Supplementary Information

Reactive two-component monolayers template bottom-up assembly of nanoparticle arrays on HOPG

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STM, TOF-MS and ML Formation and Reaction Protocols.

STM Data Collection.

STM data was collected using freshly cut 80/20 Pr/Ir tips. Bias voltage between -800 mV to -1400 mV, set currents between 10 pA to 100 pA and scan rates between 0.5 Hz to 7 Hz were employed. STM data was collected on a VEECO MS-10 STM instrument. The acquired images were flattened.

TOF-MS Data Collection.

After preparation of a given monolayer (ML) sample on HOPG, a thin film of THAP (2,4,6-trihydroxyacetophenone) was applied by drop casting 15 µL of 1.7 mg/ml (30/70 acetone/octane) solution onto the monolayer-bearing side of HOPG. The THAP solution was allowed to dry in air, yielding a fairly uniform THAP coating on HOPG. (The THAP thin film was applied to enhance TOF-MS intensities.) TOF-MS data was collected on a Brucker Daltonics Autoflex MALDI-TOF mass spectrometer (Billerica, MA) running in linear mode. The modified sample holder (MTP PAC Frame) used to hold the HOPG substrate was reported previously. The HOPG substrate was irradiated using the instrument's Smartbeam^{TM}-II Nd:YAG laser (355 nm) with 6_ultra laser profile operating at 1 kHz.[1] Data was collected in positive ion mode using 500 laser shots at 47% or 55% laser power (referred to as 47LP and 55LP, respectively). 47LP was used as it generated reproducible and non-saturating **1^{OH}** and **2** molecular ion intensities (< 75000) while minimizing molecular ion fragmentation. 55LP gave acceptable signals from the heavier surface species; use of higher LP increased their fragmentation. Other parameters are listed as follows: TOF accelerating voltage 19.45 kV, grid voltage 18.1 kV, pulsed ion extraction time 90 µs. Instrument calibration was carried out using Brucker Daltonics Peptide Calibration Standard II applied to an HOPG substrate in the modified sample holder.

Formation and Reactions Protocols for $1^{OH}1^{OH}22$ monolayer samples on HOPG.

0) Preparation of **$1^{OH}1^{OH}22$** monolayer.

6 µL of **1^{OH} : 2** = 2:3 (total concentration = 10 µM) phenyloctane solution was drop cast on a freshly cleaved 12 mm x 12 mm HOPG substrate. After 15 minutes, the HOPG (monolayer side) was rinsed dropwise with 25 µL phenyloctane and then three times with 25 µL hexanes. The HOPG substrate was allowed to dry in air. Such a ML sample is referred to as a 'rinsed **$1^{OH}1^{OH}22$** monolayer sample'.

1) Crosslinking of **$1^{OH}1^{OH}22$** monolayers with 3,3'-dithiobis-propanoic acid (DTBP).

A rinsed **$1^{OH}1^{OH}22$** monolayer sample was incubated in 1 ml N,N-dimethylformamide (DMF) solution containing 20 mM DTBP, 40 mM EDC (1-(3-dimethylaminopropyl)-3-ethylcarbodiimide hydrochloride) and 40 mM DMAP (4-dimethylaminopyridine) at room temperature for 12 hours. After the reaction, the HOPG (monolayer side) was rinsed with 0.25 ml DMF twice, 0.5 ml water once and 0.1 ml ethanol twice. The sample was then allowed to dry in air.

2) Dithiothreitol (DTT) reduction of DTBP-crosslinked **$1^{OH}1^{OH}22$** monolayers.

A monolayer sample prepared as in 1) was incubated in 1.5 ml of 50 mM DTT aqueous solution buffered with 200 mM tris(hydroxymethyl)aminomethane buffer (namely Tris buffer, pH = 7.8) at room temperature for 3 hours. After DTT reduction, the HOPG (monolayer side) was rinsed with 0.25 ml water twice and allowed to dry in air.

3) Reaction of **$1^{OH}1^{OH}22$** monolayers with DTBP mono ethyl ester (3-((3-ethoxy-3-oxopropyl)disulfaneyl)-propanoic acid).

A rinsed **$1^{OH}1^{OH}22$** monolayer sample was incubated in 1 ml of DMF containing 40 mM DTBP mono ethyl ester, 40 mM EDC and 40 mM DMAP at room temperature for 12 hours. After reaction, the HOPG (monolayer side) was rinsed with 0.25 ml DMF twice, 0.5 ml water once and 0.1 ml ethanol twice. The sample was then allowed to dry in air.

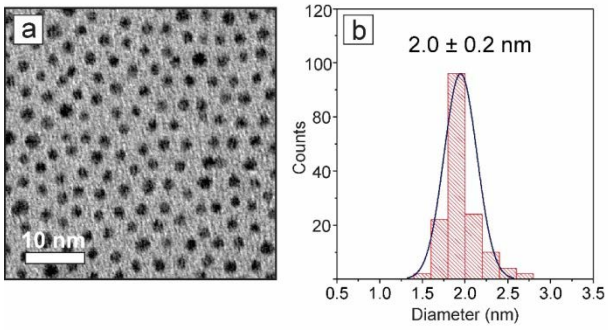
4) DTT reduction of DTBP mono ethyl ester reacted **$1^{OH}1^{OH}22$** monolayers

A monolayer sample prepared as in 3) was incubated in 1.5 ml of 50 mM DTT aqueous solution buffered with 200 mM Tris (pH = 7.8) at room temperature for 3 hours. After DTT reduction, the HOPG (monolayer side) was rinsed with 0.25 ml water twice and allowed to dry in air.

Synthesis and Characterization of AuNP

Chemicals - Hydrogen tetrachloroaurate (III) hydrate ($HAuCl_4 \cdot 3H_2O$), borane tert-butylamine complex (TBAB, 97%), oleylamine (OAm, technical grade, 70%), 1,2,3,4-tetrahydronaphthalene (tetralin, anhydrous, 99%) were purchased from Sigma Aldrich.

Synthesis of Au nanoparticles - Au nanoparticles (NPs) were synthesized using a modified literature method.[2] Typically, $HAuCl_4 \cdot 3H_2O$ (110 mg) was dissolved in a mixture of tetralin (10 mL) and OAm (5mL). The mixture was heated to 35^{o}C and stirred under flowing N_2 for 10 min. A reducing solution containing 0.5 mmol of TBAB, tetralin (1 mL), and OAm (1 mL) was mixed by sonication and quickly injected into the precursor solution. The reaction mixture was then allowed to react at 35 ^{o}C for 2 hours before acetone (30 mL) was added to precipitate the Au NPs. The Au NPs were collected by centrifugation (8000 rpm, 5 min), washed with acetone and redispersed in hexane before further use.



Characterization of synthesized Au NPs. (a) A TEM image of synthesized Au NPs. (b) Size distribution histogram of the Au NPs with an average diameter of 2.0 ± 0.2 nm.

Syntheses of 1^{OH} and 2

Part I Abbreviations

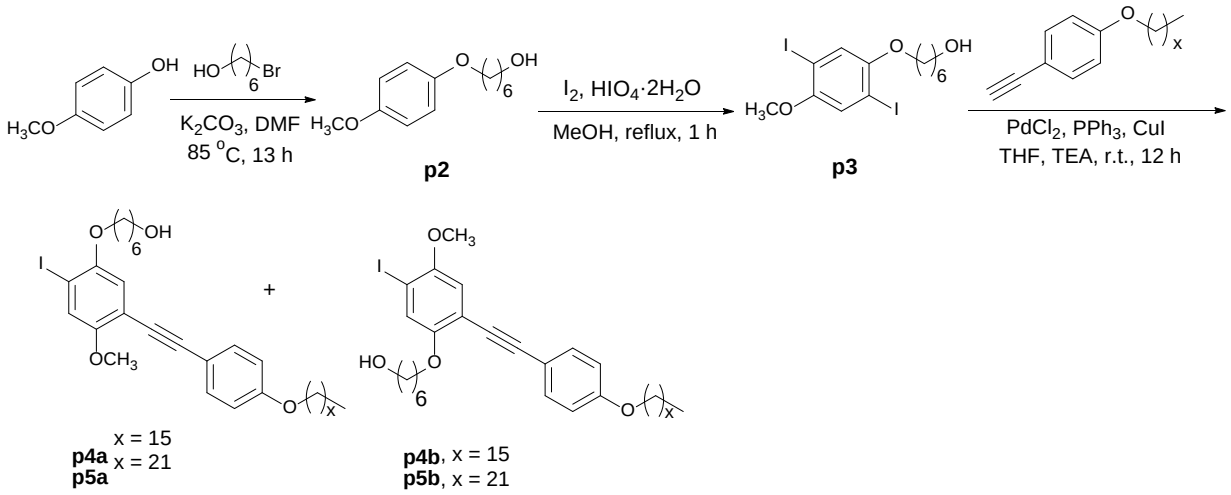
DCM = dichloromethane; DMF = dimethylformamide; DMSO = dimethyl sulfoxide; EA = ethyl acetate; TEA = triethylamine; THF = tetrahydrofuran; Ts = 4-toluenesulfonyl.

Part II General Remarks of Synthetic Protocols

Commercial reagents were used as received without further purification. Synthetic compounds including 6-hydroxyhexyl 4-methylbenzenesulfonate [3], 1,4-bis(hexyloxy)-2,5-diiodobenzene[3], 1-ethynyl-4-(hexadecyloxy)benzene[3], 3,3'-dithiobispropanoic mono-ethyl ester[4], 1-(docosyloxy)-4-ethynylbenzene[5], **p7**[3] and **p8**[3] were synthesized as previously reported. Moisture sensitive and air-sensitive reactions were carried out in flame-dried glassware with anhydrous solvents under argon atmosphere. Deoxygenated anhydrous solvents such as THF, DMF and TEA were obtained from a solvent column system. Reaction progress was monitored by thin layer chromatography (TLC) plates, visualized by UV, ninhydrin stain and phosphomolybdic acid (PMA) stain. Purification was performed with flash column chromatography when necessary (230-400 mesh silica gel, 60 Å, Whatman). Nuclear magnetic resonance (NMR) experiment were performed on Bruker 400 MHz and 600 MHz (proton) spectrometers. Spectra were referenced to the mono-protic solvent residual peak. Chemical shifts are reported in parts per million (ppm) upfield of tetramethylsilane (TMS).

Electron impact ionization (EI), electrospray ionization and matrix-assisted laser desorption ionization (MALDI) methods were used to obtain the mass spectra with Agilent Technologies 5973N, Agilent Technologies 6530 Accurate-Mass Q-TOF LC/MS and the Bruker autoflex speed MALDI-TOF mass spectrometers, respectively.

Part III Syntheses of Precursors p5 and p6



Scheme S1. Syntheses of **p4** and **p5**.

6-(4-methoxyphenoxy)hexan-1-ol (p2).

4-methoxyphenol (1.116 g, 9 mmol), 6-bromohexanol (1.086 g, 6 mmol), potassium carbonate (2.48 g, 18 mmol) were stirred in 18 ml DMF at 85 ^{o}C for 13 hours under argon atmosphere. The reaction mixture was then cooled down to room temperature. The mixture was washed with water, brine and extracted with EA. The organic layer was dried over sodium sulfate and concentrated under reduced pressure. The crude mixture was purified by column chromatography (30/70 EA/hexanes) to afford compound **p2** (622 mg, 2.8 mmol, yield 47%) as a white solid. ^{1}H NMR (600 MHz, $CDCl_3$): δ 6.85 (s, 4H), 3.93 (t, *J* = 6.5 Hz, 2H), 3.79 (s, 3H), 3.69 (q, *J* = 6.1 Hz, 2H), 1.80 (p, *J* = 7.0 Hz, 2H), 1.65-1.44 (m, 6H), 1.28-1.25 (m, 1H); ^{13}C NMR (151 MHz, $CDCl_3$): δ 153.69, 153.24, 115.42, 114.62, 68.49, 62.95, 55.76, 32.71, 29.36, 25.91, 25.56.

6-(2,5-diiodo-4-methoxyphenoxy)hexan-1-ol (p3).

H_5IO_6 (0.51 g, 1.9 mmol) and iodine (0.988 g, 3.9 mmol) were mixed in 15 ml methanol and stirred for 15 minutes in a 50 ml round bottom flask. **p2** (622 mg, 2.8 mmol) was added to the flask in 10 ml methanol, and the reaction mixture was refluxed for 1 hour. The mixture was then cooled to room temperature and was quenched with a large excess of saturated sodium thiosulfate aqueous solution followed by extraction with EA. The solvent was removed under reduced pressure. The crude product was subjected to second round of identical reaction with 70% of the initial amount of iodine and periodic acid. After work-up following the same protocol, the crude mixture was purified by column chromatography (30/70 EA/hexanes) to afford compound **p3** (590 mg, 1.2 mmol, yield 45%) as a solid. The compound was used without further purification. ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.21 (s, 1H), 7.20 (s, 1H), 3.97 (t, *J* = 6.3 Hz, 2H), 3.85 (s, 3H), 3.70 (q, *J* = 5.8 Hz, 2H), 1.85 (q, *J* = 6.9 Hz, 2H), 1.67-1.45 (m, 6H), 1.28-1.27 (m, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 153.28, 152.90, 122.94, 121.47, 86.34, 85.42, 70,20, 62.93, 57.18, 32.70, 29.10, 25.90, 25.43.

6-(5-((4-(hexadecyloxy)phenyl)ethynyl)-2-iodo-4-methoxyphenoxy)hexan-1-ol (p4a) and 6-(2-((4-(hexadecyloxy)phenyl)ethynyl)-5-iodo-4-methoxyphenoxy)hexan-1-ol (p4b).

p3 (371 mg, 0.78 mmol), 1-ethynyl-4-(hexadecyloxy)benzene (205 mg, 0.60 mmol), $PdCl_2$ (7 mg, 0.04 mmol), CuI (8 mg, 0.04 mmol), triphenyl phosphine (31 mg, 0.12 mmol), 3 ml THF and 0.75 ml TEA were loaded into a 5 ml microwave tube that was flame-dried and protected by argon atmosphere. The reaction mixture was stirred at room temperature for 12 hours. The reaction mixture was quenched with saturated ammonium chloride aqueous solution. The mixture was extracted with DCM and washed with water and with brine. The organic layer was dried over anhydrous sodium sulfate. The solution was concentrated under reduced pressure. The crude product was purified by column chromatography (25/75 EA/Hexanes) to afford **p4a** (135 mg, 0.20 mmol, yield 33%) as yellow solid and **p4b** (76 mg, 0.11 mmol, yield 18%) as yellow solids.

p4a. ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.50 (d, *J* = 8.5 Hz, 2H), 7.30 (s, 1H), 6.94 (s, 1H), 6.88 (d, *J* = 8.5 Hz, 2H), 4.01-3.97 (m, 4H), 3.88 (s, 3H), 3.70 (t, *J* = 6.5 Hz, 2H), 1.89-1.78 (m, 4H), 1.68-1.56 (m, 4H), 1.51-1.28 (m, 28H), 0.90 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$): δ 159.36, 154.44, 151.81, 133.12, 122.11, 116.35, 114.95, 114.49, 113.50, 94.55, 86.92, 83.83, 69.94,

68.09, 62.96, 56.68, 32.71, 31.94, 29.71, 29.67, 29.61, 29.58, 29.40, 29.38, 29.20, 29.15, 26.03, 25.94, 25.45, 22.71, 14.14.

p4b. ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.47 (d, *J* = 8.3 Hz, 2H), 7.32 (s, 1H), 6.93 (s, 1H), 6.89 (d, *J* = 8.4 Hz, 2H), 4.03-3.98 (m, 4H), 3.87 (s, 3H), 3.65 (t, *J* = 6.5 Hz, 2H), 1.89-1.78 (m, 4H), 1.64-1.57 (m, 4H), 1.50-1.28 (m, 28H), 0.91 (t, *J* = 6.8 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$): δ 159.35, 154.21, 152.33, 132.98, 124.10, 115.12, 114.54, 114.23, 94.57, 85.90, 84.05, 69.83, 68.11, 62.90, 56.95, 32.70, 31.94, 29.71, 29.68, 29.62, 29.59, 29.42, 29.38, 29.25, 29.21, 26.04, 25.88, 25.50, 22.71, 14.15.

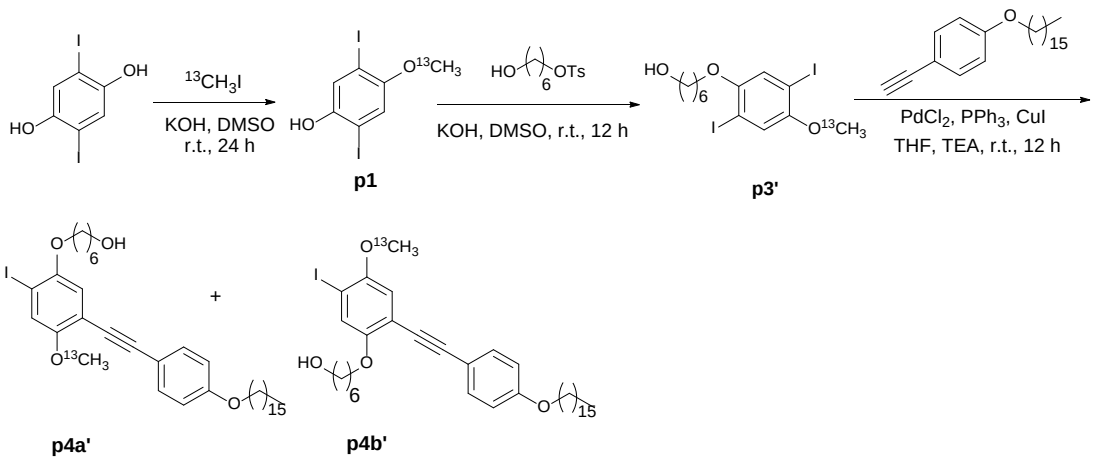
6-(5-((4-(docosyloxy)phenyl)ethynyl)-2-iodo-4-methoxyphenoxy)hexan-1-ol (p5a) and 6-(2-((4-(docosyloxy)phenyl)ethynyl)-5-iodo-4-methoxyphenoxy)hexan-1-ol (p5b).

p3 (371 mg, 0.78 mmol), 1-(docosyloxy)-4-ethynylbenzene (256 mg, 0.60 mmol), $PdCl_2$ (7 mg, 0.04 mmol), CuI (8 mg, 0.04 mmol), triphenyl phosphine (31 mg, 0.12 mmol), 3 ml THF and 0.75 ml TEA were loaded in to a 5 ml microwave tube that was flame-dried and protected by argon atmosphere. The reaction mixture was stirred at room temperature for 12 hours. The reaction mixture was quenched with saturated ammonium chloride aqueous solution. The mixture was extracted with DCM and washed with water and with brine. The organic layer was dried over anhydrous sodium sulfate. The solution was concentrated under reduced pressure. The crude product was purified by column chromatography (25/75 EA/Hexanes) to afford **p5a** (154 mg, 0.20 mmol, yield 33%) as yellow solid and **p5b** (86 mg, 0.11 mmol, yield 18%) as yellow solids.

p5a. ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.50 (d, *J* = 8.8 Hz, 2H), 7.30 (s, 1H), 6.93 (s, 1H), 6.88 (d, *J* = 8.8 Hz, 2H), 4.00-3.97 (m, 4H), 3.88 (s, 3H), 3.69 (t, *J* = 6.6 Hz, 2H), 1.88-1.78 (m, 4H), 1.67-1.56 (m, 4H), 1.50-1.28 (m, 40H), 0.90 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$): δ 159.35, 154.44, 151.81, 133.12, 122.11, 116.34, 114.97, 114.48, 113.51, 94.56, 86.93, 83.86, 69.94, 68.09, 62.92, 56.67, 32.70, 31.95, 29.73, 29.69, 29.63, 29.60, 29.42, 29.39, 29.22, 29.16, 26.04, 25.95, 25.46, 22.72, 14.16.

p5b. ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.47 (d, *J* = 8.8 Hz, 2H), 7.32 (s, 1H), 6.94 (s, 1H), 6.89 (d, *J* = 8.9 Hz, 2H), 4.03-3.97 (m, 4H), 3.87 (s, 3H), 3.65 (t, *J* = 6.6 Hz, 2H), 1.89-1.78 (m, 4H), 1.63-1.57 (m, 4H), 1.50-1.28 (m, 40H), 0.90 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$): δ 159.35, 154.22, 152.33, 132.98, 124.10, 115.13, 114.54, 114.23, 94.57, 85.90, 84.05, 69.82, 68.11, 62.89, 56.95, 32.70, 31.95, 29.72, 29.69, 29.63, 29.60, 29.42, 29.38, 29.26, 29.22, 26.04, 25.88, 25.50, 22.72, 14.15.

Note: see Part V for assignments of isomers **p4a**, **p4b**, **p5a**, **p5b**.



Scheme S2. Synthesis of **p4a'** and **p4b'**.

2,5-diiodo-4-(methoxy-^{13}C)phenol (p1).

Potassium hydroxide (132 mg, 0.235 mmol) and 2,5-Diiodohydroquinone (734 mg, 2 mmol) were loaded into a 20 ml microwave tube under argon atmosphere. After stirring for 30 minutes, Iodomethane-^{13}C (284 mg, 2 mmol) was added by syringe dropwise to the tube at 0 ^{o}C. The reaction mixture was stirred at room temperature for 24 hours. 6 ml 0.5 M HCl aqueous solution was added to quench the reaction, followed by dilution of 15 ml water. The precipitate was isolated by centrifugation and washed with water. The crude product was air-dried and used without further purification.

6-(2,5-diiodo-4-(methoxy-^{13}C)phenoxy)hexan-1-ol (p3′).

6-hydroxyhexyl 4-methylbenzenesulfonate (240 mg, 0.88 mmol), **p1** (221 mg, 0.59 mmol) and potassium hydroxide (36 mg, 0.64 mmol) were stirred in 2 ml dimethyl sulfoxide at room temperature for 12 hours. The mixture was neutralized with 0.6 ml 0.5 M HCl aqueous solution. The mixture was washed with water, brine and extracted with EA. The organic layer was dried over sodium sulfate and concentrated under reduced pressure. The crude mixture was purified by column chromatography (30/70 EA/hexanes) to afford compound **p3′** (203 mg, 0.43 mmol, yield 72%) as a colorless oil.

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.20 (s, 1H), 7.19 (s, 1H), 3.96 (t, *J* = 6.3 Hz, 2H), 3.84 (d, $^{1}J_{CH}$ = 144.7 Hz, 3H, -OCH_3), 3.69 (t, *J* = 6.6 Hz, 2H), 1.84 (p, *J* = 6.9 Hz, 2H), 1.66-1.44 (m, 6H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 153.35, 153.34, 152.96, 123.04, 121.59, 121.55, 86.39, 85.48, 85.46, 70.26, 62.88, 57.21, 32.68, 29.11, 25.90, 25.43. ESI(+)HRMS m/z Calcd for $[M+NH_4]^+$ 494.9723, found 494.9712.

6-(5-((4-(hexadecyloxy)phenyl)ethynyl)-2-iodo-4-(methoxy-^{13}C)phenoxy)hexan-1-ol (p4a′) and 6-(2-((4-(hexadecyloxy)phenyl)ethynyl)-5-iodo-4-(methoxy-^{13}C)phenoxy)hexan-1-ol (p4b′).

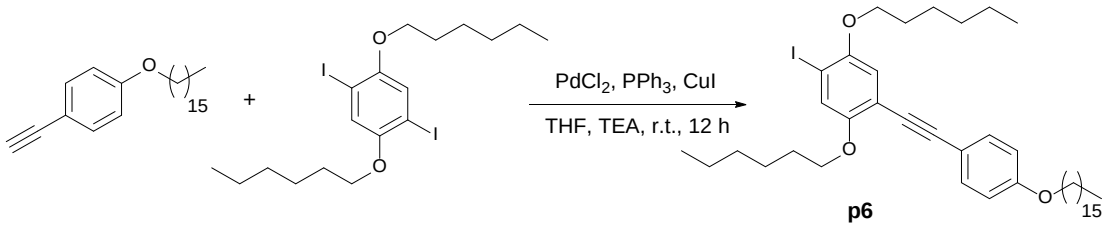
p3′ (200 mg, 0.42 mmol), 1-ethynyl-4-(hexadecyloxy)benzene (143 mg, 0.42 mmol), $PdCl_2$ (7 mg, 0.04 mmol), CuI (8 mg, 0.04 mmol), triphenyl phosphine (31 mg, 0.12 mmol), 2 ml THF and 0.5 ml TEA were loaded in to a 5 ml microwave tube that was flame-dried and protected

by argon atmosphere. The reaction mixture was stirred at room temperature overnight. The reaction mixture was quenched with saturated ammonium chloride aqueous solution. The mixture was extracted with DCM and washed with water and brine. The organic layer was dried over anhydrous sodium sulfate. The solution was concentrated under reduced pressure. The crude product was purified by column chromatography (25/75 EA/Hexanes) to afford **p4a′** (63 mg, 0.091 mmol, yield 22%) as a yellow solid and **p4b′** (43 mg, 0.062 mmol, yield 15%) as a yellow solid.

p4a′. ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.49 (d, *J* = 8.9 Hz, 2H), 7.30 (s, 1H), 6.93 (s, 1H), 6.88 (d, *J* = 8.9 Hz, 2H), 4.01-3.97 (m, 4H), 3.88 (d, $^{1}J_{CH}$ = 140.8 Hz, 3H, -OCH_3), 3.70 (t, *J* = 6.6 Hz, 2H), 1.88-1.78 (m, 4H), 1.67-1.56 (m, 4H), 1.50-1.28 (m, 28H), 0.90 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$): δ 159.39, 154.51, 151.89, 133.11, 122.28, 122.25, 116.45, 115.03, 114.52, 113.62, 94.56, 86.94, 83.86, 70.01, 68.13, 62.94, 56.73, 32.72, 31.93, 29.70, 29.68, 29.66, 29.60, 29.57, 29.40, 29.38, 29.21, 29.17, 26.03, 25.95, 25.46, 22.69, 14.11.

p4b′. ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.47 (d, *J* = 8.8 Hz, 2H), 7.32 (s, 1H), 6.94 (s, 1H), 6.89 (d, *J* = 8.8 Hz, 2H), 4.03-3.98 (m, 4H), 3.87 (d, $^{1}J_{CH}$ = 144.0 Hz, 3H, -OCH_3), 3.65 (t, *J* = 6.5 Hz, 2H), 1.89-1.78 (m, 4H), 1.63-1.57 (m, 4H), 1.50-1.28 (m, 28H), 0.90 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$): δ 159.37, 154.26, 152.41, 132.97, 124.24, 115.17, 114.64, 114.62, 114.57, 114.34, 94.56, 85.92, 85.90, 84.06, 69.90, 68.14, 62.88, 56.97, 32.70, 31.92, 29.69, 29.67, 29.65, 29.59, 29.56, 29.39, 29.35, 29.27, 29.21, 26.02, 25.87, 25.49, 22.68, 14.10.

Note: See Part V for assignments of isomers **p4a'**, **p4b'**, **p4a**, **p4b**, **p5a**, **p5b**.



Scheme S3. Synthesis of **p6**.

1-((4-(hexadecyloxy)phenyl)ethynyl)-2,5-bis(hexyloxy)-4-iodobenzene (p6).

1,4-bis(hexyloxy)-2,5-diiodobenzene (152 mg, 0.29 mmol), 1-ethynyl-4-(hexadecyloxy) benzene (89 mg, 0.26 mmol), $PdCl_2$ (5 mg, 0.03 mmol), CuI (5 mg, 0.03 mmol), triphenyl phosphine (19 mg, 0.09 mmol), 1.2 ml THF and 0.3 ml TEA were loaded in to a 5 ml microwave tube that was flame-dried and protected by argon atmosphere. The reaction mixture was stirred at room temperature for 12 hours. The reaction mixture was quenched with saturated ammonium chloride aqueous solution. The mixture was extracted with DCM and washed with water, brine. The organic layer was dried over anhydrous sodium sulfate. The solution was concentrated under reduced pressure. The crude product was purified by column chromatography (15/85 DCM/Hexanes) to afford **p6** (80 mg, 0.11 mmol, yield 41%) as yellow solid.

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.48 (d, *J* = 8.8 Hz, 2H), 7.32 (s, 1H), 6.93 (s, 1H), 6.89 (d, *J* = 8.8 Hz, 2H), 4.02-3.98 (m, 6H), 1.88-1.79 (m, 6H), 1.57-1.29 (m, 38H), 0.96-0.90 (m, 9H). ^{13}C NMR

(151 MHz, $CDCl_3$): δ 159.28, 154.22, 151.88, 132.99, 123.93, 115.90, 115.26, 114.50, 114.16, 94.42, 86.86, 84.13, 70.11, 69.96, 68.10, 31.95, 31.61, 31.52, 29.72, 29.68, 29.62, 29.59, 29.41, 29.39, 29.22, 29.18, 26.04, 25.77, 25.72, 22.72, 22.65, 22.62, 14.14, 14.07. FAB-MS m/z Calcd for $[M+H]^+$ 745.40, found 745.

Part IV Synthesis of 1^{OH} and 2

6-(5-((4-(docosyloxy)phenyl)ethynyl)-2-((4-(heptacosa-11,13,20,22-tetrayn-1-yloxy)phenyl)ethynyl)-4-methoxyphenoxy)hexan-1-ol (1^{OH}).

p5a (29 mg, 0.04 mmol), **p7** (18 mg, 0.04 mmol), $PdCl_2$ (1 mg, 0.003 mmol), CuI (1 mg, 0.003 mmol), triphenyl phosphine (2 mg, 0.009 mmol), 0.5 ml THF and 0.2 ml TEA were loaded in to a 1 ml microwave tube that was flame-dried and protected by argon atmosphere. The reaction mixture was stirred at room temperature for 12 hours. The reaction mixture was quenched with saturated ammonium chloride aqueous solution. The mixture was extracted with DCM and washed with water andbrine. The organic layer was dried over anhydrous sodium sulfate. The solution was concentrated under reduced pressure. The crude product was purified by column chromatography (80/20 DCM/Hexanes) to afford **1^{OH}** (30 mg, 0.027 mmol, yield 71%) as a yellow solid.

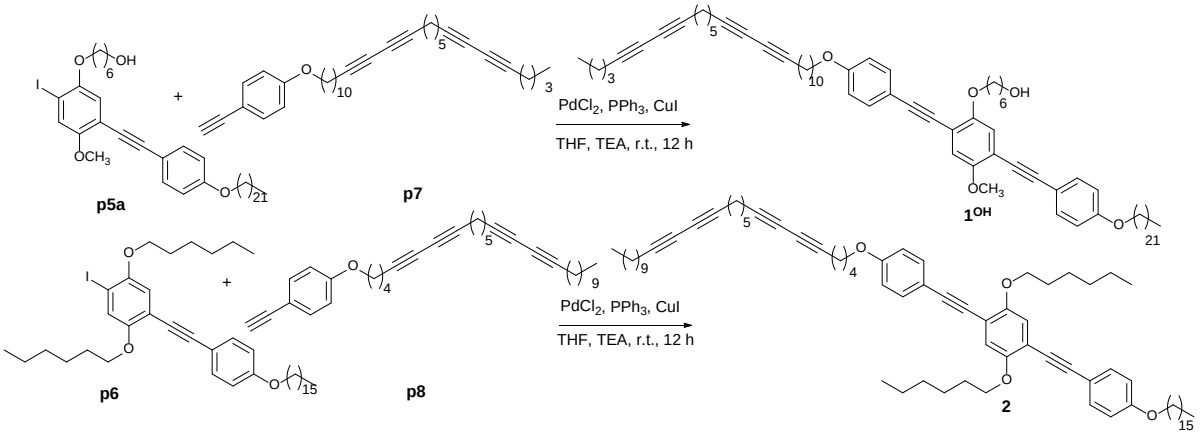
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.51 (d, *J* = 8.8 Hz, 2H), 7.48 (d, *J* = 8.8 Hz, 2H), 7.03 (s, 1H), 7.01 (s, 1H), 6.90-6.87 (m, 4H), 4.06 (t, *J* = 6.4 Hz, 2H), 4.00-3.98 (m, 4H), 3.92 (s, 3H), 3.66 (t, *J* = 6.6 Hz, 2H), 2.29-2.26 (m, 8H), 1.91-1.79 (m, 6H), 1.65-1.28 (m, 68H), 0.94-0.89 (m, 6H). ^{13}C NMR (151 MHz, $CDCl_3$): δ 159.32, 159.31, 153.79, 153.43, 133.14, 133.00, 117.38, 115.28, 115.11, 115.07, 114.54, 114.48, 114.08, 113.31, 95.03, 84.58, 84.34, 77.72, 77.69, 77.11, 69.55, 68.09, 65.50, 65.20, 65.18, 62.93, 56.45, 32.73, 31.94, 30.37, 29.69, 29.67, 29.61, 29.58, 29.50, 29.41, 29.39, 29.37, 29.36, 29.30, 29.22, 29.21, 29.07, 28.84, 28.32, 28.05, 27.86, 26.03, 26.02, 25.92, 25.52, 22.70, 21.92, 19.21, 19.11, 18.89, 14.13, 13.54. MS (MALDI) m/z Calcd for $[M]^+$ 1127, found 1127.

1-((4-(heptacosa-5,7,14,16-tetrayn-1-yloxy)phenyl)ethynyl)-4-((4-(hexadecyloxy)phenyl)ethynyl)-2,5-bis(hexyloxy)benzene (2).

p6 (22 mg, 0.03 mmol), **p8** (18 mg, 0.04 mmol), $PdCl_2$ (1 mg, 0.003 mmol), CuI (1 mg, 0.003 mmol), triphenyl phosphine (2 mg, 0.009 mmol), 0.6 ml THF and 0.15 ml TEA were loaded in to a 1 ml microwave tube that was flame-dried and protected by argon atmosphere. The reaction mixture was stirred at room temperature for 12 hours. The reaction mixture was quenched with saturated ammonium chloride aqueous solution. The mixture was extracted with DCM and washed with water and brine. The organic layer was dried over anhydrous sodium sulfate. The solution was concentrated under reduced pressure. The crude product was purified by column chromatography (30/70 DCM/Hexanes) to afford **2** (10 mg, 0.009 mmol, yield 30%) as a yellow solid.

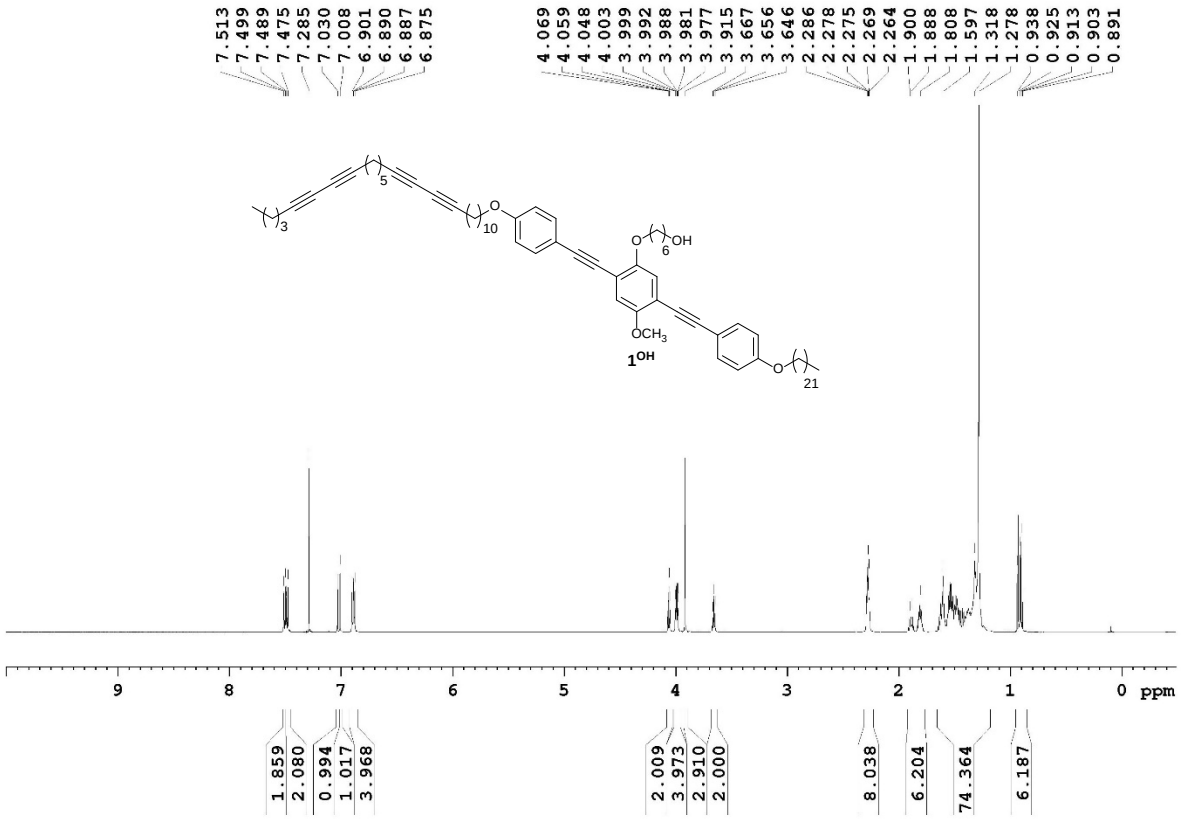
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.48-7.46 (m, 4H), 7.00 (s, 2H), 6.89-6.86 (m, 4H), 4.06-3.97 (m, 8H), 2.37 (t, *J* = 6.9 Hz, 2H), 2.29-2.24 (m, 6H), 1.95-1.73 (m, 10H), 1.55-1.28 (m, 60H), 0.93-0.88 (m, 12H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 159.23, 159.02, 153.48, 133.03, 133.01, 116.82, 115.58, 115.37, 114.48, 114.45, 113.99, 113.91, 94.92, 94.82, 84.71, 84.62, 77.79, 77.23, 76.87, 69.63, 68.08, 67.25, 65.80, 65.51, 65.36, 65.16, 31.95, 31.91, 31.64, 29.72, 29.68,

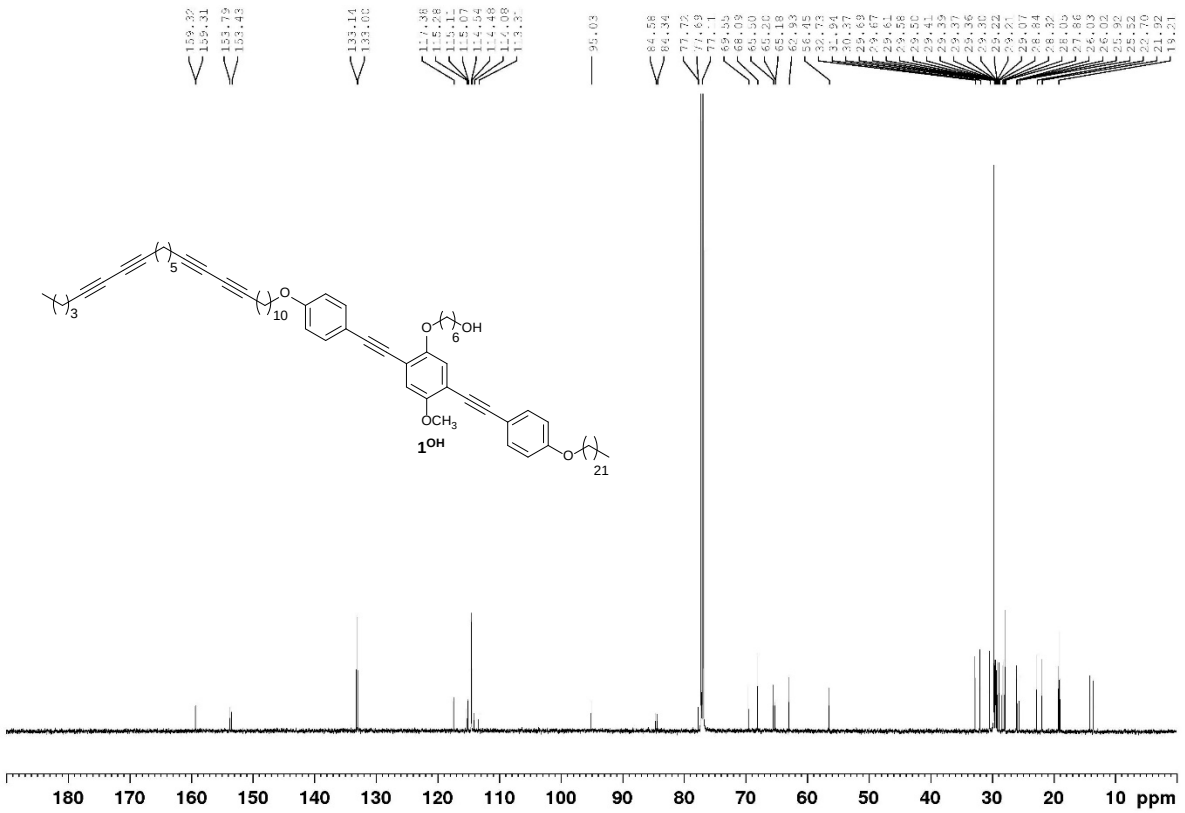
29.62, 29.59, 29.50, 29.42, 29.39, 29.35, 29.33, 29.22, 29.11, 28.87, 28.35, 28.28, 28.06, 27.86, 27.84, 26.04, 25.77, 24.91, 22.70, 22.67, 19.22, 19.12, 18.97, 14.15, 14.08. MS (MALDI) m/z Calcd for $[M]^+$ 1097, found 1097.



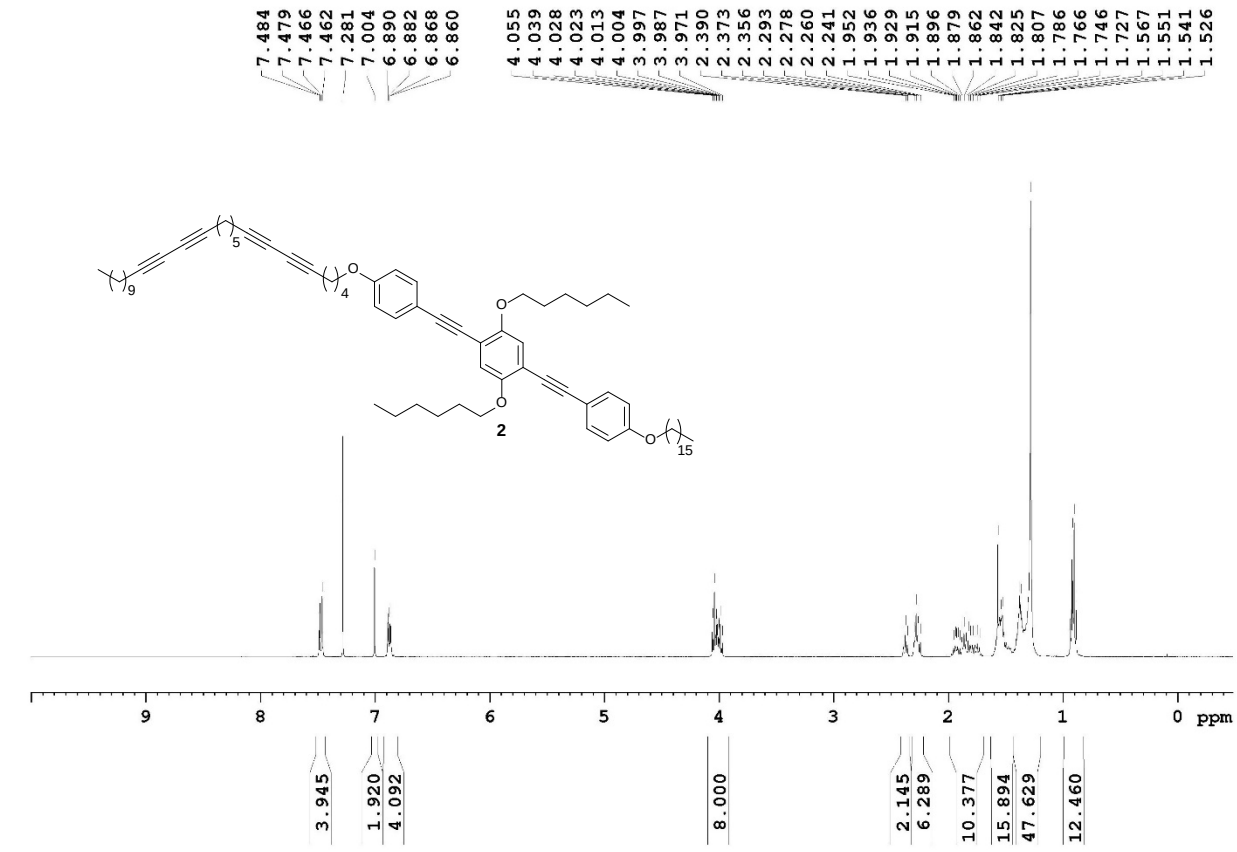
Scheme S4. Synthesis of $\mathbf{1}^{\mathbf{OH}}$ and **2**.

^{1}H and ^{13}C NMR Spectra of $\mathbf{1}^{\mathbf{OH}}$ and **2**.

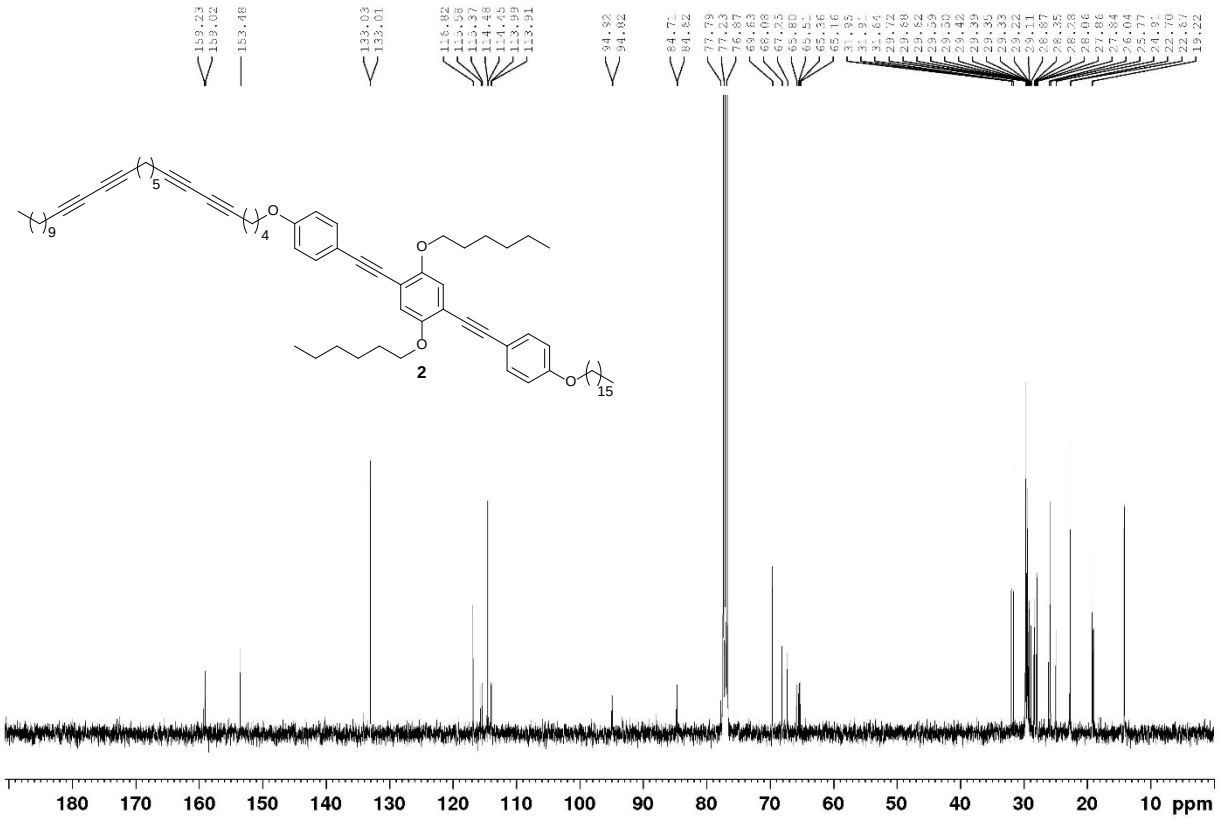



159.32
159.31
153.79
153.43
133.14
133.02
95.03
84.58
84.34
77.72
77.49
77.1
69.55
68.09
65.50
65.20
65.18
62.93
56.45
32.73
31.94
30.37
29.69
29.67
29.61
29.58
29.50
29.41
29.39
29.37
29.36
29.30
29.22
29.21
29.07
28.84
28.32
26.03
26.02
25.92
25.52
22.70
21.92
19.21
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

$\mathbf{1^{OH}}$


7.484
7.479
7.466
7.462
7.281
7.004
6.890
6.882
6.868
6.860
4.055
4.039
4.028
4.023
4.013
4.004
3.997
3.987
3.971
2.390
2.373
2.356
2.293
2.278
2.260
2.241
1.952
1.936
1.929
1.915
1.896
1.879
1.862
1.842
1.825
1.807
1.786
1.766
1.746
1.727
1.567
1.551
1.541
1.526
9 8 7 6 5 4 3 2 1 0 ppm
3.945
1.920
4.092
8.000
2.145
6.289
10.377
15.894
47.629
12.460

2



Part V Assignment of p4 and p5 isomers

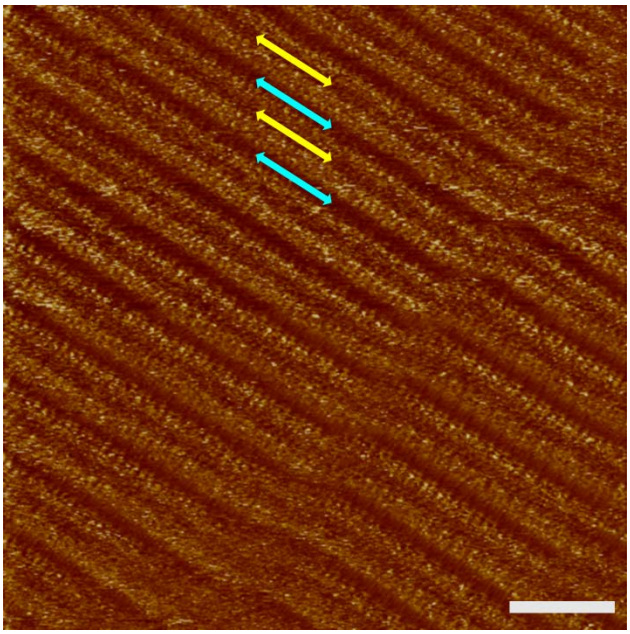
Table S1. ^{13}C resonance of p4, p4′ and p5b isomers in the 80-90 ppm Range.

	Chemical Shift (ppm)	
p4a	86.92	83.83
p4a'	86.94	83.86
p5a	86.93	83.86
p4b	85.90	84.05
p4b'	85.92, 85.90	84.06
p5b	85.90	84.05
Assignment	iodo-connected carbon	*sp*-carbon

There are two isomers of each of **p4** and **p5**, namely **p4a**, **p4b**, **p5a** and **p5b**. NMR characterization failed to provide unambiguous evidence for assignment of these two pairs of isomers. To distinguish between the a and b isomers, methoxy-^{13}C enriched isomers, **p4a' and p4b'**, were synthesized. The assignment of **p4a** and **p4b** was realized by comparing their carbon NMR to those of their methoxy-^{13}C enriched version **p4a′** and **p4b′** (substantial ^{3}J (^{13}C - ^{13}C)) and analyzing the difference of iodide-carbon peaks (between 80 to 90 ppm, there is also

a *sp*-carbon peak in this range). For **p4a′**, the carbon connected the iodide group was four bonds away from the methoxy-^{13}C carbon and was not split by methoxy-^{13}C carbon; for **p4b′**, the carbon connected to the iodide group was three bonds away from the methoxy-^{13}C carbon and correspondingly showed clear splitting due to the methoxy-^{13}C carbon. Consequently, **p4a′** and **p4b′** could be distinguished by whether the iodide-carbon was split. As shown in Table S1, **p4b′** exhibited a doublet for the ^{13}C-iodide peak but **p4a′** did not. Correspondingly, **p4a** and **p4b** were assigned by comparing the chemical shifts of their iodide-substituted carbons: the iodide-carbon of **p4a** appeared downfield to that of **p4b**, which was the same case as that of **p4a′** and **p4b′**. The assignment of **p5a** and **p5b** was accomplished by comparing their iodo-carbon chemical shifts to **p4a′** and **p4b′** in the same manner as that of **p4a** and **p4b**, based on the argument that the structure difference between a hexadecyl (**p4**) and a docosyl (**p5**) group remove from the iodinated ring has negligible impact on chemical shifts of the iodide carbons.

Fig. S1. STM image of ML formed by drop casting **1**^{OH} in phenyloctane on HOPG.



60 nm x 60 nm STM image (drop cast of 25 µM **1**^{OH})
Scale bar (white) = 10 nm

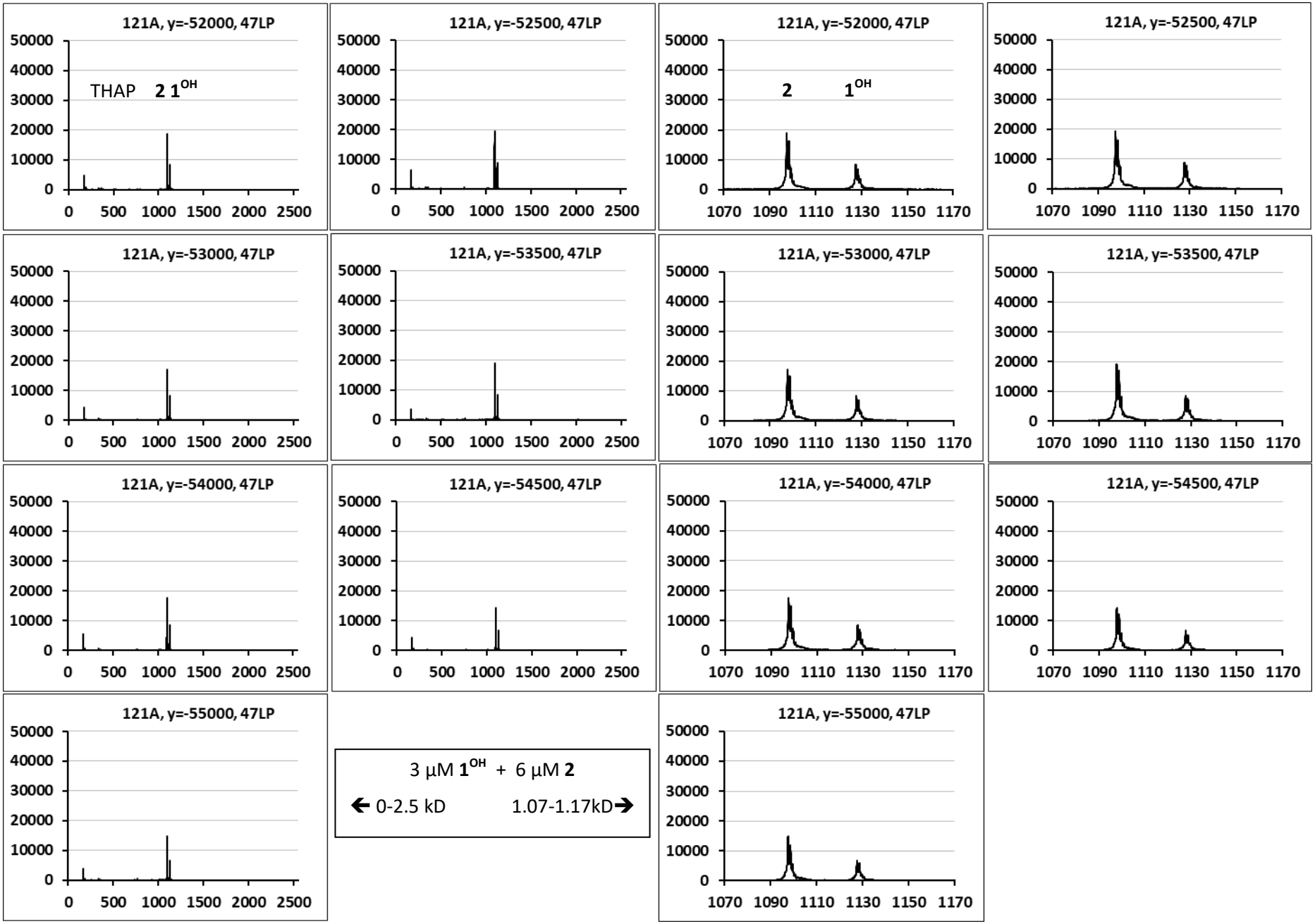
Yellow arrows mark the narrow aliphatic lamellae; Cyan arrow mark the wide aliphatic lamellae (likely [23]).

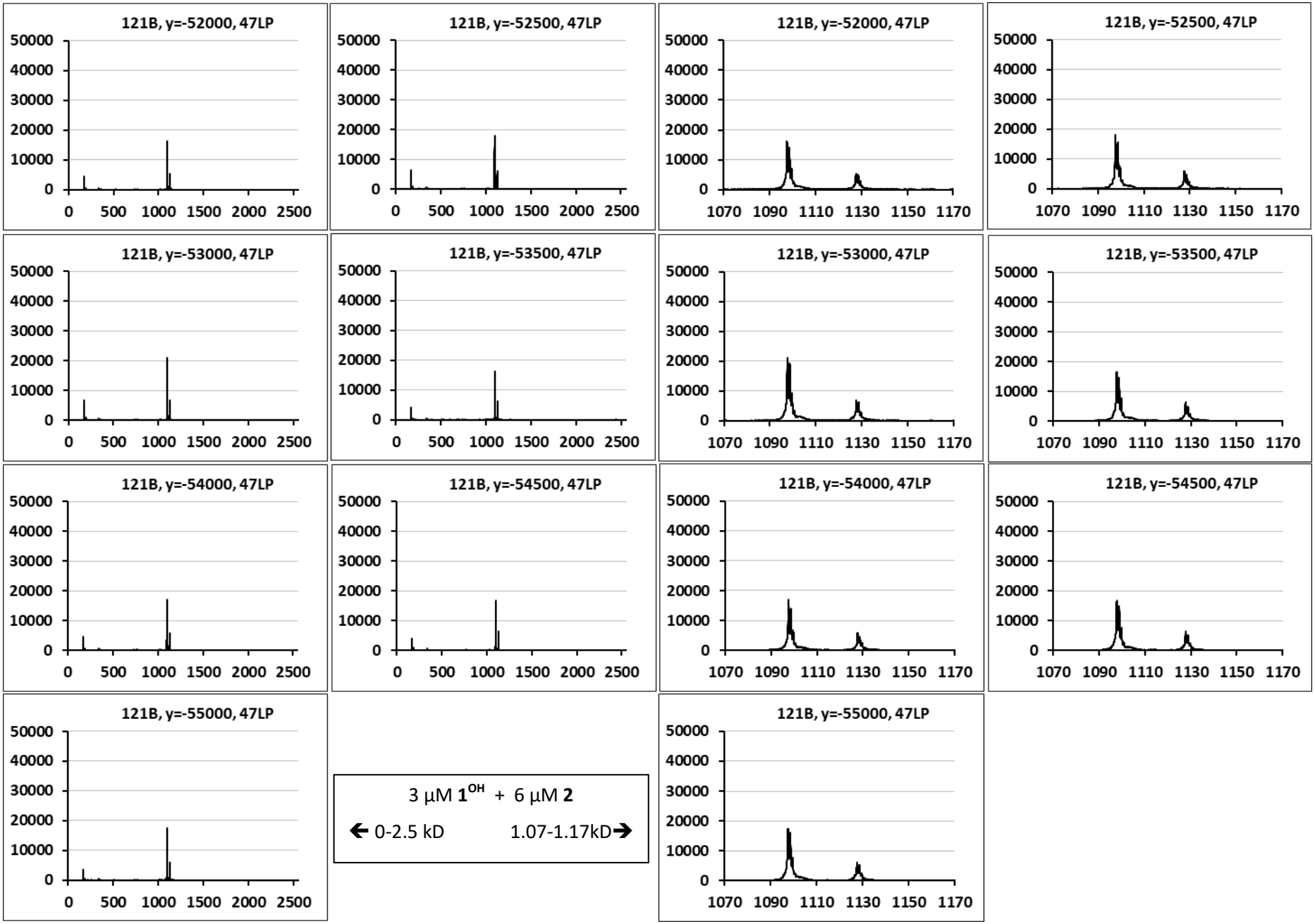
Fig. S2. Summaries and TOF-MS spectra (full range and expansions) of locations sampled to determine relative **1^{OH}** and **2** intensities as a function of **1^{OH} / 2** composition of phenyloctane solution drop cast on HOPG**.** Data collected at LP = 47 after applying a THAP thin film.

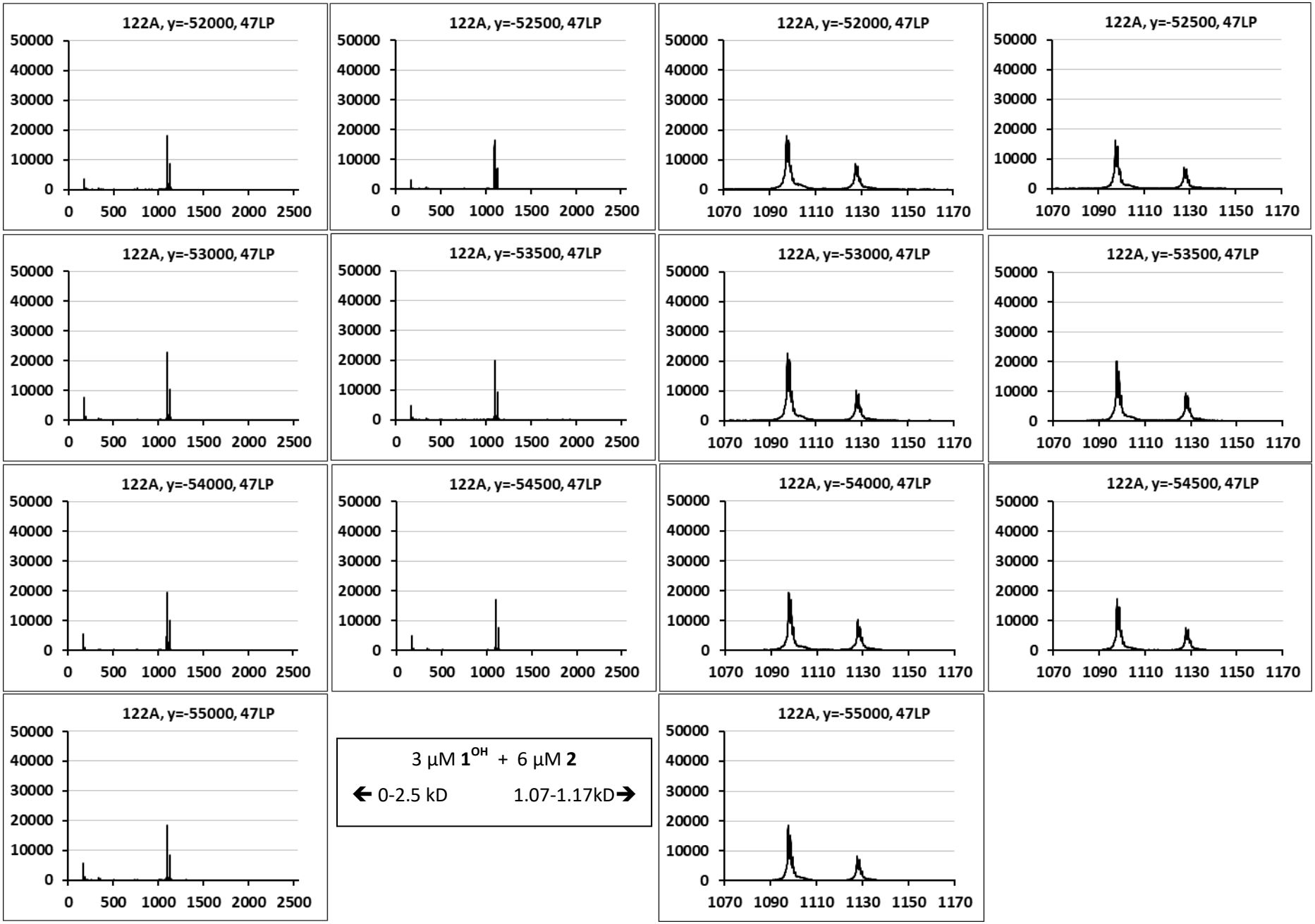
Summary of **1^{OH} / 2** phenyl octane solution with 1 : 2 concentration ratio: TOF-MS ML **1^{OH}** / **2** intensity and intensity ratio statistics from **1^{OH} / 2** phenyl octane solution with 1 : 2 concentration ratio (THAP thin film applied, LP = 47, TOF-MS measured at 14 locations on each of 4 independently prepared samples).

[1^{OH}]	[2]	# Locations	1^{OH} / 2	1^{OH} / 2	1^{OH} / 2	1^{OH} / 2	1^{OH} / 2	1^{OH}	1^{OH}	1^{OH}	1^{OH}	1^{OH}	2	2	2	2	2
µM	µM	(file names)	Max	Min	Avg	σ	σ/Avg	Max	Min	Avg	σ	σ/Avg	Max	Min	Avg	σ	σ/Avg
3	6	14 (121A,B)	0.49	0.32	0.41	0.06	15%	8809	5475	7040	1178	17%	21105	14228	17397	1804	10%
3	6	14 (122A,B)	0.52	0.32	0.42	0.06	14%	10999	6267	8568	1406	16%	27188	16252	20662	3146	15%
3	6	14 (123A,B)	0.50	0.32	0.42	0.06	15%	20450	8390	14257	3938	28%	46059	23234	33850	6509	19%
3	6	14 (124A,B)	0.51	0.34	0.41	0.06	15%	19886	11610	14606	2322	16%	48893	25813	36428	6806	19%
3	6	All	0.52	0.32	**0.41**	**0.06**	**15%**	20450	5475	**11118**	**4151**	**37%**	48893	14228	**27084**	**9609**	**35%**

[1^{OH}]	[2]	# Locations	1^{OH} / 2	1^{OH} / 2	1^{OH} / 2	1^{OH} / 2	1^{OH} / 2	1^{OH}	1^{OH}	1^{OH}	1^{OH}	1^{OH}	2	2	2	2	2
µM	µM	(file names)	Max	Min	Avg	σ	σ/Avg	Max	Min	Avg	σ	σ/Avg	Max	Min	Avg	σ	σ/Avg
4	8	14 (117A,B)	0.50	0.41	0.46	0.03	6%	20748	14208	17815	1933	11%	45386	33573	38777	3620	9%
4	8	14 (118A,B)	0.46	0.34	0.40	0.04	9%	15778	6239	10103	2751	27%	34546	15015	25108	5211	21%
4	8	14 (119A,B)	0.51	0.34	0.43	0.06	13%	16581	7395	11696	3823	33%	35077	20041	26822	5762	21%
4	8	14 (120A,B)	0.44	0.23	0.31	0.06	20%	9339	4617	6913	1456	21%	35061	13922	23152	7642	33%
4	8	All	0.51	0.23	**0.40**	**0.07**	**18%**	20748	4617	**11632**	**4756**	**41%**	45386	13922	**28465**	**8305**	**29%**


121A, y=-52000, 47LP
THAP 2 1OH
121A, y=-52500, 47LP
121A, y=-52000, 47LP
2 1OH
121A, y=-52500, 47LP
121A, y=-53000, 47LP
121A, y=-53500, 47LP
121A, y=-53000, 47LP
121A, y=-53500, 47LP
121A, y=-54000, 47LP
121A, y=-54500, 47LP
121A, y=-54000, 47LP
121A, y=-54500, 47LP
121A, y=-55000, 47LP
3 µM 1OH + 6 µM 2
← 0-2.5 kD
1.07-1.17kD→
121A, y=-55000, 47LP


121B, y=-52000, 47LP
121B, y=-52500, 47LP
121B, y=-52000, 47LP
121B, y=-52500, 47LP
121B, y=-53000, 47LP
121B, y=-53500, 47LP
121B, y=-53000, 47LP
121B, y=-53500, 47LP
121B, y=-54000, 47LP
121B, y=-54500, 47LP
121B, y=-54000, 47LP
121B, y=-54500, 47LP
121B, y=-55000, 47LP
121B, y=-55000, 47LP
3 µM 1OH + 6 µM 2
← 0-2.5 kD
1.07-1.17kD →


122A, y=-52000, 47LP
122A, y=-52500, 47LP
122A, y=-52000, 47LP
122A, y=-52500, 47LP
122A, y=-53000, 47LP
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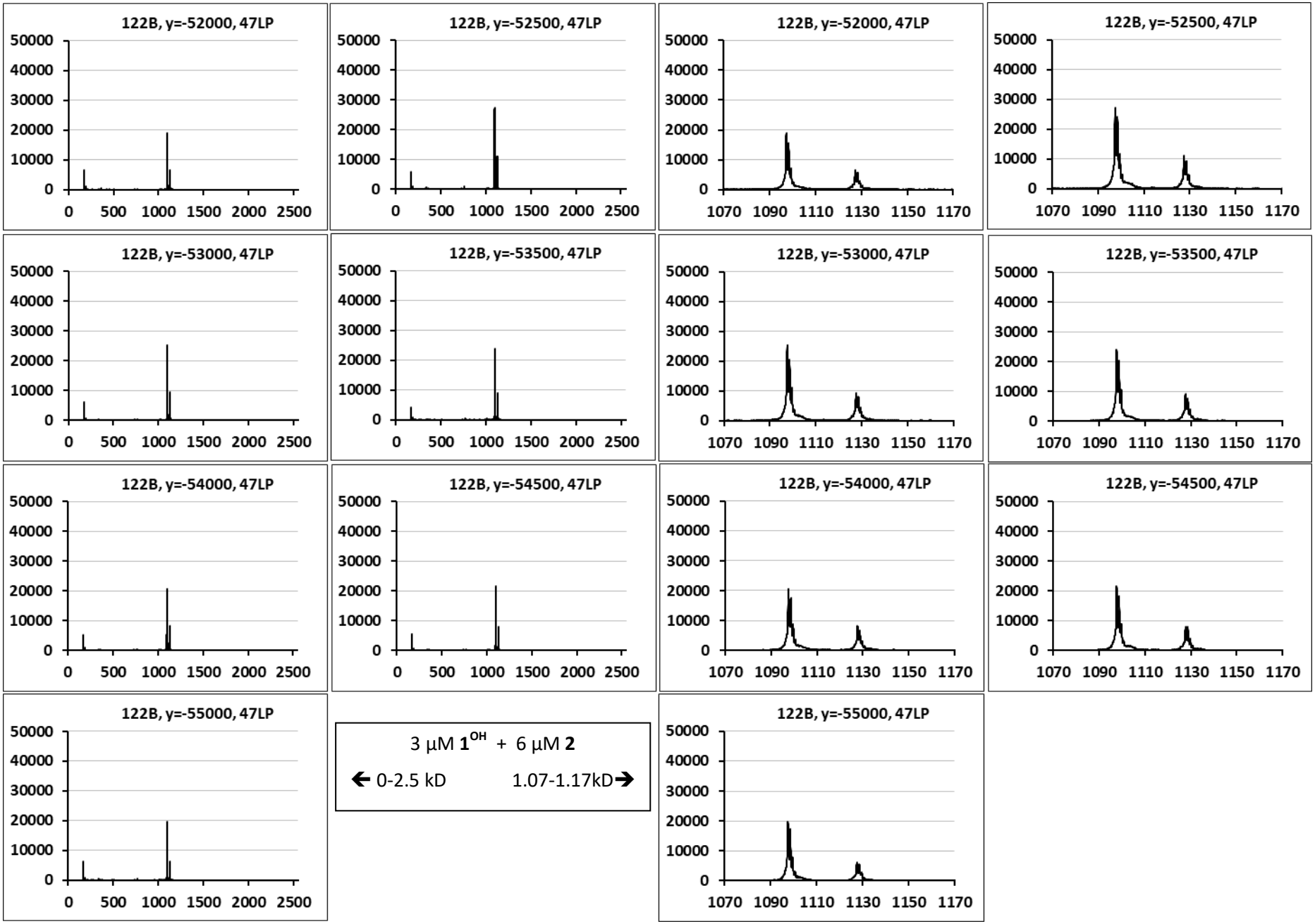
122A, y=-53000, 47LP
122A, y=-53500, 47LP
122A, y=-54000, 47LP
122A, y=-54500, 47LP
122A, y=-54000, 47LP
122A, y=-54500, 47LP
122A, y=-55000, 47LP
3 µM 1OH + 6 µM 2
← 0-2.5 kD
1.07-1.17kD →
122A, y=-55000, 47LP


122B, y=-52000, 47LP
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122B, y=-52000, 47LP
122B, y=-52500, 47LP
122B, y=-53000, 47LP
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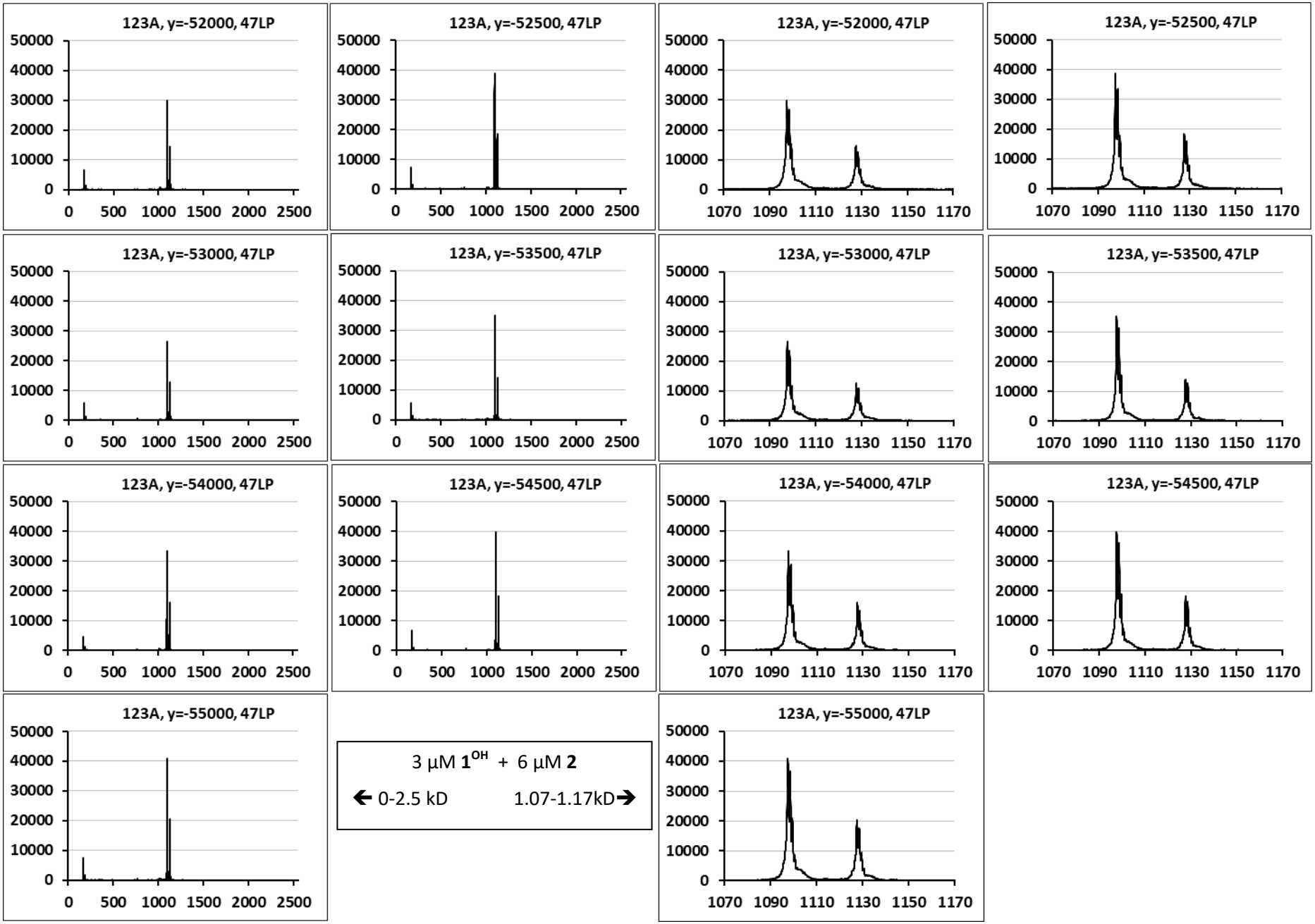
122B, y=-53000, 47LP
122B, y=-53500, 47LP
122B, y=-54000, 47LP
122B, y=-54500, 47LP
122B, y=-54000, 47LP
122B, y=-54500, 47LP
122B, y=-55000, 47LP
3 µM 1OH + 6 µM 2
← 0-2.5 kD
1.07-1.17kD→
122B, y=-55000, 47LP


123A, y=-52000, 47LP
123A, y=-52500, 47LP
123A, y=-52000, 47LP
123A, y=-52500, 47LP
123A, y=-53000, 47LP
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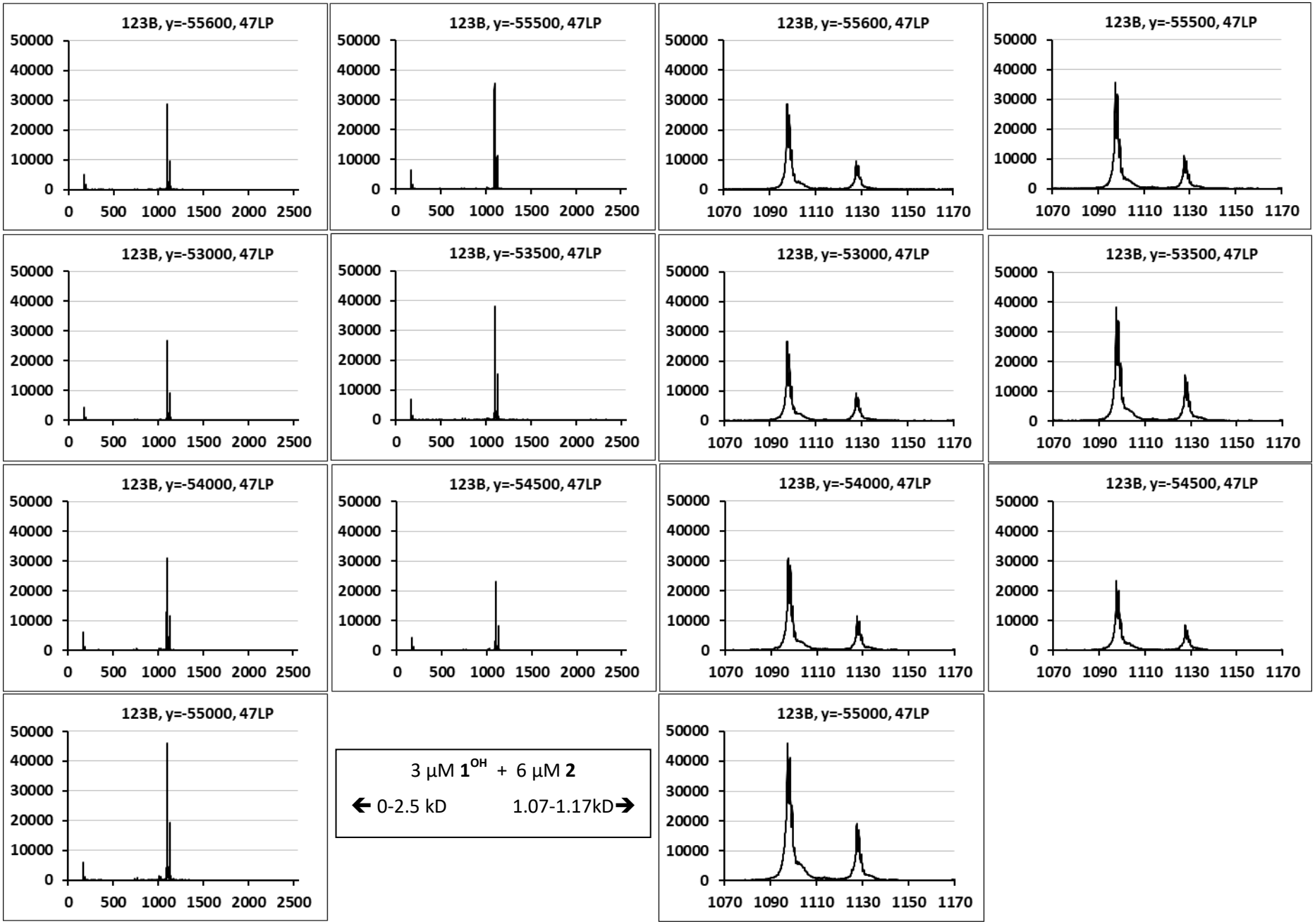
123A, y=-53000, 47LP
123A, y=-53500, 47LP
123A, y=-54000, 47LP
123A, y=-54500, 47LP
123A, y=-54000, 47LP
123A, y=-54500, 47LP
123A, y=-55000, 47LP
3 µM 1OH + 6 µM 2
← 0-2.5 kD
1.07-1.17kD→
123A, y=-55000, 47LP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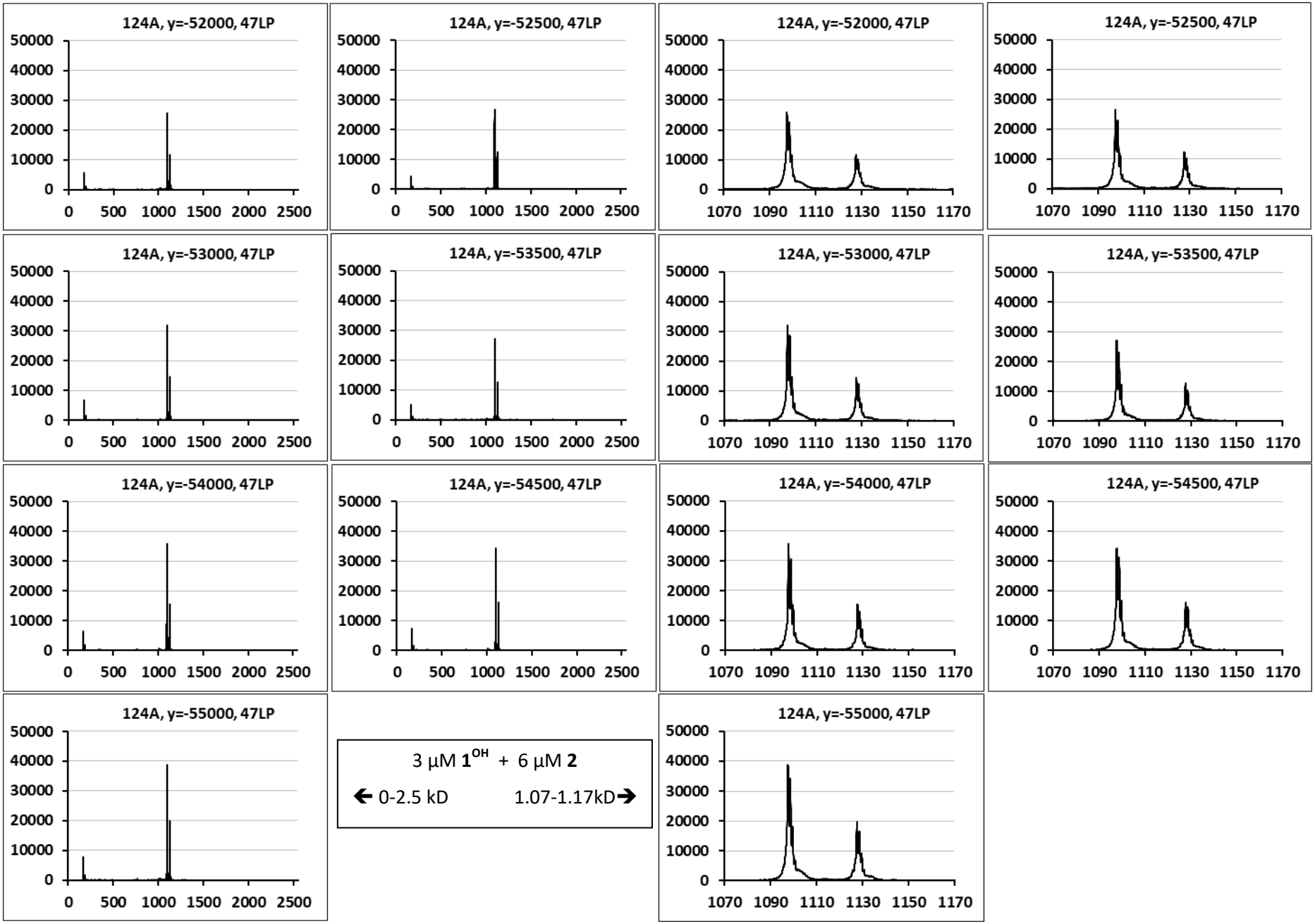
123B, y=-55500, 47LP
123B, y=-55600, 47LP
123B, y=-55500, 47LP
123B, y=-53000, 47LP
123B, y=-53500, 47LP
123B, y=-53000, 47LP
123B, y=-53500, 47LP
123B, y=-54000, 47LP
123B, y=-54500, 47LP
123B, y=-54000, 47LP
123B, y=-54500, 47LP
123B, y=-55000, 47LP
123B, y=-55000, 47LP
3 µM 1OH + 6 µM 2
← 0-2.5 kD
1.07-1.17kD→


