## Supplementary Information

**Table S1. Identification of MUC5B and MUC5AC in submucosal gland secretions.**

The major high molecular weight glycoprotein in submucosal gland secretions migrating with a molecular mass of ~4 MDa by 1D SDS-AgPAGE was excised and digested with trypsin, and the resulting peptides were analysed by MALDI-TOF-MS. PMF searching identified the presence of (A) MUC5B and (B) MUC5AC. Details of the proteins identified, masses of the tryptic peptides detected, error to the theoretical peptide masses, the position of the peptides within each protein, and the peptide sequences are shown for CF subject 1. This data is representative of all subjects.

A

| Protein | SwissProt Accession | MW (Da) | pI | Unique Peptide Hits | % Coverage |
|---|---|---|---|---|---|
| Mucin 5B | Q9HC84 | 590122 | 6.24 | 42 | 10.56 |

| *m/z* | Error (ppm) | Peptide Position | Alkylated Cysteines | Peptide Sequence |
|---|---|---|---|---|
| 605.3 | 34.2 | 5364-5368 | | FPGER |
| 722.4 | 33.3 | 1009-1014 | | TSVFIR |
| 879.4 | -56.5 | 5283-5289 | 1 | GVCSDWR |
| 893.4 | 34.5 | 155-161 | | EELPYSR |
| 906.4 | -39.5 | 1588-1593 | 1 | MCYNYR |
| 931.5 | -69.8 | 321-327 | 2 | CPELCPR |
| 960.6 | -62.2 | 678-685 | | GVQLSDWR |
| 973.6 | -46.6 | 5190-5197 | | TWLVPDSR |
| 1036.6 | -38.3 | 2362-2371, 2891-2900, 3589-3598 | | AQAQPGVPLR |
| 1077.5 | -47.6 | 1070-1078 | 1 | DPCTANPFR* |
| 1107.6 | -24.0 | 626-635 | | LTDPNSAFSR |
| 1132.5 | -33.6 | 2398-2405, 2927-2934, 3625-3632, 4154-4161 | 1 | MCFNYEIR |
| 1140.7 | 4.6 | 121-131 | | GLVGSRPVVTR |
| 1258.7 | 44.4 | 226-236 | | LTPLQFGNLQK |
| 1325.7 | -53.6 | 109-119 | | AAYEDFNVQLR |
| 1364.7 | -52.8 | 1867-1876 | 1 | FNMCFNYNVR |
| 1468.8 | -40.5 | 456-468 | 1 | CADSSFTVLAELR |
| 1498.8 | -36.9 | 1567-1578 | 2 | VHCDVHFGLVCR |
| 1508.7 | 38.8 | 1044-1057 | | SVVGDALEFGNSWK |
| 1539.8 | 2.6 | 455-468 | | KCADSSFTVLAELR |
| 1539.8 | 2.6 | 456-469 | | CADSSFTVLAELRK* |
| 1617.8 | -41.4 | 1085-1098 | 2 | QCSILHGPTFAACR |
| 1630.8 | -44.1 | 2346-2361, 2875-2890, 3573-3588, 4102-4117 | 1 | AAGGAVCEQPLGLECR |
| 1679.9 | -54.5 | 1369-1382 | 2 | GYQVCPVLADIECR |
| 1687.8 | -29.9 | 2346-2361, 2875-2890, 3573-3588, 4102-4117 | 2 | AAGGAVCEQPLGLECR* |
| 1724.9 | -40.7 | 938-954 | | IVTENIPCGTTGTTCSK* |
| 1747.8 | -37.6 | 294-307 | 3 | CPTCPCATFVEYSR |
| 1848.0 | -43.5 | 5305-5320 | 2 | VYKPCGPIQPATCNSR |
| 1938.0 | -41.2 | 5265-5280 | 2 | LEVPCQSLEAYAELCR |
| 1981.0 | -34.2 | 2372-2388, 2901-2917, 3599-3615 | 2 | ELGQVVECSLDFGLVCR |
| 2027.9 | 12.6 | 1766-1784 | | TETTMSPLTNTTTSQGTTR |
| 2113.0 | -40.1 | 659-677 | 3 | SEDCLCAALSSYVHACAAK |
| 2113.0 | -7.3 | 1182-1200 | 2 | NPSGHCLVDLPGLEGCYPK |
| 2122.9 | -21.4 | 4906-4922 | 3 | AGCHFYAVCNQHCDIDR |
| 2151.0 | 27.4 | 938-957 | 2 | IVTENIPCGTTGTTCSKAIK |
| 2257.9 | -15.6 | 5632-5649 | | YSAEAQAMQHQCTCCQER |
| 2257.9 | 39.1 | 1021-1041 | 3 | GRVCGLCGNFDDNAINDFATR |
| 2262.1 | -35.1 | 1405-1424 | | GLMCANSQQSPPLCHDYELR |
| 2285.0 | 28.1 | 1383-1402 | 1 | AAQLPDMPLEELGQQVDCDR |
| 2315.1 | -8.8 | 1021-1041 | 1 | GRVCGLCGNFDDNAINDFATR |
| 2376.1 | -28.0 | 1405-1424 | 2 | GLMCANSQQSPPLCHDYELR |
| 2878.1 | 22.1 | 328-351 | 4 | TCPLNMQHQECGSPCTDTCSNPQR |

* Sequence confirmed by MS/MS

B

| Protein | SwissProt Accession | MW (Da) | pI | Unique Peptide Hits | % Coverage |
|---|---|---|---|---|---|
| Mucin 5AC | Q8WWQ4 | 135520 | 9.11 | 10 | 14.68 |

| *m/z* | Error (ppm) | Peptide Position | Alkylated Cysteines | Peptide Sequence |
|---|---|---|---|---|
| 862.4 | -13.6 | 269-275, 567-573, 1000-1006 | 1 | EEGLVCR |
| 900.5 | 37.0 | 238-244, 969-975 | | RPEEITR* |
| 1022.5 | -12.6 | 223-230, 521-528, 954-961 | | ETYNNIIR |
| 1046.6 | -87.7 | 178-187 | | TSTSHVSISK |
| 1061.5 | -7.9 | 276-284, 574-582, 1007-1015 | | NQDQQGPFK* |
| 1084.5 | -35.1 | 285-292, 583-590, 1016-1023 | 1 | MCLNYEVR* |
| 1360.6 | -30.1 | 495-504, 928-937 | 2 | DCHPRCTWTK |
| 1848.0 | -53.6 | 269-284, 567-582, 1000-1015 | | EEGLVCRNQDQQGPFK |
| 2062.1 | -36.9 | 549-566 | 1 | SHPEVSIEHLGQVVQCSR |
| 2262.1 | 1.6 | 249-268, 980-999 | 1 | AESHPEVSIEHLGQVVQCSR |

