The first report of urine over-marking of oestrous female dung by a male white rhino

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Abstract

Although observed in other Perrisodactyla, urine over-marking in white rhinos has not been described. Using a single opportunistic sighting, we were able to collect two dung samples from one oestrous female white rhino; one unmarked and one over-marked with male urine. We hypothesised that the function of over-marking by the male was for oestrous concealment (i.e. odour masking), as observed in horses. As dung from oestrous female white rhinos emits a higher proportion of alkanes than non-oestrous dung, we expected the proportion of alkanes emitted from oestrous dung to decrease after over-marking. In contrast, we found that after over-marking the proportion of alkanes emitted increased. We suggest that the function of urine over-marking in white rhinos could be to conceal all signals of reproductive condition, so that neither oestrous nor non-oestrous signals are recognisable, or that a signal is added to indicate that the female has been mated.

Keywords *Ceratotherium simum;* odour masking; oestrus concealment.

Introduction

Over-marking occurs when one individual places its scent mark on top of a scent mark from another individual (Ferkin & Pierce, 2007). There are several hypotheses regarding the function of over-marking. One is that over-marking gives an advantage to the individual over-marking in the form of physically masking the initial odour (Johnston et al., 1994), provoking competition (Ferkin et al., 2004), or showing social rank (Rich & Hurst, 1999). This hypothesis pertains mainly to same-sex over-marking, and suggests that odours on top are investigated more frequently or given more status. For example, meadow voles (*Microtus pennsylvanicus*) and prairie voles (*Microtus ochrogaster*) exposed to an over-mark were able to distinguish the two different signals and preferred the over-mark (Ferkin et al., 2001).

A second hypothesis is that over-marking is a form of mate attraction (Ferkin & Pierce, 2007). As such, individuals over-mark the scent marks of reproductive individuals of the opposite-sex in order to facilitate mating (i.e. over-marking occurs before mating). Over-marking pre-mating has been reported in Cape mountain zebras (*Equus zebra zebra;* Penzhorn (1984)) and meadow voles (Ferkin et al., 2004). Third, over-marking is thought to be a form of mate guarding, whereby over-marking masks or devalues the odour of the initial mark (Ferkin & Pierce, 2007). This hypothesis predicts that individuals will over-mark post-reproduction in order to hide the reproductive status of an individual or indicate that the individual has been mated (Ferkin & Pierce, 2007). For example, klipspringers (*Oreotragus oreotragus*) mark with secretions from their preorbital gland, and males over-mark female scent marks as a form of chemical mate guarding (Roberts & Dunbar, 2000). Yet, it is also possible that if the signal is over-marked prior to mating, it may reduce competition from rival males that encounter the mark (Kimura, 2001).

Over-marking as a function of oestrus concealment was suggested by Penzhorn (1984) with regard to the urine over-marking of male Cape mountain zebras on female urine and dung. Recent evidence from feral horses (*Equus caballus*) indicates a similar function, where over-marking the dung of oestrous females with male urine changes the odour profile such that it is more similar to the odour of non-oestrous female dung (Kimura, 2001). Recent evidence also suggests that intrasexual over-marking in female equids (African wild ass (*Equus africanus*), Grevy's zebra (*Equus grevyi*), plains zebra (*Equus quagga*), and mountain zebra (*Equus zebra*))

helps maintain social bonds and group cohesion (Tučková et al., 2018). Ultimately, over-marking appears to be an important behaviour in Perrisodactyla.

White rhinos (*Ceratotherium simum*) defecate in communal middens (i.e. latrines) where they deposit and obtain information, including the territorial or oestrous state of the depositor (Marneweck et al., 2017a, 2018). In general, the dung of white rhino females in oestrus emits a higher proportion of hydrocarbon alkanes than non-oestrous female dung (Marneweck et al., 2017a, unpublished data). Further, the emission of the alkane 2,6-dimethylundecane from female white rhino dung is an important indicator of oestrus (Marneweck et al., 2017a), where oestrous dung odours contain a larger proportion than non-oestrous (median proportion [interquartile range] non-oestrous 0.0005 [0.0014] N = 23, oestrous 0.0030 [0.0124 N = 7; Marneweck et al., 2017a, unpublished data).

