Supporting Information

Regioselectivity in the Cu(I)-Catalyzed [4 + 2]-Cycloaddition of 2-Nitrosopyridine with Unsymmetrical Dienes

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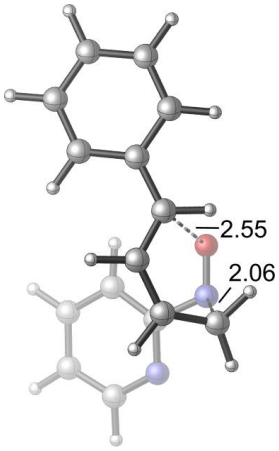
Contents

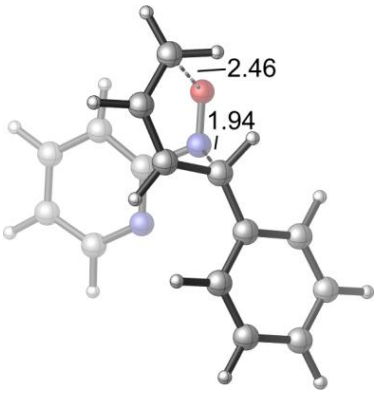
Complete Reference of Gaussian 09

Gaussian 09, Revision D.01, Frisch, M. J.; Trucks, G. W.; Schlegel, H. B.; Scuseria, G. E.; Robb, M. A.; Cheeseman, J. R.; Scalmani, G.; Barone, V.; Mennucci, B.; Petersson, G. A.; Nakatsuji, H.; Caricato, M.; Li, X.; Hratchian, H. P.; Izmaylov, A. F.; Bloino, J.; Zheng, G.; Sonnenberg, J. L.; Hada, M.; Ehara, M.; Toyota, K.; Fukuda, R.; Hasegawa, J.; Ishida, M.; Nakajima, T.; Honda, Y.; Kitao, O.; Nakai, H.; Vreven, T.; Montgomery, J. A., Jr.; Peralta, J. E.; Ogliaro, F.; Bearpark, M.; Heyd, J. J.; Brothers, E.; Kudin, K. N.; Staroverov, V. N.; Kobayashi, R.; Normand, J.; Raghavachari, K.; Rendell, A.; Burant, J. C.; Iyengar, S. S.; Tomasi, J.; Cossi, M.; Rega, N.; Millam, N. J.; Klene, M.; Knox, J. E.; Cross, J. B.; Bakken, V.; Adamo, C.; Jaramillo, J.; Gomperts, R.; Stratmann, R. E.; Yazyev, O.; Austin, A. J.; Cammi, R.; Pomelli, C.; Ochterski, J. W.; Martin, R. L.; Morokuma, K.; Zakrzewski, V. G.; Voth, G. A.; Salvador, P.; Dannenberg, J. J.; Dapprich, S.; Daniels, A. D.; Farkas, Ö.; Foresman, J. B.; Ortiz, J. V.; Cioslowski, J.; Fox, D. J. Gaussian, Inc., Wallingford CT, 2009.

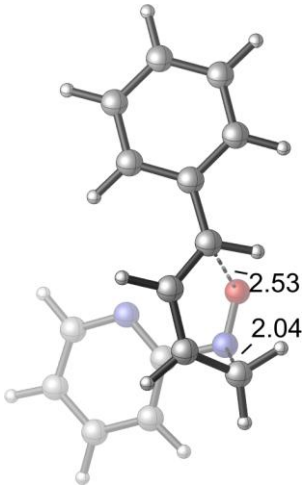
Calculated transition states for the 1-phenylbutadiene plus 2-nitrosopyridine reaction



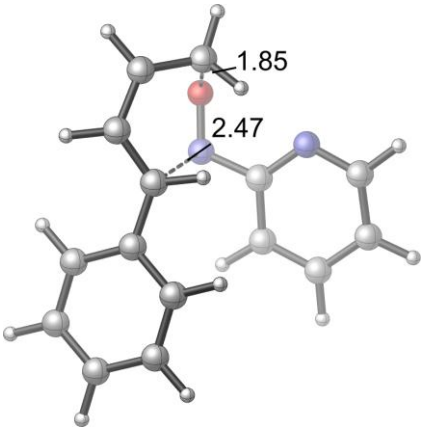
endo-prox-anti-**TS1-Ph**
$\Delta H^{\ddagger}$ = 2.8 kcal/mol
$\Delta G^{\ddagger}$ = 17.1 kcal/mol



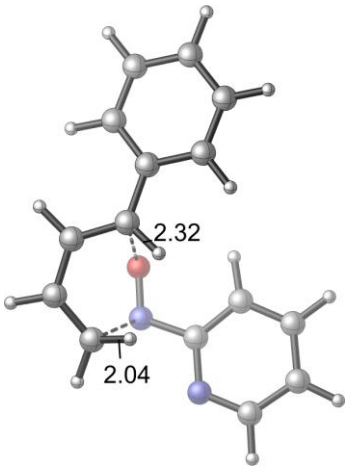
endo-dist-anti-**TS1-Ph**
$\Delta H^{\ddagger}$ = 6.2 kcal/mol
$\Delta G^{\ddagger}$ = 20.8 kcal/mol



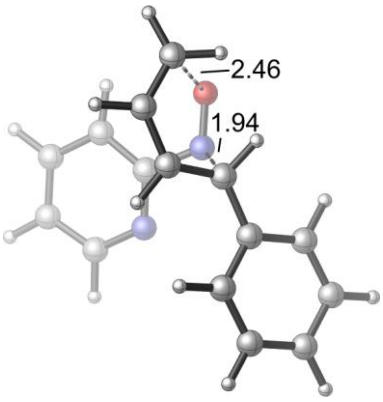
endo-prox-syn-**TS1-Ph**
$\Delta H^{\ddagger}$ = 5.0 kcal/mol
$\Delta G^{\ddagger}$ = 19.6 kcal/mol



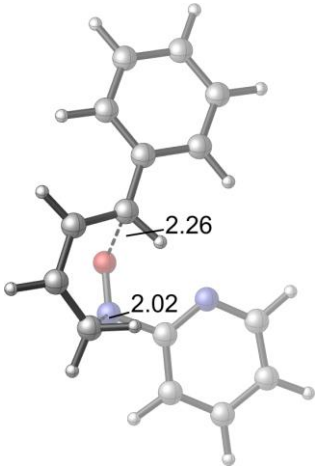
endo-dist-syn-**TS1-Ph**
$\Delta H^{\ddagger}$ = 7.1 kcal/mol
$\Delta G^{\ddagger}$ = 21.7 kcal/mol



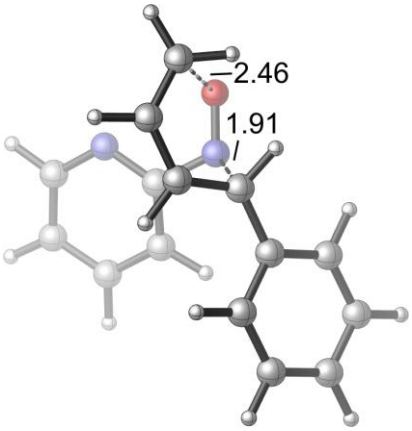
exo-prox-anti-**TS1-Ph**
$\Delta H^{\ddagger}$ = 8.3 kcal/mol
$\Delta G^{\ddagger}$ = 23.0 kcal/mol



exo-dist-anti-**TS1-Ph**
$\Delta H^{\ddagger}$ = 12.3 kcal/mol
$\Delta G^{\ddagger}$ = 27.4 kcal/mol



exo-prox-syn-**TS1-Ph**
$\Delta H^{\ddagger}$ = 10.0 kcal/mol
$\Delta G^{\ddagger}$ = 24.2 kcal/mol



exo-dist-syn-**TS1-Ph**
$\Delta H^{\ddagger}$ = 11.1 kcal/mol
$\Delta G^{\ddagger}$ = 26.0 kcal/mol

Cartesian coordinates and energies

"M06 SCF energy in solution" are M06/SDD–6-311++G(d,p) single point energies with PCM solvation model in methanol. Geometries were optimized with M06/SDD–6-31G(d) with PCM solvation model in methanol. "M06 enthalpy in solution" and "M06 free energy in solution" are M06 single point energies plus zero-point energies and thermal corrections at 298K computed with M06/SDD–6-31G(d) with PCM solvation model in methanol. For transition states, the imaginary frequencies are provided.

endo-prox-anti-TS1

M06 SCF energy in solution: -572.61312028 a.u.
M06 enthalpy in solution: -572.399196 a.u.
M06 free energy in solution: -572.450460 a.u.
Imaginary frequency: -341.8299 cm-1

Cartesian coordinates

ATOM	X	Y	Z
C	-1.026002	-2.147134	-0.221955
C	-1.043661	-1.215289	-1.237933
C	-1.807857	-0.018544	-1.220709
C	-2.674369	0.328963	-0.230500
H	-0.275574	-1.296415	-2.008473
H	-1.569488	0.732979	-1.977546
H	-1.864540	-2.268203	0.460261
H	-3.020579	-0.425336	0.474185
H	-0.345929	-2.993250	-0.279241
O	-1.037942	-0.144183	1.596796
N	-0.165602	-0.920306	1.174436
C	0.957991	-0.234639	0.539167
C	0.993426	1.152886	0.440160
N	1.951566	-1.042884	0.172923
C	2.135503	1.731343	-0.098760
H	0.141184	1.735076	0.780993
C	3.040387	-0.469258	-0.335643
C	3.183574	0.908423	-0.496428
H	2.208410	2.812031	-0.206518
H	3.847544	-1.140899	-0.632914
H	4.097370	1.318772	-0.918962
C	-3.275734	1.680424	-0.097122
H	-4.371805	1.644112	-0.174666
H	-3.050011	2.101328	0.893284
H	-2.894338	2.372558	-0.857943

endo-prox-syn-TS1

M06 SCF energy in solution: -572.60986188 a.u.
M06 enthalpy in solution: -572.395984 a.u.

M06 free energy in solution: -572.447506 a.u.
Imaginary frequency: -375.6705 cm-1

Cartesian coordinates

ATOM	X	Y	Z
C	1.005121	-2.101948	0.236854
C	1.079557	-1.160607	1.245665
C	1.893247	0.001106	1.197987
C	2.752320	0.298962	0.185673
H	0.333511	-1.209737	2.040159
H	1.714215	0.764893	1.958686
H	1.829679	-2.264874	-0.454022
H	3.030600	-0.471547	-0.531287
H	0.320111	-2.941453	0.335306
O	1.010195	-0.140873	-1.590904
N	0.146607	-0.897690	-1.130050
C	-0.983942	-0.206904	-0.498291
C	-2.072137	-1.006084	-0.147248
N	-0.969586	1.116933	-0.412381
C	-3.204728	-0.381731	0.349644
H	-2.016089	-2.086031	-0.270545
C	-2.070647	1.703135	0.066659
C	-3.206970	1.007178	0.464392
H	-4.076740	-0.965540	0.637462
H	-2.046645	2.792360	0.132883
H	-4.074219	1.541603	0.843974
C	3.438921	1.608084	0.042231
H	4.530977	1.496393	0.106311
H	3.232388	2.045602	-0.944800
H	3.117057	2.323466	0.808729

endo-dist-anti-TS1

M06 SCF energy in solution: -572.61288792 a.u.
M06 enthalpy in solution: -572.399070 a.u.
M06 free energy in solution: -572.450461 a.u.
Imaginary frequency: -353.7241 cm-1

Cartesian coordinates

ATOM	X	Y	Z
C	1.885935	1.037259	-0.085699
C	1.532473	0.247892	-1.170179
C	1.769398	-1.149866	-1.263167
C	2.416502	-1.888465	-0.326797
H	0.879063	0.697399	-1.921378
H	1.284764	-1.671011	-2.090621

H	2.683613	0.687978	0.572148
H	3.002263	-1.440734	0.470385
O	1.046924	-0.889146	1.584553
N	0.580532	0.179084	1.161221
C	-0.730706	0.031439	0.527991
C	-1.305177	-1.223100	0.347569
N	-1.332699	1.184695	0.247227
C	-2.585836	-1.273150	-0.186137
H	-0.740906	-2.111219	0.618593
C	-2.563141	1.116371	-0.260038
C	-3.233352	-0.081755	-0.497421
H	-3.074840	-2.230661	-0.356459
H	-3.046151	2.068523	-0.485864
H	-4.238700	-0.074965	-0.910925
H	2.451820	-2.973261	-0.400086
C	1.637859	2.506981	-0.063806
H	1.441121	2.851586	0.957984
H	2.523237	3.043908	-0.432429
H	0.780748	2.777532	-0.690807

endo-dist-syn-TS1

M06 SCF energy in solution: -572.60971942 a.u.
M06 enthalpy in solution: -572.395662 a.u.
M06 free energy in solution: -572.446459 a.u.
Imaginary frequency: -372.5052 cm-1

Cartesian coordinates

ATOM	X	Y	Z
C	1.854870	1.051859	-0.111222
C	1.549636	0.208430	-1.173328
C	1.834488	-1.181214	-1.196319
C	2.501491	-1.846236	-0.219353
H	0.902502	0.606775	-1.958152
H	1.376098	-1.759988	-1.999692
H	2.655260	0.758286	0.570096
H	3.064818	-1.337495	0.556546
O	1.068279	-0.805441	1.611578
N	0.573474	0.222765	1.133278
C	-0.739349	0.021208	0.505182
C	-1.491666	1.171274	0.268267
N	-1.181561	-1.214644	0.308548
C	-2.769811	1.018138	-0.247816
H	-1.078855	2.150866	0.498376
C	-2.415533	-1.343913	-0.184128
C	-3.245043	-0.268502	-0.486924

H	-3.390145	1.888110	-0.453061
H	-2.766495	-2.365014	-0.344430
H	-4.241183	-0.438018	-0.887936
H	2.576172	-2.931339	-0.235489
C	1.589398	2.518339	-0.177897
H	2.489139	3.046632	-0.522116
H	0.778691	2.753339	-0.879285
H	1.333479	2.918227	0.810965

exo-prox-anti-TS1

M06 SCF energy in solution: -572.60386615 a.u.
M06 enthalpy in solution: -572.390079 a.u.
M06 free energy in solution: -572.441249 a.u.
Imaginary frequency: -396.6232 cm-1

Cartesian coordinates

ATOM	X	Y	Z
C	0.993336	-1.879375	-0.491387
C	2.353650	-1.654608	-0.363021
C	2.902921	-0.365511	-0.375638
C	2.151925	0.783486	-0.522210
H	2.992013	-2.469634	-0.020606
H	3.947844	-0.250800	-0.079404
H	0.409747	-1.269995	-1.178448
H	1.173754	0.735090	-1.002082
H	0.582341	-2.871112	-0.319781
O	1.010649	0.201213	1.288206
N	0.199699	-0.722884	1.014538
C	-1.037310	-0.234635	0.441203
C	-1.306847	1.128815	0.326438
N	-1.908939	-1.195243	0.124842
C	-2.550735	1.506727	-0.162242
H	-0.554440	1.852611	0.627573
C	-3.096414	-0.812279	-0.339001
C	-3.469141	0.520614	-0.505165
H	-2.801583	2.560243	-0.271656
H	-3.795750	-1.610754	-0.593086
H	-4.454627	0.771721	-0.889129
C	2.703061	2.144993	-0.308165
H	2.745044	2.708435	-1.251556
H	2.057273	2.719284	0.370319
H	3.711195	2.112729	0.121682

exo-prox-syn-TS1

M06 SCF energy in solution: -572.60146634 a.u.
M06 enthalpy in solution: -572.387671 a.u.
M06 free energy in solution: -572.438557 a.u.
Imaginary frequency: -402.8394 cm-1

Cartesian coordinates

ATOM	X	Y	Z
C	0.978781	-1.852937	-0.655330
C	2.337620	-1.636504	-0.513099
C	2.873348	-0.349992	-0.409105
C	2.084282	0.793003	-0.427752
H	2.979667	-2.476002	-0.244159
H	3.913564	-0.246401	-0.093741
H	0.384405	-1.177515	-1.267078
H	1.128751	0.786510	-0.951428
H	0.567712	-2.855461	-0.554375
O	1.074918	0.148253	1.258885
N	0.222265	-0.748531	1.001248
C	-1.004421	-0.226028	0.441001
C	-2.048400	-1.145640	0.311069
N	-1.111693	1.066129	0.136188
C	-3.267775	-0.692200	-0.163756
H	-1.882554	-2.185253	0.587867
C	-2.295050	1.484796	-0.322599
C	-3.399102	0.656629	-0.490233
H	-4.106392	-1.376339	-0.276340
H	-2.366457	2.545727	-0.569298
H	-4.336736	1.059786	-0.864724
C	2.601686	2.138553	-0.075676
H	2.816081	2.725549	-0.980676
H	1.842697	2.690270	0.492999
H	3.520097	2.081839	0.520689

exo-dist-anti-TS1

M06 SCF energy in solution: -572.60055069 a.u.
M06 enthalpy in solution: -572.386827 a.u.
M06 free energy in solution: -572.437529 a.u.
Imaginary frequency: -405.8353 cm-1

Cartesian coordinates

ATOM	X	Y	Z
C	-1.604956	1.062354	-0.405752
C	-2.805204	0.363345	-0.348142
C	-2.882227	-1.023039	-0.485707

C	-1.764866	-1.818532	-0.672935
H	-3.683572	0.886286	0.036750
H	-3.824628	-1.509196	-0.232412
H	-0.848808	0.712844	-1.110726
H	-0.875251	-1.456608	-1.184497
O	-0.958595	-1.172663	1.132581
N	-0.460927	-0.013945	1.005552
C	0.868431	-0.049758	0.443284
C	1.525703	-1.251893	0.172275
N	1.434363	1.150848	0.286268
C	2.827533	-1.191078	-0.306544
H	1.020701	-2.196633	0.351820
C	2.683802	1.187411	-0.171222
C	3.425833	0.051138	-0.486328
H	3.371847	-2.106062	-0.533490
H	3.124179	2.178766	-0.292705
H	4.444051	0.143538	-0.855589
H	-1.844841	-2.900632	-0.600003
C	-1.526660	2.506692	-0.041488
H	-0.514285	2.758119	0.290863
H	-1.760041	3.136423	-0.912568
H	-2.238976	2.756291	0.754523

exo-dist-syn-TS1

M06 SCF energy in solution: -572.59856109 a.u.
M06 enthalpy in solution: -572.384785 a.u.
M06 free energy in solution: -572.435814 a.u.
Imaginary frequency: -453.1178 cm-1

Cartesian coordinates

ATOM	X	Y	Z
C	-1.526853	1.111457	-0.558212
C	-2.757152	0.514641	-0.369834
C	-2.928547	-0.870712	-0.345153
C	-1.846430	-1.747722	-0.432105
H	-3.590430	1.141707	-0.044841
H	-3.890918	-1.263364	-0.017762
H	-0.774874	0.574204	-1.135456
H	-0.969385	-1.535090	-1.038424
O	-1.037391	-1.192584	1.181800
N	-0.431687	-0.065026	1.133529
C	0.852561	-0.136623	0.512335
C	1.682258	0.971746	0.723437
N	1.225069	-1.209722	-0.191428
C	2.954390	0.959594	0.175130

H	1.322437	1.797947	1.333413
C	2.450283	-1.195993	-0.721840
C	3.352253	-0.147966	-0.570897
H	3.630940	1.797724	0.329835
H	2.733195	-2.078815	-1.298586
H	4.340741	-0.205019	-1.019614
H	-2.009663	-2.806598	-0.242821
C	-1.295289	2.569227	-0.388228
H	-0.306245	2.763000	0.048422
H	-1.308418	3.080982	-1.361941
H	-2.056775	3.031338	0.250880

endo-prox-anti-TS1-Ph

M06 SCF energy in solution: -764.25164950 a.u.
M06 enthalpy in solution: -763.981505 a.u.
M06 free energy in solution: -764.040760 a.u.
Imaginary frequency: -331.3924 cm-1

Cartesian coordinates

ATOM	X	Y	Z
C	-1.426972	-2.576730	0.495192
C	-0.998387	-1.654798	1.426207
C	0.237403	-0.962181	1.380554
C	1.200827	-1.155246	0.430670
H	-1.730941	-1.293135	2.149243
H	0.360474	-0.143600	2.092159
H	-0.731993	-3.146160	-0.118084
H	1.160967	-2.048054	-0.193232
H	-2.426730	-2.995738	0.576760
O	-0.451574	-1.019144	-1.505316
N	-1.588181	-1.247562	-1.069872
C	-2.273599	-0.052601	-0.581573
C	-1.645594	1.189031	-0.577754
N	-3.542848	-0.260395	-0.238577
C	-2.395102	2.286390	-0.174183
H	-0.604858	1.261047	-0.882851
C	-4.247218	0.804916	0.139082
C	-3.724193	2.096235	0.190339
H	-1.949032	3.278757	-0.143375
H	-5.286906	0.623703	0.416627
H	-4.347759	2.928410	0.507251
C	2.372909	-0.316279	0.235258
C	3.437565	-0.803353	-0.538942
C	2.481242	0.974740	0.778937
C	4.580497	-0.041304	-0.743604

H	3.358964	-1.798926	-0.976497
C	3.623697	1.735128	0.575379
H	1.651703	1.393450	1.348108
C	4.679676	1.229942	-0.183510
H	5.396863	-0.439528	-1.343550
H	3.690083	2.734282	1.002611
H	5.572465	1.831007	-0.345799

endo-prox-syn-TS1-Ph

M06 SCF energy in solution: -764.24818180 a.u.
M06 enthalpy in solution: -763.978013 a.u.
M06 free energy in solution: -764.036881 a.u.
Imaginary frequency: -359.5637 cm-1

Cartesian coordinates

ATOM	X	Y	Z
C	-1.433865	-2.584801	0.472335
C	-0.993688	-1.686318	1.425325
C	0.242752	-0.999883	1.380996
C	1.204400	-1.187080	0.427311
H	-1.719313	-1.336258	2.161005
H	0.368956	-0.182432	2.093301
H	-0.744388	-3.155059	-0.146387
H	1.178775	-2.084183	-0.190984
H	-2.429268	-3.015154	0.562623
O	-0.433349	-1.050458	-1.492635
N	-1.571284	-1.245914	-1.055215
C	-2.237639	-0.036091	-0.563884
C	-3.591845	-0.163944	-0.259523
N	-1.564771	1.107592	-0.533829
C	-4.282270	0.976141	0.120681
H	-4.072548	-1.138353	-0.327020
C	-2.244852	2.196098	-0.164414
C	-3.594206	2.186724	0.174330
H	-5.340577	0.926360	0.368150
H	-1.682155	3.131085	-0.141120
H	-4.093953	3.107478	0.464758
C	2.361487	-0.328590	0.229815
C	3.450901	-0.807379	-0.514526
C	2.427617	0.978482	0.740796
C	4.577675	-0.021711	-0.719515
H	3.404873	-1.815239	-0.927992
C	3.554057	1.762412	0.537103
H	1.576797	1.390521	1.282583
C	4.635454	1.265535	-0.190665

H	5.414127	-0.413732	-1.295501
H	3.586719	2.774105	0.937991
H	5.515261	1.885216	-0.354004

endo-dist-anti-TS1-Ph

M06 SCF energy in solution: -764.24603617 a.u.
M06 enthalpy in solution: -763.976067 a.u.
M06 free energy in solution: -764.034884 a.u.
Imaginary frequency: -359.1265 cm-1

Cartesian coordinates

ATOM	X	Y	Z
C	0.604397	1.343772	0.223567
C	-0.288898	1.327870	1.298890
C	-1.440530	2.141598	1.374024
C	-1.778460	3.091282	0.458588
H	-0.210258	0.517295	2.024153
H	-2.166765	1.887123	2.147574
H	0.663459	2.256946	-0.372760
H	-1.067807	3.501116	-0.251471
O	-1.395457	1.604649	-1.465363
N	-0.674859	0.670084	-1.071501
C	-1.422697	-0.471997	-0.553211
C	-2.811673	-0.436162	-0.467849
N	-0.677434	-1.542257	-0.288118
C	-3.459744	-1.591410	-0.051578
H	-3.339966	0.478083	-0.723280
C	-1.317979	-2.640848	0.108844
C	-2.702442	-2.719979	0.245867
H	-4.544178	-1.612470	0.039580
H	-0.696026	-3.510611	0.327310
H	-3.169483	-3.646190	0.571297
H	-2.751808	3.575564	0.497585
C	1.827008	0.524950	0.156945
C	2.610752	0.607680	-1.000012
C	2.249337	-0.315072	1.191829
C	3.778982	-0.132115	-1.127816
H	2.283564	1.259617	-1.810871
C	3.420768	-1.053576	1.068344
H	1.668752	-0.389427	2.109612
C	4.188317	-0.967427	-0.090616
H	4.373186	-0.056179	-2.036816
H	3.738199	-1.698534	1.886004
H	5.106020	-1.545705	-0.182592

endo-dist-syn-TS1-Ph

M06 SCF energy in solution: -764.24469838 a.u.
M06 enthalpy in solution: -763.974707 a.u.
M06 free energy in solution: -764.033511 a.u.
Imaginary frequency: -363.8548 cm-1

Cartesian coordinates

ATOM	X	Y	Z
C	-0.491016	-1.455485	0.169857
C	0.404099	-1.448800	1.247246
C	1.588924	-2.213282	1.280996
C	1.959755	-3.099548	0.316499
H	0.294113	-0.671431	2.004133
H	2.308546	-1.965876	2.062539
H	-0.528881	-2.349769	-0.456880
H	1.263971	-3.495883	-0.415366
O	1.517079	-1.508673	-1.508181
N	0.718688	-0.660363	-1.080092
C	1.340599	0.547396	-0.529626
C	0.510941	1.660467	-0.380486
N	2.651560	0.558434	-0.322320
C	1.089223	2.844647	0.052797
H	-0.549573	1.590212	-0.613797
C	3.190261	1.708343	0.088703
C	2.459231	2.873197	0.299656
H	0.481683	3.737692	0.184104
H	4.268895	1.702818	0.256014
H	2.955269	3.781175	0.633408
H	2.951898	-3.545443	0.325851
C	-1.739160	-0.669424	0.157178
C	-2.484208	-0.633207	-1.028244
C	-2.191726	0.066518	1.256727
C	-3.637526	0.133633	-1.122031
H	-2.132200	-1.203317	-1.888493
C	-3.349316	0.833309	1.165337
H	-1.646798	0.038503	2.198665
C	-4.071725	0.874332	-0.023454
H	-4.201485	0.154518	-2.052868
H	-3.688082	1.400597	2.030441
H	-4.975864	1.476525	-0.092349

exo-prox-anti-TS1-Ph

M06 SCF energy in solution: -764.24296184 a.u.

M06 enthalpy in solution: -763.972647 a.u.
M06 free energy in solution: -764.031473 a.u.
Imaginary frequency: -389.7206 cm-1

Cartesian coordinates

ATOM	X	Y	Z
C	1.491967	2.401372	-0.648896
C	0.249981	3.006388	-0.533163
C	-0.943927	2.276502	-0.473446
C	-1.005514	0.897202	-0.548877
H	0.203094	4.064075	-0.273346
H	-1.854227	2.825987	-0.229498
H	1.601219	1.519379	-1.277694
H	-0.160862	0.351337	-0.972843
H	2.399563	2.992050	-0.553912
O	0.372256	0.889771	1.319214
N	1.540835	1.141102	0.948592
C	2.250656	-0.027471	0.468734
C	1.672744	-1.295777	0.496707
N	3.503546	0.216987	0.081443
C	2.451399	-2.370174	0.086026
H	0.647196	-1.409687	0.838838
C	4.233948	-0.826274	-0.306142
C	3.759691	-2.137108	-0.323965
H	2.042454	-3.378831	0.087444
H	5.256594	-0.610329	-0.619882
H	4.403818	-2.949752	-0.650069
C	-2.187715	0.090422	-0.310068
C	-2.178810	-1.260354	-0.696457
C	-3.343478	0.597791	0.308061
C	-3.286525	-2.071215	-0.490563
H	-1.285065	-1.668279	-1.170920
C	-4.449462	-0.213652	0.514024
H	-3.370916	1.633403	0.643253
C	-4.428043	-1.549816	0.114603
H	-3.260335	-3.113869	-0.801879
H	-5.335708	0.196185	0.995386
H	-5.296997	-2.183461	0.281687

exo-prox-syn-TS1-Ph

M06 SCF energy in solution: -764.24024436 a.u.
M06 enthalpy in solution: -763.970050 a.u.
M06 free energy in solution: -764.029429 a.u.
Imaginary frequency: -408.2162 cm-1

Cartesian coordinates

ATOM	X	Y	Z
C	1.481384	2.360675	-0.667142
C	0.242196	2.984930	-0.588103
C	-0.955029	2.267533	-0.526138
C	-1.014701	0.883075	-0.561008
H	0.203691	4.049245	-0.355265
H	-1.863213	2.827470	-0.298755
H	1.587114	1.479135	-1.298606
H	-0.177142	0.321770	-0.973464
H	2.389898	2.952978	-0.576158
O	0.314397	0.876528	1.265578
N	1.494975	1.130888	0.932752
C	2.228061	-0.028743	0.450683
C	3.611895	0.122805	0.361017
N	1.580455	-1.156057	0.170887
C	4.363826	-0.971184	-0.039696
H	4.066170	1.079264	0.613201
C	2.319363	-2.197781	-0.218616
C	3.705231	-2.161706	-0.337673
H	5.446752	-0.899897	-0.116932
H	1.774310	-3.115008	-0.449332
H	4.252273	-3.046365	-0.653757
C	-2.194255	0.081483	-0.291059
C	-2.142577	-1.299208	-0.546730
C	-3.381770	0.617161	0.235418
C	-3.240167	-2.112986	-0.302404
H	-1.217482	-1.725599	-0.935721
C	-4.478178	-0.197342	0.479053
H	-3.446269	1.679550	0.465082
C	-4.414014	-1.564244	0.210044
H	-3.180654	-3.180087	-0.509219
H	-5.390714	0.234582	0.886494
H	-5.275500	-2.199890	0.406449

exo-dist-anti-TS1-Ph

M06 SCF energy in solution: -764.23680846 a.u.
M06 enthalpy in solution: -763.966402 a.u.
M06 free energy in solution: -764.024362 a.u.
Imaginary frequency: -459.1699 cm-1

Cartesian coordinates

ATOM	X	Y	Z
C	0.674277	1.047445	-0.595222
C	0.645351	2.432734	-0.560332

C	-0.532494	3.171880	-0.639319
C	-1.791717	2.565144	-0.670941
H	1.563802	2.969691	-0.318556
H	-0.478712	4.244761	-0.458853
H	-0.118949	0.524267	-1.128436
H	-1.951242	1.620964	-1.191762
O	-1.672106	1.814867	1.044025
N	-0.968969	0.742199	1.093292
C	-1.666085	-0.385706	0.573221
C	-3.013520	-0.340832	0.193896
N	-0.933858	-1.507672	0.543242
C	-3.605553	-1.509202	-0.265723
H	-3.572954	0.585520	0.290576
C	-1.522849	-2.615417	0.102759
C	-2.849921	-2.674938	-0.321065
H	-4.650668	-1.510879	-0.570159
H	-0.903788	-3.514967	0.086247
H	-3.274290	-3.611742	-0.673458
H	-2.681633	3.186876	-0.600532
C	1.855691	0.247657	-0.308438
C	1.980231	-1.026622	-0.882550
C	2.884810	0.708682	0.527357
C	3.106754	-1.804869	-0.651585
H	1.178987	-1.398741	-1.520685
C	4.008775	-0.071535	0.761287
H	2.791399	1.678124	1.015628
C	4.126293	-1.328799	0.170052
H	3.190692	-2.787813	-1.111969
H	4.795843	0.298714	1.415960
H	5.007888	-1.939134	0.357675

exo-dist-syn-TS1-Ph

M06 SCF energy in solution: -764.23873116 a.u.
M06 enthalpy in solution: -763.968190 a.u.
M06 free energy in solution: -764.026620 a.u.
Imaginary frequency: -468.3366 cm-1

Cartesian coordinates

ATOM	X	Y	Z
C	0.533932	1.182294	-0.614943
C	0.397421	2.552798	-0.505331
C	-0.844655	3.188495	-0.549365
C	-2.048222	2.475661	-0.594637
H	1.268303	3.155874	-0.243517
H	-0.882513	4.253836	-0.325163

H	-0.255050	0.633890	-1.129681
H	-2.150427	1.532935	-1.129041
O	-1.899172	1.717425	1.088931
N	-1.095707	0.724445	1.190182
C	-1.581596	-0.479169	0.619995
C	-0.795731	-1.613410	0.871892
N	-2.717836	-0.511985	-0.087250
C	-1.214764	-2.832526	0.366397
H	0.111474	-1.509422	1.463102
C	-3.096469	-1.695961	-0.572627
C	-2.392582	-2.880579	-0.377416
H	-0.634247	-3.734821	0.548942
H	-4.022763	-1.703421	-1.150565
H	-2.763434	-3.814350	-0.792660
H	-2.982350	3.024283	-0.494682
C	1.741970	0.419122	-0.367333
C	1.854025	-0.871318	-0.911033
C	2.795033	0.904375	0.426977
C	2.987662	-1.643734	-0.688609
H	1.039615	-1.259393	-1.523470
C	3.925528	0.132334	0.647123
H	2.714650	1.886463	0.891316
C	4.027999	-1.143083	0.089139
H	3.059866	-2.638861	-1.123835
H	4.731853	0.520892	1.266673
H	4.915899	-1.746324	0.269245

endo-prox-syn-TS2

M06 SCF energy in solution: -1035.33327115 a.u.
M06 enthalpy in solution: -1035.011050 a.u.
M06 free energy in solution: -1035.090942 a.u.
Imaginary frequency: -316.7743 cm-1

Cartesian coordinates

ATOM	X	Y	Z
C	2.532115	1.191812	-2.194991
C	2.138040	1.996273	-1.147364
C	0.812138	2.471239	-0.957165
C	-0.219564	2.217042	-1.808347
H	2.850395	2.167747	-0.339354
H	0.596236	3.002546	-0.027652
H	1.966859	1.142432	-3.122397
H	-0.016922	1.748438	-2.770565
H	3.565767	0.863987	-2.270728
O	0.332418	-0.341748	-1.657620

N	1.566185	-0.506203	-1.545587
C	2.014407	-0.781138	-0.192263
C	3.368085	-1.043110	0.003654
N	1.101967	-0.906480	0.772152
C	3.788422	-1.411604	1.269936
H	4.060961	-0.954590	-0.829635
C	1.515302	-1.262719	1.998128
C	2.845162	-1.517483	2.292266
H	4.838327	-1.619074	1.462192
H	0.740634	-1.353488	2.758304
H	3.132018	-1.807288	3.299028
C	-1.622994	2.632382	-1.572032
H	-1.950781	3.332759	-2.354180
H	-2.303907	1.770722	-1.634022
H	-1.753391	3.113060	-0.595475
Cu	-0.823172	-0.469524	0.287023
N	-1.547539	0.971128	1.446949
N	-2.217486	-1.685513	-0.396054
C	-1.950652	1.789696	2.158996
C	-3.039980	-2.375147	-0.828385
C	-2.457018	2.821558	3.035776
H	-1.633137	3.280293	3.591788
H	-2.961377	3.594513	2.446225
H	-3.172240	2.394348	3.745983
C	-4.067721	-3.237864	-1.366398
H	-4.810492	-2.642600	-1.907111
H	-3.625433	-3.965111	-2.054774
H	-4.567194	-3.774669	-0.553422

endo-dist-syn-TS2

M06 SCF energy in solution: -1035.33419948 a.u.
M06 enthalpy in solution: -1035.012737 a.u.
M06 free energy in solution: -1035.093454 a.u.
Imaginary frequency: -368.4719 cm-1

Cartesian coordinates

ATOM	X	Y	Z
C	-2.488769	-1.565202	1.283694
C	-1.814812	-0.511116	1.889042
C	-0.426175	-0.457403	2.181833
C	0.530864	-1.418132	1.910814
H	-2.383579	0.404386	2.067759
H	-0.080423	0.464438	2.653599
H	-2.017441	-2.549367	1.275177
H	0.261875	-2.417391	1.571248

O	-0.647459	-1.756756	-0.641968
N	-1.798845	-1.275723	-0.561649
C	-1.826460	0.127356	-0.929454
C	-3.016895	0.668516	-1.392087
N	-0.682132	0.812751	-0.837925
C	-3.010557	2.002711	-1.782097
H	-3.905902	0.045976	-1.459045
C	-0.687702	2.096826	-1.204993
C	-1.828604	2.730017	-1.686024
H	-3.917520	2.467524	-2.161080
H	0.256263	2.630134	-1.098944
H	-1.785602	3.775715	-1.976773
Cu	0.812555	-0.293778	0.140859
N	1.958300	1.427572	0.831955
N	2.393750	-1.221507	-0.809677
C	-3.973502	-1.552509	1.152758
H	-4.296683	-2.144435	0.289093
H	-4.430267	-1.993485	2.049124
H	-4.359332	-0.529597	1.051461
H	1.537094	-1.298915	2.313445
C	2.544918	2.377815	1.139695
C	3.262281	-1.815612	-1.291094
C	3.278625	3.564812	1.522644
H	2.653403	4.204123	2.154105
H	4.178320	3.285613	2.080214
H	3.573428	4.126377	0.630322
C	4.348008	-2.556849	-1.894222
H	4.936492	-1.899461	-2.542142
H	5.000822	-2.964802	-1.115882
H	3.949820	-3.382254	-2.492825

endo-prox-syn-TS3

M06 SCF energy in solution: -1285.93372160 a.u.
M06 enthalpy in solution: -1285.466630 a.u.
M06 free energy in solution: -1285.552822 a.u.
Imaginary frequency: -322.0055 cm-1

Cartesian coordinates

ATOM	X	Y	Z
C	3.508170	-0.155439	2.344923
C	2.866531	-1.324196	1.992780
C	1.477024	-1.569316	2.160387
C	0.597901	-0.696473	2.727333
H	3.432626	-2.063101	1.423957
H	1.080985	-2.496501	1.740023

H	3.089754	0.528980	3.079192
H	0.973876	0.199396	3.220506
H	4.578033	-0.052779	2.181112
O	1.496597	1.206557	1.129284
N	2.697626	1.003452	0.855591
C	2.908894	0.329235	-0.412802
C	4.219842	0.155458	-0.847323
N	1.846319	0.021591	-1.160156
C	4.428489	-0.401201	-2.097631
H	5.044822	0.456604	-0.206122
C	2.054783	-0.524440	-2.370078
C	3.324293	-0.757397	-2.872742
H	5.438281	-0.554325	-2.470403
H	1.163279	-0.768227	-2.945701
H	3.443854	-1.195214	-3.859630
C	-0.860713	-0.944068	2.843855
H	-1.132539	-1.101962	3.898579
H	-1.438244	-0.070492	2.509420
H	-1.178119	-1.820212	2.264729
Cu	0.048622	0.296521	-0.377886
N	-1.235046	-1.547355	-0.495728
N	-1.750303	1.165285	-0.285783
C	-2.743990	0.356473	-0.330736
C	-2.470010	-1.060235	-0.438005
H	-3.786904	0.685940	-0.288457
C	-1.927297	2.625549	-0.120962
C	-3.348858	3.102958	-0.386265
H	-3.377717	4.196834	-0.312453
H	-3.686773	2.826050	-1.394415
H	-4.066238	2.712650	0.347858
C	-1.524769	2.949928	1.316825
H	-0.510917	2.588076	1.533954
H	-1.546226	4.036874	1.471536
H	-2.224265	2.488363	2.027588
C	-0.967541	3.295658	-1.099457
H	-1.025940	4.386865	-0.995290
H	0.068905	2.984973	-0.904932
H	-1.219948	3.032745	-2.135690
C	-1.370574	-2.897866	-0.572809
H	-0.513459	-3.558100	-0.629335
C	-2.705708	-3.233056	-0.553978
H	-3.215775	-4.187081	-0.593731
N	-3.396714	-2.056897	-0.466949
C	-4.844643	-1.911072	-0.433354
H	-5.291167	-2.904716	-0.359527
H	-5.145524	-1.324113	0.439581
H	-5.201592	-1.424626	-1.346517

endo-dist-syn-TS3

M06 SCF energy in solution: -1285.93706106 a.u.
M06 enthalpy in solution: -1285.470061 a.u.
M06 free energy in solution: -1285.554354 a.u.
Imaginary frequency: -349.8836 cm-1

Cartesian coordinates

ATOM	X	Y	Z
C	-3.354139	0.649071	-1.736282
C	-2.552725	-0.463377	-1.957772
C	-1.157775	-0.463471	-2.219627
C	-0.311075	0.629881	-2.314686
H	-3.015347	-1.442631	-1.811483
H	-0.701800	-1.448886	-2.333876
H	-2.989786	1.627519	-2.053487
H	-0.699618	1.646111	-2.369710
O	-1.593765	1.672979	0.004358
N	-2.693713	1.077546	0.113441
C	-2.574842	-0.121351	0.914855
C	-3.707850	-0.625632	1.537652
N	-1.359360	-0.669187	1.027867
C	-3.560999	-1.762861	2.322946
H	-4.663881	-0.121356	1.418081
C	-1.228817	-1.767752	1.775236
C	-2.302945	-2.345907	2.443117
H	-4.418908	-2.189770	2.836781
H	-0.228412	-2.196351	1.822924
H	-2.150524	-3.239718	3.041316
Cu	-0.000389	0.227144	-0.284385
N	1.442971	-1.546214	-0.466538
N	1.834886	1.114104	0.271941
C	2.847614	0.337031	0.190986
C	2.642385	-1.040477	-0.207310
H	3.870499	0.656843	0.418369
C	1.958452	2.518743	0.712040
C	-4.831913	0.517248	-1.595596
H	-5.235567	1.303505	-0.947696
H	-5.311688	0.617607	-2.578700
H	-5.111503	-0.462266	-1.186235
H	0.712854	0.486188	-2.663016
C	1.366683	3.382437	-0.399933
H	0.311501	3.136153	-0.570674
H	1.431124	4.442444	-0.121193
H	1.918837	3.238734	-1.339151

C	1.115128	2.643461	1.980090
H	1.538021	2.031431	2.788301
H	1.094869	3.689369	2.313991
H	0.083688	2.315875	1.794415
C	3.388265	2.958469	0.995832
H	3.850583	2.371849	1.801418
H	4.022899	2.894110	0.101157
H	3.379306	4.006692	1.318500
C	1.649479	-2.851634	-0.786976
H	0.831358	-3.514476	-1.044594
C	2.993530	-3.139702	-0.720793
H	3.551548	-4.049509	-0.901411
N	3.616953	-1.980504	-0.350271
C	5.048197	-1.797011	-0.159912
H	5.544907	-2.755074	-0.326506
H	5.437461	-1.065340	-0.874661
H	5.256913	-1.463280	0.861169

pentadiene

M06 SCF energy in solution: -195.19653665 a.u.
M06 enthalpy in solution: -195.076568 a.u.
M06 free energy in solution: -195.112719 a.u.

Cartesian coordinates

ATOM	X	Y	Z
C	2.411320	0.774267	0.000000
C	1.405667	-0.108571	0.000000
H	2.221097	1.848289	0.000000
H	3.452990	0.461245	0.000000
H	1.628530	-1.179672	0.000000
C	0.000000	0.252126	0.000000
C	-1.000671	-0.639865	0.000000
H	-0.234999	1.321915	0.000000
H	-0.746196	-1.704298	0.000000
C	-2.449184	-0.296245	0.000000
H	-2.960319	-0.713915	0.879331
H	-2.603570	0.790074	0.000000
H	-2.960319	-0.713915	-0.879331

phenylbutadiene

M06 SCF energy in solution: -386.83419868 a.u.
M06 enthalpy in solution: -386.657746 a.u.
M06 free energy in solution: -386.702280 a.u.

Cartesian coordinates

ATOM	X	Y	Z
C	-4.416021	0.429075	0.000004
C	-3.329123	-0.353896	-0.000003
H	-4.329962	1.516060	0.000011
H	-5.422075	0.016118	0.000005
H	-3.445470	-1.441295	-0.000008
C	-1.970913	0.144572	0.000000
C	-0.892473	-0.661963	-0.000002
H	-1.853855	1.231973	0.000005
H	-1.069448	-1.742118	0.000002
C	0.511417	-0.266407	-0.000004
C	1.493733	-1.267776	-0.000007
C	0.933557	1.073086	0.000001
C	2.846713	-0.949718	0.000010
H	1.180998	-2.312524	-0.000008
C	2.284295	1.391402	-0.000005
H	0.195915	1.874990	-0.000004
C	3.248354	0.382914	0.000002
H	3.589917	-1.745350	0.000015
H	2.590142	2.436466	-0.000006
H	4.306601	0.637941	0.000004

PyrNO

M06 SCF energy in solution: -377.42349414 a.u.
M06 enthalpy in solution: -377.331134 a.u.
M06 free energy in solution: -377.368723 a.u.

Cartesian coordinates

ATOM	X	Y	Z
C	1.632223	-1.006007	0.000000
C	0.690334	-2.038066	0.000000
C	-0.662520	-1.714856	0.000000
C	-1.024711	-0.374913	0.000000
C	0.000000	0.567400	0.000000
N	1.303015	0.281474	0.000000
H	-1.420422	-2.494882	0.000000
H	2.697476	-1.238362	0.000000
H	1.018777	-3.074234	0.000000
H	-2.060502	-0.046697	0.000000
N	-0.250790	1.991769	0.000000
O	-1.426608	2.292515	0.000000

PyrNO-CuAN2-anti

M06 SCF energy in solution: -840.13699827 a.u.
M06 enthalpy in solution: -839.936839 a.u.
M06 free energy in solution: -840.007237 a.u.

Cartesian coordinates

ATOM	X	Y	Z
C	-2.389442	-0.000253	-1.846706
C	-3.769557	-0.000262	-1.613807
C	-4.240395	-0.000235	-0.306227
C	-3.322255	-0.000181	0.737805
C	-1.980928	-0.000151	0.383879
N	-1.501755	-0.000182	-0.860490
H	-5.307766	-0.000251	-0.101200
H	-1.997237	-0.000244	-2.862081
H	-4.457206	-0.000288	-2.455066
H	-3.615171	-0.000158	1.783935
N	-0.911865	-0.000094	1.341405
O	-1.210954	-0.000035	2.515031
Cu	0.812327	-0.000043	0.420519
N	1.823085	-1.619809	-0.033965
C	2.417930	-2.573596	-0.304125
C	3.163466	-3.764118	-0.640979
H	3.785297	-4.063983	0.208684
H	2.475123	-4.579476	-0.884882
H	3.807914	-3.568187	-1.503847
C	3.162374	3.764781	-0.640964
H	2.473816	4.579936	-0.884932
H	3.784046	4.064837	0.208749
H	3.806961	3.569030	-1.503768
C	2.417164	2.574051	-0.304127
N	1.822567	1.620098	-0.034004

PyrNO-CuAN2-syn

M06 SCF energy in solution: -840.13959538 a.u.
M06 enthalpy in solution: -839.939607 a.u.
M06 free energy in solution: -840.008273 a.u.

Cartesian coordinates

ATOM	X	Y	Z
C	1.907537	0.043524	-1.473399
C	3.301327	0.077445	-1.603683
C	4.096620	0.036761	-0.468782
C	3.470890	-0.039208	0.772925

C	2.084378	-0.065570	0.801193
N	1.303764	-0.025310	-0.290890
H	5.179897	0.062494	-0.545337
H	1.265401	0.072109	-2.351205
H	3.740183	0.136184	-2.595633
H	4.020916	-0.076256	1.709565
N	1.489982	-0.141506	2.094312
O	0.268811	-0.145053	2.099188
Cu	-0.704339	-0.031341	0.068766
N	-1.654847	1.684593	-0.009503
C	-2.213175	2.697088	-0.038331
C	-2.910627	3.961957	-0.075901
H	-3.904066	3.828304	-0.515952
H	-3.020541	4.357380	0.939025
H	-2.346631	4.680890	-0.678732
C	-3.280835	-3.760684	-0.360780
H	-2.732095	-4.560603	-0.868106
H	-3.576782	-4.107142	0.634742
H	-4.180880	-3.523391	-0.937238
C	-2.448935	-2.585203	-0.243296
N	-1.786684	-1.641893	-0.147732

PyNO-Cu5-16

M06 SCF energy in solution: -1090.73886967 a.u.
M06 enthalpy in solution: -1090.393541 a.u.
M06 free energy in solution: -1090.469088 a.u.

Cartesian coordinates

ATOM	X	Y	Z
O	-1.264149	-0.558371	-2.079901
N	-2.483603	-0.643424	-2.000366
C	-2.993853	-0.602489	-0.675248
C	-4.374490	-0.670869	-0.542269
N	-2.140749	-0.509077	0.361492
C	-4.911307	-0.637472	0.739986
H	-4.986539	-0.744122	-1.437415
C	-2.663368	-0.477567	1.588045
C	-4.039717	-0.540117	1.816734
H	-5.985418	-0.687548	0.894446
H	-1.961428	-0.403004	2.416017
H	-4.409546	-0.513108	2.837678
Cu	-0.227182	-0.385407	-0.085740
N	1.467158	-1.502908	0.032306
N	1.121261	1.189352	0.022019
C	2.324868	0.762846	0.098643

C	2.508036	-0.675260	0.109125
H	3.201395	1.415957	0.155235
C	0.771586	2.621600	0.016572
C	1.977696	3.547532	-0.045352
H	1.630857	4.587407	-0.075182
H	2.623430	3.443771	0.837178
H	2.578734	3.370457	-0.947803
C	-0.121855	2.840527	-1.201598
H	-1.022188	2.212689	-1.143968
H	-0.439963	3.889926	-1.250171
H	0.413629	2.595728	-2.128629
C	-0.020480	2.873898	1.297951
H	-0.373161	3.913256	1.320042
H	-0.895935	2.212619	1.354336
H	0.604987	2.699828	2.183380
C	1.976969	-2.764026	0.072244
H	1.345717	-3.641758	0.027746
C	3.345860	-2.695237	0.176607
H	4.104857	-3.463952	0.240091
N	3.670556	-1.366192	0.198780
C	5.013410	-0.811912	0.305655
H	5.249186	-0.212876	-0.578831
H	5.097674	-0.195591	1.205471
H	5.722705	-1.638894	0.373848

X-ray Crystallographic Structure Determination for 28

Experimental

A black block-shaped crystal of dimensions 0.52 x 0.41 x 0.28 mm was selected for structural analysis. Intensity data for this compound were collected using a diffractometer with a Bruker APEX ccd area detector (1) and graphite-monochromated Mo K〈 radiation (⌊ = 0.71073 Å). The sample was cooled to 100(2) K. Cell parameters were determined from a non-linear least squares fit of 5505 peaks in the range 2.40 < ⌉ < 28.07°. A total of 29951 data were measured in the range 1.64 < ⌉ < 26.00° using ⌋ and ⌈ oscillation frames. The data were corrected for absorption by the semi-empirical method (2) giving minimum and maximum transmission factors of 0.7181 and 0.8320. The data were merged to form a set of 7489 independent data with R(int) = 0.0410 and a coverage of 100.0 %.

The monoclinic space group *C*2/*c* was determined by systematic absences and statistical tests and verified by subsequent refinement. The structure was solved by direct methods and refined by full-matrix least-squares methods on F^2 (3). The positions of hydrogens bonded to carbons were initially determined by geometry and refined by a riding model. Non-hydrogen atoms were refined with anisotropic displacement parameters. Hydrogen atom displacement parameters were set to 1.2 times the isotropic equivalent displacement parameters of the bonded atoms. A total of 487 parameters were refined against 7489 data to give wR(F^2) = 0.1249 and S = 1.007 for weights of w = 1/[⌡^{2} (F^2) + (0.0420 P)^2 + 30.0000 P], where P = [F_o^2 + 2F_c^2] / 3. The final R(*F*) was 0.0524 for the 5916 observed, [*F* > 4 ⌡(*F*)], data. The largest shift/s.u. was 0.001 in the final refinement cycle. The final difference map had maxima and minima of 0.729 and -0.752 e/Å^3, respectively.

Selected bond distances (Å) and angles (^{o}) of **28**: Cu(1)-N(1B) 2.024(2), Cu(1)-N(12A) 2.028(3), Cu(1)-N(1A) 2.046(3), Cu(1)-N(12B) 2.046(3); N(1B)-Cu(1)-N(12A) 130.88(11), N(1B)-Cu(1)-N(1A) 125.50(13), N(12A)-Cu(1)-N(1A) 81.26(13), N(1B)-Cu(1)-N(12B) 81.92(11), N(12A)-Cu(1)-N(12B) 114.57(12), N(1A)-Cu(1)-N(12B) 128.87(10).

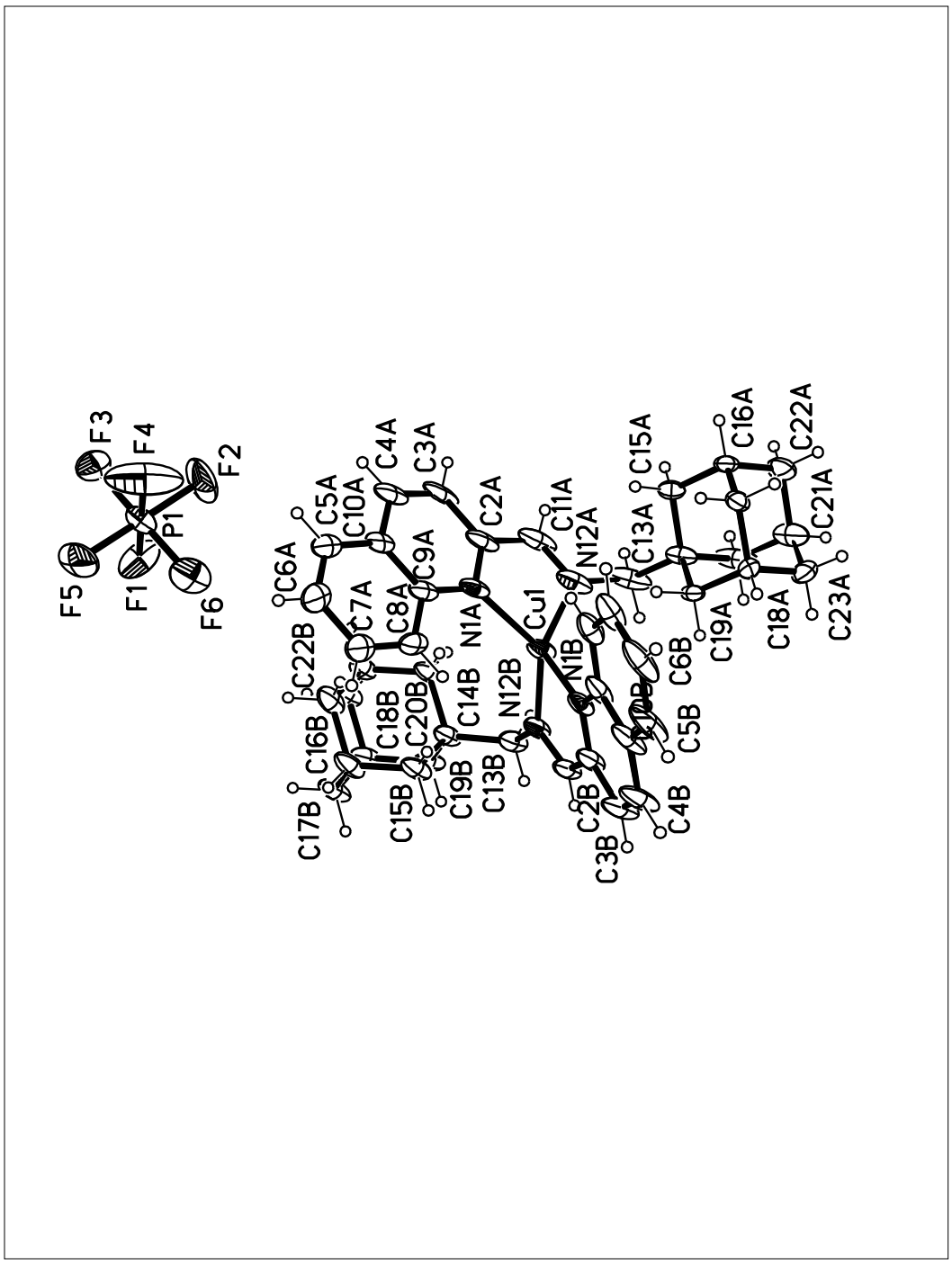

F3
F4
F2
P1
F5
F1
F6
C6A
C5A
C10A
C4A
C3A
C2A
C11A
N12A
C13A
C15A
C16A
C22A
C21A
C23A
C18A
C19A
C6B
C5B
C4B
C3B
C2B
N1B
Cu1
N1A
C9A
C8A
C7A
C14B
N12B
C13B
C19B
C15B
C20B
C18B
C16B
C17B
C22B

Table 1. Crystal data and structure refinement for **28**.

