

Reactions of cyclohexyl isocyanide, dialkyl acetylenedicarboxylates and 1-aryl-2-ene-3-acetyl-1,4-diketones: one-pot synthesis of highly functionalized 5-cyclohexylimino-2,5-dihydrofurans

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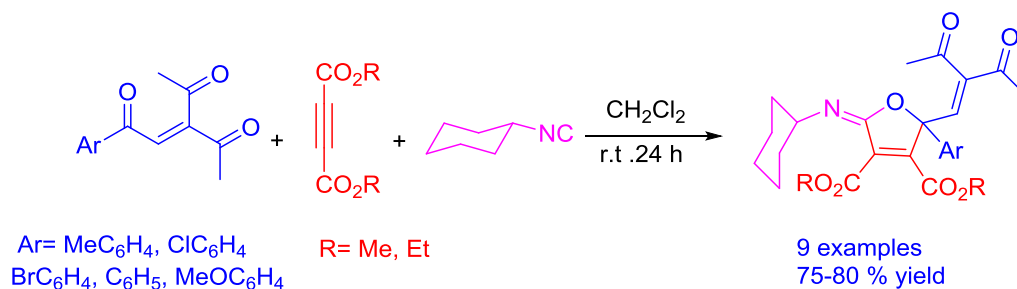
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Abstract

Dihydrofurans are important intermediates in organic synthesis, and are also important starting materials used in syntheses of a number of natural products. A facile synthesis of highly functionalized 5-cyclohexylimino-2,5-dihydrofuran derivatives by the multi-component reaction of cyclohexyl isocyanide, dialkyl acetylenedicarboxylates and 1-aryl-2-ene-3-acetyl-1,4-diketones is described.



Keywords: Isocyanide, multi-component reactions, dialkyl acetylenedicarboxylates, arylglyoxal, 5-imino-2,5-dihydrofuran

Introduction

The Michael addition of isocyanides to activated carbon-carbon multiple bonds produces reactive zwitterionic intermediates which may be trapped by carbon or hydrogen proton electrophiles to afford a wide range of carbocyclic or heterocyclic organic compounds. It has been reported that isocyanides attack dialkyl acetylenedicarboxylates (DAADs), yielding zwitterionic species which could be trapped by the carbonyl group of aldehydes,¹ ketones,² esters,³ isocyanates,⁴ acyl chlorides⁵, and even carbon dioxide⁶, to afford five- to seven-membered oxygen and nitrogen heterocyclic compounds.

Multi-component reactions of arylglyoxals have recently attracted much attention for the synthesis of a wide range of important heterocyclic compounds. The application of arylglyoxals for the preparation of three- to six-membered heterocyclic compounds has been recently reviewed.⁷

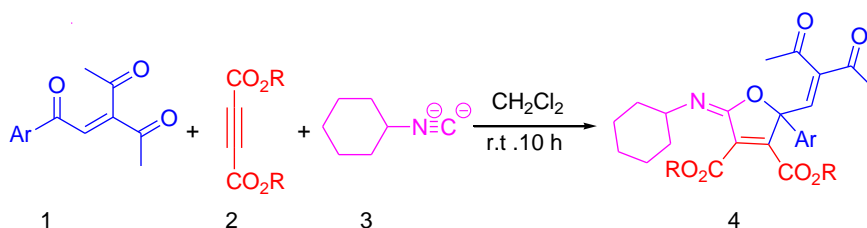
Dihydrofurans are important intermediates in organic synthesis and have been attracting much attention in synthetic studies^{8,9}. Several methods have been developed for their preparation.¹⁰⁻¹⁷ 2,5-Disubstituted dihydrofuran-3,4-dicarboxylates are important starting materials which have been used for the synthesis of some natural products.¹⁸ Thus, developing new, simple and efficient syntheses of dihydrofuran derivatives have been an area of interest.

In recent years, we have been focusing our attention on the applicability of multi-component reactions of isocyanides^{19,20} and arylglyoxals^{21,22} for the synthesis of heterocyclic compounds. In continuation of our studies in this area, we wish to report, herein, the reaction of 1-aryl-2-ene-3-acetyl-1,4-diketone derivatives, prepared by Knoevenagel condensation of arylglyoxals with acetylacetone,²² with isocyanides and DAADs to produce 5-imino-2,5-dihydrofuran derivatives in good yields.

