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Data Article

GC-HRTOF-MS dataset of metabolites extracted from sorghum and *ting* (a fermented product) produced using two strains of *Lactobacillus fermentum* (singly and in combination)

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ABSTRACT

This data article reports the untargeted metabolite profile of whole grain sorghum (*Sorghum bicolor* L.) and fermented *ting* samples obtained using two strains of *Lactobacillus fermentum*. The sorghum grains were obtained from Agricol Johannesburg (South Africa) and fermentation was done at 34 °C for 24 h. Controlled fermentation with two *Lactobacillus fermentum* strains (L. *fermentum* FUA 3165 and L. *fermentum* FUA 3321), was done using the strains singly and in combination. The samples obtained thereafter were freeze-dried and acetonitrile/methanol/water (v/v/v) were used as extraction solvent, before analyses on a gas chromatography high resolution time of flight mass spectrometry (GC–HRTOF-MS) system. Data obtained showed the presence of different compounds, classified into metabolite groups such as acids, alcohols, benzenes, furan, esters, hydrocarbons, terpenes, phytosterols, etc., with their retention time, molecular formula, observed mass and average peak areas reported herein. These

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data can be used for finding biomarkers for sorghum and their derived fermented products.

Specifications Table

Subject	*Food Science and Technology*
Specific subject area	*Fermentation, Cereal, Food Composition, Food Analysis*
Type of data	Table
How data were acquired	Using a combination of organic solvents (acetonitrile/methanol/water) [4:4:2, v/v/v], metabolites were extracted from whole grain sorghum and derived *ting* samples and analyzed on a LECO Pegasus GC–HRTOF-MS system (LECO Corporation, St Joseph, USA) equipped with an Agilent 7890A gas chromatograph (Agilent Technologies, Inc., Wilmington, DE, USA) operating in high-resolution, a Gerstel MPS multipurpose autosampler (Gerstel Inc., Mülheim an der Ruhr, Germany) and a Rxi®−5 ms column (30 m × 0.25 mm ID × 0.25 µm) (Restek, Bellefonte, USA).
Data format	Analyzed data Raw files
Parameters for data collection	Triplicate samples were collected from raw whole grain sorghum and *ting* samples fermented at 34 °C for 24 h (*i.e.* spontaneously/naturally fermented *ting, ting* fermented with L. *fermentum* FUA 3165, *ting* fermented with L. *fermentum* FUA 3321 and *ting* fermented with a combination of L. *fermentum* FUA 3165 and L. *fermentum* FUA 3321).
Description of data collection	All samples (both fermented and the raw sample) were collected immediately after freeze-drying. Metabolites were subsequently extracted from freeze-dried samples using acetonitrile/methanol/ water (4:4:2, v/v/v) and reconstituted with 1 mL of chromatographic grade methanol before being filtered into a dark amber vial. One microliter of the extract was injected into a GC–HRTOF-MS system. Metabolites were subsequently identified on NIST, Mainlib and Feihn metabolomics databases.
Data source location	Sorghum grains were sourced from Agricol (Pty) Ltd. Potchefstroom, South Africa (S26°71′83.6″E27°07′22.5″); extraction and analysis were carried out at the University of Johannesburg (Doornfontein Campus), Johannesburg, South Africa (S26 ° 11′32.6″28 ° 03′28.9″).
Data accessibility	Raw GC–HRTOF-MS data as well as the supplementary file have been deposited in Mendeley data and can be retrieved from http://dx.doi.org/10.17632/jpvxk87wp5.1

Value of the Data

- The data contributed to identification of metabolites present in whole grain sorghum and a derived fermented product (*ting*).
- The data provides an understanding of metabolite modifications occurring during natural and controlled fermentation and would be valuable to the food industry and food technology researchers working on cereal/sorghum fermentation.
- The data adds to a growing body of evidence on the transformation of compounds during food fermentation and some of the metabolites reported herein have both nutritional importance and health promoting effects.
- The data indicate that untargeted GC–HRTOF-MS analysis of fermented sorghum may lead to the discovery of compounds for the development of novel functional foods.

