

(45)

Analytical Research Division  
Research Directorate

02 Nov 84

Analysis/Evaluation of Leaf

A shipment designated 100272222-10 was received by the Analytical Research Division, 23 February 1984, from FSTC. The sample was also identified with the number TH 840209-10DL. The shipment consisted of a single leaf having a 2 mm yellow spot and containerized in a plastic bag. No further information was available about the sample.

A vapor sample withdrawn from within the plastic bag containing the leaf was subjected to analysis by gas chromatography/mass spectrometry (GC/MS). A portion of the spot was leached with chloroform. Another portion was leached in 1:1 methanol:water. The solvent soluble materials were analyzed by GC/MS, ion chromatography (IC), thin layer chromatography (TLC), and infrared spectrometry (IR).

The GC/MS spectra of the vapors associated with the leaf identified the presence of dimethyl phenyl indine. The chloroform solubles did not give a definitive GC/MS spectra. IC was negative for all ions of interest. TLC separated a UV fluorescent compound,  $R_f=0.75$ , and a red colored component,  $R_f=0.80$ . These separations are similar to those given by reference vegetation samples. Derivatization with negative ion chemical ionization MS detection for trichothecenes was negative. IR spectra identified the presence of aliphatic hydrocarbons and traces of indefinite components.

Conclusions

No evidence of any known CW agents, agent degradation products, or trichothecenes was detected. The sample appears to be innocuous.

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ANALYTICAL RESEARCH DIVISION  
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21 September 1984

Analysis/Evaluation of Vegetation and Stone Samples

A shipment designated 10027Z(4) was received by the Analytical Research Division, 28 February 1984, from FSTC. The analysis of five samples, 10027Z(4)-1 (TH840209-1DL), 10027Z(4)-4 (TH840209-4DL), 10027Z(4)-5 (TH840209-5DL), 10027Z(4)-6 (TH840209-6DL) and 10027Z(4)-7 (TH840209-7DL), have been previously reported. Additional analysis have been completed on three samples. These include a small stone with one yellow spot two mm in diameter containerized in a plastic bag identified with TH840209-3DL and designated 10027Z(4)-3, two leaves with white and rust spots from one to nine mm in diameter, containerized in a plastic bag identified with TH840209-9DL and designated 10027Z(4)-9 (Figure 1) and one leaf with one yellow spot containerized in a plastic bag identified with TH840208-1LP and designated 10027Z(4)-12 (Figure 1). No further information was available on these samples.

A separate vapor sample withdrawn from within each plastic bag was subjected to analysis by gas chromatography/mass spectrometry (GC/MS). A portion of each sample was extracted with chloroform. Another portion of each was extracted 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).

10027Z(4)-3 (TH840209-3DL) stone

The GC/MS spectra of the vapor associated with the stone identified the presence of trimethyl phenylindene. The GC/MS analysis of the chloroform solubles gave no definitive spectra. IC separated no ions of interest. No detectable components were separated by TLC. Derivatization with negative ion chemical ionization MS detection was negative for trichothecenes. IR spectra identified a trace of aliphatic hydrocarbon and a silicate possibly soil.

10027Z(4)-9 (TH840209-9DL) two leaves

The GC/MS spectra of the vapor associated with the leaves identified the presence of 2,3 dihydro, 1,1,3-trimethyl-3-phenyl-1H indene

Chloroform solubles from each of the leaves gave no definitive GC/MS spectra. IC detected no ions of interest from either leaf. TLC detected two components from the small leaf with yellow spots, a UV fluorescent component, Rf 0.77-0.83, and another reacting with the p-anisaldehyde reagent to give a red spot at Rf 0.83-0.88. Only one component was detected from the large leaf with rust spots, a UV fluorescent material, Rf 0.73-0.79. Derivatization with negative ion chemical ionization MS detection was negative for trichothecenes in both leaves. IR spectra related to the small leaf identified the presence of aliphatic hydrocarbons and a band at 1610  $\text{cm}^{-1}$ , tentatively identified as

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a carboxylic acid salt. IR spectra related to the large leaf identified the presence of aliphatic hydrocarbons and a carbonyl band at  $1720\text{ cm}^{-1}$ .

100272(4)-12 (TH840208-1LP) one leaf

The GC/MS spectra of the vapor associated with the leaf identified the presence of a compound similar to hydroxytoluene, having a di-t-butyl group with one unsaturation. The GC/MS spectra of the chloroform solubles identified the presence of phthalates and six high boiling aliphatic hydrocarbons, bp  $250^{\circ} - 290^{\circ}$ ,  $> C_{30}$ . IC was negative for all ions of interest. TLC separated one component, which yielded a purple spot Rf 0.1-0.15 on reaction with p-anisaldehyde. Derivatization with negative ion chemical ionization MS detection for trichothecenes was negative. IR spectra identified the presence of aliphatic hydrocarbons, carbonyl and C-O.

