

Optimization of Extraction Methods for NMR and LC-MS Metabolite Fingerprint Profiling of Botanical Ingredients in Food and Natural Health Products (NHPs)

Varathan Vinayagam ^{1,†}, Arunachalam Thirugnanasambandam ^{1,2,*,†}, Subramanyam Ragupathy ¹, Ragupathy Sneha ^{1,3} and Steven G. Newmaster ¹

1 College of Biological Science, University of Guelph, Guelph, ON N1G 2W1, Canada; vinaychemist89@gmail.com (V.V.); ragu@uoguelph.ca (S.R.); snehara@auamed.net (R.S.); snewmast@uoguelph.ca (S.G.N.)

2 Biological and Life Sciences, Canadian Light Source, Saskatoon, SK S7N 2V3, Canada

3 College of Medicine, American University of Antigua, Jobberwock Beach Road, Coolidge P.O. Box W1451, Antigua and Barbuda

* Correspondence: athiru@outlook.com

† These authors contributed equally to this work.

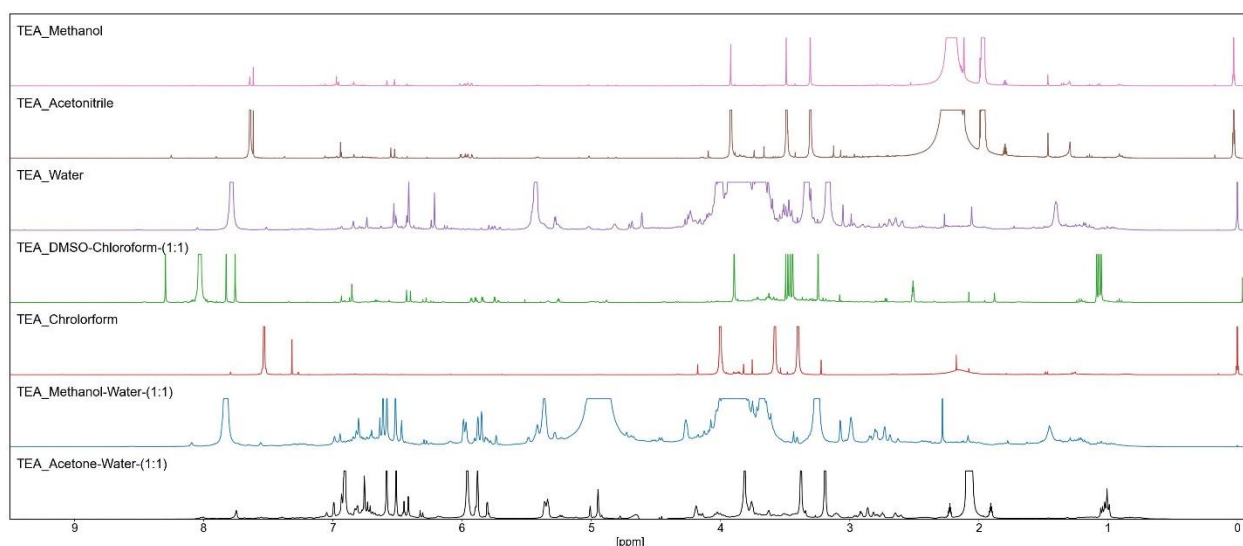


Figure S1. ¹H NMR spectra (400 MHz) of *Camellia sinensis* (tea) extracts obtained using different solvents and solvent mixtures. Spectra are displayed from top to bottom: methanol (pink), acetonitrile (brown), water (purple), DMSO-chloroform (1:1, green),

chloroform (red), methanol-water (1:1, blue), acetone-water (1:1, cyan). Chemical shifts are reported in δ (ppm) relative to TMS.