124A, y=-52000, 47LP
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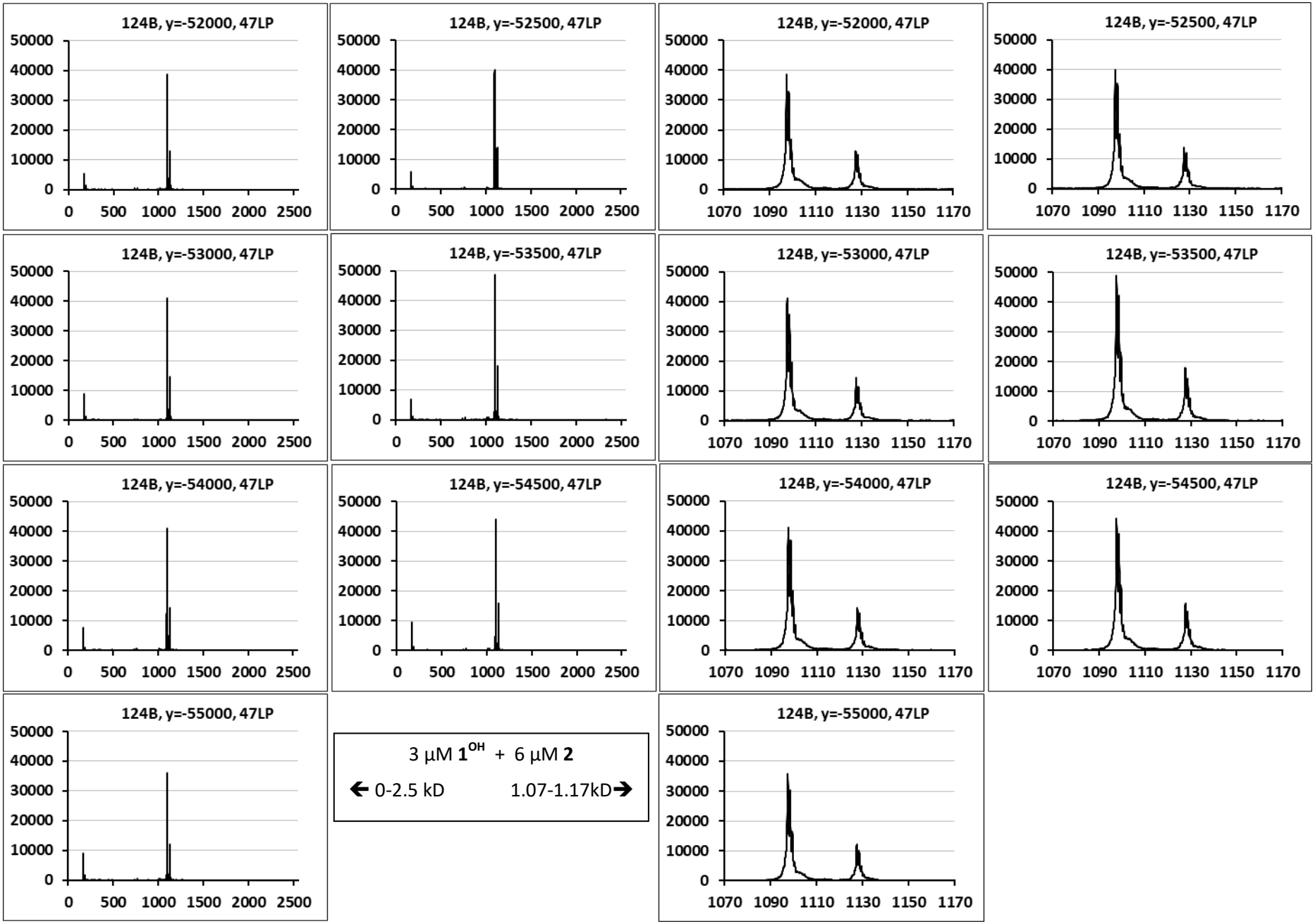
124A, y=-52000, 47LP
124A, y=-52500, 47LP
124A, y=-53000, 47LP
124A, y=-53500, 47LP
124A, y=-53000, 47LP
124A, y=-53500, 47LP
124A, y=-54000, 47LP
124A, y=-54500, 47LP
124A, y=-54000, 47LP
124A, y=-54500, 47LP
124A, y=-55000, 47LP
3 µM 1OH + 6 µM 2
← 0-2.5 kD
1.07-1.17kD→
124A, y=-55000, 47LP


124B, y=-52000, 47LP
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124B, y=-52000, 47LP
124B, y=-52500, 47LP
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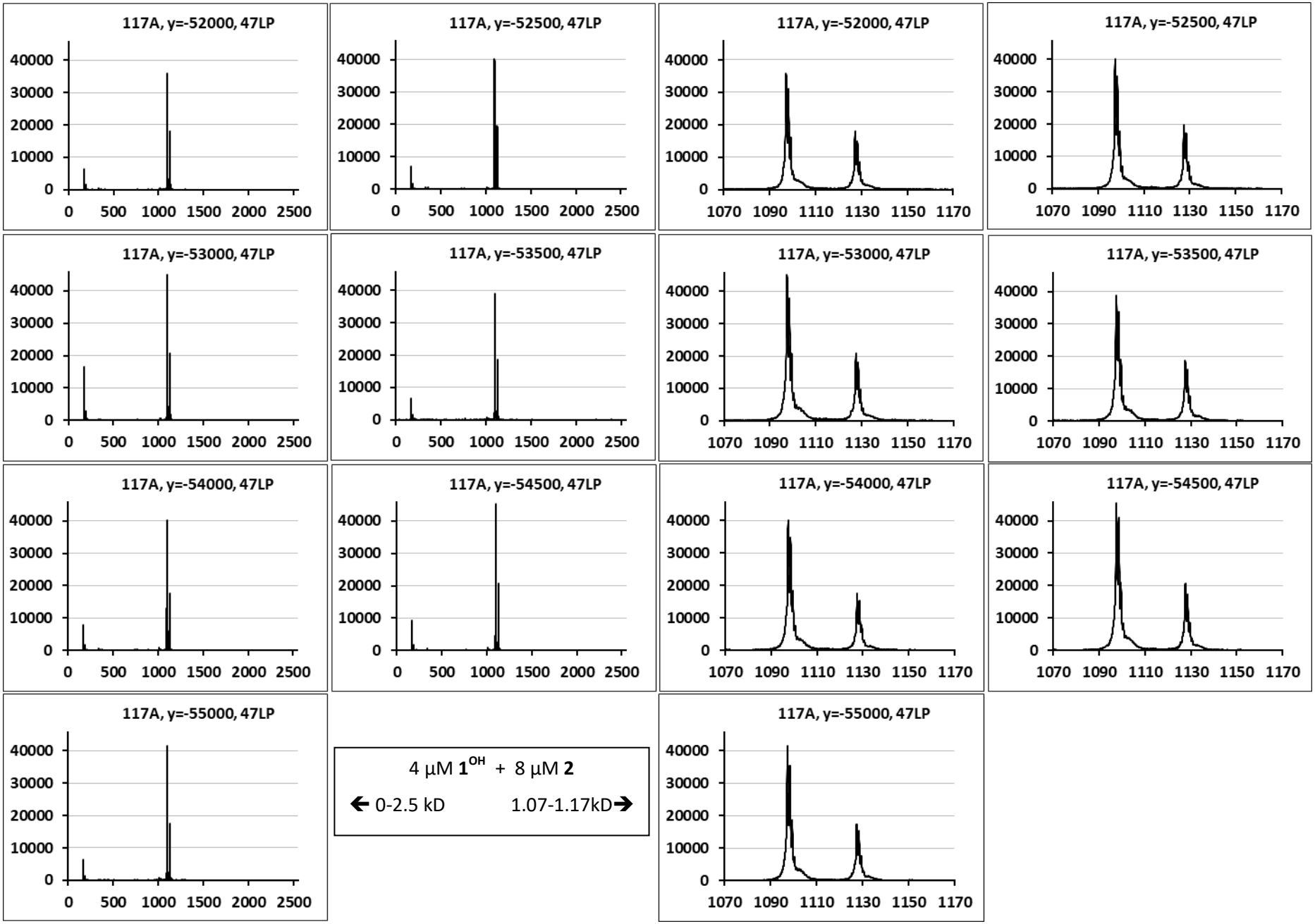
124B, y=-53000, 47LP
124B, y=-53500, 47LP
124B, y=-54000, 47LP
124B, y=-54500, 47LP
124B, y=-54000, 47LP
124B, y=-54500, 47LP
124B, y=-55000, 47LP
124B, y=-55000, 47LP
3 µM 1OH + 6 µM 2
← 0-2.5 kD
1.07-1.17kD→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117A, y=-52500, 47LP
117A, y=-52000, 47LP
117A, y=-52500, 47LP
117A, y=-53000, 47LP
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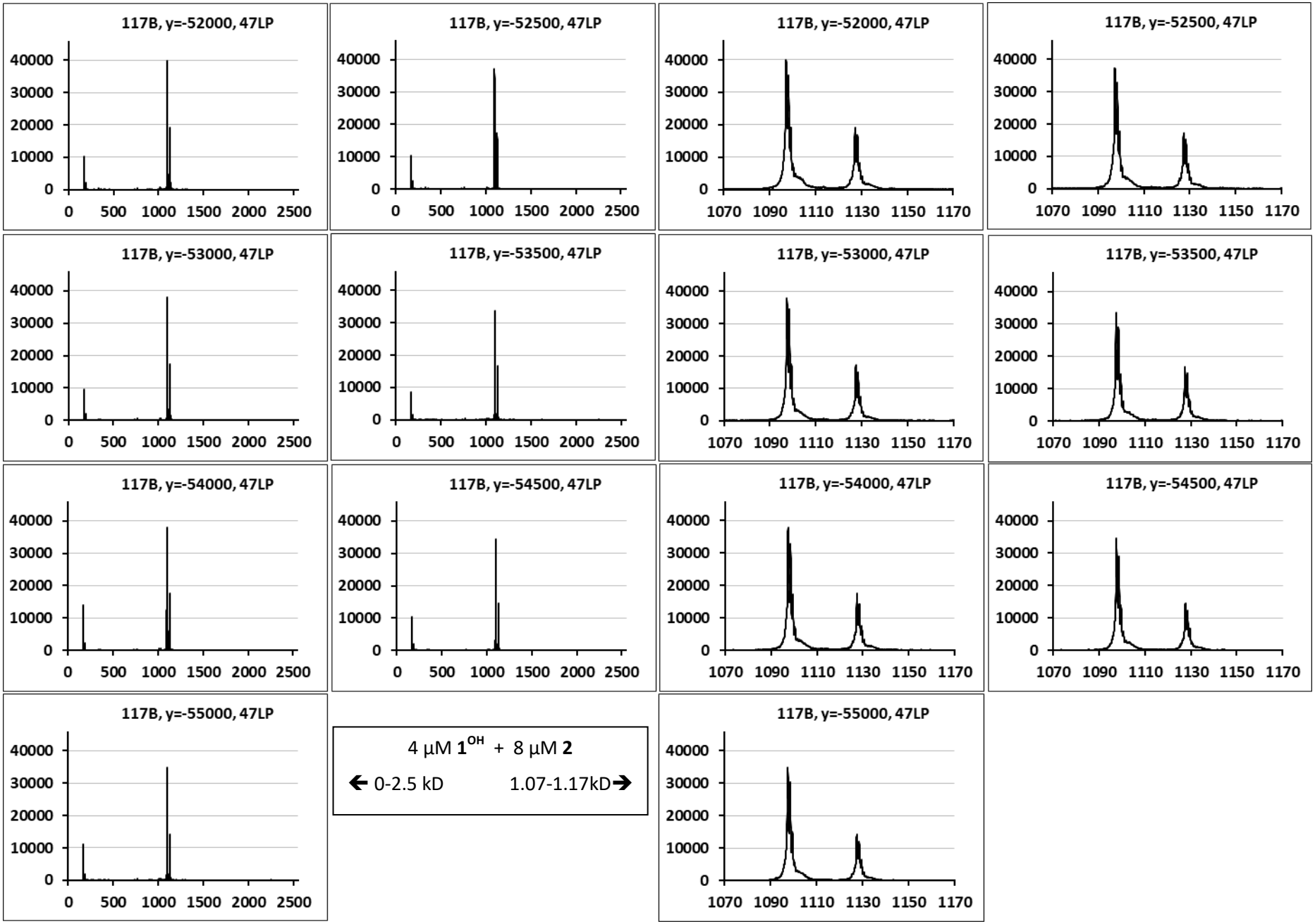
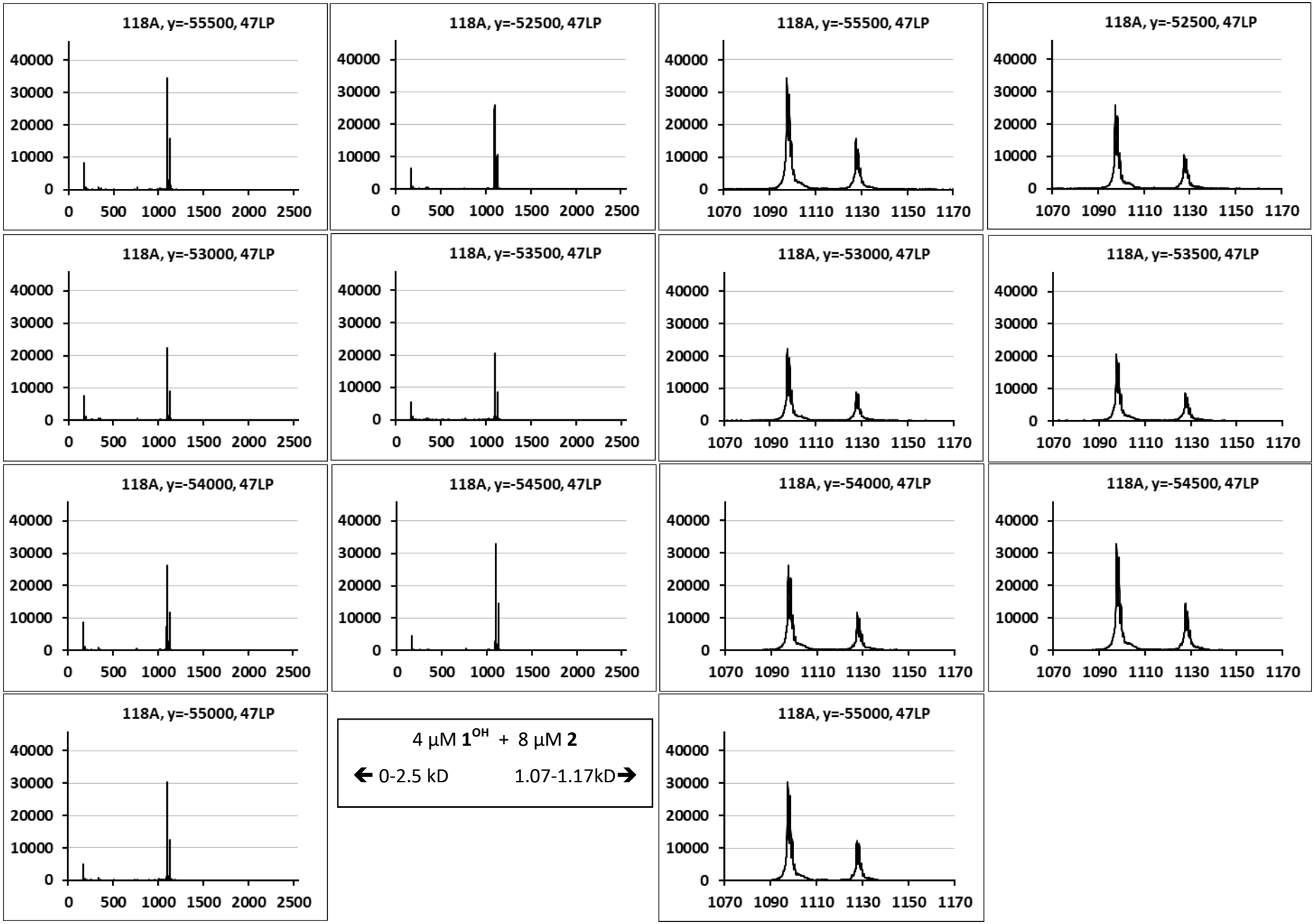
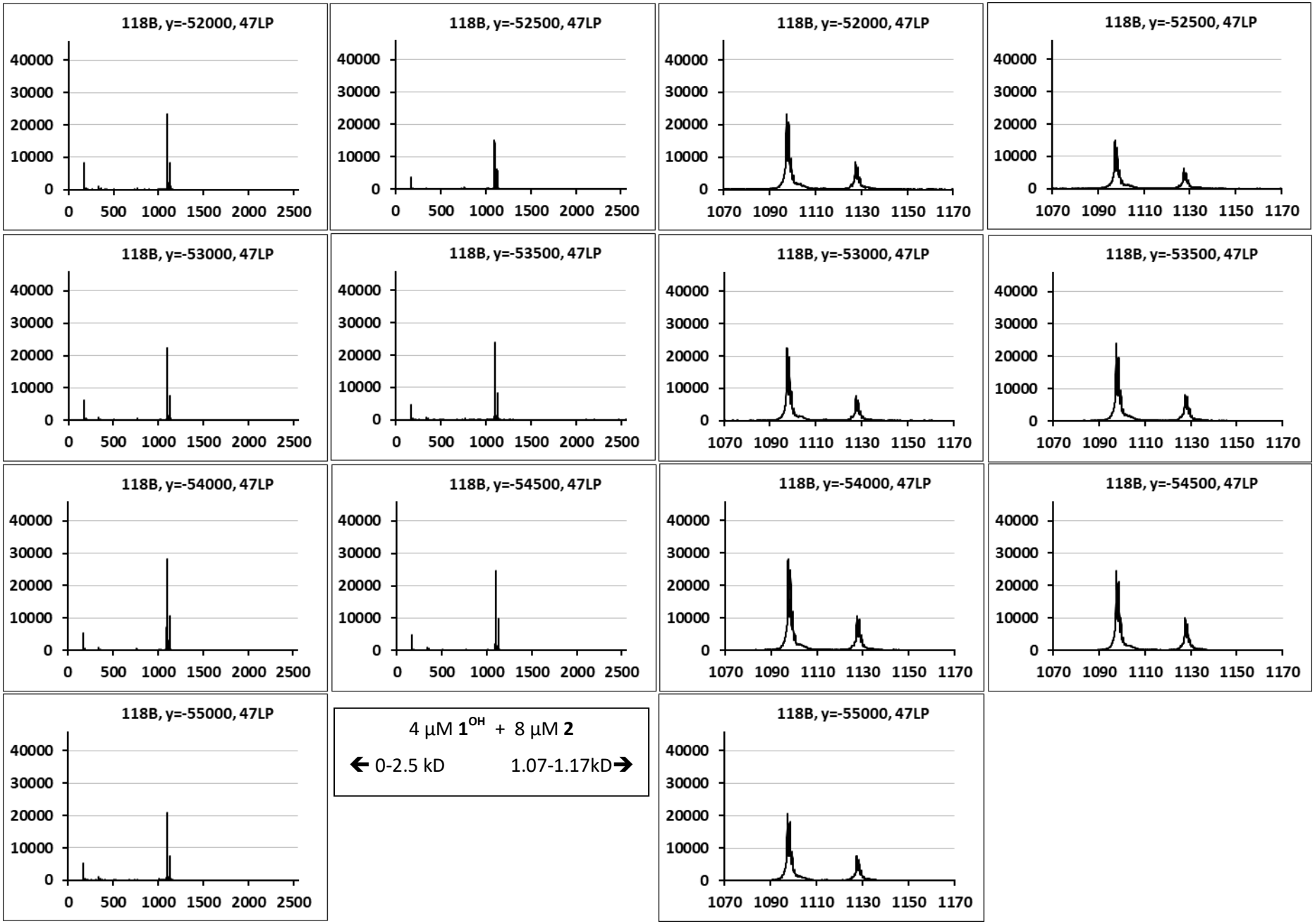
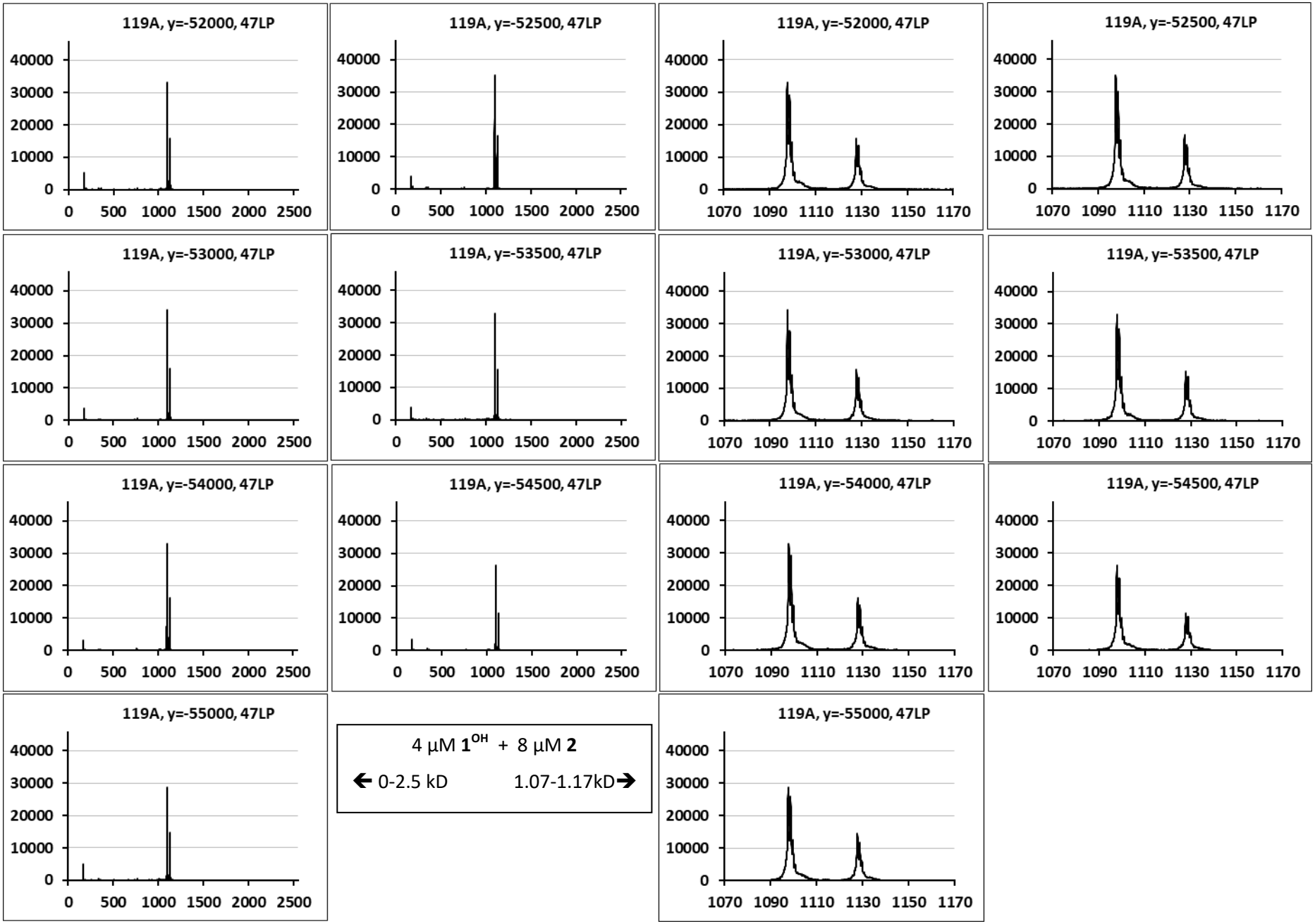
117A, y=-53500, 47LP
117A, y=-54000, 47LP
117A, y=-54500, 47LP
117A, y=-54000, 47LP
117A, y=-54500, 47LP
117A, y=-55000, 47LP
117A, y=-55000, 47LP
4 µM 1OH + 8 µM 2
← 0-2.5 kD
1.07-1.17kD→


117B, y=-52000, 47LP
117B, y=-52500, 47LP
117B, y=-52000, 47LP
117B, y=-52500, 47LP
117B, y=-53000, 47LP
117B, y=-53500, 47LP
117B, y=-53000, 47LP
117B, y=-53500, 47LP
117B, y=-54000, 47LP
117B, y=-54500, 47LP
117B, y=-54000, 47LP
117B, y=-54500, 47LP
117B, y=-55000, 47LP
117B, y=-55000, 47LP
40000
30000
20000
10000
0
0 500 1000 1500 2000 2500
1070 1090 1110 1130 1150 1170
4 µM 1OH + 8 µM 2
← 0-2.5 kD
1.07-1.17kD →


118A, y=-55500, 47LP
118A, y=-52500, 47LP
118A, y=-55500, 47LP
118A, y=-52500, 47LP
118A, y=-53000, 47LP
118A, y=-53500, 47LP
118A, y=-53000, 47LP
118A, y=-53500, 47LP
118A, y=-54000, 47LP
118A, y=-54500, 47LP
118A, y=-54000, 47LP
118A, y=-54500, 47LP
118A, y=-55000, 47LP
4 µM 1OH + 8 µM 2
← 0-2.5 kD
1.07-1.17kD→
118A, y=-55000, 47LP


118B, y=-52000, 47LP
118B, y=-52500, 47LP
118B, y=-52000, 47LP
118B, y=-52500, 47LP
118B, y=-53000, 47LP
118B, y=-53500, 47LP
118B, y=-53000, 47LP
118B, y=-53500, 47LP
118B, y=-54000, 47LP
118B, y=-54500, 47LP
118B, y=-54000, 47LP
118B, y=-54500, 47LP
118B, y=-55000, 47LP
4 µM 1OH + 8 µM 2
← 0-2.5 kD
1.07-1.17kD→
118B, y=-55000, 47LP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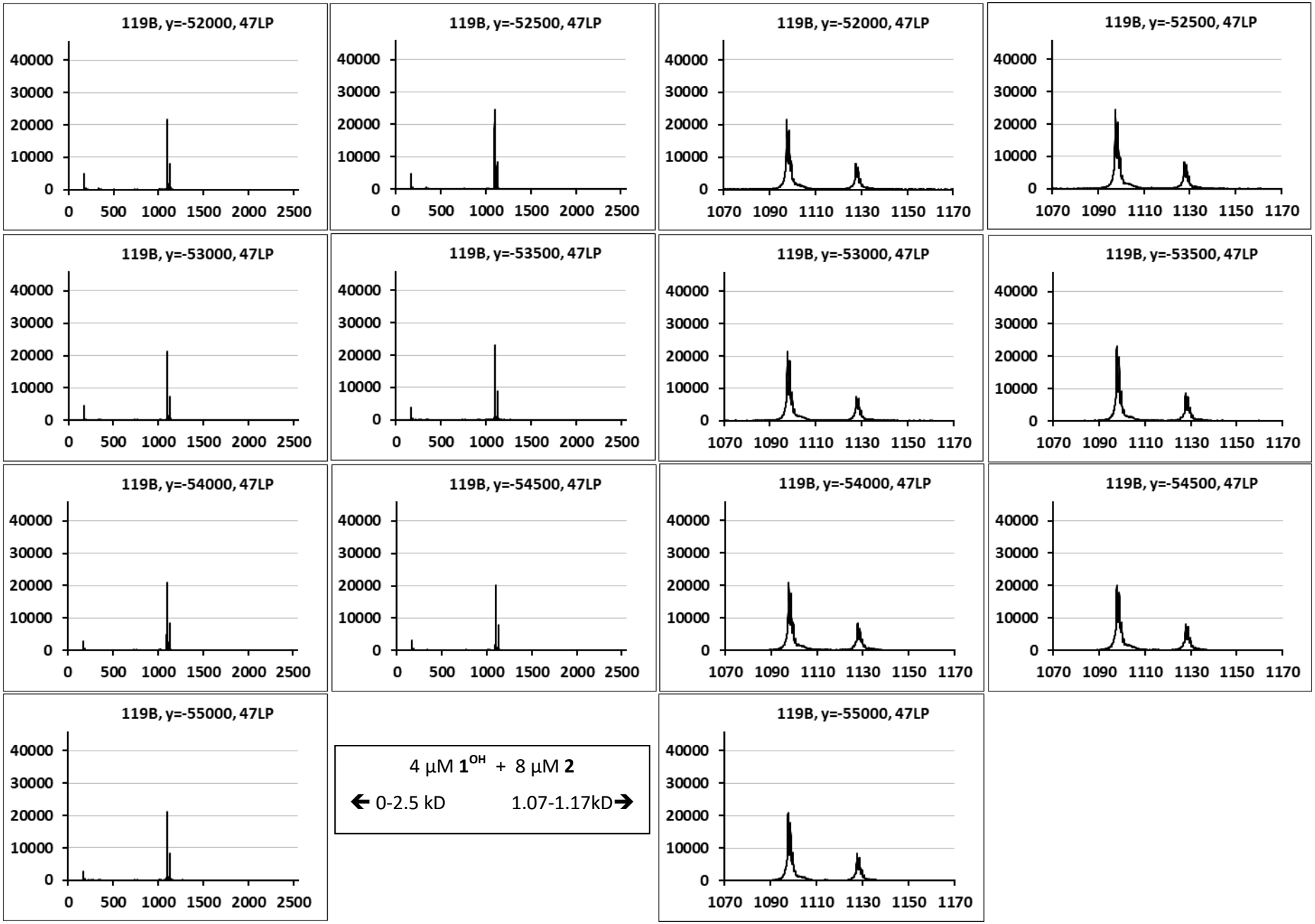
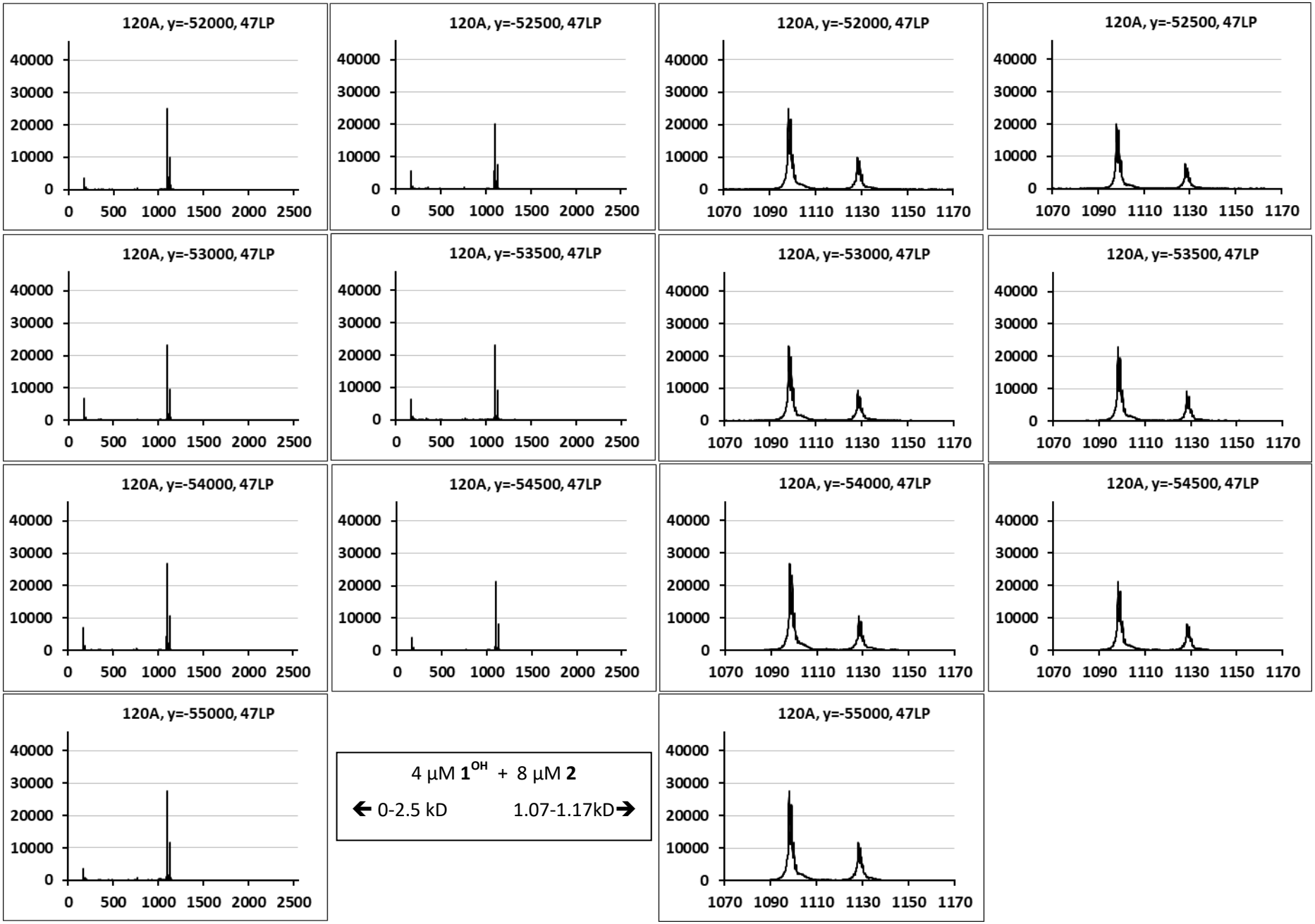
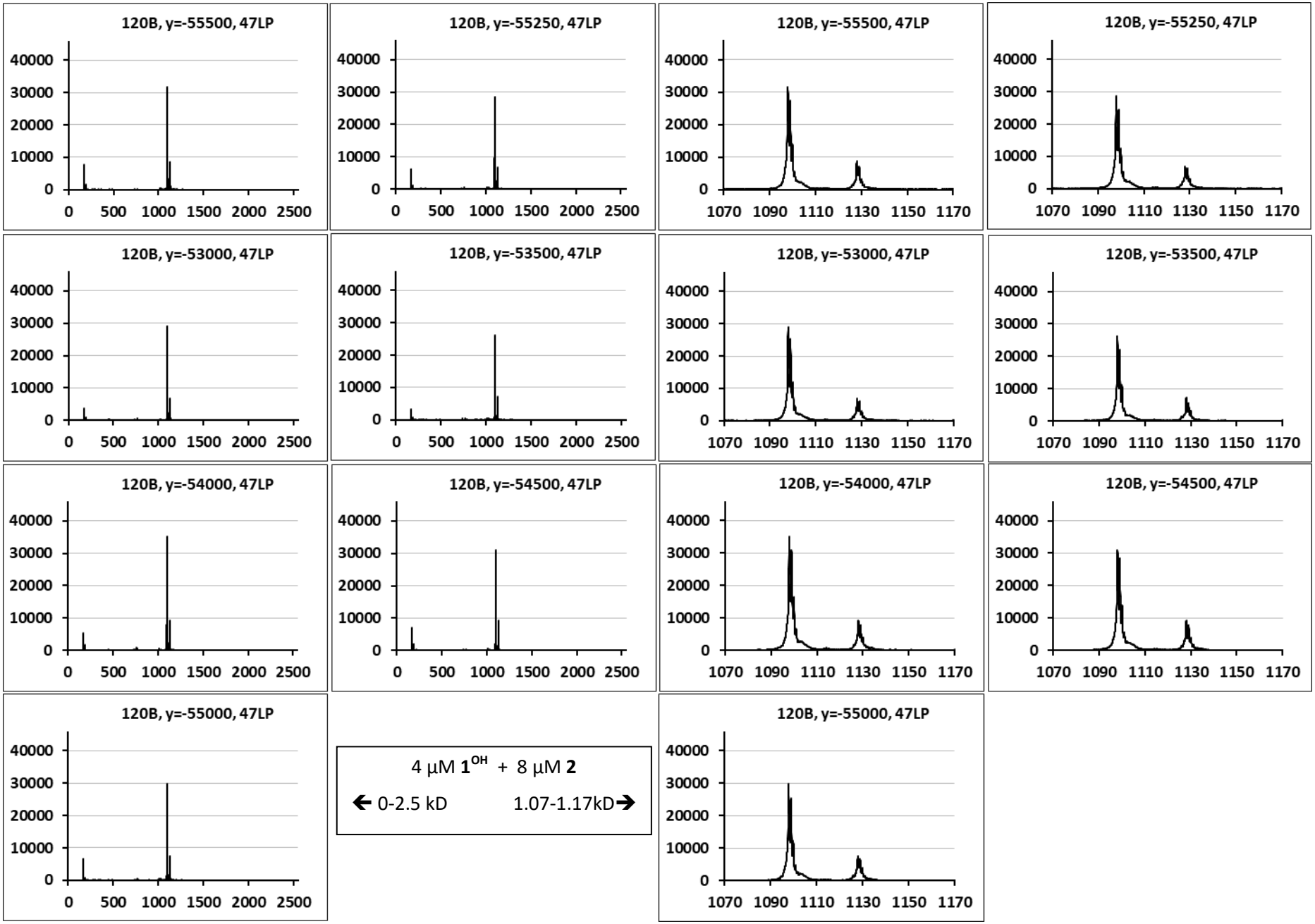
119A, y=-52500, 47LP
119A, y=-53000, 47LP
119A, y=-53500, 47LP
119A, y=-53000, 47LP
119A, y=-53500, 47LP
119A, y=-54000, 47LP
119A, y=-54500, 47LP
119A, y=-54000, 47LP
119A, y=-54500, 47LP
119A, y=-55000, 47LP
4 µM 1OH + 8 µM 2
← 0-2.5 kD
1.07-1.17kD→
119A, y=-55000, 47LP


119B, y=-52000, 47LP
119B, y=-52500, 47LP
119B, y=-52000, 47LP
119B, y=-52500, 47LP
119B, y=-53000, 47LP
119B, y=-53500, 47LP
119B, y=-53000, 47LP
119B, y=-53500, 47LP
119B, y=-54000, 47LP
119B, y=-54500, 47LP
119B, y=-54000, 47LP
119B, y=-54500, 47LP
119B, y=-55000, 47LP
119B, y=-55000, 47LP
40000
30000
20000
10000
0
0 500 1000 1500 2000 2500
1070 1090 1110 1130 1150 1170
4 µM 1OH + 8 µM 2
← 0-2.5 kD
1.07-1.17kD →

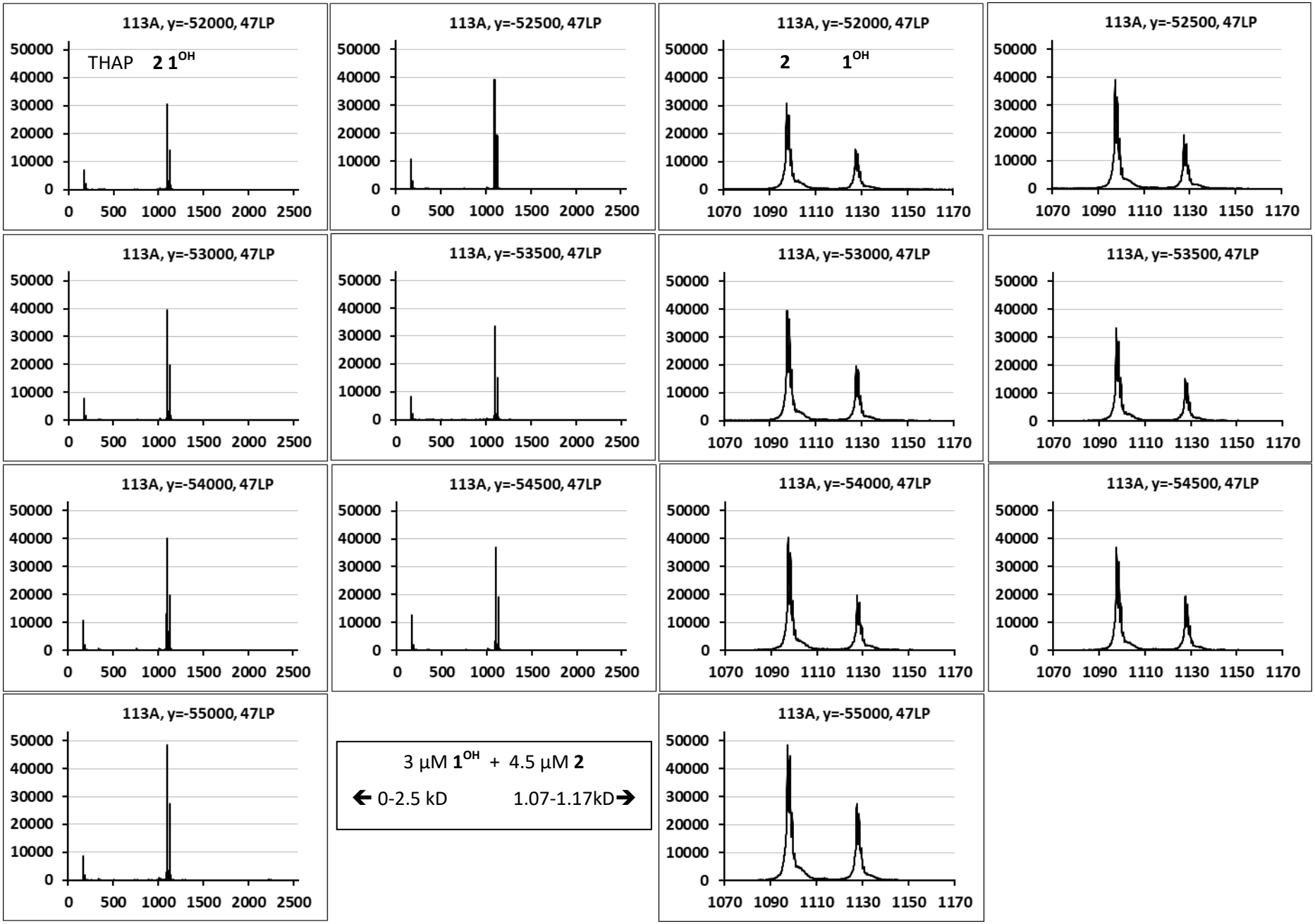

120A, y=-52000, 47LP
120A, y=-52500, 47LP
120A, y=-52000, 47LP
120A, y=-52500, 47LP
120A, y=-53000, 47LP
120A, y=-53500, 47LP
120A, y=-53000, 47LP
120A, y=-53500, 47LP
120A, y=-54000, 47LP
120A, y=-54500, 47LP
120A, y=-54000, 47LP
120A, y=-54500, 47LP
120A, y=-55000, 47LP
120A, y=-55000, 47LP
4 µM 1OH + 8 µM 2
← 0-2.5 kD
1.07-1.17kD→

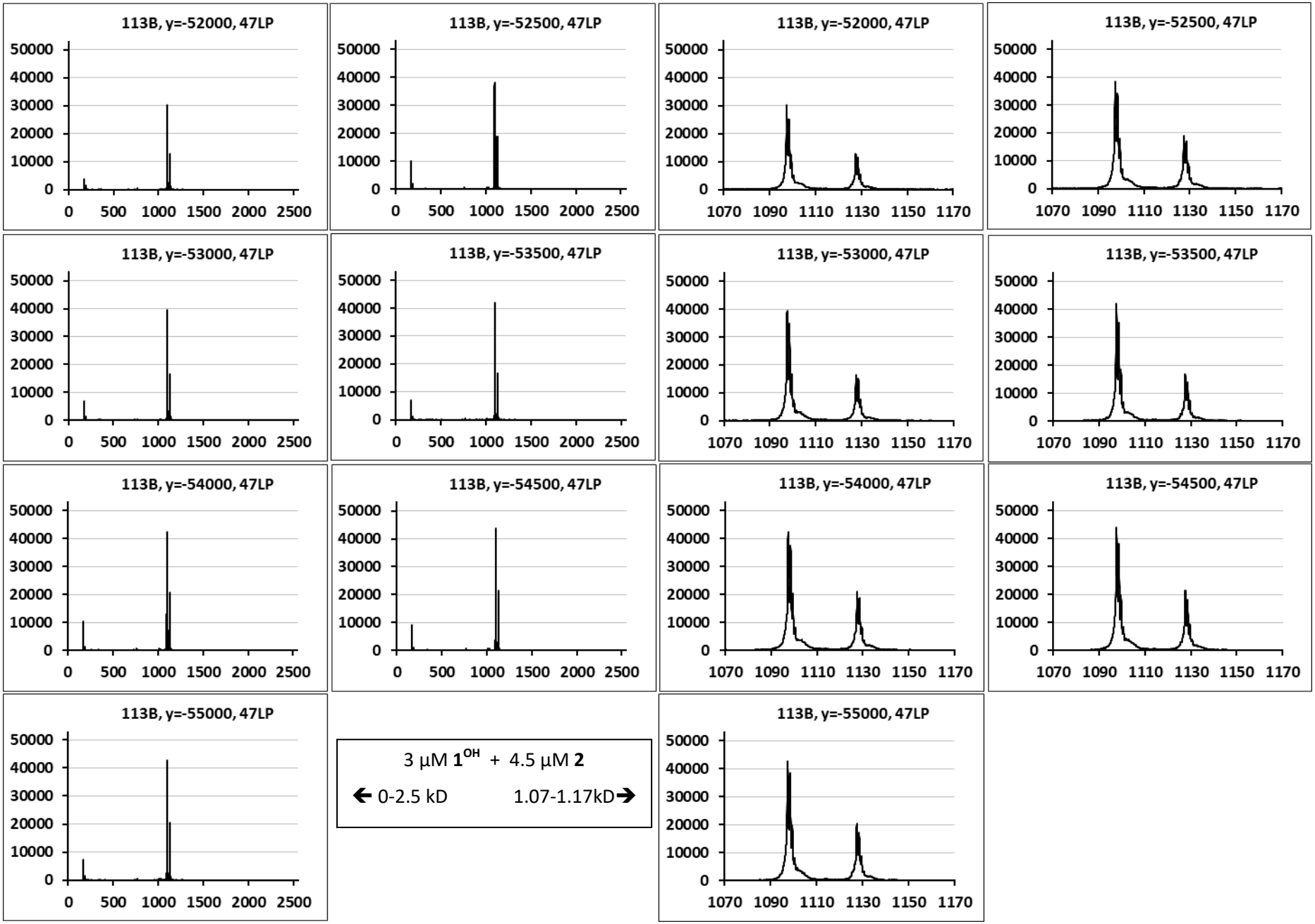

120B, y=-55500, 47LP
120B, y=-55250, 47LP
120B, y=-55500, 47LP
120B, y=-55250, 47LP
120B, y=-53000, 47LP
120B, y=-53500, 47LP
120B, y=-53000, 47LP
120B, y=-53500, 47LP
120B, y=-54000, 47LP
120B, y=-54500, 47LP
120B, y=-54000, 47LP
120B, y=-54500, 47LP
120B, y=-55000, 47LP
120B, y=-55000, 47LP
4 µM 1OH + 8 µM 2
← 0-2.5 kD
1.07-1.17kD→

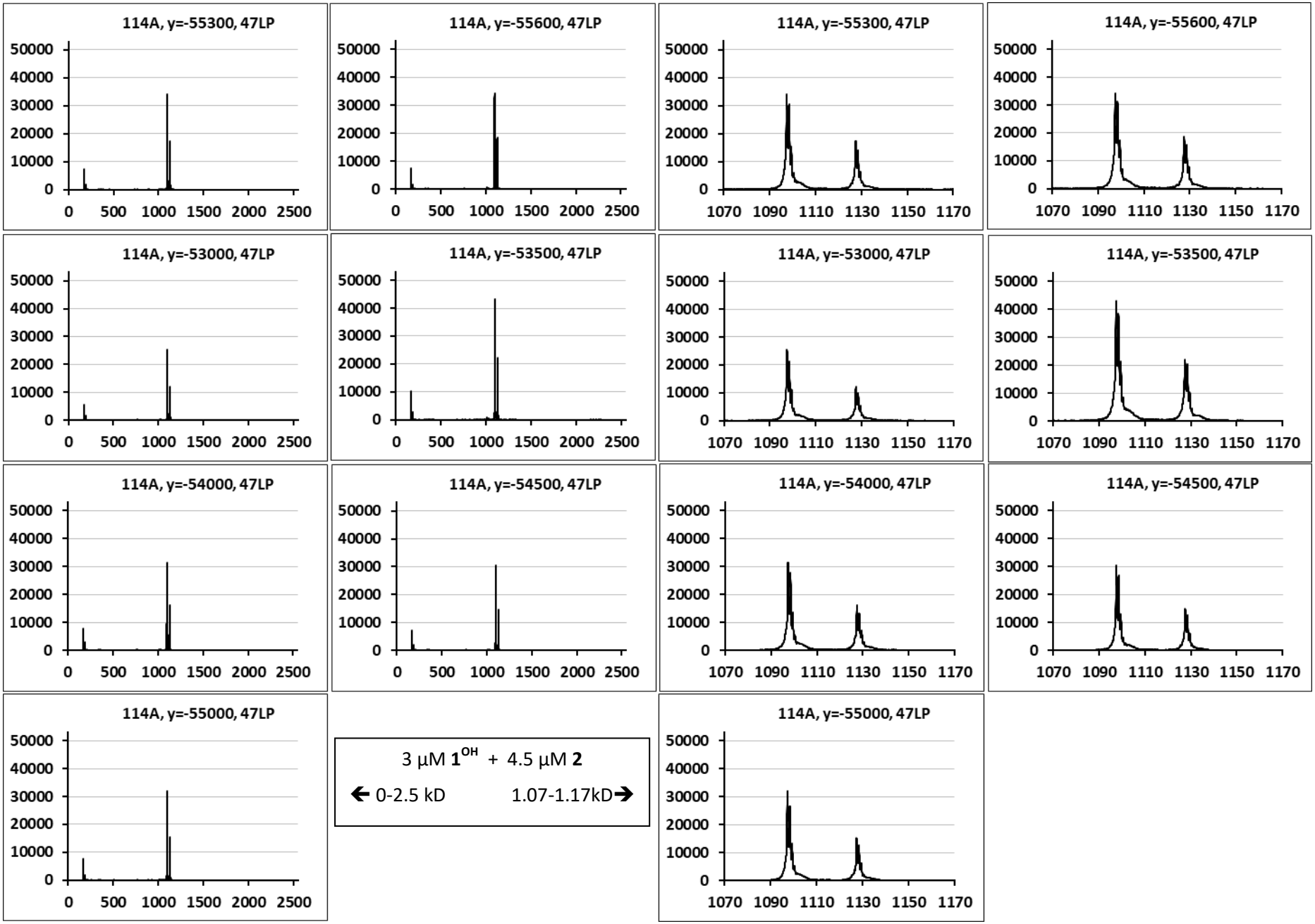
Summary of **1^{OH} / 2** phenyl octane solution with 1 : 1.5 concentration ratio: TOF-MS ML **1^{OH}** / **2** intensity and intensity ratio statistics from **1^{OH} / 2** phenyl octane solution with 1 : 1.5 concentration ratio (THAP thin film applied, LP = 47, TOF-MS measured at 14 locations on each of 4 independently prepared samples).

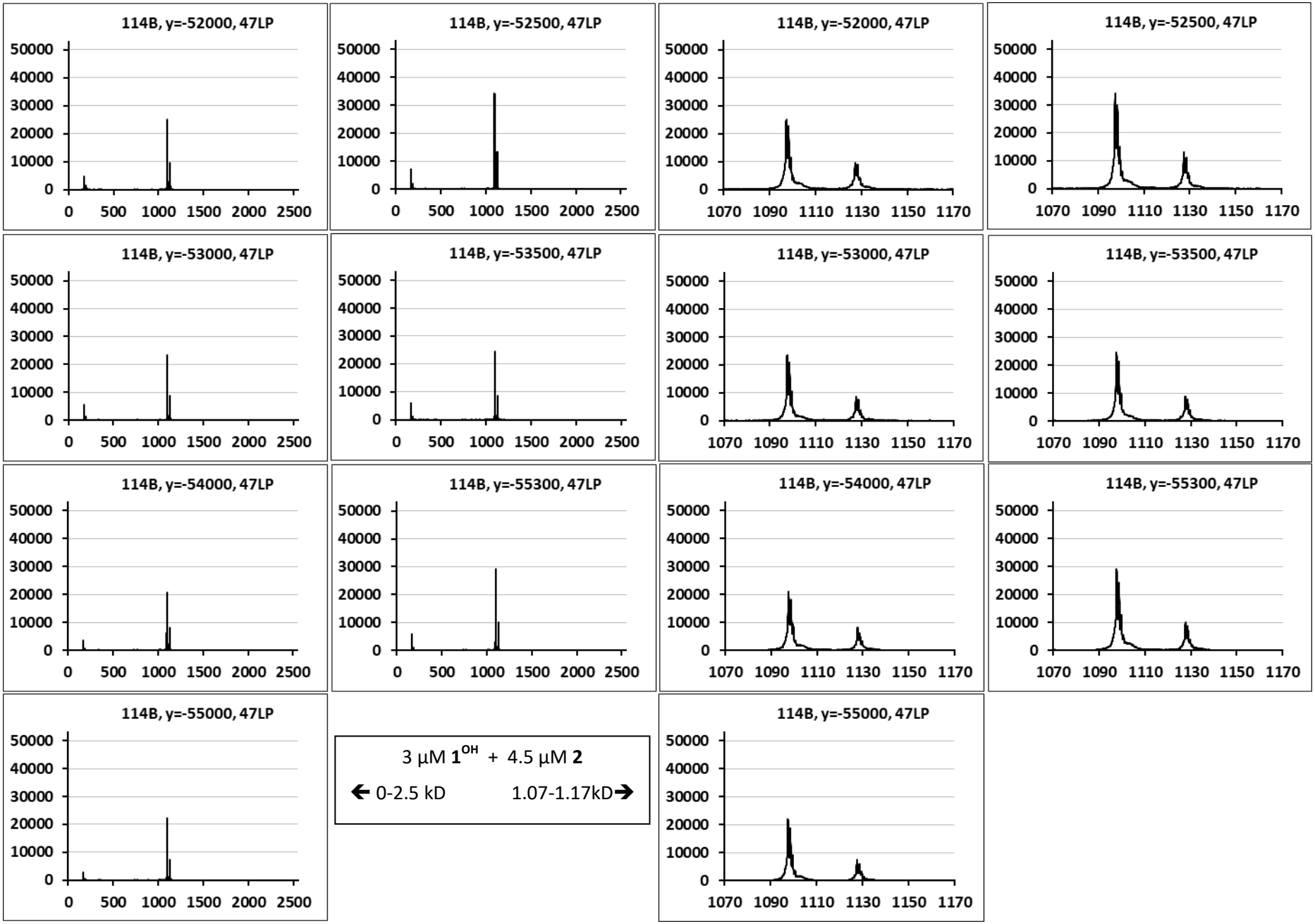
[1^{OH}]	[2]	# Locations	1^{OH} / 2	1^{OH} / 2	1^{OH} / 2	1^{OH} / 2	1^{OH} / 2	1^{OH}	1^{OH}	1^{OH}	1^{OH}	1^{OH}	2	2	2	2	2
µM	µM	(file names)	Max	Min	Avg	σ	σ/Avg	Max	Min	Avg	σ	σ/Avg	Max	Min	Avg	σ	σ/Avg
3	4.5	14 (113A,B)	0.57	0.40	0.48	0.04	9%	27414	12686	18761	3624	19%	48486	30108	39081	5065	13%
3	4.5	14 (114A,B)	0.54	0.34	0.44	0.07	17%	22135	7493	13036	4500	35%	43131	20854	29259	6128	21%
3	4.5	14 (115A,B)	0.52	0.33	0.43	0.07	16%	17051	8626	11881	2232	19%	34728	20074	27601	4180	15%
3	4.5	14 (116A,B)	0.42	0.29	0.37	0.04	11%	18484	5290	9769	4156	43%	43711	16718	26139	9391	36%
3	4.5	All	0.57	0.29	**0.43**	**0.07**	**16%**	27414	5290	**13362**	**4944**	**37%**	48486	16718	**30520**	**8126**	**27%**

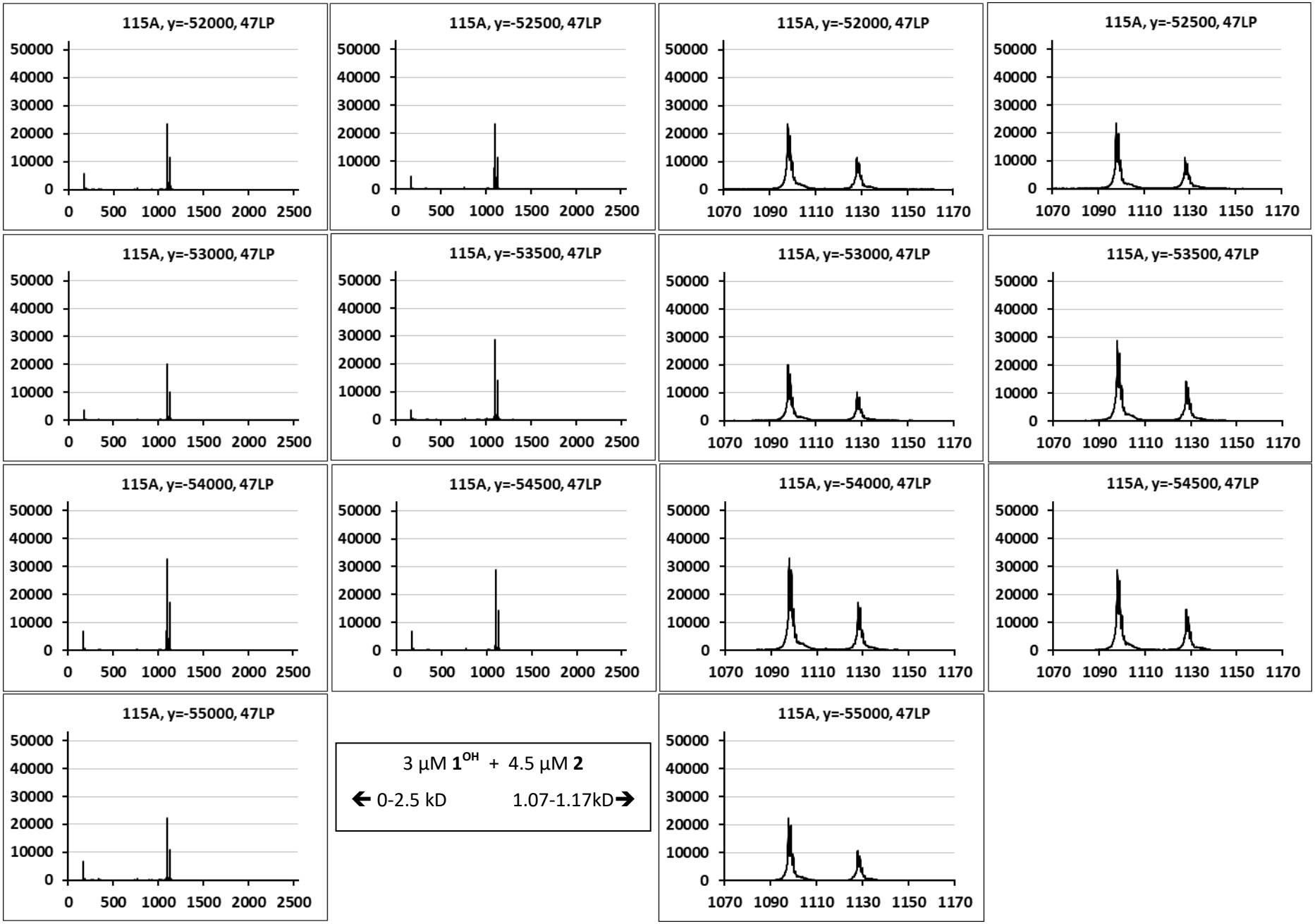
[1^{OH}]	[2]	# Locations	1^{OH} / 2	1^{OH} / 2	1^{OH} / 2	1^{OH} / 2	1^{OH} / 2	1^{OH}	1^{OH}	1^{OH}	1^{OH}	1^{OH}	2	2	2	2	2
µM	µM	(file names)	Max	Min	Avg	σ	σ/Avg	Max	Min	Avg	σ	σ/Avg	Max	Min	Avg	σ	σ/Avg
4	6	14 (109A,B)	0.51	0.34	0.43	0.05	11%	21939	8936	14193	3696	26%	42938	20536	33063	6686	20%
4	6	14 (119A,B)	0.52	0.35	0.43	0.06	13%	26487	9936	16135	4878	30%	53546	20353	37081	9035	24%
4	6	14 (111A,B)	0.53	0.34	0.43	0.06	14%	19308	10844	13690	2639	19%	38456	23294	31919	4650	15%
4	6	14 (112A,B)	0.47	0.31	0.38	0.06	14%	16765	3083	11091	3289	30%	38346	6604	29348	7780	27%
4	6	All	0.53	0.31	**0.42**	**0.06**	**14%**	26487	3083	**13777**	**4044**	**29%**	53546	6604	**32853**	**7562**	**23%**

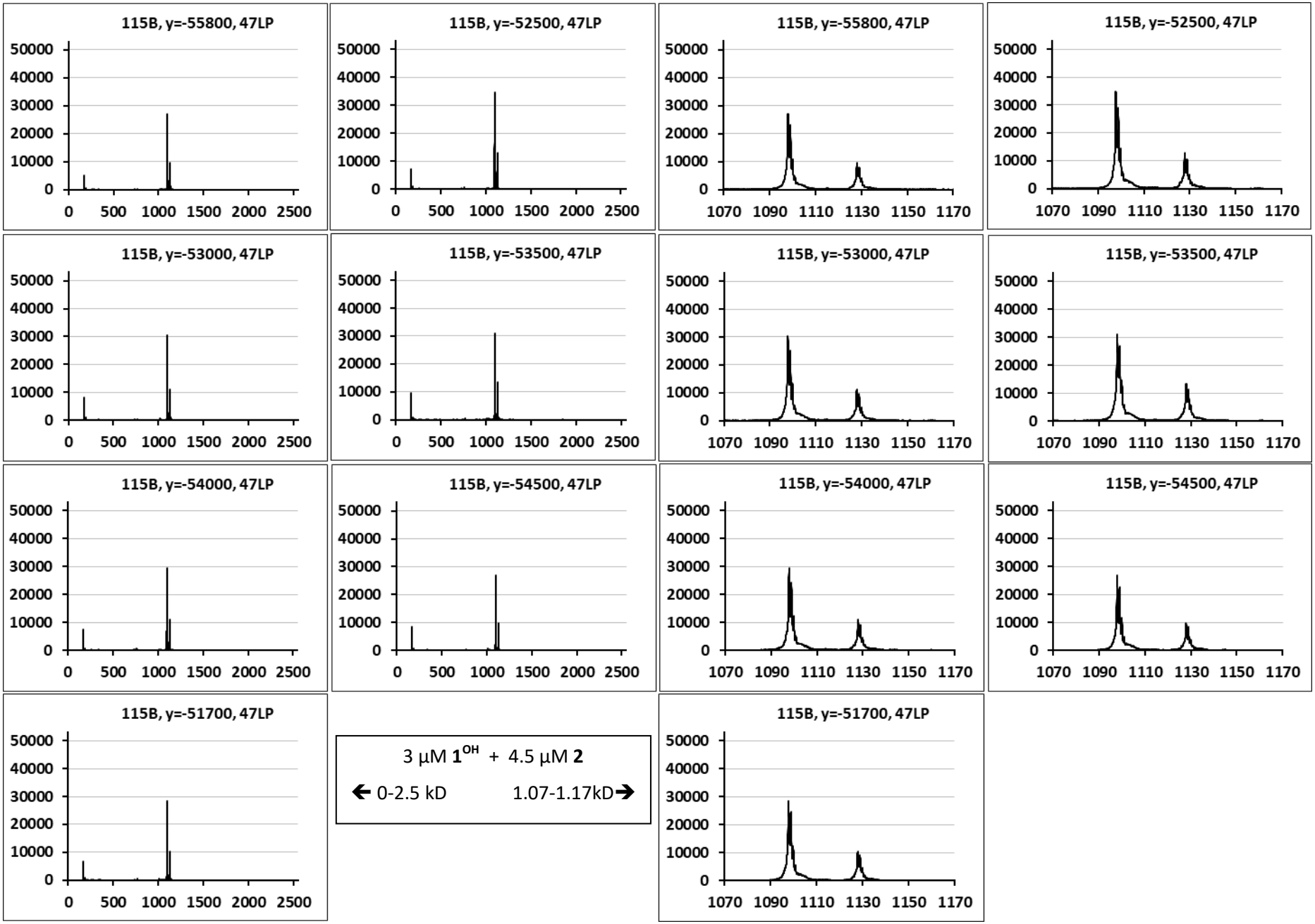

113A, y=-52000, 47LP
THAP 2 1OH
113A, y=-52500, 47LP
113A, y=-52000, 47LP
2 1OH
113A, y=-52500, 47LP
113A, y=-53000, 47LP
113A, y=-53500, 47LP
113A, y=-53000, 47LP
113A, y=-53500, 47LP
113A, y=-54000, 47LP
113A, y=-54500, 47LP
113A, y=-54000, 47LP
113A, y=-54500, 47LP
113A, y=-55000, 47LP
3 µM 1OH + 4.5 µM 2
← 0-2.5 kD
1.07-1.17kD →
113A, y=-55000, 47LP

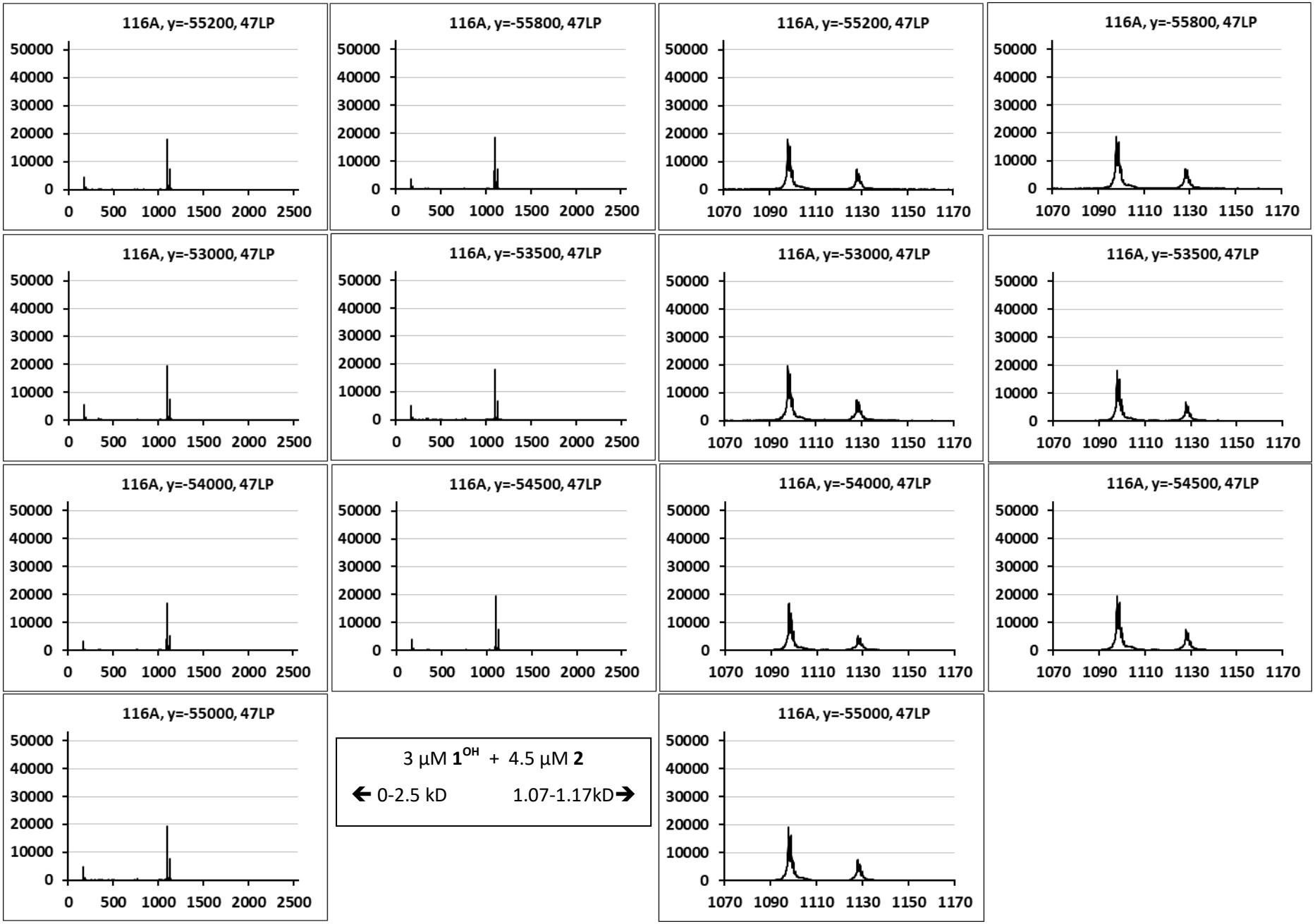

113B, y=-52000, 47LP
113B, y=-52500, 47LP
113B, y=-52000, 47LP
113B, y=-52500, 47LP
113B, y=-53000, 47LP
113B, y=-53500, 47LP
113B, y=-53000, 47LP
113B, y=-53500, 47LP
113B, y=-54000, 47LP
113B, y=-54500, 47LP
113B, y=-54000, 47LP
113B, y=-54500, 47LP
113B, y=-55000, 47LP
3 µM 1OH + 4.5 µM 2
← 0-2.5 kD
1.07-1.17kD→
113B, y=-55000, 47LP


114A, y=-55300, 47LP
114A, y=-55600, 47LP
114A, y=-55300, 47LP
114A, y=-55600, 47LP
114A, y=-53000, 47LP
114A, y=-53500, 47LP
114A, y=-53000, 47LP
114A, y=-53500, 47LP
114A, y=-54000, 47LP
114A, y=-54500, 47LP
114A, y=-54000, 47LP
114A, y=-54500, 47LP
114A, y=-55000, 47LP
114A, y=-55000, 47LP
50000
40000
30000
20000
10000
0
0 500 1000 1500 2000 2500
1070 1090 1110 1130 1150 1170
3 µM 1OH + 4.5 µM 2
← 0-2.5 kD
1.07-1.17kD→


114B, y=-52000, 47LP
114B, y=-52500, 47LP
114B, y=-52000, 47LP
114B, y=-52500, 47LP
114B, y=-53000, 47LP
114B, y=-53500, 47LP
114B, y=-53000, 47LP
114B, y=-53500, 47LP
114B, y=-54000, 47LP
114B, y=-55300, 47LP
114B, y=-54000, 47LP
114B, y=-55300, 47LP
114B, y=-55000, 47LP
114B, y=-55000, 47LP
50000
40000
30000
20000
10000
0
0 500 1000 1500 2000 2500
1070 1090 1110 1130 1150 1170
3 µM 1OH + 4.5 µM 2
← 0-2.5 kD
1.07-1.17kD →


115A, y=-52000, 47LP
115A, y=-52500, 47LP
115A, y=-52000, 47LP
115A, y=-52500, 47LP
115A, y=-53000, 47LP
115A, y=-53500, 47LP
115A, y=-53000, 47LP
115A, y=-53500, 47LP
115A, y=-54000, 47LP
115A, y=-54500, 47LP
115A, y=-54000, 47LP
115A, y=-54500, 47LP
115A, y=-55000, 47LP
115A, y=-55000, 47LP
3 µM 1OH + 4.5 µM 2
← 0-2.5 kD
1.07-1.17kD→


115B, y=-55800, 47LP
115B, y=-52500, 47LP
115B, y=-55800, 47LP
115B, y=-52500, 47LP
115B, y=-53000, 47LP
115B, y=-53500, 47LP
115B, y=-53000, 47LP
115B, y=-53500, 47LP
115B, y=-54000, 47LP
115B, y=-54500, 47LP
115B, y=-54000, 47LP
115B, y=-54500, 47LP
115B, y=-51700, 47LP
115B, y=-51700, 47LP
3 µM 1OH + 4.5 µM 2
← 0-2.5 kD
1.07-1.17kD→


116A, y=-55200, 47LP
116A, y=-55800, 47LP
116A, y=-55200, 47LP
116A, y=-55800, 47LP
116A, y=-53000, 47LP
116A, y=-53500, 47LP
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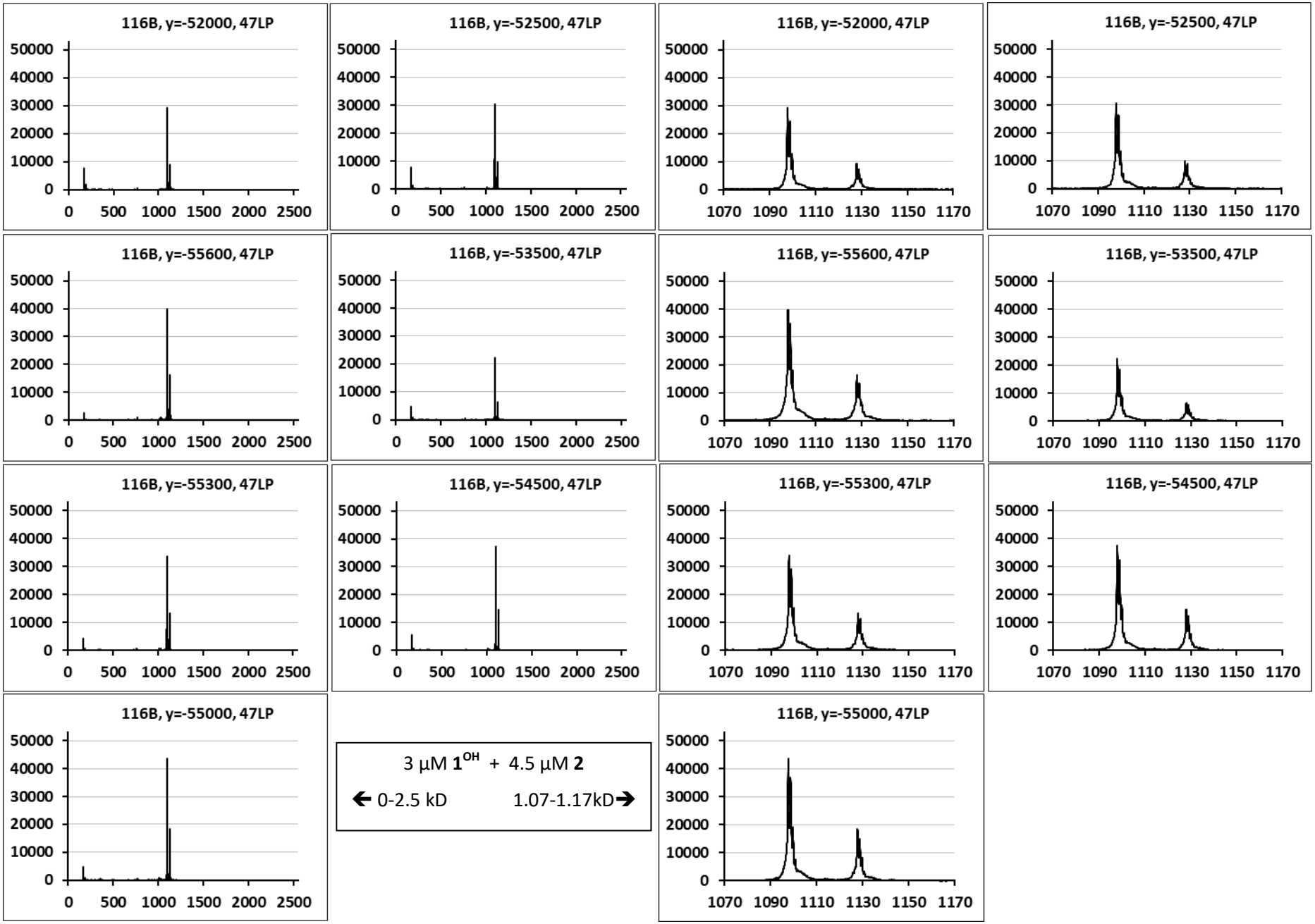
116A, y=-54000, 47LP
116A, y=-54500, 47LP
116A, y=-54000, 47LP
116A, y=-54500, 47LP
116A, y=-55000, 47LP
116A, y=-55000, 47LP
3 µM 1OH + 4.5 µM 2
← 0-2.5 kD
1.07-1.17kD→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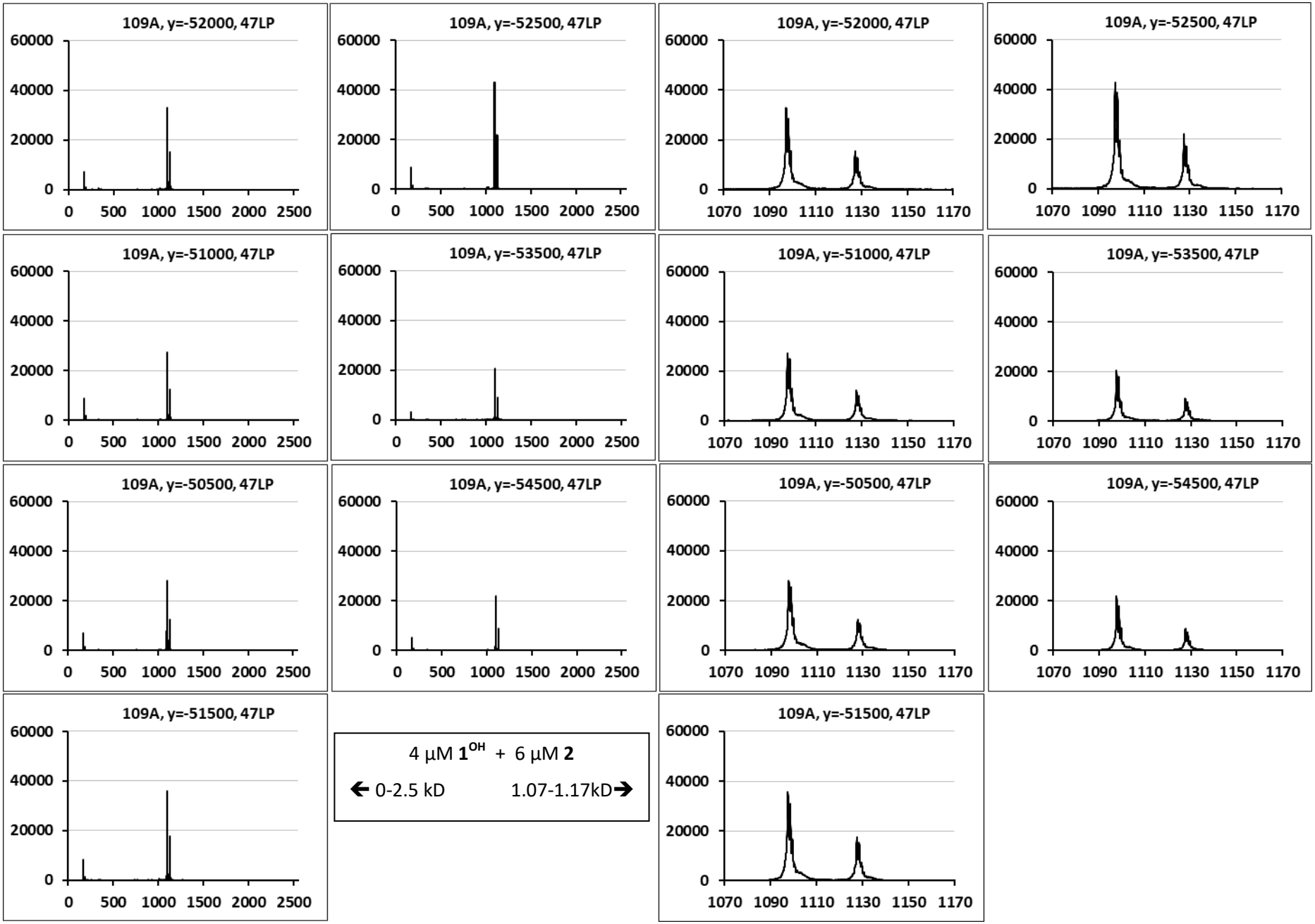
116B, y=-52500, 47LP
116B, y=-52000, 47LP
116B, y=-52500, 47LP
116B, y=-55600, 47LP
116B, y=-53500, 47LP
116B, y=-55600, 47LP
116B, y=-53500, 47LP
116B, y=-55300, 47LP
116B, y=-54500, 47LP
116B, y=-55300, 47LP
116B, y=-54500, 47LP
116B, y=-55000, 47LP
116B, y=-55000, 47LP
3 µM 1OH + 4.5 µM 2
← 0-2.5 kD
1.07-1.17kD→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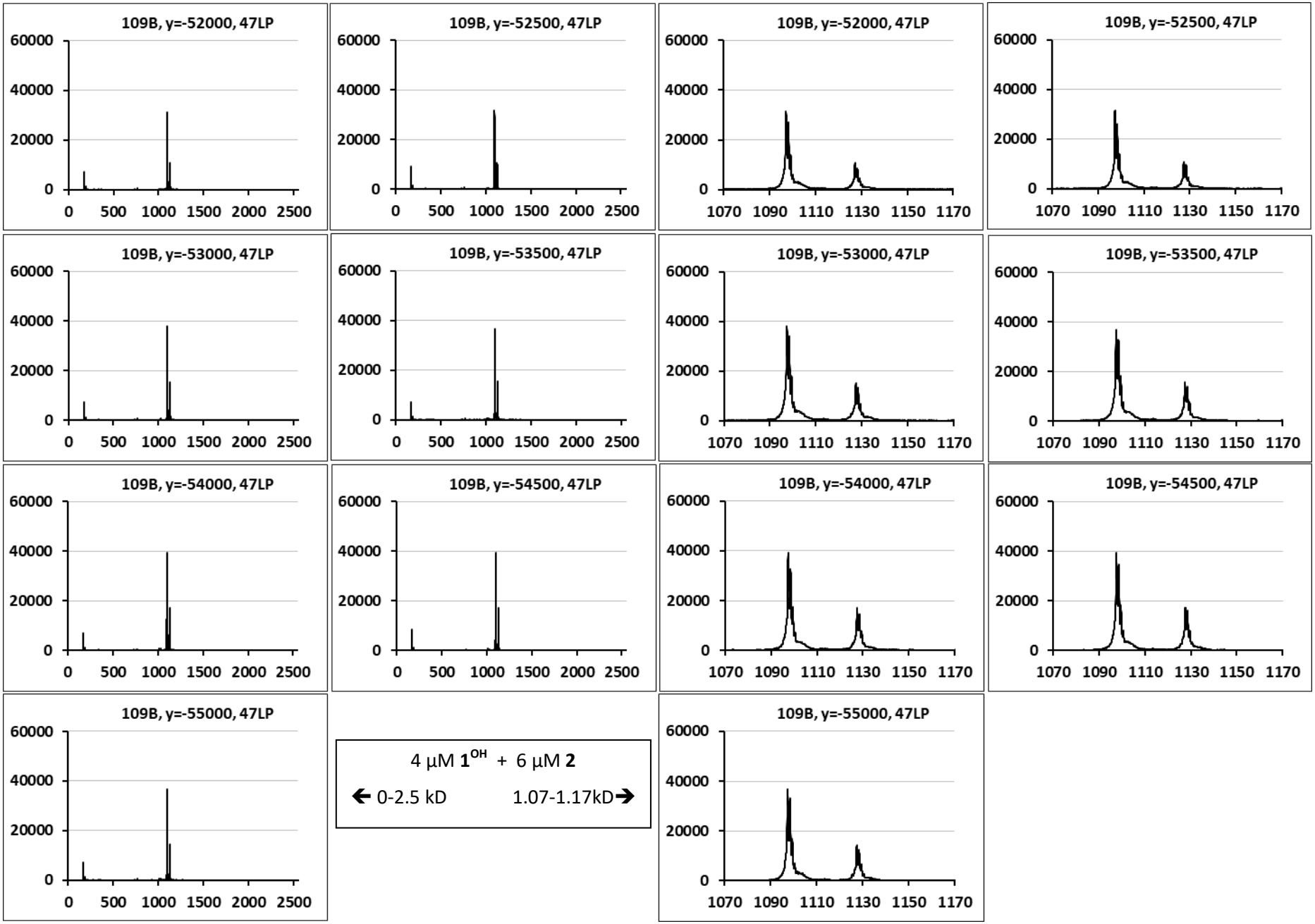
109A, y=-52500, 47LP
109A, y=-52000, 47LP
109A, y=-52500, 47LP
109A, y=-51000, 47LP
109A, y=-53500, 47LP
109A, y=-51000, 47LP
109A, y=-53500, 47LP
109A, y=-50500, 47LP
109A, y=-54500, 47LP
109A, y=-50500, 47LP
109A, y=-54500, 47LP
109A, y=-51500, 47LP
109A, y=-51500, 47LP
60000
40000
20000
0
0 500 1000 1500 2000 2500
1070 1090 1110 1130 1150 1170
4 µM 1OH + 6 µM 2
← 0-2.5 kD
1.07-1.17kD →