Results and Discussion

To investigate the reactions of cyclohexyl isocyanide, DAADs and 1-aryl-2-ene-3-acetyl-1,4-diketone derivatives, dimethyl acetylenedicarboxylate (DMAD) was added to a mixture of cyclohexyl isocyanide and 3-acetyl-1-*p*-tolyl-2-ene-1,4-dione (**1**, Ar = *p*-tolyl) in CH₂Cl₂ as solvent at room temperature. Progress of the reaction was monitored by TLC. After 10 h, TLC analysis of the reaction mixture showed the presence of only dimethyl (E)-2-(2-acetyl-3-oxobut-1-en-1-yl)-5-(cyclohexylimino)-2-(4-methylphenyl)-2,5-dihydrofuran-3,4-dicarboxylate (**4a**). Silica-gel chromatography afforded **4a** in 80% yield.

To investigate the scope of the reaction, different DAADs were treated with cyclohexyl isocyanide and different 1-aryl-2-ene-3-acetyl-1,4-diketones. The corresponding 5-cyclohexylimino-2,5-dihydrofuran derivatives **4a-i** were obtained in good yields (Scheme 1).



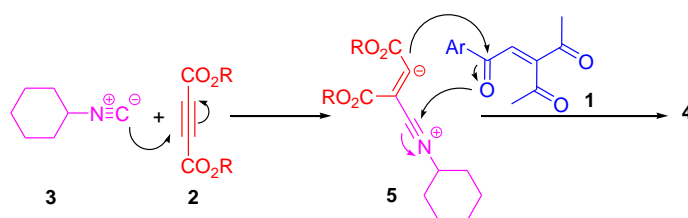
4	Ar	R	Yield%*
4a	MeC_6H_4	Me	80
4b	MeC_6H_4	Et	78
4c	BrC_6H_4	Me	80
4d	BrC_6H_4	Et	75
4e	ClC_6H_4	Me	80
4f	ClC_6H_4	Et	75
4g	C_6H_5	Me	80
4h	C_6H_5	Et	78
4i	MeOC_6H_4	Et	80

*Isolated yields

Scheme 1. Three-component reaction of cyclohexyl isocyanide, dialkyl acetylenedicarboxylates and 1-aryl-2-ene-3-acetyl-1,4-diketones.

The structures of compounds **4a-i** were deduced from their elemental analyses and their infrared (IR), ^1H NMR, and ^{13}C NMR spectral data. The 400-MHz ^1H NMR spectrum of **4a** exhibited five sharp signals at δ 2.34, 2.39, 2.42, 3.80 and 3.96 ppm for the three methyl and two methoxy groups. The cyclohexyl protons resonated as multiplets at 1.22-1.85 (5 CH_2) and at 3.51 (CH) ppm. The aromatic protons resonated as a multiplet at 7.25 ppm and the olefinic CH was observed at 7.44 ppm as a singlet. The ^{13}C NMR spectrum of compound **4a** showed twenty-five distinct resonances in agreement with the proposed structure. The structural assignment for compound **4a**, made on the basis of the NMR spectra, were supported by its IR spectrum as the ester carbonyl groups exhibited strong absorption bands at about 1755 and 1724cm^{-1} , respectively. The ketone carbonyls were observed as strong absorptions at 1684 and 1669cm^{-1} , respectively.

The suggested mechanism for formation of the 5-cyclohexylimino-2,5-dihydrofuran derivatives **4a-i** by the reaction of cyclohexyl isocyanide, DAAD and 1-aryl-2-ene-3-acetyl-1,4-diketone derivatives is shown in Scheme 2. Michael addition of cyclohexyl isocyanide to DAAD produces the reactive zwitterionic species (**5**). The formal [3+2] type cycloaddition of this reactive intermediate with the carbonyl group of 1-aryl-2-ene-3-acetyl-1,4-diketone (**1**) affords the 5-cyclohexylimino-2,5-dihydrofuran product series **4**.



Scheme 2. Suggested mechanism for formation of 5-cyclohexylimino-2,5-dihydrofuran derivatives **4a-i**.

Conclusions

In conclusion, we have developed a simple and efficient method for preparation of functionalized 5-cyclohexylimino-2,5-dihydrofuran derivatives by a three-component reaction of cyclohexyl isocyanide, dialkyl acetylenedicarboxylates and 1-aryl-2-ene-3-acetyl-1,4-diketone derivatives. This method has the advantages that the reactions can be performed under neutral conditions with readily available starting materials which require no modification or preparation.

