

1 **Point-by-point response to reviewer comments**

2 We would like to thank the reviewers for their positive and constructive reviews of this paper.

3 We address the specific points of each reviewer below. Reviewer comments in blue, author
4 response in black, text added or amended in paper in purple.

5 Reviewer comment 1

6 Stewart et al present an extremely detailed analysis of emissions of lower-volatility vapours
7 (I/SVOCs) and particulate matter from domestic fuel burning. They focused on fuels typically
8 burned in India. One novel aspect of this study is the use of GCxGC to identify hundreds of
9 individual species emitted from each fuel type.

10 Overall the paper is very well written and easy to follow. The description of the methods, in
11 particular, is very detailed and shows the high level of specificity in the measurements and
12 the extensive QA/QC. The paper is a little bit tough to follow because the section (and sub-
13 section) headers all have the same style and font. This makes it hard to determine the
14 organization of the manuscript (e.g., there are several sub-sections to C1 ACPD Interactive
15 comment Printer-friendly version Discussion paper the Methods, but the headers are
16 indistinguishable from the following Results section). Consider numbering the sections.

17 We thank the reviewer for their suggestion and all sections are now numbered.

18 This paper presents a potentially valuable dataset containing measurements of hundreds of
19 species. However, only the emission factors for PAHs are listed in detail in Table 1 of the SI. I
20 think it would be beneficial for the authors to share the full suite of measured compounds,
21 either in the SI or in an online repository, so that data can be used by other researchers in the
22 future.

23 We have added this to the SI.

24 One potential weakness is that there is only one sample for most of the fuels tested (Table 1).
25 The authors should acknowledge that there can be significant burn-to-burn differences in
26 emissions.

27 This is indeed a limitation and is now acknowledged in the main text.

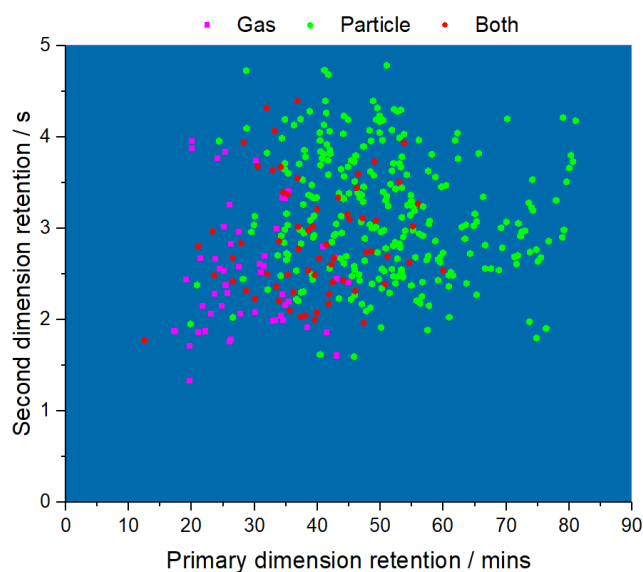
28 Variability in emission of particulate-phase PAHs in our study compared to literature was likely
29 to be highly influenced by the efficiency of combustion of different fuel types. This may also
30 be explained by measuring only once for many of the fuel types, due to significant burn-to-
31 burn differences in emissions.

32 I'm not certain that Table 2 should be in the main text. This table seems to be part of the
33 QA/QC, and it seems to me that it would be better placed in the SI.

34 This table is now in the SI.

35 Figure 5 is hard to interpret. The symbols are very small (as is the legend), and as the authors
36 note in the text, there are a lot of species shown. Since the text focuses on the PAHs, it might
37 help readability to put this version of the figure in the SI and only show the PAHs (with larger
38 symbols) in the main text.

39 Figure 5 is now in the SI and this figure has been replaced with a lighter version which just
40 focusses on PAHs as suggested. This is much clearer and highlights the regions of the
41 chromatogram dominated by species in each phase.



42
43 Figure 1. Gas and particle phase composition of PAH emissions from burning cow dung cake.

45 Figure 7 and line 548-550 suggest that more of the mass could be speciated with new
46 instruments. However Figure 7 focuses on peaks that can be positively matched with
47 something in one of the standards. Is it possible to infer composition based on the mass
48 spectra of the unidentified peaks?

49 This is tackled in the companion paper acp 2020-892, which may not have been visible at the
50 point of submission of this manuscript. High levels of total speciation are achieved (>90 %)
51 using other instruments. We will direct readers to acp 2020-892 in the text.

52 Quantitative emission factors of VOCs from the combustion of solid fuels characteristic to
53 Delhi are provided in a companion publication (Stewart et al., 2020b).

54 Grammatical comments: Line 152 and 153 - it seems like "samples" in the former line mean
55 the sampled media (filters and SPE), and in the latter it means the fuels. Please clarify.

56 This has been clarified throughout, with samples referring to the organic components
57 collected and analysed and fuel types referring to the biofuels collected and burnt.

58 Line 195 - define EtOAc Line 347 uses NVOC to indicate "non-volatile", but the paragraph
59 starting at line 389 seems to use NVOC to indicate "nitrogen-containing." Please clarify.

60 EtOAc and MeOH are now defined in the text as requested.

61 The reviewer makes a valid point about the use of NVOC as "non-volatile" and therefore the
62 nitrogen containing VOCs are no longer abbreviated as NVOCs.

Commented [JH1]:

63 Reviewer comment 2

64 Stewart et al. have developed a gas-chromatography-based analytical technique to speciate
65 and quantify semi-volatile and intermediate volatility organic compounds (S/IVOCs) and
66 applied this technique to measure emissions of S/IVOCs from domestic fuels used in Delhi,
67 India. S/IVOCs are important precursors to ozone and aerosol formation in the atmosphere
68 and there is need to develop robust analytical techniques to speciate and quantify their
69 emissions. Biomass burning is an important source of global air pollution and the type of
70 biomass burning studied here (i.e., biofuel combustion) is a particularly understudied
71 emission source. Hence, the work described in the manuscript is well motivated. I should also
72 commend the authors for a well written manuscript that provides all the necessary details to

73 comment on the methods and the interpretation of the results. The analytical method
74 development was well designed and the application was very well described, although I
75 should note that I am not trained as an analytical chemist. This should serve as a useful
76 resource for researchers doing similar work in the atmospheric community. The primary
77 results of speciation and quantification are well described too but, given the large dataset
78 that is being analyzed, only a small fraction of the data are actually presented. I recommend
79 the publication of this study in Atmospheric Chemistry and Physics after the authors have had
80 a chance to respond to my, mostly big-picture and minor, comments.

81

82 Big-picture comments: 1. The introduction seems too generic and long at the moment and
83 needs to be realigned to describe the state-of-the-science and gaps as it relates to the key
84 findings from this work. For instance, lines 53-68 discuss S/IVOC emissions generally but don't
85 focus on those emissions from biomass burning.

86 The introduction has been rewritten, and shortened, as suggested with the new structure: (1)
87 general introduction to organic emissions from biomass burning, (2) state-of-the-art studies
88 focussed on SOA formation from biomass burning, (3) state-of-the-art studies focussed on
89 GCxGC analysis of biomass burning emissions and the research gap to be filled, (4) state-of-
90 the-art studies focussed on detailed I/SVOC analyses of south Asian fuels, (5) research gaps
91 to be filled with this work in terms of analytical procedures and poorly understood, but widely
92 used, Asian fuels.

93 General discussion of historical biomass burning I/SVOC measurement procedures,
94 application of GCxGC to other source sectors and Indian air quality has now been removed. It
95 now reads:

96 Biomass burning is one of the most important global sources of trace gases and particles to
97 the atmosphere (Simoneit, 2002; Chen et al., 2017; Andreae, 2019), with residential solid fuel
98 combustion and wildfires emitting significant quantities of organic matter (Streets et al.,
99 2003; Barboni et al., 2010; Chen et al., 2017; Liu et al., 2017; Kiely et al., 2019). Emissions of
100 volatile organic compounds (VOCs) and particulate matter (PM) from biomass burning are of
101 interest due to their detrimental impact on air quality. VOCs react to form ozone and
102 secondary organic aerosol (SOA). Intermediate-volatility and semi-volatile organic

103 compounds (I/SVOCs) are also a significant emission from biomass burning (Stockwell et al.,
104 2015; Koss et al., 2018). I/SVOCs are an important class of air pollutant due to their
105 contribution to aerosol formation (Bruns et al., 2016; Lu et al., 2018). I/SVOC emissions are
106 poorly, if at all, represented in regional inventories and chemical transport models.
107 Consequently, their impact to air quality in developing regions, where solid fuel combustion
108 is a dominant fuel source, is not well understood. Recent studies have shown that the
109 inclusion of I/SVOCs leads to better agreement between modelled and measured values (Ots
110 et al., 2016; Woody et al., 2016; Jathar et al., 2017; Murphy et al., 2017). Global I/SVOC
111 emissions to the atmosphere from biomass burning were estimated to be $\sim 54 \text{ Tg yr}^{-1}$ from
112 2005-2008 (Hodzic et al., 2016), with I/SVOCs contributing in the range 8-15.5 Tg yr^{-1} to SOA
113 (Cubison et al., 2011; Hodzic et al., 2016).

114 SOA formation from biomass burning emissions is poorly understood globally. Important
115 factors include the formation of less volatile products from the oxidation of NMVOCs which
116 partition into the aerosol phase, heterogeneous oxidation of aerosol phase organics as well
117 as plume dilution followed by evaporation and further gas-phase oxidation (Lim et al., 2019).
118 Ahern et al. (2019) showed that for burning of biomass needles, biogenic VOCs were the
119 dominant class of SOA precursor. This study also found that for wiregrass, furans were the
120 most important SOA precursor. Bruns et al. (2016) showed that SOA formation from
121 combustion of beech fuel wood was dominated by 22 compounds, with phenol, naphthalene
122 and benzene contributing up to 80 % of the observed SOA. SOA formation from biomass
123 burning has been shown to be significant in laboratory studies, with SOA yields from the
124 burning of western U.S. fuels reported to be $24 \pm 4 \%$ after 6 h and $56 \pm 9 \%$ after 4 d (Lim et al.,
125 2019). However, the effect of atmospheric of aging on I/SVOCs still remains poorly
126 understood (Liu et al., 2017; Decker et al., 2019; Sengupta et al., 2020).

127 Better understanding of the quantity and composition of I/SVOCs from biomass burning is
128 needed to evaluate their impact on the atmosphere. This is a difficult analytical task, not well
129 suited to conventional analysis with gas chromatography coupled to mass spectrometry (GC-
130 MS). The reason for this is because of the exponential growth of potential isomers with carbon
131 number, which results in a large number of coeluting peaks (Goldstein and Galbally, 2007).
132 The high resolution of two-dimensional gas chromatography (GC \times GC) has been demonstrated
133 as an ideal technique to overcome this issue when analysing complex organic samples in both

134 gas (Lewis et al., 2000; Stewart et al., 2020a) and particle phases (Hamilton et al., 2004; Lyu
135 et al., 2019c). The application of GC×GC to biomass burning emissions has shown hundreds
136 of gaseous I/SVOCs using adsorption-thermal desorption cartridges (Hatch et al., 2015) or
137 solid phase extraction (SPE) disks (Hatch et al., 2018). GC×GC has also been used to analyse
138 the particle phase from samples collected onto PTFE or quartz filters (Hatch et al., 2018; Jen
139 et al., 2019), with the latter study quantifying 149 organic compounds which accounted for
140 4-37 % of the total mass of organic carbon. The process used by Hatch et al. (2018)
141 demonstrated high recoveries of non-polar species from PTFE filters, with lower recoveries
142 from SPE disks. This study highlighted the need for further evaluation of samples collected
143 onto PTFE filters and SPE disks, ideally improving the method to remove undesirable steps
144 such as trimethylsilylation derivatisation, the use of pyridine and centrifuging which led to
145 high evaporative losses. The need to develop improved sampling and measurement
146 techniques for I/SVOCs has also been highlighted. This is because these species often do not
147 transmit quantitatively through the inlet and tubing when measured using online gas-phase
148 techniques (Pagonis et al., 2017).

149 Residential combustion, agricultural crop residue burning and open municipal solid waste
150 burning in the developing world are large, poorly characterised pollution sources with the
151 potential to have a significant impact on local and regional air quality, impacting human
152 health (Venkataraman et al., 2005; Jain et al., 2014; Wiedinmyer et al., 2014). Hazardous
153 indoor air pollution from combustion of solid fuels has been shown to be the most important
154 factor from a range of 67 environmental and lifestyle risk factors causing disease in South Asia
155 (Lim et al., 2012). Despite this, nearly 76 % of rural Indian households depend on solid biomass
156 for their cooking needs (Gordon et al., 2018), using biofuels such as fuel wood, cow dung cake
157 and crop residue. Combustion often takes place indoors, without efficient emission controls,
158 which significantly increases the mean household concentration of pollutants, particularly
159 particulate matter with a diameter less than 2.5 µm (PM_{2.5}). The health effects from this are
160 significant, with an estimated 3.8 million premature deaths globally due to inefficient indoor
161 combustion from cooking (WHO, 2018).

162 Few detailed studies have been conducted examining the composition of I/SVOC emissions
163 from solid-fuel combustion sources from South Asia. Sheesley et al. (2003) used solvent
164 extraction followed by GC-MS to produce emission factors and examine molecular markers

165 from combustion of coconut leaves, rice straw, cow dung cake, biomass briquettes and
166 jackfruit branches collected from Bangladesh. A more recent study extracted PM_{2.5} samples
167 followed by analysis with GC-MS of samples collected from motorcycles, diesel- and gasoline-
168 generators, agricultural pumps, municipal solid waste burning, cooking fires using fuel wood
169 and cow dung cake, crop residue burning and brick kilns in Nepal (Jayarathne et al., 2018).
170 Lack of knowledge regarding major pollution sources hinders our ability to predict air quality,
171 but also the development of effective mitigation strategies for air pollution which leads to
172 health impacts ranging from respiratory illness to premature death (Brunekreef and Holgate,
173 2002). This results in many people living with high levels of air pollution (Cohen et al., 2005;
174 Lelieveld et al., 2015) and 13 Indian cities ranking amongst the top 20 cities in the world with
175 the highest levels of ambient PM_{2.5} pollution, based on available data (Gordon et al., 2018).

176 In this study we develop a more efficient extraction step for the SPE/PTFE technique
177 developed by Hatch et al. (2018), using accelerated solvent extraction into ethyl acetate,
178 which showed high recoveries of non-polar I/SVOCs. Domestic fuels characteristic to Northern
179 India were gathered and organic I/SVOC samples collected onto SPE disks and PTFE filters
180 from controlled laboratory combustion experiments of a variety of fuel woods, cow dung
181 cakes, municipal solid waste samples, crop residues, charcoal and LPG. The samples were
182 extracted using this new technique and analysed with GC×GC coupled to time-of-flight mass
183 spectrometry (GC×GC-ToF-MS). Molecular markers were examined from different fuels and
184 the limitations for quantification of the extremely complex samples were examined.

185

186 Text from lines 69 to 107 could be condensed into a few sentences. Earlier work relevant to
187 this paper seems to be mentioned in lines 108 to 145 and needs to be highlighted, front and
188 center.

189 This has been rectified through the new introduction paragraph.

190 Another point that could be highlighted is that S/IVOC emissions are poorly, if at all,
191 represented in emissions inventories and chemical transport models and their impacts on
192 atmospheric chemistry and air quality are uncertain (with particular relevance to regions
193 where this and similar fuel use is dominant, e.g., Asia).

194 This is now highlighted in the first paragraph of the introduction as:

195 I/SVOC emissions are poorly, if at all, represented in regional inventories and chemical
196 transport models.

197 2. Given the large variability seen in biomass burning emissions, say relative to internal
198 combustion engines, the authors should comment on the single experiments done for most
199 of the fuels. This could be done by analyzing the experiment-to-experiment variability for the
200 fuels where multiple experiments were done (i.e., cow dung cake, waste), as well as through
201 a review of similar literature.

202 This is a great comment. We now acknowledge this in the text.

203 Figure 8 shows that there was large sample to sample variability in emission factors for
204 different fuel wood samples, for which only 1 sample was taken. For this reason, emission
205 factors have been generalised for use in budget estimates to the type of fuel. Mean emission
206 factors are provided for measurements from samples of 17 fuel woods, 3 crop residues, 3 cow
207 dung cakes and 3 different collections of municipal solid waste. Despite this, for LPG and
208 charcoal samples only 1 sample was measured, and this significantly increases the uncertainty
209 in the PAH emission factors from these fuel sources.

210 We also look at this in some more detail in the companion paper, acp 2020-892, but in a more
211 quantitative manner, presenting total non-methane volatile organic compound emission
212 factors and looking at over 50 different combustion experiments of fuelwoods This is better
213 presented in this companion paper as there is a larger dataset of emissions.

