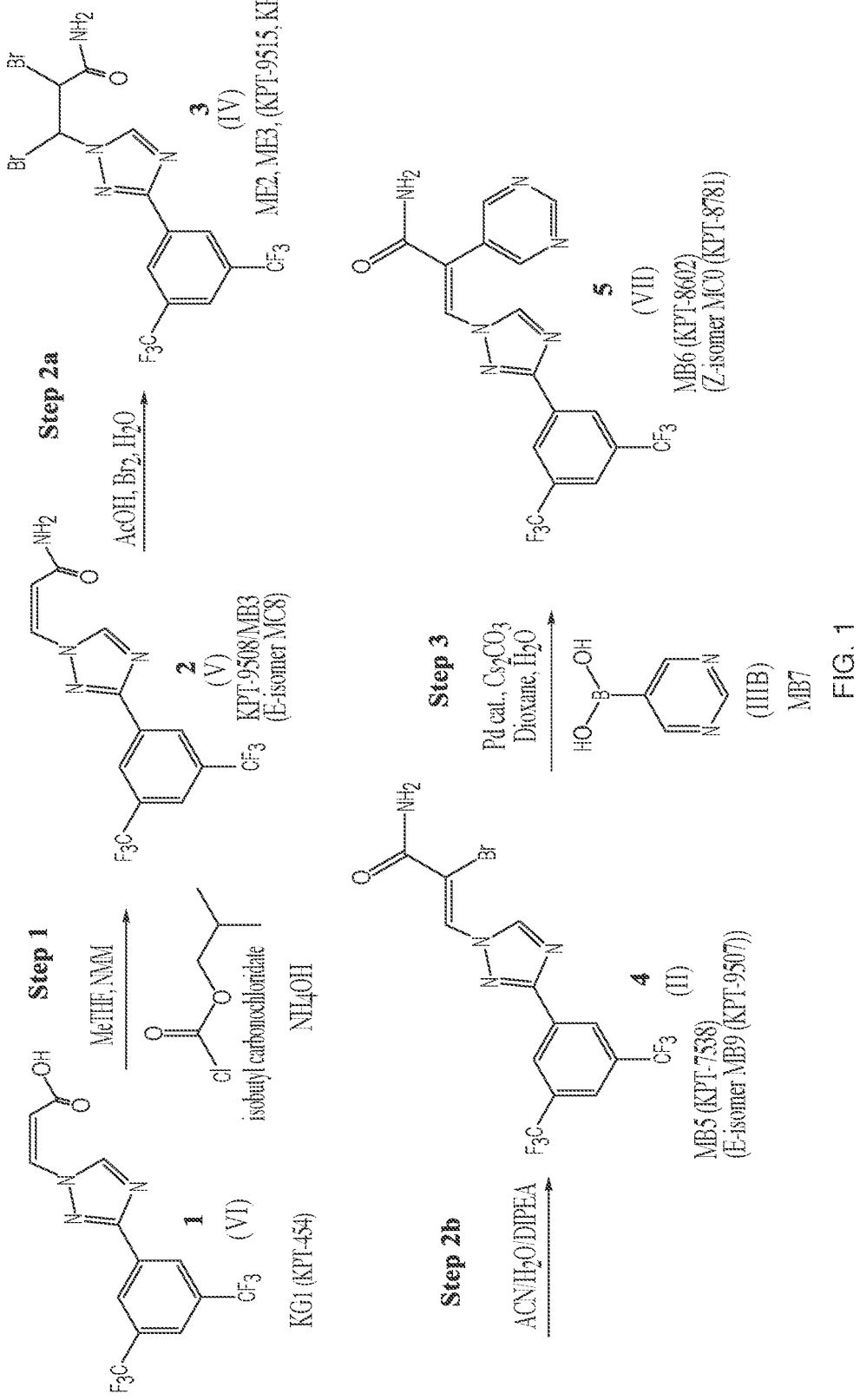

Step 1
MeTHF, NMM
isobutyl carbonochloridate
NH4OH
KG1 (KPT-454)
1
(VI)
Step 2a
AcOH, Br2, H2O
2
(V)
KPT-9508/MB3
(E-isomer MC8)
3
(IV)
ME2, ME3, (KPT-9515, KPT-9506)
Step 3
Pd cat., Cs2CO3
Dioxane, H2O
(IIIB)
MB7
5
(VII)
MB6 (KPT-8602)
(Z-isomer MC0 (KPT-8781)
Step 2b
ACN/H2O/DIPEA
4
(II)
MB5 (KPT-7538)
(E-isomer MB9 (KPT-9507))

FIG. 1

				4 h IPC (%a/a)								24 h IPC (%a/a)								
			TC	MC0	MB6	MB3/ MC8	MB9	MB8	PF9			MC0	MB6	MB3/ MC8	MB9	MB8	PF9			
			KPT#	8781	8602	9508		7838				8781	8602	9508		7838				
			m/z	429	429	351	429/ 431	429/431	699		630	429	429	351	429/ 431	429/431	699		630	
Entry	Exp (1457-PK-)	Catalyst	RT	2.9	3.0	3.2	3.3	3.5	3.9	4.1	4.2	2.9	3.0	3.2	3.3	3.5	3.9	4.1	4.2	Conversion
1	134-R1	PdDPPFCl2DCM (control)		5.2	34.2	15.8	2.3	28.9	2.3	0.0	0.0	71.1	5.4	38.6	18.1	2.3	28.3	3.7	0.0	0.0	71.7	
2	134-R2	QPhosPd(Crotyl)Cl		0.0	7.1	6.0	2.2	77.0	2.4	0.0	0.0	23.0	0.0	11.0	16.0	7.5	54.0	0.0	2.6	7.6	46.0	
3	134-R3	AmPhos Pd(crotyl)Cl		0.0	73.6	1.5	0.0	22.0	2.9	0.0	0.0	78.0	0.0	68.9	1.8	0.0	18.7	3.6	6.8	0.0	81.3	
4	134-R4	[P(tBu)3] Pd(crotyl)Cl		0.3	83.0	2.9	0.0	7.9	2.3	0.0	0.0	92.1	0.0	84.1	3.8	0.0	6.5	2.6	0.0	0.0	93.5	
5	134-R5	XPhos Pd(crotyl)Cl		2.7	17.0	5.8	0.0	61.9	6.2	0.0	0.0	38.1	3.3	22.7	10.6	0.0	36.7	6.7	0.0	0.0	43.3	
6	134-R6	RuPhos Pd(crotyl)Cl		0.0	12.9	15.9	10.7	47.6	0.0	0.0	2.0	52.4	1.8	15.1	22.3	12.7	28.2	1.0	1.7	7.0	71.8	
7	134-R7	SPhos Pd(crotyl)Cl		2.5	29.5	12.0	3.9	43.9	1.1	0.0	0.0	56.1	3.6	47.8	18.4	3.5	19.6	2.3	0.0	0.0	80.4	
8	134-R8	BrettPhos Pd(crotyl)OTf		0.6	4.2	6.1	2.5	86.0	0.0	0.0	0.0	14.0	0.0	7.0	13.9	5.7	62.9	0.0	0.0	9.0	37.1	
9	134-R9	[(R)-BINAP Pd(allyl)]Cl		0.0	0.0	3.6	3.4	83.8	8.2	0.0	0.0	16.2	0.0	0.0	7.8	7.9	67.5	9.0	0.0	4.0	32.5	
10	134-R10	[XantPhos Pd(allyl)]Cl		0.3	4.0	3.4	3.1	83.5	0.0	0.0	0.0	16.5	0.0	9.0	12.2	6.4	38.1	0.0	0.0	9.4	41.9	
11	136-R1	[P(tBu)3] Pd(crotyl)Cl		0.0	87.0	4.8	0.0	4.7	3.3	0.0	0.0	95.3	0.0	87.0	3.7	0.0	2.8	3.1	0.0	0.0	97.2	
12	136-R2	[P(Cy)3] Pd(crotyl)Cl		0.0	41.0	6.5	0.0	46.0	2.1	0.0	0.0	54.0	0.7	67.9	11.2	0.0	5.0	5.3	0.0	0.0	95.0	

FIG. 2

				4 h IPC (%a/a)								
			TC	MC0	MB6	MB3/ MC8	MB9	MB5	PF9			
			KPT#	8781	8602	9508		7538				
			m/z	429	429	351	429/ 431	429/431	699	630		
Entry	Exp (1457-PK-)	Org A (pts.)	Solvent B (pts.)	RT	2.9	3.0	3.2	3.3	3.5	3.9	4.1	4.2	Conversion
1	138-R1	Dioxane (30)	H_2O (6)		5.2	83.0	9.6	0.0	0.0	1.9	0.0	0.0	100.0
2	138-R2	THF (30)	H_2O (6)		0.0	85.0	9.9	0.0	0.0	3.6	0.0	0.0	100.0
3	138-R3	MeTHF (30)	H_2O (6)		0.0	72.0	8.7	0.0	0.0	6.3	0.0	0.0	90.3
4	138-R4	EtOH/MeTHF (45)	H_2O (6)		1.6	75.0	17.0	0.0	0.0	1.8	0.0	0.0	100.0
5	138-R5	Tol/MeTHF (45)	H_2O (6)		0.0	31.8	11.4	0.0	51.5	1.5	0.0	0.0	48.5

Notes:

1. All experiments were carried out using 3 mol% catalyst [P(tBu)3] Pd(croty)Cl loading and 0.5 eq. of Cs_2CO_3 at 70 °C (except THF – reflux).
2. Data was only collected at the 4 hour time point.
3. The impurity with m/z = 351 was initially identified as MB3 based on mass, but was likely MC8 based on retention time and expected stereochemistry of the debromination side reaction. MC8 was identified as the major debromination product by authentic spiking in later experiments.

