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The Regiospecific *N*-Sulfonylation and N-Phosphorylation of Benzoyl-Substituted Heterocyclic Ketene Aminals

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ABSTRACT: *The regiospecific N-sulfonylation and N-phosphorylation of benzoyl-substituted heterocyclic ketene aminals have been investigated. In the presence of sodium hydride, benzoyl-substituted heterocyclic ketene aminals* **1** *or* **2** *reacted with p-toluenesulfonyl chloride* **3** *to give (E)-1-(p-toluenesulfonyl)-2-(aroylmethylene)imidazolidine* **4** *or (E)-1-(p-toluenesulfonyl)-2-(aroylmethylene)hexahydropyrimidine* **5**, *respectively. Under the same condition,* **1** *reacted with diethyl chlorothiophosphate* **6** *to give diethyl [2-(aroylmethylene)imidazolidin-1-yl]thiophosphate* **7**. *However,* **2** *failed to react with* **6** *to give N-phosphorylated products.* © 1999 John Wiley & Sons, Inc. Heteroatom Chem 10: 297–301, 1999

INTRODUCTION

As important intermediates, heterocyclic ketene aminals have shown their great potential for the synthesis of a wide variety of new heterocycles and fused heterocycles. Thus, the synthesis and reactions of heterocyclic ketene aminals have attracted much attention [1–21]. Due to the effect of conjugation of the two electron-donating amino groups and of the electron-withdrawing groups, the double bond is highly polarized. Therefore, the α-carbon of heterocyclic ketene aminals possesses a relatively high electron density and is always the point of attack on the electropositive site of electrophiles [2–16]. However, in the presence of a strong base such as sodium hydride, the nitrogen atom can take part in the nucleophilic reaction rather than the α-carbon [17–21]. Recently, *O*-glucosides were obtained when the glycosylation of benzoyl-substituted heterocyclic ketene aminals was carried out [22,23]. Thus, in the case of benzoyl-substituted heterocyclic ketene aminals, the electrophiles may attack in three different ways: *C*, *N*, and *O* attack to afford different products.

Although the *N*-alkylation or *N*-acylation of heterocyclic ketene aminals can easily take place when a strong base such as sodium hydride is used, the *N*-sulfonylation and *N*-phosphorylation of these reagents have not been reported. Continuing our research on the *N*-substitution of benzoyl-substituted heterocyclic ketene aminals, **1** or **2** was reacted with *p*-toluenesulfonyl chloride **3** or diethyl chlorothiophosphate **6** in the presence of sodium hydride.

RESULTS AND DISCUSSION

Benzoyl-substituted heterocyclic ketene aminals **1** or **2** reacted with *p*-toluenesulfonyl chloride **3** in the presence of sodium hydride and anhydrous dimethylformamide (DMF) to give (*E*)-1-(*p*-toluenesulfonyl)-2-(aroylmethylene)imidazolidine **4** or (*E*)-1-(*p*-toluenesulfonyl)-2-(aroylmethylene)hexahydropyrimidine **5**, respectively (Scheme 1).

The constitution of **4** or **5**, determined by microanalyses and mass spectra, showed that the sulfonylated products are formed in a 1:1 molar ratio of **1** or **2** with **3** accompanied with the loss of 1 mol

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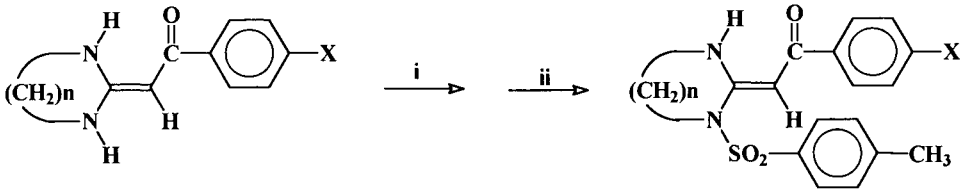


1 n=2

2 n=3

4 n=2

5 n=3

1-2, 4-5	a	b	c	d
X	H	CH_3	OCH_3	Cl

SCHEME 1 Reagents and conditions: i, NaH, DMF, r.t.; ii, *p*-$CH_3C_6H_4SO_2Cl$, C_6H_6, 0°C.

