

# Sustainable Recycling of Benzoic Acid Production Waste: Green and Highly Efficient Methods to Separate and Recover High Value-Added Conjugated Aromatic Compounds from Industrial Residues

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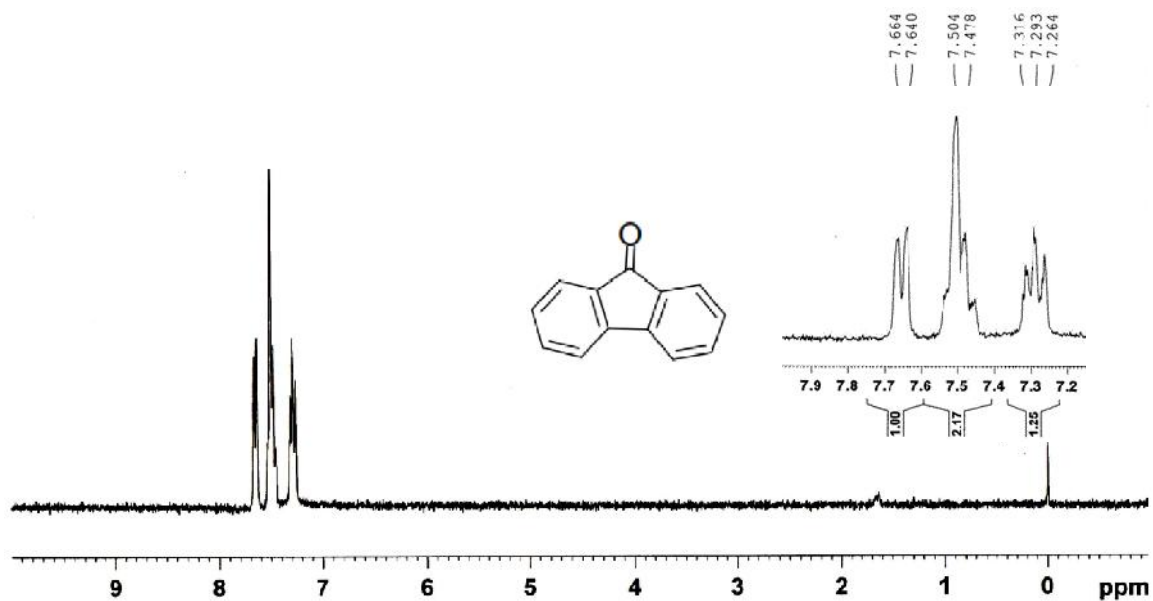
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<sup>‡</sup>Department of Chemical Engineering, Texas Tech University, 6th and Canton, Lubbock, TX 79409, USA

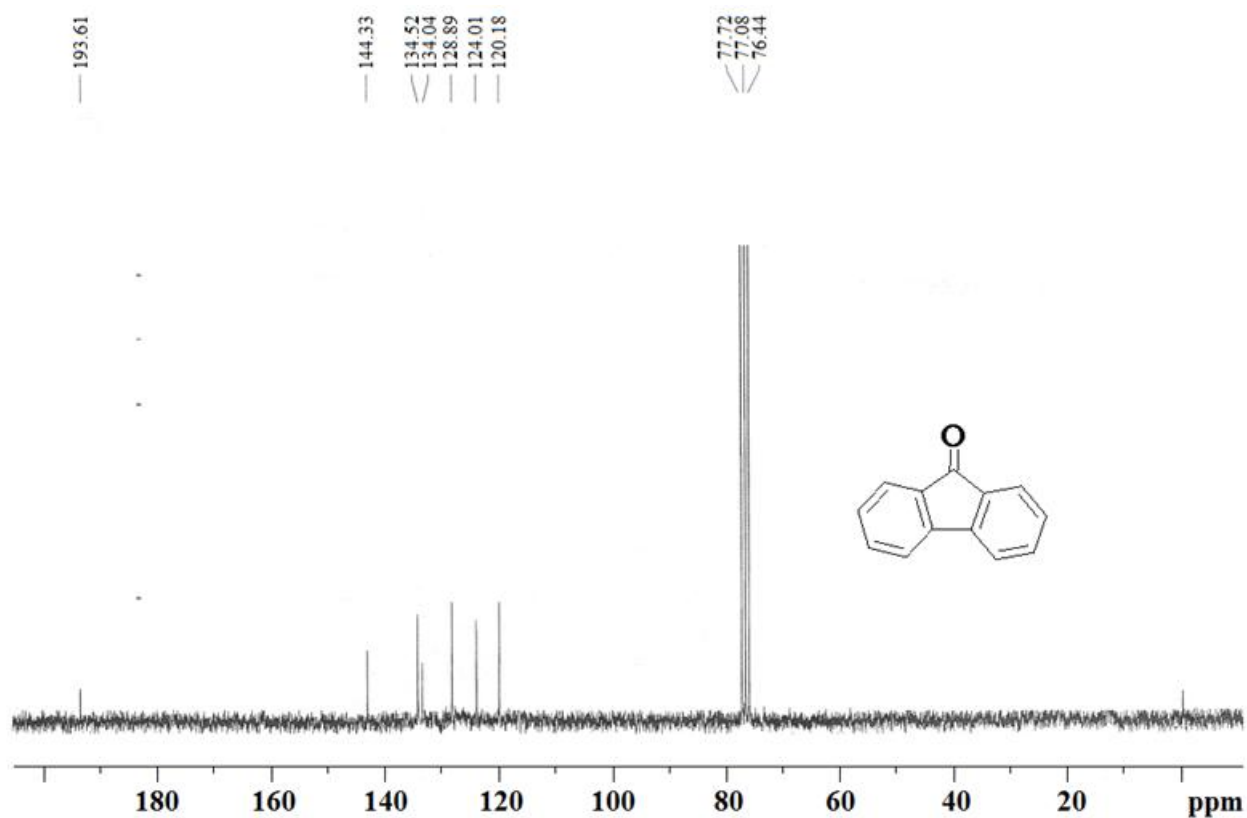
<sup>§</sup>Sphinx Scientific Laboratory Corporation, 1250 1/2 E. State Street, Sycamore, IL 60178, USA