* Sequence confirmed by MS/MS

**Table S2. Identification of gp-340 in submucosal gland secretions.**

The high molecular weight glycoprotein in submucosal gland secretions migrating with a molecular mass of ~1 MDa by 1D SDS-AgPAGE was excised and digested with trypsin, and the resulting peptides were analysed by MALDI-TOF-MS. PMF searching identified the presence of gp-340 variant protein. Details of the protein identified, masses of the tryptic peptides detected, error to the theoretical peptide masses, the position of the peptides within each protein, and the peptide sequences are shown for non-CF lung disease subject 3. This data is representative of all subjects.

| Protein | SwissProt Accession | MW (Da) | pI | Unique Peptide Hits | % Coverage |
|---|---|---|---|---|---|
| Gp-340 | Q9UKJ4 | 260591 | 5.18 | 11 | 19.9 |

| *m/z* | Error (ppm) | Peptide Position | Oxidised Methionine | Alkylated Cycteines | Peptide Sequence |
|---|---|---|---|---|---|
| 730.4 | -1.7 | 104-110, 236-242, 365-371, 496-502, 604-610, 864-870, 995-1001, 1124-1130, 1253-1259, 1382-1388, 1511-1517, 1642-1648 | | | LVNGDGR |
| 778.4 | -38.2 | 247-252, 376-381, 507-512, 615-620, 746-751, 875-880, 1145-1140, 1264-1269, 1393-1398, 1653-1658 | | | VEVLYR |
| 838.4 | 103.9 | 2360-2366 | | | CKMVVCR |
| 991.6 | -36.4 | 245-252, 1651-1658 | | | GRVEVLYR |
| 1279.7 | 53.2 | 372-381, 503-512, 611-620, 742-751, 871-880, 1131-1140, 1260-1269, 1389-1398 | | 1 | CQGRVEVLYR |
| 1420.7 | -82.3 | 2086-2098 | | | GSFTSSSNFMSIR |
| 1459.8 | -72.3 | 418-431, 549-562, 657-670, 788-801, 917-930, 1048-1061, 1177-1190, 1306-1319, 1435-1448, 1695-1708 | | | FGQGSGPIVLDDVR* |
| 1545.7 | -82.0 | 403-417, 902-916, 1291-1305, 1420-1434 | | 1 | QLGCGWATSAPGNAR |
| 1575.7 | -85.0 | 642-656, 773-787, 1162-1176, 1680-1694 | | 1 | QLGCGWAMSAPGNAR |
| 1591.7 | -49.7 | 642-656, 773-787, 1162-1176, 1680-1694 | 1 | 1 | QLGCGWAMSAPGNAR |
| 1601.8 | -51.2 | 534-548 | | 1 | QLGCGWAMLAPGNAR |

* Sequence confirmed by MS/MS

**Table S3. Monosaccharide compositions of *O*-linked oligosaccharides from MUC5B/MUC5AC.** *O*-linked oligosaccharides were released by reductive β-elimination from MUC5B/MUC5AC in submucosal gland fluid. The resulting oligosaccharide alditols were analysed by LC-ESI-MS/MS, and their relative intensities were determined based on single-ion chromatograms. GlycoComp was used to determine the monosaccharide composition of the detected oligosaccharides. Masses corresponding to oligosaccharides, the monosaccharide composition of each oligosaccharide and the normalised percent abundance of each oligosaccharide from each subject are shown.

