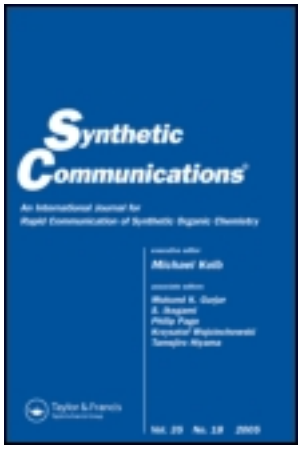

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Synthesis of Ketone from Alkyl Halide Using α-Chloro Nitrone as Oxidizing Reagent: A New Approach

Bhaskar Chakraborty [a] & Prawin Kumar Sharma [a]

[a] Organic Chemistry Laboratory, Sikkim Government College, Gangtok, Sikkim, India

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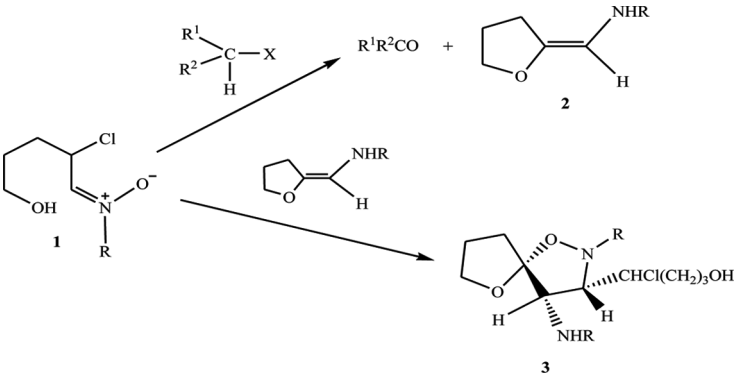
SYNTHESIS OF KETONE FROM ALKYL HALIDE USING α-CHLORO NITRONE AS OXIDIZING REAGENT: A NEW APPROACH

Bhaskar Chakraborty and Prawin Kumar Sharma
Organic Chemistry Laboratory, Sikkim Government College, Gangtok, Sikkim, India

GRAPHICAL ABSTRACT



Abstract *Consecutive S_N2 reaction of α-chloronitrones were studied with isopropyl halides, and the nitrones have remarkable oxidizing properties for the conversion of isopropyl halides to ketones with good yields. In addition, the side product obtained can serve as efficient dipolarophile in 1,3-dipolar cycloaddition reaction to produce spiro cycloadduct in good yield.*

Keywords α-Chloronitrone; ketone synthesis; reusable side product

INTRODUCTION

Conventional methods for the synthesis of ketone have been well known for a long time, including oxidation of secondary alcohols, hydration of alkynes, Friedel–Crafts acylation, and the use of Grignard reagents. Although plenty of modern methods are available for the synthesis of ketone from secondary alcohol,[1–11] no such methodologies are available for ketone synthesis from isopropyl halides using

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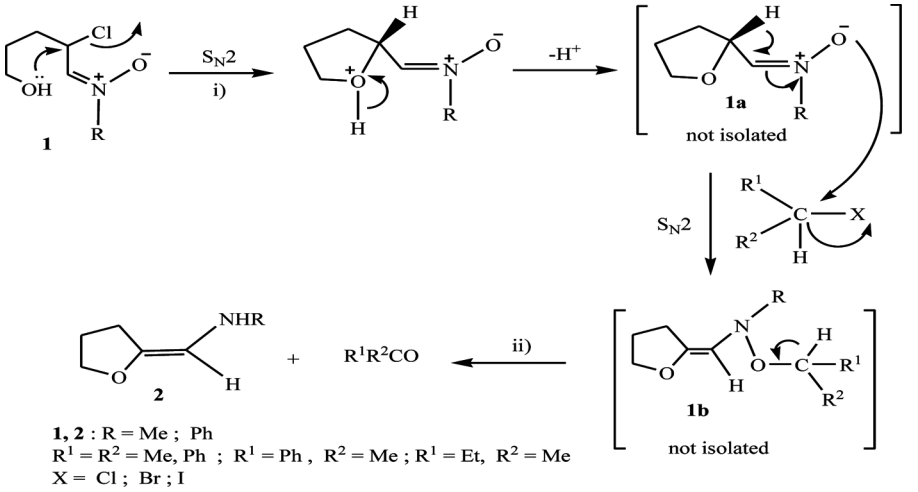
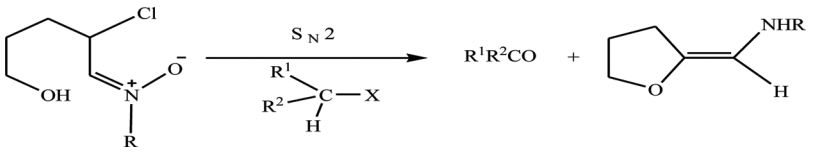
Address correspondence to Bhaskar Chakraborty, Organic Chemistry Laboratory, Sikkim Government College, Gangtok, Sikkim 737-102, India. E-mail: bchakraborty42@gmail.com



