

ANALYTICAL RESEARCH DIVISION  
RESEARCH DIRECTORATE

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20 September 1984

Analysis/Evaluation of Rice and Leaf Control Samples

A shipment designated 10027R(4) was received by the Analytical Research Division, 15 December 1983, from FSTC. The three samples included in the shipment were identified as reference materials. Each was containerized in a separate plastic bag. One sample identified with LAB310XX-5RP, designated 10027R(4)-1, consisted of approximately one cup of glutinous rice grown and milled in 1983 (Figure 1). The second sample, identified with LAB310XX-6RP, designated 10027R(4)-2, consisted of approximately one cup of non-glutinous rice grown and milled in 1983 (Figure 1). The third sample, identified with LAB31113-4WT, designated 10027R(4)-3, consisted of a yellow spotted fig leaf (Figure 1).

A separate vapor sample withdrawn from within each enclosure was subjected to analysis by gas chromatography/mass spectrometry (GC/MS). A portion of each sample 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).

10027R(4)-1 (LAB310XX-5RP) Glutinous rice

The GC/MS spectra of the vapor associated with the rice identified the presence of benzene, toluene, xylene, branched aliphatic hydrocarbons (bp 110-160°C), o-cresol, dimethylphenol, methyl benzoate and possibly benzoic acid hydride. The GC/MS spectra of the chloroform solubles was similar to that of rubber. IC separated no ions of interest. Two components were separated by TLC. One, which remained at the origin, fluoresced under UV light. The other was identical to a component detected in the 10027L(4)-1 reference rice samples, Rf 0.37-0.46 giving a purple reaction product with p-anisaldehyde. Derivatization with negative ion chemical ionization MS detection gave negative results for trichothecenes. IR spectra identified the presence of aliphatic hydrocarbons and two carbonyl bands at 1740 and 1706  $\text{cm}^{-1}$ .

10017R(4)-2 (LAB310XX-6RP) non-glutinous rice

The analysis data were similar to those of the glutinous rice, with a few exceptions. The GC/MS spectra of the vapor associated with the rice identified the presence of xylene, branched aliphatic hydrocarbons (bp 110-160°C) and methyl benzoate. The chloroform solubles did not give definitive GC/MS spectra. The IC, TLC and derivatization/MS analysis results were identical with those of the glutinous rice. IR spectra identified the presence of high concentrations of aliphatic hydrocarbons, lesser quantities of aromatic olefins, 2 carbonyls with bands at 1742 and 1711  $\text{cm}^{-1}$  could be present as an acid ester.

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10027R(4)-3 (LA831113-4WT) fig leaf pieces

The GC/MS spectra of the vapor associated with the leaf pieces identified the presence of xylene, benzaldehyde, phenol, benzonitrile acetophenone and methyl benzoate. The chloroform solubles did not give definitive GC/MS spectra. IC separated no ions of interest. TLC separated two components at Rf 0 and 0.77-0.83 which fluoresced under UV light. No reaction with p-anisaldehyde was detected. Derivatization with negative ion chemical ionization MS detection was negative for trichothecenes. IR spectra identified the presence of water, aliphatic hydrocarbons, with two weak carbonyl bands at 1733 and 1715  $\text{cm}^{-1}$ .

Conclusion:

No evidence of any known CW agent, agent degradation product or trichothecene was detected. The similarity of the analysis data from the two rice samples was as expected. The slight differences indicate definitive analyses for the two different types of rice. Each of the samples appeared to be innocuous and gave excellent, reproducible base line analyses for reference data.

