



**Testing Cert.  
#2797.01**

**ORGANIC LABORATORY ANALYSIS REPORT  
Part 2, amended October 27<sup>th</sup> 2011**

**JOB NUMBER C0BEE907  
PO NUMBER Credit Card**

for

Joe Cerniglia  
The International Group for Historic Aircraft Recovery

Prepared by:

---

David Saperstein, PhD  
Scientist, FTIR, GCMS and Raman Services  
(Tel. 408-530-3750; dsaperstein@eaglabs.com)

Reviewed by:

---

Angela Craig, Ph.D.  
Manager, FTIR, Raman, GC-MS Services  
(Tel. 408-530-3642; acraig@eaglabs.com)

**ORGANIC LABORATORY ANALYSIS REPORT**

Requester: Joe Cerniglia  
Job Number: C0BEE907  
Analysis Date: 24 Oct 2011

**Purpose:**

The purpose was to determine the identity of three additional materials in a glass bottle, labeled "Campana's Italian Balm" (apparently meant to be used with the dispensing cap positioned below the glass). The additional materials were the liner / seal in the metal cap, the liquid-like material on the inner walls and the black-looking particles also on the inner walls of the bottle, see [Table 2](#).

Reddish-brown particles stuck to the inner wall were analyzed previously in part 1 of this report (October 12, 2011).

A secondary purpose was to determine whether any of the Campana's Italian Balm ingredients listed in [Table 1](#) (and underlined in the text) were present in the analyzed materials.

Table 1

Ingredients list for Campana's Italian Balm Cosmetic in <i>Clinical Toxicology of Commercial Products</i> by Marion N. Gleason, 1957, published by Williams and Wilkins
Essential oils
Alcohol
Phenol
Benzoic Acid
Gum Tragacanth
Glycerin
Sorbitol

A tertiary purpose was to compare the findings in this report with the previously analyzed flakey white residue in a report by Dr. Jennifer Mass, Scientific Analysis of Fine Art, Berwyn PA, Sept 2007.

**Summary:**

[Table 2](#) summarizes the FTIR results of the materials from the Campana's Italian Balm bottle in parts 1 and 2 of the report, and [Table 3](#) compares the materials analyzed in this report with the analysis of the flakey white residue by Dr. Mass in 2007.

Table 2

Sample description	Possible principal components	Other possible materials
Reddish-brown particle (see part 1 of this report, Oct 12 <sup>th</sup> )	Organic acid salt similar to iron (II) oxalate dihydrate. Cellulose derivative similar to hydroxyethylcellulose.	Inorganic salts possibly containing sulfate, carbonate, hydroxide and / or silicate.
Liner / seal of the cap	Cellulose possibly paper, cotton or similar species. Saccharide (polyol) similar to corn starch.	Organic acid salts similar to iron (II) oxalate, copper (II) oxalate and/or nickel oxalate (probably as hydrates). Cellulose derivatives. Inorganic species.
Liquid-like material	Cellulose derivatives. Polyol similar to sodium alginate, <u>sorbitol</u> , <u>glycerin</u> , and/or corn starch. Organic acid salt similar to iron sodium oxalate, hydrate and / or iron potassium oxalate, trihydrate	Inorganic species. Cellulose. Ester.
Black particle – (blue features)	Aliphatic acid salt similar to copper stearate.	OH groups of moisture and/or of inorganic species.
Black particle – (brown features)	Organic acid salt similar to iron (II) oxalate, copper (II) oxalate and/or nickel oxalate (probably as hydrates). Aliphatic acid salt similar to copper stearate. Cellulose derivatives.	Inorganic species.

The compositions of the essential oils in the ingredients list ([Table 1](#)) are not known to EAG. Aromatic compounds such as phenol and benzoic acid were not observed in any of the

samples. Alcohol would be expected to have evaporated prior to analysis. A portion (i.e., a possible component) of the reference spectrum of Tragacanth gum was possibly consistent with the cellulose derivative (carboxymethyl cellulose) identified in this report.

Table 3

<b>Sample description (Campana's Italian Balm bottle)</b>	<b>Comparison to the white residue (analyzed in 2007 by Dr. Mass)</b>
Reddish-brown particle	Not a good match (see part 1 of this report, Oct 12 <sup>th</sup> 2011); possible oxalate bands appear to match
Liner / seal of the cap	Not a good match (spectra <a href="#">26</a> and <a href="#">27</a> )
Liquid-like material	Not a good overall match; possible match to small ester component of the liquid ( <a href="#">spectrum 28</a> )
Black particle – (blue features)	Not a good match; possible chemical match of ester and acid salt groups ( <a href="#">spectrum 29</a> )
Black particle – (brown features)	<b><u>Partial spectral match</u></b> ; possible chemical matches of OH, ester/acid salt and possible oxalate groups (spectra <a href="#">30</a> and <a href="#">31</a> )

### Experimental:

Representative samples of each of the materials 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:

#### *Liner / seal of the cap*

The liner / seal material appeared to be a mixture, and the material shown in [Spectrum 1](#) was possibly the majority component. Two measurements are shown in overlay format in [spectrum 1](#) (the spectral match supports the designation of the majority component), and their spectral features suggest the presence of possible cellulosic material similar to cotton or paper (e.g., bands at ~ 3349, 2924, 1162, 1112, 1060 and 1038 cm<sup>-1</sup>) in the liner / seal.

[Spectrum 2](#) shows the FTIR spectrum of a third measurement of the liner / seal and was possibly a saccharide (polyol) similar to corn starch (e.g., largest C-O band at ~ 1029 cm<sup>-1</sup> vs the C-O band of cellulose at ~ 1060 cm<sup>-1</sup> in [spectrum 1](#)). The saccharide material appeared to be a minority component in the liner / seal.

Other possible components in spectra [1](#) and [2](#) were:

possible organic acid salts similar to iron (II) oxalate, copper (II) oxalate and/or nickel (II) oxalate (possibly as hydrates), i.e., small sharp bands at  $\sim 1365$  and  $1320\text{ cm}^{-1}$  in [spectrum 1](#);

possible cellulose derivatives such as carboxymethyl cellulose (e.g., bands at  $\sim 3349$ ,  $2924$ ,  $1642$  and  $1060\text{ cm}^{-1}$  in [spectrum 1](#)), and

possible inorganic species (e.g., bands at  $\sim 3365$ ,  $1647$  and  $1045\text{ cm}^{-1}$  in spectra [1](#) and [2](#)).

Spectra [3](#), [4](#) and [5](#) show the overlays of the liner / seal measurements in spectra [1](#) and [2](#) with reference spectra of cellulose (cotton example), carboxymethyl cellulose (sodium salt) and corn starch, respectively. The matches in spectra [3](#) and [5](#) and the approximate match in spectrum [4](#) support the identifications discussed above.

#### *Liquid-like material on the inner wall surface*

[Spectrum 6](#) shows the FTIR spectra, in overlay format, of two measurements of the liquid-like material on the surface of the inner walls. Additional measurements are shown in [spectrum 7](#) (third measurement) and [spectrum 8](#) (fourth and fifth measurements). The differences in spectra [6](#), [7](#) and [8](#) indicate that the liquid-like material was a mixture. Based on the approximate fits of the reference spectra, the components of the liquid may have included:

cellulose derivatives similar to carboxymethyl cellulose (e.g., bands at  $\sim 3357$ ,  $2931$ ,  $1608$ ,  $1414$ ,  $1112$  and  $1051\text{ cm}^{-1}$  in [spectrum 6](#));

polyol similar to sodium alginate, sorbitol and/or glycerin (e.g., bands at  $\sim 3357$ ,  $2931$ ,  $1608$ ,  $1414$ ,  $1112$  and  $1051\text{ cm}^{-1}$  in [spectrum 6](#)),

polyol similar to corn starch (e.g., bands at  $\sim 3333$ ,  $2928$ ,  $1600$ ,  $1411$ ,  $1112$  and  $1079$ ,  $1024\text{ cm}^{-1}$  in [spectrum 7](#));

possible cellulose (e.g., bands at  $\sim 3357$ ,  $2931$  and  $1051\text{ cm}^{-1}$  in [spectrum 6](#));

organic acid salt similar to iron sodium oxalate, hydrate and / or to iron potassium oxalate, trihydrate (e.g., bands at  $\sim 3533$ ,  $3447$ ,  $1677$ ,  $1415$ ,  $1334$ ,  $1279$ ,  $811$  and  $780\text{ cm}^{-1}$  in [spectrum 8](#));

ester (e.g., band at  $\sim 1747\text{ cm}^{-1}$ ), and

possible inorganic species (e.g., bands at  $\sim 3345$ ,  $1677/1605$  and  $1050\text{ cm}^{-1}$  in spectra [6](#), [7](#) and [8](#)).

The possible components of the liquid-like materials in spectra [6](#), [7](#) and [8](#) are shown overlaid with appropriate library reference spectra in spectra [10](#) (reference: sodium alginate), [11](#) (reference: sorbitol), [12](#) (reference: glycerin), [13](#) (reference: cornstarch), [14](#) (reference: cotton), [15](#) (reference: iron sodium oxalate, hydrate) and [16](#) (reference: iron potassium oxalate, hydrate). The approximate match support the possible identification discussed above.

In addition to the possible components discussed above, the liquid-like material may have contained components or residue from Tragacanth gum (e.g., bands at ~ 3357, 2931, 1608, 1414, 1112 and 1051  $\text{cm}^{-1}$  in [spectrum 6](#)). The partial match in [spectrum 17](#) of the liquid-like material with the spectrum of a library reference spectrum of Tragacanth gum suggests that a component or residue of the gum cannot be ruled out as a component of the materials in the bottle.

#### *Black particle – blue features*

[Spectrum 18](#) shows the FTIR spectra, in overlay format, of two measurements of the blue features of the black particles in the bottle. The blue portion was probably aliphatic acid salt, possibly a copper salt similar to copper stearate (e.g., bands at ~ 2917, 2850, 1586, 1471 and 1413  $\text{cm}^{-1}$ ). Note that copper stearate is blue-green in color (Handbook of Chemistry and Physics, CRC Press) and possibly matches the color of the blue feature in the black particle.

The match in [spectrum 18](#) shows the spectral consistency of the blue feature.

The broad features at ~3387  $\text{cm}^{-1}$  and ~1650  $\text{cm}^{-1}$  in [spectrum 18](#) suggest the presence of OH groups and possibly signify moisture or adsorbed water of an inorganic species.

[Spectrum 19](#) shows the overlay of the blue feature in spectrum 18 with a reference spectrum of copper stearate. The match indicates that the blue feature contained aliphatic acid salt, possibly copper stearate.

#### *Black particle – brown features*

[Spectrum 20](#) shows the FTIR spectrum, in overlay format, of two measurements of the brown feature in the black particle. The brown feature was a mixture, possibly consisting of:

- one or more organic acid salts such as iron (II) oxalate hydrate, copper (II) oxalate hydrate and / or nickel (II) oxalate hydrate (e.g., bands at ~ 3389, 1651, 1362, 1319 and 823  $\text{cm}^{-1}$ );

- aliphatic acid salt such as copper stearate (e.g., bands at ~ 2954, 2916, 2850, and 1586  $\text{cm}^{-1}$ );

- cellulose derivatives such as carboxymethyl cellulose (e.g., bands at ~ 3389, 2916, 1651, 1413, and 1053  $\text{cm}^{-1}$ ), and

- inorganic species (e.g., bands at ~ 3389, 1651, and 1053  $\text{cm}^{-1}$ ).

The approximate match in [spectrum 20](#) shows a small variability of the components of the brown feature and/or show an incomplete separation of the brown and blue features in the FTIR samples.

Spectra [21](#), [22](#), [23](#), [24](#) and [25](#) show the overlays of the brown feature in [spectrum 20](#) with reference spectra of iron (II) oxalate hydrate, copper (II) oxalate hydrate, nickel (II) oxalate hydrate, copper stearate and carboxymethyl cellulose, respectively. The approximate matches support the identifications discussed above.

*Comparison of spectra (details of the summary in [Table 3](#))*

In this section, the materials in the Campana's Italian Balm bottle analyzed in parts 1 and 2 of this report (see [Table 2](#) and discussion above) are spectrally compared to the flakey white residue from the center of the bottom of the bottle fragment (identified by Dr. Mass in 2007 as a mixture, which possibly contained ester such as lanolin, linseed oil or rapeseed oil).

Spectra [26](#) and [27](#) show the overlays of the liner / seal material in spectra [1](#) and [2](#), respectively, with the flakey white residue (spec 2) in Dr. Mass's report. The large differences (i.e., strong bands of the flakey residue at  $\sim 2918$ ,  $2850$ ,  $1735$ ,  $1512$ , and  $1015\text{ cm}^{-1}$  were not apparent in the liner / seal material) outweigh the possible similarity (OH band at  $\sim 3368\text{ cm}^{-1}$  in both the cap and the flakey residue, possibly due to moisture), and hence the cap material was not a good match to the flakey residue.

[Spectrum 28](#) shows the overlay of the liquid-like material in [spectrum 6](#) with the flakey white residue (spec 2). The apparent differences (i.e., strong bands of the flakey residue at  $\sim 2918$ ,  $2850$ ,  $1512$ ,  $1172$  and  $1015\text{ cm}^{-1}$  were not apparent in the liquid-like material) suggest that the liquid-like material was not a good overall match to the white residue. However, a small ester band/shoulder at  $\sim 1747\text{ cm}^{-1}$  in the liquid-like material, possibly was related to the strong ester band in the white residue at  $\sim 1735\text{ cm}^{-1}$ , and hence the liquid-like material may have contained a small amount of ester possibly similar to some of the ester in the flakey residue.

[Spectrum 29](#) shows the overlay of the blue feature in the black particle in [spectrum 18](#) with the flakey white residue (spec 2). The differences (i.e., strong bands of the flakey residue at  $\sim 3385$ ,  $1735$ ,  $1512$ ,  $1172$  and  $1015\text{ cm}^{-1}$  were not apparent in the blue feature material) suggest that the blue feature was not a good match to the white residue.

Although there was not a good spectral match, there may be some chemical similarity of the flakey white residue and the blue feature ([Spectrum 29](#)). Both contained spectrally similar aliphatic hydrocarbon groups (bands at  $\sim 2920$  and  $2850\text{ cm}^{-1}$ ). In addition, the ester of the flakey white residue ( $1735\text{ cm}^{-1}$ ) and the aliphatic acid salt of the blue feature ( $1586\text{ cm}^{-1}$ ) may have been related if ester of the Italian balm in the authentic Campana's Italian Balm bottle hydrolyzed to form carboxylic acid, which in turn reacted with the metals in the Campana's Italian Balm cap to form the observed aliphatic acid salt.

Spectra [30](#) and [31](#) show the overlays of the brown feature in the black particle in [spectrum 20](#) with the flakey white residue (spec 2 and spec 1, respectively). The partial matches (e.g., OH band at  $3389\text{ cm}^{-1}$ , CH bands at  $\sim 2920$  and  $2850\text{ cm}^{-1}$  and possible oxalate bands at  $\sim 1362$  and  $1319\text{ cm}^{-1}$  suggest that one or more components of the brown feature and of the flakey white residue may be related (e.g., the CH bands may be related via an ester to aliphatic acid salt mechanism suggested in the paragraph above). The possibility that the flakey white residue may have also contained oxalate is tantalizing.

### **Suggestions for additional analyses:**

Other techniques may be useful, e.g., SEM-EDX to obtain the atomic elements ( $Z \geq B$ ) and Raman spectroscopy to complement the FTIR analysis, to help determine the full identity of the materials in the bottle.

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.