214 In addition, they should also comment on the differences in combustion encountered in their
215 setup versus a real-world application. For example, most municipal solid waste is probably
216 burned in a high-temperature incinerator where the combustion chemistry might be very
217 different than the combustion simulated in this work. I do understand that 'backyard' low-
218 temperature MSW fires are a major concern in India, including in Delhi.

219 This is now acknowledged in the text as shown below.

220 This study was conducted under controlled laboratory conditions. For some sample types,
221 such as municipal solid waste, the laboratory measurement may not be entirely reflective of
222 real-world conditions. Municipal solid waste combustion may occur under both flaming and

223 smouldering conditions at landfill sites and in backyards, as well as in high-temperature
224 incinerators in more developed countries. All of these are likely to have quite different
225 combustion chemistry, and consequently lead to varying levels of emission.

226 3. I commend the authors on putting together this fantastic dataset of speciation and
227 emission factors and I am fairly certain that this will serve as a comprehensive resource for
228 years to come (from studying exposure to toxic pollutants to developing accurate emissions
229 inventories for air quality modeling). [This must be an oversight but I did not see a 'data
230 availability' section that describes how and where the data will be archived for others to use].
231 However, the manuscript seems to present only a 'snapshot' of the dataset, with a mix of
232 higher-level observations and depth for only a subset of speciated organic compounds (e.g.,
233 PAHs). Correct me if I am wrong but there is so much more to the dataset than what is
234 presented. If that is indeed the case, what I would have liked to see is a structured vision for
235 how the data plans to be analyzed further (e.g., detailed source profiles, molecular markers
236 for source identification, volatility distributions) and what open, pressing questions would this
237 dataset help answer in the long run?

238 We have added a table of peaks measured on both SPE disks and PTFE filters to the SI. We
239 also now have the companion paper acp 2020-892, which gives speciated emission factors
240 from these burns and provides quantitative emission factors in the supplementary
241 information. We will provide a short reference within this paper to acp 2020-892 to
242 emphasise this.

243 We have also submitted a paper to another journal which provides comprehensive organic
244 emission profiles from 5 different instruments. This analyses the volatility distribution of
245 organic emissions from C₂-C₄₀. The paper also provides additional insight into the new
246 datasets by looking at the secondary organic aerosol production potential, OH reactivity and
247 toxicity of emissions from domestic fuels in Delhi, India.

248 In addition, the dataset (provided in acp 2020-892) will also be used to better constrain
249 NMVOC emissions through formation of local and regional emission inventories.

250

251 Minor comments: 1. Line 228: Explain what 'NIST library hit was >800' means.

252 This has now been explained in the text.

253 Peaks were assigned through comparison of retention times with known standards and
254 comparison with the National Institute of Standards and Technology (NIST) mass spectral
255 library. Peaks with no genuine standard available were tentatively identified if the NIST library
256 similarity was > 700. This provides an indication of how similar the mass spectra obtained was
257 to the database mass spectra for the peak, with more details given in Stein, (2011). Peaks with
258 a hit > 900 reflect an excellent match, 800-900 a good match and 700-800 a fair match (Stein,
259 2011). The uncertainty in this approach has been shown to be low for peaks of hits > 800, with
260 the probability of incorrect identification being around 30 % for hits between 800-900 and 14
261 % for matches above 900 (Worton et al., 2017).

262 2. Line 471-485: Clarifying questions. Is the low fraction of the speciation of the organic
263 aerosol limited to not finding a match in the NIST or does it highlight a problem with the
264 analytical method? In addition, how sensitive is the fraction speciated to the use of the filter
265 media, i.e., better with PTFE versus quartz?

266 The low fraction of speciation of organic aerosol is due to lack of genuine standards available
267 to develop detector-response curves for the mass spectrometer. Many peaks have a mass
268 spectral database hit, but this does not impact the percentage identified here.

269 Percentage identification should not be influenced by the filter sampling media. This is low in
270 complex samples due to lack of genuine standards to allow quantitative detector-response
271 curves for individual analytes to be developed. The accelerated solvent extraction method
272 used here may miss some of the most polar water soluble species as they are not soluble in
273 EtOAc.

274 3. The composition section could benefit from findings from some recent publications that
275 have studied SOA from biomass burning emissions or precursors, e.g., He et al. (ESPI, 2020) –
276 alkylfuran mixture, Joo et al., (ESC, 2019) – 3-methylfuran, Ahern et al. (JGR, 2019), Akherati
277 et al. (ES&T, 2020), and Lim et al. (ACP, 2019) – biomass burning SOA in laboratory
278 experiments with an emphasis on understanding phenolic, furanic, and monoterpene VOC
279 contributions to SOA.

280 This paper now includes references to the above papers which focus on SOA formation from
281 biomass burning emissions. Some have been included in the adjusted introduction and some
282 in the composition section.

283 Introduction:

284 Ahern et al. (2019) showed that for burning of biomass needles, biogenic VOCs were the
285 dominant class of SOA precursor.

286 SOA formation from biomass burning has been shown to be significant in laboratory studies,
287 with SOA yields from the burning of western U.S. fuels reported to be 24 ± 4 % after 6 h and
288 56 ± 9 % after 4 d (Lim et al., 2019).

289 Main body:

290 SOA formation from furanic species remains poorly understood, with a recent study showing
291 an SOA yield of 1.6-2.4 % during the oxidation of 3-methylfuran with the nitrate radical (Joo
292 et al., 2019).

293 A recent study found that, oxygenated aromatic compounds, which included phenols and
294 methoxyphenols, were responsible for just under 60 % of the SOA formed from western U.S.
295 fuels (Akherati et al., 2020).

296 4. Figure 7: Was total organic mass in the gas- and particle phase measured another way, e.g.,
297 FID-gas, Sunset OC/EC-particle, to get mass closure?

298 Organic mass in the gas phase was measured with two separate gas chromatographs and a
299 proton-transfer reaction mass spectrometer, with the data treated in the companion paper
300 acp 2020-892. No direct-FID measurements of total hydrocarbon were attempted, in part due
301 to the difficulty of dealing with the change in sensitivity of the FID for hydrocarbons and
302 OVOCs which constitute a large fraction of the gas phase VOCs. Organic mass balance is
303 treated in a subsequent publication which maps emissions onto a volatility-basis dataset and
304 has been submitted for review elsewhere. However, OC/EC was not measured on these PM
305 samples due to the small sample size.

306 5. Figure 8: Mention sample size for each fuel. Specify measurement uncertainty when $n=1$.
307 Combine measurement uncertainty and experiment-to-experiment variability when $n>1$.

308 This point has been addressed in major point 2.

309 Table 1 shows the sample size for each fuel. Figure 8 shows that there was large sample to
310 sample variability in emission factors for different wood samples, for which only 1 sample was
311 taken. For this reason, emission factors have been generalised for use in budget estimates to
312 the type of fuel. Mean emission factors have been provided for measurements from samples
313 of 17 fuel woods, 3 crop residues, 3 cow dung cakes and 3 different collections of municipal
314 solid waste. The mean values and standard deviations of measured emission factors are as
315 follows: fuelwood ($247 \pm 214 \text{ mg kg}^{-1}$), crop residue ($747 \pm 518 \text{ mg kg}^{-1}$), MSW ($1022 \pm 340 \text{ mg}$
316 kg^{-1}) and cow dung cake ($615 \pm 112 \text{ mg kg}^{-1}$). Despite this, for LPG and charcoal samples only
317 1 sample was measured, and this significantly increases the uncertainty in the PAH emission
318 factors from these fuel sources. Experiment to experiment variability is provided in the
319 companion paper acp 2020-892.

320 6. Figure 9: Was the gas/particle partitioning of PAHs analyzed further? Seems like an ideal
321 dataset to study absorptive partitioning.

322 This has not been analysed as part of this work. A subsequent publication will provide a
323 volatility-basis dataset of emissions from the fuel types studied here. It should be possible to
324 dilute this to real-world dilutions, particulate matter concentrations and temperatures to
325 better understand, and represent, the gas/particle phase partitioning of emissions from
326 biomass burning.

327 7. Lines 631- 632: Why were certain samples fully speciated and others not very much? Was
328 this relationship examined further with respect to its sensitivity to fuel, total organic mass
329 captured on filter/disc, other variables?

330 Certain fuels were not fully speciated due to the low quantity of organic matter released and
331 the availability of standards to quantify these. For extremely complex samples, genuine
332 standards were not available to develop quantitative mass-spectrometer response curves to
333 these peaks and resulted in low levels of total quantification. Future suggestions are given in
334 the text of splitting the eluent between the mass spectrometer and a flame ionisation
335 detector to provide much better semi quantification.

336 **Marked up manuscript**

337 **Emissions of intermediate-volatility and semi-volatile organic compounds from**
338 **domestic fuels used in Delhi, India**

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353 **Abstract**

354 Biomass burning emits significant quantities of intermediate-volatility and semi-volatile
355 volatile organic compounds (I/SVOCs) in a complex mixture, probably containing many
356 thousands of chemical species. These components are significantly more toxic and have poorly
357 understood chemistry compared to volatile organic compounds routinely analysed/quantified in
358 ambient air, however quantification/analysis of I/SVOCs presents a difficult analytical
359 challenge.

360 The gases and particles emitted during the test combustion of a range of domestic solid fuels
361 collected from across ~~New~~-Delhi were sampled and analysed. Organic aerosol was collected
362 onto Teflon (PTFE) filters and residual low-volatility gases were adsorbed to the surface of
363 solid-phase extraction (SPE) disks. A new method relying on accelerated solvent extraction
364 (ASE) coupled to comprehensive two-dimensional gas chromatography with time-of-flight
365 mass spectrometry (GC×GC-ToF-MS) was developed. This highly sensitive and powerful
366 analytical technique enabled over 3000 peaks from I/SVOC species with unique mass spectra
367 to be detected. 15-100 % of gas-phase emissions and 7-100 % of particle-phase emissions were
368 characterised. The method was analysed for suitability to make quantitative measurements of
369 I/SVOCs using SPE disks. Analysis of SPE disks indicated phenolic and furanic compounds

370 were important to gas-phase I/SVOC emissions and levoglucosan to the aerosol phase. Gas-
371 and particle-phase emission factors for 21 polycyclic aromatic hydrocarbons (PAHs) were
372 derived, including 16 compounds listed by the US EPA as priority pollutants. Gas-phase
373 emissions were dominated by smaller PAHs. New emission factors were measured (mg kg^{-1})
374 for PAHs from combustion of cow dung cake (615), municipal solid waste (1022), crop residue
375 (747), sawdust (1236), fuel wood (247), charcoal (151) and ~~liquified~~liquefied petroleum gas
376 (56).

377 The results of this study indicate that cow dung cake and municipal solid waste burning are
378 likely to be significant PAH sources and further study is required to quantify their impact,
379 alongside emissions from fuel wood burning.

380 1. Introduction

381 Biomass burning is one of the most important global sources of trace gases and particles to the
382 atmosphere (Simoneit, 2002; Chen et al., 2017; Andreae, 2019). ~~Emissions of volatile organic
383 compounds (VOCs) and particulate matter (PM) are of interest due to their detrimental impact
384 on air quality. VOCs react to form ozone and secondary organic aerosol (SOA), and contribute
385 0.95 Tg yr⁻¹ of SOA yearly (Shrivastava et al., 2017). Estimates of VOCs from burning often
386 do not include many intermediate volatility and semi-volatile organic compounds (I/SVOCs).
387 Wildfires emit significant quantities of organic matter over regions such as the USA, the
388 Mediterranean, South East Asia and Australia, with residential solid fuel combustion and
389 wildfires emitting significant quantities of organic matter (Streets et al., 2003; Barboni et al.,
390 2010; Chen et al., 2017; Liu et al., 2017; Kiely et al., 2019). Emissions of volatile organic
391 compounds (VOCs) and particulate matter (PM) from biomass burning are of interest due to
392 their detrimental impact on air quality. VOCs react to form ozone and secondary organic
393 aerosol (SOA). Intermediate-volatility and semi-volatile organic compounds (I/SVOCs) are
394 also a significant emission from biomass burning (Stockwell et al., 2015; Koss et al., 2018).
395 I/SVOCs are an important class of air pollutant due to their contribution to aerosol formation
396 (Bruns et al., 2016; Lu et al., 2018). ~~and residential combustion leads to substantial organic
397 emissions in the developing world (Streets et al., 2003)~~I/SVOC emissions are poorly, if at all,
398 represented in regional inventories and chemical transport models. Consequently, their impact
399 to air quality in developing regions, where solid fuel combustion is a dominant fuel source, is
400 not well understood. Recent studies have shown that the inclusion of I/SVOCs leads to better
401 agreement between modelled and measured values (Ots et al., 2016; Woody et al., 2016; Jathar
402 et al., 2017; Murphy et al., 2017). Global I/SVOC emissions to the atmosphere from biomass~~

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403 burning were estimated to be ~ 54 Tg yr⁻¹ from 2005-2008 (Hodzic et al., 2016), with I/SVOCs
404 contributing in the range 8-15.5 Tg yr⁻¹ to SOA (Cubison et al., 2011; Hodzic et al., 2016).

405 SOA formation from biomass burning emissions is poorly understood globally. Important
406 factors include the formation of less volatile products from the oxidation of NMVOCs which
407 partition into the aerosol phase, heterogeneous oxidation of aerosol phase organics as well as
408 plume dilution followed by evaporation and further gas-phase oxidation (Lim et al., 2019).
409 Ahern et al. (2019) showed that for burning of biomass needles, biogenic VOCs were the
410 dominant class of SOA precursor. This study also found that for wiregrass, furans were the
411 most important SOA precursor. Bruns et al. (2016) showed that SOA formation from
412 combustion of beech fuel wood was dominated by 22 compounds, with phenol, naphthalene
413 and benzene contributing up to 80 % of the observed SOA. SOA formation from biomass
414 burning has been shown to be significant in laboratory studies, with SOA yields from the
415 burning of western U.S. fuels reported to be 24±4 % after 6 h and 56±9 % after 4 d (Lim et al.,
416 2019). However, the effect of atmospheric aging on I/SVOCs still remains poorly understood
417 (Liu et al., 2017; Decker et al., 2019; Sengupta et al., 2020).

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418 Better understanding of the quantity and composition of I/SVOCs from biomass burning is
419 needed to evaluate their impact on the atmosphere. This is a difficult analytical task, not well
420 suited to conventional analysis with gas chromatography coupled to mass spectrometry (GC-
421 MS). The reason for this is because of the exponential growth of potential isomers with carbon
422 number, which results in a large number of coeluting peaks (Goldstein and Galbally, 2007).
423 The high resolution of two-dimensional gas chromatography (GC×GC) has been demonstrated
424 as an ideal technique to overcome this issue when analysing complex organic samples in both
425 gas ~~I/SVOCs are an important class of air pollutant due to their contribution to aerosol~~
426 formation (Bruns et al., 2016; Lu et al., 2018). ~~I/VOCs have an effective saturation~~
427 concentration of 300-3,000,000 µg m⁻³ and are predominantly in the vapour phase. Once
428 oxidised their lower volatility products can partition into the aerosol phase (Donahue et al.,
429 2006). ~~SVOCs have effective saturation concentrations of 0.3-300 µg m⁻³ (Donahue et al.,~~
430 2012) and can partition between the gas and particle phases. Many studies have focused on
431 I/SVOCs emitted from a range of sources due to their impact on aerosol formation (Robinson
432 et al., 2007; Zhao et al., 2014; Cross et al., 2015; Zhao et al., 2015; Pereira et al., 2018)-
433 ~~I/SVOCs have been shown to contribute significantly to emissions from biomass burning~~
434 (Lewis et al., 2000; Stewart et al., 2020a) and particle phases (Hamilton et al., 2004; Lyu et al.,
435 2019c). The application of GC×GC to biomass burning emissions has shown hundreds of

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436 gaseous I/SVOCs using adsorption-thermal desorption cartridges (Hatch et al., 2015) or solid
437 phase extraction (SPE) disks (Hatch et al., 2018). GCxGC has also been used to analyse the
438 particle phase from samples collected onto PTFE or quartz filters (Hatch et al., 2018; Jen et al.,
439 2019), with the latter study quantifying 149 organic compounds which accounted for 4-37 %
440 of the total mass of organic carbon. The process used by Hatch et al. (2018) demonstrated high
441 recoveries of non-polar species from PTFE filters, with lower recoveries from SPE disks. This
442 study highlighted the need for further evaluation of samples collected onto PTFE filters and
443 SPE disks, ideally improving the method to remove undesirable steps such as
444 trimethylsilylation derivatisation, the use of pyridine and centrifuging which led to high
445 evaporative losses. The need to develop improved sampling and measurement techniques for
446 I/SVOCs has also been highlighted. This is because these species often do not transmit
447 quantitatively through the inlet and tubing when measured using online gas-phase techniques
448 (Pagonis et al., 2017).

