Electronic Supplementary Information

Evaluation of a high-resolution micro-size magic angle spinning (HRμMAS) probe for NMR-based metabolomic studies of nanolitre samples

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Table S1. Tentative peak assignments of the identified metabolites found in liver and brain extracts.

assigned index	^{1}H shift / ppm (multiplicity)	metabolite	abbreviation
1	1.33(d), 4.11(q)	lactate	lac
2	1.48(d)	alanine	ala
3	1.91(t), 2.30(t)	γ-aminobutyric acid	GABA
4	2.02(s), 2.51(dd), 2.68(dd), 4.39(dd)	N-acetyl aspartate	NAA
5	2.04(s)	N-acetylaspartylglutamate	NAAG
6	2.06(m), 2.35(m), 3.76(m)	glutamate	glu
7	2.12(m), 2.45(m), 3.76(m)	glutamine	gln
8	2.37(s)	pyruvate	
9	2.40(s)	succinate	suc
10	2.81(dd)	aspartate	asp
11	3.04(s), 3.93(s)	creatine/phosphocreatine	Cr/PCr
12	3.19(s)	choline	cho
13	3.21(s)	phosphorylcholine	PC
14	3.22(s)	glycerophosphocholine	GPC
15	3.26(m), 3.43(t)	taurine	tau
16	3.28(t), 3.55(t), 3.63(t), 4.06(m)	*myo*-inositol	m-ins
17	3.56(s)	glycine	gly
18	3.3-3.9(m), 4.65(d), 5.23(d)	glucose	glc
19	6.14(d), 8.22(s)	inosine or adenosine triphosphate	Ino/ATP
20?	3.01(m)	lysine?	lys
21	2.65(s)	citrate	–
22	3.53(dd)	glycerol	–

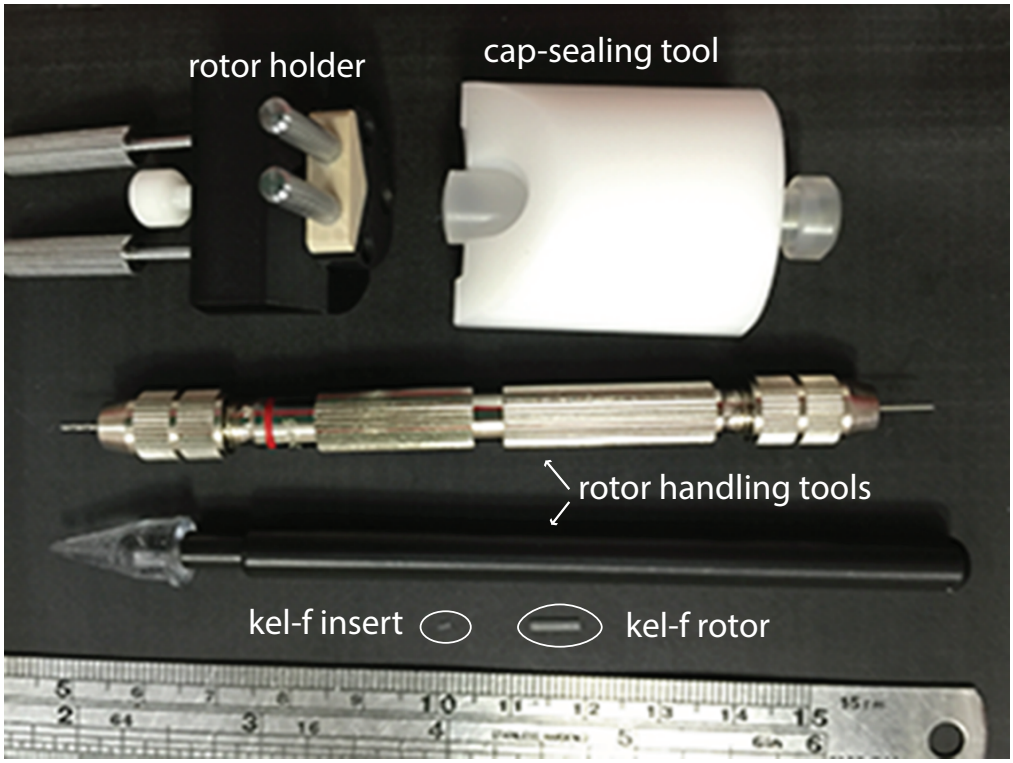

Figure S1. Custom-made tools for the sample preparations.

