SUPPLEMENTARY MATERIALS
Pharmaceutical Residual Solvent Analysis:
A Comparison of GC-FID and SIFT-MS Performance

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A. Materials and Methods

Table S1. Target solvents and the SIFT-MS reagent ion - primary product ion pairs used to quantify them (to avoid unnecessary complications, only the product ions used in the method are shown). Product ion formulae, mass-to charge ratios, and ion signal ratios are also shown.

Compound, Molecular Formula	Reagent Ion	Product Ion Formula	Product Ion m/z	Ion Signal Ratios	Reference*
Acetonitrile, C_2H_3N	H_3O^+	$C_2H_3N.H^+$	78	100%	[18]
Chlorobenzene, C_6H_5Cl	$O_2^{+\bullet}$	$C_6H_5{}^{35}Cl^{+\bullet}$	112	75%	[19]
	$O_2^{+\bullet}$	$C_6H_5{}^{37}Cl^{+\bullet}$	114	25%	
Chloroform, $CHCl_3$	$O_2^{+\bullet}$	$CH^{35}Cl_2^+$	83	56%	[20]
	$O_2^{+\bullet}$	$CH^{35}Cl^{37}Cl^+$	85	38%	
Cumene, C_9H_{12}	NO^+	$C_9H_{12}^{+\bullet}$	120	60%	[21]
Cyclohexane, C_6H_{12}	NO^+	$C_6H_{11}^+$	83	64%	[22]
	$O_2^{+\bullet}$	$C_6H_{12}^{+\bullet}$	84	74%	
1,2-Dichloroethene, $C_2H_2Cl_2$	H_3O^+	$C_2H_2{}^{35}Cl^{37}Cl.H^+$	99	20%	[21]
	$O_2^{+\bullet}$	$C_2H_2{}^{35}Cl_2^{+\bullet}$	96	60%	
	$O_2^{+\bullet}$	$C_2H_2{}^{35}Cl^{37}Cl^{+\bullet}$	98	35%	
1,2-Dimethoxyethane, $C_4H_{10}O_2$	H_3O^+	$C_4H_{10}O_2.H^+$	91	40%	[23]
	NO^+	$C_4H_9O_2^+$	89	80%	
1,4-Dioxane, $C_4H_8O_2$	H_3O^+	$C_4H_8O_2.H^+$	89	100%	[24]
	NO^+	$C_4H_7O_2^+$	87	55%	
	NO^+	$C_4H_8O_2^{+\bullet}$	88	45%	
Hexane, C_6H_{14}	NO^+	$C_6H_{13}^+$	85	100%	[25]
Methanol, CH_4O	H_3O^+	$CH_4O.H^+$	33	100%	[26]
Methylbutylketone, $C_6H_{12}O$	NO^+	$C_6H_{12}O.NO^+$	130	100%	[27]
Methylcyclohexane, C_7H_{14}	NO^+	$C_7H_{13}^+$	97	100%	[28]
Methylene chloride, CH_2Cl_2	$O_2^{+\bullet}$	$CH_2{}^{35}Cl_2^{+\bullet}$	84	56%	[20]
	$O_2^{+\bullet}$	$CH_2{}^{35}Cl^{37}Cl^{+\bullet}$	86	38%	
Nitromethane, CH_3NO_2	H_3O^+	$CH_3NO_2.H^+$	62	100%	[29]
Pyridine, C_5H_5N	H_3O^+	$C_5H_5N.H^+$	80	100%	[18]
Tetrahydrofuran, C_4H_8O	NO^+	$C_4H_7O^+$	71	100%	[23]
Tetralin, C_8H_{10}	NO^+	$C_{10}H_{12}^{+\bullet}$	132	100%	[30]
	$O_2^{+\bullet}$	$C_8H_8^{+\bullet}$	104	35%	
Toluene, C_7H_8	NO^+	$C_7H_8^{+\bullet}$	92	100%	[31]
	$O_2^{+\bullet}$	$C_7H_8^{+\bullet}$	92	100%	

Compound, Molecular Formula	Reagent Ion	Product Ion Formula	Product Ion m/z	Ion Signal Ratios	Reference*
Trichloroethylene, C_2HCl_3	$O_2^{+\bullet}$	$C_2H^{35}Cl_3^{+\bullet}$	130	42%	[20]
	$O_2^{+\bullet}$	$C_2H^{35}Cl_2^{37}Cl^{+\bullet}$	132	42%	
Xylene (all isomers; ethylbenzene), C_8H_{10}	NO^+	$C_8H_{10}^{+\bullet}$	106	100%	[31]

* These reference numbers correspond to citations in the article itself.

Table S2. Comparison of method validation parameters, sample preparation and sample scheduling for Runs 1 and 2 in the present study.

Parameter / Activity	Run 1	Run 2
Linearity	Yes	No
Precision: Repeatability	Yes (six replicates)	Yes (non-standard: triplicate measurement at start and end)
Accuracy	Yes	Yes
Recovery	Yes	Yes
Sample preparation order	Batch-wise preparation (standard stock solution re-capped after addition to vials in each step 2 to 4): 1. Addition of all test solutions (sample solutions) and water to appropriate vials 2. Calibrations (6x pre-calibration) 3. Tablet – 50 and 100% spikes 4. Oral suspension – 50 and 100% spikes	Batch-wise preparation (standard stock solution re-capped after addition to vials in each step 2 to 5): 1. Addition of all test solutions (sample solutions) and water to appropriate vials 2. Calibrations (triplicate pre-calibration) 3. Tablet and oral suspension – 100% spikes 4. Tablet and oral suspension – 50% spike 5. Calibrations (triplicate post-calibration).
Sample run order	1. Blank 2. Calibrations x6 3. Tablet (triplicate) 4. Tablet plus 50% spike (triplicate) 5. Tablet plus 100% spike (triplicate) 6. Oral suspension (triplicate) 7. Oral suspension plus 50% spike (triplicate) 8. Oral suspension plus 100% spike (triplicate) 9. Blank.	1. Blank 2. Calibrations (triplicate pre-calibration) 3. Tablet (triplicate) 4. Tablet plus 50% spike (triplicate) 5. Tablet plus 100% spike (triplicate) 6. Oral suspension (triplicate) 7. Oral suspension plus 50% spike (triplicate) 8. Oral suspension plus 100% spike (triplicate) 9. Calibrations (triplicate post-calibration) 10. Blank.

Table S3. SIFT-MS linearity data for Class 2A solvents across the concentration levels 0.2 to 1.0. SIFT-MS response is the headspace concentration in ppmV.

Concentration Level	Acetonitrile (H$_3$O$^+$ 42)	Chlorobenzene (O$_2^{+\bullet}$ 112)	Chlorobenzene (O$_2^{+\bullet}$ 114)	Cumene (NO$^+$ 120)	Cyclohexane (NO$^+$ 83)	Cyclohexane (O$_2^{+\bullet}$ 84)	1,2-Dichloroethene (H$_3$O$^+$ 99)	1,2-Dichloroethene (O$_2^{+\bullet}$ 96)	1,2-Dichloroethene (O$_2^{+\bullet}$ 98)	1,4-Dioxane (H$_3$O$^+$ 89)	1,4-Dioxane (NO$^+$ 87)	1,4-Dioxane (NO$^+$ 88)	Methanol (H$_3$O$^+$ 33)	Methylcyclohexane (NO$^+$ 97)	Methylene chloride (O$_2^{+\bullet}$ 84)	Methylene chloride (O$_2^{+\bullet}$ 86)	Tetrahydrofuran (NO$^+$ 71)	Toluene (NO$^+$ 92)	Toluene (O$_2^{+\bullet}$ 92)	Xylene (NO$^+$ 106)
0.2	0.050	0.585	0.567	0.335	7.36	7.40	2.23	4.99	7.28	0.010	0.010	0.009	0.138	1.85	8.05	1.05	0.218	2.979	2.780	5.00
0.4	0.091	1.15	1.09	0.576	13.2	12.6	3.63	9.60	13.5	0.019	0.019	0.018	0.251	3.41	13.6	1.88	0.444	5.69	5.38	9.48
0.6	0.140	1.95	1.82	0.982	22.6	20.7	6.12	16.2	23.4	0.031	0.029	0.035	0.402	5.62	22.7	3.06	0.695	9.51	9.32	15.7
0.8	0.204	2.81	2.62	1.36	32.4	28.3	9.06	23.1	32.5	0.047	0.043	0.051	0.590	7.72	31.4	4.31	1.04	13.1	13.9	22.4
1	0.251	3.60	3.39	1.73	42.5	36.0	12.6	28.5	40.4	0.060	0.051	0.056	0.688	10.4	40.3	5.65	1.28	16.0	17.3	28.4
R^2	0.995	0.995	0.994	0.994	0.991	0.995	0.976	0.996	0.996	0.993	0.993	0.972	0.991	0.992	0.994	0.993	0.995	0.997	0.993	0.996

Table S4. SIFT-MS linearity data for Class 2B solvents across the concentration levels 0.2 to 1.0. SIFT-MS response is the headspace concentration in ppmV.

Concentration Level	Chloroform ($O_2^{+\bullet}$ 83)	Chloroform ($O_2^{+\bullet}$ 85)	1,2-Dimethoxyethane (H_3O^+ 91)	1,2-Dimethoxyethane (NO^+ 89)	Hexane (NO^+ 85)	Methylbutylketone (NO^+ 130)	Nitromethane (H_3O^+ 62)	Pyridine (H_3O^+ 80)	Tetralin (NO^+ 132)	Tetralin ($O_2^{+\bullet}$ 104)	Trichloroethylene ($O_2^{+\bullet}$ 130)	Trichloroethylene ($O_2^{+\bullet}$ 132)
0.2	1.87	0.88	0.124	0.005	0.205	0.017	0.005	0.006	0.137	0.147	0.164	0.399
0.4	3.03	1.57	0.218	0.011	0.312	0.032	0.010	0.014	0.254	0.270	0.295	0.765
0.6	5.46	2.56	0.376	0.018	0.598	0.050	0.015	0.025	0.440	0.485	0.541	1.41
0.8	7.79	3.56	0.503	0.025	0.794	0.071	0.023	0.039	0.645	0.688	0.757	2.13
1	10.4	4.64	0.627	0.032	1.02	0.090	0.029	0.051	0.810	0.866	0.967	2.82
R^2	0.986	0.994	0.996	0.999	0.987	0.996	0.993	0.992	0.993	0.994	0.993	0.988

Table S5. GC-FID linearity data for Class 2A solvents across the concentration levels 0.2 to 1.0. GC-FID response is peak area. Gray values are not included in calculation of the linear regression coefficient, R^2.

Concentration Level	Acetonitrile	Chlorobenzene	Cumene	Cyclohexane	1,2-Dichloroethene	1,4-Dioxane	Methanol	Methylcyclohexane	Methylene chloride	Tetrahydrofuran	Toluene	Xylene
0.2	26352	1398280	386063	12264657	2007969	8324	62394	3684143	502816	1620569	5718237	6685712
0.4	46001	2264558	516951	18427392	3480099	17038	122188	5053745	852648	2901097	9542764	10146391
0.6	72255	3540812	813707	27743840	5166345	26725	194393	7698166	1315194	4370155	14621950	15824366
0.8	95148	4921204	1160739	37788433	6931718	37925	264046	11149814	1785599	5899704	19814090	21887560
1	86494	5640296	1204886	41735663	8289759	30686	203853	11159772	2069340	6960050	22944892	24404144
R^2	0.997	0.990	0.956	0.984	0.998	0.997	0.998	0.943	0.994	0.997	0.994	0.984

Table S6. GC-FID linearity data for Class 2B solvents across the concentration levels 0.2 to 1.0. GC-FID response is peak area. Gray values are not included in calculation of the linear regression coefficient, R^2.

Concentration Level	Chloroform	1,2-Dimethoxyethane	Hexane	Methylbutylketone	Nitromethane	Pyridine	Tetralin	Trichloroethylene
0.2	366116	18965	595394	44537	4587	4084	561991	126248
0.4	600573	26759	993657	70460	8073	13874	960476	204932
0.6	971247	36331	1442340	114791	10560	25599	1480855	306953
0.8	1358627	39338	1952682	162888	14707	40171	2045261	423603
1	1453777	37075	2374673	180496	16826	31369	2311282	506267
R^2	0.973	0.964	0.998	0.981	0.992	0.992	0.990	0.996

C. Repeatability

Table S7a. SIFT-MS repeatability data for Class 2A solvents (Run 1) at concentration level 1.0. SIFT-MS response is the headspace concentration in ppmV.

Replicate Number and Statistical Parameters	Acetonitrile (H_3O^+ 42)	Chlorobenzene ($O_2^{+\bullet}$ 112)	Chlorobenzene ($O_2^{+\bullet}$ 114)	Cumene (NO^+ 120)	Cyclohexane (NO^+ 83)	Cyclohexane ($O_2^{+\bullet}$ 84)	1,2-Dichloroethene (H_3O^+ 99)	1,2-Dichloroethene ($O_2^{+\bullet}$ 96)	1,2-Dichloroethene ($O_2^{+\bullet}$ 98)	1,4-Dioxane (H_3O^+ 89)	1,4-Dioxane (NO^+ 87)	1,4-Dioxane (NO^+ 88)	Methanol (H_3O^+ 33)	Methylcyclohexane (NO^+ 97)	Methylene chloride ($O_2^{+\bullet}$ 84)	Methylene chloride ($O_2^{+\bullet}$ 86)	Tetrahydrofuran (NO^+ 71)	Toluene (NO^+ 92)	Toluene ($O_2^{+\bullet}$ 92)	Xylene (NO^+ 106)
Repl. 1	0.265	3.75	3.62	2.06	50.5	42.5	13.6	28.0	43.8	0.061	0.052	0.055	0.741	13.3	48.4	6.15	1.35	16.1	17.5	31.4
Repl. 2	0.249	3.50	3.38	1.82	45.4	37.8	13.0	27.3	41.0	0.055	0.054	0.054	0.694	11.5	42.9	5.87	1.30	15.6	17.1	28.7
Repl. 3	0.276	3.72	3.53	1.85	43.5	37.6	11.6	28.3	42.1	0.069	0.056	0.063	0.796	11.4	42.6	5.69	1.39	16.2	17.7	29.6
Repl. 4	0.247	3.44	3.32	1.79	43.9	38.4	12.2	27.4	40.5	0.052	0.049	0.058	0.683	11.2	43.0	5.81	1.24	15.5	16.9	28.1
Repl. 5	0.242	3.54	3.29	1.81	43.4	37.8	11.4	27.3	40.1	0.055	0.053	0.064	0.711	11.4	42.6	5.60	1.25	15.3	16.9	28.3
Repl. 6	0.267	3.67	3.47	1.95	44.2	38.7	11.6	27.6	42.3	0.061	0.058	0.063	0.783	12.0	43.9	5.72	1.36	16.0	17.3	29.6
Mean	*0.258*	*3.60*	*3.43*	*1.88*	*45.1*	*38.8*	*12.3*	*27.6*	*41.6*	*0.059*	*0.054*	*0.060*	*0.734*	*11.8*	*43.9*	*5.81*	*1.31*	*15.8*	*17.2*	*29.3*
SD	*0.012*	*0.116*	*0.117*	*0.096*	*2.481*	*1.698*	*0.812*	*0.377*	*1.251*	*0.006*	*0.003*	*0.004*	*0.043*	*0.714*	*2.059*	*0.176*	*0.056*	*0.334*	*0.298*	*1.112*
%RSD	*4.8*	*3.2*	*3.4*	*5.1*	*5.5*	*4.4*	*6.6*	*1.4*	*3.0*	*9.5*	*5.3*	*7.1*	*5.8*	*6.1*	*4.7*	*3.0*	*4.3*	*2.1*	*1.7*	*3.8*

Table S7b. SIFT-MS repeatability data for Class 2A solvents (Run 2) at concentration level 1.0 made in triplicate before and after the Run 2 accuracy and recovery exercise. SIFT-MS response is the headspace concentration in ppmV. Additional calculations on pre- and post-run triplicate measurements (compared to Run 1; Table S5a) illustrate the challenge of this method (loss of volatiles causing drift).

Replicate Number and Statistical Parameters	Acetonitrile (H_3O^+ 42)	Chlorobenzene ($O_2^{+\cdot}$ 112)	Chlorobenzene ($O_2^{+\cdot}$ 114)	Cumene (NO^+ 120)	Cyclohexane (NO^+ 83)	Cyclohexane ($O_2^{+\cdot}$ 84)	1,2-Dichloroethene (H_3O^+ 99)	1,2-Dichloroethene ($O_2^{+\cdot}$ 96)	1,2-Dichloroethene ($O_2^{+\cdot}$ 98)	1,4-Dioxane (H_3O^+ 89)	1,4-Dioxane (NO^+ 87)	1,4-Dioxane (NO^+ 88)	Methanol (H_3O^+ 33)	Methylcyclohexane (NO^+ 97)	Methylene chloride ($O_2^{+\cdot}$ 84)	Methylene chloride ($O_2^{+\cdot}$ 86)	Tetrahydrofuran (NO^+ 71)	Toluene (NO^+ 92)	Toluene ($O_2^{+\cdot}$ 92)	Xylene (NO^+ 106)
Repl. 1 (Pre)	0.287	3.53	3.31	1.73	25.5	22.7	9.12	26.8	35.1	0.064	0.061	0.210	0.773	7.05	26.4	19.9	1.50	16.4	17.2	29.4
Repl. 2 (Pre)	0.262	3.47	3.32	1.75	28.5	23.7	11.3	26.7	34.5	0.064	0.060	0.188	0.700	7.24	27.6	19.5	1.38	16.9	17.4	29.5
Repl. 3 (Pre)	0.259	3.33	3.16	1.77	26.9	23.2	8.97	25.6	34.5	0.057	0.054	0.178	0.703	7.55	26.9	18.2	1.28	16.1	16.5	29.1
Repl. 4 (Post)	0.253	3.12	2.98	1.42	22.1	19.5	8.61	24.2	30.7	0.057	0.058	0.184	0.697	5.39	22.3	17.8	1.32	15.0	15.2	25.6
Repl. 5 (Post)	0.278	3.16	3.01	1.44	20.7	18.7	7.25	24.1	30.8	0.063	0.055	0.209	0.758	5.20	21.4	18.5	1.44	14.9	15.3	26.1
Repl. 6 (Post)	0.251	3.08	2.92	1.41	22.3	19.3	9.76	24.0	30.2	0.059	0.052	0.191	0.681	5.23	22.2	17.9	1.32	14.8	15.1	25.2
Mean (All)	*0.265*	*3.28*	*3.11*	*1.59*	*24.3*	*21.2*	*9.18*	*25.2*	*32.6*	*0.061*	*0.057*	*0.194*	*0.718*	*6.28*	*24.5*	*18.6*	*1.37*	*15.7*	*16.1*	*27.5*
SD (All)	*0.013*	*0.174*	*0.158*	*0.164*	*2.82*	*2.05*	*1.23*	*1.20*	*2.08*	*0.003*	*0.003*	*0.012*	*0.034*	*1.02*	*2.54*	*0.80*	*0.076*	*0.819*	*0.958*	*1.87*
%RSD (All)	*5.0*	*5.3*	*5.1*	*10.3*	*11.6*	*9.7*	*13.4*	*4.7*	*6.4*	*5.0*	*5.6*	*6.3*	*4.7*	*16.2*	*10.4*	*4.3*	*5.6*	*5.2*	*5.9*	*6.8*
Mean (Pre)	*0.270*	*3.44*	*3.26*	*1.75*	*27.0*	*23.2*	*9.81*	*26.4*	*34.7*	*0.062*	*0.059*	*0.192*	*0.725*	*7.28*	*27.0*	*19.2*	*1.39*	*16.5*	*17.0*	*29.4*
SD (Pre)	*0.013*	*0.084*	*0.073*	*0.016*	*1.23*	*0.41*	*1.08*	*0.54*	*0.28*	*0.003*	*0.003*	*0.013*	*0.034*	*0.21*	*0.49*	*0.73*	*0.090*	*0.330*	*0.386*	*0.17*
%RSD (Pre)	*4.7*	*2.4*	*2.2*	*0.93*	*4.5*	*1.8*	*11.0*	*2.1*	*0.82*	*5.4*	*5.2*	*7.0*	*4.7*	*2.8*	*1.8*	*3.8*	*6.5*	*2.0*	*2.3*	*0.58*
Mean (Post)	*0.261*	*3.12*	*2.97*	*1.42*	*21.7*	*19.2*	*8.54*	*24.1*	*30.6*	*0.060*	*0.055*	*0.195*	*0.712*	*5.27*	*22.0*	*18.1*	*1.36*	*14.9*	*15.2*	*25.7*
SD (Post)	*0.012*	*0.033*	*0.037*	*0.012*	*0.71*	*0.34*	*1.03*	*0.08*	*0.26*	*0.002*	*0.002*	*0.011*	*0.033*	*0.08*	*0.40*	*0.31*	*0.057*	*0.082*	*0.082*	*0.37*
%RSD (Post)	*4.7*	*1.0*	*1.3*	*0.88*	*3.3*	*1.8*	*12.0*	*0.34*	*0.86*	*3.9*	*3.9*	*5.4*	*4.7*	*1.6*	*1.8*	*1.7*	*4.2*	*0.55*	*0.54*	*1.4*
%Drift*	*96.8*	*90.6*	*91.0*	*81.3*	*80.5*	*82.6*	*87.1*	*91.4*	*88.1*	*96.8*	*94.1*	*101.4*	*98.2*	*72.4*	*81.4*	*94.1*	*98.1*	*90.5*	*89.2*	*87.4*

* Drift of post-run calibration replicates compared to pre-run calibration replicates.

