Electronic Supplementary Information

An assessment of spruce wood as a replacement for peat in the production of whisky: chemical and sensory analysis of new make spirits.

FT-ICR-MS

Spectral processing was carried out with CoreMS 2.5.2.beta in Python 3.11.4 starting with an intrinsic error investigation (Corilo et al., 2021, Van Rossum et al., 2009). Peaks were picked according to a signal to noise threshold based on the logarithmic intensity of each peak, including noise and analyte peaks (Zhurov et al., 2014) using standard deviations of 10 and the number of bins used in the histogram was set to 500. The minimum relative intensity required for a peak to be picked was set to 1%. Initial formula assignments were made with the elemental limits $C_{0\text{-}100}H_{0\text{-}200}O_{0\text{-}26}$ and a maximum/minimum error threshold of ±5 ppm. For each spectrum, the m/z and m/z error of each picked peak were then plotted to examine the intrinsic error distribution of the instrument. From this a ppm error range was generated from which only peaks within the range were calibrated using a polynomial function and a calibration list of known whisky compounds (Kew, 2018). Final formula assignments were made using the elemental limits $C_{1\text{-}90}H_{4\text{-}200}O_{0\text{-}26}S_{0\text{-}5}N_{0\text{-}5}$ for m/z between 100 and 800 and a maximum/minimum error threshold of ± 0.25 ppm. Formula assignments were also made on each blank spectrum to identify any potential contaminants, which were defined as any assigned peaks with a peak height greater than 5% of the highest assigned peak in each blank spectrum. The formulae corresponding to these peaks were removed from sample mass lists.

Python was used to apply a mass filter to the triplicate samples where any formula that appeared in only one replicate was removed. Average mass lists were produced from triplicate samples with each mass given an average peak height form the triplicate samples. These mass lists were analysed in Python, using PyKrev 1.2.4, and UpSet Plot 0.8.0 (Kitson et al., 2021; Seabold et al., 2010). Pykrev automatically removes isotopologues from mass lists. Attributes for each sample were determined, including average numbers of individual elements compound classes determined using both constituent elements and multidimensional stoichiometric compound classification (MSCC) (Rivas-Ubach et al., 2018).

Sample	Assigned Formula	Mean Mz	Mean C	Mean H	Mean N	Mean O	Mean S	OC	HC	DBE	AI	NOSC

Low wines-Average	2247	341.3819	17.9226	25.6088	0.2158	5.8567	0.1504	0.3687	1.4416	6.2261	0.1417	-0.6344
Fraction 1-Average	1715	359.5482	19.4781	31.3394	0.4204	5.4309	0.07755	0.3142	1.5913	5.0186	0.0911	-0.8856
Fraction 2-Average	2651	362.6694	18.6137	27.4123	0.5462	6.4085	0.07922	0.3860	1.4608	6.1807	0.1165	-0.5709
Fraction 3-Average	1803	339.7301	18.1503	26.4908	0.3644	5.5080	0.09484	0.3389	1.4766	6.0871	0.1389	-0.7192
Peated-Average	2700	368.7842	19.0863	30.2252	0.2037	6.4204	0.1485	0.3845	1.5702	5.0756	0.0665	-0.7291

Sample	**Lipid %**	**Carbohydrate %**	**Amino sugar %**	**Peptide %**	**Tannin/Lignin like %**	**CHO %**	**CHOS %**	**CHON %**	**CHNS %**	**CHS %**
Low wines-Average	49.9778	1.6911	0.2670	4.4949	37.6502	76.4130	13.6182	8.4557	0.1780	1.2461
Fraction 1-Average	70.2041	0.9329	0.1749	3.7318	21.8076	75.3353	5.9475	16.7930	0.5248	1.2828
Fraction 2-Average	49.1890	1.5089	0.5281	9.0155	34.3644	68.6533	6.4881	23.3497	0.5281	0.9053
Fraction 3-Average	53.0227	0.9429	0.3328	5.6572	36.4947	75.8181	8.0976	14.6977	0.3328	1.0538
Peated-Average	60.8889	3.1481	0.3333	5.1852	23.0000	76.7037	14.5556	8.3703	0.1852	0.1111

Table S1. Summary of attributes for the average spectra determined using PyKrev. Assigned formula excludes isotopologues. Mean elemental counts are the average amount per formula. OC and HC are elemental ratios. DBE = Double bond equivalents, AI = Aromaticity index calculated using 'rAImod' in PyKrev. NOSC = Nominal oxidative state of carbon. Lipid, Carbohydrate, Amino sugar, and Oxy-aromatic classes are assigned with MSCC.

Sample	**Calibration Points**	**Calibration RMS**	**Assigned Peaks**	**Percentage Relative Abundance**	**Assignment RMS**	**Contaminants Removed**
Spruce low	27	0.150667	3729	59.6073	0.12492	272

wines-1						
Spruce low wines-2	22	0.138726	3071	58.2558	0.11633	272
Spruce low wines-3	25	0.321008	3499	59.8082	0.12351	272
Spruce fraction 1-1	40	0.221830	4942	40.4059	0.13794	206
Spruce fraction 1-2	15	0.148770	2529	50.2861	0.12150	266
Spruce fraction 1-3	10	0.181170	2186	48.7416	0.13280	238
Spruce fraction 2-1	37	0.220510	3550	35.6446	0.14080	234
Spruce fraction 2-2	31	0.139983	4672	45.8117	0.13115	258
Spruce fraction 2-3	19	0.102657	3404	33.6696	0.11234	265
Spruce fraction 3-1	30	0.135771	3870	31.8047	0.12782	271
Spruce fraction 3-2	15	0.387611	2375	27.7162	0.13162	251
Spruce fraction 3-3	17	0.248542	2649	30.5255	0.12399	262
Peated-1	31	0.171134	3378	69.8346	0.12198	33
Peated-2	28	0.129888	4393	71.1742	0.121812	33
Peated-3	29	0.156082	5116	72.52873	0.119098	31

Table S2. Summary of assignments. Calibration points are the number of matched calibration points with the spectrum. RMS = Root Mean Square Error, assigned peaks include isotopologues.

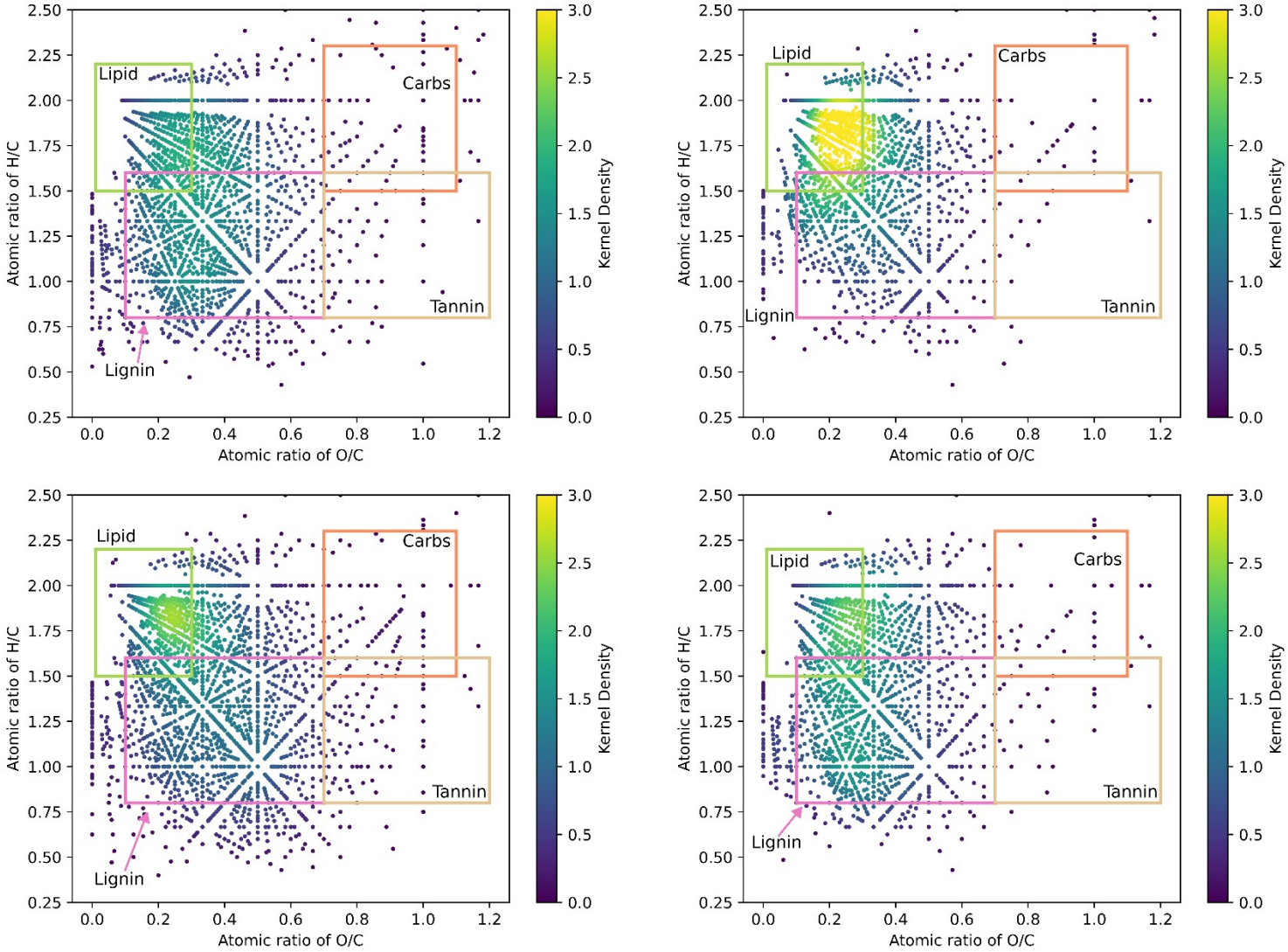

Figure S1. Van Krevelen diagrams of spruce smoked low wines (top left) and spruce smoked new make spirit fractions (fraction 1- top right, fraction 2 – bottom left, fraction 3 – bottom right)

New make spirit production

Green malt

Green malt used for new make spirit production was supplied by the Scotch Whisky Research Institute. Bulk samples of KWS Sassy barley was steeped and germinated in the laboratory to produce green malt. The steeping and germination conditions are shown in Table S3. After the end of the last germination step the green malt was stored in the freezer.

Step	Time (h)	Temperature (°C)
1st Steep	8	17
1st Rest	16	17
2nd Steep	24	17
Germination	96	17

Table S3. Steeping and germination conditions to produce green malt.

Malt smoking

Frozen green malt was thawed overnight in a fridge and then allowed to equilibrate to ambient temperature prior to use in smoking experiments. 25 g of peat (provided by a maltster who had sourced it from Bowmore, Islay) or spruce wood chippings was used to smoke 250g of green malt until smoking was complete (0.5 h) using a Carbolite EHA 12/150B horizontal tube oven (Fig. S2). Peat or spruce samples were placed inside the oven via a quartz glass sample boat. The oven has been modified at the chimney end to have a 90° elbow pipe attached to a quartz glass tube which holds the green malt. Air passes into the system via an air inlet from a benchtop air tap. The flow of air can be controlled from this tap and is measured by using an anemometer. The combustions were carried out at the same settings across all smokings; 600°C with an air flow of 2.8 m/s. Once the green malt samples had been smoked they were kilned at 50°C for 24hr. To produce enough malt for processing into spirit in duplicate, these smokings were carried out a total of 8 times for the laboratory spruce smoked malt, and the laboratory peated malt.

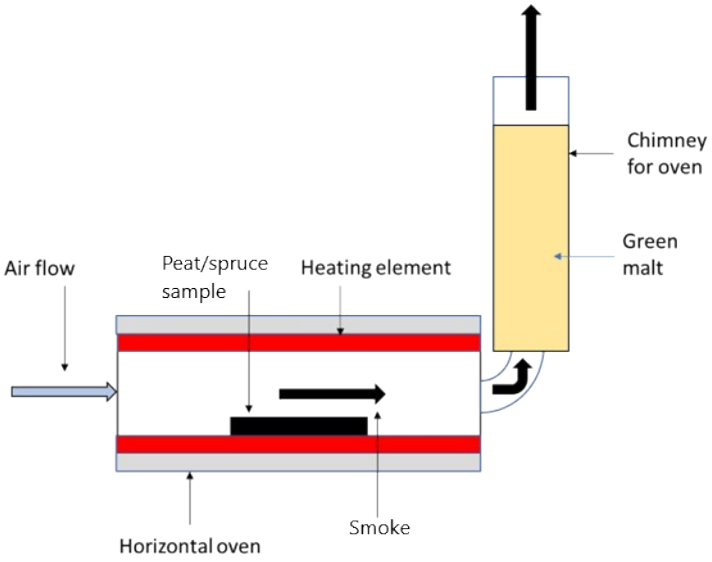

Figure S2. Diagram of the SWRI laboratory scale smoking apparatus.

Mashing

A 128 g sample of malt was weighed into a milling beaker and milled using a Buhler Miag Universal Laboratory Disc Mill (Buhler GmbH, Braunschweig, Germany, Post fach 3369, D-38023) set at 0.2 mm gap width using a feeler gauge. The resultant grist was immediately placed into a tared container and 125 g (± 0.001 g) weighed out. This process was repeated four times to obtain a total of 500 g of grist from each malt sample.

Each 125 g aliquot of grist was transferred as quantitatively as possible to a mashing beaker and placed in a 1-Cube R8 Mashing Bath at 65 °C. Distilled water was heated to 68 °C and 325 mL added to the grist in the mashing beaker, carefully rinsing any excess flour in the grist container. Each mash was stirred thoroughly using a glass rod to eliminate any lumps and covered with aluminium foil. The mash temperature was maintained at 65 °C for 1 h with regular stirring.

The mash was then transferred to a polypropylene centrifuge bottle and spun at 1200 g (2000 rpm) for 6 min in a Sanyo MSE Mistral 3000E centrifuge (MSE (UK) Limited, London, UK, SE26 5AZ). The supernatant was decanted and filtered under vacuum through a Buchner filter fitted with an unbleached Classic Calico cotton filter (John Lewis plc., London, UK, SW1E 5NN) into a 2-L Buchner flask. The residual grains were then transferred to the filter funnel and filtered under vacuum to apparent dryness. A glass reagent bottle stopper was used to tamp down the bed and maintain the vacuum. The resulting filtrate was cooled to ambient temperature and transferred to a 3L round bottom flask. The dried grains were returned to the mashing beaker and placed in the water bath which had been heated to 80 °C. The centrifuge bottle was rinsed with 200 mL boiling distilled water and the hot washings added to the grains as second water. The grains were extracted at 80 °C for 30 min, and then filtered under vacuum through the Buchner apparatus as described previously. The resulting filtrate was cooled and added to the 3L round bottom flask. The grains were returned to the mashing beaker and the beaker placed in a water bath at 100 °C and allowed to equilibrate for 5 min. Subsequently, 150 mL boiling water was added to the grains and extracted at 100 °C for 10 min. The grains were then filtered as quickly as possible in order to maximise elution of wort sugars before cooling. An appropriate volume was added to the wort in the fermenter in order to achieve a total wort volume of 2200 mL.

Fermentation

Once all the wort had been collected, 8.00 g of caked M-type distillers yeast from Kerry Biosciences (Kerry Group, Tralee, County Kerry, Ireland, V92 EH11) was pitched into the wort to give an expected pitching rate of 1-2 x 10^7 cells/mL. The fermentation flask was then sealed with a fermentation lock and placed in a water bath with a temperature programme rising from 19 °C to 33 °C over 72 hours. The resulting wash was frozen until distillation. Fermentation for each sample was performed in duplicate.

Distillation

The fermented wash was distilled using lab-scale copper stills as previously described in detail by Harrison et al.[1] Prior to distillation, the frozen wash was rapidly thawed in a sink of hot water. The still was then charged with 1.65 L of wash and 550 mL of low wines were collected. 50ml of the low wines was retained for analysis. The remaining 500 mL was charged to the spirit still and the following cut-points were used; 3 consecutive 100 mL new make spirit fractions. For the wash distillation, 10 drops of Foamdoctor F2887 were added to the still to reduce foaming. Distillation for each sample was performed in duplicate.

TGA

Samples

Sample	Mass/ mg	Temperature program	N_2 flow
PI001	82.7079 mg	(10)800[10](10)RT	100 cm^3/min
PA001	93.5400 mg	(10)800[10](10)RT	100 cm^3/min
SB001	39.9144 mg	(10)800[10](10)RT	100 cm^3/min
SNB001	43.2736 mg	(10)800[10](10)RT	100 cm^3/min

Table S4. TGA sample details.

