# AIR EMISSIONS FROM SMALL-SCALE BURNING TESTS OF CRUDE AND HEAVY FUEL OIL

Scientific Report from DCE – Danish Centre for Environment and Energy No. 515 2022

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# Data sheet

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Abstract: Combatting oil spills in high Arctic marine waters may be extremely challenging. In situ burning of oil spills is recognised as an effective removal method for oil spills in the sea. The aim of this project is to increase the knowledge and understanding of potential environmental implications related to combatting oil spills in ice-infested waters by in situ burning. The project included measurements of air emissions from burns of oil in a small-scale burning cell and a cone calorimeter.

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# Contents

# Preface

This report describes the results of small-scale laboratory experiments of burning crude oil. The study is part of the *Strategic Environmental Study Program for Northeast Greenland* and is specifically included in the research theme: “In situ burning of oil spills in high arctic ice infested waters”.

The Strategic Environmental Study Program for Northeast Greenland was funded by Environment Agency for the Mineral Resources Activities (EAMRA), Greenland Government.

The DCE study program on environment and raw material extraction in Greenland supported by the Ministry of Environment of Denmark.

The study was carried out as a collaboration between Aarhus University (AU), DCE – Danish Centre for Environment and Energy and DBI – The Danish institute of Fire Safety and Security Technology.

# Summary

Combatting oil spills in high Arctic marine waters may be extremely challenging due to the presence of ice, remoteness, darkness and difficult access. In situ burning of oil spills is recognised as an effective removal method for oil spills in the sea. Ice on the water can contribute to reduce oil weathering and also, under the right circumstances, contain an oil slick as a thick layer on the water surface that can be ignited. Therefore, in situ burning is often mentioned as a promising method for oil spill response in the Arctic.

The aim of this project is to increase the knowledge and understanding of potential environmental implications related to combatting oil spills in ice-infested waters by in situ burning. The project included measurements of air emissions from burns of oil in a small-scale burning cell and a cone calorimeter. A crude oil and a bunker fuel oil were included and burned on the top of seawater or fresh water. The measurements from the small-scale burns of oil had focus on soot, the blackish or brownish substance formed during incomplete combustion. Measured parameters were organic carbon (OC), elemental carbon (EC) as well as dioxins and PAHs. Soot is of concern in relation to the warming of the Arctic environment, and deposits of dark matter on snow and ice surfaces can reduce the albedo and thereby enhance ice melt. In situ burning of oil may generate persistent organic pollutants (POPs) such as dioxins and PAHs that pose a risk for marine mammals and humans.

The results from the burning cell showed that burning of bunker fuel on seawater produced the highest amount of soot. The soot emission decreased with increasing mass loss for the freshwater experiments, whereas the picture was not as clear for the burns on seawater. The results thus indicate that the type of water basin (salt/fresh) might influence the amount of soot formed. Further, the highest concentrations of PAHs in the soot were measured for the bunker fuel experiments on seawater. In general, regardless of oil type, for the PAH composition, there is a shift from a 2- and 3-ring dominated oil to the 4-6-ring dominated soot. These larger PAHs are more persistent in the environment than lower molecular weight PAHs. In contrast to measurements of PAHs, dioxins were below the analytical detection limit in all samples.

The cone calorimeter tests showed that increasing external heat (incident heat) led to increased soot concentrations and that the soot concentrations were higher for bunker fuel than for crude oil. Opposite to the burning cell experiments, the soot concentration increased with increasing mass loss. This highlights that it is not simple to reproduce burn tests as just slight changes in a few physical and chemical parameters can affect the actual burning process/fire dynamics significantly and hence the derived results. This also highlights the complications of reproducing full-scale offshore burning events in the laboratory.

The cone calorimeter soot measurements were extrapolated to give estimates of the total amount of soot produced during the burns, which makes up for between 0.1 and 38% of the burned oil depending on the external heat input.

# Resumé

Bekæmpelse af olieudslip i højarktiske havområder kan være ekstremt udfordrende på grund af tilstedeværelsen af is, afsides beliggenhed, mørke og vanskelig adgang. In situ afbrænding af olieudslip, dvs. direkte afbrænding af olien på havoverfladen, anerkendes som en effektiv metode til at oprense olieudslip i havet. Is på vandet kan bidrage til at reducere forvitringen af olien og også, under de rette omstændigheder, inddæmme olien, så den samles i en tykt lag på vandoverfladen, der kan antændes. Derfor nævnes in situ afbrænding ofte som en lovende metode til bekæmpelse af olieudslip i Arktis.

Formålet med dette projekt er at øge kendskabet til og forståelsen af potentielle miljømæssige konsekvenser i forbindelse med bekæmpelse af olieudslip i isede farvande ved in situ afbrænding. Projektet omfattede målinger af luftemissioner fra afbrænding af olie i en lille afbrændingscelle og et cone-kalorimeter. En råolie og en bunkerolie blev afbrændt oven på havvand eller ferskvand. Målingerne fra lille-skala-afbrændinger af olie havde fokus på sod, det sorte eller brunlige stof, der bliver dannet under en ufuldstændig afbrænding. De målte parametre var organisk kulstof (OC), elementært kulstof (EC) samt dioxiner og PAH'er. Sod er bekymrende i forhold til opvarmningen af det arktiske miljø, og aflejringer af sort kulstof på sne- og isoverflader kan reducere albedoen og derved øge isafsmeltningen. In situ afbrænding kan generere persistente organiske miljøgifte (POP) såsom dioxiner og PAH'er, der udgør en risiko for havpattedyr og mennesker.

Resultaterne fra afbrændingscellen viste, at afbrænding af bunkerolie på havvand genererede den højeste mængde sod. Sodudledningen faldt med stigende massetab i ferskvandseksperimentet, mens billedet ikke var helt så klart for afbrændingerne på havvand. Resultaterne indikerer således, at vandtypen (salt / frisk) kan påvirke mængden af dannet sod. Desuden blev de højeste koncentrationer af PAH'er i soden målt i bunkerolien på havvand. Generelt, uanset olietype, sker der et skift i PAH-sammensætningen fra en olie domineret af 2 og 3 ringe til sod domineret af 4-6-ringe. Disse større PAH'er er mere persistente i miljøet end PAH'er med lavere molekylær vægt. I modsætning til målinger af PAH'er lå dioxinerne under den analytiske detektionsgrænse i alle prøver.

Cone-kalorimetertestene viste, at stigende ekstern varme (indfaldende varme) førte til øgede sodkoncentrationer, og at sodkoncentrationerne var højere for bunkerolien end for råolien. Modsat eksperimenterne i afbrændingsceller steg sodkoncentrationen med stigende massetab. Dette understreger, at det ikke er enkelt at reproducere afbrændingstest, da blot små ændringer i nogle få fysiske og kemiske parametre kan påvirke den faktiske afbrændingsproces / branddynamik betydeligt og dermed de afledte resultater. Dette fremhæver også komplikationerne forbundet med at reproducere fuldskala offshore-afbrændinger i laboratoriet.

Cone-kalorimeterets sodmålinger blev ekstrapoleret for at opnå estimater af den samlede mængde sod produceret under afbrændingerne, hvilket udgjorde mellem 0,1 og 38 % af den afbrændte olie, afhængigt af den eksterne varmetilførsel.

# 1 Definitions of words, concepts and abbreviations

### Soot, black carbon, TC, EC, OC

Varying definitions and uses of the terms soot, black carbon (BC), elemental carbon (EC) and organic carbon (OC) exist. Often EC, BC and soot are used as synonyms for the most refractory and light-absorbing component of carbon-based combustion particles, though the underlying analyses are different (MST 2017). This complicates comparison of the results of different studies. Soot is the blackish or brownish substance formed during incomplete combustion of carbon-based materials. Soot is typically measured as BC by light absorption or – as in this project – its chemically inertness as EC.

Following the analytical method used in our study (section 4.4), the results are rendered by two separate fractions: organic carbon (OC) and elemental carbon (EC). In this report we will use the specific terms of either EC, OC or TC (total carbon, i.e. the sum of OC+EC) when referring to specific results from the experimental work. The EC fraction is considered to be more or less equivalent to the general concept of BC, while the OC fraction covers, e.g., PAHs. For the discussions etc., we will use soot as a general term for the products of incomplete combustion that mainly contain EC but also inorganic material (ash) and OC.

### Mass loss – burning efficiency

The success of an in situ burning operation is often described by the “burning efficiency [BE%]” which is a gravimetric estimation of the amount of oil consumed during the burn. The BE% is calculated as the weight difference in the initial oil amount before the burn and the amount of burn residue after the fire is extinguished.

However, burning efficiency is occasionally used qualitatively in the scientific literature when referring to the effectiveness of the actual burn process, e.g., ”how well it burns, meaning how well it converts the fuel to heat energy”.

To avoid confusions in the discussion of the results, “mass loss” is defined as the gravimetric estimation of the amount of oil consumed during the burn, whereas “combustion efficiency/effectiveness” is used as a qualitative description of “how well the oil burns”.

### Small-scale burning cell

The small-scale burning cell was used as a measure to imitate an in situ burning event with respect to ignitability. This set-up has previously been tested and verified against field burns (e.g. Fritt-Rasmussen and Brandvik 2011).

### Cone calorimeter

A cone calorimeter is a standardised set-up and a widely used technique for evaluating fire performance. The cone calorimeter monitors a comprehensive set of fire properties including mass loss rate, smoke production, CO production etc. Measurements in the cone calorimeter cover ignition followed by combustion of the sample. By using the cone calorimeter, it is possible to apply external heat (incident heat) to the sample.

