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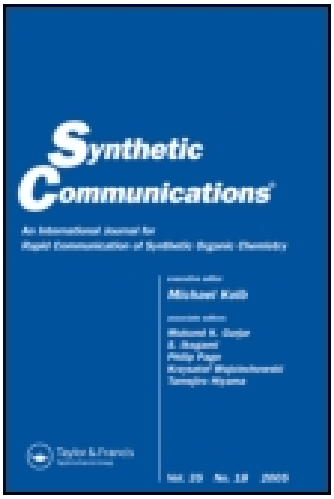

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EFFICIENT AND ALTERNATIVE APPROACH FOR PREPARATION OF *O*-BENZOYLOXIMES USING BENZOYL PEROXIDE

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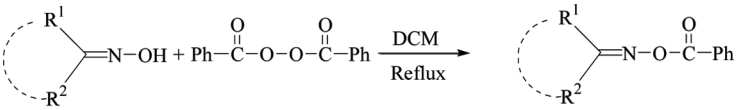
GRAPHICAL ABSTRACT



R^1 = Ph, Aryl, Alkyl

R^2 = H, Alkyl, Aryl

Abstract *Benzoyl peroxide has been used as a mild and efficient reagent for the preparation of benzoyl ester of oxime in moderate to good yields.*

Keywords Benzoyl peroxide; ester; oxime

INTRODUCTION

There are numerous reports on the use of oxime ester for peptide synthesis.[1] Because of the lesser reactivity of oxime esters toward an amine component, weak acids are used to enhance the reaction.[2] Hayashi and Shimizu[3] reported that aromatic oxime esters containing hydroxy groups are very effective for peptide synthesis (Scheme 1).

Another important use of oxime ester is as lipoprotein-associated phospholiphase A_2 inhibitors.[4] Very recently,[5] it has been observed that oxime ester photoinitiators such as OXE-1 and OXE-2 (Fig. 1) meet the specific requirements desired for color filter displays, which usually consist of translucent pixels of three primary colors (red, green, and blue) surrounded by a black matrix frame in mobile phones, notebook computers and televisions.

Hence, preparation of oxime ester is of much importance in organic synthesis. The only available methods in the literature for synthesis of acyl and benzoyl ester of

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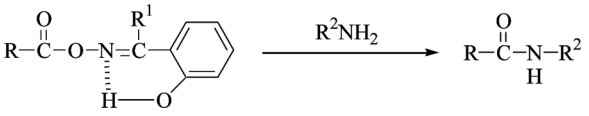
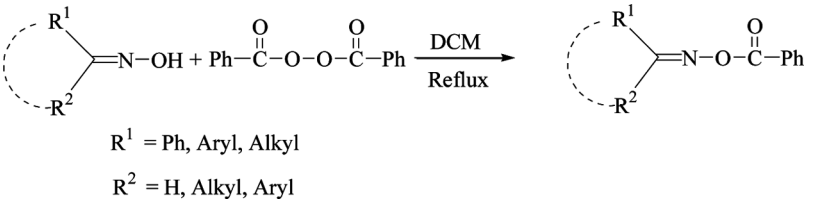


Scheme 1. Oxime ester for peptide synthesis.



Scheme 2. Synthesis of benzoyloximes.

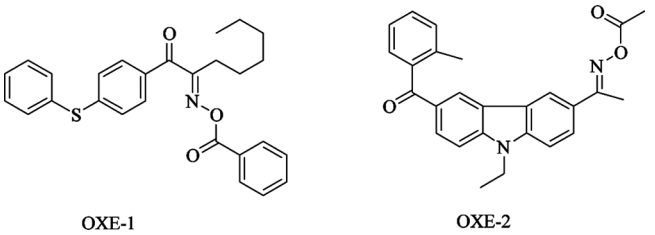


Figure 1. Photo-active oximes.

oxime are by treatment with acetic anhydride or acylchloride / benzoyl chloride in presence of triethylamine or pyridine as base.[6] Reaction of a (*E, Z*)-mixture or *Z*-oxime with benzoyl or acyl chlorides gives only *E*-isomers of ester as *Z*-isomer could be isomerized to the (*E*)-isomer by triethyl ammonium chloride.[7] Hence, to avoid this isomerization and use of pyridine in stoichiometric or even excess,[6] development of a simple and convenient method that can be applied under milder and environmentally friendly conditions is highly desirable. Benzoyl peroxide is usually used as radical initiator. It has been also used as benzoylation reagent of alcohols to form benzoylesters.[8] As a part of our research project to provide greener methodology,[9] we have observed that the reaction of benzoyl peroxide with oxime gives benzoyl ester of oxime in moderate to good yields under the present reaction conditions (Scheme 2).

