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# Characterisation of traditionally kiln produced pine tar by gas chromatography-mass spectrometry

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## Abstract

Pine tar samples from each out of six, successively filled barrels from a traditionally accomplished, yet temperature monitored, kiln production have been characterised by combined gas chromatography mass spectrometry (GC-MS). The relative abundance of resin- and fatty acids correlated in a distinctive way to the temperature development within the kiln, which made it possible to identify from which barrel each sample originated. The pine tar samples were according to an at least 300 years old tarring practice of Medieval Stave churches, subsequently seethed in a cast iron pan and again characterised by GC-MS. The characteristics of the initial samples were still evident after heating to 180–190°C. © 2002 Elsevier Science B.V. All rights reserved.

*Keywords:* Pine tar; Pine pitch; Diterpenoids; Abietic acid; Dehydro abietic acid; Kiln produced tar; Wood pyrolysis; Dry distillation; Destructive distillation; GC-MS

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## 1. Introduction

Tarring with pine tar is among the oldest surface treatments known of in Norway and elsewhere in the Nordic countries. In the Norwegian Viking age, approximately 800–1030 A.D., pine tar was used for sealing and probably coating of wooden

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ships [1]. As Christianity was established in the beginning of the second millennium, the wooden churches were to be coated with pine tar every third year, according to law regulations. Since the 1980s, the Cultural Heritage Authorities in Norway prescribe pine tar made in kilns as the proper surface treatment for the remaining medieval stave churches. This claim is realistic, because, although weak, a living tradition on tar making in kilns still exists. Yet, tar treatments today are not sufficiently successful. Tarring with newly produced tar, normally heated in water bath prior to application, seems to be easily washed and wore off due to rain, wind and sun exposure. It often takes less than one year to wear down a tarred surface, particularly those exposed to sun. One conservation aim is to develop a neat pine tar treatment able to protect a sun exposed wooden surface for at least 3 years. Both archaeological, architectural and written sources give evidence that pine tar made in kilns was the original surface treatment for the stave churches and it appears also that kiln constructions today are similar to Middle Age kilns [2,3]. Taking hand-written church records from the 17th to 18th centuries into consideration, a pre-application treatment involving heating or seething of tar is described. The records are merely specifying that the tar was reduced to either 'stir tar' or 'pitch'. The term 'stir-tar' is not known from other contexts. In this work, 'stir-tar' is understood as tar, which is still liquid at a temperate condition, contrary to pitch, which is regarded almost solid at room temperature and below ( $< 25^{\circ}\text{C}$ ).

In a traditional kiln production involving a so-called dry or destructive distillation of pine wood, the tar is drained continuously, or in portions of 10–20 l, from the bottom of the kiln into wooden barrels or other kinds of containers. This is different from an industrial retort plant, which normally accumulate the tar in one big tank. In a kiln production, the tar in the first barrel is distilled at lower temperatures than the tar in e.g. the fourth or sixth barrel, resulting in diverging chemical composition and physical properties [4,5]. The tar fractions from a kiln production thus represent a diversity of tar qualities contrary to tar from a retort plant production. One assumption is that our predecessors knew the advantages and disadvantages of every tar-quality. This knowledge is lost today, but should be possible to regain to some extent by analysing and comparing both fresh, as well as artificially and naturally aged pine tar samples according to chemical contents and optical appearance. Liquid tar samples are also characterised by physical properties like viscosity, specific weight, percentage of water soluble components, percentage of volatile components, film forming abilities, penetrating and drying capacity etc.

The aim of this work was limited to characterise a series of six tar samples (from six barrels) collected during a kiln production in 1996 called FNN, by combined gas chromatography-mass spectrometry (GC-MS), which can be used to identify and quantify the bulk part of the organic constituents of the tar [6,7]. By using silylation instead of methylation, we are able to differentiate between naturally methylated resin- and fatty acids and acids. Two of the samples were subsequently seethed by different temperatures and time periods and characterised by GC-MS. This was done in order to examine the influence of temperature and heating time upon composition. Furthermore, these experiments should provide information about

whether the characteristics of the fractions resulting from the traditional kiln collecting practice were recognisable even after heat treatment.