Experimental Section

General. All of the utilized arylglyoxals were prepared by the SeO_2 -oxidation of the related aryl methyl ketones on the basis of the reported procedure and used as their monohydrates.²⁴ Elemental analyses were performed using a Heraeus CHN-O-Rapidanalyzer. IR spectra were recorded on a Shimadzu IR-470 spectrometer. ^1H and ^{13}C NMR spectra were recorded on a Bruker DRX-400 Avance spectrometer at 400 and 100 MHz, respectively. The chemicals used in this work were purchased from Merck and used without further purification.

General procedure. To a magnetically-stirred solution of dialkyl acetylenedicarboxylate (2 mmol) and 1-aryl-2-ene-3-acetyl-1,4-diketone derivative (2 mmol) in CH_2Cl_2 (10 mL) was added a solution of cyclohexyl isocyanide (2 mmol) in CH_2Cl_2 (5 mL) dropwise at room temperature over 10 min. The mixture was then allowed to stir for 10 h. The solvent was removed under reduced pressure, and the residue was separated by column chromatography (silica gel, hexane–EtOAc, 5:1) to afford the pure title compounds.

Dimethyl (E)-2-(2-acetyl-3-oxobut-1-en-1-yl)-5-(cyclohexylimino)-2-(4-methylphenyl)-2,5-dihydrofuran-3,4-dicarboxylate (4a). Yellow solid, (770 mg, 80%). mp 169-172°C. IR (solid, KBr, ν_{max} cm^{-1}): 1755, 1724, 1684, 1669 (4C=O). ^1H NMR (400 MHz, CDCl_3): δ_{H} 1.22 -1.85 (10H, m, 5 CH_2 of cyclohexyl), 2.34, 2.39, 2.42 (9H, 3s, 3 CH_3), 3.51 (1H, m, CH of cyclohexyl), 3.80, 3.96 (6H, 2s, 2OMe), 7.25 (4H, m, 4CH, HAr), 7.44 (1H, s, CH olefinic). ^{13}C NMR (100 MHz, CDCl_3) δ_{C} 21.2, 24.5, 24.7, 25.6, 26.6, 32.3, 33.0, 33.8 (5 CH_2 of cyclohexyl and 3 CH_3), 53.1, 53.2 (2OMe), 57.0 (CH of cyclohexyl), 90.1 (C of dihydrofuran), 125.9, 129.9, 133.9, 136.4, 137.1, 139.7, 142.6, 143.7 (aromatic and olefinic carbons), 152.4 (C=N), 160.8, 161.9 (2C=O of esters), 196.3, 202.0 (2C=O of ketones). Calcd. for ($\text{C}_{27}\text{H}_{31}\text{NO}_7$): C, 67.35; H, 6.49; N, 2.91. Found: C, 67.33; H, 6.42; N, 2.95.

Diethyl (E)-2-(2-acetyl-3-oxobut-1-en-1-yl)-5-(cyclohexylimino)-2-(4-methylphenyl)-2,5-dihydrofuran-3,4-dicarboxylate (4b). Yellow oil, (794 mg, 78%). IR (neat, ν_{max} cm^{-1}): 1724, 1675 (4C=O). ^1H NMR (400 MHz, $\text{DMSO}-d_6$) δ_{H} 1.17 (3H, t, $^3J_{\text{HH}}$ 8.0 Hz, CH_3), 1.30 (3H, t, $^3J_{\text{HH}}$ 8.0 Hz, CH_3), 1.21-1.75 (10H, m, 5 CH_2 of cyclohexyl), 2.23, 2.34, 2.44 (9H, 3s, 3 CH_3), 3.35 (1H, m, CH of cyclohexyl), 4.22 (2H, m, CH_2), 4.36 (2H, m, CH_2), 7.26 (2H, d, $^3J_{\text{HH}}$ 9.0 Hz, 2CH, HAr), 7.31 (2H, d, $^3J_{\text{HH}}$ 9.0 Hz, 2CH, HAr), 7.36 (1H, s, CH olefinic). ^{13}C NMR (100 MHz, $\text{DMSO}-d_6$) δ_{C} 14.0, 14.3, 21.3, 24.5, 24.6, 25.6, 26.9, 32.4, 33.1, 34.0 (5 CH_2 of cyclohexyl and 5 CH_3), 55.3 (CH of cyclohexyl) 62.6, 62.7 (2 OCH_2), 90.4 (C of dihydrofuran), 126.2, 130.31, 134.3, 135.9, 136.9, 139.8, 143.2, 143.6 (aromatic and olefinic carbons), 153.0 (C=N), 159.9, 161.4 (2C=O of esters), 197.4, 202.4 (2C=O of ketones). Calcd. for ($\text{C}_{29}\text{H}_{35}\text{NO}_7$): C, 68.35; H, 6.92; N, 2.75. Found: C, 68.33; H, 6.85; N, 2.85.

