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

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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 Rx®–5 ms column (30 m × 0.25 mm ID × 0.25 μm) (Restek, Bellefonte, USA). |
| Data format | Analyzed data |
| Parameters for data collection | Raw files |
| 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 formula, observed mass and average peak areas of each compound were presented after comparing the GC–HRTOF-MS spectra with those from NIST, Mainlib and Feihm 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, Labotec, 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 splitless 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}O Si  | ND                 | 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_3 Si| ND                 | 399,772  | ND   | ND          | ND          | ND               |
|          |              | **Aldehydes**                                                                    |                   |                    |          |      |              |              |                  |
| 05:08    | 120.0570     | Benzeneacetaldehyde                                                             | C_{8}H_{8}O       | 2,908,518          | ND       | ND   | 6,447,350   | 4,870,543   | 5,829,043        |
| 15:53    | 234.1611     | 3,5-di–tert-Butyl-4-hydroxybenzaldehyde                                         | C_{15}H_{22}O_2   | 2,662,492          | ND       | 67,414| ND          | ND          | ND               |
|          |              | **Amines**                                                                       |                   |                    |          |      |              |              |                  |
| 05:23    | 138.0629     | N,N-Dimethylglycine                                                             | C_{4}H_{9}NO_2    | 987,321            | 1,185,134| ND   | ND          | ND          | ND               |
| 05:43    | 103.0629     | N,N-Dimethylglycine                                                             | C_{4}H_{9}NO_2    | 987,321            | 1,185,134| ND   | ND          | ND          | ND               |
| 05:47    | 136.0519     | 3,4-methylenedioxyprovalerone                                                   | C_{10}H_{21}NO_3  | 1,533,782          | ND       | ND   | ND          | ND          | ND               |
| 06:08    | 137.0468     | Piperidine-2-carboxamide, 1-amino-N-mesityl-                                   | C_{15}H_{23}N_3O  | 773,586            | ND       | ND   | ND          | ND          | ND               |
| 08:36    | 117.0573     | m-Aminophenylacetylene                                                          | C_{8}H_{7}N       | 404,802            | ND       | ND   | ND          | ND          | ND               |
| 10:08    | 149.0836     | N,N-Diethylaniline                                                              | C_{10}H_{15}N     | 480,796            | ND       | ND   | ND          | ND          | ND               |
| 10:27    | 151.0993     | Cyclohexanone pyridoline enamine                                                | C_{10}H_{17}N     | 310,684            | ND       | ND   | ND          | ND          | ND               |
| 12:36    | 163.0991     | 2-tert-Butyl-6-methylaniline                                                    | C_{11}H_{17}N     | 214,897            | ND       | ND   | ND          | ND          | ND               |