Table S8a. SIFT-MS repeatability data for Class 2B solvents (Run 1) at concentration level 1.0. SIFT-MS response is the headspace concentration in ppmV.

Replicate Number and Statistical Parameters	Chloroform (O₂⁺• 83)	Chloroform (O₂⁺• 85)	1,2-Dimethoxyethane (H₃O⁺ 91)	1,2-Dimethoxyethane (NO⁺ 89)	Hexane (NO⁺ 85)	Methylbutylketone (NO⁺ 130)	Nitromethane (H₃O⁺ 62)	Pyridine (H₃O⁺ 80)	Tetralin (NO⁺ 132)	Tetralin (O₂⁺• 104)	Trichloroethylene (O₂⁺• 130)	Trichloroethylene (O₂⁺• 132)
Repl. 1	13.2	5.22	0.714	0.034	1.12	0.093	0.028	0.058	0.861	0.925	1.02	3.04
Repl. 2	11.5	4.80	0.619	0.033	1.16	0.091	0.030	0.053	0.862	0.914	1.05	2.97
Repl. 3	11.4	4.74	0.749	0.035	0.983	0.102	0.031	0.063	0.908	0.967	1.06	3.19
Repl. 4	11.1	4.83	0.618	0.034	1.05	0.091	0.029	0.052	0.822	0.904	1.05	2.95
Repl. 5	11.2	4.70	0.646	0.034	1.04	0.094	0.029	0.057	0.849	0.887	1.04	2.96
Repl. 6	11.8	4.90	0.693	0.038	1.01	0.095	0.029	0.061	0.886	0.928	1.07	3.05
Mean	*11.7*	*4.87*	*0.674*	*0.035*	*1.06*	*0.094*	*0.029*	*0.057*	*0.864*	*0.921*	*1.05*	*3.03*
SD	*0.707*	*0.171*	*0.049*	*0.002*	*0.062*	*0.004*	*0.001*	*0.004*	*0.027*	*0.025*	*0.016*	*0.083*
%RSD	*6.0*	*3.5*	*7.3*	*5.3*	*5.8*	*3.9*	*3.8*	*6.8*	*3.1*	*2.7*	*1.5*	*2.7*

Table S8b. SIFT-MS repeatability data for Class 2B solvents (Run 2) at concentration level 1.0 made in triplicate before and after the Run 2 accuracy and recovery exercise. SIFT-MS response is the headspace concentration in ppmV. Additional calculations on pre- and post-run triplicate measurements (compared to Run 1; Table S6a) illustrate the challenge of this method (loss of volatiles causing drift).

Replicate Number and Statistical Parameters	Chloroform $(O_2^{+\bullet}\ 83)$	Chloroform $(O_2^{+\bullet}\ 85)$	1,2-Dimethoxyethane $(H_3O^+\ 91)$	1,2-Dimethoxyethane $(NO^+\ 89)$	Hexane $(NO^+\ 85)$	Methylbutylketone $(NO^+\ 130)$	Nitromethane $(H_3O^+\ 62)$	Pyridine $(H_3O^+\ 80)$	Tetralin $(NO^+\ 132)$	Tetralin $(O_2^{+\bullet}\ 104)$	Trichloroethylene $(O_2^{+\bullet}\ 130)$	Trichloroethylene $(O_2^{+\bullet}\ 132)$
Repl. 1 (Pre)	7.62	19.0	0.637	0.043	5.39	10.4	0.032	0.052	1.05	0.970	1.28	3.57
Repl. 2 (Pre)	7.96	18.1	0.621	0.037	5.79	9.90	0.030	0.056	1.03	0.906	1.25	3.41
Repl. 3 (Pre)	7.84	17.2	0.609	0.036	4.99	9.51	0.029	0.058	0.976	0.886	1.20	3.24
Repl. 4 (Post)	6.17	16.8	0.559	0.038	5.21	9.51	0.029	0.058	0.957	0.858	1.07	3.08
Repl. 5 (Post)	5.90	17.8	0.594	0.040	5.05	10.2	0.033	0.064	0.945	0.862	1.09	3.09
Repl. 6 (Post)	6.13	16.8	0.547	0.035	5.35	9.38	0.031	0.057	0.915	0.842	1.09	3.00
Mean (All)	*6.94*	*17.6*	*0.595*	*0.038*	*5.30*	*9.82*	*0.031*	*0.057*	*0.979*	*0.887*	*1.16*	*3.23*
SD (All)	*0.88*	*0.784*	*0.032*	*0.003*	*0.264*	*0.381*	*0.001*	*0.004*	*0.047*	*0.042*	*0.084*	*0.201*
%RSD (All)	*12.7*	*4.5*	*5.4*	*6.9*	*5.0*	*3.9*	*4.8*	*6.3*	*4.8*	*4.8*	*7.2*	*6.2*
Mean (Pre)	*7.81*	*18.1*	*0.623*	*0.039*	*5.39*	*9.94*	*0.030*	*0.055*	*1.02*	*0.921*	*1.24*	*3.41*
SD (Pre)	*0.14*	*0.735*	*0.011*	*0.003*	*0.327*	*0.364*	*0.001*	*0.003*	*0.031*	*0.036*	*0.033*	*0.135*
%RSD (Pre)	*1.8*	*4.1*	*1.8*	*7.3*	*6.1*	*3.7*	*4.2*	*4.8*	*3.0*	*3.9*	*2.7*	*4.0*
Mean (Post)	*6.07*	*17.1*	*0.567*	*0.038*	*5.20*	*9.70*	*0.031*	*0.059*	*0.939*	*0.854*	*1.08*	*3.06*
SD (Post)	*0.12*	*0.471*	*0.020*	*0.002*	*0.123*	*0.360*	*0.002*	*0.003*	*0.018*	*0.009*	*0.009*	*0.040*
%RSD (Post)	*2.0*	*2.8*	*3.5*	*6.1*	*2.4*	*3.7*	*5.3*	*5.5*	*1.9*	*1.0*	*0.87*	*1.3*
%Drift*	*77.7*	*94.7*	*91.1*	*97.2*	*96.5*	*97.6*	*101.2*	*107.3*	*92.2*	*92.8*	*87.1*	*89.7*

* Drift of post-run calibration replicates compared to pre-run calibration replicates.

Table S9a. GC-FID repeatability data for Class 2A solvents (Run 1) at concentration level 1.0. GC-FID response is peak area.

Replicate number and Statistical Parameters	Acetonitrile	Chlorobenzene	Cumene	Cyclohexane	1,2-Dichloroethene	1,4-Dioxane	Methanol	Methylcyclohexane	Methylene chloride	Tetrahydrofuran	Toluene	Xylene
Repl. 1	106406	6156678	1634687	55983390	8316799	46013	323222	17344731	2129041	7152420	24498135	28596751
Repl. 2	127938	6169020	1619604	52234226	8207191	48293	333215	16380995	2143251	7142169	24558243	28678885
Repl. 3	119200	5986326	1689513	48747764	7847819	42513	298512	15874989	2052739	6767718	23617614	28576446
Repl. 4	116222	5806153	1435168	48176497	7989709	48412	325844	14462826	2099052	6914298	23471716	26427020
Repl. 5	118219	5557151	1340987	44909775	7385105	43127	294075	13458818	2002259	6566068	22059820	24840337
Repl. 6	119887	5818901	1423774	49251317	7935606	48607	339777	14839289	2089555	6971522	23454416	26405306
Mean	*117979*	*5915705*	*1523956*	*49883828*	*7947038*	*46161*	*319108*	*15393608*	*2085983*	*6919033*	*23609991*	*27254124*
SD	*6948*	*235439*	*141574*	*3796733*	*326180*	*2762*	*18651*	*1409296*	*51831*	*225618*	*909329*	*1610941*
%RSD	*5.9*	*4.0*	*9.3*	*7.6*	*4.1*	*6.0*	*5.8*	*9.2*	*2.5*	*3.3*	*3.9*	*16.9*

Table S9b. GC-FID repeatability data for Class 2A solvents (Run 2) at concentration level 1.0 made in triplicate before and after the Run 2 accuracy and recovery exercise. GC-FID response is peak area. Additional calculations on pre- and post-run triplicate measurements (compared to Run 1; Table S7a) illustrate the challenge of this method (loss of volatiles causing drift).

Replicate number and Statistical Parameters	Acetonitrile	Chlorobenzene	Cumene	Cyclohexane	1,2-Dichloroethene	1,4-Dioxane	Methanol	Methylcyclohexane	Methylene chloride	Tetrahydrofuran	Toluene	Xylene
Repl. 1 (Pre)	138880	30244247	1148420	30104446	9160888	43691	302030	9235683	2534491	8111590	24051895	46697190
Repl. 2 (Pre)	129593	27591122	1099128	29001868	8850326	44850	311127	8693071	2393998	7787489	23156224	45077593
Repl. 3 (Pre)	120948	24329202	1035220	28344618	8504294	42798	304108	8620192	2212566	7278461	21422395	42341034
Repl. 4 (Post)	119935	26708821	992404	23364145	7818686	41118	263703	6839052	2161526	6970211	21157863	42074166
Repl. 5 (Post)	125407	26162893	989238	24455694	8010406	44918	324296	7042886	2150601	7088581	21293446	42058758
Repl. 6 (Post)	120094	25392587	964376	24321590	7849333	41143	301152	7041625	2102629	6946797	20874285	41058470
Mean (All)	*125810*	*26738145*	*1038131*	*26598727*	*8365656*	*43086*	*301069*	*7912085*	*2259302*	*7363855*	*21992685*	*43217869*
SD (All)	*7431*	*2047712*	*71853*	*2875891*	*561870*	*1707*	*20234*	*1051463*	*168510*	*479695*	*1292755*	*2174750*
%RSD (All)	*5.9*	*7.7*	*6.9*	*10.8*	*6.7*	*4.0*	*6.7*	*13.3*	*7.5*	*6.5*	*5.9*	*5.0*
Mean (Pre)	*129807*	*27388190*	*1094256*	*29150311*	*8838503*	*43780*	*305755*	*8849649*	*2380352*	*7725847*	*22876838*	*44705272*
SD (Pre)	*8968*	*2962739*	*56757*	*889255*	*328457*	*1029*	*4767*	*336296*	*161396*	*419971*	*1336828*	*2201815*
%RSD (Pre)	*6.9*	*10.8*	*5.2*	*3.1*	*3.7*	*2.4*	*1.6*	*3.8*	*6.8*	*5.4*	*5.8*	*4.9*
Mean (Post)	*121812*	*26088100*	*982006*	*24047143*	*7892808*	*42393*	*296384*	*6974521*	*2138252*	*7001863*	*21108531*	*41730465*
SD (Post)	*3114*	*661297*	*15350*	*595282*	*102989*	*2187*	*30577*	*117321*	*31330*	*76007*	*213891*	*582015*
%RSD (Post)	*2.6*	*2.5*	*1.6*	*2.5*	*1.3*	*5.2*	*10.3*	*1.7*	*1.5*	*1.1*	*1.0*	*1.4*
*%Drift**	*93.8*	*95.3*	*89.7*	*82.5*	*89.3*	*96.8*	*96.9*	***78.8***	*89.8*	*90.6*	*92.3*	*93.3*

* Drift of post-run calibration replicates compared to pre-run calibration replicates.

Table S10a. GC-FID repeatability data for Class 2B solvents (Run 1) at concentration level 1.0. GC-FID response is peak area.

Replicate number and Statistical Parameters	Chloroform	1,2-Dimethoxyethane	Hexane	Methylbutylketone	Nitromethane	Pyridine	Tetralin	Trichloroethylene
Repl. 1	1662928	24198	2737812	193261	18348	51621	2619107	554045
Repl. 2	1712191	24623	2669485	199435	17256	54494	2629491	588126
Repl. 3	1630508	24141	2514837	186633	17173	45462	2562518	541875
Repl. 4	1687218	22084	2506564	196848	17140	52577	2579421	551741
Repl. 5	1629062	20334	2322488	182077	16085	44770	2454000	514223
Repl. 6	1687874	22589	2520676	192998	17905	52529	2597046	542457
Mean	*1668297*	*22995*	*2545310*	*191875*	*17318*	*50242*	*2573597*	*548745*
SD	*33658*	*1642*	*145026*	*6458*	*772*	*4085*	*63569*	*23943*
%RSD	*2.0*	*7.1*	*5.7*	*3.4*	*4.5*	*8.1*	*2.5*	*4.4*

Table S10b. GC-FID repeatability data for Class 2B solvents (Run 2) at concentration level 1.0 made in triplicate before and after the Run 2 accuracy and recovery exercise. GC-FID response is peak area. Additional calculations on pre- and post-run triplicate measurements (compared to Run 1; Table S8a) illustrate the challenge of this method (loss of volatiles causing drift)

Replicate number and Statistical Parameters	Chloroform	1,2-Dimethoxyethane	Hexane	Methylbutylketone	Nitromethane	Pyridine	Tetralin	Trichloroethylene
Repl. 1 (Pre)	1867255	1264	5952698	239907	28032	24051895	3111103	671269
Repl. 2 (Pre)	1716544	1161	5746273	212631	26609	23156224	2884574	627923
Repl. 3 (Pre)	1517960	1168	5661010	190267	28150	21422395	2663100	619782
Repl. 4 (Post)	1581233	569	4857342	200953	27356	21157863	2674391	573751
Repl. 5 (Post)	1557374	1112	5139689	199307	29616	21293446	2635720	578502
Repl. 6 (Post)	1525857	1128	5143961	191724	27836	20874285	2542366	562201
Mean (All)	*1627704*	*1067*	*5416829*	*205798*	*27933*	*21992685*	*2751876*	*605571*
SD (All)	*137695*	*250*	*428860*	*18520*	*998*	*1292755*	*208770*	*41579*
%RSD (All)	*8.5*	*23.4*	*7.9*	*9.0*	*3.6*	*5.9*	*7.6*	*6.9*
Mean (Pre)	*1700586*	*1198*	*5786660*	*214268*	*27597*	*22876838*	*2886259*	*639658*
SD (Pre)	*175193*	*58*	*149979*	*24860*	*858*	*1336828*	*224006*	*27677*
%RSD (Pre)	*10.3*	*4.8*	*2.6*	*11.6*	*3.1*	*5.8*	*7.8*	*4.3*
Mean (Post)	*1554821*	*936*	*5046997*	*197328*	*28269*	*21108531*	*2617492*	*571485*
SD (Post)	*27776*	*318*	*164260*	*4922*	*1191*	*213891*	*67874*	*8383*
%RSD (Post)	*1.8*	*34.0*	*3.3*	*2.5*	*4.2*	*1.0*	*2.6*	*1.5*
%Drift*	*91.4*	*78.2*	*87.2*	*92.1*	*102.4*	*92.3*	*90.7*	*89.3*

* Drift of post-run calibration replicates compared to pre-run calibration replicates.

D.1. Tablet

Table S11a. SIFT-MS accuracy data for Class 2A solvents (Run 1) in the tableted product. Values in red do not meet acceptance criteria ($\pm$20% of spiked value).

Spike Level / %	Replicate Number and Statistical Parameters	Acetonitrile (H$_3$O$^+$ 42)	Chlorobenzene (O$_2^{+\bullet}$ 112)	Chlorobenzene (O$_2^{+\bullet}$ 114)	Cumene (NO$^+$ 120)	Cyclohexane (NO$^+$ 83)	Cyclohexane (O$_2^{+\bullet}$ 84)	1,2-Dichloroethene (H$_3$O$^+$ 99)	1,2-Dichloroethene (O$_2^{+\bullet}$ 96)	1,2-Dichloroethene (O$_2^{+\bullet}$ 98)	1,4-Dioxane (H$_3$O$^+$ 89)	1,4-Dioxane (NO$^+$ 87)	1,4-Dioxane (NO$^+$ 88)	Methanol (H$_3$O$^+$ 33)	Methylcyclohexane (NO$^+$ 97)	Methylene chloride (O$_2^{+\bullet}$ 84)	Methylene chloride (O$_2^{+\bullet}$ 86)	Tetrahydrofuran (NO$^+$ 71)	Toluene (NO$^+$ 92)	Toluene (O$_2^{+\bullet}$ 92)	Xylene (NO$^+$ 106)
50	Repl. 1	0.438	0.405	0.401	0.370	0.348	0.371	0.435	0.441	0.396	0.449	0.393	0.381	0.449	0.297	0.357	0.412	0.415	0.450	0.407	0.403
	Repl. 2	0.446	0.408	0.410	0.382	0.343	0.363	0.384	0.445	0.406	0.461	0.449	0.403	0.461	0.297	0.348	0.398	0.442	0.468	0.411	0.410
	Repl. 3	0.442	0.397	0.401	0.364	0.330	0.350	0.402	0.427	0.389	0.400	0.405	0.383	0.437	0.280	0.337	0.396	0.426	0.445	0.385	0.399
	Mean	*0.442*	*0.403*	*0.404*	*0.372*	*0.340*	*0.362*	*0.407*	*0.438*	*0.397*	*0.437*	*0.416*	*0.389*	*0.449*	*0.291*	*0.348*	*0.402*	*0.428*	*0.454*	*0.401*	*0.404*
	SD	*0.003*	*0.005*	*0.004*	*0.008*	*0.008*	*0.009*	*0.021*	*0.008*	*0.007*	*0.026*	*0.024*	*0.010*	*0.010*	*0.008*	*0.008*	*0.007*	*0.011*	*0.009*	*0.011*	*0.004*
	%RSD	*0.7*	*1.2*	*1.0*	*2.0*	*2.2*	*2.4*	*5.2*	*1.8*	*1.7*	*6.1*	*5.8*	*2.5*	*2.2*	*2.7*	*2.4*	*1.7*	*2.6*	*2.1*	*2.8*	*1.1*
100	Repl. 1	0.908	0.941	0.929	1.20	0.848	0.812	0.940	0.958	0.910	0.922	0.865	0.820	0.882	0.765	0.806	0.883	0.882	0.969	0.928	1.058
	Repl. 2	0.912	0.907	0.891	0.856	0.784	0.783	0.908	0.948	0.872	0.894	0.906	0.855	0.924	0.688	0.770	0.844	0.890	0.938	0.911	0.896
	Repl. 3	0.912	0.899	0.911	0.808	0.773	0.763	0.859	0.948	0.855	0.928	0.815	0.877	0.879	0.664	0.745	0.852	0.897	0.944	0.911	0.879
	Mean	*0.911*	*0.916*	*0.910*	*0.956*	*0.802*	*0.786*	*0.902*	*0.951*	*0.879*	*0.915*	*0.862*	*0.851*	*0.895*	*0.706*	*0.774*	*0.860*	*0.890*	*0.950*	*0.917*	*0.945*
	SD	*0.002*	*0.018*	*0.015*	*0.175*	*0.033*	*0.020*	*0.034*	*0.005*	*0.023*	*0.015*	*0.037*	*0.024*	*0.021*	*0.043*	*0.025*	*0.017*	*0.006*	*0.014*	*0.008*	*0.081*
	%RSD	*0.2*	*2.0*	*1.7*	*18.4*	*4.14*	*2.6*	*3.7*	*0.5*	*2.6*	*1.6*	*4.3*	*2.8*	*2.3*	*6.1*	*3.3*	*2.0*	*0.7*	*1.4*	*0.9*	*8.6*

Table S11b. SIFT-MS accuracy data for Class 2A solvents (Run 2) in the tableted product. Values in gray are probably outliers but are included in calculations.

