Atmospheric
Chemistry
and Physics
Discussions

1 **Emissions of intermediate-volatility and semi-volatile organic compounds from**

2 **domestic fuels used in Delhi, India**

3 Gareth J. Stewart[1], Beth S. Nelson[1], W. Joe F. Acton[2,a], Adam R. Vaughan[1], Naomi J. Farren[1],

4 James R. Hopkins[1,3], Martyn W. Ward[1], Stefan J. Swift[1], Rahul Arya[4], Arnab Mondal[4], Ritu

5 Jangirh[4], Sakshi Ahlawat[4], Lokesh Yadav[4], Sudhir K. Sharma[4], Siti S. M. Yunus[5], C. Nicholas

6 Hewitt[2], Eiko Nemitz[6], Neil Mullinger[6], Ranu Gadi[7], Lokesh. K. Sahu[8], Nidhi Tripathi[8],

7 Andrew R. Rickard[1,3], James D Lee[1,3], Tuhin K. Mandal[4] and Jacqueline F. Hamilton[1].

8 [1] Wolfson Atmospheric Chemistry Laboratories, Department of Chemistry, University of York, York, YO10 5DD, UK

9 [2] Lancaster Environment Centre, Lancaster University, Lancaster LA1 4YQ, UK

10 [3] National Centre for Atmospheric Science, University of York, York, YO10 5DD, UK

11 [4] CSIR-National Physical Laboratory, Dr. K.S. Krishnan Marg, New Delhi, Delhi 110012, India

12 [5] School of Water, Environment and Energy, Cranfield University, Cranfield, MK43 0AL, UK

13 [6] UK Centre for Ecology and Hydrology, Bush Estate, Penicuik, EH26 0QB, UK

14 [7] Indira Gandhi Delhi Technical University for Women, Kashmiri Gate, New Delhi, Delhi 110006, India

15 [8] Physical Research Laboratory (PRL), Ahmedabad 380009, India

16 [a] Now at: School of Geography, Earth and Environmental Sciences, University of Birmingham, B15 2TT, Birmingham, UK

17 **Abstract**

18 Biomass burning emits significant quantities of intermediate-volatility and semi-volatile

19 volatile organic compounds (I/SVOCs) in a complex mixture, probably containing many

20 thousands of chemical species. These components are significantly more toxic and have poorly

21 understood chemistry compared to volatile organic compounds routinely analysed in ambient

22 air, however quantification of I/SVOCs presents a difficult analytical challenge.

23 The gases and particles emitted during the test combustion of a range of domestic solid fuels

24 collected from across New Delhi were sampled and analysed. Organic aerosol was collected

25 onto Teflon (PTFE) filters and residual low-volatility gases were adsorbed to the surface of

26 solid-phase extraction (SPE) disks. A new method relying on accelerated solvent extraction

27 (ASE) coupled to comprehensive two-dimensional gas chromatography with time-of-flight

28 mass spectrometry (GC×GC-ToF-MS) was developed. This highly sensitive and powerful

29 analytical technique enabled over 3000 peaks from I/SVOC species with unique mass spectra

30 to be detected. 15-100 % of gas-phase emissions and 7-100 % of particle-phase emissions were

31 characterised. The method was analysed for suitability to make quantitative measurements of

32 I/SVOCs using SPE disks. Analysis of SPE disks indicated phenolic and furanic compounds

33 were important to gas-phase I/SVOC emissions and levoglucosan to the aerosol phase. Gas-

34 and particle-phase emission factors for 21 polycyclic aromatic hydrocarbons (PAHs) were

35 derived, including 16 compounds listed by the US EPA as priority pollutants. Gas-phase

36 emissions were dominated by smaller PAHs. New emission factors were measured (mg kg^{-1})

37 for PAHs from combustion of cow dung cake (615), municipal solid waste (1022), crop residue

38 (747), sawdust (1236), fuel wood (247), charcoal (151) and liquified petroleum gas (56).

39 The results of this study indicate that cow dung cake and municipal solid waste burning are

40 likely to be significant PAH sources and further study is required to quantify their impact,

41 alongside emissions from fuel wood burning.

42 **Introduction**

43 Biomass burning is one of the most important global sources of trace gases and particles to the

44 atmosphere (Simoneit, 2002; Chen et al., 2017; Andreae, 2019). Emissions of volatile organic

45 compounds (VOCs) and particulate matter (PM) are of interest due to their detrimental impact

46 on air quality. VOCs react to form ozone and secondary organic aerosol (SOA), and contribute

47 0-95 Tg yr^{-1} of SOA yearly (Shrivastava et al., 2017). Estimates of VOCs from burning often

48 do not include many intermediate-volatility and semi-volatile organic compounds (I/SVOCs).

49 Wildfires emit significant quantities of organic matter over regions such as the USA, the

50 Mediterranean, South East Asia and Australia (Liu et al., 2017; Barboni et al., 2010; Kiely et

51 al., 2019; Guérette et al., 2018) and residential combustion leads to substantial organic

52 emissions in the developing world (Streets et al., 2003).

53 I/SVOCs are an important class of air pollutant due to their contribution to aerosol formation

54 (Bruns et al., 2016; Lu et al., 2018). IVOCs have an effective saturation concentration of 300-

55 3,000,000 μg m^{-3} and are predominantly in the vapour phase. Once oxidised their lower

56 volatility products can partition into the aerosol phase (Donahue et al., 2006). SVOCs have

57 effective saturation concentrations of 0.3-300 μg m^{-3} (Donahue et al., 2012) and can partition

58 between the gas and particle phases. Many studies have focused on I/SVOCs emitted from a

59 range of sources due to their impact on aerosol formation (Robinson et al., 2007; Zhao et al.,

60 2014; Zhao et al., 2015; Cross et al., 2015; Pereira et al., 2018). I/SVOCs have been shown to

61 contribute significantly to emissions from biomass burning (Stockwell et al., 2015; Koss et al.,

62 2018). Global I/SVOC emissions to the atmosphere from biomass burning were estimated to

63 be ~ 54 Tg yr^{-1} from 2005-2008 (Hodzic et al., 2016) with I/SVOCs contributing in the range

64 8-15.5 Tg yr^{-1} to SOA (Cubison et al., 2011; Hodzic et al., 2016). SOA formation from

65 combustion of beech fuel wood was shown to be dominated by 22 compounds, with phenol,

66   naphthalene and benzene contributing up to 80 % of the observed SOA (Bruns et al., 2016).

67   However, the effect of atmospheric of aging on I/SVOCs still remains poorly understood (Liu

68   et al., 2017; Decker et al., 2019; Sengupta et al., 2020).

69   Residential combustion, agricultural crop residue burning, and open municipal solid waste

70   burning in the developing world are large, poorly characterised pollution sources with the

71   potential to have a significant impact on local and regional air quality, impacting human health

72   (Venkataraman et al., 2005; Jain et al., 2014; Wiedinmyer et al., 2014). Hazardous indoor air

73   pollution from combustion of solid fuels has been shown to be the most important factor from

74   a range of 67 environmental and lifestyle risk factors causing disease in South Asia (Lim et al.,

75   2012). Recent studies focussed on source apportionment of ambient VOC concentrations in

76   Delhi have shown ground-level concentrations to be predominantly traffic related, with smaller

77   contributions from solid fuel combustion (Stewart et al., 2020; Wang et al., 2020). Despite this,

78   nearly 76 % of rural Indian households are dependent on solid biomass for their cooking needs

79   (Gordon et al., 2018) with biofuels such as fuel wood, cow dung cake and crop residue being

80   used. Combustion often takes place indoors without efficient emission controls which

81   significantly increases the mean household concentration of pollutants, particularly particulate

82   matter with a diameter less than 2.5 μm ($PM_{2.5}$). Studies have shown mean 24 h concentrations

83   of $PM_{2.5}$ in kitchens to be in excess of 500 μg m^{-3} (Balakrishnan et al., 2013), with 4-8 times

84   ambient concentration enhancement of polycyclic aromatic hydrocarbons (PAHs) close to the

85   stove during cooking (Bhargava et al., 2004). This is significantly larger than the 40 μg m^{-3}

86   Indian National Air Quality Standard. For comparison, the mean population weighted $PM_{2.5}$

87   level in Delhi, Chennai, Hyderabad and Mumbai from 2015-2018 was 72 μg m^{-3} and the global

88   mean 20 μg m^{-3} (Chen et al., 2020), with various sources also leading to elevated levels of

89   PAHs in cities like Delhi (Elzein et al., 2020). The health effects from this are significant, with

90   premature deaths in India from exposure to ambient and household air pollution estimated to

91   be over 2 million (Lallukka et al., 2017).

92   Few detailed studies have been conducted examining the composition of I/SVOC emissions

93   from sources relevant to South Asia. One study examined gas- and particle-phase emissions

94   from coconut leaves, rice straw, cow dung cake, biomass briquettes and jackfruit branches in

95   Bangladesh with samples analysed by ion chromatography (IC), organic/elemental carbon

96   (OC/EC) and gas-chromatography coupled to mass spectrometry (GC-MS) to produce

97   emission factors and examine molecular markers (Sheesley et al., 2003). Another study

98   examined emissions of $PM_{2.5}$, OC/EC, metals and organics from motorcycles, diesel- and

99   gasoline-generators, agricultural pumps, municipal solid waste burning, cooking fires using

100   fuel wood and cow dung cake, crop residue burning and brick kilns in Nepal (Jayarathne et al.,

101   2018). Lack of knowledge regarding major pollution sources hinders our ability to predict air

102   quality, but also the development of effective mitigation strategies for air pollution which leads

103   to health impacts ranging from respiratory illness to premature death (Brunekreef and Holgate,

104   2002). This results in many people living with high levels of air pollution (Lelieveld et al.,

105   2015; Cohen et al., 2005) and 13 Indian cities ranking amongst the top 20 cities in the world

106   with the highest levels of ambient $PM_{2.5}$ pollution, based on available data (Gordon et al.,

107   2018).