|          |              | **Benzenes**                                                                     |                   |                    |          |      |              |              |                  |
| 25:01    | 240.2322     | Benzeneethanamine, 2-fluoro-4,3,4-trihydroxy-N-isopropyl-                        | C_{11}H_{16}NO_3  | 212,792            | 692,047  | ND   | 275,274     | 463,653     |                  |
|          |              | **Cyclic compounds**                                                            |                   |                    |          |      |              |              |                  |
| 13:03    | 180.0782     | 2,3,5,6-Tetrafluoromethoxybenzene                                               | C_{7}H_{4}F_4O    | 173,127            | 169,999  | ND   | 221,158     | ND          |                  |
|          |              | **Esters**                                                                       |                   |                    |          |      |              |              |                  |
| 03:02    | 151.0544     | 1,2-Ethanediol, diacetate                                                       | C_{6}H_{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-butenolic 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, dipropionate | C_{6}H_{16}O_{4} | 3,513,275 | ND | ND | ND | 2,467,354 |
| 06:06    | 214.9931     | 1-Alanine, n-propargyloxycarbonyl-, ethyl ester\(C_{6}H_{13}NO_{3}\) | ND | ND | ND | 995,480 | ND | ND |
| 06:14    | 173.0626     | Tetrahydropran Z-10-dodecenoate | C_{17}H_{30}O_{5} | 3,670,636 | 3,846,290 | 402,863 | 2,869,869 | 2,768,347 |
| 07:00    | 140.0469     | 2-Furanicarboxylic acid, 3-methyl-, methyl ester | C_{5}H_{10}O_{3} | ND | ND | ND | ND | 484,918 |
| 07:06    | 128.0610     | Naphthalene | C_{10}H_{8}O_{3} | 598,900 | 619,239 | ND | 519,869 | 550,418 |
| 07:21    | 150.0787     | Acetic acid, bromo-, phenyl ester | C_{6}H_{11}BrO_{2} | 533,448 | ND | ND | ND | ND |
| 07:23    | 251.9856     | Alanine, N-methyl-n-propargyloxycarbonyl-, dodecyl ester | C_{20}H_{35}NO_{4} | ND | 228,190 | ND | ND | ND |
| 08:50    | 153.0780     | dl-1-Methylpiperidine-2-carboxylic acid ethyl ester | C_{4}H_{7}NO_{2} | ND | ND | ND | ND | 1,074,456 |
| 11:18    | 195.1253     | Dibutyl methylphosphonate | C_{9}H_{21}O_{3}P | ND | ND | 306,921 | 791,483 | 339,323 |
| 12:23    | 194.0938     | Ethyl 4-ethoxybenzoate | C_{11}H_{16}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_{3} | NR | ND | 158,354 | ND | ND |
| 14:46    | 210.0886     | 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-methylethyl ester, (5a,12a,19a)- | C_{22}H_{38}N_{2}O_{2} | 94,590 | ND | ND | ND | ND |
| 17:01    | 223.0967     | Phthalic acid, 8-chloroctyl decyl ester | C_{28}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_{8}H_{17}NO_{4} | ND | 1,753,672 | 2,514,843 | ND | ND |
| 22:45    | 192.0614     | Carbonic acid, 2-dimethylaminoethyl isobutyl ester | C_{9}H_{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 |                   |          |       |       |       |       |
| 18:18    | 284.2709     | Palmitic acid, ethyl ester | C_{16}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     | 9,10-Octadecadienoic acid, methyl ester | C_{18}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_{32}O_{2} | 286,335 | ND | ND | ND | ND |