109B, y=-52000, 47LP
109B, y=-52500, 47LP
109B, y=-52000, 47LP
109B, y=-52500, 47LP
109B, y=-53000, 47LP
109B, y=-53500, 47LP
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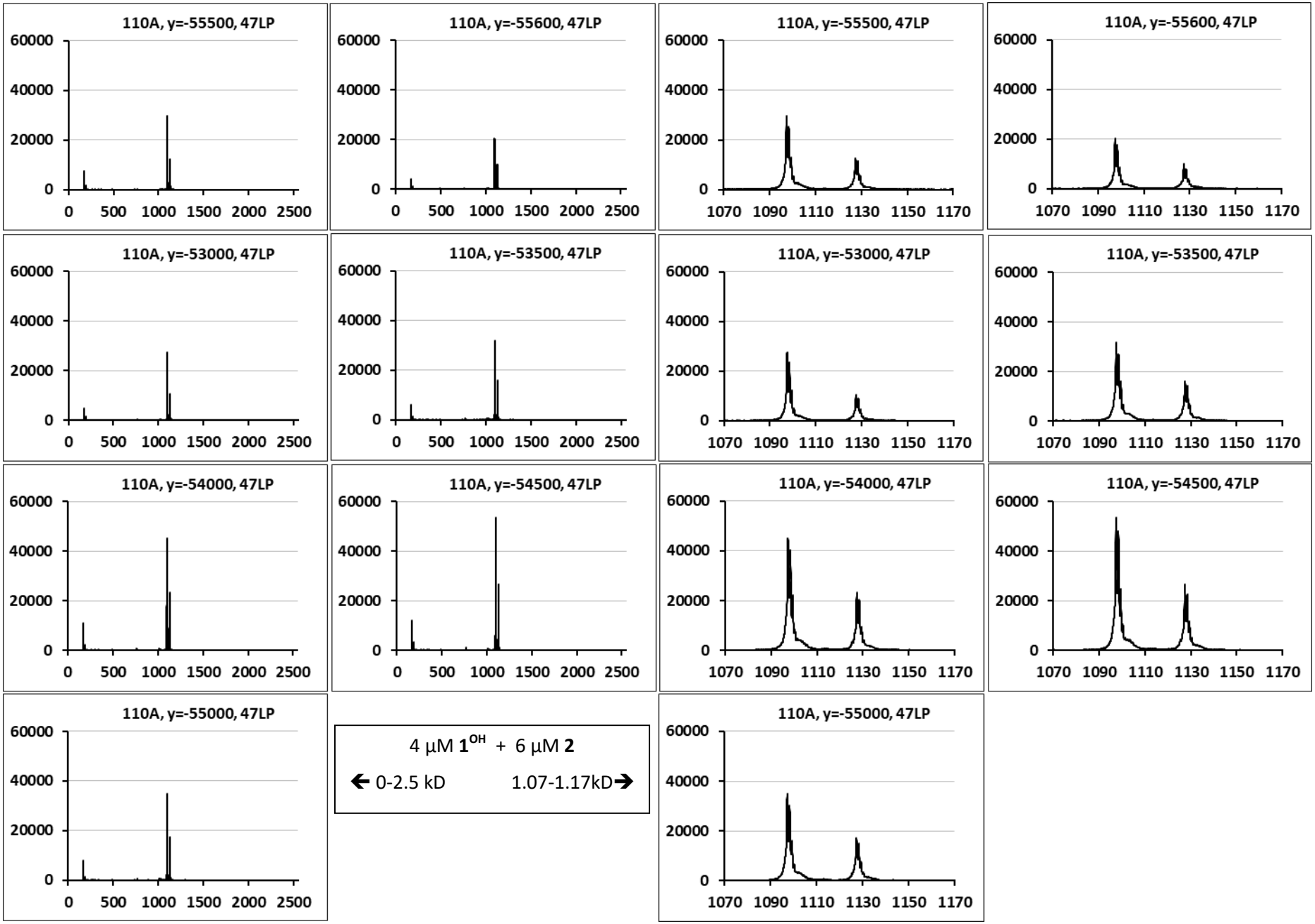
109B, y=-53500, 47LP
109B, y=-54000, 47LP
109B, y=-54500, 47LP
109B, y=-54000, 47LP
109B, y=-54500, 47LP
109B, y=-55000, 47LP
4 µM 1OH + 6 µM 2
← 0-2.5 kD
1.07-1.17kD →
109B, y=-55000, 47LP


110A, y=-55500, 47LP
110A, y=-55600, 47LP
110A, y=-55500, 47LP
110A, y=-55600, 47LP
110A, y=-53000, 47LP
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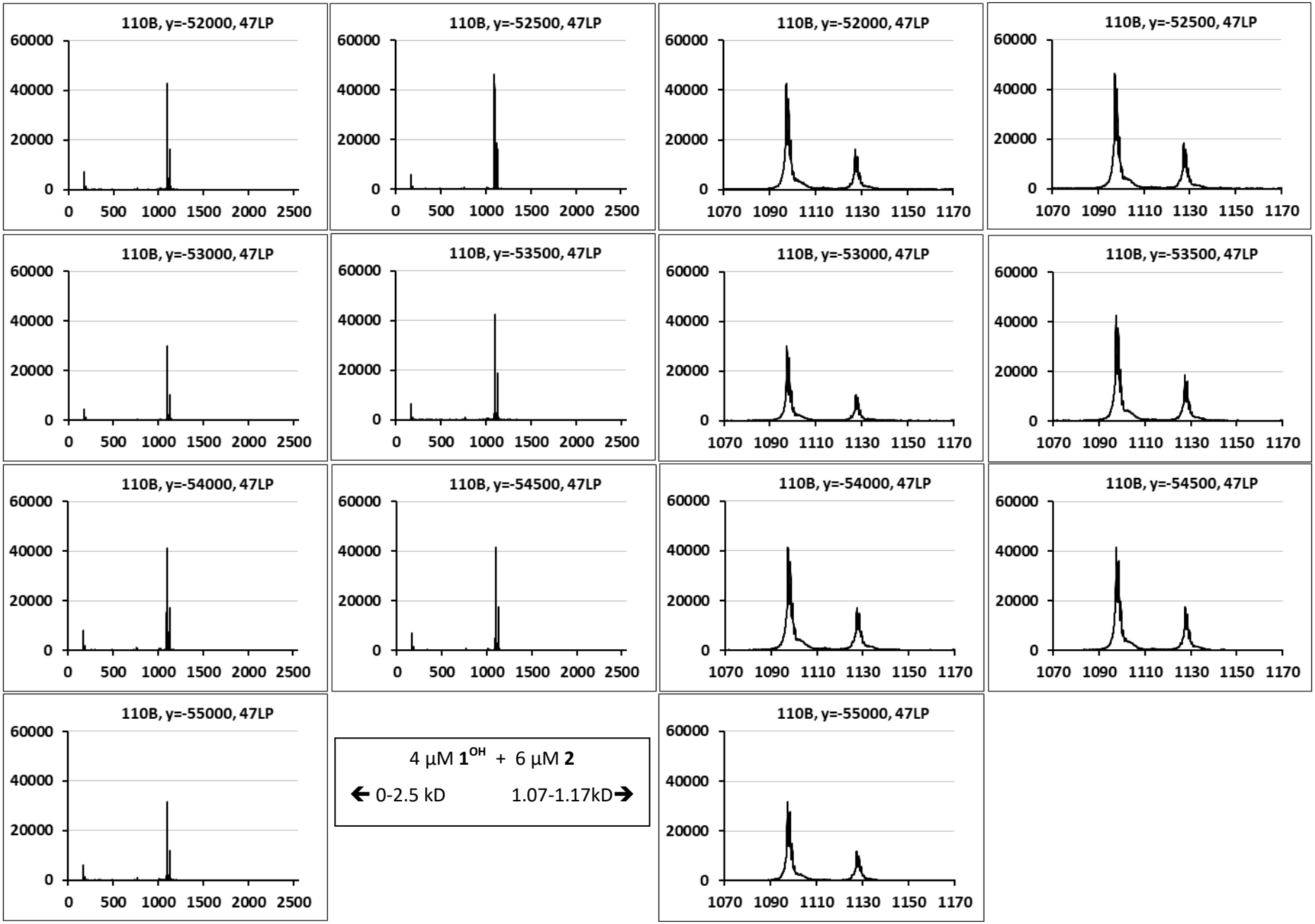
110A, y=-53500, 47LP
110A, y=-54000, 47LP
110A, y=-54500, 47LP
110A, y=-54000, 47LP
110A, y=-54500, 47LP
110A, y=-55000, 47LP
4 µM 1OH + 6 µM 2
← 0-2.5 kD
1.07-1.17kD →
110A, y=-55000, 47LP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110B, y=-52500, 47LP
110B, y=-52000, 47LP
110B, y=-52500, 47LP
110B, y=-53000, 47LP
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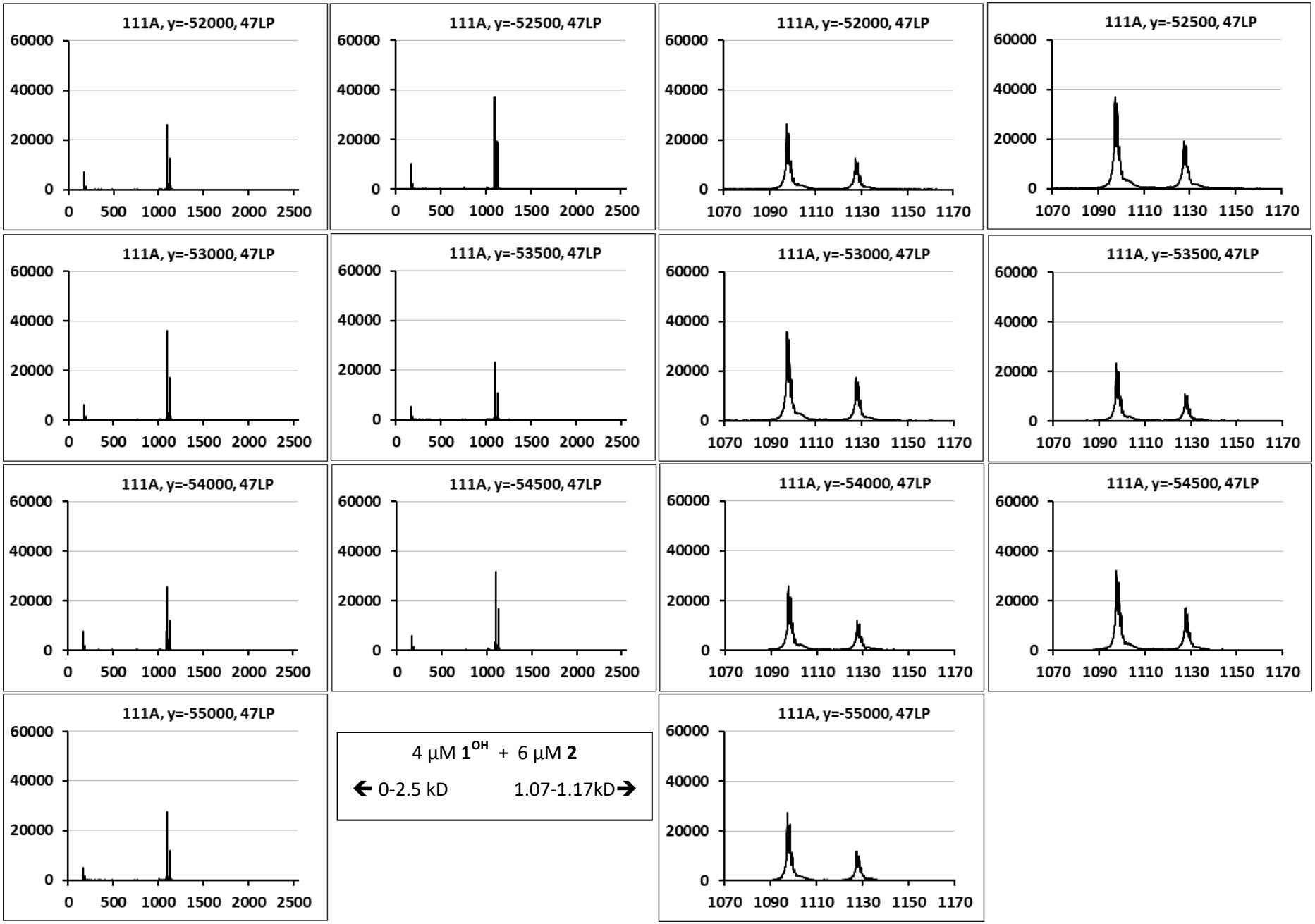
110B, y=-53000, 47LP
110B, y=-53500, 47LP
110B, y=-54000, 47LP
110B, y=-54500, 47LP
110B, y=-54000, 47LP
110B, y=-54500, 47LP
110B, y=-55000, 47LP
4 µM 1OH + 6 µM 2
← 0-2.5 kD
1.07-1.17kD→
110B, y=-55000, 47LP


111A, y=-52000, 47LP
111A, y=-52500, 47LP
111A, y=-52000, 47LP
111A, y=-52500, 47LP
111A, y=-53000, 47LP
111A, y=-53500, 47LP
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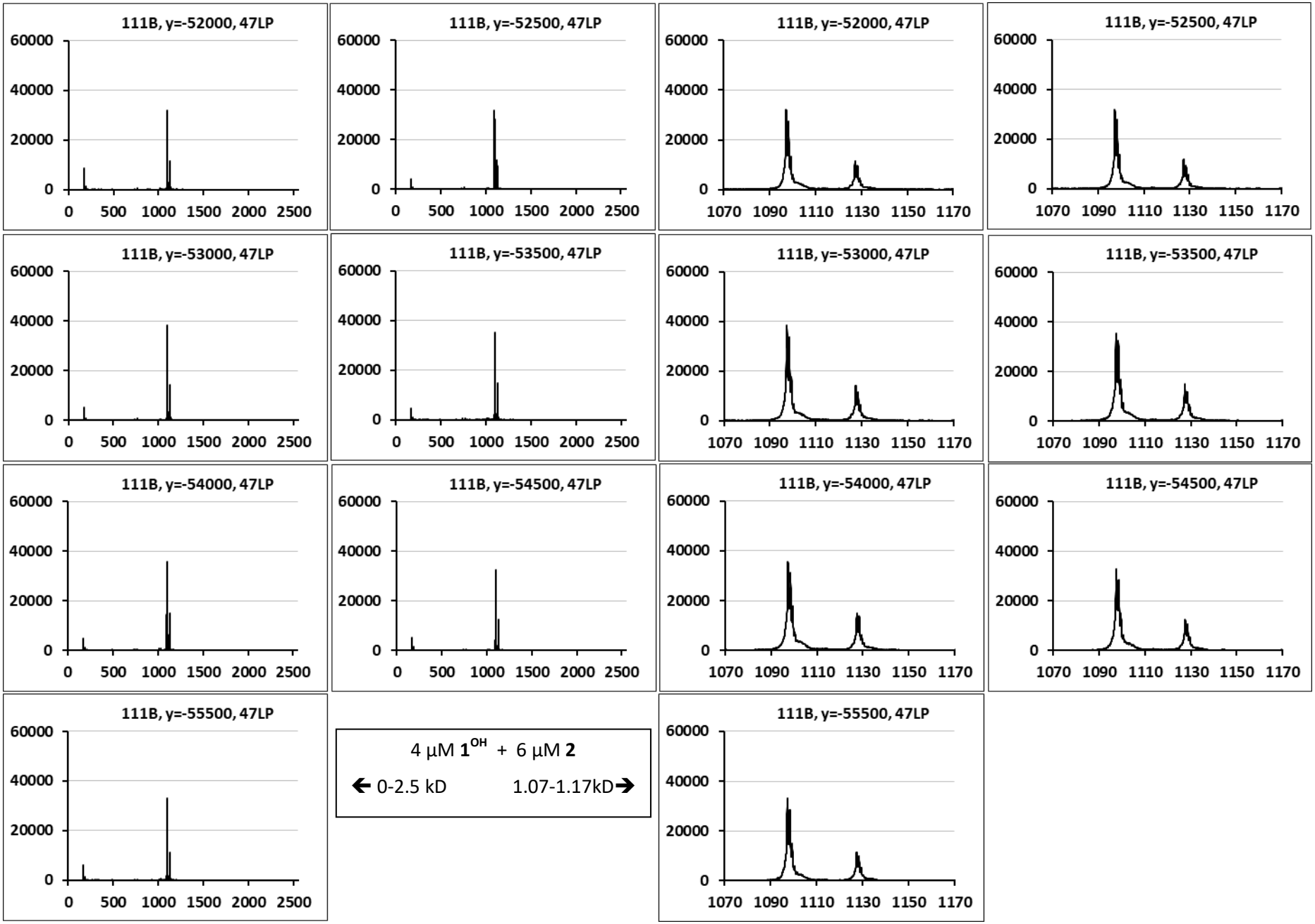
111A, y=-53500, 47LP
111A, y=-54000, 47LP
111A, y=-54500, 47LP
111A, y=-54000, 47LP
111A, y=-54500, 47LP
111A, y=-55000, 47LP
4 µM 1OH + 6 µM 2
← 0-2.5 kD
1.07-1.17kD→
111A, y=-55000, 47LP


111B, y=-52000, 47LP
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111B, y=-53000, 47LP
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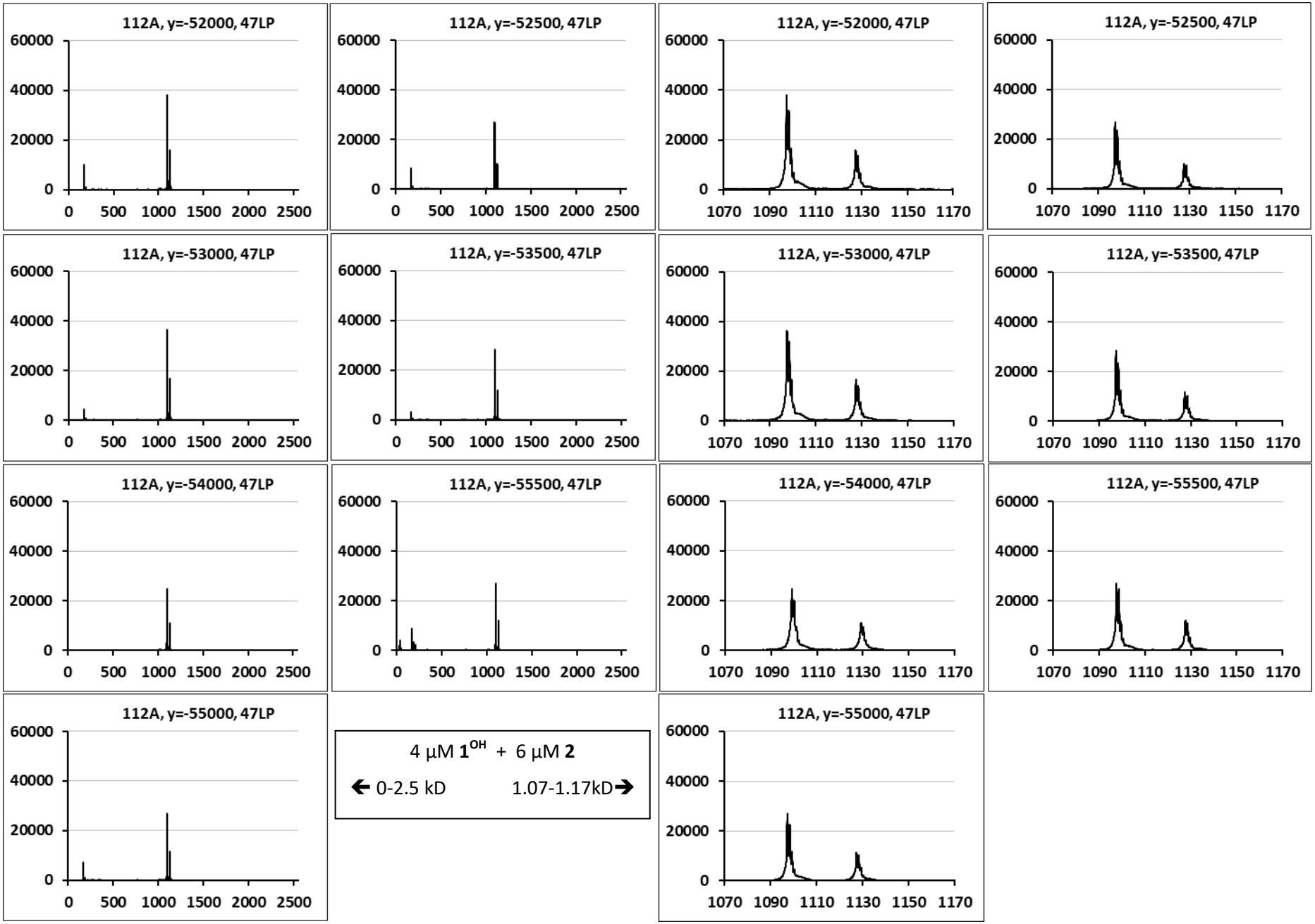
111B, y=-53500, 47LP
111B, y=-54000, 47LP
111B, y=-54500, 47LP
111B, y=-54000, 47LP
111B, y=-54500, 47LP
111B, y=-55500, 47LP
4 µM 1OH + 6 µM 2
← 0-2.5 kD
1.07-1.17kD→
111B, y=-55500, 47LP


112A, y=-52000, 47LP
112A, y=-52500, 47LP
112A, y=-52000, 47LP
112A, y=-52500, 47LP
112A, y=-53000, 47LP
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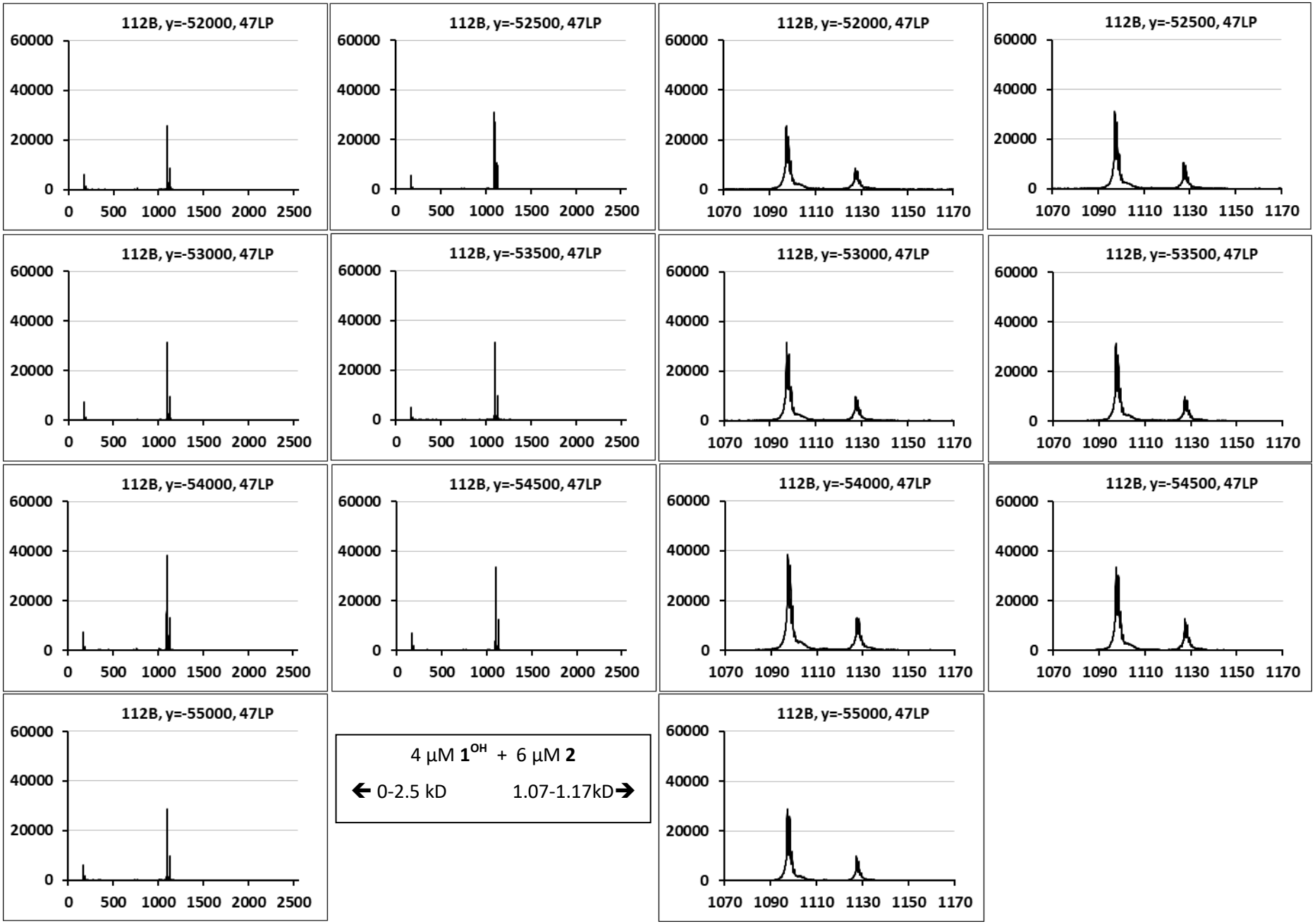
112A, y=-53000, 47LP
112A, y=-53500, 47LP
112A, y=-54000, 47LP
112A, y=-55500, 47LP
112A, y=-54000, 47LP
112A, y=-55500, 47LP
112A, y=-55000, 47LP
4 µM 1OH + 6 µM 2
← 0-2.5 kD
1.07-1.17kD →
112A, y=-55000, 47LP


112B, y=-52000, 47LP
112B, y=-52500, 47LP
112B, y=-52000, 47LP
112B, y=-52500, 47LP
112B, y=-53000, 47LP
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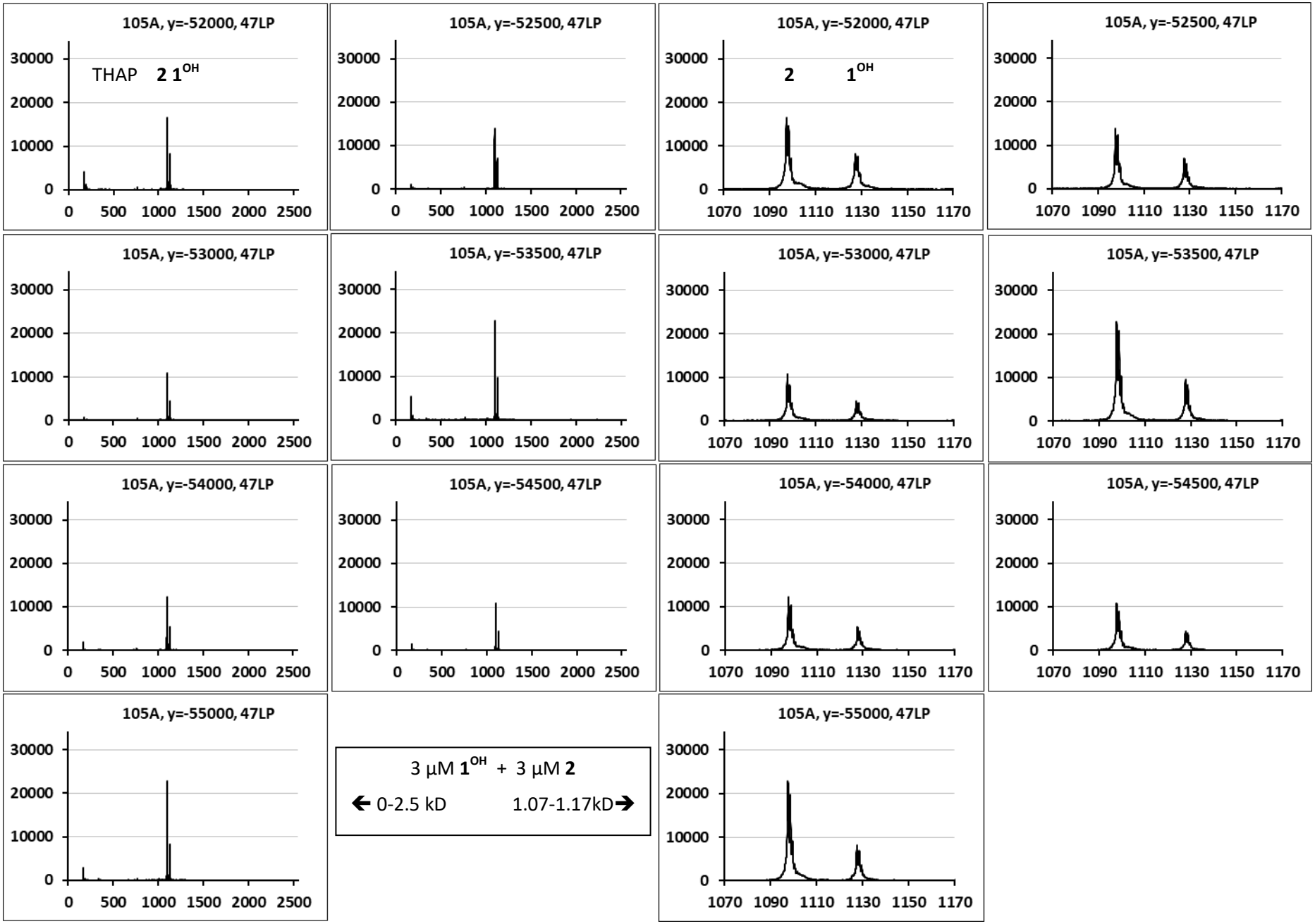
112B, y=-53000, 47LP
112B, y=-53500, 47LP
112B, y=-54000, 47LP
112B, y=-54500, 47LP
112B, y=-54000, 47LP
112B, y=-54500, 47LP
112B, y=-55000, 47LP
112B, y=-55000, 47LP
60000
40000
20000
0
0 500 1000 1500 2000 2500
1070 1090 1110 1130 1150 1170
4 µM 1OH + 6 µM 2
← 0-2.5 kD
1.07-1.17kD →

Summary of **1^{OH} / 2** phenyl octane solution with 1 : 1 concentration ratio: TOF-MS ML **1^{OH}** / **2** intensity and intensity ratio statistics from **1^{OH} / 2** phenyl octane solution with 1 : 1 concentration ratio (THAP thin film applied, LP = 47, TOF-MS measured at 14 locations on each of 4 independently prepared samples).

[1^{OH}]	[2]	# Locations	1^{OH} / 2	1^{OH} / 2	1^{OH} / 2	1^{OH} / 2	1^{OH} / 2	1^{OH}	1^{OH}	1^{OH}	1^{OH}	1^{OH}	2	2	2	2	2
µM	µM	(file names)	Max	Min	Avg	σ	σ/Avg	Max	Min	Avg	σ	σ/Avg	Max	Min	Avg	σ	σ/Avg
3	3	14 (105A,B)	0.51	0.27	0.38	0.08	20%	9644	4215	6143	1736	28%	22830	10782	16293	3780	23%
3	3	14 (106A,B)	0.60	0.39	0.48	0.07	15%	17533	5078	8589	3461	40%	31095	11492	17364	4941	28%
3	3	14 (107A,B)	0.59	0.32	0.47	0.09	18%	6624	2144	4842	1485	31%	14574	4911	10159	2422	24%
3	3	14 (108A,B)	0.53	0.38	0.46	0.04	10%	10487	3131	5818	2000	34%	22106	7149	12513	3822	31%
3	3	All	0.60	0.27	**0.45**	**0.08**	**18%**	17533	2144	**6348**	**2636**	**42%**	31095	4911	**14082**	**4745**	**34%**

[1^{OH}]	[2]	# Locations	1^{OH} / 2	1^{OH} / 2	1^{OH} / 2	1^{OH} / 2	1^{OH} / 2	1^{OH}	1^{OH}	1^{OH}	1^{OH}	1^{OH}	2	2	2	2	2
µM	µM	(file names)	Max	Min	Avg	σ	σ/Avg	Max	Min	Avg	σ	σ/Avg	Max	Min	Avg	σ	σ/Avg
4	4	14 (101A,B)	0.61	0.43	0.51	0.06	11%	21323	13650	17055	2292	13%	38278	29966	33571	2906	9%
4	4	14 (102A,B)	0.50	0.30	0.40	0.08	20%	14596	7989	10468	2336	22%	32976	17066	26578	4610	17%
4	4	14 (103A,B)	0.76	0.52	0.64	0.08	12%	29100	14971	20469	4035	20%	40570	23973	31932	4490	14%
4	4	14 (104A,B)	0.57	0.39	0.48	0.05	11%	12547	6756	9527	1470	15%	22768	16151	19930	1834	9%
4	4	All	0.76	0.30	**0.51**	**0.11**	**22%**	29100	6756	**14380**	**5295**	**37%**	40570	16151	**28003**	**6442**	**23%**

[1^{OH}]	[2]	# Locations	1^{OH} / 2	1^{OH} / 2	1^{OH} / 2	1^{OH} / 2	1^{OH} / 2	1^{OH}	1^{OH}	1^{OH}	1^{OH}	1^{OH}	2	2	2	2	2
µM	µM	(file names)	Max	Min	Avg	σ	σ/Avg	Max	Min	Avg	σ	σ/Avg	Max	Min	Avg	σ	σ/Avg
10	10	14 (141A,B)	22.0	1.8	8.9	7.7	86%	19298	5406	13538	4082	30%	6616	627	3049	2267	74%
10	10	14 (142A,B)	21.0	2.6	9.9	5.4	55%	26763	9000	18336	5609	31%	6405	652	2709	1887	70%
10	10	14 (143A,B)	7.6	1.3	3.7	1.8	48%	27105	11128	18871	4243	22%	12815	3540	6205	2963	48%
10	10	14 (144A,B)	15.6	4.9	9.0	2.9	32%	23140	7559	12790	4269	33%	3174	484	1623	838	52%
10	10	All	22.0	1.3	**7.9**	**5.5**	**69%**	27105	5406	**15884**	**5251**	**33%**	12815	484	**3396**	**2694**	**79%**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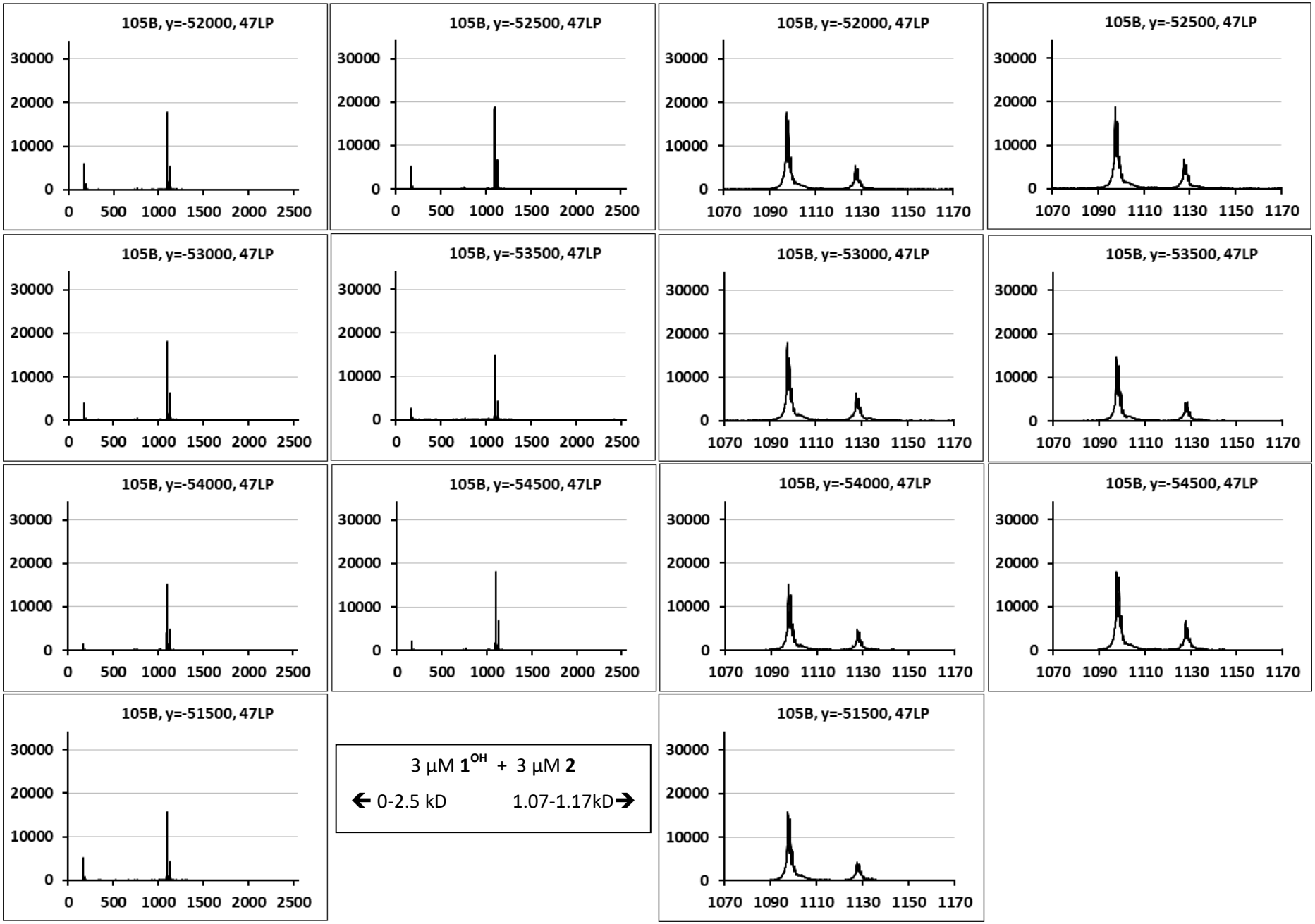
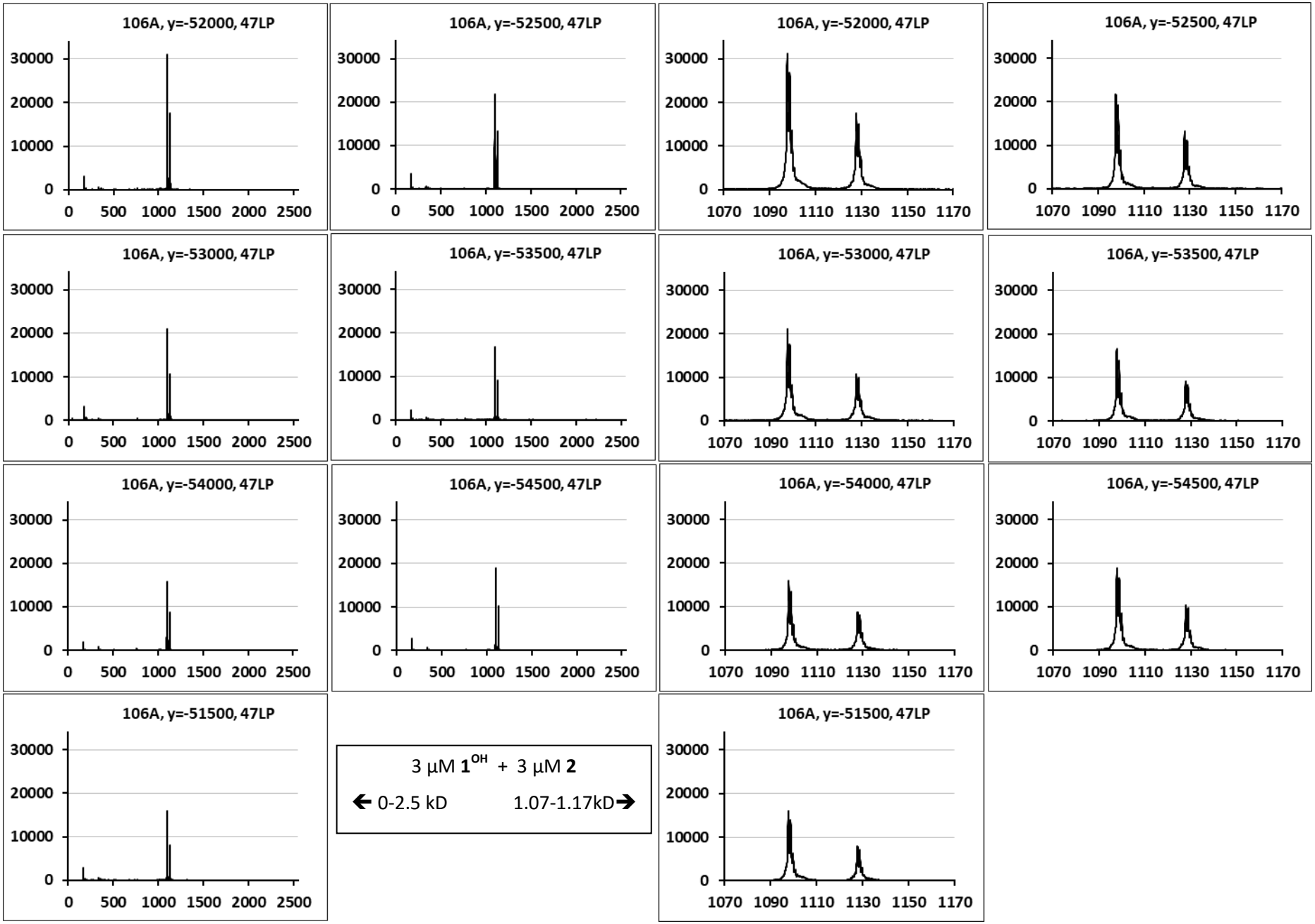
THAP 2 1OH
105A, y=-52500, 47LP
105A, y=-52000, 47LP
2 1OH
105A, y=-52500, 47LP
105A, y=-53000, 47LP
105A, y=-53500, 47LP
105A, y=-53000, 47LP
105A, y=-53500, 47LP
105A, y=-54000, 47LP
105A, y=-54500, 47LP
105A, y=-54000, 47LP
105A, y=-54500, 47LP
105A, y=-55000, 47LP
3 µM 1OH + 3 µM 2
← 0-2.5 kD
1.07-1.17kD→
105A, y=-55000, 47LP


105B, y=-52000, 47LP
105B, y=-52500, 47LP
105B, y=-52000, 47LP
105B, y=-52500, 47LP
105B, y=-53000, 47LP
105B, y=-53500, 47LP
105B, y=-53000, 47LP
105B, y=-53500, 47LP
105B, y=-54000, 47LP
105B, y=-54500, 47LP
105B, y=-54000, 47LP
105B, y=-54500, 47LP
105B, y=-51500, 47LP
105B, y=-51500, 47LP
30000
20000
10000
0
0 500 1000 1500 2000 2500
1070 1090 1110 1130 1150 1170
3 µM 1OH + 3 µM 2
← 0-2.5 kD
1.07-1.17kD→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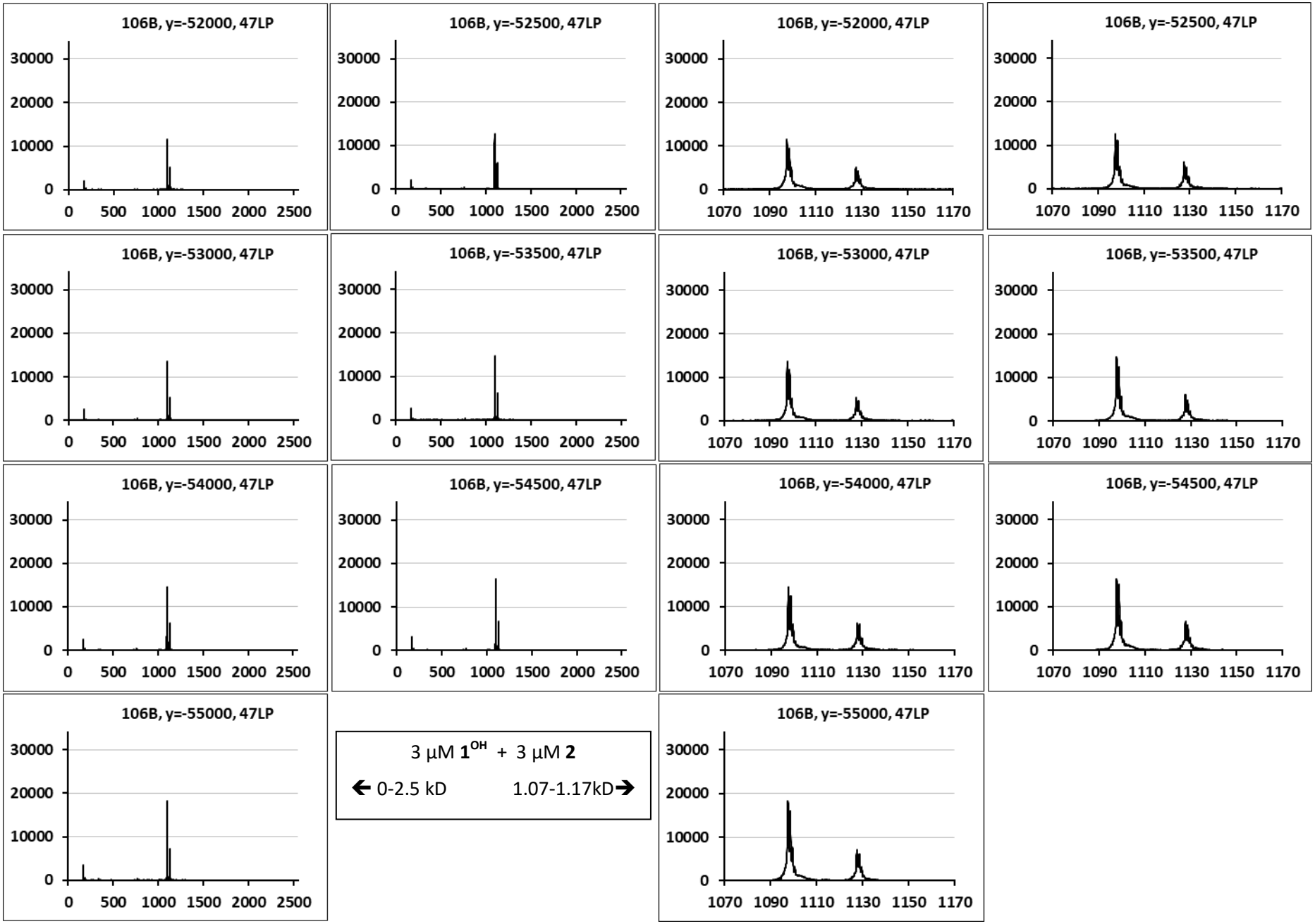
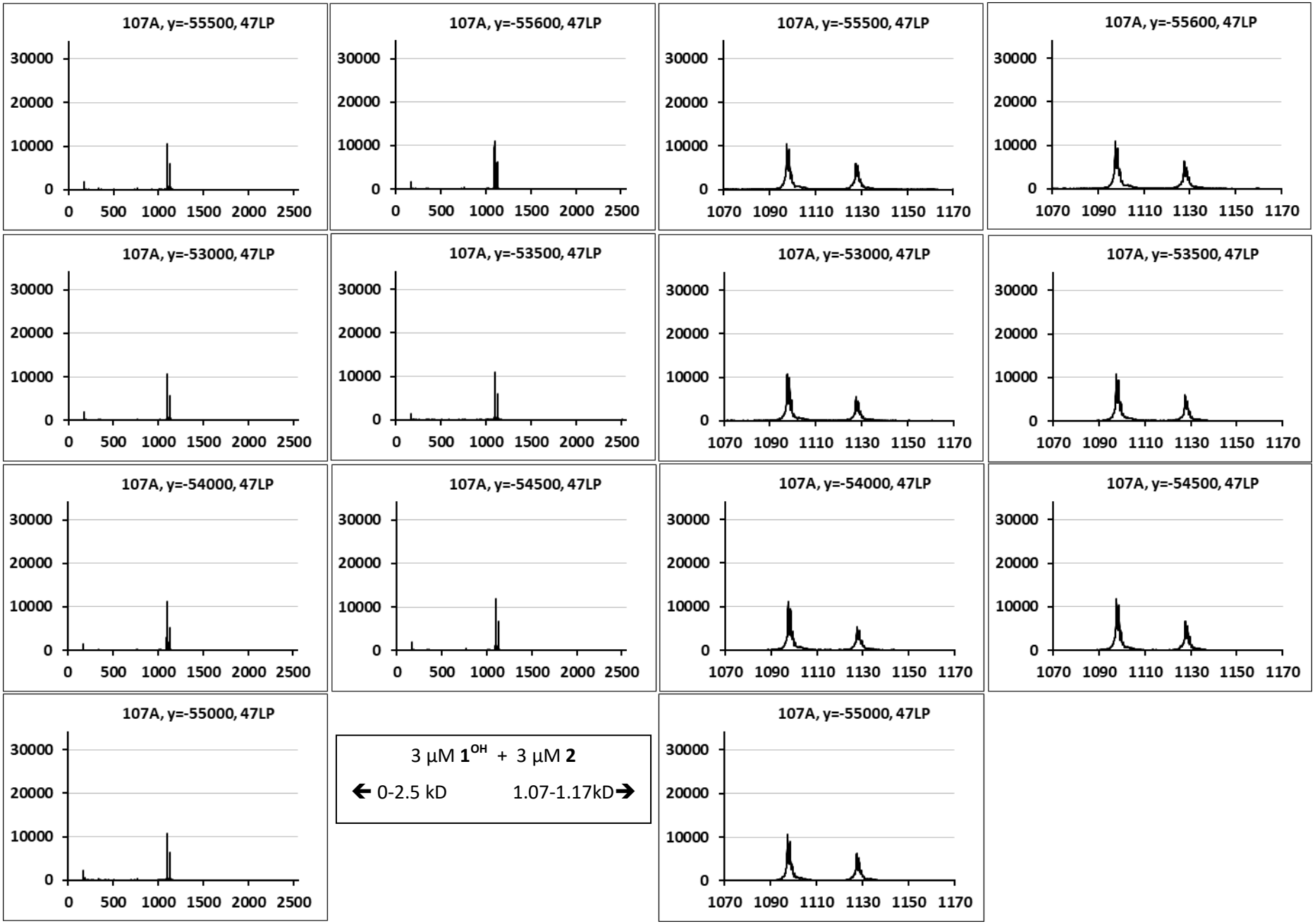
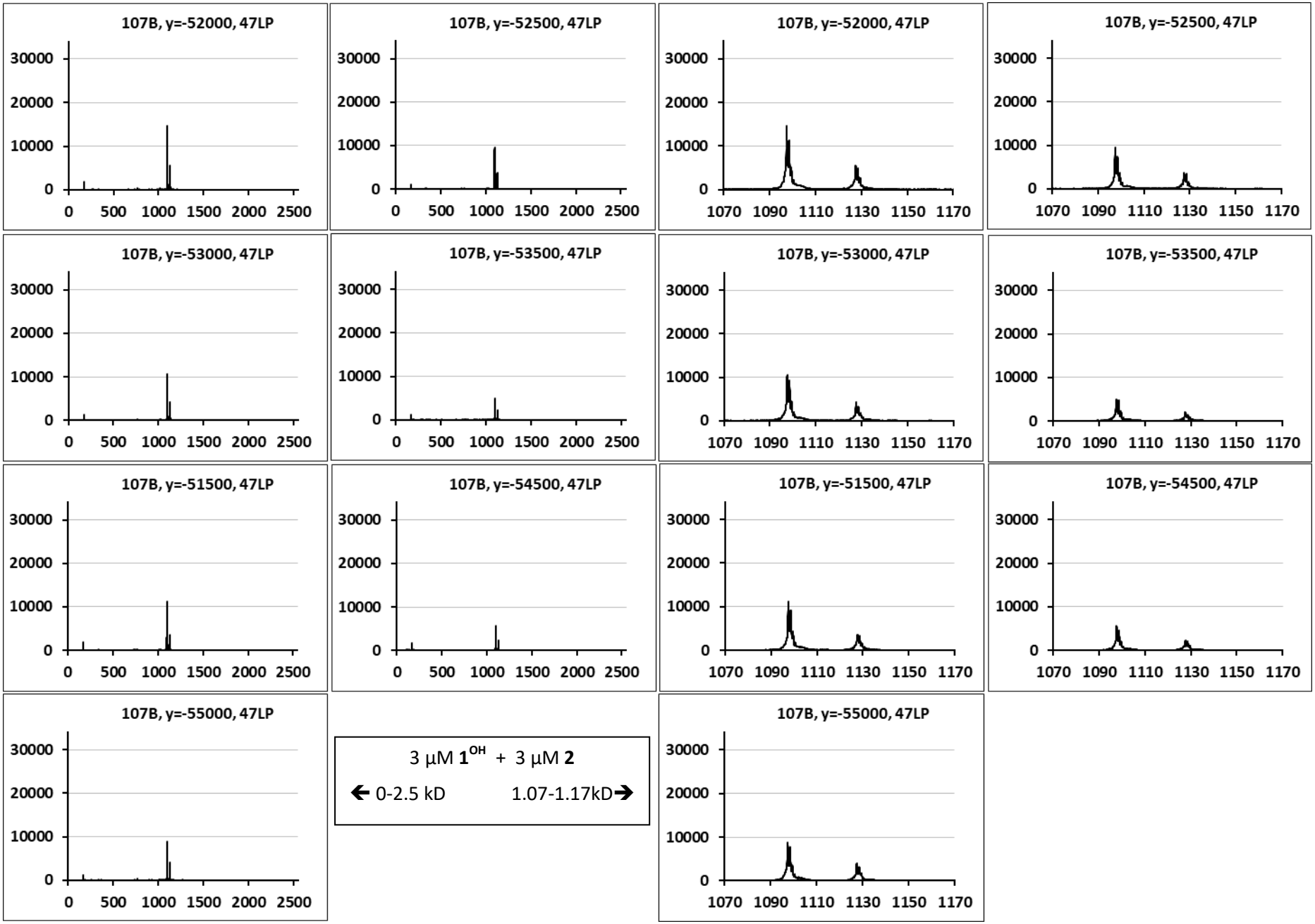
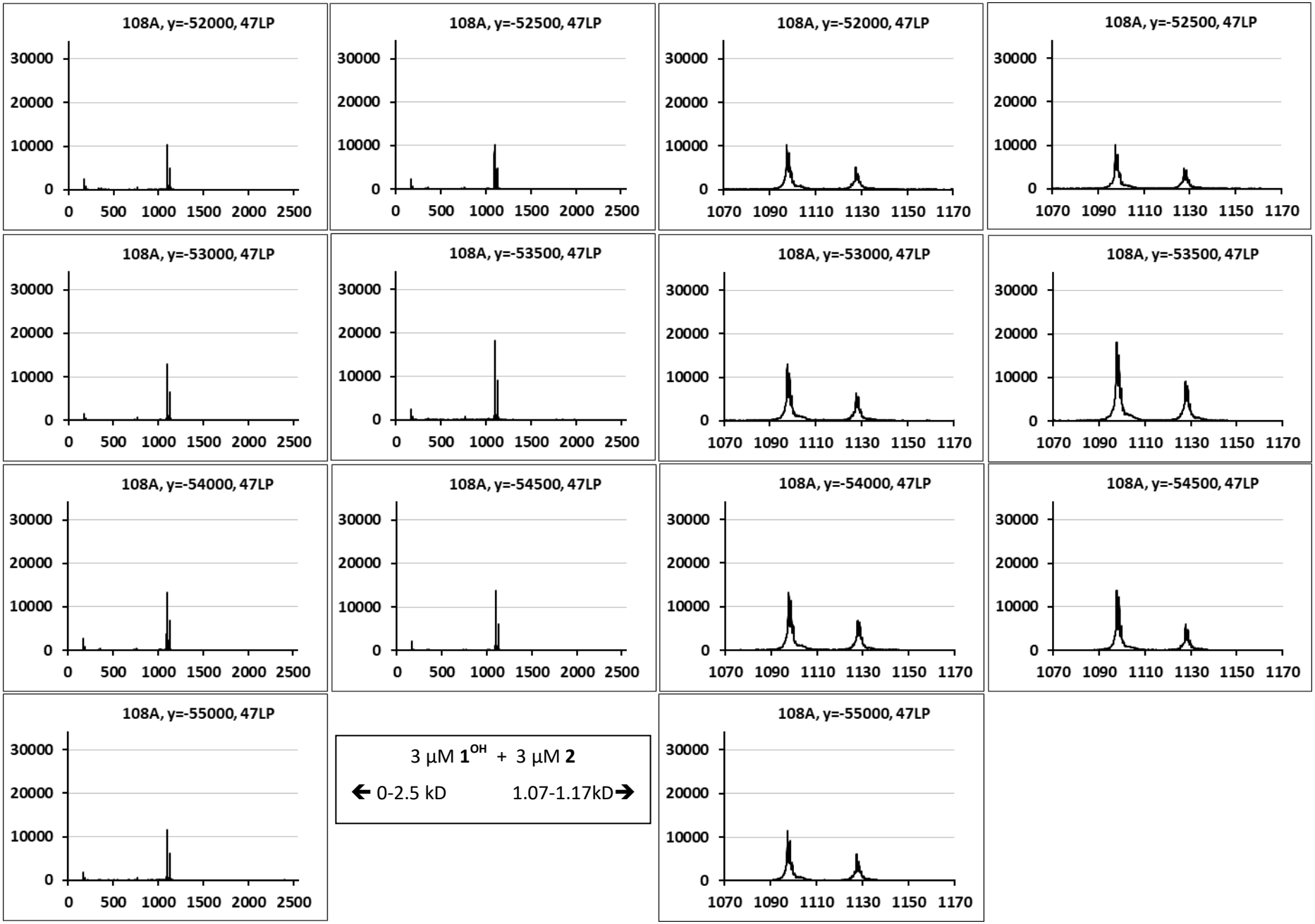
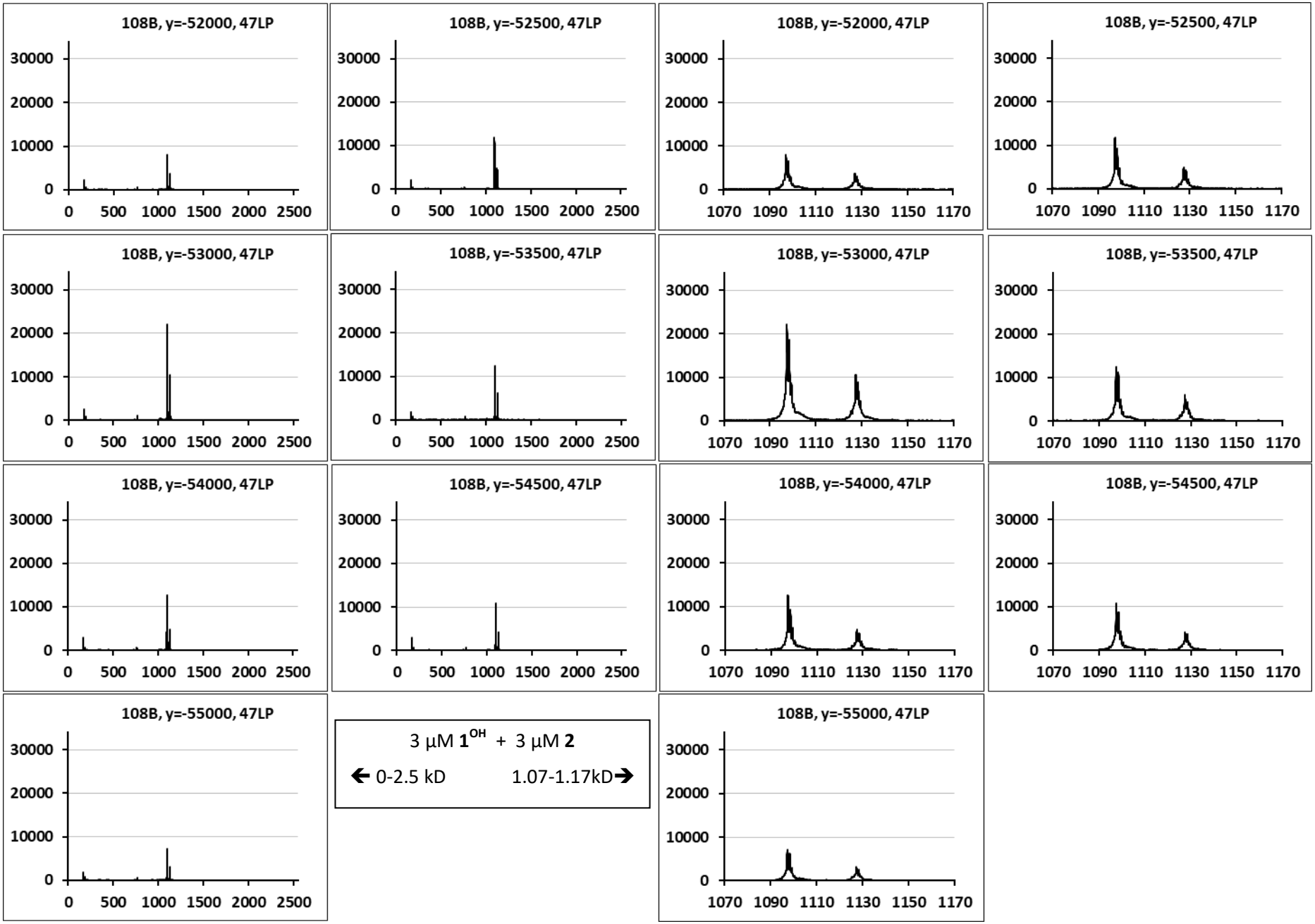
106A, y=-52500, 47LP
106A, y=-52000, 47LP
106A, y=-52500, 47LP
106A, y=-53000, 47LP
106A, y=-53500, 47LP
106A, y=-53000, 47LP
106A, y=-53500, 47LP
106A, y=-54000, 47LP
106A, y=-54500, 47LP
106A, y=-54000, 47LP
106A, y=-54500, 47LP
106A, y=-51500, 47LP
106A, y=-51500, 47LP
30000
20000
10000
0
0 500 1000 1500 2000 2500
1070 1090 1110 1130 1150 1170
3 µM 1OH + 3 µM 2
← 0-2.5 kD
1.07-1.17kD→

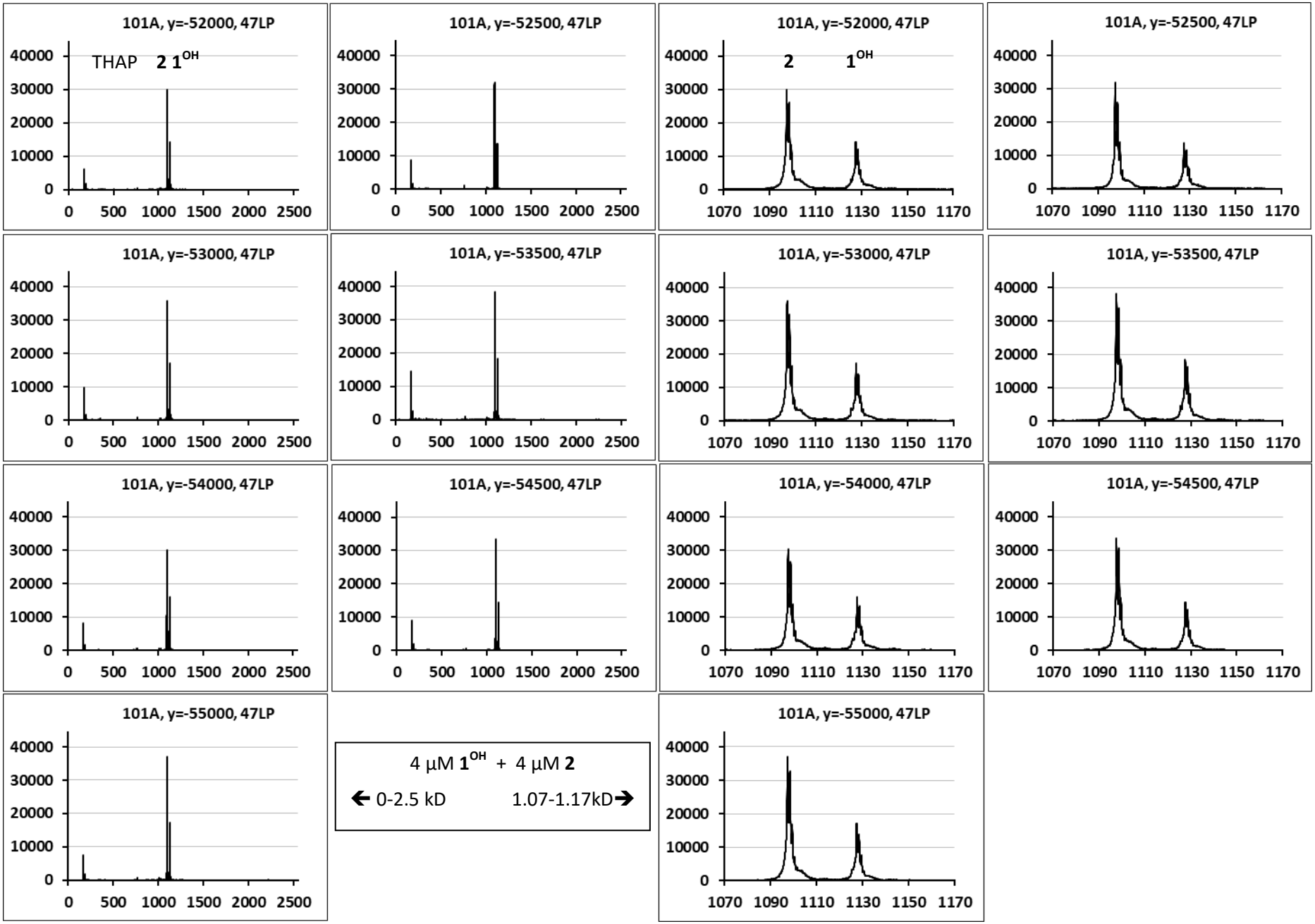

106B, y=-52000, 47LP
106B, y=-52500, 47LP
106B, y=-52000, 47LP
106B, y=-52500, 47LP
106B, y=-53000, 47LP
106B, y=-53500, 47LP
106B, y=-53000, 47LP
106B, y=-53500, 47LP
106B, y=-54000, 47LP
106B, y=-54500, 47LP
106B, y=-54000, 47LP
106B, y=-54500, 47LP
106B, y=-55000, 47LP
106B, y=-55000, 47LP
30000
20000
10000
0
0 500 1000 1500 2000 2500
1070 1090 1110 1130 1150 1170
3 µM 1OH + 3 µM 2
← 0-2.5 kD
1.07-1.17kD →

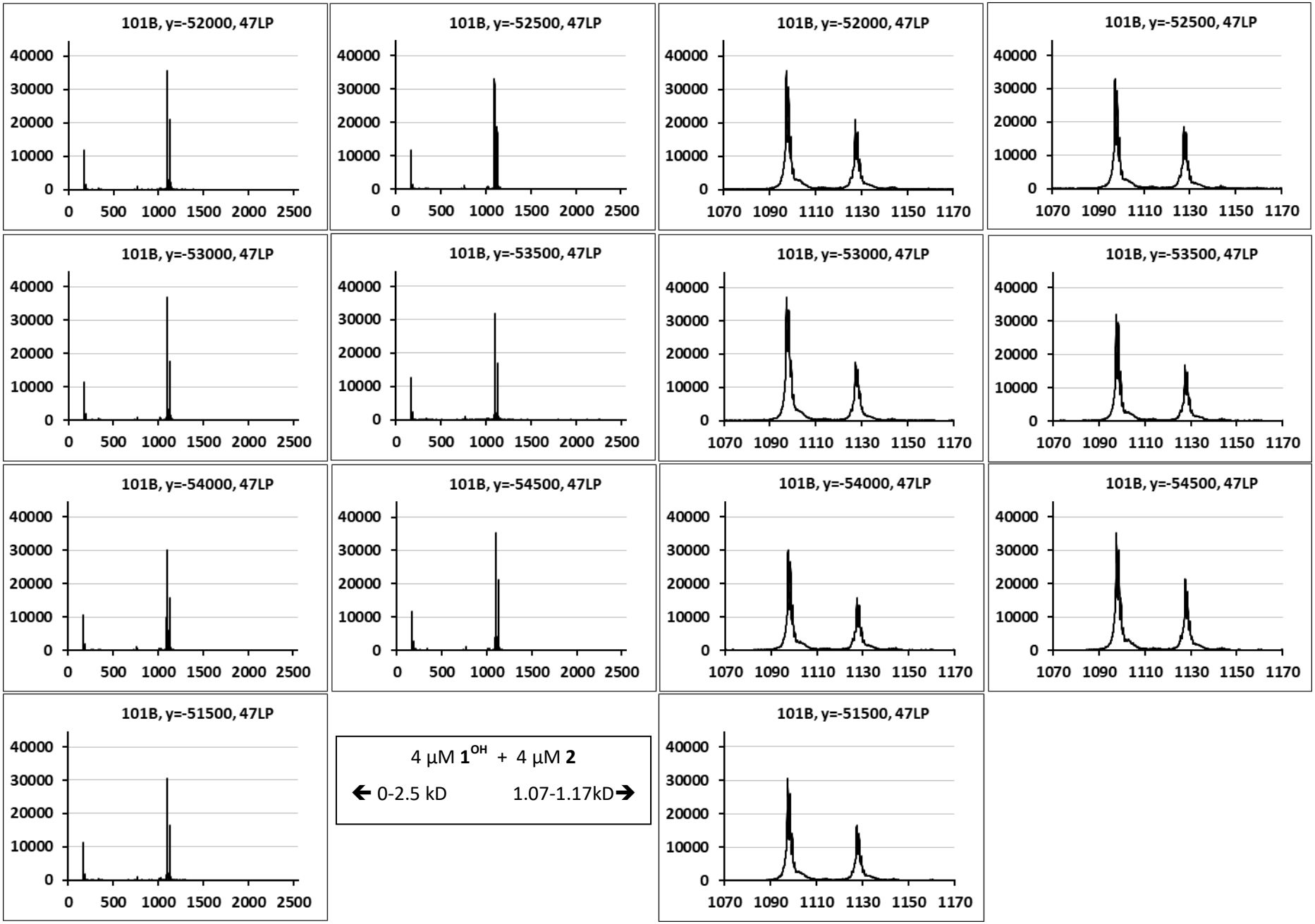

107A, y=-55500, 47LP
107A, y=-55600, 47LP
107A, y=-55500, 47LP
107A, y=-55600, 47LP
107A, y=-53000, 47LP
107A, y=-53500, 47LP
107A, y=-53000, 47LP
107A, y=-53500, 47LP
107A, y=-54000, 47LP
107A, y=-54500, 47LP
107A, y=-54000, 47LP
107A, y=-54500, 47LP
107A, y=-55000, 47LP
3 µM 1OH + 3 µM 2
← 0-2.5 kD
1.07-1.17kD→
107A, y=-55000, 47LP

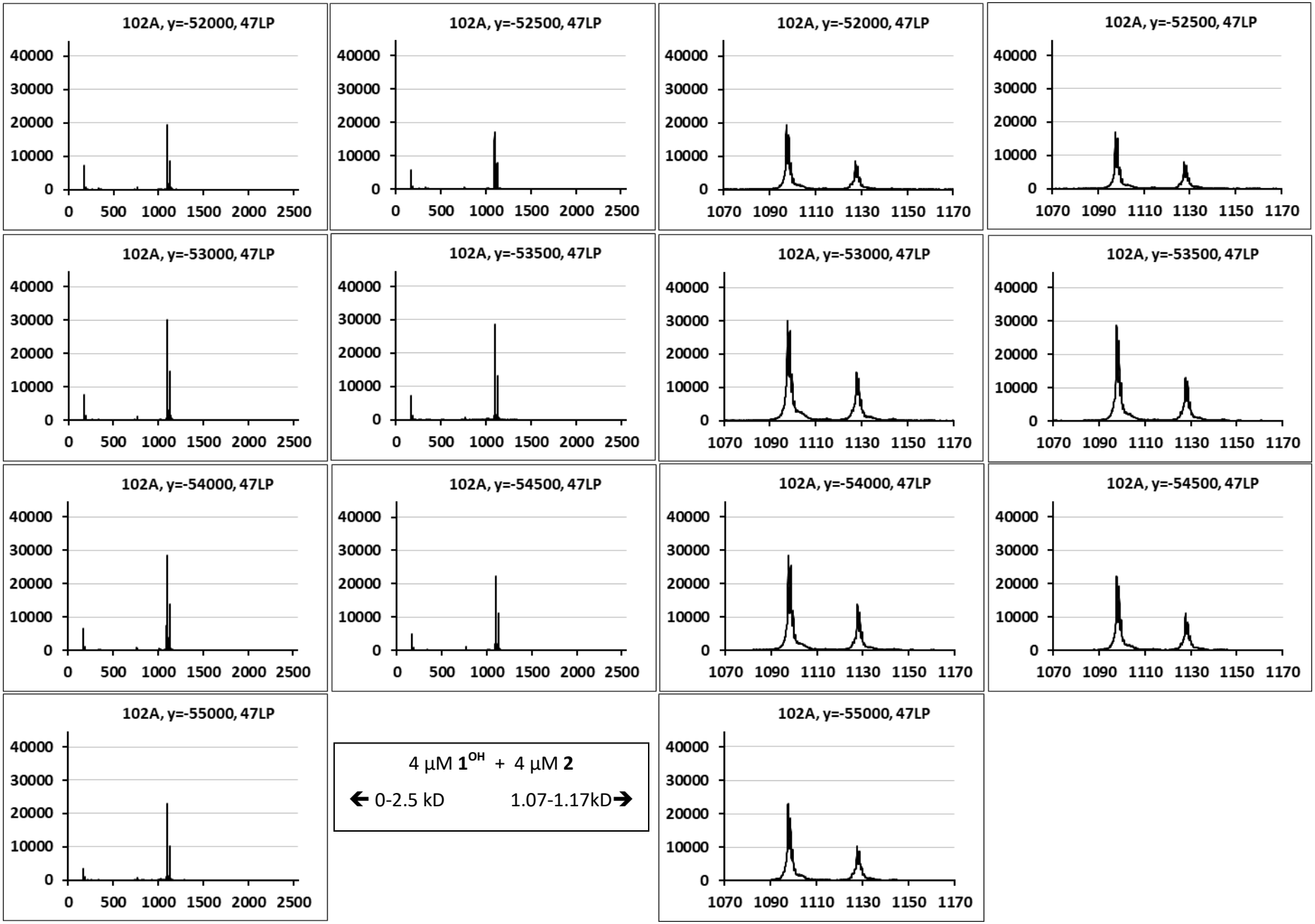

107B, y=-52000, 47LP
107B, y=-52500, 47LP
107B, y=-52000, 47LP
107B, y=-52500, 47LP
107B, y=-53000, 47LP
107B, y=-53500, 47LP
107B, y=-53000, 47LP
107B, y=-53500, 47LP
107B, y=-51500, 47LP
107B, y=-54500, 47LP
107B, y=-51500, 47LP
107B, y=-54500, 47LP
107B, y=-55000, 47LP
107B, y=-55000, 47LP
30000
20000
10000
0
0 500 1000 1500 2000 2500
1070 1090 1110 1130 1150 1170
3 µM 1OH + 3 µM 2
← 0-2.5 kD
1.07-1.17kD→

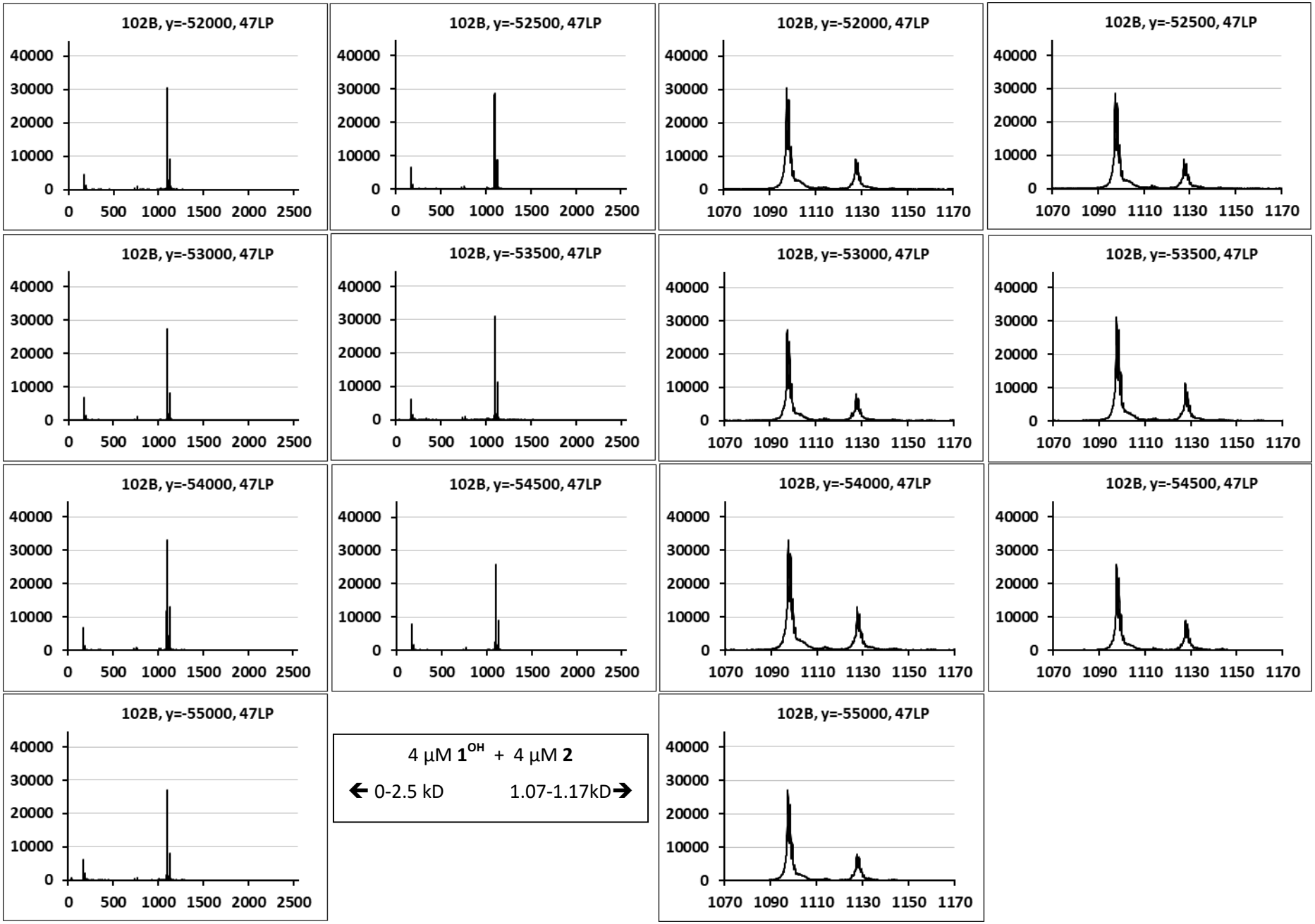

108A, y=-52000, 47LP
108A, y=-52500, 47LP
108A, y=-52000, 47LP
108A, y=-52500, 47LP
108A, y=-53000, 47LP
108A, y=-53500, 47LP
108A, y=-53000, 47LP
108A, y=-53500, 47LP
108A, y=-54000, 47LP
108A, y=-54500, 47LP
108A, y=-54000, 47LP
108A, y=-54500, 47LP
108A, y=-55000, 47LP
3 µM 1OH + 3 µM 2
← 0-2.5 kD
1.07-1.17kD→
108A, y=-55000, 47LP

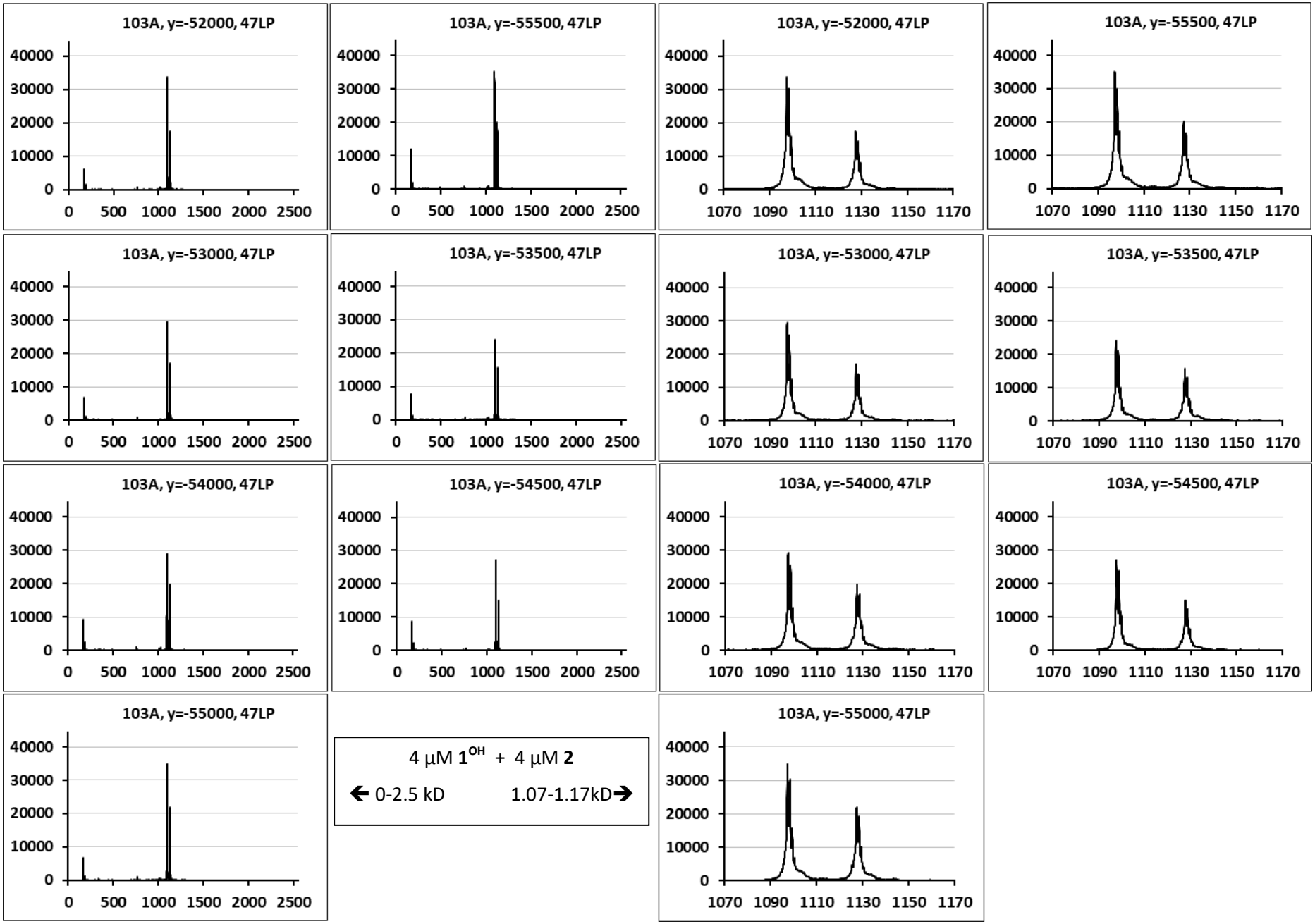

108B, y=-52000, 47LP
108B, y=-52500, 47LP
108B, y=-52000, 47LP
108B, y=-52500, 47LP
108B, y=-53000, 47LP
108B, y=-53500, 47LP
108B, y=-53000, 47LP
108B, y=-53500, 47LP
108B, y=-54000, 47LP
108B, y=-54500, 47LP
108B, y=-54000, 47LP
108B, y=-54500, 47LP
108B, y=-55000, 47LP
3 µM 1OH + 3 µM 2
← 0-2.5 kD
1.07-1.17kD→
108B, y=-55000, 47LP