Graphs

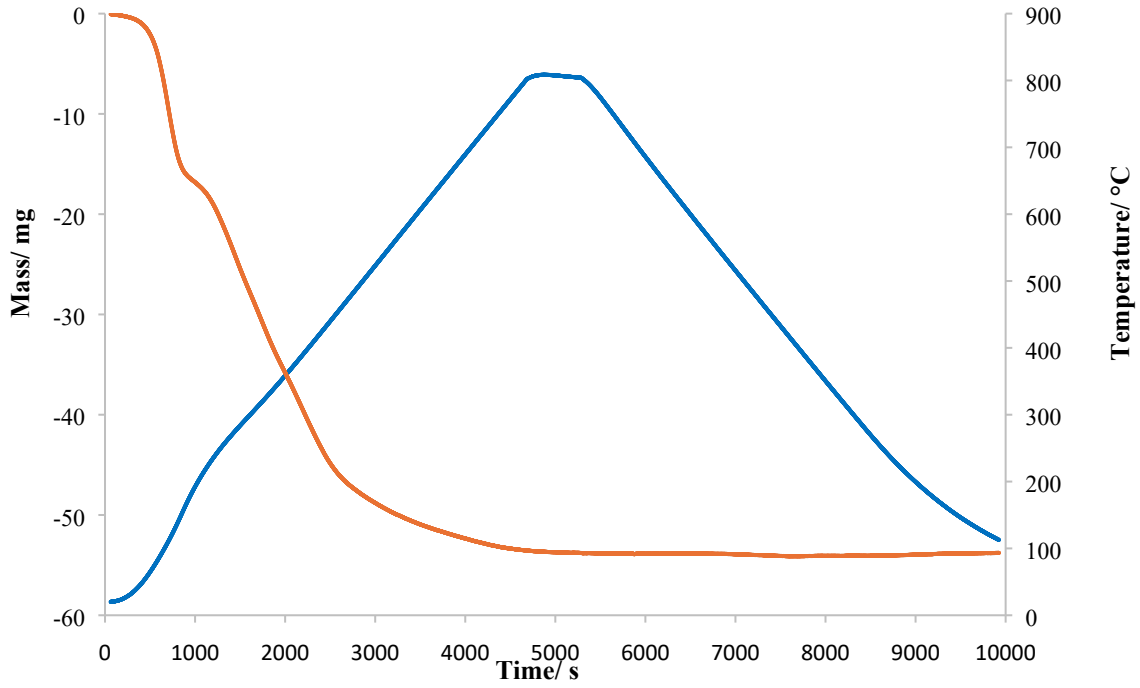

Figure S3. Islay peat TGA mass lost and temperature against time.

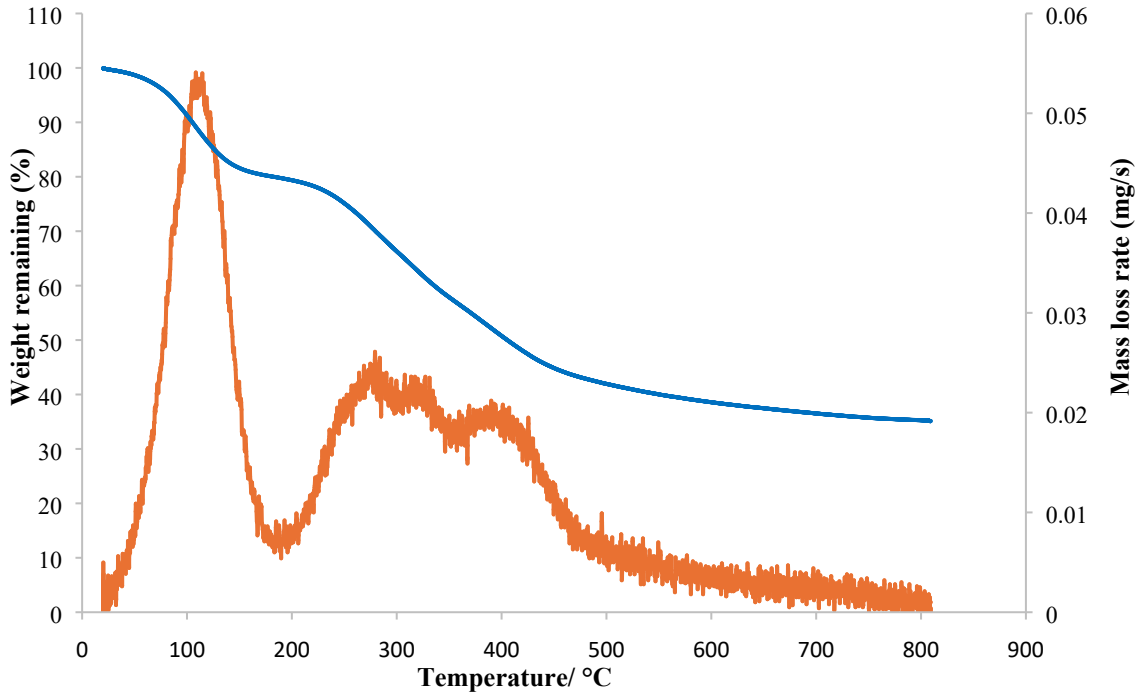

Figure S4. Islay peat TGA weight remaining and mass loss rate against temperature.

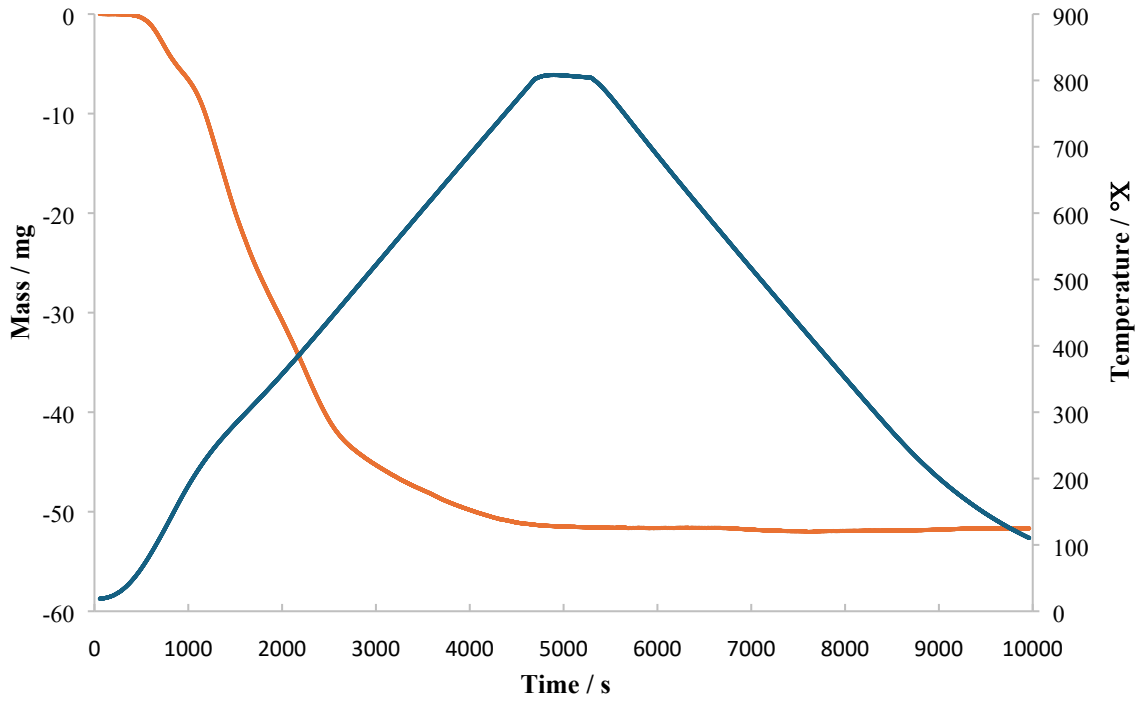

Figure S5. Aberdeenshire peat TGA mass lost and temperature against time.

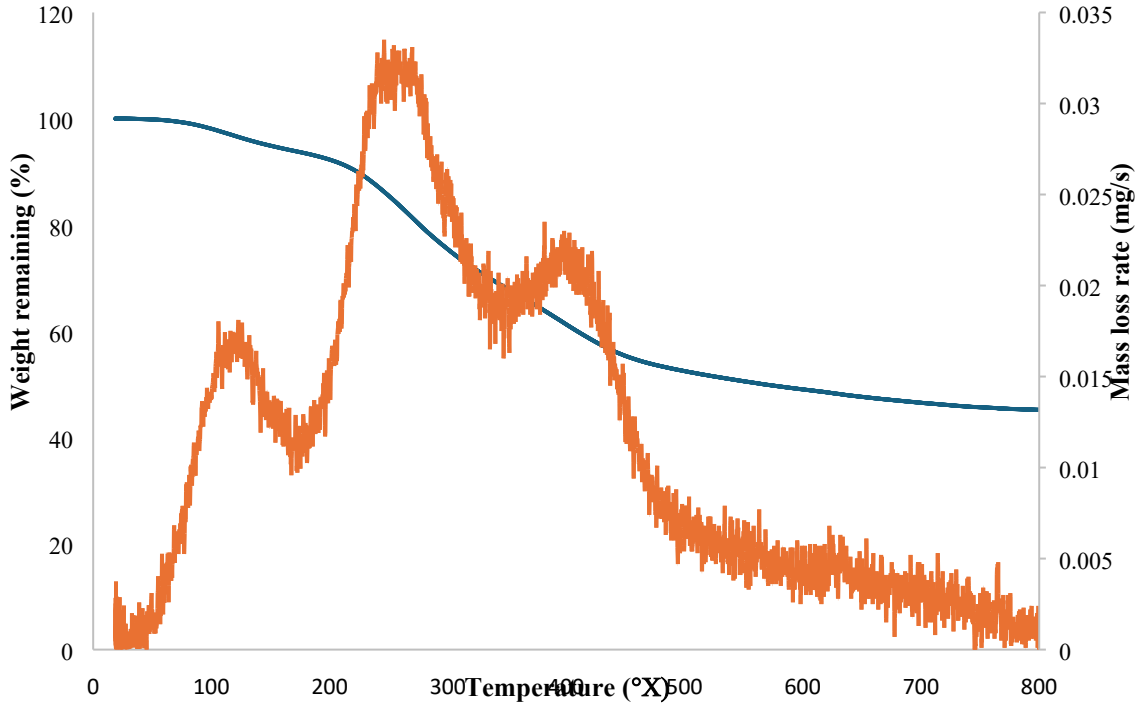

Figure S6. Aberdeenshire peat TGA weight remaining and mass loss rate against temperature.

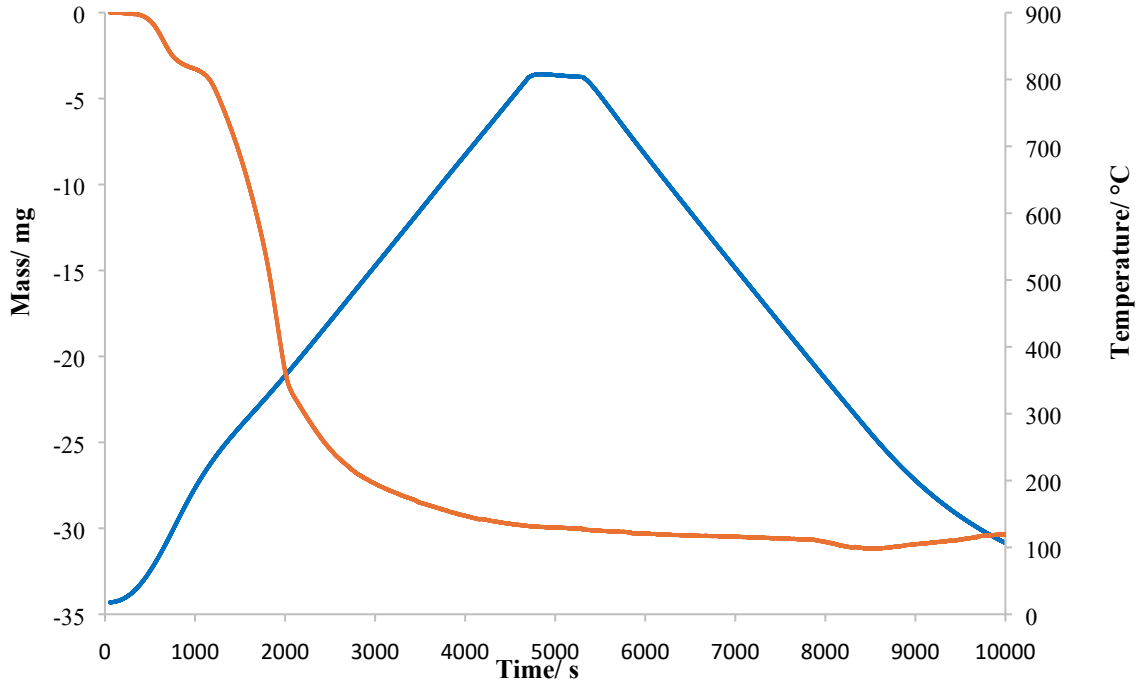

Figure S7. Spruce with bark TGA mass lost and temperature against time.

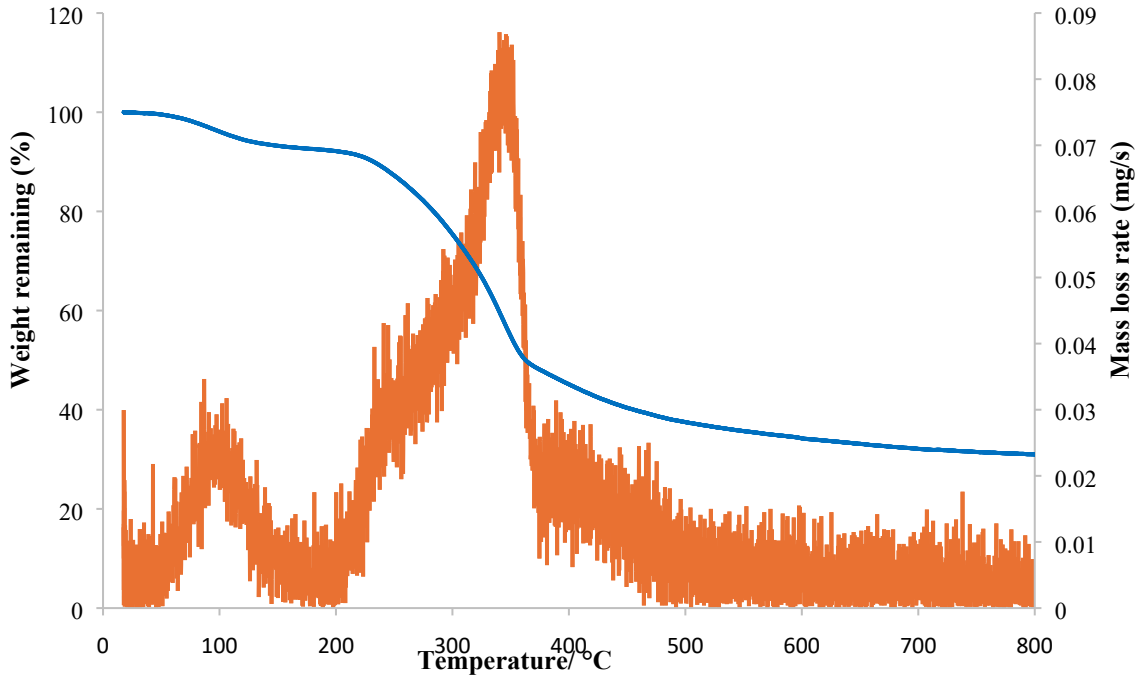

Figure S8. Spruce with bark TGA weight remaining and mass loss rate against temperature.

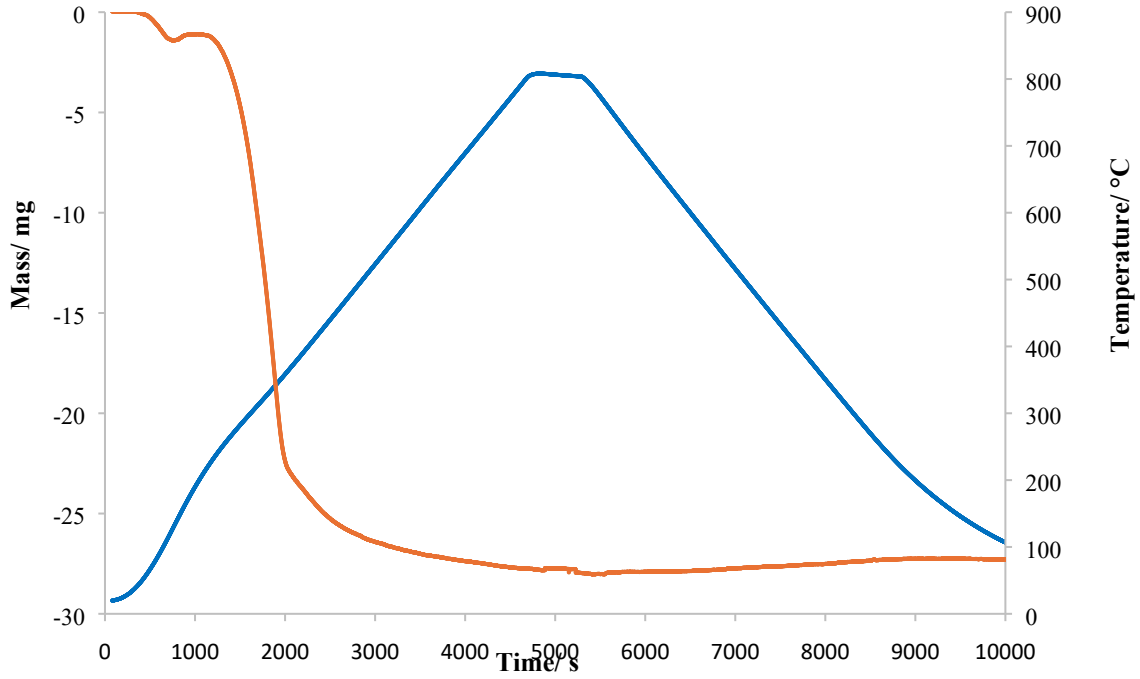

Figure S9. Spruce without bark TGA mass lost and temperature against time.

