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03/26/2008 03:49 AM

To wkreagen@mmm.com
cc JNewsted@entrinx.com
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bcc
Subject Re: Entrix Consulting

Bill:

I will forward the opportunities to you. Then you decide how you would like to proceed. I am attaching two papers that have been assigned to me to review. If you want to take them over, I will write to the journals and tell them that I can no longer review them and suggest that they be referred to whomever in 3M who is appropriate. Some journals will allow this, but others, for conflict of interest issues, will not allow an industry to review a paper about one of their products. That is where I came in for Dale. Since we had been set up as academic experts, about half of the papers published in the area in any given year came to me (continue to come to me) for review. In time sheets, I always listed these reviews as literature searches so that there was no paper trail to 3M.



I have attached the two papers that came this week. One from Environmental Pollution and one from ES&T.

Let me know if you want to take over the reviews of these papers and I will decline.

Sincerely,

John P. Giesy

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Environmental Pollution
Manuscript Draft

Manuscript Number: ENVPOL-D-08-00116

Title: Perfluorooctanoic Acid and Perfluorooctane Sulfonate in the Sediment of the Roter Main River, Bayreuth, Germany

Article Type: Full Paper (max. 5000 words)

Keywords: perfluorooctanoic acid; perfluorooctane sulfonate; sediment; aquatic environment

Abstract: Perfluorooctanoic acid (PFOA) and perfluorooctane sulfonate (PFOS) are widely distributed in aquatic ecosystems. Their sources are known but few studies about their accumulation potential in river sediments exist.

The aim of the study is to assess the concentrations of PFOA and PFOS in sediments in relation to their levels in river water receiving effluent from a waste water treatment plant (WWTP). PFOS accumulates by a factor of about 40 relative to river water, PFOA only up to three-fold. In contrast to previous suggestions, enrichment on sediment is not correlated to the total organic carbon contents.

3M_MN00110701

1 **Perfluorooctanoic Acid and Perfluorooctane Sulfonate in the Sediment**
2 **of the Roter Main River, Bayreuth, Germany**

3

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24 **Keywords:** perfluorooctanoic acid; perfluorooctane sulfonate; sediment; aquatic
25 environment

26

ABSTRACT

27 Perfluorooctanoic acid (PFOA) and perfluorooctane sulfonate (PFOS) are
28 widely distributed in aquatic ecosystems. Their sources are known but few studies about
29 their accumulation potential in river sediments exist. The aim of the study is to assess
30 the concentrations of PFOA and PFOS in sediments in relation to their levels in river
31 water receiving effluent from a waste water treatment plant (WWTP). PFOS
32 accumulates by a factor of about 40 relative to river water, PFOA only up to three-fold.
33 In contrast to previous suggestions, enrichment on sediment is not correlated to the total
34 organic carbon contents.

35

CAPSULE

36 Rivers sediments constitute a sink of perfluorinated surfactants released from the waste
37 water treatment plant.

38

39

INTRODUCTION

40 Over the last 50 years, perfluorochemicals (PFCs) such as perfluorooctanoic acid
41 (PFOA) and perfluorooctane sulfonate (PFOS) have been widely used both in industry
42 and as components of consumer products (Kissa, 2001; Schulz et al., 2003). Due to their
43 specific physical-chemical properties, especially their chemical and thermal stability,
44 and their low surface free energy, especially their water- and fat-repellent properties
45 (Kissa, 2001), they have been used in numerous applications. At the same time, their
46 persistence and potential to bioaccumulate have led to their wide-spread presence in the
47 global environment (Prevedorou et al., 2006; Houde et al., 2006). Several direct and
48 indirect sources of PFCs-emission to the environment have been reported, such as
49 manufacturing processes in the polymer and electronics industries (Prevedorou et al.,

50 2006), in the degradation of volatile precursors (Ellis et al., 2004; Dinglasan et al.,
51 2004; Lange, 2000-2002), or in the release of treated waste waters (Becker et al., 2008a-
52 b; Boulanger et al., 2005; Schulz et al. 2006a, 2006b; Sinclair and Kannan, 2006).

