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ORGANIC LABORATORY ANALYSIS REPORT

JOB NUMBER C0BEE907
PO NUMBER Credit Card

for

Joe Cerniglia
The International Group for Historic Aircraft Recovery

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ORGANIC LABORATORY ANALYSIS REPORT

Requester: Joe Cerniglia
Job Number: C0BEE907
Analysis Date: 12 Oct 2011

Purpose:

The purpose was to determine the identity of the reddish-brown spots which were stuck to the inner walls of a glass bottle.

Secondary purposes were to avoid sampling the loose blackish particles and to avoid the liquid-like residue also on the inner walls of the bottle.

Summary:

The reddish-brown spots appeared to be a mixture, possibly of:

organic acid salt similar to iron (II) oxalate dihydrate;

a cellulose derivative such as hydroxyethyl cellulose, and / or

inorganic salts possibly containing sulfate, carbonate, hydroxide and / or silicate.

The reddish-brown residue did not match the previously analyzed samples of sticky residue and flakey residue (Dr. Jennifer Mass, Scientific Analysis of Fine Art, Berwyn PA, Sept 2007).

SED-EDS can help to determine the atomic elements (Z>=B) of non-volatile solids and thereby might help to narrow the list of possible salts in the reddish-brown spots. Raman spectroscopy is complementary to FTIR and is more sensitive than FTIR to many common inorganic oxides and their related salts.

Experimental:

Representative samples of the reddish spots were transferred to an infrared transmitting substrate and examined by Fourier Transform Infrared Spectroscopy (FTIR) with the FTIR microscope in transmission mode.

Results and Interpretations:

Spectrum 1 shows the FTIR spectra, in overlay format, of two measurements of the reddish-brown spots on the inner walls of the glass bottle. The reddish-brown spots appeared to be a mixture, possibly of:

organic acid salt similar to iron (II) oxalate dihydrate (e.g., bands at ~ 3397, 1641, 1364, 1321, and 824 cm^{-1});

a cellulose derivative such as hydroxyethyl cellulose (e.g., bands at ~ 3397, 2944, 1399 and 1059 cm^{-1}), and / or

inorganic salts possibly containing sulfate (~1059 cm^{-1}), carbonate (~ 1399 cm^{-1}), hydroxide (~3397 and 1641 cm^{-1}) and / or silicate (~1059 cm^{-1}).

Spectra 2, 3 and 4 show the overlays of the reddish-brown spots with reference spectra of iron (II) oxalate dihydrate (a yellow-red material), of hydroxyethylcellulose (a cellulose derivative) and of ordinary glass (silicate material), respectively. The approximate matches support the possible identifications discussed above.

Comparison to the previous report (Scientific Analysis of Fine Art, Sept 2007)

Spectra 5 and 6, respectively, show the overlays of the reddish-brown residue in spectrum 1 with the spectrum of the previously analyzed sticky residue from the corner of a bottle (identified as wild silk by Dr. Mass) and with the spectrum of the flakey residue from the center of the bottom of the same bottle (identified by Dr. Mass as a mixture, which possibly contained ester such as lanolin, linseed oil or rapeseed oil).

The absence of a match in spectrum 5 indicates that the reddish-brown residue probably did not contain material similar to the possible wild silk in the sticky residue

The differences in spectrum 6 (i.e., strong bands of the flakey residue at ~ 2918, 2850, 1735, 1512, and 1015 cm^{-1} were not apparent in the reddish-brown spot) outweigh the possible similarities (e.g., bands at ~ 3397, 1361, 1321 and 823 cm^{-1} of the reddish-brown spot were possibly also observed in the flakey residue), and hence the reddish-brown residue was not a good match to the flakey residue.