108   Early biomass burning studies used filters to target aerosol and sorbent tubes or polyurethane

109   styrene-divinylbenzene (PUF/XAD/PUF) cartridges to sample gaseous species followed by

110   solvent extraction and analysis by GC-MS (McDonald et al., 2000; Schauer et al., 2001; Hays

111   et al., 2002; Mazzoleni et al., 2007; Dhammapala et al., 2007; Singh et al., 2013; Jordan and

112   Seen, 2005; Pettersson et al., 2011; Sheesley et al., 2003). Detailed studies have focussed on

113   quantifying the composition of the particulate matter from burning by extracting aerosol

114   samples, followed by analysis by GC-MS (Oros and Simoneit, 2001b, a; Fine et al., 2001; Oros

115   et al., 2006; Jayarathne et al., 2018). Many studies have been carried out to measure emission

116   factors of PAHs from burning, such as detailed measurements of up to 133 PAHs (Samburova

117   et al., 2016) and time-resolved PAH measurements (Eriksson et al., 2014). PAH emission

118   factors have been measured for coal (Chen et al., 2005; Lee et al., 2005; Geng et al., 2014) oil

119   (Rogge et al., 1997), fuel woods (McDonald et al., 2000; Simoneit, 2002; Hosseini et al., 2013;

120   Jimenez et al., 2017; Geng et al., 2014), peat (Iinuma et al., 2007), tyres (Iinuma et al., 2007),

121   domestic waste (Sidhu et al., 2005; Kakareka et al., 2005), cow dung cake (Gadi et al., 2012;

122   Singh et al., 2013; Tiwari et al., 2013), sawdust briquette (Kim Oanh et al., 2002) and crop

123   residue (Jenkins et al., 1996; Lu et al., 2009; Gadi et al., 2012; Singh et al., 2013; Wei et al.,

124   2014; Kim Oanh et al., 2015; Wiriya et al., 2016). Measurements of I/SVOCs in both gas- and

125   particle-phase samples using conventional GC-MS presents a difficult analytical challenge, due

126   to the exponential growth of potential isomers with carbon number which can result in a large

127   number of coeluting peaks (Goldstein and Galbally, 2007).

128   The high resolution of two-dimensional gas chromatography (GCxGC) has been demonstrated

129   as an ideal technique to overcome the issue of peak coelution in one-dimensional gas

130   chromatography and has been used to analyse complex ambient samples in the gas (Lewis et

131   al., 2000; Xu et al., 2003; Dunmore et al., 2015; Lyu et al., 2019a) and particle phases

132 (Hamilton et al., 2004; Lyu et al., 2019b; Lyu et al., 2019c). GCxGC has recently shown

133 hundreds of gaseous I/SVOCs released from biomass burning using adsorption-thermal

134 desorption cartridges or solid phase extraction (SPE) disks (Hatch et al., 2015; Hatch et al.,

135 2018). The particle phase has also been targeted by extracting samples from PTFE or quartz

136 filters (Hatch et al., 2018; Jen et al., 2019), with the latter study quantifying 149 organic

137 compounds which accounted for 4-37 % of the total mass of organic carbon. The process used

138 by Hatch et al. (2018) demonstrated high recoveries of non-polar species from PTFE filters,

139 with lower recoveries from SPE disks. This study highlighted the need for further evaluation

140 of samples collected onto PTFE filters and SPE disks, ideally improving the method to remove

141 undesirable steps such as trimethylsilyation derivatisation, the use of pyridine and centrifuging

142 which led to high evaporative losses. The need to develop improved sampling and

143 measurement techniques for I/SVOCs has been highlighted as these species often do not

144 transmit quantitatively through the inlet and tubing when measured using online gas-phase

145 techniques (Pagonis et al., 2017).

146 In this study we develop a more efficient extraction step for the SPE/PTFE technique, allowing

147 high recoveries of non-polar I/SVOCs collected from burning typical domestic fuels used in

148 Northern India. The technique is used to identify many I/SVOCs in burning samples, examined

149 for quantification of I/SVOCs from burning and used to develop emission factors for selected

150 PAHs.

151 **Sample collection and burning facility**

152 The state of New Delhi was gridded ($0.05^\circ \times 0.05^\circ$) and samples collected from across the state

153 (see Figure 1). Samples were stored in a manner akin to local practices prior to combustion, to

154 ensure that the moisture content of fuels were similar to those burnt across the state. A range

155 of solid biomass fuels were collected which included 17 fuel wood species, cow dung cake,

156 charcoal and sawdust (see Table 1). Three crop residue samples were collected and consisted

157 of dried stems from vegetable plants such as cabbage (*Brassica spp*) and aubergines (*Solanum*

158 *melongena*) as well as coconut husk (*Cocos nucifera*). Municipal solid waste samples were

159 collected from Bhalaswa, Ghazipur and Okhla landfill sites. A low-cost LPG stove was also

160 purchased to allow direct comparison to other combustion sources.

161 Samples were burnt at the CSIR-National Physical Laboratory (NPL) New Delhi under

162 controlled conditions using a combustion dilution chamber that has been well described

163 previously (Venkataraman et al., 2002; Saud et al., 2011; Saud et al., 2012; Singh et al., 2013).

164   In summary, 200 g of dry fuel was rapidly heated to spontaneous ignition with emissions driven

165   into a hood and up a flue by convection to allow enough dilution, cooling and residence time

166   to achieve the quenching of typical indoor environments. This process was designed to

167   replicate the immediate condensational processes that occur in smoke particles approximately

168   5-20 mins after emission, yet prior to photochemistry which may change composition (Akagi

169   et al., 2011). A low volume sampler (Vayubodhan Pvt.Ltd) was used to collect particulates and

170   low volatility gases passing from the top of the flue through a chamber with a flow rate of 46.7

171   L min^{-1}. As detailed in Table 1, 30 samples from a range of fuel types were burnt, and 8 blank

172   samples were collected (see the Supplementary Information S1 for an example burn and filter

173   samples collected from different sources). Prior to sample collection, SPE disks (Resprep, C_{18},

174   47 mm) were prewashed with 2 x 5 mL acetone (Fisher Scientific analytical reagent grade),

175   and 1 x 5 mL methanol (Sigma-Aldrich HPLC grade), then packed in foil and sealed in airtight

176   bags. Samples were collected onto a PTFE filter (Cole-Parmer, 47 mm, 1.2 μm pore size)

177   placed on top of an SPE disk in a filter holder (Cole-Parmer, 47 mm, PFA) for 30 mins at a

178   flow rate of 6 L min^{-1}, maintained by a mass flow controller (Alicat 0-20 SLM) connected to a

179   pump. Samples were removed from the filter holder immediately after the experiment and

180   wrapped in foil, placed inside an airtight bag and stored at – 20 °C. Samples were then

181   transported to the UK for analysis using an insulated container containing dry ice via. air freight

182   and stored at – 20 °C for around 2 months prior to analysis.
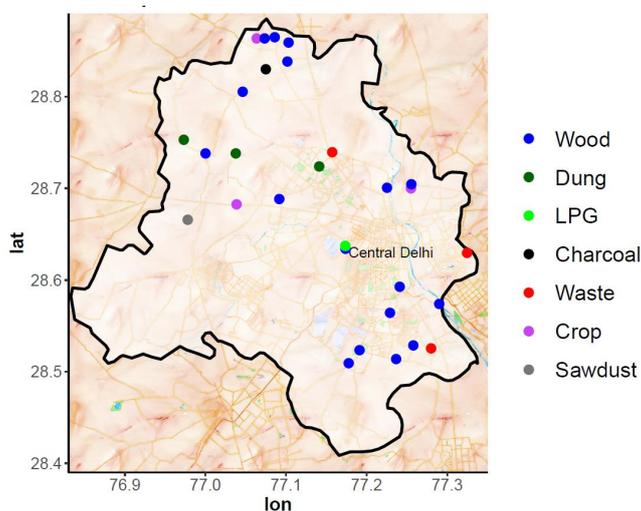
183

184   Figure 1. Locations across New Delhi used for the local surveys into fuel use and sample

185   collection. Map produced using tiles from Stamen maps.

186 Table 1. Types of sample collected where n = number of samples burned, SPE and PTFE = number of
187 blank corrected peaks detected on SPE disks and PTFE filters, respectively.

Fuel woods	n	SPE	PTFE	Other	n	SPE	PTFE
Plywood	1	149	530	Cow dung cake	3	1295	1604
Azadirachta indica	1	562	880	*Cocos nucifera*	1	614	1182
Morus spp	1	811	1108	Charcoal	1	453	211
Shorea spp	1	283	326	Sawdust	1	1113	1417
Ficus religiosa	1	504	652	Waste	3	980	1181
Syzygium spp	1	680	529	LPG	1	-	0
Ficus spp	1	277	247	Blank	8	-	-
Vachellia spp	1	702	753	Cow dung cake mix	1	932	1200
Dalbergia sissoo	1	483	561	*Brassica spp*	1	656	463
Ricinus spp	1	424	125	*Solanum melongena*	1	280	551
Holoptelea spp	1	276	263				
Saraca indica	1	517	445				
Pithecellobium spp	1	527	159				
Eucalyptus spp	1	211	77				
Melia azedarach	1	434	166				
Prosopis spp	1	237	113				
Mangifera indica	1	360	546				

188

Sample extraction

189

190 SPE disks and PTFE filters were spiked with an internal standard (50 μL at 20 μg mL^{-1})
191 containing 6 deuterated PAHs (1,4-Dichlorobenzene-d$_4$, naphthalene-d$_8$, acenaphthene-d$_{10}$,
192 phenanthrene-d$_{10}$, chrysene-d$_{12}$, perylene-d$_{12}$; EPA 8270 Semivolatile Internal Standard Mix,
193 2000 μg mL^{-1} in DCM) to result in a final internal standard concentration of 1 μg mL^{-1} in
194 solution. The solvent from the internal standard was allowed to evaporate and then SPE disks
195 and PTFE filters were cut and extracted into EtOAc using accelerated solvent extraction (ASE
196 350, Dionex, ThermoFisher Scientific). Extractions were performed at 80 ^{o}C and 1500 psi for
197 three 5 min cycles. After each cycle, the cell was purged for 60 seconds into a sample collection
198 vial. Samples were then reduced from 15 mL to 0.90 mL over a low flow of N$_2$ in an ice bath
199 over a period of 6-8 hours (Farren et al., 2015). Samples were then pipetted (glass Pasteur) to
200 sample vials (Sigma-Aldrich, amber glass, 1.5 mL), with ASE vials rinsed with 2×50 μL
201 washes of EtOAC, then added to the sample vial and sealed (Agilent 12 mm cap,
202 PTFE/silicone/PTFE). The mass of the sample vial and cap for each sample was measured
203 before and after to determine the exact volume of solvent in each sample. Extracts were frozen
204 prior to analysis to reduce evaporative losses.