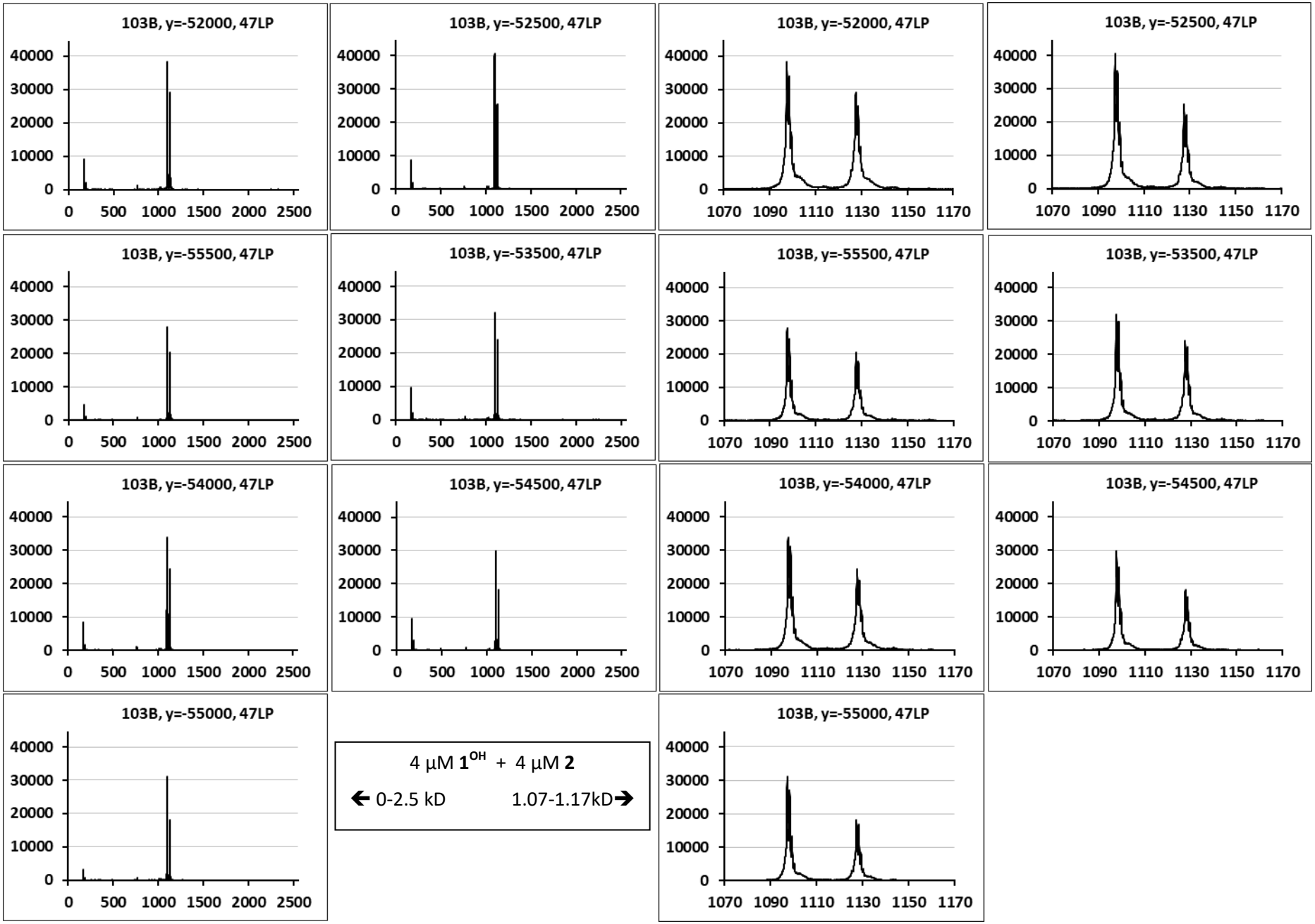

101A, y=-52000, 47LP
THAP 2 1OH
101A, y=-52500, 47LP
101A, y=-52000, 47LP
2 1OH
101A, y=-52500, 47LP
101A, y=-53000, 47LP
101A, y=-53500, 47LP
101A, y=-53000, 47LP
101A, y=-53500, 47LP
101A, y=-54000, 47LP
101A, y=-54500, 47LP
101A, y=-54000, 47LP
101A, y=-54500, 47LP
101A, y=-55000, 47LP
4 µM 1OH + 4 µM 2
← 0-2.5 kD
1.07-1.17kD →
101A, y=-55000, 47LP

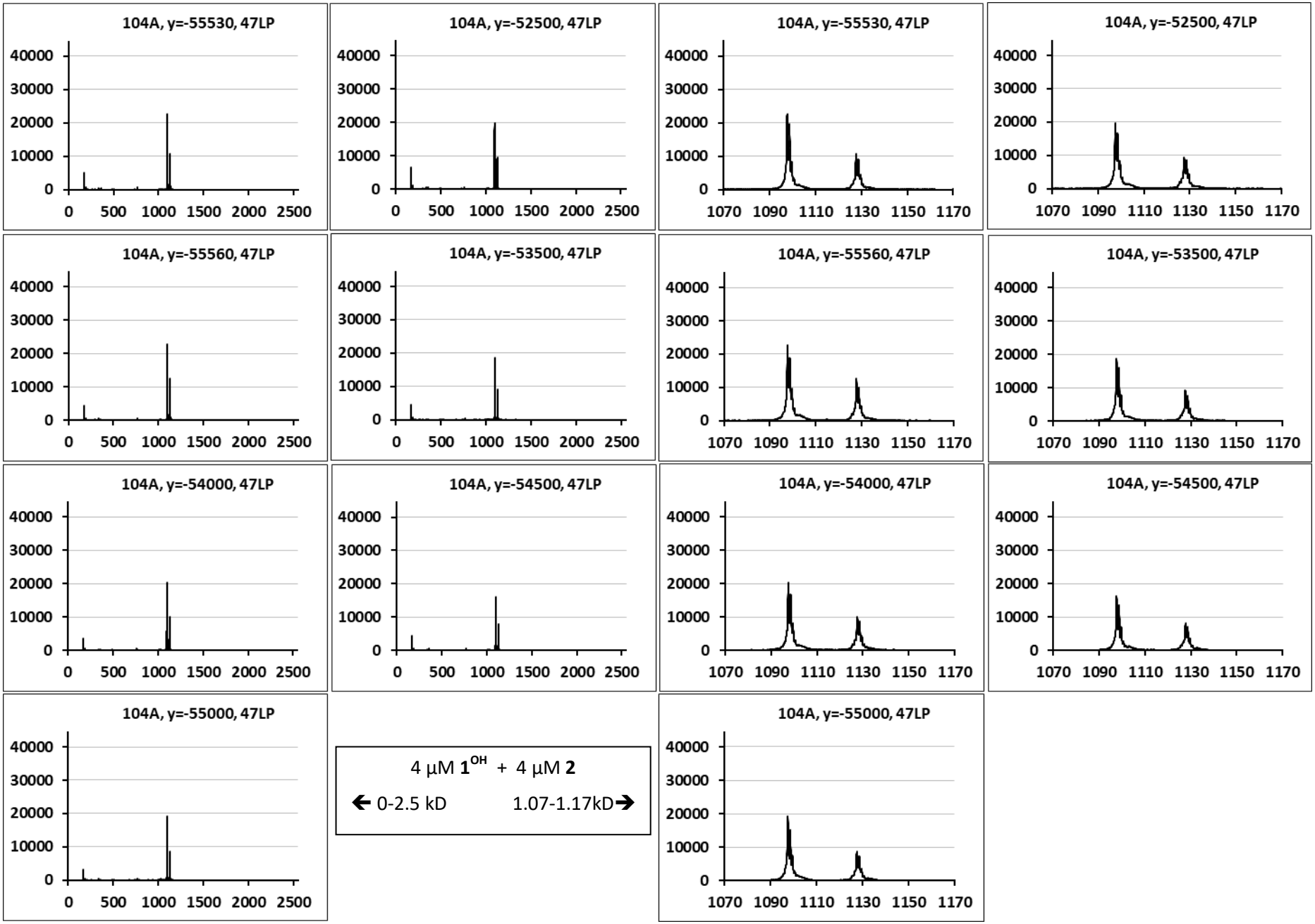

101B, y=-52000, 47LP
101B, y=-52500, 47LP
101B, y=-52000, 47LP
101B, y=-52500, 47LP
101B, y=-53000, 47LP
101B, y=-53500, 47LP
101B, y=-53000, 47LP
101B, y=-53500, 47LP
101B, y=-54000, 47LP
101B, y=-54500, 47LP
101B, y=-54000, 47LP
101B, y=-54500, 47LP
101B, y=-51500, 47LP
101B, y=-51500, 47LP
40000
30000
20000
10000
0
0 500 1000 1500 2000 2500
1070 1090 1110 1130 1150 1170
4 µM 1OH + 4 µM 2
← 0-2.5 kD
1.07-1.17kD→


102A, y=-52000, 47LP
102A, y=-52500, 47LP
102A, y=-52000, 47LP
102A, y=-52500, 47LP
102A, y=-53000, 47LP
102A, y=-53500, 47LP
102A, y=-53000, 47LP
102A, y=-53500, 47LP
102A, y=-54000, 47LP
102A, y=-54500, 47LP
102A, y=-54000, 47LP
102A, y=-54500, 47LP
102A, y=-55000, 47LP
4 µM 1OH + 4 µM 2
← 0-2.5 kD
1.07-1.17kD→
102A, y=-55000, 47LP


102B, y=-52000, 47LP
102B, y=-52500, 47LP
102B, y=-52000, 47LP
102B, y=-52500, 47LP
102B, y=-53000, 47LP
102B, y=-53500, 47LP
102B, y=-53000, 47LP
102B, y=-53500, 47LP
102B, y=-54000, 47LP
102B, y=-54500, 47LP
102B, y=-54000, 47LP
102B, y=-54500, 47LP
102B, y=-55000, 47LP
102B, y=-55000, 47LP
40000
30000
20000
10000
0
0 500 1000 1500 2000 2500
1070 1090 1110 1130 1150 1170
4 µM 1OH + 4 µM 2
← 0-2.5 kD
1.07-1.17kD →


103A, y=-52000, 47LP
103A, y=-55500, 47LP
103A, y=-52000, 47LP
103A, y=-55500, 47LP
103A, y=-53000, 47LP
103A, y=-53500, 47LP
103A, y=-53000, 47LP
103A, y=-53500, 47LP
103A, y=-54000, 47LP
103A, y=-54500, 47LP
103A, y=-54000, 47LP
103A, y=-54500, 47LP
103A, y=-55000, 47LP
103A, y=-55000, 47LP
4 µM 1OH + 4 µM 2
← 0-2.5 kD
1.07-1.17kD→


103B, y=-52000, 47LP
103B, y=-52500, 47LP
103B, y=-52000, 47LP
103B, y=-52500, 47LP
103B, y=-55500, 47LP
103B, y=-53500, 47LP
103B, y=-55500, 47LP
103B, y=-53500, 47LP
103B, y=-54000, 47LP
103B, y=-54500, 47LP
103B, y=-54000, 47LP
103B, y=-54500, 47LP
103B, y=-55000, 47LP
4 µM 1OH + 4 µM 2
← 0-2.5 kD
1.07-1.17kD→
103B, y=-55000, 47LP


104A, y=-55530, 47LP
104A, y=-52500, 47LP
104A, y=-55530, 47LP
104A, y=-52500, 47LP
104A, y=-55560, 47LP
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104A, y=-53500, 47LP
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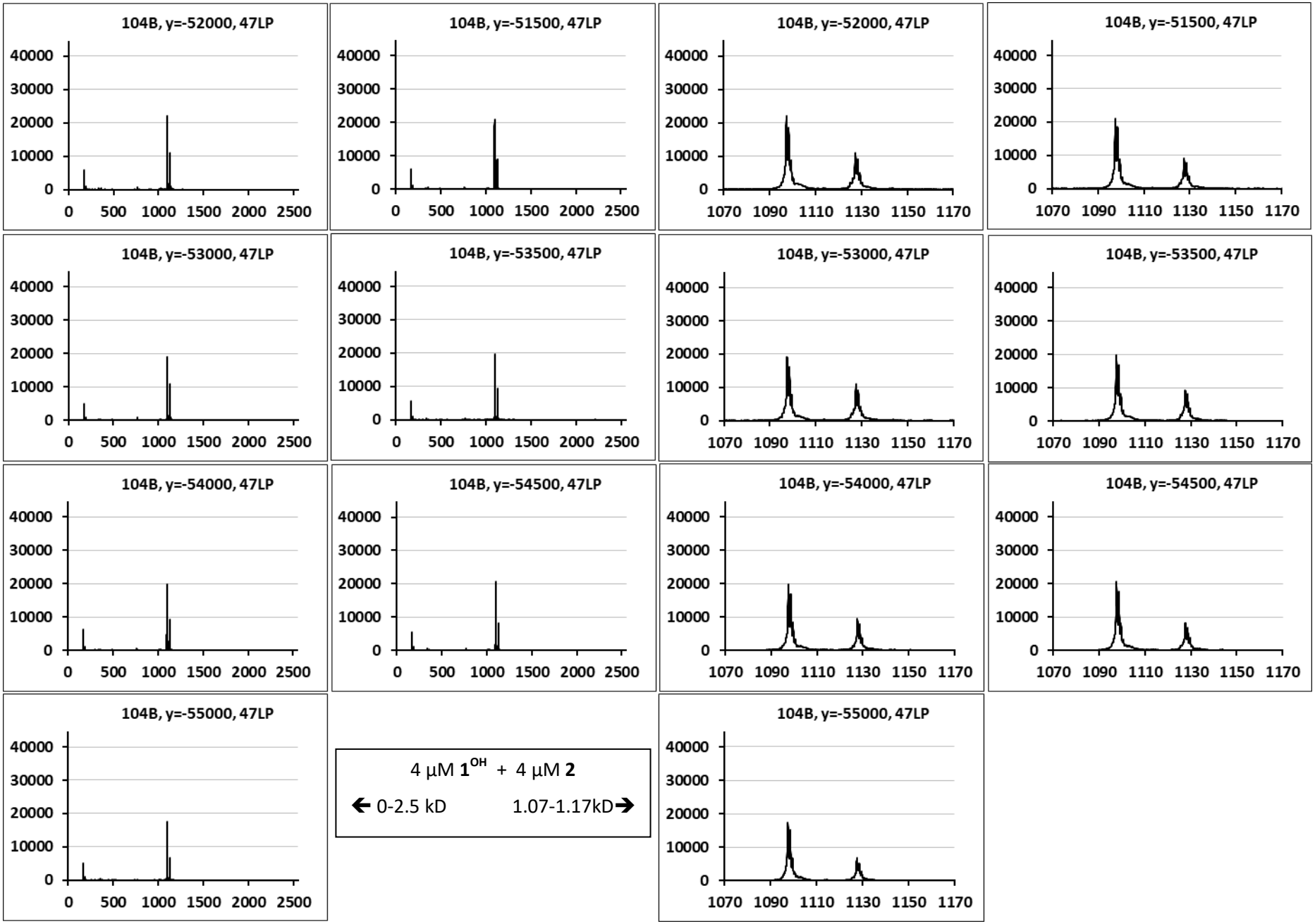
104A, y=-54500, 47LP
104A, y=-54000, 47LP
104A, y=-54500, 47LP
104A, y=-55000, 47LP
104A, y=-55000, 47LP
4 µM 1OH + 4 µM 2
← 0-2.5 kD
1.07-1.17kD→


104B, y=-52000, 47LP
104B, y=-51500, 47LP
104B, y=-52000, 47LP
104B, y=-51500, 47LP
104B, y=-53000, 47LP
104B, y=-53500, 47LP
104B, y=-53000, 47LP
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104B, y=-54000, 47LP
104B, y=-54500, 47LP
104B, y=-54000, 47LP
104B, y=-54500, 47LP
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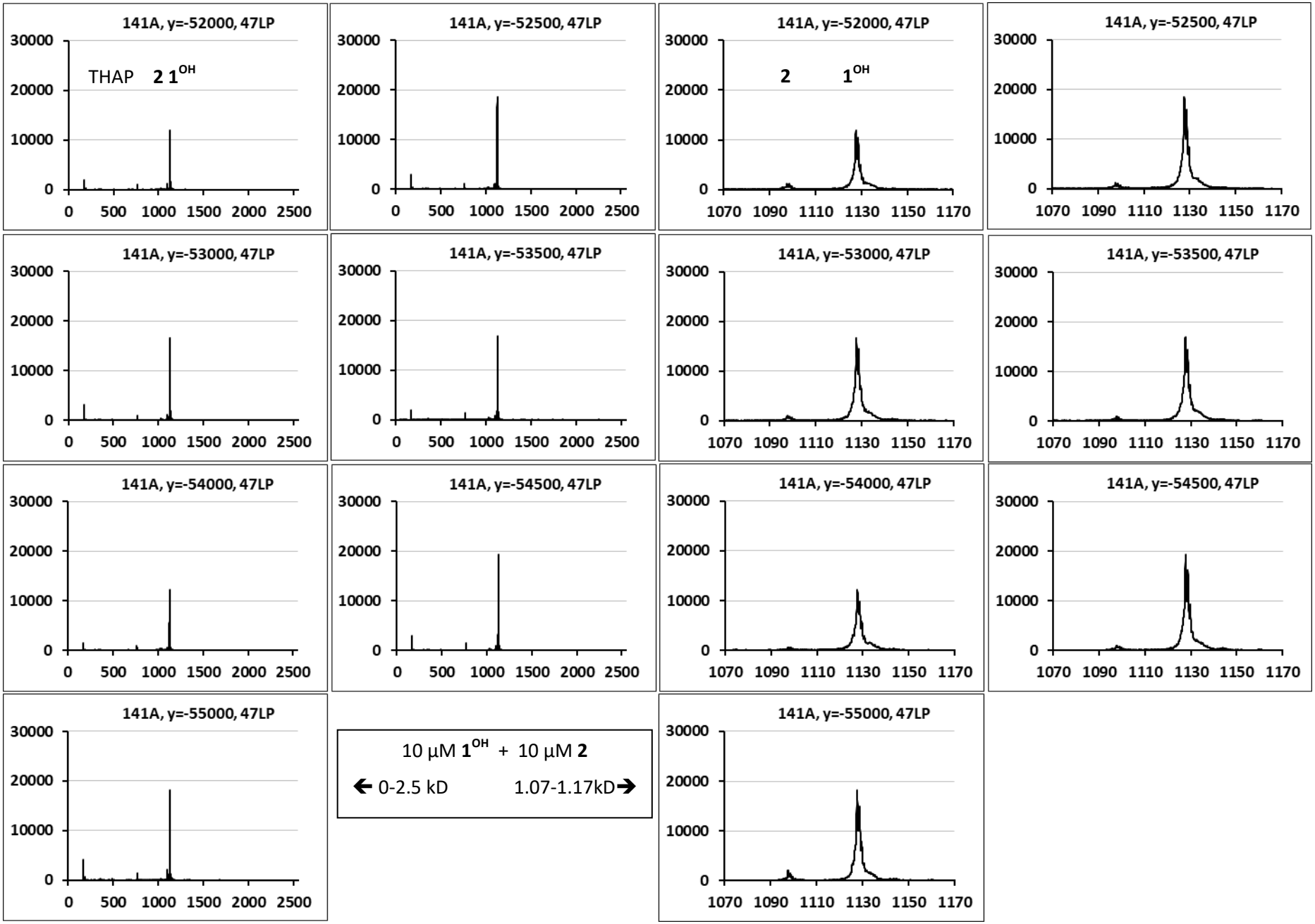
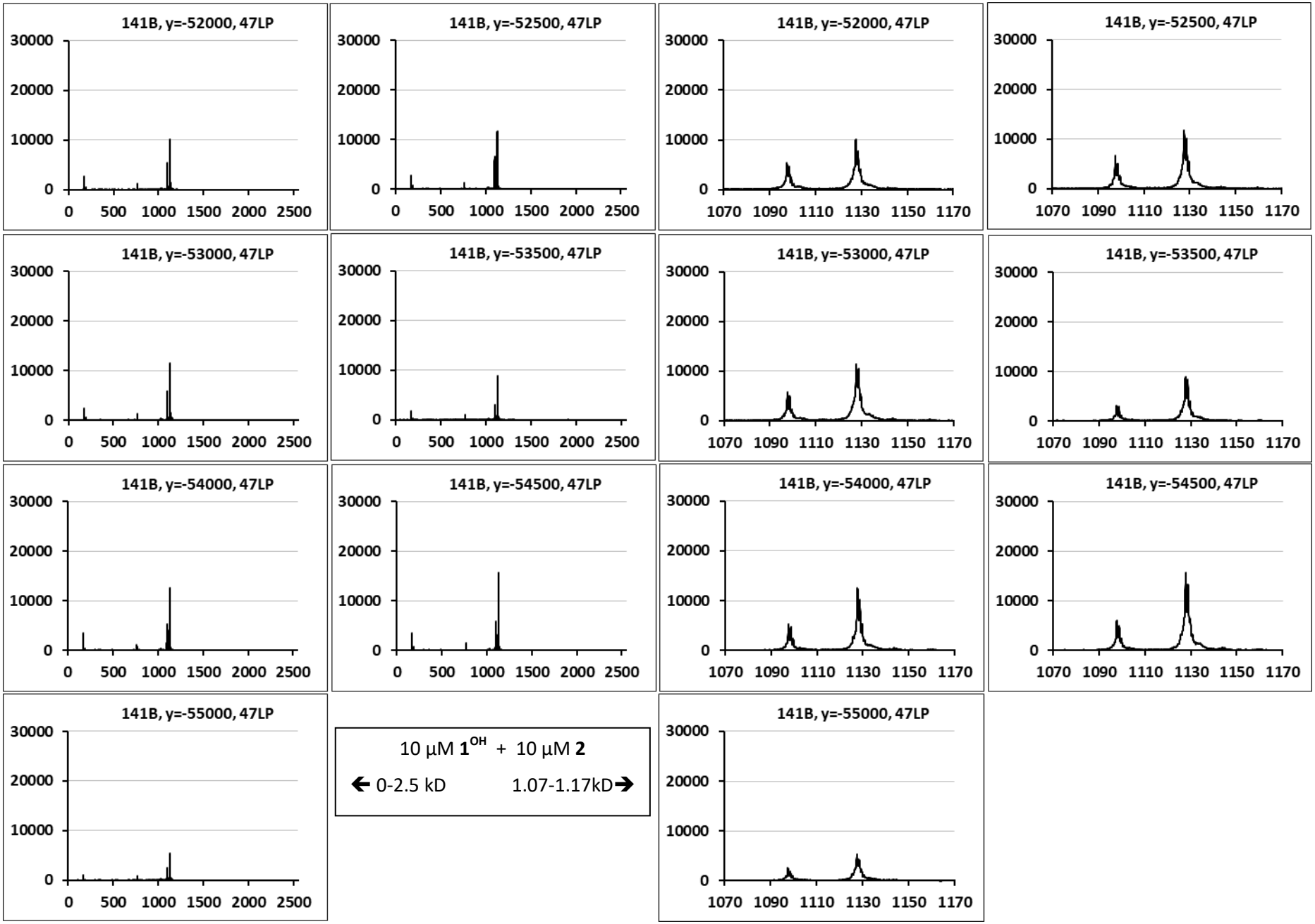
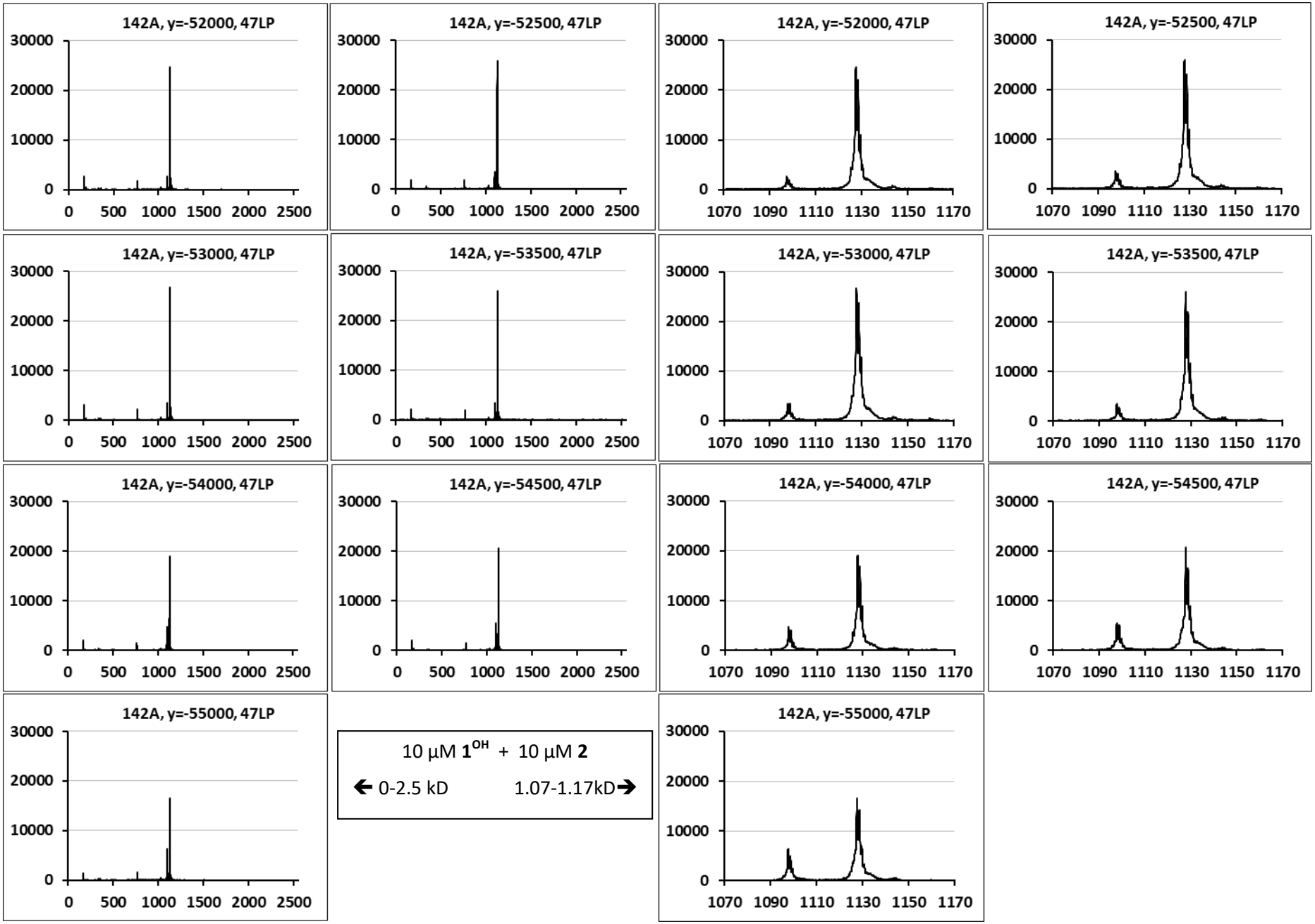
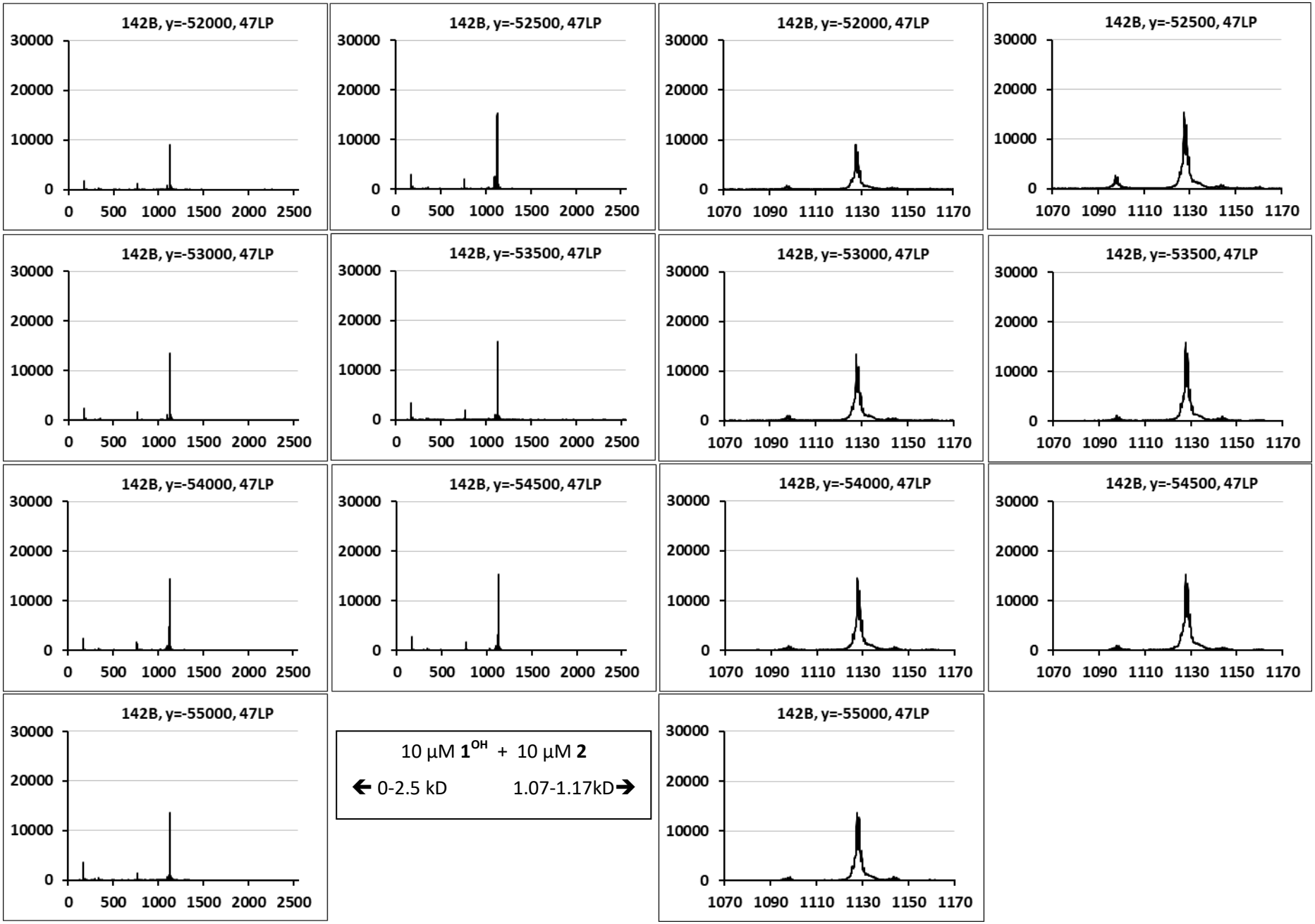
104B, y=-55000, 47LP
4 µM 1OH + 4 µM 2
← 0-2.5 kD
1.07-1.17kD→


141A, y=-52000, 47LP
THAP 2 1OH
141A, y=-52500, 47LP
141A, y=-52000, 47LP
2 1OH
141A, y=-52500, 47LP
141A, y=-53000, 47LP
141A, y=-53500, 47LP
141A, y=-53000, 47LP
141A, y=-53500, 47LP
141A, y=-54000, 47LP
141A, y=-54500, 47LP
141A, y=-54000, 47LP
141A, y=-54500, 47LP
141A, y=-55000, 47LP
10 µM 1OH + 10 µM 2
← 0-2.5 kD
1.07-1.17kD→
141A, y=-55000, 47LP


141B, y=-52000, 47LP
141B, y=-52500, 47LP
141B, y=-52000, 47LP
141B, y=-52500, 47LP
141B, y=-53000, 47LP
141B, y=-53500, 47LP
141B, y=-53000, 47LP
141B, y=-53500, 47LP
141B, y=-54000, 47LP
141B, y=-54500, 47LP
141B, y=-54000, 47LP
141B, y=-54500, 47LP
141B, y=-55000, 47LP
141B, y=-55000, 47LP
30000
20000
10000
0
0 500 1000 1500 2000 2500
1070 1090 1110 1130 1150 1170
10 µM 1OH + 10 µM 2
← 0-2.5 kD
1.07-1.17kD→


142A, y=-52000, 47LP
142A, y=-52500, 47LP
142A, y=-52000, 47LP
142A, y=-52500, 47LP
142A, y=-53000, 47LP
142A, y=-53500, 47LP
142A, y=-53000, 47LP
142A, y=-53500, 47LP
142A, y=-54000, 47LP
142A, y=-54500, 47LP
142A, y=-54000, 47LP
142A, y=-54500, 47LP
142A, y=-55000, 47LP
10 µM 1OH + 10 µM 2
← 0-2.5 kD
1.07-1.17kD→
142A, y=-55000, 47LP


142B, y=-52000, 47LP
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142B, y=-52000, 47LP
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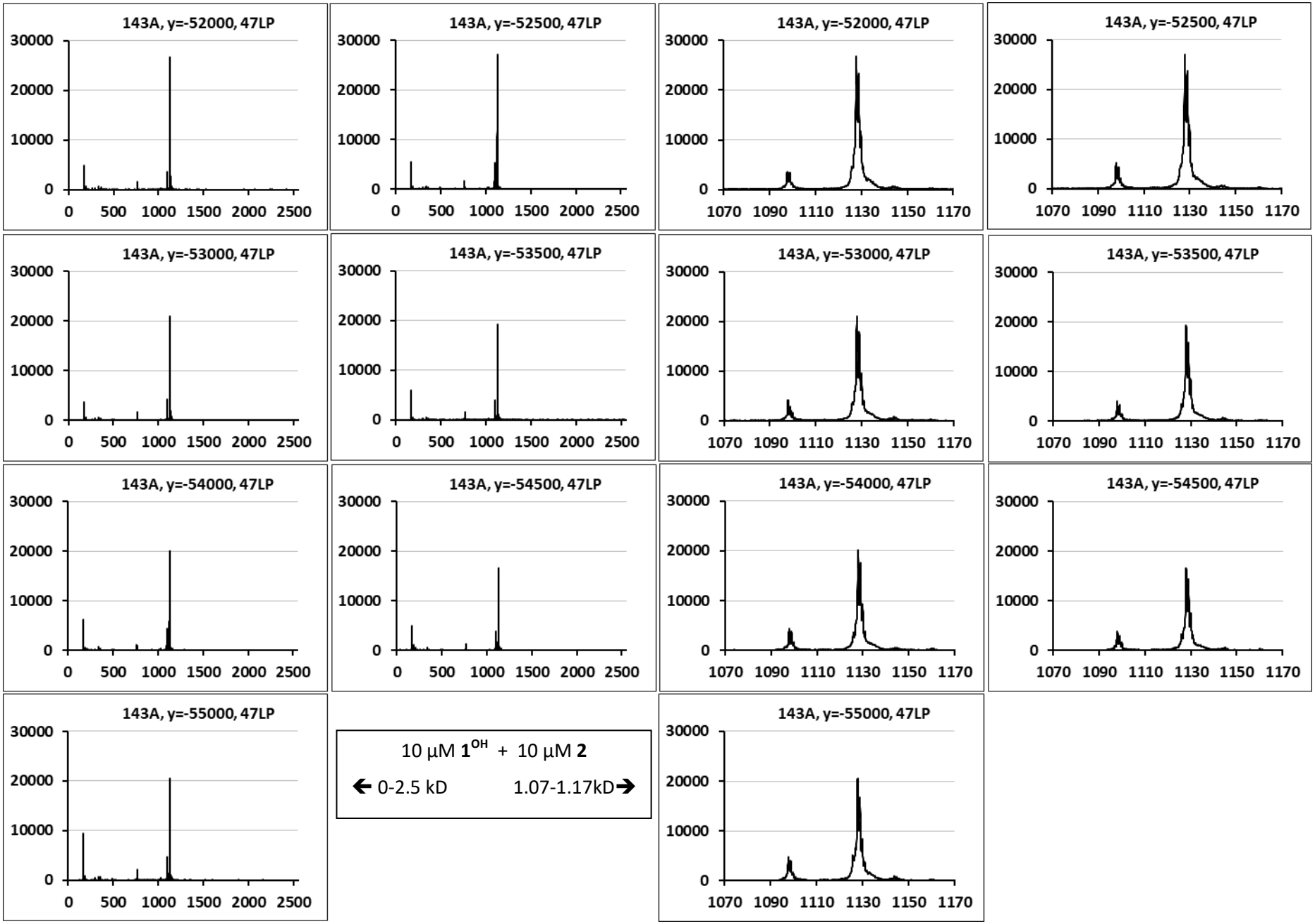
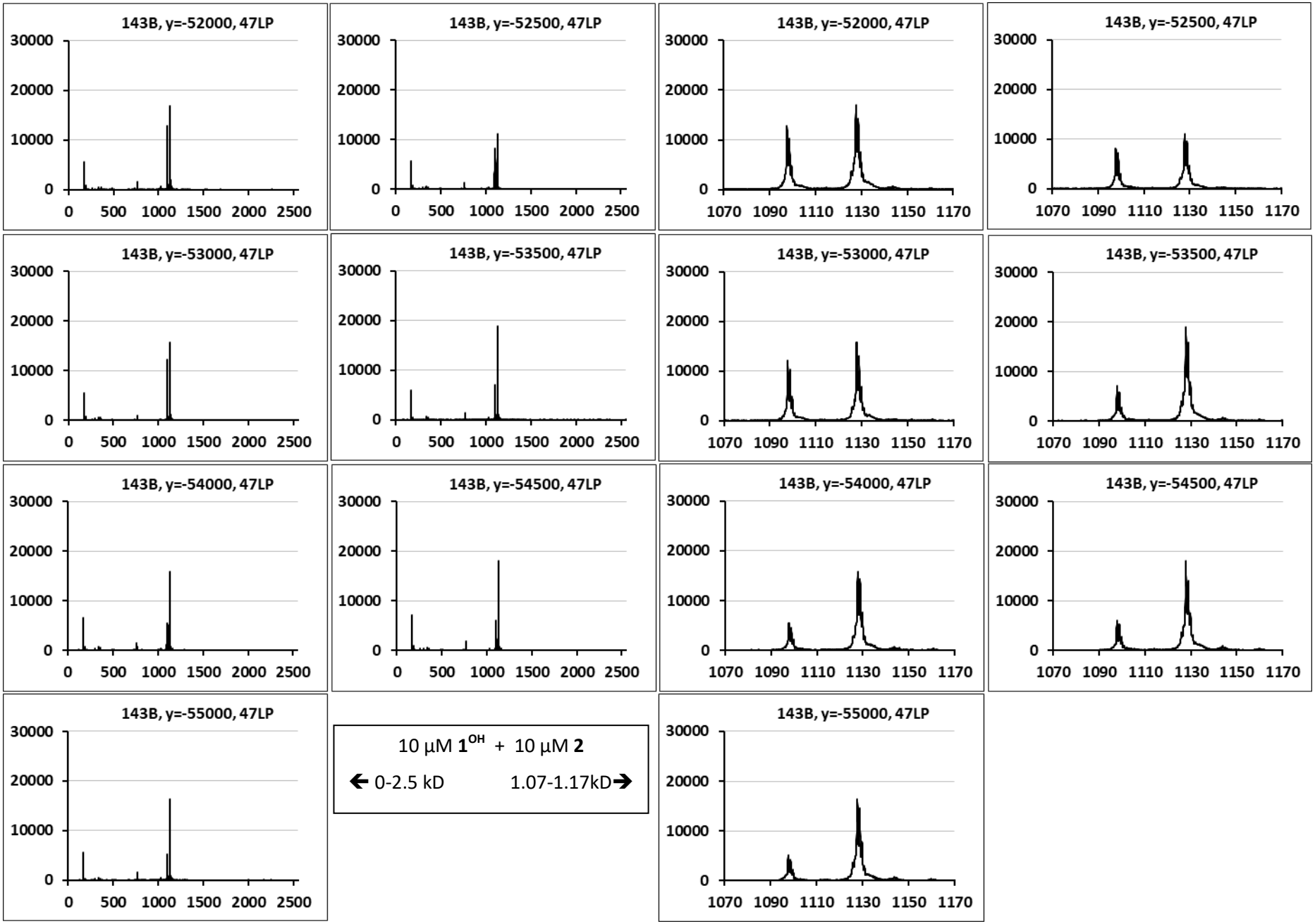
142B, y=-53000, 47LP
142B, y=-53500, 47LP
142B, y=-53000, 47LP
142B, y=-53500, 47LP
142B, y=-54000, 47LP
142B, y=-54500, 47LP
142B, y=-54000, 47LP
142B, y=-54500, 47LP
142B, y=-55000, 47LP
142B, y=-55000, 47LP
30000
20000
10000
0
0 500 1000 1500 2000 2500
1070 1090 1110 1130 1150 1170
10 µM 1OH + 10 µM 2
← 0-2.5 kD
1.07-1.17kD→


143A, y=-52000, 47LP
143A, y=-52500, 47LP
143A, y=-52000, 47LP
143A, y=-52500, 47LP
143A, y=-53000, 47LP
143A, y=-53500, 47LP
143A, y=-53000, 47LP
143A, y=-53500, 47LP
143A, y=-54000, 47LP
143A, y=-54500, 47LP
143A, y=-54000, 47LP
143A, y=-54500, 47LP
143A, y=-55000, 47LP
10 µM 1OH + 10 µM 2
← 0-2.5 kD
1.07-1.17kD→
143A, y=-55000, 47LP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143B, y=-52500, 47LP
143B, y=-52000, 47LP
143B, y=-52500, 47LP
143B, y=-53000, 47LP
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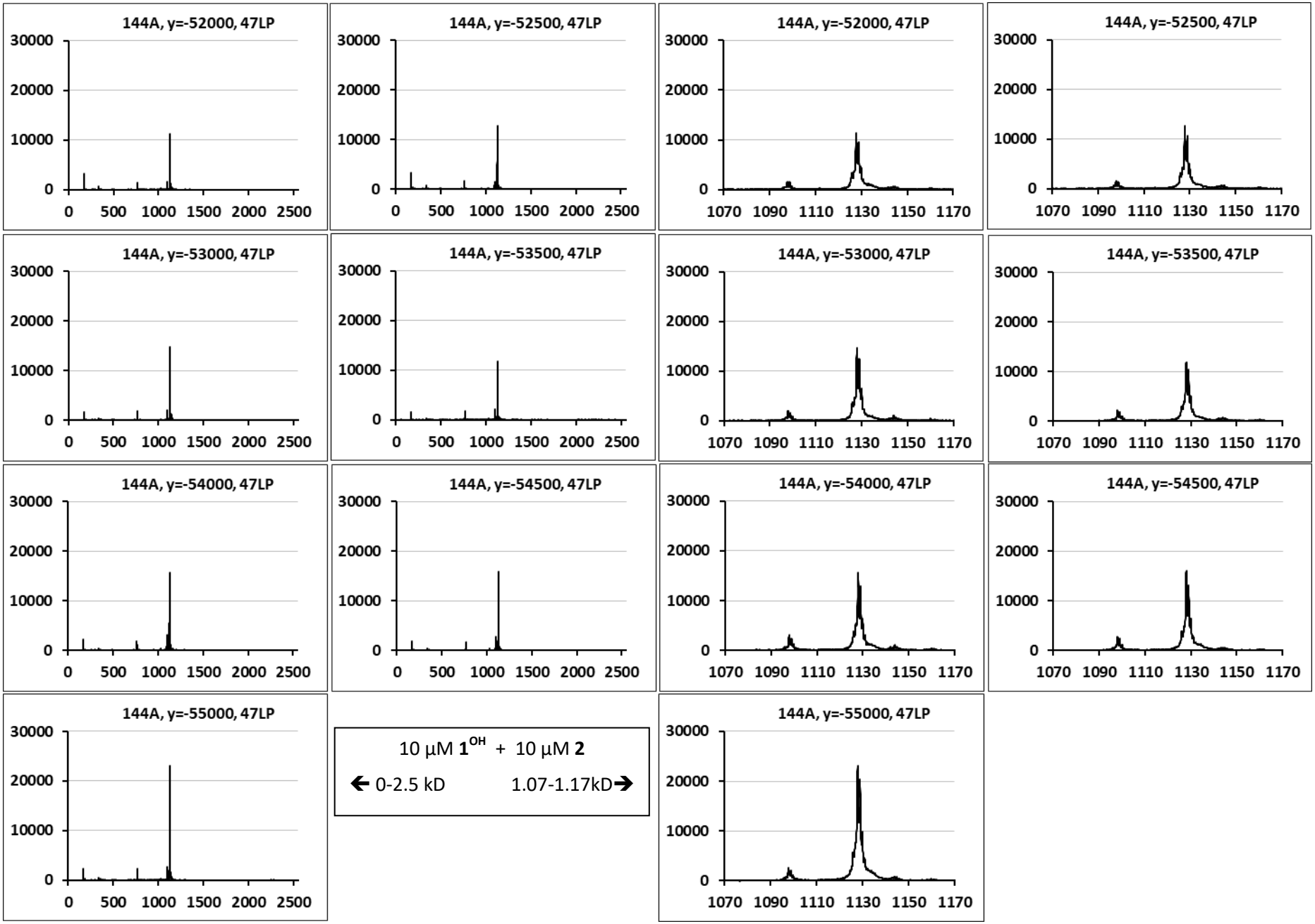
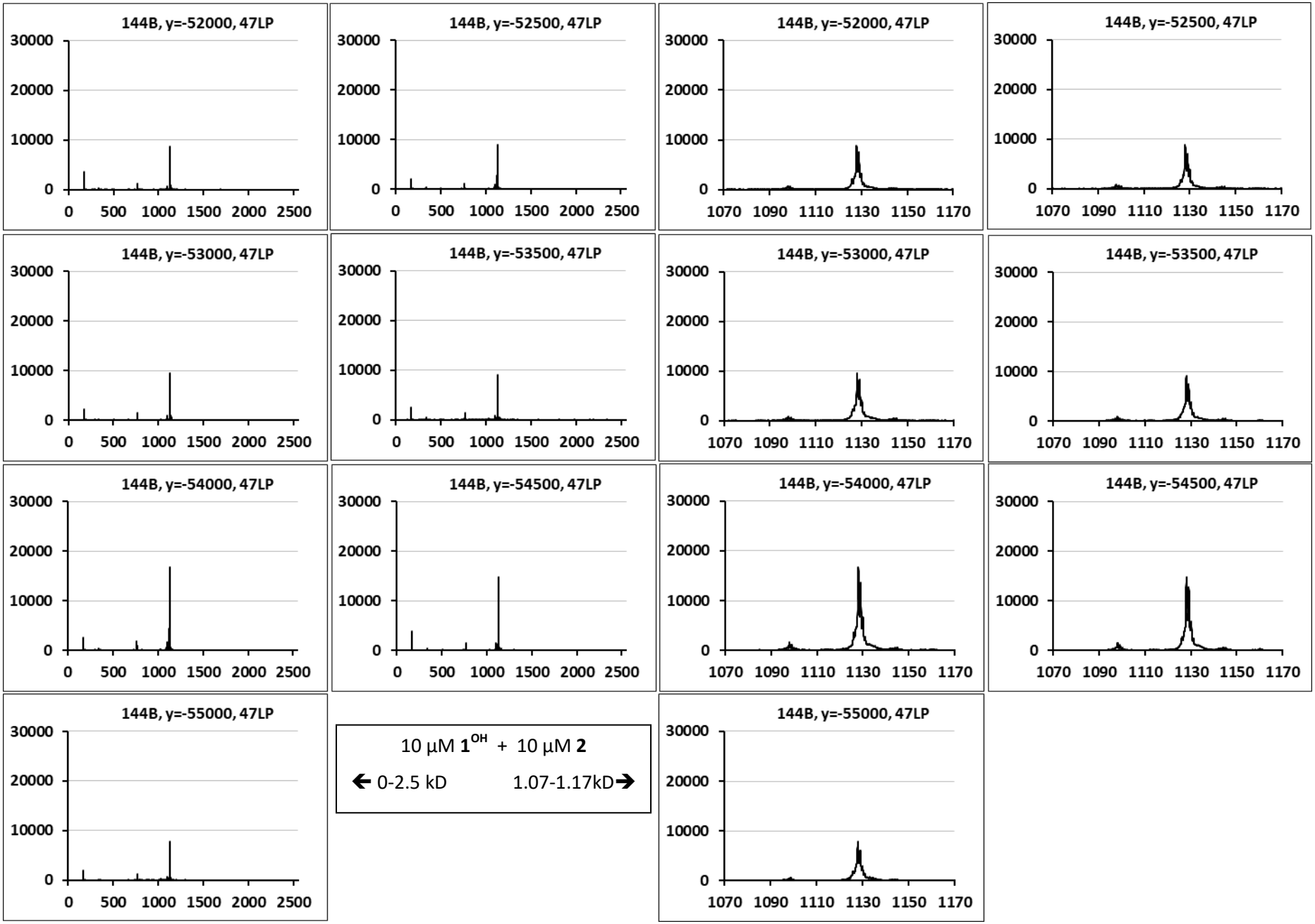
143B, y=-53000, 47LP
143B, y=-53500, 47LP
143B, y=-54000, 47LP
143B, y=-54500, 47LP
143B, y=-54000, 47LP
143B, y=-54500, 47LP
143B, y=-55000, 47LP
143B, y=-55000, 47LP
30000
20000
10000
0
0 500 1000 1500 2000 2500
1070 1090 1110 1130 1150 1170
10 µM 1OH + 10 µM 2
← 0-2.5 kD
1.07-1.17kD →

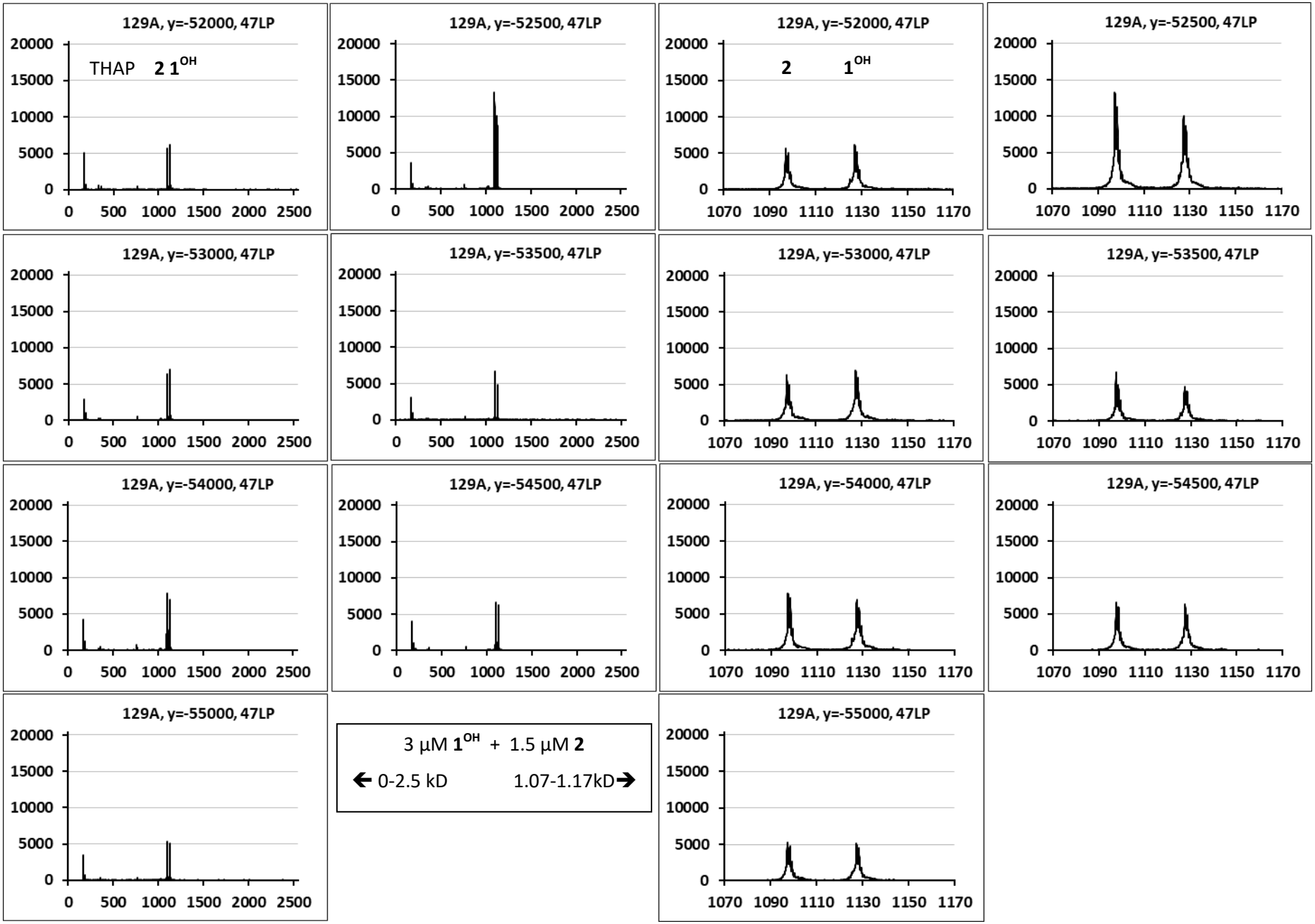

144A, y=-52000, 47LP
144A, y=-52500, 47LP
144A, y=-52000, 47LP
144A, y=-52500, 47LP
144A, y=-53000, 47LP
144A, y=-53500, 47LP
144A, y=-53000, 47LP
144A, y=-53500, 47LP
144A, y=-54000, 47LP
144A, y=-54500, 47LP
144A, y=-54000, 47LP
144A, y=-54500, 47LP
144A, y=-55000, 47LP
144A, y=-55000, 47LP
30000
20000
10000
0
0 500 1000 1500 2000 2500
1070 1090 1110 1130 1150 1170
10 µM 1OH + 10 µM 2
← 0-2.5 kD
1.07-1.17kD →

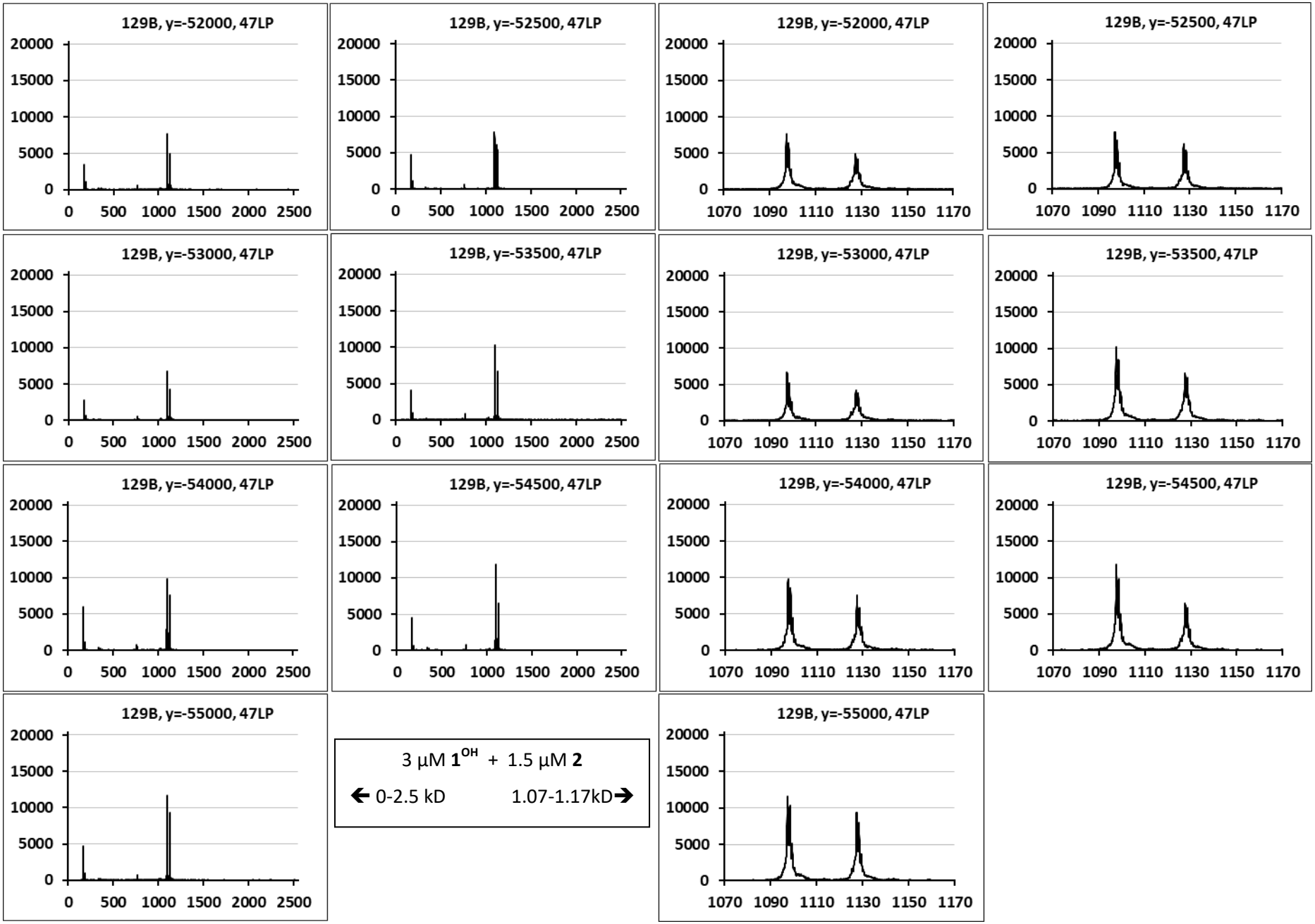

144B, y=-52000, 47LP
144B, y=-52500, 47LP
144B, y=-52000, 47LP
144B, y=-52500, 47LP
144B, y=-53000, 47LP
144B, y=-53500, 47LP
144B, y=-53000, 47LP
144B, y=-53500, 47LP
144B, y=-54000, 47LP
144B, y=-54500, 47LP
144B, y=-54000, 47LP
144B, y=-54500, 47LP
144B, y=-55000, 47LP
144B, y=-55000, 47LP
30000
20000
10000
0
0 500 1000 1500 2000 2500
1070 1090 1110 1130 1150 1170
10 µM 1OH + 10 µM 2
← 0-2.5 kD
1.07-1.17kD →

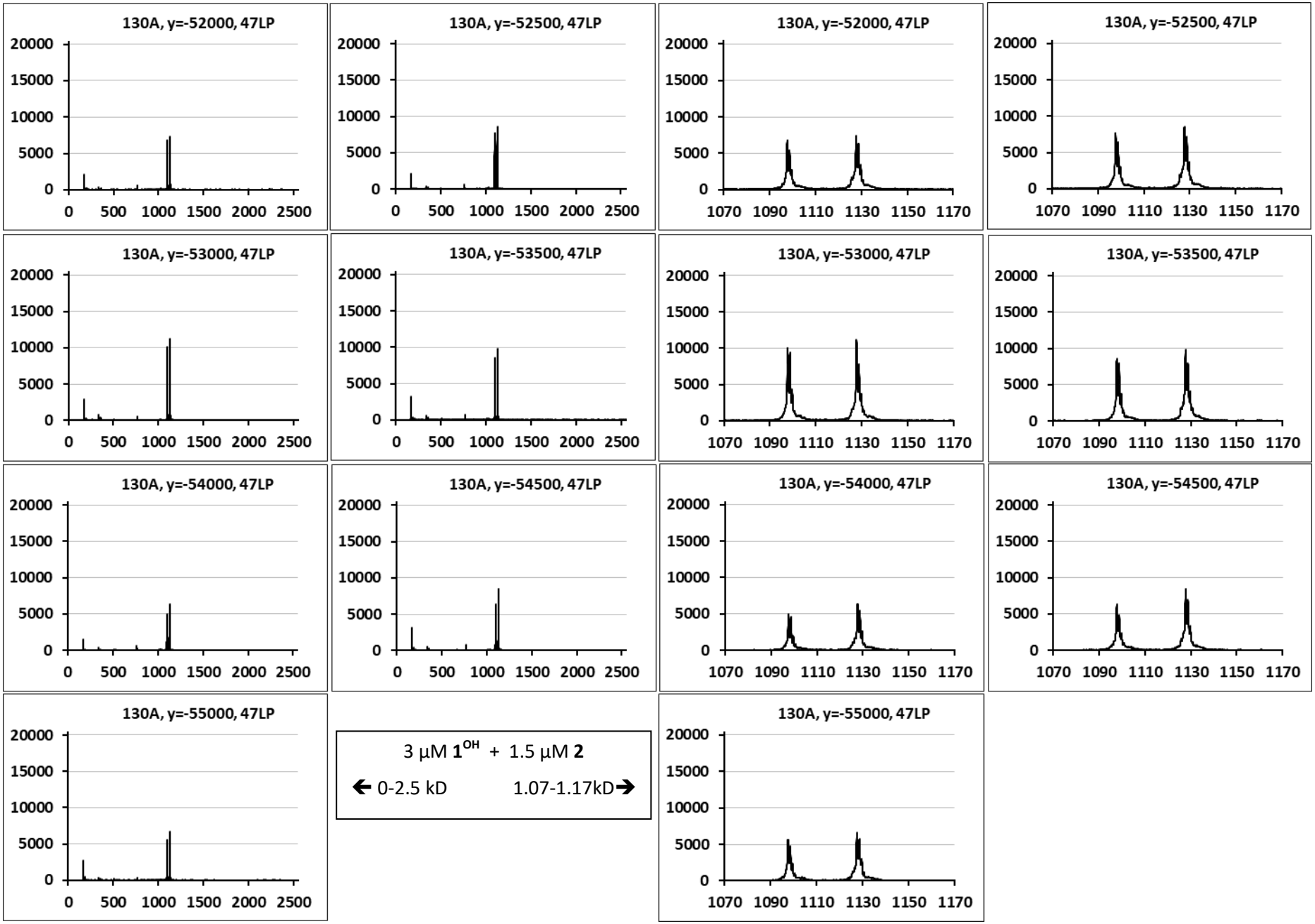
Summary of **1^{OH} / 2** phenyl octane solution with 2 : 1 concentration ratio: TOF-MS ML **1^{OH}** / **2** intensity and intensity ratio statistics from **1^{OH} / 2** phenyl octane solution with 2 : 1 concentration ratio (THAP thin film applied, LP = 47, TOF-MS measured at 14 locations on each of 4 independently prepared samples).

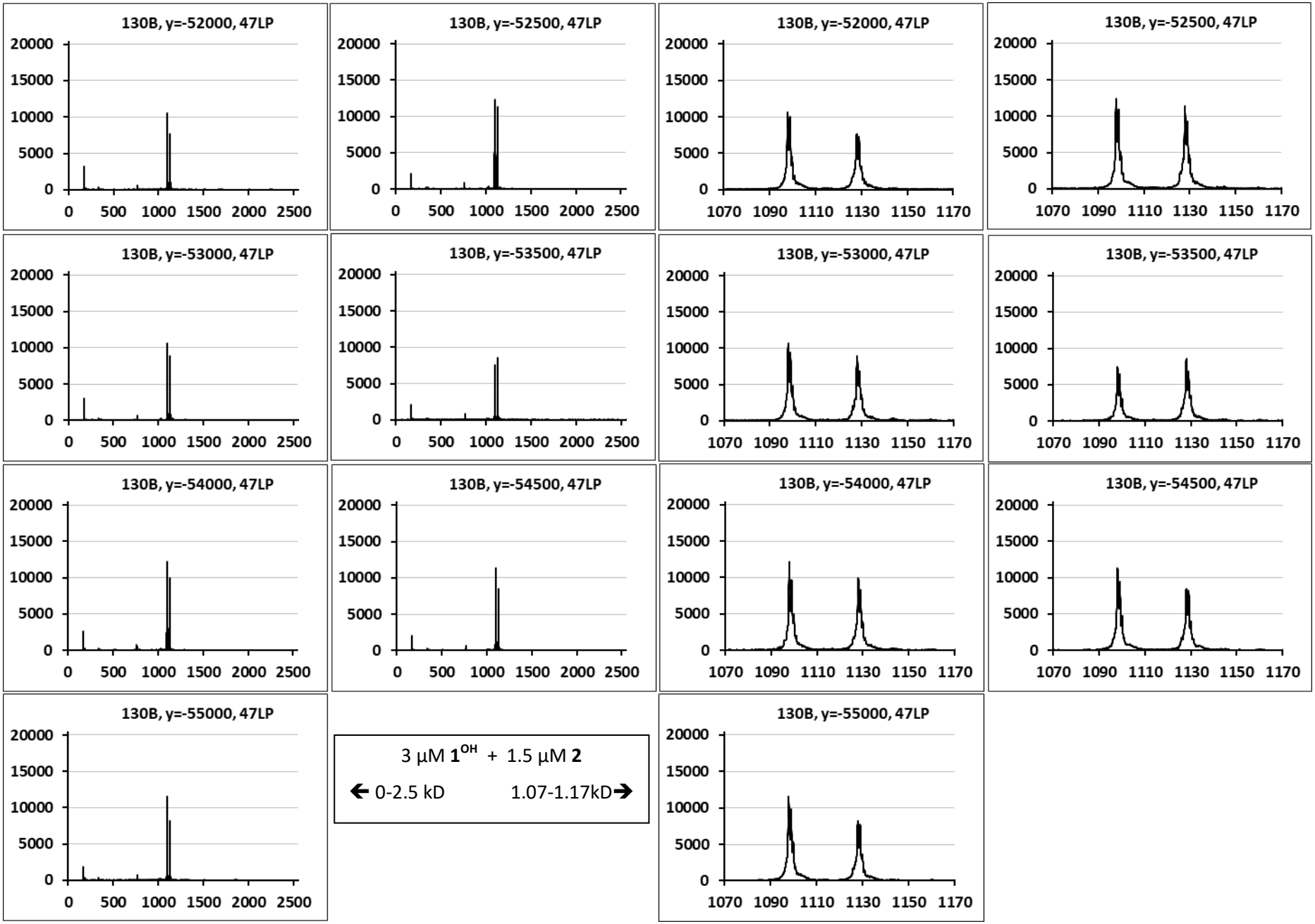
[1^{OH}]	[2]	# Locations	1^{OH} / 2	1^{OH} / 2	1^{OH} / 2	1^{OH} / 2	1^{OH} / 2	1^{OH}	1^{OH}	1^{OH}	1^{OH}	1^{OH}	2	2	2	2	2
µM	µM	(file names)	Max	Min	Avg	σ	σ/Avg	Max	Min	Avg	σ	σ/Avg	Max	Min	Avg	σ	σ/Avg
3	1.5	14 (129A,B)	1.09	0.54	0.81	0.17	21%	10027	4248	6551	1640	25%	13274	5284	8390	2527	30%
3	1.5	14 (130A,B)	1.33	0.70	1.01	0.21	21%	11369	6374	8665	1490	17%	12354	4964	9009	2537	28%
3	1.5	14 (131A,B)	1.19	0.60	0.82	0.20	24%	13370	4507	7992	2873	36%	16948	7177	9694	2808	29%
3	1.5	14 (132A,B)	0.92	0.36	0.60	0.18	31%	6316	1948	4726	1129	24%	12391	5165	8201	2216	27%
3	1.5	All	1.33	0.36	**0.81**	**0.24**	**29%**	13370	1948	**6984**	**2395**	**34%**	16948	4964	**8824**	**2530**	**29%**

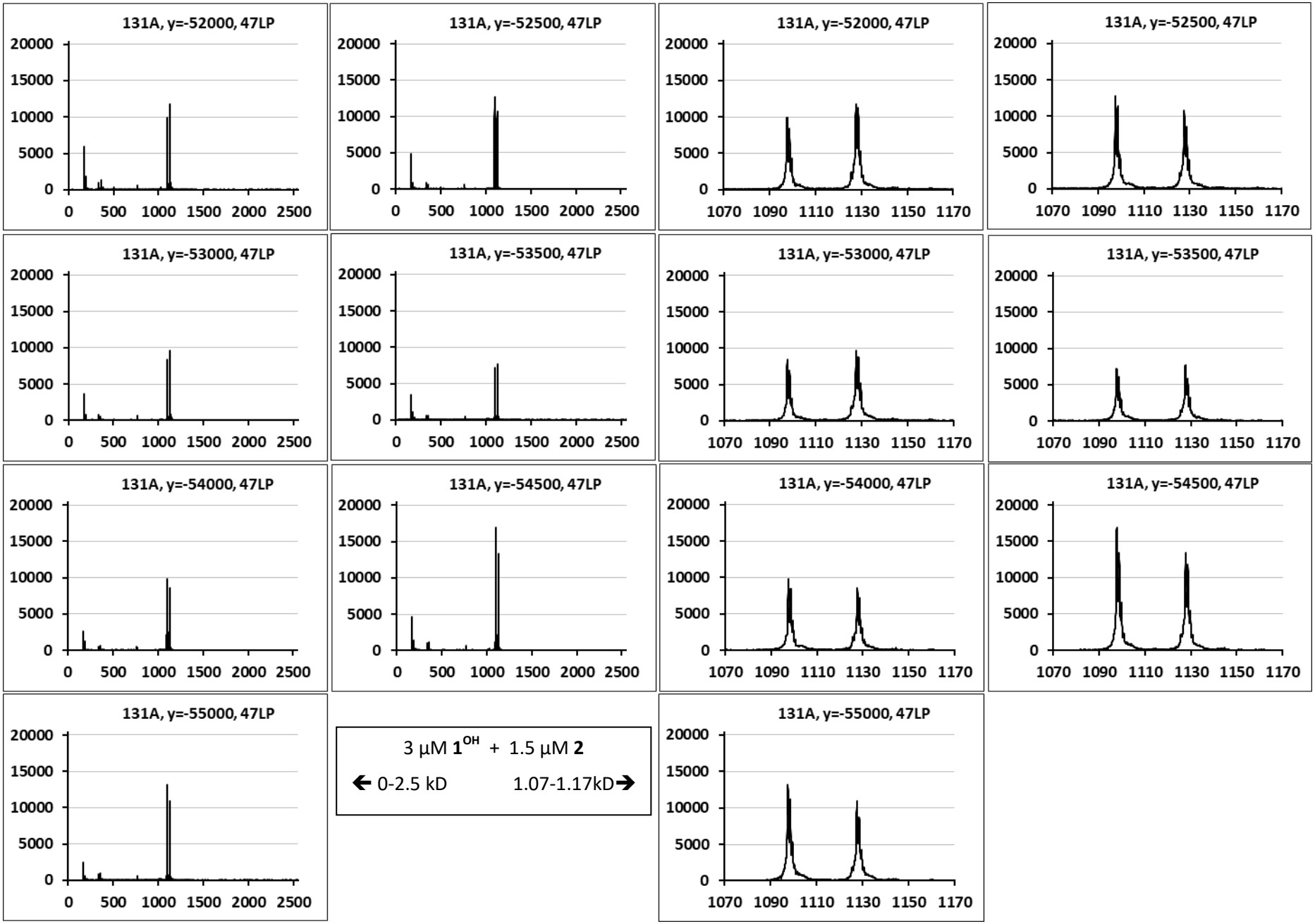
[1^{OH}]	[2]	Locations	1^{OH} / 2	1^{OH} / 2	1^{OH} / 2	1^{OH} / 2	1^{OH} / 2	1^{OH}	1^{OH}	1^{OH}	1^{OH}	1^{OH}	2	2	2	2	2
µM	µM		Max	Min	Avg	σ	σ/Avg	Max	Min	Avg	σ	σ/Avg	Max	Min	Avg	σ	σ/Avg
4	2	14 (125A,B)	0.83	0.61	0.73	0.08	10%	10589	6170	8593	1386	16%	15935	8402	11821	2231	19%
4	2	14 (126A,B)	1.23	0.58	0.85	0.23	28%	11635	3498	7495	2969	40%	12227	5121	8646	2134	25%
4	2	14 (127A,B)	2.22	0.99	1.44	0.43	30%	14120	6632	10912	2937	27%	11177	5117	7885	2276	29%
4	2	14 (128A,B)	1.39	0.73	1.00	0.19	19%	23382	8270	14574	4452	31%	20487	5954	14657	3561	24%
4	2	All	2.22	0.58	**1.01**	**0.37**	**37%**	23382	3498	**10393**	**4092**	**39%**	20487	5117	**10752**	**3724**	**35%**

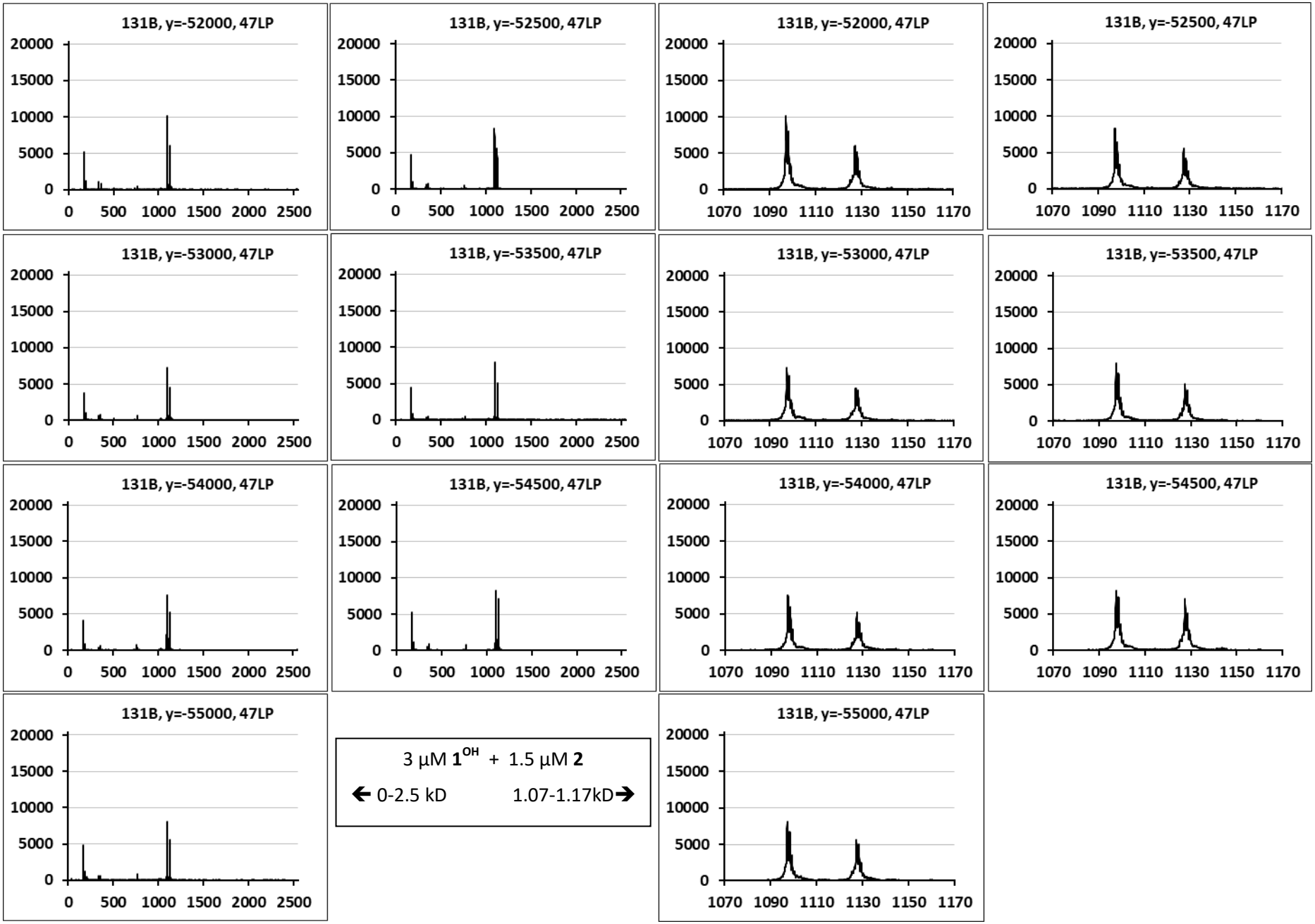

129A, y=-52000, 47LP
THAP 2 1OH
129A, y=-52500, 47LP
129A, y=-52000, 47LP
2 1OH
129A, y=-52500, 47LP
129A, y=-53000, 47LP
129A, y=-53500, 47LP
129A, y=-53000, 47LP
129A, y=-53500, 47LP
129A, y=-54000, 47LP
129A, y=-54500, 47LP
129A, y=-54000, 47LP
129A, y=-54500, 47LP
129A, y=-55000, 47LP
3 µM 1OH + 1.5 µM 2
← 0-2.5 kD
1.07-1.17kD →
129A, y=-55000, 47LP

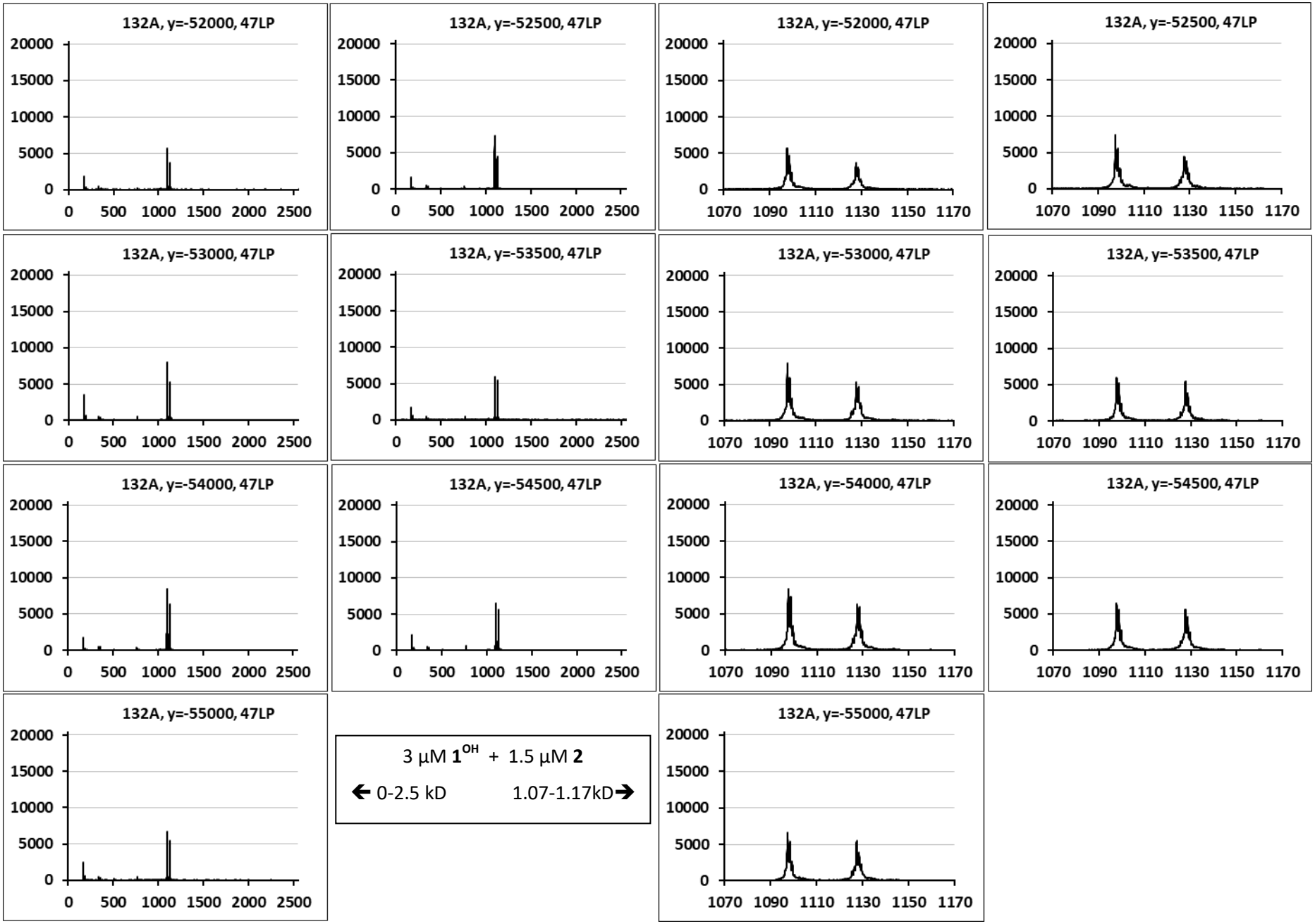

129B, y=-52000, 47LP
129B, y=-52500, 47LP
129B, y=-52000, 47LP
129B, y=-52500, 47LP
129B, y=-53000, 47LP
129B, y=-53500, 47LP
129B, y=-53000, 47LP
129B, y=-53500, 47LP
129B, y=-54000, 47LP
129B, y=-54500, 47LP
129B, y=-54000, 47LP
129B, y=-54500, 47LP
129B, y=-55000, 47LP
129B, y=-55000, 47LP
20000
15000
10000
5000
0
0 500 1000 1500 2000 2500
1070 1090 1110 1130 1150 1170
3 µM 1OH + 1.5 µM 2
← 0-2.5 kD
1.07-1.17kD →

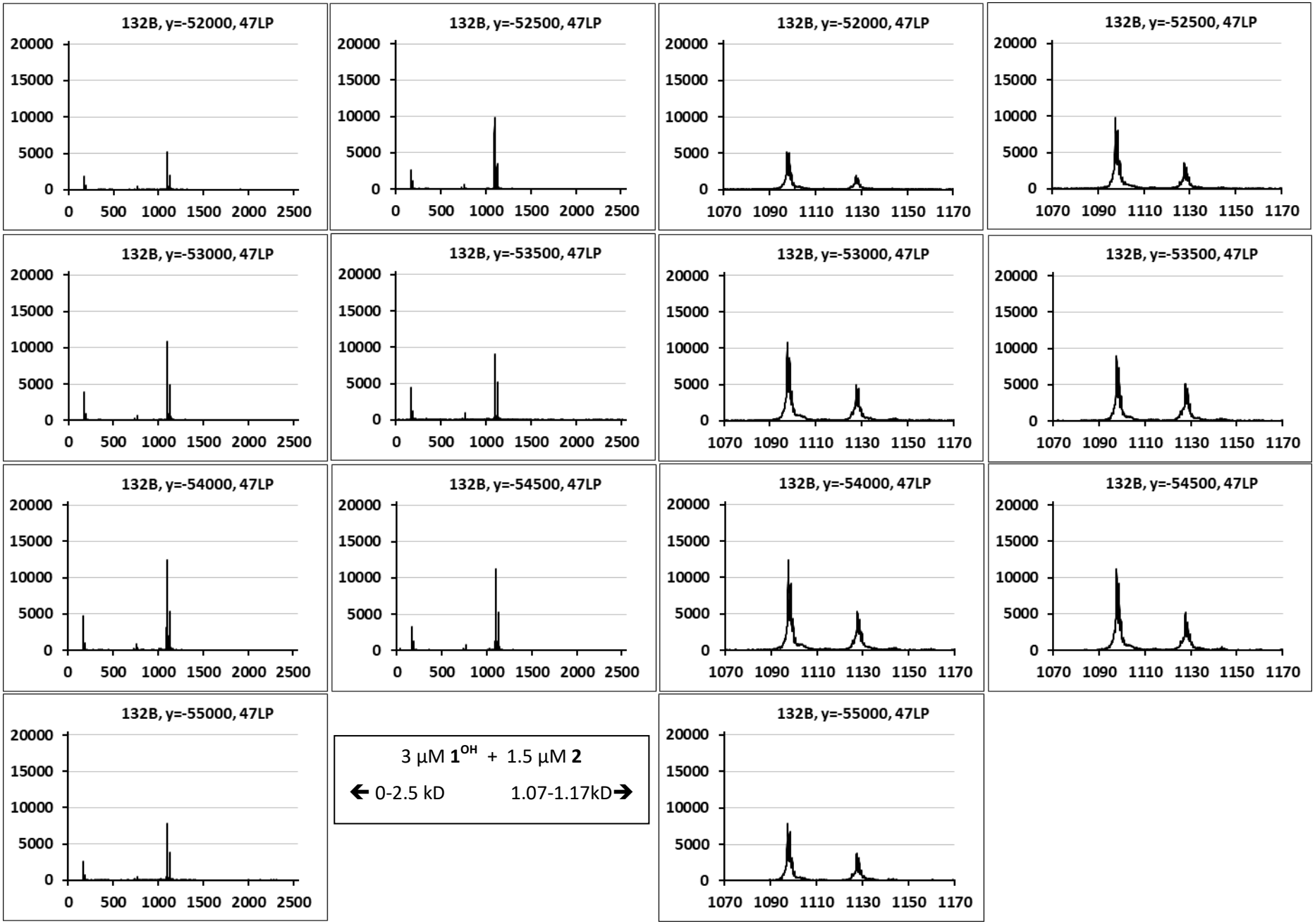

130A, y=-52000, 47LP
130A, y=-52500, 47LP
130A, y=-52000, 47LP
130A, y=-52500, 47LP
130A, y=-53000, 47LP
130A, y=-53500, 47LP
130A, y=-53000, 47LP
130A, y=-53500, 47LP
130A, y=-54000, 47LP
130A, y=-54500, 47LP
130A, y=-54000, 47LP
130A, y=-54500, 47LP
130A, y=-55000, 47LP
3 µM 1OH + 1.5 µM 2
← 0-2.5 kD
1.07-1.17kD→
130A, y=-55000, 47LP

