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ANALYSIS REPORT

REPORT NO.: SAFA076 **DATE:** 9/24/2007

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OBJECT DESCRIPTION (form, material, color, etc): Archaeological glass fragments excavated from the Republic of Kiribati in the summer of 2007: two beveled glass fragments and one bottle glass fragment containing two white residues/accretions – one with a flakey morphology on the center of the bottle bottom, and a sticky residue in the corner of the fragment.

REASON FOR ANALYSIS: Could these objects have an early twentieth-century American provenance? Could they have been manufactured prior to 7/2/37? Specific questions: Were the beveled glass fragments silvered suggesting that they were part of a mirror such as a compact mirror? The silvering could be due to elemental silver or aluminum. Was the bottle used to hold hair tonic, skin lotion, or some other material? The bottle is of the type made in Owens Illinois in 1933.

SAMPLING: Milligram sized samples were removed from the glass surfaces for scanning electron microsocopy using a diamond scribe. A size 11 scalpel blade was used to remove white residue/accretion samples from the glass bottle fragment for Fourier transform infrared spectroscopy.

ANALYSIS PROTOCOL: Fourier transform infrared spectroscopy (FTIR) was used to identify the white accretions in the glass bottle. Samples were removed from the glass fragments for scanning electron microscopy/energy-dispersive x-ray microanalysis (SEM-EDS) analysis to allow for elemental analysis of elements heavier than sodium. See Appendix I for experimental details of analyses.

RESULTS and DISCUSSION:

Bottle Residue Analysis: The FTIR spectrum of the sticky white residue/accretion from the corner of the bottle fragment was an excellent match for reference spectra of wild silk (see Figure 1). This finding, along with its fibrous morphology when viewed under the microscope, indicates that this residue is the remains of a caterpillar cocoon or other insect-derived proteinaceous fiber. The FTIR spectrum of the flakey white residue/accretion from the bottom of the bottle appears to contain more than one material. The closest matches to reference spectra included those of an oil such as rapeseed oil or linseed oil and lanolin (see Figure 2). Lanolin, a waxy material extracted from wool, is a common component of skin creams and ointments.

Glass Analysis: The side of the beveled edge glass fragments with the brown accretion was analyzed by SEM-EDS and found to contain silicon, calcium, aluminum, sodium, and magnesium. The aluminum content of this surface was not elevated in a manner that would suggest remnants of an applied aluminum surface to create a mirror (see Figure 3). Similar to the XRF experiments performed in SAFA075, silver was not identified on this surface. The iridescent

surface of the beveled edge glass fragments was found to contain silicon, aluminum, calcium, magnesium, titanium, iron, sulfur, and potassium (see Figure 4), with the major elements being calcium and phosphorus. Calcium phosphate and calcium sulfate crusts are known to form on weathered glass surfaces.1 This surface also did not appear to be enriched in aluminum beyond the amount typically observed in glasses (on the order of 2-3 weight percent) nor was silver detected.

CONCLUSIONS: The glass bottle contains a white residue that is a mixture of several compounds, including an oil and lanolin. This is consistent with its having been used as a lotion or skin cream bottle. No residues of aluminum- or silver-based mirroring were observed on the two beveled glass fragments.

APPENDIX I. EXPERIMENTAL:

FTIR analysis:

For FTIR (Fourier transform infrared) analysis, samples were transferred directly to a diamond compression cell half. The samples were rolled flat to transparency with a steel roller and then analyzed using the Thermo-Nicolet Magna 560 infrared bench with the Nic-Plan infrared microscope with MCT-A detector (range 4000-650 cm^{-1}, 120 scans, 4 cm^{-1} resolution). Resulting spectra were interpreted with the aid of commercial and art conservation infrared reference spectral libraries.

SEM-EDS Analysis:

Scanning electron microscopy and energy dispersive x-ray microanalysis were performed using a Topcon ABT 60 electron microscope with an Evex x-ray microanalysis system. A 20 kV beam voltage was used in conjunction with a 22 mm working distance and a 20° sample tilt. Samples for SEM-EDS were mounted on carbon stubs (SPI supplies) using carbon tape but not coated because a variable pressure chamber was used.

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1 R. Newton and S. Davison, Conservation of Glass, Butterworths, London, pp. 139 and 159.

