Analytical Research Division Research Directorate

26 September 1984

Analysis/Evaluation of Powder and Leaf Samples

A shipment designated 10027K(4), carrying the Registry Number C-290-83, received by the Analytical Research Division, 15 November 1983, from FSIC, contained six sub-samples. The shipment consisted of five powder samples, each wrapped in notepaper and placed in a wide mouth screw top jar, and leaves with yellow spots placed in a second wide mouth screw top jar. No information was available on any of the samples. The first powder, designated 10027K(4)-1, consisted of approximately 10 mg of flat yellow spots (figure 1). The second powder, designated 10027K(4)-2, consisted of approximately 10 mg of a yellow powder (figure 1). The third powder, designated 10027K(4)-3, consisted of approximately 10 mg of a yellow powder (figure 1). The fourth powder consisted of approximately 2 mg of brown powder, resembling soil (figure 1). The fifth powder, consisting of approximately 5 mg of brown powder will be reported at a later date. The sixth sample consisted of eight leaves, each having one yellow spot, 5 mm in diameter (figure 2).

Vapor samples withdrawn from within each sample enclosure were subjected to analysis by gas chromatography/mass spectrometry (GC/MS). A portion of each sample 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).

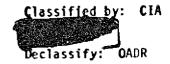
The GC/MS spectra of the vapor associated with the packets of powder identified the presence of camphor and menthol as the primary components and toluene, xylene, styrene, acetophenone, isopropyl benzene, t-butyl benzene, propyl toluene or butyl benzene and approximately 6 isomers of $C_{10}H_{14}$, β -R as minor components.

10027K(4)-1, flat yellow spots

6C/MS spectra of the chloroform solubles identified only diethylphthalate. IC detected no ions of interest. No separation of components was detected by TLC. Derivatization with negative ion chemical ionization MS detection was negative for trichothecenes. IR spectra detected the presence of aliphatic hydrocarbons and carbonyl bands at 1735 and 1710cm⁻¹.

10027K(4)-2, yellow powder

Analysis of the chloroform solubles by GC/MS gave no definitive spectra. IC detected no ions of interest. No separation of components was detected by TLC. Derivatization with negative ion chemical ionization MS detection was negative for trichothecenes. IR spectra identified heavy concentrations of aliphatic hydrocarbons, one carbonyl possibly present as an ester and a possible phthalate.



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10027K(4)-3, yellow powder

GC/MS spectra of the chloroform solubles identified the presence of C₂₅ and C₃₀ aliphatic hydrocarbons. IC was negative for all ions of interest. No separation of components was detected by TLC. Derivatization with negative ion chemical ionization MS detection was negative for trichothecenes. IR spectra identified the presence of aliphatic hydrocarbons and a carbonyl at 1734 cm⁻¹, possibly present as an ester.

10027K(4)-4, brown powder resembling soil

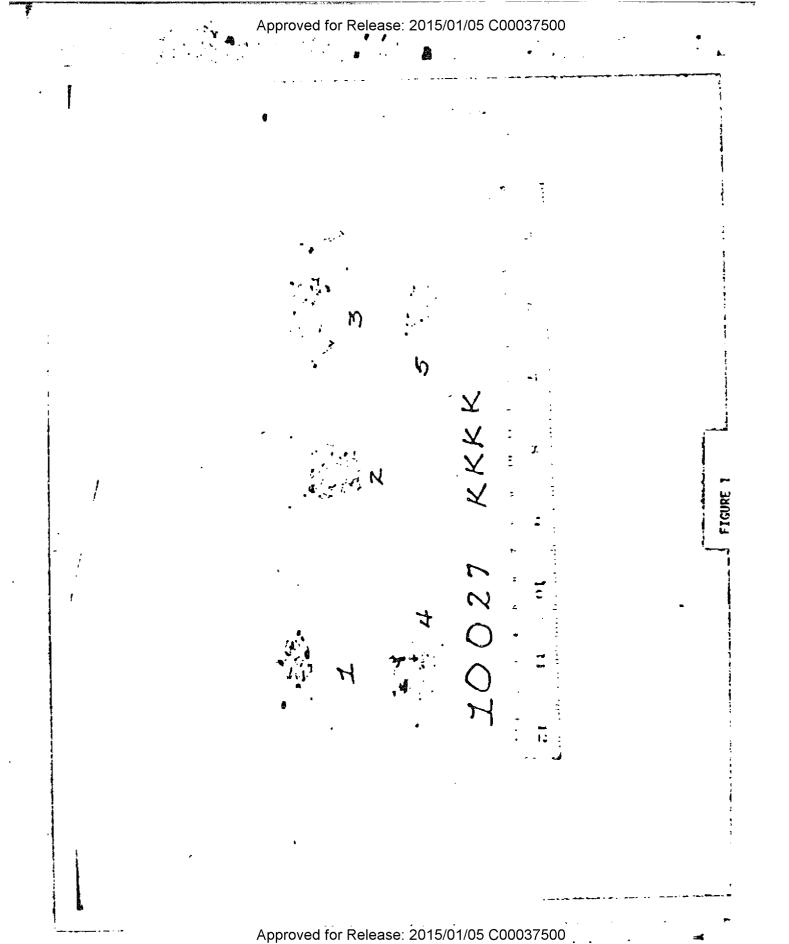
Analysis of the chloroform solubles by GC/MS gave no definitive spectra. IC separated no ions of interest. No detectable components were separated by TLC. Derivatization with negative ion chemical ionization MS detection was negative for trichothecenes. IR spectra identified the presence of aliphatic hydrocarbons, a carbonyl at 1733, and possible cellulose or soil.

10027K(4)-6, leaves

The GC/MS spectra of the vapor associated with theleaves identified the presence of toluene, ethyl benzene and benzaldéhyde. IC was negative for all 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 the presence of aliphatic hydrocarbons and possibly an inorganic nitrate.

Conclusion

No evidence of any known CW agent, agent degradation product of trichothecene was detected. Each of the components detected have either previously been detected in reference samples or are normally associated with vegetation sources. The samples appear to be innocuous.



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FIGURE 2

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