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SUPPORTING INFORMATION

Organocatalytic Asymmetric Michael/Hemiacetalization/Acyl Transfer Reaction of α-Nitroketones with *o*-Hydroxycinnamaldehydes: Synthesis of 2,4-Disubstituted Chromans

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Table Contents

1. General procedure for the synthesis compound (1)	S 1
2. General procedure for the synthesis of compounds (2)	S 1
3. Reference	S 1
4. Preliminary Catalyst screening (Table-1)	S2
5. Acid screening (Table-2)	S3
6. Solvent Screening (Table-3)	S 3
7. Concentration and Temperature screening (Table-4)	S4
8. Scale up reaction	S5
9. Crystal data	S5
10. NMR Spectra	S7
11. HPLC Spectra	S41

1. General procedure for the synthesis of compound (1):



To the stirred solution of salicylaldehyde in DCM was added (triphenylphosphoranylidene) acetaldehyde at 0 °C, and the resulting mixture was warmed to room temperature. Upon complete consumption of the starting material (monitored by TLC, 6–12 h). The intermediate products **1** were purified by flash column chromatography on silica gel (Hexane/Ethyl acetate).

2. General procedure for the synthesis of compound (2):



Nitro methane (3 mmol), corresponding aldehyde (1 mmol) and imidazole were mixed in 2 ml of distilled water. The heterogeneous reaction mixture was stirred at ambient temperature for 24 hr. After completion of the reaction, the products were extracted with 10 ml of diethyl ether. The organic layer was dried over Na₂SO₄ then concentrated in vacuo. The intermediate products **B** were purified by flash column chromatography on silica gel (Hexane/Ethyl acetate). To a stirred solution of intermediate product **B** (1 mmol) in DCM (3 ml) was added PCC (2 mmol) in portions. The resulting suspension was stirred at rt for 12 hr. The mixture was passing though short silica gel then concentrated in vacuo. The products **2** were purified by flash column chromatography (hexane/ethyl acetate).

3. Reference:

- 1. B. Padhi, D. S. Reddy, D. K. Mohapatra, Eur. J. Org. Chem., 2015, 542.
- 2. M. Phukan, K. Jyoti, R. Borah, Synthetic Communications, 2008, 38, 3068.
- 3. M. Zhou, D. Dong, B. Zhu, H. Geng, Y. Wang, X. Zhang, Org. Lett., 2013, 15, 5524.

4. Preliminary catalyst screening (Table-1):



entry ^a	catalyst	yield $(\%)^b$	dr^c	ee (%) ^d
1	Ι	80	>20:1	56
2	II	85	>20:1	78
3	III	84	>20:1	72
4	IV	82	>20:1	78
5	V	Complex mix.	>20:1	-
6	VI	75	>20:1	52
7	VII	70	>20:1	65
8	VIII	85	>20:1	-

9	IX	80	>20:1	-
10	X	85	>20:1	-
11	XI	60	>20:1	30
12 ^e	II	82	>20:1	52
13 ^f	II	80	>20:1	42

"Reactions were carried out with 0.25 mmol of 1a and 0.05 mmol of 2a in DCE (0.5 ml) at 0 °C for 2 days, ^bIsolated yield after silica gel column chromatography, ^cdr was determined by ¹H NMR, ^dee was determined by HPLC, ^{*e*}both are same eq. and f_2 eq. of **1**.

5. Acid Screening (Table-2):



entry ^a	Acid	yield $(\%)^b$	dr^c	$ee~(\%)^d$
1	PhCO ₂ H	85	>20:1	78
2	2F-PhCO ₂ H	77	>20:1	76
3	3NO ₂ -PhCO ₂ H	75	>20:1	70
4	AcOH	80	>20:1	78
5	NaOAc	-	>20:1	-

^aReactions were carried out with 0.25 mmol of 1a and 0.05 mmol of 2a in DCE (0.5 ml) at rt for 2 days, ^bIsolated yield after silica gel column chromatography, ^cdr was determined by ¹H NMR and ^dee was determined by HPLC.