1. Data Description

The current data (Table 1) shows information on the metabolites in whole grain sorghum and a fermented product (*ting*) analyzed using GC–HRTOF-MS. The retention time, molecular formular, observed mass and average peak areas of each compounds were presented after comparing the GC–HRTOF-MS spectra with those from NIST, Mainlib and Feihn metabolomics databases.

2. Experimental Design, Materials and Methods

2.1. Fermentation of whole grain sorghum into ting

Sorghum (*Sorghum bicolor* L.) grains (with variety name - Titan) were milled and sieved to obtain whole grain (WG)-sorghum flour. Spontaneous fermentation of *ting* was done by mixing WG-sorghum flour with sterile distilled water (1:1, w/v) and fermentation was done at 34 °C for 24 h [1]. Controlled fermentation with two *Lactobacillus fermentum* strains (L. *fermentum* FUA 3165 and L. *fermentum* FUA 3321) was done as described in Adebo et al. [2]. These strains were earlier isolated from *ting* [3] and subsequently were grown in MRS broth using a modified method of Sekwati-Monang and Gänzle [3]. The strains were grown in 10 mL MRS broth (Hi-Media, Mumbai, India) for 24 h in an incubator (IncoShake, Labotec, Johannesburg, South Africa) with temperature set at 34 °C. The liquid culture obtained was subsequently centrifuged (Eppendorf 5702R, Merck, Johannesburg, South Africa) at 3000 rpm and 10 °C for 5 min, to obtain cells. The cells were washed thrice with sterile phosphate buffer saline (PBS) and reconstituted in 10 mL of PBS [3]. The L. *fermentum* strains (both singly and in combination) (cell counts of approximately 10^5 cfu/mL) were then inoculated in the mixture and fermentation conducted in an incubator (IncoShake, Labotec, Johannesburg, South Africa). All fermentation processes were done in triplicates and thereafter subsequent *ting* samples were freeze-dried (at −55 °C for 24 h) and ground prior to analysis.

2.2. Metabolite extraction and GC-HRTOF-MS analysis

One gram each of the samples was weighed into 50 mL centrifuge tubes and 10 mL of the extraction solvent acetonitrile/methanol/water (4:4:2, v/v/v) was added. The mixture was thoroughly agitated and sonicated in an ultrasonic bath (Scientech 704, Labotech, South Africa) for 1 h at 4 °C, followed by centrifugation (Eppendorf 5702R, Merck South Africa) at 3500 rpm for 5 min at 4 °C. The supernatant was subsequently transferred to a round bottom flask and concentrated using a rotavapor and the dried extract was reconstituted with 1 mL of chromatographic grade methanol (Merck, South Africa) and filtered into dark amber vials for analysis. The samples were subsequently analysed on a GC–HRTOF-MS system (LECO Corporation, St Joseph, MI, USA), operating in high resolution. The instrument was equipped with a Gerstel MPS multipurpose autosampler (Gerstel Inc. Germany) and Rxi®−5 ms column (30 m × 0.25 mm ID × 0.25 µm) (Restek, Bellefonte, USA). One microliter (1 µL) of sample extracts of three biological replicates were injected in a spitless mode using helium as a carrier gas, pumped at a constant flow rate of 1 mL/min. The inlet and transfer line temperatures were 250 and 225 °C, respectively while the ion source temperature was at 250 °C. The initial oven temperature was set at 70 °C, held for 0.5 min, ramped at 10 °C/min to 150 °C, held for 2 min, ramped at 10 °C/min to 330 °C and held for 3 min for the column to 'bake-out'. Solvent blanks were also done to observe for contamination and impurities. Metabolites were identified by matching the generated

Table 1
Metabolites identified in the sorghum and *ting* samples.