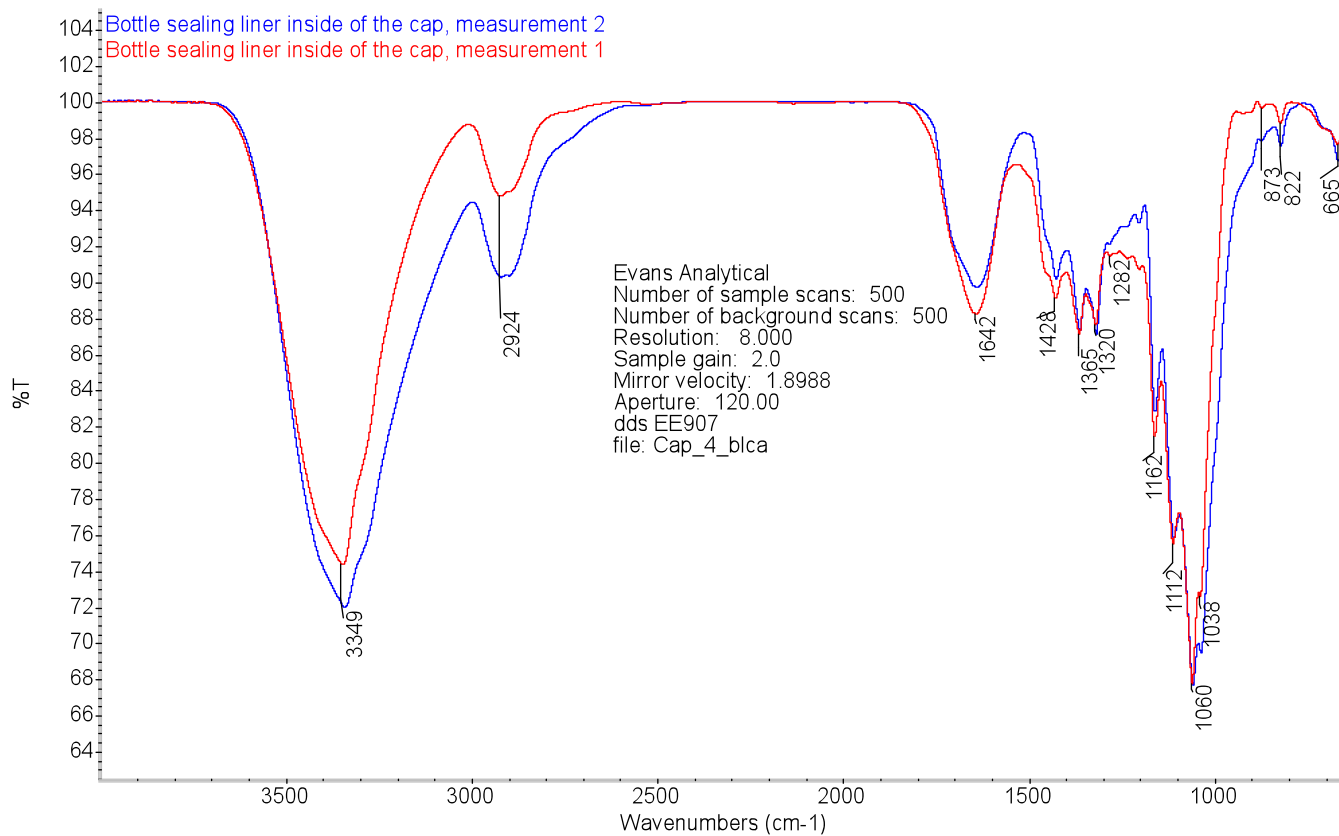
After reviewing this report, you may assess our services using an electronic service evaluation form. This can be done by clicking on the link below, or by pasting it into your internet browser. Your comments and suggestions allow us to determine how to better serve you in the future.  
<http://www.eaglabs.com/evaluate.htm?job=C0BEE907>

**This analysis report should not be reproduced except in full, without the written approval of EAG.**

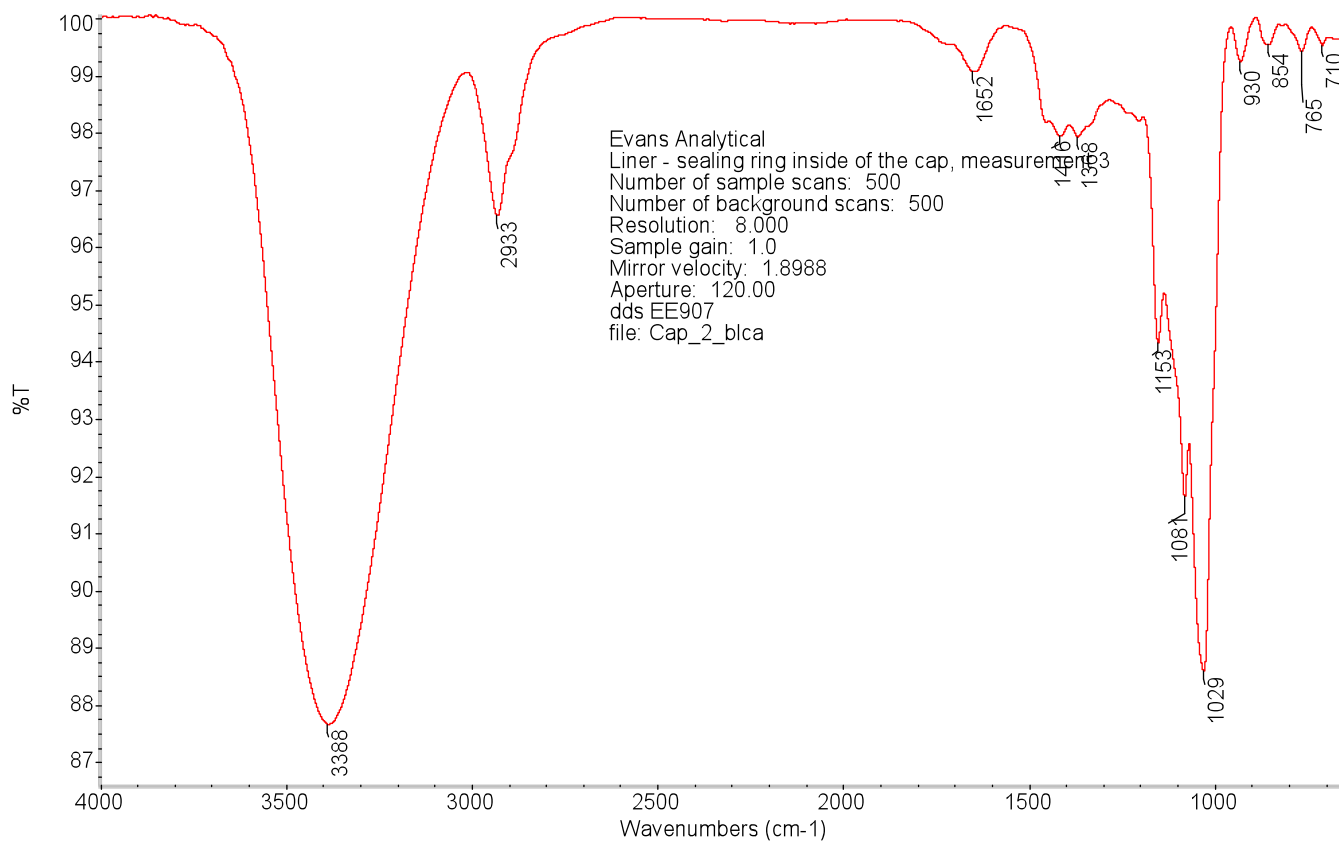
### **Appendix 1**

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.