6. Optimization of solvent Screening (Table-3):



2a

NO₂

II (20 mol%) PhCOOH (20 mol%) Solvent, RT, 2 d



entry ^a	solvent	yield $(\%)^b$	dr ^c	ee (%) ^d
1	CH ₂ Cl ₂	85	>20:1	72
2	DCE	87	>20:1	78

3	PhCH ₃	80	>20:1	68
4	MTBE	-	-	-
5	1 4 Dioxane	70	>20:1	45

^{*a*}Reactions were carried out with 0.25 mmol of **1a** and 0.05 mmol of **2a** in solvent (0.5 ml) at rt for 2 days, ^{*b*}Isolated yield after silica gel column chromatography, ^{*c*}dr was determined by ¹H NMR and ^{*d*}ee was determined by HPLC.

7. Optimization of temperature and concentration (Table-4):

0



ll (20 mol%) PhCOOH (20 mol%) DCE, Temp, time, Cocn.



entry ^a	Temp	Conc.(ml)	Time(day)	yield $(\%)^b$	dr ^c	ee (%) ^d
1	RT	0.5	2d	85	>20:1	78
2	0 °C	0.5	2d	50	>20:1	90
3	-20 °C	0.5	2d	40	>20:1	94
4	-20 °C	0.5	7d	85	>20:1	94
5	-40 °C	0.5	7d	-	>20:1	-
6	-20 °C	0.25	7d	85	>20:1	93
7	-20 °C	1	7d	85	>20:1	92
8 ^e	-20 °C	0.5	7d	85	>20:1	90
9 ^f	-20 °C	0.5	7d	85	>20:1	89

^aReactions were carried out with 0.25 mmol of **1a** and 0.05 mmol of **2a** in solvent, ^bIsolated yield after silica gel column chromatography, ^cdr was determined by ¹H NMR, ^dee was determined by HPLC, ^e4 eq. of **1a** and ^f6 eq. mmol of 1a.

8. Scale up reaction:



9. Crystal information:

Crystal data and structure refi	nement for 3p
Identification code	3p (CCDC 1581364)
Empirical formula	C ₁₇ H ₁₄ ClNO ₅
Formula weight	347.74
Temperature/K	569(2)
Crystal system	orthorhombic
Space group	P 21 21 21
a/Å	7.5946(3)
b/Å	13.4938(6)
c/Å	15.8269(7)
α/°	90.00
β/°	90.00
$\gamma/^{\circ}$	90.00
Volume/Å ³	1621.94(12)
Z	4
$\rho_{calc}mg/mm^3$	1.424
m/mm ⁻¹	0.262
F(000)	720.0
Crystal size/mm ³	$0.28 \times 0.24 \times 0.21$
2Θ range for data collection	5.94 to 50°
Index ranges	$-9 \le h \le 8, -7 \le k \le 16, -18 \le l \le 14$
Reflections collected	4970
Independent reflections	2760[R(int) = 0.0211]
Data/restraints/parameters	2760/0/217

Crystal data and ſ. c: -

Goodness-of-fit on F ²	0.991
Final R indexes [I>= 2σ (I)]	$R_1 = 0.0433, wR_2 = 0.1073$
Final R indexes [all data]	$R_1 = 0.0577, wR_2 = 0.1193$
Largest diff. peak/hole / e Å ⁻³	0.18/-0.16
Flack parameter	0.04(10)



Ortep diagram of compound 3p

10. NMR Spectra


































































Compound: 3z⁵ ¹H,¹³C spectra



11. HPLC Spectra:

RM-AMS-1R



No.	Peak Name	Ret.Time (detected)	Area	Rel.Area(ident.)	Height	Amount
		min	mAU*min	%	mAU	
	34 1	20.52	279.4747	50.1472899	585.2813	n.a.
	35 2	23.05	277.833	49.8527101	462.042	n.a.

RM-AMS-1C



No.	Peak Name	Ret.Time (detected)	Area	Rel.Area(ident.)	Height	Amount
		min	mAU*min	%	mAU	
	48 1	20.53	306.9522	96.73848084	616.5783	n.a.
	50 2	23.41	10.349	3.261519156	20.201	n.a.

RM-AMS-5R



No.	Peak Name	Ret.Time (detected)	Area	Rel.Area(ident.)	Height	Amount
		min	mAU*min	%	mAU	
	10 1	29.36	29.95109	50.23596111	39.93468	n.a.
	19 2	37.30	29.670	49.76403889	30.000	n.a.
	RM-AMS-5C					



No.	Peak Name	Ret.Time (detected)	Area	Rel.Area(ident.)	Height	Amount
		min	mAU*min	%	mAU	
	45 1	29.78	20.71624	95.6083648	21.27015	n.a.
	48 2	37.72	0.952	4.391635197	1.016	n.a.





