

# Chemical Determination of Odorants in Air from Sewage Treatment Works

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## Abstract

Public awareness and concern on odorous emissions from waste treatment facilities is increasing. Odors may be released to the air during the treatment of sewage and thus cause health effects. The volatile chemicals are commonly classified in four groups, sulfur-, nitrogen-compounds, volatile fatty acids and other chemicals (mainly aldehydes/ketones). In this study, ambient air sampling by sorbent tubes and trace chemical analysis by GC-MS were conducted in order to understand the character and component of the odorants. The analysis of odorants includes field air sampling by pulling a volume of air through a sorbent tube and laboratory testing by using a newly established method, namely Thermal Desorption Unit (TDU) coupled with GC-XLMSD analysis. The TDU-GC-XLMSD method was validated for the determination of volatile and semi-volatile odorants. The results showed main components were amines, aldehydes, acids, mercaptans and other sulfur, ether, ester compounds. The GC-XLMSD identification of odorant components in odor samples is based on their retention time and monitoring ion m/z ratio, then comparison to MS data base and the available authentic standards of 59 odorants and commercial odor standard TO-14. The developed method was applied for determination of odor nuisance in over 835 odor samples collected from three wastewater treatment facilities of Hong Kong. Study showed sludge storage zone would need to be very open not for H<sub>2</sub>S concentrate which would be impact on health. The organic acids were existed as esters in odor. Deodorizing units could reduce odor amounts between 37.4-97.6 %, if not count on including H<sub>2</sub>S. Deodorizing units could reduce H<sub>2</sub>S 86-100%,

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most removal efficiency were 95-100% in both by TDU-GC-XLMSD and Odalog meter.

## **INTRODUCTION**

Sewage treatment facilities produce odorous that can cause nuisance to neighbours. Odor complaints maybe caused by a variety of odorous compounds that are released or generated in the facilities. The major types of odors in sewage treatment facilities can be nitrogen compounds such as ammonia and amines, sulfide compounds such as hydrogen sulfide, mercaptans and other reduced sulfides, organic acids and their derivatives, and hydrocarbons such as aldehyde, ketones and aromatic hydrocarbons. Some of the odorants can have strong pungent nuisance smells and some may be harmful to human health. In this study, chemical odorants from air samples taken from sewage treatment facilities were collected and analyzed. Odorants included volatile organic compounds, amines, aldehydes, mercaptans, sulfur-, nitrogen, acids, petroleum hydrocarbons. For air sampling and chemical analysis, a combination of sorbent tube, thermal desorption unit (TDU) and GC-MS was successful in determining odorants at the required detection levels. Hydrogen sulfide (H<sub>2</sub>S) was found to be the main odorant which has a characteristic strong bad-egg smell and high in toxicity. Olfactometry method was also carried out to compare the sensory results against chemical odorant data.

## **MATERIALS AND METHODS**

Agilent Technologies 7890A GC System equipped MEGA-WAX MS fused silica capillary column was employed for determination of odorants from Waste Water Treatment Works (WWTWs) odor samples. A suitable GC column MEGA-WAX MS 0.25 $\mu$ m x 0.25 mm x 60 meters was selected and GC temperature program was optimized in order to cover most non-polar as well as relatively polar compounds. The optimized GC temperature program is described as following: inlet part at 250 °C, oven temperature start at 36 °C hold 3 min. then increase to 75 °C with increasing rate 8 °C/min, continue to increase oven to 180 °C at increasing rate 15 °C/min. then continue to increase to 240 °C with increasing rate at 10 °C/min, hold for 11 min. The injection mode was splitless with post run at 250 °C for 1 min. Electron-ionization (EI-MS) conditions includes ionization potential of 70eV in positive ion mode were optimized for achieving the best sensitivity, with scanning mass range of m/z 30-250.

A sampling apparatus capable of control air sampling rate at 30ml/min. Various sorbents were examined for sampling of target compounds in order to trap and retain compounds, Tenax® was selected as the absorbent which is a porous

polymer resin based on 2,6-diphenylene oxide. The sorbent tube has been specifically designed for the trapping of volatiles and semi-volatiles from air. Selection of this relatively hydrophobic sorbent for absorbent tube was found to be able to minimize water interrupting. The obtained data indicated that the sampling time of up no longer than 60 minutes provided stable results. Longer sampling time might cause odor overloading. Therefore, sampling time of 40 and 60 minutes, with the total odor volumes of 1.2 and 1.8 liters, were selected and validated.