## Supporting Information

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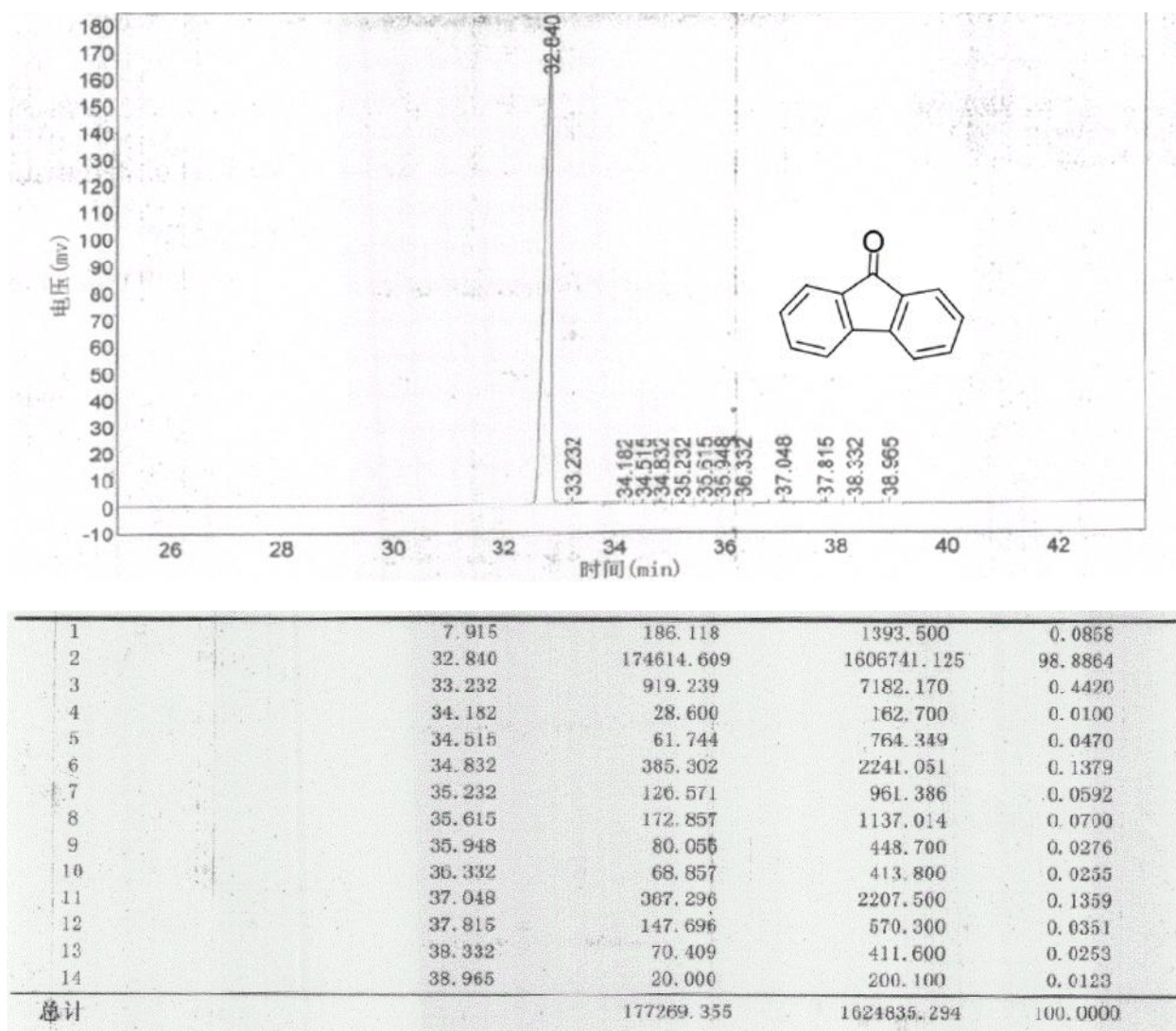


b.



**Figure S1.** <sup>1</sup>H and <sup>13</sup>C NMR spectra of 9-fluorenone (in CDCl<sub>3</sub>)

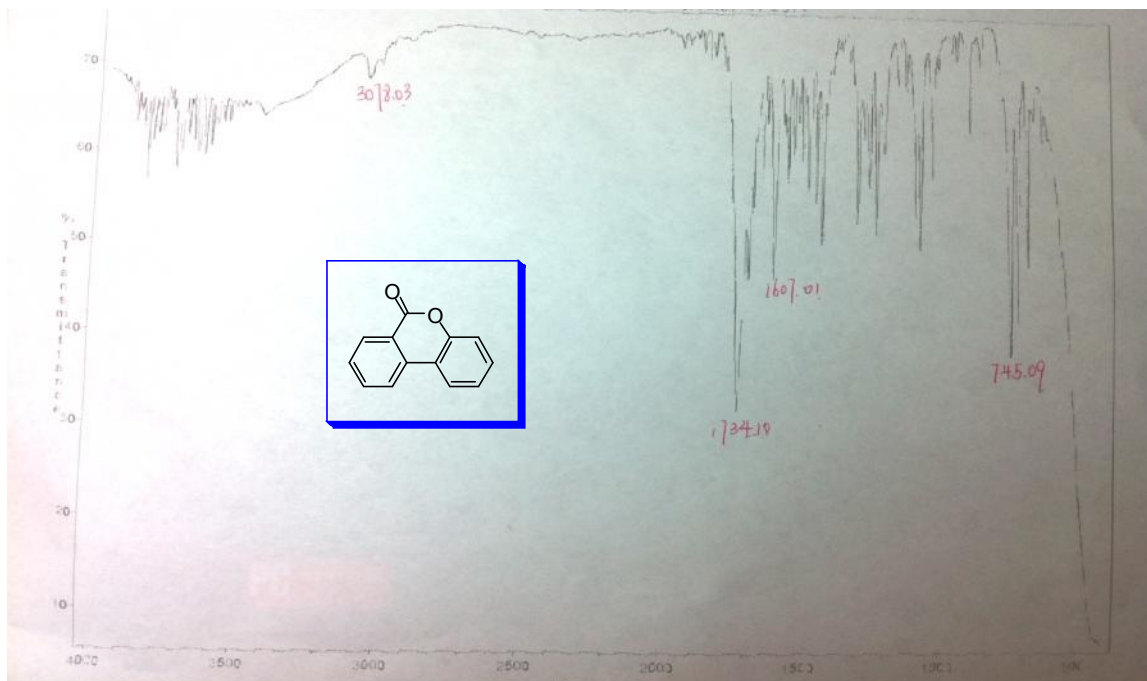
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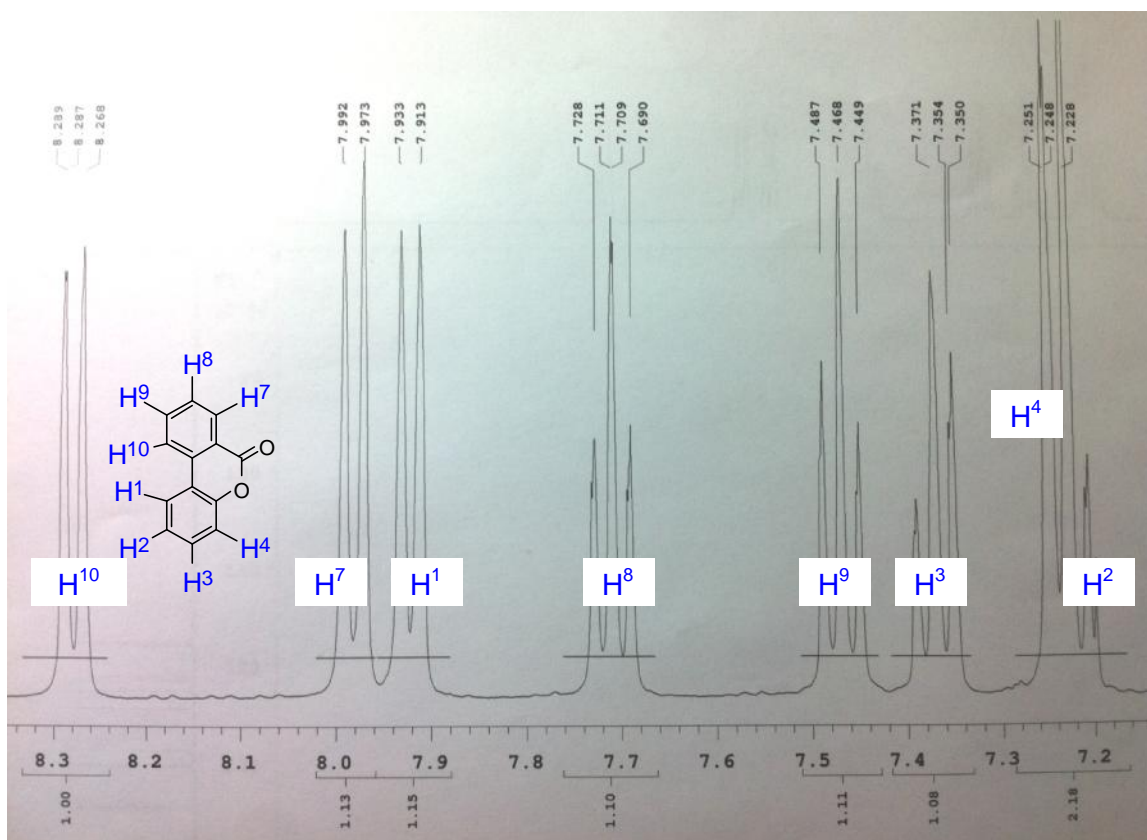
**Figure S2.** HPLC trace of 9-fluorenone