53 Sediments have been suggested as one of two final sinks of PFCs, the other
54 being the deep oceans (Prevedorou et al., 2006). Most of the earlier studies have
55 focused on water and biological matrices; little information is available on PFC
56 concentrations in soils and sediments. Relatively low concentrations of PFOA and
57 PFOS were found in sediments collected from four Rivers in the San Francisco Bay,
58 USA (Higgins et al., 2006), from the Tidal Flat Areas of Ariake Sea (Nakata et al.,
59 2006) or from the rivers Kamo, Uji, and Tanjin in Japan (Senthilkumar et al, 2007).

60 The aim of this study was to determine the accumulation potential of PFOA and
61 PFOS in sediments relative to the water of the river Roter Main; a well quantified
62 source of these compounds into the river is the municipal waste water treatment plant
63 (WWTP) of Bayreuth (Becker et al, 2008 a-b).

64 MATERIALS AND METHODS

65 Chemicals and Equipment

66 PFOA (95 %, Lancaster Eastgate, UK), [1, 2- ¹³C₂]-PFOA (98 %, 10 mg, Perkin
67 Elmer, Boston, USA), perfluorooctane sulfonate potassium salt (98 %, Fluka, Buchs,
68 Germany), [1, 2, 3, 4-¹³C₄]-perfluorooctane sulfonate sodium salt (99 %, 50 µg/mL-
69 solution in methanol, Campro Scientific, Berlin, Germany), acetic acid (100 %, Merck,
70 Darmstadt, Germany), ammonium acetate (99.0 %, Fluka, Buchs, Germany), methanol,
71 and acetonitrile (picograde, Promochem, Wesel, Germany) were used as obtained. The
72 equipment was pre-cleaned as described previously (Weremiuk et al., 2006); Teflon
73 equipment was avoided.

74 Sampling Location and Collection

75 The investigation area was the river Roter Main flowing through Bayreuth,
76 Upper Franconia, Germany, which has an average daily flow of 270 000 m³ day⁻¹ and
77 receives treated waste waters of industrial, commercial and domestic origin from the
78 municipal WWTP, having a daily flow of 40 000 m³ and serving a population of 72
79 000 inhabitants. Based on our previous studies it was estimated that about 1.2 ± 0.5 g
80 PFOA and 4.7 ± 2.3 g PFOS are daily released from the plant into the river (Becker et
81 al, 2008 a-b).

82 On 19 October 2006, sediment samples were collected at four different
83 locations: a) 1 km upstream, b) 50 m downstream, c) 500 m downstream, and d) 1 km
84 downstream the WWTP. At each location, 11 individual samples (Fig. 1) from the
85 upper sediment (~ 15 cm) were collected with a polypropylene (PP) tube (Ø = 10 cm)
86 and transferred into 250-mL PP-bottles. Water samples (n = 3) were also collected at
87 each location with 2-L PP-bottles.

88 **Sample Preparation and Analysis**

89 Sediment samples were weighed into aluminium boxes precleaned with hexane
90 and methanol, freeze-dried, sieved (0.63 mm mesh), transferred into clean 50-mL PP-
91 bottles, and stored at room temperature until analysis. Their total organic carbon (TOC)
92 content was determined and the samples were extracted as described previously (Becker
93 et al., 2008 b).

94 Mixed sediment samples were also prepared for each location by weighing 1 g
95 of each of the 11 individual samples of the respective location into a 50 mL PP-
96 centrifuge tube. Each sample was thoroughly mixed and extracted in triplicate as
97 described above.

