

# Engineering Orthogonal Polypeptide GalNAc-Transferase and UDP-Sugar Pairs

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bertozzi@stanford.edu

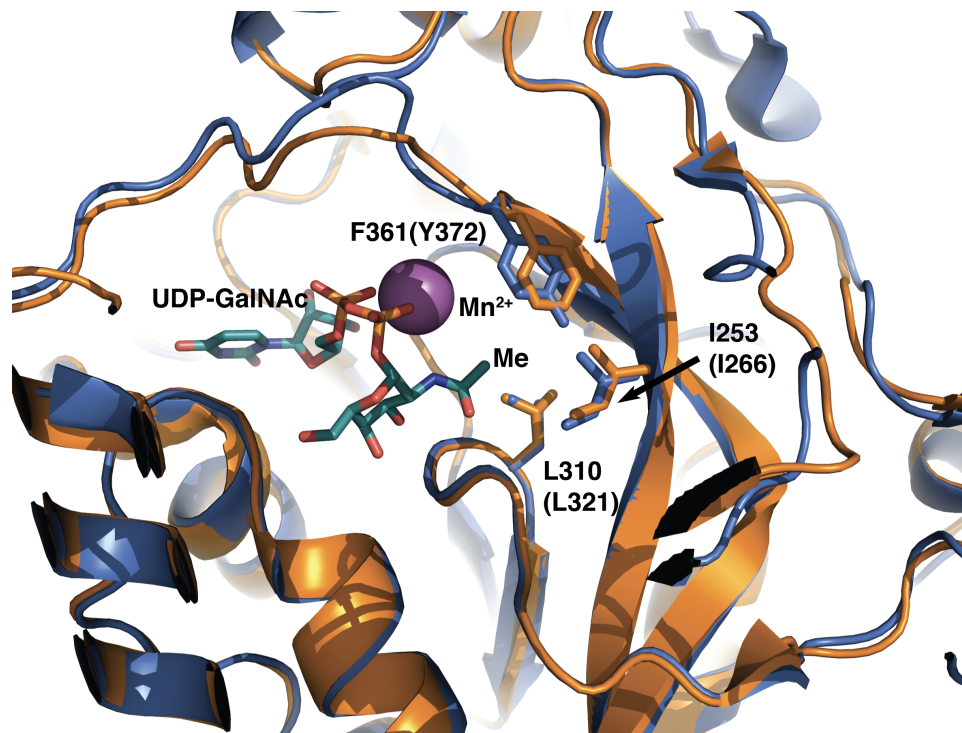
<sup>1</sup>These authors contributed equally to this work.

## Supporting Information

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## I. Supporting Figures and Tables



**Figure S1. Alignment of GalNAc-T10 and GalNAc-T2 crystal structures.** GalNAc-T10 (blue; co-crystallized with UDP and GalNAc, PDB ID: 2D7I)<sup>1</sup> aligned with GalNAc-T2 (orange; co-crystallized with UDP-GalNAc, PDB ID: 4D0T).<sup>2</sup> The three-dimensional structure of the active site of the two enzymes is closely conserved. Labeled structures include bound UDP-GalNAc (sticks), UDP-GalNAc methyl group (Me), and  $Mn^{2+}$  (purple sphere). Side chains of potential gatekeeper residues (sticks) are labeled with GalNAc-T2 residue names; GalNAc-T10 residue names are listed parenthetically. GalNAc-T2 served as the reference structure.

10 20 30 40 50 60 70 80 90 100  
hT8/1-637  
hT18/1-607  
hT9/1-603  
hT19/1-598  
hT10/1-603  
hT11/1-584  
hT7/1-657  
hT5/1-940  
hT11/1-608  
hT20/1-443  
hT15/1-639  
hT3/1-633  
hT6/1-622  
hT4/1-578  
hT12/1-581  
hT1/1-559  
hT13/1-556  
hT2/1-571  
hT14/1-552  
hT16/1-558  
MNRIRKFRGSGRVLAFIVASVIWLLFDMAALRLSFSEINTRVLIKEDIVRRERIGFRVQPDQGGKIFYSSIKEMKPPLRHGKGAWGKENVRKTEESVLKVEVDL

110 120 130 140 150 160 170 180 190 200 210  
hT8/1-637  
hT18/1-607  
hT9/1-603  
hT19/1-598  
hT10/1-603  
hT11/1-584  
hT7/1-657  
hT5/1-940  
hT11/1-608  
hT20/1-443  
hT15/1-639  
hT3/1-633  
hT6/1-622  
hT4/1-578  
hT12/1-581  
hT1/1-559  
hT13/1-556  
hT2/1-571  
hT14/1-552  
hT16/1-558  
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220 230 240 250 260 270 280 290 300 310 320  
hT8/1-637  
hT18/1-607  
hT9/1-603  
hT19/1-598  
hT10/1-603  
hT11/1-584  
hT7/1-657  
hT5/1-940  
hT11/1-608  
hT20/1-443  
hT15/1-639  
hT3/1-633  
hT6/1-622  
hT4/1-578  
hT12/1-581  
hT1/1-559  
hT13/1-556  
hT2/1-571  
hT14/1-552  
hT16/1-558  
MMF-----WRKLPKALFICLTLAIAV-----NLLVFSKGT  
MVC-----TRKTKLVSTCVILSGMNTIICLLYGVGWNTYIASVYR  
MAV-----ARKIRTLVTLNVL-----VFGIVL-----FSVYCR  
MAS-----LRRVKVLLVNLII-----AVAGFVL-----FLAKCR  
-----MRLK-----IGFILRSL-----VVGSLGLV-----  
LNVTISLSDRPRKQRSQAVANERHPASTAVPKSGEAMALNKTQSKSEVANKHKANTSLFPFKFTVNSNRLKQKISINETPLG-SLSKDDGARGAHGKLLNFSHSLVI

330 340 350 360 370 380 390 400 410 420 430  
hT8/1-637  
hT18/1-607  
hT9/1-603  
hT19/1-598  
hT10/1-603  
hT11/1-584  
hT7/1-657  
hT5/1-940  
hT11/1-608  
hT20/1-443  
hT15/1-639  
hT3/1-633  
hT6/1-622  
hT4/1-578  
hT12/1-581  
hT1/1-559  
hT13/1-556  
hT2/1-571  
hT14/1-552  
hT16/1-558  
LQNL---FTG---GLHRELPLH---LNK---RYGAVIKR---LHLEVELODL---KESMKLALRQENV---NSTLKRRAKDEVRPLLKAMÉ  
GQEP---AP---DKLEEDKG---DTLKIIE---LDHLENVIK---GCLTSATWTW---LLFYVNESFT---QPKLW---VFGK---VFKM---VFGK---  
LQGR---SQ---ELRIVSGDRRVSRAKAVGTGL---DREALQRL---LDHLEEVVY---KVLKSGSKHISRNREMSSSSLAPHVSPQSTN---HALTGGLEPAKINIFITA---KAPFS  
PIAV---RSG---DAFHEIRPRAEV---ANLSAHSASP---IQDAVLRK---LSLLEDVY---GDFVLAALVLLPNAVGLWALYRERQ---PDGTPGCSAAVAP---AAQO  
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-----VLWSSLTPRPDDPSPLSRMREDRDNDMPNRRGNGLA---PGEGRFPKVPVPH---K---K---DAMP---KMQIG---APVRON---  
ITKE-----EEQKADPKEVNSKTKTIFP-----KVLKSGSKHISRNREMSSSSLAPHVSPQSTN---HALTGGLEPAKINIFITA---KAPFS  
-----MCSVTVRYFCY-----GCLTSATWTW---LLFYVNESFT---QPKLW---VFGK---VFKM---VFGK---  
PHTLHQVTYA-QASKHSP---EA---RYR---LDGESQDWL---EA---ED---EGEYS---ED---EGEYS---  
MREVSVOY-S-KEESMERMKNKN---KML---DMLFAVNI---K---DAMP---KMQIG---APVRON---  
LHRDVSREEA-TEKPMKSLVSRKD---HVI---DMLFAMNL---R---DAMP---KMQIG---APVRON---  
-----MAVRTW---ACK---TCL---LLAFITVAYIF---VE---LLVSTF---HASAG---AGRAR---  
-----MWGR---TARRCPRELRRGR---EAL---LVLL---ALLA---GLGSVL---RAQRC---AGAC---  
-----MRRFA---YCK---VLLATSLIMVL---LDMFLLVYFSEC---NKCDL---KKEKRL---  
-----MRRSV---YCK---VLLATSLIMVL---LDMFLLVYFSEC---NKCDL---KKEKRL---  
-----MRR---RSR---VLLATSLIMVL---LDMFLLVYFSEC---NKCDL---KKEKRL---  
-----MRRLT---RRL---VLPVFGWLIT---VLLFWYTKRK---LEVPT---GPEVOT---  
-----MRKI---RAN---AIALTVAVIL---GTFYVLDQDNRRA---HAASS---GRCQAQ---

