

TECHNICAL SERVICE RESPONSE NO.: UT003

<u>Subject</u>: Identification of "Angel Hair" Found 30 Miles North of Sacramento, California on I-5 (Nov. 11, 1999)

<u>Date</u>: February 26, 2002

Requested By: Brian Boldman Mark Rodeghier

<u>Reported By</u>: P. A. Budinger Analytical Scientist

Background/Objective: Thomas Brewer was driving on I-5 about 30 miles north of Sacramento on his way to Alaska. He observed strings of fiber-like material of varying lengths (from a few inches to 50 feet) floating at different elevations. This continued for approximately 10-15 miles, when he noticed some of it on the highway shoulders, trees, signs and lawn. He collected samples of the material and noted the material diminished in size over a period of time. His detailed description may be found in Art Bell's website: <u>http://artbell.com/stringy.html</u>. No other unusual phenomena were noted in the sky. There were thin wispy clouds overhead. Brian Boldman forwarded three samples of this material to Frontier Analysis on recommendation of Mark Rodeghier. Thomas Brewer took photographs of the material at the site of the fall. Some selected pictures follow. These were obtained from the Art Bell website.



"Angel Hair" on Brewer's Truck



" Angel Hair" on Pavement (Cassette Tape for Size Estimate)



"Angel Hair" on Pavement

Conclusions:

1.) Both the white fiber and volatile materials associated with the fiber were identified. The fiber is a polymer containing protein amide type linkages. This suggests an animal/biological source is involved in its manufacture. Also coating the fiber are droplets which appear to be a natural long chain fatty ester type material. The volatiles are primarily composed of a mix of the following light hydrocarbons: 2-methyl propane; 2-methyl-1-propene; 2-methyl-1-butene; 2-methyl pentane; 3-methyl pentane; hexane; dimethyl-pentane; 2 C₆H₁₂ (MW=84) hydrocarbon structures (specific isomers unidentified); one C₈H₁₆ hydrocarbon (MW=112) (specific isomer unidentified). There are possibly trace amounts of heavier hydrocarbons such as two C₂₀H₄₂ components and a C₂₃H₄₃ component. Additionally, trace amounts of carbonyl sulfide (COS) and carbon disulfide (CS₂) are indicated.

2.) No weight loss or physical degradation of the fiber was observed over a period of 4 months.

3.) The fibrous material is identical to that from an angel hair fall in Los Gatos California (near San Jose) on October 19-20,1977 (see Frontier Analysis T.S.R. No.: UT002.)

4.) The material does not originate from spiders, i.e. spider webs. It is more similar to that of silk from caterpillars, e.g. silkworms, tent caterpillars etc. While the material has been identified as being of biological origin, this does not rule out a possible "intelligent" influence. Spider web/silk properties are desirable for many applications.

Procedure:

Samples: Three samples were submitted in vials tightly sealed with rubber stoppers. One was indicated to be fresh (unopened). The other opened and resealed. Another had asphalt particulates in it.



Infrared spectra (FT-IR) were obtained from the fibers of all samples on the Nicolet Magna 560 and Avatar 360 spectrometers using the Harrick SplitPea® cell. Additional spectra on the "fresh" fiber were also acquired over a period of four months to see if any changes occurred to the fibers over time. Also, a spectrum was obtained from a chloroform extract of the fiber. The weight of the fiber was recorded 48 days to check for weight loss. Additionally, headspace GC/MS analysis was performed on the "fresh" sample for the identification of any volatile components on the day of sample receipt. Microscope photographs were obtained using the Leika GZ6 stereomicroscope.

Results:

The results of the individual tests done on the three samples follow. These results are summarized in the conclusions section on the first page of this report.

Microscope Analysis: A microscope photograph obtained at 60x magnification shows fine fibers, of which most are bundled in a rope-like fashion. A few singlets are observed and are of very fine denier, probably less than 1 micron¹. There are tiny droplets which are more easily viewed on the single strands. A few of these strands are selected with red arrows in the photograph.