98 Briefly, 1 g of sediment was weighed into a new, clean 50-mL PP-centrifuge
99 tube and 50 µL of a standard solution containing 10 µg L⁻¹ each of ¹³C-PFOA and ¹³C-

100 PFOS were added. Each sample was sonicated in 10 mL 1 % (vol.) aqueous acetic acid,
101 centrifuged, and the supernatant was collected. The pellet was re-suspended in 2.5 mL
102 of a mixture of methanol and 1 % aqueous acetic acid (90:10, vol-%), sonicated,
103 centrifuged again, and the supernatant was combined with the first one. The procedure
104 was repeated one more time to yield 25 mL extract. A final wash with acetic acid (1 %,
105 10 mL) yielded a total volume of 35 mL. The analytes were extracted by solid phase
106 extraction (SPE) using a C18 cartridge (200 mg, 6.0 mL, Oasis HLB Waters Corp.,
107 Milford, USA), eluted with methanol, the solvent was evaporated, and the residue was
108 redissolved in 0.5 mL of a mixture (50:50, vol.) of aqueous ammonium acetate (10
109 mmol/L, pH = 4.1) and acetonitrile. The solution was filtered and transferred to PP-snap
110 ring vials (0.75 mL) with polyethylene (PE) caps, and analysed by HPLC-ESI-MS/MS
111 (Weremiuk et al., 2006).

112 Water samples were prepared and analysed as described previously (Weremiuk
113 et al., 2006, Becker et al. 2008 a).

114 **Quantification**

115 For calibration, stock solutions of ^{13}C -PFOA, ^{13}C -PFOS, PFOA, PFOS and two
116 working standard solutions, one containing $10\ \mu\text{g L}^{-1}$ of each ^{13}C -PFOA and ^{13}C -PFOS,
117 the other $10\ \mu\text{g L}^{-1}$ of each non-labelled PFOA and PFOS, were prepared as described
118 previously (Becker et al., 2008 a-b).

119 For quantitative analysis of sediment extracts, standard solutions containing non-
120 labelled PFOA and PFOS in the range from 0.5 to $2.5\ \mu\text{g L}^{-1}$ and $1\ \mu\text{g L}^{-1}$ of each ^{13}C -
121 labelled analyte were used for calibration. For analysis of water sample extracts,
122 standard solutions containing non-labelled PFOA and PFOS in a range from 1 to 8
123 $\mu\text{g L}^{-1}$ and $1\ \mu\text{g L}^{-1}$ of each ^{13}C -PFOA and ^{13}C -PFOS were used. Calibration curves
124 were constructed by plotting analyte and internal standard peak area ratios versus

125 analyte concentrations; regression coefficients were higher than 0.995. The limits of
126 quantification (LOQ, signal to noise ratio 7) for sediments were 25 ng kg⁻¹ PFOA and
127 50 ng kg⁻¹ PFOS, for river water 0.06 ng L⁻¹ PFOA and 0.12 ng L⁻¹ PFOS. Procedural
128 blanks for sediment samples were below the limit of detection (LOD), for water it was
129 0.015 ng PFOA, PFOS was below LOD.

130 **RESULTS AND DISCUSSION**

131 PFOA concentrations in sediment samples were frequently below LOQ
132 especially at the location upstream the WWTP, but also reached 175 ng kg⁻¹ dry weight
133 (Table 1). Obviously the variability between individual samples is rather high, the being
134 widest observed 0.5 km downstream the WWTP, potentially reflecting incomplete
135 mixing of waste and river waters at this location.

136 PFOS concentrations ranged from <50 and 570 ng kg⁻¹ dw, with the highest
137 variations 500 m downstream the WWTP (Table 1). PFOS was up to 17-fold higher
138 than PFOA, due to its stronger adsorption potential. A mixed sample of each location
139 was prepared, extracted and analysed. Results of the mixed samples analysis (Table 2)
140 were in a good agreement with the average values calculated for each location (Table
141 1), and confirmed that concentrations of the analytes downstream the plant were
142 significantly increased.

143 TOC of sediment samples ranged between 0.06 and 0.78 % (Table 1). PFOA and
144 PFOS concentrations in river water were between 10 and 23 ng L⁻¹, and 1.7 and 16
145 ng L⁻¹, respectively (Table 2).

