

### Supplemental Data

**Suppl. Table 1.** List of product ions from ETD-MS<sup>2</sup> of ANGPTL3+1Tn precursors ( $\text{MH}_4^{4+}$ , 200 ms reaction time, and  $\text{MH}_3^{3+}$ , 150 ms reaction time) in LTQ-Orbitrap, with deviations of experimental from theoretical  $m/z$  values (err, in ppm) calculated by Protein Prospector (<http://prospector.ucsf.edu/>) MS-Product program for HexNAc at Thr8.

		$404.4780^{4+}$		$538.9697^{3+}$	
	calc.	exp.	err (ppm)	exp.	error (ppm)
c <sub>2</sub>	243.1816	243.1843	11		
z <sub>2</sub>	244.1418	244.1446	12		
c <sub>5</sub> <sup>2+</sup>	284.1899	284.1923	8.4		
c <sub>6</sub> <sup>2+</sup>	362.2405	362.2424	5.3		
z <sub>3</sub>	391.2102	391.2121	4.9		
c <sub>3</sub>	399.2827	399.2845	4.6	399.2846	4.9
c <sub>7</sub> <sup>2+</sup>	412.7643	412.7661	4.4		
z <sub>7</sub> <sup>2+</sup>	525.2739	525.2739	1.7		
c <sub>5</sub>	567.3725	567.3737	2.0	567.3738	2.2
z <sub>9</sub> <sup>2+</sup>	609.3180	609.3191	1.9		
[c <sub>9</sub> -1] <sup>2+</sup>	612.8503			612.8513	1.6
c <sub>9</sub> <sup>2+</sup>	613.3542	613.3550	1.3		
[c <sub>10</sub> -1] <sup>2+</sup>	686.3845			686.3854	1.3
c <sub>10</sub> <sup>2+</sup>	686.8884	686.8889	0.7		
z <sub>10</sub> <sup>2+</sup>	687.3685	687.37	2.2		
c <sub>6</sub>	723.4737	723.4741	0.6	723.4742	0.8
c <sub>6</sub> +1	723.4770	724.4813	5.9		
c <sub>11</sub> <sup>2+</sup>	743.4304			743.4310	0.7
[c <sub>11</sub> +1] <sup>2+</sup>	743.9321	743.9341	2.7		
z <sub>5</sub> *	792.3900	792.3917	2.1		
c <sub>7</sub>	824.5213	824.5213	0.0	824.5212	0.2
c <sub>7</sub> +1	825.5247	825.5279	3.9		
z <sub>6</sub>	893.4377	893.4374	-0.3		
z <sub>7</sub>	1049.5388	1049.5380	-0.7	1049.5385	-0.3
z <sub>7</sub> +1	1050.5466	1050.5448	-1.7		
z <sub>9</sub>	1217.6287	1217.6266	-1.7	1217.6276	-0.9
z <sub>9</sub> +1	1218.6365	1218.6343	-1.8		
c <sub>9</sub>	1225.7011	1225.6991	-1.7	1225.6994	-1.4
c <sub>9</sub> +1	1226.7045	1226.7057	1.0		
c <sub>10</sub>	1372.7696	1372.7668	-2.0	1372.7670	-1.9
c <sub>10</sub> +1	1373.7729	1373.7751	1.6		
z <sub>10</sub> +1	1374.7376	1374.7141	-17		
c <sub>10</sub>	1485.8536	1485.8505	-2.1		
c <sub>11</sub> +1	1486.8570	1486.8583	0.9	1486.8577	0.5
c <sub>11</sub> +2	1487.8603	1487.8655	3.5		

\*Observed at 100 ms ETD reaction time.