The GC-XLMSD characterization of 74 odorants in odor sample is based on chromatographic retention time and mass spectrometric characteristic ions as well as spectrum library searching. The 74 odorant characteristic ions of the odorants under electron-ionization (EI-MS) have been established (Appendix1). The EI-MS spectrum library of the odorants has been established. The developed characteristic ions will be used in GC-XLMSD analysis for detection of possible occurrence of any odorant in the air. Library spectrum will be used for confirmation. Total 74 requested odorants, authentic standards of 59 odorants and TO-14 standards were purchased. The odorant standards were analyzed under the optimized GC-XLMSD conditions for validation of the established EI-MS characteristic ions (Appendix1). The analysis also provided information of chromatographic retention time, which will be used for convenient and accurate identification of odorants in real odor samples. For those odorants that their authentic standards were not available, their possible detection in the air will be based on the established characteristic ions and library searching. Most acids, for example, could not be detected directly under the current conditions, but their relative esters showed up at chromatography.

#### **Standard calculation curve**

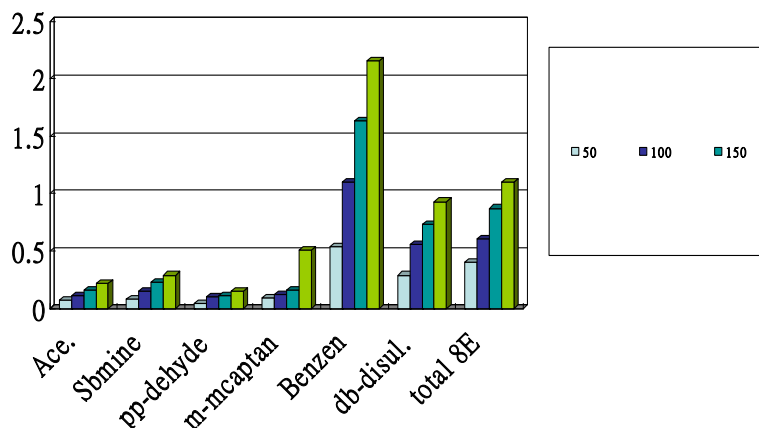
Because concentrations of standard gas mixture were very changeable with their container volume, atmosphere pressure and environment temperature, so it's not accurately to make different concentrations in a same volume in several containers, but it can be made with different injected gas mixture volume to make up a series of different amount of the gas mixture for checking their peak integration area to see if positive relationship to their amount (by different injected volumes) exists.

There were a series of injected volumes as followings:

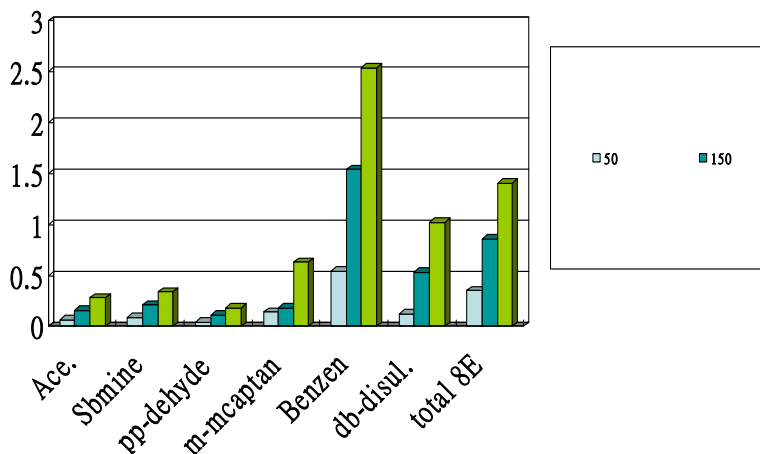
50, 100, 150, 200 ppbv for both TO-14 and 59 gas standard mixture were used. The results showed the procedure and determination results were reproducible, with relative standard deviation used the standard gas mixture TO-14 and 59 species of standard mixture within 15% (n=3). Determination limit were 0.5 ppbv

for most target compounds, with their peak area three time higher than noise. The acids existed as esters had little low sensitivity with determination limit 0.5-1.0 ppbv.

