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Assessment of polyaromatic hydrocarbon emissions from laser printers

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Abstract

The potential for polyaromatic hydrocarbon (PAH) emissions from laser printers has been examined using a simulated printing process and a detailed chemical analysis of printer toner. We have analysed the PAH content of both carbon black (a toner constituent) and toner before and after heating and have found measurable evaporation and subsequent condensation of PAHs. Based on our analysis we have estimated a maximum possible PAH emission rate of 82.1 $\mu\text{g}/\text{minute}$ of printing for a 10% page coverage. Our VOC emission results agree well with those of other authors. The concentrations of individual PAHs in the emissions were relatively low, however non-trivial, especially long term.

Keywords:

PAHs, VOCs, toner, ultrafine particles, printing

1. Introduction

1 Laser printing devices are common in workplaces, libraries and in homes.
2 There has recently been concern over the health effects of such printers;
3 specifically the generation of ultrafine particulate matter during the printing
4 process. This has in turn prompted a resurgence in inkjet technology. A
5 number of studies have measured the particle concentrations produced by
6

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7 laser printers (He et al., 2007; Schripp et al., 2008; Wensing et al., 2008;
8 Morawska et al., 2009; Koivisto et al., 2010; Wang et al., 2011; Tang et al.,
9 2012b), while others have assessed the risk of such exposures (Hänninen
10 et al., 2010). A comprehensive study by He et al. (2007) found that a large
11 proportion of commercial laser printers generated ultrafine particles and that
12 around half of those that did, did so at high concentrations.

13 This potentially represents a health risk as there is growing evidence to
14 suggest that the surface area of carbon nanoparticles (like those generated
15 by heating toner in the printing process) is the most toxicologically relevant
16 factor (Stoeger et al., 2006). Though at this stage there is little epidemiolog-
17 ical evidence linking printer emissions to health problems (Hänninen et al.,
18 2010).

19 However, work with human lung cell cultures has found that both printer
20 toner and printer emissions have a genotoxic effect (Gminski et al., 2011;
21 Tang et al., 2012a; Könzöl et al., 2013). The exact component(s) of printer
22 toner which cause this effect are, however unclear as there are many volatile
23 organic compounds (VOCs) and polyaromatic hydrocarbons (PAHs) present
24 in printer toner. The presence of such compounds is an artifact of the pro-
25 duction of carbon black (a key component in toner) via combustion in sub-
26 stoichiometric air. Additionally, the particles emitted during the printing
27 process contain inorganics, such as iron, silicon and bromine (Barthel et al.,
28 2011).

29 Depending on the temperature reached during the printing process, these
30 VOCs and PAHs may be released. He et al. (2010) studied the effect of fuser
31 roller temperature in laser printers and found that it was the most significant
32 factor in terms of the number of particles generated.

33 Given that laser printers are almost exclusively used indoors, there will
34 be a risk of exposure to printer emissions and as such consideration must be
35 given to printer emissions when monitoring indoor air quality. Kagi et al.
36 (2007) considered the implications of both chemical and ultrafine particles
37 for indoor air quality. While some studies have measured and identified
38 VOC emissions from office equipment (such as printers and photocopiers)
39 (Lee et al., 2001; Lee and Hsu, 2007), as well as considering health impacts
40 (Lee et al., 2006; Wolkoff and Nielsen, 2001), to the best of our knowledge no
41 study has identified the compounds present in emissions from laser printers.

42 The presence and release of PAHs in and from toner is important as a large
43 number of PAHs are classified as carcinogens or probable carcinogens (Straif
44 et al., 2005). This work examines the release of PAHs from toners using

45 a simulated printing process, to identify compounds that may be released
46 during the printing process, as well as the PAHs that are present in toner.