440 450 460 470 480 490 500 510 520 530 540  
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hT18/1-607  
hT9/1-603  
hT19/1-598  
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hT12/1-581  
hT1/1-559  
hT13/1-556  
hT2/1-571  
hT14/1-552  
hT16/1-558  
TKV---NETK---HKTQMKLF---PHSQLFRQWG---EDLSAQQKAA---QDLFRKFGYNAYLSQDLPNRT  
QHI---QEAPAKPEEAEAEFP---TSSLFAHWG---QELSEGRVA---LKQFOYYGNAYLSDRPLDRP  
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TEYNQSHIKALL---PDSGTHQVLR---DUTL---SPALVAPGQGRP---VVVPHCKEKA---ERWKECNFNYSVLSLIPVDRA  
GPHCPSPKFFYP---RFRGRPSVLEPQKANKI---DDVIDSRVEDPEEGHLKFSSELGMIENERDOELRDLGQKHALNMLISDRGLYHRD  
SPG---KVVHQ---QIYGEQIKPKHVIKRT---DEDAKSMGLTDFNHTNPELHK---ELLYGFNVIISRLGIERE  
-----PLEGLP---PFLSLR---EQDLLVAVALP---QARRNQSQRGGSYR---LIKOPRRQDKEAPKRDWGADEGEVESEELTPFSLDRPGLQALISARIPLQRA  
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TLFS---INQSLQ---GYTAA---ELKPV---LDRP---PQDSNAPGAGCA---FKTNLSVEEQKEV---ERGAKKCFNAFASDRISLHRD  
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-----LPA---LRAVI---SR---NQE---GPGEMGRK---VLIKDDQEMK---KELFKINQFNLMASJIALNRS  
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hT8/1-637  
hT18/1-607  
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hT2/1-571  
hT14/1-552  
hT16/1-558  
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I-PDRHANCCKHM---YLERLPNTSIIIFVNEALSIVLRSVHVSNNHTPQLLKEIILVDDFSEERHLKDKLEEMAR---F-SKVRIVRTRKREGLIRARLGASMA  
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I-EDTRPAGCAEQH---VHNNLPTTSMICFVDEWSTLLRSHVSNRSPHLLKEIILVDDFSTKDYLDKNDKYMSSQ---F-PKVRILRIRKREGLIRARLAGAQA  
V-PDTRSKMCKLQK---YPARLPTASVLCFVNEALSIVLRSVHVSNNHTPQLLKEIILVDDNSDNEELKFNLDQYVNM---RYPGLVKVIRNSREGLIRARLOGWAA  
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LCPDTRPPECIEQKFRCPPLPTTSMIVFVNEALSIVLRSVHVSNNHTPQLLKEIILVDDNSDNEELKFNLDQYVNM---RYPGLVKVIRNSREGLIRARLAGAQA  
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I-PVRWNLCKEKKYD---DNLPRTSVIAFYNEALSIVLRSVHVSNNHTPQLLKEIILVDDYSDREHLKERLANELSG---L-PKVRILRIRKREGLIRARLIGASMA  
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I-PDTRHDCQRQK---WRDPLPTASVLCFVNEALSIVLRSVHVSNNHTPQLLKEIILVDDYSDREHLKERLANELSG---L-PKVRILRIRKREGLIRARLIGASMA  
I-PDTRHDCQRQK---WRDPLPTTSMIVFVNEALSIVLRSVHVSNNHTPQLLKEIILVDDYSDREHLKERLANELSG---L-PKVRILRIRKREGLIRARLIGASMA  
I-RDTRHYSCTSVS---YSSDPLPTASVLCFVNEALSIVLRSVHVSNNHTPQLLKEIILVDDYSDREHLKERLANELSG---L-PKVRILRIRKREGLIRARLIGASMA

```

660      670      680      690      700      710      720      730      740      750      760
hT8/1-637 --TADVVAIDAHIEVNVGWAEPILARIQEDRTIVSPVFDNIRFDTKLDKYE-----LAVDGFNWLWCRYDALPQAWID--LHDVTPVKVSPIMCII--LAANRHFLG
hT18/1-607 --TAPVVALDFAHVEFNVCWAEPLTRIKENRKRILSPSFDNIKYDNFEIEEYP-----LAAQGFDWELWCRYLNPPKAWWK--LENSTAPIRSPALICG--FIVDRQYFG
hT9/1-603 --TAPVVFDAHVEFNVCWAEPLARIRKEDRITCTVPLIDVINDNTEYIIPQGGDEDCYARCAWDSMLWKRVPDLPQEKRL--RKTTEPYSFAMAGGLCAIEKEFFF
hT10/1-598 --TGQVTFDAHVEFTAGWAEPLVLRIQENRKRILPISDNIKQDNFEVQRYE-----NSAHGYSWELWCMIYSPPKDWDW--AGDPSLPKRTFAMI--GCSFVNRKFFG
hT10/1-603 --TGDVITFDLHCENVNWLPPLDRIRARNRKTIVCFMIDVIDHDDFRYEQTAG-----DAMRGAFDWEMYYKRIPIPELQ--KADPSDFESPVMAGGLFAVDRKWFV
hT17/1-584 --RGEVITFDLHCENVNWLPPLNQIALNHKTIVCFMIDVIDHNNHFEYEAQAG-----DAMRGAFDWEMYYKRIPIPELQ--RADPSDFESPVMAGGLFAVDRKWFV
hT7/1-657 --KCGVITFDLHAIECNVWYVLPVPIISKDRITICTVPLIDVINDNTEYIIPQGGDEDCYARCAWDSMLWKRVPDLPQEKRL--RKTTEPYSFAMAGGLCAIEKEFFF
hT5/1-940 --TGDVITFDLHCENVNWLPPLAAIRKEDRITCTVPLIDVINDNTEYIIPQGGDEDCYARCAWDSMLWKRVPDLPQEKRL--RKTTEPYSFAMAGGLCAIEKEFFF
hT11/1-608 --TGEVITFDLHCENVNWLPPLAAIRKEDRITCTVPLIDVINDNTEYIIPQGGDEDCYARCAWDSMLWKRVPDLPQEKRL--RKTTEPYSFAMAGGLCAIEKEFFF
hT20/1-443 --SGDVLVFDLHCENVNWLPPLAAIKADPKMVCPLIDVINDNTEYIIPQGGDEDCYARCAWDSMLWKRVPDLPQEKRL--RKTTEPYSFAMAGGLCAIEKEFFF
hT5/1-639 --TGDVITFDLHCENVNWLPPLVPIISKDRITICTVPLIDVINDNTEYIIPQGGDEDCYARCAWDSMLWKRVPDLPQEKRL--RKTTEPYSFAMAGGLCAIEKEFFF
hT3/1-633 --TAETLITFDLHCCECFWGLEPLARIAENYAVVSPDIASIDLNTFEFNKPSPY--GSHNRGNFDWLSFGWESLPDHEKOR--RKTTEPYSFAMAGGLCAIEKEFFF
hT6/1-622 --OAEVITFDLHCCECFWGLEPLARIAEDKTVVSPDVIDLNTFEFNKPSPY--GRVHNRGNFDWLSFGWESLPDHEKOR--RKTTEPYSFAMAGGLCAIEKEFFF
hT4/1-578 --TGDVITFDLHCCECFWGLEPLARIRKEDRITCTVPLIDVINDNTEYIIPQGGDEDCYARCAWDSMLWKRVPDLPQEKRL--RKTTEPYSFAMAGGLCAIEKEFFF
hT2/1-581 --RCDVITFDLHCCECFWGLEPLARIRKEDRITCTVPLIDVINDNTEYIIPQGGDEDCYARCAWDSMLWKRVPDLPQEKRL--RKTTEPYSFAMAGGLCAIEKEFFF
hT1/1-559 --KGVITFDLHCCECFWGLEPLARIRKEDRITCTVPLIDVINDNTEYIIPQGGDEDCYARCAWDSMLWKRVPDLPQEKRL--RKTTEPYSFAMAGGLCAIEKEFFF
hT13/1-556 --KGVITFDLHCCECFWGLEPLARIRKEDRITCTVPLIDVINDNTEYIIPQGGDEDCYARCAWDSMLWKRVPDLPQEKRL--RKTTEPYSFAMAGGLCAIEKEFFF
hT2/1-571 --OAKVITFDLHCCECFWGLEPLARIRKEDRITCTVPLIDVINDNTEYIIPQGGDEDCYARCAWDSMLWKRVPDLPQEKRL--RKTTEPYSFAMAGGLCAIEKEFFF
hT4/1-552 --QCTTITFDLHCCECFWGLEPLARIRKEDRITCTVPLIDVINDNTEYIIPQGGDEDCYARCAWDSMLWKRVPDLPQEKRL--RKTTEPYSFAMAGGLCAIEKEFFF
hT16/1-558 --AATVITFDLHCCECFWGLEPLARIRKEDRITCTVPLIDVINDNTEYIIPQGGDEDCYARCAWDSMLWKRVPDLPQEKRL--RKTTEPYSFAMAGGLCAIEKEFFF
770      780      790      800      810      820      830      840      850      860
hT8/1-637 EIGSLDGMGLIYGCENVELSRWQCGGCKVEIPECSRVAIAHLEHKKPYALD-----LALKRNALRVAAIWMDEKHMVYLAWNIP--LQSGIDGDFDVSRRMLREKL
hT18/1-607 EIGLLDGMCEVYGCENVELSRWQCGGCSVLEPCSRVAIAHLEHKKPYALD-----LAAHRRNARLRAEVMWDEKSHVYMAWNIQ--DQSDIGIDI--TARKLRRKL
hT9/1-603 DIGLLDGMCEVYGCENVELSRWQCGGSMELVPCSRVAIAHLEHKKPYALD-----LAAHRRNARLRAEVMWDEKSHVYMAWNIQ--DQSDIGIDI--TARKLRRKL
hT10/1-598 EIGLLDGMCEVYGCENVELSRWQCGGSMELVPCSRVAIAHLEHKKPYALD-----LAAHRRNARLRAEVMWDEKSHVYMAWNIQ--DQSDIGIDI--TARKLRRKL
hT10/1-603 EIGLLDGMCEVYGCENVELSRWQCGGSMELVPCSRVAIAHLEHKKPYALD-----LAAHRRNARLRAEVMWDEKSHVYMAWNIQ--DQSDIGIDI--TARKLRRKL
hT17/1-584 ELGGYDGLCEIWMGCEQYEIFSRVWMCQGMEDIPCSRVGHIYRKRKPYKVP-----AG--YSLARNLRVAEVMWDEKSHVYMAWNIQ--DQSDIGIDI--TARKLRRKL
hT7/1-657 ELGLYDPSLQIWMGCEQYEIFSRVWMCQGMEDIPCSRVGHIYRKRKPYKVP-----AG--YSLARNLRVAEVMWDEKSHVYMAWNIQ--DQSDIGIDI--TARKLRRKL
hT5/1-940 ELGYDTPGLDWMGCEQYEIFSRVWMCQGMEDIPCSRVGHIYRKRKPYKVP-----AG--YSLARNLRVAEVMWDEKSHVYMAWNIQ--DQSDIGIDI--TARKLRRKL
hT11/1-608 ELGYDTPGLDWMGCEQYEIFSRVWMCQGMEDIPCSRVGHIYRKRKPYKVP-----AG--YSLARNLRVAEVMWDEKSHVYMAWNIQ--DQSDIGIDI--TARKLRRKL
hT20/1-443 EIGQYDQMDVWGRLENLLESLRWMCGGLDIPCSRVGHIYRKRKPYKVP-----AG--YSLARNLRVAEVMWDEKSHVYMAWNIQ--DQSDIGIDI--TARKLRRKL
hT11/1-639 NTGAYDSLMSLGGENLELSPKAWLCCGSVEILPCSRVGHIYRKRKPYKVP-----AG--YSLARNLRVAEVMWDEKSHVYMAWNIQ--DQSDIGIDI--TARKLRRKL
hT3/1-633 EIGYDTPGLDWMGCEQYEIFSRVWMCQGMEDIPCSRVGHIYRKRKPYKVP-----AG--YSLARNLRVAEVMWDEKSHVYMAWNIQ--DQSDIGIDI--TARKLRRKL
hT6/1-622 ELGYDTPGLDWMGCEQYEIFSRVWMCQGMEDIPCSRVGHIYRKRKPYKVP-----AG--YSLARNLRVAEVMWDEKSHVYMAWNIQ--DQSDIGIDI--TARKLRRKL
hT4/1-578 YLGYDTPGLDWMGCEQYEIFSRVWMCQGMEDIPCSRVGHIYRKRKPYKVP-----AG--YSLARNLRVAEVMWDEKSHVYMAWNIQ--DQSDIGIDI--TARKLRRKL
hT12/1-581 ELGYDTPGLDWMGCEQYEIFSRVWMCQGMEDIPCSRVGHIYRKRKPYKVP-----AG--YSLARNLRVAEVMWDEKSHVYMAWNIQ--DQSDIGIDI--TARKLRRKL
hT1/1-559 ELGYDTPGLDWMGCEQYEIFSRVWMCQGMEDIPCSRVGHIYRKRKPYKVP-----AG--YSLARNLRVAEVMWDEKSHVYMAWNIQ--DQSDIGIDI--TARKLRRKL
hT2/1-571 ELGYDTPGLDWMGCEQYEIFSRVWMCQGMEDIPCSRVGHIYRKRKPYKVP-----AG--YSLARNLRVAEVMWDEKSHVYMAWNIQ--DQSDIGIDI--TARKLRRKL
hT14/1-552 YLGYDTPGLDWMGCEQYEIFSRVWMCQGMEDIPCSRVGHIYRKRKPYKVP-----AG--YSLARNLRVAEVMWDEKSHVYMAWNIQ--DQSDIGIDI--TARKLRRKL
hT16/1-558 HLGKYDQMDVWGRLENLLESLRWMCGGLDIPCSRVGHIYRKRKPYKVP-----AG--YSLARNLRVAEVMWDEKSHVYMAWNIQ--DQSDIGIDI--TARKLRRKL
880      890      900      910      920      930      940      950      960      970      980
hT8/1-637 KCKTFDHWLKNVYPLKPHLTI-----VYGRMKNLIDENVCLDQGF-----VPGNTPIMYCHEF-----SSQNVYHLLTCELYVGLQIAEAS-
hT18/1-607 KCKTFDHWLKNVYPLKPHLTI-----VYGRMKNLIDENVCLDQGF-----VPGNTPIMYCHEF-----SSQNVYHLLTCELYVGLQIAEAS-
hT9/1-603 KCKTFDHWLKNVYPLKPHLTI-----VYGRMKNLIDENVCLDQGF-----VPGNTPIMYCHEF-----SSQNVYHLLTCELYVGLQIAEAS-
hT10/1-598 KCKTFDHWLKNVYPLKPHLTI-----VYGRMKNLIDENVCLDQGF-----VPGNTPIMYCHEF-----SSQNVYHLLTCELYVGLQIAEAS-
hT10/1-603 KCKTFDHWLKNVYPLKPHLTI-----VYGRMKNLIDENVCLDQGF-----VPGNTPIMYCHEF-----SSQNVYHLLTCELYVGLQIAEAS-
hT17/1-584 KCKDFQMAAIVADVDPKYKPP-----VEPPAAWGEIRNVAALNCLVSKH-----GATGTELRLDICVKDGSERTWSHGLFTGWRDIRPGEPLHT-
hT7/1-657 NCQSFKWFMEIAYDITSHYLP-----PKNDVWGEIRGFEIAYCISDMG-----K--TNGGFVLELPCCHRM-----GNGLFRINEANQLMW-
hT5/1-940 KCKSFKWLKNVYPLKPHLTI-----VYGRMKNLIDENVCLDQGF-----VPGNTPIMYCHEF-----SSQNVYHLLTCELYVGLQIAEAS-
hT11/1-608 KCKSFKWLKNVYPLKPHLTI-----VYGRMKNLIDENVCLDQGF-----VPGNTPIMYCHEF-----SSQNVYHLLTCELYVGLQIAEAS-
hT20/1-443 KCKSFKWLKNVYPLKPHLTI-----VYGRMKNLIDENVCLDQGF-----VPGNTPIMYCHEF-----SSQNVYHLLTCELYVGLQIAEAS-
hT11/1-639 KCKTFDHWLKNVYPLKPHLTI-----VYGRMKNLIDENVCLDQGF-----VPGNTPIMYCHEF-----SSQNVYHLLTCELYVGLQIAEAS-
hT3/1-633 KCKTFDHWLKNVYPLKPHLTI-----VYGRMKNLIDENVCLDQGF-----VPGNTPIMYCHEF-----SSQNVYHLLTCELYVGLQIAEAS-
hT6/1-622 KCKSFKWLKNVYPLKPHLTI-----VYGRMKNLIDENVCLDQGF-----VPGNTPIMYCHEF-----SSQNVYHLLTCELYVGLQIAEAS-
hT4/1-578 KCKSFKWLKNVYPLKPHLTI-----VYGRMKNLIDENVCLDQGF-----VPGNTPIMYCHEF-----SSQNVYHLLTCELYVGLQIAEAS-
hT12/1-581 KCKDFQMAAIVADVDPKYKPP-----VEPPAAWGEIRNVAALNCLVSKH-----GATGTELRLDICVKDGSERTWSHGLFTGWRDIRPGEPLHT-
hT1/1-559 KCKDFQMAAIVADVDPKYKPP-----VEPPAAWGEIRNVAALNCLVSKH-----GATGTELRLDICVKDGSERTWSHGLFTGWRDIRPGEPLHT-
hT2/1-571 KCKDFQMAAIVADVDPKYKPP-----VEPPAAWGEIRNVAALNCLVSKH-----GATGTELRLDICVKDGSERTWSHGLFTGWRDIRPGEPLHT-
hT14/1-552 KCKDFQMAAIVADVDPKYKPP-----VEPPAAWGEIRNVAALNCLVSKH-----GATGTELRLDICVKDGSERTWSHGLFTGWRDIRPGEPLHT-
hT16/1-558 KCKDFQMAAIVADVDPKYKPP-----VEPPAAWGEIRNVAALNCLVSKH-----GATGTELRLDICVKDGSERTWSHGLFTGWRDIRPGEPLHT-
990      1000     1010     1020     1030     1040     1050     1060     1070     1080     1090
hT8/1-637 ASDRCLTDP-----GKAKEPTLEPCSKAA--KNRLHIYWFDFKGP--GAVIN-----RDKRCLLQEMK--KDLL--GSHVLVLTQCS-----TQVWEIQTWRDQGT
hT18/1-607 DDNRCLVDP-----GKAKEPTLEPCSKAA--KNRLHIYWFDFKGP--GAVIN-----RDKRCLLQEMK--KDLL--GSHVLVLTQCS-----TQVWEIQTWRDQGT
hT9/1-603 PDSKCLVDP-----GTRMPTLKRCEVDA--RPTQRWDFDQSG--GPIQN-----RATGRCLLEVMKSDAN--FGLRLVQRCS-----GQKWIIRNWIKHARRH-
hT10/1-598 PDSKCLVDP-----GTRMPTLKRCEVDA--RPTQRWDFDQSG--GPIQN-----RATGRCLLEVMKSDAN--FGLRLVQRCS-----GQKWIIRNWIKHARRH-
hT10/1-603 PDSKCLVDP-----GTRMPTLKRCEVDA--RPTQRWDFDQSG--GPIQN-----RATGRCLLEVMKSDAN--FGLRLVQRCS-----GQKWIIRNWIKHARRH-
hT17/1-584 --KRCFDIAI-----SHTSPVTLYDCH-----SMKGNLWLYRKKD--RTLFH-----PVSGSCMDCSESD-----HRIFMNTCNPSSLTQQLVFEHNTSTVLEK
hT7/1-657 --KRCFDIAI-----SHTSPVTLYDCH-----SMKGNLWLYRKKD--RTLFH-----PVSGSCMDCSESD-----HRIFMNTCNPSSLTQQLVFEHNTSTVLEK
hT5/1-940 --DOCLTSG-----ADGSKVMTIHCN-----LNEFKWQYIKNLRHTY-----IPSGKCLDRSEVL-----HQVFINSDCSKTKQKWEHNIHVS-----
hT11/1-608 --LLCLDMSSET--RSSDPRMLMKCH-----GSGSQWTFGKN--RFLYQ-----VSVGQLRAVDP--LQSK--GVSAMAI--CD--GSSSQWHLV-----
hT20/1-443 --HLCFAVR-----OEVILQNCITEG--LAIHQHWFDFQEN--GMIVH-----ILSGKMEAVVQENNK-----DLYLRPCD--GKARQQRVFDQINAVDER--
hT11/1-639 --QLCLVDS-----KALGLGSCHEFTGKNSQPKDEWELEAQQ--QLIRN-----SGSGTCLTSQDK-----KPMAMP--SN--SOPHQLWLFV-----
hT3/1-633 --ELCAEVP-----EQNYVGMGNCPDGFPPVANI--IWHFKED--GTIFH-----PHSGCLLSAYRTP--EGR--PDVQM--RT--CDALDKN--IWSF--
hT6/1-622 --PCCGLVDS-----RNGPVI--MLKCH-----HRLGNLWYDAERLTLR-----VNSNQLCDEP--EEDKM-----VPTMQDCS--PAL--SQWKF--TL--NLQ--
hT4/1-578 --DCLCLVDS-----VDRAPGAPV--LWLCCK-----NDGRQWTKT--GSHI--EH-----IASHLCLD--DMF--GDCG--ENK--EIV--VNP--CESS--LMS--SQW--
hT12/1-581 --DCLCLVDS-----VDRAPGAPV--LWLCCK-----NDGRQWTKT--GSHI--EH-----IASHLCLD--DMF--GDCG--ENK--EIV--VNP--CESS--LMS--SQW--
hT1/1-559 --DCLCLVDS-----VDRAPGAPV--LWLCCK-----NDGRQWTKT--GSHI--EH-----IASHLCLD--DMF--GDCG--ENK--EIV--VNP--CESS--LMS--SQW--
hT2/1-571 --DCLCLVDS-----VDRAPGAPV--LWLCCK-----NDGRQWTKT--GSHI--EH-----IASHLCLD--DMF--GDCG--ENK--EIV--VNP--CESS--LMS--SQW--
hT14/1-552 --DCLCLVDS-----VDRAPGAPV--LWLCCK-----NDGRQWTKT--GSHI--EH-----IASHLCLD--DMF--GDCG--ENK--EIV--VNP--CESS--LMS--SQW--
hT16/1-558 --DCLCLVDS-----VDRAPGAPV--LWLCCK-----NDGRQWTKT--GSHI--EH-----IASHLCLD--DMF--GDCG--ENK--EIV--VNP--CESS--LMS--SQW--
1100
hT8/1-637 NSQ-----
hT18/1-607 -----
hT9/1-603 -----
hT10/1-598 FNRN-----
hT10/1-603 FNRN-----
hT17/1-584 FNHHANS-----
hT7/1-657 -----
hT5/1-940 -----
hT11/1-608 -----
hT20/1-443 -----
hT11/1-639 -----
hT3/1-633 -----
hT6/1-622 -----
hT4/1-578 -----
hT12/1-581 -----
hT1/1-559 -----
hT13/1-556 -----
hT2/1-571 -----
hT14/1-552 -----
hT16/1-558 -----

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**Figure S2. Alignment of human GalNAc-T genes.** Amino acid sequences corresponding to full-length human GALNT1–GALNT20 (Table S1) were aligned using Clustal Omega.<sup>3</sup> GalNAc-Ts are labeled as follows: hT8(human GalNAc-T8)-1-637 (sequence contains 637 amino acids).



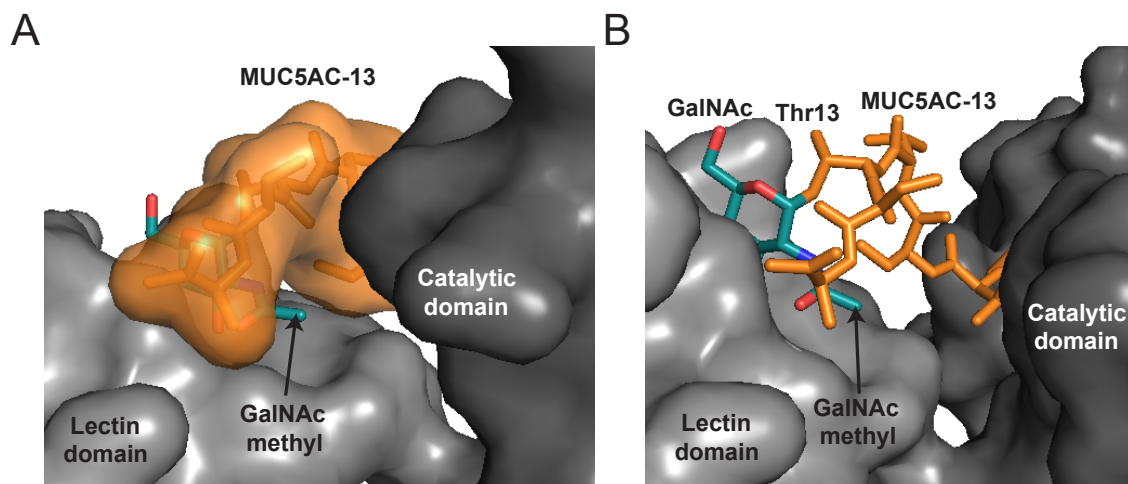
**Table S1. GalNAc-T gene names and accession numbers.**

<b>Human gene name</b>	<b>Accession number<sup>a</sup></b>
GALNT1	X85018
GALNT2	X85019
GALNT3	X92689
GALNT4	Y08564
GALNT5	NM_014568 <sup>b</sup>
GALNT6	Y08565
GALNT7	AJ002744
GALNT8	AJ271385
GALNT9	AB040672
GALNT10	AJ505950
GALNT11	Y12434
GALNT12	AJ132365
GALNT13	AJ505991
GALNT14	Y09324
GALNT15	NM_054110
GALNT16	AJ505951
GALNT17	AJ626725
GALNT18	AJ626724
GALNT19	AJ626726
GALNT20	145292

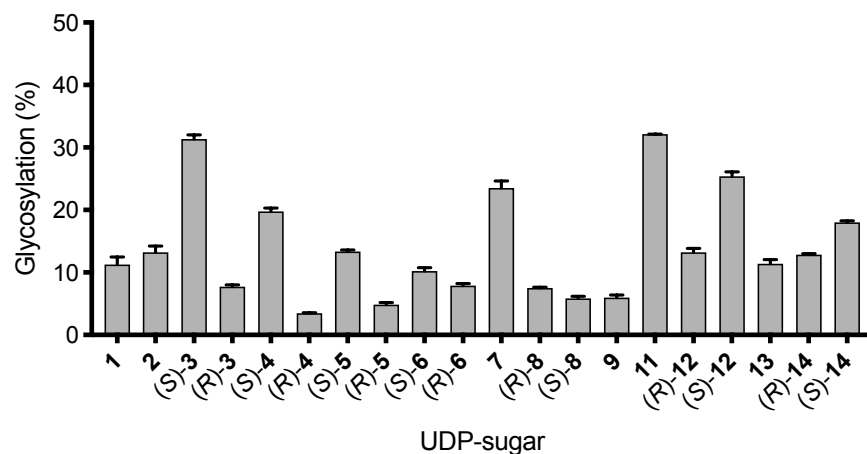
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(a) GalNAc-T amino acid sequence and accession information is summarized in Bennett et al.<sup>4</sup>

(b) Reported in Guo et al.<sup>5</sup>

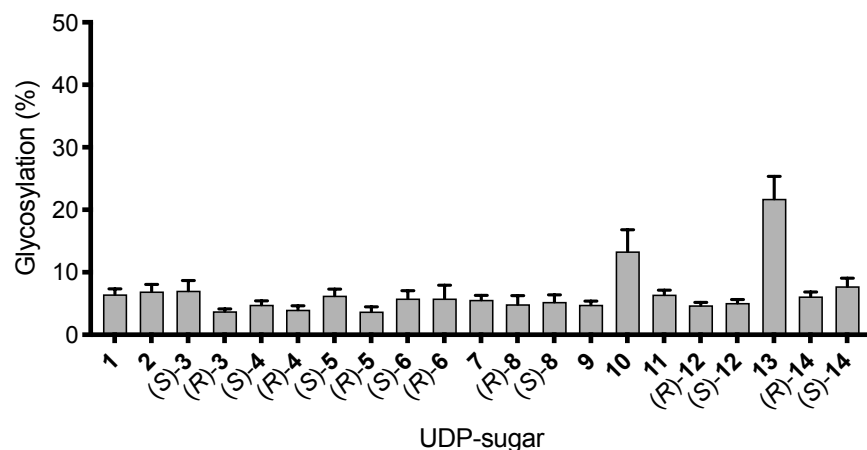


**Figure S3. GalNAc-T2 with a GalNAc-peptide, MUC5AC-13, bound to the lectin domain.** GalNAc-T2 (gray; active confirmation of the enzyme, co-crystallized with UDP and MUC5AC-13, PDB ID: 5AJP). (A) GalNAc (sticks) is attached to MUC5AC-13 (orange sticks and surface) at Thr13. The GalNAc methyl group (teal stick) extends out of the pocket formed by MUC5AC-13 and the lectin domain (light gray surface) into a solvent-exposed cleft between the acceptor peptide and the lectin and catalytic domains (dark gray surface). (B) View of the solvent exposed cleft into which GalNAc methyl extends. GalNAc (sticks) bound to Thr13 of MUC5AC-13 (orange sticks) is shown.



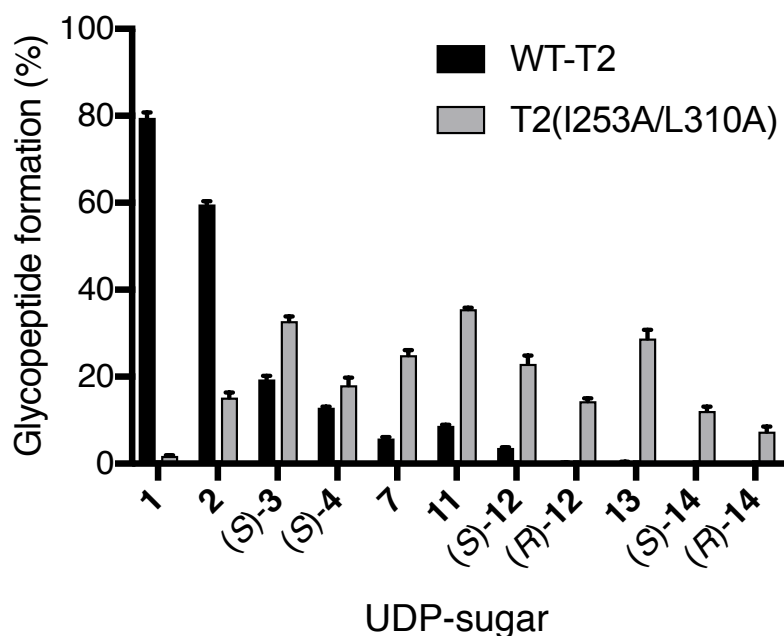
**Figure S4. T2(I253A/L310A) activity assay with UDP-GalNAc analogs.**

Glycosylation reaction by 25 nM T2(I253A/L310A) with 250  $\mu$ M UDP-GalNAc analog and 50.0  $\mu$ M peptide (GAGAPGPTPGPGAG) at 37  $^{\circ}$ C for 1 h. Enzymatic activity was measured using the Glycosyltransferase Activity Kit (R&D Systems, Inc.). All data represent the mean of technical triplicates, and the error bars represent the standard deviation.



**Figure S5. T1(I238A/L295A) activity assay with UDP-GalNAc analogs.**

Glycosylation reaction by 25 nM T1(I238A/L295A) with 500  $\mu$ M UDP-GalNAc analog and 50.0  $\mu$ M peptide (GAGAFFPTPGPAGAGK) at 37  $^{\circ}$ C for 1 h. Enzymatic activity was measured using the Glycosyltransferase Activity Kit (R&D Systems, Inc.). All data represent the mean of technical triplicates, and the error bars represent the standard deviation.



**Figure S6. Screening GalNAc-T2 for an orthogonal enzyme-substrate pair.** Glycopeptide formation by wild-type and double mutant GalNAc-T2. Peptide-1 and UDP-GalNAc or UDP-GalNAc analogs were incubated with GalNAc-T2 at 37 °C for 1 h, and the reaction was quenched with aqueous EDTA (150 mM, pH = 8.0). The percent conversion to glycopeptide product was quantified by HPLC separation and peak integration. These data are the same as those shown in Figure 3C. All data represent the mean of technical triplicates, and the error bars represent the standard deviation.



**Table S2. Synthetic gBlocks used for GalNAc-T1 and GalNAc-T10 gene assembly.**

T1_GB1 <sup>c</sup>	GACAAGCTT <u>GCGGCCGCGGATGAAAAAAGGAGAGAGGACTTCC</u> TGCTGGAGATGTTCTAGAGCCAGTACAAAAGCCTCATGAAGGTCC TGGAGAAATGGGGAAACCAGTCGTCATTCCTAAAGAGGATCAAGA AAAGATGAAAGAGATGTTTAAAATCAATCAGTTCAATTTAATGGC AAGTGAGATGATTGCACTCAACAGATCTTTACCAGATGTTAGGTTA GAAGGGTGTAAAACAAAGGTGTATCCAGATAATCTTCCTACAACA AGTGTGGTGATTGTTTTCCACAATGAGGCTTGGAGCACACTTCTGC GAACTGTCCATAGTGTCAATTAATCGCTCACCAAGACACATGATAG AAGAAATTGTT <u>GAGACCTGGTGTG</u>
T1_GB2	GACAGGAGGTCTCATTGTTCTAGTAGATGATGCCAGTGAAAGAGA CTTTTTGAAAAGGCCTTTAGAGAGTTATGTGAAAAAACTAAAAGT ACCAGTTCATGTAATTCGAATGGAACAACGTTCTGGATTGATCAG AGCTAGATTAAGAGGAGCTGCTGTGTCTAAAGGCCAAGTGATCAC CTTCCTGGATGCCATTGTGAGTGTACAGTGGGATGGCTGGAGCCT CTCTTGGCCAGGATCAAACATGACAGGAGAACAGTGGTGTGTCCC ATCATCGATGTGATCAGTGATGATACTTTTGAGTACATGGCAGGCT CTGATATGACCTATGGTGGGTTCAACTGGAAGCTCAATTTTCGCTG GTATCCTGTTCCCAAAGAGAAAATGGACAGAAGGAAAGGTGATCG GACTCTCCTGTCAGGACACCTACCATGGCAGGAGGCCTTTTTTCA ATAGACAGAGATTACTTTTT <u>GAGACCTGGTGTG</u>
T1_GB3	GACAGGAGGTCTCACTTTCAGGAAATTGGAACATATGATGCTGGA ATGGATATTTGGGGAGGAGAAAACCTAGAAAATTTCTTTAGGATT TGGCAGTGTGGAGGAACCTTTGGAAATTGTTACATGCTCACATGTTG GACATGTGTTTCGGAAAGCTACACCTTACACGTTTCCAGGAGGCA CAGGGCAGATTATCAATAAAAAATAACAGACGACTTGCAGAAGTGT GGATGGATGAATTCAGAATTTCTTCTATATAATTTCTCCAGGTGT TACAAAGGTAGATTATGGAGATATATCGTCAAGAGTTGGTCTAAG ACACAACTACAATGCAAACCTTTTTCTGTTACCTAGAGAATATA TATCCTGATTCTCAAATTCACGTCACTATTTCTCATTGGGAGAGA TACGAAATGTGGAAACGAATCAGTGTCTAGATAACATGGCTAGAA AAGAGAATGAAAAGTTGGA <u>TGAGACCTGGTGTG</u>
T1_GB4 <sup>d</sup>	GACAGGAGGTCTCATGGAATTTTAAATTGCCATGGTATGGG <b>TGGT</b> AATCAGGTTTTCTTTATACTGCCAACAAGAAATTAGAACAGAT GACCTTTGCTTGGATGTTTCCAACTTAATGGCCAGTTACAATGC TCAAATGCCACCACCTAAAAGGCAACCACTCTGGGAGTATGACC CAGTGAATTAACCCTGCAGCATGTGAACAGTAATCAGTGCCTGG ATAAAGCCACAGAAGAGGATAGCCAGGTGCCAGCATTAGAGACT GCAATGGAAGTCGGTCCCAGCAGTGGCTTCTTCGAAACGTCACCC TTCCAGAAATATTCTGAGA ATTCATCGATAG

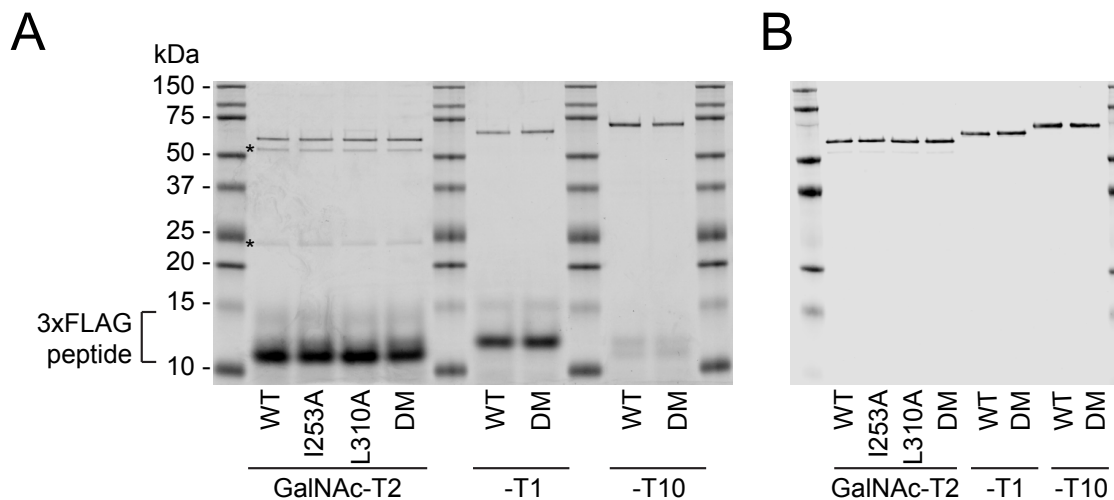
(c) BsaI restriction sites are underlined in each gBlock.

(d) Silent mutations in any gBlock are underlined and bold.

T10_GB1 <sup>e</sup>	GACAGGAGGTCTCAGGCCGCGCCTGGGGGATCGGGGGCGGCGGT GGCGCCGGCGGGGACAGGGCTCACACAGTCGACAAAAGAAAA CGTTTTTCTTGGGAGATGGGCAGAAGCTGAAGGACTGGCATGACA AGGAGGCCATCCGGAGGGACGCTCAGCGCGTAGGAAATGGAGAA CAAGGAAGACCTTACCCCATGACCGATGCTGAGAGAGTGGATCAG GCATACCGAGAAAATGGATTTAACATCTACGTCAGTGATAAAATC TCCTTGAATCGCTCTCTCCAGATATCCGGCACCCAACTGCAACA GCAAGCGCTACCTGGAGACACTTCCCAACACAAGCATCATCATCC CCTTCCACAACGAGGGCTGGTCCTCCCTCCTCCGCACCGTCCACAG TGTGCTCAATCGCTCGCCTCCAGAGCTGGTCGCCGAGATTGTACTG GTCGACGACTTCAGTGATCGAGAGCACCTGAAGAAGCCTCTTGAA GACTACATGGCCCTTTT <u>GAGACCT</u> GGTGTG
T10_GB2_ WT	GACAGGAGGTCTCACTTTTCCCCAGTGTGAGGATTCTTCGAACCAA GAAACGGGAAGGGCTGATAAGGACCCGAATGCTGGGGCCTCAG TGGCAACTGGGGATGTCATCACATTCTTGGATTCACACTGTGAAGC CAATGTCAACTGGCTTCCCCCTTGCTTGACCGCATTGCTCGGAAC CGCAAGACCATTGTGTGCCCGATGAATGATGTAATTGACCATGAC GACTTTCGGTACGAGACACAGGCAGGGGATGCCATGCGGGGAGCC TTTACTGGGAGATGTACTACAAGCGGATCCCGATCCCTCCAGAA CTGCAGAAAGCTGACCCAGCGACCCATTTGAGTCTCCCGTGATG GCCGGTGGACTGTTCCCGTGGATCGGAAGTGGTTCTGGGAACTC GGCGGGTATGACCCAGGCTTGGAGATCTGGGGAGGGGAGCAGTAT GAAATCTCCTTCAAGGTGTGGATGTGTGGGGGCCGCATGGAGGAC ATCCCCTGCTCCAGGGTGGGCCATATCTACAGGAAGTATGTGCCCT ACAAGGTCCCGCCGGAGTCAGCCTGGCCCGGAACCTTAAGCGGG TGGCCGAAGTGTGGATGGATGAGTACGCAGAGTT <u>GAGACCT</u> GGTG TG
T10_GB2_ MUT	GACAGGAGGTCTCACTTTTCCCCAGTGTGAGGATTCTTCGAACCAA GAAACGGGAAGGGCTGATAAGGACCCGAATGCTGGGGCCTCAG TGGCAACTGGGGATGTCATCACATTCTTGGATTCACACTGTGAAGC CAATGTCAACTGGCTTCCCCCTTGCTTGACCGCATTGCTCGGAAC CGCAAGACCATTGTGTGCCCGATGGCCGATGTAATTGACCATGAC GACTTTCGGTACGAGACACAGGCAGGGGATGCCATGCGGGGAGCC TTTACTGGGAGATGTACTACAAGCGGATCCCGATCCCTCCAGAA CTGCAGAAAGCTGACCCAGCGACCCATTTGAGTCTCCCGTGATG GCCGGTGGAGCCTTCCCGTGGATCGGAAGTGGTTCTGGGAACTC GGCGGGTATGACCCAGGCTTGGAGATCTGGGGAGGGGAGCAGTAT GAAATCTCCTTCAAGGTGTGGATGTGTGGGGGCCGCATGGAGGAC ATCCCCTGCTCCAGGGTGGGCCATATCTACAGGAAGTATGTGCCCT ACAAGGTCCCGCCGGAGTCAGCCTGGCCCGGAACCTTAAGCGGG TGGCCGAAGTGTGGATGGATGAGTACGCAGAGTT <u>GAGACCT</u> GGTG TG

(e) GalNAc-T10 was assembled with T10\_GB1, T10\_GB2\_WT, and T10\_GB3 (wild-type) or T10\_GB1, T10\_GB2\_MUT, and T10\_GB3 (double mutant).

T10_GB3	GACAGGAGGTCTCAGAGTACATTTACCAGCGCCGGCCTGAATACC GCCACCTCTCCGCTGGGGATGTCGCAGTCCAGAAAAAGCTCCGCA GCTCCCTTAACTGCAAGAGTTTCAAGTGGTTTATGACGAAGATAGC CTGGGACCTGCCCAAATTCTACCCACCCGTGGAGCCCCCGGCTGC AGCTTGGGGGGAGATCCGAAATGTGGGCACAGGGCTGTGTGCAGA CACAAAGCACGGGGCCTTGGGCTCCCCACTAAGGCTAGAGGGCTG CGTCCGAGGCCGTGGGGAGGCTGCCTGGAACAACATGCAGGTATT CACCTTCACCTGGAGAGAGGACATCCGGCCTGGGAGACCCCCAGCA CACCAAGAAGTTCTGCTTTGATGCCATTTCCACACCAGCCCTGTC ACGCTGTACGACTGCCACAGCATGAAGGGCAACCAGCTGTGGAAA TACCGCAAAGACAAGACCCTGTACCACCCTGTCAGTGGCAGCTGC ATGGACTGCAGTGAAAGTGACCATAGGATCTTCATGAACACCTGC AACCCATCCTCTCTCACCCAGCAGTGGCTGTTTGAACACACCAACT CAACAGTCTTGGAAAAATTCAATAGGAACTGAG AATTTGAGACCTGGTGTG
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**Figure S7. Purified soluble GalNAc-Ts.** (A) SDS-PAGE of GalNAc-T2 (predicted MW of the wild-type enzyme 61.6 kDa), GalNAc-T1 (63.1 kDa) and GalNAc-T10 (65.9 kDa) preparations (Coomassie stain). GalNAc-Ts are labeled wild-type (WT), single mutant (I253A, L310A), or double mutant (DM). Asterisks depict potential degradation products. (B) Western blot with anti-FLAG<sup>®</sup> immunolabeling.

## II. General Information

MUC5AC-3 (GTT\*PSPVPTTSTTSAP), MUC5AC-13 (GTTSPSPVPTTSTT\*SAP), EA2 (PTTDSTTPAPTTK), and a peptide optimized for GalNAc-T2 (GAGAPGPTPGPGAG) were purchased from AnaSpec, Inc. and used without further purification. A peptide optimized for GalNAc-T1 (GAGAFFPTPGPAGAGK) was synthesized on 2-chlorotrityl chloride resin by solid phase peptide synthesis using *N*-Fmoc-protected amino acids. Peptide concentrations were determined by amino acid analysis at the UC Davis Molecular Structure Facility. UDP-sugar concentrations were quantified using the molar extinction coefficient of UDP at 262 nm ( $10,000 \text{ M}^{-1}\text{cm}^{-1}$ ).

MacPyMOL was used to model all crystal structures. Integrated DNA Technologies (IDT) synthesized all gBlocks. Primer synthesis and sequencing of all plasmids prior to use was by Elim Biopharmaceuticals, Inc. (Hayward, USA). Restriction enzymes, Antarctic phosphatase, and T4 DNA ligase were purchased from New England Biolabs and used according to the manufacturer's instructions. PfuUltra II Fusion HS DNA polymerase was from Agilent. cComplete™ mini EDTA-free protease inhibitor, p3xFLAG-CMV-8, pFLAG-myc-CMV-19, recombinant human serum albumin, anti-FLAG® M2 agarose resin, and monoclonal anti-FLAG® M2 antibody were purchased from Sigma-Aldrich (now Millipore-Sigma). Colloidal Blue Staining Kit was obtained from Thermo Fisher Scientific.

Dulbecco's Modified Eagle Medium with high glucose (DMEM), Dulbecco's Phosphate-Buffered Saline without calcium or magnesium (DPBS), and Penicillin/Streptomycin were purchased from Hyclone. Fetal bovine serum (FBS) was obtained from Omega Scientific, and 0.25% trypsin/EDTA and Opti-MEM® Reduced Serum Medium were purchased from Invitrogen. TransIT®-293 transfection reagent was purchased from Mirus Bio LLC.

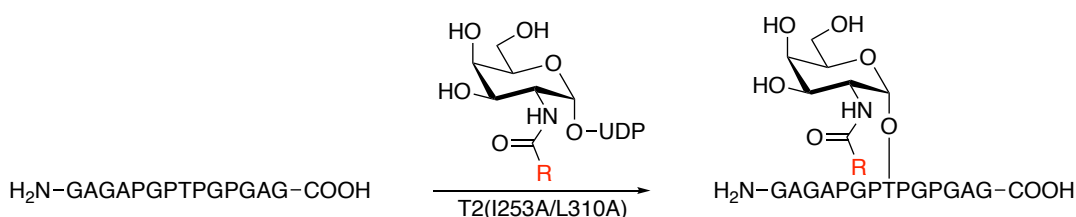
Chemical reagents were obtained from commercial sources and used without further purification unless otherwise noted. Unless stated otherwise, reactions were conducted under an atmosphere of nitrogen using anhydrous solvents. THF and  $\text{CH}_2\text{Cl}_2$  were deoxygenated and dried by sparging with nitrogen followed by passage through an activated alumina column prior to use. Deionized water ( $18.2 \text{ M}\Omega\cdot\text{cm}$ ) was prepared by a Millipore Milli-Q Biocel A10 purification unit and used to prepare all buffers and aqueous solutions.

Glycosyltransferase Activity Kit was purchased from R&D Systems, Inc., and absorbance at 620 nm was acquired using a Molecular Devices SpectraMax M3 96-well plate reader. High-performance liquid chromatography (HPLC) analyses were carried out on an Agilent 1100 series system with an Agilent Poroshell 120 EC-C18 column (length 150 mm, I.D. 4.6 mm) at 40 °C with a solvent flow rate of 0.4 mL/min using a UV detector operating at 340 nm. Glycosylation sites were analyzed by an Orbitrap Fusion™ Tribrid™ Mass Spectrometer coupled to a Dionex Ultimate 3000 HPLC with an EASY-Spray™ LC 100 Å C18 column (length 150 mm, I.D. 75  $\mu\text{m}$ ).

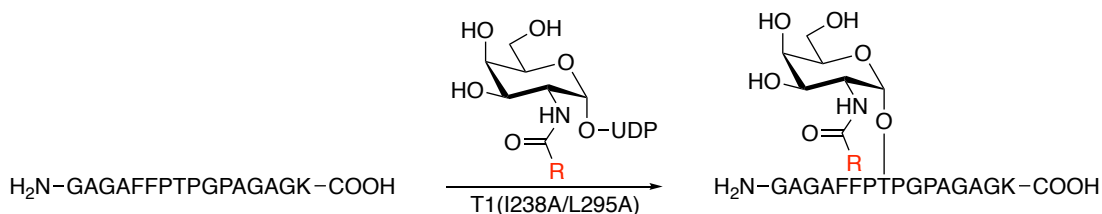


Analytical thin layer chromatography (TLC) was performed with Silicycle 60 Å silica gel plates and analyzed by UV illumination or KMnO<sub>4</sub> stain. Flash column chromatography was performed using silica gel (60 Å pore size, 40–63 μm, 230–400 mesh). Preparative HPLC was performed on a Varian ProStar system with an Agilent Microsorb 100-5 C18 column (length 250 mm, I.D. 21.4 mm) or an Agilent prep 100 Å C18 column (length 250 mm, I.D. 21.2 mm) with a solvent flow rate of 20 mL/min. <sup>1</sup>H NMR, <sup>13</sup>C NMR, and <sup>31</sup>P NMR data were collected on a Varian Inova 500 MHz spectrometer at ambient temperature.

### III. Glycosyltransferase Enzymatic Activity Assays (Figures S4, S5, S6, 3, 5A, and 5B)

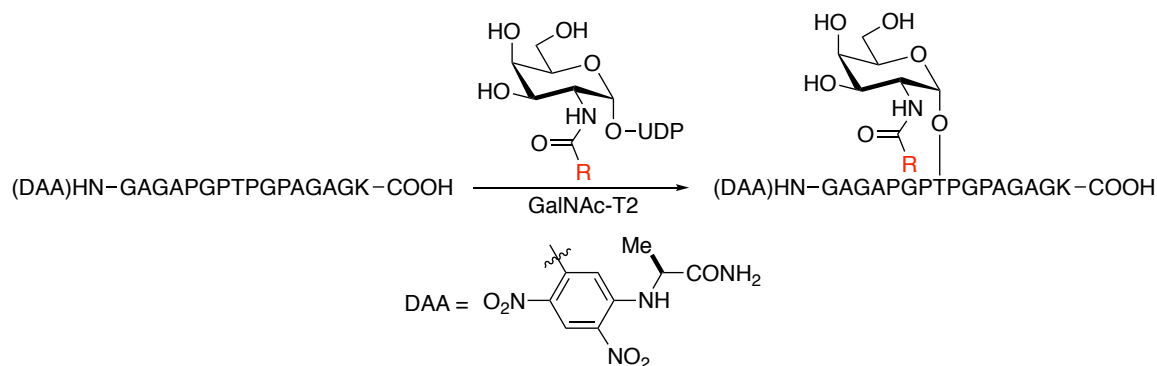


**Glycosylation by T2(I253A/L310A) with UDP-GalNAc analogs (Figure S4).** The glycosylation reaction was initiated by the addition of T2(I253A/L310A) (50.0 nM) in Tris-HCl buffer (16.7 mM Tris-HCl, 100 mM NaCl, 25% glycerol, pH = 7.4; 25.0 μL) to the mixture of UDP-GalNAc analog (500 μM), peptide (GAGAPGPTPGPGAG; 100 μM), and Coupling Phosphatase 1 (4 ng/μL) in Tris-HCl buffer (25 mM Tris-HCl, 20 mM MnCl<sub>2</sub>; 25.0 μL) at 0 °C, resulting in a final reaction mixture containing T2(I253A/L310A) (25.0 nM), peptide (50.0 μM), UDP-GalNAc analog (250 μM), and Coupling Phosphatase 1 (2 ng/μL) in Tris-HCl buffer (20.8 mM Tris-HCl, 10 mM MnCl<sub>2</sub>, 50 mM NaCl, 12.5% glycerol, pH = 7.4; 50.0 μL). The glycosylation was conducted at 37 °C for 1 h. The enzymatic activity was measured according to the manufacturer's protocol (Glycosyltransferase Activity Kit; R&D Systems, Inc.).

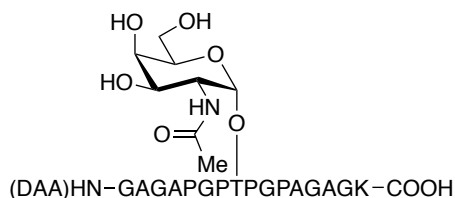


**Glycosylation by T1(I238A/L295A) with UDP-GalNAc analogs (Figure S5).** The glycosylation reaction was initiated by the addition of T1(I238A/L295A) (50.0 nM) in Tris-HCl buffer (16.7 mM Tris-HCl, 100 mM NaCl, 25% glycerol, pH = 7.4; 25.0 μL) to the mixture of UDP-GalNAc analog (1.00 mM), peptide (GAGAFFPTPGPAGAGK; 100 μM), and Coupling Phosphatase 1 (4 ng/μL) in Tris-HCl buffer (25 mM Tris-HCl, 20 mM MnCl<sub>2</sub>; 25.0 μL) at 0 °C, resulting in a final reaction mixture containing

T1(I238A/L295A) (25.0 nM), peptide (50.0  $\mu$ M), UDP-GalNAc analog (500  $\mu$ M), and Coupling Phosphatase 1 (2 ng/ $\mu$ L) in Tris-HCl buffer (20.8 mM Tris-HCl, 10 mM MnCl<sub>2</sub>, 50 mM NaCl, 12.5% glycerol, pH = 7.4; 50.0  $\mu$ L). The glycosylation was conducted at 37 °C for 1 h. The enzymatic activity was measured according to the manufacturer's protocol (Glycosyltransferase Activity Kit; R&D Systems, Inc.).



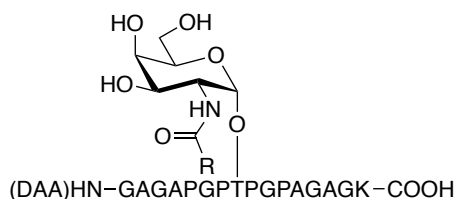
### Characterization of Peptide-1 glycosylation products of GalNAc-T2 with UDP-GalNAc and analogs (Figures S6 and 3).



#### 1-Peptide-1.

The product formation was determined by HPLC (22.5% acetonitrile/water; 0.1% trifluoroacetic acid; 0.4 mL/min) with  $t_r = 12.2$  min.

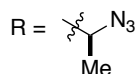
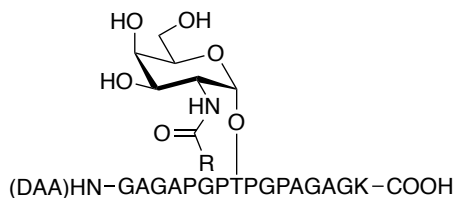
MS (ESI)  $m/z$  ( $[M+2H]^{2+}$ ) calcd for C<sub>71</sub>H<sub>110</sub>N<sub>22</sub>O<sub>28</sub>: 859.3930, found: 859.3926 (−0.46 ppm).



#### 2-Peptide-1.

The product formation was determined by HPLC (20% acetonitrile/water; 0.1% trifluoroacetic acid; 0.4 mL/min) with  $t_r = 33.6$  min.

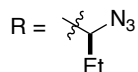
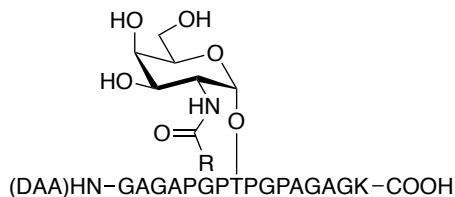
MS (ESI)  $m/z$  ( $[M+2H]^{2+}$ ) calcd for  $C_{71}H_{109}N_{25}O_{28}$ : 879.8937, found: 879.8928 (-1.02 ppm).



### (S)-3-Peptide-1.

The product formation was determined by HPLC (21.5% acetonitrile/water; 0.1% trifluoroacetic acid; 0.4 mL/min) with  $t_r = 27.2$  min.

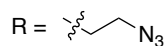
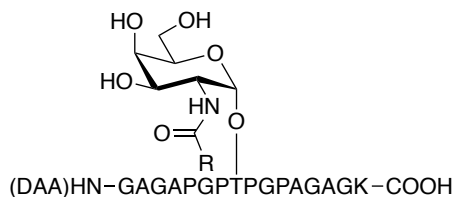
MS (ESI)  $m/z$  ( $[M+2H]^{2+}$ ) calcd for  $C_{72}H_{111}N_{25}O_{28}$ : 886.9015, found: 886.9015 (-0.02 ppm).



### (S)-4-Peptide-1.

The product formation was determined by HPLC (22.5% acetonitrile/water; 0.1% trifluoroacetic acid; 0.4 mL/min) with  $t_r = 30.2$  min.

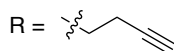
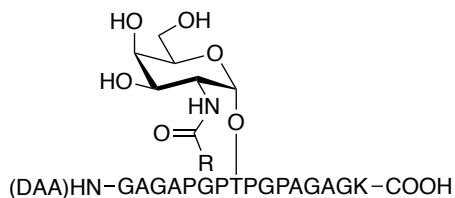
MS (ESI)  $m/z$  ( $[M+2H]^{2+}$ ) calcd for  $C_{73}H_{113}N_{25}O_{28}$ : 893.9093, found: 893.9088 (-0.61 ppm).



### 7-Peptide-1.

The product formation was determined by HPLC (21% acetonitrile/water; 0.1% trifluoroacetic acid; 0.4 mL/min) with  $t_r = 29.0$  min.

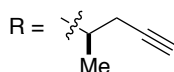
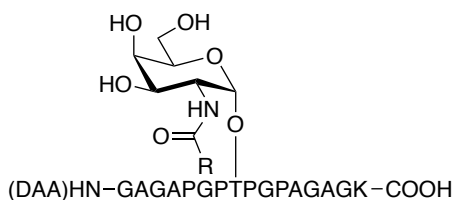
MS (ESI)  $m/z$  ( $[M+2H]^{2+}$ ) calcd for  $C_{72}H_{111}N_{25}O_{28}$ : 886.9015, found: 886.9011 (-0.47 ppm).



### 11-Peptide-1.

The product formation was determined by HPLC (21% acetonitrile/water; 0.1% trifluoroacetic acid; 0.4 mL/min) with  $t_r = 26.1$  min.

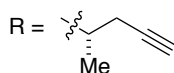
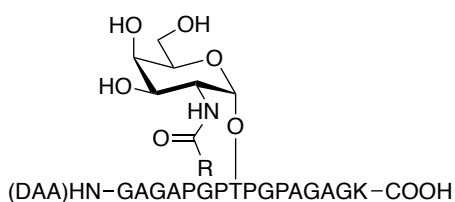
MS (ESI)  $m/z$  ( $[M+2H]^{2+}$ ) calcd for  $C_{74}H_{112}N_{22}O_{28}$ : 878.4008, found: 878.4016 (+0.89 ppm).



### (*R*)-12-Peptide-1.

The product formation was determined by HPLC (22.5% acetonitrile/water; 0.1% trifluoroacetic acid; 0.4 mL/min) with  $t_r = 22.6$  min.

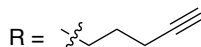
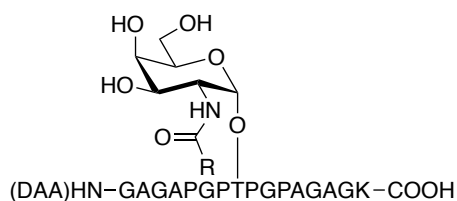
MS (ESI)  $m/z$  ( $[M+2H]^{2+}$ ) calcd for  $C_{75}H_{114}N_{22}O_{28}$ : 885.4086, found: 885.4092 (+0.63 ppm).



### (*S*)-12-Peptide-1.

The product formation was determined by HPLC (22.5% acetonitrile/water; 0.1% trifluoroacetic acid; 0.4 mL/min) with  $t_r = 21.9$  min.

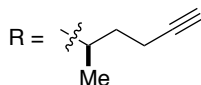
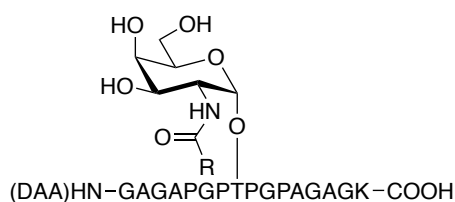
MS (ESI)  $m/z$  ( $[M+2H]^{2+}$ ) calcd for  $C_{75}H_{114}N_{22}O_{28}$ : 885.4086, found: 885.4090 (+0.40 ppm).



### 13-Peptide-1.

The product formation was determined by HPLC (22.5% acetonitrile/water; 0.1% trifluoroacetic acid; 0.4 mL/min) with  $t_r = 21.8$  min.

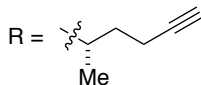
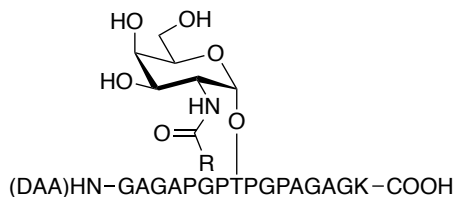
MS (ESI)  $m/z$  ( $[M+2H]^{2+}$ ) calcd for  $C_{75}H_{114}N_{22}O_{28}$ : 885.4086, found: 885.4086 (-0.05 ppm).



### (R)-14-Peptide-1.

The product formation was determined by HPLC (22.5% acetonitrile/water; 0.1% trifluoroacetic acid; 0.4 mL/min) with  $t_r = 30.4$  min.

MS (ESI)  $m/z$  ( $[M+2H]^{2+}$ ) calcd for  $C_{76}H_{116}N_{22}O_{28}$ : 892.4165, found: 892.4164 (-0.08 ppm).

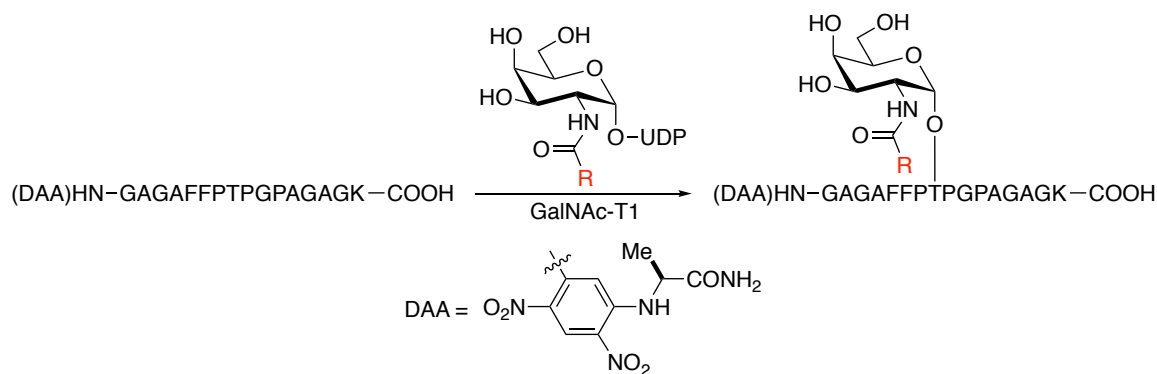


### (S)-14-Peptide-1.

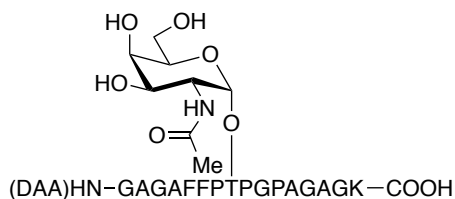
The product formation was determined by HPLC (22.5% acetonitrile/water; 0.1% trifluoroacetic acid; 0.4 mL/min) with  $t_r = 30.5$  min.



MS (ESI)  $m/z$  ( $[M+2H]^{2+}$ ) calcd for  $C_{76}H_{116}N_{22}O_{28}$ : 892.4165, found: 892.4172 (+0.82 ppm).



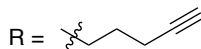
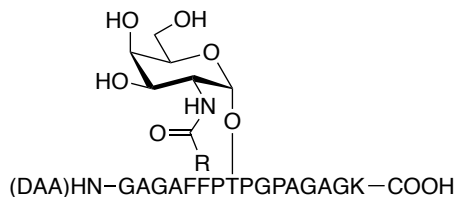
### Characterization of Peptide-2 glycosylation products of GalNAc-T1 with UDP-GalNAc and analogs (Figures 5A and 5B).



#### 1-Peptide-2.

The product formation was determined by HPLC (30% acetonitrile/water; 0.1% trifluoroacetic acid; 0.4 mL/min) with  $t_r = 25.8$  min.

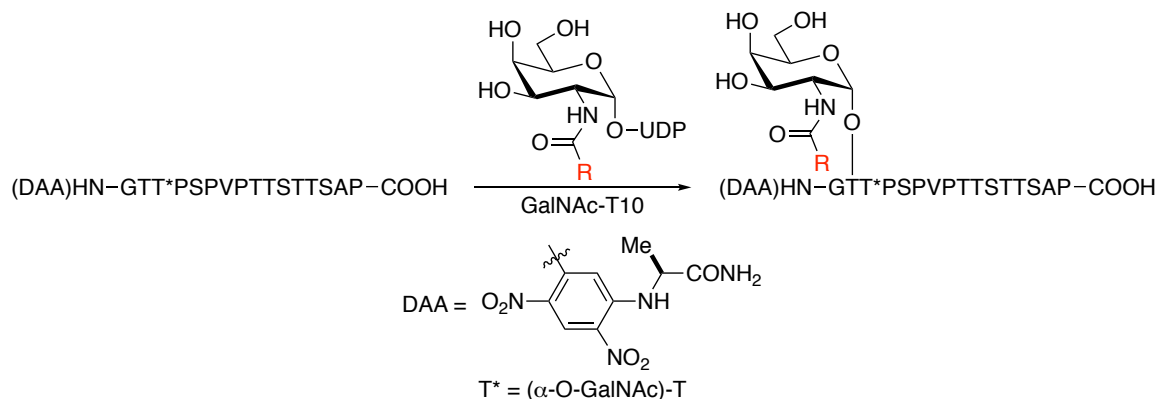
MS (ESI)  $m/z$  ( $[M+2H]^{2+}$ ) calcd for  $C_{82}H_{118}N_{22}O_{28}$ : 929.4243, found: 929.4242 (-0.10 ppm).



#### 13-Peptide-2.

The product formation was determined by HPLC (30% acetonitrile/water; 0.1% trifluoroacetic acid; 0.4 mL/min) with  $t_r = 42.1$  min.

MS (ESI)  $m/z$  ( $[M+2H]^{2+}$ ) calcd for  $C_{86}H_{122}N_{22}O_{28}$ : 955.4399, found: 955.4413 (+1.42 ppm).



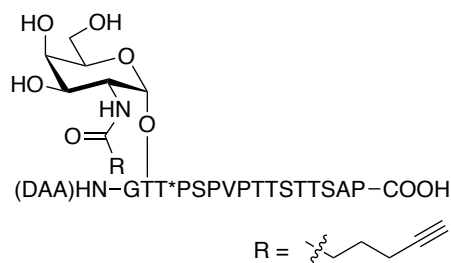
**Characterization of Peptide-3 glycosylation products of GalNAc-T10 with UDP-GalNAc and analogs (Figures 5A and 5B).**



**1-Peptide-3.**

The product formation was determined by HPLC (21% acetonitrile/water; 0.1% trifluoroacetic acid; 0.4 mL/min) with  $t_r = 18.2$  min.

MS (ESI)  $m/z$  ( $[M+2H]^{2+}$ ) calcd for  $\text{C}_{88}\text{H}_{140}\text{N}_{22}\text{O}_{41}$ : 1080.4773, found: 1080.4797 (+2.21 ppm).



**13-Peptide-3.**

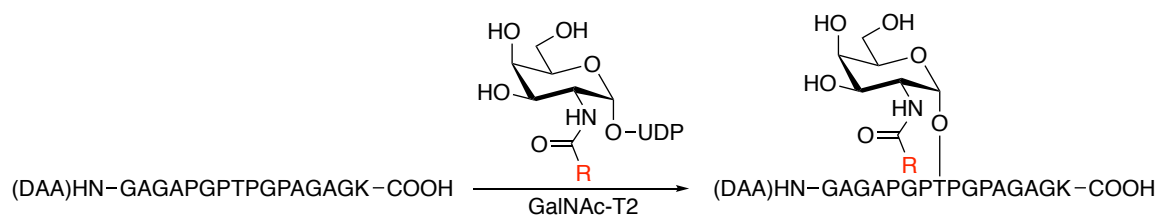
The product formation was determined by HPLC (22.5% acetonitrile/water; 0.1% trifluoroacetic acid; 0.4 mL/min) with  $t_r = 31.5$  min.

MS (ESI)  $m/z$  ( $[M+2H]^{2+}$ ) calcd for  $\text{C}_{92}\text{H}_{144}\text{N}_{22}\text{O}_{41}$ : 1106.4930, found: 1106.4939 (+0.85 ppm).

#### IV. Glycosylation Reaction Kinetics (Table 1; Figure 5C)

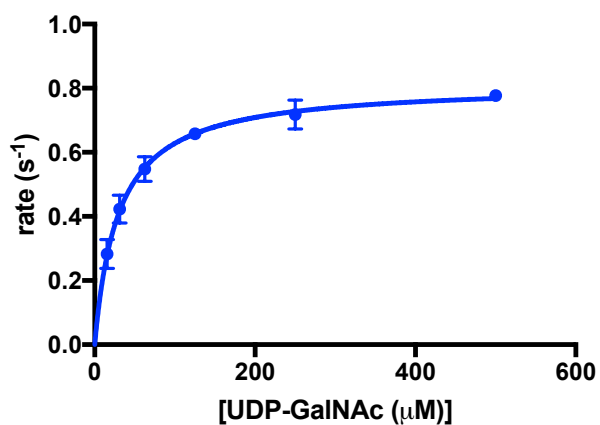
##### General reaction conditions for GalNAc-T kinetics

The glycosylation reaction was initiated by the addition of GalNAc-T in Tris-HCl buffer (16.7 mM Tris-HCl, 100 mM NaCl, 25% glycerol, pH = 7.4; 35.0  $\mu$ L) to the mixture of UDP-sugar (31.2  $\mu$ M, 62.5  $\mu$ M, 125  $\mu$ M, 250  $\mu$ M, 500  $\mu$ M and 1.00 mM), and peptide (Peptide-1 (**1**, (*S*)-**3**, and **11**) = 534  $\mu$ M; Peptide-1 (**7**, (*S*)-**12**, **13**) = 500  $\mu$ M); Peptide-2 = 500  $\mu$ M; Peptide-3 = 532  $\mu$ M) in Tris-HCl buffer (25 mM Tris-HCl, 20 mM MnCl<sub>2</sub>; 35.0  $\mu$ L) at 0  $^{\circ}$ C, resulting in a final reaction mixture containing GalNAc-T, UDP-sugar (15.6  $\mu$ M, 31.2  $\mu$ M, 62.5  $\mu$ M, 125  $\mu$ M, 250  $\mu$ M, and 500  $\mu$ M), and peptide (Peptide-1 (**1**, (*S*)-**3**, and **11**) = 267  $\mu$ M; Peptide-1 (**7**, (*S*)-**12**, **13**) = 250  $\mu$ M); Peptide-2 = 250  $\mu$ M; Peptide-3 = 266  $\mu$ M) in Tris-HCl buffer (20.8 mM Tris-HCl, 10 mM MnCl<sub>2</sub>, 50 mM NaCl, 12.5% glycerol, pH = 7.4; 70.0  $\mu$ L). The glycosylation was conducted at 37  $^{\circ}$ C. Aliquots were taken at 5, 10, and 15 min and quenched by the addition of aqueous EDTA (150 mM, pH = 8.0). The glycopeptide formation was analyzed by HPLC, and initial rates were calculated.



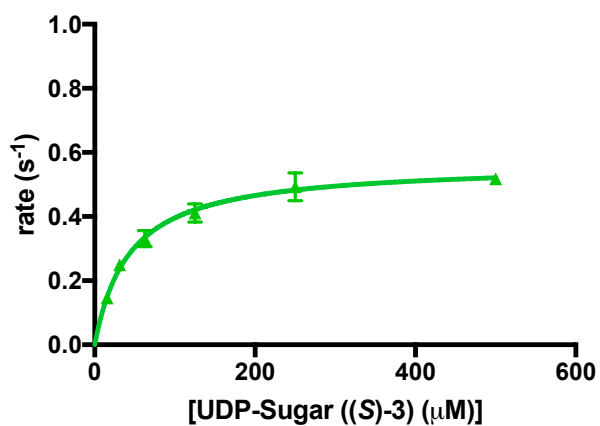
##### Determination of kinetic parameters for GalNAc-T2 with Peptide-1 (Table 1).

The reaction conditions differ from the general reaction conditions for GalNAc-T kinetics as described here. GalNAc-T concentrations were as follows: WT-T2 with **1** (initial = 8.33 nM, final = 4.17 nM); T2(I253A/L310A) with (*S*)-**3** (initial = 8.59 nM, final = 4.30 nM); T2(I253A/L310A) with **7** (initial = 20.0 nM, final = 10 nM); T2(I253A/L310A) with **11** (initial = 8.59 nM, final = 4.30 nM); T2(I253A/L310A) with (*S*)-**12** (initial = 20.0 nM, final = 10 nM); T2(I253A/L310A) with **13** (initial = 30.0 nM, final = 15.0 nM). These reactions had aliquots removed after 4, 8, and 12 min instead: T2(I253A/L310A)/**7** and T2(I253A/L310A)/(*S*)-**12**.



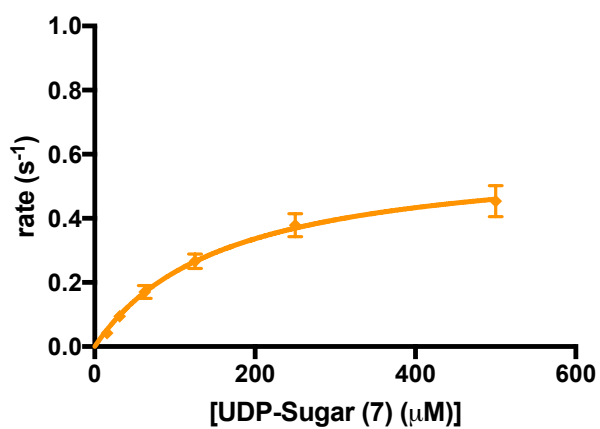
**Kinetics of WT-T2 with UDP-GalNAc (1).**

$k_{\text{cat}} = 0.813 \pm 0.017 \text{ s}^{-1}$ ;  $K_{\text{m}} = 30 \pm 2 \text{ } \mu\text{M}$ ;  $k_{\text{cat}}/K_{\text{m}} = 28 \text{ mM}^{-1} \text{ s}^{-1}$ .



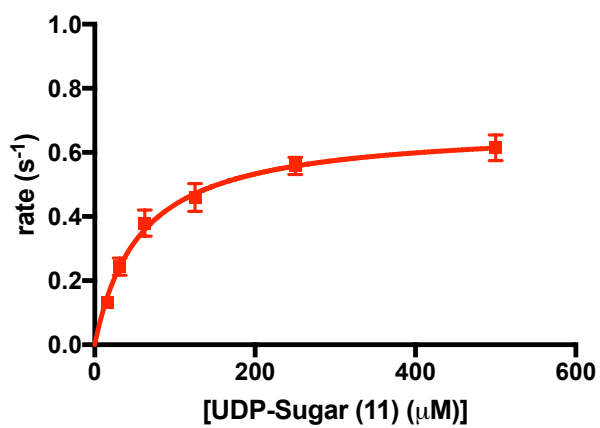
**Kinetics of T2(I253A/L310A) with UDP-Sugar ((S)-3).**

$k_{\text{cat}} = 0.566 \pm 0.014 \text{ s}^{-1}$ ;  $K_{\text{m}} = 43 \pm 4 \text{ } \mu\text{M}$ ;  $k_{\text{cat}}/K_{\text{m}} = 13 \text{ mM}^{-1} \text{ s}^{-1}$ .



**Kinetics of T2(I253A/L310A) with UDP-Sugar (7).**

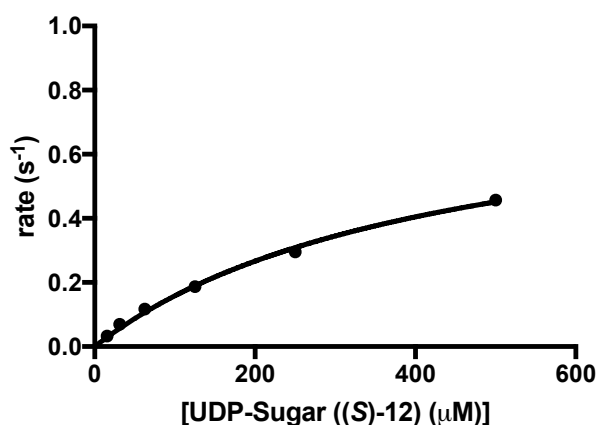
$k_{\text{cat}} = 0.61 \pm 0.03 \text{ s}^{-1}$ ;  $K_m = 1.6 \times 10^2 \pm 2 \times 10^1 \text{ μM}$ ;  $k_{\text{cat}}/K_m = 3.8 \text{ mM}^{-1} \text{ s}^{-1}$ .



**Kinetics of T2(I253A/L310A) with UDP-Sugar (11).**

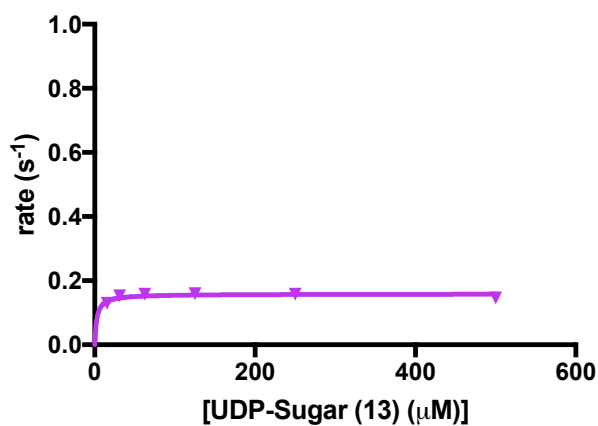
$k_{\text{cat}} = 0.68 \pm 0.02 \text{ s}^{-1}$ ;  $K_m = 56 \pm 6 \text{ μM}$ ;  $k_{\text{cat}}/K_m = 12 \text{ mM}^{-1} \text{ s}^{-1}$ .





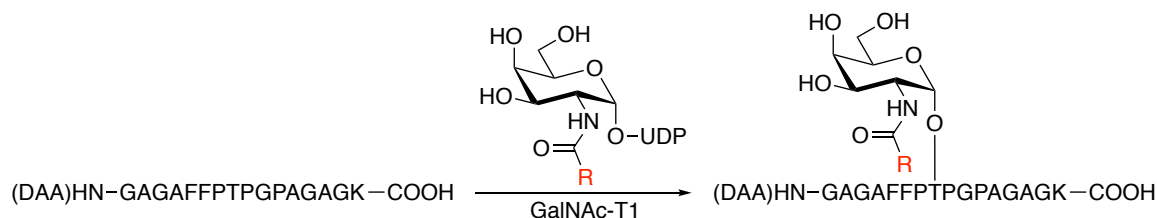
**Kinetics of T2(I253A/L310A) with UDP-Sugar ((S)-12).**

$k_{\text{cat}} = 0.84 \pm 0.05 \text{ s}^{-1}$ ;  $K_m = 4.3 \times 10^2 \pm 5 \times 10^1 \text{ μM}$ ;  $k_{\text{cat}}/K_m = 2.0 \text{ mM}^{-1} \text{ s}^{-1}$ .



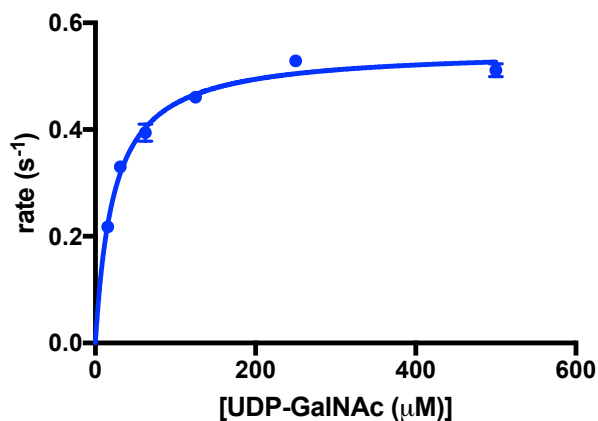
**Kinetics of T2(I253A/L310A) with UDP-Sugar (13).**

$k_{\text{cat}} = 0.158 \pm 0.003 \text{ s}^{-1}$ ;  $K_m = 2.6 \pm 0.8 \text{ μM}$ ;  $k_{\text{cat}}/K_m = 61 \text{ mM}^{-1} \text{ s}^{-1}$ .



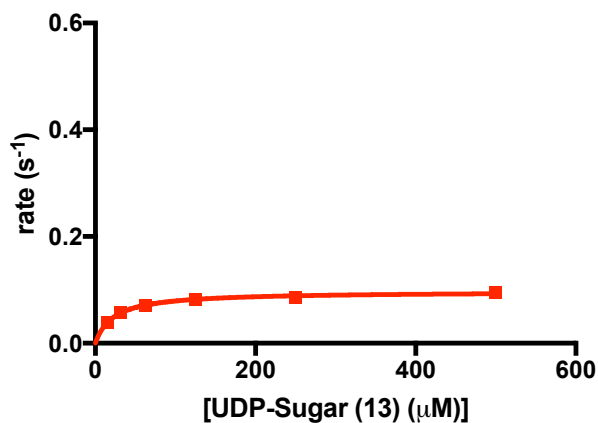
**Determination of kinetic parameters for GalNAc-T1 with Peptide-2 (Figure 5C).**

The reaction conditions differ from the general reaction conditions for GalNAc-T kinetics as described here. GalNAc-T concentrations were as follows: WT-T1 and **1** (initial = 100 nM, final = 50.0 nM); T1(I238A/L295A) and **13** (initial = 100 nM, final = 50.0 nM).



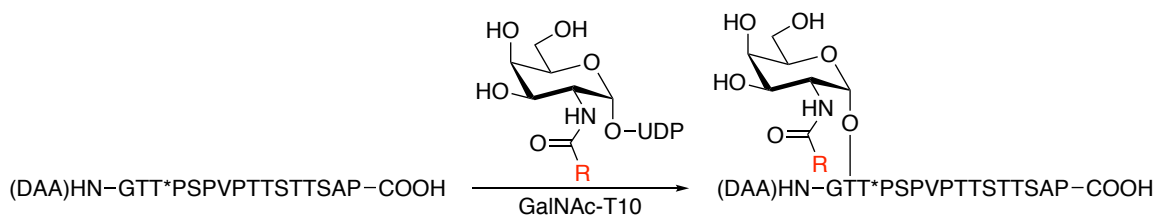
**Kinetics of WT-1 with UDP-GalNAc (1).**

$k_{\text{cat}} = 0.551 \pm 0.008 \text{ s}^{-1}$ ;  $K_{\text{m}} = 22.8 \pm 1.5 \text{ } \mu\text{M}$ ;  $k_{\text{cat}}/K_{\text{m}} = 24.2 \text{ mM}^{-1} \text{ s}^{-1}$ .



**Kinetics of T1(I238A/L295A) with UDP-Sugar (13).**

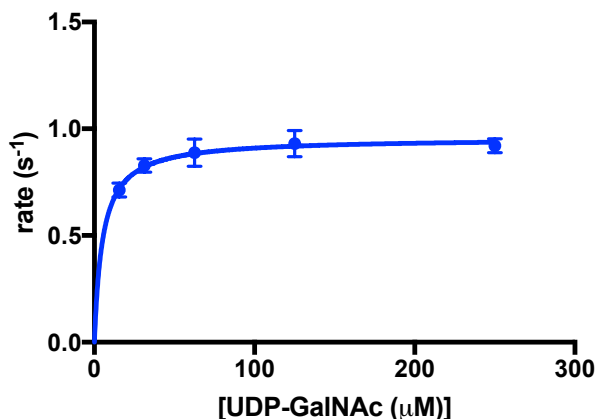
$k_{\text{cat}} = 0.097 \pm 0.002 \text{ s}^{-1}$ ;  $K_{\text{m}} = 22 \pm 2 \text{ } \mu\text{M}$ ;  $k_{\text{cat}}/K_{\text{m}} = 4.3 \text{ mM}^{-1} \text{ s}^{-1}$ .



**Determination of kinetic parameters for GalNAc-T10 with Peptide-3 (Figure 5C).**

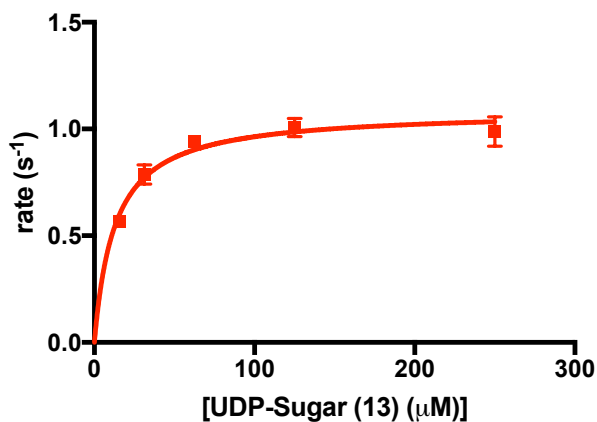
The reaction conditions differ from the general reaction conditions for GalNAc-T kinetics as described here. GalNAc-T concentrations were as follows: WT-T10 and **1** (initial = 6.18 nM, final = 3.09 nM); T10(I266A/L321A) and **13** (initial = 6.17 nM, final = 3.08

nM. For GalNAc-T10, UDP-sugar final concentrations were varied from 15.6  $\mu\text{M}$  to 250  $\mu\text{M}$  (31.2  $\mu\text{M}$  to 500  $\mu\text{M}$  initial concentrations).



#### Kinetics of WT-T10 with UDP-GalNAc (1).

$k_{\text{cat}} = 0.956 \pm 0.019 \text{ s}^{-1}$ ;  $K_{\text{m}} = 5.1 \pm 0.9 \text{ }\mu\text{M}$ ;  $k_{\text{cat}}/K_{\text{m}} = 1.9 \times 10^2 \text{ mM}^{-1} \text{ s}^{-1}$ .



#### Kinetics of T10(I266A/L321A) with UDP-Sugar (13).

$k_{\text{cat}} = 1.09 \pm 0.03 \text{ s}^{-1}$ ;  $K_{\text{m}} = 12.7 \pm 1.6 \text{ }\mu\text{M}$ ;  $k_{\text{cat}}/K_{\text{m}} = 85.4 \text{ mM}^{-1} \text{ s}^{-1}$ .

### V. UDP-Sugar Competition Experiment (Figure 4)

#### Procedure.

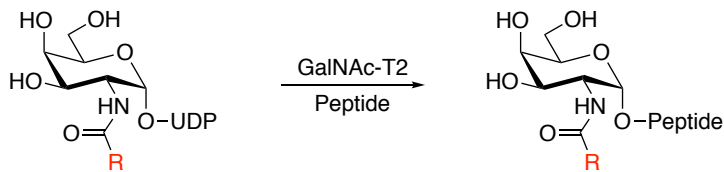
The glycosylation reaction was initiated by the addition of WT-T2 (50.0 nM) in Tris-HCl buffer (16.7 mM Tris-HCl, 100 mM NaCl, 25% glycerol, pH = 7.4; 35.0  $\mu\text{L}$ ) to the mixture of UDP-GalNAc (500  $\mu\text{M}$ ), UDP-GalNAc analog (500  $\mu\text{M}$ ) and Peptide-1 ((DAA)GAGAPGPTPGPAGAGK; 100  $\mu\text{M}$ ) in Tris-HCl buffer (25 mM Tris-HCl, 20 mM  $\text{MnCl}_2$ ; 35.0  $\mu\text{L}$ ) at 0  $^\circ\text{C}$ , resulting in a final reaction mixture containing WT-T2

(25.0 nM), UDP-GalNAc (250  $\mu$ M), UDP-GalNAc analog (250  $\mu$ M), and peptide (50.0  $\mu$ M) in Tris-HCl buffer (20.8 mM Tris-HCl, 10 mM MnCl<sub>2</sub>, 50 mM NaCl, 12.5% glycerol, pH = 7.4; 70.0  $\mu$ L). The glycosylation was conducted at 37 °C. Reaction progress was monitored by taking aliquots and quenching them by the addition of aqueous EDTA (150 mM, pH = 8.0). The ratio between the two glycopeptides was determined by HPLC.

## VI. Glycosylation of Natural Peptide Substrates by GalNAc-Ts (Figure 6)

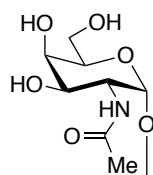
### Procedure.

The glycosylation reaction was initiated by the addition of wild-type or double mutant GalNAc-T (-T2 = 60.0 nM; -T1 = 30.0 nM; -T10 = 200 nM) in Tris-HCl buffer (16.7 mM Tris-HCl, 100 mM NaCl, 25% glycerol, pH = 7.4; 25.0  $\mu$ L) to the mixture of UDP-sugar (**1** was used for WT-T2, -T1, -T10, and **13** was used for double mutant -T2, -T1, -T10; 1.00 mM), and peptide (MUC5AC-3, MUC5AC-13, or EA2; 200  $\mu$ M) in Tris-HCl buffer (25 mM Tris-HCl, 20 mM MnCl<sub>2</sub>; 25.0  $\mu$ L) at 0 °C, resulting in a final reaction mixture containing wild-type or double mutant GalNAc-T (-T2 = 30.0 nM; -T1 = 15.0 nM; -T10 = 100 nM), UDP-sugar (**1** was used for WT-T2, -T1, -T10, and **13** was used for double mutant -T2, -T1, -T10; 500  $\mu$ M), and peptide (MUC5AC-3, MUC5AC-13, or EA2; 100  $\mu$ M) in Tris-HCl buffer (20.8 mM Tris-HCl, 10 mM MnCl<sub>2</sub>, 50 mM NaCl, 12.5% glycerol, pH = 7.4; 50.0  $\mu$ L). The glycosylation was conducted at 37 °C. Reaction progress was monitored by taking aliquots and quenching them by the addition of aqueous EDTA (150 mM, pH = 8.0; 25.0  $\mu$ L). Glycopeptide formation and glycosite occupancy were analyzed on an Orbitrap Fusion™ Tribrid™ Mass Spectrometer (Thermo) coupled to a Dionex Ultimate 3000 HPLC with an EASY-Spray™ LC (100 Å C18 column length 150 mm, I.D. 75  $\mu$ m). The samples were eluted at 0.3  $\mu$ L/min using a 90-min gradient and a 185-min instrument method. Solvent A was 0.1% formic acid in water, and solvent B was 0.1% formic acid in acetonitrile. The gradient profile varied by peptide. The instrument method used an MS1 resolution of 60,000 at FWHM of 400  $m/z$ , an automatic gain control (AGC) target of 3e5, and a mass range from 300 to 1,500  $m/z$ . Electron transfer dissociation (ETD) MS2 spectra were generated at top speed for 3 s. ETD parameters were as follows: calibrated charge dependent ETD times, 2e5 reagent target, and precursor AGC target of 1e4. Glycopeptides were manually sequenced using Xcalibur software (Thermo). Relative abundances were obtained by integrating under the extracted ion chromatograms ( $\pm$ 10 ppm).



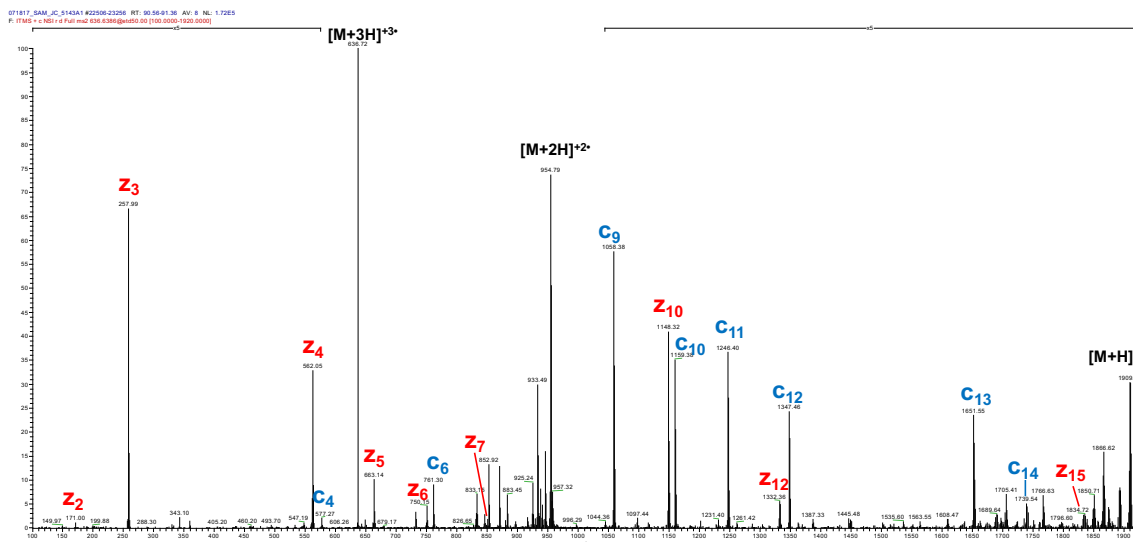
10~20% peptide conversion

### Characterization of peptides glycosylated by GalNAc-T2.



H<sub>2</sub>N-GTT\*PSPVPTTSTTSAP-COOH

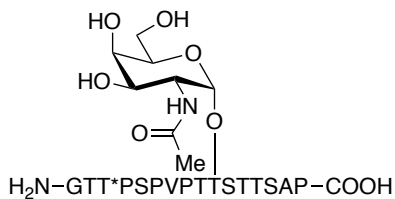
G T T \* P S P V P T T S T T \* S A P



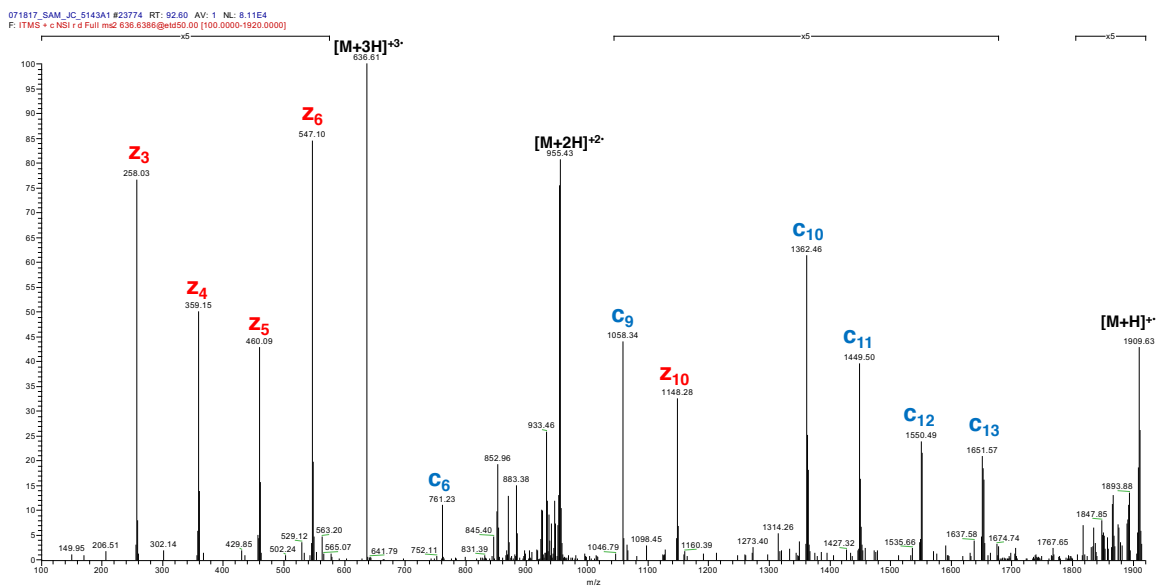
### GTT\*PSPVPTTSTTSAP.

Product formation and glycosite occupancy were determined using ETD fragmentation on an Orbitrap Fusion™ Tribrid™ Mass Spectrometer coupled to a Dionex Ultimate 3000 HPLC. The gradient profile was as follows (min/%B): 0:3, 3:5, 93:25, 103:42, 104:98, 109:98, 110:3, 185:3 with  $t_r = 91.0$  min.

MS (ESI)  $m/z$  ( $[M+2H]^{2+}$ ) calcd for C<sub>79</sub>H<sub>132</sub>N<sub>18</sub>O<sub>36</sub>: 954.4526, found: 954.4540 (+1.49 ppm).



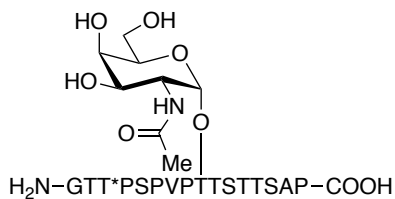
G T T \* P S P V P T T \* S T T S A P



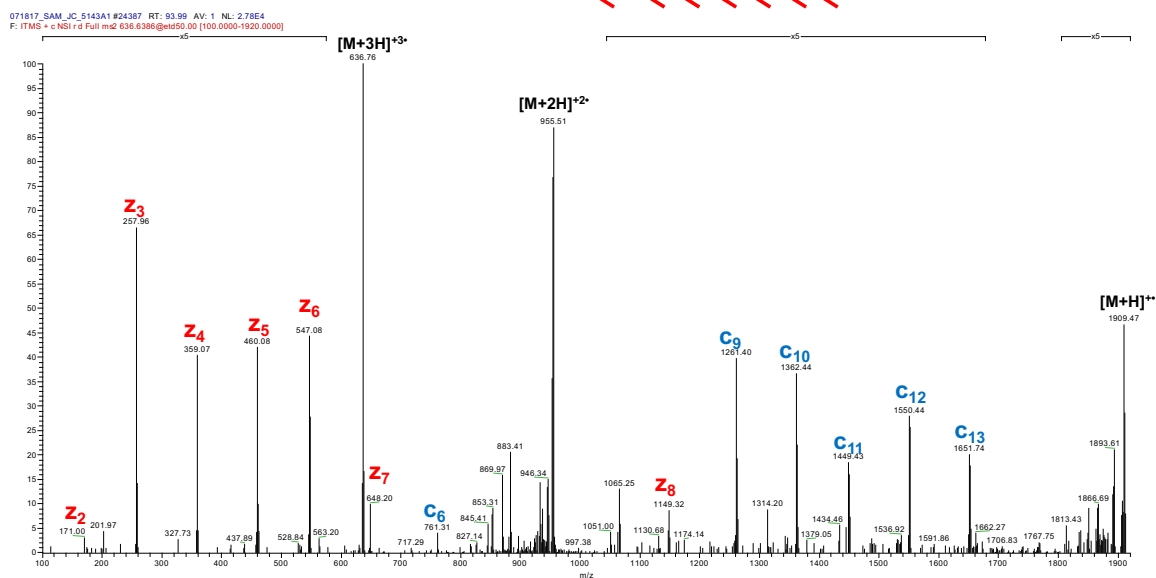
### GTT\*PSPVPTT\*STTSAP.

Product formation and glycosite occupancy were determined using ETD fragmentation on an Orbitrap Fusion™ Tribrid™ Mass Spectrometer coupled to a Dionex Ultimate 3000 HPLC. The gradient profile was as follows (min/%B): 0:3, 3:5, 93:25, 103:42, 104:98, 109:98, 110:3, 185:3 with  $t_r = 92.6$  min.

MS (ESI)  $m/z$  ( $[M+2H]^{2+}$ ) calcd for C<sub>79</sub>H<sub>132</sub>N<sub>18</sub>O<sub>36</sub>: 954.4526, found: 954.4545 (+2.01 ppm).



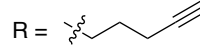
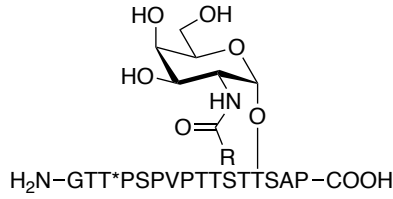
GTT\*PSPVPTTSTTSAP



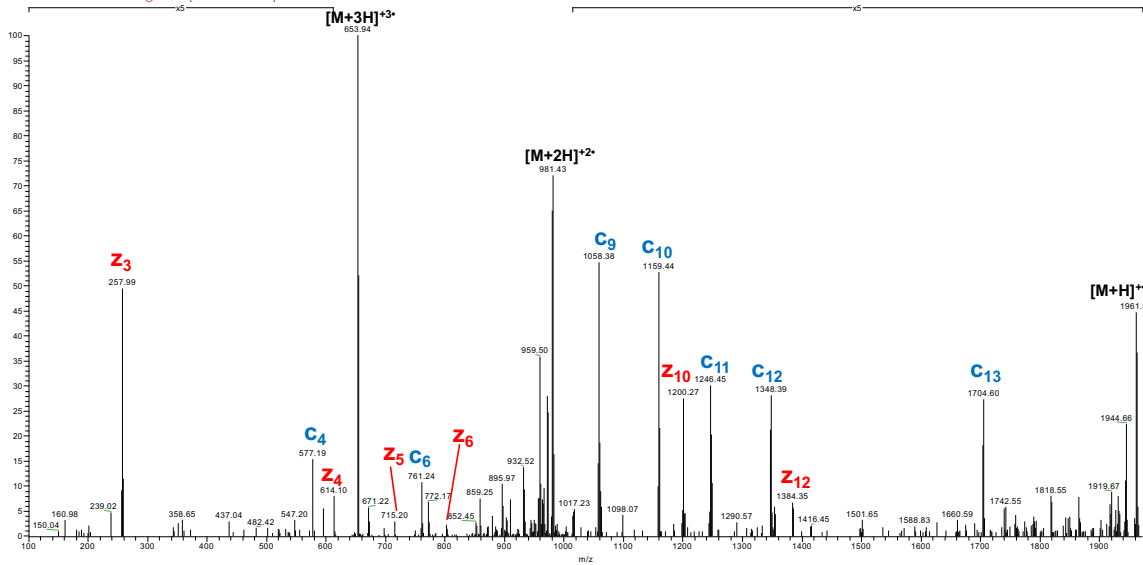
### GTT\*PSPVPTTSTTSAP.

Product formation and glycosite occupancy were determined using ETD fragmentation on an Orbitrap Fusion™ Tribrid™ Mass Spectrometer coupled to a Dionex Ultimate 3000 HPLC. The gradient profile was as follows (min/%B): 0:3, 3:5, 93:25, 103:42, 104:98, 109:98, 110:3, 185:3 with  $t_r = 94.0$  min.

MS (ESI)  $m/z$  ( $[M+2H]^{2+}$ ) calcd for C<sub>79</sub>H<sub>132</sub>N<sub>18</sub>O<sub>36</sub>: 954.4526, found: 954.4534 (+0.86 ppm).



071817\_SAM\_JC\_514382\_927320 RT: 101.20 AV: 1 NL: 1.00ES  
F: ITMS + c NSI r4 Full m/z 653.9821 @e0150.00 [100.0000-1972.0000]

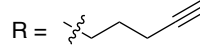
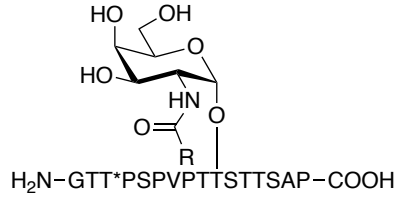


### GTT\*PSPVPTTST(13-T)SAP.

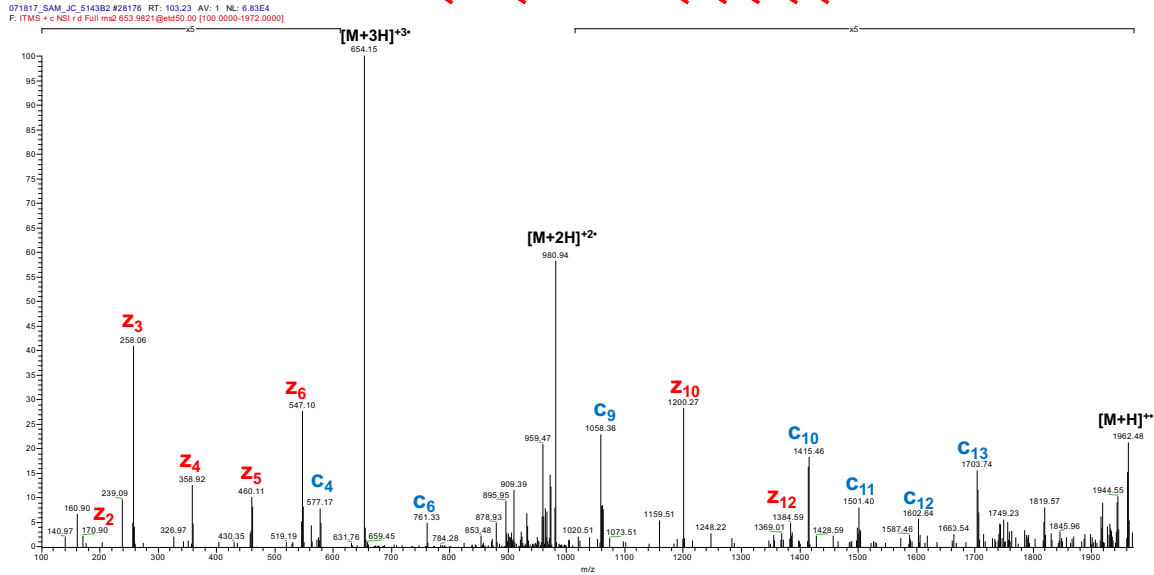
Product formation and glycosite occupancy were determined using ETD fragmentation on an Orbitrap Fusion™ Tribrid™ Mass Spectrometer coupled to a Dionex Ultimate 3000 HPLC. The gradient profile was as follows (min/%B): 0:3, 3:5, 93:25, 103:42, 104:98, 109:98, 110:3, 185:3 with  $t_r = 100.6$  min.

MS (ESI)  $m/z$  ( $[M+2H]^{2+}$ ) calcd for C<sub>83</sub>H<sub>136</sub>N<sub>18</sub>O<sub>36</sub>: 980.4682, found: 980.4717 (+3.54 ppm).





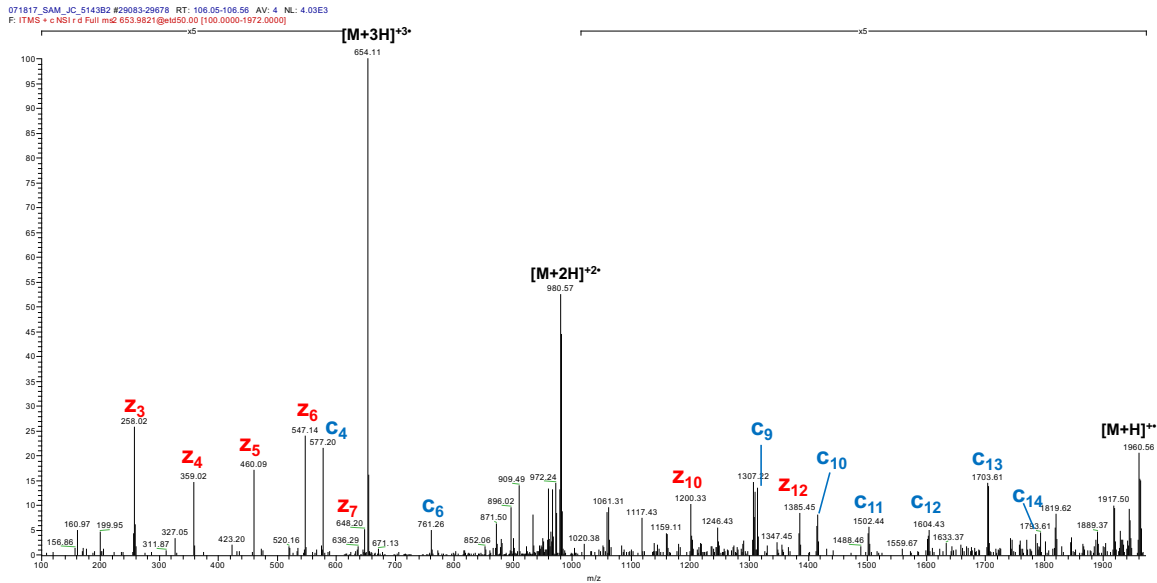
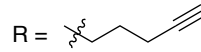
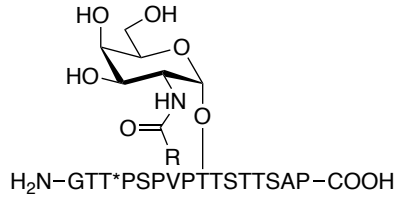
GTT\*PSPVPTT\*STTSAP



### GTT\*PSPVPT(13-T)STTSAP.

Product formation and glycosite occupancy were determined using ETD fragmentation on an Orbitrap Fusion™ Tribrid™ Mass Spectrometer coupled to a Dionex Ultimate 3000 HPLC. The gradient profile was as follows (min/%B): 0:3, 3:5, 93:25, 103:42, 104:98, 109:98, 110:3, 185:3 with  $t_r = 102.7$  min.

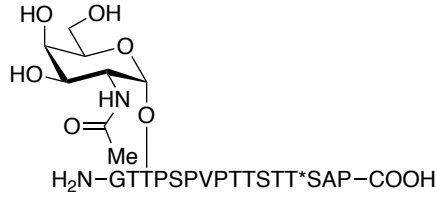
MS (ESI)  $m/z$  ( $[M+2H]^{2+}$ ) calcd for C<sub>83</sub>H<sub>136</sub>N<sub>18</sub>O<sub>36</sub>: 980.4682, found: 980.4714 (+3.23 ppm).



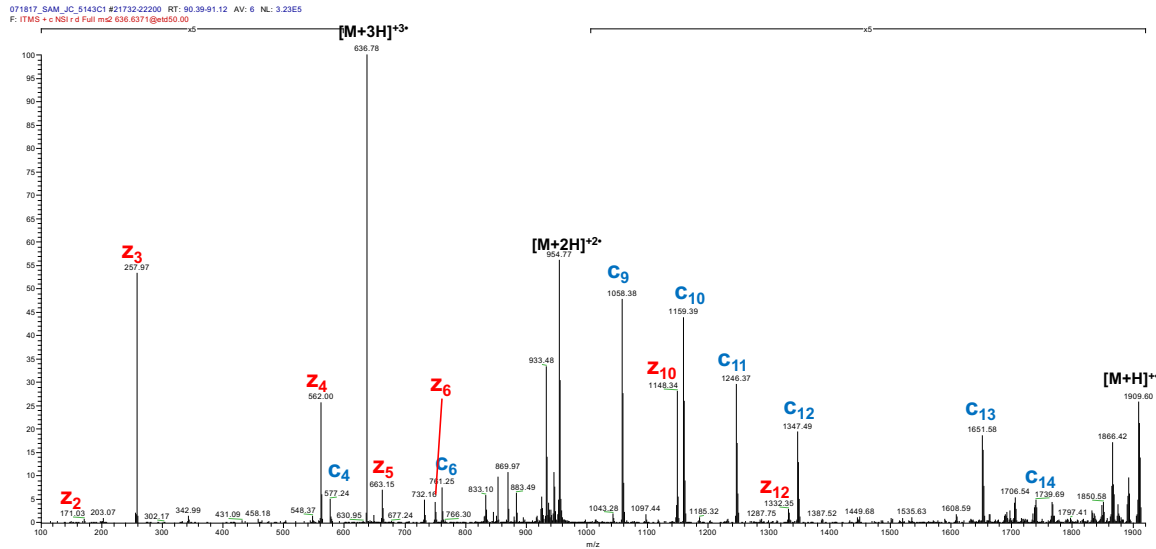
### GTT\*PSPVPTTSTTSAP.

Product formation and glycosite occupancy were determined using ETD fragmentation on an Orbitrap Fusion™ Tribrid™ Mass Spectrometer coupled to a Dionex Ultimate 3000 HPLC. The gradient profile was as follows (min/%B): 0:3, 3:5, 93:25, 103:42, 104:98, 109:98, 110:3, 185:3 with  $t_r = 105.2$  min.

MS (ESI)  $m/z$  ( $[M+2H]^{2+}$ ) calcd for C<sub>83</sub>H<sub>136</sub>N<sub>18</sub>O<sub>36</sub>: 980.4682, found: 980.4710 (+2.83 ppm).



G (TT)\* PLS P[V P T]T[S]T[S]A P

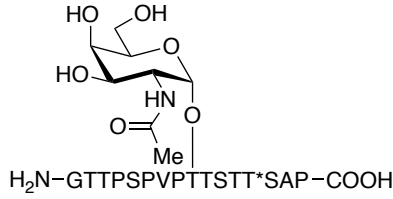


### GTT\*PSPVPTTSTT\*SAP.

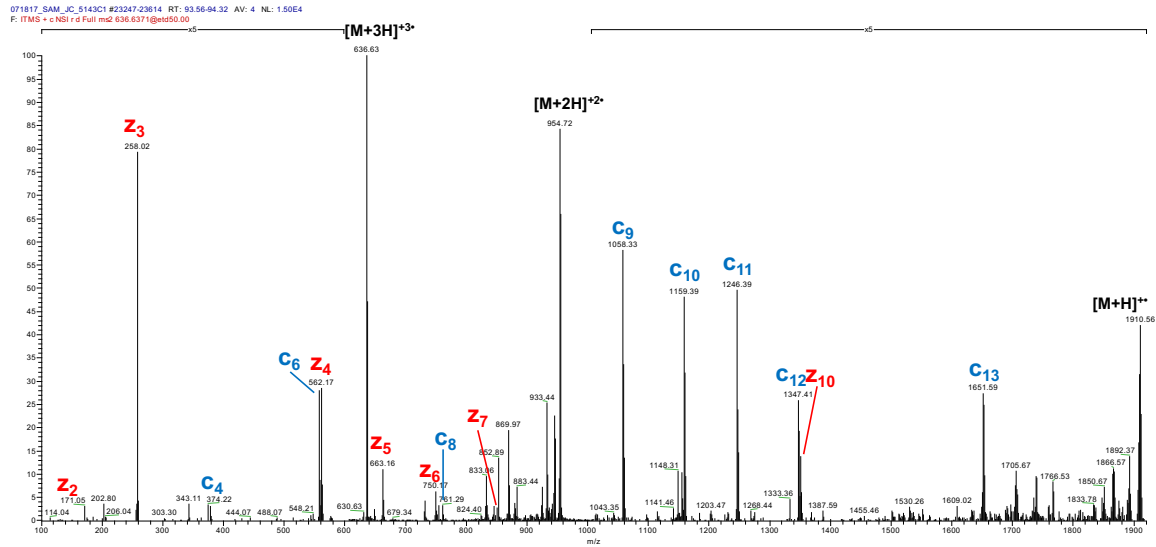
The mass spectrum for this peptide does not allow the unambiguous assignment of the glycosite to Thr2 or Thr3. However, this compound has the identical fragmentation pattern and retention time as the major product of WT-T2/1 with MUC5AC-3, indicating that the glycopeptides are likely identical and that the occupied glycosite is Thr3.

Product formation and glycosite occupancy were determined using ETD fragmentation on an Orbitrap Fusion™ Tribrid™ Mass Spectrometer coupled to a Dionex Ultimate 3000 HPLC. The gradient profile was as follows (min/%B): 0:3, 3:5, 93:25, 103:42, 104:98, 109:98, 110:3, 185:3 with  $t_r = 91.0$  min.

MS (ESI)  $m/z$  ( $[M+2H]^{2+}$ ) calcd for C<sub>79</sub>H<sub>132</sub>N<sub>18</sub>O<sub>36</sub>: 954.4526, found: 954.4551 (+2.64 ppm).



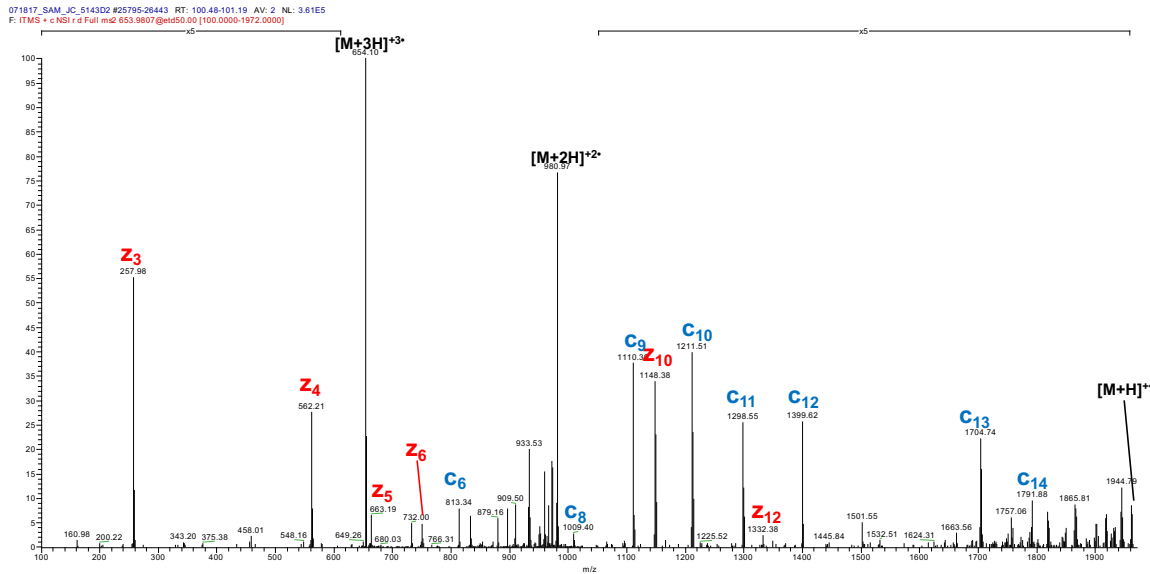
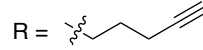
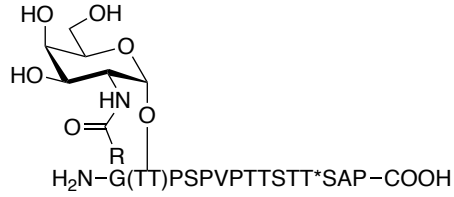
G T T P S P V P T\* T S T T\* S A P



### GTTPSPVPT\*TSTT\*SAP.

Product formation and glycosite occupancy were determined using ETD fragmentation on an Orbitrap Fusion™ Tribrid™ Mass Spectrometer coupled to a Dionex Ultimate 3000 HPLC. The gradient profile was as follows (min/%B): 0:3, 3:5, 93:25, 103:42, 104:98, 109:98, 110:3, 185:3 with  $t_r = 93.6$  min.

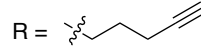
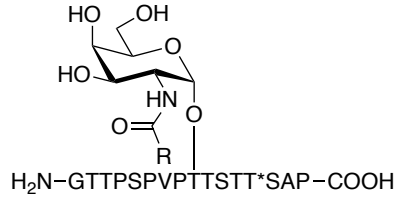
MS (ESI)  $m/z$  ( $[M+2H]^{2+}$ ) calcd for C<sub>79</sub>H<sub>132</sub>N<sub>18</sub>O<sub>36</sub>: 954.4526, found: 954.4528 (+0.23 ppm).



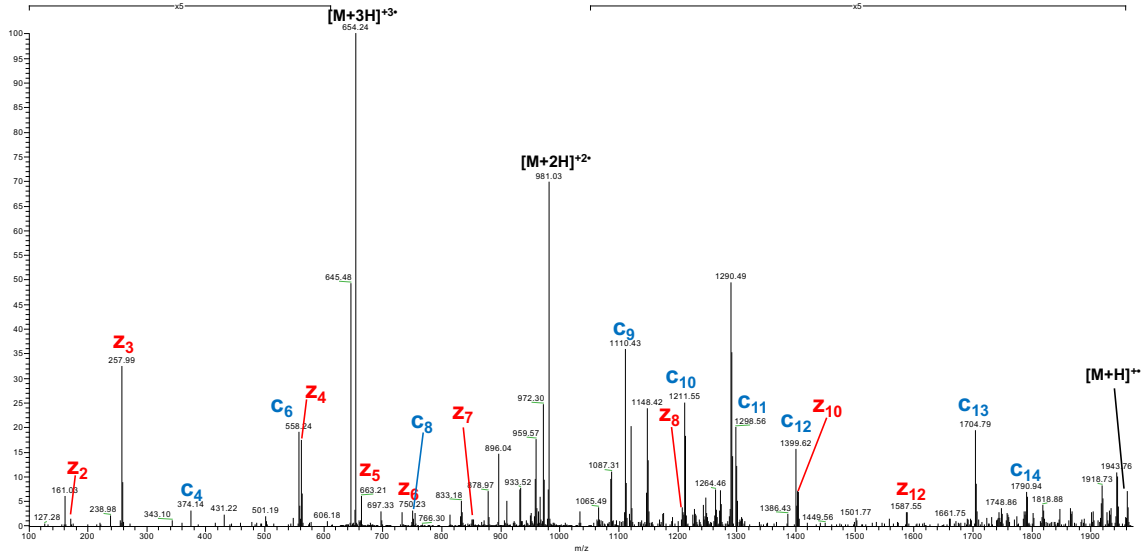
### G(13-(TT))PSPVPTTSTT\*SAP.

The glycosite cannot be unambiguously assigned, and either Thr2 or Thr3 (**13**-(TT)) is glycosylated by T2(I253A/L310A)/**13**. Product formation and glycosite occupancy were determined using ETD fragmentation on an Orbitrap Fusion™ Tribrid™ Mass Spectrometer coupled to a Dionex Ultimate 3000 HPLC. The gradient profile was as follows (min/%B): 0:3, 3:5, 93:25, 103:42, 104:98, 109:98, 110:3, 185:3 with  $t_r = 100.4$  min.

MS (ESI)  $m/z$  ( $[M+2H]^{2+}$ ) calcd for C<sub>83</sub>H<sub>136</sub>N<sub>18</sub>O<sub>36</sub>: 980.4682, found: 980.4715 (+3.34 ppm).



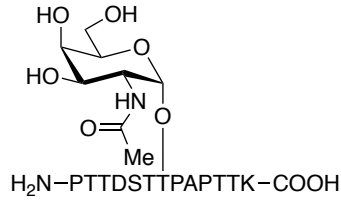
071817\_SAM\_AC\_5143D2 #27824-28251 RT: 104.84-105.13 AV: 2 NL: 4.08E4  
F: ITMS + c NSI ra Full m/z 653.9807@eld90.00 [100.0000-1972.0000]



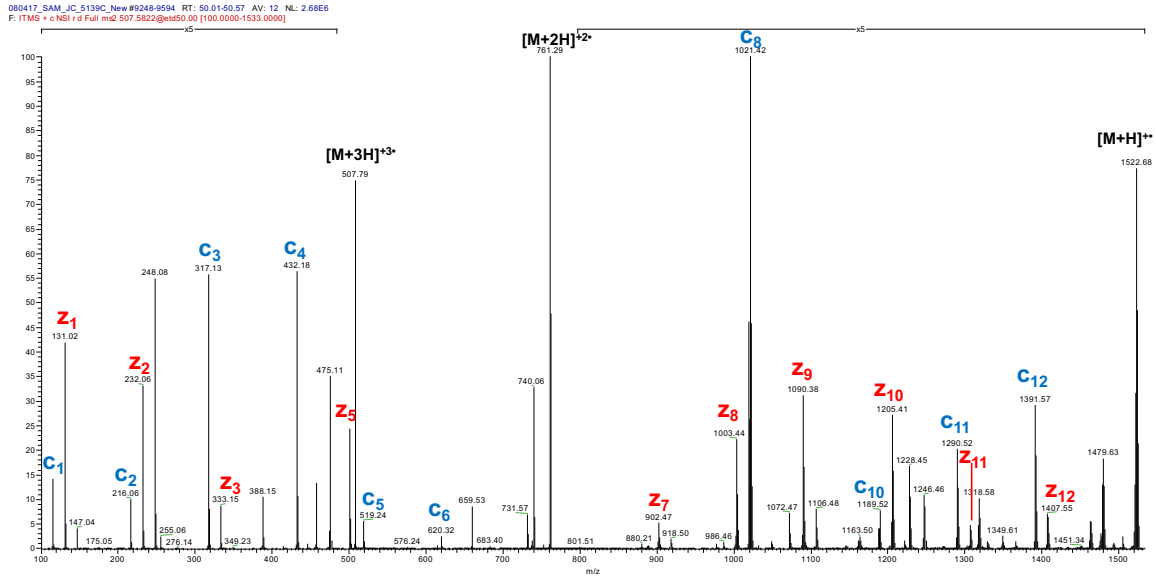
### GTTPSPVP(13-T)TSTT\*SAP.

Product formation and glycosite occupancy were determined using ETD fragmentation on an Orbitrap Fusion™ Tribrid™ Mass Spectrometer coupled to a Dionex Ultimate 3000 HPLC. The gradient profile was as follows (min/%B): 0:3, 3:5, 93:25, 103:42, 104:98, 109:98, 110:3, 185:3 with  $t_r = 105.2$  min.

MS (ESI)  $m/z$  ( $[M+2H]^{2+}$ ) calcd for C<sub>83</sub>H<sub>136</sub>N<sub>18</sub>O<sub>36</sub>: 980.4682, found: 980.4687 (+0.48 ppm).



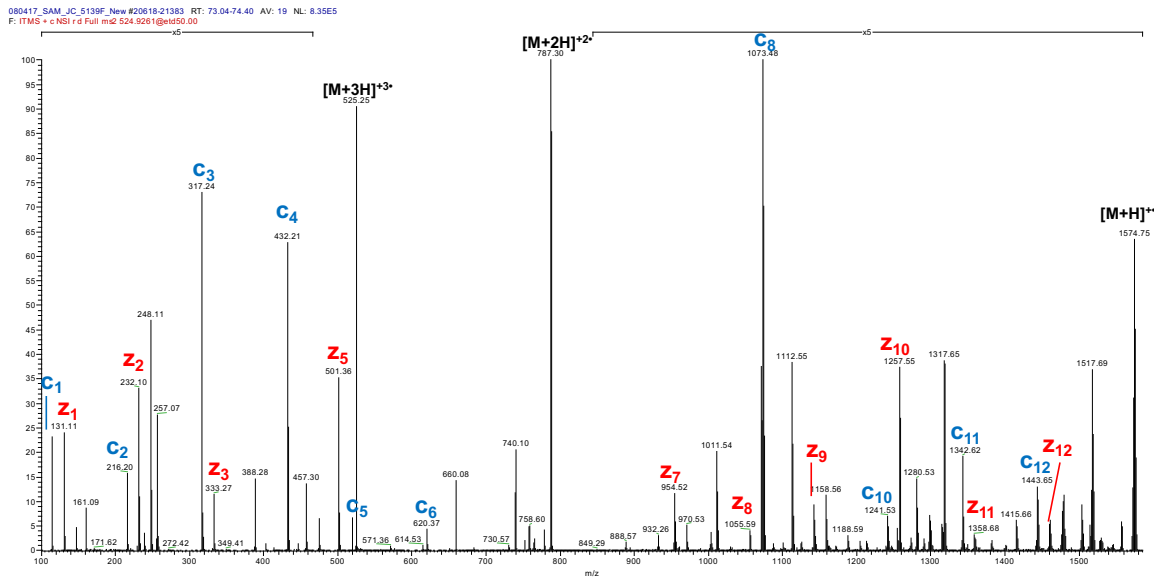
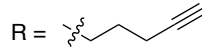
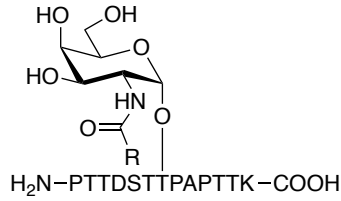
PTTDSSTT\*PAPT\*TK



### PTTDSSTT\*PAPT\*TK.

Product formation and glycosite occupancy were determined using ETD fragmentation on an Orbitrap Fusion™ Tribrid™ Mass Spectrometer coupled to a Dionex Ultimate 3000 HPLC. The gradient profile was as follows (min/%B): 0:3, 3:5, 93:20, 103:42, 104:98, 109:98, 110:3, 185:3 with  $t_r = 50.0$  min.

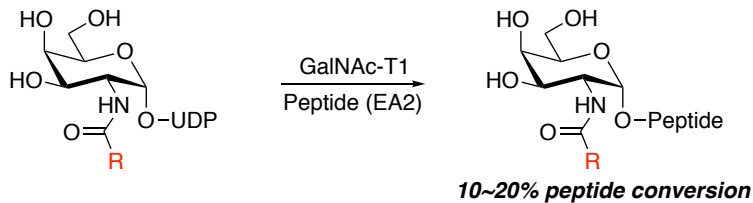
MS (ESI)  $m/z$  ( $[M+2H]^{2+}$ ) calcd for C<sub>63</sub>H<sub>107</sub>N<sub>15</sub>O<sub>28</sub>: 760.8705, found: 760.8708 (+0.39 ppm).



### PTTDSST(13-T)PAPTTK.

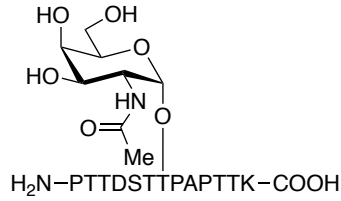
Product formation and glycosite occupancy were determined using ETD fragmentation on an Orbitrap Fusion™ Tribrid™ Mass Spectrometer coupled to a Dionex Ultimate 3000 HPLC. The gradient profile was as follows (min/%B): 0:3, 3:5, 93:20, 103:42, 104:98, 109:98, 110:3, 185:3 with  $t_r = 73.0$  min.

MS (ESI)  $m/z$  ( $[M+2H]^{2+}$ ) calcd for C<sub>67</sub>H<sub>111</sub>N<sub>15</sub>O<sub>28</sub>: 786.8862, found: 786.8867 (+0.70 ppm).

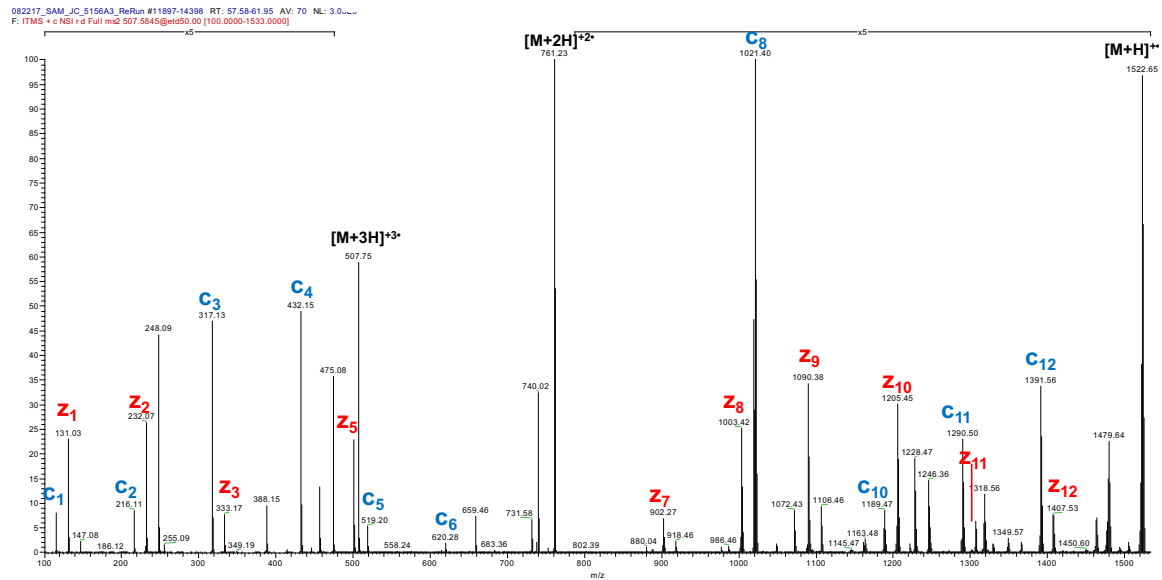


### Characterization of peptides glycosylated by GalNAc-T1.





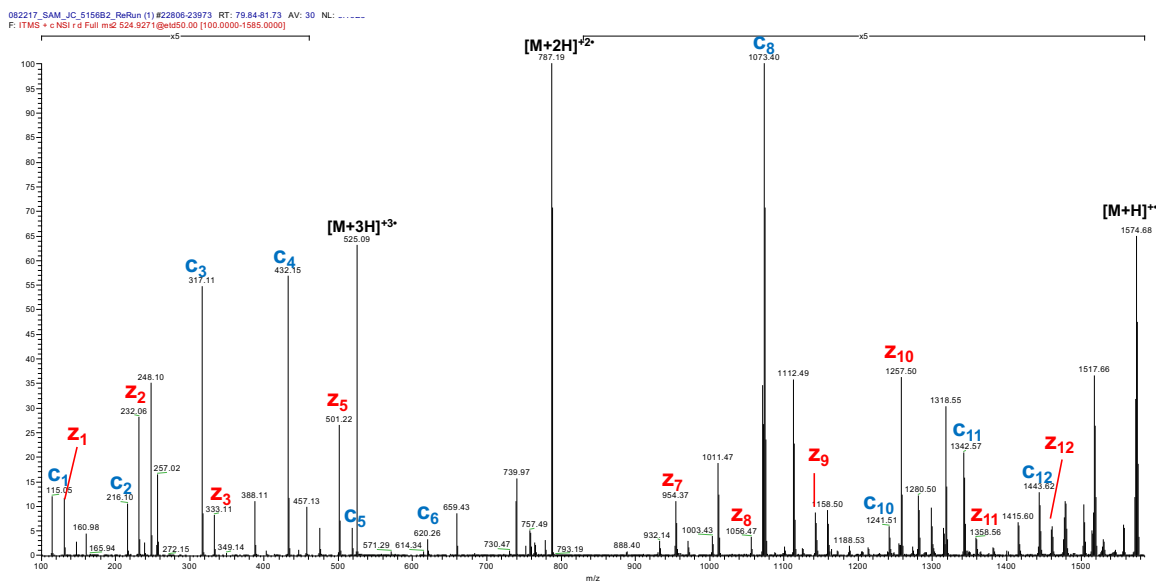
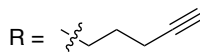
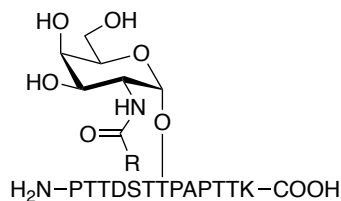
PTTDSIT\*PAPTTK



### PTTDSIT\*PAPTTK.

Product formation and glycosite occupancy were determined using ETD fragmentation on an Orbitrap Fusion™ Tribrid™ Mass Spectrometer coupled to a Dionex Ultimate 3000 HPLC. The gradient profile was as follows (min/%B): 0:3, 3:5, 93:20, 103:42, 104:98, 109:98, 110:3, 185:3 with  $t_r = 59.0$  min.

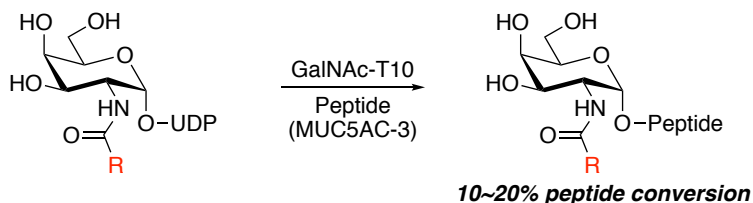
MS (ESI)  $m/z$  ( $[M+2H]^{2+}$ ) calcd for C<sub>63</sub>H<sub>107</sub>N<sub>15</sub>O<sub>28</sub>: 760.8705, found: 760.8700 (−0.66 ppm).



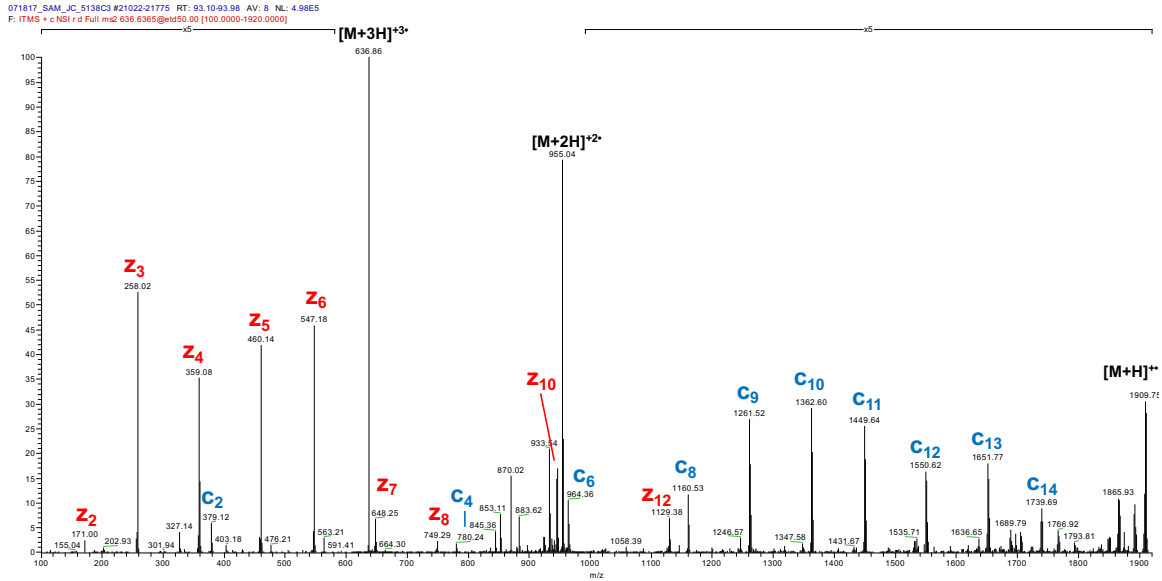
### PTTDSST(13-T)PAPTTK.

Product formation and glycosite occupancy were determined using ETD fragmentation on an Orbitrap Fusion™ Tribrid™ Mass Spectrometer coupled to a Dionex Ultimate 3000 HPLC. The gradient profile was as follows (min/%B): 0:3, 3:5, 93:20, 103:42, 104:98, 109:98, 110:3, 185:3 with  $t_r = 80.0$  min.

MS (ESI)  $m/z$  ( $[M+2H]^{2+}$ ) calcd for C<sub>67</sub>H<sub>111</sub>N<sub>15</sub>O<sub>28</sub>: 786.8862, found: 786.8859 (-0.32 ppm).



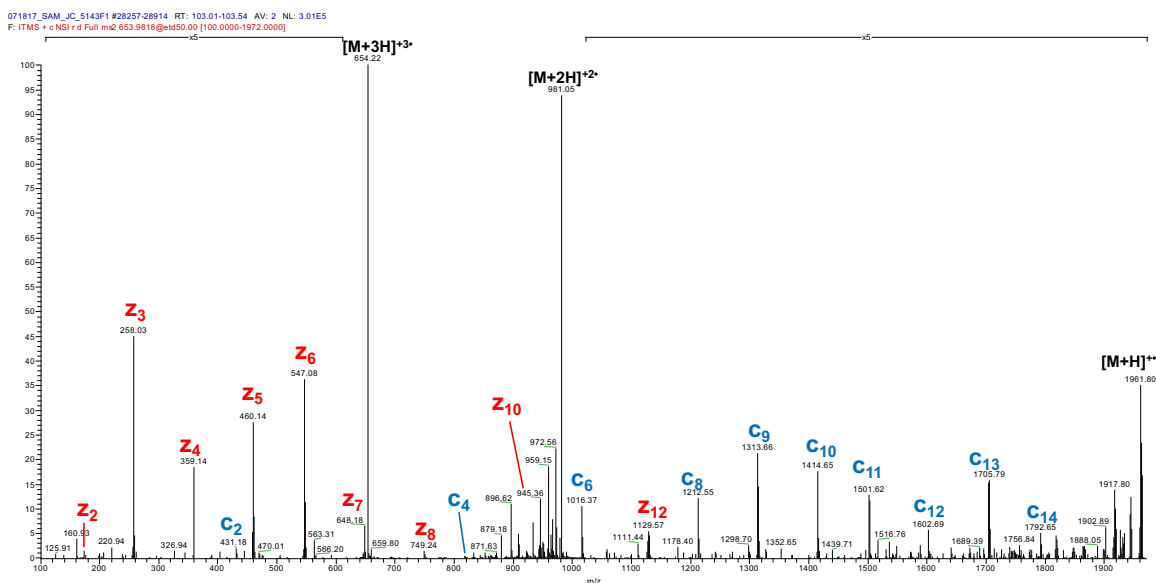
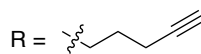
### Characterization of peptides glycosylated by GalNAc-T10.



### GT\*T\*PSPVPTTSTTSAP.

Product formation and glycosite occupancy were determined using ETD fragmentation on an Orbitrap Fusion™ Tribrid™ Mass Spectrometer coupled to a Dionex Ultimate 3000 HPLC. The gradient profile was as follows (min/%B): 0:3, 3:5, 93:25, 103:42, 104:98, 109:98, 110:3, 185:3 with  $t_r = 93.4$  min.

MS (ESI)  $m/z$  ( $[M+2H]^{2+}$ ) calcd for C<sub>79</sub>H<sub>132</sub>N<sub>18</sub>O<sub>36</sub>: 954.4526, found: 954.4554 (+2.95 ppm).



### G(13-T)\*PSPVPTTSTTSAP.

Product formation and glycosite occupancy were determined using ETD fragmentation on an Orbitrap Fusion™ Tribrid™ Mass Spectrometer coupled to a Dionex Ultimate 3000 HPLC. The gradient profile was as follows (min/%B): 0:3, 3:5, 93:25, 103:42, 104:98, 109:98, 110:3, 185:3 with  $t_r = 93.4$  min.

MS (ESI)  $m/z$  ( $[M+2H]^{2+}$ ) calcd for C<sub>83</sub>H<sub>136</sub>N<sub>18</sub>O<sub>36</sub>: 980.4682, found: 980.4716 (+3.44 ppm).

## VII. Preparation of GalNAc-Ts (Table S2; Figure S7)

### Secretion design

Soluble human GalNAc-T2 was generated by removing the N-terminal transmembrane domain and referencing published truncations.<sup>6,7</sup> For GalNAc-T1 and -T10 secretion constructs, N-terminal truncations were made by cleaving at the middle or end of the stem region that follows the transmembrane domain, as identified by online protein secondary structure predictors GlobPlot (2.3) and HMMTOP (2.0), and published truncations were also referenced for GalNAc-T1 and -T10.<sup>1,7-9</sup>

### **Cloning of truncated GalNAc-Ts.**

Full length human GalNAc-T2 (EBI accession number LC043140.1) in the plasmid pCMV-NTAP was a kind gift from Lawrence Tabak (National Institutes of Health, Bethesda, MD). A truncated version (aa E43-N571) was cloned into p3xFLAG-CMV-8 using primers (GalNAc-T2 coding sequence underlined)

GACAAGCTTGCGGCCGCGGAGGACTGGAATGAAATTG (fwd) and CGATGAATTCCTACTGCTGCAGGTTGAGC (rev) and a NotI/EcoRI restriction strategy. As the presence of a 3xFLAG tag in the secretion construct prevented elution from FLAG<sup>®</sup> affinity resin, truncated GalNAc-T2 was sub-cloned into pFLAG-myc-CMV-19 using a NotI/EcoRI restriction strategy that excluded the myc tag from the coding sequence. This construct contains an N-terminal preprotrypsin leader sequence and an ampicillin resistance cassette.

Secretion constructs for GalNAc-T1 (NCBI Genbank<sup>®</sup> accession number X85018, aa D35-F559) and GalNAc-T10 (NCBI Genbank<sup>®</sup> accession number AJ505950, aa P40-N603) were assembled by Golden Gate cloning using the gBlocks depicted in Table S2. BsaI restriction sites at the joint regions of each gBlock enabled assembly. Silent mutations were included to mask endogenous BsaI sites.

The Golden Gate reaction was carried out using 20 fmol of each gBlock and 2000 U T4 DNA ligase in 15  $\mu$ L T4 DNA ligase buffer, using 25 cycles of 37 °C (2 min), 16 °C (5 min); 60 °C (10 min); 80 °C (20 min); and 4 °C (hold). Following assembly, BsaI sites were introduced into the flanking regions of the T1 gene by PCR using the primers CACACCAGGTCTCAGGCGCGGATGAAAAAAGGAGAGAGGACTT (fwd) and CACACCAGGTCTCTAATTCTCAGAATATTTCTGGAAGGGTGACGT (rev). Amplicons were digested with BsaI to give NotI and EcoRI overhangs, and cloned into pFLAG-myc-CMV-19 while excluding the myc tag from the coding sequence.

### **Generation of GalNAc-T mutants.**

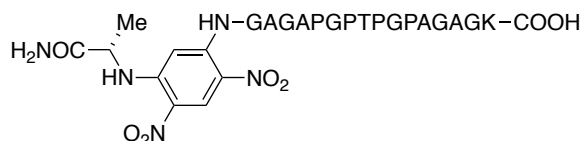
Point mutations were introduced into wild-type GalNAc-T2 (WT-T2) and WT-T1 by site-directed mutagenesis using a strategy according to a literature procedure.<sup>10</sup> The primer pairs used to generate GalNAc-T2 mutants (mismatch underlined) were CACCCATCGCCGATGTCATTAATATGGACAAC (fwd) and GACATCGGCGATGGGTGACACAACCCGAGTC (rev) for T2(I253A), GCTGGTGGGGCCTTTGTGATGGATAAGTTC (fwd) and CATCACAAAGGCCCCACCAGCAATCATGGGG (rev) for T2(L310A), GGACACGTGGCCCGGAAGCAGCACCCCTACACGTTC (fwd) and GCTTCCGGGCACGTGTCCACACGGCTGCAC (rev) for T2(F361A), and GACACGTGTCCCGGAAGCAGCAC (fwd) and CTTCCGGGACACGTGTCCAC (rev) for T2(F361S). The F361 mutants were generated from full-length GalNAc-T2 in pCMV-NTAP, and truncated versions were cloned and sub-cloned as described for the wild-type enzyme in the previous section. Primer pairs for GalNAc-T1 mutants (mismatch underlined) were CCATCGCCGATGTGATCAGTGATGATAC (fwd) and ACATCGGCGATGGGACACACC (rev) for T1(I238A), and GAGGCGCCTTTTCAATAGACAGAGATTACTTTC (fwd) and GAAAAGGCGCCTCCTGCCATGG (rev) for T1(L295A). The GalNAc-T10 double

mutant was assembled using a modified gBlock during Golden Gate assembly (Table S2).

### Protein expression.

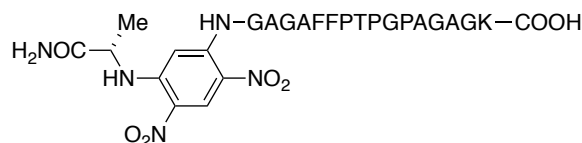
Truncated GalNAc-Ts were expressed in HEK-293T cells and purified by FLAG affinity chromatography. Briefly, cells were grown in Dulbecco's Modified Eagle Medium with 10% (v/v) fetal bovine serum in 15-cm dishes and transfected with expression plasmids using TransIT<sup>®</sup>-293 according to the manufacturer's instructions and using 37.5  $\mu$ g plasmid DNA per dish. The medium was changed after 24 h, and protein expression was allowed to continue for another 24 h. The supernatant was collected and centrifuged for 15 min at 3650 x g at 4 °C. The clarified supernatant was treated with cComplete<sup>™</sup> mini EDTA-free protease inhibitor and loaded on a column packed with anti-FLAG<sup>®</sup> M2 agarose resin (1.25 mL) pre-conditioned according to the manufacturer's instructions. The resin was washed with Tris buffered saline (TBS, 25 mM Tris-HCl, 150 mM NaCl, pH = 7.4; 2 x 10 mL). Protein was eluted by 3xFLAG<sup>®</sup> peptide (100  $\mu$ g/mL) in TBS (25 mM Tris-HCl, 150 mM NaCl, pH = 7.4; 5 mL). Glycerol was added to the pooled elution fractions to a final concentration of 25% (v/v). Proteins were aliquoted and stored at -80 °C. Protein quantification was performed by densitometric analysis of bands on SDS-PAGE gels stained with Colloidal Blue Staining Kit and comparison to known standards of human serum albumin (Figure S7A). Immunodecoration after Western blot was performed using a murine anti-FLAG<sup>®</sup> antibody (Figure S7B).

## VIII. Characterization of Chromophore-Labeled Peptides (Figure 2A)



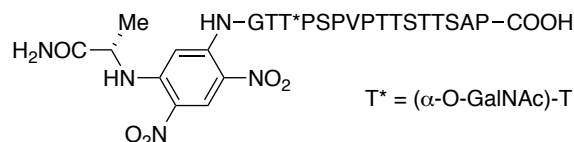
**Peptide-1.** The peptide was purified by preparative HPLC on C-18 silica gel (20→60% acetonitrile/water; 0.1% trifluoroacetic acid).

MS (ESI)  $m/z$  ( $[\text{M}+2\text{H}]^{2+}$ ) calcd for  $\text{C}_{63}\text{H}_{97}\text{N}_{21}\text{O}_{23}$ : 757.8533, found: 757.8524 (-1.20 ppm).



**Peptide-2.** The peptide was purified by preparative HPLC on C-18 silica gel (30→40% acetonitrile/water; 0.1% trifluoroacetic acid).

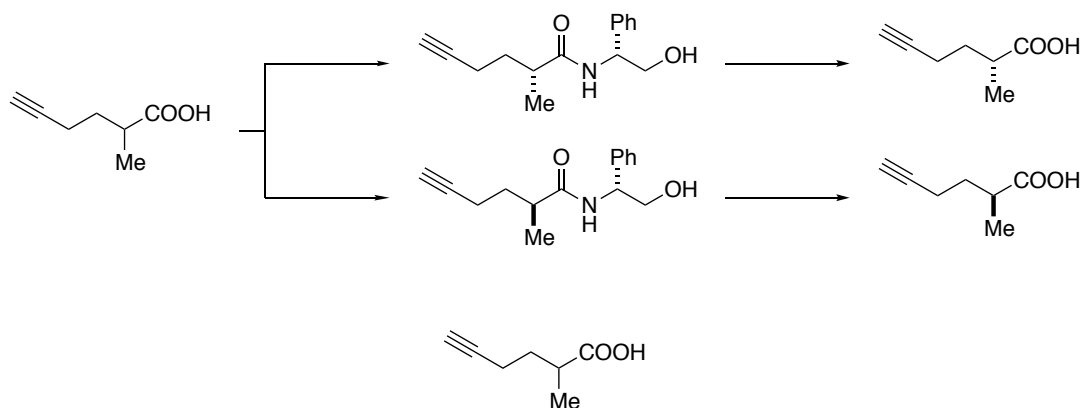
MS (ESI)  $m/z$  ( $[\text{M}+2\text{H}]^{2+}$ ) calcd for  $\text{C}_{74}\text{H}_{105}\text{N}_{21}\text{O}_{23}$ : 827.8846, found: 827.8828 (-2.19 ppm).



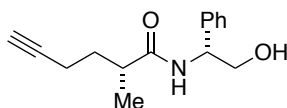
**Peptide-3.** The peptide containing a protected sugar was purified by preparative HPLC on C-18 silica gel (30→40% acetonitrile/water). The title compound was purified by preparative HPLC on C-18 silica gel (5→50% acetonitrile/water; 0.1% trifluoroacetic acid).

MS (ESI)  $m/z$  ( $[M+2H]^{2+}$ ) calcd for  $C_{80}H_{127}N_{21}O_{36}$ : 978.9376, found: 978.9376 (−0.03 ppm).

### IX. Preparation of UDP-Sugars (Figures 2B and 2C)



**Methylhex-5-ynoic acid.** LDA was prepared by the dropwise addition of *n*-BuLi (2.5 M in hexanes; 14.2 mL, 36 mmol) to a solution of *i*-Pr<sub>2</sub>NH (6.43 mL, 45.9 mmol) in THF (24.0 mL) in a 250-mL round-bottom flask at −78 °C. The reaction mixture was stirred at 0 °C for 15 min, and then it was cooled to −20 °C. A solution of propanoic acid (1.33 mL, 17.8 mmol) in HMPA (3.00 mL) was added dropwise over 10 min to the LDA solution at −20 °C. The mixture was stirred at r.t. for 30 min, and then it was cooled to 0 °C. Next, a solution of (4-bromobut-1-yn-1-yl)trimethylsilane<sup>11</sup> (3.04 g, 14.8 mmol) in THF (5.90 mL) was added. The resulting mixture was allowed to warm to r.t., and it was stirred for 2 h. The reaction was quenched by the addition of water (100 mL). The aqueous layer was rinsed with ethyl acetate (50 mL), acidified using HCl (2 M), and extracted with ethyl acetate (3 x 50 mL). The extracted organic layers were combined, dried over Na<sub>2</sub>SO<sub>4</sub>, and concentrated. The acid was used without further purification.



**(*R*)-*N*-((*R*)-2-Hydroxy-1-phenylethyl)-2-methylhex-5-ynamide.** The title compound was prepared from (*R*)-2-amino-2-phenylethan-1-ol (1.37 g, 10.0 mmol) and methylhex-5-ynoic acid (1.26 g, 10.0 mmol) according to a literature procedure.<sup>12</sup> The product was purified by column chromatography on silica gel (15%→100% ethyl acetate/hexanes)

and then on C-18 silica gel (5%→100% acetonitrile/water; 0.1% trifluoroacetic acid): 495 mg (21% over two steps). White solid.

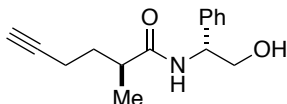
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.37–7.32 (m, 2H), 7.31–7.25 (m, 3H), 6.41 (d,  $J$  = 6.8 Hz, 1H), 5.05 (ddd,  $J$  = 7.0, 5.1, 5.1 Hz, 1H), 3.84 (d,  $J$  = 5.1 Hz, 2H), 3.08 (br s, 1H), 2.55–2.46 (m, 1H), 2.33–2.20 (m, 2H), 2.00 (t,  $J$  = 2.6 Hz, 1H), 1.91–1.82 (m, 1H), 1.65–1.56 (m, 1H), 1.14 (d,  $J$  = 6.9 Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  176.5, 139.2, 128.7, 127.6, 126.6, 83.8, 69.3, 66.1, 55.6, 39.8, 32.3, 17.6, 16.4.

FT-IR (neat) 3295, 3071, 3031, 2937, 2877, 1640, 1545, 1491, 1451, 1390, 1371, 1280, 1245, 1200, 1179, 1116, 1087, 1072, 1042, 1002, 939, 882, 841, 749, 697, 636, 614, 561, 524, 500  $\text{cm}^{-1}$ .

HRMS (ESI)  $m/z$  ( $[\text{M}+\text{Na}]^+$ ) calcd for  $\text{C}_{15}\text{H}_{19}\text{NNaO}_2$ : 268.1313, found: 268.1309.

$R_f$  = 0.38 (60% ethyl acetate/hexanes).



**(S)-N-((R)-2-Hydroxy-1-phenylethyl)-2-methylhex-5-ynamide.** The title compound was prepared from (*R*)-2-amino-2-phenylethan-1-ol (1.37 g, 10.0 mmol) and methylhex-5-ynoic acid (1.26 g, 10.0 mmol) according to a literature procedure.<sup>12</sup> The product was purified by column chromatography on silica gel (15%→100% ethyl acetate/hexanes) and then on C-18 silica gel (5%→100% acetonitrile/water; 0.1% trifluoroacetic acid): 469 mg (19% over two steps). White solid.

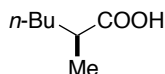
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.38–7.33 (m, 2H), 7.32–7.27 (m, 3H), 6.43 (d,  $J$  = 7.4 Hz, 1H), 5.06 (ddd,  $J$  = 7.0, 5.0, 5.0 Hz, 1H), 3.89–3.80 (m, 2H), 2.82 (br s, 1H), 2.58–2.50 (m, 1H), 2.21 (dddd,  $J$  = 17.1, 6.1, 6.1, 2.6 Hz, 1H), 2.08 (dddd,  $J$  = 17.7, 8.9, 6.3, 2.7 Hz, 1H), 1.97 (t,  $J$  = 2.6 Hz, 1H), 1.86–1.78 (m, 1H), 1.62–1.53 (m, 1H), 1.19 (d,  $J$  = 6.8 Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  176.5, 139.4, 128.8, 127.8, 126.7, 83.7, 69.4, 66.2, 55.6, 39.8, 32.5, 17.8, 16.5.

FT-IR (neat) 3271, 3069, 3033, 2966, 2947, 2928, 2876, 1642, 1538, 1492, 1472, 1453, 1385, 1367, 1347, 1292, 1270, 1252, 1218, 1195, 1184, 1115, 1095, 1067, 1046, 1002, 941, 910, 899, 882, 839, 753, 696, 679, 644, 556, 529, 502, 441  $\text{cm}^{-1}$ .

HRMS (ESI)  $m/z$  ( $[\text{M}+\text{Na}]^+$ ) calcd for  $\text{C}_{15}\text{H}_{19}\text{NNaO}_2$ : 268.1313, found: 268.1308.

$R_f$  = 0.26 (60% ethyl acetate/hexanes).



**Assignment of absolute stereochemistry of (*S*)-2-methylhexanoic acid [49642-51-5].**

A mixture of *N*-((*R*)-2-Hydroxy-1-phenylethyl)-2-methylhex-5-ynamide (80.0 mg, 0.326 mmol;  $R_f$  = 0.26 (60% ethyl acetate/hexanes)) and Pd/C (10 wt%; 14.8 mg) in MeOH (6.0 mL) in a 25-mL round-bottom flask was stirred under atmospheric pressure of  $\text{H}_2$  at r.t. for 12 h, and then the reaction mixture was filtered through a pad of Celite® and concentrated. The compound was hydrolyzed to obtain 2-methylhexanoic acid according



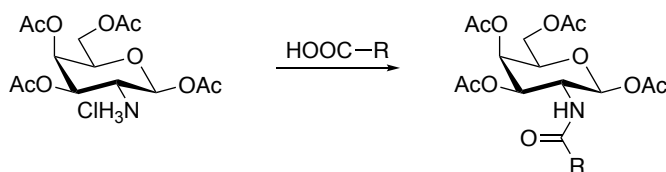
to a literature procedure.<sup>12</sup> The product was purified by column chromatography (10%→78% diethyl ether/pentane): 39.8 mg (94% over two steps). Colorless oil.

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 2.45 (h, *J* = 7.0 Hz, 1H), 1.73–1.64 (m, 1H), 1.48–1.38 (m, 1H), 1.36–1.27 (m, 4H), 1.17 (d, *J* = 6.9 Hz, 3H), 0.89 (t, *J* = 6.9 Hz, 3H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 183.9, 39.6, 33.4, 29.4, 22.7, 17.0, 14.1.

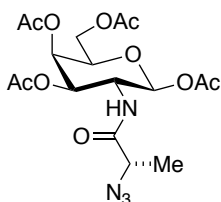
$[\alpha]_D^{23} = +18.1^\circ$  (*c* = 1.02, CHCl<sub>3</sub>).

The absolute stereochemistry was assigned as (*S*) by comparison with reported optical rotations (lit.<sup>13</sup>  $[\alpha]_D^{20} = +20.6^\circ$  (*c* = 0.5, CHCl<sub>3</sub>; *S* enantiomer); lit.<sup>14</sup>  $[\alpha]_D^{20} = +18.1^\circ$  (*c* = 0.84, CHCl<sub>3</sub>; *S* enantiomer)).



### Representative experimental procedure for the preparation of peracetylated *N*-acetyl-β-D-galactosamine derivatives (Route 1).

1,3,4,6-Tetra-*O*-acetyl-2-amino-2-deoxy-β-D-galactopyranose hydrochloride<sup>15</sup> and azido acids<sup>16</sup> were prepared according to literature procedures. A mixture of the sugar (192 mg, 0.500 mmol), the acid (0.500 mmol), and Hünig's base (0.261 mL, 1.50 mmol) in DMF (4.00 mL) in a 25-mL round-bottom flask was cooled to 0 °C. COMU<sup>®</sup> was added, and the reaction mixture was stirred at 0 °C for 1 h. The solution was allowed to warm to r.t. and stirred for 3 h. The mixture was diluted by the addition of ethyl acetate (50 mL), rinsed with HCl (1 M; 2 x 10 mL), saturated aqueous NaHCO<sub>3</sub> (2 x 10 mL), and brine (20 mL), dried over Na<sub>2</sub>SO<sub>4</sub>, and concentrated. The product was purified by column chromatography.



### 1,3,4,6-Tetra-*O*-acetyl-2-((*S*)-2-azidopropanamido)-2-deoxy-β-D-galactopyranose.

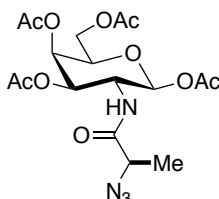
The title compound was prepared from 1,3,4,6-tetra-*O*-acetyl-2-amino-2-deoxy-β-D-galactopyranose hydrochloride (384 mg, 1.00 mmol) and (*S*)-2-azidopropanoic acid (115 mg, 1.00 mmol). The product was purified by column chromatography (15%→100% ethyl acetate/hexanes): 397 mg (89%). White solid.

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 6.77 (d, *J* = 9.5 Hz, 1H), 5.76 (d, *J* = 8.7 Hz, 1H), 5.30 (d, *J* = 3.3 Hz, 1H), 5.20 (dd, *J* = 11.3, 3.4 Hz, 1H), 4.27 (ddd, *J* = 11.3, 9.1, 9.1 Hz, 1H), 4.09–3.99 (m, 3H), 3.90 (q, *J* = 7.0 Hz, 1H), 2.06 (s, 3H), 2.01 (s, 3H), 1.94 (s, 3H), 1.90 (s, 3H), 1.32 (d, *J* = 7.0 Hz, 3H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 170.7, 170.5, 170.4, 170.2, 169.3, 92.5, 71.7, 69.7, 66.4, 61.5, 59.1, 49.6, 20.6, 20.5, 20.5, 20.4, 17.1.

FT-IR (neat) 3337, 2975, 2956, 2934, 2109, 1745, 1665, 1530, 1435, 1369, 1321, 1268, 1215, 1162, 1151, 1068, 1036, 972, 946, 915, 903, 731, 703, 668, 645, 628, 601, 556, 539, 495, 480  $\text{cm}^{-1}$ .

HRMS (ESI)  $m/z$  ( $[\text{M}+\text{Na}]^+$ ) calcd for  $\text{C}_{17}\text{H}_{24}\text{N}_4\text{NaO}_{10}$ : 467.1390, found: 467.1383.



**1,3,4,6-Tetra-*O*-acetyl-2-((*R*)-2-azidopropanamido)-2-deoxy- $\beta$ -D-galactopyranose.**

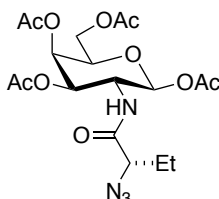
The title compound was prepared from 1,3,4,6-tetra-*O*-acetyl-2-amino-2-deoxy- $\beta$ -D-galactopyranose hydrochloride (576 mg, 1.50 mmol) and (*R*)-2-azidopropanoic acid (173 mg, 1.50 mmol). The product was purified by column chromatography (12% $\rightarrow$ 100% ethyl acetate/hexanes): 458 mg (69%). White solid.

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  6.59 (d,  $J = 9.5$  Hz, 1H), 5.81 (d,  $J = 8.8$  Hz, 1H), 5.36 (d,  $J = 3.1$  Hz, 1H), 5.22 (dd,  $J = 11.2, 3.3$  Hz, 1H), 4.35 (ddd,  $J = 11.2, 9.2, 9.2$  Hz, 1H), 4.15–4.05 (m, 3H), 3.98 (q,  $J = 7.0$  Hz, 1H), 2.14 (s, 3H), 2.09 (s, 3H), 2.01 (s, 3H), 1.97 (s, 3H), 1.41 (d,  $J = 7.0$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  170.7, 170.6, 170.5, 170.3, 169.5, 92.5, 71.9, 70.2, 66.5, 61.5, 59.2, 49.9, 20.9, 20.7, 20.6, 17.2.

FT-IR (neat) 3311, 2983, 2940, 2110, 1743, 1685, 1531, 1434, 1368, 1212, 1161, 1116, 1069, 1039, 955, 917, 904, 865, 733, 647, 601, 561, 537  $\text{cm}^{-1}$ .

HRMS (ESI)  $m/z$  ( $[\text{M}+\text{Na}]^+$ ) calcd for  $\text{C}_{17}\text{H}_{24}\text{N}_4\text{NaO}_{10}$ : 467.1390, found: 467.1381.



**1,3,4,6-Tetra-*O*-acetyl-2-((*S*)-2-azidobutanamido)-2-deoxy- $\beta$ -D-galactopyranose.**

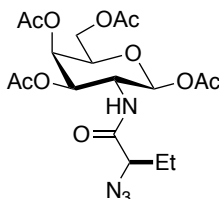
The title compound was prepared from 1,3,4,6-tetra-*O*-acetyl-2-amino-2-deoxy- $\beta$ -D-galactopyranose hydrochloride (544 mg, 1.42 mmol) and (*S*)-2-azidobutanoic acid (183 mg, 1.42 mmol). The product was purified by column chromatography (12% $\rightarrow$ 100% ethyl acetate/hexanes): 435 mg (67%). White solid.

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  6.49 (d,  $J = 9.4$  Hz, 1H), 5.78 (d,  $J = 8.8$  Hz, 1H), 5.39 (dd,  $J = 3.3, 0.7$  Hz, 1H), 5.21 (dd,  $J = 11.3, 3.3$  Hz, 1H), 4.38 (ddd,  $J = 11.3, 9.1, 9.1$  Hz, 1H), 4.17–4.10 (m, 2H), 4.09–4.04 (m, 1H), 3.91 (dd,  $J = 6.6, 4.9$  Hz, 1H), 2.15 (s, 3H), 2.09 (s, 3H), 2.03 (s, 3H), 1.99 (s, 3H), 1.90–1.74 (m, 2H), 0.92 (t,  $J = 7.4$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  170.6, 170.6, 170.3, 167.0, 169.4, 92.6, 71.9, 70.1, 66.4, 65.5, 61.4, 49.8, 25.3, 20.9, 20.8, 20.8, 20.7, 9.4.

FT-IR (neat) 3319, 2973, 2941, 2881, 2104, 1745, 1669, 1532, 1435, 1368, 1214, 1162, 1116, 1071, 1040, 941, 899, 736, 648, 601, 561, 538  $\text{cm}^{-1}$ .

HRMS (ESI)  $m/z$  ( $[M+Na]^+$ ) calcd for  $C_{18}H_{26}N_4NaO_{10}$ : 481.1547, found: 481.1535.



**1,3,4,6-Tetra-*O*-acetyl-2-((*R*)-2-azidobutanamido)-2-deoxy- $\beta$ -D-galactopyranose.**

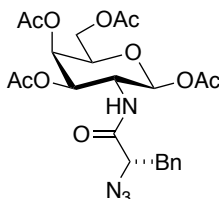
The title compound was prepared from 1,3,4,6-tetra-*O*-acetyl-2-amino-2-deoxy- $\beta$ -D-galactopyranose hydrochloride (518 mg, 1.35 mmol) and (*R*)-2-azidobutanoic acid (174 mg, 1.35 mmol). The product was purified by column chromatography (12% $\rightarrow$ 100% ethyl acetate/hexanes): 417 mg (68%). White solid.

$^1H$  NMR (500 MHz,  $CDCl_3$ )  $\delta$  6.47 (d,  $J$  = 9.5 Hz, 1H), 5.80 (d,  $J$  = 8.8 Hz, 1H), 5.38 (dd,  $J$  = 3.3, 0.7 Hz, 1H), 5.19 (dd,  $J$  = 11.3, 3.3 Hz, 1H), 4.40 (ddd,  $J$  = 11.3, 9.2, 9.2 Hz, 1H), 4.18–4.11 (m, 2H), 4.07–4.05 (m, 1H), 3.94 (dd,  $J$  = 6.2, 5.0 Hz, 1H), 2.16 (s, 3H), 2.11 (s, 3H), 2.04 (s, 3H), 1.98 (s, 3H), 1.91–1.78 (m, 2H), 0.92 (t,  $J$  = 7.4 Hz, 3H).

$^{13}C$  NMR (126 MHz,  $CDCl_3$ )  $\delta$  170.6, 170.5, 170.3, 169.8, 169.5, 92.6, 71.9, 70.2, 66.4, 65.3, 61.4, 49.8, 25.2, 21.0, 20.8, 20.7, 9.3.

FT-IR (neat) 3317, 2973, 2937, 2881, 2104, 1746, 1671, 1532, 1435, 1368, 1215, 1161, 1117, 1071, 1041, 937, 901, 863, 736, 676, 630, 601, 561, 537  $cm^{-1}$ .

HRMS (ESI)  $m/z$  ( $[M+Na]^+$ ) calcd for  $C_{18}H_{26}N_4NaO_{10}$ : 481.1547, found: 481.1550.



**1,3,4,6-Tetra-*O*-acetyl-2-((*S*)-2-azido-3-phenylpropanamido)-2-deoxy- $\beta$ -D-galactopyranose.**

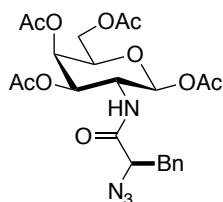
The title compound was prepared from 1,3,4,6-tetra-*O*-acetyl-2-amino-2-deoxy- $\beta$ -D-galactopyranose hydrochloride (384 mg, 1.00 mmol) and (*S*)-2-azido-3-phenylpropanoic acid (191 mg, 1.00 mmol). The product was purified by column chromatography (1% $\rightarrow$ 10% acetone/ $CH_2Cl_2$ ): 456 mg (88%). White solid.

$^1H$  NMR (500 MHz,  $CDCl_3$ )  $\delta$  7.32–7.29 (m, 2H), 7.28–7.25 (m, 1H), 7.25–7.22 (m, 2H), 6.48 (d,  $J$  = 9.3 Hz, 1H), 5.79 (d,  $J$  = 8.8 Hz, 1H), 5.39 (dd,  $J$  = 3.2, 0.7 Hz, 1H), 5.21 (dd,  $J$  = 11.3, 3.3 Hz, 1H), 4.37 (ddd,  $J$  = 11.3, 9.0, 9.0 Hz, 1H), 4.18–4.05 (m, 4H), 3.27 (dd,  $J$  = 14.0, 4.3 Hz, 1H), 2.81 (dd,  $J$  = 14.1, 9.1 Hz, 1H), 2.17 (s, 3H), 2.10 (s, 3H), 2.03 (s, 3H), 1.97 (s, 3H).

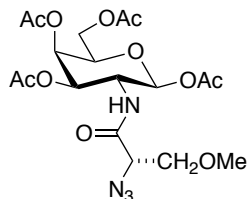
$^{13}C$  NMR (126 MHz,  $CDCl_3$ )  $\delta$  170.6, 170.6, 170.3, 169.5, 169.5, 136.1, 129.4, 128.9, 127.4, 92.7, 71.9, 70.0, 66.4, 65.9, 61.4, 50.2, 38.6, 21.0, 20.8, 20.7.

FT-IR (neat) 3327, 3084, 3065, 3031, 2929, 2110, 1745, 1675, 1531, 1498, 1454, 1433, 1368, 1301, 1216, 1162, 1115, 1073, 1040, 947, 913, 733, 702, 595, 556, 538  $cm^{-1}$ .

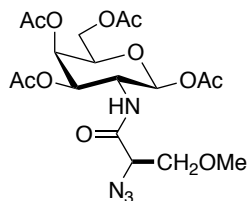
HRMS (ESI)  $m/z$  ( $[M+Na]^+$ ) calcd for  $C_{23}H_{28}N_4NaO_{10}$ : 543.1703, found: 543.1696.



**1,3,4,6-Tetra-*O*-acetyl-2-((*R*)-2-azido-3-phenylpropanamido)-2-deoxy- $\beta$ -D-galactopyranose.** The title compound was prepared from 1,3,4,6-tetra-*O*-acetyl-2-amino-2-deoxy- $\beta$ -D-galactopyranose hydrochloride (192 mg, 0.50 mmol) and (*R*)-2-azido-3-phenylpropanoic acid (95.6 mg, 0.50 mmol). The product was purified by column chromatography (1% $\rightarrow$ 10% acetone/CH<sub>2</sub>Cl<sub>2</sub>): 136 mg (52%). White solid. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.33–7.26 (m, 3H), 7.26–7.22 (m, 2H), 6.36 (d, *J* = 9.3 Hz, 1H), 5.80 (d, *J* = 8.8 Hz, 1H), 5.39 (dd, *J* = 3.2, 0.7 Hz, 1H), 5.20 (dd, *J* = 11.3, 3.4 Hz, 1H), 4.34 (ddd, *J* = 11.3, 9.1, 9.1 Hz, 1H), 4.19–4.09 (m, 3H), 4.05 (ddd, *J* = 6.5, 6.5, 1.0 Hz, 1H), 3.30 (dd, *J* = 14.1, 4.2 Hz, 1H), 2.82 (dd, *J* = 14.1, 9.0 Hz, 1H), 2.18 (s, 3H), 2.10 (s, 3H), 2.04 (s, 3H), 1.98 (s, 3H). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  170.5, 170.3, 169.4, 169.4, 136.1, 129.5, 128.9, 127.5, 92.6, 72.0, 70.1, 66.4, 65.8, 61.4, 50.2, 38.6, 21.0, 20.8, 20.8, 20.7. FT-IR (neat) 3327, 2952, 2924, 2854, 2113, 1746, 1685, 1532, 1455, 1435, 1368, 1300, 1217, 1160, 1117, 1074, 1041, 949, 919, 908, 735, 702, 601 cm<sup>-1</sup>. HRMS (ESI) *m/z* ([M+Na]<sup>+</sup>) calcd for C<sub>23</sub>H<sub>28</sub>N<sub>4</sub>NaO<sub>10</sub>: 543.1703, found: 543.1706.



**1,3,4,6-Tetra-*O*-acetyl-2-((*S*)-2-azido-3-methoxypropanamido)-2-deoxy- $\beta$ -D-galactopyranose.** The title compound was prepared from 1,3,4,6-tetra-*O*-acetyl-2-amino-2-deoxy- $\beta$ -D-galactopyranose hydrochloride (510 mg, 1.33 mmol) and (*S*)-2-azido-3-methoxypropanoic acid (193 mg, 1.33 mmol). The product was purified by column chromatography (15% $\rightarrow$ 100% ethyl acetate/hexanes): 453 mg (72%). White solid. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  6.44 (d, *J* = 9.5 Hz, 1H), 5.78 (d, *J* = 8.7 Hz, 1H), 5.40 (dd, *J* = 3.2, 0.7 Hz, 1H), 5.18 (dd, *J* = 11.3, 3.3 Hz, 1H), 4.36 (ddd, *J* = 11.3, 9.1, 9.1 Hz, 1H), 4.19–4.09 (m, 3H), 4.04 (ddd, *J* = 6.5, 6.5, 0.8 Hz, 1H), 3.80–3.69 (m, 2H), 3.38 (s, 3H), 2.17 (s, 3H), 2.11 (s, 3H), 2.05 (s, 3H), 2.02 (s, 3H). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  170.5, 170.5, 170.2, 169.3, 167.9, 92.7, 72.4, 72.0, 69.9, 66.4, 63.3, 61.4, 59.4, 50.1, 20.9, 20.8, 20.8, 20.7. FT-IR (neat) 3337, 2957, 2921, 2852, 2109, 1747, 1675, 1534, 1457, 1435, 1369, 1305, 1219, 1162, 1116, 1073, 1041, 950, 923, 900, 849, 800, 602, 562 cm<sup>-1</sup>. HRMS (ESI) *m/z* ([M+Na]<sup>+</sup>) calcd for C<sub>18</sub>H<sub>26</sub>N<sub>4</sub>NaO<sub>11</sub>: 497.1496, found: 497.1485.



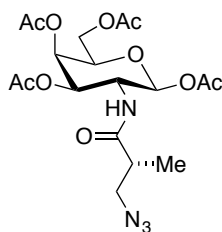
**1,3,4,6-Tetra-*O*-acetyl-2-((*R*)-2-azido-3-methoxypropanamido)-2-deoxy- $\beta$ -D-galactopyranose.** The title compound was prepared from 1,3,4,6-tetra-*O*-acetyl-2-amino-2-deoxy- $\beta$ -D-galactopyranose hydrochloride (510 mg, 1.33 mmol) and (*R*)-2-azido-3-methoxypropanoic acid (193 mg, 1.33 mmol). The product was purified by column chromatography (15% $\rightarrow$ 100% ethyl acetate/hexanes): 459 mg (73%). White solid.

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  6.43 (d,  $J = 9.4$  Hz, 1H), 5.78 (d,  $J = 8.8$  Hz, 1H), 5.39 (dd,  $J = 3.3, 0.6$  Hz, 1H), 5.19 (dd,  $J = 11.3, 3.3$  Hz, 1H), 4.37 (ddd,  $J = 11.2, 9.1, 9.1$  Hz, 1H), 4.19–4.09 (m, 3H), 4.04 (ddd,  $J = 6.6, 6.6, 0.9$  Hz, 1H), 3.81–3.65 (m, 2H), 3.37 (s, 3H), 2.17 (s, 3H), 2.13 (s, 3H), 2.05 (s, 3H), 2.00 (s, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  170.5, 170.4, 170.2, 169.4, 167.9, 92.6, 72.4, 72.1, 70.1, 66.4, 63.1, 61.3, 59.4, 50.1, 21.0, 20.8, 20.7.

FT-IR (neat) 3340, 2952, 2921, 2850, 2108, 1746, 1688, 1533, 1457, 1436, 1369, 1218, 1162, 1115, 1072, 1041, 955, 925, 901, 847, 803, 600, 557  $\text{cm}^{-1}$ .

HRMS (ESI)  $m/z$  ( $[\text{M}+\text{Na}]^+$ ) calcd for  $\text{C}_{18}\text{H}_{26}\text{N}_4\text{NaO}_{11}$ : 497.1496, found: 497.1493.



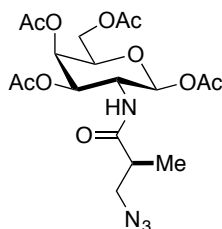
**1,3,4,6-Tetra-*O*-acetyl-2-((*R*)-3-azido-2-methylpropanamido)-2-deoxy- $\beta$ -D-galactopyranose.** The title compound was prepared from 1,3,4,6-tetra-*O*-acetyl-2-amino-2-deoxy- $\beta$ -D-galactopyranose hydrochloride (384 mg, 1.00 mmol) and (*R*)-3-azido-2-methylpropanoic acid<sup>17</sup> (129 mg, 1.00 mmol). The product was purified by column chromatography (12% $\rightarrow$ 100% ethyl acetate/hexanes): 318 mg (69%). White solid.

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  6.08 (d,  $J = 9.5$  Hz, 1H), 5.76 (d,  $J = 8.8$  Hz, 1H), 5.35 (d,  $J = 2.8$  Hz, 1H), 5.15 (dd,  $J = 11.3, 3.4$  Hz, 1H), 4.44 (ddd,  $J = 11.3, 9.2, 9.2$  Hz, 1H), 4.16–4.03 (m, 3H), 3.48 (dd,  $J = 12.1, 9.1$  Hz, 1H), 3.28 (dd,  $J = 12.1, 4.8$  Hz, 1H), 2.39–2.32 (m, 1H), 2.14 (s, 3H), 2.11 (s, 3H), 2.02 (s, 3H), 1.97 (s, 3H), 1.06 (d,  $J = 7.0$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  174.2, 170.7, 170.6, 170.3, 169.8, 92.7, 71.9, 70.3, 66.6, 61.6, 53.9, 49.6, 41.4, 20.8, 20.8, 20.6, 15.4.

FT-IR (neat) 3317, 2974, 2937, 2102, 1747, 1665, 1545, 1439, 1369, 1218, 1161, 1118, 1071, 1041, 952, 923, 900, 596  $\text{cm}^{-1}$ .

HRMS (ESI)  $m/z$  ( $[\text{M}+\text{Na}]^+$ ) calcd for  $\text{C}_{18}\text{H}_{26}\text{N}_4\text{NaO}_{10}$ : 481.1547, found: 481.1538.



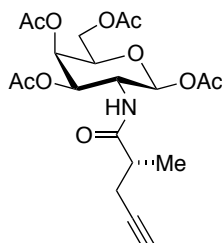
**1,3,4,6-Tetra-*O*-acetyl-2-((*S*)-3-azido-2-methylpropanamido)-2-deoxy- $\beta$ -D-galactopyranose.** The title compound was prepared from 1,3,4,6-tetra-*O*-acetyl-2-amino-2-deoxy- $\beta$ -D-galactopyranose hydrochloride (384 mg, 1.00 mmol) and (*S*)-3-azido-2-methylpropanoic acid<sup>14</sup> (129 mg, 1.00 mmol). The product was purified by column chromatography (12%→100% ethyl acetate/hexanes): 328 mg (72%). Light-yellow solid.

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  5.75 (d,  $J$  = 8.8 Hz, 1H), 5.74 (d,  $J$  = 9.4 Hz, 1H), 5.38 (dd,  $J$  = 3.4, 1.1 Hz, 1H), 5.14 (dd,  $J$  = 11.3, 3.3 Hz, 1H), 4.47 (ddd,  $J$  = 11.3, 9.1, 9.1 Hz, 1H), 4.19–4.10 (m, 2H), 4.05 (ddd,  $J$  = 6.5, 6.5, 1.2 Hz, 1H), 3.51 (dd,  $J$  = 12.0, 9.2 Hz, 1H), 3.32 (dd,  $J$  = 12.0, 4.7 Hz, 1H), 2.39–2.30 (m, 1H), 2.18 (s, 3H), 2.11 (s, 3H), 2.05 (s, 3H), 2.02 (s, 3H), 1.09 (d,  $J$  = 7.0 Hz, 3H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  174.1, 171.0, 170.6, 170.3, 169.6, 93.1, 72.1, 70.1, 66.5, 61.5, 54.0, 49.9, 41.6, 20.9, 20.8, 20.6, 15.5.

FT-IR (neat) 3312, 2977, 2937, 2102, 1745, 1664, 1545, 1437, 1369, 1216, 1162, 1118, 1071, 1041, 953, 923, 899, 850, 629, 597, 559, 538, 488 cm<sup>-1</sup>.

HRMS (ESI)  $m/z$  ([M+Na]<sup>+</sup>) calcd for C<sub>18</sub>H<sub>26</sub>N<sub>4</sub>NaO<sub>10</sub>: 481.1547, found: 481.1523.



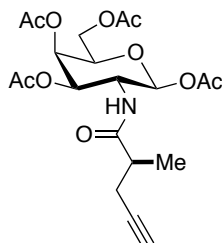
