Synthesis of Spiro Thiazolo[3,2-*a*]pyrimidine Compounds by One-Pot Sequential 1,3-Dipolar Cycloadditions

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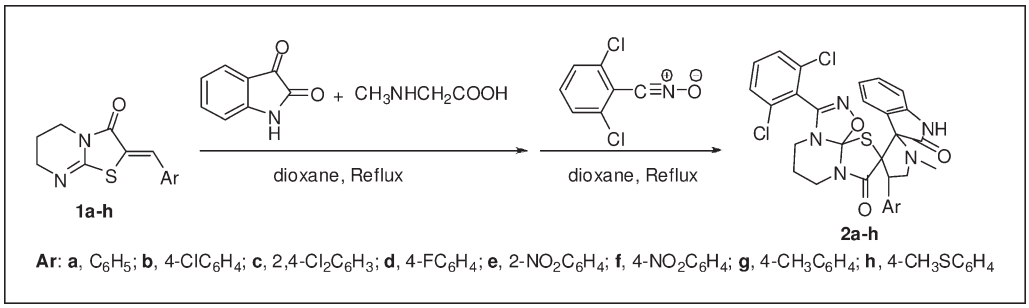

A new class of spiro thiazolo[3,2-*a*]pyrimidine compounds were synthesized by the one-pot sequential 1,3-dipolar cycloaddition of azomethine ylide (generated from isatin and sarcosine)–nitrile oxide to 2-arylmethylidene-6,7-dihydro-5*H*-thiazolo[3,2-*a*]pyrimidin-3-ones in moderate yields. The structures of all the products were characterized thoroughly by NMR, MS, IR, elemental analysis, and NMR together with X-ray crystallographic analysis.

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INTRODUCTION

Nitrile oxides are important intermediates as 1,3-dipoles in cycloaddition reaction [1]. The C, N double bond of pyridine, quinoline, isoquinoline, and 1,5-benzothiazepine as heterodipolarophile can also react with nitrile oxides to obtain oxadiazoles [2,3]. Oxadiazoles, the important bioisosteres for esters and amides in drug discovery, have been reported to have muscarinic agonist, benzodiazepine receptor agonist, 5-HT agonist, and antirhinoviral activities [4].

Thiazolo[3,2-*a*]pyrimidin-3-one derivatives are found to be associated with various biological activities such as antibacterial, antimicrobial, and anticancer activities [5].

The 1,3-dipolar cycloaddition reaction of azomethine ylides generated by a decarboxylative route from amino acids and isatin with exocyclic olefins represents an efficient method for the construction of the spirooxindole derivatives, which often have very wide biological applications as antimicrobial, antitumoral, antibiotic agents, and inhibitors of human NK-1 receptor [6,7].

The incorporation of oxadiazole, spirooxindole, and thiazolo[3,2-*a*]pyrimidin-3-one into a heterocyclic framework, which we believe could be a useful framework with potential biological activities, has not been investigated yet. In this work, we wish to report the synthesis of dispiro[indole-3,2′-pyrrolidine-3′,10″-[1,2,4]oxadiazolo[4,5-*a*][1,3]thiazolo[2,3-*b*]pyrimidine] **2** by one-pot sequential azomethine ylide (generated from isatin and sarcosine)–nitrile oxide cycloaddition to 2-arylmethylidene-6,7-dihydro-5*H*-thiazolo[3,2-*a*]pyrimidin-3-ones **1** (Scheme 1).

RESULTS AND DISCUSSION

The structures of products **2a–2h** were confirmed by different spectroscopic techniques (MS, IR, and NMR) and elemental analysis together with X-ray. The IR spectrum of **2c** exhibited two carbonyl peaks locating at 1698.2 and 1710.5 cm^{-1}, which was assigned to the carbonyl group in thiazole ring and the carbonyl group of indole ring, respectively. Further, the mass spectrum of **2c** showed a molecular ion peak at *m/z* 674 ($[M+H]^+$), which indicates the existence of **2c**. The ^1H NMR spectrum of **2c** revealed a singlet at 2.03 resulting from N—CH_3, several multiplets at δ 1.87–1.91, 2.82–2.91, and 4.09–4.14 and a triplet at δ 3.59 (J = 8.5 Hz) resulting from pyrrolidine ring and pyrimidine ring for the six protons of three methylenes. One doublet of doublets at δ 5.24 assignable to the CH in pyrrole ring, several multiplets in the range of δ 6.86–7.37 and a doublet at δ 7.96 (J = 8.5 Hz) for aromatic protons. The

