**Assessing the performance of analytical methods for propolis. A collaborative trial by the International Honey Commission**

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Table S1. Chemicals and instruments used by each laboratory (continue)

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Chemicals and Instruments | Laboratory | | | |
| L01 | L03 | L05 | L06 |
| Aluminium chloride | Fluka (S.Luis, MO, USA) | Sigma - Aldrich (S.Luis, MO, USA) | Sigma-Aldrich (Burlington,MA,USA) | Sigma - Aldrich (S.Luis, MO, USA) |
| Dinitrophenylhydrazine | Sigma - Aldrich (S.Luis, MO, USA) | Sigma - Aldrich (S.Luis, MO, USA) | Sigma-Aldrich (Burlington,MA,USA) | Fluka (Buchs, Switzerland) |
| Ethanol | J.T.Baker (Gliwice, Poland) | Fisher Scientific (Leics, UK) | Merck (Darmstadt, Germany) | Fisher Scientific (Leics, UK) |
| Ferric chloride | Merck KGaA (Darmstadt, Germany) | Sigma - Aldrich (S.Luis, MO, USA) | Sigma-Aldrich (Burlington,MA,USA) | Sigma - Aldrich (S.Luis, MO, USA) |
| Folin–Ciocalteu Reagent | Sigma - Aldrich (S.Luis, MO, USA) | Sigma - Aldrich (S.Luis, MO, USA) | Sigma-Aldrich (Burlington,MA,USA) | Panreac (Barcelona, Spain) |
| Gallic acid | Sigma - Aldrich (S.Luis, MO, USA) | Sigma - Aldrich (S.Luis, MO, USA) | Merck (Darmstadt, Germany) | Sigma - Aldrich (S.Luis, MO, USA) |
| Methanol | J.T.Baker (Gliwice, Poland) | Fisher Scientific (Leics, UK) | Merck (Darmstadt, Germany) | Fisher Scientific (Leics, UK) |
| Naringenin | Sigma - Aldrich (S.Luis, MO, USA) | Sigma - Aldrich (S.Luis, MO, USA) | Sigma-Aldrich (Burlington,MA,USA) | Sigma - Aldrich (S.Luis, MO, USA) |
| Petroleum ether | Merck KGaA (Darmstadt, Germany) | Fisher Scientific (Leics, UK) | Merck (Darmstadt, Germany) | Fisher Scientific (Leics, UK) |
| Potassium hydroxide | Merck KGaA (Darmstadt, Germany) | Merck (Darmstadt, Germany) | Merck (Darmstadt, Germany) | Sigma - Aldrich (S.Luis, MO, USA) |
| Quercetin | Sigma - Aldrich (S.Luis, MO, USA) | Sigma - Aldrich (S.Luis, MO, USA) | Sigma-Aldrich (Burlington,MA,USA) | Sigma - Aldrich (S.Luis, MO, USA) |
| Sulphuric acid | LabExpert - KEFO (Ljubljana, Slovenia) | Carl Roth GmbH + Co.KG (Karlsruhe Germany) | Sigma-Aldrich (Burlington,MA,USA) | Sigma - Aldrich (S.Luis, MO, USA) |
| Sodium carbonate | Honeywell - Fluka (Muskegon, USA) | Merck (Darmstadt, Germany) | Merck (Darmstadt, Germany) | Sigma - Aldrich (S.Luis, MO, USA) |
| Muffle furnace | Elektrosanitarij (Zagreb, Croatia) | Nabertherm HTC 08/15 (Lilienthal, Germany) | Nabertherm HTC 08/15 (Lilienthal, Germany) | SNOL, Optic Ivymen System (Utena, Lithuania) |
| Rotary evaporator | Büchi (Büchi, India) | R-100 Büchi Rotavapor (Flawil, Szitzerland) | ISOLAB Evaporator - Rotary Dik tip (Eschau, Germany) | Heidolph, model Heizbad Hei-VAP (Schwabach, Germany) |
| Soxhlet apparatus | INKKO (Zagreb, Croatia) | Behr Labor Technik, Model R 106 T, (Düsseldorf, Germany) | ISOLAB Glassware (Eschau,Germany) | Behr Labor Technik, Model R 106 T, (Düsseldorf, Germany) |
| Spetrophotometer | Mettler Toledo (Greifenses, Switzerland) | Genesis 10S UV-VIS, Thermo Fisher (USA) | Schimadzu UV-1601 (Quioto, Japan) | Analytikijena, model Specord 200 (Jena, Germany) |
| Ultrasounds | - | RK 100H, Bandelin Sonorex (Berlin, Germany) | ISOLAB Ultrasonic Bath (Eschau,Germany) | J.P. Selecta (Barcelona, Spain) |
| Water purification systems | Milli - Q ultrapure water System (Darmstadt, Germany) | GenPure Pro UV/UF, Thermo Fisher (USA) | Milli - Q ultrapure water System (Darmstadt, Germany) | TGI pure system (Houston, TX, USA) |

