CHAPTER 3

REACTIONS OF NITRONES WITH BURGESS REAGENT

3.1 Abstract

This chapter deals with the reactions of various nitrones with Burgess reagent. 1,3-Dipolar species such as nitrones and azomethine imines undergo annulation reactions with Burgess reagent. Preliminary studies indicated that nitrones undergo useful transformations with Burgess reagent. The reaction involves a [3+2] annulation followed by a rearrangement involving C-to-N aryl migration. Based on the available experimental evidence, plausible mechanisms for the rearrangement and the overall conversion have been proposed.

3.2 Introduction

Burgess reagent (1) is a versatile reagent in organic synthesis\(^1,2\) and its reactivity with a number of functional groups like alcohols, epoxides\(^3\), 1,2-diols\(^4-6\), thiols\(^7,8\) are well documented. Newer applications\(^9\) of the reagent as well as several modified forms of the reagents with improved thermal stability\(^10\) are being reported. Now, chiral versions of the reagent are also known\(^11\) and the reagent has been extensively used in natural product syntheses.
A recent report shows an unexpected $N$-demethylation of oxymorphone and oxycodone-$N$-oxide using Burgess reagent to the corresponding oxazolidines providing a direct synthetic route to naltrexone, naloxone, and other antagonists from oxymorphone.\textsuperscript{9} Though several reagents like cyanogen bromide (von Braun reaction),\textsuperscript{12} ethyl chloroformate\textsuperscript{13} etc. are available for this transformation the conversion still remains a challenge in terms of efficiency and greenness of the reagent and conditions. The conversion of oxymorphone to naloxone and other analgesics include several steps, but use of Burgess reagent for $N$-demethylation reduces the entire sequence to three one-pot operations, proceeding in excellent overall yields.\textsuperscript{9} Burgess reagent shows unexpected reactivity with $N$-oxides and the results are interesting and applicable in synthesis of several heterocyclic compounds, particularly those with pharmaceutical applications.

Nitrones being $N$-substituted 1,3-dipolar systems can undergo cycloaddition\textsuperscript{14} reactions with a variety of carbon–carbon, carbon–nitrogen, carbon–sulphur, nitrogen–phosphorus multiple bonded systems to give various heterocyclic systems. Nitrones also find application in the synthesis of a wide range of natural product target types – from sugars and nucleoside analogues through
lactams to alkaloids and other nitrogen heterocyclic natural products, both bridgehead bicyclic and monocyclic systems. Thus, nitrones represent a useful substrate for fabrication of heterocyclic systems in modern synthetic chemistry. A dipolar homo \([3+2]\) cycloaddition reaction of nitrone with cyclopropane has also been reported.

Annulation reactions are important synthetic processes for constructing a wide variety of carbocyclic and heterocyclic frameworks. Among various annulations, \([3+2]\) annulation represents a breakthrough in the field of organic synthesis. In principle, reaction between a 1,3-dipole and 1,2-dipole should yield five membered ring structures via a formal \([3+2]\) annulation sequence. Huisgen and co-workers have refined this methodology for general application in organic synthesis. Several 1,3-dipoles were investigated by Huisgen. Invariably, the dipolarophile was a \(\pi\)-system.

### 3.2.1 Objectives

To the best of our knowledge, 1,3-dipolar addition to a \(\sigma\)-bond in acyclic systems is not reported in literature. A close examination of the structure of Burgess reagent reveals that it can act as a 1,2-dipole. In principle, any 1,3-dipole possessing significant nucleophilicity should react with Burgess reagent with elimination of triethylamine to give the corresponding product having a five-membered ring by a formal dipolar addition to a \(\sigma\)-bond. With a view to verify this hypothesis, we selected nitrones
as the dipole component since significant nucleophilic activity of nitrones has been well documented.\textsuperscript{21,22}

### 3.3 Results and Discussion

In the present investigation, we have exploited the 1,2-dipolar nature of Burgess reagent that should enable it to undergo annihilation reactions with 1,3-dipolar species possessing significant nucleophilicity such as nitrones. In this preliminary investigation, we examined the reaction of two ketonitrones \textit{viz.} $N$-(diphenylmethylene)aniline-$N$-oxide (2) and $N$-(9H-fluoren-9-ylidene)aniline-$N$-oxide (3), and two aldonitrones \textit{viz.} $N$-(benzylidene)aniline-$N$-oxide (4a) and $N$-(anthracen-9-ylmethylene)aniline-$N$-oxide (4b) with Burgess reagent. Structure of different nitrones employed in the present study is given in Figure 3.2. The required nitrones were prepared according to procedures reported in literature.\textsuperscript{23-27}

![Figure 3.2](image)