47 **2. Methods**

48 Our “simulate” printing (or toner heating) methods emulate the evaporation-
49 condensation processes which are likely to occur on the fuser-roller of a laser
50 printer. We neglect the additional forces (mechanical and electrostatic) which
51 exist in a printer. We also do not have a sorbent substance (paper) present.
52 The heating rates in our system were necessarily slower than in a real printer,
53 and the toner mass higher per unit area. However, we believe our system
54 accurately replicates the evaporation of PAHs and SVOCs from toner, if not
55 the fate once released. Our two methods (TGA and large scale) produced
56 almost identical mass losses of toner, despite different heating rates, which
57 supports this conclusion. Most studies of printer emissions (Morawska et al.,
58 2009; He et al., 2007; Wang et al., 2011) have found a spike in particulate
59 emissions at the commencement of the printing process, which rapidly dimin-
60 ishes after approximately 30 s. This suggests that SVOCs and PAHs may
61 possibly deposit on the fuser roller (or other printer components), and be
62 re-released at the start of the printing process.

63 Given that the release of ultrafine particles during a single printing oper-
64 ation is relatively low, and that PAHs only make up a small fraction of the
65 toner constituents, identifying individual components in the emissions would
66 be difficult. Therefore, this work has utilised a simulated printing process
67 whereby a sample (either toner (Black MP C5503, RICOH, USA), or carbon
68 black (Printex U, Degussa, Germany) (Bredin et al., 2011)) is heated rapidly,
69 using a Thermogravimetric Analyser (TGA, NETZSCH Tarus TG209 F3).
70 This allows any mass loss from the toner due to heating to be measured.

71 A series of measurements were made using the TGA, with samples (10-30
72 mg) of either toner or carbon black, with one of three different temperature
73 profiles. All measurements were preceded by a 45 minute purge of the sample
74 chamber with either air or nitrogen at 38°. The first set of measurements
75 was conducted by heating at 50°C/min to a temperature of 250°C. The TGA
76 was then held isothermal for 5 minutes before the temperature was returned
77 to 38°C as quickly as possible. The purpose of this rapid cooling is to give
78 as close an approximation to the printing process as possible.

79 The second set of measurements the TGA was held isothermal at 250°C
80 for 1 hour, before being allowed to return to the ambient temperature.

81 The final set of measurements considered mass loss at different maximum
82 temperatures. In these measurements, the toner was heated to temperatures
83 of 150, 200, 250 and 300°C and then allowed to return to ambient temperature
84 as quickly as possible.

85 In order to allow the results of the simulated printing process to be re-
86 lated to actual printing, we evaluated the toner consumption for a printer, by
87 printing 100 identical pages and determining the mass loss of toner from the
88 cartridge. For this work we used a FujiXerox DocuCentre IV C 3375, multi-
89 function printer. These measured values, combined with measured emissions
90 in other work were then used to calculate emission rates.

91 Additional experiments were also conducted using larger size toner sam-
92 ples (1 g) in a glass vessel. This set up involved drawing emissions from
93 heated toner through a filter. The set-up is shown in Figure 1.

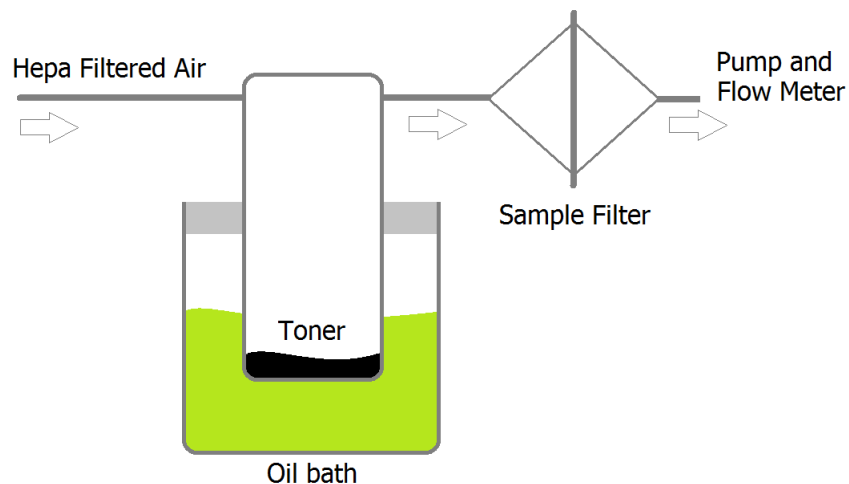


Figure 1: A schematic of the experimental set up for the large scale experiments

94 In order to maintain an evenly distributed and stable temperature a sand
95 and avocado oil bath was used to heat the glass vessel containing the sample
96 (Figure 1). The emissions from the heated toner were passed through a glass
97 fibre filter. The oil bath was pre-heated to 230 -250°C, then the glass vessel
98 place inside to simulate rapid heat-up during printing. After 10 minutes
99 the sample vessel was removed from the oil bath and the pump left running
100 for a further five minutes, so that any additional emissions could be drawn

101 through the filter. The filters were weighed before and after the tests so that
102 the collected mass of emissions could be obtained.

