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Analytical Division
Research Directorate

Analysis/Evaluation of Metal Fragments and Gloves

A shipment designated 10027C(6), received by the Analytical Division 5 Apr 85 from FSTC, contained twenty-two subsamples. A group of samples identified by the SEA team as of prime interest and all related to suspect chemical munitions were evaluated concurrently. These consist of a threaded metal piece identified with CB 850215-004XA, figure 1, several solid chunks which appear metallic identified with CB850215-002/2XA, figure 1, a second group of similar solid chunks identified with CB850215-002/3XA, figure 1, a third group of similar solid chunks identified with CB850215-002/1XA, figure 1, dark brown granules identified with CB850215-002XA, figure 2 and a pair of surgeons gloves reportedly used in taking and separating these samples identified with CB850215-003SW, figure 3. Subshipment designations for these samples are: 10027C(6)-5, 10027C(6)-6, 10027C(6)-7, 10027C(6)-8, 10027C(6)-9, and 10027C(6)-11 respectively. Each sample was contained in a glass jar, sealed, [redacted] Vapor samples withdrawn from within each sample jar enclosure were subjected to analysis by gas chromatography/mass spectrometry (GC/MS). Portions of each sample were either swabbed or leached with chloroform. Additional portions of each sample were treated similarly with 1:1 methanol:water. The solvent soluble materials were analyzed by GC/MS, ion chromatography (IC), thin layer chromatography (TLC) and infrared spectrometry (IR).

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Sample 10027C(6)-5 (Metal Piece) (CB850215-004XA)

This sample was subjected to swabbing with solvents. The GC/MS spectra of the vapor associated with the sample identified the presence of acetone, xylene, naphthalene, methyl naphthalene, biphenyl and a terpene similar to cadinene. GC/MS analysis of the chloroform solubles identified only an alcohol, possibly a glycol. No detectable ions were separated by IC. No separation of any detectable compounds was evident by TLC. IR identified the presence of water, aliphatic hydrocarbons, a carbonyl band at 1736 cm^{-1}, nitrite NO_2, and inorganic silica, SiO_2. Derivatization with negative ion chemical ionization MS detection was negative for trichothecenes.

10027C(6)-6 (Large Solid Chunks) (CB850215-002/2XA)

A chunk having white, yellow and black surface colorations was leached in chloroform. Another chunk having rust and black surface colorations was leached in 1:1 methanol:water. Upon covering the solid with methanol/water, a degassing was observed, suggesting that the material contained either pores of a size which permitted the solvent to displace air in them or an acid salt which liberated a gas on solution, eg. a carbonate giving CO_2. The GC/MS spectra of the vapors associated with the sample identified the presence of acetone,

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trichlorofluoromethane, chloroform, triethylamine, toluene, chlorobenzene, two isomers of nitrotoluene, a sesquiterpene, naphthalene, diphenylamine, E-hydroxytoluene and an unidentified aliphatic hydrocarbon. Analysis of the chloroform solubles by GC/MS identified the major component as TNT. IC separated no detectable ions. TLC separated two UV absorbers at Rf 0.75-0.80 and 0.81-0.85, supportive of aromatic nitrocompounds. IR analysis identified TNT as the major component with smaller quantities of an aliphatic hydrocarbon, possibly a paraffin wax. Derivatization with negative ion chemical ionization MS detection for trichothecenes was negative.

10027C(6)-7 (Large Solid Chunks) (CB850215-002/3XA)

Chunks similar to those described in 10027C(6)-6 were subjected to the same solvent leaches. A degassing phenomenon similar to that in 10027C(6)-6 was observed in the methanol-water leach. The GC/MS spectra of the vapor associated with the sample identified the presence of acetone, trichlorofluoromethane, either trimethyl benzene or propyl benzene, aliphatic hydrocarbons, naphthalene and one sesquiterpene $C_{15}H_{24}$ Analysis of the chloroform solubles by GC/MS identified toluene as the major component, with lesser quantities of TNT and traces of butanol and benzaldehyde. No detectable ions were separated by IC. TLC separated two UV absorbers similar to those detected in 10027C(6)-6, having Rf=0.72-0.82 and 0.81-0.82. IR analysis identified the presence of TNT and hydrocarbons. Derivatization with negative ion chemical ionization MS detection for trichothecenes was negative.

10027C(6)-8 (Large Solid Chunks) (CB850215-002/1XA)

A chunk having white and black surface colorations was leached in chloroform. Other chunks having white, black and red surface colorations were leached in 1:1 methanol:water. A degassing phenomenon similar to those of the previous two samples was observed in the methanol-water leach. GC/MS spectra of the vapor associated with the sample identified the presence of acetone, xylene, ethyl toluene or propyl benzene, naphthalene, nitrotoluene, a sesquiterpene $C_{15}H_{24}$ and diphenyl. Analysis of the chloroform solubles by GC/MS identified TNT as the major component, with a trace of toluene. No detectable ions were separated by IC. TLC separated the same UV absorbers detected in 10C27C(6)-7. IR identified the presence of TNT. Derivatization with negative ion chemical ionization MS detection for trichothecenes was negative.