| M | Composition | CF Lung Disease | | | | | Non-CF Lung Disease | | | | | Control | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | 1 | 2 | 3 | 4 | 5 | 1 | 2 | 3 | 4 | 5 | 1 | 2 | 3 |
| Neutral | | | | | | | | | | | | | | |
| 531.2 | [ 1 , 1 , 1 , 0 , 0 ] | | | | | 6.1 | | | | | 0.5 | | | |
| 588.1 | [ 2 , 1 , 0 , 0 , 0 ] | 0.8 | 0.9 | | 1.4 | | 1.7 | | 1.9 | 0.8 | 1.0 | 3.9 | | 16.2 |
| 734.2 | [ 2 , 1 , 1 , 0 , 0 ] | 3.8 | 2.5 | 1.7 | | 6.5 | | 4.4 | | 4.5 | 3.1 | | 7.5 | |
| 750.3 | [ 2 , 2 , 0 , 0 , 0 ] | | | | 1.9 | | 3.2 | 1.0 | 1.7 | 1.2 | 1.4 | 0.8 | | 2.9 |
| 791.2 | [ 3 , 1 , 0 , 0 , 0 ] | | | | | | | 2.1 | | 1.5 | 1.2 | | | 2.0 |
| 880.2 | [ 2 , 1 , 2 , 0 , 0 ] | | | 0.7 | | | | | | 0.3 | | | 1.6 | |
| 896.2 | [ 2 , 2 , 1 , 0 , 0 ] | | 2.8 | 1.9 | | 4.9 | | 4.2 | | 3.8 | 5.1 | | 4.2 | 3.5 |
| 937.2 | [ 3 , 1 , 1 , 0 , 0 ] | 6.3 | | 6.4 | | 6.2 | | 4.8 | | 3.0 | 1.1 | | 12.7 | |
| 953.3 | [ 3 , 2 , 0 , 0 , 0 ] | | | | | | 7.3 | 2.5 | 5.6 | 2.7 | 1.7 | 3.1 | | 7.8 |
| 1042.4 | [ 2 , 2 , 2 , 0 , 0 ] | | | 3.7 | | 6.9 | | 2.3 | | 4.1 | 1.3 | | 7.4 | |
| 1083.3 | [ 3 , 1 , 2 , 0 , 0 ] | 1.9 | | 1.4 | | | | 2.1 | | 0.5 | | | | |
| 1099.3 | [ 3 , 2 , 1 , 0 , 0 ] | 1.6 | 5.8 | 7.9 | | 6.6 | 4.2 | 10.6 | 5.8 | 14.2 | 5.0 | 1.6 | 7.3 | 6.8 |
| 1140.4 | [ 4 , 1 , 1 , 0 , 0 ] | 6.4 | | | | | | | | | | | | |
| 1188.3 | [ 2 , 2 , 3 , 0 , 0 ] | 1.5 | | 2.3 | | | | 2.3 | | | | | 3.1 | |
| 1204.4 | [ 2 , 3 , 2 , 0 , 0 ] | | | | | | | | | | 2.3 | | | |
| 1245.4 | [ 3 , 2 , 2 , 0 , 0 ] | | 8.1 | 16.8 | | 16.8 | | 14.7 | | 13.1 | 6.1 | | 21.9 | 2.8 |
| 1261.3 | [ 3 , 3 , 1 , 0 , 0 ] | | | | | | | | 0.7 | | 2.6 | | | |
| 1286.5 | [ 4 , 1 , 2 , 0 , 0 ] | 4.6 | | | | | 1.5 | | | | | | | |
| 1350.5 | [ 2 , 3 , 3 , 0 , 0 ] | | | | | | | | | | 2.4 | | | |
| 1391.3 | [ 3 , 2 , 3 , 0 , 0 ] | | | | | | | 8.7 | | 3.8 | 1.7 | | | |
| 1407.3 | [ 3 , 3 , 2 , 0 , 0 ] | | | | | | | | | | 1.7 | | | |
| 1448.4 | [ 4 , 2 , 2 , 0 , 0 ] | 7.0 | | 8.0 | | | | | | | | | 7.6 | |
| 1594.4 | [ 4 , 2 , 3 , 0 , 0 ] | 8.5 | | 1.2 | | | | | | | | | | |
| 1798.0 | [ 5 , 2 , 3 , 0 , 0 ] | 1.7 | | | | | | | | | | | | |
| Sialylated | | | | | | | | | | | | | | |
| 676.2 | [ 1 , 1 , 0 , 1 , 0 ] | 3.6 | | | | | | | | 0.7 | 1.0 | | | |
| 717.2 | [ 2 , 0 , 0 , 1 , 0 ] | 1.1 | | 0.8 | | | | | | 0.8 | 0.9 | | | |
| 879.1 | [ 2 , 1 , 0 , 1 , 0 ] | | 1.9 | 1.1 | 1.2 | 1.7 | 2.0 | | 1.8 | 1.4 | 1.4 | 2.2 | 0.9 | 3.5 |
| 967.2 | [ 1 , 1 , 0 , 2 , 0 ] | 1.5 | | | | | | | | | | | | |
| 1025.2 | [ 2 , 1 , 1 , 1 , 0 ] | 1.1 | | | | | | | 1.1 | 0.9 | 2.0 | | | |
| 1041.5 | [ 2 , 2 , 0 , 1 , 0 ] | 3.2 | 7.6 | 1.4 | 4.7 | | 4.9 | 1.6 | 3.0 | 2.7 | 3.4 | 4.6 | | |
| 1082.3 | [ 3 , 1 , 0 , 1 , 0 ] | 6.7 | 9.4 | 6.8 | 1.9 | 4.0 | 2.9 | 3.0 | 1.6 | 2.9 | 3.8 | | 5.4 | |
| 1187.4 | [ 2 , 2 , 1 , 1 , 0 ] | 2.4 | 9.6 | 5.3 | 9.9 | 8.7 | 7.3 | 1.4 | 12.5 | 6.4 | 6.4 | 6.3 | 2.2 | 16.5 |
| 1228.4 | [ 3 , 1 , 1 , 1 , 0 ] | | | | | | 1.7 | | 3.0 | | | | | |
| 1244.4 | [ 3 , 2 , 0 , 1 , 0 ] | 2.7 | 7.4 | 2.2 | | 4.2 | 10.5 | 8.5 | 0.8 | 3.3 | 2.6 | 13.1 | | |
| 1316.4 | [ 2 , 1 , 1 , 2 , 0 ] | | | | | | 1.0 | | | | | | | |
| 1332.3 | [ 2 , 2 , 0 , 2 , 0 ] | 6.7 | | | 4.5 | 8.4 | 2.4 | | | 4.6 | 4.7 | | | |
| 1333.4 | [ 2 , 2 , 2 , 1 , 0 ] | | 3.3 | 1.3 | | 7.4 | | | | | 3.2 | | | |