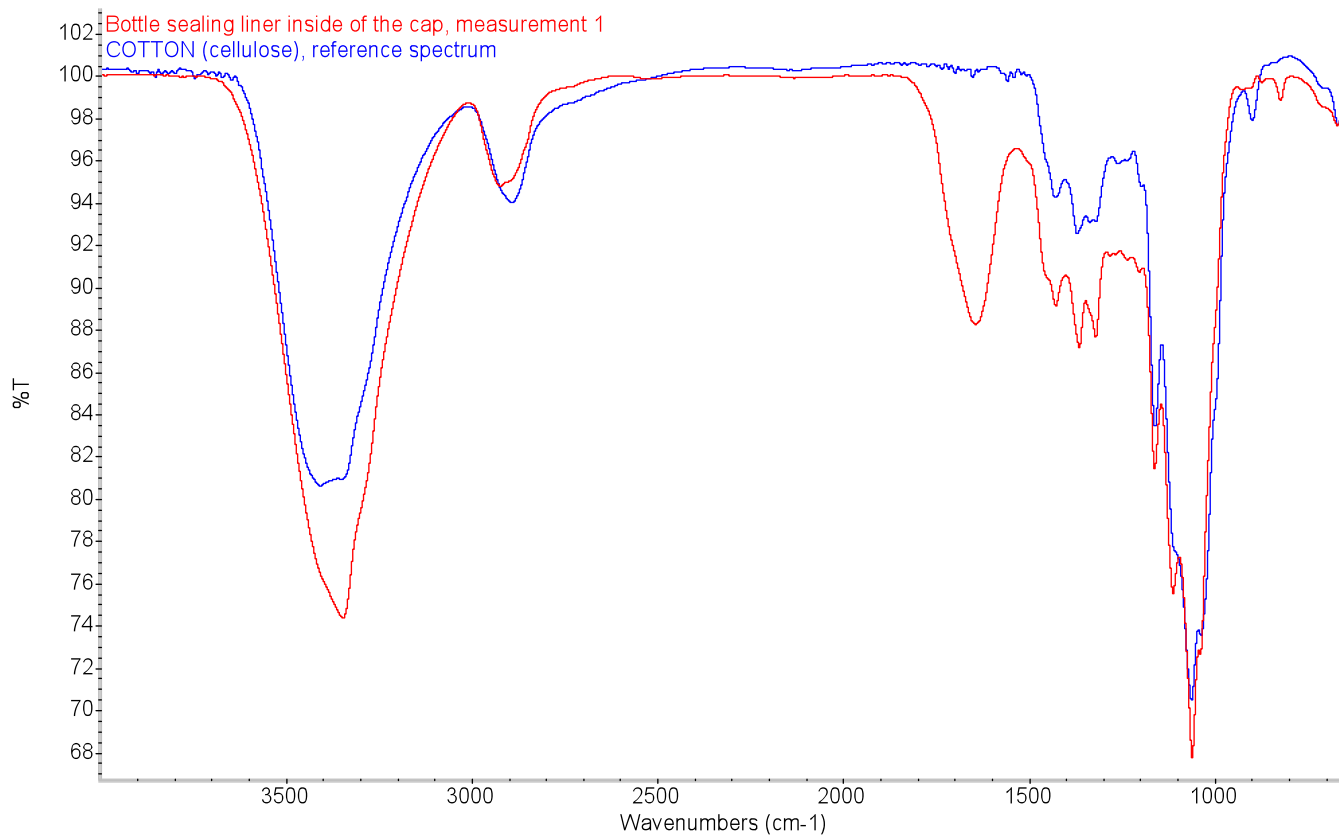




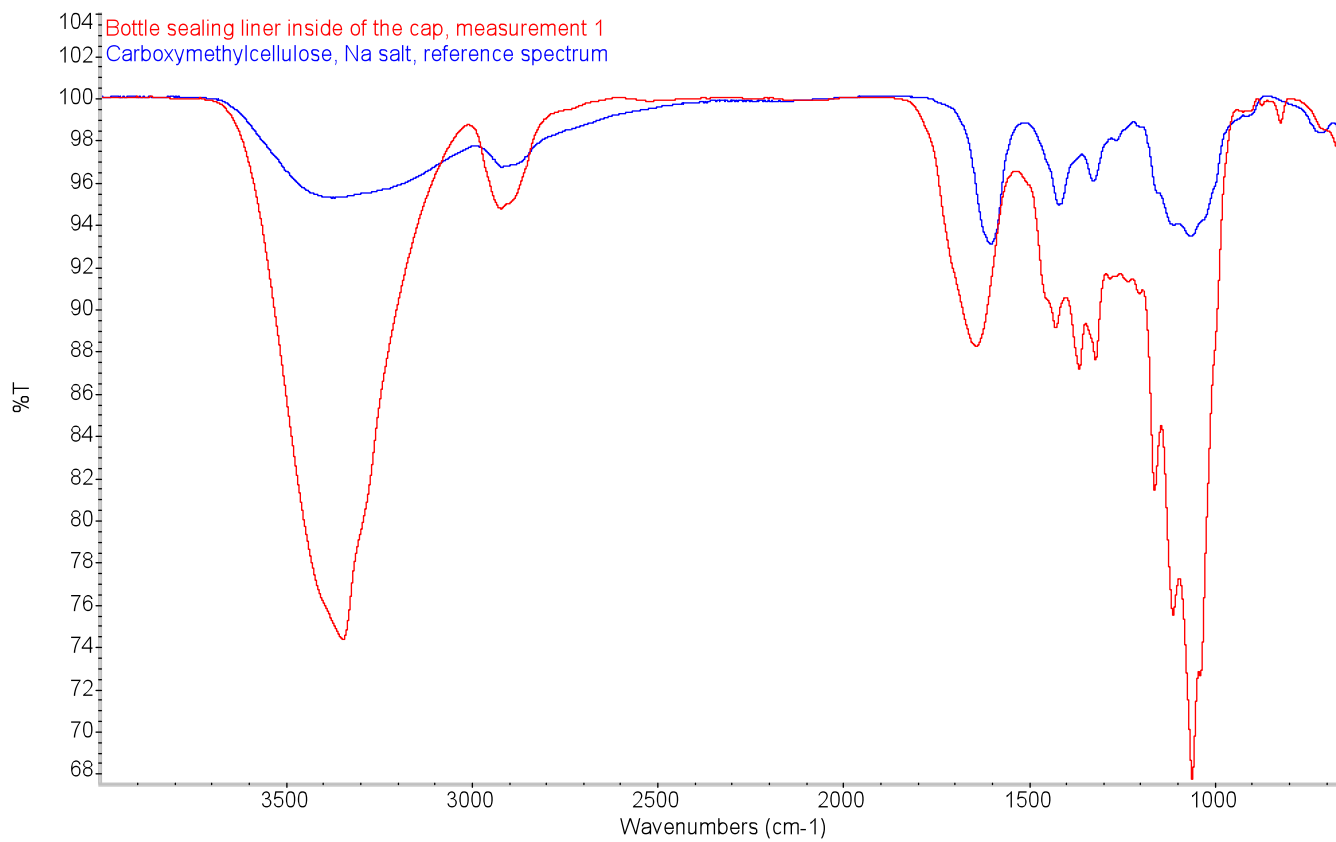
Spectrum 1



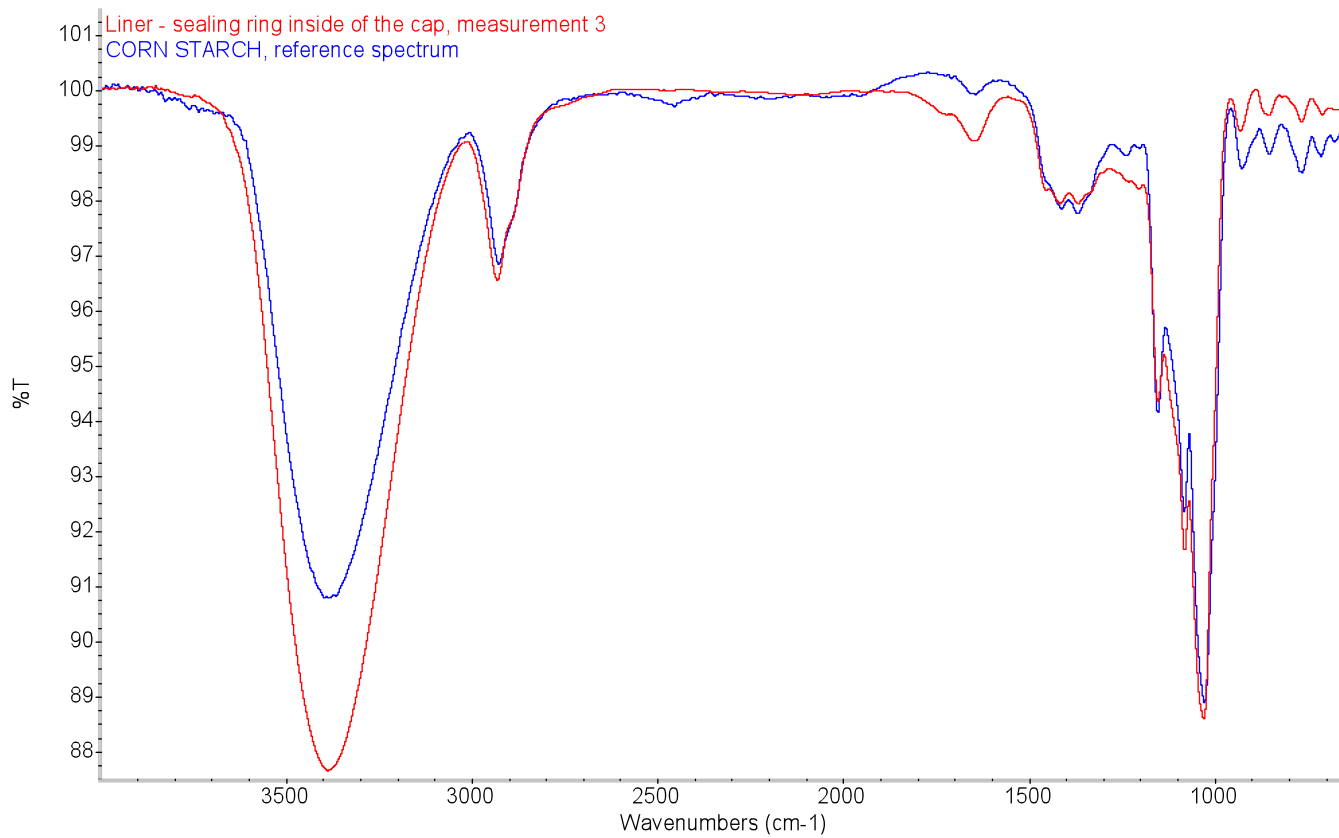
Spectrum 2



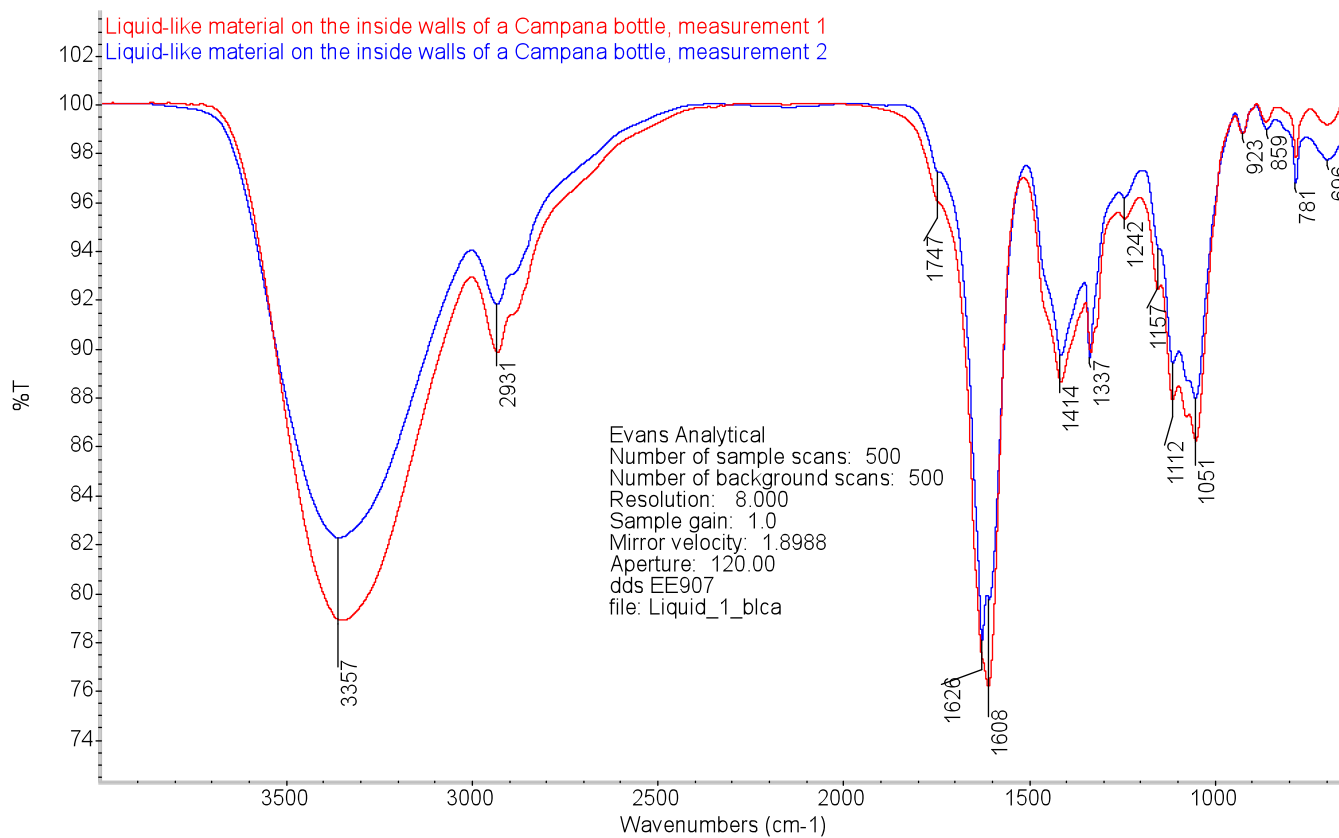
Spectrum 3



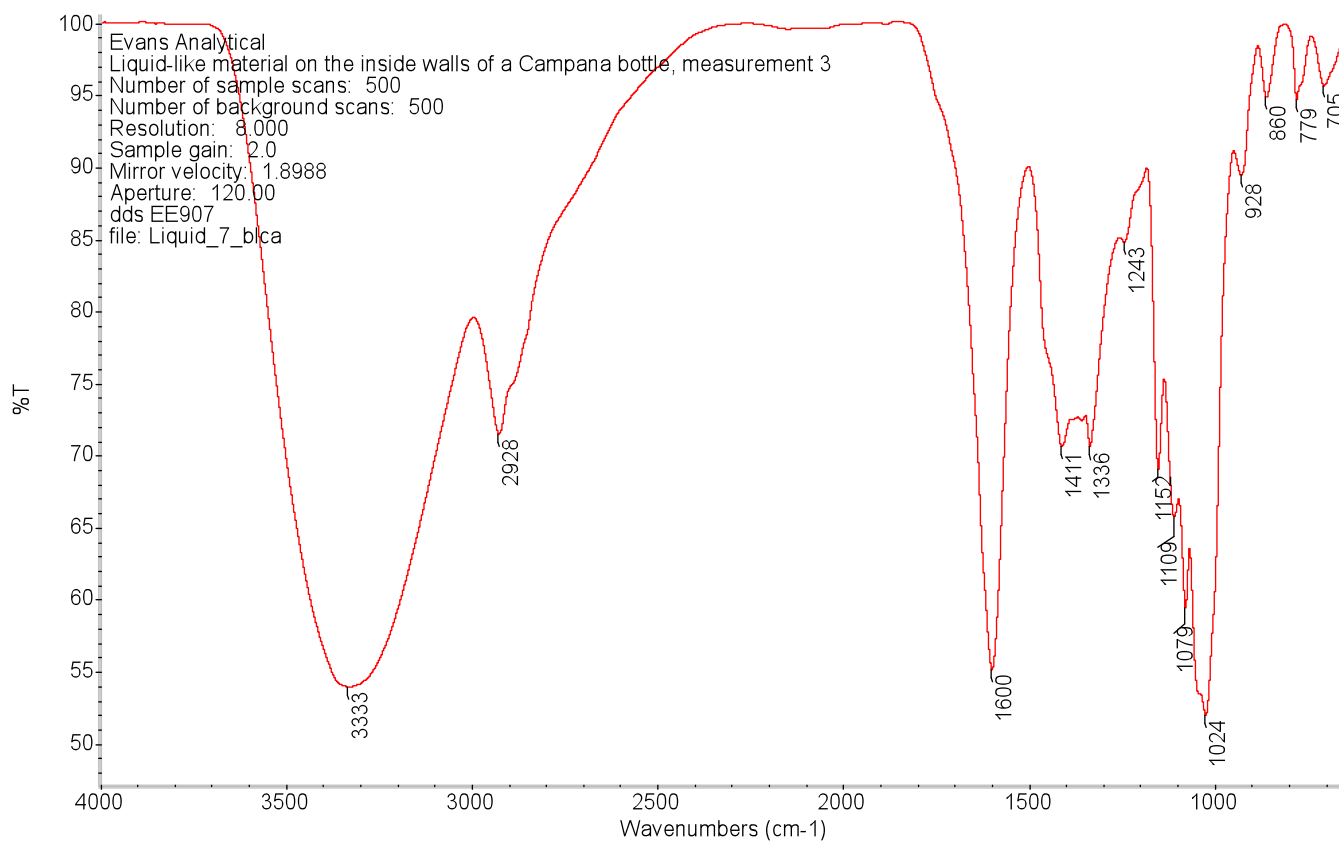
Spectrum 4



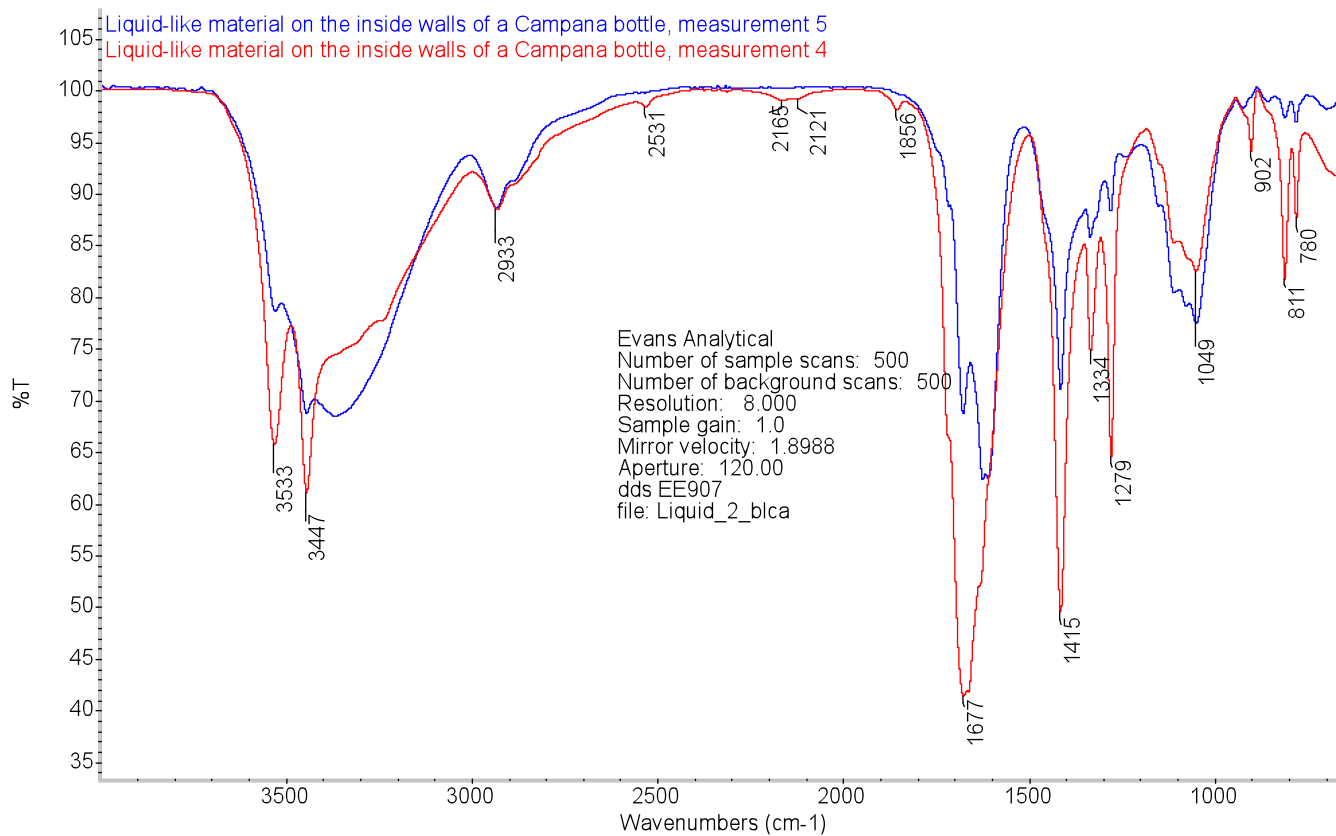
Spectrum 5