## 2. Materials and methods

### 2.1. Samples

The pine tar samples derive from a traditionally accomplished kiln production in Øverbygd (Troms, Norway). The raw material was big, old stumps from Scots pine (*Pinus Sylvestris*), from trees felled about 50 years earlier. At this stage, the remains primarily consist of resinous heartwood, which is carefully rinsed during autumn season, dried and chopped by axe in frosty weather in the wintertime into pieces approximately 30–40 cm long and 4–5 cm thick. The wood sticks were stacked in the following summer, due to tradition. The wood was stacked «hemispherically» on a funnel shaped platform on top of a birch bark layer. Eight thermo-elements were installed in the stack during construction to measure temperatures every 10 min during the burning process. The wood stack was finally covered with heather turf except along the base, where the kiln was ignited. It was allowed to catch fire properly before being covered by turf all over. In the centre of the funnel was a drain hole, which lead to a hollowed-out log underneath from which the tar was tapped off in portions of 10–20 l directly into barrels. The kiln consisted of approximately 35 m<sup>3</sup> of wood, burned for 45 h and gave about 1050 l of tar and 230 l of 'tar acid'. Tar acid is an acidic water phase, heavier than tar, which gradually separates from the tar during storage and which is usually removed after a couple of weeks. The kiln filled up six big plastic barrels of 220 l each. Six samples, each of 1.5 l, were taken just after every barrel shift (within the first 30 min) and represent as such each 220 l barrel in the production (Table 1). The samples of 1.5 l were brought to the laboratory and stored at room temperature for almost 5 months before the tar acid was removed. Additionally, 25 l samples from each barrel were brought to the laboratory in plastic cans and tar from barrel number 2 and 5, FNN2 and FNN5, were subjected to heating experiments including seething in a cast-iron pan. One portion of tar was heated at 120–30°C and subsamples (approximately 1.5–2 l) were collected for analysis and application experiments at 30 and 60 min. Another portion was heated at 180–90°C, and subsamples collected at 15, 30 and 60 min, respectively, Table 2.

### 2.2. Instrumentation

The GC-MS instrumentation consisted of a Hewlett Packard 5890 Series II gas chromatograph and a Hewlett Packard 5989 A mass spectrometer. The separations were performed on a Restek 5 MS (5% diphenyl-95% dimethyl polysiloxane) low bleed/MS capillary column (30 m × 0.25 mm i.d., 0.25 µm film thickness) from Restek (Bellefonte, USA) with a 2.5 m × 0.32 mm i.d. methyl deactivated retention gap, from Chrompack (Middelburg, The Netherlands). Helium (99.9999%) from

Table 1  
Average production temperatures for 1.5 l samples and the flow rate according to the filling of barrels

Sample number	Time when collected	Average temperature while collected (°C)	% of tar acid in 1.5 l samples (v/v)	Flow rate for each barrel (l h <sup>-1</sup> )	% of tar acid in the barrels (v/v)
FNN-1	03:28 1 July	100	30	44	25
FNN-2	08:04 1 July	183	19	73	14
FNN-3	11:14 1 July	192	18	53	14
FNN-4	15:04 1 July	264	17	42	14
FNN-5	20:04 1 July	317	34	30	17
FNN-6	03:44 2 July	380	35	14	24

AGA (Oslo, Norway) was used as carrier gas at 1.0 ml min<sup>-1</sup>. The oven temperature was programmed from 40 to 190°C at 10°C min<sup>-1</sup> and from 190 to 300°C at 4°C min<sup>-1</sup> and finally held at 300°C for 10 min. Splitless injections of 1 µl were performed with an autosampler (Hewlett Packard 7673 injector) at 250°C. The MS was operated in electron ionisation (EI) mode and the m/z range from 40 to 500 was scanned with a cycle time of 0.5 s. The transfer line was held at 300°C.