Spike Level / %	Replicate Number and Statistical Parameters	Acetonitrile (H₃O⁺ 42)	Chlorobenzene (O₂⁺• 112)	Chlorobenzene (O₂⁺• 114)	Cumene (NO⁺ 120)	Cyclohexane (NO⁺ 83)	Cyclohexane (O₂⁺• 84)	1,2-Dichloroethene (H₃O⁺ 99)	1,2-Dichloroethene (O₂⁺• 96)	1,2-Dichloroethene (O₂⁺• 98)	1,4-Dioxane (H₃O⁺ 89)	1,4-Dioxane (NO⁺ 87)	1,4-Dioxane (NO⁺ 88)	Methanol (H₃O⁺ 33)	Methylcyclohexane (NO⁺ 97)	Methylene chloride (O₂⁺• 84)	Methylene chloride (O₂⁺• 86)	Tetrahydrofuran (NO⁺ 71)	Toluene (NO⁺ 92)	Toluene (O₂⁺• 92)	Xylene (NO⁺ 106)
50	Repl. 1	0.525	0.463	0.471	0.437	0.476	0.500	0.526	0.491	0.481	0.492	0.428	0.480	0.476	0.430	0.482	0.476	0.467	0.504	0.463	0.456
	Repl. 2	0.427	0.380	0.388	0.375	0.348	0.416	0.340	0.408	0.404	0.412	0.484	0.456	0.434	0.384	0.397	0.389	0.408	0.413	0.376	0.380
	Repl. 3	0.582	0.499	0.507	0.462	0.476	0.505	0.537	0.519	0.506	0.531	0.546	0.534	0.538	0.443	0.490	0.516	0.527	0.530	0.488	0.481
	Mean	*0.511*	*0.448*	*0.455*	*0.425*	*0.434*	*0.474*	*0.468*	*0.473*	*0.464*	*0.478*	*0.486*	*0.490*	*0.483*	*0.419*	*0.456*	*0.460*	*0.467*	*0.482*	*0.443*	*0.439*
	SD	*0.064*	*0.050*	*0.050*	*0.037*	*0.061*	*0.041*	*0.091*	*0.047*	*0.043*	*0.050*	*0.048*	*0.033*	*0.043*	*0.025*	*0.042*	*0.053*	*0.049*	*0.050*	*0.048*	*0.043*
	%RSD	*12.5*	*11.1*	*10.9*	*8.6*	*14.0*	*8.7*	*19.3*	*10.0*	*9.3*	*10.4*	*9.9*	*6.7*	*8.9*	*6.0*	*9.2*	*11.5*	*10.4*	*10.4*	*10.9*	*9.8*
100	Repl. 1	0.868	0.957	0.953	1.059	1.126	1.095	0.947	0.979	1.005	0.778	0.869	0.874	0.831	1.150	1.091	0.880	0.815	1.001	0.987	0.995
	Repl. 2	0.913	1.009	1.020	1.059	1.134	1.119	1.076	1.034	1.060	0.919	0.966	0.849	0.909	1.141	1.116	0.945	0.874	1.039	1.042	1.033
	Repl. 3	0.894	0.984	0.998	1.008	1.118	1.095	1.074	1.011	1.023	0.911	0.772	0.849	0.854	1.067	1.091	0.939	0.844	1.027	1.005	0.995
	Mean	*0.892*	*0.983*	*0.990*	*1.042*	*1.126*	*1.103*	*1.032*	*1.008*	*1.030*	*0.869*	*0.869*	*0.857*	*0.865*	*1.120*	*1.100*	*0.921*	*0.844*	*1.022*	*1.011*	*1.008*
	SD	*0.019*	*0.021*	*0.028*	*0.024*	*0.007*	*0.011*	*0.060*	*0.023*	*0.023*	*0.065*	*0.079*	*0.012*	*0.033*	*0.037*	*0.012*	*0.029*	*0.024*	*0.016*	*0.023*	*0.018*
	%RSD	*2.1*	*2.2*	*2.8*	*2.3*	*0.60*	*1.0*	*5.8*	*2.3*	*2.2*	*7.4*	*9.1*	*1.4*	*3.8*	*3.3*	*1.1*	*3.2*	*2.8*	*1.6*	*2.3*	*1.8*

Table S12a. SIFT-MS accuracy data for Class 2B solvents (Run 1) in the tableted product. Values in red do not meet acceptance criteria ($\pm$20% of spiked value).

Spike Level / %	Replicate Number and Statistical Parameters	Chloroform ($O_2^{+\bullet}$ 83)	Chloroform ($O_2^{+\bullet}$ 85)	1,2-Dimethoxyethane (H_3O^+ 91)	1,2-Dimethoxyethane (NO^+ 89)	Hexane (NO^+ 85)	Methylbutylketone (NO^+ 130)	Nitromethane (H_3O^+ 62)	Pyridine (H_3O^+ 80)	Tetralin (NO^+ 132)	Tetralin ($O_2^{+\bullet}$ 104)	Trichloroethylene ($O_2^{+\bullet}$ 130)	Trichloroethylene ($O_2^{+\bullet}$ 132)
50	Repl. 1	0.312	0.389	0.378	0.489	0.409	0.453	0.557	0.339	0.380	0.390	0.420	0.347
	Repl. 2	0.304	0.379	0.407	0.432	0.405	0.453	0.519	0.406	0.389	0.405	0.407	0.350
	Repl. 3	0.291	0.375	0.391	0.403	0.433	0.443	0.465	0.423	0.384	0.379	0.399	0.340
	Mean	*0.302*	*0.381*	*0.392*	*0.441*	*0.416*	*0.449*	*0.514*	*0.389*	*0.384*	*0.391*	*0.408*	*0.346*
	SD	*0.008*	*0.006*	*0.012*	*0.036*	*0.012*	*0.004*	*0.038*	*0.036*	*0.004*	*0.011*	*0.009*	*0.004*
	%RSD	*2.8*	*1.6*	*3.1*	*8.2*	*3.0*	*1.0*	*7.3*	*9.3*	*1.0*	*2.7*	*2.1*	*1.2*
100	Repl. 1	0.787	0.853	0.900	0.807	0.934	0.862	0.996	0.838	0.850	0.832	0.942	0.859
	Repl. 2	0.729	0.808	0.881	0.844	0.851	0.855	0.955	0.954	0.806	0.809	0.907	0.819
	Repl. 3	0.693	0.820	0.859	0.838	0.850	0.862	0.908	0.941	0.833	0.833	0.926	0.833
	Mean	*0.736*	*0.827*	*0.880*	*0.830*	*0.879*	*0.860*	*0.953*	*0.911*	*0.830*	*0.825*	*0.925*	*0.837*
	SD	*0.039*	*0.019*	*0.017*	*0.016*	*0.039*	*0.003*	*0.036*	*0.052*	*0.018*	*0.011*	*0.014*	*0.016*
	%RSD	*5.3*	*2.3*	*1.9*	*2.0*	*4.5*	*0.4*	*3.80*	*5.7*	*2.2*	*1.3*	*1.56*	*2.0*

Table S12b. SIFT-MS accuracy data for Class 2B solvents (Run 2) in the tableted product. Values in gray are probably outliers but are included in calculations.

Spike Level / %	Replicate Number and Statistical Parameters	Chloroform $(O_2^{+\bullet}\ 83)$	Chloroform $(O_2^{+\bullet}\ 85)$	1,2-Dimethoxyethane $(H_3O^+\ 91)$	1,2-Dimethoxyethane $(NO^+\ 89)$	Hexane $(NO^+\ 85)$	Methylbutylketone $(NO^+\ 130)$	Nitromethane $(H_3O^+\ 62)$	Pyridine $(H_3O^+\ 80)$	Tetralin $(NO^+\ 132)$	Tetralin $(O_2^{+\bullet}\ 104)$	Trichloroethylene $(O_2^{+\bullet}\ 130)$	Trichloroethylene $(O_2^{+\bullet}\ 132)$
50	Repl. 1	0.437	0.466	0.436	0.485	0.478	0.493	0.505	0.391	0.460	0.471	0.457	0.430
	Repl. 2	0.337	0.374	0.364	0.375	0.370	0.411	0.388	0.367	0.368	0.370	0.357	0.328
	Repl. 3	0.447	0.514	0.495	0.538	0.500	0.552	0.580	0.461	0.506	0.494	0.498	0.455
	Mean	*0.407*	*0.451*	*0.432*	*0.466*	*0.449*	*0.485*	*0.491*	*0.406*	*0.445*	*0.445*	*0.438*	*0.404*
	SD	*0.049*	*0.058*	*0.054*	*0.068*	*0.057*	*0.058*	*0.079*	*0.040*	*0.057*	*0.054*	*0.059*	*0.055*
	%RSD	*12.1*	*12.8*	*12.4*	*14.5*	*12.7*	*12.0*	*16.1*	*9.8*	*12.9*	*12.1*	*13.5*	*13.6*
100	Repl. 1	1.087	0.868	0.932	0.830	0.868	0.835	0.868	0.817	0.944	0.922	1.075	0.972
	Repl. 2	1.109	0.931	0.996	0.901	0.952	0.906	0.881	0.972	0.996	0.980	1.066	1.024
	Repl. 3	1.084	0.908	0.937	0.898	0.963	0.871	0.881	0.943	0.963	0.964	1.066	1.015
	Mean	*1.093*	*0.903*	*0.955*	*0.877*	*0.928*	*0.871*	*0.876*	*0.911*	*0.968*	*0.955*	*1.069*	*1.004*
	SD	*0.011*	*0.026*	*0.029*	*0.033*	*0.042*	*0.029*	*0.006*	*0.067*	*0.022*	*0.025*	*0.004*	*0.023*
	%RSD	*1.0*	*2.9*	*3.0*	*3.7*	*4.5*	*3.3*	*0.70*	*7.4*	*2.2*	*2.6*	*0.38*	*2.3*

Table S13a. GC-FID accuracy data for Class 2A solvents (Run 1) in the tableted product. Values in red do not meet accuracy (±20% of spiked value) or repeatability (<20%RSD) acceptance criteria.

Spike Level / %	Replicate Number and Statistical Parameters	Acetonitrile	Chlorobenzene	Cumene	Cyclohexane	1,2-Dichloroethene	1,4-Dioxane	Methanol	Methylcyclohexane	Methylene chloride	Tetrahydrofuran	Toluene	Xylene
50	Repl. 1	0.435	0.436	0.382	0.350	0.441	0.426	0.410	0.284	0.442	0.439	0.441	0.430
	Repl. 2	0.336	0.437	0.386	0.347	0.430	0.486	0.458	0.294	0.440	0.443	0.442	0.431
	Repl. 3	0.532	0.416	0.325	0.238	0.359	0.503	0.532	0.186	0.426	0.404	0.395	0.391
	Mean	*0.434*	*0.430*	*0.364*	*0.311*	*0.410*	*0.472*	*0.467*	*0.254*	*0.436*	*0.429*	*0.426*	*0.417*
	SD	*0.098*	*0.012*	*0.034*	*0.064*	*0.045*	*0.041*	*0.062*	*0.060*	*0.009*	*0.022*	*0.027*	*0.023*
	%RSD	*22.5*	*2.8*	*9.4*	*20.5*	*10.9*	*8.6*	*13.2*	*23.5*	*2.0*	*5.1*	*6.3*	*5.5*
100	Repl. 1	1.24	0.938	0.830	0.826	0.948	0.961	0.976	0.725	0.973	0.968	0.948	0.915
	Repl. 2	1.17	0.905	0.747	0.726	0.892	0.901	0.911	0.592	0.903	0.920	0.905	0.864
	Repl. 3	1.06	0.939	0.878	0.863	0.933	0.968	0.966	0.806	0.958	0.949	0.945	0.924
	Mean	*1.16*	*0.927*	*0.818*	*0.805*	*0.925*	*0.943*	*0.951*	*0.707*	*0.944*	*0.946*	*0.932*	*0.901*
	SD	*0.091*	*0.020*	*0.066*	*0.071*	*0.029*	*0.037*	*0.035*	*0.108*	*0.037*	*0.024*	*0.024*	*0.033*
	%RSD	*7.8*	*2.1*	*8.1*	*8.8*	*3.1*	*3.9*	*3.6*	*15.3*	*3.9*	*2.5*	*2.6*	*3.6*

Table S13b. GC-FID accuracy data for Class 2A solvents (Run 2) in the tableted product. Values in red do not meet acceptance criteria (±20% of spiked value), while values in gray are outliers and <u>not</u> included in calculations.

Spike Level / %	Replicate Number and Statistical Parameters	Acetonitrile	Chlorobenzene	Cumene	Cyclohexane	1,2-Dichloroethene	1,4-Dioxane	Methanol	Methylcyclohexane	Methylene chloride	Tetrahydrofuran	Toluene	Xylene
50	Repl. 1	0.485	0.476	0.389	0.400	0.493	0.438	0.457	0.317	0.495	0.489	0.483	0.452
	Repl. 2	0.542	0.493	0.471	0.420	0.469	0.463	0.450	0.402	0.505	0.490	0.485	0.485
	Repl. 3	0.529	0.479	0.474	0.454	0.471	0.496	0.550	0.443	0.487	0.477	0.481	0.485
	Mean	*0.519*	*0.483*	*0.445*	*0.424*	*0.478*	*0.466*	*0.486*	*0.387*	*0.496*	*0.485*	*0.483*	*0.474*
	SD	*0.024*	*0.009*	*0.040*	*0.022*	*0.011*	*0.024*	*0.046*	*0.053*	*0.007*	*0.006*	*0.002*	*0.019*
	%RSD	*4.7*	*1.9*	*8.9*	*5.3*	*2.2*	*5.1*	*9.5*	*13.6*	*1.5*	*1.2*	*0.32*	*4.0*
100	Repl. 1	0.785	0.947	0.945	1.004	1.017	0.735	0.740	0.963	0.932	0.969	0.989	0.962
	Repl. 2	0.813	0.978	0.986	1.046	1.035	0.704	0.749	1.016	0.955	0.981	1.013	0.996
	Repl. 3	0.478	0.631	0.704	0.679	0.618	0.399	1.958	0.678	0.429	0.704	0.647	0.658
	Mean	*0.799*	*0.963*	*0.966*	*1.025*	*1.026*	*0.719*	*0.744*	*0.989*	*0.944*	*0.975*	*1.001*	*0.979*
	SD	*0.014*	*0.015*	*0.020*	*0.021*	*0.009*	*0.015*	*0.005*	*0.027*	*0.012*	*0.006*	*0.012*	*0.017*
	%RSD	*1.7*	*1.6*	*2.1*	*2.1*	*0.88*	*2.1*	*0.61*	*2.7*	*1.2*	*0.64*	*1.2*	*1.7*

Table S14a. GC-FID accuracy data for Class 2B solvents (Run 1) in the tableted product. Values in red do not meet accuracy (±20% of spiked value) or repeatability (<20%RSD) acceptance criteria.

Spike Level / %	Replicate Number and Statistical Parameters	Chloroform	1,2-Dimethoxyethane	Hexane	Methylbutylketone	Nitromethane	Pyridine	Tetralin	Trichloroethylene
50	Repl. 1	0.452	0.298	0.421	0.427	0.421	0.370	0.455	0.470
	Repl. 2	0.462	0.240	0.397	0.435	0.358	0.421	0.454	0.442
	Repl. 3	0.507	0.113	0.297	0.470	0.252	0.476	0.450	0.393
	Mean	*0.474*	*0.217*	*0.372*	*0.444*	*0.344*	*0.422*	*0.453*	*0.435*
	SD	*0.029*	*0.095*	*0.066*	*0.023*	*0.085*	*0.053*	*0.003*	*0.039*
	%RSD	*6.2*	*43.8*	*17.7*	*5.2*	*24.8*	*12.5*	*0.6*	*9.0*
100	Repl. 1	1.06	0.348	0.958	0.960	0.896	0.954	1.01	1.10
	Repl. 2	1.00	0.247	0.844	0.936	0.769	0.857	0.947	1.00
	Repl. 3	1.06	0.264	0.888	0.955	0.830	0.948	0.971	1.03
	Mean	*1.04*	*0.286*	*0.897*	*0.950*	*0.832*	*0.920*	*0.976*	*1.05*
	SD	*0.035*	*0.054*	*0.058*	*0.012*	*0.064*	*0.054*	*0.032*	*0.052*
	%RSD	*3.3*	*18.9*	*6.4*	*1.3*	*7.6*	*5.9*	*3.3*	*5.0*

Table S14b. GC-FID accuracy data for Class 2B solvents (Run 2) in the tableted product. Values in red do not meet acceptance criteria (±20% of spiked value), while values in gray are outliers and are <u>not</u> included in calculations.

Spike Level / %	Replicate Number and Statistical Parameters	Chloroform	1,2-Dimethoxyethane	Hexane	Methylbutylketone	Nitromethane	Pyridine	Tetralin	Trichloroethylene
50	Repl. 1	0.479	0.782	0.483	0.475	0.486	0.596	0.493	0.454
	Repl. 2	0.541	0.684	0.432	0.530	0.448	0.666	0.519	0.459
	Repl. 3	0.500	0.704	0.447	0.469	0.407	0.610	0.479	0.466
	Mean	*0.507*	*0.723*	*0.454*	*0.491*	*0.447*	*0.624*	*0.497*	*0.460*
	SD	*0.026*	*0.042*	*0.021*	*0.027*	*0.032*	*0.030*	*0.016*	*0.005*
	%RSD	*5.1*	*5.8*	*4.6*	*5.5*	*7.2*	*4.8*	*3.3*	*1.1*
100	Repl. 1	0.795	0.798	1.082	0.828	1.011	1.026	0.924	1.017
	Repl. 2	0.843	0.873	1.076	0.859	1.049	1.065	0.940	1.018
	Repl. 3	0.519	0.797	0.706	0.526	0.697	0.679	0.612	0.659
	Mean	*0.819*	*0.836*	*1.079*	*0.844*	*1.030*	*1.046*	*0.932*	*1.017*
	SD	*0.024*	*0.038*	*0.003*	*0.016*	*0.019*	*0.020*	*0.008*	*0.0004*
	%RSD	*2.9*	*4.5*	*0.29*	*1.9*	*1.8*	*1.9*	*0.83*	*0.04*

Table S15a. SIFT-MS accuracy data for Class 2A solvents (Run 1) in the oral suspension product. Values in red do not meet acceptance criteria (±20% of spiked value), while values in gray are probably outliers but are included in calculations.