Spectrum 6

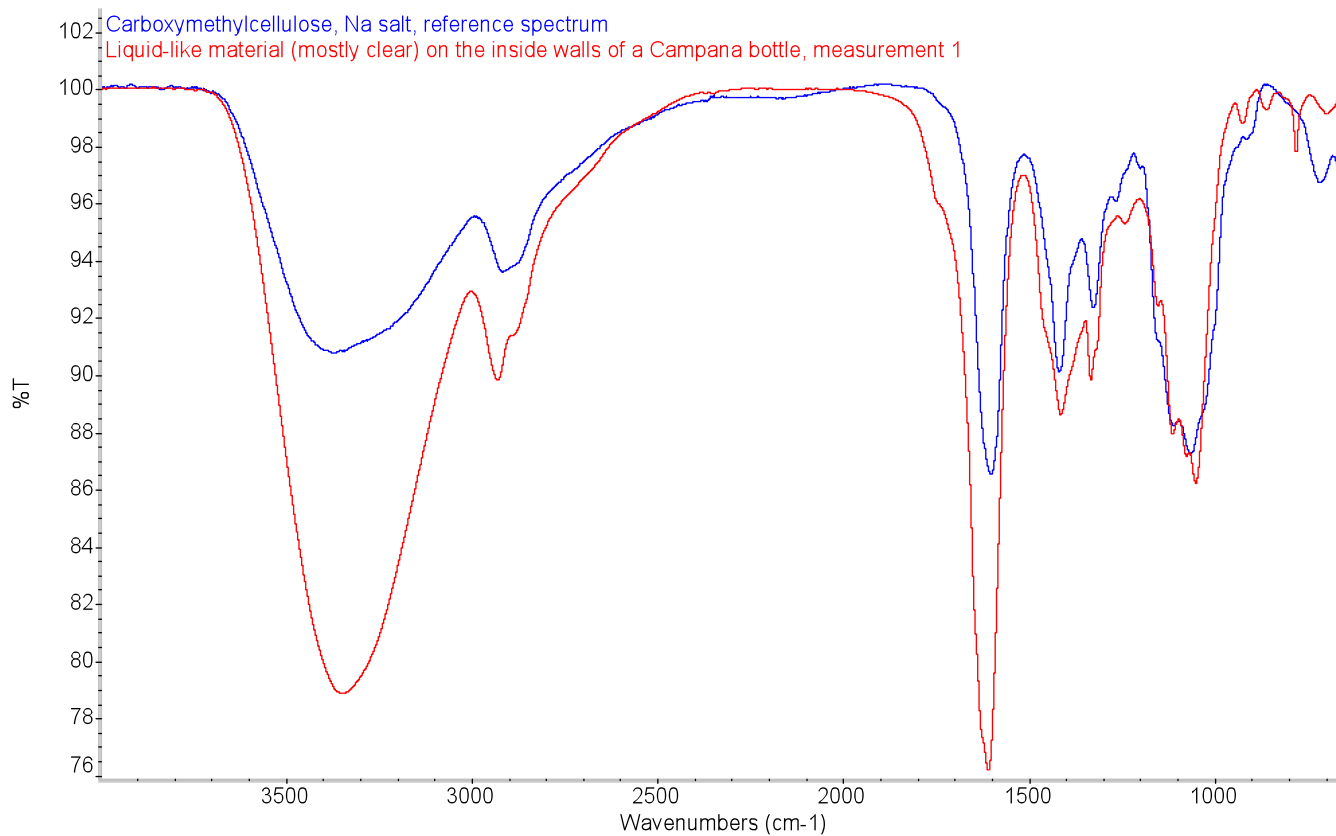


Spectrum 7

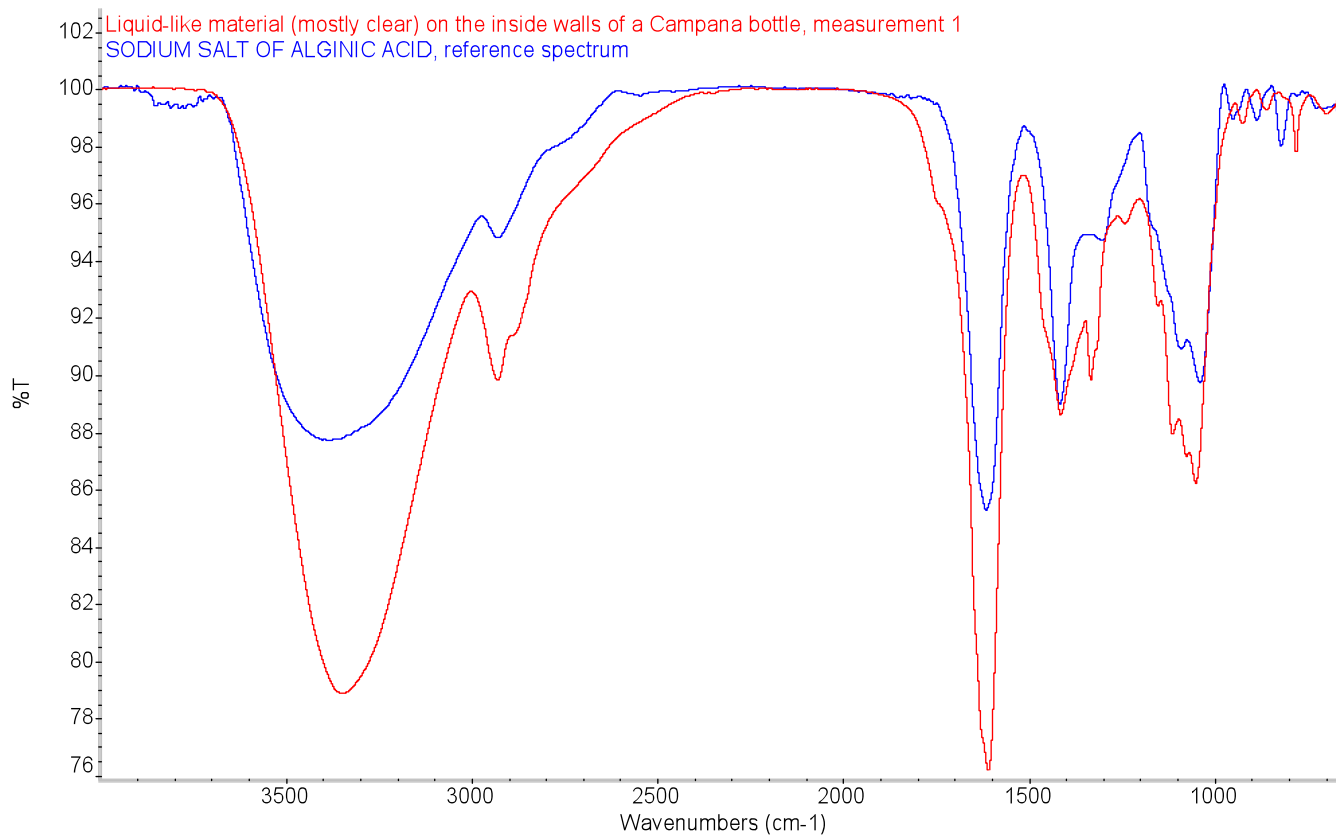


Spectrum 8

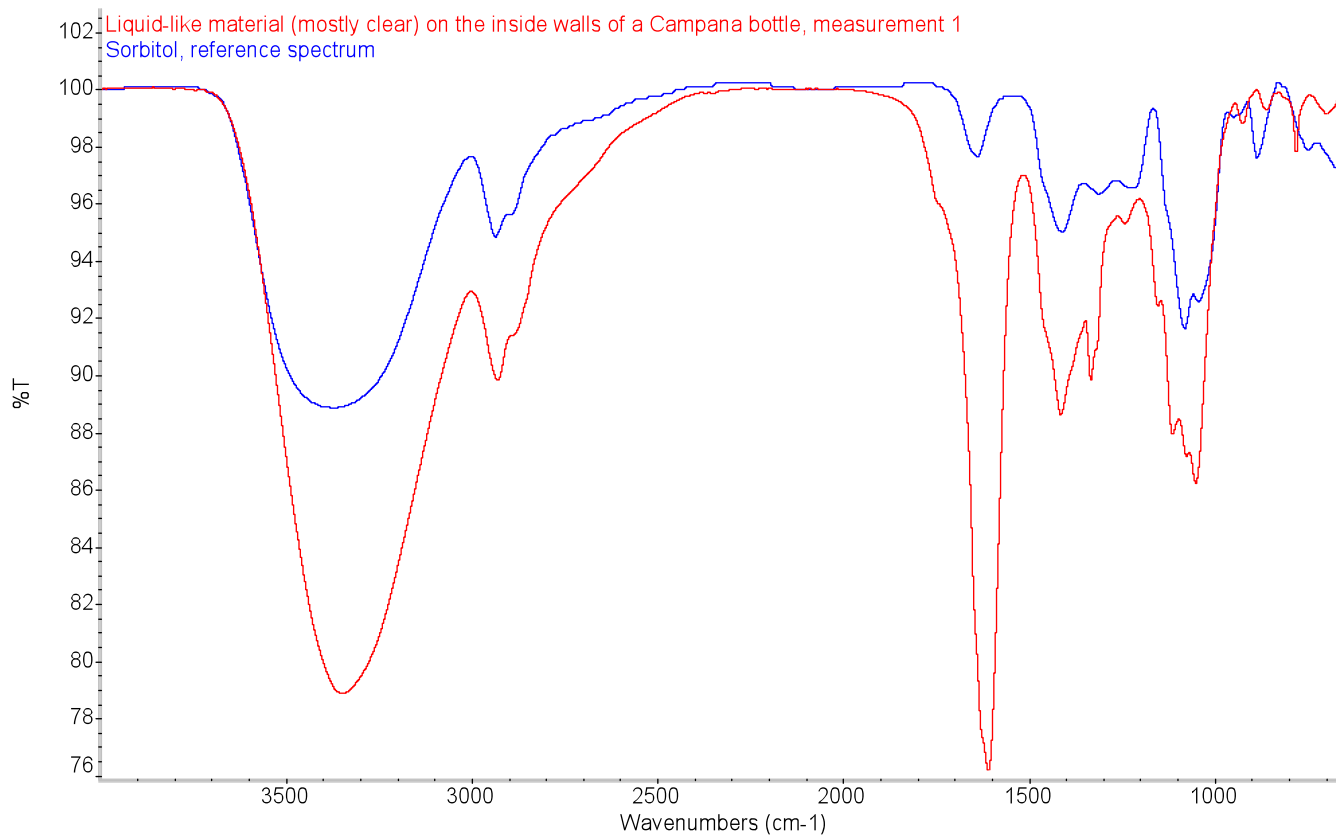




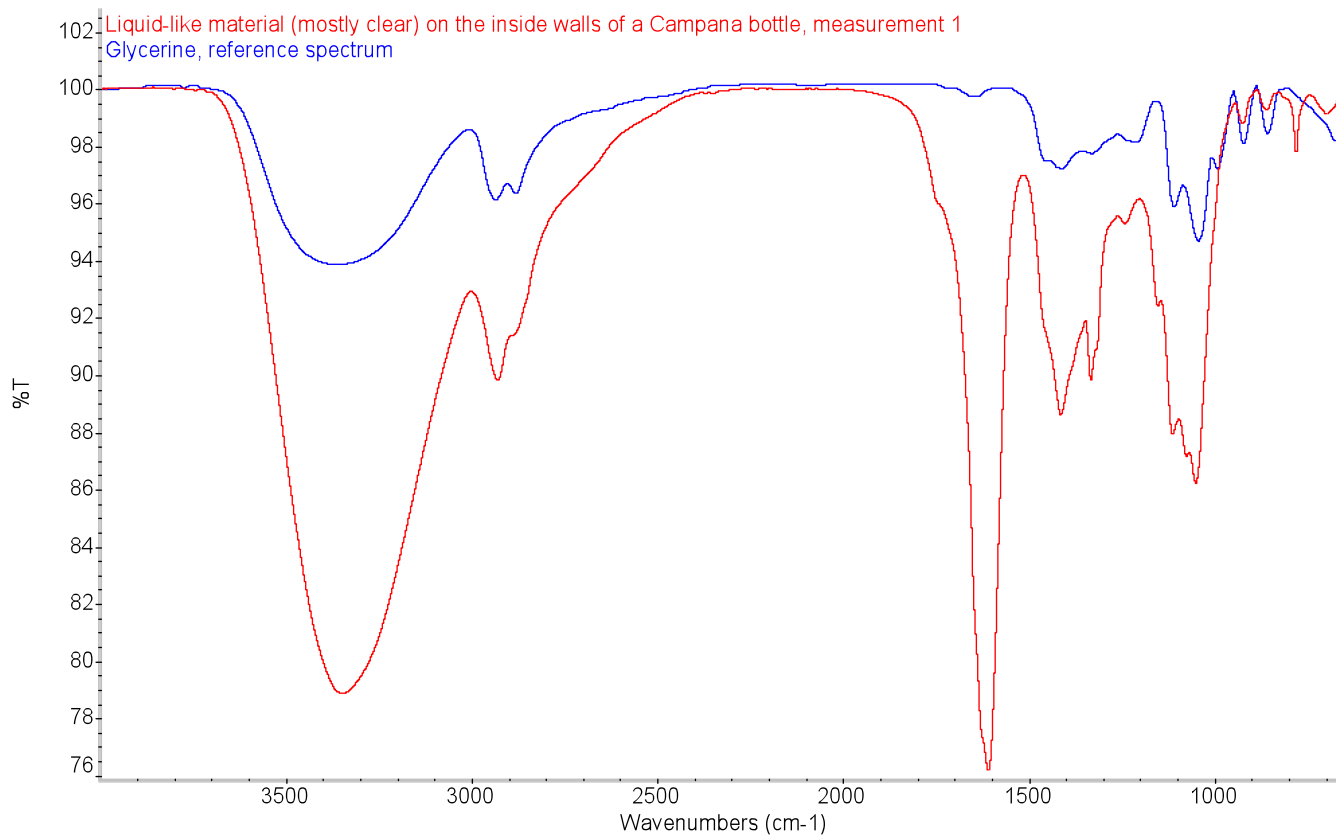
Spectrum 9



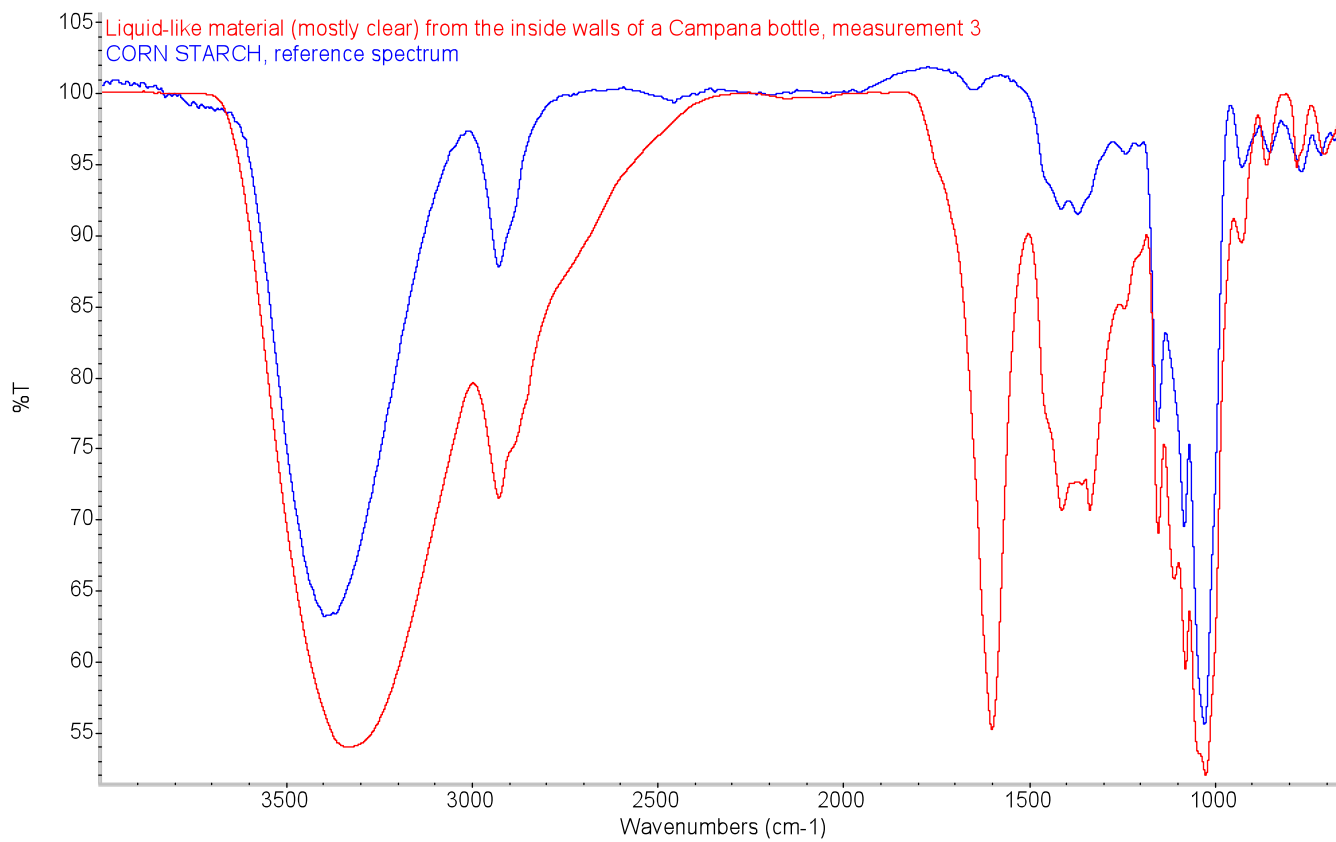
Spectrum 10



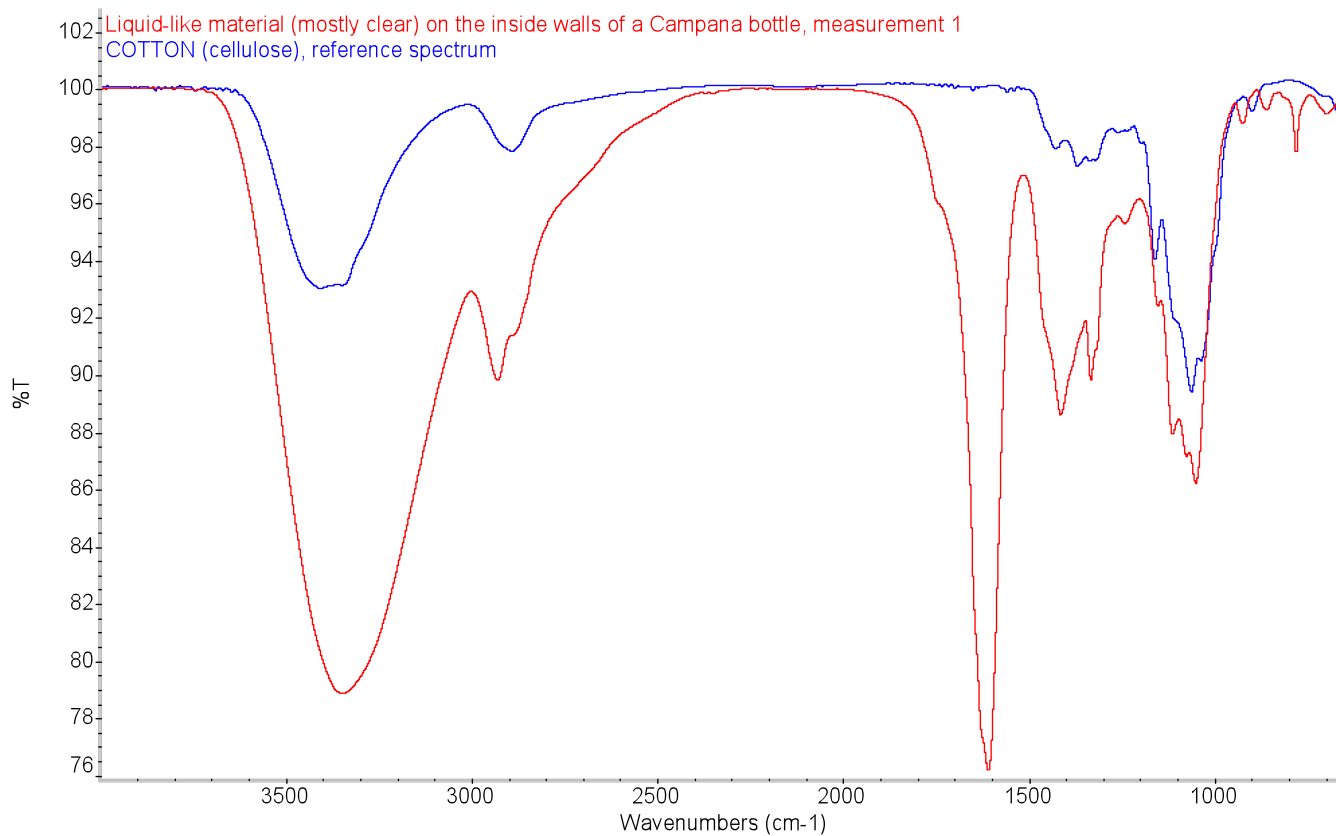