| 23:04    | 356.3548     | Cerotic acid methyl ester | C_{22}H_{44}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)
| 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** | | | | Raw | 3424 | 3424 (3165) | 3424 (3321) | 3424 (3165+3321) |
| 04:40    | 147.9655     | Chlorfenapyr    | C\textsubscript{15}H\textsubscript{11}BrClF\textsubscript{3}N\textsubscript{2}O | 930,302 | ND   | ND   | ND   | ND   |
| 10:23    | 175.1484     | 2,4,7,9-Tetramethyl-5-decyn-4,7-diol | C\textsubscript{14}H\textsubscript{26}O\textsubscript{3} | 100,538 | ND | ND | ND | ND |
| 17:43    | 279.1462     | Metalaxyl       | C\textsubscript{15}H\textsubscript{21}NO\textsubscript{4} | 3,880,783 | 3,986,179 | 5,244,149 | 3,562,770 | 4,357,238 |
| 20:31    | 248.0394     | Fludioxonil     | C\textsubscript{12}H\textsubscript{8}F\textsubscript{2}N\textsubscript{2}O\textsubscript{2} | 530,134 | 734,940 | 601,202 | 809,294 | 591,821 |
| **Furan** | 07:34        | Dihydrobenzofuran | C\textsubscript{6}H\textsubscript{6}O | ND | ND | ND | 7,806,526 | ND |
| **Hydrocarbons** | 03:38      | 1-Heptene, 4-methyl- | C\textsubscript{6}H\textsubscript{16} | ND | ND | ND | 1,236,876 | ND |
| 06:25    | 359.0655     | Cyclotetrasiloxane, (iodomethyl)heptamethyl- | C\textsubscript{16}H\textsubscript{23}I\textsubscript{4}Si\textsubscript{4} | ND | ND | 1,061,813 | ND | ND |
| 07:19    | 145.0496     | 1,2,4,5-Tetraoxane, 3,3,6,6-tetramethyl- | C\textsubscript{16}H\textsubscript{12}O\textsubscript{4} | 7,235,363 | 10,145,571 | 13,226,959 | 8,423,453 | 10,093,214 |
| 08:29    | 141.1637     | Hexadecane       | C\textsubscript{16}H\textsubscript{34} | 328,269 | ND | ND | ND | ND |
| 08:29    | 166.0635     | Tridecane        | C\textsubscript{13}H\textsubscript{28} | ND | 401,814 | ND | ND | ND |
| 13:31    | 182.0821     | Cyclohexanol, 2,2-dichloro-1-methyl- | C\textsubscript{12}H\textsubscript{12}Cl\textsubscript{2}O | ND | ND | 849,060 | 1,214,518 |
| **Ketones** | 03:15        | 4(1H)-Pyrimidinone | C\textsubscript{6}H\textsubscript{4}N\textsubscript{2}O | 2,786,884 | ND | ND | ND | ND |
| 03:15    | 96.0207      | 4-Cyclopentene-1,3-dione | C\textsubscript{6}H\textsubscript{12}O\textsubscript{2} | ND | 4,863,301 | ND | 4,297,218 | 6,952,310 |
| 06:06    | 128.0357     | 3-Acetoxy-2-methyl-pyrane-4-one | C\textsubscript{6}H\textsubscript{12}O\textsubscript{4} | ND | 1,628,612 | ND | ND | ND |
| 08:49    | 150.0675     | Ethanone, 1-(2-hydroxy-5-methylphenyl)- | C\textsubscript{16}H\textsubscript{10}O\textsubscript{2} | 3,995,900 | 1,765,853 | 945,216 | ND | 1,537,796 |
| 16:10    | 209.0832     | 1-Methyl-acridone | C\textsubscript{16}H\textsubscript{11}NO | 39,979 | 79,927 | ND | ND | ND |
| 16:10    | 209.0833     | 4-Methyl-acridone | C\textsubscript{16}H\textsubscript{11}NO | ND | ND | ND | 63,121 |
| **Miscellaneous compounds** | 03:22       | Oxime–, methoxy–, phenyl– | C\textsubscript{6}H\textsubscript{5}NO\textsubscript{2} | ND | ND | ND | ND | 259,999 |
| 03:38    | 136.1247     | 2-Pyrrolidinone, 5-(ethoxymethyl)- | C\textsubscript{6}H\textsubscript{11}NO\textsubscript{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\textsubscript{16}H\textsubscript{21}C\textsubscript{2}N\textsubscript{4}O\textsubscript{2} | ND | 3,163,741 | ND | ND | ND |
| 03:44    | 115.0394     | N-[3,3’-Dimethoxy-4’-(2-piperidin-1-yl-acetamido)-biphenyl-4-yl]-2-piperidin-1-yl-acetamide | C\textsubscript{28}H\textsubscript{38}N\textsubscript{4}O\textsubscript{4} | ND | ND | ND | 7,606,605 | ND |