Empirical formula	C_{42} H_{48} Cu F_6 N_4 P	
Formula weight	817.35	
Crystal system	monoclinic	
Space group	*C*2/*c*	
Unit cell dimensions	*a* = 32.815(8) Å	⟨= 90°
	b = 11.058(3) Å	®= 124.773(4)°
	c = 25.545(6) Å	©= 90°
Volume	7614(3) Å^3	
Z, Z'	8, 1	
Density (calculated)	1.426 Mg/m^3	
Wavelength	0.71073 Å	
Temperature	100(2) K	
F(000)	3408	
Absorption coefficient	0.682 mm^{-1}	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.8320 and 0.7181	
Theta range for data collection	1.64 to 26.00°	
Reflections collected	29951	
Independent reflections	7489 [R(int) = 0.0410]	
Data / restraints / parameters	7489 / 0 / 487	
wR(F^2 all data)	*wR*2 = 0.1249	
R(*F* obsd data)	*R*1 = 0.0524	
Goodness-of-fit on F^2	1.007	
Observed data [I > 2 ∫(I)]	5916	
Largest and mean shift / s.u.	0.001and 0.000	
Largest diff. peak and hole	0.729 and -0.752 e/Å^3	

$wR2 = \{ \copyright [w(F_o^2 - F_c^2)^2] / \copyright [w(F_o^2)^2] \}^{1/2}$

$R1 = \copyright ||F_o| - |F_c|| / \copyright |F_o|$

References

(1) (a) Data Collection: SMART Software Reference Manual (2007). Bruker-AXS, 5465 E. Cheryl Parkway, Madison, WI 53711-5373 USA. (b) Data Reduction: SAINT Software Reference Manual (2007). Bruker-AXS, 5465 E. Cheryl Parkway, Madison, WI 53711-5373 USA.

(2) G. M. Sheldrick (2001). SADABS. Program for Empirical Absorption Correction of Area Detector Data. University of Göttingen, Germany.

(3) G. M. Sheldrick (2008). *Acta Cryst., A64*, 112-122.

Acknowledgment

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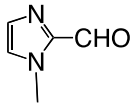
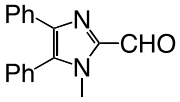
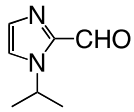
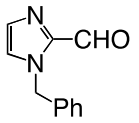
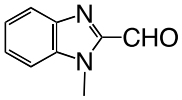
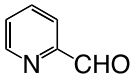
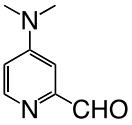
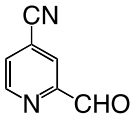
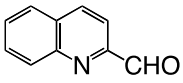
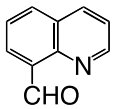
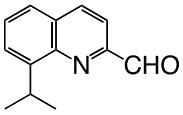
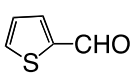
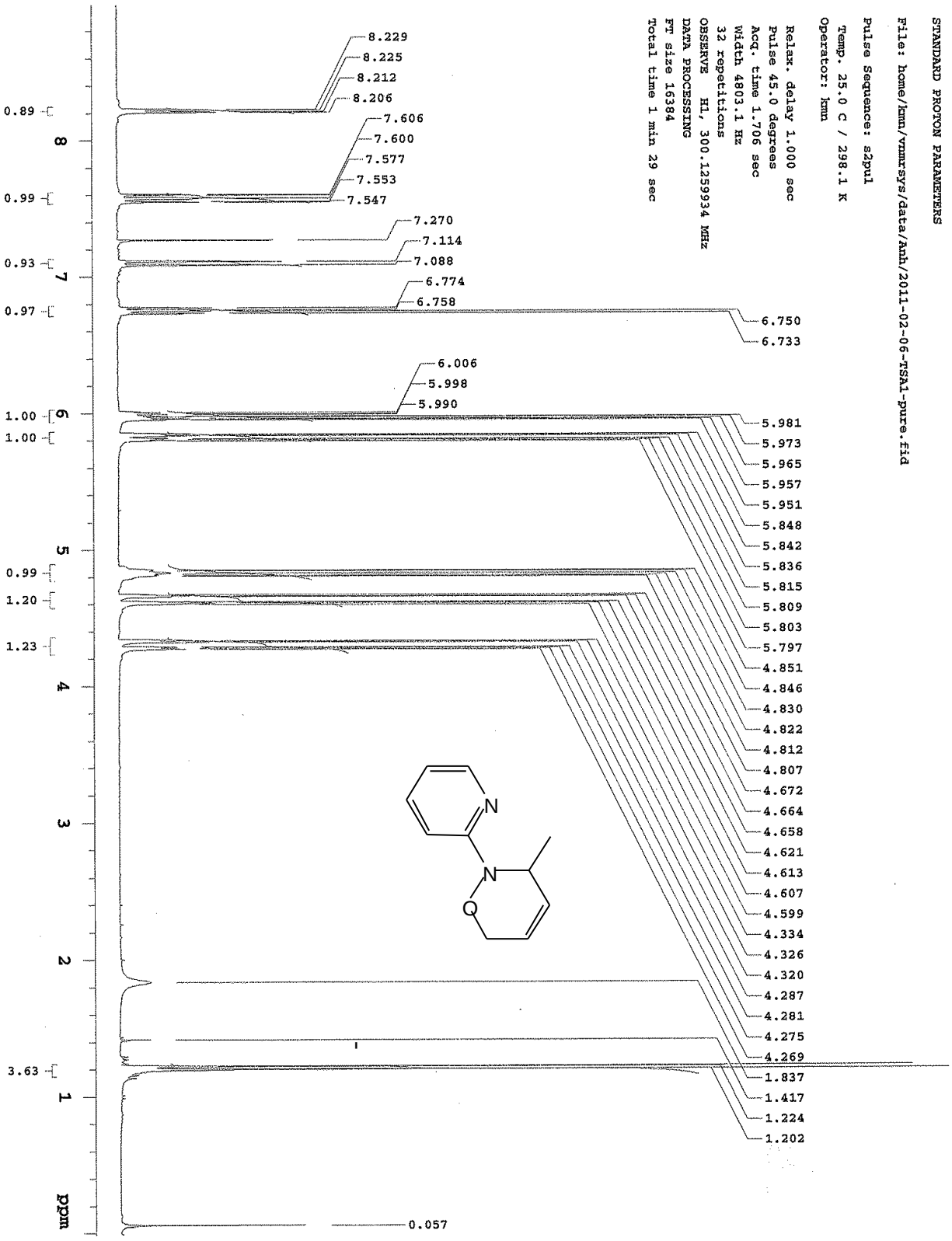
Table S1. Adduct Regioselectivity in PyrNO/pentadiene reactions catalyzed by (imine)Cu^+

Aldehyde	*Amine*	*Dist/Prox Adduct*
	**5**	40:60
	**6**	37:63
	**4** **5** **6** **7**	40:60 38:62 41:59 40:60
	**6** **7**	38:62 37:63
	**4**	40:60
	**5** **7** **11** **12**	45:55 39:61 42:58 40:60
	**5** **6** **7** **13**	37:63 38:62 37:63 38:62
	**7** **14** **15**	35:65 42:58 42:58
	**9**	37:63
	**7** **8** **10**	36:64 37:63 37:63
	**7** **8**	45:55 39:61
	**7** **8**	42:58 40:60


STANDARD PROTON PARAMETERS
File: home/kmn/vnmrsys/data/Anh/2011-02-06-TSA1-pure.fid
Pulse Sequence: s2pul
Temp. 25.0 C / 298.1 K
Operator: kmn
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.706 sec
Width 4803.1 Hz
32 repetitions
OBSERVE H1, 300.1259934 MHz
DATA PROCESSING
FT size 16384
Total time 1 min 29 sec
8.229
8.225
8.212
8.206
7.606
7.600
7.577
7.553
7.547
7.270
7.114
7.088
6.774
6.758
6.750
6.733
6.006
5.998
5.990
5.981
5.973
5.965
5.957
5.951
5.848
5.842
5.836
5.815
5.809
5.803
5.797
4.851
4.846
4.830
4.822
4.812
4.807
4.672
4.664
4.658
4.621
4.613
4.607
4.599
4.334
4.326
4.320
4.287
4.281
4.275
4.269
1.837
1.417
1.224
1.202
0.057
0.89
0.99
0.93
0.97
1.00
1.00
0.99
1.20
1.23
3.63
ppm
N
N
O

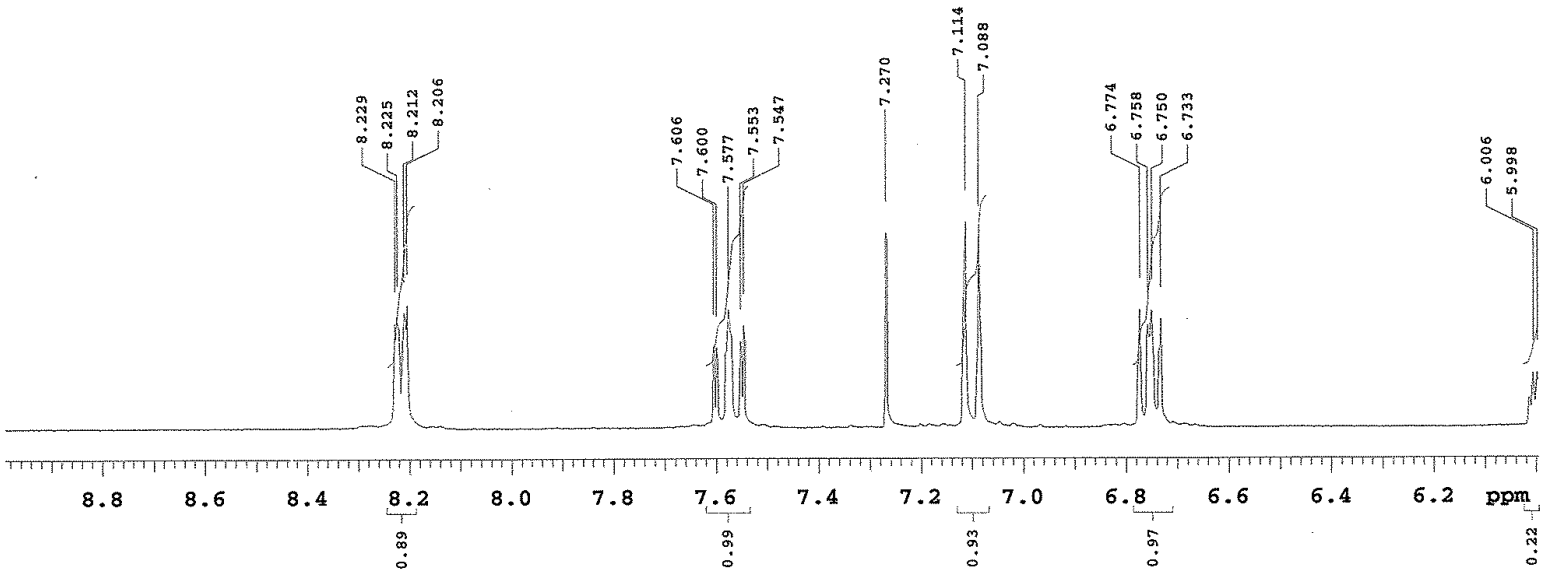
STANDARD PROTON PARAMETERS

File: home/kmn/vnmrsys/data/Anh/2011-02-06-TSA1-pure.fid

Pulse Sequence: s2pul

Temp. 25.0 C / 298.1 K
Operator: kmn

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.706 sec
Width 4803.1 Hz
32 repetitions
OBSERVE H1, 300.1259934 MHz
DATA PROCESSING
FT size 16384
Total time 1 min 29 sec



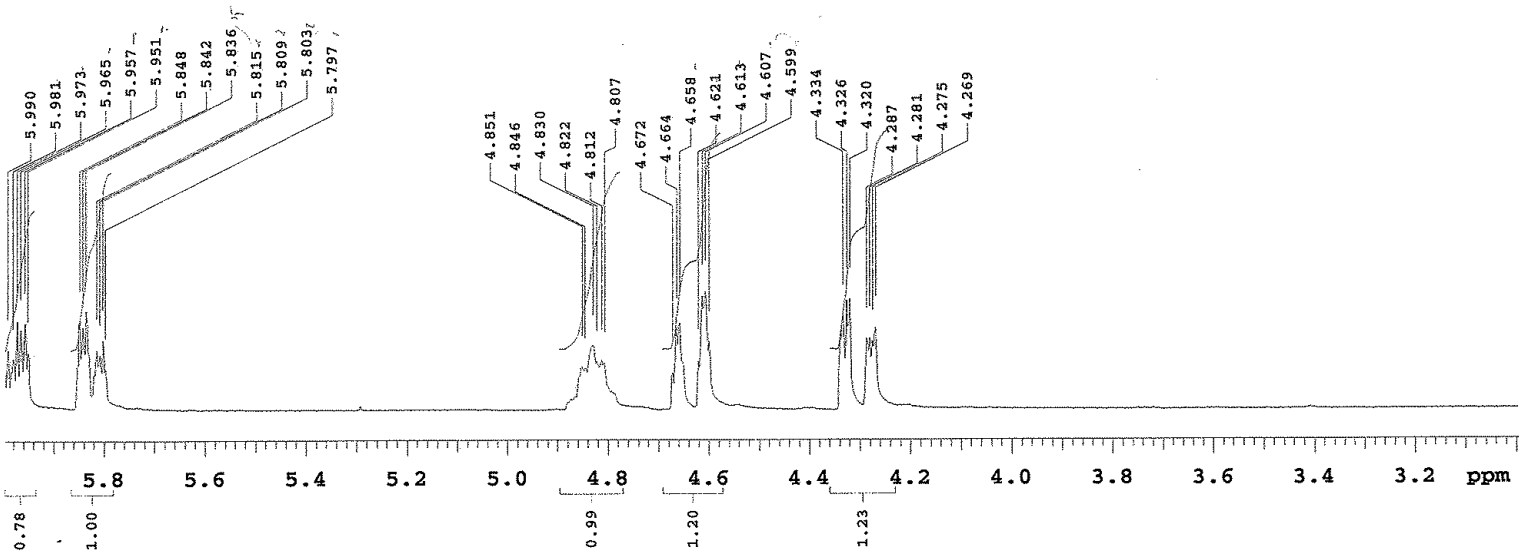
STANDARD PROTON PARAMETERS

File: home/kmn/vnmrsys/data/Anh/2011-02-06-TSA1-pure.fid

Pulse Sequence: s2pul

Temp. 25.0 C / 298.1 K
Operator: kmn

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.706 sec
Width 4803.1 Hz
32 repetitions
OBSERVE H1, 300.1259934 MHz
DATA PROCESSING
FT size 16384
Total time 1 min 29 sec



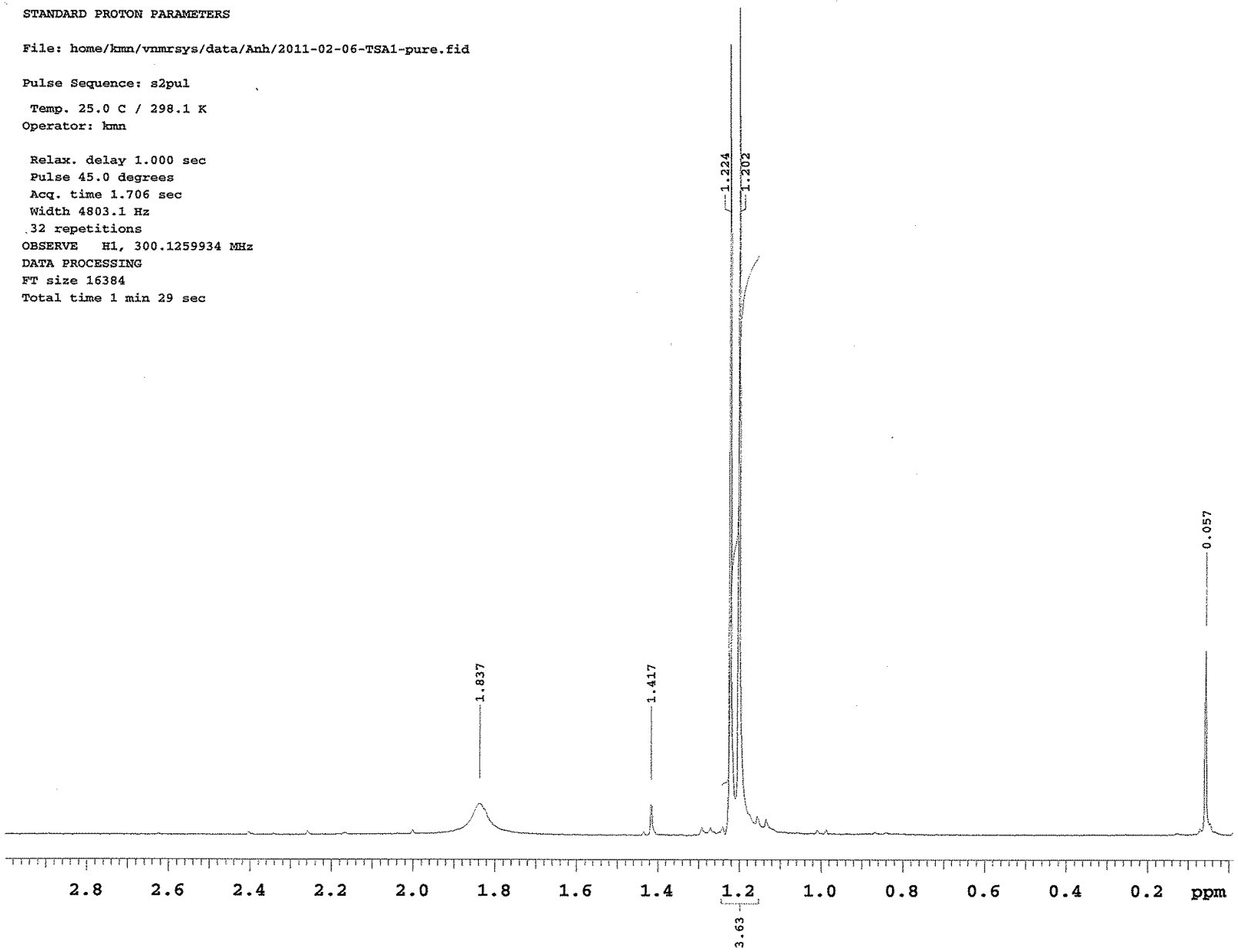
STANDARD PROTON PARAMETERS

File: home/kmn/vnmrsys/data/Anh/2011-02-06-TSA1-pure.fid

Pulse Sequence: s2pul

Temp. 25.0 C / 298.1 K
Operator: kmn

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.706 sec
Width 4803.1 Hz
32 repetitions
OBSERVE H1, 300.1259934 MHz
DATA PROCESSING
FT size 16384
Total time 1 min 29 sec



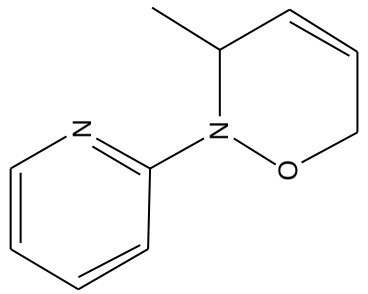
STANDARD PROTON PARAMETERS
Selective band center: 8.23 (ppm); width: 36.8 (Hz)

Temp. 21.0 C / 294.1 K
Operator: kmn

Relax. delay 1.000 sec
Pulse 90.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
360 repetitions
OBSERVE H1, 399.8659464 MHz
DATA PROCESSING
FT size 32768
Total time 120 hr, 27 min

TSA1

8.23 → NOE'S 1.24, 6.7, 7.6



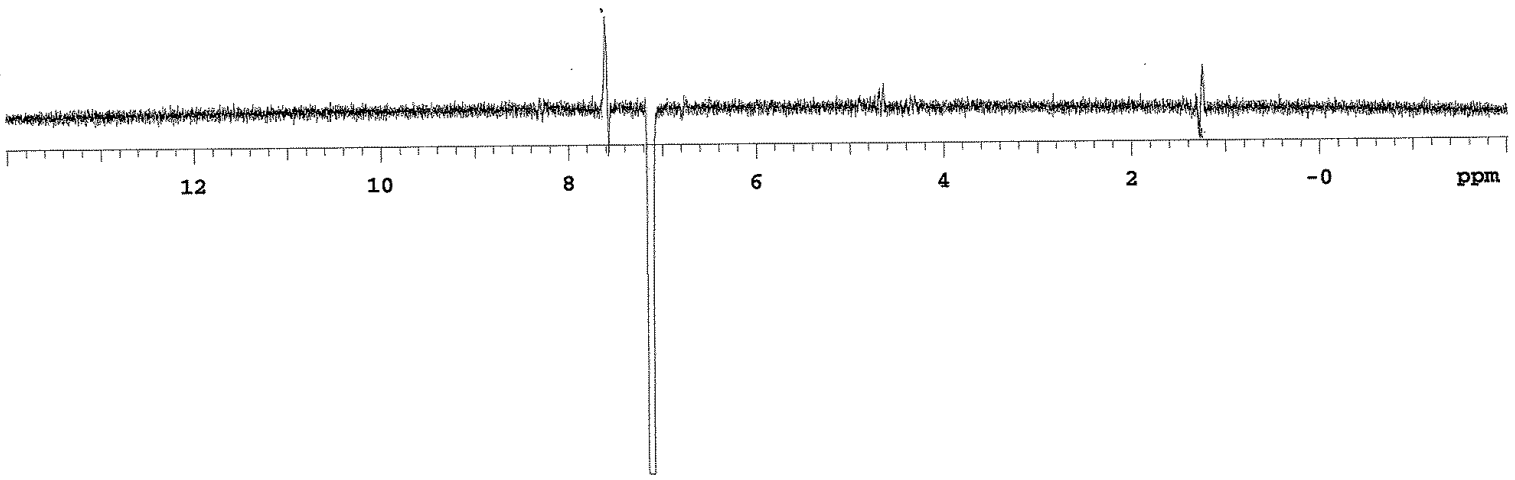
STANDARD PROTON PARAMETERS
Selective band center: 7.10 (ppm); width: 23.8 (Hz)

Temp. 21.0 C / 294.1 K
Operator: kmn

Relax. delay 1.000 sec
Pulse 90.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
104 repetitions
OBSERVE H1, 399.8659464 MHz
DATA PROCESSING
FT size 32768
Total time 1 hr, 15 min

TSA1

7.10 → NOEs 1.24, [illegible], 4.66 7.61



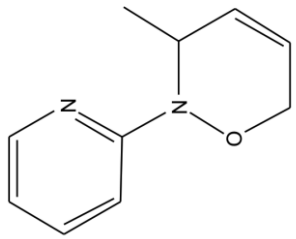
STANDARD PROTON PARAMETERS

File: xp

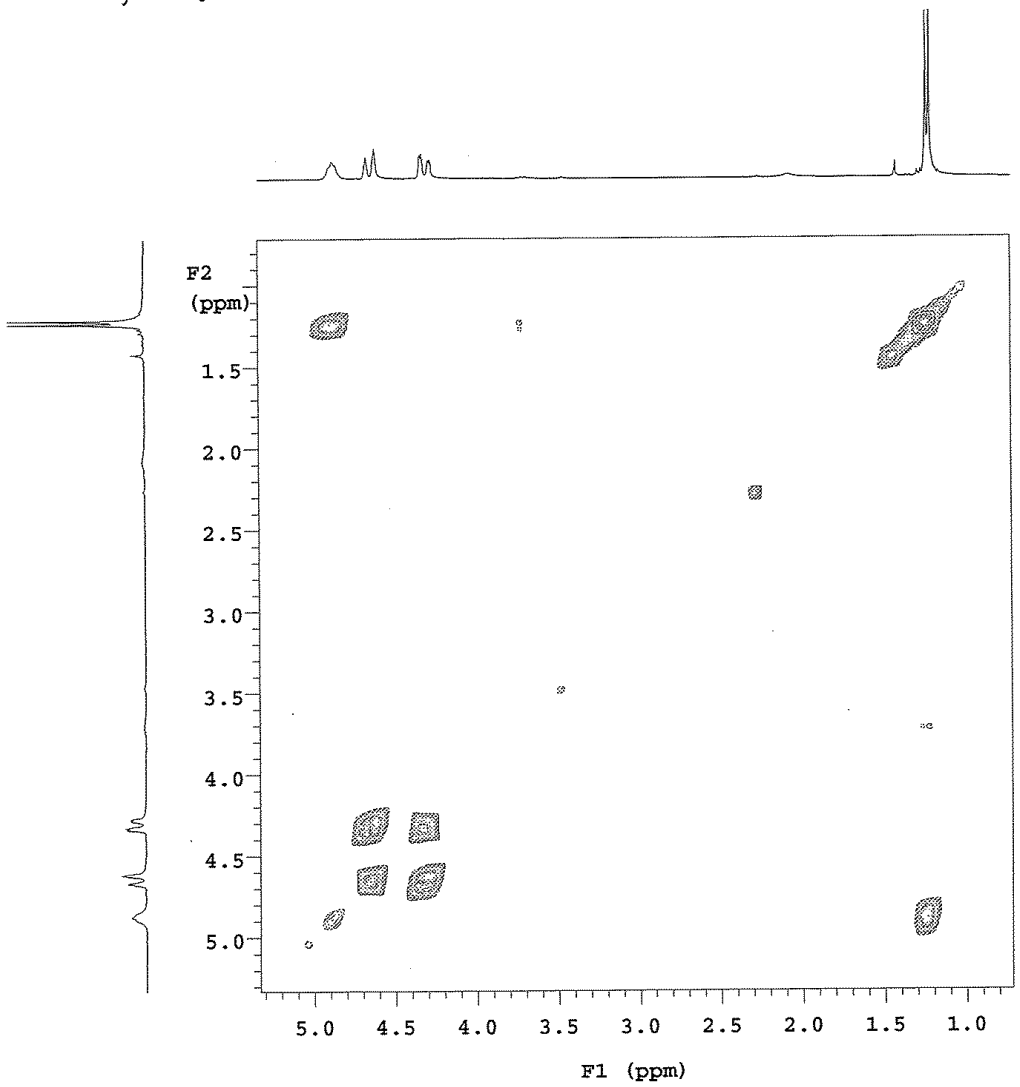
Pulse Sequence: gCOSY

Operator: kmn

Relax. delay 1.000 sec
Acq. time 0.150 sec
Width 4803.1 Hz
2D Width 4803.1 Hz
Single scan
128 increments
OBSERVE H1, 300.1259964 MHz
DATA PROCESSING
Sq. sine bell 0.075 sec
F1 DATA PROCESSING
Sq. sine bell 0.027 sec
FT size 2048 x 2048
Total time 4 min 8 sec



TSM1-gcosy-062211.fid



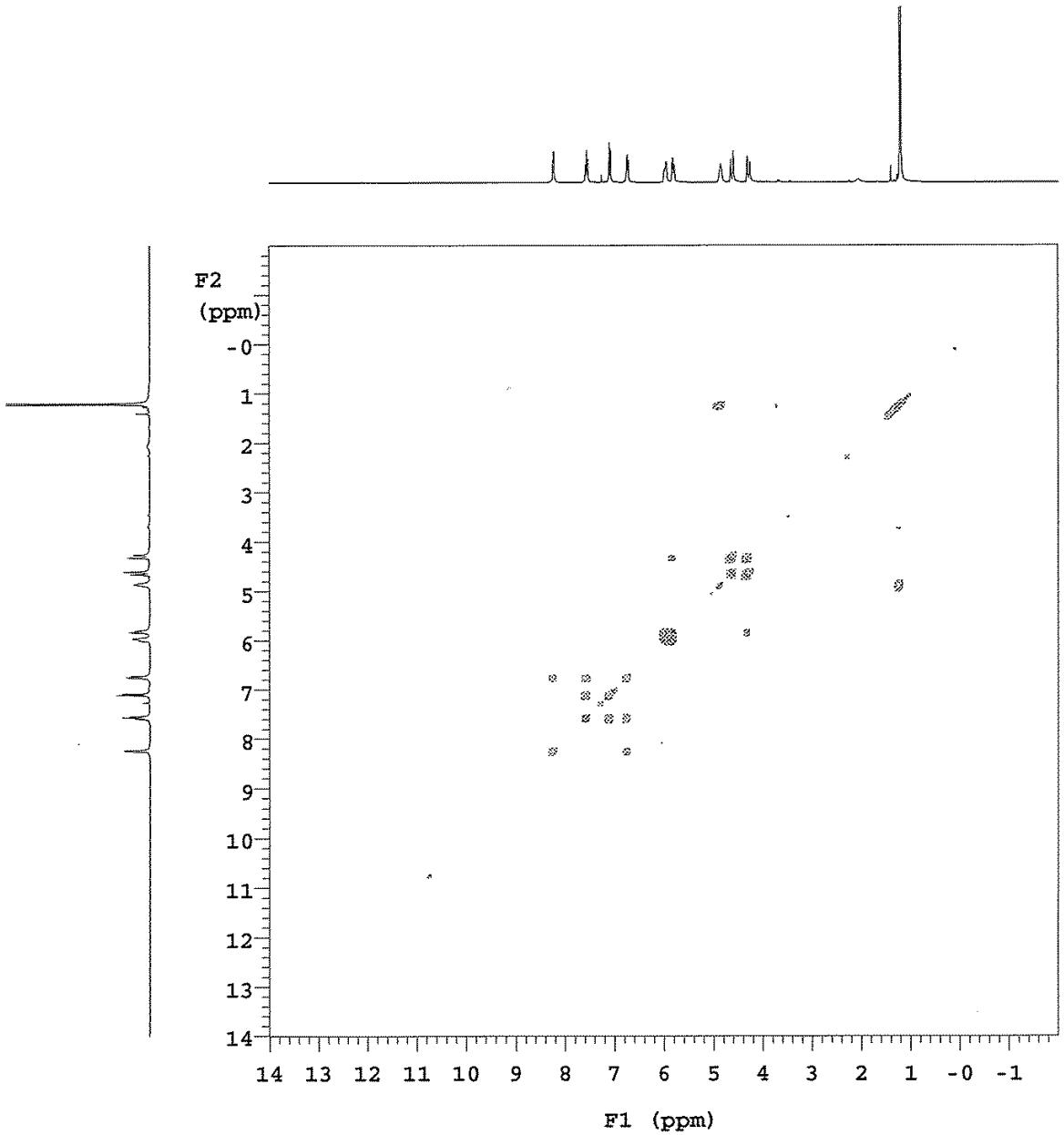
STANDARD PROTON PARAMETERS

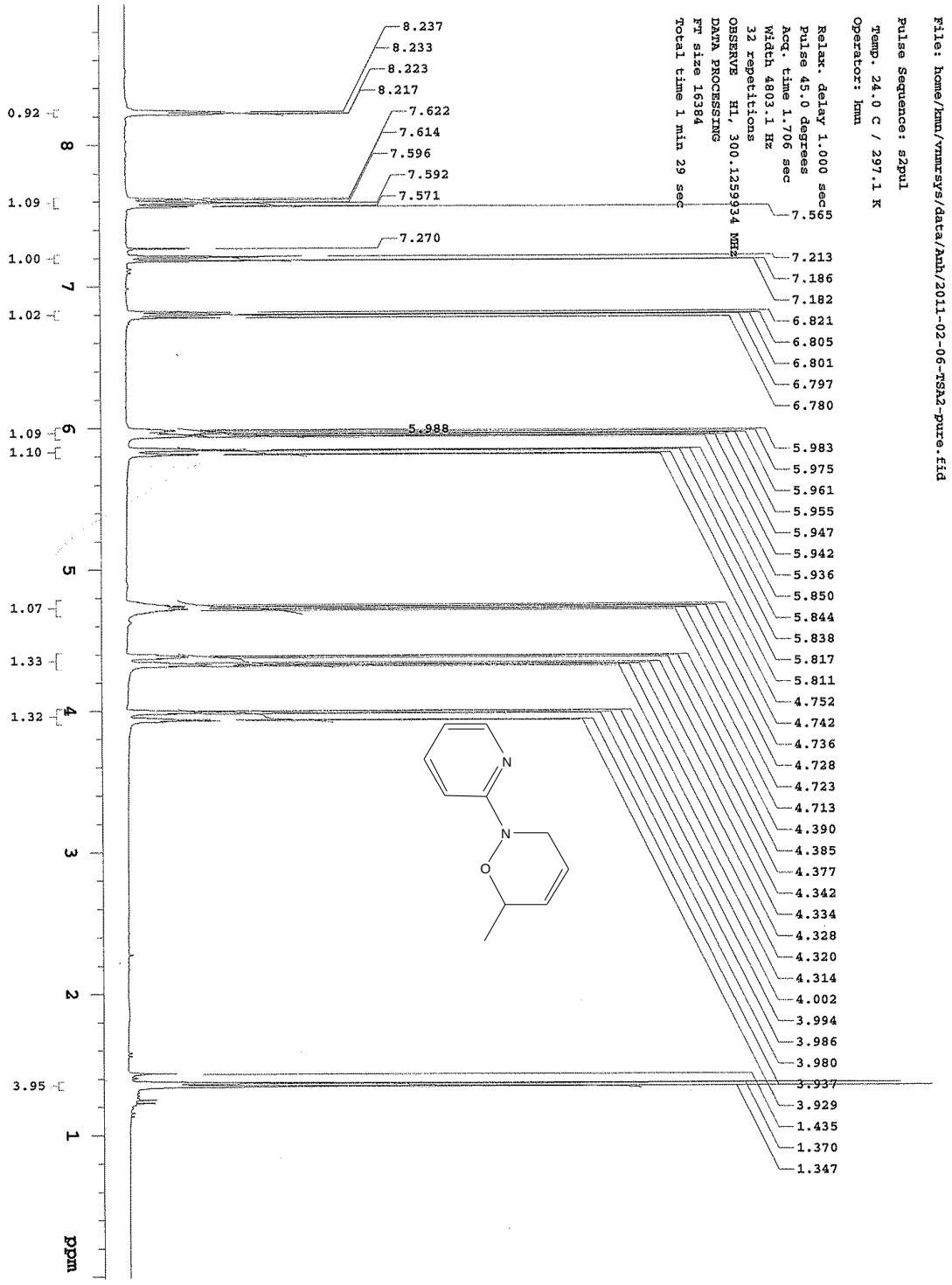
File: xp

Pulse Sequence: gCOSY

Operator: kmn

Relax. delay 1.000 sec
Acq. time 0.150 sec
Width 4803.1 Hz
2D Width 4803.1 Hz
Single scan
128 increments
OBSERVE H1, 300.1259964 MHz
DATA PROCESSING
Sq. sine bell 0.075 sec
F1 DATA PROCESSING
Sq. sine bell 0.027 sec
FT size 2048 x 2048
Total time 4 min 8 sec



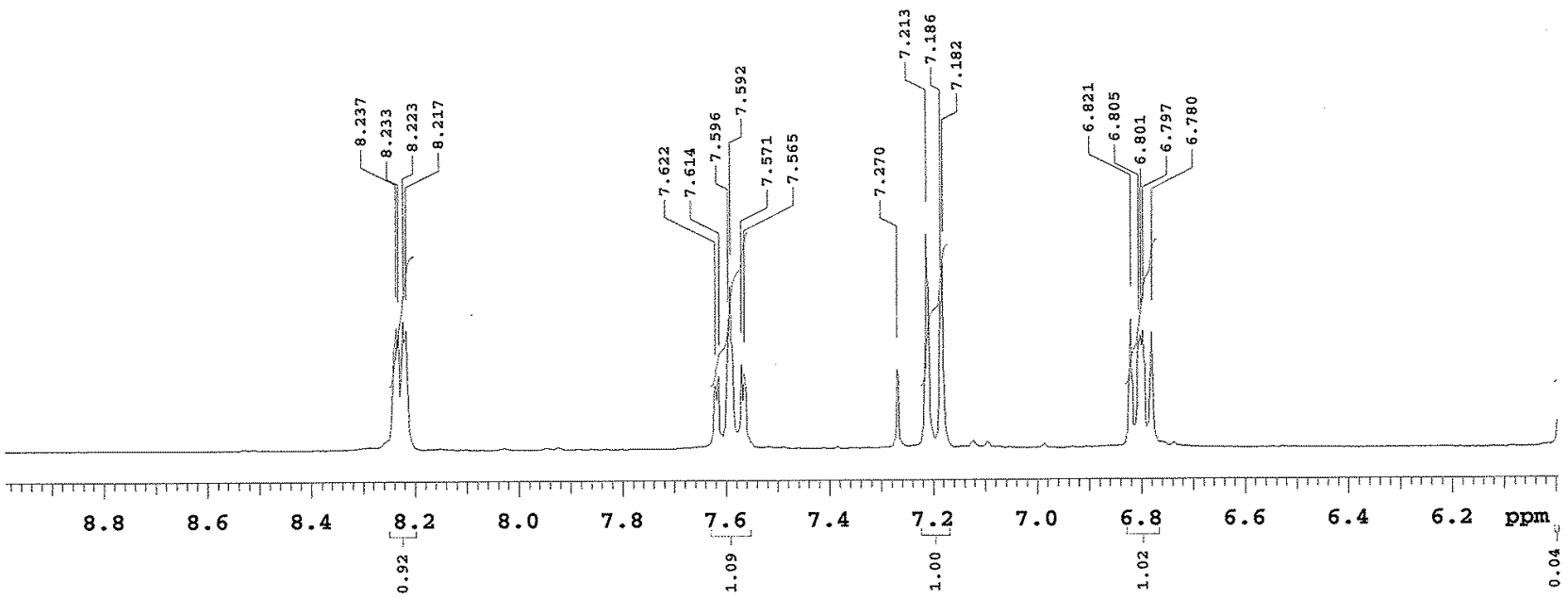

File: home/kmn/vnmrsys/data/anh/2011-02-06-TSA2-pure.fid
Pulse Sequence: s2pul
Temp. 24.0 C / 297.1 K
Operator: kmn
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.706 sec
Width 4803.1 Hz
32 repetitions
OBSERVE H1, 300.1259934 MHz
DATA PROCESSING
FT size 16384
Total time 1 min 29 sec
8.237
8.233
8.223
8.217
7.622
7.614
7.596
7.592
7.571
7.565
7.270
7.213
7.186
7.182
6.821
6.805
6.801
6.797
6.780
5.988
5.983
5.975
5.961
5.955
5.947
5.942
5.936
5.850
5.844
5.838
5.817
5.811
4.752
4.742
4.736
4.728
4.723
4.713
4.390
4.385
4.377
4.342
4.334
4.328
4.320
4.314
4.002
3.994
3.986
3.980
3.937
3.929
1.435
1.370
1.347
0.92
1.09
1.00
1.02
1.09
1.10
1.07
1.33
1.32
3.95
N
N
O
ppm

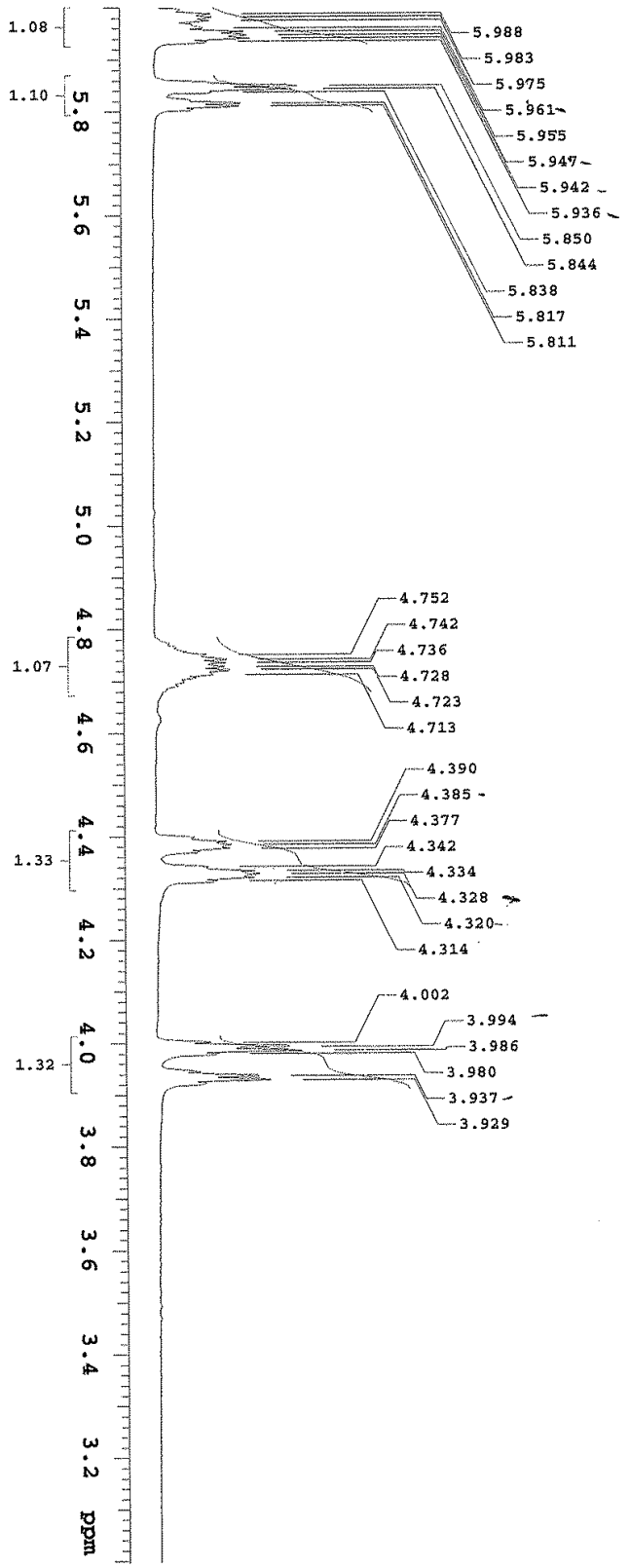
File: home/kmn/vnmrsys/data/Anh/2011-02-06-TSA2-pure.fid

Pulse Sequence: s2pul

Temp. 24.0 C / 297.1 K
Operator: kmn

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.706 sec
Width 4803.1 Hz
32 repetitions
OBSERVE H1, 300.1259934 MHz
DATA PROCESSING
FT size 16384
Total time 1 min 29 sec



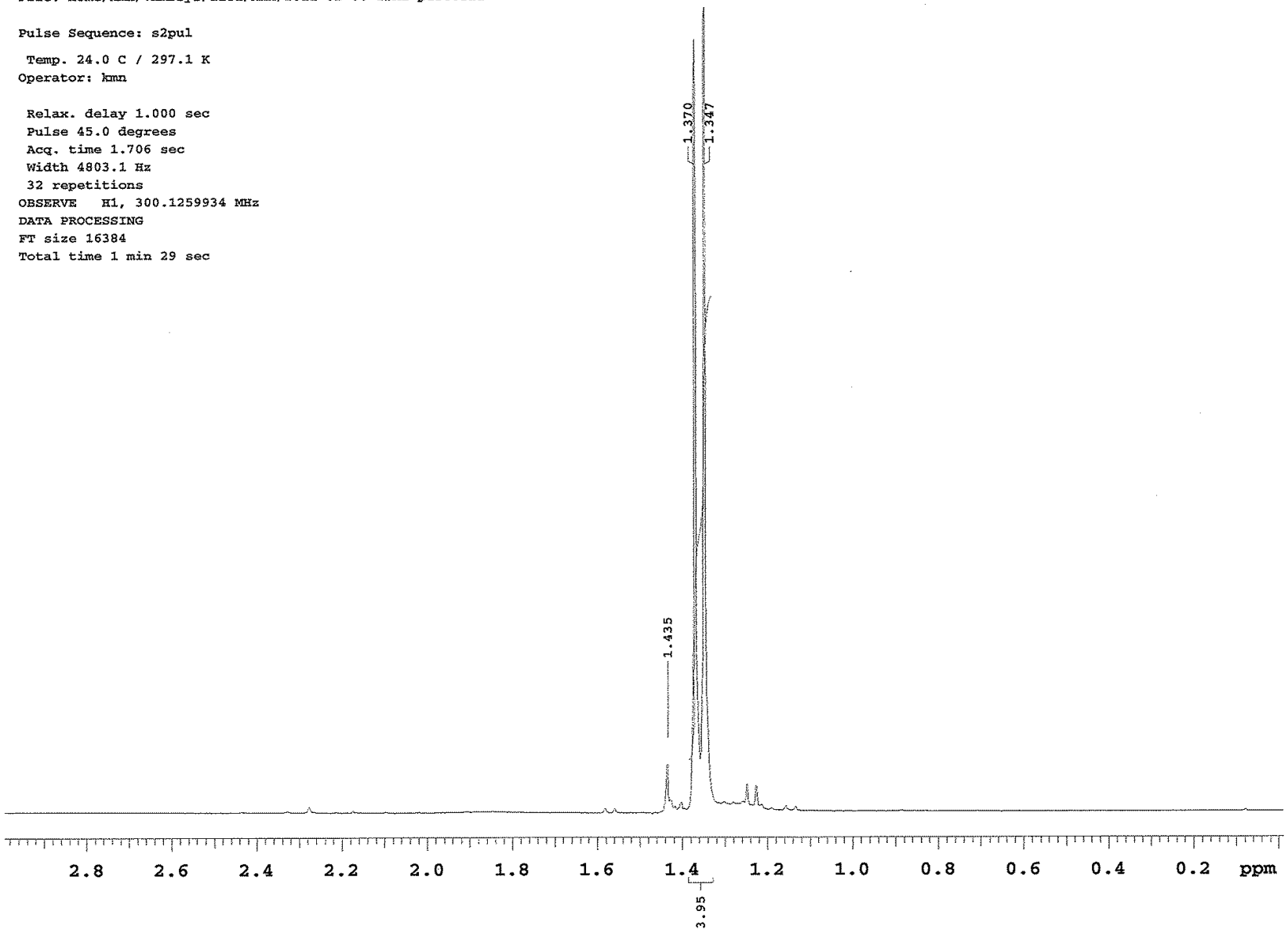

File: home/kmn/vnmrsys/data/Anh/2011-02-06-TSA2-pure.fid
Pulse Sequence: s2pul
Temp. 24.0 C / 297.1 K
Operator: kmn
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.706 sec
Width 4803.1 Hz
32 repetitions
OBSERVE H1, 300.1259934 MHz
DATA PROCESSING
FT size 16384
Total time 1 min 29 sec
5.988
5.983
5.975
5.961
5.955
5.947
5.942
5.936
5.850
5.844
5.838
5.817
5.811
4.752
4.742
4.736
4.728
4.723
4.713
4.390
4.385
4.377
4.342
4.334
4.328
4.320
4.314
4.002
3.994
3.986
3.980
3.937
3.929
1.08
1.10
1.07
1.33
1.32
5.8
5.6
5.4
5.2
5.0
4.8
4.6
4.4
4.2
4.0
3.8
3.6
3.4
3.2
ppm

File: home/kmn/vnmrsys/data/Anh/2011-02-06-TSA2-pure.fid

Pulse Sequence: s2pul

Temp. 24.0 C / 297.1 K
Operator: kmn

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.706 sec
Width 4803.1 Hz
32 repetitions
OBSERVE H1, 300.1259934 MHz
DATA PROCESSING
FT size 16384
Total time 1 min 29 sec



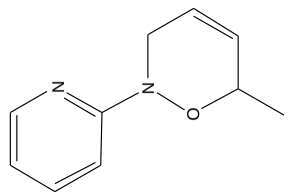
STANDARD PROTON PARAMETERS

File: xp

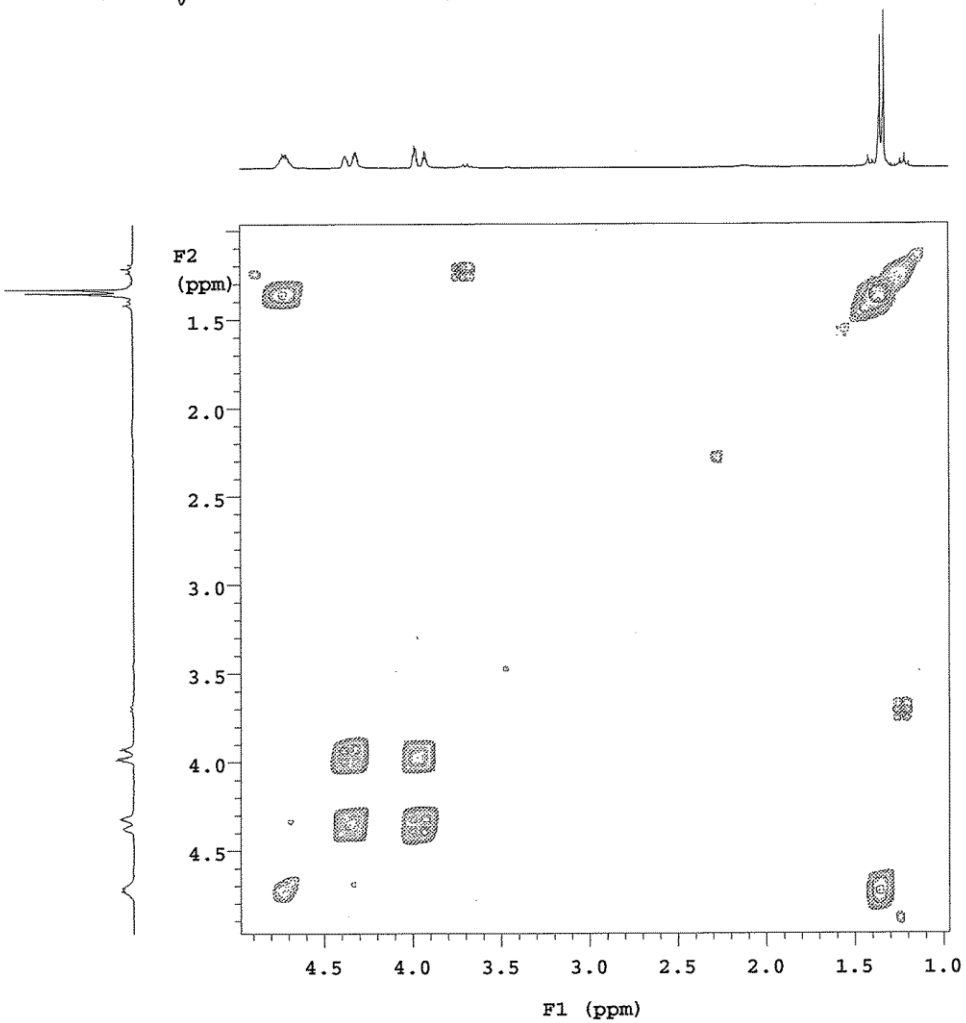
Pulse Sequence: gCOSY

Operator: kmn

Relax. delay 1.000 sec
Acq. time 0.150 sec
Width 4803.1 Hz
2D Width 4803.1 Hz
Single scan
128 increments
OBSERVE H1, 300.1259964 MHz
DATA PROCESSING
Sq. sine bell 0.075 sec
F1 DATA PROCESSING
Sq. sine bell 0.027 sec
FT size 2048 x 2048
Total time 4 min 8 sec



TSA2gCOSY-062211-fid



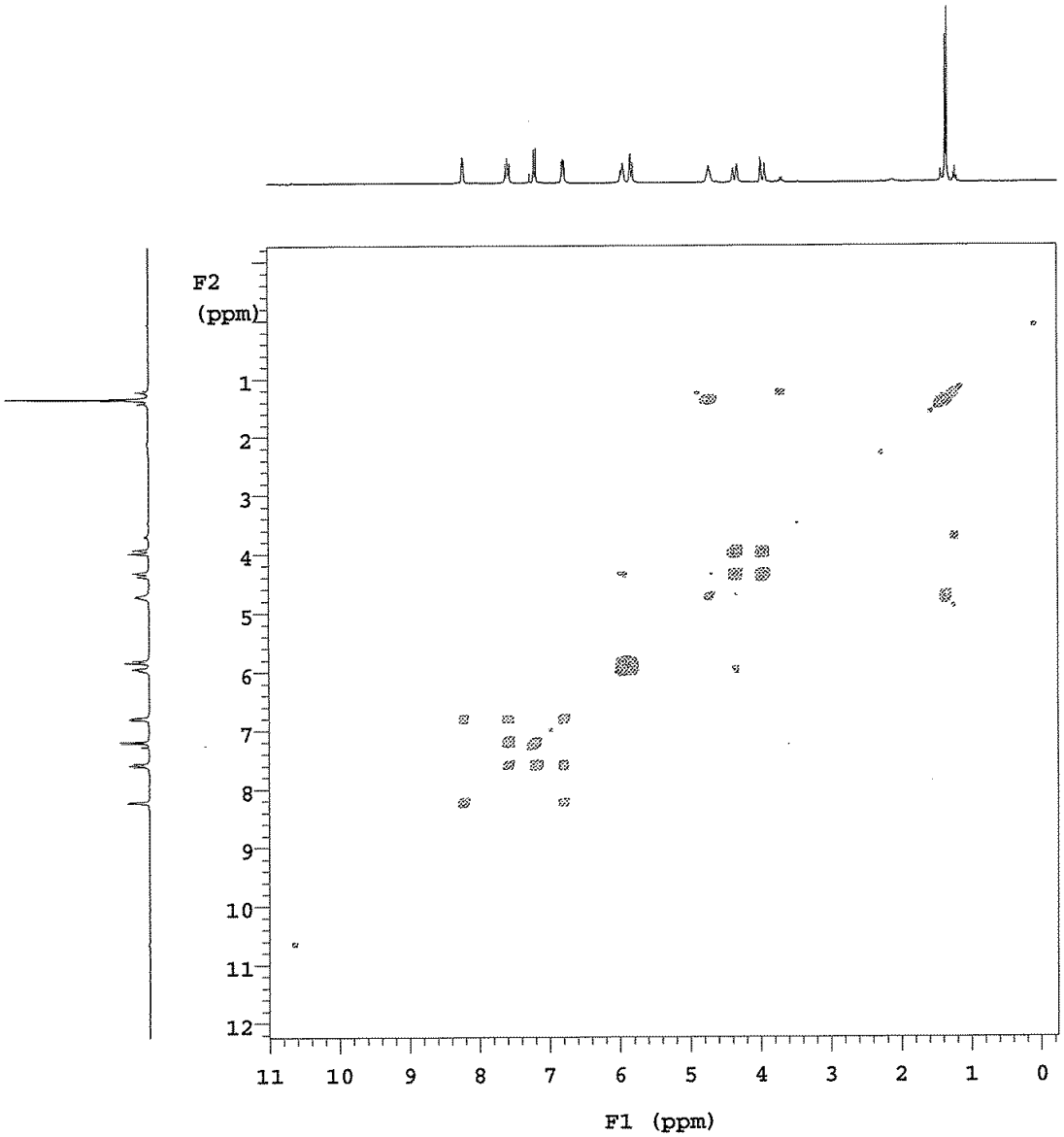
STANDARD PROTON PARAMETERS

File: xp

Pulse Sequence: gCOSY

Operator: kmn

Relax. delay 1.000 sec
Acq. time 0.150 sec
Width 4803.1 Hz
2D Width 4803.1 Hz
Single scan
128 increments
OBSERVE H1, 300.1259964 MHz
DATA PROCESSING
Sq. sine bell 0.075 sec
F1 DATA PROCESSING
Sq. sine bell 0.027 sec
FT size 2048 x 2048
Total time 4 min 8 sec



STANDARD PROTON PARAMETERS
Selective band center: 8.21 (ppm); width: 45.4 (Hz)
file=TSA2-1D-noesy-400.fid

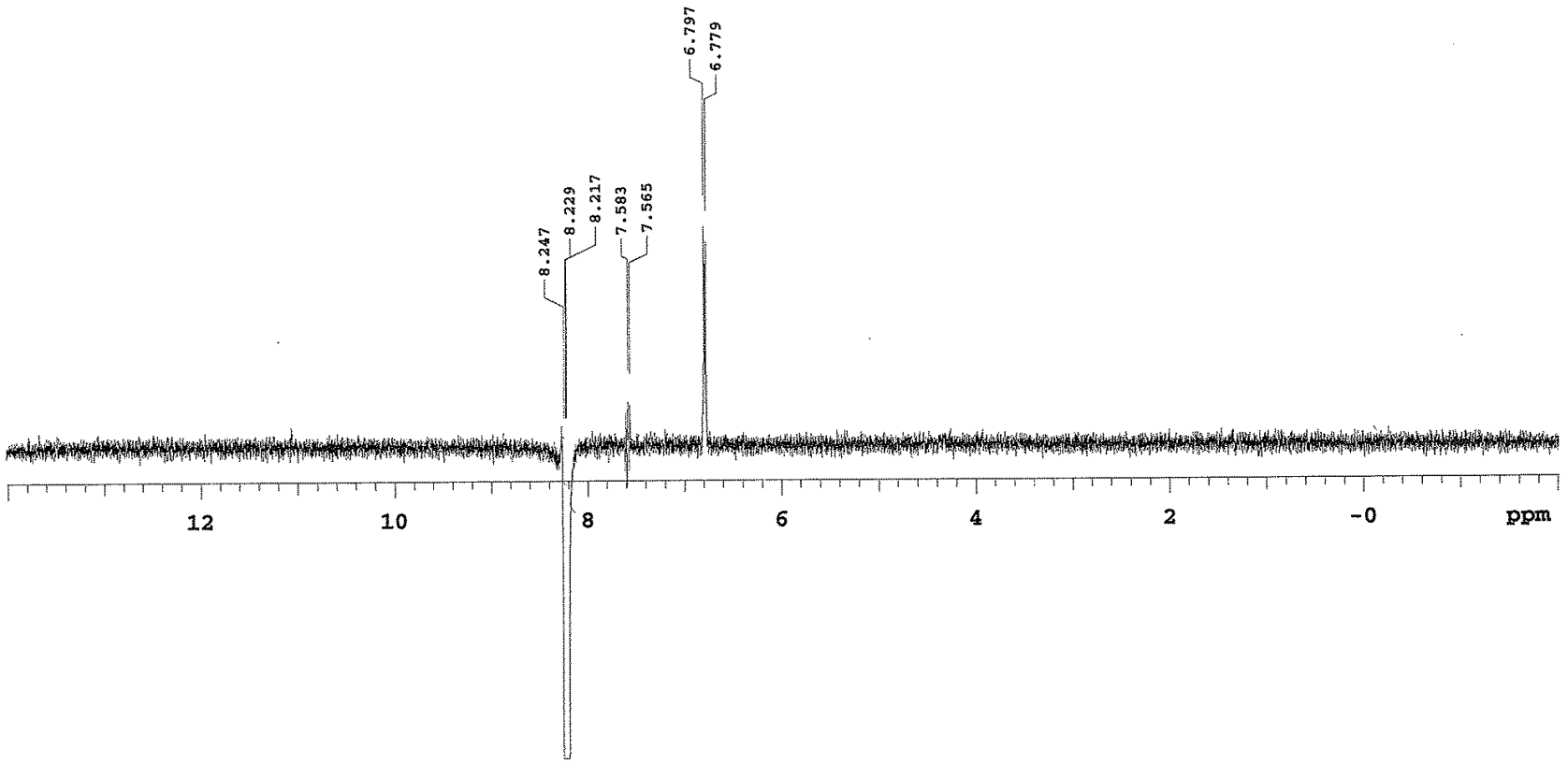
Temp. 21.0 C / 294.1 K
Operator: kmn

Relax. delay 1.000 sec
Pulse 90.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
188 repetitions
OBSERVE H1, 399.8659464 MHz
DATA PROCESSING
FT size 32768
Total time 11 hr, 56 min