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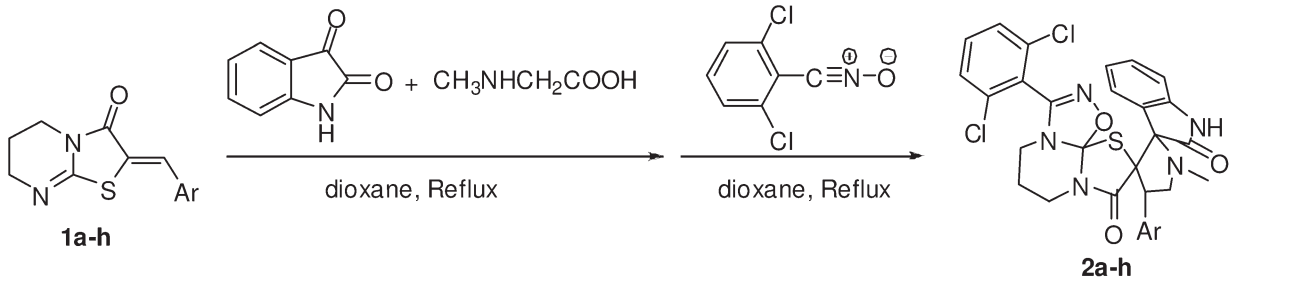
Scheme 1



Ar: **a**, C_6H_5; **b**, 4-ClC_6H_4; **c**, 2,4-$Cl_2C_6H_3$; **d**, 4-FC_6H_4; **e**, 2-$NO_2C_6H_4$; **f**, 4-$NO_2C_6H_4$; **g**, 4-$CH_3C_6H_4$; **h**, 4-$CH_3SC_6H_4$

existence of a singlet downfield at δ 7.69 corresponds to NH in indole ring.

The ^{13}C NMR spectrum of the product **2c** exhibits the presence of three methylenes carbon a*t* δ 22.86, 33.74, and 36.02, N—CH_3 carbons at δ 35.98, two carbonyl carbons at δ 170.63 and 177.53, and C=N carbon at δ 151.65 (based on HMQC). The existence of signal at δ 70.08 and 80.80 is assignable for the two spiro carbons.

Further, the structure of the product was confirmed by X-ray diffraction analysis of **2a** [8] (Fig. 1).

EXPERIMENTAL

1 [9] and 2,6-dichlorobenzonitrile oxide [10] were prepared according to the reported procedures. All NMR spectra were recorded on a Bruker AV-II 500 MHz NMR spectrometer, operating at 500 MHz for ^1H and 125 MHz for ^{13}C. TMS was used as an internal reference for ^1H and ^{13}C chemical shifts, and $CDCl_3$ was used as a solvent. Elemental analysis was measured by an Elemental analyzer (varioEL II). MS was conducted by a Finnigan LCQ Advantage MAX mass spectrometer. IR spectra were recorded on a Perkin-Elmer spectrometer (Spectrum One). Melting points were measured by a Yanaco MP500 melting point apparatus and uncorrected.

General procedure for the synthesis of 3″-(2,6-dichlorophenyl)-1′-methyl-4′-aryl-6″,7″-dihydro-5″H-dispiro[indole-3,2′-pyrrolidine-3′,10″-[1,2,4]oxadiazolo[4,5-*a*][1,3]thiazolo[2,3-*b*]pyrimidine]-2,9″(1*H*)-dione. A solution of isatin (1 mmol), sarcosine (1 mmol), and 2-arylmethylidene-6,7-dihydro-5H-thiazolo[3,2-*a*]pyrimidin-3-ones **1** (1 mmol) in dioxane (30 mL) was refluxed for 24 h, after that 2,6-dichlorobenzonitrile oxide (1.5 mmol) was added, and the resulting solution was further refluxed for 24 h; the solvent was removed *in vacuo*. The residue was subjected to column chromatography using petroleum ether–ethyl acetate (v/v 5:1) as eluent to afford the corresponding **2**.

