Note

The Decomposition Reaction of 4-Acetylsydnones Arylhydrazones

Wen-Fa Kuo[a] (• • • •), Chun-Yen Chiu[b] (• • • •), I-Tzu Lin[a] (• • • •), Shu-Ya Sheu[a] (• • • •), Kao-Hung Lin[a] (• • • •) and Mou-Yung Yeh*[a] (• • • •)
[a]*Department of Chemistry, National Cheng Kung University, Tainan, Taiwan 70101, R.O.C.*
[b]*Department of Environmental Engineering and Health, Tajen Institute of Technology, Ping Tung, Taiwan 907, R.O.C.*

The acidic decomposition of 4-acetylsydnones arylhydrazones **2** results in the formation of 4-arylhydrazo-1,2-pyrazolin-5-ones **3** and 4-arylamino-1,2,3-triazoles **4**, respectively. The reactions of 4-acetylsydnones **1** with arylhydrazines **5** afford compounds **3** as the only products in the absence of solvent.

INTRODUCTION

In the literature, only the substituted derivatives on the acyl functional group of 4-acylsydnones **1** has been investigated.[1-4] In this report, however, synthetic studies using compounds **1** through decomposition of the sydnone ring to give heterocyclic compounds are of great interest. We also present the decomposition of 4-acetylsydnones arylhydrazones **2** which would be transferred to two heterocyclic compounds, 4-arylhydrazo-1,2-pyrazolin-5-ones **3** and 4-amino-1,2,3-triazoles **4**, in the presence of hot HCl via three subsequent steps: N1-attack, sydnone ring opening and ring formation. In addition, the reactions of 4-acetylsydnones **1** with arylhydrazine **5** were also examined.

Various synthetic routes developed for 1,2-pyrazolin-5-ones[5-9] and 1,2,3-triazoles[10-16] have been reported. In this study, we report the decomposition of sydnone derivatives yielding compounds **3** and **4**.

RESULTS AND DISCUSSION

From the acidic decomposition of 4-acetylsydnones arylhydrazones **2** in the presence of hot HCl, 4-arylhydrazo-1,2-pyrazolin-5-ones **3** and 4-arylamino-1,2,3-triazoles **4** were obtained with considerably low yields. Instead, high yields of hydrolysis products, 4-acetylsydnones **1**, were obtained. In order to acquire satisfactory yields of compounds **3** and **4**, we modified the above synthetic method. Excess arylhydrazines **5** were used to react with compounds **1**. The resulting products **2** were not isolated and $HCl_{(aq)}$ was added to proceed acidic decomposition. Using this method, we obtained compound **3** and **4** with high yields. This is well understood that the condensations were reversible reactions and the excess hydrazines would result in a high quantity of hydrazones in the reaction solution to perform acidic decomposition. The yields of decomposition are summarized in Table 1.

According to the spectral data and X-ray structure (Fig. 1), compounds **3a-3d** possess two aryl substituents which originated from corresponding arylhydrazines **5**. On the other hand, we have also isolated arylamines as side products. On the spectral analysis of compounds **3**, the N-H stretching was not obvious in the IR spectra. The N-H resonance signal of ^1H NMR in each compound is broad and down field shift to 13 ppm. Besides, the C=O stretching was red shift to 1650 cm^{-1} due to the intramolecular hydrogen bonding of N-H and C=O. Hence, we proposed the reaction mechanism as outlined in Scheme I. N1-attack resulted in the ring opening of sydnone and the formation of C=N double bond by denitrosolation. The C=N was then attacked by free arylhydrazine **5** to give 4-arylhydrazo-1,2-pyrazolin-5-ones **3**. In addition, the structure identifications of 4-arylamino-1,2,3-triazoles **4** by various spectroscopic techniques were similar to those reported in the literature.[16] This proposed reaction mechanism of compounds **4** was also given in Scheme I, which refers to the photolysis mechanism of the sydnone ring reported by Marky.[15] In the presence of HCl, the sydnone ring opened and diazirine **6** was formed as an intermediate by decarboxylation. Then N1-attack resulted in heterolysis of N-N bond of diazirine **6** to give 4-arylamine-1,2,3-triazoles **4**.