Table S1. Chemicals and instruments used by each laboratory (continue)

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Chemicals and Instruments | Laboratory | | | |
| L07 | L08 | L09 | L10 |
| Aluminium chloride | Sigma - Aldrich (S.Luis, MO, USA) | Prolabo (Leuven, Belgium) | Sigma - Aldrich (S.Luis, MO, USA) | Fluka (Buchs, Switzerland) |
| Dinitrophenylhydrazine | Sigma - Aldrich (S.Luis, MO, USA) | Sigma - Aldrich (S.Luis, MO, USA) | Fluka (Buchs, Switzerland) | Sigma - Aldrich (S.Luis, MO, USA) |
| Ethanol | Carlo Erba (Chau. du Vexin, Val-de-Reuil, France) | Merck (Darmstadt, Germany) | Carlo Erba reagents (Milan, Italy) | VWR/BDH Chemicals (Fontenay sous Bois, France) |
| Ferric chloride | Sigma - Aldrich (S.Luis, MO, USA) | Prolabo (Leuven, Belgium) | Sigma - Aldrich (S.Luis, MO, USA) | Panreac (Barcelona, Spain) |
| Folin–Ciocalteu Reagent | Sigma - Aldrich (S.Luis, MO, USA) | Merck (Darmstadt, Germany) | Sigma - Aldrich (S.Luis, MO, USA) | VWR/BDH Chemicals (Fontenay sous Bois, France) |
| Gallic acid | Extrasynthèse (Genay, France) | Sigma - Aldrich (S.Luis, MO, USA) | Fluka (Buchs, Switzerland) | Panreac (Barcelona, Spain) |
| Methanol | Carlo Erba (Chau. du Vexin, Val-de-Reuil, France) | Merck (Darmstadt, Germany) | Carlo Erba reagents (Milan, Italy) | VWR/BDH Chemicals (Fontenay sous Bois, France) |
| Naringenin | Sigma - Aldrich (S.Luis, MO, USA) | Sigma - Aldrich (S.Luis, MO, USA) | Sigma - Aldrich (S.Luis, MO, USA) | Sigma - Aldrich (S.Luis, MO, USA) |
| Petroleum ether | - | - | Carlo Erba reagents (Milan, Italy) | VWR/BDH Chemicals (Fontenay sous Bois, France) |
| Potassium hydroxide | Merck (Darmstadt, Germany) | Prolabo (Leuven, Belgium) | Sigma - Aldrich (S.Luis, MO, USA) | Merck (Darmstadt, Germany) |
| Quercetin | Sigma - Aldrich (S.Luis, MO, USA) | Sigma - Aldrich (S.Luis, MO, USA) | Sigma - Aldrich (S.Luis, MO, USA) | Sigma - Aldrich (S.Luis, MO, USA) |
| Sulphuric acid | Fisher Scientific (Leics, UK) | Prolabo (Leuven, Belgium) | Sigma - Aldrich (S.Luis, MO, USA) | VWR/BDH Chemicals (Fontenay sous Bois, France) |
| Sodium carbonate | Merck (Darmstadt, Germany) | Prolabo (Leuven, Belgium) | Sigma - Aldrich (S.Luis, MO, USA) | Merck (Darmstadt, Germany) |
| Muffle furnace | - | Lenton (Asian) | Zetalab, ZE model (Padua, Italy) | Heraeus (Hanau, Germany) |
| Rotary evaporator | Heidolph, model Laborota 4003 ( Schwabach, Germany) | Heidolph Instruments GmbH & Co. KG ( Schwabach, Germany) | Steroglass, model Strike 202 (Padua, Italy) | Büchi, model R-124 (Flawil, Switzerland) |
| Soxhlet apparatus | - | Soxhlet extractor | Extraction Apparatus, Soxhlet Steroglass (Perugia, Italy) | Tecator, Soxtec System HT (Höganäs, Sweden) |
| Spetrophotometer | Perkin Elmer, Lambda 25 (Waltham, MA, USA) | GBC, model Cintra 202 (Australia) | JASCO Europe, model V-530 (Cremella, Italy) | Varian, Cary 400 Bio (Mulgrave, Vic, Australia) |
| Ultrasounds | - | - | - | J.P. Selecta (Barcelona, Spain) |
| Water purification systems | Millipore Rios 5 (Molsheim, France) | Ultra Clear TWF system (SG water) | Millipore Elix 5 Water Purification System Lab (Milan, Italy) | Millipore (Molsheim, France) |