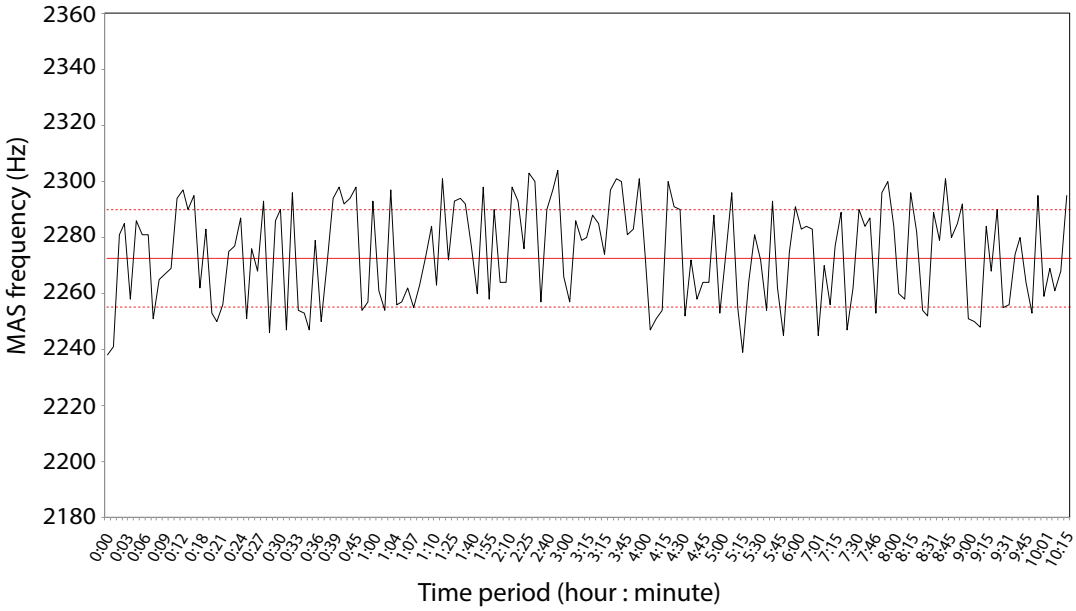

Figure S2. MAS profile of HRμMAS using a JEOL MAS pneumatic unit. It shows the fluctuation of the spinning frequency over a 10-hour timespan with a mean frequency of 2275 Hz (solid red line) with a standard deviation of ±17 Hz (dashed red lines).

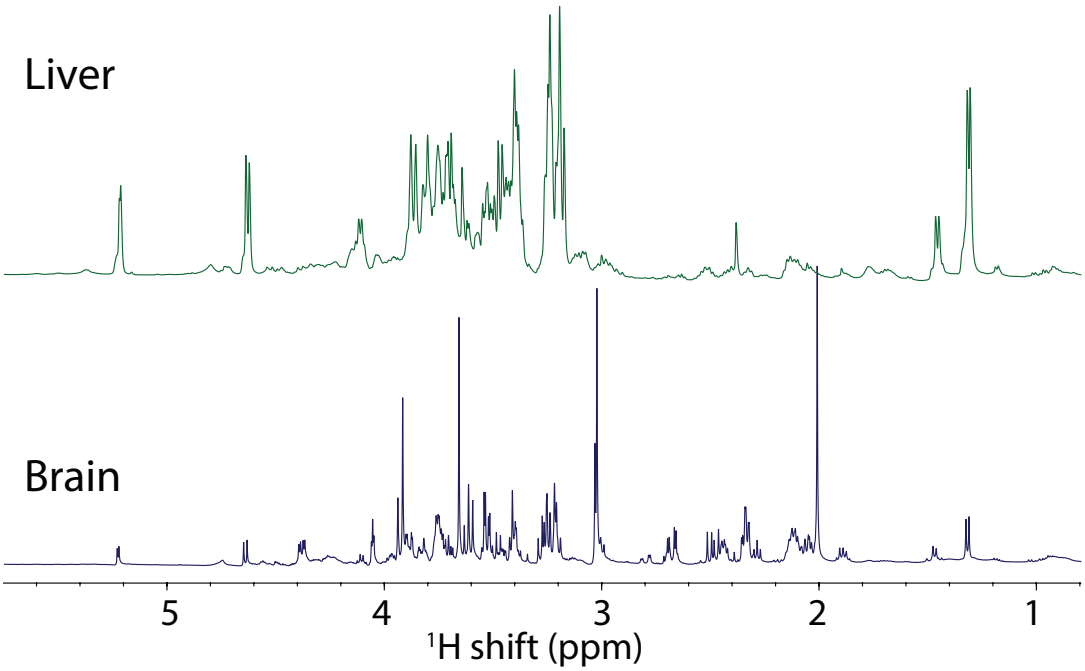

Figure S3. ^{1}H HR-MAS NMR spectra of liver and brain extract with a significant high sample mass concentration, ~7 g/ml and ~6 g/ml, respectively. The sample volume is about 50 µl. The spectra were acquired at a 500 MHz Bruker spectrometer with a water-suppression single-pulse experiment. A total of 256 scans were collected with a 8 s recycle delay.