of hydrogen chloride. The sulfonyl group absorption at ca. 1360 and 1170 cm^{-1} in the IR spectra of **4** or **5** indicated that sulfonylation occurred in the reaction. As a polyfunctionalized molecule, benzoyl-substituted heterocyclic ketene aminals may take part in the reaction through *C*, *N*, and *O* attack to give different products. However, the presence of one nitrogen proton signal (δ = 10.15–12.33) and one ethylenic proton signal (δ = 6.33–6.52) in the ^{1}H NMR spectra of **4** or **5** excludes *C*-sulfonylated products, and the presence of a carbonyl carbon signal (δ = 184.2–188.1) in the ^{13}C NMR spectra also excludes *O*-sulfonylated products. Therefore, the sulfonylation can only have occurred on the nitrogen atom to afford mono-*N*-sulfonylated products. The *E*-configuration of **4** or **5** was determined by the downfield shift of the nitrogen proton in the ^{1}H NMR, which is due to the intramolecular hydrogen bond.

In the presence of sodium hydride and anhydrous dimethylformamide (DMF), benzoyl-substituted heterocyclic ketene aminals **1** reacted with diethyl chlorothiophosphate **6** to give diethyl [2-(aroylmethylene)imidazolidin-1-yl] thiophosphate **7** (Scheme 2).

Microanalytical data and mass spectropic data showed that compounds **7** were formed in a 1:1 molar ratio of **1** with **6** accompanied with the loss of 1 mol of hydrogen chloride. The possibilities of *C*-phosphorylated and *O*-phosphorylated products were excluded by the presence of one ethylenic proton signal (δ = 6.01–6.03) in the ^{1}H NMR spectra and a carbonyl carbon signal (δ = 185.5–186.9) in the ^{13}C NMR spectra. Thus, the products **7** can only be *N*-phosphorylated products. The *E*-configuration of **7** was determined by the downfield shift of the nitrogen proton (δ = 10.30–10.39) in the ^{1}H NMR spectra.

Unexpectedly, benzoyl-substituted heterocyclic ketene aminals **2** failed to react with **6** in the presence of sodium hydride and DMF to afford *N*-phosphorylated products. The reaction was complicated, and the products could not be separated easily.

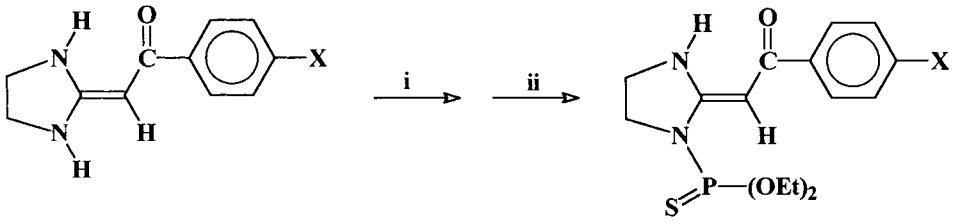
The mechanism of the *N*-sulfonylation and *N*-phosphorylation reaction, like those of the *N*-alkylation and *N*-acylation reactions, may be due to the formation of the nitrogen anion during the reaction of **1** or **2** in the presence of sodium hydride, which then leads to the formation of *N*-sulfonylated and *N*-phosphorylated products with **3** or **6**.

EXPERIMENTAL

Melting points are uncorrected. ^{1}H and ^{13}C NMR spectra were recorded with a Varian Unity 200 spectrometer. IR spectra were recorded with a Perkin-Elmer 782 spectrometer. Mass spectra were recorded on an AEI MS-50 instrument. Elemental analyses were performed at the Analytical Laboratory of the Institute.

General Procedure for the Preparation of **4–5**

NaH (4 mmol) was added to an ice-cooled solution of **1** or **2** (4 mmol) in 15 mL of anhydrous DMF under stirring. After 20 minutes, a solution of 4 mmol of *p*-toluenesulfonyl chloride **3** in 5 mL of benzene was added dropwise. After having been stirred for 1 hour, the mixture was poured into 100 mL of water and extracted with $CHCl_3$ (3 × 50 mL). The combined



1 **7**

1, 7	a	b	c	d
X	H	CH_3	OCH_3	Cl

SCHEME 2 Reagents and conditions: i, NaH, DMF, r.t.; ii, $(EtO)_2PSCl$, C_6H_6, 0°C.

extracts were dried with anhydrous $CaCl_2$, and the solvent was evaporated. The residue was subjected to column chromatography on silica gel (200–300 mesh) using $CHCl_3$ as eluant to afford the pure compound **4** or **5**, which was recrystallized from EtOH to give white crystals.