Table S1. Chemicals and instruments used by each laboratory

|  |  |  |
| --- | --- | --- |
| Chemicals and Instruments | Laboratory | |
| L11 | L12 |
| Aluminium chloride | Fluka (Buchs, Switzerland) | Merck KGaA (Darmstadt, Germany) |
| Dinitrophenylhydrazine | Carlo Erba (Chau. du Vexin, Val-de-Reuil, France) | Sigma - Aldrich (S.Luis, MO, USA) |
| Ethanol | Alkaloid (Skopje, Macedonia) | Merck KGaA (Darmstadt, Germany) |
| Ferric chloride | Acros Organics (New Jersy, USA) | Merck KGaA (Darmstadt, Germany) |
| Folin–Ciocalteu Reagent | Merck KGaA (Darmstadt, Germany) | Merck KGaA (Darmstadt, Germany) |
| Gallic acid | Sigma - Aldrich (S.Luis, MO, USA) | Merck KGaA (Darmstadt, Germany) |
| Methanol | Merck KGaA (Darmstadt, Germany) | Merck KGaA (Darmstadt, Germany) |
| Naringenin | Sigma - Aldrich (S.Luis, MO, USA) | Sigma - Aldrich (S.Luis, MO, USA) |
| Petroleum ether | Valerus (Sofia, Bulgaria) | Chemlab (Zedelgem - Belgium) |
| Potassium hydroxide | Lachema ( n.p.. Brno, Chemapol-Praque, Czechoslovakia) | Merck KGaA (Darmstadt, Germany) |
| Quercetin | Sigma - Aldrich (St. Luis, MO, USA) | Sigma - Aldrich (S.Luis, MO, USA) |
| Sulphuric acid | Valerus (Sofia, Bulgaria) | Chemlab (Zedelgem - Belgium) |
| Sodium carbonate | Valerus (Sofia, Bulgaria) | Merck KGaA (Darmstadt, Germany) |
| Muffle furnace | Lab MF2 (Labor Bio Ltd, Sofia, Bulgaria) | Nabertherm HTC 08/15 Furnaces (Bremen, Germany) |
| Rotary evaporator | Büchi, model R-3 HB (Flawil, Switzerland) | Eppendorf Concentrator plus complete system (Hamburg, Germany) |
| Soxhlet apparatus | Lenz Laborglas GmbH & Co. KG (Wertheim, Germany) | Behr Labor Technik, Model R 104 S, (Düsseldorf, Germany) |
| Spetrophotometer | Thermo Electron Corporation, Helios Gamma, Model 9423 UVG 1202E (Waltham, MS, USA) | Thermo Genesys 150 uv-vis spectrophotometer (Madison, USA) |
| Ultrasounds | Elma Schmidbauer GmbH (Singen, Germany) | - |
| Water purification systems | MK Opticoelectron (Velingrad, Bulgaria) | Sartorius H2OPRO-VF-B Arium Pro UV Ultrapure Water System (Goettingen, Germany) |



Figure S1. Mendel’s *h* plot applied in the data set of the balsamic content of propolis using mechanical agitation at room temperature. Within each lab, the columns represent the 15 propolis samples. (a) Full data; (b) Second iteration after outlier’s removal.