Over-marking of dung in middens has been observed when a territorial male is being challenged (Owen-Smith, 1975; Marneweck, pers. obs.). In this situation, the territorial male places his dung on top of the challenger's dung. However, male over-marking of female dung has not been reported in white rhinos, although observed in other Perrisodactyla (Penzhorn, 1984; Kimura, 2001). In addition, prior to copulation, female white rhinos emit repeated little squirts of urine (Owen-Smith, 1973), which may be a form of oestrus advertisement. However, as with the dung of these oestrous females, over-marking of this urine by males has also not been reported.

An opportunistic sighting allowed us to collect two dung samples from one oestrous female white rhino; one unmarked and one over-marked with male urine. Due to the polygynous mating system of white rhinos (White et al., 2007), it is unlikely that inter-sexual over-marking is for mate attraction. Males establish territories within which they actively pursue oestrous females that move through. Despite territorial males having primary access to these females, some sneaky copulations by subordinate males resident within the territories can take place (Guerier, 2012). As a result, we hypothesised that the function of male over-marking of female dung was oestrous concealment as a form of chemical mate guarding, similar to what has been observed in horses (i.e. to mask the oestrous signal; Kimura (2001)). Thus, we predicted that (1) the overall proportion of alkanes emitted by the overmarked dung would decrease, and specifically (2) the proportion of 2,6-dimethylundecane would decrease to levels found in the odour of non-oestrous female dung.

Methods

We conducted this study in the 896 km^2 Hluhluwe-iMfolozi Park, KwaZulu-Natal, South Africa. During our two-year study, we observed, on foot, over 200 separate defecation events, with at least ten of these from oestrous females. Despite the sample size, we only recorded urine over-marking by males once. Here, we opportunistically collected two dung odour samples from a single wild, free-ranging female white rhino in oestrus during June 2012. The first sample was collected at 15:25, and the second collected at 15:50 from a separate defecation ~60 m away. At the second defecation, a territorial male sniffed the dung pile, performed flehmen, and then over-marked the female's dung with his urine. From several former observations of this male over the larger study period in 2012, we established that he was a territory holder via the due to the performance of territorial behaviours he performed (i.e. dung kicking and spray urination) (Owen-Smith, 1971; Kretzschmar et al., 2001; Marneweck, pers. obs.). However, while over-marking, his urine was excreted in a stream, as in non-territorial males and females, and not sprayed in a mist, like a territorial male. We did not observe this male interact with any other individuals during the larger study period, nor did we determine the location of his territory boundary boundaries. As such, it is possible that he was outside of his territory, and thus was acting as a subordinate (i.e. not spray urinating), or this event occurred during territory challenge or take-over. Although stream urination is not ordinarily performed by territorial males, they can periodically urinate in a stream (Owen-Smith, 1973).

We identified the female as an adult (>7 years), based on body size and horn development (Hillman-Smith et al., 1986), and she was accompanied by a female calf and a female subadult (approximately three and six years old, respectively). We identified the oestrous state via the behaviour of the male. For white rhinos, there is a consort period of several days where a territorial male will move with an oestrous female. During this time, he follows her closely, restricts her movement beyond his territory boundary, and makes several mounting attempts (Owen-Smith, 1973). There is usually only one successful copulation during this courtship and, subsequently, the male can continue to follow the female loosely but does not attempt to mount further (Owen-Smith, 1973). We observed the male follow the female closely (i.e. within 10 – 20 m), for approximately 80 minutes. During this time, the male attempted to restrict the movement of the female, perform flehmen in response to her dung, and attempted to mount her once. We did not record any vocalisations by either adult.

Although the mate guarding hypothesis predicts that over-marking as a function of mate guarding would occur post-mating, we cannot confirm if the female in question had already mated with the male moving with her, but this is unlikely as males usually cease mounting attempts after successful copulation (Owen-Smith, 1973). Thus, it is possible that he over-marked her dung to reduce competition from other resident males. As the consort period in white rhinos lasts for 1 – 2 weeks, it does not begin at the onset of oestrus. The onset of oestrus is described as regular advances by the male and hiccing vocalisations from the female (Owen-Smith, 1973). The frequency of these behaviours increases until mating occurs, and then the male does not attempt to mount again after successful copulation (Owen-Smith, 1973). As we observed only one mounting attempt (no copulation), and no vocalisations, we assume the female to be in pro-oestrus.