No.	Peak Name	Ret.Time (detected)	Area	Rel.Area(ident.)	Height	Amount
		min	mAU*min	%	mAU	
	28 1	28.82	134.6121	50.54057912	181.8149	n.a.
	30 2	33.92	131.733	49.45942088	168.163	n.a.

RM-AMS-6C



No.		Peak Name	Ret.Time (detected)	Area	Rel	.Area(ident.)	Height	Amount
			min	mAU*min	%		mAU	
	146	1	28.02	81.66763		94.86015206	116.4858	n.a.
	147	2	33.27	4.425		5.1395261	5.430	n.a.





No.		Peak Name	Ret.Time (detected))	Area	Re	I.Area(ident.)	Height	Amount
			min		mAU*min	%		mAU	
	133	1	22.4	13	191.2725		49.67860443	313.9121	n.a.
	134	2	24.6	69	193.747		50.32139557	298.767	n.a.

RM-AMS-10C



No.	Peak Name	Ret.Time (detected)	Area	Rel.Area(ident.)	Height	Amount
		min	mAU*min	%	mAU	
	112 1	22.40	120.1423	94.91230482	196.8074	n.a.
	113 2	24.75	6.440	5.087695179	11.351	n.a.

RM-AMS-7R



No.		Peak Name	Ret.Time (dete	cted)	Area	Rel	.Area(ident.)	Height	Amount
			min		mAU*min	%		mAU	
	22	1		38.16	91.89341		50.34122326	88.81178	n.a.
	31	2		47.10	90.648		49.65877674	73.493	n.a.
		RM-AMS-	-7C						



No.	Peak Name	Ret.Time (detected)	Area	Rel.Area(ident.)	Height	Amount
		min	mAU*min	%	mAU	
	58 1	38.43	8.661688	95.98470805	9.00722	n.a.
	69 2	47.85	0.362	4.015291953	0.354	n.a.





No.	Peak Name	Ret.Time (detected)	Area	Rel.Area(ident.)	Height Amo	ount
		min	mAU*min	%	mAU	
5	54 1	33.61	111.3611	50.43945557	125.8012 n.a.	
7	7 2	45.27	109.421	49.56054443	96.576 n.a.	
	RM-AMS-30)C				



No.	Peak Name	Ret.Time (detected)	Area	Rel.Area(ident.)	Height	Amount
		min	mAU*min	%	mAU	
	46 1	33.46	41.07331	95.21467846	49.95123	n.a.
	61 2	44.86	2.064	4.785321535	2.090	n.a.

RM-AMS-21R



No.	Peak Name	Ret.Time (detected)	Area	Rel.Area(ident.)	Height	Amount
		min	mAU*min	%	mAU	
	58 A	28.48	289.5492	51.90623962	395.4444	n.a.
	70 B	38.63	268.282	48.09376038	275.517	n.a.

RM-AMS-21C



No.	Peak Name	Ret.Time (detected)	Area	Rel.Area(ident.)	Height	Amount
		min	mAU*min	%	mAU	
	25 1	28.97	47.96025	97.58533057	62.16524	n.a.
	35 2	39.23	1.187	2.414669433	1.564	n.a.





No.		Peak Name	Ret.Time (deter	cted)	Area	Rel	.Area(ident.)	Height	Amount
			min		mAU*min	%		mAU	
	169	1		28.05	88.05407		49.55878082	117.3973	n.a.
	198	2		36.58	89.622		50.44121918	99.858	n.a.

RM-AMS-27C



No.	Peak Name	Ret.Time (detected)	Area	Rel.Area(ident.)	Height	Amount
		min	mAU*min	%	mAU	
	50 1	28.06	48.50877	96.16414248	58.76765	n.a.
	59 2	36.93	1.935	3.835857522	2.296	n.a.

RM-AMS-26R



No.		Peak Name	Ret.Time (dete	cted)	Area	Rel	.Area(ident.)	Height	Amount
			min		mAU*min	%		mAU	
	111	1		22.62	142.5305		50.52743888	246.1211	n.a.
	119	2		29.52	139.555		49.47256112	184.588	n.a.
		RM-AMS-260	>						



No.	Peak Name	Ret.Time (detected)	Area	Rel.Area(ident.)	Height	Amount
		min	mAU*min	%	mAU	
	28 1	22.37	65.90059	97.76297079	107.7109	n.a.
	31 2	29.96	1.508	2.23702921	2.034	n.a.





