

ANALYTICAL BRANCH
RESEARCH DIVISION

23 July 1984

Analysis/Evaluation of Tan Powder

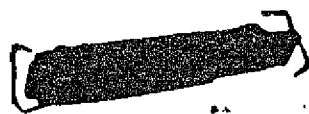
A multiple sample shipment designated 10027A(5) was received by the Analytical Branch from FSTC 3 March 1984. One sample, consisting of a tan powder carrying the identification TH 840224-1DL, was designated 10027A(5)-18. The powder was containerized in a glass screw top jar overpacked in styrofoam.

A vapor sample withdrawn from within the glass jar was subjected to analysis by gas chromatography/mass spectrometry (GC/MS). Based on this analysis, a thermal profile of the neat powder was determined by differential thermal analysis (DTA) and thermal gravimetric analysis (TGA). Methanol soluble materials were analyzed by ultraviolet spectroscopy (UV). Solvent insolubles were analyzed by infrared spectrometry (IR).


The GC/MS spectra of the vapors associated with the powder identified the presence of o-chlorobenzaldehyde, CS, and chlorobenzylcyanide. The DTA and TGA determined the melting point as 94°C and the boiling point as 311°C, with 1% water and 9% nonvolatiles. The melting and boiling points agree with those of high purity CS. UV determined the CS to be 98% pure. IR spectra identified the solvent insoluble materials to be a crystalline silicate, identical with those found in the previously reported CS samples TH 831205-1XX and TH 831205-2XX (10027V(4)-2,3). The silicate is not the amorphous silica gel or Cab-O-Sil normally used in CS formulations.

Conclusion:

The sample consists of a CS formulation containing 90% CS of 98% purity, 1% water, 9% crystalline silicon dioxide.




(254)


ANALYTICAL RESEARCH DIVISION
RESEARCH DIRECTORATE

27 September 1984

Analysis/Evaluation of Yellow Powder

A multiple sample shipment, designated 10027A(5), was received by the Analytical Research Division, 3 March 1984, from FSTC. One sample, consisting of a yellow powder, carrying the identification TH840220-1EB, was designated 10027A(5)-15. The sample reportedly was scraped from leaves and stems  b3

A vapor sample withdrawn from within the paper enclosure was analyzed by gas chromatography/mass spectrometry (GC/MS).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 solubles 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 powder identified the presence of acetophenone, 2,6-di-t-butyl-4-formylphenol, and an unidentified compound M/Z 57, 82, 97, 123. The GC/MS spectra of the chloroform solubles detected two high boiling aliphatic hydrocarbons, <C30 and phthalates. IC detected no ions of interest. TLC separated one component, Rf 0.4-0.45 which reacted as an alkylating agent with p-anisaldehyde to give a purple color. Derivatization with negative ion chemical ionization MS detection for trichothecenes was negative. IR spectra identified the presence of water and aliphatic hydrocarbons typical of a vegetation source.

Conclusion

No evidence of any known CW agent, agent degradation product or trichothecene was detected. The sample appears to be innocuous. The detection of an alkylating agent is similar to that detected on several other suspect samples and suggests that when the work load permits, further studies should be performed to identify this component.

Classified by: CIA


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Ex 104


ANALYTICAL RESEARCH DIVISION
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19 September 1984

Analysis/Evaluation of Yellow Powder

A shipment designated 1G027A(5), received by the Analytical Research Division, 3 March 1984, from FSTC, contained 19 subpackages. One container identified with TH640223-4DL was designated 1G027A(5)-3. It consisted of a few yellow spots, possibly 5mg, containerized in a black painted 4 oz wide mouth black jar. Information about the sample indicated the yellow spots had been scraped from rocks.

A vapor sample withdrawn from within the jar was subjected to analysis by gas chromatography/mass spectrometry (GC/MS). A portion of the powder was extracted with chloroform. Another portion 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 spectra of the vapor associated with the powder identified the presence of benzene, toluene, xylene, benzaldehyde, substituted isobutylene, acetophenone, methyl benzoate, nonanal and methyl naphthalene. The GC/MS of the chloroform solubles detected two high boiling aliphatic hydrocarbons, approximately C₃₀. IC separated no ions of interest. No detectable components were separated by TLC. Derivatization with negative ion chemical ion MS detection for trichothecenes was negative. IR spectra detected only a trace of hydrocarbons.

Conclusion:

No evidence of any known CW agents, agent degradation products or trichothecenes was detected. The numerous solvent and aromatic structures detected could be indicative of a solvent/delivery system.