146 PFOA concentrations in sediment samples downstream the WWTP were about
147 3-fold higher than upstream, but lower than reported for four rivers located in the San
148 Francisco Bay, USA (< LOD – 1300 ng kg⁻¹) (Higgins et al., 2005), in Tidal Flat Areas
149 of the Ariake Sea (840 – 1100 ng kg⁻¹) (Nakata et al., 2006) or in rivers Kamo, Uji and

150 Tenjin in Japan (1300 – 3900 ng kg⁻¹) (Senthilkumar et al., 2007). The sediment
151 concentrations were about 3-fold higher than in the respective water sample.

152 PFOS sediment concentrations were comparable to those of the Ariake Sea (90 –
153 140 ng kg⁻¹) (Nakata et al., 2006) or in four rivers from the San Francisco Bay (160 –
154 230 ng kg⁻¹) (Higgins et al., 2005), but lower than reported for the rivers Tenjin or
155 Osaka in Japan (3800 – 11000 ng kg⁻¹) (Senthilkumar et al., 2007). The sediment
156 concentrations were up 40-fold higher than in the respective water samples.

157 In the present study a correlation of the PFC content of the sediments to TOC
158 was not found, although this has been suggested previously (Higgins & Luthy, 2006).
159 According to Johnson et al. (2007), it is likely that adsorption of PFOA and PFOS is
160 also partially controlled by electrostatics meaning that also inorganic materials will
161 influence their fate and transport in aquatic system.

162 CONCLUSIONS

163 Treated waste water released from local WWTPs is a main source of PFOA and
164 PFOS in rivers. Once released, PFCs partition into the sediment. Higgins et al. (2007)
165 have suggested that they are readily bioavailable; thus, studies with fish from such
166 rivers are required to assess to which extent these compounds enter the aquatic food
167 chain.

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236

237

FIGURES AND TABLES

238 **Figure 1.** Sediment Sampling Scheme at the four locations of the river bed.

239

240 **Table 1.** Concentrations of PFOA, PFOS [ng kg^{-1}] and TOC [%] in river sediment
241 samples, Roter Main near Bayreuth, 19 October 2006. (UpS – upstream the WWTP,
242 DwS – downstream the WWTP, $\text{LOQ}_{\text{PFOA}} = 25 \text{ ng kg}^{-1}$, $\text{LOQ}_{\text{PFOS}} = 50 \text{ ng kg}^{-1}$). Mean
243 is calculated using a half of the LOQ.

244

245 **Table 2.** Comparison of PFOA and PFOS concentrations in water [ng L^{-1}] and sediment
246 (mixed sample) [ng kg^{-1}] at four locations (Sampling: 19 October 2006, UpS – upstream
247 the WWTP, DwS – downstream the WWTP).

248

249

Table I.

	L1	L2	L3	M1	M2	M3	M4	M5	R1	R2	R3	Mean
0.1 km UpS												
PFOA	<25	<25	<25	<25	<25	48 (6)	52 (4)	<25	49 (16)	26 (8)	46 (17)	27 ± 18
	Mean L1-L3		<25	Mean M1-M5		30	Mean RI-R3		40			
PFOS	<50	66 (1)	290 (12)	50 (10)	230 (8)	80 (9)	95 (14)	116 (9)	<50	91 (6)	76 (10)	105 ± 85
	Mean L1-L3		130	Mean M1-M5		65	Mean RI-R3		64			
TOC	0.504	0.128	0.294	0.056	0.177	0.279	0.075	0.071	0.072	0.058	0.076	
0.05 km Dws												
PFOA	<25	<25	120 (16)	-	<25	70 (4)	41 (20)	135 (17)	<25	106 (14)	175 (5)	70 ± 60
	Mean L1-L3		50	Mean M1-M5		65	Mean RI-R3		100			
PFOS	264 (2)	218 (15)	120 (10)	-	153 (14)	415 (3)	130 (3)	381 (6)	183 (13)	331 (7)	506 (10)	280 ± 120
	Mean L1-L3		235	Mean M1-M5		270	Mean RI-R3		340			
TOC	0.098	0.084	0.073	-	0.062	0.095	0.156	0.076	0.784	0.380	0.241	
0.5 km Dws												
PFOA	<25	91 (3)	60 (2)	64 (11)	48 (4)	142 (10)	168 (6)	141 (6)	34 (24)	<25	80 (12)	85 ± 60
	Mean L1-L3		76	Mean M1-M5		110	Mean RI-R3		40			
PFOS	56 (19)	226 (2)	170 (10)	348 (7)	72 (4)	227 (13)	378 (5)	235 (11)	537 (10)	179 (4)	301 (4)	250 ± 150
	Mean L1-L3		150	Mean M1-M5		250	Mean RI-R3		350			
TOC	0.133	0.084	0.140	0.105	0.084	0.089	0.061	0.086	0.200	0.260	0.151	
1 km Dws												
PFOA	<25	33 (3)	55 (2)	<25	53 (13)	77 (10)	79 (12)	105 (20)	<25	72 (2)	50 (14)	50 ± 30
	Mean L1-L3		34	Mean M1-M5		65	Mean RI-R3		45			
PFOS	176 (7)	237 (9)	100 (5)	82 (15)	143 (29)	92 (6)	296 (9)	170 (2)	348 (10)	264 (10)	307 (5)	200 ± 90
	Mean L1-L3		171	Mean M1-M5		160	Mean RI-R3		306			
TOC	0.082	0.077	0.109	0.081	0.111	0.082	0.097	0.090	0.348	0.096	0.097	