Rt (min)	Observed *m/z*	Metabolite name	Molecular formula	Average peak areas				
				Raw	3424	3424 (3165)	3424 (3321)	3424 (3165+3321)
Acids								
19:23	294.2555	Linoleic acid	$C_{18}H_{32}O_2$	7,740,820	ND	ND	4,506,995	ND
Alcohols								
03:06	167.0367	Benzyl alcohol, TBDMS derivative	$C_{13}H_{22}OSi$	ND	ND	194,896	198,094	131,173
24:04	259.1361	5,5-Dimethyl-1,3-dioxane-2-ethanol, TBDMS derivative	$C_{14}H_{30}O_3Si$	ND	399,772	ND	ND	ND
Aldehydes								
05:08	120.0570	Benzeneacetaldehyde	C_8H_8O	2,908,518	3,131,139	6,447,350	4,870,541	5,829,043
15:53	234.1611	3,5-di–tert-Butyl-4-hydroxybenzaldehyde	$C_{15}H_{22}O_2$	ND	ND	67,414	ND	ND
Amides								
05:23	138.0313	Etidocaine	$C_{17}H_{28}N_2O$	2,662,492	ND	ND	ND	ND
18:20	176.1063	Nonanamide	$C_9H_{19}NO$	ND	484,224	ND	ND	ND
18:23	128.1072	Hexadecanamide	$C_{16}H_{33}NO$	4,298,567	818,215	1,445,600	ND	1,389,080
20:22	198.1853	Dodecanamide	$C_{12}H_{25}NO$	ND	1,834,009	ND	ND	ND
20:40	86.0603	Myristamide, N-methyl-	$C_{15}H_{31}NO$	ND	ND	ND	265,169	ND
Amine-related compounds								
05:43	103.0629	N,N-Dimethylglycine	$C_4H_9NO_2$	987,321	1,185,134	ND	ND	ND
05:47	136.0519	3,4-methylenedioxypyrovalerone	$C_{16}H_{21}NO_3$	ND	1,533,782	ND	ND	ND
06:08	137.0468	Piperidine-2-carboxamide, 1-amino-N-mesityl-	$C_{15}H_{23}N_3O$	ND	773,586	ND	ND	ND
08:36	117.0573	m-Aminophenylacetylene	C_8H_7N	404,802	ND	ND	ND	ND
10:08	149.0836	N,N-Diethylaniline	$C_{10}H_{15}N$	ND	ND	ND	480,796	ND
10:27	151.0993	Cyclohexanone pyrrolidine enamine	$C_{10}H_{17}N$	ND	310,684	ND	ND	ND
11:51	151.0391	p-Methoxy-à-phenethylamine	$C_9H_{13}NO$	339,893	ND	ND	ND	ND
12:36	163.0991	2–tert-Butyl-6-methylaniline	$C_{11}H_{17}N$	ND	214,897	ND	ND	ND
Benzenes								
25:01	240.2322	Benzeneethanamine, 2-fluoro-á,3,4-trihydroxy-N-isopropyl-	$C_{11}H_{16}FNO_3$	212,792	692,047	275,274	ND	463,653
Cyclic compounds								
13:03	180.0782	2,3,5,6-Tetrafluoromethoxybenzene	$C_7H_4F_4O$	173,127	169,999	ND	221,158	ND
Esters								
03:02	151.0544	1,2-Ethanediol, diacetate	$C_6H_{10}O_4$	24,427,907	ND	ND	8,215,764	ND

(*continued on next page*)

Table 1 (*continued*)