Furthermore, the acidic decompositions of 4-acetyl-3-arylsydnones **1** were also investigated. The results revealed that only the 4-acetyl-3-(4-chlorophenyl)sydnone **1a** was transferred to compounds **3d** and **4j** with low yields (5% and 8%, respectively). For other 4-acetyl-3-arylsydnones, the acidic decomposition did not take place at all and collected compounds were only starting materials. This may be explained that compound **1a** was more unstable than other 4-acetyl-3-arylsydnones **1**. Hence a small amount of compound **1a** was easily decomposed to 4-chlorophenylhydra-

Table 1. Reactions of Sydnones **1** with Arylhydrazines **5**

Ar	Ar'	Compounds **3**[a]			Compounds **4**[a]	
			Yield% (1)	Yield% (2)		Yield% (1)
p-$MeOC_6H_4$	p-MeC_6H_4	**3a**	42	-	**4a**	35
p-MeC_6H_4	p-MeC_6H_4	**3a**	16	43	**4b**	24
p-ClC_6H_4	p-MeC_6H_4	**3a**	35	42	**4c**	52
C_6H_5	p-MeC_6H_4	**3a**	26	-	**4d**	57
p-$EtOC_6H_4$	p-MeC_6H_4	**3a**	32	-	**4e**	25
p-$MeOC_6H_4$	C_6H_5	**3b**	40	-	**4f**	14
p-MeC_6H_4	C_6H_5	**3b**	30	61	**4g**	25
p-ClC_6H_4	C_6H_5	**3b**	28	59	**4h**	27
p-$EtOC_6H_4$	C_6H_5	**3b**	32	-	**4i**	18
p-MeC_6H_4	m-MeC_6H_4	**3c**	-	67		-
p-ClC_6H_4	m-MeC_6H_4	**3c**	-	56		-
p-ClC_6H_4	p-ClC_6H_4	**3d**[b]	5	-	**4j**[b]	8

[a] Yields: (1) acidic decomposition; (2) reaction in the absence of solvent and acid.
[b] Starting material: 4-acetyl-3-(4-chlorophenyl)sydnone.

zine, which would further be condensed with remained 4-acetyl-3-(4-chlorophenyl)sydnone (**1a**) and followed by subsequent decomposition with HCl as demonstrated in Scheme I.

Further study has been carried out on reactions between 4-acetylsydnones **1** and arylhydrazine **5** under more violent conditions (100 °C) in the absence of solvent and acid. 4-Acetylsydnones arylhydrazones **2** were formed as intermediates and 4-arylhydrazo-1,2-pyrazolin-5-ones **3** were then obtained as the only products with satisfactory yields. 4-Amino-1,2,3-triazoles **4** was not produced in this reaction because the decarboxylation of the sydnone ring did not proceed in the absence of HCl. The yields of 4-arylhydrazo-1,2-pyrazolin-5-ones **3** are presented in Table 1.

CONCLUSION

A new synthetic route of 4-arylazo-1,2-pyrazolin-5-ones **3** and 4-arylamino-1,2,3-triazoles **4** are presented in this report. These results suggest that excess arylhydrazines are needed for the acidic decomposition of 4-acetylsydnones arylhydrazones **2**. The preparation of 4-arylazo-1,2-pyrazolin-5-ones **3** from the reactions of compounds **1** with arylhydrazines **5** in the absence of solvent and HCl is also a new and more efficient route.