# Supporting Information

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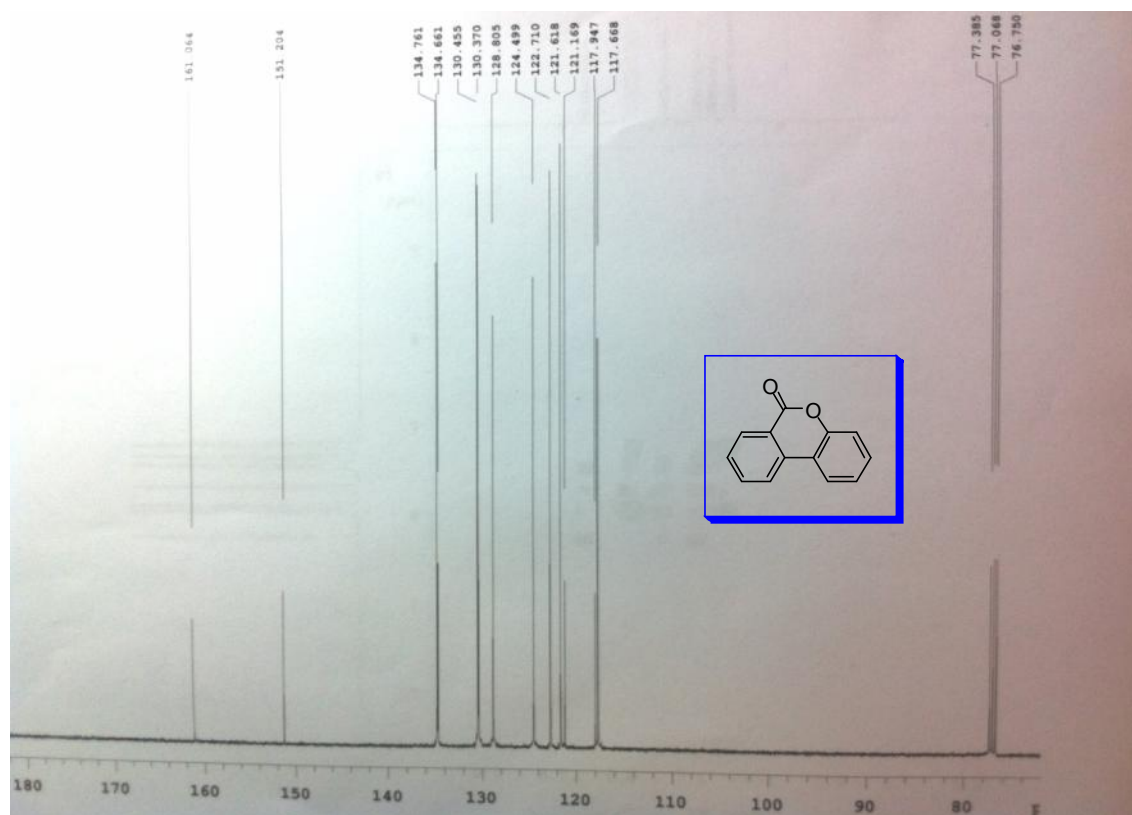


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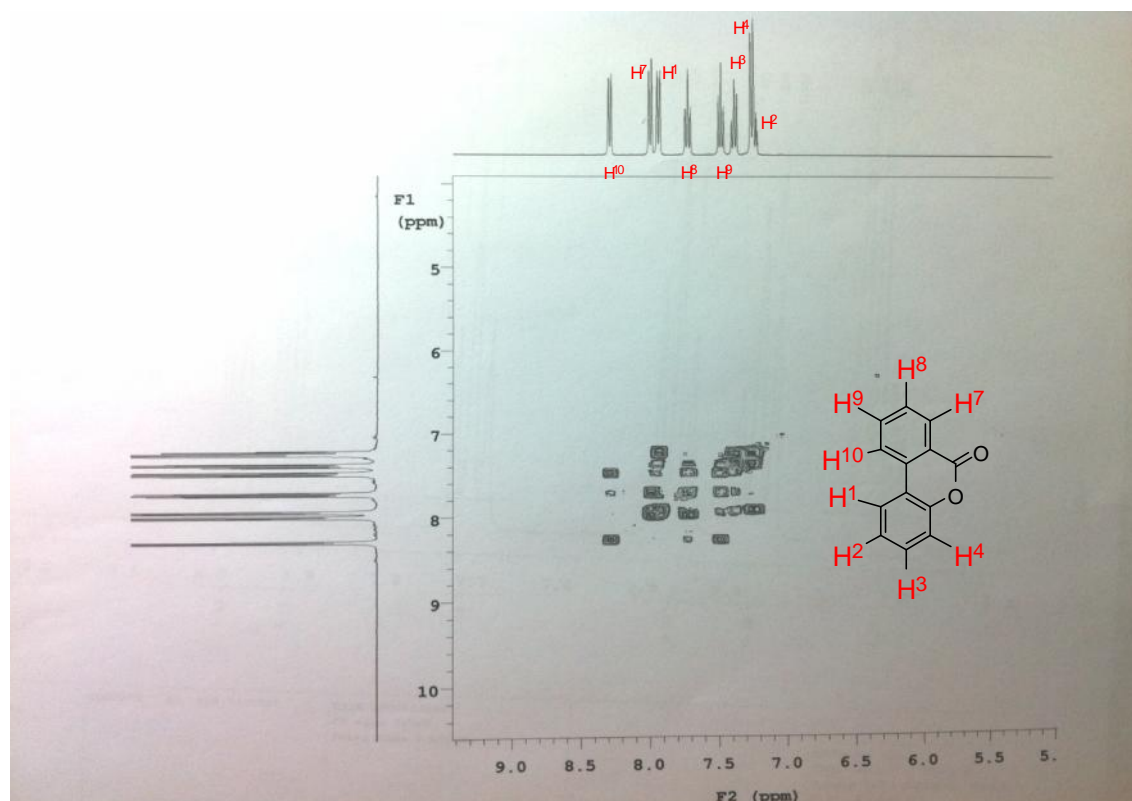


