Aloe Exudate: Characterization by Reversed Phase HPLC and Headspace GC-MS

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From the leaves of aloe, a succulent plant, a dried exudate commonly called aloe can be obtained, which is used as a natural drug for its cathartic effect and is widely employed as a bittering agent in alcoholic beverages. This investigation provides a tentative characterization of several commercial aloe exudates carried out both by reversed phase HPLC and by headspace GC-MS analysis. By means of HPLC the derivatives were evaluated, and by GC-MS the volatile fraction was investigated. Qualitative and quantitative differences among the constituents in various samples of different origins were found. In particular, these were evident in the HPLC profile of Kenya aloe and an *Aloe barbadensis* sample, which exuded a high content of isoaloeresin D and aloins, whereas GC-MS analysis showed the presence of anisole exclusively in Kenya aloe samples. Moreover, the results obtained by means of the latter technique suggested a reason for the prevailing use of Mosselbay and Port Elizabeth aloes in bitter spirits formulation.

Keywords: *Aloe; aloin; aroma; volatile compounds; HPLC-DAD; HS-GC-MS*

INTRODUCTION

The genus *Aloe* belongs to the Asphodelaceae family, Alooideae sub family, and comprises ∼420 species of succulent plants (*1*, *2*). They are indigenous to southern and eastern Africa and Madagascar but have been introduced into the West Indies, where aloes are extensively cultivated, and into tropical countries and even flourish in the Mediterranean area. Aloe has been used as a folk medicine for 3000 years; it is also now used in modern medicine in many parts of the world and is called "a pharmacy in a plant" (*3*, *4*).

From the leaves of *Aloe* plants three types of commercial products can be obtained: the dried exudate, excreted from the aloin cells present in the zone of the vascular bundle; the gel, a mucilaginous juice present in the center of the leaf (hydrenchyma); and the oil, extracted by organic solvents (*5*, *6*). The first, commonly called aloe, is a natural drug well-known for its cathartic effect and also used as a bittering agent in alcoholic beverages. The gel is utilized as a skin-care product and as a smoothening and softening agent in the cosmetic and pharmaceutical industries and as a dietary supplement in several beverages. The oil is the fatty fraction of the leaf and is used only in the cosmetic industry as a carrier of pigment and soothing agent (*6*).

In this investigation we consider only the commercial exudates, characterized by the presence of many interesting secondary metabolites belonging to different classes of compounds including alkaloids, anthraquinones, and precursors, anthrones, bianthraquinonoids, chromones, coumarins, pyrones, and their *O*- or *C*-glycosyl derivatives (*7*). The three most important constituents of aloe drug are the anthrones aloin A and B (*8*) and the chromones aloesin (*9*) and aloeresin A (*10*, *11*).

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The majority of the studies on aloe characterization have been carried out by means of TLC and HPLC (*12*−*22*), whereas there are only a few studies on the analysis of aroma chemicals in an aloe leaf extract by GC (*23*, *24*).

The aim of the present study was to characterize several samples of commercial aloe exudate both by HPLC and by GC-MS methods. The use of the latter permitted us to investigate the aroma, which was compared with HPLC results to search for any correlation that might exist. In fact, such characterization is an important parameter in choosing exudate for the alcoholic beverage industry.

MATERIALS AND METHODS

Commercial Products. This study was conducted on commercial aloe extracts that were obtained from chemist and herbalist shops and from the herbal and aroma industry. In particular, we had at our disposal three samples of *Aloe barbadensis* (purchased in chemist and herbalist shops), three samples of Cape aloe, two samples of aloe from Kenya, three from Mosselbay, and three from Port Elizabeth, gifts kindly supplied by Sessa Carlo S.p.A (Milan, Italy), Janousek Industriale S.r.l (Trieste, Italy), and Fratelli Bauer S.p.A (Trieste, Italy).