**Fig. 1. Standard calculation curve from TO-14 standard gas mixture at four different concentrations**



**Fig.2. Standard calculation curve from 56 standard gas mixture at three different concentrations**



## RESULTS AND DISCUSSION

1. The method combining sorbent tube, TDU and GC-XLMSD was developed for sampling and analysis of chemical odorant of air samples from sewage treatment facilities. The odorants of air from two wastewater treatment works (WWTWs) in Hong Kong contain volatile and semi-volatile organic compounds

and they could be classified in four groups: sulfur compounds, nitrogen compounds, organic acids, as well as aldehydes, ketones, ethers, esters. Among the odorants, hydrosulfide (H<sub>2</sub>S) is toxic and has strong bad-egg odor and it is commonly found in flume channel for incoming sewage, effluent from primary settling tanks, inlet to deodorizing units, as well as sludge handling facilities. The method developed here is multi-sorbent adsorption and thermal desorption, then concentrated by frozen – Nutech 2502 DS system with gas chromatography – XL mass spectrometry Detector (GC-XLMSD). Agilent Technologies 7890 A GC System with Agilent Technologies 5975C inert XLMSD are employed here. The method enables the sampling and determination for the wide range of organic compounds (VOC) and have detected more than 74 specified chemical odorants from various locations within two STWs including influent area, primary effluent area, ambient air, sludge handling site, and dewatered sludge cake. It showed good selectivity, sensibility and precision according to Compendium method TO-17 (US Environment Protection Agency) criteria. Limits of detection (signal to noise ratio = 3) ranges were 0.2- 0.5 ppbv. Duplicate determination results of main target compounds were within 15%, except 20% for sulfide.

2. The TDU-GC-XLMSD system was set up in 2011 and it had been used to determine on over 835 odorant samples from Stonecutters Island and Shatin Waste Water Treatment Works. Data showed the method with GC-XLMSD instrument performed very well except that a correction for hydrogen sulfide had to be made due to its degradation in the sorbent tube, and ammonia could not be determined due to its molecular weight of 17 being too small for MS to collect. Organic acids are determined in their ester forms.
3. “Direct Source Sampler” was designed. Ambient air had been collected very close to the skips for dewatered sludge cake. Despite the close proximity of the air intake point to the sludge cake, ambient air cannot be avoided. In order to investigate the true odor of the solid sample, Direct Source Sampler was a vessel constructed to hold a certain quantity of solid sample in order to maintain a stable environmental condition for air sampling to eliminate ambient air or wind effects. It aimed to collect the original odor characteristics for the solid sample as ‘source’. Air samples from dewatered sludge cake were specifically taken with this method. The results were much higher than field air sampling even very close to the sludge cake. In the confined space with sludge cake, high H<sub>2</sub>S gas in excess of 50 ppm was found.

4. Concurrent air sampling for both chemical odorant analysis and olfactometry test had been carried out. Olfactometry test was performed in accordance with EN13725 carried out by Odor Research Laboratory, Hong Kong Productivity Council (HKPC). A total of 157 concurrent odor samples have been collected and tested, both by chemical and olfactometric methods. HKPC olfactometry data would shed light on odor intensity, and Dioxin Laboratory of HKBU would describe odorants as individual chemical odorants in great details. Based on known odor threshold of most of the 74 odorants, a summated 'odor number' value was calculated based on concentrations of individual odorant concentrations and their respective odor threshold values. The actual odor unit (by EN13725) was found to be much lower than the summated 'odor number', and there appeared to have a positive correlation between the two data sets.
  
5. Deodorizing units were designed for remove hydrogen sulfide mainly, and other odorants such as amines, aldehydes, mercaptans and acid's esters would also be removed but may be not so efficient. Deodorizing unite sampling from Sept. 2013 to April 2014, There were total 157 odorant sampling have been collected. Their removal efficiency were showed on Appendix 2. Total odorants no including H<sub>2</sub>S by TDU-GC-XLMSD the deodorizing efficiency by DO units were between 37.4-97.6%. If only on H<sub>2</sub>S by TDU-GC-XLMSD calculation deodorizing efficiency were between 86.0-99.9%. From Odalog reading data, the deodorizing efficiency was between 92.3-100.0%. So in general, the DO units removal efficiency for H<sub>2</sub>S were high, which could be nearly all removed. But for amines, esters, aldehydes, mercaptans and some other sulfite compounds, except H<sub>2</sub>S, removal efficiency were wave between 37.4-97.6% (Appendix 2).
  