Rt (min)	Observed *m/z*	Metabolite name	Molecular formula	Average peak areas				
				Raw	3424	3424 (3165)	3424 (3321)	3424 (3165+3321)
03:39	195.0502	3-Methyl-2-butenoic acid, tridec–2-ynyl ester	$C_{18}H_{30}O_2$	ND	ND	305,736	ND	ND
04:23	235.9733	Carbonic acid, octyl phenyl ester	$C_{15}H_{22}O_3$	3,538,982	ND	548,414	ND	ND
04:33	76.0476	1,2-Ethanediol, dipropanoate	$C_8H_{14}O_4$	3,513,275	ND	ND	ND	2,467,354
06:06	214.9931	l-Alanine, n-propargyloxycarbonyl-, ethyl ester	$C_9H_{13}NO_4$	ND	ND	ND	995,480	ND
06:14	173.0626	Tetrahydropyran Z-10-dodecenoate	$C_{17}H_{30}O_3$	3,670,636	3,846,290	402,863	2,869,869	2,768,347
07:00	140.0469	2-Furancarboxylic acid, 3-methyl-, methyl ester	$C_7H_8O_3$	336,835	ND	ND	ND	484,918
07:06	128.0610	Naphthalene	$C_{10}H_8$	598,900	619,239	ND	519,869	550,418
07:21	150.0787	Acetic acid, bromo–, phenyl ester	$C_8H_7BrO_2$	533,448	ND	ND	ND	ND
07:23	251.9856	Alanine, N-methyl-n-propargyloxycarbonyl-, dodecyl ester	$C_{20}H_{35}NO4$	ND	228,190	ND	ND	ND
08:50	153.0780	dl-1-Methylpiperidine-2-carboxylic acid ethyl ester	$C_9H_{17}NO_2$	ND	ND	ND	ND	1,074,456
9:24	173.1174	2,2,4-Trimethyl-1,3-pentanediol diisobutyrate	$C_{16}H_{30}O_4$	364,543	490,440	685,571	474,924	522,844
9:43	178.0990	3-Hydroxy-2,2,4-trimethylpentyl 2-methylpropanoate	$C_{12}H_{24}O_3$	520,111	685,061	917,582	596,087	744,941
09:52	136.9988	2-Butenedioic acid (Z)-, dimethyl ester	$C_6H_8O_4$	ND	403,207	ND	ND	ND
11:18	195.1253	Dibutyl methylphosphonate	$C_9H_{21}O_3P$	20,690	ND	ND	ND	ND
12:23	194.0938	Ethyl 4-ethoxybenzoate	$C_{11}H_{14}O_3$	3,624,406	3,932,212	4,991,296	2,688,712	3,682,174
13:30	209.1047	Diethyl phthalate	$C_{12}H_{14}O_4$	ND	ND	158,354	ND	ND
14:46	210.0889	Ethyl homovanillate	$C_{11}H_{14}O_4$	65,310	ND	ND	ND	ND
14:46	210.0886	Hydrocinnamic acid, 4–hydroxy-3–methoxy-, methyl ester	$C_{11}H_{14}O_4$	ND	281,090	479,849	257,208	450,210
16:15	210.0703	Aspidospermidine-3-carboxylic acid, 2,3-didehydro-1-methyl-, methyl ester, (5à,12á,19à)-	$C_{22}H_{28}N_2O_2$	94,590	ND	ND	ND	ND
17:01	223.0967	Phthalic acid, 8-chlorooctyl decyl ester	$C_{26}H_{41}ClO_4$	ND	306,921	791,483	339,323	ND
17:02	224.1002	Dibutyl phthalate	$C_{16}H_{22}O_4$	ND	ND	306,831	ND	ND

(*continued on next page*)

Table 1 (*continued*)