Reversed Phase HPLC Analysis. The equipment consisted of a Spectra System P2000 pump with an SCM 100 vacuum membrane degasser and a Spectra System UV6000LP detector set at 220 nm (Thermoquest, San Jose, CA). Samples were injected with a model 7725i sample valve equipped with a 20 μL loop (Rheodyne, Rohnert Park, CA); the volume injected was 10 μL. Peak areas were calculated by means of Chromquest software (Thermoquest).

Separations were carried out with a Machery-Nagel ET 250/4 Nucleosil 100 C_{18} reversed phase column (Machery-Nagel GmbH & Co. Düren, Germany) equipped with a Waters Insert Nova-Pak C_{18} precolumn; particle size of the packing was 4 μm (Waters, Milford, MA).

The mobile phase was a linear gradient of water/acetonitrile: isocratic for 12 min with 16% acetonitrile, followed by a

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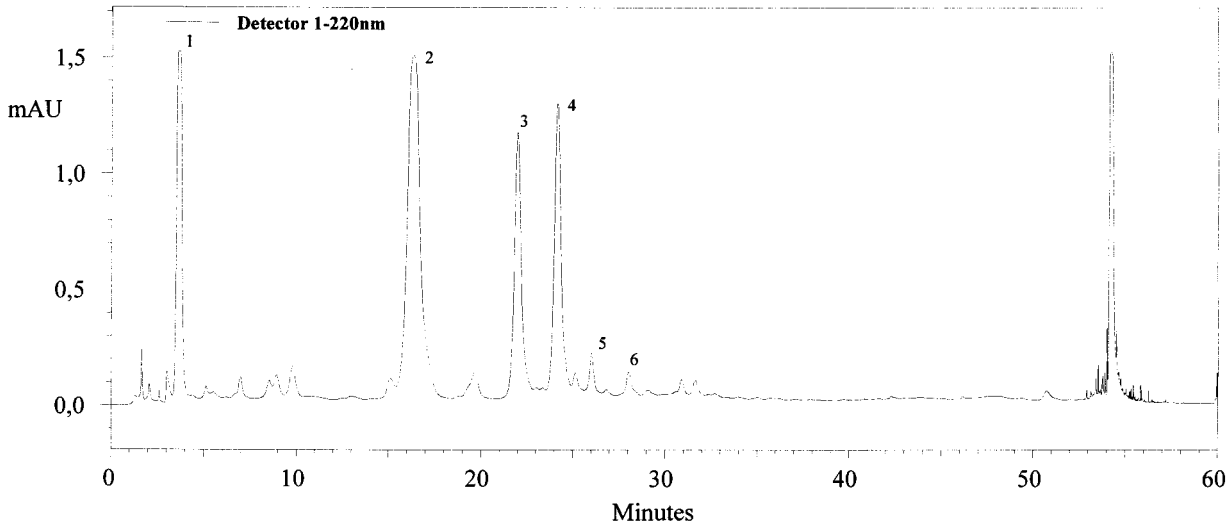
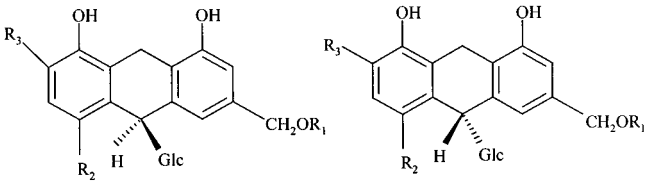
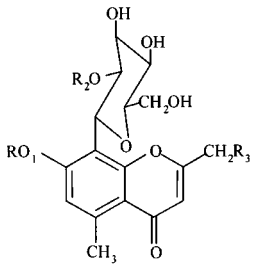


Figure 1. Reversed phase HPLC chromatogram of a Mosselbay aloe sample. Conditions were as described under Materials and Methods. Peaks: 1, aloesin; 2, aloeresin A; 3, aloin B; 4, aloin A; 5, aloinoside B; 6, aloinoside A.