103 Each of the filters used was initially heated to either 550°C or 250°C and
104 then cooled in a dessicator before use. At the conclusion of sample collection
105 the filters were removed and returned to the dessicator before being weighed.
106 After weighing the filter was heated to 100°C before being returned to the
107 dessicator and re-weighed. This process was repeated for temperatures of
108 150°C and 250°C.

109 In order to approximate the amount of carbon black in the toner, an ele-
110 mental analysis of both toner and Printex U was performed using a Thermo
111 Finnigan EA 1112 Series Flash Elemental Analyser.

112 *2.1. PAH analysis*

113 Samples of carbon black and toner were analysed for PAH content. Ad-
114 ditional samples of carbon black were heated to 250°C and 300°C and then
115 analysed to measure any loss of PAHs through evaporation, as may occur
116 during the printing process.

117 Identification of the PAHs was carried out by extraction using organic
118 solvent, followed by pre-concentration and analysis by gas chromatography
119 mass spectrometry (GC/MS). Control and laboratory blank samples were
120 analysed concurrently with the toner samples.

121 Samples were spiked with a known amount of surrogate standard (Chem-
122 Service Product CSS8250-1JM) and placed in the ASE for further extraction
123 with 4:1 mixture of hexane and ethylacetate. The sample extracts were com-
124 bined and concentrated down, a known amount of internal standard (Chem-
125 Service Product PP-HC8JM) was added prior to the sample being made up
126 to volume with equal volumes of ethylacetate and hexane. Sub-samples of
127 the extract were analysed by GC/MS.

128 For the duration of this study an Agilent 6890 Gas Chromatograph (GC)
129 coupled to a 5973 Mass Spectrometer (MS) operating in the selected ion
130 monitoring (SIM) mode was used to carry out the analyses. The GC was
131 fitted with a HP-5MS capillary column (60 m x 0.25 mm x 0.25 μ m). The
132 injector was operated in the pulsed splitless mode and was held at 300°C
133 with a target pulse pressure of 50 psi. After the run had been initiated the
134 oven was held at 50°C for 2 minutes, the column was then heated to 240°C
135 at 12°C/min and then to 310°C at 3°C/min, the column was then held at
136 this temperature for 16.5 minutes. The GC eluate was introduced into the
137 MS via a transfer line maintained at 300°C. The MS operated with a start

138 delay of 10 minutes and positive electron ionization (EI) of 70 eV. To improve
139 sensitivity the system was set to monitor five groups of ions ranging from 82
140 to 172 atomic mass units (amu) with a dwell time of 40 milliseconds (ms)
141 in Group 1, to 138 to 300 amu with a 40 ms dwell time in Group 5. The
142 GC/MS system was calibrated using five calibration standards, plus a zero
143 point.

144 3. Results and Discussion

145 In order for laser printers to produce emissions (from the toner) there
146 must be a mass loss of toner during the printing process. Figure 2 shows
147 the mass loss of samples of toner, during the simulated printing process
148 at different temperatures. It is clear that significant mass loss (and hence
149 release of emissions) occurs at temperatures higher than 250°C. There is also
150 minimal mass loss at 150°C, indicating minimal water and VOC content in
151 the toner. The significantly higher mass loss at 300°C, is possibly due to
152 thermal decomposition of the binder (a styrene based polymer).