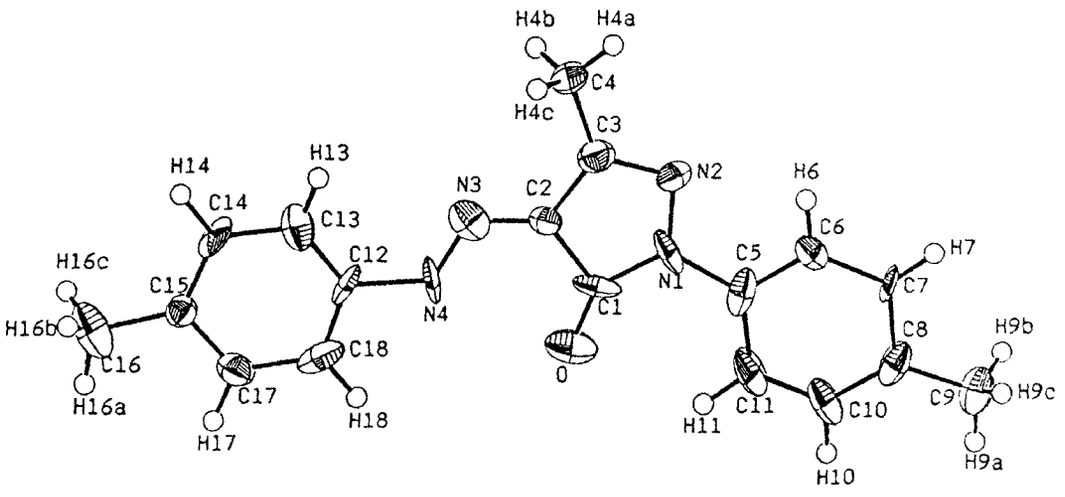
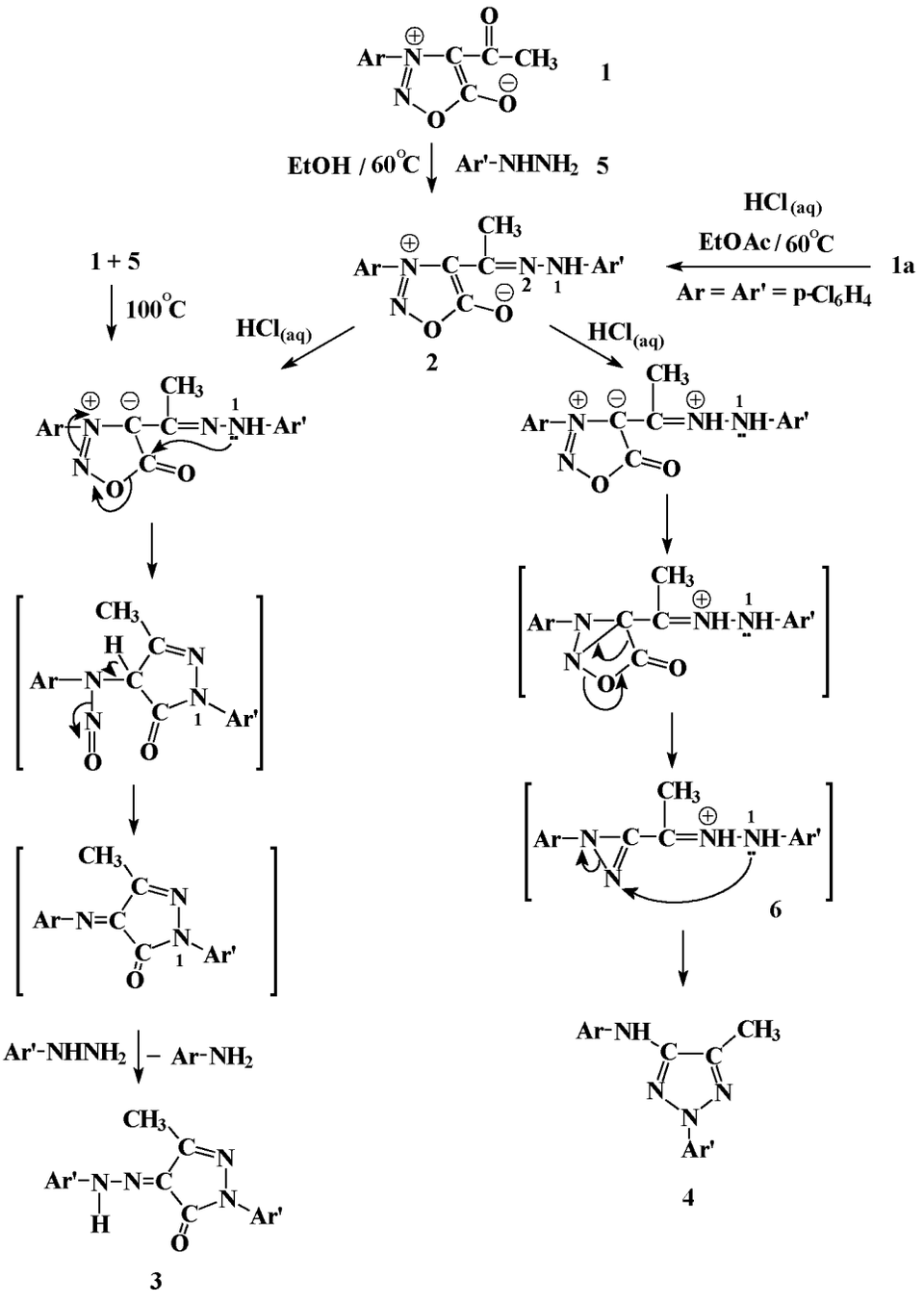


Fig. 1. ORTEP drawing of the X-ray structure of **3a**.

