One-Pot Three-Component Synthesis of Spirooxindoles Catalyzed by Hexamethylenetetramine in Water

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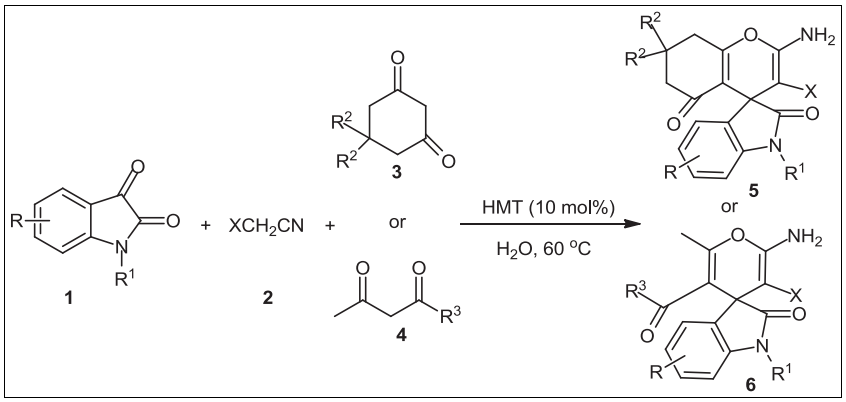

A simple, convenient, and multicomponent strategy for synthesis of spirooxindole derivatives has been developed. This strategy provides a rapid access to construct a diversity-oriented library of spirooxindoles by using three simple and readily available isatin, malononitrile or cyanoacetic ester, and 1,3-dicarbonyl compound catalyzed by hexamethylenetetramine in water.

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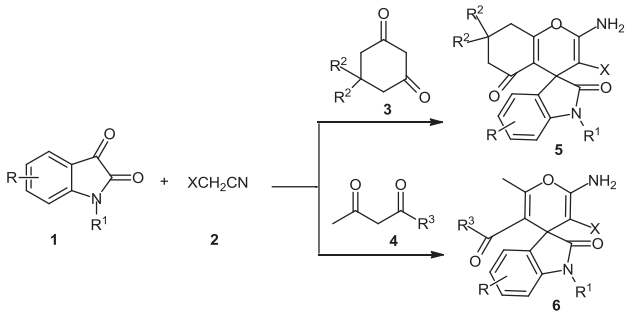
INTRODUCTION

Multicomponent reactions (MCRs) represent a highly valuable synthetic tool for the construction of novel and complex molecular structures because of their environmentally friendly atom economy and high-throughput generation of organic compounds. If such reaction could be run in an innocuous solvent, they would thus comply with most of the green chemistry principles [1].

Spirooxindoles occupy an important place in the area of heterocyclic chemistry because they are frequently found in numerous natural and synthetic products along with useful biopharmaceutical, physiopharmaceutical, and pharmaceutical activities [2–7]. As a consequence, great efforts have been devoted to the construction of diversely structured spirooxindole-fused heterocycles in the past few years [8–23]. However, there have been few reports on the synthesis of one unique spirocyclic oxindole, incorporating a 2-amino-4*H*-pyran-3-carbonitrile ring at the C3 position of oxindole [24]. The classical method for the preparation of these compounds involves a two-step reaction [3]. Recently, three-component reactions of isatin, malononitrile, and 1,3-dicarbonyl compound provide new elegant procedures for the clean synthesis of these functionalized spirocyclic oxindoles in the presence of triethylamine [25], sodium stearate [26], tris(2-hydroxyethyl) amine [27], ammonium chloride [28], ethylenediamine diacetate [29], $InCl_3$ [30], tetrabutylammonium bromide [31], triethylbenzylammonium chloride [32], tetrabutylammonium fluoride [33], β-cyclodextrin [34], L-proline [35,36], and lipase [37]. This reaction can also be performed by electrocatalytic strategy [38] or in poly(ethylene glycol) [39]. Despite the availability of these methods, because of the importance of these heterocyclic compounds from pharmaceutical, industrial, and synthetic points of view, there still remains a high demand for the development of an efficient, general, low-cost, and clean protocol to assemble these compounds.