In the cone calorimeter test, the incident heat was gradually increased to mimic the large radiative heat feedback to the oil occurring during large-scale burns.

# 2 Introduction

In situ burning of oil spills on water is recognised as an effective removal method for oil spills in both open and ice-infested waters. During open water burning, the oil slick is contained by fire resistant booms. In ice-infested waters, ice floes can be used to secure an ignitable thickness and a continuous slick. The presence of ice can also contribute to reduce the weathering of the oil, thus enlarging the window of opportunity for an in situ burning operation. Therefore, the method is often mentioned as the most promising method for oil spill response in the Arctic. Oil spill responses in High Arctic waters are facing great challenges. This is primarily due to the ice as it complicates the accessibility to the spill site and renders use of conventional response methods impossible / less efficient. Remoteness, darkness for many months of the year and lack of infrastructure also add to the challenges.

In situ burning is an oxygen-starved combustion process where the oil is converted to $CO_2$, water, soot and other combustion products. The mass loss, e.g., how much of the oil is removed from the water surface, is typically in the range of 85-90%. During the *Deepwater Horizon* incident in the Gulf of Mexico 411 burns were conducted (Allen et al. 2011), and the estimated mass loss was 85% (Stout and Payne 2016). After flame out, burn residues are left at the place of the burning. These residues might sink and potentially impact the seabed community. Furthermore, though the overall amount of oil is reduced by the burn, there seems to be a relative increase in larger poly-aromatic hydrocarbons in the residue (Fritt-Rasmussen et al. 2015). Hence, formation of pyrogenic compounds and other contaminants may be a direct result of in situ burning.

The aim of this project was to analyse potential air emission of substances of concern in relation to in situ burning in order to enhance our understanding of the resulting environmental impacts from such oil spill response operations. A number of laboratory experiments simulating burning of an oil slick were undertaken to measure the development of soot, i.e. the blackish or brownish substance formed during incomplete combustion (Andrea and Gelencsér 2006). Soot is typically measured by exploiting its light-absorbing properties as black carbon (BC) or, as in this project, its chemical inertness as EC. Also measured were PAHs and dioxins. The dominant source of dioxins in the Arctic is long transport from lower latitudes (Burkow and Kallenborn 2000). In situ burning of oil may become a local source of these persistent compounds if generated in significant quantities as the prerequisite conditions for formation are present i.e. incomplete combustion and availability of chloride from seawater (Aurell and Gullett 2010). Dioxins as well as PAHs generated during burning of oil are of concern due to their high persistence and risk of toxic effects on marine mammals and humans (AMAP 2004). The presence of soot and BC in the Arctic may have both direct and indirect climatic effects due to the strong absorption capacity for solar radiation (AMAP 2015). Soot and BC deposited on snow and ice will lower the albedo (i.e. the surface reflection) and increase surface melting (AMAP 2015). Owing to the pristine atmospheric environment at high latitudes (Villum Research Station Nord 2018), even small absolute increases in soot concentrations may have a relatively large impact on the Arctic climate. Hence, it is relevant to understand the possible formation and emissions of soot from in situ burning operations.

This project included small-scale laboratory burns and cone calorimeter tests, offering a simple means of investigating the burning of oil. The results should be regarded as input in order to catalyse the understanding and discussions of the derived environmental impacts when burning oil in the Arctic. Measurements of soot as well as dioxins and PAHs were made. Emission was measured from burning bunker oil and crude oil.

# 3 Background

In situ burning is an oxygen-starved combustion process in which not enough air / oxygen is drawn into the fire to convert the oil to a black smoke plume containing $CO_2$ (75%), water vapour (12%), soot (10%), CO (3%) and other combustion products (0.2%, including PAHs, dioxins etc.) (Tenneyson 1994). If soot particles deposit on ice, this may reduce the reflective effect of the ice and hence lead to warming and ice melt (reduced albedo effect). Soot consists primarily of carbon, but in large-scale burnings also droplets of oil can be found (NRT 1995; NRT 1992). It is challenging to actually measure the soot production from large-scale burnings as it is difficult to use the methods applicable for small-scale burnings (Fingas 2010). However, previous studies indicate a correlation between the type of burning oil and the amount of soot produced. Day et al. (1979) found that the amount of soot formed was 2-5% of the original oil mass. Fingas (2010) reported values from 1 to 16%, and Buist et al. (2013) recorded soot values from 2 to 20% for both laboratory and field test burns. During the in situ burning operations included in the response to the *Deepwater Horizon* oil blowout incident, it was estimated that $1.35 \pm 0.72 \times 10^6$ kg black carbon was released into the atmosphere, corresponding to approximately 4% of the combusted material (Perring et al. 2011).

The smoke yield expresses the amount smoke produced during a burning event. Fraser et al. (1997) estimated the smoke yield based on the fire diameter, where the smoke yield increases with the fire size diameter up to diameters of approximately 2 m (Fraser et al. 1997), after which it becomes steady (Evans et al. 2001; Notarianni et al. 1993). Experimental burnings of the Alaskan North Slope crude oil showed that the plume height of the emitted smoke could extend between 400 and 550 m into the atmosphere (McGratten et al. 1997). The time to clear the air depends on meteorology and topography. If the weather conditions are calm, the plume might remain stationary and diffuse slowly both horizontally and vertically (Day et al. 1979).

The smoke from the burn may cause health problems for humans if inhaled in high concentrations (Buist 2000). This could also be an issue for wildlife and livestock (animal congregations) nearby. According to the Alaska Regional Response Team (ARRT 2008), the estimated safety zone, i.e., the minimum distance to inhabited areas or animal congregations, is 3-4 miles (5-6.5 km) in downwind direction. This is based on the standards for air pollution of the US Environmental Protection Agency (US EPA) and modelling of particle concentrations in the smoke in the wind direction. This distance also corresponds to that identified by Potter and Buist (2008), who found that the soot concentration on the sea surface is insignificant up to a distance of 3-6 km (2-4 nautical miles) from the in situ burning operation. This is presumably due to the rise of the smoke into the air because of the heat from the burning.

The type and amounts of PAHs in the smoke produced during a burn may vary depending on the chemical composition of the original oil. PAHs, especially higher molecular weight PAHs with a high ring number, are of concern due to their moderate degradability, high bioaccumulation potential and toxicity to aquatic organisms. PAHs are largely consumed during the fire, so, overall, there is a net loss of PAHs. However, the concentration of PAHs with a higher ring number (five or more rings) is higher in the smoke than in the crude oil (Benner et al. 1990; Fingas et al. 1996 Fingas et al. 2001).

Therefore, the larger PAHs are either created or produced during the fire (Fingas et al. 1996).

Dioxins and dibenzofurans are toxic and environmentally persistent compounds that – until recently – were not thought to be produced during the burning of oil (Fingas et al. 1993; Fingas et al. 1996). During the *Deepwater Horizon* oil spill response operations in 2010, emissions from the burns were sampled. Here it was found that PCDD/PCDF (polychlorinated dibenzodioxins/polychlorinated dibenzofurans) could be detected in the smoke from the burns (Aurell and Gullett 2010), indicating the occurrence of a possibly overlooked environmental impact of in situ burns.

# 4 Materials and methods

The experimental work included small-scale laboratory burns and cone calorimeter tests.

## 4.1 Small-scale laboratory burns

A small-scale burning cell was used to simulate an in situ burning event. This set-up has previously been tested and verified against field burns (e.g. Fritt-Rasmussen and Brandvik 2011). The burning cell consists of a steel cup filled with water (seawater or fresh water was used; T = 0 < 10 °C) on which the oil sample (approximately 120 g of oil) was burned. Typically, laboratory burns on water involve tap water with a low salt content. Hence, to see if the water bath, either fresh or salt, had any influence on the results, both types were included in the experiments. The burning cell was double layered, wherein cooling water flowed (500 ml / min, <20 °C). Three thermo elements measured the temperature in the seawater, oil and flame. The oil was ignited with a butane torch. The burn residue was collected with pre-weighed oil absorption pads (3M) and reweighed to determine the mass loss.

The experiments were conducted under a large open hood (3x3 m) at the laboratory facilities of the Danish Institute of Fire and Security Technology. The inlet of the hood (D = 400 mm) had a flow of 0.6 $m^3$/s. A special set-up was built to sample smoke from the controlled small-scale laboratory burns. For each burn event, samples for determination of elemental and organic carbon (EC / OC) particulates were sampled on a planar quartz filter (Whatman QMA grade filter, D=85 mm). Samples for dioxin measurements were sampled on a depth filter / XAD-2 combined filter. Three replicate burns were completed for each sample for the dioxin measurements. A pump was connected to the filter holder as well as to the dioxin tube with a flow of 17 L / min and 11 L / min, respectively. Both filter holders were placed in the fume hood inlet. See pictures below for illustrations of the set-up (Figure 4.1) as well as Table 4.1 for the experimental set-up matrix.