Figure 1

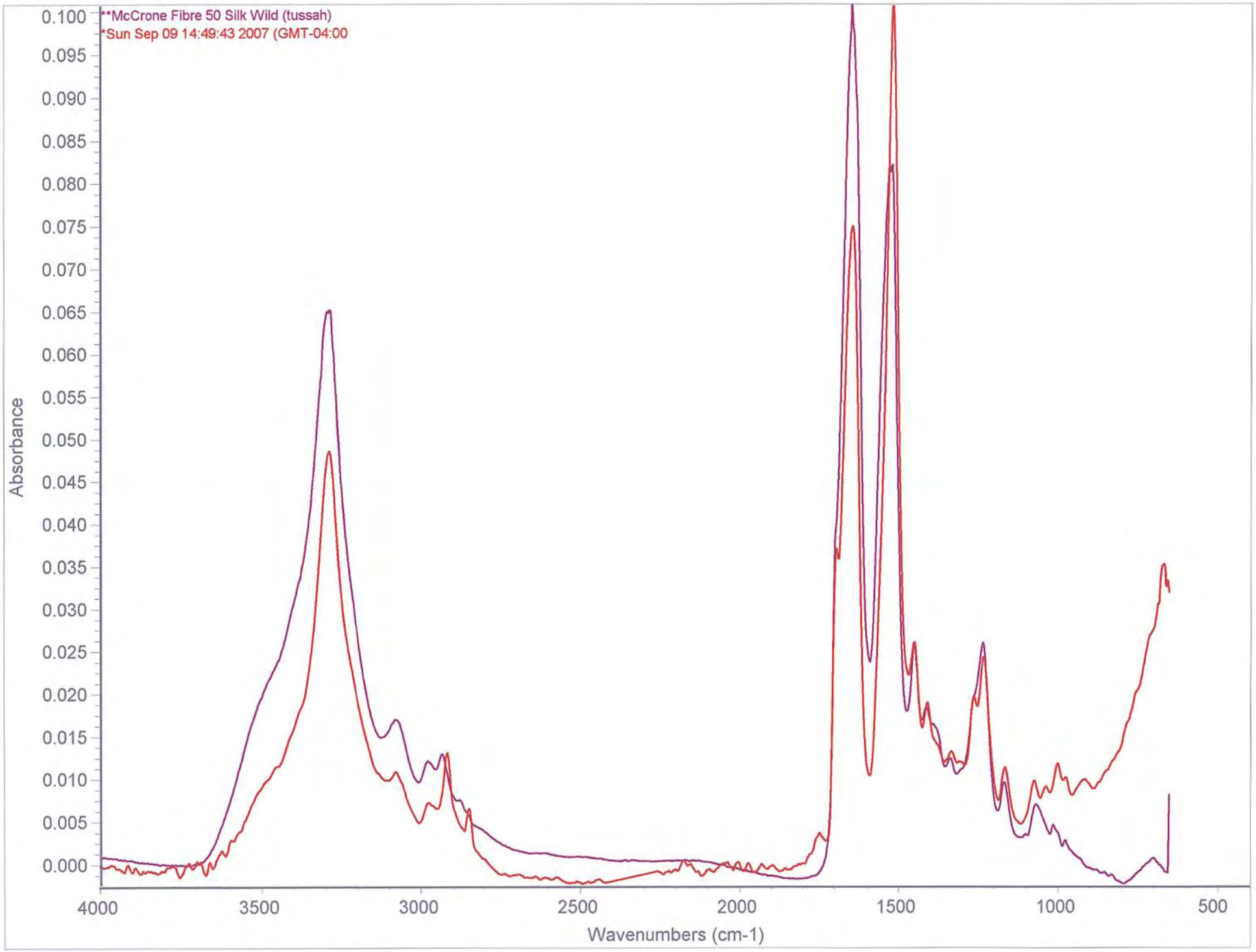