Over the past few years, hexamethylenetetramine (HMT) has been shown to be effective, alternative, and promising catalyst because of its low price, ready availability, nontoxicity, and stability. It has been utilized as a catalyst for the Baylis–Hillman reaction [40], transesterification of β-keto esters [41], and synthesis of dihydropyrano[3,2-*c*]chromene derivatives [42]. Additionally, organic reactions performed in aqueous media have become increasing useful tools not only in light of environmental concerns but also with respect to the unique properties of water in promoting reactions and enhancing selectivities. As part of an ongoing research devoted to the development of efficient protocols for the preparation of substituted heterocycles and in a continuation of our recently reported works on MCRs [43–50], herein, we report HMT-catalyzed three-component coupling of isatin, malononitrile or cyanoacetic ester, and 1,3-dicarbonyl compound for the synthesis of spirooxindoles in water (Scheme 1).

Scheme 1. Synthesis of spirooxindoles catalyzed by hexamethylenetetramine.



RESULTS AND DISCUSSION

Initially, the efficacy of various catalysts was examined for the model reaction of isatin, malononitrile, and dimedone under different reaction conditions. The results are summarized in Table 1. It was showed that the reaction was rather sluggish and resulted in poor yield (18%) in the absence of catalysts when the reaction was carried out in water for 5 h at 60°C (Table 1, entry 22), which indicated that the catalysts should be necessary for this transformation. Various catalysts, including tetrabutylammonium bromide, LiBr, $(NH_4)_2Ce(NO_3)_6$, $SrCl_2$, H_3BO_3, tungstophosphoric acid, phosphomolybdic acid, silicotungstic acid, CuO, MgO, $(NH_4)_2HPO_4$, and HMT were screened in our model reaction. Among them, HMT was proven to be the most efficient catalyst for this reaction. The preceding reaction was also examined in various solvents. The results indicated that a very low yield of the desired product was obtained when dichloromethane, acetonitrile, tetrahydrofuran were used as solvents. The best yield was obtained when the reaction was performed in water, which maybe be attributable to the proton nature of water, and it accelerated the reaction compared with other solvents and solvent-free condition. It was also observed that the use of 10 mol% catalyst is sufficient to promote the reaction. Larger amount of the catalyst did not improve the yields. We also examined the influence of temperature on the reaction yields. It was found that no desired product was obtained at room temperature (Table 1, entry 25). The reaction time dramatically decreased from 1.5 h to 20 min when the reaction temperature increased from 40°C to 60°C, and the yield of 95% was obtained (Table 1, entry 12).

Under the preceding optimized conditions, the scope of this new MCR process was next investigated using a variety of structurally different isatins, malononitrile or cyanoacetic ester, and 1,3-cyclohexanedione or 5,5-dimethyl-1,3-cyclohexadione as substrates, and the results are summarized in Table 2. Gratifyingly, we found that this HMT-catalyzed three-component reaction worked well for a wide range of isatins carrying electron-donating and

Table 1

Optimization of reaction conditions for the model reaction of isatin, malononitrile, and dimedone.

Entry	Catalyst	Catalyst loading (mol%)	Solvent	Temperature (°C)	Time (min)	Yield (%)[a]
1	Tetrabutylammonium bromide	10	H_2O	60	60	90
2	LiBr	10	H_2O	60	60	85
3	$(NH_4)_2Ce(NO_3)_6$	10	H_2O	60	120	75
4	$SrCl_2$	10	H_2O	60	120	76
5	H_3BO_3	10	H_2O	60	120	83
6	Tungstophosphoric acid	10	H_2O	60	80	83
7	Phosphomolybdic acid	10	H_2O	60	90	86
8	Silicotungstic acid	10	H_2O	60	90	90
9	CuO	10	H_2O	60	40	83
10	MgO	10	H_2O	60	600	15
11	$(NH_4)_2HPO_4$	10	H_2O	60	100	90
12	HMT	10	H_2O	60	20	95
13	HMT	10	no	60	300	20
14	HMT	10	CH_2Cl_2	Reflux	360	32
15	HMT	10	MeOH	Reflux	40	81
16	HMT	10	EtOH	Reflux	40	84
17	HMT	10	CH_3CN	Reflux	250	12
18	HMT	10	DMF	60	20	90
19	HMT	10	THF	60	250	13
20	HMT	10	[BMIM][BF_4]	60	45	94
21	HMT	10	[BMIM][PF_6]	60	120	8
22	HMT	0	H_2O	60	300	18
23	HMT	5	H_2O	60	40	85
24	HMT	20	H_2O	60	20	95
25	HMT	10	H_2O	20	180	0
26	HMT	10	H_2O	40	90	81
27	HMT	10	H_2O	80	20	95

[a]Isolated yields.

electron-withdrawing substituents on the benzene ring and provided easy access to annulated 2-amino-spiro[(3′*H*)-indol-3′,4-(4*H*)-pyrans] (**5**) in good to excellent yields. It could be found that this transformation involving less reactive cyanoacetic esters (Table 2, entries 19–25) gave corresponding spirooxindoles in slight lower yield and required longer reaction time than malononitrile.

