



Data Article

Chemical composition of steam and solvent crude oil extracts from *Azadirachta indica* leaves

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ABSTRACT

This work identifies the chemical components of *Azadirachta indica* (neem plant) leaf extracts. *A. indica* is a vascular plant which belongs to the Meliaceae family and its use as herb in folk medicine is widely acclaimed. Essential oils were extracted from leaves of *A. indica*. Steam and solvent extraction methods were used with two solvents: Ethanol and Hexane. The crude oil extracted using both extraction methods were analyzed using GC-MS. The result of the analyses show that the major constituents were Eicosane (9.7662%), Diacenaphtho[1,2-j:1',2'-l]fluoranthene (11.301%), Phenol, 4-[[[4-methoxyphenyl)methylene]amino]- (11.84%) and (3Ar,6S,9ar)-1,2,3,4,5,6,7,9a-octahydro-8-methyl-3a,6-methano-3ah-cyclopentacycloocten-10-one (36.883%) in steam extracted oil; Eicosane (10.259%), Diacenaphtho[1,2-j:1',2'-l]fluoranthene (13.51%) and Butanamide, N-(2-methoxyphenyl)-3-oxo- (16.615%) in the ethanol extracted oil, and (3Ar,6S,9ar)-1,2,3,4,5,6,7,9a-octahydro-8-methyl-3a,6-methano-3ah-cyclopentacycloocten-10-one (10.72%), n-Hexadecanoic acid (14.688%) and 9,12,15-Octadecatrienoic acid, (Z,Z,Z)- (34.719%) in the hexane extracted *A. indica* essential oil.

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Specifications Table

Subject area	Chemical Engineering, Biochemistry
Compounds	Active Ingredient
Data category	data of bioactive compounds in crude essential oil from neem and lemon plants
Data acquisition format	Mass spectra
Data type	Raw, analyzed
Procedure	Oil samples were analyzed using a gas chromatography-mass spectrometry analyzer, Agilent technologies 7890B GC system, operating at ionization energy of 70 eV with a HP-5MS capillary column (30 m × 0.25 mm; film thickness 0.50 μm). 1 μl of essential oil was injected in split/split less mode at a split ratio of 20:1 and an inlet temperature of 250 °C. The carrier gas used to aid the analysis of the essential oils was helium at a constant pressure mode of 9.4 psi. The oven was programmed to have a 3 °C/min incremental raise until it reached 240 °C from 60 °C. The mass spectrometry analyser was operated at a scan mode in 40–400 m/z range with an ion source and transfer line temperature at 230 and 300 °C respectively. ChemStation software was used in the analysis of the data acquired from the GC-MS. The constituents of the essential oils was determined based on their Kovats indices (KI), retention time (RT) and mass spectra with NIST.

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Data accessibility

A detailed data of compounds identified by GC-MS analysis of crude essential oil extracts from neem, lemon grass and commercial essential oil is provided in this article

1. Rationale

It has long been observed that plants' roots, flowers, seeds and leaves contain some active ingredients. Some are being extracted with organic solvent such as ether while some are odoriferous and very volatile [1–3]. *Azadirachta indica* is a vascular perennial plant belonging to the family Meliaceae and has *A. excelsa* as its only congener. It is a fast growing evergreen tree. Its leaves are pinnate and consist of 20–30 serrated leaflets which are dark green in colour when they are matured. *A. indica* adapts to tropical and subtropical climates and grows best where annual rainfall is between 450–1200 mm. Although *A. indica* can grow within soil pH of 5.0–8.0 and is known to survive in variety of soil types, it is best adapted to deep, permeable sandy soil and soil pH between 6.2–7.0 [4]. *A. indica* extracts are used in cosmetics, insecticides and repellents. The plant is also known to have diverse medicinal values. Constituents of extracts from the leaves and other parts of the plant have been reported to exhibit antidiabetic, immunomodulatory, diuretic, antiseptic, anti-inflammatory, antipyretic, antihyperglycaemic, antiulcer, antiarthritic, antimalarial, spermicidal, antifungal, antibacterial, hypoglycemic, antioxidant, antimutagenic and anticarcinogenic properties [5–8]. *A. indica* leave extracts and some other leaves are active against certain dermatophytes, microorganisms and parasitic insects such as bacteria, fungi and mosquitoes [9–11]. The wide range of bioactive constituents and the versatility of the use of *A. indica* plant extracts have positioned the plant as one of the most important herbal and medicinal plants [12].