RESULTS AND DISCUSSION

In a typical experimental procedure, a mixture of oxime (1 mmol) and benzoyl peroxide (1.5 mmol) in dichloromethane (2 ml) was refluxed in open atmospheric pressure for a certain period of time as required for the reaction (as monitored by thin-layer chromatography, TLC). After completion of the reaction, the crude

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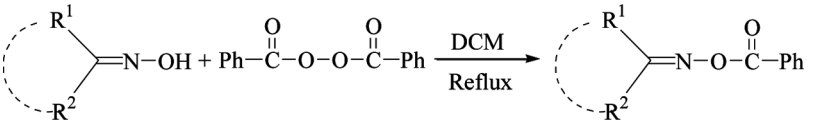
Table 1. Preparation of benzoyl ester of oxime



Entry	R^1	R^2	Time (h)	Yield (%)[a]	Ref.
1	C_6H_5	H	7	84	[10]
2	4-CH_3-C_6H_4	H	7	86	—
3	C_6H_5	CH_3	5	80	[10]
4	C_6H_5	C_2H_5	8	70	—
5	4-OCH_3-C_6H_4	H	6	87	—
6	4-OAllyl-C_6H_4	CH_3	6	85	—
7	C_6H_5	C_6H_5	4	85	[10]
8	$(CH_3)_2CHCH_2$	CH_3	3	78	—
9	C_2H_5	C_2H_5	3	90	—
10	α-Tetralone		6	88	—
11	Cyclohexanone		4	90	—
12	Cyclopentanone		3	90	—

[a]Pure isolated product.

mixture was washed with sodium bicarbonate solution and finally with brine solution. Evaporation of the organic solvent under reduced pressure afforded the product. A wide range of structurally diverse oxime was subjected to this procedure to get the corresponding ester as summarized in Table 1.

It has been observed that the aromatic ring containing electron-donating group such as –O-allyl, OMe, and Me is unaffected under the present reaction conditions and gives ester as the sole product. No amide or other product has been isolated in this reaction condition. Aldoxime with electron-withdrawing group such as -NO_2 and Cl in the benzene ring is inert under the present reaction conditions. The yields of some products were verified by repeating some experiments two or three times. The reaction conditions are same for all the reactions.

CONCLUSION

In conclusion, we have developed a highly efficient methodology for the synthesis of O-benzoyloxime derivatives with moderate to good yields under mild conditions without use of a base such as triethyl amine or pyridine. Operational simplicity and the compatibility with various functional groups are the advantages of the present procedure. We believe that this will present a very simple and more practical alternative to the existing methodologies for the synthesis of oxime ester.

EXPERIMENTAL

Melting points were determined on a glass disk with an electrical bath and were uncorrected. ^{1}H NMR (400-MHz) and ^{13}C NMR (100-MHz) spectra were run in

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$CDCl_3$ solutions. Infrared (IR) spectra were taken as KBr pellets in a Shimazdu 8400S FTIR. Elemental analyses were done on a Perkin-Elmer autoanalyzer.

Synthesis of Oxime Ester (Entry 3)

A mixture containing acetophenoneoxime (120 mg, 1 mmol) and benzoyl peroxide (363 mg, 1.5 mmol) in dichloromethane (2 ml) was refluxed in an open atmospheric pressure for a certain period of time as required for the reaction (TLC). After completion of the reaction, the crude mixture was washed with sodium bicarbonate solution (3×10 ml) and finally with brine solution (2×5 ml). Evaporation of the organic solvent under reduced pressure afforded the product. The crude product was purified (192 mg, 80%) by column chromatography (ethyl acetate and petroleum ether 60–80 °C). Solid; mp 96 °C (lit. 98–99 °C)[10]; IR (KBr, cm^{-1}): 3021, 2921, 1740, 1596, 1490, 1448. ^1H NMR ($CDCl_3$): δ 2.85 (s, 3H), 7.39–7.52 (m, 6H), 7.57–7.62 (m, 1H), 7.80–7.83 (m, 1H), 8.05–8.14 (m, 2H). ^{13}C NMR ($CDCl_3$): δ 15.12, 127.56 (2C), 128.82, 129.02 (2C), 129.29, 130.06, 130.19 (2C), 131.10, 133.78, 134.74, 164.09, 164.24.

All products were characterized by their spectral and analytical data.