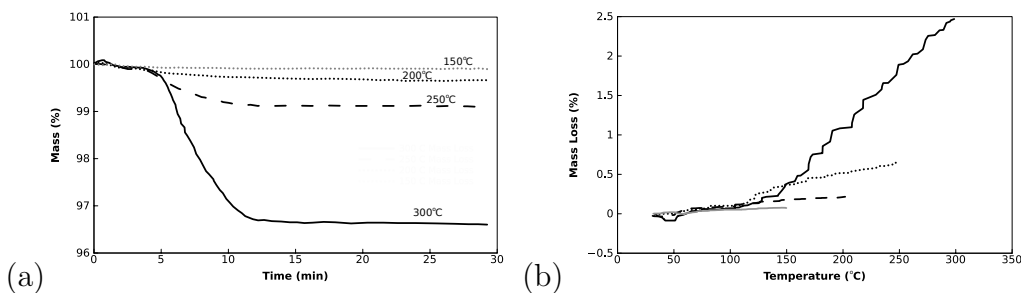


Figure 2: Mass loss of printer toner during a simulated printing process at different temperatures, (a) shows the change in the original mass of toner during the simulated printing process and, (b) shows the cumulative mass loss during heating.

153 This is in contrast to Printex U, which begins to lose mass at lower
154 temperatures. This can be seen clearly in Figure 3 where results for both
155 Printex U and toner are shown. All samples were heated to 250°C. The
156 temperature profile is shown so that the mass loss can be seen in relation to
157 temperature. All samples lost a significant proportion of their total mass loss
158 during heating. The higher mass loss of Printex U samples at the beginning
159 of the measurement, was attributed to the presence of moisture in the sample
160 (despite dessication prior to analysis, which suggests a high surface area).

161 In comparing the results, it should be noted that carbon black is a com-
 162 ponent of toner and therefore would be expected to show a higher mass loss
 163 than the toner itself, where it has effectively been diluted with binder. It is
 164 useful in this study, to consider Printex U as it contains a higher proportion
 165 of PAHs and therefore they should be more readily measured.

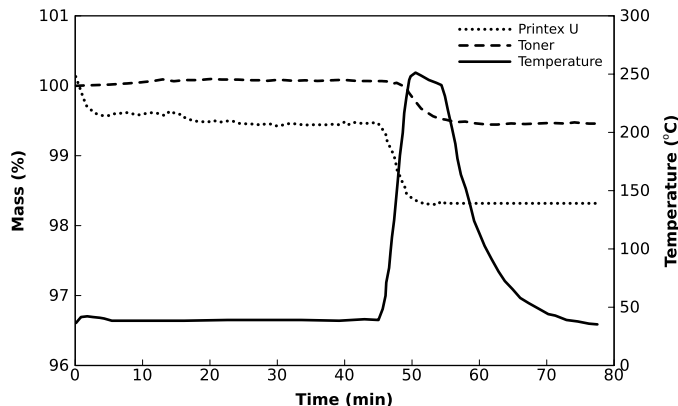


Figure 3: Mass loss of toner and Printex U samples during the simulated printing process, for 4 different maximum temperatures.

166 Given that printer toner contains carbon black in combination with binder
 167 and other components, the VOC and PAH analysis was carried out on the
 168 Printex U, as this is essentially the carbon black component of toner in
 169 isolation. The results of the VOC analysis are given in Table 1, where c is
 170 the concentration of the compound in the carbon black (w/w).

171 The VOCs shown in Table 1 only make up 0.015 % of the mass of Printex
 172 U, and so are likely present in toner in very low (difficult to measure) con-
 173 centrations.

174 The results of the PAH analysis are shown in Table 2. Multiple samples
 175 were analysed, with the resultant mean concentration, c_m , and standard
 176 deviation (SD) given. Samples of Printex U were analysed as supplied as well
 177 as after heating to 250°C and 300°C. None of the PAHs present in Printex U,
 178 as supplied, were detectable in the samples heated to 300°C, and so no data is
 179 shown in Table 2. Only compounds detected are listed, quantities indicated
 180 by $<$ were below the detection limit. The standard deviations for some of
 181 the heated samples are not listed as only one of the three samples gave a
 182 quantity over the detection limit. In these cases the concentration given is
 183 for the single measurement not a mean concentration. The large standard

Table 1: VOC content of Printex U

Compound	c (mg/kg)
Benzene	30
Styrene	4.4
Toluene	95
Xylene, m- & p-	12
Xylene, o-	0.7
1,2,4-trimethylbenzene	0.6
Ethylbenzene	0.9
Chloroform	2.8
Tetrachloro-ethylene	1.4
Dichloromethane	2.5

184 deviations for some compounds are attributed to samples being taken from
 185 different batches of Printex U. Some individual Printex U samples showed
 186 overall PAH levels up to 5x that of a typical diesel engine soot (also analysed
 187 by the authors), which is concerning.

