Frontier Analysis, Ltd.

TECHNICAL SERVICE RESPONSE NO.: UT006

Subject: Analysis of Samples Related to a Fire in England, Arkansas (March 9, 2000)

Date: October 19, 2000

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BLT Research

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Background/Objective:.

An unusual fire occurred on March 9, 2000 in Pulaski County, Arkansas. The fire was unusual in that the burn area was ca. “a mile-long, 150 yard-wide area of intermittent fires; in some burning areas the fire was reported to be excessively hot and burning with a sparkly effect.[1]” There were reports from various witnesses of such things as explosions and aerial light phenomena. A number of people who were on-site were interviewed and seemed to feel the fires were “bizarre”. 20 samples of ash and soil, which included controls, were examined to determine the cause of the fire. Some possibilities that were suggested include: illegal methamphetamine lab having blown up; 2) multiple meteorites; 3) misfired missile from the Little Rock Air Force Base; 4) plane wreckage (though there was no debris or report of a missing plane). Other observations were that a crop duster had been at work in the area; a power Plant (White Bluff) is 5 miles SE of the site; The US Natural Center for Toxicological Research is about 7-8 miles SE of the site. These sources could have contributed unrelated contamination to the fire debris. The following two photographs were submitted with the samples.

Heavily Burned Area

Possible Point of Origin

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[1] Field Report written by JoAnne Scarpellini, March 2000.

Conclusions:

This analysis does not detect material that may suggest an unusual cause of the fire. Typical mineral components of soil and burn residues from vegetation are detected. And in one sample (a purple/gray ash) an additional component, $KHCO_3$, typically used as a fire-extinguishing agent is present. I suspect the samples examined were not directly from the points of fire origin. Samples from such sources would reveal more pertinent information. There are ignition sources that could be ruled out if (per chance) any of the samples are from a point of origin. For example, we can say that no trace hydrocarbon residues were obvious which may rule out a fuel source. The water extracts for the most part are noted to be on the basic side which possibly eliminates strong acids. Chloroform extracts reveal esters which are natural to soil, i.e. long chain fatty acid esters. Following are the more detailed conclusions provided by the analyses for the site soils and ashes.

All soil samples (S1 – S6) and controls (C1 – C2) are expectedly composed of ***mostly silicate minerals (quartz type predominates), very small amounts of carbonate (probably mostly calcium carbonate), occasional trace amounts of sulfate and possibly natural organics.*** Water extractable materials range from ca. 1.4 to 1.8 wt.% (pHs ranges between 5 – 6). These consist of mostly residual colloidal silicate fines and a trace amount of soluble carbonate. Trace amounts of chloroform soluble materials consist of natural organics, e.g. long chain fatty acid esters.

The ash samples (S1 – S6) and controls (C1 – C2) are not unusual. These samples contain ***primarily silicate minerals (mostly quartz type), some carbonate (probably mostly calcium carbonate type) and uncombusted humate substances and possibly trace amounts of natural ester organics.*** (Note: The control ashes contain much more calcium carbonate than the S1 – S6 ashes.) Water extractable materials range from 1.5 – 2.2 wt.% (pHs range between 6 – 7.) The extracts contain a major amount of residual colloidal silicate fines, and moderate amounts of soluble carbonate[2] and humate substances. Very trace amounts of chloroform soluble materials are present. Like the above soil samples, these are indicated to be natural organics which are probably long chain fatty esters.

The white ash is typical of wood which had been extensively combusted by a very hot fire[3]. It is composed of ***primarily calcium carbonate, some sulfate, a small amount of residual silicate minerals (mostly quartz type), trace amounts of residual uncombusted humates, and very trace amounts of possible natural esters.*** Water soluble material amounts to 5.8 wt.% (pH = 9). These are composed of sulfate, soluble carbonate, and humate substances. The insolubles (94.2 wt.%) are mostly calcium carbonate and some silicate. There are trace amounts of chloroform solubles which are probably natural ester organics.

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[2] Calcium carbonate is very slightly soluble in water.

[3] M. K. Misra, K.W. Ragland, A. J. Baker, *Biomass & Bioenergy,* Vol. 4, No. 2, pp. 103-116, 1993 (In depth excellent research article on "Wood Ash Composition as a Function of Furnace Temperatures".)

The purple/gray ash does differ from the others. It contains at least ***45 wt.% $KHCO_3$ (potassium bicarbonate), a major amount of calcium carbonate, some sulfate, and very small amounts of carbon, silicate minerals, and humate materials, and possibly very trace amounts of natural ester organics.*** Potassium bicarbonate is commonly used as a fire-extinguishing agent. The calcium carbonate and sulfate are typical of wood ash residue. The small amount of carbon in presence of the other components most likely produces the purple/gray hue of the sample. Also the presence of carbon and humate substances indicates a cooler fire probably because it was extinguished by the potassium bicarbonate. The water extract amounts to 48 wt.% (pH = 10). The extract consists of the very soluble potassium bicarbonate and some sulfate. The insolubles (ca. 52 wt.%) consist of mostly calcium carbonate and some silicate, carbon, and humate substances. Trace amounts of chloroform solubles may be from natural ester organics.

There is a possibility that elemental analysis by EDX or ICP for trace elements may lend additional information[4]. I would suggest the tests be done on select samples because of the very high cost to run all of the tests (literally thousands of dollars) unless there is someone trained and with access to EDX or ICP who is interested enough to volunteer his/her services. Each test runs from ca. $150 (EDX) to $450 (ICP). These analyses should be done on only samples that can be confirmed as coming from the fire's point of origin. Also there is a method used by the US Army which involves soil extraction with an organic solvent followed by High Precision Liquid Chromatography (HPLC) to identify organic residues typical of some exploded missiles. This methodology would have to be sent to a contract lab with HPLC capabilities[5]. They would have to set up the method and run the samples which would be extremely expensive (probably well in excess of $5000.00 and closer to $10,000). Again confidence in proper sampling at the site would be a definite prerequisite before investing this amount of money in this analysis. I can access this method should the submitter have this sort of funding and feel it is worth proving the fire is the result of a misfired missile.

Procedure:

Samples: Twenty soil and ash samples were received for analysis. These include control samples. The locations of the Soils and Ashes labeled S1 – S6 from the fire site can be found on a hand drawn site map which was submitted with the samples. The map is included in the appendix. "All ash samples (sampled by J. Scarpellini) were scooped from the surface and bagged, then as much ash as possible scraped away. Then a soil sample was scooped from that spot so the soil sample is what was directly under the ash sample. …As best as I can tell the enclosed soil samples come from an area not associated with the most severe burned spots and also not associated with the areas where the white "sparkly" ash or purple "ash" was retrieved (by Al Collier) [6]".

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[4] Frontier Analysis does not have EDX or ICP capabilities.

[5] Frontier Analysis does not have HPLC capabilities.

[6] Ref. 1.

Following is a listing of the samples.

(Soils) Sampled 3/13/2000 by J. Scarpellini

- S1 ⎫
- S2 ⎩ From the south side (most severely burned)
- S3 ⎧
- S4 ⎭
- S5 ⎩ From the north side
- S6 ⎧

(Soil Controls)

- C1 ⎩ Taken from what appeared to be an agricultural burn ¼ to ½ mile from the woods burn
- C2 ⎧
- C3 Taken from under burned grass and leaves, High Ridge, Mo. (400 miles away)

(Ashes) Sampled 3/13/2000 by J. Scarpellini

- S1 ⎫
- S2 ⎩ From the south side (most severely burned)
- S3 ⎧
- S4 ⎭
- S5 ⎩ From the north side.
- S6 ⎧

(Soil Controls)

- C1 ⎩ Taken from what appeared to be an agricultural burn ¼ to ½ mile from the woods burn
- C2 ⎧
- C3 Taken from under burned grass and leaves, High Ridge, Mo. (400 miles away).
- White Ash Sampled 3/14/2000 by A. Collier
- Purple Ash Sampled 3/14/2000 by A. Collier

Infrared spectra were obtained from all samples (as received). Water and chloroform extracts were done on selected samples in order to isolate and detect any other components. Approximately 3 grams quantities were extracted except in the case of the purple ash for which roughly ½ gram was extracted. (There was a limited amount of sample available). The water extracts were done quantitatively. pH tests were done on each water extract. Additional infrared spectra were acquired from all the extracts and two water insolubles (the white and purple ashes).

Results:

The results of each individual test done on the 20 samples are included in this section. These results are summarized in the conclusions section on the first page of the report. The following table shows results of the FT-IR analysis of the “as received” samples and selected water extracts of the soil and ash samples. (The infrared spectra are attached in the appendix as well as pertinent references.) Also included in the table are the amounts of extracted material as well as the pH of the water after the extraction.

Samples	Composition (As Determined by FT-IR)	H_2O Ext. (Wt. %)	pH	H_2O Ext. Composition	H_2O Insol. Composition
(SOILS)					
S1	Mostly Silicate (mostly Quartz type); Trace Carbonate (Probably Calcium Carbonate) **(FT-IR Fig. 1a)**	1.84	6	Residual Colloidal Silicate; Trace Carbonate **(FT-IR Fig. 11a)**	-
S2	Mostly Silicate (mostly Quartz type); Trace Carbonate (Probably Calcium Carbonate); Possible Trace Sulfate **(FT-IR Fig. 1b)**	-		-	
S3	Mostly Silicate (mostly Quartz type); Trace Carbonate (Probably Calcium Carbonate) **(FT-IR Fig. 2a)**	-		-	
S4	Mostly Silicate (mostly Quartz type); Trace Carbonate (Probably Calcium Carbonate); Possible Trace Sulfate **(FT-IR Fig. 2b)**	-		-	
S5	Mostly Silicate (mostly Quartz type); Trace Carbonate (Probably Calcium Carbonate); Possible Trace Sulfate **(FT-IR Fig. 3a)**	1.37	5	Residual Colloidal Silicate; Trace Carbonate **(FT-IR Fig. 11b)**	-
S6	Mostly Silicate (mostly Quartz type); Trace Carbonate (Probably Calcium Carbonate); Possible Trace Sulfate **(FT-IR Fig. 3b)**	-		-	-
C1	Mostly Silicate (mostly Quartz type); Trace Carbonate (Probably Calcium Carbonate); Possible Trace Sulfate **(FT-IR Fig. 4a)**	-		-	-
C2	Mostly Silicate (mostly Quartz type); Trace Carbonate (Probably Calcium Carbonate); Possible Trace Sulfate **(FT-IR Fig. 4b)**	-		-	-
C3	Mostly Silicate (mostly Quartz type); Small Amount Carbonate (Probably Calcium Carbonate); Possible Trace Sulfate **(FT-IR Fig. 5a)**	-		-	-

Samples	**Composition (As Determined by FT-IR)**	**H_2O Ext.** **(Wt. %)**	**pH**	**H_2O Ext. Composition**	**H_2O Insol. Composition**
(ASHES)					
S1	Mostly Silicate (mostly Quartz type); Small Amount Carbonate (Probably Calcium Carbonate); Humate **(FT-IR Fig. 5b)**	2.20	7	Residual Colloidal Silicate; Humate Substance (Probably Fulvic Acids); Small Calcium Carbonate **(FT-IR Fig. 12a)**	
S2	Mostly Silicate (mostly Quartz type); Small Amount Carbonate (Probably Calcium Carbonate); Humate **(FT-IR Fig. 6a)**	-		-	
S3	Mostly Silicate (mostly Quartz type); Moderate Amount Carbonate (Probably Calcium Carbonate); Possible Humate **(FT-IR Fig. 6b)**	-		-	
S4	Mostly Silicate (mostly Quartz type); Small/Moderate Amount Carbonate (Probably Calcium Carbonate); Possible Humate **(FT-IR Fig. 7a)**	-		-	
S5	Primarily Calcium Carbonate; Small Amount Silicate; Humate **(FT-IR Fig. 7b)**	1.47	6	Residual Colloidal Silicate (mostly Quartz type); Moderate Carbonate; Humate Substance (Probably Fulvic Acids) **(FT-IR Fig. 12b)**	
S6	Calcium Carbonate; Silicate; Humate **(FT-IR Fig. 8a)**	2.09	6	Residual Colloidal Silicate (mostly Quartz type); Moderate Calcium Carbonate; Humate Substance (Probably Fulvic Acids) **(FT-IR Fig. 13a)**	

