214	124-273	125-106	125-167	125-195	126-26	126-88
Components (%wt)									
SB-XBO	83.0	82.0	80.5	78.2	69.3	78.2	X	79.5	79.5
Inhibitor A	2.0	2.0	2.0	4.0	4.0	2.0	X	2.0	2.0
Additive A	15.0	X	X	X	X	X	X	X	X
Additive B	X	16.0	16.0	16.0	16.0	16.0	X	16.0	16.0
Additive (detergent A)	X	X	0.5	0.8	0.8	X	X	X	X
Additive D (lubricity agent)	X	X	1.0	1.0	0.5	0.5	X	0.5	0.5
Methyl soyate	X	X	X	X	9.4	X	X	X	X
Mercury Marine QuickSilver Fluid	X	X	X	X	X	X	100.0	X	X
Addtive (detergent B)	X	X	X	X	X	0.5	X	X	X
Additive (detergent C)	X	X	X	X	X	X	X	2.0	2.0
Blue dye	100 ppm	100 ppm	100 ppm	100 ppm	100 ppm	100 ppm	100 ppm	100 ppm	100 ppm
	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
Physical Tests									
Kinematic Viscosity (D445)									
cSt@100C	6.6	8.0	8.7	8.0	9.1	8.0	8.5	8.5	8.6
Brookfield Viscosity (D2983)									
cPs@-25C	4810	4800	4700	4750	2540	4500	--	--	4750
Flash Point (D97)									
F	>450	>450	>450	>450	>400	>400	>400	--	420
Rust Test									
Candidate oil, %	9.75	0.53	--	--	--	--	--	--	10.53
Reference oil, %	8.79	8.79	--	--	--	--	--	--	8.79
Filter Plugging Test									
%change	-1	-0.5	--	--	--	--	--	--	-0.5
PDSC (Fluid Oxidation)									
Onset Temperature (°C)	--	246.6	--	267.1	251.6	--	249.0	247.1	246.9
Thermogravimetric Analysis (TGA)									
%off@200C	--	3.7	--	3.7	0.8	--	0.7	1.3	1.4
@300C	--	21.6	--	28.6	13.9	--	13.2	16.2	17.4
@400C	--	92.1	--	93.9	70.3	--	86.4	86.5	87.4
@425C	--	95.4	--	96.8	86.3	--	94.9	94.9	95.8
@450C	--	97.7	--	99.2	99.2	--	98.7	98.7	99.2
@475C	--	98.2	--	99.6	99.8	--	99.7	99.7	99.7
Engine Tests									
Mercury 15 HP (100 hrs.)									
Stuck rings	Pass	Pass	--	--	--	--	--	--	--
Scuffing	0%	0%	--	--	--	--	--	--	--
NMMA bearing stickiness	Pass	Pass	--	--	--	--	--	--	--
Compression loss	Fail	Pass	--	--	--	--	--	--	--
West Bend Two Cycle Engine Test	--	--	--	Fail	Fail	Fail	Pass (Repeat FAIL)	--	--
Honda Dio 3 hr. ISO Detergency Test									
Detergency Index	--	--	--	76	--	--	--	--	94
Field Tests									
Mercury Optimax 150 HP (200 hrs.)									
Rating	--	Fail	Fail	Fail	--	--	Pass	--	Pass
Overall piston condition	--	Very poor	Poor	Poor	--	--	Good	--	Good
Failure analysis	--	Heavy piston deposits	Moderate piston deposits	Moderate piston deposits	--	--	Very light deposit	--	Very light deposit
		Stuck rings	No stuck rings	No stuck rings	--	--	No stuck rings	--	No stuck rings
		Piston scoring	No scoring	No scoring	--	--	No scoring	--	No scoring
		Piston scratching	Very light scratching	No piston scratching	--	--	No piston scratching	--	No piston scratching
		Cylinder damage	No cylinder damage	No cylinder scratching	--	--	No cylinder scratching	--	No cylinder scratching