Methods for organic composition analysis

205

206 GCxGC-ToF-MS: PTFE samples were analysed using GCxGC-ToF-MS (Leco Pegasus BT
207 4D) using a splitless injection (1 μL injection, 4mm taper focus liner, SHG 560302). The

208 primary dimension column was a RXI-5SilMS (Restek, 30 m × 0.25 μm × 0.25 mm) connected

209 to a second column of RXI-17SilMS (Restek, 0.25 μm × 0.25 mm, 0.17 m primary GC oven,

210 0.1 m modulator, 1.42 m secondary oven, 0.31 m transfer line) with a He flow of 1.4 mL min^{-1}

211 . The primary oven was held at 40 °C for 1 min then ramped at 3 °C min^{-1} to 322 °C where it

212 was held for 3 min. The secondary oven was held at 62 °C for 1 min then ramped at 3.2 °C to

213 190 °C after which it was ramped at 3.6 °C min^{-1} to 325 °C and held for 19.5 mins. The inlet

214 was held at 280 °C and the transfer line at 340 °C. A 5 s cryogenic modulation was used with

215 a 1.5 s hot pulse and 1 s cool time between stages. Using two separate wash vials, the syringe

216 (10 μL Gerstel) was cleaned prior to injection with two cycles of 3 × 5 μL washes in EtOAc

217 and rinsed post injection with two cycles of 2 × 5 μL washes in EtOAc. Samples with high

218 concentrations of levoglucosan were reanalysed using a faster method, injected split (75:1 and

219 125:1) with the primary oven held at 40 °C for 1 min, then ramped at 10 °C min^{-1} to 220 °C.

220 The secondary oven was held at 62 °C for 1 min and then ramped at 10 °C min^{-1} to 245 °C.

221 SPE samples were injected split (10:1) and analysed with a shorter analysis time with the

222 primary oven held at 40 °C for 1 min then ramped at 3 °C min^{-1} to 202 °C where it was held for

223 4 seconds. The secondary oven was held at 62 °C for 1 min then ramped at 3.2 °C min^{-1} to 235

224 °C. A 75:1 split injection was used for quantitation of concentrations outside of the detector

225 response range for furans, phenolics, benzaldehydes, naphthalenes and benzonitrile. Peaks

226 were assigned through comparison of retention times with known standards and comparison

227 with the National Institute of Standards and Technology (NIST) mass spectral library. Peaks

228 with no genuine standard available were tentatively identified if the NIST library hit was >

229 800. The uncertainty in this approach has been shown to be low, with the probability of

230 incorrect identification being around 30 % for hits between 800-900 and 14 % for matches

231 above 900 (Worton et al., 2017). Integration was carried out within the ChromaTOF 5.0

232 software package (Leko, 2019). Calibration was performed using a 6-point calibration using

233 either a linear or second-order polynomial fit covering the ranges 0.1-2.5 μg ml^{-1} (splitless),

234 0.5-15 μg ml^{-1} (10:1 split), 15-400 μg ml^{-1} (75:1 split) and 400-800 μg ml^{-1} (125:1 split). Eight

235 blank measurements were made at the beginning and end of the day by passing air from the

236 chamber (6 L min^{-1} for 30 mins) through the filter holder containing PTFE filters and SPE disks

237 (see the Supplementary Information S2 for examples of blank chromatograms). Blank

238 corrections were applied by calculating the average blank value for each compound using blank

239 samples collected using the same sample collection parameters as real samples before and after

240 the relevant burning experiments.

241 PTR-ToF-MS: Online measurements of naphthalene, methylnaphthalenes and
242 dimethylnaphthalenes were made using a proton transfer reaction-time of flight-mass
243 spectrometer PTR-ToF-MS (PTR 8000; Ionicon Analytik, Innsbruck) and assigned as masses
244 129.058, 143.08 and 157.097, respectively. Additional details of the PTR-ToF-MS from
245 Physical Research Laboratory (PRL), Ahmedabad used in this study are given in previous
246 papers (Sahu and Saxena, 2015; Sahu et al., 2016). A ¼ inch OD PFA sample line ran from the
247 top of the flue to the instrument which was housed in an air-conditioned laboratory with a
248 sample flow rate of 4.3 L min^{-1}. The sample air was diluted either 5 or 6.25 times into zero air,
249 generated by passing ambient air (1 L min^{-1}) through a heated platinum filament at 550 ºC,
250 before entering the instrument with an inlet flow of 250 ml min^{-1}. The instrument was operated
251 with a reduced electric field strength (E/N, where N is the buffer gas density and E is the electric
252 field strength) of 120 Td. The drift tube temperature was 60 °C with a pressure of 2.3 mbar and
253 560 V applied across it.

254 Calibrations of the PTR-ToF-MS were performed twice a week using a gas calibration unit
255 (Ionicon Analytik, Innsbruck). The calibration gas (Apel-Riemer Enironmental Inc., Miami)
256 contained 18 compounds: methanol, acetonitrile, acetaldehyde, acetone, dimethyl sulphide,
257 isoprene, methacrolein, methyl vinyl ketone, 2-butanol, benzene, toluene, 2-hexanone, m-
258 xylene, heptanal, α-pinene, 3-octanone and 3-octanol at 1000 ppb (±5 %) and β-caryophyllene
259 at 500 ppb (±5 %). This standard was dynamically diluted into zero air to provide a 6-point
260 calibration. The normalised sensitivity (ncps/ppbv) was then determined for all masses using a
261 transmission curve derived from these standard compounds (Taipale et al., 2008).

262 Mass calibration and peak fitting of the PTR-ToF-MS data were performed using PTRwid
263 software (Holzinger, 2015). Count rates (cps) of each mass spectral peak were normalised to
264 the primary ion (H_3O^+) and water cluster ($H_3O.H_2O)^+$ peaks and mixing ratios were then
265 determined for each mass using the normalised sensitivity (ncps). Where compounds known to
266 fragment in the PTR-ToF-MS were identified, the mixing ratio of these species was calculated
267 by summing parent ion and fragment ion mixing ratios. Before each burning study ambient air
268 was sampled to provide a background for the measurement.

269 **Quantification of recovery and breakthrough**
270 Standards were used for 136 species (see Figure 2) including two commercially available
271 standard mixes containing 33 alkanes (C_7-C_{40} saturated alkane standard, certified 1000 µg m^{-1}
272 in hexane, Sigma Aldrich 49452-U) and 64 semi volatiles (EPA CLP Semivolatile Calibration

273   Mix, 1000 µg mL^{-1} in DCM:benzene 3:1, Sigma Aldrich 506508). Further standards were

274   produced in-house, by dissolving high quality standards (> 99 % purity), for a range of

275   additional species also found in samples including nitrogen containing VOCs (NVOCs),

276   furans, alkyl-substituted monoaromatics, oxygenated aromatics, ketones, aldehydes, methoxy

277   phenols, aromatic acids, PAHs and levoglucosan (see Table 2). Stock solutions of around 1000

278   µg mL^{-1} were prepared by dissolving 0.01 g into 10 mL EtOAc. Polar components, such as

279   levoglucosan, were dissolved into MeOH for stock solutions and those not soluble at room

280   temperature were heated and pipetted using hot pipette tips to make quantitative dilutions.

281   Six separate PTFE filters and SPE disks were spiked with the standard solution containing 136

282   compounds (50 µL at 20 µg mL^{-1}), extracted and analysed. Recovery levels were calculated by

283   comparing the signal to direct injection of the diluted standards to the GCxGC-ToF-MS. The

284   recoveries are shown in Table 2. SPE disks showed poor recoveries (S_{rec}) of *n*-nonane to *n*-

285   tridecane and C_2 substituted monoaromatics, likely due to volatilisation of these more-volatile

286   components. Poorer recoveries were also observed of nitroanilines and levoglucosan. Non-

287   polar species showed good recoveries, with high recoveries of C_{14}-C_{20} alkanes, furans, phenols,

288   chlorobenzenes and PAHs. PTFE filters demonstrated high recoveries (P_{rec}) of PAHs with more

289   than three rings in their structure (81.6-100 %). Recoveries were low, or zero, for volatile

290   components with boiling points < 200 °C, indicating no retention, which is consistent with the

291   method being well-suited to target the aerosol phase. The recoveries of non-polar species in

292   EtOAc from SPE disks were higher than those reported into MeOH (Hatch et al., 2018).
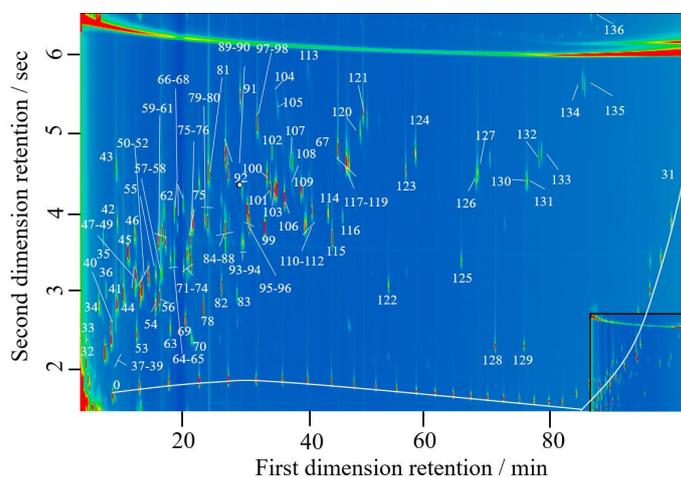
294   Figure 2. GCxGC-ToF-MS chromatogram of a mixed standard, numbered according to species listed
295   in Table 2.

296 Table 2. Species used in calibration where Comp No. refers to the peak number in Figure 2, Q_{ms} = split
297 method used for SPE quantitation, Q_{mp} = split method used for PTFE quantitation, S = splitless method,
298 S_{rec} = % recovery SPE, P_{rec} = % recovery PTFE, [a] = Sigma-Aldrich n-alkanes standard, [b] = Sigma-
299 Aldrich semivolatiles standard, [c] = Sigma-Aldrich deuterated internal standard, [d] = in-house solution
300 and - = not measured either due to being outside of SPE method range or due to volatilisation from
301 PTFE filters. Slight over-recoveries of > 100 % are reported as 100 % and accounted for in blank
302 subtractions.