Benzaldehyde Oxime Benzoyl Ester (1)

Yellow solid; mp 98 °C (lit. 99–100 °C)[10]; IR (KBr): 1764, 1741, 1596, 1450, 1226 cm^{-1}; ^1H NMR ($CDCl_3$): δ 7.44–7.62 (m, 5H), 7.79–7.83 (m, 2H), 8.07–8.15 (m, 3H), 8.5 (s, 1H, -NH). ^{13}C NMR ($CDCl_3$): δ 128.43, 128.53, 128.54, 128.83, 128.91, 129.75, 129.81, 130.21, 131.76, 133.38, 133.67, 134.21, 156.68, 171.79.

p-Methyl Benzaldehyde Oxime Benzoyl Ester (2)

Solid; mp 97 °C; IR (KBr): 1737, 1606, 1449 cm^{-1}; ^1H NMR ($CDCl_3$): δ 2.41 (s, 3H), 7.26 (d, 2H, $J = 8$ Hz), 7.49 (t, 2H, $J = 8$ Hz), 7.60 (t, 1H, $J = 8$ Hz), 7.71 (d, 2H, $J = 8$ Hz), 8.13 (d, 2H, $J = 3$ Hz), 8.53 (s, 1H, NH). ^{13}C NMR ($CDCl_3$): δ 21.65, 127.30, 128.50, 128.55, 128.73, 129.67, 129.71, 133.36, 142.40, 156.78, 164.07. Anal. calcd. for $C_{15}H_{13}NO_2$: C, 75.30; H, 5.48; N, 5.85. Found: C, 75.25; H, 5.41; N, 5.69.

Propeophenoneoxime Benzoyl Ester (4)

Solid; mp 98 °C; IR (KBr): 1747, 1604, 1452 cm^{-1}; ^1H NMR ($CDCl_3$): δ 1.29 (t, 3H, $J = 7.5$ Hz), 2.96 (t, 3H, $J = 7.5$ Hz), 7.25–7.36 (m, 6H), 7.93 (d, 2H, $J = 8.7$ Hz), 8.10 (d, 2H, $J = 7.8$ Hz). ^{13}C NMR ($CDCl_3$): δ 11.14, 21.81, 127 (2C), 128.11, 128.22, 128.34, 128.46, 128.79, 129.21, 130.11, 133.08, 133.48, 163.58, 168.14. Anal. calcd. for $C_{16}H_{15}NO_2$: C, 75.87; H, 5.97; N, 5.53. Found: C, 75.69; H, 5.91; N, 5.48.

4-Methoxybenzaldehyde Oxime Benzoyl Ester (5)

IR (KBr): 1746, 1607, 1461 cm^{-1}; ^1H NMR ($CDCl_3$): δ 1.19 (s, 3H), 3.84 (s, 3H), 6.94 (m, 2H), 7.23–7.26 (d, 1H, $J = 9$ Hz), 7.28–7.33 (t, 1H, $J = 7.5$ Hz), 7.43–7.49 (t, 2H, $J = 9$ Hz), 7.57–7.63 (m, 2H), 7.92–7.93 (d, 1H, $J = 1.5$ Hz), 7.96–8.12 (m, 1H).

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^{13}C NMR ($CDCl_3$): δ 19.95, 55.33, 113.52 (2C), 125.10, 127.99, 128.28, 128.52, 128.56, 129.40, 130.02, 161.56, 172.37. Anal. calcd. for $C_{15}H_{13}NO_3$: C, 70.58; H, 5.13; N, 5.49. Found: C, 70.45; H, 5.09; N, 5.46.

4-O-Allylacetophenone Oxime Benzoyl Ester (6)

IR (KBr): 1743, 1602, 1452 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 2.51 (s, 3 H), 4.56 (d, 2H, $J = 5.2$ Hz), 5.30 (d, 1H, $J = 10.5$ Hz), 5.41 (d, 1H, $J = 17.1$ Hz), 5.99–6.09 (m, 1H), 6.94 (d, 2H, $J = 8.0$ Hz), 7.48 (t, 2H, $J = 7.5$ Hz), 7.76 (d, 1H, $J = 7.5$ Hz), 7.78 (d, 2H, $J = 8$ Hz), 8.12 (d, 2H, $J = 7.5$ Hz). ^{13}C NMR ($CDCl_3$): δ 14.26, 68.78, 114.33, 114.65, 118.00, 127.17, 128.47, 128.58, 129.29, 129.51, 129.97, 130.38, 130.46, 132.73, 133.13, 160.60, 162.92, 163.78. Anal. calcd. for $C_{18}H_{17}NO_3$: C, 73.20; H, 5.80; N, 4.74. Found: C, 73.15; H, 5.76; N, 4.71.

Benzophenone Oxime Benzoyl Ester (7)

Solid; mp 99 °C (lit. 98–99 °C)[10]; IR (KBr): 1758, 1596, 1450, 1317 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 7.25–7.70 (m, 11H), 7.77 (t, 1H, $J = 3$ Hz), 7.81 (t, 1H, $J = 3$ Hz), 8.05 (t, 1H, $J = 3$ Hz), 8.09 (t, 1H, $J = 3$ Hz). ^{13}CNMR ($CDCl_3$): δ 125.58, 128.14 (2C), 128.32, 128.73 (2C), 129.00, 129.52, 129.66 (2C), 129.92 (2C), 130.89, 132.25 (2C), 134.11 (2C), 137.49, 162.79, 165.26.