Conclusions

No evidence of any known CW agents, agent degradation products or trichothecenes was detected. The significance of the detection of trimethyl-phenyl indene in two of the samples is not evident at this time. The samples appear to be innocuous.

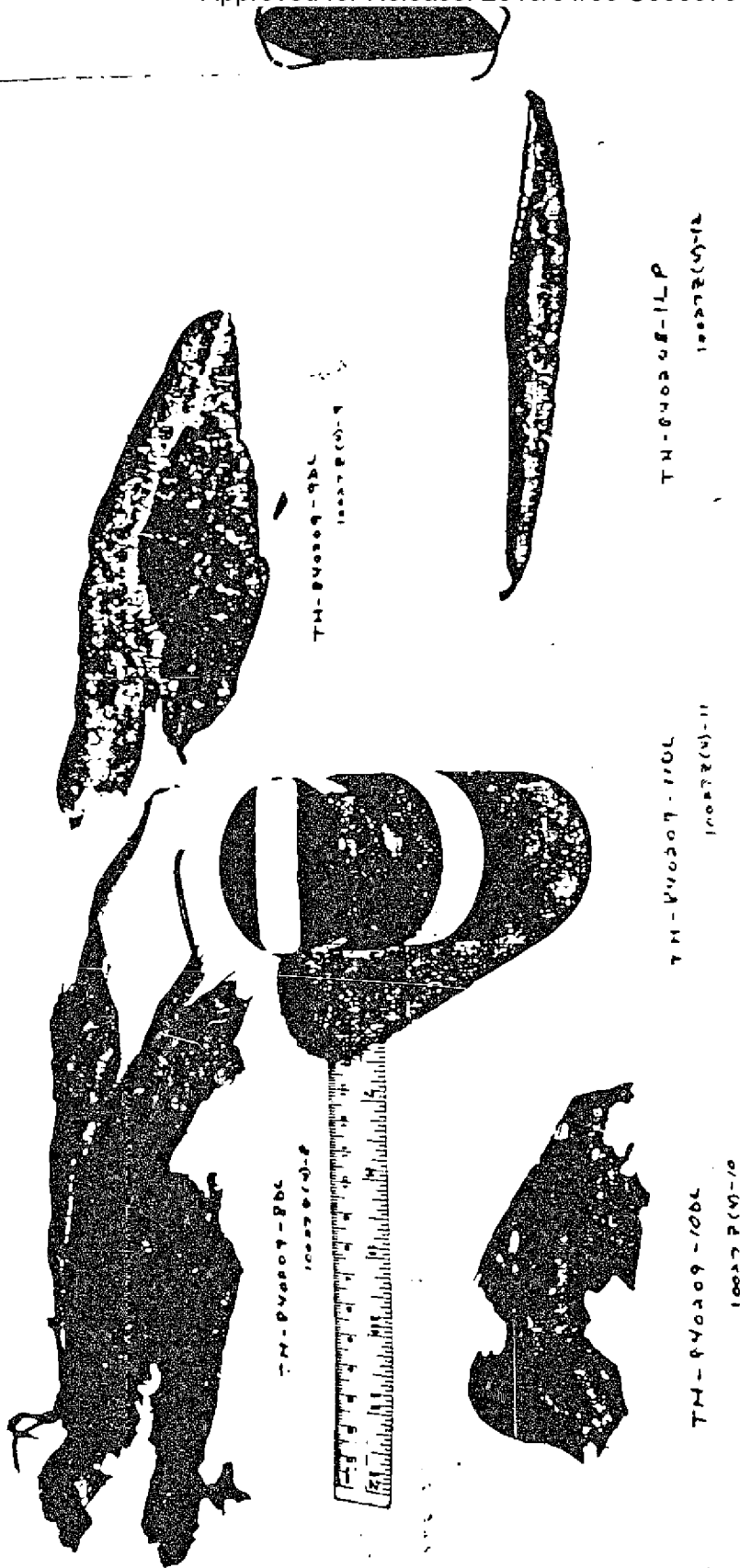


FIGURE 7

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Analysis/Evaluation of Yellow Powder

A shipment designated 10027Z(4), received by the Analytical Research Division, 23 February 1984, from FSTC, container # 12 subpackages. One container identified with TH840209-101 and designated 10027Z(4)-1. It consisted of a 4 oz wide mouth screw cap, flange gasketed glass jar (Figure 1) which reportedly contained a quantity of yellow spots scraped from rocks.