Table 2.

Sampling location	PFOA			PFOS		
	Water	Sediment	Ratio (Sediment/ Water)	Water	Sediment	Ratio (Sediment/ Water)
1 km UpS WWTP	10 ± 1	18 ± 5	1.8	1.7 ± 0.3	72 ± 14	42
0.05 km DwS WWTP	23 ± 2	68 ± 2	3.0	16 ± 0.3	310 ± 20	19
0.5 km DwS WWTP	23 ± 1	40 ± 14	1.7	14 ± 0.5	230 ± 20	17
1 km DwS WWTP	23 ± 3	35 ± 9	1.5	11 ± 0.2	240 ± 22	22

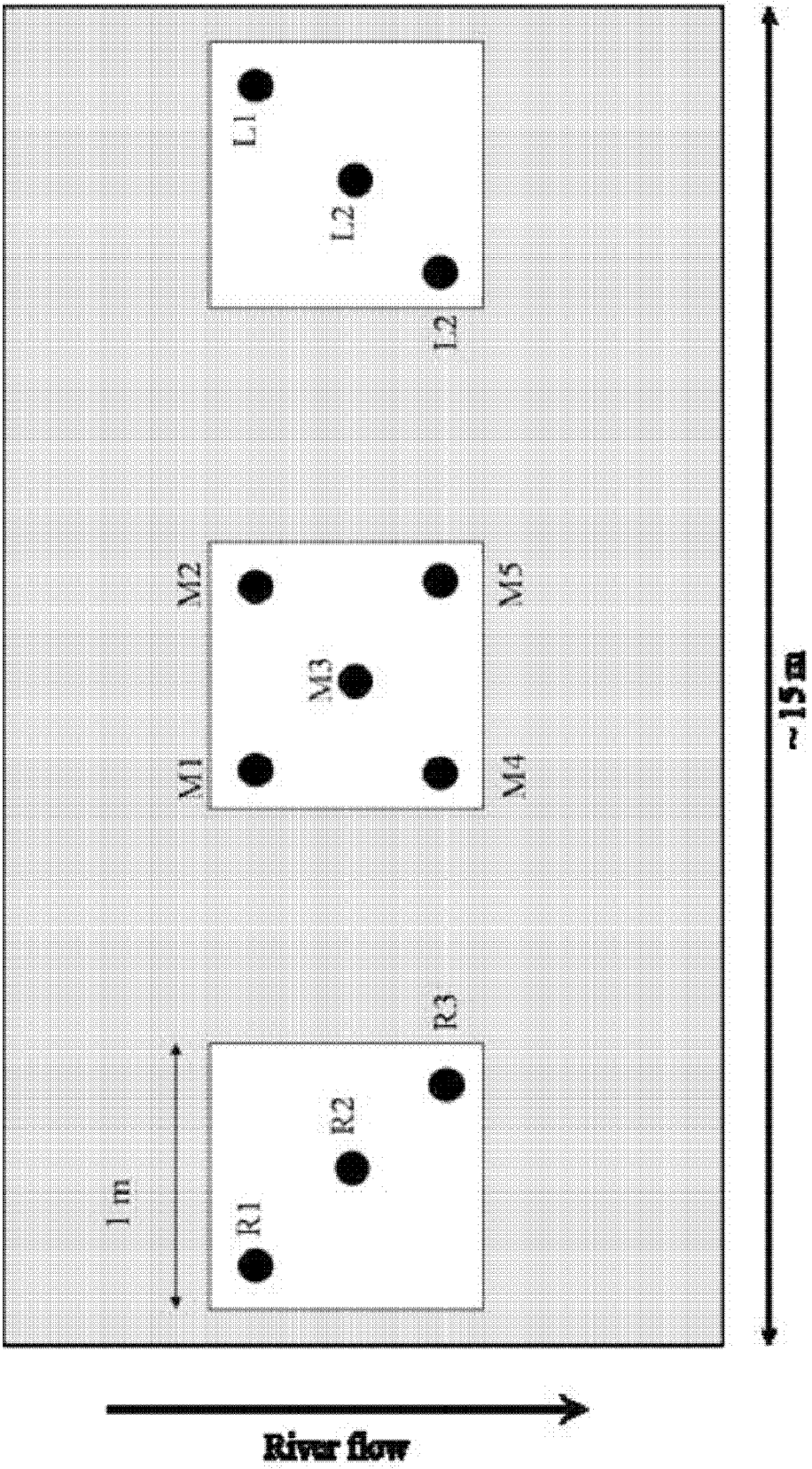


Figure 1
 Click here to download high resolution imageFigure 1.tif

Table 1. Instrument performance standards used to account for matrix effects for each analyte and MS/MS Multiple Reaction Monitoring Parameters are provided. For analytes in which two transitions were monitored the first transition listed was used for quantitation.

Analyte	Performance Standard	Mass Transition	Cone voltage	Collision energy
Perfluoroheptanoate (PFHpA)	1,2- ¹³ C perfluorooctanoate	362.9 → 318.8	10	15
Perfluorooctanoate (PFOA)	1,2- ¹³ C perfluorooctanoate	412.9 → 368.8 412.9 → 168.8	10	15