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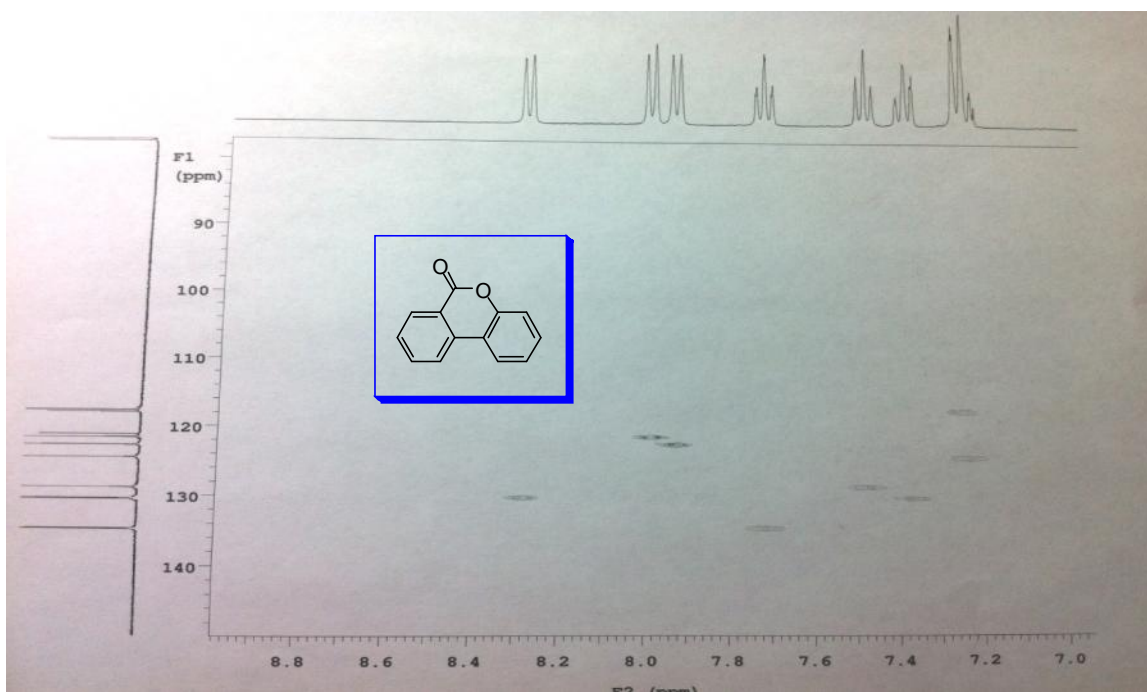
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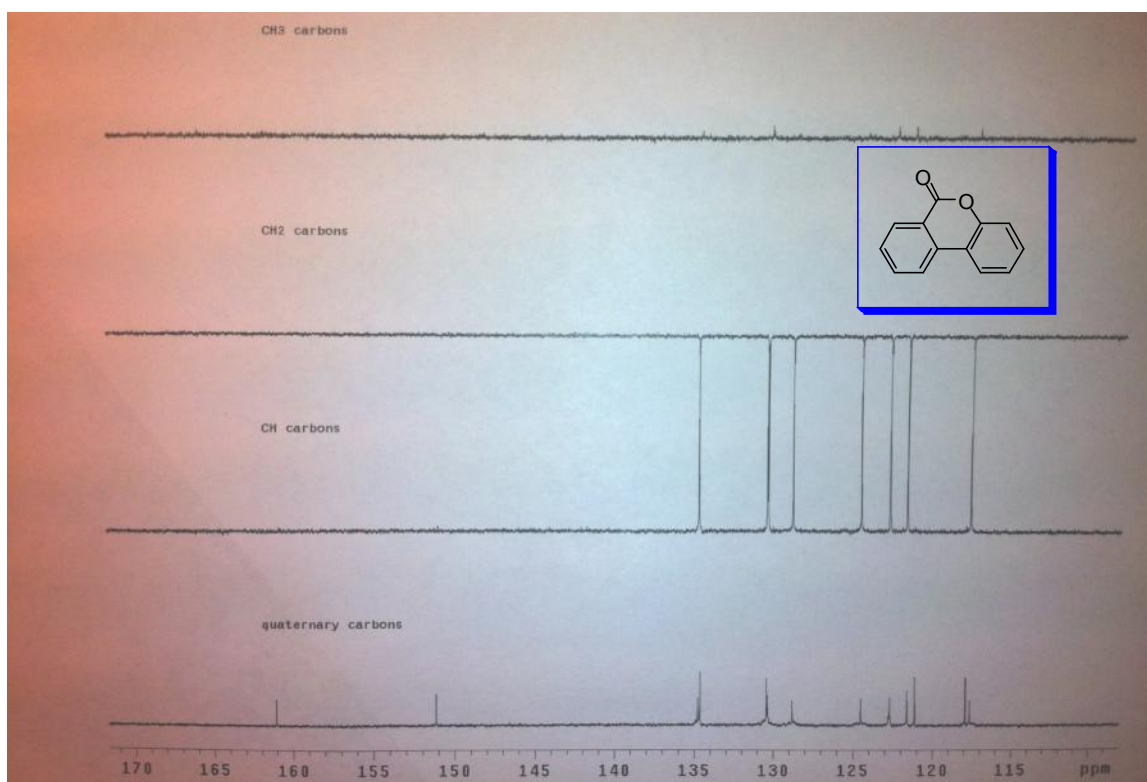


## Supporting Information

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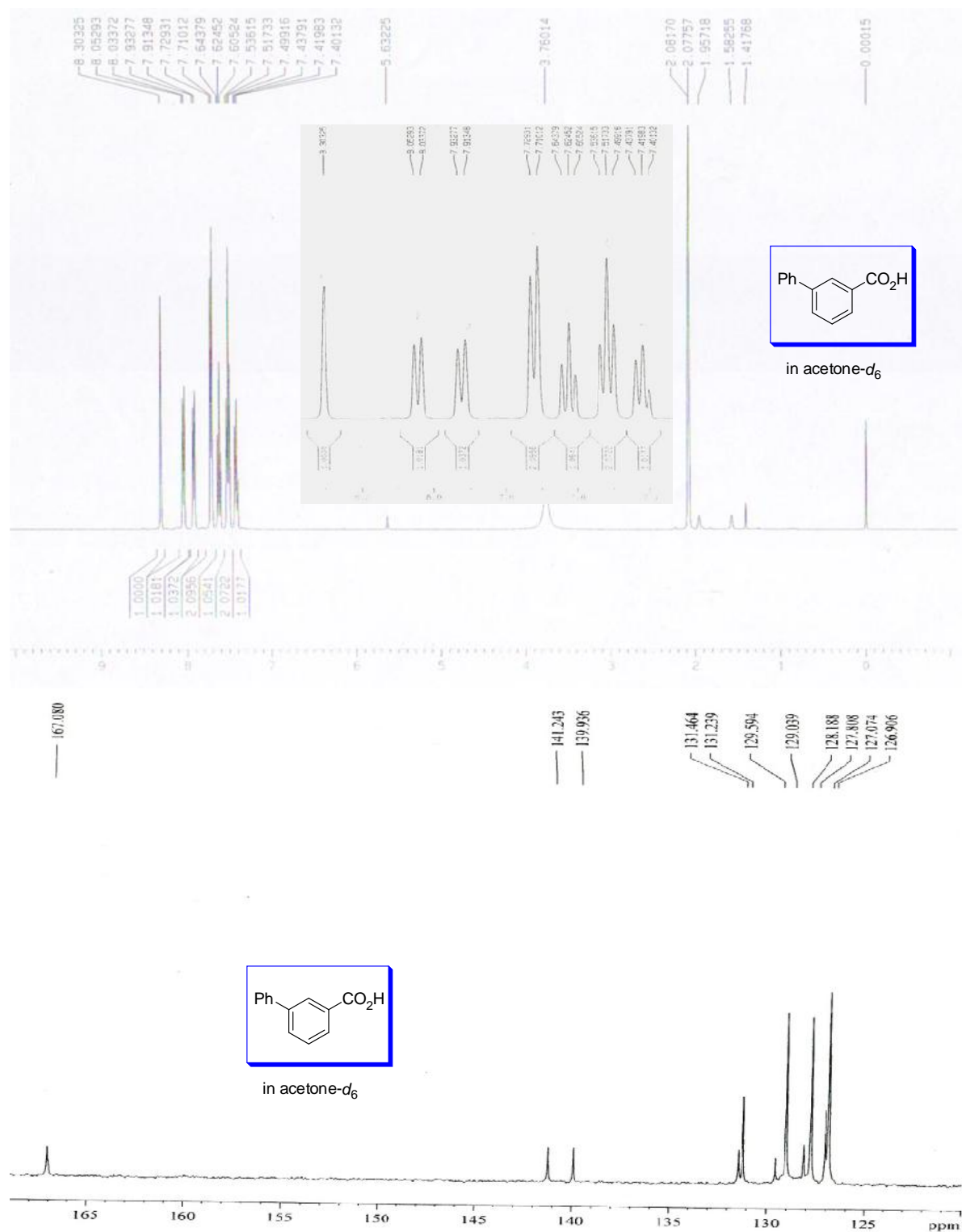