Spectrum 11



Spectrum 12

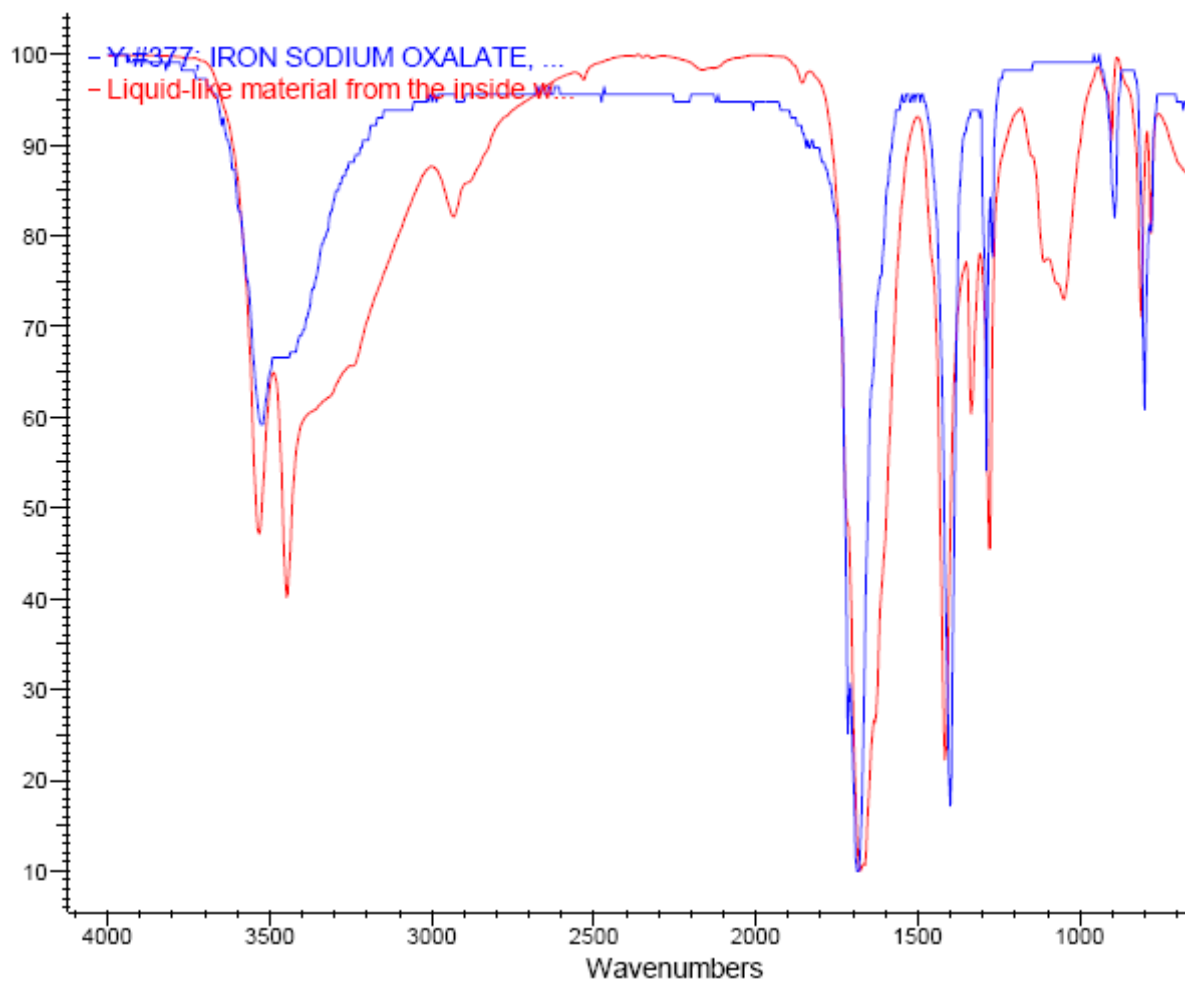


Spectrum 13



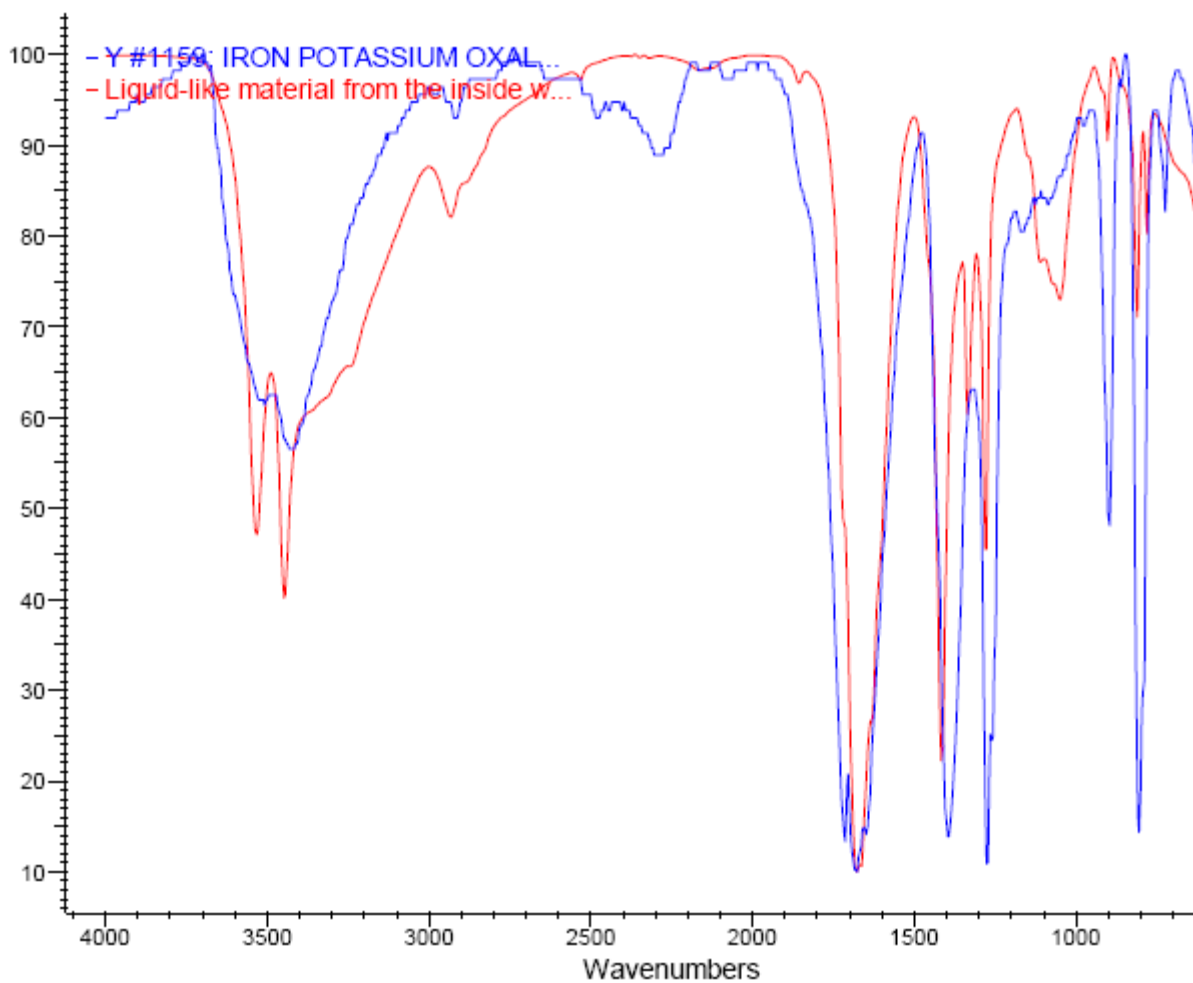
Spectrum 14

**Reference (blue trace) is iron sodium oxalate, hydrate**



Spectrum 15

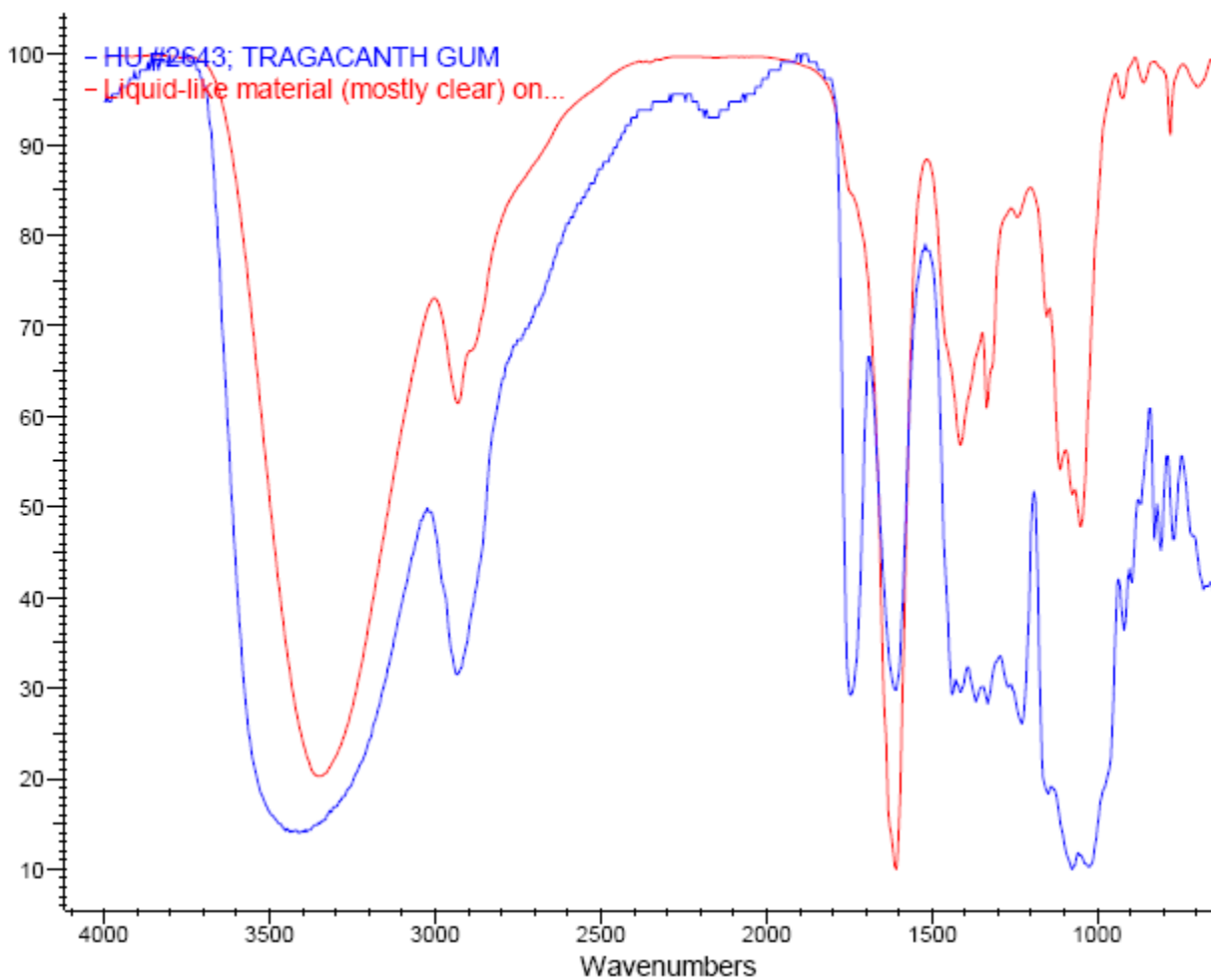
**Reference (blue trace) is iron potassium oxalate, trihydrate**