The scope of this methodology was further broadened by using acyclic 1,3-dicarbonyl compounds such as methyl acetoacetate, ethyl acetoacetate, 2-methoxyethyl acetoacetate, and acetylacetone in the three-compound reaction. A series of nonannulated 2-amino-spiro[(3′*H*)-indol-3′,4-(4*H*)-pyrans] (**6**) was also obtained in high yields as expected (Table 3).

To assess the feasibility of applying this method on a preparative scale, we carried out the model reaction on a 10 mmol scale. As expected, the reaction proceeded smoothly, similar to the case in a smaller scale, and compound **5h** was obtained in 93% yield. This preliminary result opens the possibility of a gram-scale entry to biologically relevant spirooxindoles.

The structures of the compounds **5** and **6** were elucidated through IR, ^{1}H NMR, ^{13}C NMR, and elemental analysis. High purity of products was also confirmed by performing single XRD of crystalline compound **5g** as shown in Figure 1.

Table 2

Three-component synthesis of annulated 2-amino-spiro[(3′*H*)-indol-3′,4-(4*H*)-pyrans] (**5**).

Entry	R	R^1	X	R^2	Products	Time (min)	Yield (%)[a]	mp (°C)	Lit. mp (°C)
1	H	H	CN	H	**5a**	30	95	308–310	310–311 [36]
2	H	Me	CN	H	**5b**	35	83	243–245	245–246 [36]
3	H	COMe	CN	H	**5c**	35	85	253–254	251–252 [36]
4	5-Me	H	CN	H	**5d**	30	90	294–295	291–293 [29]
5	5-Cl	H	CN	H	**5e**	60	91	289–291	288–290 [37]
6	5-Br	H	CN	H	**5f**	60	92	299–301	298–300 [29]
7	5-NO_2	H	CN	H	**5g**	65	88	>300	>300 [37]
8	H	H	CN	Me	**5h**	20	95	286–288	287–289 [34]
9	H	Me	CN	Me	**5i**	25	82	254–256	253–255 [39]
10	H	COMe	CN	Me	**5j**	25	83	235–236	232–233 [36]
11	5-Me	H	CN	Me	**5k**	25	86	280–281	275–276 [29]
12	5,7-Me_2	H	CN	Me	**5l**	30	85	>350	
13	5-Cl	H	CN	Me	**5m**	60	91	288–290	286–288 [37]
14	7-Cl	H	CN	Me	**5n**	60	90	280–281	278–280 [39]
15	4-Br	H	CN	Me	**5o**	80	88	313–315	312–315 [34]
16	5-Br	H	CN	Me	**5p**	95	86	306–307	304–305 [36]
17	5-I	H	CN	Me	**5q**	90	88	345–347	
18	5-NO_2	H	CN	Me	**5r**	60	85	302–304	302–304 [39]
19	H	H	COOEt	H	**5s**	50	90	282–283	280–283 [35]
20	H	Me	COOEt	H	**5t**	60	86	290–292	289–291 [35]
21	5-Me	H	COOEt	H	**5u**	60	88	256–258	257 [27]
22	H	H	COOMe	Me	**5v**	60	92	256–257	251–253 [32]
23	H	H	COOEt	Me	**5w**	50	91	270–272	269–271 [37]
24	5-Cl	H	COOEt	Me	**5x**	150	75	272–273	271–272 [37]
25	7-Cl	H	COOMe	Me	**5y**	120	80	280–281	278–279 [32]

[a]Isolated yields.

Table 3

Three-component synthesis of nonannulated 2-amino-spiro[(3′*H*)-indol-3′,4-(4*H*)-pyrans] (**6**).