Table S2. Number of valid laboratories per parameter/method.

|  |  |  |
| --- | --- | --- |
| Parameter | Method | Laboratories |
| Ash | - | 9 |
| Wax | Soxhlet | 9 |
| Ultrasounds | 6 |
| Balsam | Room Temperature | 9 |
| Ultrasounds | 6 |
| Phenolics | Room Temperature | 9 |
| Ultrasounds | 6 |
| Flavone/flavonol | Room Temperature | 9 |
| Ultrasounds | 6 |
| Flavanone/dihydroflavonol | Room Temperature | 8 |
| Ultrasounds | 4 |

Table S3. Outliers analysis.

|  |  |  |  |
| --- | --- | --- | --- |
| Parameter | Method | Number outliers | Valid results  % |
| Ash | - | 22 | 95 |
| Wax | Soxhlet | 18 | 94 |
| Ultrasounds | 11 | 96 |
| Balsam | Room Temperature | 6 | 99 |
| Ultrasounds | 7 | 97 |
| Phenolics | Room Temperature | 0 | 100 |
| Ultrasounds | 5 | 98 |
| Flavone/flavonol | Room Temperature | 9 | 98 |
| Ultrasounds | 18 | 93 |
| Flavanone/dihydroflavonol | Room Temperature | 5 | 99 |
| Ultrasounds | 1 | 99 |

Table S4. Average values (in percentage of raw propolis) and mean range between laboratories, after outlier exclusion