Scheme 1. Regents and conditions: (i) dry ether, pyridine, rt, N_2 atmosphere; and (ii) dry ether, Na_2CO_3, rt, N_2 atmosphere.

nitrones. In this communication, we report an efficient one-pot synthesis of ketone from isopropyl halides using α-chloronitrones **1**[12,13] as oxidizing reagent (Scheme 1, Table 1). In addition, the side product (furan derivative, **2**) obtained during ketone synthesis has been successfully used as dipolarophile in 1,3-dipolar

Table 1. Ketone synthesis using α-chloronitrones

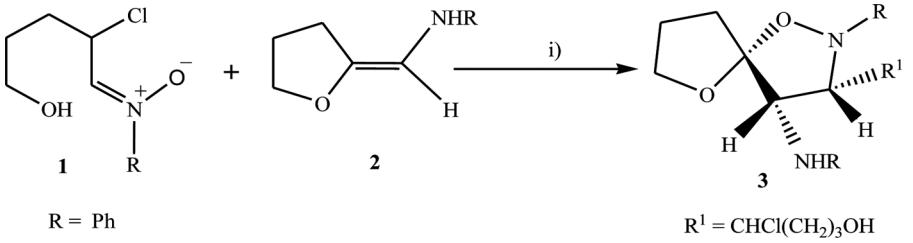


Entry	Nitrone	Alkyl halide[a]	Product[b]	Time (h)	Yield[c] (%)
1	R = Me	Isopropyl bromide	Acetone	5	79
2	R = Me	Isopropyl chloride	Acetone	6	78
3	R = Me	Isopropyl iodide	Acetone	6	77
4	R = Ph	Isopropyl bromide	Acetone	5	77
5	R = Ph	Isopropyl chloride	Acetone	6	76
6	R = Ph	Isopropyl iodide	Acetone	6	75
7	R = Me	1-Bromo ethyl benzene	Acetophenone	7	81
8	R = Ph	2-Bromo butane	Ethyl methyl ketone	6	75
9	R = Me	Bromo diphenyl methane	Benzophenone	7	83

[a]Reaction conditions: α-chloronitrone (3.0198 mmol), isopropyl halide (1 equivalent), dry ether, N_2 atmosphere, rt.

[b]All the compounds were characterized by IR, ^{1}H NMR, ^{13}C NMR, MS, and HRMS spectral data.

[c]Isolated yield after purification.



Scheme 2. (i) Reaction conditions: dry ether, rt, N_2 atmosphere, 6 h.

cycloaddition reaction with nitrone **1** for the production of spirocycloadduct **3** in good yield (85%; Scheme 2). Simple nitrones can be also employed for the ketone synthesis, but the oxidation side products are waste and the reaction is not atom efficient. α-Chloronitrones (**1**) are more reactive than normal nitrones because of the electron-withdrawing effect of chlorine and therefore can act as more powerful oxidizing reagents than normal nitrones. A literature survey reveals that ketone synthesis using nitrone as active oxidizing reagent and further use of side product (obtained during ketone synthesis) as dipolarophile in cycloaddition reaction has not yet known and hence can be incorporated as an important application in nitrone chemistry.