**1,3,4,6-Tetra-*O*-acetyl-2-((*R*)-2-methylpent-4-ynamido)-2-deoxy- $\beta$ -D-galactopyranose.** (*R*)-2-Methylpent-4-ynoic acid was prepared according to a literature procedure.<sup>12</sup> The title compound was prepared from 1,3,4,6-tetra-*O*-acetyl-2-amino-2-deoxy- $\beta$ -D-galactopyranose hydrochloride (312 mg, 0.813 mmol) and (*R*)-2-methylpent-4-ynoic acid (91.0 mg, 0.812 mmol). The product was purified by column chromatography (1st purification: 8%→70% acetone/hexanes; 2nd purification: 12%→100% ethyl acetate/hexanes): 225 mg (63%). White solid.

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  5.69 (d,  $J$  = 8.8 Hz, 1H), 5.49 (d,  $J$  = 9.6 Hz, 1H), 5.38 (dd,  $J$  = 3.3, 0.8 Hz, 1H), 5.10 (dd,  $J$  = 11.3, 3.3 Hz, 1H), 4.52 (ddd,  $J$  = 11.1, 9.3, 9.3 Hz, 1H), 4.18 (dd,  $J$  = 11.3, 6.7 Hz, 1H), 4.12 (dd,  $J$  = 11.3, 6.4 Hz, 1H), 4.02 (ddd,  $J$  = 6.5, 6.5, 1.0 Hz, 1H), 2.45–2.33 (m, 2H), 2.29 (ddd,  $J$  = 15.8, 4.9, 2.8 Hz, 1H), 2.17 (s, 3H), 2.12 (s, 3H), 2.05 (s, 3H), 2.01 (s, 3H), 1.98 (dd,  $J$  = 2.7, 2.7 Hz, 1H), 1.15 (d,  $J$  = 6.6 Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  174.8, 170.8, 170.5, 170.3, 169.7, 93.1, 81.8, 72.0, 70.5, 70.4, 66.5, 61.4, 49.6, 41.0, 23.2, 21.2, 20.8, 20.8, 17.4.

FT-IR (neat) 3291, 2968, 2919, 2852, 1747, 1663, 1543, 1457, 1432, 1369, 1220, 1162, 1114, 1070, 1042, 956, 923, 898, 710, 675, 659, 643, 630, 595, 536  $\text{cm}^{-1}$ .

HRMS (ESI)  $m/z$  ( $[\text{M}+\text{Na}]^+$ ) calcd for  $\text{C}_{20}\text{H}_{27}\text{NNaO}_{10}$ : 464.1533, found: 464.1520.



**1,3,4,6-Tetra-*O*-acetyl-2-((*S*)-2-methylpent-4-ynamido)-2-deoxy- $\beta$ -D-galactopyranose.**

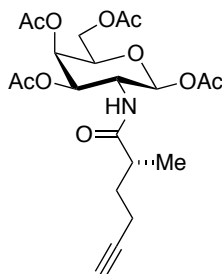
(*S*)-2-Methylpent-4-ynoic acid was prepared according to a literature procedure.<sup>12</sup> The title compound was prepared from 1,3,4,6-tetra-*O*-acetyl-2-amino-2-deoxy- $\beta$ -D-galactopyranose hydrochloride (472 mg, 1.23 mmol) and (*S*)-2-methylpent-4-ynoic acid (138 mg, 1.23 mmol). The product was purified by column chromatography (1st purification: 12% $\rightarrow$ 100% ethyl acetate/hexanes; 2nd purification: 8% $\rightarrow$ 70% acetone/hexanes): 322 mg (59%). White solid.

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  5.72 (d,  $J = 8.8$  Hz, 1H), 5.52 (d,  $J = 9.5$  Hz, 1H), 5.38 (dd,  $J = 3.3, 0.7$  Hz, 1H), 5.08 (dd,  $J = 11.3, 3.3$  Hz, 1H), 4.51 (ddd,  $J = 11.4, 9.2, 9.2$  Hz, 1H), 4.18 (dd,  $J = 11.4, 6.7$  Hz, 1H), 4.12 (dd,  $J = 11.3, 6.5$  Hz, 1H), 4.02 (ddd,  $J = 6.5, 6.5, 1.2$  Hz, 1H), 2.45–2.29 (m, 3H), 2.17 (s, 3H), 2.12 (s, 3H), 2.05 (s, 3H), 2.01 (s, 3H), 1.98 (dd,  $J = 2.6, 2.6$  Hz, 1H), 1.16 (d,  $J = 6.7$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  174.8, 170.8, 170.5, 170.3, 169.6, 93.1, 81.8, 72.1, 70.4, 70.4, 66.4, 61.4, 49.6, 40.8, 23.1, 21.0, 20.9, 20.8, 17.4.

FT-IR (neat) 3294, 2967, 2918, 2851, 1748, 1665, 1542, 1458, 1431, 1370, 1221, 1162, 1115, 1073, 1042, 953, 921, 904, 674, 657, 642, 621, 596, 562, 553, 539  $\text{cm}^{-1}$ .

HRMS (ESI)  $m/z$  ( $[\text{M}+\text{Na}]^+$ ) calcd for  $\text{C}_{20}\text{H}_{27}\text{NNaO}_{10}$ : 464.1533, found: 464.1524.



**1,3,4,6-Tetra-*O*-acetyl-2-((*R*)-2-methylhex-5-ynamido)-2-deoxy- $\beta$ -D-galactopyranose.**

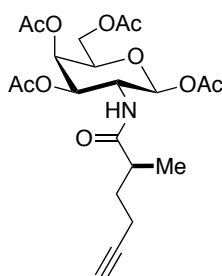
The title compound was prepared from 1,3,4,6-tetra-*O*-acetyl-2-amino-2-deoxy- $\beta$ -D-galactopyranose hydrochloride (255 mg, 0.664 mmol) and (*R*)-2-methylhex-5-ynoic acid (83.7 mg, 0.663 mmol). The product was purified by column chromatography (15% $\rightarrow$ 100% ethyl acetate/hexanes): 208 mg (69%). White solid.

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  5.73 (d,  $J = 8.8$  Hz, 1H), 5.47 (d,  $J = 9.6$  Hz, 1H), 5.38 (dd,  $J = 3.2, 0.6$  Hz, 1H), 5.12 (dd,  $J = 11.2, 3.3$  Hz, 1H), 4.49 (ddd,  $J = 11.4, 9.3, 9.3$  Hz, 1H), 4.17 (dd,  $J = 11.3, 6.7$  Hz, 1H), 4.11 (dd,  $J = 11.3, 6.5$  Hz, 1H), 4.03 (ddd,  $J = 6.5, 6.5, 0.9$  Hz, 1H), 2.43–2.36 (m, 1H), 2.26–2.05 (m, 2H), 2.17 (s, 3H), 2.13 (s, 3H), 2.05 (s, 3H), 2.01 (s, 3H), 1.95 (dd,  $J = 2.7, 2.7$  Hz, 1H), 1.83–1.76 (m, 1H), 1.57–1.50 (m, 1H), 1.09 (d,  $J = 6.9$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  176.0, 170.8, 170.6, 170.3, 169.5, 92.8, 83.3, 72.1, 70.6, 69.6, 66.6, 61.4, 49.4, 40.1, 32.0, 21.0, 20.8, 20.7, 18.0, 16.3.

FT-IR (neat) 3285, 3081, 2967, 2936, 1746, 1660, 1541, 1434, 1369, 1217, 1161, 1118, 1070, 1041, 956, 928, 902, 736, 648, 597, 538  $\text{cm}^{-1}$ .

HRMS (ESI)  $m/z$  ( $[\text{M}+\text{Na}]^+$ ) calcd for  $\text{C}_{21}\text{H}_{29}\text{NNaO}_{10}$ : 478.1689, found: 478.1682.



### 1,3,4,6-Tetra-*O*-acetyl-2-((*S*)-2-methylhex-5-ynamido)-2-deoxy- $\beta$ -D-galactopyranose.

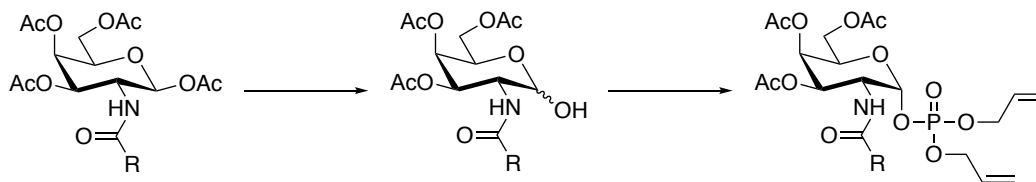
The title compound was prepared from 1,3,4,6-tetra-*O*-acetyl-2-amino-2-deoxy- $\beta$ -D-galactopyranose hydrochloride (265 mg, 0.690 mmol) and (*S*)-2-methylhex-5-ynoic acid (87.1 mg, 0.690 mmol). The product was purified by column chromatography (15%→100% ethyl acetate/hexanes): 229 mg (73%). White solid.

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  5.73 (d,  $J = 8.8$  Hz, 1H), 5.49 (d,  $J = 9.5$  Hz, 1H), 5.39 (dd,  $J = 3.2, 0.8$  Hz, 1H), 5.08 (dd,  $J = 11.5, 3.3$  Hz, 1H), 4.49 (ddd,  $J = 11.5, 9.2, 9.2$  Hz, 1H), 4.18 (dd,  $J = 11.4, 6.6$  Hz, 1H), 4.12 (dd,  $J = 11.4, 6.5$  Hz, 1H), 4.03 (ddd,  $J = 6.5, 6.5, 1.2$  Hz, 1H), 2.44–2.36 (m, 1H), 2.25–2.19 (m, 1H), 2.17 (s, 3H), 2.11 (s, 3H), 2.11–2.06 (m, 1H), 2.05 (s, 3H), 2.02 (s, 3H), 1.95 (dd,  $J = 2.7, 2.7$  Hz, 1H), 1.82–1.75 (m, 1H), 1.57–1.50 (m, 1H), 1.09 (d,  $J = 6.9$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  176.1, 170.7, 170.5, 170.3, 169.6, 93.3, 83.3, 72.1, 70.3, 69.6, 66.4, 61.5, 49.3, 40.2, 32.0, 20.9, 20.8, 20.8, 18.0, 16.4.

FT-IR (neat) 3287, 2966, 2932, 1745, 1661, 1540, 1434, 1369, 1216, 1162, 1118, 1069, 1040, 957, 920, 902, 733, 647, 597, 537  $\text{cm}^{-1}$ .

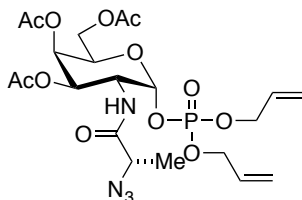
HRMS (ESI)  $m/z$  ( $[\text{M}+\text{Na}]^+$ ) calcd for  $\text{C}_{21}\text{H}_{29}\text{NNaO}_{10}$ : 478.1689, found: 478.1689.



### Preparation of tri-*O*-acetylated *N*-acetyl- $\alpha$ -D-galactosamine 1-diallyl phosphates (Route 1).



The 1-*O*-acetyl group was deprotected according to a literature procedure,<sup>18</sup> and the crude product was used for the next step without further purification. These compounds were prepared according to a literature procedure.<sup>19</sup>



**Diallyl 3,4,6-tri-*O*-acetyl-2-((*S*)-2-azidopropanamido)-2-deoxy- $\alpha$ -D-galactopyranosyl-1-phosphate.** The title compound was prepared from 3,4,6-tri-*O*-acetyl-2-((*S*)-2-azidopropanamido)-2-deoxy- $\beta$ -D-galactopyranose (748 mg, 1.86 mmol). The product was purified by column chromatography on silica gel (15%→100% ethyl acetate/hexanes) and then on C-18 silica gel (5%→100% acetonitrile/water): 701 mg (67%). White solid.

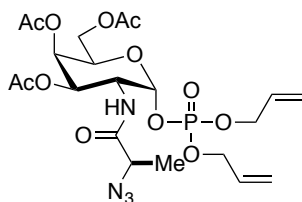
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.01 (d, *J* = 9.0 Hz, 1H), 5.90–5.81 (m, 2H), 5.65 (dd, *J* = 6.3, 3.3 Hz, 1H), 5.39 (dd, *J* = 3.2, 1.3 Hz, 1H), 5.32–5.26 (m, 2H), 5.22–5.18 (m, 2H), 5.15 (dd, *J* = 11.5, 3.2 Hz, 1H), 4.54–4.47 (m, 5H), 4.39 (ddd, *J* = 6.4, 6.4, 0.8 Hz, 1H), 4.07–3.99 (m, 2H), 3.89 (q, *J* = 7.0 Hz, 1H), 2.07 (s, 3H), 1.94 (s, 3H), 1.89 (s, 3H), 1.39 (d, *J* = 7.0 Hz, 3H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)  $\delta$  170.9, 170.4, 170.2, 170.0, 132.0 (d, *J* = 6.9 Hz), 131.9 (d, *J* = 6.7 Hz), 118.8, 118.7, 96.4 (d, *J* = 6.7 Hz), 68.6 (d, *J* = 3.7 Hz), 68.5, 67.1, 66.7, 61.4, 58.5, 47.6 (d, *J* = 7.7 Hz), 20.5, 20.5, 17.1.

<sup>31</sup>P NMR (202 MHz, CDCl<sub>3</sub>)  $\delta$  -2.1.

FT-IR (neat) 3270, 3079, 2983, 2114, 1745, 1690, 1529, 1454, 1427, 1371, 1217, 1164, 1137, 1103, 1052, 1018, 990, 945, 675, 647, 626, 601, 558, 531, 474 cm<sup>-1</sup>.

HRMS (ESI) *m/z* ([M+Na]<sup>+</sup>) calcd for C<sub>21</sub>H<sub>31</sub>N<sub>4</sub>NaO<sub>12</sub>P: 585.1574, found: 585.1571.



**Diallyl 3,4,6-tri-*O*-acetyl-2-((*R*)-2-azidopropanamido)-2-deoxy- $\alpha$ -D-galactopyranosyl-1-phosphate.** The title compound was prepared from 3,4,6-tri-*O*-acetyl-2-((*R*)-2-azidopropanamido)-2-deoxy- $\beta$ -D-galactopyranose (277 mg, 0.688 mmol). The product was purified by column chromatography on silica gel (15%→100% ethyl acetate/hexanes) and then on C-18 silica gel (5%→100% acetonitrile/water): 132 mg (34%). White solid.

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  6.51 (d, *J* = 9.5 Hz, 1H), 6.00–5.90 (m, 2H), 5.76 (dd, *J* = 6.0, 3.4 Hz, 1H), 5.45 (dd, *J* = 3.3, 1.3 Hz, 1H), 5.43–5.37 (m, 2H), 5.33–5.28 (m, 2H), 5.23 (dd, *J* = 11.4, 3.2 Hz, 1H), 4.65–4.57 (m, 5H), 4.42 (ddd, *J* = 6.5, 6.5, 1.4 Hz, 1H),

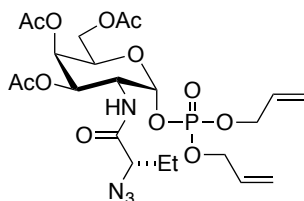
4.15–4.06 (m, 2H), 4.04 (q,  $J = 7.1$  Hz, 1H), 2.16 (s, 3H), 2.03 (s, 3H), 1.99 (s, 3H), 1.49 (d,  $J = 7.0$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  170.7, 170.4, 170.2, 132.1 (d,  $J = 6.7$  Hz), 119.2, 119.0, 96.5 (d,  $J = 6.1$  Hz), 69.0 (d,  $J = 5.3$  Hz), 68.9, 68.8 (d,  $J = 5.3$  Hz), 67.5, 66.9, 61.5, 59.0, 47.8 (d,  $J = 7.8$  Hz), 20.8, 20.8, 20.7, 17.1.

$^{31}\text{P}$  NMR (202 MHz,  $\text{CDCl}_3$ )  $\delta$  -1.4.

FT-IR (neat) 3268, 3083, 2959, 2928, 2854, 2112, 1749, 1691, 1534, 1457, 1427, 1372, 1235, 1164, 1138, 1102, 1052, 1024, 992, 949, 893, 875, 650, 625, 598, 536, 473  $\text{cm}^{-1}$ .

HRMS (ESI)  $m/z$  ( $[\text{M}+\text{Na}]^+$ ) calcd for  $\text{C}_{21}\text{H}_{31}\text{N}_4\text{NaO}_{12}\text{P}$ : 585.1574, found: 585.1571.



**Diallyl 3,4,6-tri-*O*-acetyl-2-((*S*)-2-azidobutanamido)-2-deoxy- $\alpha$ -D-galactopyranosyl-1-phosphate.** The title compound was prepared from 3,4,6-tri-*O*-acetyl-2-((*S*)-2-azidobutanamido)-2-deoxy- $\beta$ -D-galactopyranose (275 mg, 0.660 mmol). The product was purified by column chromatography on silica gel (15%→100% ethyl acetate/hexanes) and then on C-18 silica gel (5%→100% acetonitrile/water): 124 mg (33%). White solid.

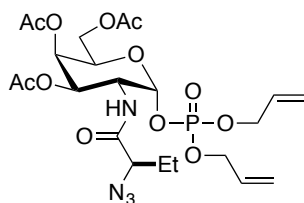
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.20 (d,  $J = 9.0$  Hz, 1H), 5.90–5.82 (m, 2H), 5.64 (dd,  $J = 6.3, 3.3$  Hz, 1H), 5.40 (dd,  $J = 3.2, 1.3$  Hz, 1H), 5.32–5.27 (m, 2H), 5.23–5.19 (m, 2H), 5.16 (dd,  $J = 11.6, 3.1$  Hz, 1H), 4.56 (dddd,  $J = 12.0, 9.1, 3.2, 3.2$  Hz, 1H), 4.53–4.46 (m, 4H), 4.42 (ddd,  $J = 6.5, 6.5, 1.3$  Hz, 1H), 4.78–4.01 (m, 2H), 3.68 (dd,  $J = 7.3, 5.7$  Hz, 1H), 2.07 (s, 3H), 1.95 (s, 3H), 1.88 (s, 3H), 1.86–1.77 (m, 1H), 1.77–1.68 (m, 1H), 0.90 (t,  $J = 7.4$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  170.5, 170.4, 170.2, 170.0, 132.0 (d,  $J = 6.9$  Hz), 131.9 (d,  $J = 6.5$  Hz), 118.8, 118.7, 96.6 (d,  $J = 6.8$  Hz), 69.0 (d,  $J = 5.3$  Hz), 68.8, 68.8 (d,  $J = 5.9$  Hz), 67.1, 66.7, 64.5, 61.4, 47.5 (d,  $J = 7.9$  Hz), 25.3, 20.6, 20.5, 20.5, 9.9.

$^{31}\text{P}$  NMR (202 MHz,  $\text{CDCl}_3$ )  $\delta$  -1.5.

FT-IR (neat) 3271, 3077, 2966, 2924, 2852, 2105, 1746, 1688, 1554, 1461, 1427, 1371, 1219, 1164, 1138, 1102, 1052, 1019, 990, 956, 873, 806, 736, 648, 627, 533, 475  $\text{cm}^{-1}$ .

HRMS (ESI)  $m/z$  ( $[\text{M}+\text{Na}]^+$ ) calcd for  $\text{C}_{22}\text{H}_{33}\text{N}_4\text{NaO}_{12}\text{P}$ : 599.1730, found: 599.1721.



**Diallyl 3,4,6-tri-*O*-acetyl-2-((*R*)-2-azidobutanamido)-2-deoxy- $\alpha$ -D-galactopyranosyl-1-phosphate.** The title compound was prepared from 3,4,6-tri-*O*-acetyl-2-((*R*)-2-azidobutanamido)-2-deoxy- $\beta$ -D-galactopyranose (376 mg, 0.903 mmol). The product

was purified by column chromatography on silica gel (15%→100% ethyl acetate/hexanes) and then on C-18 silica gel (5%→100% acetonitrile/water): 293 mg (56%). White solid.

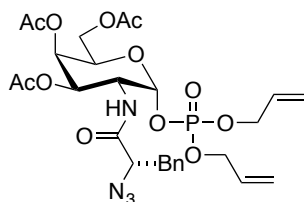
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 6.51 (d, *J* = 9.6 Hz, 1H), 6.01–5.91 (m, 2H), 5.76 (dd, *J* = 5.9, 3.4 Hz, 1H), 5.46 (dd, *J* = 3.2, 1.3 Hz, 1H), 5.44–5.38 (m, 2H), 5.33–5.29 (m, 2H), 5.21 (dd, *J* = 11.4, 3.1 Hz, 1H), 4.70–4.58 (m, 5H), 4.42 (ddd, *J* = 6.6, 6.6, 0.7 Hz, 1H), 4.15–4.07 (m, 2H), 4.00 (dd, *J* = 6.2, 4.7 Hz, 1H), 2.17 (s, 3H), 2.03 (s, 3H), 1.98 (s, 3H), 1.96–1.85 (m, 2H), 0.95 (t, *J* = 7.4 Hz, 3H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 170.7, 170.4, 170.2, 169.5, 132.1 (d, *J* = 6.8 Hz), 119.2, 119.0, 96.6 (d, *J* = 6.1 Hz), 69.9 (d, *J* = 5.3 Hz), 68.8, 68.8 (d, *J* = 5.3 Hz), 67.5, 66.9, 65.1, 61.5, 47.6 (d, *J* = 7.8 Hz), 25.1, 20.8, 20.8, 20.8, 9.2.

<sup>31</sup>P NMR (202 MHz, CDCl<sub>3</sub>) δ -1.4.

FT-IR (neat) 3274, 3077, 2968, 2927, 2106, 1747, 1689, 1526, 1460, 1427, 1371, 1232, 1164, 1139, 1103, 1021, 991, 942, 875, 808, 677, 649, 626, 593, 535, 471 cm<sup>-1</sup>.

HRMS (ESI) *m/z* ([M+Na]<sup>+</sup>) calcd for C<sub>22</sub>H<sub>33</sub>N<sub>4</sub>NaO<sub>12</sub>P: 599.1730, found: 599.1723.



**Diallyl 3,4,6-tri-*O*-acetyl-2-((*S*)-2-azido-3-phenylpropanamido)-2-deoxy- $\alpha$ -D-galactopyranosyl-1-phosphate.** The title compound was prepared from 3,4,6-tri-*O*-acetyl-2-((*S*)-2-azido-3-phenylpropanamido)-2-deoxy- $\beta$ -D-galactopyranose (325 mg, 0.679 mmol). The product was purified by column chromatography on silica gel (15%→100% ethyl acetate/hexanes) and then on C-18 silica gel (5%→100% acetonitrile/water): 188 mg (43%). Light-yellow solid.

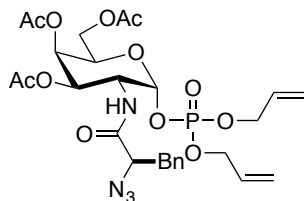
<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.39 (d, *J* = 8.9 Hz, 1H), 7.25–7.21 (m, 2H), 7.20–7.16 (m, 3H), 5.89–5.80 (m, 2H), 5.64 (dd, *J* = 6.3, 3.3 Hz, 1H), 5.41 (dd, *J* = 3.1, 1.3 Hz, 1H), 5.32–5.24 (m, 2H), 5.22–5.19 (m, 1H), 5.18–5.14 (m, 2H), 4.57 (dddd, *J* = 11.9, 8.9, 3.1, 3.1 Hz, 1H), 4.53–4.43 (m, 5H), 4.09–4.03 (m, 2H), 3.98 (dd, *J* = 9.5, 4.3 Hz, 1H), 3.14 (dd, *J* = 14.1, 4.3 Hz, 1H), 2.90 (dd, *J* = 14.1, 9.5 Hz, 1H), 2.09 (s, 3H), 1.95 (s, 3H), 1.86 (s, 3H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 170.3, 170.2, 170.0, 136.4, 132.0 (d, *J* = 7.1 Hz), 131.8 (d, *J* = 6.4 Hz), 129.1, 128.6, 127.1, 118.9, 118.7, 96.5 (d, *J* = 6.9 Hz), 68.6 (d, *J* = 5.5 Hz), 68.6, 68.6 (d, *J* = 5.1 Hz), 67.1, 66.6, 64.5, 61.3, 47.6 (d, *J* = 7.8 Hz), 38.3, 20.6, 20.5, 20.5.

<sup>31</sup>P NMR (202 MHz, CDCl<sub>3</sub>) δ -2.5.

FT-IR (neat) 3270, 3076, 3030, 2974, 2116, 1746, 1688, 1559, 1455, 1426, 1371, 1217, 1163, 1140, 1114, 1052, 1017, 987, 940, 876, 805, 731, 700, 647, 626, 601, 590, 556, 529, 482 cm<sup>-1</sup>.

HRMS (ESI) *m/z* ([M+Na]<sup>+</sup>) calcd for C<sub>27</sub>H<sub>35</sub>N<sub>4</sub>NaO<sub>12</sub>P: 661.1887, found: 661.1890.



**Diallyl 3,4,6-tri-*O*-acetyl-2-((*R*)-2-azido-3-phenylpropanamido)-2-deoxy- $\alpha$ -D-galactopyranosyl-1-phosphate.** The title compound was prepared from 3,4,6-tri-*O*-acetyl-2-((*R*)-2-azido-3-phenylpropanamido)-2-deoxy- $\beta$ -D-galactopyranose (351 mg, 0.734 mmol). The product was purified by column chromatography on silica gel (15% $\rightarrow$ 100% ethyl acetate/hexanes) and then on C-18 silica gel (5% $\rightarrow$ 100% acetonitrile/water): 172 mg (37%). White solid.

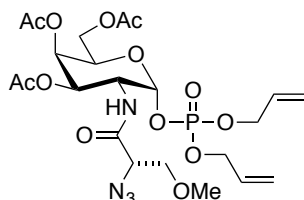
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.26–7.21 (m, 2H), 7.19–7.16 (m, 3H), 7.11 (d,  $J = 9.1$  Hz, 1H), 5.89–5.78 (m, 2H), 5.67 (dd,  $J = 6.3, 3.3$  Hz, 1H), 5.41 (d,  $J = 2.7$  Hz, 1H), 5.31–5.24 (m, 2H), 5.20–5.14 (m, 3H), 4.59 (dddd,  $J = 12.1, 9.1, 3.2, 3.2$  Hz, 1H), 4.51–4.40 (m, 5H), 4.09–4.02 (m, 3H), 3.22 (dd,  $J = 14.1, 4.0$  Hz, 1H), 2.82 (dd,  $J = 14.2, 9.6$  Hz, 1H), 2.09 (s, 3H), 1.94 (s, 3H), 1.87 (s, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  170.4, 170.1, 169.9, 169.5, 136.3, 131.9 (d,  $J = 7.4$  Hz), 131.9 (d,  $J = 7.2$  Hz), 129.1, 128.5, 127.0, 118.7, 96.3 (d,  $J = 6.6$  Hz), 68.6, 68.6 (d,  $J = 5.2$  Hz), 68.4 (d,  $J = 5.3$  Hz), 67.1, 66.6, 64.7, 61.4, 47.5 (d,  $J = 7.8$  Hz), 37.9, 20.5, 20.5, 20.4.

$^{31}\text{P}$  NMR (202 MHz,  $\text{CDCl}_3$ )  $\delta$  -2.2.

FT-IR (neat) 3271, 3073, 3029, 2957, 2111, 1748, 1691, 1535, 1455, 1427, 1371, 1236, 1163, 1140, 1115, 1022, 991, 945, 877, 743, 701, 675, 649, 625, 594, 528, 473  $\text{cm}^{-1}$ .

HRMS (ESI)  $m/z$  ( $[\text{M}+\text{Na}]^+$ ) calcd for  $\text{C}_{27}\text{H}_{35}\text{N}_4\text{NaO}_{12}\text{P}$ : 661.1887, found: 661.1882.



**Diallyl 3,4,6-tri-*O*-acetyl-2-((*S*)-2-azido-3-methoxypropanamido)-2-deoxy- $\alpha$ -D-galactopyranosyl-1-phosphate.** The title compound was prepared from 3,4,6-tri-*O*-acetyl-2-((*S*)-2-azido-3-methoxypropanamido)-2-deoxy- $\beta$ -D-galactopyranose (268 mg, 0.620 mmol). The product was purified by column chromatography on silica gel (15% $\rightarrow$ 100% ethyl acetate/hexanes) and then on C-18 silica gel (5% $\rightarrow$ 100% acetonitrile/water): 117 mg (32%). Light-yellow solid.

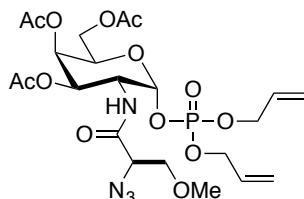
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  6.89 (d,  $J = 9.2$  Hz, 1H), 5.92–5.83 (m, 2H), 5.65 (dd,  $J = 6.0, 3.3$  Hz, 1H), 5.38 (dd,  $J = 3.3, 1.3$  Hz, 1H), 5.34–5.28 (m, 2H), 5.24–5.19 (m, 2H), 5.15 (dd,  $J = 11.5, 3.2$  Hz, 1H), 4.56–4.47 (m, 5H), 4.36 (dd,  $J = 6.6, 6.6$  Hz, 1H), 4.07–3.98 (m, 3H), 3.72 (dd,  $J = 10.2, 3.9$  Hz, 1H), 3.60 (dd,  $J = 10.1, 7.0$  Hz, 1H), 3.30 (s, 3H), 2.07 (s, 3H), 1.94 (s, 3H), 1.91 (s, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  170.4, 170.2, 167.0, 167.9, 132.0 (d,  $J = 7.4$  Hz), 132.0 (d,  $J = 7.5$  Hz), 118.7, 96.3 (d,  $J = 6.4$  Hz), 72.6, 68.6 (d,  $J = 5.2$  Hz), 68.6 (d,  $J = 5.8$  Hz), 68.5, 67.1, 66.7, 62.5, 61.3, 59.1, 47.7 (d,  $J = 8.1$  Hz), 20.6, 20.5.

$^{31}\text{P}$  NMR (202 MHz,  $\text{CDCl}_3$ )  $\delta$  -1.4.

FT-IR (neat) 3279, 3078, 2959, 2926, 2855, 2108, 1745, 1688, 1527, 1458, 1427, 1371, 1219, 1163, 1113, 1020, 991, 945, 875, 803, 627, 602, 555, 533,  $481\text{ cm}^{-1}$ .

HRMS (ESI)  $m/z$  ( $[\text{M}+\text{Na}]^+$ ) calcd for  $\text{C}_{22}\text{H}_{33}\text{N}_4\text{NaO}_{13}\text{P}$ : 615.1679, found: 615.1672.



**Diallyl 3,4,6-tri-*O*-acetyl-2-((*R*)-2-azido-3-methoxypropanamido)-2-deoxy- $\alpha$ -D-galactopyranosyl-1-phosphate.** The title compound was prepared from 3,4,6-tri-*O*-acetyl-2-((*R*)-2-azido-3-methoxypropanamido)-2-deoxy- $\beta$ -D-galactopyranose (284 mg, 0.657 mmol). The product was purified by column chromatography on silica gel (15% $\rightarrow$ 100% ethyl acetate/hexanes) and then on C-18 silica gel (5% $\rightarrow$ 100% acetonitrile/water): 134 mg (34%). White solid.

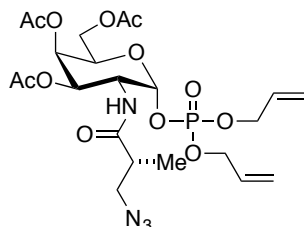
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  6.63 (d,  $J$  = 9.5 Hz, 1H), 6.01–5.91 (m, 2H), 5.77 (dd,  $J$  = 6.0, 3.4 Hz, 1H), 5.45 (dd,  $J$  = 3.3, 1.3 Hz, 1H), 5.44–5.38 (m, 2H), 5.33–5.29 (m, 2H), 5.22 (dd,  $J$  = 11.4, 3.2 Hz, 1H), 4.67–4.58 (m, 5H), 4.42 (ddd,  $J$  = 6.6, 6.6, 0.8 Hz, 1H), 4.16–4.06 (m, 3H), 3.82 (dd,  $J$  = 10.2, 3.5 Hz, 1H), 3.73 (dd,  $J$  = 10.2, 6.2 Hz, 1H), 3.39 (s, 3H), 2.16 (s, 3H), 2.03 (s, 3H), 1.99 (s, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  170.7, 170.4, 170.2, 167.7, 132.1 (d,  $J$  = 6.7 Hz), 132.1 (d,  $J$  = 6.9 Hz), 119.2, 119.0, 96.5 (d,  $J$  = 6.0 Hz), 72.5, 69.0 (d,  $J$  = 5.4 Hz), 68.9, 68.8 (d,  $J$  = 5.3 Hz), 67.4, 66.9, 63.1, 61.5, 59.4, 47.8 (d,  $J$  = 7.9 Hz), 20.8, 20.8, 20.7.

$^{31}\text{P}$  NMR (202 MHz,  $\text{CDCl}_3$ )  $\delta$  -1.4.

FT-IR (neat) 3268, 3081, 2929, 2110, 1748, 1688, 1527, 1459, 1427, 1372, 1234, 1164, 1137, 1115, 1023, 992, 948, 874, 804, 648, 628, 603, 593, 553, 534, 500,  $472\text{ cm}^{-1}$ .

HRMS (ESI)  $m/z$  ( $[\text{M}+\text{Na}]^+$ ) calcd for  $\text{C}_{22}\text{H}_{33}\text{N}_4\text{NaO}_{13}\text{P}$ : 615.1679, found: 615.1675.



**Diallyl 3,4,6-tri-*O*-acetyl-2-((*R*)-3-azido-2-methylpropanamido)-2-deoxy- $\alpha$ -D-galactopyranosyl-1-phosphate.** 3,4,6-Tri-*O*-acetyl-2-((*R*)-3-azido-2-methylpropanamido)-2-deoxy- $\beta$ -D-galactopyranose (148 mg, 0.355 mmol) and 1*H*-tetrazole (74.6 mg, 1.06 mmol) were suspended in toluene (5.0 mL) in a 20-mL vial. The mixture was sonicated for 1 h, and the toluene was removed under reduced pressure. The vial was charged with nitrogen, and dichloromethane (3.5 mL) was added. The resulting mixture was cooled to 0 °C, and then diallyl *N,N*-diisopropylphosphoramidite (0.132 mL, 0.499 mmol) was added dropwise over 5 min. The reaction mixture was stirred at 0 °C

for 1 h, and then cooled to  $-78\text{ }^{\circ}\text{C}$ . 3-Chloroperbenzoic acid (123 mg, 0.711 mmol) was added, and the mixture was stirred at  $0\text{ }^{\circ}\text{C}$  for 1 h. The mixture was diluted with dichloromethane (100 mL), washed with aqueous  $\text{Na}_2\text{SO}_3$  (10%; 50 mL), saturated aqueous  $\text{NaHCO}_3$  (50 mL), and brine (50 mL), dried over  $\text{Na}_2\text{SO}_4$ , and concentrated. The crude product was purified by column chromatography on silica gel (15% $\rightarrow$ 100% ethyl acetate/hexanes) and then on C-18 silica gel (5% $\rightarrow$ 100% acetonitrile/water): 121 mg (59%). White solid.

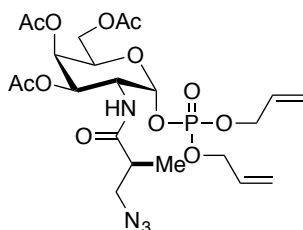
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  6.89 (d,  $J = 9.2$  Hz, 1H), 5.92–5.83 (m, 2H), 5.63 (dd,  $J = 6.2, 3.3$  Hz, 1H), 5.39 (dd,  $J = 3.1, 1.3$  Hz, 1H), 5.33–5.29 (m, 2H), 5.23–5.20 (m, 2H), 5.16 (dd,  $J = 11.5, 3.1$  Hz, 1H), 4.62 (dddd,  $J = 12.1, 9.2, 3.2, 3.2$  Hz, 1H), 4.57–4.48 (m, 4H), 4.44 (dd,  $J = 6.6, 6.6$  Hz, 1H), 4.08–4.02 (m, 2H), 3.52 (dd,  $J = 12.1, 7.9$  Hz, 1H), 3.20 (dd,  $J = 12.1, 5.9$  Hz, 1H), 2.50–2.43 (m, 1H), 2.08 (s, 3H), 1.95 (s, 3H), 1.89 (s, 3H), 1.06 (d,  $J = 7.0$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  174.4, 170.5, 170.3, 170.1, 132.2 (d,  $J = 6.9$  Hz), 132.0 (d,  $J = 6.7$  Hz), 118.8, 118.7, 97.0 (d,  $J = 7.1$  Hz), 68.7, 68.6 (d,  $J = 6.1$  Hz), 68.6 (d,  $J = 5.0$  Hz), 67.4, 66.8, 61.5, 53.7, 47.3 (d,  $J = 7.6$  Hz), 40.7, 20.6, 20.6, 20.5, 15.5.

$^{31}\text{P}$  NMR (202 MHz,  $\text{CDCl}_3$ )  $\delta$   $-2.5$ .

FT-IR (neat) 3280, 3082, 2975, 2938, 2880, 2101, 1745, 1679, 1544, 1458, 1426, 1371, 1216, 1163, 1141, 1109, 1053, 1017, 989, 942, 805, 733, 702, 645, 626, 591, 542, 529, 474  $\text{cm}^{-1}$ .

HRMS (ESI)  $m/z$  ( $[\text{M}+\text{Na}]^+$ ) calcd for  $\text{C}_{22}\text{H}_{33}\text{N}_4\text{NaO}_{12}\text{P}$ : 599.1730, found: 599.1715.



**Diallyl 3,4,6-tri-*O*-acetyl-2-((*S*)-3-azido-2-methylpropanamido)-2-deoxy- $\alpha$ -D-galactopyranosyl-1-phosphate.** 3,4,6-Tri-*O*-acetyl-2-((*S*)-3-azido-2-methylpropanamido)-2-deoxy- $\beta$ -D-galactopyranose (247 mg, 0.593 mmol) and 1*H*-tetrazole (125 mg, 1.78 mmol) were suspended in toluene (5.0 mL) in a 20-mL vial. The mixture was sonicated for 1 h, and the toluene was removed under reduced pressure. The vial was charged with nitrogen, and dichloromethane (5.9 mL) was added. The resulting mixture was cooled to  $0\text{ }^{\circ}\text{C}$ , and then diallyl *N,N*-diisopropylphosphoramidite (0.220 mL, 0.832 mmol) was added dropwise over 5 min. The reaction mixture was stirred at  $0\text{ }^{\circ}\text{C}$  for 1 h, and then cooled to  $-78\text{ }^{\circ}\text{C}$ . 3-Chloroperbenzoic acid (205 mg, 1.19 mmol) was added, and the mixture was stirred at  $0\text{ }^{\circ}\text{C}$  for 1 h. The mixture was diluted with dichloromethane (100 mL), washed with aqueous  $\text{Na}_2\text{SO}_3$  (10%; 50 mL), saturated aqueous  $\text{NaHCO}_3$  (50 mL), and brine (50 mL), dried over  $\text{Na}_2\text{SO}_4$ , and concentrated. The crude product was purified by column chromatography on silica gel (15% $\rightarrow$ 100% ethyl acetate/hexanes) and then on C-18 silica gel (5% $\rightarrow$ 100% acetonitrile/water): 249 mg (73%). Viscous light-yellow oil.

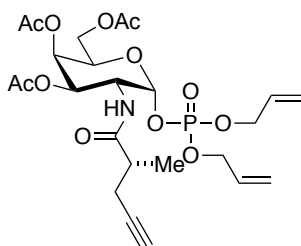
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  6.83 (d,  $J = 9.0$  Hz, 1H), 5.92–5.80 (m, 2H), 5.63 (dd,  $J = 6.4, 3.3$  Hz, 1H), 5.40 (dd,  $J = 3.2, 1.3$  Hz, 1H), 5.33–5.27 (m, 2H), 5.24–5.18 (m, 2H), 5.15 (dd,  $J = 11.5, 3.2$  Hz, 1H), 4.61 (dddd,  $J = 12.0, 9.0, 3.2, 3.2$  Hz, 1H), 4.55–4.46 (m, 4H), 4.43 (dd,  $J = 6.6, 6.6$  Hz, 1H), 4.08–4.01 (m, 2H), 3.44 (dd,  $J = 12.0, 8.7$  Hz, 1H), 3.21 (dd,  $J = 12.0, 5.4$  Hz, 1H), 2.49–2.42 (m, 1H), 2.09 (s, 3H), 1.95 (s, 3H), 1.91 (s, 3H), 1.06 (d,  $J = 7.0$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  174.2, 170.9, 170.4, 170.2, 132.2 (d,  $J = 6.8$  Hz), 132.0 (d,  $J = 6.6$  Hz), 119.1, 119.1, 97.0 (d,  $J = 6.7$  Hz), 68.8 (d,  $J = 5.4$  Hz), 68.8, 68.7 (d,  $J = 5.3$  Hz), 67.3, 66.9, 61.6, 54.1, 47.6 (d,  $J = 7.6$  Hz), 41.0, 20.8, 20.7, 20.6, 15.3.

$^{31}\text{P}$  NMR (202 MHz,  $\text{CDCl}_3$ )  $\delta$  -2.4.

FT-IR (neat) 3283, 3085, 2975, 2938, 2876, 2100, 1746, 1680, 1542, 1458, 1427, 1372, 1232, 1163, 1141, 1110, 1052, 1021, 991, 948, 888, 711, 676, 650, 626, 603, 526, 477  $\text{cm}^{-1}$ .

HRMS (ESI)  $m/z$  ( $[\text{M}+\text{Na}]^+$ ) calcd for  $\text{C}_{22}\text{H}_{33}\text{N}_4\text{NaO}_{12}\text{P}$ : 599.1730, found: 599.1720.



**Diallyl 3,4,6-tri-*O*-acetyl-2-((*R*)-2-methylpent-4-ynamido)-2-deoxy- $\alpha$ -D-galactopyranosyl-1-phosphate.** The title compound was prepared from 3,4,6-tri-*O*-acetyl-2-((*R*)-2-methylpent-4-ynamido)-2-deoxy- $\beta$ -D-galactopyranose (148 mg, 0.371 mmol). The product was purified by column chromatography (12% $\rightarrow$ 100% ethyl acetate/hexanes): 179 mg (86%). White solid.

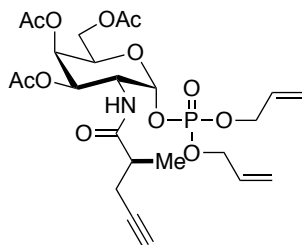
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  6.53 (d,  $J = 9.2$  Hz, 1H), 5.93–5.83 (m, 2H), 5.65 (dd,  $J = 6.0, 3.3$  Hz, 1H), 5.40 (dd,  $J = 3.2, 1.3$  Hz, 1H), 5.35–5.30 (m, 2H), 5.25–5.21 (m, 2H), 5.16 (dd,  $J = 11.5, 3.1$  Hz, 1H), 4.63 (dddd,  $J = 12.2, 9.3, 3.2, 3.2$  Hz, 1H), 4.55–4.50 (m, 4H), 4.41 (dd,  $J = 6.7, 6.7$  Hz, 1H), 4.01–4.01 (m, 2H), 2.45–2.38 (m, 2H), 2.27–2.21 (m, 1H), 2.09 (s, 3H), 1.98 (dd,  $J = 2.5, 2.5$  Hz, 1H), 1.96 (s, 3H), 1.90 (s, 3H), 1.13 (d,  $J = 6.6$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  175.2, 170.6, 170.3, 170.1, 132.2 (d,  $J = 6.8$  Hz), 132.0 (d,  $J = 6.7$  Hz), 119.0, 118.9, 97.1 (d,  $J = 6.9$  Hz), 81.8, 70.2, 68.7 (d,  $J = 5.3$  Hz), 68.7, 68.6 (d,  $J = 5.3$  Hz), 67.4, 66.9, 61.5, 47.2 (d,  $J = 7.8$  Hz), 40.0, 22.6, 20.7, 20.6, 20.6, 17.1.

$^{31}\text{P}$  NMR (202 MHz,  $\text{CDCl}_3$ )  $\delta$  -2.1.

FT-IR (neat) 3296, 2973, 2251, 1746, 1674, 1542, 1459, 1426, 1371, 1232, 1163, 1140, 1103, 1053, 1018, 988, 947, 907, 726, 646, 593, 552, 527, 473  $\text{cm}^{-1}$ .

HRMS (ESI)  $m/z$  ( $[\text{M}+\text{Na}]^+$ ) calcd for  $\text{C}_{24}\text{H}_{34}\text{NNaO}_{12}\text{P}$ : 582.1716, found: 582.1708.



**Diallyl 3,4,6-tri-*O*-acetyl-2-((*S*)-2-methylpent-4-ynamido)-2-deoxy- $\alpha$ -D-galactopyranosyl-1-phosphate.** The title compound was prepared from 3,4,6-tri-*O*-acetyl-2-((*S*)-2-methylpent-4-ynamido)-2-deoxy- $\beta$ -D-galactopyranose (219 mg, 0.548 mmol). The product was purified by column chromatography on silica gel (12% $\rightarrow$ 100% ethyl acetate/hexanes) and then on C-18 silica gel (10% $\rightarrow$ 100% acetonitrile/water): 234 mg (76%). Viscous colorless oil.

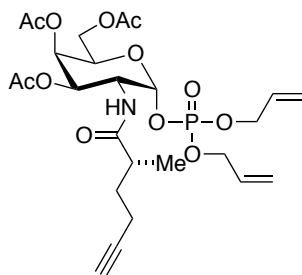
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  6.44 (d,  $J = 9.2$  Hz, 1H), 5.94–5.83 (m, 2H), 5.65 (dd,  $J = 6.1, 3.3$  Hz, 1H), 5.40 (d,  $J = 2.3$  Hz, 1H), 5.36–5.29 (m, 2H), 5.26–5.20 (m, 2H), 5.13 (dd,  $J = 11.5, 3.2$  Hz, 1H), 4.64 (dddd,  $J = 12.2, 9.3, 3.2, 3.2$  Hz, 1H), 4.56–4.49 (m, 4H), 4.40 (dd,  $J = 6.5, 6.5$  Hz, 1H), 4.09–4.02 (m, 2H), 2.44–2.34 (m, 2H), 2.27–2.19 (m, 1H), 2.10 (s, 3H), 1.96 (s, 3H), 1.94 (dd,  $J = 2.7, 2.7$  Hz, 1H), 1.91 (s, 3H), 1.14 (d,  $J = 6.4$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  175.0, 170.6, 170.3, 170.2, 132.1 (d,  $J = 6.7$  Hz), 132.0 (d,  $J = 6.7$  Hz), 119.0, 119.0, 97.0 (d,  $J = 6.8$  Hz), 81.9, 70.1, 68.7 (d,  $J = 5.3$  Hz), 68.7, 68.6 (d,  $J = 5.4$  Hz), 67.5, 66.8, 61.5, 47.2 (d,  $J = 7.8$  Hz), 40.1, 23.0, 20.8, 20.7, 20.6, 17.0.

$^{31}\text{P}$  NMR (202 MHz,  $\text{CDCl}_3$ )  $\delta$  -2.0.

FT-IR (neat) 3281, 2976, 2938, 2248, 1745, 1676, 1540, 1459, 1426, 1371, 1219, 1164, 1139, 1104, 1052, 1018, 989, 945, 910, 728, 645, 600, 591, 526, 481  $\text{cm}^{-1}$ .

HRMS (ESI)  $m/z$  ( $[\text{M}+\text{Na}]^+$ ) calcd for  $\text{C}_{24}\text{H}_{34}\text{NNaO}_{12}\text{P}$ : 582.1716, found: 582.1702.



**Diallyl 3,4,6-tri-*O*-acetyl-2-((*R*)-2-methylhex-5-ynamido)-2-deoxy- $\alpha$ -D-galactopyranosyl-1-phosphate.** The title compound was prepared from 3,4,6-tri-*O*-acetyl-2-((*R*)-2-methylhex-5-ynamido)-2-deoxy- $\beta$ -D-galactopyranose (150 mg, 0.363 mmol). The product was purified by column chromatography (15% $\rightarrow$ 100% ethyl acetate/hexanes): 173 mg (83%). White solid.

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  6.29 (d,  $J = 9.2$  Hz, 1H), 5.93–5.84 (m, 2H), 5.64 (dd,  $J = 5.8, 3.3$  Hz, 1H), 5.39 (dd,  $J = 3.3, 1.4$  Hz, 1H), 5.35–5.29 (m, 2H), 5.24–5.20 (m, 2H), 5.15 (dd,  $J = 11.5, 3.1$  Hz, 1H), 4.63 (dddd,  $J = 12.6, 9.4, 3.3, 3.3$  Hz, 1H), 4.57–4.48 (m, 4H), 4.39 (ddd,  $J = 6.4, 6.4, 1.2$  Hz, 1H), 4.09–4.00 (m, 2H), 2.40–2.33 (m, 1H), 2.17–



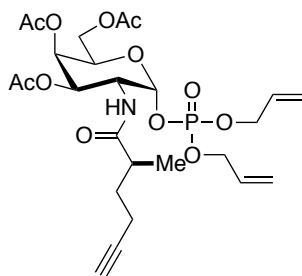
2.06 (m, 2H), 2.09 (s, 3H), 1.96 (s, 3H), 1.91 (s, 3H), 1.90 (dd,  $J = 2.7, 2.7$  Hz, 1H), 1.85–1.78 (m, 1H), 1.53–1.46 (m, 1H), 1.04 (d,  $J = 6.9$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  176.3, 170.6, 170.3, 170.1, 132.2 (d,  $J = 6.6$  Hz), 132.0 (d,  $J = 6.6$  Hz), 118.9, 118.8, 97.1 (d,  $J = 6.9$  Hz), 83.5, 69.1, 68.6 (d,  $J = 5.1$  Hz), 68.6, 68.6 (d,  $J = 6.3$  Hz), 67.5, 66.9, 61.5, 47.2 (d,  $J = 8.0$  Hz), 39.8, 31.9, 20.7, 20.6, 20.6, 17.6, 16.3.

$^{31}\text{P}$  NMR (202 MHz,  $\text{CDCl}_3$ )  $\delta$  -1.9.

FT-IR (neat) 3287, 2966, 2935, 1745, 1676, 1535, 1459, 1428, 1371, 1218, 1162, 1138, 1109, 1054, 1017, 990, 943, 808, 734, 627, 600, 527, 472  $\text{cm}^{-1}$ .

HRMS (ESI)  $m/z$  ( $[\text{M}+\text{Na}]^+$ ) calcd for  $\text{C}_{25}\text{H}_{36}\text{NNaO}_{12}\text{P}$ : 596.1873, found: 596.1866.



**Diallyl 3,4,6-tri-*O*-acetyl-2-((*S*)-2-methylhex-5-ynamido)-2-deoxy- $\alpha$ -D-galactopyranosyl-1-phosphate.** The title compound was prepared from 3,4,6-tri-*O*-acetyl-2-((*S*)-2-methylhex-5-ynamido)-2-deoxy- $\beta$ -D-galactopyranose (169 mg, 0.409 mmol). The product was purified by column chromatography (15%→100% ethyl acetate/hexanes): 185 mg (79%). White solid.

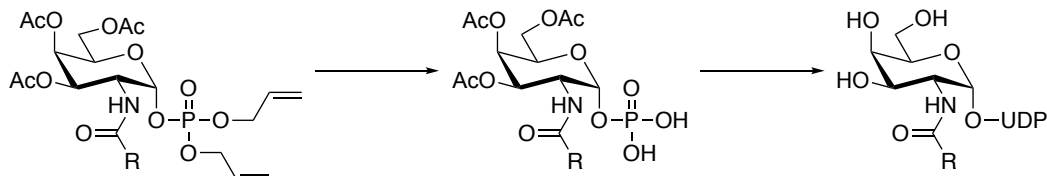
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  6.36 (d,  $J = 9.1$  Hz, 1H), 5.92–5.82 (m, 2H), 5.64 (dd,  $J = 6.2, 3.3$  Hz, 1H), 5.39 (dd,  $J = 3.2, 1.3$  Hz, 1H), 5.34–5.28 (m, 2H), 5.24–5.19 (m, 2H), 5.12 (dd,  $J = 11.6, 3.1$  Hz, 1H), 4.62 (dddd,  $J = 12.1, 9.1, 3.2, 3.2$  Hz, 1H), 4.54–4.47 (m, 4H), 4.38 (ddd,  $J = 6.5, 6.5, 1.4$  Hz, 1H), 4.08–4.01 (m, 2H), 2.41–2.34 (m, 1H), 2.17–2.07 (m, 1H), 2.09 (s, 3H), 2.06–1.99 (m, 1H), 1.95 (s, 3H), 1.92 (s, 3H), 1.89 (dd,  $J = 2.6, 2.6$  Hz, 1H), 1.79–1.70 (m, 1H), 1.52–1.46 (m, 1H), 1.05 (d,  $J = 6.8$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  176.1, 170.5, 170.3, 170.1, 132.2 (d,  $J = 6.8$  Hz), 132.0 (d,  $J = 6.6$  Hz), 118.9, 118.8, 97.0 (d,  $J = 6.9$  Hz), 83.3, 69.3, 68.6 (d,  $J = 6.5$  Hz), 68.6, 68.6 (d,  $J = 7.0$  Hz), 67.3, 66.7, 61.5, 47.0 (d,  $J = 7.7$  Hz), 39.6, 32.4, 20.7, 20.6, 17.4, 16.2.

$^{31}\text{P}$  NMR (202 MHz,  $\text{CDCl}_3$ )  $\delta$  -2.1.

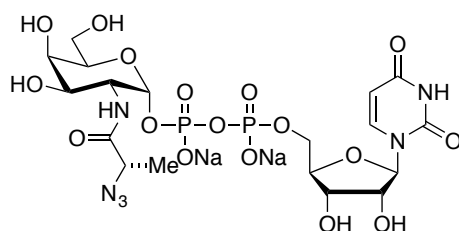
FT-IR (neat) 3283, 3079, 2967, 2937, 1745, 1676, 1540, 1458, 1428, 1371, 1217, 1163, 1139, 1110, 1052, 1017, 990, 944, 807, 734, 711, 642, 627, 601, 528, 472, 433  $\text{cm}^{-1}$ .

HRMS (ESI)  $m/z$  ( $[\text{M}+\text{Na}]^+$ ) calcd for  $\text{C}_{25}\text{H}_{36}\text{NNaO}_{12}\text{P}$ : 596.1873, found: 596.1861.



### Representative procedure for the preparation of UDP-*N*-acetyl- $\alpha$ -D-galactosamine derivatives (Route 1)

Tri-*O*-acetylated *N*-acetyl- $\alpha$ -D-galactosamine 1-phosphate derivatives were prepared from tri-*O*-acetylated *N*-acetyl- $\alpha$ -D-galactosamine 1-diallyl phosphates according to a literature procedure.<sup>19</sup> Then, UDP-sugars were prepared using a modified literature procedure.<sup>19,20</sup> A mixture of the sugar 1-phosphate (0.200 mmol), uridine 5'-monophosphomorpholidate 4-morpholine-*N,N'*-dicyclohexylcarboxamide salt (224 mg, 0.326 mmol), 1-methylimidazole hydrochloride (128 mg, 1.08 mmol), and NEt<sub>3</sub> (55.8  $\mu$ L, 0.400 mmol) in DMF (3.92 mL) in a 25-mL round-bottom flask was stirred at r.t. for 12 h. The tri-*O*-acetylated UDP-sugar was purified by column chromatography on C-18 silica gel (MeOH/water; water was doped with 10 mM *n*-Bu<sub>3</sub>NH•HCO<sub>3</sub>) and then preparative HPLC on C-18 silica gel (MeOH/water; water was doped with 10 mM *n*-Bu<sub>3</sub>NH•HCO<sub>3</sub>). Aqueous *n*-Bu<sub>3</sub>NH•HCO<sub>3</sub> solution (10 mM) was prepared by bubbling CO<sub>2</sub> through a solution of *n*-Bu<sub>3</sub>NH (10 mM) in water until all *n*-Bu<sub>3</sub>NH dissolved into the water. Pure fractions were collected, concentrated, redissolved in water (100 mL), rinsed with CH<sub>2</sub>Cl<sub>2</sub> (3 x 50 mL), and concentrated. The compound was dissolved in MeOH/water/NEt<sub>3</sub> (5 mL, 5:2:1) in a 25-mL round-bottom flask, and the reaction mixture was stirred at r.t. overnight. The product was purified by preparative HPLC on C-18 silica gel (MeOH/water; water was doped with 10 mM *n*-Bu<sub>3</sub>NH•HCO<sub>3</sub>). Pure fractions were collected, concentrated, redissolved in water (100 mL), rinsed with CH<sub>2</sub>Cl<sub>2</sub> (3 x 50 mL), and concentrated. Finally, the purified compound was passed through a Bio-Rad AG<sup>®</sup> 50W-X8 resin (sodium form) and lyophilized.



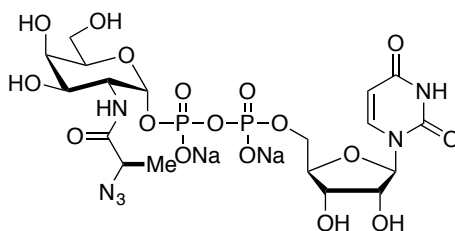
**UDP-Sugar (S)-3.** The title compound was prepared from diallyl 3,4,6-tri-*O*-acetyl-2-((*S*)-2-azidopropanamido)-2-deoxy- $\alpha$ -D-galactopyranosyl-1-phosphate (415 mg, 0.738 mmol). The tri-*O*-acetylated UDP-sugar was purified by column chromatography on C-18 silica gel (0%→100% MeOH/water; water was doped with 10 mM *n*-Bu<sub>3</sub>NH•HCO<sub>3</sub>) and then preparative HPLC on C-18 silica gel (10%→60% MeOH/water; water was doped with 10 mM *n*-Bu<sub>3</sub>NH•HCO<sub>3</sub>). The compound was dissolved in MeOH/water/NEt<sub>3</sub> (5:2:1), and the reaction mixture was stirred at r.t. overnight. The product was purified by preparative HPLC on an Agilent prep 100 Å C18 column (MeOH/water (min:%): 0:10, 10:10, 60:60; water was doped with 10 mM *n*-Bu<sub>3</sub>NH•HCO<sub>3</sub>; 20 mL/min; t<sub>r</sub> = 36.8 min): 148 mg (28%). White solid.

$^1\text{H}$  NMR (500 MHz,  $\text{D}_2\text{O}$ )  $\delta$  7.94 (d,  $J = 8.1$  Hz, 1H), 5.97–5.94 (m, 2H), 5.56 (dd,  $J = 7.0, 3.4$  Hz, 1H), 4.37–4.33 (m, 2H), 4.29–4.21 (m, 4H), 4.21–4.15 (m, 2H), 4.04 (d,  $J = 3.0$  Hz, 1H), 3.98 (dd,  $J = 11.0, 3.2$  Hz, 1H), 3.79–3.72 (m, 2H), 1.47 (d,  $J = 7.1$  Hz, 3H).  $^{13}\text{C}$  NMR (126 MHz,  $\text{D}_2\text{O}$ )  $\delta$  175.0, 166.8, 152.4, 142.3, 103.2, 95.3 (d,  $J = 6.2$  Hz), 89.2, 83.8 (d,  $J = 9.0$  Hz), 74.4, 72.7, 70.2, 69.1, 68.1, 65.6 (d,  $J = 4.9$  Hz), 61.6, 58.8, 50.5 (d,  $J = 8.6$  Hz), 17.5.

$^{31}\text{P}$  NMR (202 MHz,  $\text{D}_2\text{O}$ )  $\delta$  -10.5 (d,  $J = 20.8$  Hz), -12.3 (d,  $J = 20.8$  Hz).

FT-IR (neat) 3234, 2115, 1665, 1544, 1468, 1425, 1392, 1342, 1227, 1114, 1076, 1037, 985, 916, 861, 814, 780, 766, 717, 685, 620, 533, 503, 492, 444  $\text{cm}^{-1}$ .

HRMS (ESI)  $m/z$  ( $[\text{M}+\text{H}]^+$ ) calcd for  $\text{C}_{18}\text{H}_{27}\text{N}_6\text{Na}_2\text{O}_{17}\text{P}_2$ : 707.0703, found: 707.0706.



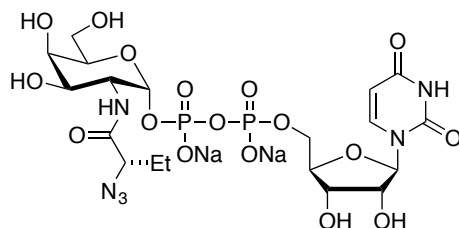
**UDP-Sugar (R)-3.** The title compound was prepared from diallyl 3,4,6-tri-*O*-acetyl-2-((*R*)-2-azidopropanamido)-2-deoxy- $\alpha$ -D-galactopyranosyl-1-phosphate (132 mg, 0.235 mmol). The tri-*O*-acetylated UDP-sugar was purified by column chromatography on C-18 silica gel (0%→100% MeOH/water; water was doped with 10 mM *n*- $\text{Bu}_3\text{NH}\cdot\text{HCO}_3$ ) and then preparative HPLC on C-18 silica gel (10%→60% MeOH/water; water was doped with 10 mM *n*- $\text{Bu}_3\text{NH}\cdot\text{HCO}_3$ ). The compound was dissolved in MeOH/water/ $\text{NEt}_3$  (5:2:1), and the reaction mixture was stirred at r.t. overnight. The product was purified by preparative HPLC on an Agilent Microsorb 100-5 C18 column (MeOH/water (min:%): 0:10, 10:10, 60:60; water was doped with 10 mM *n*- $\text{Bu}_3\text{NH}\cdot\text{HCO}_3$ ; 20 mL/min;  $t_r = 51.4$  min): 53.6 mg (32%). White solid.

$^1\text{H}$  NMR (500 MHz,  $\text{D}_2\text{O}$ )  $\delta$  7.93 (d,  $J = 8.1$  Hz, 1H), 5.97–5.93 (m, 2H), 5.54 (dd,  $J = 7.1, 3.5$  Hz, 1H), 4.37–4.32 (m, 2H), 4.28–4.23 (m, 2H), 4.23–4.15 (m, 4H), 4.03 (d,  $J = 3.1$  Hz, 1H), 3.98 (dd,  $J = 10.9, 3.2$  Hz, 1H), 3.78–3.71 (m, 2H), 1.47 (d,  $J = 6.9$  Hz, 3H).  $^{13}\text{C}$  NMR (126 MHz,  $\text{D}_2\text{O}$ )  $\delta$  174.8, 167.3, 152.7, 142.2, 103.3, 95.1 (d,  $J = 6.2$  Hz), 89.2, 83.7 (d,  $J = 9.0$  Hz), 74.4, 72.7, 70.2, 69.0, 67.9, 65.6 (d,  $J = 4.9$  Hz), 61.6, 58.8, 50.6 (d,  $J = 8.2$  Hz), 17.1.

$^{31}\text{P}$  NMR (202 MHz,  $\text{D}_2\text{O}$ )  $\delta$  -10.6 (d,  $J = 20.6$  Hz), -12.3 (d,  $J = 20.7$  Hz).

FT-IR (neat) 3254, 2115, 1669, 1536, 1464, 1426, 1391, 1346, 1232, 1116, 1078, 1037, 984, 921, 858, 814, 779, 718, 689, 630, 506  $\text{cm}^{-1}$ .

HRMS (ESI)  $m/z$  ( $[\text{M}+\text{H}]^+$ ) calcd for  $\text{C}_{18}\text{H}_{27}\text{N}_6\text{Na}_2\text{O}_{17}\text{P}_2$ : 707.0703, found: 707.0699.



**UDP-Sugar (S)-4.** The title compound was prepared from diallyl 3,4,6-tri-*O*-acetyl-2-((*S*)-2-azidobutanamido)-2-deoxy- $\alpha$ -D-galactopyranosyl-1-phosphate (124 mg, 0.215 mmol). The tri-*O*-acetylated UDP-sugar was purified by column chromatography on C-18 silica gel (0% $\rightarrow$ 100% MeOH/water; water was doped with 10 mM *n*-Bu<sub>3</sub>NH•HCO<sub>3</sub>) and then preparative HPLC on C-18 silica gel (10% $\rightarrow$ 60% MeOH/water; water was doped with 10 mM *n*-Bu<sub>3</sub>NH•HCO<sub>3</sub>). The compound was dissolved in MeOH/water/NEt<sub>3</sub> (5:2:1), and the reaction mixture was stirred at r.t. overnight. The product was purified by preparative HPLC on an Agilent Microsorb 100-5 C18 column (MeOH/water (min:%): 0:10, 10:10, 60:60; water was doped with 10 mM *n*-Bu<sub>3</sub>NH•HCO<sub>3</sub>; 20 mL/min; t<sub>r</sub> = 53.5 min): 70.1 mg (45%). White solid.

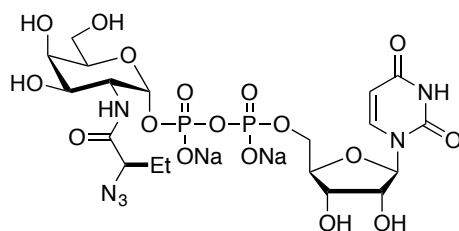
<sup>1</sup>H NMR (500 MHz, D<sub>2</sub>O)  $\delta$  7.92 (d, *J* = 8.1 Hz, 1H), 5.97–5.92 (m, 2H), 5.55 (dd, *J* = 6.9, 3.5 Hz, 1H), 4.36–4.32 (m, 2H), 4.30–4.14 (m, 5H), 4.09 (dd, *J* = 6.6, 6.6 Hz, 1H), 4.02 (d, *J* = 3.2 Hz, 1H), 3.95 (dd, *J* = 11.0, 3.2 Hz, 1H), 3.78–3.71 (m, 2H), 1.89–1.75 (m, 2H), 0.96 (t, *J* = 7.4 Hz, 3H).

<sup>13</sup>C NMR (126 MHz, D<sub>2</sub>O)  $\delta$  174.2, 167.4, 152.8, 142.2, 103.3, 95.4 (d, *J* = 6.6 Hz), 89.2, 83.7 (d, *J* = 9.1 Hz), 74.4, 72.7, 70.3, 69.2, 68.0, 65.6 (d, *J* = 5.4 Hz), 64.6, 61.6, 50.5 (d, *J* = 8.6 Hz), 25.7, 9.8.

<sup>31</sup>P NMR (202 MHz, D<sub>2</sub>O)  $\delta$  -10.5 (d, *J* = 20.8 Hz), -12.3 (d, *J* = 20.2 Hz).

FT-IR (neat) 3294, 2107, 1671, 1537, 1465, 1427, 1392, 1238, 1118, 1080, 1053, 987, 919, 855, 814, 779, 714, 685, 611, 504, 438 cm<sup>-1</sup>.

HRMS (ESI) *m/z* ([M+H]<sup>+</sup>) calcd for C<sub>19</sub>H<sub>29</sub>N<sub>6</sub>Na<sub>2</sub>O<sub>17</sub>P<sub>2</sub>: 721.0860, found: 721.0852.



**UDP-Sugar (R)-4.** The title compound was prepared from diallyl 3,4,6-tri-*O*-acetyl-2-((*R*)-2-azidobutanamido)-2-deoxy- $\alpha$ -D-galactopyranosyl-1-phosphate (293 mg, 0.508 mmol). The tri-*O*-acetylated UDP-sugar was purified by column chromatography on C-18 silica gel (0% $\rightarrow$ 100% MeOH/water; water was doped with 10 mM *n*-Bu<sub>3</sub>NH•HCO<sub>3</sub>) and then preparative HPLC on C-18 silica gel (10% $\rightarrow$ 60% MeOH/water; water was doped with 10 mM *n*-Bu<sub>3</sub>NH•HCO<sub>3</sub>). The compound was dissolved in MeOH/water/NEt<sub>3</sub> (5:2:1), and the reaction mixture was stirred at r.t. overnight. The product was purified by preparative HPLC on an Agilent Microsorb 100-5 C18 column (MeOH/water (min:%): 0:10, 10:10, 60:60; water was doped with 10 mM *n*-Bu<sub>3</sub>NH•HCO<sub>3</sub>; 20 mL/min; t<sub>r</sub> = 53.8 min): 113 mg (31%). White solid.

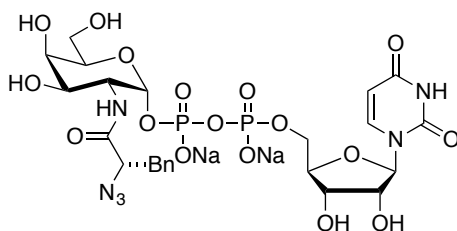
$^1\text{H}$  NMR (500 MHz,  $\text{D}_2\text{O}$ )  $\delta$  7.95 (d,  $J$  = 8.2 Hz, 1H), 5.97–5.95 (m, 2H), 5.56 (dd,  $J$  = 6.8, 3.5 Hz, 1H), 4.38–4.34 (m, 2H), 4.31–4.16 (m, 5H), 4.07–4.03 (m, 2H), 4.00 (dd,  $J$  = 10.9, 3.1 Hz, 1H), 3.80–3.72 (m, 2H), 1.94–1.76 (m, 2H), 0.98 (t,  $J$  = 7.4 Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{D}_2\text{O}$ )  $\delta$  174.0, 166.8, 152.3, 142.3, 103.2, 95.2 (d,  $J$  = 5.7 Hz), 89.2, 83.7 (d,  $J$  = 8.9 Hz), 74.4, 72.6, 70.2, 69.0, 67.9, 65.5 (d,  $J$  = 3.8 Hz), 64.7, 61.6, 50.5 (d,  $J$  = 8.5 Hz), 25.3, 9.9.

$^{31}\text{P}$  NMR (202 MHz,  $\text{D}_2\text{O}$ )  $\delta$  -10.6 (d,  $J$  = 20.8 Hz), -12.4 (d,  $J$  = 20.6 Hz).

FT-IR (neat) 3232, 2109, 1669, 1464, 1429, 1391, 1232, 1115, 1079, 1038, 987, 918, 811, 717, 687, 511, 444, 425  $\text{cm}^{-1}$ .

HRMS (ESI)  $m/z$  ( $[\text{M}+\text{H}]^+$ ) calcd for  $\text{C}_{19}\text{H}_{29}\text{N}_6\text{Na}_2\text{O}_{17}\text{P}_2$ : 721.0860, found: 721.0860.



**UDP-Sugar (S)-5.** The title compound was prepared from diallyl 3,4,6-tri-*O*-acetyl-2-((*S*)-2-azido-3-phenylpropanamido)-2-deoxy- $\alpha$ -D-galactopyranosyl-1-phosphate (188 mg, 0.294 mmol). The tri-*O*-acetylated UDP-sugar was purified by column chromatography on C-18 silica gel (0%→100% MeOH/water; water was doped with 10 mM  $n\text{-Bu}_3\text{NH}\cdot\text{HCO}_3$ ). The compound was dissolved in MeOH/water/ $\text{NET}_3$  (5:2:1), and the reaction mixture was stirred at r.t. overnight. The product was purified by preparative HPLC on an Agilent Microsorb 100-5 C18 column (MeOH/water (min:%): 0:0, 10:0, 60:50; water was doped with 10 mM  $n\text{-Bu}_3\text{NH}\cdot\text{HCO}_3$ ; 20 mL/min;  $t_r$  = 52.4 min): 107 mg (46%). White solid.

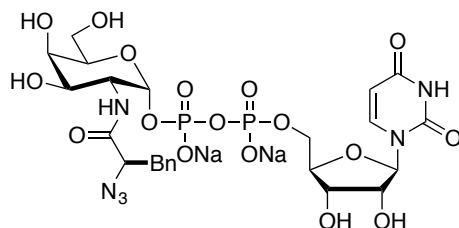
$^1\text{H}$  NMR (500 MHz,  $\text{D}_2\text{O}$ )  $\delta$  7.86 (dd,  $J$  = 8.1, 1.3 Hz, 1H), 7.37–7.32 (m, 4H), 7.32–7.27 (m, 1H), 5.90 (d,  $J$  = 3.5 Hz, 1H), 5.85 (d,  $J$  = 8.0 Hz, 1H), 5.56 (dd,  $J$  = 7.1, 3.4 Hz, 1H), 4.43 (dd,  $J$  = 9.2, 5.3 Hz, 1H), 4.32–4.13 (m, 7H), 4.01 (d,  $J$  = 2.5 Hz, 1H), 3.94 (dd,  $J$  = 11.1, 3.0 Hz, 1H), 3.79–3.70 (m, 2H), 3.20 (dd,  $J$  = 14.1, 5.1 Hz, 1H), 3.01 (dd,  $J$  = 14.0, 9.4 Hz, 1H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{D}_2\text{O}$ )  $\delta$  173.6, 166.9, 152.4, 142.2, 137.2, 130.1, 129.4, 127.9, 103.2, 95.4 (d,  $J$  = 5.3 Hz), 89.2, 83.8 (d,  $J$  = 9.3 Hz), 74.5, 72.8, 70.3, 69.2, 68.2, 65.7 (d,  $J$  = 4.9 Hz), 64.6, 61.7, 50.6 (d,  $J$  = 8.6 Hz), 38.5.

$^{31}\text{P}$  NMR (202 MHz,  $\text{D}_2\text{O}$ )  $\delta$  -10.5 (d,  $J$  = 21.4 Hz), -12.2 (d,  $J$  = 20.6 Hz).

FT-IR (neat) 3250, 2115, 1670, 1539, 1458, 1426, 1394, 1338, 1235, 1113, 1079, 1052, 987, 921, 859, 812, 742, 700, 621, 501  $\text{cm}^{-1}$ .

HRMS (ESI)  $m/z$  ( $[\text{M}+\text{Na}]^+$ ) calcd for  $\text{C}_{24}\text{H}_{30}\text{N}_6\text{Na}_3\text{O}_{17}\text{P}_2$ : 805.0836, found: 805.0833.



**UDP-Sugar (R)-5.** The title compound was prepared from diallyl 3,4,6-tri-*O*-acetyl-2-((*R*)-2-azido-3-phenylpropanamido)-2-deoxy- $\alpha$ -D-galactopyranosyl-1-phosphate (172 mg, 0.269 mmol). The tri-*O*-acetylated UDP-sugar was purified by column chromatography on C-18 silica gel (0% $\rightarrow$ 100% MeOH/water; water was doped with 10 mM *n*-Bu<sub>3</sub>NH•HCO<sub>3</sub>). The compound was dissolved in MeOH/water/NEt<sub>3</sub> (5:2:1), and the reaction mixture was stirred at r.t. overnight. The product was purified by preparative HPLC on an Agilent Microsorb 100-5 C18 column (MeOH/water (min:%): 0:0, 10:0, 60:50; water was doped with 10 mM *n*-Bu<sub>3</sub>NH•HCO<sub>3</sub>; 20 mL/min;  $t_r$  = 51.5 min): 52.2 mg (25%). White solid.

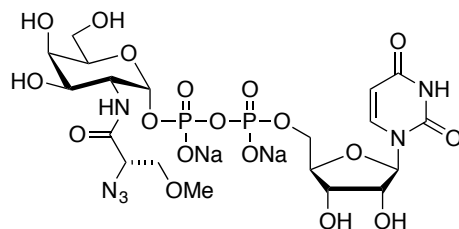
<sup>1</sup>H NMR (500 MHz, D<sub>2</sub>O)  $\delta$  7.80 (d,  $J$  = 8.0 Hz, 1H), 7.40–7.25 (m, 5H), 5.85 (d,  $J$  = 3.3 Hz, 1H), 5.80 (d,  $J$  = 8.2 Hz, 1H), 5.54 (dd,  $J$  = 6.8, 3.4 Hz, 1H), 4.38 (dd,  $J$  = 10.0, 4.2 Hz, 1H), 4.30–4.17 (m, 6H), 4.17–4.11 (m, 1H), 4.04 (d,  $J$  = 3.1 Hz, 1H), 3.98 (dd,  $J$  = 11.0, 3.0 Hz, 1H), 3.80–3.71 (m, 2H), 3.27 (dd,  $J$  = 14.2, 4.2 Hz, 1H), 2.98 (dd,  $J$  = 14.2, 10.0 Hz, 1H).

<sup>13</sup>C NMR (126 MHz, D<sub>2</sub>O)  $\delta$  173.4, 166.9, 152.3, 142.2, 137.2, 130.0, 129.4, 127.8, 103.2, 95.3 (d,  $J$  = 5.9 Hz), 89.4, 83.7 (d,  $J$  = 9.0 Hz), 74.4, 72.8, 70.2, 69.1, 68.2, 65.8 (d,  $J$  = 4.8 Hz), 64.6, 61.7, 50.7 (d,  $J$  = 8.4 Hz), 38.0.

<sup>31</sup>P NMR (202 MHz, D<sub>2</sub>O)  $\delta$  -10.5 (d,  $J$  = 20.8 Hz), -12.4 (d,  $J$  = 21.4 Hz).

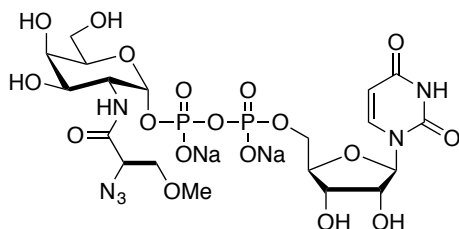
FT-IR (neat) 3294, 2114, 1665, 1543, 1459, 1425, 1390, 1342, 1232, 1112, 1079, 1053, 986, 921, 861, 812, 741, 699, 626, 510 cm<sup>-1</sup>.

HRMS (ESI)  $m/z$  ([M+H]<sup>+</sup>) calcd for C<sub>24</sub>H<sub>31</sub>N<sub>6</sub>Na<sub>2</sub>O<sub>17</sub>P<sub>2</sub>: 783.1016, found: 783.1027.

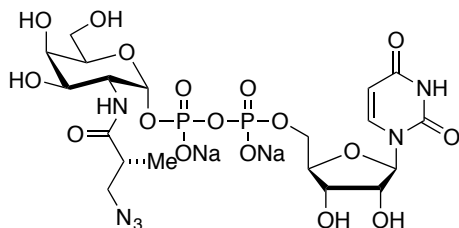


**UDP-Sugar (S)-6.** The title compound was prepared from diallyl 3,4,6-tri-*O*-acetyl-2-((*S*)-2-azido-3-methoxypropanamido)-2-deoxy- $\alpha$ -D-galactopyranosyl-1-phosphate (117 mg, 0.197 mmol). The tri-*O*-acetylated UDP-sugar was purified by column chromatography on C-18 silica gel (0% $\rightarrow$ 100% MeOH/water; water was doped with 10 mM *n*-Bu<sub>3</sub>NH•HCO<sub>3</sub>) and then preparative HPLC on C-18 silica gel (10% $\rightarrow$ 60% MeOH/water; water was doped with 10 mM *n*-Bu<sub>3</sub>NH•HCO<sub>3</sub>). The compound was dissolved in MeOH/water/NEt<sub>3</sub> (5:2:1), and the reaction mixture was stirred at r.t. overnight. The product was purified by preparative HPLC on an Agilent Microsorb 100-5 C18 column (MeOH/water (min:%): 0:10, 10:10, 60:60; water was doped with 10 mM *n*-Bu<sub>3</sub>NH•HCO<sub>3</sub>; 20 mL/min;  $t_r$  = 52.4 min): 72.5 mg (50%). White solid.

$^1\text{H}$  NMR (500 MHz,  $\text{D}_2\text{O}$ )  $\delta$  7.94 (d,  $J$  = 8.1 Hz, 1H), 5.98–5.93 (m, 2H), 5.56 (dd,  $J$  = 6.9, 3.0 Hz, 1H), 4.50–4.46 (m, 1H), 4.38–4.32 (m, 2H), 4.31–4.15 (m, 5H), 4.03 (d,  $J$  = 3.2 Hz, 1H), 3.98 (dd,  $J$  = 10.9, 2.8 Hz, 1H), 3.84–3.70 (m, 4H), 3.41 (s, 3H).  
 $^{13}\text{C}$  NMR (126 MHz,  $\text{D}_2\text{O}$ )  $\delta$  171.3, 166.8, 152.4, 142.3, 103.2, 95.2 (d,  $J$  = 6.4 Hz), 89.2, 83.8 (d,  $J$  = 9.1 Hz), 74.4, 72.8, 72.7, 70.2, 69.1, 68.0, 65.7 (d,  $J$  = 5.0 Hz), 62.7, 61.6, 59.2, 50.6 (d,  $J$  = 8.6 Hz).  
 $^{31}\text{P}$  NMR (202 MHz,  $\text{D}_2\text{O}$ )  $\delta$  -10.4 (d,  $J$  = 19.4 Hz), -12.2 (d,  $J$  = 17.8 Hz).  
 FT-IR (neat) 3250, 2111, 1670, 1539, 1466, 1427, 1392, 1234, 1111, 1079, 1056, 986, 920, 854, 812, 766, 715, 690, 625, 512  $\text{cm}^{-1}$ .  
 HRMS (ESI)  $m/z$  ( $[\text{M}+\text{H}]^+$ ) calcd for  $\text{C}_{19}\text{H}_{29}\text{N}_6\text{Na}_2\text{O}_{18}\text{P}_2$ : 737.0809, found: 737.0800.



**UDP-Sugar (R)-6.** The title compound was prepared from diallyl 3,4,6-tri-*O*-acetyl-2-((*R*)-2-azido-3-methoxypropanamido)-2-deoxy- $\alpha$ -D-galactopyranosyl-1-phosphate (133 mg, 0.224 mmol). The tri-*O*-acetylated UDP-sugar was purified by column chromatography on C-18 silica gel (0% $\rightarrow$ 100% MeOH/water; water was doped with 10 mM *n*-Bu<sub>3</sub>NH $\cdot$ HCO<sub>3</sub>) and then preparative HPLC on C-18 silica gel (10% $\rightarrow$ 60% MeOH/water; water was doped with 10 mM *n*-Bu<sub>3</sub>NH $\cdot$ HCO<sub>3</sub>). The compound was dissolved in MeOH/water/NEt<sub>3</sub> (5:2:1), and the reaction mixture was stirred at r.t. overnight. The product was purified by preparative HPLC on an Agilent Microsorb 100-5 C18 column (MeOH/water (min:%): 0:10, 10:10, 60:60; water was doped with 10 mM *n*-Bu<sub>3</sub>NH $\cdot$ HCO<sub>3</sub>; 20 mL/min;  $t_r$  = 52.4 min): 48.7 mg (29%). White solid.  
 $^1\text{H}$  NMR (500 MHz,  $\text{D}_2\text{O}$ )  $\delta$  7.95 (d,  $J$  = 8.1 Hz, 1H), 5.99–5.94 (m, 2H), 5.56 (dd,  $J$  = 6.4, 2.9 Hz, 1H), 4.44 (dd,  $J$  = 6.4, 3.9 Hz, 1H), 4.39–4.33 (m, 2H), 4.31–4.16 (m, 5H), 4.04 (d,  $J$  = 3.1 Hz, 1H), 4.01 (dd,  $J$  = 11.0, 2.6 Hz, 1H), 3.90–3.70 (m, 4H), 3.42 (s, 3H).  
 $^{13}\text{C}$  NMR (126 MHz,  $\text{D}_2\text{O}$ )  $\delta$  171.2, 168.7, 153.8, 142.1, 103.3, 95.1 (d,  $J$  = 6.4 Hz), 89.3, 83.7 (d,  $J$  = 9.0 Hz), 74.4, 72.7, 72.3, 70.3, 69.0, 67.9, 65.7 (d,  $J$  = 5.3 Hz), 62.8, 61.6, 59.3, 50.74 (d,  $J$  = 8.3 Hz).  
 $^{31}\text{P}$  NMR (202 MHz,  $\text{D}_2\text{O}$ )  $\delta$  -10.6 (d,  $J$  = 20.8 Hz), -12.3 (d,  $J$  = 20.8 Hz).  
 FT-IR (neat) 3246, 2109, 1668, 1466, 1427, 1391, 1232, 1110, 1079, 1056, 986, 917, 857, 813, 768, 714, 688, 624, 506  $\text{cm}^{-1}$ .  
 HRMS (ESI)  $m/z$  ( $[\text{M}+\text{Na}]^+$ ) calcd for  $\text{C}_{19}\text{H}_{28}\text{N}_6\text{Na}_3\text{O}_{18}\text{P}_2$ : 759.0628, found: 759.0619.



**UDP-Sugar (R)-8.** The title compound was prepared from diallyl 3,4,6-tri-*O*-acetyl-2-((*R*)-3-azido-2-methylpropanamido)-2-deoxy- $\alpha$ -D-galactopyranosyl-1-phosphate (67.0 mg, 0.116 mmol). The tri-*O*-acetylated UDP-sugar was purified by column chromatography on C-18 silica gel (0% $\rightarrow$ 100% MeOH/water; water was doped with 10 mM *n*-Bu<sub>3</sub>NH•HCO<sub>3</sub>) and then preparative HPLC on C-18 silica gel (40% $\rightarrow$ 60% MeOH/water; water was doped with 10 mM *n*-Bu<sub>3</sub>NH•HCO<sub>3</sub>). The compound was dissolved in MeOH/water/NEt<sub>3</sub> (5:2:1), and the reaction mixture was stirred at r.t. overnight. The product was purified by preparative HPLC on an Agilent prep 100 Å C18 column (MeOH/water (min:%): 0:10, 10:10, 60:60; water was doped with 10 mM *n*-Bu<sub>3</sub>NH•HCO<sub>3</sub>; 20 mL/min; t<sub>r</sub> = 39.9 min): 18.8 mg (22%). White solid.

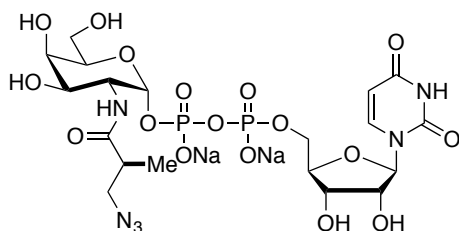
<sup>1</sup>H NMR (500 MHz, D<sub>2</sub>O)  $\delta$  7.92 (d, *J* = 8.1 Hz, 1H), 5.97–5.92 (m, 2H), 5.53 (dd, *J* = 6.8, 3.4 Hz, 1H), 4.36–4.32 (m, 2H), 4.30–4.14 (m, 5H), 4.01 (d, *J* = 3.1 Hz, 1H), 3.95 (dd, *J* = 10.9, 3.2 Hz, 1H), 3.77–3.69 (m, 2H), 3.52 (dd, *J* = 12.4, 8.2 Hz, 1H), 3.36 (dd, *J* = 12.4, 5.8 Hz, 1H), 2.85–2.77 (m, 1H), 1.14 (d, *J* = 7.0 Hz, 3H).

<sup>13</sup>C NMR (126 MHz, D<sub>2</sub>O)  $\delta$  178.7, 168.3, 153.5, 142.2, 103.3, 95.6 (d, *J* = 6.5 Hz), 89.2, 83.8 (d, *J* = 8.9 Hz), 74.4, 72.7, 70.3, 69.2, 68.3, 65.7 (d, *J* = 5.4 Hz), 61.6, 54.1, 50.3 (d, *J* = 8.7 Hz), 41.0, 15.4.

<sup>31</sup>P NMR (202 MHz, D<sub>2</sub>O)  $\delta$  -10.5 (d, *J* = 20.8 Hz), -12.4 (d, *J* = 20.8 Hz).

FT-IR (neat) 3263, 2105, 1671, 1549, 1464, 1430, 1391, 1350, 1235, 1115, 1080, 1058, 1035, 986, 921, 814, 717, 697, 617, 513 cm<sup>-1</sup>.

HRMS (ESI) *m/z* ([M+H]<sup>+</sup>) calcd for C<sub>19</sub>H<sub>29</sub>N<sub>6</sub>Na<sub>2</sub>O<sub>17</sub>P<sub>2</sub>: 721.0860, found: 721.0845.



**UDP-Sugar (S)-8.** The title compound was prepared from diallyl 3,4,6-tri-*O*-acetyl-2-((*S*)-3-azido-2-methylpropanamido)-2-deoxy- $\alpha$ -D-galactopyranosyl-1-phosphate (48.3 mg, 0.0838 mmol). The tri-*O*-acetylated UDP-sugar was purified by column chromatography on C-18 silica gel (0% $\rightarrow$ 100% MeOH/water; water was doped with 10 mM *n*-Bu<sub>3</sub>NH•HCO<sub>3</sub>) and then preparative HPLC on C-18 silica gel (40% $\rightarrow$ 60% MeOH/water; water was doped with 10 mM *n*-Bu<sub>3</sub>NH•HCO<sub>3</sub>). The compound was dissolved in MeOH/water/NEt<sub>3</sub> (5:2:1), and the reaction mixture was stirred at r.t. overnight. The product was purified by preparative HPLC on an Agilent prep 100 Å C18 column (MeOH/water (min:%): 0:10, 10:10, 60:60; water was doped with 10 mM *n*-Bu<sub>3</sub>NH•HCO<sub>3</sub>; 20 mL/min; t<sub>r</sub> = 41.2 min): 14.4 mg (24%). White solid.



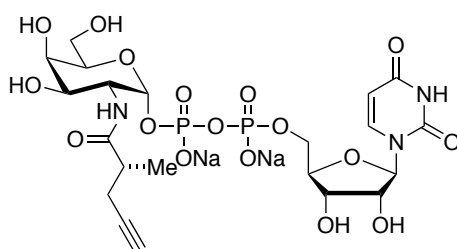
$^1\text{H}$  NMR (500 MHz,  $\text{D}_2\text{O}$ )  $\delta$  7.96 (d,  $J = 8.1$  Hz, 1H), 5.99–5.95 (m, 2H), 5.54 (dd,  $J = 7.1, 3.4$  Hz, 1H), 4.39–4.34 (m, 2H), 4.31–4.16 (m, 5H), 4.04 (d,  $J = 3.2$  Hz, 1H), 3.98 (dd,  $J = 10.9, 2.9$  Hz, 1H), 3.80–3.69 (m, 2H), 3.49 (dd,  $J = 12.3, 9.1$  Hz, 1H), 3.41 (dd,  $J = 12.4, 5.4$  Hz, 1H), 2.86–2.77 (m, 1H), 1.16 (d,  $J = 7.0$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{D}_2\text{O}$ )  $\delta$  178.8, 167.1, 152.6, 142.3, 103.3, 95.4 (d,  $J = 6.1$  Hz), 89.2, 83.9 (d,  $J = 9.3$  Hz), 74.4, 72.7, 70.3, 69.1, 68.2, 65.6 (d,  $J = 4.1$  Hz), 61.7, 54.2, 50.4 (d,  $J = 8.2$  Hz), 41.0, 15.4.

$^{31}\text{P}$  NMR (202 MHz,  $\text{D}_2\text{O}$ )  $\delta$  -10.5 (d,  $J = 20.8$  Hz), -12.3 (d,  $J = 21.2$  Hz).

FT-IR (neat) 3276, 2106, 1673, 1548, 1465, 1427, 1392, 1347, 1235, 1115, 1079, 1058, 1035, 986, 919, 814, 782, 768, 714, 696, 643, 625, 509  $\text{cm}^{-1}$ .

HRMS (ESI)  $m/z$  ( $[\text{M}+\text{H}]^+$ ) calcd for  $\text{C}_{19}\text{H}_{29}\text{N}_6\text{Na}_2\text{O}_{17}\text{P}_2$ : 721.0860, found: 721.0853.



**UDP-Sugar (R)-12.** The title compound was prepared from diallyl 3,4,6-tri-*O*-acetyl-2-((*R*)-2-methylpent-4-ynamido)-2-deoxy- $\alpha$ -D-galactopyranosyl-1-phosphate (64.6 mg, 0.115 mmol). The tri-*O*-acetylated UDP-sugar was purified by column chromatography on C-18 silica gel (0% $\rightarrow$ 100% MeOH/water; water was doped with 10 mM *n*- $\text{Bu}_3\text{NH}\cdot\text{HCO}_3$ ) and then preparative HPLC on C-18 silica gel (30% $\rightarrow$ 60% MeOH/water; water was doped with 10 mM *n*- $\text{Bu}_3\text{NH}\cdot\text{HCO}_3$ ). The compound was dissolved in MeOH/water/ $\text{NEt}_3$  (5:2:1), and the reaction mixture was stirred at r.t. overnight. The product was purified by preparative HPLC on an Agilent prep 100  $\text{\AA}$  C18 column (MeOH/water (min:%): 0:10, 10:10, 60:60; water was doped with 10 mM *n*- $\text{Bu}_3\text{NH}\cdot\text{HCO}_3$ ; 20 mL/min;  $t_r = 38.8$  min): 44.8 mg (55%). White solid.

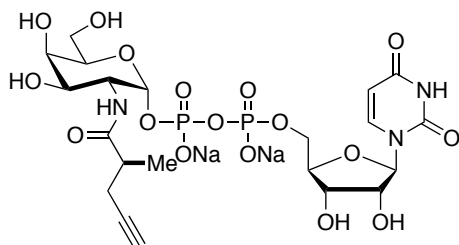
$^1\text{H}$  NMR (500 MHz,  $\text{D}_2\text{O}$ )  $\delta$  7.94 (d,  $J = 8.0$  Hz, 1H), 5.97–5.93 (m, 2H), 5.51 (dd,  $J = 6.5, 3.4$  Hz, 1H), 4.37–4.33 (m, 2H), 4.30–4.15 (m, 5H), 4.03 (d,  $J = 3.0$  Hz, 1H), 3.96 (dd,  $J = 11.0, 3.2$  Hz, 1H), 3.78–3.69 (m, 2H), 2.75 (apparent sextet,  $J = 7.0$  Hz, 1H), 2.47–2.31 (m, 3H), 1.18 (d,  $J = 6.9$  Hz, 3H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{D}_2\text{O}$ )  $\delta$  179.7, 167.7, 153.0, 142.2, 103.3, 95.7 (d,  $J = 6.5$  Hz), 89.1, 83.8 (d,  $J = 9.5$  Hz), 83.2, 74.4, 72.7, 71.6, 70.3, 69.1, 68.2, 65.6 (d,  $J = 4.6$  Hz), 61.6, 50.2 (d,  $J = 9.0$  Hz), 40.2, 23.0, 17.1.

$^{31}\text{P}$  NMR (202 MHz,  $\text{D}_2\text{O}$ )  $\delta$  -10.5 (d,  $J = 20.8$  Hz), -12.5 (d,  $J = 20.8$  Hz).

FT-IR (neat) 3274, 1672, 1539, 1463, 1428, 1394, 1339, 1241, 1117, 1078, 1054, 986, 919, 814, 780, 714, 690, 655, 628, 513  $\text{cm}^{-1}$ .

HRMS (ESI)  $m/z$  ( $[\text{M}+\text{H}]^+$ ) calcd for  $\text{C}_{21}\text{H}_{30}\text{N}_3\text{Na}_2\text{O}_{17}\text{P}_2$ : 704.0846, found: 704.0848.



**UDP-Sugar (S)-12.** The title compound was prepared from diallyl 3,4,6-tri-*O*-acetyl-2-((*S*)-2-methylpent-4-ynamido)-2-deoxy- $\alpha$ -D-galactopyranosyl-1-phosphate (59.0 mg, 0.105 mmol). The tri-*O*-acetylated UDP-sugar was purified by column chromatography on C-18 silica gel (0% $\rightarrow$ 100% MeOH/water; water was doped with 10 mM *n*-Bu<sub>3</sub>NH•HCO<sub>3</sub>) and then preparative HPLC on C-18 silica gel (30% $\rightarrow$ 60% MeOH/water; water was doped with 10 mM *n*-Bu<sub>3</sub>NH•HCO<sub>3</sub>). The compound was dissolved in MeOH/water/NEt<sub>3</sub> (5:2:1), and the reaction mixture was stirred at r.t. overnight. The product was purified by preparative HPLC on an Agilent prep 100 Å C18 column (MeOH/water (min:%): 0:10, 10:10, 60:60; water was doped with 10 mM *n*-Bu<sub>3</sub>NH•HCO<sub>3</sub>; 20 mL/min; t<sub>r</sub> = 39.1 min): 38.0 mg (51%). White solid.

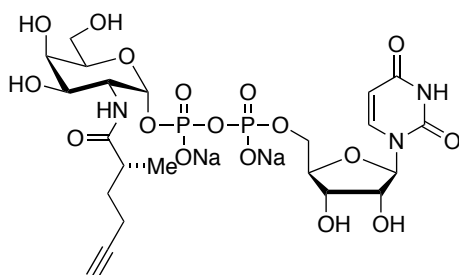
<sup>1</sup>H NMR (500 MHz, D<sub>2</sub>O)  $\delta$  7.93 (d, *J* = 8.1 Hz, 1H), 5.97–5.93 (m, 2H), 5.52 (dd, *J* = 7.0, 3.4 Hz, 1H), 4.36–4.32 (m, 2H), 4.29–4.15 (m, 5H), 4.02 (d, *J* = 3.2 Hz, 1H), 3.95 (dd, *J* = 11.0, 3.2 Hz, 1H), 3.78–3.69 (m, 2H), 2.79–2.71 (m, 1H), 2.46–2.39 (m, 1H), 2.36–2.30 (m, 2H), 1.18 (d, *J* = 6.9 Hz, 3H).

<sup>13</sup>C NMR (126 MHz, D<sub>2</sub>O)  $\delta$  179.7, 167.8, 153.1, 142.2, 103.3, 95.4 (d, *J* = 6.4 Hz), 89.1, 83.8 (d, *J* = 9.1 Hz), 83.4, 74.4, 72.7, 71.2, 70.3, 69.1, 68.1, 65.6 (d, *J* = 5.3 Hz), 61.6, 50.3 (d, *J* = 8.6 Hz), 40.4, 22.9, 17.3.

<sup>31</sup>P NMR (202 MHz, D<sub>2</sub>O)  $\delta$  -10.6 (d, *J* = 20.8 Hz), -12.3 (d, *J* = 20.8 Hz).

FT-IR (neat) 3260, 1675, 1541, 1463, 1427, 1392, 1237, 1117, 1079, 1055, 988, 920, 860, 816, 717, 688, 664, 647, 623, 510 cm<sup>-1</sup>.

HRMS (ESI) *m/z* ([M+Na]<sup>+</sup>) calcd for C<sub>21</sub>H<sub>29</sub>N<sub>3</sub>Na<sub>3</sub>O<sub>17</sub>P<sub>2</sub>: 726.0665, found: 726.0660.



**UDP-Sugar (R)-14.** The title compound was prepared from diallyl 3,4,6-tri-*O*-acetyl-2-((*R*)-2-methylhex-5-ynamido)-2-deoxy- $\alpha$ -D-galactopyranosyl-1-phosphate (62.5 mg, 0.109 mmol). The tri-*O*-acetylated UDP-sugar was purified by column chromatography on C-18 silica gel (0% $\rightarrow$ 100% MeOH/water; water was doped with 10 mM *n*-Bu<sub>3</sub>NH•HCO<sub>3</sub>) and then preparative HPLC on C-18 silica gel (30% $\rightarrow$ 60% MeOH/water; water was doped with 10 mM *n*-Bu<sub>3</sub>NH•HCO<sub>3</sub>). The compound was dissolved in MeOH/water/NEt<sub>3</sub> (5:2:1), and the reaction mixture was stirred at r.t. overnight. The product was purified by preparative HPLC on an Agilent prep 100 Å C18 column

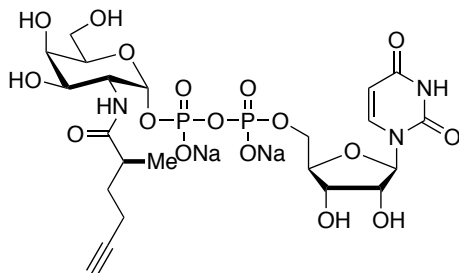
(MeOH/water (min: %): 0:10, 10:10, 60:60; water was doped with 10 mM *n*-Bu<sub>3</sub>NH•HCO<sub>3</sub>; 20 mL/min; t<sub>r</sub> = 42.8 min): 38.7 mg (50%). White solid.

<sup>1</sup>H NMR (500 MHz, D<sub>2</sub>O) δ 7.93 (d, *J* = 8.1 Hz, 1H), 5.98–5.93 (m, 2H), 5.52 (dd, *J* = 6.6, 3.4 Hz, 1H), 4.36–4.32 (m, 2H), 4.28–4.15 (m, 5H), 4.02 (d, *J* = 2.8 Hz, 1H), 3.95 (dd, *J* = 11.0, 3.2 Hz, 1H), 3.78–3.69 (m, 2H), 2.66–2.58 (m, 1H), 2.32 (t, *J* = 2.6 Hz, 1H), 2.28–2.13 (m, 2H), 1.84–1.75 (m, 1H), 1.64–1.56 (m, 1H), 1.12 (d, *J* = 6.9 Hz, 3H).  
<sup>13</sup>C NMR (126 MHz, D<sub>2</sub>O) δ 181.1, 167.6, 153.0, 142.3, 103.3, 95.6 (d, *J* = 6.7 Hz), 89.2, 85.7, 83.8 (d, *J* = 8.8 Hz), 74.4, 72.6, 70.2, 69.1, 68.3, 65.6 (d, *J* = 5.1 Hz), 61.6, 50.2 (d, *J* = 8.9 Hz), 40.5, 32.5, 17.7, 16.5.

<sup>31</sup>P NMR (202 MHz, D<sub>2</sub>O) δ –10.6 (d, *J* = 20.6 Hz), –12.5 (d, *J* = 20.6 Hz).

FT-IR (neat) 3266, 1674, 1538, 1465, 1430, 1392, 1349, 1234, 1117, 1079, 1057, 1038, 987, 918, 850, 813, 775, 687, 631, 511 cm<sup>-1</sup>.

MS (ESI) *m/z* ([M+H]<sup>+</sup>) calcd for C<sub>22</sub>H<sub>32</sub>N<sub>3</sub>Na<sub>2</sub>O<sub>17</sub>P<sub>2</sub>: 718.1002, found: 718.0994.



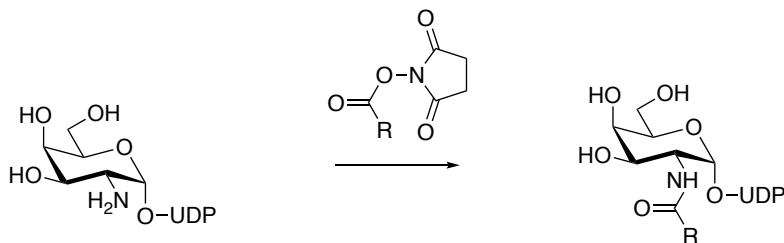
**UDP-Sugar (S)-14.** The title compound was prepared from diallyl 3,4,6-tri-*O*-acetyl-2-((*S*)-2-methylhex-5-ynamido)-2-deoxy- $\alpha$ -D-galactopyranosyl-1-phosphate (60.5 mg, 0.105 mmol). The tri-*O*-acetylated UDP-sugar was purified by column chromatography on C-18 silica gel (0%→100% MeOH/water; water was doped with 10 mM *n*-Bu<sub>3</sub>NH•HCO<sub>3</sub>) and then preparative HPLC on C-18 silica gel (30%→60% MeOH/water; water was doped with 10 mM *n*-Bu<sub>3</sub>NH•HCO<sub>3</sub>). The compound was dissolved in MeOH/water/NEt<sub>3</sub> (5:2:1), and the reaction mixture was stirred at r.t. overnight. The product was purified by preparative HPLC on an Agilent prep 100 Å C18 column (MeOH/water (min: %): 0:10, 10:10, 60:60; water was doped with 10 mM *n*-Bu<sub>3</sub>NH•HCO<sub>3</sub>; 20 mL/min; t<sub>r</sub> = 39.7 min): 35.8 mg (47%). White solid.

<sup>1</sup>H NMR (500 MHz, D<sub>2</sub>O) δ 7.93 (d, *J* = 8.1 Hz, 1H), 5.98–5.92 (m, 2H), 5.52 (dd, *J* = 6.9, 3.4 Hz, 1H), 4.37–4.32 (m, 2H), 4.28–4.14 (m, 5H), 4.01 (d, *J* = 3.2 Hz, 1H), 3.94 (dd, *J* = 11.0, 3.2 Hz, 1H), 3.78–3.69 (m, 2H), 2.70–2.62 (m, 1H), 2.33 (t, *J* = 2.7 Hz, 1H), 2.26–2.13 (m, 2H), 1.81–1.72 (m, 1H), 1.65–1.57 (m, 1H), 1.12 (d, *J* = 6.8 Hz, 3H).  
<sup>13</sup>C NMR (126 MHz, D<sub>2</sub>O) δ 181.0, 167.0, 152.5, 142.3, 103.3, 95.4 (d, *J* = 6.5 Hz), 89.1, 85.7, 83.9 (d, *J* = 9.1 Hz), 74.4, 72.6, 70.3, 70.2, 69.2, 68.1, 65.6 (d, *J* = 5.1 Hz), 61.6, 50.2 (d, *J* = 8.6 Hz), 40.3, 32.7, 17.6, 16.3.

<sup>31</sup>P NMR (202 MHz, D<sub>2</sub>O) δ –10.6 (d, *J* = 20.6 Hz), –12.3 (d, *J* = 20.8 Hz).

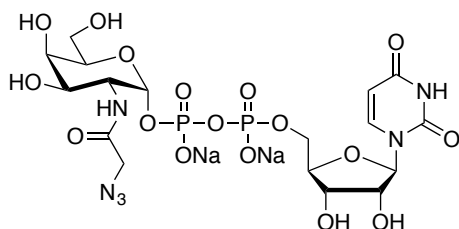
FT-IR (neat) 3252, 1674, 1540, 1463, 1430, 1393, 1350, 1235, 1115, 1078, 1058, 1036, 986, 920, 851, 814, 779, 691, 624, 511 cm<sup>-1</sup>.

HRMS (ESI) *m/z* ([M+H]<sup>+</sup>) calcd for C<sub>22</sub>H<sub>32</sub>N<sub>3</sub>Na<sub>2</sub>O<sub>17</sub>P<sub>2</sub>: 718.1002, found: 718.0995.



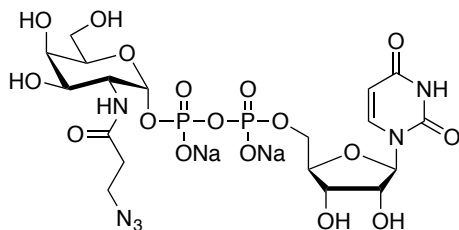
### Preparation of UDP-*N*-acetyl- $\alpha$ -D-galactosamine derivatives (Route 2).

NHS esters were prepared according to a literature procedure.<sup>21</sup> A solution of the NHS ester (0.150 mmol) in DMF (1.08 mL) was added to a mixture of UDP- $\alpha$ -D-galactosamine disodium salt (30.5 mg, 0.0500 mmol) in HEPES buffer (0.1 M, pH = 8.0; 1.08 mL) at 0 °C.<sup>22</sup> The reaction mixture was allowed to warm to r.t. and stirred overnight. Next, the mixture was purified by column chromatography on C-18 silica gel (MeOH/water; water was doped with 10 mM *n*-Bu<sub>3</sub>NH•HCO<sub>3</sub>). Aqueous *n*-Bu<sub>3</sub>NH•HCO<sub>3</sub> solution (10 mM) was prepared by bubbling CO<sub>2</sub> through a solution of *n*-Bu<sub>3</sub>NH (10 mM) in water until all *n*-Bu<sub>3</sub>NH dissolved into the water. Fractions containing the sugar were collected, concentrated, redissolved in water (100 mL), rinsed with CH<sub>2</sub>Cl<sub>2</sub> (3 x 50 mL), and concentrated. The product was purified by preparative HPLC on C-18 silica gel (MeOH/water; water was doped with 10 mM *n*-Bu<sub>3</sub>NH•HCO<sub>3</sub>). Pure fractions were collected, concentrated, redissolved in water (100 mL), rinsed with CH<sub>2</sub>Cl<sub>2</sub> (3 x 50 mL), and concentrated. Finally, the purified compound was passed through a Bio-Rad AG<sup>®</sup> 50W-X8 resin (sodium form) and lyophilized.



**UDP-GalNAz 2.** The title compound was prepared from UDP- $\alpha$ -D-galactosamine ditributylammonium salt (41.7 mg, 0.0445 mmol) and 2,5-dioxopyrrolidin-1-yl 2-azidoacetate (71.0 mg, 0.358 mmol). The product was purified by column chromatography on C-18 silica gel (0%→100% MeOH/water; water was doped with 10 mM *n*-Bu<sub>3</sub>NH•HCO<sub>3</sub>) and then preparative HPLC on an Agilent prep 100 Å C18 column (MeOH/water (min:%): 0:10, 10:10, 60:60; water was doped with 10 mM *n*-Bu<sub>3</sub>NH•HCO<sub>3</sub>; 20 mL/min; *t*<sub>r</sub> = 37.5 min): 10.8 mg (35%). White solid.  
<sup>1</sup>H NMR (500 MHz, D<sub>2</sub>O)  $\delta$  7.91 (d, *J* = 8.1 Hz, 1H), 5.96 (d, *J* = 4.2 Hz, 1H), 5.93 (d, *J* = 8.0 Hz, 1H), 5.55 (dd, *J* = 7.2, 3.4 Hz, 1H), 4.37–4.31 (m, 2H), 4.31–4.05 (m, 7H), 4.02 (d, *J* = 2.6 Hz, 1H), 3.96 (dd, *J* = 10.9, 2.9 Hz, 1H), 3.79–3.69 (m, 2H).  
<sup>13</sup>C NMR (126 MHz, D<sub>2</sub>O)  $\delta$  171.8, 167.7, 153.0, 142.2, 103.4, 95.2 (d, *J* = 6.3 Hz), 89.3, 83.8 (d, *J* = 9.0 Hz), 74.4, 72.8, 70.3, 69.1, 68.2, 65.7 (d, *J* = 5.5 Hz), 61.7, 52.3, 50.6 (d, *J* = 8.4 Hz).  
<sup>31</sup>P NMR (202 MHz, D<sub>2</sub>O)  $\delta$  -10.6 (d, *J* = 20.8 Hz), -12.2 (d, *J* = 20.6 Hz).  
 FT-IR (neat) 3251, 2113, 1672, 1549, 1465, 1426, 1233, 1111, 1080, 1042, 987, 923, 853, 815, 719, 690, 623, 512 cm<sup>-1</sup>.

MS (ESI)  $m/z$  ( $[M-2Na+H]^-$ ) calcd for  $C_{17}H_{25}N_6O_{17}P_2$ : 647.0751, found: 647.0738.



**UDP-Sugar 7.** The title compound was prepared from UDP- $\alpha$ -D-galactosamine disodium salt and 2,5-dioxopyrrolidin-1-yl 3-azidopropanoate. The product was purified by preparative HPLC on an Agilent prep 100 Å C18 column (MeOH/water (min:%): 0:10, 10:10, 60:60; water was doped with 10 mM  $n$ -Bu<sub>3</sub>NH•HCO<sub>3</sub>; 20 mL/min;  $t_r$  = 42.1 min). White solid.

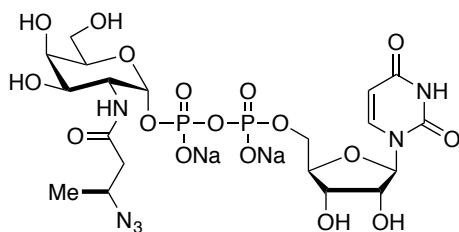
<sup>1</sup>H NMR (500 MHz, D<sub>2</sub>O)  $\delta$  7.92 (d,  $J$  = 8.1 Hz, 1H), 5.97–5.92 (m, 2H), 5.53 (dd,  $J$  = 7.1, 3.5 Hz, 1H), 4.36–4.31 (m, 2H), 4.30–4.13 (m, 5H), 4.02 (d,  $J$  = 3.1 Hz, 1H), 3.94 (dd,  $J$  = 10.9, 3.1 Hz, 1H), 3.78–3.69 (m, 2H), 3.58 (t,  $J$  = 6.4 Hz, 2H), 2.69–2.58 (m, 2H).

<sup>13</sup>C NMR (126 MHz, D<sub>2</sub>O)  $\delta$  175.0, 167.5, 152.9, 142.2, 103.3, 95.4 (d,  $J$  = 6.3 Hz), 89.2, 83.8 (d,  $J$  = 9.1 Hz), 74.4, 72.8, 70.3, 69.1, 68.3, 65.7 (d,  $J$  = 5.5 Hz), 61.7, 50.4 (d,  $J$  = 8.5 Hz), 47.7, 35.5.

<sup>31</sup>P NMR (202 MHz, D<sub>2</sub>O)  $\delta$  -10.6 (d,  $J$  = 21.4 Hz), -12.2 (d,  $J$  = 20.6 Hz).

FT-IR (neat) 3259, 2100, 1672, 1547, 1463, 1424, 1389, 1345, 1234, 1114, 1080, 1049, 985, 919, 851, 812, 767, 717, 692, 636, 620, 509 cm<sup>-1</sup>.

MS (ESI)  $m/z$  ( $[M+H]^+$ ) calcd for  $C_{18}H_{27}N_6Na_2O_{17}P_2$ : 707.0703, found: 707.0706.



**UDP-Sugar 9.** The title compound was prepared from UDP- $\alpha$ -D-galactosamine disodium salt and 2,5-dioxopyrrolidin-1-yl (*S*)-3-azidobutanoate. The product was purified by preparative HPLC on an Agilent prep 100 Å C18 column (MeOH/water (min:%): 0:10, 10:10, 60:60; water was doped with 10 mM  $n$ -Bu<sub>3</sub>NH•HCO<sub>3</sub>; 20 mL/min;  $t_r$  = 45.1 min). White solid.

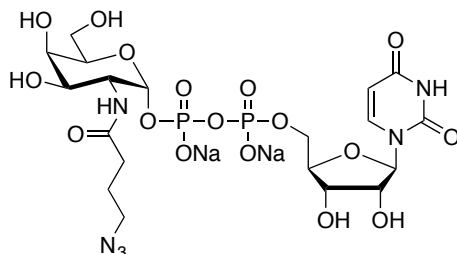
<sup>1</sup>H NMR (500 MHz, D<sub>2</sub>O)  $\delta$  7.93 (d,  $J$  = 8.1 Hz, 1H), 5.97–5.92 (m, 2H), 5.53 (dd,  $J$  = 7.1, 3.5 Hz, 1H), 4.36–4.31 (m, 2H), 4.28–4.13 (m, 5H), 4.02 (d,  $J$  = 3.0 Hz, 1H), 4.00–3.91 (m, 2H), 3.78–3.69 (m, 2H), 2.58–2.49 (m, 2H), 1.28 (d,  $J$  = 6.6 Hz, 3H).

<sup>13</sup>C NMR (126 MHz, D<sub>2</sub>O)  $\delta$  174.6, 167.5, 152.9, 142.3, 103.3, 95.3 (d,  $J$  = 6.3 Hz), 89.2, 83.8 (d,  $J$  = 9.1 Hz), 74.4, 72.7, 70.3, 69.1, 68.2, 65.7 (d,  $J$  = 5.3 Hz), 61.7, 55.7, 50.4 (d,  $J$  = 8.3 Hz), 42.7, 19.3.

<sup>31</sup>P NMR (202 MHz, D<sub>2</sub>O)  $\delta$  -10.6 (d,  $J$  = 21.4 Hz), -12.2 (d,  $J$  = 21.2 Hz).

FT-IR (neat) 3219, 2112, 1673, 1546, 1462, 1424, 1387, 1342, 1233, 1112, 1080, 1040, 986, 917, 852, 813, 707, 688, 620, 509  $\text{cm}^{-1}$ .

MS (ESI)  $m/z$  ( $[M+H]^+$ ) calcd for  $\text{C}_{19}\text{H}_{29}\text{N}_6\text{Na}_2\text{O}_{17}\text{P}_2$ : 721.0860, found: 721.0858.



**UDP-Sugar 10.** The title compound was prepared from UDP- $\alpha$ -D-galactosamine disodium salt (12.6 mg, 0.0207 mmol) and 2,5-dioxopyrrolidin-1-yl 4-azidobutanoate (14.0 mg, 0.0619 mmol). The product was purified by column chromatography on C-18 silica gel (0% $\rightarrow$ 100% MeOH/water; water was doped with 10 mM  $n\text{-Bu}_3\text{NH}\cdot\text{HCO}_3$ ) and then preparative HPLC on an Agilent prep 100 Å C18 column (MeOH/water (min.%): 0:10, 10:10, 60:60; water was doped with 10 mM  $n\text{-Bu}_3\text{NH}\cdot\text{HCO}_3$ ; 20 mL/min;  $t_r$  = 41.3 min): 7.4 mg (50%). White solid.

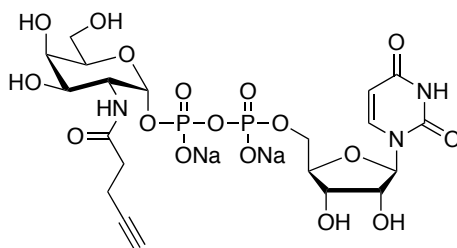
$^1\text{H}$  NMR (500 MHz,  $\text{D}_2\text{O}$ )  $\delta$  7.93 (d,  $J$  = 8.1 Hz, 1H), 5.99–5.92 (m, 2H), 5.52 (dd,  $J$  = 7.2, 3.3 Hz, 1H), 4.37–4.31 (m, 2H), 4.29–4.13 (m, 5H), 4.01 (d,  $J$  = 3.2 Hz, 1H), 3.94 (dd,  $J$  = 11.0, 2.8 Hz, 1H), 3.78–3.68 (m, 2H), 3.35 (t,  $J$  = 6.9 Hz, 2H), 2.42 (t,  $J$  = 7.5 Hz, 2H), 1.87 (p,  $J$  = 7.2 Hz, 2H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{D}_2\text{O}$ )  $\delta$  177.3, 168.1, 153.3, 142.2, 103.4, 95.4 (d,  $J$  = 6.4 Hz), 89.2, 83.81 (d,  $J$  = 9.2 Hz), 74.5, 72.7, 70.3, 69.1, 68.3, 65.7 (d,  $J$  = 5.3 Hz), 61.7, 51.1, 50.4 (d,  $J$  = 8.3 Hz), 33.6, 25.2.

$^{31}\text{P}$  NMR (202 MHz,  $\text{D}_2\text{O}$ )  $\delta$  -10.6 (d,  $J$  = 21.4 Hz), -12.2 (d,  $J$  = 20.6 Hz).

FT-IR (neat) 3263, 2106, 1671, 1546, 1464, 1425, 1388, 1351, 1231, 1112, 1079, 1053, 1037, 985, 918, 851, 812, 719, 688, 623, 510  $\text{cm}^{-1}$ .

MS (ESI)  $m/z$  ( $[M+H]^+$ ) calcd for  $\text{C}_{19}\text{H}_{29}\text{N}_6\text{Na}_2\text{O}_{17}\text{P}_2$ : 721.0860, found: 721.0862.



**UDP-Sugar 11.** The title compound was prepared from UDP- $\alpha$ -D-galactosamine disodium salt (50.0 mg, 0.0821 mmol) and 2,5-dioxopyrrolidin-1-yl pent-4-ynoate (48.0 mg, 0.246 mmol). The product was purified by column chromatography on C-18 silica gel (0% $\rightarrow$ 100% MeOH/water; water was doped with 10 mM  $n\text{-Bu}_3\text{NH}\cdot\text{HCO}_3$ ) and then preparative HPLC on an Agilent prep 100 Å C18 column (MeOH/water (min.%): 0:10, 10:10, 60:60; water was doped with 10 mM  $n\text{-Bu}_3\text{NH}\cdot\text{HCO}_3$ ; 20 mL/min;  $t_r$  = 43.5 min): 29.5 mg (52%). White solid.

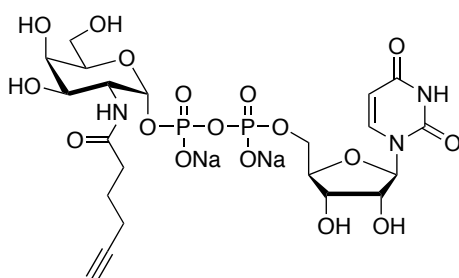
$^1\text{H}$  NMR (500 MHz,  $\text{D}_2\text{O}$ )  $\delta$  7.95 (d,  $J = 8.1$  Hz, 1H), 5.99–5.93 (m, 2H), 5.53 (dd,  $J = 7.1, 3.4$  Hz, 1H), 4.37–4.32 (m, 2H), 4.30–4.14 (m, 5H), 4.02 (d,  $J = 3.1$  Hz, 1H), 3.95 (dd,  $J = 11.0, 3.0$  Hz, 1H), 3.79–3.69 (m, 2H), 2.63–2.51 (m, 2H), 2.51–2.45 (m, 2H), 2.34 (t,  $J = 2.3$  Hz, 1H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{D}_2\text{O}$ )  $\delta$  176.0, 167.0, 152.5, 142.3, 103.3, 95.5 (d,  $J = 6.5$  Hz), 89.2, 84.3, 83.8 (d,  $J = 9.1$  Hz), 74.4, 72.7, 70.6, 70.3, 69.1, 68.2, 65.6 (d,  $J = 5.3$  Hz), 61.7, 50.4 (d,  $J = 8.7$  Hz), 35.0, 15.0.

$^{31}\text{P}$  NMR (202 MHz,  $\text{D}_2\text{O}$ )  $\delta$  -10.6 (d,  $J = 20.8$  Hz), -12.3 (d,  $J = 20.6$  Hz).

FT-IR (neat) 3268, 1671, 1547, 1469, 1423, 1391, 1234, 1113, 1080, 1045, 986, 919, 852, 814, 765, 712, 687, 626, 552, 514  $\text{cm}^{-1}$ .

MS (ESI)  $m/z$  ( $[\text{M}+\text{H}]^+$ ) calcd for  $\text{C}_{20}\text{H}_{28}\text{N}_3\text{Na}_2\text{O}_{17}\text{P}_2$ : 690.0689, found: 690.0677.



**UDP-Sugar 13.** The title compound was prepared from UDP- $\alpha$ -D-galactosamine disodium salt (50.0 mg, 0.0821 mmol) and 2,5-dioxopyrrolidin-1-yl hex-5-ynoate (51.4 mg, 0.246 mmol). The product was purified by column chromatography on C-18 silica gel (0%→100% MeOH/water; water was doped with 10 mM  $n\text{-Bu}_3\text{NH}\cdot\text{HCO}_3$ ) and then preparative HPLC on an Agilent prep 100 Å C18 column (MeOH/water (min:%): 0:10, 10:10, 60:60; water was doped with 10 mM  $n\text{-Bu}_3\text{NH}\cdot\text{HCO}_3$ ; 20 mL/min;  $t_r = 38.2$  min): 25.2 mg (44%). White solid.

$^1\text{H}$  NMR (500 MHz,  $\text{D}_2\text{O}$ )  $\delta$  7.94 (dd,  $J = 8.2, 1.4$  Hz, 1H), 5.98–5.92 (m, 2H), 5.52 (dd,  $J = 7.2, 3.4$  Hz, 1H), 4.37–4.31 (m, 2H), 4.29–4.13 (m, 5H), 4.02 (d,  $J = 3.1$  Hz, 1H), 3.94 (dd,  $J = 10.8, 2.2$  Hz, 1H), 3.79–3.68 (m, 2H), 2.44 (t,  $J = 7.4$  Hz, 2H), 2.34 (t,  $J = 2.6$  Hz, 1H), 2.26–2.21 (m, 2H), 1.80 (p,  $J = 7.4$  Hz, 2H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{D}_2\text{O}$ )  $\delta$  177.6, 167.0, 152.5, 142.3, 103.3, 95.4 (d,  $J = 6.5$  Hz), 89.1, 85.6, 83.8 (d,  $J = 9.3$  Hz), 74.4, 72.7, 70.4, 70.3, 69.1, 68.3, 65.6 (d,  $J = 5.3$  Hz), 61.7, 50.3 (d,  $J = 8.6$  Hz), 35.3, 24.9, 17.8.

$^{31}\text{P}$  NMR (202 MHz,  $\text{D}_2\text{O}$ )  $\delta$  -10.6 (d,  $J = 21.4$  Hz), -12.2 (d,  $J = 20.6$  Hz).

FT-IR (neat) 3274, 1676, 1547, 1467, 1425, 1392, 1345, 1234, 1113, 1080, 1054, 1042, 986, 919, 852, 813, 768, 715, 687, 652, 627, 512  $\text{cm}^{-1}$ .

MS (ESI)  $m/z$  ( $[\text{M}+\text{H}]^+$ ) calcd for  $\text{C}_{21}\text{H}_{30}\text{N}_3\text{Na}_2\text{O}_{17}\text{P}_2$ : 704.0846, found: 704.0836.

## X. References

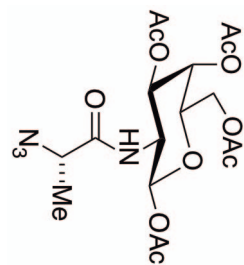
- (1) Kubota, T.; Shiba, T.; Sugioka, S.; Furukawa, S.; Sawaki, H.; Kato, R.; Wakatsuki, S.; Narimatsu, H. Structural Basis of Carbohydrate Transfer Activity by Human UDP-GalNAc: Polypeptide  $\alpha$ -N-Acetylgalactosaminyltransferase (pp-GalNAc-T10). *J. Mol. Biol.* **2006**, 359 (3), 708–727.

- (2) Lira-Navarrete, E.; Iglesias-Fernández, J.; Zandberg, W. F.; Compañón, I.; Kong, Y.; Corzana, F.; Pinto, B. M.; Clausen, H.; Peregrina, J. M.; Vocadlo, D. J.; Rovira, C.; Hurtado-Guerrero, R. Substrate-Guided Front-Face Reaction Revealed by Combined Structural Snapshots and Metadynamics for the Polypeptide N-Acetylgalactosaminyltransferase 2. *Angew. Chem., Int. Ed.* **2014**, *53* (31), 8206–8210.
- (3) Chojnacki, S.; Cowley, A.; Lee, J.; Foix, A.; Lopez, R. Programmatic Access to Bioinformatics Tools from EMBL-EBI Update: 2017. *Nucleic Acids Res.* **2017**, *45* (W1), W550–W553.
- (4) Bennett, E. P.; Mandel, U.; Clausen, H.; Gerken, T. A.; Fritz, T. A.; Tabak, L. A. Control of Mucin-Type O-Glycosylation: A Classification of the Polypeptide GalNAc-Transferase Gene Family. *Glycobiology* **2012**, *22* (6), 736–756.
- (5) Guo, H.; Zhao, L.; Shi, B.; Bao, J.; Zheng, D.; Zhou, B.; Shi, J. GALNT5 uaRNA Promotes Gastric Cancer Progression through Its Interaction with HSP90. *Oncogene* **2018**, *37* (33), 4505–4517.
- (6) White, T.; Bennett, E. P.; Takio, K.; Sørensen, T.; Bonding, N.; Clausen, H. Purification and cDNA Cloning of a Human UDP-*N*-Acetyl- $\alpha$ -D-Galactosamine:Polypeptide *N*-Acetylgalactosaminyltransferase. *J. Biol. Chem.* **1995**, *270* (41), 24156–24165.
- (7) Wandall, H. H.; Hassan, H.; Mirgorodskaya, E.; Kristensen, A. K.; Roepstorff, P.; Bennett, E. P.; Nielsen, P. A.; Hollingsworth, M. A.; Burchell, J.; Taylor-Papadimitriou, J.; Clausen, H. Substrate Specificities of Three Members of the Human UDP-*N*-Acetyl- $\alpha$ -D-Galactosamine:Polypeptide *N*-Acetylgalactosaminyltransferase Family, GalNAc-T1, -T2, and -T3. *J. Biol. Chem.* **1997**, *272* (38), 23503–23514.
- (8) Homa, F. L.; Hollander, T.; Lehman, D. J.; Thomsen, D. R.; Elhammer, A. P. Isolation and Expression of a cDNA Clone Encoding a Bovine UDP-GalNAc:Polypeptide *N*-Acetylgalactosaminyltransferase. *J. Biol. Chem.* **1993**, *268* (17), 12609–12616.
- (9) Cheng, L.; Tachibana, K.; Zhang, Y.; Guo, J.; Tachibana, K. K.; Kameyama, A.; Wang, H.; Hiruma, T.; Iwasaki, H.; Togayachi, A.; Kudo, T.; Narimatsu, H. Characterization of a Novel Human UDP-GalNAc Transferase, pp-GalNAc-T10. *FEBS Lett.* **2002**, *531* (2), 115–121.
- (10) Zheng, L.; Baumann, U.; Reymon, J.-L. An Efficient One-Step Site-Directed and Site-Saturation Mutagenesis Protocol. *Nucleic Acids Res.* **2004**, *32* (14), e115.
- (11) Sibbersen, C.; Palmfeldt, J.; Hansen, J.; Gregersen, N.; Jørgensen, K. A.; Johannsen, M. Development of a Chemical Probe for Identifying Protein Targets of  $\alpha$ -Oxoaldehydes. *Chem. Commun.* **2013**, *49* (38), 4012–4014.
- (12) Pettigrew, J. D.; Freeman, R. P.; Wilson, P. D. Total Synthesis of (–)-Xyloketal D and Its Enantiomer—Confirmation of Absolute Stereochemistry. *Can. J. Chem.* **2005**, *82* (11), 1640–1648.
- (13) Smith, S. M.; Takacs, J. M. Amide-Directed Catalytic Asymmetric Hydroboration of Trisubstituted Alkenes. *J. Am. Chem. Soc.* **2010**, *132* (6), 1740–1741.
- (14) Studte, C.; Breit, B. Zinc-Catalyzed Enantiospecific  $sp^3$ – $sp^3$  Cross-Coupling of  $\alpha$ -Hydroxy Ester Triflates with Grignard Reagents. *Angew. Chem., Int. Ed.* **2008**, *47* (29), 5451–5455.



- (15) Agarwal, K.; Kaul, R.; Garg, M.; Shajahan, A.; Jha, S. K.; Sampathkumar, S.-G. Inhibition of Mucin-Type O-Glycosylation through Metabolic Processing and Incorporation of *N*-Thioglycolyl-D-Galactosamine Peracetate (Ac<sub>5</sub>GalNTGc). *J. Am. Chem. Soc.* **2013**, *135* (38), 14189–14197.
- (16) Lundquist, J. T., IV; Pelletier, J. C. Improved Solid-Phase Peptide Synthesis Method Utilizing  $\alpha$ -Azide-Protected Amino Acids. *Org. Lett.* **2001**, *3* (5), 781–783.
- (17) Rej, R.; Nguyen, D.; Go, B.; Fortin, S.; Lavallée, J.-F. Total Synthesis of Cryptophycins and Their 16-(3-Phenylacryloyl) Derivatives. *J. Org. Chem.* **1996**, *61* (18), 6289–6295.
- (18) Andersen, S. M.; Heuckendorff, M.; Jensen, H. H. 3-(Dimethylamino)-1-Propylamine: A Cheap and Versatile Reagent for Removal of Byproducts in Carbohydrate Chemistry. *Org. Lett.* **2015**, *17* (4), 944–947.
- (19) Beahm, B. J.; Dehnert, K. W.; Derr, N. L.; Kuhn, J.; Eberhart, J. K.; Spillmann, D.; Amacher, S. L.; Bertozzi, C. R. A Visualizable Chain-Terminating Inhibitor of Glycosaminoglycan Biosynthesis in Developing Zebrafish. *Angew. Chem., Int. Ed.* **2014**, *53* (13), 3347–3352.
- (20) Tsukamoto, H.; Kahne, D. *N*-Methylimidazolium Chloride-Catalyzed Pyrophosphate Formation: Application to the Synthesis of Lipid I and NDP-Sugar Donors. *Bioorg. Med. Chem. Lett.* **2011**, *21* (17), 5050–5053.
- (21) Zhang, Z.; Hejesen, C.; Kjelstrup, M. B.; Birkedal, V.; Gothelf, K. V. A DNA-Mediated Homogeneous Binding Assay for Proteins and Small Molecules. *J. Am. Chem. Soc.* **2014**, *136* (31), 11115–11120.
- (22) Masuko, S.; Bera, S.; Green, D. E.; Weïwer, M.; Liu, J.; DeAngelis, P. L.; Linhardt, R. J. Chemoenzymatic Synthesis of Uridine Diphosphate-GlcNAc and Uridine Diphosphate-GalNAc Analogs for the Preparation of Unnatural Glycosaminoglycans. *J. Org. Chem.* **2012**, *77* (3), 1449–1456.

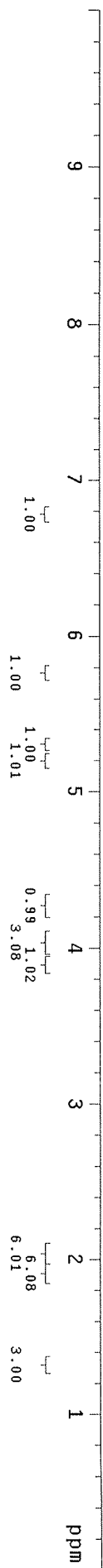
# XI. NMR Spectra



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i41 n
i42 n
i43 n
i44 n
i45 n
i46 n
i47 n
i48 n
i49 n
i50 n
i51 n
i52 n
i53 n
i54 n
i55 n
i56 n
i57 n
i58 n
i59 n
i60 n
i61 n
i62 n
i63 n
i64 n
i65 n
i66 n
i67 n
i68 n
i69 n
i70 n
i71 n
i72 n
i73 n
i74 n
i75 n
i76 n
i77 n
i78 n
i79 n
i80 n
i81 n
i82 n
i83 n
i84 n
i85 n
i86 n
i87 n
i88 n
i89 n
i90 n
i91 n
i92 n
i93 n
i94 n
i95 n
i96 n
i97 n
i98 n
i99 n
i100 n