Spike Level / %	Replicate Number and Statistical Parameters	Acetonitrile (H$_3$O$^+$ 42)	Chlorobenzene (O$_2^{+\bullet}$ 112)	Chlorobenzene (O$_2^{+\bullet}$ 114)	Cumene (NO$^+$ 120)	Cyclohexane (NO$^+$ 83)	Cyclohexane (O$_2^{+\bullet}$ 84)	1,2-Dichloroethene (H$_3$O$^+$ 99)	1,2-Dichloroethene (O$_2^{+\bullet}$ 96)	1,2-Dichloroethene (O$_2^{+\bullet}$ 98)	1,4-Dioxane (H$_3$O$^+$ 89)	1,4-Dioxane (NO$^+$ 87)	1,4-Dioxane (NO$^+$ 88)	Methanol (H$_3$O$^+$ 33)	Methylcyclohexane (NO$^+$ 97)	Methylene chloride (O$_2^{+\bullet}$ 84)	Methylene chloride (O$_2^{+\bullet}$ 86)	Tetrahydrofuran (NO$^+$ 71)	Toluene (NO$^+$ 92)	Toluene (O$_2^{+\bullet}$ 92)	Xylene (NO$^+$ 106)
50	Repl. 1	0.466	0.374	0.375	0.355	0.332	0.345	0.366	0.416	0.377	6.846	0.552	0.751	0.457	0.288	0.330	0.439	0.649	0.434	0.377	0.382
	Repl. 2	0.563	0.444	0.436	0.386	0.354	0.363	0.419	0.463	0.418	9.258	0.710	1.068	0.571	0.297	0.348	0.505	0.806	0.482	0.428	0.425
	Repl. 3	0.368	0.327	0.325	0.288	0.283	0.307	0.339	0.369	0.339	6.115	0.500	0.674	0.375	0.237	0.291	0.379	0.545	0.362	0.324	0.321
	Mean	*0.466*	*0.382*	*0.379*	*0.343*	*0.323*	*0.338*	*0.375*	*0.416*	*0.378*	*7.406*	*0.587*	*0.831*	*0.468*	*0.274*	*0.323*	*0.441*	*0.667*	*0.426*	*0.376*	*0.376*
	SD	*0.079*	*0.048*	*0.045*	*0.041*	*0.030*	*0.024*	*0.034*	*0.038*	*0.032*	*1.343*	*0.090*	*0.171*	*0.080*	*0.026*	*0.024*	*0.051*	*0.107*	*0.049*	*0.042*	*0.043*
	%RSD	*17.0*	*12.5*	*12.0*	*11.9*	*9.2*	*7.0*	*9.0*	*9.2*	*8.6*	*18.1*	*15.2*	*20.6*	*17.2*	*9.6*	*7.3*	*11.6*	*16.1*	*11.5*	*11.2*	*11.4*
100	Repl. 1	1.056	0.916	0.908	0.819	0.784	0.781	0.842	0.976	0.906	9.020	1.208	1.434	1.070	0.675	0.768	0.939	1.255	0.950	0.923	0.905
	Repl. 2	1.021	0.899	0.908	0.824	0.791	0.778	0.850	0.969	0.889	8.425	1.249	1.349	1.048	0.689	0.765	0.930	1.217	0.976	0.934	0.889
	Repl. 3	0.986	0.882	0.864	0.824	0.791	0.786	0.883	0.951	0.877	8.544	1.169	1.434	1.018	0.712	0.774	0.914	1.164	0.938	0.923	0.883
	Mean	*1.021*	*0.899*	*0.894*	*0.823*	*0.788*	*0.782*	*0.859*	*0.966*	*0.890*	*8.663*	*1.208*	*1.406*	*1.045*	*0.692*	*0.769*	*0.928*	*1.212*	*0.955*	*0.926*	*0.892*
	SD	*0.029*	*0.014*	*0.021*	*0.003*	*0.003*	*0.003*	*0.018*	*0.011*	*0.012*	*0.257*	*0.033*	*0.040*	*0.021*	*0.015*	*0.004*	*0.010*	*0.037*	*0.016*	*0.005*	*0.009*
	%RSD	*2.8*	*1.5*	*2.3*	*0.3*	*0.40*	*0.4*	*2.1*	*1.1*	*1.3*	*3.0*	*2.7*	*2.9*	*2.0*	*2.2*	*0.5*	*1.1*	*3.1*	*1.7*	*0.6*	*1.0*

Table S15b. SIFT-MS accuracy data for Class 2A solvents (Run 2) in the oral suspension product. Values in red do not meet acceptance criteria ($\pm$20% of spiked value).

Spike Level / %	Replicate Number and Statistical Parameters	Acetonitrile (H$_3$O$^+$ 42)	Chlorobenzene (O$_2$$^{+\bullet}$ 112)	Chlorobenzene (O$_2$$^{+\bullet}$ 114)	Cumene (NO$^+$ 120)	Cyclohexane (NO$^+$ 83)	Cyclohexane (O$_2$$^{+\bullet}$ 84)	1,2-Dichloroethene (H$_3$O$^+$ 99)	1,2-Dichloroethene (O$_2$$^{+\bullet}$ 96)	1,2-Dichloroethene (O$_2$$^{+\bullet}$ 98)	1,4-Dioxane (H$_3$O$^+$ 89)	1,4-Dioxane (NO$^+$ 87)	1,4-Dioxane (NO$^+$ 88)	Methanol (H$_3$O$^+$ 33)	Methylcyclohexane (NO$^+$ 97)	Methylene chloride (O$_2$$^{+\bullet}$ 84)	Methylene chloride (O$_2$$^{+\bullet}$ 86)	Tetrahydrofuran (NO$^+$ 71)	Toluene (NO$^+$ 92)	Toluene (O$_2$$^{+\bullet}$ 92)	Xylene (NO$^+$ 106)
50	Repl. 1	0.536	0.478	0.487	0.474	0.485	0.505	0.567	0.499	0.493	1.049	0.538	0.497	0.501	0.463	0.490	0.501	0.515	0.497	0.465	0.474
	Repl. 2	0.536	0.460	0.468	0.450	0.472	0.491	0.547	0.495	0.484	0.972	0.475	0.528	0.505	0.438	0.478	0.485	0.487	0.499	0.464	0.450
	Repl. 3	0.533	0.454	0.474	0.438	0.485	0.477	0.578	0.479	0.475	0.985	0.510	0.523	0.481	0.424	0.466	0.479	0.482	0.483	0.449	0.443
	Mean	*0.535*	*0.464*	*0.477*	*0.454*	*0.481*	*0.491*	*0.564*	*0.491*	*0.484*	*1.002*	*0.508*	*0.516*	*0.496*	*0.442*	*0.478*	*0.489*	*0.495*	*0.493*	*0.459*	*0.456*
	SD	*0.002*	*0.010*	*0.008*	*0.015*	*0.006*	*0.012*	*0.013*	*0.009*	*0.008*	*0.034*	*0.026*	*0.014*	*0.010*	*0.016*	*0.010*	*0.009*	*0.014*	*0.007*	*0.008*	*0.013*
	%RSD	*0.33*	*2.2*	*1.7*	*3.2*	*1.2*	*2.4*	*2.2*	*1.7*	*1.6*	*3.4*	*5.1*	*2.6*	*2.1*	*3.7*	*2.1*	*1.9*	*2.9*	*1.4*	*1.7*	*2.9*
100	Repl. 1	1.015	1.067	1.069	1.078	1.101	1.109	1.116	1.070	1.088	1.471	0.987	0.952	0.964	1.107	1.112	1.041	1.005	1.065	1.086	1.062
	Repl. 2	0.883	0.984	0.975	1.034	1.126	1.114	1.149	1.003	1.023	1.261	0.867	0.833	0.811	1.122	1.112	0.918	0.852	1.014	1.011	1.002
	Repl. 3	0.932	0.999	1.020	1.027	1.138	1.095	1.138	1.022	1.036	1.351	0.973	0.890	0.872	1.110	1.091	0.971	0.910	1.020	1.024	1.024
	Mean	*0.943*	*1.017*	*1.021*	*1.046*	*1.122*	*1.106*	*1.134*	*1.032*	*1.049*	*1.361*	*0.943*	*0.892*	*0.883*	*1.113*	*1.105*	*0.977*	*0.922*	*1.033*	*1.040*	*1.030*
	SD	*0.054*	*0.036*	*0.038*	*0.022*	*0.015*	*0.008*	*0.014*	*0.028*	*0.028*	*0.086*	*0.053*	*0.049*	*0.063*	*0.006*	*0.010*	*0.051*	*0.063*	*0.023*	*0.033*	*0.025*
	%RSD	*5.8*	*3.5*	*3.7*	*2.1*	*1.4*	*0.73*	*1.2*	*2.7*	*2.7*	*6.3*	*5.7*	*5.4*	*7.1*	*0.55*	*0.87*	*5.2*	*6.8*	*2.2*	*3.1*	*2.4*

* %Acc. is the accuracy (as percentage) with respect to the spiked concentration.

Table S16a. SIFT-MS accuracy data for Class 2B solvents (Run 1) in the oral suspension product. Values in red do not meet acceptance criteria (±20% of spiked value).

Spike Level / %	Replicate Number and Statistical Parameters	Chloroform $(O_2^{+\bullet}\ 83)$	Chloroform $(O_2^{+\bullet}\ 85)$	1,2-Dimethoxyethane $(H_3O^+\ 91)$	1,2-Dimethoxyethane $(NO^+\ 89)$	Hexane $(NO^+\ 85)$	Methylbutylketone $(NO^+\ 130)$	Nitromethane $(H_3O^+\ 62)$	Pyridine $(H_3O^+\ 80)$	Tetralin $(NO^+\ 132)$	Tetralin $(O_2^{+\bullet}\ 104)$	Trichloroethylene $(O_2^{+\bullet}\ 130)$	Trichloroethylene $(O_2^{+\bullet}\ 132)$
50	Repl. 1	0.284	0.451	0.404	0.807	9.183	0.468	0.506	0.322	0.349	0.360	0.482	0.311
	Repl. 2	0.303	0.514	0.462	1.000	11.929	0.561	0.540	0.407	0.408	0.413	0.569	0.363
	Repl. 3	0.246	0.387	0.312	0.602	8.111	0.364	0.397	0.495	0.276	0.304	0.406	0.258
	Mean	*0.278*	*0.451*	*0.393*	*0.803*	*9.741*	*0.464*	*0.481*	*0.408*	*0.344*	*0.359*	*0.486*	*0.311*
	SD	*0.024*	*0.052*	*0.062*	*0.163*	*1.608*	*0.080*	*0.061*	*0.071*	*0.054*	*0.044*	*0.067*	*0.043*
	%RSD	*8.6*	*11.5*	*15.7*	*20.2*	*16.5*	*17.3*	*12.7*	*17.3*	*15.7*	*12.4*	*13.7*	*13.8*
100	Repl. 1	0.734	0.946	0.902	1.441	10.612	1.002	1.003	0.796	0.792	0.789	1.068	0.809
	Repl. 2	0.728	0.929	0.899	1.404	9.954	0.962	0.959	0.854	0.764	0.801	1.068	0.809
	Repl. 3	0.743	0.915	0.880	1.398	10.330	0.871	0.867	0.815	0.719	0.733	0.992	0.760
	Mean	*0.735*	*0.930*	*0.893*	*1.415*	*10.299*	*0.945*	*0.943*	*0.822*	*0.759*	*0.775*	*1.043*	*0.793*
	SD	*0.006*	*0.013*	*0.010*	*0.019*	*0.270*	*0.055*	*0.057*	*0.024*	*0.030*	*0.030*	*0.036*	*0.023*
	%RSD	*0.86*	*1.4*	*1.1*	*1.4*	*2.6*	*5.8*	*6.0*	*2.9*	*4.0*	*3.9*	*3.4*	*2.9*

Table S16b. SIFT-MS accuracy data for Class 2B solvents (Run 2) in the oral suspension product. Values in red do not meet acceptance criteria (±20% of spiked value).

Spike Level / %	Replicate Number and Statistical Parameters	Chloroform ($O_2^{+\bullet}$ 83)	Chloroform ($O_2^{+\bullet}$ 85)	1,2-Dimethoxyethane (H_3O^+ 91)	1,2-Dimethoxyethane (NO^+ 89)	Hexane (NO^+ 85)	Methylbutylketone (NO^+ 130)	Nitromethane (H_3O^+ 62)	Pyridine (H_3O^+ 80)	Tetralin (NO^+ 132)	Tetralin ($O_2^{+\bullet}$ 104)	Trichloroethylene ($O_2^{+\bullet}$ 130)	Trichloroethylene ($O_2^{+\bullet}$ 132)
50	Repl. 1	0.456	0.495	0.473	0.498	0.651	0.520	0.525	0.395	0.477	0.485	0.492	0.433
	Repl. 2	0.437	0.477	0.458	0.496	0.614	0.512	0.466	0.471	0.468	0.468	0.479	0.433
	Repl. 3	0.430	0.473	0.456	0.483	0.615	0.490	0.473	0.426	0.456	0.456	0.478	0.421
	Mean	*0.441*	*0.482*	*0.462*	*0.492*	*0.627*	*0.507*	*0.488*	*0.431*	*0.467*	*0.469*	*0.483*	*0.429*
	SD	*0.011*	*0.010*	*0.008*	*0.007*	*0.017*	*0.013*	*0.026*	*0.032*	*0.009*	*0.012*	*0.007*	*0.006*
	%RSD	*2.5*	*2.0*	*1.6*	*1.4*	*2.8*	*2.5*	*5.4*	*7.3*	*1.9*	*2.5*	*1.4*	*1.4*
100	Repl. 1	1.113	1.033	1.061	1.029	1.112	0.983	0.953	1.042	1.041	1.042	1.126	1.074
	Repl. 2	1.103	0.897	0.949	0.912	1.057	0.842	0.829	0.971	0.932	0.961	1.075	0.987
	Repl. 3	1.088	0.954	0.981	0.959	1.106	0.912	0.874	1.011	0.984	0.977	1.083	1.021
	Mean	*1.101*	*0.961*	*0.997*	*0.966*	*1.092*	*0.912*	*0.885*	*1.008*	*0.986*	*0.994*	*1.095*	*1.027*
	SD	*0.010*	*0.056*	*0.047*	*0.048*	*0.025*	*0.057*	*0.051*	*0.029*	*0.045*	*0.035*	*0.023*	*0.036*
	%RSD	*0.91*	*5.8*	*4.8*	*5.0*	*2.3*	*6.3*	*5.8*	*2.9*	*4.5*	*3.5*	*2.1*	*3.5*

Table S17a. GC-FID accuracy data for Class 2A solvents (Run 1) in the oral suspension product. Values in red do not meet acceptance criteria (±20% of spiked value).

Spike Level / %	Replicate Number and Statistical Parameters	Acetonitrile	Chlorobenzene	Cumene	Cyclohexane	1,2-Dichloroethene	1,4-Dioxane	Methanol	Methylcyclohexane	Methylene chloride	Tetrahydrofuran	Toluene	Xylene
50	Repl. 1	0.539	0.441	0.377	0.344	0.441	0.494	0.458	0.286	0.446	0.445	0.441	0.466
	Repl. 2	0.675	0.429	0.352	0.301	0.410	0.544	0.521	0.239	0.456	0.442	0.425	0.457
	Repl. 3	0.523	0.420	0.371	0.340	0.430	0.470	0.455	0.284	0.441	0.441	0.436	0.456
	Mean	*0.579*	*0.430*	*0.367*	*0.328*	*0.427*	*0.503*	*0.478*	*0.270*	*0.448*	*0.443*	*0.434*	*0.460*
	SD	*0.084*	*0.010*	*0.013*	*0.024*	*0.016*	*0.038*	*0.037*	*0.026*	*0.008*	*0.002*	*0.008*	*0.005*
	%RSD	*14.4*	*2.4*	*3.6*	*7.3*	*3.7*	*7.6*	*7.8*	*9.8*	*1.7*	*0.5*	*1.9*	*1.1*
100	Repl. 1	1.23	0.886	0.855	0.861	0.931	0.986	0.930	0.805	0.936	0.922	0.923	0.930
	Repl. 2	1.20	0.777	0.592	0.776	0.898	0.946	0.904	0.648	0.915	0.899	0.828	0.767
	Repl. 3	1.27	0.900	0.815	0.834	0.925	0.975	0.963	0.745	0.940	0.937	0.923	0.926
	Mean	*1.23*	*0.854*	*0.754*	*0.824*	*0.918*	*0.969*	*0.932*	*0.732*	*0.931*	*0.919*	*0.891*	*0.874*
	SD	*0.035*	*0.068*	*0.142*	*0.043*	*0.018*	*0.021*	*0.029*	*0.079*	*0.014*	*0.019*	*0.055*	*0.093*
	%RSD	*2.9*	*7.9*	*18.8*	*5.3*	*2.0*	*2.1*	*3.1*	*10.8*	*1.5*	*2.1*	*6.1*	*10.6*

Table S17b. GC-FID accuracy data for Class 2A solvents (Run 2) in the oral suspension product. GC-FID response is peak area. Values in red do not meet acceptance criteria (±20% of spiked value), while values in gray are outliers and are <u>not</u> included in calculations.

Spike Level / %	Replicate Number and Statistical Parameters	Acetonitrile	Chlorobenzene	Cumene	Cyclohexane	1,2-Dichloroethene	1,4-Dioxane	Methanol	Methylcyclohexane	Methylene chloride	Tetrahydrofuran	Toluene	Xylene
50	Repl. 1	0.365	0.273	0.180	0.249	0.295	0.325	0.783	0.217	0.280	0.151	0.283	0.256
	Repl. 2	0.518	0.452	0.438	0.442	0.481	0.499	0.504	0.397	0.474	0.477	0.478	0.468
	Repl. 3	0.474	0.458	0.457	0.463	0.473	0.461	0.499	0.443	0.467	0.461	0.472	0.470
	Mean	*0.496*	*0.455*	*0.447*	*0.453*	*0.477*	*0.480*	*0.502*	*0.420*	*0.470*	*0.469*	*0.475*	*0.469*
	SD	*0.022*	*0.004*	*0.009*	*0.011*	*0.004*	*0.019*	*0.003*	*0.023*	*0.004*	*0.008*	*0.003*	*0.002*
	%RSD	*4.5*	*1.0*	*2.0*	*2.4*	*0.81*	*4.0*	*0.53*	*5.5*	*0.81*	*1.7*	*0.64*	*0.37*
100	Repl. 1	0.741	0.957	0.979	1.079	1.030	0.699	0.689	1.059	0.930	0.975	0.998	0.982
	Repl. 2	0.824	0.933	0.995	1.080	1.009	0.799	0.775	1.084	0.925	0.962	0.989	0.981
	Repl. 3	0.815	0.949	0.997	1.078	1.015	0.819	0.850	1.075	0.918	0.963	0.988	0.980
	Mean	*0.793*	*0.946*	*0.991*	*1.079*	*1.018*	*0.772*	*0.771*	*1.072*	*0.924*	*0.967*	*0.992*	*0.981*
	SD	*0.037*	*0.013*	*0.008*	*0.001*	*0.009*	*0.052*	*0.066*	*0.010*	*0.005*	*0.006*	*0.005*	*0.001*
	%RSD	*4.7*	*1.3*	*0.80*	*0.08*	*0.86*	*6.8*	*8.6*	*1.0*	*0.54*	*0.62*	*0.45*	*0.11*

Table S18a. GC-FID accuracy data for Class 2B solvents (Run 1) in the oral suspension product. Values in red do not meet acceptance criteria ($\pm$20% of spiked value).

Spike Level / %	Replicate Number and Statistical Parameters	Chloroform	1,2-Dimethoxyethane	Hexane	Methylbutylketone	Nitromethane	Pyridine	Tetralin	Trichloroethylene
50	Repl. 1	0.479	0.253	0.408	0.390	0.388	0.414	0.440	0.466
	Repl. 2	0.531	0.171	0.366	0.492	0.390	0.458	0.458	0.439
	Repl. 3	0.474	0.219	0.398	0.418	0.376	0.425	0.427	0.470
	Mean	*0.495*	*0.214*	*0.391*	*0.433*	*0.385*	*0.432*	*0.442*	*0.458*
	SD	*0.032*	*0.041*	*0.022*	*0.053*	*0.008*	*0.023*	*0.015*	*0.017*
	%RSD	*6.4*	*19.1*	*5.6*	*12.2*	*2.0*	*5.2*	*3.5*	*3.7*
100	Repl. 1	1.04	0.280	0.900	0.907	0.874	0.843	0.894	1.02
	Repl. 2	1.00	0.190	0.857	0.863	0.822	0.802	0.671	0.928
	Repl. 3	1.05	0.274	0.885	0.892	0.825	0.844	0.877	1.03
	Mean	*1.03*	*0.248*	*0.881*	*0.887*	*0.840*	*0.830*	*0.814*	*0.991*
	SD	*0.027*	*0.050*	*0.022*	*0.022*	*0.029*	*0.024*	*0.124*	*0.055*
	%RSD	*2.7*	*20.3*	*2.5*	*2.5*	*3.5*	*2.9*	*15.3*	*5.6*

Table S18b. GC-FID accuracy data for Class 2B solvents (Run 2) in the oral suspension product. Values in red do not meet acceptance criteria (±20% of spiked value), while values in gray are outliers and are <u>not</u> included in calculations.