|  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Sample | Ash | | Wax | | | | Balsam | | | | Total Phenolics | | | | Flavone/Flavonol | | | | Flavanone/Dihydroflavonol | | | |
| Soxhlet | | Ultrasounds | | RT | | Ultrasounds | | RT | | Ultrasounds | | RT | | Ultrasounds | | RT | | Ultrasounds | |
| S01 | 0,9 | 0,8-1,1 | 23 | 22-23 | 23 | 19-25 | 69 | 59-76 | 69 | 65-74 | 15 | 13-17 | 15 | 11-17 | 2,2 | 1,8-2,5 | 2,4 | 1,7-3,1 | 6,8 | 2,4-11,0 | 8,8 | 7,2-10,1 |
| S02 | 1,4 | 1,1-1,4 | 21 | 19-23 | 19 | 17-21 | 72 | 65-76 | 73 | 67-79 | 18 | 14-22 | 17 | 13-20 | 5,6 | 4,9-6,2 | 5,1 | 4,1-5,7 | 8,8 | 2,9-14,3 | 10,5 | 8,8-13,5 |
| S03 | 1,2 | 1,0-1,3 | 35 | 32-37 | 32 | 26-39 | 59 | 52-64 | 51 | 38-60 | 13 | 10-15 | 12 | 14-10 | 3,6 | 3,1-4,1 | 3,2 | 3,1-3,4 | 7,0 | 1,8-10,8 | 8,4 | 7,2-9,7 |
| S04 | 0,8 | 0,6-0,9 | 30 | 28-33 | 31 | 22-40 | 62 | 58-65 | 61 | 56-66 | 12 | 9-15 | 12 | 11-13 | 2,5 | 2,2-2,9 | 2,4 | 2,1-2,7 | 7,4 | 3,2-10,2 | 8,4 | 7,2-9,5 |
| S05 | 1,4 | 1,1-1,6 | 47 | 42-51 | 46 | 38-60 | 49 | 43-54 | 48 | 43-54 | 13 | 11-15 | 13 | 12-14 | 4,4 | 3,8-5,1 | 4,4 | 4,1-4,7 | 7,0 | 2,6-9,7 | 8,3 | 7,0-9,4 |
| S06 | 2,9 | 2,8-3,3 | 19 | 17-22 | 15 | 12-17 | 59 | 54-63 | 58 | 47-68 | 11 | 9-13 | 11 | 10-12 | 3,1 | 2,7-3,5 | 3,0 | 2,8-3,1 | 7,2 | 2,6-9,7 | 7,8 | 6,3-9,9 |
| S07 | 1,8 | 1,5-2,0 | 28 | 25-32 | 25 | 21-29 | 54 | 48-60 | 55 | 45-64 | 7 | 6-8 | 7 | 6-9 | 1,2 | 0,8-1,8 | 1,1 | 1,0-1,4 | 10,4 | 5,1-18,1 | 9,1 | 5,3-11,2 |
| S08 | 2,2 | 2,0-2,3 | 40 | 33-47 | 34 | 30-41 | 56 | 50-60 | 53 | 42-59 | 11 | 9-13 | 11 | 9-14 | 2,8 | 2,2-3,2 | 2,6 | 2,3-2,8 | 7,1 | 3,6-10,0 | 8,3 | 5,1-11,3 |
| S09 | 1,0 | 0,8-1,0 | 32 | 31-32 | 31 | 27-36 | 64 | 59-70 | 64 | 58-72 | 16 | 13-21 | 16 | 15-17 | 6,2 | 5,1-8,0 | 6,4 | 5,7-8,1 | 9,6 | 7,4-13,0 | 11,0 | 7,9-14,1 |
| S10 | 0,4 | 0,2-0,4 | 10 | 7-13 | 11 | 6-15 | 72 | 65-77 | 67 | 59-71 | 17 | 15-21 | 16 | 14-19 | 6,7 | 5,7-8,4 | 6,2 | 5,0-7,0 | 10,4 | 5,1-13,1 | 10,1 | 8,7-12,4 |
| S11 | 2,4 | 1,6-3,1 | 20 | 17-23 | 19 | 17-25 | 69 | 66-75 | 70 | 67-74 | 18 | 15-24 | 19 | 16-22 | 7,5 | 6,3-9,5 | 7,2 | 6,6-7,6 | 11,1 | 6,7-14,0 | 10,0 | 9,0-11,6 |
| S12 | 1,3 | 1,1-1,4 | 28 | 25-31 | 27 | 25-30 | 64 | 62-67 | 64 | 59-68 | 16 | 14-19 | 16 | 14-19 | 6,5 | 5,3-8,2 | 6,1 | 5,7-6,7 | 8,9 | 4,8-11,6 | 9,3 | 7,2-13,3 |
| S13 | 1,0 | 0,5-0,9 | 21 | 19-24 | 21 | 16-23 | 72 | 65-79 | 70 | 68-73 | 20 | 15-26 | 19 | 17-22 | 7,7 | 6,7-9,8 | 7,3 | 6,8-8,0 | 11,6 | 9,1-13,8 | 10,5 | 7,7-14,9 |
| S14 | 0,7 | 0,2-0,4 | 18 | 14-23 | 20 | 14-27 | 73 | 68-76 | 73 | 70-75 | 23 | 19-29 | 22 | 19-25 | 8,9 | 8,2-10,0 | 8,7 | 8,0-9,7 | 13,2 | 6,9-18,2 | 12,5 | 10,3-16,6 |
| S15 | 0,7 | 0,5-0,8 | 45 | 44-45 | 42 | 36-49 | 53 | 50-57 | 53 | 51-55 | 14 | 12-19 | 14 | 12-16 | 4,0 | 3,7-4,8 | 3,9 | 3,7-4,1 | 8,5 | 4,6-11,4 | 8,2 | 5,3-11,4 |
|  | 1,3 | | 28 | | 26 | | 63 | | 62 | | 15 | | 15 | | 4,8 | | 4,7 | | 9,0 | | 9,4 | |
| Int | 0,4-2,9 | | 10-47 | | 11-46 | | 49-73 | | 48-73 | | 7-23 | | 7-22 | | 1,2-8,9 | | 1,1-8,7 | | 6,8-13,2 | | 7,8-12,5 | |