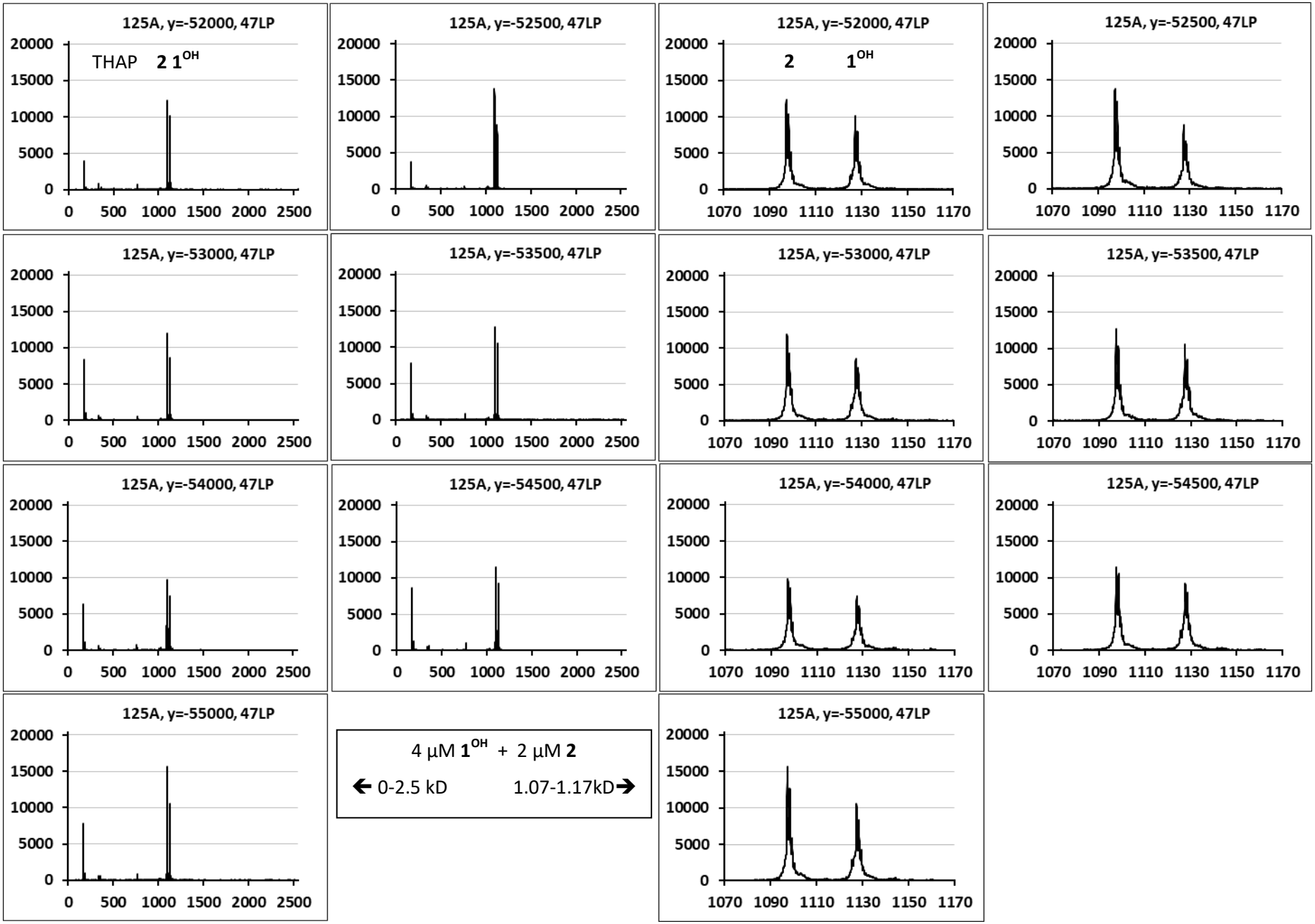

130B, y=-52000, 47LP
130B, y=-52500, 47LP
130B, y=-52000, 47LP
130B, y=-52500, 47LP
130B, y=-53000, 47LP
130B, y=-53500, 47LP
130B, y=-53000, 47LP
130B, y=-53500, 47LP
130B, y=-54000, 47LP
130B, y=-54500, 47LP
130B, y=-54000, 47LP
130B, y=-54500, 47LP
130B, y=-55000, 47LP
3 µM 1OH + 1.5 µM 2
← 0-2.5 kD
1.07-1.17kD→
130B, y=-55000, 47LP

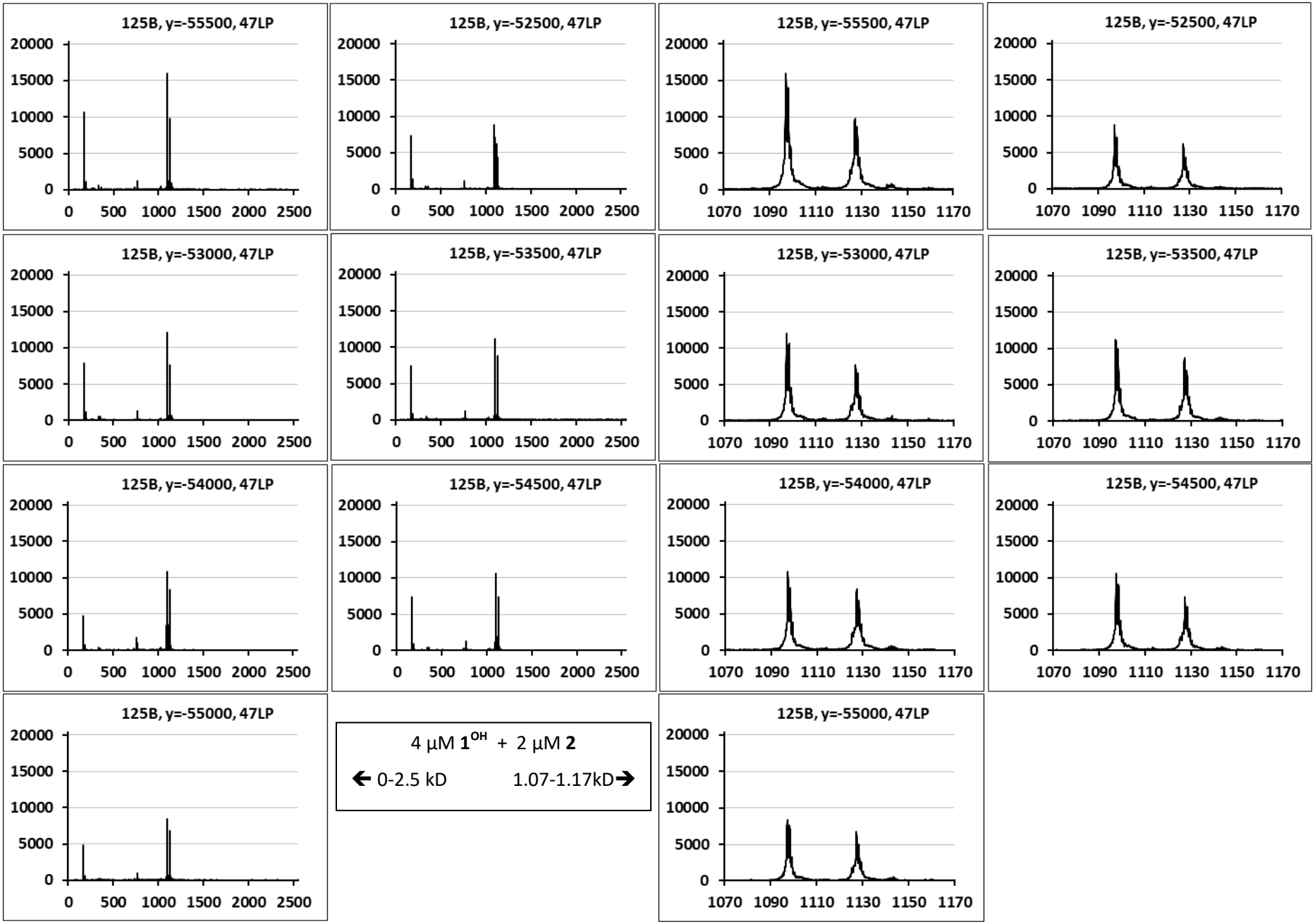

131A, y=-52000, 47LP
131A, y=-52500, 47LP
131A, y=-52000, 47LP
131A, y=-52500, 47LP
131A, y=-53000, 47LP
131A, y=-53500, 47LP
131A, y=-53000, 47LP
131A, y=-53500, 47LP
131A, y=-54000, 47LP
131A, y=-54500, 47LP
131A, y=-54000, 47LP
131A, y=-54500, 47LP
131A, y=-55000, 47LP
3 µM 1OH + 1.5 µM 2
← 0-2.5 kD
1.07-1.17kD→
131A, y=-55000, 47LP


131B, y=-52000, 47LP
131B, y=-52500, 47LP
131B, y=-52000, 47LP
131B, y=-52500, 47LP
131B, y=-53000, 47LP
131B, y=-53500, 47LP
131B, y=-53000, 47LP
131B, y=-53500, 47LP
131B, y=-54000, 47LP
131B, y=-54500, 47LP
131B, y=-54000, 47LP
131B, y=-54500, 47LP
131B, y=-55000, 47LP
131B, y=-55000, 47LP
20000
15000
10000
5000
0
0 500 1000 1500 2000 2500
1070 1090 1110 1130 1150 1170
3 µM 1OH + 1.5 µM 2
← 0-2.5 kD
1.07-1.17kD →


132A, y=-52000, 47LP
132A, y=-52500, 47LP
132A, y=-52000, 47LP
132A, y=-52500, 47LP
132A, y=-53000, 47LP
132A, y=-53500, 47LP
132A, y=-53000, 47LP
132A, y=-53500, 47LP
132A, y=-54000, 47LP
132A, y=-54500, 47LP
132A, y=-54000, 47LP
132A, y=-54500, 47LP
132A, y=-55000, 47LP
3 µM 1OH + 1.5 µM 2
← 0-2.5 kD
1.07-1.17kD→
132A, y=-55000, 47LP


132B, y=-52000, 47LP
132B, y=-52500, 47LP
132B, y=-52000, 47LP
132B, y=-52500, 47LP
132B, y=-53000, 47LP
132B, y=-53500, 47LP
132B, y=-53000, 47LP
132B, y=-53500, 47LP
132B, y=-54000, 47LP
132B, y=-54500, 47LP
132B, y=-54000, 47LP
132B, y=-54500, 47LP
132B, y=-55000, 47LP
3 µM 1OH + 1.5 µM 2
← 0-2.5 kD
1.07-1.17kD→
132B, y=-55000, 47LP


125A, y=-52000, 47LP
THAP 2 1OH
125A, y=-52500, 47LP
125A, y=-52000, 47LP
2 1OH
125A, y=-52500, 47LP
125A, y=-53000, 47LP
125A, y=-53500, 47LP
125A, y=-53000, 47LP
125A, y=-53500, 47LP
125A, y=-54000, 47LP
125A, y=-54500, 47LP
125A, y=-54000, 47LP
125A, y=-54500, 47LP
125A, y=-55000, 47LP
4 µM 1OH + 2 µM 2
← 0-2.5 kD
1.07-1.17kD→
125A, y=-55000, 47LP


125B, y=-55500, 47LP
125B, y=-52500, 47LP
125B, y=-55500, 47LP
125B, y=-52500, 47LP
125B, y=-53000, 47LP
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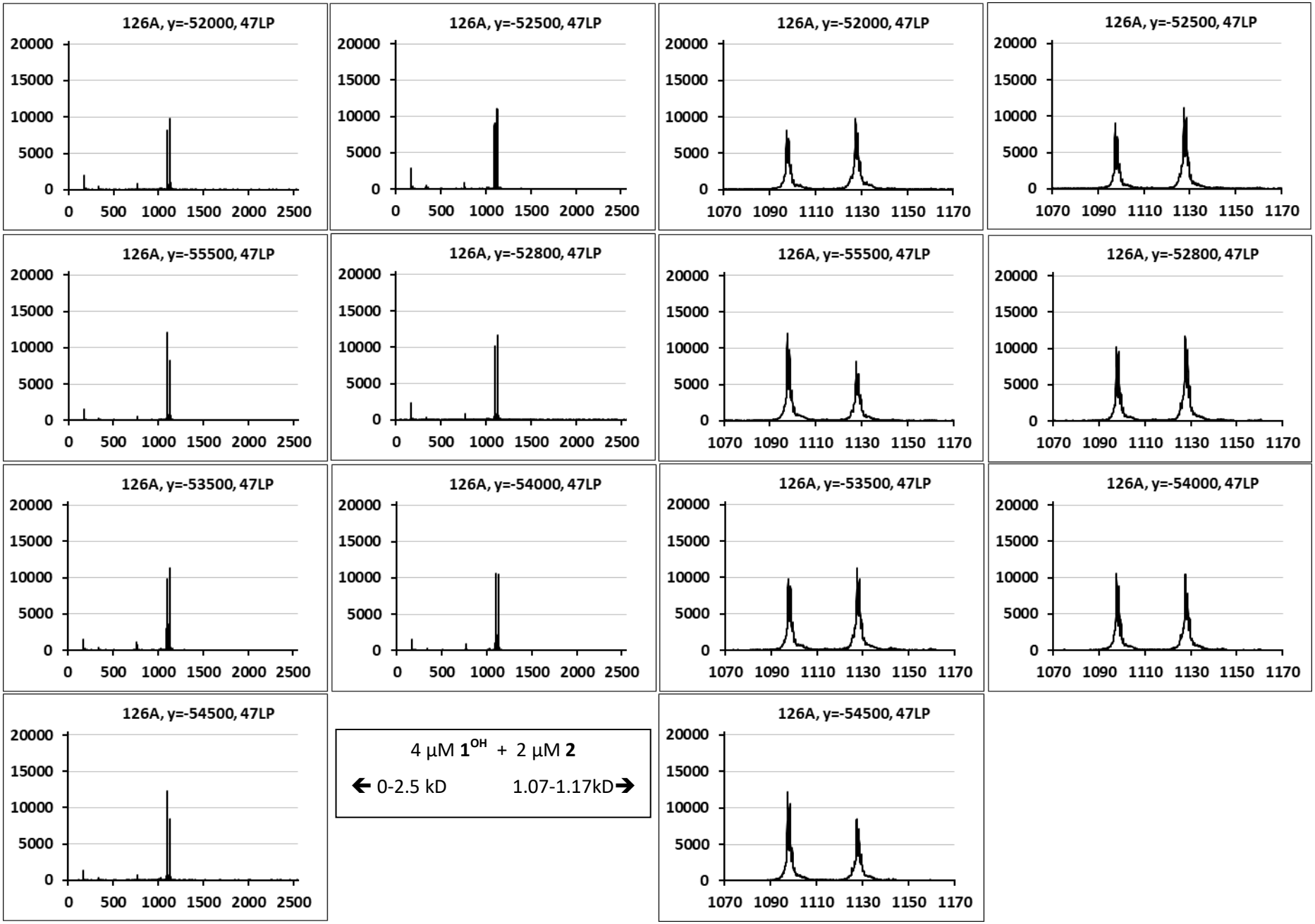
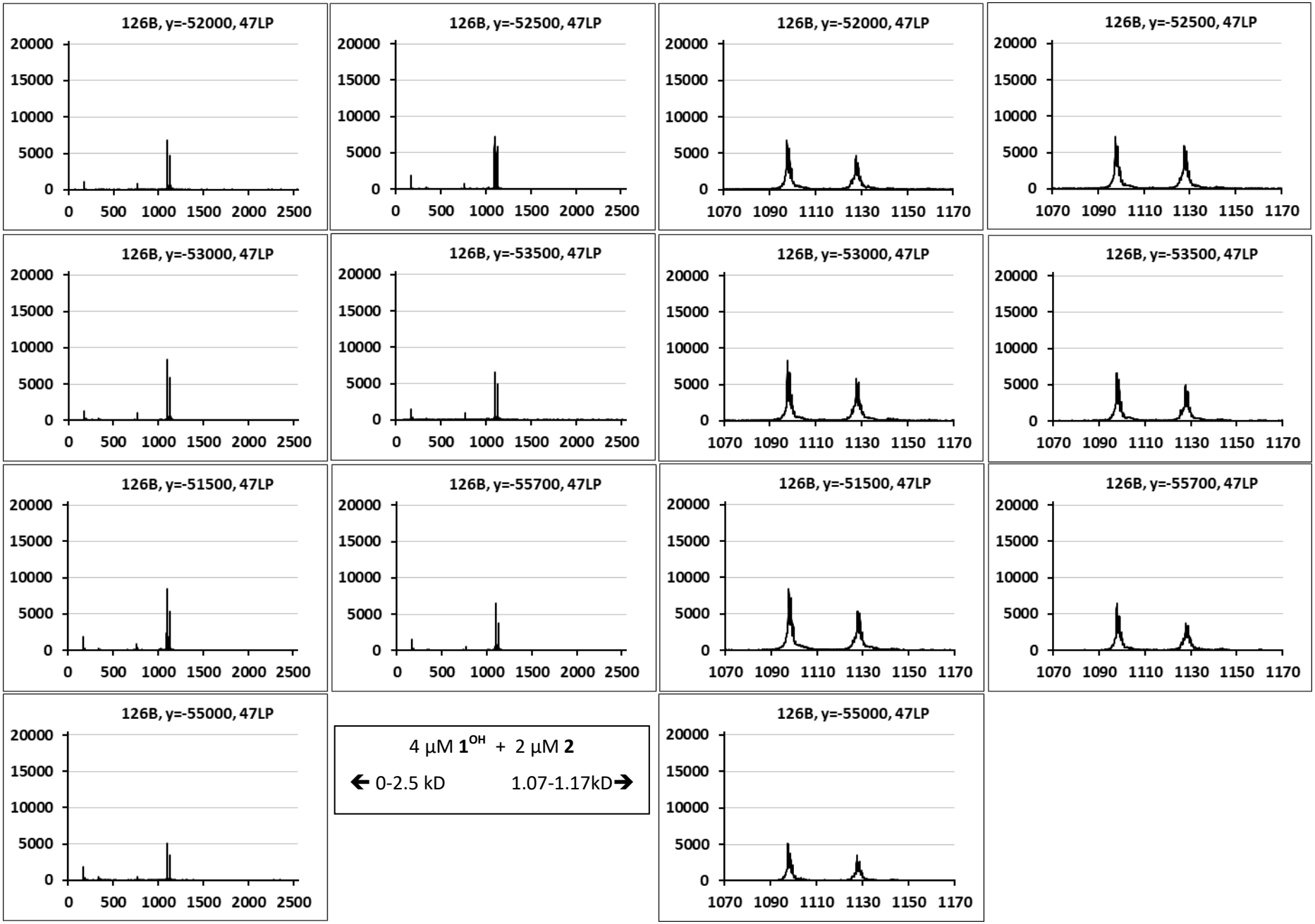
125B, y=-53000, 47LP
125B, y=-53500, 47LP
125B, y=-54000, 47LP
125B, y=-54500, 47LP
125B, y=-54000, 47LP
125B, y=-54500, 47LP
125B, y=-55000, 47LP
4 µM 1OH + 2 µM 2
← 0-2.5 kD
1.07-1.17kD →
125B, y=-55000, 47LP


126A, y=-52000, 47LP
126A, y=-52500, 47LP
126A, y=-52000, 47LP
126A, y=-52500, 47LP
126A, y=-55500, 47LP
126A, y=-52800, 47LP
126A, y=-55500, 47LP
126A, y=-52800, 47LP
126A, y=-53500, 47LP
126A, y=-54000, 47LP
126A, y=-53500, 47LP
126A, y=-54000, 47LP
126A, y=-54500, 47LP
126A, y=-54500, 47LP
20000
15000
10000
5000
0
0 500 1000 1500 2000 2500
1070 1090 1110 1130 1150 1170
4 µM 1OH + 2 µM 2
← 0-2.5 kD
1.07-1.17kD →


126B, y=-52000, 47LP
126B, y=-52500, 47LP
126B, y=-52000, 47LP
126B, y=-52500, 47LP
126B, y=-53000, 47LP
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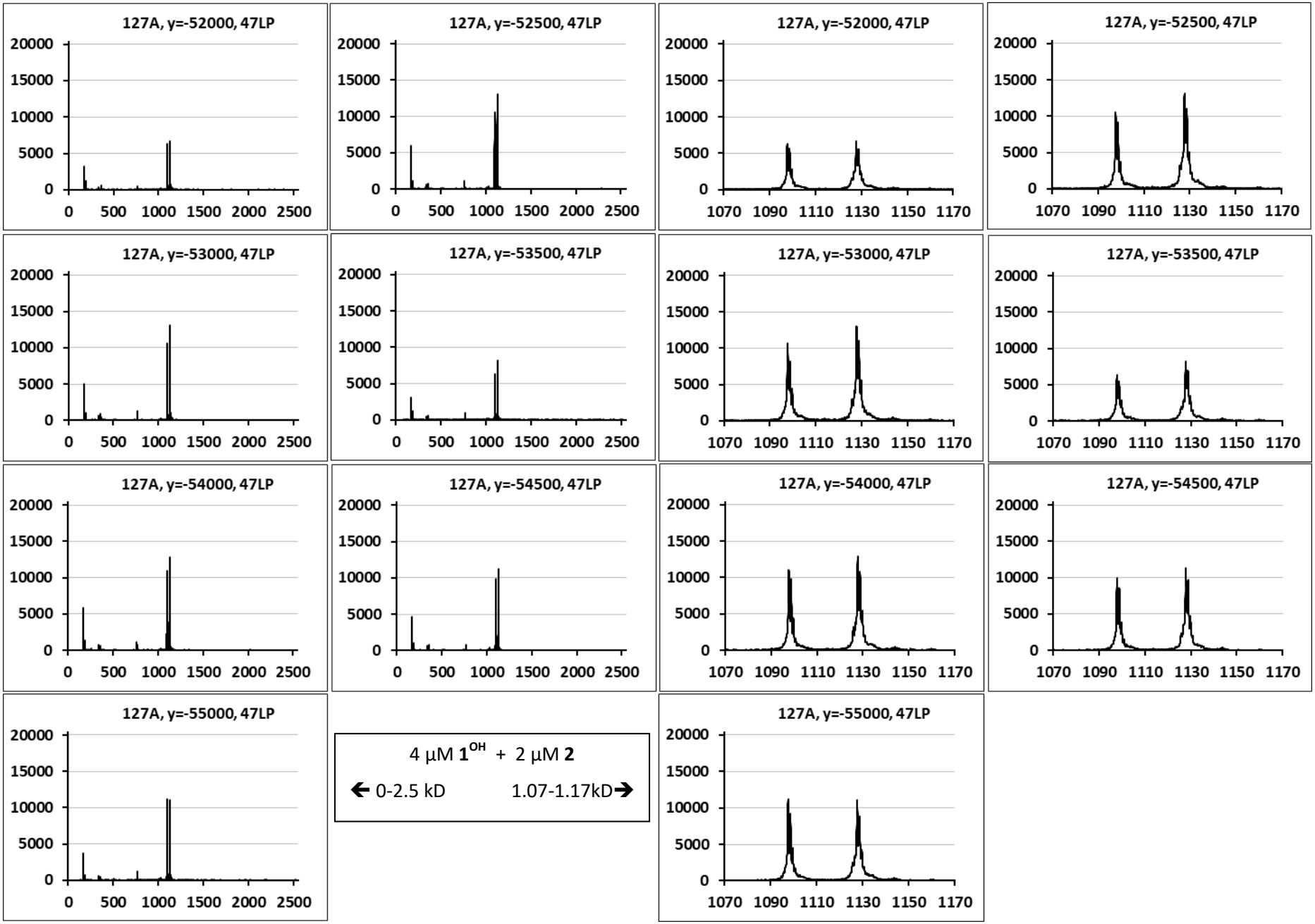
126B, y=-53000, 47LP
126B, y=-53500, 47LP
126B, y=-51500, 47LP
126B, y=-55700, 47LP
126B, y=-51500, 47LP
126B, y=-55700, 47LP
126B, y=-55000, 47LP
126B, y=-55000, 47LP
20000
15000
10000
5000
0
0 500 1000 1500 2000 2500
1070 1090 1110 1130 1150 1170
4 µM 1OH + 2 µM 2
← 0-2.5 kD
1.07-1.17kD →


127A, y=-52000, 47LP
127A, y=-52500, 47LP
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127A, y=-52500, 47LP
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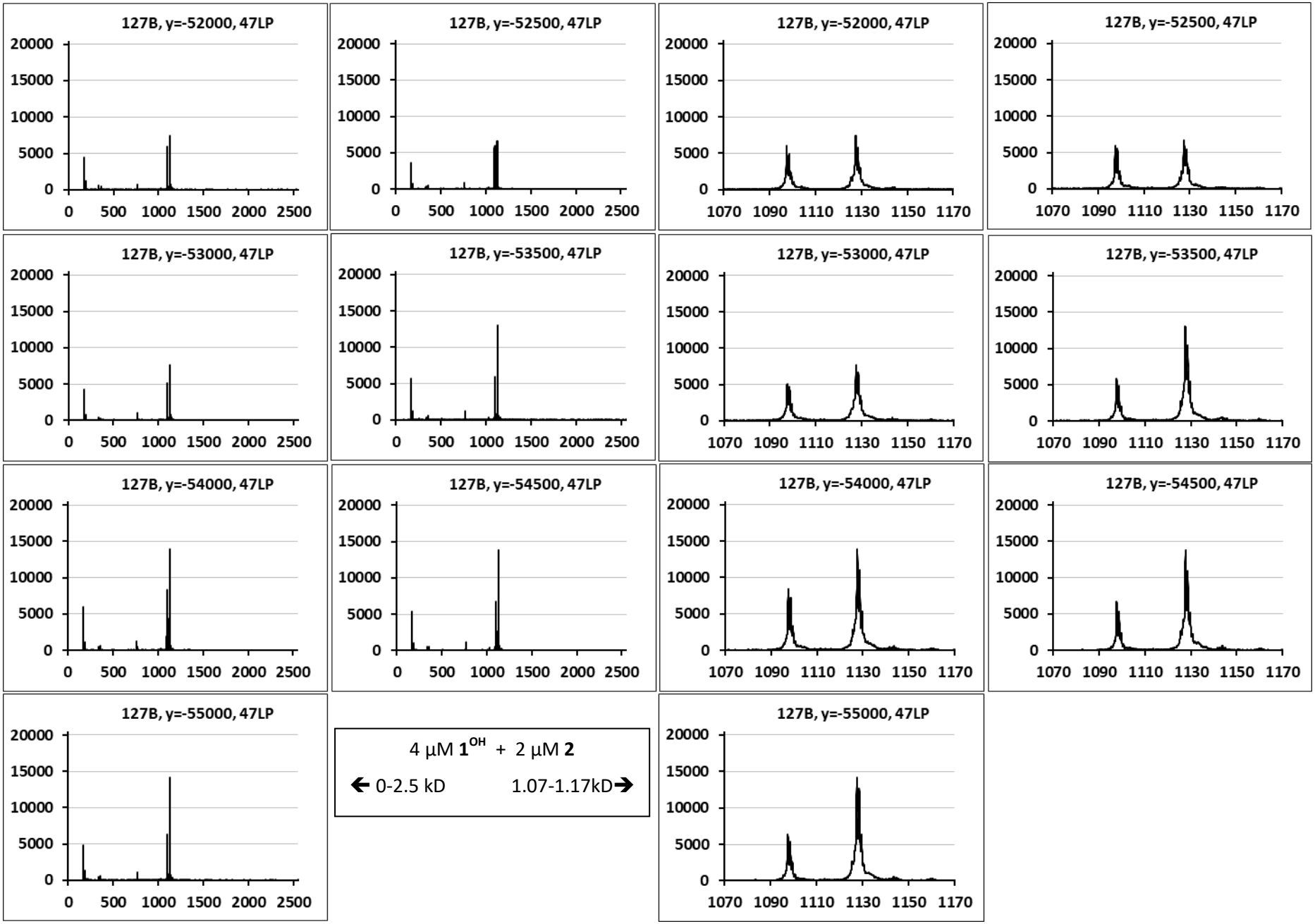
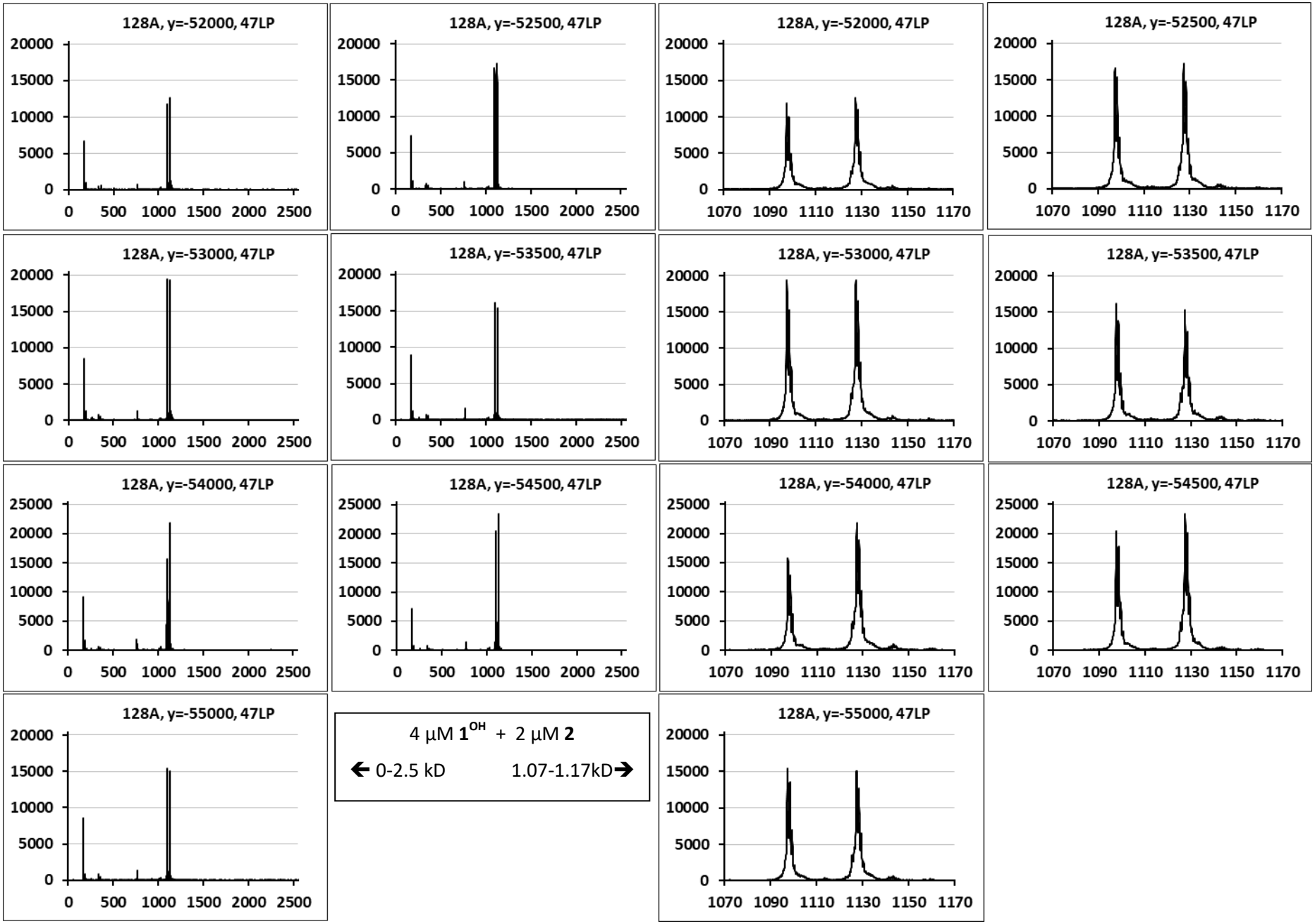
127A, y=-53500, 47LP
127A, y=-54000, 47LP
127A, y=-54500, 47LP
127A, y=-54000, 47LP
127A, y=-54500, 47LP
127A, y=-55000, 47LP
4 µM 1OH + 2 µM 2
← 0-2.5 kD
1.07-1.17kD→
127A, y=-55000, 47LP


127B, y=-52000, 47LP
127B, y=-52500, 47LP
127B, y=-52000, 47LP
127B, y=-52500, 47LP
127B, y=-53000, 47LP
127B, y=-53500, 47LP
127B, y=-53000, 47LP
127B, y=-53500, 47LP
127B, y=-54000, 47LP
127B, y=-54500, 47LP
127B, y=-54000, 47LP
127B, y=-54500, 47LP
127B, y=-55000, 47LP
4 µM 1OH + 2 µM 2
← 0-2.5 kD
1.07-1.17kD→
127B, y=-55000, 47LP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128A, y=-52500, 47LP
128A, y=-52000, 47LP
128A, y=-52500, 47LP
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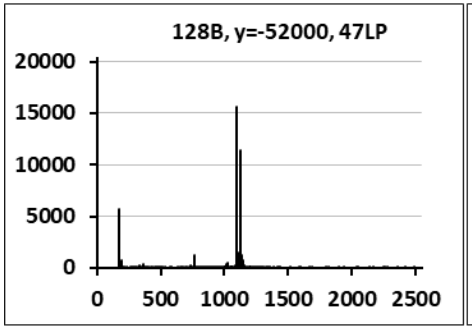
128A, y=-54500, 47LP
128A, y=-54000, 47LP
128A, y=-54500, 47LP
128A, y=-55000, 47LP
4 µM 1OH + 2 µM 2
← 0-2.5 kD
1.07-1.17kD→
128A, y=-55000, 47LP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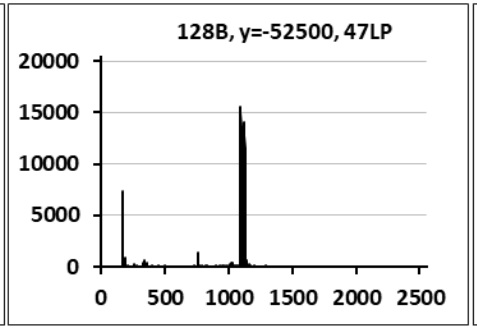
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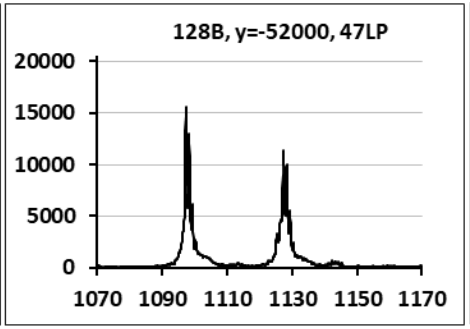
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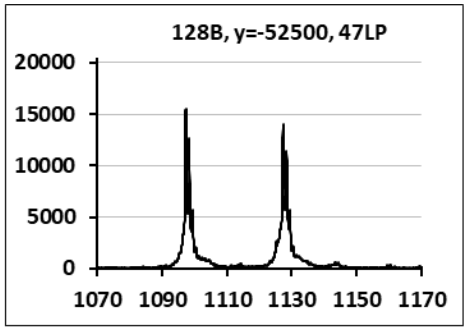
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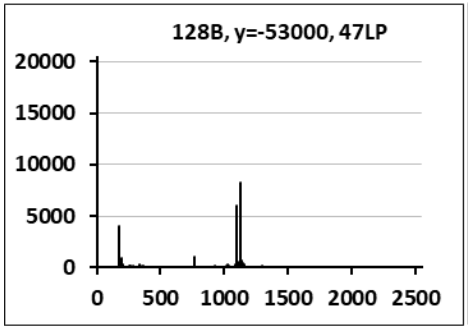
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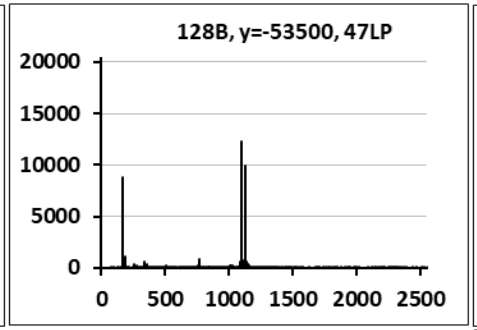
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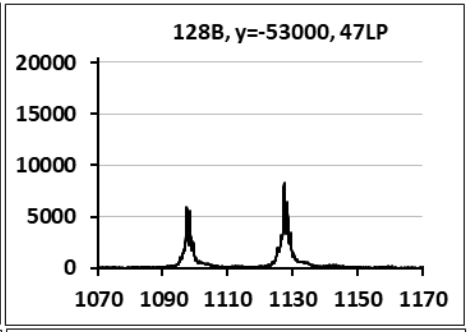
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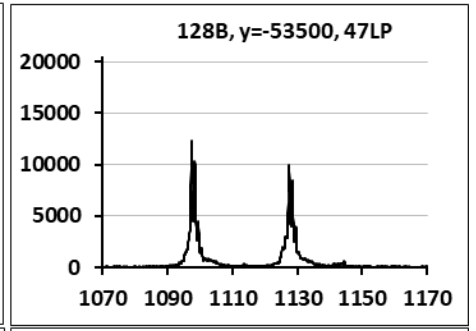
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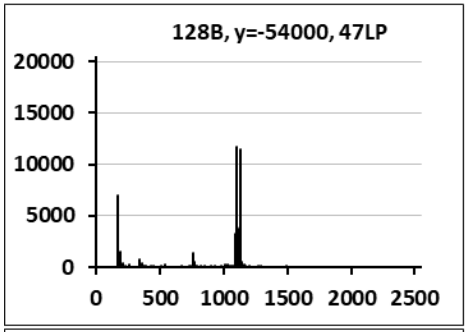
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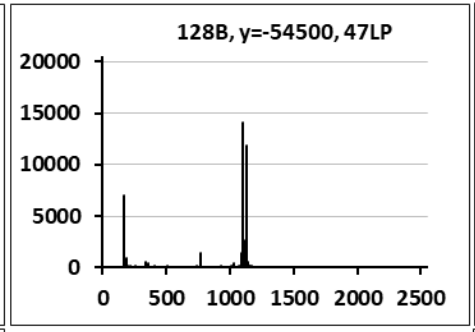
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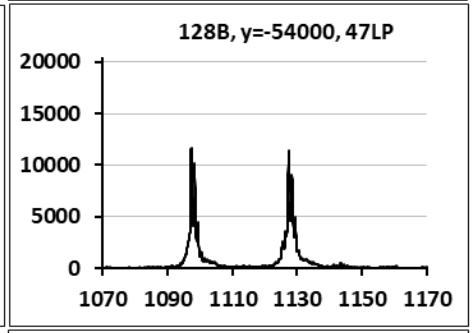
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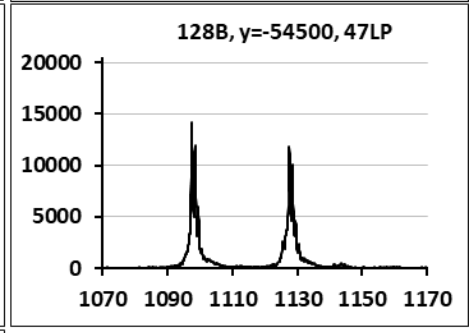
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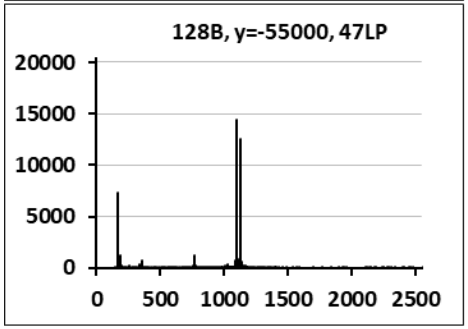
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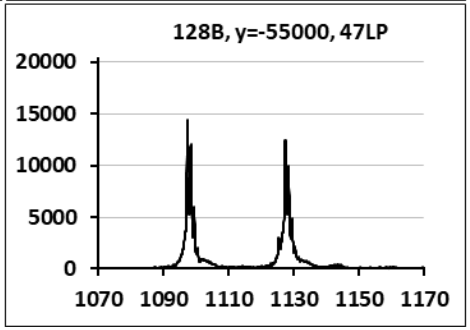
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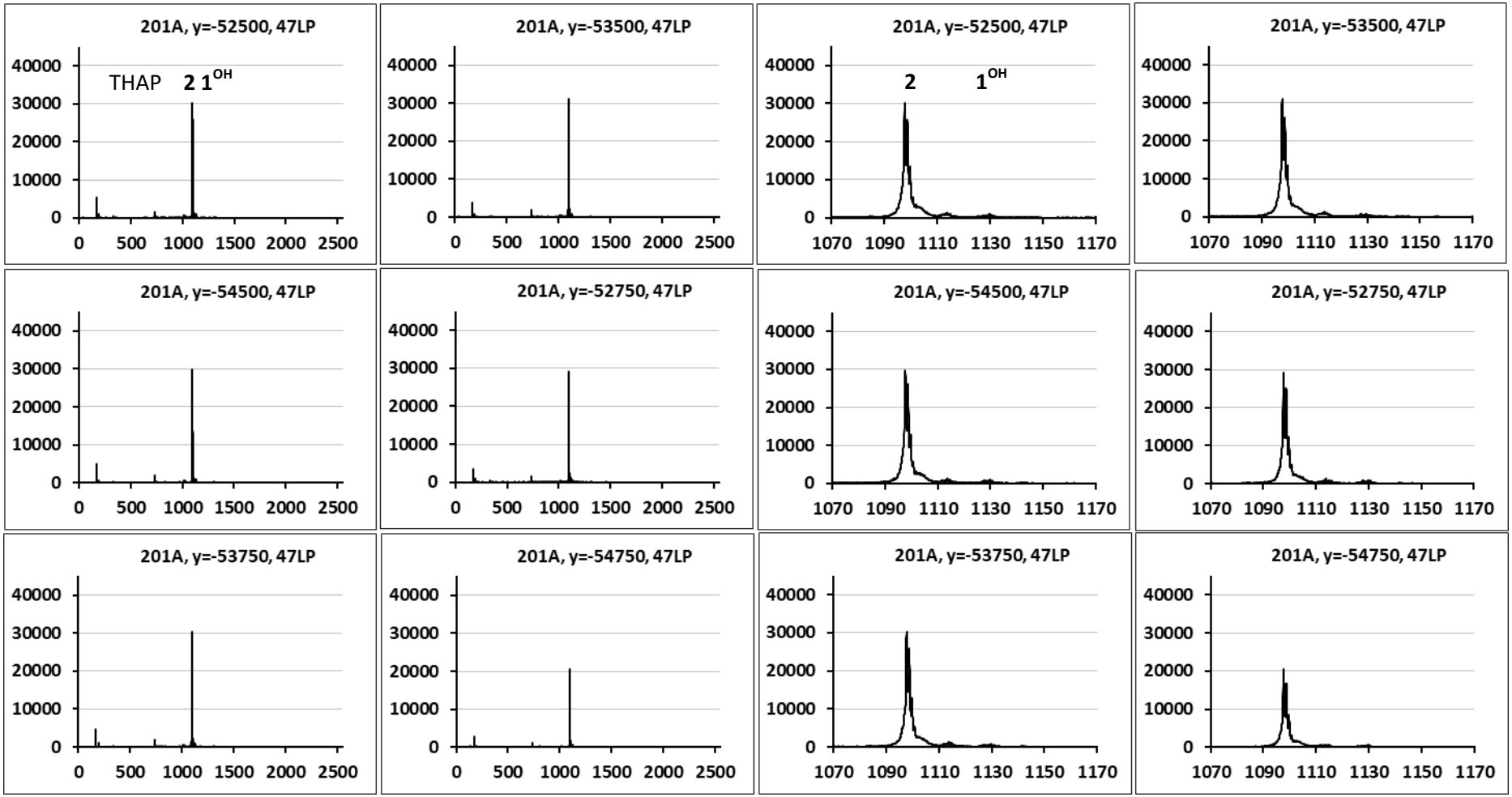
1070 1090 1110 1130 1150 1170

Fig. S3. Summaries and TOF-MS spectra (full range and expansions) of locations sampled to determine disappearance of $\mathbf{1}^{OH}$ following reaction of ($\mathbf{1}^{OH}\mathbf{1}^{OH}\mathbf{22}$) ML with 20 mM DTBP. Data collected at LP = 47 after applying a THAP thin film.

Summary: TOF-MS ML $\mathbf{1}^{OH}$ / **2** intensity and intensity ratio statistics after esterification with 20 mM DTBP, 40 mM EDC, 40 mM DMAP THAP thin film applied, LP = 47. (TOF-MS measured at 12 locations on each of 6 independently prepared samples.)

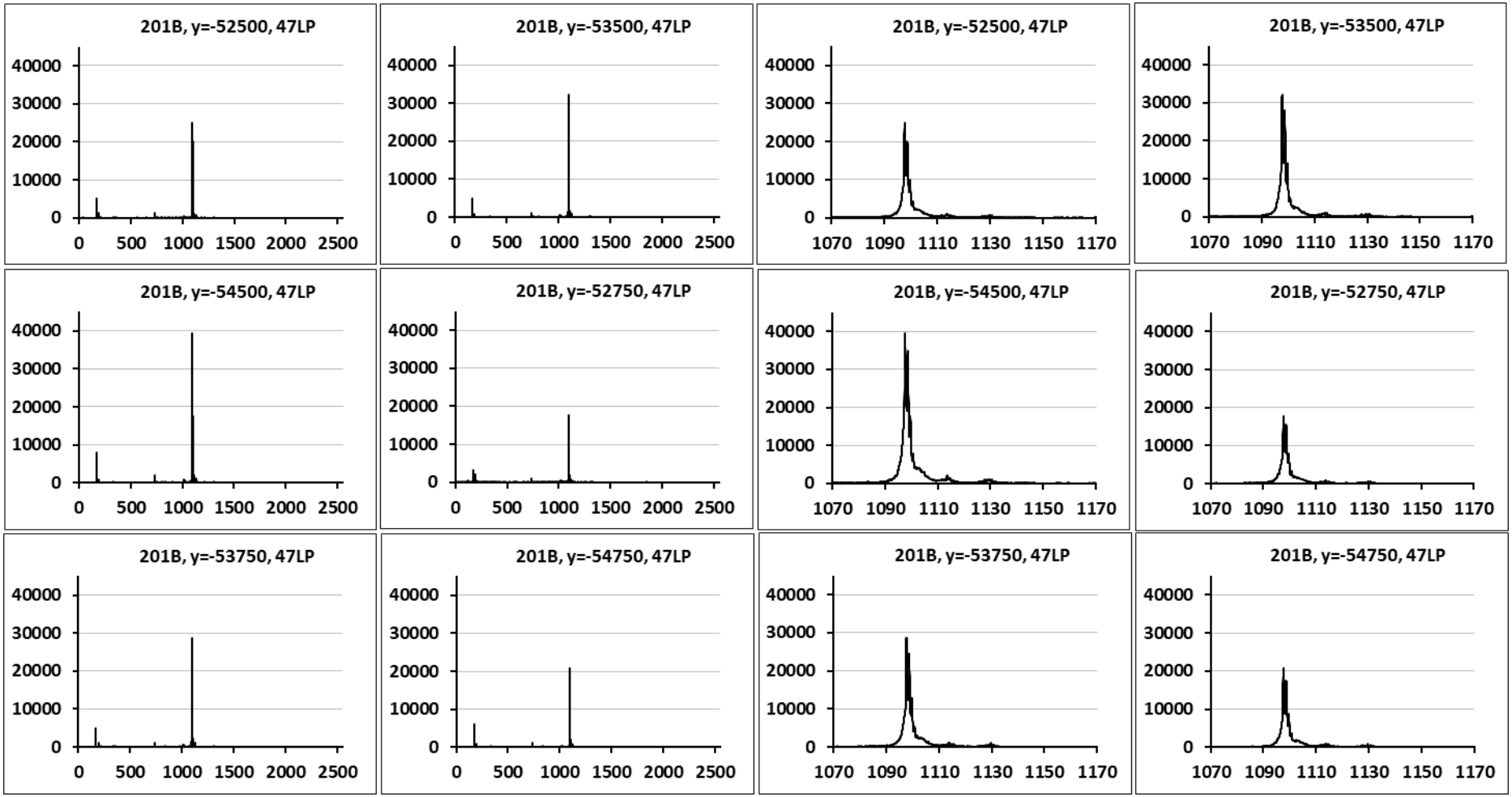
[1^{OH}]	[**2**]	**# Locations**	$\mathbf{1}^{OH}$ **/ 2**	$\mathbf{1}^{OH}$ **/ 2**	$\mathbf{1}^{OH}$ **/ 2**	$\mathbf{1}^{OH}$ **/ 2**	$\mathbf{1}^{OH}$ **/ 2**	$\mathbf{1}^{OH}$	$\mathbf{1}^{OH}$	$\mathbf{1}^{OH}$	$\mathbf{1}^{OH}$	$\mathbf{1}^{OH}$	**2**	**2**	**2**	**2**	**2**
µM	µM		**Max**	**Min**	**Avg**	**σ**	**σ/Avg**	**Max**	**Min**	**Avg**	**σ**	**σ/Avg**	**Max**	**Min**	**Avg**	**σ**	**σ/Avg**
4	6	12 (201A,B)	0.030	0.019	0.023	0.003	13%	1006	376	650	186	29%	39395	17736	27883	5975	21%
4	6	12 (202A,B)	0.025	0.016	0.020	0.003	13%	750	436	550	85	15%	30267	25121	27897	1534	5%
4	6	12 (203A,B)	0.026	0.017	0.021	0.003	14%	557	248	435	90	21%	26974	14350	20542	3210	16%
4	6	12 (204A,B)	0.025	0.014	0.020	0.003	17%	773	432	599	99	17%	39974	24974	30394	3680	12%
4	6	12 (230A,B)	0.024	0.015	0.019	0.003	13%	879	309	573	179	31%	44668	19514	29767	7950	27%
4	6	12 (231A,B)	0.028	0.016	0.020	0.003	16%	802	401	563	121	22%	34969	21822	27709	3755	14%
4	6	72	0.030	0.014	**0.020**	**0.003**	**15%**	1006	248	**561**	**144**	**26%**	44668	14350	**27365**	**5663**	**21%**



4 µM **1**^{OH} + 6 µM **2**

20 mM DTBP, 40 mM EDC, 40 mM DMAP

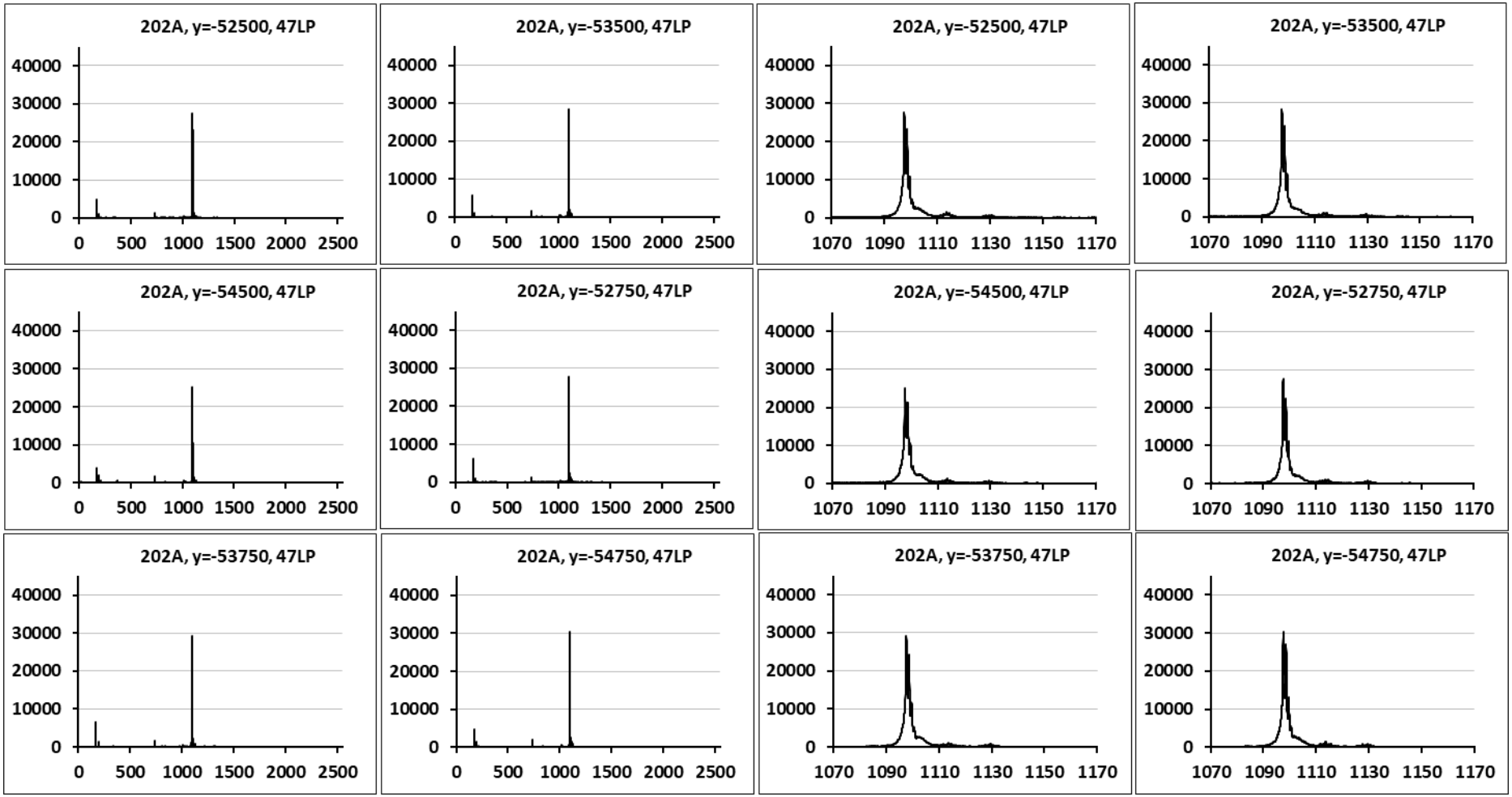
← 0-2.5 kD 1.07-1.17kD→



4 µM **1**^{OH} + 6 µM **2**

20 mM DTBP, 40 mM EDC, 40 mM DMAP

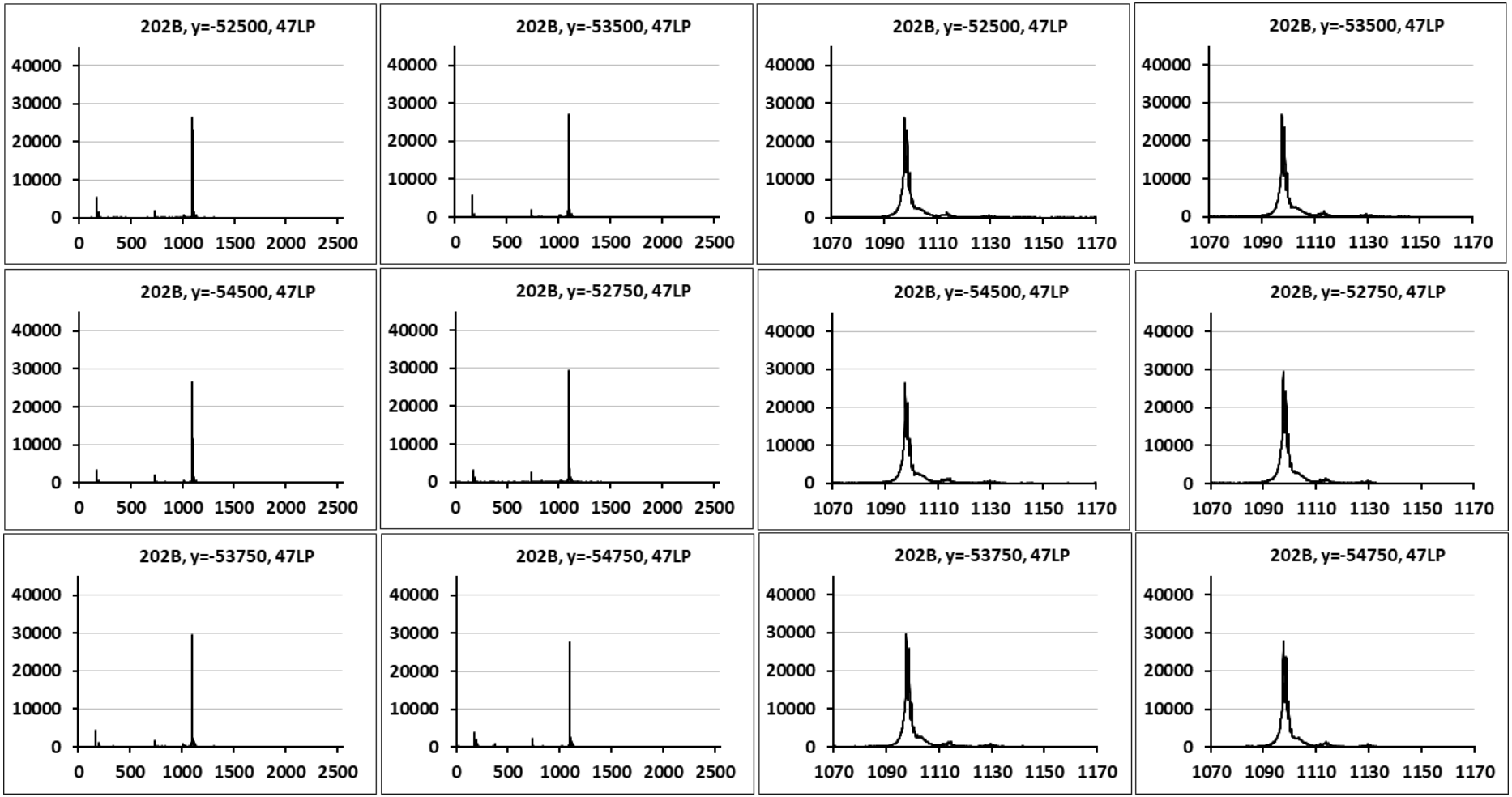
← 0-2.5 kD 1.07-1.17kD→



4 μM **1**^{OH} + 6 μM **2**

20 mM DTBP, 40 mM EDC, 40 mM DMAP

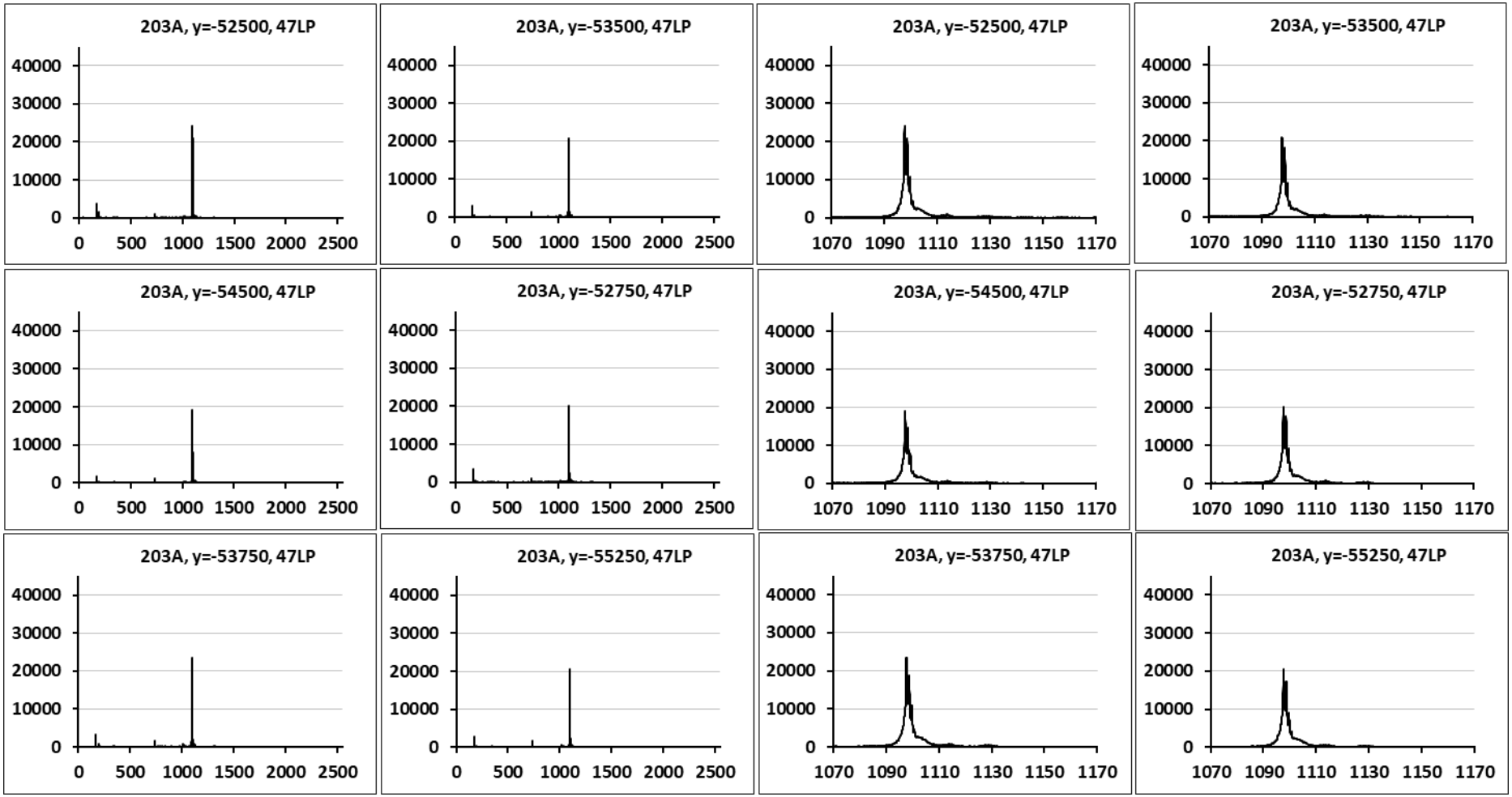
← 0-2.5 kD 1.07-1.17kD→



4 µM **1**^{OH} + 6 µM **2**

20 mM DTBP, 40 mM EDC, 40 mM DMAP

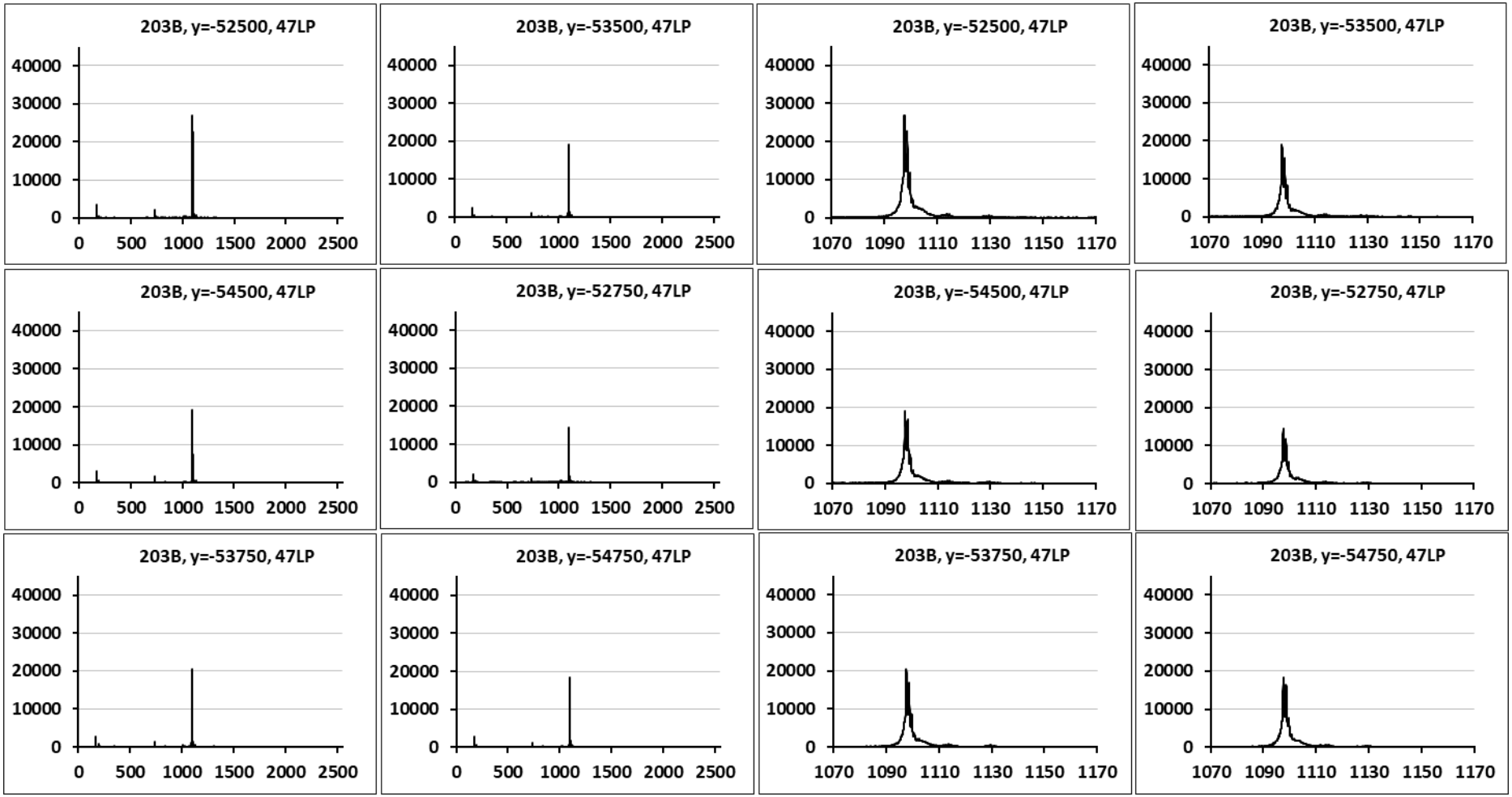
← 0-2.5 kD 1.07-1.17kD→



4 µM **1**^{OH} + 6 µM **2**

20 mM DTBP, 40 mM EDC, 40 mM DMAP

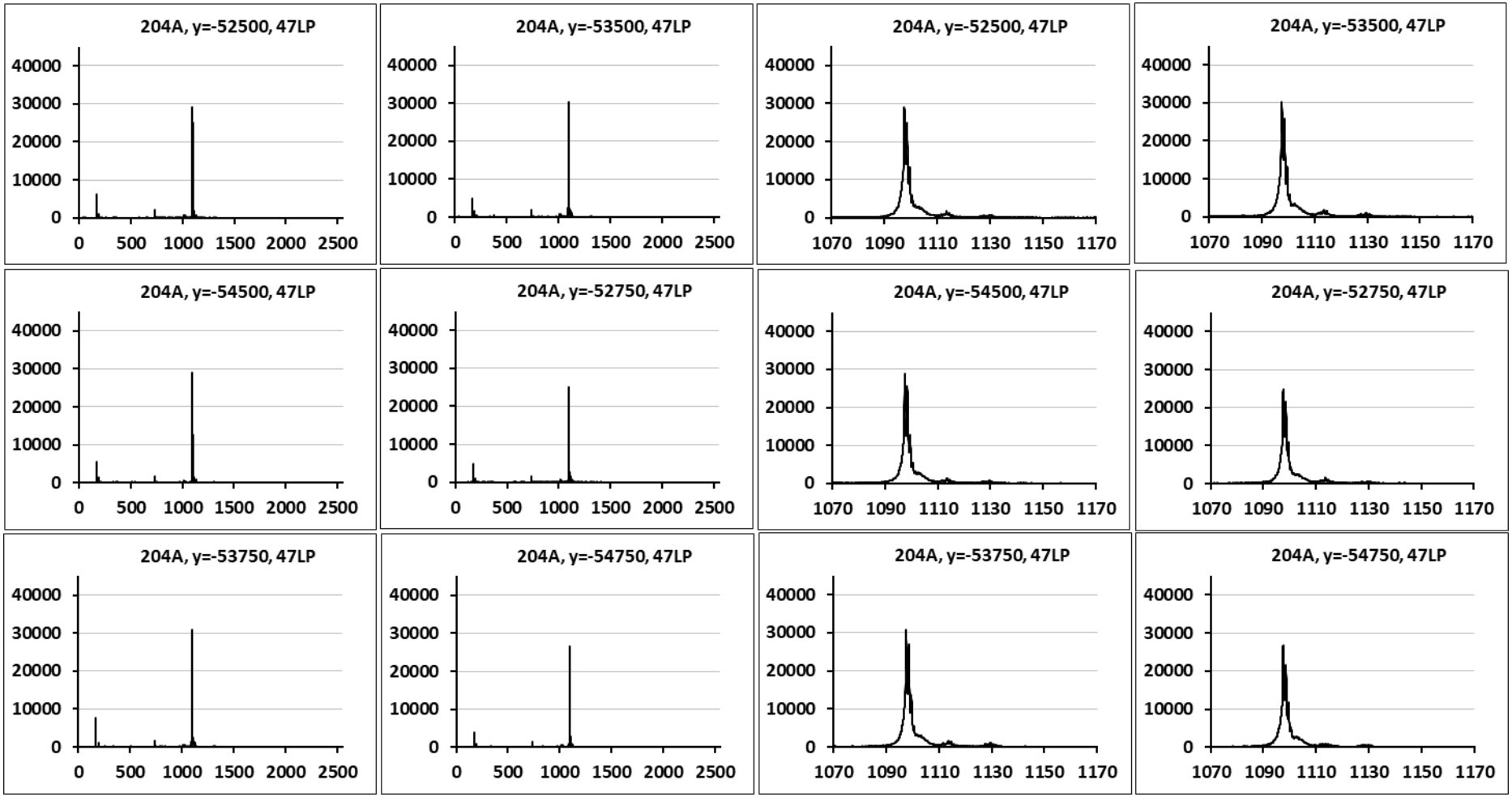
← 0-2.5 kD 1.07-1.17kD→



4 μM **1**^{OH} + 6 μM **2**

20 mM DTBP, 40 mM EDC, 40 mM DMAP

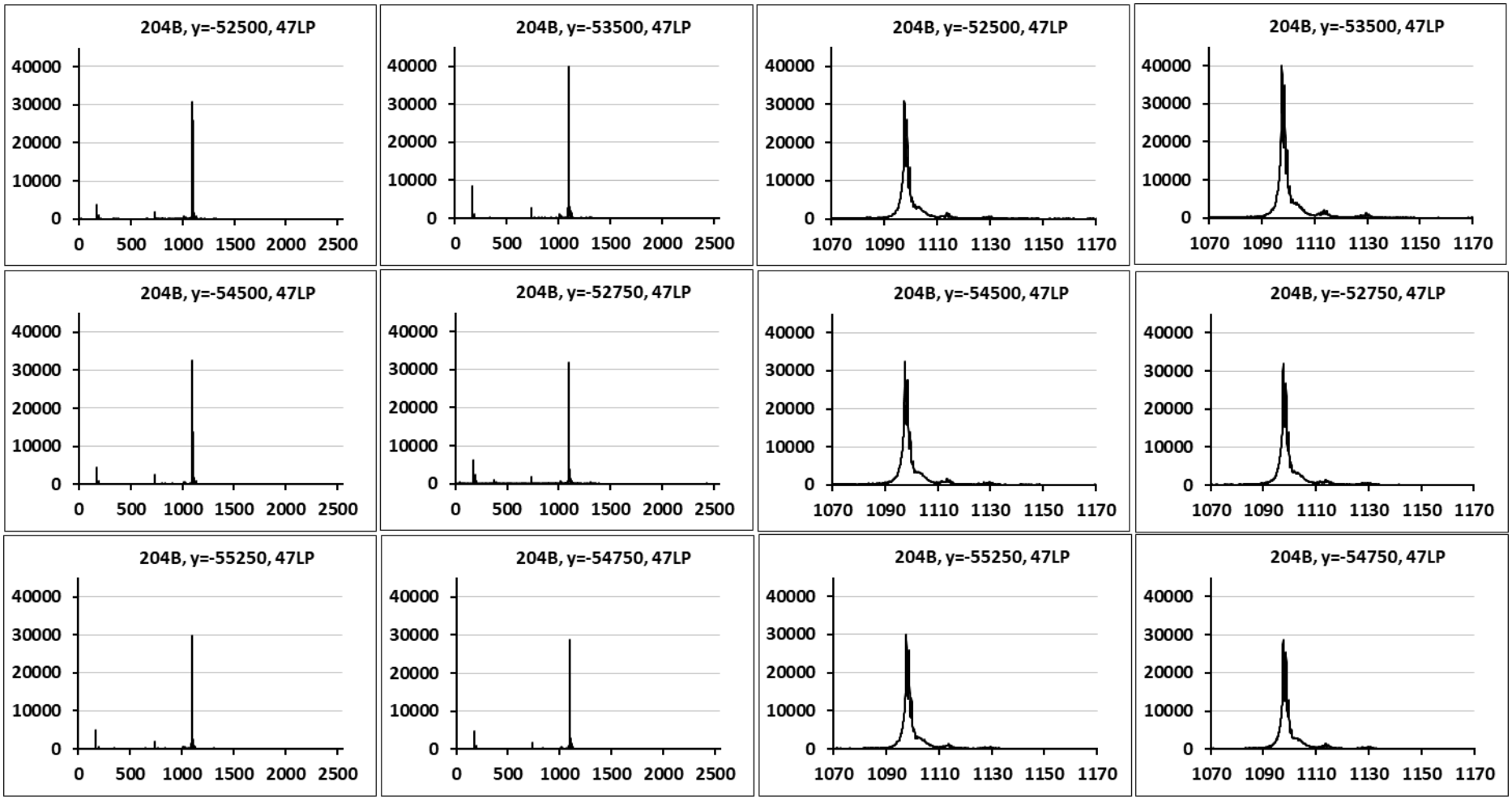
← 0-2.5 kD 1.07-1.17kD→



4 µM $\mathbf{1}^{OH}$ + 6 µM **2**

20 mM DTBP, 40 mM EDC, 40 mM DMAP

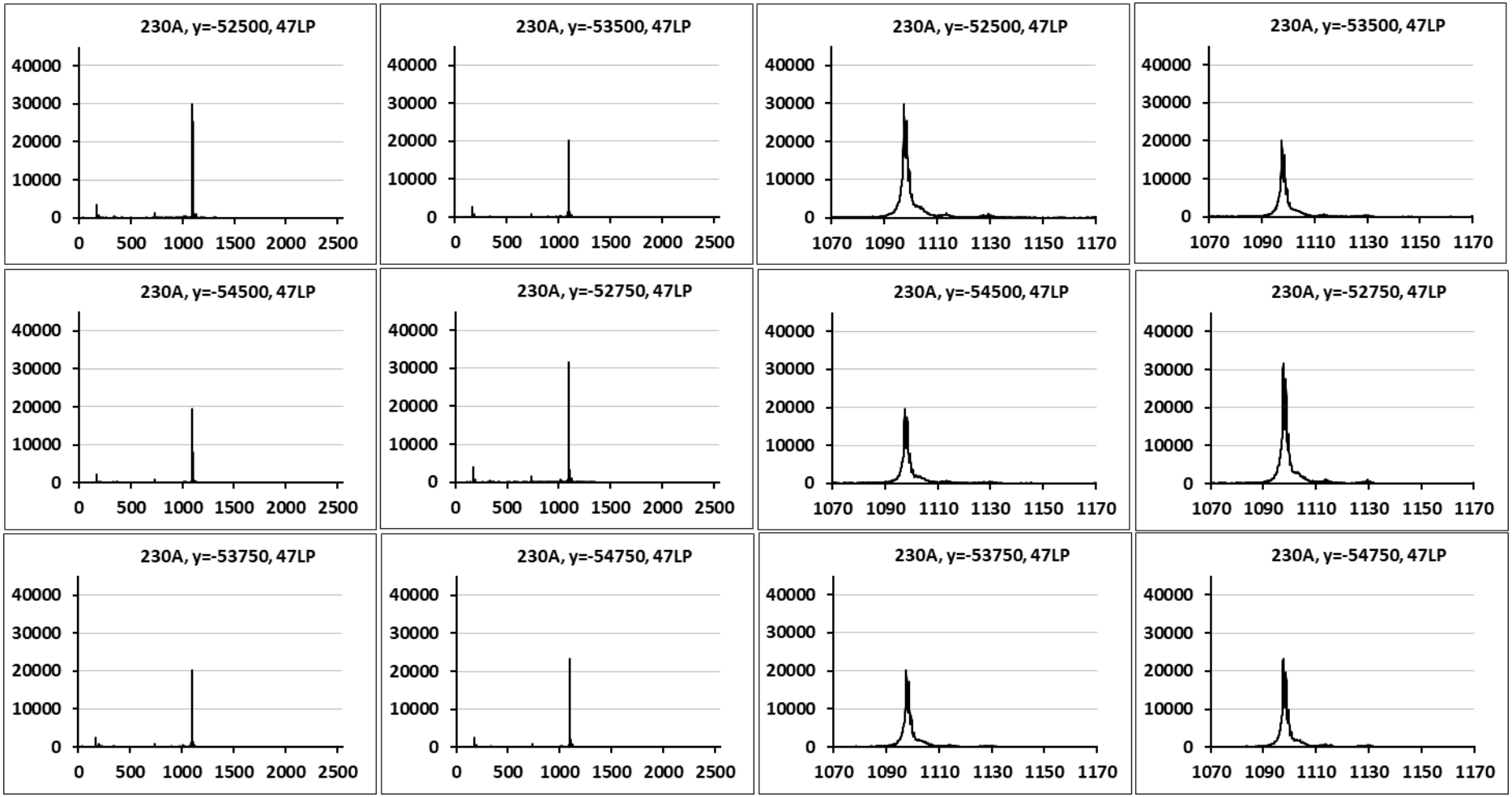
← 0-2.5 kD 1.07-1.17kD→



4 µM **1**^{OH} + 6 µM **2**

20 mM DTBP, 40 mM EDC, 40 mM DMAP

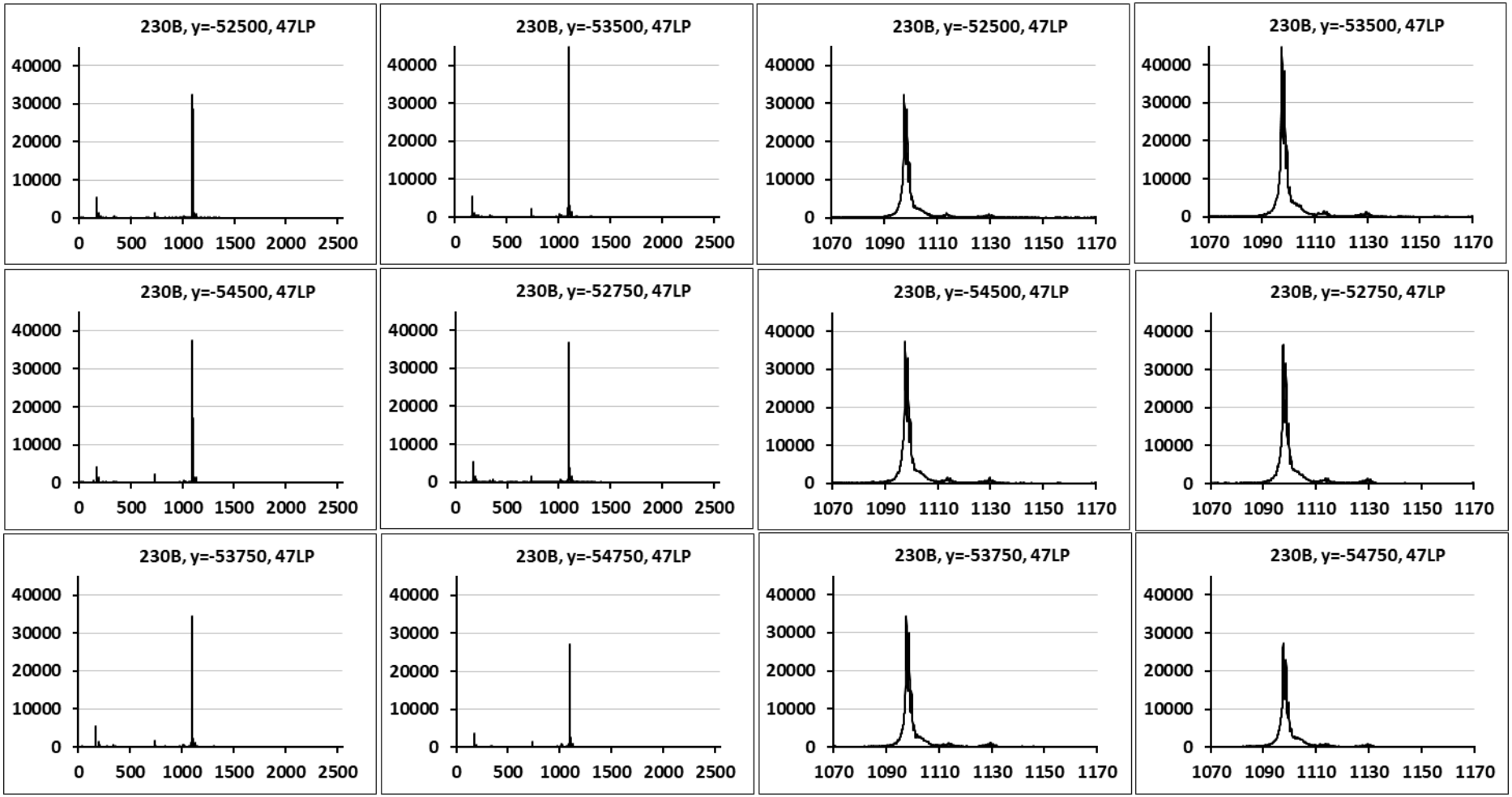
← 0-2.5 kD 1.07-1.17kD→



4 µM **1**^{OH} + 6 µM **2**

20 mM DTBP, 40 mM EDC, 40 mM DMAP

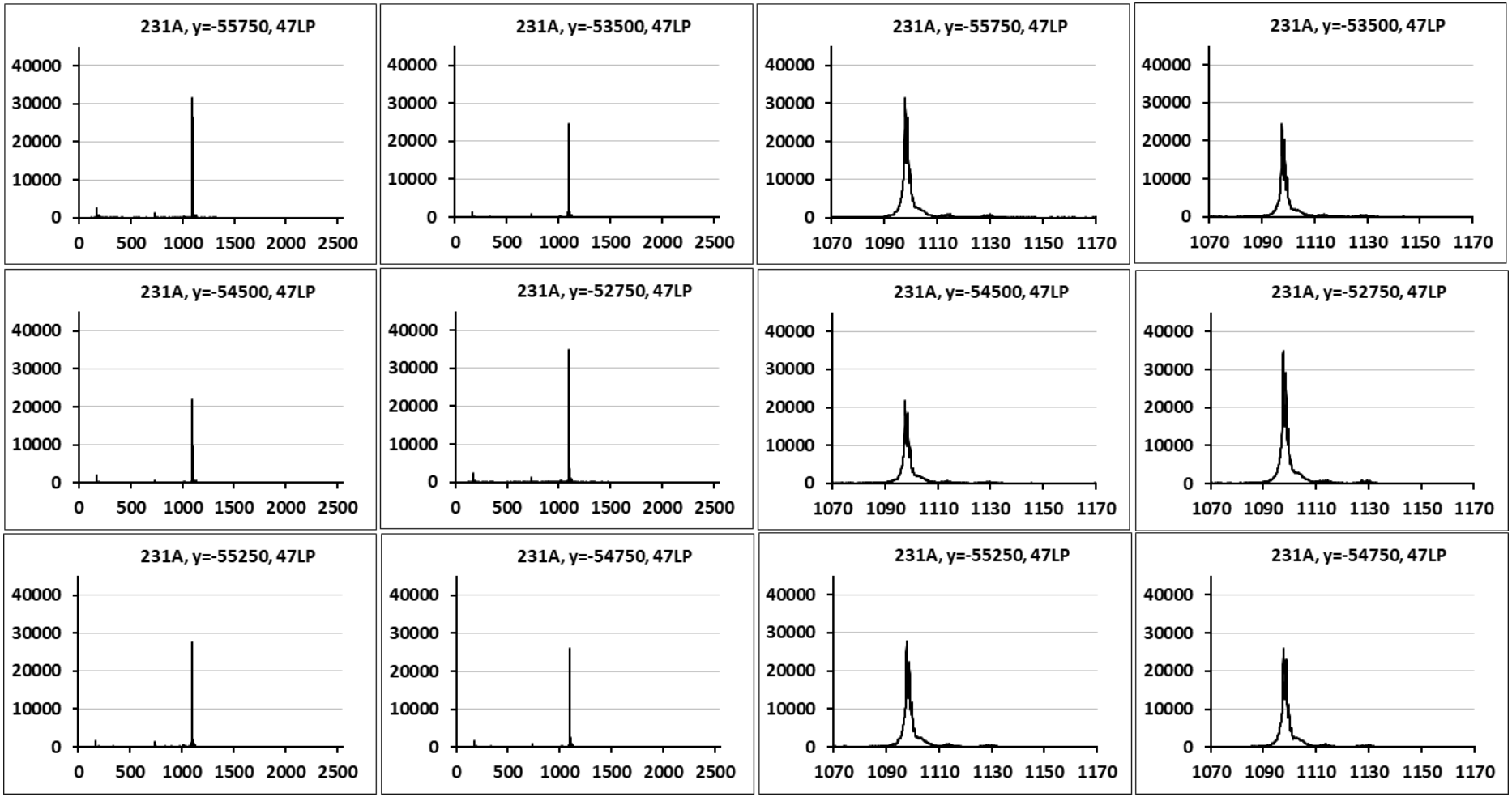
← 0-2.5 kD 1.07-1.17kD→



4 μM **1**^{OH} + 6 μM **2**

20 mM DTBP, 40 mM EDC, 40 mM DMAP

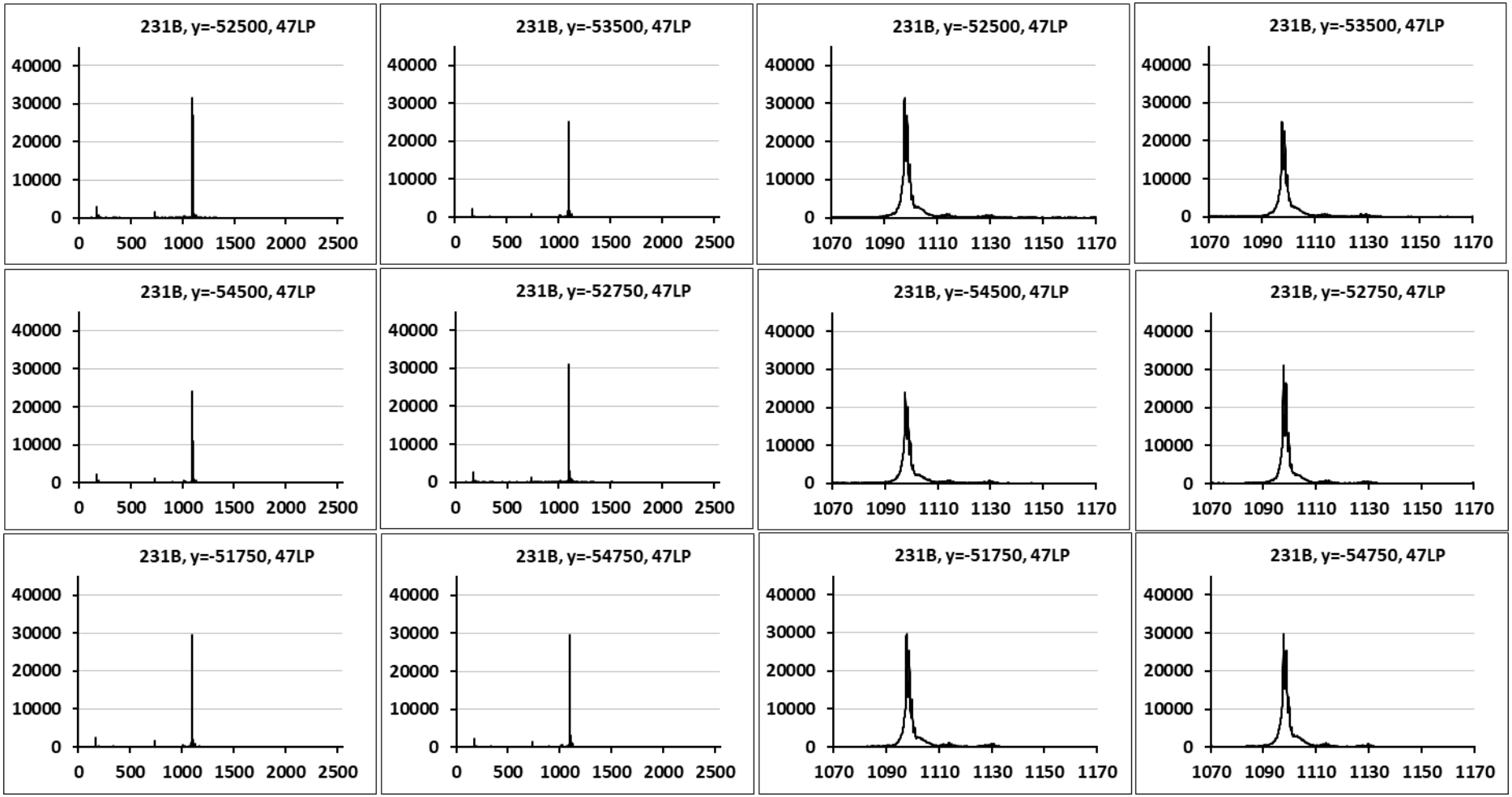
← 0-2.5 kD 1.07-1.17kD→



4 µM **1**^{OH} + 6 µM **2**

20 mM DTBP, 40 mM EDC, 40 mM DMAP

← 0-2.5 kD 1.07-1.17kD→



4 μM **1**^{OH} + 6 μM **2**

20 mM DTBP, 40 mM EDC, 40 mM DMAP

← 0-2.5 kD 1.07-1.17kD→

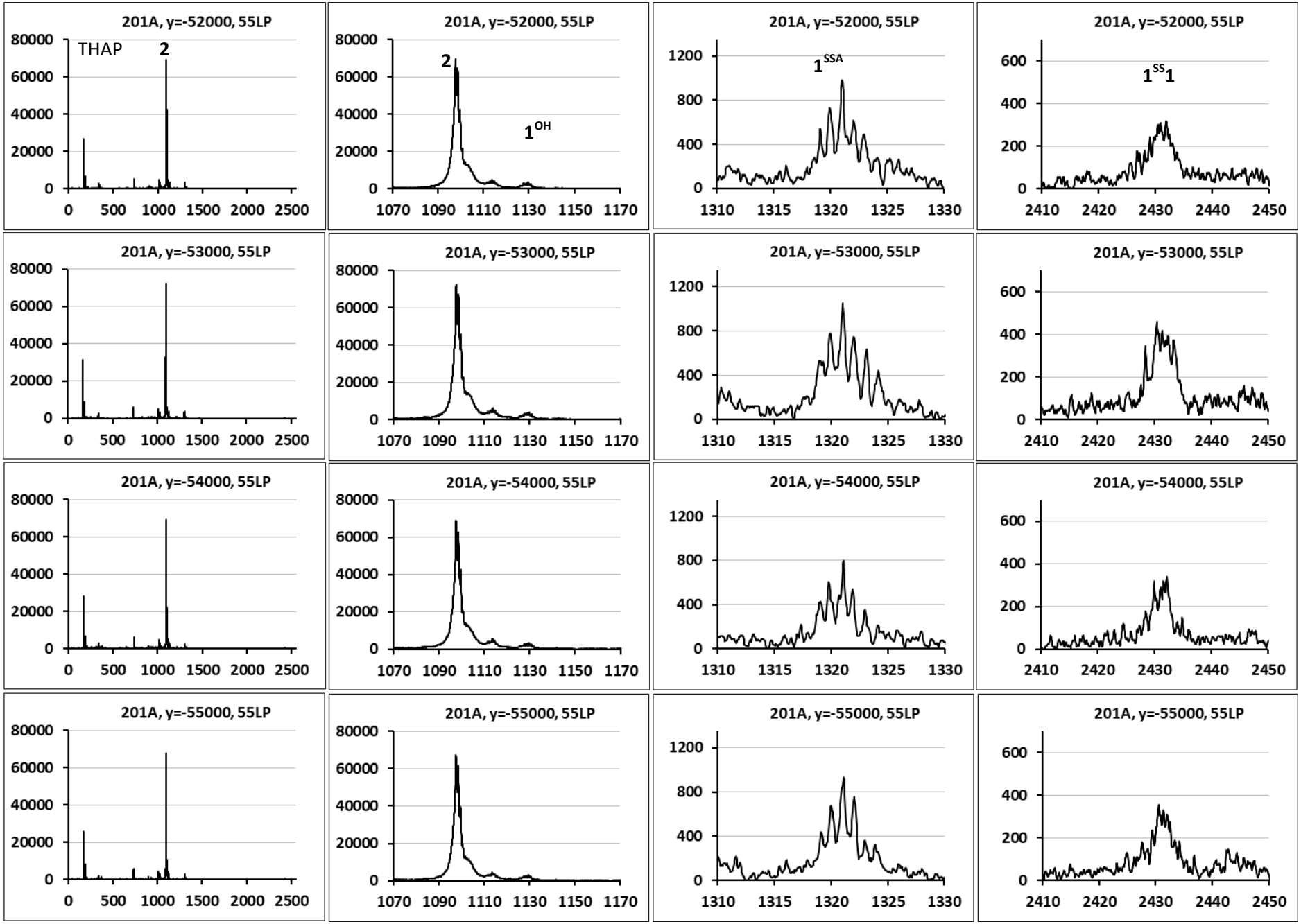
Fig. S4. Summaries and TOF-MS spectra (full range and expansions) of locations sampled to determine $\mathbf{1}^{OH}$, $\mathbf{1}^{SSA}$ and $\mathbf{1}^{SS}\mathbf{1}$ intensities following reaction of ($\mathbf{1}^{OH}\mathbf{1}^{OH}\mathbf{22}$) ML with 20 mM or 100 mM DTBP. Data collected at LP = 55 after applying a THAP thin film.