(continued on next page)
| Rt (min) | Observed m/z | Metabolite name                                                                 | Molecular formula | Average peak areas |
|---------|--------------|--------------------------------------------------------------------------------|------------------|--------------------|
|         |              |                                                                                 | Raw              | 3424 (3165) | 3424 (3321) | 3424 (3165+3321) |
| 03:25   | 120.9949     | Propane, 1-(chloromethoxy)-2-methyl-2H,6-methanol-1,5,5,5-hexamethylene-3-[(trimethylsilyl)oxy]- | C₆H₁₁ClO        | ND          | ND          | 231,723.5     | ND          | 245,486 |
| 07:23   | 297.0460     | Octamethylcyclotetrasiloxane                                                   | C₂H₂₄O₈Si₄       | 86,003     | 292,340    | 876,624       | 486,157    | 96,684  |
| 07:23   | 283.0488     | trisiloxane, 1,1,5,5,5,5-hexamethylene-3-[(trimethylsilyl)oxy]-                | C₅H₂₆O₈Si₄       | ND          | ND          | ND          | ND          | 516,456 |
| 07:34   | 156.0783     | 4-tert-Butoxy styrene                                                           | C₁₂H₁₆O          | 2,761,756  | 6,531,976  | 11,059,650    | ND          | 7,299,878 |
| 08:39   | 167.0663     | 1-Butanol, 2-[[1-methyl-1H-pyrrrol-2-yl]methyl]lmino]-                          | C₁₀H₁₈N₂O        | ND          | 180,947    | ND          | ND          | ND      |
| 08:48   | 434.0843     | Dodecamethylcyclohexasiloxane                                                   | C₁₂H₃₆O₈Si₈      | 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            | C₁₃H₁₇NOS        | 353,088    | ND          | 707,267      | 536,547    | ND      |
| 09:53   | 358.0682     | Decamethylcyclopentasiloxane                                                    | C₁₀H₁₀O₈Si₅      | 300,166    | 308,866    | 364,179       | 412,638    | 293,075 |
| 10:08   | 149.0836     | Vanillinitrile                                                                 | C₄H₇NO₂         | 404,103    | 431,904    | 441,067       | ND          | 419,421 |
| 10:22   | 192.1876     | Butanamide, N-acetyl-N-(4-hydroxyphenyl)-                                         | C₁₂H₁₄NO₃       | ND          | 86,709     | ND          | ND          | ND      |
| 11:47   | 508.1035     | Tetradecamethylcycloheptasiloxane                                                | C₁₄H₄₂O₈Si₇      | 6,837,696  | 7,642,982  | 8,850,394     | 6,258,189  | 6,858,344 |
| 13:02   | 418.0348     | 3-Isopropoxy-1,1,7,7,7-hexamethyl-3,5,5-tris[(trimethylsilyloxy)tetrasiloxane   | C₁₈H₈₂O₈Si₇      | 749,976    | 884,384    | 1,102,092     | ND          | 781,574 |
| 13:21   | 157.0886     | 3-Methyl-4-phenyl-1H-pyrrole                                                    | C₁₁H₁₁N         | 429,072    | 302,532    | ND          | ND          | 233,259 |
| 14:01   | 232.1822     | 4-Amino-7-diethylamino-chromen-2-one                                              | C₁₃H₁₆N₂O₂       | ND          | ND          | 33,438       | ND          | ND      |
| 14:27   | 579.1255     | Hexadecamethylcyclooctasiloxane                                                  | C₁₆H₄₈O₈Si₈      | 5,538,284  | 6,504,556  | 8,541,053     | 4,811,020  | 6,051,080 |
| 14:32   | 277.0233     | 1,3-Dioxolane, 2-pentadecyl-                                                    | C₁₈H₃₆O₂         | 102,687    | ND          | ND          | ND          | ND      |
| 15:27   | 127.0122     | 2-Butyl-1,3-dioxolane                                                           | C₁₈H₄₂O₂         | 3,067,053  | 3,622,500  | 4,249,587     | 2,865,447  | 3,301,935 |
| 15:47   | 153.0785     | 2-trans-1-Methyldecachydroquinoline                                              | C₁₀H₁₉N         | ND          | ND          | ND          | ND          | 3,750,268 |
| 15:47   | 153.1639     | Quinoline, decahydro-1-methyl-, cis-                                             | C₁₀H₁₉N         | 4,421,995  | ND          | ND          | ND          | ND      |
| 16:82   | 443.9760     | 1,1,1,5,7,7-Heptamethyl-3,3-bis[(trimethylsilyloxy)tetrasiloxane                 | C₁₃H₄₀O₈Si₆      | 5,410,633  | 5,144,545  | 6,376,632     | 3,513,283  | 4,665,388 |
| 20:54   | 154.1341     | 3-Cyclopentylpropionamide, N,N-dimethyl-                                         | C₁₀H₁₉NO        | 164,178    | ND          | 415,751       | 326,475    | 288,838 |
| 22:31   | 169.1013     | Bis[2-(dimethylamino)ethyl] ether                                                | C₆H₂₀N₂O        | 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*]]- | C₃₃H₃₈N₄O₄      | 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-4,3,4-trihydroxy-N-isopropyl- | C_{11}H_{16}FNO_{3} | 212,792 | 692,047 | 275,274 | ND | 463,653 |
| 28:00    | 532.9913      | Hexadecamethylpentasiloxane | C_{10}H_{48}O_{6}Si_{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(3.6)]decan- | C_{12}H_{18}Si_{2} | 130,002 | ND | ND | ND | ND |