### 2.3. Reagents and solutions

HPLC grade methyl tert butyl ether (MTBE) was obtained from Rathburn (Walkerburn, UK). Myristic acid (99–100%) and myristic 14,14,14-D<sub>3</sub> acid were obtained from Sigma Chemical Co. (St. Louis, MO, USA). TriSil<sup>®</sup> was from Pierce Chemical Co. (Rockford, IL, USA).

Stock solutions of the internal standard, tri-deuterated myristic acid, were prepared by dissolving 25.5 mg in 50 or 25 ml MTBE dried over molecular sieve (Fluka, Buchs, Switzerland). The stock solutions were kept at 4°C.

### 2.4. Procedures

A weighed sample (about 100 mg) of tar was dissolved in 25 ml dried MTBE. To a 300 µl aliquot of this sample was added 200 µl of a 0.153 mg ml<sup>-1</sup> internal standard solution and 100 µl of Trisil [8]. The solution was shaken for 5 min and allowed to stand for at least 2 h at ambient temperature before GC-MS analysis. Two derivatization replicates were performed. Quantification was done assuming equal response factors. The chromatogram was integrated from 5 to 45 min and the relative concentration of the individual components in the tar sample was found by the following equation, where *A* is area of the compound of interest and *A*<sub>I.S.</sub> is area of the internal standard.

$$\% \text{ of compound in tar} = \frac{A \cdot \text{weight of I.S.} \cdot 100\%}{(A_{\text{I.S.}} \cdot \text{weight of sample})}$$

Identification of the compounds was achieved using computer matching of the mass spectra with the NIST library.

Table 2  
Heating treatments for FNN2 and FNN5

Samples	Treatment	
FNN2	FNN5	Untreated
FNN2-1	FNN5-1	120–30°C for 30 min
FNN2-2	FNN5-2	120–30°C for 60 min
FNN2-3	FNN5-3	180–90°C for 15 min
FNN2-4	FNN5-4	180–90°C for 30 min
FNN2-5	FNN5-5	180–90°C for 60 min

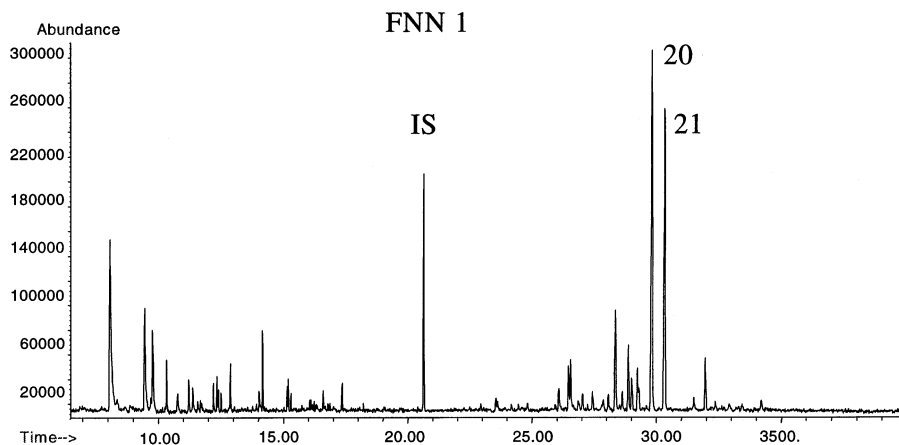


Fig. 1. Full plot total ion chromatogram of FNN1. Compound 20 and 21 are dehydro abietic acid and abietic acid, respectively.

### 3. Results and discussion

Pine tars are known to contain e.g. tricyclic diterpenoid resin acids, tricyclic diterpene hydrocarbons, alkylphenanthrenes and fatty acids [6,9–11]. Identification of compounds and tar characterisation are now usually achieved by GC-MS. Methylation of the acids with diazomethane has been the most reported derivatisation method, however, silylation has also been used [12]. In this work, silylation was preferred to methylation since the former method allows determination of naturally occurring methyl esters in the tar samples [13].