Summary: TOF-MS ML $\mathbf{1}^{OH}$, $\mathbf{1}^{SSA}$ and $\mathbf{1}^{SS}\mathbf{1}$ intensity statistics after esterification with 20 mM DTBP, 40 mM EDC, 40 mM DMAP. THAP thin film applied, LP = 55. (TOF-MS measured at 8 locations on each of 6 independently prepared samples.)

[$\mathbf{1}^{OH}$]	[**2**]	**# Locations**	$\mathbf{1}^{OH}$	$\mathbf{1}^{OH}$	$\mathbf{1}^{OH}$	$\mathbf{1}^{OH}$	$\mathbf{1}^{OH}$	$\mathbf{1}^{SSA}$	$\mathbf{1}^{SSA}$	$\mathbf{1}^{SSA}$	$\mathbf{1}^{SSA}$	$\mathbf{1}^{SSA}$	$\mathbf{1}^{SS}\mathbf{1}$	$\mathbf{1}^{SS}\mathbf{1}$	$\mathbf{1}^{SS}\mathbf{1}$	$\mathbf{1}^{SS}\mathbf{1}$	$\mathbf{1}^{SS}\mathbf{1}$
µM	µM	(file names)	**Max**	**Min**	**Avg**	**σ**	**σ/Avg**	**Max**	**Min**	**Avg**	**σ**	**σ/Avg**	**Max**	**Min**	**Avg**	**σ**	**σ/Avg**
4	6	8 (201A,B)	3756	2259	3177	391	12%	1255	590	809	222	27%	560	318	409	86	21%
4	6	8 (202A,B)	3863	1519	2895	546	19%	994	380	659	185	28%	618	228	450	138	31%
4	6	8 (203A,B)	2417	1955	1950	296	15%	878	332	591	194	33%	523	176	317	113	36%
4	6	8 (204A,B)	3029	1955	2442	334	14%	839	437	641	128	20%	506	177	341	111	33%
4	6	8 (230A,B)	3180	1928	2482	501	20%	958	591	749	139	19%	389	158	271	74	27%
4	6	8 (231A,B)	3264	1529	2374	520	22%	1005	435	784	200	26%	428	165	313	77	25%
4	6	48	3863	1519	**2553**	**576**	**23%**	1255	332	**705**	**189**	**27%**	618	158	**350**	**115**	**33%**

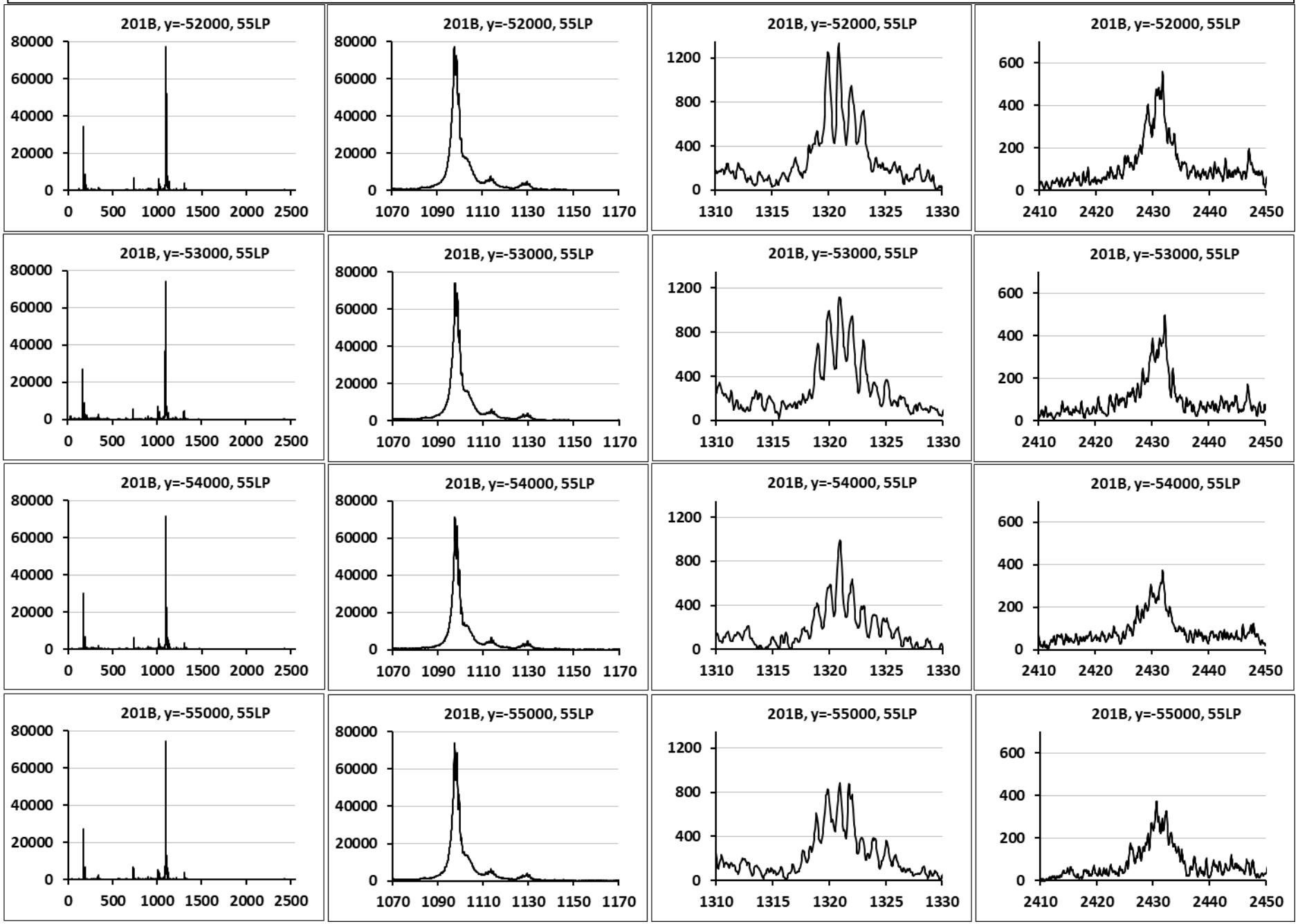
4 μM $\mathbf{1}^{OH}$ + 6 μM **2** 20 mM DTBP, 40 mM EDC, 40 mM DMAP

0-2.5 kD	1.07-1.17kD	1.31-1.33 kD	2.41-2.45 kD



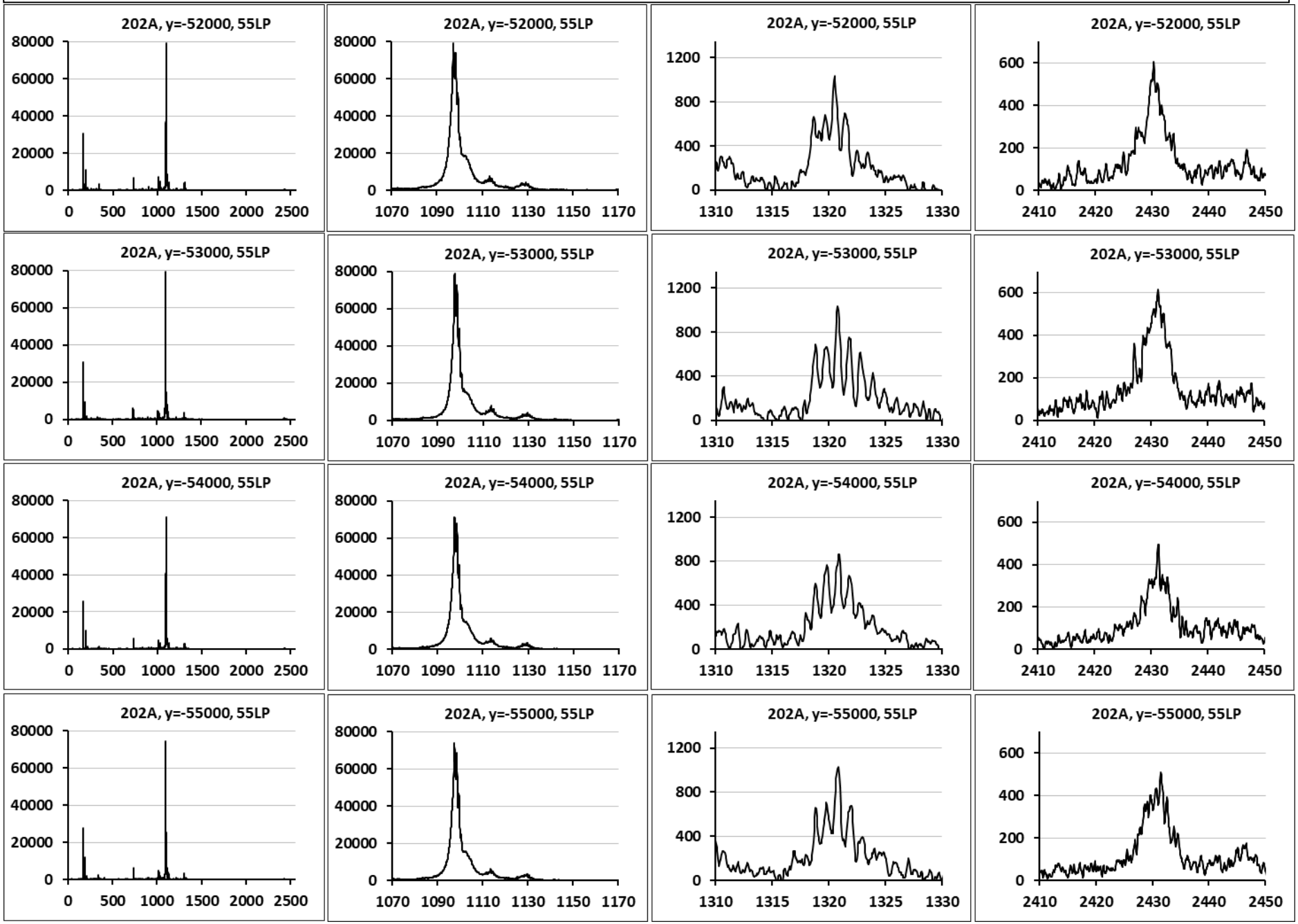
4 μM **1**^{OH} + 6 μM **2** 20 mM DTBP, 40 mM EDC, 40 mM DMAP

0-2.5 kD	1.07-1.17kD	1.31-1.33 kD	2.41-2.45 kD



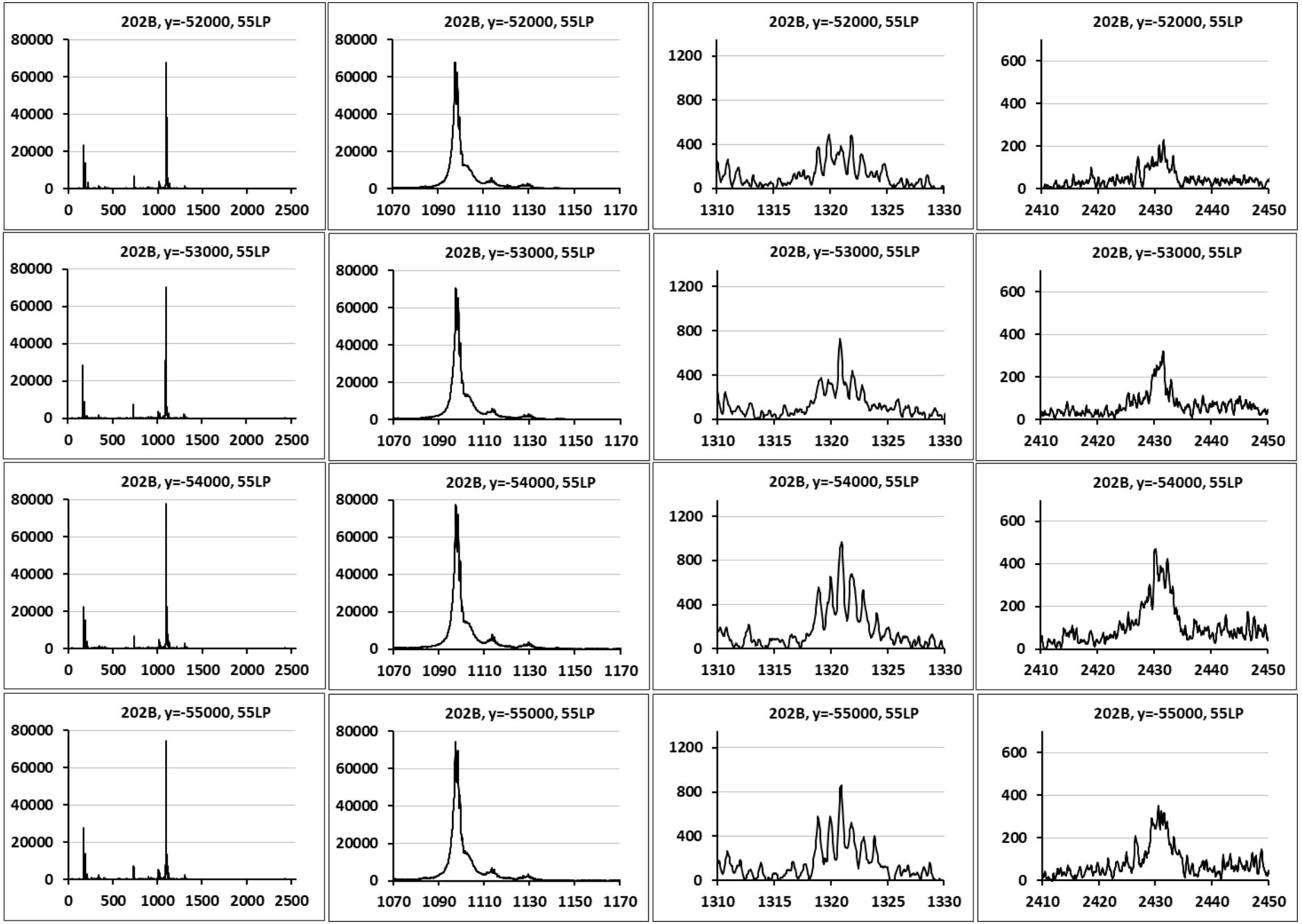
4 µM **1**^{OH} + 6 µM **2** 20 mM DTBP, 40 mM EDC, 40 mM DMAP

0-2.5 kD	1.07-1.17kD	1.31-1.33 kD	2.41-2.45 kD



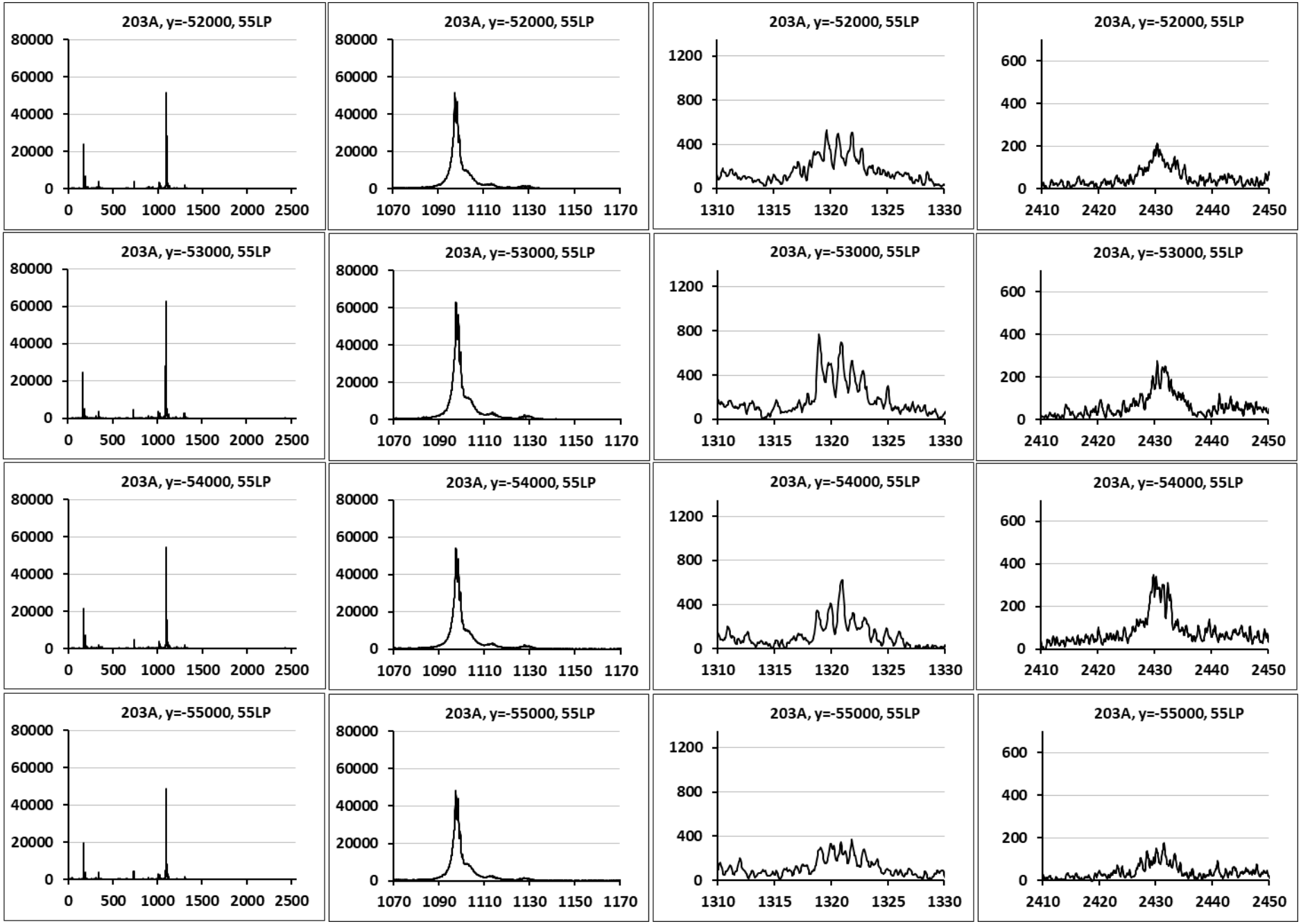
4 μM **1**^{OH} + 6 μM **2** 20 mM DTBP, 40 mM EDC, 40 mM DMAP

0-2.5 kD	1.07-1.17kD	1.31-1.33 kD	2.41-2.45 kD



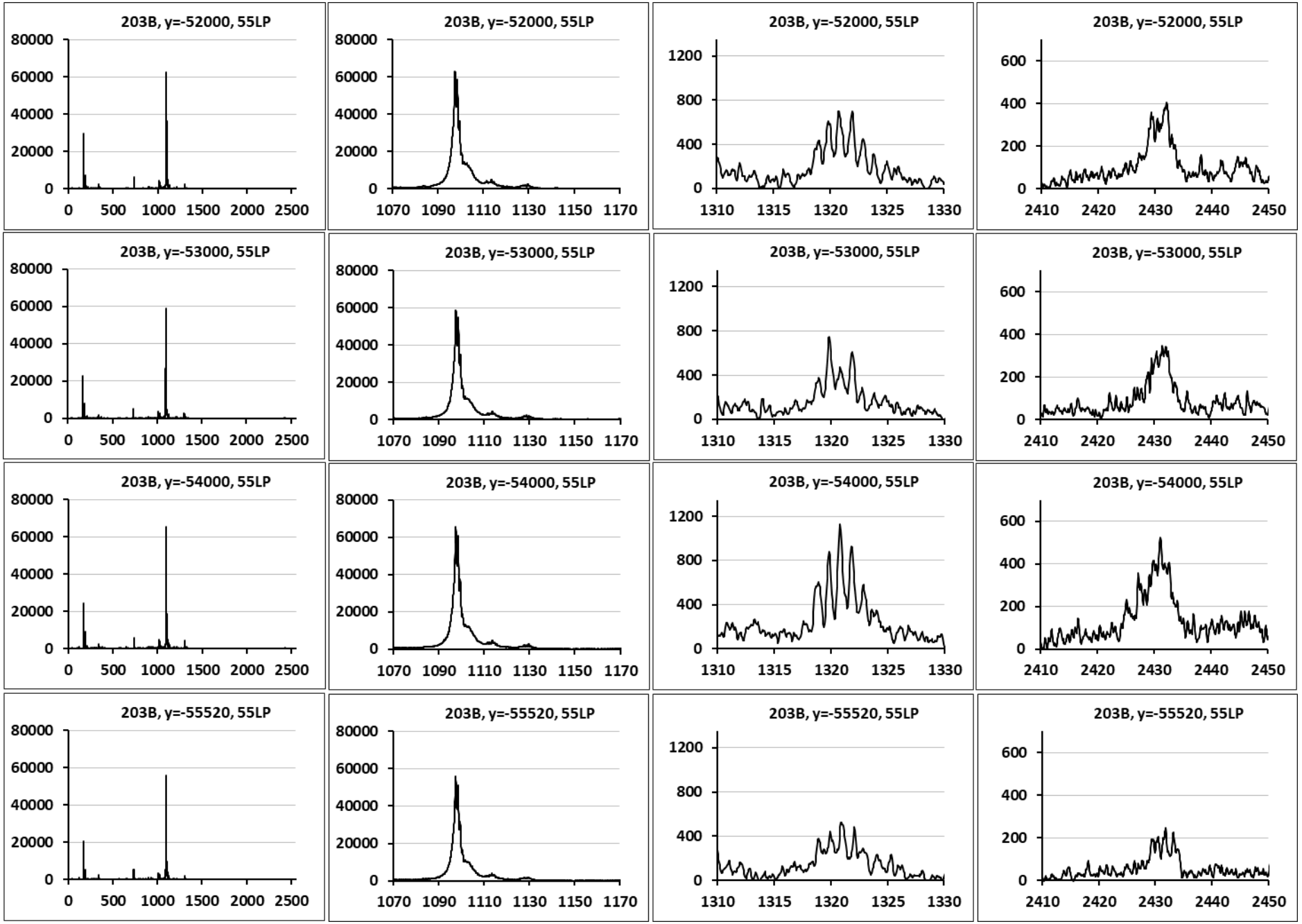
4 μM **1**^{OH} + 6 μM **2** 20 mM DTBP, 40 mM EDC, 40 mM DMAP

0-2.5 kD	1.07-1.17kD	1.31-1.33 kD	2.41-2.45 kD



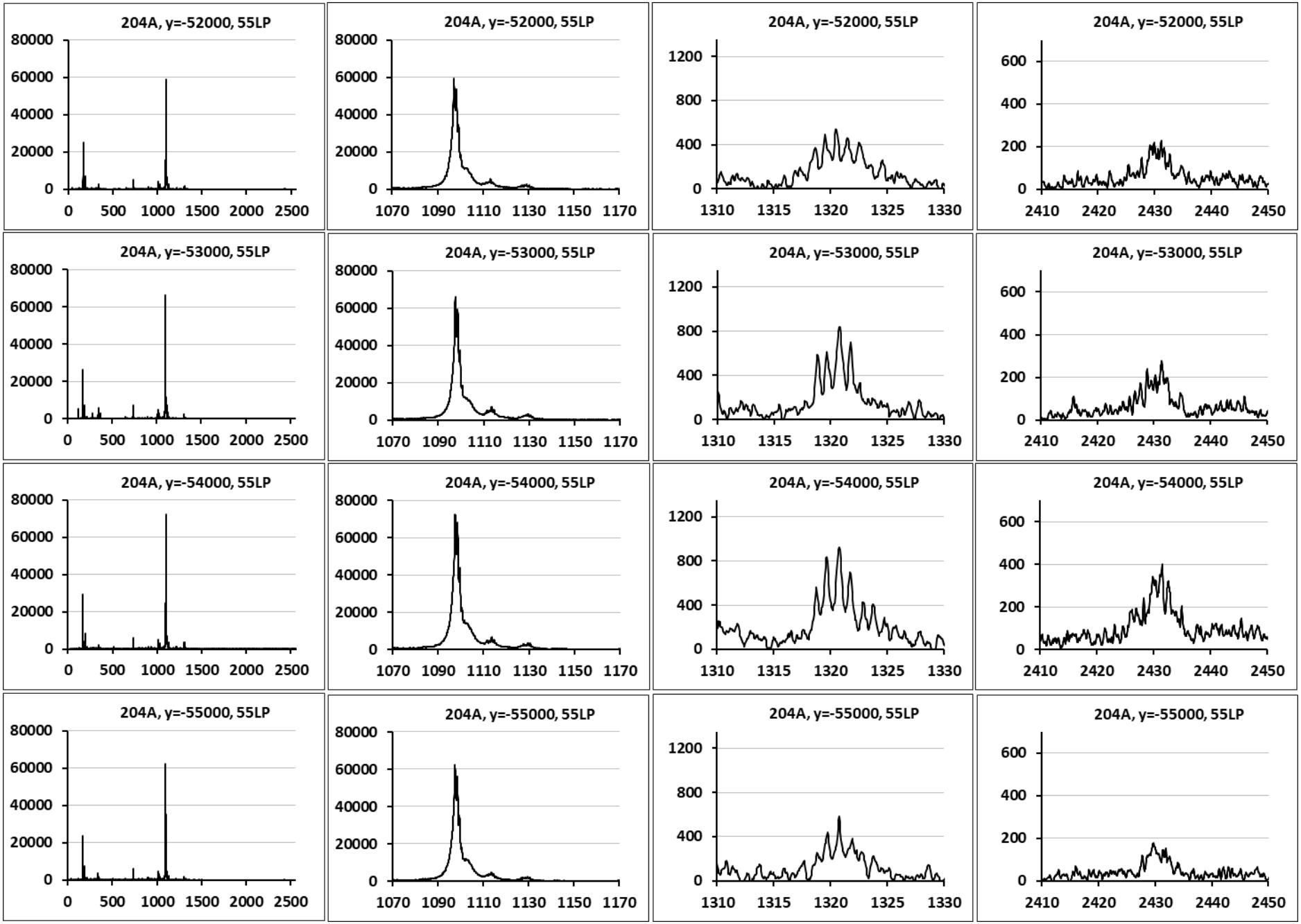
4 μM **1**^{OH} + 6 μM **2** 20 mM DTBP, 40 mM EDC, 40 mM DMAP

0-2.5 kD	1.07-1.17kD	1.31-1.33 kD	2.41-2.45 kD



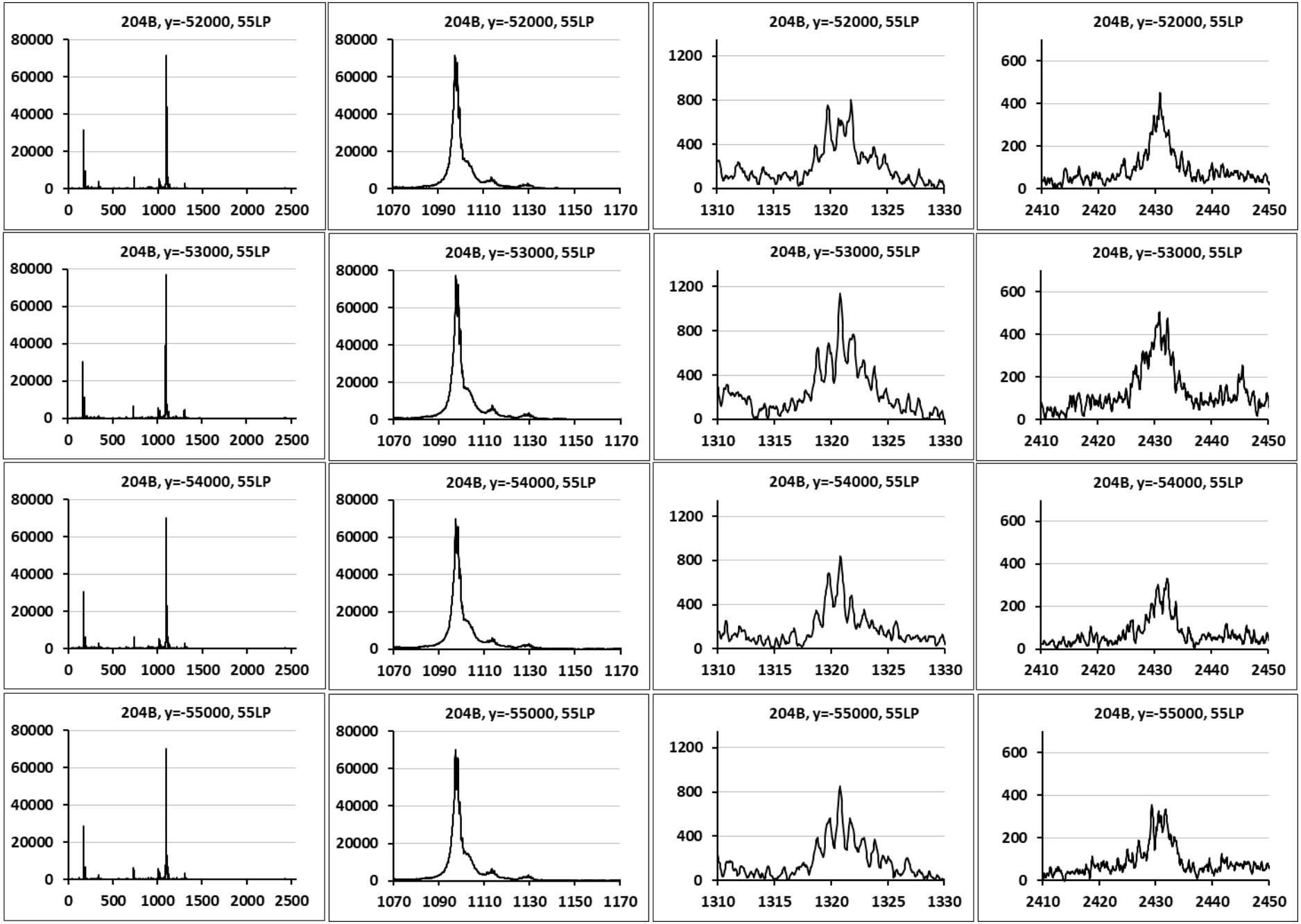
4 μM $\mathbf{1}^{OH}$ + 6 μM **2** 20 mM DTBP, 40 mM EDC, 40 mM DMAP

0-2.5 kD	1.07-1.17kD	1.31-1.33 kD	2.41-2.45 kD



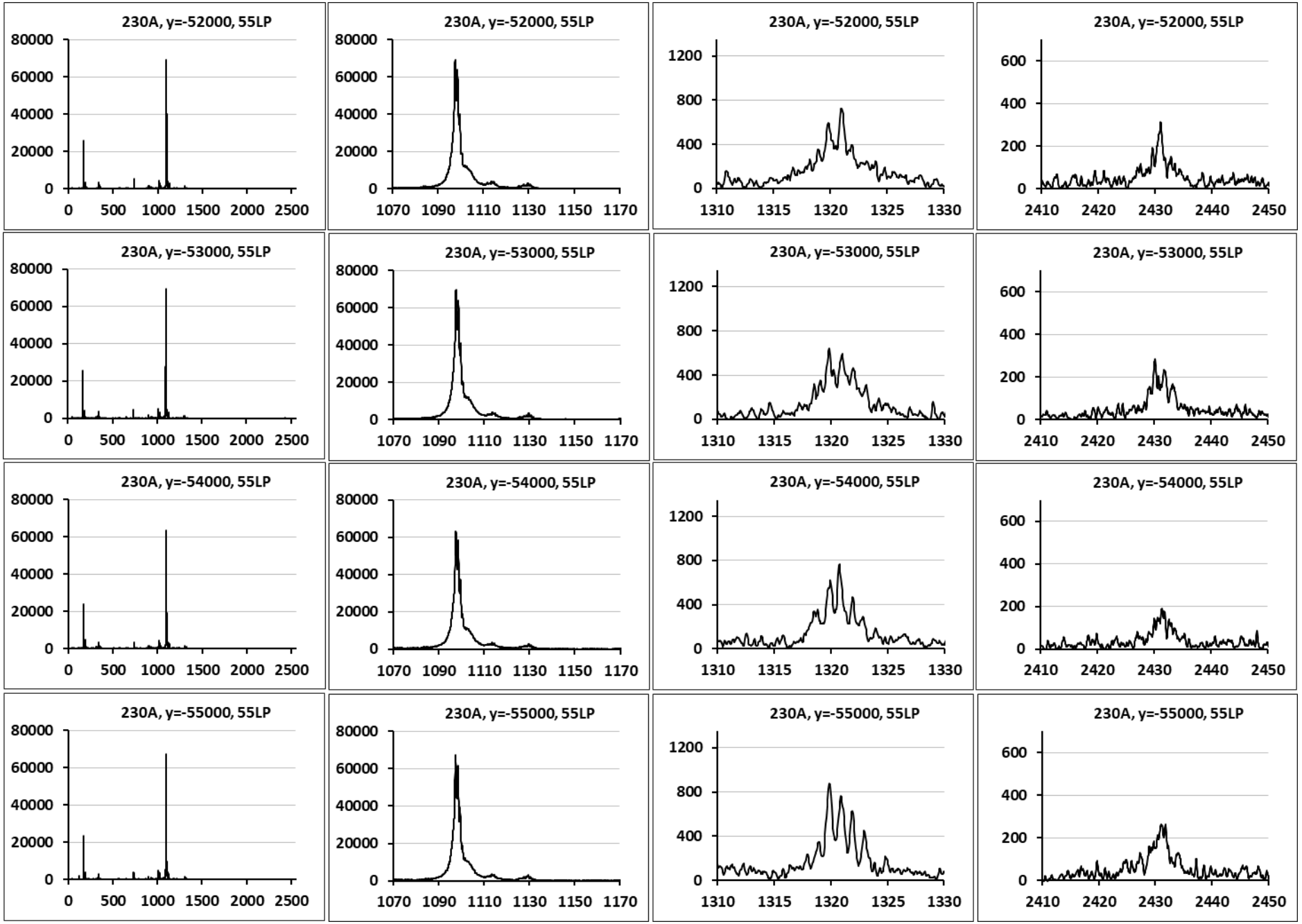
4 μM $\mathbf{1}^{OH}$ + 6 μM **2** 20 mM DTBP, 40 mM EDC, 40 mM DMAP

0-2.5 kD	1.07-1.17kD	1.31-1.33 kD	2.41-2.45 kD



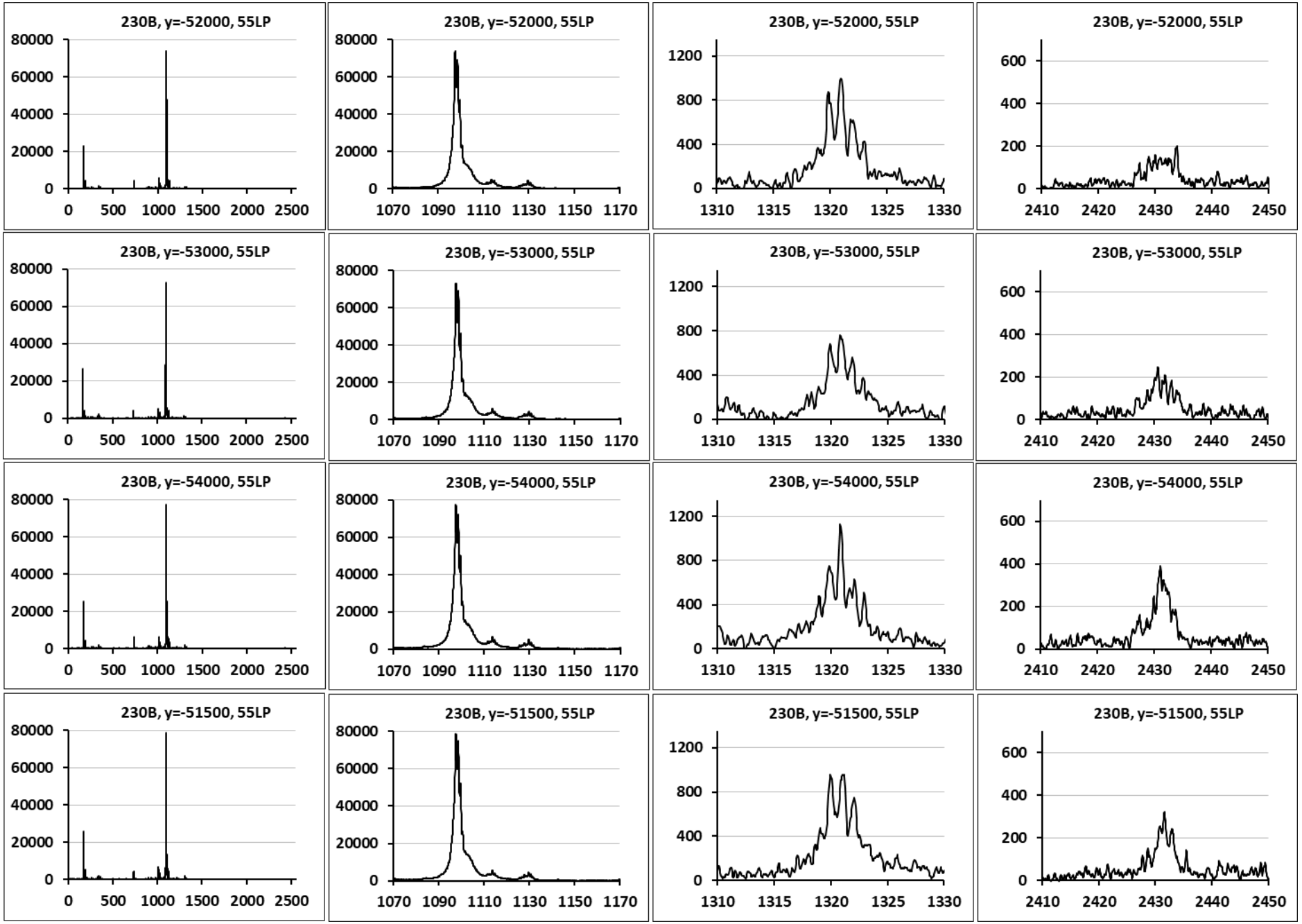
4 μM **1**^{OH} + 6 μM **2** 20 mM DTBP, 40 mM EDC, 40 mM DMAP

0-2.5 kD	1.07-1.17kD	1.31-1.33 kD	2.41-2.45 kD



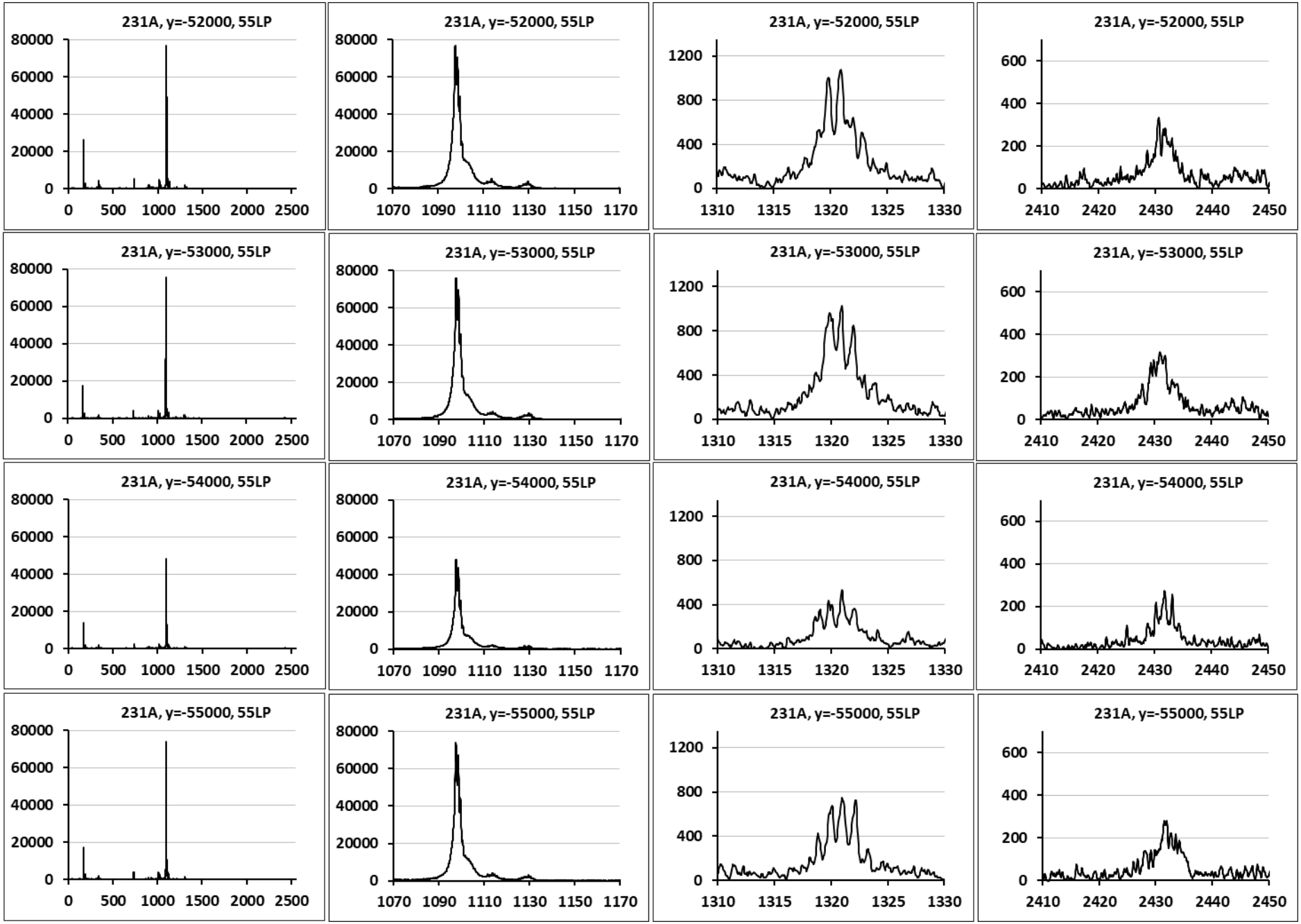
4 µM $\mathbf{1}^{OH}$ + 6 µM **2** 20 mM DTBP, 40 mM EDC, 40 mM DMAP

0-2.5 kD	1.07-1.17kD	1.31-1.33 kD	2.41-2.45 kD



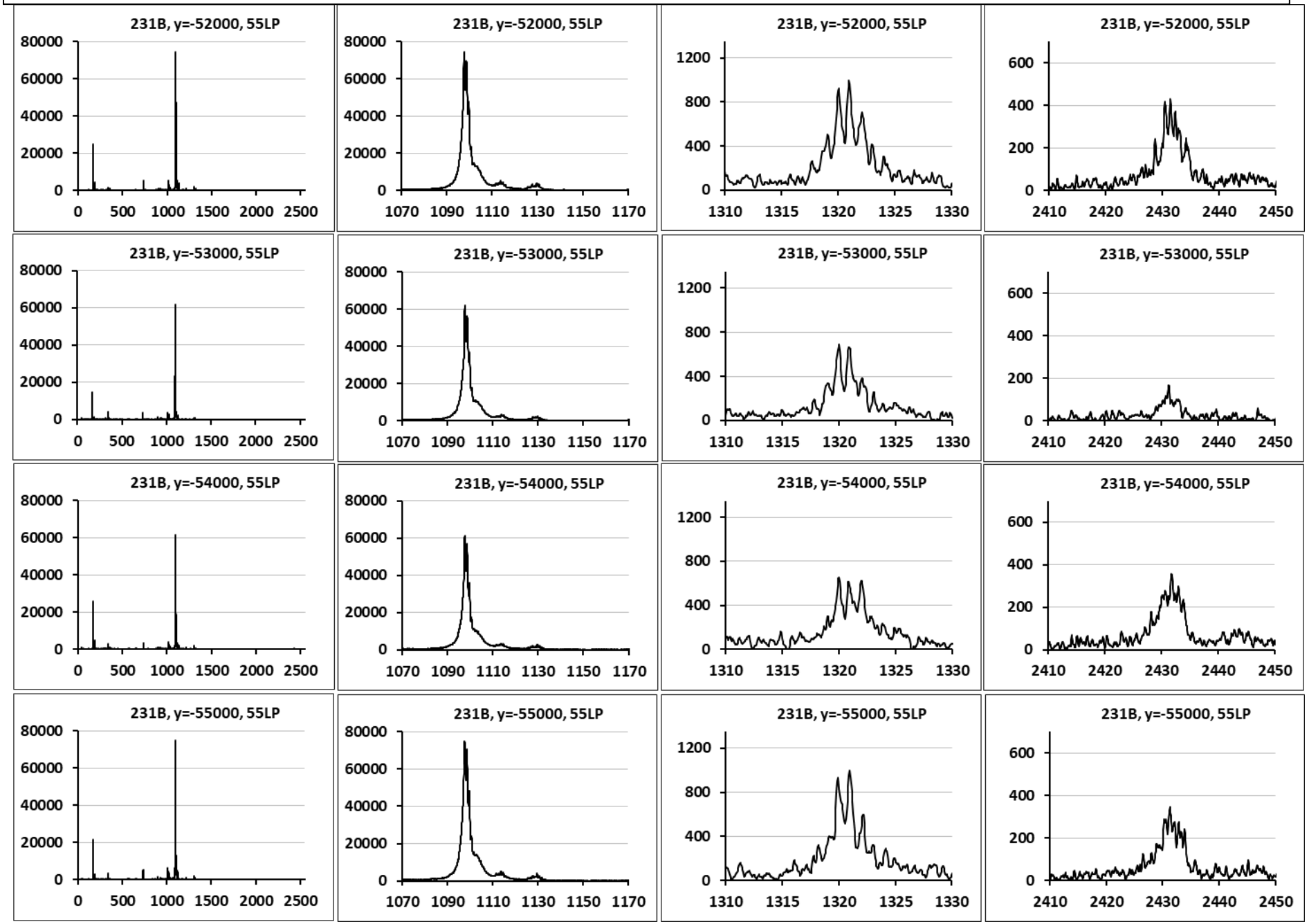
4 μM **1**^{OH} + 6 μM **2** 20 mM DTBP, 40 mM EDC, 40 mM DMAP

0-2.5 kD	1.07-1.17kD	1.31-1.33 kD	2.41-2.45 kD



4 µM **1**^{OH} + 6 µM **2** 20 mM DTBP, 40 mM EDC, 40 mM DMAP

0-2.5 kD	1.07-1.17kD	1.31-1.33 kD	2.41-2.45 kD

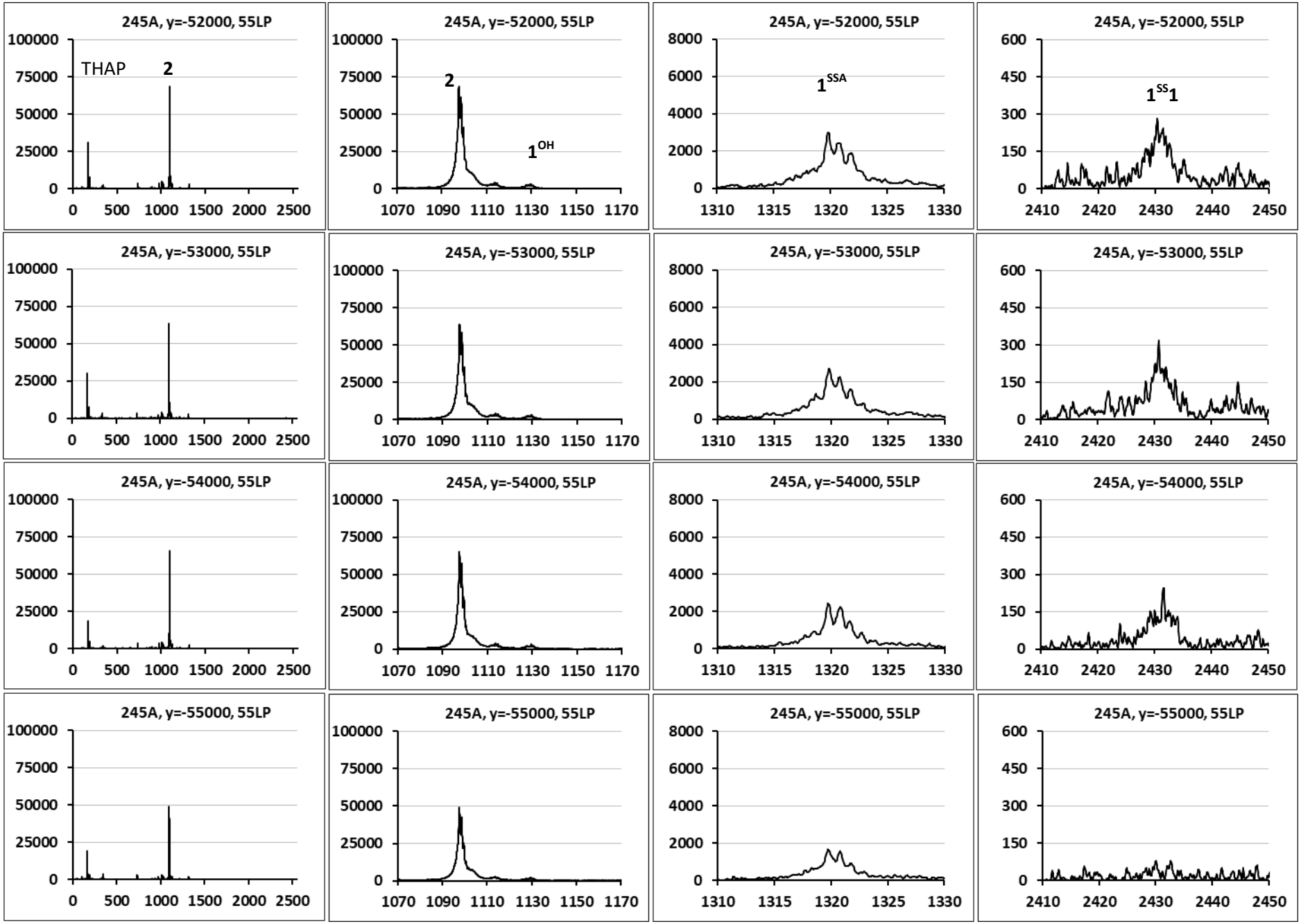


Summary: TOF-MS ML $\mathbf{1}^{OH}$, $\mathbf{1}^{SSA}$ and $\mathbf{1}^{SS}\mathbf{1}$ intensity statistics after esterification with 100 mM DTBP, 20 mM EDC, 40 mM DMAP. THAP thin film applied, LP = 55. (TOF-MS measured at 8 locations on each of 4 independently prepared samples.)

[$\mathbf{1}^{OH}$]	[**2**]	**# Locations**	$\mathbf{1}^{OH}$	$\mathbf{1}^{OH}$	$\mathbf{1}^{OH}$	$\mathbf{1}^{OH}$	$\mathbf{1}^{OH}$	$\mathbf{1}^{SSA}$	$\mathbf{1}^{SSA}$	$\mathbf{1}^{SSA}$	$\mathbf{1}^{SSA}$	$\mathbf{1}^{SSA}$	$\mathbf{1}^{SS}\mathbf{1}$	$\mathbf{1}^{SS}\mathbf{1}$	$\mathbf{1}^{SS}\mathbf{1}$	$\mathbf{1}^{SS}\mathbf{1}$	$\mathbf{1}^{SS}\mathbf{1}$
µM	µM	(file names)	**Max**	**Min**	**Avg**	**σ**	**σ/Avg**	**Max**	**Min**	**Avg**	**σ**	**σ/Avg**	**Max**	**Min**	**Avg**	**σ**	**σ/Avg**
4	6	8 (245A,B)	3823	1414	2656	721	27%	5593	1653	3295	1288	39%	672	80	324	167	52%
4	6	8 (246A,B)	4923	2064	2896	977	34%	7527	1943	4382	1719	39%	839	185	388	201	52%
4	6	8 (247A,B)	3384	1842	2674	488	18%	3613	2129	2625	478	18%	353	222	299	48	16%
4	6	8 (248A,B)	3251	1842	2390	443	19%	3142	1664	2298	516	22%	519	104	272	129	48%
4	6	32	4923	1414	**2654**	**681**	**26%**	7527	1653	**3150**	**1345**	**43%**	839	80	**321**	**147**	**46%**

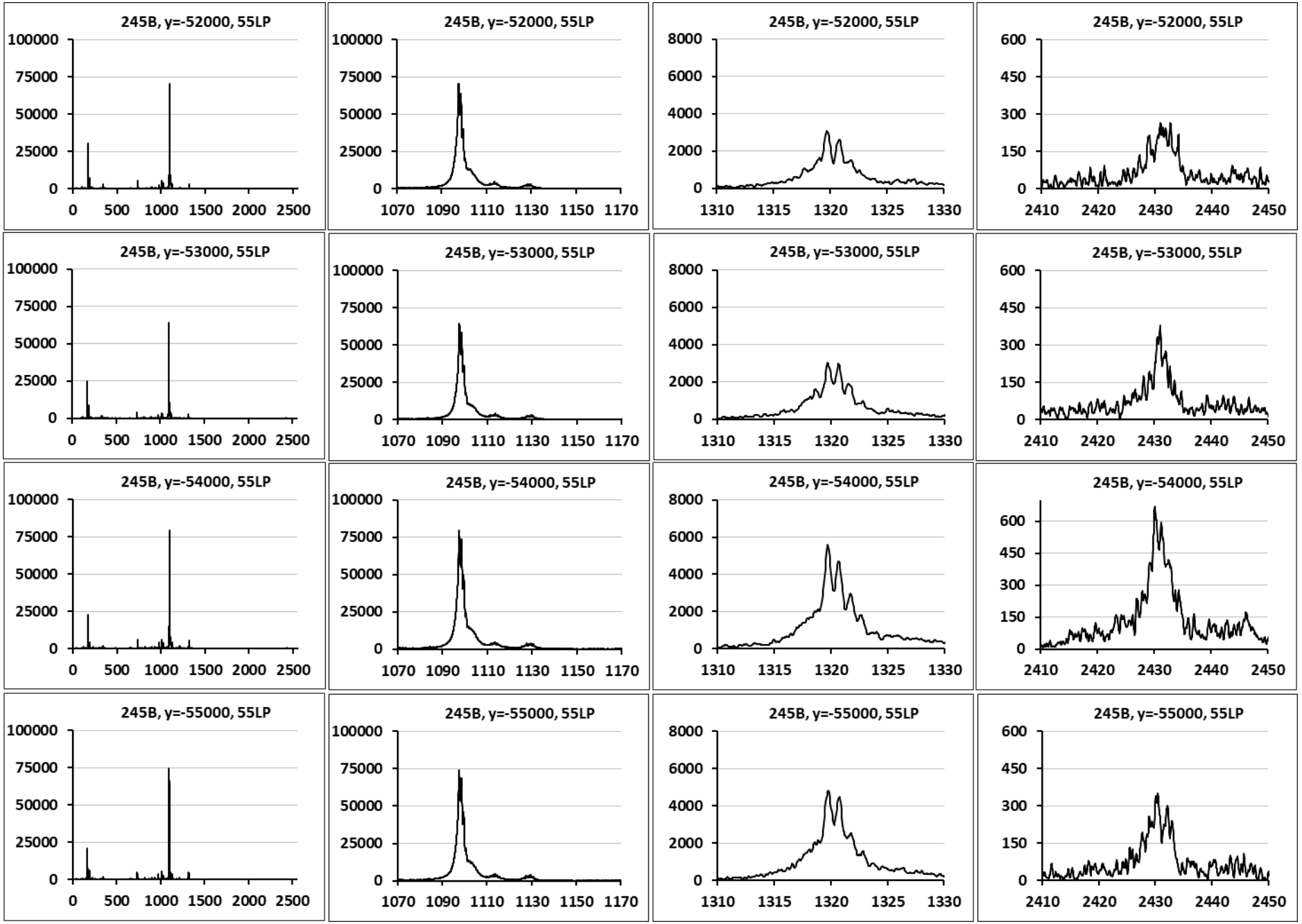
4 µM $\mathbf{1}^{OH}$ + 6 µM **2** 100 mM DTBP, 20 mM EDC, 40 mM DMAP

0-2.5 kD	1.07-1.17kD	1.31-1.33 kD	2.41-2.45 kD



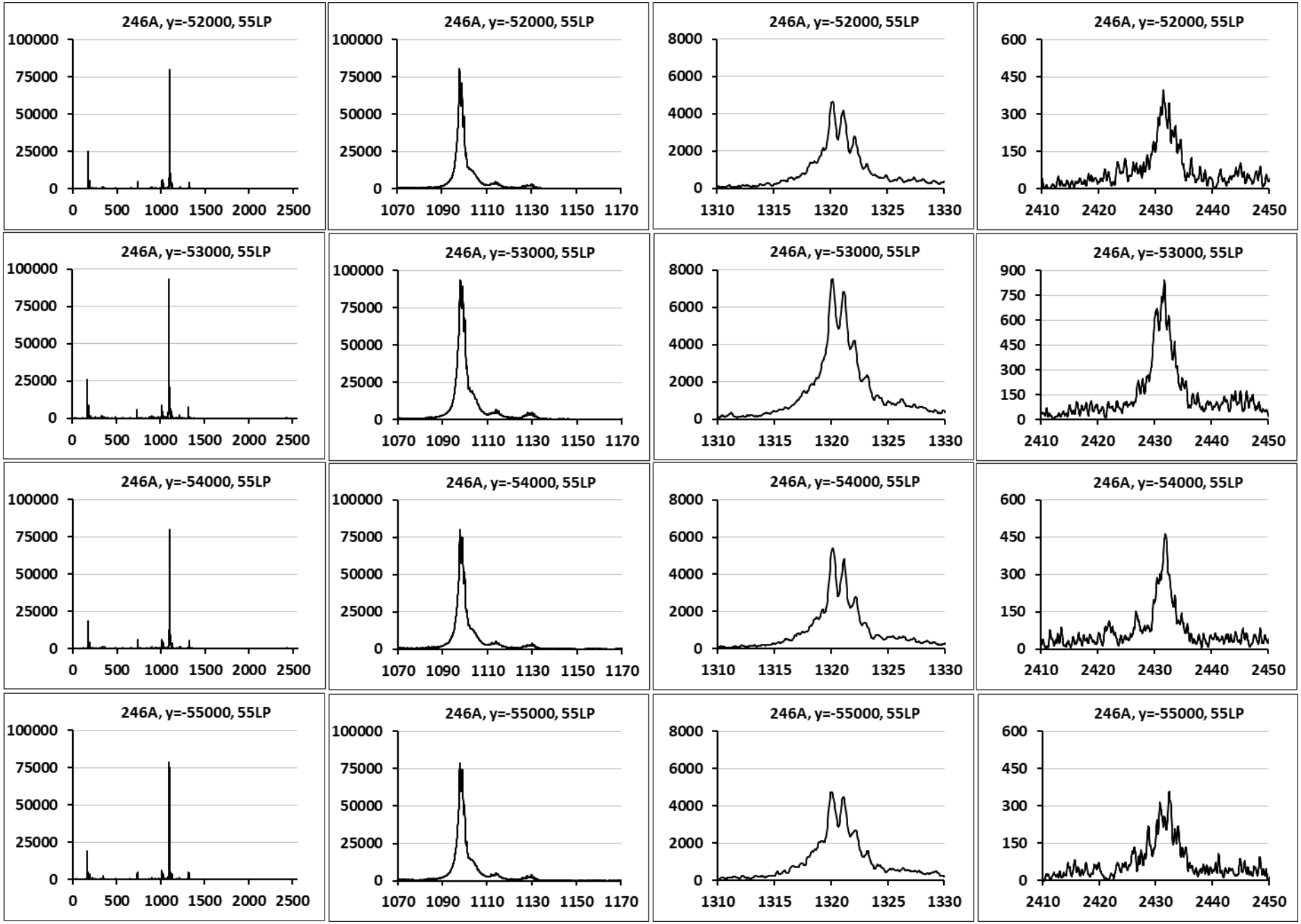
4 μM $\mathbf{1}^{OH}$ + 6 μM **2** 100 mM DTBP, 20 mM EDC, 40 mM DMAP

0-2.5 kD	1.07-1.17kD	1.31-1.33 kD	2.41-2.45 kD



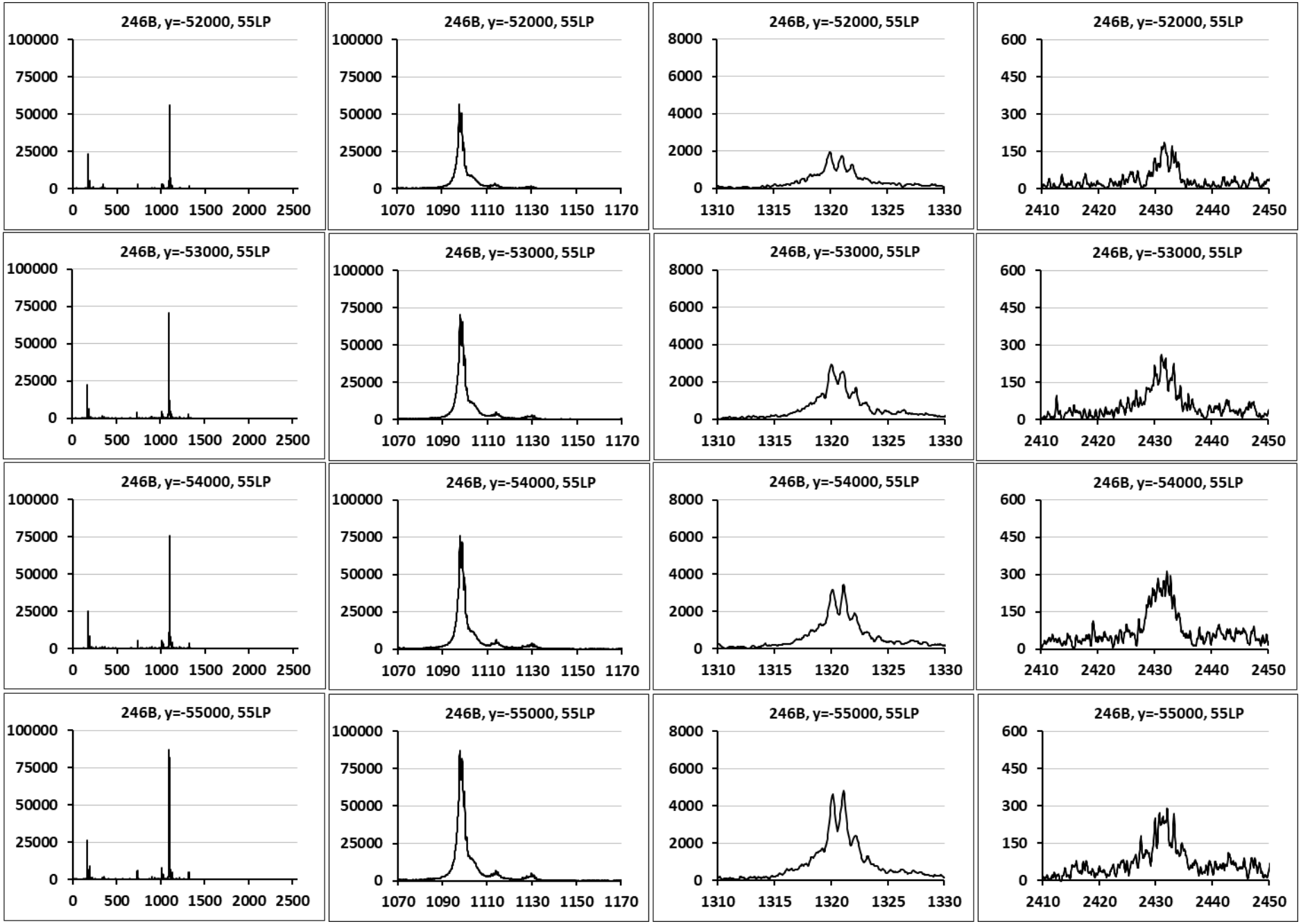
4 μM **1**^{OH} + 6 μM **2** 100 mM DTBP, 20 mM EDC, 40 mM DMAP

0-2.5 kD	1.07-1.17kD	1.31-1.33 kD	2.41-2.45 kD



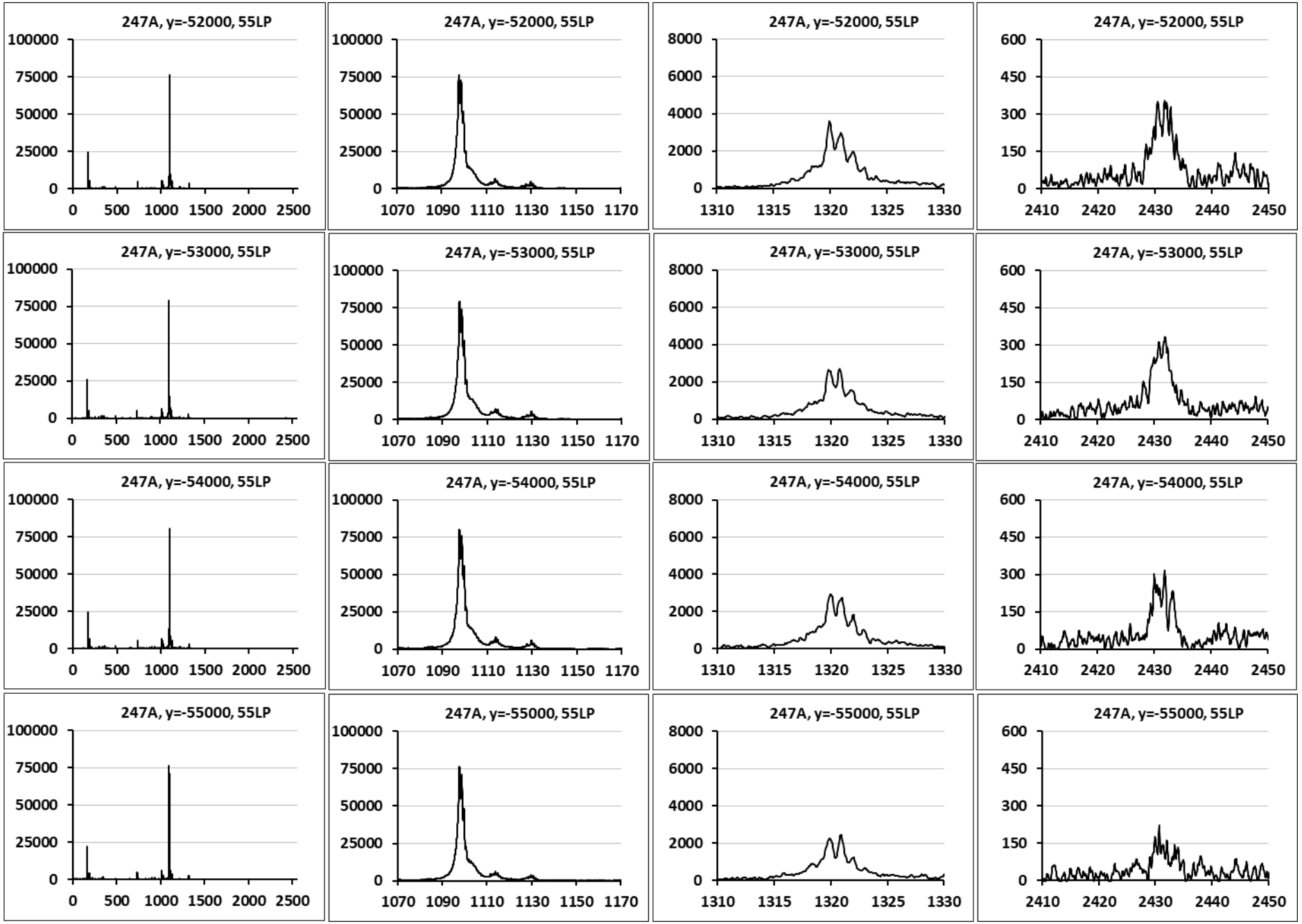
4 µM $\mathbf{1}^{OH}$ + 6 µM **2** 100 mM DTBP, 20 mM EDC, 40 mM DMAP

0-2.5 kD	1.07-1.17kD	1.31-1.33 kD	2.41-2.45 kD



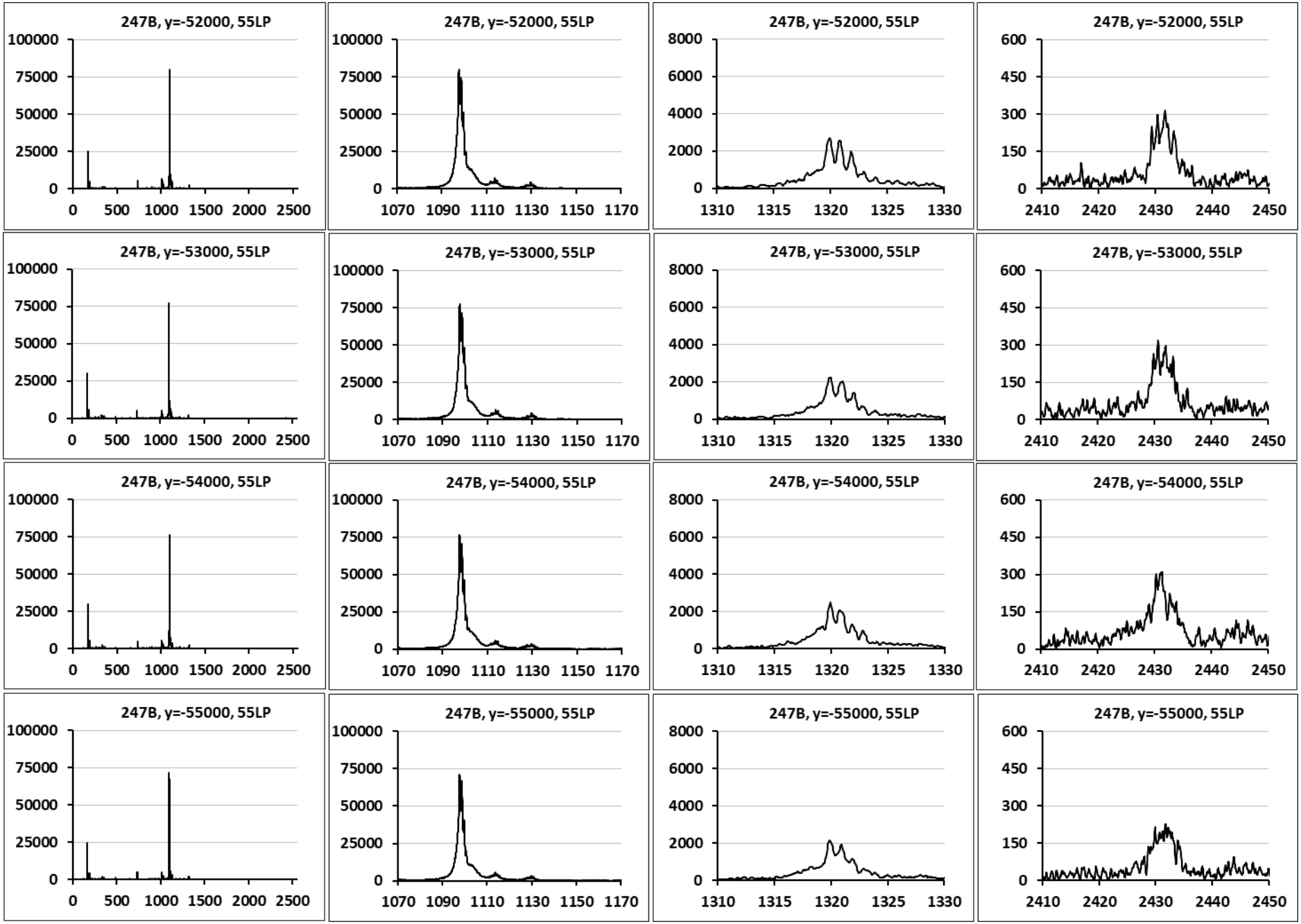
4 µM $\mathbf{1}^{OH}$ + 6 µM **2** 100 mM DTBP, 20 mM EDC, 40 mM DMAP

0-2.5 kD	1.07-1.17kD	1.31-1.33 kD	2.41-2.45 kD



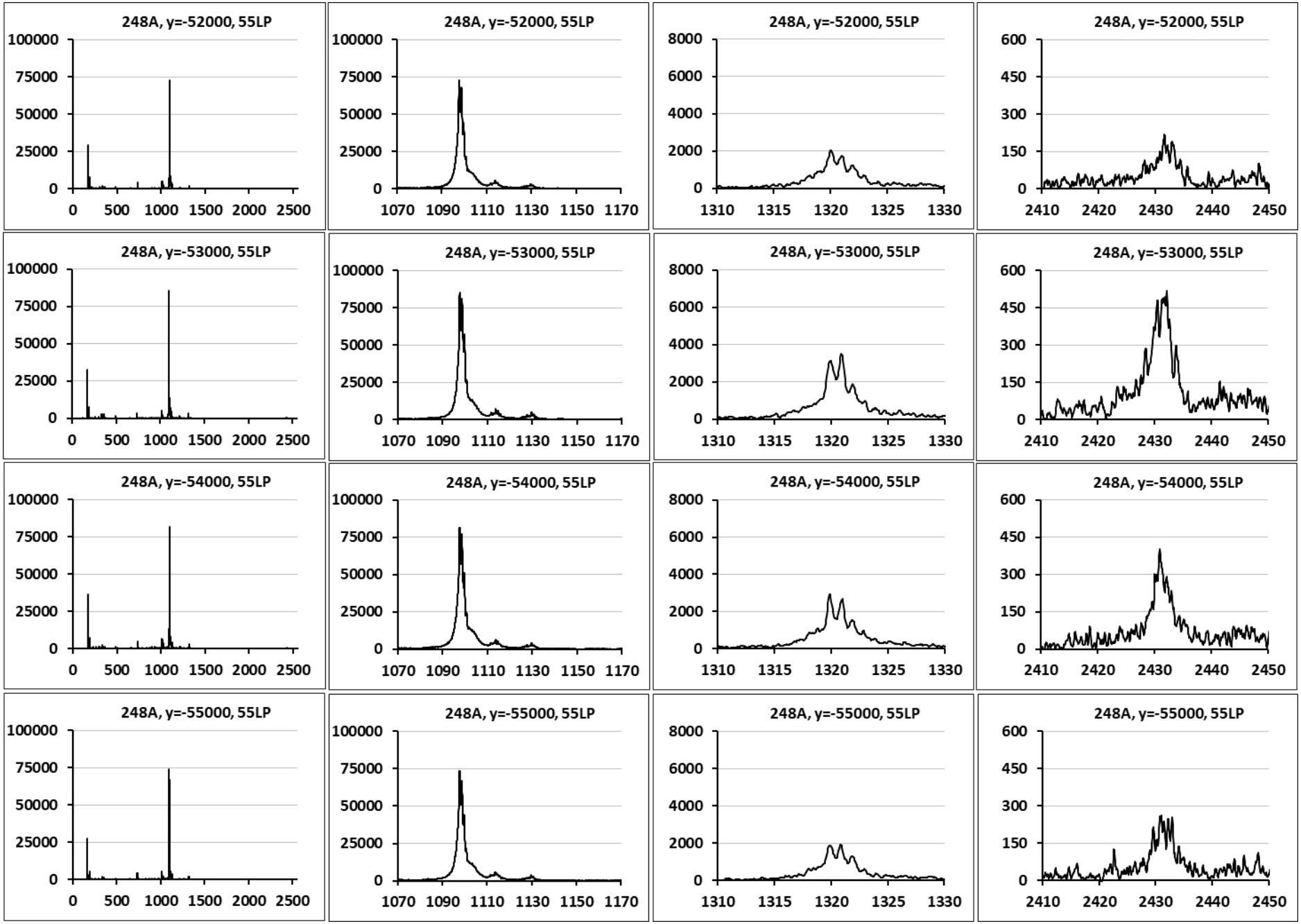
4 μM $\mathbf{1}^{OH}$ + 6 μM **2** 100 mM DTBP, 20 mM EDC, 40 mM DMAP

0-2.5 kD	1.07-1.17kD	1.31-1.33 kD	2.41-2.45 kD



4 μM **1**^{OH} + 6 μM **2** 100 mM DTBP, 20 mM EDC, 40 mM DMAP

0-2.5 kD	1.07-1.17kD	1.31-1.33 kD	2.41-2.45 kD



4 µM **1**^{OH} + 6 µM **2** 100 mM DTBP, 20 mM EDC, 40 mM DMAP

0-2.5 kD	1.07-1.17kD	1.31-1.33 kD	2.41-2.45 kD

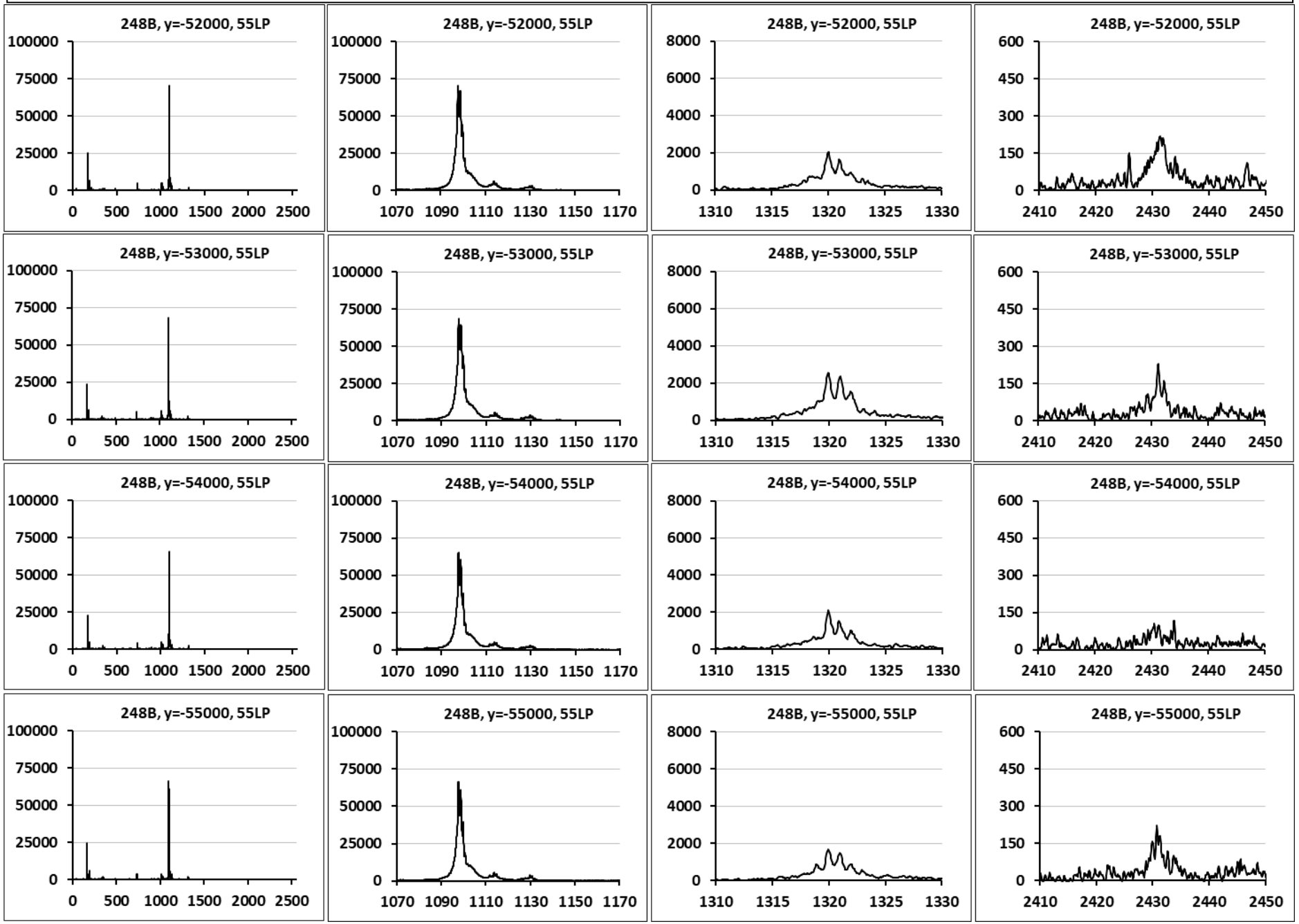


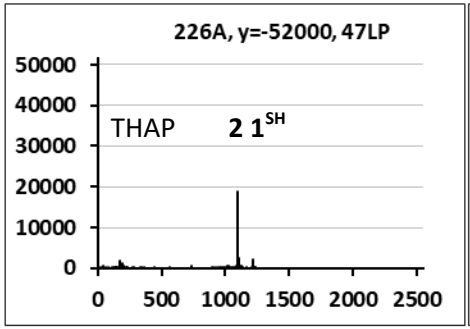
Fig. S5. Summaries and TOF-MS spectra (full range and expansions) of locations sampled to determine relative $\mathbf{1}^{SH}$ and **2** intensities following reaction of ($\mathbf{1}^{OH}\mathbf{1}^{OH}\mathbf{22}$) ML with DTBP followed by reaction with DTT reducing agent. Data collected at LP = 47 after applying a THAP thin film.