Dimethyl (E)-2-(2-acetyl-3-oxobut-1-en-1-yl)-2-(4-bromophenyl)-5-(cyclohexylimino)-2,5-dihydrofuran-3,4-dicarboxylate (4c). White solid, (874 mg, 80%). mp 137-140 °C. IR (solid, KBr, ν_{max} cm^{-1}): 1752, 1731, 1688, 1674 (4C=O). ^1H NMR (400 MHz, CDCl_3) δ_{H} 1.22-1.83 (10H, m, 5 CH_2 of cyclohexyl), 2.33, 2.41 (6H, 2s, 2 CH_3), 3.51 (1H, m, CH of cyclohexyl), 3.82, 3.96 (6H, 2s, 2OMe), 7.26 (2H, d, $^3J_{\text{HH}}$ 9.0 Hz, 2CH, HAr), 7.38 (1H, s, CH olefinic), 7.57 (2H, d, $^3J_{\text{HH}}$ 9.0 Hz, 2CH, HAr). ^{13}C NMR (100 MHz, CDCl_3) δ_{C} 24.5, 24.7, 25.5, 26.7, 32.2, 33.0,

33.8 (5CH₂ of cyclohexyl and 2CH₃), 53.2, 53.3 (2OMe), 57.1 (CH of cyclohexyl), 89.5 (C of dihydrofuran), 124.0, 127.7, 132.4, 136.1, 136.2, 136.7, 142.0, 144.2 (aromatic and olefinic carbons), 152.0 (C=N), 160.7, 161.7 (2C=O of esters), 196.1, 202.1 (2C=O of ketones). Calcd. for (C₂₆H₂₈BrNO₇): C, 57.15; H, 5.17; N, 2.56. Found: C, 57.18; H, 5.12; N, 2.50.

Diethyl (E)-2-(2-acetyl-3-oxobut-1-en-1-yl)-2-(4-bromophenyl)-5-(cyclohexylimino)-2,5-dihydrofuran-3,4-dicarboxylate (4d). Yellow oil (861 mg, 75%). IR (neat, ν_{\max} cm⁻¹): 1724, 1676 (4C=O). ¹H NMR (400 MHz, CDCl₃): δ_{H} 1.25 (3H, t, ³J_{HH} 7.0 Hz, CH₃), 1.36 (3H, t, ³J_{HH} 7.0 Hz, CH₃), 1.23 - 1.80 (10H, m, 5CH₂ of cyclohexyl), 2.28, 2.37 (6H, 2s, 2CH₃), 3.48 (1H, m, CH of cyclohexyl), 4.22 (2H, m, CH₂), 4.38 (2H, q, ³J_{HH} 7.0 Hz, CH₂), 7.24 (2H, d, ³J_{HH} 9.0 Hz, 2CH, HAr), 7.38 (1H, s, CH olefinic), 7.53 (2H, d, ³J_{HH} 9.0 Hz, 2CH, HAr). ¹³C NMR (100 MHz, CDCl₃) δ_{C} 13.8, 14.0, 24.4, 24.6, 25.6, 26.6, 32.2, 33.0, 33.8 (5CH₂ of cyclohexyl and 4CH₃), 56.9 (CH of cyclohexyl), 62.4, 62.5 (2OCH₂), 89.5 (C of dihydrofuran), 123.8, 127.8, 132.3, 136.3, 136.4, 136.6, 141.8, 143.9 (aromatic and olefinic carbons), 152.1 (C=N), 160.3, 161.3 (2C=O of esters), 196.2, 201.8 (2C=O of ketones). Calcd. for (C₂₈H₃₂BrNO₇): C, 58.54; H, 5.61; N, 2.44. Found: C, 58.28; H, 5.82; N, 2.50.