Spike Level / %	Replicate Number and Statistical Parameters	Chloroform	1,2-Dimethoxyethane	Hexane	Methylbutylketone	Nitromethane	Pyridine	Tetralin	Trichloroethylene
50	Repl. 1	0.339	0.318	0.208	0.314	0.193	0.421	0.308	0.247
	Repl. 2	0.447	0.436	0.484	0.448	0.507	0.573	0.469	0.474
	Repl. 3	0.446	0.336	0.474	0.434	0.423	0.568	0.459	0.467
	Mean	*0.446*	*0.386*	*0.479*	*0.441*	*0.465*	*0.570*	*0.464*	*0.470*
	SD	*0.001*	*0.050*	*0.005*	*0.007*	*0.042*	*0.003*	*0.005*	*0.004*
	%RSD	*0.12*	*12.9*	*1.1*	*1.6*	*9.0*	*0.49*	*1.1*	*0.75*
100	Repl. 1	0.790	0.611	1.070	0.790	1.023	1.014	0.888	0.976
	Repl. 2	0.796	0.749	1.062	0.783	1.011	1.016	0.895	0.977
	Repl. 3	0.800	0.946	1.071	0.803	1.063	1.013	0.895	0.984
	Mean	*0.796*	*0.769*	*1.068*	*0.792*	*1.032*	*1.014*	*0.893*	*0.979*
	SD	*0.004*	*0.137*	*0.004*	*0.008*	*0.022*	*0.001*	*0.004*	*0.004*
	%RSD	*0.53*	*17.9*	*0.35*	*1.0*	*2.1*	*0.11*	*0.41*	*0.38*

E.1. Tablet

Table S19a. SIFT-MS recovery data for Class 2A solvents (Run 1) in the tableted product. All data are percentages. Values in red do not meet acceptance criteria (±20% of spiked value).

Spike Level / %	Replicate Number and Statistical Parameters	Acetonitrile (H₃O⁺ 42)	Chlorobenzene (O₂⁺• 112)	Chlorobenzene (O₂⁺• 114)	Cumene (NO⁺ 120)	Cyclohexane (NO⁺ 83)	Cyclohexane (O₂⁺• 84)	1,2-Dichloroethene (H₃O⁺ 99)	1,2-Dichloroethene (O₂⁺• 96)	1,2-Dichloroethene (O₂⁺• 98)	1,4-Dioxane (H₃O⁺ 89)	1,4-Dioxane (NO⁺ 87)	1,4-Dioxane (NO⁺ 88)	Methanol (H₃O⁺ 33)	Methylcyclohexane (NO⁺ 97)	Methylene chloride (O₂⁺• 84)	Methylene chloride (O₂⁺• 86)	Tetrahydrofuran (NO⁺ 71)	Toluene (NO⁺ 92)	Toluene (O₂⁺• 92)	Xylene (NO⁺ 106)
50	Repl. 1	87.7	81.0	80.2	74.0	69.5	74.2	87.0	88.2	79.2	89.9	78.7	76.3	89.8	59.5	71.5	82.3	82.9	90.1	81.4	80.6
	Repl. 2	89.2	81.5	81.9	76.5	68.6	72.7	76.7	89.0	81.2	92.3	89.9	80.6	92.2	59.3	69.7	79.5	88.4	93.5	82.3	81.9
	Repl. 3	88.4	79.3	80.2	72.8	66.0	70.1	80.3	85.3	77.8	80.0	80.9	76.6	87.3	56.1	67.4	79.2	85.2	89.1	77.1	79.9
	Mean	*88.4*	*80.6*	*80.8*	*74.4*	*68.0*	*72.3*	*81.3*	*87.5*	*79.4*	*87.4*	*83.2*	*77.8*	*89.8*	*58.3*	*69.5*	*80.4*	*85.5*	*90.9*	*80.2*	*80.8*
	SD	*0.63*	*0.94*	*0.82*	*1.5*	*1.5*	*1.7*	*4.3*	*1.6*	*1.4*	*5.3*	*4.8*	*2.0*	*2.0*	*1.6*	*1.7*	*1.4*	*2.2*	*1.9*	*2.3*	*0.85*
	%RSD	*0.72*	*1.2*	*1.0*	*2.0*	*2.2*	*2.4*	*5.2*	*1.8*	*1.7*	*6.1*	*5.8*	*2.5*	*2.2*	*2.7*	*2.4*	*1.7*	*2.6*	*2.1*	*2.8*	*1.1*
100	Repl. 1	90.8	94.1	92.9	120.2	84.8	81.2	94.0	95.8	91.0	92.2	86.5	82.0	88.2	76.5	80.6	88.3	88.2	96.9	92.8	105.8
	Repl. 2	91.2	90.7	89.1	85.6	78.4	78.3	90.8	94.8	87.2	89.4	90.6	85.5	92.4	68.8	77.0	84.4	89.0	93.8	91.1	89.6
	Repl. 3	91.2	89.9	91.1	80.8	77.3	76.3	85.9	94.8	85.5	92.8	81.5	87.7	87.9	66.4	74.5	85.2	89.7	94.4	91.1	87.9
	Mean	*91.1*	*91.6*	*91.0*	*95.6*	*80.2*	*78.6*	*90.2*	*95.1*	*87.9*	*91.5*	*86.2*	*85.1*	*89.5*	*70.6*	*77.4*	*86.0*	*89.0*	*95.0*	*91.7*	*94.5*
	SD	*0.18*	*1.8*	*1.5*	*17.5*	*3.32*	*2.0*	*3.4*	*0.51*	*2.3*	*1.5*	*3.7*	*2.4*	*2.1*	*4.3*	*2.5*	*1.7*	*0.62*	*1.4*	*0.82*	*8.1*
	%RSD	*0.20*	*2.0*	*1.7*	*18.4*	*4.14*	*2.6*	*3.7*	*0.54*	*2.6*	*1.6*	*4.3*	*2.8*	*2.3*	*6.1*	*3.3*	*2.0*	*0.70*	*1.4*	*0.90*	*8.6*

Table S19b. SIFT-MS recovery data for Class 2A solvents (Run 2) in the tableted product. All data are percentages.

Spike Level / %	Replicate Number and Statistical Parameters	Acetonitrile (H₃O⁺ 42)	Chlorobenzene (O₂⁺• 112)	Chlorobenzene (O₂⁺• 114)	Cumene (NO⁺ 120)	Cyclohexane (NO⁺ 83)	Cyclohexane (O₂⁺• 84)	1,2-Dichloroethene (H₃O⁺ 99)	1,2-Dichloroethene (O₂⁺• 96)	1,2-Dichloroethene (O₂⁺• 98)	1,4-Dioxane (H₃O⁺ 89)	1,4-Dioxane (NO⁺ 87)	1,4-Dioxane (NO⁺ 88)	Methanol (H₃O⁺ 33)	Methylcyclohexane (NO⁺ 97)	Methylene chloride (O₂⁺• 84)	Methylene chloride (O₂⁺• 86)	Tetrahydrofuran (NO⁺ 71)	Toluene (NO⁺ 92)	Toluene (O₂⁺• 92)	Xylene (NO⁺ 106)
50	Repl. 1	105.0	92.6	94.2	87.4	95.3	100.0	105.3	98.3	96.2	98.4	85.5	96.0	95.1	86.0	96.4	95.1	93.4	100.7	92.7	91.2
	Repl. 2	85.4	76.1	77.5	75.1	69.5	83.1	68.0	81.6	80.9	82.3	96.8	91.1	86.8	76.8	79.5	77.7	81.6	82.6	75.2	75.9
	Repl. 3	116.3	99.9	101.3	92.5	95.3	101.0	107.5	103.8	101.1	106.3	109.1	106.7	107.7	88.6	98.0	103.2	105.3	106.0	97.7	96.2
	Mean	*102.3*	*89.5*	*91.0*	*85.0*	*86.7*	*94.7*	*93.6*	*94.6*	*92.7*	*95.7*	*97.1*	*97.9*	*96.5*	*83.8*	*91.3*	*92.0*	*93.4*	*96.4*	*88.5*	*87.8*
	SD	*12.8*	*9.9*	*10.0*	*7.3*	*12.1*	*8.2*	*18.1*	*9.4*	*8.6*	*10.0*	*9.6*	*6.5*	*8.6*	*5.1*	*8.4*	*10.6*	*9.7*	*10.0*	*9.6*	*8.6*
	%RSD	*12.5*	*11.1*	*10.9*	*8.6*	*14.0*	*8.7*	*19.3*	*10.0*	*9.3*	*10.4*	*9.9*	*6.7*	*8.9*	*6.0*	*9.2*	*11.5*	*10.4*	*10.4*	*10.9*	*9.8*
100	Repl. 1	86.8	95.7	95.3	105.9	112.6	109.5	94.7	97.9	100.5	77.8	86.9	87.4	83.1	115.0	109.1	88.0	81.5	100.1	98.7	99.5
	Repl. 2	91.3	100.9	102.0	105.9	113.4	111.9	107.6	103.4	106.0	91.9	96.6	84.9	90.9	114.1	111.6	94.5	87.4	103.9	104.2	103.3
	Repl. 3	89.4	98.4	99.8	100.8	111.8	109.5	107.4	101.1	102.3	91.1	77.2	84.9	85.4	106.7	109.1	93.9	84.4	102.7	100.5	99.5
	Mean	*89.2*	*98.3*	*99.0*	*104.2*	*112.6*	*110.3*	*103.2*	*100.8*	*103.0*	*86.9*	*86.9*	*85.7*	*86.5*	*112.0*	*110.0*	*92.1*	*84.4*	*102.2*	*101.1*	*100.8*
	SD	*1.9*	*2.1*	*2.8*	*2.4*	*0.67*	*1.1*	*6.0*	*2.3*	*2.3*	*6.5*	*7.9*	*1.2*	*3.3*	*3.7*	*1.2*	*2.9*	*2.4*	*1.6*	*2.3*	*1.8*
	%RSD	*2.1*	*2.2*	*2.8*	*2.3*	*0.60*	*1.0*	*5.8*	*2.3*	*2.2*	*7.4*	*9.1*	*1.4*	*3.8*	*3.3*	*1.1*	*3.2*	*2.8*	*1.6*	*2.3*	*1.8*

Table S20a. SIFT-MS recovery data for Class 2B solvents (Run 1) in the tableted product. All data are percentages. Values in red do not meet acceptance criteria (±20% of spiked value).

Spike Level / %	Replicate Number and Statistical Parameters	Chloroform ($O_2^{+\bullet}$ 83)	Chloroform ($O_2^{+\bullet}$ 85)	1,2-Dimethoxyethane (H_3O^+ 91)	1,2-Dimethoxyethane (NO^+ 89)	Hexane (NO^+ 85)	Methylbutylketone (NO^+ 130)	Nitromethane (H_3O^+ 62)	Pyridine (H_3O^+ 80)	Tetralin (NO^+ 132)	Tetralin ($O_2^{+\bullet}$ 104)	Trichloroethylene ($O_2^{+\bullet}$ 130)	Trichloroethylene ($O_2^{+\bullet}$ 132)
50	Repl. 1	62.3	77.8	75.5	97.9	81.9	90.5	111.4	67.8	76.0	78.0	83.9	69.3
	Repl. 2	60.8	75.8	81.5	86.3	80.9	90.5	103.9	81.1	77.9	81.0	81.4	70.0
	Repl. 3	58.2	74.9	78.2	80.6	86.6	88.6	93.0	84.6	76.7	75.8	79.7	68.0
	Mean	*60.4*	*76.2*	*78.4*	*88.2*	*83.1*	*89.9*	*102.7*	*77.9*	*76.9*	*78.2*	*81.7*	*69.1*
	SD	*1.7*	*1.2*	*2.4*	*7.2*	*2.5*	*0.90*	*7.5*	*7.2*	*0.76*	*2.1*	*1.7*	*0.82*
	%RSD	*2.8*	*1.6*	*3.1*	*8.2*	*3.0*	*1.0*	*7.3*	*9.3*	*1.0*	*2.7*	*2.1*	*1.2*
100	Repl. 1	78.7	85.3	90.0	80.7	93.4	86.2	99.6	83.8	85.0	83.2	94.2	85.9
	Repl. 2	72.9	80.8	88.1	84.4	85.1	85.5	95.5	95.4	80.6	80.9	90.7	81.9
	Repl. 3	69.3	82.0	85.9	83.8	85.0	86.2	90.8	94.1	83.3	83.3	92.6	83.3
	Mean	*73.6*	*82.7*	*88.0*	*83.0*	*87.9*	*86.0*	*95.3*	*91.1*	*83.0*	*82.5*	*92.5*	*83.7*
	SD	*3.9*	*1.9*	*1.7*	*1.6*	*3.9*	*0.35*	*3.6*	*5.2*	*1.8*	*1.1*	*1.4*	*1.6*
	%RSD	*5.3*	*2.3*	*1.9*	*2.0*	*4.5*	*0.41*	*3.8*	*5.7*	*2.2*	*1.3*	*1.6*	*2.0*

Table S20b. SIFT-MS recovery data for Class 2B solvents (Run 2) in the tableted product. All data are percentages.

Spike Level / %	Replicate Number and Statistical Parameters	Chloroform $(O_2^{+\bullet}$ 83)	Chloroform $(O_2^{+\bullet}$ 85)	1,2-Dimethoxyethane $(H_3O^+$ 91)	1,2-Dimethoxyethane $(NO^+$ 89)	Hexane $(NO^+$ 85)	Methylbutylketone $(NO^+$ 130)	Nitromethane $(H_3O^+$ 62)	Pyridine $(H_3O^+$ 80)	Tetralin $(NO^+$ 132)	Tetralin $(O_2^{+\bullet}$ 104)	Trichloroethylene $(O_2^{+\bullet}$ 130)	Trichloroethylene $(O_2^{+\bullet}$ 132)
50	Repl. 1	87.4	93.2	87.2	97.1	95.5	98.6	101.1	78.2	92.0	94.2	91.4	86.0
	Repl. 2	67.5	74.9	72.8	75.1	74.0	82.1	77.6	73.3	73.6	73.9	71.5	65.6
	Repl. 3	89.4	102.8	99.0	107.5	100.1	110.4	116.1	92.2	101.2	98.7	99.7	90.9
	Mean	*81.4*	*90.3*	*86.3*	*93.2*	*89.9*	*97.0*	*98.2*	*81.3*	*88.9*	*88.9*	*87.5*	*80.8*
	SD	*9.9*	*11.6*	*10.7*	*13.5*	*11.4*	*11.6*	*15.9*	*8.0*	*11.5*	*10.8*	*11.8*	*11.0*
	%RSD	*12.1*	*12.8*	*12.4*	*14.5*	*12.7*	*12.0*	*16.1*	*9.8*	*12.9*	*12.1*	*13.5*	*13.6*
100	Repl. 1	108.7	86.8	93.2	83.0	86.8	83.5	86.8	81.7	94.4	92.2	107.5	97.2
	Repl. 2	110.9	93.1	99.6	90.1	95.2	90.6	88.1	97.2	99.6	98.0	106.6	102.4
	Repl. 3	108.4	90.8	93.7	89.8	96.3	87.1	88.1	94.3	96.3	96.4	106.6	101.5
	Mean	*109.3*	*90.3*	*95.5*	*87.7*	*92.8*	*87.1*	*87.6*	*91.1*	*96.8*	*95.5*	*106.9*	*100.4*
	SD	*1.1*	*2.6*	*2.9*	*3.3*	*4.2*	*2.9*	*0.62*	*6.7*	*2.2*	*2.5*	*0.41*	*2.3*
	%RSD	*1.0*	*2.9*	*3.0*	*3.7*	*4.5*	*3.3*	*0.70*	*7.4*	*2.2*	*2.6*	*0.38*	*2.3*

Table S21a. GC-FID recovery data for Class 2A solvents (Run 1) in the tableted product. All data are percentages. Values in red do not meet acceptance criteria (±20% of spiked value).

Spike Level / %	Replicate Number and Statistical Parameters	Acetonitrile	Chlorobenzene	Cumene	Cyclohexane	1,2-Dichloroethene	1,4-Dioxane	Methanol	Methylcyclohexane	Methylene chloride	Tetrahydrofuran	Toluene	Xylene
50	Repl. 1	87.0	89.2	74.5	71.3	98.3	81.6	81.6	57.5	94.4	94.0	92.9	86.7
	Repl. 2	72.7	89.5	75.2	70.8	96.0	94.6	91.9	58.8	93.9	94.8	93.2	86.9
	Repl. 3	100.8	85.5	66.0	53.8	81.7	98.1	107.5	44.0	91.2	86.9	84.2	80.0
	Mean	*86.8*	*88.0*	*71.9*	*65.3*	*92.0*	*91.4*	*93.7*	*53.4*	*93.1*	*91.9*	*90.1*	*84.5*
	SD	*11.5*	*1.8*	*4.2*	*8.2*	*7.3*	*7.1*	*10.7*	*6.7*	*1.4*	*3.5*	*4.2*	*3.9*
	%RSD	*13.2*	*2.1*	*5.9*	*12.5*	*8.0*	*7.8*	*11.4*	*12.5*	*1.5*	*3.8*	*4.6*	*4.7*
100	Repl. 1	101.4	91.9	70.8	73.0	100.2	97.9	100.9	58.9	98.9	99.2	94.5	86.0
	Repl. 2	96.4	88.7	64.6	65.2	94.6	91.5	94.0	49.8	92.1	94.5	90.4	81.5
	Repl. 3	88.4	92.0	74.4	75.9	98.7	98.7	99.8	64.4	97.4	97.4	94.2	86.8
	Mean	**95.4**	**90.9**	**69.9**	**71.4**	**97.8**	**96.0**	**98.2**	**57.7**	**96.1**	**97.0**	**93.0**	**84.8**
	SD	**5.3**	**1.5**	**4.1**	**4.5**	**2.4**	**3.2**	**3.0**	**6.0**	**2.9**	**1.9**	**1.9**	**2.8**
	%RSD	**5.6**	**1.7**	**5.8**	**6.3**	**2.4**	**3.3**	**3.1**	**10.5**	**3.0**	**2.0**	**2.0**	**3.3**

Table S21b. GC-FID recovery data for Class 2A solvents (Run 2) in the tableted product. All data are percentages. Values in red do not meet acceptance criteria (±20% of spiked value), while values in gray are outliers and are <u>not</u> included in calculations.

Spike Level / %	Replicate Number and Statistical Parameters	Acetonitrile	Chlorobenzene	Cumene	Cyclohexane	1,2-Dichloroethene	1,4-Dioxane	Methanol	Methylcyclohexane	Methylene chloride	Tetrahydrofuran	Toluene	Xylene
50	Repl. 1	97.0	95.3	77.8	79.9	98.5	87.6	91.3	63.4	99.1	97.7	96.7	90.8
	Repl. 2	108.4	98.6	94.2	84.0	93.9	92.6	90.0	80.4	100.9	98.1	97.0	97.5
	Repl. 3	105.8	95.7	94.9	90.8	94.3	99.3	110.1	88.7	97.3	95.4	96.3	97.0
	Mean	*103.8*	*96.5*	*88.9*	*84.9*	*95.6*	*93.2*	*97.1*	*77.5*	*99.1*	*97.1*	*96.7*	*95.1*
	SD	*6.0*	*1.8*	*9.7*	*5.5*	*2.6*	*5.9*	*11.2*	*12.9*	*1.8*	*1.4*	*0.4*	*3.7*
	%RSD	*5.8*	*1.9*	*10.9*	*6.4*	*2.7*	*6.3*	*11.6*	*16.7*	*1.8*	*1.5*	*0.4*	*3.9*
100	Repl. 1	78.5	94.7	94.5	100.4	101.7	73.5	74.0	96.3	93.2	96.9	98.9	95.9
	Repl. 2	81.3	97.8	98.6	104.6	103.5	70.4	74.9	101.6	95.5	98.1	101.3	99.2
	Repl. 3	47.8	63.1	67.8	70.4	67.9	39.9	42.9	70.4	61.8	64.7	65.9	65.5
	Mean	*79.9*	*96.3*	*96.6*	*102.5*	*102.6*	*71.9*	*74.4*	*98.9*	*94.4*	*97.5*	*100.1*	*97.6*
	SD	*2.0*	*2.2*	*2.9*	*3.0*	*1.3*	*2.2*	*0.6*	*3.8*	*1.6*	*0.9*	*1.7*	*2.3*
	%RSD	*2.5*	*2.3*	*3.0*	*2.9*	*1.2*	*3.0*	*0.9*	*3.8*	*1.7*	*0.9*	*1.7*	*2.4*

Table S22a. GC-FID recovery data for Class 2B solvents (Run 1) in the tableted product. All data are percentages. Values in red do not meet acceptance criteria (±20% of spiked value).