FIG. 3

						24 h IPC (%a/a)									24 h IPC (%a/a)								
					TC	MC 0	MB 6	MB3 / MC8	MB 9	MB5	PF 9				MC 0	MB 6	MB3 / MC8	MB 9	MB5	PF 9			
					KPT #	8781	8602	9508		7538					8781	8602	9508		7538				
					m/z	429	429	351	429/431	429/431	699		630		429	429	351	429/431	429/431	699		630	
Entry	Exp (1457-PK-)	Catalyst	Org A (pts.)	Base (eq.)	RT	2.9	3.0	3.2	3.3	3.5	3.9	4.1	4.2	Conversion	2.9	3.0	3.2	3.3	3.5	3.9	4.1	4.2	Conversion
1	140-R1	AmPhos Pd(crotyl)Cl	Dioxane (30)	Cs_2CO_3 (0.5)		0.0	70.5	6.6	0.0	19.5	3.3	0	0.0	80.5	ND	ND	ND	ND	ND	ND	ND	ND	ND
2	140-R2	AmPhos Pd(crotyl)Cl	Dioxane (30)	Cs_2CO_3 (1)		0.9	47.9	15.9	3.0	30.1	1.5	0	0.0	69.9	ND	ND	ND	ND	ND	ND	ND	ND	ND
3	144-R1	[P(tBu)3] Pd(crotyl)Cl	MeTHF (30)	Cs_2CO_3 (1)		1.0	63.9	15.0	3.0	15.9	0.0	0.0	0.0	84.1	1.4	69.0	19.8	3.3	3.1	2.3	0.0	0.0	96.9
4	144-R2	[P(tBu)3] Pd(crotyl)Cl	MeTHF (30)	$NaHCO_3$ (2)		0.8	46.9	12.4	3.1	35.5	0.0	0.0	0.0	64.5	1.5	51.6	16.2	3.6	20.1	1.9	0.0	0.0	79.9
5	144-R3	[P(tBu)3] Pd(crotyl)Cl	MeTHF (30)	K_2CO_3 (1)		0.7	55.6	12.1	2.8	25.2	0.0	0.0	1.9	74.8	1.1	68.8	17.9	3.3	5.8	2.2	0.0	0.0	94.2
6	144-R4	[P(tBu)3] Pd(crotyl)Cl	MeTHF (30)	K_3PO_4 (2)		0.7	55.6	12.1	2.8	25.3	0.0	0.0	1.8	74.7	7.1	55.7	21.8	3.4	12.5	1.6	0.0	0.0	87.5

Notes:

1. All experiments were carried out using 3 mol% catalyst loading in 30 pts of solvent A (dioxane or MeTHF) and 6 pts of solvent B (water) at 70 °C.
2. ND = not determined.
3. The impurity with m/z = 351 was initially identified as MB3 based on mass, (LCMS), but was likely MC8 based on retention time and expected stereochemistry of the debromination side reaction. MC8 was identified as the major debromination product by authentic spiking in later experiments.

FIG. 4

No	Reaction Conditions (MB5: 1.0 eq; MB7: 2.0 eq; Cs_2CO_3: 0.5 eq; 5g)	Pd Catalyst	IPC Result (MB5 wrt MB6 % a/a)	MB6 Purity in Reaction Mixture (% a/a)	Major By-products wrt MB6 (% a/a)	Conclusions	Ref.
1	Degassed THF:H_2O 7:1 (16 parts); at reflux (65 °C); 4 hrs	$^tBu_3PPd(crotyl)Cl$, 3%	115	37	MC8: 21.3 PF9: 1.5	1.Reaction stalled 2.High levels of by-products	1473-JZ-001
2	Degassed THF:H_2O 15:2 (34 parts); at reflux (63 °C); 6 hrs	$^tBu_3PPd(crotyl)Cl$, 3%	1.1	80	MC8: 7.9 PF9: 3.7	High levels of by-products	1473-JZ-004
3	Degassed THF:H_2O 15:2 (34 parts); at reflux (63 °C); 8 hrs	$^tBu_3PPd(crotyl)Cl$, 3%	86	43	MC8: 22 PF9: 3.2	High levels of by-products	1473-JZ-009
4	Repeated the above trial using a new lot Pd catalyst	$^tBu_3PPd(crotyl)Cl$, 3%	Almost no reaction	-	-	No reaction	1473-JZ-018
5	Degassed dioxane:H_2O 15:2 (34 parts); 70-76 °C, 4 hrs	$^tBu_3PPd(crotyl)Cl$, 3%	86	37	MC8: 29 PF9: 9.0	1.Reaction stalled 2.High levels of by-products	1473-JZ-007

FIG. 5

No	Reaction Conditions (MB5: 1.0 eq; MB7: 2.0 eq; Cs_2CO_3: 0.5 eq)	Pd Catalyst	IPC Result (MB5 wrt MB6 % a/a)	MB6 Purity in Reaction Mixture (% a/a)	Major By-products wrt MB6 (% a/a)	Conclusions	Ref.
1	Degassed THF:H_2O 15:2 (17 parts); at reflux (64 °C); 7 hrs	Strem Pd 46-0232, 2%	Almost no reaction	-	-	No reaction	1473-JZ-013
2	Degassed THF:H_2O 15:2 (17 parts); at reflux (64 °C); 7 hrs	Strem SPhos Palladacycle Gen. 2 (Cat #46-0283), 2%	Almost no reaction	-	-	No reaction	1473-JZ-015
3	Degassed THF:H_2O 15:2 (17 parts); at reflux (63 °C); 8 hrs	Strem Amphos Palladacycle Gen. 2 (Cat #46-0342), 2%	10.9	76.1	MC8: 3.1 PF9: 13.7	1.Reaction stalled 2.High levels of by-products	1473-JZ-011
4	Degassed THF:H_2O 15:2 (22 parts); at reflux (63 °C); 7 hrs	Strem Amphos Palladacycle Gen. 2 (Cat #46-0342), 3%	4.8	77.6	MC8: 13.9 PF9: 9.7	1.Reaction stalled 2.High levels of by-products	1473-JZ-016
5	Degassed dioxane:H_2O 15:2 (34 parts); 70-76 °C; 4 hrs	Strem Amphos Palladacycle Gen. 2 (Cat #46-0342), 3%	No MB5 detected	89.2	MC8: 1.6 PF9: 3.1	Good reaction	1473-JZ-019
6	Repeated the above trial at 11g scale	Strem Amphos Palladacycle Gen. 2 (Cat #46-0342), 3%	0.1	86.5	MC8: 0.6 PF9: 3.0	Good reaction	1473-JZ-028
7	Degassed dioxane:H_2O 15:2 (34 parts); 60-65 °C; 4 hrs	JM Amphos Pd(crotyl)Cl (Pd-161), 3%	No MB5 detected	88.3	MC8: 1.7 PF9: 3.8	Good reaction	1473-JZ-021

FIG. 6

No	Reaction Conditions (MB7: 2.0 eq; Cs_2CO_3; 0.5 eq; 5g scale)	Amount of Strem Amphos Paladacycle Gen. 2	IPC Result (% a/aMB5 wrt MB6)	MB6 Purity in Reaction Mixture (% a/a)	MC0 wrt MB6 (% a/a)	MC8 wrt MB6 (% a/a)	PF9 wrt MB6 (% a/a)	Note	Ref.
1	Degassed dioxane:H_2O (7:1, 24 parts); 60-65 °C; 4 hrs	3 mol %	0.1	87.7	0.5	2.2	2.6	Good result	1473-JZ-153
2	Degassed dioxane:H_2O (7:1, 24 parts); 60-65 °C; 4 hrs	2 mol %	0.2	85.5	0.3	2.6	3.9	Good result	1473-JZ-155
3	Degassed dioxane:H_2O (7:1, 24 parts); 60-65 °C; 4 hrs	1 mol %	19.0	70.1	0.3	4.5	2.0	Reaction stalled	1473-JZ-157