(*E*)-*1*-(*p-Toluenesulfonyl*)-*2*-(*benzoylmethylene*)*imidazolidine*

4a: Yield 64%; mp 174–176°C; IR (KBr): *v* 3440 (NH), 1615 (CO), 1354 (SO_2), 1160 (SO_2) cm^{-1}; ^1H NMR ($CDCl_3$): δ 10.23 (s, 1H, NH), 7.98–7.88 (m, 2H, Ar-H), 7.82 (d, 2H, Ar-H), 7.50–7.40 (m, 3H, Ar-H), 7.32 (d, 2H, Ar-H), 6.52 (s, 1H, C=C-H), 3.88 (t, 2H, N-CH_2), 3.60 (t, 2H, N-CH_2), 2.41 (s, 3H, CH_3); ^{13}C NMR ($CDCl_3$): δ 188.1, 158.4, 145.4, 139.9, 133.3, 130.9, 130.0, 128.2, 127.5, 127.0, 77.8, 46.6, 41.4, 21.6; MS: m/z 342 [M^+] (5), 277 (24), 250 (58), 105 (100). Anal. calcd for $C_{18}H_{18}N_2O_3S$: C, 63.14; H, 5.30; N, 8.18. Found: C, 62.80; H, 5.43; N, 8.25.

(*E*)-*1*-(*p-Toluenesulfonyl*)-*2*-(*4-methylbenzoylmethylene*)*imidazolidine*

4b: Yield 59%; mp 188–190°C; IR (KBr): *v* 3430 (NH), 1624 (CO), 1360 (SO_2), 1168 (SO_2) cm^{-1}; ^1H NMR ($CDCl_3$): δ 10.20 (s, 1H, NH), 7.83 (d, 2H, Ar-H), 7.83 (d, 2H, Ar-H), 7.33 (d, 2H, Ar-H), 7.26 (d, 2H, Ar-H), 6.51 (s, 1H, C=C-H), 3.89 (t, 2H, N-CH_2), 3.60 (t, 2H, N-CH_2), 2.42 (s, 3H, CH_3), 2.41 (s, 3H, CH_3); ^{13}C NMR ($CDCl_3$): δ 188.1, 158.2, 145.3, 141.3, 137.2, 133.4, 130.0, 128.9, 127.5, 127.1, 77.6, 46.7, 41.4, 21.6, 21.4; MS: m/z 356 [M^+] (6), 291 (18), 264 (63), 119 (100). Anal. calcd for $C_{19}H_{20}N_2O_3S$: C, 64.02; H, 5.66; N, 7.86. Found: C, 63.91; H, 5.83; N, 7.68.

(*E*)-*1*-(*p-Toluenesulfonyl*)-*2*-(*4-methoxybenzoylmethylene*)*imidazolidine*

4c: Yield 69%; mp 202–204°C; IR (KBr): *v* 3430 (NH), 1620 (CO), 1358 (SO_2), 1165 (SO_2) cm^{-1}; ^1H NMR ($CDCl_3$): δ 10.15 (s, 1H, NH), 7.92 (d, 2H, Ar-H), 7.83 (d, 2H, Ar-H), 7.33 (d, 2H, Ar-H), 6.95 (d, 2H, Ar-H), 6.48 (s, 1H, C=C-H), 3.91 (t, 2H, N-CH_2), 3.90 (s, 3H, OCH_3), 3.60 (t, 2H, N-CH_2), 2.42 (s, 3H, CH_3); ^{13}C NMR ($CDCl_3$): δ 187.4, 161.9, 158.0, 145.3, 133.4, 132.6, 130.0, 129.0, 127.5, 113.4, 77.4, 55.3, 46.7, 41.4, 21.6; MS: m/z 372 [M^+] (6), 307 (5), 280 (42), 135 (100). Anal. calcd for $C_{19}H_{20}N_2O_4S$: C, 61.27; H, 5.41; N, 7.52. Found: C, 61.49; H, 5.45; N, 7.15.