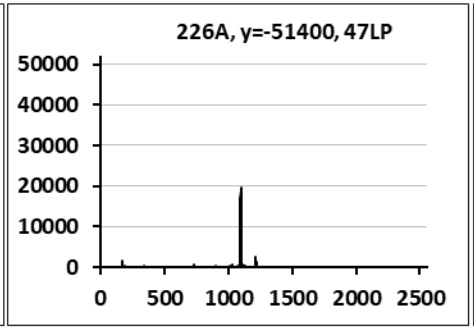
Summary: TOF-MS ML $\mathbf{1}^{SH}$ / **2** intensity and intensity ratio statistics after esterification with 20 mM DTBP, 40 mM EDC, 40 mM DMAP followed by DTT reduction. THAP thin film applied, LP = 47. (TOF-MS measured at 14 locations on each of 4 independently prepared samples.)

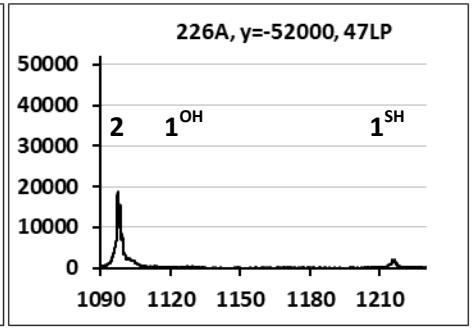
[1^{OH}]	[**2**]	**# Locations**	$\mathbf{1}^{SH}$ **/ 2**	$\mathbf{1}^{SH}$ **/ 2**	$\mathbf{1}^{SH}$ **/ 2**	$\mathbf{1}^{SH}$ **/ 2**	$\mathbf{1}^{SH}$ **/ 2**	$\mathbf{1}^{SH}$	$\mathbf{1}^{SH}$	$\mathbf{1}^{SH}$	$\mathbf{1}^{SH}$	$\mathbf{1}^{SH}$	**2**	**2**	**2**	**2**	**2**
µM	µM	(file names)	**Max**	**Min**	**Avg**	**σ**	**σ/Avg**	**Max**	**Min**	**Avg**	**σ**	**σ/Avg**	**Max**	**Min**	**Avg**	**σ**	**σ/Avg**
4	2	14 (226A,B)	0.15	0.11	0.12	0.01	9%	3356	1652	2620	502	19%	26908	14533	21024	3149	15%
4	2	14 (227A,B)	0.12	0.07	0.10	0.01	13%	5381	2169	3899	987	25%	51887	26301	38732	7504	19%
4	2	14 (228A,B)	0.14	0.09	0.11	0.01	13%	5998	2706	4086	958	23%	48922	27151	35552	6540	18%
4	2	14 (229A,B)	0.13	0.10	0.12	0.01	7%	6135	3199	4771	827	17%	49019	26493	41277	6245	15%
4	2	All	0.15	0.07	**0.11**	**0.01**	**13%**	6135	1652	**3844**	**1133**	**29%**	51887	14533	**34146**	**9880**	**29%**

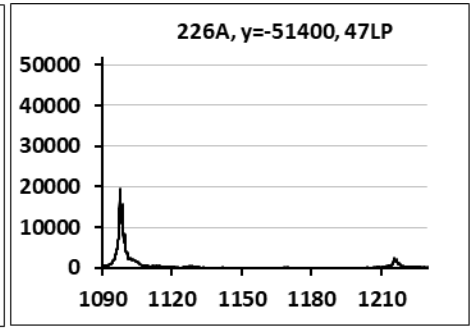
4 µM **1**^{OH} + 6 µM **2** 20 mM DTBP, 40 mM EDC, 40 mM DMAP followed by DTT reduction

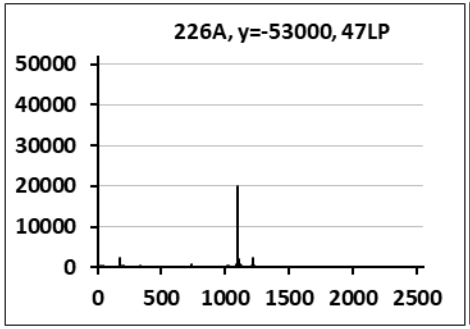
0-2.5 kD 1.09-1.23kD

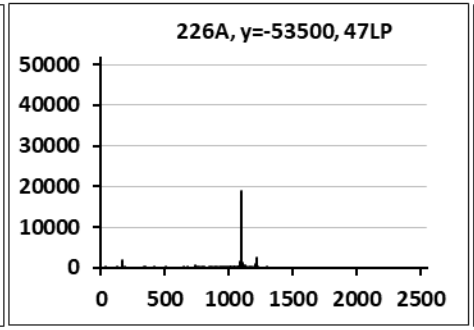


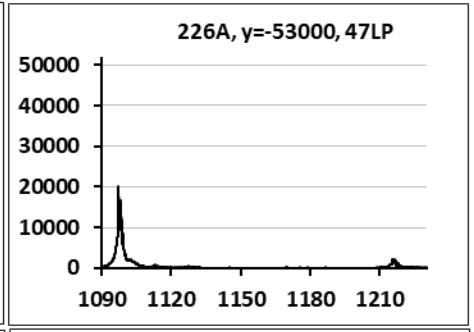


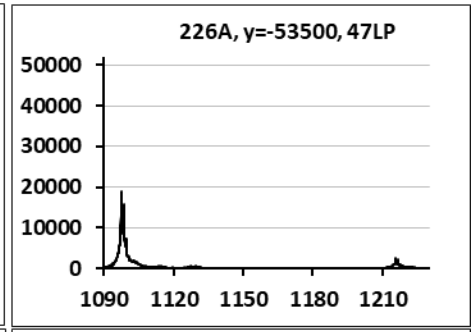


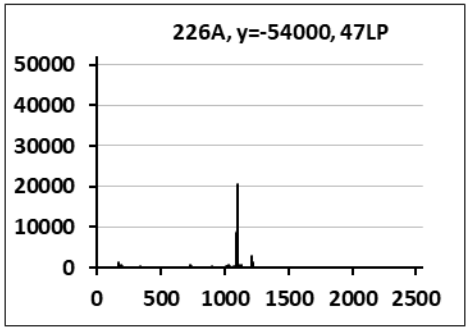


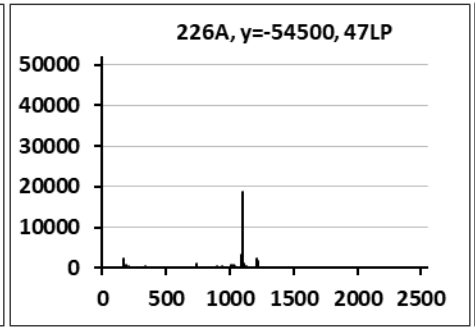


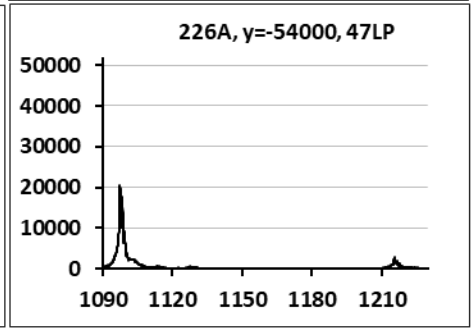


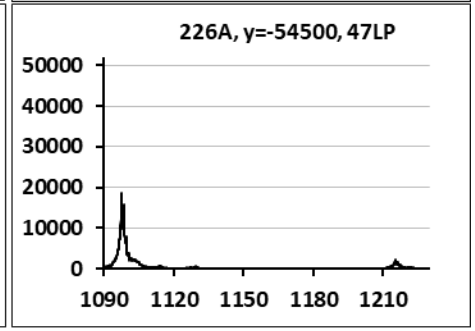


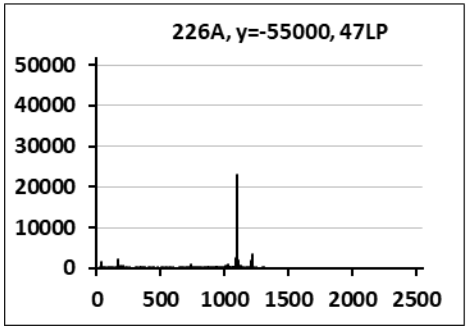


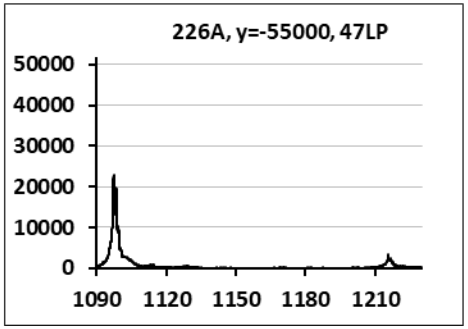




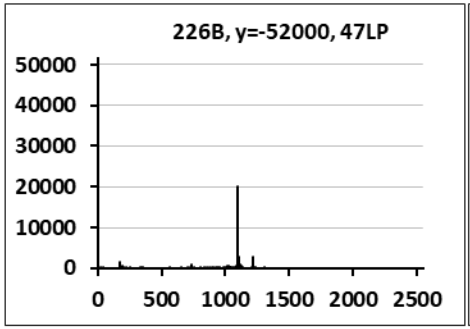


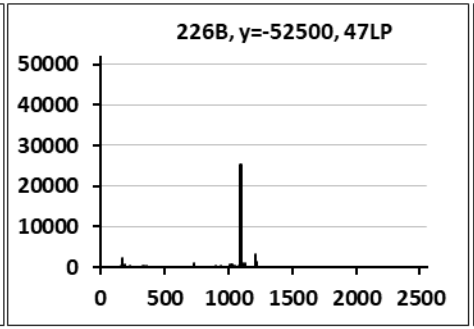


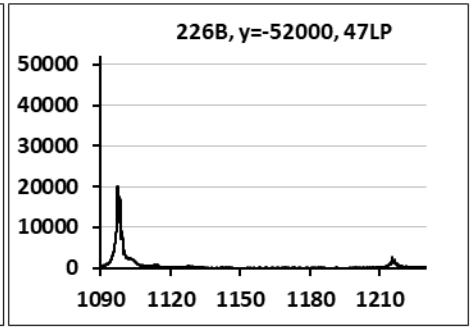


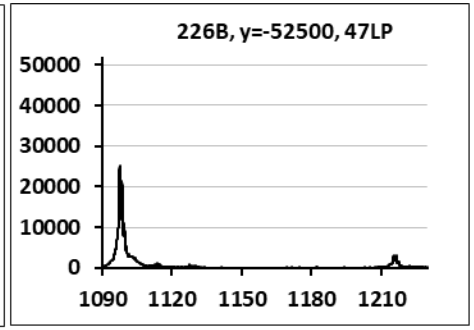


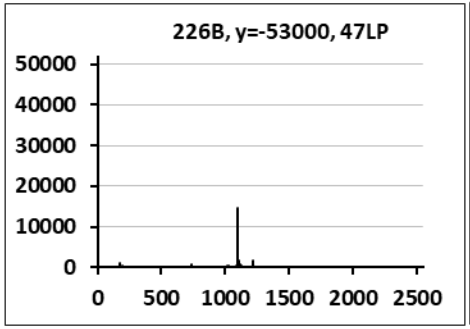
4 μM $\mathbf{1}^{OH}$ + 6 μM **2**	20 mM DTBP, 40 mM EDC, 40 mM DMAP followed by DTT reduction	
0-2.5 kD		1.09-1.23kD

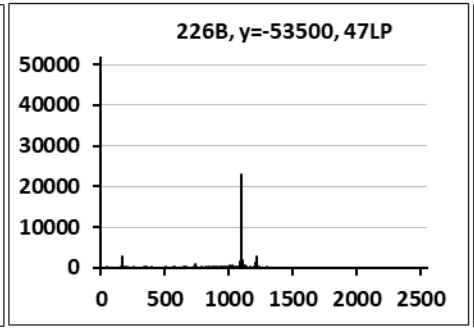


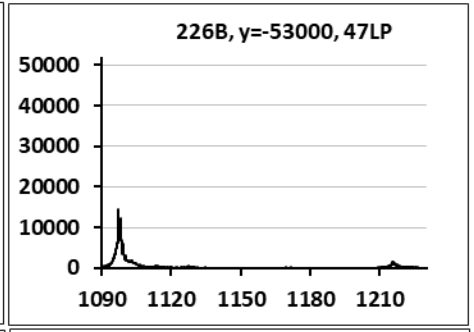


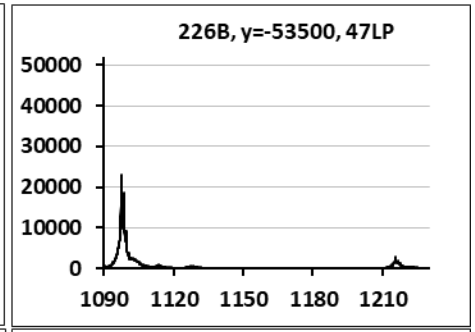


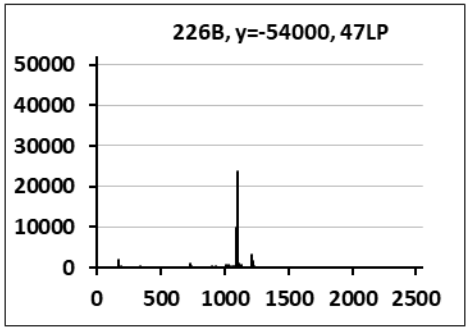


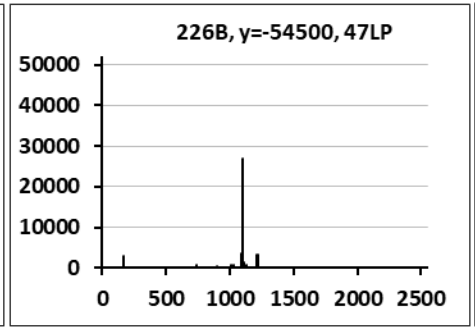


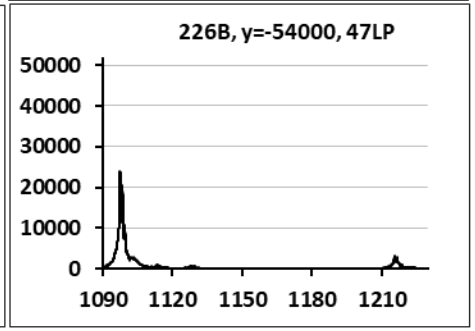


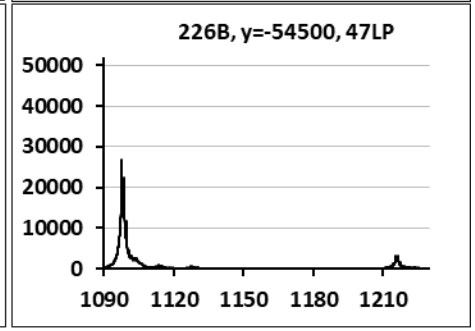


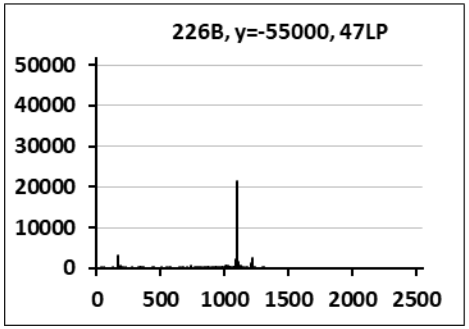


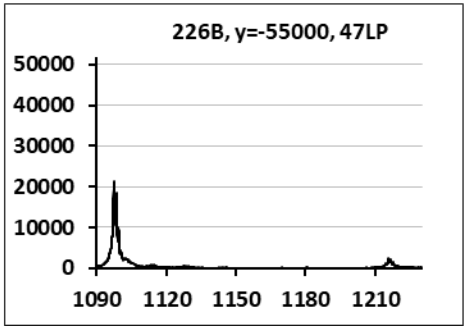




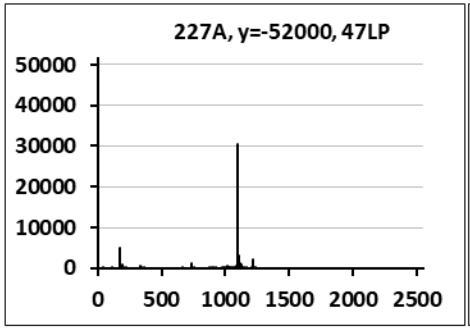


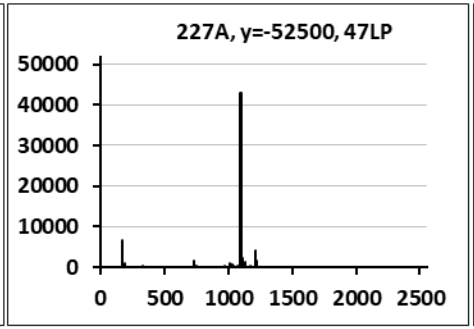


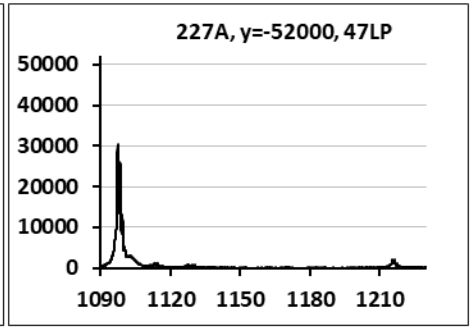


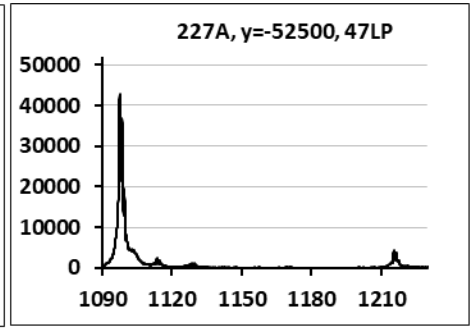


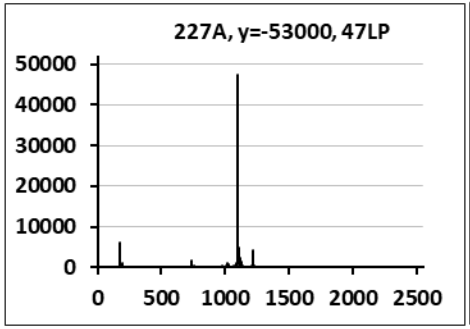
4 µM **1**^{OH} + 6 µM **2**	20 mM DTBP, 40 mM EDC, 40 mM DMAP followed by DTT reduction	
0-2.5 kD		1.09-1.23kD

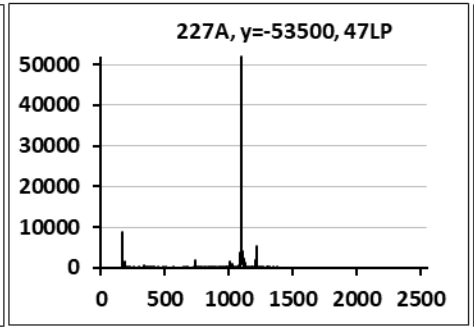


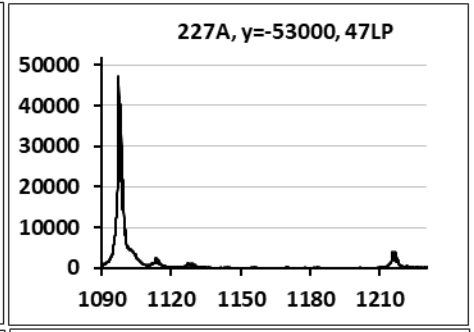


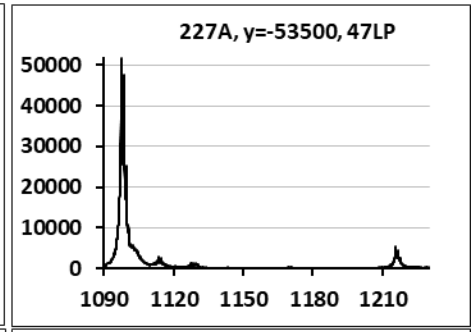


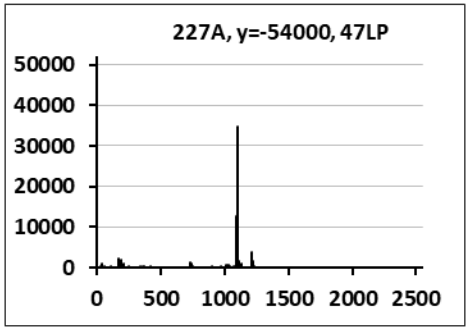


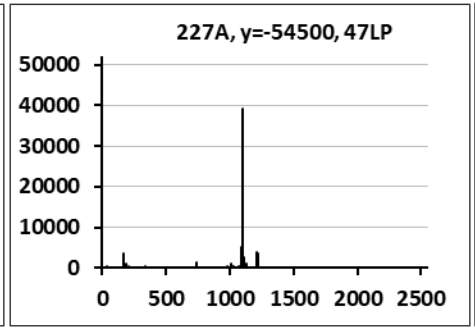


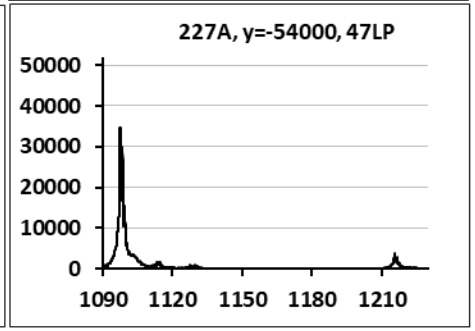


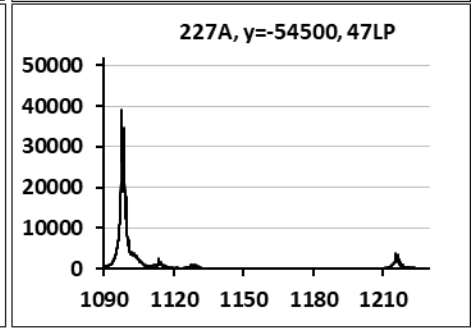


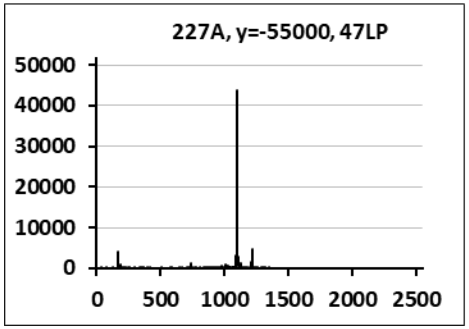


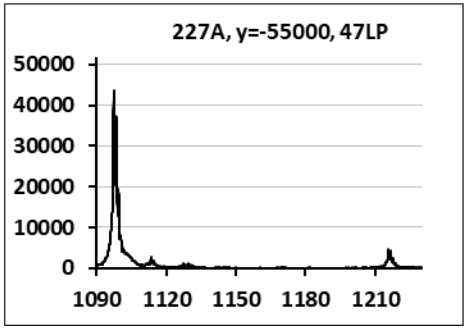












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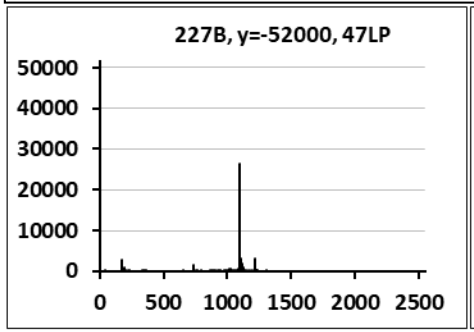
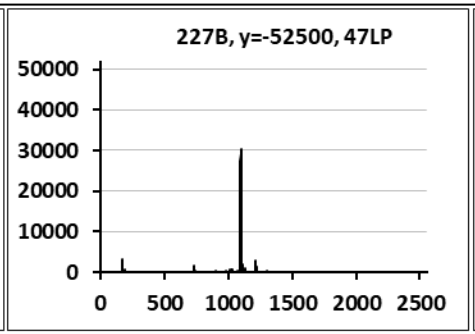
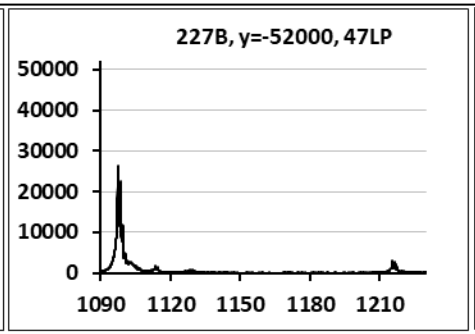
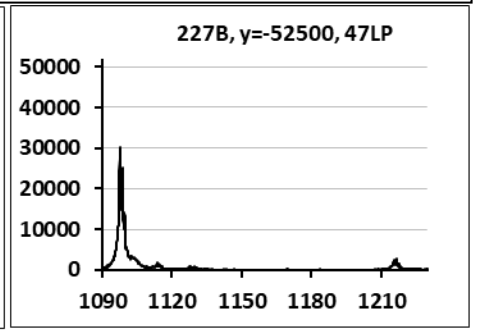
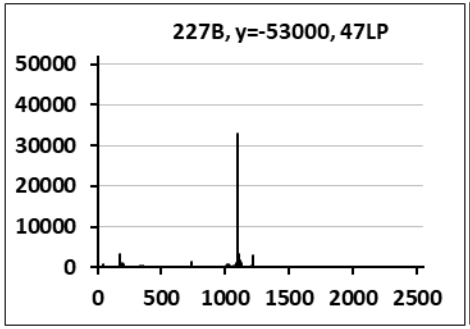
0-2.5 kD 1.09-1.23kD

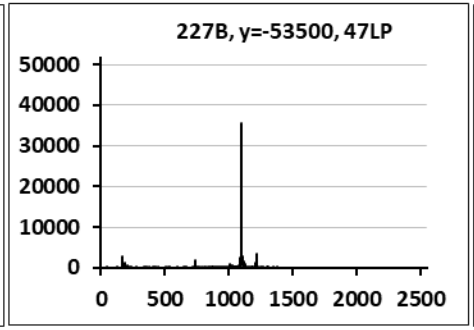


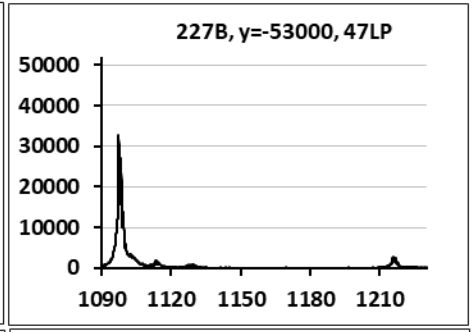


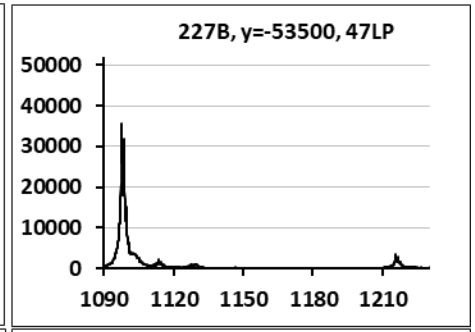


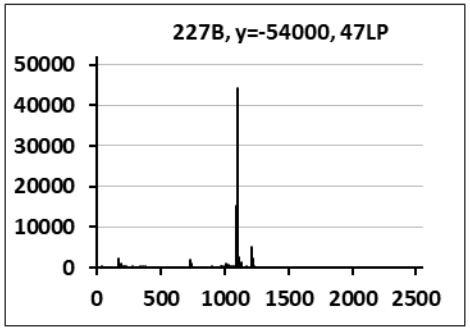


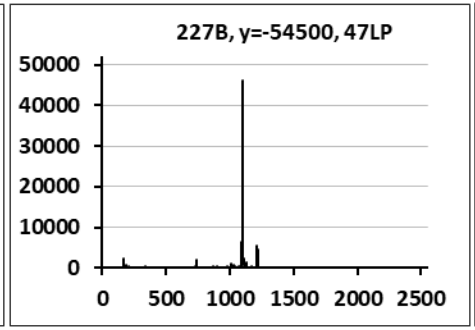


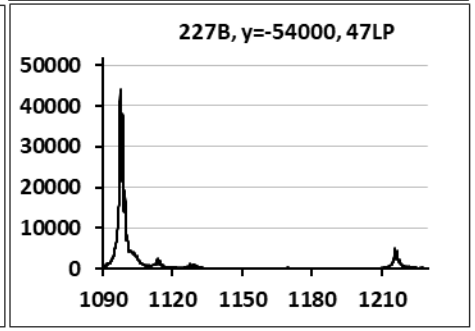


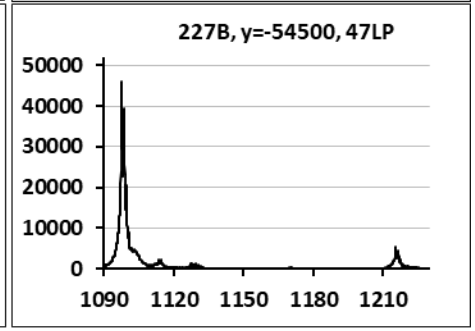


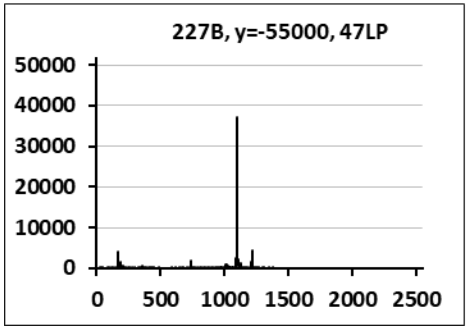


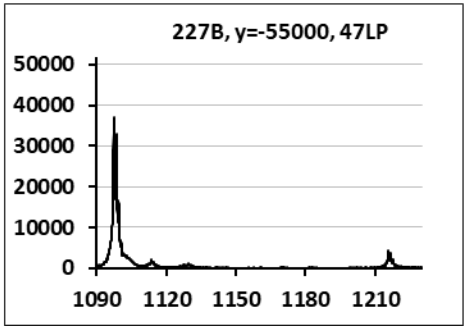












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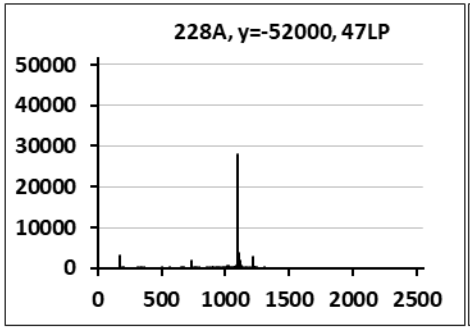
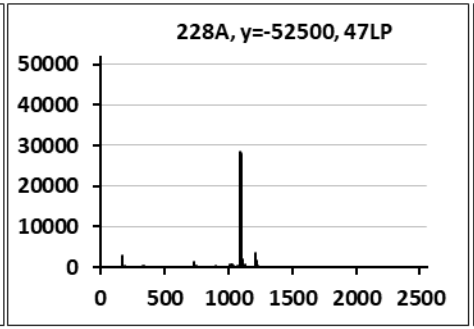
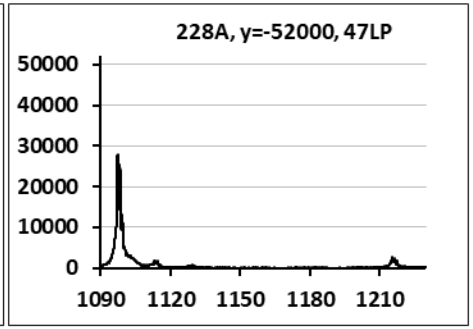
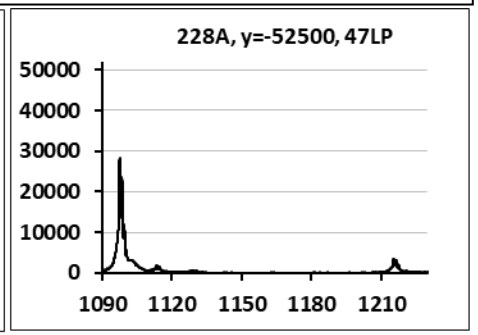
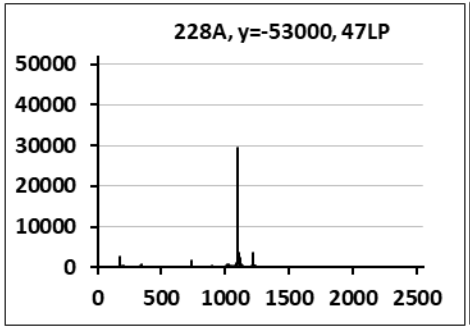
0-2.5 kD 1.09-1.23kD

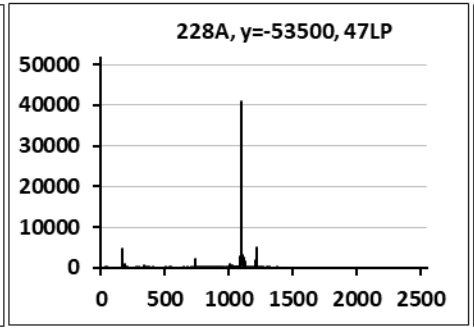


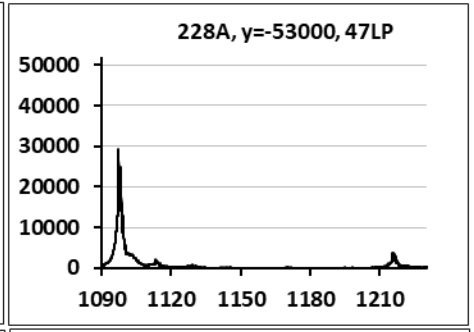


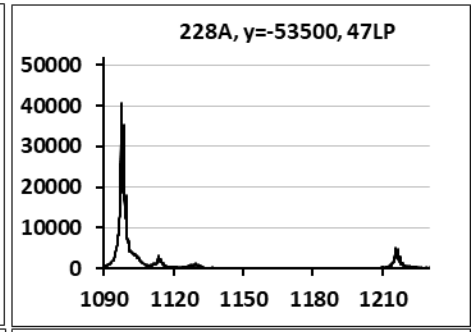


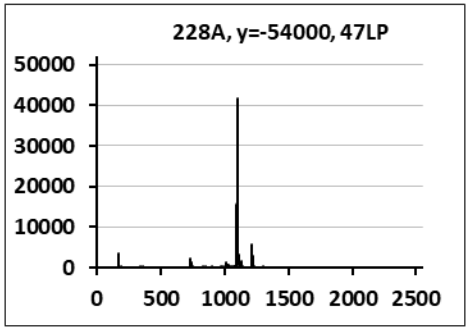


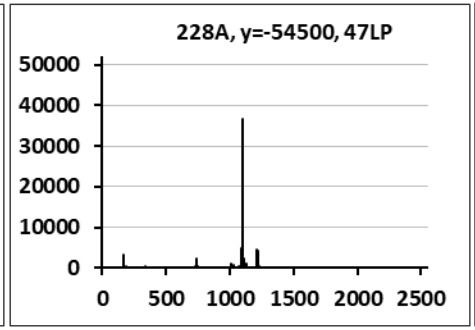


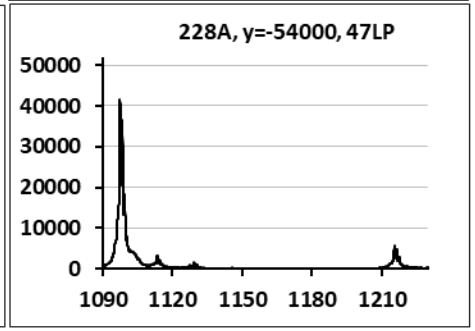


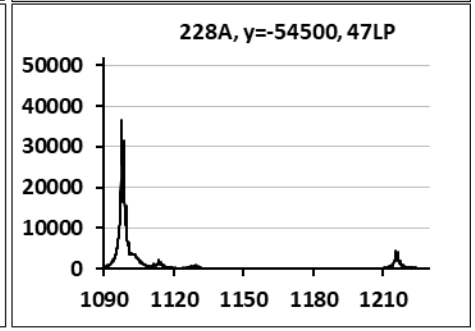


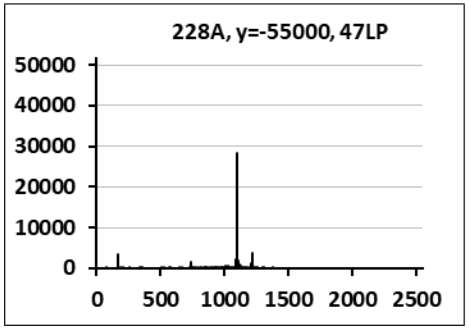


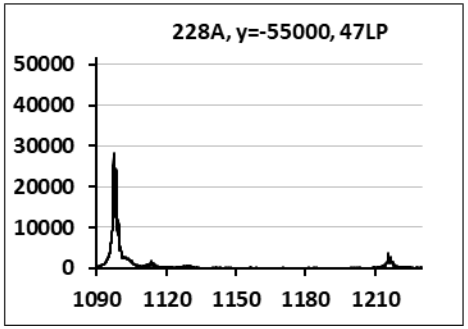












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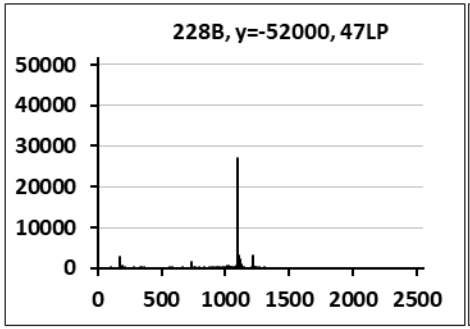

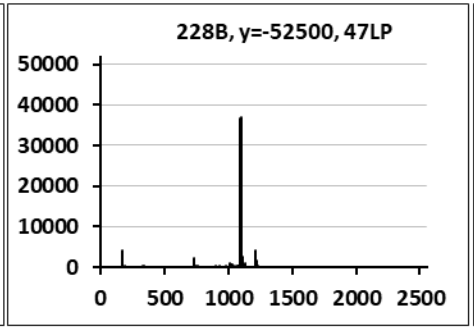

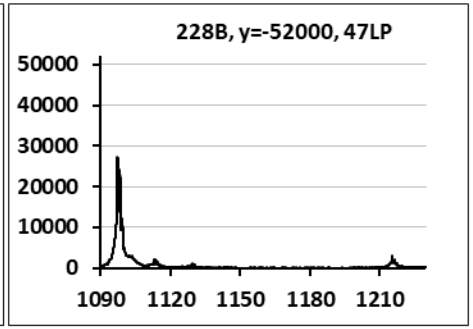

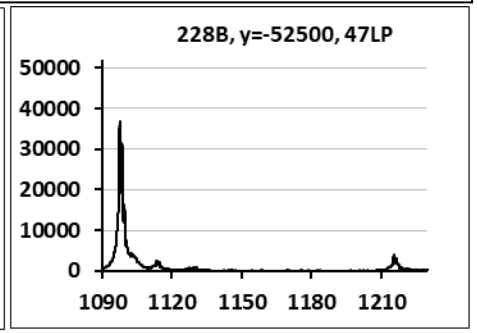

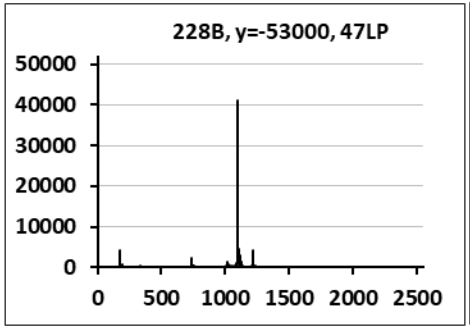

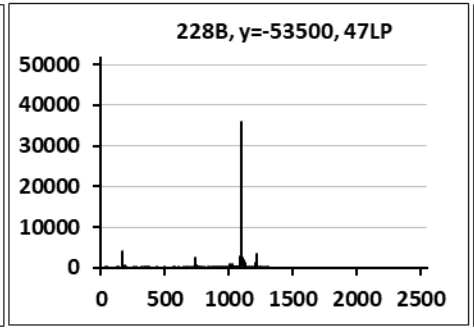

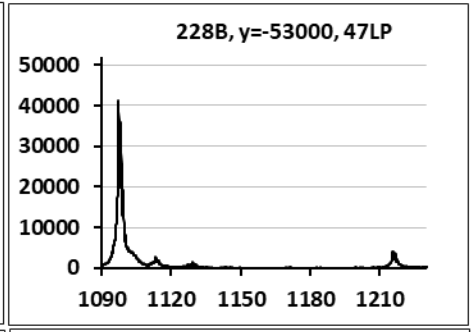

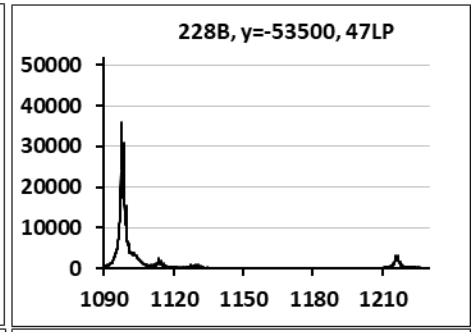

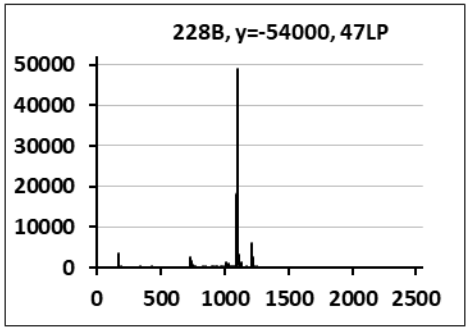

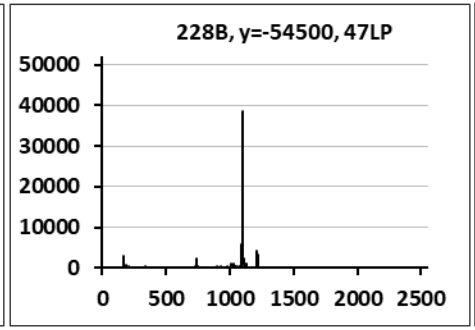

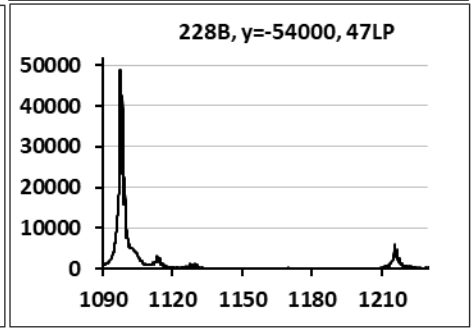

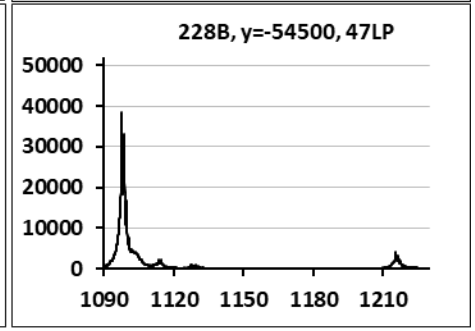

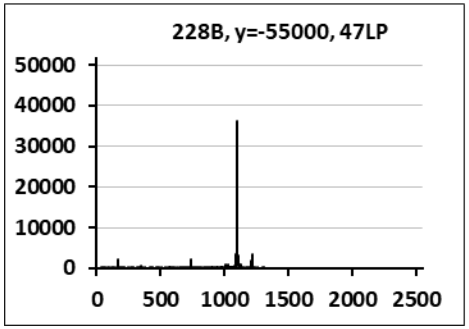

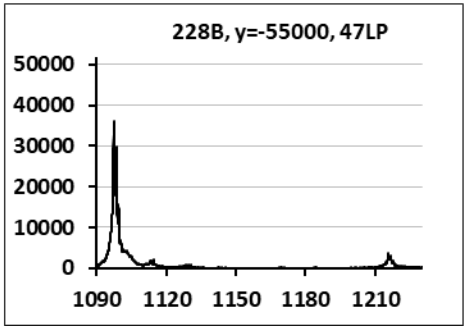

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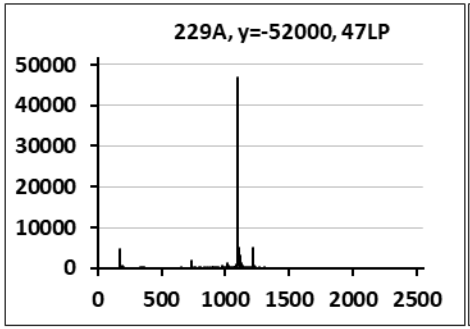
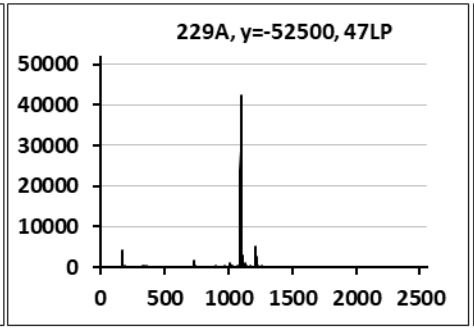
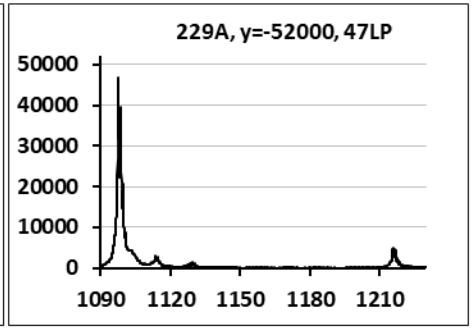
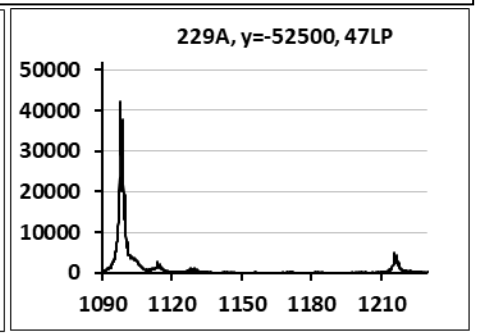
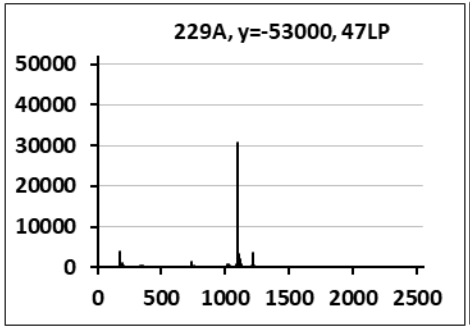
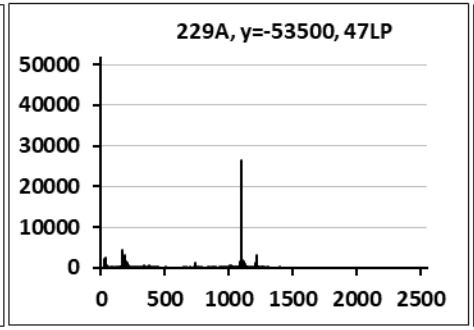


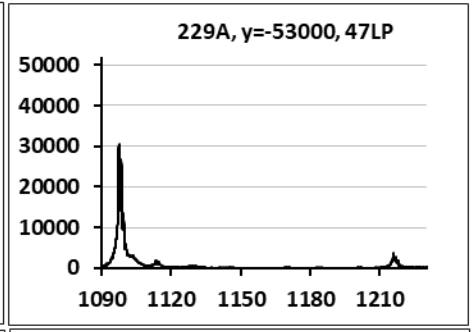


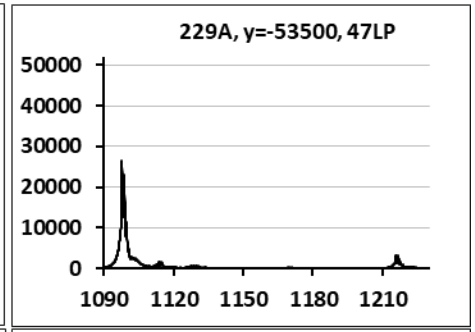


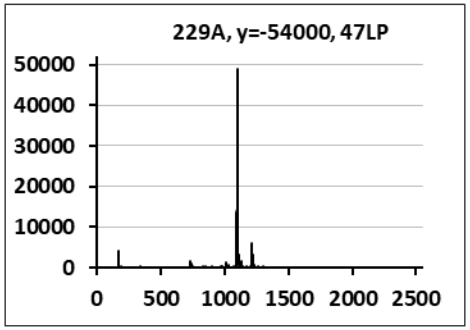


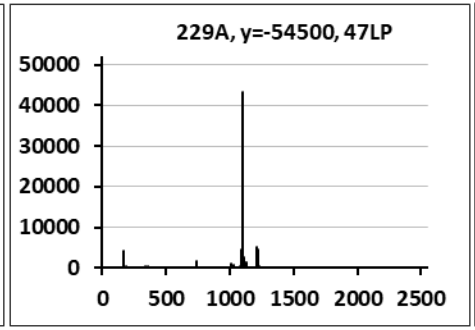


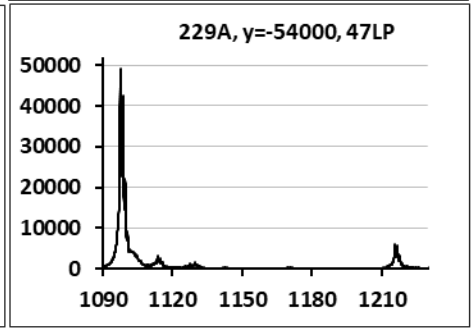


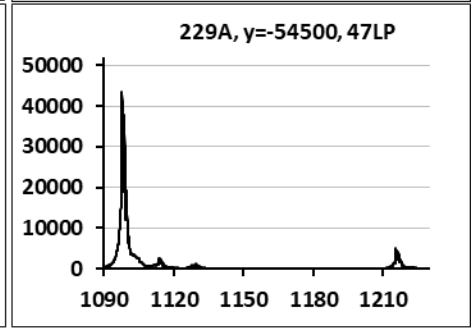


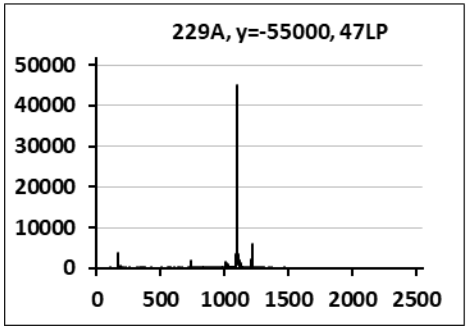


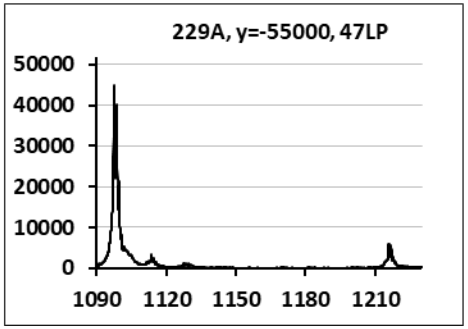






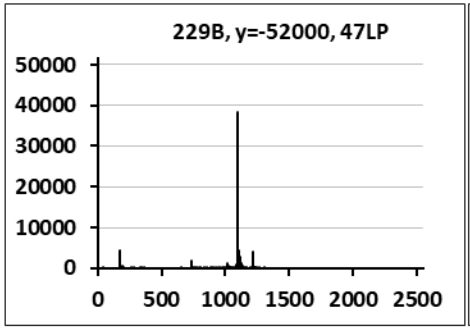


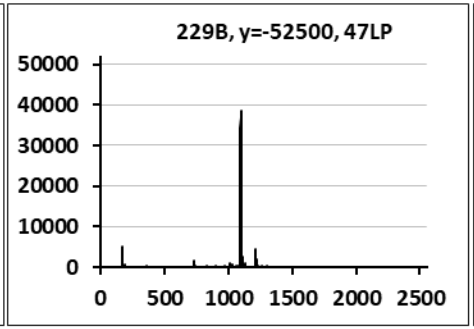


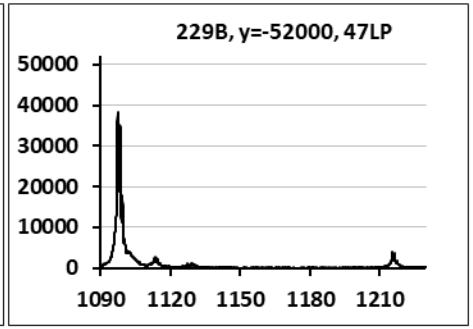


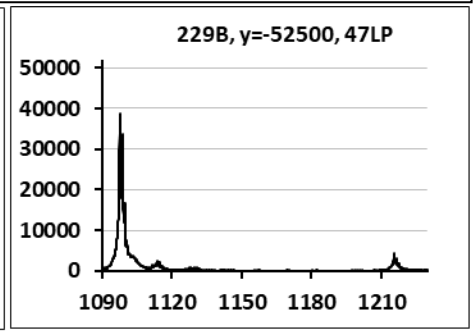
4 µM **1**^{OH} + 6 µM **2** 20 mM DTBP, 40 mM EDC, 40 mM DMAP followed by DTT reduction

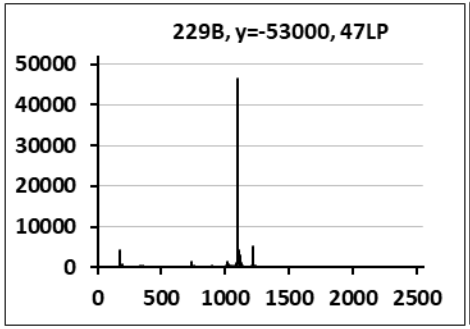
0-2.5 kD 1.09-1.23kD

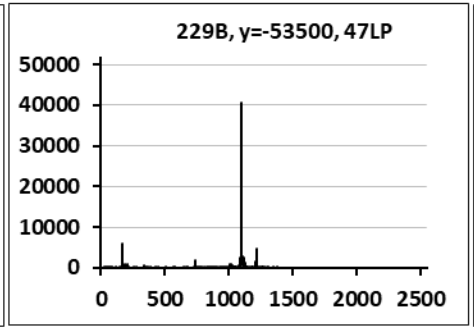


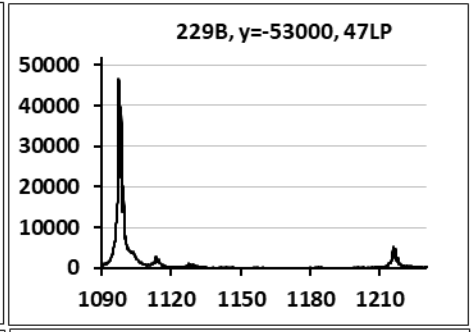


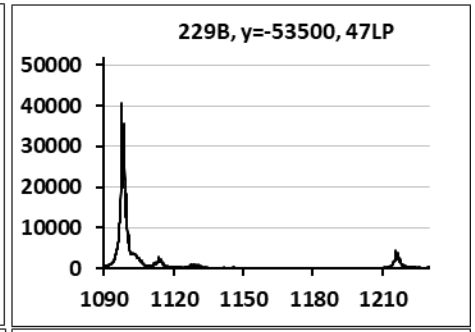


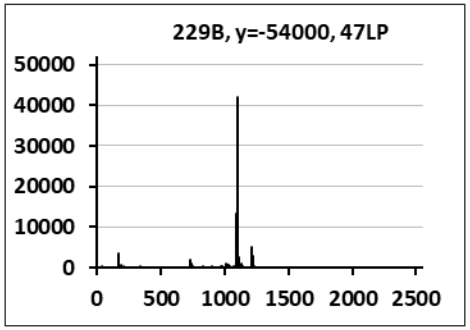


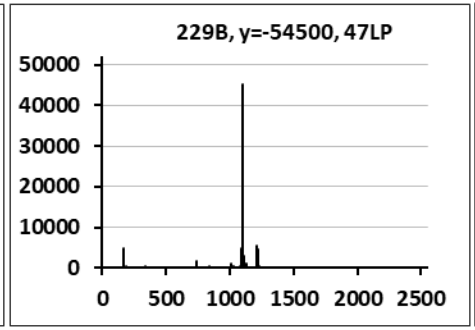


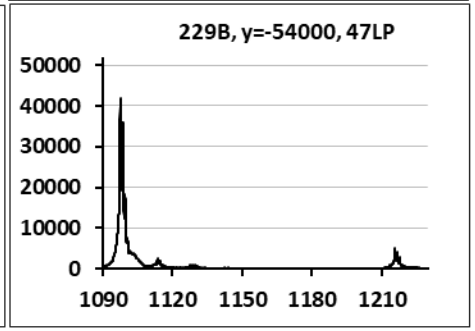


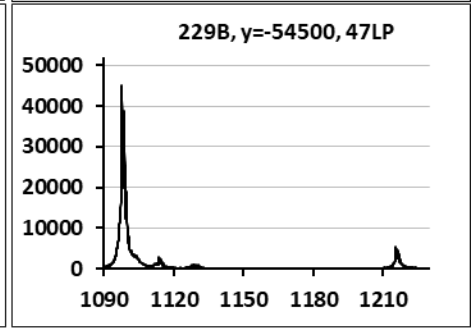


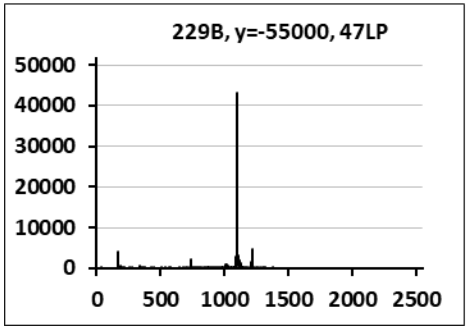












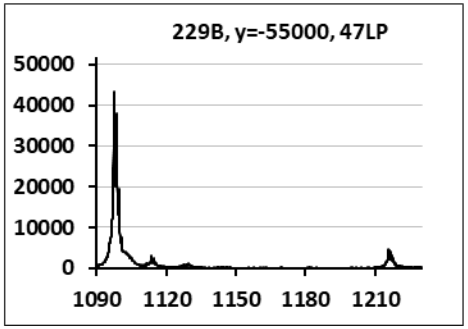


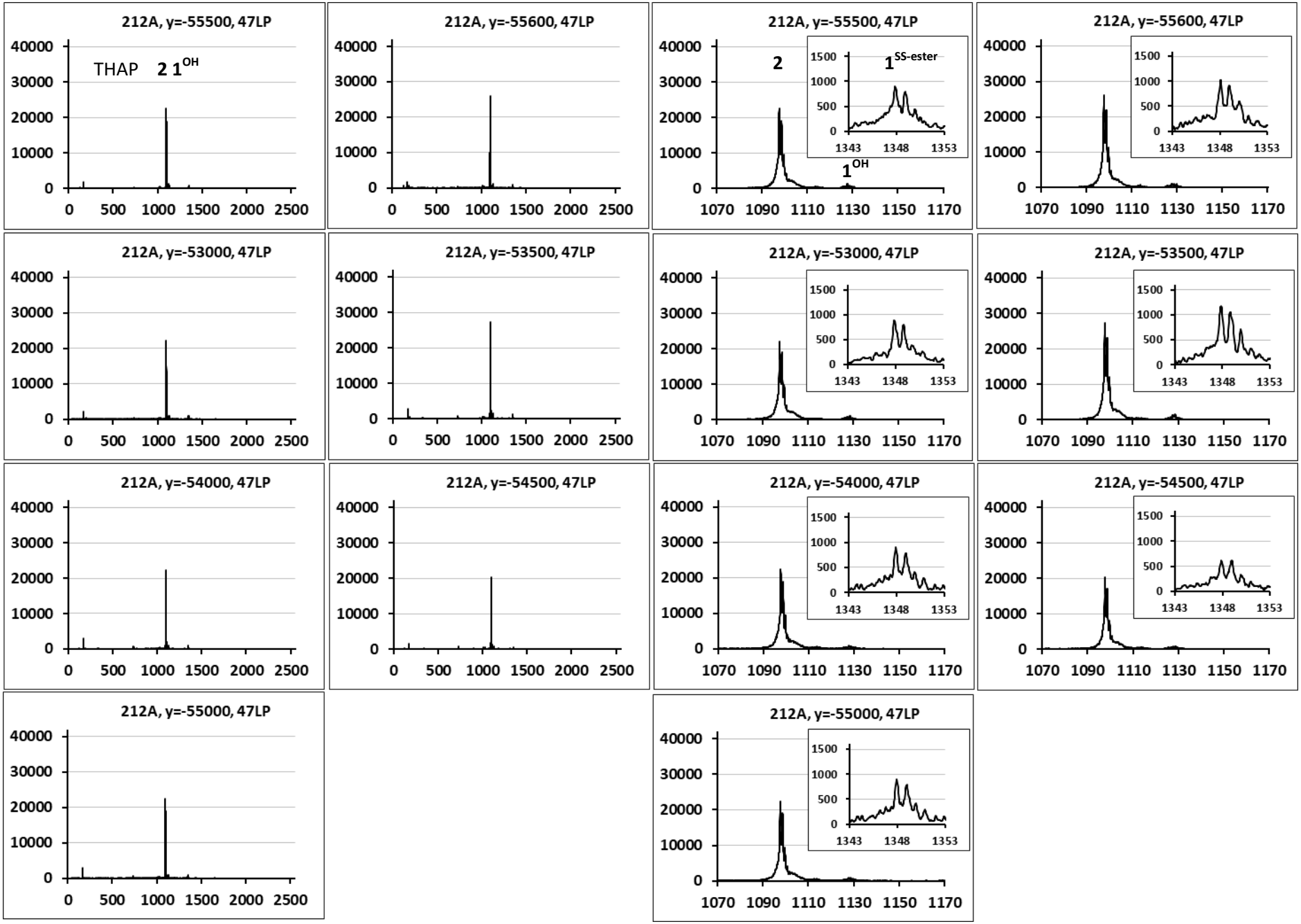
Fig. S6. Summaries and TOF-MS (full range and expansions) of location sampled to determine disappearance of $\mathbf{1}^{OH}$ following reaction of ($\mathbf{1}^{OH}\mathbf{1}^{OH}\mathbf{22}$) ML with the mono-ethyl ester of DTBP and to determine relative $\mathbf{1}^{SH}$ and **2** intensities following reaction of ($\mathbf{1}^{OH}\mathbf{1}^{OH}\mathbf{22}$) ML with the mono-ethyl ester followed by reaction with DTT reducing agent**.** Data collected at LP = 47 after applying a THAP thin film.

Summary: TOF-MS ML $\mathbf{1}^{OH}$ / **2** intensity and intensity ratio statistics after esterification with 40 mM mono-ethyl ester of DTBP, 40 mM EDC and 40 mM DMAP. THAP thin film applied, LP = 47. (TOF-MS measured at 14 locations on each of 4 independently prepared samples.)

[$\mathbf{1}^{OH}$]	[**2**]	**# Locations**	$\mathbf{1}^{OH}$ **/ 2**	$\mathbf{1}^{OH}$ **/ 2**	$\mathbf{1}^{OH}$ **/ 2**	$\mathbf{1}^{OH}$ **/ 2**	$\mathbf{1}^{OH}$ **/ 2**	$\mathbf{1}^{OH}$	$\mathbf{1}^{OH}$	$\mathbf{1}^{OH}$	$\mathbf{1}^{OH}$	$\mathbf{1}^{OH}$	**2**	**2**	**2**	**2**	**2**
µM	µM	(file names)	**Max**	**Min**	**Avg**	**σ**	**σ/Avg**	**Max**	**Min**	**Avg**	**σ**	**σ/Avg**	**Max**	**Min**	**Avg**	**σ**	**σ/Avg**
4	2	14 (212A,B)	0.043	0.024	0.035	0.005	15%	1444	626	925	227	24%	37330	16644	26182	5248	20%
4	2	14 (213A,B)	0.034	0.023	0.030	0.003	10%	1260	371	890	216	24%	40531	15942	29894	6048	20%
4	2	14 (224A,B)	0.057	0.038	0.048	0.006	13%	1585	851	1246	234	19%	32956	19472	26092	3879	15%
4	2	14 (225A,B)	0.059	0.037	0.045	0.008	18%	1484	743	1059	197	19%	28240	19374	23670	2423	10%
4	2	All	0.059	0.023	**0.039**	**0.009**	**24%**	1585	371	**1030**	**255**	**25%**	40531	15942	**26459**	**5014**	**19%**

4 µM **1**^{OH} + 6 µM **2** 40 mM DTBP mono-ethyl ester, 40 mM EDC, 40 mM DMAP

0-2.5 kD 1.07-1.17kD (inset 1.343-1.353 kD)



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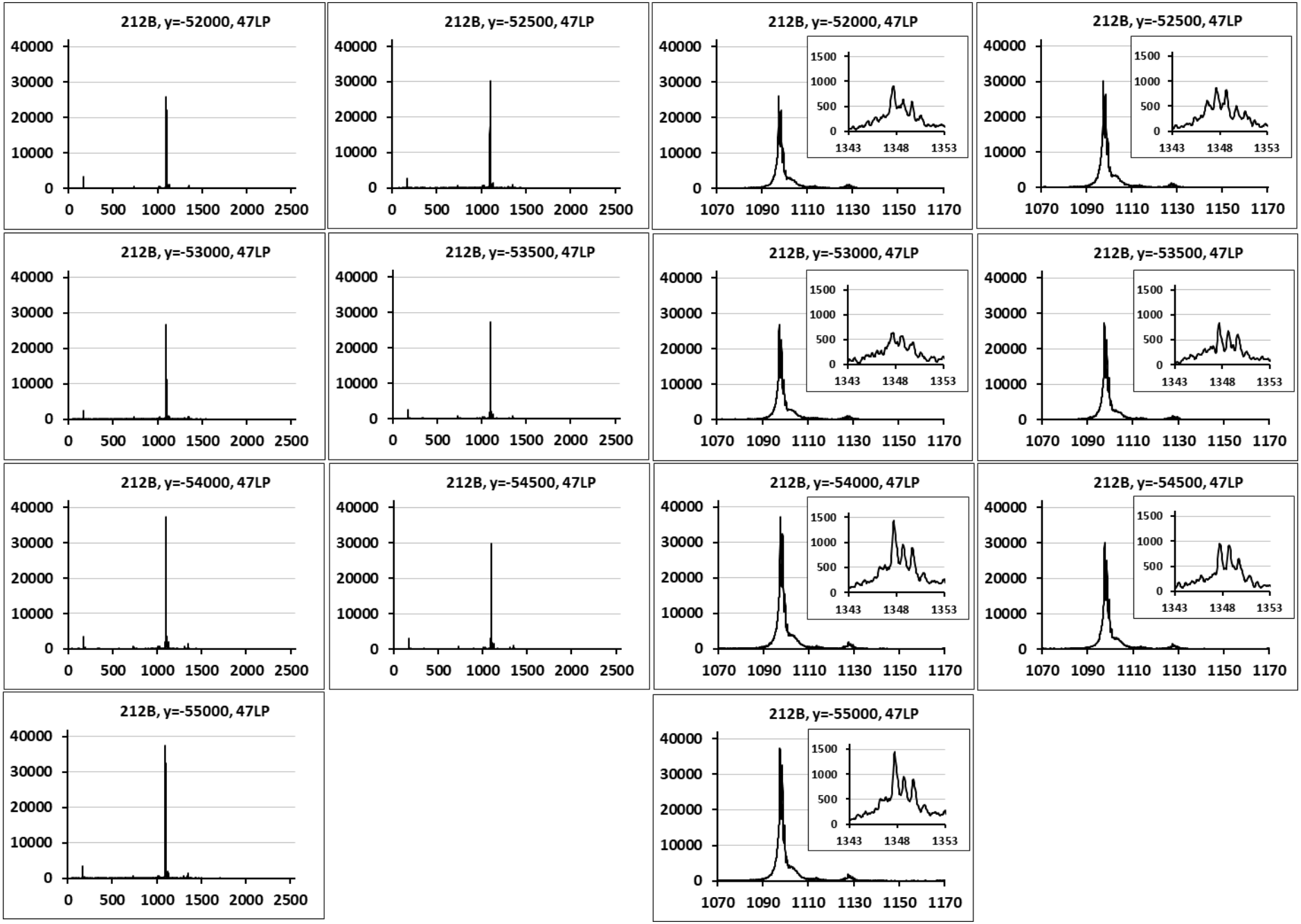
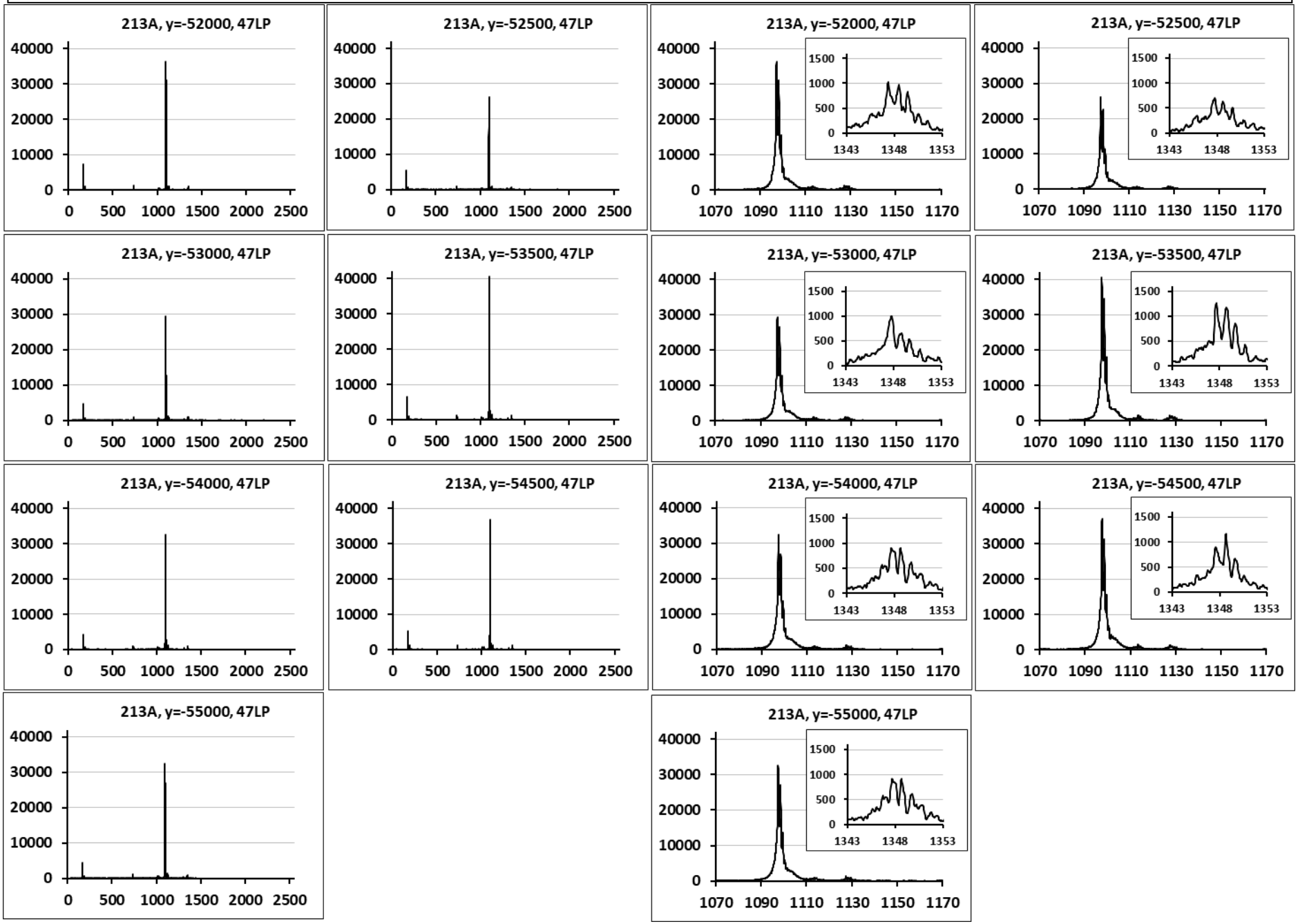
0-2.5 kD 1.07-1.17kD (inset 1.343-1.353 kD)



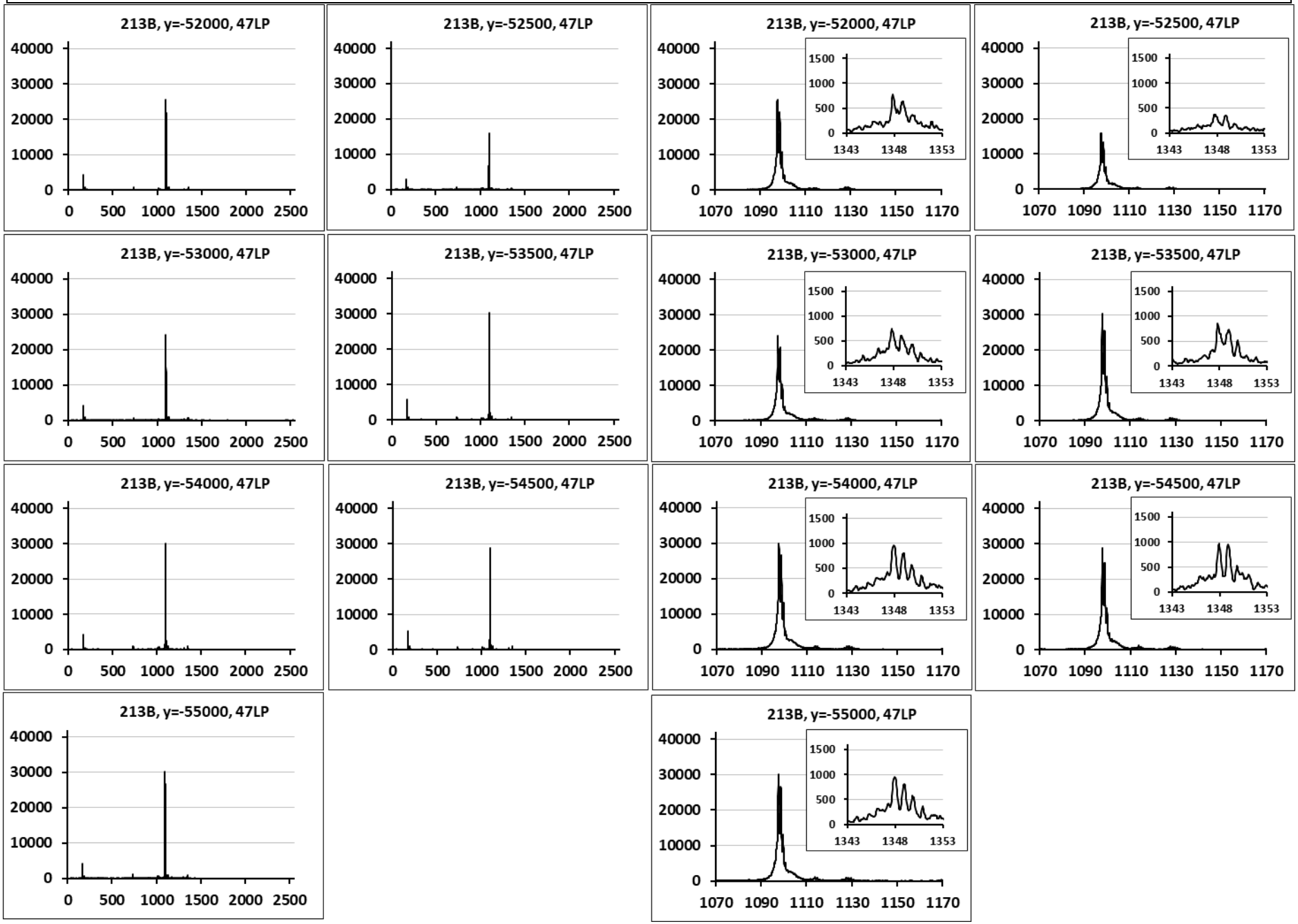
4 µM **1**^{OH} + 6 µM **2** 40 mM DTBP mono-ethyl ester, 40 mM EDC, 40 mM DMAP

0-2.5 kD 1.07-1.17kD (inset 1.343-1.353 kD)



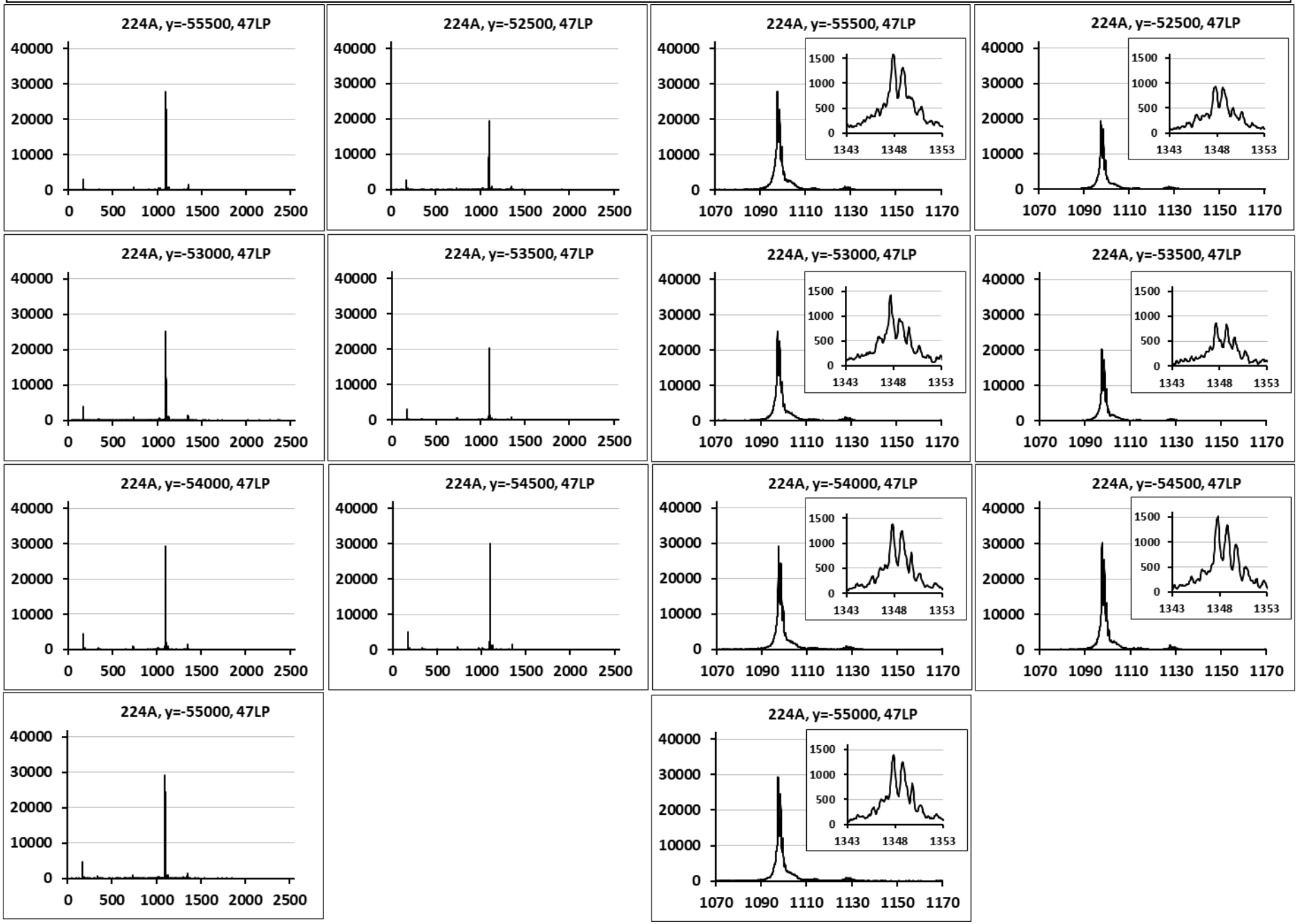
4 µM **1**^{OH} + 6 µM **2** 40 mM DTBP mono-ethyl ester, 40 mM EDC, 40 mM DMAP

