

Repeatability of methods of isotopic analysis of acetic acid and water in vinegar, and of acetic acid, water and ethanol from must fermentation in AMB

| acetic acid |                                 |                       |                       | water  |                                 |                       |                       | ethanol            |                       |
|-------------|---------------------------------|-----------------------|-----------------------|--------|---------------------------------|-----------------------|-----------------------|--------------------|-----------------------|
| Sample      | (D/H) <sub>CH<sub>3</sub></sub> | δ <sup>13</sup> C / ‰ | δ <sup>18</sup> O / ‰ | Sample | (D/H) <sub>CH<sub>3</sub></sub> | δ <sup>13</sup> C / ‰ | δ <sup>18</sup> O / ‰ | (D/H) <sub>i</sub> | δ <sup>13</sup> C / ‰ |
| vinegar 1   | 100,8                           | -27,7                 | 0,7                   | ABM 1  | 104,9                           | -25,5                 | -1,5                  | 101,9              | -25,8                 |
| vinegar 2   | 101,7                           | -27,5                 | 0,8                   | ABM 2  | 104,9                           | -25,6                 | -1,5                  | 101,5              | -26,2                 |
| vinegar 3   | 100,9                           | -27,6                 | 0,7                   | ABM 3  | 104,7                           | -25,6                 | -1,5                  | 102,1              | -25,7                 |
| vinegar 4   | 101,3                           | -27,4                 | 0,7                   | ABM 4  | 104,4                           | -26,1                 | -1,2                  | 102,5              | -25,7                 |
| vinegar 5   | 101,0                           | -27,5                 | 0,6                   | ABM 5  | 104,9                           | -25,7                 | -1,4                  | 101,7              | -25,8                 |
| vinegar 6   | 100,7                           | -27,5                 | 0,6                   | ABM 6  | 104,8                           | -25,8                 | -1,1                  | 102,3              | -25,7                 |
| vinegar 7   | 101,8                           | -27,4                 | 0,5                   | ABM 7  | 104,5                           | -25,6                 | -1,4                  | 102,5              | -25,6                 |
| vinegar 8   | 101,2                           | -27,3                 | 0,5                   | ABM 8  | 104,6                           | -25,6                 | -1,2                  | 103                | -25,7                 |
| vinegar 9   | 101,6                           | -27,3                 | 0,4                   | ABM 9  | 104,3                           | -25,8                 | -1,2                  | 102,7              | -25,8                 |
| vinegar10   | 101,0                           | -27,3                 | 0,6                   | ABM 10 | 104,8                           | -25,7                 | -1,3                  | 103                | -25,8                 |
| mean        | 101,2                           | -27,5                 | 0,6                   |        | 104,7                           | -25,7                 | -1,3                  | 102,3              | -25,8                 |
| std dev     | 0,4                             | 0,1                   | 0,1                   |        | 0,2                             | 0,2                   | 0,1                   | 0,5                | 0,2                   |

Reproducibility of methods of isotopic analysis of acetic acid and water in vinegar, and of acetic acid, water and ethanol from must fermentation in AMB. The D/H and δ<sup>13</sup>C of ABM vinegar are the values of acetic acid; those of ABM must are of the fermentation ethanol

| Sample        | (D/H) <sub>CH<sub>3</sub></sub> |       |       | SR   | δ <sup>13</sup> C / ‰ |       | Diff. | δ <sup>18</sup> O / ‰ |      | Diff. |
|---------------|---------------------------------|-------|-------|------|-----------------------|-------|-------|-----------------------|------|-------|
|               | lab1                            | lab2  | lab3  |      | lab1                  | lab2  |       | lab1                  | lab2 |       |
| vinegar 1     | 104.8                           | 105.4 | 104.8 | 0.4  | -26.3                 | -26.5 | -0.2  | 3.3                   | 3.0  | -0.3  |
| vinegar 2     | 105.0                           | 105.0 | 104.5 | 0.3  | -26.1                 | -26.2 | -0.1  | 0.5                   | 0.6  | 0.1   |
| vinegar 3     | 106.0                           | 106.0 | 104.8 | 0.7  | -26.6                 | -27.0 | -0.4  | 4.5                   | 4.2  | -0.3  |
| vinegar 4     | 106.2                           | 106.5 | 106.9 | 0.3  | -26.4                 | -26.5 | -0.1  | 6.0                   | 5.8  | -0.2  |
| vinegar 5     | 106.9                           | 106.6 | 106.6 | 0.2  | -25.3                 | -25.3 | 0.0   | 4.6                   | 4.6  | 0.0   |
| vinegar 6     | 107.7                           | 108.2 | 107.6 | 0.3  | -24.1                 | -24.3 | -0.2  | 3.6                   | 3.8  | 0.2   |
| vinegar 7     | 107.3                           | 107.9 | 107.3 | 0.3  | -24.2                 | -24.1 | 0.1   | 3.2                   | 3.4  | 0.2   |
| vinegar 8     | 108.5                           | 107.5 | 108.6 | 0.6  | -22.6                 | -22.7 | -0.1  | 1.4                   | 1.6  | 0.2   |
| vinegar 9     | 109.2                           | 109.2 | 107.9 | 0.8  | -21.5                 | -21.5 | 0.0   | 0.0                   | 0.4  | 0.4   |
| vinegar 10    | 108.9                           | 109.5 | 107.5 | 1.0  | -21.2                 | -21.4 | -0.2  | 0.0                   | 0.2  | 0.2   |
| mean          |                                 |       |       | 0.5  |                       |       | -0.1  |                       |      | 0.1   |
| ABM 1 vinegar | 105.2                           | 104.0 | 105.0 | 0.6  | -26.2                 | -26.3 | -0.1  | 2.5                   | 2.3  | -0.2  |
| ABM 2 vinegar | 104.4                           | 104.4 | 104.8 | 0.2  | -26.0                 | -26.2 | -0.2  | 3.5                   | 3.7  | 0.2   |
| ABM 3 vinegar | 106.1                           | 105.5 | 105.3 | 0.4  | -26.6                 | -26.8 | -0.2  | 5.2                   | 5.2  | 0.0   |
| ABM 4 vinegar | 106.9                           | 105.7 | 106.8 | 0.6  | -26.1                 | -26.5 | -0.4  | 6.4                   | 5.9  | -0.5  |
| ABM 5 vinegar | 108.2                           | 108.2 | 108.6 | 0.3  | -21.3                 | -21.5 | -0.2  | 1.1                   | 1.6  | 0.5   |
| mean          |                                 |       |       | 0.4  |                       |       | -0.2  |                       |      | 0.0   |
| ABM 1 must    | 101.6                           | 102.0 |       | -0.4 | -26.4                 | -26.4 | 0.0   |                       |      |       |
| ABM 2 must    | 101.3                           | 101.8 |       | -0.5 | -26.3                 | -26.4 | 0.1   |                       |      |       |
| ABM 3 must    | 103.1                           | 103.6 |       | -0.5 | -24.2                 | -24.3 | 0.1   |                       |      |       |
| ABM 4 must    | 102.4                           | 102.6 |       | -0.2 | -26.2                 | -25.9 | -0.3  |                       |      |       |
| ABM 5 must    | 102.3                           | 103.1 |       | -0.8 | -26.3                 | -26.3 | 0.0   |                       |      |       |
| mean          |                                 |       |       | -0.5 |                       |       | -0.0  |                       |      |       |