FIG. 7

No	Reaction Conditions (MB7: 2.0 eq; Cs_2CO_3: 0.5 eq; 5g scale)	Amount of MB7 (Eq)	IPC Result (% a/a MB5 wrt MB6)	MB6 Purity in Reaction Mixture (% a/a)	MC0 wrt MB6 (% a/a)	MC8 wrt MB6 (% a/a)	PF9 wrt MB6 (% a/a)	Note	Ref.
1	3% Strem Pd 46-0342, 24 parts of degassed dioxane: H_2O 7:1, 60-65 °C, 4 hrs	1.5	9.0 8.4 after 7 hours	80.8	0.2	2.0	5.1	Reaction stalled even extending the reaction time	1473-JZ-141
2	3% Strem Pd 46-0342, 34 parts of degassed dioxane: H_2O 7:1, 60-65 °C, 4 hrs	1.5	9.6 8.8 after 9 hours	81.3	0.2	1.9	4.6	Reaction stalled even extending the reaction time	1473-JZ-151
3	2% Strem Pd 46-0342, 24 parts of degassed	1.8	0.6	88.2	0.1	1.7	2.9	Good result, but the residual MB5	1499-JZ-001

FIG. 8

No	Reaction Conditions (2.0 eq MB7; 0.5 eq Cs_2CO_3; 2% Strem Pd 46-0342; 5g scale)	Degassed Solvents	IPC Result (% a/a MB5 wrt MB6)	MB6 Purity in Reaction Mixture (% a/a)	MC0 wrt MB6 (% a/a)	MC8 wrt MB6 (% a/a)	PF9 wrt MB6 (% a/a)	Note	Ref.
1	3% Strem Pd 46-0342; 60-65 °C; 4 hrs	Degassed dioxane:H_2O 7:1 24 parts	0.1	87.7	0.5	2.2	2.6	Good result	1473-JZ-153
2	60-65 °C; 4 hrs	Degassed THF:H_2O 3:1 24 parts	3.6	75.6	0.2	11.0	2.9	Reaction stalled and higher level Impurities	1473-JZ-161
3	60-65 °C; 4 hrs	Degassed THF:H_2O 1:1 24 parts	0.03	79.5	1.6	9.2	2.0	Higher level Impurities	1473-JZ-163
4	60-65 °C; 4 hrs	Degassed MeOH:H_2O 5:1 24 parts	0.6	58.5	4.6	32.1	3.0	Higher level Impurities	1473-JZ-162
5	60-65 °C; 4 hrs 8 hours more	Degassed MeTHF:H_2O 7:1, 24 parts	36.8 14.8	66.4 72.5	0.1 0.1	2.1 2.7	5.5 10.8	Reaction stalled Reaction stalled again and higher level PF9	1473-JZ-169

FIG. 9

No	Reaction Conditions (MB7: 2.0 eq; Cs_2CO_3: 0.5 eq; 2% Strem Pd 46-0342); 5g scale	Degassed Dioxane:H_2O Ratio (v/v)	IPC Result (% a/a MB5 wrt MB6)	MB6 Purity in Reaction Mixture (% a/a)	MC0 wrt MB6 (% a/a)	MC8 wrt MB6 (% a/a)	PI9 wrt MB6 (% a/a)	Note	Ref.
1	24 parts solvents; 60-65 °C; 8 hrs	14:1	6.6	83.5	0.2	1.9	3.4	Reaction stalled even extending the reaction time	1473-JZ-174
			3.9 after 13 hours	84.2 after 13 hours	0.2	2.0	4.4		
2	24 parts solvents; 60-65 °C; 4 hrs	7:1	0.2	85.5	0.3	2.6	3.9	Good reaction	1473-JZ-155
3	24 parts solvents; 60-65 °C; 4 hrs	2:1	0.1	80.9	3.5	6.4	0.9	Higher level Impurities than the above one	1473-JZ-165

FIG. 10

No	Reaction Conditions (2.0 eq MB7; 0.5 eq Cs_2CO_3; 5g scale)	Degassed Dioxane:H_2O 7:1 v/v Parts	IPC Result (% a/a MB5 wrt MB6)	MB6 Purity in Reaction Mixture (% a/a)	MC0 wrt MB6 (% a/a)	MC8 wrt MB6 (% a/a)	PP9 wrt MB6 (% a/a)	Note	Ref.
1	3% Strem Pd 46-0342; 60-65 °C; 4 hrs	34	No MB5 detected	89.2	0.7	1.6	3.1	Good reaction	1473-JZ-019
2	3% Strem Pd 46-0342; 60-65 °C; 4 hrs	24	0.1	87.2	0.5	2.2	3.4	Good reaction	1473-JZ-153
3	2% Strem Pd 46-0342; 60-65 °C; 4 hrs	24	0.2	85.5	0.3	2.6	3.9	Good reaction too, comparing with above one	1473-JZ-155
4	2% Strem Pd 46-0342; 55-60 °C; 5.5 hrs	24	0.1	87.9	0.3	2.1	3.9	Good reaction too, comparing with above one	1473-JZ-180
5	2% Strem Pd 46-0342; 60-65 °C; 6 hrs	18	0.9	86.3	0.1	2.4	4.8	Good reaction, but the residual MB5 close to the limit (<1%)	1473-JZ-172
6	Reducing top volume to 12 parts,	12	9.6	79.0	0.1	2.7	3.5	Reaction stalled. A biphasic mixture	1473-JZ-167

FIG. 11

No	Reaction Conditions (MB7; 2.0 eq; Cs_2CO_3; 0.5 eq; 24 parts of degassed dioxane:H_2O 7:1; 2% Strem Pd 46-0342); 5g scale	Reaction Time Needed	IPC Result (% a/a MB5 wrt MB6)	MB6 Purity in Reaction Mixture (% a/a)	MC0 wrt MB6 (% a/a)	MC8 wrt MB6 (% a/a)	PF9 wrt MB6 (% a/a)	Note	Ref.
1	50-55 °C	14 hrs	1.3	86.8	0.1	2.1	3.1	Cleaner reaction, but required longer time	1473-JZ-177
2	55-60 °C	5.5 hrs	0.1	87.9	0.3	2.1	3.9	Best one	1473-JZ-180
3	60-65 °C	4 hrs	0.2	85.5	0.3	2.6	3.9	Good one	1473-JZ-155
4	Reflux (71-72 °C)	4 hrs	0.1	79.4	5.8	3.0	4.8	Much higher level of MC0	1473-JZ-176

FIG. 12

No	Reaction Conditions	Time for Reaction Mixture Degassing After Reagents Combined	IPC Result (% a/a MB5 wrt MB6)	MB6 Purity in Reaction Mixture (% a/a)	MC0 wrt MB6 (% a/a)	MC8 wrt MB6 (% a/a)	PF9 wrt MB6 (% a/a)	Note	Ref.
1	MB7: 2.0 eq; Cs_2CO_3: 0.5 eq; 24 parts of degassed dioxane:H_2O 7:1; 2% Strem Pd 46-0342; 55-60 °C; 8.5 hrs; 5g scale	55 min.	N/D	85.9	0.1	2.4	3.9	Good reaction	1473-IZ-180
2	MB7: 2.0 eq; Cs_2CO_3: 0.5 eq; 24 parts of degassed dioxane:H_2O 7:1; 2% Strem Pd 46-0342; 55-60 °C; 7 hrs; 5g scale	0 min.	0.08	89.2	0.1	2.0	2.9	Comparable one, even though no degassing of the reaction mixture before heating	1473-IZ-203

FIG. 13

No	Reaction Conditions (2.0 eq MB7; 2% Strem Pd 46-0342; 5g scale)	Base	IPC Result (% a/a MB5 wrt MB6)	MB6 Purity in Reaction Mixture (% a/a)	MC0 wrt MB6 (% a/a)	MC8 wrt MB6 (% a/a)	PP9 wrt MB6 (% a/a)	Note	Ref.
1	Degassed dioxane:H_2O (7:1, 24 parts); 60-65 °C; 4 hrs	Cs_2CO_3 0.5 eq	0.1	87.7	0.5	2.2	2.6	Good reaction	1473-JZ-180
2	Degassed dioxane:H_2O (7:1, 24 parts); 60-65 °C; 4 hrs	K_3PO_4 0.5 eq	29.4	67.0	0.1	2.9	5.2	Reaction stalled	1473-JZ-159
3	Degassed dioxane:H_2O (7:1, 24 parts); 60-65 °C; 7.5 hrs	Cs_2CO_3/CsHCO_3 0.25 eq each	34.0	65.3	0.1	2.9	3.7	Reaction stalled	1499-JZ-026
	Charged more $CsHCO_3$ and heated for 5 more hrs	0.25 eq more $CsCO_3$	N/D after 5 hrs	84.5 after 5 hrs	0.1	2.7	5.7	Good reaction after charging more base	
4	Degassed dioxane:H_2O (7:1, 24 parts); 55-60 °C; 7.5 hrs	Cs_2CO_3/CsOH 0.25 eq/0.50 eq	N/D	86.8	0.1	2.4	3.8	As good as Cs_2CO_3. Similar impurity profile	1499-JZ-034