| 1390.4 | [ 3 , 2 , 1 , 1 , 0 ] | 7.9 | 25.3 | 12.0 | 6.4 | 11.6 | 12.9 | 13.5 | 17.1 | 15.4 | 10.6 | 19.8 | 12.1 | 18.5 |
| 1478.4 | [ 2 , 2 , 1 , 2 , 0 ] | 3.7 | 5.8 | | 12.2 | | 5.0 | | 3.4 | 1.4 | 4.9 | 3.9 | | |
| 1535.4 | [ 3 , 2 , 0 , 2 , 0 ] | | | 2.5 | | | | | | 1.4 | 1.9 | 5.3 | | |
| 1536.7 | [ 3 , 2 , 2 , 1 , 0 ] | 0.6 | 7.2 | 7.3 | | | 1.7 | 4.5 | 3.9 | 3.5 | 3.1 | | 6.1 | |
| 1552.3 | [ 3 , 3 , 1 , 1 , 0 ] | | | | 4.1 | | 2.3 | | | | 3.7 | | | |
| 1593.5 | [ 4 , 2 , 1 , 1 , 0 ] | 9.8 | | 2.2 | | | | | | | | 10.0 | | |
| 1681.3 | [ 3 , 2 , 1 , 2 , 0 ] | | 2.6 | 1.7 | | | | | 9.0 | | | | | |
| 1698.0 | [ 3 , 3 , 2 , 1 , 0 ] | | | | | | | | | 0.4 | 1.8 | | | |
| 1698.8 | [ 3 , 3 , 0 , 2 , 0 ] | | | | 1.7 | | | | | 1.2 | 0.8 | | | |
| 1739.4 | [ 4 , 2 , 2 , 1 , 0 ] | 3.3 | | | | | | | | | | | | |
| 1756.0 | [ 4 , 3 , 1 , 1 , 0 ] | | | | | | | | | 0.6 | | | | |
| 1828.0 | [ 3 , 2 , 2 , 2 , 0 ] | | | | 1.7 | | | | 2.4 | | | | | |
| 1844.0 | [ 3 , 3 , 1 , 2 , 0 ] | | | | 3.0 | | | | | | | | | |
| 1901.0 | [ 4 , 3 , 0 , 2 , 0 ] | | | | | | | | | | 1.5 | | | |
| 1902.2 | [ 4 , 3 , 2 , 1 , 0 ] | 0.9 | | | | | | | | 1.7 | | | | |
| 1990.4 | [ 3 , 3 , 2 , 2 , 0 ] | | | | 3.9 | | 2.2 | | 1.6 | | | 4.1 | | |
| 2064.2 | [ 4 , 4 , 2 , 1 , 0 ] | | | | 2.1 | | | | | | | | | |
| 2192.8 | [ 4 , 3 , 2 , 2 , 0 ] | | | | | | | | 1.4 | | | | | |
| 2208.6 | [ 4 , 4 , 1 , 2 , 0 ] | | | | 2.1 | | | | 0.5 | | 0.9 | | | |
| 2209.8 | [ 4 , 4 , 3 , 1 , 0 ] | | | | | | 2.1 | | 1.2 | | | 1.5 | | |
| 2280.8 | [ 3 , 3 , 2 , 3 , 0 ] | | | | 4.2 | | 1.6 | | 2.6 | | 1.5 | | | |
| 2282.9 | [ 3 , 3 , 4 , 2 , 0 ] | | | | 2.5 | | | | | | | | | |
| 2354.8 | [ 4 , 4 , 2 , 2 , 0 ] | | | | | | | | | | | 3.0 | | |
| 2499.6 | [ 4 , 4 , 1 , 3 , 0 ] | | | | 4.1 | | 2.1 | | 1.5 | | | 3.8 | | |
| 2500.8 | [ 4 , 4 , 3 , 2 , 0 ] | | | | | | | | 1.8 | | | | | |
| 2646.4 | [ 4 , 4 , 2 , 3 , 0 ] | | | | 5.9 | | 4.2 | | 3.7 | | | 5.3 | | 4.3 |
| 2791.2 | [ 4 , 4 , 1 , 4 , 0 ] | | | | 4.1 | | | | | | | | | |
| 2792.4 | [ 4 , 4 , 3 , 3 , 0 ] | | | | | | 2.0 | | | | | 2.7 | | |
| 2866.6 | [ 5 , 5 , 3 , 2 , 0 ] | | | | 3.6 | | | | | | | | | |
| 2994.6 | [ 5 , 4 , 1 , 4 , 0 ] | | | | | | | | 1.6 | | | | | |
| 3156.4 | [ 5 , 5 , 1 , 4 , 0 ] | | | | 3.1 | | | | 3.5 | | | | | |
| 3157.0 | [ 5 , 5 , 3 , 3 , 0 ] | | | | 3.7 | | | | | | | 4.8 | | |
| Sulfated | | | | | | | | | | | | | | |
| 959.1 | [ 2 , 1 , 0 , 1 , 1 ] | 2.1 | | | | | | 1.3 | | | | | | |
| 1105.3 | [ 2 , 1 , 1 , 1 , 1 ] | | | | | | | 1.4 | | | | | | |
| 1121.2 | [ 2 , 2 , 0 , 1 , 1 ] | | | | | | | 5.1 | 1.3 | | | | | |
| 1179.2 | [ 3 , 2 , 1 , 0 , 1 ] | | | | | | 2.2 | | 1.3 | | 1.0 | | | 4.5 |
| 1267.1 | [ 2 , 2 , 1 , 1 , 1 ] | 0.5 | | | | | 1.9 | | | | 0.7 | | | |
| 1470.2 | [ 3 , 2 , 1 , 1 , 1 ] | | | | | | 2.0 | | | | | | | |
| 1471.6 | [ 3 , 2 , 3 , 0 , 1 ] | | | 1.7 | | | | | | | | | | |
| 2014.0 | [ 4 , 5 , 0 , 1 , 1 ] | | | | | | | | 1.0 | | | | | |
| 2143.6 | [ 4 , 4 , 2 , 1 , 1 ] | | | | | | 2.0 | | | | | | | |
| 2433.8 | [ 4 , 4 , 0 , 3 , 1 ] | | | | | | | | | | | | | 5.2 |
| 2580.6 | [ 4 , 4 , 1 , 3 , 1 ] | | | | | | 2.3 | | 1.5 | | | | | |
| 2799.9 | [ 5 , 5 , 2 , 2 , 1 ] | | | | | | | | | | | | | 5.4 |
| 2945.4 | [ 5 , 5 , 1 , 3 , 1 ] | | | | 6.2 | | | | | | | | | |
| 2946.2 | [ 5 , 5 , 3 , 2 , 1 ] | | | | | | | | | | 2.0 | | | |
| 3311.0 | [ 6 , 6 , 3 , 2 , 1 ] | | | | | | 2.9 | | | | | | | |