6. Odorant data analysis showed at radial plots. From the plot at top, odor samples from the locations of wastewater influents, DO inlet, had high H<sub>2</sub>S, with high concentrations of acid group, Dewater house marked as cake area, ambient air and DO outlet had low concentrations of H<sub>2</sub>S. Ambient air plot showed high concentrations of S-group and acid group (second plot). From the third pair plots, left plot showed primary effluent had high concentration of H<sub>2</sub>S, but at cake area which original location was Dewater House, the plot showed abundant of N-group, aldehyde group, S-group and acid-group, but not H<sub>2</sub>S, in where there was very strong pungent nuisance relieved from sludge containing N-, Aldehyde-,S-, mercaptan, acid-groups compounds, but no exist H<sub>2</sub>S at both determination, TDU-GC-XLMSD or Odalog meter. From

the plots in the bottom showed Deodorizing units inlet contained high concentration of H<sub>2</sub>S, but outlet showed low concentrations of H<sub>2</sub>S and other compounds (Appendix 3).

7. Other than 74 specified chemical odorants, petroleum hydrocarbon compounds were found in air samples taken from various locations inside the two WWTWs. It was found that, for all air samples taken, there was around couple ppm level, except H<sub>2</sub>S. For non-H<sub>2</sub>S compounds, their maximum concentrations detected (in the order of one to a few hundred ppb) were not high when compared to their occupational safety and health exposure limits (for example, 50 ppm for 1,2-dichlorobenzene and 5 ppm for n-butylamine).

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Appendix 1. The 74 odorants, their detection limits (DL) and characteristic mass spectrometric ions.

<b>No.</b>	<b>Odorant</b>	<b>DOL (ppbv)</b>	<b>Monitored ion <i>m/z</i></b>
1	n-Butylamine	0.5	30, 73
2	sec-Butylamine	0.5	44, 58, 18
3	tert-Butylamine	0.5	58, 41
4	Diethylamine	0.5	58, 30
5	Di isopropylamine	0.5	44, 86
6	Dimethylamine	0.5	44, 45
7	Dipropylamine	0.5	72, 30
8	Ethylamine	0.5	30, 28
9	Isobutylamine	0.5	30, 73
10	Isopropylamine	0.5	44, 42
11	Propylamine	0.5	30, 59
12	Tri ethylamine	0.5	86, 58
13	Trimethylamine	0.2	58, 59
14	Ammonia	0.5	17
15	Indole	0.5	117, 90
16	Skatole	0.2	130, 131
17	Methylamine	0.5	30, 31, 28
18	Formaldehyde	0.5	30, 29, 28
19	Acetaldehyde	0.5	29, 44
20	Propionaldehyde	0.5	58, 29
21	Crotonaldehyde	0.5	39, 41, 70
22	n-Butyraldehyde	0.5	44, 43, 72
23	iso-Butylaldehyde	0.2	43,41,72
24	Benzaldehyde	0.5	106, 105, 77
25	Isovaleraldehyde	0.2	44, 43, 58
26	Valeraldehyde	0.2	44, 58
27	o-Tolualdehyde	0.5	120, 119, 91
28	m-Tolualdehyde	0.5	120, 119, 91
29	p-Tolualdehyde	0.5	121, 119, 91
30	2,5-Dimethylbenzaldehyde	0.5	134,133,105
31	Acetone	0.5	43, 58
32	Benzene	0.5	78, 77
33	Tetrachloroethylene	0.5	166, 164
34	Toluene	0.5	91, 92