Rt (min)	Observed *m/z*	Metabolite name	Molecular formula	Average peak areas				
				Raw	3424	3424 (3165)	3424 (3321)	3424 (3165+3321)
17:22	268.2396	9-Hexadecenoic acid, methyl ester, (Z)-	$C_{17}H_{32}O_2$	189,854	ND	ND	ND	ND
21:06	239.2366	Octanoic acid, 2-dimethylaminoethyl ester	$C_{12}H_{25}NO_2$	377,687	ND	951,054	699,376	ND
21:43	336.3021	n-Propyl 9,12-octadecadienoate	$C_{21}H_{38}O_2$	ND	4,102,774	ND	ND	ND
21:45	338.3184	cis-9-Octadecenoic acid, propyl ester	$C_{21}H_{40}O_2$	ND	2,699,372	ND	ND	ND
22:32	200.6480	Carbonic acid, 2-dimethylaminoethyl 2-methoxyethyl ester	$C_8H_{17}NO_4$	ND	1,753,672	2,514,843	1,544,231	ND
22:45	192.0614	Carbonic acid, 2-dimethylaminoethyl isobutyl ester	$C_9H_{19}NO_3$	ND	ND	238,730	ND	ND
23:17	279.1596	Phthalic acid, dicyclohexyl ester	$C_{20}H_{26}O_4$	126,800	158,291	119,863	138,556	239,456
30:16	468.3959	Lupeol acetate	$C_{32}H_{52}O_2$	331,898	241,628	271,657	ND	257,440
Fatty acid derivatives								
20:21	226.2167	Myristic acid amide	$C_{14}H_{29}NO$	1,058,677	14,433,972	ND	ND	ND
FAEEs								
18:18	284.2709	Palmitic acid, ethyl ester	$C_{18}H_{36}O_2$	3,187,863	1,834,849	ND	ND	ND
FAMEs								
15:13	211.2057	Tridecanoic acid, 12-methyl-, methyl ester	$C_{15}H_{30}O_2$	ND	247,848	ND	ND	ND
17:37	270.2558	Palmitic acid, methyl ester	$C_{17}H_{34}O_2$	18,720,903	12,278,043	16,215,036	10,984,629	11,811,787
19:22	294.2552	9,12-Octadecadienoic acid, methyl ester	$C_{19}H_{34}O_2$	ND	5,263,382	ND	ND	ND
19:26	296.2709	trans-13-Octadecenoic acid, methyl ester	$C_{19}H_{36}O_2$	ND	5,618,353	ND	ND	ND
19:27	296.2717	cis-13-Octadecenoic acid, methyl ester	$C_{19}H_{36}O_2$	2,290,714	3,540,200	ND	ND	2,301,480
19:37	298.2867	Stearic acid, methyl ester	$C_{19}H_{38}O_2$	ND	1,273,294	1,212,922	ND	1,030,362
21:26	228.2045	Tridecanoic acid, methyl ester	$C_{14}H_{28}O_2$	286,335	ND	ND	ND	ND
23:04	356.3548	Cerotic acid methyl ester	$C_{27}H_{54}O_2$	457,864	794,013	759,293	709,138	733,315
24:35	382.3808	Lignoceric acid methyl ester	$C_{25}H_{50}O_2$	496,426	497,908	ND	ND	ND

(*continued on next page*)

Table 1 (*continued*)

Rt (min)	Observed *m/z*	Metabolite name	Molecular formula	Average peak areas				