Samples	**Composition (As Determined by FT-IR)**	**H_2O Ext. (Wt. %)**	**pH**	**H_2O Ext. Composition**	**H_2O Insol. Composition**
C1	Silicate (mostly Quartz type); Calcium Carbonate; Possible Humate **(FT-IR Fig. 8b)**	-		-	
C2	Silicate (mostly Quartz type); Calcium Carbonate; Possible Humate **(FT-IR Fig. 9a)**	-		-	
C3	Silicate (mostly Quartz type); Calcium Carbonate; Possible Humate **(FT-IR Fig. 9b)**	-		-	
White	Calcium Carbonate; Moderate Silicate **(FT-IR Fig. 10a) The spectrum compares to that of white ash found in a fireplace. See reference in the Addendum. Also these components are typical of that found in combusted wood ash** [7]	5.77	9	Residual Calcium Carbonate Fines; Sulfate; Humate Substance (Probably Fulvic Acids) **(FT-IR Fig. 13b)**	Calcium Carbonate; Moderate Silicate; **(FT-IR Fig. 14b)**
Purple	Potassium Hydrogen Carbonate Indications; Calcium Carbonate; Moderate/Strong Amount Sulfate **(FT-IR Fig. 10b) The components are typical of wood ash and has additional absorption from Potassium Hydrogen Carbonate.**	48.28	10	Potassium Hydrogen Carbonate; Small/Moderate Amount Sulfate **(FT-IR Fig. 14a) A reference spectrum of Potassium Hydrogen Carbonate can be found in the Addendum for comparison.**	**(Water Insols)** Insoluble Humate; Small Probable Silicate **(FT-IR Fig. 15a)** **(HCl Acid Treated Water Insolubles)** Carbon; Humate Substance; Probable Silicate **(FT-IR Fig. 15b)**

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[7] Ref. 3.

Chloroform extractions done on selected ashes and soils of S1, S5, S6, and the purple ash amounted to trace levels (sub milligram). The extracts in most cases contained inorganic fines which could not be avoided and were in greater amounts than the extracted material. These inorganics produces strong absorption in the infrared spectra which sometimes masked pertinent absorption belonging to the extracts. The following table shows the individual results of these tests. The extracts appear to be long chain fatty esters, i.e. the spectral absorbance bands are typical of natural esters found in soils.

Samples	**Chloroform Extracts (As Determined by FT-IR)**
(Soils)	
S1	Mostly Natural Ester; Some Silicate Fines **(FT-IR Fig. 16a)**
S5	Mostly Natural Ester; Some Silicate Fines **(FT-IR Fig. 16b)**
S6	Mostly Natural Ester; Some Silicate Fines **(FT-IR Fig. 17a)**
(Ashes)	
S1)	Mostly Silicate Fines; Small Carbonate Fines; Small Natural Ester **(FT-IR Fig. 17b)**
S5	Moderate Silicate Fines; Moderate Calcium Carbonate Fines; Small Natural Ester **(FT-IR Fig. 18a)**
S6	Predominate Calcium Carbonate Fines; Moderate Silicate Fines: Small Natural Ester **(FT-IR Fig. 18b)**
Purple Ash	Prominent Calcium Carbonate Fines; Moderate Silicate Fines: Very Trace Natural Ester **(FT-IR Fig. 19)**

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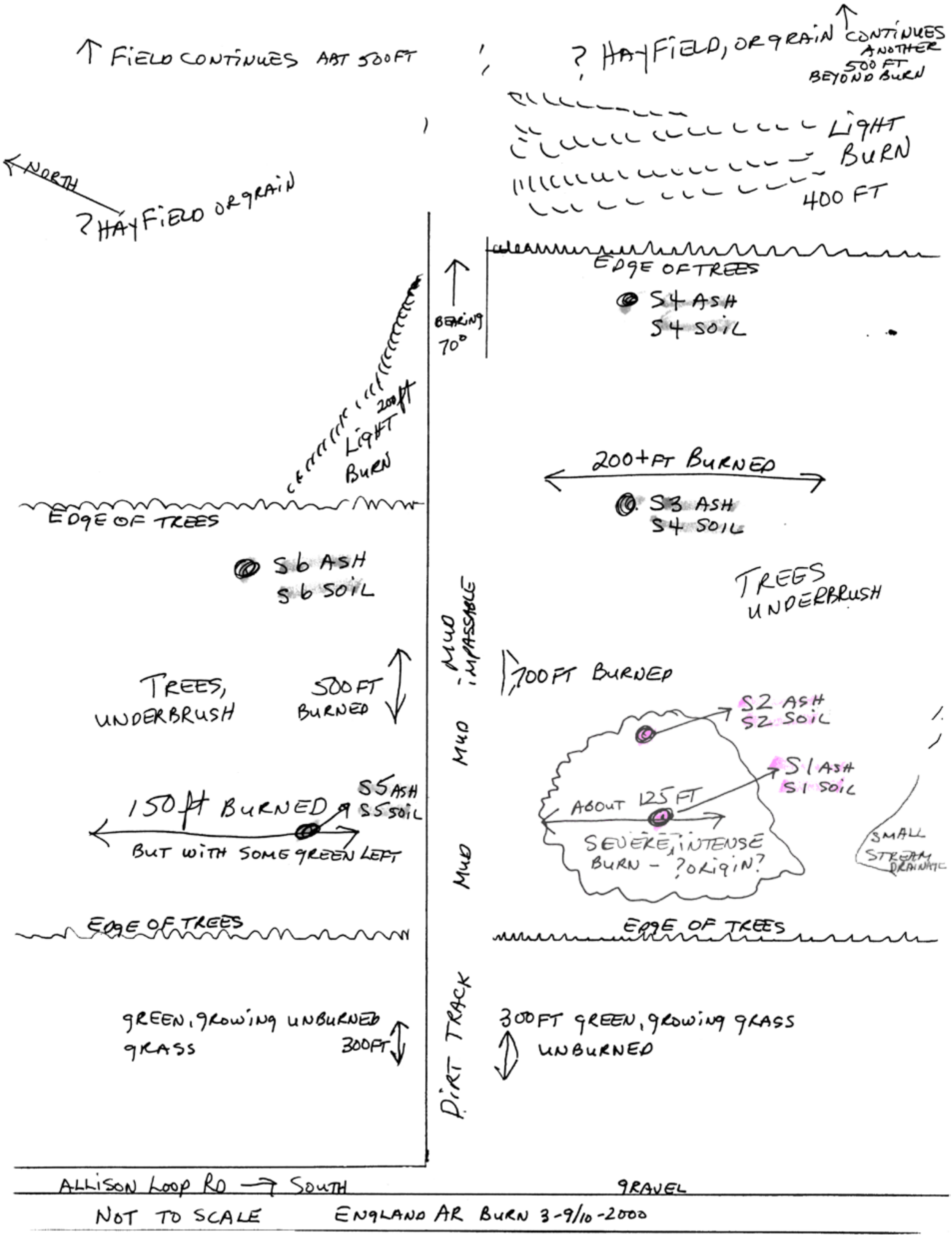
Phyllis A. Budinger

APPENDIX



Site map showing the locations of the samples

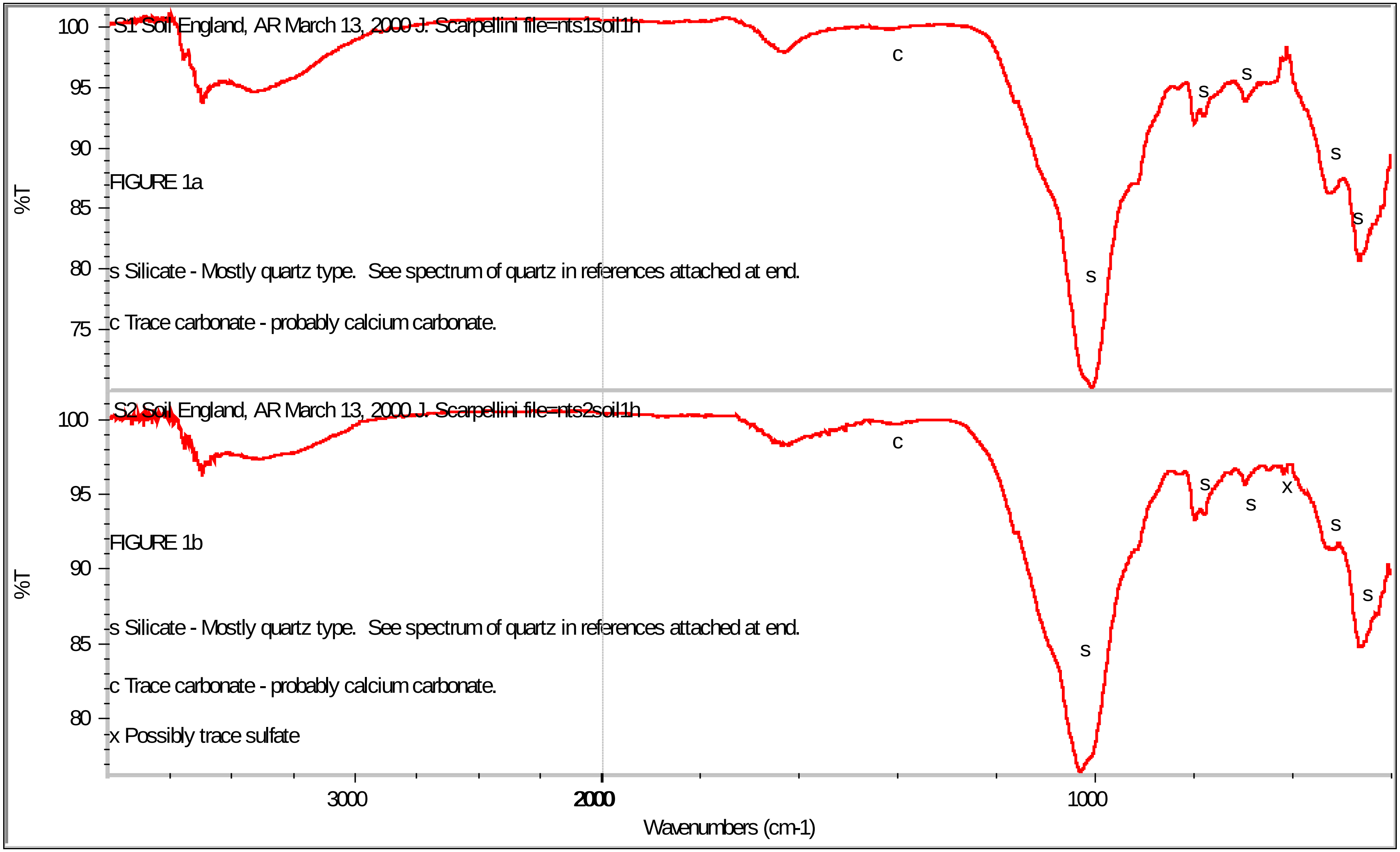


Figure 1. Infrared spectra of “as received” soils: a) S1; b) S2.