3″-(2,6-Dichlorophenyl)-1′-methyl-4′-phenyl-6″,7″-dihydro-5″H-dispiro[indole-3,2′-pyrrolidine-3′,10″-[1,2,4]oxadiazolo[4,5-*a*][1,3]thiazolo[2,3-*b*]pyrimidine]-2,9″(1*H*)-dione (2a). Yield 55%. m.p. 202–204°C; ^1H NMR ($CDCl_3$, 500 MHz) δ: 1.83–1.87 (m, 2H), 2.29 (s, 3H, —NCH_3), 2.51–2.58 (m, 1H), 2.74–2.85 (m, 2H), 3.60 (t, J = 8.5 Hz, 1H), 4.04–4.14 (m, 2H), 4.77 (t, J = 8.5 Hz, 1H), 6.91 (d, J = 7.5 Hz, 1H), 7.00 (t, J = 7.5 Hz, 1H), 7.27–7.38 (m, 8H), 7.52 (d, J = 8.0 Hz, 2H), 8.61 (br, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ: 22.67 (C-2), 33.78 (C-3), 35.82 (C-9), 35.97 (C-1), 50.94 (C-7), 59.01 (C-8), 67.08 (C-5), 81.08 (C-10), 110.26 (C-4), 115.82, 122.60, 122.73, 122.82, 124.82, 125.73, 128.37, 128.45, 128.62, 130.34, 130.38, 130.52, 132.58, 135.82, 136.73, 139.11, 142.93, 151.46 (C-13), 171.75 (C-6), 177.69 (C-11); IR (KBr) ν: 1718.2, 1700.1 cm^{-1}; ESI MS *m/z*: 606 $[M+H]^+$. Anal. calcd for $C_{30}H_{25}Cl_2N_5O_3S$: C, 59.41; H, 4.15; N, 11.55; found C, 59.70; H, 4.11; N, 11.60.

3″-(2,6-Dichlorophenyl)-1′-methyl-4′-(4-chlorophenyl)-6″,7″-dihydro-5″H-dispiro[indole-3,2′-pyrrolidine-3′,10″-[1,2,4]oxadiazolo[4,5-*a*][1,3]thiazolo[2,3-*b*]pyrimidine]-2,9″(1*H*)-dione (2b). Yield 48%. m.p. 249–251°C; ^1H NMR (DMSO-d_6, 500 MHz) δ: 1.74–1.80 (m, 2H), 2.09 (s, 3H), 2.35–2.41 (m, 1H), 2.64–2.70 (m, 1H), 2.91–2.95 (m, 1H), 3.40–3.42 (m, 1H), 3.81 (t, J = 9.0 Hz, 1H), 3.93 (dd, J = 8.5, 13.5 Hz, 1H), 4.57 (t, J = 8.5 Hz, 1H), 6.85 (d, J = 7.5 Hz, 1H), 6.96 (t, J = 7.5 Hz, 1H), 7.10 (d, J = 7.5 Hz, 1H), 7.28 (t, J = 7.5 Hz, 1H), 7.41–7.43 (m, 2H), 7.46–7.47 (m, 2H), 7.59–7.65 (m, 3H), 10.68 (s, 1H); ^{13}C NMR (d_6-DMSO, 125 MHz) δ: 22.45 (C-2), 33.92 (C-3), 35.06 (C-9), 35.49 (C-1), 49.85 (C-7), 57.92 (C-8), 70.73 (C-5), 79.89 (C-10), 109.76 (C-4), 115.28, 121.71, 124.53, 125.26, 128.46, 128.99, 130.12, 131.85, 131.96, 133.94, 135.02, 135.16, 138.12, 143.97, 151.26 (C-13), 170.74 (C-6), 176.66 (C-11); IR (KBr) ν: 1717.4, 1685.7 cm^{-1}; ESI MS *m/z*: 640 $[M+H]^+$. Anal. calcd for $C_{30}H_{24}Cl_3N_5O_3S$: C, 56.22; H, 3.77; N, 10.93; found C, 56.33; H, 3.69; N, 11.01.