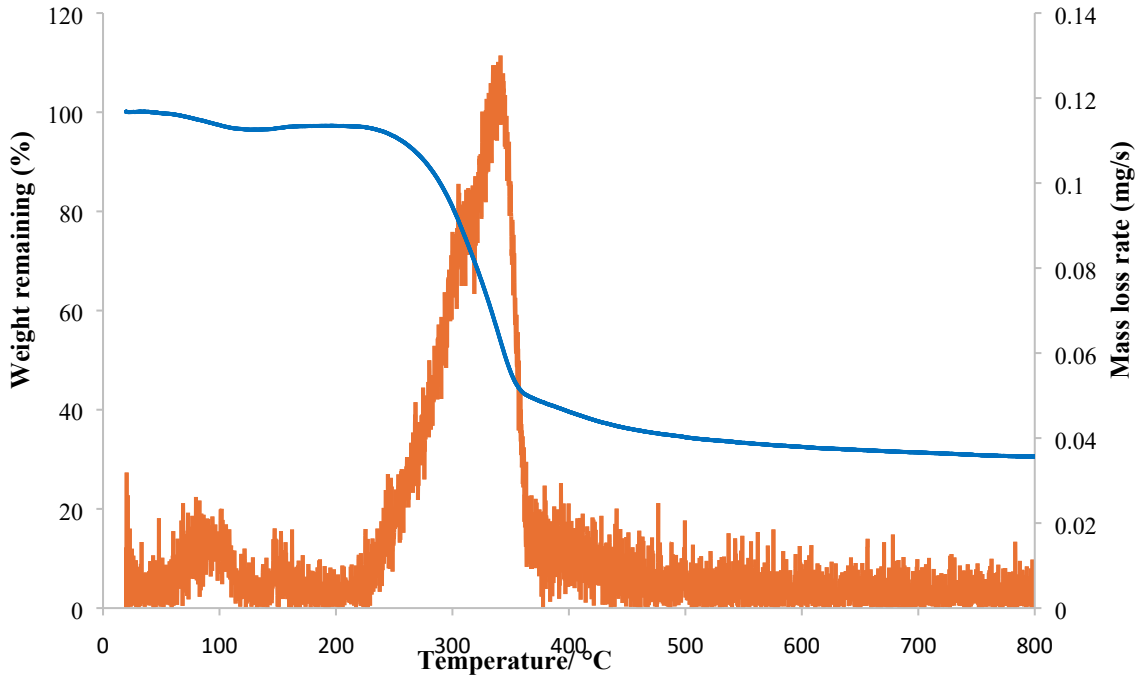

Figure S10. Spruce without bark TGA weight remaining and mass loss rate against temperature.

PyGCMS

Samples

Sample	Mass/ mg	Pyrolysis temperature/ °C	Description
PI1	0.533	550	Islay peat, 1:250 split
PA4	0.520	550	Aberdeenshire peat, 1:100 split
SB2	0.521	550	Spruce with bark, 1:250 split
SNB2	0.494	550	Spruce no bark, 1:250 split

Table S5. PyGCMS sample details.

Signal assignments

RT/min	Name	m/z	Molecular Weight
1.55	Carbon dioxide	44	44
1.81	Acetic acid	43,44,45,60	60
1.94	Hydroxyacetone	43,44	74
2.25	Unknown	67,79	-
2.35	Toluene	91,92	92
2.48	2(3H)-Furanone	84,54	84
2.71	Furfural	95,96	96
3.10	*n*-C9 Alkene	56,55,126*	126
3.20	Styrene	104,103	104
3.49	1,2-Cyclopentanedione	98,55	98
3.65	2-Pentenoic acid, 3-methyl-2,5-furandione	55,68,100,39	100, 112
3.86	5-Methylfurfural	110,109	110
4.02	$C_5H_6N_2O$	110,68	110
4.08	Phenol	94,66	94
4.27	4-Hydroxy-5,6-(2H)-pyran-2-one	114,58	114
4.45	Unknown	107,95	-
4.51	Unknown	112,52	-
4.65	Corylone	112,69	112
4.77	Dianhydrorhamnose	128,113	128
4.86	Indene	115,116	116
4.96	*o*-Cresol	108,107	108
5.13	Acetophenone	105,77	120
5.21	*p*-Cresol	107,108	108
5.34	n-C11 Alkene, Furaneol	43,55,128*	154, 128
5.40	Guaiacol	109,124	124
5.61	2,6-Xylenol	122,107	122
5.66	2-Methylbenzofuran	131,132	132
5.77	Maltol	126,71	126
6.06	Benzyl nitrile	117,90	117
6.16	2,5-Xylenol	122,107	122
6.20	Unknown	115,130	-
6.31	Methylindene	130,115	130
6.42	4-Ethylphenol	107,122	122
6.57	Octanoic acid	60,73	144

6.65	*n*-C12 Alkene	41,55	168
6.78	4-Methylguaiacol	138,123	138
6.87	3,5-Dihydroxy-2-methyl-4H-pyran-4-one	142,68	142
7.12	1,4:3,6-Dianhydro-α-d-glucopyranose, 2-hydroxymethylcyclohexanone	110,69	144, 128
7.17	4-Vinylphenol	120,91	120
7.38	4-Ethyl-2-methylphenol	121,136	136
7.53	Unknown	134,104	-
7.63	Hexylbenzene	91,92,162*	162
7.74	Unknown	105,152	-
7.81	Methoxypyrocatechol	140,125	140
7.85	Unknown	129,140	-
7.94	4-Ethylguaiacol	137,152	152
8.01	*n*-C13 Alkene	41,55,182*	182
8.05	1-Indanone	104,132	132
8.12	*n*-C13 Alkane	57,71,184*	184
8.21	Unknown	117,142,141	-
8.43	4-Vinylguaiacol	150,135	150
8.54	Unknown	138,144	-
8.62	Unknown	43,97	-
8.83	4-Allylphenol	133,134	134
8.92	Syringol	154,139	154
8.98	Eugenol	164,149	164
9.02	2-Benzyl-3-hydroxybutanoic acid	91,92	194
9.05	Unknown (likely 2 compounds)	131,132	-
9.10	4-Propylguaiacol	137,105	166
9.13	Unknown	154,139	-
9.17	Unknown	191	-
9.22	Aliphatic compound	81,55	-
9.33	*n*-C14 Alkene	83,55	196
9.43	*n*-C14 Alkane, 5-Methyl-1H-indole	57,71,130,131	198,131
9.49	*cis*-Isoeugenol	164,149	164
9.57	Unknown	123,164	-
9.65	Vanillin	151,152	152
9.90	Dimethylnaphthalene	156,141	156
10.15	4-Methylsyringol	168,153	168
10.20	*trans*-Isoeugenol	164,149	164
10.30	Unknown (likely 2 compounds)	55,41,141,156	-
10.40	Unknown	107,146	-
10.50	Aliphatic compound	55,41	-
10.60	*n*-C15 Alkene	83,97	210
10.69	*n*-C15 Alkane	57,71	212
10.76	Apocynin	166,151	166
10.89	Unknown	121,95	-
11.02	Unknown	161,107	-
11.10	Unknown	173,168	-

11.13	3,4,5-Trimethoxytoluene	182,167	182
11.27	Guaiacylacetone	137,180	180
11.62	3’,5’-Dimethoxyacetophenone	180,165	180
11.81	*n*-C16 Alkene	97,83	224
11.90	*n*-C16 Alkane	57,71	226
11.96	Trimethylnaphthalene	155,170	170
12.55	Levoglucosan	60,73	162
12.82	Decylbenzene	92,91,218*	218
12.97	*n*-C17 Alkene	97,83	238
13.05	*n*-C17 Alkane	57,71	240
13.21	*trans*-4-Propenylsyringol	194,179	194
13.39	Unknown	69,186	-
13.53	Aliphatic compound	83,97	-
13.65	4’-Hydroxy-3’,5’-dimethoxyacetophenone	196,181	
13.77	Acetophenone	181,196	196
13.81	Tetradecanoic acid	73,60,228*	228
13.98	Undecylbenzene	92,91,232*	232
14.03	Desaspidinol	167,210	210
14.06	*n*-C18 Alkene	97,83	252
14.13	*n*-C18 Alkane	57,71	254
14.18	Unknown	178,58	-
15.11	*n*-C19 Alkene	97,83	266
15.17	*n*-C19 Alkane	57,71	268
15.89	Hexadecanoic acid	73,60	256
16.11	*n*-C20 Alkene	97,83	280
16.17	*n*-C20 Alkane	57,71	282
17.06	*n*-C21 Alkene	97,83	294
17.12	*n*-C21 Alkane	57,71	296
17.71	Octadecanoic acid	73,60	284
17.88	*n*-C22 Alkene	97,83	308
17.91	*n*-C22 Alkane	57,71	310
18.48	*n*-C23 Alkene	97,83	322
18.51	*n*-C23 Alkane	57,71	324
18.96	*n*-C24 Alkene	97,83	336
19.37	*n*-C25 Alkene	97,83	350
19.73	*n*-C26 Alkene	97,83	364
20.10	*n*-C27 Alkene	97,83	378
20.43	*n*-C28 Alkene	97,83	392
20.81	*n*-C29 Alkene	97,83	406
21.20	*n*-C30 Alkene	97,83	420

Table S6. Compounds identified in Islay peat pyrogram obtained at 550 °C. *Ions with low intensity important for compound identification.

RT/min	Name	m/z	Molecular Weight
1.86	Carbon dioxide	44	44
1.95	Unknown	43,39	-
2.00	Acetic acid	43,45	60

2.06	3-Methylfuran	82,81	82
2.16	Hydroxyacetone	43,74	74
2.27	2,5-Dimethylfuran	96,95	96
2.42	Pyridine	79,52	79
2.53	Toluene	91,92	92
2.62	2(3H)-Furanone	84,54	84
2.71	3-Furaldehyde	95,96	96
2.75	2-Methylpyridine	93,66	93
2.83	Furfural	95,96	96
3.03	2-Propylfuran	81,53,110	110
3.05	Unknown	34,44,98	-
3.14	*p*-Xylene	91,106	106
3.17	Cyclopent-4-ene-1,3-dione	96,68	96
3.22	*n*-C9 Alkene	43,56	126
3.30	Styrene	104,103	104
3.32	Unknown	91,100	-
3.38	2(5H)-Furanone, Acetylfuran	95,55	84,110
3.45	Unknown	70,42	-
3.50	1,2-Cyclopentanedione	98,40	98
3.63	5,6-Dihydro-2H-pyran-2-one	68,39	98
3.77	Unknown	57,43	-
3.88	Hexahydro-3,6-pyridazinedione	55,86	114
3.98	Phenol	94,66	94
4.05	6-Methyl-4(1H)-Pyrimidinone	110,40	110
4.12	Unknown	97,105	-
4.16	*n*-C10 Alkene	41,56	140
4.22	α-Acetobutyrolactone	43,86	128
4.27	Unknown	114,58	-
4.36	1H-Pyrrole-2-carboxaldehyde	95,56	95
4.42	Corylone	112	112
4.48	Unknown	52,110	-
4.54	3-Methyl-1,2-cyclopentanedione	112,69	112
4.79	Dianhydrorhamnose	58,113,128	128
4.82	*o*-Toluidine	91	107
4.86	*o*-Cresol	108,107	108
4.91	Indene	115,116	116
4.99	Unknown	60,91	-
5.11	*p*-Cresol	107,108	108
5.17	2,5-Furandicarboxaldehyde	124,123	124
5.30	Furaneol	43,128	128
5.34	Guaiacol	109,124	124
5.46	*n*-C11 Alkane	57,43	156
5.64	Maltol	126,71	126
5.69	2-Methylbenzofuran	131,132,95	132
5.77	Cyclopent-4-ene-1,3-dione	96,40	96
5.81	4-Piperidone	99,57	99
5.92	5-Hydroxy-2-methyl-4H-pyran-4-one	126,69	126
6.02	2H-Pyran-2-one	96,40	96
6.08	2,5-Xylenol	107,122	122

6.30	4-Ethylphenol	107,122	122
6.37	Unknown	57,73	-
6.58	3,5-Dihydroxy-2-methyl-4H-pyan-4-one	142,68	142
6.67	Catechol	110,64	110
6.70	4-Methylguaiacol	123,138	138
6.79	*n*-C12 Alkane	57,43	170
6.83	Unknown	114,55	-
6.87	6-Methyl-3-pyridinol	109,81	109
7.01	4-Vinylphenol	120,91	120
7.09	5-Hydroxymethylfurfural	97,126	126
7.23	3,4-Anhydro-d-galactosan	69,71	144
7.28	4-Ethyl-2-methylphenol	121,136	136
7.34	Unknown	113,114	-
7.44	*cis*-1,2-Cyclohexanediol	42,43,116*	116
7.58	Cellulose degradation product	57,73	-
7.85	4-Ethylguaiacol	137,110,152	152
7.96	4-Methylcatechol	124,78	124
8.02	*n*-C13 Alkene	41,55	182
8.14	Indole, *n*-C13 Alkane	117,57	117,184
8.20	2-Hydroxy-6,8-dioxabicyclo[3.2.1]octan-4-one	87,144	-
8.34	2-Methoxy-4-vinylphenol	150,135	150
8.69	4-Allylphenol	134,133	134
8.79	Syringol	154,139	154
8.89	Eugenol	164,74	164
8.94	Unknown	121,154	-
9.09	1,2,3-Benzenetriol	126,74	126
9.20	Anhydrocarbohydrate	60,73	-
9.35	*n*-C14 Alkene	55,41	196
9.47	Vanillin	151,152	152
9.57	2-Methyl-5-hydroxybenzofuran	147,148	148
9.71	Inositol	73	180
9.75	2-Methyl-5-hydroxybenzofuran	147,148	148
9.79	Unknown	57,71	-
9.96	Acetophenone	121,136	136
10.01	4-Methylsyringol	168,153	168
10.09	Anhydrocarbohydrate	60,73	-
10.12	*trans*-Isoeugenol	60,164	164
10.39	*n*-C15 Alkene	55,41	210
10.56	Apocynin	151,166	166
10.89	Levoglucosan	60,73	162
11.06	Guaiacylacetone	137,180	180
11.24	Unknown	43,60	-
11.43	3-Hydroxy-4-methoxybenzoic acid	168,153	168
11.48	3',5'-Dimethoxyacetophenone	180,165	180
11.76	Butyrovanillone	151,123	194
11.83	*n*-C16 Alkene	55,43	224
11.92	1,6-Anhydro-β-D-glucofuranose	73,43	162

11.95	1,6-Anhydro-α-D-galactofuranose	73,69	162
12.58	Syringaldehyde	182,181	182
12.82	Tetrahydroactinidiolide	43,57	182
12.88	5-Ethynyl-1,2,3-trimethoxybenzene	192,177	192
12.98	*n*-C17 Alkene	55,83	238
13.06	*n*-C17 Alkane	57,71	240
13.27	Unknown	186,93	-
13.36	Unknown	56,57	-
13.40	Acetosyringone	181,196	196
13.52	Coniferyl alcohol	137,180	180
13.67	Tetradecanoic acid	73,60	228
13.75	Desapidinol	167,210	210
13.99	Unknown	91,92	-
14.07	*n*-C18 Alkene	83,97	252
14.14	*n*-C18 Alkane	57,71	254
15.11	*n*-C19 Alkene	83,97	266
15.18	*n*-C19 Alkane	57,71	268
16.11	*n*-C20 Alkene	83,97	280
16.17	*n*-C20 Alkane	57,71	282
17.05	*n*-C21 Alkene	83,97	294
17.11	*n*-C21 Alkane	57,71	296
17.81	*n*-C22 Alkene	83,97	308
17.84	*n*-C22 Alkane	57,71	310
18.34	*n*-C23 Alkene	83,97	322
18.36	*n*-C23 Alkene	83,97	322
18.38	*n*-C23 Alkane	57,71	324
18.43	*n*-C21 Ketone	58,59,310*	310
18.80	*n*-C24 Alkene	83,97	336
18.82	*n*-C24 Alkane	57,71	338
19.17	*n*-C25 Alkene	83,97	350
19.51	*n*-C26 Alkene	83,97	364
19.86	*n*-C27 Alkene	83,97	378
19.92	Aliphatic ketone	58,59	-
20.57	*n*-C29 Alkene	83,97	392
20.64	Aliphatic ketone	58,59	-
21.37	*n*-Alkane	57,71	-
21.50	Aliphatic ketone	58,59	-

Table S7. Compounds identified in Aberdeenshire peat pyrogram obtained at 550 °C. *Ions with low intensity important for compound identification.