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449 ~~Global I/SVOC emissions to the atmosphere from biomass burning were estimated to be ~54~~
450 ~~Tg yr⁻¹ from 2005-2008 (Hodzic et al., 2016) with I/SVOCs contributing in the range 8-15.5~~
451 ~~Tg yr⁻¹ to SOA (Cubison et al., 2011; Hodzic et al., 2016). SOA formation from combustion of~~
452 ~~beech fuel wood was shown to be dominated by 22 compounds, with phenol, naphthalene and~~
453 ~~benzene contributing up to 80 % of the observed SOA (Bruns et al., 2016). However, the effect~~
454 ~~of atmospheric aging on I/SVOCs still remains poorly understood (Liu et al., 2017; Decker~~
455 ~~et al., 2019; Sengupta et al., 2020).~~

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456 Residential combustion, agricultural crop residue burning, and open municipal solid waste
457 burning in the developing world are large, poorly characterised pollution sources with the
458 potential to have a significant impact on local and regional air quality, impacting human health
459 (Venkataraman et al., 2005; Jain et al., 2014; Wiedinmyer et al., 2014). Hazardous indoor air
460 pollution from combustion of solid fuels has been shown to be the most important factor from
461 a range of 67 environmental and lifestyle risk factors causing disease in South Asia (Lim et al.,
462 2012). ~~Recent studies focussed on source apportionment of ambient VOC concentrations in~~
463 ~~Delhi have shown ground-level concentrations to be predominantly traffic related, with smaller~~
464 ~~contributions from solid fuel combustion (Stewart et al., 2020a; Wang et al., 2020). Despite~~
465 ~~this, nearly 76 % of rural Indian households are dependent on solid biomass for their cooking~~
466 ~~needs (Gordon et al., 2018) with biofuels such as fuel wood, cow dung cake and crop residue~~
467 ~~being used. Combustion often takes place indoors without efficient emission controls which~~
468 ~~significantly increases the mean household concentration of pollutants, particularly particulate~~

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469 ~~matter with a diameter less than 2.5 μm ($\text{PM}_{2.5}$). Studies have shown mean 24 h concentrations~~
470 ~~of $\text{PM}_{2.5}$ in kitchens to be in excess of 500 $\mu\text{g m}^{-3}$ (Balakrishnan et al., 2013), with 4-8 times~~
471 ~~ambient concentration enhancement of polycyclic aromatic hydrocarbons (PAHs) close to the~~
472 ~~stove during cooking (Bhargava et al., 2004). This is significantly larger than the 40 $\mu\text{g m}^{-3}$~~
473 ~~Indian National Air Quality Standard. For comparison, the mean population-weighted $\text{PM}_{2.5}$~~
474 ~~level in Delhi, Chennai, Hyderabad and Mumbai from 2015-2018 was 72 $\mu\text{g m}^{-3}$ and the global~~
475 ~~mean 20 $\mu\text{g m}^{-3}$ (Chen et al., 2020), with various sources also leading to elevated levels of~~
476 ~~PAHs in cities like Delhi (Elzein et al., 2020). The health effects from this are significant, with~~
477 ~~premature deaths in India from exposure to ambient and household air pollution estimated to~~
478 ~~be over 2 million (Lallukka et al., 2017).~~

479 ~~Few detailed studies have been conducted examining the composition of I/SVOC emissions~~
480 ~~from sources relevant to South Asia. One study examined gas- and particle-phase emissions~~
481 ~~from coconut leaves, rice straw, cow dung cake, biomass briquettes and jackfruit branches in~~
482 ~~Bangladesh with samples analysed by ion chromatography (IC), organic/elemental carbon~~
483 ~~(OC/EC) and gas chromatography coupled to mass spectrometry (GC-MS) to produce~~
484 ~~emission factors and examine molecular markers (Sheesley et al., 2003). Another study~~
485 ~~examined emissions of $\text{PM}_{2.5}$, OC/EC, metals and organics. Despite this, nearly 76 % of rural~~
486 ~~Indian households depend on solid biomass for their cooking needs (Gordon et al., 2018), using~~
487 ~~biofuels such as fuel wood, cow dung cake and crop residue. Combustion often takes place~~
488 ~~indoors, without efficient emission controls, which significantly increases the mean household~~
489 ~~concentration of pollutants, particularly particulate matter with a diameter less than 2.5 μm~~
490 ~~($\text{PM}_{2.5}$). The health effects from this are significant, with an estimated 3.8 million premature~~
491 ~~deaths globally due to inefficient indoor combustion from cooking (WHO, 2018).~~

492 ~~Few detailed studies have been conducted examining the composition of I/SVOC emissions~~
493 ~~from solid-fuel combustion sources from South Asia. Sheesley et al. (2003) used solvent~~
494 ~~extraction followed by GC-MS to produce emission factors and examine molecular markers~~
495 ~~from combustion of coconut leaves, rice straw, cow dung cake, biomass briquettes and jackfruit~~
496 ~~branches collected from Bangladesh. A more recent study extracted $\text{PM}_{2.5}$ samples followed by~~
497 ~~analysis with GC-MS of samples collected from motorcycles, diesel- and gasoline-generators,~~
498 ~~agricultural pumps, municipal solid waste burning, cooking fires using fuel wood and cow~~
499 ~~dung cake, crop residue burning and brick kilns in Nepal (Jayarathne et al., 2018). Lack of~~
500 ~~knowledge regarding major pollution sources hinders our ability to predict air quality, but also~~
501 ~~the development of effective mitigation strategies for air pollution which leads to health~~

502 impacts ranging from respiratory illness to premature death (Brunekreef and Holgate, 2002).
503 This results in many people living with high levels of air pollution (Cohen et al., 2005;
504 Lelieveld et al., 2015) and 13 Indian cities ranking amongst the top 20 cities in the world with
505 the highest levels of ambient PM_{2.5} pollution, based on available data (Gordon et al., 2018).

506 ~~Early biomass burning studies used filters to target aerosol and sorbent tubes or polyurethane~~
507 ~~styrene-divinylbenzene (PUF/XAD/PUF) cartridges to sample gaseous species followed by~~
508 ~~solvent extraction and analysis by GC-MS (McDonald et al., 2000; Schauer et al., 2001; Hays~~
509 ~~et al., 2002; Sheesley et al., 2003; Jordan and Seen, 2005; Dhammapala et al., 2007; Mazzoleni~~
510 ~~et al., 2007; Pettersson et al., 2011; Singh et al., 2013). Detailed studies have focussed on~~
511 ~~quantifying the composition of the particulate matter from burning by extracting aerosol~~
512 ~~samples, followed by analysis by GC-MS (Fine et al., 2001; Oros and Simoneit, 2001b, a; Oros~~
513 ~~et al., 2006; Jayarathne et al., 2018). Many studies have been carried out to measure emission~~
514 ~~factors of PAHs from burning, such as detailed measurements of up to 133 PAHs (Samburova~~
515 ~~et al., 2016) and time-resolved PAH measurements (Eriksson et al., 2014). PAH emission~~
516 ~~factors have been measured for coal (Chen et al., 2005; Lee et al., 2005; Geng et al., 2014) oil~~
517 ~~(Rogge et al., 1997), fuel woods (McDonald et al., 2000; Simoneit, 2002; Hosseini et al., 2013;~~
518 ~~Geng et al., 2014; Jimenez et al., 2017), peat (Iinuma et al., 2007), tyres (Iinuma et al., 2007);~~
519 ~~domestic waste (Kakareka et al., 2005; Sidhu et al., 2005), cow dung cake (Gadi et al., 2012;~~
520 ~~Singh et al., 2013; Tiwari et al., 2013), sawdust briquette (Kim Oanh et al., 2002) and crop~~
521 ~~residue (Jenkins et al., 1996; Lu et al., 2009; Gadi et al., 2012; Singh et al., 2013; Wei et al.,~~
522 ~~2014; Kim Oanh et al., 2015; Wiriya et al., 2016). Measurements of I/SVOCs in both gas and~~
523 ~~particle phase samples using conventional GC-MS presents a difficult analytical challenge, due~~
524 ~~to the exponential growth of potential isomers with carbon number which can result in a large~~
525 ~~number of coeluting peaks (Goldstein and Galbally, 2007).~~

526 ~~The high resolution of two-dimensional gas chromatography (GCxGC) has been demonstrated~~
527 ~~as an ideal technique to overcome the issue of peak coelution in one-dimensional gas~~
528 ~~chromatography and has been used to analyse complex ambient samples in the gas (Lewis et~~
529 ~~al., 2000; Xu et al., 2003; Dunmore et al., 2015; Lyu et al., 2019a) and particle phases~~
530 ~~(Hamilton et al., 2004; Lyu et al., 2019b; Lyu et al., 2019c). GCxGC has recently shown~~
531 ~~hundreds of gaseous I/SVOCs released from biomass burning using adsorption thermal~~
532 ~~desorption cartridges or solid-phase extraction (SPE) disks (Hatch et al., 2015; Hatch et al.,~~
533 ~~2018). The particle phase has also been targeted by extracting samples from PTFE or quartz~~
534 ~~filters (Hatch et al., 2018; Jen et al., 2019), with the latter study quantifying 149 organic~~

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535 compounds which accounted for 4.37 % of the total mass of organic carbon. The process used
536 by Hatch et al. (2018) demonstrated high recoveries of non-polar species from PTFE filters,
537 with lower recoveries from SPE disks. This study highlighted the need for further evaluation
538 of samples collected onto PTFE filters and SPE disks, ideally improving the method to remove
539 undesirable steps such as trimethylsilylation derivatisation, the use of pyridine and centrifuging
540 which led to high evaporative losses. The need to develop improved sampling and
541 measurement techniques for I/SVOCs has been highlighted as these species often do not
542 transmit quantitatively through the inlet and tubing when measured using online gas phase
543 techniques (Pagonis et al., 2017).

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544 In this study we develop a more efficient extraction step for the SPE/PTFE technique, allowing
545 developed by Hatch et al. (2018), using accelerated solvent extraction into ethyl acetate, which
546 showed high recoveries of non-polar I/SVOCs collected from burning typical domestic,
547 Domestic fuels used in characteristic to Northern India. The technique is used to identify many
548 I/SVOCs in burning samples, were gathered and organic I/SVOC samples collected onto SPE
549 disks and PTFE filters from controlled laboratory combustion experiments of a variety of fuel
550 woods, cow dung cakes, municipal solid waste samples, crop residues, charcoal and LPG. The
551 samples were extracted using this new technique and analysed with GC×GC coupled to time-
552 of-flight mass spectrometry (GC×GC-ToF-MS). Molecular markers were examined from
553 different fuels and the limitations for quantification of I/SVOCs from burning and used to
554 develop emission factors for selected PAHs the extremely complex samples using a mass
555 spectrometer were examined.

556 2. Methods Sample

557 2.1 Fuel collection and burning facility

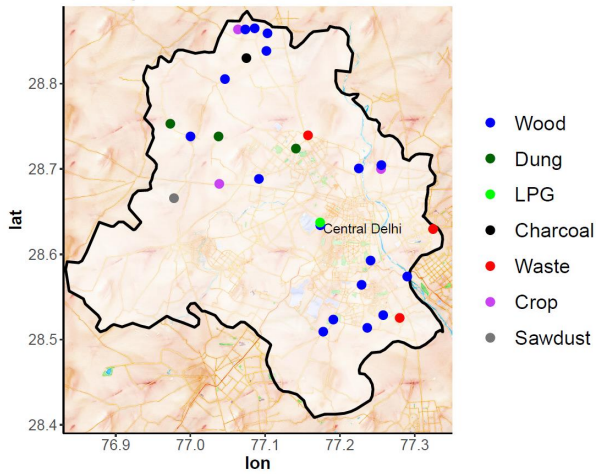
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558 The state of New-Delhi was gridded (0.05°05×0.05°05) and samples a diverse range of fuel
559 types collected from across the state (see Figure 2). Samples Fuels were stored in a manner akin
560 to local practices prior to combustion, to ensure that the moisture content of fuels were similar
561 to those burnt across the state. A range of solid biomass fuels were collected which included
562 17 fuel wood species, cow dung cake, charcoal and sawdust (see Table 1). Three crop residue
563 samples fuel types were collected and consisted of dried stems from vegetable plants such as
564 cabbage (*Brassica spp*) and aubergines (*Solanum melongena*) as well as coconut husk (*Cocos*
565 *nucifera*). Municipal solid waste samples were was collected from Bhalaswa, Ghazipur and

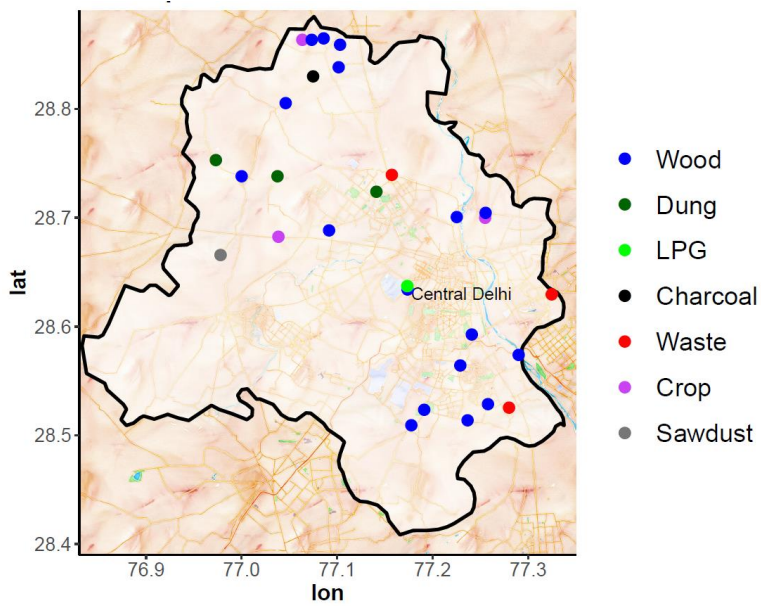
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566 Okhla landfill sites. A low-cost liquefied petroleum gas (LPG) stove was also purchased to
567 allow direct comparison to other combustion sources.

568 Samples/Fuels were burnt at the CSIR-National Physical Laboratory (NPL) New Delhi under
569 controlled conditions using a combustion dilution chamber that has been well described
570 previously (Venkataraman et al., 2002; Saud et al., 2011; Saud et al., 2012; Singh et al., 2013).
571 In summary, 200 g of dry fuel was rapidly heated to spontaneous ignition with emissions driven
572 into a hood and up a flue by convection to allow enough dilution, cooling and residence time
573 to achieve the quenching of typical indoor environments. This process was designed to
574 replicate the immediate condensational processes that occur in smoke particles approximately
575 5-20 mins after emission, yet prior to photochemistry which may change composition (Akagi
576 et al., 2011). A low volume sampler (Vayubodhan Pvt.Ltd) was used to collect particulates and
577 low volatility gases passing from the top of the flue through a chamber with a flow rate of 46.7
578 L min⁻¹. As detailed in Table 1, ~~30 samples from a range of fuel types were burnt, and 8 blank~~
579 ~~samples were collected~~ from 30 fuels alongside 8 blank measurements (see the Supplementary
580 Information S1 for an example burn and filter ~~samples collected from different~~
581 ~~sources)-sample~~). Prior to sample collection, SPE disks (Resprep, C₁₈, 47 mm) were prewashed
582 with 2 ~~×~~ 5 mL acetone (Fisher Scientific analytical reagent grade), and 1 ~~×~~ 5 mL methanol
583 (Sigma-Aldrich HPLC grade), then packed in foil and sealed in airtight bags. Samples were
584 collected onto a PTFE filter (Cole-Parmer, 47 mm, 1.2 µm pore size) placed on top of an SPE
585 disk in a filter holder (Cole-Parmer, 47 mm, PFA) for 30 mins at a flow rate of 6 L min⁻¹,
586 maintained by a mass flow controller (Alicat 0-20 SLM) connected to a pump. Samples were
587 removed from the filter holder immediately after the experiment ~~and~~, wrapped in foil, placed
588 inside into an airtight bag and stored at - 20 °C. Samples were ~~then~~ transported to the UK for
589 analysis using an insulated container containing dry ice via. air freight and stored at - 20 °C
590 for around 2 months prior to analysis.



591



592

593 Figure 2. Locations across New Delhi used for the local surveys into fuel use and sample
 594 collection. Map produced using tiles from Stamen maps.