	R_1	R_2	R_3	Config
Aloin A	H	H	H	S, S
Aloin B	H	H	H	R, S
Aloinoside A	α–L-rhamnosyl	H	H	S, S
Aloinoside B	α–L-rhamnosyl	H	H	R, S



	R_1	R_2	R_3
Aloesin	H	H	$COCH_3$
Aloeresin A	H	*p*-coumaroyl	$COCH_3$

Figure 2. Chemical structures of the main compounds identified in aloe exudates (see chromatogram shown in Figure 1).

linear gradient up to 33% acetonitrile in 25 min; a second linear gradient from 33 to 60% acetonitrile in 13 min; and then continued with a third linear gradient up to 100% of acetonitrile followed by an isocratic step (100% acetonitrile for 9 min) and re-equilibration step (to 16% acetonitrile) for 15 min before the next injection. The column was thermostated at 45 °C with an LC 100 column oven (Perkin-Elmer, Norwalk, CT); the flow rate was 1 mL/min. The identification and the purity of the chromatographic peaks were estimated using a UV6000LP photodiode array detector (DAD) (Thermoquest).

All solvents were of HPLC grade (J. T. Baker, Mallinckrodt Baker B.V., Deventer, Holland). Aloin of 53% purity by HPLC and aloe-emodine of 95% purity by HPLC were supplied by Sigma Chemical Co. (St. Louis, MO).

Headspace GC-MS Analysis. The equipment consisted of a headspace connected to a GC 8000 coupled with an MD 800 mass spectrometer (Fisons Instruments, Milan, Italy). All chromatographic data were recorded and processed by suitable software (Mass Lab, Fisons). The analytical column was a Mega PS 264 (50 m × 0.32 mm, film thickness = 3.0 μm). The carrier was helium with a flow rate of 2 mL/min.

The headspace sampling technique used was that previously described by Barcarolo et al. (*25*, *26*). Volatile components are driven into a cryogenic trap (capillary tube), cooled at −110 °C by liquid nitrogen, which is connected to the capillary gas chromatograph. Desorption of volatile components takes place by rapid heating of the trap to 240 °C (5s) and transfer of analytes to the analytical column.

During the transfer of the volatile components the temperature of the column was held at 40 °C; then, after 6 min, the column oven was programmed at 5 °C/min to 180 °C, which was held for 5 min, then at 7 °C/min to 200 °C, held for 2 min, and finally at 7 °C/min to 240 °C, held for 20 min. Transfer line temperature was kept at 250 °C.

The mass spectrometer was scanned from *m*/*z* 29 to 300 with a cycle time of 0.5 s. The ion source was maintained at 200 °C, and spectra were obtained by electron impact (70 eV). Identification of compounds was carried out by comparison of retention times and mass spectra of standards, when available, or tentatively from the MS spectra in comparison with members of the NIST library. Quantification of each component of the GC profile was made by the internal standard method, using ethyl propionate purchased from Sigma Chemical Co.

Preparation of Sample Solution. *HPLC Sample.* The crystalline sample of aloe, without specification of purity (50 mg), suitably powdered, was dissolved in 40% aqueous ethanol (20 mL). The sample was sonicated in an ultrasonic bath for 10 min. The solution was used for HPLC analysis after filtration with a 0.22 μm PTFE membrane (Lida, Kenosha, WI).

GC Sample. The powdered aloe (5 g) was exactly weighed into a 100 mL vial and sealed with an aluminum−rubber septum (Supelco, Bellefonte, PA). Internal standard (0.02 μL of ethyl propionate) was added to the vials, which were conditioned in an oven at 105 °C for 30 min before analysis. Headspace stripping was carried out for 150 s with helium at a rate of 8 mL/min.