Spike Level / %	Replicate Number and Statistical Parameters	Chloroform	1,2-Dimethoxyethane	Hexane	Methylbutylketone	Nitromethane	Pyridine	Tetralin	Trichloroethylene
50	Repl. 1	87.9	207.1	83.9	86.6	94.5	52.3	88.6	91.1
	Repl. 2	89.7	178.9	79.5	88.2	82.6	64.3	88.3	86.1
	Repl. 3	97.5	106.9	61.9	94.8	62.5	77.5	87.7	77.3
	Mean	*91.7*	*164.3*	*75.1*	*89.8*	*79.8*	*64.7*	*88.2*	*84.8*
	SD	*4.2*	*42.2*	*9.5*	*3.5*	*13.2*	*10.3*	*0.4*	*5.7*
	%RSD	*4.6*	*25.7*	*12.7*	*3.9*	*16.5*	*15.9*	*0.4*	*6.7*
100	Repl. 1	97.8	114.5	89.6	93.9	92.3	95.8	93.8	102.0
	Repl. 2	92.2	91.2	79.4	91.7	80.3	84.3	88.1	92.8
	Repl. 3	97.0	95.3	83.4	93.5	86.0	95.2	90.3	95.9
	Mean	*95.7*	*100.3*	*84.1*	*93.0*	*86.2*	*91.8*	*90.7*	*96.9*
	SD	*2.5*	*10.2*	*4.2*	*1.0*	*4.9*	*5.3*	*2.3*	*3.8*
	%RSD	*2.6*	*10.1*	*5.0*	*1.0*	*5.7*	*5.8*	*2.6*	*3.9*

Table S22b. GC-FID recovery data for Class 2B solvents (Run 2) in the tableted product. All data are percentages. Values in red do not meet acceptance criteria (±20% of spiked value), while values in gray are outliers and are <u>not</u> included in calculations.

Spike Level / %	Replicate Number and Statistical Parameters	Chloroform	1,2-Dimethoxyethane	Hexane	Methylbutylketone	Nitromethane	Pyridine	Tetralin	Trichloroethylene
50	Repl. 1	95.8	156.3	96.5	95.1	97.2	119.3	98.6	90.7
	Repl. 2	108.2	136.8	86.5	105.9	89.5	133.2	103.7	91.9
	Repl. 3	99.9	140.8	89.5	93.8	81.5	121.9	95.8	93.3
	Mean	*101.3*	*144.6*	*90.8*	*98.3*	*89.4*	*124.8*	*99.4*	*92.0*
	SD	*6.3*	*10.3*	*5.2*	*6.6*	*7.9*	*7.4*	*4.0*	*1.3*
	%RSD	*6.2*	*7.1*	*5.7*	*6.8*	*8.8*	*5.9*	*4.0*	*1.4*
100	Repl. 1	79.5	79.8	108.2	82.8	101.1	102.6	92.4	101.7
	Repl. 2	84.3	87.3	107.6	85.9	104.9	106.5	94.0	101.8
	Repl. 3	51.9	79.7	70.6	52.6	69.7	67.9	61.2	65.9
	Mean	*81.9*	*83.6*	*107.9*	*84.4*	*103.0*	*104.6*	*93.2*	*101.7*
	SD	*3.4*	*5.4*	*0.4*	*2.3*	*2.7*	*2.8*	*1.1*	*0.1*
	%RSD	*4.2*	*6.4*	*0.4*	*2.7*	*2.6*	*2.7*	*1.2*	*0.1*

E.2. Oral suspension

Table S23a. SIFT-MS recovery data for Class 2A solvents (Run 1) in the oral suspension product. All data are percentages. Values in red do not meet acceptance criteria (±20% of spiked value).

Spike Level / %	Replicate Number and Statistical Parameters	Acetonitrile (H$_3$O$^+$ 42)	Chlorobenzene (O$_2^{+\bullet}$ 112)	Chlorobenzene (O$_2^{+\bullet}$ 114)	Cumene (NO$^+$ 120)	Cyclohexane (NO$^+$ 83)	Cyclohexane (O$_2^{+\bullet}$ 84)	1,2-Dichloroethene (H$_3$O$^+$ 99)	1,2-Dichloroethene (O$_2^{+\bullet}$ 96)	1,2-Dichloroethene (O$_2^{+\bullet}$ 98)	1,4-Dioxane (H$_3$O$^+$ 89)	1,4-Dioxane (NO$^+$ 87)	1,4-Dioxane (NO$^+$ 88)	Methanol (H$_3$O$^+$ 33)	Methylcyclohexane (NO$^+$ 97)	Methylene chloride (O$_2^{+\bullet}$ 84)	Methylene chloride (O$_2^{+\bullet}$ 86)	Tetrahydrofuran (NO$^+$ 71)	Toluene (NO$^+$ 92)	Toluene (O$_2^{+\bullet}$ 92)	Xylene (NO$^+$ 106)
50	Repl. 1	93.1	74.9	74.9	70.9	66.4	69.1	73.1	83.2	75.4	1369.2	110.4	150.2	91.4	57.6	66.0	87.8	129.8	86.8	75.4	76.5
	Repl. 2	112.5	88.7	87.2	77.2	70.8	72.7	83.9	92.6	83.6	1851.5	142.0	213.7	114.3	59.3	69.7	100.9	161.2	96.4	85.5	85.0
	Repl. 3	73.7	65.4	65.0	57.6	56.7	61.3	67.7	73.8	67.7	1223.1	99.9	134.7	75.1	47.5	58.3	75.8	109.0	72.5	64.9	64.2
	Mean	*93.1*	*76.3*	*75.7*	*68.6*	*64.6*	*67.7*	*74.9*	*83.2*	*75.6*	*1481.3*	*117.4*	*166.2*	*93.6*	*54.8*	*64.7*	*88.2*	*133.3*	*85.2*	*75.3*	*75.2*
	SD	*15.9*	*9.6*	*9.1*	*8.2*	*5.9*	*4.7*	*6.7*	*7.7*	*6.5*	*268.5*	*17.9*	*34.2*	*16.1*	*5.2*	*4.8*	*10.3*	*21.5*	*9.8*	*8.4*	*8.5*
	%RSD	*17.0*	*12.5*	*12.0*	*11.9*	*9.2*	*7.0*	*9.0*	*9.2*	*8.6*	*18.1*	*15.2*	*20.6*	*17.2*	*9.6*	*7.3*	*11.6*	*16.1*	*11.5*	*11.2*	*11.4*
100	Repl. 1	105.6	91.6	90.8	81.9	78.4	78.1	84.2	97.6	90.6	902.0	120.8	143.4	107.0	67.5	76.8	93.9	125.5	95.0	92.3	90.5
	Repl. 2	102.1	89.9	90.8	82.4	79.1	77.8	85.0	96.9	88.9	842.5	124.9	134.9	104.8	68.9	76.5	93.0	121.7	97.6	93.4	88.9
	Repl. 3	98.6	88.2	86.4	82.4	79.1	78.6	88.3	95.1	87.7	854.4	116.9	143.4	101.8	71.2	77.4	91.4	116.4	93.8	92.3	88.3
	Mean	*102.1*	*89.9*	*89.4*	*82.3*	*78.8*	*78.2*	*85.9*	*96.6*	*89.0*	*866.3*	*120.8*	*140.6*	*104.5*	*69.2*	*76.9*	*92.8*	*121.2*	*95.5*	*92.6*	*89.2*
	SD	*2.9*	*1.4*	*2.1*	*0.25*	*0.31*	*0.3*	*1.8*	*1.1*	*1.2*	*25.7*	*3.3*	*4.0*	*2.1*	*1.5*	*0.39*	*1.0*	*3.7*	*1.6*	*0.55*	*0.93*
	%RSD	*2.8*	*1.5*	*2.3*	*0.30*	*0.40*	*0.4*	*2.1*	*1.1*	*1.3*	*3.0*	*2.7*	*2.9*	*2.0*	*2.2*	*0.50*	*1.1*	*3.1*	*1.7*	*0.59*	*1.0*

Table S23b. SIFT-MS recovery data for Class 2A solvents (Run 2) in the oral suspension product. All data are percentages. Values in red do not meet acceptance criteria (±20% of spiked value).

Spike Level / %	Replicate Number and Statistical Parameters	Acetonitrile (H$_3$O$^+$ 42)	Chlorobenzene (O$_2$$^{+\bullet}$ 112)	Chlorobenzene (O$_2$$^{+\bullet}$ 114)	Cumene (NO$^+$ 120)	Cyclohexane (NO$^+$ 83)	Cyclohexane (O$_2$$^{+\bullet}$ 84)	1,2-Dichloroethene (H$_3$O$^+$ 99)	1,2-Dichloroethene (O$_2$$^{+\bullet}$ 96)	1,2-Dichloroethene (O$_2$$^{+\bullet}$ 98)	1,4-Dioxane (H$_3$O$^+$ 89)	1,4-Dioxane (NO$^+$ 87)	1,4-Dioxane (NO$^+$ 88)	Methanol (H$_3$O$^+$ 33)	Methylcyclohexane (NO$^+$ 97)	Methylene chloride (O$_2$$^{+\bullet}$ 84)	Methylene chloride (O$_2$$^{+\bullet}$ 86)	Tetrahydrofuran (NO$^+$ 71)	Toluene (NO$^+$ 92)	Toluene (O$_2$$^{+\bullet}$ 92)	Xylene (NO$^+$ 106)
50	Repl. 1	107.3	95.6	97.5	94.7	96.9	101.0	113.4	99.9	98.6	209.7	107.7	99.5	100.2	92.7	98.0	100.3	103.0	99.5	93.1	94.8
	Repl. 2	107.3	91.9	93.6	90.1	94.5	98.2	109.4	99.1	96.8	194.3	95.0	105.7	101.0	87.6	95.6	97.1	97.5	99.8	92.8	90.1
	Repl. 3	106.5	90.7	94.9	87.7	96.9	95.3	115.5	95.9	95.0	196.9	102.1	104.6	96.3	84.7	93.1	95.8	96.4	96.7	89.7	88.6
	Mean	*107.0*	*92.8*	*95.3*	*90.8*	*96.1*	*98.2*	*112.8*	*98.3*	*96.8*	***200.3***	*101.6*	*103.3*	*99.1*	*88.3*	*95.6*	*97.7*	*99.0*	*98.7*	*91.9*	*91.2*
	SD	*0.36*	*2.1*	*1.6*	*2.9*	*1.2*	*2.3*	*2.5*	*1.7*	*1.5*	*6.7*	*5.2*	*2.7*	*2.1*	*3.3*	*2.0*	*1.9*	*2.9*	*1.4*	*1.5*	*2.6*
	%RSD	*0.33*	*2.2*	*1.7*	*3.2*	*1.2*	*2.4*	*2.2*	*1.7*	*1.6*	*3.4*	*5.1*	*2.6*	*2.1*	*3.7*	*2.1*	*1.9*	*2.9*	*1.4*	*1.7*	*2.9*
100	Repl. 1	101.5	106.7	106.9	107.8	110.1	110.9	111.6	107.0	108.8	147.1	98.7	95.2	96.4	110.7	111.2	104.1	100.5	106.5	108.6	106.2
	Repl. 2	88.3	98.4	97.5	103.4	112.6	111.4	114.9	100.3	102.3	126.1	86.7	83.3	81.1	112.2	111.2	91.8	85.2	101.4	101.1	100.2
	Repl. 3	93.2	99.9	102.0	102.7	113.8	109.5	113.8	102.2	103.6	135.1	97.3	89.0	87.2	111.0	109.1	97.1	91.0	102.0	102.4	102.4
	Mean	*94.3*	*101.7*	*102.1*	*104.6*	*112.2*	*110.6*	*113.4*	*103.2*	*104.9*	***136.1***	*94.3*	*89.2*	*88.3*	*111.3*	*110.5*	*97.7*	*92.2*	*103.3*	*104.0*	*103.0*
	SD	*5.4*	*3.6*	*3.8*	*2.2*	*1.5*	*0.80*	*1.4*	*2.8*	*2.8*	*8.6*	*5.3*	*4.9*	*6.3*	*0.61*	*1.0*	*5.1*	*6.3*	*2.3*	*3.3*	*2.5*
	%RSD	*5.8*	*3.5*	*3.7*	*2.1*	*1.4*	*0.73*	*1.2*	*2.7*	*2.7*	*6.3*	*5.7*	*5.4*	*7.1*	*0.55*	*0.87*	*5.2*	*6.8*	*2.2*	*3.1*	*2.4*

Table S24a. SIFT-MS recovery data for Class 2B solvents (Run 1) in the oral suspension product. All data are percentages. Values in red do not meet acceptance criteria (±20% of spiked value).

Spike Level / %	Replicate Number and Statistical Parameters	Chloroform ($O_2^{+\bullet}$ 83)	Chloroform ($O_2^{+\bullet}$ 85)	1,2-Dimethoxyethane (H_3O^+ 91)	1,2-Dimethoxyethane (NO^+ 89)	Hexane (NO^+ 85)	Methylbutylketone (NO^+ 130)	Nitromethane (H_3O^+ 62)	Pyridine (H_3O^+ 80)	Tetralin (NO^+ 132)	Tetralin ($O_2^{+\bullet}$ 104)	Trichloroethylene ($O_2^{+\bullet}$ 130)	Trichloroethylene ($O_2^{+\bullet}$ 132)
50	Repl. 1	56.8	90.1	80.9	161.3	1836.6	93.7	101.1	64.3	69.8	72.1	96.3	62.3
	Repl. 2	60.6	102.9	92.5	200.0	2385.9	112.1	108.0	81.5	81.6	82.5	113.9	72.6
	Repl. 3	49.1	77.4	62.5	120.4	1622.1	72.7	79.4	99.0	55.2	60.8	81.3	51.7
	Mean	*55.5*	*90.1*	*78.6*	*160.6*	*1948.2*	*92.8*	*96.2*	*81.6*	*68.9*	*71.8*	*97.2*	*62.2*
	SD	*4.8*	*10.4*	*12.3*	*32.5*	*321.6*	*16.1*	*12.2*	*14.2*	*10.8*	*8.9*	*13.3*	*8.6*
	%RSD	*8.6*	*11.5*	*15.7*	*20.2*	*16.5*	*17.3*	*12.7*	*17.3*	*15.7*	*12.4*	*13.7*	*13.8*
100	Repl. 1	73.4	94.6	90.2	144.1	1061.2	100.2	100.3	79.6	79.2	78.9	106.8	80.9
	Repl. 2	72.8	92.9	89.9	140.4	995.4	96.2	95.9	85.4	76.4	80.1	106.8	80.9
	Repl. 3	74.3	91.5	88.0	139.8	1033.0	87.1	86.7	81.5	71.9	73.3	99.2	76.0
	Mean	*73.5*	*93.0*	*89.3*	*141.5*	*1029.9*	*94.5*	*94.3*	*82.2*	*75.9*	*77.5*	*104.3*	*79.3*
	SD	*0.6*	*1.3*	*1.0*	*1.9*	*27.0*	*5.5*	*5.7*	*2.4*	*3.0*	*3.0*	*3.6*	*2.3*
	%RSD	*0.86*	*1.4*	*1.1*	*1.4*	*2.6*	*5.8*	*6.0*	*2.9*	*4.0*	*3.9*	*3.4*	*2.9*

Table S24b. SIFT-MS recovery data for Class 2B solvents (Run 2) in the oral suspension product. All data are percentages. Values in red do not meet acceptance criteria (±20% of spiked value).

Spike Level / %	Replicate Number and Statistical Parameters	Chloroform ($O_2^{+\bullet}$ 83)	Chloroform ($O_2^{+\bullet}$ 85)	1,2-Dimethoxyethane (H_3O^+ 91)	1,2-Dimethoxyethane (NO^+ 89)	Hexane (NO^+ 85)	Methylbutylketone (NO^+ 130)	Nitromethane (H_3O^+ 62)	Pyridine (H_3O^+ 80)	Tetralin (NO^+ 132)	Tetralin ($O_2^{+\bullet}$ 104)	Trichloroethylene ($O_2^{+\bullet}$ 130)	Trichloroethylene ($O_2^{+\bullet}$ 132)
50	Repl. 1	91.1	99.0	94.6	99.7	130.3	103.9	105.0	78.9	95.5	96.9	98.5	86.6
	Repl. 2	87.4	95.4	91.6	99.1	122.7	102.5	93.2	94.3	93.6	93.5	95.7	86.6
	Repl. 3	85.9	94.5	91.3	96.5	123.1	98.0	94.5	85.2	91.2	91.3	95.6	84.1
	Mean	*88.1*	*96.3*	*92.5*	*98.4*	*125.4*	*101.5*	*97.6*	*86.1*	*93.4*	*93.9*	*96.6*	*85.8*
	SD	*2.2*	*1.9*	*1.5*	*1.4*	*3.5*	*2.5*	*5.3*	*6.3*	*1.8*	*2.3*	*1.3*	*1.2*
	%RSD	*2.5*	*2.0*	*1.6*	*1.4*	*2.8*	*2.5*	*5.4*	*7.3*	*1.9*	*2.5*	*1.4*	*1.4*
100	Repl. 1	111.3	103.3	106.1	102.9	111.2	98.3	95.3	104.2	104.1	104.2	112.6	107.4
	Repl. 2	110.3	89.7	94.9	91.2	105.7	84.2	82.9	97.1	93.2	96.1	107.5	98.7
	Repl. 3	108.8	95.4	98.1	95.9	110.6	91.2	87.4	101.1	98.4	97.7	108.3	102.1
	Mean	*110.1*	*96.1*	*99.7*	*96.6*	*109.2*	*91.2*	*88.5*	*100.8*	*98.6*	*99.4*	*109.5*	*102.7*
	SD	*1.0*	*5.6*	*4.7*	*4.8*	*2.5*	*5.7*	*5.1*	*2.9*	*4.5*	*3.5*	*2.3*	*3.6*
	%RSD	*0.91*	*5.8*	*4.8*	*5.0*	*2.3*	*6.3*	*5.8*	*2.9*	*4.5*	*3.5*	*2.1*	*3.5*

Table S25a. GC-FID recovery data for Class 2A solvents (Run 1) in the oral suspension product. All data are percentages. Values in red do not meet acceptance criteria (±20% of spiked value).

Spike Level / %	Replicate Number and Statistical Parameters	Acetonitrile	Chlorobenzene	Cumene	Cyclohexane	1,2-Dichloroethene	1,4-Dioxane	Methanol	Methylcyclohexane	Methylene chloride	Tetrahydrofuran	Toluene	Xylene
50	Repl. 1	101.8	90.2	73.8	70.4	98.1	96.3	91.9	57.8	95.1	95.2	93.0	92.8
	Repl. 2	121.4	88.0	70.0	63.6	91.9	106.9	105.3	51.4	97.1	94.6	90.0	91.3
	Repl. 3	99.5	86.3	72.9	69.7	96.0	91.0	91.1	57.5	94.2	94.2	92.0	91.0
	Mean	*107.6*	*88.2*	*72.2*	*67.9*	*95.4*	*98.1*	*96.1*	*55.5*	*95.5*	*94.7*	*91.7*	*91.7*
	SD	*9.8*	*1.6*	*1.6*	*3.0*	*2.6*	*6.6*	*6.5*	*2.9*	*1.2*	*0.4*	*1.2*	*0.9*
	%RSD	*9.1*	*1.8*	*2.2*	*4.5*	*2.7*	*6.7*	*6.8*	*5.3*	*1.3*	*0.4*	*1.4*	*1.0*
100	Repl. 1	100.7	87.0	72.7	75.8	98.5	100.6	96.0	64.3	95.4	94.7	92.1	87.1
	Repl. 2	98.3	76.7	53.0	69.1	95.1	96.4	93.3	53.6	93.3	92.4	83.1	73.0
	Repl. 3	103.4	88.3	69.7	73.7	97.9	99.4	99.5	60.2	95.7	96.2	92.1	86.8
	Mean	*100.8*	*84.0*	*65.1*	*72.9*	*97.2*	*98.8*	*96.3*	*59.4*	*94.8*	*94.4*	*89.1*	*82.3*
	SD	*2.1*	*5.2*	*8.7*	*2.8*	*1.5*	*1.8*	*2.5*	*4.4*	*1.1*	*1.6*	*4.2*	*8.1*
	%RSD	*2.1*	*6.2*	*13.3*	*3.8*	*1.5*	*1.8*	*2.6*	*7.5*	*1.1*	*1.6*	*4.7*	*9.8*

Table S25b. GC-FID recovery data for Class 2A solvents (Run 2) in the oral suspension product. All data are percentages. Values in red do not meet acceptance criteria (±20% of spiked value), while values in gray are outliers and are <u>not</u> included in calculations.