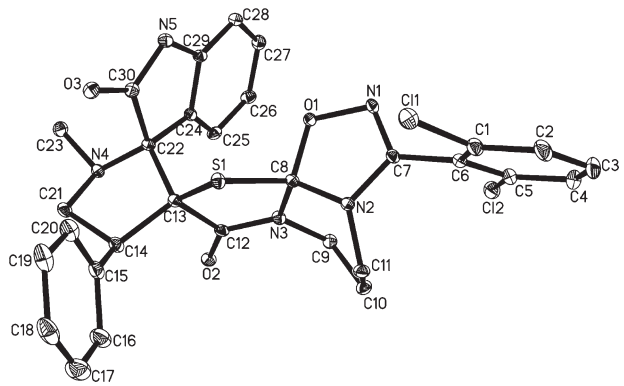


Figure 1. ORTEP diagram of **2a** (H atoms have been omitted for clarity).

3″-(2,6-Dichlorophenyl)-1′-methyl-4′-(2,4-dichlorophenyl)-6″,7″-dihydro-5″H-dispiro[indole-3,2′-pyrrolidine-3′,10″-[1,2,4]oxadiazolo[4,5-*a*][1,3]thiazolo[2,3-*b*]pyrimidine]-2,9″(1*H*)-dione (2c). Yield 50%. m.p. 245–246°C; ^{1}H NMR ($CDCl_3$, 500 MHz) δ: 1.87–1.91 (m, 2H, H-2), 2.30 (s, 3H, —NCH_3), 2.82–2.91 (m, 3H, H-1, H-3), 3.59 (t, J = 8.5 Hz, 1H, H-8), 4.09–4.14 (m, 2H, H-3, H-8), 5.24 (dd, J = 8.5, 9.5 Hz, 1H, H-7), 6.86–6.87 (m, 1H), 7.01–7.04 (m, 1H), 7.28–7.31 (m, 2H), 7.32–7.37 (m, 5H), 7.69 (s, 1H, H-11), 7.96 (d, J = 8.5 Hz, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ: 22.86 (C-2), 33.74 (C-3), 35.98 (C-9), 36.02 (C-1), 45.04 (C-7), 57.14 (C-8), 70.08 (C-5), 80.80 (C-10), 110.34 (C-4), 116.03, 122.92, 124.08, 126.04, 127.70, 128.34, 128.62, 128.95, 130.56, 131.96, 132.60, 133.54, 135.54, 135.89, 136.76, 136.85, 142.44, 151.65 (C-13), 170.63 (C-6), 177.53 (C-11); IR (KBr) ν: 1710.5, 1698.2 cm^{-1}; ESI MS *m/z*: 674 $[M+H]^+$. Anal. calcd for $C_{30}H_{23}Cl_4N_5O_3S$: C, 53.35; H, 3.43; N, 10.37; found C, 53.05; H, 3.39; N, 10.26.

3″-(2,6-Dichlorophenyl)-1′-methyl-4′-(4-fluorophenyl)-6″,7″-dihydro-5″H-dispiro[indole-3,2′-pyrrolidine-3′,10″-[1,2,4]oxadiazolo[4,5-*a*][1,3]thiazolo[2,3-*b*]pyrimidine]-2,9″(1*H*)-dione (2d). Yield 55%. m.p. 190–192°C; ^{1}H NMR ($CDCl_3$, 500 MHz) δ: 1.82–1.91 (m, 2H), 2.29 (s, 3H, H-9), 2.51–2.58 (m, 1H), 2.77–2.84 (m, 2H), 3.60 (dd, J = 8.0, 9.0 Hz, 1H), 4.00–4.07 (m, 2H), 4.73 (dd, J = 8.0, 10.0 Hz, 1H, H-7), 6.88 (d, J = 7.5 Hz, 1H), 7.00–7.05 (m, 3H), 7.28–7.35 (m, 5H), 7.46–7.49 (m, 2H), 7.78 (s, 1H, H-12); ^{13}C NMR ($CDCl_3$, 125 MHz) δ: 22.93, 33.68, 35.93, 35.99, 50.17, 59.15, 71.29, 80.97, 110.11, 115.51, 122.68, 124.60, 125.68, 128.31, 128.59, 130.41, 131.77, 131.84, 132.57, 134.80, 135.77, 136.72, 142.57, 151.46, 171.53, 177.40; IR (KBr) ν: 1720.5, 1689.8 cm^{-1}; ESI MS *m/z*: 624 $[M+H]^+$. Anal. calcd for $C_{30}H_{24}Cl_2FN_5O_3S$: C, 57.70; H, 3.87; N, 11.21; found C, 57.45; H, 3.91; N, 11.07.