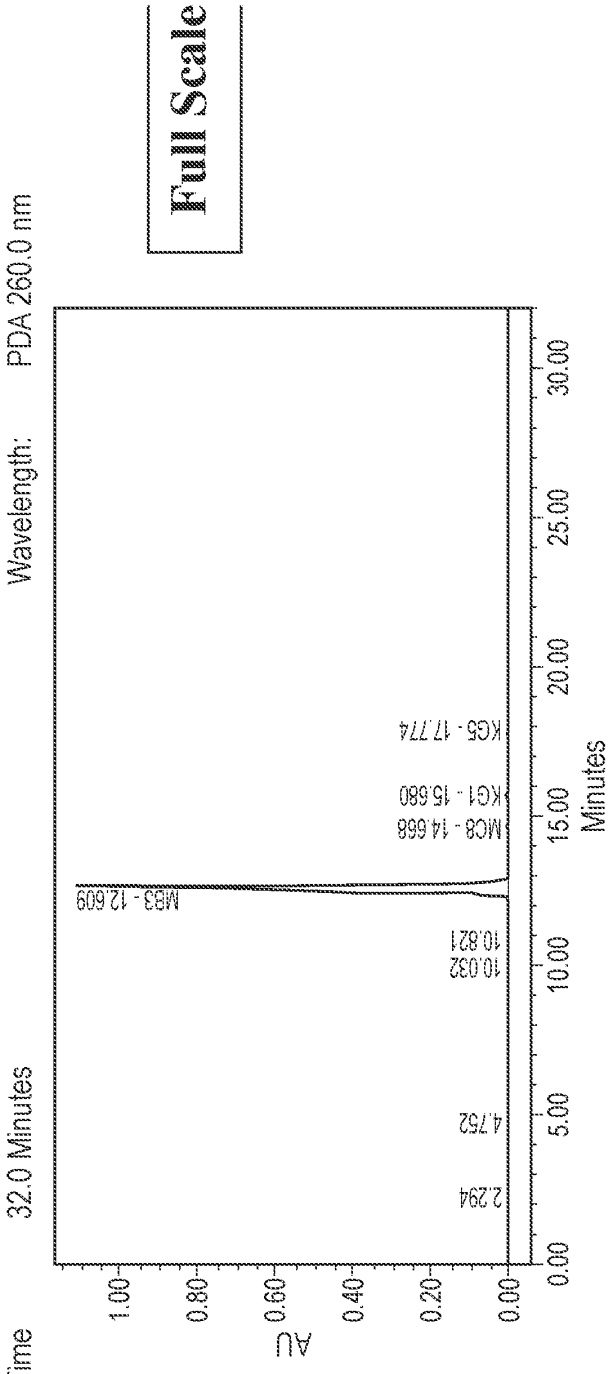
FIG. 14

Entry	Parameter	Change	Comment
1	Catalyst Type	Catalyst changed from tetrakis(triphenylphosphine)palladium to Strem Amphos Palladacycle Gen. 2, cat. #46-0342	High conversion, less side reactions
2	Catalyst loading	Loading Reduced from 10 mol% to 2 mol%	Potential reduce in cost and lower burden on Pd removal
3	MB7 eq.	Increased form 1.5 eq. to 2.0 eq.	Increased cost and burden on isolation purging Offset by significant increase in performance and yield. Purging studies indicate that MB7 can be effectively purged
4	Cs_2Co_3 eq.	Decreased from 0.75 eq. to 0.50 eq.	Reduced cost and lower side reactions
5	Dioxane volume	Increased from 16 pts to 21 pts.	Ensures single phase reaction to improve robustness
6	Water Volume	Increased from 2 pts to 3 pts	Ensures single phase

FIG. 15A

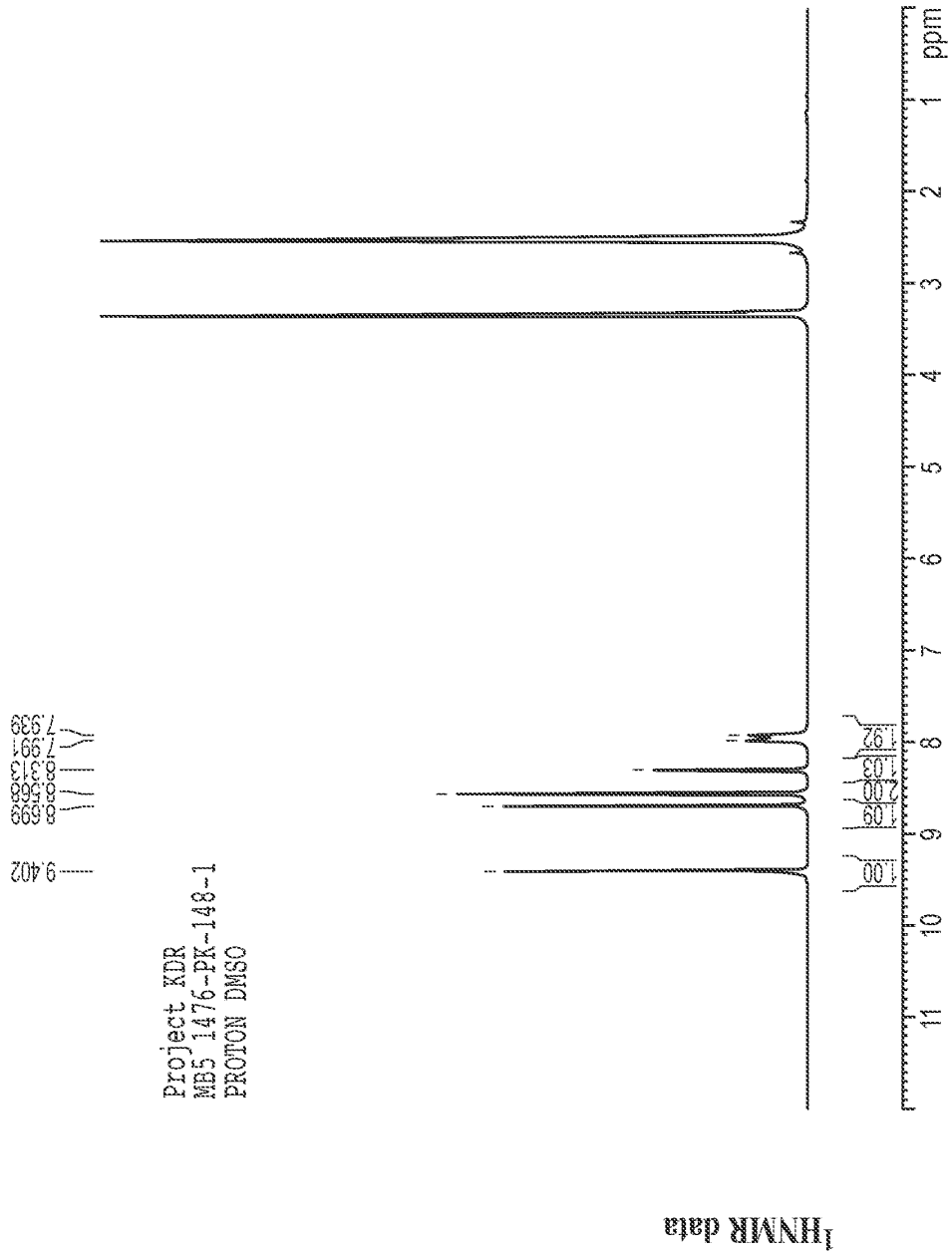
Entry	Parameter	Change	Comment
			reaction to improve robustness
7	Reaction Temperature	Reduced from 70-75°C to 55-60 °C	Less energy intensive and lower side reactions
8	Reaction Time	Reduced from 24 hours to ~7 hours	Reduced cycle times.

FIG. 15B



	Name	RT	Area	% Area	In: Type	USP Plate Count	USP Tailing	USP Resolution
1		2.294	168.44	0.00	bb	3224	0.867	
2		4.752	1488.90	0.01	bb	11843	0.812	16.677
3		10.032	534.76	0.00	bb	38200	0.821	21.444
4		10.821	459.18	0.00	bb	34997	0.661	2.742
5	MB3	12.609	13898415.37	98.27	bv	23056	0.794	5.719
6	MC8	14.668	62154.46	0.44	vb	36375	0.906	6.595
7	KG1	15.680	140789.13	1.00	bb	50950	0.971	3.521
8	KG5	17.774	38882.03	0.27	bb	70027	0.937	7.719
Sum			14142892.3					

FIG. 16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0.050
0.048
0.046
0.044
0.042
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0.038
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0.024
0.022
0.020
0.018
0.016
0.014
0.012
0.010
0.008
0.006
0.004
0.002
0.000
ppm
1
2
3
4
5
6
7
8
9
10
11
1.00
1.09
2.00
1.03
1.92