As there are renewed interests in herbal based medications in order to forestall the side effects of synthetic drugs, the quest to find new and unique molecular structures of plant origin as major constituents of some natural products, and those of *modern drugs* as means of combating recalcitrant diseases is also on the increase [13–15]. Inasmuch as essential oils and other plant extracts are principally products of complex biological processes; and genetics and environment play crucial role in determining the morphology, chemical composition and ultimately the properties of herbal and medicinal plants [16,17], expanded knowledge rooted in holistic scientific research will open up a wealth of possibilities. As demand for plant based medication increases, studies on taxonomy, toxicity, chemical analysis and pharmacology of plant secondary metabolites will among other things, prevent problems associated with indiscriminate use caused by incorrect identification, improper documentation and lack of standardization of plant based extracts and their products. This work presents the chemical components of essential oils from *A. indica* leaves of Nigerian origin, extracted using steam, ethanol and hexane for the purpose of further scientific investigations.

2. Procedure

2.1. Source of raw materials

Several *A. indica* trees are found within the premises of Covenant University, Ota, Ogun State, Nigeria. Ota is on latitude 6.41 °N and longitude 3. 41 °E, has a tropical climate and a mean annual rainfall of 1280 mm. Fresh leaves were plucked from one of the *A. indica* trees found on sandy - loam soil within the academic area of the University, air dried and partly ground to powder.

2.2. Extraction of the essential oil by steam distillation

The air dried leaves was placed in a conical flask with distilled water at the bottom of the flask and an electrically powered hot plate with a control dial to set the temperature as desired served as the heat source for generating the required steam for the extraction process. A T-connector, having a thermometer at one end and a condenser at the other was attached to the conical flask with the aid of a cork. The condenser had an L-connector at its other end by which it was attached to a flat bottom flask with a cork. The mixture of extracted essential oil and water flowed from the conical flask to the flat bottom flask.

2.3. Solvent extraction of the essential oil

Using the Soxhlet apparatus, essential oil was extracted from the plant material with hexane and ethanol. The ground plant material was placed in a muslin cloth material and put in the extraction chamber of the Soxhlet apparatus. Hexane or ethanol was placed in the flat bottom flask; 300 ml of each of the solvent was used to cause a siphoned flow in the apparatus without totally evaporating from the flat bottom flask. As the solvent is evaporated from the flat bottom flask at a temperature between 50–70 °C for hexane and 78.24 °C for ethanol, it goes through the distillation path through which it gets to the middle section of the Soxhlet apparatus before the vapour flows to the condenser at the top of the apparatus. The vapour condenses and falls back into the middle section of the Soxhlet apparatus where it comes in contact with the plant material placed there. Based on the design of the apparatus, the solvent has enough retention time in the middle