Table 1. Compounds Identified by HPLC in Different Aloe Exudates[a]

t_R (min)		B (a)	B (b)	B (c)	C (a)	C (b)	C (c)	K (a)	K (b)	M (a)	M (b)	M (c)	PE (a)	PE (b)	PE (c)
3.65	aloesin	+	+	+	+	+				+	+	+	+	+	+
4.00	8-*C*-glucosyl-(*S*)-aloesol									+				+	
7.75	8-*O*-methyl-7-hydroxyaloin B			+											
7.96	8-*O*-methyl-7-hydroxyaloin A			+											
8.57	aloenin							+	+						
16.18	aloeresin A	+	+		+	+				+	+	+	+	+	+
21.86	aloin B	+	+	+	+	+	+	+	+	+	+	+	+	+	+
24.05	aloin A	+	+	+	+	+	+	+	+	+	+	+	+	+	+
24.33	isoaloeresin D			+				+	+						
25.97	aloinoside B						+	+	+		+	+			
27.98	aloinoside A						+	+	+		+	+			
44.02	aloe-emodin			+	+				+	+			+		+

[a] B, *Aloe barbadensis*; C, Cape aloe; K, Kenya aloe; M, Mosselbay aloe; PE, Port Elizabeth aloe; a−c, individual samples.

RESULTS AND DISCUSSION

The reversed phase HPLC gradient elution system allows the simultaneous separation of many components of aloe because of the large differences in hydrophobicity, due to the presence of glycoside and aglycon derivatives of each class of compounds. The characteristic HPLC profile of an alcoholic solution of aloe is shown in Figure 1, and the structures of the relative identified substances are shown in Figure 2.

Table 1 lists identified peaks for each aloe sample. The identification of the peaks was carried out either by comparison with standards when possible (aloin A, aloin B, and aloe-emodin) or by comparison of the spectral data with those in the literature [aloesin (*9*), aloeresin A (*10*), aloenin (*27*), aloinosides A and B (*17*)] or provided by Professor Okamura (University of Fukuyama, Japan). Because the HPLC gradient elution reported by Okamura et al. (*21*) was slightly different from ours, the first step consisted in reproducing this method. We then verified the retention time and absorption spectrum of each component to compare them with those obtained by our method. This permitted us to definitively assign the identity of several peaks. The use of DAD also permitted the purity of each peak to be verified.

On the basis of the HPLC results we can state that the major compounds present are aloesin, aloeresin A, and aloins A and B, as reported in the literature (*8*, *10*, *12*). Aloesin was absent in one sample, labeled Cape aloe "c", and in both Kenya aloe samples; aloenin was present in Kenya aloe only. Aloe-emodin is a degradation product of aloins and so is not important in the characterization of commercial aloe; its quantity depends on the technique employed in preparing the exudates and on conservation conditions.

Comparison of the chromatograms from the different samples shows a high quantity of aloins in *Aloe barbadensis* and in Kenya aloe. In the case of *Aloe barbadensis* "c" and Kenya aloe we also observed a high content of isoaloeresin D. The latter peculiarity could be a reason for their not being employed in the alcoholic beverage industry, a conclusion supported by the analysis of Mosselbay aloe samples, which are widely utilized in bitter spirits formulation, although these contained similarly high amount of aloins.

For GC-MS analysis, the reversed carrier gas flow technique we used permitted us to eliminate those substances not retained by the cryogenic trap. This was reflected in the absence of a broad initial peak and in the optimal resolution of substances eluted. In Table 2 the main identified peaks are listed. *A. barbadensis* samples show a higher content of aromatic compounds, especially of aldehydes such as butanal, pentanal, hexanal, and 2-hexanal, and ketones, such as 2-butanone, 2-heptanone, and 2-octanone. In these samples we also found a high content of some substances that could be very important in a characterization, such as dimethyl sulfone, methyl acetate, methyl formate, eucalyptol, and β-thujone. Whereas Kenya aloe samples showed a high content of β-pinene and anisole, the latter was present in these samples only and could therefore be decisive in the total aroma definition of commercial aloe coming from Kenya.