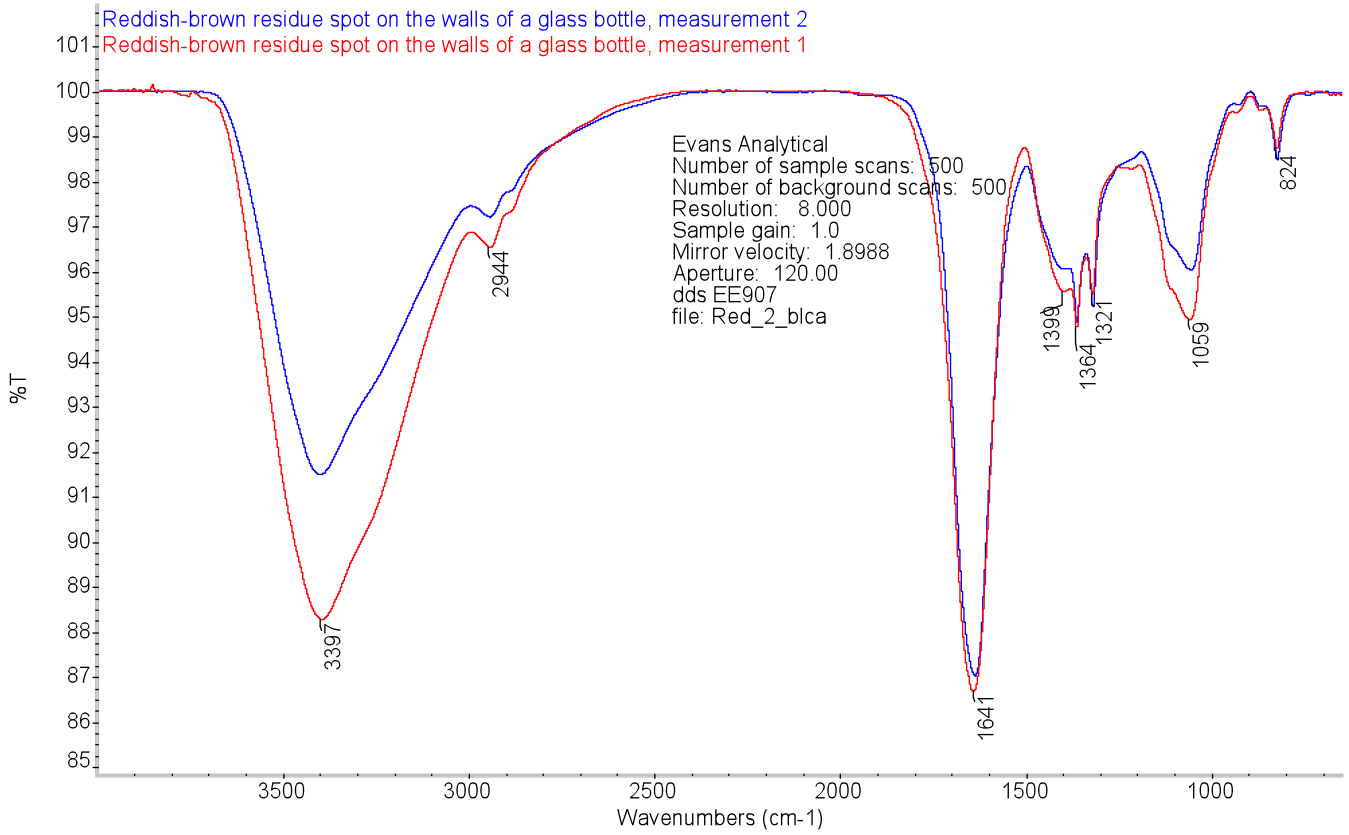
FTIR is often used for the qualitative identification of functional groups or for the identification of entire organic compounds, typically with the aid of spectral databases. Assignment of spectral features to functional groups or the identification of a compound can be made with relative certainty, in some instances. However in many cases the presence of spectral features and functional groups cannot be traced unambiguously to one specific compound, especially in the analysis of mixtures. Where identification is relatively certain, the report will so state. Where identification is ambiguous, the report will provide possible compounds or classes of materials that may be present in the sample.

If questions arise as you review the results of this analysis, the report author or any other member of our technical staff will be available for consultation.

After reviewing this report, you may assess our services using an electronic service evaluation form. This can be done by clicking on the link below, or by pasting it into your internet browser. Your comments and suggestions allow us to determine how to better serve you in the future. http://www.eaglabs.com/evaluate.htm?job=C0BEE907