Spike Level / %	Replicate Number and Statistical Parameters	Acetonitrile	Chlorobenzene	Cumene	Cyclohexane	1,2-Dichloroethene	1,4-Dioxane	Methanol	Methylcyclohexane	Methylene chloride	Tetrahydrofuran	Toluene	Xylene
50	Repl. 1	73.0	54.5	35.9	49.9	59.1	65.0	52.2	43.3	56.0	30.2	56.7	52.1
	Repl. 2	103.7	90.4	87.7	88.4	96.1	99.9	100.9	79.4	94.8	95.5	95.6	94.1
	Repl. 3	94.7	91.6	91.3	92.7	94.6	92.3	99.8	88.5	93.3	92.3	94.3	94.3
	Mean	*99.2*	*91.0*	*89.5*	*90.5*	*95.3*	*96.1*	*100.4*	*84.0*	*94.1*	*93.9*	*94.9*	*94.2*
	SD	*6.3*	*0.88*	*2.6*	*3.0*	*1.1*	*5.4*	*0.7*	*6.5*	*1.1*	*2.3*	*0.9*	*0.18*
	%RSD	*6.4*	*0.97*	*2.9*	*3.3*	*1.2*	*5.6*	*0.7*	*7.7*	*1.1*	*2.4*	*0.9*	*0.20*
100	Repl. 1	74.1	95.7	97.9	107.9	103.0	69.9	68.9	105.9	93.0	97.5	99.8	97.9
	Repl. 2	82.4	93.3	99.5	108.0	100.9	79.9	77.5	108.4	92.5	96.2	98.9	97.8
	Repl. 3	81.5	94.9	99.7	107.8	101.5	81.9	85.0	107.5	91.8	96.3	98.8	97.6
	Mean	*79.3*	*94.6*	*99.1*	*107.9*	*101.8*	*77.2*	*77.1*	*107.2*	*92.4*	*96.7*	*99.2*	*97.8*
	SD	*4.6*	*1.26*	*1.0*	*0.1*	*1.1*	*6.4*	*8.1*	*1.3*	*0.6*	*0.7*	*0.6*	*0.15*
	%RSD	*5.8*	*1.33*	*1.0*	*0.1*	*1.1*	*8.3*	*10.5*	*1.2*	*0.7*	*0.8*	*0.6*	*0.16*

Table S26a. GC-FID recovery data for Class 2B solvents (Run 1) in the oral suspension product. All data are percentages. Values in red do not meet acceptance criteria (±20% of spiked value).

Spike Level / %	Replicate Number and Statistical Parameters	Chloroform	1,2-Dimethoxyethane	Hexane	Methylbutylketone	Nitromethane	Pyridine	Tetralin	Trichloroethylene
50	Repl. 1	92.6	185.0	81.5	79.6	88.3	62.8	86.0	90.4
	Repl. 2	101.8	141.6	74.1	98.9	88.7	73.2	89.1	85.5
	Repl. 3	91.7	168.0	79.8	84.8	86.0	65.5	83.6	91.2
	Mean	*95.4*	*164.9*	*78.5*	*87.8*	*87.7*	*67.2*	*86.2*	*89.0*
	SD	*4.6*	*17.9*	*3.2*	*8.2*	*1.2*	*4.4*	*2.2*	*2.5*
	%RSD	*4.8*	*10.8*	*4.1*	*9.3*	*1.3*	*6.6*	*2.6*	*2.8*
100	Repl. 1	95.3	99.2	84.4	88.8	90.2	82.6	83.3	94.3
	Repl. 2	92.1	76.0	80.6	84.7	85.3	77.8	63.5	86.4
	Repl. 3	96.8	97.7	83.1	87.4	85.6	82.7	81.8	95.4
	Mean	*94.7*	*91.0*	*82.7*	*87.0*	*87.0*	*81.1*	*76.2*	*92.0*
	SD	*2.0*	*10.6*	*1.6*	*1.7*	*2.3*	*2.3*	*9.0*	*4.0*
	%RSD	*2.1*	*11.6*	*1.9*	*2.0*	*2.6*	*2.9*	*11.9*	*4.4*

Table S26b. GC-FID recovery data for Class 2B solvents (Run 2) in the oral suspension product. All data are percentages. Values in red do not meet acceptance criteria (±20% of spiked value), while values in gray are outliers and are <u>not</u> included in calculations.

Spike Level / %	Replicate Number and Statistical Parameters	Chloroform	1,2-Dimethoxyethane	Hexane	Methylbutylketone	Nitromethane	Pyridine	Tetralin	Trichloroethylene
50	Repl. 1	67.8	63.5	41.6	62.8	38.5	84.2	61.7	49.5
	Repl. 2	89.4	87.2	96.8	89.6	101.4	114.7	93.8	94.7
	Repl. 3	89.1	67.3	94.8	86.8	84.6	113.5	91.8	93.3
	Mean	*89.2*	*77.2*	*95.8*	*88.2*	*93.0*	*114.1*	*92.8*	*94.0*
	SD	*0.15*	*14.0*	*1.4*	*2.0*	*11.9*	*0.79*	*1.4*	*1.0*
	%RSD	*0.17*	*18.2*	*1.5*	*2.2*	*12.8*	*0.69*	*1.5*	*1.1*
100	Repl. 1	79.0	61.1	107.0	79.0	102.3	101.4	88.8	97.6
	Repl. 2	79.6	74.9	106.2	78.3	101.1	101.6	89.5	97.7
	Repl. 3	80.0	94.6	107.1	80.3	106.3	101.3	89.5	98.4
	Mean	*79.6*	*76.9*	*106.8*	*79.2*	*103.2*	*101.4*	*89.3*	*97.9*
	SD	*0.52*	*16.8*	*0.45*	*1.0*	*2.7*	*0.14*	*0.45*	*0.45*
	%RSD	*0.65*	*21.9*	*0.42*	*1.3*	*2.6*	*0.13*	*0.50*	*0.46*