The large number of compounds in the samples calls for a high resolution method as capillary GC. However, capillary GC alone does not give complete resolution, and a pre-fractionation may be required. This, on the other hand, is quite time consuming, which is unfortunate when a large number of samples are to be analysed. Hence, we decided to use GC without pre-fractionation in our study. The components present at a concentration higher than 1% in at least one of the samples in the series were sought identified, and the major components were quantified using tri-deuterated myristic acid as internal standard. Silylated myristic acid (tetradecanoic acid) elutes in a retention window containing few tar components (Fig. 1 and Fig. 2). Since myristic acid may be naturally present in tar samples, tri-deuterated myristic acid was chosen as internal standard.

About 27 peaks in a retention time area from 19–35 min, here called ‘the resin acid region’, were further selected for characterisation (Fig. 3 and Fig. 4). Since a large number of samples are going to be compared, it is beneficial to choose a limited number of components, but from an essential and characteristic part of the chromatogram.

### 3.1. Characterisation of FNN-tar fractions from the kiln

In an earlier study, tar sample series from three likewise temperature-monitored kiln productions were analysed by GC-MS, only methylated instead of silylated [4,5]. Clear patterns emerged, regarding both the temperature development during kiln productions and that the relative concentration of each component correlated to the temperature within the kiln. The FNN 96 data presented here confirm these earlier results to a large extent (Table 3). The degree of tar acid separation is obviously affected by storing temperature and/or storing time (Table 1). Comparison of the full plot chromatograms of FNN-1 and FNN-6 (Fig. 1 and Fig. 2) shows that the prior contains a larger amount of compounds in the retention time region below 10 min than the latter. This was expected due to the difference in production temperatures within the kiln.

The most abundant components identified in ‘the resin acid region’ of FNN1–6 are presented in Table 3 by relative abundance of identified components. Compared with the earlier results from three kiln productions [5], the relative concentrations of the FNN components increase/decrease less linearly, as if FNN2 and FNN3 have been exchanged. However, this is not the case. The more even temperatures in the present kiln compared with the temperatures measured in the other kilns might explain the divergence during this interval (Table 1). The temperature of the other kilns showed a greater gap between maximum and minimum temperatures in this same time stage of production, which was 10–13 h after ignition. The fact that the flow rate (Table 1) for barrel 2 was considerably higher than for the rest might also indicate a somewhat earlier maximum yield than expected, though this expectation

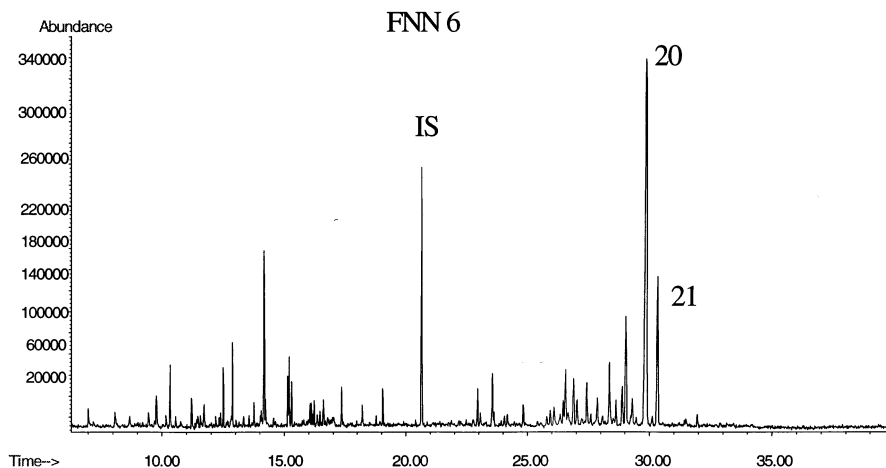


Fig. 2. Full plot total ion chromatogram of FNN6. Compound 20 and 21 are dehydro abietic acid and abietic acid, respectively.