TSA 2

NOE

8.21 → 6.79, 7.58

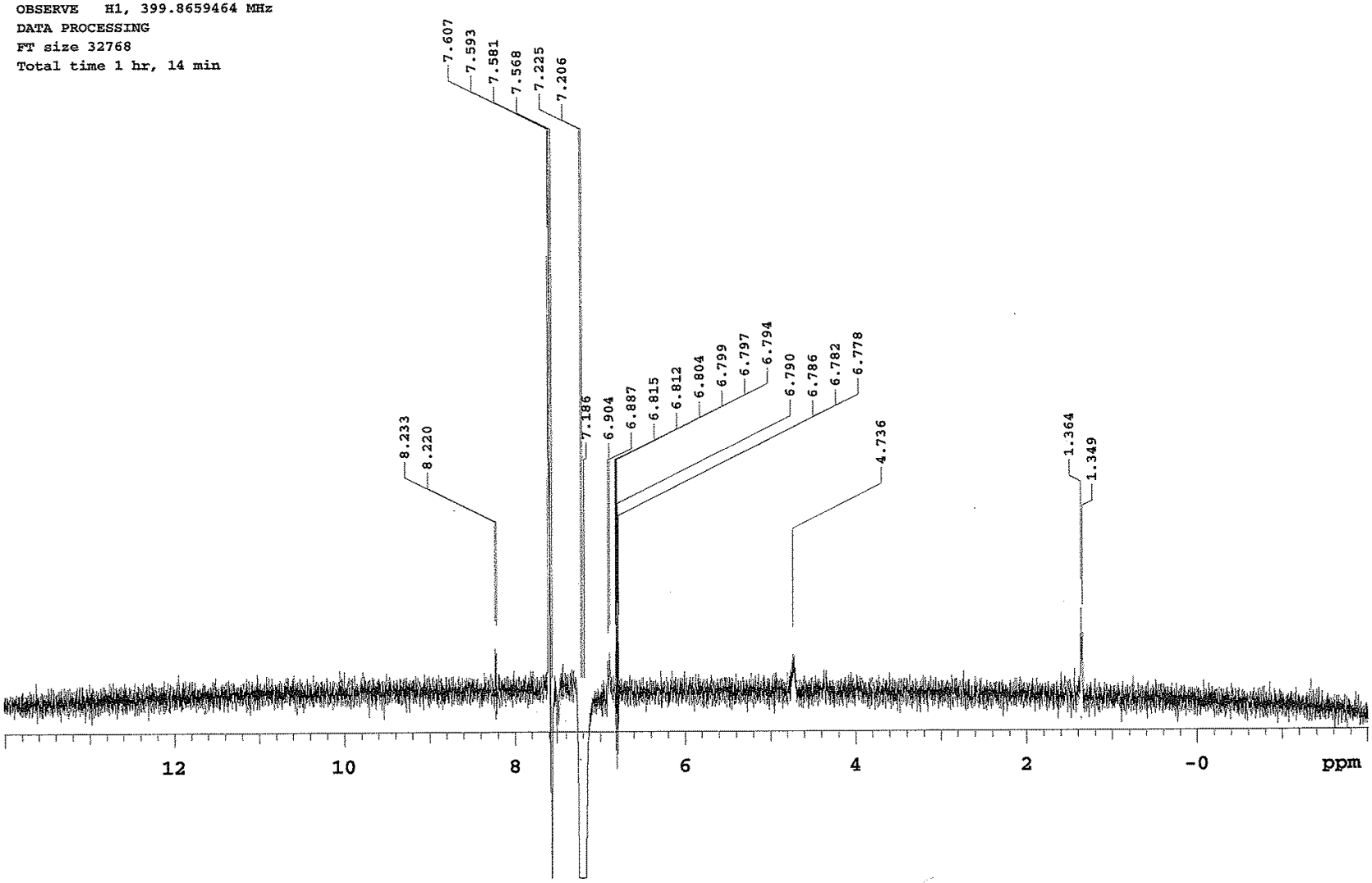


STANDARD PROTON PARAMETERS
Selective band center: 7.19 (ppm); width: 31.6 (Hz)

Temp. 21.0 C / 294.1 K
Operator: kmn

Relax. delay 1.000 sec
Pulse 90.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
116 repetitions
OBSERVE H1, 399.8659464 MHz
DATA PROCESSING
FT size 32768
Total time 1 hr, 14 min

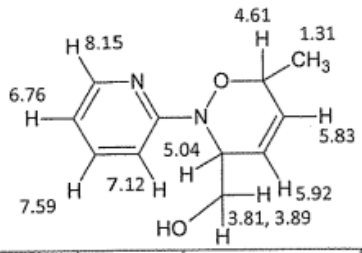
TSA2
7.19 → NOE 8.23, 7.6, 6.8, 4.74, 1.37

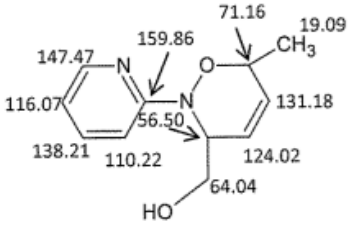


Summary NMR of (6-methyl-2-(pyridin-2-yl)-3,6-dihydro-2H-1,2-oxazin-3-yl)methanol (Distal 2)

Proton referenced to chloroform relative to TMS at 7.26 ppm and carbon referenced to 77.23 ppm

Fraction 1





Carbon	mult	Proton (HSQC)	Mult (J Hz)	COSY	Proton(HBMC)	NOE
19.09	CH_3	1.31	d(6.7)	4.61	5.83, 4.61	
56.50	CH	5.04	m	3.89, 5.92*	5.92, 5.83, 3.89	
64.04	CH_2	3.81	dd(11.6, 3.3)	5.04, 3.92	5.83	
64.04	CH_2	3.89	dd(11.6, 7.1)	3.81		
71.16	CH	4.61	m	1.31	5.92, 5.83, 1.31	
110.22	CH	7.12	dd(7.9,0.98)	7.59	8.15, 7.59, 6.76	7.59, 4.61, 1.31
116.07	CH	6.76	ddd(7.9, 5.0, 0.98)	7.59, 8.15	8.15 7.12	
124.02	CH	5.92	ddd(10.5,4.5,2)	3.89*	5.92*, 4.61, 3.89, 3.81, 1.31	
131.18	CH	5.83	dt(10.5, 1.6)		1.31	
138.21	CH	7.59	dt(7.9, 7.9, 1.7)	6.76, 7.12	8.15, 6.76	
147.47	CH	8.15	dd(5.0, 1.7)	6.76	7.59, 6.76	
159.86	C	---			8.15, 7.59, 6.76, 5.92, 3.81	

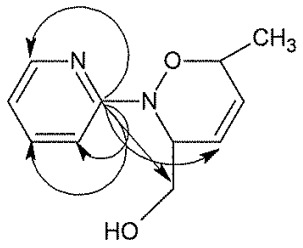
April 10, 2012

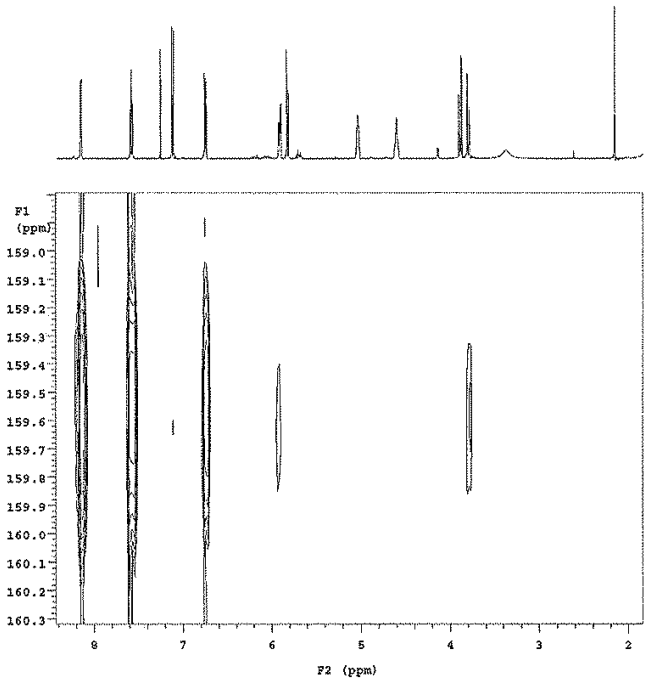
Fraction 1

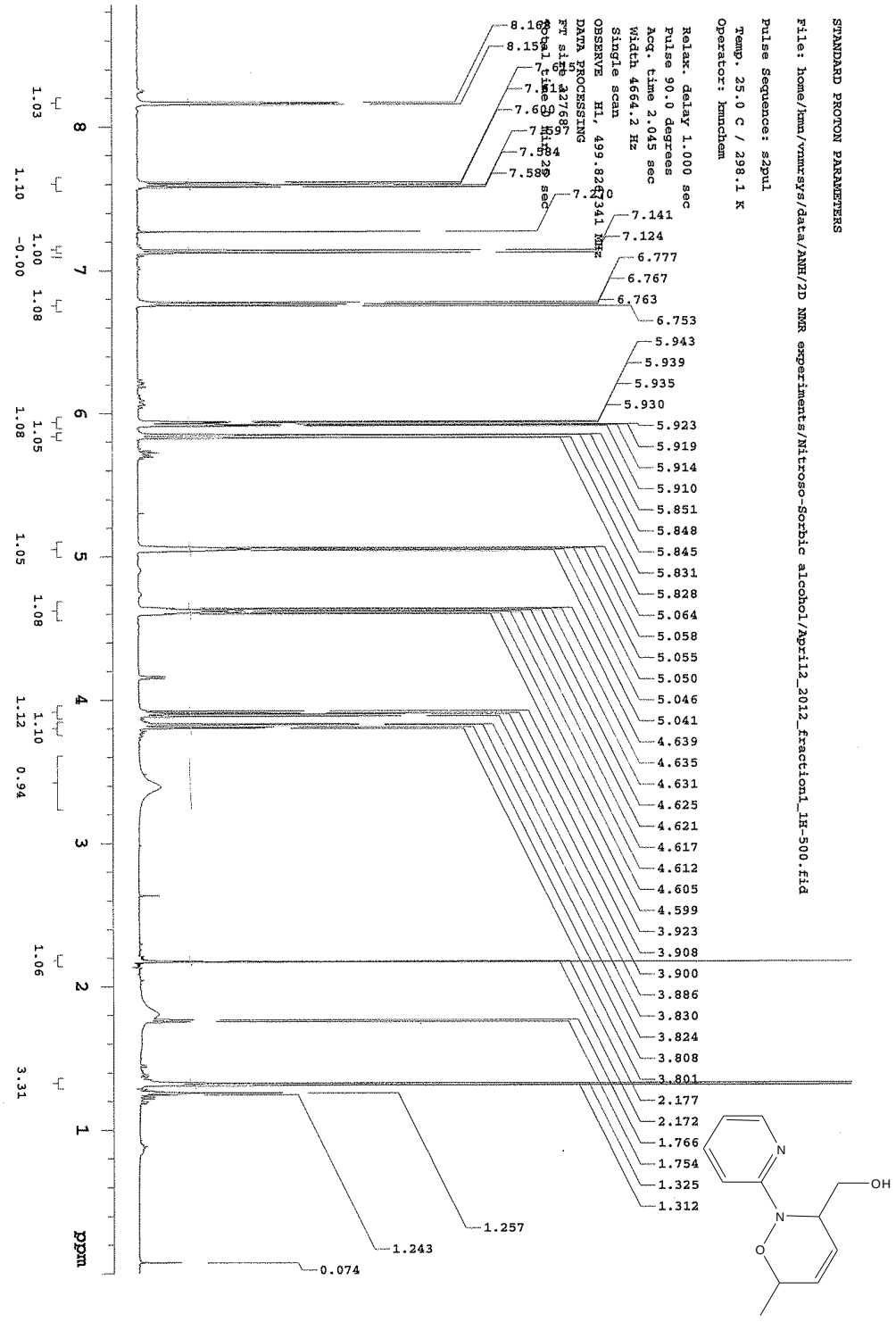
Nitroso-Sorbic- alcohol

gHMBC

Through Bond C-H long range coupling






STANDARD PROTON PARAMETERS
File: home/hma/vnmrsys/data/ANH/2D NMR experiments/Nitroso-Sorbic alcohol/April2_2012_fraction1_1H-500.fid
Pulse Sequence: s2pul
Temp. 25.0 C / 298.1 K
Operator: hmnchem
Relax. delay 1.000 sec
Pulse 90.0 degrees
Acq. time 2.045 sec
Width 4664.2 Hz
Single scan
OBSERVE H1, 499.8247341 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 29 sec
8.163
8.151
7.600
7.597
7.584
7.580
7.141
7.124
6.777
6.767
6.763
6.753
5.943
5.939
5.935
5.930
5.923
5.919
5.914
5.910
5.851
5.848
5.845
5.831
5.828
5.064
5.058
5.055
5.050
5.046
5.041
4.639
4.635
4.631
4.625
4.621
4.617
4.612
4.605
4.599
3.923
3.908
3.900
3.886
3.830
3.824
3.808
3.801
2.177
2.172
1.766
1.754
1.325
1.312
1.257
1.243
0.074
1.03
1.10
1.00
-0.00
1.08
1.08
1.05
1.05
1.08
1.12
1.10
0.94
1.06
3.31
ppm
N
N
O
OH

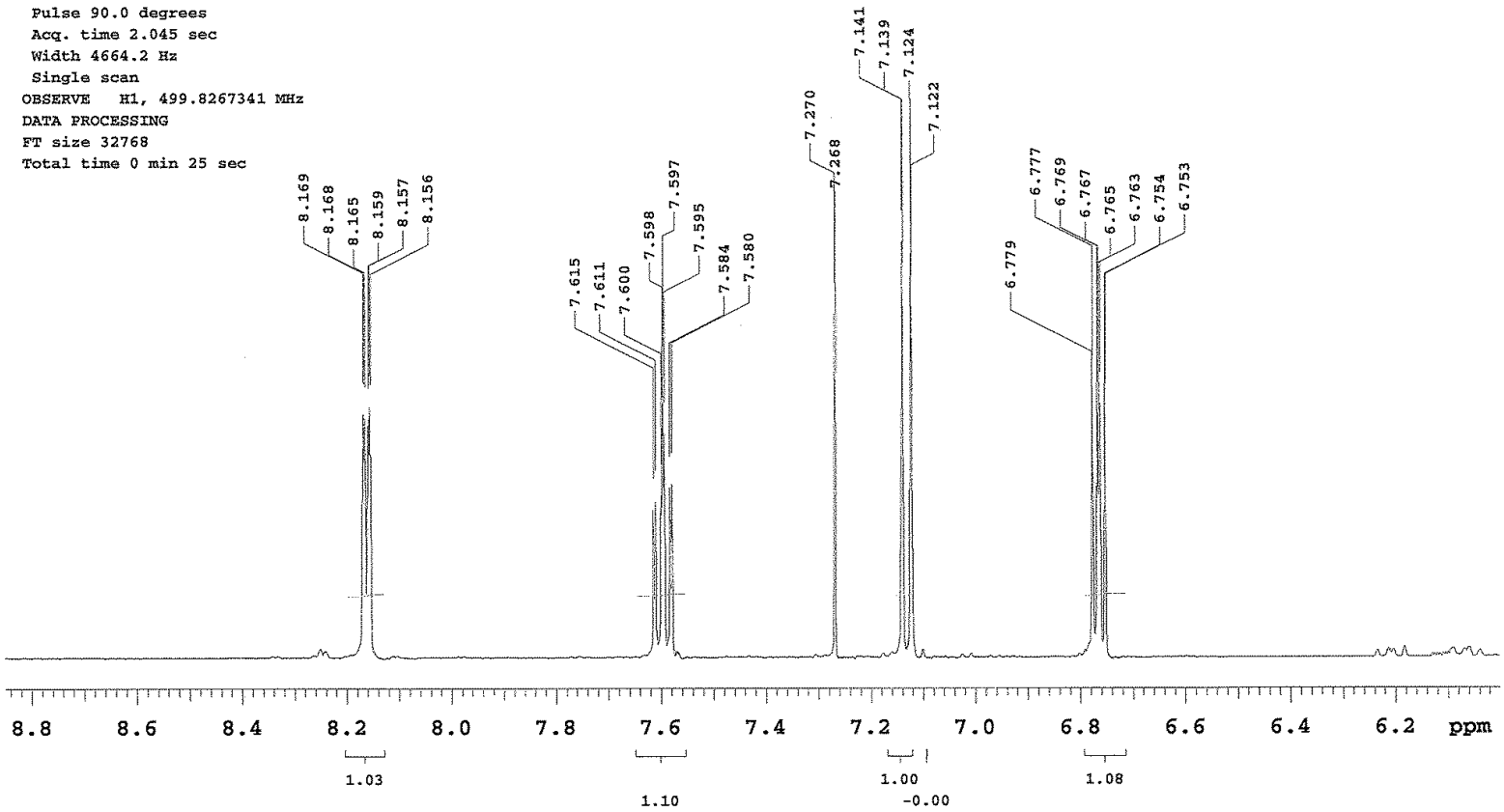
STANDARD PROTON PARAMETERS

File: home/kmn/vnmrsys/data/ANH/2D NMR experiments/Nitroso-Sorbic alcohol/April2_2012_fraction1_1H-500.fid

Pulse Sequence: s2pul

Temp. 25.0 C / 298.1 K
Operator: kmnchem

Relax. delay 1.000 sec
Pulse 90.0 degrees
Acq. time 2.045 sec
Width 4664.2 Hz
Single scan
OBSERVE H1, 499.8267341 MHz
DATA PROCESSING
FT size 32768
Total time 0 min 25 sec



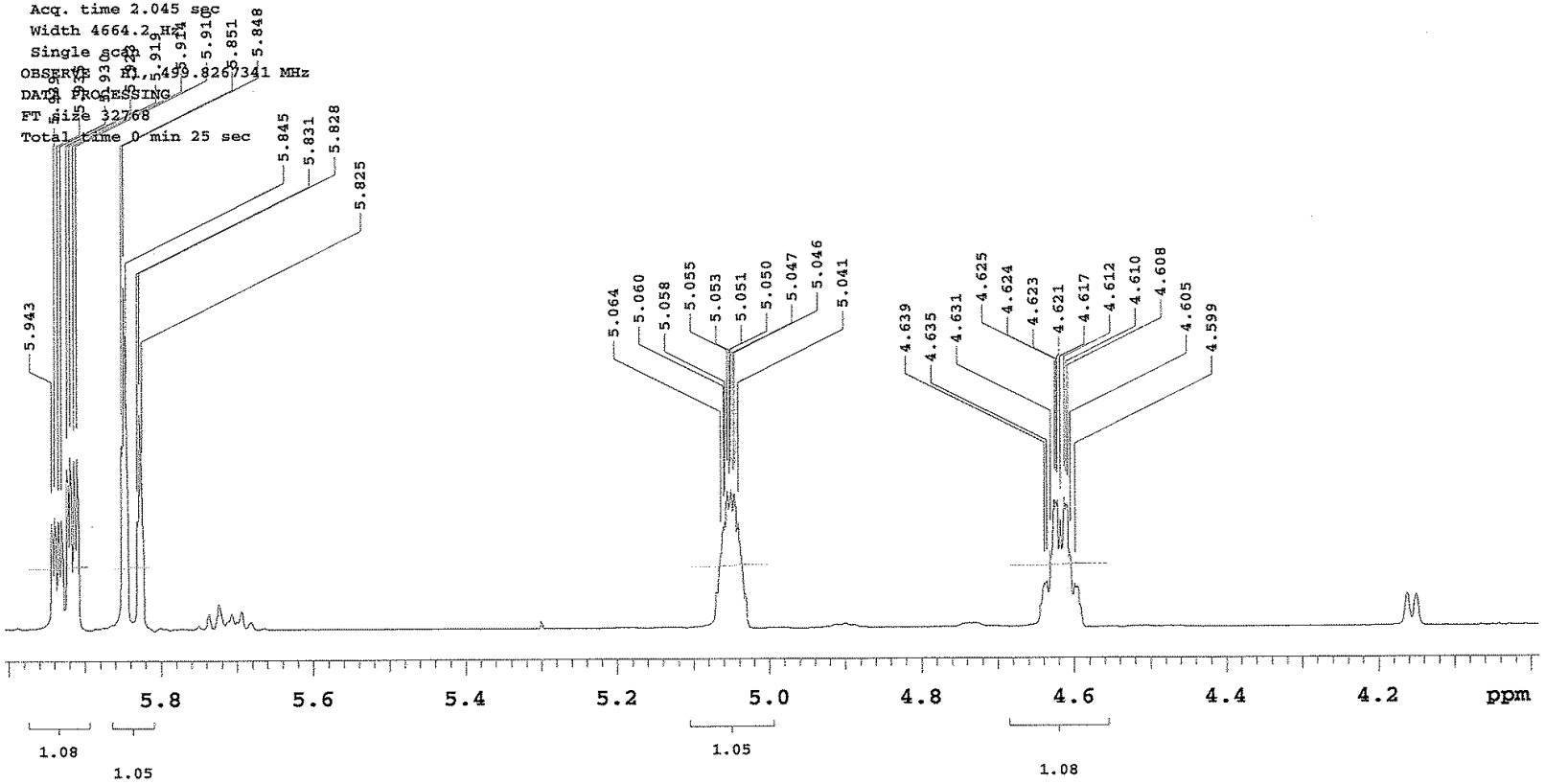
STANDARD PROTON PARAMETERS

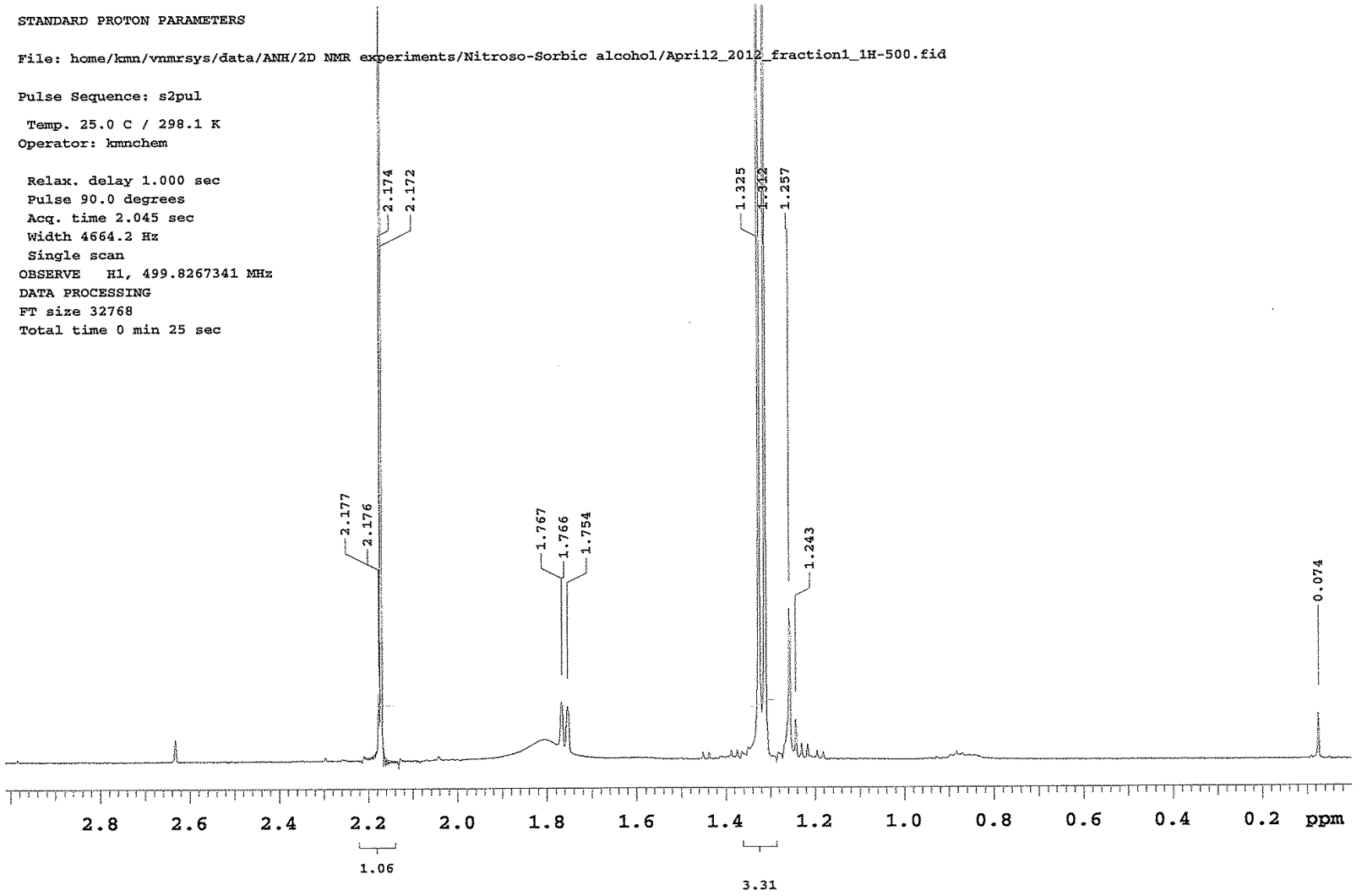
File: home/kmn/vnmrsys/data/ANH/2D NMR experiments/Nitroso-Sorbic alcohol/April2_2012_fraction1_1H-500.fid

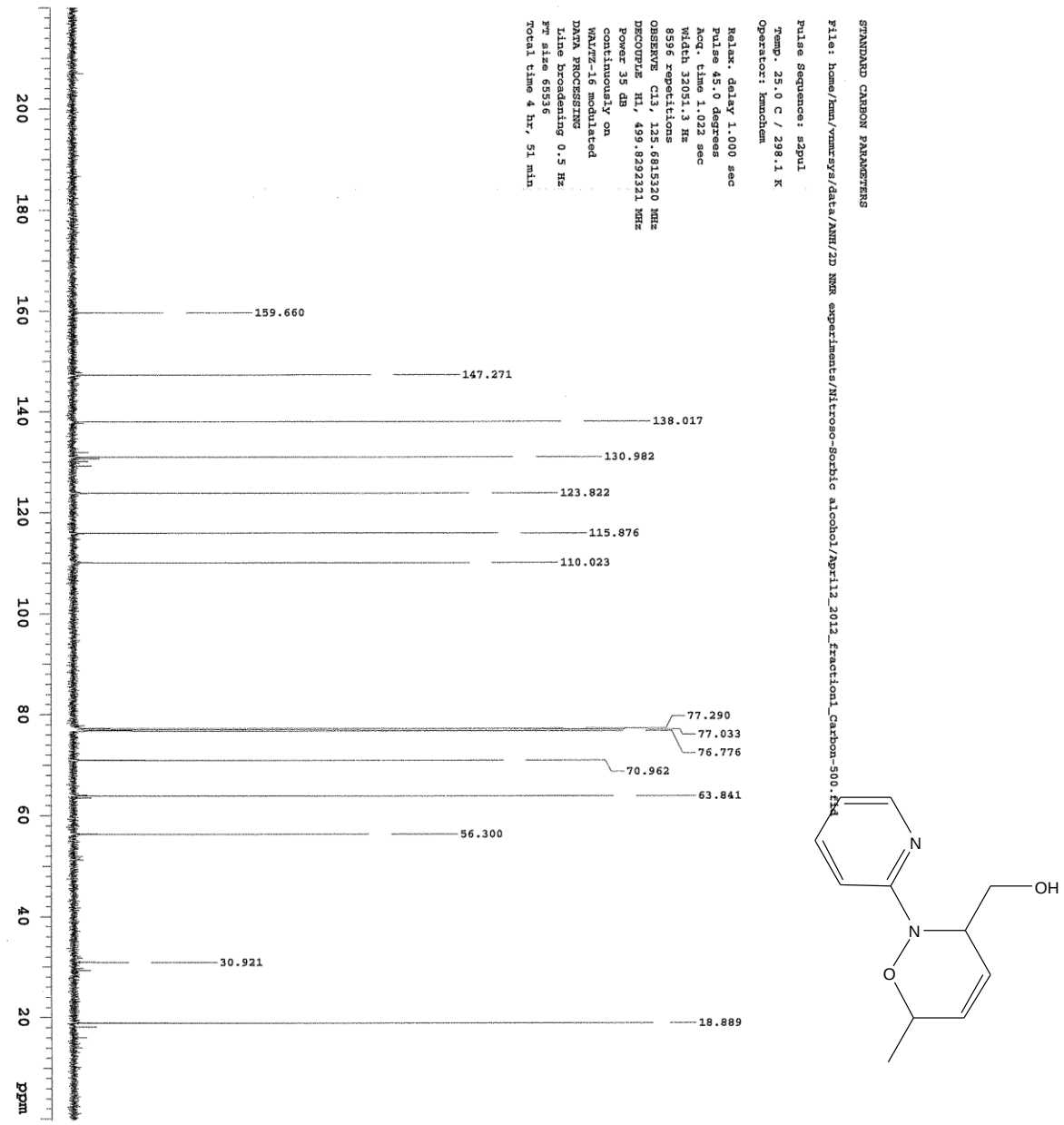
Pulse Sequence: s2pul

Temp. 25.0 C / 298.1 K
Operator: kmnchem

Relax. delay 1.000 sec
Pulse 90.0 degrees
Acq. time 2.045 sec
Width 4664.2 Hz
Single scan
OBSERVE H1, 499.8267341 MHz
DATA PROCESSING
FT size 32768
Total time 0 min 25 sec




STANDARD PROTON PARAMETERS
File: home/kmn/vnmrsys/data/ANH/2D NMR experiments/Nitroso-Sorbic alcohol/April2_2012_fraction1_1H-500.fid
Pulse Sequence: s2pul
Temp. 25.0 C / 298.1 K
Operator: kmnchem
Relax. delay 1.000 sec
Pulse 90.0 degrees
Acq. time 2.045 sec
Width 4664.2 Hz
Single scan
OBSERVE H1, 499.8267341 MHz
DATA PROCESSING
FT size 32768
Total time 0 min 25 sec
2.177
2.176
2.174
2.172
1.767
1.766
1.754
1.325
1.312
1.257
1.243
0.074
2.8
2.6
2.4
2.2
2.0
1.8
1.6
1.4
1.2
1.0
0.8
0.6
0.4
0.2
ppm
1.06
3.31


STANDARD CARBON PARAMETERS
File: home/hnm/vnmrsys/data/ANH/2D NMR experiments/Nitroso-Sorbic alcohol/April2_2012_fraction1_Carbon-500.fid
Pulse Sequence: s2pul
Temp. 25.0 C / 298.1 K
Operator: hmnchem
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.022 sec
Width 32051.3 Hz
8596 repetitions
OBSERVE C13, 125.6815320 MHz
DECOUPLE H1, 499.8292321 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 4 hr, 51 min
159.660
147.271
138.017
130.982
123.822
115.876
110.023
77.290
77.033
76.776
70.962
63.841
56.300
30.921
18.889
200
180
160
140
120
100
80
60
40
20
ppm
N
N
O
OH

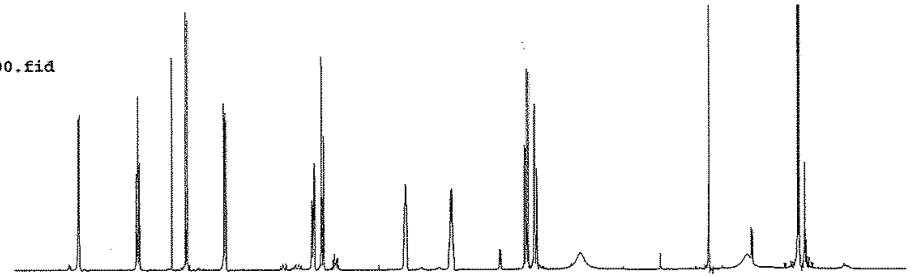
STANDARD PROTON PARAMETERS

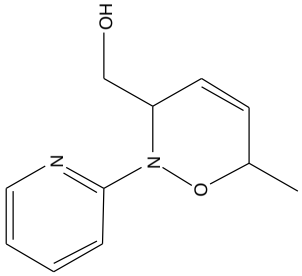
File: home/kmn/Desktop/ahn-500/April2_2012_fraction1_gHMBC-500.fid

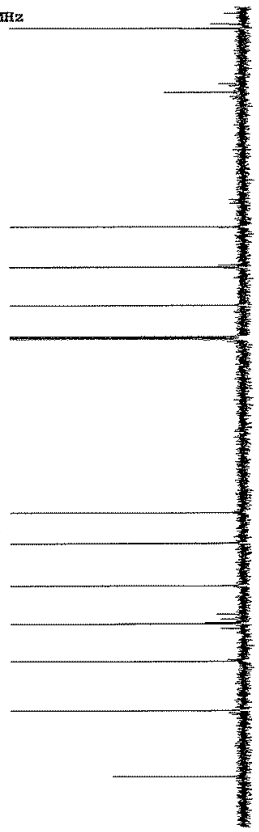
Pulse Sequence: gHMBC

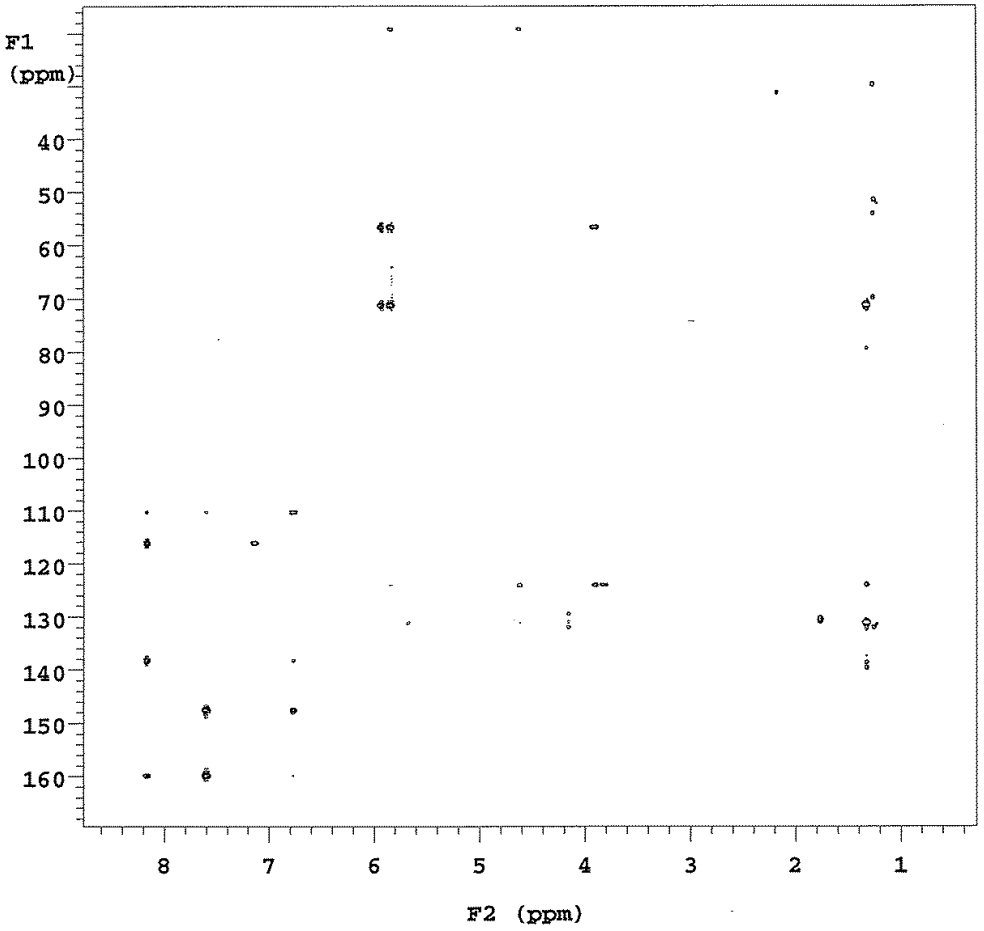
Temp. 25.0 C / 298.1 K
Operator: kmnchem

Relax. delay 1.000 sec
Acq. time 0.150 sec
Width 4664.2 Hz
2D Width 30165.9 Hz
32 repetitions
2 x 256 increments
OBSERVE H1, 499.8267329 MHz
DATA PROCESSING
Sq. sine bell 0.075 sec
F1 DATA PROCESSING
Sq. sine bell 0.027 sec
Shifted by -0.027 sec
FT size 2048 x 4096
Total time 5 hr, 43 min

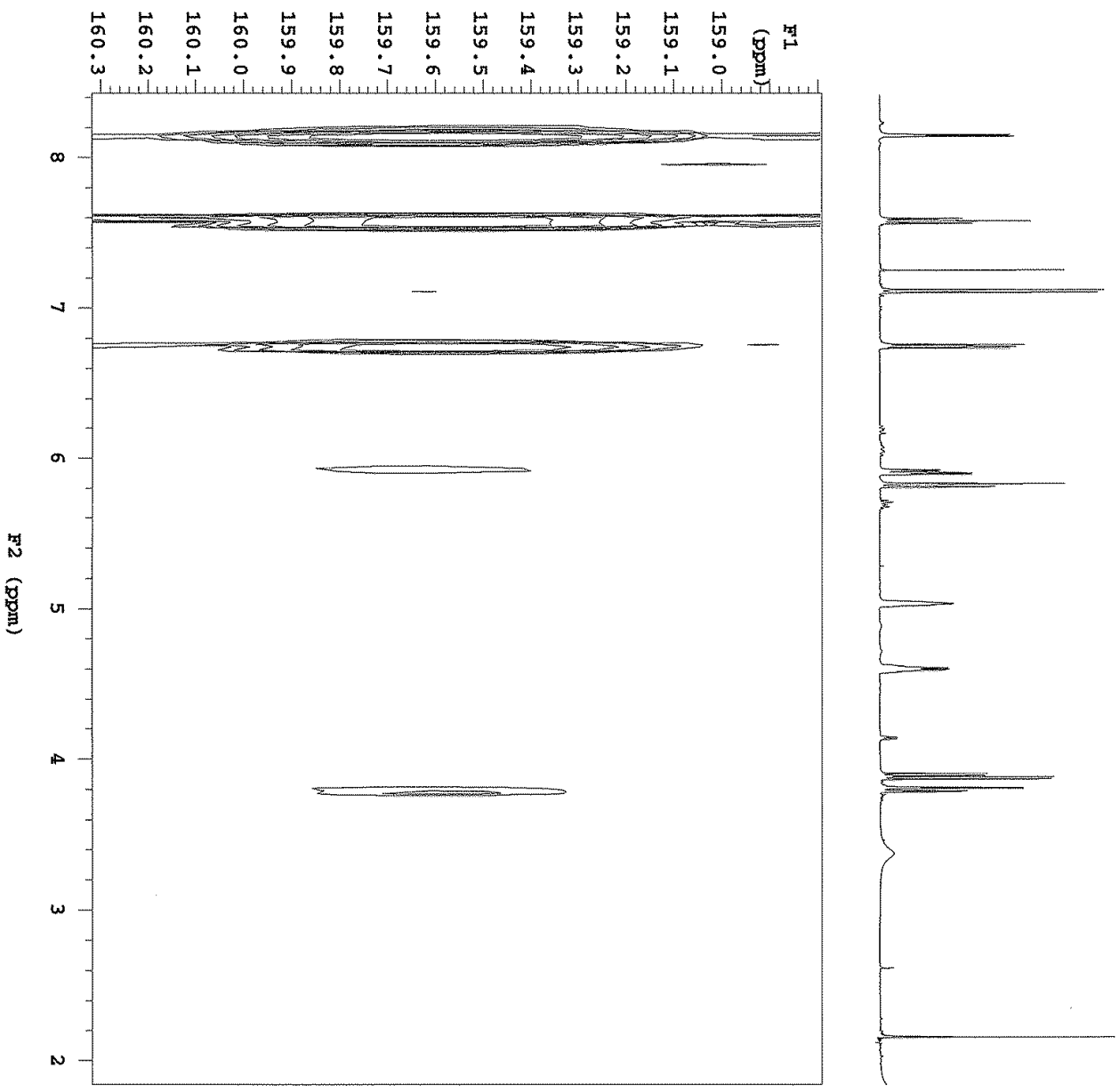


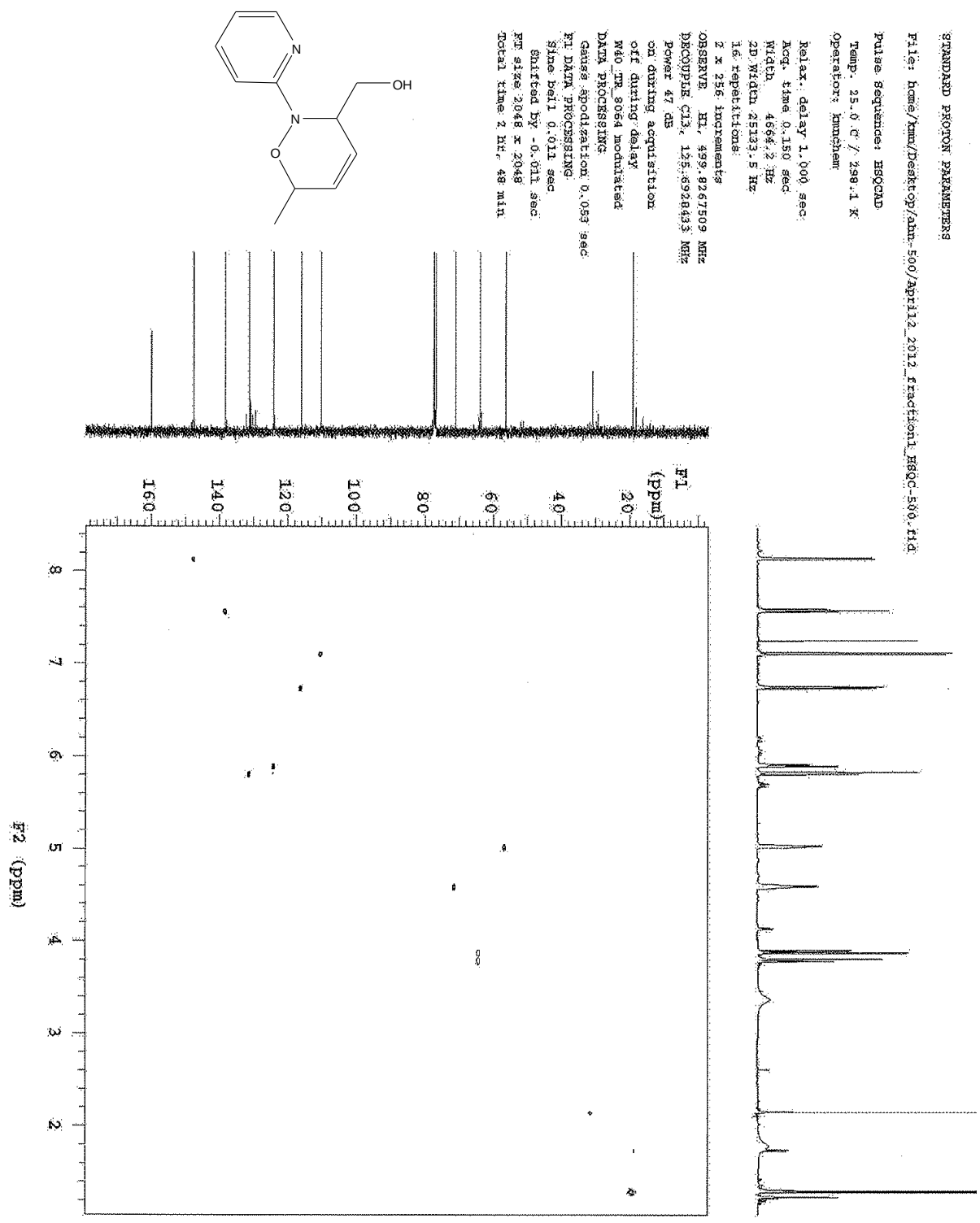






CCC


F1 (ppm)
160.3
160.2
160.1
160.0
159.9
159.8
159.7
159.6
159.5
159.4
159.3
159.2
159.1
159.0
F2 (ppm)
8
7
6
5
4
3
2


STANDARD PROTON PARAMETERS
File: home/kmm/Desktop/abn-500/April2_2012_fraction1_HSQC-500.fid
Pulse Sequence: HSQCAD
Temp: 25.0 C / 298.1 K
Operator: kmmchem
Relax. delay 1.000 sec
Acq. time 0.150 sec
Width 4664.2 Hz
2D Width 25133.5 Hz
16 repetitions
2 x 256 increments
OBSERVE H1, 499.8267509 MHz
DECOUPLE C13, 125.692843 MHz
Power 47 dB
on during acquisition
off during delay
W40.TR.8064 modulated
DATA PROCESSING
Gauss apodization 0.053 sec
F1 DATA PROCESSING
Sine bell 0.011 sec
Shifted by -0.011 sec
FT size 2048 x 2048
Total time 2 hr, 48 min
N
O
N
OH
F1 (ppm)
F2 (ppm)

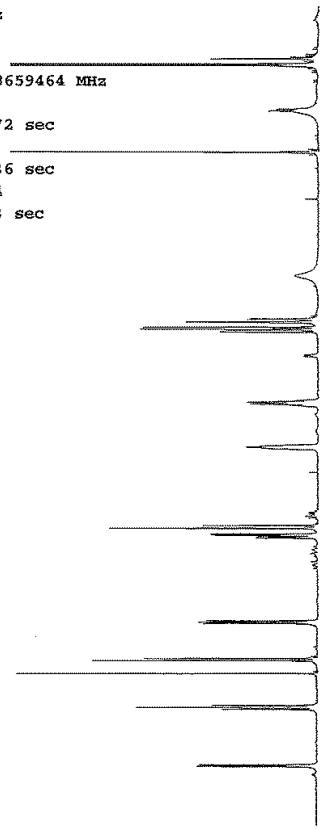
STANDARD PROTON PARAMETERS
Fraction 1
KMN-AHN
March 29,2012

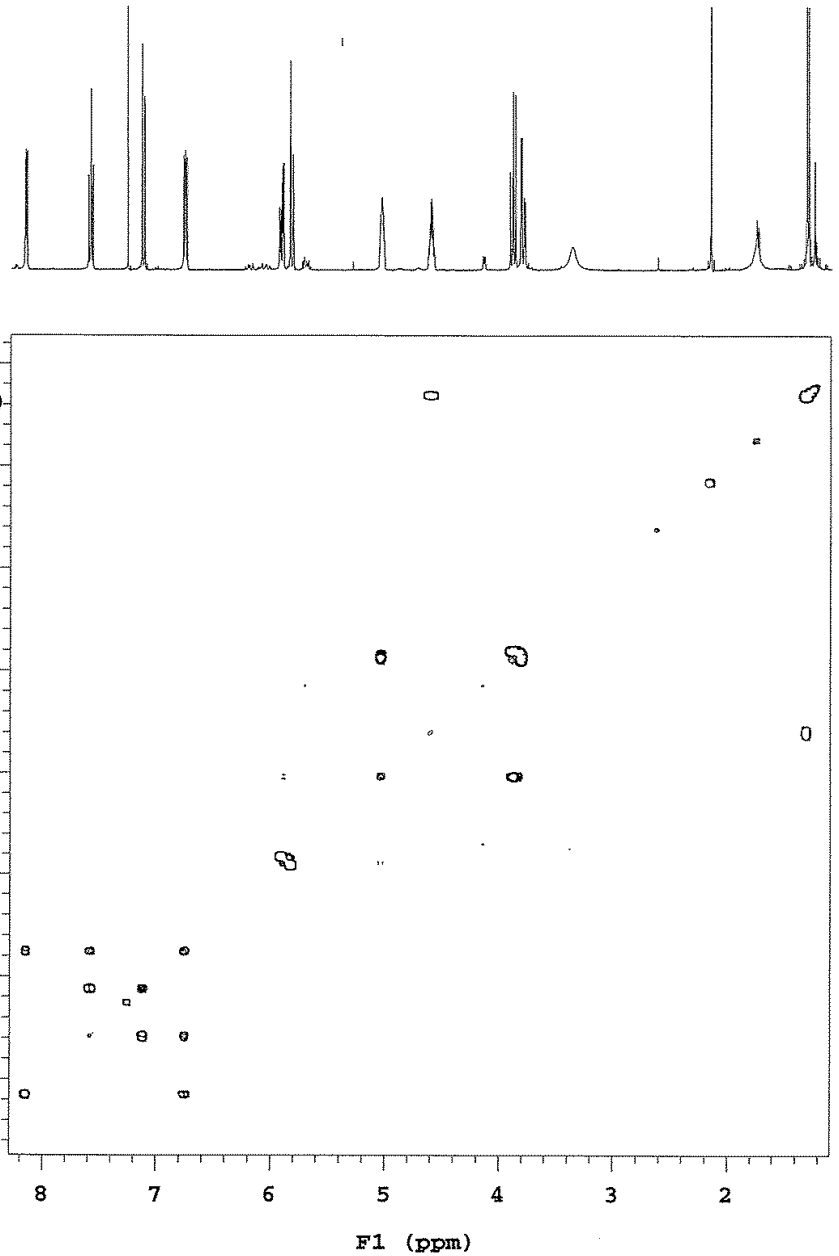
File: home/kmn/Desktop/ahn-500/fraction1_gCOSY_400.fid

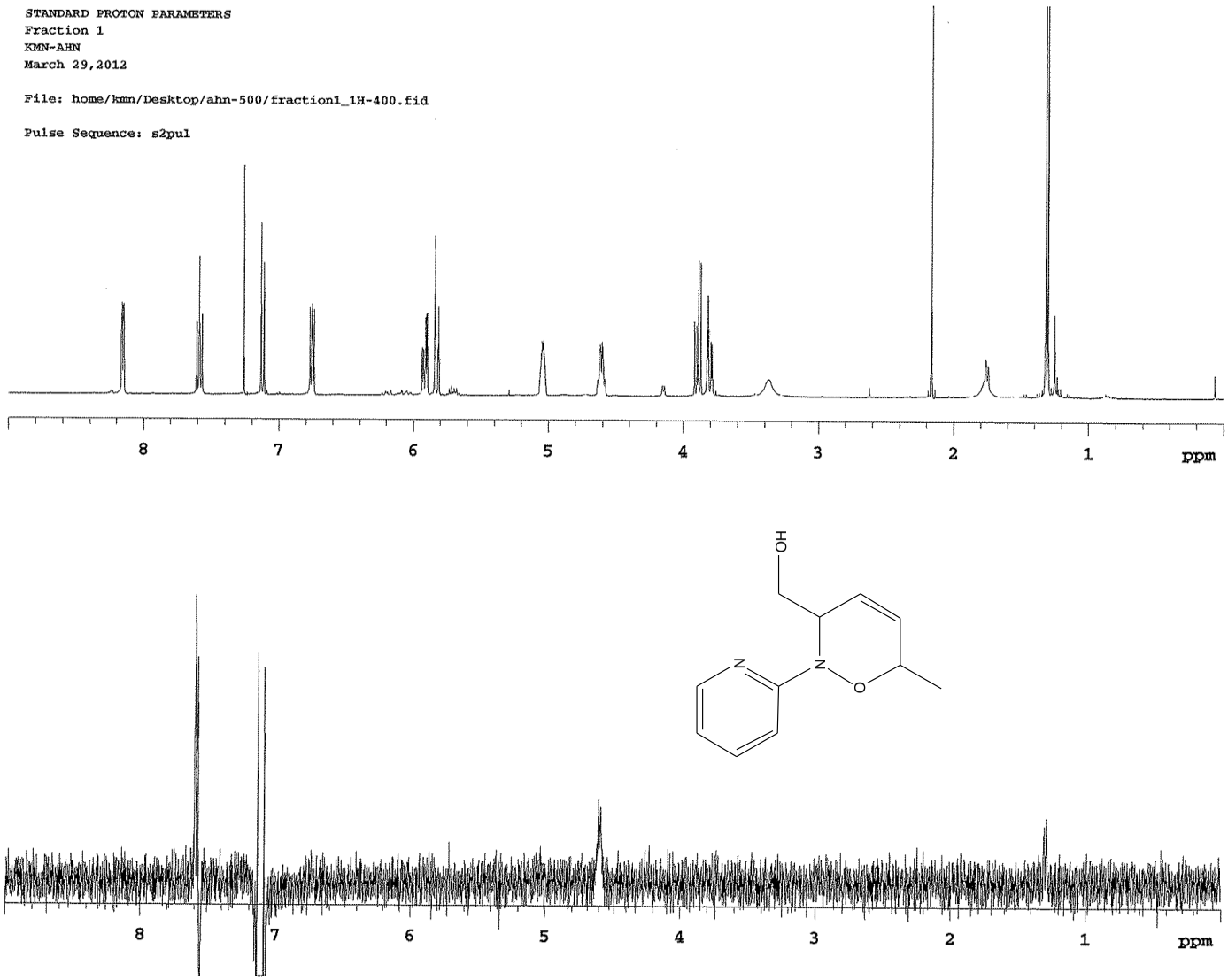
Pulse Sequence: gCOSY

Temp. 25.0 C / 298.1 K
Operator: kmn

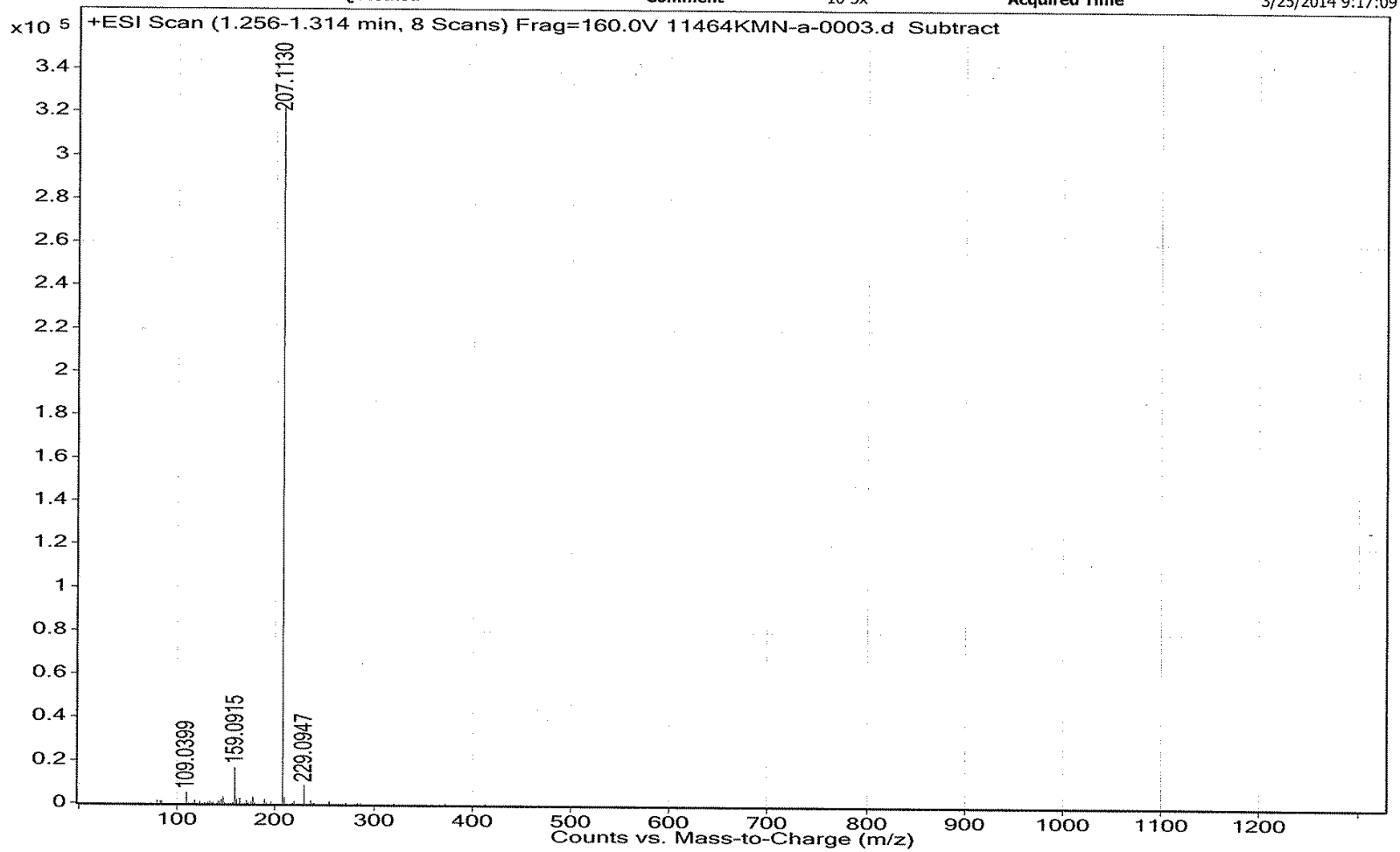
Relax. delay 1.000 sec
Acq. time 0.150 sec
Width 3571.4 Hz
2D Width 3571.4 Hz
Single scan
128 increments
OBSERVE H1, 399.8659464 MHz
DATA PROCESSING
Sq. sine bell 0.072 sec
F1 DATA PROCESSING
Sq. sine bell 0.036 sec
FT size 1024 x 1024
Total time 3 min 13 sec