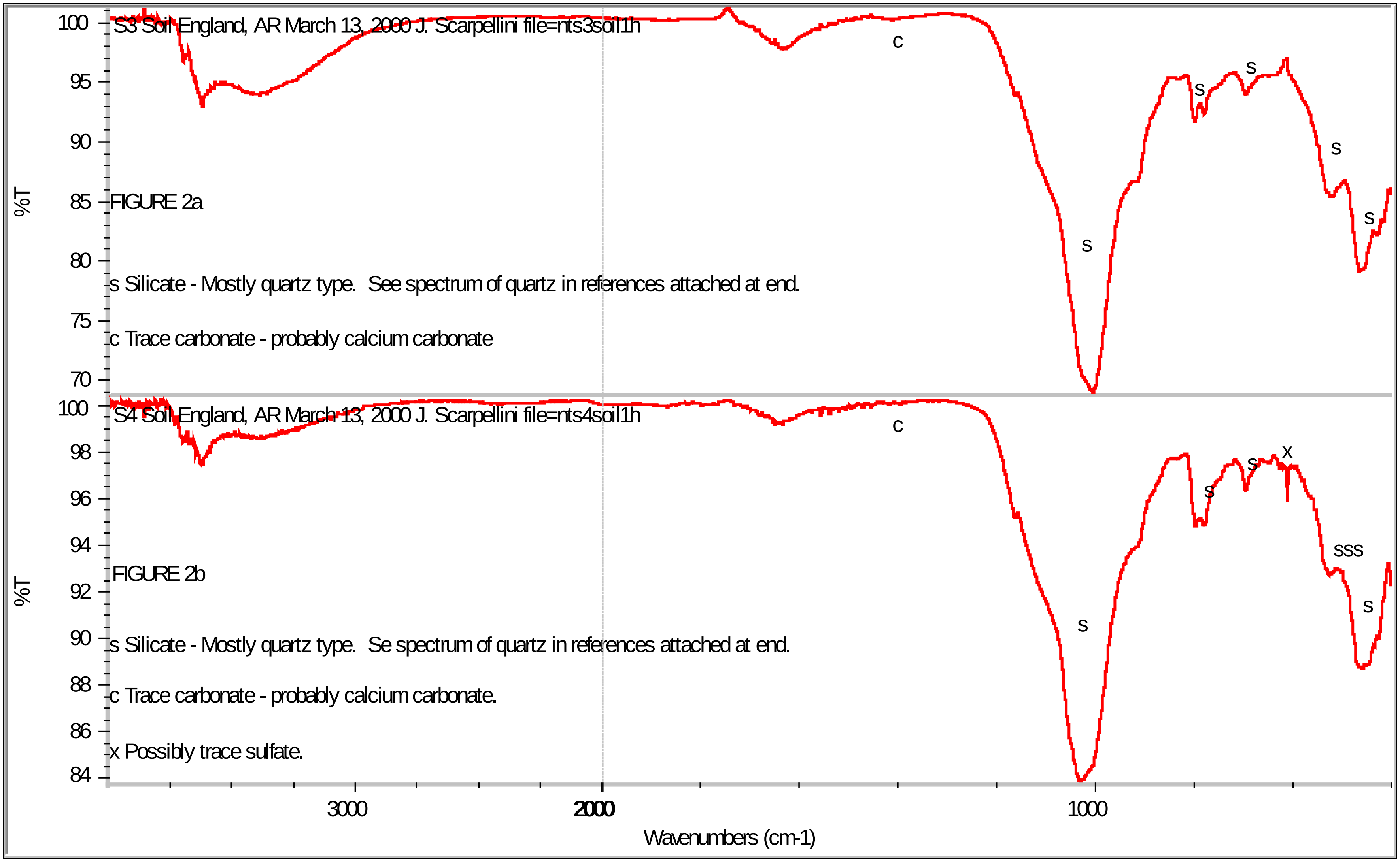


Figure 2. Infrared spectra of “as received” soils: a) S3; b) S4.

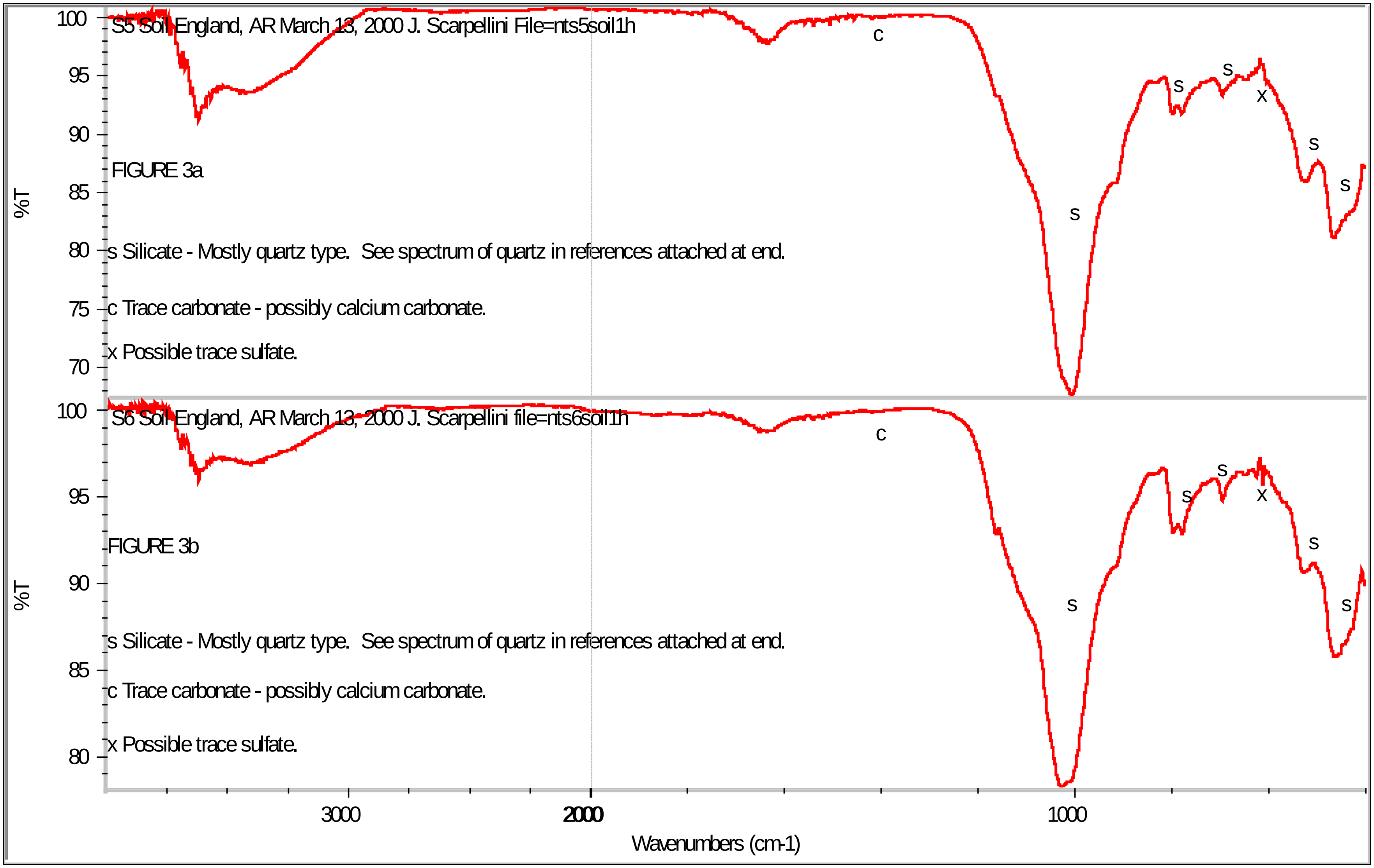


Figure 3. Infrared spectra of “as received” soils: a) S5; b) S6.

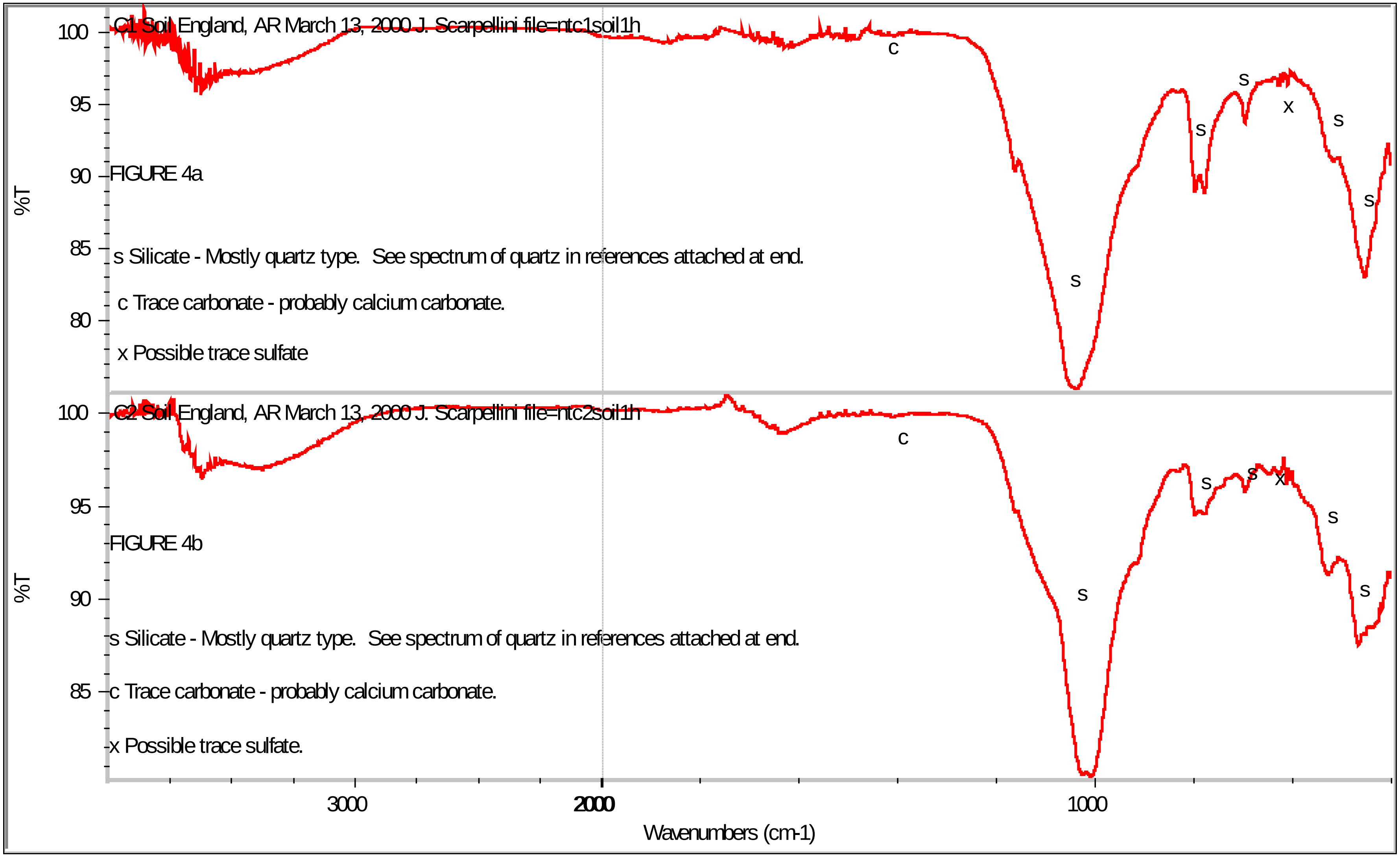


Figure 4. Infrared spectra of “as received” control soils: a) C1; b) C2.