303

Comp No.	Species	Q_{ms}	Q_{mp}	S_{rec}	P_{rec}	Comp No.	Species	Q_{ms}	Q_{mp}	S_{rec}	P_{rec}
	Alkane						**NVOC**				
0	n-Nonane [a]	10:1	S	60.0	-	32	Pyridine [d]	10:1	S	75.1	-
1	n-Decane [a]	10:1	S	77.6	19.5	33	n-Nitrosodimethylamine [b]	10:1	S	-	-
2	n-Undecane [a]	10:1	S	100	57.2	44	2,3-lutidine [d]	10:1	S	99.4	-
3	n-Dodecane [a]	10:1	S	85.7	22.0	46	Benzonitrile [d]	75:1	S	86.9	-
4	n-Tridecane [a]	10:1	S	91.4	75.0	57	n-Nitrosodipropylamine [b]	10:1	S	100	-
5	n-Tetradecane [a]	10:1	S	97.8	97.8	62	Nitrobenzene [b]	10:1	S	88.5	-
6	n-Pentadecane [a]	10:1	S	99.7	92.3	67	2-Nitrophenol [b]	10:1	S	100	-
7	n-Hexadecane [a]	10:1	S	100	100	68	Pyrrole 2-carbonitrile [d]	10:1	S	-	-
8	n-Heptadecane [a]	10:1	S	100	98.0	77	4-chloroananline [b]	10:1	S	7.78	-
9	n-Octadecane [a]	10:1	S	100	99.9	98	2-Nitroanaline [b]	10:1	S	100	-
10	n-Nonadecane [a]	10:1	S	100	98.9	102	2,6-dinitrotoluene [b]	10:1	S	99.9	-
11	n-Eicosane [a]	10:1	S	100	96.8	105	3-Nitroanaline [b]	10:1	S	34.2	-
12	n-Heneicosane [a]	10:1	S	-	100	107	2,4-Dinitrotoluene [b]	10:1	S	100	-
13-23	n-Docosane [a] – n-Dotriacontane [a]	10:1	S	-	100	108	4-Nitrophenol [b]	10:1	S	-	-
24	n-Tritriacontane	-	-	-	96.5	112	Azobenzene [b]	10:1		100	100
25	n-Tetratriacontane	-	-	-	78.9	113	p- Nitroaniline [b]	10:1	S	64.5	-
26	n-Pentatriacontane	-	-	-	58.3	121	Caffeine [d]	10:1	S	-	-
27	n-Hexatriacontane	-	-	-	49.9		**Aromatics**				
28	n-Heptatriacontane	-	-	-	35.4	37	Ethylbenzene [d]	10:1	S	44.6	-
29	n-Octatriacontane	-	-	-	32.1	38	m-Xylene [d]	10:1	S	34.5	-
30	n-Nonatriacontane	-	-	-	29.1	39	o-Xylene [d]	10:1	S	32.4	-
31	n-Tetracontane	-	-	-	27.9	40	Styrene [d]	10:1	S	58.4	-
	PAH					69	Pentylbenzene [d]	10:1	S	99.0	24.4
76	Naphthalene [b/c]	75:1	S	93.9	37.1	82	Pentamethylbenzene [d]	10:1	S	68.6	39.5
81	Quinoline [d]	10:1	S	28.6	-		**Halogenated**				
87	2-Methylnapthalene [b]	75:1	S	90.8	72.4	48	2-Chlorophenol [b]	10:1	S	100	-
89	Indole [d]	10:1	S	81.6	-	50	1,3-Dichlorobenzene [b]	10:1	S	85.5	-
90	Azulene [d]	10:1	S	38.5	-	51	1,4-Dichlorobenzene [b,c]	10:1	S	87.2	-
91	1(3H)-Isobenzofuranone [d]	10:1	S	100	-	52	1,2-Dichlorobenzene [b]	10:1	S	70.3	-
96	Biphenyl [d]	10:1	S	99.5	75.0	56	Hexachloroethane [b]	10:1	S	83.7	-
97	1,4-Naphthoquinone [d]	10:1	S	100	-	74	2,4-Dichlorophenol [b]	10:1	S	100	83.9
99	2,3-Dimethylnaphthalene [d]	10:1	S	100	-	75	1,2,4-trichlorobenzene [b]	10:1	S	85.6	-
100	Acenaphthylene [b]	10:1	S	98.5	84.1	78	Hexachlorobutadiene [b]	10:1	S	61.6	-
103	Acenapthene [b/c]	10:1	S	100	88.2	83	Hexachlorocyclopentadiene [b]	10:1	S	100	-
106	Dibenzofuran [b]	10:1	S	100	86.4	88	4-Chloro-3-methylphenol [b]		S	90.8	-
109	Fluorene [b]	10:1	S	100	86.0	93	2,4,6-Trichlorophenol [b]	10:1	S	95.8	-
117	9H-Fluoren-9-one [d]	10:1	S	100	100	94	2,4,5-Trichlorophenol [b]	10:1	S	100	-
118	Phenanthrene [b]	10:1	S	100	96.7	95	2-Chloronapthalene [b]	10:1	S	99.6	-
119	Anthracene [b]	10:1	S	98.6	95.9	110	4-Chlorophenylphenylether [b]	10:1	S	100	-
120	Carbazole [b]	10:1	S	100	85.2	114	4-Bromophenylphenylether [b]	10:1	S	100	-
123	Fluoranthene [b]	10:1	S	100	97.2	115	Hexachlorobenzene [b]	10:1	S	100	-
124	Pyrene [b]	10:1	S	-	100	116	Pentachlorophenol [b]	10:1	S	100	-
126	Benzo(a)anthracene [b]	-	S	-	100		**Furans**				

#	Compound	Ratio	S	V1	V2	#	Compound	Ratio	S	V1	V2
127	Chrysene [b/c]	-	S	-	100	34	Furfural [d]	75:1	S	84.3	-
130	Benzo(b)fluoranthene [b]	-	S	-	100	35	Maleic anhydride [d]	10:1	S	54.9	-
131	Benzo(k)fluoranthene [b]	-	S	-	100	36	α-Angelica lactone [d]	10:1	S	52.1	-
132	Benzo(a)pyrene [b]	-	S	-	89.5	43	2-5(H)-furanone [d]	75:1	S	100	-
133	Perylene-D12 [c]	-	S	-	92.4	**Phthalates**					
134	Indeno(1,2,3-CD)pyrene [b]	-	S	-	94.0	101	Dimethyl phthalate [b]	10:1	S	100	-
135	Dibenz(A,H)anthracene [b]	-	S	-	92.9	111	Diethyl phthalate [b]	10:1	S	100	-
136	Benzo(G,H,I)perylene [b]	-	S	-	96.6	122	Di-n-butyl-phthalate [b]	10:1	S	-	-
Oxygenated aromatics						125	Benzyl butyl phthalate [b]	-	S	-	92.0
41	Anisole [d]	10:1	S	20.4	-	128	Bis(2-ethylhexyl)phthalate [b]	-	S	-	97.4
42	p-Benzoquinone [d]	10:1	S	94.8	-	129	Di-n-octyl phthalate [b]	-	S	-	90.6
45	Benzaldehyde [d]	10:1	S	82.8	-	**Others**					
47	Phenol [b]	75:1	S	100	-	49	Bis(2-chloroethyl)ether [b]	10:1	S	84.5	-
55	o-Cresol [b]	10:1	S	100	-	53	2-Octanone [d]	10:1	S	97.0	-
58	p-Cresol [b]	75:1	S	100	-	54	Bis(2-chloro-1-methylethyl)ether [b]	10:1	S	100	-
59	3-Methylbenzaldehyde [d]	10:1	S	99.9	-	63	Nonanal [d]	10:1	S	100	52.3
60	2-Methylbenzaldehyde [d]	75:1	S	100	-	65	Isophorone [b]	10:1	S	96.4	-
61	2-Methoxyphenol [d]	75:1	S	100	-	70	1-nonanol [d]	10:1	S	98.6	-
64	2,6-Dimethylphenol [d]	75:1	S	100	100	72	Bis(2-chloroethoxy)methane [b]	10:1	S	100	-
66	2,3-dimethyl-2,5-cyclohexadiene-1,4-dione [d]	10:1	S	100	-	84	Pinane diol [d]	10:1	S	-	-
71	2,4-dimethylphenol [b]	10:1	S	89.5	-	104	Levoglucosan [d]	10:1	S	0	70.0
73	Benzoic acid [d]	10:1	S	-	-						
79	Mequinol [d]	10:1	S	60.4	-						
80	m-Guaiacol [d]	10:1	S	44.0	-						
85	Hydroquinone [d]	10:1	S	34.8	-						
86	Resorcinol [d]	10:1	S	76.0	-						
92	2,6-Dimethoxyphenol [d]	10:1	S	93.6	-						

304

305 To quantify the additional effect of breakthrough during sampling, tests were conducted for

306 SPE disks to examine the retention of components adsorbed to their surface when subject to an

307 air flow equivalent to the sample volume. SPE disks were spiked with the calibration mixture

308 containing 96 compounds of interest (50 μL at 20 μg mL^{-1}, $n = 4$) and subject to a purified air

309 flow of 6 L min^{-1} for 30 mins. The samples were extracted and analysed, and the signal

310 compared with 4×50 μL spikes directly into 0.95 mL EtOAc. Figure 3 shows the relative

311 enhancement of unpurged over purged samples. For more volatile components a value greater

312 than zero was observed (Figure 3), which indicated breakthrough of the most volatile

313 components and indicated good retention of components with a boiling point of around 225 °C

314 (see the Supplementary Information S3 for breakthrough tests). Concentrations measured for

315 n-alkanes on SPE disks were also compared with concurrent measurements made during

316 burning experiments using online thermal-desorption two-dimensional gas chromatography

317 coupled to a flame ionisation detector. The measured concentrations for n-alkanes from n-

318 nonane to n-dodecane were compared using both techniques, with measured concentrations

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319 similar for *n*-undecane/*n*-dodecane (bp = 216 ºC, see the Supplementary Information S4) but

320 not the smaller alkanes. This was interpreted to indicate little breakthrough for components less

321 volatile than *n*-dodecane. These findings are in line with the US EPA certified methods for

322 Resprep SPE disks (525.1, 506, 550.1, and 549.1), when used to quantitatively analyse drinking

323 water, which show their suitability for quantitative measurement of species with a molecular

324 weight of around naphthalene/acenaphthylene (bp = 218-280 ºC). These results indicate that

325 for more volatile species with boiling points below 250 ºC, SPE disks can only be used to make

326 qualitative measurements at these sample times and flow rates. Such qualitative information is

327 highly complementary to quantitative measurements using other, less specific, techniques, such

328 as PTR-ToF-MS, where it can assist in identification of the contributors to *m/z* ions.
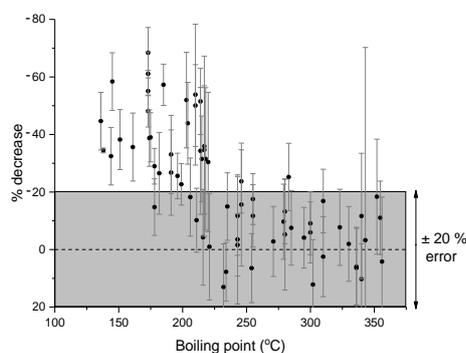
329

330 Figure 3. Relative reduction of purged over unpurged samples, presented as a percentage decrease of
331 purged to unpurged signal. The standard deviation of replicate measurements is indicated by error bars.