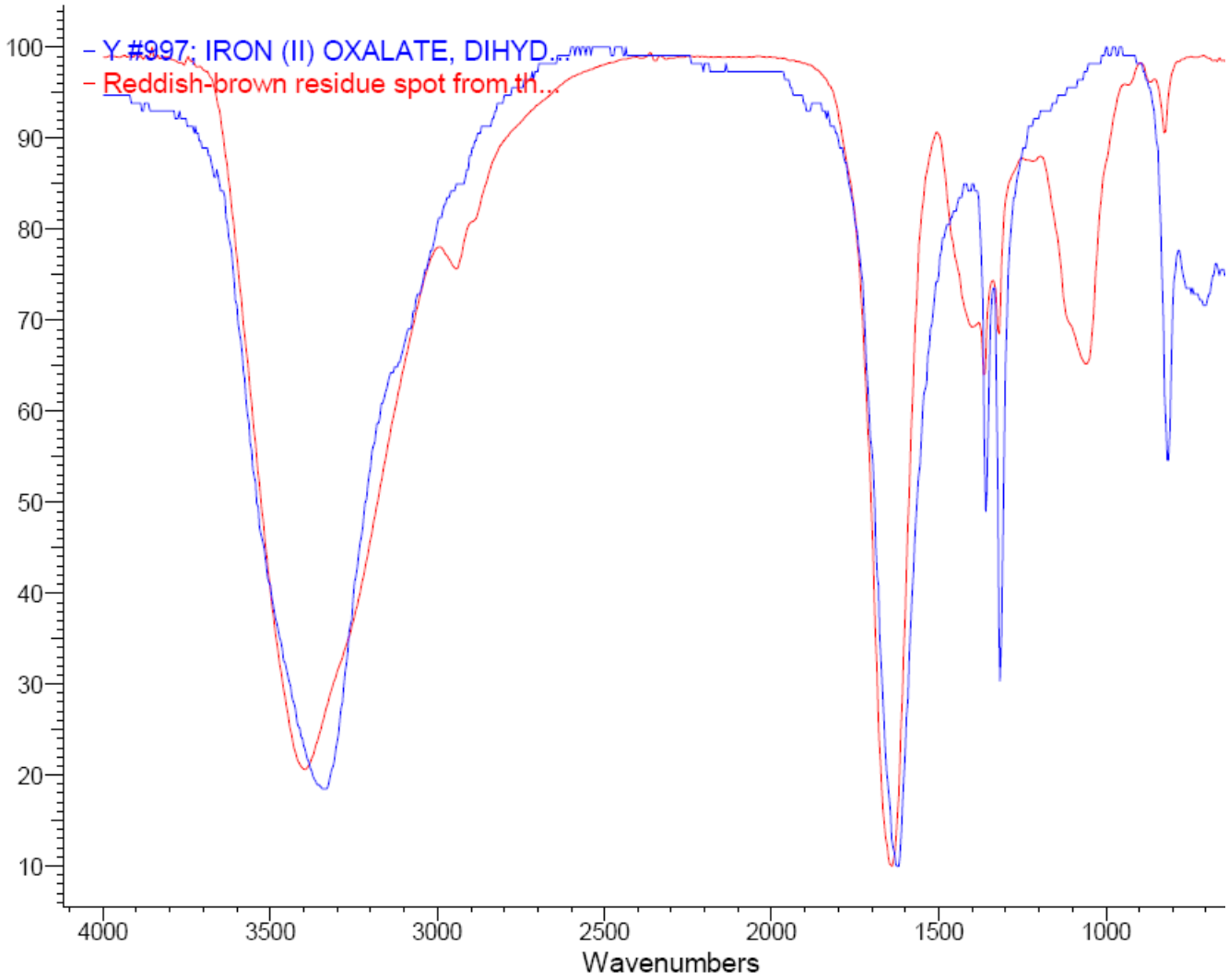
Appendix 1

Infrared Spectroscopy (IR) is the study of molecular vibrations. It is an extremely useful analytical technique, providing specific information about chemical bonding and molecular structure, most suitably for organic materials. The technique is based on the fact that bonds and groups of bonds vibrate at characteristic frequencies. When exposed to infrared radiation, a molecule selectively absorbs infrared frequencies that match those of its allowed vibrational modes. Therefore, the infrared absorption spectrum of a material reveals which vibrations, and thus functional groups, are present in its structure. It should be noted that vibrations that do not involve a change in dipole moment, as in O_2 and N_2, do not absorb infrared radiation. Thus, IR spectra can be collected in air.