α-Chloronitrones (**1**) are moderately stable and can be isolated, whereas transient nitrone **1a** could not be isolated because of its high instability and undergoes decomposition at room temperature. The lone pair of electrons of the OH group of α-chloronitrone facilitates intramolecular S_N2 reaction in the presence of pyridine and is actually the driving force for the development of transient nitrone **1a**. Nitrone **1a** reacts very quickly with different isopropyl halides (S_N2 reaction) and develops an intermediate compound **1b**. The labile N-O bond of **1b** undergoes cleavage[14] when the reaction mixture is stirred with solid sodium carbonate, which plays an important role for the development of ketone and furan derivative **2** as side products in a Kornblum-type process (Scheme 1; Table 1).

The synthetic potential of furan derivative (**2**) has been found to be tremendous in the regioselective synthesis of novel 5-substituted spiro cycloadduct **3** with nitrone **1** exclusively (Scheme 2), and the spectroscopic data of **3** are similar to the spiro cycloadducts reported earlier.[15–17]

The furan derivatives have been isolated from the organic layer and acetone from the aqueous mixture after completion of the reaction. The yield of isolated acetone (75–79%) was comparatively less because after a certain amount of acetone was removed from aqueous mixture by fractional distillation, the resulting solution became stable because of the formation of an azeotropic mixture and resisted fractional distillation. The same procedure was followed for the isolation of ethyl methyl ketone while benzophenone and acetophenone were isolated directly from the reaction mixture by column chromatography along with furan derivatives (**2**), and consequently the yields are comparatively greater than acetone and ethyl methyl ketone. The results are summarized in Table 1.

The beauty of the reaction is the addition of pyridine at the begining to generate transient nitrone **1a** and is capable of developing furan derivative **2**, which is

utilized as a new efficient dipolarophile in 1,3-dipolar cycloaddition reaction and thereby the reaction as a whole becomes atom efficient. At the outset of this work, we were not sure about the development of transient nitrone **1a**, but after completion of the study and spectral analysis of side product **2**, the development of transient nitrone **1a** was confirmed. The products, especially acetone, benzophenone, acetophenone, and ethyl methyl ketone, are known compounds and spectral data of the synthesized ketones are almost identical to the values found in literature. For example, a single sharp singlet signal at δ 2.12 and 202.00 in the NMR spectrum (^{1}H, ^{13}C respectively) along with molecular ion peak at 58 and base peak at 43 in the MS spectrum give strong evidence in favor of acetone formation. The R_f value of the synthesized acetone was found in the solvent front because of its volatility (difficult to identify because it evaporates rapidly) and was compared with acetone obtained from commercial suppliers. Expected signals for benzophenone, acetophenone, and ethyl methyl ketone has been found in ^{1}H NMR and ^{13}C NMR spectra while molecular ion peak and base peak values in the MS spectrum follow a general pattern of fragmentation of ketones in these compounds. The oxidation side product **2** was obtained as single isomer having *E* configuration in all the cases, and the yield of the side product was almost 10–13% when isolated in pure condition. The spiro cycloadduct **3** was obtained as regioselective single isomer predominantly in 1,3-DCR of α-chloronitrone **1** with side product **2** in good yield (85%) when isolated in pure condition. The stereochemistry of the 5-substituted regioselective spirocycloadduct **3** was rationalized by considering the multiplicity of the proton signals at 3-H, 4-H, and CHCl asymmetric centers along with their constant coupling values.[18] In the ^{1}H NMR spectrum of cycloadduct **3**, 3-H resonates around δ_H 5.84 ppm while 4–H resonates around δ_H 3.60 ppm. The coupling constant is $J_{3,4} \sim 8.36$ Hz, implying a *cis* relationship between H-3 and H-4. The CHCl proton also resonates around δ_H 2.68 ppm. The 3-H and CHCl protons are also *syn*, as evidenced from their coupling constant values ($J_{3,CHCl} \sim 8.70$ Hz). ^{1}H NMR spectrum of **3** also shows significant long-range coupling between H-4 with H-3′ and vice versa. In the mass spectrum, in addition to a molecular ion peak a prominent base peak value is obtained for the cycloadduct and significant $M^+ + 2$ peak of characteristic relative height is also obtained for **3**, which may be due to isotopic abundance of Cl^{37} atom. Studies of high-resolution mass spectrographic (HRMS) spectra show almost exact mass for the majority of the compounds. A preferential conformation for the spiroregioselective isoxazolidine derivative **3** may be represented in Fig. 1.