A vapor sample withdrawn from the container was subjected to analysis by gas chromatography/mass spectrometry (GC/MS). A portion of the powder was extracted with chloroform. Another portion of the powder was extracted 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).

The GC/MS analysis of the vapors associated with the powder gave no definitive spectra. The GC/MS spectra of the chloroform solubles showed only a possible trace of chloro compounds. IC separated no compounds of interest. No detectable components were separated by TLC. Derivatization with negative ion chemical ionization MS detection for trichothecenes was negative. IR spectra identified the presence of water, traces of hydrocarbons and carbonyl, and silicates, typical of soil.

Conclusion:

No evidence of any known CW agent, agent degradation product or trichothecene was detected. As received by the Analytical Research Division, the sample appears to be innocuous.

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19 September 1984

Analysis/Evaluation of Leaves

A shipment designated 100272(4), received by the Analytical Research Division, 23 February 1984, from FSTC, contained 12 subpackages. One container identified with TH840209-4DL was designated 100272(4)-4. It reportedly consisted of leaves (Figure 1) with yellow spots from 2 to 5mm diameter  b3

A vapor sample withdrawn  was subjected to analysis by gas chromatography/mass spectrometry (GC/MS). A portion of the leaves was leached in chloroform. Another portion of the leaves was leached in 1:1 methanol:water. The solvent soluble materials were analyzed by GC/MS, ion chromatography (IC), thin layer chromatography (TLC) and infrared spectrometry (IR). b3

The GC/MS spectra of the vapors associated with the leaves identified the presence of a series of fatty acids including hexanoic, heptanoic and isoocetyl acids, and an unidentified compound of molecular weight 208 having a base peak 123. The GC/MS spectra of the chloroform solubles showed only a possible trace of chloro compounds. IC separated no ions of interest. No detectable components were separated by TLC. Derivatization with negative ion chemical ionization MS detection for trichothecenes was negative. IR spectra identified the presence of water and aliphatic hydrocarbons.

Conclusion:

No evidence of any known CW agents, agent degradation products or trichothecenes was detected. The detection of volatilized fatty acids in this sample, 100272(4)-5 and 100272(4)-6, but not in the reference vegetation would indicate they are not naturally occurring vegetation components. They are components of the M-4 thickener for napalm, but the aluminum salts used in the M-4 were not detected.

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19 September 1984

Analysis/Evaluation of Leaves

A shipment designated 10027Z(4), received by the Analytical Research Division, 23 February 1984, from FSTC, contained 12 subpackages. One container, identified with TH84029-5DL was designated 10027Z(4)-5. It reportedly consisted of leaves (figure 1) with white spots from 2 to 13mm in diameter. 

b3

A vapor sample withdrawn  was subjected to analysis by gas chromatography/mass spectrometry (GC/MS). A portion of the leaves was leached in chloroform. Another portion was leached in 1:1 methanol:water. The solvent soluble materials were analyzed by GC/MS, ion chromatography (IC), thin layer chromatography (TLC), and infrared spectrometry (IR). 

b3

The GC/MS spectra of the vapor associated with the leaves identified the presence of the same fatty acids, hexanoic, heptanoic and iso-octanoic detected in the vapors of the 10027Z(4)-4 (TH830209-4DL) sample. The GC/MS spectra of the chloroform solubles identified only a possible trace of chloro compounds. IC separated no ions of interest. No detectable components were separated by TLC. Derivatization with negative ion chemical ionization MS detection for trichothecenes was negative. IR spectra identified the presence of water, methyl groups, a trace carbonyl and silicates typical of soils.

Conclusion:

No evidence of any known CW agent, agent degradation products or trichothecene was detected. The detection of volatilized fatty acids in this sample, 10027Z(4)-4 and 10027Z(4)-6, but not in the reference vegetation would indicate they are not naturally occurring vegetation components. They are components of the M-4 thickener for napalm, but the aluminum salts used in the M-4 were not detected.

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**ANALYTICAL RESEARCH DIVISION  
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19 September 1984

Analysis/Evaluation of Leaves

A shipment designated 10027Z(4), received by the Analytical Research Division, 23 February 1984, from PSIC, contained 12 subpackages. One container identified with TH840209-7DL was designated 10027Z(4)-7. It reportedly consisted of reference leaves [redacted]

b3

The sample was subjected to the same series of analyses performed on suspect samples. A vapor sample withdrawn [redacted] was subjected to analysis by gas chromatography/mass spectrometry (GC/MS). A portion of the leaves was leached in chloroform. Another portion was leached in 1:1 methanol:water. The solvent soluble materials were analyzed by GC/MS, ion chromatography (IC), thin layer chromatography (TLC) and infrared spectrometry (IR).

b3<sup>7</sup>

The GC/MS analysis of the vapors associated with the powder identified the presence of dimethyl phenylindene. The GC/MS analysis of the chloroform solubles gave no definitive spectra. IC separated no ions of interest. TLC separated two components Rf 0.15 and 0.40 which reacted with the p-anisaldehyde detection reagent to give a purple color. Derivatization with negative ion chemical ionization MS detection did not detect any trichothecenes. The MS spectra was identical to that of 10027Z(4)-6, indicating a large quantity of an unidentified compound which underwent derivatization. The IR spectra identified the presence of water and fragmentary alkyl hydrocarbons.