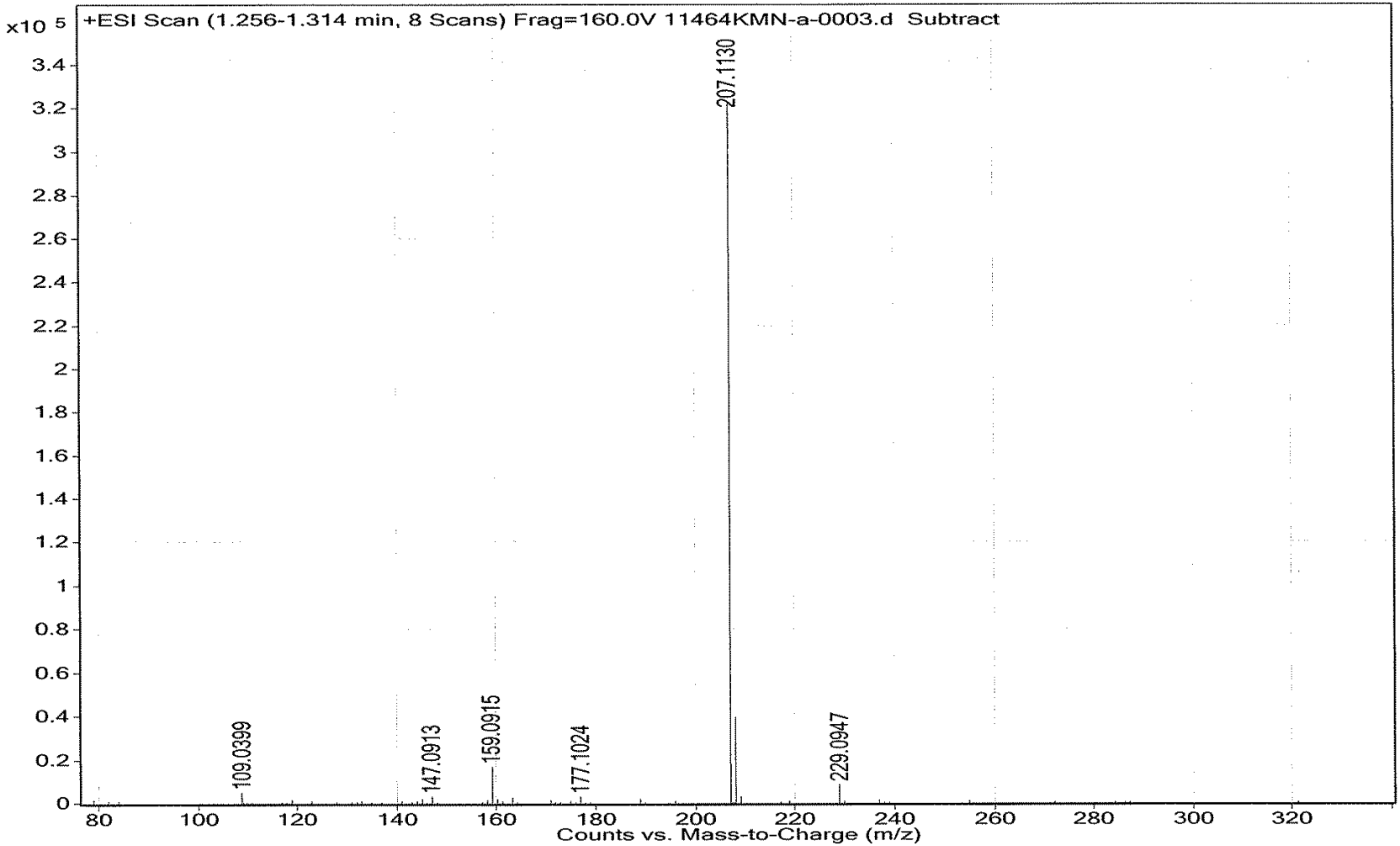



STANDARD PROTON PARAMETERS
Fraction 1
KMN-AHN
March 29,2012
File: home/kmn/Desktop/ahn-500/fraction1_1H-400.fid
Pulse Sequence: s2pul
8
7
6
5
4
3
2
1
ppm
OH
N
N
O
8
7
6
5
4
3
2
1
ppm

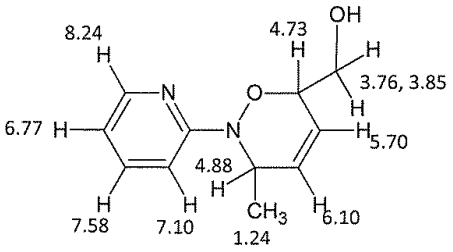
Sample Name	anhtran-240	**Position**	P1-E1	**Instrument Name**	Instrument MS	**User Name**	Foster
Inj Vol	5	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	11464KMN-a-0003.d	**ACQ Method**		**Comment**	10-3x	**Acquired Time**	3/25/2014 9:17:09 A

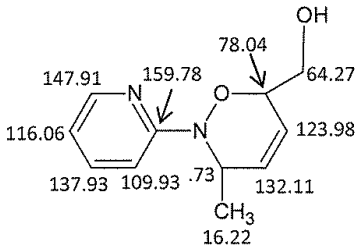


Sample Name	anhtran-240	**Position**	P1-E1	**Instrument Name**	Instrument MS	**User Name**	Foster
Inj Vol	5	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	11464KMN-a-0003.d	**ACQ Method**		**Comment**	10-3x	**Acquired Time**	3/25/2014 9:17:09 AM



Summary NMR of (3-methyl-2-(pyridin-2-yl)-3,6-dihydro-2H-1,2-oxazin-6-yl)methanol (Proximal 2)





Fraction 2

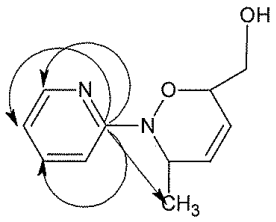
Carbon	mult	Proton (HSQC)	Mult (J Hz)	COSY	Proton(HBMC)	NOE
16.22	CH_3	1.24	d(6.7)	4.88	4.88	
51.47	CH	4.88	m	1.24	6.10, 5.70	
64.27	CH_2	3.76	dd(12.2, 6.5)	3.85		
64.27	CH_2	3.85	dd(12.2, 3.0)	3.76	5.70	
78.04	CH	4.73	m	3.85, 3.76	6.10, 5.70, 3.85	
109.93	CH	7.10	d(7.8)	7.58	8.24, 7.58, 6.77	7.58, 4.88, 4.73, 1.24
116.06	CH	6.77	dd(7.8, 5.06)	8.24, 7.58	8.24, 7.10	
123.98	CH	5.70	dt(10.2, 1.3)	6.10	4.73, 3.85, 3.76, 1.24	
132.11	CH	6.10	ddd(10.2, 4.8, 2.1)	5.70	4.73, 1.24	
137.93	CH	7.58	dd(7.8, 7.8, 1.8)	7.10, 6.77	8.24, 6.77	
147.91	CH	8.24	dd (5.06, 1.8)	6.77	7.58, 6.77	
159.78	C	---			8.24, 7.58, 6.77, 1.24	

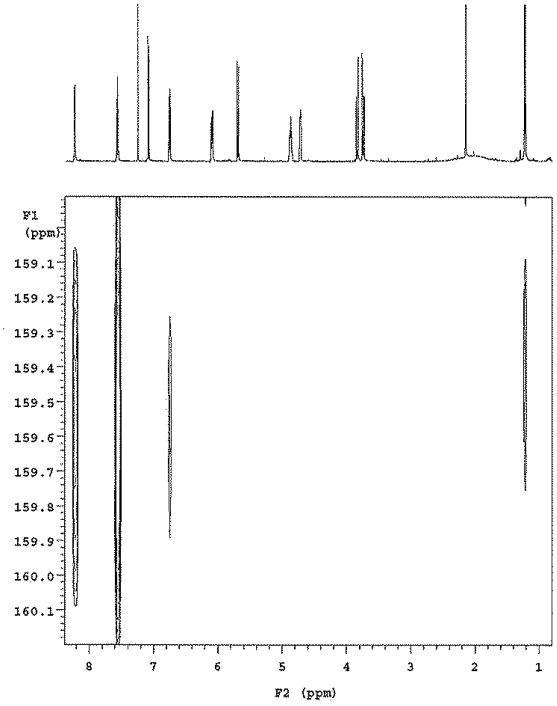
Fraction 2

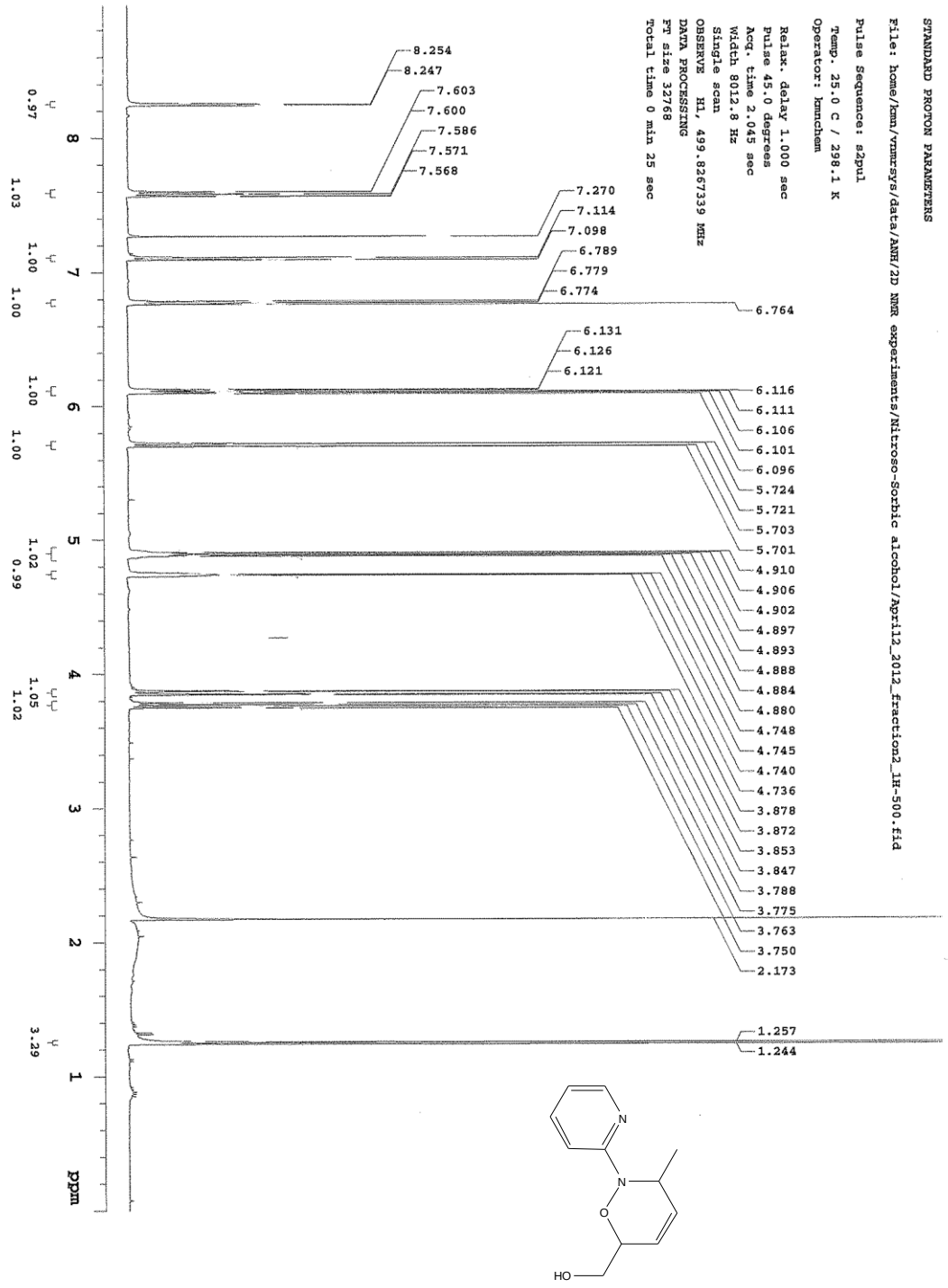
Nitroso-Sorbic- alcohol

gHMBC

Through Bond C-H long range coupling






STANDARD PROTON PARAMETERS
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Pulse Sequence: s2pul
Temp. 25.0 C / 298.1 K
Operator: kmmchem
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.045 sec
Width 8012.8 Hz
Single scan
OBSERVE H1, 499.8267339 MHz
DATA PROCESSING
FT size 32768
Total time 0 min 25 sec
8.254
8.247
7.603
7.600
7.586
7.571
7.568
7.270
7.114
7.098
6.789
6.779
6.774
6.764
6.131
6.126
6.121
6.116
6.111
6.106
6.101
6.096
5.724
5.721
5.703
5.701
4.910
4.906
4.902
4.897
4.893
4.888
4.884
4.880
4.748
4.745
4.740
4.736
3.878
3.872
3.853
3.847
3.788
3.775
3.763
3.750
2.173
1.257
1.244
0.97
1.03
1.00
1.00
1.00
1.00
1.02
0.99
1.05
1.02
3.29
ppm
N
N
O
HO

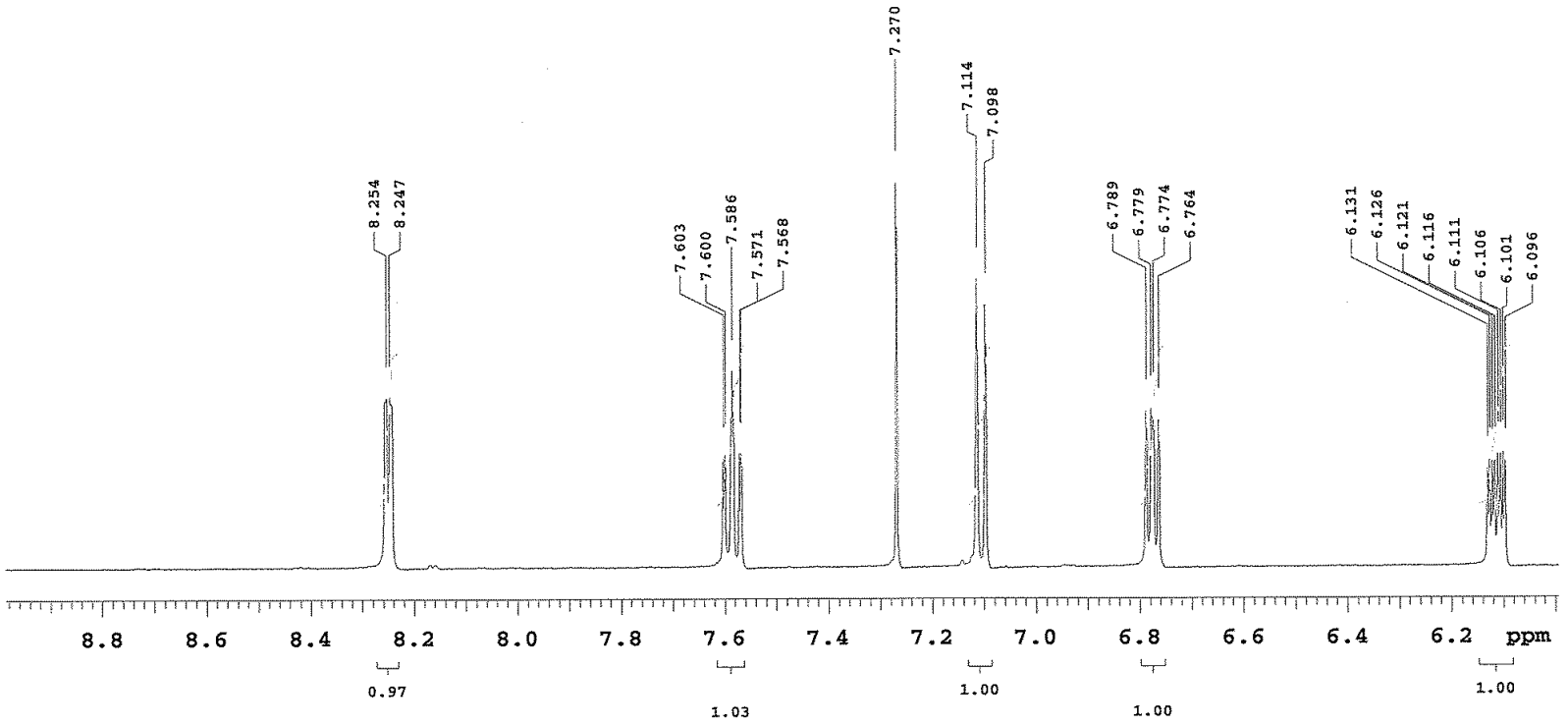
STANDARD PROTON PARAMETERS

File: home/kmn/vnmrsys/data/ANH/2D NMR experiments/Nitroso-Sorbic alcohol/April2_2012_fraction2_1H-500.fid

Pulse Sequence: s2pul

Temp. 25.0 C / 298.1 K
Operator: kmnchem

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.045 sec
Width 8012.8 Hz
Single scan
OBSERVE H1, 499.8267339 MHz
DATA PROCESSING
FT size 32768
Total time 0 min 25 sec



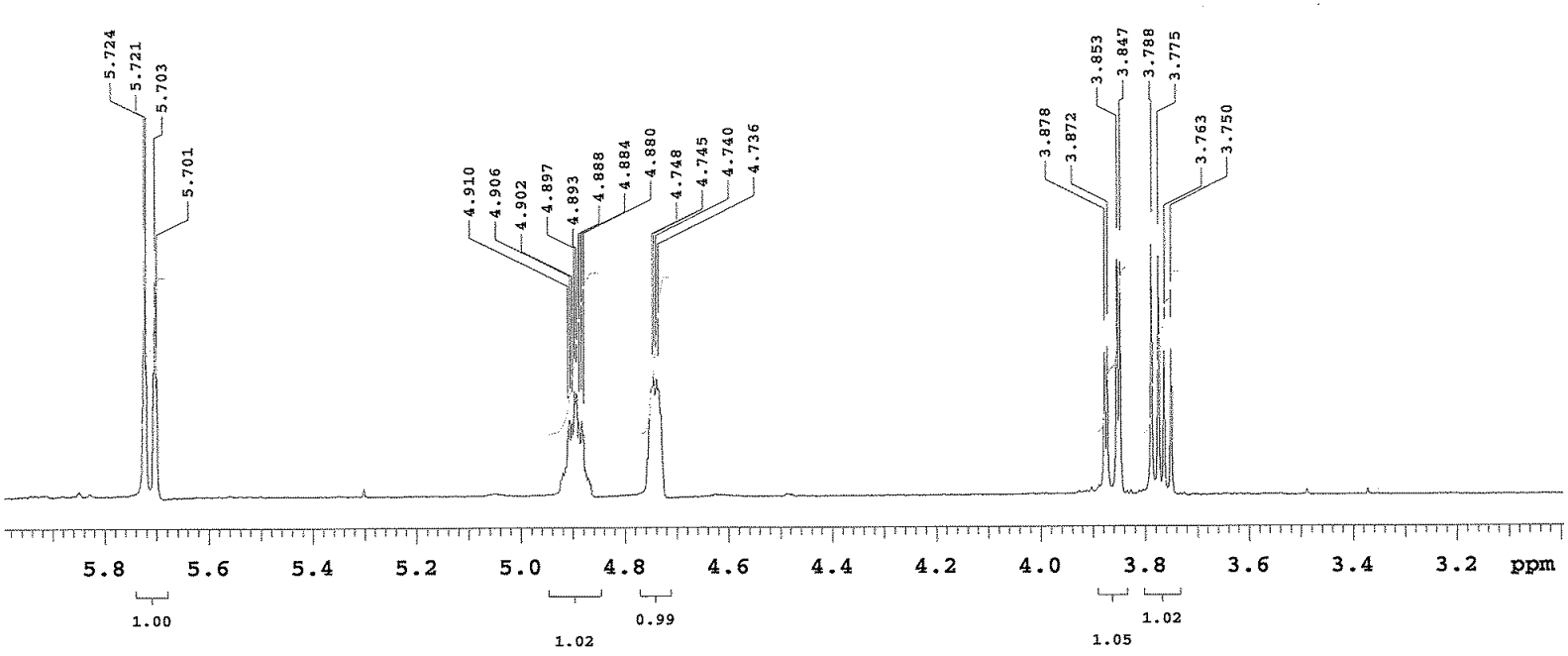
STANDARD PROTON PARAMETERS

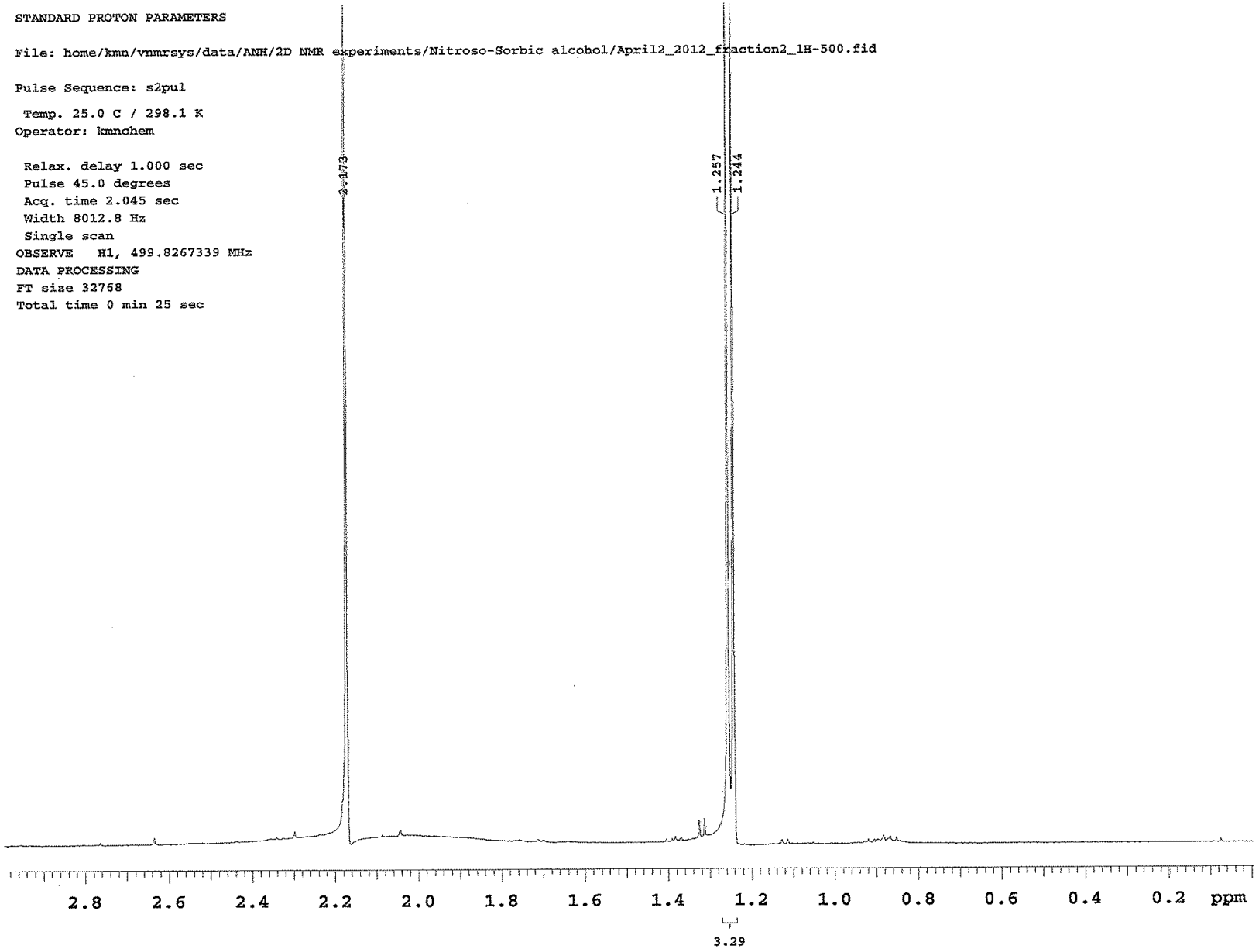
File: home/kmn/vnmrsys/data/ANH/2D NMR experiments/Nitroso-Sorbic alcohol/April2_2012_fraction2_1H-500.fid

Pulse Sequence: s2pul

Temp. 25.0 C / 298.1 K
Operator: kmnchem

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.045 sec
Width 8012.8 Hz
Single scan
OBSERVE H1, 499.8267339 MHz
DATA PROCESSING
FT size 32768
Total time 0 min 25 sec




STANDARD PROTON PARAMETERS
File: home/kmn/vnmrsys/data/ANH/2D NMR experiments/Nitroso-Sorbic alcohol/April2_2012_fraction2_1H-500.fid
Pulse Sequence: s2pul
Temp. 25.0 C / 298.1 K
Operator: kmnchem
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.045 sec
Width 8012.8 Hz
Single scan
OBSERVE H1, 499.8267339 MHz
DATA PROCESSING
FT size 32768
Total time 0 min 25 sec
2.173
1.257
1.244
2.8
2.6
2.4
2.2
2.0
1.8
1.6
1.4
1.2
1.0
0.8
0.6
0.4
0.2
ppm
3.29

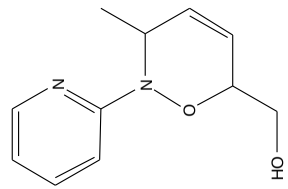
STANDARD CARBON PARAMETERS
Fraction 2
13C 500 NMR
April 2, 2012

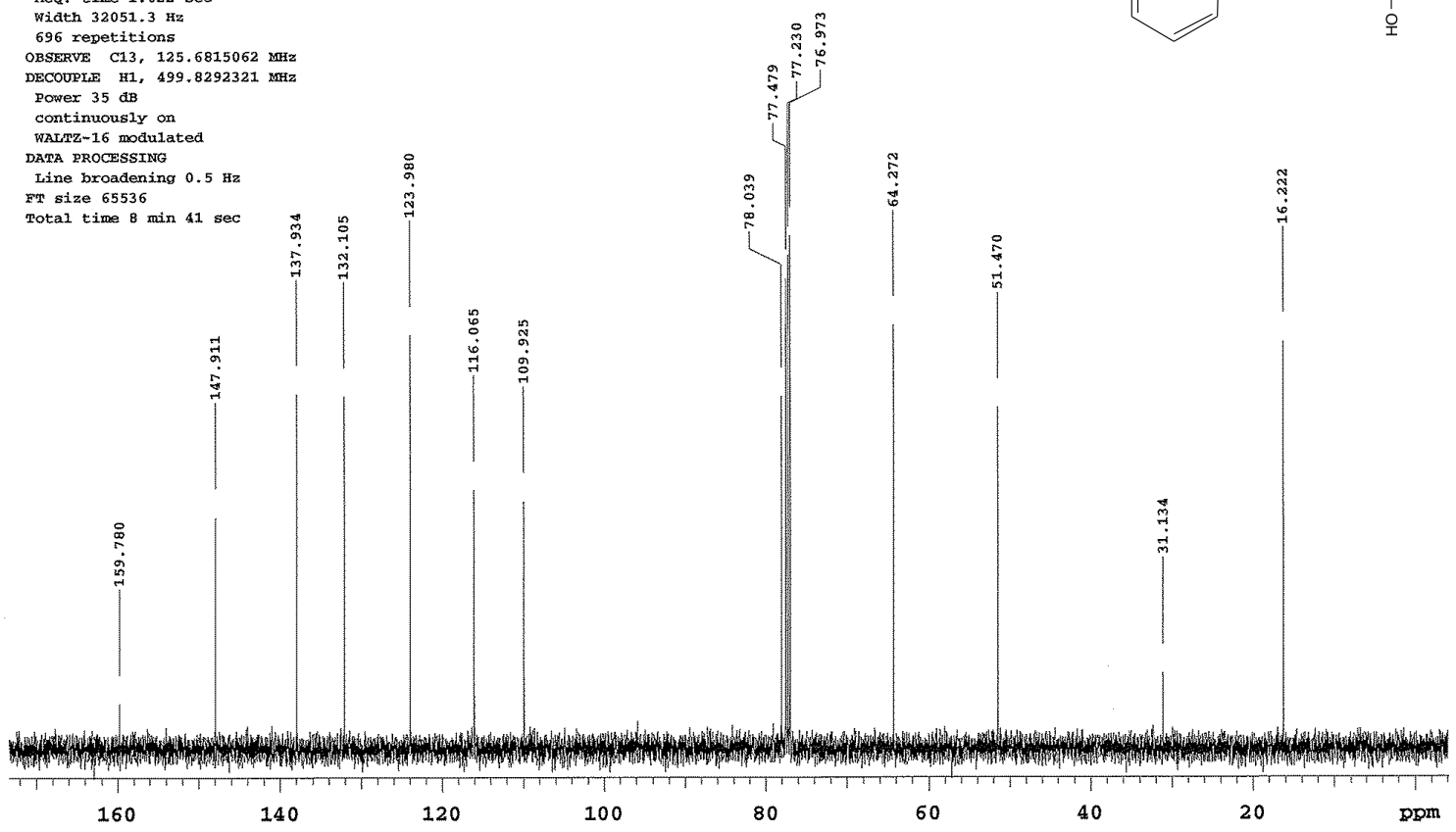
File: home/kmn/Desktop/ahn-500/April2_2012_fraction2_Carbon-500.fid

Pulse Sequence: s2pul

Temp. 25.0 C / 298.1 K
Operator: kmnchem

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.022 sec
Width 32051.3 Hz
696 repetitions
OBSERVE C13, 125.6815062 MHz
DECOUPLE H1, 499.8292321 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 8 min 41 sec





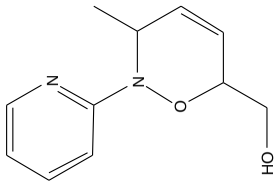
STANDARD PROTON PARAMETERS

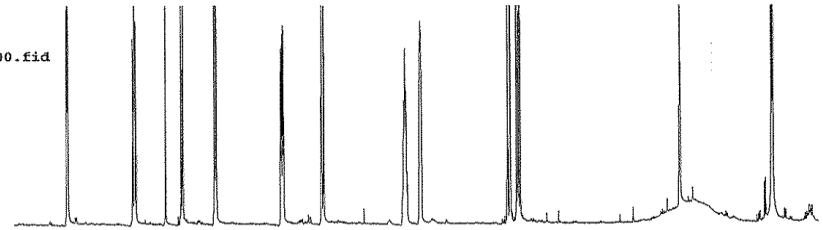
File: home/kmn/Desktop/ahn-500/April2_2012_fraction2_gHMBC-500.fid

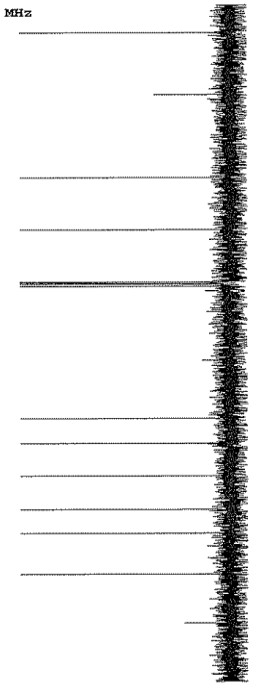
Pulse Sequence: gHMBC

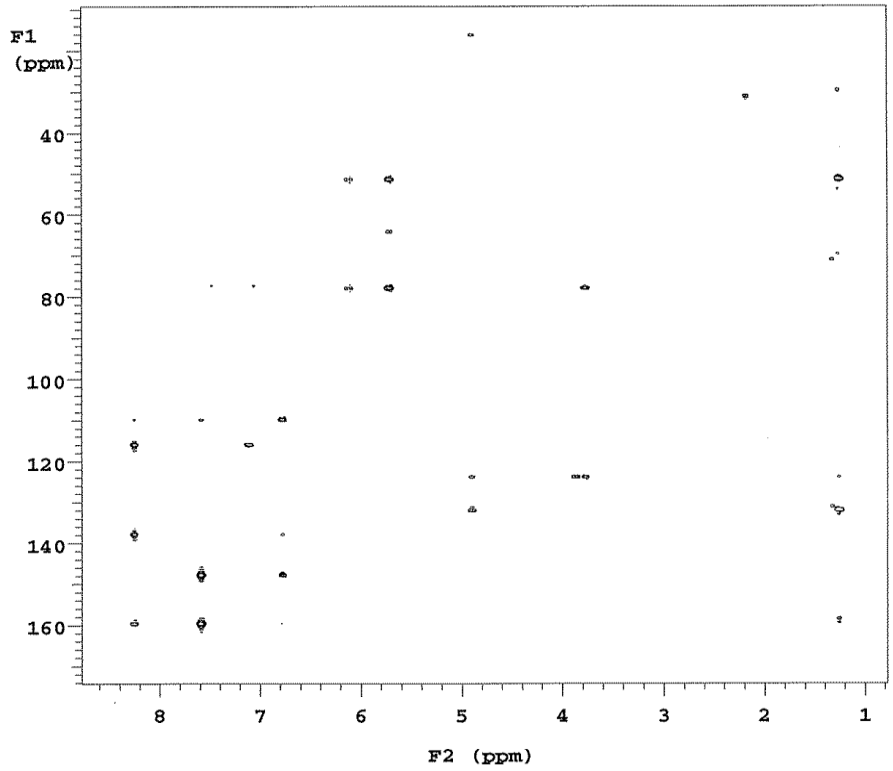
Temp. 25.0 C / 298.1 K
Operator: kmnchem

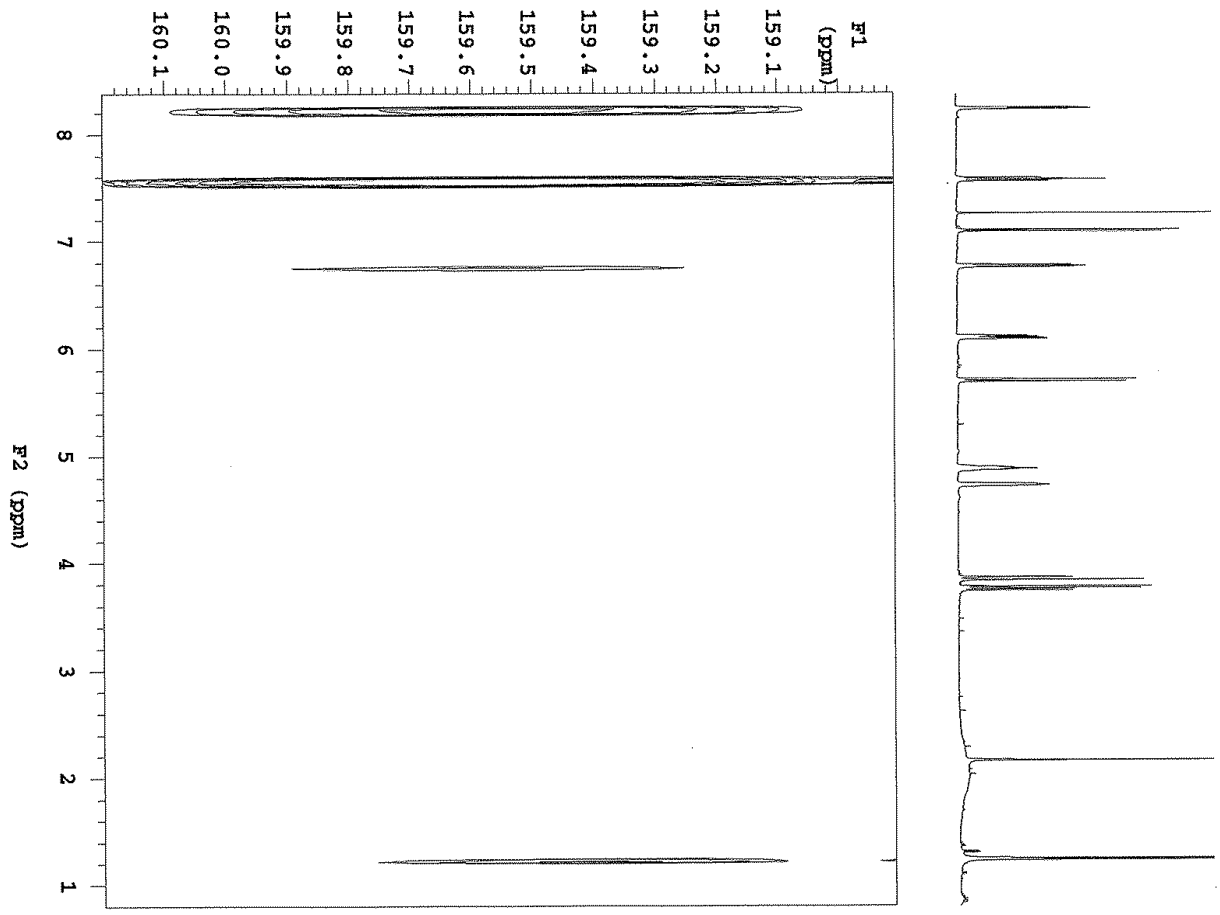
Relax. delay 1.000 sec
Acq. time 0.150 sec
Width 4166.7 Hz
2D Width 30165.9 Hz
16 repetitions
2 x 256 increments
OBSERVE H1, 499.8267329 MHz
DATA PROCESSING
Sq. sine bell 0.061 sec
F1 DATA PROCESSING
Sq. sine bell 0.027 sec
Shifted by -0.027 sec
FT size 1024 x 4096
Total time 2 hr, 51 min

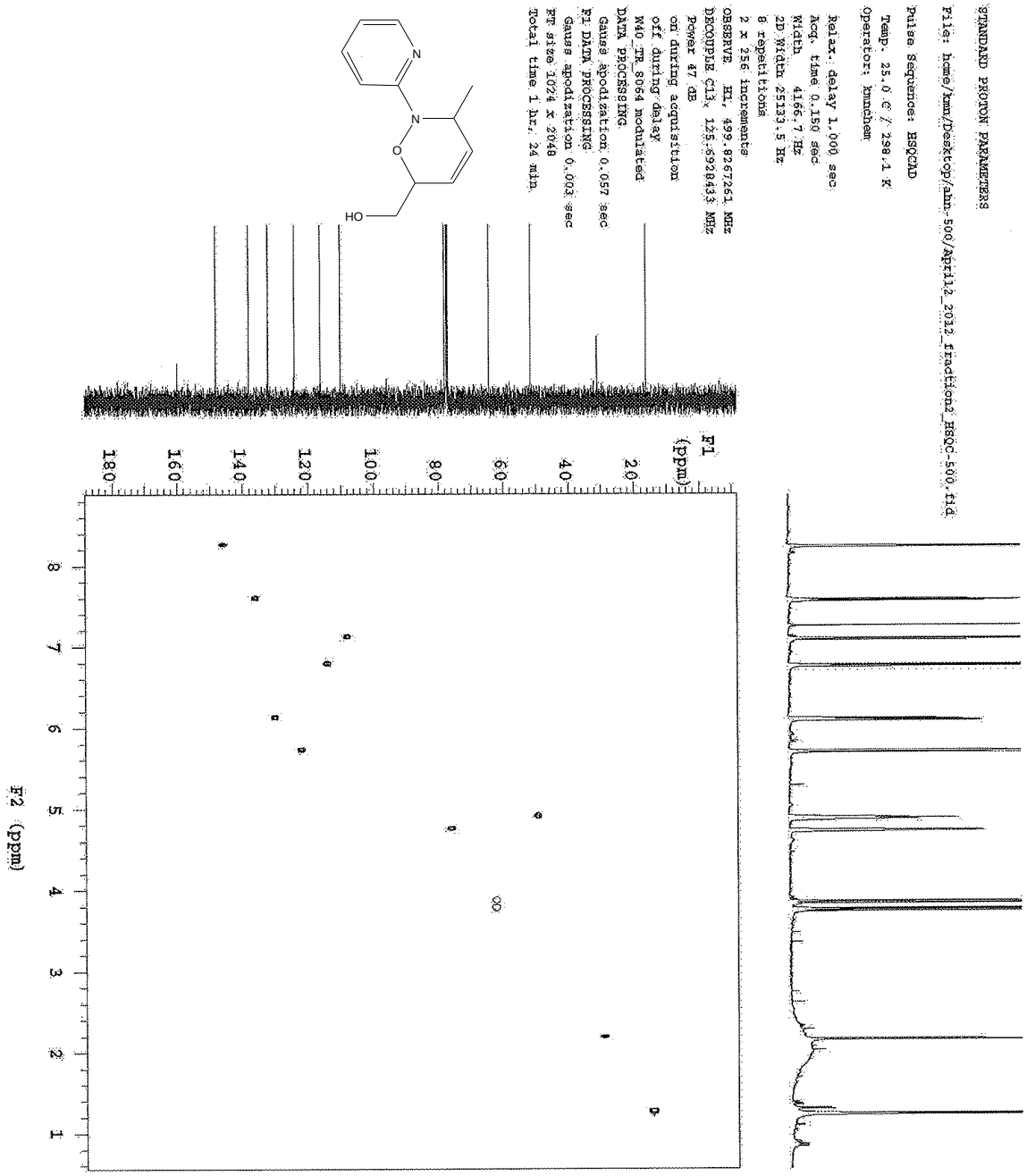


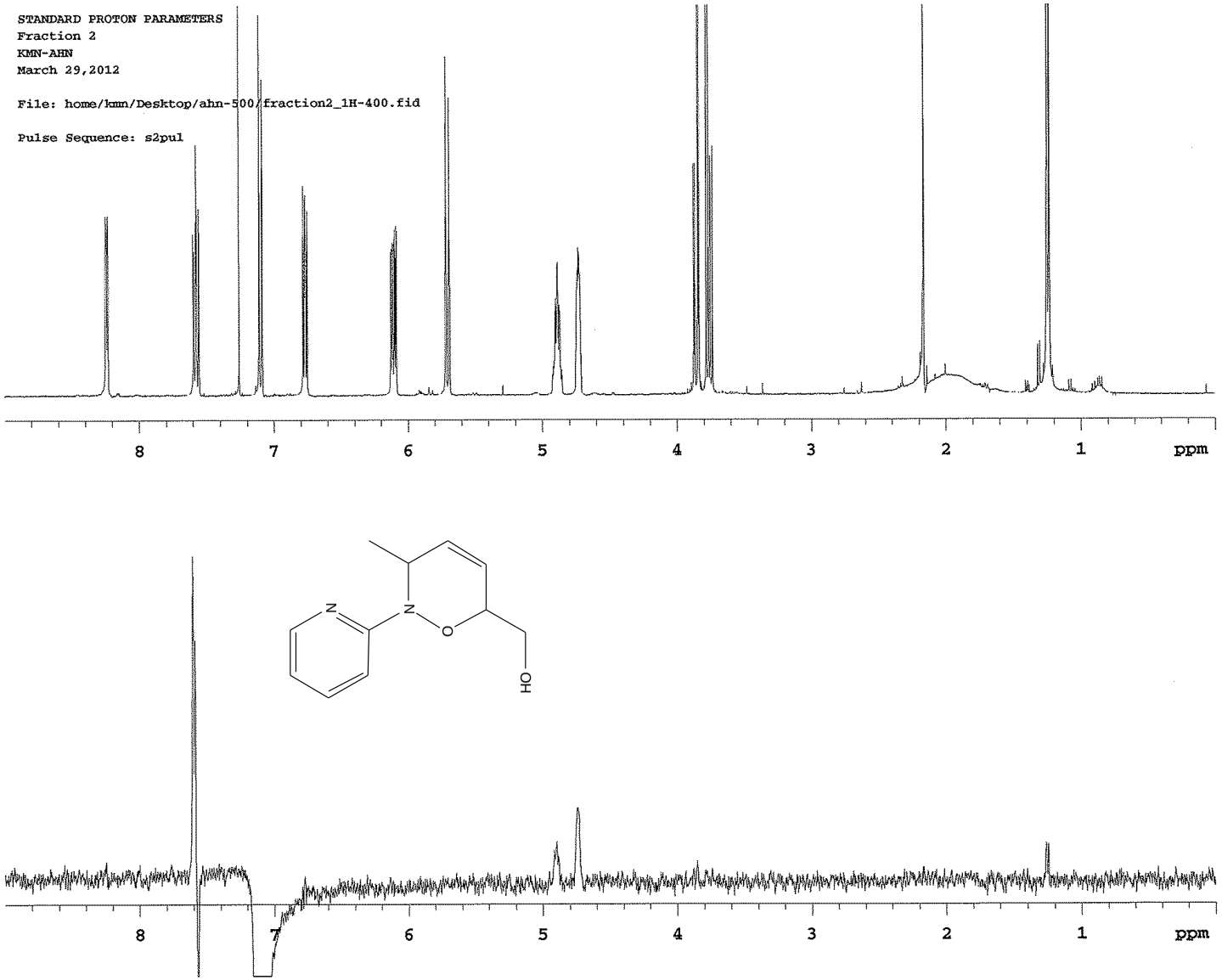


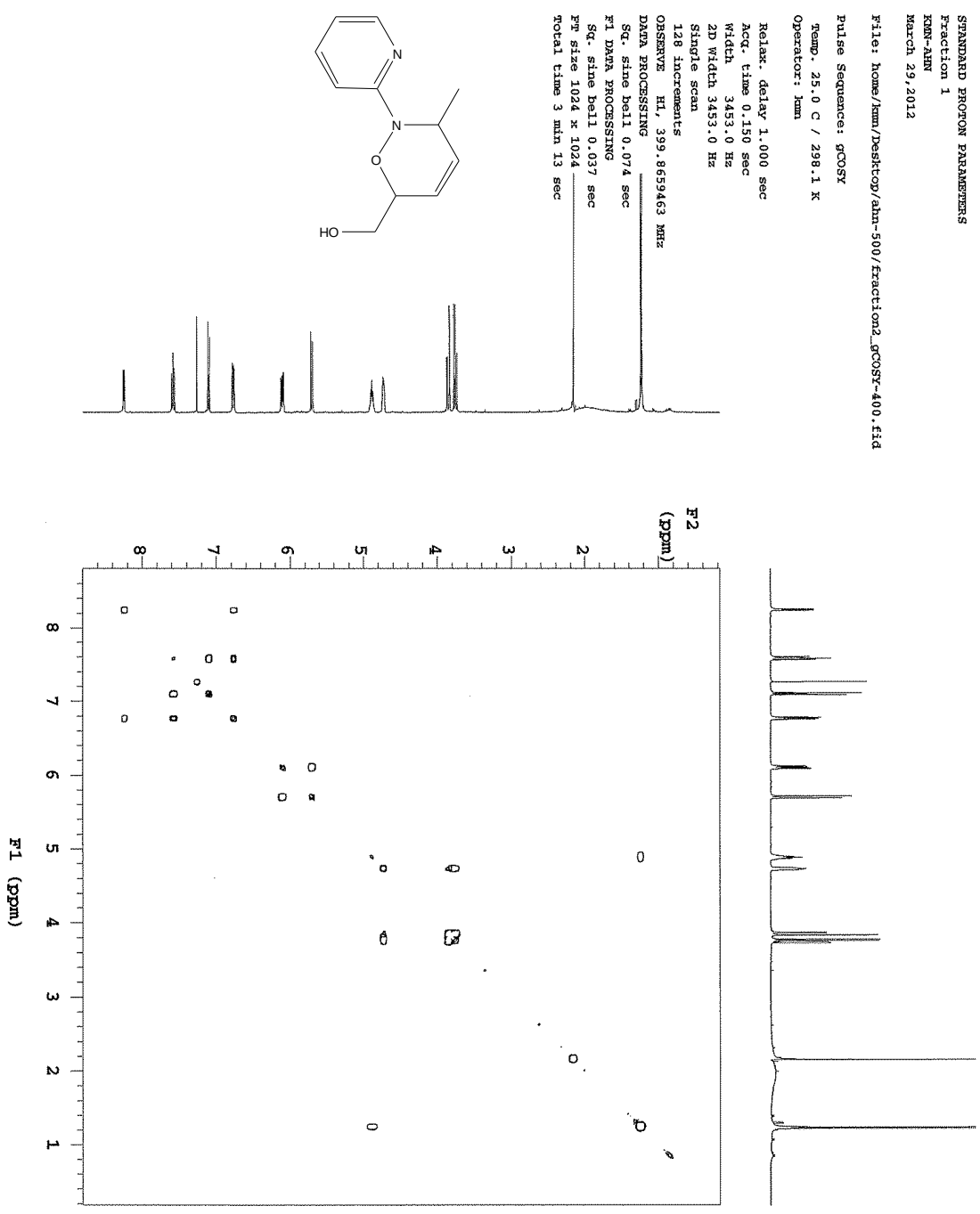




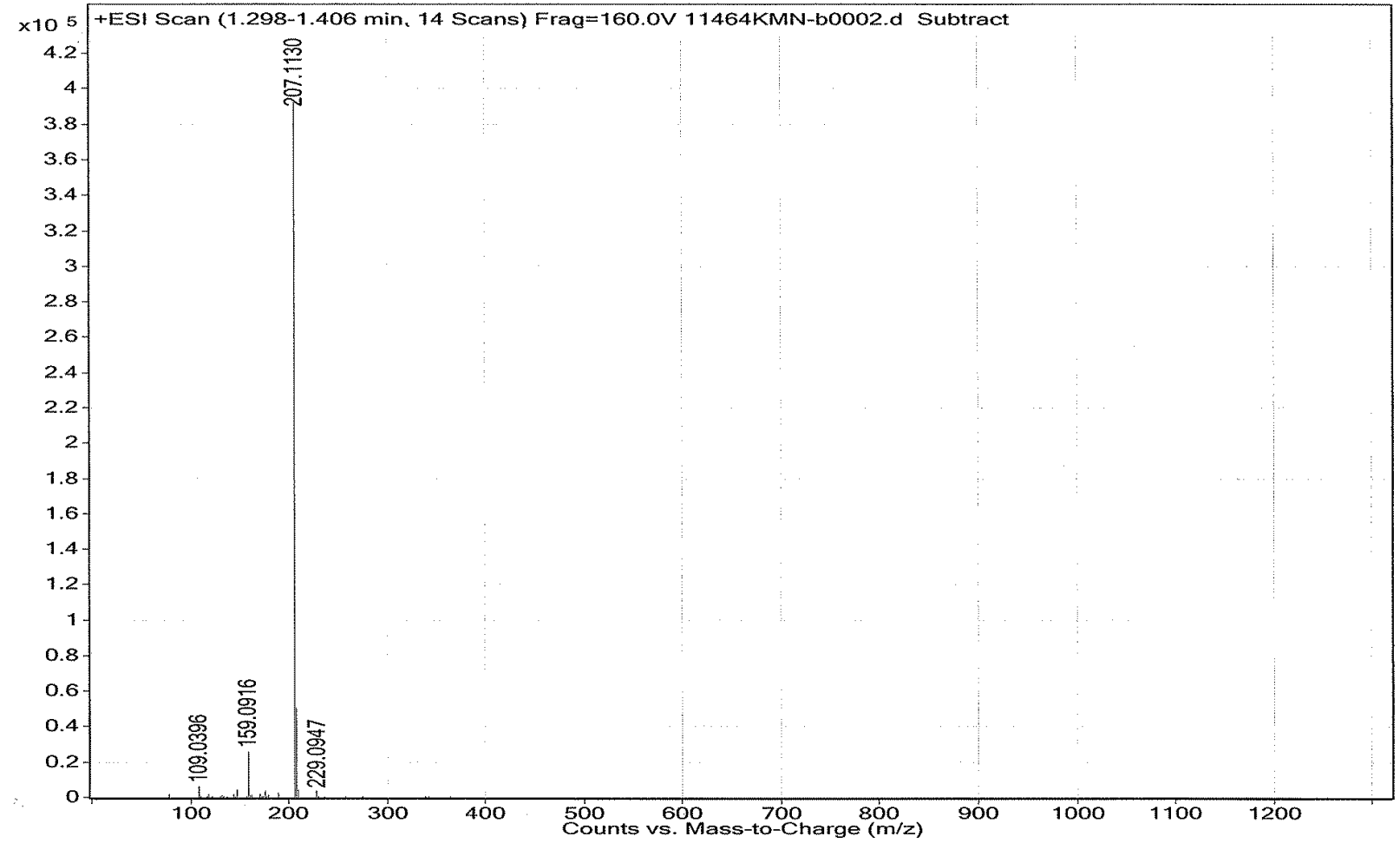

F1 (ppm)
160.1
160.0
159.9
159.8
159.7
159.6
159.5
159.4
159.3
159.2
159.1
F2 (ppm)
8
7
6
5
4
3
2
1


STANDARD PROTON PARAMETERS
File: home/hmm/Desktop/ahm-500/April2_2012_fraction2_HSQC-500.fid
Pulse Sequence: HSQCAD
Temp. 25.0 C / 298.1 K
Operator: hmmcbem
Relax. delay 1.000 sec
Acq. time 0.150 sec
Width 4166.7 Hz
2D Width 25133.5 Hz
8 repetitions
2 x 256 increments
OBSERVE H1, 499.8267261 MHz
DECOUPLE c13, 125.6920433 MHz
Power 47 dB
on during acquisition
off during delay
W40_TR_8064 modulated
DATA PROCESSING
Gauss apodization 0.057 sec
F1 DATA PROCESSING
Gauss apodization 0.003 sec
FT size 1024 x 2048
Total time 1 hr, 24 min
N
N
O
HO
F1 (ppm)
F2 (ppm)

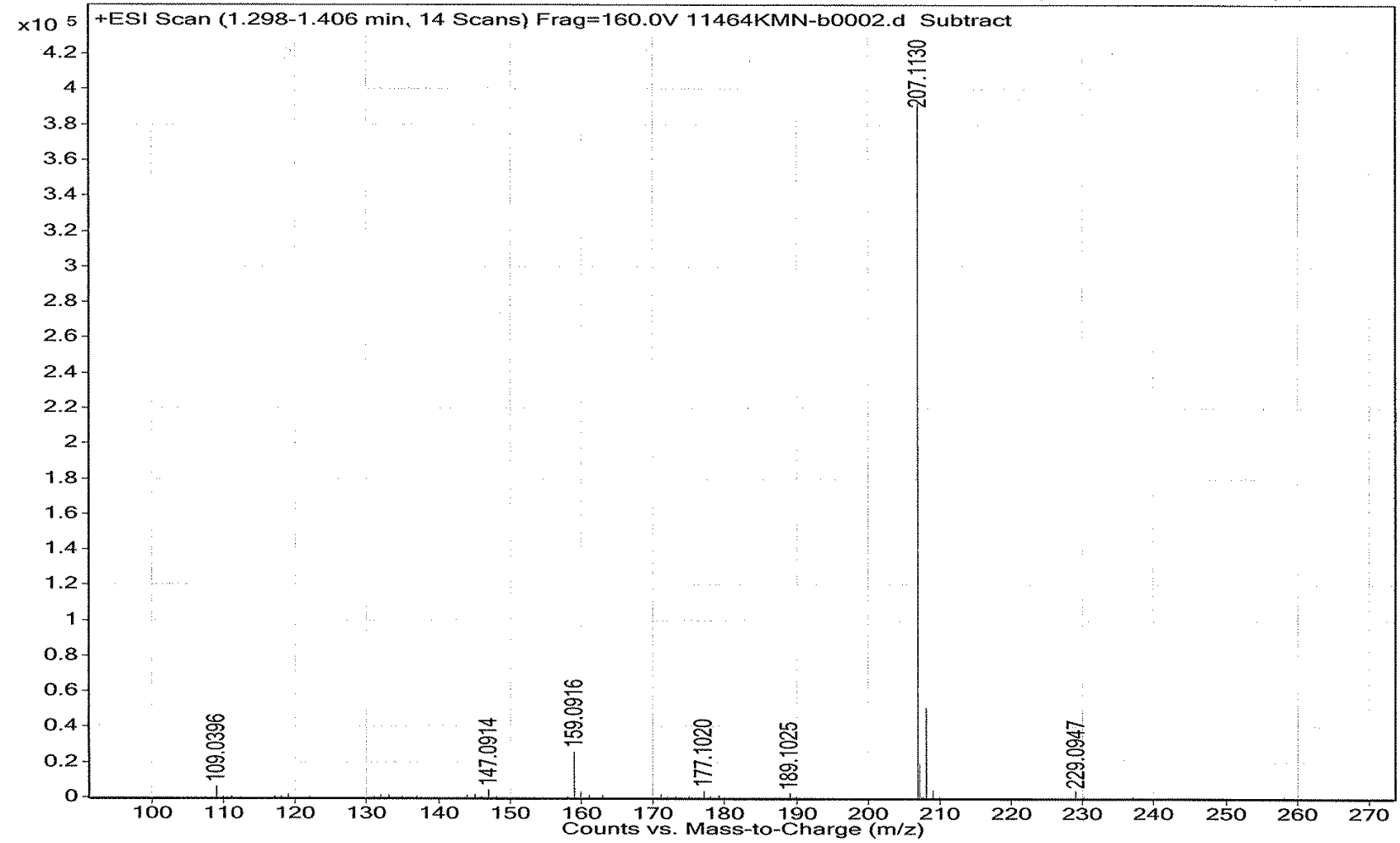

STANDARD PROTON PARAMETERS
Fraction 2
KMN-AHN
March 29,2012
File: home/kmn/Desktop/ahn-500/fraction2_1H-400.fid
Pulse Sequence: s2pul
8
7
6
5
4
3
2
1
ppm
N
N
O
HO
8
7
6
5
4
3
2
1
ppm