<b>35</b>	o-Xylene	0.5	91, 106
<b>36</b>	m-Xylene	0.5	91, 106
<b>37</b>	p-Xylene	0.5	91, 106
<b>38</b>	Acetic ester	0.5	43, 45, 60
<b>39</b>	Butanoic ester (Butyric ester)	0.2	60, 73
<b>40</b>	Heptanoic ester	0.5	60, 73
<b>41</b>	Hexanoic ester	0.5	60, 73
<b>42</b>	iso-Hexanoic ester	0.2	57, 74, 43
<b>43</b>	2-Methylbutyric ester	0.5	74, 57
<b>44</b>	Isovaleric ester	0.2	60, 43
<b>45</b>	Isobutyric ester	0.5	43, 73
<b>46</b>	2-Methylvaleric ester	0.5	74, 43
<b>47</b>	3-Methylvaleric ester	0.5	60, 41
<b>48</b>	4-Methylvaleric ester	0.5	57, 74
<b>49</b>	octanoic ester	0.5	60, 73
<b>50</b>	Valeric ester	0.2	60, 73
<b>51</b>	Propionic ester	0.5	74, 45, 28
<b>52</b>	2-ethylhexanoic ester	0.5	73, 88
<b>53</b>	Nonanoic ester	0.5	60, 73
<b>54</b>	n-Butyl mercaptan	0.2	41, 56, 27
<b>55</b>	tert-Butyl mercaptan	0.2	41, 56, 27
<b>56</b>	Carbon disulfide	0.5	76, 44
<b>57</b>	Carbonyl sulfide	0.5	60, 32
<b>58</b>	Diethyl disulfide	0.5	122, 66, 94
<b>59</b>	Diethyl sulfide	0.2	75, 90
<b>60</b>	Dimethyl disulfide	0.5	94, 79
<b>61</b>	Dimethyl sulfide	0.5	62, 47, 45
<b>62</b>	2,5-Dimethylthiophene	0.5	111, 112, 97
<b>63</b>	Ethyl mercaptan	0.2	62, 29, 47
<b>64</b>	Ethyl methyl sulfide	0.5	61, 76
<b>65</b>	2-Ethylthiophene	0.5	97, 112
<b>66</b>	Hydrogen sulfide	0.2	34, 33, 32
<b>67</b>	Isobutyl mercaptan	0.2	41, 43, 90
<b>68</b>	Isopropyl mercaptan	0.2	43, 76
<b>69</b>	Methyl mercaptan	0.2	47, 48
<b>70</b>	3-Methyl thiophene	0.5	97, 98
<b>71</b>	n-Propyl mercaptan	0.2	76, 43, 47
<b>72</b>	Tetrahydrothiophene	0.5	60, 88

<b>73</b>	Thiophene	0.5	84, 58
<b>74</b>	Sulfur dioxide	0.5	64, 48

Appendix 2. Removal Efficiency of DO Units from Stonecutters Island (SC) and Shatin (ST).

Removal Efficiency (RE) of DO units from Stonecutters Island (SC) and Shatin(ST)						
Stonecutters Island				Shatin		
DOU 2 #3	Sept. 26, 2013		RE %	DOU 12 #1	Sept. 24, 2013	
	inlet	Outlet		inlet	Outlet	RE %
Total Odorants	642.5	322.8	849.8	1171.2	302.3	74.2
Hydeogen sulfide	2191.5	179.1	91.8	8230.6	127.9	98.4
Odalog reading ppm	/	/	/	/	/	/
DOU2 #4 Oct. 17, 2013				Biotrickling	Filter #2	Oct. 7, 2013
	inlet	Outlet	RE %	inlet	Outlet	RE %
Total Odorants	11195.2	370.2	96.7	10283.9	461.0	95.5
Hydeogen sulfide	11204.6	215.2	98.1	10150.1	314.6	96.9
Odalog reading ppm	11.6	0.3	97.4	10.7	0.3	97.2
DOU2 #1 Oct. 28, 2013				DO 4 #1	Oct.21, 2013	
	inlet	Outlet	RE %	inlet	Outlet	RE %
Total Odorants	1017.5	172.6	83.0	984.9	569.8	42.1
Hydeogen sulfide	6120.9	218.7	96.4	33117.1	379.4	98.9
Odalog reading ppm	7.2	0.3	95.8	41.1	0.4	99.0
DOU2 #2 Nov. 21, 2013				DO2 #2	Nov. 12, 2013	
	inlet	Outlet	RE %	inlet	Outlet	RE %
Total Odorants	956.6	357.0	62.7	1190.2	636.0	46.6
Hydeogen sulfide	5110.2	182.7	96.4	4982.6	13.6	99.7
Odalog reading ppm	5.8	0.2	96.6	5.7	0.1	98.2
DOU1 #3 Dec. 2, 2013				DO12 #1	Nov. 25, 2013	
	inlet	Outlet	RE %	inlet	Outlet	RE %
Total Odorants	912.6	399.6	56.2	1373.4	802.9	41.5
Hydeogen sulfide	6429.1	7.8	99.9	2744.8	7.5	99.7
Odalog reading ppm	7.2	0.0	100.0	2.6	0.1	96.2