Mosselbay aloe samples were characterized by a greater variability. In particular, in sample a we found dimethyl sulfone, methyl acetate, and acetophenone, which were not present in the others, whereas sample c had a high concentration of ethyl acetate. With regard to samples of Port Elizabeth origin, differences among the three samples were not so evident, but sample a showed components that were absent in the others, such as methyl formate, butanal, acetophenone, β-pinene, and camphor, which could be important in the characterization of each product.

In this study it was difficult for us to know the exact origin of each sample. For example, the sample labeled Mosselbay "a" probably came from Port Elizabeth; in fact, in this sample aloinosides were absent, whereas they are found in samples b and c according to other authors (*28*). Similarly, sample c of *A. barbadensis* was probably the only "true" *barbadensis* sample, because its HPLC profile was the same as that we recorded for an aloin standard from Sigma Chemical Co., labeled "from Curaçao aloe". In addition, some intragroup differences noticed in the amount of some compounds may depend on the age of the plants, climatic conditions, harvesting time, and shelf-life conditions. From these considerations a higher number of samples would be necessary to lead to significant evaluation of origin. Nevertheless, we can state that analysis of volatile components, in addition to the HPLC method, should be a useful analytical tool for commercial aloe characterization. In fact, we can observe that some compounds are highly represented in some types of aloe and negligible or absent in the other types. Moreover, if the overall GC results are examined, in Port Elizabeth and Mosselbay aloes, which are generally preferred in bitter

Table 2. Aroma Compound Content (Milligrams per Kilogram) of Different Commercial Aloe Extracts[a]