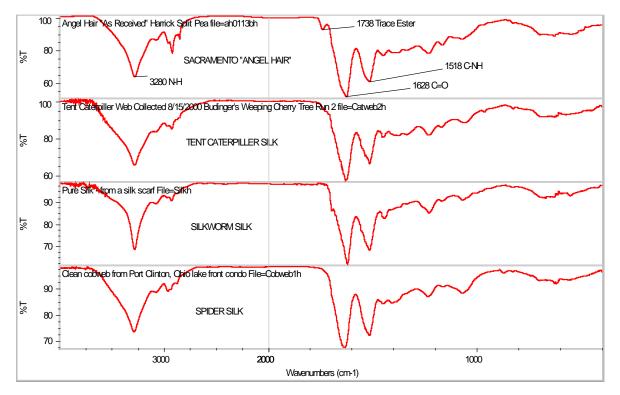
¹ SEM microscope analysis of 'angel hair' fibers from the Los Gatos, California fall shows fibers were <1 micron $(0.017-0.27\mu)$. (T.S.R. No.: 002)

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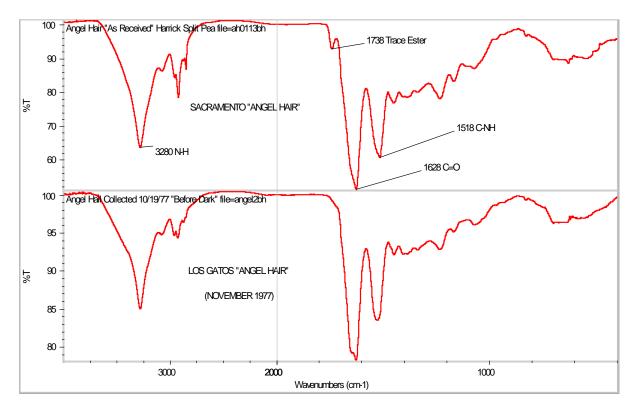


Headspace GC/MS Analysis: Headspace volatiles from the tightly sealed container of the fresh sample was carefully sampled and examined before any outside air could be introduced. This analysis shows primarily a light hydrocarbon mix consisting of: 2-methyl propane; 2-methyl-1-propene; 2-methyl-1-butene; 2-methyl pentane; 3-methyl pentane; hexane; dimethyl-pentane; 2 C₆H₁₂ (MW=84) hydrocarbon structures (specific isomers unidentified); one C₈H₁₆ hydrocarbon (MW=112) (specific isomer unidentified). Also indicated are carbonyl sulfide (COS) and carbon disulfide (CS₂). There are possibly heavier hydrocarbons present such as two C₂₀H₄₂ components and a C₂₃H₄₃ component. None of these materials were detected in a blank run for comparison to the sample headspace. The detailed information, i.e. chromatogram with identified peaks is included in an addendum.

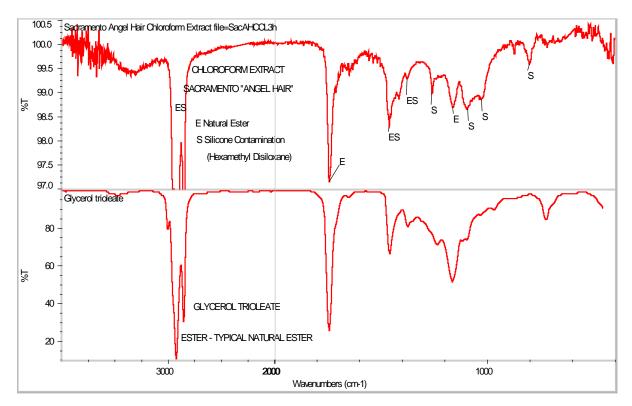
FT-IR: Infrared spectra of the filaments from the three samples are identical and show they are composed of a material with a prominent secondary amide linkage which is typical of protein. Characteristic amide C=O and NH modes are present at 1628 and 1518 cm⁻¹. NH stretch occurs between 3600-3100 cm⁻¹ (max 3280 cm⁻¹). More specifically, the spectra are similar to references of spider web, but are not a match. There are subtle band ratio differences and frequency shifts. These are significant enough to rule spiders out as a source of the material. The spectra are more akin to references of silk derived from caterpillars such as silkworms or tent caterpillars, etc. However, it is not conclusive that this is the source. Following are some spectra for comparison. These include: a representative spectrum from the many runs of the Sacramento "angel hair", tent caterpillar silk, 'silkworm' silk, spider silk.



The spectral bands from the filaments also match bands from an angel hair fall in Los Gatos, California in November 1977. Following are spectra comparing the Sacramento and Los Gatos "angel hair".