RT/min	Name	m/z	Molecular Weight
1.54	Carbon dioxide	44	44
1.83	Acetic acid	45,43,60	60
1.90	Hydroxyaxcetone	43,74	74
2.36	Toluene	91,92	92
2.45	2(3H)-Furanone	84,54	84
2.69	Furfural	95,96	96

2.83	2-Furanmethanol	98,97	98
2.87	Acetoxyacetone	43,86	116
3.02	Xylene	91,106	106
3.10	Cyclopent-4-ene-1,3-dione	96,68	96
3.18	Styrene	104,103	104
3.32	2-Ethyl-5-methylfuran	110,95	110
3.40	2(5H)-Furanone	84,55	84
3.52	1,2-Cyclopentanedione	98,55	98
3.63	Unknown	68,83	-
3.77	Unknown	57,43	-
3.84	5-Methylfurfural	109,110	110
4.07	Phenol	94,66	94
4.27	Unknown	114,58	-
4.43	α-Terpinolene	121,136	136
4.53	*o*-Cymene	119,112	134
4.67	Corylone	112,55	112
4.77	Dianhydrhramnose	91,67	128
4.88	Unknown	57,69	-
4.94	*o*-Cresol	108,107	108
5.11	Cumene	105,120	120
5.21	*p*-Cresol	107,108	108
5.30	*n*-C11 Alkene	41,55,154*	154
5.39	Guaiacol	124,109	124
5.54	Unknown	44,57	-
5.64	2-Methylbenzofuran	132,131	132
5.80	Maltol	126,71	126
6.03	Benzyl nitrile	117,90	117
6.16	2,5-Xylenol	107,122	122
6.30	3-Hydroxy-4-methylbenzaldehyde, 2-methylindene	135,136,130	136,130
6.46	4-Ethylphenol	107,122	122
6.55	Heptanoic acid, Octenoic acid	60,55	130,142
6.63	*n*-C12 Alkene	83,97	168
6.77	4-Methylguaiacol	123,138	138
7.06	Catechol	110,64	110
7.18	4-Vinylphenol	120,91	120
7.32	2,3-Dimethoxytoluene	124,152,137	152
7.38	4-Ethyl-2-methylphenol	121,136	136
7.58	Unknown	97,126	-
7.62	2-Ethylbenzaldehyde	134,133	134
7.67	4-Propylphenol	107,121,136	136
7.82	4-Methylcatechol	124,78	124
7.92	4-Ethylguaiacol	137,152	152
7.98	*n*-C13 Alkene	83,97	182
8.12	2-Allylphenol	134,133	134
8.21	1-Methylnaphthalene, Indole	117,57	142,117
8.32	4-Methylcatechol	124,123	124
8.44	4-Vinylguaiacol	150,135	150
8.53	Unknown	138,109	-

8.80	4-Allylphenol	134,133	134
8.98	Eugenol	164,149	164
9.09	4-Propylguaiacol	137,166	166
9.21	Bicyclo[3.3.1]nonane	81,124	124
9.31	*n*-C14 Alkene	83,97	196
9.41	3-Methylindole	130,131	131
9.47	Unknown	149,135	-
9.56	Ethylresorcinol	123,138	138
9.63	*cis-I*soeugenol	164,149	164
9.66	Vanillin	151,152	152
9.77	2-Allyl-4-methylphenol	148,133	148
10.20	*trans*-Isoeugenol	164,149	164
10.31	Unknown	41,55	-
10.39	Unknown	107,146	-
10.58	*n*-C15 Alkene	83,97	210
10.66	6-Methoxy-3-methylbenzofuran	147,162	162
10.76	Apocynin	151,166	166
11.27	Guaiacylacetone	137,180	180
11.35	α-Calacorene	157,142	200
11.62	β-Calacorene	157,142	200
11.79	*n*-C16 Alkene	83,97	224
11.90	1,2-Dimethoxy-4-n-propylbenzene	151,180	180
11.95	Butyrovanillone	151,123	194
12.20	Levoglucosan	60,73	162
12.62	*n*-C17 Alkene	83,97	238
12.70	4-Hydroxy-3-methoxybenzenepropanol	137,182	182
12.80	*n*-C17 Alkene	69,83	238
12.86	3-Methoxy-2-naphthalenol	174,131	174
12.94	Unknown	83,97	-
12.99	Coniferyl alcohol	137,124	180
13.01	1'-Hydroxyeugenol	137,124	180
13.15	Unknown	137,149	-
13.21	Unknown	82,43	-
13.27	Aliphatic alkane	57,71	-
13.37	Unknown	56,69	-
13.40	Unknown	186,93	-
13.61	Unknown	70,83	-
13.72	Oplopanone	153,135	238
13.77	Coniferyl alcohol isomer	137,180	180
13.97	Unknown	81,95	-
14.05	*n*-C18 Alkene	83,97	252
14.11	*n*-C18 Alkane	57,71	254
14.54	Neophytadiene	68,95,278*	278
14.62	Unknown	43,123	-
14.80	Unknown	70,55	-
14.86	Pentadecanoic acid	73,60,242*	242
14.92	Unknown	206,150	-
14.99	Hexadecanol	83,97	242

15.09	*n*-C19 Alkene	97,83	266
15.15	*n*-C19 Alkane	57,71	268
15.23	Tetradecylfuran	81,95,264*	264
15.36	Unknown	74,55	-
15.73	Heptadecanol	83,97,256*	256
15.91	Hexadecenoic acid	73,60,256*	256
16.09	*n*-C20 Alkene	83,97	266
16.24	Unknown	239	-
16.38	Unknown	197,239	-
16.55	Unknown	239,254	-
16.58	Unknown	213,185	-
16.63	Kaur-16-ene	55,83	272
16.92	Epimanool	137,81	290
16.98	Aliphatic alcohol	83,97	-
17.04	*n*-C21 Alkene	83,97	280
17.09	*n*-C21 Alkane	57,71	282
17.51	Linoleic acid	81,67	280
17.70	Octadecanoic acid	73,129	284
17.85	*n*-C22 Alkene	83,97	294
17.89	*n*-C22 Alkane	57,71	296
17.98	Unknown	157,239	-
18.05	Unknown	254	-
18.20	Unknown	109,123	-
18.45	*n*-C23 Alkene	83,97	308
18.49	*n*-C23 Alkane	57,71	310
18.56	Aliphatic ketone	58,59	-
18.66	Unknown	269,201	-
18.74	Unknown	83,280	-
18.81	Podocarpa-6,8,11,13-tetraen-15-oic acid	237,197	312
18.83	Methyl dehyroabietate	239,240	314
18.88	Eicosanoic acid	73,43,312*	312
18.93	Unknown	167,197	-
18.94	*n*-C24 Alkene	83,97	322
18.98	Dehydroabietol	253,271	286
19.36	*n*-C25 Alkene	83,97	336
19.83	7-Oxodehydroabietic acid, Methyl ester	253,328	328
20.09	*n*-C27 Alkene	83,97	364
20.73	Unknown	113,100	-

Table S8. Compounds identified in spruce with bark pyrogram obtained at 550 °C. *Ions with low intensity important for compound identification.

RT/min	Name	m/z	Molecular Weight
1.55	Carbon dioxide	44	44
1.71	Unknown	44,31	-
1.86	Acetic acid	31,45,60	60
2.01	Hydroxyacetone	43,31,74	74
2.26	1,2-Ethanediol, monoacetate	43,73	104
2.40	Unknown	58,43	-
2.68	Furfural	95,96	96

2.87	2,3-Dihydro-2,5-dimethylfuran	43,98	98
3.17	Styrene	104,103	104
3.31	2,4-Dimethylfuran	67,96	96
3.47	2(5H)-furanone	55,84	84
3.56	1,2-Cyclopentanedione	98,55	98
3.66	Unknown	68,83	-
3.88	5-Methylfurfural	86,109	110
4.07	α-Methylstyrene	41,118	118
4.13	Phenol	94,66	94
4.33	Unknown	114,58	-
4.70	Corylone	112,69	112
4.82	Salicylaldehyde	122,121	122
4.96	*o*-Cresol	108,107	108
5.25	*p*-Cresol	107,108	108
5.29	*n*-C11 Alkene	85,41	154
5.40	Guaiacol	109,124	124
5.64	2,6-Xylenol	44,57,122*	122
5.85	Maltol	126,71	126
6.17	2,5-Xylenol	107,122	122
6.36	Unknown	42,85	-
6.49	4-Ethylphenol,2,3-dihydroxybenzaldehyde	107,138	122,138
6.56	5-Methylguaiacol	123,138	138
6.63	*n*-C12 Alkene	41,55,168*	168
6.79	4-Methylguaiacol	123,138,95	138
6.99	5-Hydroxymaltol	142,43	142
7.22	Catechol	110,64	110
7.33	Dimethoxytoluene	152,137	152
7.37	4-Ethyl-2-methylphenol	121,136	136
7.72	5-Hydroxymethylfurfural	97,126	126
7.93	4-Ethylguaiacol	137,152	152
7.99	*n*-C13 Alkene	55,83	182
8.09	*n*-C13 Alkane	57,71	184
8.14	*p*-Isopropenylphenol	134,133	134
8.46	4-Vinylguaiacol	150,135	150
8.54	5-Methyl-2-allylphenol	148,133	148
8.79	Unknown	144,87	-
9.00	Eugenol	164,149	164
9.06	8,9-Dehydrothymol methyl ether, 1H-indenol	132,131,162	162,132
9.11	4-Propylguaiacol	137,166	166
9.31	*n*-C14 Alkene	83,97	196
9.40	*n*-C14 Alkane	57,71	198
9.57	4-Ethylcatechol	123,138	138
9.65	*cis*-Isoeugenol	164,149	164
9.73	Vanillin	151,152	152
9.80	6-Allyl-*o*-cresol	148,133	148
10.23	*trans*-Isoeugenol	164,149	164
10.43	Propylguaiacol isomer	137,166	166
10.58	*n*-C15 Alkene	83,97	210

10.67	*n*-C15 Alkane,6-methoxy-3-methylbenzofuran	147,162,57	212,162
10.82	Apocynin	151,166	166
10.99	4-Hydroxy-3-propylbenozic acid	151,180	180
11.09	4-Hydroxy-3-methoxybenzoic acid, methyl ester	151,182	182
11.32	Guaiacylacetone	137,180	180
11.38	Unknown	131,178	-
11.80	*n*-C16 Alkene	83,97	224
11.82	1'-Hydroxyeugenol	137,180	180
11.87	*n*-C16 Alkane	57,71	226
11.92	1-(2-hydroxy-4-methoxyphenyl)propan-1-one	151,180	180
11.97	Butyrovanillone	151,123	194
12.73	4-Hydroxy-3-methoxybenzenepropanol	137,182	182
12.86	Levoglucosan	60,57	162
12.94	*n*-C17 Alkene	83,97	238
13.02	*n*-C17 Alkane	85,71	240
13.76	Coniferyl aldehyde	178,135	178
13.85	Tetradecanoic acid	73,60	228
14.55	Unknown	200,157	-
14.78	Unknown	178,55	-
14.84	Pentadecanoic acid	73,60,242*	242
15.21	Heptadecanone	58,43,254*	254
15.43	Hexadecenoic acid, methyl ester	74,87,270*	270
16.08	Hexadecenoic acid	73,60,256*	256
16.83	Heptadecanoic acid	73,60,270*	270
16.93	Epimanool	81,95	290
17.19	Nonadecanone	58,43,282*	282
17.43	Methyl stearate	74,87	298
17.91	Octadecanoic acid	73,129,284	284
18.22	Retene	219,234	234
18.31	Unknown	246,256	-
18.64	1,4-Dimethyl-6-phenyl-naphthalene, eicosanoic acid, methyl ester	87,74	232,326
18.67	Unknown	272,273	-
18.75	Unknown	286,130	-
18.83	Unknown	137	-
18.84	Methyl dehydroabietate	239,240	314
18.90	Unknown	137	-
18.95	Unknown	97,83	-
19.00	Unknown	59,72	-
19.09	Unknown	151,161	-
19.16	Unknown	137,286	-
19.21	Unknown	98,43	-
19.28	Unknown	91,117	-
19.36	*n*-C25 Alkene	83,97	336
19.40	Unknown	137,286	-
19.78	Unknown	131,103	-

19.83	Unknown	253,131	-

Table S9. Compounds identified in spruce without bark pyrogram obtained at 550 °C. *Ions with low intensity important for compound identification.

Pyrograms

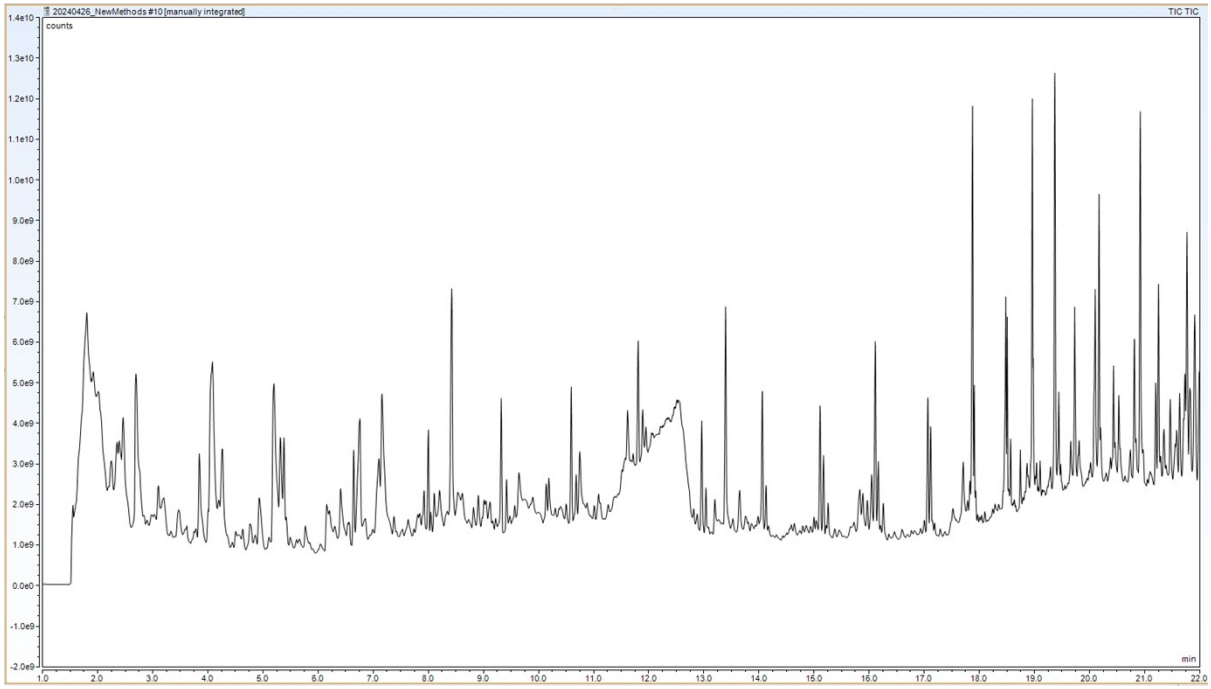

Figure S11. Islay peat pyrogram.

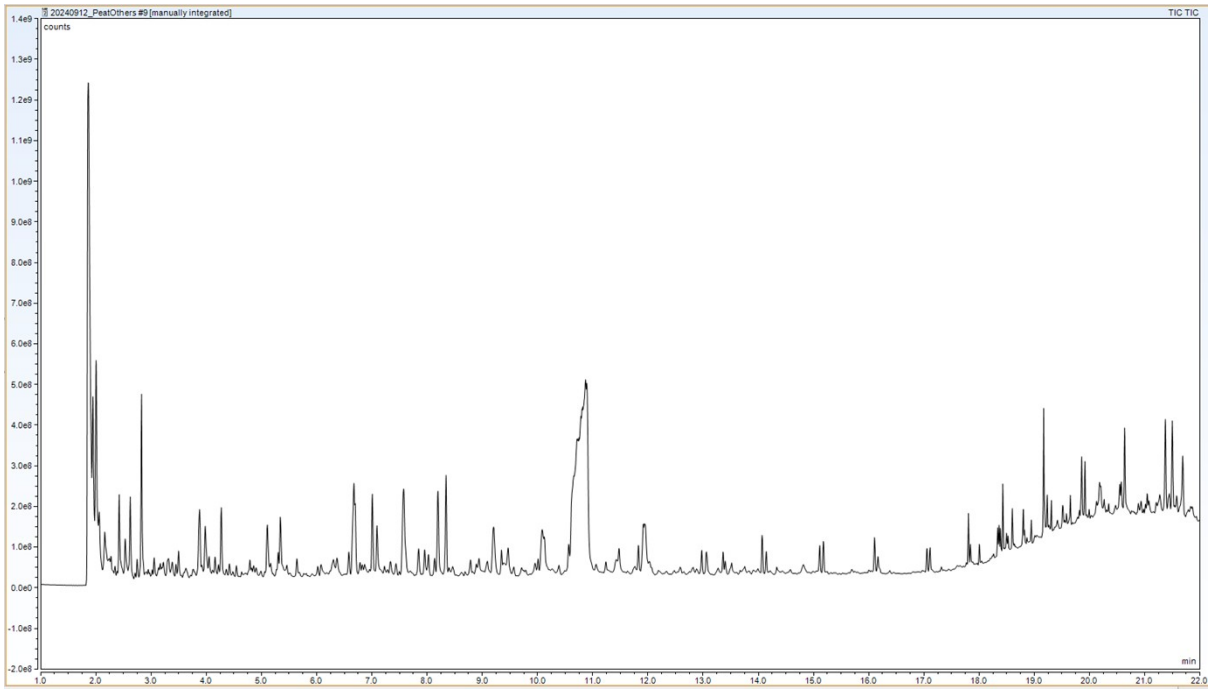

Figure S12. Aberdeenshire peat pyrogram.

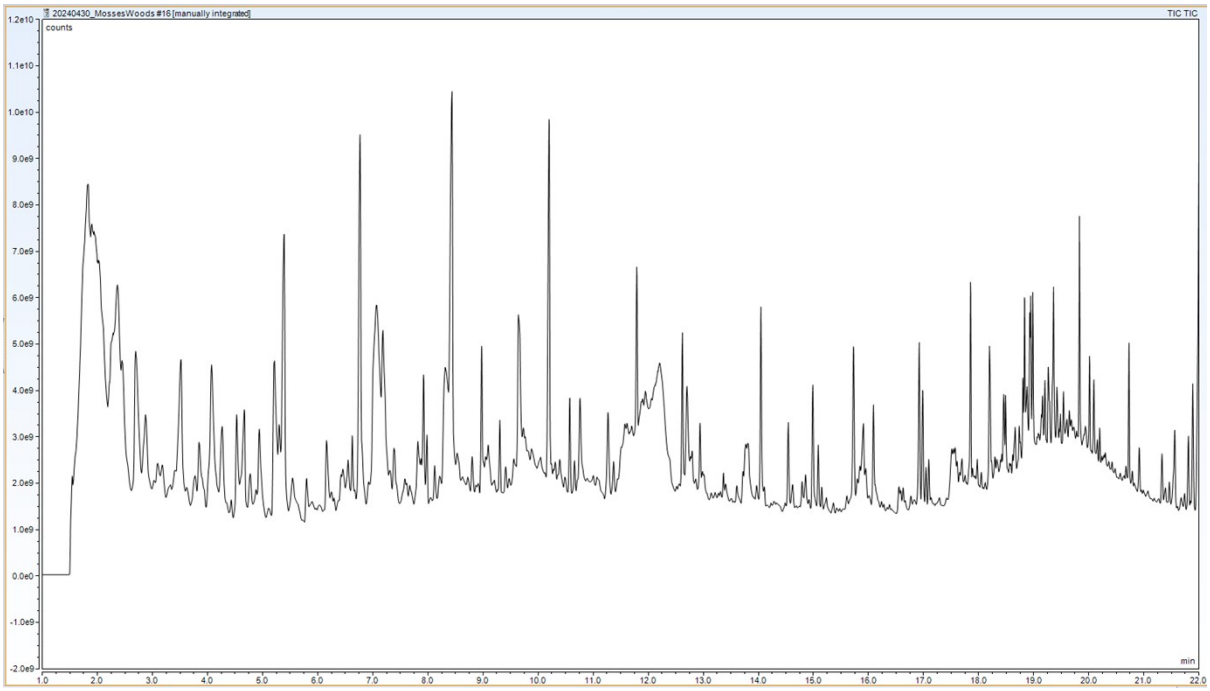

Figure S13. Spruce with bark pyrogram.