No.	Peak Name	Ret.Time (detected)	Area	Rel.Area(ident.)	Height	Amount
		min	mAU*min	%	mAU	
	59 1	34.92	175.2922	49.917343	192.712	n.a.
	75 2	46.61	175.873	50.082657	149.484	n.a.

RM-AMS-31C



No.	Peak Name	Ret.Time (detected)	Area	Rel.Area(ident.)	Height	Amount
		min	mAU*min	%	mAU	
	<mark>51 1</mark>	35.35	73.55517	93.90978632	78.95802	n.a.
	76 2	47.63	4.770	6.090213681	4.619	n.a.

RM-AMS-22R



No.		Peak Name	Ret.Time (dete	cted)	Area	Rel	.Area(ident.)	Height	Amount
			min		mAU*min	%		mAU	
	194	1		55.60	90.79799		52.21674219	66.02942	n.a.
	269	2		74.96	83.089		47.78325781	43.930	n.a.
		RM-AMS-22	2C						



No.		Peak Name	Ret.Time (detected)	Area	Rel	.Area(ident.)	Height	Amount
			min	mAU*min	%		mAU	
	114	1	55.85	58.33509		97.56841222	39.87429	n.a.
	187	2	75.63	1.454		2.431587785	0.896	n.a.

RM-AMS-12R



No.		Peak Name	Ret.Time (detected)	Area	Re	I.Area(ident.)	Height	Amount
			min	mAU*min	%		mAU	
	106	1	27.73	111.7665		50.28176471	133.002	n.a.
	112	2	32.23	110.514		49.71823529	110.553	n.a.
		RM-AMS-12	C					



No.	Peak Name	Ret.Time (detected)	Area	Rel.Area(ident.)	Height	Amount
		min	mAU*min	%	mAU	
	54 1	26.13	179.1302	91.86658626	208.8371	n.a.
	64 2	31.27	15.858	8.132943741	17.765	n.a.





No.		Peak Name	Ret.Time (detected)	Area	Rel	.Area(ident.)	Height	Amount
			min	mAU*min	%		mAU	
	105	1	27.53	145.4057		50.67646502	204.1363	n.a.
	108	2	31.43	141.524		49.32353498	170.654	n.a.

RM-AMS-3C



No.	Peak Name	Ret.Time (detected)	Area	Rel.Area(ident.)	Height	Amount
		min	mAU*min	%	mAU	
	36 A	28.51	81.53621	91.21334626	100.0774	n.a.
	37 B	32.91	7.854	8.786653735	10.165	n.a.





No.	Peak Name	Ret.Time (detected)	Area	Rel.Area(ident.)	Height	Amount
		min	mAU*min	%	mAU	
	33 1	30.67	48.98738	50.78795208	63.00821	n.a.
	34 2	34.38	47.467	49.21204792	53.512	n.a.

RM-AMS-2C



No.		Peak Name	Ret.Time (detected)	Area	Rel.Area(ident.)	Height	Amount
			min	mAU*min	%	mAU	
	149	1	30.4	3 76.99119	94.03105934	95.44135	n.a.
	150	2	34.88	3 4.887	5.968940128	6.422	n.a.

RM-AMS-4R



No.		Peak Name	Ret.Time (detected)	Area	Re	l.Area(ident.)	Height	Amount
			min	mAU*min	%		mAU	
	31	1	23.70	51.06273		92.63291195	85.94157	n.a.
	32	2	27.79	4.061		7.367088045	6.002	n.a.





No.		Peak Name	Ret.Time (detected)	Area	Rel	I.Area(ident.)	Height	Amount
			min	mAU*min	%		mAU	
	163	1	26.4	3 111.8901		49.89969195	149.1592	n.a.
	164	2	28.8	7 112.340		50.10030805	119.799	n.a.

RM-AMS-13R



No.	Peak Name	Ret.Time (detected)	Area	Rel.Area(ident.)	Height	Amount
		min	mAU*min	%	mAU	
	93 1	25.82	140.2529	92.92505871	201.7818	n.a.
	94 2	28.61	<mark>10.67</mark> 8	7.074941294	15.271	n.a.