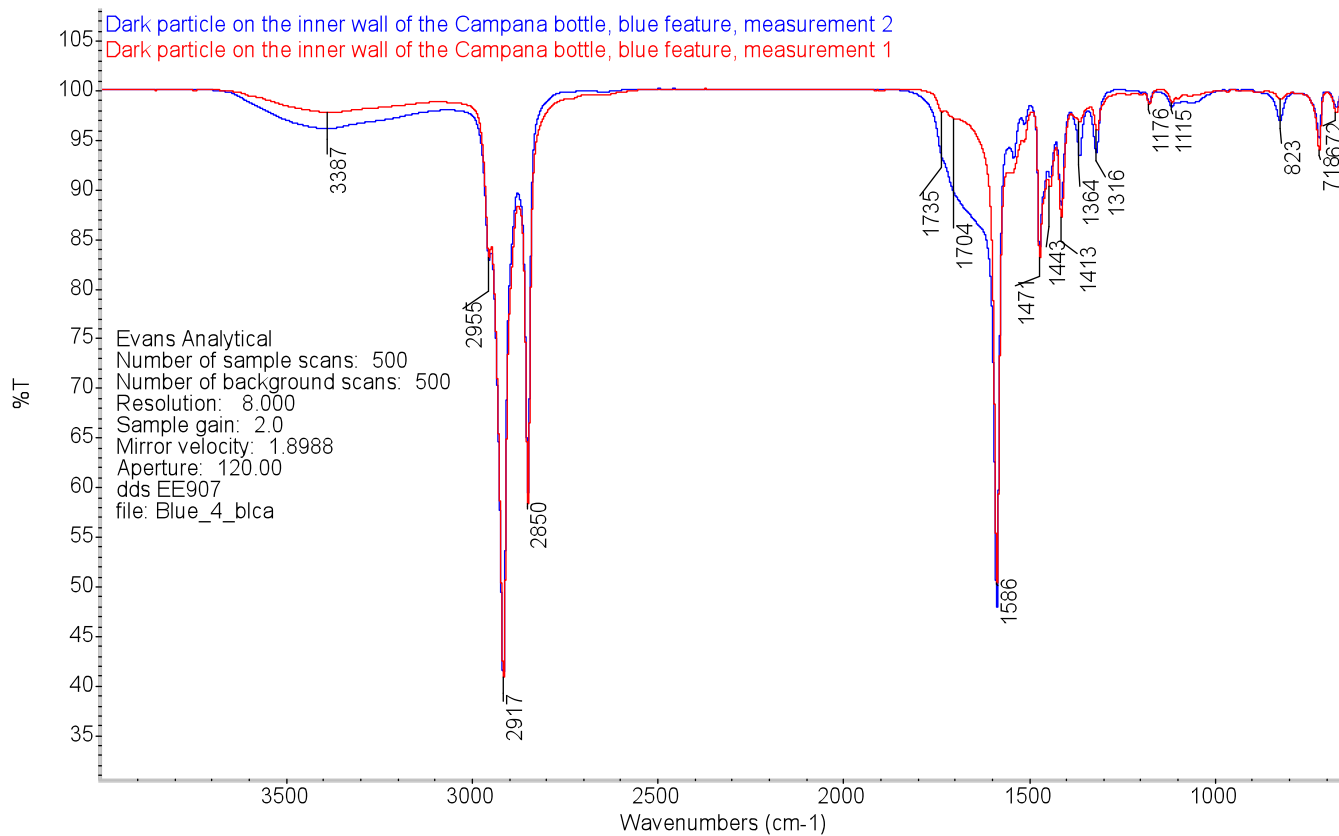
Spectrum 16



**Reference (blue trace) is the dried sap of Astragalus species**

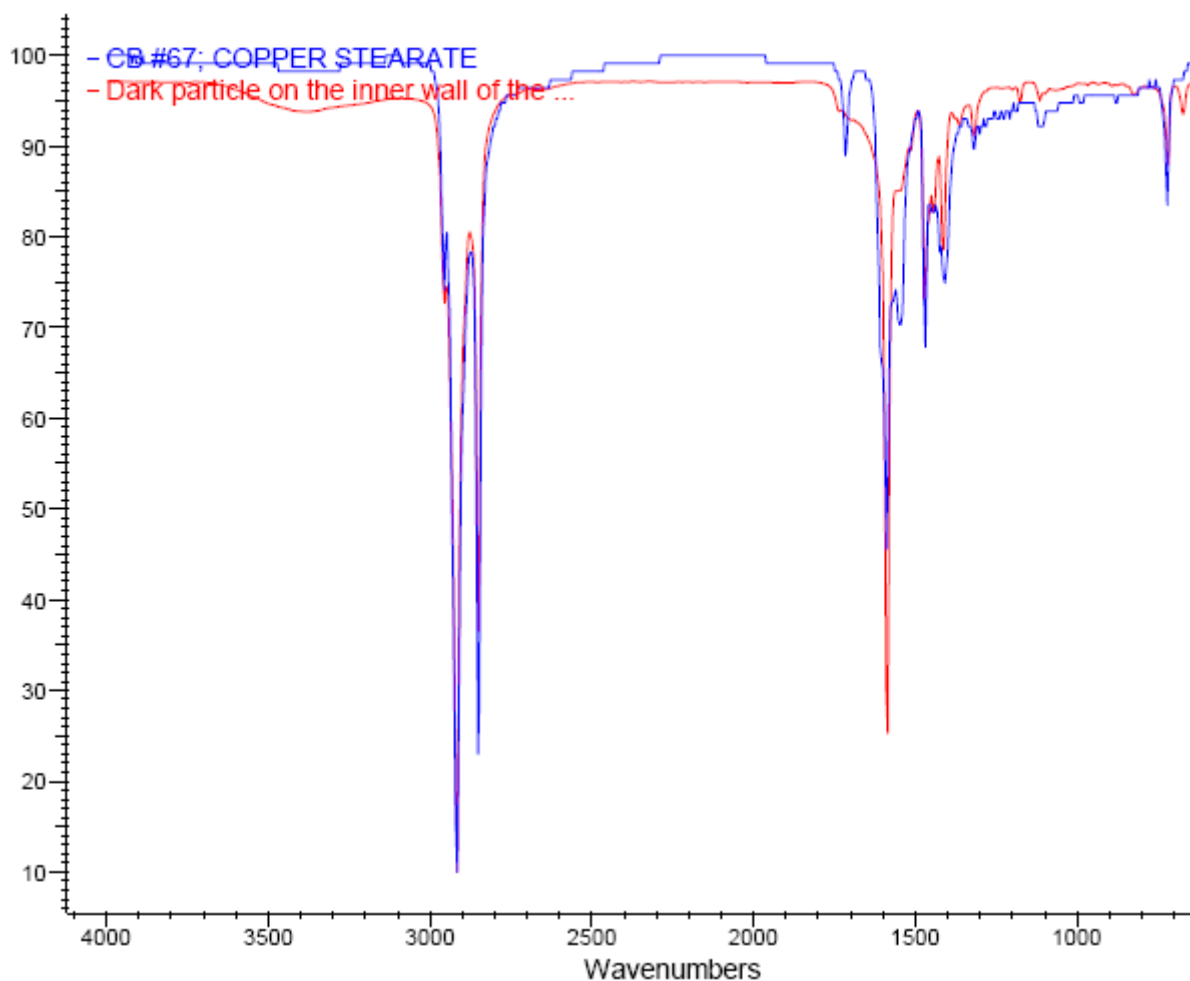


Spectrum 17

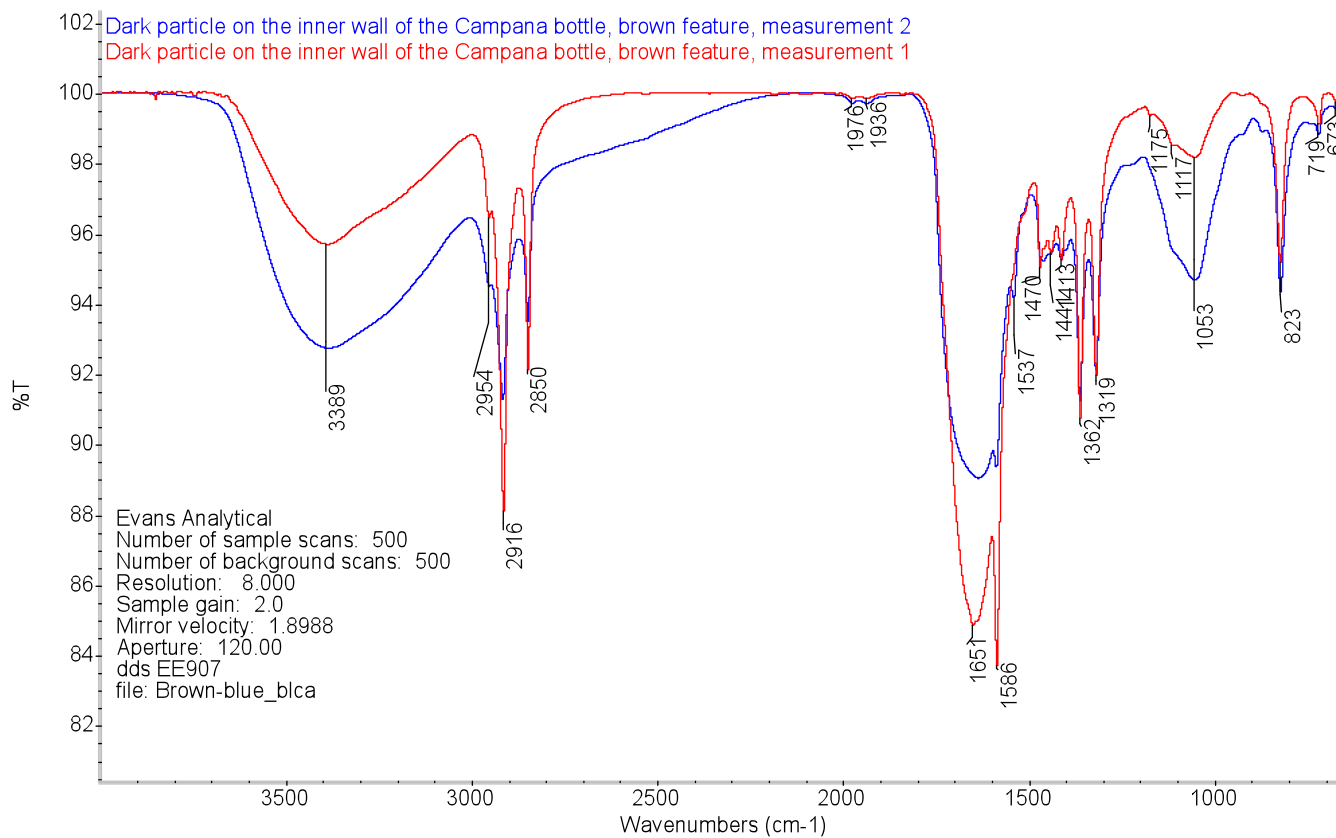


Spectrum 18

**Reference (blue trace) is copper stearate**

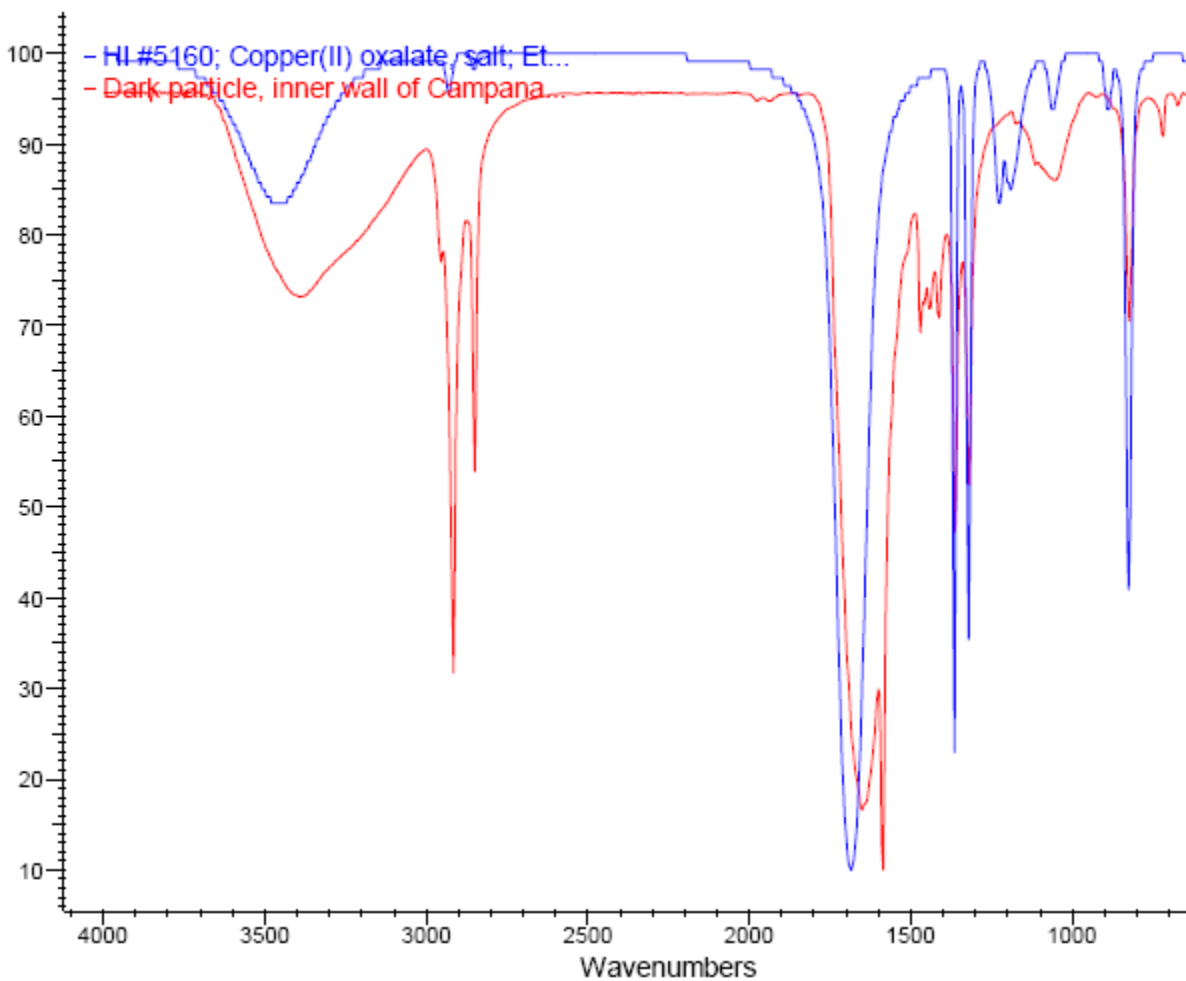


Spectrum 19



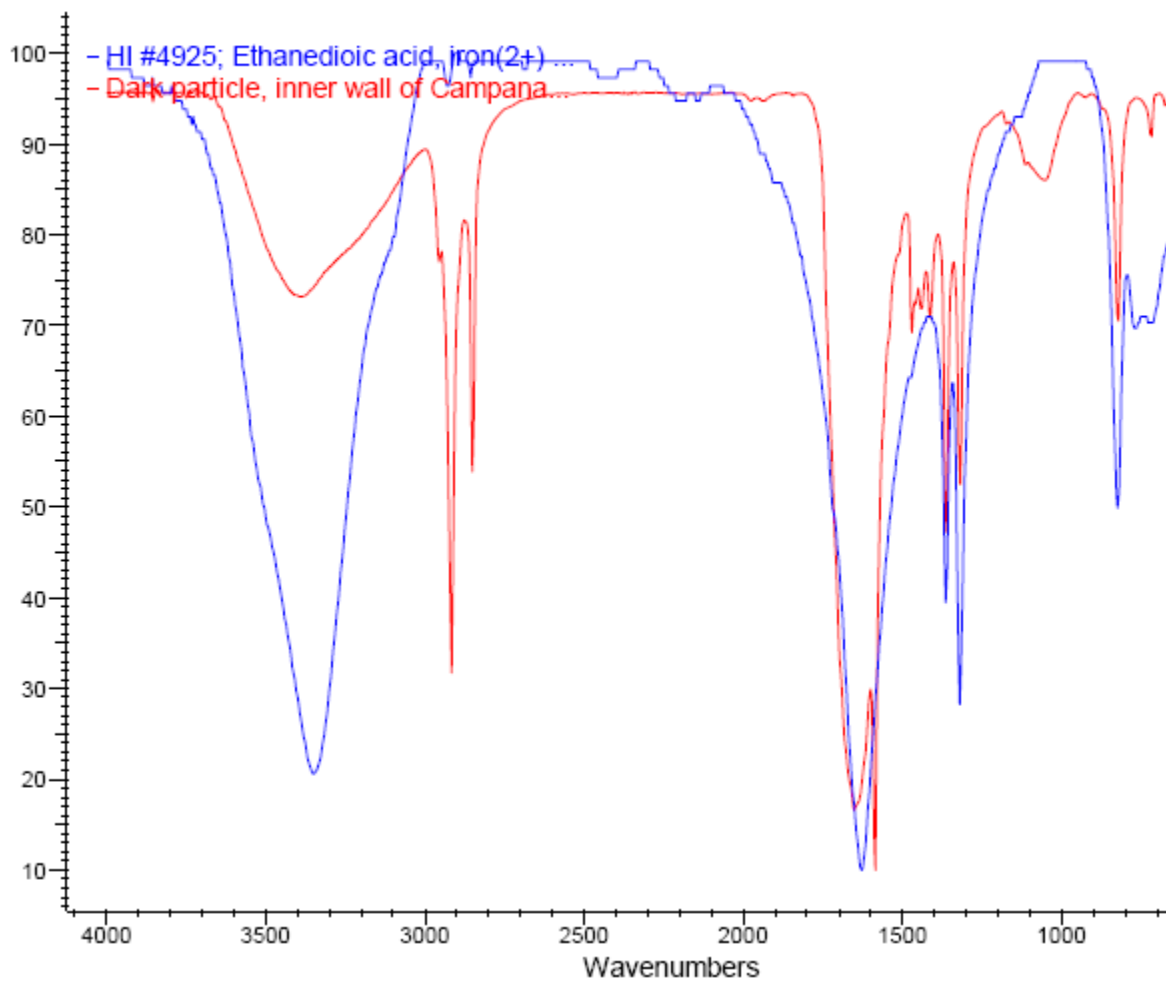