STANDARD PROTON PARAMETERS
Fraction 1
KMN-AHN
March 29,2012
File: home/kmn/desktop/ahn-500/fraction2_gCOSY-400.fid
Pulse Sequence: gCOSY
Temp. 25.0 C / 298.1 K
Operator: kmn
Relax. delay 1.000 sec
Acq. time 0.150 sec
Width 3453.0 Hz
2D Width 3453.0 Hz
Single scan
128 increments
OBSERVE H1, 399.8659463 MHz
DATA PROCESSING
Sq. sine bell 0.074 sec
F1 DATA PROCESSING
Sq. sine bell 0.037 sec
FT size 1024 x 1024
Total time 3 min 13 sec
N
N
O
HO
F2 (ppm)
F1 (ppm)

Sample Name	anhtran-241	**Position**	P1-E2	**Instrument Name**	Instrument MS	**User Name**	Foster
Inj Vol	5	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	11464KMN-b0002.d	**ACQ Method**		**Comment**	10-3x	**Acquired Time**	3/25/2014 9:36:58 AM

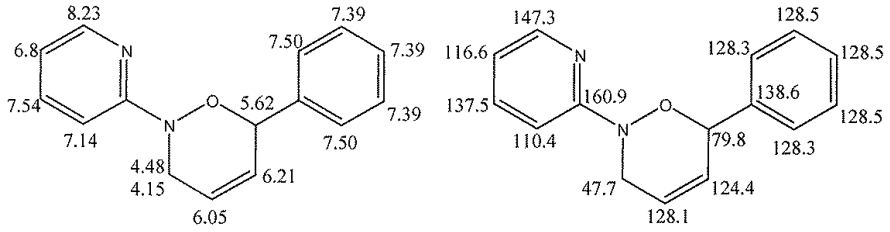


Sample Name	anhtran-241	**Position**	P1-E2	**Instrument Name**	Instrument MS	**User Name**	Foster
Inj Vol	5	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	11464KMN-b0002.d	**ACQ Method**		**Comment**	10-3x	**Acquired Time**	3/25/2014 9:36:58 AM



Summary NMR of 6-phenyl-2-(pyridin-2-yl)-3,6-dihydro-2H-1,2-oxazine (Proximal **3**)

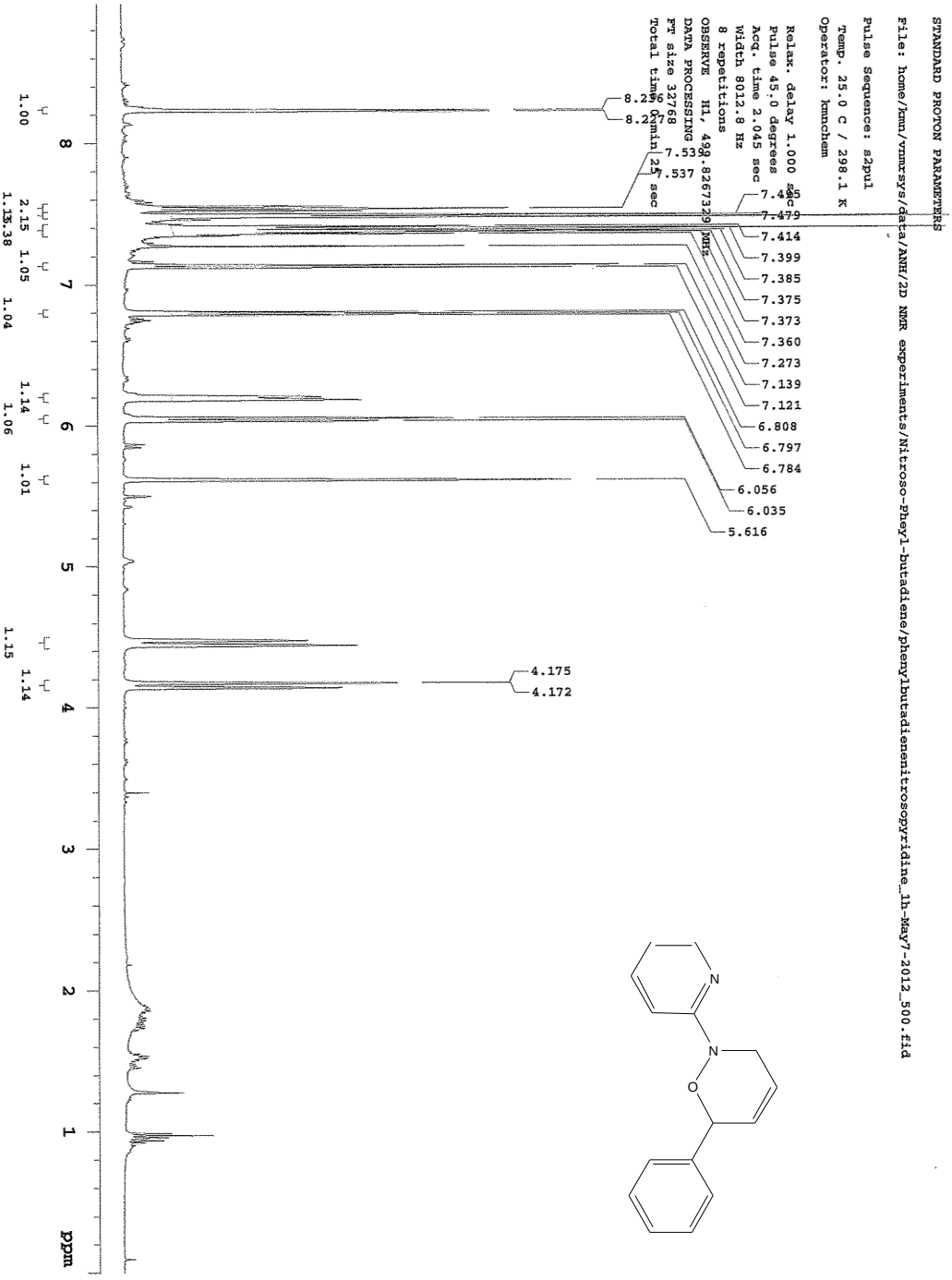
2-17-2012
KMN
Ahn

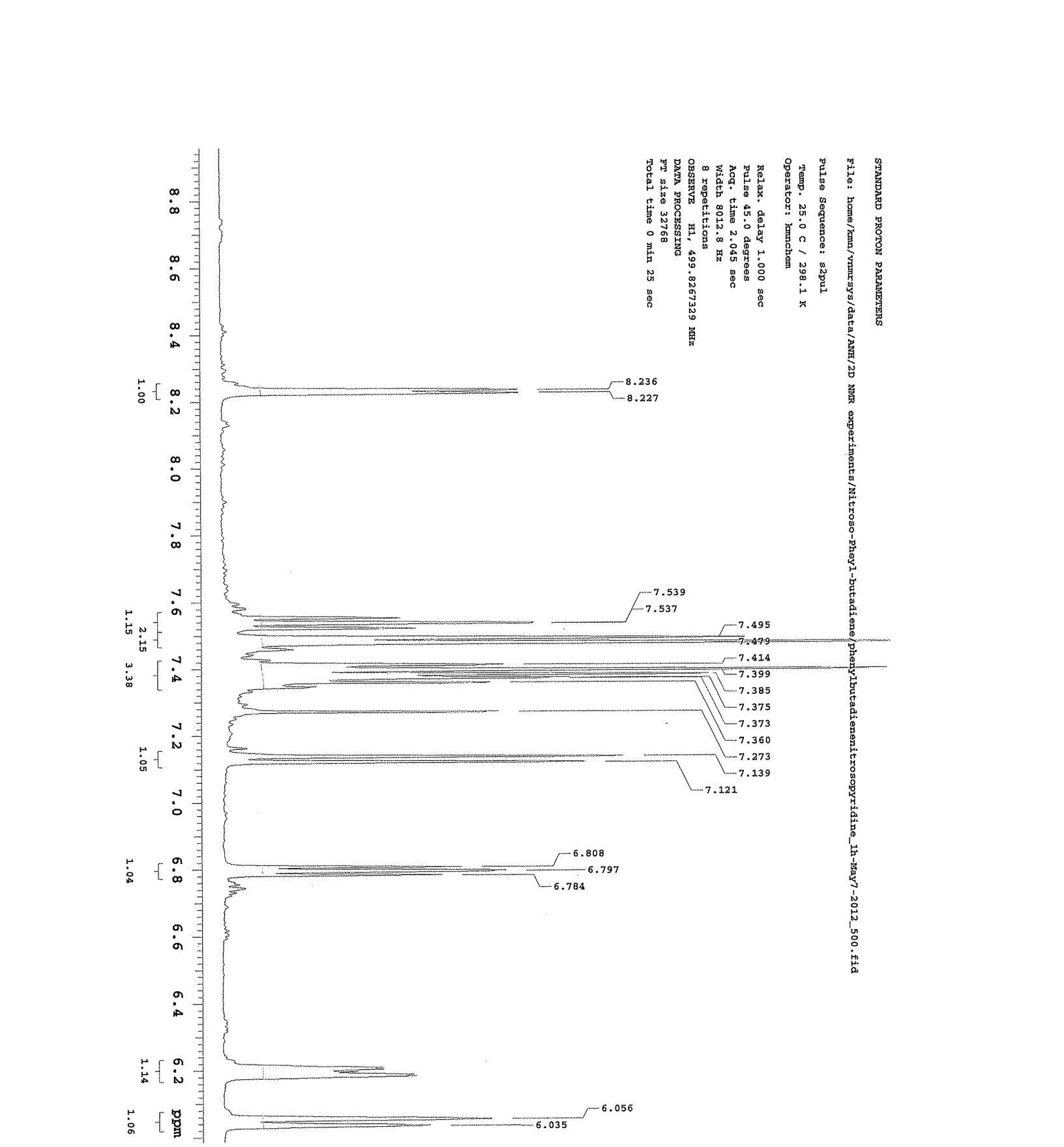


1H	Mult	J (hz)	COSY	HSQC	gHMBC	NOESY
4.15	m	17.2	4.48, 5.62	47.7	128.1, 124.4	4.48, 6.21
4.48	m	17.2	4.15, 5.62	47.7	128.1, 124.4	4.15, 6.21
5.62	m		4.14, 4.48	79.8	128.4, 138.6	
6.05	m	10.4	6.21, 4.48	128.1	124.4, 79.8, 47.7	
6.21	m	10.4	6.05	124.4	128.1, 79.8	
6.80	dd	7.9, 4.6	7.54, 8.23	116.6	110.4, 147.3	
7.14	d	7.9	7.54	110.4	116.6	5.62, 7.54
7.39	m			128.5	138.6, 128.5	
7.50	m			128.3	128.5, 79.8	
7.54	td	7.9, 1.8	6.8, 7.14, 8.23	137.5	160.9, 147.3	
8.23	dd	4.6, 1.8	6.8, 7.54	147.3	160.9, 137.5, 116.6	6.8

Experimental Details:
The experiments were carried out on a Varian VNMRS-500 MHz NMR. The proton spectra was collected with 8 scans. 1H and 13C chemical shifts were assigned by 2D methods. gCOSY, HSQC, and gHMBC were run using pulse sequences supplied by Varian, Inc., without modification. The gCOSY was collected with 1 scan and 128 increments. The HSQC was collected with 32 scans and 256 increments and the gHMBC was collected with 60 scans and 256 increments. 1D-NOESY was run to investigate conformation. Resonances at 4.15, 4.48, 7.14 and 8.23 were irradiated with 344 scans per experiment.

gCOSY: 1H-1H through bond coupling
HSQC: 1H-13C 1 bond coupling
gHMBC: 1H-13C long rang coupling: 2 -3 bond lengths
NOESY: 1H – 1H through space coupling, normally less than 4 angstroms.


STANDARD PROTON PARAMETERS
File: home/kmn/vnmrsys/data/ANH/2D NMR experiments/Nitroso-Phenyl-butadiene/phenylbutadienenitrosopyridine_1h-May7-2012_500.fid
Pulse Sequence: s2pul
Temp. 25.0 C / 298.1 K
Operator: kmnchem
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.045 sec
Width 8012.8 Hz
8 repetitions
OBSERVE H1, 499.8267329 MHz
DATA PROCESSING
FT size 32768
Total time 0 min 25 sec
8.236
8.234
7.539
7.537
7.479
7.414
7.399
7.385
7.375
7.373
7.360
7.273
7.139
7.121
6.808
6.797
6.784
6.056
6.035
5.616
4.175
4.172
1.00
2.15
1.05
1.04
1.14
1.06
1.01
1.15
1.14
ppm


STANDARD PROTON PARAMETERS
File: home/kmn/vnmrsys/data/ANH/2D NMR experiments/Nitroso-Phenyl-butadiene/phenylbutadienenitrosopyridine_1h-May7-2012_500.fid
Pulse Sequence: s2pul
Temp. 25.0 C / 298.1 K
Operator: kmnchem
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.045 sec
Width 8012.8 Hz
8 repetitions
OBSERVE H1, 499.8267329 MHz
DATA PROCESSING
FT size 32768
Total time 0 min 25 sec
8.236
8.227
7.539
7.537
7.495
7.479
7.414
7.399
7.385
7.375
7.373
7.360
7.273
7.139
7.121
6.808
6.797
6.784
6.056
6.035
8.8
8.6
8.4
8.2
8.0
7.8
7.6
7.4
7.2
7.0
6.8
6.6
6.4
6.2
ppm
1.00
2.15
3.38
1.05
1.04
1.14
1.06
1.15

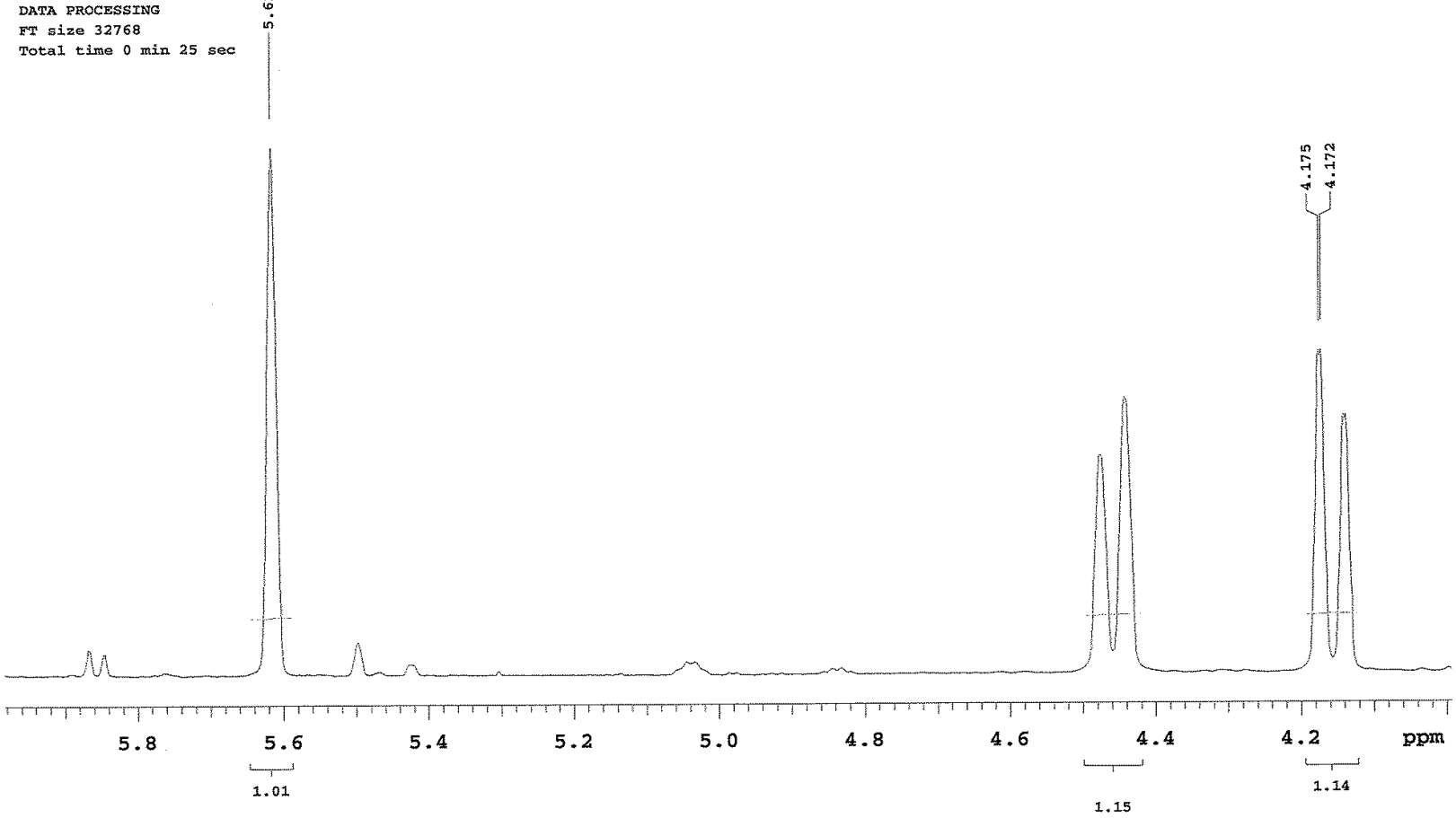
STANDARD PROTON PARAMETERS

File: home/kmn/vnmrsys/data/ANH/2D NMR experiments/Nitroso-Pheyl-butadiene/phenylbutadienenitrosopyridine_1h-May7-2012_500.fid

Pulse Sequence: s2pul

Temp. 25.0 C / 298.1 K
Operator: kmnchem

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.045 sec
Width 8012.8 Hz
8 repetitions
OBSERVE H1, 499.8267329 MHz
DATA PROCESSING
FT size 32768
Total time 0 min 25 sec



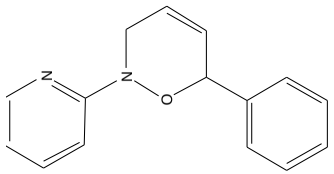
STANDARD PROTON PARAMETERS

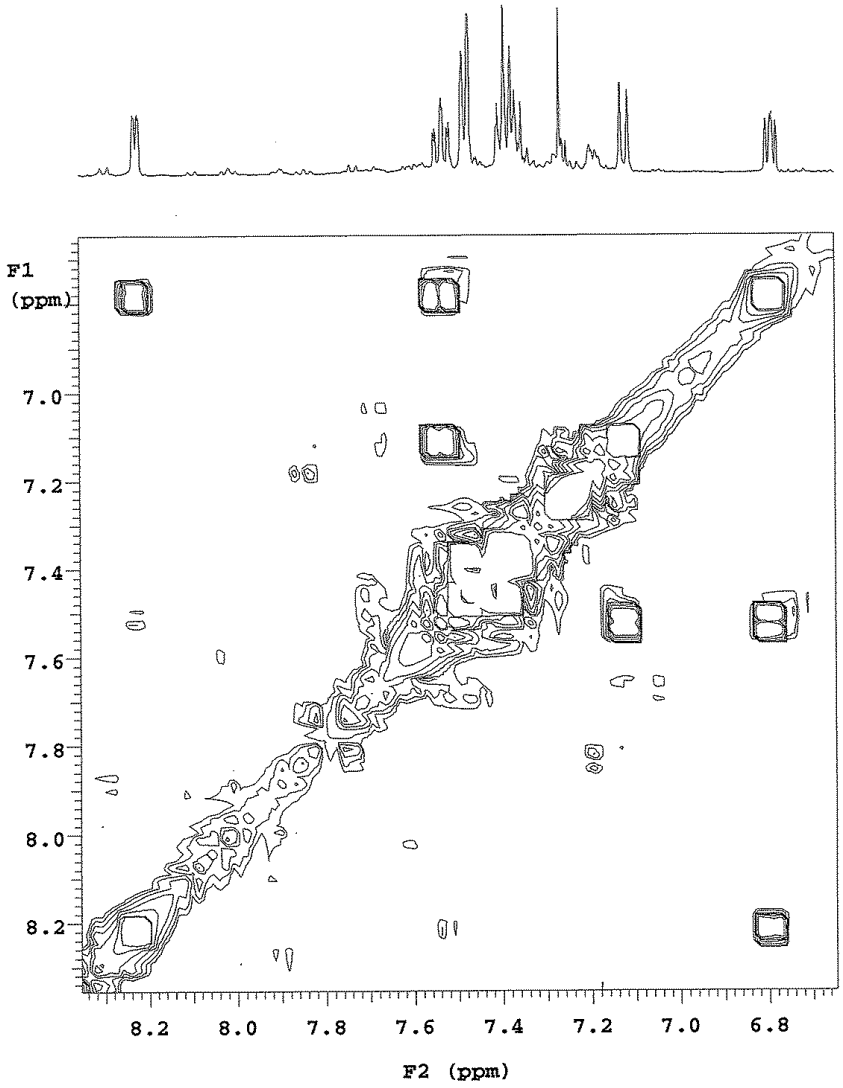
File: home/kmn/Desktop/ahn-500-0217-2012/phenylbutadienenitrosopyridine_gCOSY.fid

Pulse Sequence: gCOSY

Temp. 25.0 C / 298.1 K
Operator: kmnchem

Relax. delay 1.000 sec
Acq. time 0.150 sec
Width 8012.8 Hz
2D Width 8012.8 Hz
Single scan
128 increments
OBSERVE H1, 499.8267304 MHz
DATA PROCESSING
Sq. sine bell 0.064 sec
F1 DATA PROCESSING
Sq. sine bell 0.016 sec
FT size 2048 x 2048
Total time 3 min 13 sec





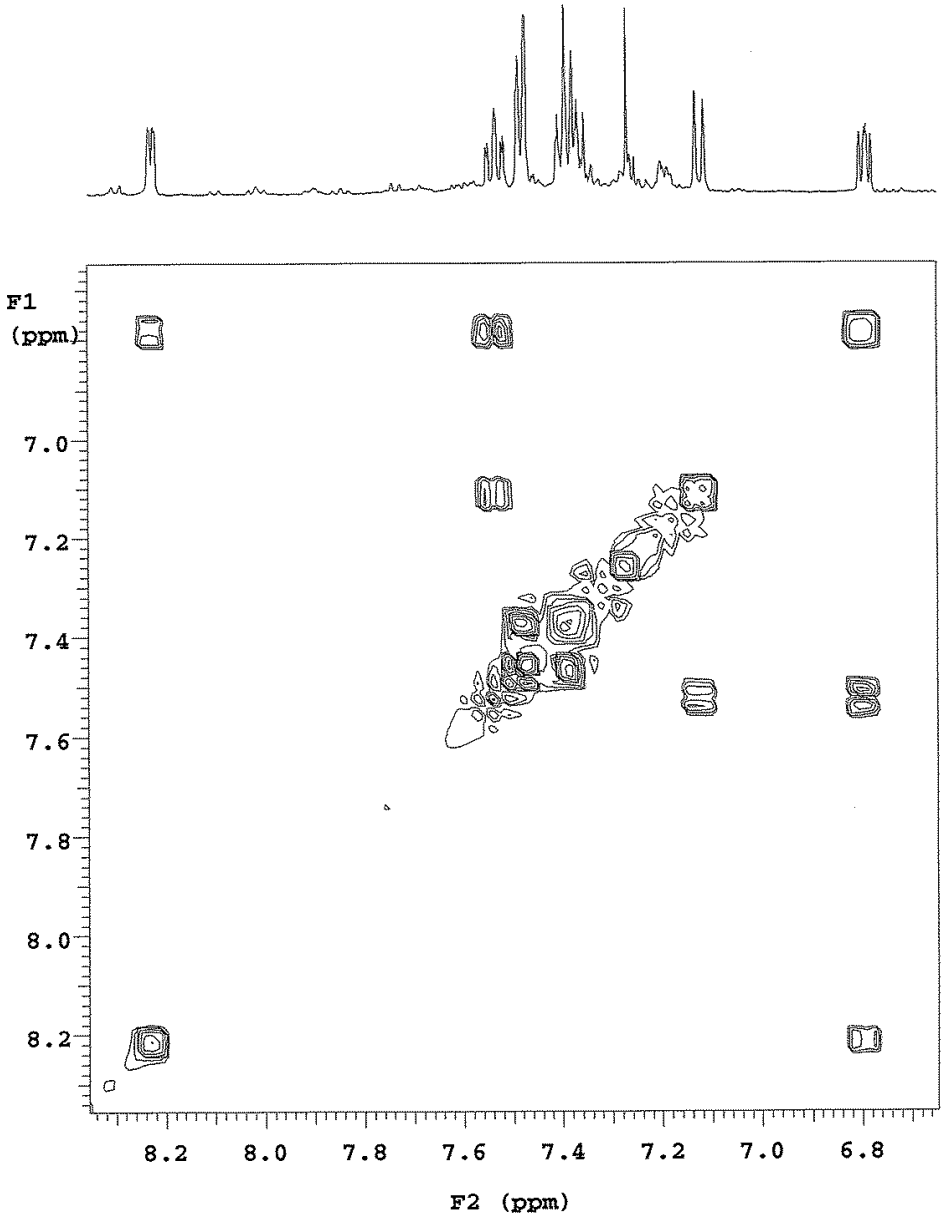
STANDARD PROTON PARAMETERS

File: home/kmn/Desktop/ahn-500-0217-2012/phenylbutadienenitrosopyridine_gCOSY.fid

Pulse Sequence: gCOSY

Temp. 25.0 C / 298.1 K
Operator: kmnchem

Relax. delay 1.000 sec
Acq. time 0.150 sec
Width 8012.8 Hz
2D Width 8012.8 Hz
Single scan
128 increments
OBSERVE H1, 499.8267304 MHz
DATA PROCESSING
Sq. sine bell 0.064 sec
F1 DATA PROCESSING
Sq. sine bell 0.016 sec
FT size 2048 x 2048
Total time 3 min 13 sec



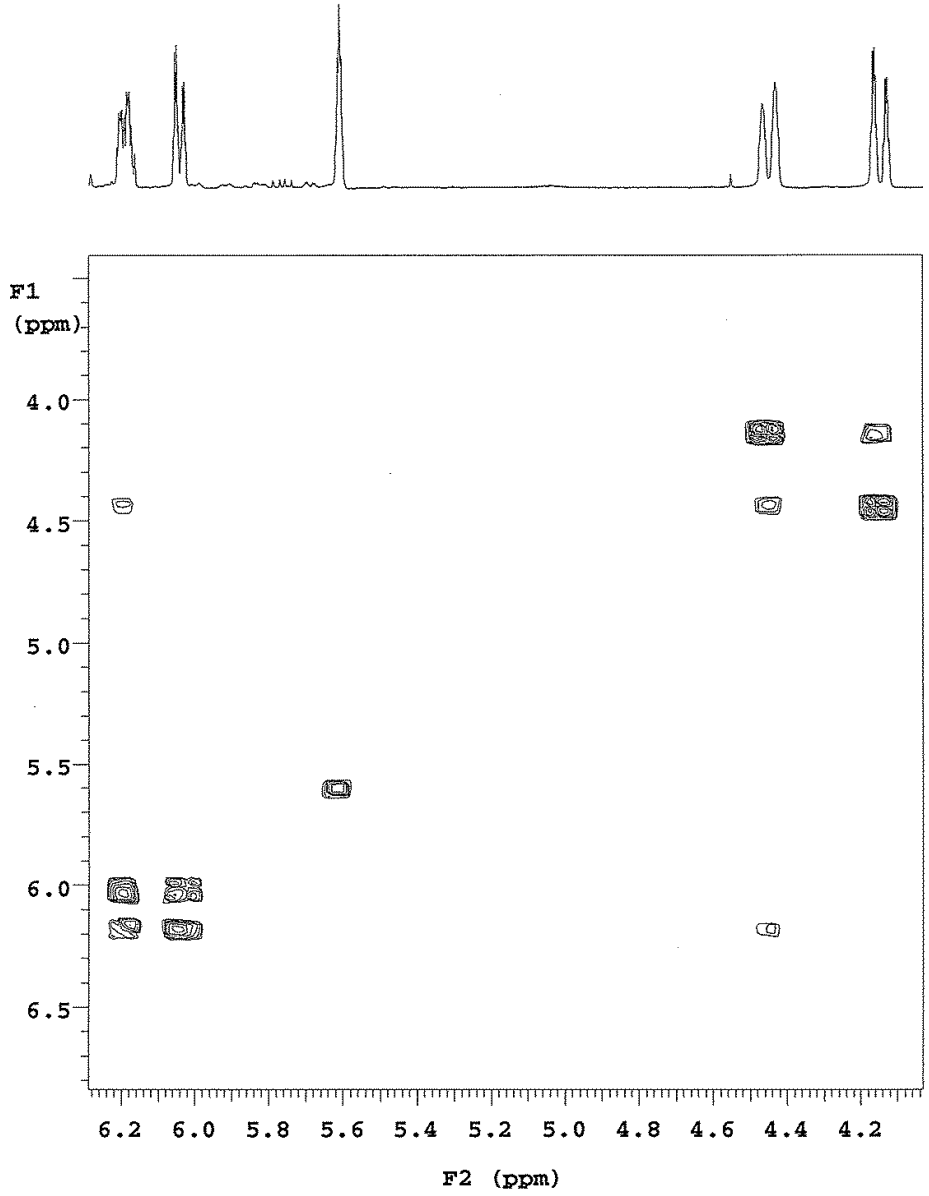
STANDARD PROTON PARAMETERS

File: home/kmn/Desktop/ahn-500-0217-2012/phenylbutadienenitrosopyridine_gCOSY.fid

Pulse Sequence: gCOSY

Temp. 25.0 C / 298.1 K
Operator: kmnchem

Relax. delay 1.000 sec
Acq. time 0.150 sec
Width 8012.8 Hz
2D Width 8012.8 Hz
Single scan
128 increments
OBSERVE H1, 499.8267304 MHz
DATA PROCESSING
Sq. sine bell 0.064 sec
F1 DATA PROCESSING
Sq. sine bell 0.016 sec
FT size 2048 x 2048
Total time 3 min 13 sec



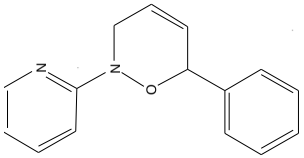
STANDARD PROTON PARAMETERS

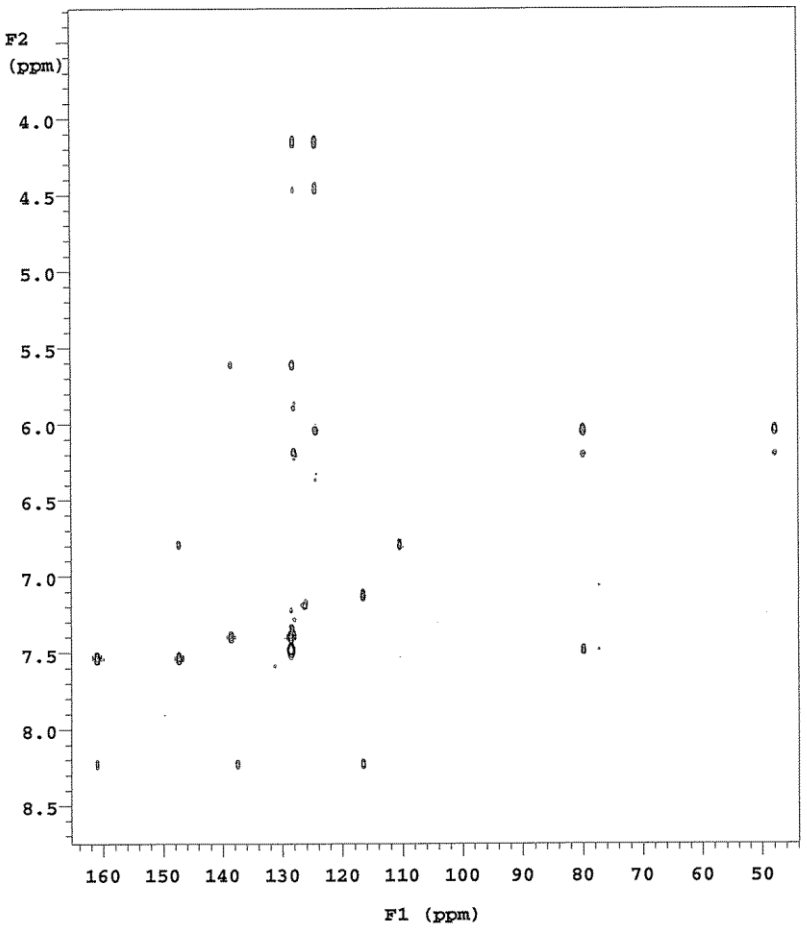
File: home/kmn/Desktop/ahn-500-0217-2012/phenylbutadienenitrosopyridine_HMBC.fid

Pulse Sequence: gHMBC

Temp. 25.0 C / 298.1 K
Operator: kmnchem

Relax. delay 1.000 sec
Acq. time 0.150 sec
Width 4006.4 Hz
2D Width 30165.9 Hz
60 repetitions
2 x 256 increments
OBSERVE H1, 499.8267329 MHz
DATA PROCESSING
Gauss apodization 0.059 sec
F1 DATA PROCESSING
Gauss apodization 0.016 sec
FT size 1024 x 4096
Total time 10 hr, 42 min





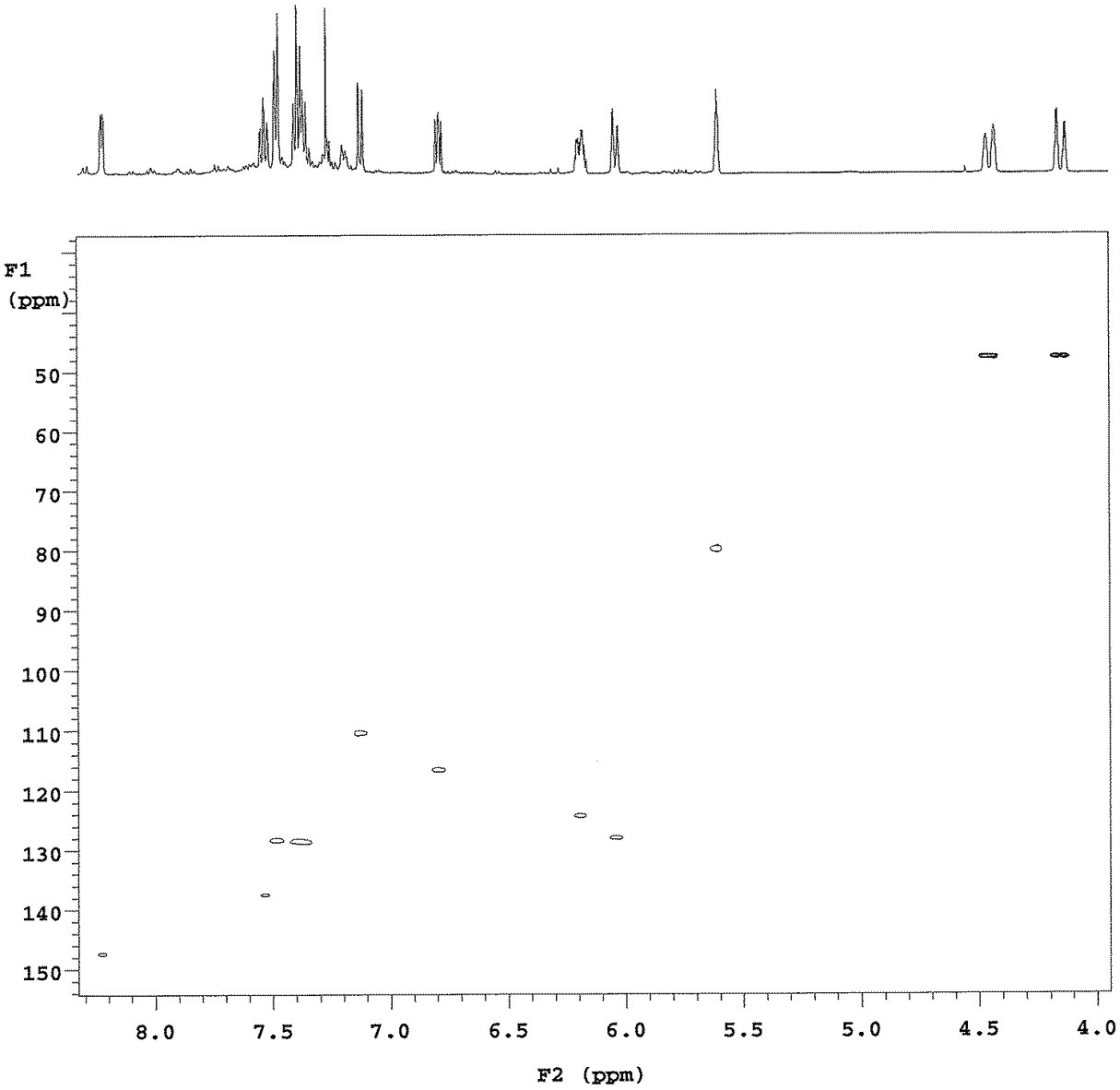
STANDARD PROTON PARAMETERS

File: home/kmn/Desktop/ahn-500-0217-2012/phenylbutadienenitrosopyridine_HSQC.fid

Pulse Sequence: HSQC

Temp. 25.0 C / 298.1 K
Operator: kmnchem

Relax. delay 1.000 sec
Acq. time 0.150 sec
Width 4006.4 Hz
2D Width 21367.5 Hz
32 repetitions
2 x 256 increments
OBSERVE H1, 499.8267329 MHz
DECOUPLE C13, 125.6909580 MHz
Power 46 dB
on during acquisition
off during delay
W40_TR_8064 modulated
DATA PROCESSING
Gauss apodization 0.088 sec
F1 DATA PROCESSING
Sine bell 0.013 sec
Shifted by -0.013 sec
FT size 1024 x 4096
Total time 5 hr, 34 min



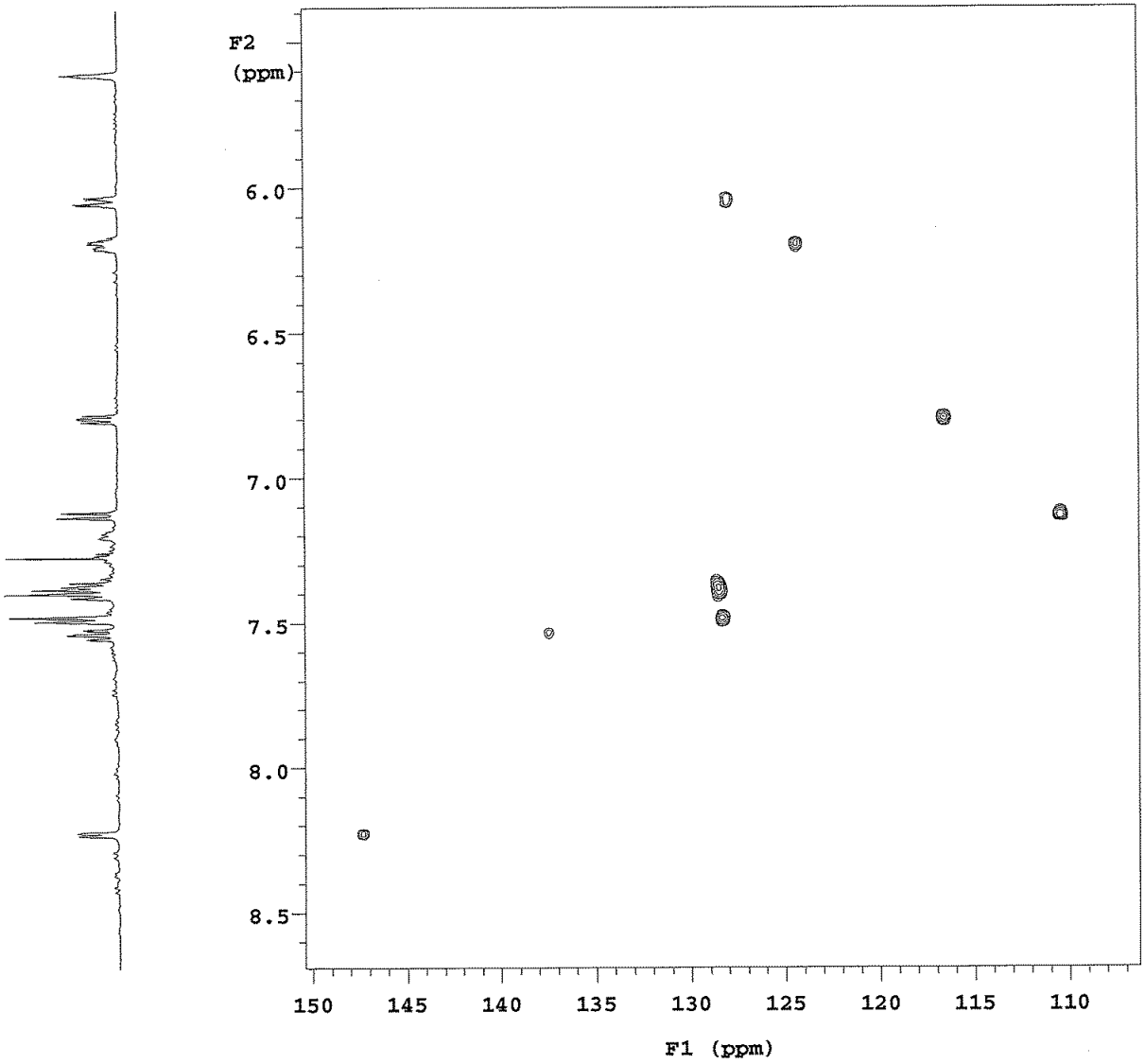
STANDARD PROTON PARAMETERS

File: home/kmn/Desktop/ahn-500-0217-2012/phenylbutadienenitrosopyridine_HSQC.fid

Pulse Sequence: HSQC

Temp. 25.0 C / 298.1 K
Operator: kmnchem

Relax. delay 1.000 sec
Acq. time 0.150 sec
Width 4006.4 Hz
2D Width 21367.5 Hz
32 repetitions
2 x 256 increments
OBSERVE H1, 499.8267329 MHz
DECOUPLE C13, 125.6909580 MHz
Power 46 dB
on during acquisition
off during delay
W40_TR_8064 modulated
DATA PROCESSING
Gauss apodization 0.088 sec
F1 DATA PROCESSING
Sine bell 0.013 sec
Shifted by -0.013 sec
FT size 1024 x 4096
Total time 5 hr, 34 min



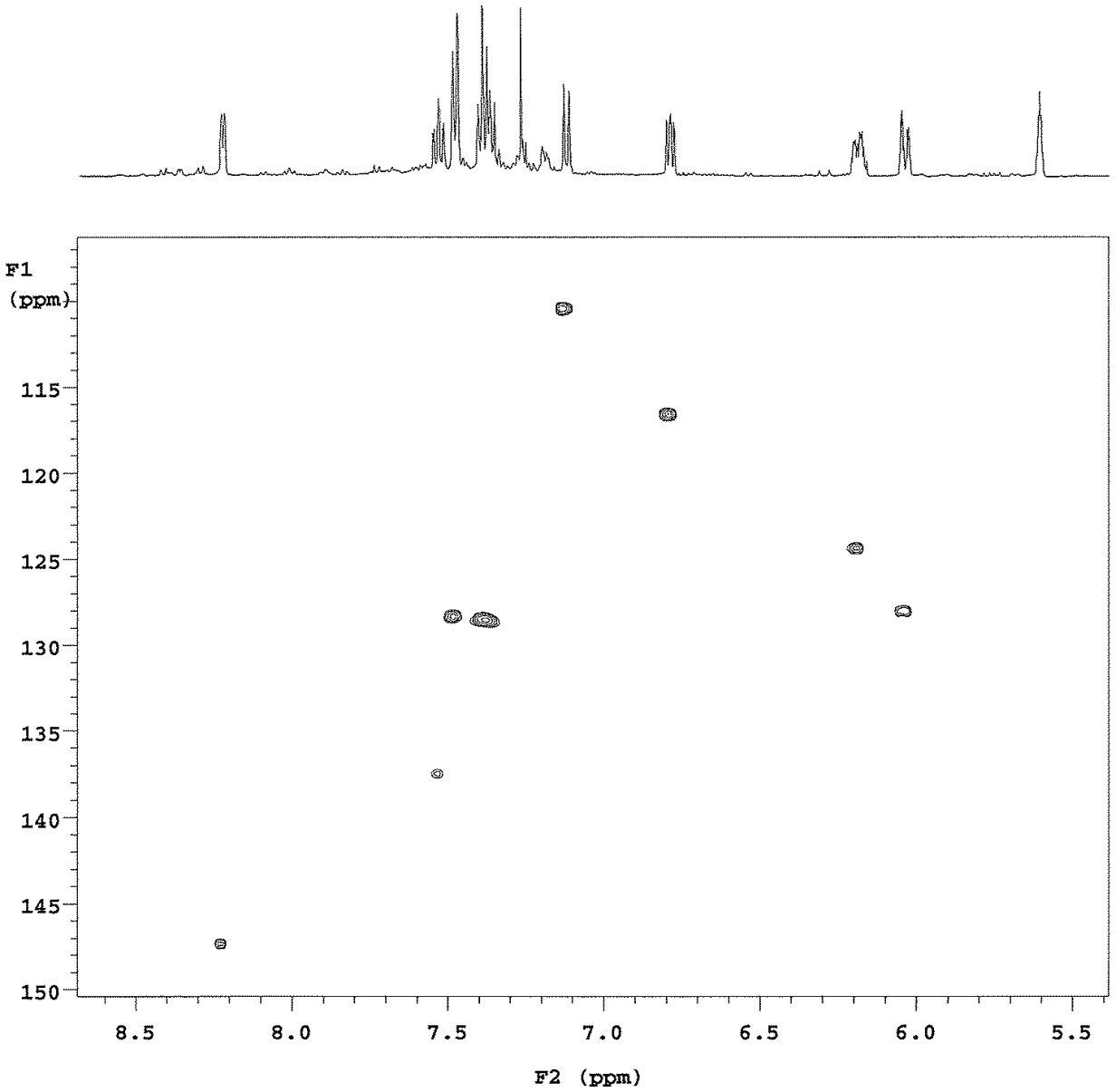
STANDARD PROTON PARAMETERS

File: home/kmn/Desktop/ahn-500-0217-2012/phenylbutadienenitrosopyridine_HSQC.fid

Pulse Sequence: HSQC

Temp. 25.0 C / 298.1 K
Operator: kmnchem

Relax. delay 1.000 sec
Acq. time 0.150 sec
Width 4006.4 Hz
2D Width 21367.5 Hz
32 repetitions
2 x 256 increments
OBSERVE H1, 499.8267329 MHz
DECOUPLE C13, 125.6909580 MHz
Power 46 dB
on during acquisition
off during delay
W40_TR_8064 modulated
DATA PROCESSING
Gauss apodization 0.088 sec
F1 DATA PROCESSING
Sine bell 0.013 sec
Shifted by -0.013 sec
FT size 1024 x 4096
Total time 5 hr, 34 min



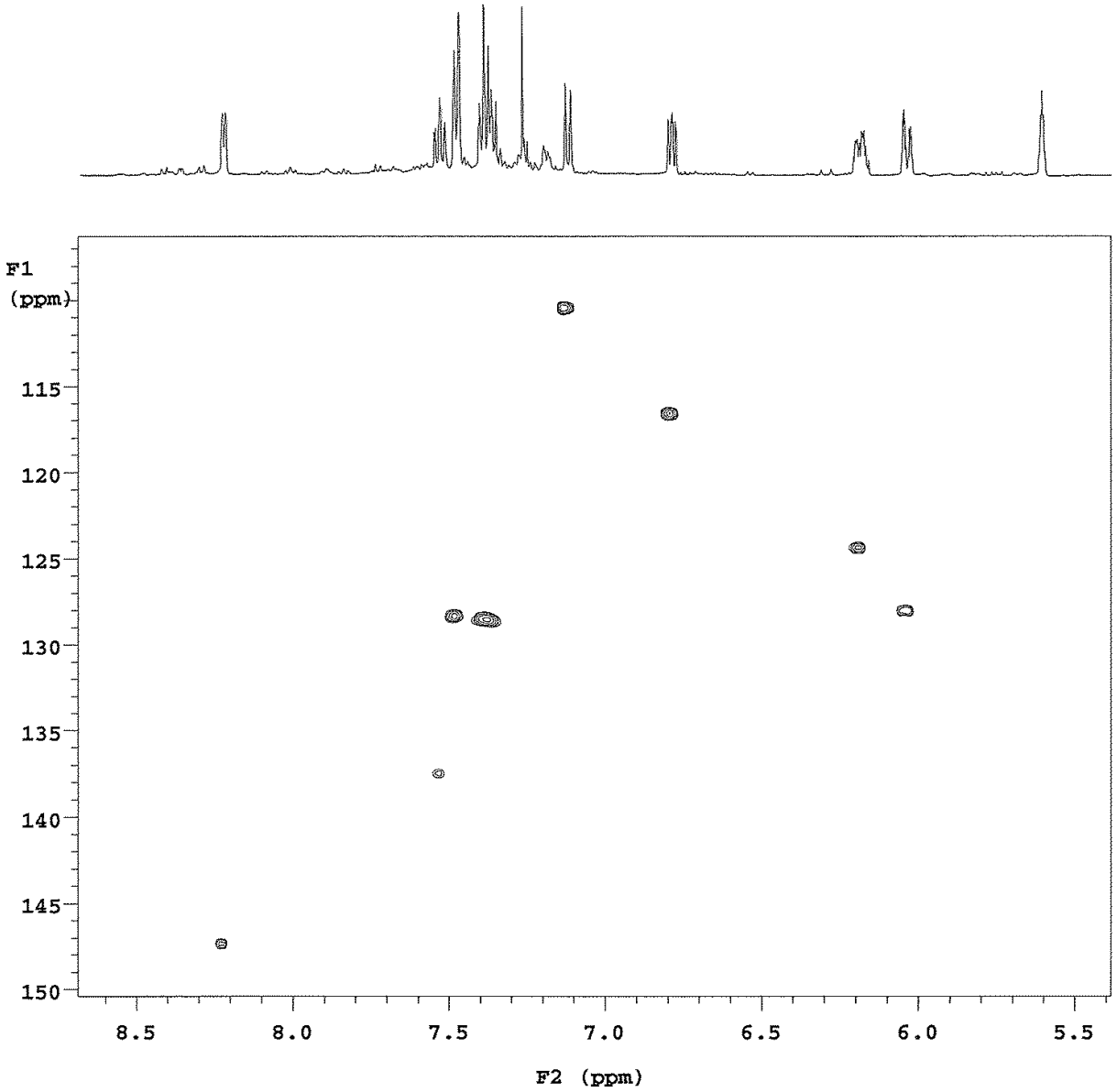
STANDARD PROTON PARAMETERS

File: home/kmm/Desktop/ahn-500-0217-2012/phenylbutadienenitrosopyridine_HSQC.fid

Pulse Sequence: HSQC

Temp. 25.0 C / 298.1 K
Operator: kmmchem

Relax. delay 1.000 sec
Acq. time 0.150 sec
Width 4006.4 Hz
2D Width 21367.5 Hz
32 repetitions
2 x 256 increments
OBSERVE H1, 499.8267329 MHz
DECOUPLE C13, 125.6909580 MHz
Power 46 dB
on during acquisition
off during delay
W40_TR_8064 modulated
DATA PROCESSING
Gauss apodization 0.088 sec
F1 DATA PROCESSING
Sine bell 0.013 sec
Shifted by -0.013 sec
FT size 1024 x 4096
Total time 5 hr, 34 min



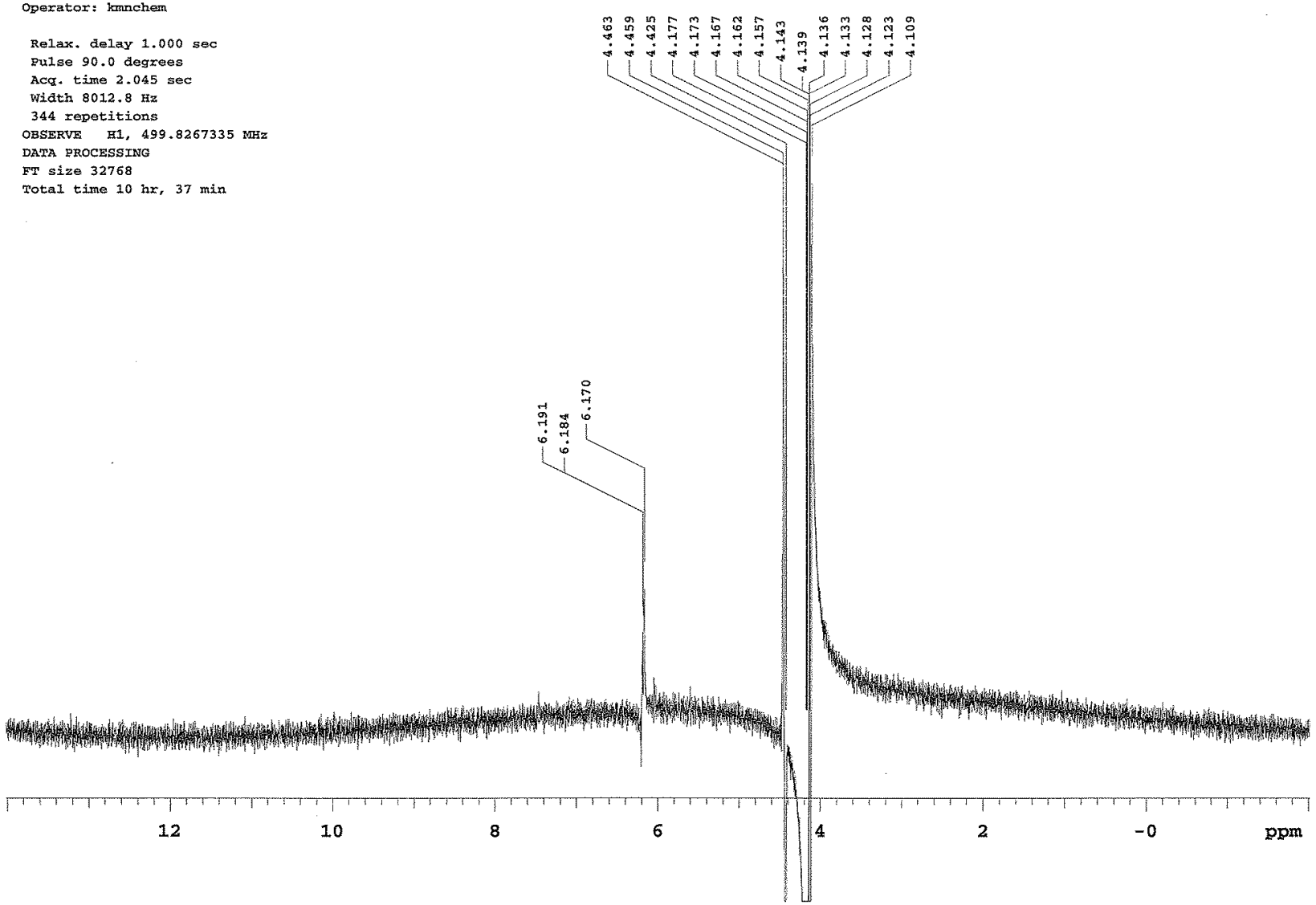
STANDARD PROTON PARAMETERS
Selective band center: 4.15 (ppm); width: 38.7 (Hz)