M, measured neutral reduced mass; Composition, [*N* -acetylhexosamine, hexose, fucose, *N* -acetylneuraminic acid, sulfate]. Assumption: All deoxyhexoses are fucose.

**Table S4. Monosaccharide compositions of *O*-linked oligosaccharides from gp-340.**

*O*-linked oligosaccharides were released by reductive β-elimination from gp-340 in submucosal gland secretions. The resulting oligosaccharide alditols were analysed by LC-ESI-MS/MS, and their relative intensities were determined based on single-ion chromatograms. GlycoComp was used to determine the monosaccharide composition of the detected oligosaccharides. Masses corresponding to oligosaccharides, the monosaccharide composition of each oligosaccharide and the normalised percent abundance of each oligosaccharide from each subject are shown. Although neutral oligosaccharides were only detected from subjects with non-CF lung disease, the low abundance of these components means that there are no significant differences in monosaccharide abundance between the subject groups.

| M | Composition | CF Lung Disease | | | | | Non-CF Lung Disease | | | | | Control | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | 1 | 2 | 3 | 4 | 5 | 1 | 2 | 3 | 4 | 5 | 1 | 2 | 3 |
| Neutral | | | | | | | | | | | | | | |
| 588.1 | [ 2 , 1 , 0 , 0 , 0 ] | | | | | | 1.9 | | | | | | | |
| 750.3 | [ 2 , 2 , 0 , 0 , 0 ] | | | | | | 3.7 | | | | | | | |
| 896.2 | [ 2 , 2 , 1 , 0 , 0 ] | | | | | | 1.0 | | | | | | | |
| 953.3 | [ 3 , 2 , 0 , 0 , 0 ] | | | | | | 6.6 | | | | | | | |
| 1042.4 | [ 2 , 2 , 2 , 0 , 0 ] | | | | | | | | | 3.2 | | | | |
| 1099.3 | [ 3 , 2 , 1 , 0 , 0 ] | | | | | | 4.9 | | | 8.3 | | | | |
| 1188.3 | [ 2 , 2 , 3 , 0 , 0 ] | | | | | | | | | | 1.0 | | | |
| 1245.4 | [ 3 , 2 , 2 , 0 , 0 ] | | | | | | | | | 7.7 | | | | |
| 1391.3 | [ 3 , 2 , 3 , 0 , 0 ] | | | | | | | | | 2.2 | | | | |
| 2064.8 | [ 4 , 4 , 4 , 0 , 0 ] | | | | | | | | | | 3.6 | | | |
| Sialylated | | | | | | | | | | | | | | |
| 676.2 | [ 1 , 1 , 0 , 1 , 0 ] | 16.5 | 6.7 | | | | | | | | | | | 3.6 |
| 879.1 | [ 2 , 1 , 0 , 1 , 0 ] | | | | | | 1.9 | | | | | | | |
| 967.3 | [ 1 , 1 , 0 , 2 , 0 ] | | | | | | | | | | | | | 6.0 |
| 1041.5 | [ 2 , 2 , 0 , 1 , 0 ] | | | | | | 6.3 | | | 5.2 | | | | |
| 1187.4 | [ 2 , 2 , 1 , 1 , 0 ] | | | 14.4 | | | 11.7 | | | 5.2 | 1.2 | | | |
| 1244.4 | [ 3 , 2 , 0 , 1 , 0 ] | | | | | | 9.9 | | | | | | | |
| 1316.4 | [ 2 , 1 , 1 , 2 , 0 ] | 26.8 | 7.2 | 9.0 | 9.9 | | | | | | | | | |
| 1332.3 | [ 2 , 2 , 0 , 2 , 0 ] | 27.7 | 50.5 | 48.5 | 48.1 | 62.2 | 7.0 | 29.2 | 40.2 | 26.2 | 18.9 | 25.3 | 21.6 | 11.4 |
| 1390.4 | [ 3 , 2 , 1 , 1 , 0 ] | | | | | | 22.6 | | | 7.1 | | 3.9 | | |
| 1478.4 | [ 2 , 2 , 1 , 2 , 0 ] | 16.7 | 15.0 | 18.5 | 42.3 | 37.8 | 11.9 | 14.8 | 14.7 | 10.5 | 20.0 | 15.1 | 16.8 | 25.1 |
| 1844.0 | [ 3 , 3 , 1 , 2 , 0 ] | | | | | | | | | | 1.4 | | | |
| 1990.4 | [ 3 , 3 , 2 , 2 , 0 ] | | | | | | | | | | 4.6 | 3.0 | | 13.2 |
| 2208.8 | [ 4 , 4 , 1 , 2 , 0 ] | | | | | | | | | | 1.6 | | | |
| 2353.6 | [ 4 , 4 , 0 , 3 , 0 ] | | | | | | | 8.3 | 8.5 | 6.4 | | 10.1 | 20.0 | |
| 2280.8 | [ 3 , 3 , 2 , 3 , 0 ] | | | | | | | | | | 4.2 | | | |
| 2499.6 | [ 4 , 4 , 1 , 3 , 0 ] | | | | | | | 17.2 | 19.1 | 9.0 | | 14.8 | 30.0 | 8.1 |
| 2500.4 | [ 4 , 4 , 3 , 2 , 0 ] | 12.4 | 20.6 | 7.3 | | | 3.5 | 5.4 | | | 12.4 | | | |
| 2646.4 | [ 4 , 4 , 2 , 3 , 0 ] | | | | | | | 14.4 | 9.9 | 6.2 | 13.7 | 9.4 | 11.6 | 14.8 |
| 2791.2 | [ 4 , 4 , 1 , 4 , 0 ] | | | | | | | | 7.7 | | | | | |
| 2792.4 | [ 4 , 4 , 3 , 3 , 0 ] | | | | | | 6.1 | 10.8 | | | 13.6 | 7.6 | | 13.9 |
| Sulfated | | | | | | | | | | | | | | |
| 1105.3 | [ 2 , 1 , 1 , 1 , 1 ] | | | | | | | | | 2.9 | | | | |
| 2579.8 | [ 4 , 4 , 1 , 3 , 1 ] | | | | | | | | | 3.9 | | 5.0 | | |