332

Burning results

334 Figure 4 shows chromatograms from I/SVOCs in the gas and particle phase from burning a

335 cow dung cake sample collected from SPE disks and PTFE filters during a whole 30-minute

336 burn, after passing through a dilution and cooling chamber. The saturation concentration C_i^* at

337 298 K is provided as an alternative x-axis and has been calculated for each *n*-alkane, *i*, using:

$$C_i^* = \frac{M_i 10^6 \zeta_i P_{L,i}^o}{760RT} \tag{E1}$$

338

339 where M_i = molecular weight of VOC *i* (g mol^{-1}), ζ_i = activity coefficient of VOC *i* in the

340 condensed phase (assumed to be 1), $P_{L,i}^o$ = liquid vapour pressure of VOC *i* in Torr, R = gas

13

341   constant (8.206×10^{-5} m^3 atm mol^{-1} K^{-1}) and T = temperature in Kelvin (Lu et al., 2018). The

342   constant 760 Torr has been used to convert between units of atm and Torr where 1 atm = 760

343   Torr. $P^{o}_{L,i}$ values have been calculated from EPA Estimation Programme Interface Suite data at

344   298 K (EPA, 2012)**.** The SPE disks showed 1295 peaks with unique mass spectra and captured

345   gaseous VOCs and I/VOCs with $C*$ ~ 1×10^8 - 5×10^2 μg m^{-3} at 298 K. The largest peaks were

346   from alkanes, 1-alkenes, limonene, phenolics, substituted naphthalenes, furans and substituted

347   pyridines. The PTFE filters captured 1604 I/SVOCs and low/non-volatility VOCs (L/NVOC)

348   with unique mass spectra present in the aerosol phase from $C*$ ~ 5×10^6-1×10^{-5} μg m^{-3} at 298

349   K. A transition can be seen in the two chromatograms from the gas to the aerosol phase. Species

350   with a saturation vapour concentration less than 5×10^4 μg m^{-3} at 298 K were predominantly in

351   the aerosol phase after passing though the dilution chamber. A large region of more polar

352   components was present in the I/SVOC region from $C*$ 5×10^4-5×10^0 μg m^{-3} at 298 K and

353   contained sugar pyrolysis products and highly substituted aromatics such as those with ketone,

354   ether and di and trisubstituted phenol substituents. Many alkanes, from *n*-octadecane to *n*-

355   triatriacontane were present, mainly in the SVOC region. The LVOC region was dominated by

356   a series of sterols and stanols. GCxGC provided extremely high resolution to allow

357   deconvolution of complex samples. The insert in Figure 4 shows how the complexity of the

358   SPE chromatogram can be further resolved by looking at a single ion chromatogram, for

359   example m/z = 57, which highlighted aliphatic non-polar peaks, with large peaks for alkanes

360   from *n*-nonane to *n*-nonadecane.

361   Figure 5 shows overlaid peak markers from SPE disks and PTFE filters from a 30-minute cow

362   dung cake burn coloured by functionality and phase. Over 3000 peaks with individual mass

363   spectra were identified. The complexity of emissions was vast, with 473 PAHs (light brown)

364   forming a group towards the top centre to right of the chromatogram. The most abundant

365   calibrated PAH in the gas phase was naphthalene, followed by methyl and dimethyl

366   naphthalene isomers. A range of methyl, dimethyl, tri and tetramethyl naphthalenes as well as

367   ethyl, propyl, butyl and methyl propyl isomers were detected. Naphthalene isomers substituted

368   with aldehydes, carboxylic acids and nitriles were also released. Biphenyl and a range of

369   methyl, dimethyl and ethyl biphenyls were also released. A range of other PAHs such as

370   acenaphthylene, fluorene, azulene, quinoline, chamazulene, benzophenone, stilbene and

371   benzofurans along with their alkyl substituted isomers were also in the gas phase. A large

372   amount of highly substituted, larger PAHs with more than 3 aromatics rings in their structure

373   were present in the aerosol phase.
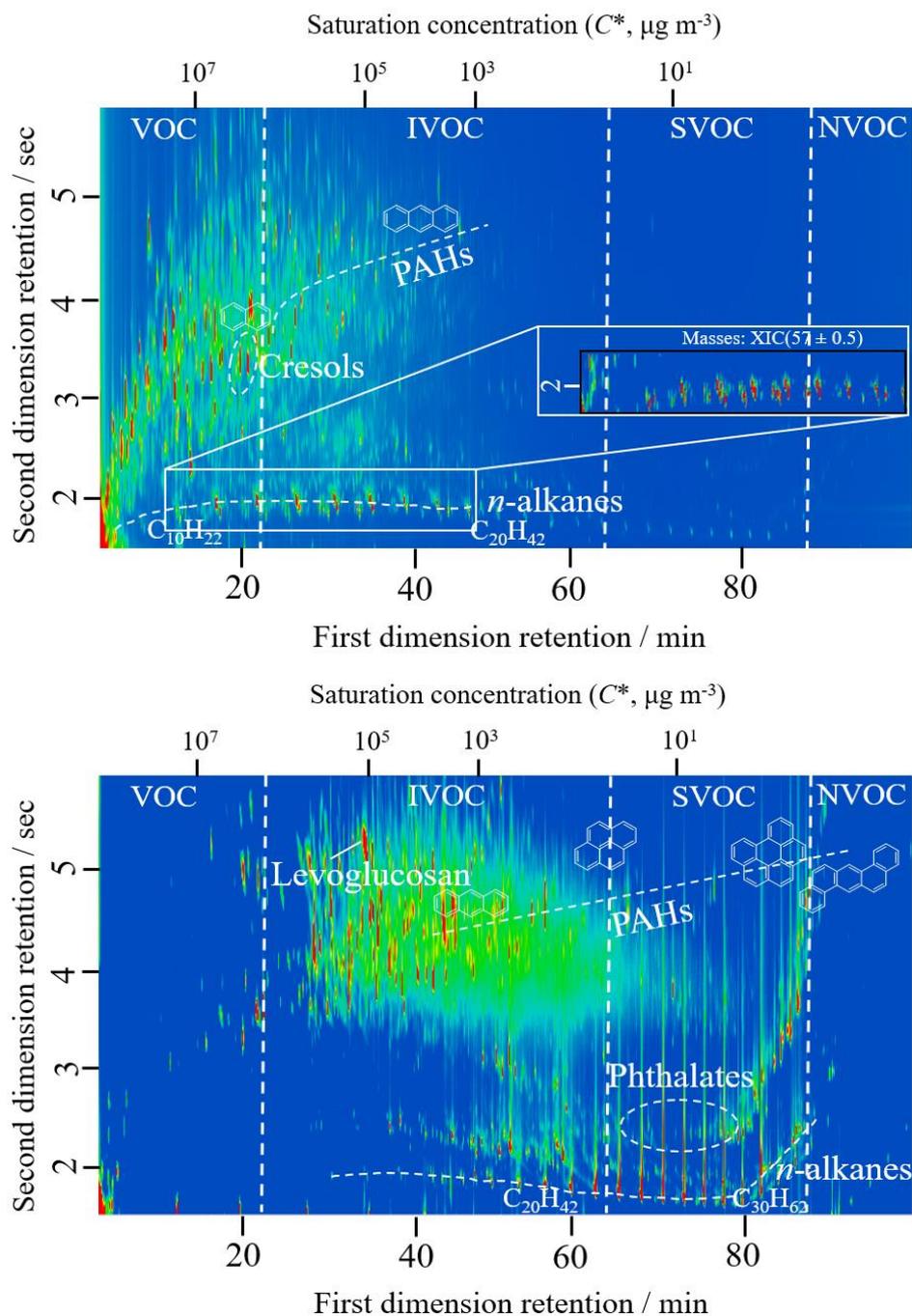
374 Figure 4. Chromatogram of SPE (top) and PTFE (bottom) extracted samples from the entire
375 burn of cow dung cake. *n*-Alkane and PAH series are marked on the chromatograms. The
376 saturation concentration scale matches the *n*-alkane series.
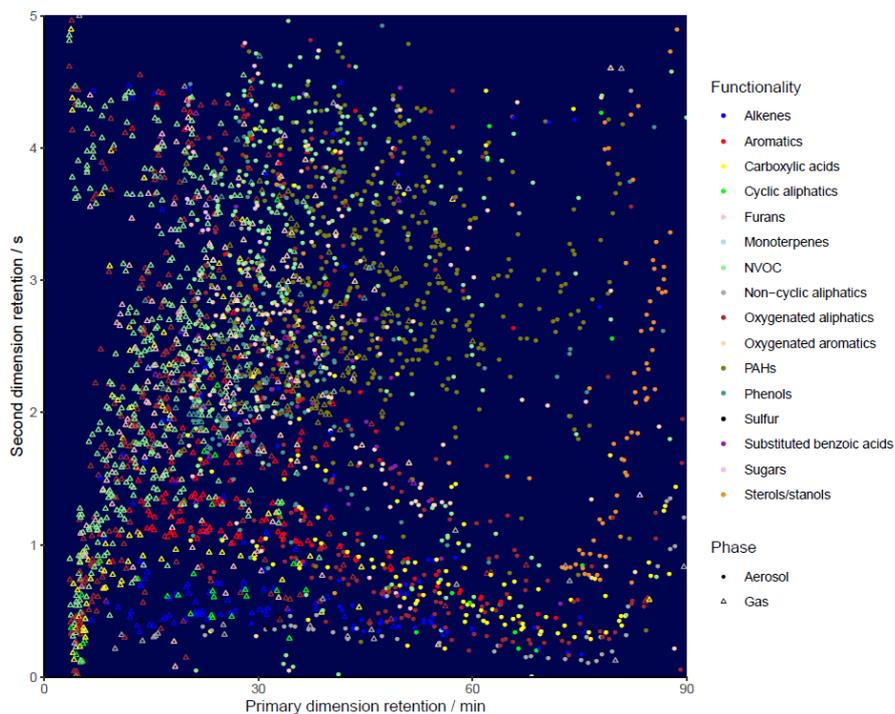
Figure 5. Gas and particle phase composition of I/SVOC emissions from burning cow dung cake collected onto SPE disks and PTFE filters, split by functionality where empty triangles indicate peaks in the gas phase and solid circles show peaks in the aerosol phase.