Table 2: PAH content of Printex U

Compound	As Supplied		After Heating to 250°C	
	c_m (mg/kg)	SD \pm	c_m (mg/kg)	SD \pm
Naphthalene	70.1	46.0	5	-
Acenaphthylene	83.4	58.4	<	-
Acenaphthene	12	-	<	-
Phenanthrene	402.8	143.4	48.5	31.8
Anthracene	47.6	12.4	<	-
Fluoranthene	173.3	73.2	86.5	33.2
Pyrene	109.4	50.1	56	22.6
Benz(a)anthracene	37.5	-	<	-
Chrysene	30.2	-	<	-
Benzo(b)fluoranthene	11	1.4	8.5	2.1
Benzo(k)fluoranthene	16.8	2.9	7	-
Benzo(e)pyrene	5.7	0.6	<	-
Benzo(a)pyrene	9.5	5.7	<	-
Indeno(1,2,3-cd)pyrene	5	0	<	-

188 It is evident from Table 2 that PAHs will be released when Printex U and

189 hence toner is heated, as is the case in laser printers. The Printex U samples
190 contained between 0.08 and 0.13 % PAH by mass.

191 Table 3 shows the results of the PAH analysis for printer toner, again
192 only compounds present in quantities above the detection limit are shown.

Table 3: PAH content of toner

Compound	c_m (mg/kg)	SD
Phenanthrene	5.1	1.7
Anthracene	5.8	1.7
Fluoranthene	5.9	0.4
Pyrene	2.7	1.7
Benz(a)anthracene	9.1	-
Chrysene	7.1	-
Benzo(k)fluoranthene	3.5	1.0
Benzo(a)pyrene	5	1.4

193 Based on the results of the elemental analysis, and assuming a styrene
194 binder (or binder with comparable hydrogen to carbon ratio) the amount of
195 carbon black in the toner was found to be approximately 4.7 %. Considering
196 the significant amount of dilution of carbon black in toner, it is not surprising
197 that fewer PAHs were detectable in the toner samples. These are mostly the
198 compounds present in the highest concentrations in Printex U.

199 In order to relate these results back to ‘real world’ printing we consider
200 the rate of printing and toner consumption for a typical office printer. For our
201 given printer (see Section 2) the results of toner consumption tests are shown
202 in Table 4, where toner usage per page is shown in relation to a percentage
203 of page coverage. Alternatively, we can also express toner consumption in
204 relation to a unit printed area (100% coverage), which in this case is 6.19
205 $\mu\text{g}/\text{cm}^2$.

206 Using the manufacturers listed printing rate of 35 pages/minute and as-
207 suming a 10% coverage per page, the PAH emissions in relation to printing
208 time are estimated at 82.1 μg PAH/minute. It should be noted that this
209 would be an overestimate as this assumes that all PAHs are emitted to air.
210 In reality, it is likely that given the proximity of the paper to the fuser roller,
211 some (or most) of the PAHs emitted will be absorbed by the paper. The
212 other consideration is that the heating of the toner by the fuser roller will
213 likely be more rapid than in the simulated printing process we have used,
214 which may lead to reduced emissions. The estimate provided here should be

Table 4: Toner consumption during printing

% page coverage	toner used (mg/page)	PAH* ($\mu\text{g}/\text{page}$)
5	19.19	1.17
10	38.39	2.35
15	57.58	3.52

* Estimated value, based on carbon black composition. The majority of PAH emitted would remain in the printed area or be absorbed into the paper.