<b>DOU1 #3 Dec. 19, 2013</b>				<b>DO2 Dec.12, 2013</b>		
	<b>inlet</b>	<b>Outlet</b>	<b>RE %</b>	<b>inlet</b>	<b>Outlet</b>	<b>RE %</b>
Total Odorants	1307.6	359.9	72.5	1373.9	859.4	37.4
Hydeogen sulfide	3275.2	0.1	100.0	9859.6	14.7	99.9
Odalog reading ppm	3.9	0.1	97.4	10.1	0.1	99.0
<b>DOU1 #3 Jan. 9, 2014</b>				<b>DO8 Jan. 2, 2014</b>		
	<b>inlet</b>	<b>Outlet</b>	<b>RE %</b>	<b>inlet</b>	<b>Outlet</b>	<b>RE %</b>
Total Odorants	1227.9	719.6	41.4	36438.4	885.5	97.6
Hydeogen sulfide	2751.4	111.2	96.0	2341.5	193.4	91.7
Odalog reading ppm	3.2	0.1	96.9	2.5	0.2	92.0
<b>DOU2 #3 Jan.24, 2014</b>				<b>DO4 Jan. 2, 2014</b>		
	<b>inlet</b>	<b>Outlet</b>	<b>RE %</b>	<b>inlet</b>	<b>Outlet</b>	<b>RE %</b>
Total Odorants	1468.0	594.8	59.5	1312.8	702.2	46.5
Hydeogen sulfide	2644.5	322.2	87.8	64758.9	9066.2	86.0
Odalog reading ppm	3.0	0.4	86.7	70.0	15.0	78.6
<b>DOU2 #3 Feb.27, 2014</b>				<b>DO 12 #1 Jan.16, 2014</b>		
	<b>inlet</b>	<b>Outlet</b>	<b>RE %</b>	<b>inlet</b>	<b>Outlet</b>	<b>RE %</b>
	901.6	461.4	48.8	1312.8	758.0	42.3
	13248.8	10.7	99.9	1124.7	13.3	98.8
	15.0	0.0	100.0	1.3	0.1	92.3
Total Odorants	<b>DOU1 #3</b>	<b>March 13, 2014</b>		<b>DO 2 Feb. 21, 2014</b>		
Hydeogen sulfide	<b>inlet</b>	<b>Outlet</b>	<b>RE %</b>	<b>inlet</b>	<b>Outlet</b>	<b>RE %</b>
Total Odorants	1457.2	514.3	64.7	1659.6	758.0	54.3
Hydeogen sulfide	3577.5	317.6	91.1	3785.2	13.4	99.6
Odalog reading ppm	4.9	0.3	93.9	4.1	0.0	100.0
<b>DOU2 #4 March 26, 2014</b>				<b>Biotrickling DO 2 March 6, 2014</b>		
	<b>inlet</b>	<b>Outlet</b>	<b>RE %</b>	<b>inlet</b>	<b>Outlet</b>	<b>RE %</b>
Total Odorants	1407.0	605.9	56.9	945.8	311.0	67.0
Hydeogen sulfide	2752.4	5.2	99.8	2237.4	10.7	99.5
Odalog reading ppm	2.9	0.0	100.0	2.4	0.1	95.8

	DOU1 #2 April 9, 2014			DO 4 #2 March 19, 2014		
	inlet	Outlet	RE %	inlet	Outlet	RE %
Total Odorants	872.7	298.6	65.8	987.0	550.3	44.2
Hydeogen sulfide	3950.1	6.6	99.8	1987.4	97.8	95.1
Odalog reading ppm	4.2	0.0	100.0	2.1	0.1	95.2
<b>Biotrickling DO 12 #1 April 2, 2014</b>						
	inlet	Outlet	RE %			
	1126.1	536.7	52.3			
	2451.0	11.0	99.6			
	2.8	0.0	100.0			
<b>DO 2 April 14, 2014</b>						
	inlet	Outlet	RE %			
	1291.6	646.0	50.0			
	9754.3	451.7	95.4			
	10.5	0.5	95.2			

### Appendix 3. Radial plots for odorants profile presentation