Other peaks present in Figure 5 included 145 alkenes, mostly towards the bottom of the chromatogram, along with a row of 95 non-cyclic aliphatic and 44 cyclic aliphatic species. Above was a row of 406 substituted aromatics, 208 carboxylic acids and 79 sterols/stanols as well as 753 oxygenated hydrocarbons containing a range of ether, alcohol and aldehyde functionalities. 250 peaks were from oxygenated aromatics, 170 from phenols and 44 from substituted benzoic acids. In addition to this there were 118 furanic species, 3 monoterpenes and 161 sulphur containing VOCs.

A wide array of NVOCs were present in the cow dung cake samples, with over 600 nitrogen containing peaks including aromatics such as pyridines and pyrizines (123), amines (82), amides (77), nitriles (74), 7-membered heterocycles (1), 6-membered heterocycles (28), 5-membered heterocycles including aromatics such as pyroles as well as pyrolines and pyrolidines (97), 4-membered heterocycles (6), 3-membered heterocycles (6), nitrogen containing PAHs (38) imidazoles (22), imines (4), isocyanates (3), hydrazines (7), carbamic

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395   acids (3), azoles (33) oximines (3) and sulfur containing nitrogen compounds (14). Previous

396   studies have measured the nitrogen content of cow dung cake to be as high as 1.9 % (Stockwell

397   et al., 2014) in comparison to other fuel types such as fuel woods (0.14-0.35 %), rice straws

398   (0.4 %) and coal (0.6 %). The large amount of NVOCs are likely formed from the volatilisation

399   and decomposition of nitrogen-containing compounds within the cow dung cake, such as free

400   amino acids, pyrroline, pyridine and chlorophyll (Leppalahti and Koljonen, 1995; Burling et

401   al., 2010; Ren and Zhao, 2015). NVOCs are of concern because they can be extremely toxic

402   (Ozel et al., 2010b; Ozel et al., 2010a; Ozel et al., 2011; Ramírez et al., 2012, 2014; Ramírez

403   et al., 2015; Farren et al., 2015) and amines in particular can change the hydrological cycle by

404   leading to the creation of new particles (Smith et al., 2008; Kirkby et al., 2011; Yu and Luo,

405   2014) which act as cloud condensation nuclei (Kerminen et al., 2005; Laaksonen et al., 2005;

406   Sotiropoulou et al., 2006).

407   Figure 6 shows a comparison of organic aerosol composition observed from different fuel types

408   (LPG, fuel wood, sawdust and municipal solid waste). The measured emissions had very

409   different compositions, reflecting the variability of organic components produced from

410   different sample types. Sawdust, municipal solid waste and cow dung cake (shown in Figure

411   4) emitted a wide range and complexity of species. Particle phase emissions from LPG burning

412   were minimal, with most peaks from the internal standard or contaminants in the solvent. Fuel

413   wood samples released more organic components into the aerosol phase, with the majority of

414   IVOCs with $C^* \sim 1.2 \times 10^5 - 7 \times 10^1$ μg m^{-3} at 298 K. The largest peak belonged to levoglucosan,

415   with other peaks from monoaromatics with several polar substituents such as ethers and

416   phenols, for example dimethoxyhydrotoluene and syringyl acetone. These were likely from the

417   depolymerisation of lignin (Simoneit et al., 1993; Sekimoto et al., 2018), an amorphous

418   polymer constituting about 25 % of fuel woods (Sjöström, 1993) and formed of randomly

419   linked, high-molecular weight phenolic compounds (Shafizadeh, 1982).

420   Sawdust, not a widely used fuel source, released many I/S/L/NVOC components in the aerosol

421   phase over a much wider range ($C^* \sim 5.8 \times 10^5 - 1 \times 10^{-3}$ μg m^{-3} at 298 K). The largest peak was

422   from levoglucosan with another large peak from squalene. Many peaks were from polar

423   substituted aromatics as well as many PAHs and their substituents, such as 2-methyl-9,10-

424   anthracenedione. The largest peak from municipal solid waste burning was also levoglucosan,

425   but these samples released fewer of the polar substituted monoaromatics than other samples.

426   Municipal solid waste released alkanes and SVOC species such as terphenyls, alkanes and

427   many PAHs.
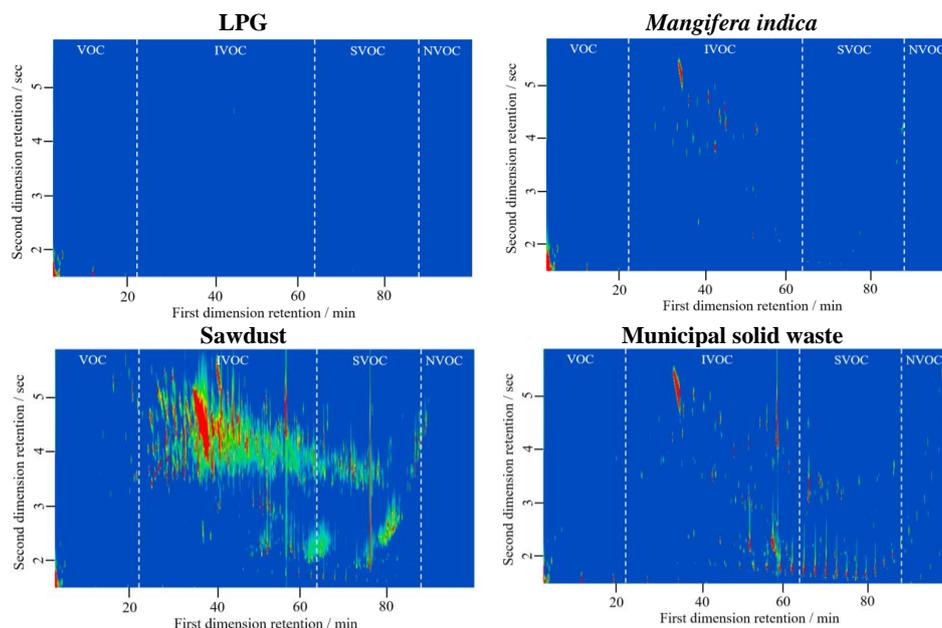
428 Figure 6. Measurements of organic aerosol from a range of different fuel types, with the contrast at the
429 same scale.

Molecular markers for domestic fuels

431 Cow dung cake samples emitted a range of sterols/stanols, which have been reported previously

432 (Sheesley et al., 2003). This study suggested that 5β-stigmastanol, coprostanol, and cholestanol

433 could be used as tracers for emissions from cow dung cake burning. This is because in higher

434 animals, anaerobic microbial reduction of sitosterol and cholesterol forms the distinctive β

435 configuration of the C-5 proton of 5β-stigmastanol and coprostanol. This contrasts with the α

436 C-5 proton caused by aerobic digestion in aquatic environments. Jayarathne et al. (2018)

437 reported 5β-stigmastanol emissions from hardwood, and Fine et al. (2001) reported 5α-

438 stigmastanol emissions from hardwood. Four fuel wood samples in our study showed

439 emissions of an isomer of stigmastanol, a result similar to Jayarathne et al. (2018) that 5β-

440 stigmastanol was not unique to cow dung cake burning or the MS measurement method used

441 was unable to distinguish between 5α- and 5β-stigmastanol. Cholestanol and coprostanol were

442 found uniquely in cow dung cake samples in our study, and suggested that they can be used as

443 unique tracers for cow dung cake burning.

444 Fuel wood samples generally released fewer organic components into the aerosol phase than

445 samples such as cow dung cake, MSW and sawdust. Levoglucosan has been traditionally

446 suggested as a tracer for biomass burning emissions, however, emissions of levoglucosan from

447 a range of sources mean that this is of limited use as a unique tracer of woodsmoke emissions

448 in regions with multiple burning sources. This could potentially be resolved in future studies

449 by examining the ratio of levoglucosan to other sugars in different source types to differentiate

450 different biomass burning sources as the chemical composition of different sources should

451 determine the emission ratio of levoglucosan to other sugar pyrolysis products (Sheesley et al.,

452 2003).

453 The presence of a wide range of terphenyls in municipal solid waste samples in this study was

454 not unique. Jayarathne et al. (2018) suggested triphenyl benzene to be a unique tracer of waste

455 burning emissions. Whilst this study found triphenyl benzene present in one cow dung cake

456 sample and in municipal solid waste samples, the waste samples emitted on average 19

457 terphenyls, many more than in the cow dung cake samples (2). Terphenyls have been

458 previously reported from incineration of waste (Tong et al., 1984) and our study suggests that

459 these compounds are good indicators of municipal solid waste burning.

460 **Total identification**

461 Figures 7A and 7B show a comparison of the relative abundance of peaks identified, defined

462 here as the sum of peak areas identified and calibrated using genuine standards for compounds

463 present in the SPE and PTFE samples compared to the total observed peak area (using the blank

464 subtracted total ion current, TIC).

465 Figure 7A shows that between 15 and 100 % of the peak area of the TIC in the SPE

466 chromatogram could be identified. The highest proportion of species that could be identified

467 was from fuel wood (67 %), followed by crop residue (57 %), charcoal (48 %), municipal solid

468 waste (46 %), cow dung cake (39 %) and sawdust (16 %). Lower total identification in samples

469 such as cow dung cake was due to increased complexity of emissions, which were not covered

470 by the standards used.