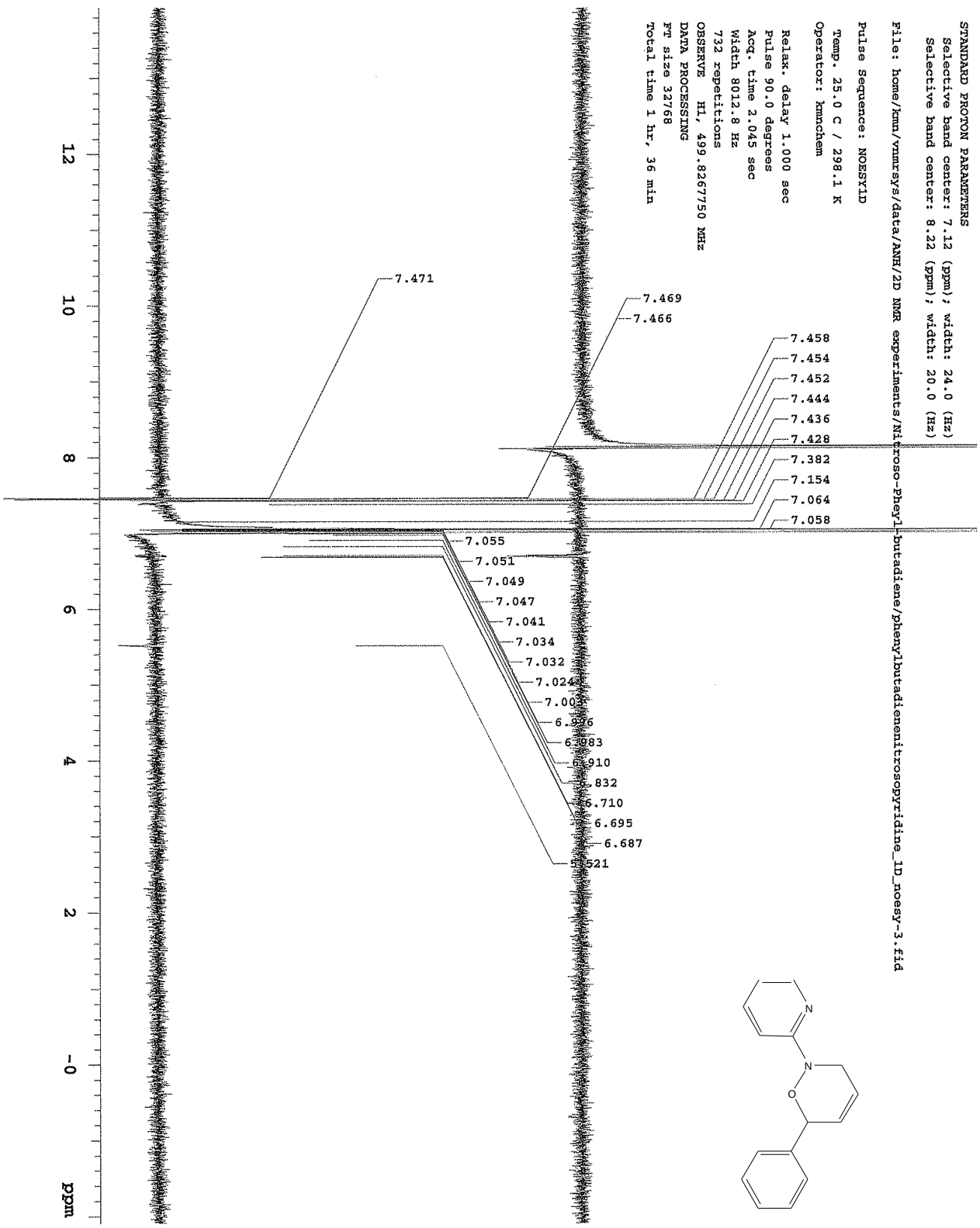
File: home/kmn/vnmrsys/data/ANH/2D NMR experiments/Nitroso-Pheyl-butadiene/phenylbutadienenitrosopyridine_1D_noesy-1.fid

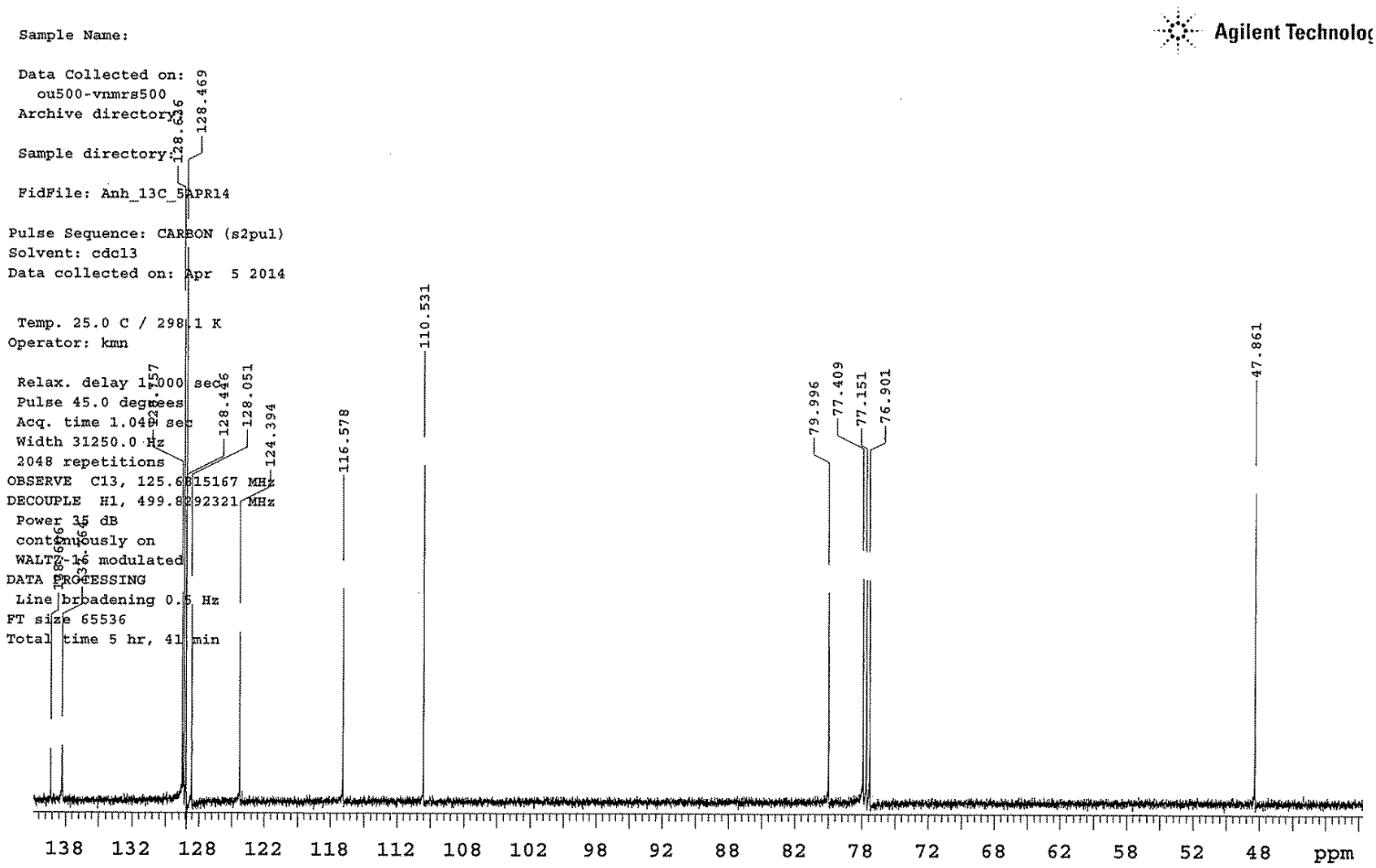
Pulse Sequence: NOESY1D

Temp. 25.0 C / 298.1 K
Operator: kmnchem

Relax. delay 1.000 sec
Pulse 90.0 degrees
Acq. time 2.045 sec
Width 8012.8 Hz
344 repetitions
OBSERVE H1, 499.8267335 MHz
DATA PROCESSING
FT size 32768
Total time 10 hr, 37 min




STANDARD PROTON PARAMETERS
Selective band center: 7.12 (ppm); width: 24.0 (Hz)
Selective band center: 8.22 (ppm); width: 20.0 (Hz)
File: home/kmm/vnmrsys/data/ANH/2D NMR experiments/Nitroso-Phenylbutadiene/phenylbutadienenitrosopyridine_1D_noesy-3.fid
Pulse Sequence: NOESY1D
Temp. 25.0 C / 298.1 K
Operator: kmmchem
Relax. delay 1.000 sec
Pulse 90.0 degrees
Acq. time 2.045 sec
Width 8012.8 Hz
732 repetitions
OBSERVE H1, 499.8267750 MHz
DATA PROCESSING
FT size 32768
Total time 1 hr, 36 min
7.471
7.469
7.466
7.458
7.454
7.452
7.444
7.436
7.428
7.382
7.154
7.064
7.058
7.055
7.051
7.049
7.047
7.041
7.034
7.032
7.024
7.00
6.976
6.983
6.910
6.832
6.710
6.695
6.687
6.521
12
10
8
6
4
2
-0
ppm
N
N
O


Sample Name:
Data Collected on:
ou500-vnmrs500
Archive directory
Sample directory
FidFile: Anh_13C_5APR14
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Apr 5 2014
Temp. 25.0 C / 298.1 K
Operator: kmn
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.049 sec
Width 31250.0 Hz
2048 repetitions
OBSERVE C13, 125.6815167 MHz
DECOUPLE H1, 499.8292321 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 5 hr, 41 min
128.469
128.236
22.757
128.446
128.051
124.394
116.578
110.531
79.996
77.409
77.151
76.901
47.861
138
132
128
122
118
112
108
102
98
92
88
82
78
72
68
62
58
52
48
ppm

Sample Name:

Data Collected on:
ou500-vnmrs500
Archive directory:

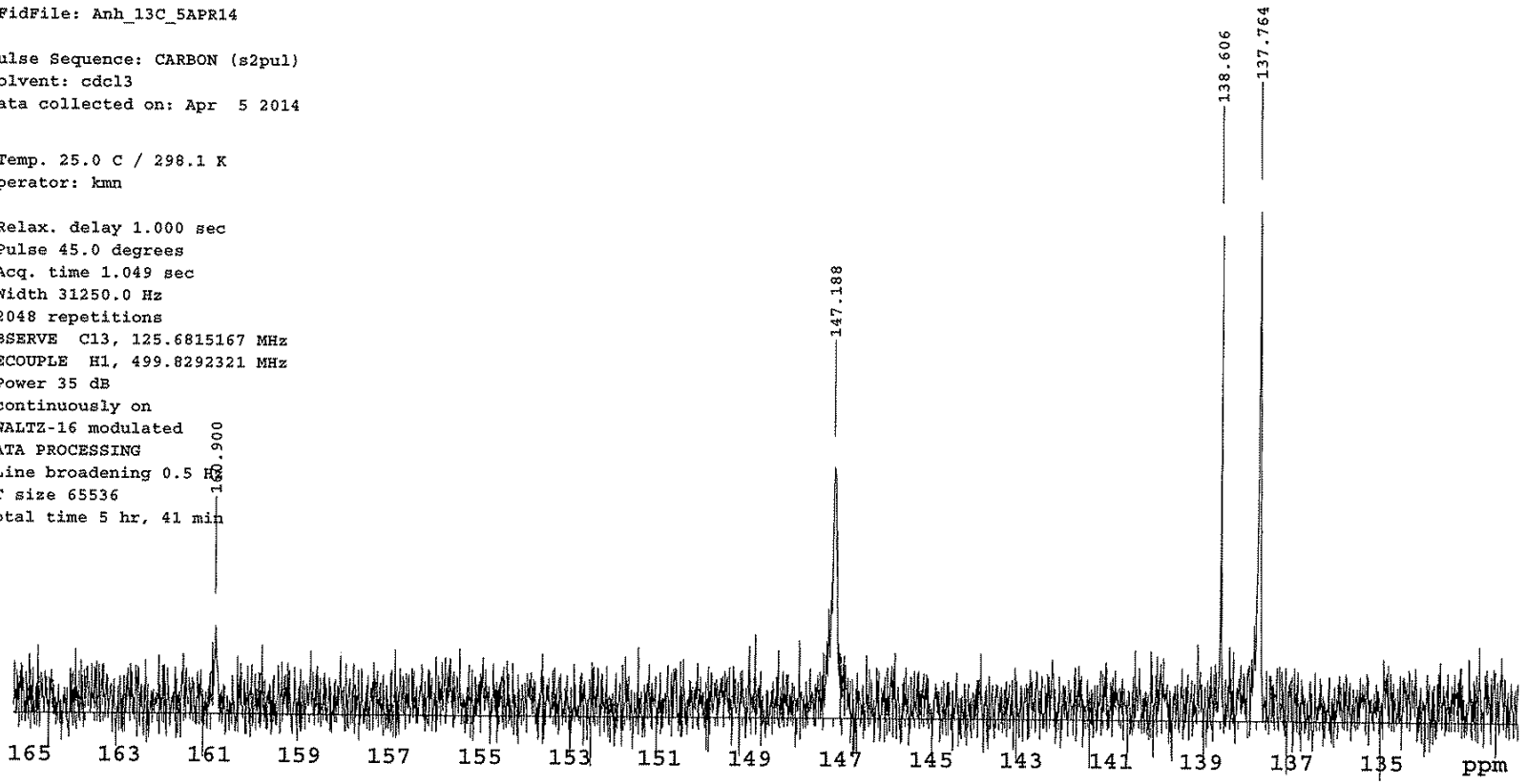
Sample directory:

FidFile: Anh_13C_5APR14

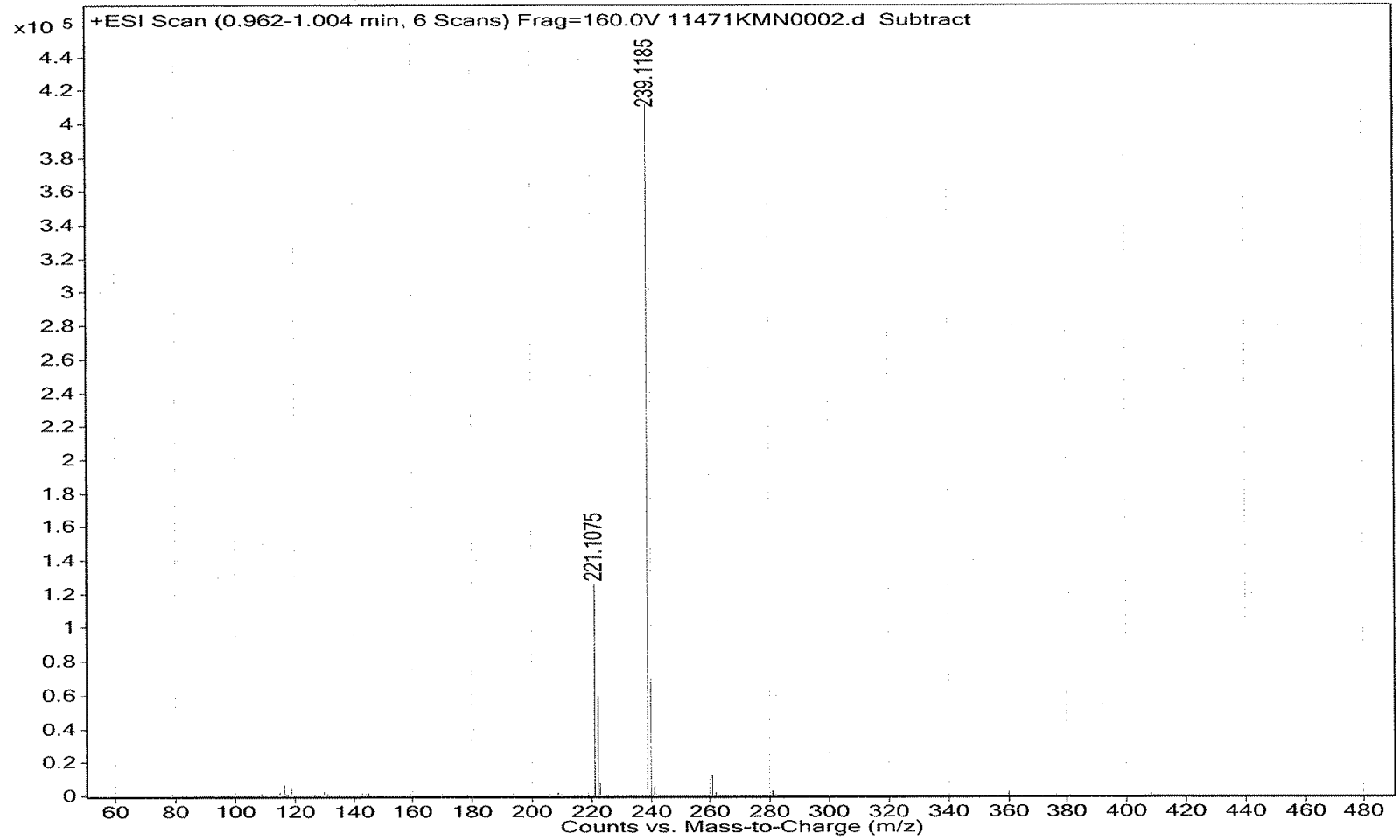
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Apr 5 2014

Temp. 25.0 C / 298.1 K
Operator: kmn

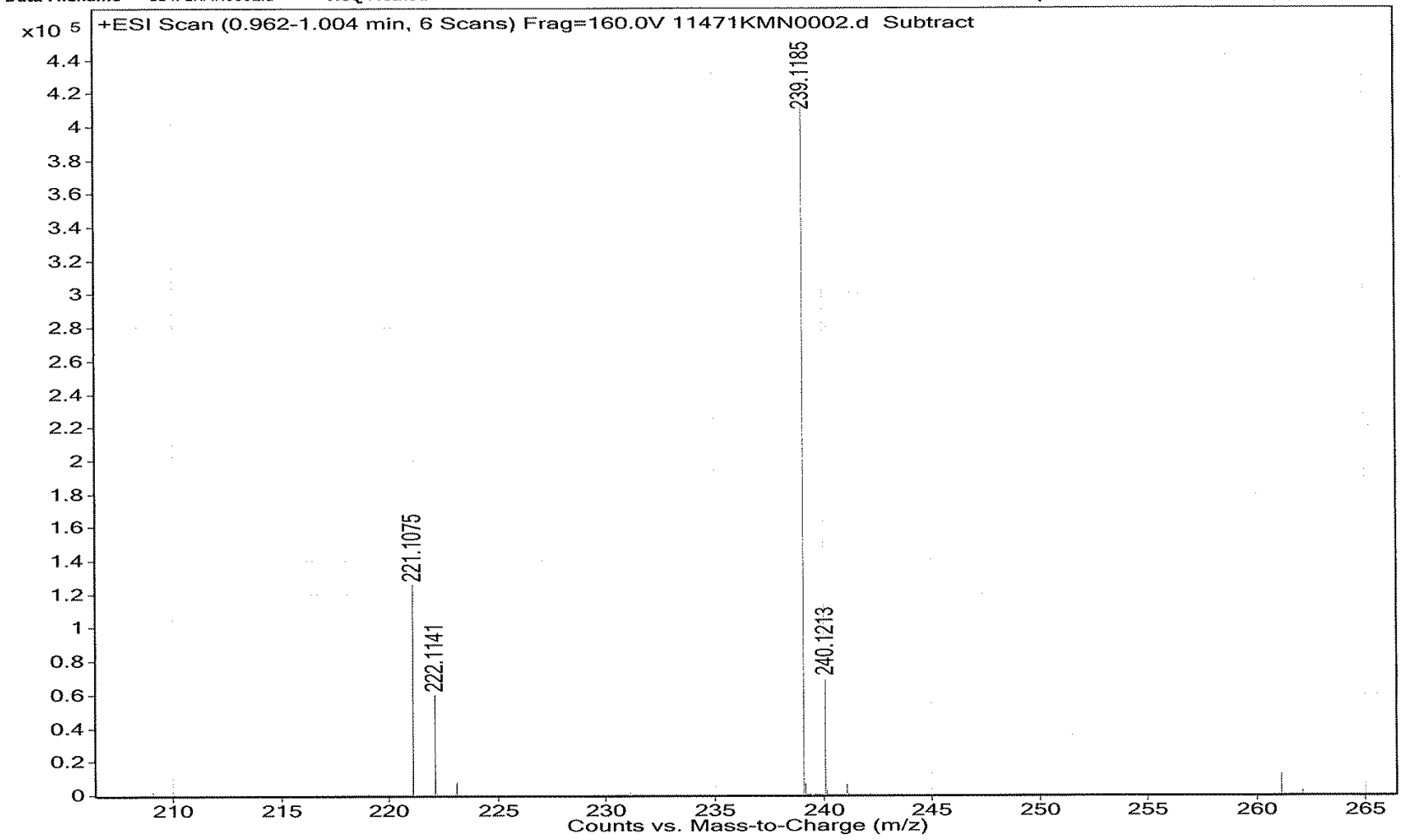
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.049 sec
Width 31250.0 Hz
2048 repetitions
OBSERVE C13, 125.6815167 MHz
DECOUPLE H1, 499.8292321 MHz
Power 35 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 5 hr, 41 min

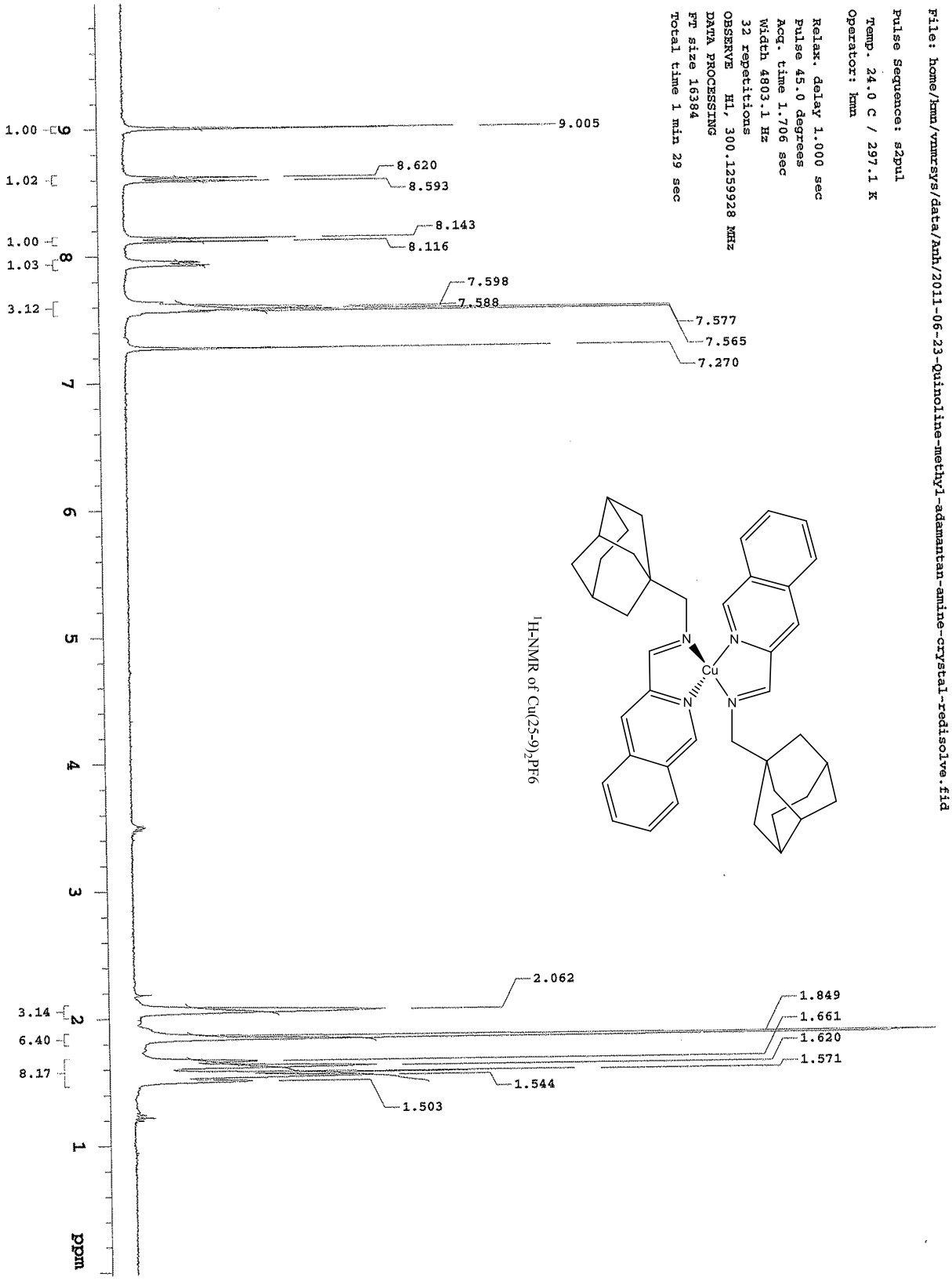


Sample Name	anhtran242	**Position**	P1-B3	**Instrument Name**	Instrument MS	**User Name**	Foster
Inj Vol	5	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	11471KMN0002.d	**ACQ Method**		**Comment**	10-3x	**Acquired Time**	4/3/2014 9:27:20 AM

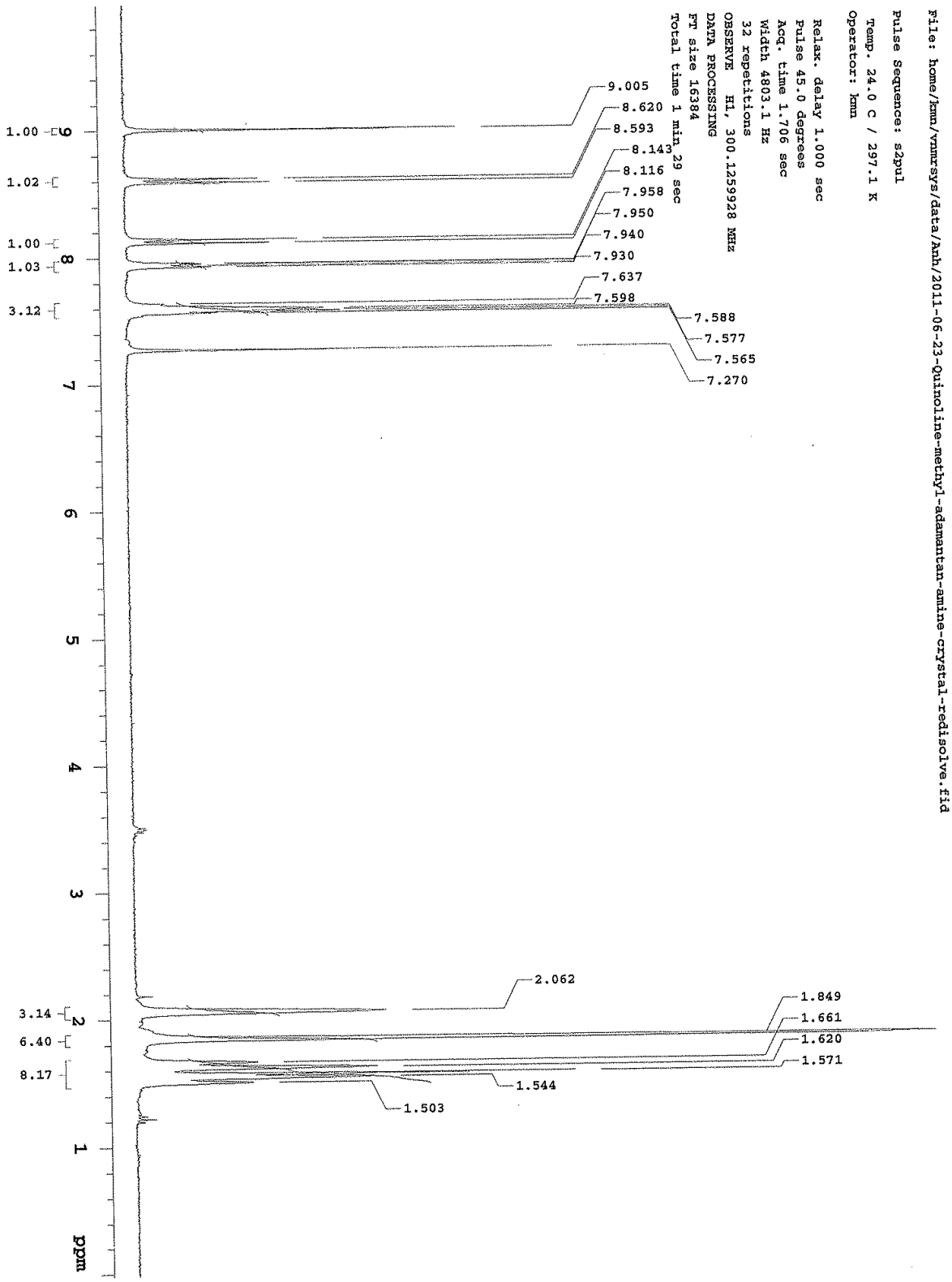


Sample Name	anhtran242	**Position**	P1-B3	**Instrument Name**	Instrument MS	**User Name**	Foster
Inj Vol	5	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	11471KMN0002.d	**ACQ Method**		**Comment**	10-3x	**Acquired Time**	4/3/2014 9:27:20 AM





^{1}H-NMR of Cu(25-9)$_2$PF6

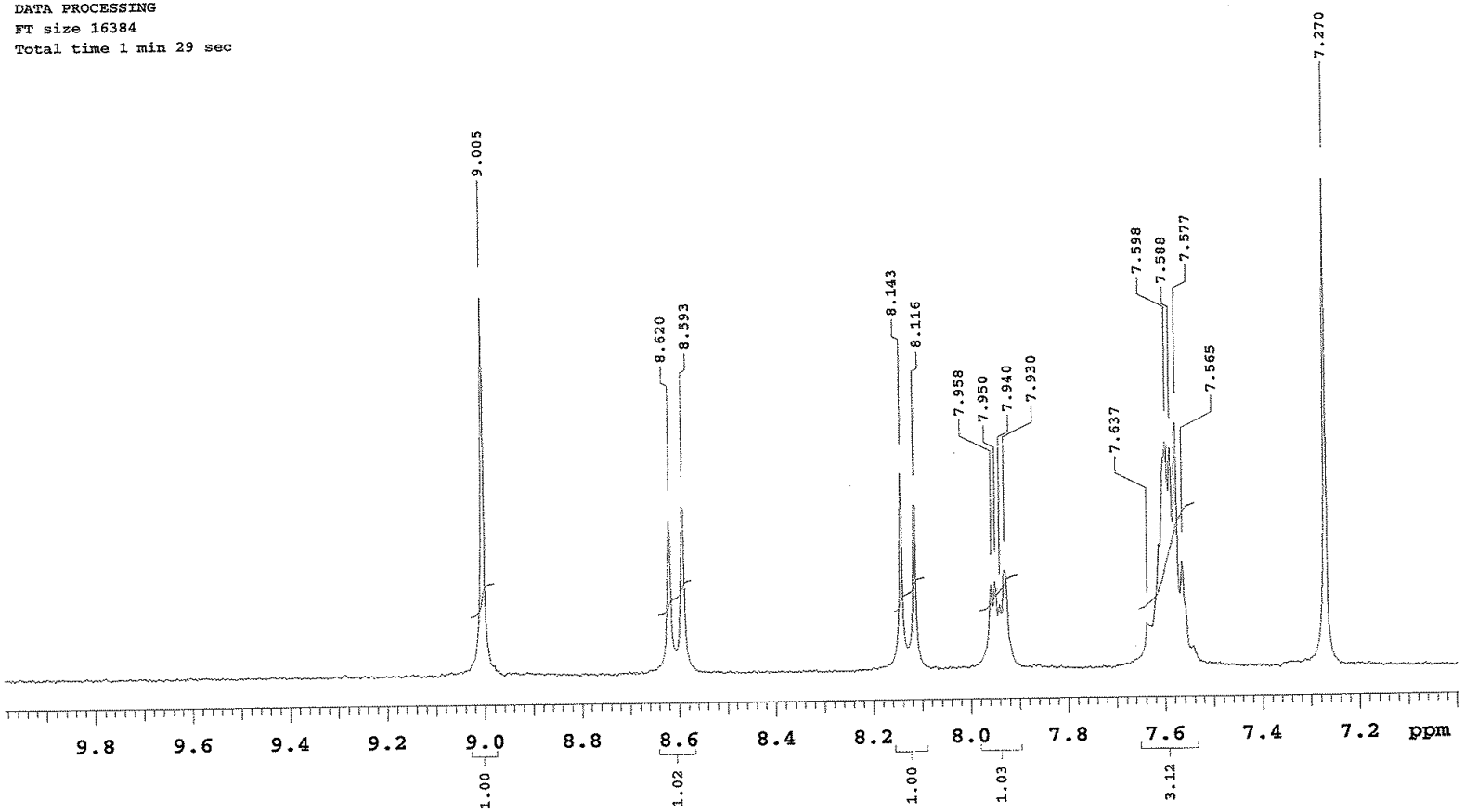

File: home/kmn/vnmrsys/data/Anh/2011-06-23-Quinoline-methyl-adamantan-amine-crystal-redisolve.fid
Pulse Sequence: s2pul
Temp. 24.0 C / 297.1 K
Operator: kmn
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.706 sec
Width 4803.1 Hz
32 repetitions
OBSERVE H1, 300.1259928 MHz
DATA PROCESSING
FT size 16384
Total time 1 min, 29 sec
9.005
8.620
8.593
8.143
8.116
7.958
7.950
7.940
7.930
7.637
7.598
7.588
7.577
7.565
7.270
2.062
1.849
1.661
1.620
1.571
1.544
1.503
1.00
1.02
1.00
1.03
3.12
3.14
6.40
8.17
ppm

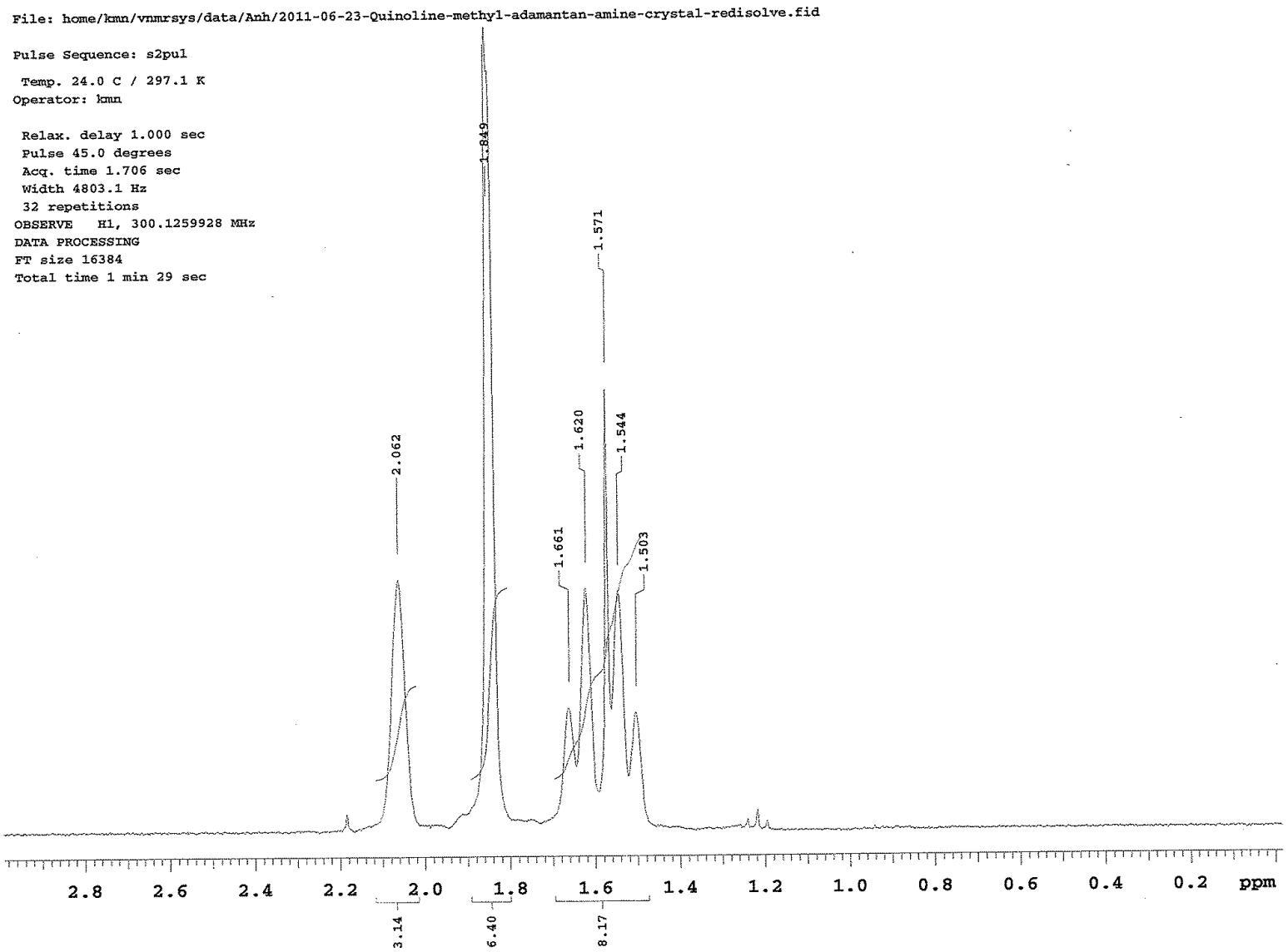
File: home/kmn/vnmrsys/data/Anh/2011-06-23-Quinoline-methyl-adamantan-amine-crystal-redisolve.fid

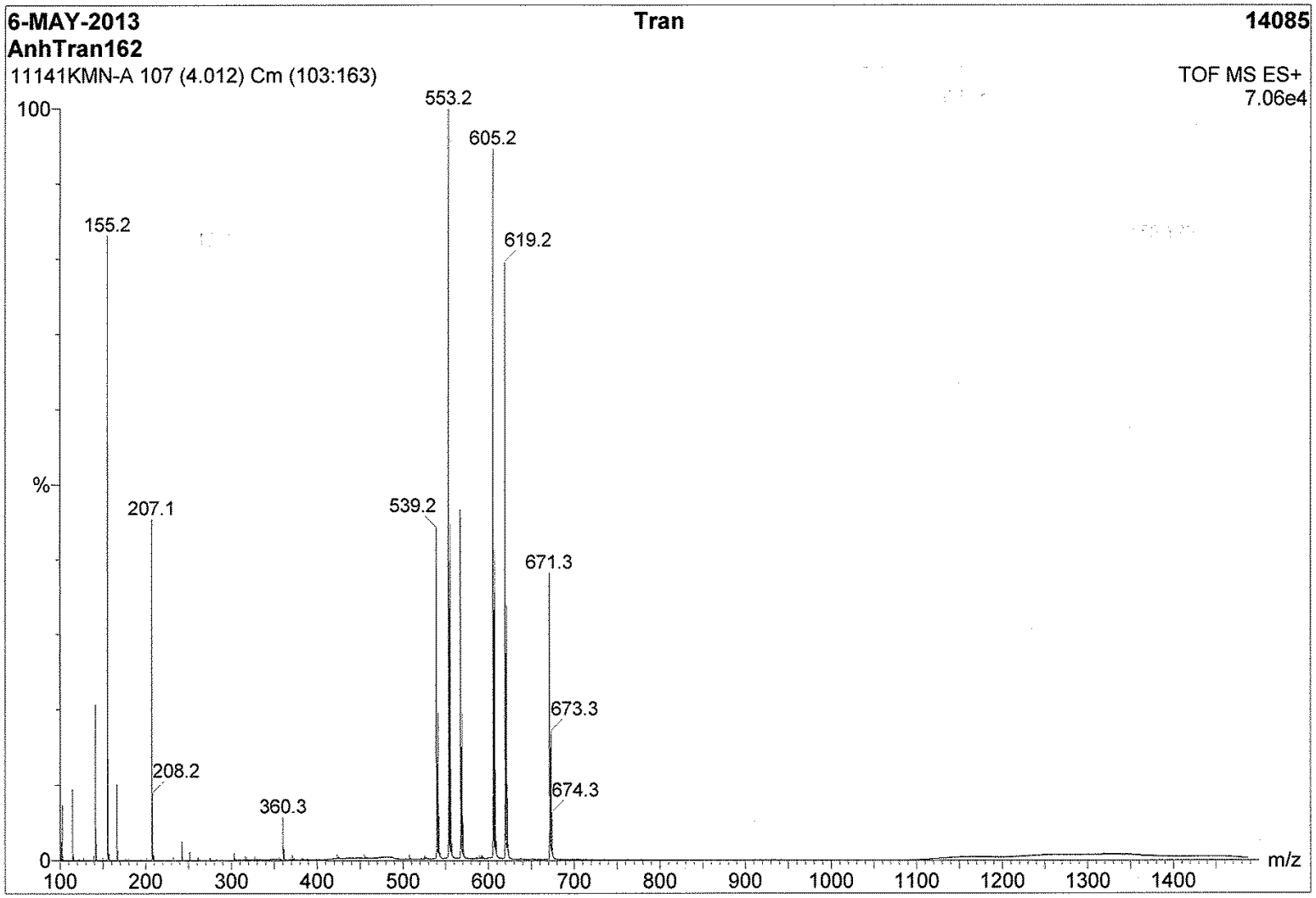
Pulse Sequence: s2pul

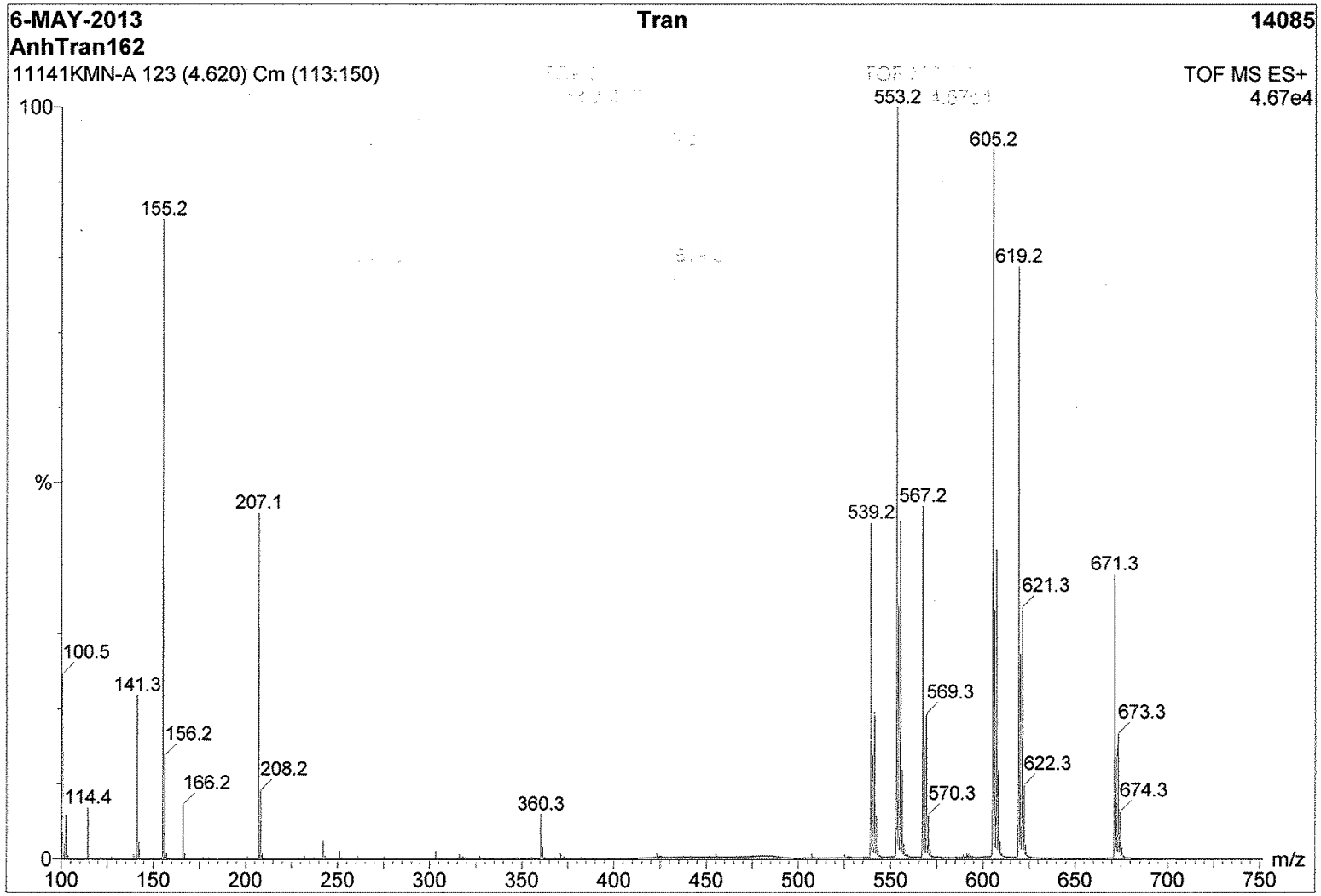
Temp. 24.0 C / 297.1 K
Operator: kmn

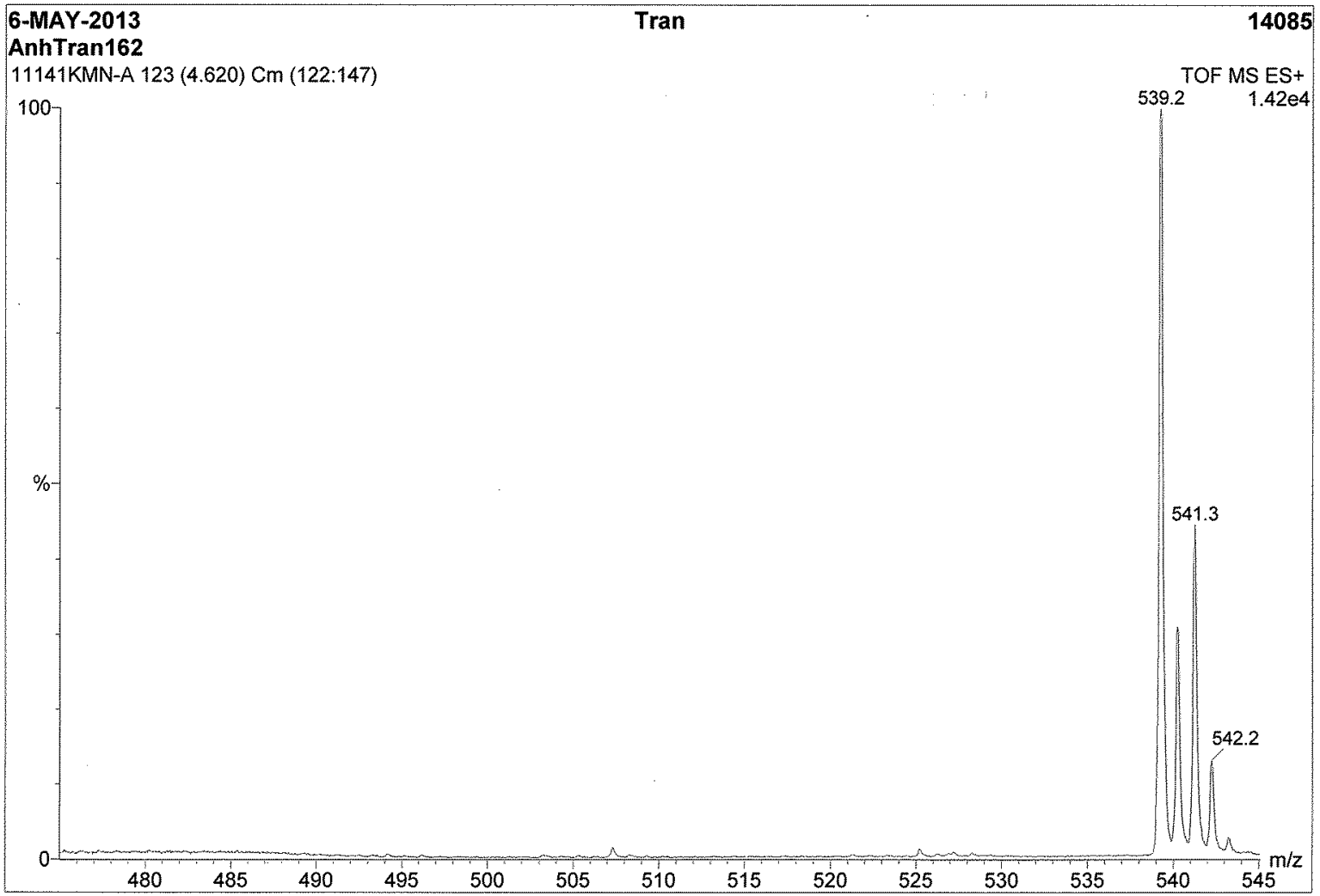
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.706 sec
Width 4803.1 Hz
32 repetitions
OBSERVE H1, 300.1259928 MHz
DATA PROCESSING
FT size 16384
Total time 1 min 29 sec



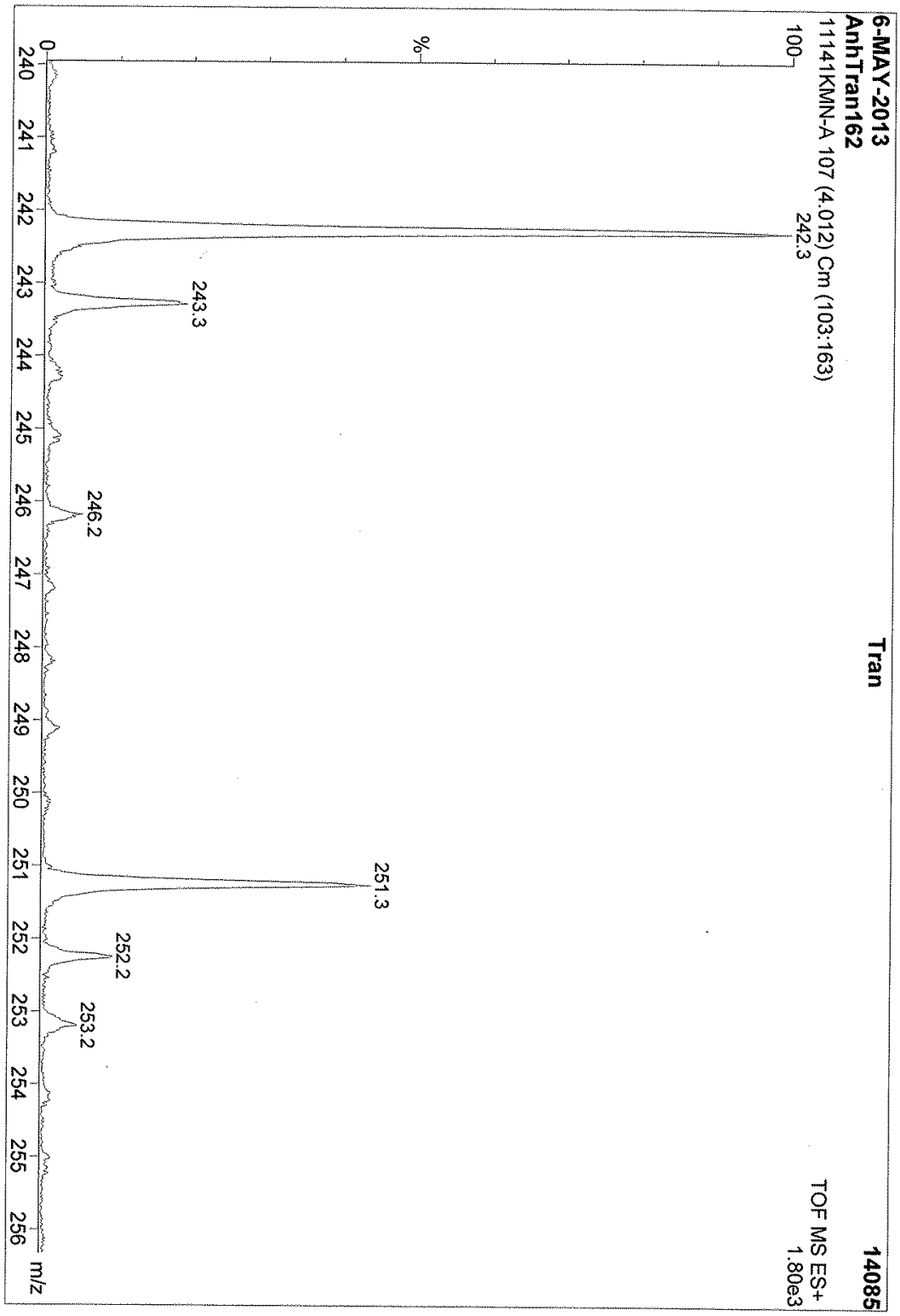

File: home/kmn/vnmrsys/data/Anh/2011-06-23-Quinoline-methyl-adamantan-amine-crystal-redisolve.fid
Pulse Sequence: s2pul
Temp. 24.0 C / 297.1 K
Operator: kmn
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.706 sec
Width 4803.1 Hz
32 repetitions
OBSERVE H1, 300.1259928 MHz
DATA PROCESSING
FT size 16384
Total time 1 min 29 sec
2.062
1.849
1.661
1.620
1.571
1.544
1.503
2.8
2.6
2.4
2.2
2.0
1.8
1.6
1.4
1.2
1.0
0.8
0.6
0.4
0.2
ppm
3.14
6.40
8.17


6-MAY-2013
AnhTran162
Tran
14085
11141KMN-A 107 (4.012) Cm (103:163)
TOF MS ES+
7.06e4
100
%
0
553.2
605.2
155.2
619.2
207.1
539.2
671.3
673.3
208.2
674.3
360.3
m/z
100
200
300
400
500
600
700
800
900
1000
1100
1200
1300
1400

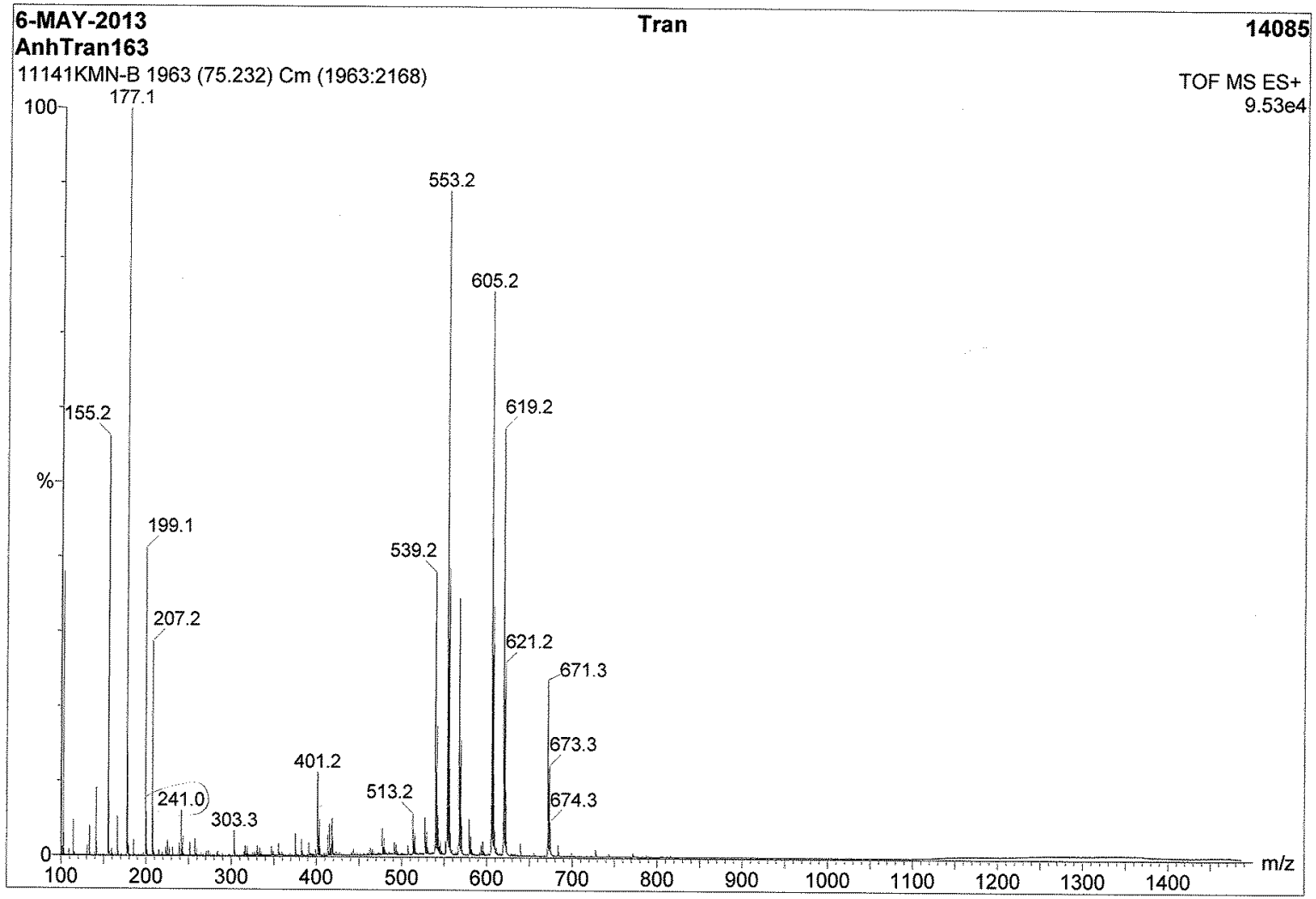

6-MAY-2013
Tran
14085
AnhTran162
11141KMN-A 123 (4.620) Cm (113:150)
TOF MS ES+
4.67e4
100
%
0
553.2
605.2
155.2
619.2
207.1
539.2
567.2
671.3
621.3
100.5
141.3
569.3
673.3
156.2
622.3
208.2
166.2
570.3
674.3
114.4
360.3
m/z
100
150
200
250
300
350
400
450
500
550
600
650
700
750