M, measured neutral reduced mass; Composition, [*N* -acetylhexosamine, hexose, fucose, *N* -acetylneuraminic acid, sulfate].
Assumption: All deoxyhexoses are fucose.

**Table S5. Structures of *O*-linked oligosaccharides from MUC5B/MUC5AC.** *O*-linked oligosaccharides were released by reductive β-elimination from MUC5B/MUC5AC in submucosal gland secretions. The resulting oligosaccharide alditols were analysed by LC-ESI-MS/MS, and their relative intensities were determined based on single-ion chromatograms. GlycosidIQ was used to assign structures to the detected oligosaccharides, which were also manually validated. Oligosaccharides were found to contain all major *O*-glycan cores, both chain types, and had terminal epitopes dominated by Lewis-type and blood group antigens. Masses corresponding to oligosaccharides, assigned oligosaccharide structures and the normalised percent abundance of each oligosaccharide from each subject are shown. Abbreviations: Fuc, fucose; Gal, galactose; GlcNAc, *N*-acetylglucosamine; GalNAc, *N*-acetylgalactosamine; NeuAc, *N*-acetylneuraminc acid; a – α linkage; b – β linkage; 1, 2, 3, 4, 6 – linked carbon position.

| M | Structure | CF Diseased |  |  |  |  | non-CF Diseased |  |  |  |  | Control |  |  |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
|  |  | 1 | 2 | 3 | 4 | 5 | 1 | 2 | 3 | 4 | 5 | 1 | 2 | 3 |
|  | Neutral |  |  |  |  |  |  |  |  |  |  |  |  |  |
| 531.2 | Fuc(a1-2)Gal(b1-3)GalNAc |  |  |  |  | 6.1 |  |  |  |  |  |  |  |  |
| 588.2 | Gal(b1-3)GlcNAc(b1-3)GalNAc |  |  |  |  |  |  |  |  |  |  | 3.9 |  | 6.5 |
| 588.2 | Gal(b1-4)GlcNAc(b1-3)GalNAc | 1.0 |  |  | 1.4 |  | 1.3 |  | 1.7 |  |  |  |  |  |
| 588.2 | *Gal(b1-3)**[GlcNAc(b1-6)]**GalNAc |  |  |  |  |  |  |  |  |  |  |  |  | 1.3 |
| 734.0 | Fuc(a1-2)Gal(b1-3)**[GlcNAc(b1-6)]**GalNAc | 1.1 |  |  |  |  |  |  |  |  |  |  | 1.5 |  |
| 734.0 | Fuc(a1-2)[GalNAc(a1-3)]Gal(b1-3)GalNAc | 1.0 |  |  |  |  |  |  |  |  |  |  |  |  |
| 734.2 | Fuc(a1-3/4)[Gal(b1-3/4)]GlcNAc(b1-3)GalNAc | 0.4 |  |  |  |  |  | 3.9 |  | 3.9 | 3.1 |  |  |  |
| 734.2 | Fuc(a1-2)Gal(b1-3)GlcNAc(b1-3)GalNAc |  | 2.5 |  |  | 4.8 |  |  |  |  |  |  | 4.8 |  |
| 734.2 | Fuc(a1-2)Gal(b1-4)GlcNAc(b1-3)GalNAc |  |  |  |  | 1.7 |  |  |  |  |  |  |  |  |
| 750.3 | Gal(b1-3)**[Gal(b1-4)GlcNAc(b1-6)]**GalNAc |  |  |  | 1.9 |  | 2.9 | 1.0 | 1.5 | 1.2 | 1.7 | 0.8 |  | 2.5 |
| 750.3 | Gal(b1-3)GlcNAc(b1-3)Gal(b1-3)GalNAc |  |  |  |  |  |  |  |  |  |  |  |  | 0.7 |
| 791.2 | GlcNAc(b1-3)**[Gal(b1-3)GlcNAc(b1-6)]**GalNAc |  |  |  |  |  |  | 2.1 |  |  |  |  |  | 2.2 |
| 791.2 | GlcNAc(b1-3)**[Gal(b1-4)GlcNAc(b1-6)]**GalNAc |  |  |  |  |  |  |  |  | 1.5 | 1.6 |  |  |  |
| 880.3 | Fuc(a1-2)Gal(b1-3/4)[Fuc(a1-3/4)]GlcNAc(b1-3)GalNAc |  |  |  |  |  |  |  |  |  |  |  | 1.6 |  |
| 896.2 | Fuc(a1-3/4)[Gal(b1-3/4)]GlcNAc(b1-3)Gal(b1-3)GalNAc |  |  |  |  |  |  |  |  |  | 3.5 |  |  | 3.9 |
| 896.2 | Gal(b1-3)**[Fuc(a1-2)Gal(b1-4)GlcNAc(b1-6)]**GalNAc |  | 2.5 | 1.3 |  | 2.1 |  | 1.8 |  | 1.7 | 0.8 |  | 2.6 |  |
| 896.2 | Fuc(a1-2)Gal(b1-3)**[Gal(b1-3)GlcNAc(b1-6)]**GalNAc |  |  |  |  | 2.8 |  |  |  | 1.4 |  |  |  |  |
| 896.2 | Fuc(a1-2)Gal(b1-3)**[Gal(b1-4)GlcNAc(b1-6)]**GalNAc |  |  |  |  |  |  | 1.6 |  |  | 1.1 |  |  |  |
| 937.3 | Fuc(a1-2)[GalNAc(a1-3)]Gal(b1-3/4)GlcNAc(b1-3)GalNAc |  |  |  |  |  |  |  |  |  |  |  | 4.4 |  |
| 937.3 | GlcNAc(b1-3)**[Fuc(a1-2)Gal(b1-3)GlcNAc(b1-6)]**GalNAc | 6.3 |  | 1.4 |  |  |  |  |  | 3.0 |  |  |  |  |
| 937.3 | GlcNAc(b1-3)**[Fuc(a1-2)Gal(b1-4)GlcNAc(b1-6)]**GalNAc |  |  | 6.0 |  | 6.2 |  | 4.1 |  |  | 1.4 |  | 8.3 |  |
| 953.3 | Gal(b1-3/4)GlcNAc(b1-3)**[Gal(b1-3/4)GlcNAc(b1-6)]**GalNAc |  |  |  |  |  | 7.3 | 2.4 | 5.6 | 2.7 | 2.2 | 3.1 |  | 8.6 |
| 1042.4 | Fuc(a1-2)Gal(b1-3)**[Fuc(a1-2)Gal(b1-4)GlcNAc(b1-6)]**GalNAc |  |  | 4.3 |  | 6.9 |  | 2.3 |  | 3.2 | 1.6 |  | 7.4 |  |
| 1083.0 | Fuc(a1-2)[GalNAc(a1-3)]Gal(b1-3/4)[Fuc(a1-3/4)]GlcNAc(b1-3)GalNAc | 2.2 |  |  |  |  |  |  |  |  |  |  |  |  |
| 1083.0 | GlcNAc(b1-3)**[Fuc(a1-2)Gal(b1-3/4)[Fuc(a1-3/4)]GlcNAc(b1-6)]**GalNAc |  |  | 1.6 |  |  |  |  |  |  |  |  |  |  |
| 1099.3 | Gal(b1-3)**[Fuc(a1-2)[GalNAc(a1-3)]Gal(b1-4)GlcNAc(b1-6)**]GalNAc | 2.1 |  |  |  |  |  |  |  |  |  |  |  |  |
| 1099.3 | Fuc(a1-3/4)[Gal(b1-3/4)]GlcNAc(b1-3)**[Gal(b1-3/4)GlcNAc(b1-6)]**GalNAc |  | 2.4 | 3.2 |  | 2.4 | 4.2 |  | 5.2 |  | 1.2 |  |  |  |