```



JCS131\_13C\_CDC13

exp2 s2pul1

SAMPLE DEC. & VT

date Jun 14 2017 dn H1

solvent CDC13 dof -499.0

file exp dm yyy

ACQUISITION 125.674 dmm 11400

sfrq C13 dpwr 43

tn 1.500 PROCESSING 2.00

at 99016 1b fn not used f

np 33003.3 math

fb 18000 4

bs 7.0 weff

pw 7.0 wexp

tpwr 51 wds

di 0.500 wnt

tof 100.0 sp

nt 1024 wp

ct 36 vs

atock n SC

gain 54 WC

11 n hzmm

1n n ts

1p y rffl

1s n rffl

1t th ins

1u ai

DISPLAY

-4500.1

33002.8

2567 0

250

5.03

500.00

14196.7

96936.1

34

800

169.33

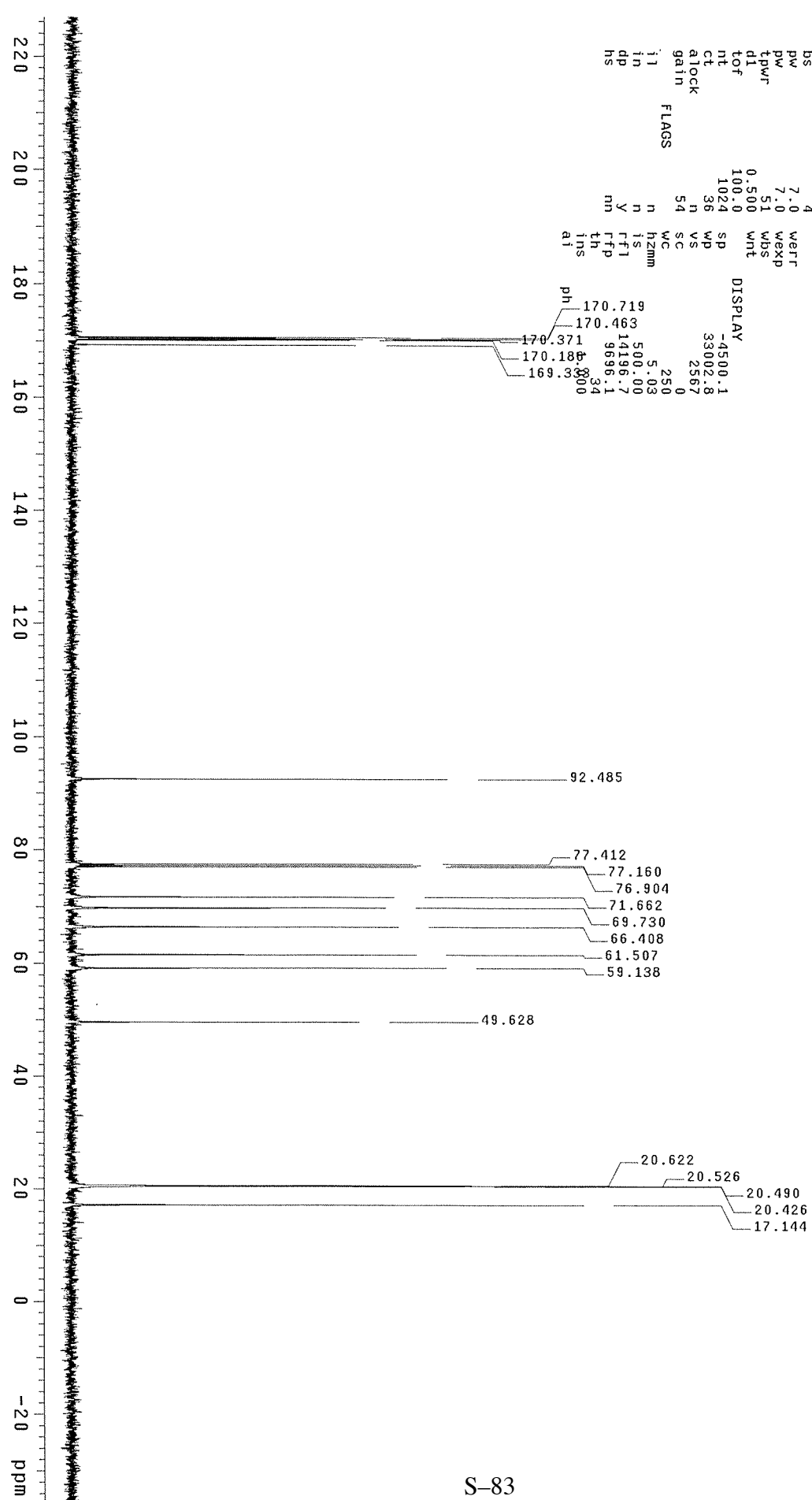
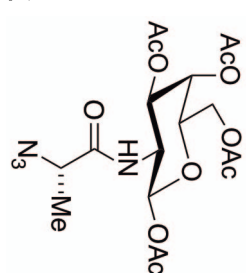
170.18

170.371

170.463

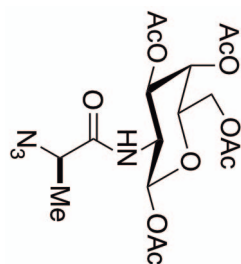
170.719

ph

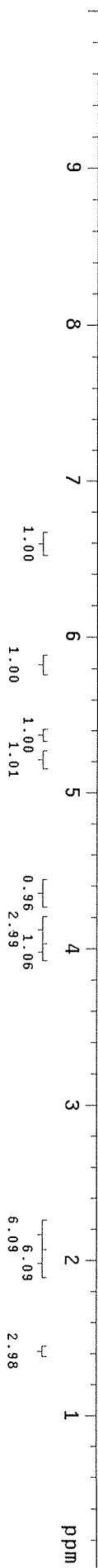


JC1171\_1H\_CDC13

exp2 s2pul1

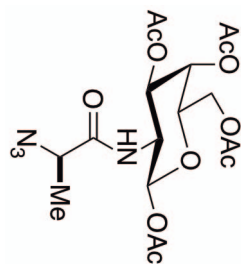


SAMPLE DEC. & VT  
 date Feb 21 2017 dfrq 499.751  
 solvent CDC13 dn H1  
 file exp dpwr 30  
 ACQUISITION dof 0  
 sfrq 499.751 dm nm  
 tn H1 dmm C  
 at 4.000 dmf 200  
 np 64000 dseq  
 sw 8000.0 dres 1.0  
 fb 4000 homo n  
 bs 4 dfrq2 DEC2  
 tpwr 60 dn2 0  
 pw 8.0 dpwr2  
 dl 0 dof2 1  
 tof 0 dm2 0  
 nt 16 dmm2 n  
 ct 16 dmf2 C  
 atlock n dseq2 200  
 gain 40 dres2 1.0  
 FLAGS n homo2 n  
 i1 n PROCESSING  
 in n wtfile  
 dp y nm  
 hs nm proc ft  
 DISPLAY fn 65536  
 math f  
 SP 0.0  
 WP 4997.3 weff  
 VS 151 wexp  
 SC 0 wds  
 WC 250 wnt  
 hzmm  
 IS 19.59  
 ffl 33.57  
 rffl 5142.1  
 lh 3628.2  
 ins 7  
 nm cdc ph 1.000



JC1171\_13C\_CDCl3

exp1 s2pu1



DEC. & VT

H1

-499.0

YYY

W

11400

43

not used

f

PROCESSING

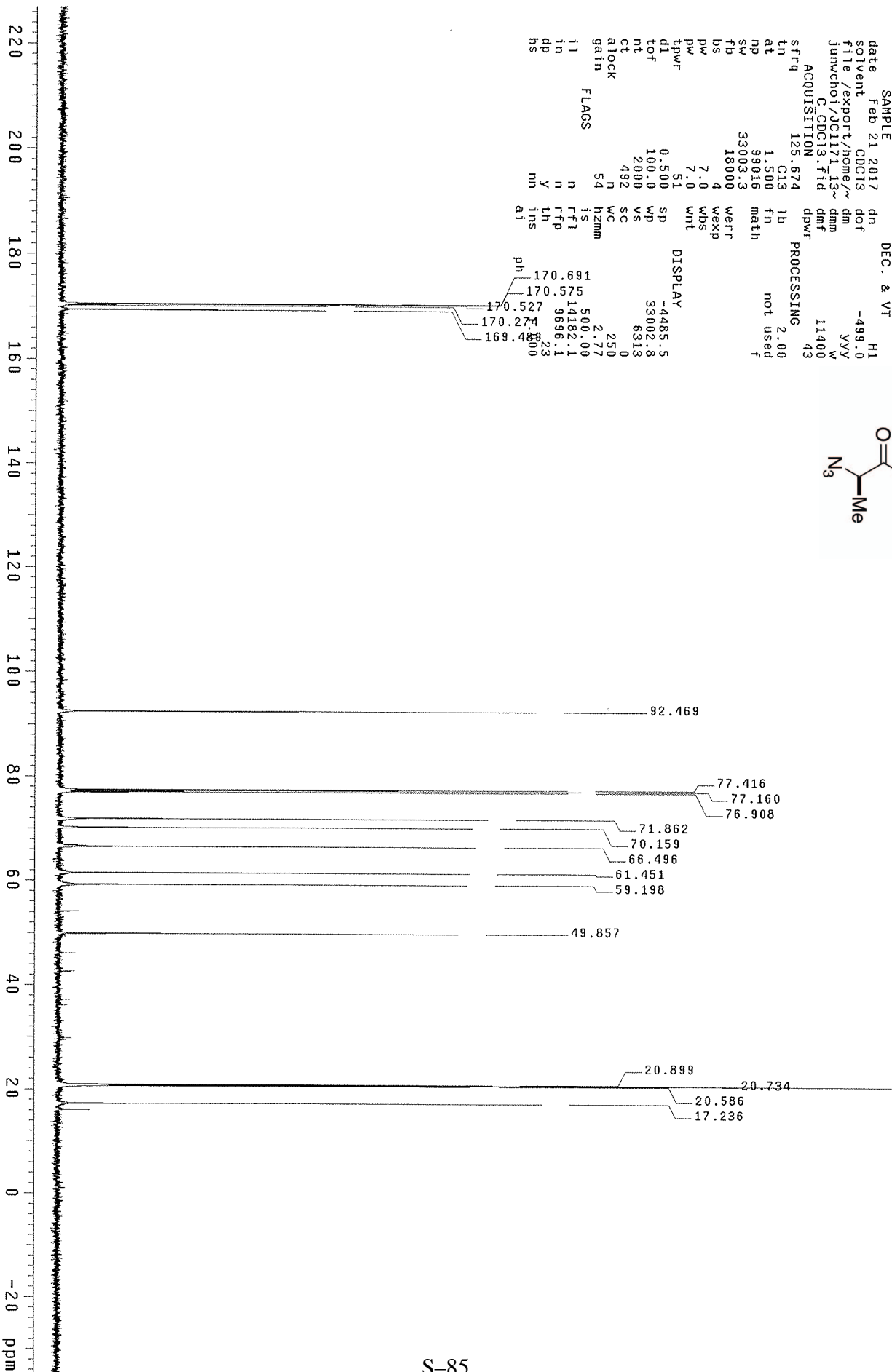
2.00

2.00

not used

f

SAMPLE Feb 21 2017 dn  
 solvent CDC13 dof  
 file /export/home/~ Junwchoi/JC1171\_13~ dm  
 C:\CDC13.Fid dmf  
 ACQUISITION 125.674 dpwr  
 sfrq 125.674 PROCESSING  
 tn C13 lb  
 at 1.500 fn  
 np 99016 math  
 sw 33003.3  
 fb 18000 wefr  
 bs 4 wexp  
 pw 7.0 wds  
 tpwr 7.0 wnt  
 dl 51  
 tof 0.500 sp  
 nt 100.0 wp  
 ct 2000 vs  
 atlock 492 SC  
 gain 54 hzmm  
 FLAGS n n  
 ll n n  
 in n n  
 dp y y  
 hs nn ins  
 ai ai



92.469

77.416  
77.160  
76.908

71.862  
70.159  
66.496  
61.451  
59.198

49.857

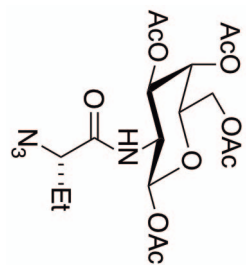
20.899

20.734

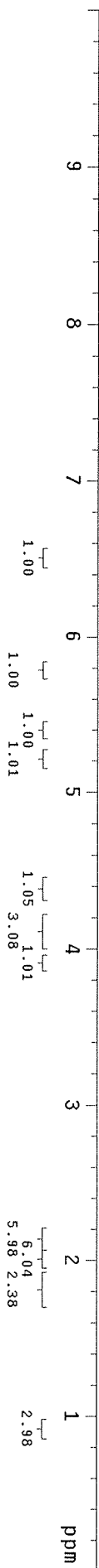
20.586  
17.236

JC1167\_1H\_CDC13

expi s2pu1

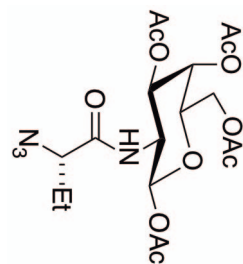


SAMPLE DEC. & VT  
 date Feb 21 2017 dfrq 499.751  
 solvent CDC13 dn H1  
 file exp dpwr 30  
 ACQUISITION dof 0  
 sfrq 499.751 dm mm  
 tn H1 dmm C  
 at 4.000 dmf 200  
 np 64000 dseq  
 sw 8000.0 dres 1.0  
 fb 4000 homo n  
 bs 4  
 tpwr 60 dfrq2 DEC2 0  
 pv 8.0 dn2 1  
 di 0 dpwr2 1  
 tof 0 dof2 0  
 nt 16 dmm2 n  
 ct 16 dmf2 C  
 atlock n dseq2 200  
 gain 40  
 FLAGS  
 i1 n homo2 1.0  
 in n  
 dp y wfile PROCESSING  
 hs nm proc fn ft  
 DISPLAY 65536 f  
 SP 0.0 math  
 WP 4997.3 weff  
 VS 130 wexp  
 SC WDS  
 WC 250 wnt  
 hzmm  
 IS 19.59  
 IS 33.57  
 rffl 5142.3  
 rfp 3628.2  
 th 7  
 ins 1.000  
 nm cdc ph

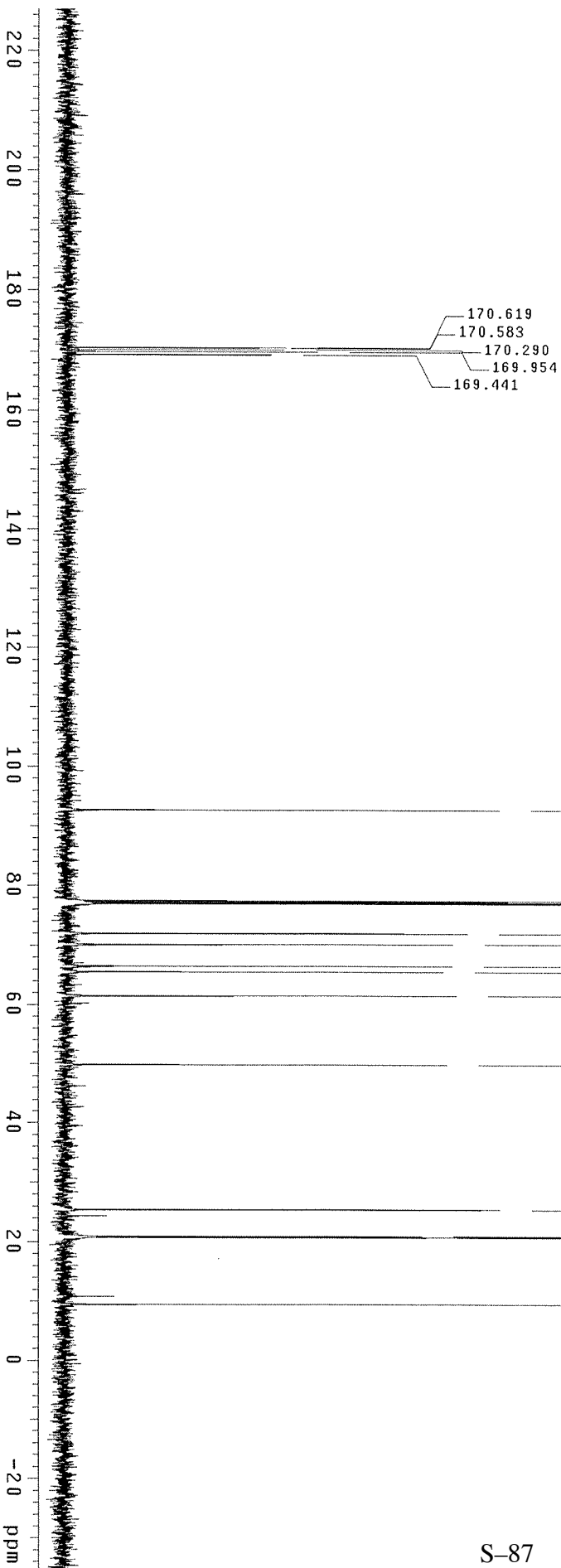


JC1167\_13C-CDCl3

exp2 s2pu1

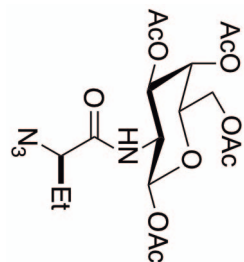


SAMPLE DEC. & VT H1  
date Feb 21 2017 dn  
solvent CDCl3 dof -499.0  
file exp dm  
ACQUISITION exp dmm  
sfrq 125.674 dmf 11400  
in C13 dpwr 43  
at 1.500 1b PROCESSING 2.00  
np 99016 fn not used f  
sw 33003.3 math  
fb 18000  
bs 4  
pw 7.0 weff  
pw 7.0 wexp  
tpwr 51 wbs  
di 0.500 wnt  
tof 100.0  
nt 2000 sp DISPLAY -4481.0  
ct 200 wp 33002.8  
atock n vs 9281  
gain 54 SC 9281  
i 1 WC 250  
in n hzmm 3.82  
in n is 500.00  
ddp y rffl 1417.6  
hs nn rffp 9696.1  
th ins 23  
ai ph 1.000

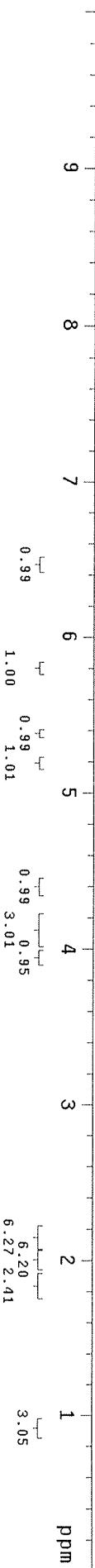


JCI169\_1H\_CDC13

expt1 s2pu1



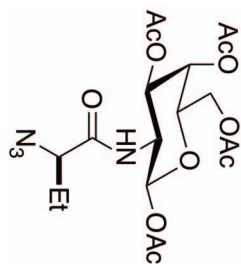
SAMPLE DEC. & VT  
 date Feb 21 2017 499.751  
 solvent CDC13 dn H1  
 file exp dpwr H1  
 ACQUISITION 30  
 dof 0  
 sfrq 499.751 dm nm  
 tn H1 dmm C  
 at 4.000 dmr 200  
 np 64000 dseq  
 sw 8000.0 dres 1.0  
 fb 4000 homo n  
 bs 4  
 tpwr 60 dfrq2 DEC2  
 pw 8.0 dn2 0  
 dt 0 dpwr2 1  
 tof 0 dof2 0  
 nt 16 dm2 n  
 ct 16 dmm2 n  
 alock n dmf2 C  
 gain 40 dseq2 200  
 flags n dres2 1.0  
 i1 n homo2 n  
 in y wtfile PROCESSING  
 dp y wproc  
 hs nn math  
 DISPLAY 0.0 ft  
 SP 4997.3 fn 65536  
 WP 119 weff f  
 VS 0 wexp  
 SC 250 wds  
 WC 19.99 wnt  
 hzmm 33.57  
 IS 5142.3  
 rfl 3628.2  
 rfp 7  
 th 1.000  
 ins nm cdc ph



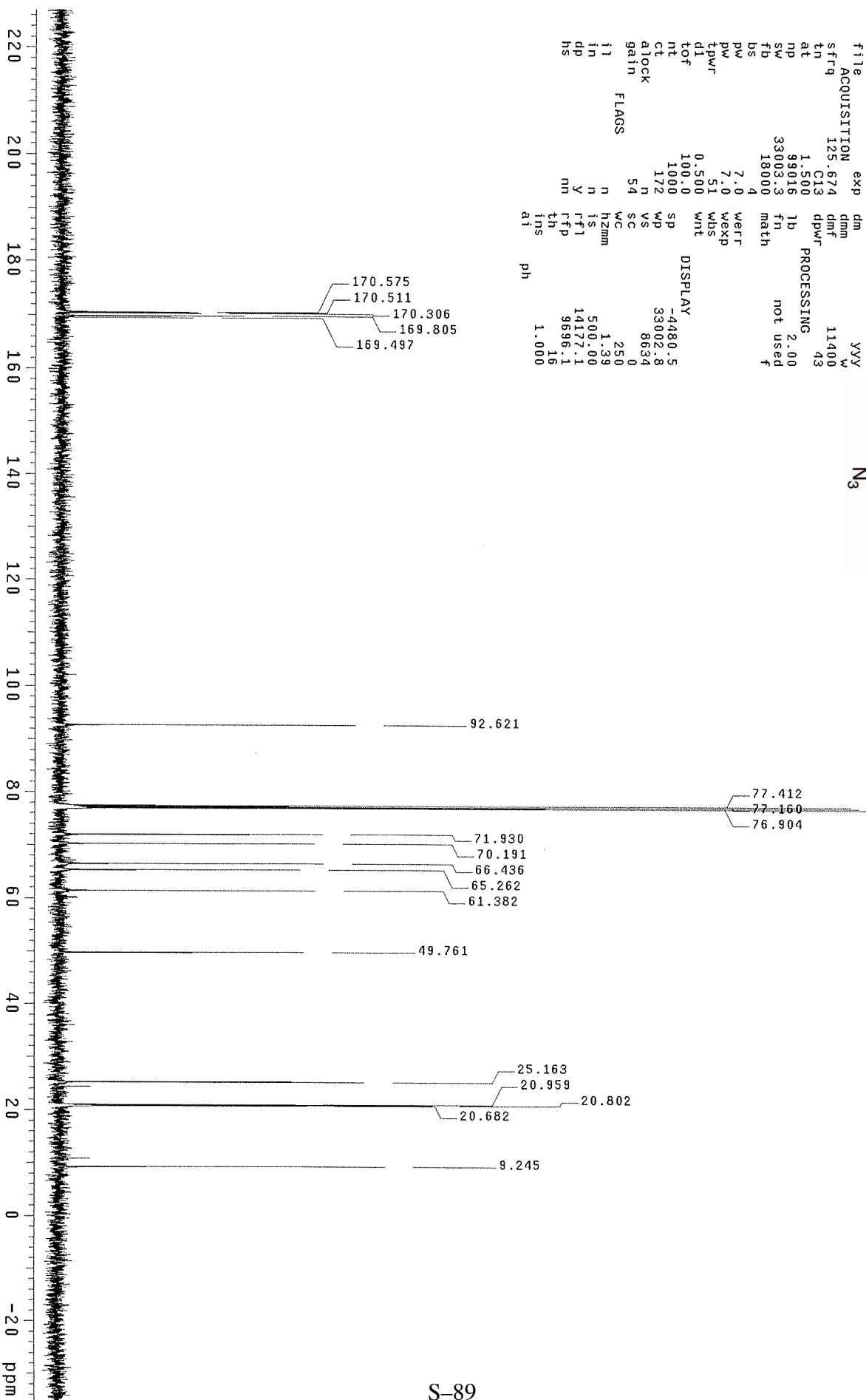


JC1169\_13C\_CDCl3

exp2 s2pu1

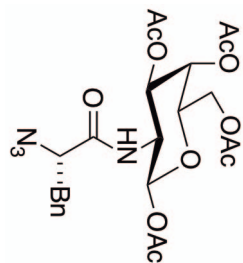


SAMPLE DEC. & VT H1  
 date Feb 21 2017 dn  
 solvent CDC13 dof -499.0  
 file exp dm  
 ACQUISITION exp dmm yyy  
 sfrq 125.674 dmf 11400  
 tn C13 dpwr 43  
 at 1.500 PROCESSING  
 np 99016 lb 2.00  
 sw 33003.3 fn not used f  
 fb 18000 math  
 bs 4  
 pw 7.0 weff  
 dw 7.0 wexp  
 tpwr 51 wds  
 dl 0.500 wnt  
 lof 100.0  
 nt 1000 sp -4480.5  
 ct 172 wp 33002.8  
 alock n vs 8634  
 gain 54 sc 0  
 FLAGS hzmm 250  
 i1 n 1.39  
 in n 509.00  
 dp y rfl 1417.1  
 hs nn rfp 9696.1  
 th 16  
 ins  
 ai ph 1.000



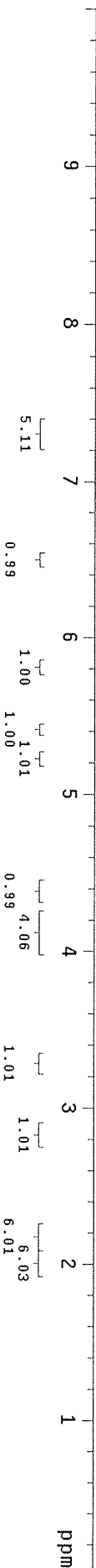
JC1083\_1H\_CDCl3

exp2 s2pu1



SAMPLE 5 2017  
 date May 5 2017  
 solvent CDC13  
 file exp  
 ACQUISITION 499.751  
 sfrq 499.751  
 tn H1  
 at H1  
 np 4.000  
 sw 64000  
 fb 8000.0  
 bs 4000  
 tpwr 4  
 pw 60  
 dt 8.0  
 tof 0  
 nt 0  
 ct 8  
 atlock n  
 gain 40  
 flags 40  
 i1 n  
 in n  
 dp y  
 hs n  
 DISPLAY -0.2  
 SP 4997.3  
 WP 160  
 VS 0  
 SC 0  
 WC 250  
 hzmm 19.99  
 IS 9333.43  
 ffl 1501.2  
 fth 0  
 ins 7  
 nm cdc 1.000  
 ph

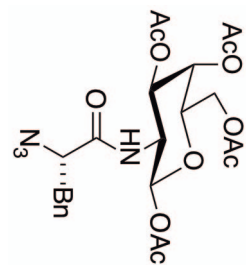
DEC. & VT 499.751  
 dfrq H1  
 dn 30  
 dpwr 0  
 dof 0  
 dm nnn  
 dmm C  
 dmf 200  
 dseq 1.0  
 dres n  
 homo  
 dfrq2 0  
 dn2  
 dpwr2 1  
 dof2 0  
 dmf2 n  
 dmm2 C  
 dmf2 200  
 dseq2 1.0  
 dres2  
 homo2 n  
 PROCESSING  
 y  
 wtfile ft  
 n  
 proc f  
 math  
 65536  
 f  
 wft



JC1083\_13C\_CDCl3

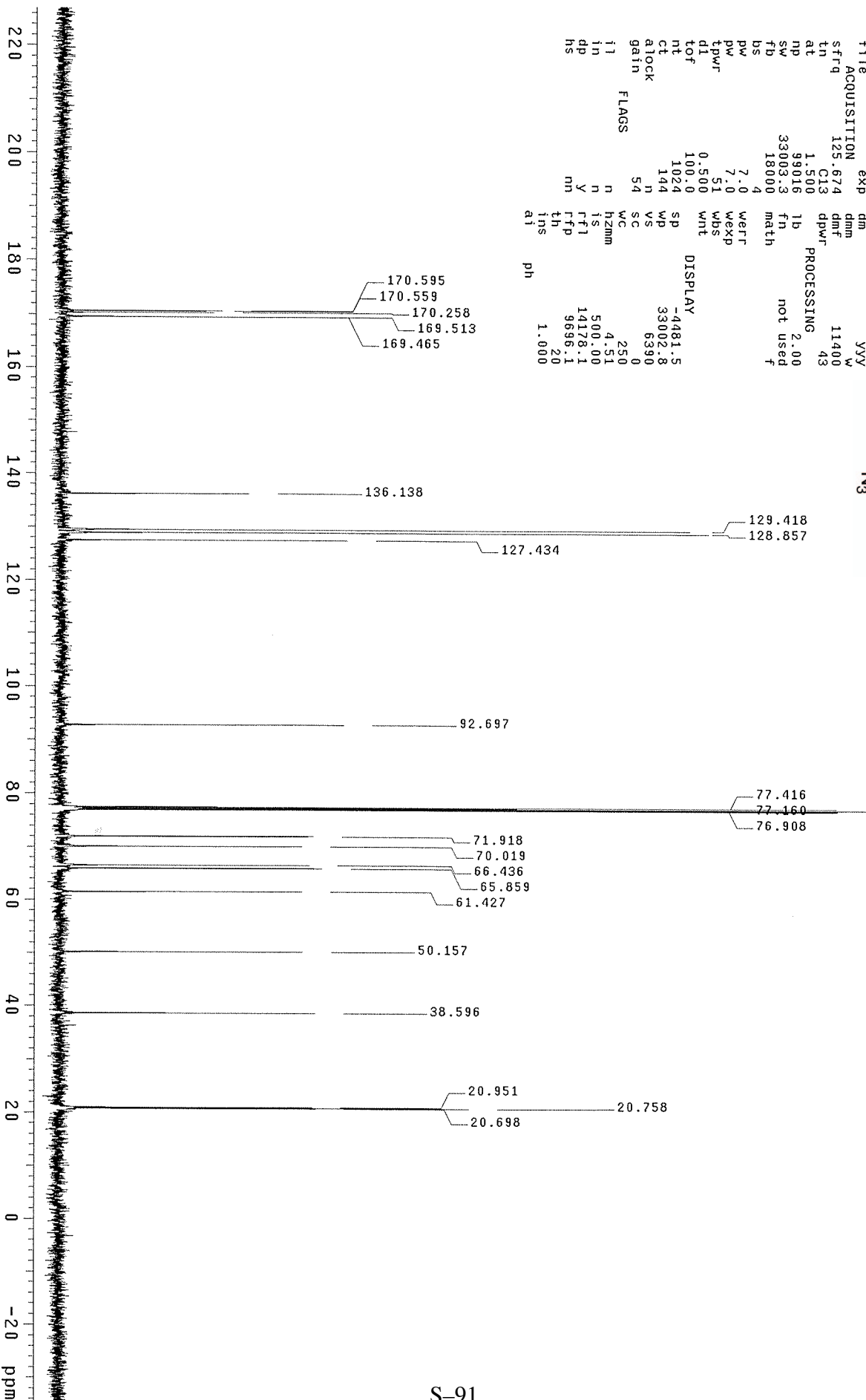
expi s2pu1

DEC. & VT



PARAMETER	VALUE	UNIT
date	May 5 2017	
solvent	CDCl3	
file	exp	
ACQUISITION	125.674	dmf
sfrq	125.674	dmf
tn	C13	dmf
at	1.500	dmf
np	99016	lb
sw	33003.3	fn
fb	18000	math
bs	4	
pw	7.0	werr
pw	7.0	wexp
tpwr	51	wbs
di	0.500	wnt
tof	100.0	
nt	1024	sp
ct	144	wp
atlock	n	vs
gain	54	sc
FLAGS	n	wc
il	n	hzm
in	n	is
dp	y	ftl
hs	nn	rfl
		th
		ins
		ai

PARAMETER	VALUE
DISPLAY	-4481.5
ph	33002.8
	6390
	0
	250
	4.51
	500.00
	14178.1
	9696.1
	20
	1.000



JC1081\_1H\_CDCl3

expt szpu1

SAMPLE 5 2017 DEC. & VT 499.751

date May 5 2017

solvent CDC13

file exp

ACQUISITION 499.751

td H1 dm nmn

at 4.000 dmf C

np 64000 dseq 200

sw 8000.0 dres 1.0

fb 4000 homo n

bs 4 dfrq2 DEC2 0

tpwr 60 dn2

pw 8.0 dpwr2 1

dl 0 dof2 0

tof 16 dm2 n

nt 12 dmm2 C

alock n dmf 200

gain 40 dseq2

flags n dres2 1.0

il n homo2 n

in n

dp y wfile PROCESSING

hs nn proc ft

sp 0.0 math f

wp 4997.3 werr 65536

vs 151 wexp

sc 0 wbs

wc 250 wnt

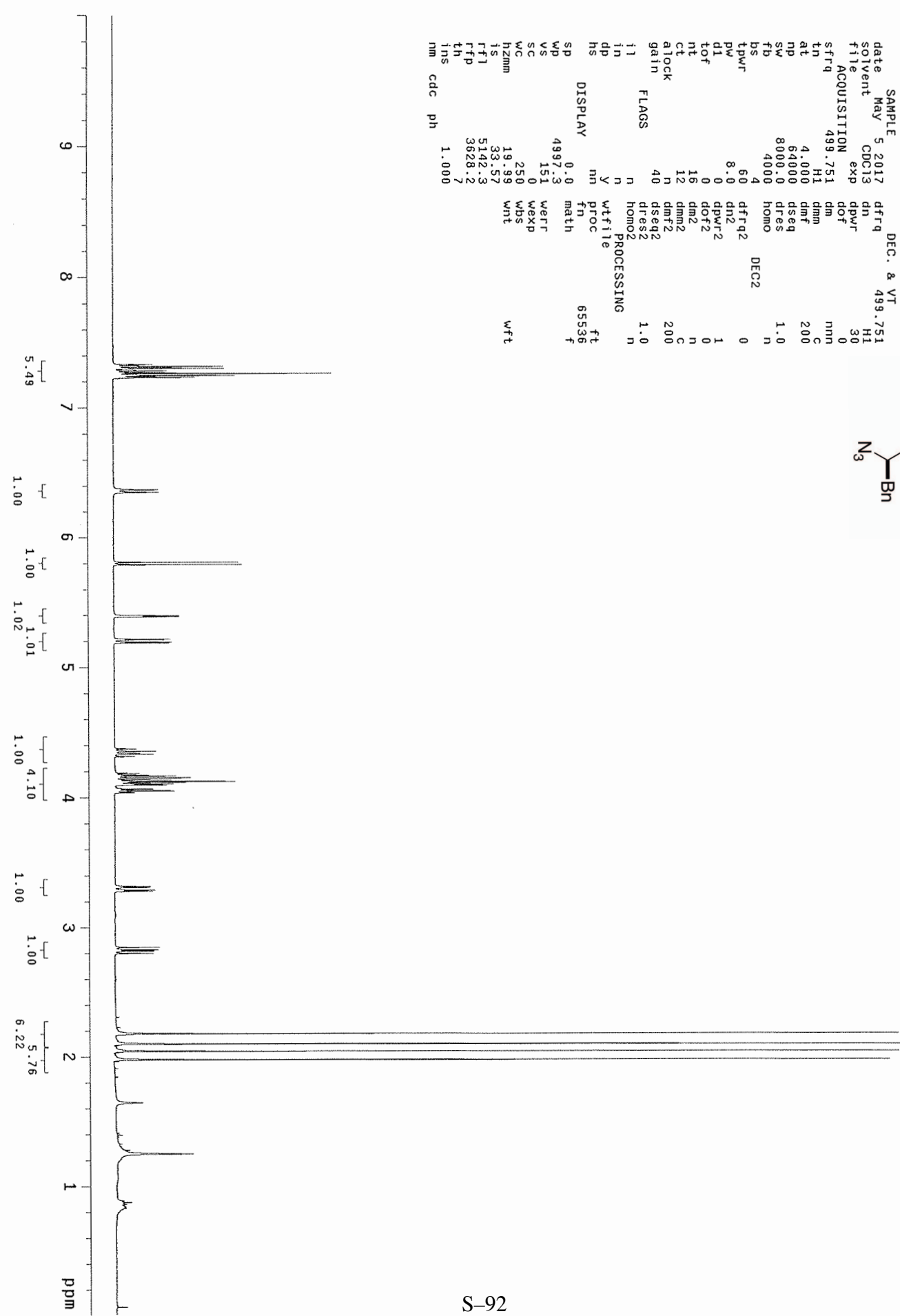
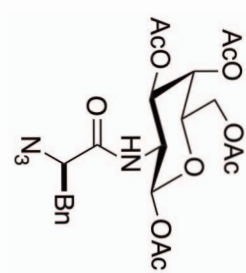
h2mm 19.99

is 33.57

ftf 5142.3

lth 3628.2

ins 1.000



JC1081\_13C\_CDCl3

exp2 s2pul1

DEC. & VT

H1

-499.0

YYY

W

11400

43

not used

f

PROCESSING

2.00

1b

fn

math

werf

wexp

wbs

wnt

DISPLAY

-4477.5

33002.8

26403

0

250

3.64

18000

170.25

170.53

169.433

169.1

169.1

169.1

169.1

169.1

169.1

169.1

169.1

169.1

169.1

169.1

169.1

169.1

169.1

169.1

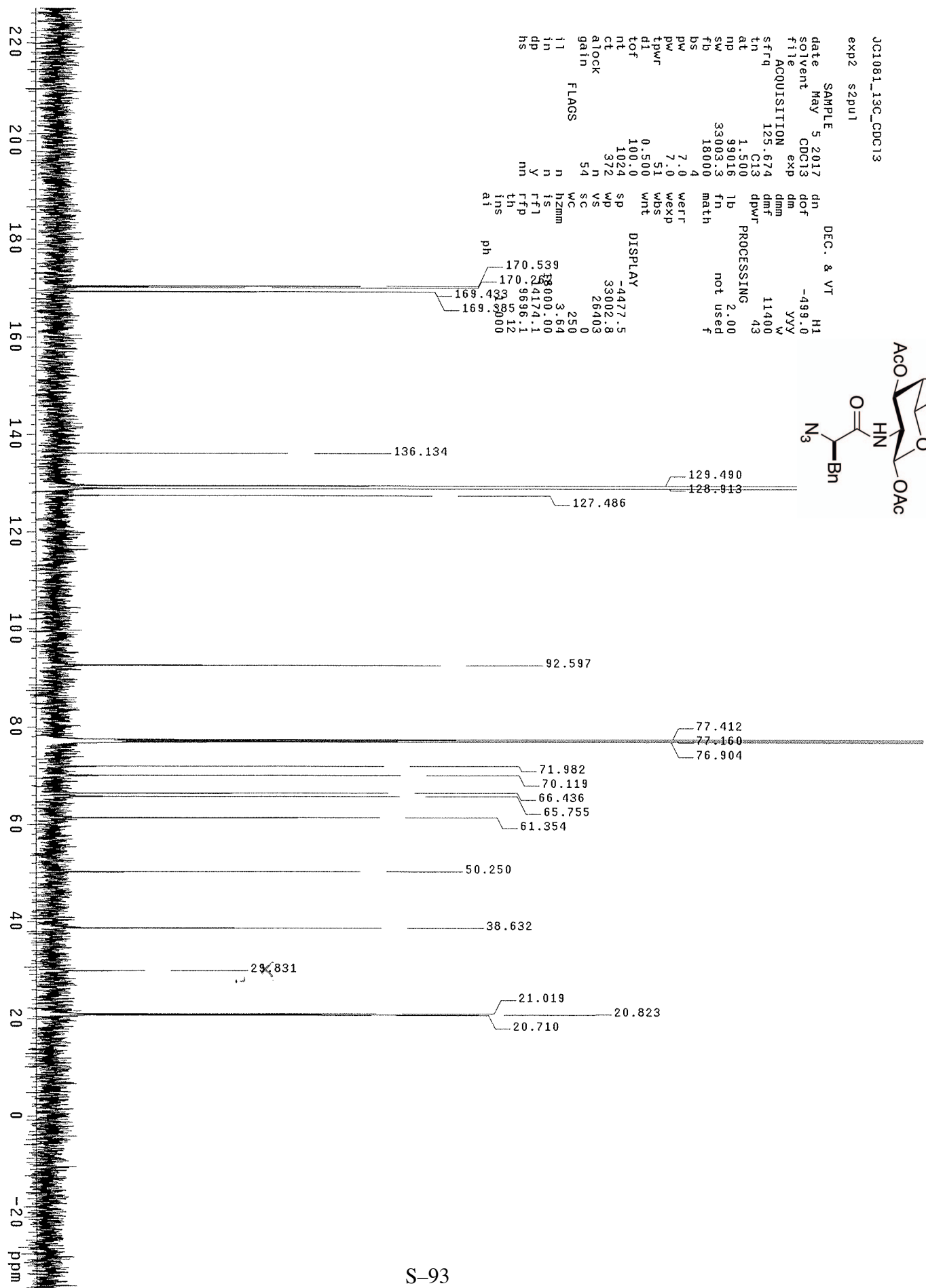
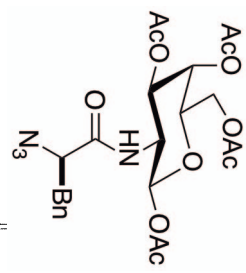
169.1

169.1

169.1

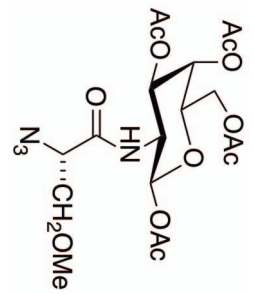
169.1

169.1



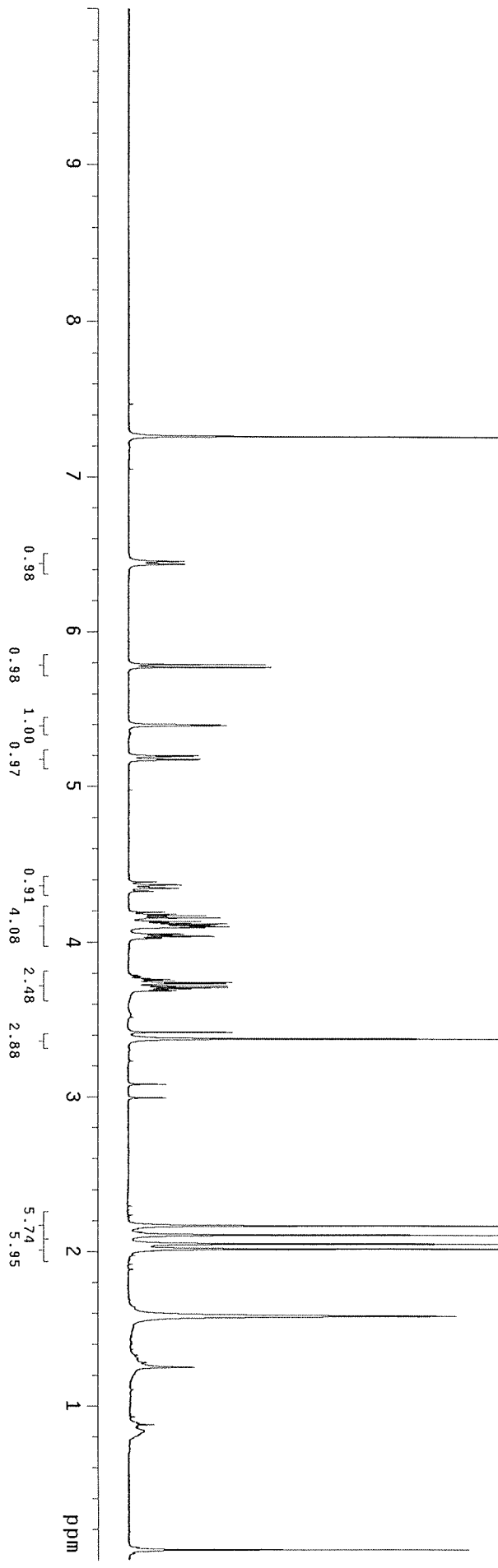
JC1201(S)\_1H\_CDCl3

expt1 s2pul1



SAMPLE 8 2017  
 date May 8 2017  
 solvent CDCl3  
 file exp  
 ACQUISITION  
 sfrq 499.751  
 tn H1  
 at 4.000  
 np 64000  
 sw 8000.0  
 fb 4000  
 bs 4  
 tpwr 60  
 pw 8.0  
 dl 0  
 tof 0  
 nt 128  
 ct 128  
 alock n  
 gain 40  
 flags n  
 i1 n  
 in y  
 dp n  
 hs n  
 DISPLAY 0.0  
 sp 4997.3  
 wp 151  
 vs 0  
 sc 250  
 wc 19.99  
 hzmm 49820.65  
 fs 5142.3  
 ffl 3628.7  
 fh 1.000  
 ins nm  
 cdc ph

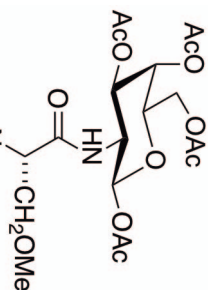
DEC. & VT 499.751  
 dfrq dn H1  
 dn H1  
 dpwr 30  
 dof 0  
 dm nnn  
 dmm c  
 dmf 200  
 dseq n  
 dres 1.0  
 homo n  
 dfrq2 0  
 dn2 1  
 dpwr2 1  
 dof2 0  
 dm2 n  
 dmm2 n  
 dmt2 c  
 dseq2 200  
 dres2 1.0  
 homo2 n  
 PROCESSING  
 y wtfile  
 n proc  
 ft  
 ft  
 65536  
 f  
 math  
 wft



JC1201(s)\_13C\_CDC13

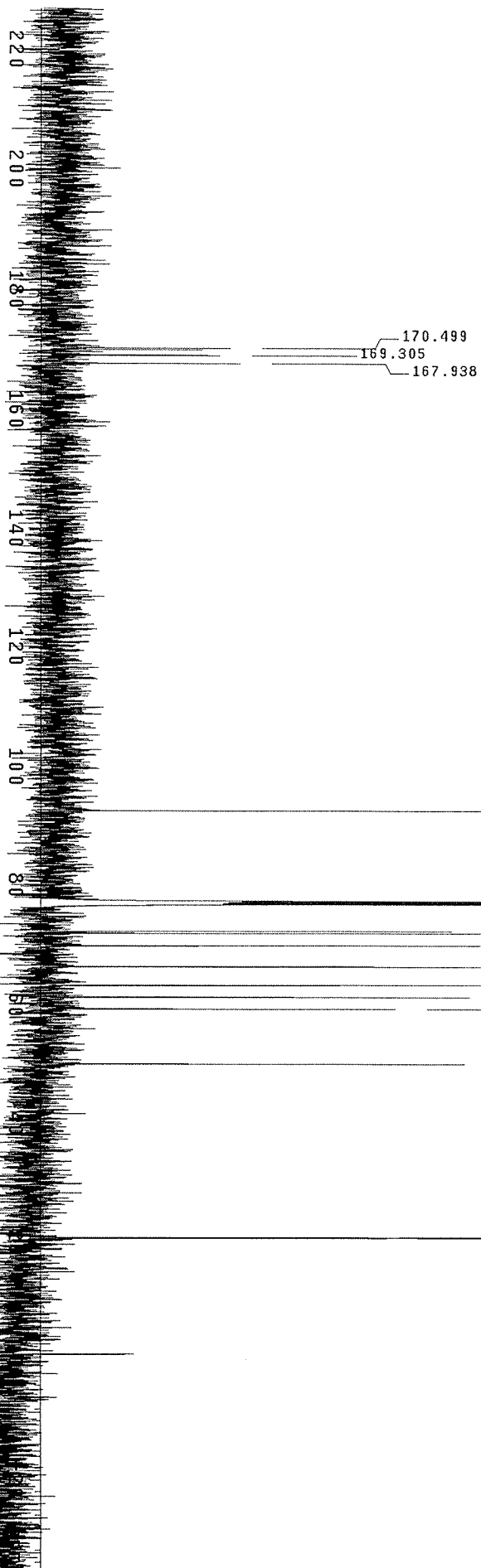
exp2 s2pu1

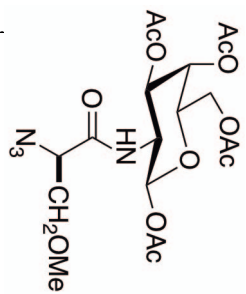
DEC. & VT



date	8 2017	dn	H1
solvent	CDCl3	dof	-499.0
file	exp	dm	YYY
ACQUISITION		dmm	W
sfrq	125.674	dmf	11400
tn	C13	dpwr	43
at	1.500	1b	
nd	99016	PROCESsing	2.00
sw	33003.3	fn	not used
fd	18000	math	f
bs	4	werr	
pw	7.0	wexp	
tpwr	7.0	wbs	
dl	51	wrt	
tof	0.500	SP	-4475.5
nt	100.0	WP	33002.8
ct	20000	VS	140159
atlock	4500	SC	0
gain	N	WC	250
FLAGS	54	hzm	3.64
il	n	is	500.00
in	n	rfl	14172.0
dp	Y	rflp	9696.1
hs	nm	th	21
		ins	1.000
		at	ph

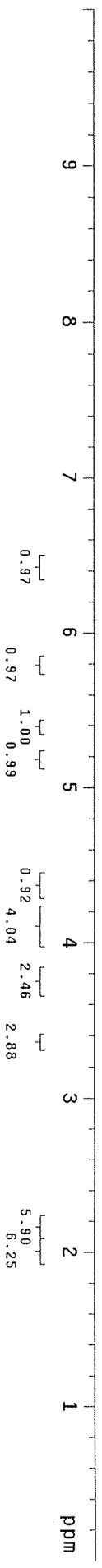
DISPLAY	-4475.5
SP	33002.8
WP	140159
VS	0
SC	250
WC	3.64
hzm	500.00
is	14172.0
rfl	9696.1
rflp	21
th	1.000
ins	
at	ph





```

JC1201(R)_1H_CDCl3
exp1 s2pu1
SAMPLE 9 2017 DEC. & VT 499.751
date May 9 2017 dfrq 499.751
solvent CDCl3 dn HI 30
file exp dpwr 30
ACQUISITION dof 0
sfrq 499.751 dm nnn
tn H1 dmm c
at 4.000 dmf 200
nd 64000 dseq
sw 8000.0 dres 1.0
fd 4000 homo n
bs 4 DEC2
tpwr 60 dfrq2 0
pw 8.0 dn2
d1 0 dpwr2 1
tof 0 dof2 0
ct 128 dmm2 n
atlock 128 dmt2 c
gain 40 dseq2 200
flags n dres2 1.0
i1 homo2 n
in n wtfile PROCESSING
dp y wfile ft
hs mn proc fn 65536
DISPLAY math wnt
SP 0.0
WP 4997.3 werr
VS 184 wexp
SC 0 wbs
WC 250 wnt
hzmm 19.99
is 33.57
f1 5142.3
f1p 3628.2
lh 1.000
nm cdc ph
  
```

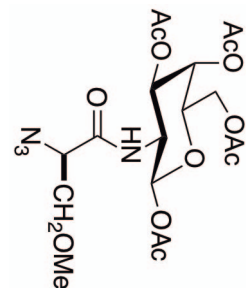




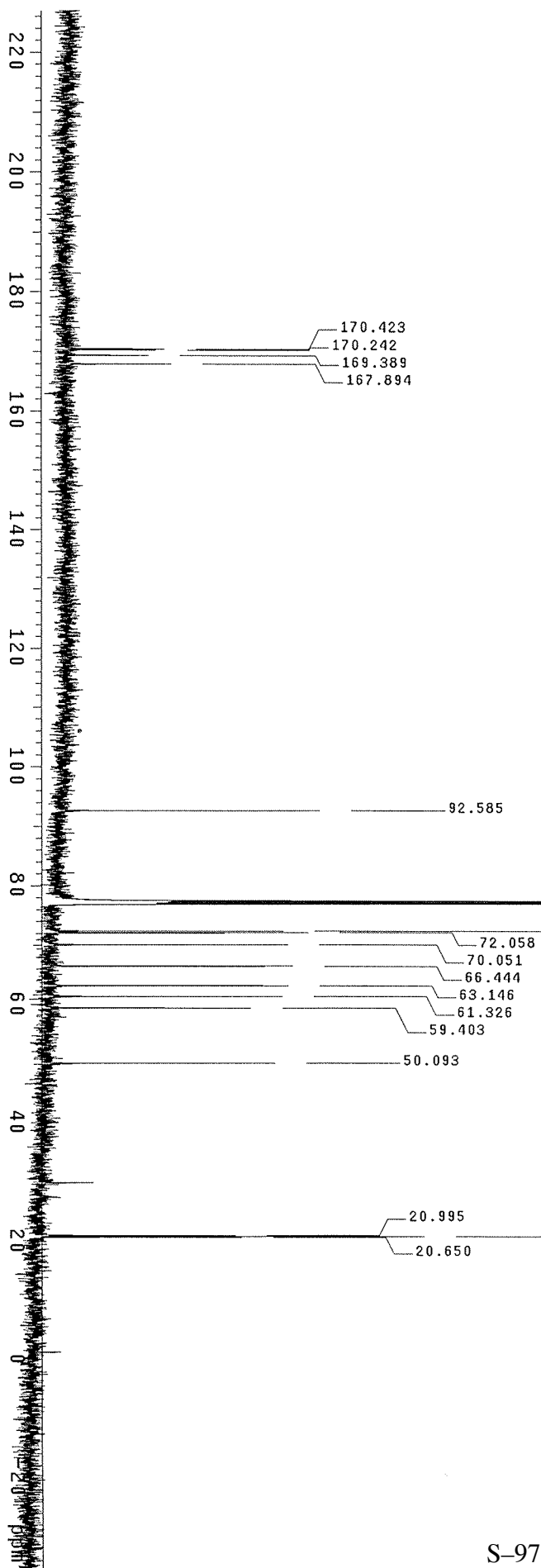
JC1201(R)\_13C\_CDC13

exp2 s2pul1

DEC. & VT

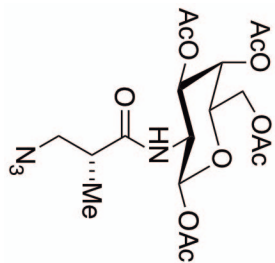


SAMPLE 9 2017 dn H1  
 date May 9 2017 dn -499.0  
 solvent CDC13 dof  
 title exp dm YYY  
 ACQUISITION dmm W  
 sfrq 125.674 dmf 11400  
 tn C13 dpwr 43  
 at 1.500 1b  
 np 99016 fn not used f  
 sw 33003.3 math  
 fd 18000  
 bs 4  
 pw 7.0 weff  
 pv 7.0 wexp  
 tpwr 51 wbs  
 dl 0.500 wnt  
 tof 100.0  
 nt 16000 SP -4475.5  
 ct 15780 WP 33002.8  
 atlock N VS 88854  
 gain 54 WC 250  
 FLAGS  
 i1 n hzmm 4.29  
 in n is 500.00  
 dp y rffl 14172.0  
 hs nn rfp 9696.1  
 th  
 ins 1.00  
 at ph