FIG. 17

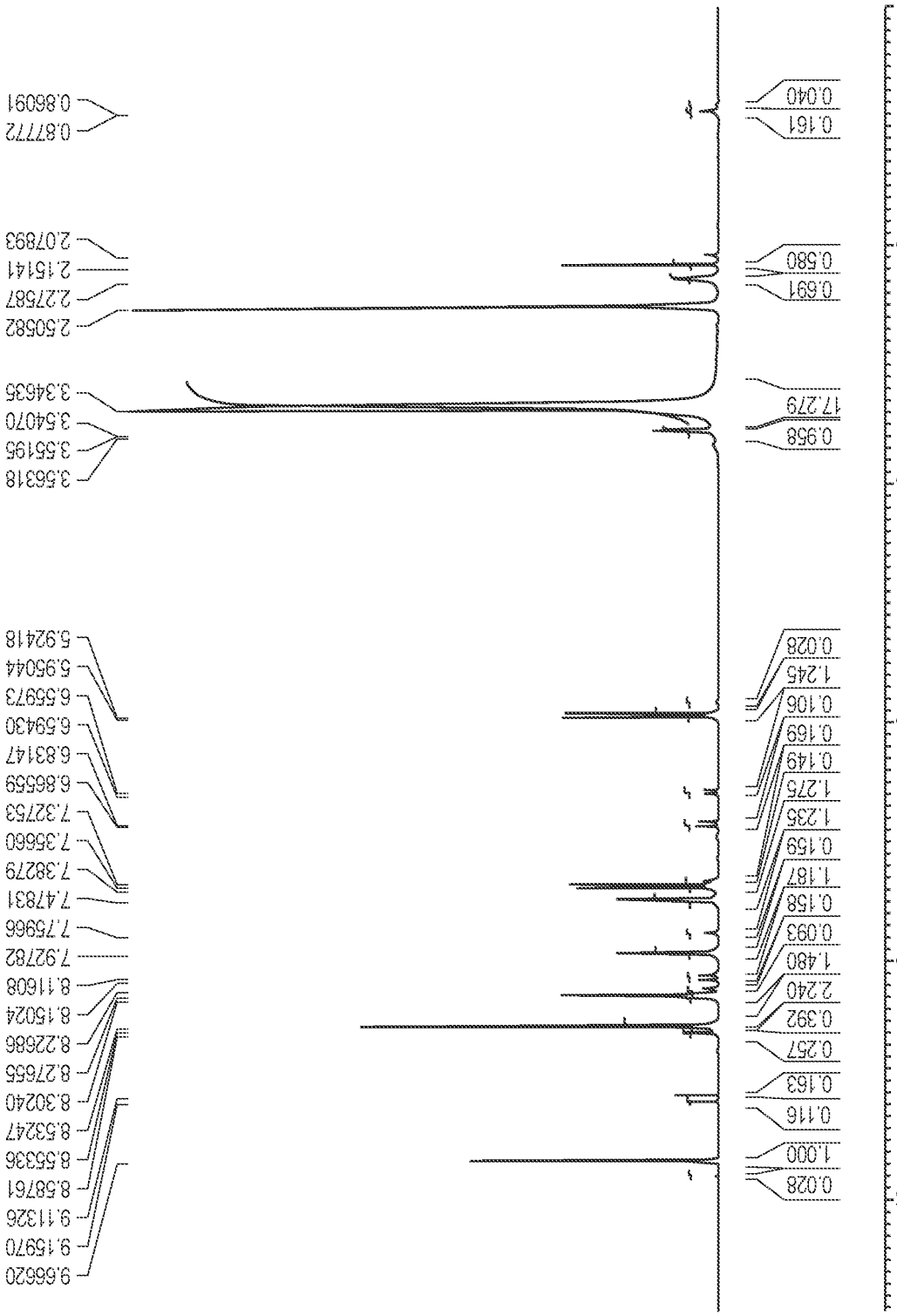
0.86091
0.87772
2.07893
2.15141
2.27587
2.50582
3.34635
3.54070
3.55195
3.56318
5.92418
5.95044
6.55973
6.59430
6.83147
6.86559
7.32753
7.35660
7.38279
7.47831
7.75966
7.92782
8.11608
8.15024
8.22686
8.27655
8.30240
8.53247
8.55336
8.58761
9.11326
9.15970
9.56620
0.040
0.161
0.580
0.691
17.279
0.958
0.028
1.245
0.106
0.169
0.149
1.275
1.235
0.159
1.187
0.158
0.093
1.480
2.240
0.392
0.257
0.163
0.116
1.000
0.028
2
4
6
8
10