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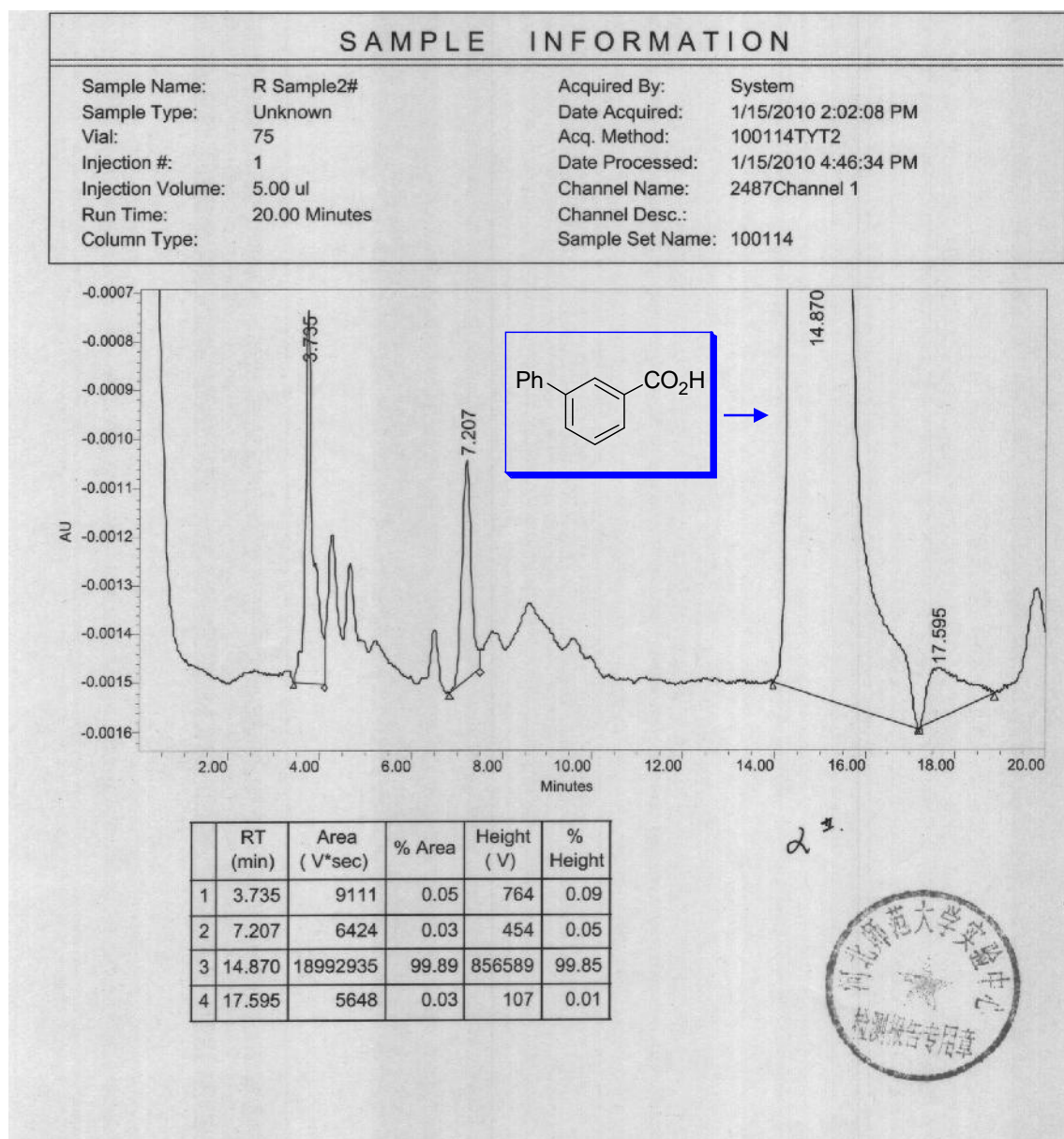
**Figure S3.** FTIR, <sup>1</sup>H and <sup>13</sup>C NMR, COSY, and DEPT spectra of 6H-benzo[c]chromen-6-one