RT: Room temperature. Int: Interval range between samples

Table S5. Estimation of variance components for ash, wax and balsam content

|  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Sample | Ash | | | Wax | | | | | | Balsam | | | | | |
|  | S2replicates | S2Laboraries | Soxhlet | | | Ultrasounds | | | RT | | | Ultrasounds | | |
|  | S2replicates | S2Laboraries |  | S2replicates | S2Laboraries |  | S2replicates | S2Laboraries |  | S2replicates | S2Laboraries |
| S01 | 0,9 | 0,013 | 0,010 | 23 | 0,95 | 0,00 | 23 | 5,21 | 3,90 | 69 | 7,13 | 24,94 | 69 | 13,8 | 7,6 |
| S02 | 1,4 | 0,041 | 0,000 | 21 | 0,67 | 1,92 | 19 | 0,92 | 3,62 | 72 | 2,50 | 10,00 | 73 | 9,2 | 40,5 |
| S03 | 1,2 | 0,005 | 0,010 | 35 | 1,15 | 3,05 | 32 | 15,20 | 31,60 | 59 | 0,97 | 16,64 | 51 | 2,0 | 458,3 |
| S04 | 0,8 | 0,013 | 0,009 | 30 | 0,56 | 2,81 | 31 | 7,46 | 41,46 | 62 | 0,54 | 4,97 | 61 | 3,2 | 88,9 |
| S05 | 1,4 | 0,014 | 0,017 | 47 | 2,55 | 11,34 | 46 | 14,32 | 65,66 | 49 | 1,78 | 12,82 | 48 | 2,5 | 549,8 |
| S06 | 2,9 | 0,010 | 0,044 | 19 | 0,24 | 3,10 | 15 | 0,47 | 7,50 | 59 | 9,88 | 4,17 | 58 | 8,4 | 190,5 |
| S07 | 1,8 | 0,039 | 0,009 | 28 | 0,60 | 7,68 | 25 | 2,91 | 8,71 | 54 | 6,29 | 12,79 | 55 | 4,7 | 291,0 |
| S08 | 2,2 | 0,029 | 0,000 | 40 | 1,52 | 28,55 | 34 | 7,44 | 18,01 | 56 | 5,08 | 11,07 | 53 | 8,0 | 320,2 |
| S09 | 1,0 | 0,013 | 0,002 | 32 | 2,01 | 0,00 | 31 | 5,72 | 8,69 | 64 | 1,65 | 11,11 | 64 | 2,9 | 49,1 |
| S10 | 0,4 | 0,012 | 0,000 | 10 | 1,69 | 3,78 | 11 | 2,96 | 8,96 | 72 | 4,67 | 13,23 | 67 | 11,8 | 20,5 |
| S11 | 2,4 | 0,057 | 0,298 | 20 | 0,40 | 6,14 | 19 | 1,73 | 7,88 | 69 | 2,91 | 8,82 | 70 | 19,5 | 6,4 |
| S12 | 1,3 | 0,013 | 0,014 | 28 | 2,59 | 4,01 | 27 | 1,50 | 5,54 | 64 | 1,91 | 3,88 | 64 | 11,0 | 37,2 |
| S13 | 1,0 | 0,028 | 0,010 | 21 | 0,33 | 3,91 | 21 | 1,76 | 5,84 | 72 | 2,64 | 21,53 | 70 | 1,5 | 4,5 |
| S14 | 0,7 | 0,006 | 0,004 | 18 | 0,48 | 9,05 | 20 | 5,80 | 27,01 | 73 | 4,71 | 9,58 | 73 | 8,3 | 20,2 |
| S15 | 0,7 | 0,003 | 0,009 | 45 | 0,69 | 0,00 | 42 | 23,59 | 13,03 | 53 | 0,85 | 7,62 | 53 | 8,0 | 302,8 |
|  |  | 0,020 | 0,029 |  | 1,1 | 5,7 |  | 6,5 | 17,2 |  | 3,6 | 11,5 |  | 7,7 | 159,2 |

RT: Room temperature. S2replicates: Variance of replicate. S2Laboraries: Variance of laboratories

