I. Scope

This policy and procedure guideline establishes a standard method for the analysis and comparison of Composition C-4. Composition C-4 is frequently encountered in casework, requiring the identification of its components and sometimes differentiation and comparison of questioned and known samples.

Composition C-4 contains cyclotrimethylene trinitramine (RDX), dioctyl sebacate or adipate (DOS or DOA), polyisobutylene (PIB), and oil (known as a process or lubricating oil). A taggant, usually dimethyl dinitrobutane (DMDNB), is present in Composition C-4 manufactured in 1996 or later.

Both the military and commercial companies produce Composition C-4, but due to the distribution process, it is generally not possible to determine the difference chemically. (REDACTED)

A variety of methods can be used to identify the components, such as XRD, FTIR, GC-MS, HTGC-MS, and LC. Isotope ratio-mass spectrometry (IR-MS) may also be used to gather further information on the RDX and plasticizer. Examiners should refer to the Standard Approach for the Examination of Explosives as well as the appropriate instrument protocols.

II. References

Department of Defense Standard Practice, MIL-STD-1168B. Ammunition lot numbering and ammunition data card. 1998.

International Civil Aviation Organization. Convention on the marking of plastic explosives for the purpose of detection. 1991.

Military Standard, MIL-STD-1461E. Ammunition manufacturers and their symbols. 1990.

Military Specification, MIL-C-45010A (MU). Composition C-4. 1963.

Military Specification, MIL-P-14536 (Ord). Polyisobutylene Binder. 1957.

Keto RO. Improved method for the analysis of the military explosive Composition C-4. J Forensic Sci 1986;31:241-49.

Midkiff CR, Washington WD. Systematic approach to the detection of explosive residues. IV. Military explosives. J AOAC 1976;59:1357-74.

Peimer RE, Washington WD, Snow KB. On the examination of the military explosive, C-4. J Forensic Sci 1980;25:398-400.

Wilson A. personal communication with MR Reardon, 2004.

Reardon MR and Bender EC, Differentiation of Composition C-4 based on the analysis of the process oil, Journal of Forensic Sciences, 50(3):564-570, 2005.

III. Validation

The various instruments mentioned in this protocol are all well-known and scientifically accepted techniques for the analysis of various materials, including those found in Composition C-4. Standards and known Composition C-4 samples have been analyzed on all instruments.

IV. Apparatus/Reagents

- Appropriate solvents, such as pentane, acetone, and chloroform
- Instruments capable of identifying the major components in Composition C-4, including FTIR, XRD, GC-MS, HTGC-MS, and LC-MS
- GC-MS, specifically HTGC-MS, capable of providing sufficient information to compare process (lubricating) oils

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

VI. Procedures

Examiners should refer to the current Standard Approach for the Examination of Explosives for a list of instruments that are appropriate to identify each Composition C-4 component. The following protocols should be referenced (in either ATF Explosives or Fire Debris protocols) for the proper instrumental procedures, including performance check, quality control, and sample preparation:

- EGIS
- FTIR
- GC-MS
- High-temperature GC-MS
- LC-MS
- XRD

In order to compare Composition C-4 samples, all major components (RDX, DOA or DOS, PIB, and taggant (if present)) must first be identified using the proper techniques. Given the similarities between DOA and DOS, the FTIR is often not suitable to differentiate the two plasticizers. If distinction is not possible on the FTIR, then the examiner should use HTGC-MS, which will provide retention time and mass spectral data specific to each plasticizer.

If the analysis and identification of all major components results in no differences between the questioned and known samples, then further examination of the process (lubricating) oil is required. The procedure listed below is suggested for the analysis of the process oil and may be modified as needed:

1) Extract approximately 0.5-0.6 g of sample in 5-6 mL of pentane. Smaller extraction amounts are acceptable, as long as the proper ratio of sample to solvent is maintained.

2) Prepare a 1 g silica cartridge by flushing it with approximately 12 mL of pentane. Discard the collected pentane.
3) Pour the sample extract onto the cartridge and add another 5-6 mL of pentane, collecting the filtrate.
4) Analyze the filtrate on the HTGC-MS and compare the resulting questioned and known chromatograms. Concentration of the extract under nitrogen may be necessary, depending on the initial HTGC-MS results.

If further comparison is necessary, samples may be sent to another laboratory for stable isotope ratio analysis or any other technique not currently used in the ATF laboratory system.

VII. Quality Assurance/Quality Control

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

VIII. Sources of Error

Error potentials for each instrumental technique are referred to in the instrument protocols. Extract concentration is important when comparing process (lubricating) oils, since a sample that is too concentrated will produce a chromatogram profile that is distorted from its true shape. Care should be taken to prepare questioned and known samples in a similar manner. Refer to the ATF Fire Debris protocol on the analysis of lubricating oils for further information on comparisons of these oils and sources of error.