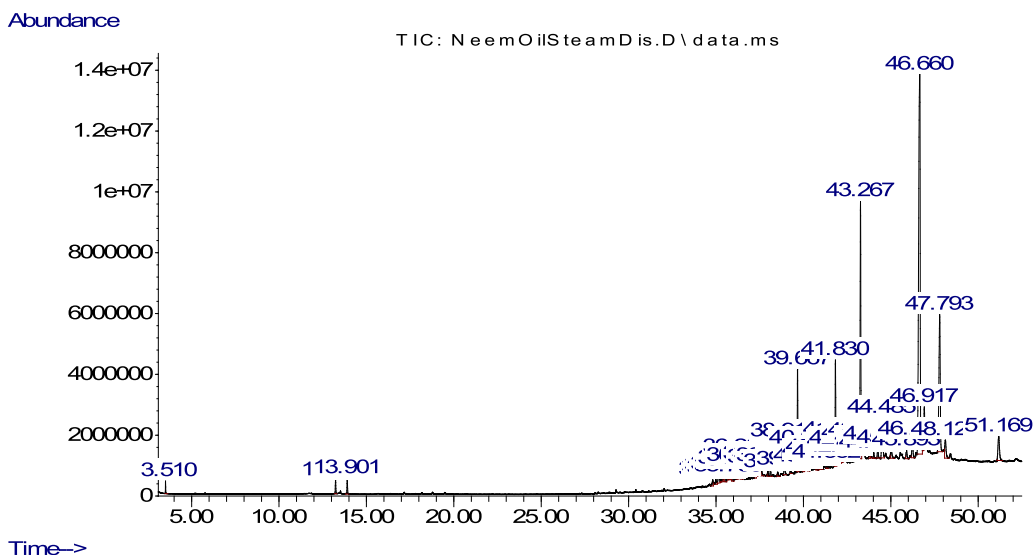


Fig. 1. Gas chromatogram of steam extracted essential oil from *A. indica* leaves.

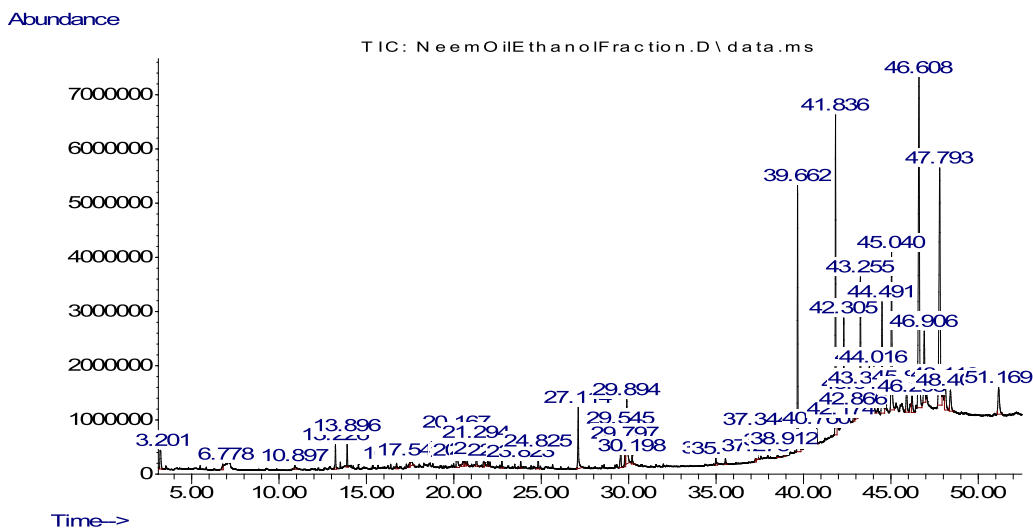


Fig. 2. Gas chromatogram of ethanol extracted essential oil from *A. indica* leaves.

section to enable it to extract the desired substances from the plant material there before it is siphoned back into the flat bottom flask.

2.4. Oil extract analysis

The composition of the oil extracts was determined with the use of a gas chromatography-mass spectrometry analyser, Agilent technologies 7890B GC system, operating at ionization energy of 70 eV with a HP-5MS capillary column (30 m × 0.25 mm; film thickness 0.50 μm). 1 μl of essential oil was injected in split/splitless mode at a split ratio of 20:1 and an inlet temperature of 250 °C. The carrier gas used to aid the analysis of the essential oils was helium at a constant pressure mode of 9.4 psi. The oven was programmed to have a 3 °C/min incremental raise until it reached 240 °C from 60 °C. The mass spectrometry analyser was operated at a scan mode in 40–400 m/z range with an ion source and transfer line temperature at 230 and 300 °C respectively. ChemStation software was used in the analysis of the data acquired from the GC-MS. The constituents of the essential oils was determined based on their Kovats indices (KI), retention time (RT) and mass spectra with NIST.