Entry	R	X	R^3	Products	Time (min)	Yield (%)[a]	mp (°C)	Lit. mp (°C)
1	H	CN	OMe	**6a**	120	75	280–281	
2	H	CN	OEt	**6b**	100	92	260–261	255–256 [29]
3	H	CN	OCH_2CH_2OMe	**6c**	100	93	179–181	
4	5-Cl	CN	OEt	**6d**	120	90	277–278	263–265 [37]
5	5-Cl	CN	OCH_2CH_2OMe	**6e**	120	88	235–236	
6	5-Br	CN	OCH_2CH_2OMe	**6f**	120	91	221–222	
7	5-NO_2	CN	OEt	**6g**	150	89	251–252	247–249 [37]
8	5-NO_2	CN	OCH_2CH_2OMe	**6h**	130	91	215–217	
9	H	CN	Me	**6i**	150	65	242–243	240 [27]
10	H	COOEt	Me	**6j**	150	60	235–236	235 [27]

[a]Isolated yields.

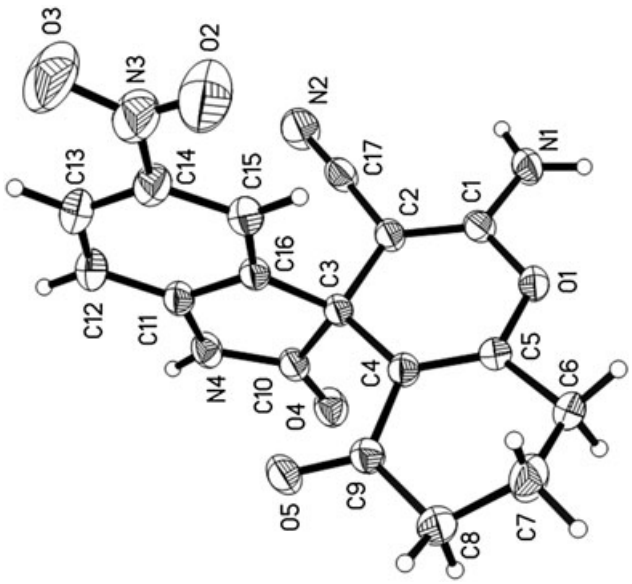


Figure 1. ORTEP diagram of compound **5g**.

In summary, we have developed a highly efficient HMT-catalyzed three-component, one-pot methodology for the synthesis of spirooxindoles via condensation of isatin, malononitrile or cyanoacetic ester, and 1,3-dicarbonyl compound. Simplicity, high yield, short reaction time, easy purification, efficiency, and economic availability of the catalyst are the salient features of this method. Further expansion of the reaction scope and synthetic application of this protocol are in progress in our laboratory.

EXPERIMENTAL

IR spectra were recorded with a Shimadzu FTIR-8900 spectrometer (Japan) using KBr plates. ^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker DRX-500 spectrometer at 500 MHz (^{1}H) and 125 MHz (^{13}C), respectively, using DMSO-d_6 as solvent and TMS as internal standard. Elemental analyses were carried out on a Vario EL III CHNOS elemental analyzer. Crystal data were collected with Bruker Smart Apex-II CCD diffractometer using graphite monochromated Mo Kα radiation ($\lambda = 0.71073$ Å) at 298 K. Commercially available reagents were used without further purification.

General procedure for synthesis of spirooxindole (5 and 6). HMT (0.1 mmol) was added to a mixture of isatin (1 mmol), malononitrile or cyanoacetic ester (1 mmol), and 1,3-dicarbonyl compound (1 mmol) in water (5 mL). The mixture was stirred at 60°C for an appropriate time. After completion of the reaction confirmed by TLC, the reaction mixture was cooled to room temperature. The precipitate was filtered and then recrystallized from EtOH to afford pure desired products.