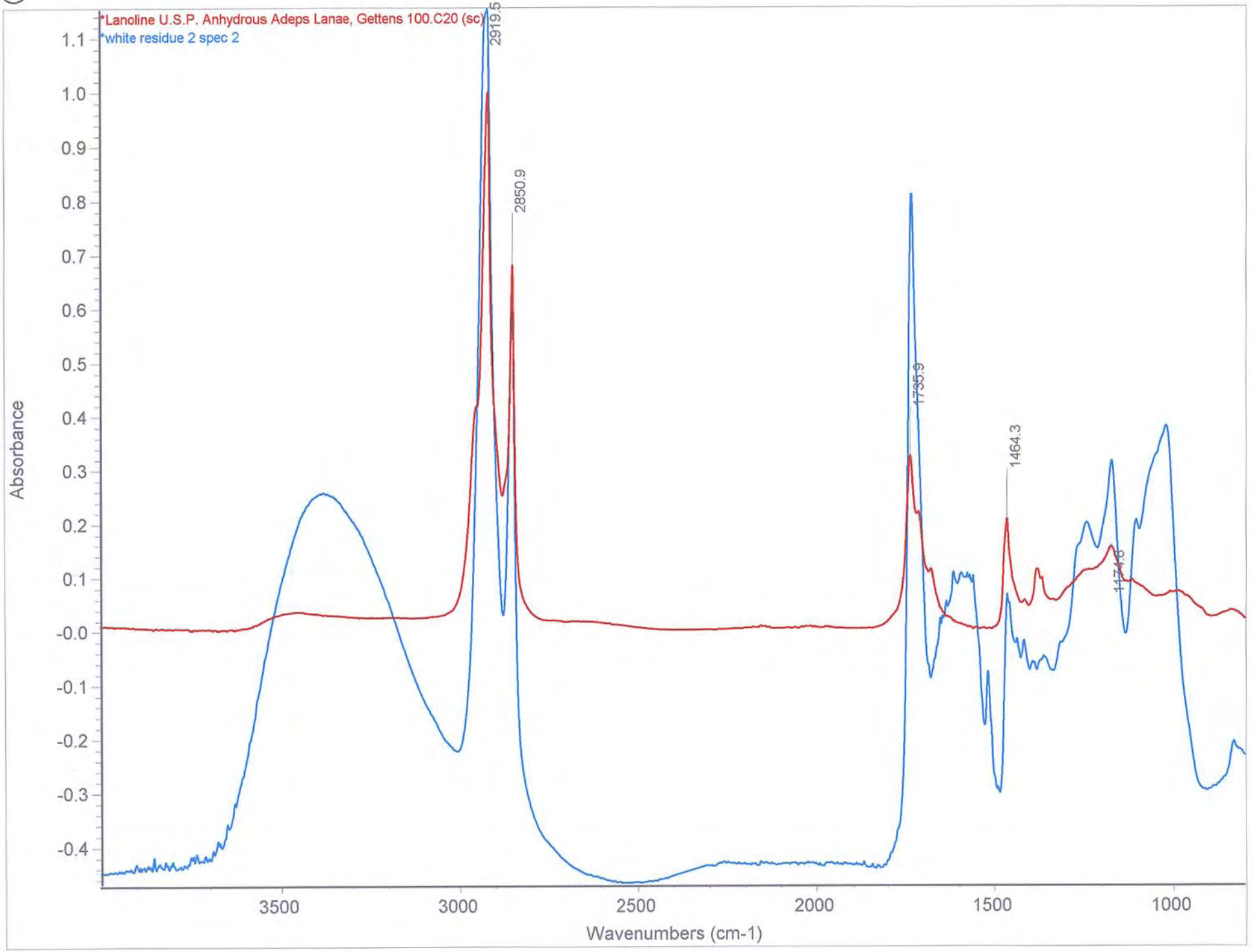