No.	Peak Name	Ret.Time (detected)	Area	Rel.Area(ident.)	Height	Amount
		min	mAU*min	%	mAU	
	30 1	18.41	79.9158	50.41014123	158.1795	n.a.
	34 2	28.80	78.615	49.58985877	51.054	n.a.

RM-AMS-9C



No.	Peak Name	Ret.Time (detected)	Area	Rel.Area(ident.)	Height	Amount
		min	mAU*min	%	mAU	
	26 1	19.05	64.99365	97.83643263	117.1505	n.a.
	31 2	32.27	1.437	2.163491051	1.193	n.a.





No.	Peak Name	Ret.Time (detected)	Area	Rel.Area(ident.)	Height	Amount
		min	mAU*min	%	mAU	
	8 1	32.37	127.9147	49.36263466	116.5426	n.a.
1	3 2	40.05	131.218	50.63736534	118.188	n.a.

RM-AMS-14C



No.	Peak Name	Ret.Time (detected)	Area	Rel.Area(ident.)	Height	Amount
		min	mAU*min	%	mAU	
	25 1	33.79	5.012605	5.199683194	5.72466	n.a.
	31 2	41.06	91.390	94.80031681	83.015	n.a.





No.	1	Peak Name	Ret.Time (detected)	Area	Re	I.Area(ident.)	Height	Amount
			min	mAU*min	%		mAU	
1	151	1	26.5	1 170.0181		50.88109035	261.7212	n.a.
1	165	2	35.2	7 164.129		49.11867925	184.195	n.a.

RM-AMS-8C



No.	Peak Name	Ret.Time (detected)	Area	Rel.Area(ident.)	Height	Amount
		min	mAU*min	%	mAU	
	31 1	26.44	35.91638	92.31371183	54.37508	n.a.
	32 2	35.43	2.990	7.686288168	3.952	n.a.

RM-AMS-19R



Peak Name	Ret. Time (detected)	Area	Rel.Area(ident.)	Height Amount	
	min	mAU*min	%	mAU	
27 1	17.93	6.917124	50.68787914	15.22748 n.a.	
36 2	27.82	<u>6.729</u>	49.31212086	6.649 n.a.	

RM-AMS-19C



No.	Peak Name	Ret.Time (detected)	Area	Rel.Area(ident.)	Height	Amount
		min	mAU*min	%	mAU	
3	5 1	17.84	12.34744	97.23821715	26.88638	n.a.
4	8 2	28.60	0.351	2.76178285	0.548	n.a.

RM-AMS-28R



No.	Peak Name	Ret.Time (detected)	Area	Rel.Area(ident.)	Height	Amount
		min	mAU*min	%	mAU	
	36 1	13.75	88.87419	49.92720803	221.711	n.a.
	37 2	15.67	89.133	50.07279197	202.241	n.a.

RM-AMS-28C-2



No.	Peak Name	Ret.Time (detected)	Area	Rel.Area(ident.)	Height Am	ount
		min	mAU*min	%	mAU	
	23 1	13.69	2.395948	97.34811892	5.93645 n.a	
	26 2	15.64	0.065	2.651881078	0. <mark>1</mark> 85 n.a	í.

RM-AMS-41R



No.		Peak Name	Ret.Time (detec	cted)	Area	Rel	.Area(ident.)	Height	Amount
			min		mAU*min	%		mAU	
	41	1		26.64	48.96193		49.92510831	52.71095	n.a.
	43	2		33.86	49.109		50.07489169	47.014	n.a.

RM-AMS-41C



No.		Peak Name	Ret.Time (detecte	ed)	Area	Rel	.Area(ident.)	Height	Amount
			min		mAU*min	%		mAU	
	128	1	26	3.44	266.0192		95.21554153	282.3179	n.a.
	143	2	34	4.30	13.367		4.784458471	15.266	n.a.

RM-AMS-34R



No.	Peak Name	Ret.Time (detected)	Area	Rel.Area(ident.)	Height	Amount
		min	mAU*min	%	mAU	
	32 1	32.45	6.1046	8.127743906	7.39003	n.a.
	33 2	34.64	69.004	91.87225609	72.457	n.a.





No.	Peak Name	Ret.Time (detected)	Area	Rel.Area(ident.)	Height	Amount
		min	mAU*min	%	mAU	
54	1	40.73	244.0032	49.57047356	158.4591	n.a.
55	2	46.16	248.232	50.42952644	139.151	n.a.