2-Amino-5′,7,7,7′-tetramethyl-2′,5-dioxo-5,6,7,8-tetrahydrospiro[chromene-4,3′-indoline]-3-carbonitrile (5l). This compound was obtained as white solid; IR: 3323, 3157, 2191, 1724, 1685, 1654, 1624, 1604, 1481, 1473, 1352, 1301, 1224, 1327, 1301, 1224, 1201, 1055, 869 cm^{-1}; ^{1}H NMR (500 MHz, DMSO-d_6): 1.00 (s, 3H), 1.03 (s, 3H), 2.08–2.16 (m, 2H), 2.17 (s, 3H), 2.18 (s, 3H), 2.55 (s, 2H), 6.58 (s, 1H), 6.76 (s, 1H), 7.16 (s, 2H), 10.31 (s, 1H) ppm; ^{13}C NMR (125 MHz, DMSO-d_6): 16.7, 19.0, 21.0, 27.6, 32.4,47.5, 50.5, 56.5, 58.4, 111.5, 117.9, 118,5, 121.4, 130.5, 130.8, 134.7, 138.6, 159.1, 178.8, 195.2 ppm. *Anal*. Calcd for $C_{21}H_{21}N_3O_3$: C, 69.41; H, 5.82; N, 11.56. Found: C, 69.36; H, 6.01; N, 11.68.

2-Amino-5′-iodo-7,7-dimethyl-2′,5-dioxo-5,6,7,8-tetrahydrospiro[chromene-4,3′-indoline]-3-carbonitrile (5q). This compound was obtained as white solid; IR: 3289, 2195, 1726, 1662, 1612, 1599, 1473, 1352, 1328, 1223, 1168, 1055, 810 cm^{-1}; ^{1}H NMR (500 MHz, DMSO-d_6): 1.00 (s, 3H), 1.03 (s, 3H), 1.98–2.15 (m, 2H), 2.58 (s, 2H), 6.65 (s, 1H), 7.31–7.47 (m, 4H), 10.52 (s, 1H) ppm; ^{13}C NMR (125 MHz, DMSO-d_6): 27.1, 27.5, 31.9, 46.8, 49.9, 56.8, 84.3, 110.2, 111.7, 117.2, 131.3, 136.7, 137.0, 141.9, 158.8, 160.5, 177.4, 195.0 ppm. *Anal*. Calcd for $C_{19}H_{16}IN_3O_3$: C, 49.47; H, 3.50; N, 9.11. Found: C, 49.66; H, 3.32; N, 8.99.

Methyl 2′-amino-3′-cyano-6′-methyl-2-oxospiro[indoline-3,4′-pyran]-5′-carboxylate (6a). This compound was obtained as orange solid; IR: 3303, 3145, 2196, 1724, 1678, 1631, 1616, 1596, 1471, 1411, 1328, 1288, 1209, 1132, 1070, 759 cm^{-1}; ^{1}H NMR (500 MHz, DMSO-d_6): 2.31 (s, 3H), 3.34 (s, 3H), 6.79 (d, $J = 7.5$ Hz, 1H), 6.93 (t, $J = 7.5$ Hz, 1H), 7.04 (d, $J = 7.5$ Hz, 1H), 7.11 (s, 2H), 7.16 (t, $J = 7.5$ Hz, 1H), 10.36 (s, 1H) ppm; ^{13}C NMR (125 MHz, DMSO-d_6): 19.3, 49.5, 51.9, 56.9, 105.3, 109.7, 117.9, 121.3, 123.8, 129.0, 134.9, 142.4, 158.9, 159.4, 165.6, 178.9 ppm. *Anal*. Calcd for $C_{16}H_{13}N_3O_4$: C, 61.73; H, 4.21; N, 13.50. Found: C, 61.90; H, 4.03; N, 13.32.

2-Methoxyethyl 2′-amino-3′-cyano-6′-methyl-2-oxospiro[indoline-3,4′-pyran]-5′-carboxylate (6c). This compound was obtained as white solid; IR: 3190, 2191, 1721, 1679, 1620, 1595, 1472, 1385, 1288, 1216, 1137, 1072, 755 cm^{-1}; ^{1}H NMR (500 MHz, DMSO-d_6): 2.32 (s, 3H), 3.12 (s, 3H), 3.16–3.23 (m, 2H), 3.83–3.93 (m, 2H), 6.79 (d, $J = 7.5$ Hz, 1H), 6.92 (t, $J = 7.5$ Hz, 1H), 7.05 (d, $J = 7.5$ Hz, 1H), 7.14 (s, 2H), 7.17 (t, $J = 7.5$ Hz, 1H), 10.36 (s, 1H) ppm; ^{13}C NMR (125 MHz, DMSO-d_6): 19.2, 49.4, 57.1, 58.4, 63.8, 69.5, 105.2, 109.9, 117.9, 122.3, 123.8, 128.9, 134.9, 142.5, 159.2, 165.0, 178.9 ppm. *Anal*. Calcd for $C_{18}H_{17}N_3O_5$: C, 60.84; H, 4.82; N, 11.83. Found: C, 61.02; H, 5.01; N, 11.66.