Table 3

FNN 96 untreated tar series (1–6); amount of components given in % of sample; identification was achieved using computer matching of the mass spectra with the NIST library; probability over 90% named S, over 70% P and below T

Chemical name provided by the NIST library	Peak number	RT (min)	FNN 1	ID	FNN 2	FNN 3	FNN 4	FNN 5	FNN 6
			% in sample		% in sample				
Podocarpa-8,11,13-trien-16-al, 13 isopropyl, isomer <sup>a</sup>	2	22.99	0.55	P	1.10	1.27	1.43	1.59	1.71
Podocarpa-8,11,13-trien-16-al, 13 isopropyl, isomer <sup>b</sup>	3	23.59	0.89	P	1.45	1.92	2.10	2.32	2.50
Hexadecanoic acid, TMS	4	23.67	0.86	S	1.13	1.20	1.16	0.98	0.80
Phenanthrene, 2,5-dimethyl (m/z 253) <sup>c</sup>	5	24.09	0.42	P	0.49	0.56	0.66	0.66	0.74
Cyclopent A indene,3,8-dihydro-1,2,3,3,8,8-hexamethyl	6	24.88	0.74	P	1.16	1.21	1.34	1.43	1.49
Phenanthrene, 2,3,5-trimethyl (m/z 330) <sup>d</sup>	7	25.84	0.42	P	0.63	0.75	0.76	0.77	1.04
Podocarp-8(14)-en-15al, 13a-methyl-13-vinyl	8	25.98	0.72	P	1.05	1.19	1.22	1.17	1.24
Linoleic acid, TMS	9	26.52	2.63	S	2.83	2.78	2.19	1.64	1.35
Oleic acid, TMS	10	26.60	3.63	S	3.91	4.14	3.59	3.09	2.81
Phenanthrene, 2,4,5,7-tetramethyl	11	26.93	1.23	P	1.82	2.11	2.31	2.54	2.83
Isopimaric acid, TMS, Isomer (m/z 374)	12	27.48	1.64	P	2.38	2.46	2.49	2.42	2.90
Isopimaric acid, TMS, Isomer (m/z 374)	13	27.92	1.43	P	2.23	2.35	2.51	2.52	2.90
Pimaric acid, TMS	14	28.41	7.19	S	6.99	6.70	5.58	4.57	4.08
Sandaracopimaric acid, TMS	15	28.67	1.41	S	1.69	1.75	1.73	1.63	1.52
Isopimaric acid, TMS, Isomer (m/z 374)	16	28.93	3.91	S	3.90	3.69	3.32	2.71	2.45
Podocarpa-8,11,13-trien-15-oic acid, 13-isopropyl, methyl ester	17	29.07	2.10	S	3.88	5.20	5.40	5.77	6.07
Palustric acid, TMS	18	29.29	2.57	P	3.30	3.33	2.24	1.75	1.11
Laevopimaric acid, TMS	19	29.34	1.26	T	1.49	1.50	1.59	1.45	1.60
Dehydroabietic acid, TMS	20	29.89	25.09	S	30.21	31.64	30.84	31.51	30.54
Abietic acid, TMS	21	30.37	20.31	S	20.26	18.50	14.41	11.38	8.87
Neobietic acid, TMS	22	32.01	3.65	P	3.51	3.40	2.34	1.52	1.40
Sum % response:			82.65		95.41	97.68	89.21	83.42	79.95

<sup>a</sup> Full name from NIST library, 1-Phenanthrenecarboxaldehyde, 1,2,3,4,4A,9,10,10A-octahydro-1,4A-Dimethyl-7-(1-Methylethyl)-, 1S-(1.alpha,4a.alpha., 10a.beta.).

<sup>b</sup> Full name from NIST library, 1-Phenanthrenecarboxaldehyde, 1,2,3,4,4A,9,10,10A-octahydro-1,4A-Dimethyl-7-(1-Methylethyl)-, 1S-(1.alpha,4a.alpha., 10a.beta.).

<sup>c</sup> m/z 253 also found for this compound.

<sup>d</sup> m/z 253 also found for this compound.