Figures

A. Linearity

Figure S1. SIFT-MS linearity plots for Class 2A solvents across the concentration levels 0.2 to 1.0. SIFT-MS response is the headspace concentration in ppmV.
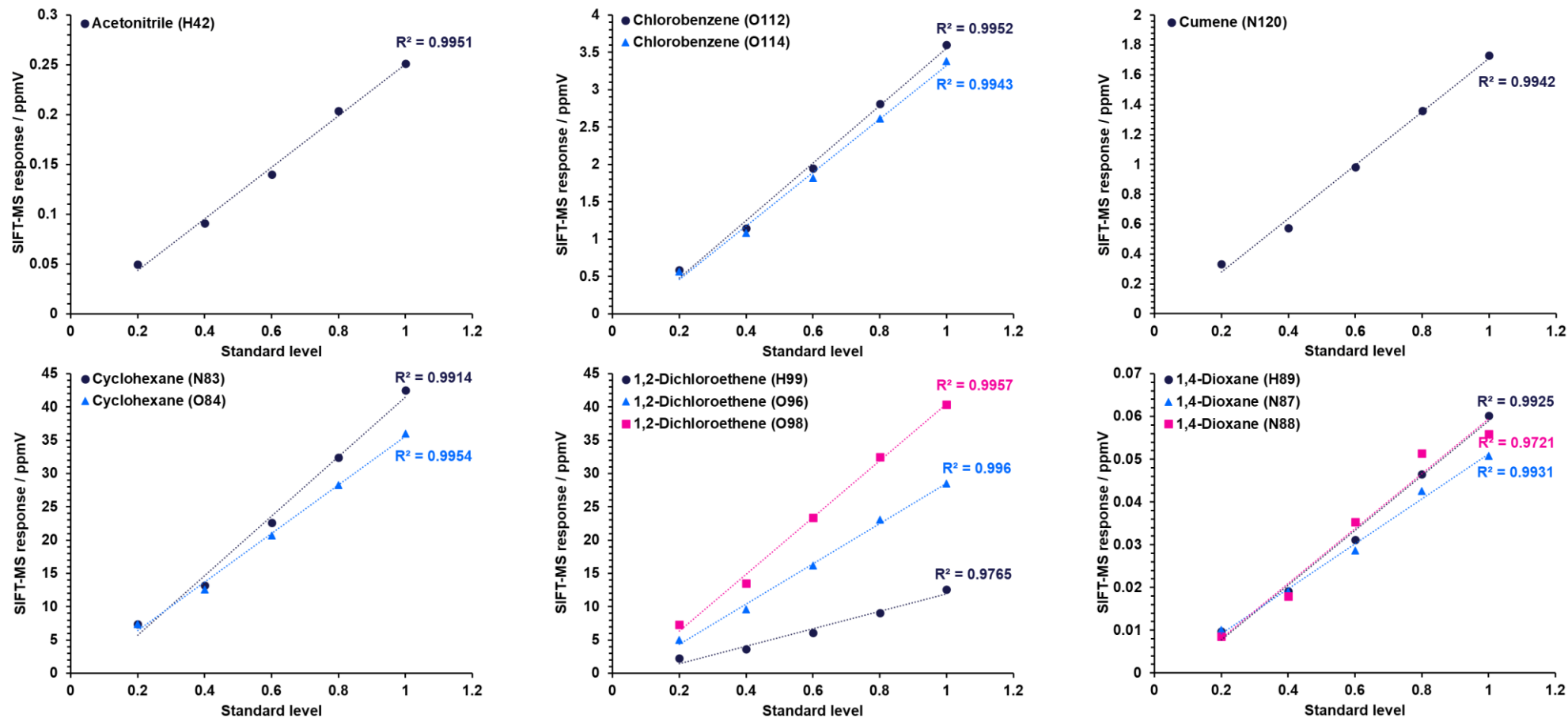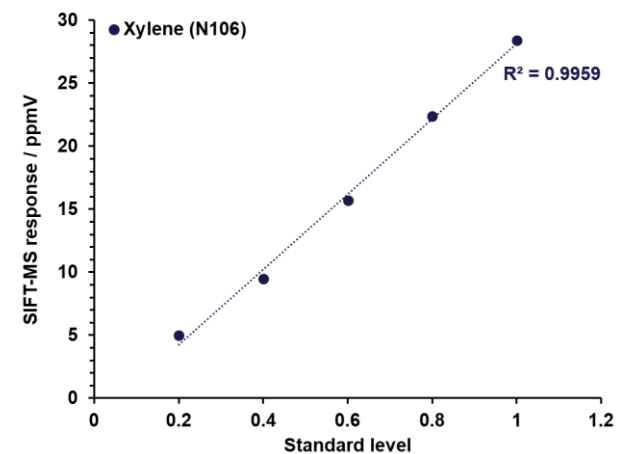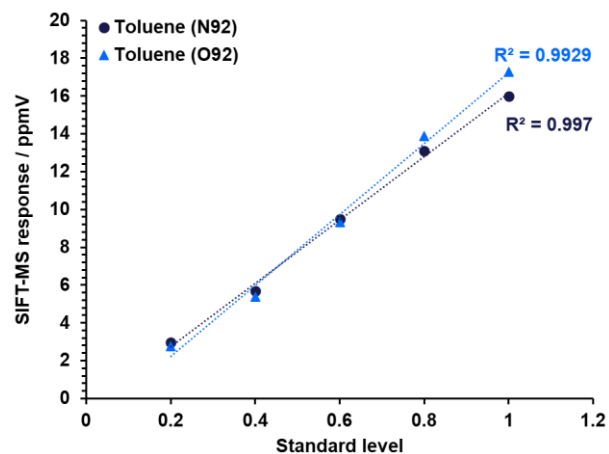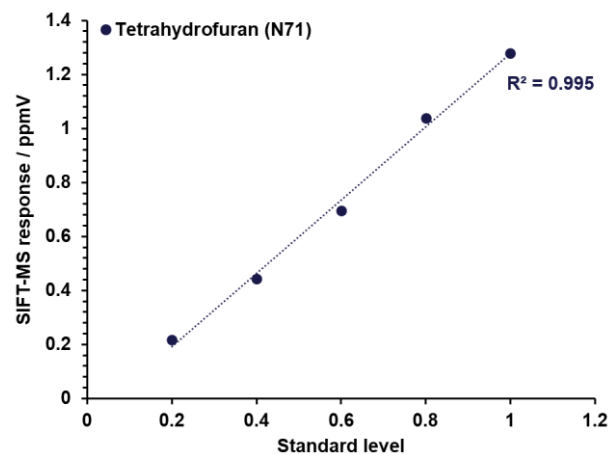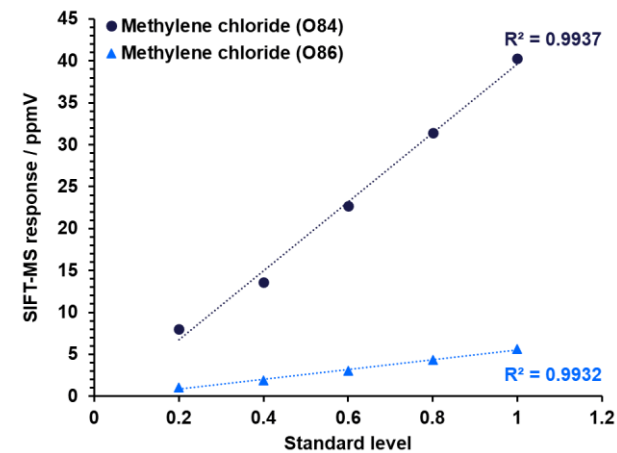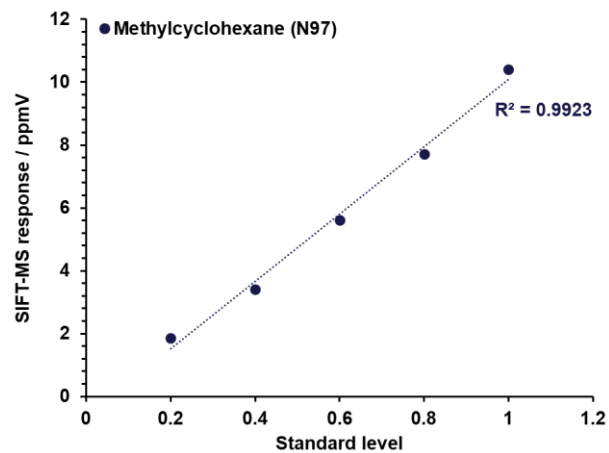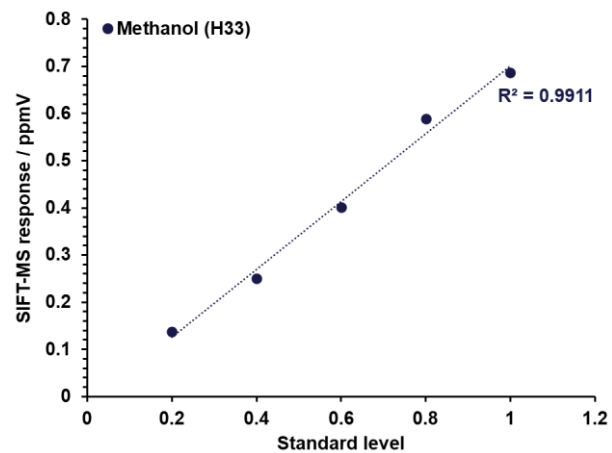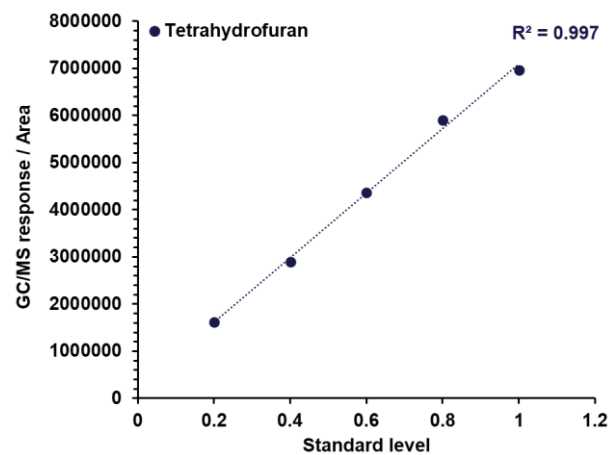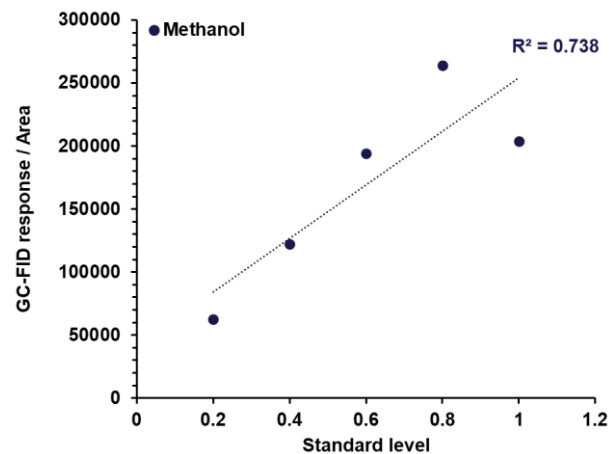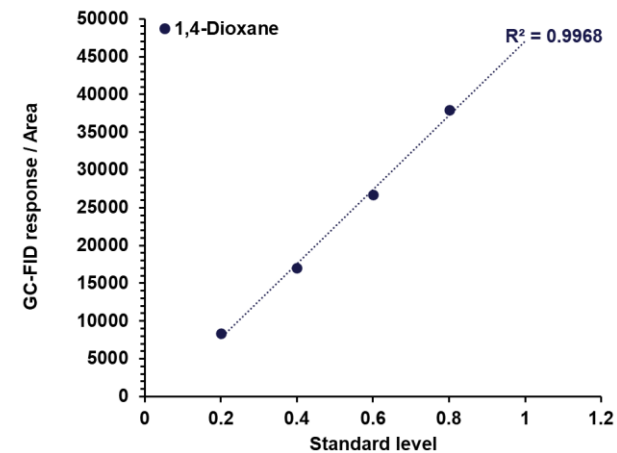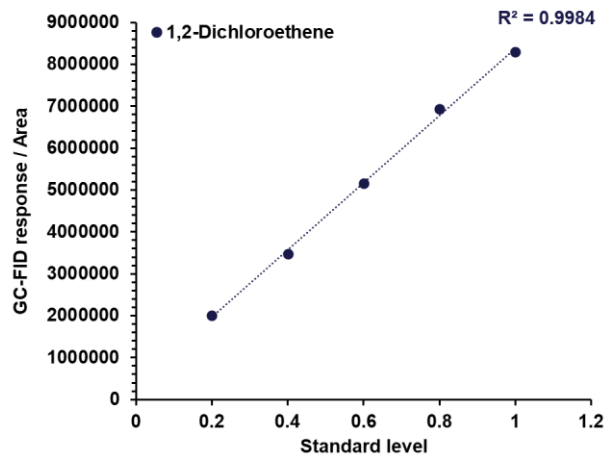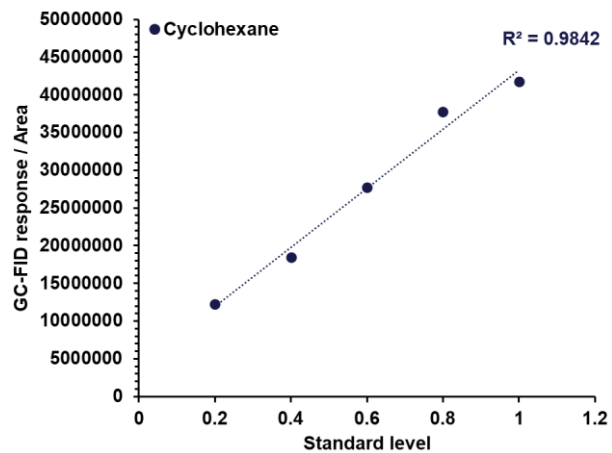
Figure S2. SIFT-MS linearity plots for Class 2B solvents across the concentration levels 0.2 to 1.0. SIFT-MS response is the headspace concentration in ppmV.
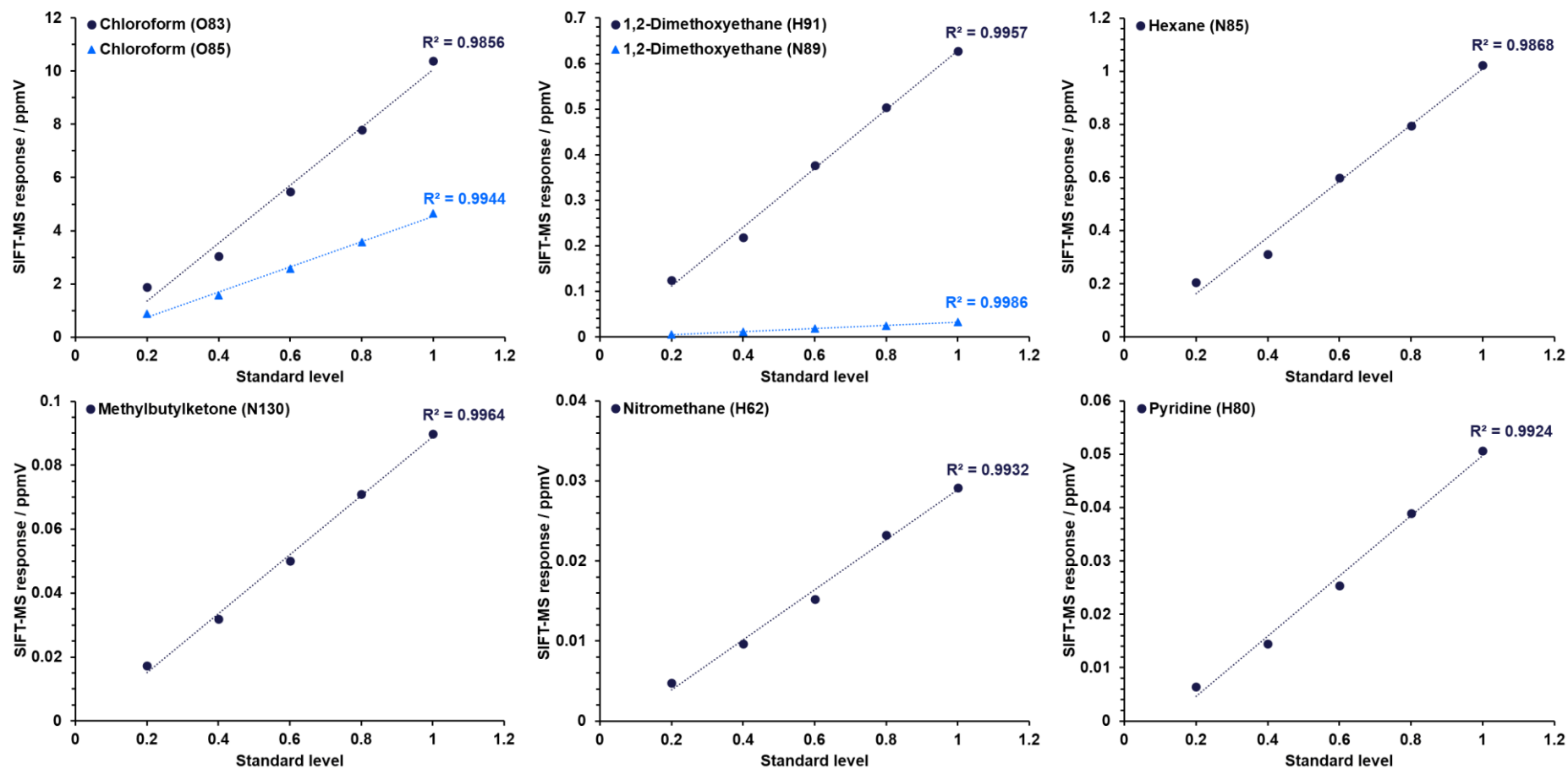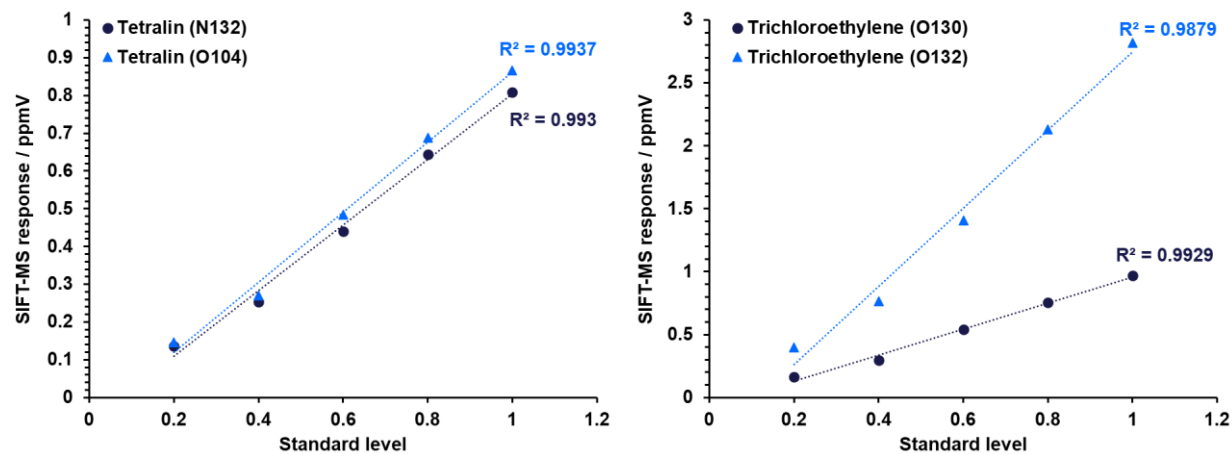
Figure S3. GC-FID linearity plots for Class 2A solvents across the concentration levels 0.2 to 1.0. GC-FID response is peak area. Note that for some analytes level 1.0 is not included as it significantly skewed the linear fit.
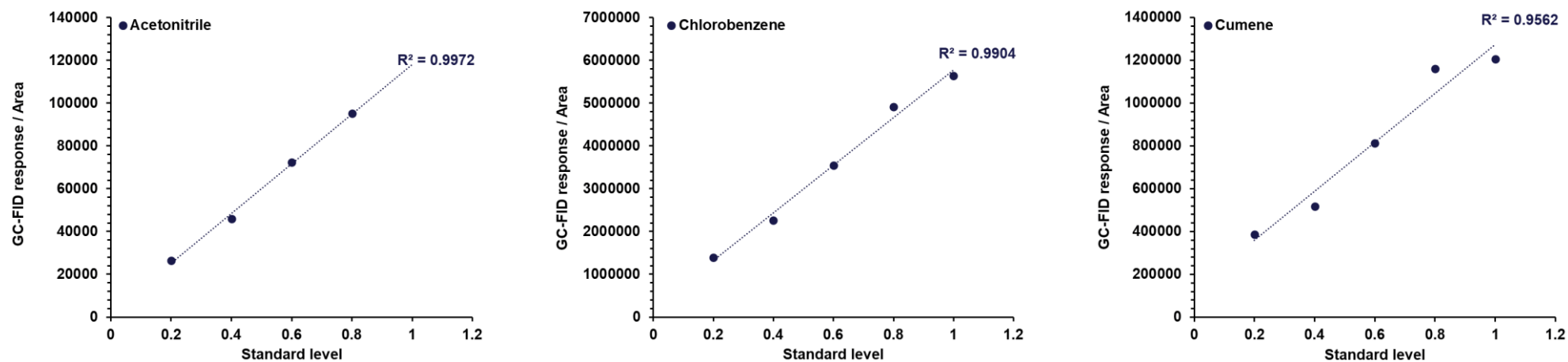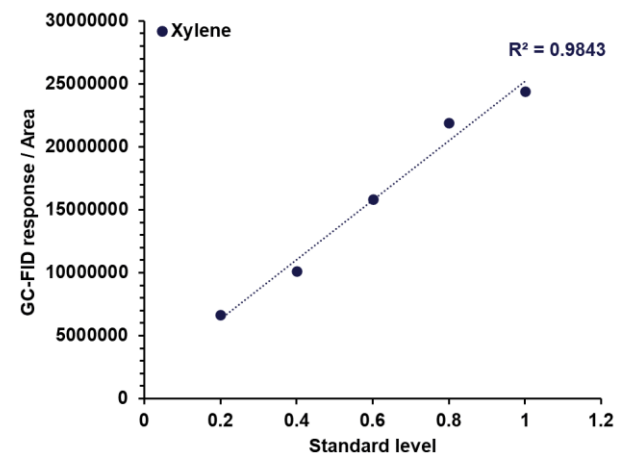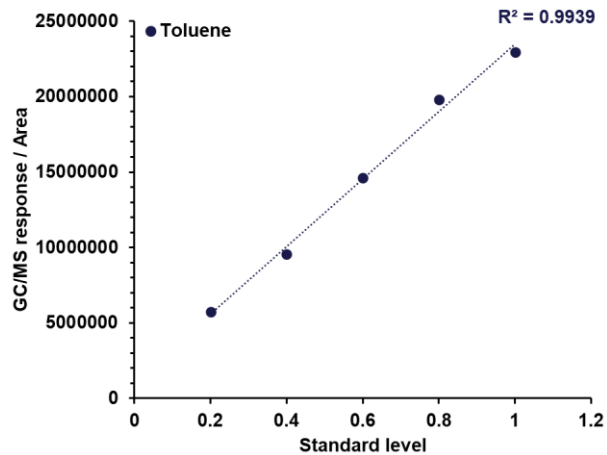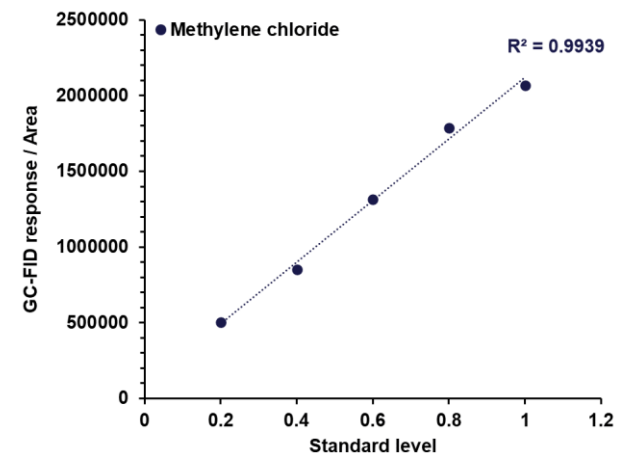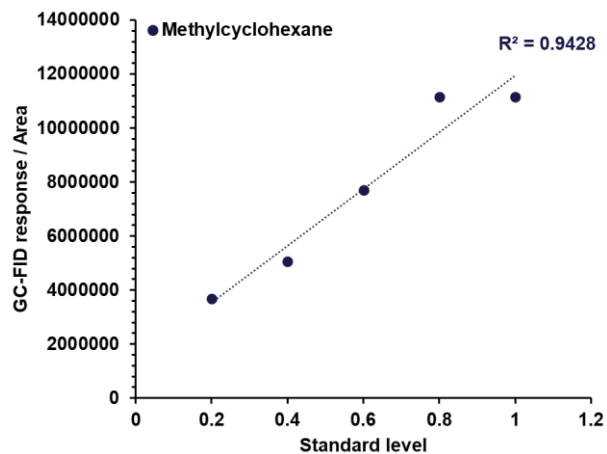
Figure S4. GC-FID linearity plots for Class 2B solvents across the concentration levels 0.2 to 1.0. GC-FID response is peak area. Note that for some analytes level 1.0 is not included as it significantly skewed the linear fit.
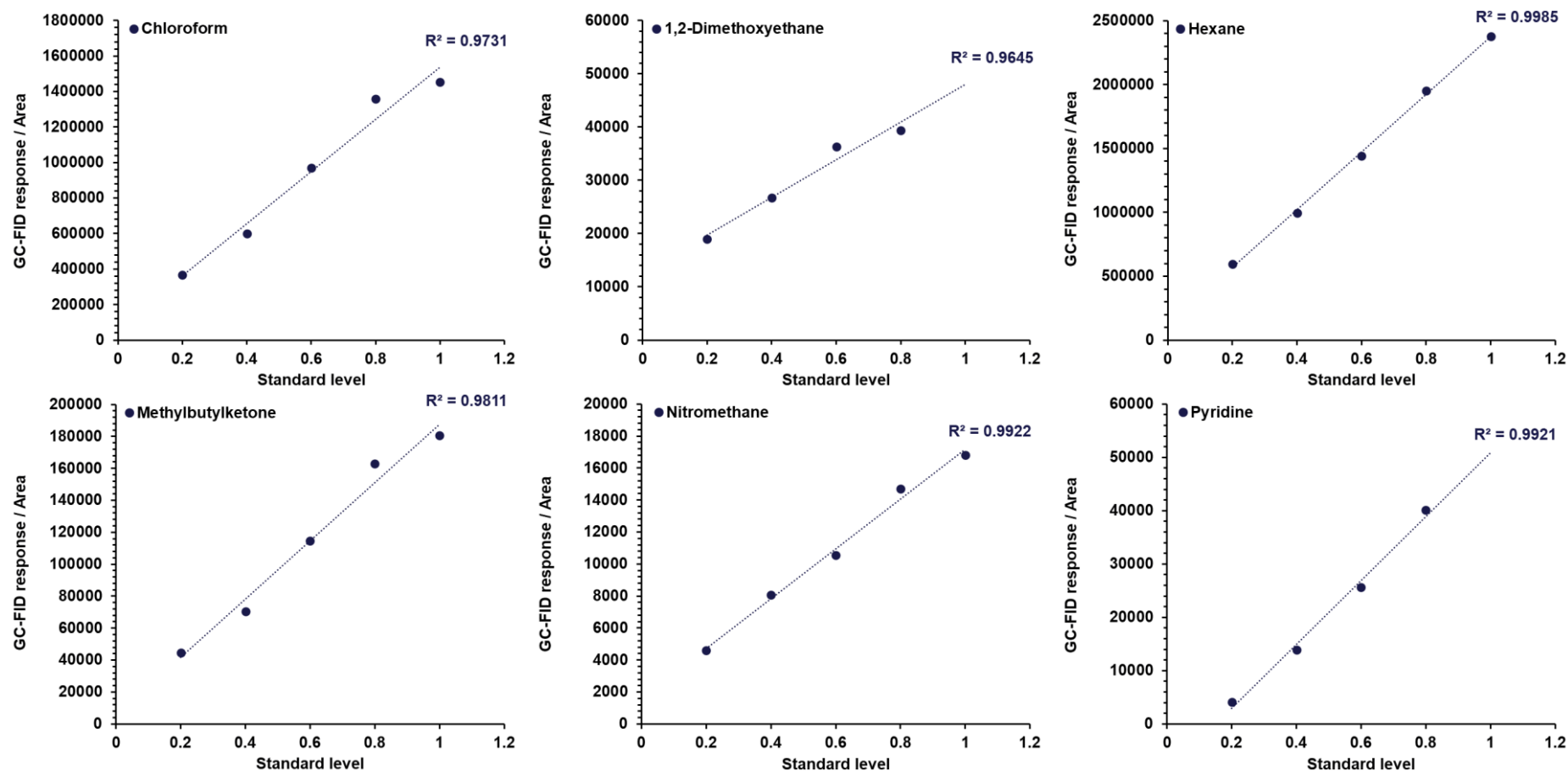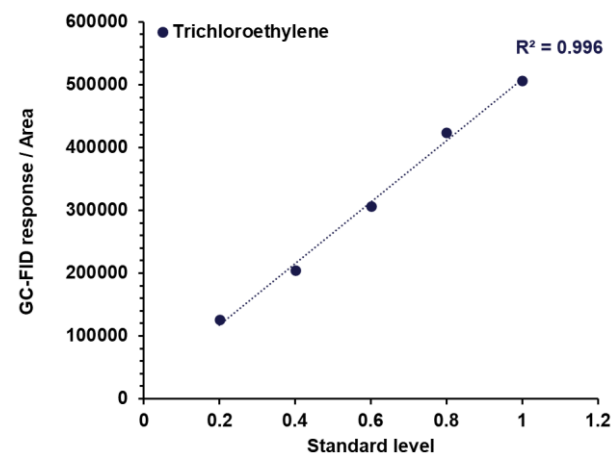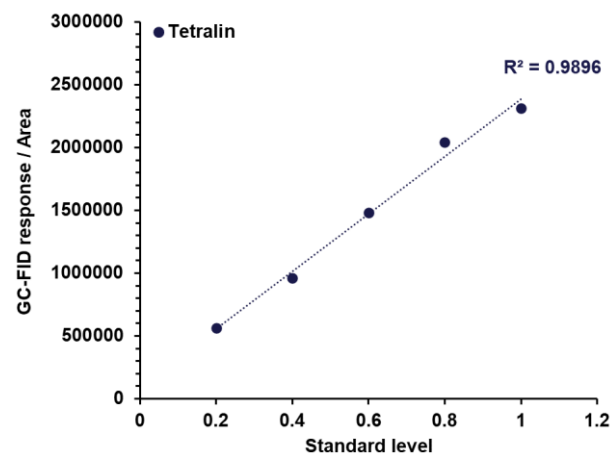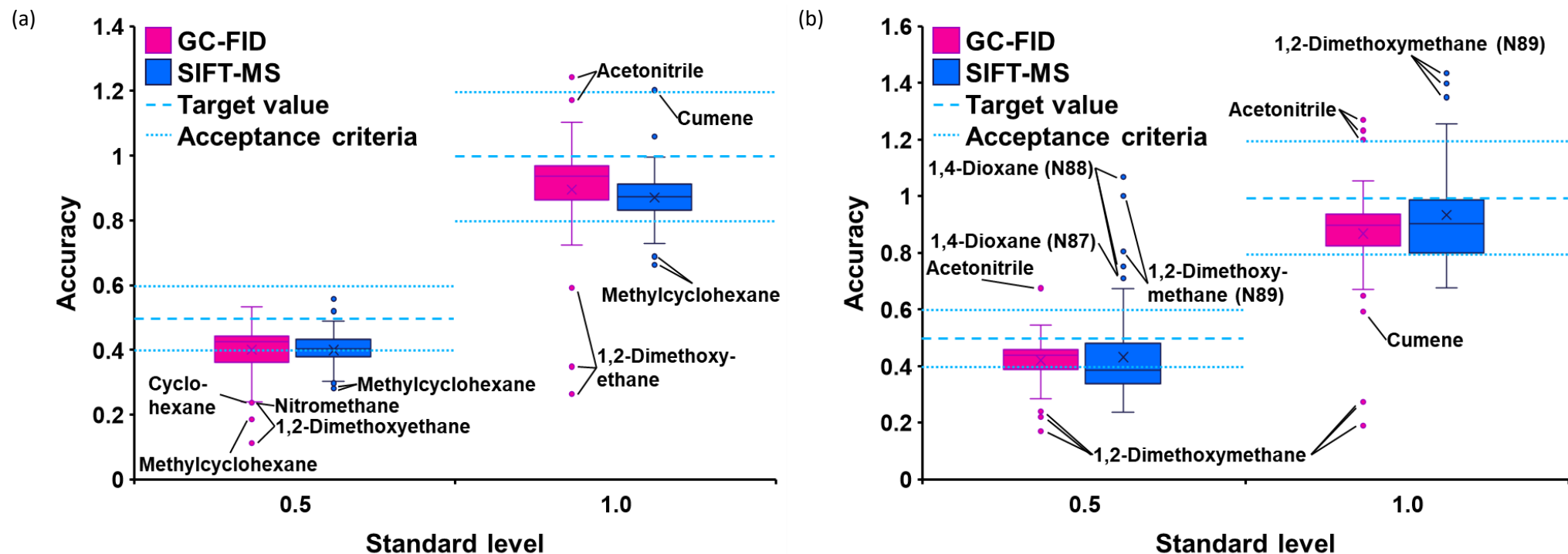
B. Accuracy

Figure S5. Accuracy data from "Run 1" GC-FID and SIFT-MS measurements of 0.5 and 1.0 level solvent spikes in solutions prepared from (a) tableted and (b) oral suspension formulations of acetaminophen (paracetamol). The box-and-whisker plots summarize all replicate data in Tables S11a to S18a, except for (i) the GC-FID replicates that were identified as outliers, and (ii) SIFT-MS ions experiencing significant matrix interference (oral suspension only; 1,4-dioxane (using H_3O^+ and m/z 89) and hexane (NO^+ 85)). "Run 2" data are shown in Figure 6 of the article and in Tables S11b to S18b.

Figure S6. Recovery data from "Run 1" GC-FID and SIFT-MS measurements of 0.5 and 1.0 level solvent spikes in solutions prepared from (a) tableted and (b) oral suspension formulations of acetaminophen (paracetamol). The box-and-whisker plots summarize all replicate data in Tables S19a to S26a, except for (i) the GC-FID replicates that were identified as outliers, and (ii) SIFT-MS ions experiencing significant matrix interference (oral suspension only; 1,4-dioxane (using H_3O^+ and m/z 89) and hexane (NO^+ 85)). "Run 2" data are shown in Figure 7 of the article and in Tables S19b to S26b.