Scheme I



EXPERIMENTAL SECTION

Melting points (Buchi 535 apparatus) are uncorrected. IR spectra were recorded on a Hitachi 270-30 infrared spectrometer. NMR spectra were measured on a Bruker AMX-200 NMR spectrometer with tetramethylsilane as internal standard. The mass spectra were registered on a Finnigan MAT TSQ-46C spectrometer at an ionizing potential 70 eV. Elemental analyses were performed on Heraeus CHN-O-Rapid and Tacussel Coulomax 78 analyzers. X-ray analysis was made with a Nonius CAD-4 diffractometer. Column chromatography was carried out on silica gel (Kieselgel 100, 230-400

mesh, E. Merck).

Preparation of 4-arylhydrazo-1,2-pyrazolin-5-ones 3 and 4-arylamino-1,2,3-triazoles 4

A solution of 4-acetyl-3-(4-methoxyphenyl)sydnone (0.3 g, 1.3 mmol) and 4-methylphenylhydrazine hydrochloride (0.6 g, 3.8 mmol) in 10 mL of EtOH was stirred at 60 °C for 24 h. HCl (2 mL, 37%) was added and stirred continuously for 3 h. After reactions were complete, the precipitate was collected by filtration and recrystallization with EtOAc to give 0.17 g of **3a** (42%). The filtrate was removed under reduced pressure and the crude solid was subjected to chromatography (EtOAc : n-hexane = 1 : 4) to give 0.13 g of **4a** (35%).

The acidic decomposition of 4-acetyl-3-(4-chlorophenyl)-sydnone 1a

A solution of compound **1a** (0.5 g, 2.1 mmol) in 5 mL of EtOAc was added into 1.0 mL of HCl (37%). The reaction mixture was stirred at 60 °C for 72 h. The precipitated solid, **3d**, was collected by filtration and then recrystallized in EtOAc with 0.036 g of **3d** (5%). The filtrate was subjected to chromatography (EtOAc : n-hexane = 1 : 5) to give 0.054 g of **4j** (8%).

The acidic decomposition of 4-acetyl-3-arylsydnones arylhydrazones 2

A solution of 0.3 g of compounds **2** and 1 mL of HCl (37%) in 5 mL of EtOH was stirred at 60 °C for 24 h. The precipitated solid was collected and recrystallized in EtOAc, and 8-12% of compound **3** was obtained. The filtrate was subjected to chromatography (EtOAc : n-hexane = 1 : 4) to give compounds **4** (3-7%) and 4-acetyl-3-arylsydnones (70-80%).

The reactions of 4-acetyl-3-arylsydnones 1 with arylhydrazines 5 in the absence of HCl and solvent

0.25 g of 4-acety-3-(4-methylphenyl)sydnone (1.1 mmol) was added to 2 mL of phenylhydrazine (20 mmol). The solution was then heated to 100 °C and stirred for 5 h. After reactions were complete, the solution was dropped into acidic ice water (30 mL of water and 2 mL (37%) of HCl), precipitate was then collected and recrystallization was accomplished with EtOAc or acetone to give 0.2 g of **3b** (61%).

1-(4-Methylphenyl)-4-(4-methylphenylhydrazo)-3-methyl-1,2-pyrazolin-5-one (3a)

Red powder; mp 218-219 °C, IR (KBr), cm^{-1}: 1653 (ν C=O), 1593 (ν C=N), ^1H NMR (DMSO-d_6), δ 13.4 (br, 1H), 7.78 (d, J = 8.5 Hz, 2H), 7.49 (d, J = 8.4 Hz, 2H), 7.27-7.22 (m, 4H), 2.31 (s, 3H), 2.30 (s, 3H), 2.27 (s, 3H), EIMS (70 eV), *m/z* (%): 306 (M^+, 100), 215 (21), 107 (31), 91 (78), 79 (27), 65 (33). Anal. Calcd. for $C_{18}H_{18}N_4O$: C, 70.57; H, 5.92; N, 18.29. Found C, 70.40; H, 5.95; N, 18.18.