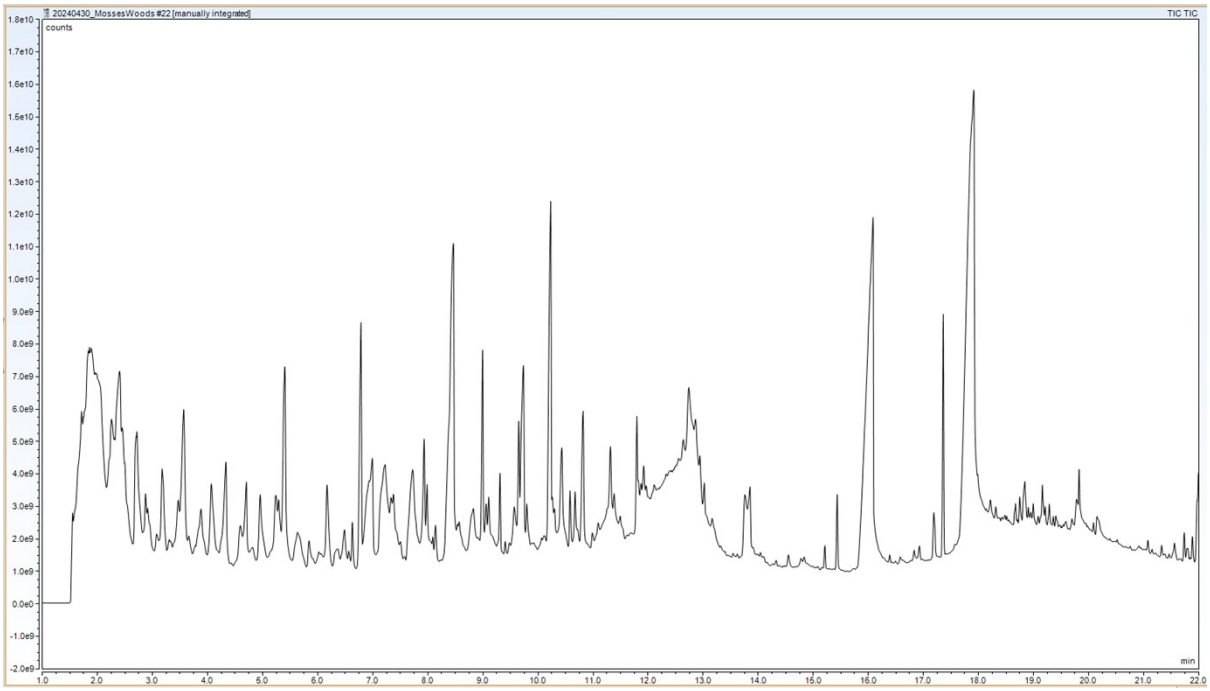

Figure S14. Spruce without bark pyrogram.

Sensory Analysis

Sensory Characteristics	p-value	PNMS1	PNMS2	SNMS1	SNMS2
Overall peat intensity	≤0.001	1.61 a	1.39 ab	0.80 c	1.11 bc
Burnt	≤0.001	1.19 a	1.14 ab	0.46 c	0.68 bc
Smoky	0.01	1.23 a	1.18 a	0.67 b	0.89 ab
Medicinal	0.67	0.46	0.49	0.40	0.34
Sulphury	0.77	0.19	0.25	0.21	0.13

Meaty	0.02	0.06 b	0.19 ab	0.15 ab	0.30 a
Feinty	0.57	0.49	0.58	0.54	0.43
Cereal	0.12	0.49	0.56	0.81	0.55
Green/ grassy	0.74	0.22	0.26	0.30	0.32
Floral	0.94	0.14	0.14	0.14	0.18
Fruity/estery	1.00	0.35	0.34	0.36	0.34
Solventy	0.29	0.30	0.31	0.15	0.24
Soapy	0.18	0.09	0.12	0.38	0.41
Sweet	0.38	0.18	0.14	0.24	0.19
Nutty	0.49	0.10	0.09	0.21	0.14
Oily	0.53	0.39	0.55	0.40	0.53
Sour	0.56	0.08	0.22	0.20	0.26
Stale	0.27	0.19	0.19	0.08	0.15

Table S10. Mean panel scores and results of ANOVA and Tukey's post-hoc tests (a,b,c represent sample groupings in Tukey's post-hoc test; $p<0.05$)

NMR

Sample	Description
PLW	Low wines produced using peated malt
PNMS1	First distillation fraction produced using peated malt
PNMS2	Second distillation fraction produced using peated malt
PNMS3	Third distillation fraction produced using peated malt
SLW	Low wines produced using malt smoked with spruce wood
SNMS1	First distillation fraction produced using malt smoked with spruce wood
SNMS2	Second distillation fraction produced using malt smoked with spruce wood
SNMS3	Third distillation fraction produced using malt smoked with spruce wood

Table S10. Details of samples analysed using NMR.

Spectra

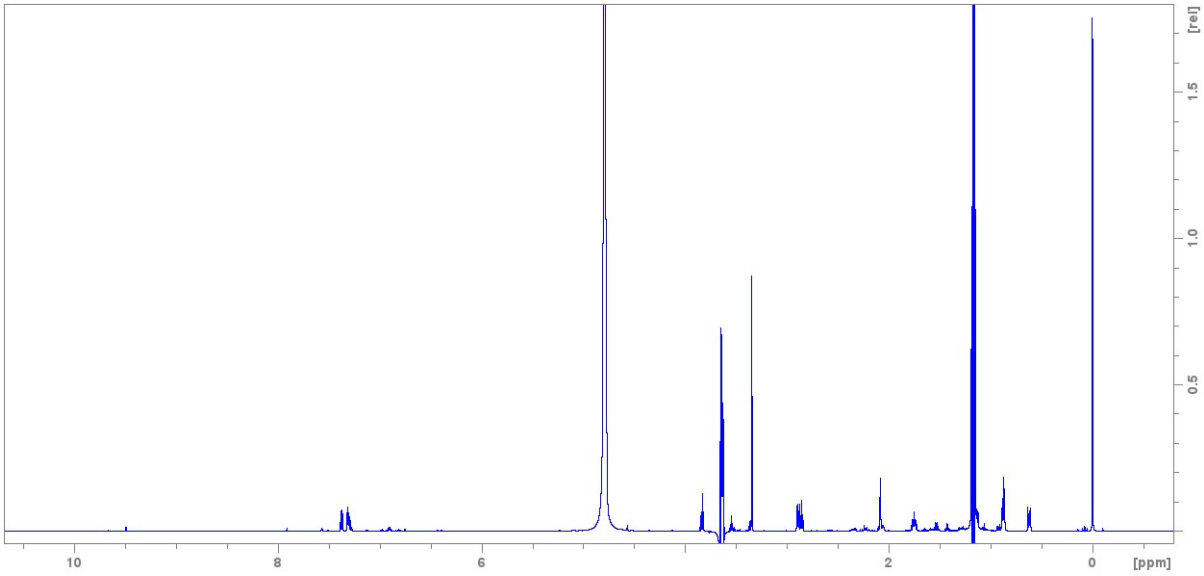

Figure S15. ^{1}H NMR spectrum of peated low wines recorded at 600 MHz.

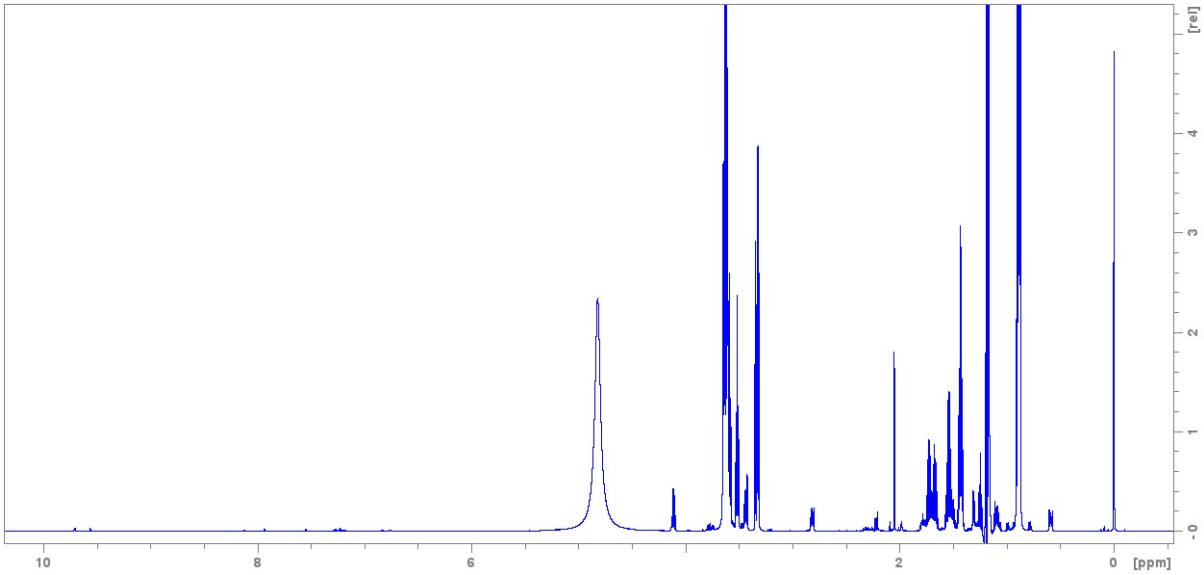

Figure S16. ^{1}H NMR spectrum of peated new make spirit fraction 1.

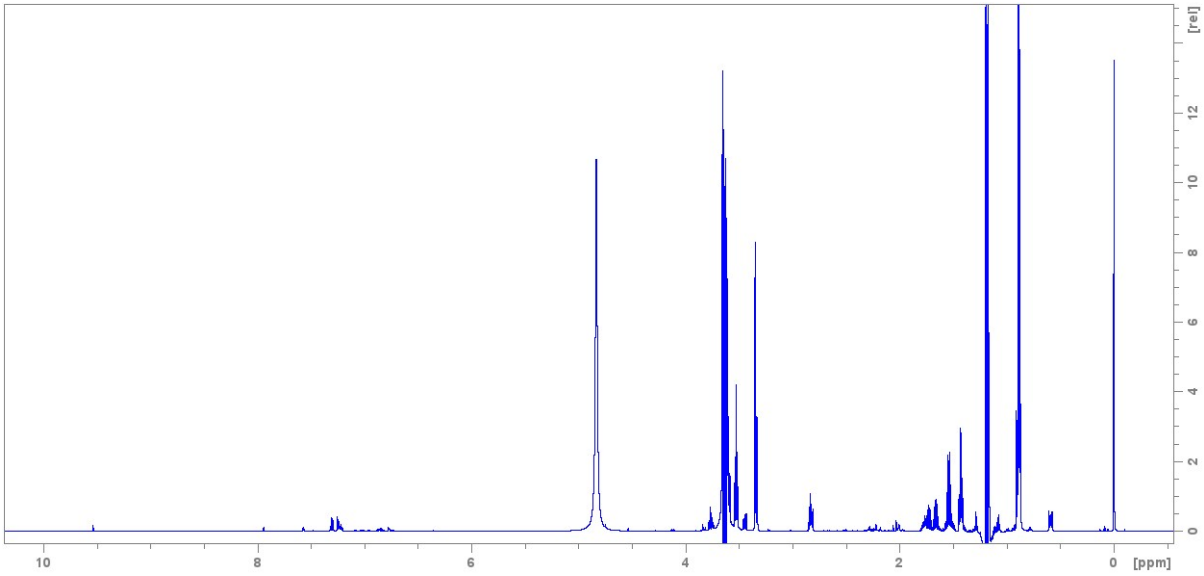

Figure S17. ^{1}H NMR spectrum of peated new make spirit fraction 2.

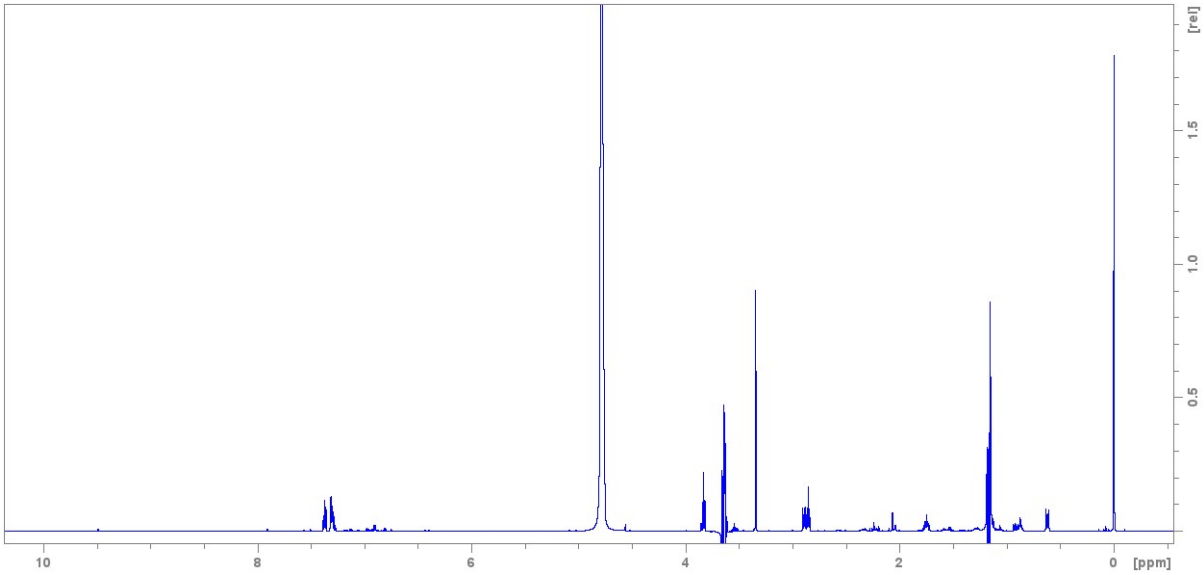

Figure S18. ^{1}H NMR spectrum of peated new make spirit fraction 3.

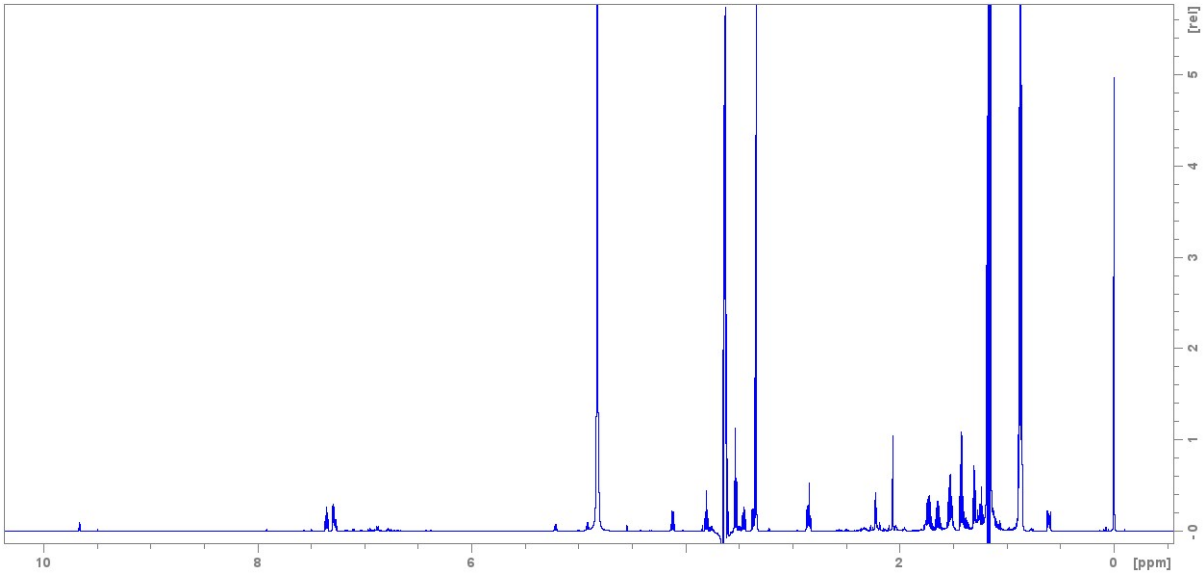

Figure S19. ^{1}H NMR spectrum of spruce smoked low wines.