Spectrum 20

**Reference (blue trace) is copper(II) oxalate**



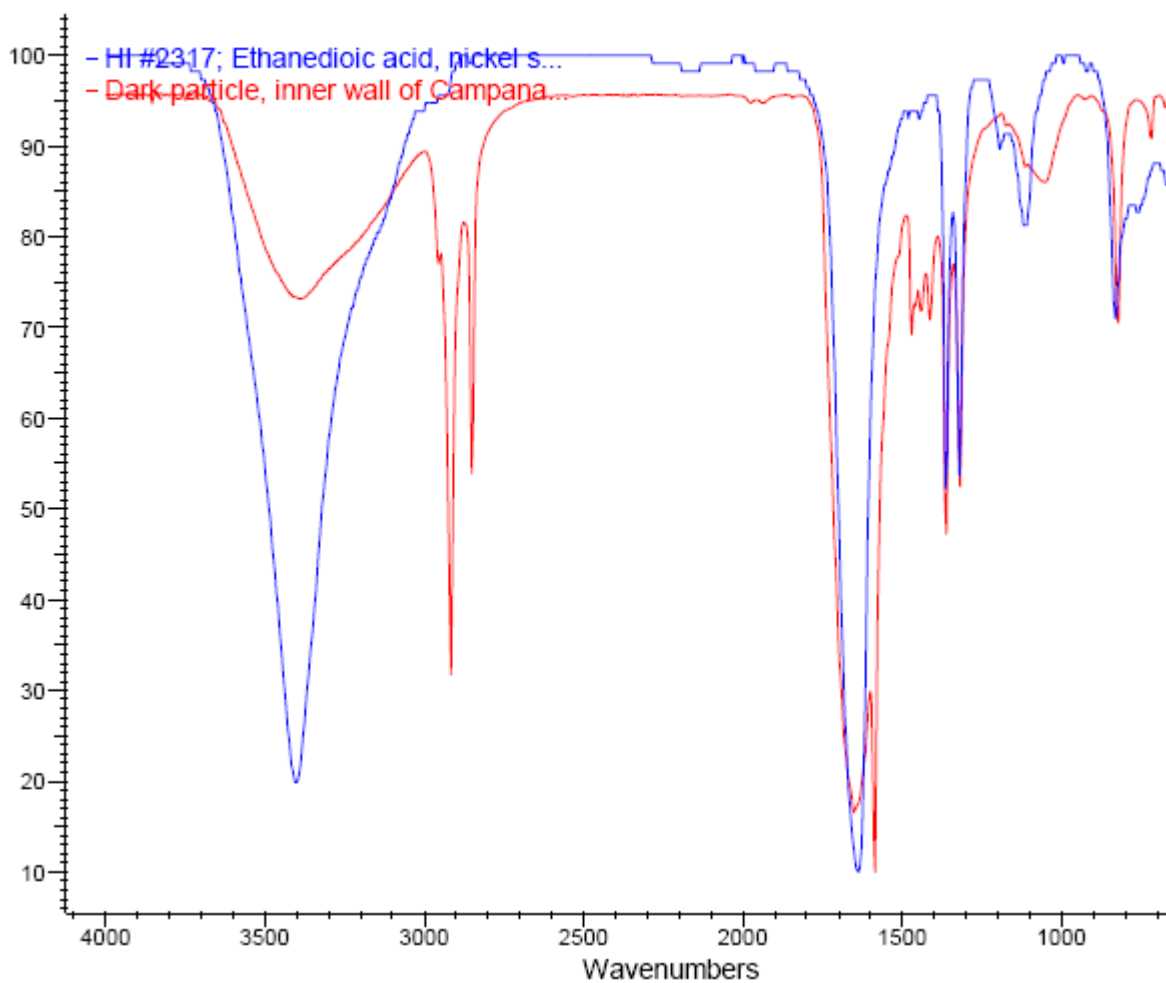
Spectrum 21

Reference (blue trace) is iron (II) oxalate



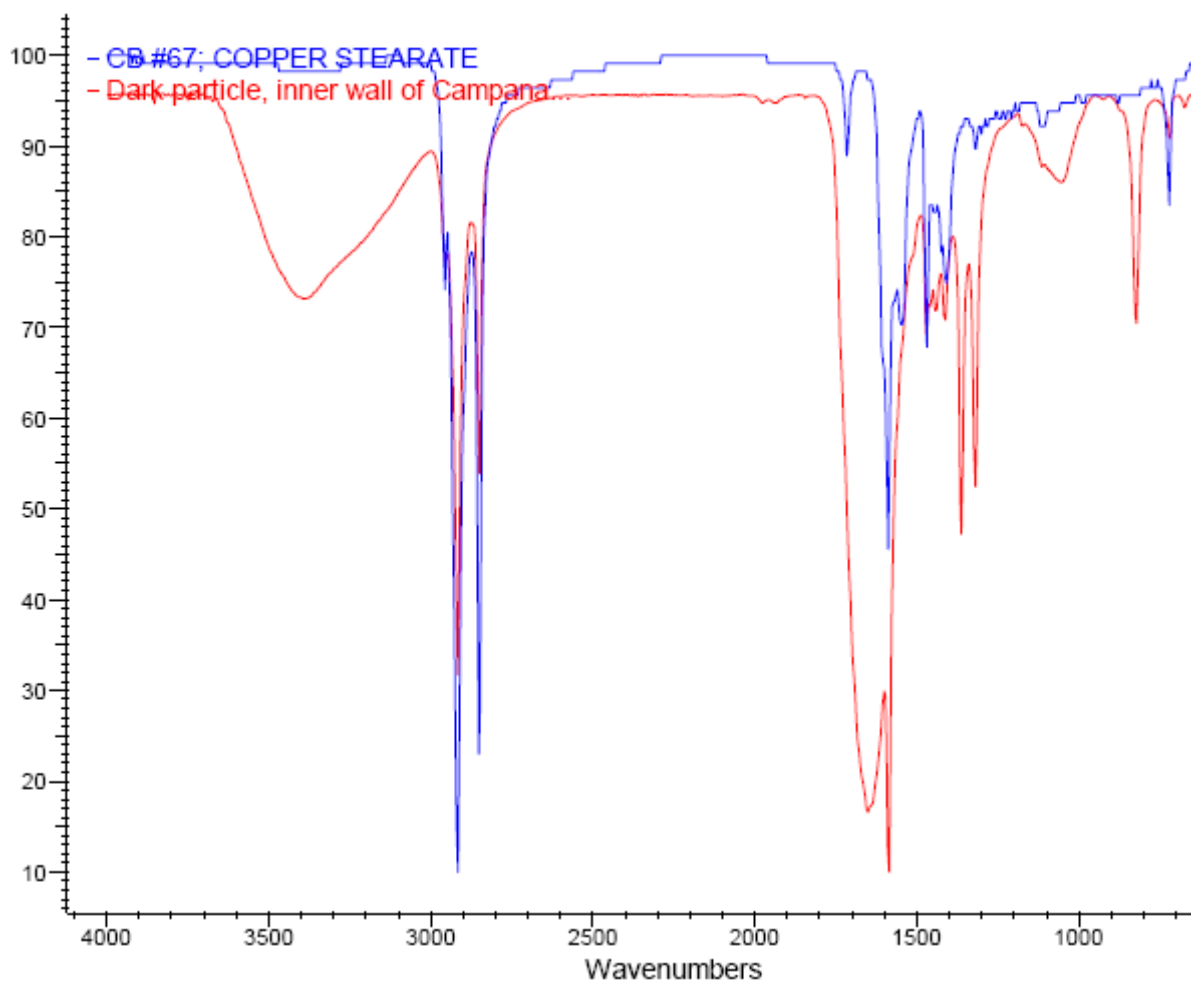
Spectrum 22

**Reference (blue trace) is nickel oxalate**



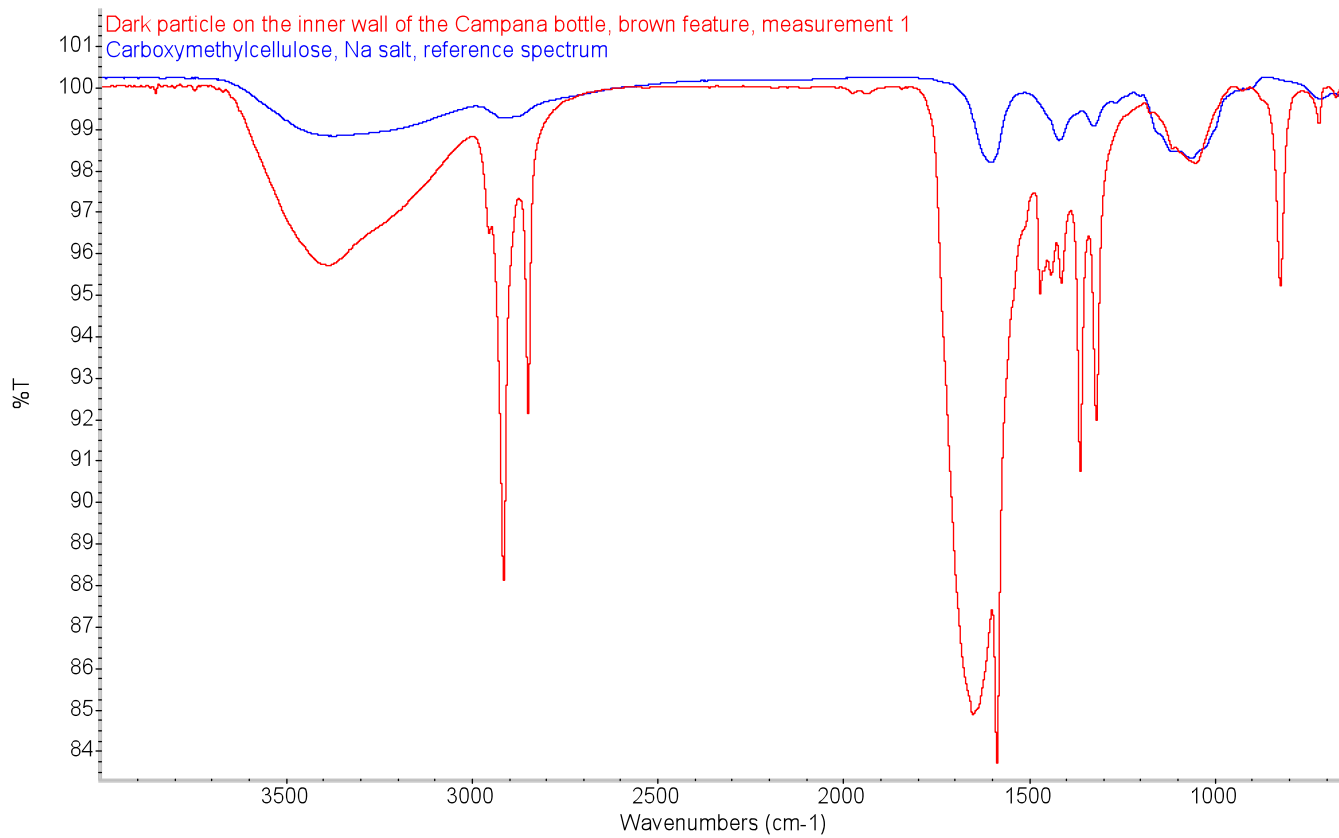
Spectrum 23