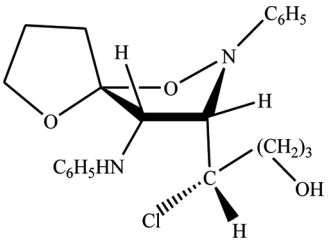


Figure 1. General conformational structure of spirocycloadduct **3**.

EXPERIMENTAL

^{1}H NMR spectra were recorded with a Bruker Avance DRX-300 spectrometer (300 MHz, FT NMR) using tetramethylsilane (TMS) as internal standard. ^{13}C NMR spectra were recorded on the same instrument at 75 MHz. The coupling constants (J) are given in hertz (Hz). Infrared (IR) spectra were obtained with a Perkin-Elmer RX 1-881 machine as film or KBr pellets for all the products. MS spectra were recorded with a Jeol SX-102 (FAB) instrument. The HRMS spectra were recorded on a DART-HRMS, JMS-T100LC, Accu-TOF instrument. Elemental analyses (CHN) were performed with a Perkin-Elmer 2400 series CHN analyzer. Thin-layer chromatography (TLC) was carried out on Fluka silica-gel TLC cards, while column chromatography was performed with silica gel (E. Merck, India), 60–200 mesh. All other reagents and solvents were purified after receiving from commercial suppliers. *N*-Methylhydroxylamine was purchased from Aldrich Chemical Company and was used as received. *N*-Phenylhydroxylamine was prepared following standard methods available in literature and has been used in various reported syntheses.[19–25]

General Procedure for Synthesis of Aliphatic Ketone and Furan Derivative 2 (Entry 1, Table 1)

Pyridine (1 equivalent) was added to a stirred solution of nitrone **1** (R=Me; 500 mg, 3.0198 mmol) in dry ether (25 mL) and stirred at rt with a magnetic stirrer under N_2 atmosphere for 1 h, while the formation of transient nitrone **1a** (not isolated) was monitored by TLC ($R_f = 0.38$). Isopropyl bromide (371.1002 mg, 1 equivalent) was added at this stage, and the reaction mixture was stirred for another 3 h until the intermediate compound **1b** (not isolated) developed (monitorted by TLC; $R_f = 0.40$). Two g of solid Na_2CO_3 were added at this stage, and the reaction mixture was stirred for another 1 h while the progress of the reaction was again monitored by TLC ($R_f = 0.50$, 0.96). The reaction was typically completed when the N-O bond was cleaved. Usual workup, removal of pyridine hydrochloride, and silica-gel column chromatographic purification using ethyl acetate–hexane provided furan derivative **2** (R=Me) as a pale yellow gummy liquid (10%; $R_f = 0.50$). During workup, the furan derivative went into the organic layer while the aqueous part containing acetone was separated from water by fractional distillation (79%; $R_f = 0.96$). This procedure was followed for the substrates **1–6** and **8** listed in Table 1.

Spectroscopic Data for Ethyl Methyl Ketone (Entry 8)

Colorless liquid (78%); $R_f = 0.74$; IR (KBr): 2940 (m), 1710 (s), 1450 (m), 1360 (m) cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 2.55 (q, 2H, $\underline{CH_2}CH_3$), 2.14 (s, 3H, CH_3), 1.24 (t, 3H, $CH_2\underline{CH_3}$); ^{13}CNMR ($CDCl_3$): δ 204.72 (C=O), 46.22 ($\underline{CH_2}CH_3$), 37.26 (CH_3), 17.43 ($CH_2\underline{CH_3}$); FAB-MS: m/z 72 (M^+), 57 (B.P), 29. HRMS-EI: calcd. for C_4H_8O (M), 72.0510; found M^+, 72.0496.