Dimethyl (E)-2-(2-acetyl-3-oxobut-1-en-1-yl)-2-(4-chlorophenyl)-5-(cyclohexylimino)-2,5-dihydrofuran-3,4-dicarboxylate (4e). White solid (803 mg, 80%). mp 170-173°C. IR (solid, KBr, ν_{\max} cm⁻¹): 1754, 1723, 1683 (4C=O). ¹H NMR (400 MHz, CDCl₃): δ_{H} 1.18 - 1.77 (10H, m, 5CH₂ of cyclohexyl), 2.29, 2.37 (6H, 2s, 2CH₃), 3.47 (1H, m, CH of cyclohexyl), 3.77, 3.92 (6H, 2s, 2OMe), 7.28 (2H, d, ³J_{HH} 8.0 Hz, 2CH, HAr), 7.35 (1H, s, CH olefinic), 7.37 (2H, d, ³J_{HH} 8.0 Hz, 2CH, HAr). ¹³C NMR (100 MHz, CDCl₃) δ_{C} 24.4, 24.6, 26.6, 32.2, 33.0, 33.8 (5CH₂ of cyclohexyl and 2CH₃), 53.2 (2OMe), 57.1 (CH of cyclohexyl), 89.5 (C of dihydrofuran), 127.4, 129.4, 135.6, 135.7, 136.2, 136.7, 142.0, 144.1 (aromatic and olefinic carbons), 152.0 (C=N), 160.7, 161.7 (2C=O of esters), 196.0, 201.7 (2C=O of ketones). Calcd. for (C₂₆H₂₈ClNO₇): C, 62.21; H, 5.62; N, 2.79. Found: C, 62.19; H, 5.59; N, 2.77.

Diethyl (E)-2-(2-acetyl-3-oxobut-1-en-1-yl)-2-(4-chlorophenyl)-5-(cyclohexylimino)-2,5-dihydrofuran-3,4-dicarboxylate (4f). White solid (795 mg, 75%). mp 155-158°C IR (solid, KBr, ν_{\max} cm⁻¹): 1724, 1676 (4C=O). ¹H NMR (400 MHz, CDCl₃): δ_{H} 1.23 (3H, t, ³J_{HH} 7.1 Hz, CH₃), 1.34 (3H, t, ³J_{HH} 7.1 Hz, CH₃), 1.21-1.76 (10H, m, 5CH₂ of cyclohexyl), 2.27, 2.35 (6H, 2s, 2CH₃), 3.47 (1H, m, CH of cyclohexyl), 4.20 (2H, m, CH₂), 4.36 (2H, q, ³J_{HH} 7.0 Hz, CH₂), 7.27-7.36 (5H, m, CH aromatic and olefinic). ¹³C NMR (100 MHz, CDCl₃) δ_{C} 13.7, 14.0, 24.3, 24.5, 25.5, 26.5, 32.1, 33.0, 33.8 (5CH₂ of cyclohexyl and 4CH₃), 56.9 (CH of cyclohexyl), 62.4 (2OCH₂), 89.4 (C of dihydrofuran), 127.5, 129.3, 135.5, 135.8, 136.4, 136.6, 141.9, 144.0 (aromatic and olefinic carbons), 152.0 (C=N), 160.3, 161.3 (2C=O of esters), 196.0, 201.7 (2C=O of ketones). Calcd. for (C₂₈H₃₂ClNO₇): C, 63.45; H, 6.09; N, 2.64. Found: C, 63.40; H, 6.03; N, 2.60.

Dimethyl (E)-2-(2-acetyl-3-oxobut-1-en-1-yl)-5-(cyclohexylimino)-2-phenyl-2,5-dihydrofuran-3,4-dicarboxylate (4g). Yellow solid (748 mg, 80%), mp 178-180°C. IR (solid, KBr, ν_{\max} cm⁻¹): 1754, 1719, 1681, 1669 (4C=O). ¹H NMR (400 MHz, CDCl₃): δ_{H} 1.19-1.82 (10H, m, 5CH₂ of cyclohexyl), 2.30, 2.38 (6H, 2s, 2CH₃), 3.50 (1H, m, CH of cyclohexyl), 3.77, 3.92 (6H, 2s, 2OMe), 7.34-7.42 (6H, m, CH aromatic and olefinic). ¹³C NMR (100 MHz, CDCl₃) δ_{C} 24.5, 24.7, 25.6, 26.5, 32.2, 33.0, 33.8 (5CH₂ of cyclohexyl and 2CH₃), 53.0, 53.1 (2OMe), 57.0 (CH of cyclohexyl), 90.0 (C of dihydrofuran), 125.9, 129.1, 129.5, 136.5, 136.9, 137.0, 142.5, 143.8 (aromatic and olefinic carbons), 152.2 (C=N), 160.8, 161.8 (2C=O of esters), 196.1, 201.8 (2C=O of ketones). Calcd. for (C₂₆H₂₉NO₇): C, 66.80; H, 6.25; N, 3.00. Found: C, 66.78; H, 6.20; N, 3.07.