				Raw	3424	3424 (3165)	3424 (3321)	3424 (3165+3321)
Fungicides/Herbicides/Insecticides/Pesticides								
04:40	147.9655	Chlorfenapyr	$C_{15}H_{11}BrClF_3N_2O$	930,302	ND	ND	ND	ND
10:23	175.1484	2,4,7,9-Tetramethyl-5-decyn-4,7-diol	$C_{14}H_{26}O_2$	100,538	ND	ND	ND	ND
17:43	279.1462	Metalaxyl	$C_{15}H_{21}NO_4$	3,880,783	3,986,179	5,244,149	3,562,770	4,357,238
20:31	248.0394	Fludioxonil	$C_{12}H_6F_2N_2O_2$	530,134	734,940	601,202	809,294	591,821
Furan								
07:34	120.0569	Dihydrobenzofuran	C_8H_8O	ND	ND	ND	7,806,526	ND
Hydrocarbons								
03:38	102.0552	1-Heptene, 4-methyl-	C_8H_{16}	ND	ND	ND	1,236,876	ND
06:25	359.0655	Cyclotetrasiloxane, (iodomethyl)heptamethyl-	$C_8H_{23}IO_4Si_4$	ND	ND	1,061,813	ND	ND
07:19	145.0496	1,2,4,5-Tetroxane, 3,3,6,6-tetramethyl-	$C_6H_{12}O_4$	7,235,363	10,145,571	13,226,959	8,423,453	10,093,214
08:29	141.1637	Hexadecane	$C_{16}H_{34}$	328,269	ND	ND	ND	ND
08:29	166.0635	Tridecane	$C_{13}H_{28}$	ND	401,814	ND	ND	ND
13:31	182.0821	Cyclohexanol, 2,2-dichloro-1-methyl-	$C_7H_{12}C_{l2}O$	ND	ND	ND	849,060	1,214,518
Ketones								
03:15	96.0207	4(1H)-Pyrimidinone	$C_4H_4N_2O$	2,786,884	ND	ND	ND	ND
03:15	96.0207	4-Cyclopentene-1,3-dione	$C_5H_4O_2$	ND	4,863,301	ND	4,297,218	6,952,310
06:06	128.0357	3-Acetoxy-2-methyl-pyran-4-one	$C_8H_8O_4$	ND	ND	1,628,612	ND	ND
08:49	150.0675	Ethanone, 1-(2–hydroxy-5-methylphenyl)-	$C_9H_{10}O_2$	3,995,900	1,765,853	945,216	ND	1,537,796
16:10	209.0832	1-Methyl-acridone	$C_{14}H_{11}NO$	39,979	79,927	ND	ND	ND
16:10	209.0833	4-Methyl-acridone	$C_{14}H_{11}NO$	ND	ND	ND	ND	63,121
Miscellaneous compounds								
03:22	151.0241	Oxime-, methoxy–phenyl-_	$C_8H_9NO_2$	ND	ND	ND	ND	259,999
03:38	136.1247	2-Pyrrolidinone, 5-(ethoxymethyl)-	$C_7H_{13}NO_2$	ND	ND	ND	592,985	ND
03:43	100.0409	Pyrido[2,3-*d*]pyrimidine-2,4(1H,3H)–dione, 6,7-dichloro-5-[(1-*ethylpyrrolidin*-2-*yl*)*methylamino*]−1,3-dimethyl-	$C_{16}H_{21}C_{l2}N_5O_2$	ND	3,163,741	ND	ND	ND
03:44	115.0394	N-[3,3′-D*imethoxy*-4′-(2-*piperidin*-1-*yl*-*acetylamino*)-*biphenyl*-4-*yl*]−2-piperidin-1-yl-acetamide	$C_{28}H_{38}N_4O_4$	ND	ND	ND	7,606,605	ND