1-Phenyl-4-phenylhydrazo-3-methyl-1,2-pyrazolin-5-one (3b)

Red powder; mp 153.5-154.5 °C, IR (KBr), cm^{-1}: 1659 (ν C=O), 1596 (ν C=N), ^1H NMR (DMSO-d_6), δ 13.27 (br, 1H), 7.92 (d, J = 7.4 Hz, 2H), 7.62 (d, J = 7.4 Hz, 2H), 7.48-7.18 (m, 6H), 2.29 (s, 3H), EIMS (70 eV), *m/z* (%): 278 (M^+, 100), 252 (21), 201 (42), 173 (10), 160 (13), 93 (39), 77 (75). Anal. Calcd. for $C_{16}H_{14}N_4O$: C, 69.05; H, 5.07; N, 20.13. Found C, 69.11; H, 5.13; N, 20.22.

1-(3-Methylphenyl)-4-(3-methylphenylhydrazo)-3-methyl-1,2-pyrazolin-5-one (3c)

Red powder; mp 116-116.5 °C, IR (KBr), cm^{-1}: 1650 (ν C=O), 1590 (ν C=N), ^1H NMR (DMSO-d_6), δ 13.31 (br, 1H), 7.74-7.71 (m, 2H), 7.41-7.01 (m, 6H), 2.34 (s, 3H), 2.32 (s, 3H), 2.29 (s, 3H), EIMS (70 eV), *m/z* (%): 306 (M^+, 100), 215 (21), 187 (4), 91 (31), 77 (6). Anal. Calcd. for $C_{18}H_{18}N_4O$: C, 70.57; H, 5.92; N, 18.29. Found C, 70.56; H, 5.96; N, 18.30.

1-(4-Chlorophenyl)-4-(4-chlorophenylhydrazo)-3-methyl-1,2-pyrazolin-5-one (3d)

Red powder; mp 232-233 °C, IR (KBr), cm^{-1}: 1659 (ν C=O), 1595 (ν C=N), ^1H NMR (DMSO-d_6), δ 13.15 (br, 1H), 7.92 (d, J = 9.0 Hz, 2H), 7.75 (d, J = 8.9 Hz, 2H), 7.53-7.46 (m, 4H), 2.32 (s, 3H). EIMS (70 eV), *m/z* (%): 350 (M^++4, 11), 348 (M^++2, 65), 346 (M^+, 100), 235 (27), 207 (10), 192 (6), 111 (39), 99 (14). Anal. Calcd. for $C_{16}H_{12}N_4OCl_2$: C, 55.35; H, 3.48; N, 16.14. Found C, 55.30; H, 3.56; N, 16.25.

2-(4-Methylphenyl)-4-(4-methoxyphenylamino)-5-methyl-*2H*-1,2,3-triazole (4a)

Yellow powder; mp 135.5-136.5 °C, IR (KBr), cm^{-1}: 3382 (ν NH), 1608 (ν C=N), ^1H NMR (DMSO-d_6), δ 8.30 (s, 1H), 7.74 (d, J = 8.4 Hz, 2H), 7.52 (d, J = 9.0 Hz, 2H), 7.28 (d, J = 8.4 Hz, 2H), 6.89 (d, J = 9.0 Hz, 2H), 3.70 (s, 3H), 2.32 (s, 6H). EIMS (70 eV), *m/z* (%): 294 (M^+, 100), 279 (42), 147 (6), 119 (6), 105 (21), 91 (22). Anal. Calcd. for $C_{17}H_{18}N_4O$: C, 69.37; H, 6.16; N, 19.03. Found C, 69.22; H, 6.18; N, 19.15.

2-(4-Methylphenyl)-4-(4-methylphenylamino)-5-methyl-*2H*-1,2,3-triazole (4b)

Yellow powder; mp 115.5-116 °C, IR (KBr), cm^{-1}: 3400 (ν NH), 1602 (ν C=N), ^1H NMR (DMSO-d_6), δ 8.36 (s, 1H), 7.74 (d, J = 8.4 Hz, 2H), 7.44 (d, J = 8.4 Hz, 2H), 7.28 (d, J = 8.4 Hz, 2H), 7.08 (d, J = 8.4 Hz, 2H), 2.32 (s, 6H), 2.22 (s, 3H). EIMS (70 eV), *m/z* (%): 278 (M^+, 100), 105 (38), 91 (30), 78 (13), 65 (18), 51 (5). Anal. Calcd. for $C_{17}H_{18}N_4$: C, 73.35;

H, 6.52; N, 20.13. Found C, 73.24; H, 6.59; N, 20.21.