Diethyl (E)-2-(2-acetyl-3-oxobut-1-en-1-yl)-5-(cyclohexylimino)-2-phenyl-2,5-dihydrofuran-3,4-dicarboxylate (4h). White solid (773 mg, 78%) mp 150-153°C. IR (solid, KBr, ν_{\max} cm⁻¹): 1747, 1717, 1673 (4C=O). ¹H NMR (400 MHz, CDCl₃): δ_{H} 1.24 (3H, t, ³J_{HH} 7.0 Hz, CH₃), 1.36 (3H, t, ³J_{HH} 7.0 Hz, CH₃), 1.22-1.79 (10H, m, 5CH₂ of cyclohexyl), 2.30, 2.38 (6H, 2s, 2CH₃), 3.50 (1H, m, CH of cyclohexyl), 4.22 (2H, m, CH₂), 4.38 (2H, q, ³J_{HH} 8.0 Hz, CH₂), 7.35 - 7.42 (5H, m, 5CH, HAr), 7.45 (1H, s, CH olefinic). ¹³C NMR (100 MHz, CDCl₃) δ_{C} 13.7, 14.0, 24.4, 24.6,

25.6, 26.5, 32.2, 33.0, 33.8 (5CH₂ of cyclohexyl and 4CH₃), 56.8 (CH of cyclohexyl), 62.2, 62.3 (2OCH₂), 90.0 (C of dihydrofuran), 126.0, 129.1, 129.4, 136.4, 137.1, 137.2, 142.3, 143.7 (aromatic and olefinic carbons), 152.3 (C=N), 160.4, 161.4 (2C=O of esters), 196.2, 201.8 (2C=O of ketones). Calcd. for (C₂₈H₃₃NO₇): C, 67.86; H, 6.71; N, 2.83. Found: C, 67.86; H, 6.71; N, 2.83.

Diethyl (E)-2-(2-acetyl-3-oxobut-1-en-1-yl)-5-(cyclohexylimino)-2-(4-methoxyphenyl)-2,5-dihydrofuran-3,4-dicarboxylate (4i). Yellow oil (841 mg, 80%). IR (neat, ν_{\max} cm⁻¹): 1720 (4C=O). ¹H NMR (400 MHz, CDCl₃): δ_{H} 1.24 (3H, t, ³J_{HH} 7.1 Hz, CH₃), 1.34 (3H, t, ³J_{HH} 7.1 Hz, CH₃), 1.23-1.77 (10H, m, 5CH₂ of cyclohexyl), 2.29, 2.37 (6H, 2s, 2CH₃), 3.48 (1H, m, CH of cyclohexyl), 3.80 (3H, s, OMe), 4.21 (2H, m, CH₂), 4.37 (2H, m, CH₂), 7.27 (4H, m, 4CH aromatic), 7.41 (1H, s, CH olefinic). ¹³C NMR (100 MHz, CDCl₃) δ_{C} 13.8, 14.2, 24.3, 24.6, 25.7, 26.2, 32.2, 33.1, 33.8 (5CH₂ of cyclohexyl and 4CH₃), 53.2 (OCH₃), 56.7 (CH of cyclohexyl), 62.3, 62.5 (2OCH₂), 90.0 (C of dihydrofuran), 123.0, 125.1, 126.4, 137.1, 137.2, 142.3, 143.7, 151.3 (aromatic and olefinic carbons), 152.3 (C=N), 160.2, 161.5 (2C=O of esters), 196.0, 201.2 (2C=O of ketones). Calcd. for (C₂₉H₃₅NO₈): C, 66.27; H, 6.71; N, 2.66. Found: C, 66.36; H, 6.78; N, 2.83.

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