**Table 4.1.** Experimental set-up matrix for the small-scale laboratory burns. Numbers represent burns, samples for EC/OC measurements and samples for dioxin measurements.

| | Water bath | IFO180 | Crude oil | No oil | Comments |
|---|---|---|---|---|---|
| Tests | Salt | 3-3-1 | 3-3-1 | | |
| | Fresh | 3-3-1 | 3-3-1 | | |
| Controls | Fresh | | 2-2-0 | | |
| | | | | 0-1-1 | Placed in fume hood |
| | | | | 0-1-1 | Placed in the laboratory for background measurements |

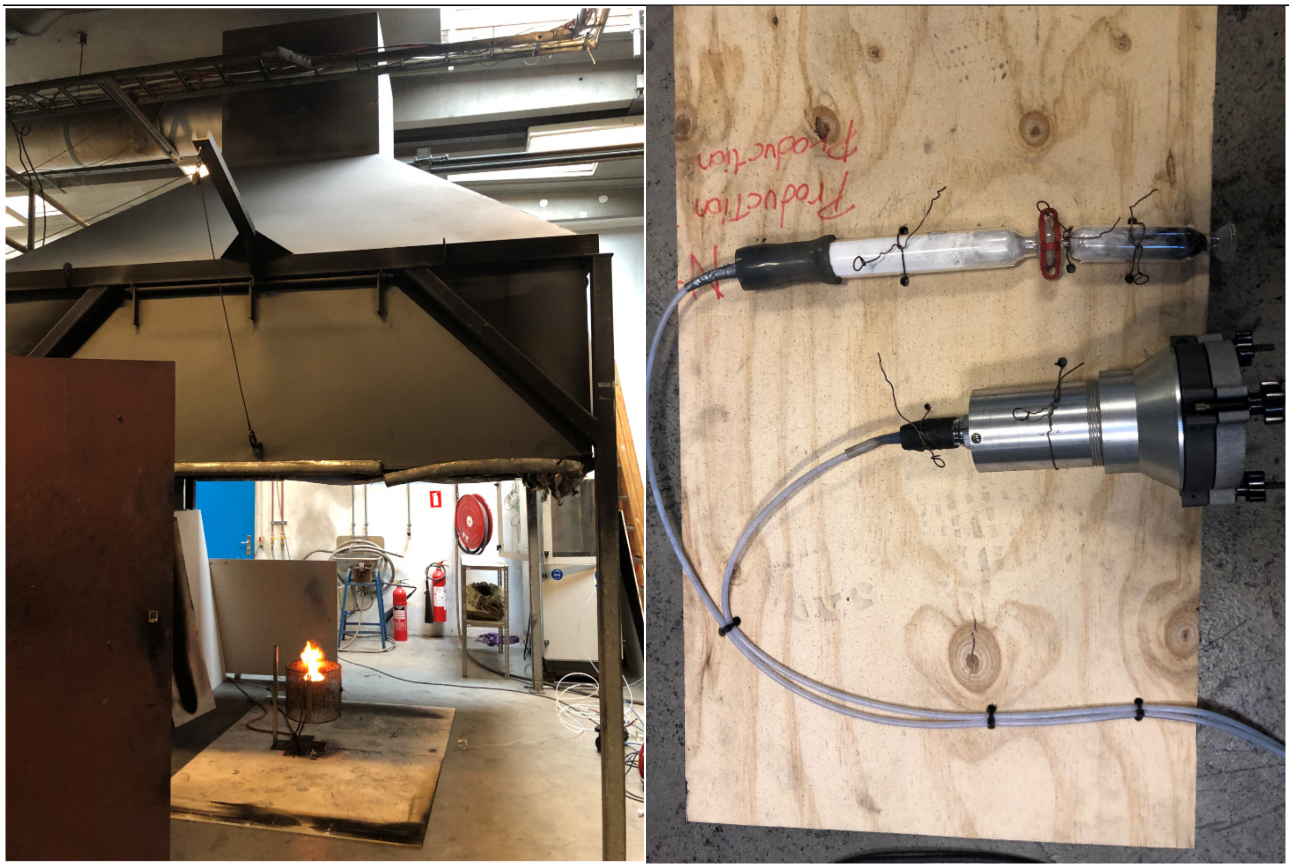


**Figure 4.1.** Pictures of the small-scale laboratory burn set-up (left) and the set-up for smoke sampling (right). Top filters were used for dioxin measurements and the bottom filter for EC/OC measurements.

## 4.2 Cone calorimeter

To evaluate the impact of incident heat on the formation of burn products and burn residues, burn tests were conducted using a cone calorimeter with controlled incident heat fluxes. A cone calorimeter is a standardised set-up and a widely used technique for evaluating fire performance. The cone calorimeter monitors a comprehensive set of fire properties, including mass loss rate, smoke production, CO production etc. Measurements in the cone calorimeter cover ignition followed by combustion of the sample. It is possible to apply different external heat fluxes (incident heat) to the sample.

A steel container (inner height 100 mm and inner diameter 82 mm) was filled with seawater (T = 0 - 10 °C), and an oil layer of approximately 5 mm was added on top. The freeboard from the oil surface to the top of the container was approximately 5 mm. The steel container was placed on a scale in the cone calorimeter to continuously measure the weight loss during the burn. The oil was ignited by use of a butane torch. Incident heat fluxes of 0, 13, 38.7 and 64.6 kW/m$^2$ were tested. The burn experiments using an incident heat flux of 64.6 kW/m$^2$ produced a vigorous fire that had to be extinguished before completion to avoid damage to the cone calorimeter. Smoke was sampled on a planar quartz filter (Whatman QMA grade filter, D=55 mm). The filter pump flow was 0.27 L/min in most of the experiments and 0.55 L/min. in a few additional tests.

The overall experimental matrix for the cone calorimeter burn tests is given in Table 4.2. The cone calorimeter is shown in Figure 4.2. The cone calorimeter tests followed the ISO 56660 – “Part 1, regarding heat release rate (cone calorimeter method) and smoke production rate (dynamic measurement)”.

**Table 4.2.** Experimental matrix for the cone calorimeter burn tests.

| **Burn ID** | **Oil type** | **Water bath** | **Filter pump flow [L/min]** | **Incident heat [kW/m$^2$]** | **Total oil mass [g]** | **Total residue mass [g]** | **Oil film thickness [mm]** |
|---|---|---|---|---|---|---|---|
| Control 1 | Crude oil | salt | Unknown | 0 | 47.6 | 19.9 | 10 |
| Control 2 | Crude oil | salt | unknown | 0 | 25.5 | 8.4 | 5 |
| Control 3 | Crude oil | salt | - | 0 | 24.5 | 9.3 | 5 |
| Control 4 | Crude oil | salt | 0.6 | 0 | 25.2 | 8.7 | 5 |
| Test 5 | Crude oil | salt | 0.27 | 0 | 25.1 | 8.7 | 5 |
| Test 6 | Crude oil | salt | 0.27 | 0 | 26.4 | 9.5 | 5 |
| Test 9 | Crude oil | salt | 0.27 | 13 | 24.9 | 9.5 | 5 |
| Test 10 | Crude oil | salt | 0.27 | 13 | 25.2 | 10.2 | 5 |
| Test 13 | Crude oil | salt | 0.27 | 38.7 | 24.6 | 12.8 | 5 |
| Test 14 | Crude oil | salt | 0.27 | 38.7 | 24.7 | 12.8 | 5 |
| Test 21 | Crude oil | salt | 0.55 | 64.6 | 24.7 | 32.5 | 5 |
| Test 7 | IFO180 | salt | 0.27 | 0 | 25.6 | 4.5 | 5 |
| Test 8 | IFO180 | salt | 0.27 | 0 | 25.4 | 3.5 | 5 |
| Test 11 | IFO180 | salt | 0.27 | 13 | 25.6 | 9.6 | 5 |
| Test 12 | IFO180 | salt | 0.27 | 13 | 25.1 | 9.5 | 5 |
| Test 15 | IFO180 | salt | 0.27 | 38.7 | 25.9 | 14.6 | 5 |
| Test 16 | IFO180 | salt | 0.27 | 38.7 | 24.7 | 17 | 5 |
| Test 19 | Crude oil | salt | 0.55 | 0 | 25.3 | 9.8 | 5 |
| Test 20 | Crude oil | salt | 0.55 | 0 | 25.2 | 9.6 | 5 |
| Test 17 | IFO180 | salt | 0.55 | 0 | 53.6 | 9.6 | 10 |
| Test 18 | IFO180 | salt | 0.55 | 0 | 50.3 | 9.6 | 10 |
| Test 22 | Crude oil | fresh | 0.55 | 64.6 | - | 28.2 | |
| Test 23 | IFO180 | fresh | 0.55 | 64.6 | - | 16.4 | |

**Figure 4.2.** Cone calorimeter at DBI.

## 4.3 Oil types

Two oil types were included in the study – a crude oil and a bunker fuel oil.

According to the SEIA for Northeast Greenland (Boertmann and Mosbech 2011), the paraffinic Statfjord crude oil is suggested as a representative crude oil for the Northeast Greenland area. However, as it was not possible to obtain the necessary amounts of Statfjord crude oil for our experiments, we used a naphthenic North Sea crude instead. The physical and chemical properties of the oils can be found in Table 7.3 together with the properties of Statfjord crude.

The bunker fuel oil included was a heavy fuel oil – IFO180. This oil type has somewhat different properties compared to the crude oil, as is evident in Table 4.3, and is a shipping fuel oil.