Conclusion:

No evidence of any known CW agent, agent degradation product or trichothecene was detected. Submitted as a reference sample, the components separated by thin layer chromatography and the component which underwent derivatization afford excellent base line data for evaluation of suspect samples.

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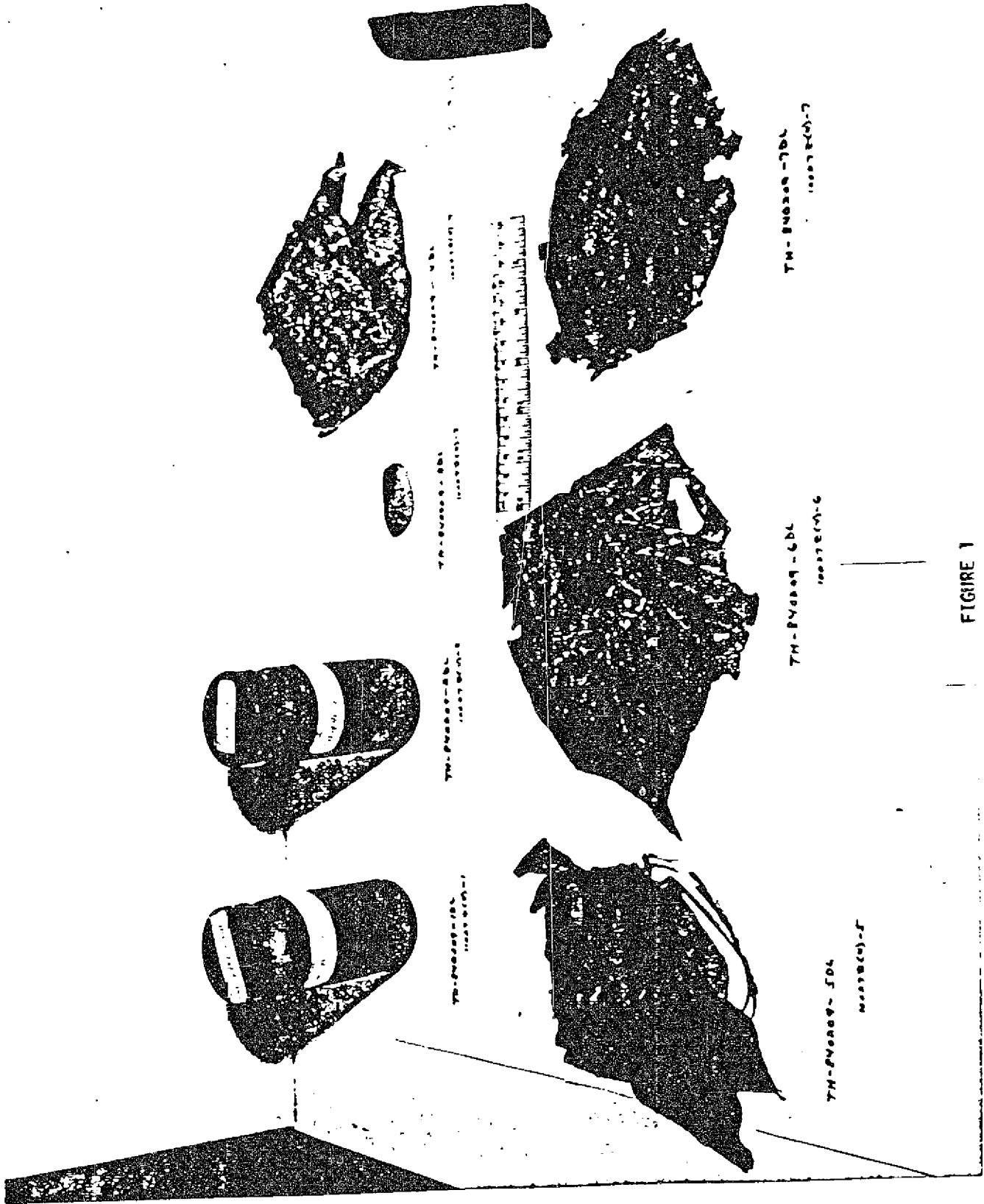


FIGURE 1