t_R (min)	identification	B (a)	B (b)	B (c)	C (a)	C (b)	C (c)	K (a)	K (b)	M (a)	M (b)	M (c)	PE (a)	PE (b)	PE (c)
4.25	acetaldehyde	185.79	165.30	214.52	146.75	102.49	19.37	63.32	86.26	221.03	23.60	33.75	167.51	17.39	115.59
5.08	methyl formate	4.25	12.32	9.50		5.09			3.17	13.89		3.28	3.72		
5.76	ethanol	1884.76	608.01	2774.45	3380.22	2950.39	281.94	3288.93	2545.07	967.84	383.68		2842.97	291.84	1745.68
6.76	2-propenal	7.01	3.36	26.91				3.51		10.56		1.75	18.25		1.61
6.98	acetone	1256.32	3371.37	3102.65		1847.00	82.16	772.22	476.29	1968.52		2287.86	2810.89	834.26	1017.06
7.14	2-propanol	924.29		1593.60	6257.65	7520.03	2079.02		45.68	4314.14					
8.55	methyl acetate	7.97	78.51	54.56	5.10	8.91		8.04	34.68	18.10		6.79	11.29	1.60	4.38
9.13	dimethyl sulfone	2.02	7.39	2.29						1.22				0.83	2.41
9.81	1-propanol	303.49	799.35			302.79								99.64	901.36
9.87	isobutanal			1307.83			8.54	132.25	89.07	1962.95	43.78	24.56	586.85		
10.41	2-butenal	7.50	2.64	25.63	697.50	3.06		2.18		12.64		7.44	4.53	1.60	
11.03	2,3-butanedione	21.75	10.25	31.46			6.49	5.64	10.03	4.72	16.68	4.05	11.71		9.27
11.26	3-methyl-2-butanone	187.33	40.59	41.04	20.26	85.38	5.94	40.62	59.41	164.78	8.64	12.10	138.12	3.62	24.87
11.52	butanal	27.76	4.95	105.06	44.41	8.22	1.89	5.34	5.03	19.62	2.30	7.58	17.60	1.24	1.96
11.72	2-butanone	191.04	234.53	540.15	4.74	56.24	5.44	46.66	68.51	218.24	12.97	80.37	82.33	4.29	63.6
12.20	2-methylfuran	7.77	49.29	12.69		6.34	0.74	2.30	4.23		1.32	72.93	6.5		7.58
12.48	2-methyl-3-buten-2-ol	74.38	7.14	105.42	0.77	1.81	2.73	0.38	2.09	9.73	1.12	3.63	2.31		1.38
12.68	ethyl acetate	107.36	604.33	46.89	16.90	33.56	18.71	35.46	64.49	57.8	22.09	111.34	43.42	28.36	95.21
13.11	propyl formate		7.21	2.35	1.30					7.77			0.43		3.62
13.30	isobutanol	69.51		72.73	13.42	17.07	1.82	6.09	8.52	27.60	6.94		29.80		7.10
14.60	2-butenal	99.37	1.30	30.84	2.30	1.68	2.09	7.45	4.93	2.98	0.56	3.02	5.13		
14.87	3-methylbutanal	494.57	84.99	2541.92	238.43	143.22	24.96	364.39	146.90	1061.14	35.23	27.70	469.86	10.39	45.76
15.20	butylformate	76.16	32.25	182.54	14.34	16.54	4.98	3.16	14.22	19.00	6.51		25.18	2.37	7.42
15.40	2-methylbutanal	340.21	56.85	1865.20	177.98	178.43	7.63	207.40	133.56	755.71	25.55	14.62	380.86	3.90	38.01
16.51	2-pentanone	30.58	15.87	65.93	9.58	9.65	3.61	13.04	19.33	26.17	7.26	12.52	15.34		11.23
17.05	pentanal	79.06	31.76	340.52	21.60	30.08	15.00	25.33	15.74	59.92	12.76	24.49	4.50	8.95	17.37
18.88	3-methyl-1-butanol	96.24	23.59	88.50	12.66	16.15	25.33	26.47	28.02	6.85	4.44	22.56	30.32	3.37	26.81
19.10	2-methyl-1-butanol	25.61	12.20	1275.08	6.15	7.35	3.06	14.11	12.94	27.39	1.71	4.61	14.03	0.65	14.05
19.30	4-methyl-2-pentanone	56.00		144.56	14.36	19.37	7.06		37.96	37.34	5.27	11.21	33.67	4.47	13.50
19.55	2-methyl-2-butenal	80.27	20.30	167.71	14.75	29.02	7.73	12.55	7.70	49.88	8.35	31.59	51.92	3.76	11.73
21.43	2,4-pentanedione	80.96	328.57	254.79	17.27	45.02	4.95	4.78	3.79	31.17	17.03	59.79	73.42	16.89	54.05
21.74	2-hexanone	11.15		30.96	4.09	7.73		2.70	3.84	11.34	3.77	10.52	6.94	2.41	8.49
21.96	3-hexanol			16.04	6.58	7.26		5.57	18.50	27.18	5.11	16.35	6.00	1.91	1.99
22.26	hexanal	183.59	43.69	597.54	57.11	76.36	48.13	95.83	21.83	112.01	40.89	33.94	109.65	51.61	22.61
