I. Scope: This policy and procedure guideline establishes a standard method for the analysis and identification of waxes and petrolatum encountered in evidentiary samples. Petroleum-derived, paraffin waxes contain normal alkanes above C_{17} with a lesser abundance of branched alkanes. Some waxes, including naturally-derived waxes such as beeswax and soybean wax, may contain a variety of esters, acids, and other oxygenated compounds instead of or in addition to alkanes, the distribution of which may differ from petroleum-derived waxes. Petrolatum, also known as petroleum jelly, is generally comprised of wax and lubricating oil components. Since petrolatum is a semi-solid substance, the analysis procedures are similar to waxes.

II. References

ASTM D5442 - Standard Test Method for Analysis of Petroleum Waxes by Gas Chromatography.

ASTM E 2881 - Standard Test Method for Extraction and Derivatization of Vegetable Oils and Fats from Fire Debris and Liquid Samples with Analysis by Gas Chromatography-Mass Spectrometry

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Giles, JJ, The analysis of waxes and greases using high resolution gas chromatography, Forensic Science Society, Vol 27, 1987, pp 231-9.

Griffin, RME, Doolan, K, Campbell, M, Hamill, J, and Kee, TG, Analysis of wax-based products by capillary gas chromatography-mass spectrometry, Science and Justice, Vol 36, 1996, pp 229-244.

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Kuk, RJ, Analysis of artificial fireplace logs by high temperature gas chromatography, Journal of Forensic Science, Vol 47, 2002, pp 1288-93.

U.S. Customs Laboratory Methods (USCL Method 34-07), Quantitation of paraffin in beeswax and other waxes by high temperature capillary gas chromatography, March 2003.

Wampler, Thomas P. (ed.), Applied Pyrolysis Handbook, Marcel Dekker, Inc., 1995.

Blackledge, R, Cabiness, L., Examination for Petrolatum Based Lubricants in Evidence from Rapes and Sodomies. Journal of Forensic Science, Vol 28(2), April 1983, pp 451-462.

III. Apparatus/Reagents

A. Appropriate solvents, such as trichloroethylene (TCE) or pentane
B. Instruments capable of identifying organic chemical unknowns, including Fourier transform Infrared Spectroscopy (FTIR) and high temperature gas chromatography-mass spectrometry (HTGC-MS).

IV. Safety Precautions

Personal protective equipment such as lab coat, safety glasses and gloves should be available and used, if necessary, when preparing samples and conducting analyses.

When using solvents, such as TCE and pentane, examiners should utilize laboratory hoods to minimize exposure to solvent vapors.

Material Safety Data Sheet (MSDS) references for solvents should be available and read by the user.

Pentane should be kept away from open flames and sparks.

V. Procedures

Wax and petrolatum may be *identified* using physical characteristics (e.g. appearance, texture, solubility, pliability, clarity, melting point) and/or optical properties of the wax crystals using polarized light microscopy (PLM) in conjunction with at least one of the following techniques:

- Fourier transform infrared spectroscopy (FTIR): refer to ATF Trace protocol
- High-temperature gas chromatography-mass spectrometry (HTGC-MS): refer to ATF Fire Debris protocol
- Sample derivatization and analysis of fatty acid methyl esters (FAMES) using GC-MS: refer to ATF Fire Debris protocol (Note: may not apply for petrolatum.)
- Pyrolysis gas chromatography-mass spectrometry (PyGC-MS): refer to ATF Trace protocol

Samples to be analyzed by HTGC-MS will be dissolved in a solvent at an appropriate concentration, such as approximately 0.1% by weight. Heat may be necessary for complete dissolution of the solid sample. This preparation may be modified at examiner discretion.

Samples to be analyzed by the FAME GC-MS must be extracted and derivatized according to the ATF Fire Debris protocol on Vegetable Oils.

Paraffin waxes and petrolatum may be extracted during routine ignitable liquid testing using

passive charcoal adsorption (ATF Fire Debris protocol) or solvent extraction (ATF Fire Debris protocol). If a physical examination of the debris does not result in finding solid material to further analyze, the wax or petrolatum should be considered indicated only and an identification should not be made. If petrolatum is suspected, examiners should also refer to the analysis of lubricating oils protocol (ATF Fire Debris protocol) for further guidance on sample preparation.

Wax and petrolatum comparisons must be performed with extreme caution on solid/semi-solid samples or visible residues only and shall include analysis by HTGC-MS, PyGC-MS, or FAME GC-MS (if applicable). The identification and comparison of waxes and petrolatum by GC-MS is done using overall pattern comparison and the retention time and spectral data compared with a known reference. Minor differences in GC-MS patterns do not necessarily constitute a conclusion of "chemically different". If possible, at least three replicates of the questioned and known samples shall be prepared in a similar manner and analyzed to determine if differences seen can be attributed to the samples or the instrument. While minor heating for sample dissolution is acceptable, it should be noted that waxes can also be affected by excessive heat, resulting in changes in the chromatographic pattern. Refer to Section VIII, Part 2 for suggested wording for comparisons. Note that sample concentration may affect the resulting data. When feasible, samples should be prepared so that the extract concentrations are as similar as possible.

VI. Quality Control

Examiners should follow the appropriate instrument protocols for quality control for each instrument.

VII. Sources of Error

Error potentials for each instrumental technique are referred to in the instrument protocols.

Sample concentration may adversely alter the chromatographic data and affect the resulting conclusion. Care should be taken to ensure that samples being compared are prepared in a similar manner and at a similar concentration.