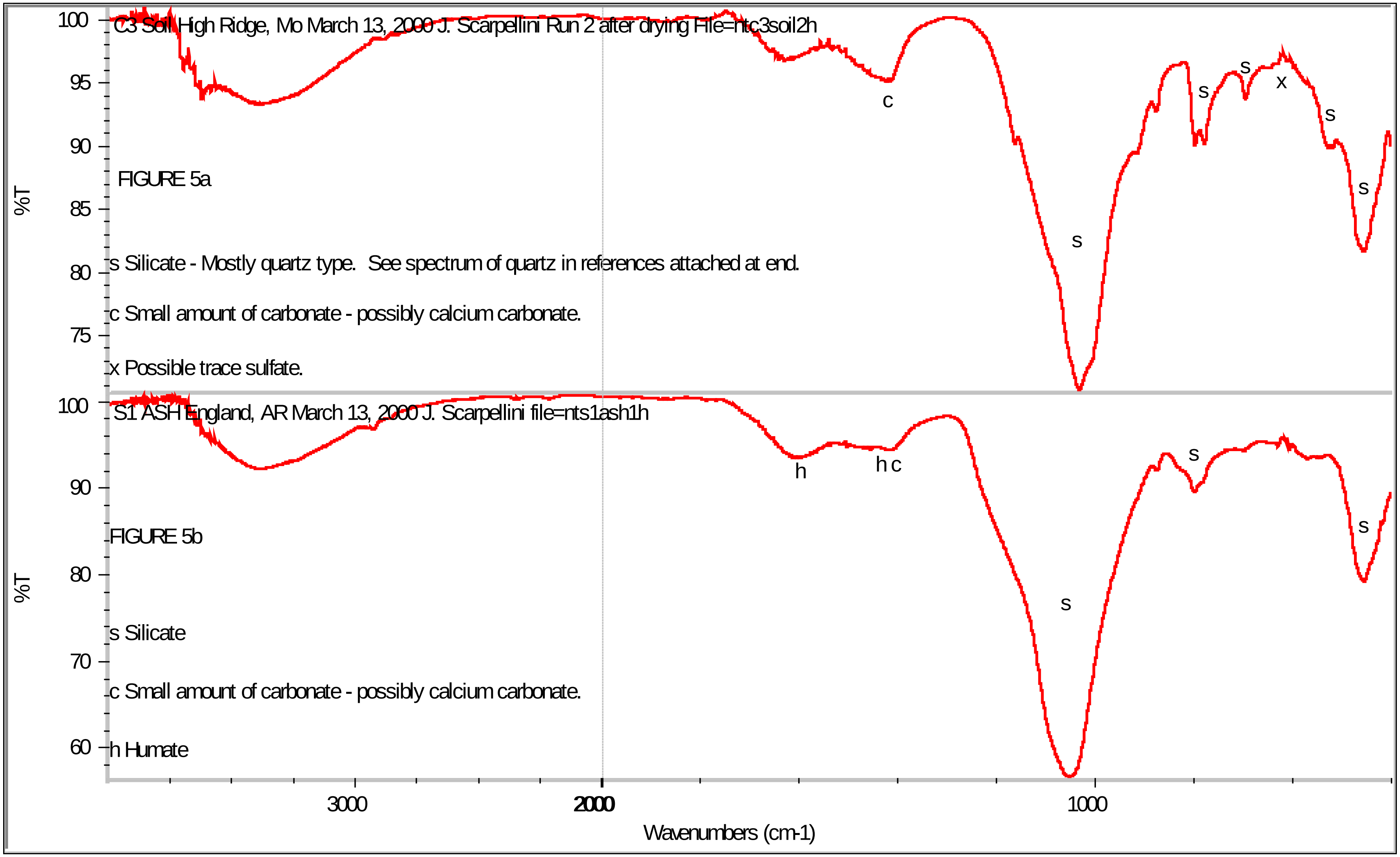


Figure 5. Infrared spectra of “as received” samples: a) C3 control soil; b) S1 ash.

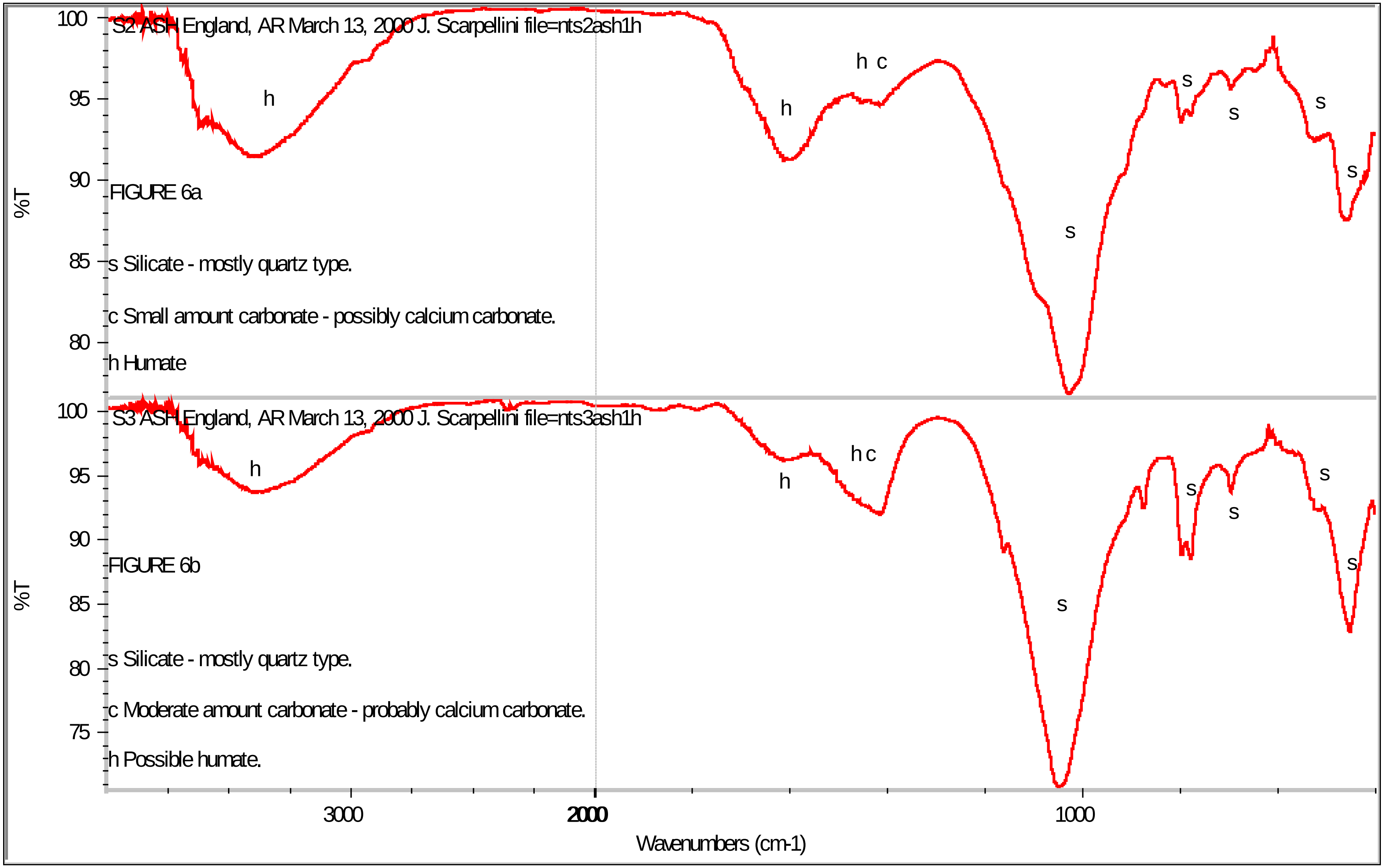


Figure 6. Infrared spectra of “as received” ashes: a) S2; b) S3.

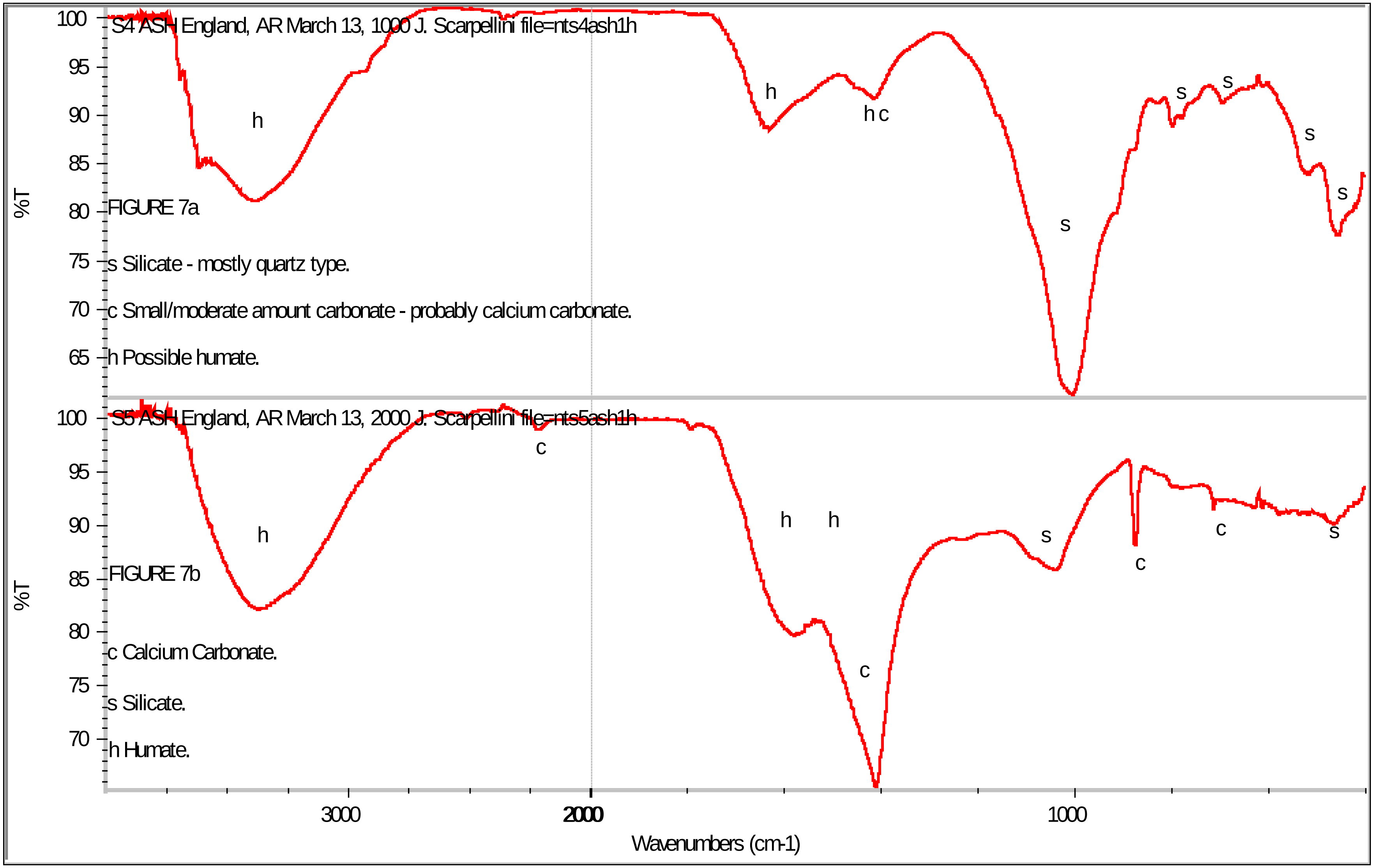


Figure 7. Infrared spectra of “as received” ashes: a) S4; b) S5.