**McCrone Fibre 50 Silk Wild (tussah)
*Sun Sep 09 14:49:43 2007 (GMT-04:00
Absorbance
0.100
0.095
0.090
0.085
0.080
0.075
0.070
0.065
0.060
0.055
0.050
0.045
0.040
0.035
0.030
0.025
0.020
0.015
0.010
0.005
0.000
4000
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)

Figure 2a


*Lanoline U.S.P. Anhydrous Adeps Lanae, Gettens 100.C20 (sc)
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2850.9
1735.9
1464.3
1171.8
Absorbance
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1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
-0.0
-0.1
-0.2
-0.3
-0.4
3500
3000
2500
2000
1500
1000
Wavenumbers (cm-1)

Figure 2b

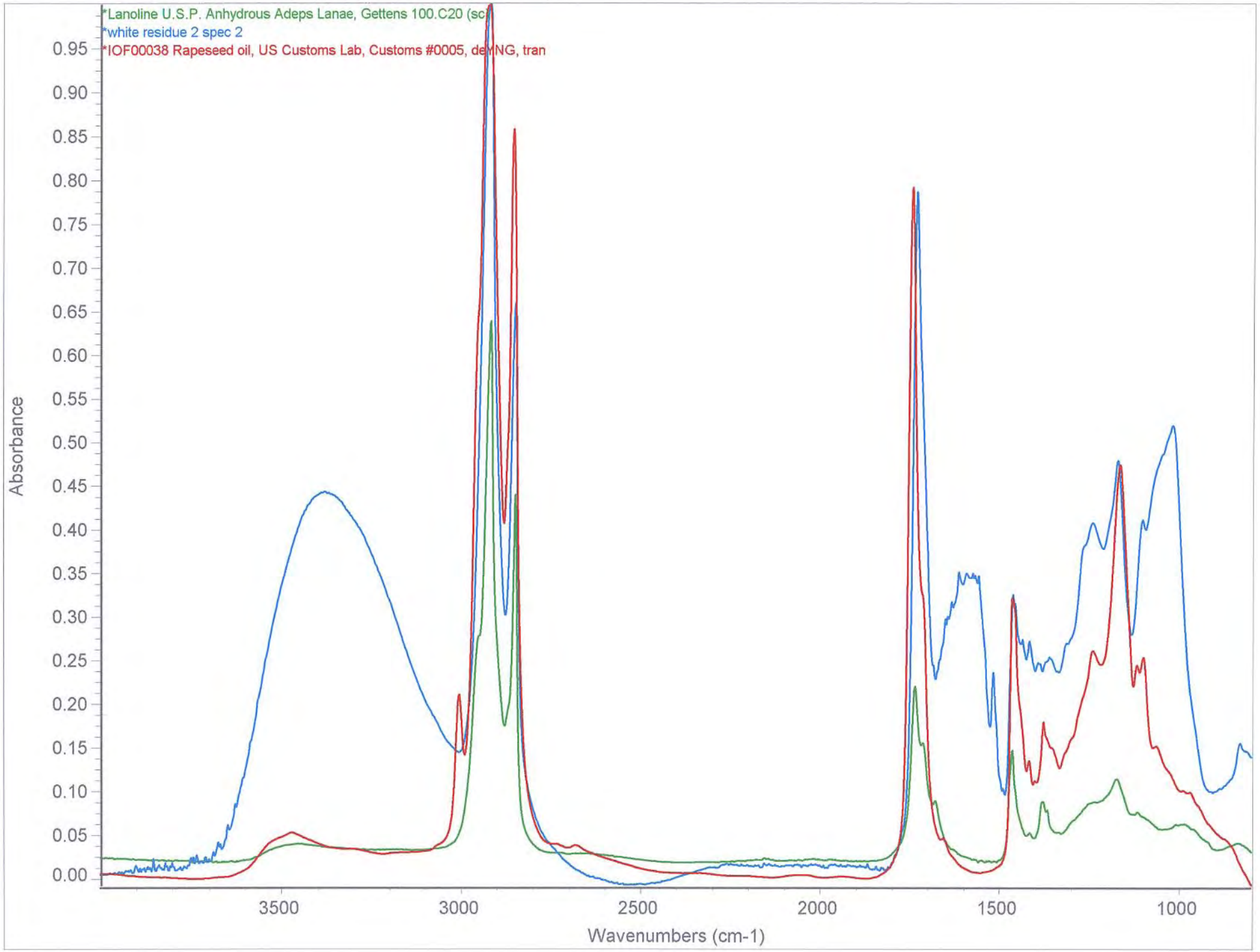
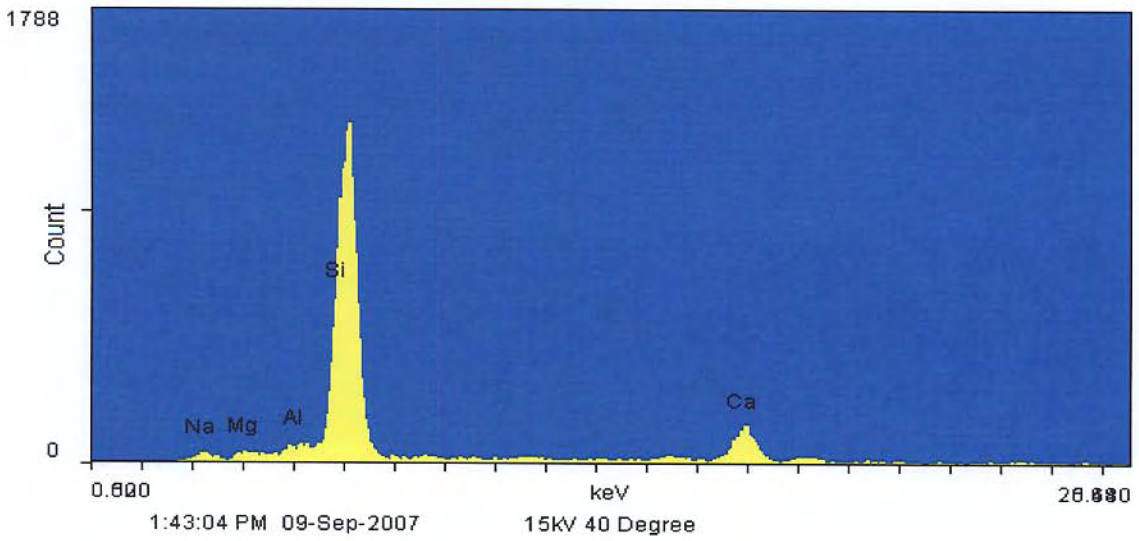