Spectroscopic Data for 2 (R=Me; α-N-Methyl Furan Derivative; Entry 1) [(E)-1-(Dihydrofuran-2-(3H)-ylidene)-N-Methyl Methanamine)]

Pale yellow gummy liquid (10%); $R_f = 0.50$; IR (KBr): 3120–3060 (br), 2835 (m), 1660 (s), 1450 (m), 1215 (m) cm^{-1}; ^1H NMR ($CDCl_3$): δ 4.81 (br, 1H, N-H), 4.56 (s, 1H, C=CH), 3.30 (N-Me), 2.50–2.16 (m, 6H); ^{13}C NMR ($CDCl_3$): δ 105.26, 102.70 (double bonded carbons), 27.32, 25.00, 24.12 (3 CH_2 carbons); FAB-MS: m/z 113 (M^+), 98, 97; HRMS-EI: calcd. for $C_6H_{11}ON$ (M), 113.0850; found M^+, 113.0832. Anal. found C, 63.52; H, 9.67; N, 12.30. $C_6H_{11}ON$ requires C, 63.67; H, 9.79; N, 12.38%.

Spectroscopic Data for 2 (R=Ph; α-N-Phenyl Furan Derivative; Entry 4) [(E)-1-(Dihydrofuran-2-(3H)-ylidene)-N-phenyl Methanamine)]

Dark yellow viscous liquid (11.5%); $R_f = 0.52$; IR (KBr): 3154–3065 (br), 2865 (m), 1640 (s), 1440 (m), 1144 (m), 776 (s) cm^{-1}; ^1H NMR ($CDCl_3$): δ 7.83–7.74 (m, 5H, C_6H_5), 6.24 (br, 1H, N-H), 2.17 (s, 1H, C=CH), 1.79–1.18 (m, 6H); ^{13}C NMR ($CDCl_3$): δ 136.00, 135.18, 134.00, 132.62 (aromatic carbons), 104.20, 101.15 (double bonded carbons), 28.55, 26.43, 24.00 (3 CH_2 carbons). FAB-MS (m/z): 175 (M^+), 98, 97, 77. HRMS-EI: calcd. for $C_{11}H_{13}ON$ (M), 175.0993; found M^+, 175.0976. Anal. found C, 75.28; H, 7.41; N, 7.88. $C_{11}H_{13}ON$ requires C, 75.39; H, 7.47; N, 7.99%.

General Procedure for Synthesis of Aromatic Ketone and Furan Derivative 2 (Entry 9, Table 1)

Pyridine (1 equivalent) was added to a stirred solution of nitrone 1 (R=Me, 500 mg, 3.0198 mmol) in dry ether (25 mL) and stirred at rt with a magnetic stirrer under an N_2 atmosphere for 1 h while the formation of transient nitrone **1a** (not isolated) was monitored by TLC ($R_f = 0.38$). Bromo diphenyl methane (749 mg, 1 equivalent) was added at this stage, and the reaction mixture was stirred for another 5 h until the intermediate compound **1b** (not isolated) was developed (monitorted by TLC; $R_f = 0.40$). Two g of solid Na_2CO_3 were added at this stage, and the reaction mixture was stirred for another 1 h, while the progress of the reaction was again monitored by TLC ($R_f = 0.42$, 0.50). The reaction was typically completed when the N-O bond was cleaved. Usual workup, removal of pyridine hydrochloride, and silica-gel column chromatographic purification using ethyl acetate–hexane provided benzophenone as white flecks (83%; $R_f = 0.76$) and furan derivative 2 (R=Me) as pale yellow gummy liquid (12%; $R_f = 0.50$). The same procedure was followed for the isolation of acetophenone.

Spectroscopic Data for Benzophenone (Entry 9)

White flecks (78%); mp 47 °C (uncorrected); $R_f = 0.42$; IR (KBr): 3030 (m), 1685 (s), 776 (s) cm^{-1}; ^1H NMR ($CDCl_3$): δ 7.63–7.38 (m, 5H, C_6H_5); ^{13}CNMR ($CDCl_3$): δ 195.70 (C=O), 138.22, 137.56, 137.05, 135.92, 133.74, 132.10, 130.83,

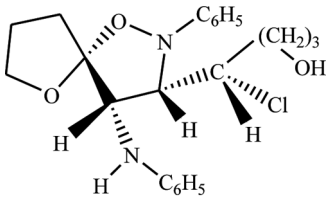
129.14; FAB-MS: m/z 182 (M^+), 105 (B.P), 77, 51, HRMS-EI: calcd. for $C_{13}H_{10}O$ (M), 182.0720; found M^+, 182.0706.