Table S6. Estimation of variance components for the phenolic content

|  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Sample | Total Phenolics | | | Flavone/Flavonol | | | Flavanone/Dihydroflavonol | | |
| RT | | | RT | | | RT | | |
|  | S2rep. | S2Lab. |  | S2rep. | S2Lab. |  | S2rep. | S2Lab. |
| S01 | 15 | 0,72 | 2,04 | 2,2 | 0,006 | 0,073 | 6,8 | 0,10 | 8,46 |
| S02 | 18 | 0,18 | 6,07 | 5,6 | 0,009 | 0,213 | 8,8 | 0,10 | 14,00 |
| S03 | 13 | 0,27 | 2,49 | 3,6 | 0,007 | 0,148 | 7,0 | 0,18 | 10,10 |
| S04 | 12 | 0,15 | 3,04 | 2,5 | 0,006 | 0,078 | 7,4 | 0,06 | 7,48 |
| S05 | 13 | 0,09 | 2,08 | 4,4 | 0,018 | 0,184 | 7,0 | 0,14 | 8,63 |
| S06 | 11 | 0,35 | 0,69 | 3,1 | 0,006 | 0,052 | 7,2 | 0,31 | 5,84 |
| S07 | 7 | 0,06 | 0,60 | 1,2 | 0,004 | 0,107 | 10,4 | 0,21 | 12,80 |
| S08 | 11 | 0,18 | 2,08 | 2,8 | 0,006 | 0,156 | 7,1 | 0,42 | 5,03 |
| S09 | 16 | 0,67 | 8,91 | 6,2 | 0,026 | 1,169 | 9,6 | 0,25 | 5,80 |
| S10 | 17 | 0,06 | 6,45 | 6,7 | 0,022 | 0,921 | 10,4 | 0,32 | 7,12 |
| S11 | 18 | 0,44 | 10,28 | 7,5 | 0,013 | 0,982 | 11,1 | 0,48 | 6,98 |
| S12 | 16 | 0,13 | 2,03 | 6,5 | 0,022 | 0,830 | 8,9 | 0,17 | 6,21 |
| S13 | 20 | 0,35 | 14,14 | 7,7 | 0,033 | 1,108 | 11,6 | 0,46 | 2,06 |
| S14 | 23 | 1,21 | 12,61 | 8,9 | 0,016 | 0,563 | 13,2 | 0,31 | 15,71 |
| S15 | 14 | 0,24 | 4,13 | 4,0 | 0,014 | 0,168 | 8,5 | 0,31 | 4,76 |
|  |  | 0,34 | 5,18 |  | 0,014 | 0,450 |  | 0,25 | 8,07 |

RT: Room temperature. S2rep: Variance of replicate. S2Lab: Variance of laboratories

Table S7. Percentage of results with questionable (2<Z<3) and unsatisfactory performance (Z>3) Z-Scores

|  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Laboratory | Ash | | Wax | | | | Balsam | | | | Total Phenolics | | Flavone/Flavonol | | Flavanone/Dihydroflavonol | |
| Soxhlet | | Ultrasounds | | RT | | Ultrasounds | | RT | | RT | | RT | |
|  | Q | U | Q | U | Q | U | Q | U | Q | U | Q | U | Q | U | Q | U |
| L01 | 7 | - | 7 | 7 | - | 7 | - | 7 | - | - | - | - | - | - | - | 7 |
| L03 | 14 | 7 | - | - | - | - | - | - | - | - | - | - | - | - | - | - |
| L05 | - | - |  |  | 7 | - |  |  |  |  |  |  |  |  |  |  |
| L06 | - | - | - | - | 7 | - | - | - | - | - | - | - | - | - | - | - |
| L07 |  |  |  |  |  |  | - | - | - | - | - | - | - | - | - | - |
| L08 | - | - | - | - |  |  | - | - |  |  | - | - | - | - | - | - |
| L09 | - | - | - | - |  |  | 7 | - |  |  | - | - | - | - | - | - |
| L10 | - | 20 | - | 14 |  |  | - | - | 14 | 14 | - | - | - | 14 | - | - |
| L11 | 7 | - | 7 | - | - | - | - | - |  |  | 7 | - | - | - | - | - |
| L12 | - | 7 |  |  | - | - | 7 | 7 | - | - | - | - | 7 | 47 |  |  |

Q: Questionable performance. U: Unsatisfactory performance. RT: Room temperature