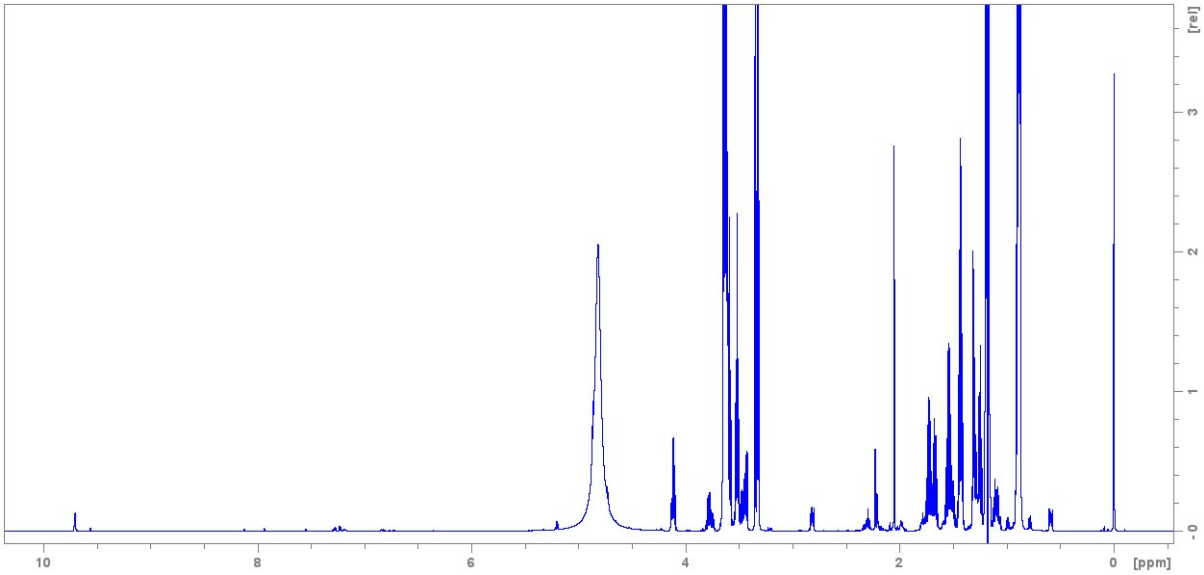

Figure S20. ^{1}H NMR spectrum of spruce smoked new make spirit fraction 1.

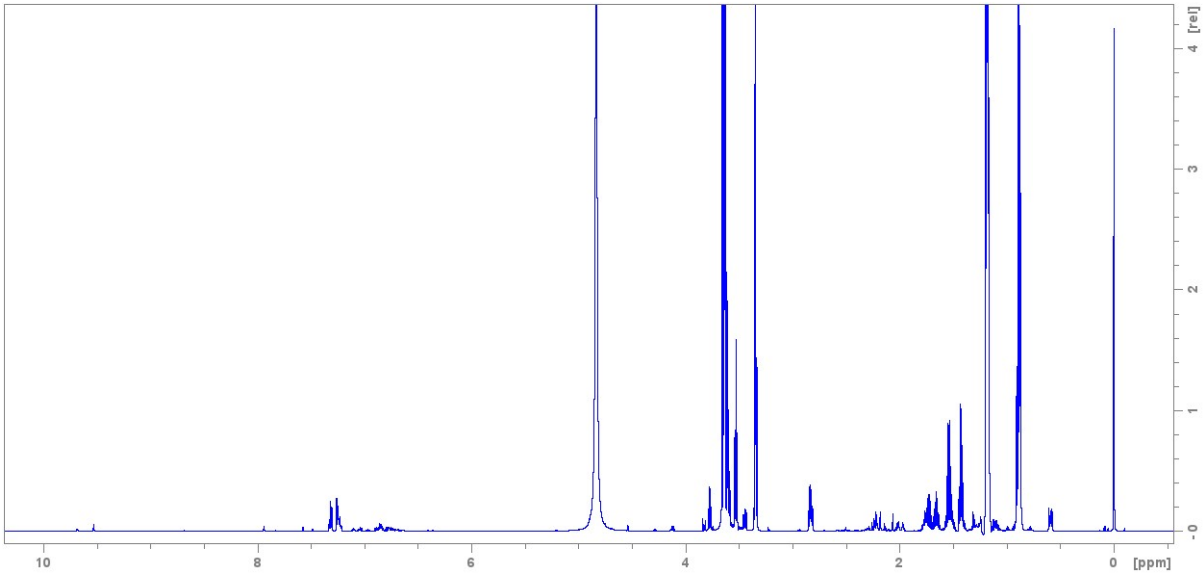

Figure S21. ^{1}H NMR spectrum of spruce smoked new make spirit fraction 2.

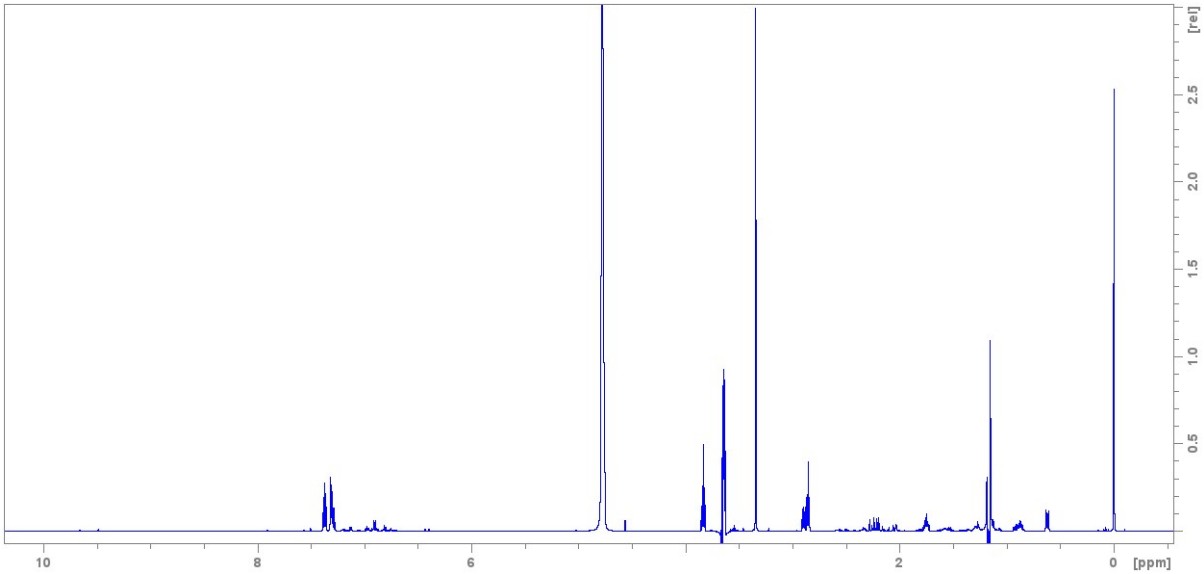

Figure S22. ^{1}H NMR spectrum of spruce smoked new make spirit fraction 3.

Signal assignments

δ_H / ppm	Multiplicity	Integration	Inferences	Concentration/ mmol L^{-1}
0	Singlet	10	DSS 3xCH_3	1.00
0.62	Multiplet	2.23	DSS CH_2	1.00
0.77	Multiplet	-	Fusel alcohols CH_3 groups carbon satellites	-
0.87	Multiplet	-	Fusel alcohols CH_3 groups	-
0.98	Multiplet	-	Fusel alcohols CH_3 groups carbon satellites	-
1.082	Triplet, J = 7.5 Hz	-	Fusel alcohol ethyl ester CH_3	-
1.17	Distorted	-	Ethanol CH_3 group, suppressed	-
1.24	Tripplet, J = 7.2 Hz	0.32	Ethyl acetate CH_3 next to CH_2	0.096

1.27	Triplet, J = 7.0 Hz	0.40	Fusel alcohol ethyl ester CH_3	-
1.32	Doublet, J = 5.3 Hz	0.17	Lactic acid CH_3 next to CH	0.050
1.43	Quartet, J = 6.9 Hz	0.41	3-methyl-1-butanol CH_2 next to CH and CH_2	0.18
1.53	Sextet, J = 7.1 Hz	0.80	n-propanol CH_2 next to CH_2 and CH_3	0.36
1.65	Nonet, J = 6.7 Hz	0.22	3-methyl-1-butanol CH next to 2xCH_3 and CH_2	0.20
1.75	Multiplet	2.39	DSS CH_2	1.07
2.00	Multiplet	-	Acetate esters	-
2.05	Multiplet	0.52	DSS	0.23
2.08	Singlet	2.14	Acetic acid CH_3	0.64
2.10	Singlet	0.12	Ethyl acetate isolated CH_3	0.035
2.20	Singlet	0.062	*o*-cresol CH_3	0.018
2.22	Singlet	0.068	Acetone 2xCH_3	0.010
2.23	Doublet, J = 2.9 Hz	0.13	Acetaldehyde CH_3	0.039
2.26	Singlet	0.039	*p*-cresol CH_3	0.012
2.33	Triplet, J = 7.3 Hz	0.27	Ethyl ester	-
2.86	Triplet, J = 6.7 Hz	1.56	2-phenylethanol CH_2 next to CH_2	0.70
2.95	Triplet, J = 6.7 Hz	0.012	2-phenylethyl acetate CH_2 next to CH_2	0.0053
3.22	Singlet	0.021	Methanol CH_3 carbon satellite	-
3.34	Singlet	4.31	Methanol CH_3 isolated	1.29
3.36	Doublet, J = 6.6 Hz	0.41	Isobutanol CH_2 next to CH and OH	0.18
3.38	Singlet	0.022	Part of 2-methylbutanol diastereotopic CH_2	-
3.39	Doublet, J = 6.6 Hz	0.055	Part of 2-methylbutanol diastereotopic CH_2	-
3.472	DD, J = 10.8 Hz, 5.9 Hz	0.088	Part of 2-methylbutanol diastereotopic CH_2	0.079
3.55	Triplet, J = 6.6 Hz	0.52	n-propanol CH_2 next to CH_2 and OH	0.24
3.64	Distorted	-	Ethanol CH_2 suppressed	-
3.83	Triplet, J = 6.8 Hz	1.43	Phenylethanol CH_2	0.64
3.86	Singlet	0.074	Guaiacol OCH_3	0.022
4.12	Quartet, J = 7.1 Hz	0.046	Lactic acid CH	0.041
4.56	Singlet	0.15	Furfuryl alcohol CH_2	0.067
4.79	Broad singlet	-	Water OH suppressed	-
5.23	Quartet, J = 5.3 Hz	-	Acetaldehyde water hemiacetal	-
6.39	Doublet, J = 3.2 Hz	0.036	2-methylfuran CH	0.032
6.43	DD, J = 3.1 Hz, 1.9 Hz	0.034	2-methylfuran CH	0.031
6.75	DD, J = 3.6 Hz, 1.6 Hz	0.12	Furfural CH next to 2xCH	0.11

6.80	Doublet, J = 8.5 Hz	0.072	o-cresol aromatic CH next to CH	0.065
6.90	Doublet, J = 7.9 Hz	0.29	Phenol 2xCH next to CH	0.13
6.97	Triplet, J = 7.3 Hz	0.14	o-cresol aromatic CH next to 2xCH	0.13
7.06	Doublet, J = 7.7 Hz	0.022	p-cresol 2xCH next to CH	0.078
7.28	Triplet, J = 7.3 Hz	0.47	Phenol 2xCH next to 2xCH	0.21
7.31	Doublet, J = 7.9 Hz	1.88	2-phenylethanol 2x aromatic CH	0.85
7.37	Triplet, J = 7.6 Hz	1.40	2-phenylethanol 2x aromatic CH	0.63
7.57	Doublet, J = 3.6 Hz	0.11	Furfural CH next to CH	0.099
7.91	Singlet	0.11	Furfural CH	0.097
9.49	Singlet	0.11	Furfural CHO	0.097
9.66	Quartet, J = 3.0 Hz	0.023	Acetaldehyde CHO	0.021

Table S11. Signal assignment for NMR spectrum of low wines produced using peated malt, recorded at University of Edinburgh using a 600 MHz spectrometer, PLW.

δ_H / ppm	Multiplicity	Integration	Inferences	Concentration/ mmol L^{-1}
0	Singlet	10	DSS 3xCH_3	1.00
0.59	Multiplet	2.21	DSS CH_2	0.99
0.78	Multiplet	1.24	Fusel alcohols CH_3 groups carbon satellites	-
0.89	Multiplet	200.05	Fusel alcohols CH_3 groups	-
1.00	Multiplet	1.37	Fusel alcohols CH_3 groups carbon satellites	-
1.09	Triplet, J = 7.5 Hz	-	Ethanol CH_3 carbon satellite	-
1.11	Triplet, J = 7.4 Hz	4.74	Fusel alcohol ethyl ester CH_3	-
1.18	Distorted	-	Ethanol CH_3 suppressed	-
1.25	Triplet, J = 7.1 Hz	7.43	Ethyl acetate CH3 next to CH_2	2.23
1.29	Singlet	1.24	Fusel alcohol ethyl ester CH_3	-
1.31	Doublet, J = 4.8 Hz	2.97	Lactic acid CH_3 next to CH	0.89
1.43	Quartet, J = 7.0 Hz	29.09	3-methyl-1-butanol CH_2 next to CH and CH_2	13.09
1.49	Multiplet	-	n-butanol CH_2 groups	-
1.54	Sextet, J = 7.2 Hz	19.6	n-propanol CH_2 next to CH_2 and CH_3	8.82
1.68	Nonet, J = 6.7 Hz	12.4	3-methyl-1-butanol CH next to 2xCH_3 and CH_2	11.2
1.73	Nonet, J = 6.6 Hz	11.5	Isobutanol CH next to 2xCH_3 and CH_2	5.18
1.78	Multiplet	1.77	DSS CH_2	0.80
1.94	Singlet	0.028	Aacetic acid CH_3 carbon satellite	-
1.99	Multiplet	-	Acetate esters	-
2.05	Singlet	4.86	Acetic acid CH_3	1.46
2.08	Singlet	0.054	Ethyl acetate CH_3 isolated	0.016
2.16	Singlet	0.032	Acetic acid CH_3 carbon satellite	-
2.18	Singlet	0.073	o-cresol CH_3	0.022

2.21	Singlet	0.49	Acetone 2xCH_3	0.074
2.23	Doublet, J = 2.9 Hz	0.65	Acetaldehyde CH_3 next to CH	0.19
2.30	Triplet, J = 7.4 Hz	0.63	Ethyl ester	-
2.82	Multiplet	2.24	DSS CH_2	1.01
2.93	Doublet, J = 7.7 Hz	0.032	2-phenylethyl acetate CH_2	0.014
3.21	Doublet, J = 6.7 Hz	0.16	Isobutanol CH_2 carbon satellite	-
3.23	Singlet	0.028	Methanol CH_3 carbon satellite	-
3.32	Doublet, J = 6.6 Hz	24.56	Isobutanol CH_2 next to CH and OH	11.05
3.35	Singlet	2.86	Methanol CH_3	0.86
3.44	DD, J = 10.6 Hz, 6.0 Hz	6.14	Part of 2-methylbutanol diastereotopic CH_2	2.76
3.48	Quartet, J = 7.1 Hz	-	Ethanol CH_2 carbon satellite	-
3.52	Triplet, J = 6.8 Hz	15.03	n-propanol CH_2	6.76
3.59	Triplet, J = 7.1 Hz	23.23	3-methyl-1-butanol CH_2 next to CH_2	10.45
3.63	Distorted	-	Ethanol CH_2 suppressed	-
3.75	Quartet, J = 6.8 Hz	-	Ethanol CH_2 carbon satellite	-
3.78	Quartet, J = 7.2 Hz	-	Phenylethanol CH_2	-
4.12	Quartet, J = 7.2 Hz	-	Lactic acid CH	-
4.13	Quartet, J = 6.8 Hz	-	Ethyl acetate CH_2 next to CH_3	-
4.27	Triplet, J = 6.8 Hz	0.19	2-phenylethyl acetate CH_2	0.087
4.53	Singlet	-	Furfuryl alcohol CH_2	-
4.82	Broad singlet	-	Water OH suppressed	-
4.87	Quartet, J = 5.2 Hz	-	Acetaldehyde ethyl hemiacetal	-
5.20	Quartet, J = 5.2 Hz	-	Acetaldehyde water hemiacetal	-
6.33	Doublet, J = 3.2 Hz	0.013	2-methylfuran CH	0.011
6.35	Doublet, J = 4.0 Hz	0.0086	2-methylfuran CH	0.0077
6.73	Multiplet	0.018	o-cresol	0.016
6.76	DD, J = 3.6 Hz, 1.6 Hz	0.12	Furfural CH next to 2xCH	0.11
6.83	Doublet, J = 8.5 Hz	0.070	Phenol 2xCH next to CH	0.032
6.92	Multiplet	0.0090	p-cresol 2xCH next to CH	0.0041
6.97	Doublet, J = 8.2 Hz	0.015	o-cresol CH next to CH	0.013
7.04	Doublet, J = 8.5 Hz	0.011	o-cresol CH next to CH	0.010
7.19	Doublet, J = 7.3 Hz	0.12	Phenol 2xCH next to CH	0.054
7.23	Doublet, J = 7.3 Hz	0.22	2-phenylethanol 2x aromatic CH	0.098
7.28	Triplet, J = 7.5 Hz	0.21	2-phenylethanol 2x aromatic CH	0.093
7.55	Doublet, J = 3.8 Hz	0.13	Furfural CH	0.11
7.94	Singlet	0.12	Furfural CH	0.11
8.12	Singlet	0.036	Ethyl formate CH	0.032
9.56	Singlet	0.12	Furfural CHO	0.11
9.59	Doublet, J = 1.9 Hz	0.009	5-HMF CHO	0.0081
9.71	Quartet, J = 2.9 Hz	0.19	Acetaldehyde CHO	0.17
9.99	Singlet	0.0024	Vanillin CHO	0.0021

Table S12. Signal assignment for NMR spectrum of new make spirit distillate fraction 1 produced using peated malt, recorded at University of Edinburgh using a 600 MHz spectrometer, PNMS1.