0-2.5 kD 1.07-1.17kD (inset 1.343-1.353 kD)



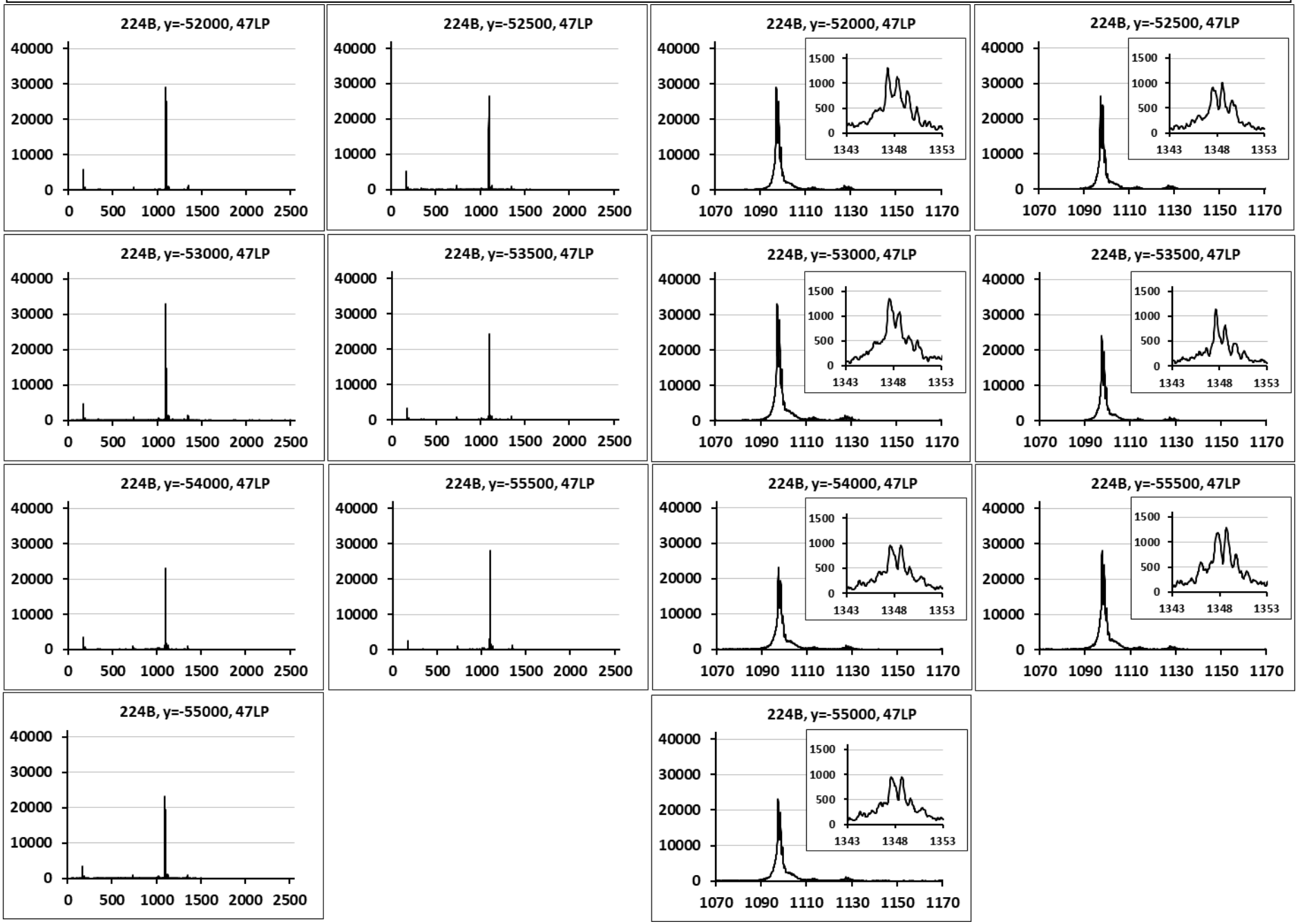
4 µM **1**^{OH} + 6 µM **2** 40 mM DTBP mono-ethyl ester, 40 mM EDC, 40 mM DMAP

0-2.5 kD 1.07-1.17kD (inset 1.343-1.353 kD)



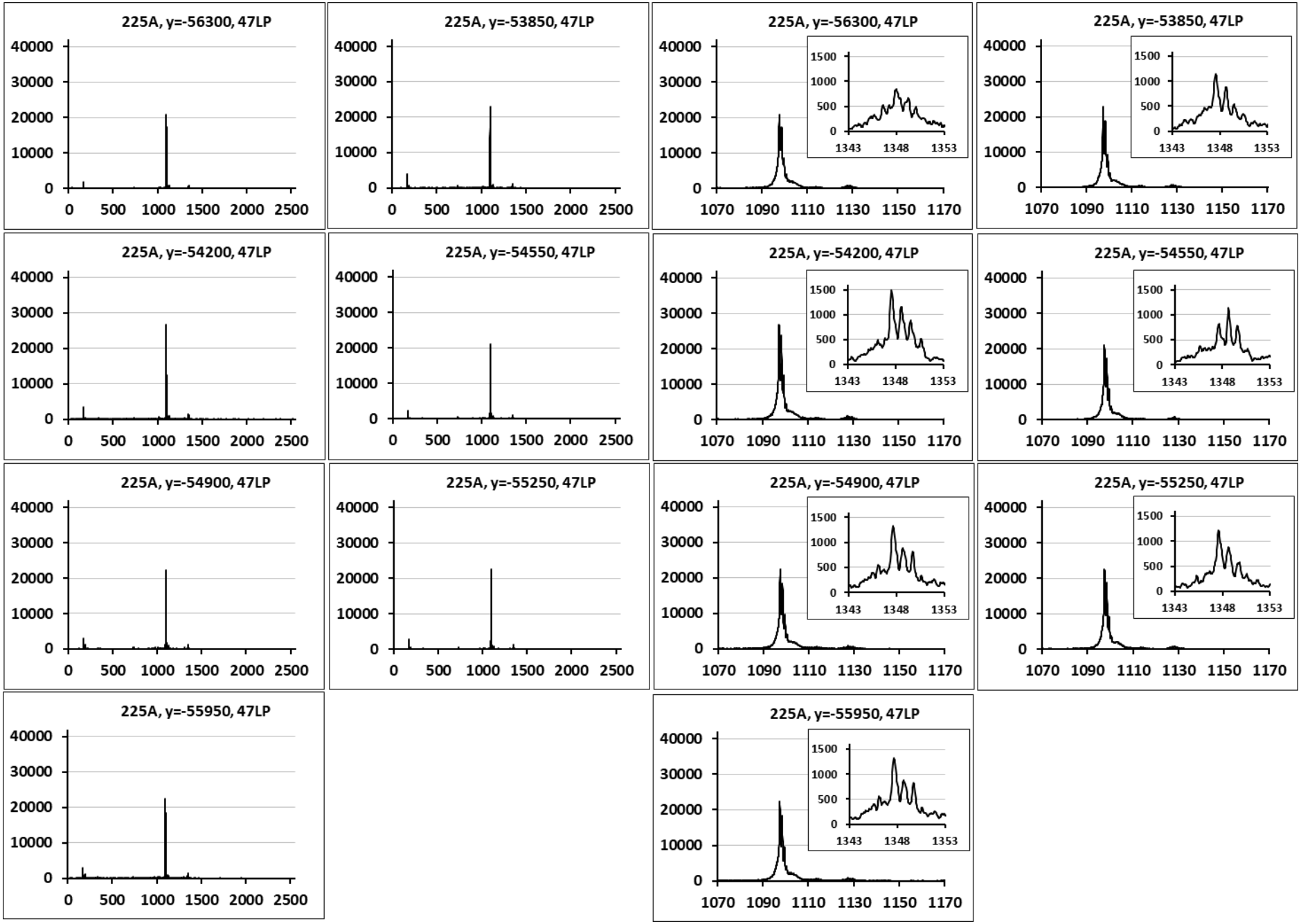
4 µM **1**^{OH} + 6 µM **2** 40 mM DTBP mono-ethyl ester, 40 mM EDC, 40 mM DMAP

0-2.5 kD 1.07-1.17kD (inset 1.343-1.353 kD)



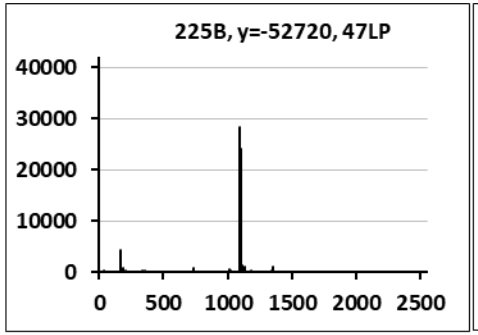
4 µM **1**^{OH} + 6 µM **2** 40 mM DTBP mono-ethyl ester, 40 mM EDC, 40 mM DMAP

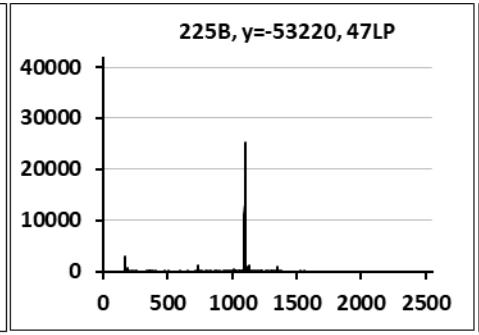
0-2.5 kD 1.07-1.17kD (inset 1.343-1.353 kD)

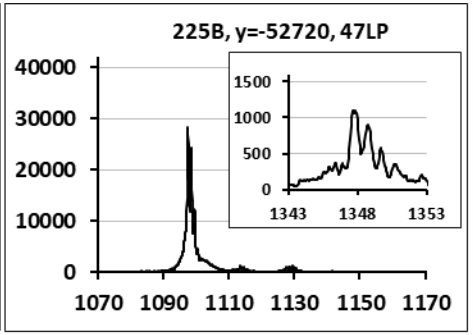


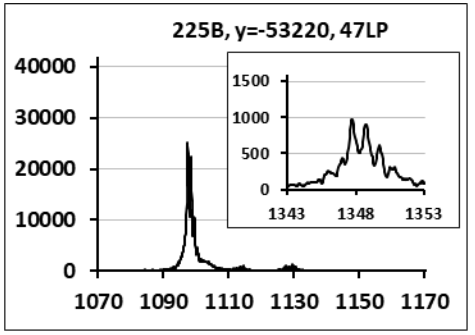
4 μM **1**^{OH} + 6 μM **2** 40 mM DTBP mono-ethyl ester, 40 mM EDC, 40 mM DMAP

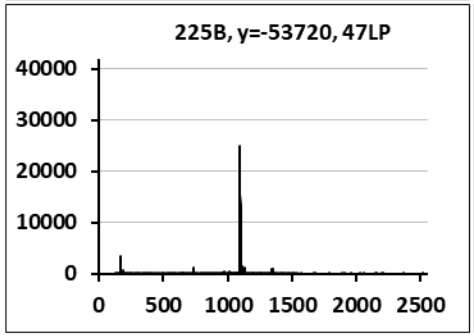
0-2.5 kD 1.07-1.17kD (inset 1.343-1.353 kD)

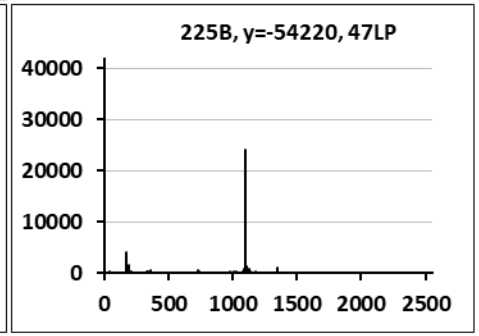


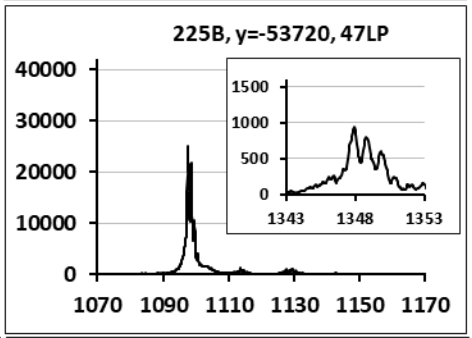


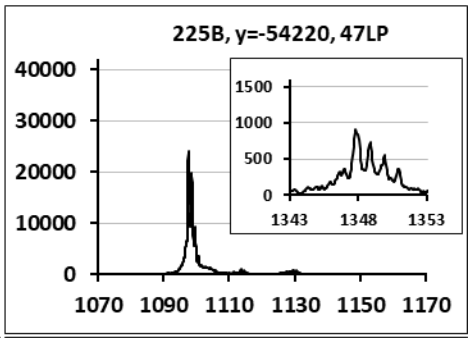


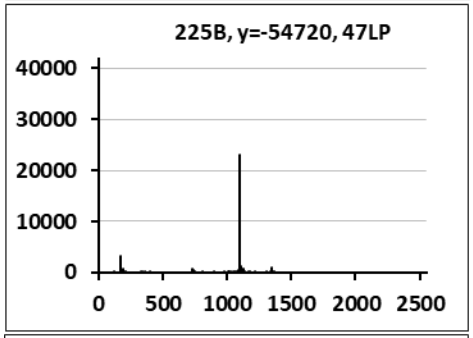


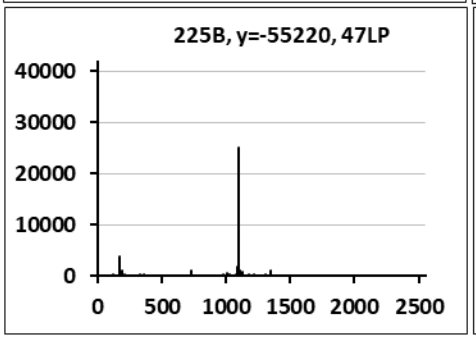


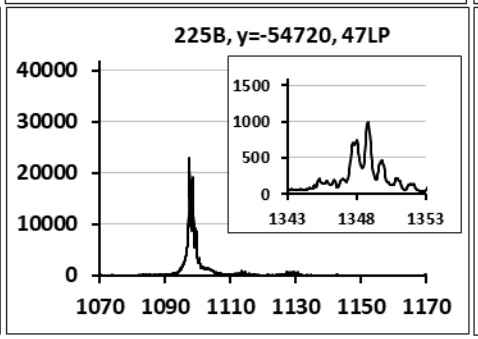


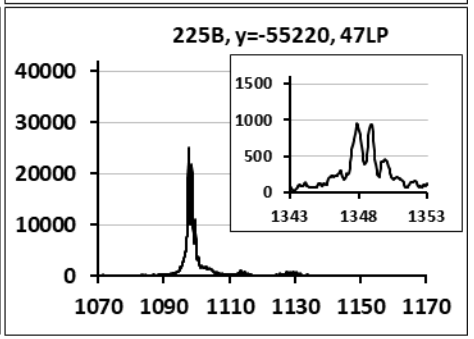


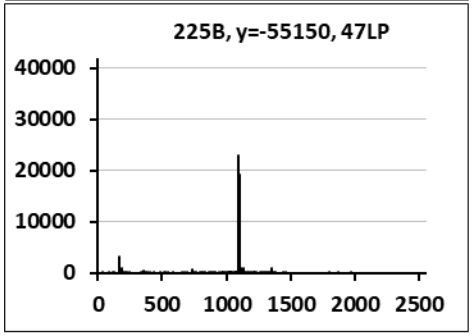


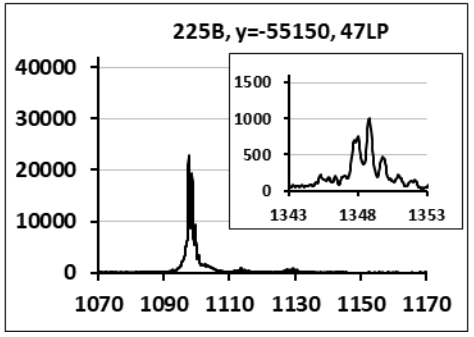










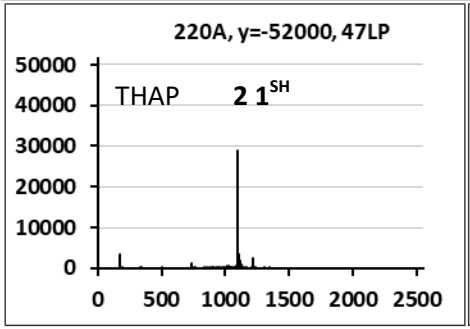


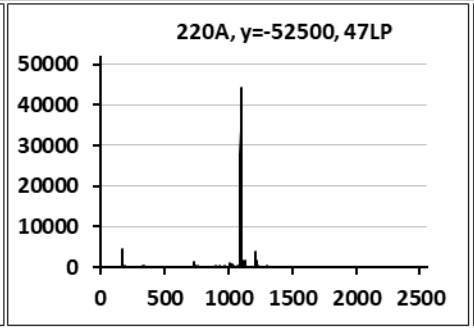
Summary: TOF-MS ML **1^{SH} / 2** intensity and intensity ratio statistics after esterification with 40 mM mono-ethyl ester of DTBP, 40 mM EDC and 40 mM DMAP followed by reduction with DTT reducing agent. THAP thin film applied, LP = 47. (TOF-MS measured at 14 locations on each of 4 independently prepared samples.)

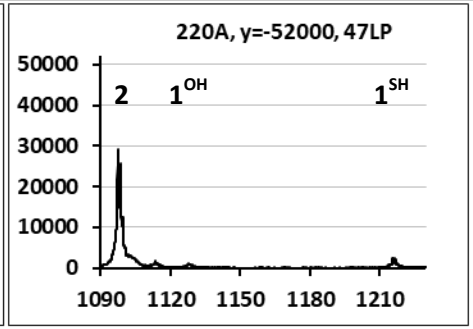
[1^{OH}]	[2]	**# Locations**	**1^{SH} / 2**	**1^{SH} / 2**	**1^{SH} / 2**	**1^{SH} / 2**	**1^{SH} / 2**	**1^{SH}**	**1^{SH}**	**1^{SH}**	**1^{SH}**	**1^{SH}**	**2**	**2**	**2**	**2**	**2**
µM	µM	(file names)	**Max**	**Min**	**Avg**	**σ**	**σ/Avg**	**Max**	**Min**	**Avg**	**σ**	**σ/Avg**	**Max**	**Min**	**Avg**	**σ**	**σ/Avg**
4	2	14 (220A,B)	0.11	0.07	0.08	0.01	15%	3717	1218	2598	703	27%	44045	17333	31878	7341	23%
4	2	14 (221A,B)	0.14	0.09	0.11	0.01	11%	6746	2222	3997	1206	30%	48964	21660	36834	7383	20%
4	2	14 (222A,B)	0.10	0.08	0.09	0.01	9%	4690	2112	2859	684	24%	45653	24863	32199	5841	18%
4	2	14 (223A,B)	0.10	0.06	0.08	0.01	11%	4124	1973	2897	603	21%	48148	29602	35714	5517	15%
4	2	All	0.14	0.06	**0.09**	**0.01**	**17%**	6746	1218	**3088**	**975**	**32%**	48964	17333	**34156**	**6754**	**20%**

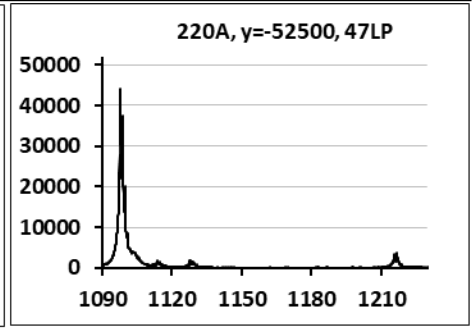
4 µM **1**^{OH} + 6 µM **2** 40 mM DTBP mono-ethyl ester, 40 mM EDC, 40 mM DMAP followed by DTT reduction

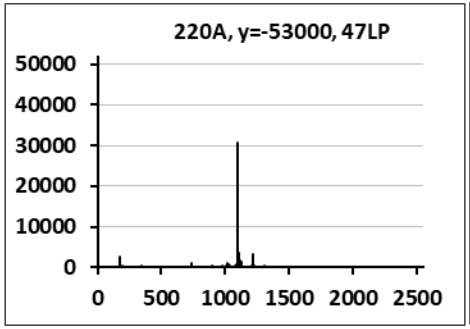
0-2.5 kD 1.09-1.23kD

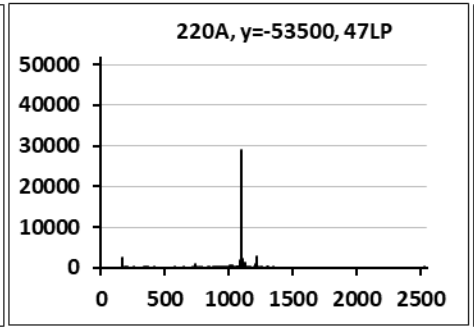


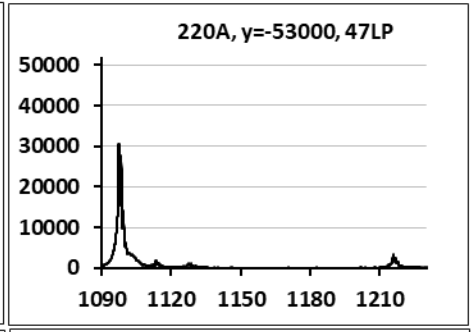


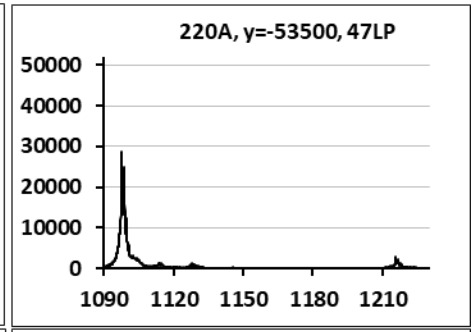


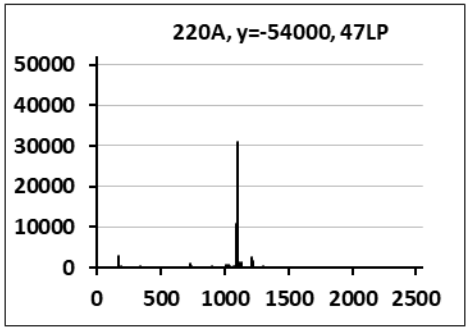


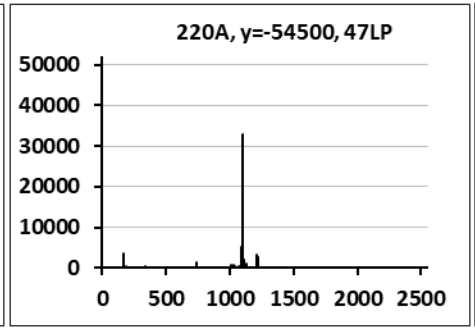


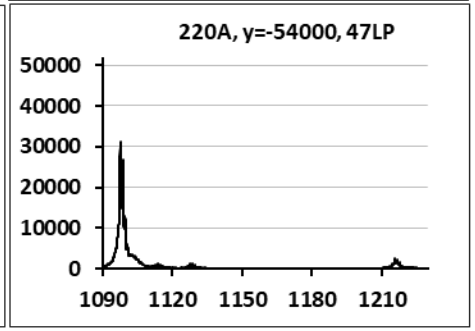


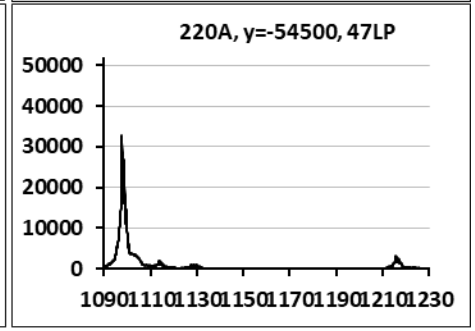


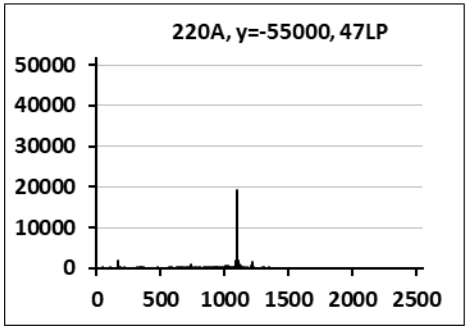


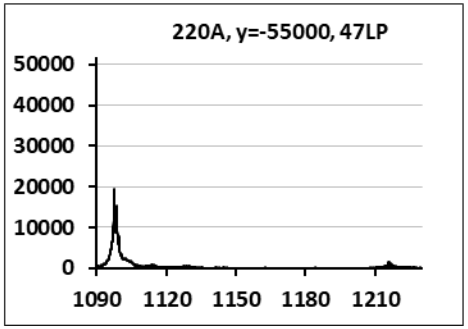






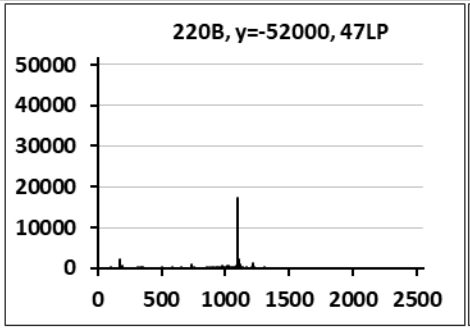


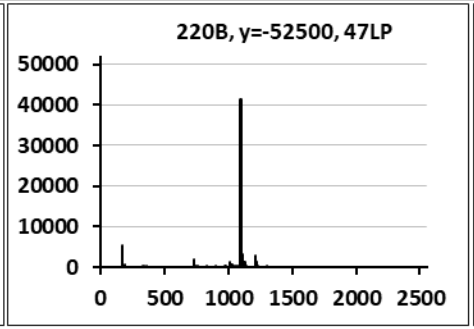


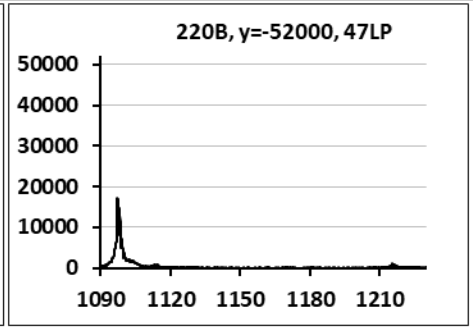


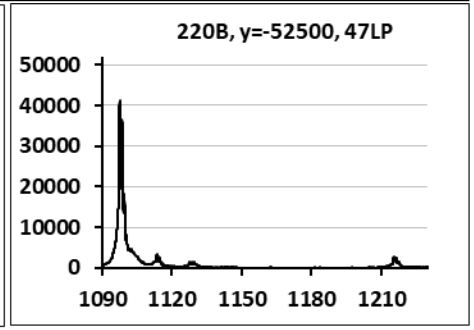
4 µM **1**^{OH} + 6 µM **2** 40 mM DTBP mono-ethyl ester, 40 mM EDC, 40 mM DMAP followed by DTT reduction

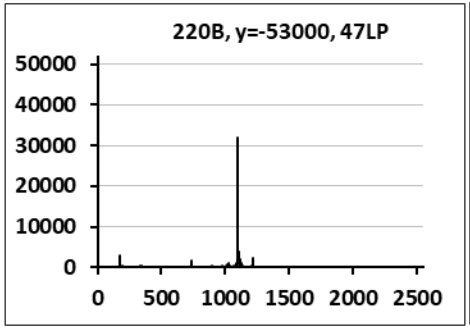
0-2.5 kD 1.09-1.23kD

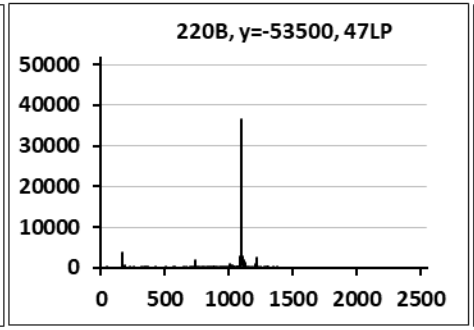


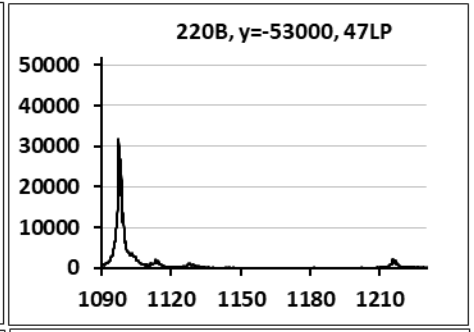


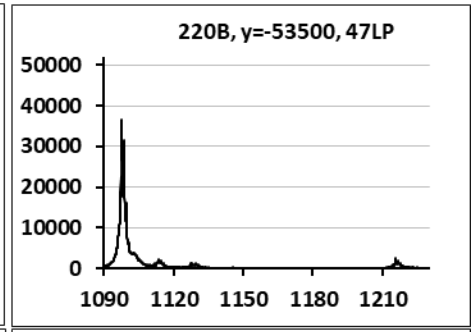


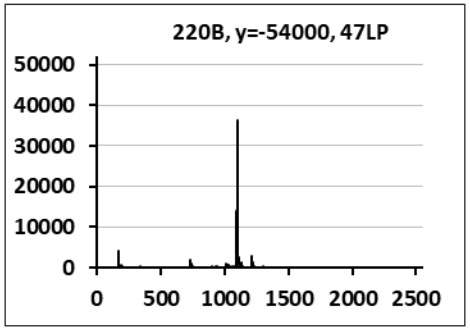


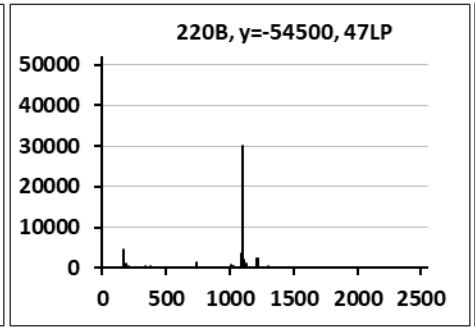


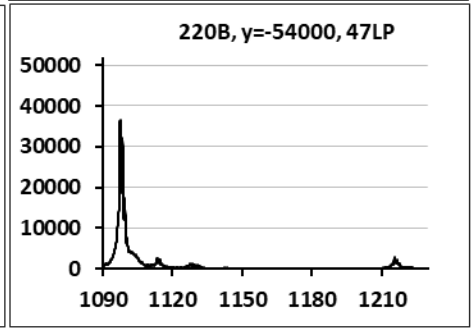


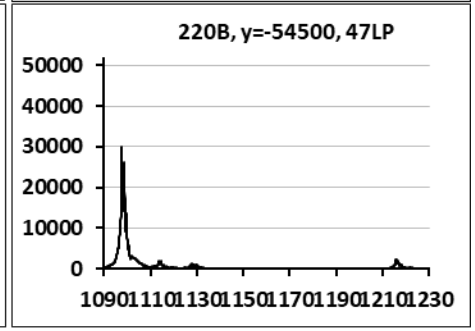


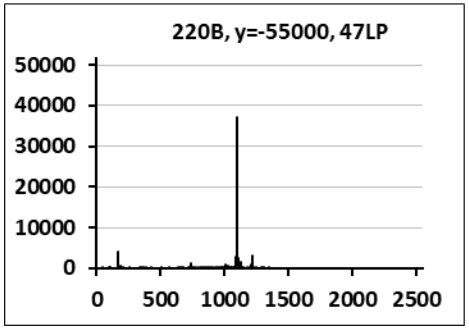


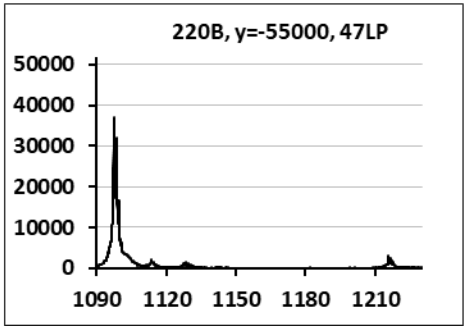






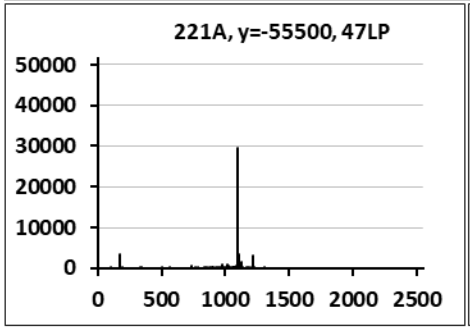


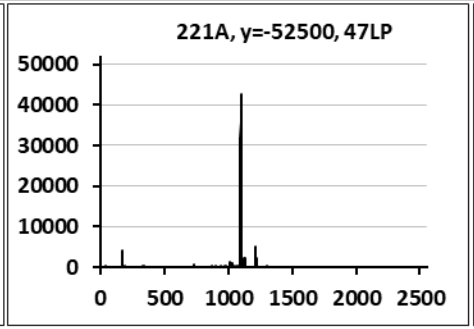


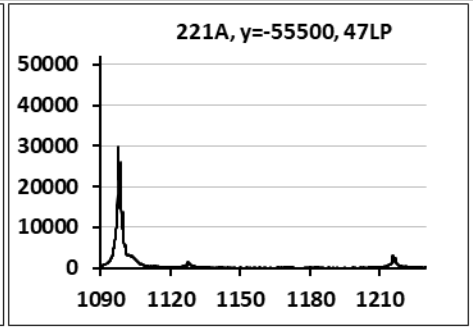


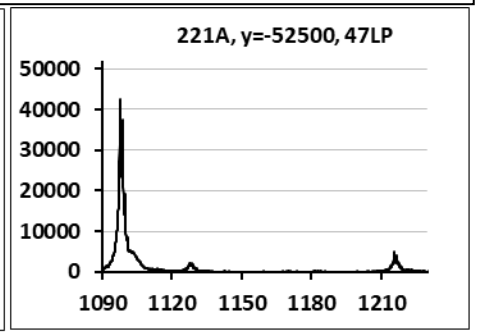
4 µM **1**^{OH} + 6 µM **2** 40 mM DTBP mono-ethyl ester, 40 mM EDC, 40 mM DMAP followed by DTT reduction

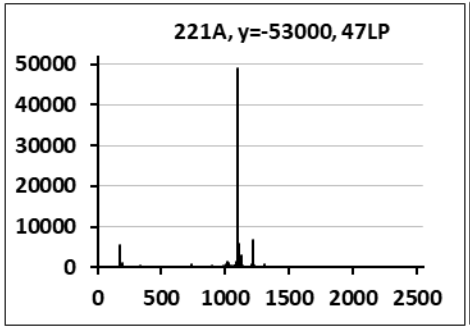
0-2.5 kD 1.09-1.23kD

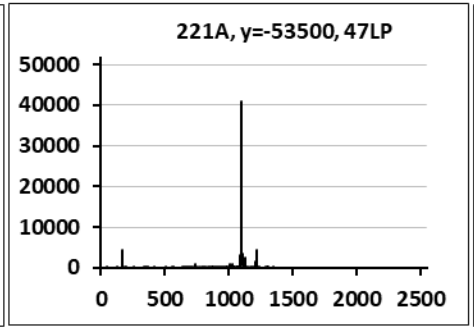


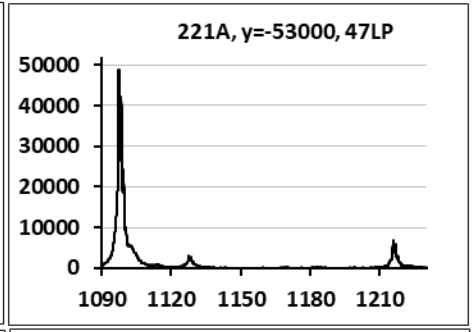


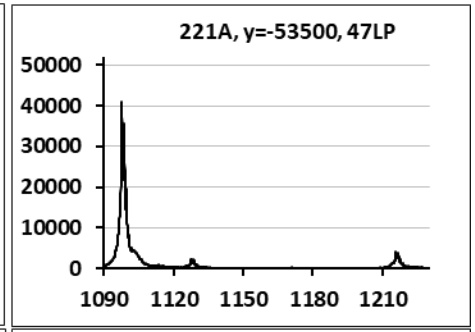


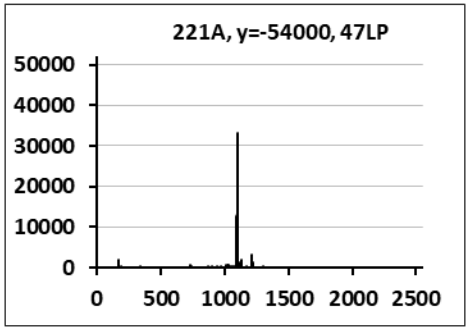


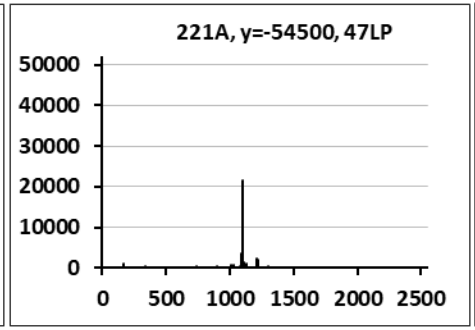


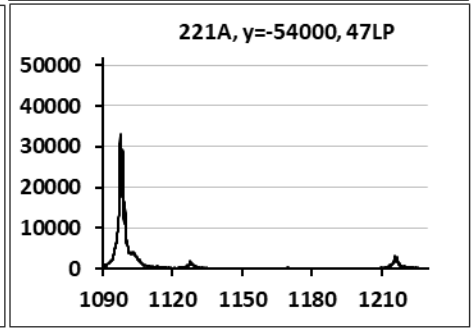


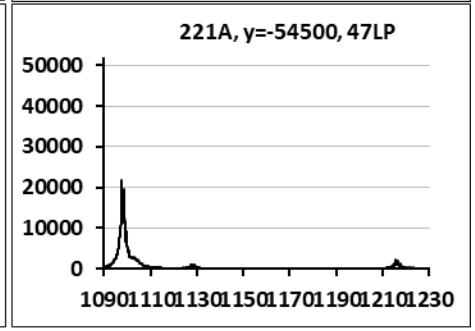


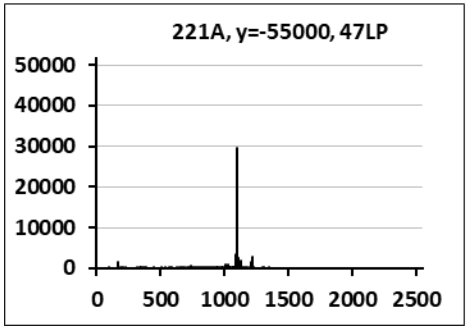


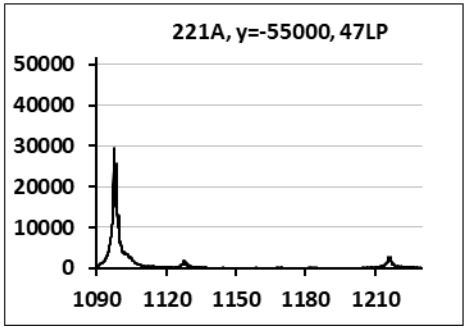




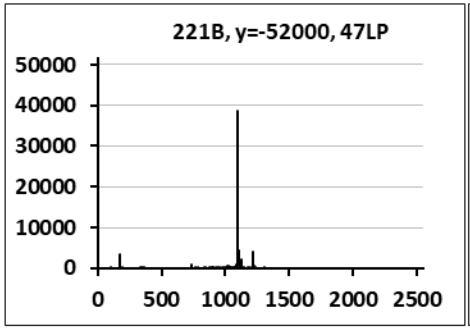


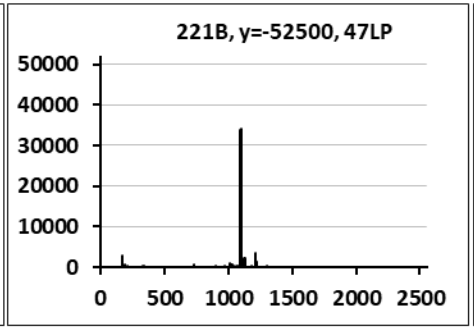


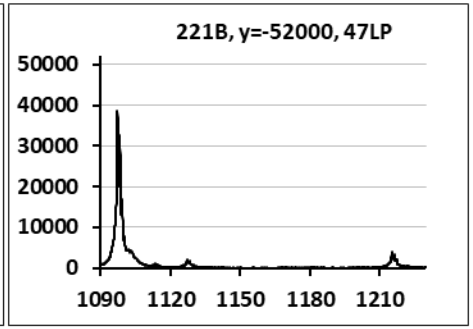


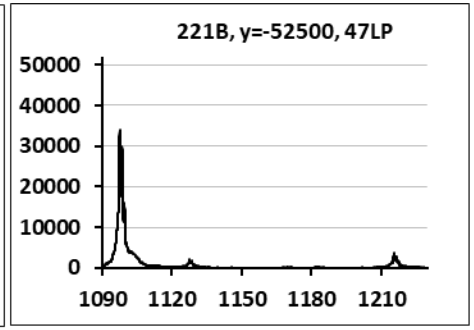


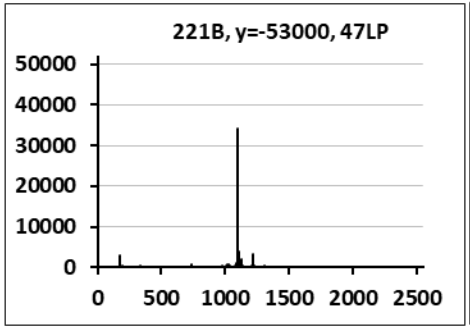
4 µM $\mathbf{1}^{OH}$ + 6 µM **2**	40 mM DTBP mono-ethyl ester, 40 mM EDC, 40 mM DMAP followed by DTT reduction
0-2.5 kD	1.09-1.23kD

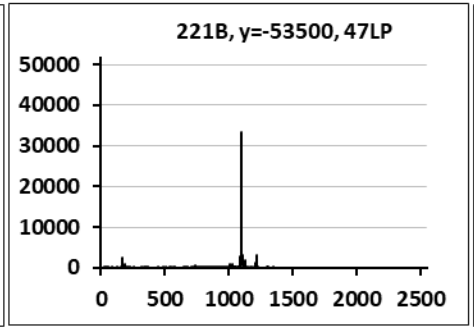


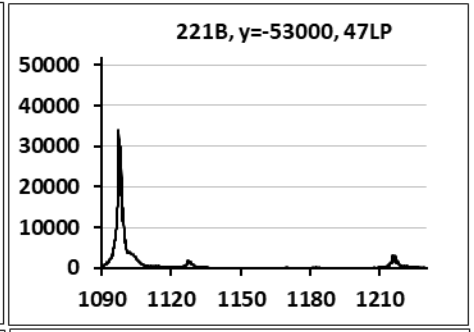


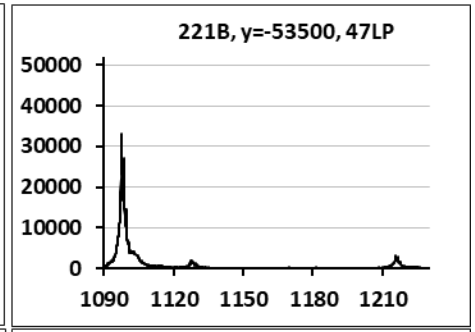


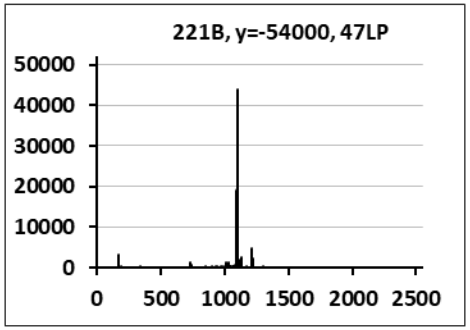


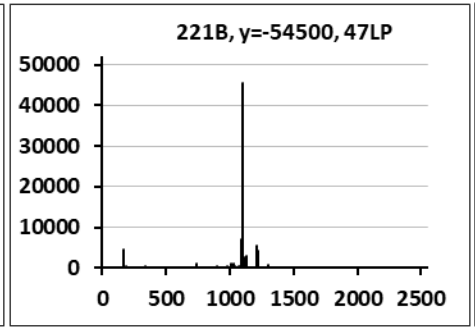


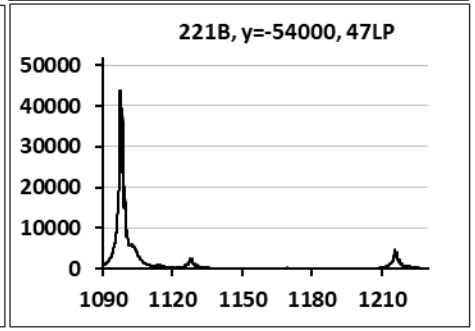


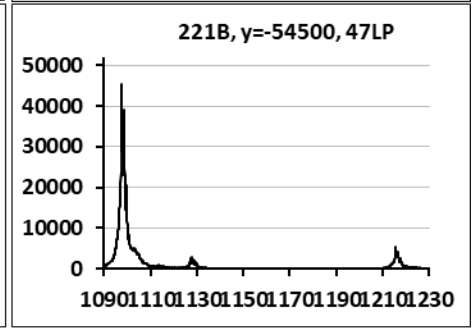


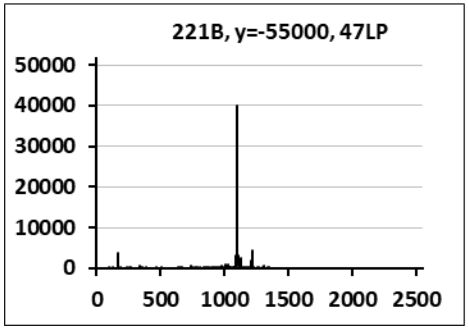


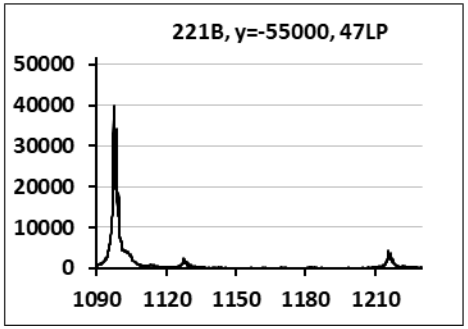












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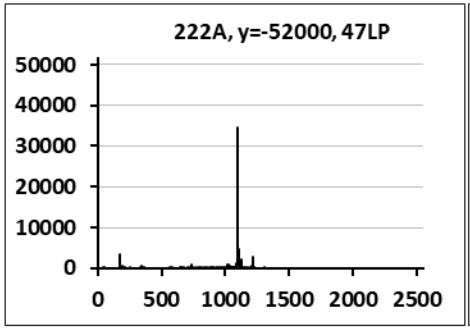
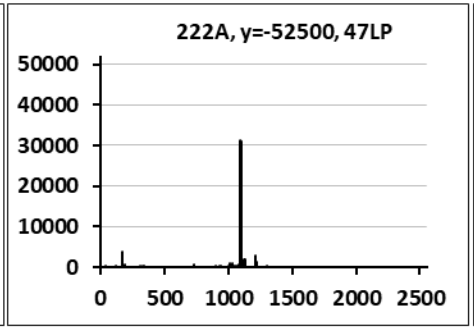
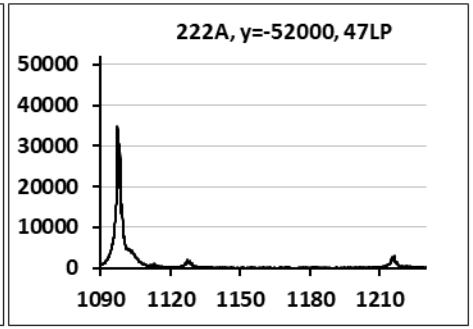
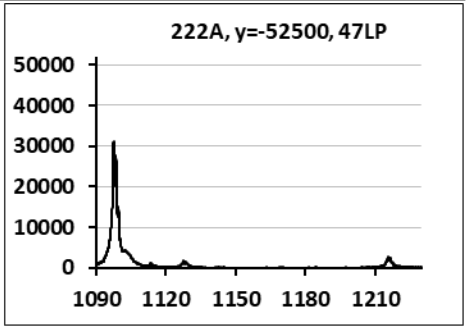
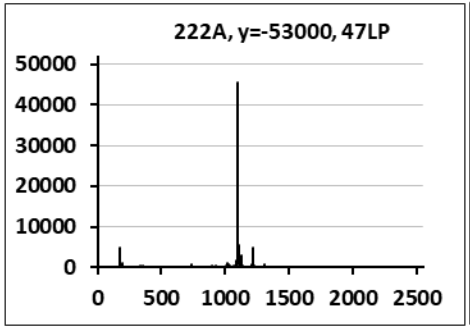
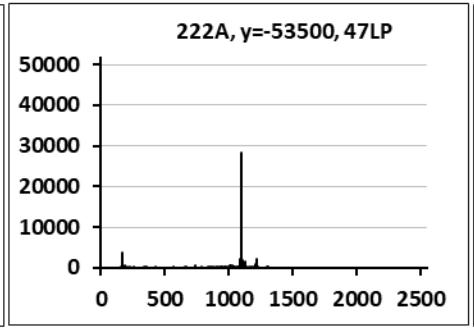


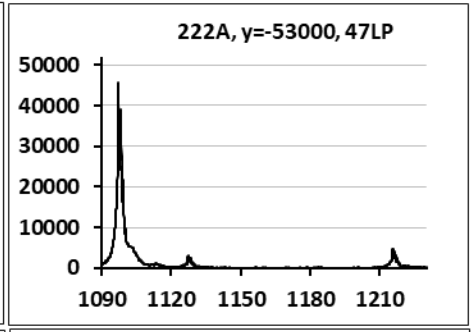


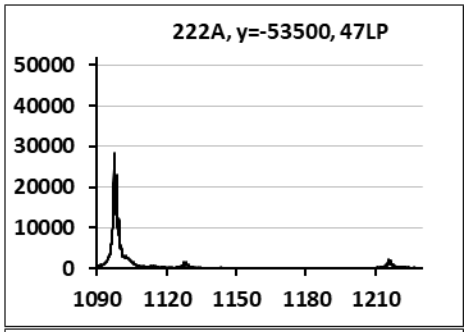


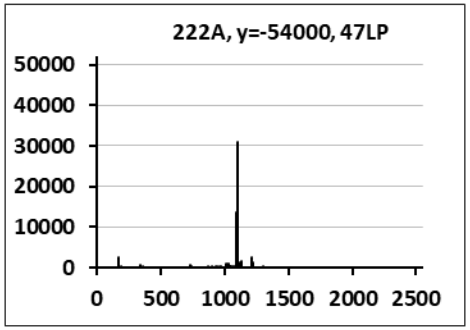


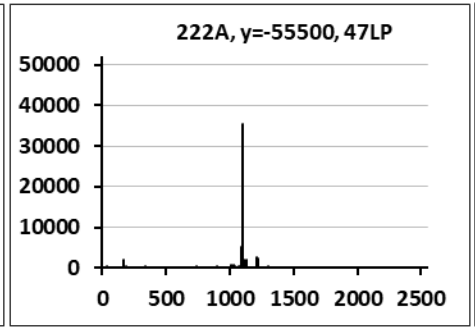


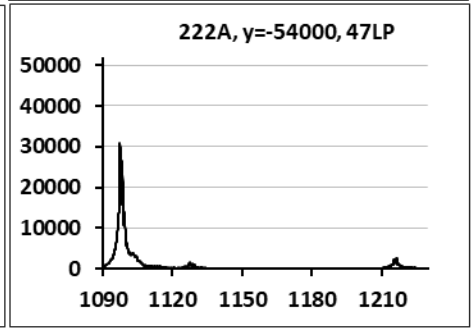


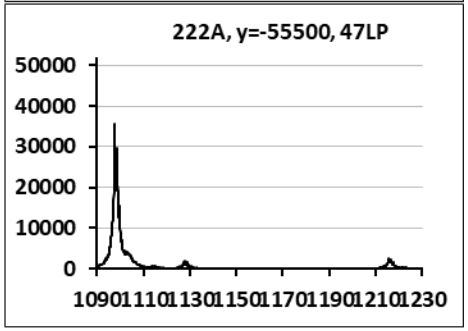


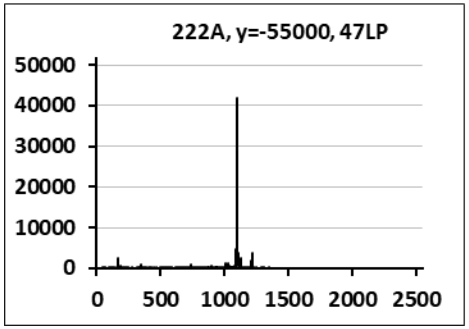


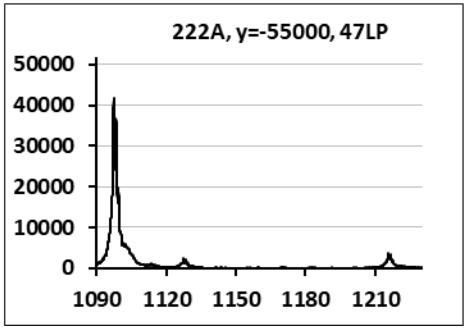




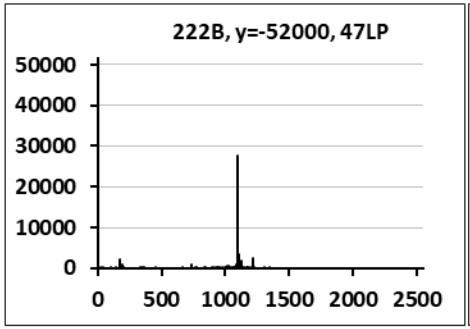


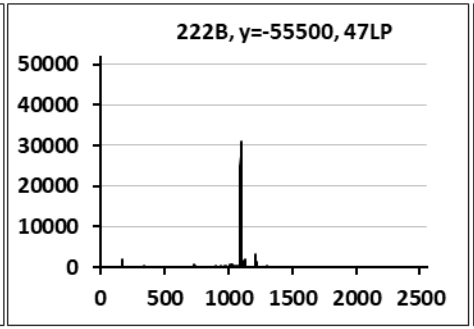


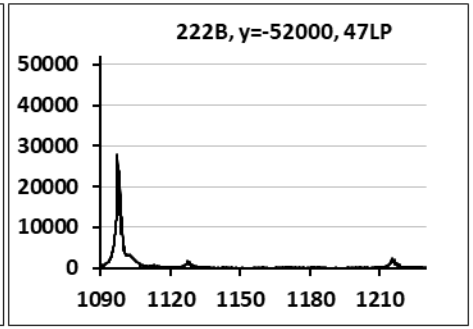


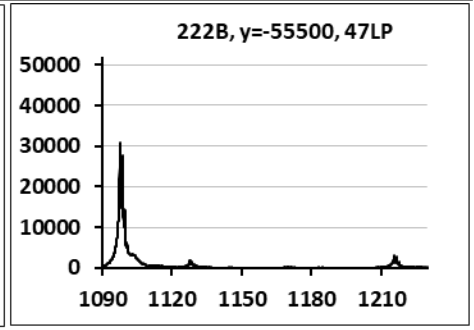


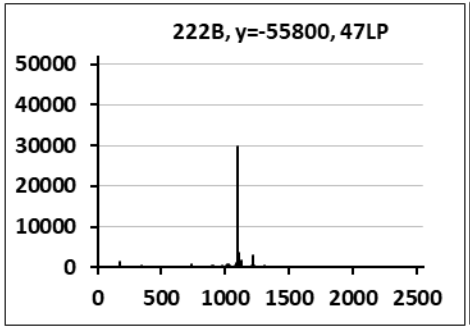
4 µM **1**^{OH} + 6 µM **2**	40 mM DTBP mono-ethyl ester, 40 mM EDC, 40 mM DMAP followed by DTT reduction
0-2.5 kD	1.09-1.23kD

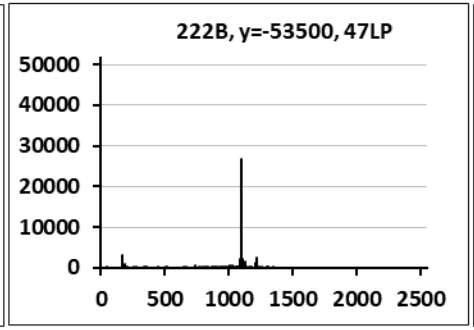


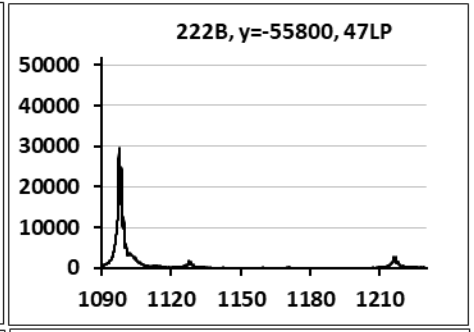


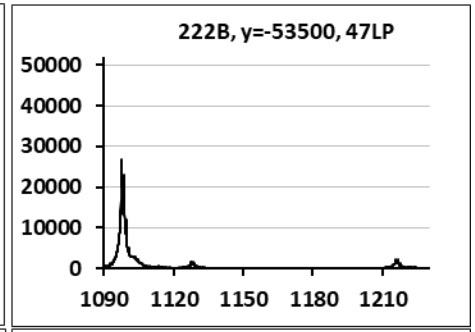


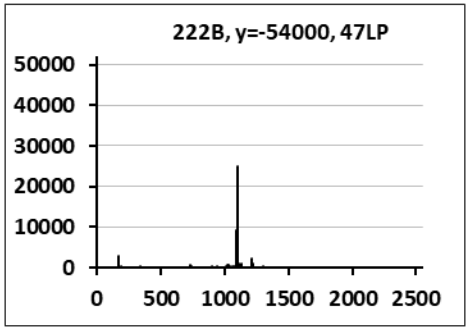


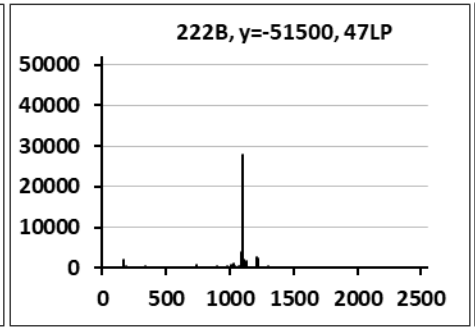


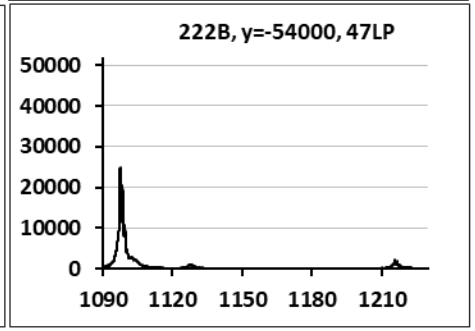


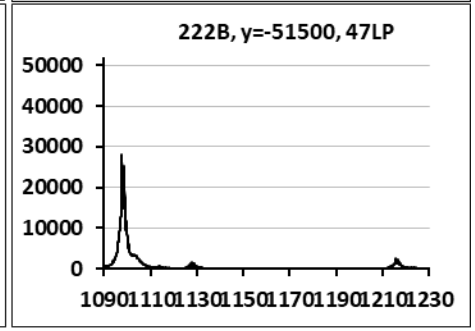


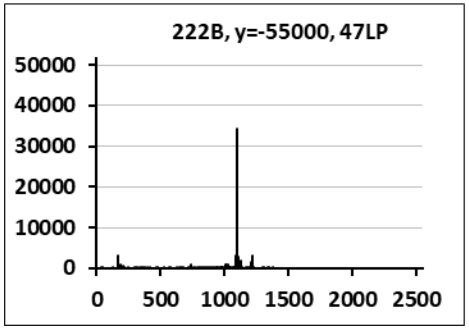


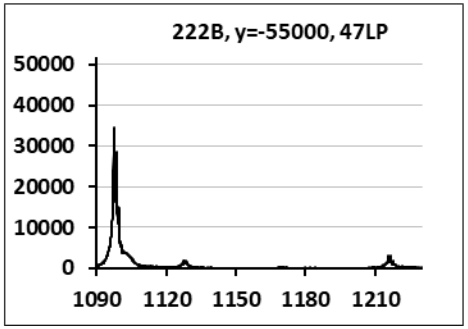




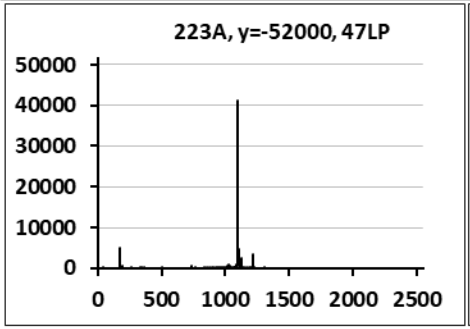


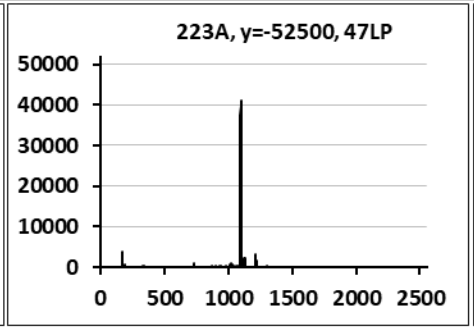


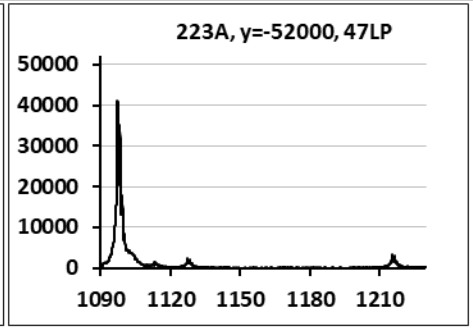


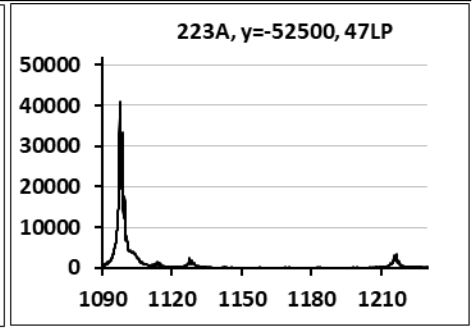


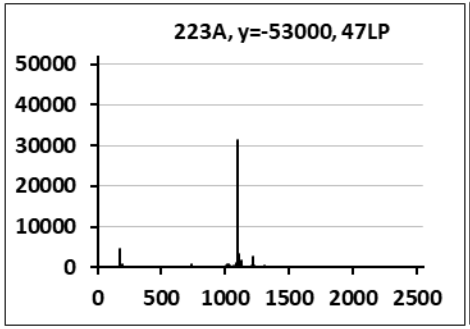
4 µM **1**^{OH} + 6 µM **2**	40 mM DTBP mono-ethyl ester, 40 mM EDC, 40 mM DMAP followed by DTT reduction
0-2.5 kD	1.09-1.23kD

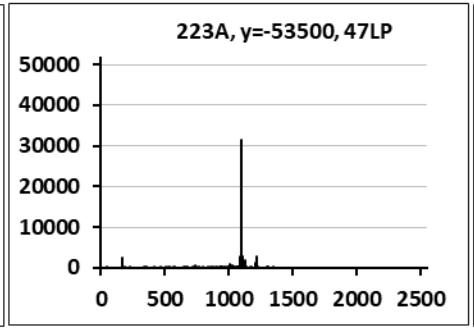


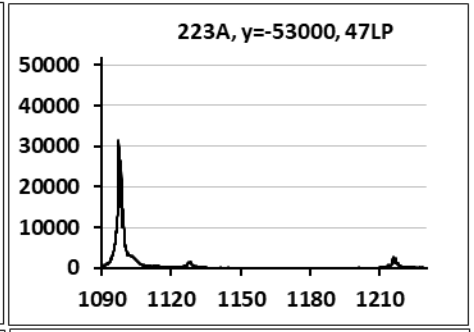


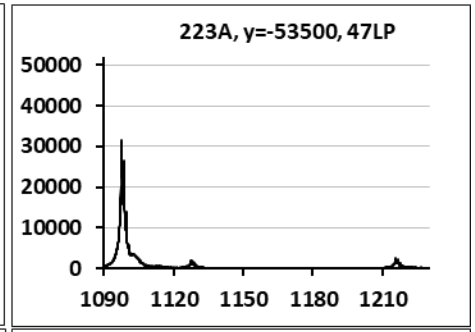


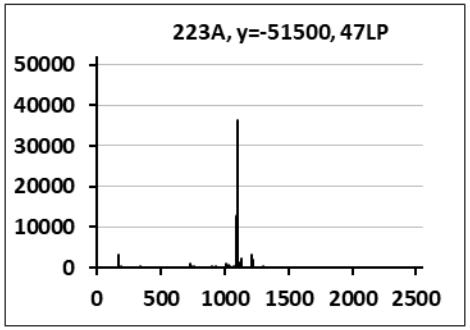


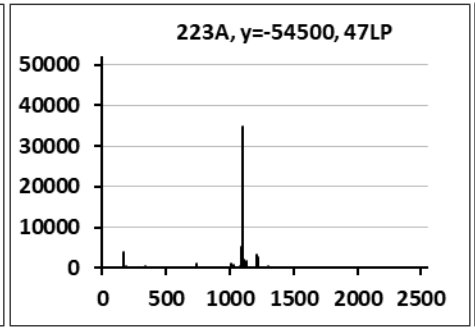


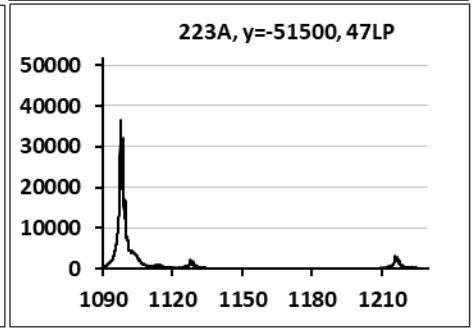


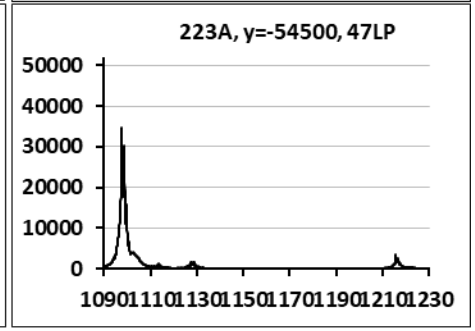


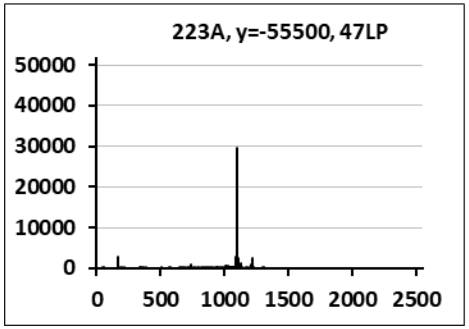


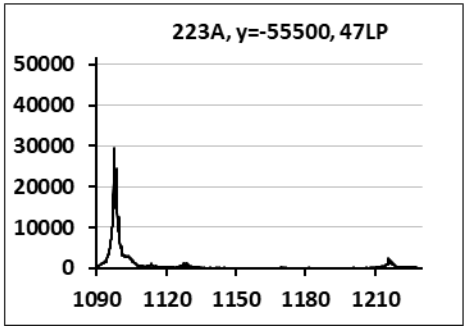




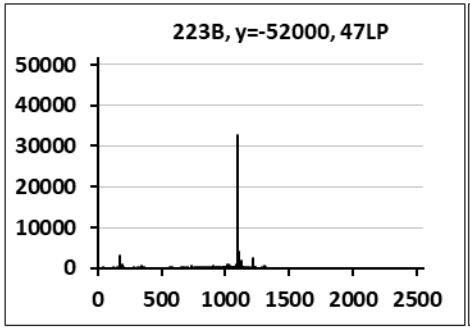


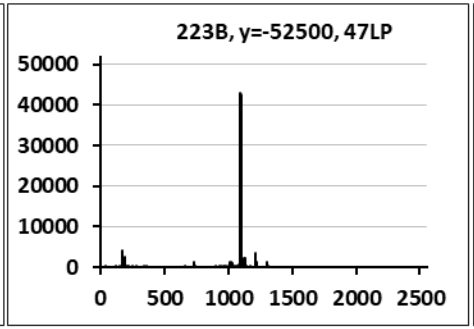


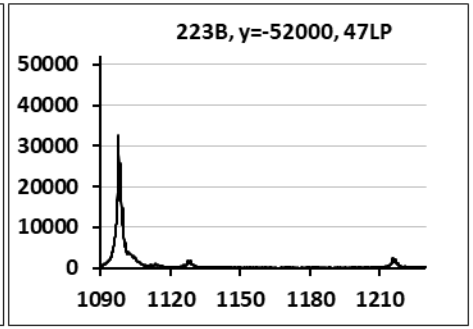


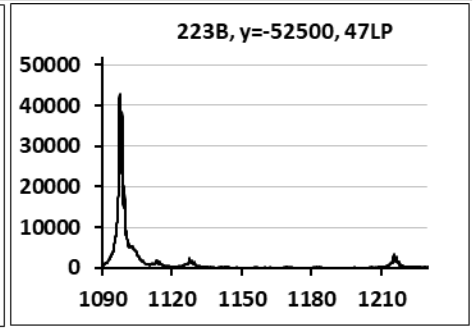


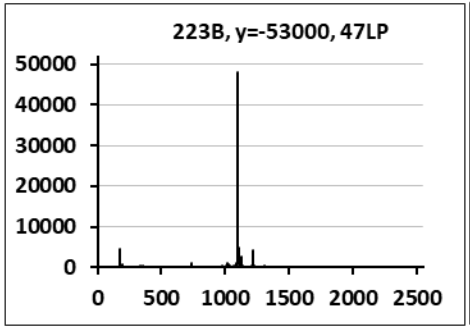
4 µM **1**^{OH} + 6 µM **2**	40 mM DTBP mono-ethyl ester, 40 mM EDC, 40 mM DMAP followed by DTT reduction
0-2.5 kD	1.09-1.23kD

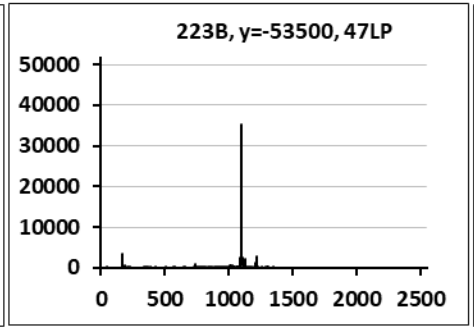


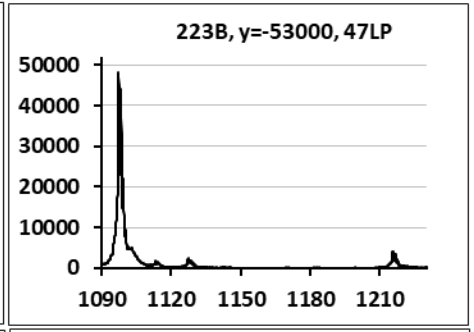


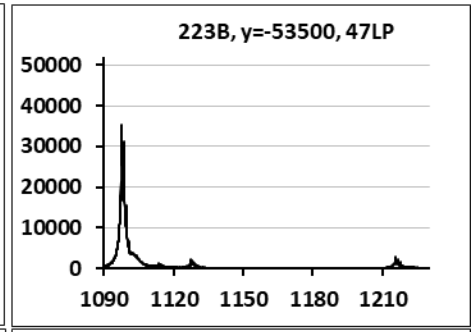


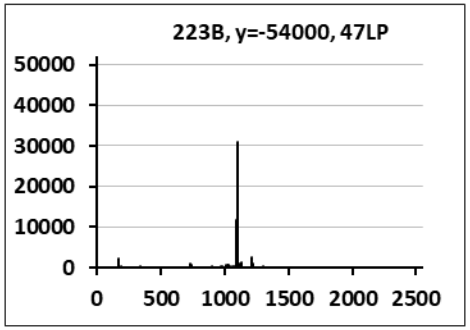


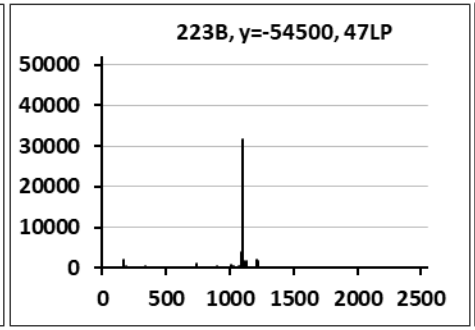


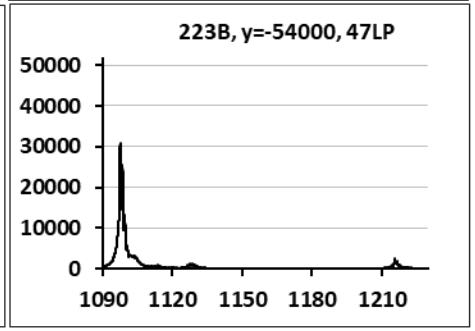


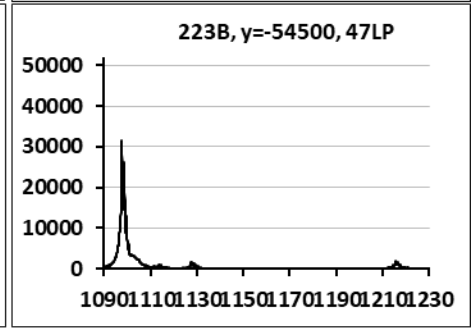


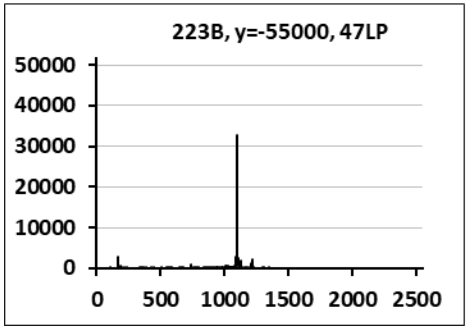












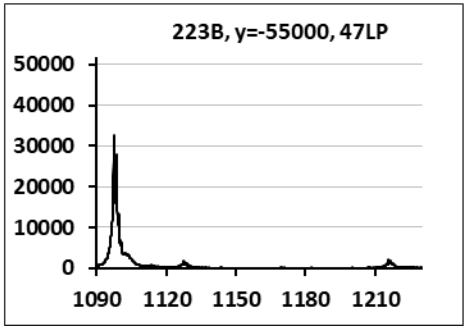
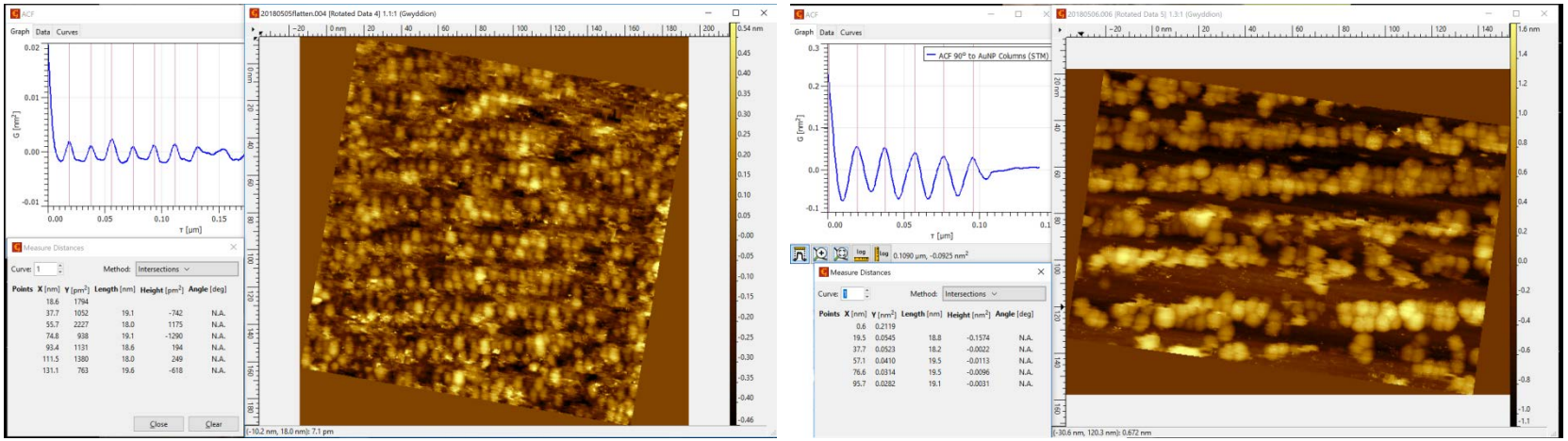
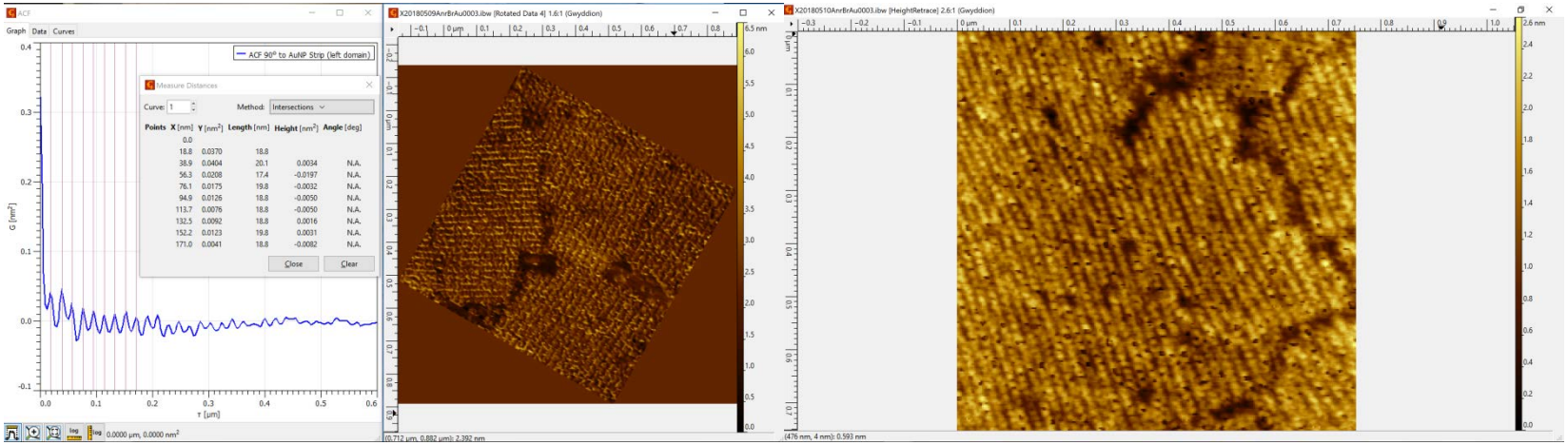


Fig. S7. Additional STM and AFM images of templated AuNP films and AFM control experiments.

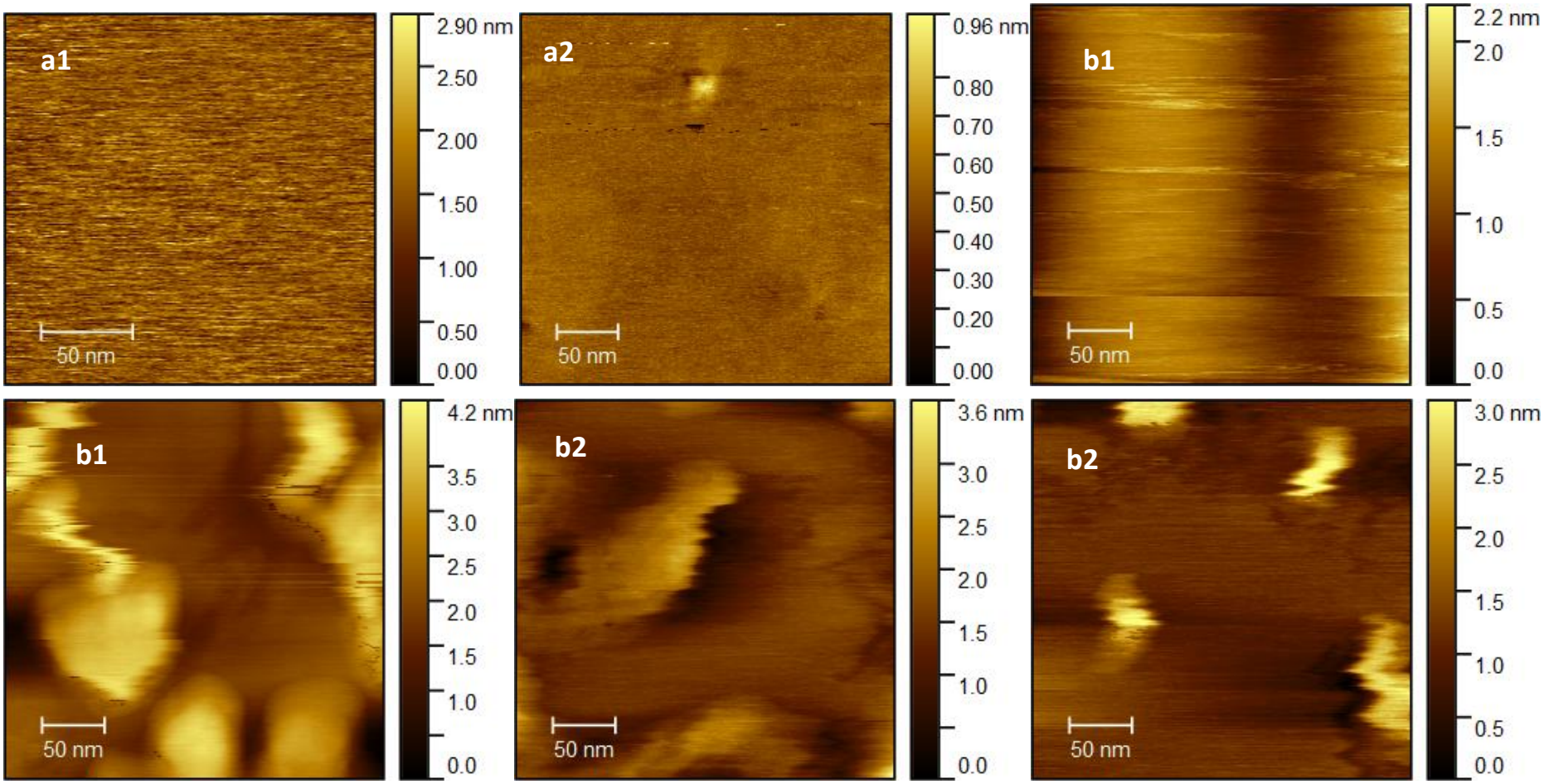
STM images collected from two different samples. The scans have been rotated so that the AuNP columns are aligned horizontally. The height difference autocorrelation (upper left box in each image) was calculated along a vertical axis. Column 3 in each table lists the spacings between autocorrelation maxima.



(Below) AFM images collected from two different samples. The left scan has been rotated so that one domain's AuNP columns are aligned horizontally. The height difference autocorrelation (upper left box in image) was calculated along a vertical axis using the entire image. Column 3 in the table lists the spacings between autocorrelation maxima. (right) 750 nm scan.



AFM control experiments. (a1,a2) AFM scans of patterned [$\mathbf{1}^{OH}\mathbf{1}^{OH}\mathbf{22}$] ML self assembled from 4 µM $\mathbf{1}^{OH}$ / 6 µM **2** phenyloctane solutions (two samples). (b1, b2) AFM images collected from patterned [$\mathbf{1}^{OH}\mathbf{1}^{OH}\mathbf{22}$] ML (self assembled from 4 µM $\mathbf{1}^{OH}$ / 6 µM **2** phenyloctane solution) incubated with 5µg / mL AuNP for 20 minutes followed by dropwise rinsing with 2 mL hexane (two samples).



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