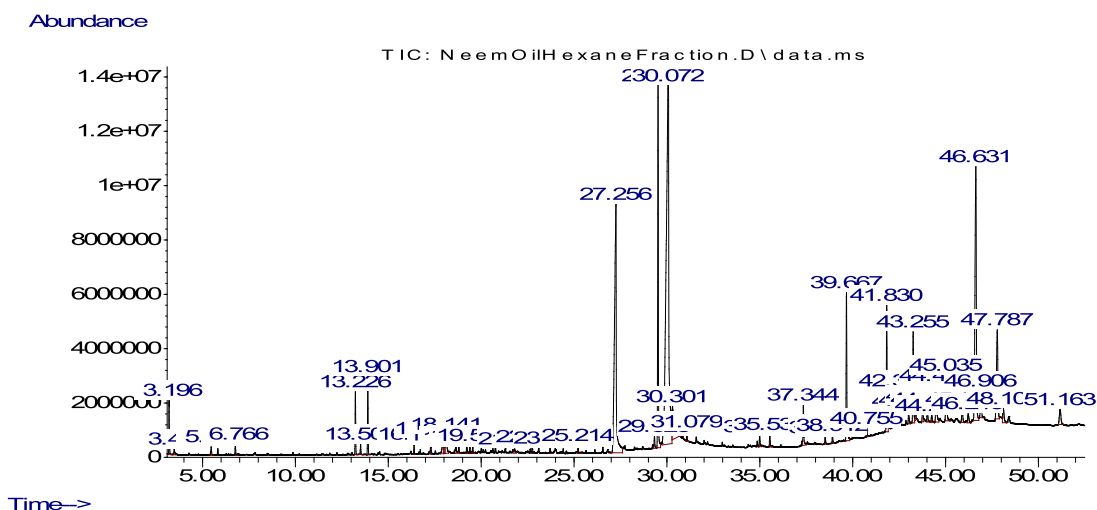


Fig. 3. Gas chromatogram of hexane extracted essential oil from *A. indica* leaves.

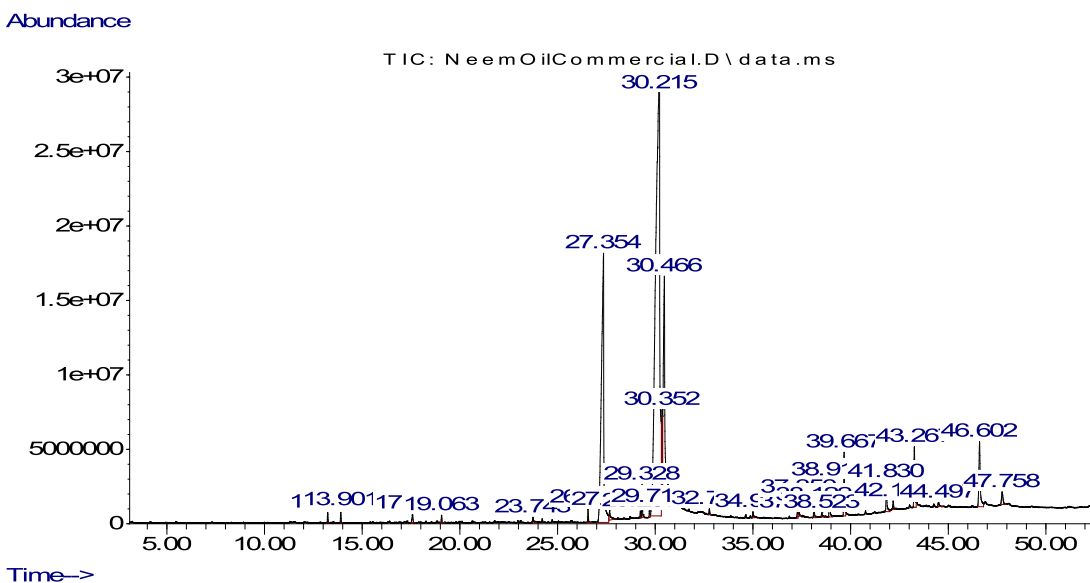


Fig. 4. Gas chromatogram of commercial neem oil.

