**Figure S1.** Overview of the experimental designs.
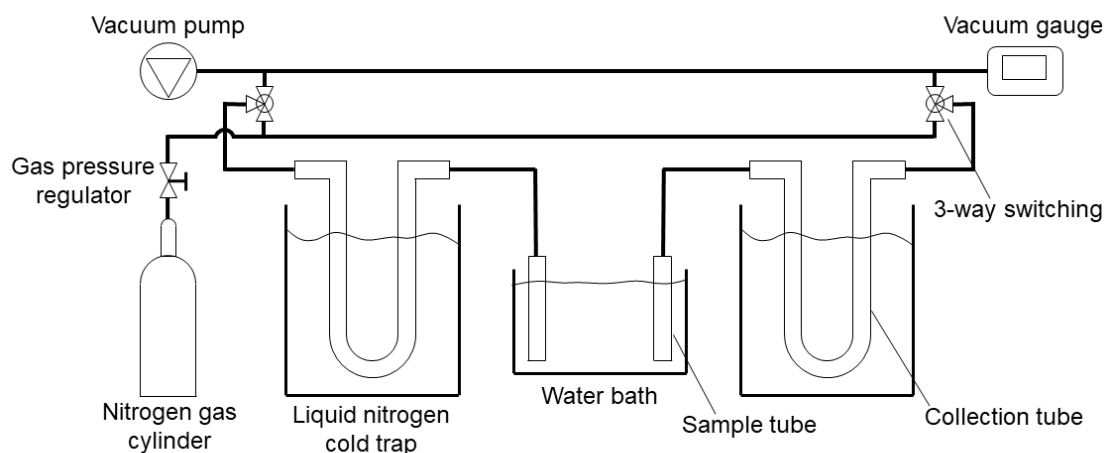
**Figure S2.** Schematic overview of the cryogenic vacuum distillation extraction setup. The setup is mainly composed of three units, i.e., sample unit, collection unit and pressure control unit. The sample unit includes sample tubes (12 ml Exetainer, Labco Ltd., Lampeter, UK) and a water bath (SBK 25D, Salvis AG, Reussbühl, Switzerland). The collection unit includes U-shaped collection tubes and liquid nitrogen cold traps (Stainless steel Dewar flask, Cole-Parmer Instrument Company, Vernon Hills, IL, US). The pressure control unit includes a vacuum pump (BS2212, Brook Crompton Ltd, Doncaster, UK), a vacuum gauge (TPG 252, Balzers) and a nitrogen gas cylinder. The setup consists of four independent extraction lines, each comprising five collection units, resulting in a total of 20 extraction slots. The lines are mainly composed of different types of Swagelok fittings (Swagelok Company, Solon, OH, US), flanges, flexible hoses, and steel tubing. An Ultra-Torr vacuum adapter (SS-12-UT-A-16, Swagelok), which was welded to a welding connector (SS-10M0-1-4W, Swagelok), was used for connections between all glassware (i.e., the sample tubes and collection tubes) and the stainless-steel tubing (10 mm diameter). Rubber O-ring was inserted between the Ultra-Torr vacuum adapter and the glassware to ensure a vacuum proof connection.

**Table S1.** Hydrogen and oxygen isotope compositions (mean ± SD) of the materials used in the experiments

| Material | $\delta^2H$ (‰) | $\delta^{18}O$ (‰) |
|---|---|---|
| Stem pieces | -210.00 ± 3.85* | 14.39 ± 0.10 |
| Stem powder | -211.07 ± 2.53* | 14.14 ± 0.18 |
| Stem segments | -207.63 ± 3.54* | 14.14 ± 0.10 |
| Stem cellulose powder | -177.02 ± 0.74* | 17.95 ± 0.25 |
| Twig pieces | -121.53 ± 1.34* | 22.44 ± 0.21 |
| Twig segments | -109.40 ± 2.08* | 20.63 ± 0.07 |
| Cellulose triacetate | -103.93 ± 3.81 | 32.66 ± 0.15 |
| Caffeine | -144.06 ± 1.24 | 11.92 ± 1.79 |

Note: Bulk organic materials were used for the isotope analyses. * marks $\delta^2H_{ne}$, which represents the calculated $\delta^2H$ of the non-exchangeable hydrogen.