## Supporting Information



**Figure S4.** <sup>1</sup>H and <sup>13</sup>C NMR spectra of biphenyl-3-carboxylic acid

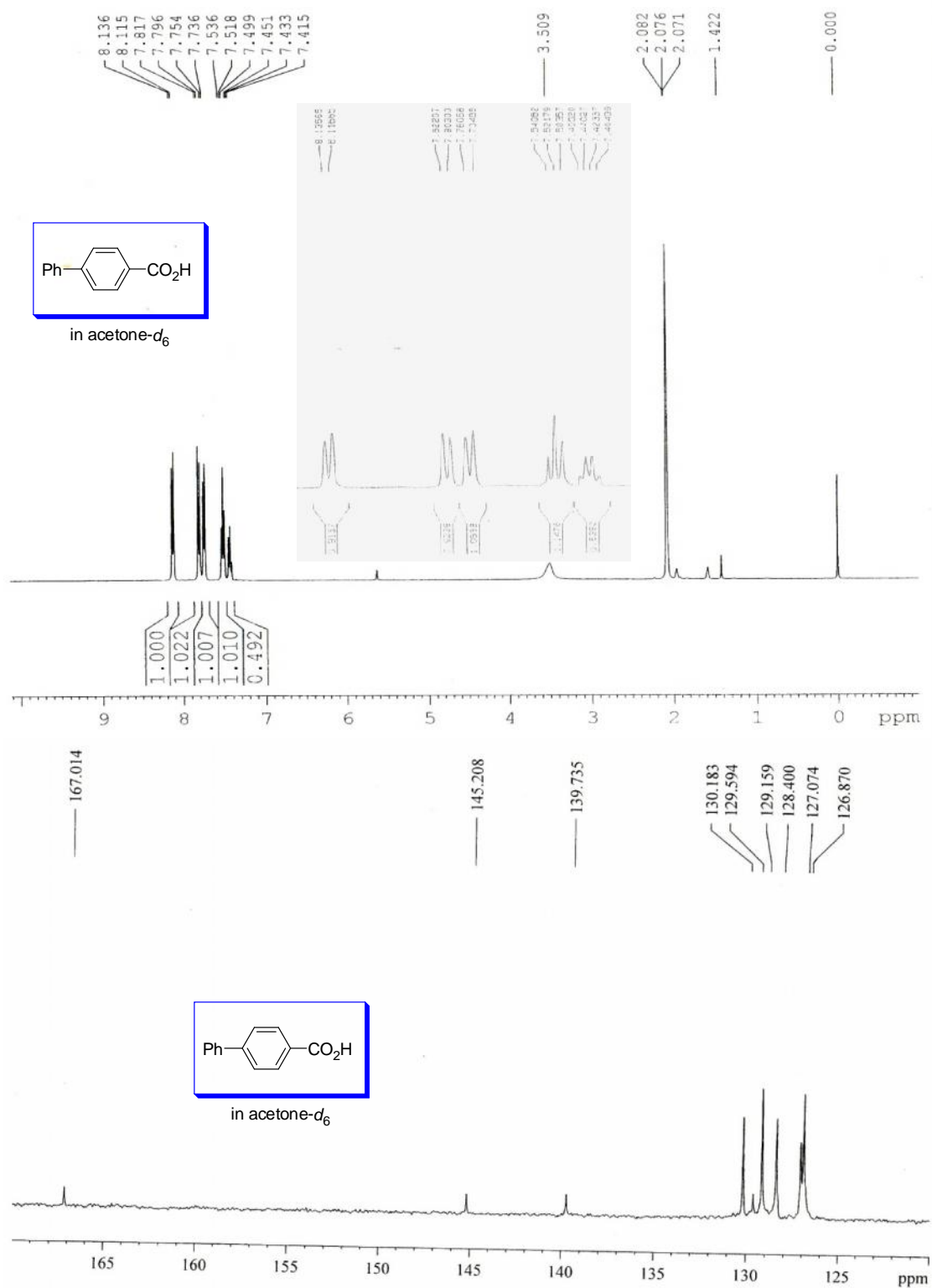
## Supporting Information



**Figure S5.** HPLC trace of biphenyl-3-carboxylic acid

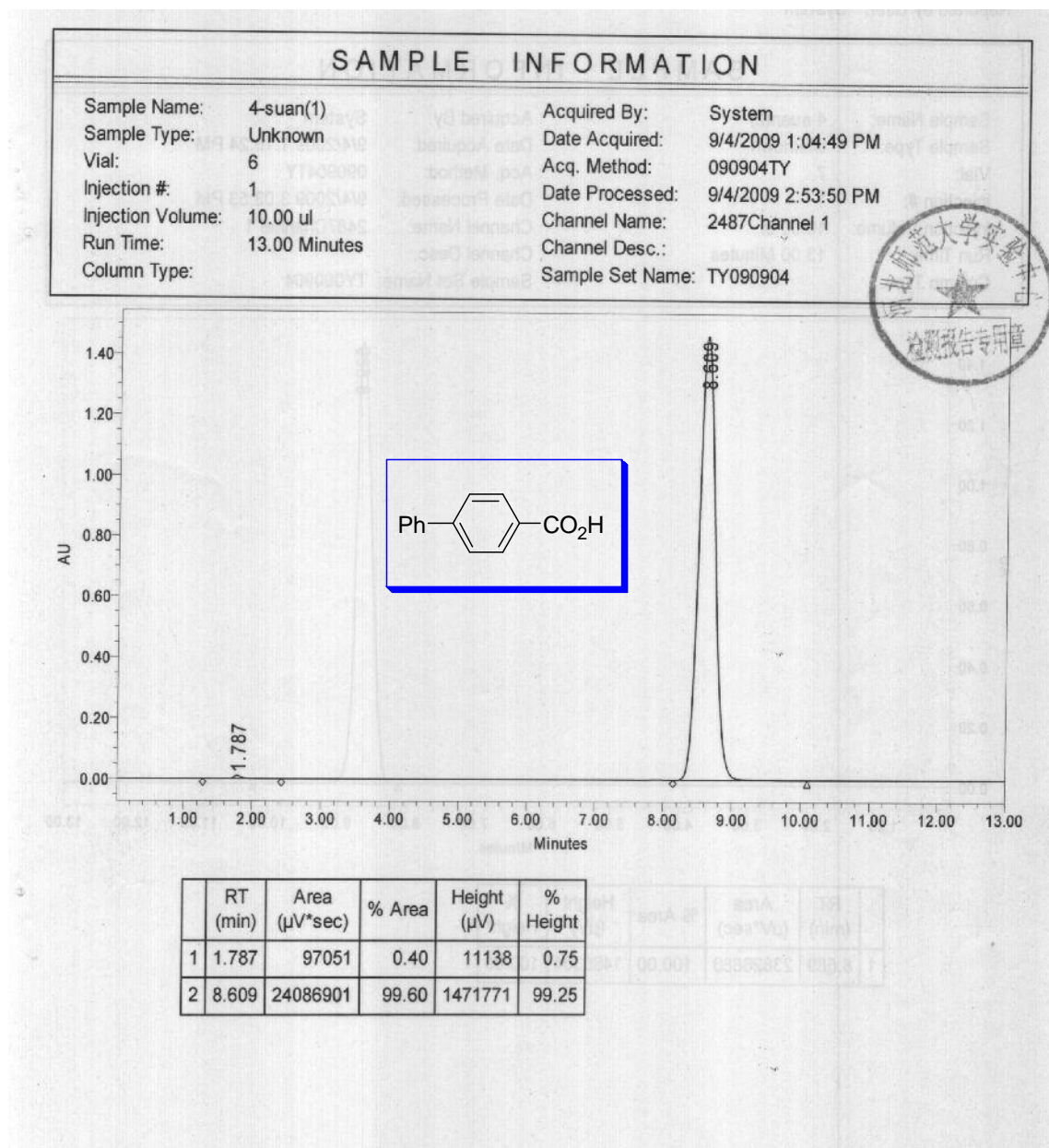


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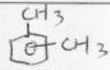

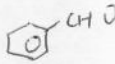
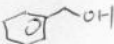
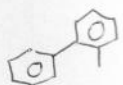
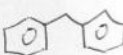
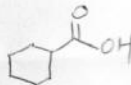
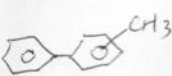
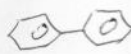
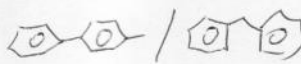
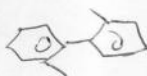
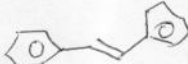
**Figure S6.** <sup>1</sup>H and <sup>13</sup>C NMR spectra of biphenyl-4-carboxylic acid

## Supporting Information

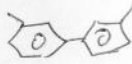
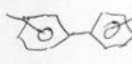



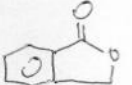
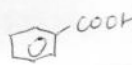
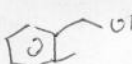
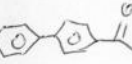
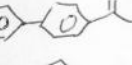
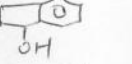
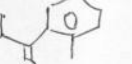



**Figure S7.** HPLC trace of biphenyl-4-carboxylic acid

# Supporting Information

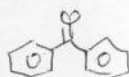
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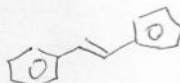
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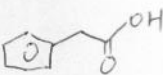
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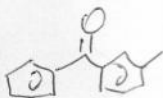


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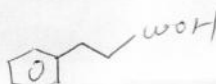


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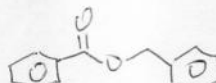
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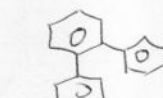


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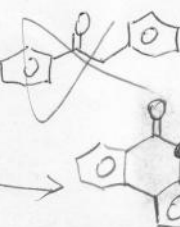


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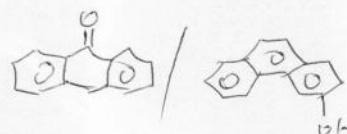
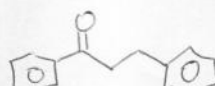
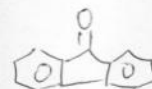
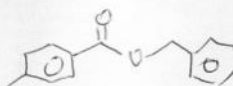
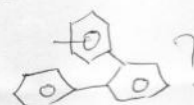
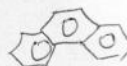
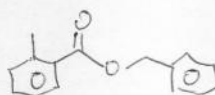


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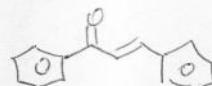
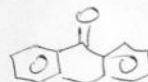
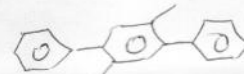
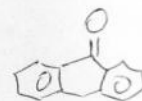
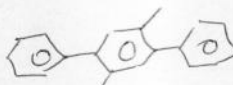
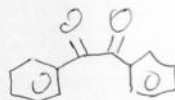
# Supporting Information