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Analytical Research Division
Research Directorate

02 Nov 84

Analysis/Evaluation of Leaf Samples

Attached are the final reports on the analysis/evaluation of several samples included in the shipment 10027AAAAA, received by the Analytical Research Division 3 March 1984. Included in these samples are several leaf samples: 10027AAAAA-9, 10027AAAAA-11, 10027AAAAA-16 and 10027AAAAA-17. It should be noted that the vapor analysis of samples 10027AAAAA-9,-11,-16 each detected components other than those normally found associated with vegetation samples. These included sulfur dioxide, dimethyl disulfide and dimethyl sulfone in the 10027AAAAA-9 analyses, sulfur dioxide, cyanobenzene and methyl benzoate in 10027AAAAA-11, and cyanobenzene and isooctane in 10027AAAAA-16. Analysis of 10027AAAAA-17 identified only normal vegetation components. Since the unusual components were detected only in the vapors, further chemical analyses can not be performed. These components are not normally found in the packaging materials used. At this time the source of these components can not be determined. They could be from exposure to a fungicide, herbicide, or an unknown source. A literature and intelligence study would be appropriate to determine if these compounds could be components of a viable volatile agent.

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Declassify: OADR

(25c)

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Analytical Research Division
Research Directorate

30 Oct 84

Analysis/Evaluation of Leaves

A shipment designated 10027AAAA-9A was received by the Analytical Research Division from FSTC. The shipment consisted of three groups of leaves (Figure 1). Each group of leaves was contained in a plastic bag. There was no information available concerning the samples.

Vapor samples withdrawn from within the plastic bags containing the leaves were subjected to analysis by gas chromatography/mass spectrometry (GC/MS). Several areas on each type of leaves were leached in chloroform, others were leached in a 1:1 methanol: water mixture. The solvent soluble materials were analyzed by GC/MS, ion chromatography (IC), thin layer chromatography (TLC), and infrared spectrometry (IR).

10027AAAA-9A

The GC/MS spectra of the vapor associated with the leaves identified the presence of dimethyldisulfide as the major component, with sulfur dioxide and benzaldehyde as minor components. The GC/MS spectra of the chloroform solubles identified the presence of copaene and phthalates. IC was negative for all ions of interest. TLC separated one component, a pink spot $R_f = .83-.91$, which became dark pink on reaction with p-anisaldehyde. A similar component has been detected on previous reference leaf samples. Derivatization with negative ion chemical ionization MS detection was negative for trichothecenes. IR spectra identified the presence of water, aliphatic hydrocarbons, a trace of carbonyl and possibly soil.

10027AAAA-9B

The GC/MS spectra of the vapor associated with these leaves identified the presence of a complex mixture, including sulfur dioxide, dimethyl disulfide, dimethyl pyridine, terpene, aliphatic hydrocarbons, limonene, nonane, phenoxy compounds, dodecane, camphor, 4-t-butyl-o-cresol, C₁₅H₃₂ and C₁₅H₃₀ isomers and sesquiterpene. The GC/MS spectra of the chloroform solubles identified the presence of phthalates only. No components were separated by TLC. IC was negative for all detectable ions. Derivatization with negative ion chemical ionization MS detection was negative for trichothecenes. IR spectra identified the presence of water, aliphatic hydrocarbons and a carbonyl at 1735 cm⁻¹, possibly present as an acid.

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Encl 10E

10027AAAAA-9C

The GC/MS spectra of the vapors in equilibrium with these leaves identified the presence of sulfur dioxide, benzene, toluene, xylene, dimethyl sulfone, pinene, acetophenone, benzonitrile, terpenoid (C₁₄H₁₀O), naphthalene, cyclic paraffins, siloxane, alpha muurolene (C₁₅H₂₄), and calomenene with caryphyllene as the major component. The GC/MS spectra of the chloroform solubles identified only the presence of phthalates. IC was negative for all ions of interest. No components were separated by TLC. Derivatization with negative ion chemical ionization MS detection was negative for trichothecenes. IR spectra identified the presence of aliphatic hydrocarbons, a carbonyl at 1735 cm⁻¹, and a possible second carbonyl present as a phthalate, an ester or cellulose.

Conclusions

No evidence of any known CW agents, agent degradation products, or trichothecenes was detected. The phthalates detected were probably inherent to the plastics bags used in packaging. The various terpenes and aromatic solvents appear to be associated with foliage from the area. The presence of sulfur dioxide, dimethyl disulfide and dimethyl pyridine in the vapors associated with the leaves suggests the possibility of contamination with a volatile substance. Since no information was available as to whether the leaves were collected from a suspected chemical attack area, the significance of these findings can not be evaluated.