10027C(6)-9 (Dark Brown Granules) (CB 850215-002XA)

A few granules were leached in chloroform. Another portion of granules were leached in 1:1 methanol:water. The GC/MS spectra of the vapor associated with the sample identified the presence of acetone, trichlorofluoromethane, xylene, either diethyl benzene or tetramethyl benzene, naphthalenes, methyl naphthalenes, several aliphatic hydrocarbons and 4 or 5 sesquiterpenes, each $C_{15}H_{24}$. Analysis of the chloroform solubles by GC/MS identified toluene, TNT and an alcohol. No

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detectable ions were separated by IC. TLC separated only one component, a UV absorber at Rf 0.76-0.84 similar to one of the components detected in samples 10027C(6)-6,7 and 8. IR identified the presence of aliphatic hydrocarbons, aromatic nitrates, TNT and silicates which appear to be sand from the environment. Derivatization with negative ion chemical ionization MS detection for trichothecenes was negative.

10027C(6)-11 (Surgeon's Gloves) (CB850215-003SW)

The blackened area of two fingers were cut out. One was leached in chloroform, the other in 1:1 water:methanol. The GC/MS of the vapor associated with the gloves identified the presence of acetone, a compound similar to $C_{10}H_{18}O_2$, B-hydroxy toluene (BHT) and dehydro BHT. GC/MS spectra of the chloroform solubles identified toluene, BHT and 1,1,3-trimethyl-3 phenylindene. No detectable ions were separated by IC. TLC separated one component, identical with that detected in sample 10027C(6)-9. IR spectra identified the presence of high concentrations of hydrocarbons, possible aromatic structures, a C-O band and C=O band (1745 cm^{-1}), with no evidence of nitrocompounds. Derivatization with negative ion chemical ionization MS detection for trichothecenes was negative.

Conclusion:

No evidence of any known CW agent, agent degradation product or trichothecene was detected. The primary component of each of these samples was TNT and structures normally associated with it in a munition propellant. The detection of sesquiterpenes in several samples could be indicative of either a natural contamination of the samples or inclusions of a natural product, eg. trichothecenes, in the munition fill. As evidenced from the photographs, it is difficult to determine the source of the terpene structures.

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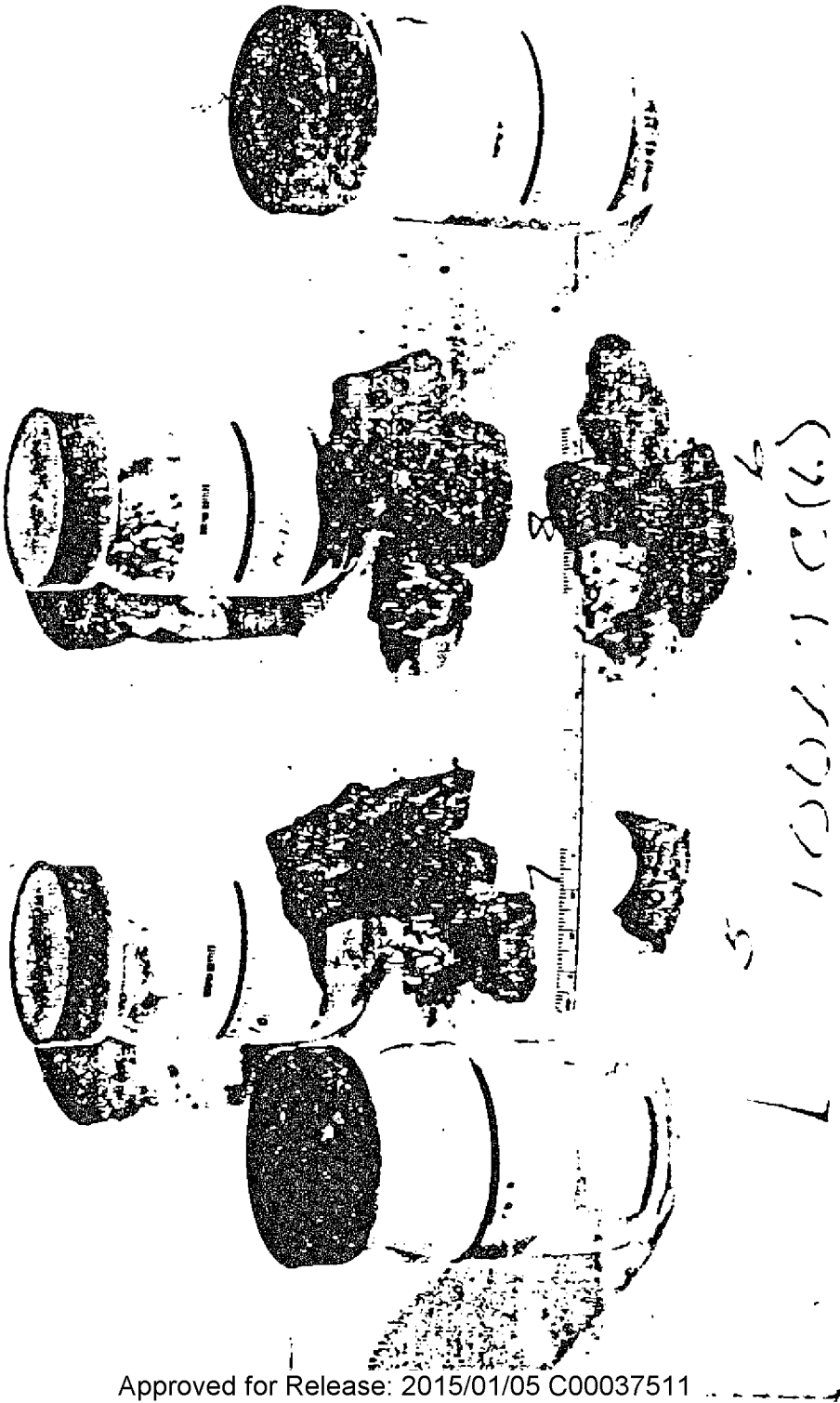