37	28.74	0.15 D:\Database\NIST02.L	
		benzyl o-toluate	73819 067157-60-2 49
		1-Bromo-1-phenylpropane X	55327 002114-36-5 43
		N-benzyl-1H-benzimidazole X	61570 004981-92-4 43
38	28.87	0.88 D:\Database\NIST02.L	
		Naphthalene, 2-(1-methyl-2-phen...	84947 017181-02-1 95
		Coumarine, 8-allyl-7-hydroxy-6-	84801 298686-57-4 53
		2H-1-Benzopyran-2-one, 7-methox...	84806 000581-31-7 50
39	29.20	0.23 D:\Database\NIST02.L	
		Benzeneacetic acid, phenylmethy...	73855 000102-16-9 58
		4-Benzoyloxybenzonitrile	62301 052805-36-4 52
		Benzene, (iodomethyl)-	68359 000620-05-3 50
40	29.62	0.06 D:\Database\NIST02.L	
		Benz[3,4]anthra[1,2-b]oxirene,...	93474 001155-38-0 55
		2',5'-Dimethyl-p-terphenyl	93489 020260-22-4 53
		1,2,3,10,11,12-Hexahydroperylene	93491 007350-92-7 50
41	30.54	1.15 D:\Database\NIST02.L	
		Naphthalene, 2-91-methyl-2-phen...	84947 017181-02-1 86
		Benzoic acid, 4-methyl-, phenyl...	73863 005467-99-2 40
		1,1',4,1"-Terphenyl-, 3'-methyl-	84945 033776-38-4 30
42	30.89	0.13 D:\Database\NIST02.L	
		Phenanthrene	41356 000085-01-8 64
		2-Cyclopropen-1-one, 2,3-diphenyl-	60289 000886-38-4 64
		Anthracene	41350 000120-12-7 64
43	31.00	0.11 D:\Database\NIST02.L	
		1,4-Cyclohexadiene, 6-methylene...	84948 018636-59-4 87
		1,1',4,1"-terphenyl-, 3'-methyl-	84945 033776-38-4 64
		1,1'-Biphenyl, 2-(phenylmethyl)-	84943 000606-97-3 58
44	31.49	0.52 D:\Database\NIST02.L	
		Benzyl p-toluate	73820 001711-35-9 91
		Benzoic acid, 4-methyl-, phenyl...	73863 005467-99-2 70
		Benzamide, 2-methyl-N-phenyl-	63642 007055-03-0 59
45	31.78	3.52 D:\Database\NIST02.L	
		9H-Fluoren-9-one ✓	42768 000486-25-9 93
		9H-Fluoren-9-one ✓	42769 000486-25-9 93
		9H-Fluoren-9-one	42766 000486-25-9 91
46	32.13	1.06 D:\Database\NIST02.L	
		.beta.-Phenylpropiophenone	63006 001083-30-3 94
		.beta.-Phenylpropiophenone	63010 001083-30-3 86
		1-Pentanone, 4,4-dimethyl-1,3-d...	98159 057847-40-2 64
47	32.83	0.06 D:\Database\NIST02.L	
		4-Benzylbiphenyl	84938 000613-42-3 93
		1,1',4,1"-terphenyl-, 3'-methyl-	84945 033776-38-4 84
		1,1',3,1"-terphenyl-, 5'-methyl-	84944 033733-07-2 81
48	33.15	0.22 D:\Database\NIST02.L	
		Anthrone	52034 000090-44-8 95
		Anthrone	52035 000090-44-8 95
		3-Phenanthrol	52038 000605-87-8 94



# Supporting Information

49	33.57	0.65 D:\Database\NIST02.L Benzenecetic acid, alpha, -hyd... Benzenecetic acid, 4-ethoxy- 1-Butanol, 4-(phenylmethoxy)-	42323 000774-40-3 53 42222 004919-33-9 47 42462 004541-14-4 47
50	33.67	0.52 D:\Database\NIST02.L Ethanedione, diphenyl- beta-Phenylpropiophenone Benzoic acid, (4-benzoyloxy-2-c...	62903 000134-81-6 78 63006 001083-30-3 78 141897 122842-83-5 50
51	34.42	0.38 D:\Database\NIST02.L 1,1',4,1"-terphenyl-, 3'-methyl- 1,1'-Biphenyl, 2-(phenylmethyl)- 1,1',3,1"-terphenyl-, 5'-methyl-	84945 033776-38-4 98 84943 000606-97-3 94 84944 033733-07-2 93
52	34.71	0.25 D:\Database\NIST02.L 4,5-Dimethoxy-2-biphenylcarboxy... 2',5'-Dimethyl-p-terphenyl 5,7-Dimethyl-6-phenyl-1,3-diaza...	93227 162137-38-4 45 93489 020260-22-4 43 93371 1000216-37-9 38
53	35.70	0.12 D:\Database\NIST02.L Ethanone, 2,2-dichloro-1-(4-met... Benzoic acid, 4-methyl-, phenyl... 3,4,6-Trimethylthiopyrazolo[3,4...	57712 004974-59-8 30 64427 001900-85-2 25 93563 130224-57-6 25
54	36.57	0.28 D:\Database\NIST02.L Anthrone Anthrone Anthrone	52034 000090-44-8 93 52036 000090-44-8 91 52035 000090-44-8 87
55	36.67	0.12 D:\Database\NIST02.L 2',5'-Dimethyl-p-terphenyl 4,5,7,8,9,10-Hexahydrobenzo(a)p... E-1-(9'-Fluorenylidene)-2-cyano...	93489 020260-22-4 46 93494 073712-75-1 46 93450 083026-87-3 43
56	38.21	0.15 D:\Database\NIST02.L Anthrone Anthrone Anthrone	52036 000090-44-8 94 52034 000090-44-8 94 52035 000090-44-8 91
57	40.05	0.25 D:\Database\NIST02.L 4-Benzylbiphenyl 1,1',4,1"-terphenyl-, 3'-methyl- 1,1'-Biphenyl, 2-(phenylmethyl)-	84939 000613-42-3 96 84945 033776-38-4 93 84943 000606-97-3 89
58	44.15	0.88 D:\Database\NIST02.L 2-Propen-1-one, 1,3-diphenyl- 2-Propen-1-one, 1,3-diphenyl- 2-Propen-1-one, 1,3-diphenyl-	61681 000094-41-7 94 61689 000614-47-1 90 61683 000094-41-7 90
59	44.71	0.69 D:\Database\NIST02.L 1,1',4,1"-terphenyl-, 3'-methyl- 4-Benzylbiphenyl 4-Benzylbiphenyl	84945 033776-38-4 91 84939 000613-42-3 90 84938 000613-42-3 90
60	45.00	0.26 D:\Database\NIST02.L 1,1',4,1"-terphenyl-, 3'-methyl- 1,4-Cyclonexdiene,6-methylene... 1,1',3,1"-Terphenyl, 5'-methyl-	84945 033776-38-4 93 84948 018636-59-4 90 84944 033733-07-2 76



