I. Scope: This policy and procedure guideline establishes a standard method for extracting and analyzing lubricating oils encountered in neat liquids and debris samples. Lubricating oils are derived from a heavy boiling fraction of crude oil. They generally range from C_{18} to C_{40+} and contain an unresolved envelope of hydrocarbons. There are several types of lubricating oils which include conventional lubricating oils (from crude oil), synthetic lubricating oils (modified petroleum components), and synthetic blends (mixture of conventional and synthetic). Lubricating oils may also contain additional components such as glycols and esters that are added to alter the properties of the oil. Lubricating oils are not volatile and may not be detected using typical fire debris headspace concentration techniques. If lubricating oils are suspected, a solvent extraction should be performed after routine ignitable liquid or explosive analysis. Though some components of lubricating oils can be observed using a typical gas chromatograph-mass spectrometer (GC-MS), a high temperature instrument (HTGC-MS) may be necessary for identification of heavier oils. For comparisons of lubricating oils, HTGC-MS analysis is required.

II. References:

ASTM E1386 - Standard Practice for Separation and Concentration of Ignitable Liquid Residues from Fire Debris Samples by Solvent Extraction

ASTM E1618 - Guide for Ignitable Liquid Residues in Extracts from Fire Debris Samples by Gas Chromatography-Mass Spectrometry

Gary JH, Handwerk GE. *Petroleum Refining Technology and Economics*. 4th edition. New York: Marcel Dekker, 2001.

Reardon MR, Allen L, Bender EC, Boyle KM. "Comparison of Motor Oils Using High-Temperature Gas Chromatography-Mass Spectrometry." *J Forensic Sci* 2007; 52:656-663.

Reardon MR, Bender EC. "Differentiation of Composition of C-4 Based on the Analysis of Process Oil." *J Forensic Sci* 2005; 50:564-570

Hibbard R, Goodpaster JV, Evans MR. "Factors Affecting the Forensic Examination of Automotive Lubricating Oils." *J Forensic Sci* 2011; 56: 741-753.

Material Safety Data Sheet for Pentane

III. Apparatus/Reagents:

A. Solvent - Pentane or other appropriate organic solvent
B. Drying agent - anhydrous sodium sulfate or equivalent
C. Filter apparatus - free of extractable hydrocarbons

D. Beakers or similar containers- free of extractable hydrocarbons
E. GC-MS instrument with capillary column capable of providing sufficient information to identify and compare (as necessary) lubricating oils (refer to ATF Fire Debris protocols on GC-MS and HTGC-MS)
F. Reference collection of various lubricating oils

IV. Safety Precautions:

Personal protective equipment including but not limited to safety glasses, gloves, and lab coat should be worn.

Pentane should be used in a fume hood and kept away from open flames and sparks.

V. Procedures

The following procedures are suggested for the extraction of neat liquids and debris samples and may be modified depending on sample material and size. HTGC-MS is required for comparisons of lubricating oils (if requested by the submitter).

Neat liquids:

1. Dilute the sample in an appropriate volume of solvent, such as pentane, to obtain acceptable instrument signal.
2. Filter as necessary and analyze the extract by GC-MS and/or HTGC-MS.

Debris Samples:
Lubricating oils may be extracted during routine ignitable liquid testing using passive charcoal adsorption (ATF Fire Debris protocol); however, solvent extraction (ATF Fire Debris protocol) may be necessary to recover heavier oils. See the appropriate protocol for extraction procedure. Solvent extraction of debris should be performed after routine ignitable liquid or explosives analysis.

Co-extraction of interfering compounds may be a concern with some porous matrices using this technique. Clean-up procedures using silica gel cartridges or similar products may be needed to separate lubricating oils from interfering products.

For identification of lubricating oils, samples should be compared to a reference lubricating oil with similar characteristics such as peak distribution and chemical content. Comparisons of lubricating oils must be performed with extreme caution and must include analysis by HTGC-MS. Comparisons should be based on overall chromatographic patterns such as shape of the unresolved envelope and apex position. Minor differences do not necessarily indicate that the two samples are chemically different and may be due to instrumental variation. Additionally, comparisons of lubricating oils may be affected by sample concentration. Oils that are too concentrated will skew the pattern and potentially affect the comparison conclusion. If possible, the compared lubricating oils should be analyzed in triplicate to assess any minor instrumental and concentration variations.

VI. Quality Control/Quality Assurance:

Examiners should follow the Quality Control/Quality Assurance procedures outlined in the ATF Fire Debris protocols for GC-MS and HTGC-MS.

VII. Sources of Error:

Error potentials for GC-MS and HTGC-MS are referred to in the instrument protocols.

Concentration differences may affect conclusions; thus every effort should be made to ensure that the oils being compared are similar in abundance/concentration.

VIII. Extract Storage

All solvent extracts will be preserved with charcoal and returned with the evidence to the submitting agency.