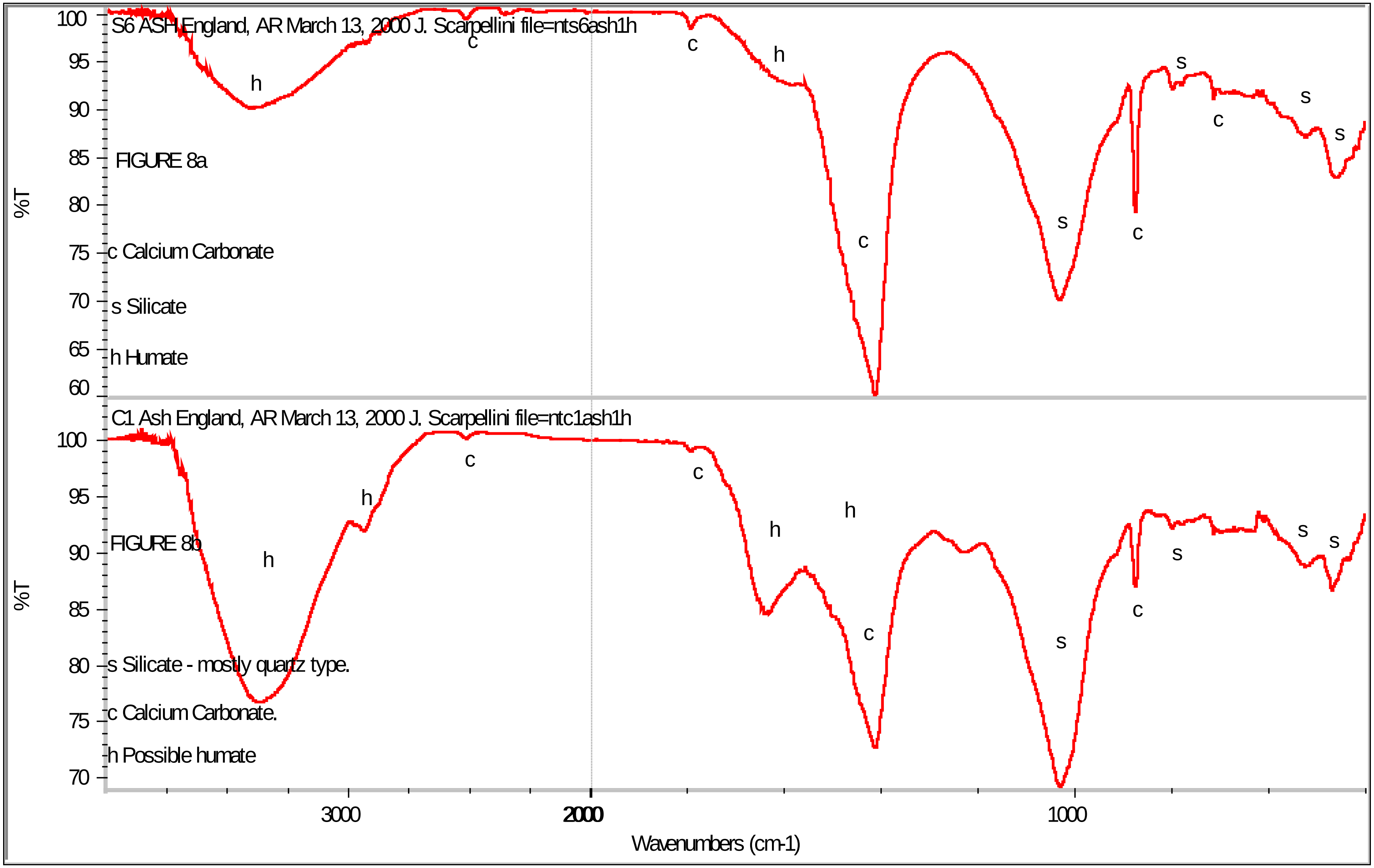


Figure 8. Infrared spectra of “as received” ashes: a) S6; b) C1 - control.

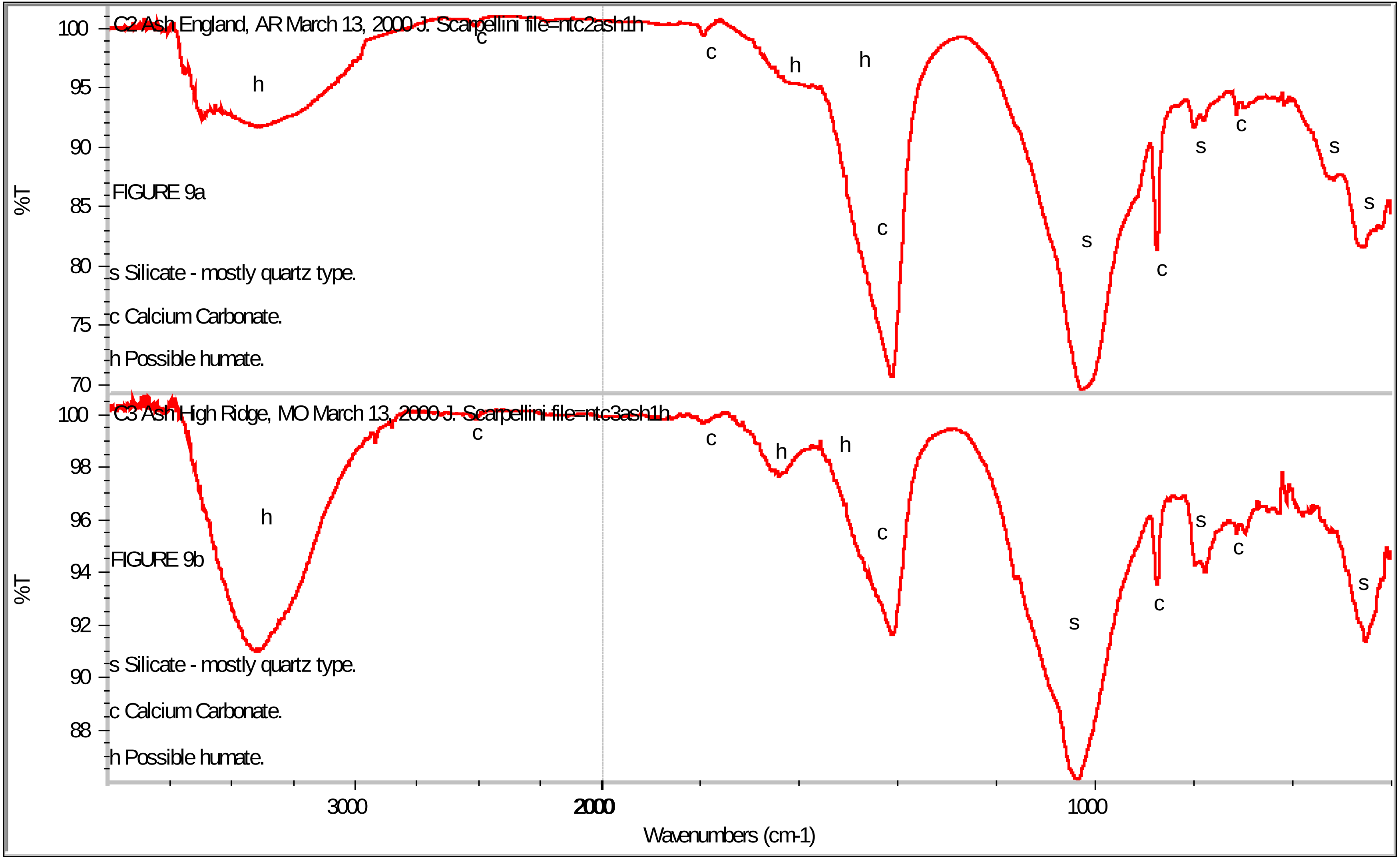


Figure 9. Infrared spectra of "as received" control ashes: a) C2; b) C3.

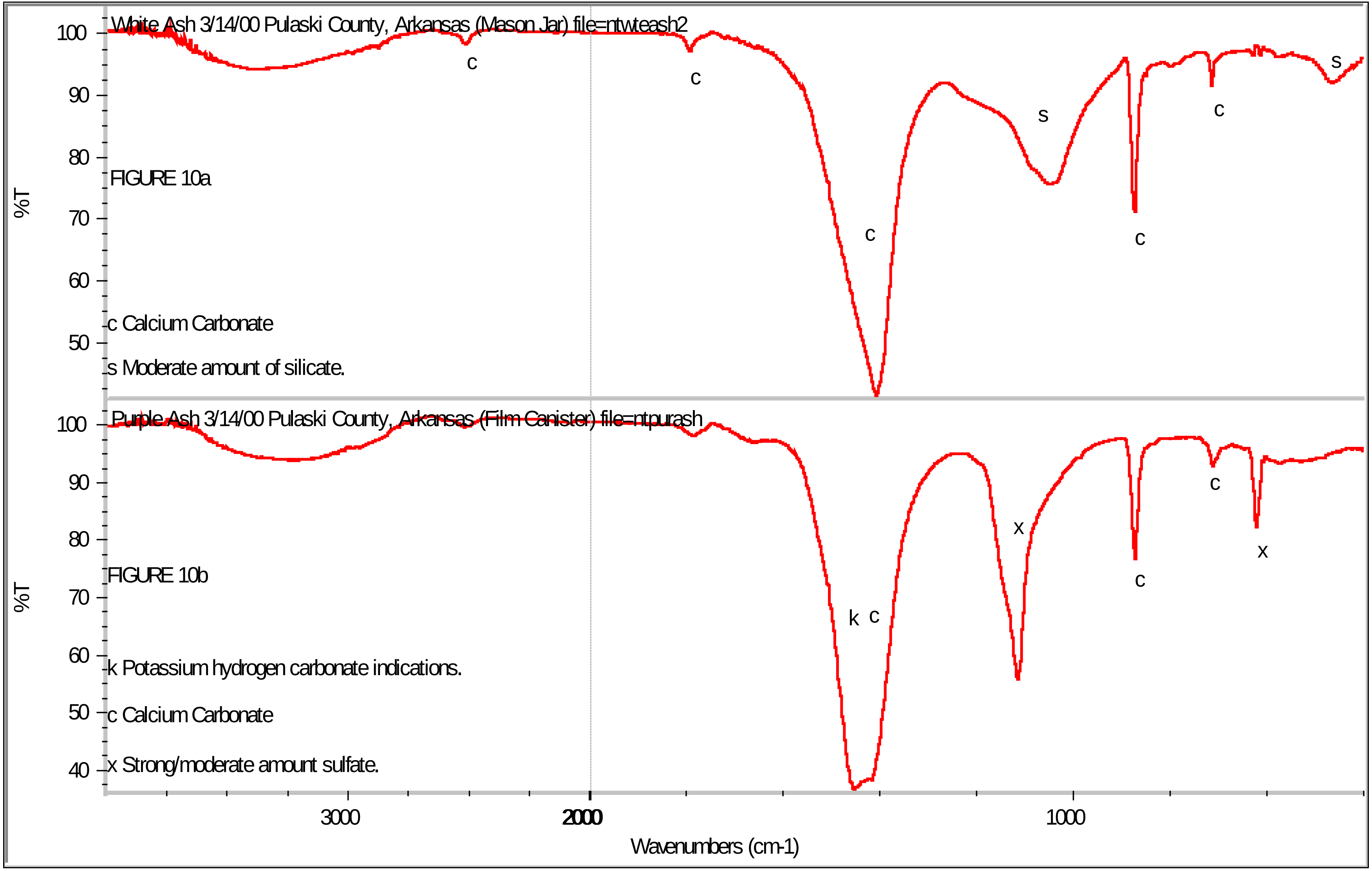


Figure 10. Infrared spectra of “as received” ashes: a) White Ash; b) Purple Ash.