**Table 4.3.** Indications of the physical/chemical properties of the fresh oils used in the experiments from https://www.equinor.com/en/what-we-do/crude-oil-and-condensate-assays.html and Faksness 2008 (viscosity, North Sea crude) and Fritt-Rasmussen 2010 (Statfjord). IFO180 properties are derived from the SINTEF Oil Weathering Model (Johansen et al. 2010). Note that the values are guidance values and that variations are likely to occur.

| **Oil type** | **Density [g/cm$^3$]** | **Pour point [°C]** | **Wax [wt. %]** | **Viscosity [cP]** |
|---|---|---|---|---|
| North Sea naphthenic crude oil | 0.847 | -30 | 4.2 | 299 at 2 °C |
| Statfjord | 0.835 | -6 | 4.3 | 824 at 3 °C |
| IFO180 | 0.956 | -12 | n.a. | 10970 at 2 °C |

## 4.4 Thermal optical analysis of organic carbon and elemental carbon

The Quartz filter samples from the small-scale laboratory burn cell and cone calorimeter tests were analysed for organic carbon (OC) and elemental carbon (EC) using a Sunset Laboratory Carbon Analyzer (Department of Environmental Science, Aarhus University), which applies a thermal/optical technique by which carbonaceous material on the quartz filter is thermally desorbed in a quartz oven in two stages (Birch and Cary 1996). First, OC evolves from the sample in an inert atmosphere. EC evolves in a second step in an oxygen-containing atmosphere along with tarred material formed from pyrolysis of OC during the first stage. In each stage, the temperature is ramped up according to a protocol, EUSAAR2, which has been optimised for thermal/optical analysis of ambient aerosol to minimise tarring, harmonise OC/EC results and ease the comparison of different studies (Cavalli et al. 2010). During the entire process, the change in light transmission through the filter is monitored. Pyrolysis of OC may occur during the first stage, which will reduce the transmission as a response to the formed tar. In the second stage, it is assumed that pyrolysed OC will evolve prior to any EC in the sample and bring the laser transmission back to its initial value. Thus, the carbon detected in stage 2 until the laser reading matches its initial value is ascribed to OC. However, the assumption is not necessarily valid, and pyrolysis should therefore be reduced to a minimum (Cavalli et al. 2010). Ultimately, the “true” EC evolves, bringing transmission through the filter to a higher value than at the starting point provided that EC was present. The carbon evolved in the Thermal/Optical Carbon Analyzer is catalytically converted to methane ($CH_4$) and detected in a Flame Ionization Detector (Fine et al. 2004; McDonald et al. 2000; Sippula et al. 2007).

## 4.5 Dioxin measurements

The samples for dioxin measurements were analysed at Eurofins ÖKOMETRIC GmbH according to DIN EN 1948-2:2006 and DIN EN 1948-3:2006.

## 4.6 PAH analyses

The soot samples from the small-scale laboratory burn cell tests were analysed for the content of polyaromatic hydrocarbons (PAHs).

The samples were spiked with recovery standards (deuterium and carbon-13 labelled PAHs) and extracted by soxleth extraction for 24 hours with toluene. Next, the samples were evaporated and cleaned up on SPE silica columns, and the columns were then eluted with pentane and the solvent was changed to toluene for the GC-MS analysis.

The PAH samples were analysed on GC-MS in SIM mode. Internal standards: deuterium and carbon-13 labelled PAHs. Column: HP-5MS, length: 30 m, diameter: 0.250 mm, film thickness: 0.25 µm. Temperature program: 90 °C 1 min, increase 7 °C/min to 240 °C, 240 °C 4 min, increase 20 °C/min to 270 °C, 270 °C 18.5 min.

## 4.7 Calculations of derived parameters from the experiments

**Mass loss** is a gravimetric estimation of the amount of oil consumed during the burn. The mass loss is calculated as the weight difference in the initial oil amount before the burn and from the amount of burn residue after the fire is extinguished:

$$Mass\ loss\ [\%] = 100\left(1 - m_f/m_0\right) \qquad \textit{Equation 1}$$

$m_0$ is the weight of the initial amount of oil (g), $m_f$ is the weight of the oil residue (g).

Another and often used measure to express the burning is the **regression rate**, which is the oil thickness reduction over time (SL Ross, 1998):

$$\dot{r}''\ [mm/min] = \frac{\dot{\mathrm{m}}}{\mathrm{A}\rho_0}$$

*Equation 2 (SL Ross 1998)*

$\rho_0$ is the initial oil density of 0.850 g/cm$^3$, A is the oil slick area, and $\dot{m}$ is the **mass burning rate** defined as the mass lost per unit time of burning.

Here, we only calculated the overall mass burning rate as it was not possible to measure the weight loss during the experiments (burning cell experiments). The global mass burning rate can be calculated by the following equation:

$$\dot{m}_{global}[g/s] = \frac{(m_0 - m_f)}{t_f}$$

*Equation 3*

$t_f$ is the time of the burning experiment, $m_0$ is the mass of the initial amount of oil, and $m_f$ is the mass of the oil residues

# 5 Results and discussion

## 5.1 Small-scale burning cell

### 5.1.1 Mass loss and flame temperature

The measured mass losses were in the range 38-67% (Table 5.1). No apparent pattern emerged when comparing the measured mass losses between the two oil types. The type of water, whether salt or fresh, in the water bath beneath the oil did not appear to have any impact on the mass loss either. The lower mass losses reported here (38-67%) compared to large-scale field burning results (>80 %) were expected due to the small diameter of the burning cell and / or variations in the ambient oxygen supply to the fire. The pool size, i.e. the diameter (D) of the pool, largely determines the fire dynamics and for a fire within the range of 0.05 m < D < 1 m, the flames are considered to be in the laminar-turbulent transition regime, whereas for larger pool fires (D > 1 m) the flame regime is considered turbulent. In the transition regime, the regression rate (mm oil consumed / min burn time) increases with increasing pool diameter until the steady regression rate level in the turbulent regime is reached. The regression rates calculated for our experiments (Table 5.1) were within the same range as otherwise reported for transition regime burns, e.g., by van Gelderen (2017), having the same diameter as our set-up. The highest regression rates were found for the IFO180 burn on freshwater. This is mainly due to the duration of the burn, which was 25-40% shorter than for seawater, correlating with variations in initial oil pool film thickness and burn time. In contrast, no correlations between initial oil film thickness and mass loss or initial oil film thickness and burn rate were observed. This highlights that the burn ends when the oil film becomes too thin. The picture is, however, not as clear for IFO180 with many variations in the result, e.g., large variations in the residue amount. This is considered to be a result of the boil-over phase at the end of the burn. Boil-over occurs for oils with relatively high boiling temperatures when the water sub-layer is superheated (Brogaard et al. 2014); this is considered to be a laboratory phenomenon and unlikely to occur in the field where there is sufficient water exchange beneath the burning oil slick. In general, the regression rate, flame height and heat release rate increase markedly during a boil-over (Evans et al. 1988; Garo et al. 1994; Guénette et al. 1994).

With respect to the flame temperature, our results showed that the crude oil produced a higher flame temperature than the bunker fuel, IFO180 (Figure 5.1). The heat increase of IFO180, however, was more pronounced at the end of the burn due to the occurrence of boil-over. The higher flame temperatures found for the crude oil could be a result of a more efficient combustion process for crude oil than for bunker fuel as bunker fuel contains a considerable amount of residual heavy end oil products with a high flame point.

To sum up, the crude oil burns resulted in higher flame temperatures and more uniform results, whereas the bunker fuel, IFO180, had lower flame temperatures and demonstrated larger variation in burn results. Thus, the IFO180 burns on fresh water had the shortest burn time and the highest regressions rates, indicating that the type of water below the burn might have an impact on it.

**Table 5.1.** Key parameters of the small-scale burn experiments and derived key results of the experiments.

| Burn ID | Oil type | Water type | Total oil mass [g] | Initial oil film thickness [mm] | Residue thickness [mm] | Residue mass [g] | Burn time [min] | Mass loss* [%] | Regression rate [mm/min] |
|---|---|---|---|---|---|---|---|---|---|
| **Crude 1.F** | Crude | Fresh | 137.5 | 10.5 | 4.5 | 68.3 | 13 | 50 | 0.41 |
| **Crude 2.F** | Crude | Fresh | 137.5 | 10.5 | 4.4 | 66.9 | 11 | 51 | 0.49 |
| **Crude 3.F** | Crude | Fresh | 137.5 | 10.5 | 4.9 | 74.1 | 10 | 46 | 0.49 |
| **Crude 1.S** | Crude | Salt | 138 | 10.6 | 3.7 | 56.8 | 10 | 59 | 0.62 |
| **Crude 2.S** | Crude | Salt | 140 | 10.7 | 3.8 | 58.4 | 11 | 58 | 0.57 |
| **Crude 3.S** | Crude | Salt | 139 | 10.6 | 4.1 | 62.9 | 15 | 55 | 0.39 |
| **IFO180 1.F** | IFO180 | Fresh | 130.5 | 10.0 | 2.8 | 42.3 | 6 | 68 | 1.12 |
| **IFO180 2.F** | IFO180 | Fresh | 128.5 | 9.8 | 5.2 | 79.2 | 6 | 38 | 0.63 |
| **IFO180 3.F** | IFO180 | Fresh | 131.5 | 10.1 | 3.5 | 53.5 | 6 | 59 | 0.99 |
| **IFO180 1.S** | IFO180 | Salt | 126.5 | 9.7 | 4.1 | 62.7 | 10 | 50 | 0.49 |
| **IFO180 2.S** | IFO180 | Salt | 124.5 | 9.5 | 5.0 | 76.4 | 8[a] | 39 | 0.46 |
| **IFO180 3.S** | IFO180 | Salt | 130 | 9.9 | 2.8 | 43.2 | 8 | 67 | 0.83 |

a) = estimated from temperature measurements. * measured as oil removal during the burning