(*E*)-*1*-(*p-Toluenesulfonyl*)-*2*-(*4-chlorobenzoylmethylene*)*imidazolidine*

4d: Yield 61%; mp 197–199°C; IR (KBr): *v* 3430 (NH), 1625 (CO), 1360 (SO_2), 1168 (SO_2) cm^{-1}; ^1H NMR ($CDCl_3$): δ 10.24 (s, 1H, NH), 7.90 (d, 2H, Ar-H), 7.82 (d, 2H, Ar-H), 7.40 (d, 2H, Ar-H), 7.33 (d, 2H, Ar-H), 6.45 (s, 1H, C=C-H), 3.92 (t, 2H, N-CH_2), 3.63 (t, 2H, N-CH_2), 2.42 (s, 3H, CH_3); ^{13}C NMR (CDCl3): δ 188.7, 158.6, 145.5, 138.3, 136.9, 133.4, 130.0, 128.4, 128.4, 127.5, 77.2, 46.7, 41.5, 21.6; MS: m/z 376 [M^+] (6), 311 (25), 284 (90), 139 (100). Anal. calcd for $C_{18}H_{17}ClN_2O_3S$: C, 57.36; H, 4.55; N, 7.44. Found: C, 57.41; H, 4.90; N, 7.43.

(*E*)-*1*-(*p-Toluenesulfonyl*)-*2*-(*benzoylmethylene*)*hexahydropyrimidine*

5a: Yield 51%; mp 159–161°C; IR (KBr): *v* 3440 (NH), 1610 (CO), 1348 (SO_2), 1160 (SO_2) cm^{-1}; ^1H NMR ($CDCl_3$): δ 12.33 (s, 1H, NH), 7.83–7.73 (m, 2H, Ar-

H), 7.77 (d, 2H, Ar-H), 7.45–7.37 (m, 3H, Ar-H), 7.29 (d, 2H, Ar-H), 6.38 (s, 1H, C=C-H), 3.99 (t, 2H, N-CH_2), 3.35 (t, 2H, N-CH_2), 2.42 (s, 3H, CH_3), 2.00 (quin, 2H, C-CH_2-C); ^{13}C NMR ($CDCl_3$): δ 185.8, 156.5, 145.0, 140.4, 135.7, 130.3, 129.9, 128.1, 127.2, 126.6, 82.2, 44.6, 38.8, 21.8, 21.5; MS: m/z 356 [M^+] (4), 291 (31), 263 (100). Anal. calcd for $C_{19}H_{20}N_2O_3S$: C, 64.02; H, 5.66; N, 7.86. Found: C, 63.91; H, 5.93; N, 7.86.

(*E*)-*1*-(*p-Toluenesulfonyl*)-*2*-(*4-methylbenzoylmethylene*)*hexahydropyrimidine*

5b: Yield 57%; mp 115–117°C; IR (KBr): ν 3440 (NH), 1610 (CO), 1350 (SO_2), 1164 (SO_2) cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 12.28 (s, 1H, NH), 7.81 (d, 2H, Ar-H), 7.72 (d, 2H, Ar-H), 7.30 (d, 2H, Ar-H), 7.21 (d, 2H, Ar-H), 6.39 (s, 1H, C=C-H), 3.99 (t, 2H, N-CH_2), 3.35 (t, 2H, N-CH_2), 2.42 (s, 3H, CH_3), 2.40 (s, 3H, CH_3), 2.00 (quin, 2H, C-CH_2-C); ^{13}C NMR ($CDCl_3$): δ 185.7, 156.3, 144.9, 140.6, 137.7, 135.7, 129.8, 128.8, 127.2, 126.7, 82.0, 44.6, 38.7, 21.8, 21.5, 21.3; MS: m/z 370 [M^+] (4), 305 (27), 277 (89), 187 (85), 119 (100). Anal. calcd for $C_{20}H_{22}N_2O_3S$: C, 64.84; H, 5.99; N, 7.56. Found: C, 64.57; H, 5.97; N, 7.58.