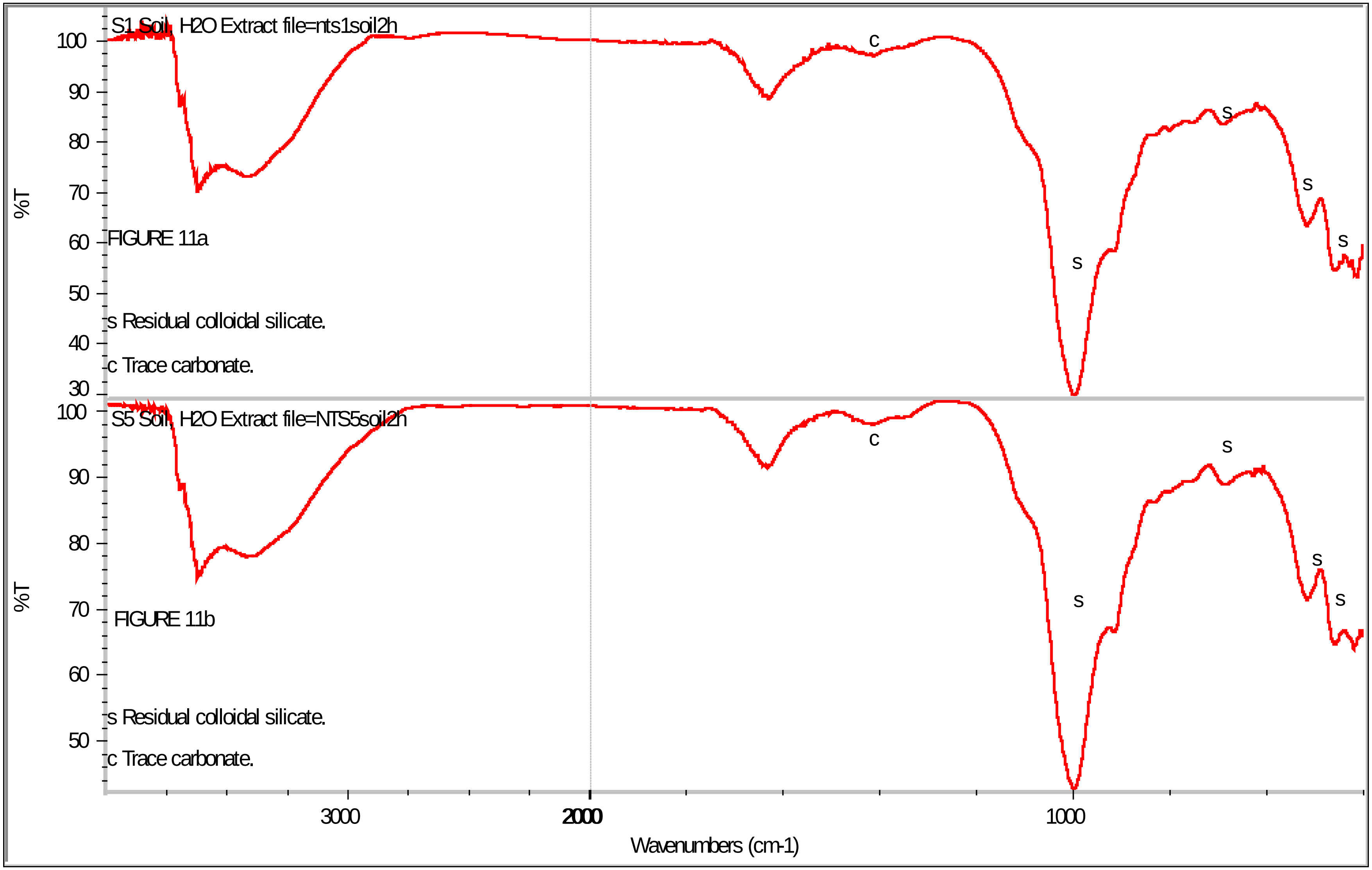


Figure 11. Infrared spectra of water extracts of soils: a) S1; b) S5.

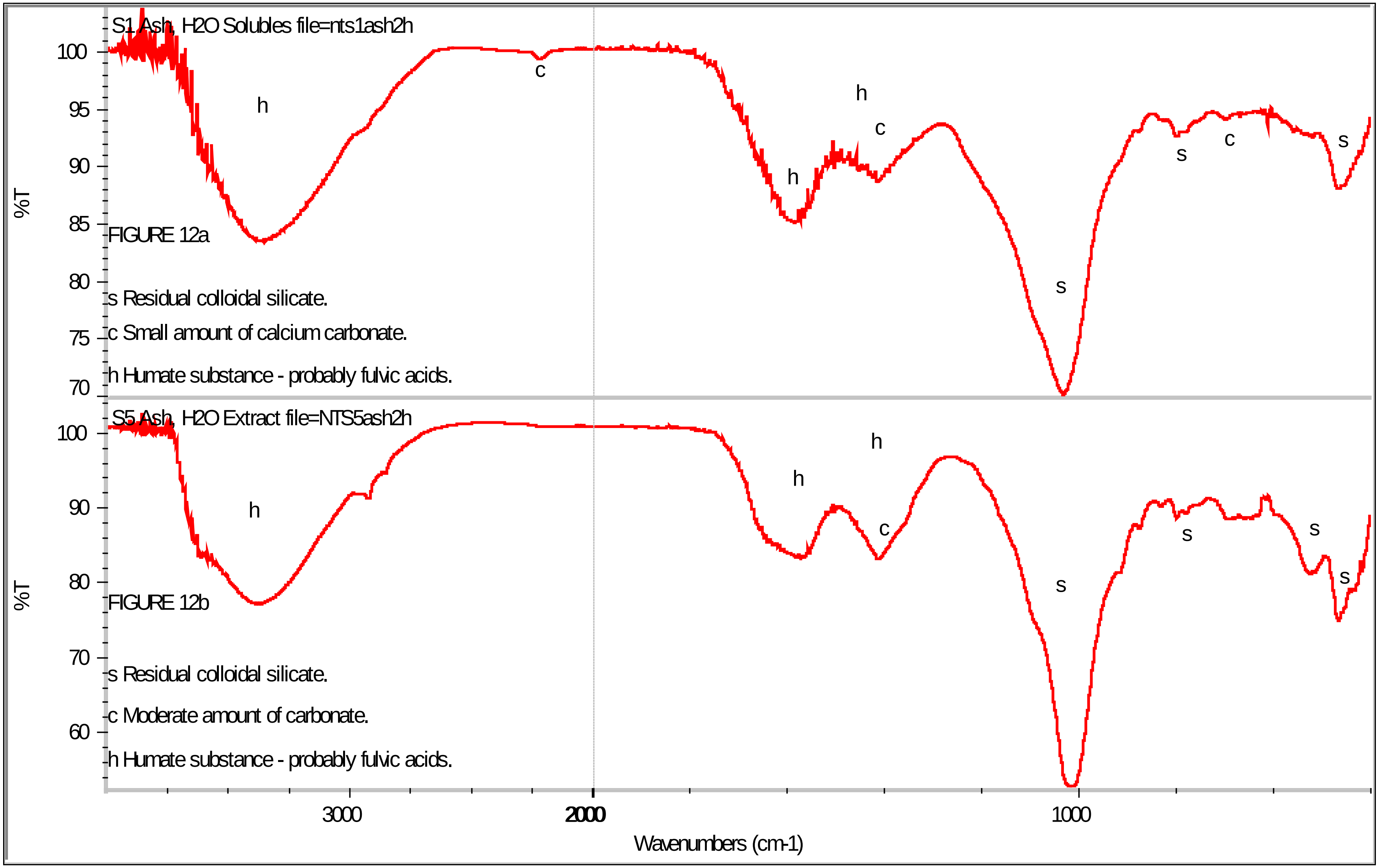


Figure 12. Infrared spectra of water extracts of ashes: a) S1; b) S5.

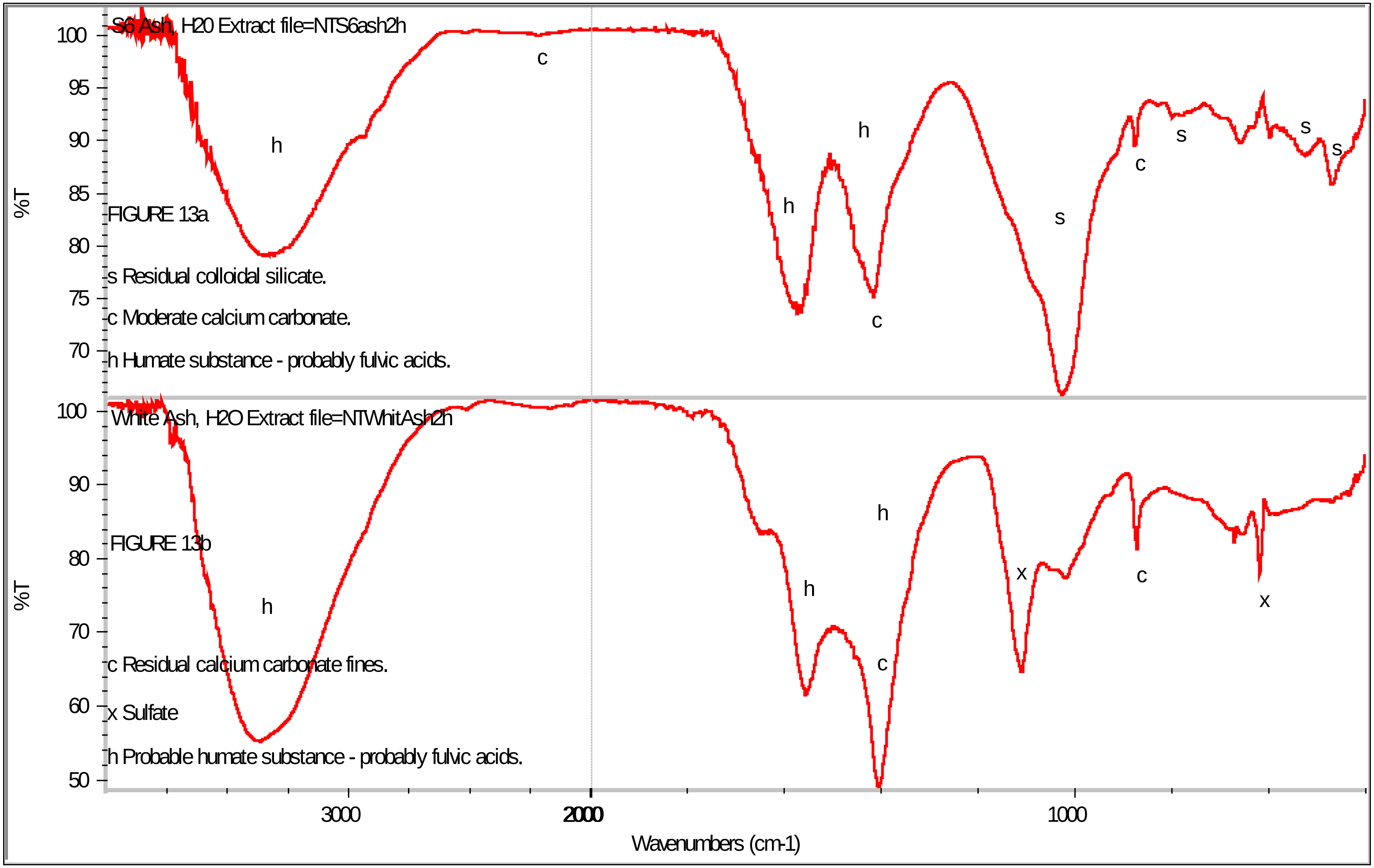


Figure 13. Infrared spectra of water extracts of ashes: a) S6; b) White Ash.