We collected odour samples using a dynamic headspace extraction method (Amirav & Dagan, 1997) to collect air for 25 minutes from approximately 800 g (one bolus) of fresh (<5 minutes old) dung enclosed in a polyacetate bag using a micro-air sampler (Supelco PAS-500) with a realised flow rate of 150 ml/min. The VOCs emitted from the dung were captured in a small thermodesorption trap filled with 1 mg of Tenax® and 1 mg of Carbotrap®. We confirmed that both dung samples were from the same adult female by following her and observing her defecate.

We analysed the thermodesorption traps using gas chromatography-mass spectrometry (GC-MS). We carried out analysis on a Bruker 450 GC with a 30 m x 0.25 mm internal diameter (film thickness 0.25 µm) Varian VF-5ms column, connected to a Varian VF-1ms column (11 m x 0.25 mm internal diameter, film thickness 0.25 µm) coupled to a Bruker 300 quadrupole mass spectrometer in electron-impact ionization mode at 70 eV. Thermodesorption traps were placed in a Varian 1079 injector equipped with a chromatoprobe thermal desorption device. The flow of helium carrier gas was 1 ml min-1. We held the injector at an initial temperature of 250°C for 20 minutes. The split vent was programmed to start with a 10:1 split for 2 minutes and then to switch to splitless mode for 2 minutes to allow for thermal desorption, followed by a 100:1 split after 4.2 minutes to clean the injector. After an initial temperature at 45°C the temperature of the GC oven was increased to 260°C at 7°C min-1 and, after reaching 260°C, held at this temperature for a total run time of 35 minutes. We identified VOCs using Varian Workstation software with the NIST 2011 mass spectral library (NIST/EPA/NIH Mass Spectral Library, data version: NIST 2011; MS search

software version 2.0 d). We verified the identification of VOCs with retention times of authentic standards and published Kovats indices wherever possible (Supplementary Material Table S1).

To compare the odour of the over-marked dung, we created an MDS plot (using the R package *vegan*; Oksanen et al. (2015)) including dung odour samples from both oestrous (N = 3) and non-oestrous (N = 15) females collected during the same season for comparison (Marneweck et al., 2017a, 2017b). Some females may have been mis-identified as non-oestrous due to the fact that they were observed alone, which may explain the inclusion of some non-oestrous markers close to the oestrous core in Figure 1. Alternatively, it may be that oestrous and non-oestrous odours are very similar and, as a result, will overlap. Yet, due to the small sample size of oestrous females in the dry season (N = 3), it makes it difficult to differentiate. As we believe the female in this study to be in pro-oestrus, this may further explain the close relationship with non-oestrous odours, (i.e. it may be a continual drift from non-oestrous, to pro-oestrus, to oestrus, to an-oestrus).

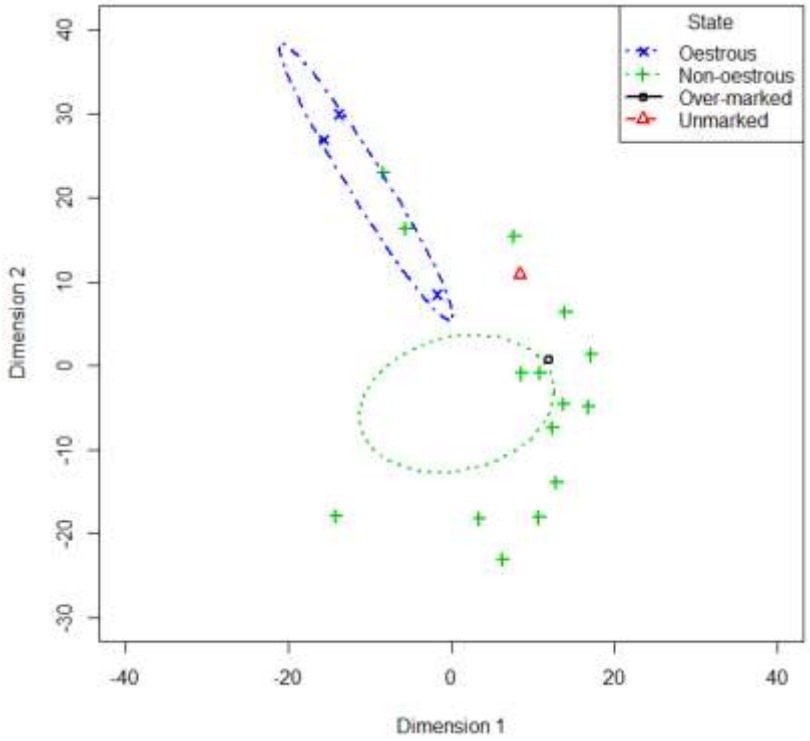

Figure 1. Multidimensional scaling (MDS) plot based on Bray-Curtis similarities of VOCs emitted from female white rhino dung during the dry season. Encompassing circles represent 95% confidence intervals. Stress = 0.15.