595 Table 1. Locations across Delhi used for the local surveys into fuel usage and collection. Map tiles by
 596 Stamen Design. Data by © OpenStreetMap contributors 2020. Distributed under a Creative Commons
 597 BY-SA License.

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599 **Table 1.** Types of ~~sample collected~~ **fuel sampled** where n = number of ~~samples burned~~ **burns of a specific**
 600 **fuel type**, SPE and PTFE = number of blank corrected peaks detected on SPE disks and PTFE filters,
 601 respectively.

Fuel woods	n	SPE	PTFE	Other	n	SPE	PTFE
Plywood	1	149 201	530 516	Cow dung cake	3	129 51235	1604 1562
<i>Azadirachta indica</i>	1	562 557	880 862	<i>Cocos nucifera</i>	1	614 620	1197 1182
<i>Morus spp</i>	1	811 805	1108 1132	Charcoal	1	453 439	211 280
<i>Shorea spp</i>	1	283 296	326 360	Sawdust	1	1113 1112	1417 1486
<i>Ficus religiosa</i>	1	504 500	652 712	Waste	3	980 948	1182 1184
<i>Syzygium spp</i>	1	680 661	529 571	LPG	1	-	0 -
<i>Ficus spp</i>	1	277 306	247 292	Blank	8	-	-
<i>Vachellia spp</i>	1	702 697	753 800	Cow dung cake mix	1	932 931	1200 1241
<i>Dalbergia sissou</i>	1	483 501	561 611	<i>Brassica spp</i>	1	656 652	463 536
<i>Ricinus spp</i>	1	424	125 271	<i>Solanum melongena</i>	1	280 314	551 559
<i>Holoptelea spp</i>	1	276 274	263 324				
<i>Saraca indica</i>	1	517 525	445 484				
<i>Pithecellobium spp</i>	1	527 525	159 235				
<i>Eucalyptus spp</i>	1	211 238	771 144				
<i>Melia azedarach</i>	1	434 444	166 213				
<i>Prosopis spp</i>	1	237 248	113 144				
<i>Mangifera indica</i>	1	360 387	546 628				

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603 **Sample extraction**

604 **2.2 Extraction**

605 SPE disks and PTFE filters were spiked with an internal standard ($50 \mu\text{L}$ at $20 \mu\text{g mL}^{-1}$)
 606 containing 6 deuterated PAHs (1,4-Dichlorobenzene- d_4 , naphthalene- d_8 , acenaphthene- d_{10} ,
 607 phenanthrene- d_{10} , chrysene- d_{12} , perylene- d_{12} ; EPA 8270 Semivolatile Internal Standard Mix,
 608 $2000 \mu\text{g mL}^{-1}$ in DCM) to result in a final internal standard concentration of $1 \mu\text{g mL}^{-1}$ in
 609 solution. The solvent from the internal standard was allowed to evaporate and then SPE disks
 610 and PTFE filters were cut and extracted into **ethyl acetate (EtOAc)** using accelerated solvent
 611 extraction (ASE 350, Dionex, ThermoFisher Scientific). Extractions were performed at $80 \text{ }^\circ\text{C}$
 612 and 1500 psi for three 5 min cycles. After each cycle, the cell was purged for 60 seconds into
 613 a sample collection vial. Samples were then reduced from 15 mL to 0.90 mL over a low flow
 614 of N_2 in an ice bath over a period of 6-8 hours (Farren et al., 2015). Samples were then pipetted
 615 (glass Pasteur) to sample vials (Sigma-Aldrich, amber glass, 1.5 mL), with ASE vials rinsed
 616 with $2 \times 50 \mu\text{L}$ washes of EtOAc, then added to the sample vial and sealed (Agilent 12 mm
 617 cap, PTFE/silicone/PTFE). The mass of the sample vial and cap for each sample was measured
 618 before and after to determine the exact volume of solvent in each sample. Extracts were frozen
 619 prior to analysis to reduce evaporative losses.

2.3 Organic Methods for organic composition analysis

GC×GC×GC-ToF-MS: PTFE samples were analysed using GC×GC×GC-ToF-MS (Leco Pegasus BT 4D) using a splitless injection (1 µL injection, 4mm taper focus liner, SHG 560302). The primary dimension column was a RXI-5SilMS (Restek, 30 m × 0.25 µm × 0.25 mm) connected to a second column of RXI-17SilMS (Restek, 0.25 µm × 0.25 mm, 0.17 m primary GC oven, 0.1 m modulator, 1.42 m secondary oven, 0.31 m transfer line) with a He flow of 1.4 mL min⁻¹. The primary oven was held at 40 °C for 1 min then ramped at 3 °C min⁻¹ to 322 °C where it was held for 3 min. The secondary oven was held at 62 °C for 1 min then ramped at 3.2 °C min⁻¹ to 190 °C after which it was ramped at 3.6 °C min⁻¹ to 325 °C and held for 19.5 mins. The inlet was held at 280 °C and the transfer line at 340 °C. A 5 s cryogenic modulation was used with a 1.5 s hot pulse and 1 s cool time between stages. Using two separate wash vials, the syringe (10 µL Gerstel) was cleaned prior to injection with two cycles of 3 × 5 µL washes in EtOAc and rinsed post injection with two cycles of 2 × 5 µL washes in EtOAc. Samples with high concentrations of levoglucosan were reanalysed using a faster method, injected split (75:1 and 125:1) with the primary oven held at 40 °C for 1 min, then ramped at 10 °C min⁻¹ to 220 °C. The secondary oven was held at 62 °C for 1 min and then ramped at 10 °C min⁻¹ to 245 °C.

SPE samples were injected split (10:1) and analysed with a shorter analysis time with the primary oven held at 40 °C for 1 min then ramped at 3 °C min⁻¹ to 202 °C where it was held for 4 seconds. The secondary oven was held at 62 °C for 1 min then ramped at 3.2 °C min⁻¹ to 235 °C. A 75:1 split injection was used for quantitation of concentrations outside of the detector response range for ~~furans~~ furanics, phenolics, benzaldehydes, naphthalenes and benzonitrile. Peaks were assigned through comparison of retention times with known standards and comparison with the National Institute of Standards and Technology (NIST) mass spectral library. Peaks with no genuine standard available were tentatively identified if the NIST library ~~hit was > 800. The uncertainty in this approach has been shown to be low~~ similarity was > 700. This provides an indication of how similar the mass spectra obtained was to the database mass spectra for the peak, with more details given in Stein, (2011). Peaks with a hit > 900 reflect an excellent match, 800-900 a good match and 700-800 a fair match (Stein, 2011). The uncertainty in this approach has been shown to be low for peaks of hits > 800, with the probability of incorrect identification being around 30 % for hits between 800-900 and 14 % for matches above 900 (Worton et al., 2017). Integration was carried out within the ChromaTOF 5.0 software package (Leko, 2019). Calibration was performed using a 6-point calibration using

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653 either a linear or second-order polynomial fit covering the ranges 0.1-2.5 $\mu\text{g ml}^{-1}$ (splitless),
654 0.5-15 $\mu\text{g ml}^{-1}$ (10:1 split), 15-400 $\mu\text{g ml}^{-1}$ (75:1 split) and 400-800 $\mu\text{g ml}^{-1}$ (125:1 split). Eight
655 blank measurements were made at the beginning and end of the day by passing air from the
656 chamber (6 L min^{-1} for 30 mins) through the filter holder containing PTFE filters and SPE disks
657 (see the Supplementary Information S2 for examples of blank chromatograms). Blank
658 corrections were applied by calculating the average blank value for each compound using blank
659 samples collected using the same sample collection parameters as real samples before and after
660 the relevant burning experiments.

661 PTR-ToF-MS: Online measurements of naphthalene, methylnaphthalenes and
662 dimethylnaphthalenes were made using a proton transfer reaction-time of flight-mass
663 spectrometer PTR-ToF-MS (PTR 8000; Ionicon Analytik, Innsbruck) and assigned as masses
664 129.058, 143.08 and 157.097, respectively. Additional details of the PTR-ToF-MS from
665 Physical Research Laboratory (PRL), Ahmedabad used in this study are given in previous
666 papers (Sahu and Saxena, 2015; Sahu et al., 2016). A $\frac{1}{4}$ inch OD PFA sample line ran from the
667 top of the flue to the instrument which was housed in an air-conditioned laboratory with a
668 sample flow rate of 4.3 L min^{-1} . The sample air was diluted either 5 or 6.25 times into zero air,
669 generated by passing ambient air (1 L min^{-1}) through a heated platinum filament at 550 $^{\circ}\text{C}$,
670 before entering the instrument with an inlet flow of 250 ml min^{-1} . The instrument was operated
671 with a reduced electric field strength (E/N , where N is the buffer gas density and E is the electric
672 field strength) of 120 Td. The drift tube temperature was 60 $^{\circ}\text{C}$ with a pressure of 2.3 mbar and
673 560 V applied across it.

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

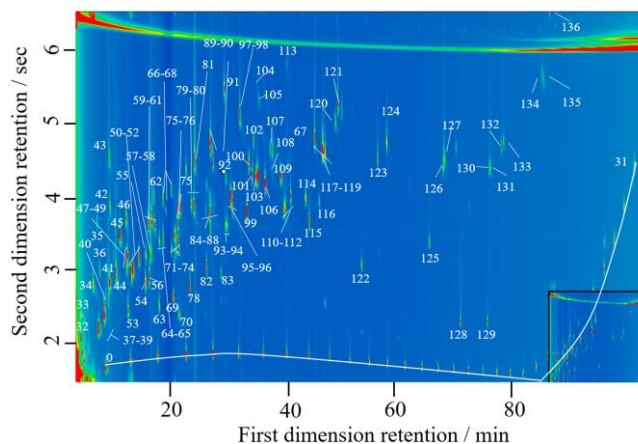
682 Mass calibration and peak fitting of the PTR-ToF-MS data were performed using PTRwid
683 software (Holzinger, 2015). Count rates (cps) of each mass spectral peak were normalised to
684 the primary ion (H_3O^+) and water cluster ($\text{H}_3\text{O}\cdot\text{H}_2\text{O}^+$) peaks and mixing ratios were then

685 determined for each mass using the normalised sensitivity (ncps). Where compounds known to
686 fragment in the PTR-ToF-MS were identified, the mixing ratio of these species was calculated
687 by summing parent ion and fragment ion mixing ratios. Before each burning study ambient air
688 was sampled to provide a background for the measurement.

689 **2.4 Quantification of recovery and breakthrough**

690 Standards were used for 136 species (see [Figure 2](#) [the Supplementary Information S3](#))
691 including two commercially available standard mixes containing 33 alkanes (C₇-C₄₀ saturated
692 alkane standard, certified 1000 µg m⁻¹ in hexane, Sigma Aldrich 49452-U) and 64 semi
693 volatiles (EPA CLP Semivolatile Calibration Mix, 1000 µg mL⁻¹ in DCM:benzene 3:1, Sigma
694 Aldrich 506508). Further standards were produced in-house, by dissolving high quality
695 standards (> 99 % purity), for a range of additional species also found in samples including
696 nitrogen containing VOCs ~~(NVOCs)~~, ~~furans~~, ~~furans~~, alkyl-substituted monoaromatics,
697 oxygenated aromatics, ketones, aldehydes, methoxy phenols, aromatic acids, PAHs and
698 levoglucosan ~~(see Table 2)~~. Stock solutions of around 1000 µg mL⁻¹ were prepared by
699 dissolving 0.01 g into 10 mL EtOAc. Polar components, such as levoglucosan, were dissolved
700 into [methanol \(MeOH\)](#) for stock solutions and those not soluble at room temperature were
701 heated and pipetted using hot pipette tips to make quantitative dilutions.

702 Six separate PTFE filters and SPE disks were spiked with the standard solution containing 136
703 compounds (50 µL at 20 µg mL⁻¹), extracted and analysed. Recovery levels were calculated by
704 comparing the signal to direct injection of the diluted standards to the ~~GC×GC×GC~~-ToF-
705 MS. The recoveries are shown in [Table 2](#) [the Supplementary Information S3](#). SPE disks
706 showed poor recoveries (S_{rec}) of *n*-nonane to *n*-tridecane and C₂ substituted monoaromatics,
707 likely due to volatilisation of these more-volatile components. Poorer recoveries were also
708 observed of nitroanilines and levoglucosan. Non-polar species showed good recoveries, with
709 high recoveries of C₁₄-C₂₀ alkanes, ~~furans~~ ~~furans~~, phenols, chlorobenzenes and PAHs. PTFE
710 filters demonstrated high recoveries (P_{rec}) of PAHs with more than three rings in their structure
711 (81.6-100 %). Recoveries were low, or zero, for volatile components with boiling points < 200
712 °C, indicating no retention, which is consistent with the method being well-suited to target the
713 aerosol phase. The recoveries of non-polar species ~~in~~ [into](#) EtOAc from SPE disks were higher
714 than those reported into MeOH (Hatch et al., 2018).



715
716 **Figure 2.** GCxGC ToF MS chromatogram of a mixed standard, numbered according to species listed
717 **in Table 2.**

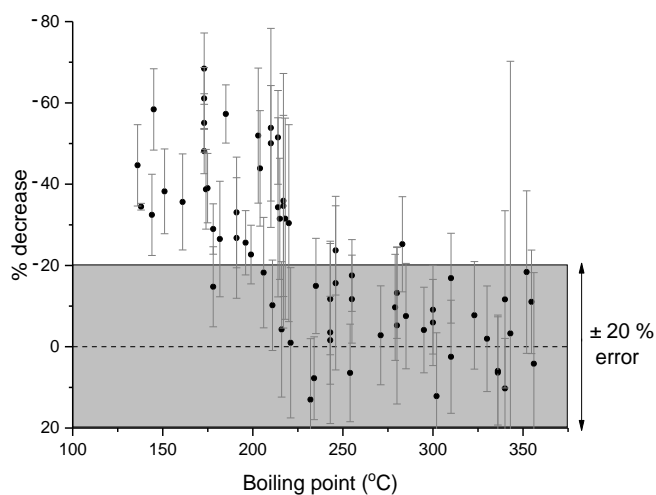
718 **Table 2.** Species used in calibration where Comp.No. refers to the peak number in Figure 2, Q_{ms} = split
719 method used for SPE quantitation, Q_{mp} = split method used for PTFE quantitation, S = splitless method,
720 S_{rec} = % recovery SPE, P_{rec} = % recovery PTFE, ^a = Sigma Aldrich *n* alkanes standard, ^b = Sigma
721 Aldrich semivolatiles standard, ^c = Sigma Aldrich deuterated internal standard, ^d = in-house solution
722 and — = not measured either due to being outside of SPE method range or due to volatilisation from
723 PTFE filters. Slight over-recoveries of > 100 % are reported as 100 % and accounted for in blank
724 subtractions.