3. Data, value and validation

The chemical components of neem essential oil from commercial source and those extracted from *A. indica* leaves with steam, ethanol and hexane are presented in Table 1 and the gas chromatograms are shown in Figs. 1–4.

Respectively, a total of 39, 44, 44 and 25 compounds were identified in essential oil extracted using steam, ethanol, hexane and the commercial neem oil. 2,6-Octadienal, 3,7-dimethyl-, (Z)-; Citral; Eicosane and 1,2-Bis(trimethylsilyl)benzene were found to be common chemical components of essential oils from steam extraction, ethanol extraction, hexane extraction and the commercial neem oil. Eicosane is known to possess antifungal, antibacterial, antitumor and cytotoxic properties [18]. Anticancer, antimicrobial, antioxidant and hypercholesterolemic properties of 9,12,15-Octadecatrienoic acid, (Z,Z,Z)- have been reported [19]. Several fatty acids like oleic acid and hexadecanoic acid exhibit antibacterial and antifungal activities [20]. Octadecanoic acid is also known to possess antitumor activity in addition to antibacterial and antifungal activities [18,21]. The roles of minor components in increasing the activity of essential oils and providing synergistic effects have been shown [22]; hence, probable contributions of minor components to the overall properties of essential oils should not be underestimated. As an antioxidant, Vitamin E has the capability to protect organisms from oxidative stress [23].

Table 1Constituents of crude oils extracts from *Azadirachta indica* leaves and a commercial source as identified by GC-MS analysis.