Results and Discussion

The odour of the over-marked female dung sample was more similar to that of non-oestrous female dung (Fig. 1). After over-marking, two alkanes were eliminated from the female's dung odour (3-methylpentane and dodecane; Table 1). Additionally, three alkanes appeared after over-marking, that were not present in the unmarked sample ((3-methylbutylidene)cyclopentane, 2-methylundecane, and 6-methyloctadecane; Table 1). The total proportion of alkanes emitted from the unmarked dung was 0.0347, and this increased to 0.0962 after overmarking (177% increase; Table 1). Further, the proportion of 2,6-dimethylundecane increased by 143% after overmarking; from 0.0027 to 0.0066 (Table 1). For a list of all tentatively identified VOCs and their relative proportions, see Supplementary Material Table S1.

Although over-marking increased the proportion of 2,6-dimethylundecane emitted, the proportion was above the average found in oestrous dung odour. Thus, over-marking did not conceal oestrous by mimicking non-oestrous dung odour as expected. Rather, it could be that urine over-marking in white rhinos makes the oestrous condition unrecognisable, or perhaps adds a signal to show that the female has already been mated.

Interestingly, the over-marked dung odour was still similar to female dung odour in general, and the over-marking behaviour did not create a new, unique odour. The most important indicator of territorial status in male dung odour is the alkane nonane, where territorial male dung emits a larger proportion than non-territorial (mean proportion $\pm$ SE non-territorial 0.0120 $\pm$ 0.0027, N = 29; territorial 0.0158 $\pm$ 0. 0031, N = 30; Marneweck et al., 2017a, unpublished data). The proportion of nonane in the dung odour of the oestrous female increased by only 10% after overmarking, and did not reach the level of nonane emitted from either non-territorial or territorial male dung. This suggests that the indicator for territorial status may be different in urine than in dung.

Due to the polygynous mating system of white rhinos, it is most likely that inter-sexual over-marking is for mate guarding, only this did not occur in the way we expected. White rhino males hold exclusive territories while females hold larger, unexclusive home ranges that encompass several male territories (Owen-Smith, 1975). Territorial males have primary access to females within their territory but sneaky copulations by subordinate males resident within the territories do occur (Guerier, 2012). As a result, by rendering a female's reproductive condition unrecognisable, a male would likely

Table 1. The tentatively identified VOCs belonging to the alkane function group, and their relative proportion, present in dung odour of unmarked and over-marked female dung.

			Proportion contribution to dung odour	
Name	CAS number	Weight (g/mol)	Sample one (unmarked)	Sample two (over-marked)
Nonane	111-84-2	128	0.0083	0.0092
Tridecane	629-50-5	184	0.0079	0.0383
Hexane	110-54-3	86	0.0073	0.0040
2,6,10-Trimethyldodecane	3891-98-3	212	0.0039	0.0148
2,6-Dimethylundecane	17301-23-4	184	0.0027	0.0066
Dodecane [-]	112-40-3	170	0.0012	0.0000
2,3-Dimethylundecane	17312-77-5	184	0.0008	0.0022
Tetradecane	629-59-4	198	0.0007	0.0025
3-Methylpentane [-]	96-14-0	86	0.0006	0.0000
3-Methyldecane	13151-34-3	156	0.0005	0.0016
Hexadecane	544-76-33	226	0.0005	0.0071
Heptadecane	629-78-7	240	0.0002	0.0027
Octadecane	593-45-3	254	0.0001	0.0010
(3-Methylbutylidene)cyclopentane [+]	53366-51-1	138	0.0000	0.0051
2-Methylundecane [+]	7045-71-8	170	0.0000	0.0006
6-Methyloctadecane [+]	10544-96-4	268	0.0000	0.0005
		Total	0.0347	0.0962

[-] denotes VOC eliminated after over marking

reduce the possibility of these subordinate males trying to mate with her. Yet, it could also be that the female defecated in a midden along the male's territory boundary. Neighbouring territorial males often explore the middens along their territorial boundaries (Owen-Smith, 1973). Thus, by over-marking her dung, the territorial male could have been trying to prevent the neighbouring male from detecting that the female was reproductively receptive. If so, this would then prevent aggressive interactions with the neighbouring male.