725

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	<i>n</i> -Nonane ^a	10:1	S	60.0	-	32	Pyridine ^d	10:1	S	75.1	-
1	<i>n</i> -Decane ^a	10:1	S	77.6	19.5	33	<i>n</i> -Nitrosodimethylamine ^b	10:1	S	-	-
2	<i>n</i> -Undecane ^a	10:1	S	100	57.2	44	2,3-lutidine ^d	10:1	S	99.4	-
3	<i>n</i> -Dodecane ^a	10:1	S	85.7	22.0	46	Benzonitrile ^d	75:1	S	86.9	-
4	<i>n</i> -Tridecane ^a	10:1	S	91.4	75.0	57	<i>n</i> -Nitrosodipropylamine ^b	10:1	S	100	-
5	<i>n</i> -Tetradecane ^a	10:1	S	97.8	97.8	62	Nitrobenzene ^b	10:1	S	88.5	-
6	<i>n</i> -Pentadecane ^a	10:1	S	99.7	92.3	67	2-Nitrophenol ^b	10:1	S	100	-
7	<i>n</i> -Hexadecane ^a	10:1	S	100	100	68	Pyrrole-2-carbonitrile ^d	10:1	S	-	-
8	<i>n</i> -Heptadecane ^a	10:1	S	100	98.0	77	4-chloroaniline ^b	10:1	S	7.78	-
9	<i>n</i> -Octadecane ^a	10:1	S	100	99.9	98	2-Nitroaniline ^b	10:1	S	100	-
10	<i>n</i> -Nonadecane ^a	10:1	S	100	98.9	102	2,6-dinitrotoluene ^b	10:1	S	99.9	-
11	<i>n</i> -Eicosane ^a	10:1	S	100	96.8	105	3-Nitroaniline ^b	10:1	S	34.2	-
12	<i>n</i> -Heneicosane ^a	10:1	S	-	100	107	2,4-Dinitrotoluene ^b	10:1	S	100	-
13-23	<i>n</i> -Docosane ^a — <i>n</i> -Dotriacontane ^a	10:1	S	-	100	108	4-Nitrophenol ^b	10:1	S	-	-
24	<i>n</i> -Tritriacontane	-	-	-	96.5	112	Azobenzene ^b	10:1	-	100	100
25	<i>n</i> -Tetratriacontane	-	-	-	78.9	113	<i>p</i> -Nitroaniline ^b	10:1	S	64.5	-
26	<i>n</i> -Pentatriacontane	-	-	-	58.3	121	Caffeine ^d	10:1	S	-	-
27	<i>n</i> -Hexatriacontane	-	-	-	49.9	Aromatics					
28	<i>n</i> -Heptatriacontane	-	-	-	35.4	37	Ethylbenzene ^d	10:1	S	44.6	-
29	<i>n</i> -Octatriacontane	-	-	-	32.1	38	<i>m</i> -Xylene ^d	10:1	S	34.5	-
30	<i>n</i> -Nonatriacontane	-	-	-	29.1	39	<i>o</i> -Xylene ^d	10:1	S	32.4	-
31	<i>n</i> -Tetracontane	-	-	-	27.9	40	Styrene ^d	10:1	S	58.4	-

PAH				69	Pentylbenzene ^d	40:1	S	99.0	24.4		
76	Naphthalene ^{b,c}	75:1	S	93.9	37.1	82	Pentamethylbenzene ^d	40:1	S	68.6	39.5
81	Quinoline ^d	40:1	S	28.6	-	Halogenated					
87	2-Methylnaphthalene ^b	75:1	S	90.8	72.4	48	2-Chlorophenol ^b	40:1	S	100	-
89	Indole ^d	40:1	S	81.6	-	50	1,3-Dichlorobenzene ^b	40:1	S	85.5	-
90	Azulene ^d	40:1	S	38.5	-	51	1,4-Dichlorobenzene ^{b,c}	40:1	S	87.2	-
91	1(3H)- Isobenzofuranone ^d	40:1	S	100	-	52	1,2-Dichlorobenzene ^b	40:1	S	70.3	-
96	Biphenyl ^d	40:1	S	99.5	75.0	56	Hexachloroethane ^b	40:1	S	83.7	-
97	1,4-Naphthoquinone ^d	40:1	S	100	-	74	2,4-Dichlorophenol ^b	40:1	S	100	83.9
99	2,3- Dimethylnaphthalene ^d	40:1	S	100	-	75	1,2,4-trichlorobenzene ^b	40:1	S	85.6	-
100	Acenaphthylene ^b	40:1	S	98.5	84.1	78	Hexachlorobutadiene ^b	40:1	S	61.6	-
103	Acenaphthene ^{b,c}	40:1	S	100	88.2	83	Hexachlorocyclopentadiene ^b	40:1	S	100	-
106	Dibenzofuran ^b	40:1	S	100	86.4	88	4-Chloro-3-methylphenol ^b	S	90.8	-	
109	Fluorene ^b	40:1	S	100	86.0	93	2,4,6-Trichlorophenol ^b	40:1	S	95.8	-
117	9H-Fluoren-9-one ^d	40:1	S	100	100	94	2,4,5-Trichlorophenol ^b	40:1	S	100	-
118	Phenanthrene ^b	40:1	S	100	96.7	95	2-Chloronaphthalene ^b	40:1	S	99.6	-
119	Anthracene ^b	40:1	S	98.6	95.9	110	4-Chlorophenylphenylether ^b	40:1	S	100	-
120	Carbazole ^b	40:1	S	100	85.2	114	4-Bromophenylphenylether ^b	40:1	S	100	-
123	Fluoranthene ^b	40:1	S	100	97.2	115	Hexachlorobenzene ^b	40:1	S	100	-
124	Pyrene ^b	40:1	S	-	100	116	Pentachlorophenol ^b	40:1	S	100	-
126	Benzo(a)anthracene ^b	-	S	-	100	Furans					
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	40:1	S	54.9	-
131	Benzo(k)fluoranthene ^b	-	S	-	100	36	α -Angelica lactone ^d	40:1	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	40:1	S	100	-
135	Dibenz(A,H)anthracene ^b	-	S	-	92.9	111	Diethyl-phthalate ^b	40:1	S	100	-
136	Benzo(G,H,I)perylene ^b	-	S	-	96.6	122	Di-n-butyl-phthalate ^b	40:1	S	-	-
Oxygenated aromatics						125	Benzyl-butyl-phthalate ^b	-	S	-	92.0
41	Anisole ^d	40:1	S	20.4	-	128	Bis(2-ethylhexyl)phthalate ^b	-	S	-	97.4
42	p-Benzoquinone ^d	40:1	S	94.8	-	129	Di-n-octyl-phthalate ^b	-	S	-	90.6
45	Benzaldehyde ^d	40:1	S	82.8	-	Others					
47	Phenol ^b	75:1	S	100	-	49	Bis(2-chloroethyl)ether ^b	40:1	S	84.5	-
55	<i>o</i> -Cresol ^b	40:1	S	100	-	53	2-Octanone ^d	40:1	S	97.0	-
58	<i>p</i> -Cresol ^b	75:1	S	100	-	54	Bis(2-chloro-1- methyl)ethyl ether ^b	40:1	S	100	-
59	3-Methylbenzaldehyde ^d	40:1	S	99.9	-	63	Nonanal ^d	40:1	S	100	52.3
60	2-Methylbenzaldehyde ^d	75:1	S	100	-	65	Isophorone ^b	40:1	S	96.4	-
61	2-Methoxyphenol ^d	75:1	S	100	-	70	1-nonanol ^d	40:1	S	98.6	-
64	2,6-Dimethylphenol ^d	75:1	S	100	100	72	Bis(2- chloroethoxy)methane ^b	40:1	S	100	-
66	2,3-dimethyl-2,5- eyclohexadiene-1,4- dione ^d	40:1	S	100	-	84	Pinane-diol ^d	40:1	S	-	-
71	2,4-dimethylphenol ^b	40:1	S	89.5	-	104	Levogluconan ^d	40:1	S	0	70.0
73	Benzoic acid ^d	40:1	S	-	-						
79	Mequinol ^d	40:1	S	60.4	-						
80	<i>m</i> -Guaiacol ^d	40:1	S	44.0	-						
85	Hydroquinone ^d	40:1	S	24.8	-						
86	Resorcinol ^d	40:1	S	76.0	-						
92	2,6-Dimethoxyphenol ^d	40:1	S	93.6	-						

727 To quantify the additional effect of breakthrough during sampling, tests were conducted for
728 SPE disks to examine the retention of components adsorbed to their surface when subject to an
729 air flow equivalent to the sample volume. SPE disks were spiked with the calibration mixture
730 containing 96 compounds of interest ($50 \mu\text{L}$ at $20 \mu\text{g mL}^{-1}$, $n = 4$) and subject to a purified air
731 flow of 6 L min^{-1} for 30 mins. The samples were extracted and analysed, and the signal
732 compared with $4 \times 50 \mu\text{L}$ spikes directly into 0.95 mL EtOAc . Figure 3 shows the relative
733 enhancement of unpurged over purged samples. For more volatile components a value greater
734 than zero was observed (Figure 3), which indicated breakthrough of the most volatile
735 components and indicated good retention of components with a boiling point of around $225 \text{ }^\circ\text{C}$
736 (see the Supplementary Information [S3S4](#) for breakthrough tests). Concentrations measured
737 for *n*-alkanes on SPE disks were also compared with concurrent measurements made during
738 burning experiments using online thermal-desorption two-dimensional gas chromatography
739 coupled to a flame ionisation detector. The measured concentrations for *n*-alkanes from *n*-
740 nonane to *n*-dodecane were compared using both techniques, with measured concentrations
741 similar for *n*-undecane/*n*-dodecane (bp = $216 \text{ }^\circ\text{C}$, see the Supplementary Information [S4S5](#)) but
742 not the smaller alkanes. This was interpreted to indicate little breakthrough for components less
743 volatile than *n*-dodecane. These findings are in line with the US EPA certified methods for
744 Resprep SPE disks (525.1, 506, 550.1, and 549.1), when used to quantitatively analyse drinking
745 water, which show their suitability for quantitative measurement of species with a molecular
746 weight of around naphthalene/acenaphthylene (bp = $218\text{-}280 \text{ }^\circ\text{C}$). These results indicate that
747 for more volatile species with boiling points below $250 \text{ }^\circ\text{C}$, SPE disks can only be used to make
748 qualitative measurements at these sample times and flow rates. Such qualitative information is
749 highly complementary to quantitative measurements using other, less specific, techniques, such
750 as PTR-ToF-MS, where it can assist in identification of the contributors to *m/z* ions.



751
 752 Figure 3. Relative reduction of purged over unpurged samples, presented as a percentage decrease of
 753 purged to unpurged signal. The standard deviation of replicate measurements is indicated by error bars.

754
 755 **Burning results**

756 **3. Results**
 757 **3.1 Chromatography**

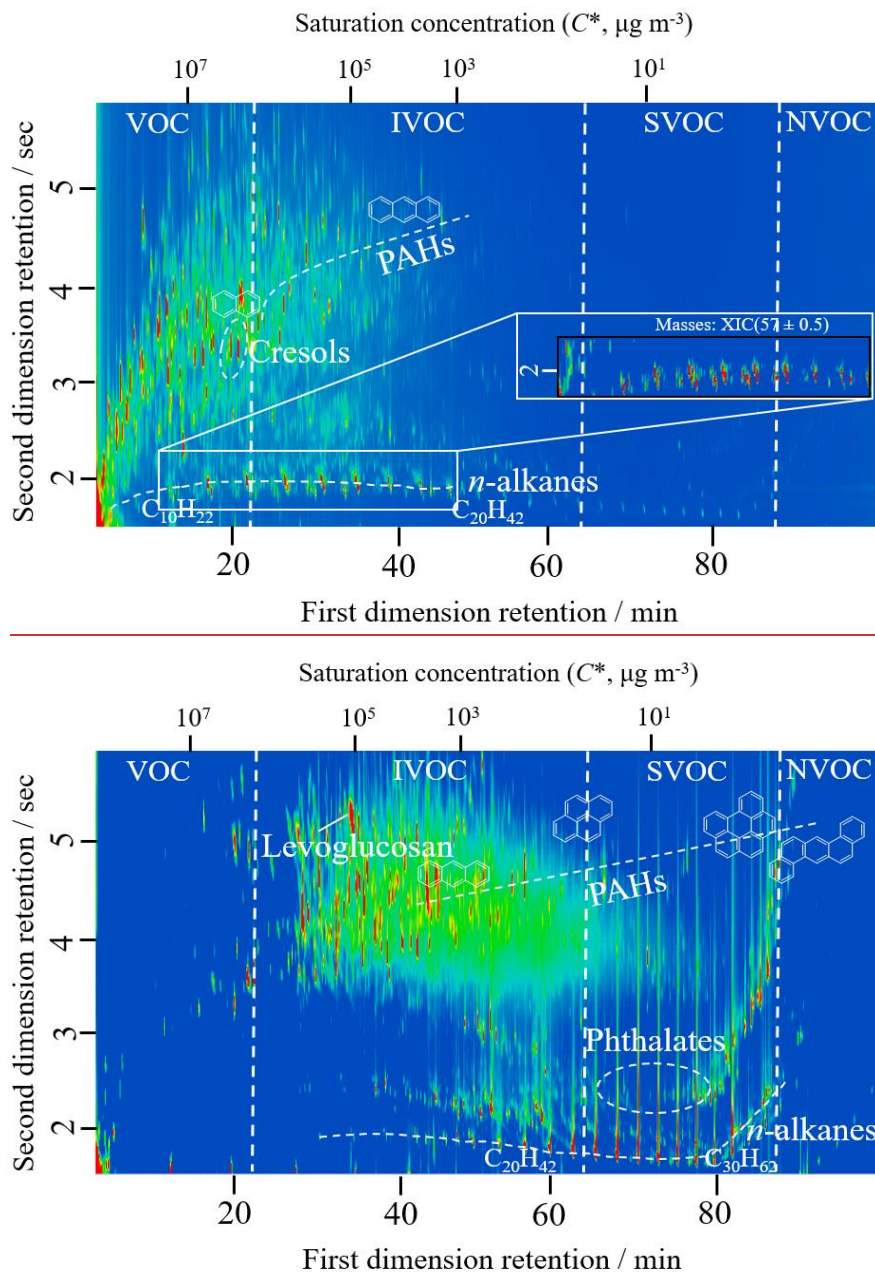
758 Figure 4 shows chromatograms from I/SVOCs in the gas and particle phase from burning a
 759 cow dung cake sample collected from SPE disks and PTFE filters during a whole 30-minute
 760 burn, after passing through a dilution and cooling chamber. The saturation concentration C_i^* at
 761 298 K is provided as an alternative x-axis and has been calculated for each n -alkane, i , using:

$$C_i^* = \frac{M_i 10^6 \zeta_i P_{L,i}^0}{760RT} \quad \text{E1}$$

762
 763 where M_i = molecular weight of VOC i (g mol^{-1}), ζ_i = activity coefficient of VOC i in the
 764 condensed phase (assumed to be 1), $P_{L,i}^0$ = liquid vapour pressure of VOC i in Torr, R = gas
 765 constant ($8.206 \times 10^{-5} \text{ m}^3 \text{ atm mol}^{-1} \text{ K}^{-1}$) and T = temperature in Kelvin (Lu et al., 2018). The
 766 constant 760 Torr has been used to convert between units of atm and Torr where 1 atm = 760
 767 Torr. $P_{L,i}^0$ values have been calculated from EPA Estimation Programme Interface Suite data at

768 298 K (EPA, 2012). The SPE disks showed ~~1295~~1297 peaks with unique mass spectra and
769 captured gaseous VOCs and I/VOCs with $C^* \sim 1 \times 10^8 - 5 \times 10^2 \mu\text{g m}^{-3}$ at 298 K. The largest
770 peaks were from alkanes, 1-alkenes, limonene, phenolics, substituted naphthalenes,
771 ~~furans~~furans and substituted pyridines. The PTFE filters captured ~~1604~~1617 I/SVOCs and
772 low/non-volatility VOCs (L/NVOC) with unique mass spectra present in the aerosol phase
773 from $C^* \sim 5 \times 10^6 - 1 \times 10^{-5} \mu\text{g m}^{-3}$ at 298 K. A transition can be seen in the two chromatograms
774 from the gas to the aerosol phase. Species with a saturation vapour concentration less than
775 $5 \times 10^4 \mu\text{g m}^{-3}$ at 298 K were predominantly in the aerosol phase after passing through the
776 dilution chamber. A large region of more polar components was present in the I/SVOC region
777 from $C^* 5 \times 10^4 - 5 \times 10^0 \mu\text{g m}^{-3}$ at 298 K and contained sugar pyrolysis products and highly
778 substituted aromatics such as those with ketone, ether and di and trisubstituted phenol
779 substituents. Many alkanes, from *n*-octadecane to *n*-triacontane were present, mainly in the
780 SVOC region. The LVOC region was dominated by a series of sterols and stanols. GCxGC
781 provided extremely high resolution to allow deconvolution of complex samples. The insert in
782 [Figure 4](#) shows how the complexity of the SPE chromatogram can be further resolved by
783 looking at a single ion chromatogram, for example $m/z = 57$, which highlighted aliphatic non-
784 polar peaks, with large peaks for alkanes from *n*-nonane to *n*-nonadecane.