22.65	2-methyltetrahydro-furan-3-one	4.08	11.57	26.38				9.49	8.22		0.76	2.58	0.67		3.28
23.59	2-hexenal	6.53	5.34	27.37		0.88					1.49	1.80	1.20		9.39
24.00	furfural	28.72	14.20	231.21	19.13	21.83	13.28	72.72	32.26	12.80	8.19	3.27	53.12	3.72	14.9
24.29	tetrahydro-2-furan-methanol	27.07		117.01			2.12	4.52	4.28	4.39				0.65	
25.34	1-hexanol			130.15	9.38	19.14	8.89	15.75	18.28	16.84		7.61	20.62	3.11	15.88
26.46	2-heptanone	21.43	9.91	55.90	5.91	10.30	2.64	5.53	1.03	15.90	2.32	2.13	12.18	1.19	9.34
26.89	2,5-dimethylcyclo-pentanone	59.32	20.76	59.20			1.88		15.74				12.01		
27.01	heptanal	32.22	19.72	209.22	39.32	27.89	8.10	48.79	3.15	25.40	16.65	32.42	33.25	14.64	15.97
28.11	anisole							3.53	14.61						
28.32	1-heptanol	230.22		231.24	53.35	26.00	1.72	9.69	21.45	160.86	37.14	130.29	48.88	3.67	19.88
28.93	5-methyl-2-tetrahydro-furanmethanol	11.72	3.98	27.51		2.66			1.40	3.33	3.20	9.11	3.78		2.36
29.31	6-methyl-2-heptanone	96.92	60.40	42.23			4.92			82.42	19.85	32.53	57.39		
29.49	2-heptenal			12.77	9.48		4.24						6.51		
29.59	1-octanol	113.02	79.58	73.10	24.20	31.72		9.25	6.22	56.77	28.34	96.14	51.09	11.44	35.56
30.79	2-octanone	81.51	18.21	22.27	7.09		2.32	2.72	22.40	18.99	6.68	14.73	19.95	3.96	6.86
31.00	eucalyptol	558.47	165.14	331.34	37.58	77.77	14.77	348.86	29.58	133.75	79.57	148.63	139.01	17.24	31.68
31.29	octanal	34.78	21.54							243.19	44.98	97.00			
31.35	7-methyl-4-octanol			201.15	38.88	20.90	10.52	28.79	15.43		13.91	8.94	31.34	19.32	17.03
32.75	*p*-cymene		18.80		40.41	11.12		9.24	354.53	239.83	13.22	29.79	35.37	1.67	27.16
32.98	limonene	470.80	21.96		15.49	11.00		10.53		56.61	17.78	24.92	17.50	1.27	30.16
34.55	acetophenone	280.81		60.82				5.05	13.01	20.66			4.96		
34.64	epoxylinalol		145.10		65.36	40.70	3.54	16.30	18.81	111.89	47.58	32.98	85.83	37.18	63.69
35.26	linalol	106.75	70.29	27.64	49.42	18.67		4.96	55.83		22.47		32.76	10.69	89.09
35.39	nonanal		82.62	41.52	114.47	61.65	29.11	332.80	38.84	304.29	66.93	96.58	96.01	68.88	33.44
35.82	2-isobutylnorbornane			10.04	15.67	46.20	1.25	3.39	13.07	63.09	46.94	85.39	89.86	7.65	7.91
36.00	β-thujone	4.64		25.13			0.73		2.84						2.69
36.32	β-pinene		9.94	22.80				10.43	105.28			10.90	17.50		
36.60	*p*-menth-1-ene		9.59	1495.89	14.09	7.27		3.86	49.71	62.26	6.18	19.38	9.91		17.81
38.00	2-nonenal	23.49	3.37	7.14	39.56	4.81	1.13	13.99	9.33	35.76	4.54	2.06	14.52	2.93	6.38
38.37	camphor			18.60			0.82		36.43				17.63		
39.15	mentha-1,8-diene			26.35											
39.40	2-decanone	7.86		10.27		8.27	2.26	24.76	45.87	4.63			3.02	1.96	0.37
40.11	decanal	46.49	41.78	13.68	27.95	27.86	13.55	10.62	18.88	21.19	21.13	11.58	26.77		12.88
41.64	1-*p*-menthen-9-al	77.10	38.57		52.54	63.92	3.83	34.71	60.79	85.57	36.69	37.93	170.66	8.66	14.46

[a] B, *Aloe barbadensis*l C, Cape aloe; K, Kenya aloe; M, Mosselbay aloe; PE, Port Elizabeth aloe.

spirits formulation, a major aromatic balance arises. This means that these samples showed an equilibrium in their headspace composition, because none of the aromatics is present in an elevated quantity to such a degree as to prevail over the other volatile components.

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