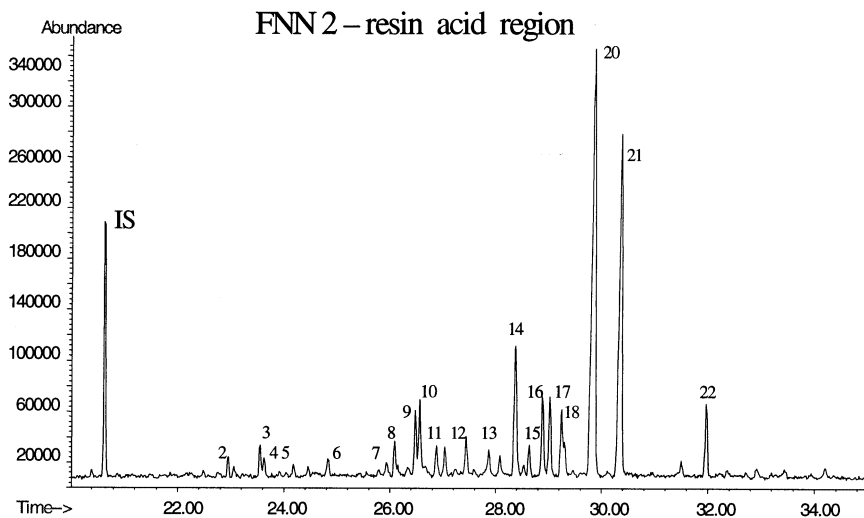


Fig. 3. 'Resin acid region' plot of FNN2. See Table 3 for identification of compounds.

is based solely on a general impression rather than flow rate measurements from the other tar kilns. Barrel 2-tar is more viscous and grainy compared with barrel 5-tar, which is smooth.

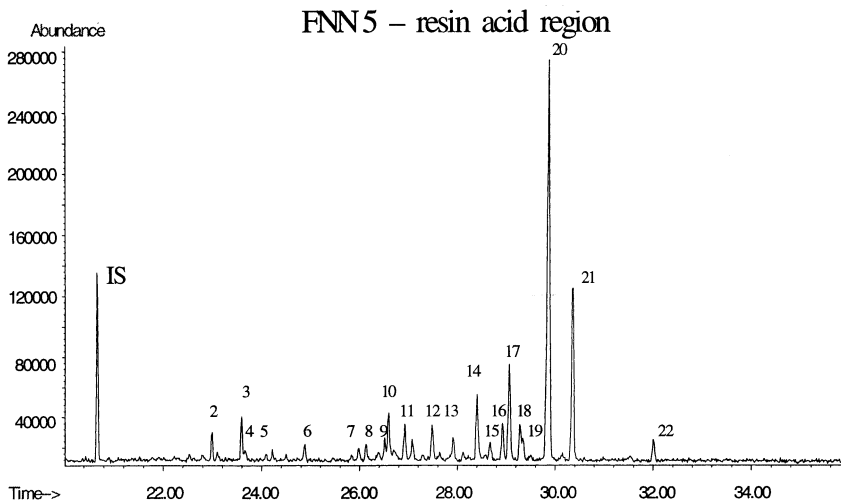


Fig. 4. 'Resin acid region' plot of FNN5. See Table 3 for identification of compounds.