# Supporting Information

61	48.06	0.39 D:\Database\NIST02.L m-Terphenyl m-Terphenyl m-Terphenyl	76475 000092-06-8 98 76474 000092-06-8 98 76471 000092-94-4 96	
62	48.75	0.64 D:\Database\NIST02.L 9,10-Anthracenedione 9H-Fluoren-9-one 1H-Phenalen-1-one	61655 000084-65-1 90 42767 000486-25-9 60 42770 000548-39-0 55	
63	52.48	0.98 D:\Database\NIST02.L 3H-Indazol-3-one, 1,2-dihydra-2... Benzamide, N-(2'-iodophenyl)-N-... Benzene, 1-methoxy-2-(2-phenyle...	62792 017049-65-9 53 135712 138883-34-8 47 63038 052805-92-2 47	
64	53.07	2.82 D:\Database\NIST02.L 4-Hydroxy-9-fluorenone 9H-Fluoren-9-one, 1-hydroxy- 4-Hydroxy-9-fluorenone	53564 001986-00-1 95 53565 006344-60-1 93 53563 001986-00-1 93	
65	54.21	0.37 D:\Database\NIST02.L p-Terphenyl p-Terphenyl m-Terphenyl	76473 000092-94-4 97 76471 000092-94-4 95 76467 000092-06-8 93	
66	54.70	1.12 D:\Database\NIST02.L 4-Benzylbiphenyl 4-Benzylbiphenyl Triphenylmethane	84939 000613-42-3 98 84938 000613-42-3 95 84940 000519-73-3 81	
67	72.37	1.28 D:\Database\NIST02.L 2-Biphenylcarboxylic acid 2-Biphenylcarboxylic acid Biphenyl-4-carboxylic acid	54844 000947-84-2 91 54843 000947-84-2 90 54846 000092-92-2 62	
68	76.01	1.25 D:\Database\NIST02.L 3-(1-naphthyl)acrylaldehyde 2,4,6-cycloheptatrien-1-one, 2-... Mopholine, 4-(1-cyclopenten-1-...	44116 001504-73-0 52 44124 014562-09-5 47 43350 007182-08-3 46	
69	97.57	0.56 D:\Database\NIST02.L (1,1'-Biphenyl)-2-acetic acid 9H-Fluoren-9-one, hydrazone Anthrone	64391 014676-52-9 74 51937 013629-22-5 62 52036 000090-44-8 62	
70	108.26	1.05 D:\Database\NIST02.L 4-Amino-4'-hydroxystilbene (1,1'-Biphenyl)-2-ol, 5-(1,1-di... 10H-Phenoxasim, 10,10-dimethyl-	63634 000836-44-2 43 73950 000577-92-4 38 73733 018414-62-5 35	
71	111.45	17.42 D:\Database\NIST02.L (1,1'-Biphenyl)-3-carboxylic acid ✓ 2-Biphenylcarboxylic acid Biphenyl-4-carboxylic acid	54854 000716-76-7 97 54843 000947-84-2 87 54846 000092-92-2 81	
72	117.52	20.38 D:\Database\NIST02.L Biphenyl-4-carboxylic acid ✓ Biphenyl-4-carboxylic acid Biphenyl-4-carboxylic acid	54846 000092-92-2 98 54850 000092-92-2 97 54851 000092-92-2 93	

**Figure S8.** LC-MS analytical report of yellow solid mixture

## Supporting Information

**Table S1.** Waste water analytical reports for separating pure 9-fluorenone from the benzyl benzoate residue (originals given in Chinese are followed by English translation)

苯甲酸生产废料回收利用项目生产废水抽样检测结果(2) 省环评项目

序号	测试项目	水样测试结果 <sup>a,b</sup>		
		水样 1 (2010.03.23)	水样 2 (2010.03.24)	水样 3 (2010.03.25)
1	pH 值	6.0	6.5	6.5
2	色度	8	12	18
3	悬浮物	16	18	21
4	五日生化需氧量	5	8	6
5	化学需氧量	25	22	30
6	氨氮 (以 N 计)	2	3	2
7	总氮	3	4	3
8	总磷	0.1	0.1	0.1
9	总有机碳	16	18	12
10	硫化物	未检出	未检出	未检出
11	硝基苯类	未检出	未检出	未检出
12	苯胺类	未检出	未检出	未检出
13	总氰化物	未检出	未检出	未检出
14	总汞	未检出	未检出	未检出
15	六价铬	未检出	未检出	未检出

a. 废水样品从车间废水排放口采集;

b. 测试项目单位为 mg/L (pH 值和色度除外)。

## Supporting Information

Entry	Test Item	Test Result <sup>a,b</sup>		
		Waste Sample 1 (2010.03.23)	Waste Sample 2 (2010.03.24)	Waste Sample 3 (2010.03.25)
1	pH Value	6.0	6.5	6.5
2	Chroma	8	12	18
3	Suspended Material	16	18	21
4	BOD5	5	8	6
5	Chemical Oxygen Demand	25	22	30
6	Ammonia Nitrogen	2	3	2
7	Total Nitrogen	3	4	3
8	Total Phosphorus	0.1	0.1	0.1
9	Total Organic Carbon	16	18	12
10	Sulfide	undetected	undetected	undetected
11	Nitrobenzenes	undetected	undetected	undetected
12	Phenyl amines	undetected	undetected	undetected
13	Total Cyanide	undetected	undetected	undetected
14	Total Mercury	undetected	undetected	undetected
15	Hexavalent Chrome	undetected	undetected	undetected

a. The wastewater samples were collected from from workshop wastewater outfalls;

b. The unit is mg/L (except pH and Chroma).



## Supporting Information

**Table S2.** Waste water analytical reports for separating 6H-benzo[c]chromen-6-one, biphenyl-3-carboxylic acid and biphenyl-4-carboxylic acid mixture from the yellow solid mixture (originals given in Chinese are followed by English translation)

苯甲酸生产废料回收利用项目生产废水抽样检测结果(1) 混合苯甲酸分离项目

序号	测试项目	水样测试结果 a,b		
		水样 1 (2009.09.02)	水样 2 (2009.09.03)	水样 3 (2009.09.04)
1	pH 值	6.5	6.0	6.0
2	色度	15	10	21
3	悬浮物	22	15	25
4	五日生化需氧量	8	6	5
5	化学需氧量	16	12	25
6	氨氮 (以 N 计)	1	1	0.6
7	总氮	2	1.6	1
8	总磷	0.1	0.1	0.1
9	总有机碳	18	12	20
10	硫化物	未检出	未检出	未检出
11	硝基苯类	未检出	未检出	未检出
12	苯胺类	未检出	未检出	未检出
13	总氰化物	未检出	未检出	未检出
14	总汞	未检出	未检出	未检出
15	六价铬	未检出	未检出	未检出

a. 废水样品从车间废水排放口采集;  
b. 测试项目单位为 mg/L (pH 值和色度除外)。

*Handwritten notes:*

- 左侧: 主要污染物是什么? (Main pollutants are what?)
- 左侧: 来自有机碳吗? (From organic carbon?)
- 左侧: 检测过吗? 不用管 (Detected? No need to worry)
- 右侧: 氨氮和总氮来自哪里? (Ammonia nitrogen and total nitrogen from where?)
- 右侧: 无氰化物、硝基苯类、苯胺类、总氰化物、总汞、六价铬 (No cyanide, nitrobenzene, aniline, total cyanide, total mercury, hexavalent chromium)

## Supporting Information

Entry	Test Item	Test Result <sup>a,b</sup>		
		Waste Sample 1 (2009.09.02)	Waste Sample 2 (2009.09.03)	Waste Sample 3 (2009.09.04)
1	pH Value	6.5	6.0	6.0
2	Chroma	15	10	21
3	Suspended Material	22	15	25
4	BOD5	8	6	5
5	Chemical Oxygen Demand	16	12	25
6	Ammonia Nitrogen	1	1	0.6
7	Total Nitrogen	2	1.6	1
8	Total Phosphorus	0.1	0.1	0.1
9	Total Organic Carbon	18	12	20
10	Sulfide	undetected	undetected	undetected
11	Nitrobenzenes	undetected	undetected	undetected
12	Phenyl amines	undetected	undetected	undetected
13	Total Cyanide	undetected	undetected	undetected
14	Total Mercury	undetected	undetected	undetected
15	Hexavalent Chrome	undetected	undetected	undetected

a. The wastewater samples were collected from from workshop wastewater outfalls;

b. The unit is mg/L (except pH and Chroma).