Perfluorononanoate (PFNA)	1,2,3,4,5- ¹³ C perfluorononanoate	462.8 → 418.8 462.8 → 218.8	12 16	18 18
Perfluorodecanoate (PFDA)	1,2- ¹³ C perfluorodecanoate	512.8 → 468.9 512.8 → 218.9	15 15	18 18
Perfluoroundecanoate (PFUA)	1,2- ¹³ C perfluorodecanoate	562.9 → 518.9 562.9 → 268.8	15	15
Perfluorododecanoate (PFDoDA)	1,2- ¹³ C perfluorodecanoate	612.8 → 568.8 612.8 → 318.9	12	30
Perfluorotetradecanoate (PFTeDA)	1,2- ¹³ C perfluorodecanoate	712.9 → 668.9 712.9 → 168.8	17	22
Perfluorooctane sulfonate (PFOS)	¹⁸ O ₂ perfluorooctane sulfonate	498.9 → 98.9 498.9 → 79.9	50	60
6:2 Fluorotelomer unsaturated carboxylate (6:2 FTUCA)	2H-Perfluoro-[1,2- ¹³ C ₂]-2-octenoate	356.9 → 292.9	15	17
8:2 Fluorotelomer unsaturated carboxylate (8:2 FTUCA)	2H-Perfluoro-[1,2- ¹³ C ₂]-2-decenoate	457 → 392.9	18	20
10:2 Fluorotelomer unsaturated carboxylate (10:2 FTUCA)	2H-Perfluoro-[1,2- ¹³ C ₂]-2-dodecenoate	557 → 493	20	25