2-(4-Methylphenyl)-4-(4-chlorophenylamino)-5-methyl-*2H*-1,2,3-triazole (4c)

Yellow powder; mp 113-114 °C, IR (KBr), cm^{-1}: 3424 (ν NH), 1602 (ν C=N), ^{1}H NMR (DMSO-d_6), δ 8.68 (s, 1H), 7.76 (d, *J* = 8.5 Hz, 2H), 7.56 (d, *J* = 8.9 Hz, 2H), 7.33-7.27 (m, 4H), 2.33 (s, 6H). EIMS (70 eV), *m/z* (%): 300 (M^++2, 34), 298 (M^+, 100), 152 (7), 105 (42), 91 (19), 78 (10), 65 (11). Anal. Calcd. for $C_{16}H_{15}N_4Cl$: C, 64.32; H, 5.06; N, 18.75. Found C, 64.30; H, 5.11; N, 18.79.

2-(4-Methylphenyl)-4-phenylamino-5-methyl-*2H*-1,2,3-triazole (4d)

Yellow powder; mp 112.5-113.5 °C, IR (KBr), cm^{-1}: 3436 (ν NH), 1599 (ν C=N), ^{1}H NMR (DMSO-d_6), δ 8.50 (s, 1H), 7.77 (d, *J* = 8.4 Hz, 2H), 7.55 (d, *J* = 8.2 Hz, 2H), 7.32-7.23 (m, 4H), 6.87-6.80 (m, 1H), 2.33 (s, 3H), 2.34 (s, 3H). EIMS (70 eV), *m/z* (%): 264 (M^+, 100), 187 (6), 142 (5), 118 (6), 107 (23), 105 (36), 91 (21), 77 (20), 65 (11). Anal. Calcd. for $C_{16}H_{16}N_4$: C, 72.70; H, 6.10; N, 21.19. Found C, 72.85; H, 6.36; N, 21.46.

2-(4-Methylphenyl)-4-(4-ethoxyphenylamino)-5-methyl-*2H*-1,2,3-triazole (4e)

Yellow powder; mp 120-121 °C, IR (KBr), cm^{-1}: 3388 (ν NH), 1602 (ν C=N), ^{1}H NMR (DMSO-d_6), δ 8.27 (s, 1H), 7.73 (d, *J* = 8.1 Hz, 2H), 7.49 (d, *J* = 8.9 Hz, 2H), 7.28 (d, *J* = 8.1 Hz, 2H), 6.87 (d, *J* = 8.9 Hz, 2H), 3.96 (q, *J* = 6.9 Hz, 2H), 2.32 (s, 6H), 1.30 (t, *J* = 6.9 Hz, 3H). EIMS (70 eV), *m/z* (%): 308 (M^+, 100), 279 (65), 105 (29), 91 (30), 78 (10), 65 (14), 57 (9). Anal. Calcd. for $C_{18}H_{20}N_4O$: C, 70.11; H, 6.54; N, 18.17. Found C, 70.19; H, 6.67; N, 18.31.

4-(4-Methoxyphenylamino)-2-phenyl-5-methyl-*2H*-1,2,3-triazole (4f)

Yellow powder; mp 97.5-98.5 °C, IR (KBr), cm^{-1}: 3370 (ν NH), 1611 (ν C=N), ^{1}H NMR (DMSO-d_6), δ 8.34 (s, 1H), 7.85 (d, *J* = 7.8 Hz, 2H), 7.56-7.43 (m, 4H), 7.20-7.27 (m, 1H), 6.89 (d, *J* = 9.1 Hz, 2H), 3.71 (s, 3H), 2.33 (s, 3H). EIMS (70 eV), *m/z* (%): 280 (M^+, 100), 265 (43), 92 (11), 91 (19), 77 (23). Anal. Calcd. for $C_{16}H_{16}N_4O$: C, 68.55; H, 5.75; N, 19.99. Found C, 68.48; H, 5.83; N, 20.06.