2-Methoxyethyl 2′-amino-5-chloro-3′-cyano-6′-methyl-2-oxospiro[indoline-3,4′-pyran]-5′-carboxylate (6e). This compound was obtained as white solid; IR: 3203, 2208, 1718, 1701, 1654, 1596, 1477, 1419, 1380, 1282, 1249, 1224, 1078, 829 cm^{-1}; ^{1}H NMR (500 MHz, DMSO-d_6): 2.25 (s, 3H), 3.03 (s, 3H), 3.13 (t, $J = 5.0$ Hz, 2H), 3.37–3.91 (m, 2H), 6.71 (d, $J = 8.5$ Hz, 1H), 7.08 (d, $J = 2.0$ Hz, 1H), 7.12 (dd, $J = 8.5$, 2.0 Hz, 1H), 7.13 (s, 2H), 10.41 (s, 1H); ^{13}C NMR (125 MHz, DMSO-d_6): 19.4, 49.8, 56.5, 58.4, 63.9, 69.6, 104.3, 111.3, 117.8, 124.0, 126.2, 128.8, 137.2, 141.4, 159.3, 160.2, 164.9, 178.7 ppm. *Anal*. Calcd for $C_{18}H_{16}ClN_3O_5$: C, 55.46; H, 4.14; Cl, 9.10; N, 10.78. Found: C, 5.62; H, 3.96; N, 10.93.

2-Methoxyethyl 2′-amino-5-bromo-3′-cyano-6′-methyl-2-oxospiro[indoline-3,4′-pyran]-5′-carboxylate (6f). This compound was obtained as white solid; IR: 3318, 2196, 1672, 1474, 1290, 1069, 981, 747 cm^{-1}; ^{1}H NMR (500 MHz, DMSO-d_6): 2.34 (s, 3H), 3.12 (s, 3H), 3.23 (t, $J = 5.0$ Hz, 2H), 3.87–4.01 (m, 2H), 6.76 (d, $J = 8.5$ Hz, 1H), 7.23 (s, 2H), 7.28 (d, $J = 1.5$ Hz, 1H), 7.35 (dd, $J = 8.5$, 1.5 Hz, 1H), 10.51 (s, 1H) ppm; ^{13}C NMR (125 MHz, DMSO-d_6): 18.9, 49.3, 56.1, 57.9, 63.5, 69.1, 103.7, 110.3, 111.4, 117.3, 126.2, 131.2, 137.1, 140.7, 141.3, 145.6, 158.9, 159.8, 164.4, 178.1 ppm. *Anal*. Calcd for $C_{18}H_{16}BrN_3O_5$: C, 49.79; H, 3.71; N, 9.68. Found: C, 49.96; H, 3.58; N, 9.85.

2-Methoxyethyl 2′-amino-3′-cyano-6′-methyl-5-nitro-2-oxospiro [indoline-3,4′-pyran]-5′-carboxylate (6h). This compound was obtained as white solid; IR: 3326, 2195, 1674, 1603, 1524, 1480, 1383, 1338, 1290, 1258, 1212, 1136, 1070, 982, 840 cm^{-1}; ^1H NMR (500 MHz, DMSO-d_6): 2.30 (s, 3H), 3.00 (s, 3H), 3.08–3.15 (m, 2H), 3.77–3.92 (m, 2H), 6.92 (d, $J = 8.5$ Hz, 1H), 7.25 (s, 2H), 7.93 (d, $J = 2.0$ Hz, 1H), 8.09 (dd, $J = 8.5$, 2.0 Hz, 1H), 11.40 (s, 1H) ppm; ^{13}C NMR (125 MHz, DMSO-d_6): 19.6, 49.7, 55.8, 58.3, 64.0, 69.6, 103.5, 110.1,117.6, 119.5, 126.4, 136.3, 142.9, 159.4, 161.2, 164.8, 179.4 ppm. *Anal*. Calcd for $C_{18}H_{16}N_4O_7$: C, 54.00; H, 4.03; N, 13.99. Found: C, 53.82; H, 3.95; N, 14.16.

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