(*continued on next page*)

Table 1 (*continued*)

Rt (min)	Observed *m/z*	Metabolite name	Molecular formula	Average peak areas				
				Raw	3424	3424 (3165)	3424 (3321)	3424 (3165+3321)
03:25	120.9949	Propane, 1-(chloromethoxy)−2-methyl-	$C_5H_{11}ClO$	ND	ND	231,723.5	ND	245,486
07:23	297.0460	Octamethylcyclotetrasiloxane	$C_8H_{24}O_4Si_4$	86,003	292,340	876,624	486,157	96,684
07:23	283.0488	trisiloxane, 1,1,1,5,5,5-hexamethyl-3-[(*trimethylsilyl*)*oxy*]-	$C_9H_{28}O_3Si_4$	ND	ND	ND	ND	516,456
07:34	156.0783	4–tert-Butoxystyrene	$C_{12}H_{16}O$	2,761,756	6,531,976	11,059,650	ND	7,299,878
08:39	167.0663	1-Butanol, 2-[[(1-methyl-1H-pyrrol-2-yl)methyl]amino]-	$C_{10}H_{18}N_2O$	ND	180,947	ND	ND	ND
08:48	434.0843	Dodecamethylcyclohexasiloxane	$C_{12}H_{36}O_6Si_6$	3,938,587	4,483,024	4,287,897	3,847,269	3,883,265
09:52	157.0495	1,3-Dimethyl-7,7-diphenyl-6-oxa-4-thia-2-azabicyclo[3.2.0]hept–2-ene	$C_{18}H_{17}NOS$	353,088	ND	707,267	536,547	ND
09:53	358.0682	Decamethylcyclopentasiloxane	$C_{10}H_{30}O_5Si_5$	300,166	308,866	364,179	412,638	293,075
10:08	149.0836	Vanillonitrile	$C_8H_7NO_2$	404,103	431,904	441,067	ND	419,421
10:22	192.1876	Butanamide, N-acetyl-N-(4-hydroxyphenyl)-	$C_{12}H_{15}NO_3$	ND	86,709	ND	ND	ND
11:47	508.1035	Tetradecamethylcycloheptasiloxane	$C_{14}H_{42}O_7Si_7$	6,837,698	7,642,982	8,850,394	6,258,189	6,858,344
13:02	418.0348	3-Isopropoxy-1,1,1,7,7,7-hexamethyl-3,5,5-tris(trimethylsiloxy)tetrasiloxane	$C_{18}H_{52}O_7Si_7$	749,976	884,384	1,102,092	ND	781,574
13:21	157.0886	3-Methyl-4-phenyl-1H-pyrrole	$C_{11}H_{11}N$	429,072	302,532	ND	ND	233,259
14:01	232.1822	4-Amino-7-diethylamino-chromen-2-one	$C_{13}H_{16}N_2O_2$	ND	ND	33,438	ND	ND
14:27	579.1255	Hexadecamethylcyclooctasiloxane	$C_{16}H_{48}O_8Si_8$	5,538,284	6,504,556	8,541,053	4,811,020	6,051,080
14:32	277.0233	1,3-Dioxolane, 2-pentadecyl-	$C_{18}H_{36}O_2$	102,687	ND	ND	ND	ND
15:27	127.0122	2-Butyl-1,3-dioxolane	$C_7H_{14}O_2$	3,067,053	3,622,500	4,249,587	2,865,447	3,301,935
15:47	153.0785	trans-1-Methyldecahydroquinoline	$C_{10}H_{19}N$	ND	ND	ND	ND	3,750,268
15:47	153.1639	Quinoline, decahydro-1-methyl-, cis-	$C_{10}H_{19}N$	4,421,995	ND	ND	ND	ND
16:82	443.9760	1,1,1,5,7,7,7-Heptamethyl-3,3-bis(trimethylsiloxy)tetrasiloxane	$C_{13}H_{40}O_5Si_6$	5,410,633	5,144,545	6,376,632	3,513,283	4,665,388
20:54	154.1341	3-Cyclopentylpropionamide, N,N-dimethyl-	$C_{10}H_{19}NO$	164,178	ND	415,751	326,475	288,838
22:31	169.1013	Bis(2-(Dimethylamino)ethyl) ether	$C_8H_{20}N_2O$	1,249,460	1,130,845	ND	ND	ND
22:59	303.2687	Butanamide, 2-(dimethylamino)-N-[5,8-dioxo-3-phenyl-7-(phenylmethyl)−2-oxa-6,9-diazabicyclo[10.2.2]hexadeca-10,12,14,15-tetraen-4-yl]−3-methyl-, [3R-[3R*,4S*(S*),7S*]]-	$C_{33}H_{38}N_4O_4$	147,174	ND	ND	ND	ND

(*continued on next page*)

Table 1 (*continued*)