| 1099.3 | Fuc(a1-2)Gal(b1-3)GlcNAc(b1-3)**[Gal(b1-3/4)GlcNAc(b1-6)]**GalNAc |  |  |  |  |  |  | 8.2 |  | 4.2 | 2.9 |  | 4.7 |  |
| 1099.3 | Gal(b1-3/4)GlcNAc(b1-3)**[Fuc(a1-2)Gal(b1-4)GlcNAc(b1-6)]**GalNAc |  | 3.4 | 3.8 |  | 4.2 |  | 2.3 |  | 3.2 |  |  |  |  |
| 1099.3 | Gal(b1-3/4)GlcNAc(b1-3)**[Fuc(a1-3/4)[Gal(b1-3/4)]GlcNAc(b1-6)]**GalNAc |  |  |  |  |  |  |  |  |  |  | 1.6 |  | 7.5 |
| 1140.0 | GlcNAc(b1-3)**[Fuc(a1-2)[GalNAc(a1-3)]Gal(b1-4)GlcNAc(b1-6)]**GalNAc | 6.6 |  |  |  |  |  |  |  |  |  |  |  |  |
| 1188.3 | Fuc(a1-2)Gal(b1-3)**[Fuc(a1-2)Gal(b1-3/4)[Fuc(a1-3/4)]GlcNAc(b1-6)]**GalNAc |  |  | 1.8 |  |  |  | 2.3 |  |  |  |  |  |  |
| 1245.4 | Fuc(a1-2)Gal(b1-4)GlcNAc(b1-3/6)[Fuc(a1-2)Gal(b1-3/4)GlcNAc(b1-3/6)]GalNAc |  | 8.1 | 16.8 |  | 16.8 |  | 14.7 |  | 13.1 | 4.5 |  | 4.7 |  |
| 1245.4 | Fuc(a1-2)Gal(b1-4)GlcNAc(b1-3)**[Fuc(a1-2)Gal(b1-4)GlcNAc(b1-6)]**GalNAc |  |  |  |  |  |  |  |  |  |  |  | 13.0 |  |
| 1245.4 | Fuc(a1-2)Gal(b1-3)**[Fuc(a1-2)[GalNAc(a1-3)]Gal(b1-3/4)GlcNAc(b1-6)]**GalNAc |  |  |  |  |  |  |  |  |  |  |  | 4.2 |  |
| 1261.3 | Fuc(a1-3/4)[Gal(b1-3/4)]GlcNAc(b1-3)Gal(b1-3)**[Gal(b1-3/4)GlcNAc(b1-6)]**GalNAc |  |  |  |  |  |  |  |  |  | 1.1 |  |  |  |
| 1286.0 | GlcNAc(b1-3)**[Fuc(a1-2)[GalNAc(a1-3)]Gal(b1-3/4)[Fuc(a1-3/4)]GlcNAc(b1-6)]**GalNAc | 4.5 |  |  |  |  |  |  |  |  |  |  |  |  |
| 1391.3 | Fuc(a1-2)Gal(b1-3)GlcNAc(b1-3)**[Fuc(a1-2)Gal(b1-3/4)[Fuc(a1-3/4)]GlcNAc(b1-6)]**GalNAc |  |  |  |  |  |  | 6.9 |  | 3.2 |  |  |  |  |
| 1448.4 | Fuc(a1-3/4)[Gal(b1-3/4)]GlcNAc(b1-3)**[Fuc(a1-2)[GalNAc(a1-3)]Gal(b1-4)GlcNAc(b1-6)]**GalNAc |  |  | 5.3 |  |  |  |  |  |  |  |  |  |  |
| 1448.4 | Fuc(a1-2)[GalNAc(a1-3)]Gal(b1-4)GlcNAc(b1-3)**[Fuc(a1-2)Gal(b1-4)GlcNAc(b1-6)]**GalNAc |  |  | 4.0 |  |  |  |  |  |  |  |  |  |  |
| 1448.4 | Fuc(a1-2)[GalNAc(a1-3)]Gal(b1-3)GlcNAc(b1-3)**[Fuc(a1-2)Gal(b1-3)GlcNAc(b1-6)]**GalNAc |  |  |  |  |  |  |  |  |  |  |  | 3.4 |  |
| 1448.4 | Fuc(a1-2)Gal(b1-3)GlcNAc(b1-3)**[Fuc(a1-2)[GalNAc(a1-3)]Gal(b1-4)GlcNAc(b1-6)]**GalNAc |  |  |  |  |  |  |  |  |  |  |  | 4.2 |  |
|  | Sialylated |  |  |  |  |  |  |  |  |  |  |  |  |  |
| 676.1 | NeuAc(a2-3/6)Gal(b1-3)GalNAc | 0.9 |  |  |  |  |  |  |  |  |  |  |  |  |
| 676.1 | Gal(b1-3)**[NeuAc(a2-6)]**GalNAc | 2.5 |  |  |  |  |  |  |  |  | 1.0 |  |  |  |
| 717.2 | GlcNAc(b1-3)**[NeuAc(a2-6)]**GalNAc |  |  |  |  |  |  |  |  | 0.8 | 1.1 |  |  |  |
| 879.1 | Gal(b1-3/4)GlcNAc(b1-3)**[NeuAc(a2-6)]**GalNAc |  |  |  |  |  |  |  |  |  |  | 2.2 | 0.9 | 3.8 |
| 879.1 | NeuAc(a2-3/6)Gal(b1-3/4)GlcNAc(b1-3)GalNAc |  |  |  | 1.2 |  |  |  |  |  |  |  |  |  |
| 879.1 | NeuAc(a2-3/6)Gal(b1-4)GlcNAc(b1-3)GalNAcol |  | 1.9 |  |  | 1.7 |  |  |  |  |  |  |  |  |
| 879.1 | NeuAc(a2-3/6)Gal(b1-3)**[GlcNAc(b1-6)]**GalNAc |  |  |  |  |  |  |  |  |  | 0.9 |  |  |  |
| 967.0 | NeuAc(a2-3/6)Gal(b1-3)**[NeuAc(a2-6)]**GalNAc | 1.5 |  |  |  |  |  |  |  |  |  |  |  |  |
| 1025.2 | Fuc(a1-2)Gal(b1-3)GlcNAc(b1-3)**[NeuAc(a2-6)]**GalNAc |  |  |  |  |  |  |  |  |  | 1.5 |  |  |  |
| 1025.2 | Fuc(a1-3/4)[Gal(b1-3/4)]GlcNAc(b1-3)**[NeuAc(a2-6)]**GalNAc |  |  |  |  |  |  |  |  | 1.2 |  |  |  |  |
| 1041.5 | Gal(b1-3)**[NeuAc(a2-3/6)Gal(b1-3/4)GlcNAc(b1-6)]**GalNAc | 1.0 | 7.6 | 1.6 |  |  |  | 1.6 | 3.0 | 2.2 | 4.2 |  |  |  |
| 1041.5 | Gal(b1-3)**[NeuAc(a2-3/6)Gal(b1-4)GlcNAc(b1-6)]**GalNAc |  |  |  |  |  | 4.1 |  |  |  |  | 1.3 |  |  |
| 1041.5 | NeuAc(a2-3/6)Gal(b1-3)**[Gal(b1-3/4)GlcNAc(b1-6)]**GalNAc | 0.8 |  |  | 3.5 |  |  |  |  |  |  | 3.3 |  |  |
| 1082.3 | GlcNAc(b1-3)**[NeuAc(a2-3/6)Gal(b1-3/4)GlcNAc(b1-6)]**GalNAc |  | 9.4 |  |  | 4.0 | 2.9 | 3.0 |  |  | 4.8 |  | 5.4 |  |
| 1082.3 | NeuAc(a2-3/6)Gal(b1-3/4)GlcNAc(b1-3)**[GlcNAc(b1-6)]**GalNAc |  |  | 7.8 |  |  |  |  |  |  |  |  |  |  |
| 1187.4 | NeuAc(a2-3)Gal(b1-3/4)[Fuc(a1-3/4)]GlcNAc(b1-3)Gal(b1-3)GalNAc | 2.2 |  |  | 5.0 |  |  |  |  |  |  |  |  |  |
| 1187.4 | NeuAc(a2-3/6)Gal(b1-3)**[Fuc(a1-3/4)[Gal(b1-3/4)]GlcNAc(b1-6)]**GalNAc |  | 1.5 |  |  | 4.3 |  |  |  |  |  | 4.6 |  |  |
| 1187.4 | Gal(b1-3)**[NeuAc(a2-3)Gal(b1-3/4)[Fuc(a1-3/4)]GlcNAc(b1-6)]**GalNAc |  | 1.0 |  | 4.9 |  | 7.4 |  | 12.5 |  | 4.2 |  |  | 7.1 |
| 1187.4 | Fuc(a1-2)Gal(b1-3)**[NeuAc(a2-3/6)Gal(b1-3/4)GlcNAc(b1-6)]**GalNAc |  | 7.1 | 3.2 |  | 4.4 |  | 1.4 |  | 5.1 | 3.9 | 1.7 |  |  |
| 1187.4 | NeuAc(a2-3/6)Gal(b1-3)**[Fuc(a1-2)Gal(b1-4)GlcNAc(b1-6)]**GalNAc |  |  | 2.9 |  |  |  |  |  | 1.1 |  |  | 2.2 |  |