215 treated as a ‘worst case scenario’ and indicates that we need at least to be
 216 aware of PAH emissions from printers, or PAH levels absorbed into printed
 217 paper.

218 The results of our work may also be compared to results in the literature
 219 of measured printer emissions. Wang et al. (2011) measured the emissions
 220 of VOCs during printing, calculating total VOC emissions of 2.14 mg, over
 221 a 1000 page print cycle, giving 2.14 $\mu\text{g}/\text{page}$. Based on our VOC analysis
 222 of carbon black, we estimate VOC emissions of 2.7 $\mu\text{g}/\text{page}$. The difference
 223 between the two values is likely due to a difference in compositions in the
 224 VOC components of the toner used by Wang et al. (2011) and carbon black.
 225 With that considered, the two results agree well.

226 To further confirm the release of compounds from toner, large scale tests
 227 were conducted. These tests heated the toner and collected any particulate
 228 produced in a filter. Toner mass losses during these tests were equivalent to
 229 those given in Figure 2(a). An increase in mass in the filter was observed
 230 indicating the presence of such substances in toner emissions. After initial
 231 weighing, the filters were heated, then weighed. It was found that all collected
 232 emissions could be evaporated from the filter at or before 250°C. The results
 233 are shown in Figure 4.

234 It can be seen in Figure 4 that all mass collected by the filter is driven off
 235 at 250°C. On this basis, and given the known presence of PAHs and VOCs
 236 it is assumed that all collected emissions are either, water, SVOCs or PAHs.
 237 Given that over 80% of the mass collected on the filter is still present after
 238 heating to 100°C, it is assumed that the majority of the emissions are VOCs
 239 and PAHs. Additionally, based on the results of the VOC analysis, where
 240 the only (detected) compound with a boiling point above 150°C was 1,2,4-
 241 trimehtylbenzene, we propose that the majority of the collected emissions
 242 are PAHs. We do note however, that during the printing process, long chain

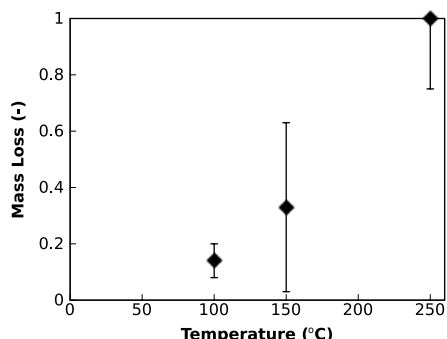


Figure 4: Cumulative mass loss from the filters, each data point represents a mean value, with error bars indicating ± 1 SD

243 alkanes and siloxanes have been detected by other authors (Barthel et al.,
 244 2011; Morawska et al., 2009). These however, are considered to be too volatile
 245 (and in too low concentrations) to form persistent particles (Morawska et al.,
 246 2009) and so were not specifically tested for. Furthermore, the carbon and/or
 247 toner contains no silicon and therefore siloxanes must have another source
 248 (such as printer components - therefore presumably more of an issue with
 249 newer printers). This leads to the interesting question of source (and gener-
 250 ation mechanism) apportionment of printer emissions, which is however
 251 beyond the scope of this work.

252 We have only considered the evaporation-condensation mechanism of
 253 aerosol formation, neglecting additional mechanical forces. Other forces (me-
 254 chanical, electrostatic) and processes exist, which may serve to either gener-
 255 ate additional particles, or collect those generated from heating and fusing
 256 toner.

257 4. Conclusion

258 This work identified 14 different PAHs in toner and its constituents. A
 259 number of these were released during a simulated printing process and are
 260 therefore likely to be released during normal laser printing operations. We
 261 have estimated maximum possible PAH emissions at $82.1 \mu\text{g}/\text{minute}$ for 10%
 262 page coverage at 35 pages/minute. Due to differences between the actual
 263 printing process and our simulated printing process actual emissions are ex-
 264 pected to be lower than this estimate. As manufacturers are beginning to
 265 use lower printing temperatures the amount of PAHs released will likely be

266 decreased. Nevertheless it may be advisable for carbon black manufacturers
267 to attempt to reduce PAH levels on their products. and more consideration
268 given to PAHs.

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