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Analytical Research Division
Research Directorate

01 Nov 84

Analysis/Evaluation of Liquid

A shipment designated 10027AAAAA-10 was received by the Analytical Research Division, 3 March 1984, from FSTC. The sample was also identified with the number TH 840201-1XX. The shipment consisted of approximately 1.5 ml of dark brown liquid containerized in a 5 ml glass vial stoppered with a rubber septum (Figure 1). Information concerning the sample indicated it reportedly was a poison, possibly to be used to poison water supplies.

A vapor sample withdrawn from within the vial containing the liquid was subjected to analysis by gas chromatography/mass spectrometry (GC/MS). A portion of the liquid was extracted with chloroform. The solvent soluble materials were analyzed by GC/MS, thin layer chromatography (TLC), and infrared spectrometry (IR). The neat liquid was analyzed by GC/MS, ion chromatography (IC), TLC and IR.

The vapor associated with the liquid did not give a definitive GC/MS spectra. The chloroform soluble materials also did not give a definitive GC/MS spectra. The neat liquid did not yield a GC/MS spectra. IC was negative for all ions of interest. No components were separated by TLC. Derivatization with negative ion chemical ionization MS detection for trichothecenes was negative. IR spectra identified the presence of traces of aliphatic hydrocarbons, and the possible presence of cellulose, carbonyls and alcohols.

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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Excl 107

(254)


Analytical Research Division
Research Directorate

01 Nov 84

Analysis/Evaluation of Solid

A shipment designated 11027AAAAA-11 was received by the Analytical Research Division, 3 March 1984, from FSTC. The sample was also identified with the number TH 840213-1AC. The shipment consisted of two pieces of off-white solid containerized in a plastic bag (Figure 1). No information was available about the sample.

A vapor sample withdrawn from within the plastic bag containing the solids was subjected to analysis by gas chromatography/mass spectrometry (GC/MS). A portion of the solids was extracted with chloroform. Another portion was extracted with a mixture of 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 chloroform solubles identified the presence of aliphatic hydrocarbons, from C23 to C33, with C27 having the highest concentration. The spectra are nearly identical to those of beeswax previously studied.

The GC/MS spectra of the vapor associated with the solids identified cyanobenzene as the major component, with sulfur dioxide, benzene, benzaldehyde and methyl benzoate as minor components. The GC/MS spectra of the chloroform solubles identified the presence of aliphatic hydrocarbons, from C23 to C33, with C27 having the highest concentration. The spectra is nearly identical to those of beeswax run in previous studies. IC was negative for all ions of interest. TLC separated one component, $R_f = .87-.92$ which yielded a dark spot on reaction with p-anisaldehyde. This was similar to the separation and reaction yielded by beeswax. Derivatization with negative ion chemical ionization MS detection was negative for trichothecenes. IR spectra identified the material as beeswax.

Conclusions

No evidence of any known CW agents, agent degradation products, or trichothecenes was detected. The sample was identified as beeswax.

Classified by: CIA



Declassify: OADR

Encl 10K

(254)

Analytical Research Division
Research Directorate

01 Nov 84

Analysis/Evaluation of Leaves

A shipment designated 16027AAAAA-16 was received by the Analytical Research Division, 3 March 1984, from FSTC. The sample was also identified with the number TH 840223-1MS. The shipment consisted of several mango blossoms and leaves, each having a 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 leaves was subjected to analysis by gas chromatography/mass spectrometry (GC/MS). A portion of the leaves 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 leaves identified thujenene as the major component, with benzene, toluene, xylene, cyanobenzene, alpha fenchene, isooctane and three terpenoids as minor components. The chloroform solubles did not give a definitive GC/MS spectra. IC was negative for all ions of interest. No components were separated by TLC. Derivatization with negative ion chemical ionization MS detection for trichothecenes was negative. IR spectra identified the presence of aliphatic hydrocarbons, C=O and C-O.

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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Declassify: OADR

Encl 10i


Analytical Research Division
Research Directorate

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01 Nov 84

Analysis/Evaluation of Leaves

A shipment designated 16027AAAAA-17 was received by the Analytical Research Division, 3 March 1984, from FSTC. The sample was also identified with the number TH 840223-1DL. The shipment consisted of several banana leaves each having a 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 leaves was subjected to analysis by gas chromatography/mass spectrometry (GC/MS). A portion of the leaves 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 leaves identified the presence of toluene, xylene and benzaldehyde. The GC/MS spectra of the chloroform solubles identified only the presence of a trace of terpenes. IC was negative for all ions of interest. No components were separated by TLC. Derivatization with negative ion chemical ionization MS detection for trichothecenes was negative. IR spectra identified the presence of aliphatic hydrocarbons, C=O and water.