FIG. 18

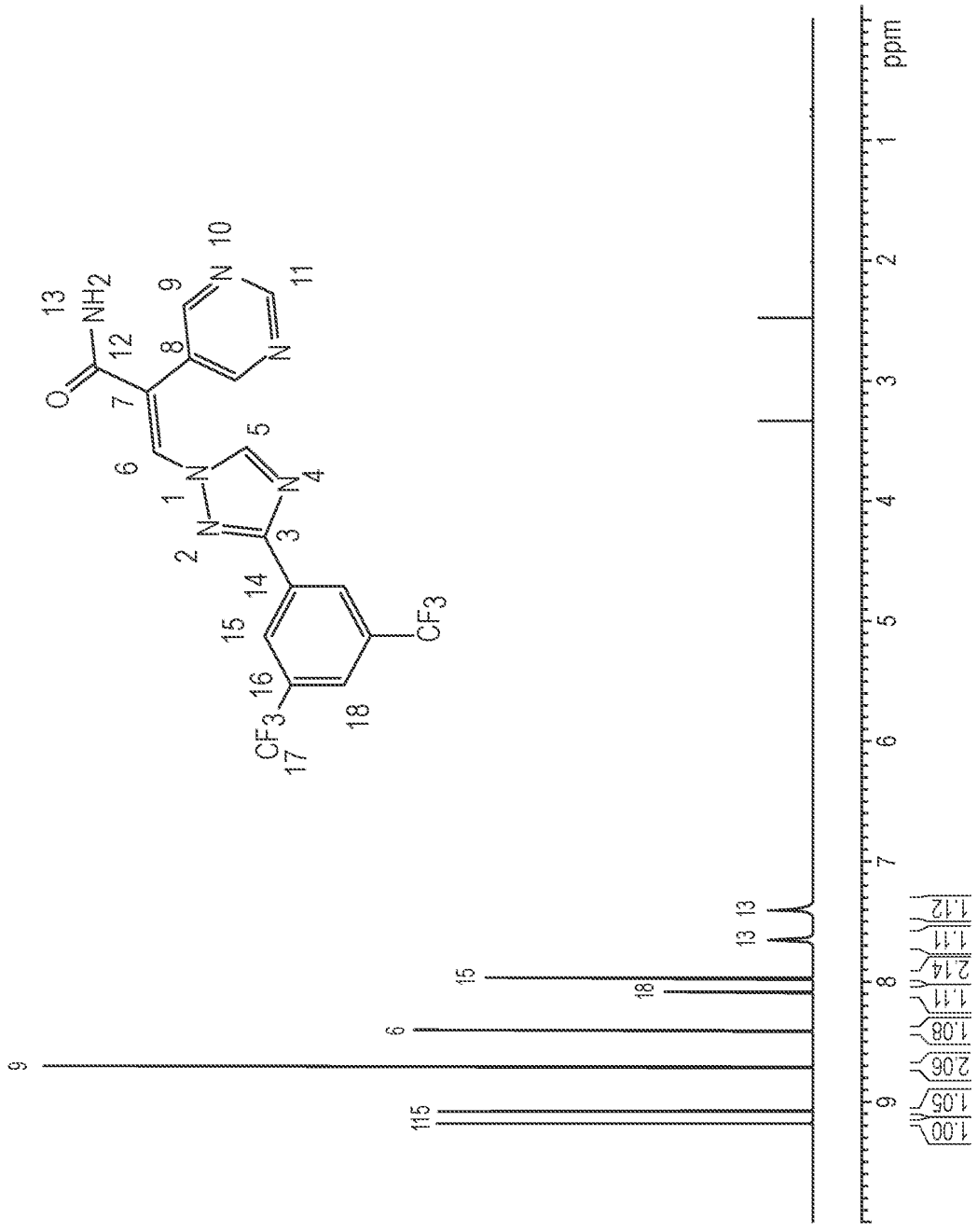

FIG. 19

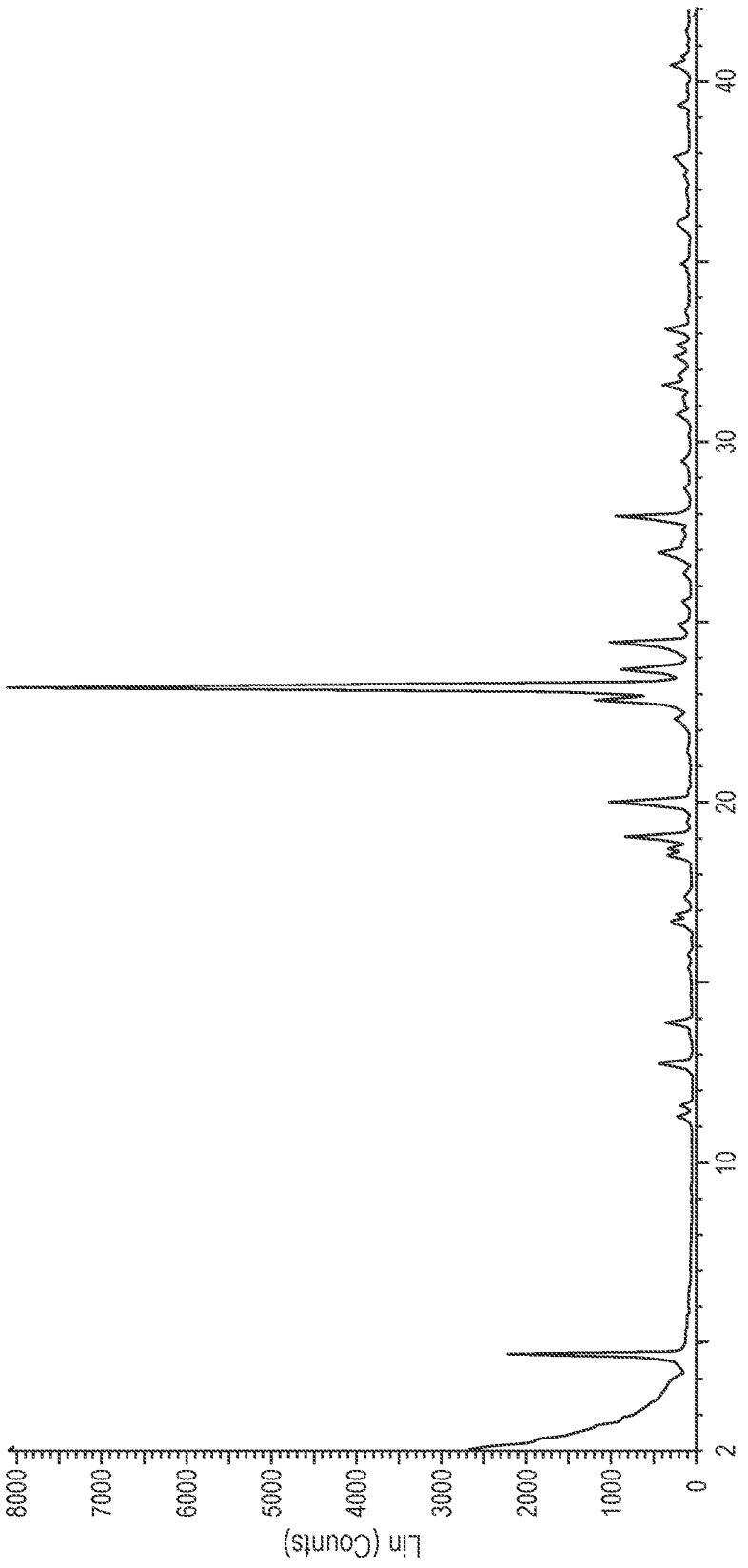
Lin (Counts)
8000
7000
6000
5000
4000
3000
2000
1000
0
2
10
20
30
40
2-Theta - Scale

FIG. 20

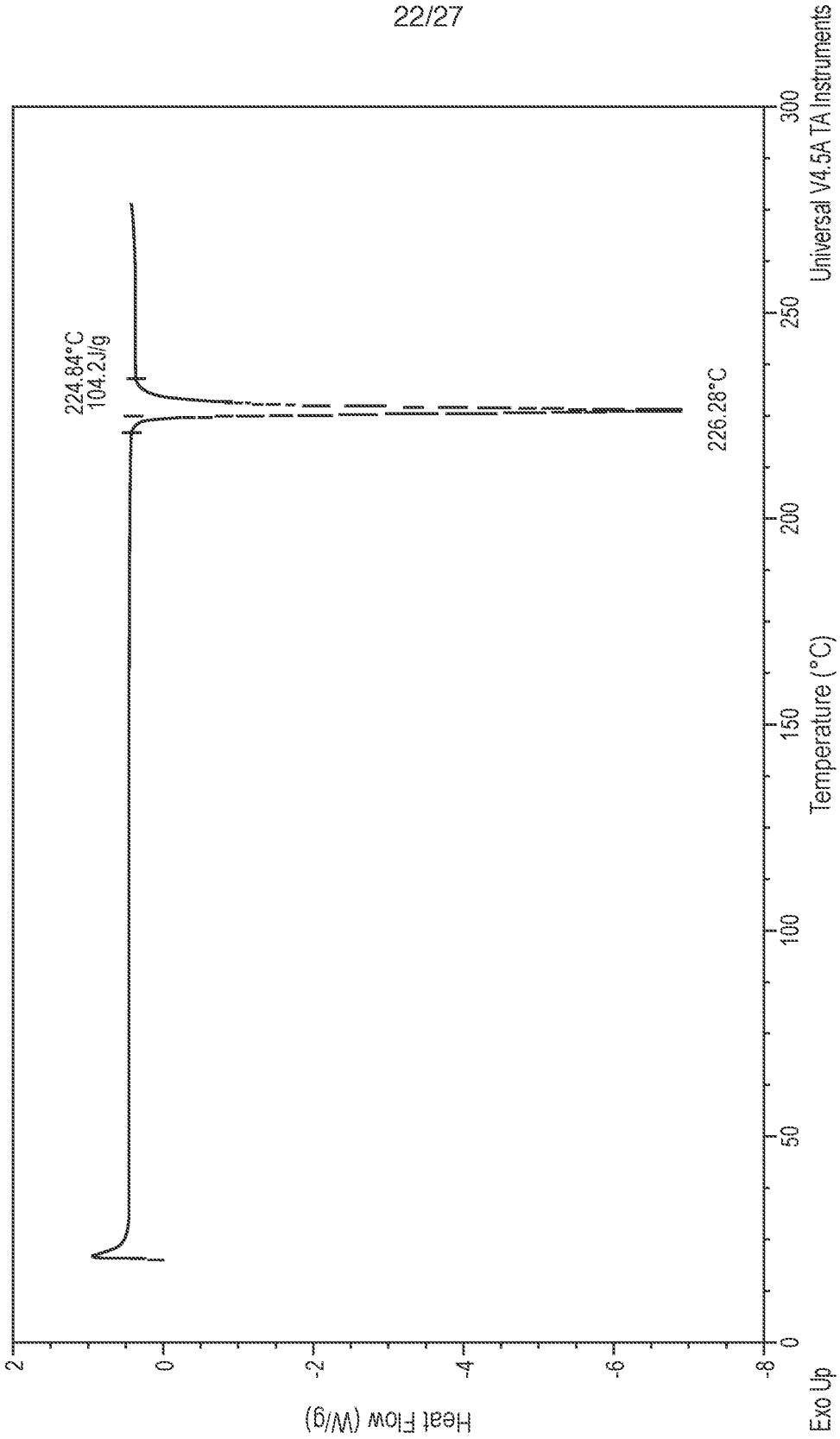
224.84°C
104.2J/g
226.28°C
Heat Flow (W/g)
Exo Up
Temperature (°C)
Universal V4.5A TA Instruments

FIG. 21

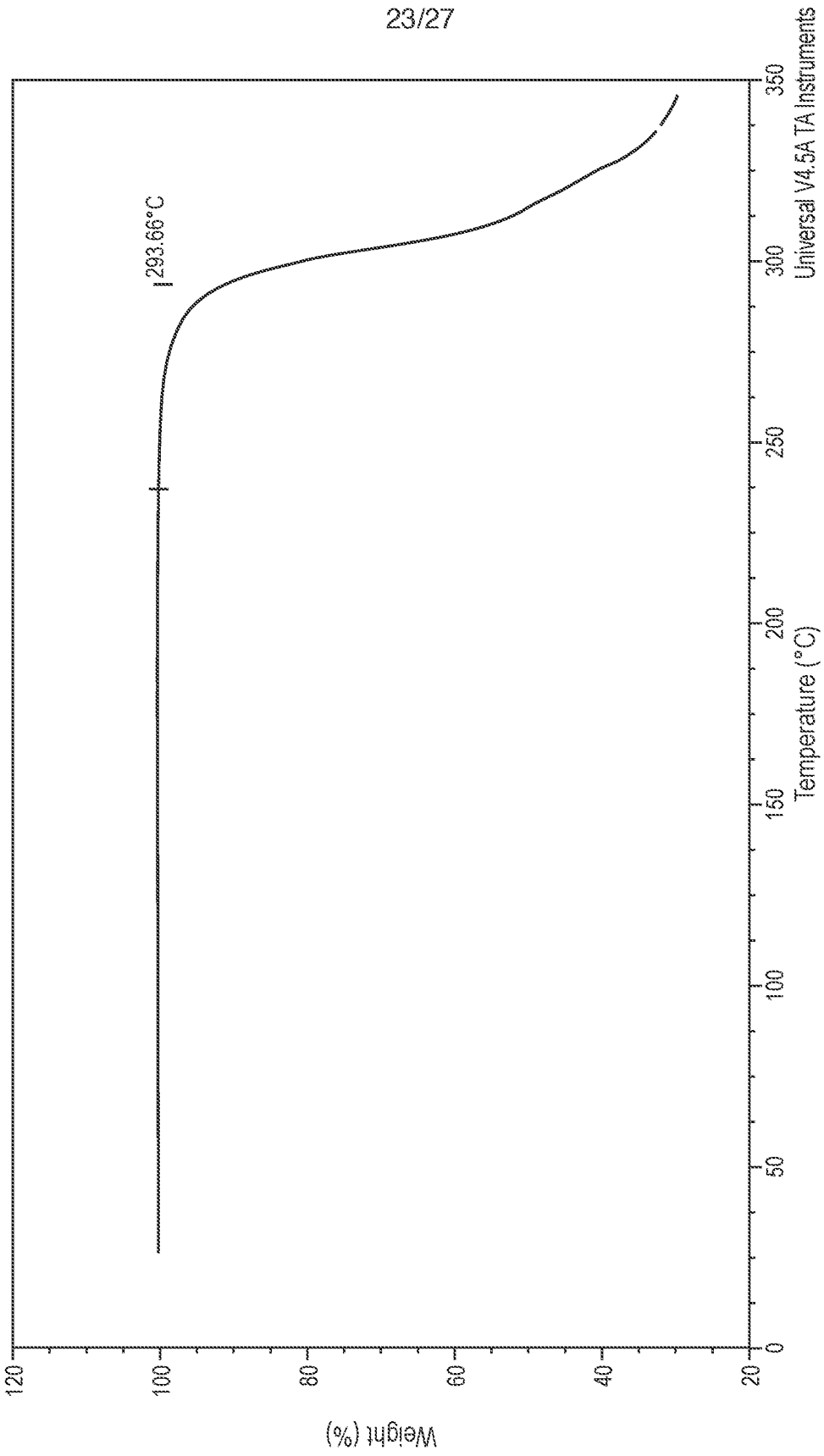
293.66°C
Weight (%)
120
100
80
60
40
20
0
50
100
150
200
250
300
350
Temperature (°C)
Universal V4.5A TA Instruments