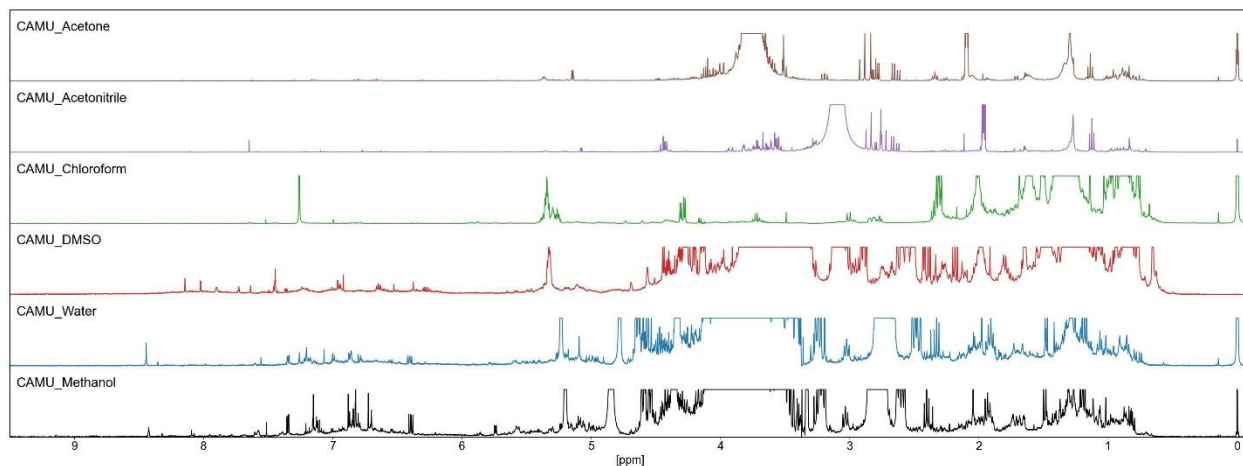


Figure S2. ¹H NMR spectra (400 MHz) of *Myrciaria dubia* (camu camu) extracts obtained using different solvents. Spectra are displayed from top to bottom: acetone (brown), acetonitrile (purple), chloroform (green), DMSO (red), water (blue), methanol (black). Chemical shifts are reported in δ (ppm) relative to TMS.