We fully acknowledge that the sample size is a key limitation of our study, but present these findings as a way to urge further investigation on the subject. Future studies could investigate the chemical implications of over-marking, as well as the function of urine marking in white rhinos. Yet, we suggest that this research be conducted on wild, free-roaming populations, and not captive white rhinos, as the chemical components/concentrations in the urine of captive individuals would likely differ to free-ranging individuals. This is because captive males do not display territorial behaviour, thus they will likely not have the same concentration of testosterone present in their urine (or volatile compounds that represent testosterone; Marneweck et al. (2017a)). Moreover, testosterone levels are significantly affected by social housing (i.e. number of females present; Kretzschmar et al. (2004), Christensen et al. (2009)). In addition, captive females do not show normal oestrous cycles, with cycles being erratic, shorter or longer than average, or females being acyclic (Brown et al., 2001). These hormonal fluctuations will also make it difficult to collect odours representative of free-roaming oestrous females. Finally, the volatile compounds emitted from dung and urine would likely be further impacted by the fact that captive animals do not eat a natural diet. As odours are influenced by diet, this would also change the volatile compounds emitted from dung and urine (Macdonald et al., 2008; Kean et al., 2011).

Despite the limited sample size, our results suggest that urine over-marking in white rhinos could be a function of mate guarding, and highlights the potential for urine to portray a different message than dung as a scent marking source. Ultimately, this is the first study to describe, and hypothesise on the function of, urine over-marking behaviour in white rhinos.