FIGURE 1

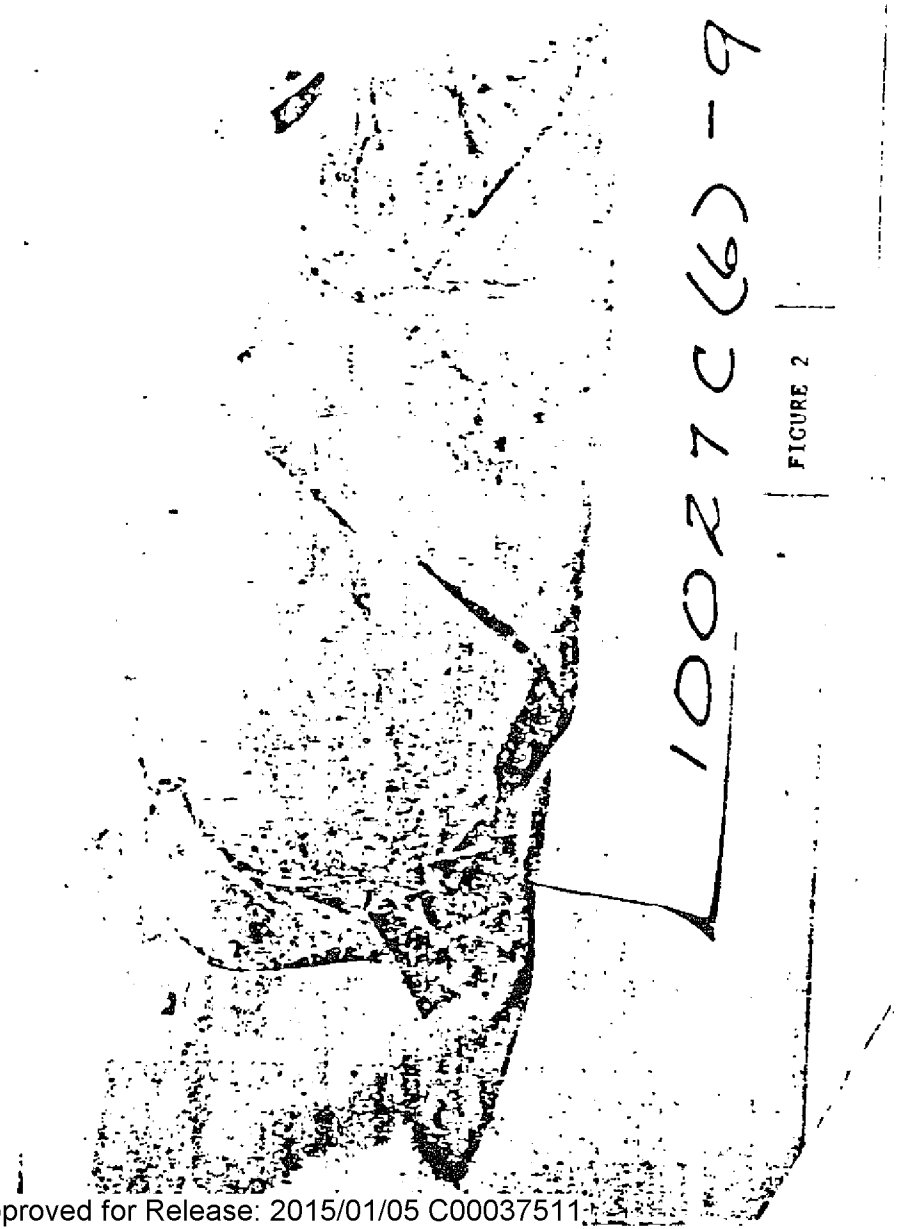

FIGURE 2

FIGURE 3