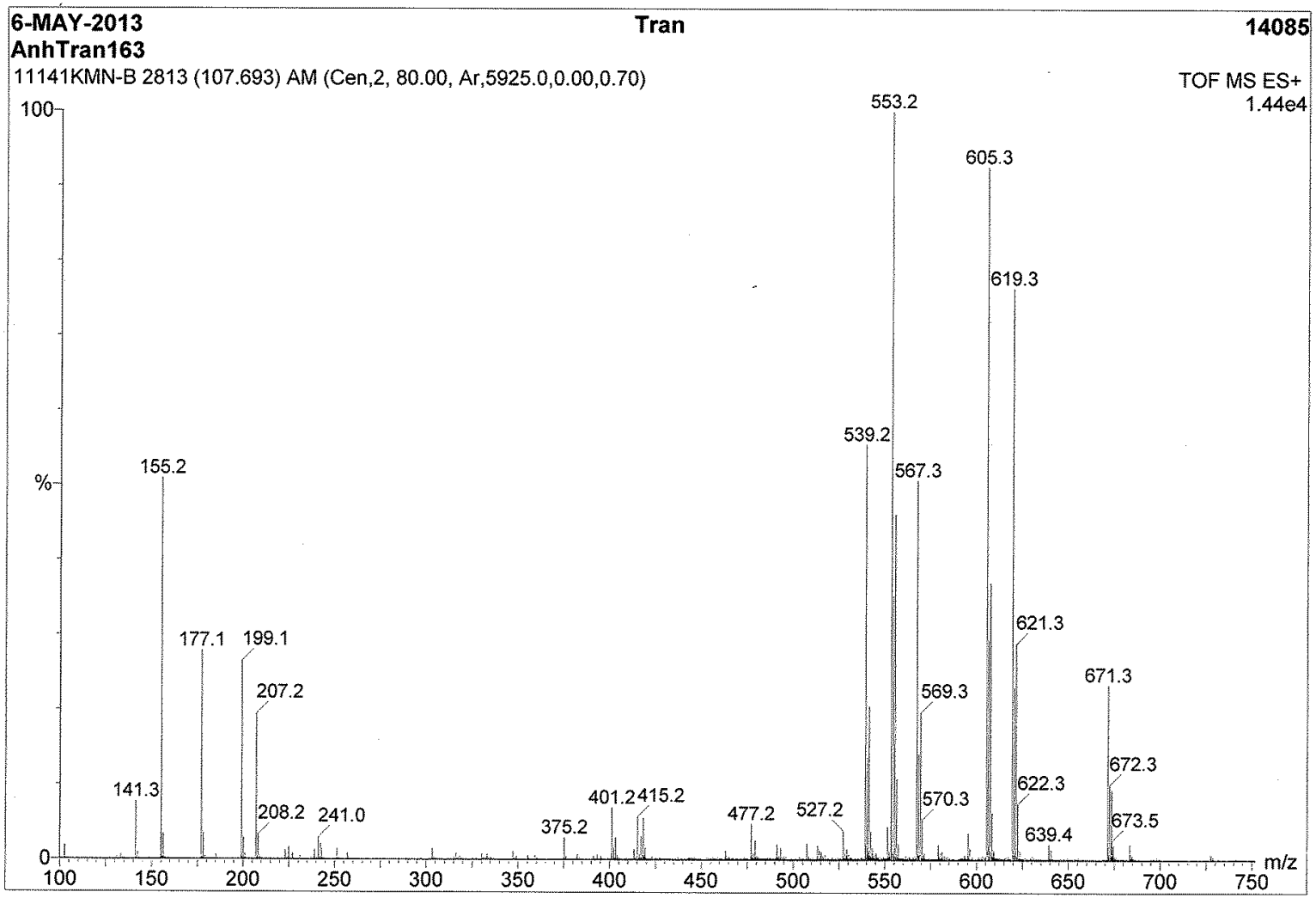

6-MAY-2013
Tran
14085
AnhTran162
11141KMN-A 123 (4.620) Cm (122:147)
TOF MS ES+
1.42e4
539.2
541.3
542.2
100
%
0
480
485
490
495
500
505
510
515
520
525
530
535
540
545
m/z

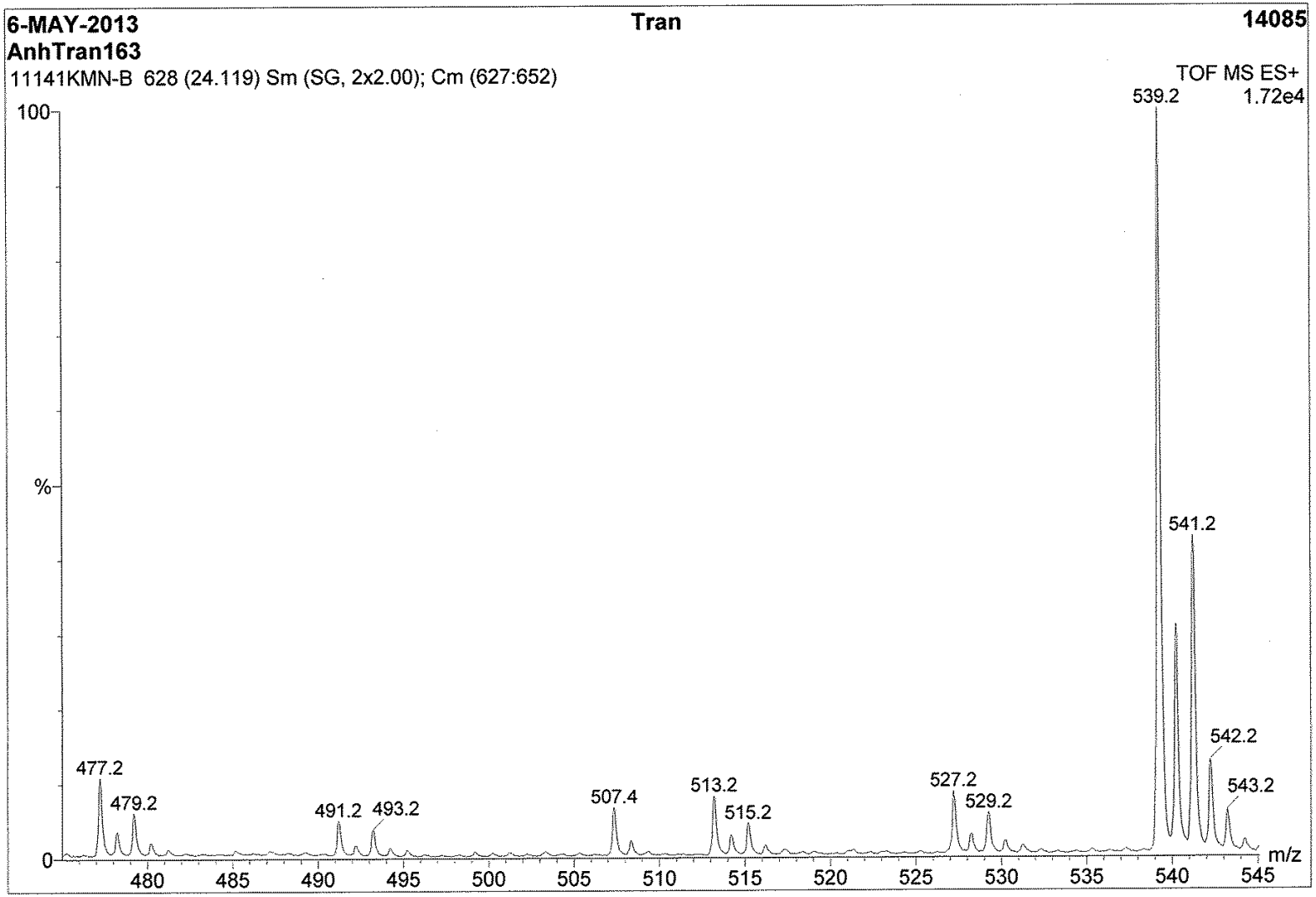
$(tButyl)_4N^+$ m/z= 242 as internal standar



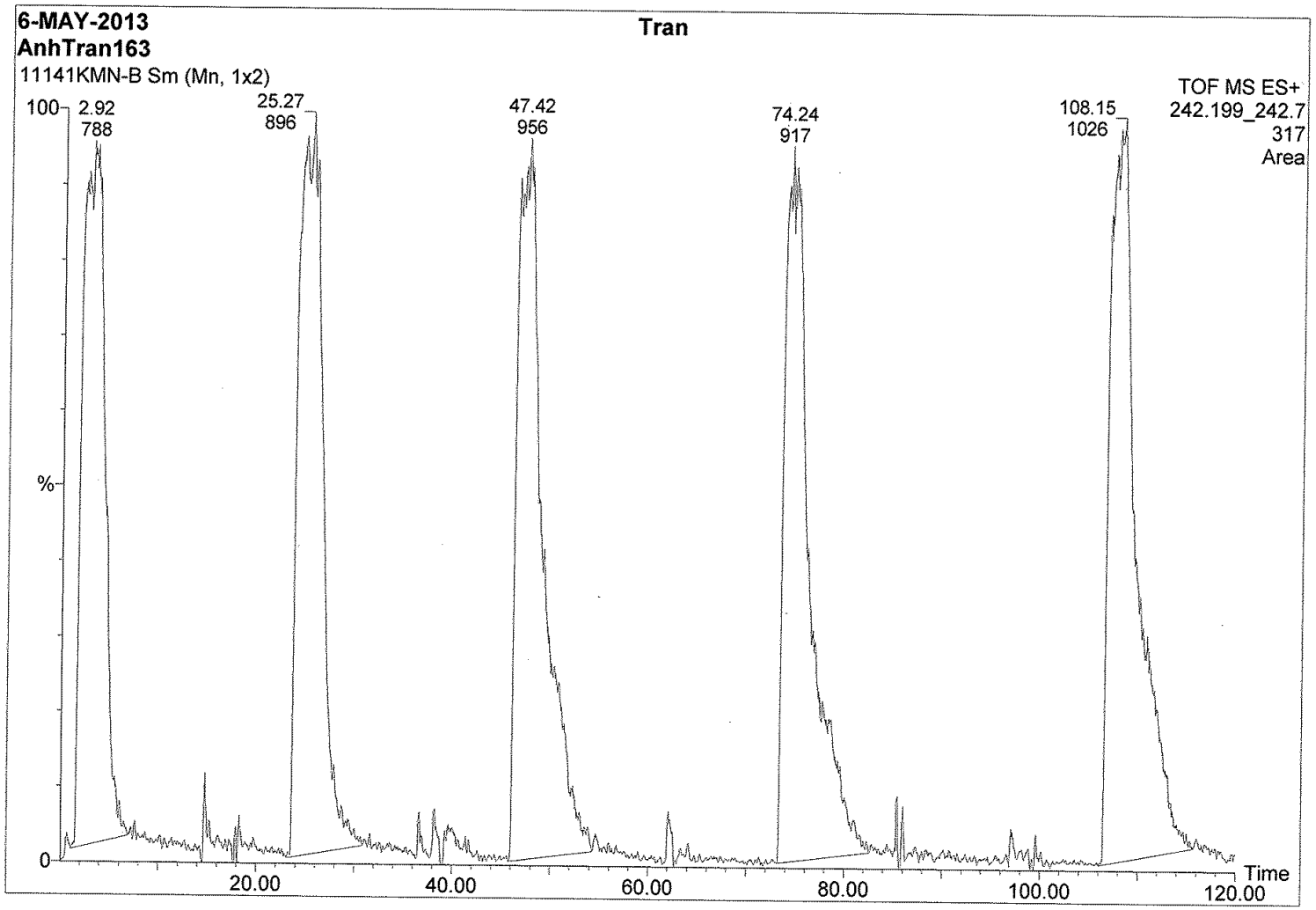
MS library of $CU(CH_3CN)_4PF_6$; **25; 7**; **8**; **9** + 100 equiv of **Prox-TSA 1**



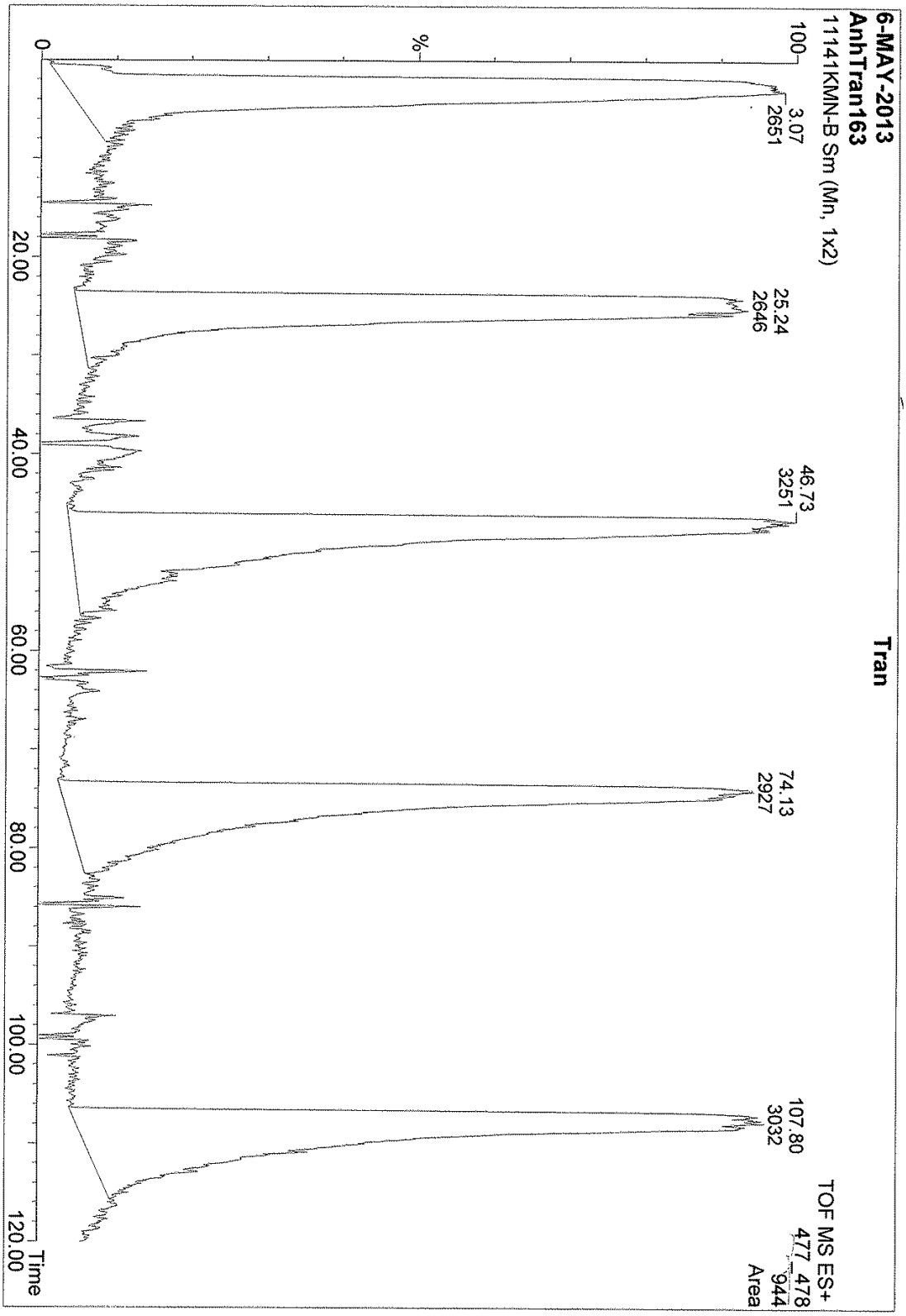

6-MAY-2013
AnhTran163
Tran
14085
11141KMN-B 2813 (107.693) AM (Cen,2, 80.00, Ar,5925.0,0.00,0.70)
TOF MS ES+
1.44e4
100
%
0
553.2
605.3
619.3
539.2
567.3
155.2
621.3
671.3
177.1
199.1
207.2
569.3
672.3
622.3
141.3
208.2
241.0
401.2
415.2
375.2
477.2
527.2
570.3
673.5
639.4
m/z
100
150
200
250
300
350
400
450
500
550
600
650
700
750


6-MAY-2013
AnhTran163
Tran
14085
11141KMN-B 628 (24.119) Sm (SG, 2x2.00); Cm (627:652)
TOF MS ES+
1.72e4
100
%
0
539.2
541.2
542.2
543.2
477.2
479.2
491.2
493.2
507.4
513.2
515.2
527.2
529.2
m/z
480
485
490
495
500
505
510
515
520
525
530
535
540
545

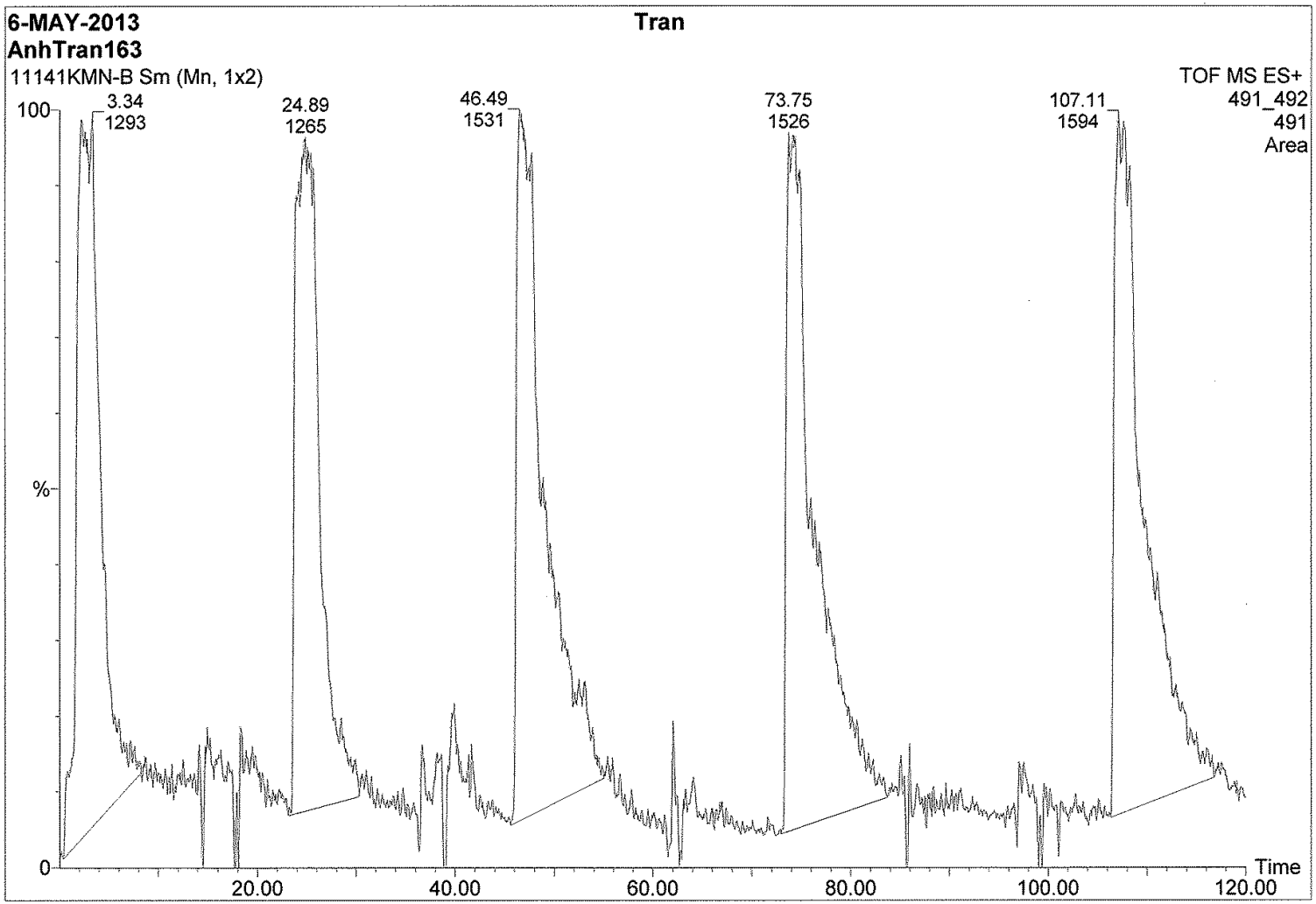
Ion count of $(tButyl)_4N^+$ m/z= 242 as internal standar



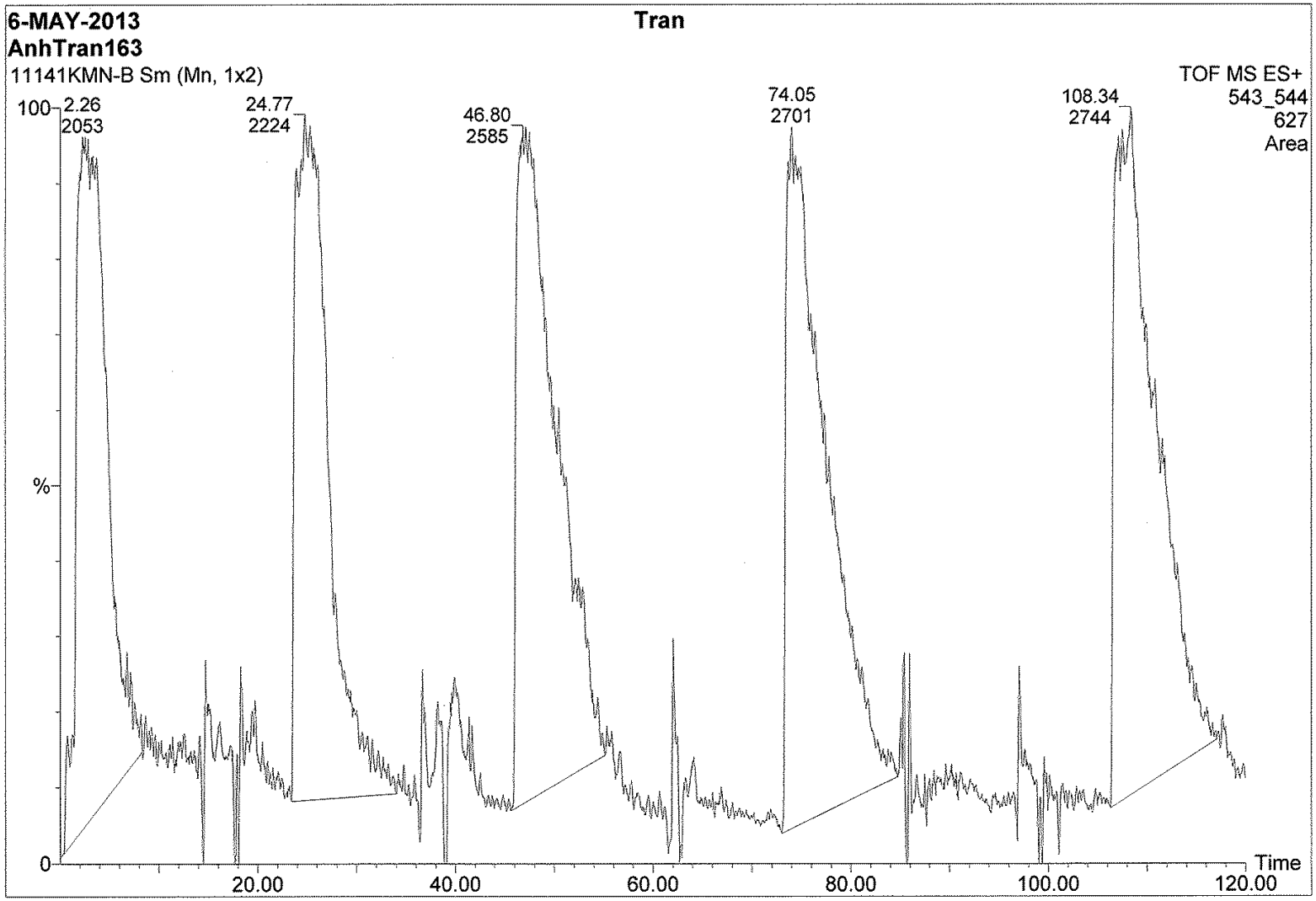
Ion count of $[Cu(\mathbf{25\text{-}7})\mathbf{Prox\text{-}TSA1}]^+$ m/z= 477



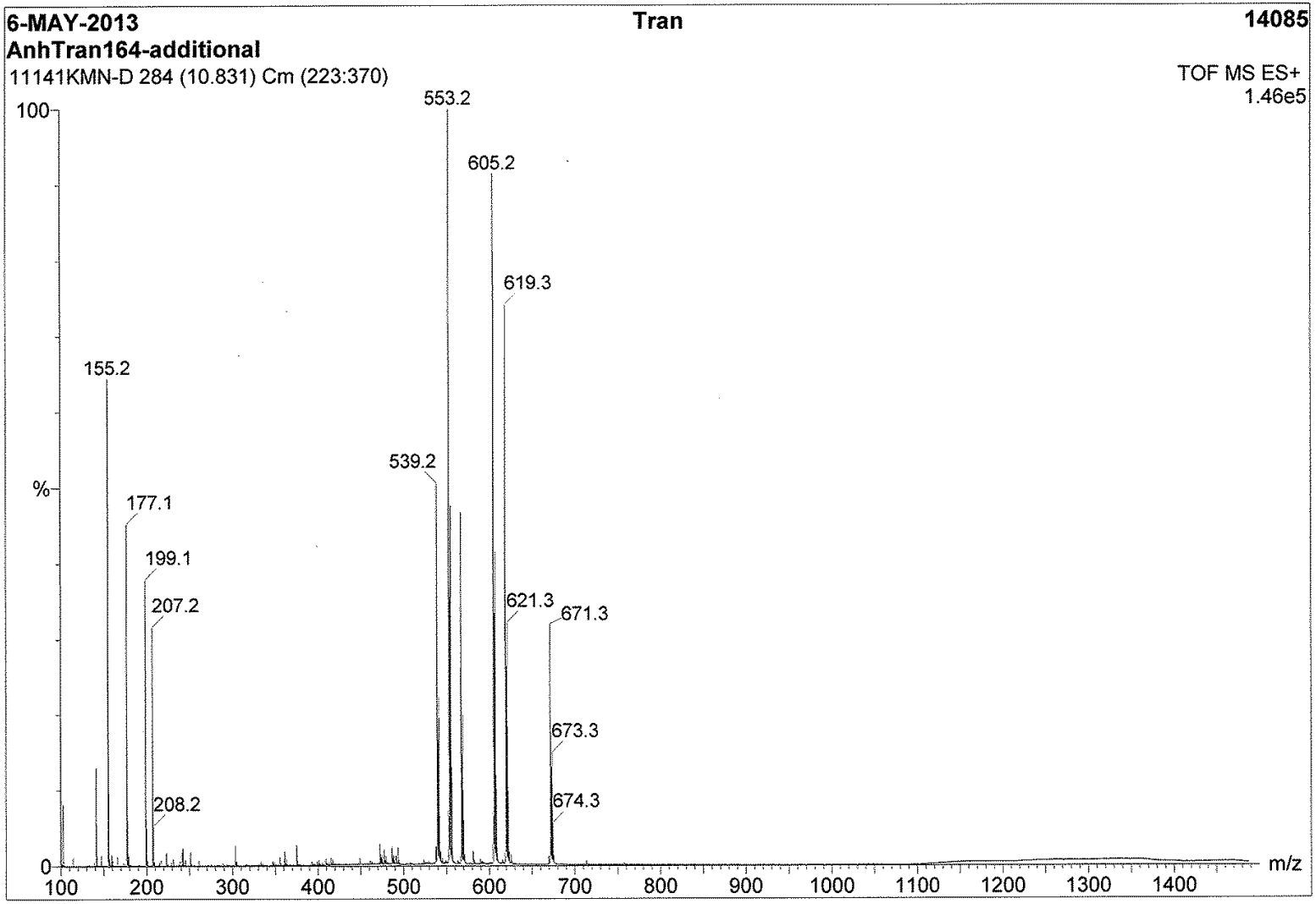
Ion count of [Cu(**25-8**)**Prox-TSA1**]$^{+}$ m/z= 491

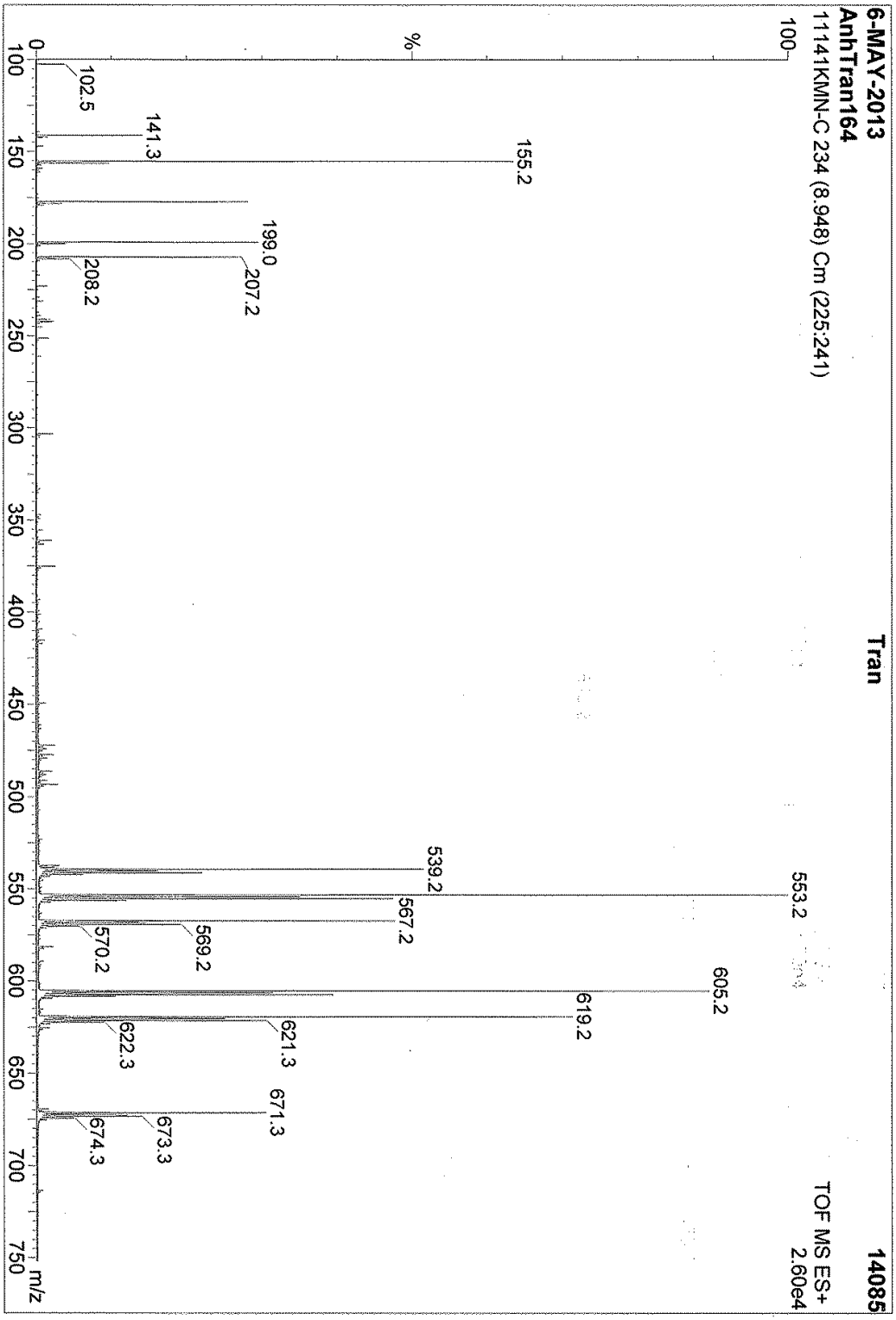


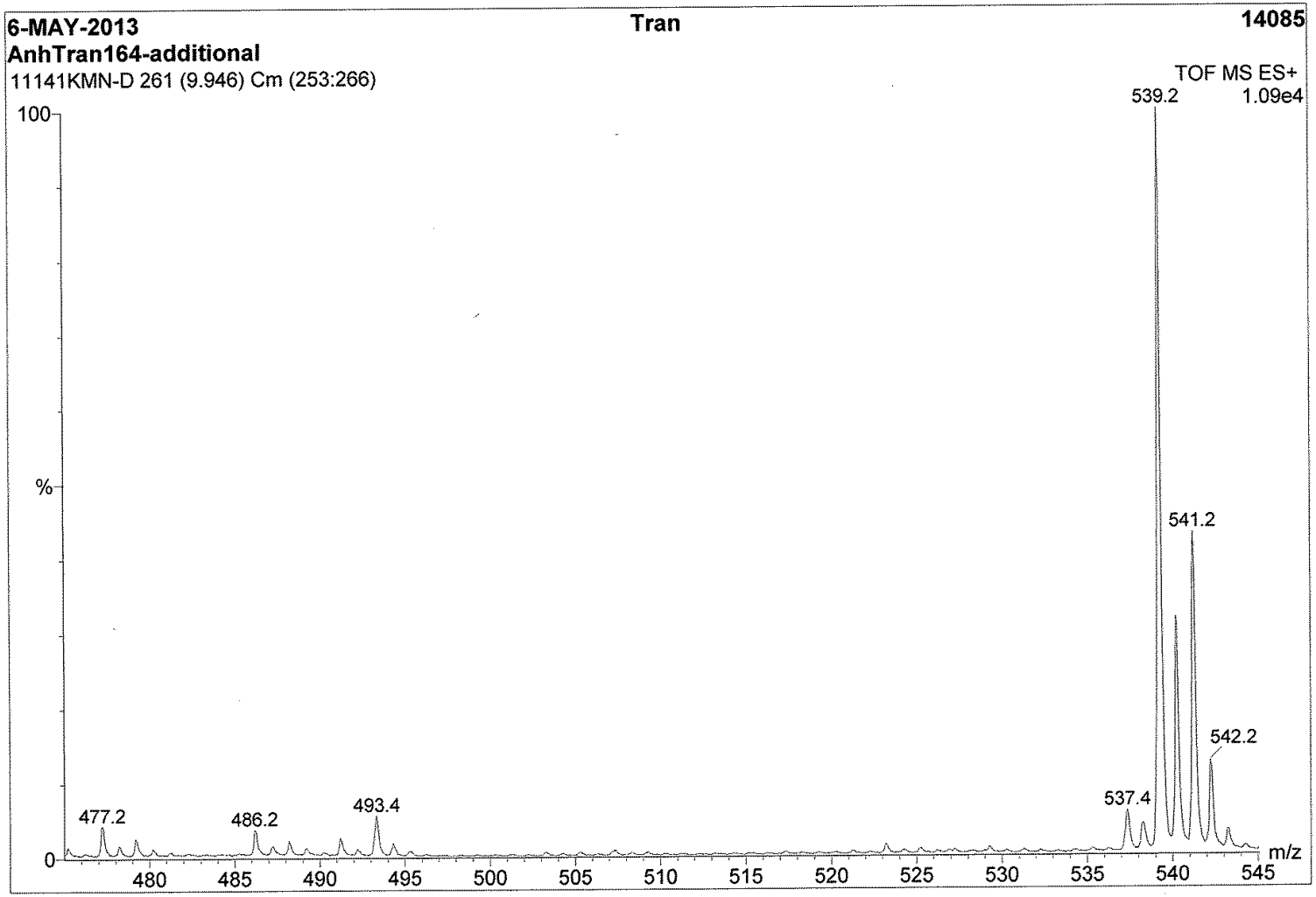
Ion count of [Cu(**25-9**)**Prox-TSA1**]$^{+}$ m/z= 543



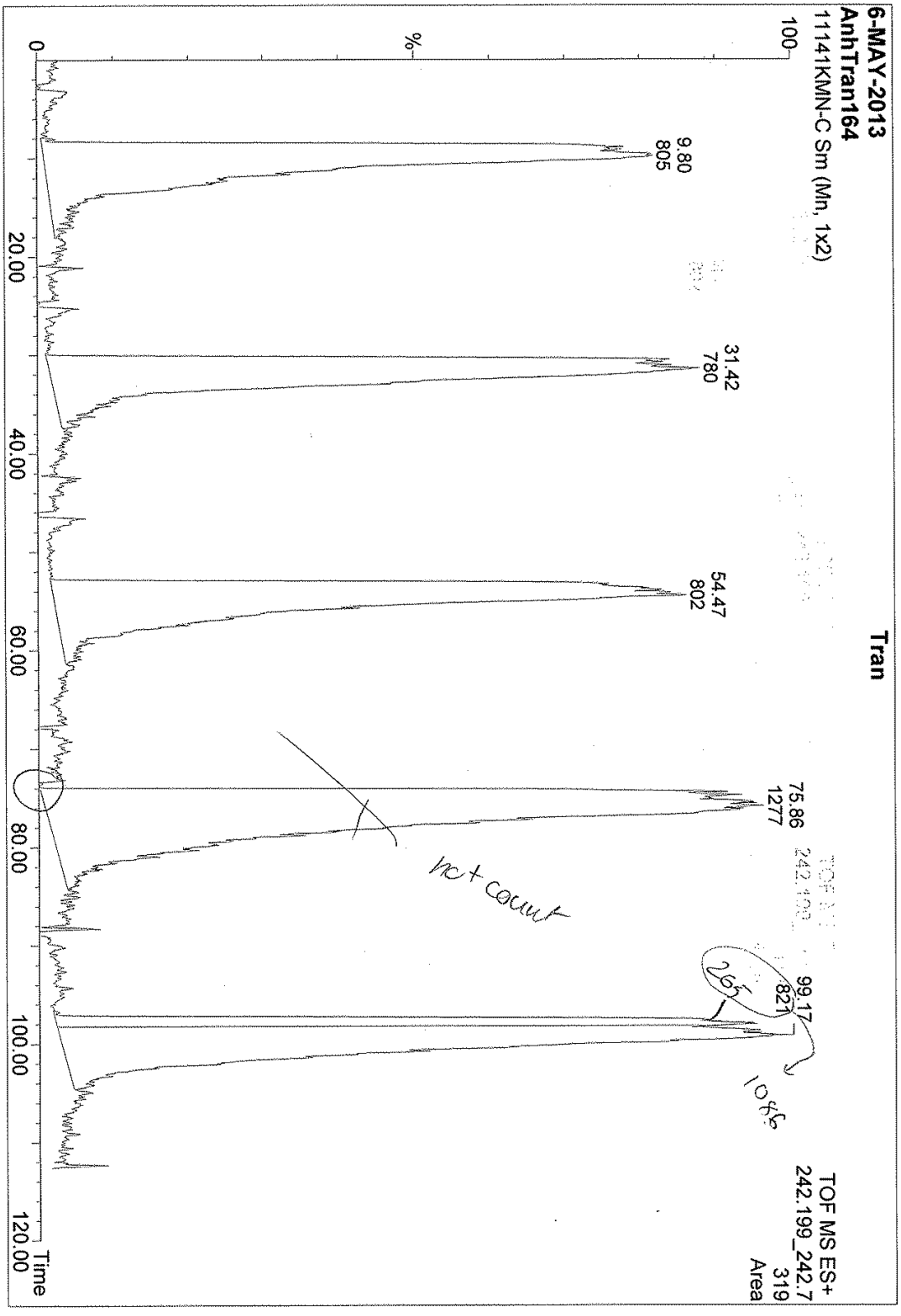
MS library of $CU(CH_3CN)_4PF_6$; **25; 7**; **8**; **9** + 100 equiv of **Distal 1**

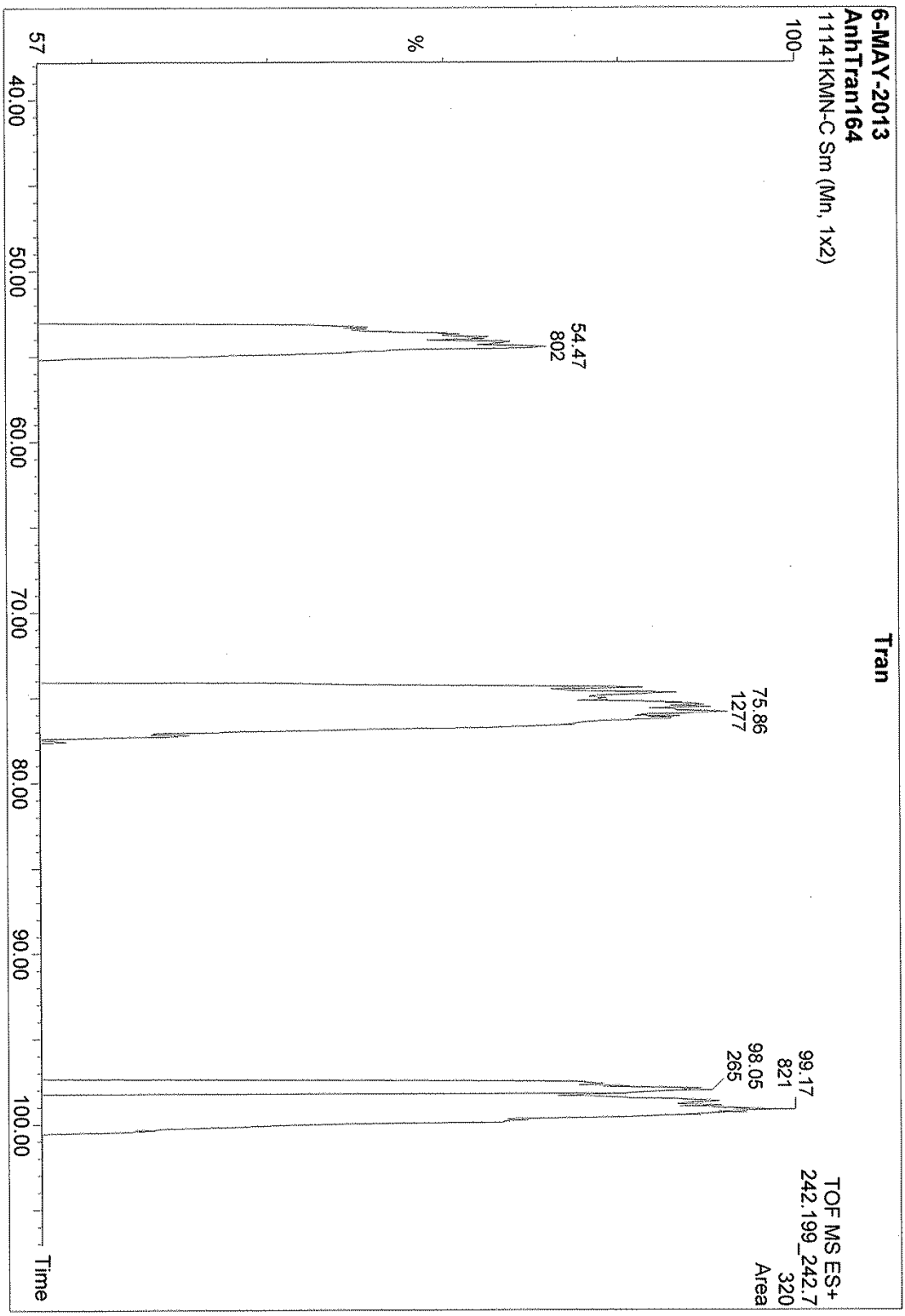


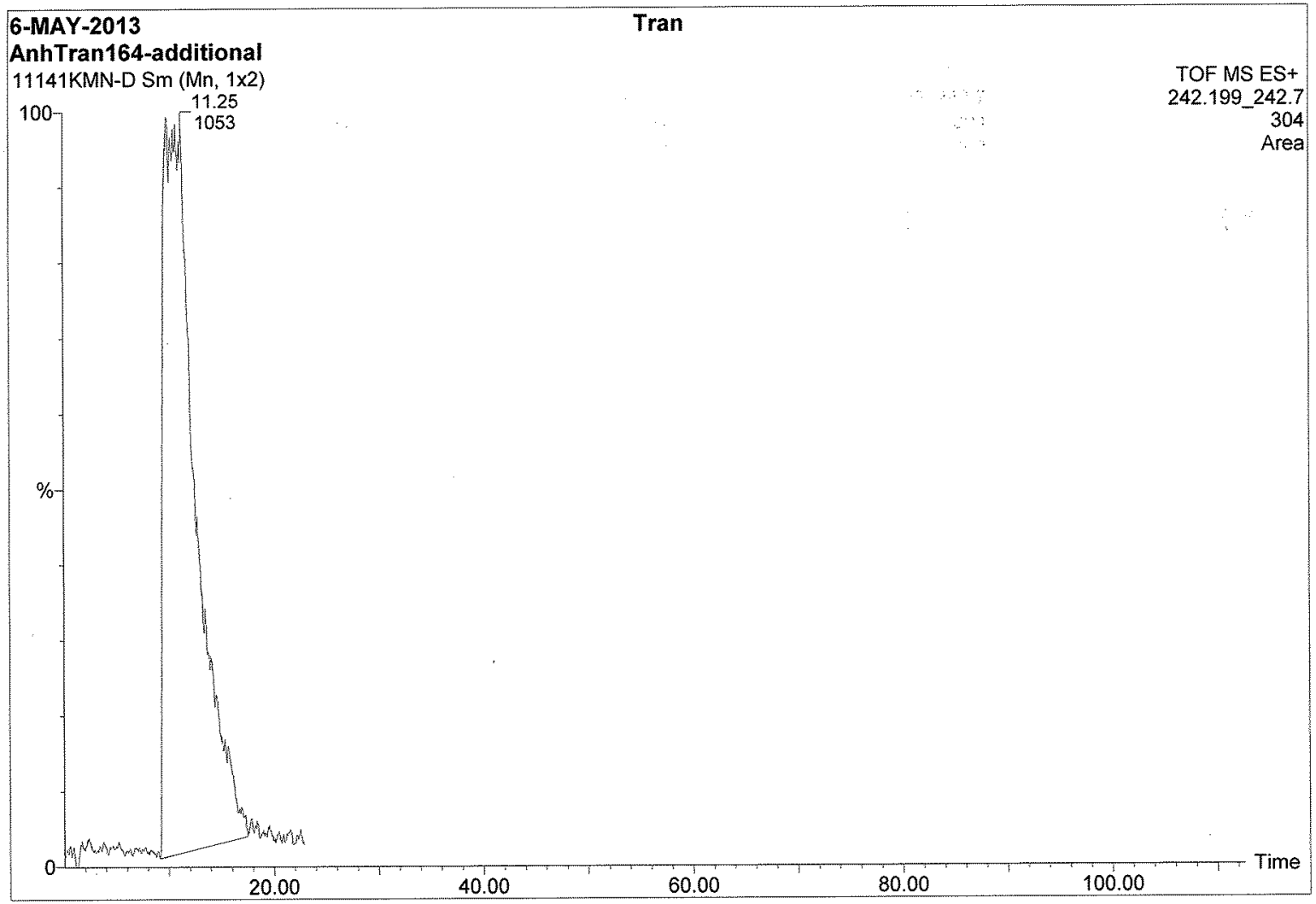

6-MAY-2013
AnhTran164
11141KMN-C 234 (8.948) Cm (225:241)
Tran
14085
TOF MS ES+
2.60e4
%
m/z
102.5
141.3
155.2
199.0
207.2
208.2
539.2
553.2
567.2
569.2
570.2
605.2
619.2
621.3
622.3
671.3
673.3
674.3


6-MAY-2013
AnhTran164-additional
11141KMN-D 261 (9.946) Cm (253:266)
Tran
14085
TOF MS ES+
1.09e4
539.2
541.2
542.2
537.4
493.4
486.2
477.2
100
%
0
480
485
490
495
500
505
510
515
520
525
530
535
540
545
m/z

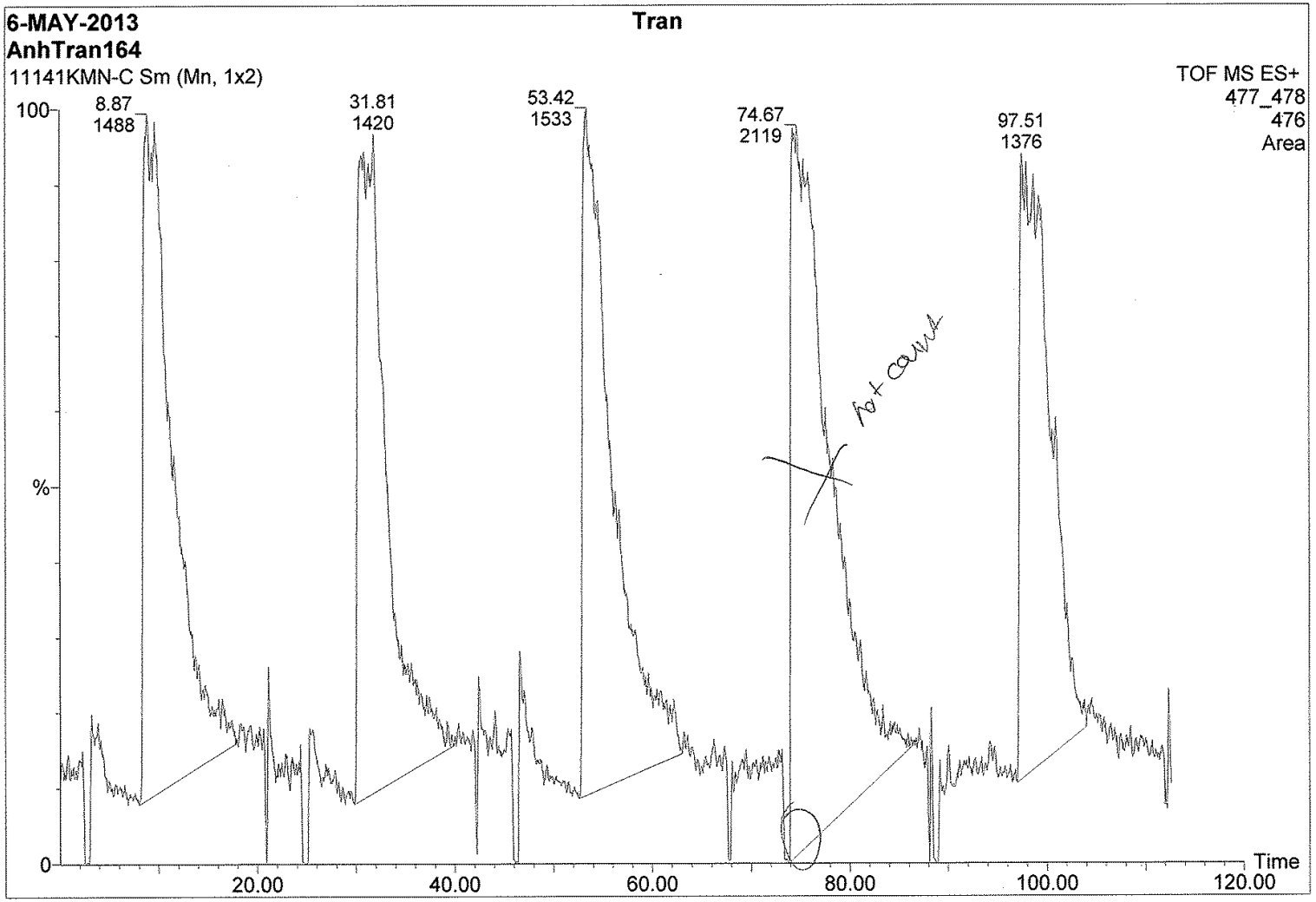
Ion count of $(tButyl)_4N^+$ m/z= 242 as internal standar

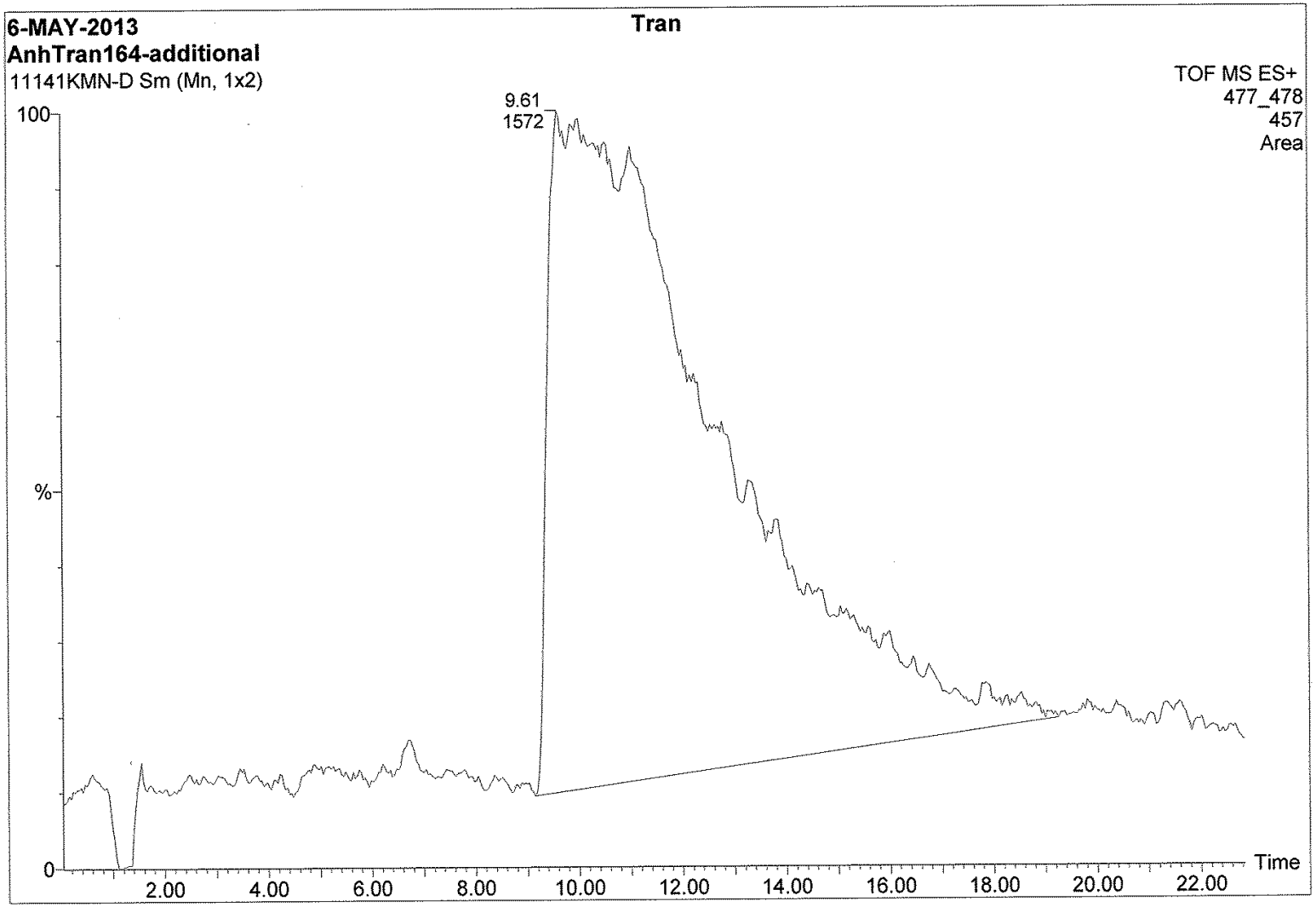



6-MAY-2013
AnhTran164
11141KMN-C Sm (Mn, 1x2)
Tran
100
%
57
40.00
50.00
60.00
70.00
80.00
90.00
100.00
Time
54.47
802
75.86
1277
99.17
821
98.05
265
TOF MS ES+
242.199_242.7
320
Area

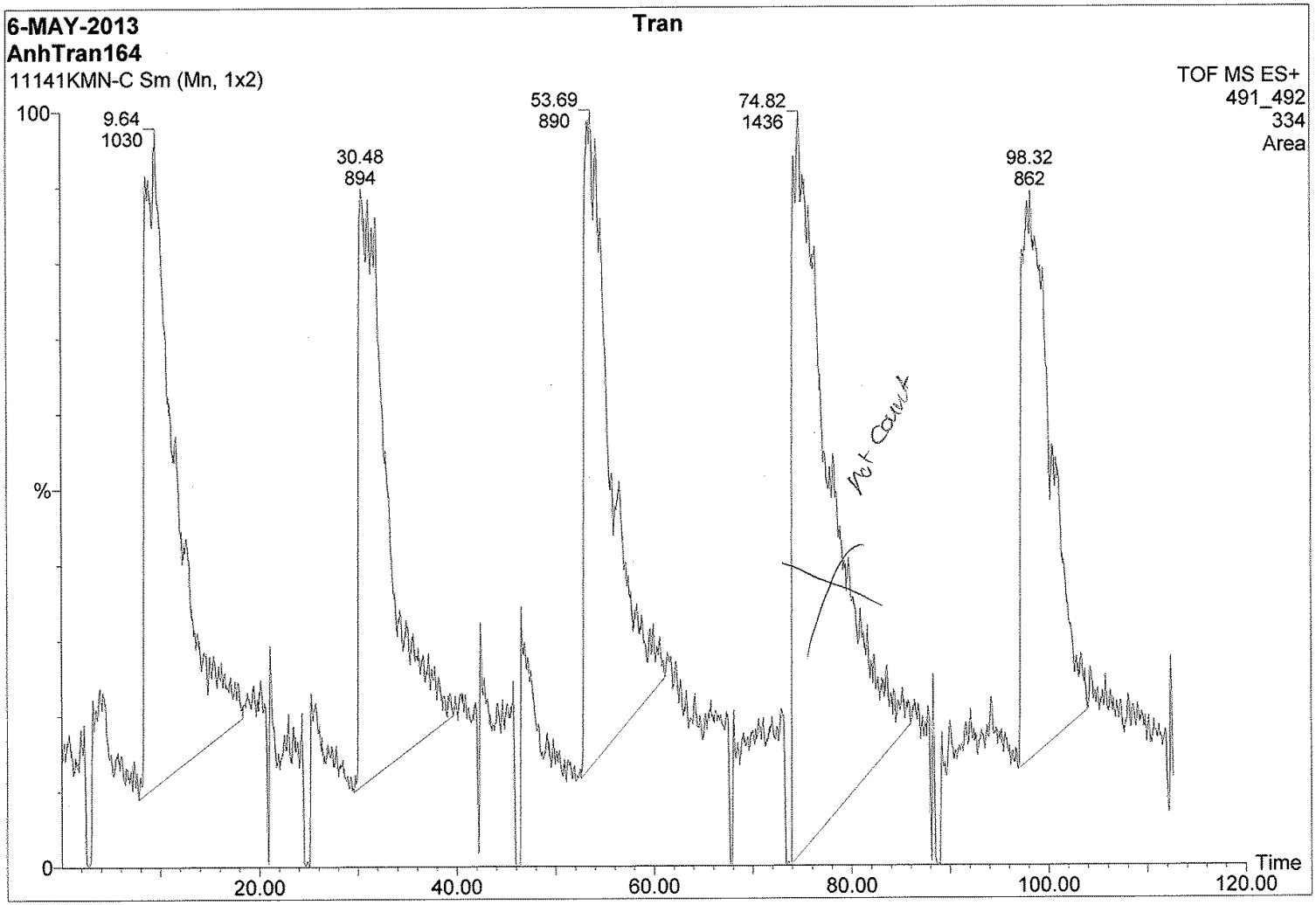

6-MAY-2013
AnhTran164-additional
11141KMN-D Sm (Mn, 1x2)
Tran
TOF MS ES+
242.199_242.7
304
Area
11.25
1053
100
%
0
20.00
40.00
60.00
80.00
100.00
Time