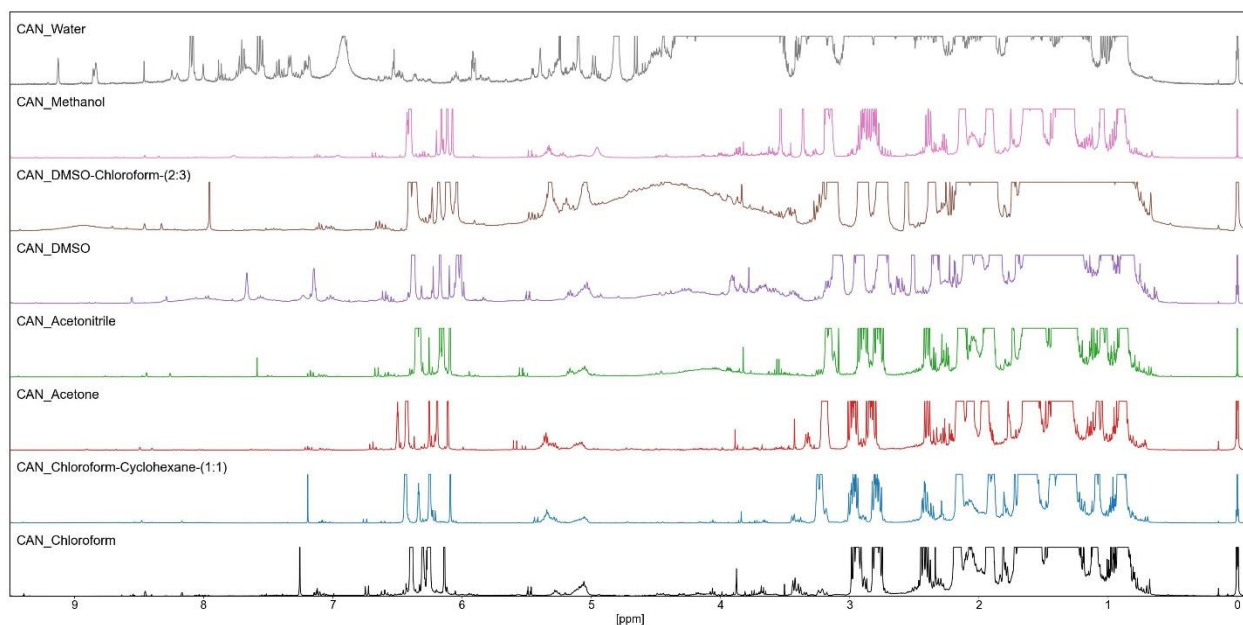
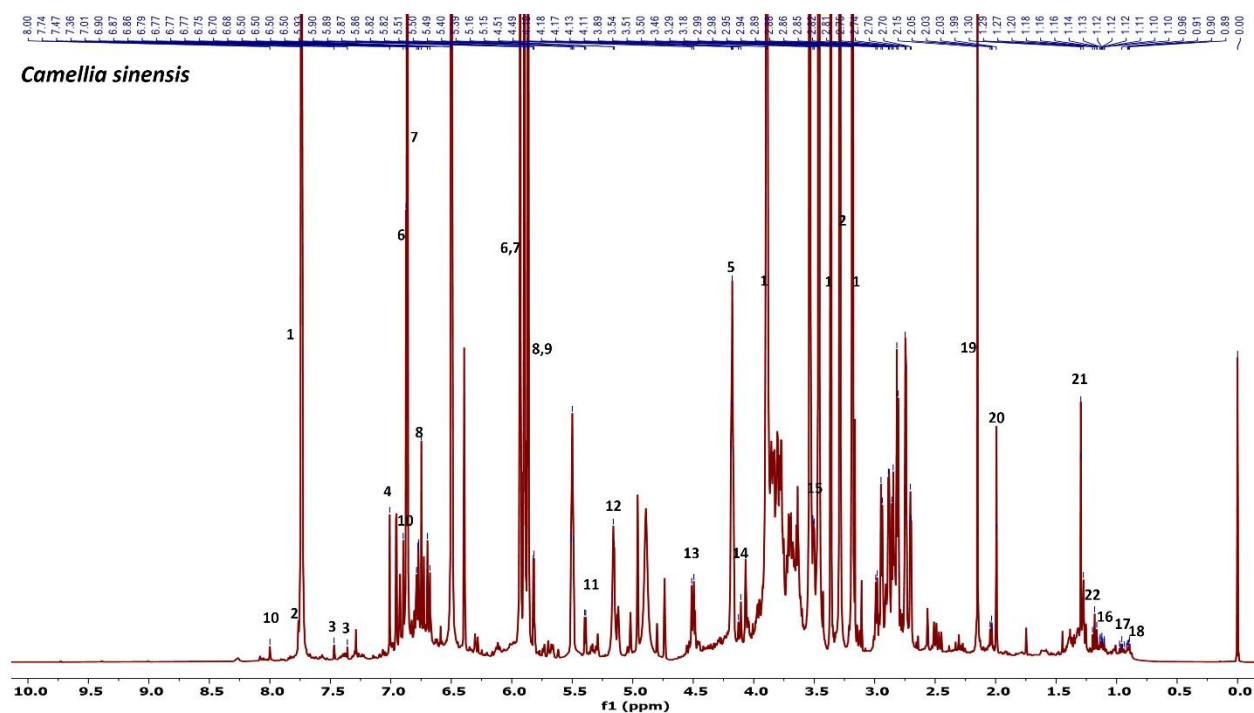
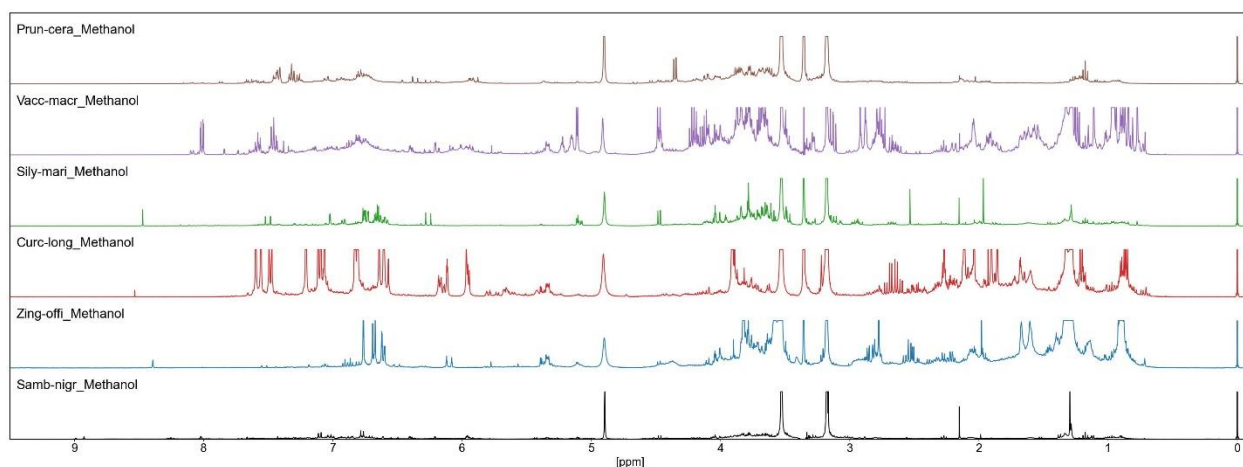
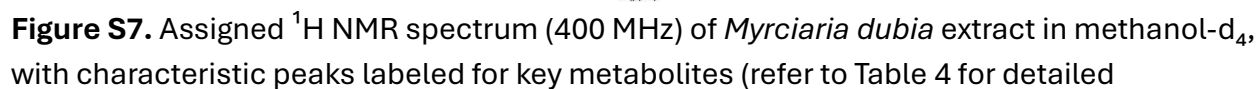
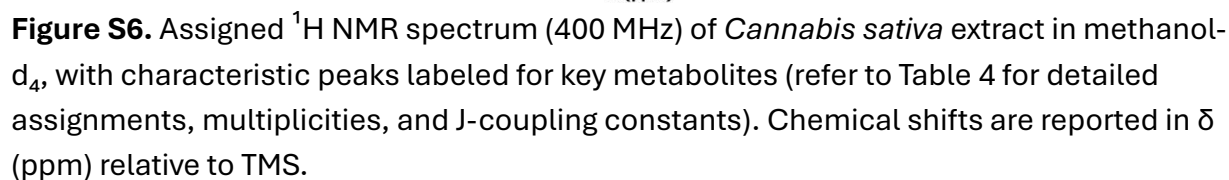


Figure S3. ¹H NMR spectra (400 MHz) of *Cannabis sativa* extracts obtained using different solvents and solvent mixtures. Spectra are displayed from top to bottom: water (black), methanol (pink), DMSO-chloroform (2:3, brown), DMSO (purple), acetonitrile (blue), acetone (green), chloroform-cyclohexane (1:1, red), chloroform (blue). Chemical shifts are reported in δ (ppm) relative to TMS.





assignments, multiplicities, and J-coupling constants). Chemical shifts are reported in δ (ppm) relative to TMS.