δ_H / ppm	Multiplicity	Integration	Inferences	Concentration/ mmol L^{-1}
0	Singlet	10	DSS 3xCH_3	1.00
0.59	Multiplet	2.19	DSS CH_2	0.98
0.79	Multiplet	0.47	Fusel alcohols CH_3 groups carbon satellites	-
0.89	Multiplet	71.33	Fusel alcohols CH_3 groups	-
0.99	Multiplet	0.10	Fusel alcohols CH_3 groups carbon satellites	-
1.12	Triplet, J = 7.5 Hz	-	Fusel alcohol ethyl ester CH_3	-
1.19	Distorted	-	Ethanol CH_3 suppressed	-
1.25	Triplet, J = 7.2 Hz	-	Ethyl acetate CH_3 next to CH_2	-
1.29	Triplet, J = 7.0 Hz	0.99	Fusel alcohol ethyl ester CH_3	-
1.31	Doublet, J = 5.3 Hz	0.082	Lactic acid CH_3	0.025
1.43	Quartet, J = 7.0 Hz	10.2	3-methyl-1-butanol CH_2 next to CH and CH_2	4.59
1.54	Sextet, J = 7.2 Hz	10.6	n-propanol CH_2 next to CH_2 and CH_3	4.79
1.66	Nonet, J = 6.7 Hz	4.00	3-methyl-1-butanol CH next to 2xCH_3 and CH_2	3.60
1.73	Nonet, J = 6.7 Hz	4.55	Isobutanol CH next to 2xCH_3 and CH_2	4.09
1.77	Multiplet	-	DSS CH_2	-
2.01	Quintet, J = 2.1 Hz	0.56	DSS	0.25
2.03	Singlet	0.076	Acetate ester	-
2.03	Singlet	0.43	Acetic acid CH_3	0.13
2.06	Singlet	0.14	Ethyl acetate CH_3 isolated	0.043
2.18	Multiplet	-	o-cresol CH_3	-
2.20	Singlet	0.072	Acetone 2xCH_3	0.011
2.22	Multiplet	-	Acetaldehyde CH_3	-
2.26	Singlet	0.084	p-cresol CH_3	0.025
2.28	Triplet, J = 7.3 Hz	0.28	Ethyl ester	-
2.83	Multiplet	3.58	2-phenylethanol CH_2 triplet, DSS CH_2 overlap	-
2.93	Triplet	-	2-phenylethyl acetate CH_2	-
3.22	Doublet, J = 6.5 Hz	0.057	Isobutanol carbon satellite	-
3.23	Singlet	0.021	Methanol CH_3 carbon satellite	-
3.34	Doublet, J = 7.1 Hz	7.14	Isobutanol CH_2 next to CH and OH	3.21
3.35	Singlet	2.87	Methanol CH_3	0.86
3.45	DD, J = 10.7 Hz, 5.9 Hz	2.06	Part of 2-methylbutanol diastereotopic CH_2	1.86
3.53	Triplet, J = 6.8 Hz	9.75	n-propanol CH_2	4.39
3.60	Triplet, J = 7.2 Hz	-	3-methyl-1-butanol CH_2 next to CH_2	-
3.64	Distorted	-	Ethanol CH_2 suppressed	-

3.77	Quartet, J = 7.0 Hz	2.46	Phenylethanol CH_2	1.10
3.82	Singlet	0.12	Eugenol OCH_3	0.035
3.84	Singlet	0.21	Guaiacol OCH_3	0.064
4.12	Quartet, J = 7.2 Hz	0.17	Lactic acid CH next to CH_3	0.15
4.28	Triplet, J = 6.7 Hz	0.060	2-phenylethyl acetate CH_2	0.027
4.54	Singlet	0.12	Furfuryl alcohol CH_2	0.052
4.84	Broad singlet	-	Water OH suppressed	-
5.21	Quartet, J = 5.1 Hz	-	Acetaldehyde water hemiacetal	-
6.35	Doublet, J = 3.1 Hz	0.040	2-methylfuran CH	0.036
6.40	DD, J = 3.1 Hz, 1.9 Hz	0.041	2-methylfuran CH	0.037
6.75	Doublet, J = 3.5 Hz	0.038	p-cresol 2xCH next to CH	0.017
6.77	DD, J = 3.7 Hz, 1.6 Hz	0.33	Furfural CH next to 2xCH	0.30
6.79	Multiplet	0.025	o-cresol aromatic CH	0.023
6.85	Doublet, J = 9.0 Hz	0.40	Phenol 2xCH next to CH	0.36
7.02	Triplet, J = 9.0 Hz	0.20	o-cresol aromatic CH	0.18
7.08	Doublet, J = 7.5 Hz	0.069	o-cresol aromatic CH	0.062
7.22	Triplet, J = 7.3 Hz	0.94	Phenol 2xCH next to 2xCH	0.42
7.25	Doublet, J = 7.2 Hz	1.48	2-phenylethanol 2x aromatic CH	0.66
7.31	Triplet, J = 7.5 Hz	1.42	2-phenylethanol 2x aromatic CH	0.64
7.57	Doublet, J = 3.5 Hz	0.25	Furfural CH	0.22
7.94	Singlet	0.24	Furfural CH	0.22
9.54	Singlet	0.23	Furfural CHO	0.21
9.69	Quartet, J = 2.9 Hz	0.013	Acetaldehyde CHO	0.012

Table S13. Signal assignment for NMR spectrum of new make spirit distillate fraction 2 produced using peated malt, recorded at University of Edinburgh using a 600 MHz spectrometer, PNMS2.

δ_H / ppm	Multiplicity	Integration	Inferences	Concentration/ mmol L^{-1}
0	Singlet	10	DSS 3xCH_3	1.00
0.62	Multiplet	2.22	DSS CH_2	1.00
0.87	Multiplet	-	Fusel alcohols CH_3 groups carbon satellites	-
1.17	Distorted	-	Ethanol CH_3 suppressed	-
1.24	Triplet, J = 7.3 Hz	-	Ethyl acetate CH_3 next to CH_2	-
1.31	Multiplet	-	Lactic acid CH_3 next to CH	-
1.43	Quartet, J = 6.9 Hz	0.11	3-methyl-1-butanol CH_2 next to CH and CH_2	0.049
1.53	Sextet, J = 7.1 Hz	0.48	n-propanol CH_2 next to CH_2 and CH_3	0.21
1.75	Multiplet	2.30	DSS CH_2	1.04
2.04	Quintet, J = 2.0 Hz	0.50	DSS	0.22
2.07	Singlet	0.74	Acetic acid CH_3	0.22
2.10	Singlet	0.089	Ethyl acetate CH_3 isolated	0.027
2.19	Singlet	0.029	o-cresol CH_3	0.0088
2.20	Singlet	0.13	Acetone 2xCH_3	0.020

2.26	Singlet	0.086	p-cresol CH_3	0.026
2.86	Triplet, J = 6.7 Hz	2.60	2-phenylethanol CH_2	1.17
2.89	Multiplet	2.29	DSS CH_2	1.03
2.96	Triplet, J = 6.6 Hz	0.044	2-phenylethyl acetate CH_2	0.020
3.35	Singlet	4.37	Methanol CH_3	1.31
3.55	Triplet, J = 6.6 Hz	0.29	n-propanol CH_2 next to CH_2 and OH	0.13
3.58	Triplet, J = 6.6 Hz	-	3-methyl-1-butanol CH_2 next to CH_2	-
3.83	Triplet, J = 6.7 Hz	2.48	2-phenylethanol CH_2 next to CH_2 and OH	1.12
4.13	Quartet, J = 7.3 Hz	-	Lactic acid CH next to CH_3	-
4.30	Triplet, J = 7.4 Hz	0.0012	2-phenylethyl acetate CH_2	0.00054
4.56	Singlet	0.18	Furfuryl alcohol CH_2	0.082
4.79	Broad Singlet	-	Water OH suppressed	-
5.23	Quartet, J = 5.1 Hz	0.018	Acetaldehyde water hemiacetal	-
6.40	Doublet, J = 3.1 Hz	0.056	2-methylfuran CH	0.050
6.44	DD, J = 3.0 Hz, 1.8 Hz	0.056	2-methylfuran CH	0.051
6.75	DD, J = 3.6 Hz, 1.6 Hz	0.093	Furfural CH	0.084
6.78	Doublet, J = 8.0 Hz	0.008	p-cresol 2xCH next to CH	0.0034
6.80	Doublet, J = 8.4 Hz	0.17	o-cresol CH	0.15
6.90	Doublet, J = 7.7 Hz	0.50	Phenol 2xCH next to CH	0.22
6.98	Triplet, J = 7.4 Hz	0.23	o-cresol CH next to 2xCH	0.21
7.06	Doublet, J = 7.9 Hz	0.052	p-cresol 2xCH next to CH	0.023
7.13	Doublet, J = 8.0 Hz	0.18	o-cresol CH next to CH	0.16
7.29	Triplet, J = 7.3 Hz	0.78	Phenol 2xCH next to 2xCH	0.35
7.31	Doublet, J = 8.0 Hz	3.12	2-phenylethanol 2x aromatic CH	1.40
7.37	Triplet, J = 7.5 Hz	2.29	2-phenylethanol 2x aromatic CH	1.03
7.57	Doublet, J = 3.7 Hz	0.070	Furfural CH	0.063
7.91	Singlet	0.070	Furfural CH	0.063
9.49	Singlet	0.068	Furfural CHO	0.061
9.66	Quartet, J = 2.9 Hz	0.0076	Acetaldehyde CHO	0.0068

Table S14. Signal assignment for NMR spectrum of new make spirit distillate fraction 3 produced using peated malt, recorded at University of Edinburgh using a 600 MHz spectrometer, PNMS3.

δ_H / ppm	Multiplicity	Integration	Inferences	Concentration/ mmol L^{-1}
0	Singlet	10	DSS 3xCH_3	1.00
0.61	Multiplet	2.19	DSS CH_2	0.99
0.77	Multplet	0.27	Fusel alcohols CH_3 groups carbon satellites	-
0.88	Multiplet	53.4	Fusel alcohols CH_3 groups	-
0.98	Multiplet	0.46	Fusel alcohols CH_3 groups carbon satellites	-
1.10	Triplet, J = 6.8 Hz	-	Fusel alcohol ethyl ester CH_3	-

1.17	Triplet, J = 7.7 Hz	-	Ethanol CH_3 suppressed	-
1.24	Triplet, J = 7.2 Hz	2.80	Ethyl acetate CH_3 next to CH_2	0.84
1.27	Triplet, J = 7.1 Hz	2.37	Fusel alcohol ethyl ester CH_3	-
1.31	Triplet, J = 5.4 Hz	3.70	Lactic acid CH_3 next to CH	1.11
1.36	Doublet, J = 5.3 Hz	0.32	Acetate ester	-
1.42	Quartet, J = 7.0 Hz	6.41	3-methyl-1-butanol CH_2 next to CH and CH_2	2.89
1.53	Sextet, J = 7.1 Hz	6.75	n-propanol CH_2 next to CH_2 and CH_3	3.04
1.65	Nonet, J = 6.7 Hz	3.34	3-methyl-1-butanol CH next to 2xCH_3 and CH_2	3.01
1.72	Nonet, J = 5.2 Hz	5.21	Isobutanol CH next to 2xCH_3 and CH_2	4.69
1.77	Multiplet	-	DSS	-
2.02	Singlet	0.068	Acetate ester	-
2.04	Quintet, J = 2.1 Hz	0.56	DSS	0.25
2.06	Singlet	2.26	Acetic acid CH_3	0.68
2.10	Singlet	0.15	Ethyl acetate CH_3 isolated	0.044
2.19	Singlet	0.29	o-cresol CH_3	0.087
2.22	Singlet	0.51	Acetone 2xCH_3	0.077
2.23	Doublet, J = 2.8 Hz	1.56	Acetaldehyde CH_3 next to CH	0.47
2.25	Singlet	0.14	p-cresol CH_3	0.041
2.33	Multiplet	-	Ethyl ester	-
2.84	Triplet, J = 6.9 Hz	2.76	2-phenylethanol CH_2 triplet, DSS CH_2 overlap	-
2.96	Triplet, J = 6.4 Hz	0.048	2-phenylethyl acetate CH_2	0.021
3.22	Singlet	0.056	Methanol CH_3 carbon satellite	-
3.23	Doublet, J = 6.5 Hz	0.030	Isobutanol carbon satellite	-
3.34	Singlet	11.57	Methanol CH_3	3.47
3.35	Doublet, J = 6.8 Hz	6.66	Isobutanol CH_2 next to CH and OH	3.00
3.36	Singlet	0.40	Part of 2-methylbutanol diastereotopic CH_2	-
3.38	Doublet, J = 6.6 Hz	1.02	Part of 2-methylbutanol diastereotopic CH_2	-
3.46	DD, J = 10.7 Hz, 6.0 Hz	1.66	Part of 2-methylbutanol diastereotopic CH_2	1.50
3.54	Triplet, J = 6.7 Hz	4.29	n-propanol CH_2	1.93
3.61	Triplet, J = 6.9 Hz	-	3-methyl-1-butanol CH_2 next to CH_2	-
3.64	Distorted	-	Ethanol CH_2 suppressed	-
3.81	Triplet, J = 6.9 Hz	1.81	Phenylethanol CH_2	0.81
3.84	Singlet	0.10	Guaiacol OCH_3	0.031
4.12	Quartet, J = 7.1 Hz	1.32	Lactic acid CH next to CH_3	1.19
4.32	Triplet, J = 6.6 Hz	0.028	2-phenylethyl acetate CH_2	0.013
4.55	Singlet	0.16	Furfuryl alcohol CH_2	0.070

4.83	Broad singlet	-	Water OH suppressed	-
4.92	Quartet, J = 5.2 Hz	-	Acetaldehyde ethyl hemiacetal	-
5.22	Quartet, J = 5.2 Hz	0.41	Acetaldehyde water hemiacetal	-
6.38	Doublet, J = 3.2 Hz	0.068	2-methylfuran CH	0.062
6.42	DD, J = 2.9 Hz, 2.0 Hz	0.066	2-methylfuran CH	0.059
6.72	Mutiplet	0.056	p-cresol 2x CH	0.025
6.76	DD, J = 3.6 Hz, 1.6 Hz	0.084	Furfural CH next to 2xCH	0.075
6.78	Doublet, J = 8.3 Hz	0.15	o-cresol CH	0.14
6.88	Doublet, J = 7.9 Hz	0.48	Phenol 2xCH next to CH	0.21
6.95	Triplet, J = 7.3 Hz	0.20	o-cresol CH	0.18
7.03	Doublet, J = 7.4 Hz	0.058	p-cresol 2xCH next to CH	0.026
7.10	Doublet, J = 8.5 Hz	0.17	o-cresol CH	16.2
7.26	Doublet, J = 7.3 Hz	0.71	Phenol 2xCH next to 2xCH	0.32
7.29	Doublet, J = 7.3 Hz	2.39	2-phenylethanol 2x aromatic CH	1.07
7.36	Triplet, J = 7.5 Hz	1.81	2-phenylethanol 2x aromatic CH	0.82
7.57	Doublet, J = 3.7 Hz	0.067	Furfural CH	0.060
7.92	Singlet	0.064	Furfural CH	0.058
9.49	Singlet	0.058	Furfural CHO	0.052
9.66	Quartet, J = 2.9 Hz	0.44	Acetaldehyde CHO	0.40

Table S15. Signal assignment for NMR spectrum of low wines produced using spruce smoked malt, recorded at University of Edinburgh using a 600 MHz spectrometer, SLW.