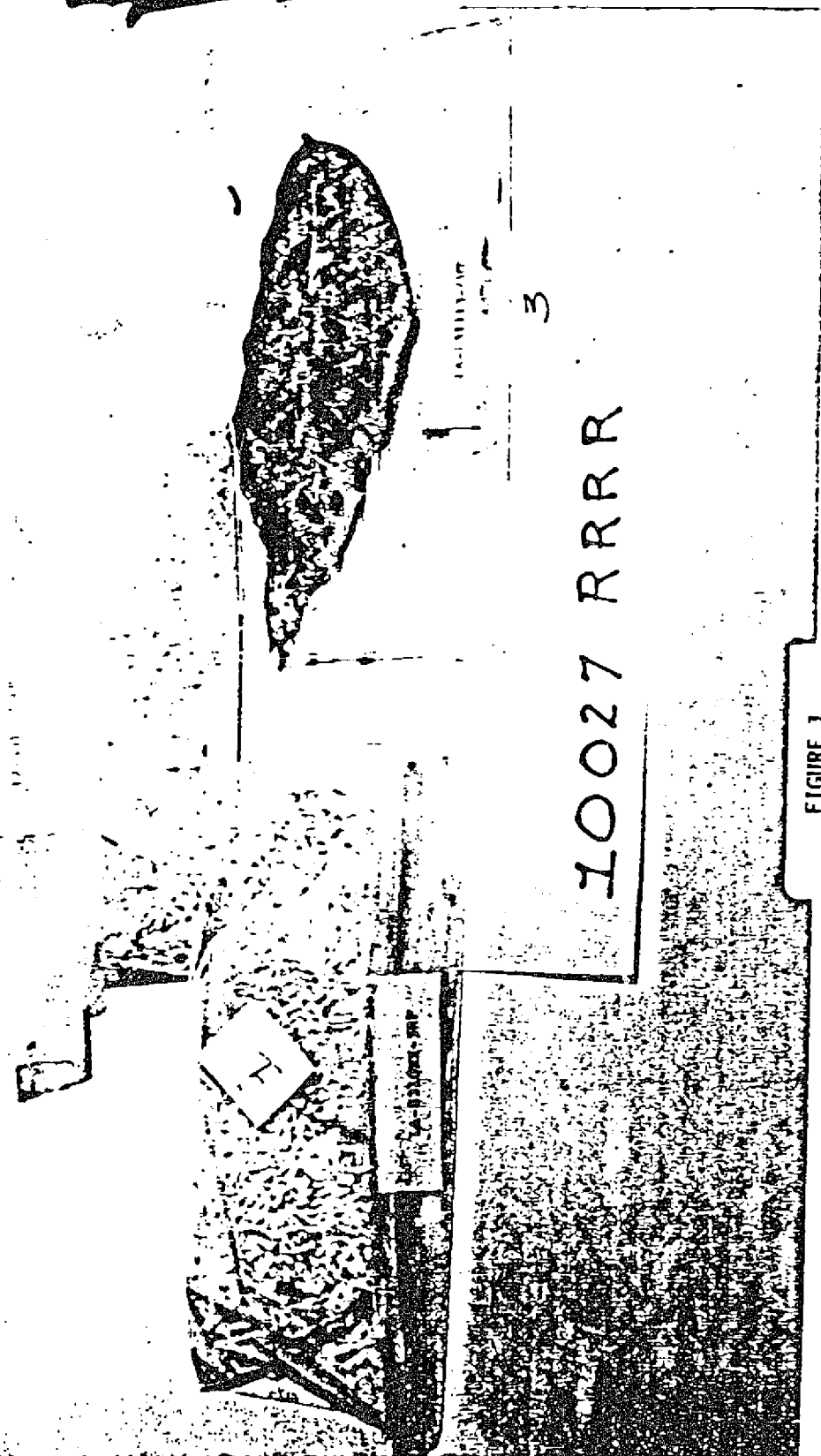


FIGURE 7

  
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29 Oct 84

Analysis/Evaluation of Dark Liquid

A shipment designated 10027QQQ was received by the Analytical Research Division, 14 December 1983, from FSTC. The sample was also identified with the number TH 831117-2XX. The shipment consisted of approximately 4 oz. of a dark amber liquid containerized in a screw top glass jar. There was no information available concerning the sample.

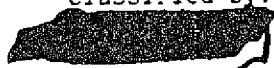
A vapor sample withdrawn from within the glass bottle was subjected to analysis by gas chromatography/mass spectrometry (GC/MS). The neat liquid was analyzed by GC-MS, thin layer chromatography (TLC), and infrared spectrometry (IR).

The GC/MS spectra of the vapor associated with the liquid identified the presence of 2 isomers of cresol as the major components, and toluene, xylene, phenol, two isomers of dimethyl phenol naphthalene and methyl naphthalene as minor components. The GC/MS spectra of the neat liquid identified the presence of a complex mixture. The major component was phenol. Minor components identified included toluene, xylenes, thiophenol, two cresols, a series of substituted phenols, thymol and dimethylbenzaldehyde. TLC yielded a smear from which components could not be separated. IR spectra identified both aromatic and aliphatic hydrocarbons, carbonyl and OH bonds typical of phenol, and p-mono or disubstitution.

Conclusions

No evidence of any known CW agents, agent degradation products, or trichothecenes was detected. The liquid appears to be an industrial grade of phenol.

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