*Lanoline U.S.P. Anhydrous Adeps Lanae, Gettens 100.C20 (sc
*white residue 2 spec 2
*IOF00038 Rapeseed oil, US Customs Lab, Customs #0005, de NG, tran
Absorbance
0.95
0.90
0.85
0.80
0.75
0.70
0.65
0.60
0.55
0.50
0.45
0.40
0.35
0.30
0.25
0.20
0.15
0.10
0.05
0.00
3500
3000
2500
2000
1500
1000
Wavenumbers (cm-1)

Figure 3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Na
Mg
Al
Ca
keV
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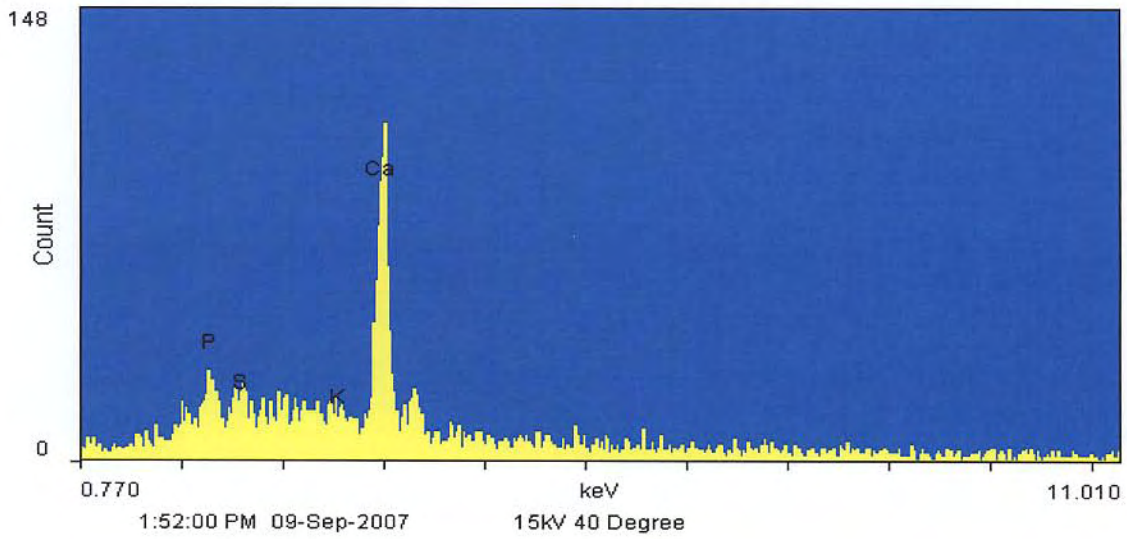
15kV 40 Degree

Figure 4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Ca
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S
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keV
11.010
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15kV 40 Degree