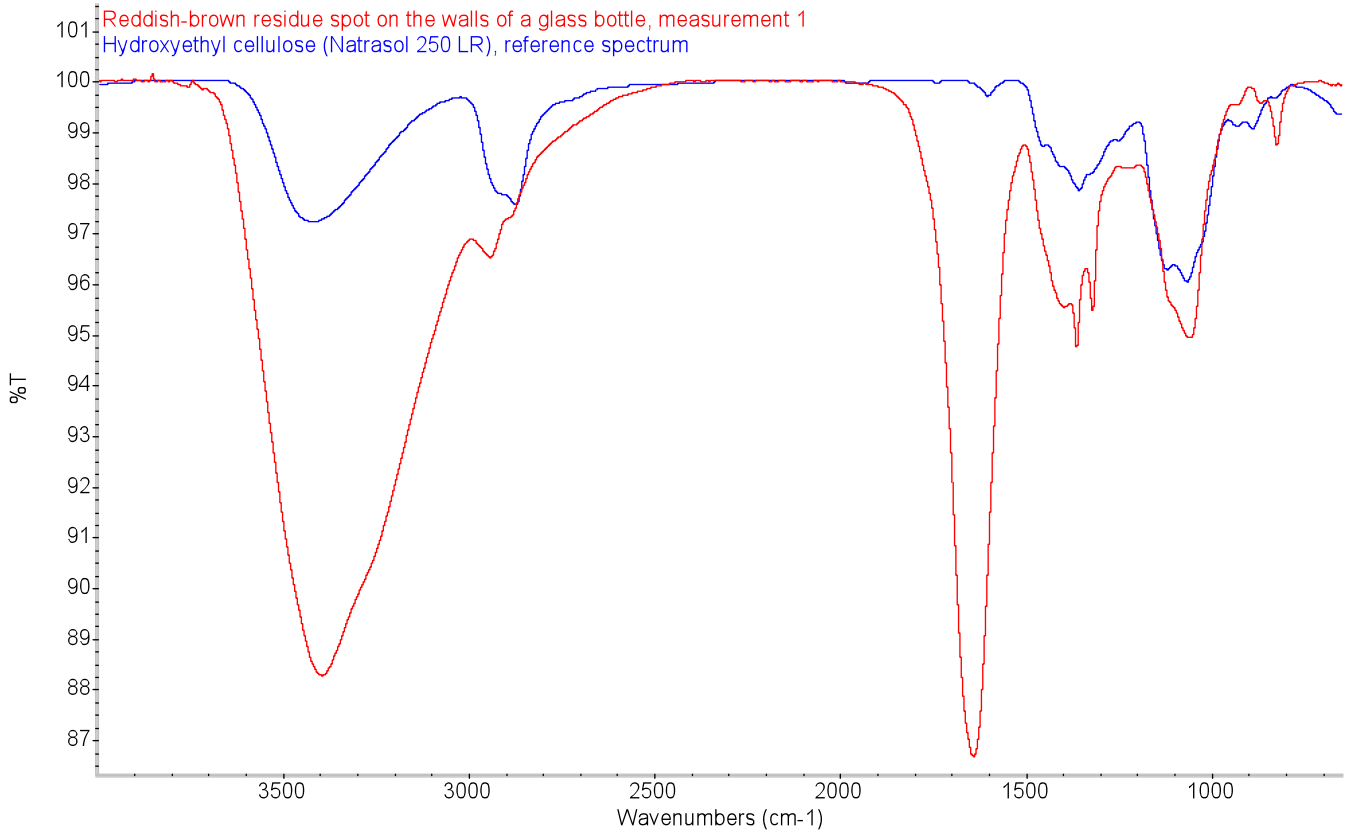

Reddish-brown residue spot on the walls of a glass bottle, measurement 2
Reddish-brown residue spot on the walls of a glass bottle, measurement 1
Evans Analytical
Number of sample scans: 500
Number of background scans: 500
Resolution: 8.000
Sample gain: 1.0
Mirror velocity: 1.8988
Aperture: 120.00
dds EE907
file: Red_2_blca
%T
Wavenumbers (cm-1)
3397
2944
1641
1399
1364
1321
1059
824

Spectrum 1

Reference (blue) is iron (II) oxalate dihydrate



Spectrum 2


Reddish-brown residue spot on the walls of a glass bottle, measurement 1
Hydroxyethyl cellulose (Natrasol 250 LR), reference spectrum
%T
101
100
99
98
97
96
95
94
93
92
91
90
89
88
87
3500
3000
2500
2000
1500
1000
Wavenumbers (cm-1)