785

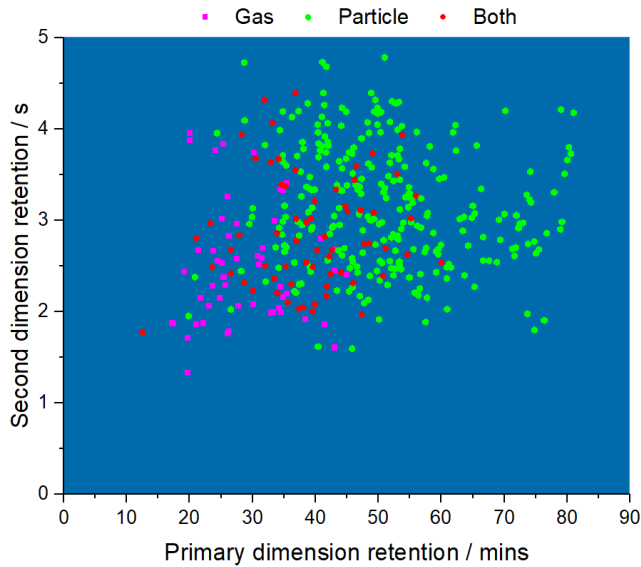


786 **Figure 4.** Chromatogram of SPE (top) and PTFE (bottom) extracted samples from the entire
 787 burn of cow dung cake. *n*-Alkane and PAH series are marked on the chromatograms. The
 788 saturation concentration scale matches the *n*-alkane series. *Figure 5 shows overlaid peak*
 789 *markers from SPE disks and PTFE filters from* See the Supplementary Information S6 for a

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790 ~~30 minute cow dung cake burn coloured~~ detailed compositional breakdown, by functionality
791 and phase. ~~Over 3000 peaks with individual mass spectra were identified. The~~

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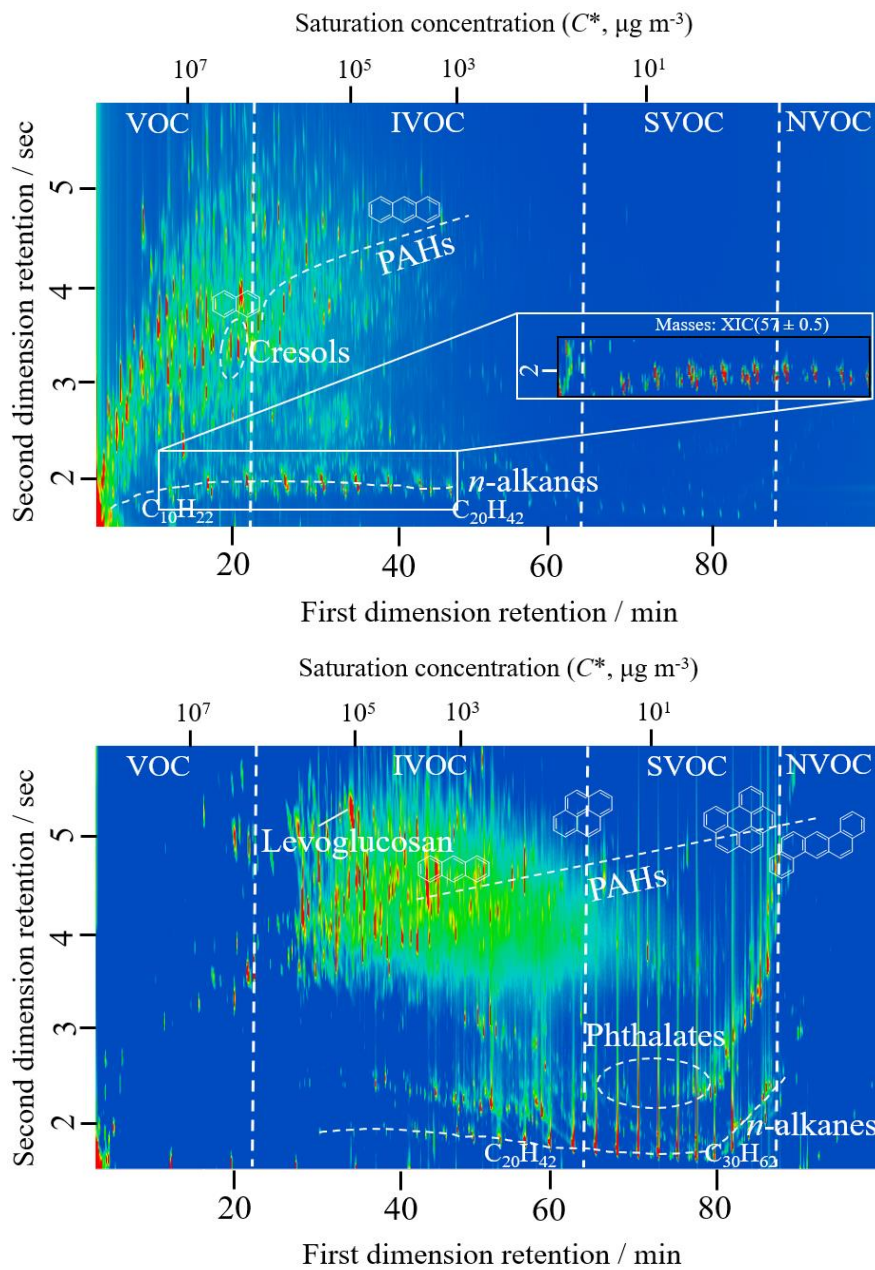


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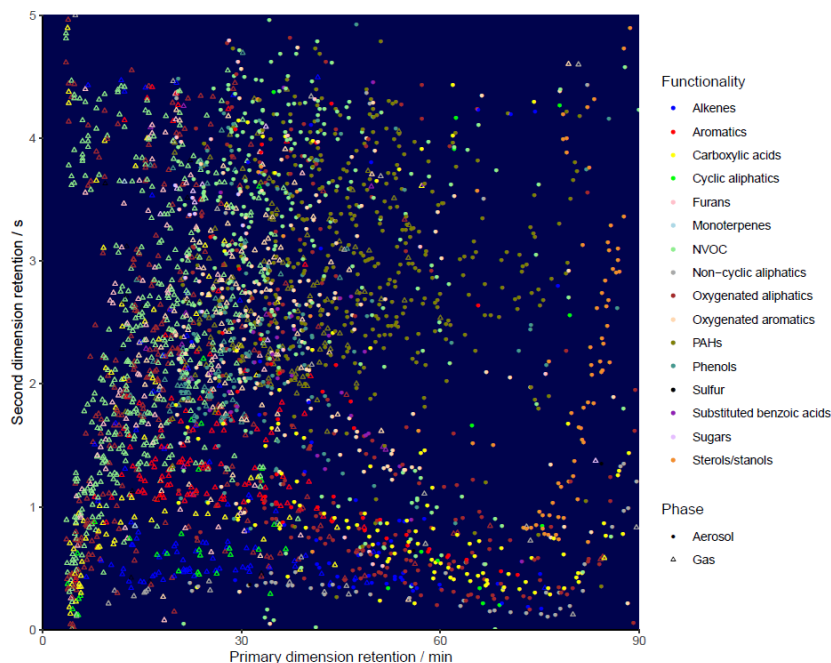
793 Figure 5. Gas and particle phase composition of PAH emissions from burning cow dung cake.

794

795 Figure 5 shows that the complexity of emissions was vast, with ~~473~~ almost 400 PAHs (~~light~~
796 ~~brown~~) forming a group towards the top centre to right of the chromatogram. The most
797 abundant calibrated PAH in the gas phase was naphthalene, followed by methyl and dimethyl
798 naphthalene isomers. A range of methyl, dimethyl, tri and tetramethyl naphthalenes as well as
799 ethyl, propyl, butyl and methyl propyl isomers were detected. Naphthalene isomers substituted
800 with aldehydes, carboxylic acids and nitriles were also released. Biphenyl and a range of
801 methyl, dimethyl and ethyl biphenyls were also released. A range of other PAHs such as
802 acenaphthylene, fluorene, azulene, quinoline, chamazulene, benzophenone, stilbene and
803 benzofurans along with their alkyl substituted isomers were also in the gas phase. A large
804 amount of highly substituted, larger PAHs with more than 3 aromatics rings in their structure
805 were present in the aerosol phase.



806 **Figure 4.** Chromatogram of SPE (top) and PTFE (bottom) extracted samples from the entire
 807 burn of cow dung cake. *n*-Alkane and PAH series are marked on the chromatograms. The
 808 saturation concentration scale matches the *n*-alkane series.



809
 810 ~~Figure 5. Gas and particle phase composition of I/SVOC emissions from burning cow dung~~
 811 ~~cake collected onto SPE disks and PTFE filters, split by functionality~~
 812 ~~where empty triangles indicate peaks in the gas phase and solid circles show peaks in the~~
 813 ~~aerosol phase.~~

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814
 815 ~~Other peaks present in Figure 5 (SPE/PTFE) included 145 alkenes (84/43), mostly towards the~~
 816 ~~bottom of the chromatogram, along with a row of 95 non-cyclic aliphatic (23/35) and 44 cyclic~~
 817 ~~aliphatic species (25/6) (see the Supplementary Information S6). Above was a row of 406~~
 818 ~~substituted aromatics, 208 (103/35), carboxylic acids (68/118) and 79 sterols/stanols (1/63) as~~
 819 ~~well as 753 oxygenated hydrocarbons containing a range of ether, alcohol and aldehyde~~
 820 ~~functionalities, 250 peaks (229/234). Peaks were also present from oxygenated aromatics, 170~~
 821 ~~from (106/145), phenols and 44 from (54/122), substituted benzoic acids. In addition to this~~
 822 ~~there were 118 (15/27), furanic species, 3 (72/42), monoterpenes (2/1) and 161 sulphur~~
 823 ~~containing VOCs species (13/4).~~

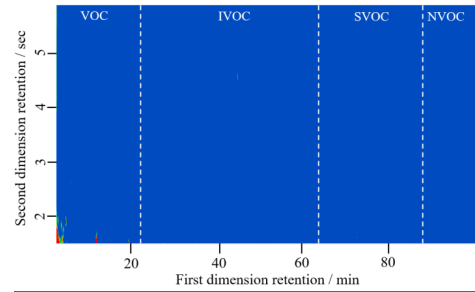
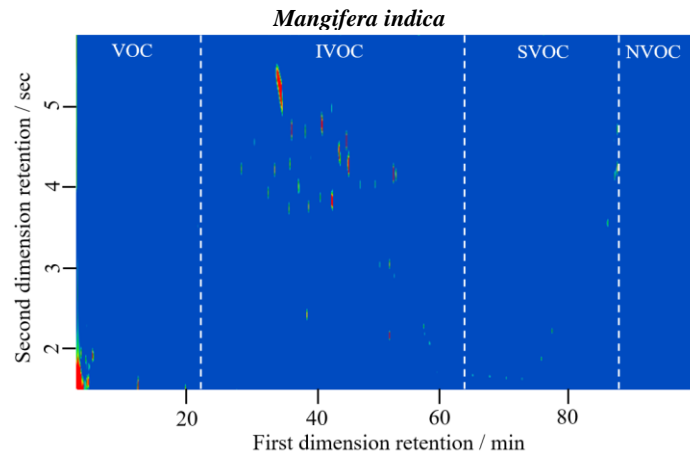
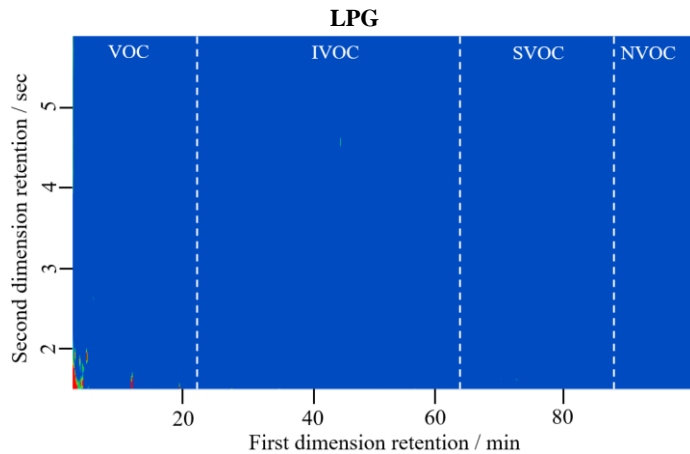
824 A wide array of ~~NVOCs~~ nitrogen containing VOCs were present in the cow dung cake samples,
 825 with ~~over 600 nitrogen containing peaks including aromatics such as peaks on SPE disks and~~
 826 ~~PTFE filters (SPE/PTFE) from pyridines and pyrazines (123 pyrazines (43/35), amines~~

827 (~~8247/28~~), amides (~~7738/37~~), nitriles (~~74~~, ~~742/31~~), 6-membered heterocycles (~~1~~), ~~613/14~~, 5-
828 membered heterocycles (~~28~~), ~~5-membered heterocycles~~ including aromatics such as
829 ~~pyrolespyrroles~~ as well as ~~pyrolinespyrazolines~~ and ~~pyrolidines~~ (~~97pyrrolidines~~ (~~50/45~~), 4-
830 membered heterocycles (~~6~~), ~~3/3~~, 3-membered heterocycles (~~64/1~~), nitrogen containing PAHs
831 (~~38~~)~~14/24~~, imidazoles (~~229/12~~), imines (~~4~~), ~~isocyanates~~ (~~3~~), ~~hydrazines~~ (~~7~~), ~~carbamic acids~~
832 (~~3~~), ~~1~~ and azoles (~~33~~) ~~oximines~~ (~~3~~) and ~~sulfur containing nitrogen compounds~~ (~~1423/10~~).
833 Previous studies have measured the nitrogen content of cow dung cake to be as high as 1.9 %
834 (Stockwell et al., 2014) in comparison to other fuel types such as fuel woods (0.14-0.35 %),
835 rice straws (0.4 %) and coal (0.6 %). The large amount of ~~NVOCs~~~~nitrogen containing VOCs~~
836 are likely formed from the volatilisation and decomposition of nitrogen-containing compounds
837 within the cow dung cake, such as free amino acids, pyrroline, pyridine and chlorophyll
838 (Leppalahti and Koljonen, 1995; Burling et al., 2010; Ren and Zhao, 2015). ~~NVOCs~~~~Nitrogen~~
839 ~~containing VOCs~~ are of concern because they can be extremely toxic (Ramírez et al., 2014;
840 Farren et al., 2015) and amines in particular can change the hydrological cycle by leading to
841 the creation of new particles (Smith et al., 2008; Kirkby et al., 2011; Yu and Luo, 2014) which
842 act as cloud condensation nuclei (Kerminen et al., 2005; Laaksonen et al., 2005; Sotiropoulou
843 et al., 2006).

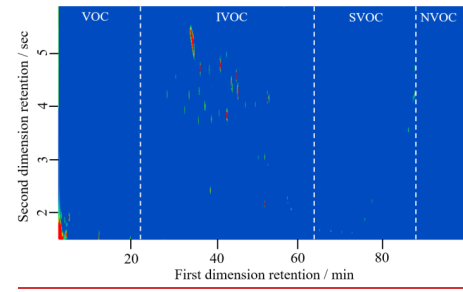
844 Figure 6 shows a comparison of organic aerosol composition observed from different fuel types
845 (LPG, fuel wood, sawdust and municipal solid waste). The measured emissions had very
846 different compositions, reflecting the variability of organic components produced from
847 different ~~sample types~~~~fuel types~~ (see the Supplementary Information S7 and S8 for species
848 ~~observed from different sample types~~). Quantitative emission factors of VOCs from the
849 ~~combustion of solid fuels characteristic to Delhi are provided in a companion publication~~
850 (Stewart et al., 2020b). Sawdust, municipal solid waste and cow dung cake (shown in Figure
851 4) emitted a wide range and complexity of species. Particle phase emissions from LPG burning
852 were minimal, with most peaks from the internal standard or contaminants in the solvent. Fuel
853 wood ~~samples~~~~combustion~~ released more organic components into the aerosol phase, with the
854 majority of IVOCs with $C^* \sim 1.2 \times 10^5 - 7 \times 10^2 \times 10^5 - 7 \times 10^1 \mu\text{g m}^{-3}$ at 298 K. The largest peak
855 belonged to levoglucosan, with other peaks from monoaromatics with several polar
856 substituents such as ethers and phenols, for example dimethoxyhydrotoluene and syringyl
857 acetone. These were likely from the depolymerisation of lignin (Simoneit et al., 1993;
858 Sekimoto et al., 2018), an amorphous polymer constituting about 25 % of fuel woods

859 (Sjöström, 1993) and formed of randomly linked, high-molecular weight phenolic compounds
860 (Shafizadeh, 1982).

861 Sawdust, although not a widely used fuel source, released many I/S/L/NVOC components in
862 the aerosol phase over a much wider range ($C^* \sim 5.8 \times 10^5 - 1 \times 10^{-3} \mu\text{g m}^{-3}$ at 298 K). The largest
863 peak was from levoglucosan, with another large peak from squalene. Many peaks were from
864 polar substituted aromatics as well as many PAHs and their substituents, such as 2-methyl-
865 9,10-anthracenedione. The largest peak from municipal solid waste burning was also
866 levoglucosan, but ~~these samples~~ this fuel type released fewer of the polar substituted
867 monoaromatics than other ~~samples~~ fuels. Municipal solid waste released alkanes and SVOC
868 species such as terphenyls, alkanes and many PAHs.