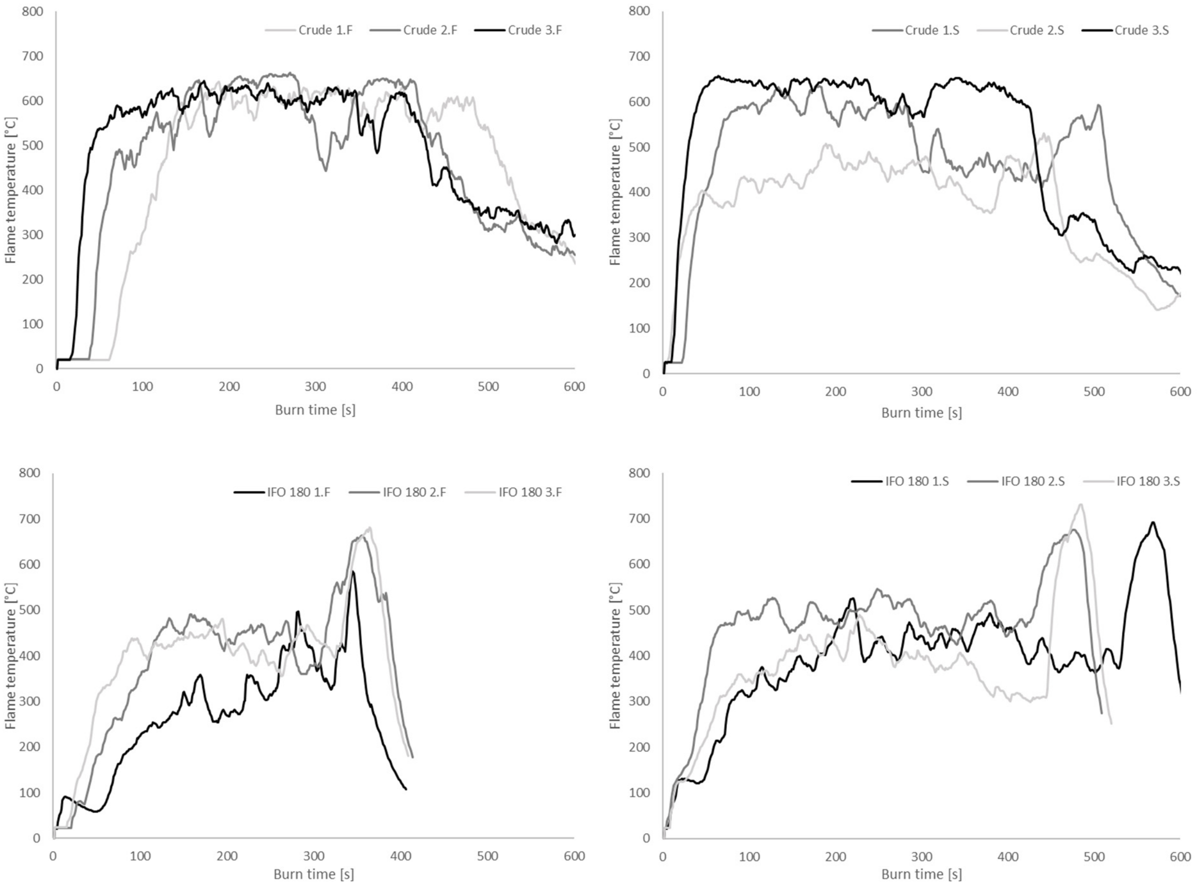


**Figure 5.1.** Flame temperature measurements in the 12 burn experiments. Top left is crude oil burns on fresh water, top right is crude oil burns on seawater, bottom left is IFO180 burns on fresh water, and bottom right is IFO180 burns on seawater. The experiments were in triplicates (1, 2, 3) on either seawater (s) or fresh water (f).

### 5.1.2 Dioxin measurements

Dioxin concentrations were not detected above the analytical detection limits for any of the samples in the small-scale burn experiments. In contrast, formation of dioxins has been indicated in large-scale in situ burn events, e.g., during the burns used as part of the response to the oil spill from the *Deepwater Horizon* blow-out incident in the Gulf of Mexico (Schaum et al. 2010). Thus, it is possible that our laboratory conditions are not suitable for generating dioxins including the set-up such as oil types, water parameters, type of flame regime (i.e. diameter of burn), which all differs from the *Deep Water Horizon* incident burns. Thus, conclusions cannot be drawn as to the decisive factors for generating dioxins, and more studies are needed to understand this issue in more detail. However, a possible mechanism behind dioxin production in the case of large-scale in situ burns could be that the large amount of oil involved and the production of heat may introduce seawater into the fire at very high temperatures, which is not seen in the smaller-scale experiments. Under real world conditions, the prolonged heat production at the immediate ocean surface may favour the conversion of chlorine in seawater into dioxins, which are then emitted to the atmosphere as either gases, particulates or droplets. It is possible that a lower thermal threshold may exist under which the conditions for dioxin formation and / or emission are not met. Dioxins could be concentrated in the burn residue (which we did not measure) instead of being emitted. Thus, which is the driving factor for dioxin production and / or emission during an in situ burn is still an open question.

### 5.1.3 Elemental carbon and organic carbon measurements

Quartz filter samples were analysed for organic carbon (OC) and elemental carbon (EC). The results are illustrated in Figure 5.2 including total carbon (∑OC+EC).

It is not possible to use the absolute values of these measurements as iso-kinetic sampling of the smoke was not obtainable in the laboratory. Hence, OC / EC / TC values were normalised to the unit “microgram C / $cm^2$ filter / gram oil burned”. The results allow us to gain an understanding of the processes and the sensitivity of the different variables (mass loss, water bath (fresh or salt) and oil type) of the study on the generation of organic and elemental carbon, i.e. the soot production (Figure 5.2).

A linear correlation between mass loss and TC content (in the soot) emerged in the freshwater experiments, where a high mass loss resulted in low TC (Figure 5.3). High mass loss is likely a result of a more effective burning process that generates less soot / smoke. The trend was not as clear for the seawater basin experiments and in particular not for the IFO180 experiments where the variation between replicates was more pronounced. The latter is considered to be a result of the vigorous boil-over at the end of these experiments (see section 4.1).

Overall, from our results it seems as if IFO180 on seawater produces the highest amount of TC and thereby has the highest potential deposition and impact on the environment surrounding the burn.

**Figure 5.2.** Organic carbon (OC), elemental carbon (EC) and total carbon content (TC) from the burns. All OC/EC/TC values are normalised to the unit “microgram C per cm$^2$ filter per gram oil burned”.