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Supplementary Material

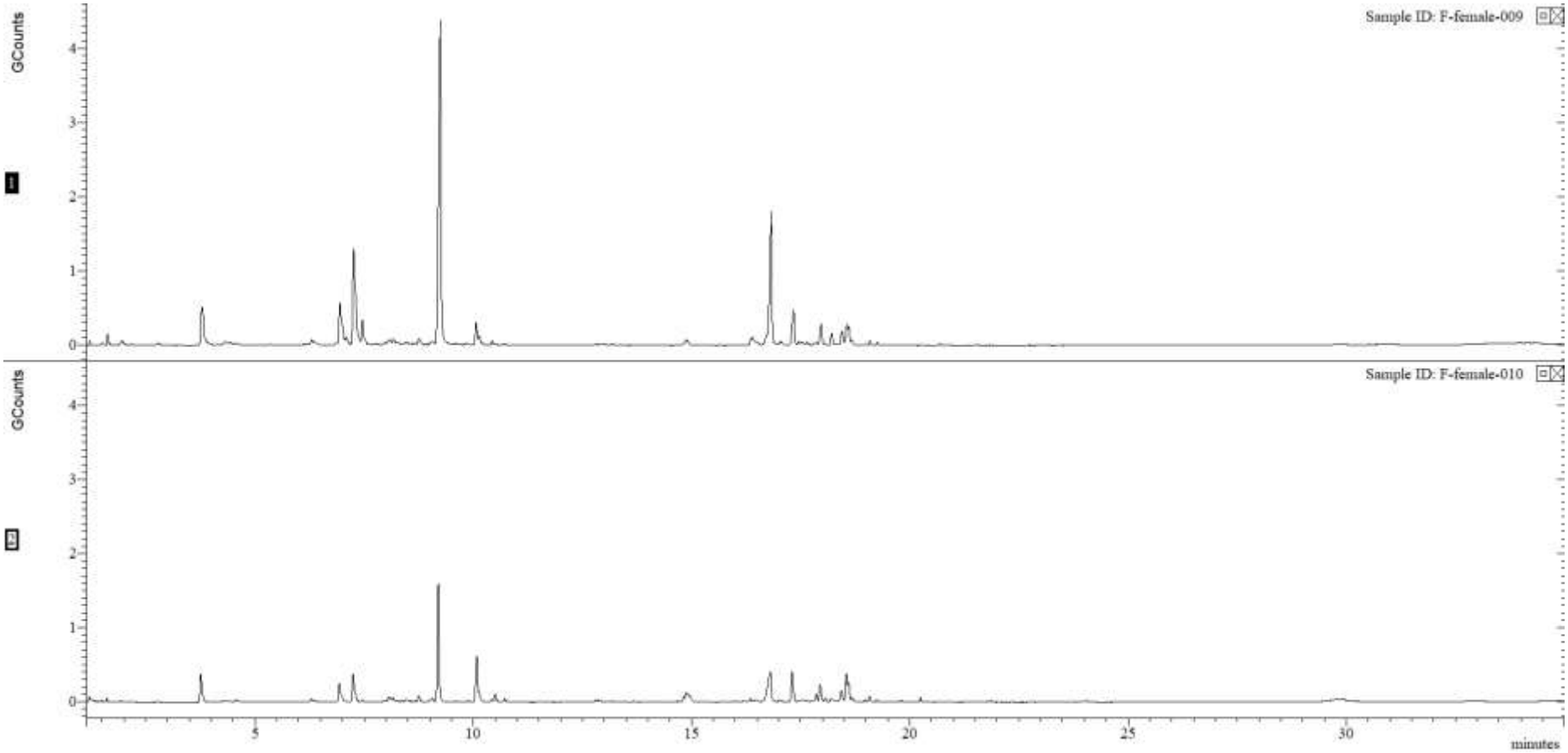

Figure S1. Chromatograms representing (1) the unmarked female dung odour (above), and (2) the over-marked female dung odour (below).

Table S1. The tentatively identified VOCs, and their relative proportion, present in dung odour of unmarked and over-marked female white rhino dung.

VOC name	Functional group	CAS number	Weight (g/mol)	Proportion contribution to dung odour	
				Sample one (unmarked)	Sample two (over-marked)
Limonene[c]	Monoterpene	138-86-3	136	0.3785	0.2329
(5*E*)-2,3,5,8-Tetramethyl-1,5,9-decatriene[a]	Alkadiene	230646-72-7	192	0.1276	0.0921
3,4-dihydro-β-ocimene[a]	Monoterpene	2436-90-0	138	0.1225	0.0711
Toluene[c]	Aromatic compound	108-88-3	92	0.0563	0.0667
2,7-Dimethyl-1,7-octadiene[a]	Monoterpene	40195-09-3	138	0.0545	0.0443
β-Caryophyllene[c]	Sesquiterpene	87-44-5	204	0.0379	0.0589
Camphene[b]	Monoterpene	79-92-5	136	0.0207	0.0049
α-Caryophyllene[a]	Sesquiterpene	6753-98-6	204	0.0204	0.0373
p-Cresol[c]	Aromatic compound	106-44-5	108	0.0181	0.0949
Cyclosativene[a]	Sesquiterpene	22469-52-9	204	0.0125	0.0043
Bicyclo[10.1.0]tridec-1-ene[a]	Alkene	54766-91-5	178	0.0113	0.0274
α-Muurolene[b]	Sesquiterpene	10208-80-7	204	0.0112	0.0085
Geranial[a]	Monoterpene	141-27-5	152	0.0094	0.0218
Nonane[c]	Alkane	111-84-2	128	0.0083	0.0092
Cymene[a-]	Monoterpene	99-87-6	134	0.0082	0.0000

Tridecane[c]	Alkane	629-50-5	184	0.0079	0.0383
Hexane[c]	Alkane	110-54-3	86	0.0073	0.0040
(2*E*)-1,4-dihydro-β-ocimene[b]	Alkadiene	2609-23-6	138	0.0073	0.0110
(*E*)-Oct-2-ene[b]	Alkene	13389-42-9	112	0.0070	0.0061
α-Pinene[c]	Monoterpene	80-56-8	136	0.0067	0.0013
(3*E*)-3-Ethyl-2,5-dimethyl-1,3-hexadiene[a]	Alkadiene	62338-07-2	138	0.0062	0.0141
(*Z*)-Oct-2-ene[b]	Alkene	7642/04/08	112	0.0058	0.0039
(*Z*)-1,4-dihydroocimene[a]	Monoterpene	2492-22-0	138	0.0054	0.0049
6-Methyl-5-heptene-2-one[b]	Irregular terpene	110-93-0	126	0.0045	0.0121
Pentanal[b]	Aliphatic aldehyde	110-62-3	86	0.0039	0.0024
α-Copaene[a]	Sesquiterpene	3856-25-5	204	0.0039	0.0011
Farnesane[a]	Sesquiterpene	3891-98-3	212	0.0039	0.0148
α-Panasinsen[a]	Sesquiterpene	56633-28-4	204	0.0034	0.0091
p-Mentha-1,4(8)-diene[a]	Monoterpene	586-62-9	136	0.0032	0.0038
β-Gurjunene[a]	Sesquiterpene	17334-55-3	204	0.0029	0.0010
6,11-Dimethyl-2,6,10-dodecatrien-1-ol[b]	Aliphatic alcohol		208	0.0028	0.0040
2,6-Dimethylundecane[b]	Alkane	17301-23-4	184	0.0027	0.0066
Tricyclene[a]	Monoterpene	508-32-7	136	0.0021	0.0005
Nonanal[c]	Aliphatic aldehyde	124-19-6	142	0.0018	0.0083
Butyric acid[b-]	Aliphatic acid	107-92-6	88	0.0018	0.0000