No	RT	Compound	Area% for extraction methods considered			Area% for commercial essential oil
			Steam	Ethanol	Hexane	
1	3.2014	Toluene	ND	1.0011	0.8454	ND
2	3.476	Cyclopentanol, 1-methyl-	ND	ND	0.369	ND
3	3.5103	Silanediol, dimethyl-	0.2081	ND	ND	ND
4	5.4672	3-Pentanol, 2-methyl-	ND	ND	0.2057	ND
5	6.7776	1,2,3,4-Butanetetrol, [S-(R*,R*)]-	ND	0.251	ND	ND
6	10.898	4H-Pyran-4-one, 2,3-dihydro-3,5-dihydroxy-6-methyl-	ND	0.2448	ND	ND
7	13.226	2,6-Octadienal, 3,7-dimethyl-, (Z)-	0.4485	0.744	1.1123	0.1863
8	13.507	Geraniol	ND	ND	0.1885	ND
9	13.896	Citral	0.5734	0.7511	1.4285	0.2286
10	16.385	2,6-Octadien-1-ol, 3,7-dimethyl-, (Z)-	ND	ND	0.1808	ND
11	16.722	Undecane	ND	0.1974	ND	ND
12	17.289	Caryophyllene	ND	ND	0.3518	ND
13	17.483	Cyclopentaneundecanoic acid	ND	0.224	ND	ND
14	17.546	N-acetyl-4-fluoroamphetamine	ND	0.4074	ND	ND
15	17.575	1,3,6,10-Dodecatetraene, 3,7,11-trimethyl-, (Z,E)-	ND	ND	ND	0.2578
16	17.947	Diethylmalonic acid, di(3-chlorobenzyl) ester	ND	ND	0.659	ND
17	18.021	1-Decanol, 2-hexyl-	ND	ND	0.2578	ND
18	18.141	4,5-Dimethyl-3-heptanol	ND	ND	0.7893	ND
19	18.645	Octanal, 7-hydroxy-3,7-dimethyl-	ND	ND	0.2364	ND
20	18.799	Pentadecane	ND	ND	0.1359	ND
21	19.063	.beta.-Bisabolene	ND	ND	ND	0.1402
22	19.223	Naphthalene, 1,2,4a,5,6,8a-hexahydro-4,7-dimethyl-1-(1-methylethyl)-	ND	ND	0.2427	ND
23	19.549	2(4H)-Benzofuranone, 5,6,7,7a-tetrahydro-4,4,7a-trimethyl-	ND	ND	0.1652	ND
24	20.167	1-Piperidinethiocarboxamide	ND	1.3956	ND	ND
25	20.528	Imidazole-2-carboxylic acid, 4-methyl-	ND	0.4337	ND	ND
26	20.631	Hexadecane, 1-(ethenyl)-	ND	0.2265	ND	ND
27	20.768	Hexadecane	ND	0.1809	ND	ND
28	21.294	Azulene, 1,2,3,3a,4,5,6,7-octahydro-1,4-dimethyl-7-(1-methylethenyl)-, [1R-(1.alpha.,3a.beta.,4.alpha.,7.beta.)]-	0.4568	0.6594	ND	ND
29	21.706	.alpha.-Cadinol	ND	0.3012	ND	ND
30	21.798	l-[-]-4-Hydroxy-1-methylproline	ND	ND	0.1621	ND
31	21.952	1,4-Methano-1H-indene, octahydro-1,7a-dimethyl-4-(1-methylethenyl)-, [1S-(1.alpha.,3a.beta.,4.alpha.,7a.beta.)]-	ND	0.224	ND	ND
32	22.748	1-Naphthalenol, decahydro-1,4a-dimethyl-7-(1-methylethylidene)-, [1R-(1.alpha.,4a.beta.,8a.alpha.)]-	ND	0.2223	ND	ND
33	22.753	Pentadecane, 2,6,10,14-tetramethyl-	ND	ND	0.1602	ND
34	23.709	Tetradecanoic acid	ND	ND	0.1533	0.1998
35	23.823	N-Benzhydrylimidazole	ND	0.2007	ND	ND
36	24.825	2-Naphthalenemethanol, 1,2,3,4,4a,5,6,8a-octahydro-.alpha.,.alpha.,4a,8-tetramethyl-, (2.alpha.,4a.alpha.,8a.alpha.)-	ND	0.514	ND	ND
37	25.214	2-Pentadecanone, 6,10,14-trimethyl-	ND	ND	0.1843	ND
38	26.558	Hexadecanoic acid, methyl ester	ND	ND	ND	0.2843
39	27.114	n-Hexadecanoic acid	ND	2.117	14.688	19.463
40	27.668	Ethyl 13-methyl-tetradecanoate	ND	ND	ND	0.4315
41	29.236	9,12-Octadecadienoic acid (Z,Z)-, methyl ester	ND	ND	ND	0.1296
42	29.328	11-Octadecenoic acid, methyl ester	ND	ND	0.322	ND
43	29.328	6-Octadecenoic acid, methyl ester, (Z)-	ND	ND	ND	0.6452
44	29.522	Phytol	ND	ND	8.0087	ND
45	29.545	3H-Pyrazolo[4,3-c]quinolin-3-one, 8-fluoro-1,2-dihydro-	ND	1.1577	ND	ND
46	29.711	Methyl stearate	ND	ND	ND	0.2046
47	29.797	9,12-Octadecadienoic acid (Z,Z)-	ND	0.8601	ND	ND
48	29.894	9,12,15-Octadecatrienoic acid, (Z,Z,Z)-	ND	2.8747	34.719	ND
49	30.198	Octadecanoic acid	ND	0.2942	ND	10.187
50	30.215	Oleic Acid	ND	ND	ND	55.723
51	30.352	9-Octadecenoic acid, (E)-	ND	ND	ND	2.2079
52	31.079	Tridecanedial	ND	ND	0.1968	ND
53	34.809	Thujone	0.4116	ND	ND	ND
54	34.987	2-Dodecen-1-yl(-)succinic anhydride	0.396	ND	ND	ND
55	34.993	15-Hydroxypentadecanoic acid	ND	0.3036	0.2885	ND
56	34.998	cis-9-Hexadecenal	ND	ND	ND	0.1752