Isobutyl Methyl Ketone Oxime Benzoyl Ester (8)

IR (KBr): 1746, 1600, 1453 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 1.07 (d, 6H, $J = 7.4$ Hz), 1.98–2.05 (m, 1H), 2.10 (s, 3H), 2.32 (d, 2H, $J = 7.4$ Hz), 7.60 (t, 2H, $J = 6.4$ Hz), 7.66 (t, 1H, $J = 6.4$ Hz), 8.07 (d, 2H, $J = 6.4$ Hz). ^{13}C NMR ($CDCl_3$): δ 15.60, 22.36, 22.78, 25.93, 44.46, 125.25, 128.49, 128.87, 129.55, 129.78, 134.38, 163.07, 167.05. Anal. calcd. for $C_{13}H_{17}NO_2$: C, 71.21; H, 7.81; N, 6.39. Found: C, 71.11; H, 7.75; N, 6.35.

Diethylketone Oxime Benzoyl Ester (9)

IR (KBr): 1743, 1633, 1456 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 1.18–1.24 (m, 6 H), 2.42–2.69 (m, 4H), 7.46 (t, 2H, $J = 7.2$ Hz), 7.55 (d, 1H, $J = 7.2$ Hz), 8.06–8.12 (d, 2H, $J = 7.2$ Hz). ^{13}C NMR ($CDCl_3$): δ 10.29, 10.57, 22.44, 27.12, 128.03, 128.23, 129.03, 129.15, 129.70, 132.87, 163.74, 172.47. Anal. calcd. for $C_{12}H_{15}NO_2$: C, 70.22; H, 7.37; N, 6.82. Found: C, 70.17; H, 7.31; N, 6.79.

α-Tetraloneoxime Benzoyl Ester (10)

IR (KBr): 1735, 1596, 1452 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 1.92–1.99 (m, 2H), 2.83 (t, 2H, $J = 6.4$ Hz), 3.04 (t, 2H, $J = 6.4$ Hz), 7.2 (d, 1H, $J = 7.6$ Hz), 7.27 (t, 1H, $J = 7.6$ Hz), 7.38 (t, 1H, $J = 7.6$ Hz), 7.50 (t, 1H, $J = 7.6$ Hz), 7.61 (t, 2H, $J = 7.6$ Hz), 7.66 (d, 1H, $J = 7.6$ Hz), 8.19 (d, 2H, $J = 7.6$ Hz), 8.31 (d, 1H, $J = 7.6$ Hz). ^{13}C NMR ($CDCl_3$): δ 21.37, 25.85, 29.56, 125.91, 126.65, 128.57 (2C), 128.68, 128.95, 129.33 (2C), 130.85, 133.27, 140.94, 162.47, 163.85. Anal. calcd. for $C_{17}H_{15}NO_2$: C, 76.96; H, 5.70; N, 5.28. Found: C, 76.79; H, 5.62; N, 5.20.

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Cyclohexanoneoxime Benzoyl Ester (11)

IR (KBr): 1741, 1637, 1448 cm^{-1}; ^1H NMR ($CDCl_3$): δ 1.58–1.76 (m, 6 H), 2.41 (t, 2H, $J = 6.3$ Hz), 2.62 (t, 2H, $J = 6.3$ Hz), 7.43 (t, 2H, $J = 6.9$ Hz), 7.55 (t, 1H, $J = 6.9$ Hz), 8.01 (d, 2H, $J = 6.9$ Hz). ^{13}C NMR ($CDCl_3$): δ 25.49, 25.66, 26.57, 26.92, 31.98, 128.11 (2C), 128.27, 129.14 (2C), 129.32, 129.79, 132.94, 164.09, 164.41. Anal. calcd. for $C_{13}H_{15}NO_2$: C, 71.87; H, 6.96; N, 6.45. Found: C, 71.80; H, 6.90; N, 6.41.

Cyclopentanone Oxime Benzoyl Ester (12)

IR (KBr): 1742, 1658, 1450, 1255 cm^{-1}; ^1H NMR ($CDCl_3$): δ 1.81 (bs, 4H), 2.60 (t, 2H, $J = 6.8$ Hz), 2.66 (t, 2H, $J = 6.8$ Hz), 7.44 (t, 2H, $J = 7.3$ Hz), 7.56 (t, 1H, $J = 7.3$ Hz), 8.05 (d, 2H, $J = 7.3$ Hz). ^{13}C NMR ($CDCl_3$): δ 24.09, 24.69, 28.92, 31.06, 128.08 (2C), 128.69, 129.08 (2C), 132.79, 163.59, 175.88. Anal. calcd. for $C_{12}H_{13}NO_2$: C, 70.92; H, 6.45; N, 6.89. Found: C, 70.81; H, 6.41; N, 6.79.

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