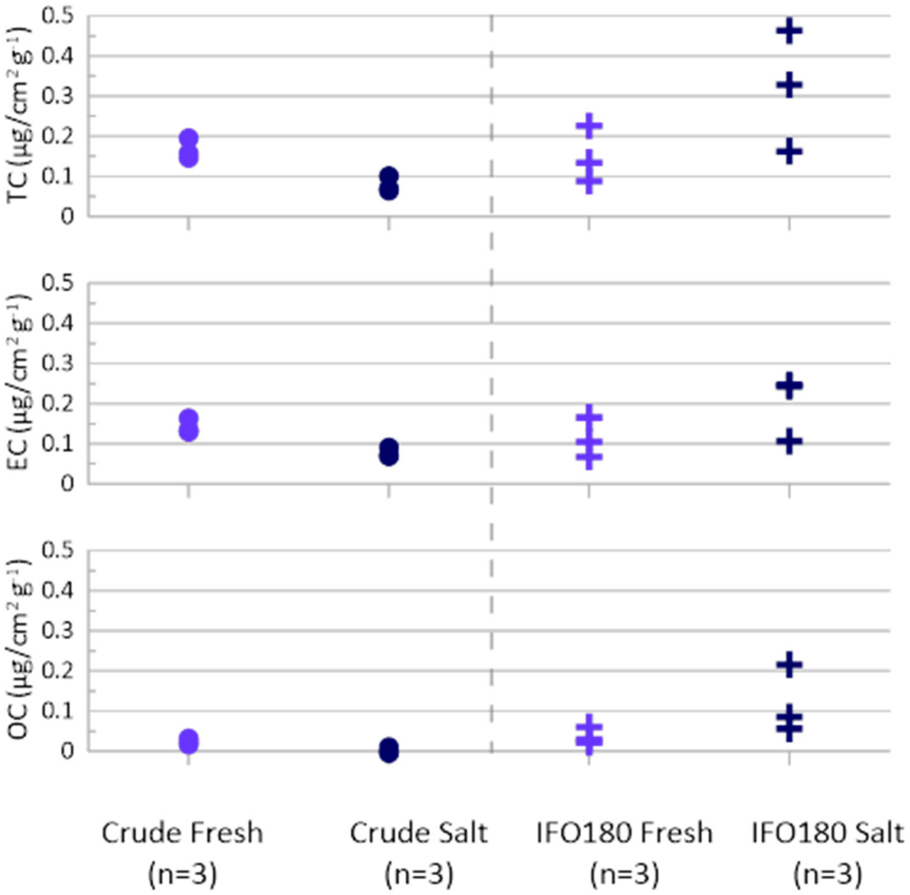
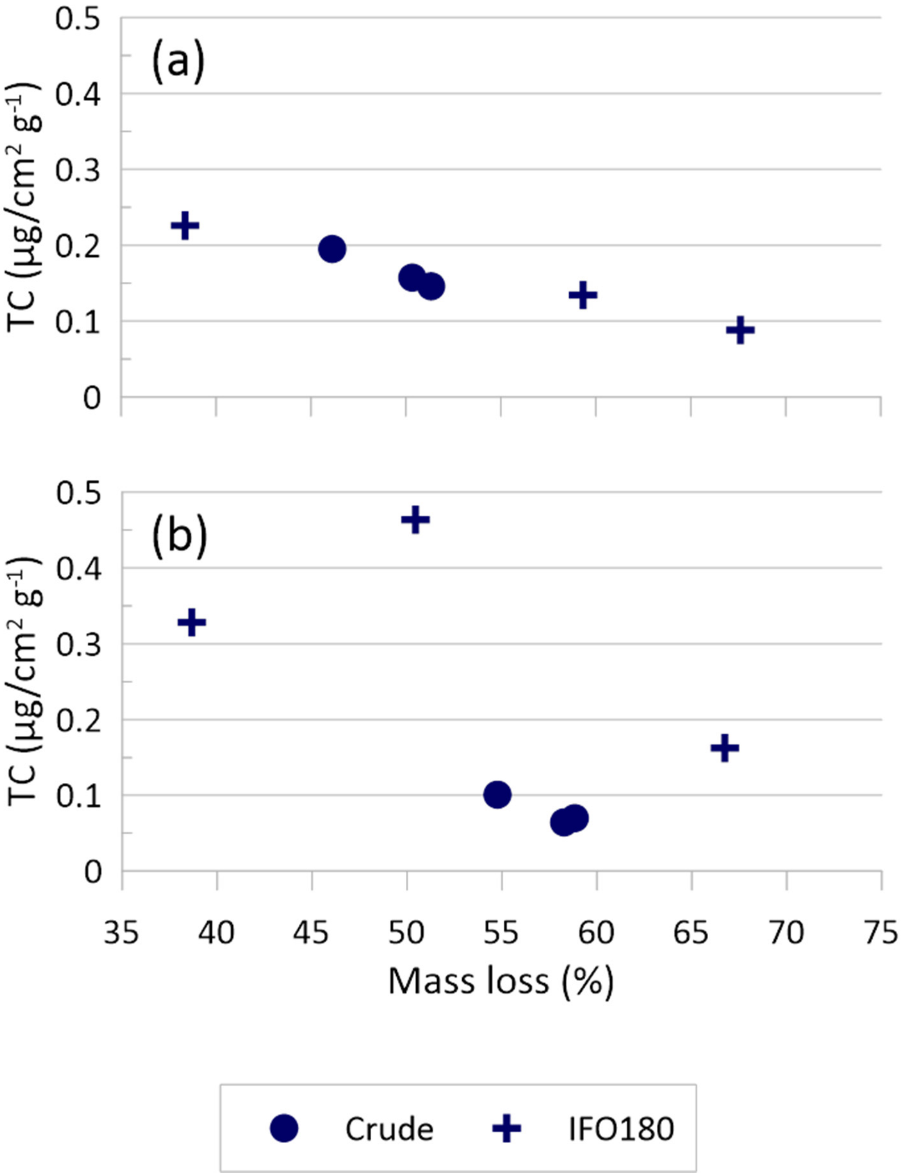


**Figure 5.3.** Carbon deposition (TC) vs mass loss. All OC/EC/TC values are normalised to the unit "microgram C per cm$^2$ filter per gram oil burned".
(a) = fresh water and (b) = seawater.



### 5.1.4 PAH content

The total concentrations of PAHs on the filter samples showed that more PAHs were measured in the IFO180 burns than in the crude oil burns (Table 5.2). Further, the highest concentrations were recorded in the IFO180 burn experiments on seawater, and these also had the highest TC production (Table 5.2 and Figure 5.2).

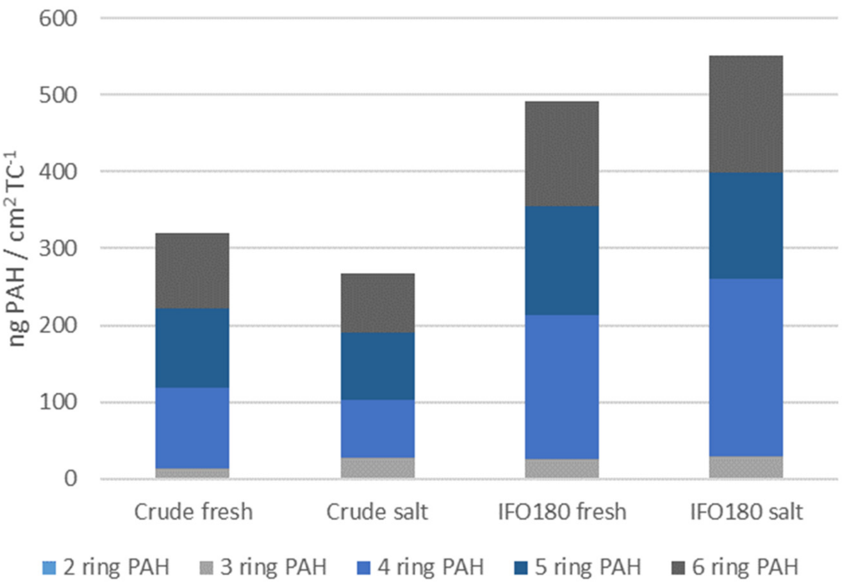
The PAHs (grouped according to the number of aromatic rings in the chemical structure) were normalised against produced TC (Figure 5.4). From this, it is evident that IFO180 burns generate more soot and that this soot also contains more PAHs.

**Table 5.2.** Total concentrations of PAHs on the filter in the different burn tests (n=3) and average TC, OC and EC values (n=3).

| Sample ID | ∑PAH [ng/cm$^2$] | Average TC [µg/cm$^2$ g$^{-1}$ oil] | Average OC [µg/cm$^2$ g$^{-1}$ oil] | Average EC [µg/cm$^2$ g$^{-1}$ oil] |
|---|---|---|---|---|
| Crude fresh | 3836 | 0.166 | 0.024 | 0.142 |
| Crude salt | 1574 | 0.078* | 0.01* | 0.077* |
| IFO180 fresh | 6240 | 0.149 | 0.037 | 0.112 |
| IFO180 salt | 12974 | 0.318 | 0.119 | 0.199 |

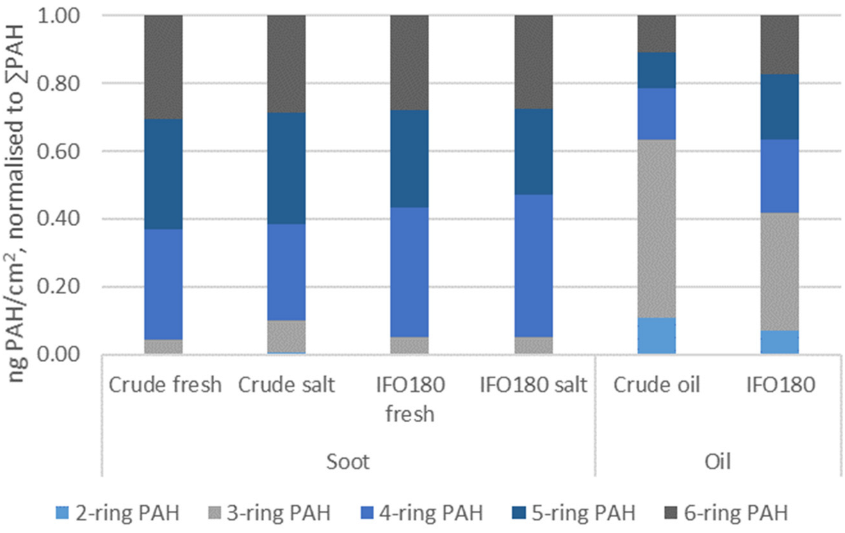
*n=1 as the rest of the samples had negative values

**Figure 5.4.** PAHs normalised to the sum of TC produced and grouped according to the number of aromatic rings in the chemical structure.



The distribution of the different PAHs in the soot is shown in Figure 5.5, together with the PAH composition of the related oils. There is a clear shift from a 2- and 3-ring dominated oil to the 4-6-ring dominated soot. The PAH composition of the soot is similar regardless of oil type and water type. This dominance of 4-6 ring PAHs is also described in Faksness et al. (2018).

**Figure 5.5.** Distribution of PAHs in the soot and the related original oil samples.



The results indicated that water type may influence PAH formation and that bunker fuels (IFO180) may produce higher PAH concentrations, particularly for in situ burns on seawater.

## 5.2 Cone calorimeter

### 5.2.1 Elemental carbon and organic carbon measurements

Figure 5.6 shows the mass loss, TC, EC and OC results for the crude oil and IFO180 as a function of the incident heat (0, 13 and 38 kW/m$^2$) and for the tests with a filter pump flow of 0.27 L/min. Also, incident heat of 64 kW/m$^2$ was tested; however, this resulted in quite violent burns that had to be extinguished to avoid damage to the apparatus. With increasing incident heat, the mass losses and TC concentrations also increase (Figure 5.6). Higher TC concentrations were measured for IFO180 than for the crude oil. This is in line with the findings from the small-scale burning cell experiments. However, it should be noted that the specific TC concentrations between the small-scale burn cell experiment and the cone experiments cannot be compared due to the non-isokinetic sampling conditions. It is possible to compare the relative trends in the development, though.