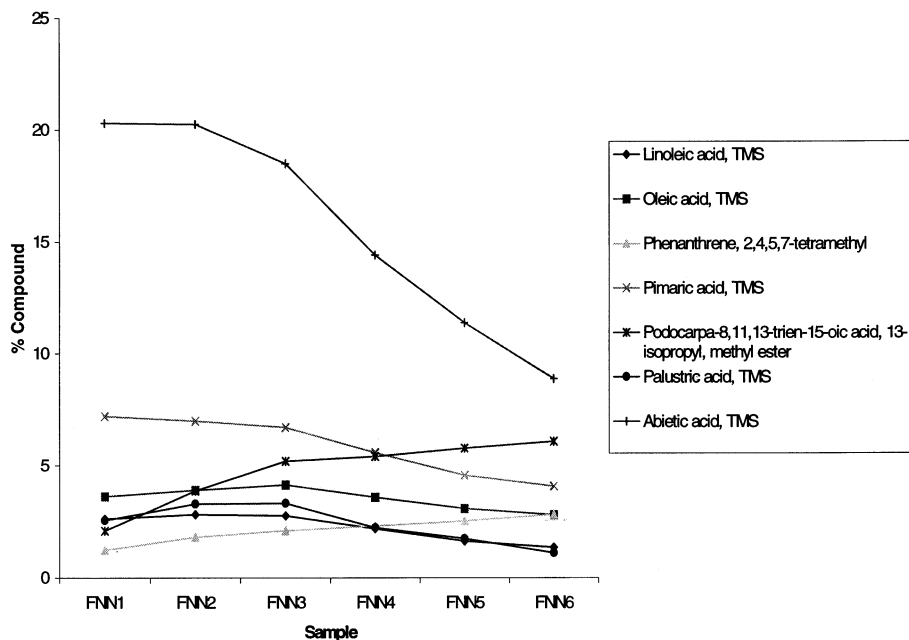


Fig. 5. Relative abundance of some fatty- and resin acids in FNN1–6.

The percentages of total response given in Table 3 assume equal response factors (RF) of components and internal standard. This simplification is necessary due to the lack of references and known RF for every single component. Aliphatic compounds probably have a lower RF than aromatic compounds at the chosen MS conditions.

The main components in the pine tar samples are dehydro abietic acid and abietic acid, peak number 20 and 21 in Figs. 1–3 and Fig. 4. As in the earlier study [5], the content of abietic acid was decreasing during kiln production. The amount of dehydro abietic acid increased in the earlier study, while in the present, dehydro abietic acid remains at a constant level during the production. However, the peak height ratio between the peak 20 and 21 shows a linear evolution. Fig. 5 presents graphically the relative abundance of some resin- and fatty acids. The change in relative abundance of the components with increased temperature, reflects a tendency of dehydrogenation and decarboxylation of components, towards a higher degree of aromatisation of the cyclic compounds. This is typical for tar made in kilns and tar derived from different temperature stages in the production. In traditional tar trade, both first, second, as well as third class tar were marketed [14,15], a fact which verifies that the tar from the kiln stages were kept separate. In all of the investigated samples, only one naturally methylated methyl ester, number 17 (Table 3 and Figs. 3 and 4), was identified in the resin acid region.

Table 4

FNN 96-2 series (FNN2-1–FNN2-5); amount of components given in % of sample; identification was achieved using computer matching of the mass spectra with the NIST library; probability over 90% named S, over 70% P and below T

Chemical name provided by the NIST library	Peak number	RT (min)	FNN 2	ID	FNN 2-1	FNN 2-2	FNN 2-3	FNN 2-4	FNN 2-5
			% in sample	% in sample					
Podocarpa-8,11,13-trien-16-al,13 isopropyl, isomer <sup>a</sup>	2	22.99	1.10	P	1.20	1.42	1.32	1.22	1.25
Podocarpa-8,11,13-trien-16-al,13 isopropyl, isomer <sup>b</sup>	3	23.59	1.47	P	1.84	2.37	1.84	1.84	1.85
Hexadecanoic acid, TMS	4	23.67	1.10	S	1.18	1.28	1.10	1.13	1.15
Phenanthrene, 2,5-dimethyl (m/z 253) <sup>c</sup>	5	24.09	0.56	P	1.12	1.19	0.62	0.61	0.64
Cyclopent A indene,3,8-dihydro-1,2,3,3,8,8-hexamethyl	6	24.88	1.17	P	1.21	1.45	1.28	1.33	1.34
Phenanthrene, 2,3,5-trimethyl (m/z 330) <sup>d</sup>	7	25.84	0.50	P	0.69	1.10	0.69	0.75	0.66
Podocarp-8(14)-en-15al, 13a-methyl-13-vinyl	8	25.98	1.13	P	1.18	1.49	1.18	1.30	1.25
Linoleic acid, TMS	9	26.52	2022	S	2.59	2.73	2.66	2.93	3.08
Oleic acid, TMS	10	26.60	3.88	S	4.11	4.41	4.08	4.19	4.33
Phenanthrene, 2,4,5,7-tetramethyl	11	26.93	1.91	P	2.30	2.37	2.03	2.04	2.05
Isopimaric acid, TMS, Isomer (m/z 374)	12	27.48	2.35	P	2.42	2.54	2.49	2.50	2.60
Isopimaric acid, TMS, isomer (m/z 374)	13	27.92	1.75	P	1.98	2.58	2.37	2.41	2.35
Pimaric acid, TMS	14	28.41	6.89	S	7.20	7.89	7.22	7.32	7.62
Sandaracopimaric acid, TMS	15	28.67	1.83	S	2.19	2.30	1.84	1.91	1.94
Isopimaric acid, TMS, Isomer (m/z 374)	16	28.93	3.48	S	3.75	4.23	3.90	4.03	4.18
Podocarpa-8,11,13-trien-15-oic acid, 13-isopropyl, methyl Ester	17	29.07	3.94	S	4.45	4.87	4.46	4.63	4.71
Palustric acid, TMS	18	29.29	3.30	P	4.73	4.72	4.33	4.80	4.21
Laevopimaric acid, TMS	19	29.34	1.49	T	1.89	1.90	1.74	1.73	1.67
Dehydroabietic acid, TMS	20	29.89	28.55	S	29.00	30.79	31.60	32.43	32.91
Abietic acid, TMS	21	30.37	18.32	S	18.16	19.78	21.70	23.55	24.30
Neoabietic acid, TMS	22	32.01	3.56	P	4.06	4.77	3.77	3.53	3.38
Sum % response:			90.50		97.25	106.18	102.22	106.18	107.47