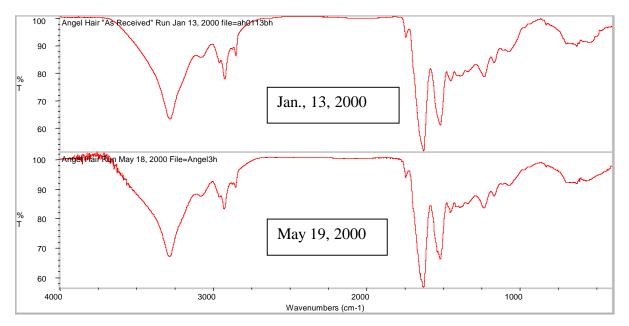


However, the Sacramento samples additionally show a very weak ester C=O band. A chloroform solvent extract from the "angel hair" concentrated this ester². An infrared spectrum of the extract more specifically suggests the ester is similar to a glycerol ester with long hydrocarbon chains. This is a typical natural ester. Also detected is a silicone contaminant which is most likely an additive from the rubber stopper used to seal the "angel hair" container. Spectra of the "angel hair" extract and a reference of a glycerol triester follow.



Several spectra of the same angel hair fiber (fresh sample) taken over a four month period show no differences. This indicates no deterioration of the sample occurred. Following are the first and last spectra of the series.

² The "angel hair" fiber is insoluble in chloroform.



Additionally, weights from the "fresh" sample taken over a period of 48 days reveal no evidence for weight loss. The data follow.

Days	Weight (mg)
0	5.4
1	5.7
2	5.5
3	5.5
13	5.5
17	5.3
48	5.7

Acknowledgment: I would like to thank Richard Wilson for the GC/MS headspace analysis and some initial FT-IR analysis.

File: UT003.DOC

Phyllis A. Budinger

Distribution:

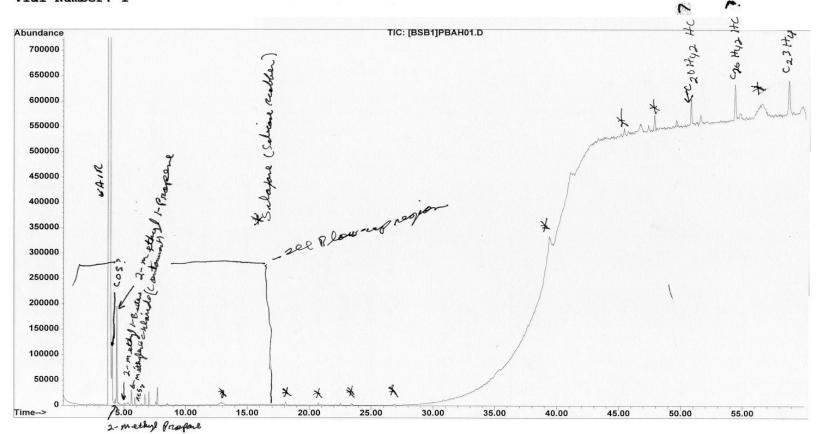
Richard Wilson Ted Phillips Mark Rodeghier Brian Boldman Nancy Talbott T. S. R. No.: UT002 P. A. Budinger Page 8

ADDENDUM

Headspace GC-MS Analysis of Sacramento "Angel Hair" (GC Chromatogram – Full 1 Hour Run)

Headspore of Sample

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File : C:\HPCHEM\4\DATA\BSB\PBAH01.D
Operator : [BSB1]RLW 1/13/00
Acquired : 13 Jan 2000 14:11 using AcqMethod HSDB1
Instrument : GC/MS #4
Sample Name: <u>Angel Hair</u> 1/13/00
Misc Info : HS of Angel Hair 0.1 ul Split 5 DB-1
Vial Number: 1
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Headspace GC-MS Analysis of Sacramento "Angel Hair" (GC Chromatogram – Expanded 0-14 minutes)

File : C:\HPCHEM\4\DATA\BSB\PBAH01.D Operator : [BSB1]RLW 1/13/00 Acquired : 13 Jan 2000 14:11 using AcqMethod HSDB1 Instrument : GC/MS #4 Sample Name: Angel Hair 1/13/00 Misc Info : <u>HS of Angel Hair</u> 0.1 ul Split 5 DB-1 Vial Number: I