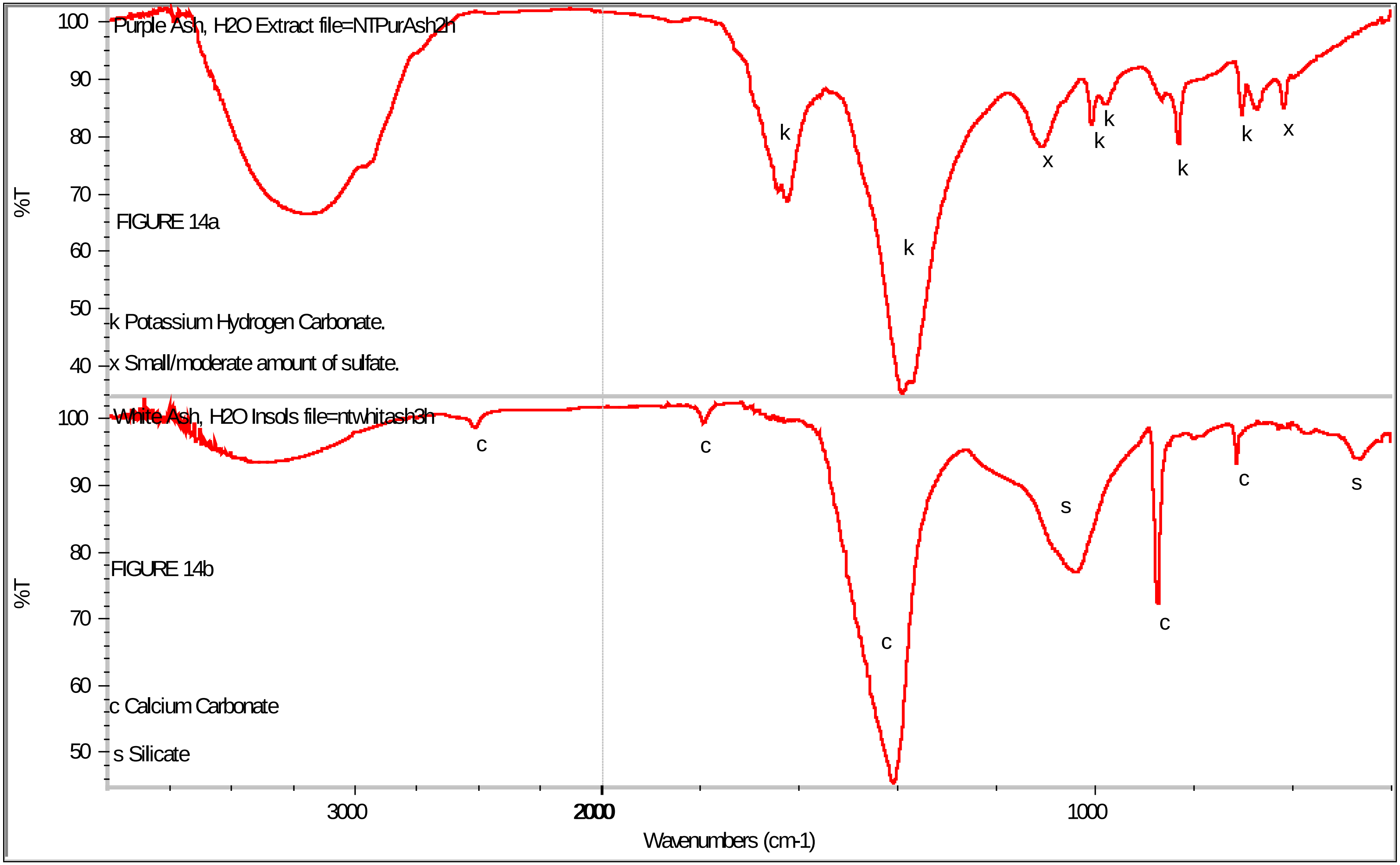


Figure 14. Infrared spectra of: a) water extract of Purple Ash; b) water insolubles of White Ash.

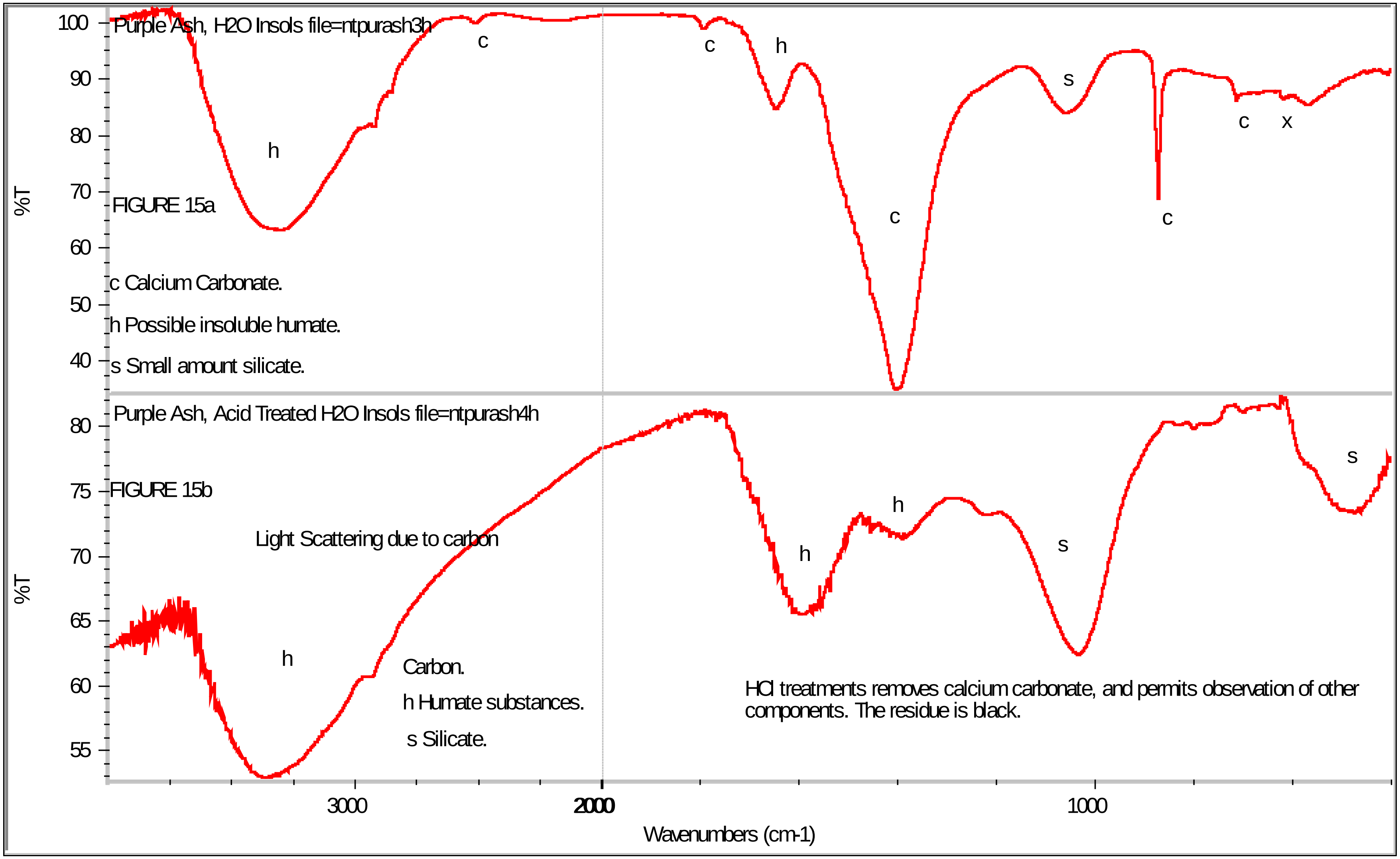


Figure 15. Infrared spectra of: a) water insolubles of Purple Ash; b) HCl treated insolubles.

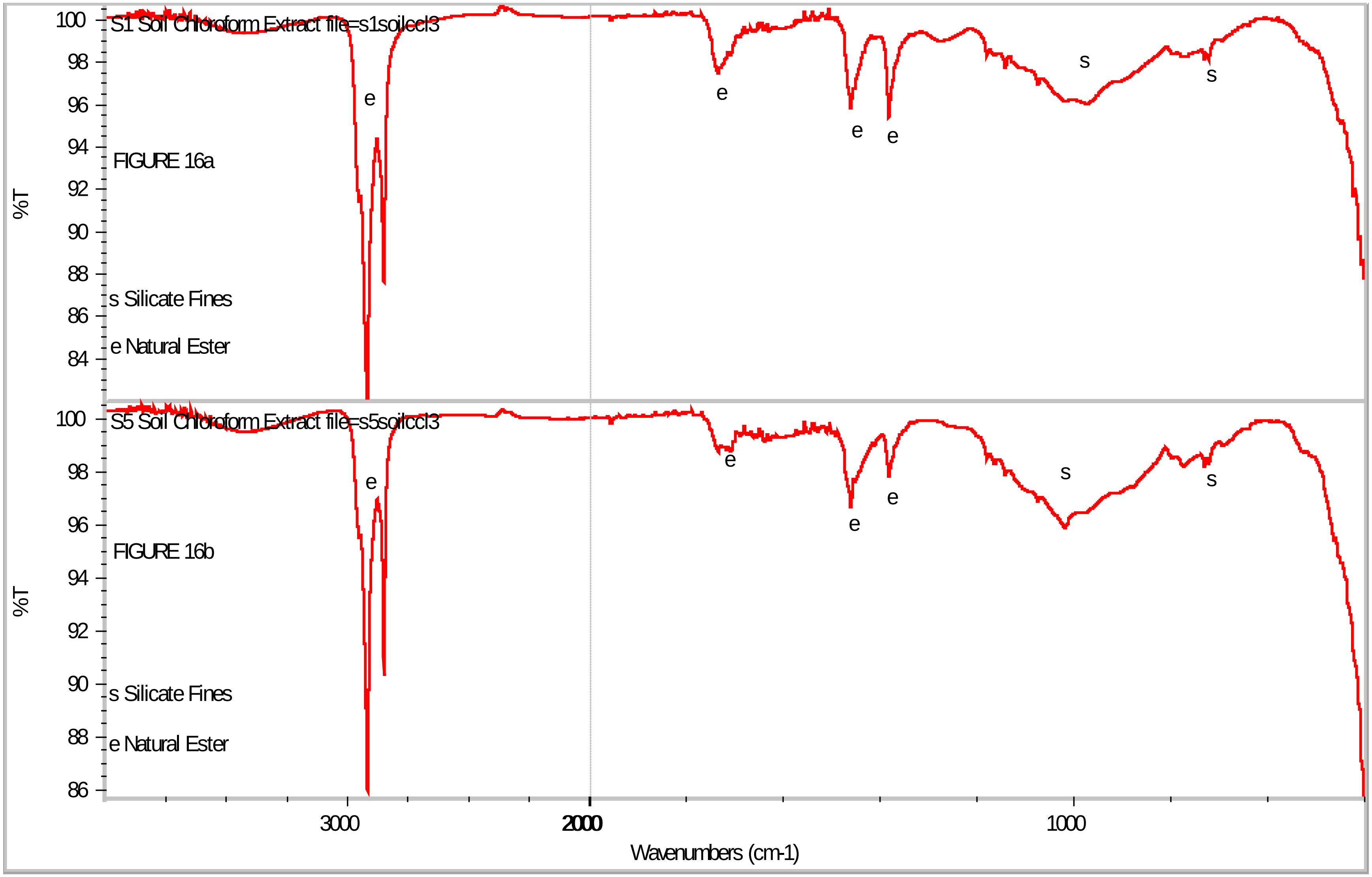


Figure 16. Infrared spectra of chloroform extracts of: a) S1 Soil; b) S5 Soil.