3″-(2,6-Dichlorophenyl)-1′-methyl-4′-(2-nitrophenyl)-6″,7″-dihydro-5″H-dispiro[indole-3,2′-pyrrolidine-3′,10″-[1,2,4]oxadiazolo[4,5-*a*][1,3]thiazolo[2,3-*b*]pyrimidine]-2,9″(1*H*)-dione (2e). Yield 52%. m.p. 220–222°C; ^{1}H NMR ($CDCl_3$, 500 MHz) δ: 1.85–1.93 (m, 1H), 2.05–2.10 (m, 1H), 2.29 (s, 3H), 2.75–2.89 (m, 3H), 3.59 (t, J = 8.5 Hz, 1H), 4.11 (t, J = 9.0 Hz, 1H), 4.17 (dd, J = 8.5, 13.5 Hz, 1H), 4.92 (t, J = 9.0 Hz, 1H), 6.88 (d, J = 7.5 Hz, 1H), 7.04 (t, J = 7.5 Hz, 1H), 7.29–7.37 (m, 5H), 7.42 (t, J = 7.5 Hz, 1H), 7.59 (s, 1H), 7.68 (t, J = 7.5 Hz, 1H), 7.73 (d, J = 7.5 Hz, 1H), 8.41 (d, J = 8.0 Hz, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ: 22.86 (C-2), 33.82 (C-3), 35.83 (C-9), 36.07 (C-1), 44.52 (C-7), 58.89 (C-8), 70.58 (C-5), 80.75 (C-10), 109.98 (C-4), 116.44, 122.93, 122.98, 123.68, 124.16, 126.37, 128.21, 128.38, 128.50, 130.54, 132.32, 132.52, 133.10, 133.54, 135.95, 136.67, 142.33, 151.67 (C-13) 170.05 (C-6), 177.30 (C-11); IR (KBr) ν: 1709.7, 1693.6 cm^{-1}; ESI MS *m/z*: 651 $[M+H]^+$. Anal. calcd for $C_{30}H_{24}Cl_2N_6O_5S$: C, 55.30; H, 3.71; N, 12.90; found C, 55.00; H, 3.83; N, 12.78.

3″-(2,6-Dichlorophenyl)-1′-methyl-4′-(4-nitrophenyl)-6″,7″-dihydro-5″H-dispiro[indole-3,2′-pyrrolidine-3′,10″-[1,2,4]oxadiazolo[4,5-*a*][1,3]thiazolo[2,3-*b*]pyrimidine]-2,9″(1*H*)-dione (2f). Yield 55%. m.p. 226–228°C; ^{1}H NMR ($CDCl_3$, 500 MHz) δ: 1.79–1.96 (m, 2H), 2.30 (s, 3H), 2.49–2.55 (m, 1H), 2.79–2.85 (m, 2H), 3.64 (t, J = 8.0 Hz, 1H), 4.05–4.09 (m, 2H), 4.85 (dd, J = 8.0, 9.5 Hz, 1H), 6.91 (d, J = 7.5 Hz, 1H), 7.03 (t, J = 7.5 Hz, 1H), 7.29–7.31 (m, 1H), 7.32–7.36 (m, 4H), 7.71 (t, J = 8.5 Hz, 2H), 7.78 (br, 1H), 8.20 (d, J = 8.5 Hz, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ: 22.91 (C-2), 33.84 (C-3), 35.81 (C-9), 36.05, 50.48, 58.99, 70.56, 80.93 (C-10), 110.18 (C-4), 115.79, 122.64, 122.89, 123.87, 124.26, 125.76, 128.39, 128.68, 130.68, 131.31, 132.70, 135.81, 136.78, 142.48, 146.79, 147.33, 151.57 (C-13), 171.16 (C-6), 176.98 (C-11); IR (KBr) ν: 1711.6, 1699.9 cm^{-1}; ESI MS *m/z*: 651 $[M+H]^+$. Anal. calcd for $C_{30}H_{24}Cl_2N_6O_5S$: C, 55.30; H, 3.71; N, 12.90; found C, 55.39; H, 3.94; N, 12.80.