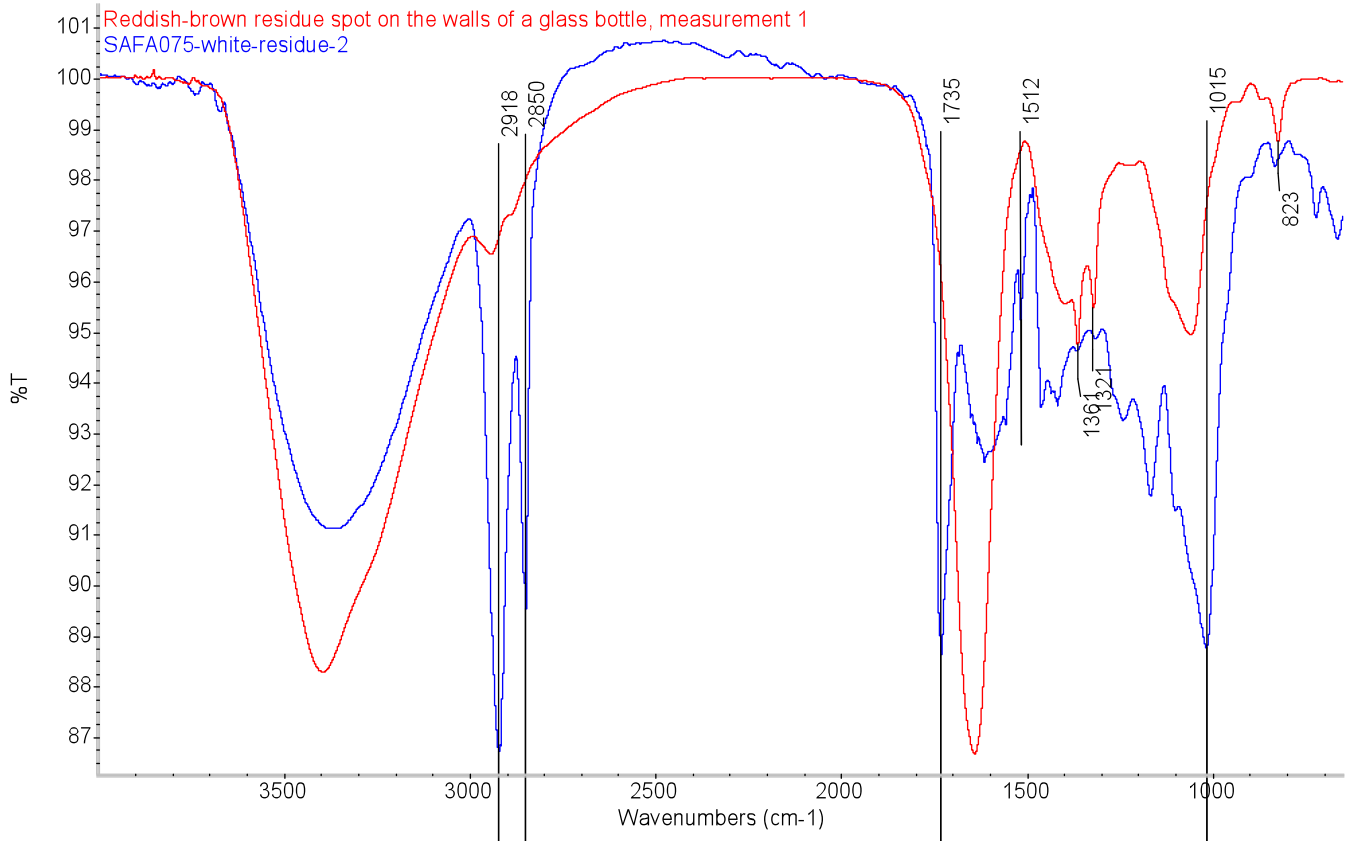
Spectrum 3


Reddish-brown residue spot on the walls of a glass bottle, measurement 1
Ordinary glass, reference spectrum
%T
102
101
100
99
98
97
96
95
94
93
92
91
90
89
88
87
3500
3000
2500
2000
1500
1000
Wavenumbers (cm-1)

Spectrum 4


Reddish-brown residue spot on the walls of a glass bottle, measurement 1
SAFA075-white bottle contents
%T
101
100
99
98
97
96
95
94
93
92
91
90
89
88
87
3500
3000
2500
2000
1500
1000
Wavenumbers (cm-1)

Spectrum 5


Reddish-brown residue spot on the walls of a glass bottle, measurement 1
SAFA075-white-residue-2
%T
Wavenumbers (cm-1)
2918
2850
1735
1512
1015
1391
1321
823

Spectrum 6