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ANALYTICAL RESEARCH DIVISION RESEARCH DIRECTORATE

18 September 1984

Analysis/Evaluation of Yellow Powder

A shipment designated 10027A(4), also identified with TH830513-38DL, was received by the Analytical Research Division, 3 June 1983, from FSTC. Information concerning the sample indicated it could be the residue from a chemical attack. The sample consisted of approximately 50mg of a yellow powder containerized in a glass vial fitted with a rubber septum closure (Figure 1).

A vapor sample withdrawn from within the glass tube was analyzed by gas chromatography/mass spectrometry (GC/HS). A portion of the powder was extracted with chloroform. Another portion was extracted with 1:1 methanol:water. The solvent extracts were subjected to analysis by GC/HS, ion chromatography (IC), thin layer chromatography (TLC) and infrared spectrometry (IR). In addition, a portion of the neat powder was examined by scanning electron microscopy (SEM).

The GC/HS spectra of the vapor associated with the powder identified the presence of the solvents toluene and xylene. The GC/HS spectra of the chloroform solubles detected a series of aliphatic hydrocarbons, C23, C25, C27, C29, C31 with the C25and C27 having the highest concentration. This is similar to the spectra of beeswax. In addition, pentanol and cyclopropyl methylketone were detected. Ion chromatography detected 9 ppm fluoride and possible traces of cyanide. Thin layer chromatography separated two components which reacted with the detection reagent for alkylating agents to yield purple spots at Rf 0.28 to 0.36 and 0.80 to 0.84. Known Deeswax gave a similar reaction at Rf .80 to .84. A reference rice sample gave a similar reaction at approximately the same Rf as the 0.28 to 0.36 component. Derivatization with negative ion chemical ionization, MS detection was negative for trichothecenes. Gas chromatography gave a separation of components with retention times identical to those of beeswax. IR spectra identified the presence of aliphatic hydrocarbons, two carbonyl bands at 1735 and 1710 cm⁻¹, possible aromatics and esters. The SEM examination (Figure 2) showed the powder consisted of tightly packed clumps of pollen. Five different pollen species were detected. Each pollen species had been detected in one pr more previous samples.

Conclusion.

No evidence of any known CW agent, agent degradation product or trichothecene was detected. The various analyses suggest the major components are pollen and beeswax. Although this combination could be indicative of bee feces, the detection of (1) trace levels of fluoride, (2) an unidentified alkylating agent not a component of beeswax or bee feces, and (3) five species of pollen rather than one or two species normally found in bee feces suggest another, at present, unidentified source.



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