**Reference (blue trace) is copper stearate**

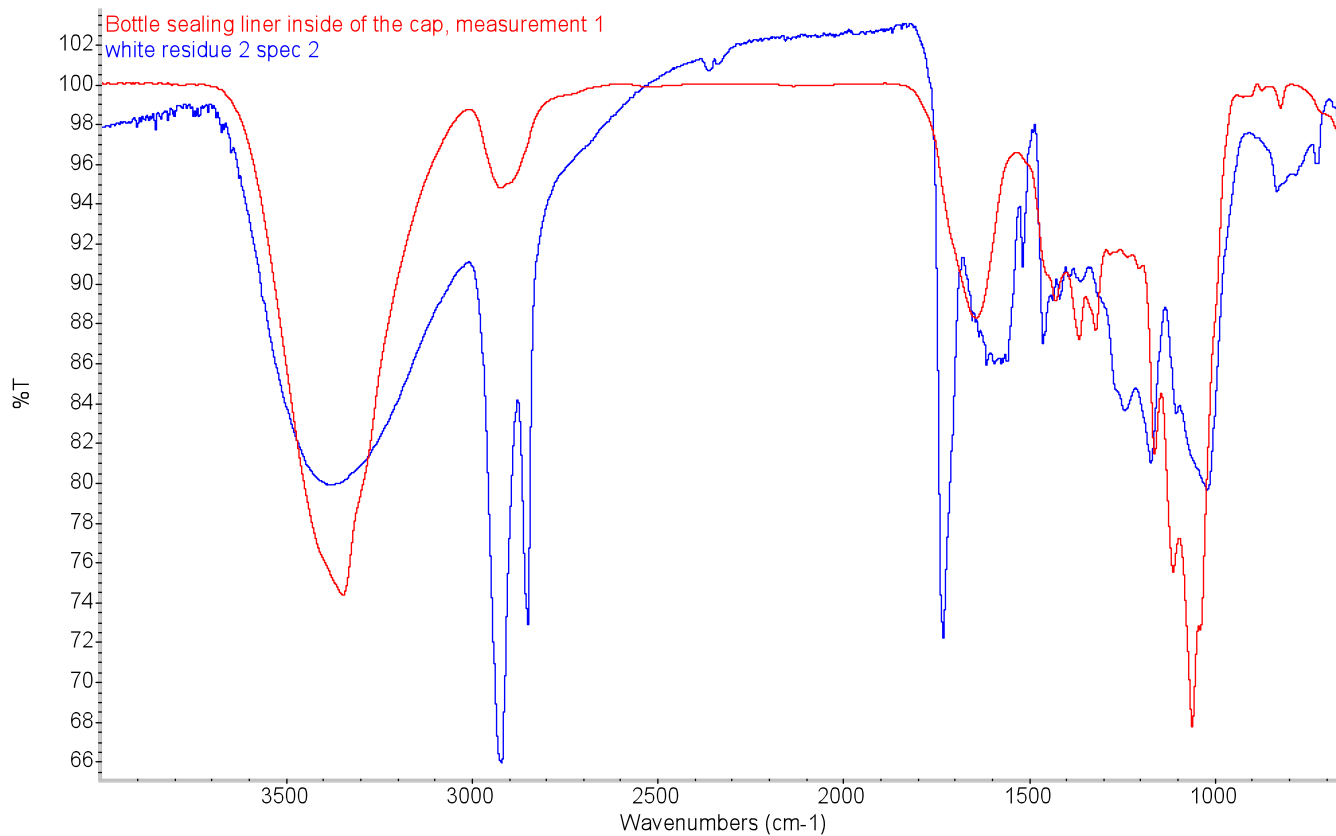


Spectrum 24

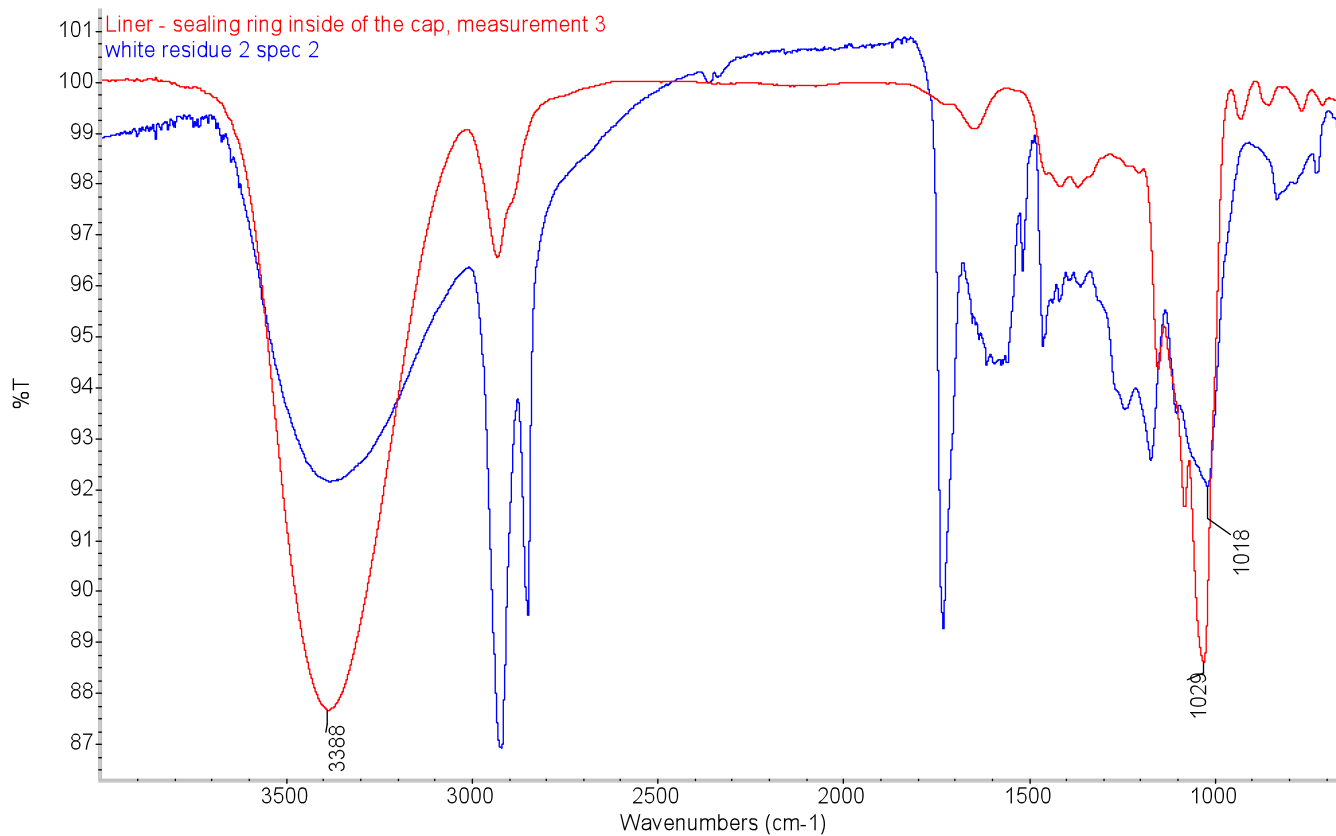




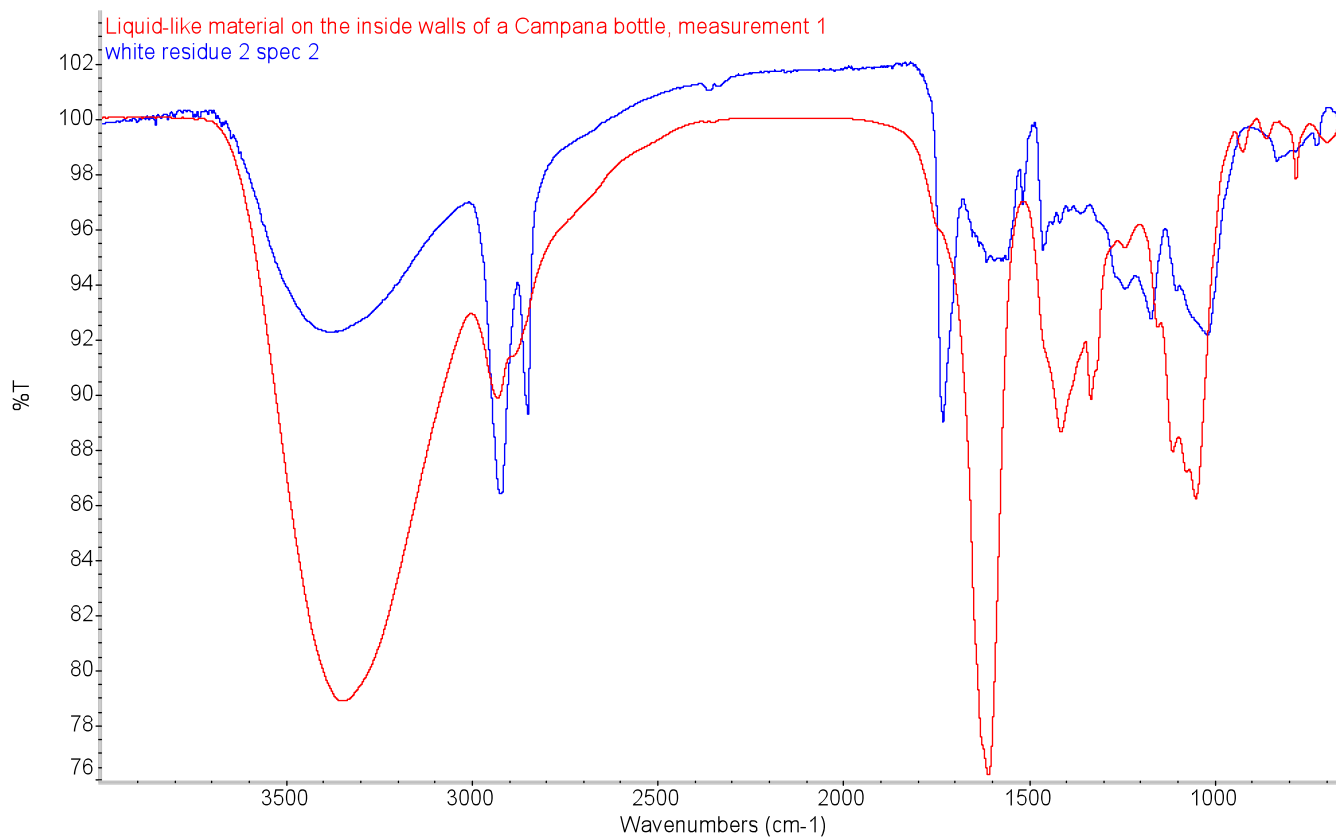
Spectrum 25



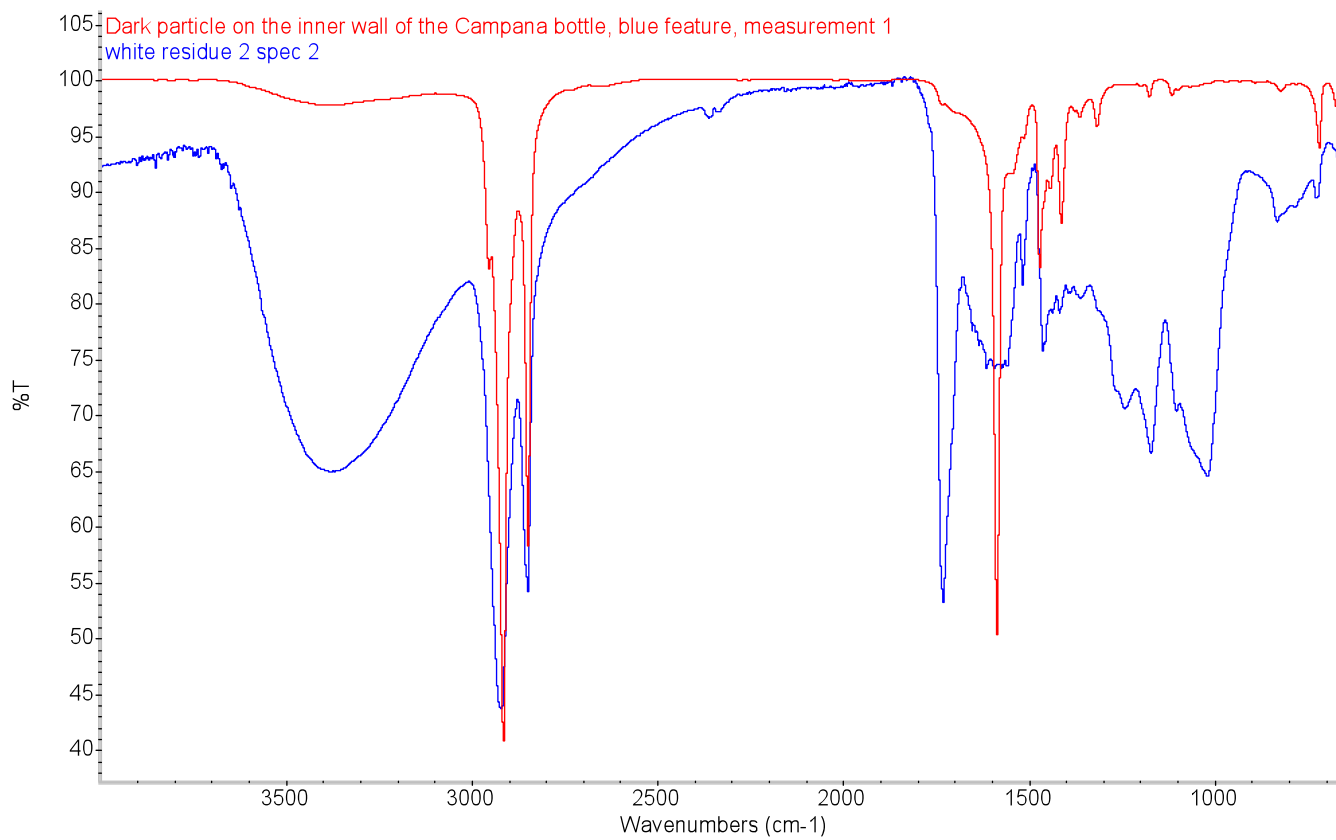
Spectrum 26



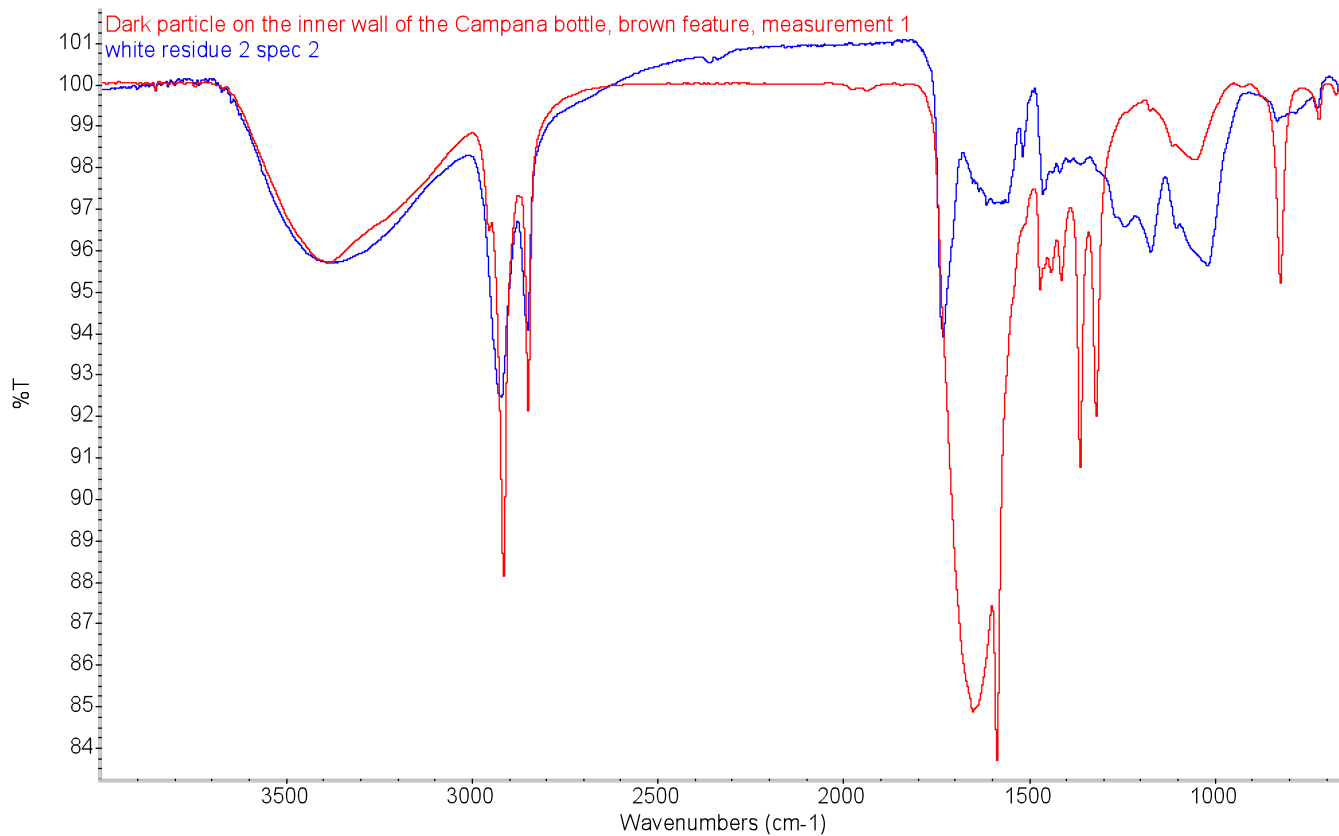
Spectrum 27



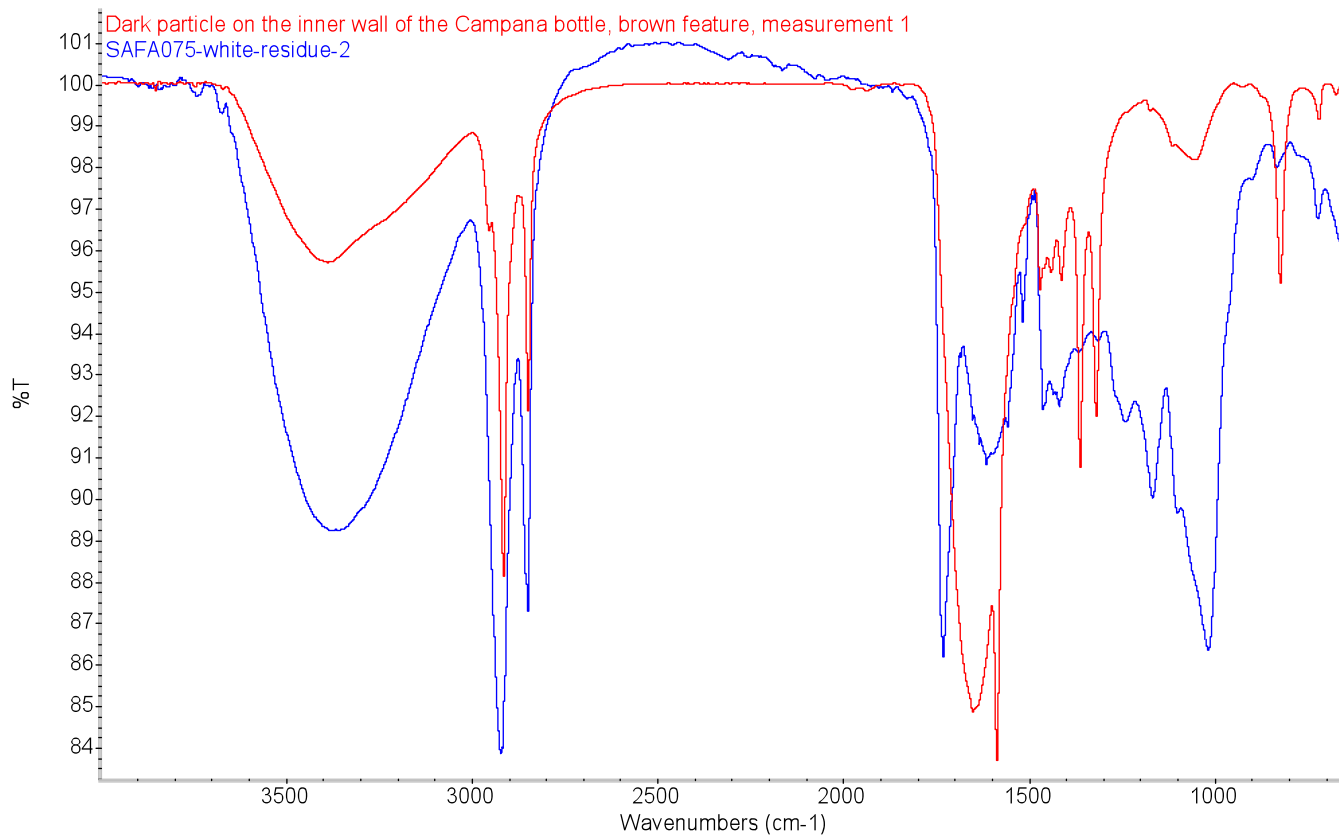
Spectrum 28



Spectrum 29



Spectrum 30



Spectrum 31