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February 12, 2014

RE: Biodegradable, Toxicological, Bio-accumulation up-dated for VGP EALs

Vessel General Permit (VGP): On 19 December 2013, the United States Environmental Protection Agency's (EPA) VGP took effect. As defined in Appendix A of the permit, "Environmentally acceptable lubricants" (EALs) means lubricants that are "biodegradable" and "minimally-toxic" and are "not bio-accumulative". In addition to the definitions in the VGP, products meeting the permit's definitions of being an "Environmentally Acceptable Lubricant" include those labeled by the following labeling programs: Blue Angel, European Ecolabel, Nordic Swan, the Swedish Standards SS 155434 and 155470, Convention for the Protection of the Marine Environment of the North-East Atlantic (OSPAR) requirements, and EPA's Design for the Environment (DfE). It has been also recognized by the industry and EPA that there are some discrepancies between the VGP and other labeling programs. One example of this came with a bio-accumulation question to EPA's Ryan Albert, who is listed as a contact for EAL questions. Because the VGP definition wasn't clear on the question, he recommended checking and using the European Ecolabel requirements. After reviewing the "European Ecolabel application pack for lubricant documents, and it was stated on page 27 under "Data and documents to be submitted on the bioaccumulation potential", that "Data on the bioaccumulation must be submitted if the substance is not ultimately nor inherently biodegradable". Based on this statement and the fact that the final fluid has been tested to be ultimately and/or readily biodegradable, bioaccumulation data is not required and this has been confirmed in an EPA e-mail copied to RLI.

History: Since 1991, Renewable Lubricants Inc. (RLI) has been under an intensive R&D program collecting confidential data for RLI's biobased technology. Many different additives and base oils were reviewed for performance and toxicological properties. In 1993, RLI received a USA/DOD Grant where RLI collaborated with Lubrizol and State Universities. During the R&D, Lubrizol and RLI, under a confidentiality agreement, reviewed and tested many additives and shared the data. The R&D continued with Grants from the United Soybean Board and additional collaborations from Dow AgroSciences, Ohio Soybean Council, Battelle Institute, National & Ohio Corn Growers Associations, and other government organizations. A later collaboration with Chevron Phillips Chemical provided 4 years of STLE published papers on the performance and biodegradability of the technology. Eco-toxicity and Biodegradability Tests were conducted and shared on many different additives in vegetable oils and synthetic base oils during the R&D. Most of RLI's formulas total .5 to 4 percent additive concentration. The remaining percentages are made up of biodegradable base oils that meet or exceed the environmental requirements for the U.S. EPA acute toxicity criteria and/or food grade NSF approval.

RLI's Bio-HVO and Bio-HVO2 were specially formulated to offer the lowest toxicity in aquatic conditions (see HVO and HVO2 data sheets). The HVO and HVO2 are 96-97% biobased according to the formulations and contain RLI's patented additive technology that is in most all RLI Products. Because the HVO and HVO2 are close to 100% biobased, they performed best in the Eco-toxicity and Biodegradability Tests. In fact, these two formulas ran parallel to the reference canola in the Biodegradability Tests. In outside independent lab testing performed, RLI was given the Biodegradability from 80% to 90% in 28 days in the ASTM-5864 and the OECD 301B (Mod Sturm). In the CEC-L33-T-82 and CEC-L-33-A-94 the range was from 90% to 100% in 21 days.

Ecotoxicity: Bio-HVO and Bio-HVO2 Hydraulic Fluids can be considered the top performance baseline for RLI's additive technology. RLI's Bio-Penetrants, Bio-Gear Oils, Bio-Fleet Hydraulic Fluids, Bio-Ultimax Hydraulic Fluid line of products and many other RLI product formulas contain the same types of additive components as the Bio-HVO and Bio-HVO2 Hydraulic Fluids. RLI's fluids, including additive technology evaluation for each constituent substance, have been reviewed and tested to be environmentally non-toxic and exceed the U.S. Fish and Wildlife Service and the U.S EPA VGP acute toxicity criteria100 mg/L for hydraulic fluids and 1000 mg/L for total lost fluids. In addition, the Bio-Ultimax 1000 ISO 32 Hydraulic Fluid was reviewed and tested by Northrop Grumman in the U.S Navy Offgassing Analysis (Report test 20050112NG01) at the U.S. Naval Warfare Testing Florida and was approved by the Navy Medical as non-toxic for the Navy SEALS diver safety (See Department of the Navy, Naval Sea Systems Command 09-399A38-0127 ECP ASDS-ECP-081 Revision B, Hydraulic Oil Replacement).