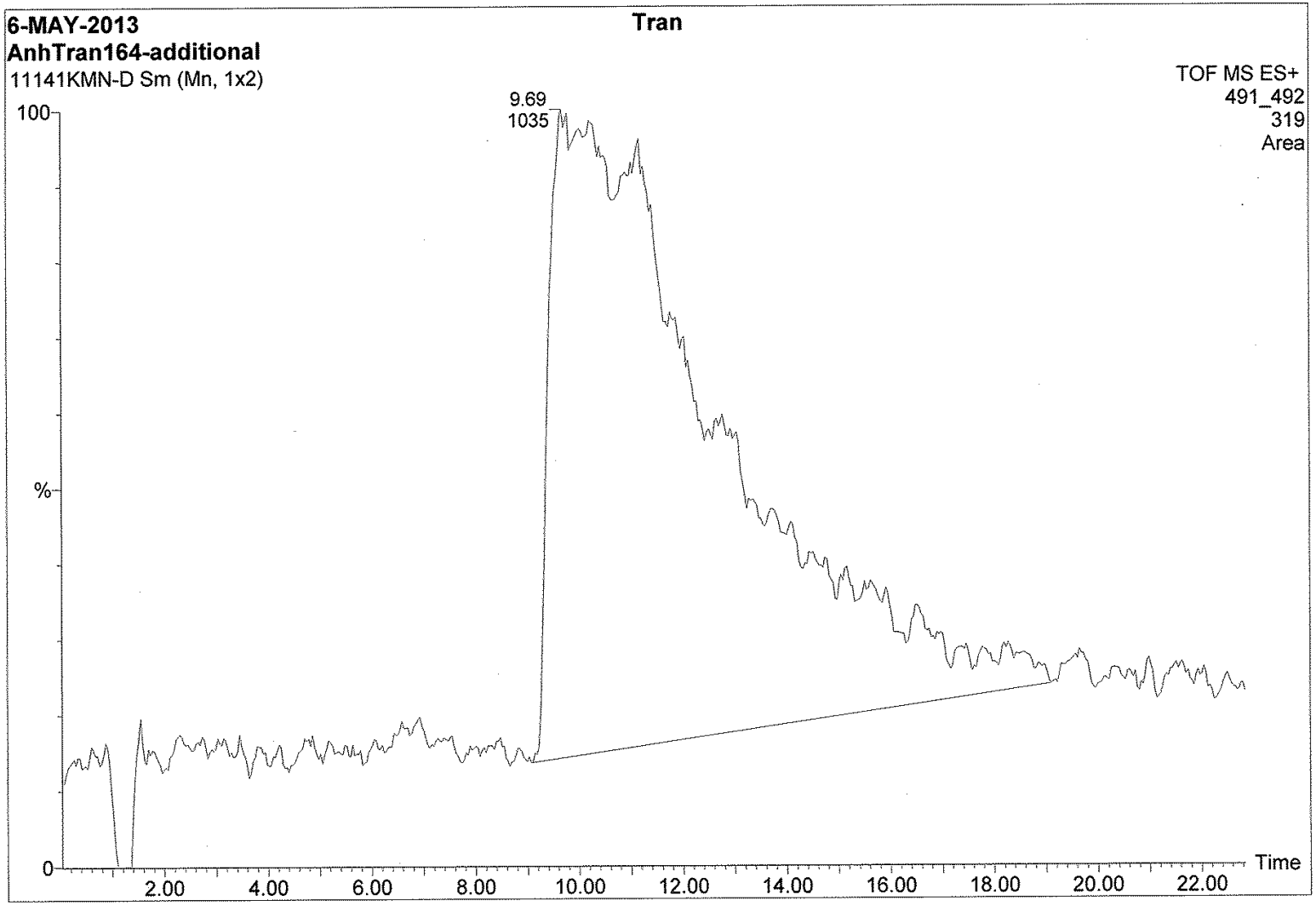
Ion count of $[Cu(25\text{-}7)Dist\text{-}TSA1]^{+}$ m/z= 477



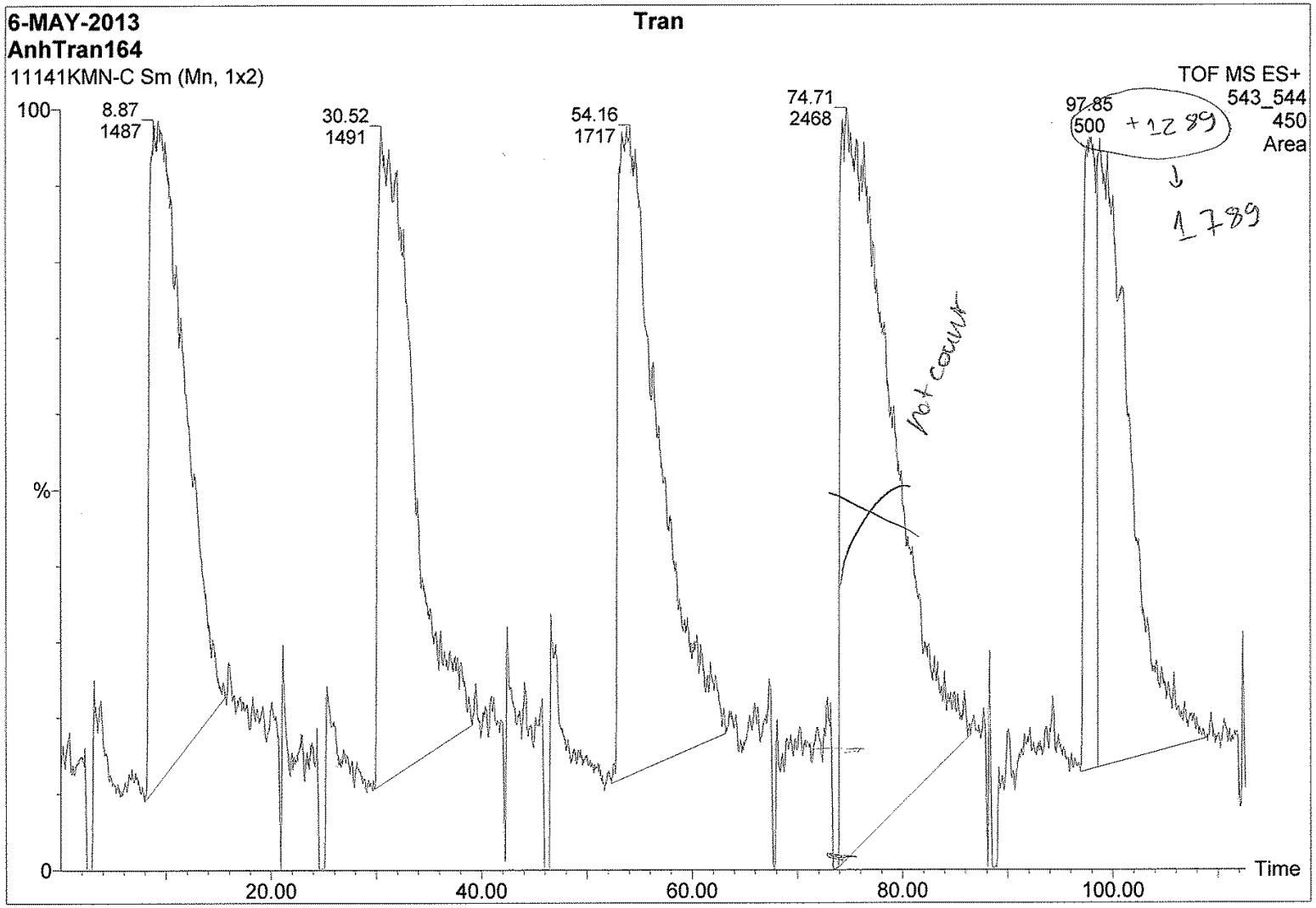

6-MAY-2013
AnhTran164-additional
11141KMN-D Sm (Mn, 1x2)
Tran
TOF MS ES+
477_478
457
Area
9.61
1572
100
%
0
Time
2.00
4.00
6.00
8.00
10.00
12.00
14.00
16.00
18.00
20.00
22.00

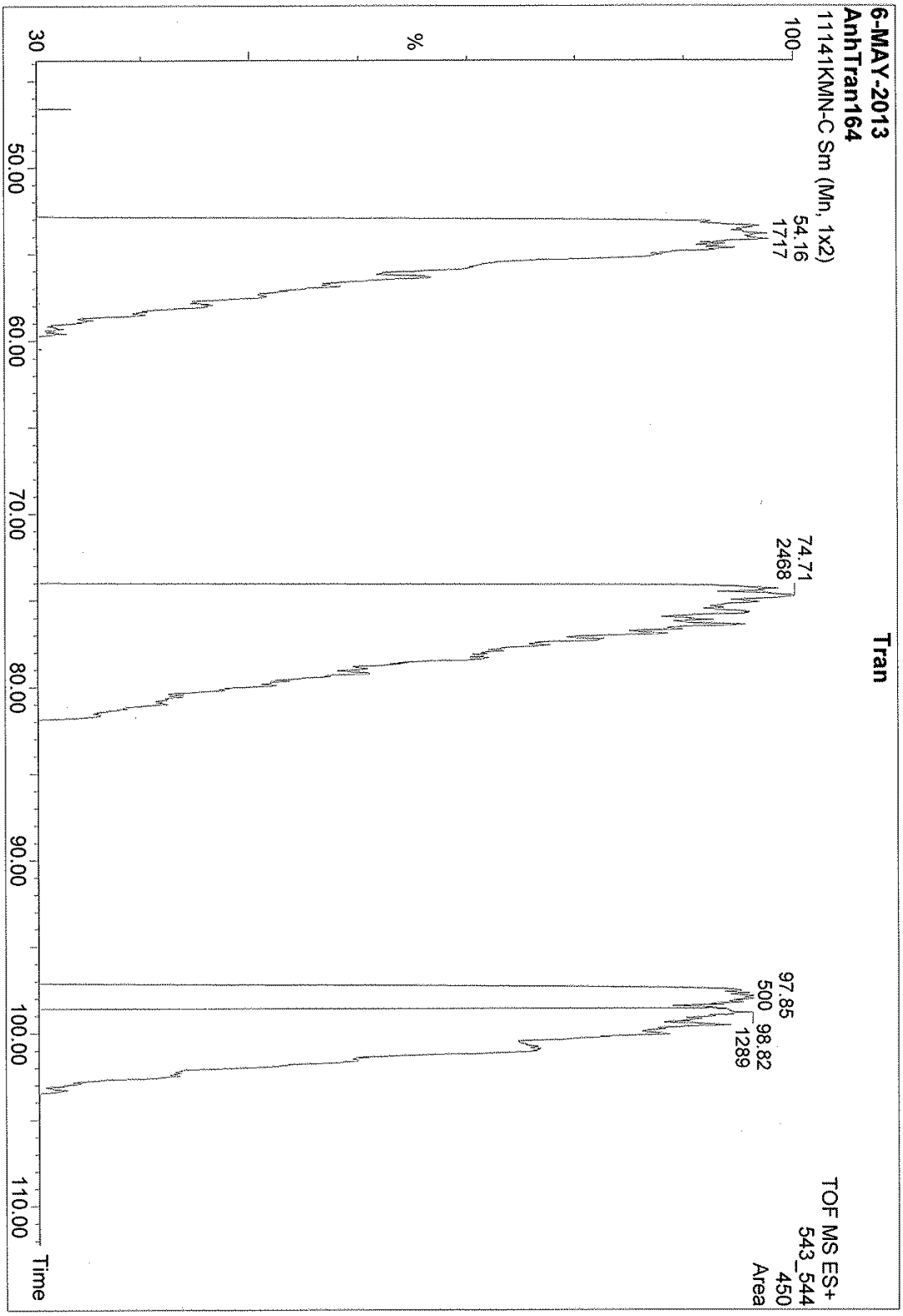
Ion count of [Cu(**25-8**)**Dist-TSA1**]$^{+}$ m/z= 491

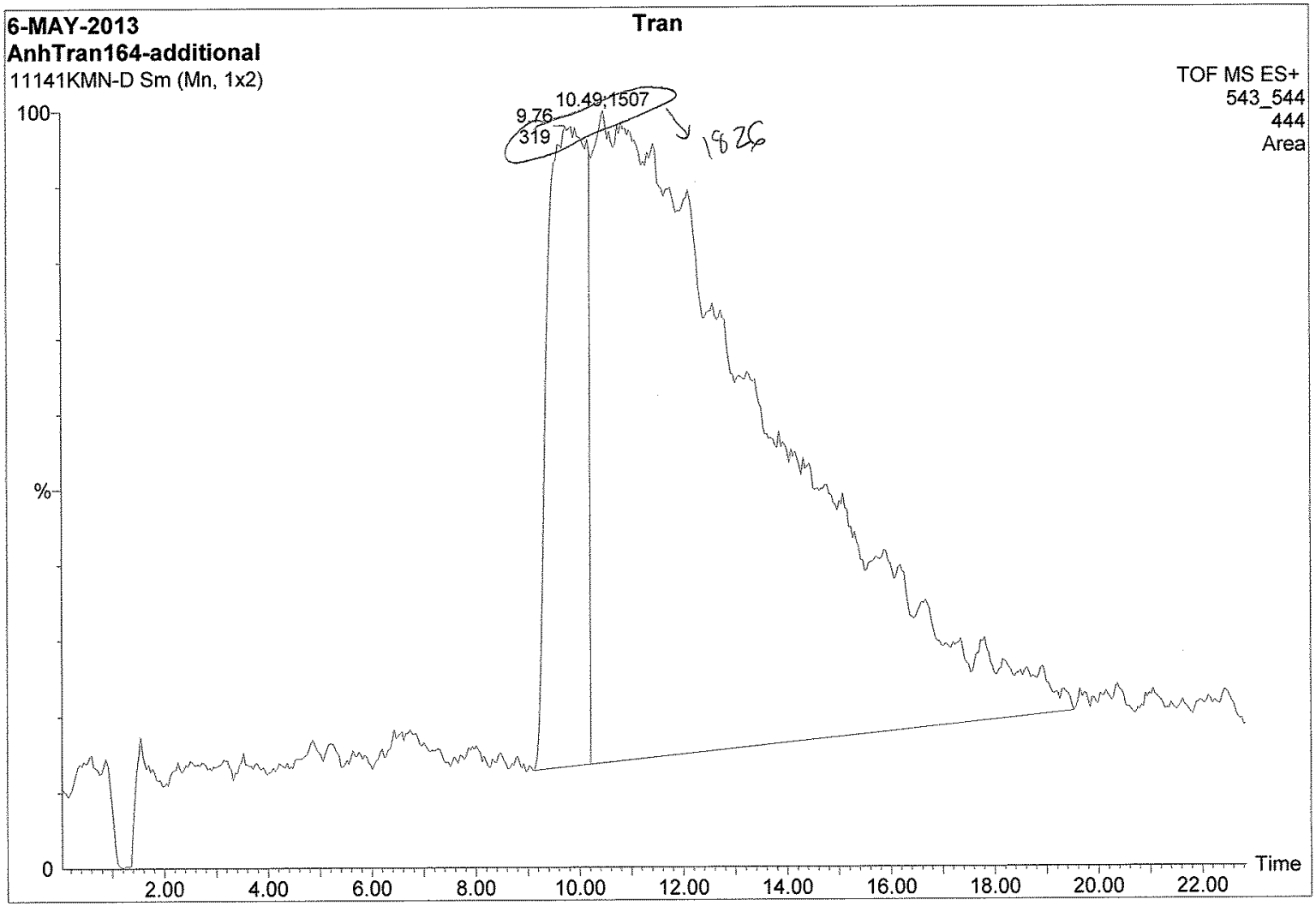



6-MAY-2013
AnhTran164-additional
11141KMN-D Sm (Mn, 1x2)
Tran
TOF MS ES+
491_492
319
Area
9.69
1035
100
%
0
Time
2.00
4.00
6.00
8.00
10.00
12.00
14.00
16.00
18.00
20.00
22.00

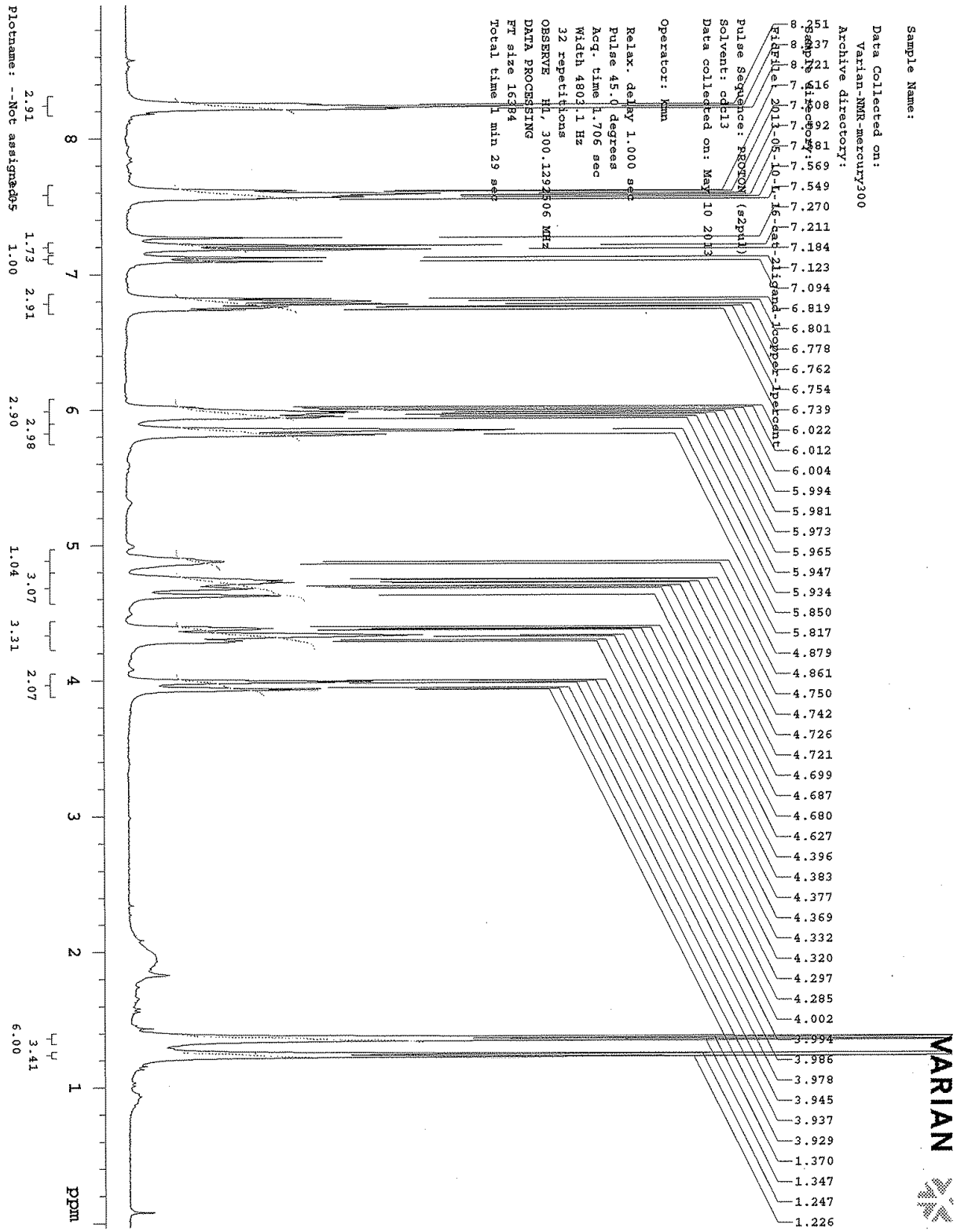
Ion count of [Cu(**25-9**)**Dist-TSA1**]$^{+}$ m/z= 543

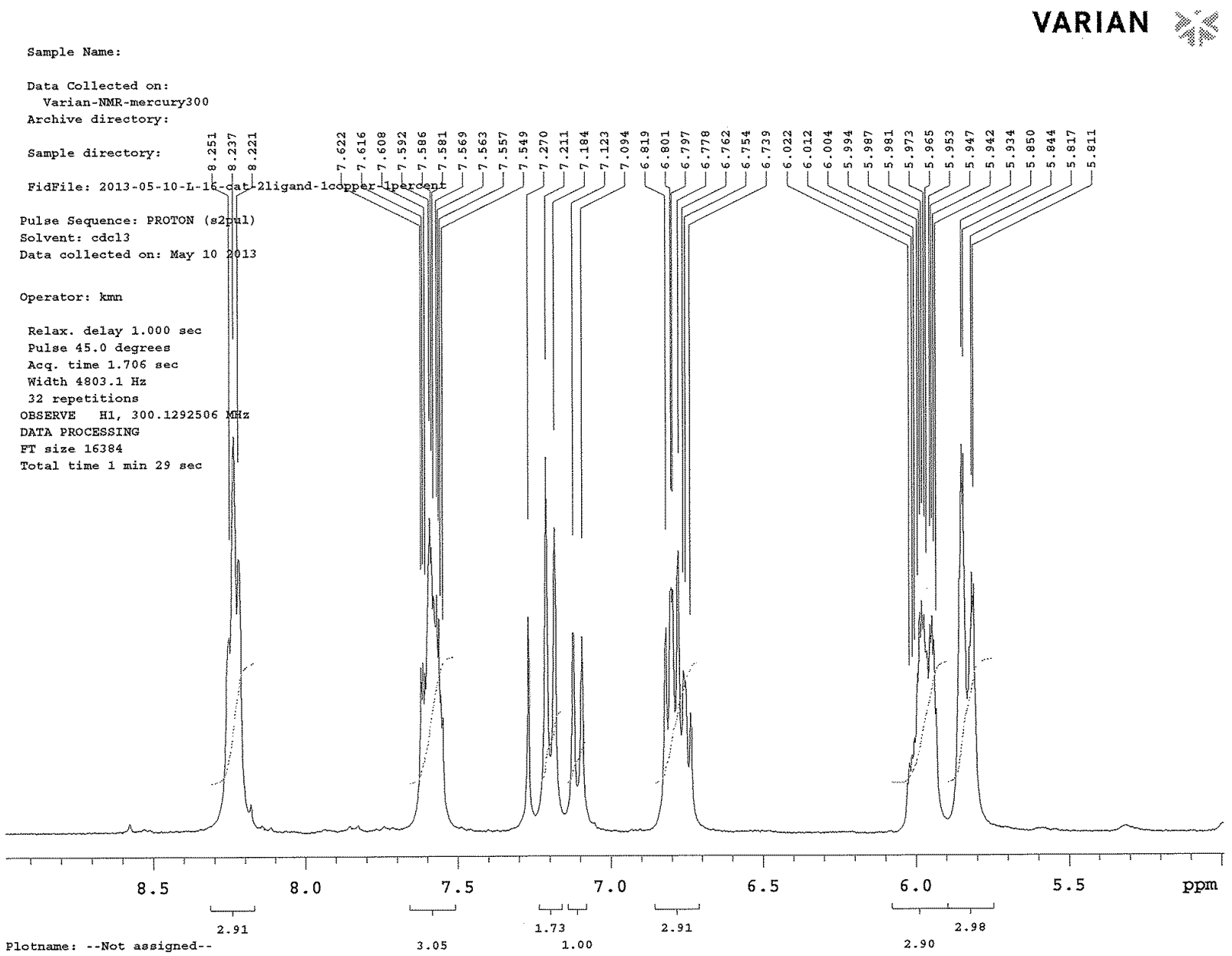


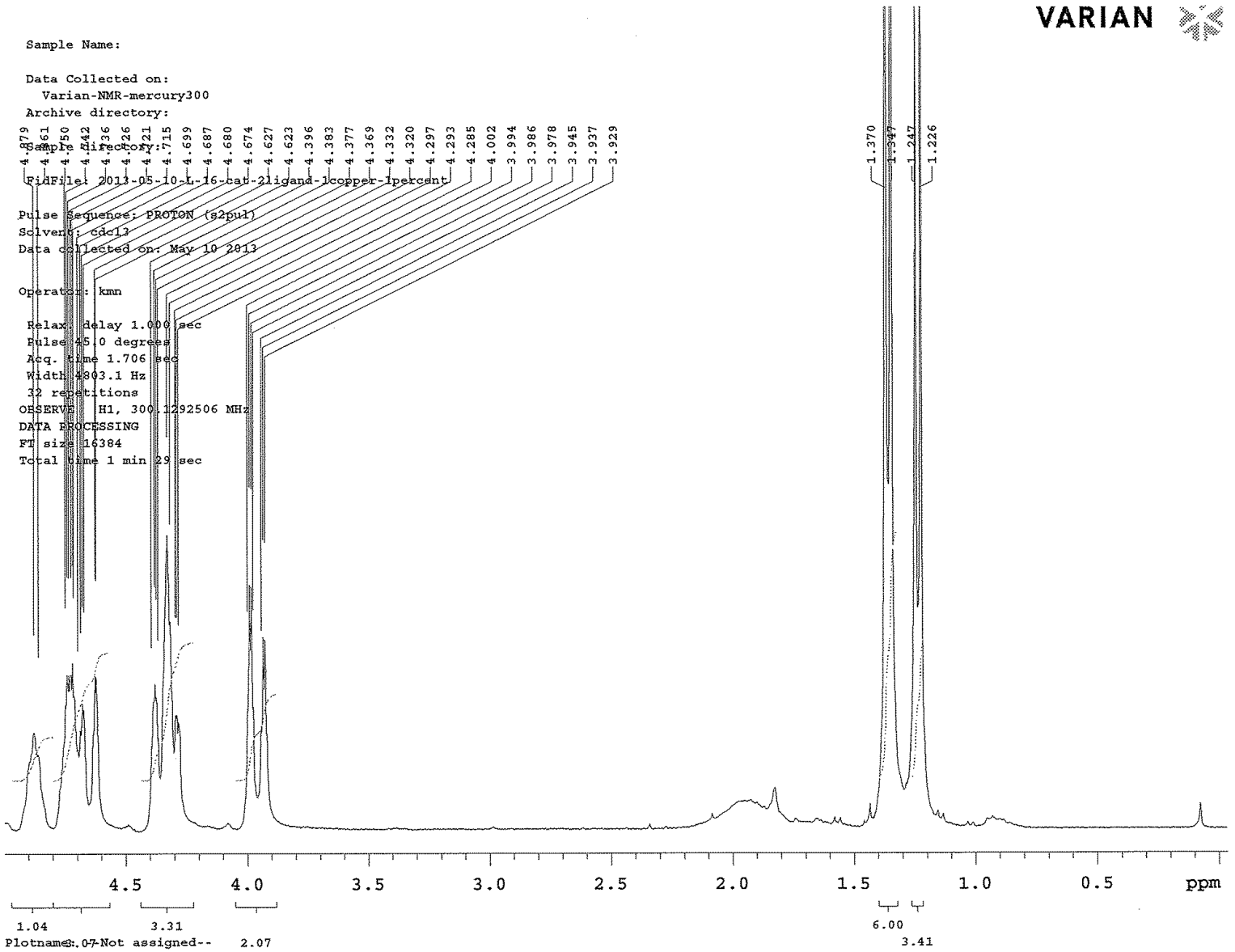

6-MAY-2013
AnhTran164
11141KMN-C Sm (Mn, 1x2)
Tran
TOF MS ES+
543_544
450
Area
100
%
54.16
1717
74.71
2468
97.85
500
98.82
1289
30
50.00
60.00
70.00
80.00
90.00
100.00
110.00
Time


6-MAY-2013
AnhTran164-additional
11141KMN-D Sm (Mn, 1x2)
Tran
TOF MS ES+
543_544
444
Area
10.49,1507
9.76
319
1826
100
%
0
Time
2.00
4.00
6.00
8.00
10.00
12.00
14.00
16.00
18.00
20.00
22.00

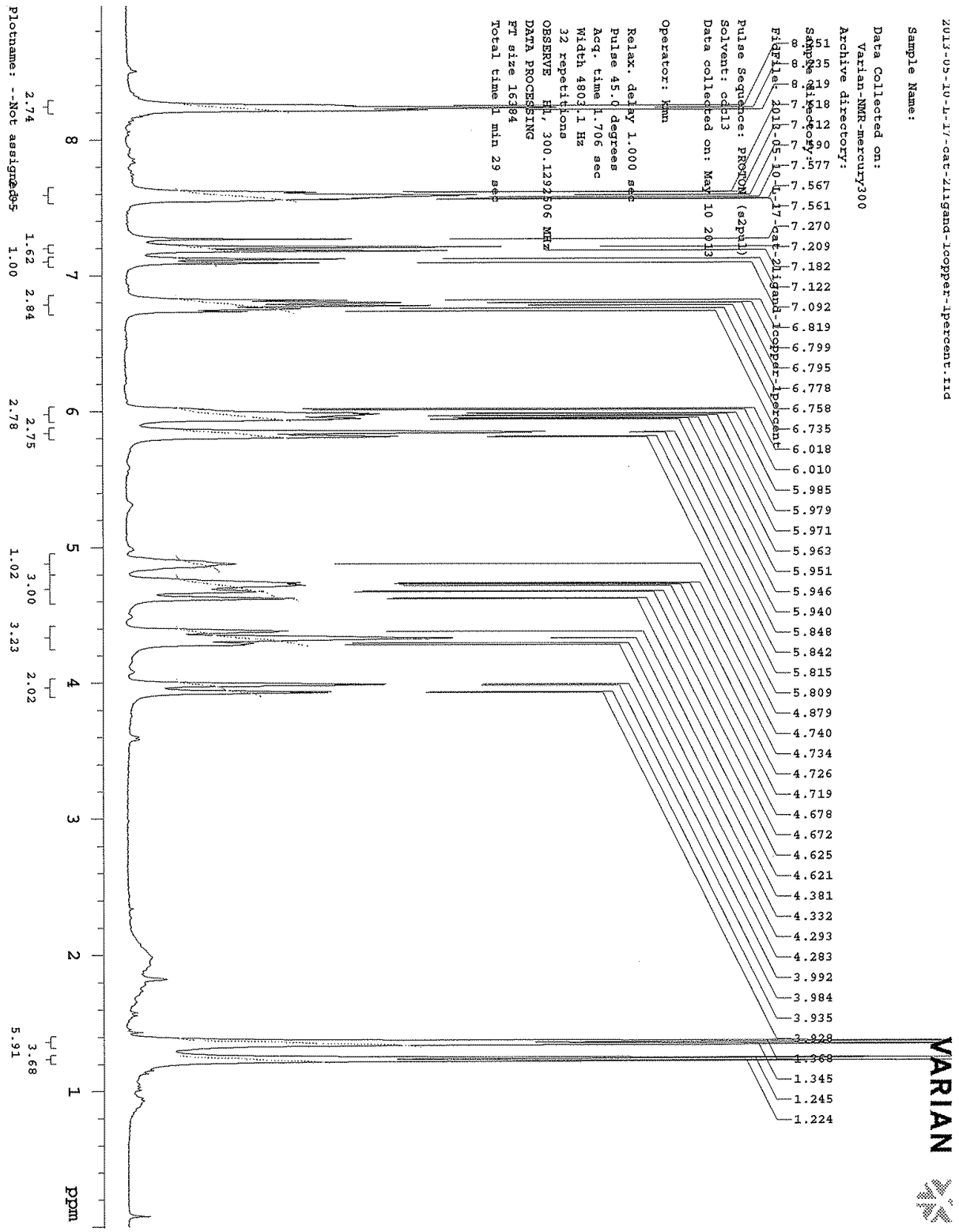
^{1}H-NMR Product mixture of $Cu(\mathbf{25\text{-}7})_2PF_6$ catalytic test

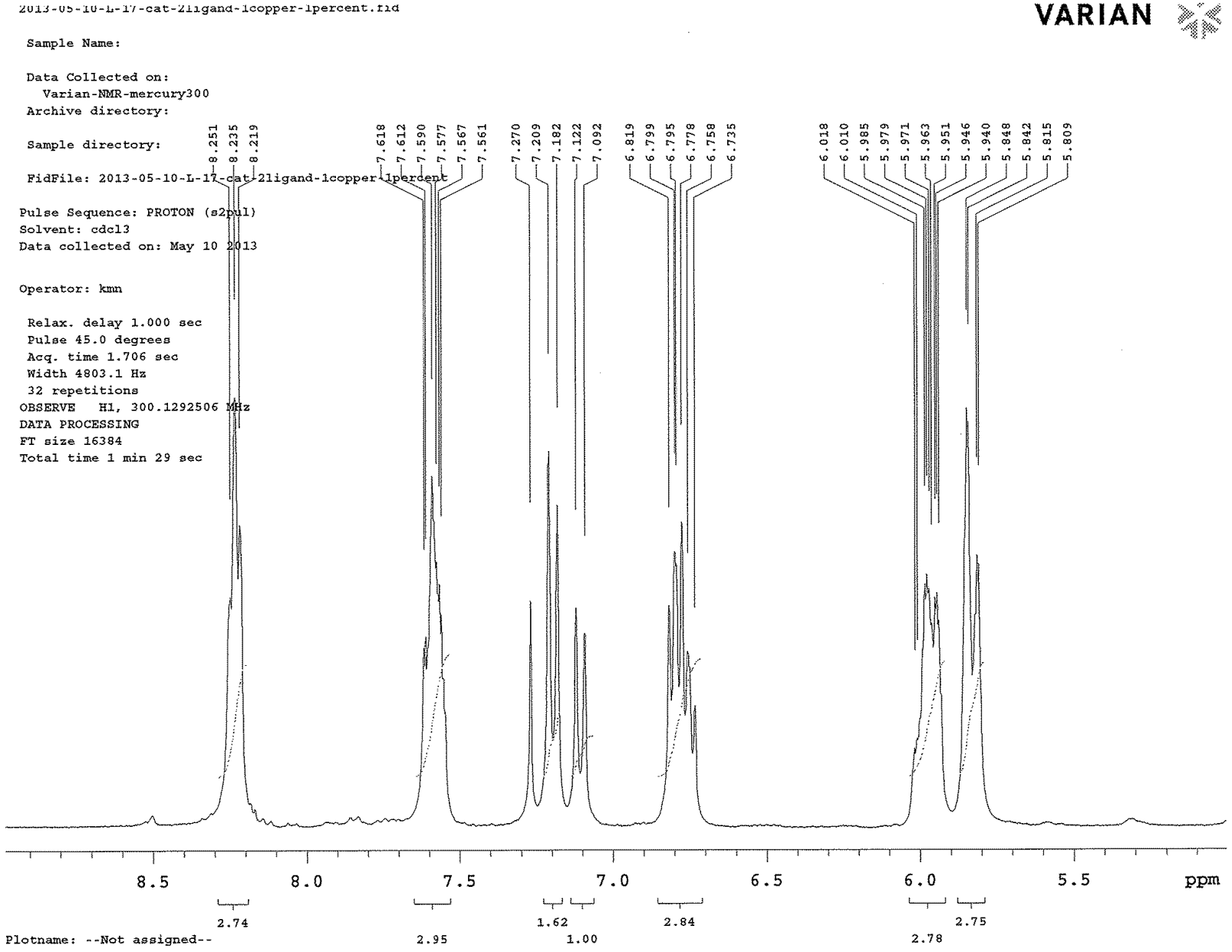


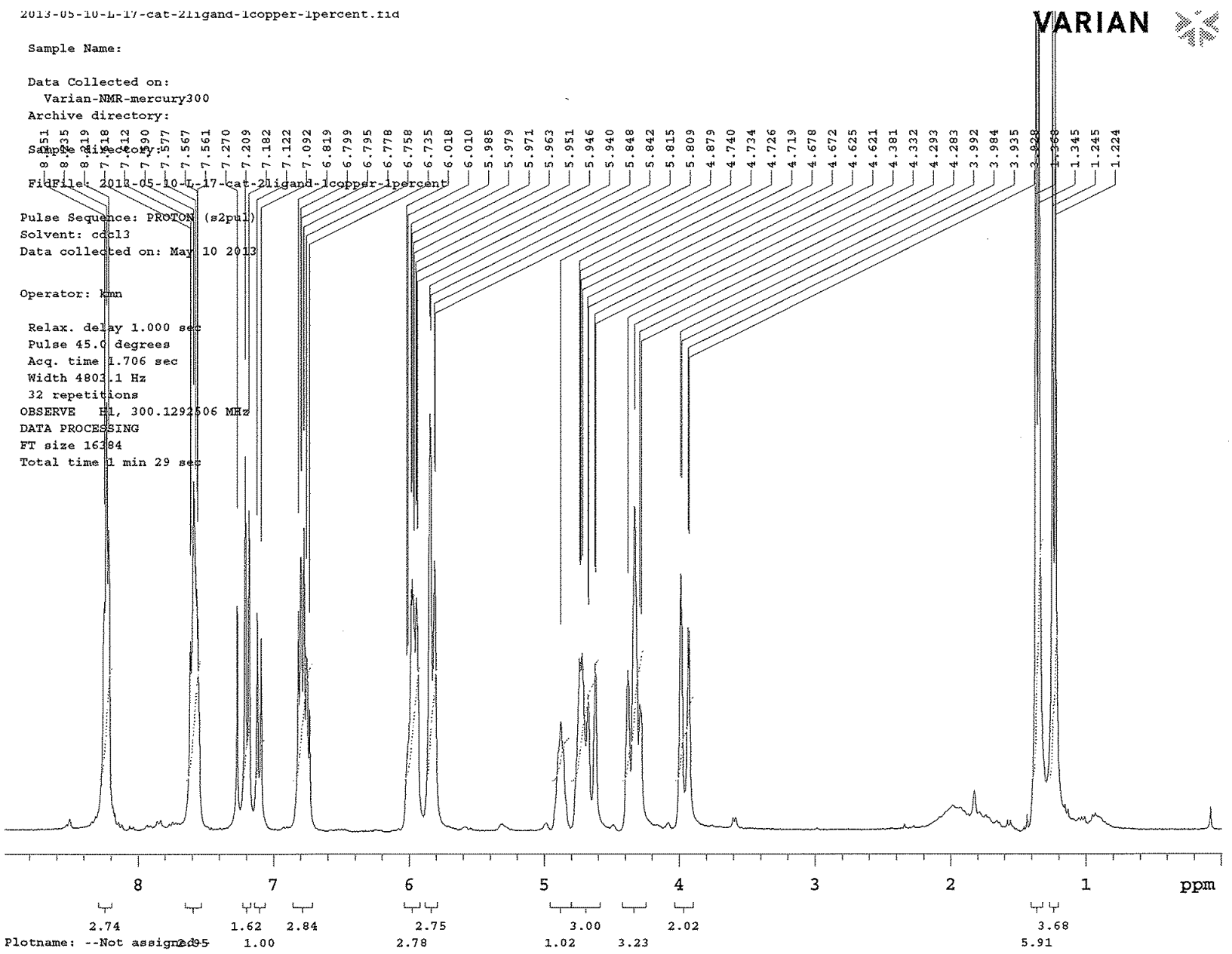

VARIAN
Sample Name:
Data Collected on:
Varian-NMR-mercury300
Archive directory:
Sample directory:
FidFile: 2013-05-10-L-16-cat-2ligand-1copper-1percent
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 10 2013
Operator: kmn
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.706 sec
Width 4803.1 Hz
32 repetitions
OBSERVE H1, 300.1292506 MHz
DATA PROCESSING
FT size 16384
Total time 1 min 29 sec
8.251
8.237
8.221
7.622
7.616
7.608
7.592
7.586
7.581
7.569
7.563
7.557
7.549
7.270
7.211
7.184
7.123
7.094
6.819
6.801
6.797
6.778
6.762
6.754
6.739
6.022
6.012
6.004
5.994
5.987
5.981
5.973
5.965
5.953
5.947
5.942
5.934
5.850
5.844
5.817
5.811
8.5
8.0
7.5
7.0
6.5
6.0
5.5
ppm
2.91
3.05
1.73
1.00
2.91
2.98
2.90
Plotname: --Not assigned--


VARIAN
Sample Name:
Data Collected on:
Varian-NMR-mercury300
Archive directory:
Sample directory:
FidFile: 2013-05-10-L-16-cat-2ligand-1copper-1percent
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 10 2013
Operator: kmn
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.706 sec
Width 4803.1 Hz
32 repetitions
OBSERVE H1, 300.1292506 MHz
DATA PROCESSING
FT size 16384
Total time 1 min 29 sec
4.879
4.861
4.850
4.842
4.836
4.826
4.821
4.715
4.699
4.687
4.680
4.674
4.627
4.623
4.396
4.383
4.377
4.369
4.332
4.320
4.297
4.293
4.285
4.002
3.994
3.986
3.978
3.945
3.937
3.929
1.370
1.347
1.247
1.226
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.04
3.31
2.07
6.00
3.41
Plotname: 07-Not assigned--

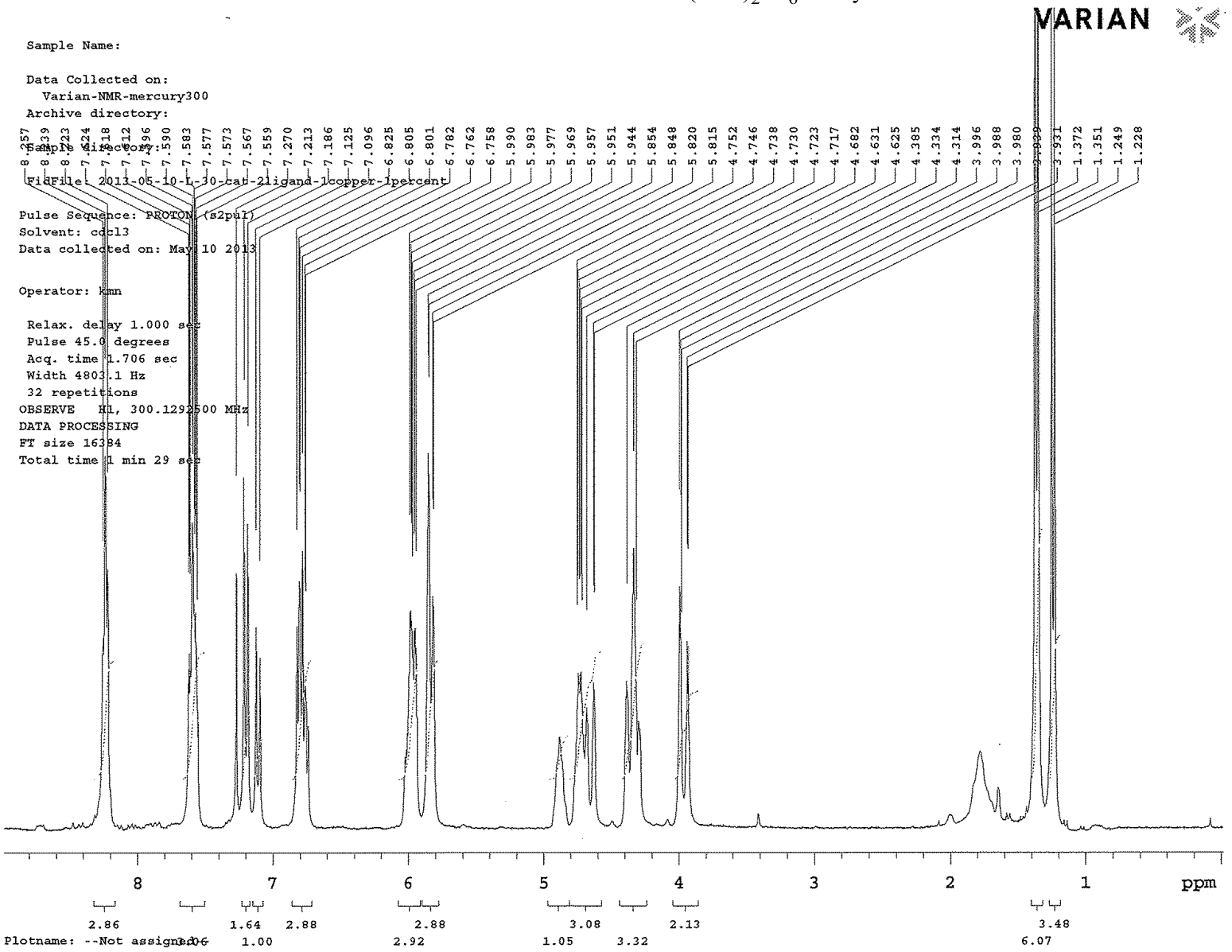
^{1}H-NMR Product mixture of Cu(**25-8**)$_2$PF$_6$ catalytic test

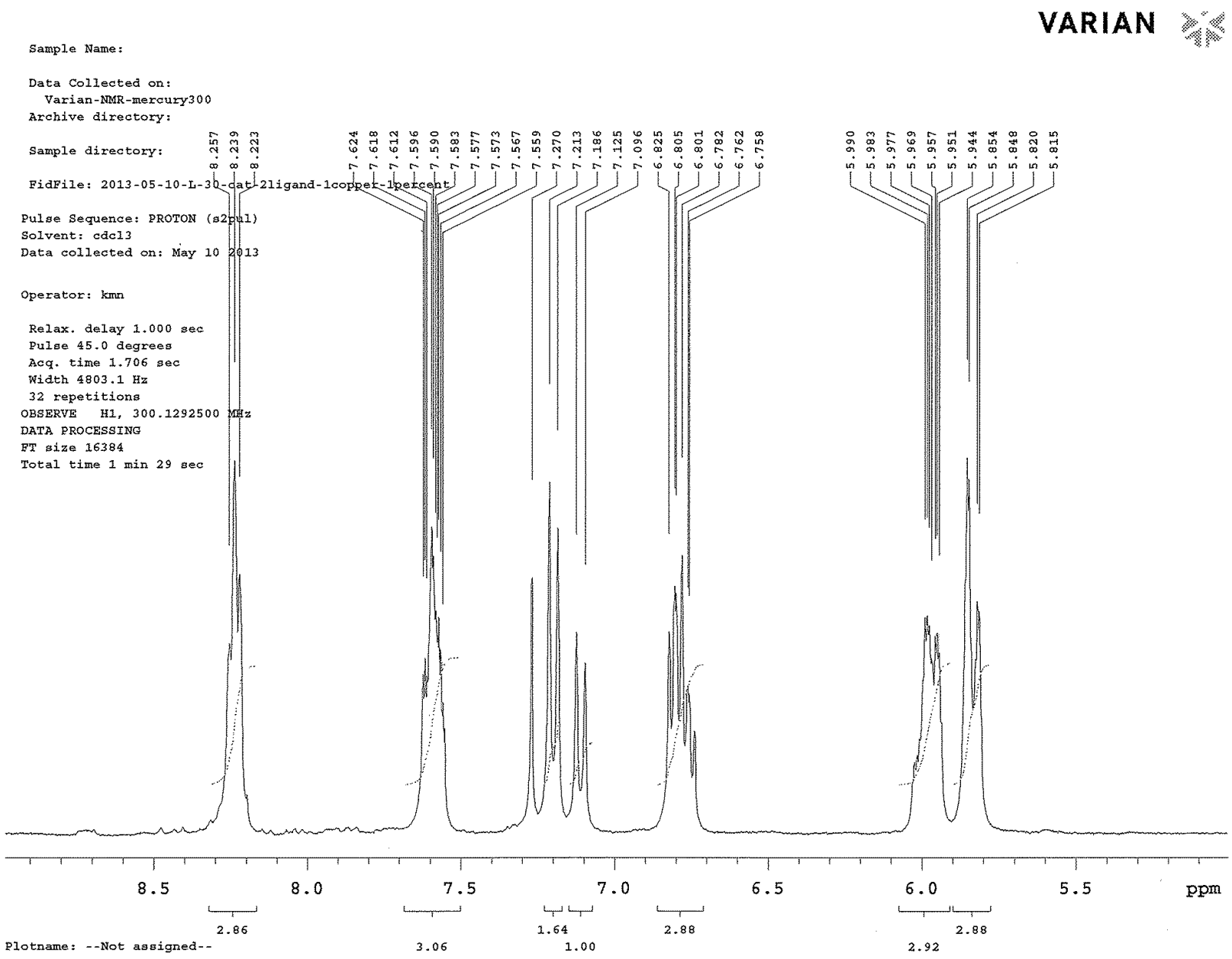


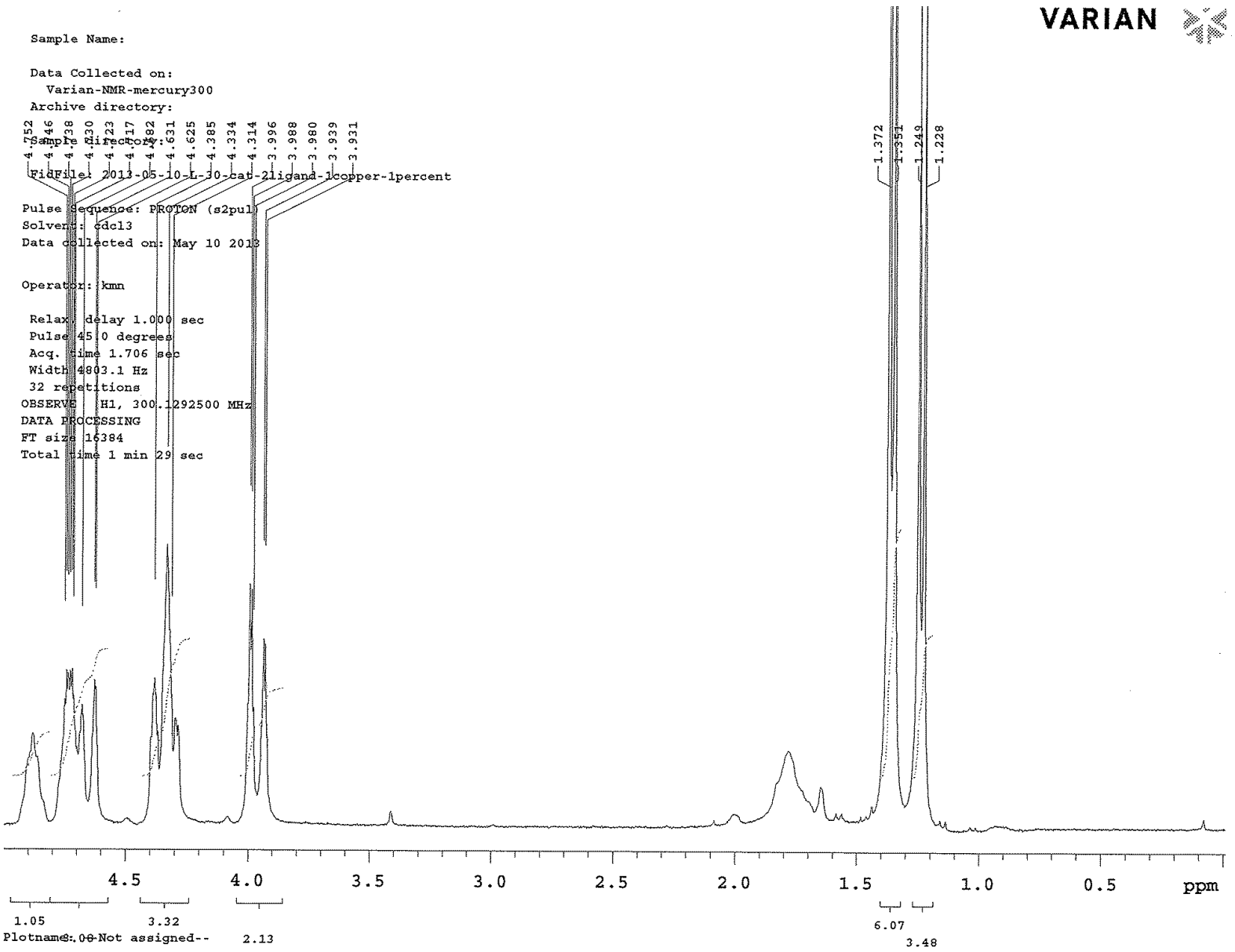

2013-05-10-L-17-cat-2ligand-1copper-1percent.fid
Sample Name:
Data Collected on:
Varian-NMR-mercury300
Archive directory:
Sample directory:
FidFile: 2013-05-10-L-17-cat-2ligand-1copper-1percent
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 10 2013
Operator: kmn
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.706 sec
Width 4803.1 Hz
32 repetitions
OBSERVE H1, 300.1292506 MHz
DATA PROCESSING
FT size 16384
Total time 1 min 29 sec
8.251
8.235
8.219
7.618
7.612
7.590
7.577
7.567
7.561
7.270
7.209
7.182
7.122
7.092
6.819
6.799
6.795
6.778
6.758
6.735
6.018
6.010
5.985
5.979
5.971
5.963
5.951
5.946
5.940
5.848
5.842
5.815
5.809
8.5
8.0
7.5
7.0
6.5
6.0
5.5
ppm
2.74
2.95
1.62
1.00
2.84
2.78
2.75
Plotname: --Not assigned--


2013-05-10-L-17-cat-2ligand-1copper-1percent.fid
Sample Name:
Data Collected on:
Varian-NMR-mercury300
Archive directory:
Sample directory:
FidFile: 2013-05-10-L-17-cat-2ligand-1copper-1percent
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 10 2013
Operator: kmn
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.706 sec
Width 4803.1 Hz
32 repetitions
OBSERVE H1, 300.1292506 MHz
DATA PROCESSING
FT size 16384
Total time 1 min 29 sec
VARIAN
8.351 8.335 8.319 7.718 7.712 7.590 7.577 7.567 7.561 7.270 7.209 7.182 7.122 7.092 6.819 6.799 6.795 6.778 6.758 6.735 6.018 6.010 5.985 5.979 5.971 5.963 5.951 5.946 5.940 5.848 5.842 5.815 5.809 4.879 4.740 4.734 4.726 4.719 4.678 4.672 4.625 4.621 4.381 4.332 4.293 4.283 3.992 3.984 3.935 3.928 1.368 1.345 1.245 1.224
8 7 6 5 4 3 2 1 ppm
2.74 2.95 1.62 2.84 2.75 3.00 2.02 3.68
1.00 2.78 1.02 3.23 5.91
Plotname: --Not assigned--

^{1}H-NMR Product mixture of Cu(**25-9**)$_2$PF$_6$ catalytic test




VARIAN
Sample Name:
Data Collected on:
Varian-NMR-mercury300
Archive directory:
Sample directory:
FidFile: 2013-05-10-L-30-cat-2ligand-1copper-1percent
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 10 2013
Operator: kmn
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.706 sec
Width 4803.1 Hz
32 repetitions
OBSERVE H1, 300.1292500 MHz
DATA PROCESSING
FT size 16384
Total time 1 min 29 sec
8.257
8.239
8.223
7.624
7.618
7.612
7.596
7.590
7.583
7.577
7.573
7.567
7.559
7.270
7.213
7.186
7.125
7.096
6.825
6.805
6.801
6.782
6.762
6.758
5.990
5.983
5.977
5.969
5.957
5.951
5.944
5.854
5.848
5.820
5.815
8.5
8.0
7.5
7.0
6.5
6.0
5.5
ppm
2.86
3.06
1.64
1.00
2.88
2.88
2.92
Plotname: --Not assigned--


VARIAN
Sample Name:
Data Collected on:
Varian-NMR-mercury300
Archive directory:
Sample directory:
FidFile: 2013-05-10-L-30-cat-2ligand-1copper-1percent
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: May 10 2013
Operator: kmn
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.706 sec
Width 4803.1 Hz
32 repetitions
OBSERVE H1, 300.1292500 MHz
DATA PROCESSING
FT size 16384
Total time 1 min 29 sec
4.752
4.746
4.738
4.730
4.723
4.717
4.682
4.631
4.625
4.385
4.334
4.314
3.996
3.988
3.980
3.939
3.931
1.372
1.351
1.249
1.228
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.05
3.32
2.13
6.07
3.48
Plotname: 00-Not assigned--