RM-AMS-35C



No.	Peak Name	Ret.Time (detected)	Area	Rel.Area(ident.)	Height	Amount
		min	mAU*min	%	mAU	
	217 1	41.39	120.0172	96.26303483	83.97359	n.a.
	218 2	46.02	4.659	3.736965172	3.630	n.a.



No.		Peak Name	Ret.Time (detected)	Area	Rel.Area(ident.)	Height	Amount
			min	mAU*min	%	mAU	
	63	1	50.78	184.5528	49.36136141	37.73442	n.a.
	96	2	75.00	189.328	50.63863859	27.345	n.a.

RM-AMS-37C



No.	Peak Name	Ret.Time (detected)	Area	Rel.Area(ident.)	Height	Amount
		min	mAU*min	%	mAU	
	36 1	52.67	5.548119	4.787834233	2.0196	n.a.
	49 2	76.60	110.331	95.21216577	15.116	n.a.



No.	Peak Name	Ret.Time (detected)	Area	Rel.Area(ident.)	Height	Amount
		min	mAU*min	%	mAU	
138	1	101.98	198.382	50.10066496	50.82367	n.a.
146	2	122.62	197.585	49.89933504	44.106	n.a.

RM-AMS-36C



No.		Peak Name	Ret.Time (detected)	Area	Re	I.Area(ident.)	Height	Amount
			min	mAU*min	%		mAU	
	253	1	106.66	6.163661		2.073575543	2.14347	n.a.
	299	2	126.01	291.084		97.92642446	87.199	n.a.





No.	F	Peak Name	Ret.Time (deter	cted)	Area	Rel.	Area(ident.)	Height	Amount
			min		mAU*min	%		mAU	
	48 ´	1		25.84	107.4142		49.2991849	179.1218	n.a.
	49 2	2		33.00	110.467		50.70044991	129.422	n.a.

RM-AMS-32C



No.	Peak Name	Ret.Time (detected)	Area	Rel.Area(ident.)	Height	Amount
		min	mAU*min	%	mAU	
	36 1	26.02	114.4702	79.44106964	178.541	n.a.
	47 2	33.50	29.624	20.55892309	36.959	n.a.





ams-51c



AMS-42R



AMS-42C



RM-AMS-49R-PHCO2PH



No.		Peak Name	Ret.Time (dete	cted)	Area	Rel	.Area(ident.)	Height	Amount
			min		mAU*min	%		mAU	
	142	1		26.17	66.24177		49.92456002	64.72089	n.a.
	143	2		29.71	66.442		50.07543998	60.863	n.a.

RM-AMS-49C-PHCO2PH



No.	Peak Name	Ret.Time (detected)	Area	Rel.Area(ident.)	Height	Amount
		min	mAU*min	%	mAU	
	38 1	25.93	19.58077	92.72817613	19.30432	n.a.
	39 2	29.43	1.536	7.271823868	1.565	n.a.





No.	Peak Name	Ret.Time (detected)	Area	Rel.Area(ident.)	Height	Amount
		min	mAU*min	%	mAU	
	29 Q	26.90	275.4331	50.22193381	385.8548	n.a.
	30 R	31.48	272.999	49.77806619	268.800	n.a.

RM-AMS-44C-BOC



No.	Peak Name	Ret.Time (detected)	Area	Rel.Area(ident.)	Height	Amount
		min	mAU*min	%	mAU	
	116 1	25.82	53.10009	96.37384594	66.2957	n.a.
	117 2	31.05	1.998	3.626154062	2.426	n.a.




No.		Peak Name	Ret.Time (detec	ted)	Area	Rel	.Area(ident.)	Height	Amount
			min		mAU*min	%		mAU	
	46	1		38.21	64.57679		50.16603231	47.83468	n.a.
	48	2	14	47.75	64.149		49.83396769	37.116	n.a.

RM-AMS-45C-IM



No.	Peak Name	Ret.Time (detected)	Area	Rel.Area(ident.)	Height	Amount
		min	mAU*min	%	mAU	
	76 1	38.38	6.684822	3.553845847	5.40461	n.a.
	77 2	47.06	181.416	96.44615415	108.107	n.a.





No.	Peak Name	Ret.Time (detected)	Area	Rel.Area(ident.)	Height	Amount
		min	mAU*min	%	mAU	
1	1	11.76	138.5414	96.40144282	355.2209	n.a.
2	2	22.38	5.172	3.598557176	10.672	n.a.