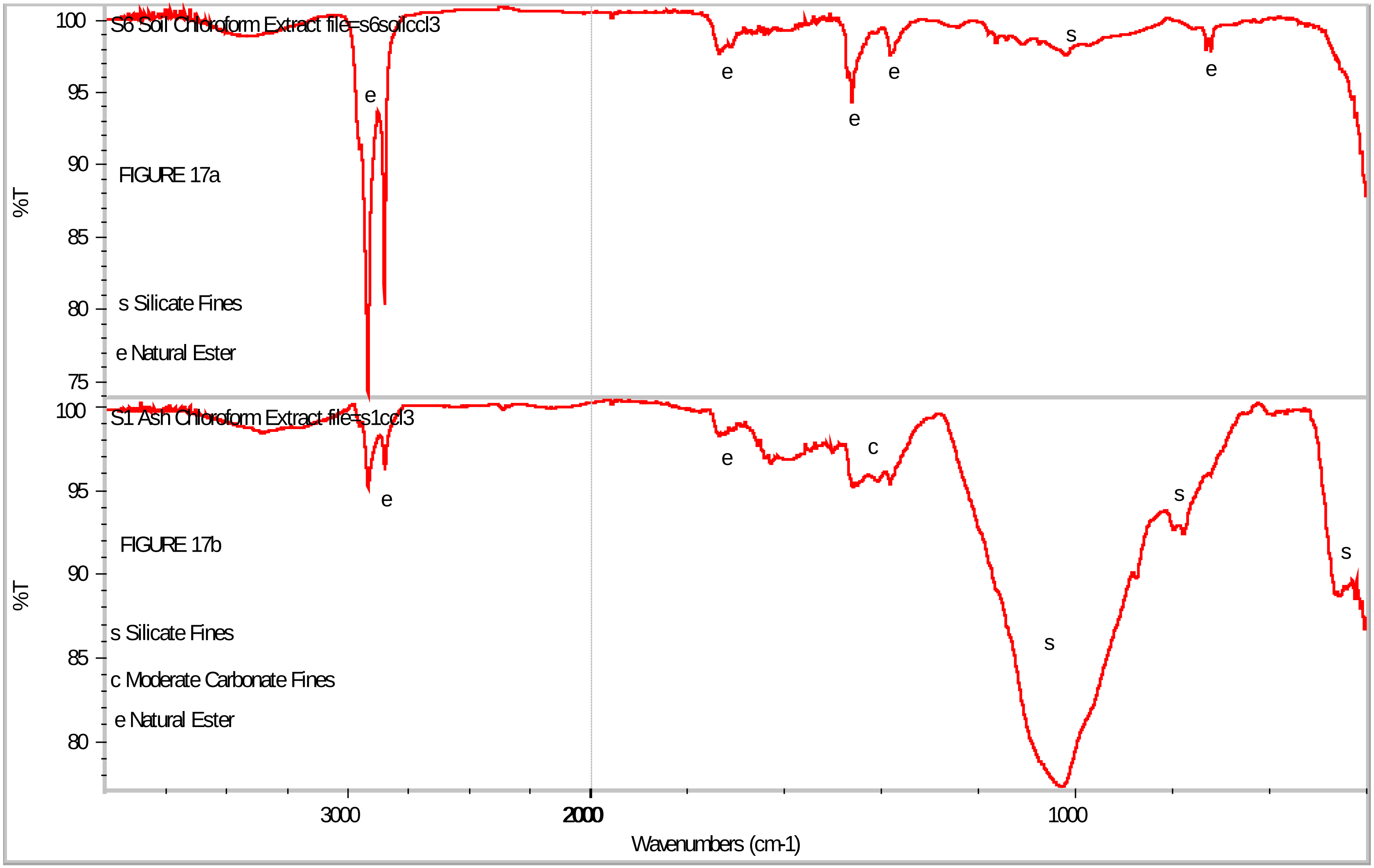


Figure 17. Infrared spectra of chloroform extracts of: a) S6 Soil; b) S1 Ash.

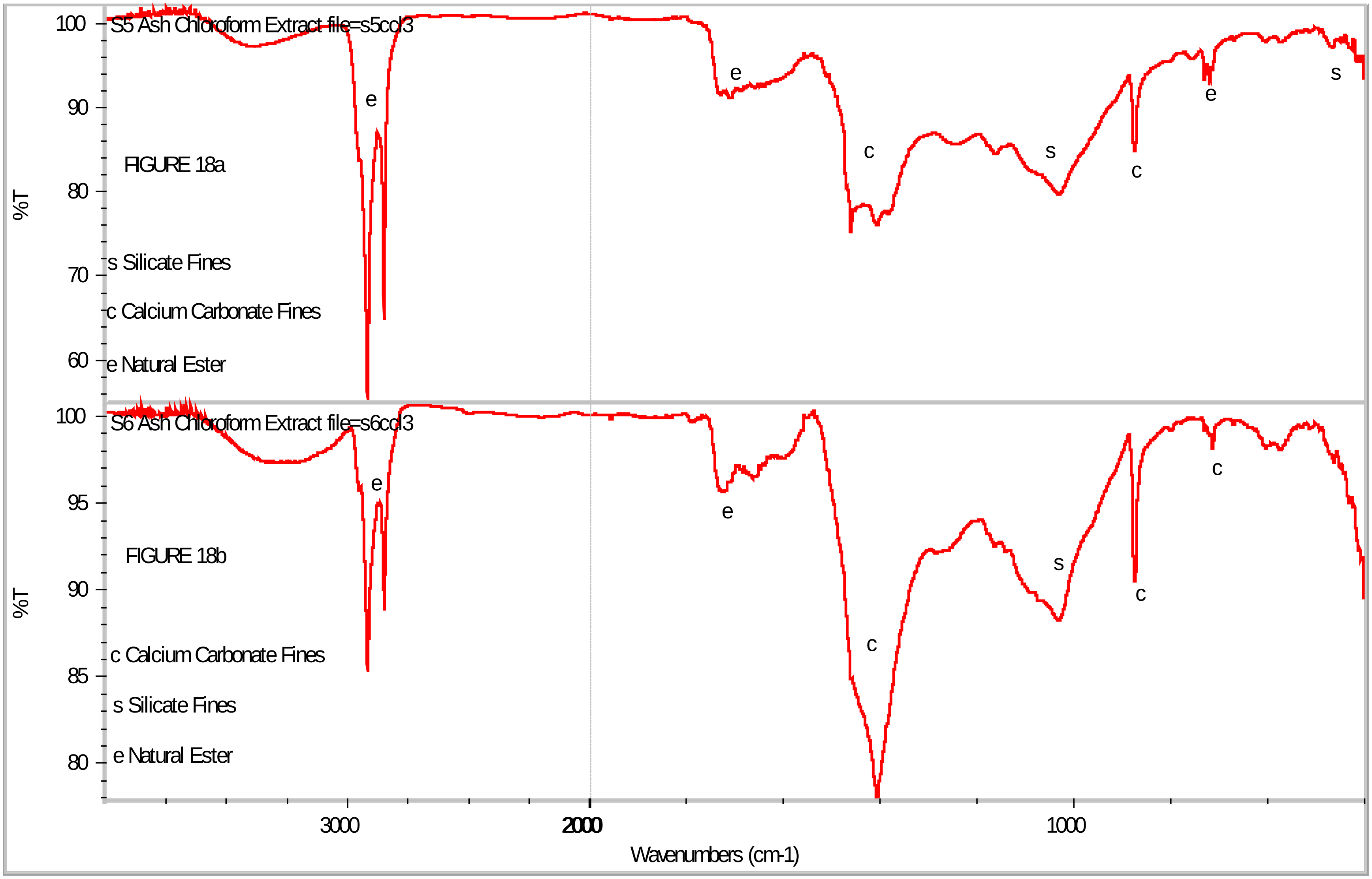


Figure 18. Infrared spectra of chloroform extracts of: a) S5 Soil; b) S6 Soil.

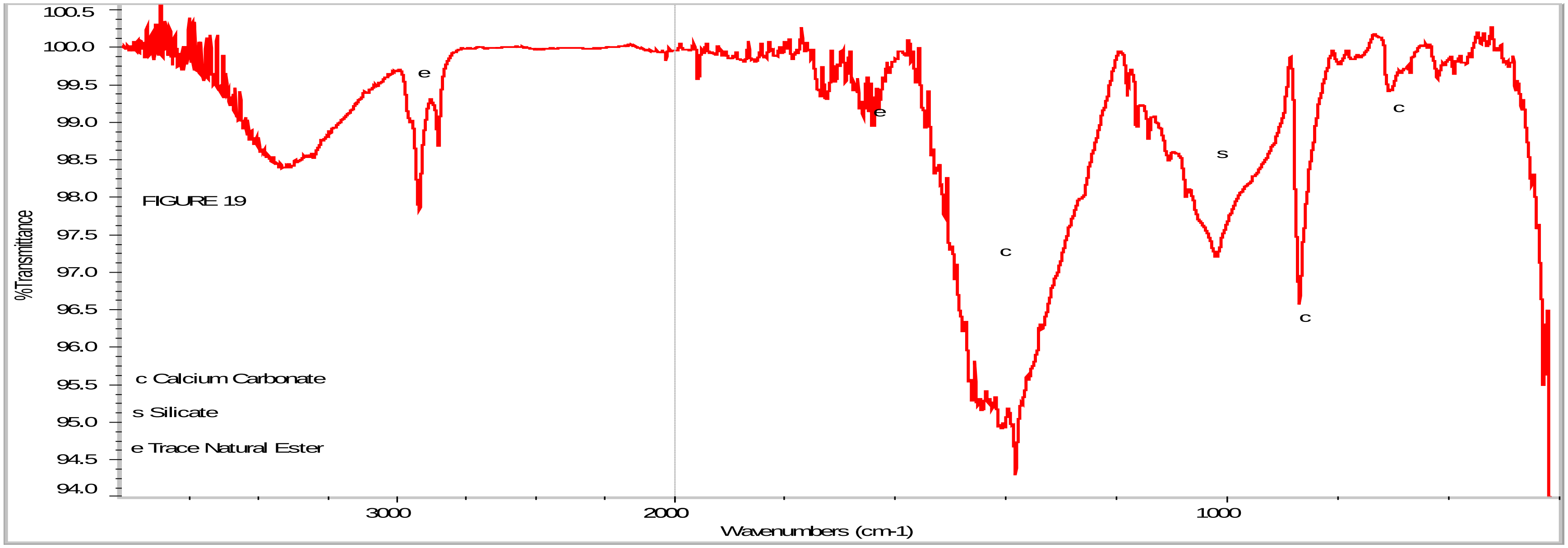


Figure 19. Infrared spectrum of a chloroform extract of the purple ash.