471 Figure 7B shows that between 7 – 100 % of the organic composition of aerosol released from

472 burning was identified. Generally, a much lower proportion of organic matter within aerosol

473 samples was identified due to a lack of genuine standards available, particularly in complex

474 samples. The lowest mean relative contribution identified from samples was sawdust (9 %),

475 followed by cow dung cake (11 %) and municipal solid waste (16 %). A larger relative

476 contribution was identified from fuel woods (34 %) and charcoal (39 %) and due to less

477 complex emissions. A large relative contribution of some fuel woods was identified from

478 *Saraca indica* (91 %) and *Pithecellobium spp* (82 %) due to a low amount of organic matter

479 released from these samples. This also influenced the percentage identification from crop

480 residue which achieved 46 % identification, due to only 3 samples with 98 % identification

481 from *Solanum melongena* but only 26 % from *Cocos nucifera* and 13 % from *Brassica spp.*

482 100 % of the aerosol released from LPG was quantified due to little being released into the

483 aerosol phase and this was principally composed of PAHs. These low levels of identification

484 of organic aerosol are in line with those reported by Jen et al. (2019) where unknown chemical

485 species represented 35-90 % of the observed organic aerosol mass from biomass burning

486 samples.

487 **Composition**

488 Figure 7C provides an indication of I/SVOC composition on SPE disks by mass of quantified

489 species, assuming no compound breakthrough. Phenolic and furanic compounds are the most

490 abundant I/SVOC species released from all sample types, except for LPG. As a proportion of

491 the total mass of species quantified with genuine standards on SPE disks, phenols released from

492 fuel woods (22-80 %) represented the largest range, with large amounts released from

493 municipal solid waste (24-37 %), cow dung cake (32-36 %), crop residue (32-57 %) and

494 sawdust (46 %). High emissions of phenolic compounds were of significance because

495 phenolics contribute significantly to SOA production from biomass-burning emissions (Yee et

496 al., 2013; Lauraguais et al., 2014; Gilman et al., 2015; Finewax et al., 2018).

497 Large emissions of furanic species were measured from fuel wood (6-59 %), municipal solid

498 waste (35-45 %), cow dung cake (39-42 %), crop residue (25-44 %) and sawdust (43 %). These

499 were important as furans can be toxic and mutagenic (Ravindranath et al., 1984; Peterson,

500 2006; Monien et al., 2011; WHO, 2016) and have been shown to be some of the species with

501 the highest OH reactivity from biomass burning emissions (Hartikainen et al., 2018; Coggon

502 et al., 2019). Furans have also been shown to result in SOA production (Gómez Alvarez et al.,

503 2009; Strollo and Ziemann, 2013) with 8-15 % of SOA produced from combustion of black

504 spruce, cut grass, Indonesian peat and ponderosa pine estimated to originate from furans and

505 28-50 % of SOA from rice straw and wiregrass (Hatch et al., 2015). Furans from biomass

506 burning emissions are thought to come from low temperature depolymerisation of hemi-

507 cellulose (Sekimoto et al., 2018) and from large alcohols and enols in high-temperature regions

508 of hydrocarbon flames (Johansson et al., 2016).

509 Emissions of alkanes were most important from combustion of cow dung cake and municipal

510 solid waste (4-9 %), with only small quantities released from various fuel wood samples (< 2

511 %) and crop residue (< 1%). This reinforced previous studies which found emissions of C_{12}-

512 C_{39} n-alkanes from municipal waste incinerators (Karasek and Tong, 1985). PAH emissions

513 represented (3 – 15 %) of the total quantified emission by mass for samples other than LPG

514 and have carcinogenic and mutagenic properties (IARC, 1983, 1984; Nisbet and LaGoy, 1992;

515 Lewtas, 2007; Zhang and Tao, 2009; Jia et al., 2011). They can damage cells through the

516 formation of adducts with DNA in many organs such as the kidneys, liver and lungs (Vineis

517 and Husgafvel-Pursiainen, 2005; Xue and Warshawsky, 2005).

518 Figure 7D shows the quantified aerosol mass was largely dominated by levoglucosan, with a

519 particularly significant contribution in the fuel wood samples (13-98 %). This was similar to a

520 previous study of fuel wood samples from Bangladesh, where levoglucosan was the largest

521 contributor to aerosol mass (Sheesley et al., 2003). Levoglucosan emissions were also large

522 from cow dung cake (30-58 %), which contrasted with the findings of Sheesley et al. (2003).

523 This could be due to differences in the feeding of cows leading to differences in residual

524 undigested organic matter in cow dung cake samples as well as differences in preparation

525 between samples collected in Bangladesh and those in this study, which had additional dried

526 biogenic material, such as straw, mixed into samples. Levoglucosan emissions were also high

527 from sawdust (91 %), crop residue (19-85 %) and municipal solid waste (58-75 %), with

528 municipal solid waste emissions likely from cellulosic material collected with samples.

529 Levoglucosan emissions from charcoal (76 %) were significant as a proportion of emissions.

530 Emissions from charcoal were low, which meant that a small emission of levoglucosan

531 represented a large proportion of total emissions. It was likely that the sample collected here

532 may have contained small amounts of cellulosic organic matter that led to the emission of

533 levoglucosan.

534 Emissions of alkanes in the gas and particle phases were similar by source type, with particulate

535 alkanes emitted principally from cow dung cake and municipal solid waste samples. Emissions

536 of particulate phenolics were large as a proportion of total quantified mass with genuine

537 standards when the total emission of other components was low. For example, phenolics

538 represented a large proportion of emissions from the fuel wood species *Morus spp* and

539 *Pithecellobium spp* with the mass principally from dimethoxyphenols. Emissions from LPG

540 were mainly PAHs and very low.

541 Whilst SPE samples for these compounds remained semi-quantitative due to slight

542 breakthrough, the detection of high emissions of phenolics and furanics in the gas phase from

543 burning was in line with recently published studies (Hatch et al., 2015; Stockwell et al., 2015;

544 Koss et al., 2018). Relatively low levels of total quantified material within the aerosol phase

545 was in line with the current literature (Jen et al., 2019) but meant that this analysis was not

546 entirely reflective of the organic fraction for complex samples. It is likely that this study

547 overemphasises the contribution of levoglucosan in complex aerosol samples, relative to other

548 components present at lower levels (Sheesley et al., 2003; Jen et al., 2019). Future instrument

549 development could allow better quantification of complex burning and ambient samples by

550 splitting the eluent between a -MS and -FID. This study suggests that future research uses lower

551 sample volumes, thicker SPE disks and studies the adsorption characteristics of VOCs to the

552 surfaces of these disks.
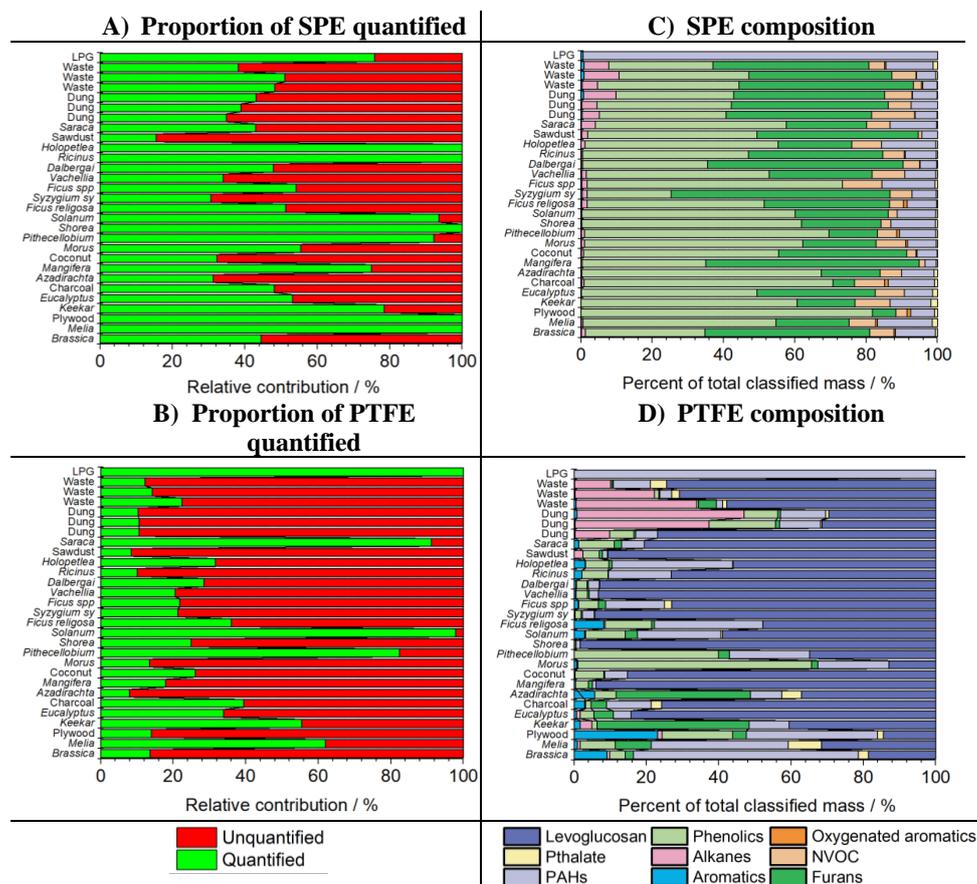
553 Figure 7. Area of organic matter quantified with genuine standards, as a fraction of total ion current
554 (TIC) (7A and 7B, left panel). Semi-quantitative/quantitative analysis of SPE/PTFE fraction (7C and
555 7D, right panel).

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Development of emission factors

Emission factors have been developed for PAHs (see Figure 8 and the Supplementary Information S5 for table of emission factors by individual fuel type) by calculating the total volume of air convectively drawn up the flue and relating this to the mass of fuel burnt (see the Supplementary Information S6 for details of calculation). Emission factors for sawdust (1240 mg kg^{-1}), municipal solid waste (1020 mg kg^{-1}), crop residue (747 mg kg^{-1}) and cow dung cake (615 mg kg^{-1}) were generally larger than for fuel wood (247 mg kg^{-1}), charcoal (151 mg kg^{-1}) and LPG (56 mg kg^{-1}). The measurement of higher emission factors for cow dung cake than fuel wood was consistent with that observed in other studies (Bhargava et al., 2004; Gadi et al., 2012).
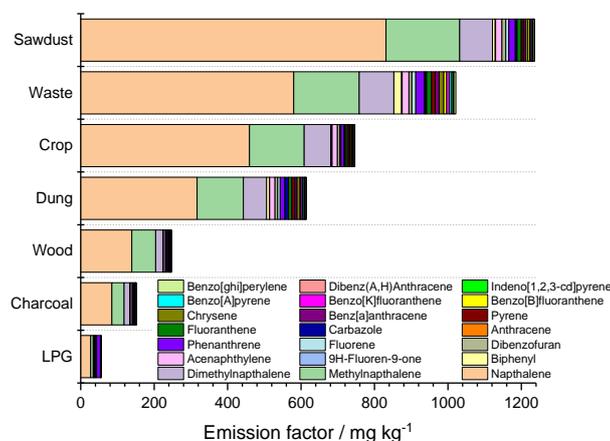
Figure 8. Mean PAH emission factors by fuel type.