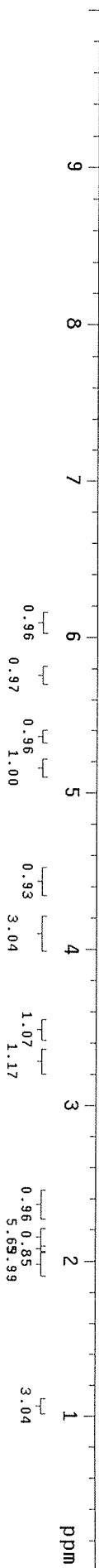


JC2141\_1H\_CDC13

exp1 s2pu1



SAMPLE DEC. & VT  
 date 9 2017 499.751  
 solvent CDC13 H1  
 file exp H1  
 ACQUISITION 499.751 dm  
 sfreq 499.751 dm  
 tn H1 dmm  
 at 4.000 dmf  
 np 64000 dseq  
 sw 8000.0 dres  
 fb 4000 homo  
 bs 4  
 tpwr 60 dffq2 DEC2  
 dl 8.0 dp2  
 tof 0 dpwr2 1  
 nt 64 dof2 0  
 ct 36 dmm2 n  
 atlock n dmf2 C  
 gain 40 dseq2 200  
 i1 n dres2 1.0  
 in n homo2 n  
 dp y wtfile PROCESSING  
 hs nm fn ft  
 DISPLAY 0.0 math f  
 SP 4997.3 werr 65536  
 VS 151 wexp f  
 SC 0 WBS  
 WC 250 WBS  
 hzmm 19.99 Wnt  
 IS 33.97  
 ffl 5142.1  
 rffl 3628.2  
 lh 7  
 ins 1.000  
 nm cdc ph



JC2141\_13C\_CDC13

exp2 s2pu1

SAMPLE 9 2017 DEC. & VT

date May 9 2017 dn H1

solvent CDC13 dof -499.0

file exp dm YYY

ACQUISITION exp dmm W

sfrq 125.674 dmf 11400

tn C13 dpwr 43

at 1.500 PROCESSING 2.00

np 99016 lb 1b

sw 33003.3 fn not used f

fb 18000 math

bs 4

pw 7.0 wefr

pt 7.0 wexp

dl 51 wbs

tpwr 0.500 wnt

dl 100.0

tof 2000

nt 148

ct 148

atock n

gain 54

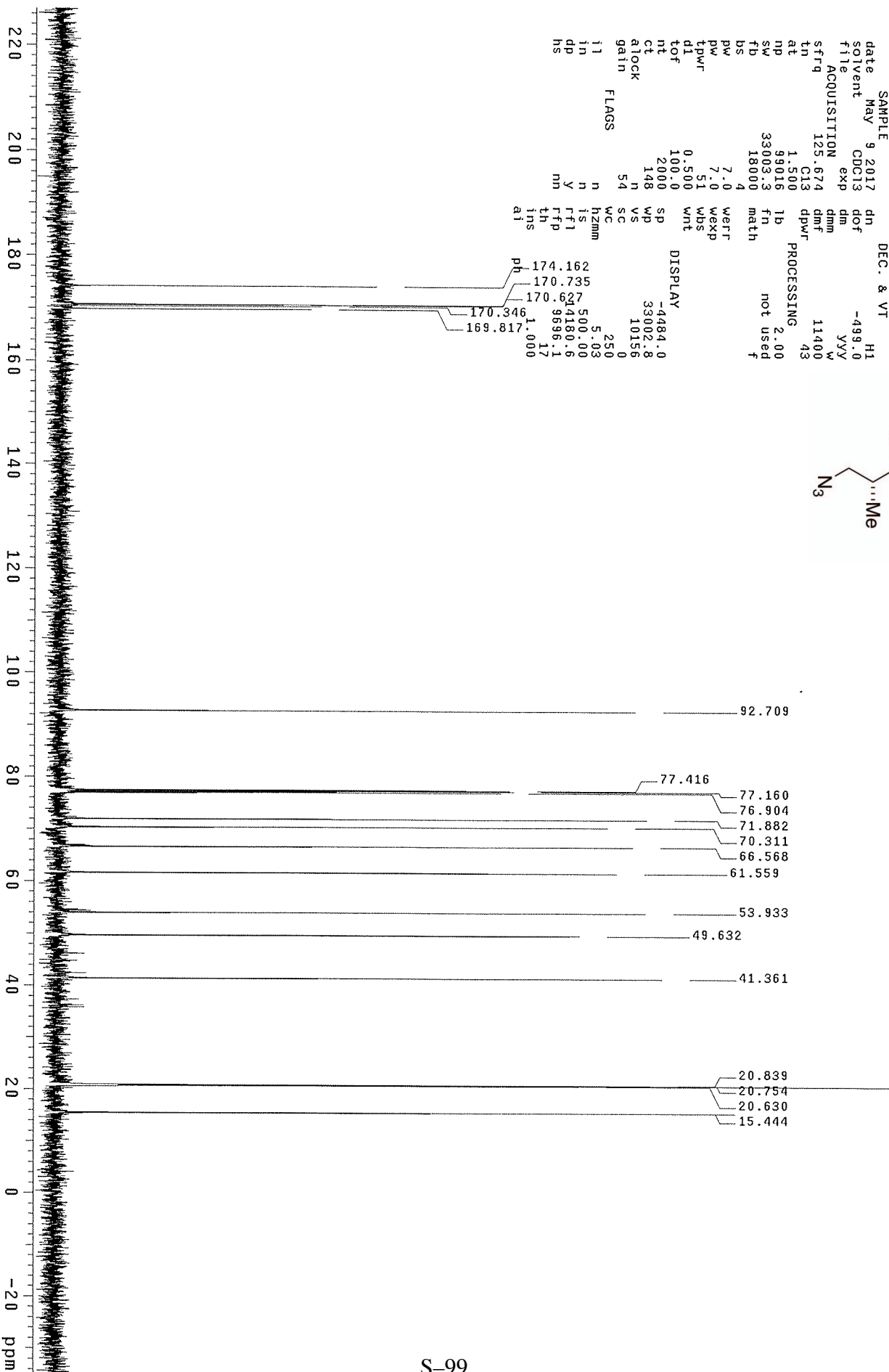
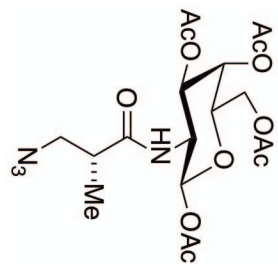
FLAGS

il n

in n

dp Y

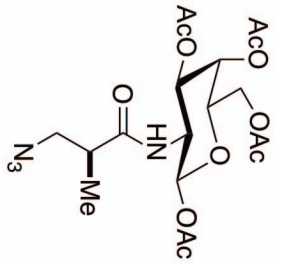
hs nm



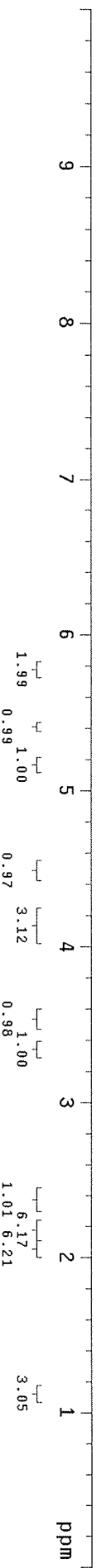
DISPLAY -4484.0  
 sp 33002.8  
 wp 10156  
 vs 0  
 sc 250  
 hc 0  
 wczmm 5.03  
 n 500.00  
 is 4180.6  
 ftl 9696.1  
 rfp 17  
 th 17  
 ins

JG5127\_1H\_CDC13

exp2 s2put1

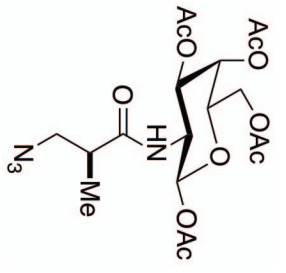


SAMPLE Jun 14 2017 DEC. & VT 499.751  
solvent CDC13 dn H1  
file exp dpwr H1  
ACQUISITION 499.751 dm 30  
sfrq 499.751 dm 0  
tn H1 dmm nnn  
at 4.000 dmf c  
np 64000 dseq 200  
sw 8000.0 dres 1.0  
fb 4000 homo n  
bs 4  
tpwr 60 dfrq2 DEC2 0  
pw 8.0 dn2  
d1 0 dpwr2 1  
tof 0 dof2 n  
nt 16 dm2 n  
ct 16 dmm2 c  
alock n dmf2 200  
gain 40 dseq2  
flags n dres2 1.0  
homoz n  
PROCESSING  
ft  
wtfile f  
nm proc 65536  
hs y math f  
DISPLAY -0.2  
SP wp 4997.3 werr  
VS vs 151 wexp  
WC sc 250 wbs  
hzmh 19.99 wnt  
IS 33.57  
rffl 1501.2  
th 0  
ins 7  
nm cdc ph 1.000



JC5127\_13C\_CDCl3

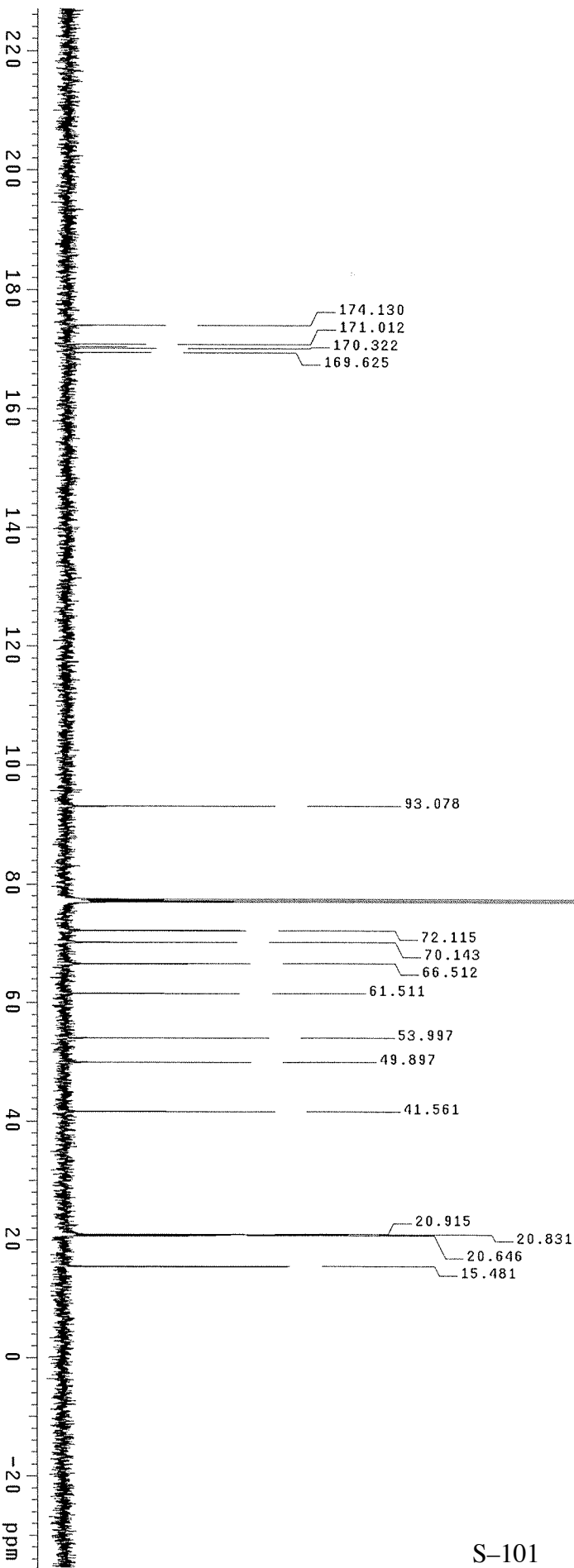
exp2 s2pul



SAMPLE Jun 14 2017  
 solvent CDC13  
 file exp  
 ACQUISITION 125.674  
 sfrq C13  
 tn 1.500  
 at 99016  
 np 33003.3  
 sw 18000  
 fb 4  
 bs 7.0  
 pw 7.0  
 tpwr 5.1  
 dl 0.500  
 tof 100.0  
 nt 1024  
 ct 224  
 atlock n  
 gain 54  
 FLAGS  
 i1 n  
 in n  
 dp y  
 hs nn  
 ai ins  
 ph 1.000

DEC. & VT H1  
 -499.0  
 yy  
 W  
 11400  
 43  
 PROCESSING  
 2.00  
 not used  
 f

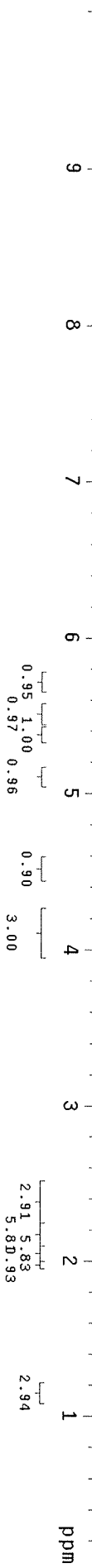
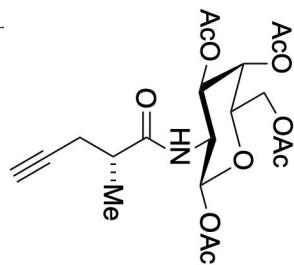
DISPLAY  
 -4478.5  
 33002.8  
 9076  
 0  
 250  
 4.86  
 509.00  
 14175.1  
 9696.1  
 10  
 1.000



JC2179\_1H\_CDC13

exptl s2pu1

SAMPLE      date      May 10 2017      DEC. & VT      499.751  
 solvent      CDC13      exp      H1  
 file      ACQUISITION      499.751      dm      30  
 sfrq      499.751      dm      0  
 tn      H1      dm      mmm  
 at      4.000      dmf      C  
 np      64000      dseq      200  
 sw      8000.0      dres      1.0  
 fb      4000      homo      n  
 bs      4      dfrq2      DEC2      0  
 tpwr      60      dp2      0  
 d1      8.0      dpwr2      1  
 tof      0      dof2      0  
 nt      256      dmf2      n  
 ct      128      dmm2      n  
 atlock      n      dmf2      C  
 gain      40      dseq2      200  
 i1      n      dres2      n  
 in      n      homo2      1.0  
 ip      n      wfile      n  
 dp      y      wfile      PROCESSING  
 hs      n      fn      ft  
                 proc      65536  
                 math      f  
 DISPLAY      0.0  
 SP      4997.3      werrf      wft  
 VS      151      wexp      wft  
 SC      0      wbs      wft  
 WC      250      wnt      wft  
 hzmm      19.99  
 IS      33.57  
 ffl      5142.1  
 rffl      3628.2  
 th      7  
 ins      1.000  
 nm      cdc      ph



JC2179\_13C\_CDC13

exp2 s2pu1

SAMPLE DEC. & VT

date May 10 2017 dn H1

solvent CDC13 dof -499.0

file exp dm dmm YYY

ACQUISITION exp dm 11400

sfrq 125.674 dpwf 43

at 1.500 PROCESSING 2.00

np 99016 lb 2.00

sw 33003.3 fn not used f

fb 18000 math

bs 4

pw 7.0 weff

pt 7.0 wexp

td 51 wbs

dl 0.500 wrt

tof 100.0 sp

nt 15000 wp

ct 15000 vs

alock n n

gain 54 sc

FLAGS hzmm

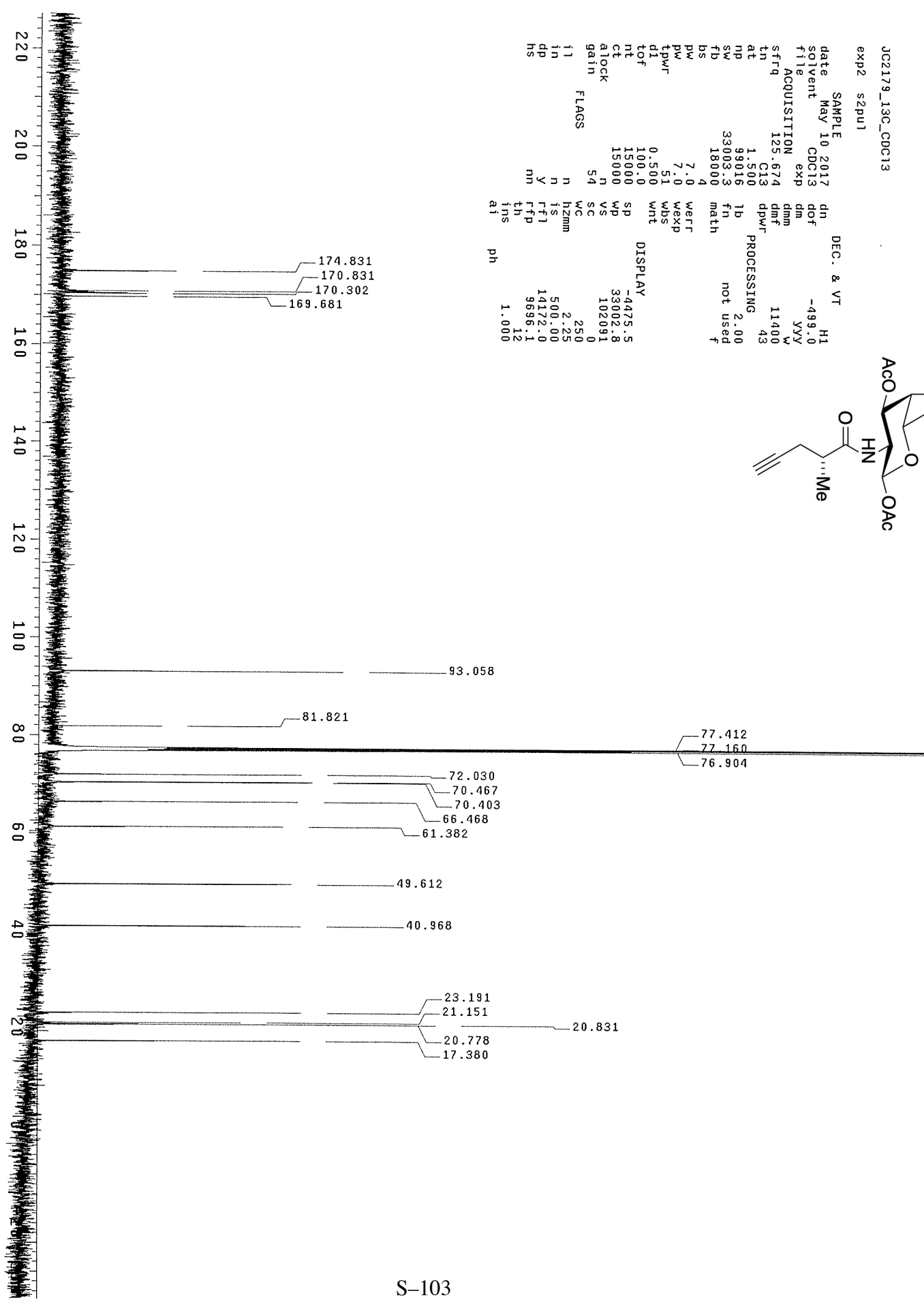
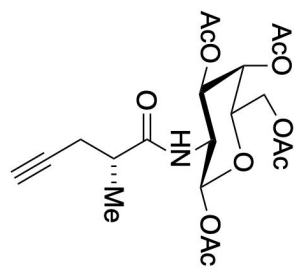
i1 n

in n

dp y

hs nn

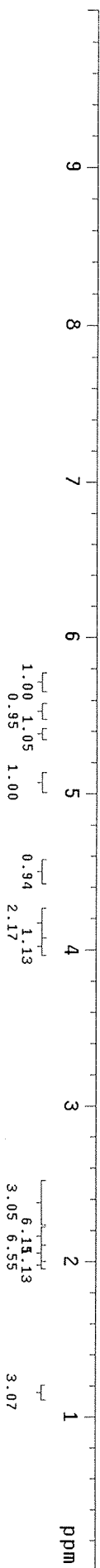
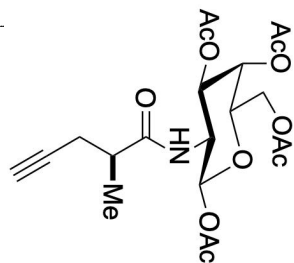
ai	ins	th	h	ftj	is	h	zmm	wt	vs	wp	sp	DISPLAY
1.000	12	9696.1	14172.0	500.00	2.25	0	250	102091	102091	33002.8	-475.5	



JC2175\_1H\_CDC13

exp1 s2pul1

SAMPLE	date	9	2017	dfrrq	DEC. & VT	499.751
solvent	file	CDC13	exp	dn	H1	30
ACQUISITION	dfpwr	499.751	dm	doF	0	0
tn	dm	H1	dm	dm	mm	mm
at	dmf	H1	dmf	dmf	C	200
np	dseq	64000	dseq	dseq	1.0	n
sw	dres	8000.0	dres	dres	1.0	n
fb	homo	4000	homo	homo	1.0	n
bs	dfrrq2	4	dfrrq2	dfrrq2	0	0
tpwr	dn2	60	dn2	dn2	0	0
pw	dpwr2	8.0	dpwr2	dpwr2	1	1
d1	doF2	0	doF2	doF2	0	0
tof	dm2	0	dm2	dm2	n	n
nt	dmm2	128	dmm2	dmm2	n	n
ct	dmf2	128	dmf2	dmf2	C	200
alock	dseq2	n	dseq2	dseq2	200	200
gain	dres2	40	dres2	dres2	1.0	n
flags	homo2	n	homo2	homo2	1.0	n
in	wtfile	n	wtfile	wtfile	ft	ft
dp	proc	Y	proc	proc	f	f
hs	math	nn	math	math	65536	65536
DISPLAY	0.0	0.0	0.0	0.0	0.0	0.0
wp	werr	4997.3	werr	werr	151	151
vs	wexp	151	wexp	wexp	0	0
sc	wbs	250	wbs	wbs	0	0
wc	wnt	250	wnt	wnt	0	0
h2mm	wft	19.99	wft	wft	33.97	33.97
is	5142.3	5142.3	5142.3	5142.3	3628.2	3628.2
ftf1	3628.2	3628.2	3628.2	3628.2	7	7
th	1.000	1.000	1.000	1.000	1.000	1.000
ins	nm	nm	nm	nm	nm	nm
cdc	ph	ph	ph	ph	ph	ph





JC2175\_13C\_CDC13

exp2 s2pu1

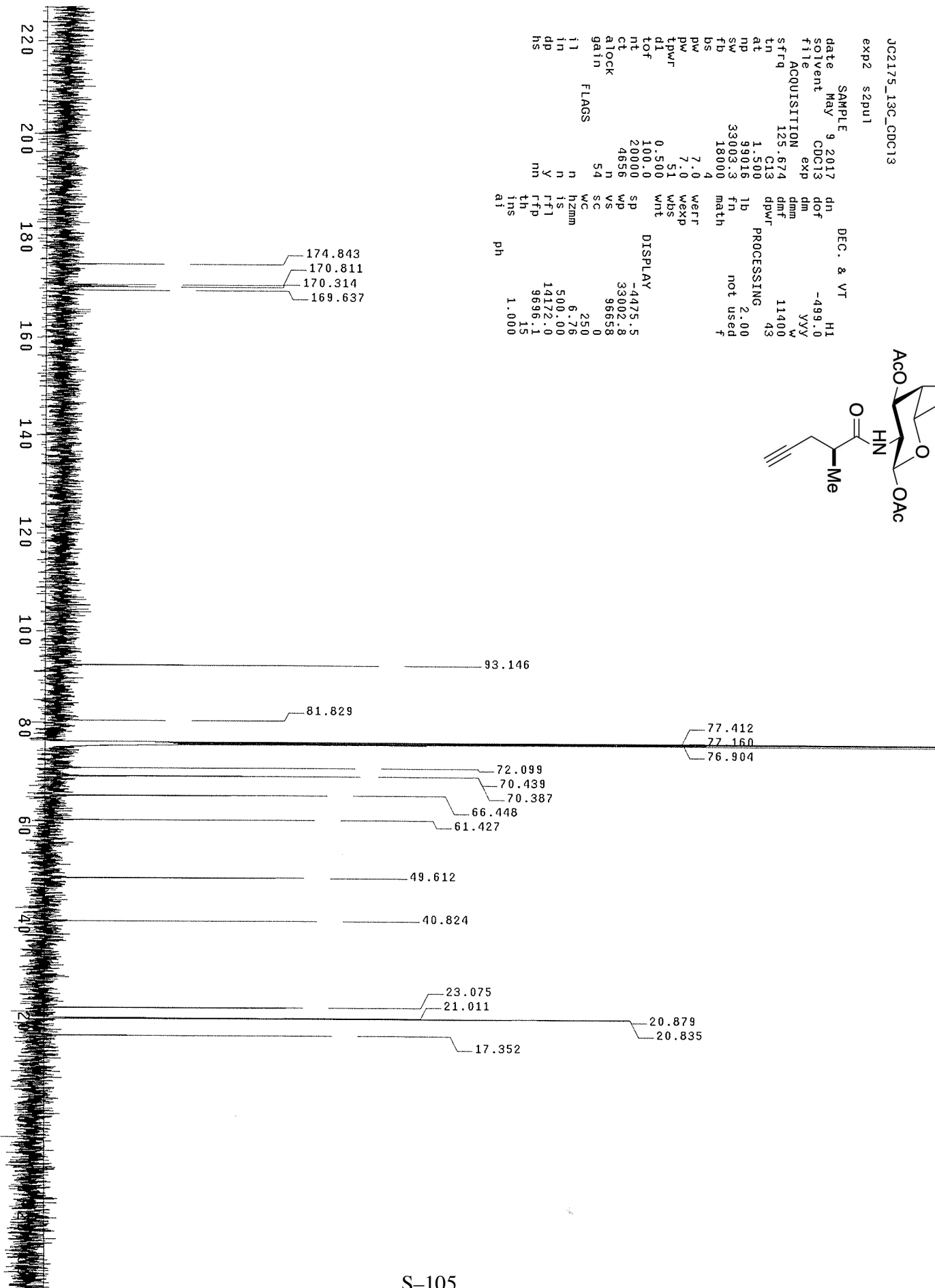
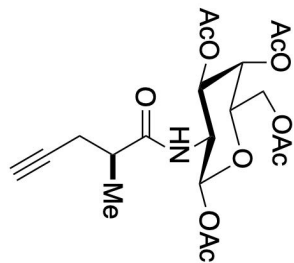
SAMPLE 9 2017  
 date May 9 2017  
 solvent CDC13  
 file exp  
 ACQUISITION  
 sfrq 125.674  
 tn 613  
 at 1.500  
 mp 93016  
 sw 33003.3  
 fb 18000  
 bs 4  
 pw 7.0  
 pwr 7.0  
 tpwr 51  
 d1 0.500  
 tof 100.0  
 nt 20000  
 ct 4656  
 a10ck n  
 gain 54  
 FLAGS  
 i1 n  
 in n  
 dp y  
 hs nn

DEC. & VT  
 H1  
 -499.0  
 YYY  
 W  
 11400  
 43

PROCESSING  
 2.00  
 not used  
 f

DISPLAY  
 -4475.5  
 33002.8  
 96658  
 0  
 250  
 6.76  
 500.00  
 14172.0  
 9696.1  
 15  
 1.000

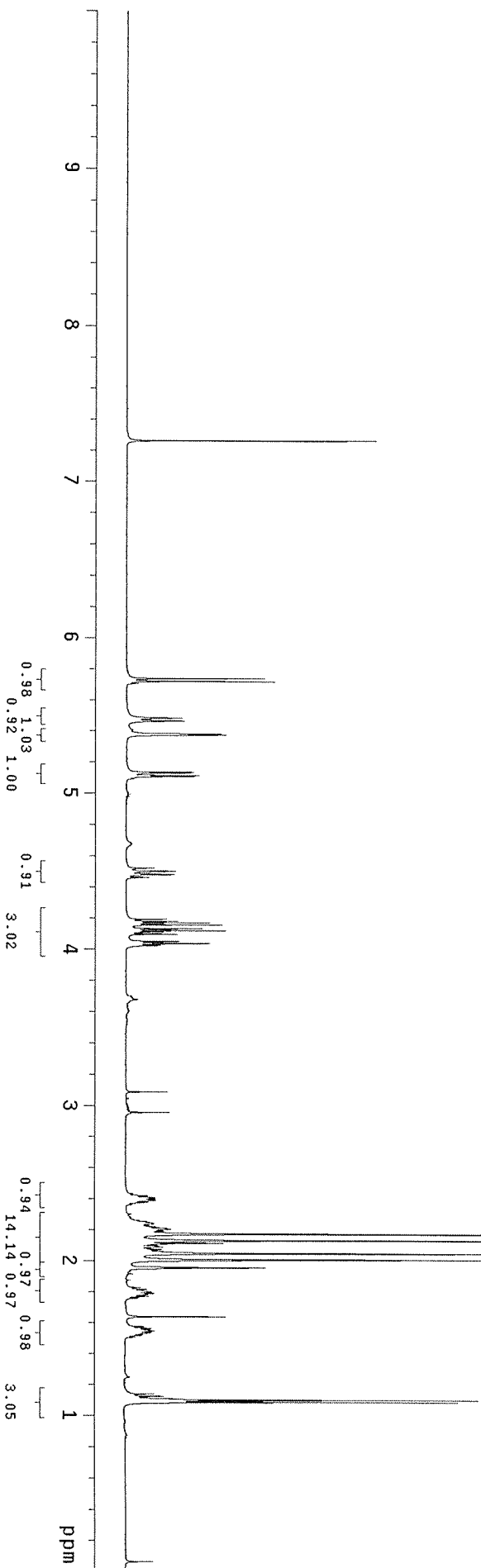
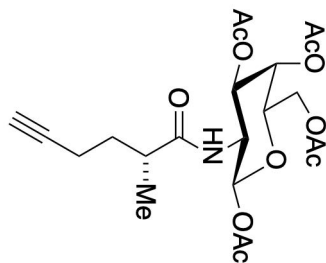
ph



JC3047\_1H\_CDCl3

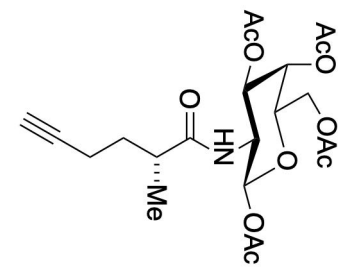
expt1 s2pul1

date	MAY 10 2017	DEC. & VT	499.751
solvent	CDCl3	exp	H1
file	exp	dpwr	30
file	ACQUISITION	dof	0
sfrq	499.751	dm	nmn
fn	H1	dmm	nmn
at	4.000	dmt	C
np	64000	dseq	200
sw	8000.0	dres	1.0
fb	4000	homo	n
bs	4	dfreq2	DEC2
tpwr	60	dn2	0
pw	8.0	dpwr2	1
dl	0	dot2	0
tof	0	dm2	n
nt	128	dmm2	n
ct	100	dmf2	C
alock	n	dres2	200
gain	40	homo2	1.0
fl	n	PROCESSING	n
in	Y	wtfile	ft
dp	nm	proc	fn
hs	nm	math	f
sp	0.0	DISPLAY	ft
wp	4997.3	werr	65536
vs	157	wexp	f
sc	0	wds	f
wc	250	wnt	wft
h2mm	19.99		
is	33.97		
rfl	5142.3		
rflp	3628.2		
th	7		
ins	1.000		
nm	cdc	ph	



JC3047\_13C\_CDC13

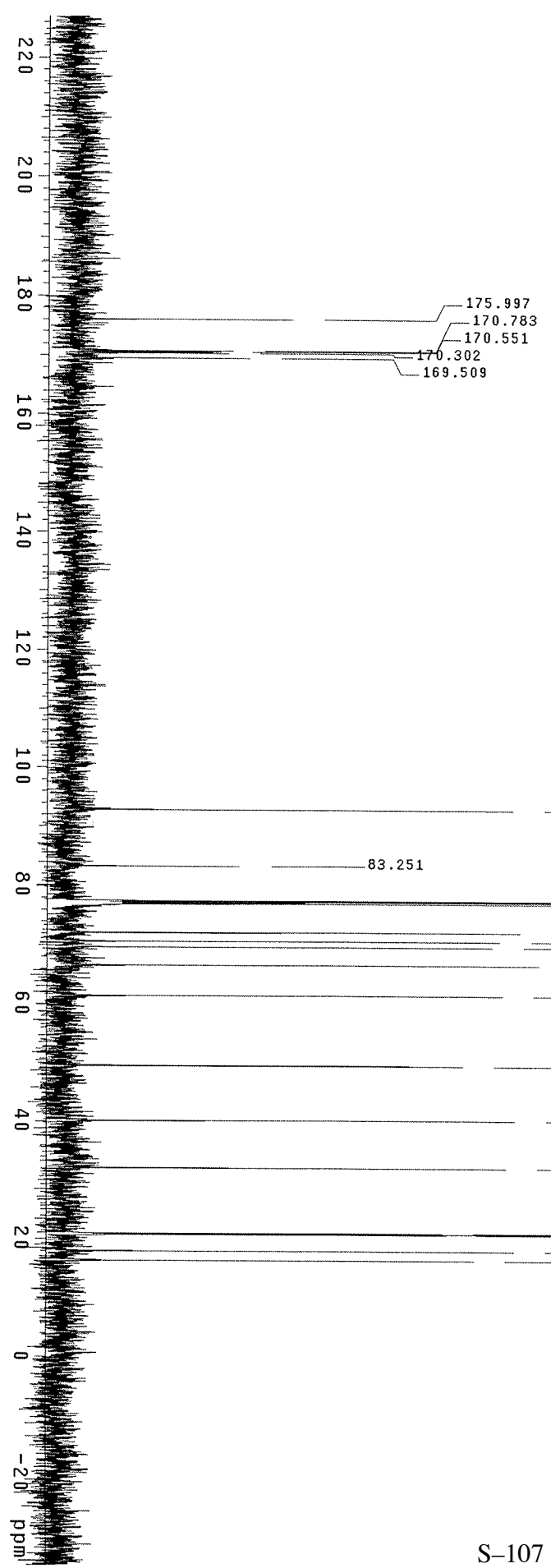
exp2 s2pu1



SAMPLE May 10 2017  
 solvent CDC13  
 file exp  
 ACQUISITION  
 sfrq 125.674  
 tn C13  
 at 1.500  
 np 99016  
 sw 33003.3  
 fb 18000  
 bs 4  
 pw 7.0  
 wexp 7.0  
 tpwr 51  
 di WBS  
 tof 0.500  
 nt 100.0  
 ct 2000  
 gain 904  
 alock n  
 gain 54  
 i1 n  
 in n  
 dp y  
 hs nn

DEC. & VT H1  
 dn -499.0  
 dof YYY  
 dm W  
 dmm 11400  
 dmf 43  
 dpwr  
 PROCESSING 2.00  
 lb 2.00  
 fn not used  
 math f

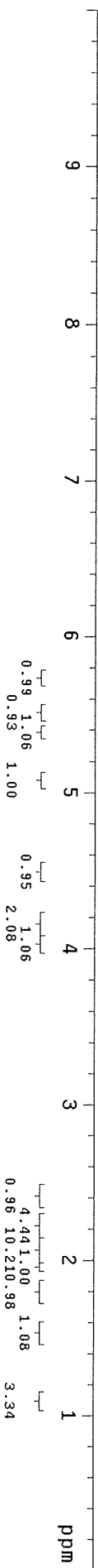
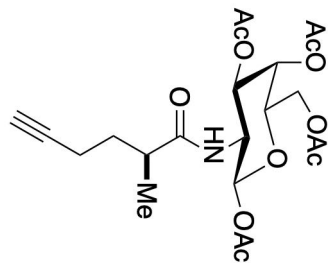
DISPLAY  
 sp -4476.0  
 wp 33002.8  
 vs 43648  
 sc 0  
 wc 250  
 hzmm 1.49  
 is 509.00  
 rfi 14172.5  
 rfp 9696.1  
 th 21  
 ins 1.000  
 ai



JC3061\_1H\_CDCl3

exp1 s2pu1

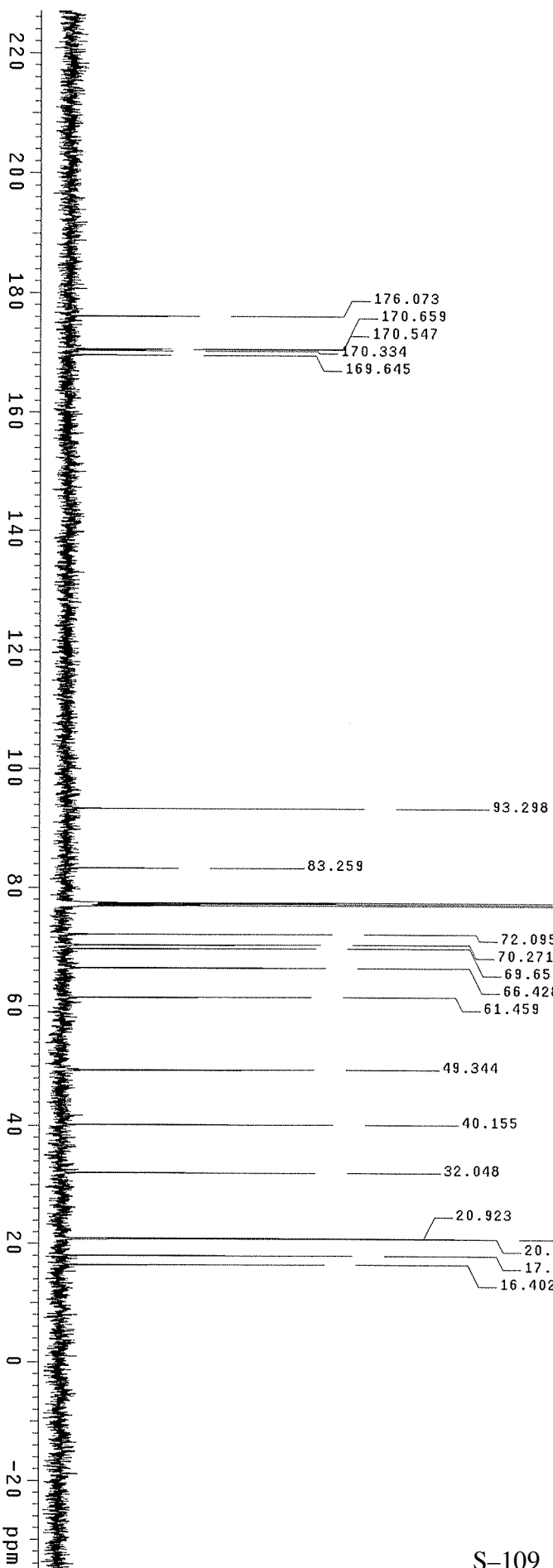
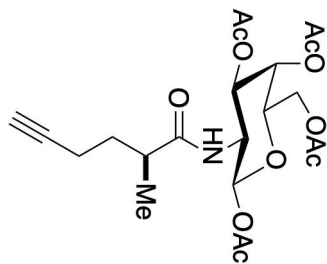
date	MAY 10 2017	DEC. & VT	499.751
solvent	CDCl3	exp	H1
file	exp	dof	30
ACQUISITION	499.751	dm	0
sfreq	499.751	dm	nmn
tn	H1	dmm	nmn
at	4.000	dmt	C
np	64000	dseq	200
sw	8000.0	dres	1.0
fb	4000	homo	n
bs	4	DEC2	n
tpwr	60	dfreq2	0
pw	8.0	dn2	0
d1	0	dpwr2	1
tof	0	dof2	0
nt	128	dm2	n
ct	100	dmm2	n
clock	n	dmf2	C
gain	40	dseq2	200
FLAGS	n	dres2	1.0
il	n	homo2	n
in	n	PROCESSING	n
dp	y	wf file	ft
hs	nn	proc	f
DISPLAY	nn	math	65536
sp	0.0	math	f
wp	4997.3	werr	
vs	381	wexp	
sc	0	wbs	
wc	250	wnt	
h2mm	19.99	wft	
f	33.37		
rfl	5142.3		
rflp	3628.2		
th	7		
ins	1.000		
nm	cdc	ph	



JC3061\_13C\_CDCl3

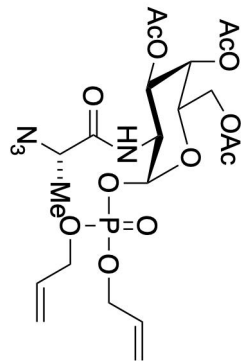
exp2 s2pu1

SAMPLE	date	May 10 2017	DEC. & VT	H1
solvent	CDCl3			-499.0
file	exp		dm	YY
ACQUISITION	exp		dmm	YY
sfrq	125.674		w	11400
tn	C13		dpwr	43
at	1.500		PROCESSING	2.00
np	99016		lb	not used
sw	33003.3		fn	f
fb	18000		math	
bs	4		weff	
pw	7.0		wexp	
tpwr	51		wbs	
di	0.500		wmt	
tof	100.0		DISPLAY	
nt	2000		sp	-4476.5
ct	1264		wp	33002.8
atlock	n		vs	23392
gain	54		sc	0
FLAGS			wc	250
i1	n		h2mm	6.07
in	n		is	500.00
dp	y		ftl	14173.0
hs	nm		rftl	9696.1
			th	12
			ins	1.000
			ai	

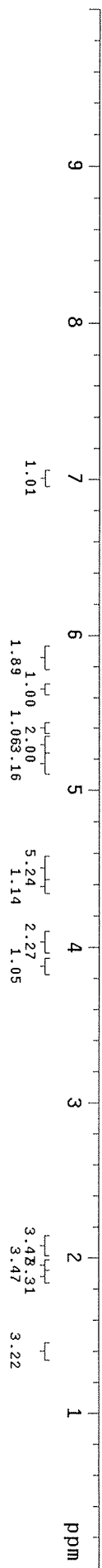


JC5142\_1H\_CDC13

exp2 s2pu1



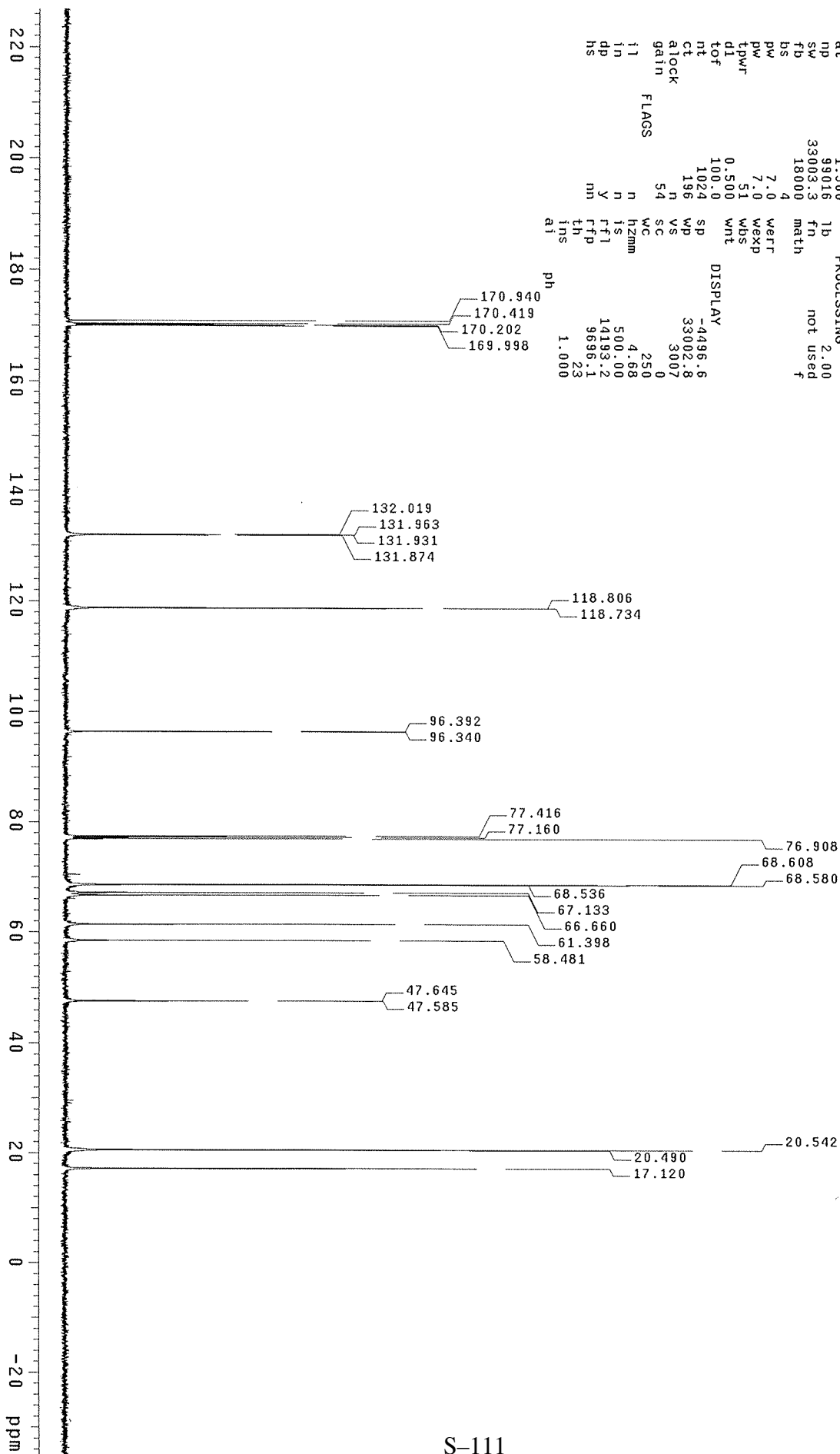
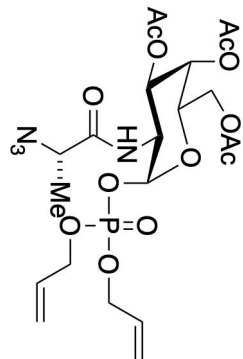
SAMPLE DEC. & VT  
 date Jun 14 2017 499.751  
 solvent CDC13 dn H1  
 file exp dpwr 30  
 ACQUISITION dof 0  
 sfrq 499.751 dm mmm  
 tn H1 dm C  
 at 4.000 dmf 200  
 np 64000 dseq  
 sw 8000.0 dres 1.0  
 fb 4000 homo n  
 bs 4  
 tpwr 60 dffq2 DEC2  
 pw 8.0 dn2 0  
 dl 0 dpwr2 1  
 tof 0 dof2 0  
 nt 16 dmm2 n  
 ct 16 dmf2 C  
 atlock n dseq2 200  
 gain 40  
 FLAGS  
 i1 n homo2 1.0  
 in n  
 dp y wfile PROCESSING  
 hs nm wfile  
 DISPLAY 0.0 ft  
 sp 4997.3 fn 65536  
 wd 151 math f  
 vs 150 weff  
 sc 250 wexp  
 wc WDS  
 hzmm 19.99 wnt  
 is 33.57  
 rfl 5141.8  
 rfp 3628.2  
 th 7  
 ins 1.000  
 nm cdc ph



JC5142\_13C\_CDCl3

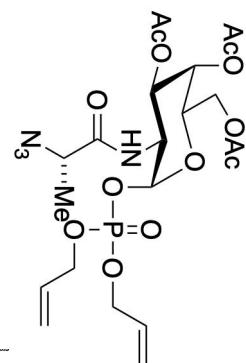
exp2 s2pu1

SAMPLE Jun 14 2017 dh DEC. & VT H1  
solvent CDC13 dof -499.0  
file exp dm  
ACQUISITION 125.674 dmm 11400  
sfrq 43  
in C13 dpwr  
at 1.500 1b PROCESSING 2.00  
np 99016 fn not used f  
sw 33003.3 math  
fb 18000  
bs 4  
pw 7.0 weft  
pv 7.0 wexp  
tpwr 51 wbs  
dl 0.500 wnt  
tof 100.0  
nt 1024 sp DISPLAY -4996.6  
ct 196 wp 33002.8  
atlock n vs 3007  
gain 54 SC 250  
FLAGS n WC 4.68  
i1 n hzmm  
in n 500.00  
dp y ffl 14193.2  
hs nn rffl 9696.1  
th ins 23  
ai 1.000



JCS142\_31P\_CD013

exp2 s2pu1



SAMPLE DEC. & VT  
 date Jun 14 2017 dfrq 499.751  
 solvent CDC13 dn H1  
 file exp dpwr 43  
 ACQUISITION exp dof 0  
 sfrq 202.297 dm YYY  
 tn P31 dmm W  
 at 1.002 dmf 11400  
 np 160254 dseq  
 SW 80000.0 dres 1.0  
 fb 44000 homo n  
 bs 4  
 tpwr 51 dfrq2 DEC2  
 pw 6.6 dn2 0  
 dl 2.000 dpwr2  
 lof 0 dot2 1  
 nt 32 dm2 n  
 ct 24 dmm2 n  
 alock n dmf2 C  
 gain 30 dseq2 9900  
 FLAGS dres2 1.0  
 i1 n homo2 n  
 in y lb PROCESSING  
 dp n wtfile 2.00  
 hs nn  
 DISPLAY  
 sp -45138.5 fn ft  
 wp 79999.4 math not used  
 vs 819  
 SC WC 250 Weff  
 WC hzmm 1.37 WeXP  
 IS 500.00 WBS  
 r-fl 45139.2 Wnt  
 r-fl 0  
 th 17  
 ins 1.000  
 ai cdc ph





JC1191\_1H\_CDCl3

exp2 s2pul

SAMPLE DEC. & VT

date Jun 8 2017 dfrq 499.751

solvent CDCl3 dn H1

file exp dpwr 30

ACQUISITION 499.751 dm 0

sfreq 499.751 dm mm

tn H1 dmm C

at 4.000 dmf 200

np 64000 dseq

sw 8000.0 dres 1.0

fb 4000 homo n

bs 4 dfrq2 DEC2

tpwr 60 dn2 0

pw 8.0 dpwr2 1

d1 0 dot2 0

tof 1000 dm2 n

nt 52 dmm2 n

ct dmf2 C

alock n dmf2 200

gain 40 dseq2

flags 40 dres2 1.0

il n homo2 n

in n wtfile PROCESSING

dp y fn math ft

hs nm proc fn 65536

sp 0.0 math f

wp 4997.3 werr

vs 151 wexp

sc 0 wds

wc 250 wnt

hzmm 19.99

ts 33.57

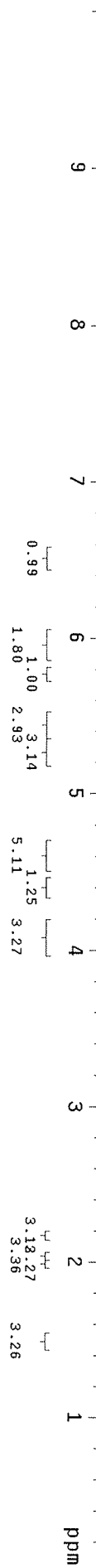
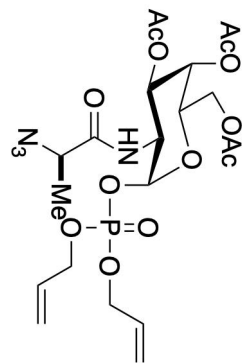
rfl 5142.3

rtp 3628.2

th 7

ins 1.000

nm cdc ph



JC1191\_13C\_CDC13

exp2 s2pul1

SAMPLE DEC. & VT

date Jun 8 2017 dn H1

solvent CDC13 dof -499.0

file exp dm dmm yyy

ACQUISITION 125.674 dmf 11400

sfrq 125.674 dpwr 43

tn 1.500 PROCESSING 2.00

at 99016 1b fn not used

np 33003.3 math

sw 18000

fb 4

bs 7.0 weff

pw 7.0 wexp

tpwr 51 wds

dl 0.500 wnt

tof 100.0

nt 17000

ct 2296

gain 54

flags n

in n

dp n

hs y

ai n

ins n

th n

ph 1.000

ai

ai

ai

ai

ai

ai

ai

ai

ai

ai

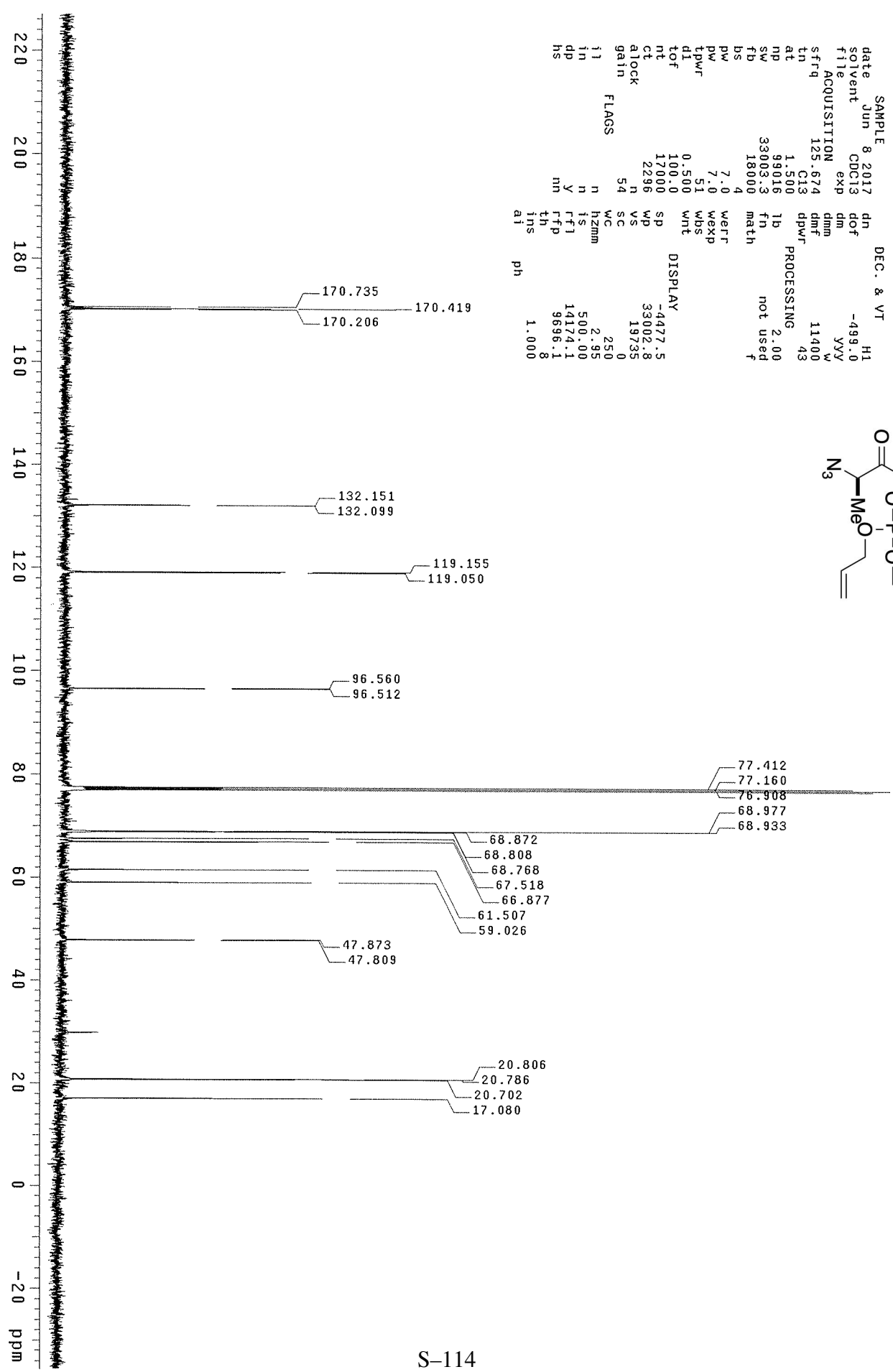
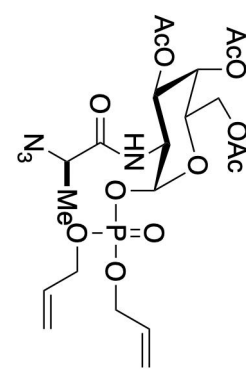
ai

ai

ai

ai

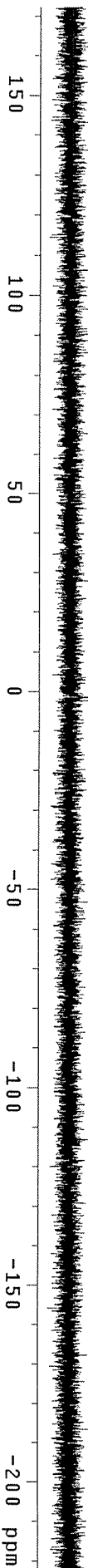
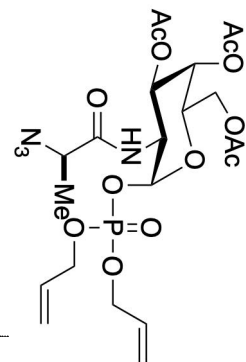
ai



JC1191\_31P\_CDC13

exp2 s2pul1

SAMPLE DEC. & VT  
 date Jun 9 2017 dfrq 499.751  
 solvent CDC13 dn H1  
 file exp dpwr 43  
 ACQUISITION exp dof 0  
 sfrq 202.297 dm YYY  
 tn P31 dmm W  
 at 1.002 dmf 11400  
 np 160254 dseq  
 sw 80000.0 dres 1.0  
 fb 44000 homo n  
 bs 4  
 tpwr 51 dfrq2 DEC2  
 pw 6.6 dn2 0  
 d1 2.000 dpwr2  
 lof 0 dot2 1  
 nl 10000 dm2 0  
 ct 24 dmm2 n  
 alock n dmf2 C  
 gain 30 dseq2 9900  
 FLAGS dres2 1.0  
 i1 n homo2 n  
 in n  
 dp Y lb PROCESSING  
 hs nn wfile 2.00  
 DISPLAY  
 sp -45159.3 fn ft  
 wp 79999.4 fn not used  
 vs 31275 math f  
 sc 0 werr  
 wc 250 wexp  
 hzmm 150.09  
 IS 500.00 WDS  
 rffl 45159.9 wnt  
 th 0  
 ins 17  
 ai cdc ph 1.000



JC5155\_1H\_CDCl3

exp2 s2pu1

SAMPLE

DEC. & VT

499.751

date Jun 24 2017

dfreq

H1

solvent ODC13

dn

30

file

dpwr

0

ACQUISITION

dof

0

sfrq

dm

nnn

tn

dmm

c

at

dmf

200

mp

dseq

4.000

sw

dres

64000

fd

homo

8000.0

bs

homo

40000

tpwr

DEC2

4

pw

dfreq2

60

d1

dn2

8.0

tof

dpwr2

0

nt

dof2

0

ct

dm2

16

atock

dmm2

c

gain

dmf2

200

FLAGS

dseq2

40

i1

dres2

1.0

ih

homo2

n

dp

wtfile

PROCESSING

ns

fn

ft

sp

proc

65536

wp

math

f

vs

werr

4997.3

sc

wexp

151

wc

wbs

0

hzmm

wnt

19.99

ts

wft

33.57

rfl

5141.6

th

3628.2

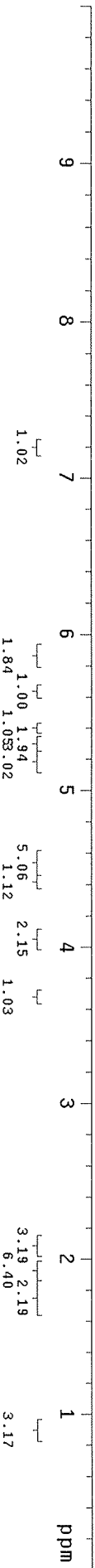
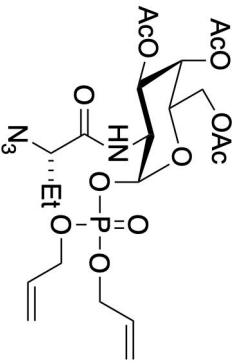
ins

1.000

nm

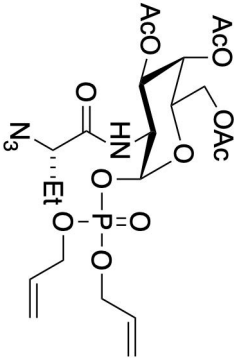
cdc

ph

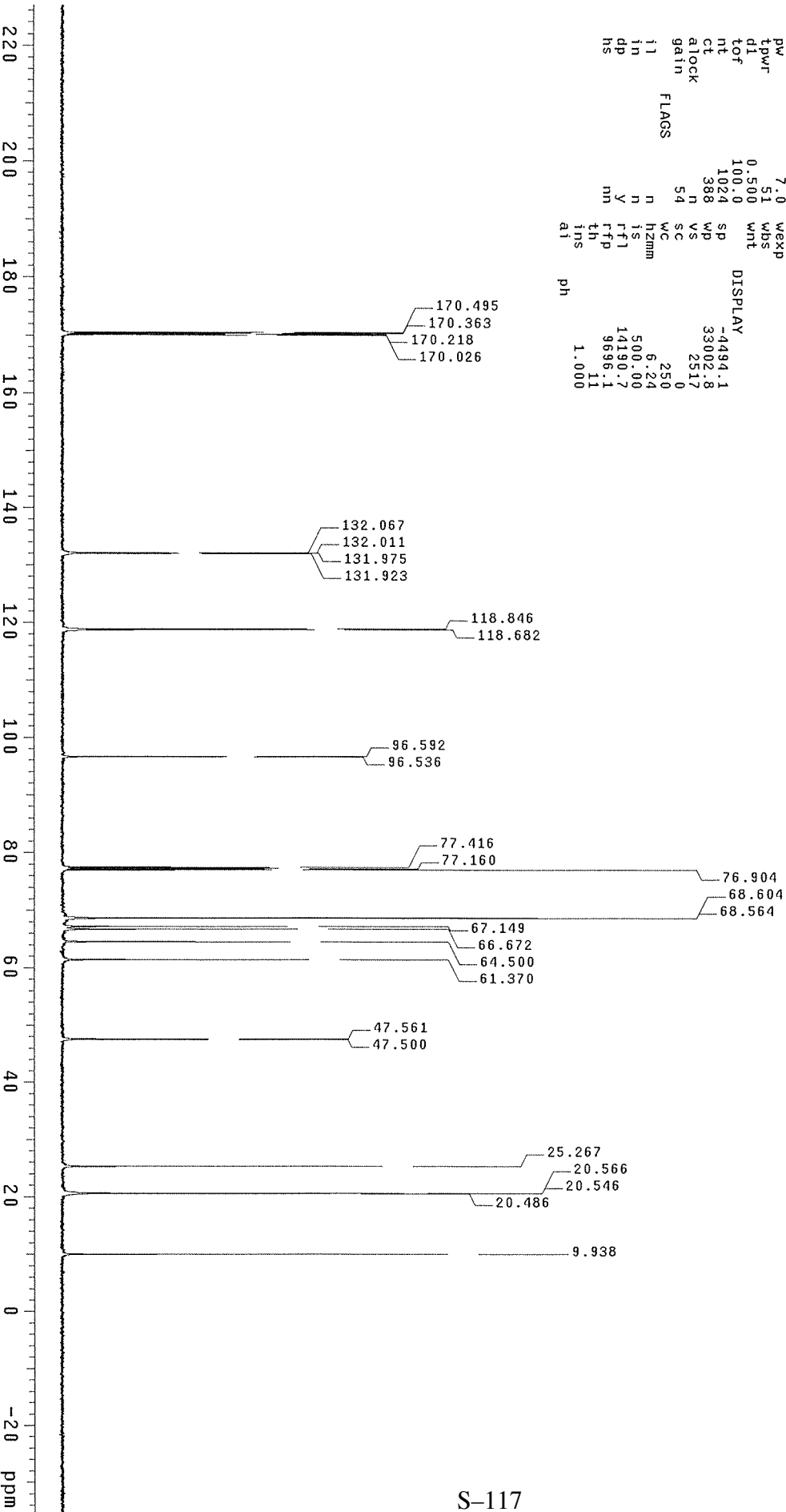


JG5155\_13C\_CDCl3

exp2 s2pu1



SAMPLE Jun 24 2017 DEC. & VT H1  
 solvent CDC13 dof -499.0  
 file exp dm YYY  
 ACQUISITION dm 11400  
 sfrq 125.674 dpwr 43  
 tn C13  
 at 1.500 1b  
 mp 99016 fn  
 sw 33003.3 not used f  
 fd 18000 math  
 bs 4  
 pw 7.0 weff  
 tpwr 7.0 wexp  
 dl 51 wbs  
 tof 0.500 wnt  
 nt 100.0  
 ct 1024 SP  
 alock 388 WP  
 gain 54 N VS  
 54 SC  
 FLAGS WC  
 l1 n hzmm 250  
 in n is 500.00  
 dp y rfl 14190.7  
 hs mn rfp 9696.1  
 th ins 11  
 al 1.000  
 DISPLAY  
 -4494.1  
 33002.8  
 2517  
 0  
 6.24  
 500.00  
 14190.7  
 9696.1  
 11  
 1.000  
 ph



JC1187\_31P\_CDC13

exp1 s2pu1

SAMPLE DEC. & VT

date Jun 9 2017 dfrq 499.751

solvent CDC13 dn H1

file ACQUISITION exp dppwr 43

sfrq 202.297 dm 0

tn P31 dmm YYY

at 1.002 dmf W

np 160254 dseq 11400

sw 80000.0 dres 1.0

fb 44000 homo n

bs 4 DECC2

tpwr 51 dfrq2 0

pv 6.6 dn2 1

d14 2.000 dppwr2 1

tof 0 dof2 n

nt 1000 dm2 n

ct 40 dmm2 C

atlock n dmf2 9900

gain 30 dres2 1.0

il n homo2 n

in n PROCESsing 2.00

dp y lb wtfille

hs nm wtfille ft

sp DISPLAY -45156.3 fn not used

wp 79999.4 math f

vs 116832

sc 0 weff

wc 250 wexp

hzmm 8.83 wbs

is 500.00 wnt

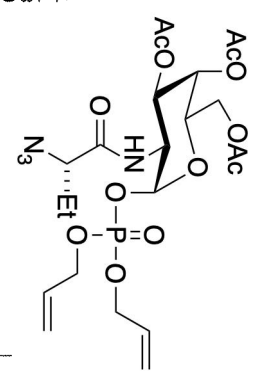
fft 45156.9

fft 0

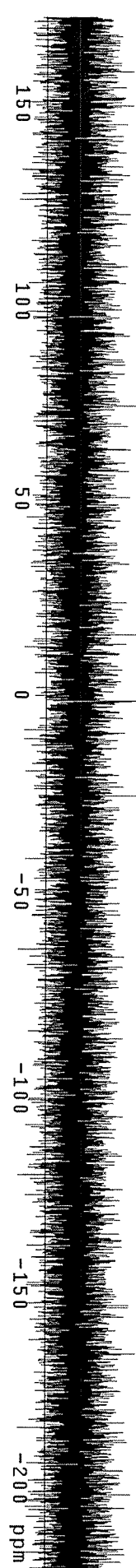
th 17

ins 1.000

ai cdc ph

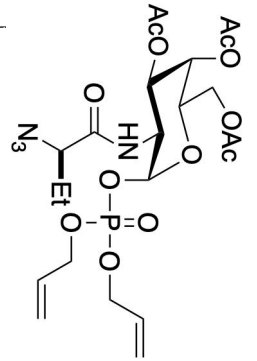


-1.472



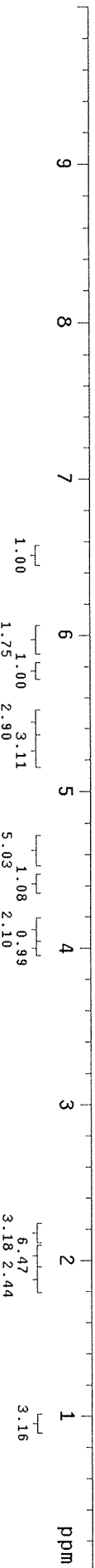
JC2017\_1H\_CDCl3

exp2 s2put1



SAMPLE Jun 13 2017 DEC. & VT 499.751  
 solvent CDCl3 dn H1  
 file ACQUISITION exp dpwr H1  
 ACQUISITION 499.751 dm 30  
 dof 0  
 mnm 0  
 tn H1 dm C  
 at 4.000 dmf 200  
 np 64000 dseq  
 sw 8000.0 dres  
 fb 4000 homo 1.0  
 bs 4  
 tpwr 60 dftq2 DEC2  
 pw 8.0 dn2 0  
 dl 0 dpwr2 1  
 tof 0 dof2 0  
 nt 128 dmm2 n  
 ct 128 dmm2 C  
 alock 40 dmf2 200  
 gain 40 dseq2  
 dres2  
 homo2 1.0  
 n

PROCESSING  
 wftlle  
 fn  
 math  
 DISPLAY 0.0  
 SP WP 4997.3 werr ft  
 VS SC 0 wexp f  
 WC 250 wbs  
 hzmm 19.99 wnt  
 is 33.57  
 rffl 5142.3  
 rfp 3628.2  
 th  
 ins 1.000  
 nm cdc ph



JC2017\_13C\_CDCl3

exp2 s2pu1

SAMPLE DEC. & VT

date Jun 13 2017 dn H1

solvent CDCl3 dof -499.0

file ACQUISITION exp dm YYY

strq 125.674 dmm W

th C13 dpwr 11400

at 1.500 43

mp 99016 1b

sw 33003.3 fn not used

fd 18000 math

bs 4

pw 7.0 wert

tpwr 7.0 wexp

d1 51 wnt

tof 0.500

nt 20000 sp -4476.0

ct 17776 wp 33002.8

atlock N VS 65342

gain 54 SC 250

11 n hzmm 0.25

in n IS 500.00

dp Y rffl 14172.5

hs n th rffp 9696.13

at ins 1.000

PROCESSING 2.00

not used f

DISPLAY -4476.0

33002.8

65342

250

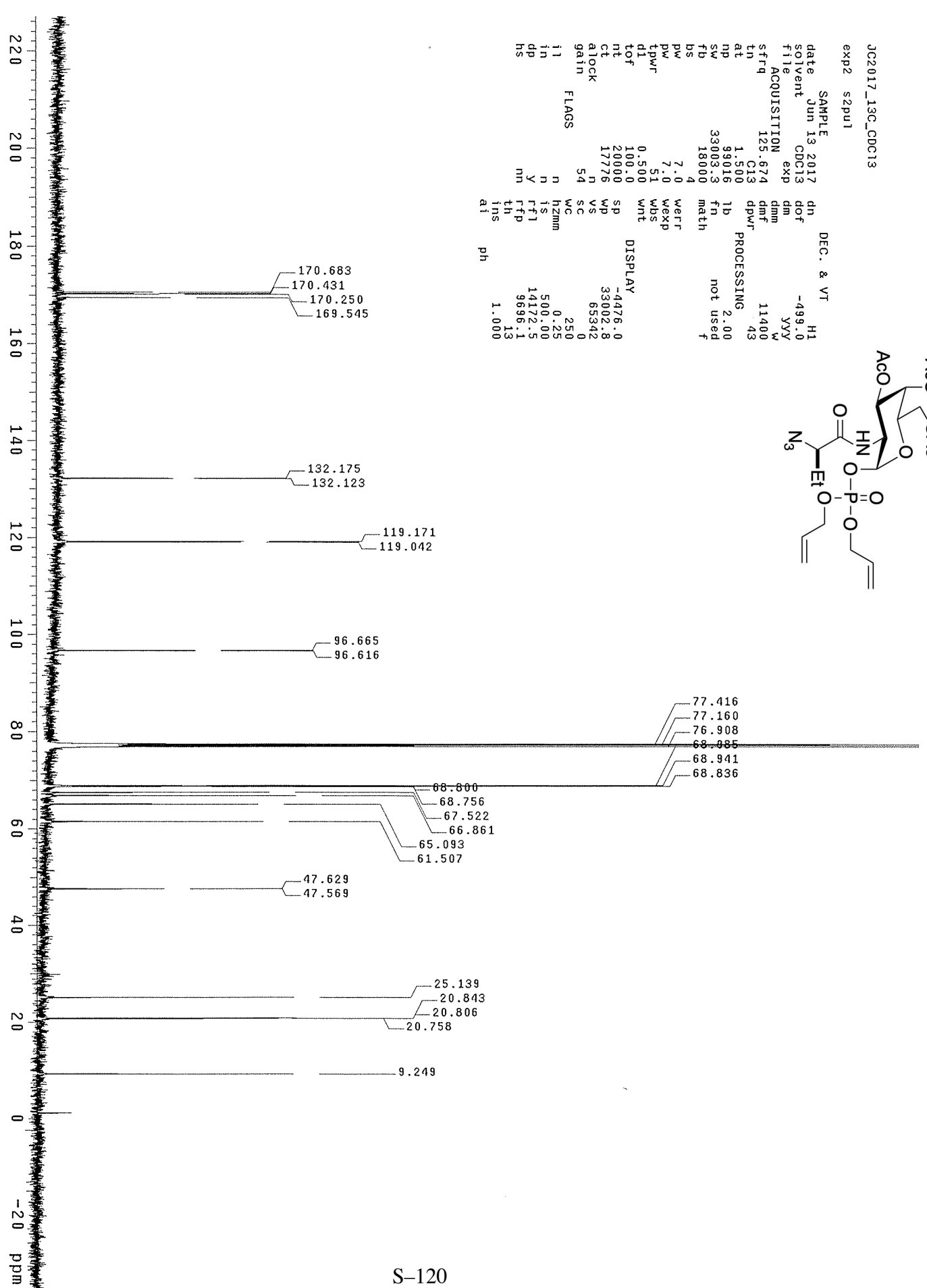
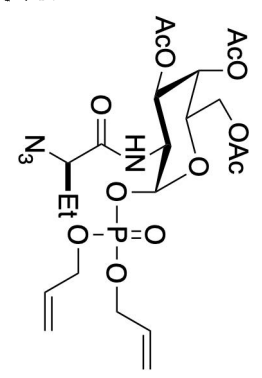
0.25

500.00

14172.5

9696.13

1.000





JC2017\_31P\_CDCC13

exp2 s2pu1

SAMPLE

DEC. & VT

499.751

date Jun 13 2017

dfreq

499.751

Hi

solvent CDC13

dn

43

Hi

f1file exp

dpwr

43

0

f2file ACQUISITION

doz

0

0

sfrq 202.297

dm

YYY

W

tn P31

dmm

11400

W

at 1.002

dmf

11400

W

mp 160254

dseq

1.0

n

sw 80000.0

dres

1.0

n

fb 44000.0

homo

1.0

n

bs 4

homo

1.0

n

tpwr 51

dfreq2

DEC2

0

pw 6.6

dn2

1

0

d1 2.000

dpwr2

1

0

tof 0

doz2

0

n

nt 1024

dm2

9900

C

ct 112

dmm2

9900

C

atlock n

dmf2

9900

C

gain 30

dseq2

1.0

n

flags n

dres2

1.0

n

il n

homo2

1.0

n

in n

proc

2.00

n

dp y

wtfile

2.00

n

hs nh

proc

not used

f

sp DISPLAY

fn

not used

f

wp -45162.4

math

not used

f

vs 79993.4

math

not used

f

sc 28709

weff

0

0

wc 0

wexp

0

0

hzmm 250

wbs

0

0

is 9.25

wnt

0

0

ffl 500.00

ffl

0

0

rfp 45163.0

th

17

17

th 17

th

17

17

als 1.000

als

1.000

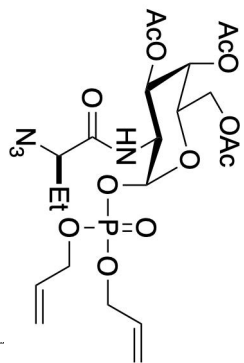
1.000

at cdc ph

at

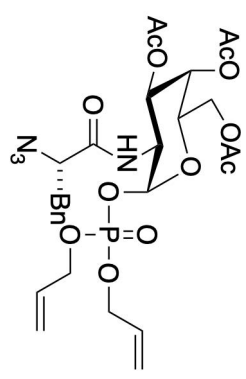
cdc

ph

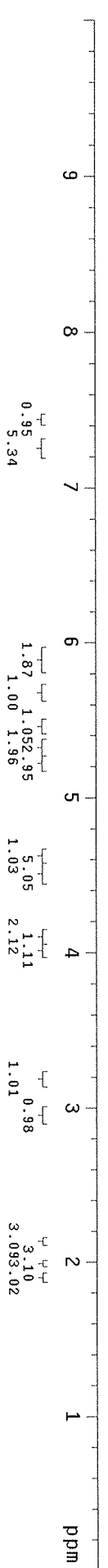


1.358





JC5136\_1H\_CDCl3  
 exp2 s2pul1  
 SAMPLE Jun 13 2017 DEC. & VT 499.751  
 solvent CDCl3 dn  
 file exp dpwr HI  
 ACQUISITION 30  
 dof 0  
 sfrq 499.751 dm nnn  
 tn H1 dmm C  
 at 4.000 dmf 200  
 np 64000 dseq  
 sw 8000.0 dres  
 fd 40000 homo 1.0 n  
 ds 4  
 tpwr 60 dfrq2 DEC2  
 pw 8.0 dn2 0  
 dl 0 dpwr2 1  
 tof 0 dof2 0  
 nt 16 dmm2 n  
 ct 16 dmf2 C  
 atock n dmf2 200  
 gain 40 dseq2  
 flags n dres2  
 l1 n homo2 1.0 n  
 in n  
 dp y wtfile PROCESSING  
 hs n fn  
 DISPLAY 65536 f  
 SD 0.0 math  
 WP 4997.3 werr  
 VS 151 wexp  
 SC 0 wbs  
 WC 250 wnt  
 hzmm 19.99  
 is 33.57  
 ffl 5129.6  
 rfp 3628.2  
 th  
 ins 1.000  
 nm cdc ph



JCS136\_13C\_CDCl3

exp2 s2pu1

SAMPLE Jun 13 2017 DEC. & VT H1

solvent CDCl3 dof -439.10

file ACQUISITION exp dm YYY

sfrq 125.674 dmf W

in C13 dpwr 11400

at 1.500 1b 43

mp 99016 fn not used

sw 33003.3 math

fd 18000

bs 4

pw 7.0 werr

tpwr 7.0 wexp

d1 51 wbs

nt 0.500 wnt

tof 100.0

nt 1024 sp

ct 132 wp

atlock N VS

gain 54 SC

11 n hzmm

in n is

dp Y rffl

hs mn th

ins

at

ph

1.000

500.00

14197.2

9996.1

17

2.250

1.250

33002.8

2374

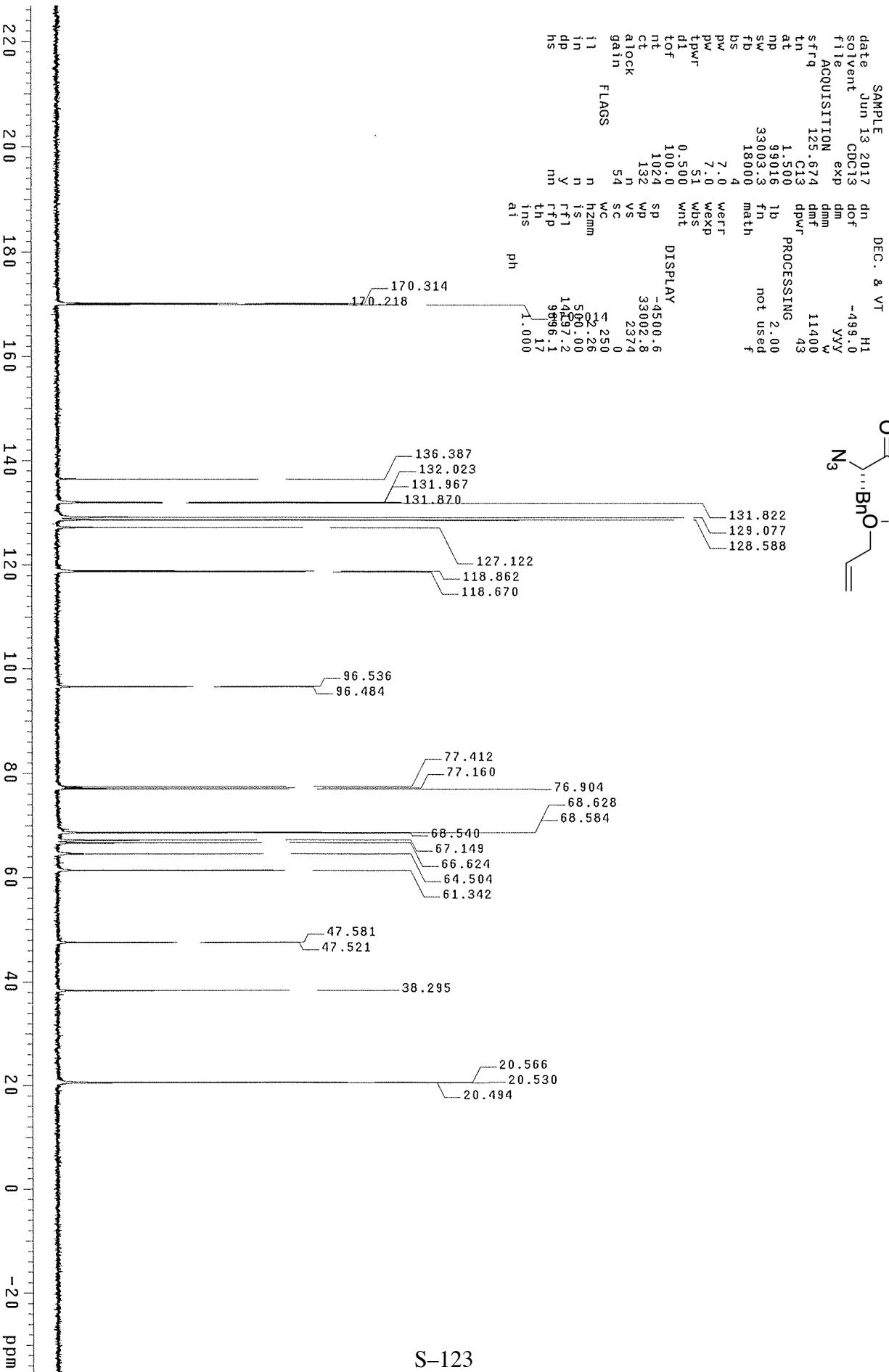
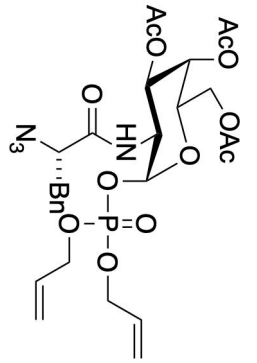
0

0

0

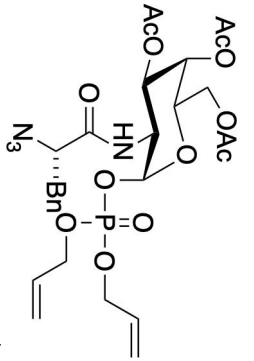
0

0



JC5136\_31P\_CD013

exp2 s2pu1



SAMPLE Jun 13 2017 DEC. & VT 499.751  
 solvent CDC13 dn H1  
 file exp dpwr 43  
 ACQUISITION dof 0  
 sfrq 202.297 dm YYY  
 tn P31 dmm W  
 at 1.002 dmf 11400  
 np 160254 dseq  
 SW 80000.0 dres 1.0  
 fd 44000 homo n  
 bs 4  
 tpwr 51 dfrq2 DEC2 0  
 pw 6.6 dn2  
 dl 2.000 dpwr2 1  
 tof 0 dof2 0  
 nt 64 dmm2 n  
 ct 12 dmf2 C  
 alock n  
 gain 30 dseq2 9900  
 FLAGS dres2  
 i1 n homo2 1.0  
 in n  
 dp y  
 hs n  
 DISPLAY nn  
 SP -45149.5  
 WD 79999.4 math not used  
 VS 981  
 SC 0 weff  
 WC 250 wexp  
 hzmm 7.99 wbs  
 is 500.00 wnt  
 rfi 45150.1  
 rfp 0  
 th 17  
 ins 1.000  
 at cdc ph



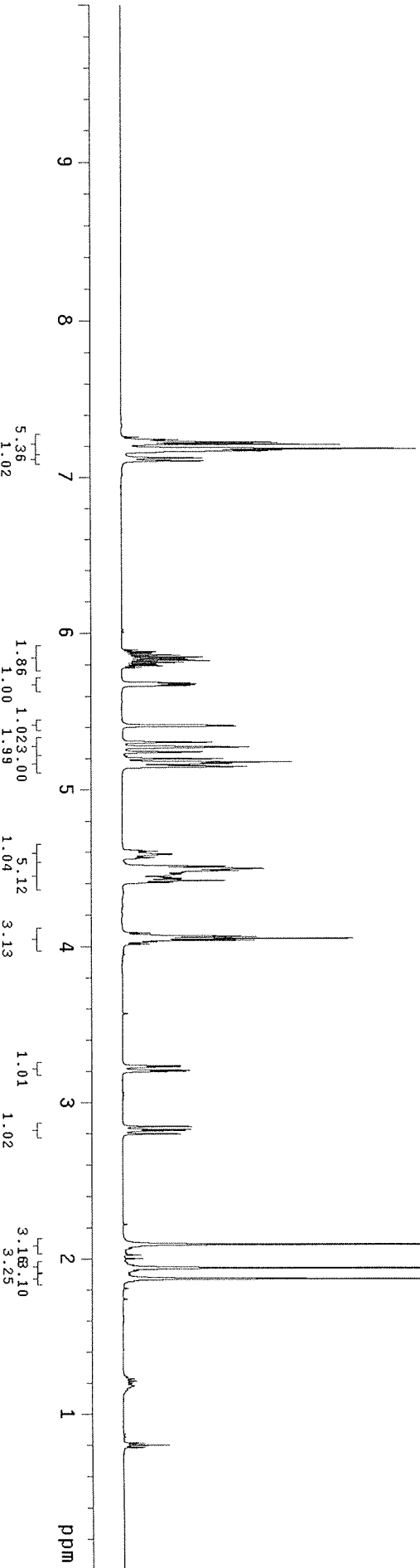
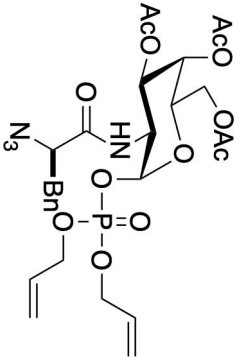
JG5137\_1H\_CDCl3

exp2 s2pu1

```

SAMPLE          DEC. & VT
date  Jun 13 2017  499.751
solvent  CDC13
f1le      exp
f2le      dpwr
ACQUISITION  499.751
sfrq      H1
in        dm
at        dmm
np        dmf
sw        dseq
fb        dres
hd        homo
bs        4
tpwr      60
pw        8.0
d1        0
tof       0
nt        16
ct        16
alock     n
gain      40
flags     40
l1        n
ln        n
dp        y
hs        n
          wtfile
          PROCESsing
          ft
          fn
          math
          65536
          f

DISPLAY  0.0
          4997.3
          IS1
          0
          250
          19.99
          33.57
          1514.4
          0
          7
          1.000
nm  cdc  ph
  
```



JG5137\_13C\_CDCl3

exp2 s2pu1

SAMPLE Jun 13 2017 DEC. & VT H1

solvent CDC13 dof -499.0

file ACQUISITION exp dim YYY

sfrq 125.674 dmf dmm W

th C13 dpwr 11400 43

at 1.500 1b PROCESSING 2.00

np 99016 fn not used f

sw 33003.3 math

fd 18000 4

bs 7.0 werr

pw 7.0 wexp

tpwr 51 wbs

dl 0.500 wnt

tof 100.0 sp

nt 1024 wp

ct 192 vs

atlock N SC

gain 54 WC

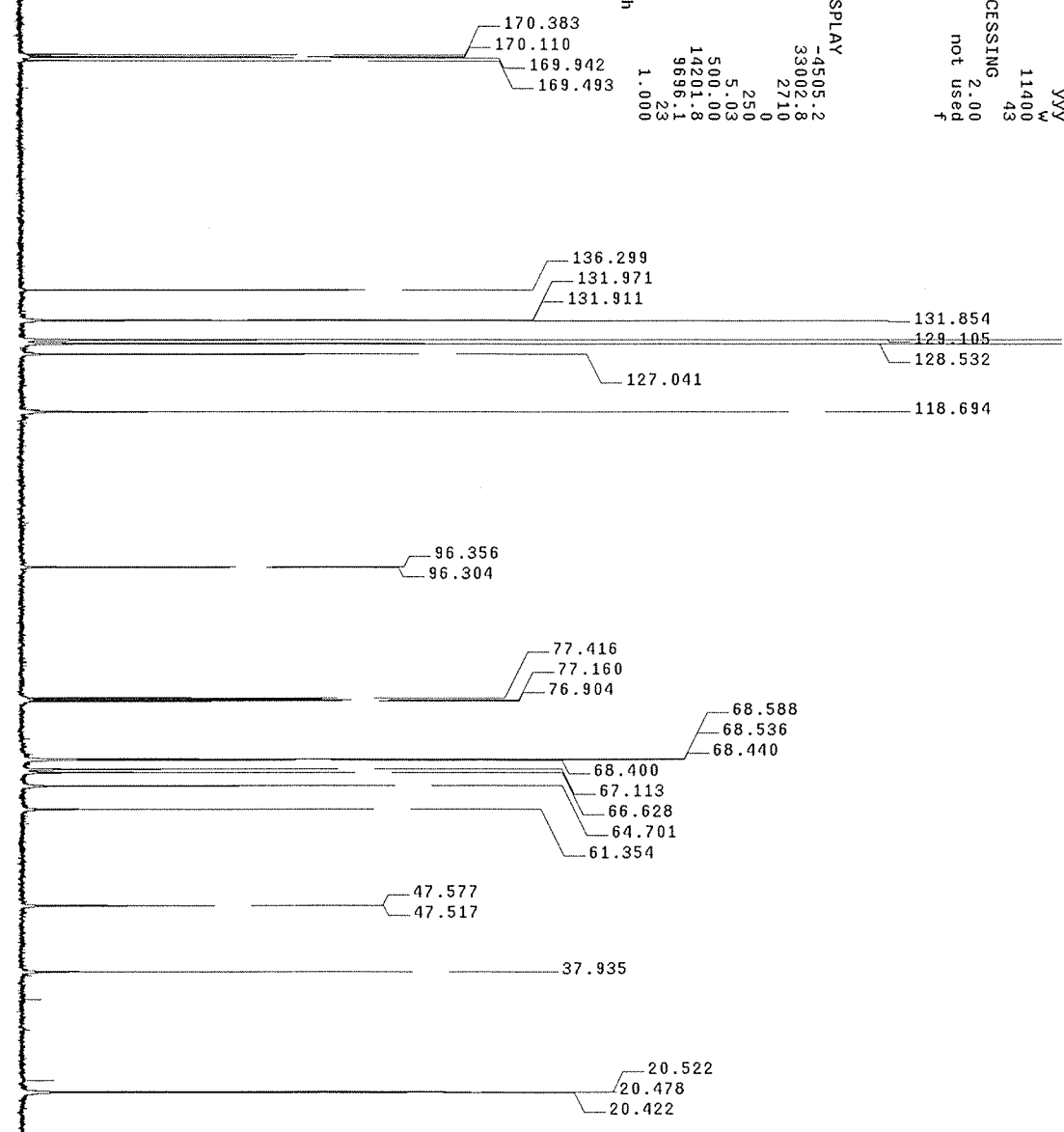
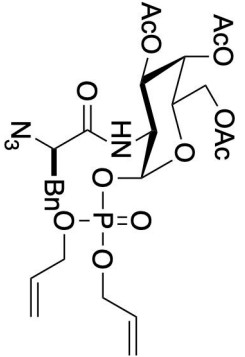
l1 n hzmm

in n is

dp Y rfl

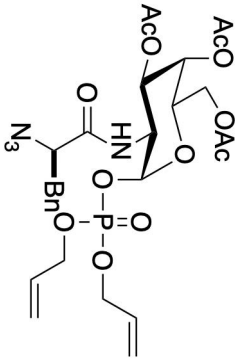
hs mn ths

PARAMETER	VALUE
DATE	Jun 13 2017
SOLVENT	CDC13
FILE	ACQUISITION
SFRQ	125.674
TH	C13
AT	1.500
NP	99016
SW	33003.3
FD	18000
BS	7.0
PW	7.0
TPWR	51
DL	0.500
TOF	100.0
NT	1024
CT	192
ATLOCK	N
GAIN	54
L1	n
IN	n
DP	Y
HS	mn



JCS137\_31P\_CD013

exp2 s2pu1

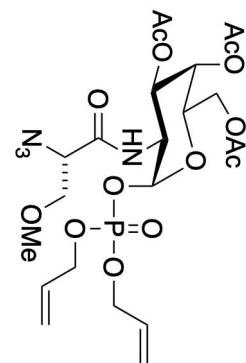


SAMPLE Jun 13 2017 DEC. & VT 499.751  
 solvent CDC13 dn H1  
 file exp dpwr 43  
 ACQUISITION dof 0  
 srrq 202.297 dm YYY W  
 tn P31 dmm 11400  
 at 1.002 dmf 1.0  
 np 160254 dseq 1.0  
 sw 80000.0 dres n  
 fd 44000 homo  
 bs 4  
 tpwr 51 dfrq2 DEC2 0  
 pw 6.6 dn2  
 dl 2.000 dpwr2 1  
 tof 0 dof2 0  
 nt 64 dm2 n  
 ct 16 dmm2 C  
 atock n dmf2 9900  
 gain 30 dseq2  
 dres2  
 homoz 1.0  
 i1 n  
 in n PROCESsing 2.00  
 dp y lb  
 hs mn wtfile  
 DISPLAY -45149.5 ft  
 sp -79999.4 fn not used  
 wd 1166 math  
 vs 0 werr  
 sc 250 wexp  
 wc 0.38 wbs  
 hzmm 500.00 wnt  
 is 45150.1  
 rfi 0  
 rfp 17  
 th  
 ins 1.000  
 al cdc ph