Spectroscopic Data for Acetophenone (Entry 7)

Colorless liquid (81%); $R_f = 0.70$; IR (KBr): 3035 (m), 1682 (s) cm^{-1}; ^1H NMR ($CDCl_3$): δ 7.16–7.04 (m, 5H, C_6H_5), 2.38 (s, 3H, CH_3); $^{13}CNMR$ ($CDCl_3$): δ 200.56 (C=O), 134.88, 132.90, 131.34, 129.54 (aromatic carbons), 28.33 (Me carbon); FAB-MS: m/z 120 (M^+), 105, 77 (B.P), 51; HRMS-EI: calcd. for C_8H_8O (M), 120.0550; found M^+, 120.0537.

General Procedure for Cycloaddition Reaction of Nitrone 1 (R=Ph) with Furan Derivative 2 (R=Ph)

Compound **2** (R=Ph, 50 mg, 0.2855 mmol, 1 equivalent) was added to a stirred solution of *N*-phenyl-α-chloronitrone **1** (R=Ph; 61.8375 mg, 0.2855 mmol) in 25 mL dry ether and stirred at rt with a magnetic stirrer under N_2 atmosphere for 6 h. The progress of the reaction was monitored by TLC ($R_f = 0.46$). After completion of the reaction, the solvent was evaporated using a rotary evaporator to afford crude cycloadduct **3**, which was purified by column chromatography using ethyl acetate–hexane and was obtained as a dark red viscous liquid **3** (85%; Scheme 2).



3

(S)-4-Chloro-4-((3 S,4 S,5R)-2-phenyl-4-(phenylamino)-1,6-dioxa-2-azaspiro[4.4]nonan-3-yl)butan-1-ol 3

3 Dark red viscous liquid. Yield 85%, $R_f = 0.46$; IR (KBr): 3480–3296 (br), 2960 (m), 2422 (m), 1620 (s), 1480 (s), 1265 (m), 1044 (m), 780 (s) cm^{-1}; ^1H NMR ($CDCl_3$): δ 6.98–6.92 (m, 10H, $2 \times C_6H_5$), 5.84 (dd, 1H, $J = 8.55$, 8.20 Hz, C_3H), 5.00 (br, 1H, CH_2OH, exchanged in D_2O), 3.60 (dt, 1H, $J = 8.34$, 8.08 Hz, C_4H), 3.40 (s, 1H, N–H proton of NHPh), 2.68 (dt, $J = 8.60$, 8.84 Hz, 1H, CHCl), 1.90 (dt, 2H, $J = 6.82$, 6.64 Hz, C_3·H), 1.50–1.12 (m, 10H); ^{13}C NMR ($CDCl_3$): δ 137.40, 136.10, 135.42, 133.00, 132.05, 130.54, 129.00, 128.13 (aromatic carbons), 94.16 (CHCl), 87.50 (C_5), 74.12 (C_3), 53.00 (C_4), 31.34, 28.00, 27.18, 25.00, 24.30, 22.92 (6 CH_2 carbons); MS (m/z): 404 ($M^+ + 2$), 402 (M^+), 325, 310, 309, 218 (B.P), 107, 91, 77. HRMS-EI: calcd. for $C_{22}H_{27}O_3N_2Cl$ (M), 402.1710, found M^+, 402.1702. Anal. found C, 65.58; H, 6.69; N, 6.85; $C_{22}H_{27}O_3N_2Cl$ requires C, 65.64; H, 6.76; N, 6.96%.

CONCLUSION

Finally, we developed a new atom-efficient methodology for the ketone synthesis using α-chloronitrones as oxidizing reagent and considered a further reaction carried out on the side product with α-chloronitrones in 1,3-dipolar cycloaddition reaction for the development of stereochemically important spirocycloadducts. The desired cycloadducts were obtained in good yields within a short reaction time. The newly developed side products (furan derivatives, **2**) are equally effective as dipolarophile in cycloaddition reactions, like other conventional dipolarophiles used for cycloaddition reactions. The notable advantage offered by this method is successful use of side products as dipolarophiles that can be used in various 1,3-dipolar cycloaddition reactions for the synthesis of spirocycloadducts. The procedure also involves simple operation, easy workup, and mild and fast reaction conditions with good yield of products.

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