(continued on next page)

Table 1 (continued)

No	RT	Compound	Area% for extraction methods considered			Area% for commercial essential oil
			Steam	Ethanol	Hexane	
57	35.147	Longifolene	0.7151	ND	ND	ND
58	35.29	2,6,10,14,18-Pentamethyl-2,6,10,14,18-eicosapentaene	0.1915	ND	ND	ND
59	35.439	1,3,3-Trimethyl-2-hydroxymethyl-3,3-dimethyl-4-(3-methylbut-2-enyl)-cyclohexene	1.0775	ND	ND	ND
60	35.536	Phthalic acid, di(2-propylpentyl) ester	ND	ND	0.2928	ND
61	35.542	3-OXO-18-NOR-ENT-ROS-4-ENE-15.beta.,16-ACETONIDE	ND	0.2014	ND	ND
62	35.708	1,6,10,14-Hexadecatetraen-3-ol, 3,7,11,15-tetramethyl-, (E,E)-	0.2251	ND	ND	ND
63	35.862	4-(2,2,6-Trimethyl-bicyclo[4.1.0]hept-1-yl)-butan-2-one	0.9811	ND	ND	ND
64	36.206	Tricyclo[4.3.1.1(3,8)]undecane	0.8226	ND	ND	ND
65	37.276	7-Pentadecyne	0.5684	ND	ND	ND
66	37.276	Heptasiloxane, 1,1,3,3,5,5,7,7,9,9,11,11,13,13-tetradecamethyl-	ND	0.2393	1.4927	ND
67	37.281	6-Octadecenoic acid, (Z)-	ND	ND	ND	0.1436
68	37.344	Nonadecane	ND	ND	1.0061	1.4981
69	37.344	Eicosane	9.7662	10.259	0.1472	1.3947
70	37.608	Phenol, 4-[2,3-dihydro-7-methoxy-3-methyl-5-(1-propenyl)-2-benzofuranyl]-2-methoxy-	0.4073	ND	ND	ND
71	37.968	2,6,10,14-Hexadecatetraen-1-ol, 3,7,11,15-tetramethyl-, acetate, (E,E,E)-	0.4922	ND	ND	ND
72	38.117	Silanol, trimethyl-, phosphite (3:1)	0.4224	ND	ND	ND
73	38.123	2-Pyridinamine, 5-methyl-	ND	ND	ND	0.3278
74	38.523	Octadecane, 3-ethyl-5-(2-ethylbutyl)-	ND	0.2227	ND	ND
75	38.912	Squalene	1.4021	ND	ND	0.8635
76	38.912	Trichothec-9-en-4-ol, 7,8:12,13-diepoxy-, 2-butenate, [4.beta.(Z),7.beta.,8.beta.]-	0.1926	0.2308	0.1759	ND
77	39.662	Heptadecane	ND	7.5868	ND	ND
78	39.667	Tetracosane	ND	ND	3.5068	ND
79	40.011	trans-Geranylgeraniol	1.4076	ND	ND	ND
80	40.263	Indolizine, 2-(4-methylphenyl)-	0.2395	ND	ND	ND
81	40.755	Octadecane, 1-iodo-	ND	0.4477	0.188	ND
82	40.76	Heneicosane, 3-methyl-	0.3953	ND	ND	ND
83	40.966	4-Dehydroxy-N-(4,5-methylenedioxy-2-nitrobenzylidene)tyramine	0.6149	ND	ND	ND
84	41.333	2-(Acetoxymethyl)-3-(methoxycarbonyl)biphenylene	0.2009	ND	ND	ND
85	41.83	Octadecane	ND	1.6801	3.0339	ND
86	41.956	1,2,4-Oxadiazole, 3-(1,3-benzodioxol-5-yl)-5-[(4-iodo-1H-pyrazol-1-yl)methyl]-	1.1733	ND	ND	ND
87	42.122	1,2-Bis(trimethylsilyl)benzene	2.3602	4.4112	1.417	0.1613
88	42.174	1,4-Bis(trimethylsilyl)benzene	1.2851	2.1832	ND	ND
89	42.179	5-Methyl-2-phenylindolizine	0.4894	ND	ND	0.1866