(*E*)-*1*-(*p-Toluenesulfonyl*)-*2*-(*4-methoxybenzoylmethylene*)*hexahydropyrimidine*

5c: Yield 53%; mp 133–135°C; IR (KBr): ν 3440 (NH), 1600 (CO), 1355 (SO_2), 1162 (SO_2) cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 12.20 (s, 1H, NH), 7.81 (d, 2H, Ar-H), 7.79 (d, 2H, Ar-H), 7.30 (d, 2H, Ar-H), 6.92 (d, 2H, Ar-H), 6.36 (s, 1H, C=C-H), 3.99 (t, 2H, N-CH_2), 3.86 (s, 3H, OCH_3), 3.33 (t, 2H, N-CH_2), 2.41 (s, 3H, CH_3), 1.99 (quin, 2H, C-CH_2-C); ^{13}C NMR ($CDCl_3$): δ 185.0, 161.3, 156.0, 144.8, 135.6, 132.9, 129.7, 128.3, 127.0, 113.2, 81.5, 55.1, 44.5, 38.6, 21.7, 21.4; MS: m/z 386 [M^+] (3), 321 (15), 293 (62), 187 (45), 135 (100). Anal. calcd for $C_{20}H_{22}N_2O_4S$: C, 62.15; H, 5.74; N, 7.25. Found: C, 61.80; H, 5.83; N, 7.33.

(*E*)-*1*-(*p-Toluenesulfonyl*)-*2*-(*4-chlorobenzoylmethylene*)*hexahydropyrimidine*

5d: Yield 60%; mp 170–172°C; IR (KBr): ν 3440 (NH), 1611 (CO), 1344 (SO_2), 1166 (SO_2) cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 12.32 (s, 1H, NH), 7.79 (d, 2H, Ar-H), 7.73 (d, 2H, Ar-H), 7.36 (d, 2H, Ar-H), 7.30 (d, 2H, Ar-H), 6.33 (s, 1H, C=C-H), 4.00 (t, 2H, N-CH_2), 3.35 (t, 2H, N-CH_2), 2.41 (s, 3H, CH_3), 2.00 (quin, 2H, C-CH_2-C); ^{13}C NMR ($CDCl_3$): δ 184.2, 156.6, 145.1, 138.9, 136.2, 135.6, 129.9, 128.3, 128.0, 127.1, 81.9, 44.6, 38.8, 21.7, 21.5; MS: m/z 390 [M^+] (2), 325 (26), 297 (87), 187 (100), 139 (52). Anal. calcd for $C_{19}H_{19}ClN_2O_3S$: C, 58.38; H, 4.90; N, 7.17. Found: C, 58.11; H, 4.94; N, 6.95.

General Procedure for the Preparation of **7**

NaH (4 mmol) was added to an ice-cooled solution of **1** (4 mmol) in 15 mL of anhydrous DMF under stirring. After 20 minutes, a solution of 4 mmol of diethyl chlorothiophosphate **6** in 5 mL of benzene was added dropwise. The reaction and workup procedure was the same as for the synthesis of **4**, and **7** was obtained as white crystals.

Diethyl [2-(Benzoylmethylene)imidazolidin-1-yl]thiophosphate

7a: Yield 70%; mp 74–76°C; IR (KBr): ν 3280 (NH), 1600 (CO), 1010 (PO), 760 (P=S), 655 (P=S) cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 10.39 (s, 1H, NH), 7.99–7.33 (m, 5H, Ar-H), 6.03 (s, 1H, C=C-H), 4.19 (q, 4H, CH_2), 3.99 (t, 2H, N-CH_2), 3.68 (t, 2H, N-CH_2), 1.36 (t, 6H, CH_3); ^{13}C NMR ($CDCl_3$): δ 186.4, 160.0 (29.2), 140.4, 130.1, 127.9, 127.1, 78.1, 63.8 (19.0), 48.6 (23.4), 42.1 (32.2), 15.7 (33.6) ppm (Hz); ^{31}P NMR ($CDCl_3$): δ 68.8; MS: m/z 340 [M^+] (65), 312 (51), 263 (48), 187 (62), 105 (100). Anal. calcd for $C_{15}H_{21}N_2O_3PS$: C, 52.93; H, 6.22; N, 8.23. Found: C, 52.36; H, 6.46; N, 8.31.

