

Appendix V: Comparison of methods and experimental setup

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Jotun/the doctoral thesis of Kidane Fanta Gebremariam (KFG/Jotun), 2009-2016

Instrument: CDS Pyroprobe 5250 (Chemical Data Systems) pyrolyser coupled online with 7890A gas chromatography system (Agilent Technologies) and a 5975C mass selective detector (Agilent Technologies).

The Chemical Specialisation Project of Tim Lauscke (TL), 2016, and this thesis by Marie-Louise Tambo Magni (MTM), 2017-2019

Instrument: Pyrola 2000 (Pyrol AB) pyrolyser coupled online with Trace GC Ultra gas chromatography system (Thermo Scientific) and an ITQ 1100 ion trap mass spectrometer (Thermo Scientific).

Table AV-1: Sample preparation

Parameter	KFG/Jotun	TL	MTM
Sample size	0,1 mg or less	0,1 mg or less	0.5-2 mm ² /1-5 µL
TMAH	8 µL, 25 % in H ₂ O	3 µL, 25 % in H ₂ O	1 or 3 µL, 25 % in H ₂ O
Sample application site	In quartz tube w/quartz wool beneath and on top, placed in pyroprobe wire	Directly on platinum filament	Directly on platinum filament

Table AV-2: Pyrolysis parameters

Parameter	KFG/Jotun	TL	MTM
Chamber temperature, T _c	<i>No chamber in CDS pyroprobe</i>	175°C	175°C
Pyrolysis temperature (T _p , time (t ₂))	850 °C, 10 sec	700°C, 2 sec	700°C, 2 sec

Table AV-3: GC parameters

Parameter	KFG/Jotun	TL	MTM
Split ratio	Split 1:50	Split 1:15	Split 1:15
Inlet temperature	280°C	280°C	280°C
Column parameters	30 m x 0.25 mm x 0.0250 µm	30 m x 0.25 mm x 0.0250 µm	30 m x 0.25 mm x 0.0250 µm
Column type	DB-5M UI Phenyl-arylene/methyl polysiloxane,	DB-5-MS fused silica WCOT 5 % phenyl 95 % dimethyl arylene siloxane	DB-5-MS fused silica WCOT 5 % phenyl 95 % dimethyl arylene siloxane
GC Temperature Programme	60°C (6min)-> 20°C/min -> 340(2 min)	40°C(5min)-> 5°C/min - > 250°C -> 10°C/min - >300°C(5 min)	<u>Samples (TP1):</u> 40°C (5min)-> 5°C/min -> 250°C -> 10°C/min - >300°C(5 min) <u>Blanks (TP2):</u> 40°C-> 15°C/min - >300°C(6 min)
Carrier gas type	Helium	Helium	Helium
Carrier gas flow	1 mL/min	1,5 mL/min	1,5 mL/min
Transfer line temperature	300°C	300°C	300°C

Table AV-4: MS parameters

Parameter	KFG/Jotun	TL	MTM
EI	70 eV	70 eV	70 eV
Scanning range	Unknown	<u>Samples pyrolysed in presence of TMAH:</u> 45-600 for 2 min -> 60-600m/z (ScA in MTM method).	<u>Sample:</u> ScA: 45-600 for 2 min -> 60-600m/z ScB: 60-600 for 2 min-> 45-600m/z <u>Blanks:</u> ScC: 40-600 m/z

Table AV-5: Identification

Parameter	KFG/Jotun	TL	MTM
Identifications	NIST 08 MS Library	NIST 2.0f (2008) NIST 08 MS Library and reference material	NIST 2.0f (2008) NIST 08 MS Library and reference material
Software	ChemStation	Xcalibur	Xcalibur