δ_H / ppm	Multiplicity	Integration	Inferences	Concentration/ mmol L^{-1}
0	Singlet	10	DSS 3xCH_3	1.00
0.59	Multiplet	2.23	DSS CH_2	1.00
0.78	Multiplet	1.38	Fusel alcohols CH_3 groups carbon satellites	-
0.89	Multiplet	219.24	Fusel alcohols CH_3 groups	-
0.99	Multiplet	1.08	Fusel alcohols CH_3 groups carbon satellites	-
1.09	Triplet, J = 8.4 Hz	-	Ethanol CH_3 carbon satellite	-
1.11	Triplet, J = 7.3 Hz	4.25	Fusel alcohol ethyl ester CH_3	-
1.18	Distorted	-	Ethanol CH_3 suppressed	-
1.25	Triplet, J = 7.1 Hz	13.63	Ethyl acetate CH_3 next to CH_2	4.09
1.29	Doublet, J = 5.6 Hz	4.58	Fusel alcohol ethyl ester CH_3	-
1.31	Doublet, J = 5.2 Hz	14.29	Lactic acid CH_3 next to CH	4.29
1.36	Multiplet	-	Acetate ester	-
1.43	Quartet, J = 7.0 Hz	30.05	3-methyl-1-butanol CH_2 next to CH and CH_2	13.52
1.49	Multiplet	-	n-butanol CH_2 groups	-
1.54	Sextet, J = 7.2 Hz	21.22	n-propanol CH_2 next to CH_2 and CH_3	9.55
1.68	Nonet, J = 6.7 Hz	12.52	3-methyl-1-butanol CH next to 2xCH_3 and CH_2	11.27

1.73	Nonet, J = 6.7 Hz	13.43	Isobutanol CH next to 2xCH_3 and CH_2	12.08
1.78	Multiplet	1.80	DSS CH_2	0.81
1.94	Singlet	0.052	Acetic acid CH_3 carbon satellite	-
2.05	Singlet	8.95	Acetic acid CH_3	2.69
2.08	Singlet	0.060	Ethyl acetate CH_3 isolated	0.018
2.16	Singlet	0.075	Acetic acid CH_3 carbon satellite	-
2.18	Singlet	0.22	o-cresol CH_3	0.067
2.21	Singlet	0.76	Acetone 2xCH_3	0.11
2.23	Doublet, J = 2.9 Hz	3.65	Acetaldehyde CH_3	1.09
2.27	Multiplet	0.10	p-cresol CH_3	-
2.30	Triplet, J = 7.4 Hz	1.68	Ethyl ester	-
2.82	Multiplet	2.35	DSS CH_2	1.06
2.85	Singlet	0.80	Part of 2-phenylethanol CH_2	-
2.93	Triplet, J = 7.0 Hz	0.092	2-phenylethyl acetate	0.042
3.21	Doublet, J = 6.6 Hz	0.19	Isobutanol carbon satellite	-
3.23	Singlet	0.079	Methanol CH_3 carbon satellite	-
3.33	Doublet, J = 6.6 Hz	28.51	Isobutanol CH_2 next to CH and OH	12.83
3.35	Singlet	9.28	Methanol CH_3	2.78
3.44	DD, J = 10.6 Hz, 6.0 Hz	7.40	Part of 2-methylbutanol diastereotopic CH_2	6.66
3.48	Quartet, J = 7.1 Hz	-	Ethanol CH_2 carbon satellite	-
3.52	Triplet, J = 6.8 Hz	16.75	n-propanol CH_2	7.54
3.59	Triplet, J = 7.1 Hz	23.10	3-methyl-1-butanol CH_2 next to CH_2	10.39
3.63	Distorted	-	Ethanol CH_2 suppressed	-
3.75	Quartet, J = 7.0 Hz	-	Ethanol CH_2 carbon satellite	-
4.12	Quartet, J = 7.2 Hz	7.84	Lactic acid CH next to CH_3	2.35
4.27	Triplet, J = 6.8 Hz	0.22	2-phenylethyl acetate CH_2	0.097
4.54	Singlet	-	Furfuryl alcohol CH_2	-
4.82	Broad singlet	-	Water OH suppressed	-
5.21	Quartet, J = 5.2 Hz	1.07	Acetaldehyde water hemiacetal	-
6.33	Doublet, J = 3.0 Hz	0.017	2-methylfuran CH	0.015
6.36	Singlet	0.012	2-methylfuran CH	0.010
6.73	Multiplet	0.048	p-cresol 2xCH next to CH	0.043
6.76	DD, J = 3.3 Hz, 1.6 Hz	0.099	Furfural CH next to 2xCH	0.089
6.84	Doublet, J = 8.1 Hz	0.12	Phenol 2xCH next to CH	0.055
6.90	Doublet, J = 7.1 Hz	0.010	Guaiacol CH	0.0091
6.92	Doublet, J = 7.5 Hz	0.018	p-cresol 2xCH next to CH	0.0083
6.98	Triplet, J = 8.4 Hz	0.050	o-cresol CH next to 2xCH	0.044
7.04	Doublet, J = 7.4 Hz	0.039	o-cresol CH next to CH	0.035
7.19	Triplet, J = 7.2 Hz	0.21	Phenol 2xCH next to 2xCH	0.094
7.23	Doublet, J = 7.4 Hz	0.35	2-phenylethanol 2x aromatic CH	0.16
7.28	Triplet, J = 7.4 Hz	0.30	2-phenylethanol 2x aromatic CH	0.14

7.55	Doublet, J = 3.7 Hz	0.095	Furfural CH	0.086
7.94	Singlet	0.095	Furfural CH	0.086
8.12	Singlet	0.048	Ethyl formate CH	0.043
9.56	Singlet	0.096	Furfural CHO	0.086
9.59	Singlet	0.012	5-HMF CHO	0.010
9.71	Quartet, J = 2.9 Hz	1.08	Acetaldehyde CHO	0.98
9.99	Singlet	0.0036	Vanillin CHO	0.0032

Table S16. Signal assignment for NMR spectrum of new make spirit distillate fraction 1 produced using spruce smoked malt, recorded at University of Edinburgh using a 600 MHz spectrometer, SNMS1.

δ_H / ppm	Multiplicity	Integration	Inferences	Concentration/ mmol L^{-1}
0	Singlet	10	DSS 3xCH_3	1.00
0.59	Multiplet	2.22	DSS CH_2	1.00
0.79	Multiplet	0.49	Fusel alcohols CH_3 groups carbon satellites	-
0.89	Multiplet	67.24	Fusel alcohols CH_3 groups	-
0.99	Multiplet	0.43	Fusel alcohols CH_3 groups carbon satellites	-
1.11	Multiplet	-	Fusel alcohol ethyl ester CH_3	-
1.19	Distorted	-	Ethanol CH_3 suppressed	-
1.25	Triplet, J = 7.2 Hz	-	Ethyl acetate CH_3 next to CH_2	-
1.29	Multiplet	-	Fusel alcohol ethyl ester CH_3	-
1.31	Doublet, J = 5.1 Hz	0.79	Lactic acid CH_3 next to CH	0.24
1.35	Multiplet	-	Acetate ester	-
1.43	Quartet, J = 6.9 Hz	8.85	3-methyl-1-butanol CH_2 next to CH and CH_2	3.98
1.54	Sextet, J = 7.1 Hz	10.6	n-propanol CH_2 next to CH_2 and CH_3	4.78
1.66	Nonet, J = 6.7 Hz	4.08	3-methyl-1-butanol CH next to 2xCH_3 and CH_2	3.67
1.73	Nonet, J = 6.7 Hz	4.30	Isobutanol CH next to 2xCH_3 and CH_2	3.87
1.76	Multiplet	-	DSS	-
2.01	Quintet, J = 2.1 Hz	0.61	DSS	0.27
2.03	Singlet	0.16	Acetate ester	-
2.04	Singlet	0.18	Acetic acid CH_3	0.056
2.06	Singlet	0.32	Ethyl acetate CH_3 isolated	0.096
2.18	Singlet	0.60	o-cresol CH_3	0.18
2.21	Singlet	0.25	Acetone 2xCH_3	0.037
2.26	Singlet	0.25	p-cresol CH_3	0.075
2.29	Triplet, J = 7.3 Hz	0.43	Ethyl ester	-
2.83	Multipet	4.44	2-phenylethanol CH_2, DSS CH_2 overlap	-
2.94	Triplet, J = 6.8 Hz	0.13	2-phenylethyl acetate CH_2	0.058
3.22	Doublet, J = 6.7 Hz	0.056	Isobutanol carbon satellite	-

3.23	Singlet	0.043	Methanol CH_3	-
3.34	Doublet, J = 6.7 Hz	7.31	Isobutanol CH_2 next to CH and OH	3.29
3.35	Singlet	6.96	Methanol CH_3	2.09
3.45	DD, J = 10.7 Hz, 5.9 Hz	1.84	Part of 2-methylbutanol diastereotopic CH_2	1.66
3.53	Triplet, J = 6.8 Hz	9.06	n-propanol CH_2	4.08
3.60	Triplet, J = 7.0 Hz	-	3-methyl-1-butanol CH_2 next to CH_2	-
3.64	Distorted	-	Ethanol CH_2 suppressed	-
3.78	Triplet, J = 7.2 Hz	2.25	Phenylethanol CH_2	1.01
3.82	Singlet	-	Eugenol OCH_3	-
3.84	Singlet	0.28	Guaiacol OCH_3	-
4.12	Quartet, J = 7.2 Hz	0.35	Lactic acid CH next to CH_3	0.31
4.29	Triplet, J = 6.7 Hz	0.014	2-phenylethyl acetate CH_2	0.055
4.54	Singlet	0.17	Furfuryl alcohol CH_2	0.075
4.84	Broad Singlet	-	Water OH suppressed	-
5.21	Quartet, J = 5.3 Hz	0.076	Acetaldehyde water hemiacetal	-
6.36	Doublet, J = 3.2 Hz	0.068	2-methylfuran CH	0.061
6.40	Multiplet	0.063	2-methylfuran CH	0.057
6.77	DD, J = 3.6 Hz, 1.5 Hz	0.23	Furfural CH	0.21
6.80	Multiplet	0.27	o-cresol	0.24
6.85	Doublet, J = 7.6 Hz	0.69	Phenol 2xCH next to CH	0.29
7.04	Triplet, J = 8.5 Hz	0.35	o-cresol CH next to 2xCH	0.32
7.10	Doublet, J = 6.8 Hz	0.24	o-cresol CH next to CH	0.22
7.23	Triplet, J = 7.3 Hz	1.46	Phenol 2xCH next to 2xCH	0.66
7.26	Doublet, J = 7.1 Hz	2.33	2-phenylethanol 2x aromatic CH	1.05
7.32	Triplet, J = 7.5 Hz	2.18	2-phenylethanol 2x aromatic CH	0.98
7.58	Doublet, J = 3.8 Hz	0.19	Furfural CH	0.17
7.94	Singlet	0.19	Furfural CH	0.17
9.53	Singlet	0.18	Furfural CHO	0.16
9.69	Quartet, J = 2.7 Hz	0.023	Acetaldehyde CHO	0.069

Table S17. Signal assignment for NMR spectrum of new make spirit distillate fraction 2 produced using spruce smoked malt, recorded at University of Edinburgh using a 600 MHz spectrometer, SNMS2.

δ_H / ppm	Multiplicity	Integration	Inferences	Concentration/ mmol L^{-1}
0	Singlet	10	DSS 3xCH_3	1.00
0.62	Multiplet	2.22	DSS CH_2	1.00
0.87	Multiplet	-	Fusel alcohols CH_3 groups	-
1.17	Distorted	-	Ethanol CH_3 suppressed	-
1.24	Triplet, J = 7.4 Hz	-	Ethyl acetate CH_3 next to CH_2	-
1.31	Multuplet	-	Lactic acid CH_3 next to CH	-
1.43	Quartet, J = 6.9 Hz	0.10	3-methyl-1-butanol CH_2 next to CH and CH_2	0.048
1.53	Quartet, J = 7.1 Hz	0.37	n-propanol CH_2 next to CH_2 and	0.17

			CH_3	
1.76	Multiplet	2.30	DSS	1.04
2.04	Quintet, J = 2.1 Hz	0.55	DSS	0.25
2.06	Singlet	0.19	Acetic acid CH_3	0.056
2.19	Singlet	0.045	o-cresol CH_3	0.013
2.22	Singlet	-	Acetone CH_3	-
2.26	Singlet	0.16	p-cresol CH_3	0.049
2.86	Triplet, J = 6.7 Hz	3.92	2-phenylethanol CH_2	1.76
2.90	Multiplet	2.26	DSS CH_2	1.02
2.96	Singlet	0.017	Phenylethyl acetate CH_2	0.0077
3.35	Singlet	10.3	Methanol CH_3	3.09
3.55	Triplet, J = 6.6 Hz	-	n-propanol CH_2	-
3.58	Triplet, J = 6.6 Hz	-	3-methyl-1-butanol CH_2 next to CH_2	-
3.83	Triplet, J = 6.7 Hz	3.93	2-phenylethanol CH_2	1.77
4.13	Quartet, J = 7.2 Hz	0.022	Lactic acid CH next to CH_3	0.020
4.30	Triplet, J = 7.3 Hz	0.0006	2-phenylethyl acetate CH_2	0.00027
4.57	Singlet	0.25	Furfuryl alcohol CH_2	0.11
4.78	Singlet	-	Water OH suppressed	-
5.24	Quartet, J = 5.2 Hz	0.034	Acetaldehyde water hemiacetal	-
6.40	Doublet, J = 3.1 Hz	0.10	2-methylfuran CH	0.094
6.44	DD, J = 3.2 Hz, 1.9 Hz	0.10	2-methylfuran CH	0.094
6.76	Singlet	-	Furfural CH	-
6.78	Doublet, J = 8.1 Hz	0.057	p-cresol 2xCH next to CH	0.026
6.81	Triplet, J = 7.9 Hz	0.42	o-cresol CH next to 2xCH	0.38
6.91	Doublet, J = 7.9 Hz	0.90	Phenol 2xCH next to CH	0.80
6.98	Triplet, J = 7.3 Hz	0.38	o-cresol CH next to 2xCH	0.35
7.06	Doublet, J = 7.8 Hz	0.080	p-cresol 2xCH next to CH	0.036
7.29	Triplet, J = 7.3 Hz	1.68	Phenol 2xCH next to 2xCH	0.76
7.31	Doublet, J = 7.9 Hz	4.34	2-phenylethanol 2x aromatic CH	1.95
7.38	Triplet, J = 7.6 Hz	3.51	2-phenylethanol 2x aromatic CH	1.58
7.57	Doublet, J = 3.6 Hz	0.052	Furfural CH	0.046
7.91	Singlet	0.051	Furfural CH	0.043
9.49	Singlet	0.048	Furfural CHO	0.043
9.66	Quartet, J = 1.8 Hz	0.061	Acetaldehyde CHO	0.055

Table S18. Signal assignment for NMR spectrum of new make spirit distillate fraction 3 produced using spruce smoked malt, recorded at University of Edinburgh using a 600 MHz spectrometer, SNMS3.

ABV measurements

ABV measurements were performed through comparison of integration values of the OH and CH_3 signals, accounting for the overlap of ethanol and water OH signals. Each measurements was performed in triplicate.

SLW1	OH signal integration	CH_3 signal integration	mol%	w%	Density (g/mL)	ABV
Sample 1	6.95	1	9.15	20.47	0.9679	25.67
Sample 2	6.97	1	9.12	20.42	0.9680	25.61
Sample 3	6.97	1	9.12	20.43	0.9680	25.61
Average						25.63 ± 0.03
SLW2						
Sample 1	6.84	1	9.29	20.74	0.9676	26.00
Sample 2	6.88	1	9.24	20.65	0.9677	25.88
Sample 3	6.86	1	9.27	20.71	0.9676	25.95
Average						25.94 ± 0.06
SNMS1_1						
Sample 1	0.85	1	43.84	66.62	0.8756	75.55
Sample 2	0.85	1	43.83	66.61	0.8756	75.55
Sample 3	0.86	1	43.81	66.59	08757	75.52
						75.54 ± 0.01
SNMS1_2						
Sample 1	0.84	1	44.21	66.95	0.8748	75.86
Sample 2	0.84	1	44.19	66.94	0.8749	75.85
Sample 3	0.84	1	44.19	66.93	0.8749	75.84
						75.85 ± 0.01
SNMS 2_1						
Sample 1	2.66	1	22.28	42.29	0.9304	50.96
Sample 2	2.66	1	22.27	42.28	0.9304	50.95
Sample 3	2.66	1	22.28	42.29	0.9304	50.96
						50.95 ± 0.01
SNMS 2_2						
Sample 1	2.56	1	23.06	43.38	0.9281	52.14
Sample 2	2.55	1	23.09	43.43	0.9280	52.20
Sample 3	2.55	1	23.11	43.46	0.9279	52.22
						52.19 ± 0.04
SNMS 3_1						
Sample 1	43.43	1	1.52	3.80	0.9915	4.88
Sample 2	43.48	1	1.52	3.80	0.9915	4.88
Sample 3	43.51	1	1.52	3.80	0.9915	4.88
						4.88 ± 0.01
SNMS 3_2						
Sample 1	37.74	1	1.75	4.36	0.9905	5.59
Sample 2	37.63	1	1.76	4.37	0.9905	5.60
Sample 3	37.74	1	1.75	4.36	0.9905	5.59
						5.60 ± 0.01

PLW1						
Sample 1	23.63	1	2.78	6.82	0.9866	8.71
Sample 2	23.64	1	2.78	6.81	0.9866	8.71
Sample 3	23.66	1	2.78	6.81	0.9866	8.70
						8.70 ± 0.01
PLW2						
Sample 1	124.75	1	0.53	1.35	0.9958	1.74
Sample 2	124.03	1	0.54	1.36	0.9958	1.75
Sample 3	124.23	1	0.54	1.36	0.9958	1.75
						1.75 ± 0.01
PNMS1_1						
Sample 1	0.82	1	44.79	67.47	0.8736	76.34
Sample 2	0.82	1	44.98	67.64	0.8732	76.50
Sample 3	0.81	1	45.05	67.70	0.8731	76.55
						76.46 ± 0.11
PNMS1_2						
Sample 1	0.82	1	44.77	67.45	0.8736	76.32
Sample 2	0.82	1	44.96	67.62	0.8732	76.48
Sample 3	0.82	1	44.83	67.51	0.8735	76.37
						76.39 ± 0.08
PNMS2_1						
Sample 1	2.14	1	26.96	48.55	0.9170	57.66
Sample 2	2.13	1	27.04	48.65	0.9168	57.76
Sample 3	2.13	1	27.05	48.66	0.9167	57.78
						57.73 ± 0.06
PNMS2_2						
Sample 1	2.23	1	26.01	47.33	0.9196	56.37
Sample 2	2.23	1	26.01	47.33	0.9196	56.37
Sample 3	2.23	1	26.03	47.36	0.9196	56.40
						56.38 ± 0.02
PNMS3_1						
Sample 1	28.42	1	2.32	5.72	0.9883	7.32
Sample 2	28.43	1	2.32	5.72	0.9883	7.32
Sample 3	28.44	1	2.32	5.72	0.9883	7.32
						7.32 ± 0.01
PNMS3_2						
Sample 1	30.14	1	2.19	5.41	0.9887	6.93
Sample 2	30.16	1	2.18	5.40	0.9887	6.92
Sample 3	30.14	1	2.19	5.41	0.9887	6.92
						6.92 ± 0.01

Table S19. ABV measurements performed on low wines and new make spirit samples using ^{1}H NMR.

References

1. B. Harrison, O. Fagnen, F. Jack and J. Brosnan, *J. Inst. Brew.*, 2011, **117**, 106–112.