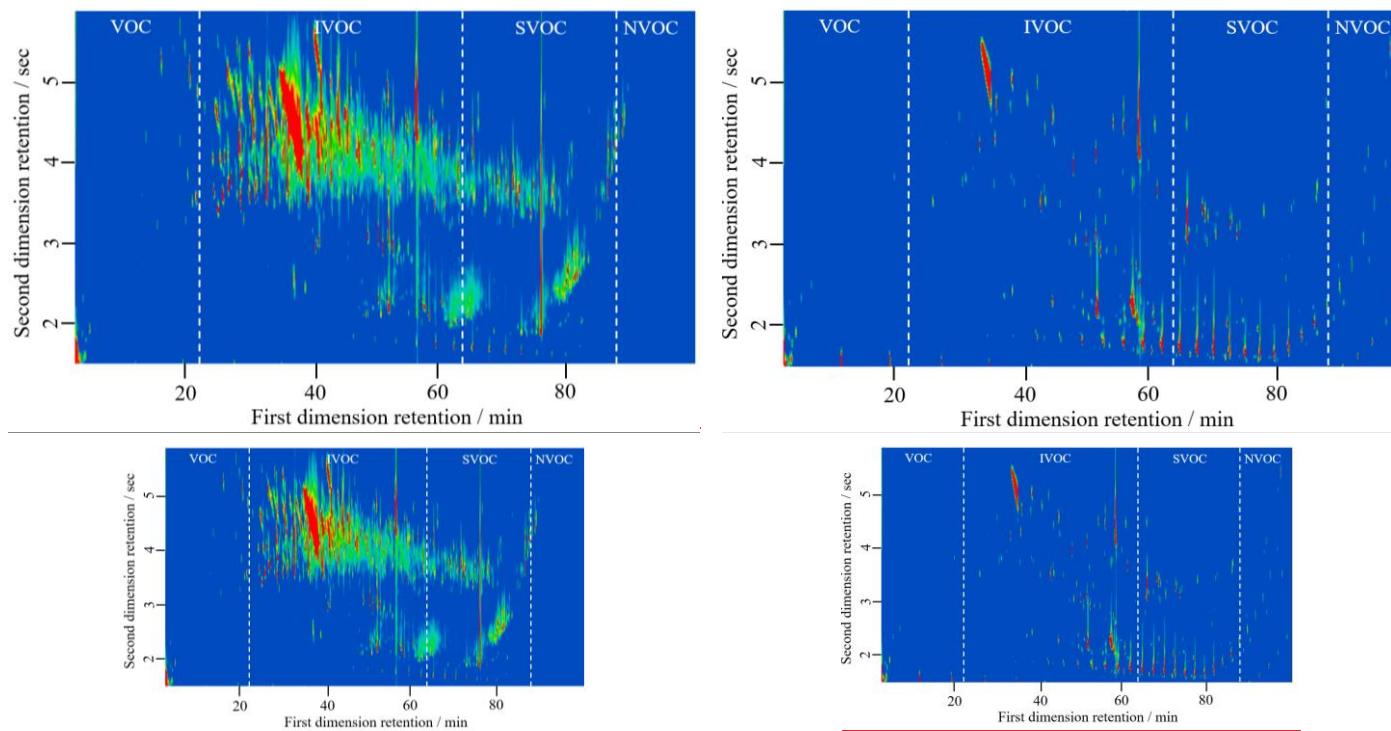


Sawdust



Municipal solid waste

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869 Figure 6. Measurements of organic aerosol from a range of different fuel types, with the contrast at the same scale.

870 **3.2 Molecular markers for domestic fuels**

871 Cow dung cake ~~samples~~combustion emitted a range of sterols/stanols, which have been
872 reported previously (Sheesley et al., 2003). This study suggested that 5 β -stigmastanol,
873 coprostanol, and cholesterol could be used as tracers for emissions from cow dung cake
874 burning. This is because in higher animals, anaerobic microbial reduction of sitosterol and
875 cholesterol forms the distinctive β configuration of the C-5 proton of 5 β -stigmastanol and
876 coprostanol. This contrasts with the α C-5 proton caused by aerobic digestion in aquatic
877 environments. Jayarathne et al. (2018) reported 5 β -stigmastanol emissions from hardwood, and
878 Fine et al. (2001) reported 5 α - stigmastanol emissions from hardwood. Four fuel wood
879 ~~samples~~combustion experiments in our study showed emissions of an isomer of stigmastanol,
880 a result similar to Jayarathne et al. (2018) that 5 β -stigmastanol was not unique to cow dung
881 cake burning or the MS measurement method used was unable to distinguish between 5 α - and
882 5 β -stigmastanol. Cholesterol and coprostanol were found uniquely ~~in~~during cow dung cake
883 ~~samples~~combustion in our study, and suggested that they can be used as unique tracers for cow
884 dung cake burning.

885 Fuel wood ~~samples~~combustion generally released fewer organic components into the aerosol
886 phase than ~~samples~~fuels such as cow dung cake, MSW and sawdust. Levoglucosan has been
887 traditionally suggested as a tracer for biomass burning emissions, however, emissions of
888 levoglucosan from a range of sources mean that this is of limited use as a unique tracer of
889 woodsmoke emissions in regions with multiple burning sources. This could potentially be
890 resolved in future studies by examining the ratio of levoglucosan to other sugars in different
891 source types ~~to~~. This may differentiate different biomass burning sources as the chemical
892 composition of different sources should determine the emission ratio of levoglucosan to other
893 sugar pyrolysis products (Sheesley et al., 2003).

894 The presence of a wide range of terphenyls ~~in~~from municipal solid waste ~~samples~~combustion
895 in this study was not unique. Jayarathne et al. (2018) suggested triphenyl benzene to be a unique
896 tracer of waste burning emissions. Whilst this study found triphenyl benzene present in one
897 cow dung cake sample and in municipal solid waste samples, the waste ~~samples~~combustion
898 emitted on average 19 terphenyls, many more than ~~in the~~from cow dung cake
899 ~~samples~~combustion (2). Terphenyls have been previously reported from incineration of waste
900 (Tong et al., 1984) and our study suggests that these compounds are good indicators of
901 municipal solid waste burning.

3.3 Total identification

Figures 7A and 7B show a comparison of the relative abundance of peaks identified, defined here as the sum of peak areas identified and calibrated using genuine standards for compounds present in the SPE and PTFE samples compared to the total observed peak area (using the blank subtracted total ion current, TIC).

Figure 7A shows that between 15 and 100 % of the peak area of the TIC in the SPE chromatogram could be identified. The highest proportion of species that could be identified was from fuel wood (67 %), followed by crop residue (57 %), charcoal (48 %), municipal solid waste (46 %), cow dung cake (39 %) and sawdust (16 %). Lower total identification in samples such as cow dung cake was due to increased complexity of emissions, which were not covered by the standards used.

Figure 7B shows that between 7 – 100 % of the organic composition of aerosol released from burning was identified. and could be quantified with genuine standards. Generally, a much lower proportion of organic matter within aerosol samples was identified due to a lack of genuine standards available, particularly in complex samples. The lowest mean relative contribution identified from samples was sawdust (9 %), followed by cow dung cake (11 %) and municipal solid waste (16 %). A larger relative contribution was identified from fuel woods (34 %) and charcoal (39 %) ~~and~~ due to less complex emissions. A large relative contribution of some fuel woods was identified from *Saraca indica* (91 %) and *Pithecellobium spp* (82 %) due to a low amount of organic matter released from these samples. This also influenced the percentage identification from crop residue which achieved 46 % identification, due to only 3 samples with 98 % identification from *Solanum melongena* but only 26 % from *Cocos nucifera* and 13 % from *Brassica spp*. 100 % of the aerosol released from LPG was quantified due to little being released into the aerosol phase and this was principally composed of PAHs. These low levels of identification of organic aerosol are in line with those reported by Jen et al. (2019) where unknown chemical species represented 35-90 % of the observed organic aerosol mass from biomass burning samples. The percentage identification in this study should not be influenced by the filter sampling media and is low in complex samples due to lack of genuine standards to allow quantitative detector-response curves for individual analytes to be developed.

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932 **3.4 Composition**

933 ~~Figure 7~~Figure 7C provides an indication of I/SVOC composition on SPE disks by mass of
934 quantified species, assuming no compound breakthrough. Phenolic and furanic compounds are
935 the most abundant I/SVOC species released from all ~~sample~~fuel types, except for LPG. As a
936 proportion of the total mass of species quantified with genuine standards on SPE disks, phenols
937 released from fuel woods (22-80 %) represented the largest range, with large amounts released
938 from municipal solid waste (24-37 %), cow dung cake (32-36 %), crop residue (32-57 %) and
939 sawdust (46 %). High emissions of phenolic compounds were of significance because
940 phenolics contribute significantly to SOA production from biomass-burning emissions (Yee et
941 al., 2013; Lauraguais et al., 2014; Gilman et al., 2015; Finewax et al., 2018). A recent study
942 found that, oxygenated aromatic compounds, which included phenols and methoxyphenols,
943 were responsible for just under 60 % of the SOA formed from western U.S. fuels (Akherati et
944 al., 2020).

945 Large emissions of furanic species were measured from fuel wood (6-59 %), municipal solid
946 waste (35-45 %), cow dung cake (39-42 %), crop residue (25-44 %) and sawdust (43 %). These
947 were important as furans can be toxic and mutagenic (Ravindranath et al., 1984; Peterson,
948 2006; Monien et al., 2011; WHO, 2016) and have been shown to be some of the species with
949 the highest OH reactivity from biomass burning emissions (Hartikainen et al., 2018; Coggon
950 et al., 2019). Furans have also been shown to result in SOA production (Gómez Alvarez et al.,
951 2009; Strollo and Ziemann, 2013) with 8-15 % of SOA produced from combustion of black
952 spruce, cut grass, Indonesian peat and ponderosa pine estimated to originate from furans and
953 28-50 % of SOA from rice straw and wiregrass (Hatch et al., 2015). Akherati et al. (2020)
954 found that heterocyclic compounds, which included furans, were responsible for approximately
955 20 % of SOA from biomass burning emissions from fuels from the western U.S. SOA
956 formation from furanic species remains poorly understood, with a recent study showing an
957 SOA yield of 1.6-2.4 % during the oxidation of 3-methylfuran with the nitrate radical (Joo et
958 al., 2019). Furans from biomass burning emissions are thought to come from low temperature
959 depolymerisation of hemi-cellulose (Sekimoto et al., 2018) and from large alcohols and enols
960 in high-temperature regions of hydrocarbon flames (Johansson et al., 2016).

961 Emissions of alkanes were most important from combustion of cow dung cake and municipal
962 solid waste (4-9 %), with only small quantities released from ~~combustion of~~ various fuel ~~wood~~
963 ~~samples~~woods (< 2 %) and crop ~~residue~~residues (< 1 %). This reinforced previous studies
964 which found emissions of C₁₂-C₃₉ *n*-alkanes from municipal waste incinerators (Karasek and

965 Tong, 1985). PAH emissions represented (3 – 15 %) of the total quantified emission by mass
966 for ~~samples~~~~fuel types~~ other than LPG and have carcinogenic and mutagenic properties (IARC,
967 1983, 1984; Nisbet and LaGoy, 1992; Lewtas, 2007; Zhang and Tao, 2009; Jia et al., 2011).
968 They can damage cells through the formation of adducts with DNA in many organs such as the
969 kidneys, liver and lungs (Vineis and Husgafvel-Pursiainen, 2005; Xue and Warshawsky, 2005).

970 ~~Figure 7~~Figure 7D shows the quantified aerosol mass was largely dominated by levoglucosan,
971 with a particularly significant contribution in the fuel wood samples (13-98 %). This was
972 similar to a previous study of fuel wood samples from Bangladesh, where levoglucosan was
973 the largest contributor to aerosol mass (Sheesley et al., 2003). Levoglucosan emissions were
974 also large from cow dung cake (30-58 %), which contrasted with the findings of Sheesley et
975 al. (2003). This could be due to differences in the feeding of cows leading to differences in
976 residual undigested organic matter in cow dung cake ~~samples~~~~fuel~~ as well as differences in
977 preparation between samples collected in Bangladesh and those in this study, which had
978 additional dried biogenic material, such as straw, mixed into samples. Levoglucosan emissions
979 were also high from sawdust (91 %), crop residue (19-85 %) and municipal solid waste (58-75
980 %), with municipal solid waste emissions likely from cellulosic material collected with
981 ~~sample~~~~the fuel~~.

982 Levoglucosan emissions from charcoal (76 %) were significant as a proportion of emissions.
983 Emissions from charcoal were low, which meant that a small emission of levoglucosan
984 represented a large proportion of total emissions. It was likely that the ~~sample~~~~fuel~~ collected
985 here may have contained small amounts of cellulosic organic matter that led to the emission of
986 levoglucosan.

987 Emissions of alkanes in the gas and particle phases were similar by source type, with particulate
988 alkanes emitted principally ~~from~~~~during combustion of~~ cow dung cake and municipal solid
989 waste ~~samples~~~~fuels~~. Emissions of particulate phenolics were large as a proportion of total
990 quantified mass with genuine standards when the total emission of other components was low.
991 For example, phenolics represented a large proportion of emissions from the fuel wood species
992 *Morus spp* and *Pithecellobium spp* with the mass principally from dimethoxyphenols.
993 Emissions from LPG were mainly PAHs and very low.

994 Whilst SPE samples for these compounds remained semi-quantitative, due to ~~slight~~
995 breakthrough, the detection of high emissions of phenolics and furanics in the gas phase from
996 burning was in line with recently published studies (Hatch et al., 2015; Stockwell et al., 2015;

997 Koss et al., 2018). Relatively low levels of total quantified material within the aerosol phase
998 was in line with the current literature (Jen et al., 2019), but meant that this analysis was not
999 entirely reflective of the organic fraction for complex samples. It is likely that this study
1000 overemphasises the contribution of levoglucosan in complex aerosol samples, relative to other
1001 components present at lower levels (Sheesley et al., 2003; Jen et al., 2019). Future instrument
1002 development could allow better quantification of complex burning and ambient samples by
1003 splitting the eluent between a -MS and -FID. This study suggests that future research uses lower
1004 sample volumes, thicker SPE disks and studies the adsorption characteristics of VOCs to the
1005 surfaces of these disks.

~~This study suggests that future research uses lower sample volumes, thicker SPE disks or disks
1006 coated with less polar coatings to minimise breakthrough of more volatile compounds to extend
1007 the range of species which can be quantified. Future work should also study the adsorption
1008 characteristics of VOCs to the surfaces of these disks.~~

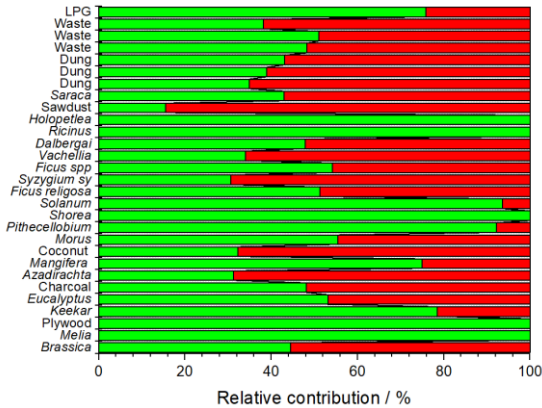
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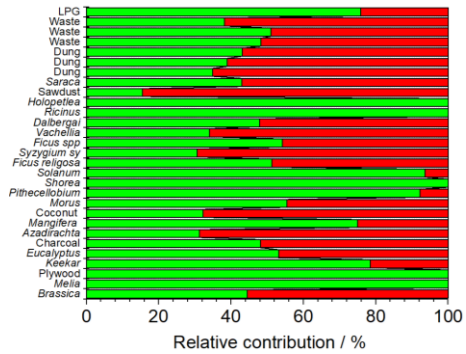
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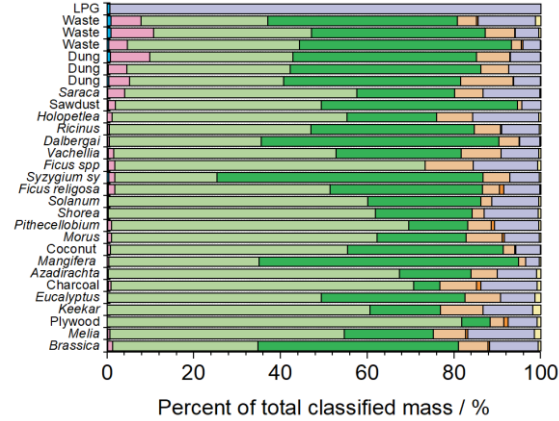
A) Proportion of SPE quantified