90	42.305	Vitamin E	0.6673	3.3997	0.9438	ND
91	42.866	Phenylacetic acid, 2-(1-adamantyl)ethyl ester	ND	0.3971	ND	ND
92	43.015	Silane, trimethyl[5-methyl-2-(1-methylethyl)phenoxy]-	ND	1.7215	0.3501	0.5514
93	43.255	Benzenamine, N-[(4-methoxyphenyl)methylene]-, N-oxido	ND	ND	2.3849	ND
94	43.255	4-[(3-Methoxy-phenylimino)-methyl]-phenol	ND	4.7733	ND	ND
95	43.267	Phenol, 4-[[4-methoxyphenyl)methylene]amino]-	11.84	ND	ND	ND
96	43.267	1-Isopropoxy-2-phenylmethylbenzene	ND	ND	ND	1.7079
97	43.387	Benzo[h]quinoline, 2,4-dimethyl-	4.4605	ND	0.2026	ND
98	43.764	2,4-Cyclohexadien-1-one, 3,5-bis(1,1-dimethylethyl)-4-hydroxy-	0.5062	ND	ND	ND
99	43.764	1H-Indole-2-carboxylic acid, 6-(4-ethoxyphenyl)-3-methyl-4-oxo-4,5,6,7-tetrahydro-, isopropyl ester	ND	1.7602	ND	ND
100	43.862	4-(1,1-Dimethylpropyl)phenol, trimethylsilyl ether	0.2728	ND	ND	ND
101	44.016	Tris(tert-butyltrimethylsilyloxy)arsane	0.26	ND	ND	ND
102	44.022	Octasiloxane, 1,1,3,3,5,5,7,7,9,9,11,11,13,13,15,15-hexadecamethyl-	ND	ND	0.3901	ND
103	44.251	Silicic acid, diethyl bis(trimethylsilyl) ester	0.6732	ND	ND	ND
104	44.485	.beta.-Sitosterol	2.4735	ND	ND	ND
105	44.491	.gamma.-Sitosterol	ND	4.675	1.2889	ND
106	45.035	Arsenous acid, tris(trimethylsilyl) ester	ND	ND	1.5873	ND
107	45.04	2-[4-Cyclohexylbutanoylamino]-3-chloro-1,4-naphthoquinone	ND	6.5549	ND	ND
108	46.602	5-Oxo-.delta.4-decahydrobenzindene	ND	ND	ND	2.702
109	46.608	Butanamide, N-(2-methoxyphenyl)-3-oxo-	ND	16.615	ND	ND
110	46.631	(3Ar,6S,9ar)-1,2,3,4,5,6,7,9a-octahydro-8-methyl-3a,6-methano-3ah-cyclopentacycloocten-10-one	36.883	ND	10.72	ND

(continued on next page)

Table 1 (continued)

No	RT	Compound	Area% for extraction methods considered			Area% for commercial essential oil
			Steam	Ethanol	Hexane	
111	46.906	Tetrasiloxane, decamethyl-	ND	ND	0.9463	ND
112	46.906	3-Quinolincarboxylic acid, 6,8-difluoro-4-hydroxy-, ethyl ester	ND	3.3455	ND	ND
113	46.917	2-Ethylacridine	2.7375	ND	ND	ND
114	47.787	Anthra[2,3-a]coronene	ND	ND	3.8743	ND
115	47.793	Diacenaphtho[1,2-j:1',2'-l]fluoranthene	11.301	13.51	ND	ND
TOTAL			100	100	100	100

ND-not detected.

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Supplementary material

Supplementary material associated with this article can be found, in the online version, at doi:[10.1016/j.cdc.2019.100208](https://doi.org/10.1016/j.cdc.2019.100208).

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