<sup>a</sup> Full name from NIST library, 1-Phenanthrenecarboxaldehyde, 1,2,3,4,4A,9,10,10A-octahydro-1,4A-Dimethyl-7-(1-Methylethyl)-, 1S-(1.alpha,4a.alpha.,10a.beta.).

<sup>b</sup> Full name from NIST library, 1-Phenanthrenecarboxaldehyde, 1,2,3,4,4A,9,10,10A-octahydro-1,4A-Dimethyl-7-(1-Methylethyl)-, 1S-(1.alpha,4a.alpha.,10a.beta.).

<sup>c</sup> m/z 253 also found for this compound.

<sup>d</sup> m/z 253 also found for this compound.

### 3.2. Stir tar and pitch obtained after heat treatment of tar

This work shows that the temperature of the kiln has a significant impact on the relative concentration of the components, which makes fingerprint characterisation for each fraction possible and relevant. Furthermore, the question was whether a following heat treatment would make the composition of samples less distinct, that is to make an early tar sample more similar to a late tar sample. Tar from FNN barrel number 2 and 5 were selected for heat treatment and two subsamples from each of the 25 l samples were used. The untreated samples (in Table 4), FNN 2 and 5, were derived from the 1.5 l bottles. One of the FNN2 subsamples was heated at 120–30°C, FNN 2-1, 2-2, and one at 180–190°C, FNN 2-3, 2-4, 2-5, Table 4. Provided that the samples were homogenous, it was expected that the relative abundance of each component would increase proportionally with increase in temperature and heating time. This is due to the fact that the components in the resin acid region is less volatile, and thus will constitute a larger part of the sample after heating and increased evaporation. This is illustrated in the second from the bottom row in Table 4. The increase in concentration of ‘resin acid’ compounds with increased temperature/time is larger than their initial subsamples variations. Therefore, it can be concluded that temperatures below 190°C do not result in any degradation of the compounds, but their concentration increase due to evaporation of the more volatile compounds of the tar. The only component of the FNN2-sample not showing a steady increase in concentration was palustric acid (peak number 18 in Fig. 3 and Fig. 4). The same tendency as discussed for FNN2 was observed with the FNN5 (results not shown).

## 4. Concluding remarks

The GC-MS data presented in this work show that successively drained tar fractions from a traditional kiln production can be recognised by their content of compounds in the resin acid region. These fractions are still recognisable by GC-MS after subsequent heat treatments at 180–190°C for 60 min. Boiling of the tar does not seem to make the initial fractions more similar in resin acid composition. The viscosity of both initial tar fractions increased after heat treatments, however, the difference in viscosity of the fractions is still evident. Further studies will focus upon which of the initial tar fractions ought to be used in specific climatic conditions concerning the maintenance of stave churches, and whether the initial characteristics are still recognisable by GC-MS after weathering.

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