Dodecane[c-]	Alkane	112-40-3	170	0.0012	0.0000
Styrene[c-]	Aromatic compound	100-42-5	104	0.0011	0.0000
γ-Terpinen[a]	Monoterpene	99-85-4	136	0.0010	0.0008
δ-Cadinene[a]	Sesquiterpene	483-76-1	204	0.0008	0.0026
2,3-Dimethylundecane[a]	Alkane	17312-77-5	184	0.0008	0.0022
Tetradecane[b]	Alkane	629-59-4	198	0.0007	0.0025
(2*E*,6*E*)-4-Methyl-2,6-octadiene[a]	Alkadiene	74498-94-5	124	0.0006	0.0027
α-Calacorene[a]	Sesquiterpene	21391-99-1	200	0.0006	0.0015
3-Methylpentane[b-]	Alkane	96-14-0	86	0.0006	0.0000
4,8-Dimethyl-1,7-nonadiene[a]	Alkadiene	62108-28-5	152	0.0006	0.0012
Isobutyric acid[b-]	Aliphatic acid	79-31-2	88	0.0005	0.0000
Hexadecane[c]	Alkane	544-76-33	226	0.0005	0.0071
1,2-Dimethyl-1,3-cyclopentadiene[a]	Alkadiene	4784-86-5	94	0.0005	0.0010
3-Methyldecane[a]	Alkane	13151-34-3	156	0.0005	0.0016
2,3-Dimethyldodecane[a]	Sesquiterpene	6117-98-2	198	0.0004	0.0012
Quinoline[a]	Nitrogen compound	91-22-5	129	0.0004	0.0026
1-Methyl-4-(1-hydroxy-1-methylethyl)benzene[a-]	Aromatic compound	1197-01-9	150	0.0002	0.0000
3-Propylphenol[a]	Aromatic compound	621-27-2	136	0.0002	0.0013
Heptadecane[c]	Alkane	629-78-7	240	0.0002	0.0027
Undecan-2-one[b]	Aliphatic ketone	112-12-9	170	0.0002	0.0010

(2*E*)-3,7,11,15-Tetramethyl-2-hexadecene[a]	Alkene	14237-73-1	280	0.0001	0.0030
6,10,14-Trimethyl-2-pentadecanone[a]	Aliphatic ketone	502-69-2	268	0.0001	0.0014
Octadecane[c]	Alkane	593-45-3	254	0.0001	0.0010
Pentan-1-ol[b+]	Aliphatic alcohol	71-41-0	88	0.0000	0.0008
2-Ethylhexan-1-ol[b+]	Aliphatic alcohol	104-76-7	130	0.0000	0.0078
3-Methyl-1H-indole[c+]	Nitrogen compound	83-34-1	131	0.0000	0.0035
(3-Methylbutylidene)cyclopentane[b+]	Alkane	53366-51-1	138	0.0000	0.0051
Undec-1-ene[a+]	Alkene	821-95-4	154	0.0000	0.0153
Decanal[c+]	Aliphatic aldehyde	112-31-2	156	0.0000	0.0016
2-Methylundecane[b+]	Alkane	7045-71-8	170	0.0000	0.0006
Geranylacetone[a+]	Monoterpene	3796-70-1	194	0.0000	0.0026
α-Longipinene[b+]	Sesquiterpene	5989-08-2	204	0.0000	0.0011
Germacrene D[a+]	Sesquiterpene	23986-74-5	204	0.0000	0.0020
6-Methyloctadecane[a+]	Alkane	10544-96-4	268	0.0000	0.0005

Compound identification criteria and notes:

[a] denotes comparison of MS with published data

[b] denotes comparison of MS and retention time with published data

[c] denotes comparison of MS and retention time with authentic standard

[-] denotes VOC eliminated after over-marking

[+] denotes VOC appeared after over-marking