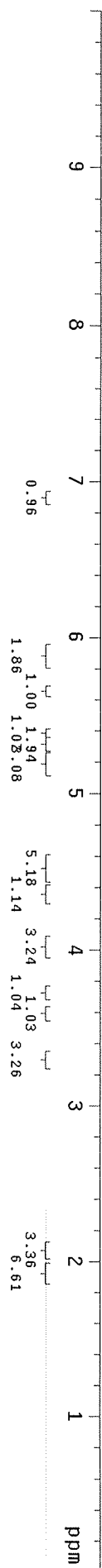


JCS146\_1H\_CDCl3

exp2 s2pul1



SAMPLE DEC. & VT  
 date Jun 24 2017 499.751  
 solvent CDC13 dn H1  
 file exp dpwr 30  
 ACQUISITION dof 0  
 sfrq 499.751 dm mm  
 tn H1 dmm C  
 at 4.000 dmf 200  
 np 64000 dseq  
 sw 8000.0 dres 1.0  
 fb 4000 homo n  
 bs 4  
 tpwr 60 dffq2 DEC2 0  
 pw 8.0 dn2  
 dl 0 dpwr2 1  
 lof 0 dof2 0  
 nt 16 dm2 n  
 ct 16 dmm2 C  
 atlock n dmf2 200  
 gain 40 dres2 1.0  
 f1 n homo2 n  
 in n  
 dp y wftfile PROCESSING  
 hs nm wftfile ft  
 DISPLAY 0.0 fn 65536  
 sp 4997.3 math  
 ws 151 weft  
 SC 250 weXP  
 WC 250 WDS  
 hzmm 19.99 wnt  
 is 33.57  
 rfi 5141.6  
 rfp 3628.2  
 th 7  
 ins 1.000  
 nm cdc ph





JC5146\_13C\_CDCl3

exp2 s2pul

SAMPLE DEC. & VT

date Jun 24 2017 dn H1

solvent CDC13 dof -499.0

file ACQUISITION exp dm yy

sfrq 125.674 dmm W

tn C13 dpwr 11400

at 1.500 PROCESSING 43

np 99016 1b 2.00

sw 33003.3 fn not used

fb 18000 math f

bs 4

pw 7.0 weff

pv 7.0 wexp

tpwr S1 WDS

d1 0.500 Wnt

tof 100.0 DISPLAY

nt 1024 SP -4494.6

ct 620 WP 33002.8

atock n VS 3639

gain 54 SC 250

FLAG

l1 n hzmm

ln n IS

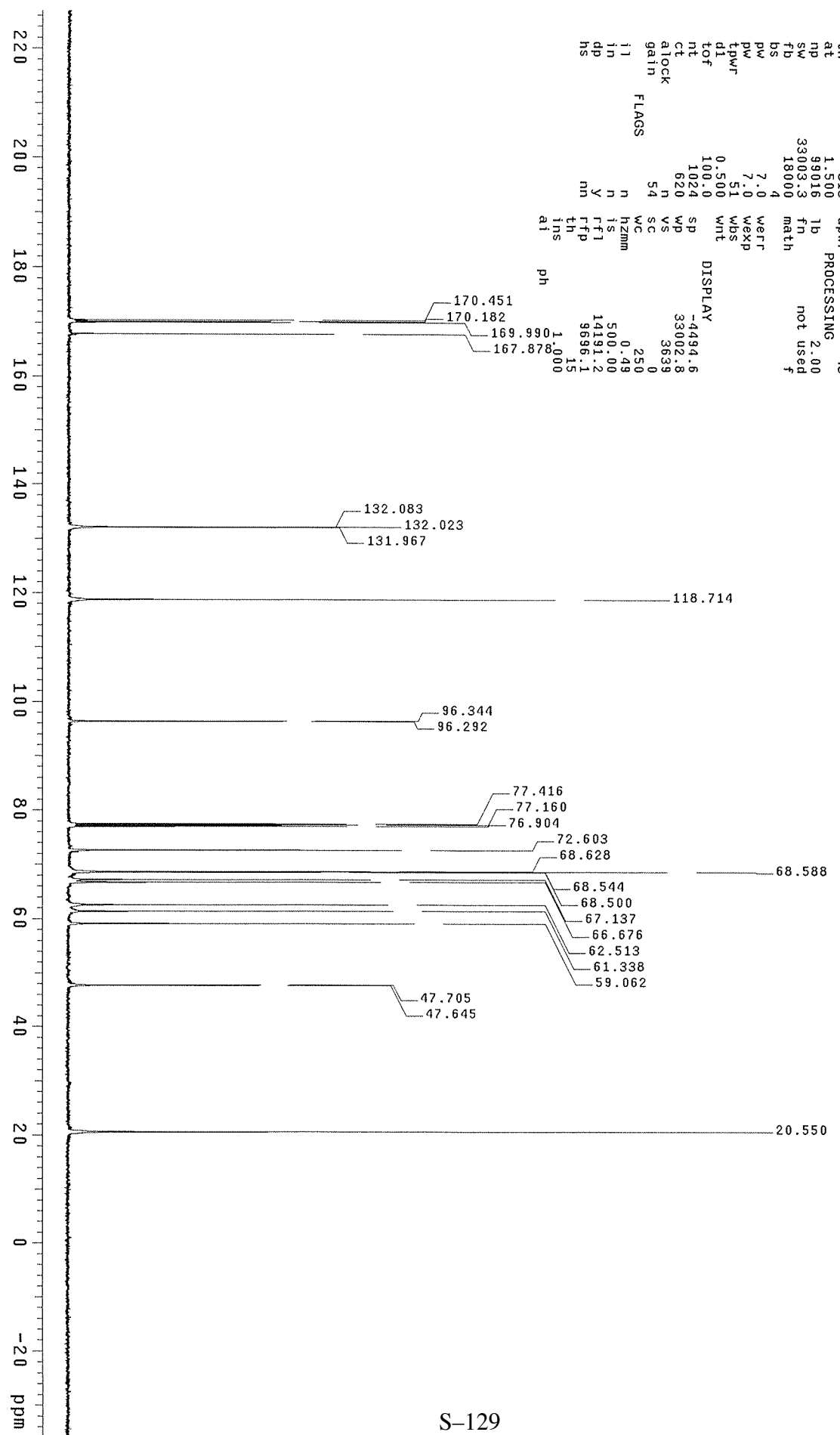
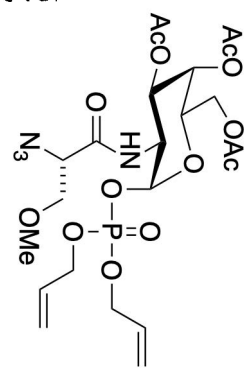
dp Y Ff1

hs mn Rffp

ai th ins

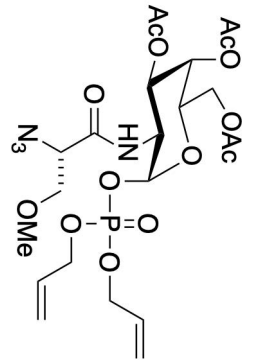
ph

ai



JC2011\_31P\_CD013

exp2 s2pul1

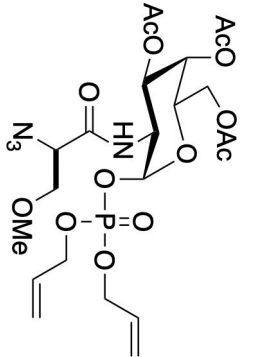


SAMPLE Jun 15 2017 DEC. & VT 499.751  
 solvent CDCl3 dn H1 43  
 file exp dpwr 0  
 ACQUISITION 202.297 dmf YYY 0  
 tn P31 dmm 11400  
 at 1.002 dmf W  
 np 160254 dseq  
 sw 80000.0 dres 1.0  
 fd 44000 homo n  
 bs 4  
 tpwr 51 dfrq2 DEC2 0  
 pw 6.6 dn2  
 dl 2.000 dpwr2 1  
 tof 0 dof2 0  
 nt 2048 dm2 n  
 ct 44 dmm2 c  
 alock n dmf2 9900  
 gain 30 dseq2  
 dres2 1.0  
 i1 n homo2  
 in n  
 dn y  
 dp n  
 hs n  
 DISPLAY nm  
 SD -45163.0 proc ft  
 WP 79999.4 fn not used  
 VS 24520 math  
 SC 0 werr  
 WC 250 wexp  
 hzmm 9.67 wbs  
 is 500.00 wnt  
 rffl 45163.6  
 rfh 0  
 th 17  
 ins 1.000  
 ai cdc ph

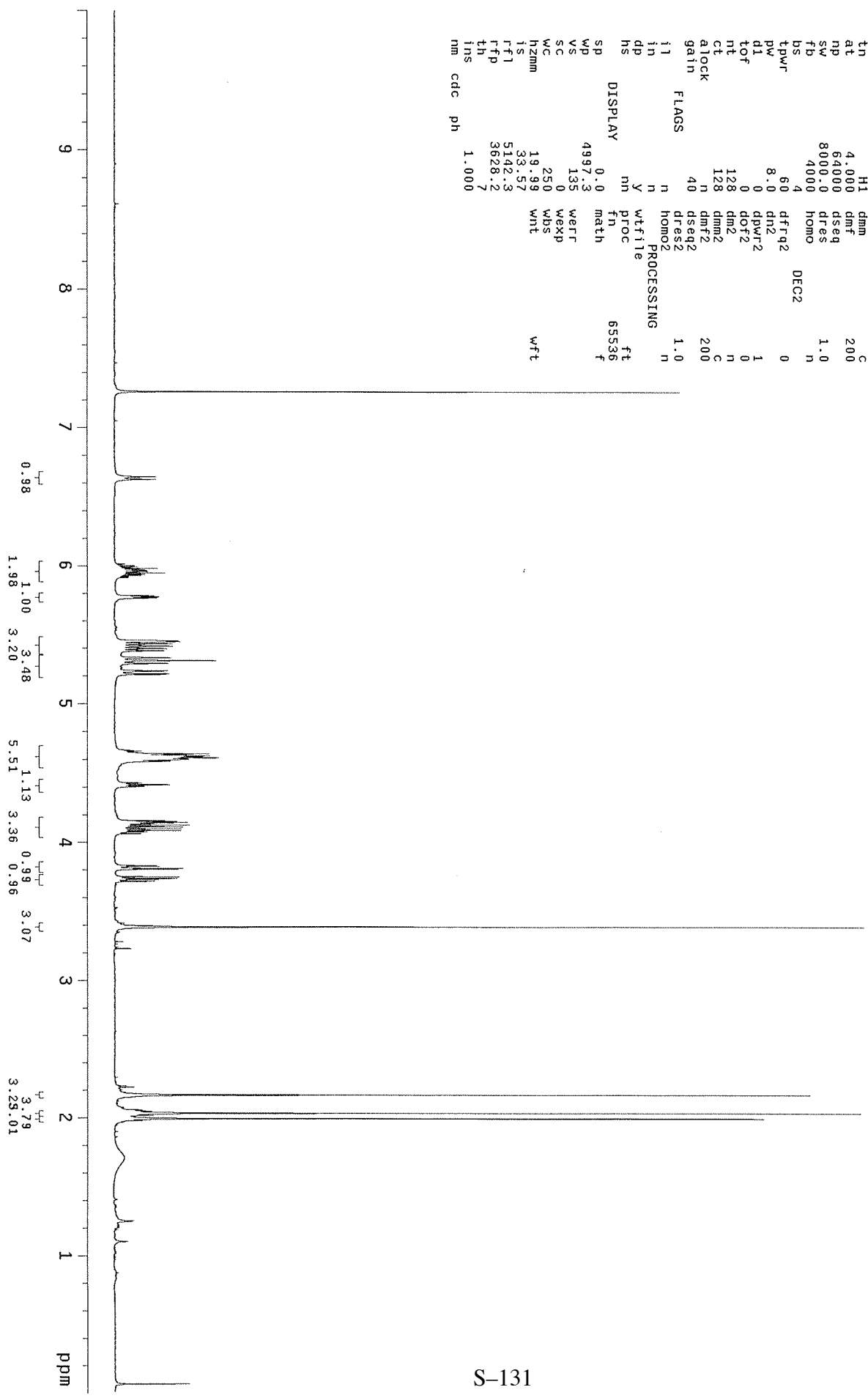


JC2013\_1H\_CDC13

exp2 s2pu1



SAMPLE DEC. & VT  
 date Jun 15 2017 499.751  
 solvent CDC13 dn H1  
 file ACQUISITION exp H1  
 ACQUISITION 30  
 dof 0  
 sfrq 499.751 dm mhn  
 tn H1 dmm C  
 at 4.000 dmf 200  
 mp 64000 dseq  
 sw 8000.0 dres 1.0  
 fd 4000 homo n  
 bs 4  
 tpwr 60 dfrq2 DEC2  
 pw 8.0 dn2 0  
 dl 0 dpwr2 1  
 tof 0 dof2 0  
 nt 128 dm2 n  
 ct 128 dmm2 C  
 atlock n dmf2 200  
 gain 40 dseq2  
 flags dres2  
 i1 n homoz 1.0  
 in n  
 dp y wftile  
 hs mn PROCESsing  
 DISPLAY ft  
 sp 0.0 fn 65536  
 wp 4997.3 math f  
 vs 135 werr  
 sc 0 wexp  
 wc 250 wbs  
 hzmm 19.99 wnt  
 is 33.57  
 rffl 5142.3  
 rfp 3628.2  
 th  
 ins 1.000  
 nm cdc ph



JC2013\_13C\_CDCl3

exp2 s2pu1

SAMPLE Jun 15 2017 DEC. & VT H1

solvent CDC13 -499.0

file exp dm

ACQUISITION 125.674 dm

sfrq C13 11400 w

in 1.500 dpwr 43

at 99016 1b PROCESSING 2.00

sw 33003.3 fn not used f

fb 18000 math

bs 4

pw 7.0 wefr

tpwr 7.0 wexp

dl 0.500 wbs

nt 100.0 wnt

ct 25000 sp

atock 17944 wp

gain 54 vs

FLAGS 54 SC

ii n WC

in n hzmm

dp y ts

hs nn rffl

ai ins th

ph 1.000

DISPLAY

-4476.0

33002.8

62348

0

250

5.03

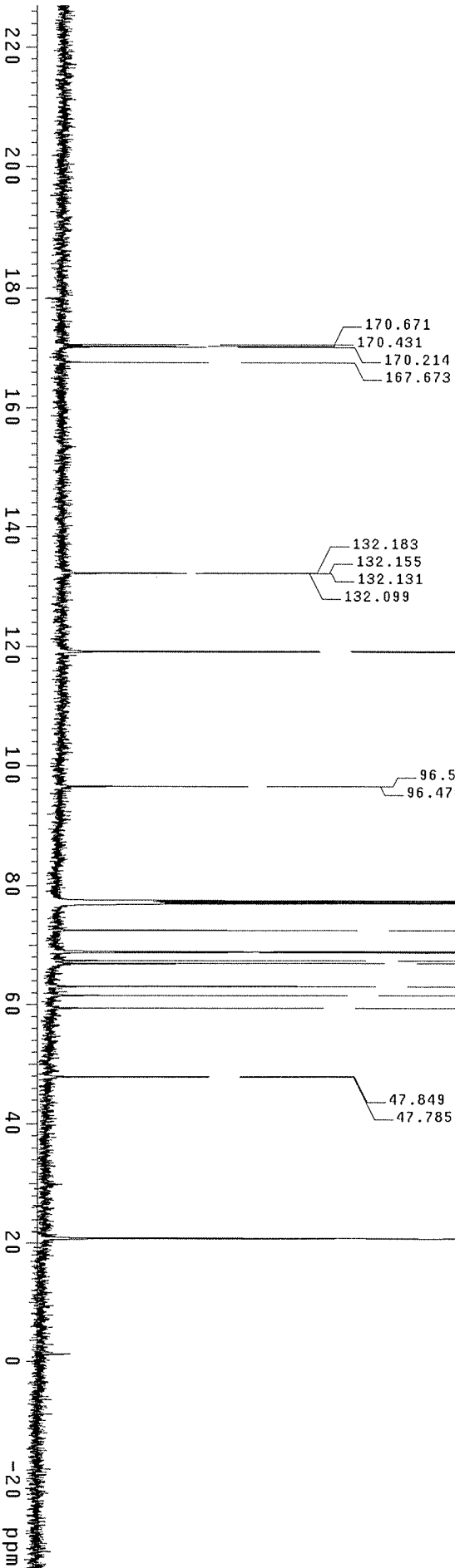
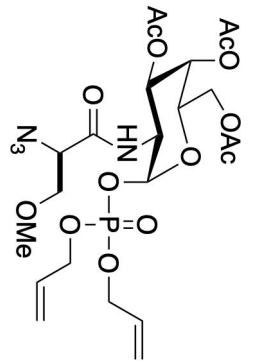
500.00

14172.5

9696.1

12

1.000



JC2013\_31P\_CD013

exp2 s2pul1

SAMPLE

date Jun 16 2017

solvent CDCl3

file exp

ACQUISITION

sfrq 202.297

in P31

at 1.002

mp 160254

sw 80000.0

fb 44000

bs 4

tpwr 51

pw 6.6

d1 2.000

tof 0

nt 128

ct 128

atlock n

gain 30

flags n

i1 n

in n

dp y

hs n

DISPLAY

sp -45160.5

wp 79999.4

vs 27298

sc 0

wc 250

hzmm 0.69

ts 500.00

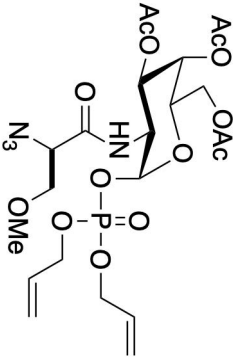
ffl 45161.1

ffp 0

th 17

ins 1.000

at cdc ph



-1.364



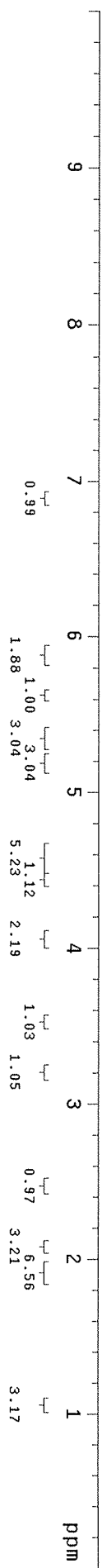
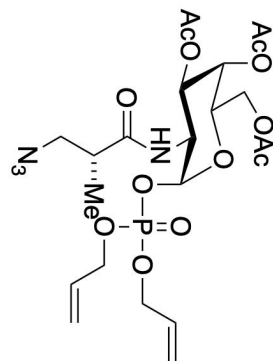
JC5152\_1H\_CDCl3

exp3 s2pu1

```

SAMPLE          DEC. & VT
date    Jun 24 2017    499.751
solvent  GDC13         dn
file    /export/home/~ dpwr
Junwchoi/JC5152_1H~ dof
          _GDC13.f1d   dm
ACQUISITION
sfrq    499.751      dmf
tn      H1          dseq
at      4.000       dres
np      64000      homo
sw      8000.0     DECC2
fb      40000     dffq2
ds      4          dh2
tpwr    60         dpwr2
pw      8.0       dof2
dl      0         dm2
tof     0         dmm2
nt      16        dmf2
ct      16        dseq2
atock   n         dres2
gain    40        homoz
          FLAGS
i1       n        wfttle
in       n        n
dp       y        fn
hs       n        math
          DISPLAY
sp      0.0       werr
wd      4997.3   wexp
vs      151      wds
sc      0        wnt
wc      250
hzmm    19.99
is      33.57
rfi     5141.8
rfp     3628.2
th      7
ins     1.000
nm      cdc
ph

```



JC5152\_13C\_CDCl3

exp2 s2pul1

DEC. & VT

dn dn

date Jun 24 2017

solvent CDC13

file exp

ACQUISITION

sfrq 125.674

tn 1.500

at 99016

np 33003.3

fb 18000

bs 4

pw 7.0

tpwr 7.0

d1 31

tof 0.500

nt 102.4

ct 132

atlock 54

gain 54

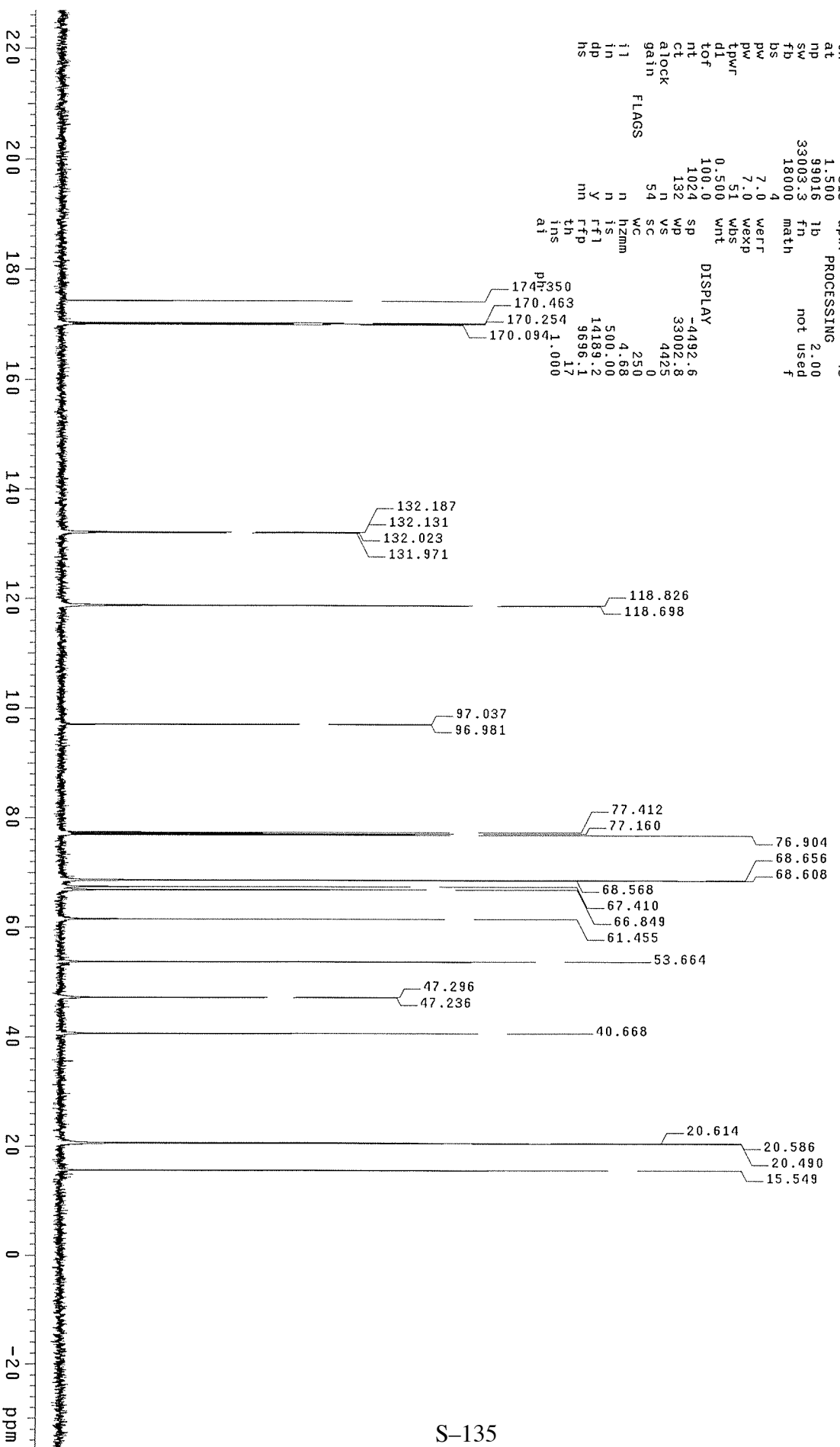
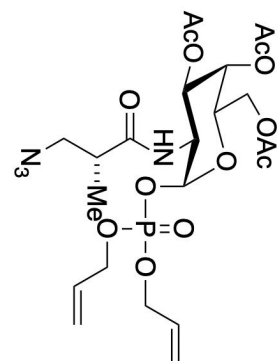
flags

i1 n

in n

dp y

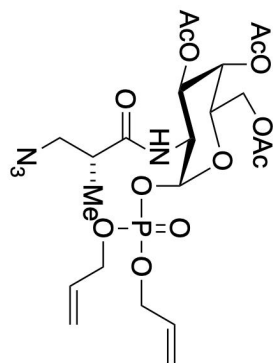
hs mn



JC5152\_31P\_CDC13

exp2 s2pu1

date	Jun 24 2017	DEC. & VT	499.751
solvent	CDC13	dn	H1
file	exp	dpwr	43
ACQUISITION	202.297	dm	0
sfrq	202.297	dm	yyy
tn	R31	dmm	w
at	1.002	dmf	11400
np	160254	dseq	
sw	80000.0	dres	1.0
fb	44000	homo	n
bs	4	DECE2	
tpwr	51	dfreq2	0
pv	6.6	dn2	
d1	2.000	dpwr2	1
tof	0	dot2	0
nt	1024	dm2	n
ct	16	dmm2	c
atock	n	dmf2	9900
gain	30	dseq2	
FLAGS		dres2	1.0
i1	n	homo2	n
in	n	PROCESSING	
dp	y	lb	2.00
hs	nm	wfille	
DISPLAY		proc	ft
sp	-45137.3	fn	not used
wp	79999.4	math	f
vs	885	werf	
SC	0	wexp	
WC	250	wbs	
h2mm	5.004	wnt	
is	500.00		
ffl	45137.9		
ffp	0		
th	17		
ins	1.000		
ai	cdc	ph	



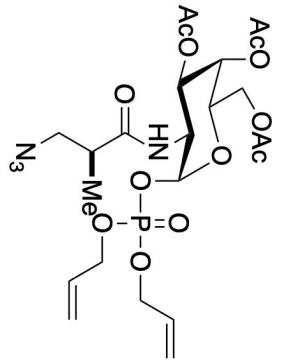
-2.501



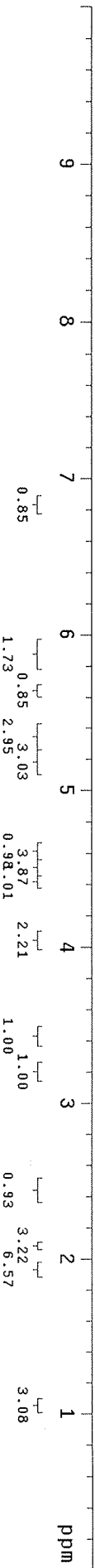


J05134\_1H\_CDCl3

exp2 s2pu1



SAMPLE DEC. & VT  
 date Jun 13 2017 499.751  
 solvent CDC13 dn H1  
 file exp CDC13 dpwr H1  
 ACQUISITION 30  
 sfrq 499.751 dm mm  
 tn H1 dmm c  
 at 4.000 dmf 200  
 np 64000 dseq  
 sw 800.0 dres 1.0  
 fd 4000 homo n  
 bs 4  
 tpwr 60 dfq2 DEC2  
 pw 8.0 dm2 0  
 dl 0 dpwr2 1  
 tof 0 dof2 0  
 nt 16 dm2 n  
 ct 16 dmf2 c  
 atock n  
 gain 40 dseq2 200  
 flags n  
 i1 n homoz 1.0  
 in n  
 dp y wtfile PROCESSING  
 hs nm fn  
 DISPLAY 0.0 math f  
 SP 4997.3 weft  
 WD 151 weft  
 VS 250 weft  
 SC 0 wexp  
 WC 250 wds  
 hzmm 19.99 wnt  
 IS 33.57  
 FFI 5141.8  
 FFP 3628.2  
 TH 1.000  
 INS  
 NM CDC PH



JCS134\_13C\_CDC13

exp2 s2pu1

SAMPLE DEC. & VT

date Jun 13 2017 dn H1

solvent CDC13 dof -499.0

file exp dm yy

ACQUISITION 125.674 dmf W

sfrq 125.674 dmf 11400

tn C13 dpwr 43

at 1.500 1b PROCESSING 2.00

np 99016 fn not used f

sw 33003.3 fn

fb 18000 math

bs 4

pw 7.0 weff

pw 7.0 wexp

tpwr 51 wds

d1 0.500 wnt

nt 100.0 sp

ct 268 wp

atock n vs

gain 54 SC

FLAGS WC

i1 n hzmm

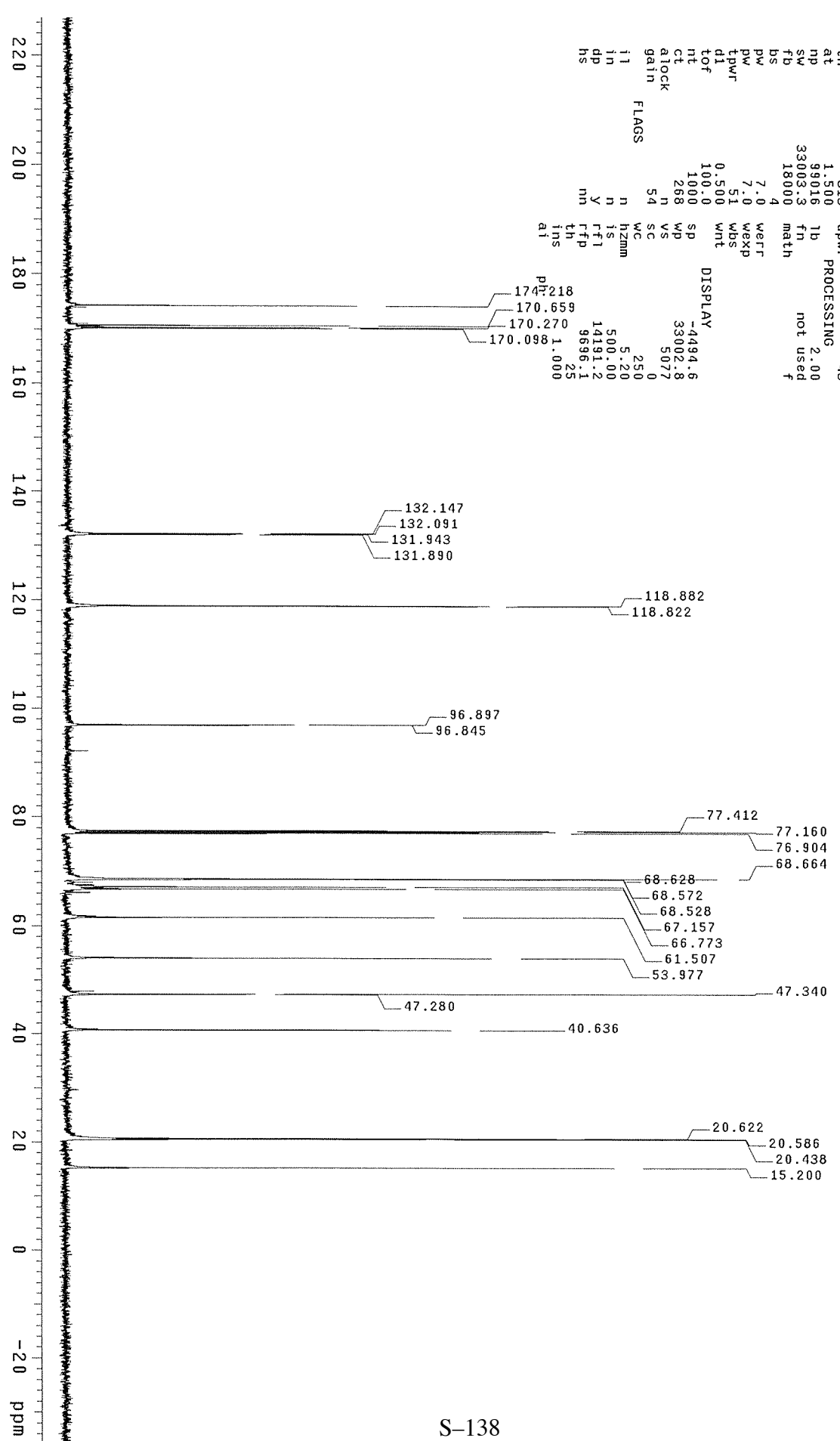
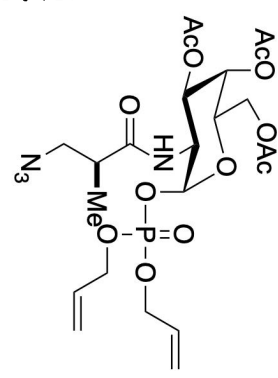
in n is

dp y rfi

hs n rff

th ins

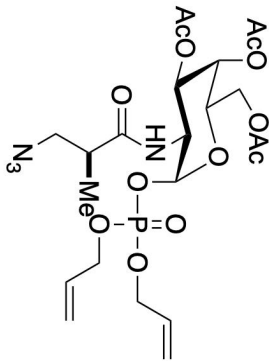
ai



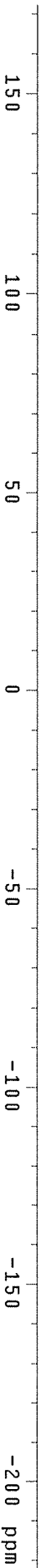
DISPLAY -4494.6  
 33002.8  
 5077.0  
 250  
 5.20  
 500.00  
 14191.2  
 9696.1  
 25  
 1.000

JC5134\_31P\_CDCl3

exp2 s2pu1



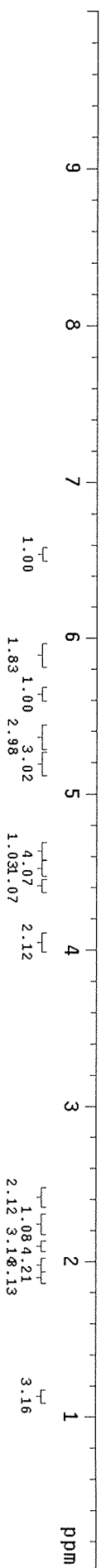
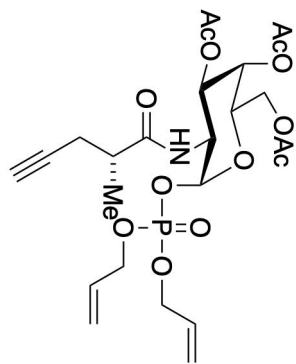
SAMPLE Jun 13 2017 DEC. & VT  
 solvent CDC13 dn 499.751  
 file exp dpwr H1  
 ACQUISITION exp dof 43  
 sfrq 202.287 dm YYY 0  
 tn P31 dmm W  
 at 1.002 dmf 11400  
 np 160254 dseq  
 SW 80000.0 dres 1.0  
 fb 44000 homo n  
 bs 4  
 tpwr 51 dffq2 DEC2 0  
 pw 6.6 dn2  
 dl 2.000 dpwr2 1  
 tof 0 dof2 n  
 nt 64 dm2 n  
 ct 24 dmm2 c  
 atlock n dmf2 9900  
 gain 30 dseq2  
 dres2  
 homoz 1.0  
 n  
 PROCESSING  
 2.00  
 DISPLAY nm  
 SP -45142.2  
 WD 79999.4 math not used  
 VS 889  
 SC 0 WERT  
 WC 250 WEXP  
 hzmm 8.83 WBS  
 IS 500.00 Wnt  
 rfl 45142.8  
 rfp 0  
 th 17  
 ins 1.000  
 ai cdc ph



JC3017\_1H\_CDC13

exp2 s2pu1

date	Jun 10 2017	DEC. & VT	499.751
solvent	CDC13	dn	H1
file	ACQUISITION	exp	30
sfreq	499.751	dm	0
tn	H1	dmm	mmn
at	4.000	dmf	C
np	64000	dseq	200
sw	8000.0	dres	1.0
fb	4000	homo	n
bs	4	homo2	n
tpwr	60	dfreq2	DEC2
pv	8.0	dn2	0
d1	0	dpwr2	1
tof	0	do2	0
nt	64	dm2	n
ct	64	dmm2	C
atock	n	dmf2	200
gain	40	dseq2	1.0
il	n	homo2	n
in	n	wtfile	PROCESSING
dp	Y	proc	ft
hs	nm	fn	65536
sp	DISPLAY	math	f
wd	0.0	werr	
vs	4997.3	wexp	
sc	151	wbs	
wc	250	wnt	wft
h2mm	19.99		
is	33.57		
ffl	5141.8		
ffp	3628.2		
th	7		
ins	1.000		
nm	cdc		
ph			



JC3017\_13C\_CDCl3

exp1 s2pu1

SAMPLE DEC. & VT

date Jun 10 2017 dn H1

solvent CDC13 dof -499.0

file exp dm YYY

ACQUISITION 125.674 dmm 11400

sfrq 125.674 dmf 43

tn 1.500 PROCESSING 2.00

at 9.9016 1b not used

np 33003.3 fn math

fb 18000 4

bs 7.0 wefr

pw 7.0 wexp

tpwr 51 wbs

d1 wnt

tof 0.500 DISPLAY

nt 100.0 sp -4990.6

ct 1000 wp 33002.8

atlock 576 vs 4596

gain 54 hzmm 0

il n 3.47

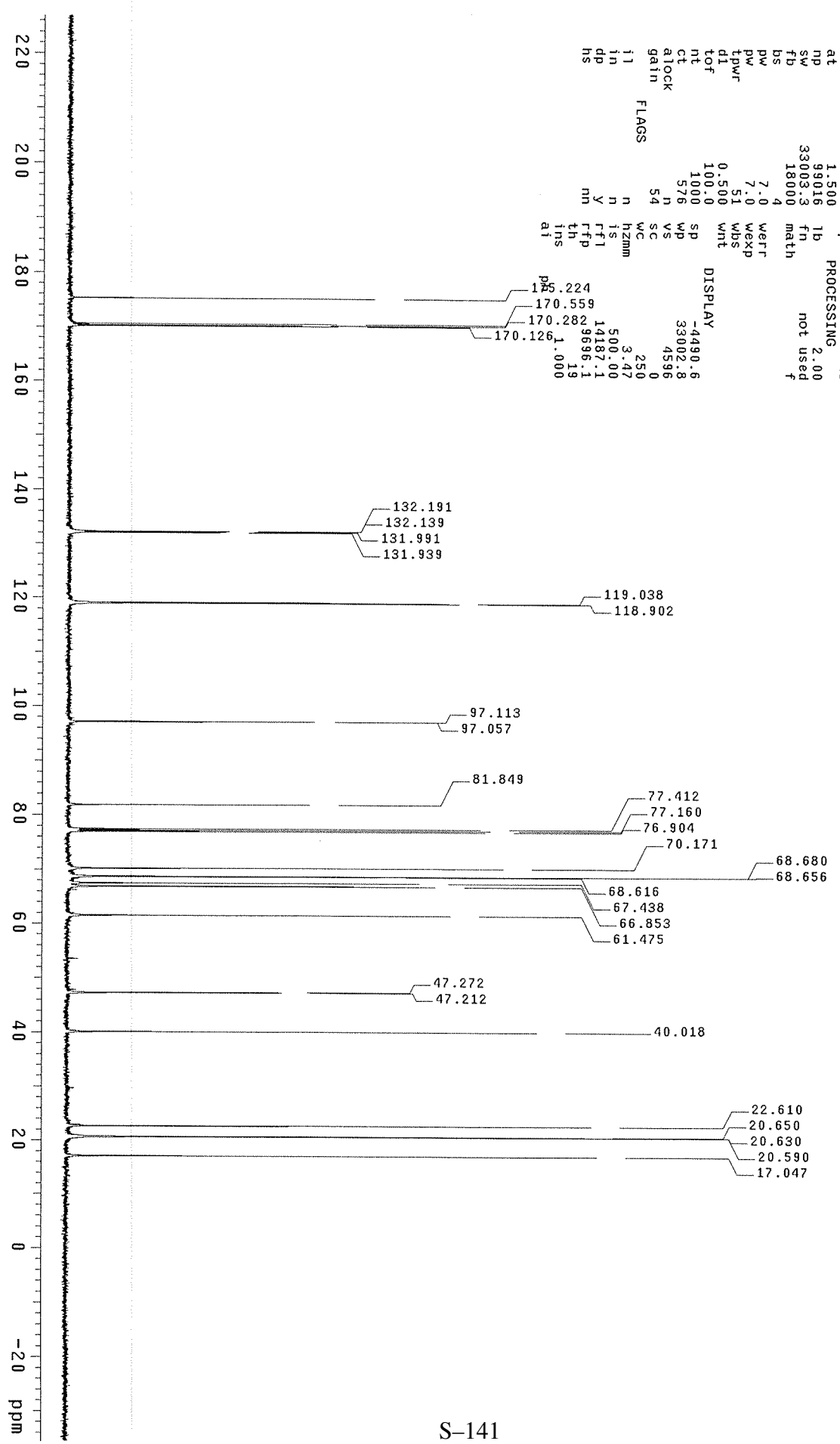
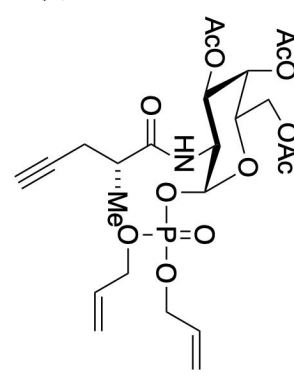
in n 500.00

dp y 14187.1

hs nm 9696.1

ins th 19

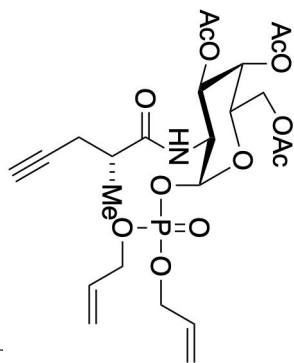
ai 1.000



JC3017\_31P\_CD013

expt1 s2pul1

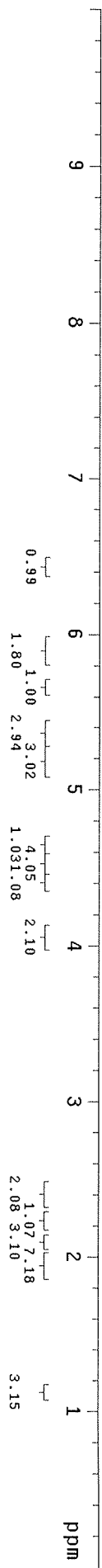
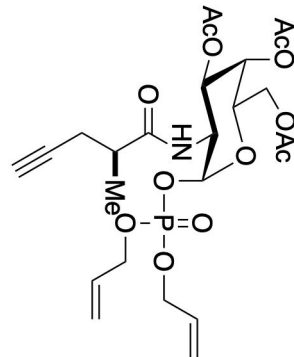
SAMPLE DEC. & VT  
 date Jun 10 2017 dfrq 499.751  
 solvent CDC13 dn H1  
 file exp dof 43  
 ACQUISITION exp dof 0  
 sfrq 202.297 dm YYY  
 tn F31 dmm W  
 at 1.002 dmf 11400  
 np 160254 dseq  
 sw 80000.0 dres 1.0  
 fb 44000 homo n  
 bs 4  
 tpwr 51 dfrq2 DEC2  
 pw 6.6 dn2 0  
 dl 2.000 dpwr2 1  
 tof 0 dof2 0  
 nt 32 dm2 n  
 ct 20 dmm2 n  
 atlock n dmf2 C  
 gain 30 dseq2 9900  
 FLAGS dres2 1.0  
 il n homo2 n  
 in n PROCESSING 2.00  
 dp y lb wfile  
 hs nm wfile  
 DISPLAY ft  
 sp -45144.0 fn not used  
 wp 79999.4 math f  
 vs 1093  
 sc 0 wefr  
 wc 250 wekp  
 hzmm 1.81 wds  
 IS 500.00 wnt  
 rffl 45144.7  
 rffp 0  
 lh 17  
 ins 1.000  
 ai cdc ph



JC3013\_1H\_CDCl3

exp2 s2pu1

SAMPLE DEC. & VT  
 date Jun 10 2017 499.751  
 solvent CDC13 H1  
 file exp dpwr 30  
 ACQUISITION dof 0  
 sfrq 499.751 dm mm  
 tn H1 dm C  
 at 4.000 dmf 200  
 np 64000 dseq  
 sw 8000.0 dres 1.0  
 fb 4000 homo n  
 bs 4 DEC2  
 tpwr 60 dfrq2 0  
 pv 8.0 dpwr2 1  
 d1 0 dof2 0  
 tof 0 dm2 n  
 nt 16 dmm2 C  
 ct 16 dmf2 200  
 atlock n dseq2  
 gain 40 dres2 1.0  
 flags n  
 i1 n homo2 n  
 in n  
 dn Y wfile  
 hs nm math ft  
 DISPLAY 0.0 fn 65536  
 sp 4997.3 weft  
 wp 151 weXP  
 vs 0 WBS  
 SC 250 Wnt  
 WC h2mm  
 hzmm 19.99  
 IS 33.57  
 rfl 5141.6  
 rfp 3628.2  
 th 7  
 ins 1.000  
 nm cdc ph



JC3013\_13C\_CDCl3

exp2 s2pu1

SAMPLE DEC. & VT

date Jun 10 2017 dn H1

solvent CDC13 dof -499.0

file ACQUISITION exp dm yyy

sfrq 125.674 dmf 11400

tn C13 dpwr 43

at 1.500 1b 2.00

np 99016 fn not used

sw 33003.3 math

fb 18000 4

bs 7.0 weff

pw 7.0 wexp

tpwr 0.500 wds

d1 100.0 wnt

tof 1000.0

nt 176 sp -4489.6

ct 176 wp 33002.8

atlock gain 54 SC 5743

gain 54 SC 5743

flags 54 SC 5743

i 1 WC 250

in n hzmm 2.95

dp n IS 500.00

hs y rffl 4186.1

ns th rffp 9696.1

ai ins 23

ai ins 23

ai ins 23

ai ins 23

ai ins 23

ai ins 23

ai ins 23

ai ins 23

ai ins 23

ai ins 23

ai ins 23

ai ins 23

ai ins 23

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ai ins 23

ai ins 23

ai ins 23

ai ins 23

ai ins 23

ai ins 23

ai ins 23

ai ins 23

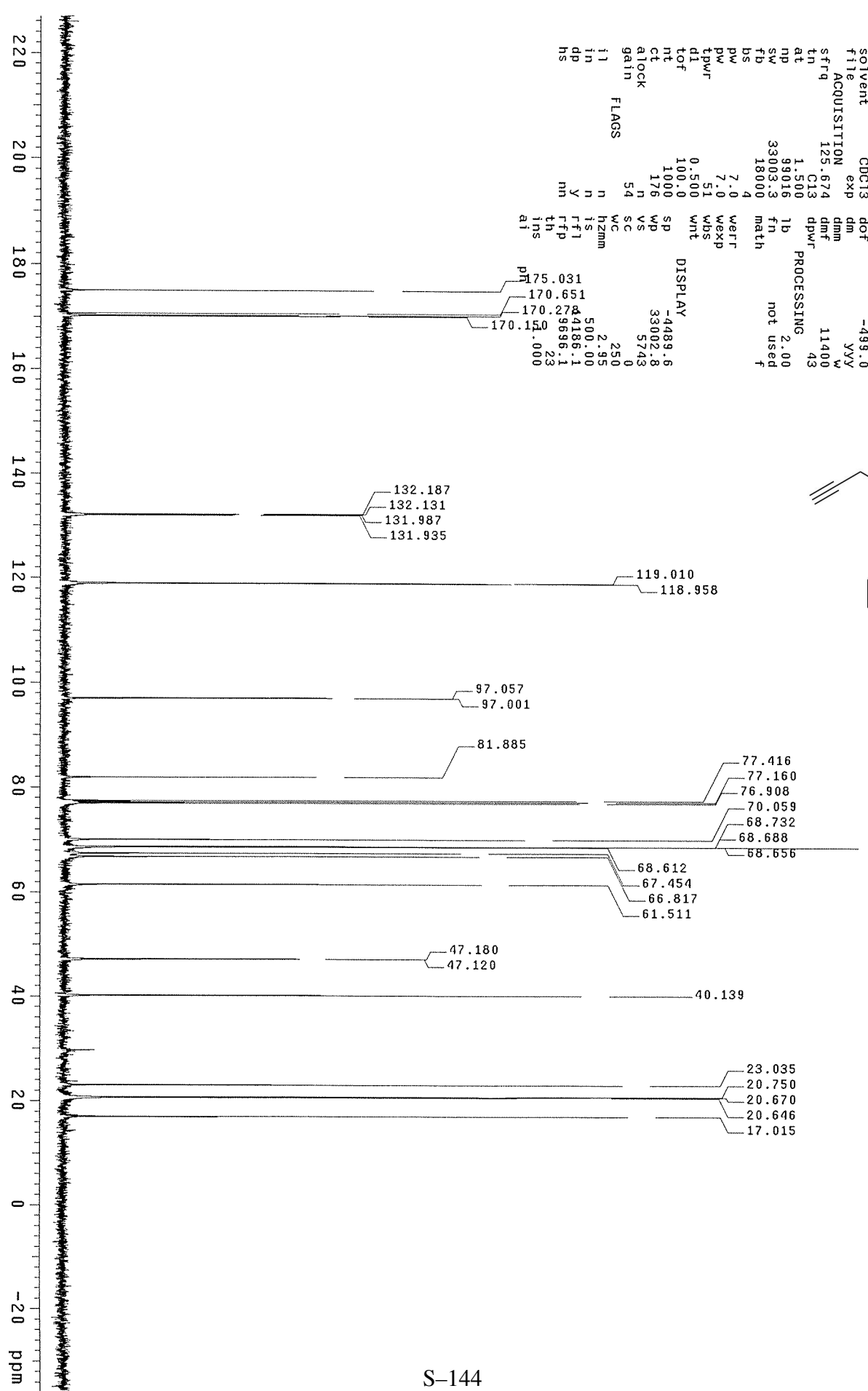
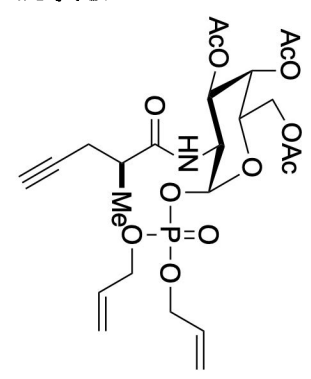
ai ins 23

ai ins 23

ai ins 23

ai ins 23

ai ins 23

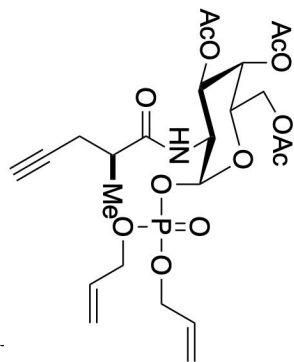




JC3013\_31P\_CDC13

exp3 s2pu1

SAMPLE DEC. & VT  
 date Jun 10 2017 dfrq 499.751  
 solvent CDC13 dn H1  
 file exp dpwr 43  
 ACQUISITION exp dof 0  
 sfrq 202.297 dm YYY  
 tn P31 dmm W  
 at 1.002 dmf 11400  
 np 160254 dseq  
 sw 80000.0 dres 1.0  
 fb 44000 homo n  
 bs 4 dfrq2 DEC2  
 tpwr 51 dn2 0  
 pw 6.6 dpwr2  
 d1 2.000 dpwr2 1  
 tof 0 dof2 n  
 nt 64 dm2 n  
 ct 28 dmm2 C  
 atlock n dmf2 9900  
 gain 30 dseq2  
 dres2  
 flags 1.0  
 i1 n homo2 n  
 in n PROCESSING  
 dp y lb wfile 2.00  
 hs nm wfile  
 DISPLAY ft  
 sp -45145.9 fn not used  
 wd 79999.4 math f  
 vs 1265  
 SC 0 werr  
 WC 250 wexp  
 hzmm 0.32 wbs  
 is 500.00 wnt  
 ffl 45146.5  
 ffp 0  
 th 17  
 ins 1.000  
 ai cdc ph



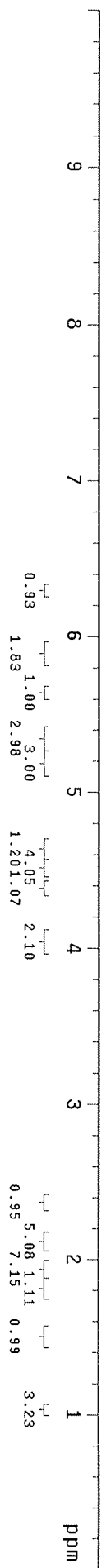
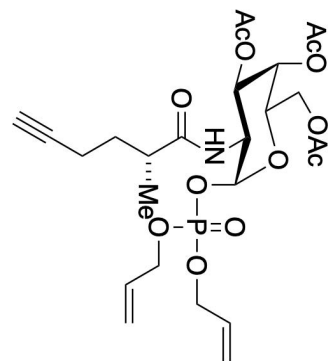
-2.033



JC3063\_1H\_GDC13

exp2 s2pu1

SAMPLE	DEC. & VT	499.751
date	Jun 13 2017	
solvent	GDC13	
file	exp	
ACQUISITION	exp	
sfrq	499.751	mm
ln	H1	dm
at	4.000	dmf
np	64000	dseq
sw	8000.0	dres
fb	4000	homo
bs	4	
tpwr	60	dfrq2
pw	8.0	dn2
di	0	dpwr2
tof	0	doF2
nt	16	dm2
ct	16	dmm2
atock	n	dmf2
gain	40	dseq2
FLAGS		dres2
il	n	homo2
in	n	PROCESsing
dp	y	wfille
hs	nm	proc
DISPLAY	0.0	fn
sp	4997.3	math
wd	151	werr
vs	0	wexp
sc	250	wbs
wc	19.99	wnt
h2mm	33.57	wft
is	5141.8	
rfl	3628.2	
rffp	7	
th	1.000	
ins		
nm		
cdc		
ph		



JC3063\_13C\_CDCl3

exp2 s2pu1

SAMPLE Jun 13 2017 DEC. & VT H1

solvent CDCl3 -499.0

file exp dm

ACQUISITION 125.674 dmm

sfrq 125.674 dmf 11400

tn C13 dpwr 43

at 1.500 1b

mp 99016 fn

sw 33003.3 fn not used

fd 18000 math

bs 4

pw 7.0 wert

pw 7.0 wexp

tpwr 51 wbs

d1 0.500 wnt

tof 100.0

nt 1000

ct 164

atlock n

gain 54

FLAGS

11 n hzmm

11 n is

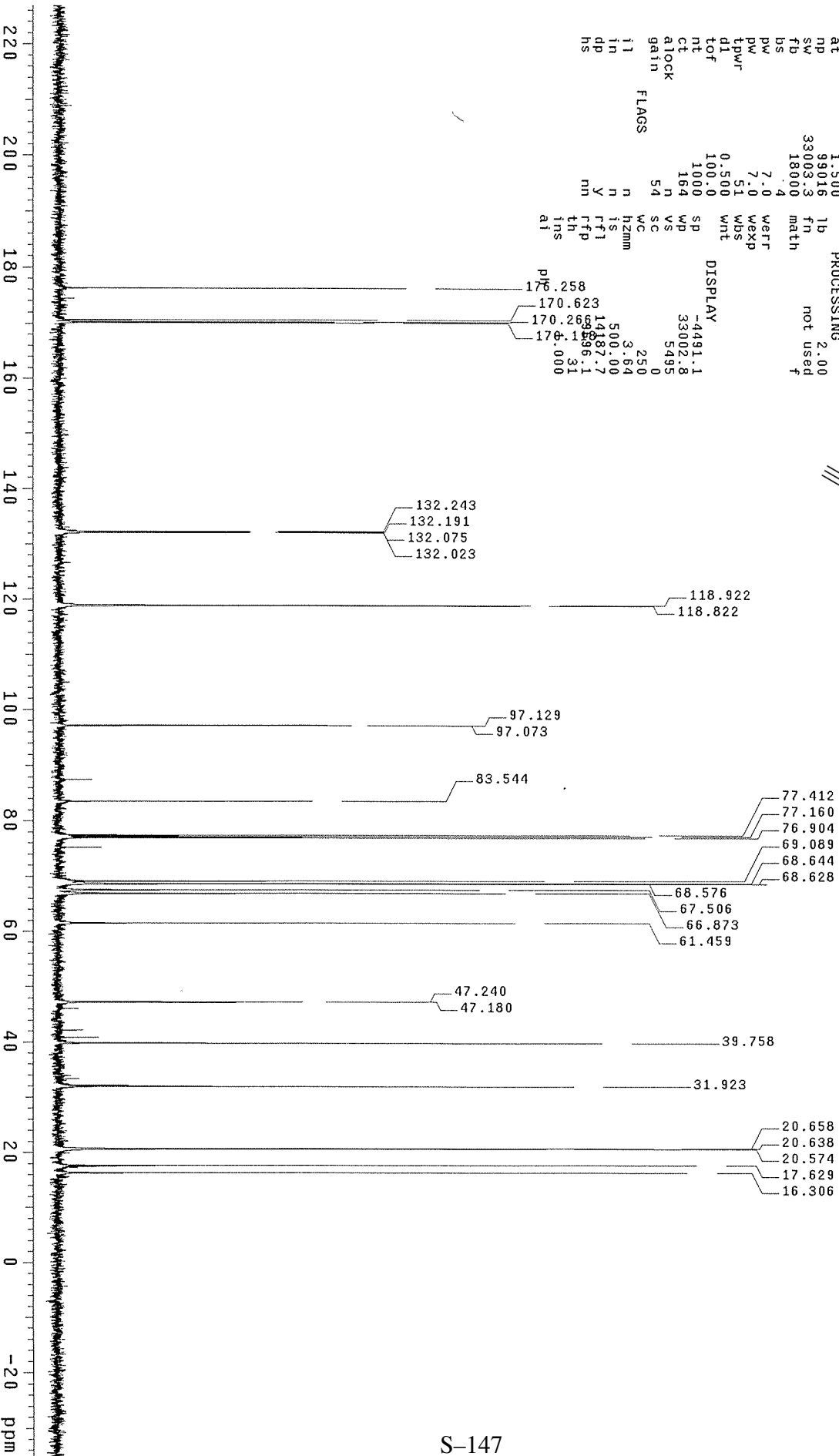
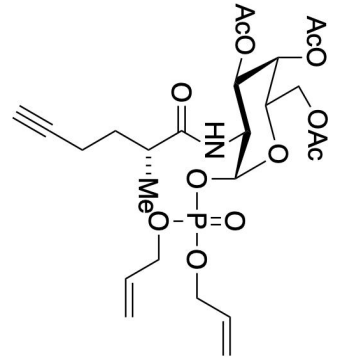
11 n rfi

11 n rfp

11 n th

11 n ins

11 n al



DISPLAY

-4491.1

33002.8

5495

250

3.64

500.00

14187.7

14187.7

14187.7

14187.7

14187.7

14187.7

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14187.7

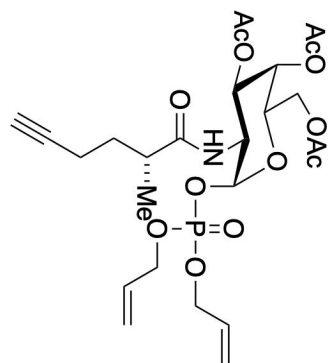
14187.7

14187.7

JC3063\_31P\_CDC13

exp2 s2pul1

SAMPLE	date	Jun 13 2017	DEC. & VT	499.751
solvent	file	CDC13	exp	H1
ACQUISITION	sfrq	202.297	dm	43
tn	at	1.002	dmf	0
np	sw	160254	dseq	yyy
sv	fb	80000.0	dres	w
bs	tpwr	44000	homo	11400
pw	di	51	dfreq2	0
tof	nt	6.6	dn2	1
ct	atlock	2.000	dpwr2	1
gain	flags	0	doF2	0
il	in	64	dm2	n
dp	hs	20	dmm2	n
hs	DISPLAY	n	dmf2	C
sp	WD	30	dseq2	9900
wp	VS	n	homo2	1.0
vs	SC	n	PROCESsing	n
WC	WC	0	1b	2.00
h2mm	h2mm	250	wfille	ft
IS	IS	5.89	fn	not used
rfl	rfl	500.00	math	f
rfl	rfl	45144.7		
th	th	0		
ins	ins	88		
al	al	1.000		



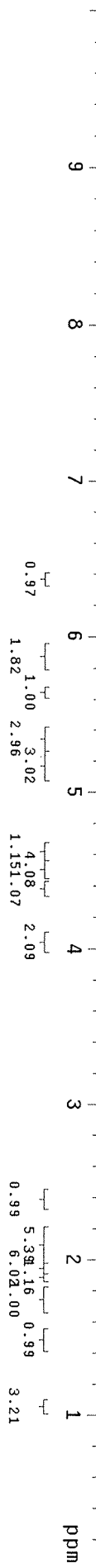
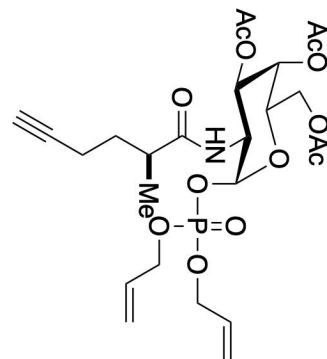
-1.946



JC3081\_1H\_CDCl3

exp2 s2pu1

date	Jun 10 2017	DEC. & VT	499.751
solvent	CDCl3	dn	H1
file	CDCl3	dpwr	30
ACQUISITION	exp	def	0
sfrq	499.751	dm	mm
tn	H1	dmm	C
at	4.000	dsmf	200
np	64000	dseq	
sw	8000.0	dres	1.0
fb	40000	homo	n
bs	4	DECC2	
tpwr	60	dfrq2	0
pw	8.0	dn2	
d1	0	dpwr2	1
tof	0	do2	n
nt	32	dmm2	n
ct	32	dmf2	C
atock	n	dseq2	200
gain	40	dres2	1.0
il	n	homo2	n
in	n	PROCESsing	
dp	y	wf file	ft
hs	nm	proc	fn
DISPLAY	0.0	math	f
SP	4997.3	weft	65536
VS	151	wexp	
SC	250	wbs	
WC	19.99	wnt	wft
h2mm	33.57		
ts	5141.8		
rfi	3628.2		
th	7		
ins	1.000		
nm	cdc	ph	



JC3081\_13C\_CDC13

exp2 s2pul1

DEC. & VT

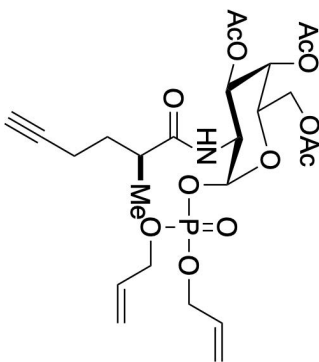
H1

-499.0

YYY

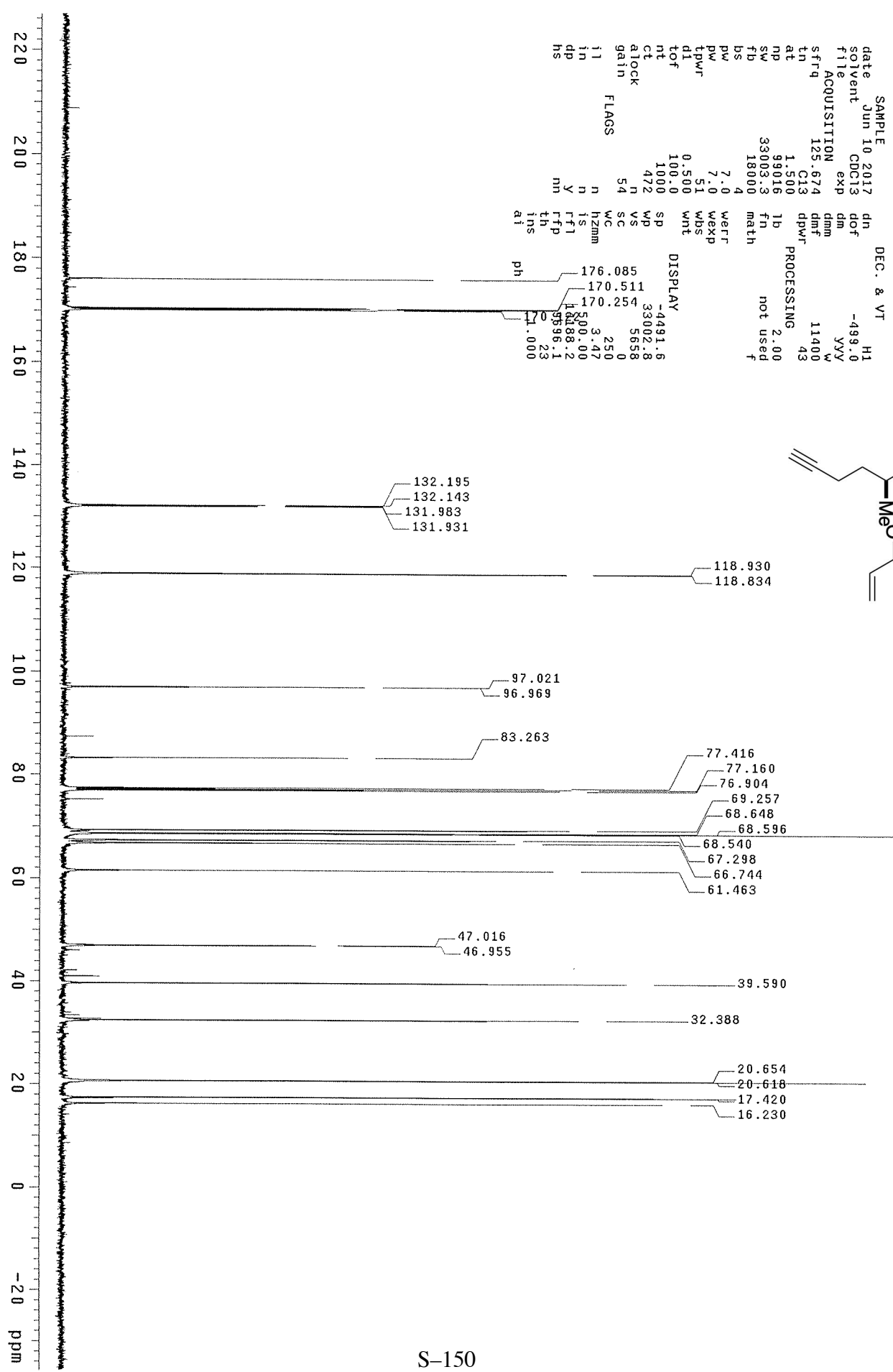
W

43



SAMPLE Jun 10 2017 dn  
 solvent CDC13 dof  
 file exp dm  
 ACQUISITION 125.674 dmm  
 sfrq 125.674 dmf  
 tn 1.500 dpwr  
 at 99016 lb  
 np 33003.3 fn  
 sw 18000 math  
 fb 4 math  
 bs 7.0 weff  
 pw 7.0 wexp  
 dw 51 wds  
 tpwr 0.500 wnt  
 dl 100.0  
 tof 1000 sp  
 nt 472 wp  
 ct 1000 vs  
 atlock 54 sc  
 gain 54 wc  
 flags n hzmm  
 i1 n  
 in n  
 dp y  
 pp rfl  
 th rfp  
 ins th  
 ai ai

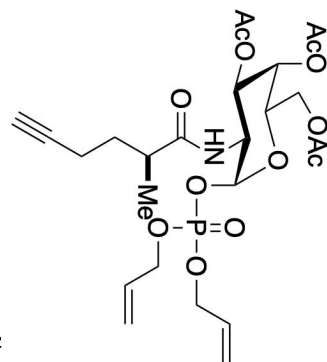
DISPLAY  
 -4991.6  
 33002.8  
 5658  
 0  
 250  
 3.47  
 500.00  
 1.8188.2  
 15996.1  
 23  
 1.000



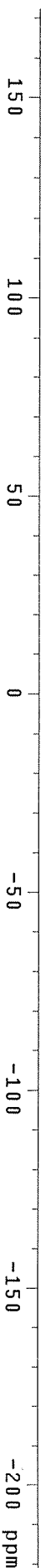
JC3081\_31P\_CDC13

exp2 s2pul1

SAMPLE date Jun 10 2017 DEC. & VT 499.751  
 solvent CDC13 dn H1 43  
 file ACQUISITION exp dpwr 0  
 ACQUISITION 202.297 dm dof 0  
 sfrq 202.297 dm yyy W  
 tn 1.002 dmf 11400  
 at 160254 dseq  
 np 80000.0 dres 1.0  
 sw 44000 homo n  
 fb 44000  
 bs 4  
 tpwr 51 dfrq2 DEC2 0  
 pw 6.6 dp2  
 d1 2.000 dpwr2 1  
 tof 0 dof2 0  
 nt 64 dm2 n  
 ct 16 dmm2 C  
 atock n dmf2 9900  
 gain 30 dseq2  
 dres2 1.0  
 i1 n homo2 n  
 in n  
 dp Y lb PROCESSING 2.00  
 hs Y wfile  
 DISPLAY -45142.2 fn ft  
 WP 79999.4 math not used f  
 VS 822  
 SC 0 weft  
 WC 250 Mexp  
 hzmm 5.04 WBS  
 IS 500.00 Wnt  
 ffl 45142.8  
 fth 0  
 lns 17  
 ai cdc ph 1.000

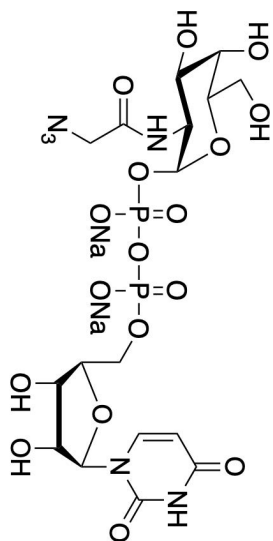


-2.082

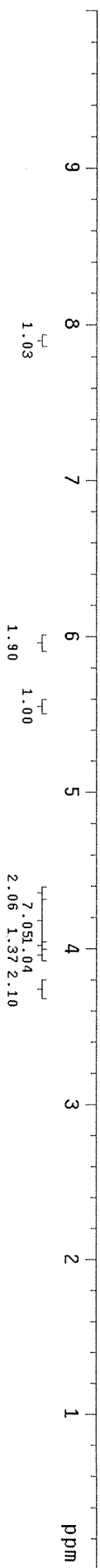


UDP-GalNAz\_1H\_D20  
exp2 s2pu1

SAMPLE	DEC. & VT	499.752
date Jul 22 2017	dfreq	499.752
solvent D2O	dn	H1
file	dpwr	30
ACQUISITION	dof	0
sfrq 499.752	dm	mm
tn H1	dmm	C
at 4.000	dmf	200
np 64000	dseq	
sw 8000.0	dres	1.0
fb 4000	homo	n
bs 4	DEFC2	0
tpwr 60	dfreq2	
pw 8.0	dn2	
d1 0	dpwr2	1
tof 0	dof2	n
nt 32	dmm2	n
ct 32	dmf2	C
atock n	dseq2	200
gain 40	dres2	1.0
FLAGS	homo2	n
il n	PROCESSING	
in n	wfitle	ft
dp y	nm	65536
hs nm	fn	f
DISPLAY	math	
sp -0.2	weff	
wd 4997.3	wexp	
vs 785	wds	
SC 0	wnt	wft
WC 250		
h2mm 19.99		
ts 4352.12		
rfl 3836.2		
rffp 2393.8		
th 7		
ins 1.000		
nm cdc ph		



UDP-GalNAz 2

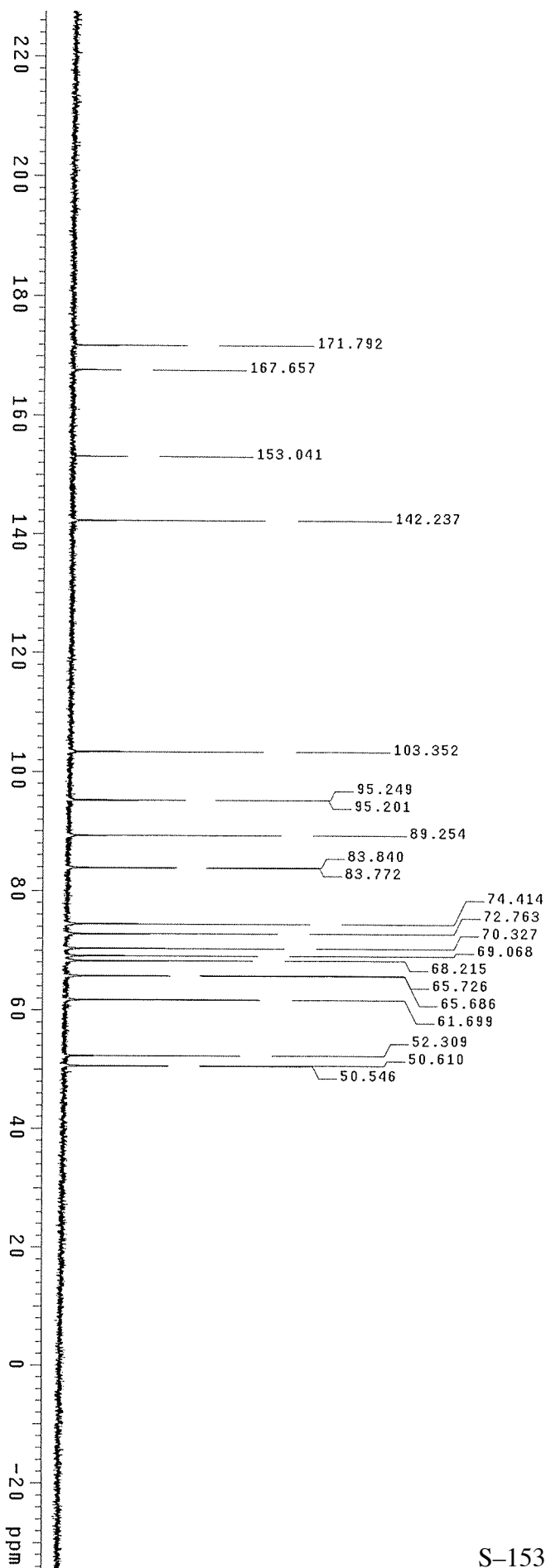
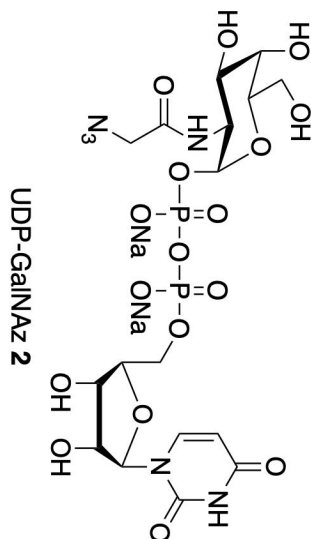




JC2119\_13C\_D20

exp3 s2pu1

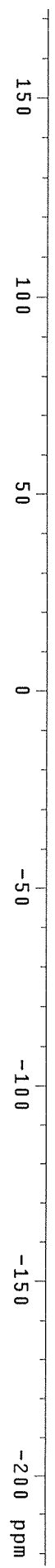
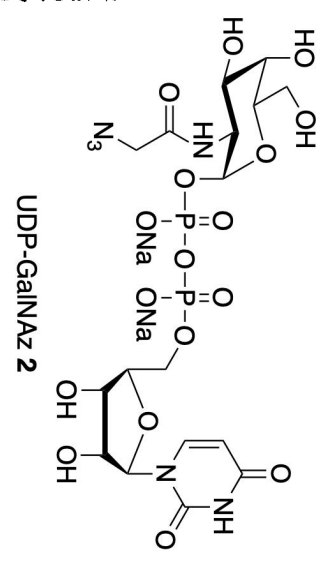
SAMPLE		DEC. & VT	
date	Jul 24 2017	dn	H1
solvent	D2O	do	-499.0
file	exp	dm	yyy
ACQUISITION	125.674	dmm	w
sfrq	125.674	dmf	11400
tn	1.500	dpwr	43
at	99016	PROCESSING	2.00
np	33003.3	fn	not used
sw	18000	math	f
fb	7.0	weff	
bs	7.0	wexp	
pw	51	wds	
tpwr	1.000	wnt	
dl	100.0	DISPLAY	
tof	20000	sp	-4411.1
nt	15668	wp	33002.8
ct	54	vs	30529
atock	n	sc	0
gain	54	wc	250
FLAGS		h2mm	6.24
il	n	ts	500.00
in	n	rs	10984.8
dp	y	rfl	6573.3
hs	nm	th	6
		ins	1.000
		ai	ph



UDP-Ga1NAz\_Na-form\_1H\_D2O

exp1 s2pu1

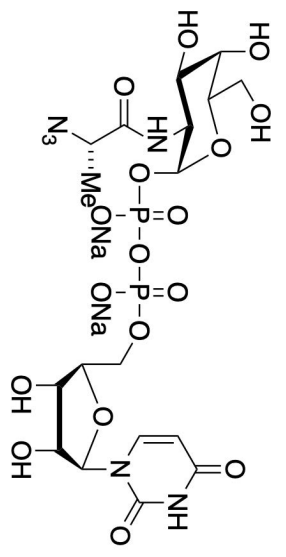
```
SAMPLE      DEC. & VT
date      Jul 26 2017  dfrq      499.752
solvent   D2O          dn        H1
file      ACQUISITION exp      dpwr      43
          202.297      dm        0
sfrq      202.297      dm        0
tn        P31         dmm       11400
at        1.002       dmf       11400
np        160254      dseq      1.0
sw        80000.0     dres      n
fb        44000      homo      n
bs        4          dfrq2     0
tpwr      51         dn2       1
pv        6.6        dpwr2     1
d11       2.000      dof2      n
tof       0         dmm2     n
nt       128        dmf2     C
ct       128        dseq2    9900
atlock   n          dres2    1.0
gain     30         dn2      n
          flags      homo2    n
i1       n          wtfile   2.00
in       n          proc     ft
dp       y          math     not used
hs       nm         wbs      f
          DISPLAY
sp      -45131.2
wp      79999.4
vs      21468
sc      0
wc      250
h2mm   2.81
ls      500.00
rfl    45131.8
rflp   0
th      17
ins     1.000
ai     cdc ph
```



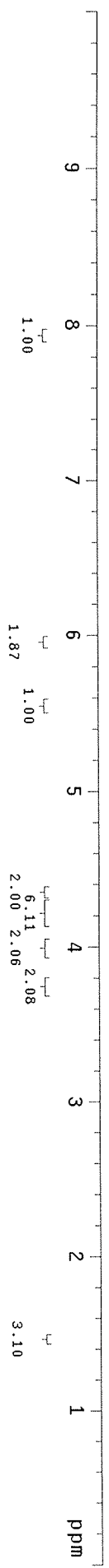
-10.520  
-10.623  
-12.159  
-12.261

JG1125\_1H\_d20  
 exp2 s2pu1

SAMPLE date Jul 22 2017 DEC. & VT 499.752  
 solvent D2O dn H1  
 title ACQUISITION exp dpuw 30  
 sfrmq 499.752 dm dof 0  
 tn H1 dmm nnn  
 at 4.000 dmf C  
 mp 64000 dseq 200  
 sw 8000.0 dres homo 1.0  
 fd 4000  
 bs 4  
 tpwr 60 dfrq2 DEC2  
 pw 8.0 dn2 0  
 dl 0 dpuw2 1  
 tof 0 dof2 0  
 nt 64 dmm2 n  
 ct 56 dmf2 C  
 atock n  
 gain 40 dseq2 200  
 flags 40 dres2 1.0  
 i1 n homo2  
 in n  
 dp Y wfile PROCESSING  
 hs n fn ft  
 DISPLAY -0.2 math fn 65536  
 sp 4997.3 werr  
 wd 277 wexp  
 vs 0 wbs  
 sc 250 wnt  
 wc 19.99  
 hzmm 33.57  
 is 3836.7  
 rfi 2393.8  
 th 1.000  
 ins cdc ph



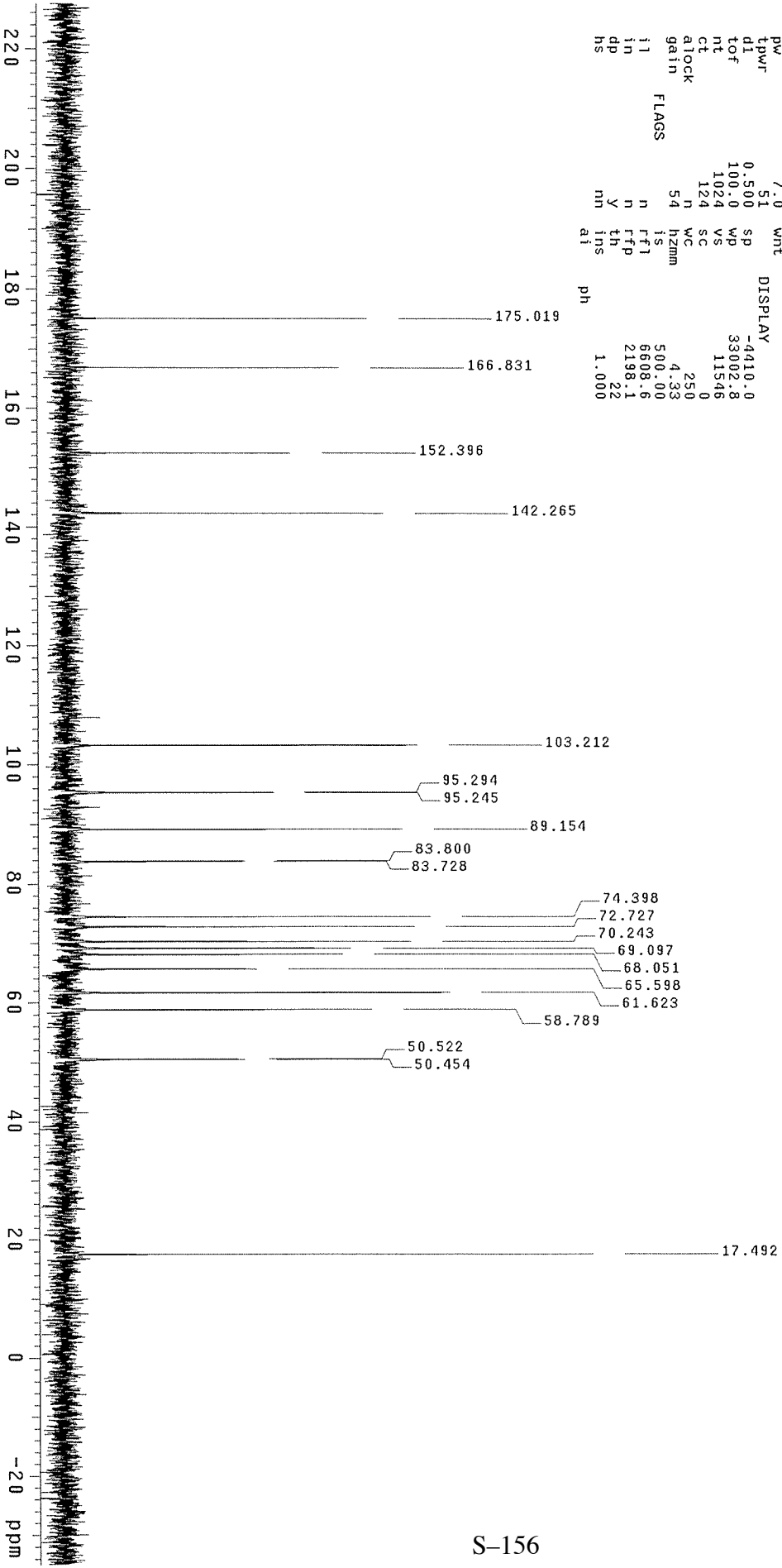
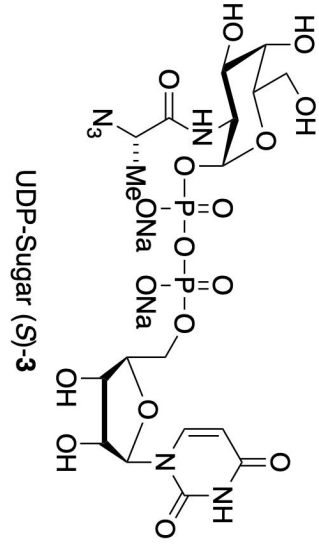
UDP-Sugar (S)-3



JC1125\_13C\_D20

exp2 s2pu1

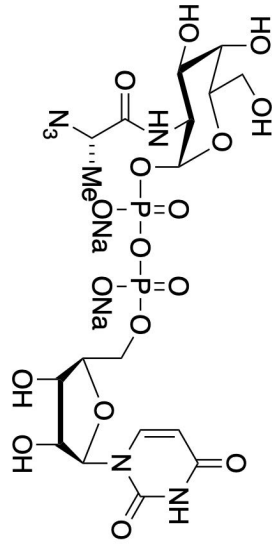
SAMPLE DEC. & VT H1  
 date Jul 7 2017 dn  
 solvent D2O dof -499.0  
 file /export/home/~ dm  
 Junwei/JC1125\_13~ dmm yyy  
 C=D20.f1d dmf 11400  
 ACQUISITION dpwr 43  
 srfq 125.674 PROCESSING 2.00  
 tn C13 1b not used  
 at 1.500 fn  
 mp 99016 math  
 sw 33003.3  
 fd 18000 werr  
 bs 4 wbs  
 pw 7.0 wexp  
 tpwr 51 wnt  
 dl 0.500 sp  
 tof 100.0 wd -4410.0  
 nt 1024 vs 33002.8  
 ct 124 SC 11546  
 atlock N WC 250  
 gain 54 hzmm 4.33  
 il n IS 500.00  
 in n rfp 6608.6  
 dp Y th 2198.1  
 bs mn ins 22  
 ai ph 1.000



JC1125\_31P\_D20

exp2 s2pu1

SAMPLE DEC. & VT  
 date Jul 7 2017 499.752  
 solvent D2O dn H1 43  
 file exp dpwr 0  
 ACQUISITION dof  
 sfrq 202.297 dm yyy  
 tn P31 dmm 11400  
 at 1.002 dmf  
 np 160254 dseq  
 sw 80000.0 dres 1.0  
 fb 44000 homo n  
 bs 4  
 tpw 51 dffq2 DEC2 0  
 pw 6.6 dn2  
 dl 2.000 dpwr2 1  
 tof 0 dof2 0  
 nt 16 dm2 n  
 ct 8 dmm2 c  
 atlock n dmf2 9900  
 gain 30 dseq2  
 dres2  
 flags 1.0  
 i1 n homoz  
 in y PROCESsing 2.00  
 dp n  
 hs n  
 DISPLAY nm  
 sp -45146.5 ft  
 wp 73999.4 fn not used  
 vs 13477 math  
 sc 0 werr  
 wc 250 wexp  
 hzmm 10.93 wbs  
 is 500.00 wnt  
 ffl 45147.1  
 ffd 0  
 th 17  
 ins 1.000  
 al cdc ph



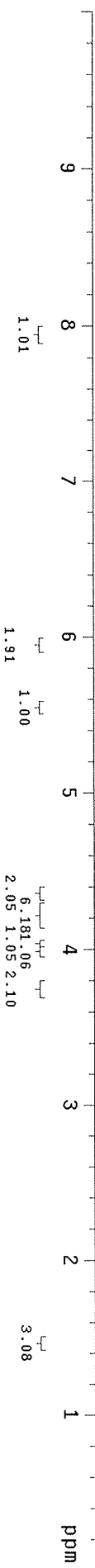
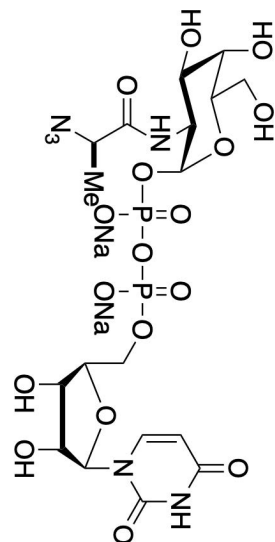
-10.451  
 -10.554  
 -12.219  
 -12.322



JC2009\_1H\_D20

exp2 s2pu1

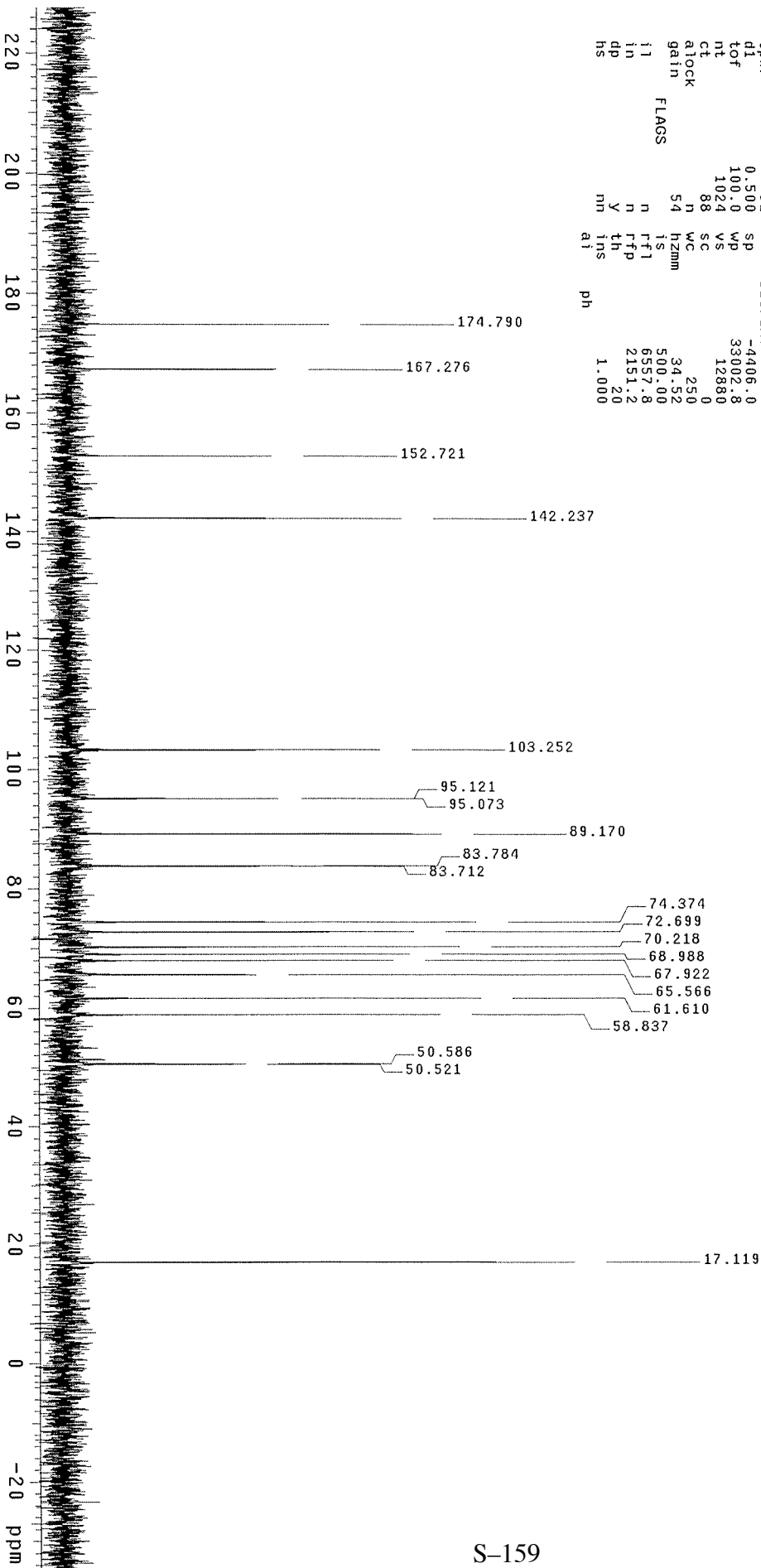
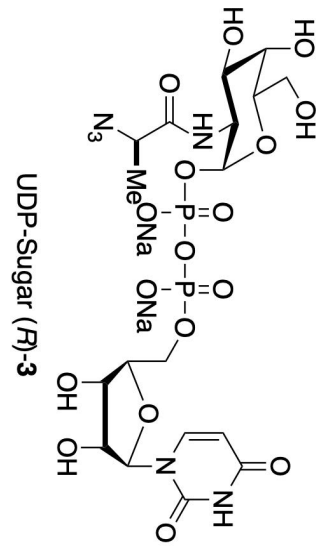
SAMPLE 7 2017 DEC. & VT 499.752  
 date Jul 7 2017 dfrq dn H1  
 solvent D2O d1 dn H1  
 file ACQUISITION exp dpwr 30  
 sfrq 499.752 dm dof 0  
 tn H1 dm mm  
 at 4.000 dmf C  
 np 64000 dseq 200  
 sw 8000.0 dres 1.0  
 fd 4000 homo n  
 bs 4  
 tpwr 60 dfrq2 DEC2 0  
 pw 8.0 dn2 0  
 d1 0 dpwr2 1  
 tof 0 dot2 1  
 nt 64 dm2 n  
 ct 28 dmm2 n  
 atock n dmf2 C  
 gain 40 dres2 200  
 flags 1.0  
 i1 n homo2  
 in n PROCESSING  
 dp y wfillie  
 hs nm math 65536  
 DISPLAY -0.2 ft  
 sp 4997.3 fn  
 wd 151 weft  
 vs 250 wexp  
 sc 0 wds  
 wc 250 wnt  
 hzmm 19.99  
 is 33.57  
 rff1 3832.5  
 rffp 2393.8  
 th 7  
 ins 1.000  
 nm cdc ph



JC2009\_13C\_D20

exp2 s2pu1

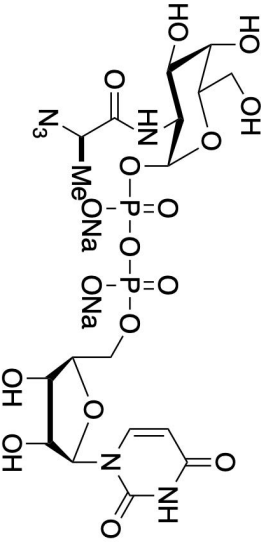
SAMPLE DEC. & VT H1  
 date Jul 7 2017 dn  
 solvent D2O dof -499.0  
 file /export/home/~ Junwcho1/JC2009\_13~ dm yyy  
 Junwcho1/JC2009\_13~ dm W  
 C\_D20.f1d dmf 11400  
 ACQUISITION dpwr 43  
 sfrq 125.674 PROCESSING 2.00  
 tn C13 1b fn not used f  
 at 1.500 math  
 md 99016  
 sw 33003.3 weff  
 fd 18000 wbs  
 bs 7.0 wds  
 pw 7.0 wnt  
 tpwr 51 DISPLAY -4406.0  
 d1 0.500 wp 33002.8  
 tof 100.0 vs 12880  
 nt 1024 SC 0  
 ct 88 WC 250  
 atlock n hzmm 34.52  
 gain 54 TS 500.00  
 i1 n rffl 6557.8  
 in n rfp 2151.2  
 dp Y th ins 20  
 hs mn ai ph 1.000