3″-(2,6-Dichlorophenyl)-1′-methyl-4′-(4-methylphenyl)-6″,7″-dihydro-5″H-dispiro[indole-3,2′-pyrrolidine-3′,10″-[1,2,4]oxadiazolo[4,5-*a*][1,3]thiazolo[2,3-*b*]pyrimidine]-2,9″(1*H*)-dione (2g). Yield 50%. m.p. 200–202°C; ^{1}H NMR ($CDCl_3$, 500 MHz) δ: 1.82–1.88 (m, 2H), 2.29 (s, 3H), 2.35 (s, 3H, —CH_3), 2.55–2.61 (m, 1H), 2.77–2.84 (m, 2H), 3.57 (t, J = 8.5 Hz, 1H), 4.02–4.06 (m, 2H), 4.71 (t, J = 9.0 Hz, 1H), 6.87 (d, J = 7.5 Hz, 1H), 7.01 (t, J = 7.5 Hz, 1H), 7.15 (t, J = 7.5 Hz, 2H), 7.27–7.28 (m, 2H), 7.32–7.35 (m, 3H), 7.38 (d, J = 7.5 Hz, 2H), 7.59 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ: 21.20, 23.04, 33.68, 35.85, 36.03, 50.46, 59.12, 71.47, 80.97, 109.89, 115.79, 122.96, 124.86, 125.84, 128.36, 128.58, 129.34, 130.12, 130.32, 132.51, 135.87, 135.94, 136.80, 136.99, 142.53, 151.49, 171.70, 176.96; IR (KBr) ν: 1719.9, 1697.9 cm^{-1}; ESI MS *m/z*: 620 $[M+H]^+$. Anal. calcd for $C_{31}H_{27}Cl_2N_5O_3S$: C, 60.00; H, 4.39; N, 11.29; found C, 59.85; H, 4.58; N, 11.24.

3″-(2,6-Dichlorophenyl)-1′-methyl-4′-(4-methylthiophenyl)-6″,7″-dihydro-5″H-dispiro[indole-3,2′-pyrrolidine-3′,10″-[1,2,4]oxadiazolo[4,5-*a*][1,3]thiazolo[2,3-*b*]pyrimidine]-2,9″(1*H*)-dione (2h). Yield 51%. m.p. 200–203°C; ^{1}H NMR ($CDCl_3$, 500 MHz) δ: 1.80–1.91 (m, 2H), 2.28 (s, 3H), 2.49 (s, 3H), 2.55–2.59 (m, 1H), 2.78–2.84 (m, 2H), 3.58 (dd, J = 8.0, 9.0 Hz, 1H), 4.00–4.07 (m, 2H), 4.70 (dd, J = 8.0, 10.0 Hz, 1H), 6.88 (d, J = 7.5 Hz, 1H), 7.00 (t, J = 7.5 Hz, 1H), 7.22 (d, J = 8.5 Hz, 2H), 7.27–7.29 (m, 2H), 7.31–7.35 (m, 3H), 7.44 (d, J = 8.0 Hz, 2H), 7.97 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ: 15.66 (—SCH_3), 23.04, 33.73, 35.91, 36.04, 50.38, 59.08, 71.34, 81.03, 110.21, 115.81, 122.64, 122.82, 124.74, 125.70, 126.48, 128.35, 128.63, 130.41, 130.78, 132.58, 135.82, 135.92, 136.80, 137.34, 142.79, 151.50, 171.69, 177.51; IR (KBr) ν: 1705.8, 1689.3 cm^{-1}; ESI MS *m/z*: 652 $[M+H]^+$. Anal. calcd for $C_{31}H_{27}Cl_2N_5O_3S_2$: C, 57.05; H, 4.17; N, 10.73; found C, 57.27; H, 4.26; N, 10.59.

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