Ecotoxicity OECD 201, 202, and 203 for Bio-HVO and Bio-HVO2 (includes the same additives in all RLI hydraulic and stern tube fluids)Fathead minnow, 96h LC50, ppm>10,000 ppmDaphnis magna, 48 h, EC50, ppm>10,000 WAFSludge respiration inhibition, EC50, ppm>10,000 ppm

In addition to evaluating the fully formulated fluids, the formulation can be <u>evaluated for each constituent substance, rather than the</u> <u>complete formulation and main compounds.</u> The EPA 2013 VGP guidelines for EALs provides in **Appendix A Definition** "Minimally-Toxic" means a substance must pass either OECD 201, 202, and 203 for acute toxicity testing, or OECD 210 and 211 for chronic toxicity testing. For purposes of the VGP, equivalent toxicity data for marine species, including methods ISO/DIS 10253 for algae, ISO TC147/SC5/W62 for crustacean, and OSPAR 2005 for fish, may be substituted for OECD 201, 202, and 203. If a

substance is evaluated for the formulation and main constituents, the LC50 of fluids must be at least 100 mg/L and the LC50 of greases, two-stroke oils, and all other total loss lubricants must be at least 1000 mg/L. If a substance is evaluated for each constituent substance, rather than the complete formulation and main compounds, then constituents comprising less than 20 percent of fluids can have an LC50 between 10-100 mg/L or a no observed effect concentration (NOEC) between 1-10 mg/L, constituents comprising less than 5 percent of fluids can have an LC50 between 1-10 mg/L or a NOEC between 0.1-1 mg/L, and constituents comprising less than 1 mg/L or a NOEC between 0-0.1 mg/L.

RLI has reviewed and evaluated the formulation main constituents and each constituent substance and the products listed on **RLI's VGP Vessel General Permit Compliance Declaration** will meet the **VGP** "Minimally-Toxic" requirements.

Bio-accumulation: RLI has evaluated each constituent substance US MSDS, new US SDS and EU SDS provided by the suppliers. The information under the bio-accumulative shows no data, or not to be expected to bio-accumulate, or no potential to bio-accumulate. In addition to searching and reviewing SDSs, RLI was given from major chemical companies the listings of constituent substance (additive components and some base oils) that are listed for use as allowable lubricant substances list or additives and allowable percentages in compliance for German Blue Angle, Swedish Standards, or European Ecolabel, which are included within the VGP as EALs. Most all RLI lubricant substances are in these component listings. These listings included substances in question by RLI or substance where the SDS showed no bio-accumulative data. Using this information, RLI has reviewed and evaluated the formulation main constituents, and each constituent substance and there percentages, and has reviewed the European Ecolable application pack for lubricant documents as recommended by the EPA, and based on the fact that the final fluid has been tested to be ultimately and/or readily biodegradable, the products listed on **RLI's VGP Vessel General Permit Compliance Declaration** will meet the **VGP** "not bio-accumulative" requirements.

Biodegradability: For the purpose of the VGP regarding environmentally acceptable lubricants and greases, <u>biodegradable means</u> <u>lubricant formulations that contain at least 90% (w/w (weight in weight concentration)) or grease formulations that contain at least</u> 75% (w/w) of a constituent substance or constituent substances (only stated substances present above 0.10% shall be assessed) that each demonstrate either the removal of at least 70 percent of dissolved organic carbon, production of at least 60 percent of the theoretical carbon dioxide, or consumption of at least 60 percent of the theoretical oxygen demand within 28 days. Acceptable test methods include: Organization for Economic Co-operation and Development Test Guidelines 301 A-F, 306, and 310, ASTM 5864, ASTM D-7373, OCSPP Harmonized Guideline 835.3110, and International Organization for Standardization 14593:1999. Again there are discrepancies between the VGP and other labeling programs. The VGP does not directly address the CEC-L33-T-82 and CEC-L-33-A-94 methods for biodegradability, but these CEC tests are accepted in the German Blue Angle for the readily and inherently biodegradable tests and some petroleum base oils can be used in the German Blue Angle where the European Ecolable is not in favor of petroleum base oils. The VGP doesn't address whether petroleum derived base oils are allowable or not allowable, but it is obvious the EPA's main purpose in the VGP is to address the biodegradability and toxicity of the formulas.

Included with the previous independent biodegradability tests, that were conducted and shared on many different additives in vegetable oils and synthetic base oils, in 2002, RLI in collaboration with Chevron Phillips Chemical constructed at RLI's laboratory the ASTM D-5864 Biodegradability Test. From November 2002 to January 2005 RLI ran 42 Biodegradability Tests (attached). Two weeks prior to officially starting ASTM D-5864, sludge microorganisms can be pre-exposed to the test samples in order to enhance results as part of an optional inoculum pre-adaptation techniques listed in ASTM 5864 Sec 8.3.1. Inoculum pre-adaption has been reported to improve biodegradability up to 10 percent in samples expected to be slow to adapt and biodegrade in 28 days. Because of the higher biobased and biodegradable content in these formulations and the necessity to save time and money, the optional inoculum pre-adaptation techniques of RLI's tests to enhance biodegradability results of the test samples. The last 6 samples that used the optional inoculum pre-adaptation techniques are reported in RLI's biodegradability summary, including data showing how the inoculum pre-adaptation technique improved the biodegradability of synthetic PAO 2 and PAO 4 approximately 10%.

The biodegradability of RLI's Bio-Products will vary depending on formulation and viscosity. When looking at the ultimate biodegradability averages at the end of the test, RLI's hydraulic fluids biodegraded very close to the reference canola oil. The Bio-Ultimax 1000 ISO 68 Hydraulic Fluid that used the optional inoculum pre-adaptation techniques, biodegraded 30% in 6 days, 44% in 10 days, and over 60% in 15 days and 83% in 28 days showing very rapid Ultimate/Readily biodegradability. The biodegradability reports are attached and the products listed on **RLI's VGP Vessel General Permit Compliance Declaration** will meet the **VGP** "biodegradable" requirements.

Respectfully Submitted,

William Garmier, Vice President Renewable Lubricants, Inc.