JC2009\_31P\_D20

exp2 s2pu1

SAMPLE DEC. & VT  
 date Jul 7 2017 dfrq 499.752  
 solvent D2O dn H1 43  
 file dpwr 0  
 ACQUISITION exp 202.297  
 sfrq 202.297 dm YYY  
 th P31 dmm W  
 at 1.002 dmf 11400  
 mp 160254 dseq  
 sw 80000.0 dres 1.0  
 fb 44000 homo n  
 bs 4  
 tpwr 51 dfrq2 DEC2 0  
 pw 6.6 dn2  
 dl 2.000 dpwr2 1  
 tof 0 dof2 0  
 nt 16 dm2 n  
 ct 8 dmm2 C  
 atock n dmf2 9900  
 gain 30 dseq2  
 dres2  
 homo2 1.0  
 i1 n  
 in n  
 dp y  
 hs n  
 DISPLAY nm  
 sp -45146.5 wtfile 2.00  
 wp 79999.4 fn  
 vs 12462 math not used  
 sc 0 werr  
 wc 250 wexp  
 hzmm 10.93 wbs  
 is 500.00 wnt  
 rffl 45147.1  
 rfp 0  
 th 17  
 ins 1.000  
 al cdc ph



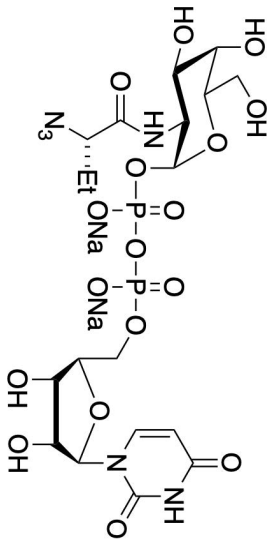
10.566  
 10.668  
 12.289  
 12.385



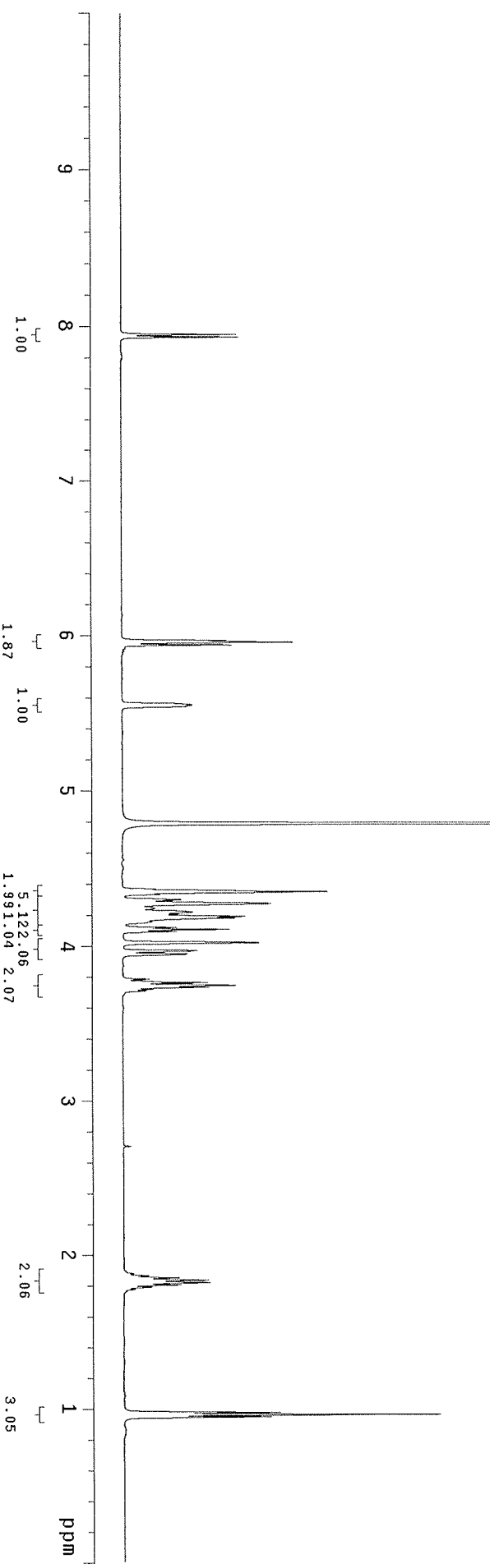


JC2007\_1H\_D20  
 exp2 s2pu1

SAMPLE DEC. & VT 499.752  
 date Jul 22 2017  
 solvent D2O  
 title ACQUISITION  
 ACQUISITION  
 sfrq 499.752  
 tn H1  
 at 4.000  
 mp 64000  
 sw 8000.0  
 fd 40000  
 bs 4  
 tpwr 60  
 pw 8.0  
 d1 0  
 tof 0  
 nt 64  
 ct 64  
 alock n  
 gain 40  
 flags  
 il n  
 in n  
 dp y  
 hs n  
 DISPLAY -0.2  
 SP 4997.3  
 WD 234  
 VS 0  
 SC 0  
 WC 250  
 hzmm 19.99  
 is 33.57  
 ffl 3836.4  
 ffp 2393.8  
 th 1.000  
 ins 1.000  
 nm cdc ph



UDP-Sugar (S)-4



JC2007\_13C\_D20

exp2 s2pu1

SAMPLE DEC. & VT

date Jul 7 2017 dn H1

solvent D2O dof -499.0

file /export/home/~ Junwchoi/JC2007\_13~ dmm yyy

Junwchoi/JC2007\_13~ dmm yyy

C\_D20\_T1d dmt 11400

ACQUISITION dpwr 43

strq 125.674 PROCESSING 43

th C13 1b 2.00

at 1.500 fn not used f

np 99016 math

sw 33003.3

fd 18000 werr

bs 4 wexp

pw 7.0 wds

tpwr 51 wnt

dl 0.500 sp DISPLAY

tof 100.0 wp -4405.4

nt 1024 vs 33002.8

ct 240 SC 13872

atlock N WC 0

gain 54 hzmm 250

l1 n IS 3.47

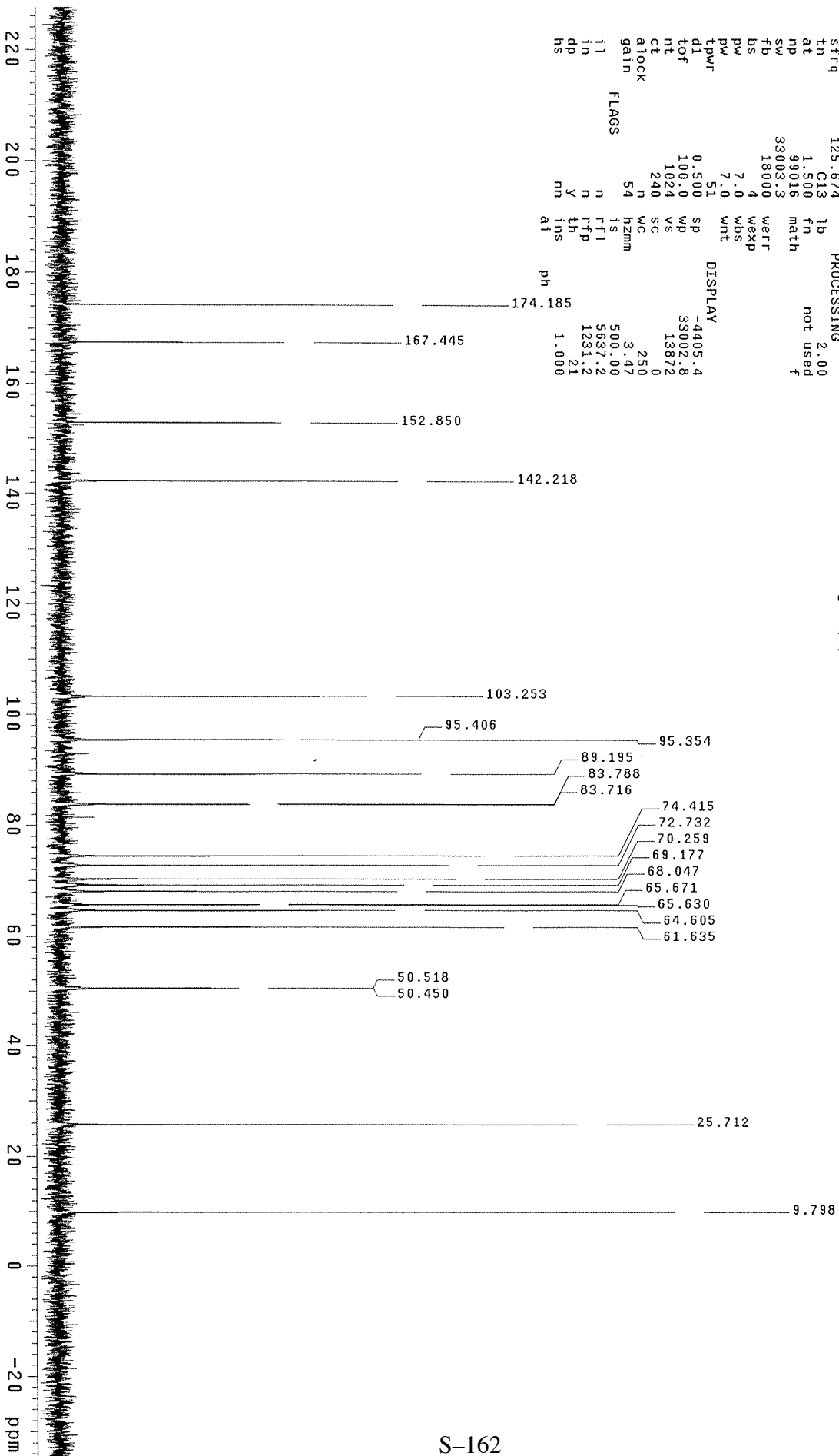
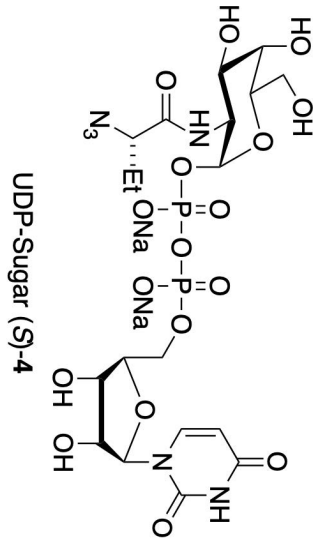
in n rffl 500.00

dp y th 5637.2

hs mn ins 1231.2

ai ph 21

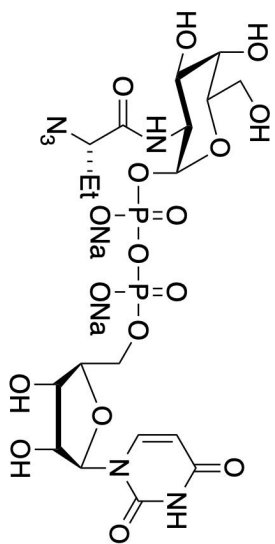
1.000



JC2007\_31P\_D20

exp2 s2pu1

SAMPLE DEC. & VT  
 date Jul 7 2017 dfrq 499.752  
 solvent D2O dn H1  
 file exp dpwr 43  
 ACQUISITION exp dof 0  
 sfrq 202.297 dm YYY  
 ln P31 dmm W  
 at 1.002 dmf 11400  
 np 160254 dseq  
 sw 80000.0 dres 1.0  
 fb 44000 homo n  
 bs 4  
 tpwr 51 dfrq2 DEC2  
 pw 6.6 dn2 0  
 dl 2.000 dpwr2 1  
 tof 0 dof2 0  
 nt 16 dm2 n  
 ct 8 dmm2 C  
 atlock n dmf2 9900  
 gain 30 dseq2  
 dres2 1.0  
 homo2 n  
 il n  
 in n  
 dp y lb  
 hs nm wfile 2.00  
 PROCESsing  
 ft  
 sp -45146.5 fn  
 wd 79999.4 math not used  
 vs 8724 math f  
 SC 250 weff  
 WC 10.93 wexp  
 hzmm 500.00 wbs  
 ts 45147.1 wnt  
 rfp 0  
 th 17  
 ins 1.000  
 al cdc ph



UDP-Sugar (S)-4

-10.454  
 -10.557  
 -12.285  
 -12.385



JC2029\_1H\_D20

exp2 s2pu1

SAMPLE

date Jul 22 2017

solvent D2O

file ACQUISITION

sfrq 499.752

tn H1

at 4.000

np 64000

sw 8000.0

fb 40000

ds 4

tpwr 60

pw 8.0

d1 0

tof 0

nt 64

ct 44

atock n

gain 40

flags n

il n

in n

dp y

hs n

DISPLAY

sp -0.2

wd 4997.3

vs 363

sc 0

wc 250

hzmm 19.99

is 33.57

rffl 3836.9

th 2393.7

ins 1.000

nm cdc ph

DEC. & VT

dfreq 499.752

dn H1

dpwr 30

doof 0

dm nmn

dmm C

dsef 200

dres n

homo 1.0

DEC2

dfreq2 0

dn2

dpwr2 1

dof2 0

dm2 n

dmm2 C

dseq2 200

dres2 n

homo2 1.0

PROCESSING

wf file

proc ft

fn 65536

math f

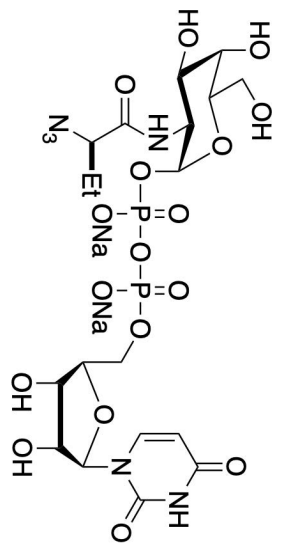
weff

wexp

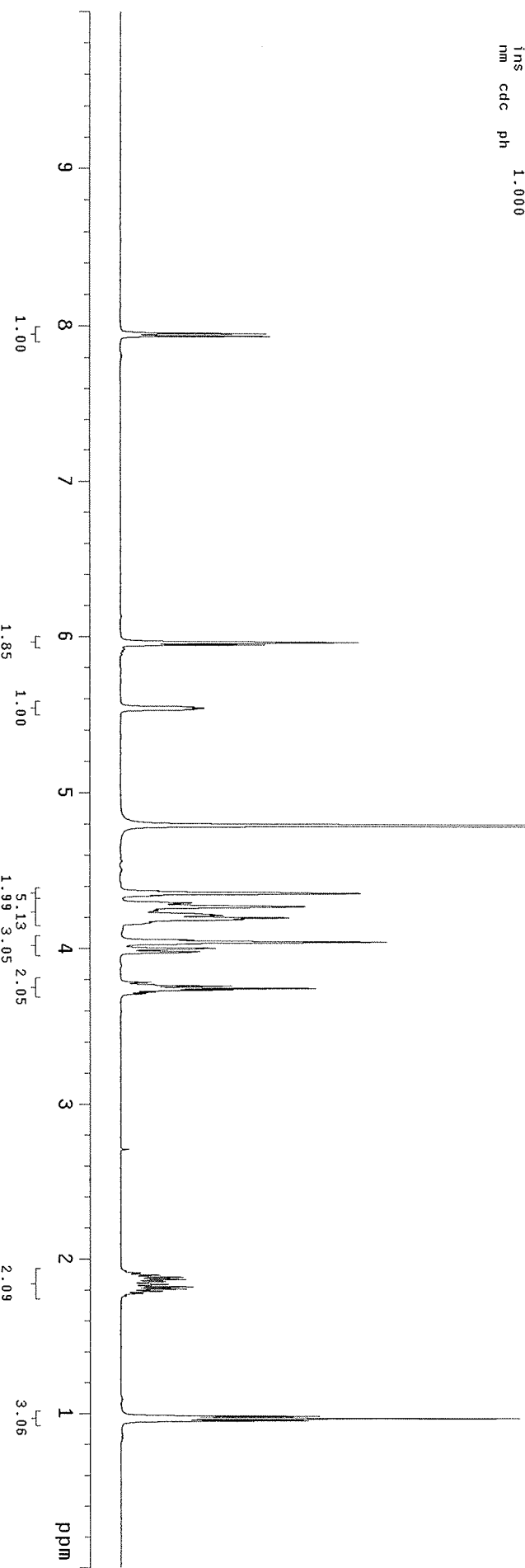
wbs

wnt

wft



UDP-Sugar (R)-4



JC2029\_13C\_D20

exp2 s2pu1

SAMPLE DEC. & VT

date Jul 7 2017 dn H1

solvent D2O dof -499.0

file /export/home/~ junwchoi/JC2029\_13~ dm YYY

Junwchoi/JC2029\_13~ dmm W

C D20.F1d dmf 11400

ACQUISITION dpwr 43

sfrq 125.674 PROCESSING 2.00

tn C13 1b not used f

at 1.500 fn math

mp 99016 math

sw 33003.3

fd 18000 weff

bs 7.0 wexp

pw 7.0 wds

tpwr 51 wnt

d1 0.500 sp DISPLAY -4412.0

tof 100.0 wp 33002.8

nt 1024 vs 6366

ct 60 SC 0

atock n WC 250

gain 54 nzm 4.51

l1 n IS 500.00

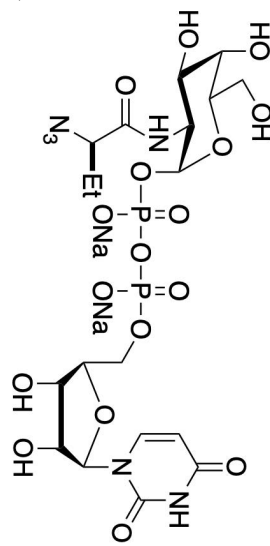
l1 n rfl 5661.9

dp y rfp 1249.3

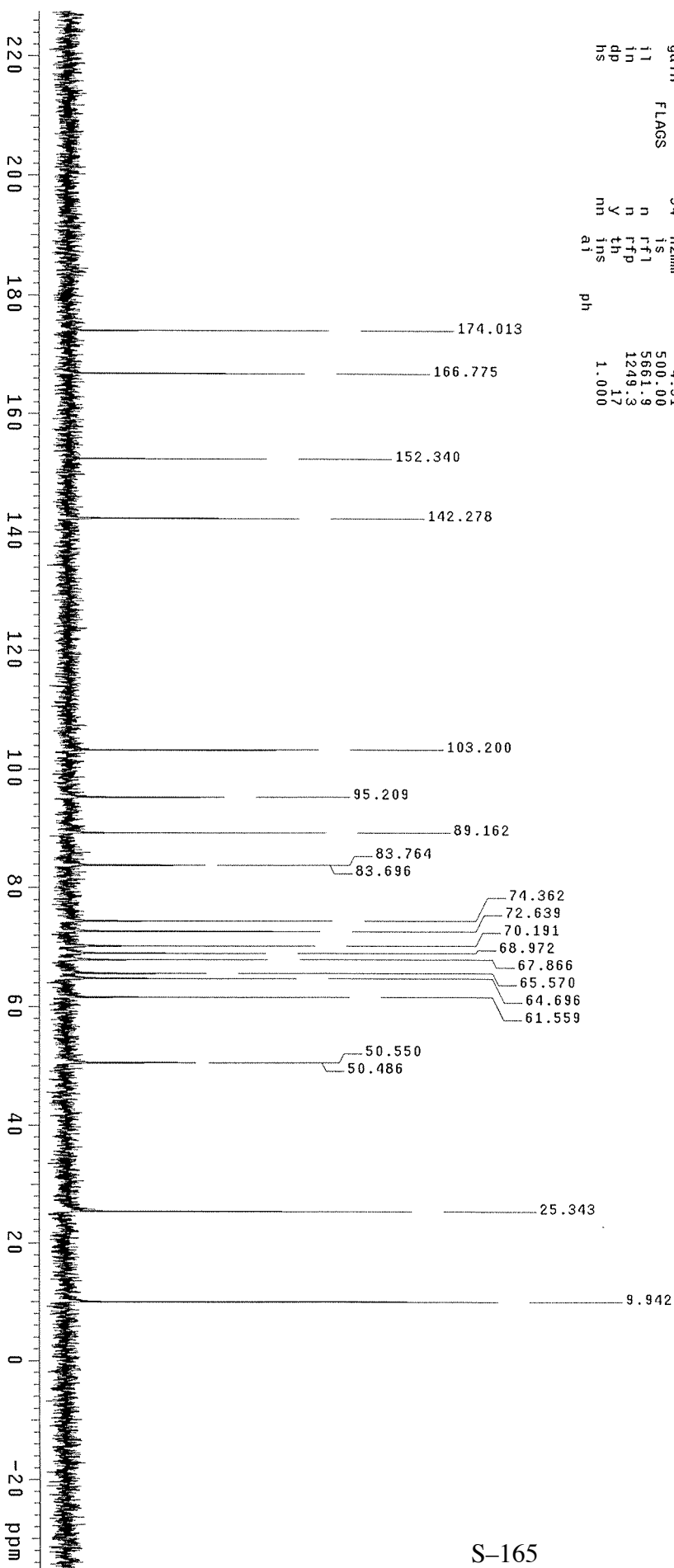
hs nn ins 17

ai

ph 1.000



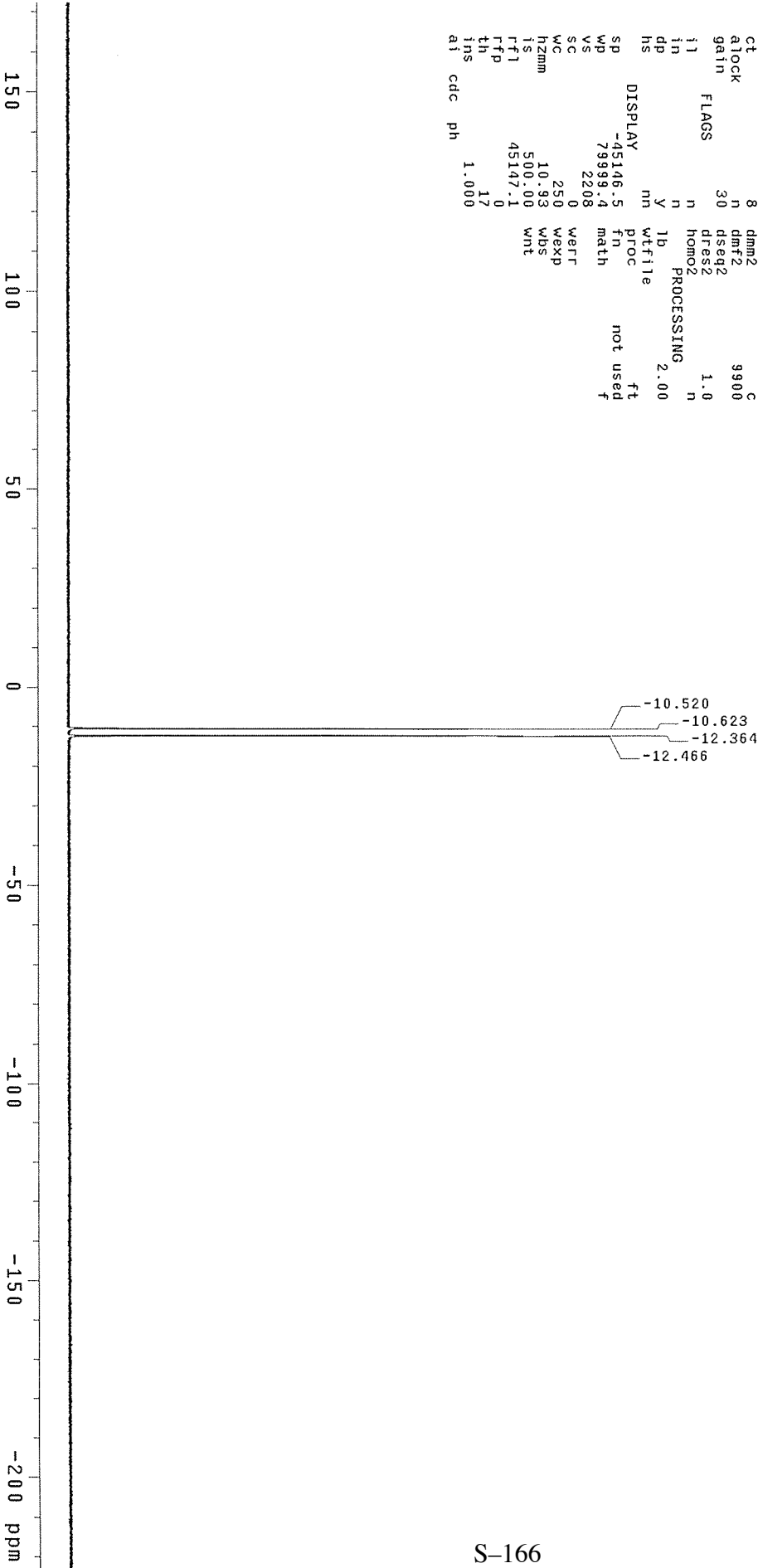
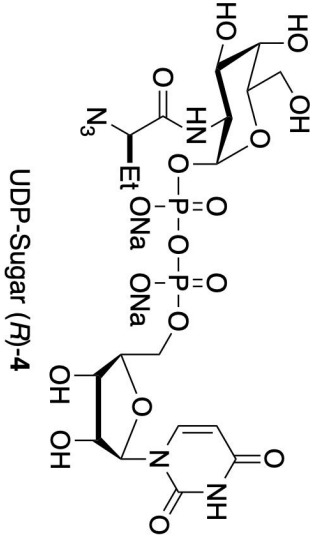
UDP-Sugar (F)-4



JC2029\_31P\_D20

exp2 s2pul1

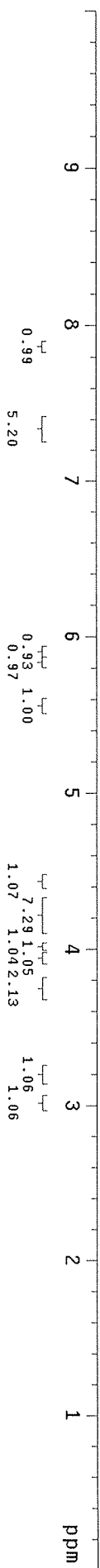
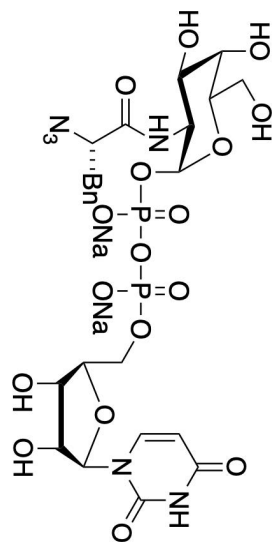
SAMPLE	date	7	2017	DEC. & VT	499.752
SOLVENT	solvent	D2O	dn	H1	43
FILE ACQUISITION	exp	202.297	dm	YYY	W
	dm	P31	dmm	dmf	11400
	at	1.002	dseq	dres	1.0
	mp	160254	dres	homo	n
	sw	80000.0	homo	DEC2	0
	fb	44000	dfiq2	dn2	1
	bs	4	dpwr2	dof2	n
	tpwr	51	dm2	dmf2	n
	pw	6.6	dseq2	dres2	n
	d1	2.000	homo2	PROCRESSING	2.00
	tof	0	PROCESSING	ft	not used
	nt	16	ft	math	f
	ct	8	math		
	atock	n			
	gain	30			
	FLAGS				
	i1	n			
	in	n			
	dp	y			
	hs	nm			
	DISPLAY	-45146.5			
	sp	79999.4			
	wp	2208			
	vs	0			
	sc	250			
	wc	10.93			
	hzmm	500.00			
	ts	45147.1			
	rfl	0			
	rffl	0			
	th	17			
	ins	1.000			
	at	cdc			
		ph			



JC1135\_1H\_D20

exp2 s2pu1

SAMPLE	date	Jul 18 2017	DEC. & VT	499.752
SOLVENT	d2o		H1	30
FILE	ACQUISITION	exp	dpwr	30
		499.752	doF	0
			dm	mm
			dmf	C
			dmm	200
			dseq	
			dres	
			homo	1.0
			homo2	n
			dfreq2	0
			dn2	
			dpwr2	1
			dot2	0
			dm2	n
			dmm2	C
			dseq2	200
			dres2	
			homo2	1.0
			homo2	n
			PROCESsing	
			wfille	ft
			proc	fn
			math	65536
			math	f
			weff	
			wexp	
			wds	
			wnt	wft
			h2mm	
			h1	
			ffl	
			ffp	
			th	
			ins	
			nm	1.000
			cdc	ph



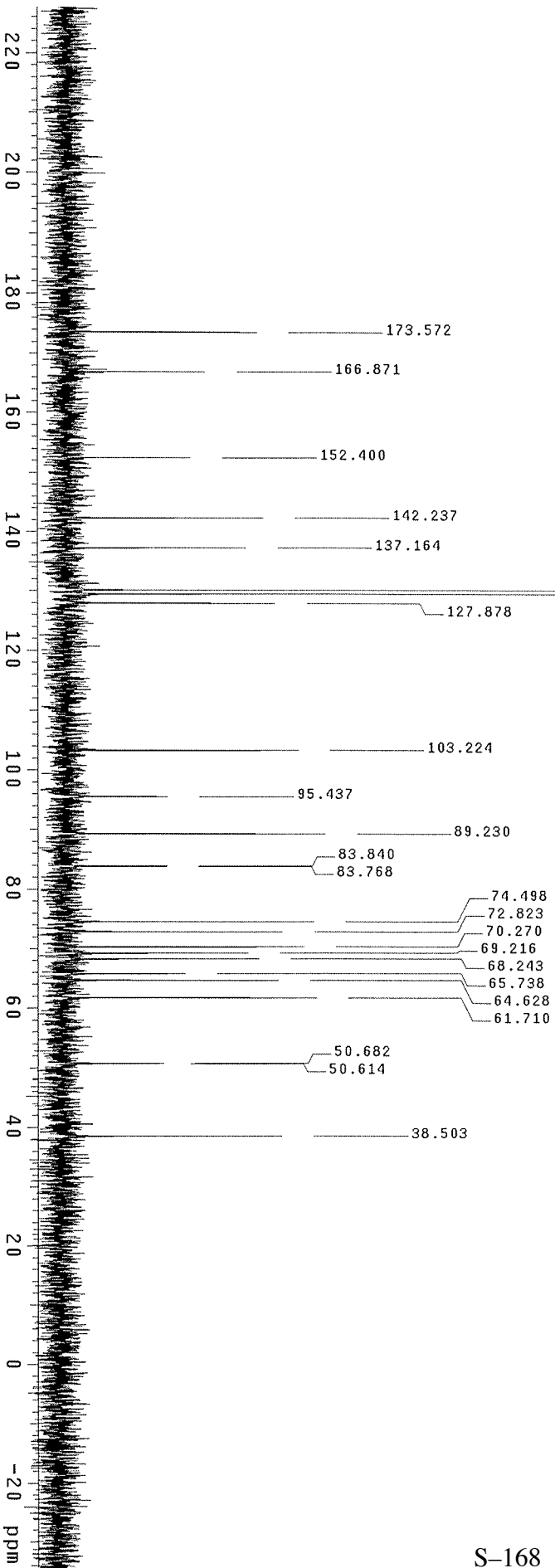
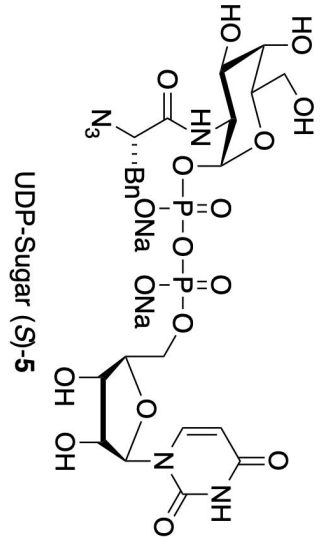
JC1135\_13C\_D20

exp3 s2pul1

SAMPLE DEC. & VT H1  
 date Jul 18 2017 dn  
 solvent D2O dof -499.10  
 file /export/home/~ Junwchoi/JC1135\_13~ dm  
 Junwchoi/JC1135\_13~ dmm YYY  
 C\_D20\_T1d dmf W  
 11400  
 43

ACQUISITION  
 sfrq 125.674 dpwr  
 tn C13 1b  
 at 1.500 fn not used f  
 mp 99016 math  
 sw 33003.3  
 fd 18000 weff  
 bs 4 wexp  
 pw 7.0 wbs  
 tpwr 51 wnt  
 d1 1.000 sp  
 tof 100.0 wp -4398.0  
 nt 1024 vs 33002.8  
 ct 256 SC 21565  
 atlock N WC 250  
 gain S4 hzmm 3.30  
 i1 n ts 500.00  
 in n rffl 9236.9  
 dp Y th 4838.4  
 hs mn ins 10  
 ai ph 1.000

DISPLAY  
 -4398.0  
 33002.8  
 21565  
 0  
 250  
 3.30  
 500.00  
 9236.9  
 4838.4  
 10  
 1.000

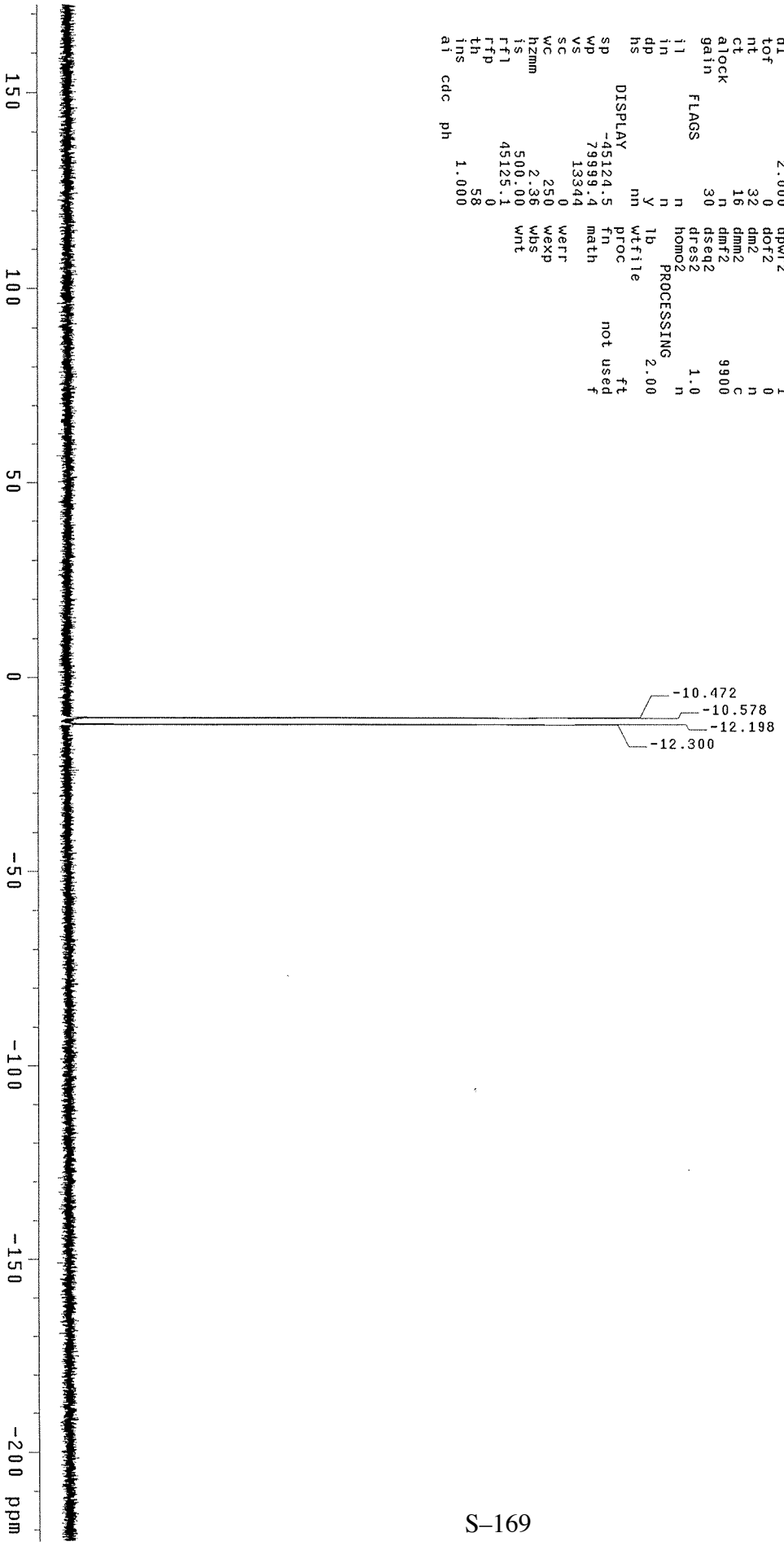
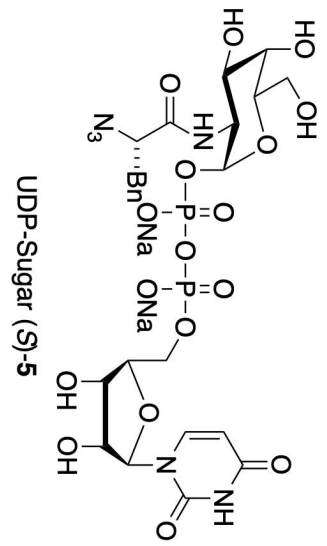




JC1135\_31P\_D20

exp2 s2pu1

SAMPLE		DEC. & VT	
date	Jul 18 2017	dfrq	499.752
solvent	D2O	dn	H1
file	exp	dpwr	43
ACQUISITION	exp	dot	0
sfrq	202.297	dm	yyy
tn	P31	dmm	w
at	1.002	dmt	11400
np	160254	dseq	
sw	80000.0	dres	1.0
fb	44000	homo	n
bs	4	DECC2	
tpwr	51	dfrq2	0
pw	6.6	dn2	
d1	2.000	dpwr2	1
tof	0	dot2	0
nt	32	dm2	n
ct	16	dmm2	c
atlock	n	dsef2	9900
gain	30	dres2	1.0
FLAGS		homo2	n
il	n	PROCESSING	2.00
in	y	lb	
dp	n	wtfile	
hs	mn	proc	not used
DISPLAY		fn	ft
sp	-45124.5	math	f
wd	79999.4	weff	
vs	13344	wexp	
sc	0	wbs	
WC	250	wnt	
hzm	2.36		
is	500.00		
rfl	45125.1		
rfd	0		
th	58		
ins	1.000		
at	cdc		
ph			



JC1137\_1H\_D20

exp2 s2pu1

SAMPLE DEC. & VT

date Jul 18 2017 dfrq 499.752

solvent D2O dn H1

file exp H1

ACQUISITION dof 30

sfrq 499.752 dm 0

fn H1 dmm nmm

at 4.000 dmf 200

np 64000 dseq

sw 8000.0 dres

fb 4000 homo 1.0

bs 4 dfrq2 0

tpwr 60 dn2

pw 8.0 dn2

d1 0 dpwr2 1

tof 0 dof2 0

nt 32 dmm2 n

ct 32 dmf2 n

atlock n dmf2 200

gain 40 dseq2 1.0

il n homo2 n

in n wtfile

dp y proc

hs n math

DISPLAY ft

SP 4997.3 fn 65536

WP -0.2 math

VS 263 wert

SC 0 wexp

WC 250 wbs

h2mm 19.99 wft

is 33.57

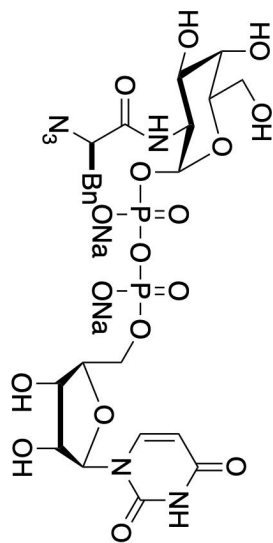
ftl 3836.2

ftf 2393.8

th 7

ins 1.000

nm cdc ph



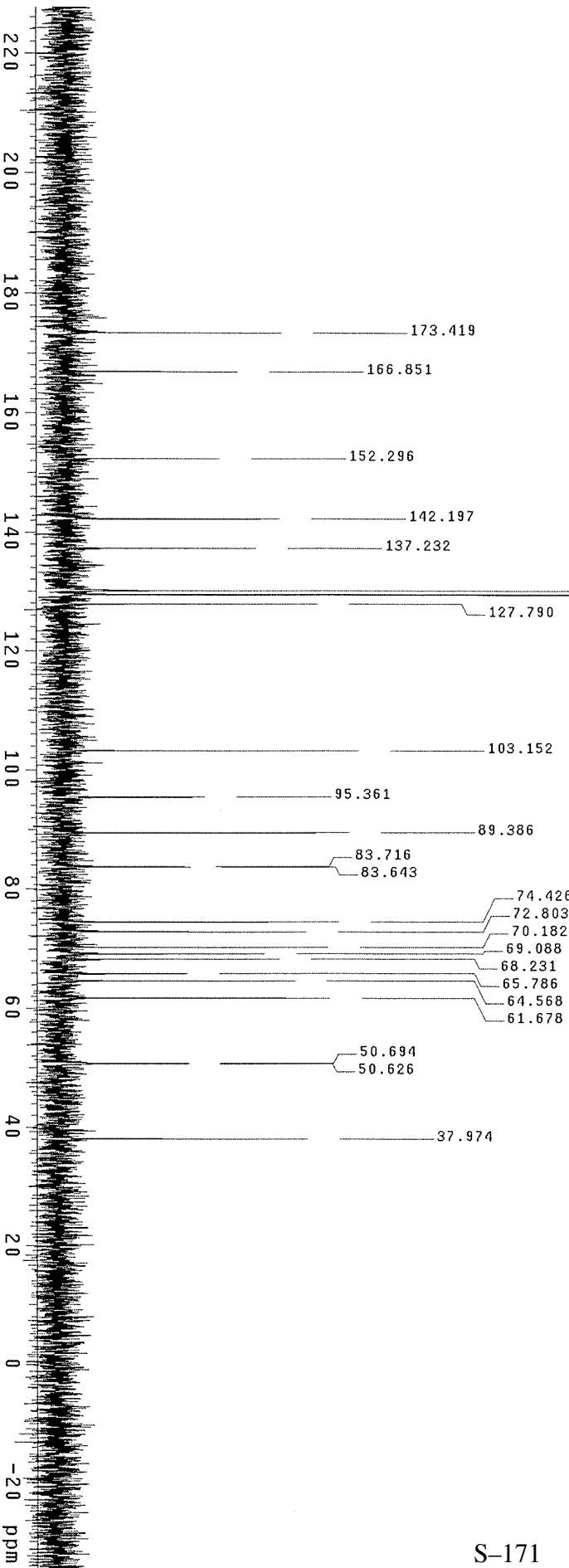
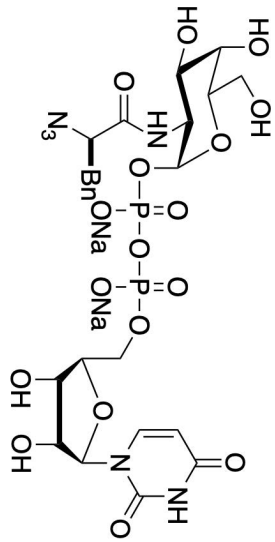
UDP-Sugar (R)-5



JC1137\_13C\_D20

exp3 s2pu1

SAMPLE DEC. & VI H1  
 date Jul 18 2017 dn  
 solvent D2O dof -499.0  
 title /export/home/~ dm  
 Junwchoi/JC1137\_13~ dmm W  
 C\_D20.f1d dmf 11400  
 ACQUISITION dpwr 43  
 sfrq 125.674 PROCESSING 2.00  
 tn C13 1b fn not used  
 at 1.500 math  
 np 99016  
 sw 33003.3 wert  
 fb 18000 wbs  
 bs 4 wexp  
 pw 7.0 wnt  
 tpwr 51  
 dl 1.000 SP -4402.0  
 tof 100.0 WP 33002.8  
 nt 1024 VS 24701  
 ct 240 SC 0  
 atlock n WC 250  
 gain 54 hzmm 1.56  
 i1 n ts 500.00  
 in n rfl 9174.4  
 dp y th 4771.9  
 hs nm ins 15  
 al ph 1.000



JC1137\_31P\_D20

exp2 s2pu1

SAMPLE date Jul 18 2017  
solvent D2O  
file exp  
ACQUISITION 202.297  
sfreq 202.297  
tn P31  
at 1.002  
np 160234  
sw 80000.0  
fb 44000  
bs 4  
tpwr 51  
pw 6.6  
d1 2.000  
tof 0  
nt 32  
ct 16  
atlock n  
gain 30  
flags n  
il n  
in n  
dp y  
hs mn  
DISPLAY -45124.5  
sp 79999.4  
vs 13344  
sc 0  
wc 250  
h2mm 2.36  
is 500.00  
rfi 45125.1  
rft 0  
th 72  
ins 1.000  
ai cdc ph

DEC. & VT 499.752

dn H1

dpwr 43

do 0

dm yyy

dmm W

dmt 11400

dres 1.0

homo n

dftg2 0

dn2 1

dpwr2 n

dotf2 0

dm2 n

dmm2 C

dmf2 9300

dseq2 1.0

homo2 n

PROCESSING 2.00

lb wtfile

proc ft

fn not used

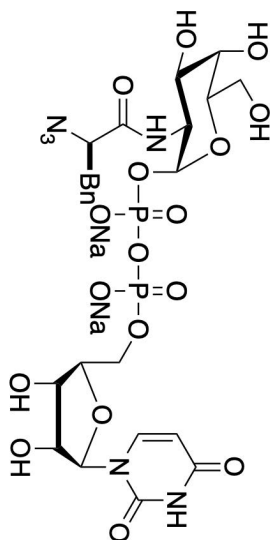
math f

werr

wexp

wbs

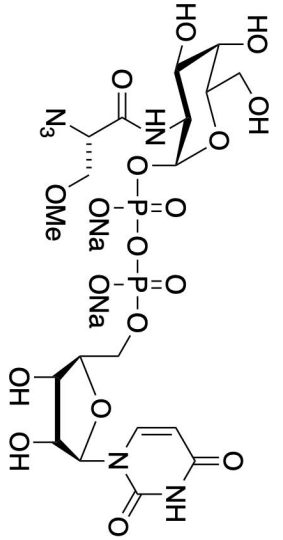
wrl



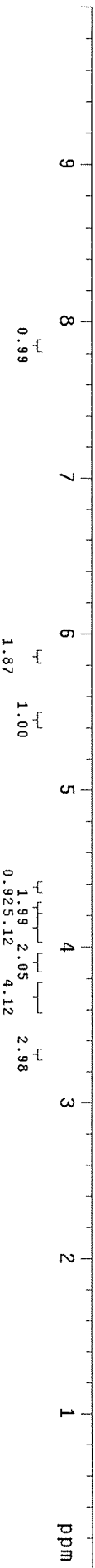
JC2025\_1H\_D20

exp2 s2pu1

SAMPLE	date	Jul 24 2017	DEC. & VT	499.752
solvent	D2O		dn	H1
file	ACQUISITION	exp	dpwr	30
			dot	0
sfrq	499.752	dm	mn	0
in	H1	dmm	c	200
at	4.000	dmf	dres	1.0
np	64000	dseq	homo	n
sw	8000.0	dres	DECC2	0
fb	4000	homo	dfreq2	0
bs	4		dn2	8.0
tpwr	60		dpwr2	0
pw	8.0		do2	0
d1	0		dm2	64
tof	0		dmm2	n
nt	64		dmf2	c
ct	64		dseq2	200
atock	n		dmf2	c
gain	40		dres2	1.0
FLAGS			homo2	n
il	n		PROCRESSING	
in	y		wfille	ft
dp	mn		proc	fn
hs	math		math	65536
DISPLAY				f
sp	-0.2			
wp	4997.3			
vs	198			
sc	0			
WC	250			
h2mm	19.99			wft
is	33.57			
ff1	3836.4			
ffp	2343.8			
th	7			
ins	1.000			
nm	cdc			
ph				



UDP-Sugar (S)-6



JC2025\_13C\_D20

exp2 s2pu1

SAMPLE DEC. & VT

date Jul 7 2017 dn H1

solvent D2O dof -499.0

file /export/home/~ Junwchoi/JC2025\_13~ dm YYY

Junwchoi/JC2025\_13~ dmm W

C D20 Tid dmf 11400

ACQUISITION dpwr 43

sfrq 125.674 PROCESSING

in C13 1b 2.00

at 1.500 fn not used f

mp 99016 math

sw 33003.3

fb 18000 weff

bs 7.0 wexp

pw 7.0 wds

tpwr 7.0 wnt

dl 51 DISPLAY

dl 0.500 sp -4410.5

nt 100.0 wp 33002.8

ct 1024 vs 12651

atlock n 168 SC 0

gain n WC 250

l1 n Hzmm 4.88

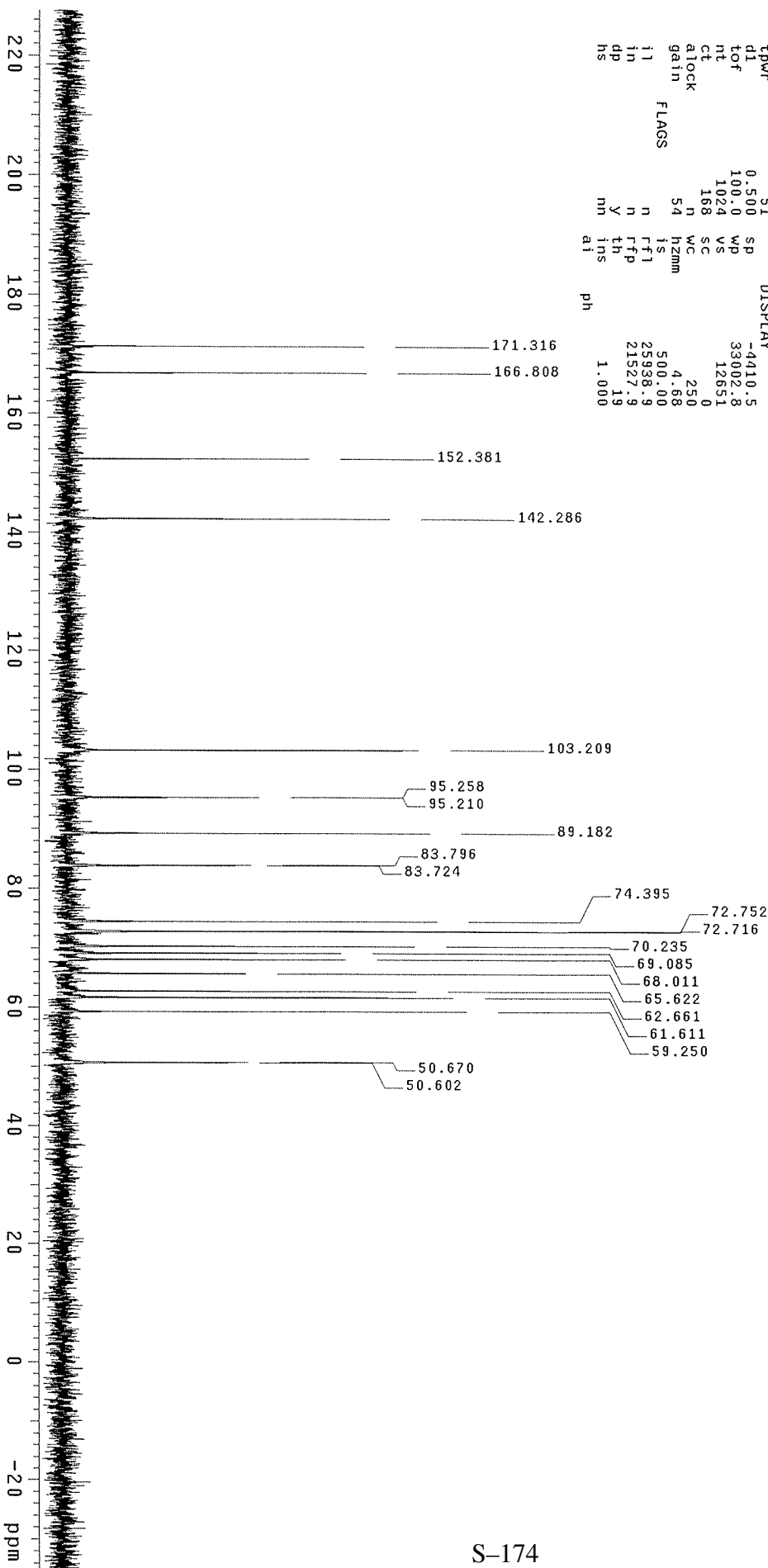
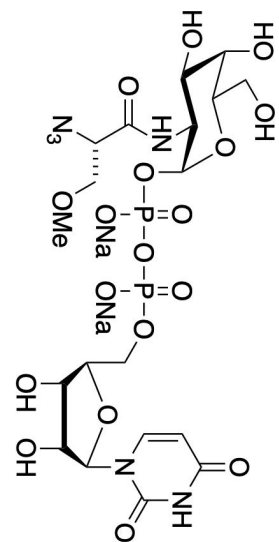
in n f1 500.00

dp n rfp 25938.9

hs n th 21527.9

ai ins 19

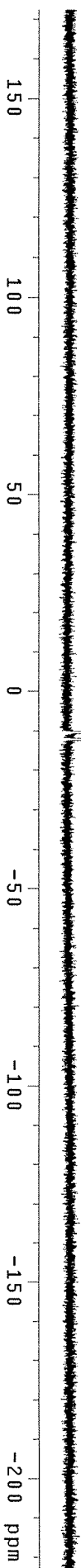
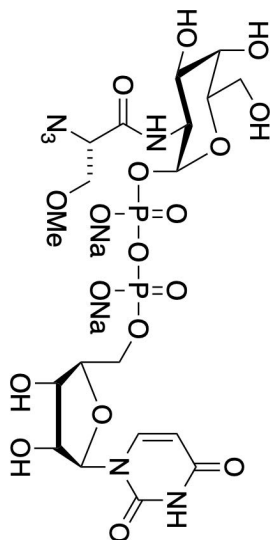
ph 1.000



JC2025\_31P\_D20

exp3 s2pul1

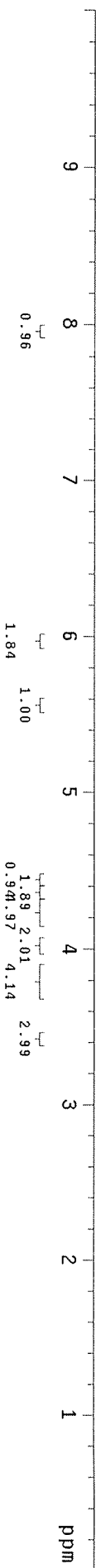
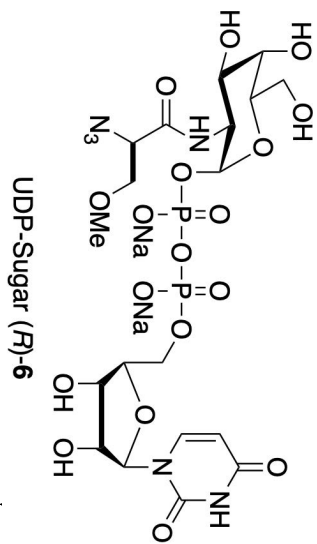
SAMPLE DEC. & VT  
 date Jul 7 2017 dfrrq 499.752  
 solvent D2O dn H1  
 file exp dpwr 43  
 ACQUISITION 202.297 dm 0  
 sffrq 202.297 dm YYY  
 tn P31 dmm W  
 at 1.002 dmf 11400  
 np 160254 dseq  
 sw 80000.0 dres 1.0  
 fb 44000 homo n  
 bs 4  
 tpwr 51 dfrrq2 DEC2  
 pv 6.6 dn2 0  
 di 2.000 dpwr2 1  
 tof 0 dof2 0  
 nt 32 dmm2 n  
 ct 16 dmf2 C  
 atlock n dseq2 9900  
 gain 30 dres2 1.0  
 f1 n homo2 n  
 in n  
 dp y 1b PROCESSING 2.00  
 hs nm wfile  
 DISPLAY -45131.8 ft  
 sp 79999.4 fn not used  
 ws 11837 math f  
 SC 0 weff  
 WC 250 wexp  
 hzmm 24.39 wbs  
 ls 500.00 wnt  
 rffl 45132.4  
 ffp 0  
 th 17  
 ins 1.000  
 ai cdc ph



JC2027\_1H\_D20

exp2 s2pu1

SAMPLE	date	2017	DEC. & VT	499.752
solvent	D2O	exp	H1	30
file	ACQUISITION	499.752	dm	0
sfreq	H1	dm	nmn	0
tn	H1	dmm	C	200
at	4.000	dmt	n	1.0
np	64000	dseq	homo	0
sw	8000.0	dres	DEC2	0
fb	4000	homo	dfreq2	60
bs	4	dn2	dpwr2	8.0
tpwr	60	dn2	dof2	0
pw	8.0	dn2	dm2	0
dl	0	dn2	dm2	0
tof	0	dn2	dm2	0
nt	64	dm2	dm2	0
ct	56	dm2	dm2	0
atlock	n	dm2	dm2	0
gain	40	dseq2	dres2	1.0
il	n	homo2	homo2	1.0
in	n	wtfile	wtfile	ft
dp	y	proc	proc	f
hs	nm	math	math	f
sp	-0.2	math	math	f
wp	4997.3	werr	werr	0
vs	220	wexp	wexp	0
sc	0	wbs	wbs	0
wc	250	wrt	wrt	0
h2mm	19.99	wrt	wrt	0
is	33.97	wrt	wrt	0
fft	3836.7	wrt	wrt	0
ffp	2393.8	wrt	wrt	0
th	7	wrt	wrt	0
ins	1.000	wrt	wrt	0
nm	cdc	ph	ph	0





JC2027\_13C\_D20

exp3 s2pu1

SAMPLE

DEC. & VT

H1

date Jul 7 2017

dn

-499.0

solvent D2O

dof

YYY

file /export/home/~

dm

w

Junwchoi/JC2027.13~

dmm

11400

C-D2O.fid

dmf

43

ACQUISITION

PROCESsing

2.00

sfrq 125.674

lb

not used

tn C13

fn

f

at 1.500

math

0

np 99016

weff

0

sw 33003.3

wexp

0

fb 18000

wbs

0

bs 4

wnt

0

pw 7.0

sp

0

tpwr 7.0

wp

0

di 51

vs

0

tof 0.500

sc

0

nt 100.0

hzm

250

ct 1024

is

500.00

atlock 284

rfp

25917.8

gain 54

th

21514.4

il n

ins

14

in n

ph

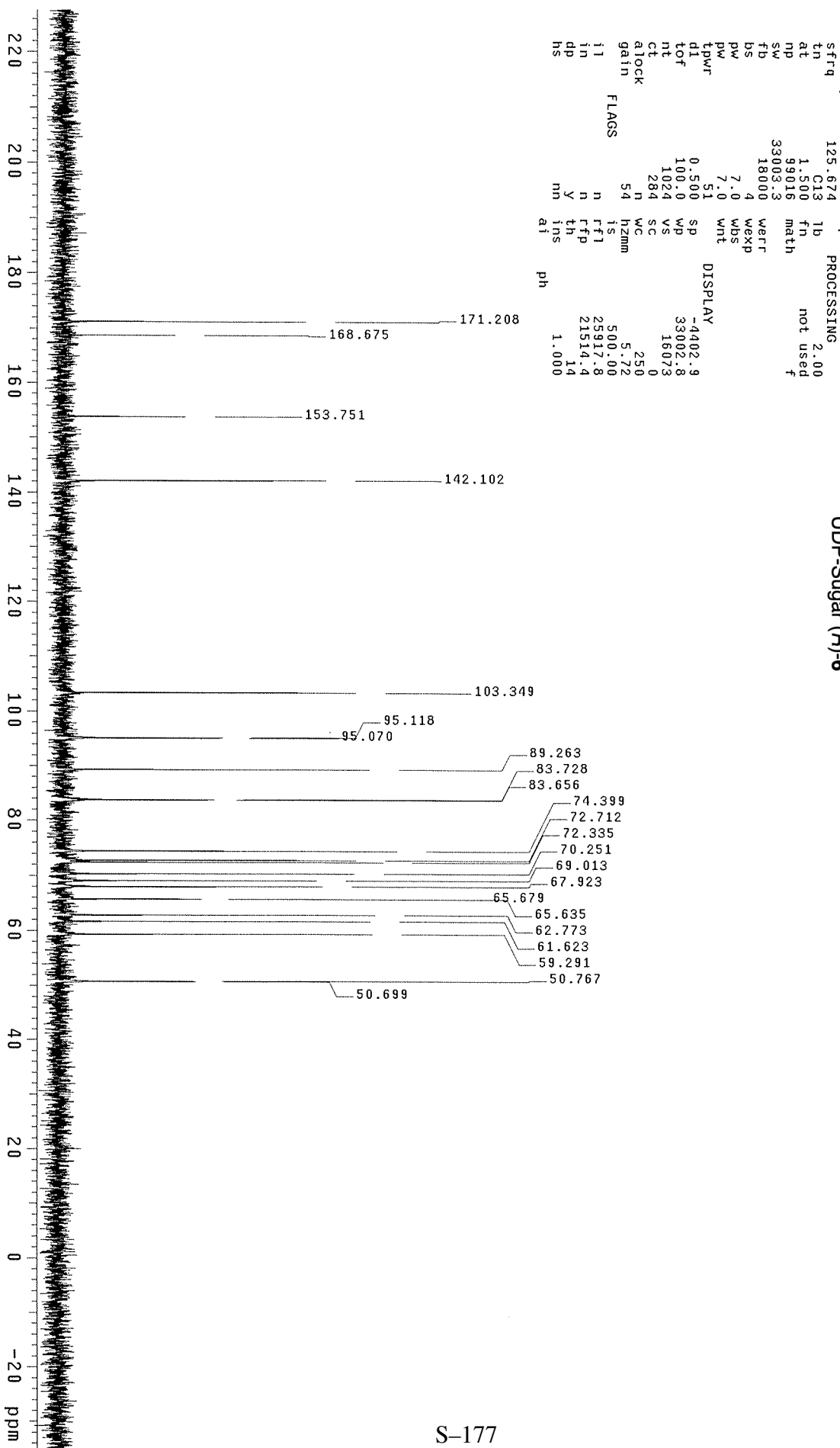
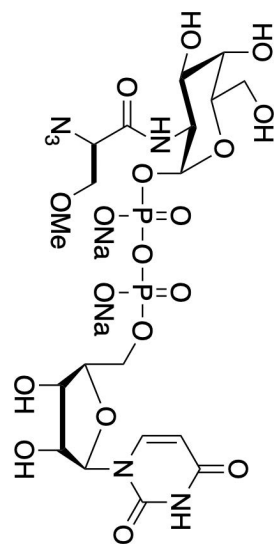
1.000

dp y

ai

0

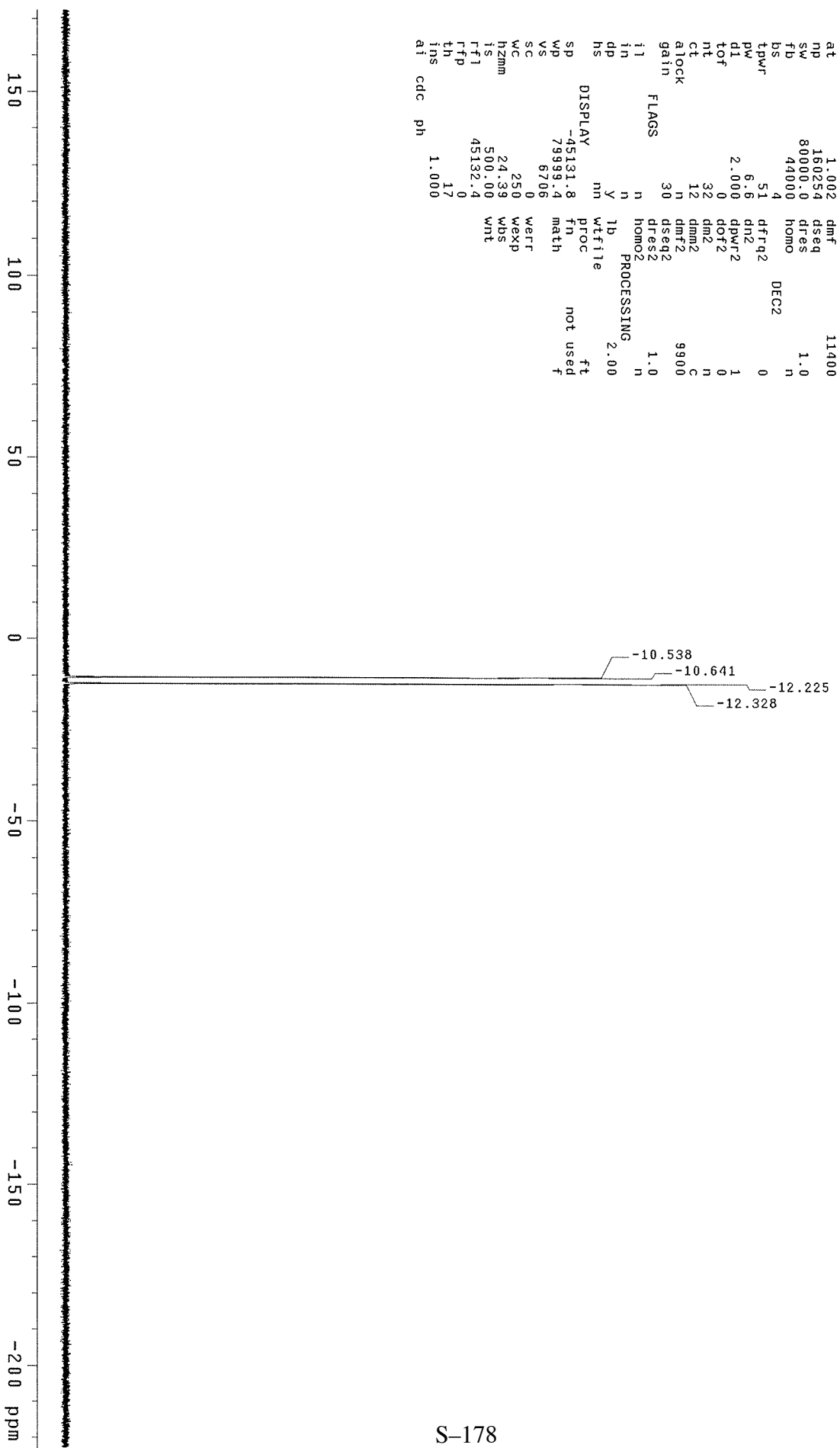
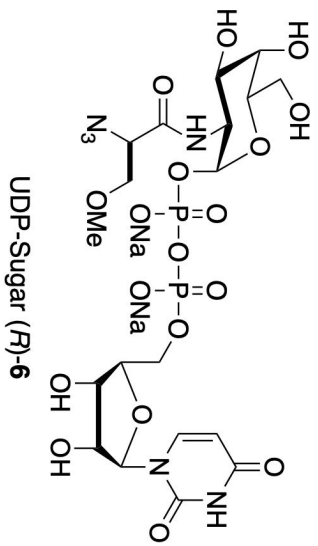
hs n



JC2027\_31P\_D20

exp3 s2pu1

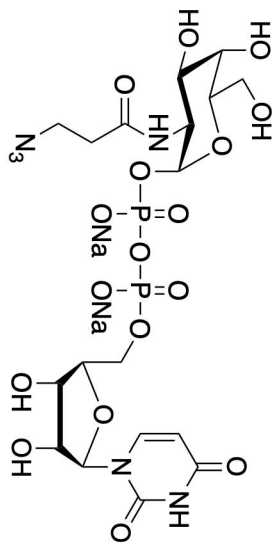
SAMPLE		DEC. & VT	
date	Jul 7 2017	dfrq	499.752
solvent	D2O	dn	H1
file	exp	dpwr	43
ACQUISITION	202.297	dof	0
sfrq	202.297	dm	yyy
tn	P31	dmm	w
at	1.002	dmt	11400
np	160254	dseq	
sw	80000.0	dres	1.0
fb	44000	homo	n
bs	4	dfrq2	DEC2
tdwr	51	dn2	0
pw	6.6	dpwr2	1
d1	2.000	dof2	0
tof	0	dm2	n
nt	32	dmm2	n
ct	12	dmt2	C
atlock	n	dseq2	9900
gain	30	dres2	1.0
FLAGS		homo2	n
i1	n	PROCESSING	2.00
in	y	wf1	ft
dp	n	proc	not used
hs	nn	math	f
DISPLAY			
sp	-45131.8		
wp	79999.4		
vs	6706		
sc	0	weff	
wc	250	wexp	
h2mm	24.39	wds	
is	500.00	wnt	
fft	45132.4		
th	0		
ins	17		
ai	1.000		



UDP-GalNAcCH2N3\_1H\_D2O

exp3 s2pu1

SAMPLE	date	Jul 13 2017	DEC. & VT	499.752
solvent	D2O	dn	H1	30
file	ACQUISITION	exp	dpwr	30
		doF		0
sfrq	499.752	dm	mm	
tn	H1	dmm	C	200
at	4.000	dmf		
np	64000	dseq		
sw	8000.0	dres	1.0	
fb	4000	homo	n	
bs	4			
tpwr	60	dfreq2	DEC2	0
pw	8.0	dn2		
dl	0	dpwr2		1
tof	0	doF2		n
nt	128	dm2		n
ct	28	dmm2	C	200
atock	n	dmf2		
gain	40	dseq2		1.0
flags		homo2		n
il	n		PROCESSING	
in	y	wfille		ft
dp	nm	proc		fn
hs	DISPLAY	math		65536
sp	-0.2			f
wd	4997.3	weft		
vs	284	wexp		
sc	0	wds		
wc	250	wnt		wft
h2mm	19.99			
ts	33.57			
rf1	3834.5			
rfp	2393.8			
th	7			
ins	1.000			
nm	cdc	ph		



UDP-Sugar 7



UDP-GalNAcCH2N3\_13C\_D20

exp3 s2pu1

SAMPLE DEC. & VT

date Jul 13 2017 dn H1

solvent D2O dof -499.0

file /export/home/~ junwchoi/UDP-GalNA~

CH2N3\_13C\_D20.fid dm 11400

ACQUISITION dpwr 43

sfrq 125.674 PROCESSING

tn C13 1b 2.00

at 1.500 fn not used

np 99016 math f

sw 33003.3

fb 18000 weff

bs 4 wexp

pw 7.0 wds

tpwr 7.0 wnt

dl 51 DISPLAY

tof 1.000 sp -4399.9

nt 100.0 wp 33002.8

ct 4000 vs 23001

atlock 1268 sc 0

gain 54 WC 250

FLAGS hzmm 2.25

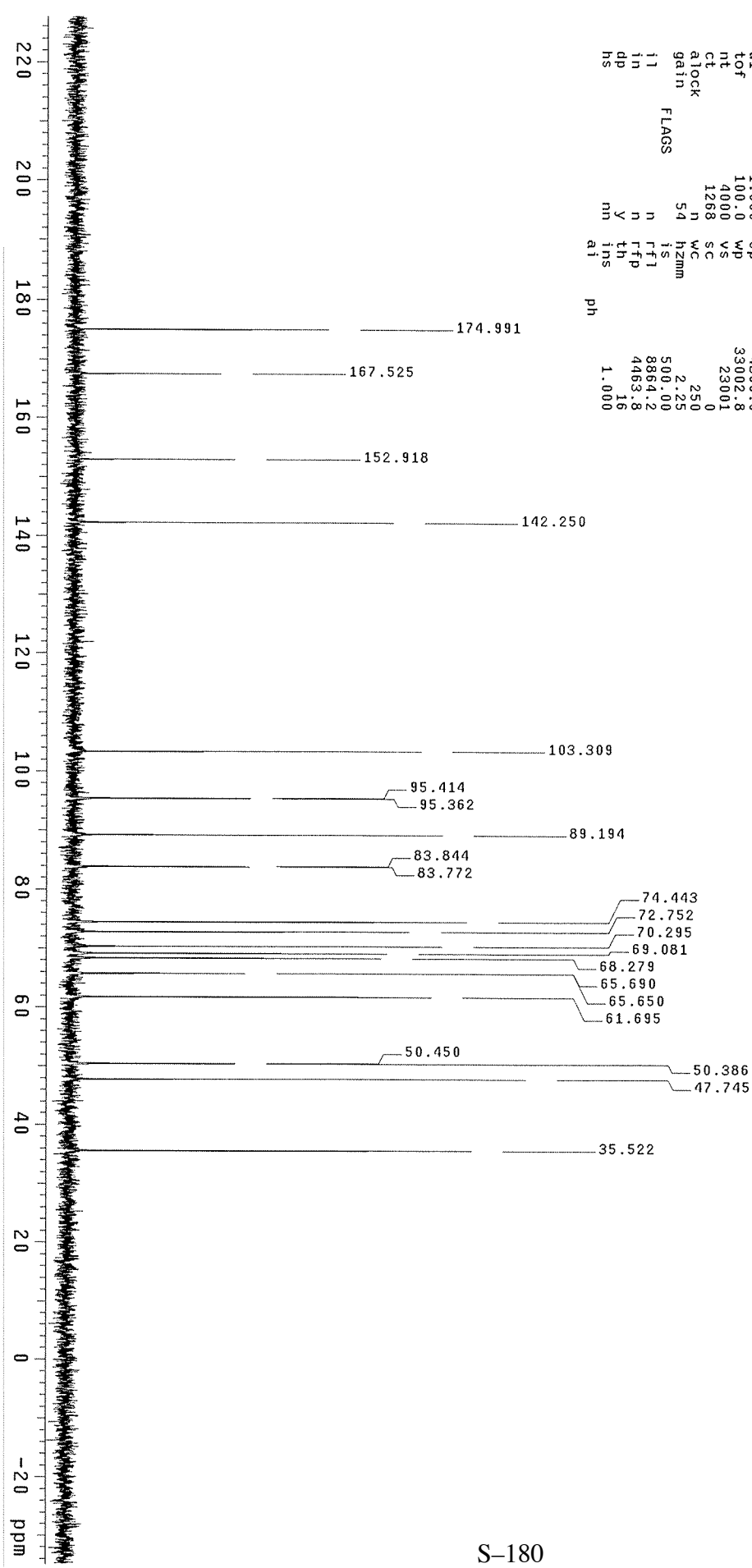
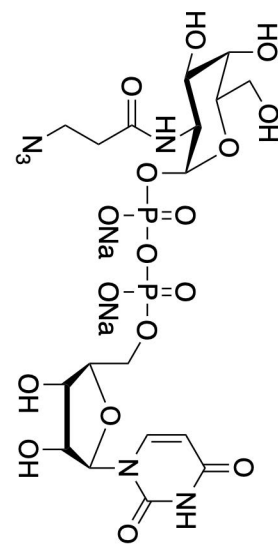
l1 n IS 509.00

in n rfl 8864.2

dp y rfp 4463.8

hs n ins 16

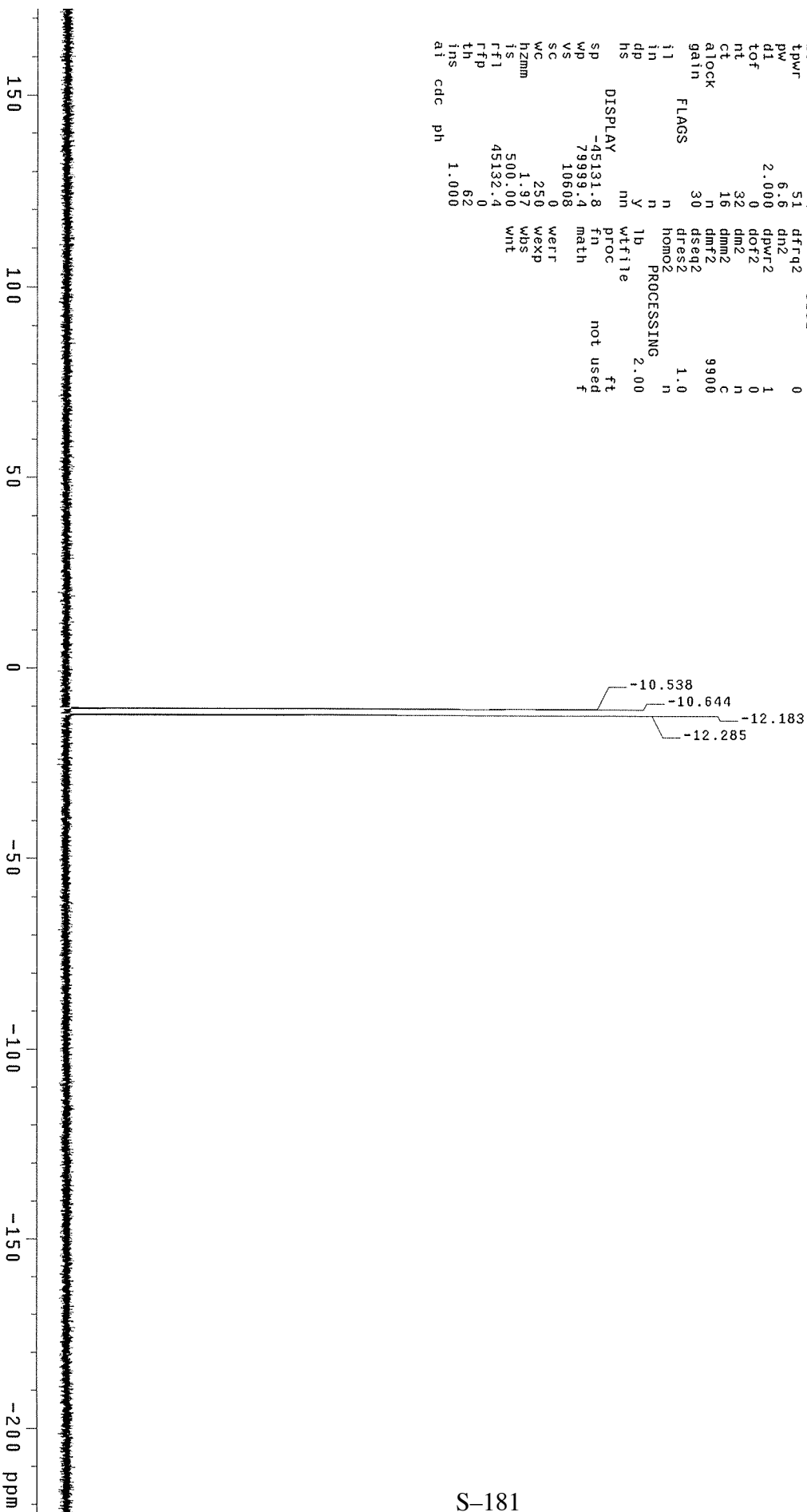
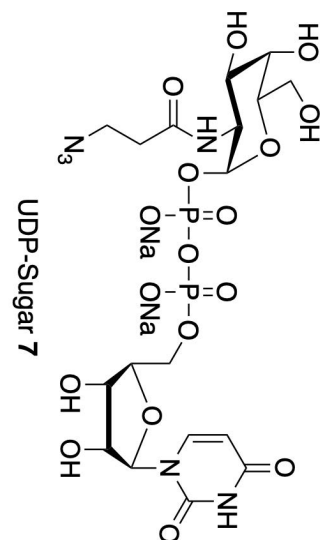
ph 1.000



UDP-GA1MACCH2N3\_31P\_D20

exp2 s2pul1

SAMPLE	date	Jul 13 2017	DEC. & VT	499.752
SAMPLE	solvent	D2O	dn	H1
SAMPLE	file		dpwr	43
ACQUISITION	exp		dof	0
ACQUISITION	sfrq	202.297	dm	yyy
ACQUISITION	tn	F31	dmm	w
ACQUISITION	at	1.002	dmt	11400
ACQUISITION	np	160254	dseq	
ACQUISITION	sw	80000.0	dres	1.0
ACQUISITION	fb	44000	homo	n
ACQUISITION	bs	4	DECC2	0
ACQUISITION	tpwr	51	dfreq2	0
ACQUISITION	pv	6.6	dn2	1
ACQUISITION	d1	2.000	dpwr2	0
ACQUISITION	tof	0	dot2	n
ACQUISITION	nt	32	dm2	n
ACQUISITION	ct	16	dmm2	c
ACQUISITION	atock	n	dmt2	9900
ACQUISITION	gain	30	dseq2	1.0
ACQUISITION	flags		homo2	n
ACQUISITION	i1	n	PROCESsing	2.00
ACQUISITION	in	n	wtfile	ft
ACQUISITION	dp	y	proc	fn
ACQUISITION	hs	nm	math	not used
ACQUISITION	sp	-45131.8	werf	f
ACQUISITION	wp	79999.4	wexp	
ACQUISITION	vs	10608	wbs	
ACQUISITION	sc	0	wnt	
ACQUISITION	wc	250		
ACQUISITION	hzm	1.97		
ACQUISITION	is	500.00		
ACQUISITION	ffl	45132.4		
ACQUISITION	rfp	0		
ACQUISITION	th	62		
ACQUISITION	ins	1.000		
ACQUISITION	ai	cdc		
ACQUISITION	ph			



JC3083\_1H\_D20

exp3 s2pu1

SAMPLE

date Jul 8 2017

solvent D2O

file exp

ACQUISITION

sfrq 499.752

tn H1

at 4.000

np 64000

sw 8000.0

fb 4000

bs 4

tpwr 60

di 8.0

tof 0

nt 32

ct 32

atlock n

gain 40

flags n

in n

dp y

ns n

sp -0.2

wd 4997.3

vs 383

sc 0

wc 250

h2mm 19.99

is 33.97

ftl 3835.2

ffp 2393.8

th 7

ins 1.000

nm cdc

ph

DEC. & VT

dfreq 499.752

dn H1

dpwr 30

dof 0

dm nmm

dmm nmm

dmf 200

dseq 200

dres 1.0

homo n

dfreq2 0

dn2 0

dpwr2 1

dof2 0

dmm2 n

dmf2 n

dseq2 200

dres2 1.0

homo2 n

PROCESSING

ft ft

proc fn

math 65536

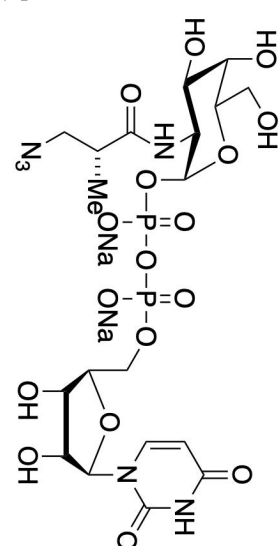
DISPLAY

weft wft

wexp wft

wbs wft

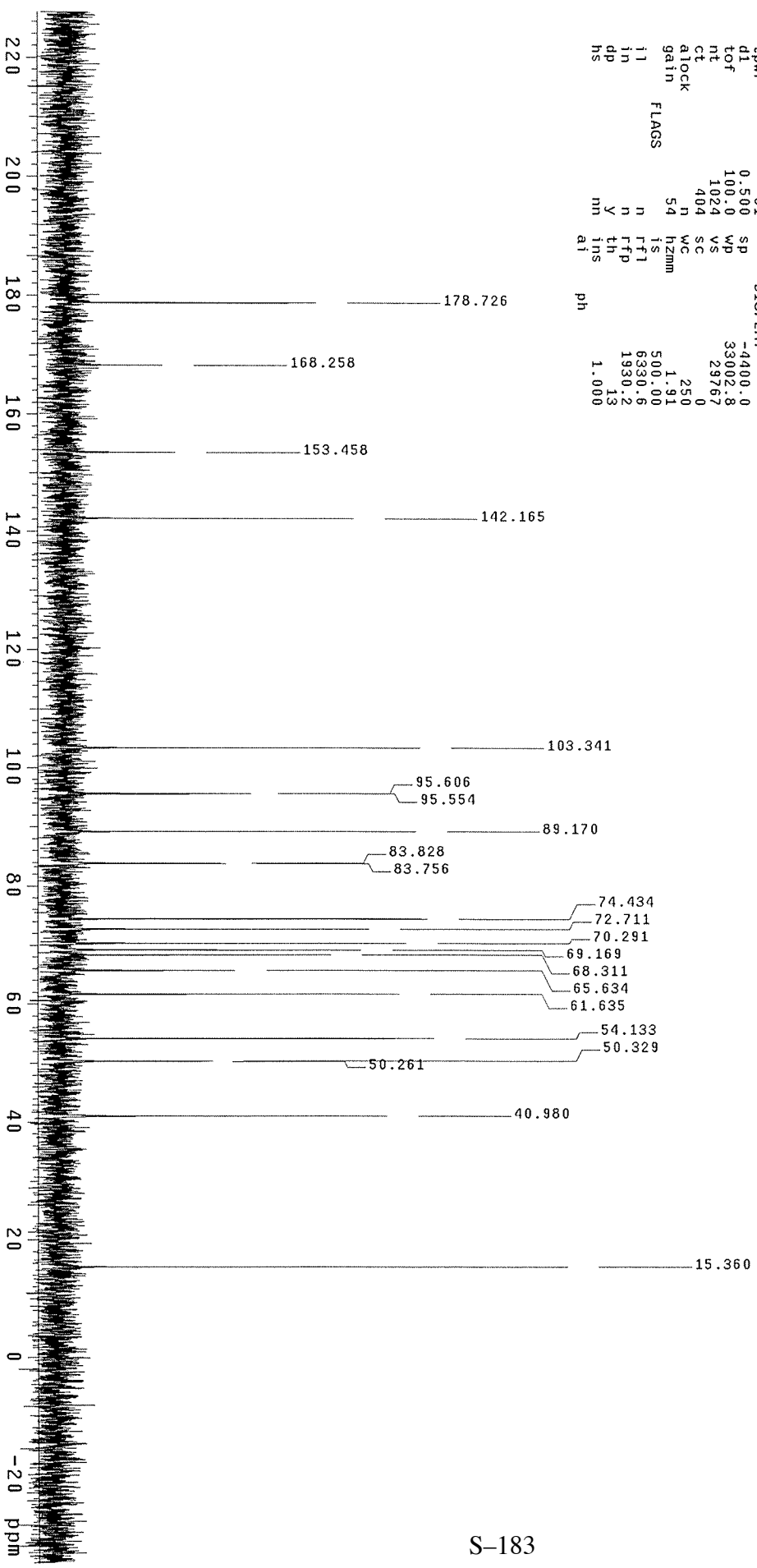
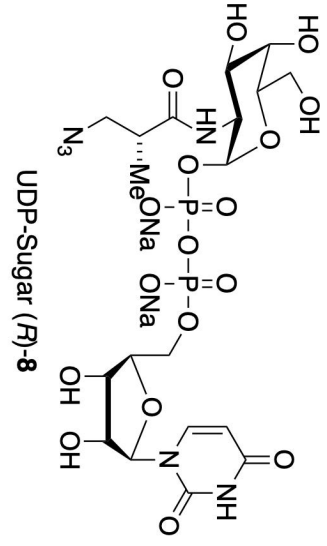
wnt wft



JC3083\_13C\_D20

exp2 s2pul1

SAMPLE	date	Jul 8 2017	dn	DEC. & VT	H1
solvent	D2O	dof	-499.0		
file	/export/home/~	yyy			
Junwecho1	JC3083_13	w			
	C_D20.f1d	dmm	11400		
ACQUISITION		dmf	43		
sfrq	125.674	dpwr		PROCESSING	2.00
tn	C13	lb	1b	fn	not used
at	1.500	math			
mp	99016				
sw	33003.3	weft			
fd	18000	wexp			
bs	7.0	wds			
pw	7.0	wnt			
tpwr	51	sp	-4400.0		
d1	0.500	wp	33002.8		
tof	100.0	vs	29767.0		
nt	1024	sc	250		
ct	404	wc	1.91		
atlock	n	hzm	500.00		
gain	54	ts	6330.6		
fl	n	rfl	1930.2		
in	n	th	13		
dp	y	ins	1.000		
hs	mn	ai			



JC3083\_31P\_D20

exp2 s2pu1

SAMPLE

DEC. & VT

date Jul 8 2017 dfrq 499.752

solvent D2O dn H1

file exp dpwr 43

ACQUISITION dof 0

sfrq 202.247 dm YYY

tn P31 dmm W

at 1.002 dmf 11400

np 160254 dseq

sw 80000.0 dres 1.0

fb 44000 homo n

bs 4 dfrq2 DEC2 0

tbwr 51 dn2 0

pw 6.6 dpwr2 1

d1 2.000 dof2 0

tof 0 dmm2 n

nt 32 dmm2 n

ct 20 dmf2 C

atlock n dseq2 9900

gain 30 dres2 1.0

flags n homo2 n

in n PROCESSING 2.00

in y lb wfile

dp nm wfile

hs y lb wfile

sp DISPLAY -45120.8 ft

wp 79999.4 fn not used

vs 10399 math

sc 0 weff

wc 250 wexp

hzm 3.81 wbs

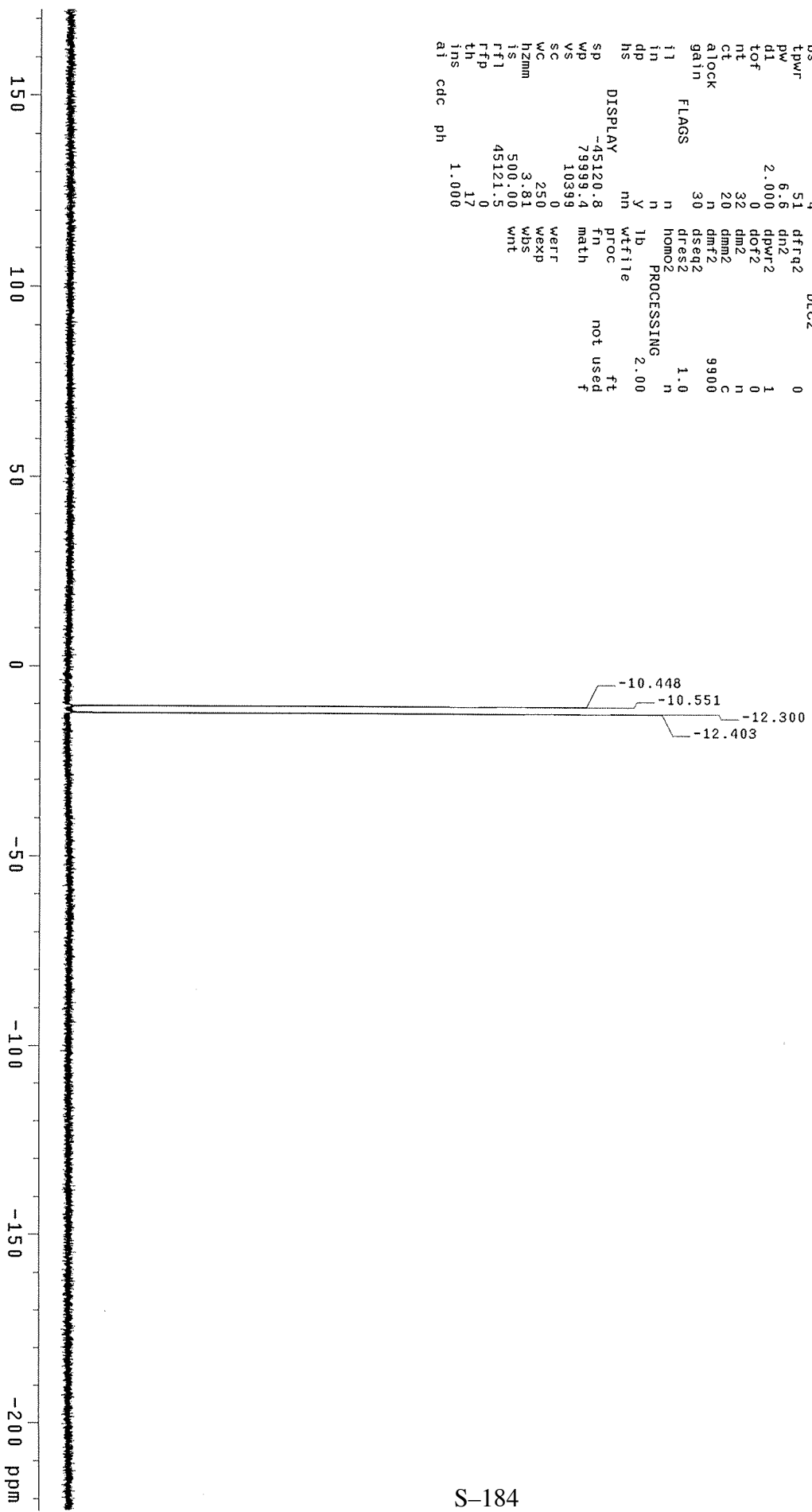
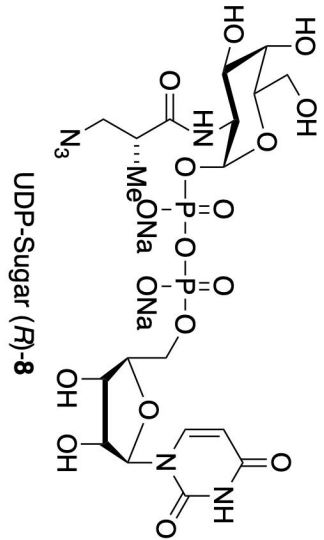
is 500.00 wnt

ftl 45121.5

ftf 0

th 17

ai cdc ph 1.000

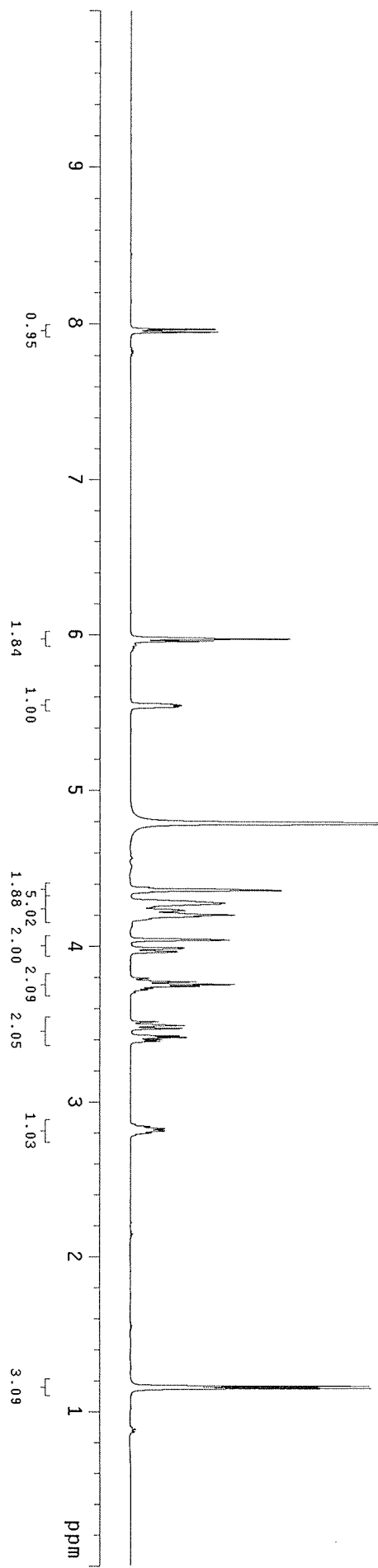
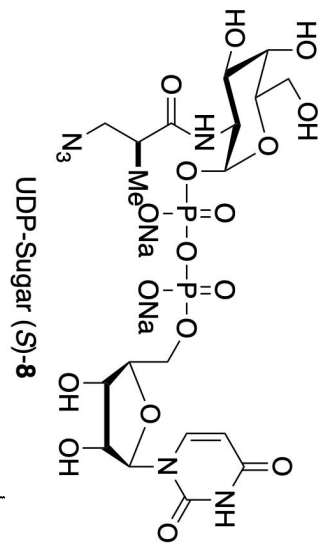