FIG. 22

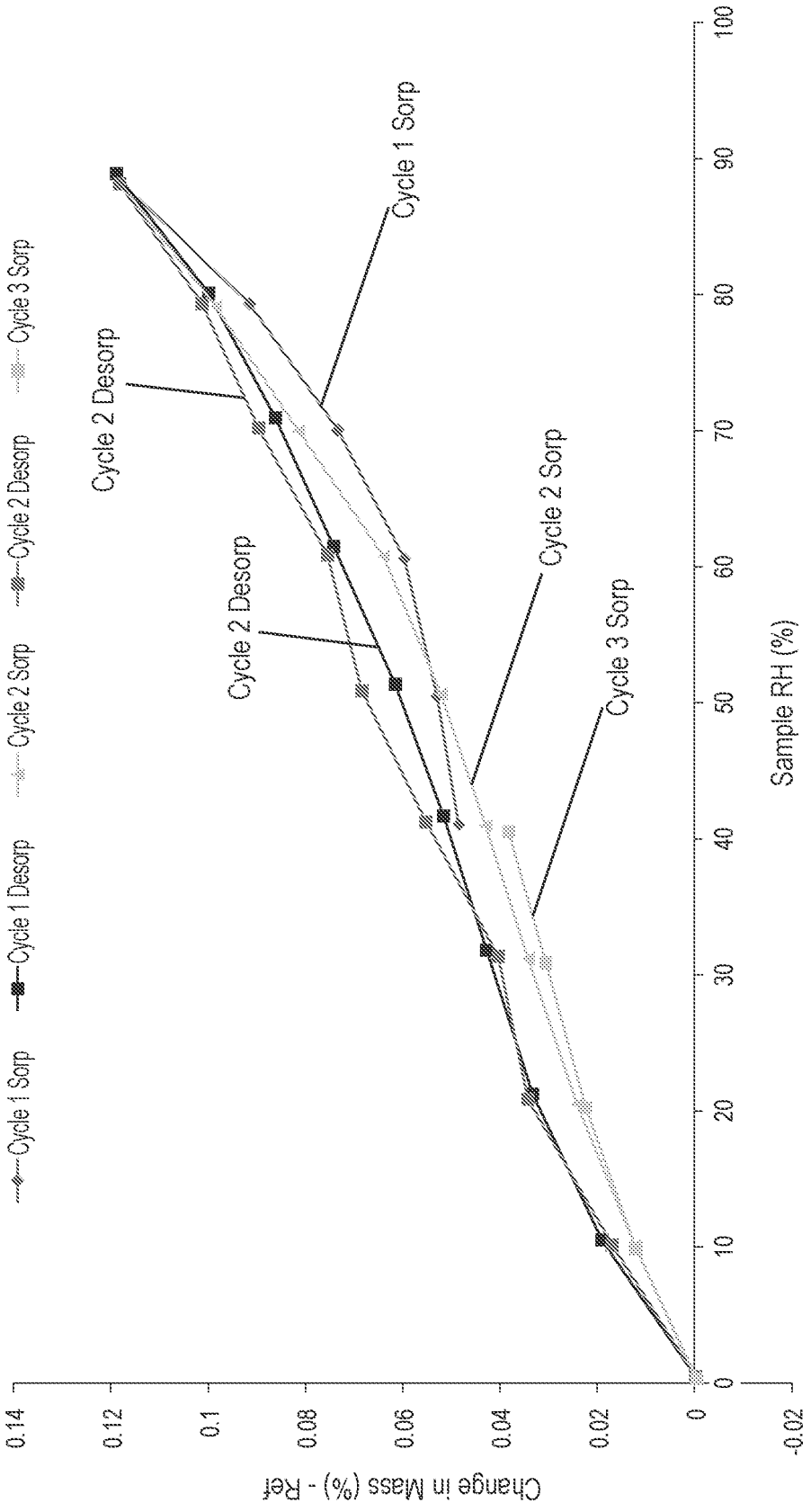
Cycle 1 Sorp
Cycle 1 Desorp
Cycle 2 Sorp
Cycle 2 Desorp
Cycle 3 Sorp
Cycle 1 Sorp
Cycle 2 Desorp
Cycle 2 Desorp
Cycle 2 Sorp
Cycle 3 Sorp
Change in Mass (%) - Ref
0.14
0.12
0.1
0.08
0.06
0.04
0.02
0
-0.02
0
10
20
30
40
50
60
70
80
90
100
Sample RH (%)

FIG. 23

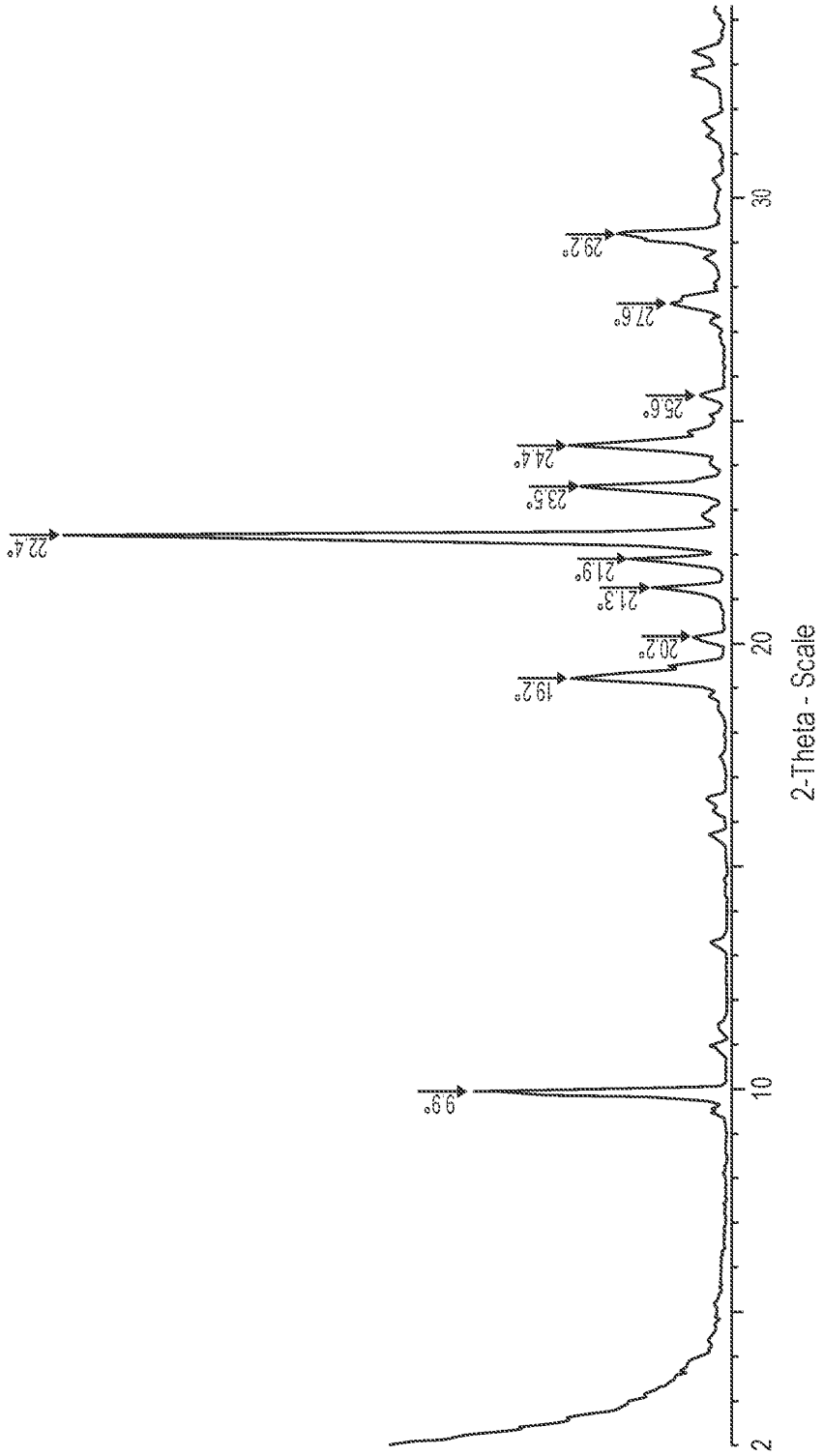
9.9°
19.2°
20.2°
21.3°
21.9°
22.4°
23.5°
24.4°
25.6°
27.6°
29.2°
2
10
20
30
2-Theta - Scale