4-(4-Methylphenylamino)-2-phenyl-5-methyl-*2H*-1,2,3-triazole (4g)

Yellow powder; mp 110-111 °C, IR (KBr), cm^{-1}: 3406 (ν NH), 1599 (ν C=N), ^{1}H NMR (DMSO-d_6), δ 8.42 (s, 1H), 7.86 (d, *J* = 8.0 Hz, 2H), 7.53-7.43 (m, 4H), 7.28-7.20 (m, 1H), 7.09 (d, *J* = 8.2 Hz, 2H), 2.34 (s, 3H), 2.23 (s, 3H). EIMS (70 eV), *m/z* (%): 264 (M^+, 100), 222 (16), 132 (13), 91 (39), 77 (26). Anal. Calcd. for $C_{16}H_{16}N_4$: C, 72.70; H, 6.10; N, 21.20. Found C, 72.58; H, 6.15; N, 21.33.

4-(4-Chlorophenylamino)-2-phenyl-5-methyl-*2H*-1,2,3-triazole (4h)

Yellow powder; mp 91.5-92.5 °C, IR (KBr), cm^{-1}: 3400 (ν NH), 1599 (ν C=N), ^{1}H NMR (DMSO-d_6), δ 8.73 (s, 1H), 7.89 (d, *J* = 7.7 Hz, 2H), 7.63-7.46 (m, 4H), 7.35-7.24 (m, 3H), 2.36 (s, 3H). EIMS (70 eV), *m/z* (%): 286 (M^++2, 43), 284 (M^+, 100), 92 (9), 77 (30), 64 (12). Anal. Calcd. for $C_{15}H_{13}N_4Cl$: C, 63.27; H, 4.60; N, 19.68. Found C, 63.21; H, 4.77; N, 19.78.

4-(4-Ethoxyphenylamino)-2-phenyl-5-methyl-*2H*-1,2,3-triazole (4i)

Yellow powder; mp 102.5-103 °C, IR (KBr), cm^{-1}: 3436 (ν NH), 1599 (ν C=N), ^{1}H NMR (DMSO-d_6), δ 8.32 (s, 1H), 7.84 (d, *J* = 7.8 Hz, 2H), 7.55-7.40 (m, 4H), 7.26-7.19 (m, 1H), 6.87 (d, *J* = 8.9 Hz, 2H), 3.95 (q, *J* = 6.9 Hz, 2H), 2.33 (s, 3H), 1.29 (t, *J* = 6.9 Hz, 3H). EIMS (70 eV), *m/z* (%): 294 (M^+, 100), 266 (32), 265 (76), 92 (21), 77 (36). Anal. Calcd. for $C_{17}H_{18}N_4O$: C, 69.37; H, 6.16; N, 19.03. Found C, 69.31; H, 6.20; N, 19.06.

2-(4-Chlorophenyl)-4-(4-chlorophenylamino)-5-methyl-*2H*-1,2,3-triazole (4j)

Yellow powder; mp 127.5-128.5 °C, IR (KBr), cm^{-1}: 3436 (ν NH), 1602 (ν C=N), ^{1}H NMR (DMSO-d_6), δ 8.77 (s, 1H), 7.87 (d, *J* = 8.8 Hz, 2H), 7.60 (d, *J* = 8.5 Hz, 2H), 7.55 (d, *J* = 8.8 Hz, 2H),7.32 (d, *J* = 8.5 Hz, 2H), 2.35 (s, 3H). EIMS (70 eV), *m/z* (%): 322 (M^++4, 11), 320 (M^++2, 63), 318 (M^+, 100), 264 (10), 219 (11), 193 (19), 179 (10), 163 (13), 152 (15), 149 (10), 138 (16), 127 (35),. Anal. Calcd. for $C_{15}H_{12}N_4Cl_2$: C, 56.44; H, 3.79; N, 17.55. Found C, 56.37; H, 3.82; N, 17.59.

ACKNOWLEDGMENT

We thank the National Science Council of the Republic of China for financial support of the work.

Received February 25, 2000.

Key Words

Acidic decomposition; 4-Acetylsydnones arylhydrazones; 4-Arylazo-1,2-pyrazolin-5-ones; 4-Arylamino-1,2,3-triazoles.

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