Rt (min)	Observed *m/z*	Metabolite name	Molecular formula	Average peak areas				
				Raw	3424	3424 (3165)	3424 (3321)	3424 (3165+3321)
25:01	240.2322	Benzeneethanamine, 2-fluoro-á,3,4-trihydroxy-N-isopropyl-	$C_{11}H_{16}FNO_3$	212,792	692,047	275,274	ND	463,653
28:00	532.9913	Hexadecamethylheptasiloxane	$C_{16}H_{48}O_6Si_7$	727,189	1,350,666	865,316	379,352	2,449,784
29:56	218.2026	4,4,9,9-Tetramethyl-4,9-disilatricyclo[6.2.0.0(3,6)]decane	$C_{12}H_{18}Si_2$	130,002	ND	ND	ND	ND
Nitrogen/sulfur containing compounds								
20:35	223.0816	Mercaptoacetic acid, 2TMS derivative	$C_8H_{20}O_2SSi_2$	23,235	ND	ND	ND	ND
Phenols								
05:44	124.0519	Guaiacol	$C_7H_8O_2$	ND	2,045,932	2,456,041	ND	2,108,401
08:20	152.0833	p-Ethylguaiacol	$C_9H_{12}O_2$	220,478	ND	ND	ND	ND
09:21	154.0626	Phenol, 2,6-dimethoxy-	$C_8H_{10}O_3$	ND	454,495	435,606	ND	483,847
09:21	165.1243	Phenol, 2,6-dimethoxy-, acetate	$C_{10}H_{12}O_4$	ND	ND	ND	298,748	ND
10:21	182.0575	5–tert-Butylpyrogallol	$C_{10}H_{14}O_3$	ND	85,825	ND	ND	ND
10:31	163.0992	2-Pyrrolidinophenol	$C_{10}H_{13}NO$	ND	ND	172,094	ND	ND
12:09	206.1666	2,4-Di–tert-butylphenol	$C_{14}H_{22}O$	4,083,231	3,927,580	5,204,311	3,688,644	3,979,680
22:21	340.2394	Phenol, 2,2′-methylenebis[6-(1,1-dimethylethyl)–4-methyl-	$C_{23}H_{32}O_2$	1,117,780	137,809	1,776,491	1,234,881	1,125,653
Phytosterols								
06:08	137.0474	Pregna-5,9(11)–dien-20-ol-3-one ethylene ketal	$C_{23}H_{34}O_3$	412,227	260,391	286,454	ND	ND
28:19	400.3695	Campesterol	$C_{28}H_{48}O$	343,940	396,949	323,256	341,618	343,918
28:31	412.3694	Stigmasterol	$C_{29}H_{48}O$	298,062	458,511	412,642	421,566	ND
28:54	414.3853	ç-Sitosterol	$C_{29}H_{50}O$	485,428	546,542	500,070	468,495	497,362
Pyrazine/Pyridines								
07:52	123.0679	4(H)-Pyridine, N-acetyl-	C_7H_9NO	845,953	1,383,077	1,274,105	715,043	962,421
08:30	133.0523	3-Methyl-pyrrolo(2,3-b)pyrazine	$C_7H_7N_3$	ND	ND	583,668	437,987	554,724
13:34	169.0882	Pyridine, 3-methyl-2-phenyl-	$C_{12}H_{11}N$	ND	132,824	ND	ND	ND
Terpenes/Terpenoids								
13:37	204.1509	Berkheyaradulene	$C_{15}H_{24}$	ND	146,413	ND	112,116	137,551
25:20	341.3190	Squalene	$C_{30}H_50$	590,224	698,571	ND	ND	712,384
25:21	367.3367	Supraene	$C_{30}H_50$	ND	ND	888,934	473,966	ND
Vitamins								
26:59	416.3648	ç-Tocopherol	$C_{28}H_{48}O_2$	1,165,509	1,177,085	966,059	736,754	823,814
27:32	430.3808	dl-à-Tocopherol	$C_{29}H_{50}O_2$	420,008	507,138	399,010	301,833	357,667

FAEEs – fatty acid ethyl esters; FAME – fatty acid methyl ester; FAPE – fatty acid pentyl ester; Raw – whole grain sorghum; 3424 – spontaneously (naturally) fermented *ting*; 3165 – *ting* fermented with L. *fermentum* FUA 3165; 3321 – *ting* fermented with L. *fermentum* FUA 3321; (3165 + 3321) – *ting* fermented with a combination of L. *fermentum* FUA 3165 and L. *fermentum* FUA 3321; ND – not detected; Rt – retention time.

spectra with the NIST,[1] Mainlib[2] and Feihn[3] reference library databases on ChromaTOF-HRT® (LECO Corporation, St Joseph, MI, USA). The data reported represents the average of triplicate determinations, after a brief processing of the raw data available in the supplementary files. Parameters adopted for processing included a signal to noise ratio (S/N) of 100, similarity match of above 70% and data presented in Table 1, represents only compounds occurring at least twice in triplicate injections. The compounds were also classified into metabolite groups. Raw spectra of each compound is presented in the supplementary file.

CRediT Author Statement

Janet Adeyinka Adebiyi: Formal analysis, Investigation, Methodology, Validation, Visualization, Writing - original draft; **Patrick Berka Njobeh:** Conceptualization, Funding acquisition, Project administration, Resources, Software, Writing - review & editing; **Eugenie Kayitesi:** Conceptualization, Funding acquisition, Project administration, Resources, Writing - review & editing; **Oluwafemi Ayodeji Adebo:** Conceptualization, Data curation, Formal analysis, Investigation, Methodology, Project administration, Resources, Software, Validation, Visualization, Writing - review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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