Diethyl [2-(4-Methylbenzoylmethylene)imidazolidin-1-yl]thiophosphate

7b: Yield 62%; mp 115–117°C; IR (KBr): ν 3220 (NH), 1605 (CO), 1010 (PO), 755 (P=S), 650 (P=S) cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 10.36 (s, 1H, NH), 7.76 (d, 2H, Ar-H), 7.20 (d, 2H, Ar-H), 6.02 (s, 1H, C=C-H), 4.18 (q, 4H, CH_2), 3.94 (t, 2H, N-CH_2), 3.66 (t, 2H, N-CH_2), 1.99 (s, 3H, CH_3), 1.37 (t, 6H, CH_3); ^{13}C NMR ($CDCl_3$): δ 186.9, 160.2 (27.8), 140.5, 137.7, 128.7, 126.7, 77.5, 63.8 (19.0), 48.8 (24.6), 42.0 (32.0), 21.3, 15.7 (32.0) ppm (Hz); ^{31}P NMR ($CDCl_3$): δ 63.9; MS: m/z 354 [M^+] (66), 326 (32), 277 (34), 201 (51), 119 (100). Anal. calcd for $C_{16}H_{23}N_2O_3PS$: C, 54.22; H, 6.54; N, 7.89. Found: C, 54.28; H, 6.57; N, 7.84.

Diethyl [2-(4-Methoxybenzoylmethylene)imidazolidin-1-yl]thiophosphate

7c: Yield 50%; mp 61–63°C; IR (KBr): ν 3280 (NH), 1600 (CO), 1015 (PO), 770 (P=S), 650 (P=S) cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 10.30 (s, 1H, NH), 7.82 (d, 2H,

Ar-H), 6.91 (d, 2H, Ar-H), 6.03 (s, 1H, C=C-H), 4.19 (q, 4H, CH_2), 3.99 (t, 2H, N-CH_2), 3.85 (s, 3H, OCH_3), 3.66 (t, 2H, N-CH_2), 1.37 (t, 6H, CH_3); ^{13}C NMR ($CDCl_3$): δ 186.2, 161.3 (27.8), 159.8, 132.9, 128.3, 113.1, 77.0, 63.6 (20.4), 55.0, 48.6 (24.8), 41.8 (32.0), 15.5 (32.0) ppm (Hz); ^{31}P NMR ($CDCl_3$): δ 62.2; MS: m/z 370 [M^+] (52), 325 (26), 293 (20), 217 (37), 135 (100). Anal. calcd for $C_{16}H_{23}N_2O_4PS$: C, 51.88; H, 6.26; N, 7.56. Found: C, 51.77; H, 6.39; N, 7.66.

Diethyl [2-(4-Chlorobenzoylmethylene)imidazolidin-1-yl]thiophosphate

7d: Yield 65%; mp 89–91°C; IR (KBr): ν 3240 (NH), 1600 (CO), 1010 (PO), 760 (P=S), 655 (P=S) cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 10.38 (s, 1H, NH), 7.80 (d, 2H, Ar-H), 7.39 (d, 2H, Ar-H), 6.01 (s, 1H, C=C-H), 4.19 (q, 4H, CH_2), 4.00 (t, 2H, N-CH_2), 3.72 (t, 2H, N-CH_2), 1.37 (t, 6H, CH_3); ^{13}C NMR ($CDCl_3$): δ 185.7, 160.8 (29.0), 139.0, 136.4, 128.3, 128.2, 77.6, 64.0 (19.0), 48.8 (24.8), 42.1 (32.0), 15.8 (32.0) ppm (Hz); ^{31}P NMR ($CDCl_3$): δ 62.2; MS: m/z 374 [M^+] (68), 346 (37), 297 (50), 221 (55), 139 (100). Anal. calcd for $C_{15}H_{20}ClN_2O_3PS$: C, 48.06; H, 5.38; N, 7.48. Found: C, 48.00; H, 5.37; N, 7.53.

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