A wide range of emission factors were measured for fuel wood samples from 50 mg kg^{-1} for *Prosopis* to 907 mg kg^{-1} for *Ficus religosa*. For most samples, PAH emissions in the gas phase were dominated by naphthalene, methylnaphthalenes and dimethylnaphthalenes with gas-phase PAHs observed up to pyrene. For fuel wood, crop residue, municipal solid waste and cow dung cake the percentage of PAHs in the gas phase decreased from 97 %, 96 %, 91 % to 89 %. PAHs from LPG showed the largest fraction in the gas phase (99.9 %) compared to the aerosol phase (0.1 %). Figure 9 shows gas and particle phase PAH emissions by individual sample type, excluding naphthalene as well as C_1- and C_2-substituted naphthalenes. PAHs were present in the aerosol phase from dibenzofuran ($C_{12}H_8O$) to benzo(ghi)perylene ($C_{22}H_{12}$).

23

578
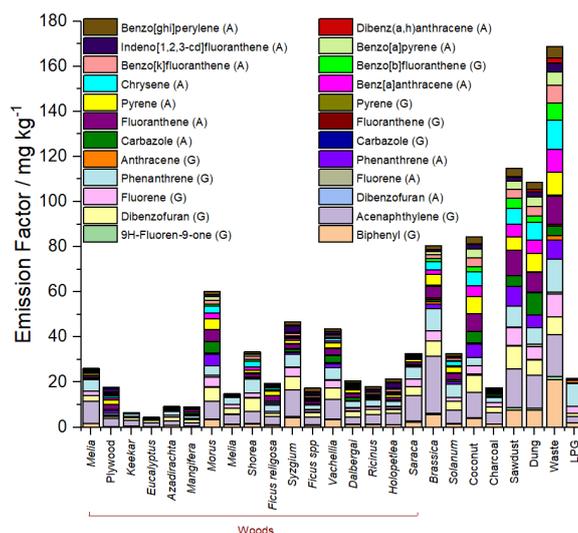
Figure 9. Emission factors of PAHs measured from SPE/PTFE where (G) and (A) represent gas- and aerosol-phase samples, respectively, excluding naphthalene as well as naphthalenes with C_1 and C_2 substituents.

Table 3 shows a comparison of the mean emission factors measured in our study with previous studies. The mean fuel wood total PAH emission factor measured in our study (247 mg kg^{-1}) was a factor 4.7-5.6 larger than those measured by Gadi et al. (2012) and Singh et al. (2013) of 44 and 53 mg kg^{-1}, respectively, for similar fuel woods collected across New Delhi and the Indo-Gangetic Plain. The PAH emission factor measured for cow dung cake (615 mg kg^{-1}) was around a factor of 10 larger than those previously measured (60 mg kg^{-1}). The larger total emission factors for fuel wood and cow dung cake was a result of high emissions of gas-phase PAHs measured using PTR-ToF-MS (51-896 mg kg^{-1} for fuel wood and 446-660 mg kg^{-1} for cow dung cake) compared with previous measurements made using PUF plugs (7 mg kg^{-1}). This indicated that either the PTR-ToF-MS was able to better detect and characterise gas-phase emissions than previous methods and suggested either breakthrough or off gassing of smaller gas-phase PAHs from PUF plugs or measurement of significant quantities of other $C_{10}H_8$ isomers on the PTR-ToF-MS. This may highlight an underestimation of 2-ring gas-phase PAH emissions in previous burning studies. Gadi et al. (2012) measured PAH emissions in the particle phase, with the mean emission for fuel wood (44 mg kg^{-1}) greater than our study (9 mg kg^{-1}). Particulate phase emissions of PAHs measured by Singh et al. (2013) from fuel wood (45 mg kg^{-1}) were also larger than our study. By contrast, particle phase PAH emissions from cow dung cake in our study (66 mg kg^{-1}) were comparable to those measured previously of 57-60 mg kg^{-1} (Gadi et al., 2012; Singh et al., 2013). Variability in emission of particulate-phase

601 PAHs in our study compared to literature was likely to be highly influenced by the efficiency
602 of combustion of samples. Although not measured in our study, differences in moisture content
603 between samples in our study and literature were likely have a large influence on the total
604 amount of PAHs emitted and may explain the differences in particle-phase emissions.

605 The particulate phase PAH emission factors from municipal solid waste combustion in our
606 study (14-181 mg kg^{-1}) were much smaller than those of previous studies (1910-8486 mg kg^{-1}
607), but the number of samples was limited. Emissions from coconut shell have not been well
608 studied, making comparisons difficult (Gulyurtlu et al., 2003). The emission of particulate
609 phase PAHs from sawdust in our study (62 mg kg^{-1}) was less than that previously reported 259
610 mg kg^{-1}, but our study found large gas phase PAH emissions (1175 mg kg^{-1}). Particulate PAH
611 emissions from the crop residue burnt in our study (13-53 mg kg^{-1}) fell within the range
612 reported by Kim Oanh et al. (2015) of 0.34-34 mg kg^{-1} for rice straw. Those reported by Wiriya
613 et al. (2016) were smaller (0.47 mg kg^{-1}), but were from samples dried in an oven at 80 °C for
614 24 hours and ignited by an LPG burner and were likely to represent more complete combustion
615 conditions. Emissions of PAHs from charcoal in our study (151 mg kg^{-1}) were larger than those
616 measured for South Asian fuels (25 mg kg^{-1}), caused principally by larger measurement of gas-
617 phase species by PTR-ToF-MS. Both our study, and that of Kim Onah et al. (1999) showed
618 charcoal released the least amount of PAH per kg burnt for biofuels. LPG combustion released
619 less particulate PAHs (0.1 mg kg^{-1}) than previous studies (0.8 mg kg^{-1}), but also included a
620 small gas-phase emission (56 mg kg^{-1}). Differences in the distribution of PAHs found in the
621 gas and aerosol phases between our study and literature were also likely to be influenced by
622 the different sample dilutions and gas-to-aerosol partitioning prior to measurement.

623 Table 3. PAH emission factors measured in our study compared to values from literature for similar
624 fuel types.

Fuel	PAH (mg kg^{-1})			
	Gas	Particle	Total	Ref
Wood	51-896	0.4-34	51-907	Our study
		1-12		(Hosseini et al., 2013)
	22-111	0.4-6	24-114	(Kim Oanh et al., 2005)
	-	44	44	(Gadi et al., 2012)
	7	45	52	(Singh et al., 2013)
		805-7294		(Kakareka et al., 2005)
			43	(Lee et al., 2005)
	66	0.8	67	(Kim Oanh et al., 2002)
	105	4	105	(Kim Oanh et al., 1999)
Dung	446-660	48-98	493-710	Our study
	-	59	-	(Gadi et al., 2012)
	3	57	60	(Singh et al., 2013)

Waste	696-1233	14-181	776-1414	Our study
	-	8486	8486	(Kakareka et al., 2005)
	-	1910	1910	(Young Koo et al., 2013)
Crop	205-1231	13-53	219-1255	Our study
	-	-	5-683	(Jenkins et al., 1996)
	-	-	3-50	(Lu et al., 2009)
	-	-	129-569	(Wei et al., 2014)
	5-230	0.3-34	5-264	(Kim Oanh et al., 2015)
	-	0.47	-	(Wiriya et al., 2016)
Sawdust	1175	62	1236	Our study
	259	261		(Kim Oanh et al., 2002)
Charcoal	147	4	151	Our study
	25	0.1	25	(Kim Oanh et al., 1999)
LPG	56	0.1	56	Our study
	-	0.8	-	(Geng et al., 2014)

625

Conclusions

626

627 This paper demonstrated an extraction technique for biomass burning samples collected onto

628 SPE disks and PTFE filters, which was well suited to analysis of non-polar species. A range of

629 samples relevant to burning in India were collected and analysed, which showed large

630 differences in the composition of organic matter released. The separation power of GCxGC has

631 been used to identify an extensive range of I/SVOCs in both gas and particle phases with 15-

632 100 % of gas-phase emissions and 7-100 % of particle-phase emissions characterised.

633 The ability to quantify species on SPE disks was assessed and scope for future studies which

634 should assess the adsorption characteristics of IVOCs onto SPE disks has been provided. It is

635 recommended that breakthrough of IVOCs collected onto SPE disks at lower sample volumes

636 is evaluated, and better methods for quantification of complex samples are developed. Further

637 samples from a wider range of sources would enable a better understanding of the drivers of

638 poor air quality in the developing world, such as crop residue burning. This study found that

639 cholestanol and coprostanol were unique to cow dung cake burning samples and these species

640 were therefore suggested as tracers for emissions from cow dung cake burning. Similarly,

641 municipal solid waste burning released many terphenyls, which could act as good indicators of

642 this source. This study found that phenolic and furanic species were the most important gas-

643 phase emissions by mass of I/SVOCs from biomass burning. New emission factors were

644 developed for US EPA criteria PAHs present in gas and aerosol phases from a large range of

645 fuel types. This suggested that many sources important to air quality in the developing world

646 are larger sources of PAHs than conventional fuel wood burning.

647

648 *Author contributions.* GJS developed the ASE method, GC method, collected samples,
649 organised logistics, extracted/analysed samples and lead the paper. BSN collected samples and
650 assisted with logistics. WJFA measured VOCs by PTR-ToF-MS, supported by CNH, LKS and
651 NT. ARV assisted in running and organising of experiments. NJF, JRH and MWW assisted in
652 GCxGC-ToF-MS method development. SJS assisted in ASE method development. RA, AM,
653 RJ, SA and LY collected samples, carried out the burning experiments and measured gas
654 volumes up the flue. SSBMY aided complex sample analysis. EN, NM, RG, SKS and JDL
655 contributed to logistics and data interpretation. TKM and JFH provided overall guidance with
656 setup, conducting, running and interpreting experiments.

657 *Competing interests*. The authors declare that they have no conflict of interest.

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