The correlation between mass loss and TC shows that TC increases with higher mass losses (Figure 5.7). This finding contrasts the measurements made using the small-scale laboratory burning cell, which highlights the difficulties in reproducing burn tests as both physical and chemical parameters (oil film thickness, oil type, oxygen supply, pool diameter, water bath etc.) will affect the actual burning process/fire dynamics and hence the derived results. It is hypothesised that a higher external incident heat flux to the oil will enhance the absolute amount of oil combusted (mass loss increase) and hence also enhance soot production (TC increase). However, a more efficient burn could also be expected to be less starved of atmospheric oxygen, leading to lower soot production – as seen with the small-scale burning cell experiments. Our results do not allow us to determine where these threshold limits occur. This also highlights the complications in reproducing full-scale offshore burning events in the laboratory. Correlating the total heat released (MJ/m$^2$ i.e. rate of heat generated by the fire) from the fire with TC and incident heat demonstrates that the combustion efficiency of the two oil types differs (Figure 5.8). With increasing incident heat, the total heat released from the crude oil burns declines as does TC. For IFO180, the trend is opposite. IFO180 is a residual fuel with a high content of complex hydrocarbon compounds, typically with a high flame point. The controlled conditions in the cone with increasing incident heat might improve the conditions for burning these heavy end compounds.

**Figure 5.6.** Mass loss, organic carbon (OC), elemental carbon (EC) and total carbon content (TC) as a function of incident heat in cone calorimeter tests. All OC/EC/TC values are normalised to the unit “microgram C per cm$^2$ filter per gram oil burned”. Note that the y-axis varies.

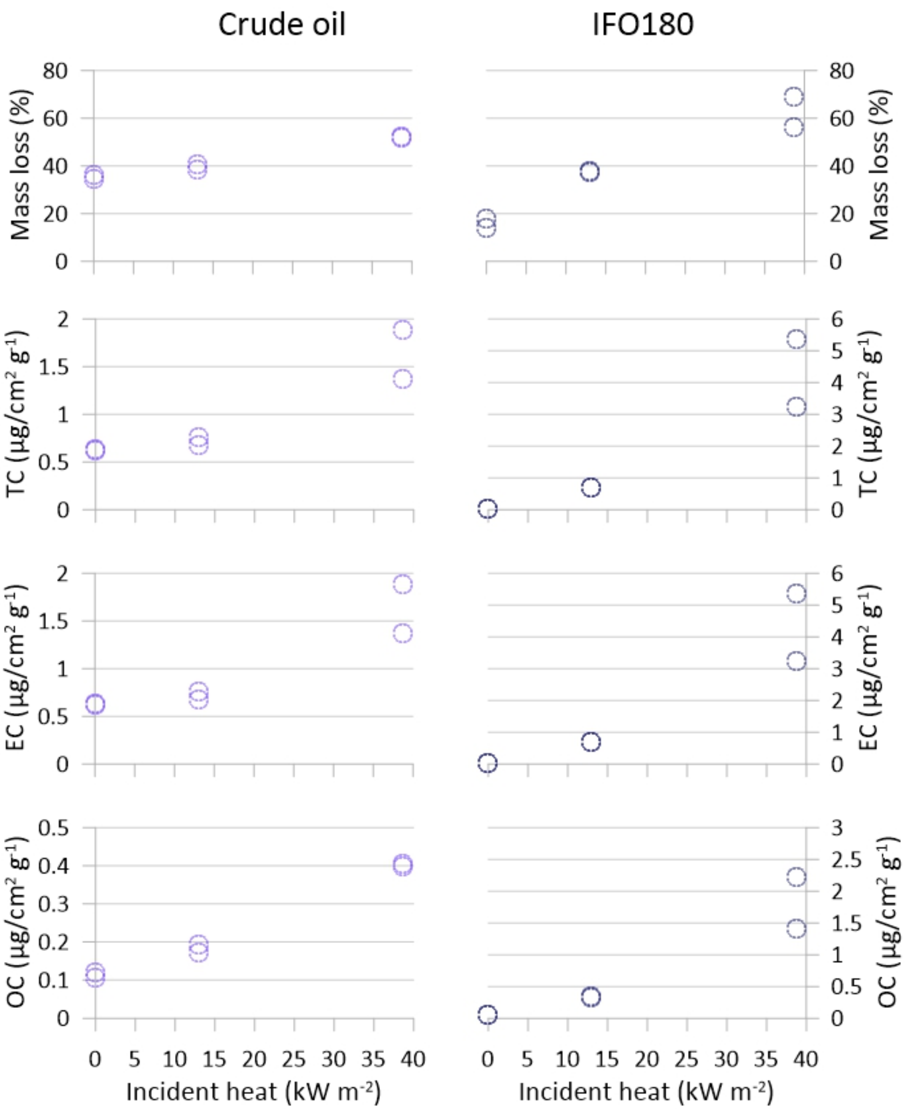


**Figure 5.7.** Carbon deposition (TC) vs mass loss. All OC/EC/TC values are normalised to the unit “microgram C per cm$^2$ filter per gram oil burned”. + = IFO180 experiments and ● = crude oil experiments.

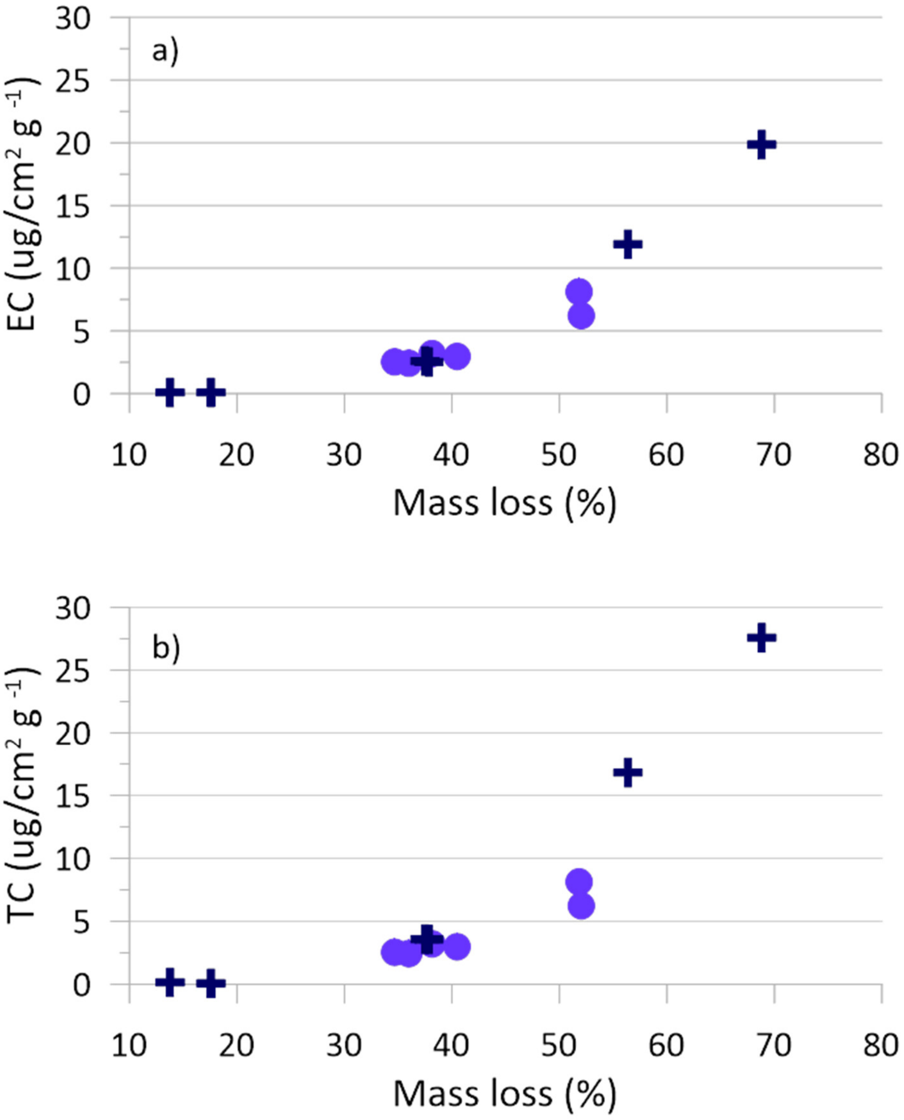
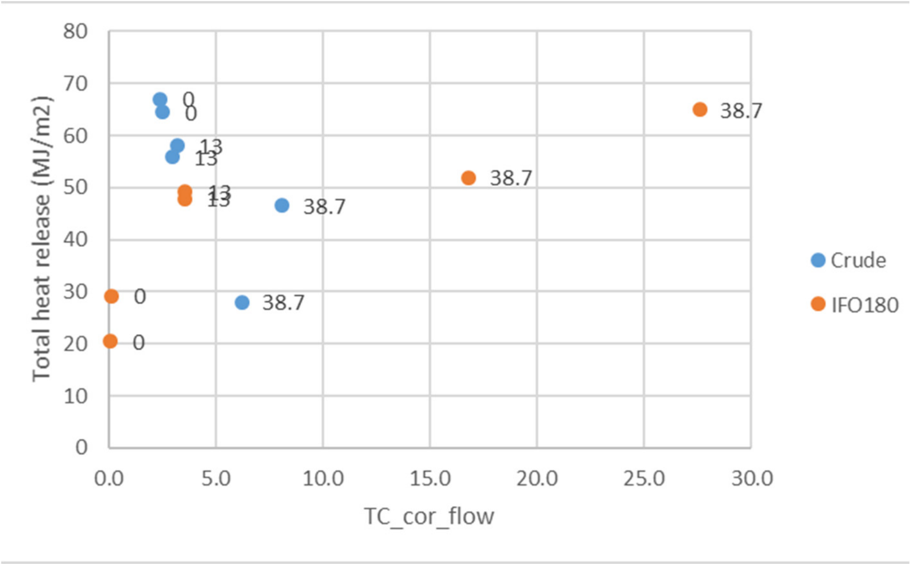


**Figure 5.8.** Carbon deposition (TC) vs total heat released. All TC values are normalised to the unit “microgram C per cm$^2$ filter per gram oil burned”. Incident heat (kW/m$^2$) is given for each point.