| 1228.4 | GlcNAc(b1-3)**[NeuAc(a2-3)Gal(b1-3/4)[Fuc(a1-3/4)]GlcNAc(b1-6)]**GalNAc |  |  |  |  |  | 1.7 |  |  |  |  |  |  |  |
| 1228.4 | NeuAc(a2-3)Gal(b1-3/4)[Fuc(a1-3/4)]GlcNAc(b1-3/6)[GlcNAc(b1-3/6)]GalNAc |  |  |  |  |  |  |  | 3.0 |  |  |  |  |  |
| 1244.4 | NeuAc(a2-3/6)Gal(b1-3/4)GlcNAc(b1-3)**[Gal(b1-3/4)GlcNAc(b1-6)]**GalNAc |  |  |  |  |  | 10.5 |  |  |  |  |  |  |  |
| 1244.4 | NeuAc(a2-3/6)Gal(b1-3/4)GlcNAc(b1-3/6)[Gal(b1-3/4)GlcNAc(b1-3/6)]GalNAc |  |  |  |  | 4.2 |  |  | 0.8 | 2.7 | 3.3 | 13.1 |  |  |
| 1244.4 | NeuAc(a2-3/6)Gal(b1-3/4)GlcNAc(b1-3)**[Gal(b1-3/4)GlcNAc(b1-6)]**GalNAc |  | 7.4 |  |  |  |  |  |  |  |  |  |  |  |
| 1332.3 | [#]NeuAc(a2-3/6)Gal-- | 6.6 |  |  |  | 8.4 |  |  |  | 4.5 |  |  |  |  |
| 1332.3 | NeuAc(a2-3/6)Gal(b1-3)**[NeuAc(a2-3/6)Gal(b1-3/4)GlcNAc(b1-6)]**GalNAc |  |  | 5.9 |  |  |  |  |  |  | 4.2 |  |  |  |
| 1333.4 | NeuAc(a2-3/6)Gal(b1-3)**[Fuc(a1-2)Gal(b1-3/4)[Fuc(a1-3/4)]GlcNAc(b1-6)]**GalNAc |  |  |  |  | 2.2 |  |  |  |  |  |  |  |  |
| 1333.4 | Fuc(a1-2)Gal(b1-3)**[NeuAc(a2-3)Gal(b1-3/4)[Fuc(a1-3/4)]GlcNAc(b1-6)]**GalNAc |  | 3.3 |  |  | 5.2 |  |  |  |  | 3.3 |  |  |  |
| 1390.4 | Fuc(a1-3/4)[Gal(b1-3/4)]GlcNAc(b1-3)**[NeuAc(a2-3/6)Gal(b1-3/4)GlcNAc(b1-6)]**GalNAc |  | 21.9 | 10.6 |  |  |  |  |  |  | 10.3 |  |  |  |
| 1390.4 | Fuc(a1-3/4)[Gal(b1-3/4)]GlcNAc(b1-3/6)[NeuAc(a2-3/6)Gal(b1-3/4)GlcNAc(b1-3/6)]GalNAc |  |  |  |  |  |  |  | 17.1 |  |  |  |  |  |
| 1390.4 | Gal(b1-3/4)GlcNAc(b1-3)**[NeuAc(a2-3)Gal(b1-3/4)[Fuc(a1-3/4)]GlcNAc(b1-6)]**GalNAc |  |  |  |  | 11.6 |  |  |  |  |  | 19.8 |  | 20.3 |
| 1390.4 | NeuAc(a2-3)Gal(b1-3/4)[Fuc(a1-3/4)]GlcNAc(b1-3)**[Gal(b1-3/4)GlcNAc(b1-6)]**GalNAc |  |  |  |  |  |  |  |  | 13.8 | 3.1 |  |  |  |
| 1390.4 | NeuAc(a2-3)Gal(b1-3/4)[Fuc(a1-3/4)]GlcNAc(b1-3/6)[Gal(b1-3/4)GlcNAc(b1-3/6)]GalNAc |  |  |  | 6.4 |  |  |  |  |  |  |  |  |  |
| 1390.4 | Fuc(a1-2)Gal(b1-3)GlcNAc(b1-3)**[NeuAc(a2-3/6)Gal(b1-3/4)GlcNAc(b1-6)]**GalNAc |  | 3.4 |  |  |  |  | 13.5 |  | 1.2 |  |  | 12.1 |  |
| 1390.4 | Fuc(a1-2)Gal(b1-4)GlcNAc(b1-3)**[NeuAc(a2-3/6)Gal(b1-3/4)GlcNAc(b1-6)]**GalNAc |  |  | 3.2 |  |  |  |  |  |  |  |  |  |  |
| 1478.4 | NeuAc(a2-3/6)Gal(b1-3)**[NeuAc(a2-3)Gal(b1-3/4)[Fuc(a1-3/4)]GlcNAc(b1-6)]**GalNAc |  | 5.8 |  |  |  |  |  | 3.4 |  |  |  |  |  |
| 1478.4 | [#]NeuAc(a2-3)Gal(b1-3/4)[Fuc(a1-3/4)]GlcNAc-- |  |  |  | 12.1 |  |  |  |  |  |  |  |  |  |
| 1536.7 | Fuc(a1-3/4)[Gal(b1-3/4)]GlcNAc(b1-3)**[NeuAc(a2-3)Gal(b1-3/4)[Fuc(a1-3/4)]GlcNAc(b1-6)]**GalNAc |  | 5.1 |  |  |  |  |  |  |  |  |  |  |  |
| 1536.7 | Fuc(a1-2)Gal(b1-3/4)[Fuc(a1-3/4)]GlcNAc(b1-3)**[NeuAc(a2-3/6)Gal(b1-3/4)GlcNAc(b1-6)]**GalNAcol |  | 2.0 |  |  |  |  |  |  |  |  |  |  |  |
| 1536.7 | Fuc(a1-2)Gal(b1-3/4)GlcNAc(b1-3)**[NeuAc(a2-3)Gal(b1-3/4)[Fuc(a1-3/4)]GlcNAc(b1-6)]**GalNAc |  |  |  |  |  |  | 4.5 |  | 2.6 |  |  |  |  |
| 1536.7 | NeuAc(a2-3)Gal(b1-3/4)[Fuc(a1-3/4)]GlcNAc(b1-3/6)[Fuc(a1-2)Gal(b1-3/4)GlcNAc(b1-3/6)]GalNAc |  |  | 4.9 |  |  |  |  |  |  | 3.9 |  |  |  |
| 1536.7 | NeuAc(a2-3)Gal(b1-3/4)[Fuc(a1-3/4)]GlcNAc(b1-3/6)[Gal(b1-3/4)[Fuc(a1-3/4)]GlcNAc(b1-3/6)]GalNAc |  |  |  |  |  |  |  | 3.9 |  |  |  |  |  |
| 1552.3 | [#]NeuAc(a2-3)Gal(b1-3/4)[Fuc(a1-3/4)]GlcNAc-- |  |  |  | 4.1 |  |  |  |  |  |  |  |  |  |
| 1681.3 | NeuAc(a2-3)Gal(b1-3/4)[Fuc(a1-3/4)]GlcNAc(b1-3/6)[NeuAc(a2-3/6)Gal(b1-3/4)GlcNAc(b1-3/6)]GalNAc |  |  |  |  |  |  |  | 7.4 |  |  |  |  |  |
| 1681.3 | NeuAc(a2-3)Gal(b1-3/4)[Fuc(a1-3/4)]GlcNAc(b1-3)**[NeuAc(a2-3/6)Gal(b1-3/4)GlcNAc(b1-6)]**GalNAc |  | 2.6 |  |  |  |  |  |  |  |  |  |  |  |
| 1828.0 | NeuAc(a2-3)Gal(b1-3/4)[Fuc(a1-3/4)]GlcNAc(b1-3) **[NeuAc(a2-3)Gal(b1-3/4)[Fuc(a1-3/4)]GlcNAc(b1-6)]**GalNAc |  |  |  |  |  |  |  | 2.4 |  |  |  |  |  |
|  | Sulfated |  |  |  |  |  |  |  |  |  |  |  |  |  |
| 1179.2 | Gal(b1-3/4)GlcNAc(b1-3)**[HSO3(-3/6)Gal(b1-3/4)[Fuc(a1-3/4)]GlcNAc(b1-6)]**GalNAc |  |  |  |  |  | 2.2 |  |  |  |  |  |  |  |
| 1179.2 | HSO3(-3/6)Gal(b1-3/4)GlcNAc(b1-3)**[Fuc(a1-2)Gal(b1-3/4)GlcNAc(b1-6)]**GalNAc |  |  |  |  |  |  |  |  |  | 1.224 |  |  |  |

* Bold letters indicate the C-6 branch of the core GalNAc. [#] Only a portion of the structure was assigned. M, neutral reduced mass; Structure, assigned structure or substructure. Assumptions: a) All deoxyhexoses are fucose. b) All hexoses are galactose. c) The core HexNAcol is a GalNAcol. d) All HexNAcs are GlcNAc except for those in sequences assigned as blood group A epitopes, which are GalNAc.