FIG. 24

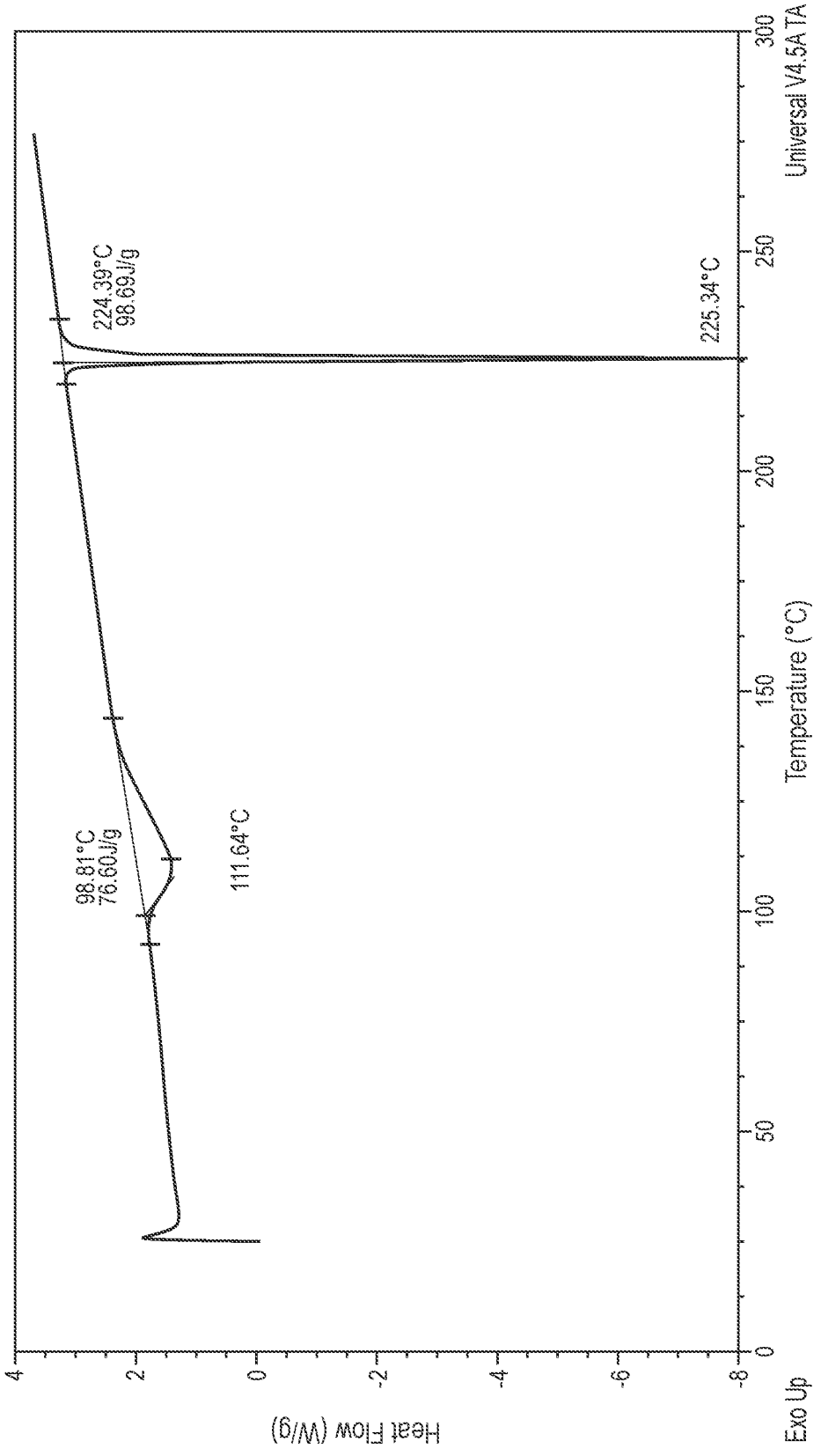
224.39°C
98.69J/g
225.34°C
98.81°C
76.60J/g
111.64°C
Heat Flow (W/g)
Exo Up
Temperature (°C)
Universal V4.5A TA

FIG. 25

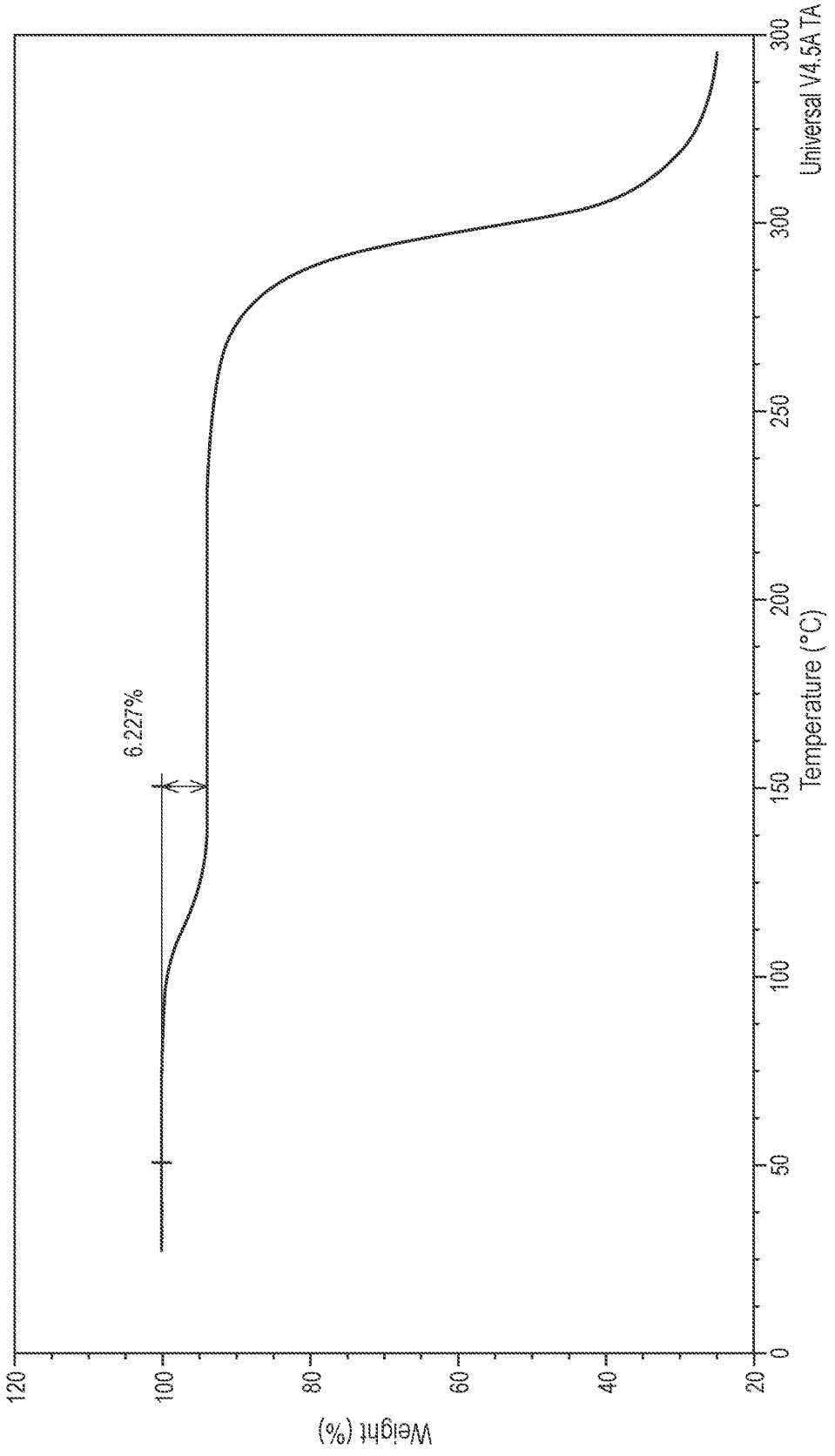
Weight (%)
120
100
80
60
40
20
0
50
100
150
200
250
300
Temperature (°C)
Universal V4.5A TA
6.227%

FIG. 26