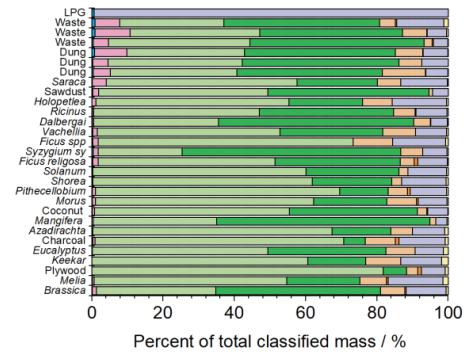
B) Proportion of PTFE quantified



C) SPE composition



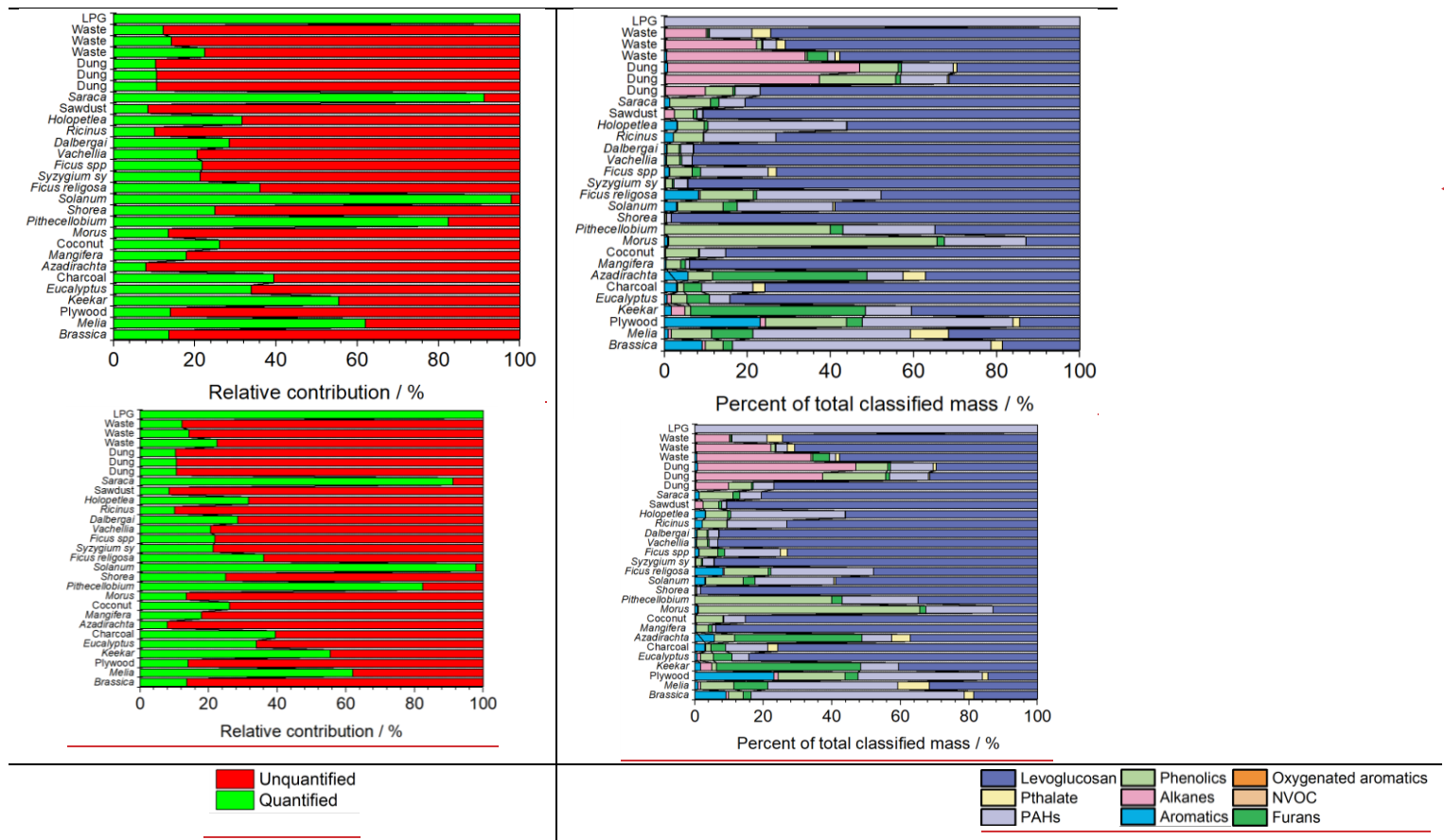
D) PTFE composition



Unquantified
Quantified

Levoglucosan
Pthalate
PAHs
Oxygenated aromatics
NVOC
Furans
Phenolics
Alkanes
Aromatics

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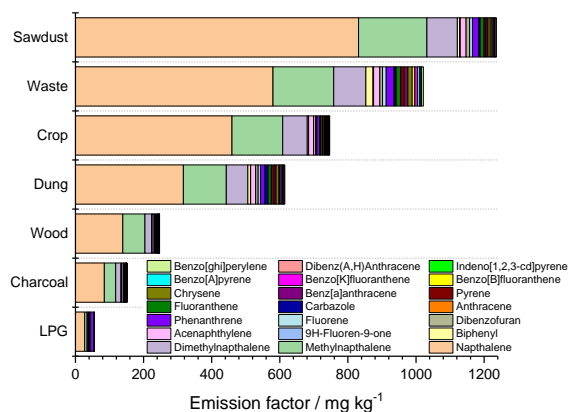
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1013 Figure 7. Area of organic matter quantified with genuine standards, as a fraction of total ion current (TIC) (7A and 7B, left panel). Semi-quantitative/quantitative
 1014 analysis of SPE/PTFE fraction (7C and 7D, right panel).

1015

1016 3.5 Development of emission factors

1017 Emission factors have been developed for PAHs (see Figure 8 and the Supplementary
1018 Information [S5S9](#) for table of emission factors by individual fuel type) by calculating the total
1019 volume of air convectively drawn up the flue and relating this to the mass of fuel burnt (see the
1020 Supplementary Information [S6S10](#) for details of calculation). Emission factors for sawdust
1021 (1240 mg kg^{-1}), municipal solid waste (1020 mg kg^{-1}), crop residue (747 mg kg^{-1}) and cow
1022 dung cake (615 mg kg^{-1}) were generally larger than for fuel wood (247 mg kg^{-1}), charcoal (151
1023 mg kg^{-1}) and LPG (56 mg kg^{-1}). The measurement of higher emission factors for cow dung
1024 cake than fuel wood was consistent with that observed in other studies (Bhargava et al., 2004;
1025 Gadi et al., 2012). [Quantitative emission factors of VOCs are provided in Stewart et al. \(2020b\).](#)

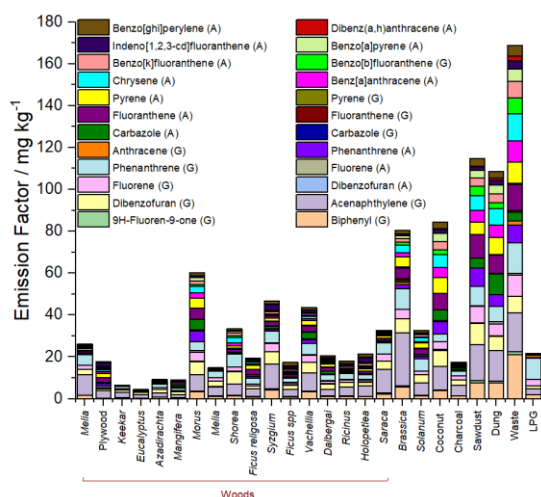


1026

1027 Figure 8. Mean PAH emission factors by fuel type.

1028 A wide range of emission factors were measured ~~for~~ from combustion of fuel wood
1029 samples woods from 50 mg kg^{-1} for *Prosopis* to 907 mg kg^{-1} for *Ficus religiosa*. Figure 9 shows
1030 that there was large sample to sample variability in emission factors for different fuel wood
1031 samples, for which only 1 sample was taken. For this reason, emission factors have been
1032 generalised for use in budget estimates to the type of fuel. Mean emission factors are provided
1033 for measurements from samples of 17 fuel woods, 3 crop residues, 3 cow dung cakes and 3
1034 different collections of municipal solid waste. Despite this, for LPG and charcoal samples only
1035 1 sample was measured, and this significantly increases the uncertainty in the PAH emission
1036 factors from these fuel sources.

1037 For most sample fuel types, PAH emissions in the gas phase were dominated by naphthalene,
 1038 methylnaphthalenes and dimethylnaphthalenes with gas-phase PAHs observed up to pyrene.
 1039 For fuel wood, crop residue, municipal solid waste and cow dung cake the percentage of PAHs
 1040 in the gas phase decreased from 97 %, 96 %, 91 % to 89 %. PAHs from LPG showed the largest
 1041 fraction in the gas phase (99.9 %) compared to the aerosol phase (0.1 %). Figure 9 shows gas-
 1042 and particle-phase PAH emissions by individual sample fuel type, excluding naphthalene as
 1043 well as C₁- and C₂-substituted naphthalenes. PAHs were present in the aerosol phase from
 1044 dibenzofuran (C₁₂H₈O) to benzo(ghi)perylene (C₂₂H₁₂).



1045
 1046 Figure 9. Emission factors of PAHs measured from SPE/PTFE where (G) and (A) represent gas- and
 1047 aerosol-phase samples, respectively, excluding naphthalene as well as naphthalenes with C₁ and C₂
 1048 substituents.

1049 Table 2 shows a comparison of the mean emission factors measured in our study with previous
 1050 studies. The mean fuel wood total PAH emission factor measured in our study (247 mg kg⁻¹)
 1051 was a factor 4.7-5.6 larger than those measured by Gadi et al. (2012) and Singh et al. (2013) of
 1052 44 and 53 mg kg⁻¹, respectively, for similar fuel woods collected across New-Delhi and the
 1053 Indo-Gangetic Plain. The PAH emission factor measured for cow dung cake (615 mg kg⁻¹) was
 1054 around a factor of 10 larger than those previously measured (60 mg kg⁻¹). The larger total
 1055 emission factors for fuel wood and cow dung cake was a result of high emissions of gas-phase
 1056 PAHs measured using PTR-ToF-MS (51-896 mg kg⁻¹ for fuel wood and 446-660 mg kg⁻¹ for
 1057 cow dung cake) compared with previous measurements made using PUF plugs (7 mg kg⁻¹).
 1058 This indicated that either the PTR-ToF-MS was able to better detect and characterise gas-phase

1059 emissions than previous methods and suggested either breakthrough or off gassing of smaller
1060 gas-phase PAHs from PUF plugs or measurement of significant quantities of other C₁₀H₈
1061 isomers on the PTR-ToF-MS. This may highlight an underestimation of 2-ring gas-phase PAH
1062 emissions in previous burning studies. Gadi et al. (2012) measured PAH emissions in the
1063 particle phase, with the mean emission for fuel wood (44 mg kg⁻¹) greater than our study (9 mg
1064 kg⁻¹). Particulate phase emissions of PAHs measured by Singh et al. (2013) from fuel wood
1065 (45 mg kg⁻¹) were also larger than our study. By contrast, particle phase PAH emissions from
1066 cow dung cake in our study (66 mg kg⁻¹) were comparable to those measured previously of 57-
1067 60 mg kg⁻¹ (Gadi et al., 2012; Singh et al., 2013). Variability in emission of particulate-phase
1068 PAHs in our study compared to literature was likely to be highly influenced by the efficiency
1069 of combustion of ~~samples~~different fuel types. This may also be explained by measuring only
1070 once for many of the fuel types, due to significant burn-to-burn differences in emissions.
1071 Although not measured in our study, differences in moisture content between ~~samples~~fuel types
1072 in our study and literature were likely have a large influence on the total amount of PAHs
1073 emitted and may explain the differences in particle-phase emissions.

1074 This study was conducted under controlled laboratory conditions. For some sample types, such
1075 as municipal solid waste, the laboratory measurement may not be entirely reflective of real-
1076 world conditions. Municipal solid waste combustion may occur under both flaming and
1077 smouldering conditions at landfill sites and in backyards, as well as in high-temperature
1078 incinerators in more developed countries. All of these are likely to have quite different
1079 combustion chemistry, and consequently lead to varying levels of emission.

1080 The particulate phase PAH emission factors from municipal solid waste combustion in our
1081 study (14-181 mg kg⁻¹) were much smaller than those of previous studies (1910-8486 mg kg⁻
1082 ¹), but the number of samples was limited. Emissions from coconut shell have not been well
1083 studied, making comparisons difficult (Gulyurtlu et al., 2003). The emission of particulate
1084 phase PAHs from sawdust in our study (62 mg kg⁻¹) was less than that previously reported 259
1085 mg kg⁻¹, but our study found large gas phase PAH emissions (1175 mg kg⁻¹). Particulate PAH
1086 emissions from the crop residue burnt in our study (13-53 mg kg⁻¹) fell within the range
1087 reported by Kim Oanh et al. (2015) of 0.34-34 mg kg⁻¹ for rice straw. Those reported by Wiriya
1088 et al. (2016) were smaller (0.47 mg kg⁻¹), but were from ~~samples~~fuel dried in an oven at 80 °C
1089 for 24 hours and ignited by an LPG burner and were likely to represent more complete
1090 combustion conditions. Emissions of PAHs from charcoal in our study (151 mg kg⁻¹) were
1091 larger than those measured for South Asian fuels (25 mg kg⁻¹), caused principally by larger

1092 measurement of gas-phase species by PTR-ToF-MS. Both our study, and that of Kim Oanh et
 1093 al. (1999) showed charcoal released the least amount of PAH per kg burnt for biofuels. LPG
 1094 combustion released less particulate PAHs (0.1 mg kg^{-1}) than previous studies (0.8 mg kg^{-1}),
 1095 but also included a small gas-phase emission (56 mg kg^{-1}). Differences in the distribution of
 1096 PAHs found in the gas and aerosol phases between our study and literature were also likely to
 1097 be influenced by the different sample dilutions and gas-to-aerosol partitioning prior to
 1098 measurement.

1099
 1100 Table 2. PAH emission factors measured in our study compared to values from literature for similar
 1101 fuel types.

Fuel	PAH (mg kg^{-1})			Ref
	Gas	Particle	Total	
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	43	(Kakareka et al., 2005)
	66	0.8	67	(Lee et al., 2005)
	105	4	105	(Kim Oanh et al., 2002)
				(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)

1102

1103 4. Conclusions

1104 This paper demonstrated an extraction technique for ~~biomass burning samples analysis of~~
 1105 SVOCs collected onto SPE disks and PTFE filters from combustion of biofuels, which was

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1106 well suited to the analysis of non-polar species. A range of samplesfuels relevant to burning in
1107 India were combusted with organic components collected and analysed, which showed large
1108 differences in the composition of organic matter released. The separation power of
1109 ~~GC×GC×GC~~ has been used to identify an extensive range of I/SVOCs in both gas and
1110 particle phases, with 15-100 % of gas-phase emissions and 7-100 % of particle-phase emissions
1111 characterised.

1112 The ability to quantify species on SPE disks was assessed and scope for future studies which
1113 should assess the adsorption characteristics of IVOCs onto SPE disks has been provided. It is
1114 recommended that breakthrough of IVOCs collected onto SPE disks at lower sample volumes
1115 is evaluated, and better methods for quantification of complex samples are developed. Further
1116 samplescharacterisation of fuel types from a wider range of sources would enable a better
1117 understanding of the drivers of poor air quality in the developing world, ~~such as crop residue~~
1118 ~~burning~~. This study found that cholestanol and coprostanol were unique to cow dung cake
1119 burning samples and these species were therefore suggested as tracers for emissions from cow
1120 dung cake burning. Similarly, municipal solid waste burning released many terphenyls, which
1121 could act as good indicators of this source. This study found that phenolic and furanic species
1122 were the most important gas-phase emissions by mass of I/SVOCs from biomass burning. New
1123 emission factors were developed for US EPA criteria PAHs present in gas and aerosol phases
1124 from a large range of fuel types. This suggested that many sources important to air quality in
1125 the developing world are larger sources of PAHs than conventional fuel wood burning.

1126 *Data Availability:* Peaks measured in this study with NIST library hits > 700 are given in the
1127 *Supplementary Information S7 and S8. Quantitative VOC emission factors measured using*
1128 *PTR-ToF-MS and gas chromatography from the fuel types studied are given in Stewart et al.*
1129 *(2020b).*

1130 *Author contributions.* GJS developed the ASE method, GC method, collected samples,
1131 organised logistics, extracted/analysed samples and lead the paper. BSN collected samples and
1132 assisted with logistics. WJFA measured VOCs by PTR-ToF-MS, supported by CNH, LKS and
1133 NT. ARV assisted in running and organising of experiments. NJF, JRH and MWW assisted in
1134 ~~GC×GC×GC~~-ToF-MS method development. SJS assisted in ASE method development.
1135 RA, AM, RJ, SA and LY collected samplesfuels, carried out the burning experiments and
1136 measured gas volumes up the flue. SSBMY aided complex sample analysis. EN, NM, RG, SKS

1137 and JDL contributed to logistics and data interpretation. TKM and JFH provided overall
1138 guidance with setup, conducting, running and interpreting experiments.

1139 *Competing interests.* The authors declare that they have no conflict of interest.

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1146 MoES/16/19/2017/APHH (DelhiFlux) to conduct this research. The paper does not discuss
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