JC41007\_1H\_D20

exp2 s2pu1

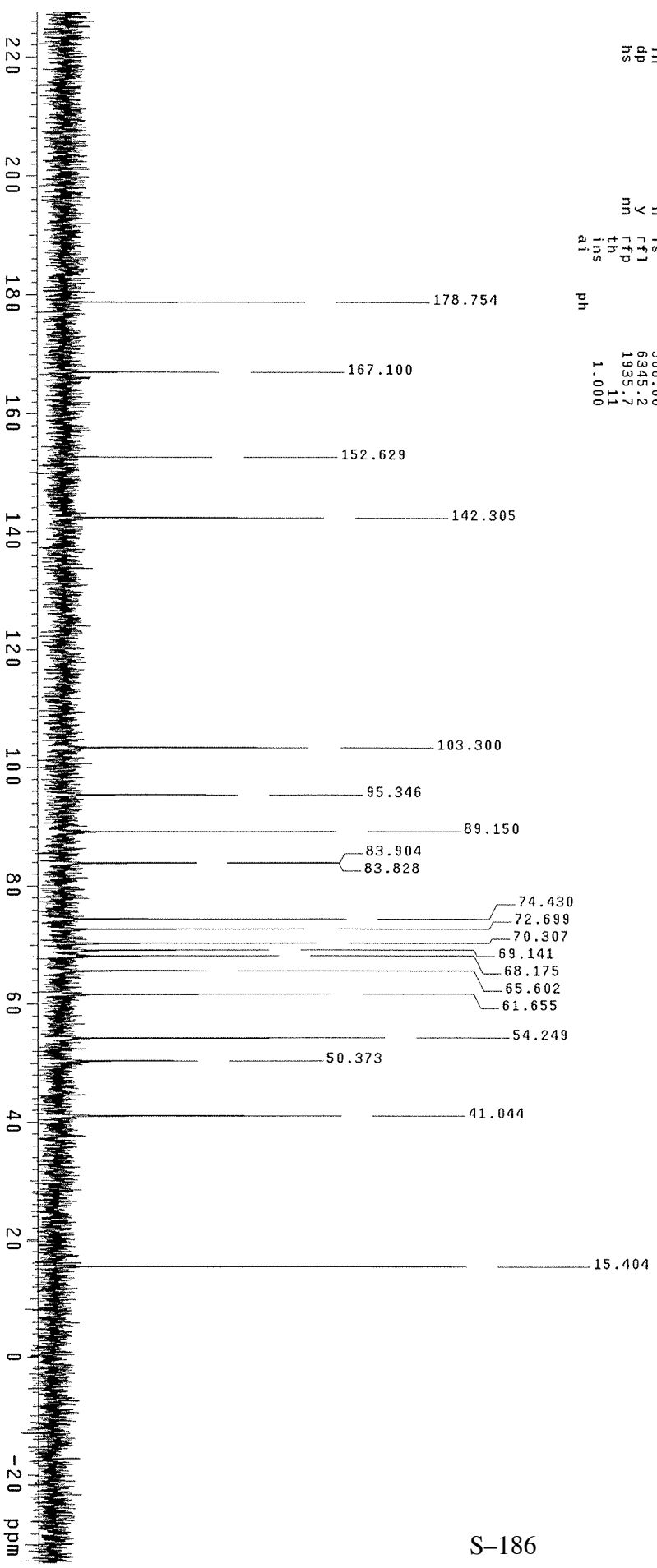
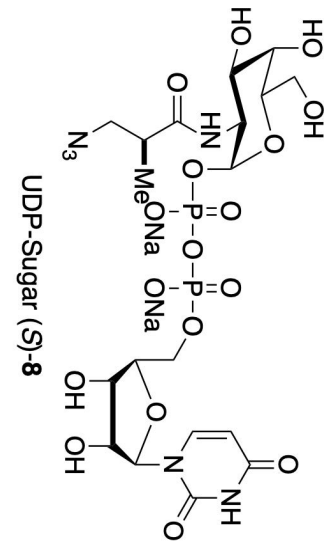
SAMPLE DEC. & VT  
 date Jul 24 2017 dfrq 499.752  
 solvent D2O dn H1  
 file ACQUISITION exp dpwr 30  
 sfrq 499.752 dm 0  
 tn H1 dmm mm  
 at 4.000 dmf C  
 np 64000 dseq 200  
 sw 8000.0 dres 1.0  
 fb 4000 homo n  
 bs 4  
 tprw 60 dfrq2 DEC2 0  
 pw 8.0 dn2  
 dl 0 dpwr2 1  
 tof 0 dof2 0  
 nt 64 dmm2 n  
 ct 64 dmf2 C  
 atock n  
 gain 40 dres2 200  
 flags  
 il n homo2 1.0  
 in n  
 dn y wfile PROCESSING  
 hs nm wfile  
 DISPLAY -0.2 ft  
 sp 4997.3 fn 65536  
 wd 231 weff  
 vs 230 wexp  
 sc WDS  
 WC 250 wnt  
 hzmm  
 is 19.99  
 ffl 33.57  
 rfp 3835.4  
 th 2393.8  
 ins 7  
 nm cdc ph 1.000



JC41007\_13C\_D20

exp3 s2pu1

SAMPLE	date	Jul 24 2017	dn	H1
solvent	D2O	dm	do	-499.0
file	exp	dm	dof	yyy
ACQUISITION	exp	dmm	ww	w
sfrq	129.674	dmt	ww	11400
tn	C13	dpwr	math	43
at	1.500	fn	not used	f
np	99016	fn	not used	f
sw	33003.3	fn	not used	f
fb	18000	math		
bs	4	weff		
pw	7.0	wexp		
tpwr	7.0	wbs		
di	51	wnt		
tof	1.000	sp	DISPLAY	
nt	2048	wp	-4409.0	
ct	720	vs	33002.8	
atlock	n	sc	33222.0	
gain	54	wc	250	
FLAGS		hzm	4.29	
il	n	ts	500.00	
in	n	rfl	6345.2	
dp	y	rffp	1935.7	
hs	mn	th	11	
ai	ins	ph	1.000	

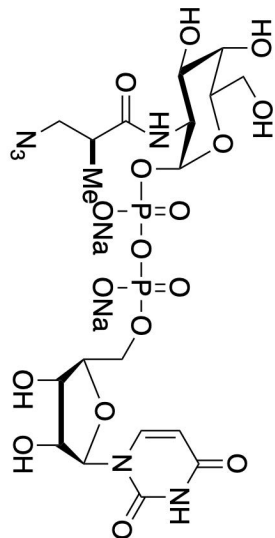


JC4007\_31P\_D20

exp3 s2pu1

SAMPLE DEC. & VT  
 date Jun 24 2017 dfrq 499.752  
 solvent D2O dn H1  
 file /export/home/~ junwchoi/JC4007\_31~ dpwr 43  
 P\_D20.F1d dm 0  
 yyy  
 W

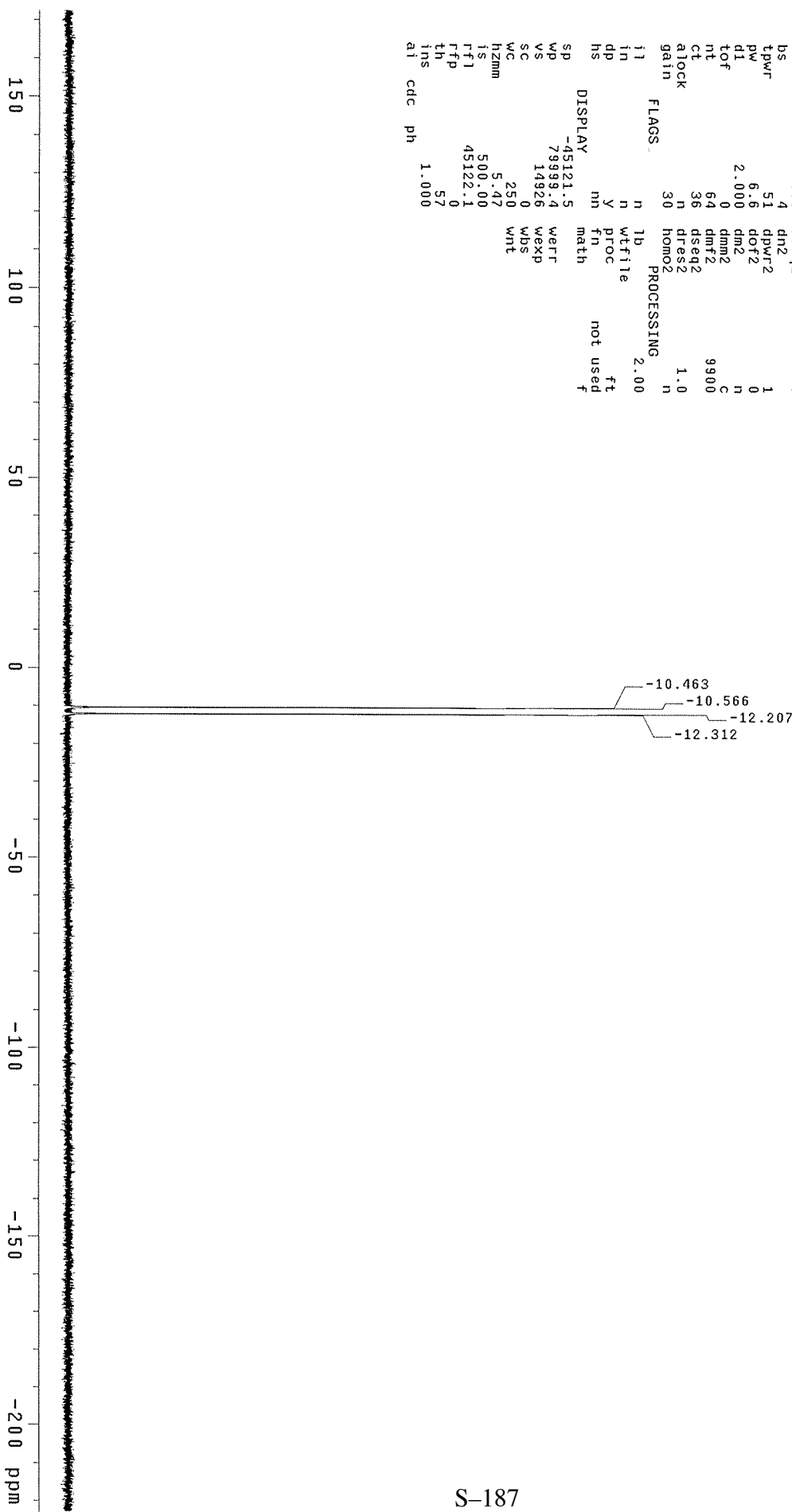
UDP-Sugar (S)-8



ACQUISITION  
 sfrq 202.297 dmf 11400  
 tn P31 dseq  
 at 1.002 dres 1.0  
 np 160254 homo n  
 sw 80000.0  
 fb 44000 dfrq2 DEC2 0  
 bs 4 dn2  
 tpwr 51 dpwr2 1  
 pw 6.6 dof2 0  
 dl 2.000 dm2 n  
 tof 0 dmm2 n  
 nt 64 dmf2 c  
 ct 36 dseq2 9900  
 atock n dres2 1.0  
 gain 30 homo2 n

FLAGS: n lb 1b wfile 2.00  
 i1 n wfile  
 in y proc ft  
 dp y math not used f  
 hs nm

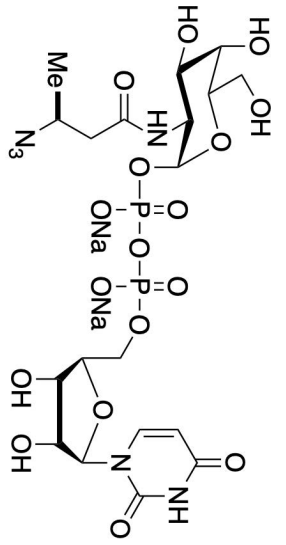
DISPLAY  
 sp -45121.5 weff  
 wd 79999.4 wexp  
 vs 14926 wds  
 sc 0 wnt  
 wc 250  
 hzmm 5.47  
 ts 500.00  
 rfl 45122.1  
 rfp 0  
 th 57  
 ins 1.000  
 al cdc ph



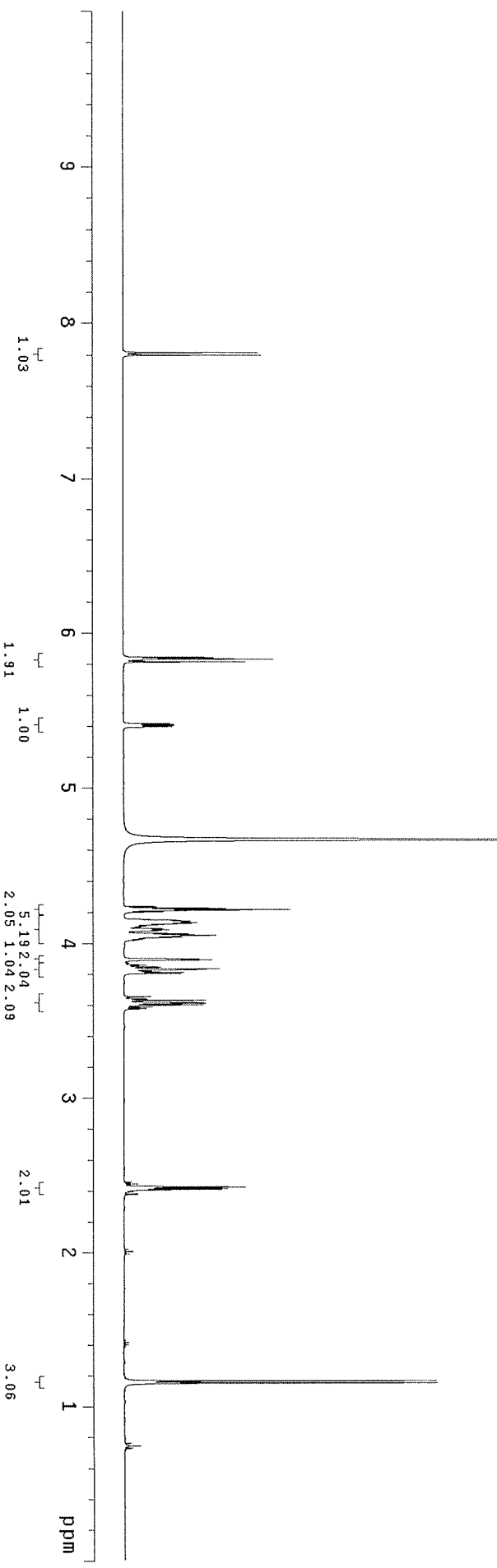
UDP-GA1NACCHMEN3(S)\_1H\_D20

exp3 s2pu1

SAMPLE	DATE	2017	DEC. & VT	499.752
SOLVENT	D2O		H1	30
FILE	EXP	DPWR	DOF	0
ACQUISITION	499.752	DM	MMN	0
SFRQ	H1	DM	MMN	0
IN	4.000	DMF	MMN	200
AT	64000	DSEQ		
MP	8000.0	DRES		1.0
SW	4000	HOMO		n
FB	4	DEC2		
BS	60	DFRQ2		0
TPWR	8.0	DM2		1
PW	0	DPWR2		0
DI	0	DOF2		0
TOF	64	DM2		n
NT	0	DMM2		n
CT	0	DMF2		c
ATOCK	40	DSEQ2		200
GAIN	40	DRES2		1.0
FLAGS		HOMO2		n
I1	n	PROCESsing		
IN	y	Wtfile		ft
DP	nm	PROC		fn
HS	DISP	MATH		65536
SP	-0.2			f
WD	4997.3	WEFT		
VS	236	WEXP		
SC	0	WBS		
WC	250	WNT		wft
HZMM	19.99			
IS	33.57			
FFI	1501.1			
FFP	0			
TH	7			
INS	1.000			
MM				
CD				
PH				



UDP-Sugar 9



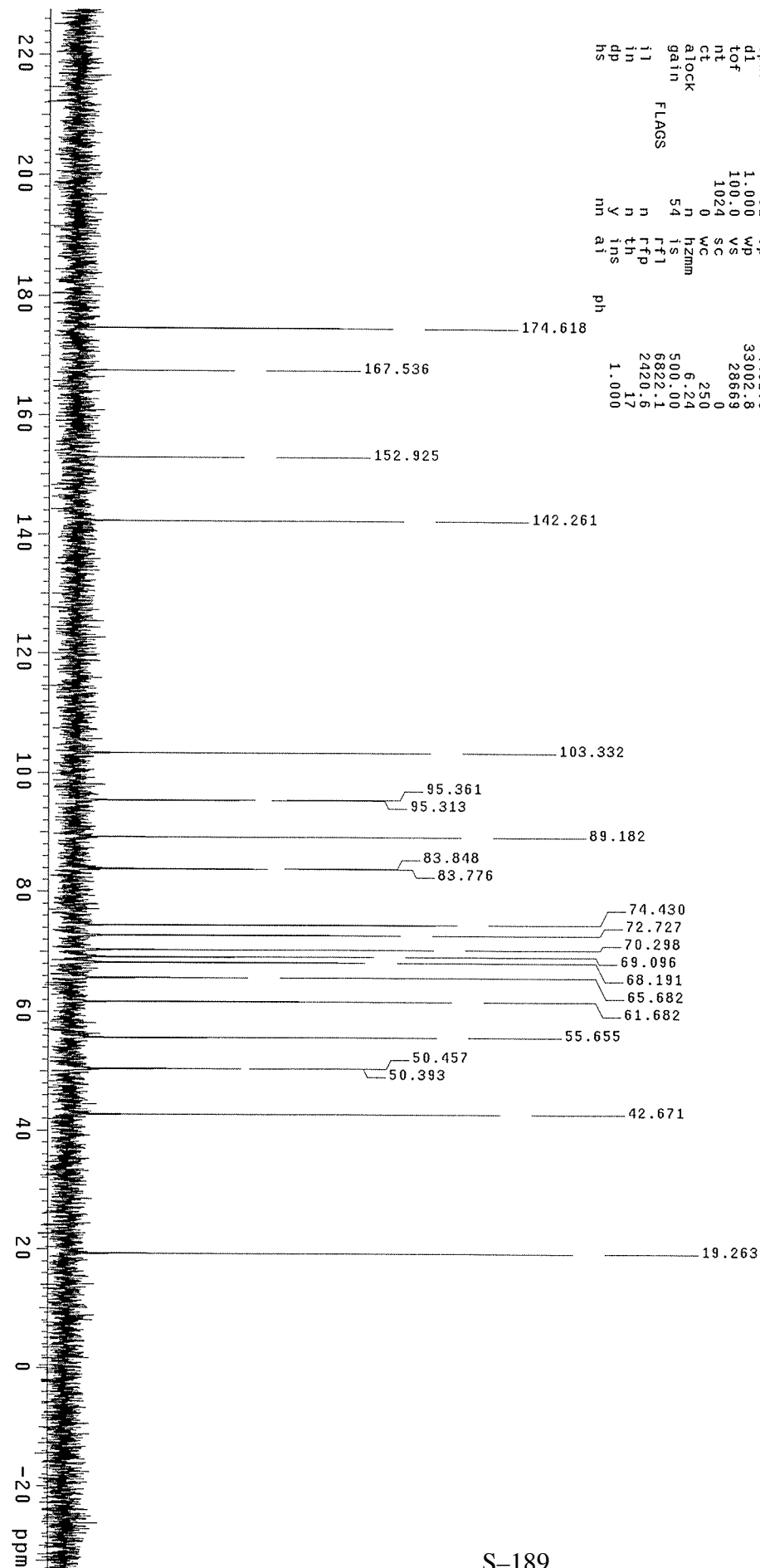
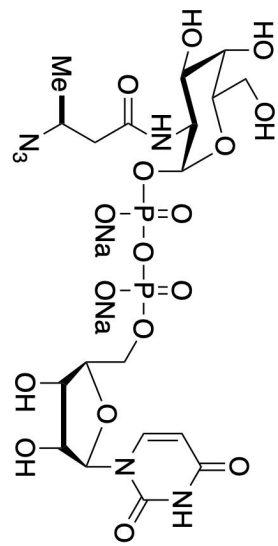
UDP-Ga1NACCHMEN3(S)\_13C\_D20

exp3 s2pu1

SAMPLE DEC. & VT  
 date Jul 13 2017 dn  
 solvent D2O dof -499.0  
 file /export/home/~ junwchoi/UDP-Ga1NA~ dm  
 CCHMEN3\_S\_13C\_D20~ dmf 11400  
 ACQUISITION f1d dpwr 43

PROCESSING 2.00  
 sfrq 125.674 lb  
 tn C13 fn not used f  
 at 1.500 math  
 mp 99016  
 sw 33003.3 weff  
 fd 18000 wexp  
 bs 4 wbs  
 pw 7.0 wnt  
 tpwr 51 sp  
 dl 1.000 wp -4401.0  
 tof 100.0 vs 33002.8  
 nt 1024 sc 28669  
 ct 0 wc 0  
 atock p hzmm 6.24  
 gain 54 ls 500.00  
 i1 n rfp 6822.1  
 in n th 2420.6  
 dp y ins 17  
 hs mn ai 1.000

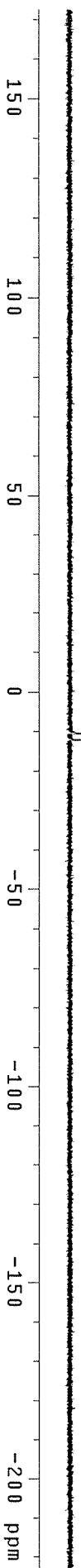
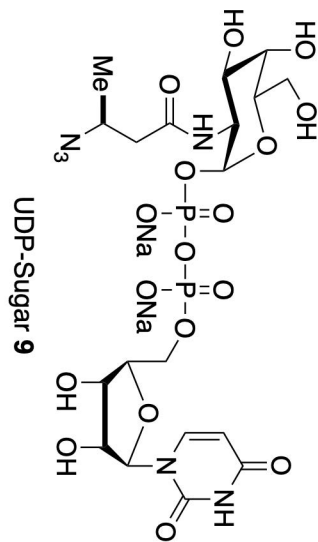
DISPLAY  
 -4401.0  
 33002.8  
 28669  
 0  
 250  
 500.00  
 6822.1  
 2420.6  
 17  
 1.000



UDP-GalNAc6Hmen3\_S-31P\_D20

exp2 s2pul

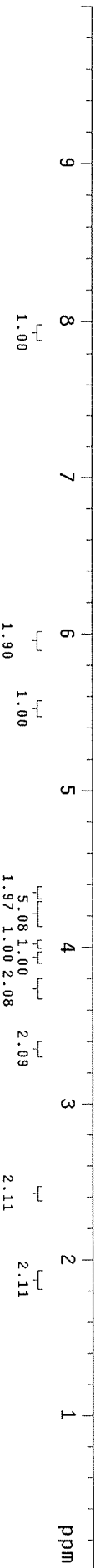
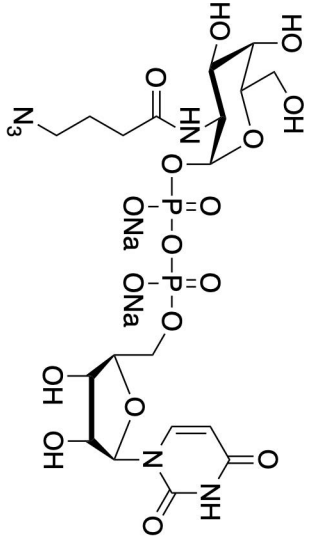
SAMPLE		DEC. & VT	
date	Jul 13 2017	dfrrq	499.752
solvent	D2O	dn	H1
file	exp	dpwr	43
ACQUISITION	exp	dof	0
sfrq	202.297	dm	yyy
tn	P31	dmm	w
at	1.002	dmf	11400
np	160254	dseq	
sw	80000.0	dres	1.0
fb	44000	homo	n
bs	4		
tpwr	51	dfrrq2	DEC2
pw	6.6	dn2	0
dl	2.000	dpwr2	1
tof	0	dof2	0
nt	32	dmm2	n
ct	0	dmf2	C
atock	n	dseq2	9900
gain	30	dres2	1.0
flags	n	homo2	n
fl	n	PROCESsing	2.00
in	y	1b	wtfile
dp	nm	proc	ft
hs	DISPLAY	fn	not used
SP	-45124.5	math	f
WD	79999.4	werr	
VS	7839	wexp	
SC	7839	wbs	
WC	250	wnt	
hzm	2.99		
ls	500.00		
ffl	45125.1		
ffp	0		
th	17		
ins	1.000		
aj	cdc		
ph			



JC3151\_1H\_D20

exp2 s2pu1

date	JUL 19 2017	DEC. & VT	499.752
solvent	D2O	dn	H1
file		dpwr	30
file		dof	0
file		dm	nmh
sfreq	499.752	dm	nmh
tn	H1	dmm	C
at	4.000	dmf	200
np	64000	dseq	
sw	8000.0	dres	1.0
fb	4000	homo	n
bs	4	homo	n
tpwr	60	dfreq2	DEC2
pw	8.0	dn2	0
d1	0	dpwr2	
tof	0	dof2	1
nt	128	dm2	0
ct	116	dmm2	n
alock	n	dmf2	C
gain	40	dseq2	200
flags		dres2	1.0
il	n	homo2	n
in	n	homo2	n
dp	Y	wtfile	ft
hs	mn	proc	fn
hs	mn	math	65536
hs	mn	math	f
sp	DISP	math	f
wp	-0.2	math	f
vs	4997.3	weff	
vs	234	wexp	
sc	250	wbs	
wc	0	wnt	
h2mm	19.99	wnt	wft
ts	33.57		
rfl	3835.9		
th	2393.8		
ins	1.000		
nm	cdc		
nm	ph		



JC3151\_13C\_D2O

exp3 s2pul

SAMPLE DEC. & VT

date Jul 19 2017 dn H1

solvent D2O dof -499.0

file /export/home/~ Junwchoi/JC3151\_13~ dmm yyy

Junwchoi/JC3151\_13~ dmm yyy

C\_D2O\_T1d dmt 11400

ACQUISITION 125.674 dpwr 43

sfrq 125.674 PROCESSING 43

tn C13 1b 2.00

at 1.500 fn not used f

np 99016 math

sw 33003.3

fb 18000 weff

bs 4 wexp

pw 7.0 wds

tpwr 51 wnt

d1 1.000 sp DISPLAY

tof 100.0 wp -4397.9

nt 2048 vs 33002.8

ct 572 SC 35154

atlock 0

gain 54 n WC 250

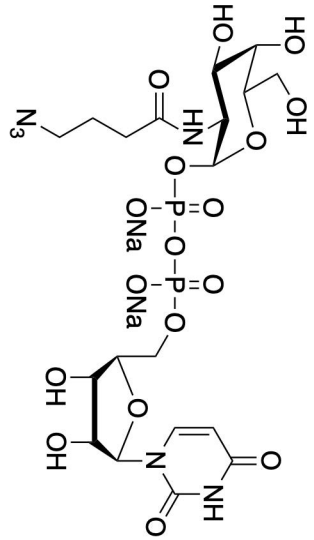
l1 n hzmm 509.00

in n rffl 7565.9

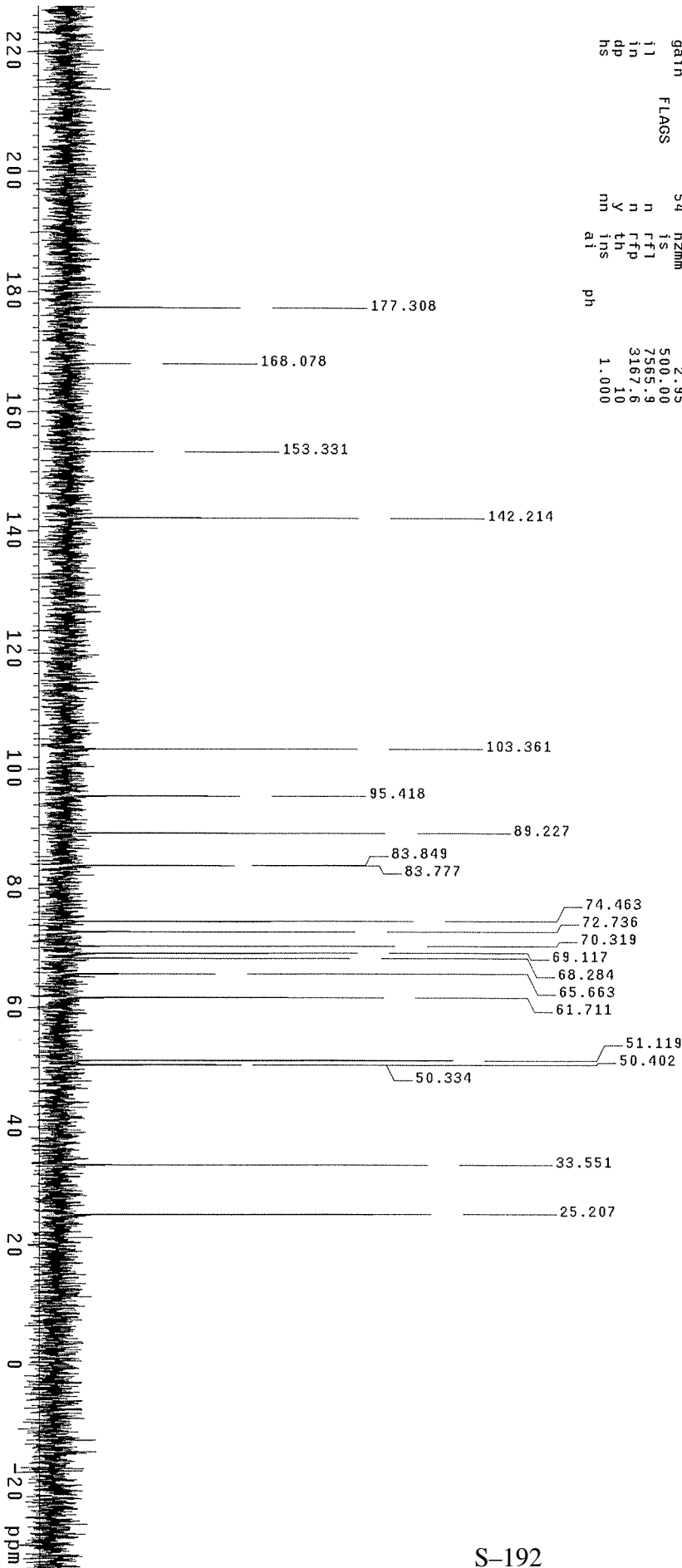
dp y rffl 3167.6

hs mn ins 10

ph 1.000



UDP-Sugar 10

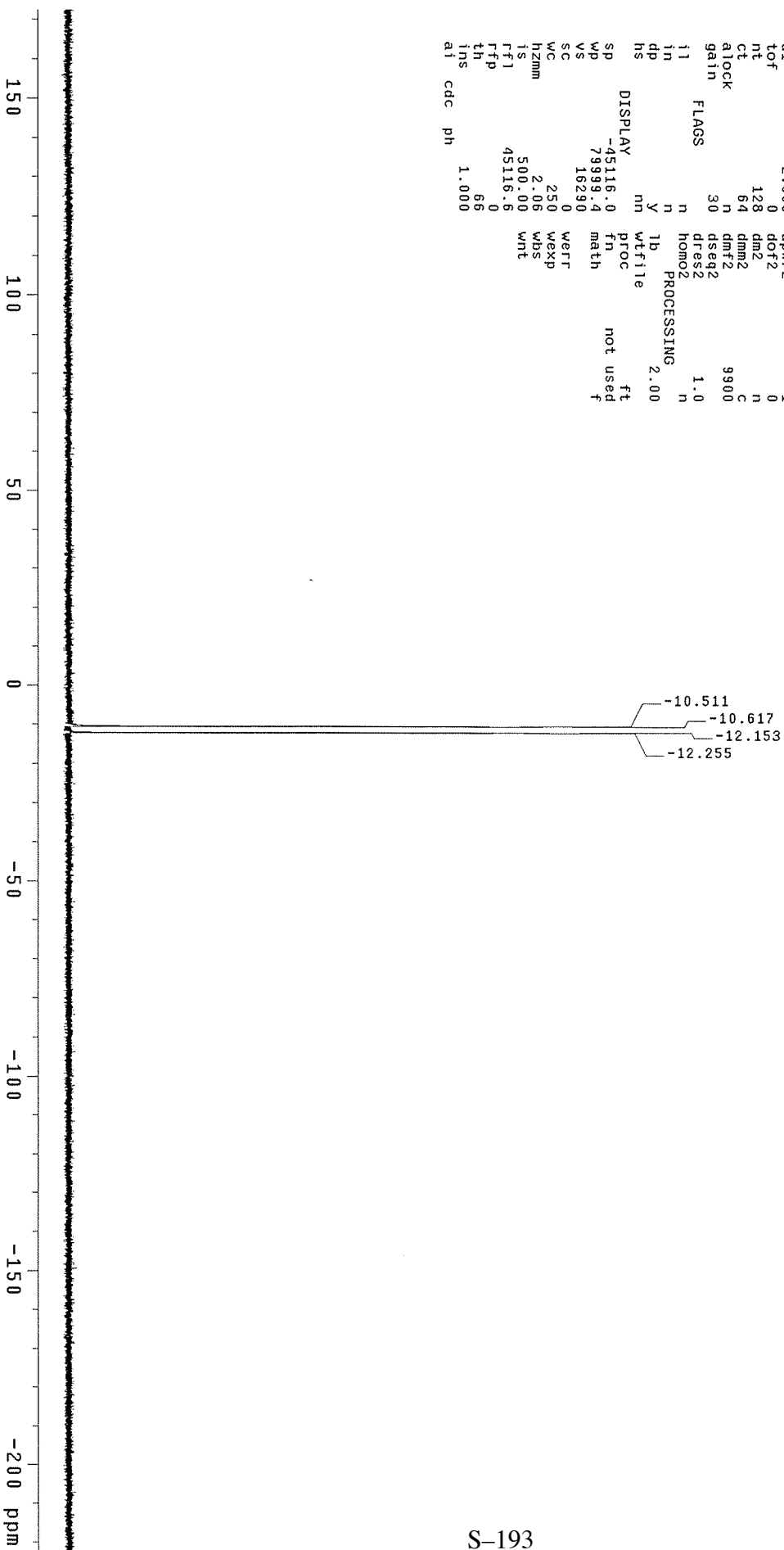
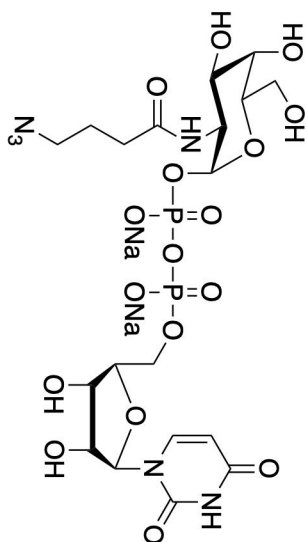




JC3151\_31P\_D20

exp2 s2pul1

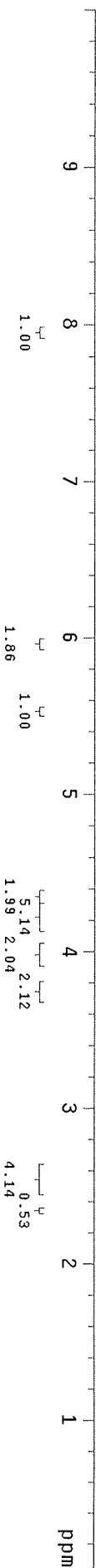
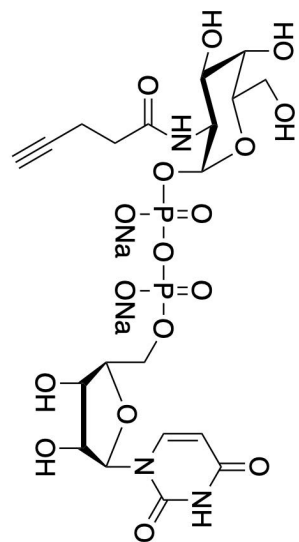
SAMPLE DEC. & VT  
date Jul 19 2017 dfrq 499.752  
solvent D2O dn H1  
file exp dpwr 43  
ACQUISITION 202.287 dof 0  
sfrq 202.287 dm yyy  
tn P31 dmm W  
at 1.002 dmf 11400  
np 160254 dseq  
sw 80000.0 dres 1.0  
fb 44000 homo n  
bs 4  
tpwr 51 dfrq2 DEC2 0  
pw 6.6 dn2  
dl 2.000 dpwr2 1  
tof 0 dot2 0  
nt 128 dm2 n  
ct 64 dmm2 C  
atock n dmf2 9900  
gain 30 dseq2  
FLAGS n dres2 1.0  
i1 n homo2  
i1 n PROCESSING 2.00  
in y lb  
dp nm wtfile  
hs DISPLAY ft  
SP -45116.0 fn not used  
WD 79999.4 math f  
VS 16290 werr  
WC 250 wexp  
SC 250 wbs  
h2mm 500.00 wnt  
f1 45116.6  
f1p 0  
th 66  
ins 1.000  
ai cdc ph



JCS061\_1H\_D20

expt1 s2pu1

SAMPLE	date	Jul 28 2017	DEC. & VT	499.752
SOLVENT	D2O	exp	H1	30
FILE	ACQUISITION	499.752	dm	mm
sfreq	499.752	H1	dmf	C
tn	4.000	dmf	dseq	200
at	64000	dseq	dres	1.0
np	8000.0	dres	homo	n
sw	4000	homo	dfreq2	0
fb	4000	homo	dn2	0
bs	4	dfreq2	dpwr2	1
tpwr	60	dn2	dof2	0
pv	8.0	dpwr2	dm2	n
d1	0	dof2	dmm2	n
tof	0	dm2	dmf2	C
nt	1024	dmm2	dseq2	200
ct	52	dmf2	homo2	1.0
atock	n	dseq2	PROCESsing	n
gain	40	homo2	wtfile	ft
il	n	PROCESsing	nm	65536
in	n	wtfile	fn	f
dp	Y	nm	math	
hs	DISP	math		
sp	WP	4997.3	werr	
wd	VS	261	wexp	
sc	WC	250	wds	
h2mm	19.99	wnt		
is	33.57			
ff1	3835.9			
ffp	2393.8			
th	7			
ins	1.000			
nm	cdc	ph		



JC5061\_13C\_D20

exp3 s2pu1

DATE: Jul 12 2017

SAMPLE: D20

SOLVENT: /export/home/~

FILE: JC5061\_13~

INSTR: C D20

PROBHD: 5mm

PROC: 43

ACQUISITION: 125.674

INSTR: C13

NUC1: 13

NUC2: 13

NUC3: 13

NUC4: 13

NUC5: 13

NUC6: 13

NUC7: 13

NUC8: 13

NUC9: 13

NUC10: 13

NUC11: 13

NUC12: 13

NUC13: 13

NUC14: 13

NUC15: 13

NUC16: 13

NUC17: 13

NUC18: 13

NUC19: 13

NUC20: 13

NUC21: 13

NUC22: 13

NUC23: 13

NUC24: 13

NUC25: 13

NUC26: 13

NUC27: 13

NUC28: 13

NUC29: 13

NUC30: 13

NUC31: 13

NUC32: 13

NUC33: 13

NUC34: 13

NUC35: 13

NUC36: 13

NUC37: 13

NUC38: 13

NUC39: 13

NUC40: 13

NUC41: 13

NUC42: 13

NUC43: 13

NUC44: 13

NUC45: 13

NUC46: 13

NUC47: 13

NUC48: 13

NUC49: 13

NUC50: 13

NUC51: 13

NUC52: 13

NUC53: 13

NUC54: 13

NUC55: 13

NUC56: 13

NUC57: 13

NUC58: 13

DEC. & VT

DN

DOF

DM

DMM

W

DMF

DPWR

PROCESsing

2.00

not used

f

math

99016

33003.3

18000

4

wexp

7.0

MBS

7.0

WNT

51

TPWR

0.500

SP

-4402.0

DIspLAY

33002.8

WP

100.0

VS

25076

SC

1024

VS

592

SC

54

WC

hzm

250

hzm

509.00

IS

509.00

IS

6285.8

IS

1883.3

IS

15

IS

1.000

IS

ph

ai

ns

th

ins

th

ins

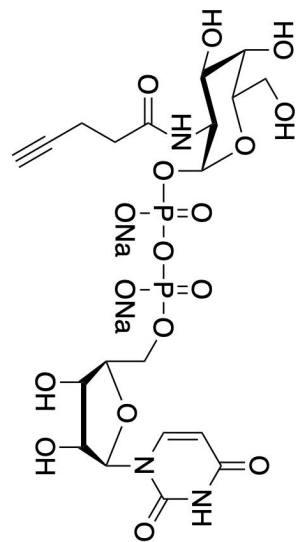
th

ins

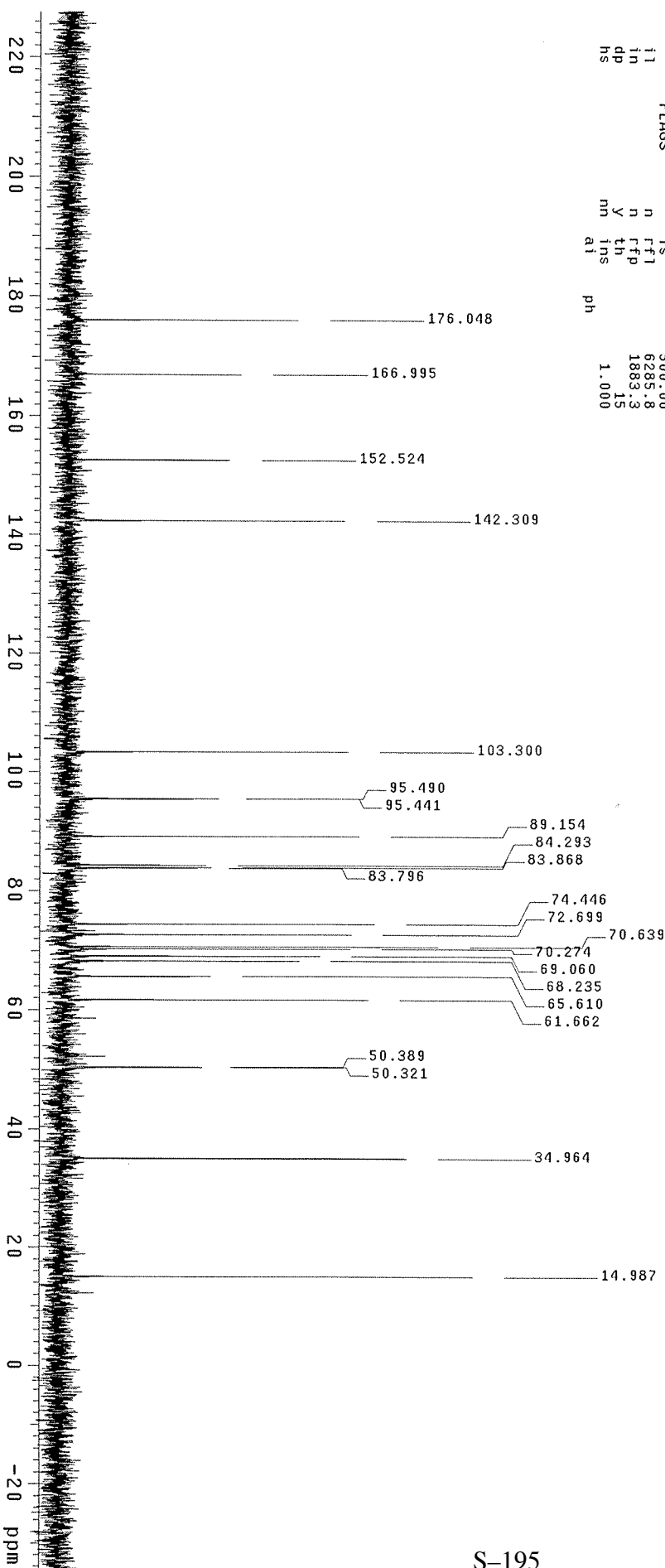
th

ins

th



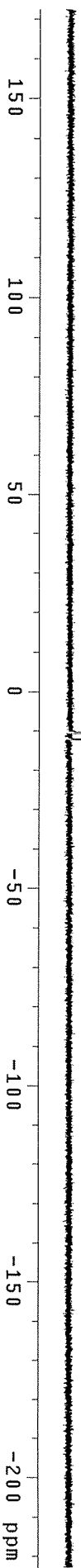
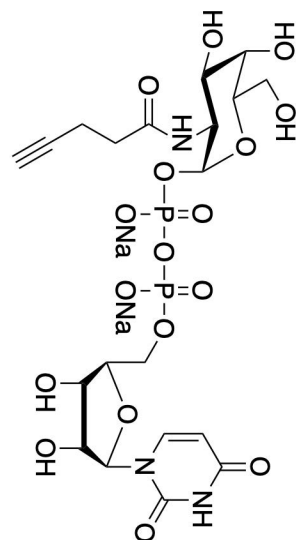
UDP-Sugar 11



JC5061\_31P\_D20

exp3 s2pu1

SAMPLE DEC. & VT  
date Jul 13 2017 dfrq 499.752  
solvent D2O dn H1  
file exp dpwr 43  
ACQUISITION exp dof 0  
sfrq 202.297 dm yyy  
tn 1.002 dmm W  
at 160254 dseq 11400  
np 80000.0 dres  
sw 44000 homo 1.0  
fb 44000  
bs 4  
tdwr 51 dfrq2 DEC2 0  
pw 6.6 dp2 1  
d1 2.000 dpwr2 1  
tof 0 dof2 0  
nt 32 dm2 n  
ct 0 dmm2 C  
atlock gain 30 dmf2 9900  
gain 30 dseq2  
dres2  
i1 n homos 1.0  
in n PROCESSING 2.00  
dp y lb wfile  
hs n y wfile  
DISPLAY -45124.5 ft  
WD 79999.4 fn not used  
VS 78339 math f  
WC 0 weff  
hZmm 250 weXP  
IS 500.00 WDS  
rfl 45125.1 Wt  
th 0  
ins 17  
ai cdc ph 1.000

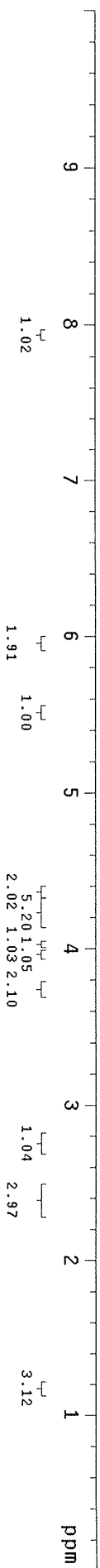
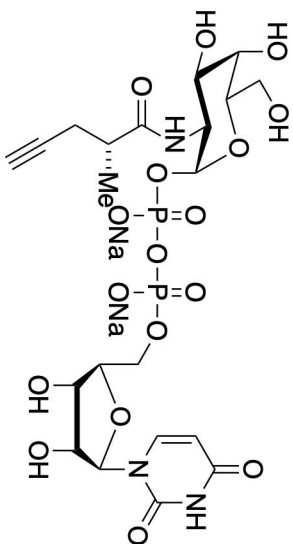


-10.508  
-10.611  
-12.210  
-12.312

JC3099\_1H\_D20

exp2 s2pu1

SAMPLE	DEC. & VT
date Jul 10 2017	499.752
solvent D2O	H1
file ACQUISITION	exp 30
sfreq 499.752	dm 0
in H1	dm 200
at 4.000	dmm
mp 64000	dseq
sw 8000.0	dres
fb 4000	homo
bs 4	DEC2
tpwr 60	dfreq 0
pw 8.0	dn2 1
d1 0	dpwr2 1
tof 0	dof2 0
nt 64	dm2 n
ct 24	dmm2 c
atock n	dmf2 200
gain 40	dseq2
flags	dres2
il n	homo2 1.0
in n	PROCESSING
dp y	wfille
hs nm	proc ft
DISPLAY	fn 65536
sp -0.2	math f
wp 4997.3	weff
vs 176	wexp
sc 0	wbs
wc 250	wnt
h2mm 19.99	wft
is 33.57	
rfl 3834.7	
rff 2393.8	
th 7	
ins 1.000	
nm cdc ph	



JC3098\_13C\_D20

exp2 s2pu1

SAMPLE DEC. & VT

date Jul 10 2017 dn H1

solvent D2O dof -499.0

file /export/home/~ dm YYY

Junwcho1/JC3098\_13~ dmm W

C\_D20.f1d dmf 11400

ACQUISITION dpwr 43

sfrq 125.674 PROCESSING 2.00

tn C13 1b not used f

at 1.500 fn math

np 99016 math

sw 33003.3 weff

fd 18000 wbs

bs 4 wexp

pw 7.0 wnt

tpwr 51 DISPLAY

dl 0.500 sp -4403.0

tof 100.0 wp 33002.8

nt 1024 vs 24431

ct 272 SC 24431

atlock N WC 250

gain 54 hzmm 1.56

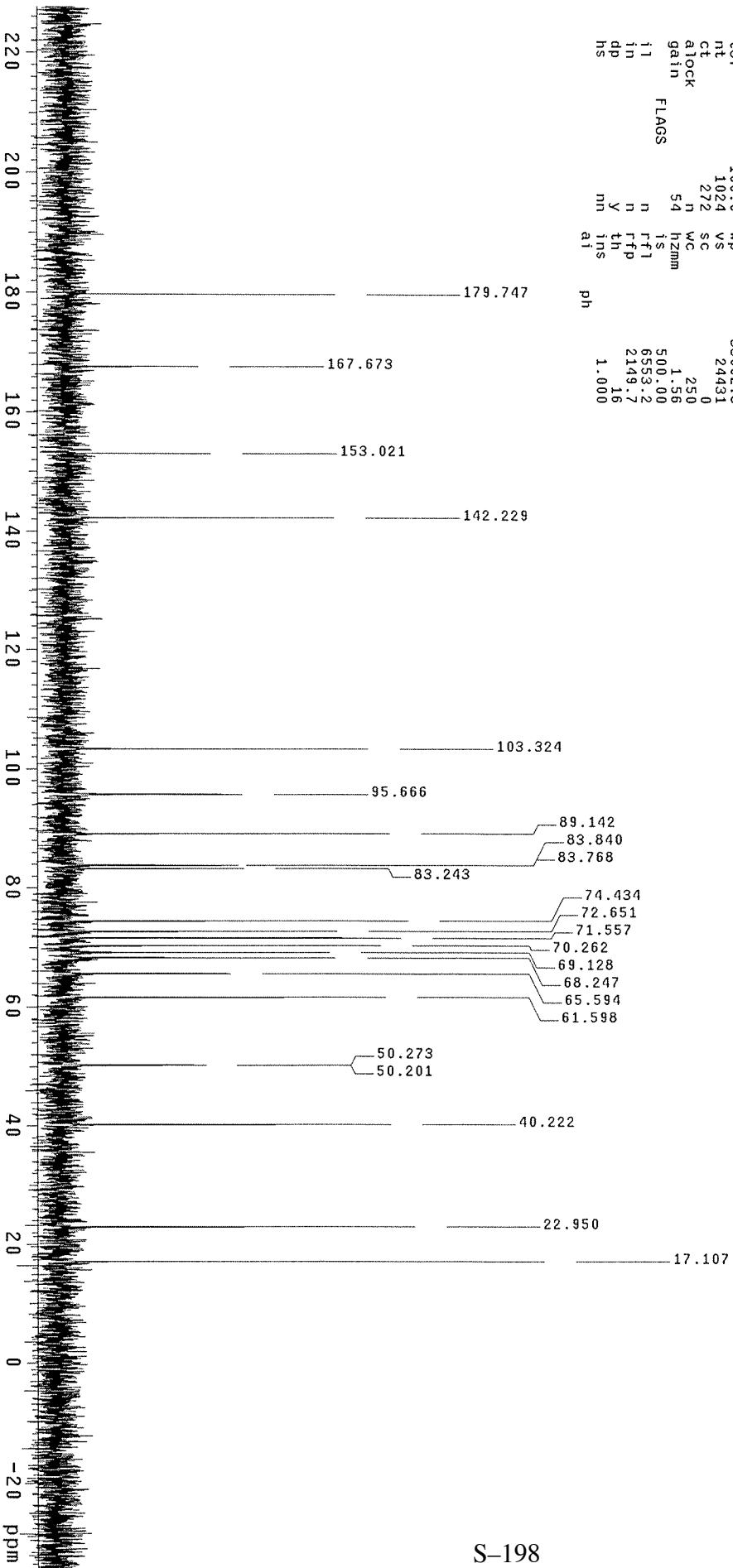
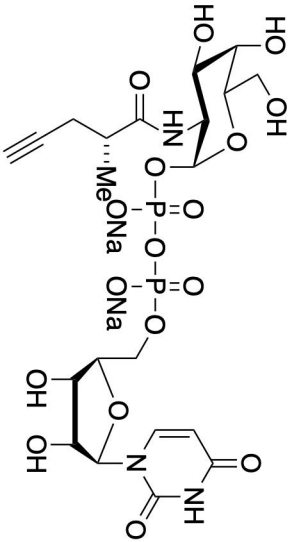
l1 n rffl 500.00

in n rfp 6553.2

dp Y th 2149.7

hs mn ins 16

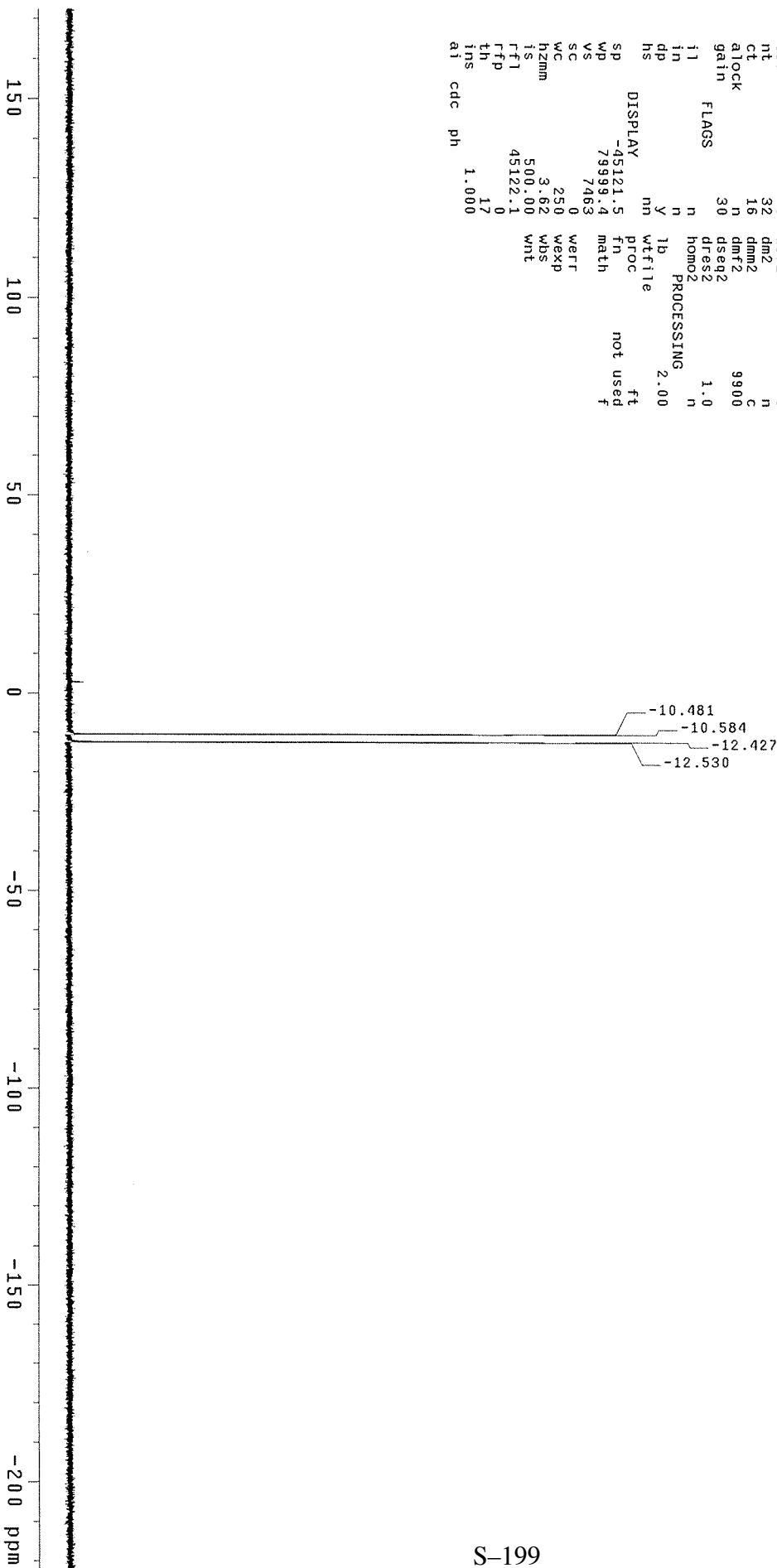
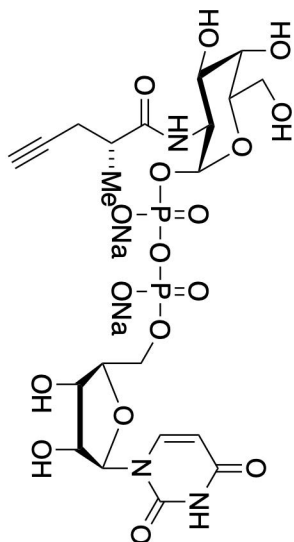
at ph 1.000



JC3099\_31P\_D20

exp2 s2pu1

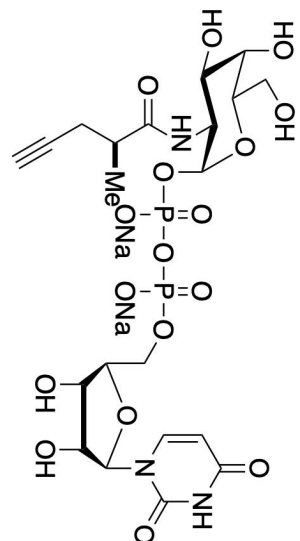
SAMPLE	date	Jul 10 2017	DEC. & VT	499.752
solvent	D2O		H1	43
file	ACQUISITION	exp	dpwr	0
sfrq	202.297	dm	dof	0
tn	P31	dmm	yyy	w
at	1.002	dmt	11400	w
np	160254	dseq		
sw	80000.0	dres		1.0
fb	44000	homo		n
bs	4	DECE		n
tpwr	51	dfrq2		0
pw	6.6	dn2		1
dl	2.000	dpwr2		0
tof	0	dof2		n
nt	32	dm2		n
ct	16	dmm2		c
atock	n	dmf2		9900
gain	30	dseq2		1.0
flags		dres2		n
il	n	homo2		n
in	n	PROCE		2.00
dp	y	1b		wtfile
hs	nm	proc		ft
DISP		fn		not used
sp	-45121.5	math		f
wd	79999.4	werr		
vs	7463	wexp		
sc	0	wbs		
wc	250	wnt		
h2mm	3.62			
is	500.00			
rfl	45122.1			
rfl	0			
th	17			
ins	1.000			
ai	cdc			
ph				



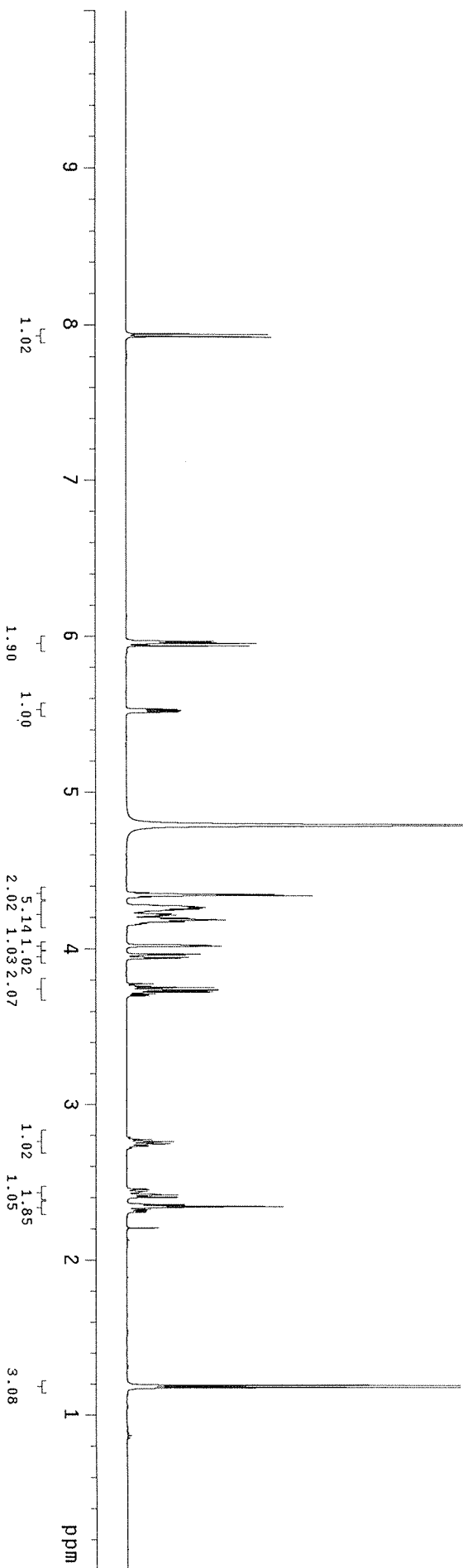
JC3101\_1H\_D20

exp2 s2pu1

SAMPLE	date	Jul 10 2017	DEC. & VT	499.752
SOLVENT	solvent	D2O	dn	H1
FILE	file	ACQUISITION	exp	30
DOF	DOF	0	dpwr	0
SFRQ	sfrq	499.752	dm	nmn
IN	in	H1	dmm	c
AT	at	4.000	dmf	200
NP	np	64000	dseq	
SW	sw	8000.0	dres	1.0
FB	fb	4000	homo	n
DS	ds	4	DEC2	0
TPWR	tpwr	60	dfrq2	0
PW	pw	8.0	dn2	1
D1	d1	0	dpwr2	1
TOF	tof	0	dof2	0
NT	nt	64	dm2	n
CT	ct	44	dmm2	c
ATOCK	atock	n	dmf2	200
GAIN	gain	40	dseq2	
FLAGS	flags		dres2	1.0
I1	i1	n	homo2	n
IN	in	n	PROCESsing	
DP	dp	nm	wtfile	ft
HS	hs	nm	PROC	65536
DISP	DISPLAY	-0.2	fn	f
SP	sp	4997.3	math	
WP	wp	286	WEFT	
VS	vs	0	WEXP	
SC	sc	250	WBS	
WC	wc	19.99	wnt	wft
HZMM	hzmm	33.57		
IS	is	3835.2		
RFL	rfl	2393.8		
TH	th	7		
INS	ins	1.000		
MM	mm			
CD	cdc	ph		



UDP-Sugar (S)-12





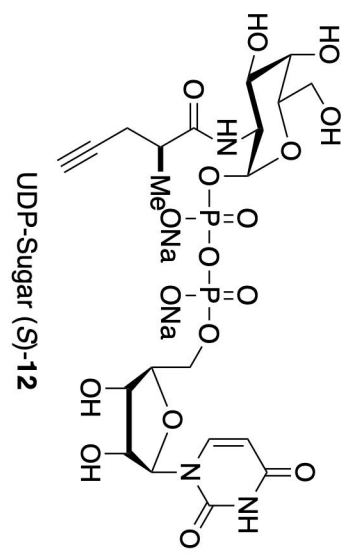
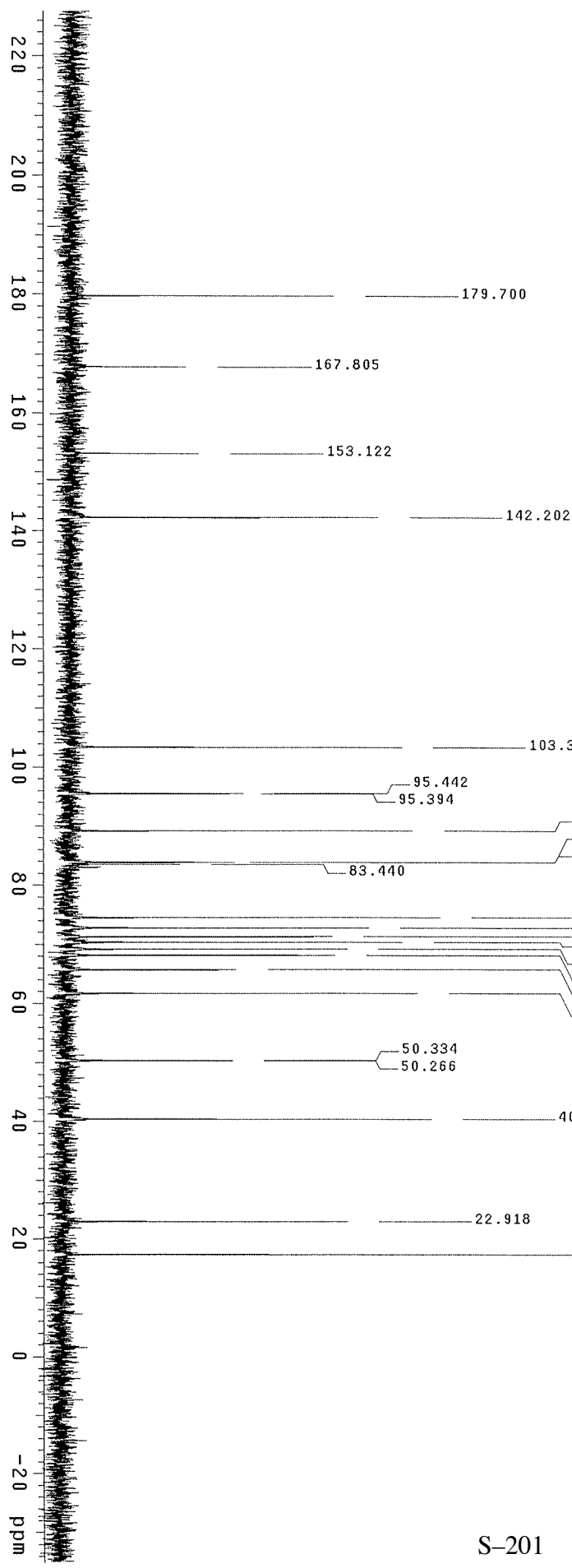
JG3101\_13C\_D20  
 exp2 s2pu1

SAMPLE date Jul 20 2017  
 solvent D2O  
 title ACQUISITION  
 sfrq 125.674  
 tn C13  
 at 1.500  
 np 99016  
 sw 33003.3  
 fd 18000  
 bs 4  
 pw 7.0  
 tpwr 7.0  
 dl 51  
 tof 1.000  
 nt 2000  
 ct 1152  
 atlock N  
 gain 54

DEC. & VT H1  
 dof -499.0  
 dm YYY  
 dmm W  
 dpwr 11400  
 PROCESSING 43  
 lb 2.00  
 fn not used  
 math f

DISPLAY  
 -4402.4  
 33002.8  
 31933  
 0  
 250  
 0.95  
 500.00  
 6578.9  
 2176.0  
 15  
 1.000

ph





JC5158\_1H\_D20

exp3 s2pu1

SAMPLE

date Aug 4 2017

solvent D2O

file exp

ACQUISITION

sfrq 499.752

tn H1

at 4.000

np 64000

sw 8000.0

fb 4000

bs 4

tbwr 60

d1 8.0

tof 0

nt 0

ct 512

alock 196

gain n

flags 40

i1 n

in n

dp y

hs n

DISPLAY

sp -0.2

wp 4997.3

vs 277

sc 0

wc 250

h2mm 19.98

ls 3857.35

ftl 3835.9

ffp 2393.8

lh 114

ins 1.000

nm cdc ph

DEC. & VT

dfreq 499.752

dn H1

dpwr 30

dof 0

dm nnn

dmm C

dntf 200

dresq 1.0

homo n

dfreq2 0

dn2 0

dpwr2 1

dof2 0

dm2 n

dmf2 C

dseq2 200

homo2 1.0

PROCESSING

wtfile ft

proc fn

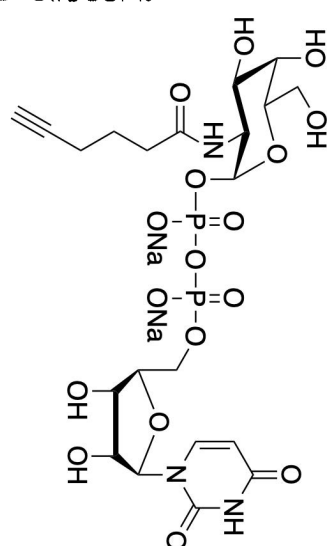
math 65536

weft f

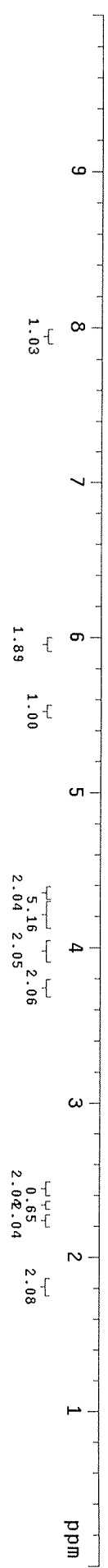
wexp

wbs

wnt wft



UDP-Sugar 13



JC5062\_13C\_D20

exp3 s2pul1

SAMPLE DEC. & VT

date Jul 12 2017 dn H1

solvent D2O dof -499.0

file /export/home/~ junwchoi/JC5062\_13~ dm yyy

Junwchoi/JC5062\_13~ dmm 11400

C D20 T1d dmf 43

ACQUISITION 125.674 dpwr 43

PROCCESSING 2.00

at 1.500 fn not used f

mp 99016 math

sw 33003.3

fb 18000 weff

bs 7.0 wds wexp

pw 7.0 Wnt

tipwr 51

dl 1.000 sp DISPLAY

nt 100.0 wp -4401.4

ct 1024 vs 33002.8

atlock n SC 50108

gain n WC 0

l1 54 hzmm 250

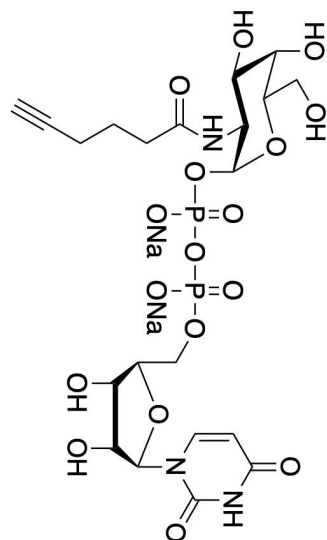
in n IS 8.15

dp n rffl 500.00

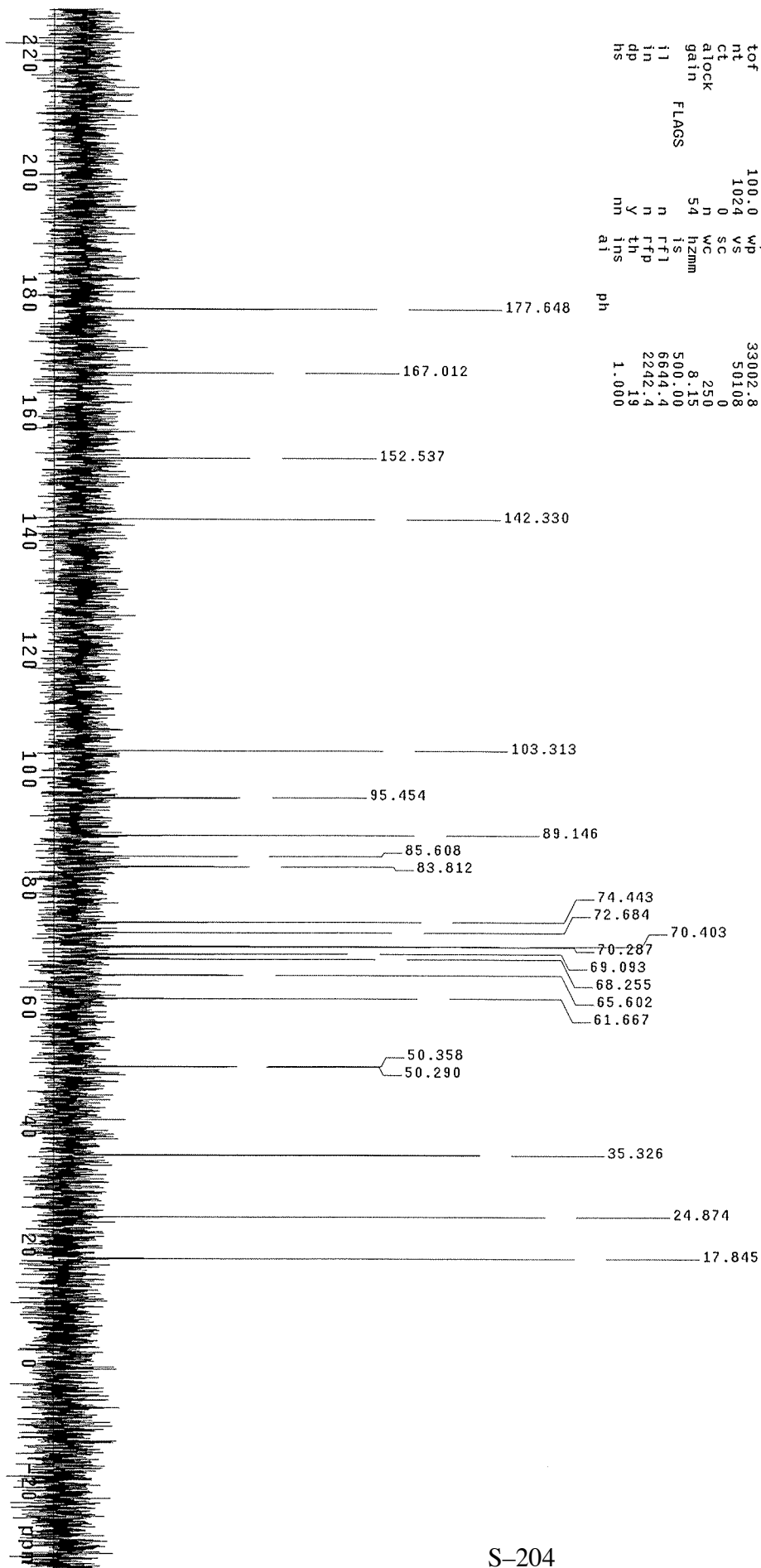
hs y th 6644.4

ai ins 2242.4

ph 1.000 19



UDP-Sugar 13





JC3103\_1H\_D20

exp2 s2pu1

SAMPLE DEC. & VT 499.752

date Jul 11 2017

solvent D2O

file exp

ACQUISITION

sfrq 499.752

tn H1

at 4.000

np 64000

sw 8000.0

fb 4000

bs 4

tdwr 60

pw 8.0

dl 0

tof 0

nt 32

ct 32

atlock n

gain 40

flags n

il n

in n

dp y

hs nm

DISPLAY

sp -0.2

wp 4997.3

vs 278

sc 0

wc 250

hZmm 19.99

is 33.57

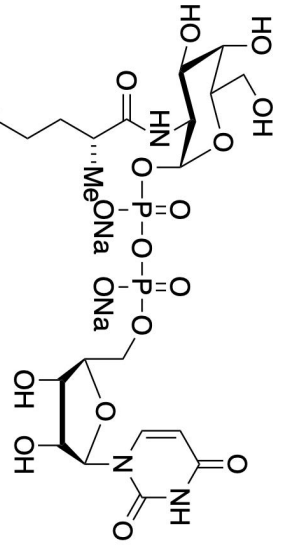
ftj 3834.7

ftf 2393.8

th 7

ins 1.000

mm cdc ph



UDP-Sugar (R)-14

DEC. & VT 499.752

ft H1

30

0

nmn

C

200

1.0

n

DEC2

0

dn2

1

dpwr2

1

dof2

n

dm2

n

dmm2

C

dmf2

200

dseq2

1.0

dres2

n

homoz

PROCESSING

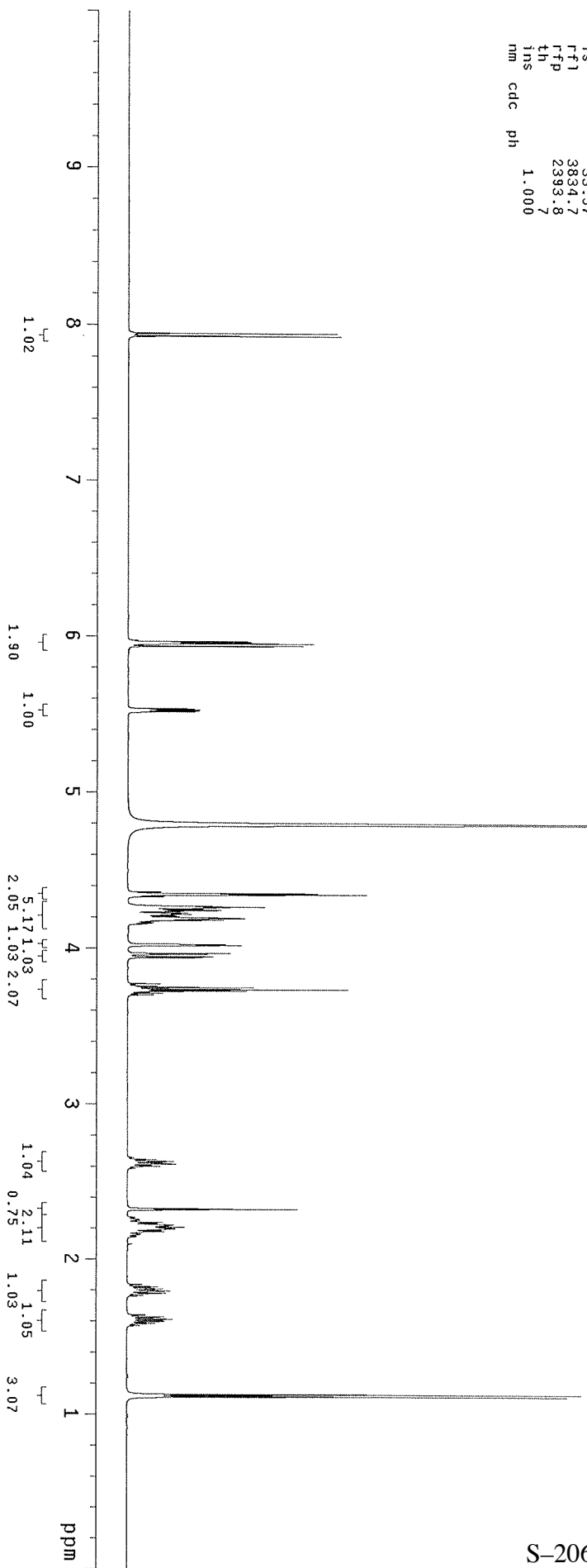
ft

65536

f

math

wft

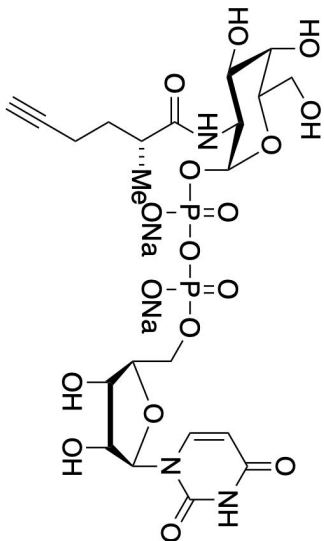




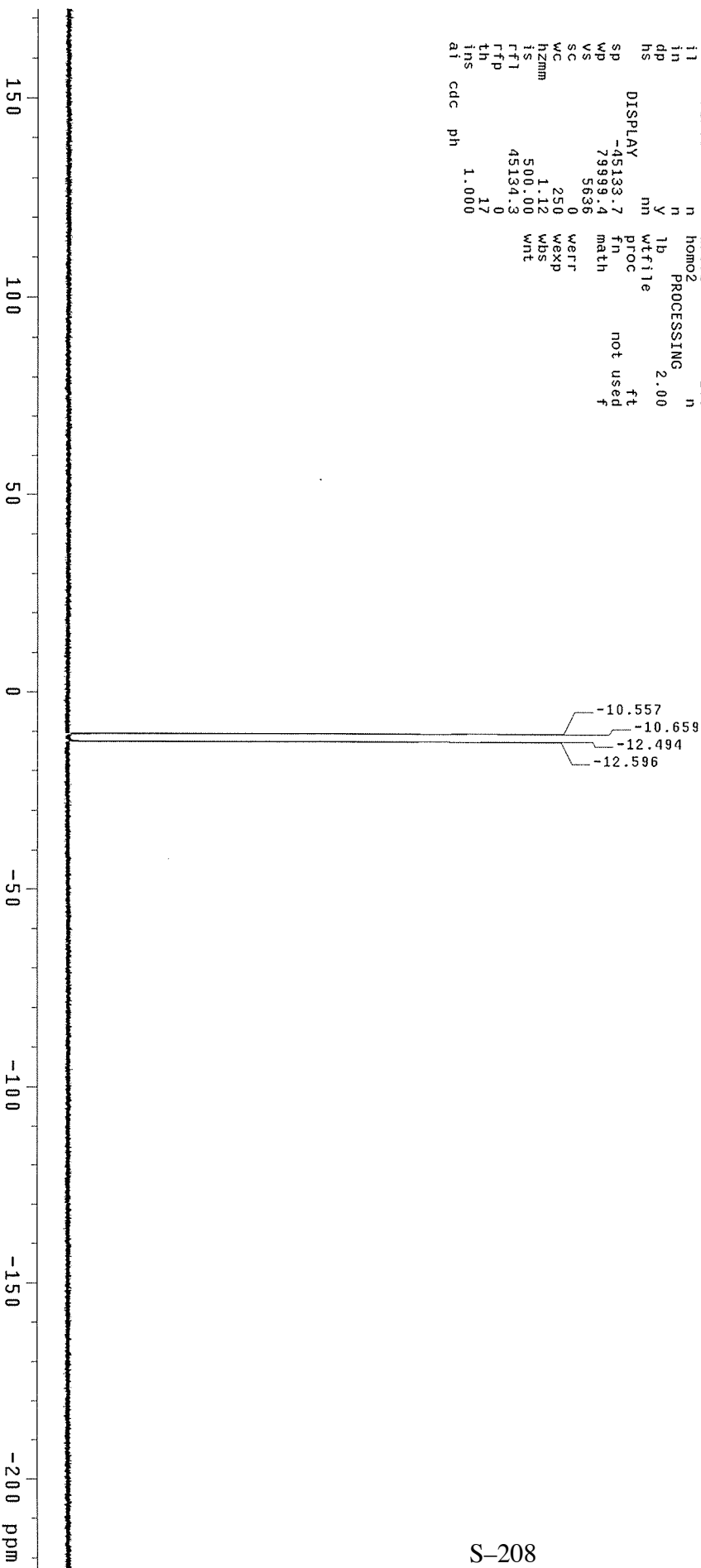
JC3103\_31P\_D20

exp3 s2pu1

SAMPLE DEC. & VT 499.752  
 date Jul 11 2017 dfrq dn H1  
 solvent D2O dpwr H1  
 file exp dof 43  
 ACQUISITION 202.297 dm 0  
 sfreq 202.297 dmm YYY  
 tn 1.002 dmf W  
 at 1.002 dmf 11400  
 np 160254 dseq  
 sw 80000.0 dres  
 fb 44000 homo 1.0  
 bs 4  
 tpwr 51 dfrq2 DEC2 0  
 pw 6.6 dn2  
 dl 2.000 dpwr2 1  
 tof 0 dof2 0  
 nt 16 dm2 n  
 ct 16 dmm2 C  
 atlock n dmf2 9900  
 gain 30 dseq2  
 dres2 1.0  
 flags homo2  
 i1 n  
 in n  
 dp y lb wtfile PROCESSING 2.00  
 hs nm  
 DISPLAY -45133.7 ft  
 sp -79999.4 fn  
 wp 5636 math not used f  
 vs 0 weff  
 sc 250 wexp  
 wcm hzmm 1.12 wbs  
 is 500.00 wnt  
 rfl 45134.3  
 rfp 0  
 th 17  
 ins 1.000  
 ai cdc ph



UDP-Sugar (R)-14

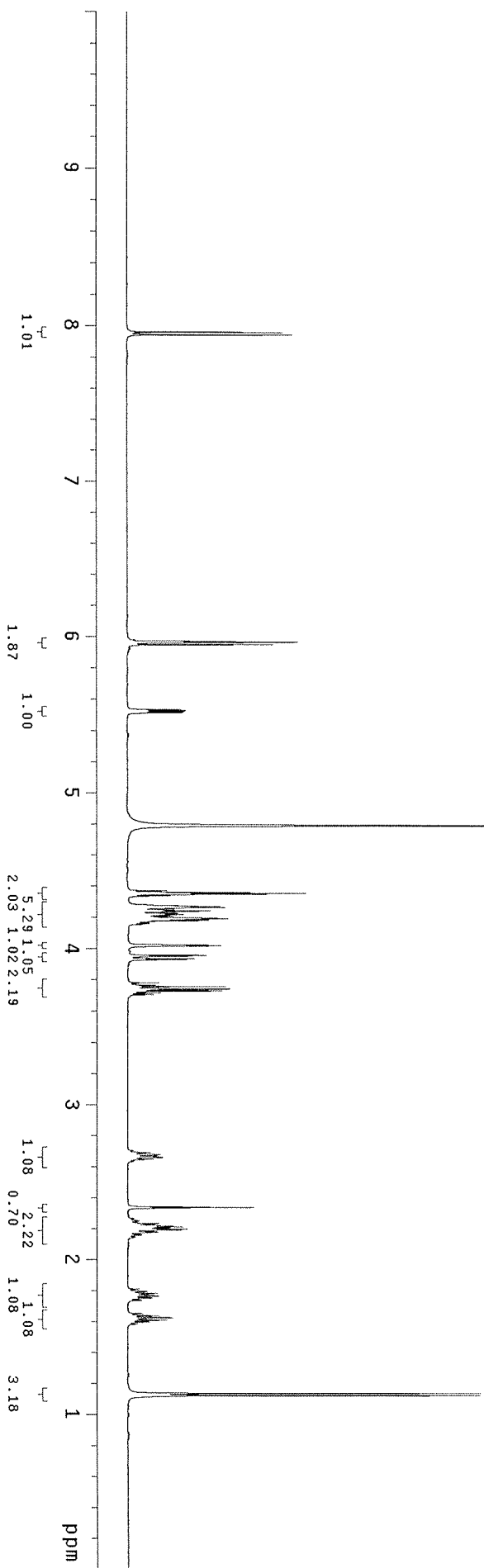
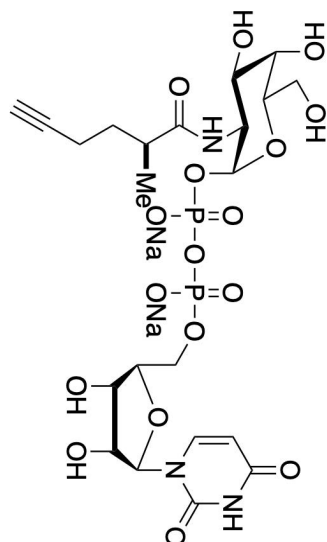




JC5163\_1H\_D20

exp1 s2pu1

date	Aug 12 2017	DEC. & VT	499.752
solvent	D2O	dn	H1
file	exp	dpwr	30
ACQUISITION	exp	doF	0
sfrq	499.752	dm	mm
tn	H1	dmm	C
at	4.000	dmt	200
np	64000	dseq	
sw	8000.0	dres	1.0
fb	4000	homo	n
bs	4	dfreq2	DEC2
tpwr	60	dn2	0
pw	8.0	dpwr2	1
dl	0	doF2	0
tof	0	dmm2	n
nt	64	dmf2	C
ct	64	dseq2	200
atock	n	dres2	1.0
gain	40	homo2	n
FLAGS		PROCESsing	
il	n	wfitle	ft
in	y	proc	65536
dp	nm	fn	f
hs	DISPLAY	math	
sp	-0.2	weff	
wd	4997.3	wexp	
vs	503	wds	
sc	0	wnt	
wc	250		
h2mm	19.99	wft	
is	1007.20		
rfl	3836.2		
rffp	2393.8		
th	7		
ins	1.000		
nm	cdc		
ph			



JCS163\_13C\_D20

expt1 s2pul1

SAMPLE DEC. & VT

date Aug 12 2017 dn H1

solvent D2O dof -499.0

file exp dm yyy

ACQUISITION exp dmm 11400

sfrq 125.674 dmf 43

tn C13 dpwr

at 1.500 PROCESSING 2.00

np 99016 1b fn not used f

sw 33003.3 math

fb 18000 4

bs 7.0 weff

pw 7.0 wexp

tpwr 51 wds

di 1.000 wnt

tof 100.0 DISPLAY

nt 2048 sp -4403.5

ct 992 wp 33002.8

atock n vs 29382

gain 54 SC 0

flags WC 250

i1 n hzmm 17.87

in n is 500.00

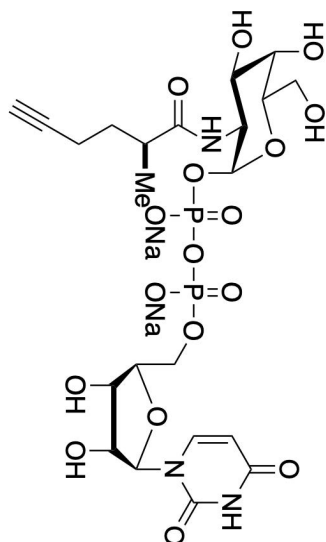
dp y rfl 6617.7

hs nm rfp 2213.7

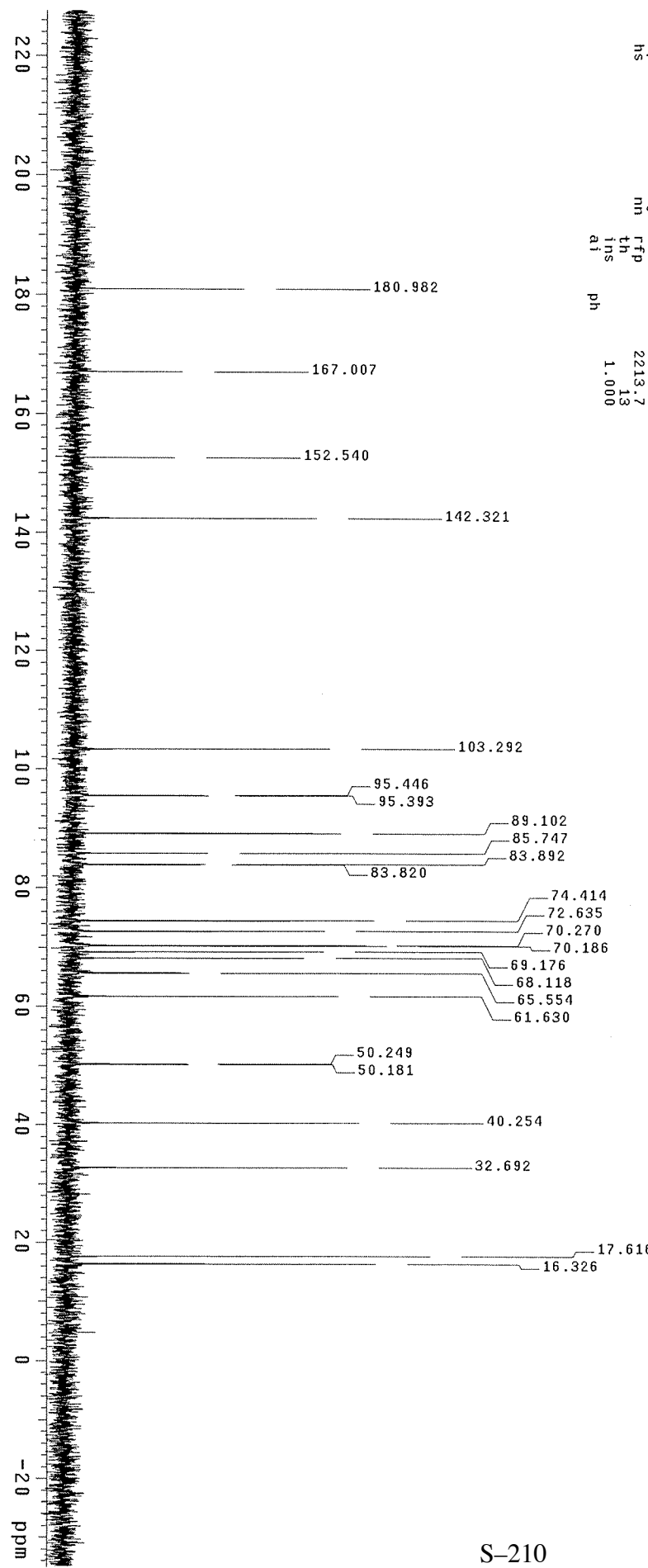
th th 13

ins ai 1.000

ph



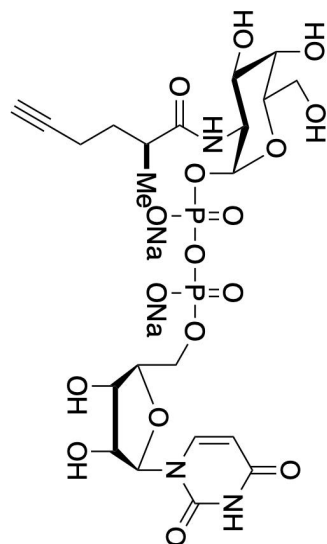
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JC3105\_31P\_D20

exp3 s2pu1

SAMPLE	date	Jul 11 2017	DEC. & VT	499.752
SOLVENT	solvent	D2O	H1	43
FILE	file	exp	dpwr	0
ACQUISITION	exp	202.297	dot	0
SFRQ	sfrq	202.297	dm	yyy
IN	in	1.002	dmm	w
AT	at	160254	dseq	11400
NP	np	80000.0	dres	1.0
SW	sw	44000	homo	n
FB	fb	44000	homo	n
BS	bs	4	dfreq2	0
TPWR	tpwr	51	dpwr2	0
PW	pw	6.6	dpwr2	1
D14	d14	2.000	dotf2	0
TOF	tof	0	dotf2	0
NT	nt	16	dmm2	n
CT	ct	16	dmm2	c
ATLOCK	atlock	n	dseq2	9900
GAIN	gain	30	dres2	1.0
FLAGS	flags	30	homo2	n
I1	i1	n	homo2	n
IN	in	n	PROCESSING	2.00
DP	dp	y	wtfile	
HS	hs	nm	proc	ft
DISPLAY	display	-45133.7	fn	not used
SP	sp	79999.4	math	f
WP	wp	5636	werr	
VS	vs	0	wexp	
SC	sc	0	wds	
WC	wc	250	wnt	
H2MM	h2mm	1.12		
IS	is	500.00		
FF1	ff1	45134.3		
FFP	ffp	0		
TH	th	17		
INS	ins	1.000		
AI	ai	cdc		
		ph		



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