Conclusions

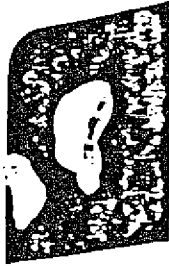
No evidence of any known CW agents, agent degradation products, or trichothecenes was detected. The sample appears to be innocuous.

Classified by: CIA


Declassify: OADR



-9C



TH840213-3AC
10027 ACE)-13



-9B

TH840213-2AC
10027A(5)-12



TH840212-1AC
10027 ACE)-11



-9A

LEAVES FROM DR. TOWNSEND
10027 ACE)-9



TH-840201-1XX
10027 A(5)-10

FIGURE 1

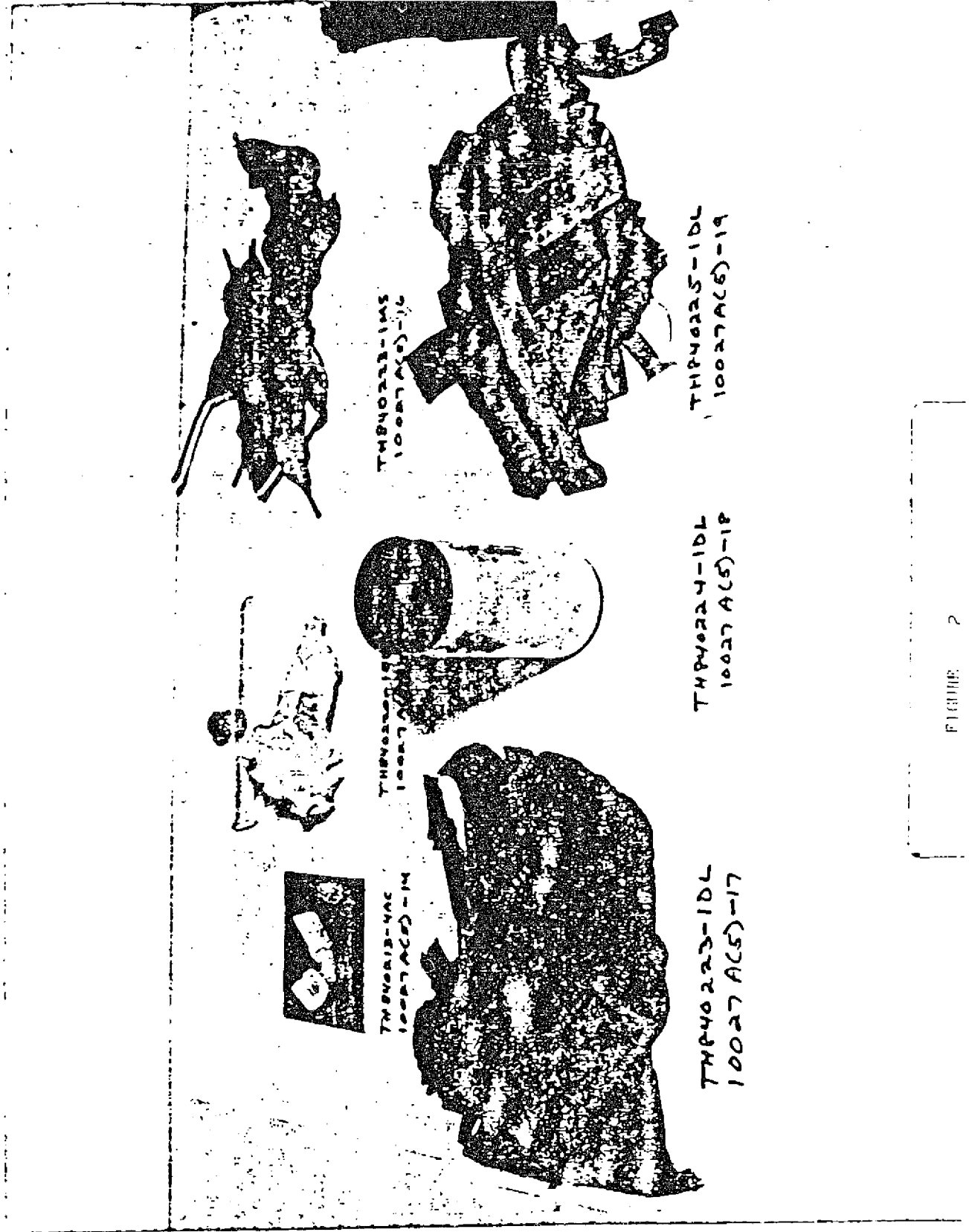


FIGURE 2