The samplings in the cone calorimeter were performed under iso-kinetic sampling conditions. It is therefore possible to extrapolate the total amount of soot produced during the burns (Figure 5.9). Soot makes up for between 0.1 and 38% of the burned oil depending on and increasing with the external heat input. Higher incident heat results in higher generation of TC. Compared to literature values for IFO180 (incident heat 25 kW / m$^2$, BC calculated as the amount of soot on the filter sample and the amount of oil burned), our results are in line with previous findings (Faksness et al. 2018). In Section 3, different values of soot are reported from various studies, ranging from approximately 1 to 20%. These variations in the amount of soot again highlight the large impact of the dependency of oil type and actual burning conditions on the results.

In the Strategic Environmental Impact Assessment for the Greenland Sea (Boertmann et al. 2021), oil spill simulations were made, including examples of instantaneous surface oil spills of 15,000 tons oil. If all that oil was burned in response to the oil spill, between 15 and 5,700 tons of soot would be generated based on the cone calorimeter results of our experiments. For comparison, residential burns in Denmark in 2015 produced 2,000 tons of black carbon (Press-Kristensen 2019). Preliminary results show that the background black carbon mass concentrations at St. North in Northeast Greenland were 20–40 ng / m$^3$ in March 2018, which is only about 1 / 20 of the levels observed in East Asia (Villum Research Station Nord 2018). Soot particle deposits on ice from a burn operation may reduce the reflective effect of the ice and thus lead to warming and melting of the ice (reduced albedo effect). Previous modelling of the plume trajectory has shown that the concentrations of particulate matter would be reduced to acceptable concentrations at ground level > 5-10 km downwind of the fire (McGrattan et al. 1993; McGrattan et al. 1997) relative to human / animal health issues. However, in remote Arctic locations, the detrimental health effects from the smoke plume should be negligible, but the environmental effects, including increased melting of the ice, from nearby precipitating soot and particulate matter are not known but may be of concern.

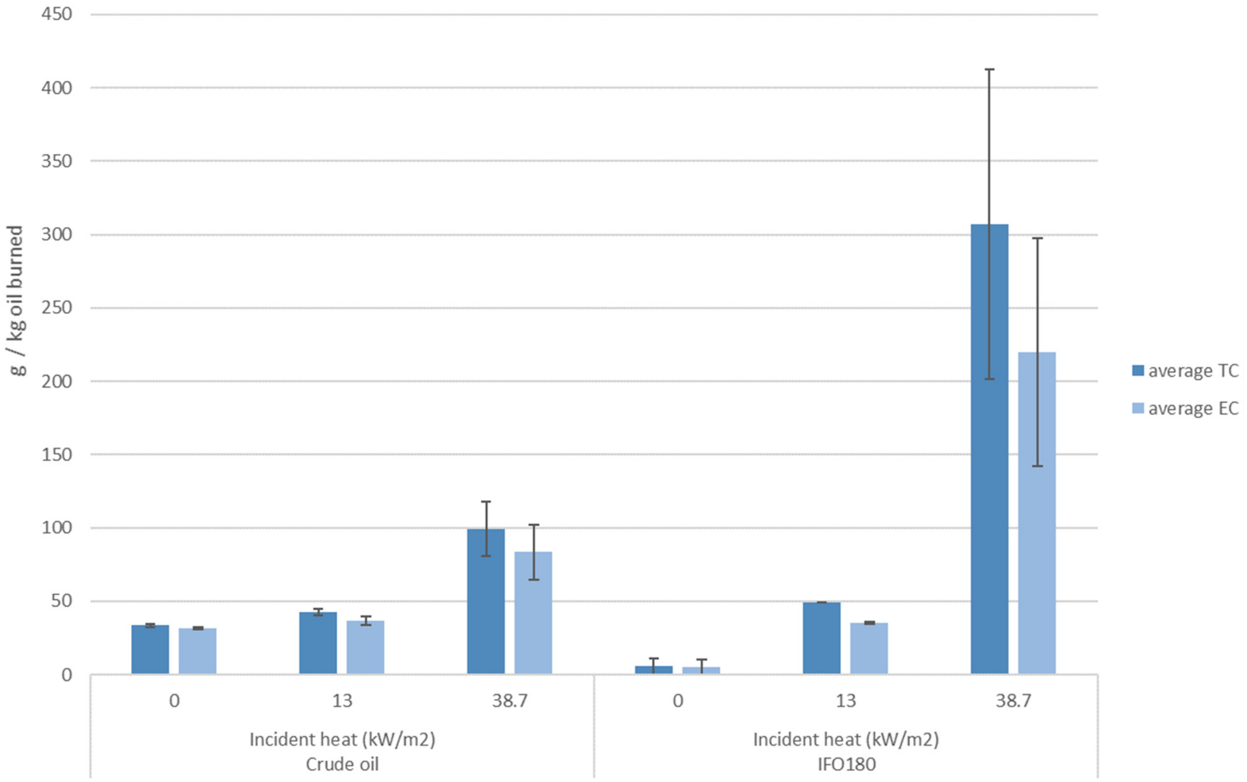


**Figure 5.9.** Total carbon (TC) and elemental carbon (EC) produced during the burning of crude oil and IFO180 with different incident heat values (kW/m$^2$). Average values (n=2; for 0 kW/m$^2$ experiments n=4).

# 6 Conclusion

The overall purpose of this work was to strengthen and improve the knowledge about the potential environmental implications following in situ burning of surface oils spills. The primary focus was to improve the understanding of air emissions and exposure of the smoke formation to the pristine Arctic environment. Formation of pyrogenic compounds and contaminants may be a direct result of in situ burning of oil spills. In small-scale laboratory experiments, the development of soot, PAHs and dioxins following burning of a simulated oil spill was measured.

Two oil types were included in small-scale burn experiments, a crude oil and a heavy fuel oil (IFO180). The oil was burned on the surface of either seawater or fresh water. The mass losses ranged between 38 and 67%, and the calculated regression rates were within the range reported for similar experiments. However, the burning of the IFO180 resulted in a violent boil-over phase at the end of the burn, implying less uniform results, and had lower flame temperatures compared to the crude oil experiments.

Dioxin concentrations were below the analytical detection limit value in all samples. As dioxin production is reported in the literature, it is still an open question, what is the driving factor (oil type, combustion effectiveness, water type etc.) for whether dioxins are produced and / or emitted during an in situ oil spill burn operation. We recommend that this topic is studied further as dioxin generated from an in situ burning operation could be a local source in the Arctic.

The soot generation from the small-scale burn experiments is reported as total carbon (TC) (sum of elemental carbon (EC) and organic carbon (OC)). For the burns on fresh water, there was a linear relationship between TC content and mass loss, where high mass loss resulted in low TC formation. For burns on seawater, the trend was less clear, particularly for the IFO180 experiments. Overall, IFO180 on seawater produced the highest amount of TC and thereby had the highest potential deposition on the surrounding environment. In the IFO180 on seawater test, these experiments also had the highest total concentrations of PAHs. This evidences that IFO180 burns generated more soot and contained more PAHs than the tested heavy fuel.

For both oil types, there was a shift in the PAH composition from a 2- and 3-ring PAH dominated oil to 4-6 ring PAH dominated soot. These 4-6-ring PAHs are more persistent in the environment than low ring-numbered PAHs.

The controlled experiments in the cone calorimeter showed that with increasing incident heat, the soot concentration also increased. Note that a higher soot concentration was measured for IFO180 than for the crude oil. This result is in line with the findings from the small-scale burning cell experiments.

The correlation between mass loss and TC demonstrated that TC increased with higher mass losses (this was opposite to the small-scale laboratory burning cell results). It may be hypothesised that a higher external incident heat flux to the oil will increase the absolute amount of oil combusted (mass loss increase) and hence also increase soot production (TC increase). However, a more efficient burn could also be expected to be less starved of oxygen and therefore produce less soot – as demonstrated in burning cell experiments. Our results did not allow us to determine threshold limits, which also highlights the complications involved in reproducing full-scale offshore burning events in the laboratory.

Based on our findings, focus on the development of in situ burning methodologies should be on increasing the mass loss by improving the combustion conditions. The oil type is also of importance, and heavy fuel oils should be avoided as they produce more soot than other oil types. This is in line with the newest IMO regulation introducing a complete HFO ban in the Arctic as from 2029.

# 7 Acknowledgement

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# AIR EMISSIONS FROM SMALL-SCALE BURNING TESTS OF CRUDE AND HEAVY FUEL OIL

Combatting oil spills in high Arctic marine waters may be extremely challenging. In situ burning of oil spills is recognised as an effective removal method for oil spills in the sea. The aim of this project is to increase the knowledge and understanding of potential environmental implications related to combatting oil spills in ice-infested waters by in situ burning. The project included measurements of air emissions from burns of oil in a small-scale burning cell and a cone calorimeter.

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