#### Nitrogen/sulfur containing compounds

| Rt (min) | Observed m/z | Metabolite name | Molecular formula | Average peak areas |
|----------|---------------|-----------------|-------------------|--------------------|
|          |               |                 |                   | Raw  | 3424 | 3424 (3165) | 3424 (3321) | 3424 (3165+3321) |
| 20:35    | 223.0816      | Mercaptoacetic acid, 2TMS derivative | C_{6}H_{20}O_{2}SS_{2} | 23,235 | ND | ND | ND | ND |

#### Phenols

| Rt (min) | Observed m/z | Metabolite name | Molecular formula | Average peak areas |
|----------|---------------|-----------------|-------------------|--------------------|
| 05:44    | 124.0519      | Guaiacol | C_{6}H_{12}O | ND | 2,045,932 | 2,456,041 | ND | 2,108,401 |
| 08:20    | 152.0833      | p-Tolylguaiacol | C_{7}H_{12}O_{2} | 220,478 | ND | ND | ND | ND |
| 09:21    | 154.0626      | Phenol, 2.6-dimethoxy- | C_{8}H_{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-tetra-Butylpyrogallo | C_{10}H_{14}O_{2} | 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-tetra-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[(1,1-dimethyl)4-methyl- | C_{22}H_{32}O_{2} | 1,117,780 | 137,809 | 1,776,491 | 1,234,881 | 1,125,653 |

#### Phytosterols

| Rt (min) | Observed m/z | Metabolite name | Molecular formula | Average peak areas |
|----------|---------------|-----------------|-------------------|--------------------|
| 06:08    | 137.0474      | Detoxadien-20-ol-3-one ethylene ketal | C_{23}H_{34}O_{3} | 421,227 | 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

| Rt (min) | Observed m/z | Metabolite name | Molecular formula | Average peak areas |
|----------|---------------|-----------------|-------------------|--------------------|
| 07:52    | 123.0679      | 4(H)-Pyridine, N-acetyl- | C_{6}H_{13}NO | 845,953 | 1,383,077 | 1,274,105 | 715,043 | 962,421 |
| 08:30    | 133.0523      | 3-Methyl-pyridolo(2,3-b)pyrazine | C_{7}H_{13}N_{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

| Rt (min) | Observed m/z | Metabolite name | Molecular formula | Average peak areas |
|----------|---------------|-----------------|-------------------|--------------------|
| 13:37    | 204.1509      | Berkheyaradulene | C_{15}H_{34} | ND | 146,413 | ND | 112,116 | 137,551 |
| 25:20    | 341.3190      | Squalene | C_{30}H_{50}O | 590,224 | 698,571 | ND | ND | 712,384 |
| 25:21    | 367.3367      | Supraene | C_{30}H_{50}O | ND | ND | 888,934 | 473,966 | ND |

#### Vitamins

| Rt (min) | Observed m/z | Metabolite name | Molecular formula | Average peak areas |
|----------|---------------|-----------------|-------------------|--------------------|
| 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_{28}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 pentlyl ester; Raw – whole grain sorghum; 3424 – spontaneously (naturally) fermented; 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 Mainlib² and Feihnt³ 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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