Burgess reagent was prepared from chlorosulphonyl isocyanate and triethylamine \textit{via} a two-step reaction\textsuperscript{28,29} (Scheme 3.1). In the first step, chlorosulphonyl isocyanate was treated with methanol to give methyl-(chlorosulphonyl)carbamate, which was
then reacted with triethylamine to give Burgess reagent (Methyl-$N$-(triethylammoniumsulphonyl)carbamate 1) in excellent overall yield. The reagent is oxidation and moisture sensitive, and needs to be stored under dry, oxygen free conditions at low temperature. A cyclic Burgess reagent was also prepared in a more convenient one-step process starting with an appropriate β-aminoalcohol\(^{30}\) (Scheme 3.1).

\[
\begin{align*}
\text{Cl} & \quad \text{SO}_3 \quad \text{N} \quad \text{C} \quad \text{O} \\
\text{CH}_3 \text{OH}, \text{C}_6\text{H}_6 & \quad 25 \text{ - } 30 \degree \text{C} \\
\text{Cl} & \quad \text{SO}_3 \quad \text{N} \quad \text{C} \quad \text{O} \\
\text{Et}_3\text{N} & \quad \text{O} \quad \text{OCH}_3 \\
\end{align*}
\]

Scheme 3.1

### 3.3.1 Reactions of $N$-(diphenylmethylene)aniline-$N$-oxide with Burgess reagent

1,3-Dipolar reaction between $N$-(diphenylmethylene)-aniline-$N$-oxide (2) and Burgess reagent (1) was conducted in a 1:3 molar ratio in dry dichloromethane at room temperature. The product 6 precipitated on adding hexane was separated, purified and further characterized by \(^1\)H NMR, \(^{13}\)C NMR, and MS (FAB). \(^1\)H NMR spectrum exhibited a characteristic signal of ester methyl proton at $\delta$ 3.40 ppm. Similarly \(^{13}\)C NMR spectrum exhibited
characteristic carbon signal at 52.70 ppm for ester methyl group and a signal at 163.4 ppm for carbonyl carbon. IR spectrum exhibited characteristic carbonyl absorption at 1713 cm$^{-1}$ and C=N absorption at 1616 cm$^{-1}$. These spectral characteristics support the presence of carbamate group in structure 6. MS (FAB) analysis gave molecular ion peak at 331.17 corresponding to the molecular formula C$_{21}$H$_{18}$O$_{2}$N$_{2}$. All data were consistent with the proposed structure 6. In a repeat run, careful work up of the reaction mixture under absolutely moisture free conditions afforded, in addition to 6, triethylamine-sulphur trioxide complex as colorless needles. Generation of 6 in the reaction between 2 and Burgess reagent mandates carbon to nitrogen aryl group migration. This rearrangement is reminiscent of a similar C to N aryl migration observed in the chlorosulphonyl isocyanate mediated transformation of nitrones.\textsuperscript{21,22} Though Burgess reagent is known to exhibit myriad reactivity, this is the first example for a C to N aryl migration overseen by this versatile reagent. We focused our attention on unraveling the mechanistic underpinnings, generality and possible synthetic utility of the novel C to N aryl migration discovered by us.
Scheme 3.2

Figure 3.3. $^1$H NMR spectrum of 6.
Structure of carbamate 6 was further confirmed by chemical transformations. Acid hydrolysis of 6 gave diphenylamine (7) along with 8 in quantitative yields. Structure of 8 was arrived at on the basis of spectral and analytical data. IR spectrum of 8 showed a peak at 3278 cm\(^{-1}\) attributable to NH stretch and two carbonyl stretching frequencies at 1778 and 1651 cm\(^{-1}\). In the \(^1\)H NMR spectrum of 8, a broad singlet (1H, D\(_2\)O-exchangeable) was observed at 8 8.2. A sharp singlet (3H) attributable to methoxy group was observed at 8 3.8 and three sets of multiplets (5H) attributable to a mono-substituted benzene ring were observed in the 8 7.4-7.8 range. Based on available data, this new compound was identified as methyl benzoylecarbamate (8).
3.3.2 Reaction of $N$-(9H-fluoren-9-ylidene)aniline-$N$-oxide (3) with Burgess reagent

In order to establish the generality of the novel C to N aryl migration observed by us, we examined the reaction of $N$-(9H-fluoren-9-ylidene)aniline-$N$-oxide 3 with Burgess reagent (Scheme 3.3). The product 10 precipitated on adding hexane was separated, purified and characterized on the basis of $^1$H NMR, $^{13}$C NMR, MS (FAB) data.

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**Scheme 3.3**
Figure 3.6. $^1$H NMR spectrum of compound 10.

Figure 3.7. $^{13}$C NMR spectrum of compound 10.
Figure 3.8. $^{13}$C DEPT-135 spectrum of 10.

Figure 3.9. $^{13}$C DEPT-90 spectrum of 10.

$^1$H NMR spectrum of 10 exhibited the characteristic peak of methyl proton at $\delta$ 3.32 ppm. The $^{13}$C NMR spectrum exhibited characteristic carbon peak at $\delta$ 52.20 ppm for ester methyl group.
and a peak at δ 160.0 ppm for carbonyl carbon. IR spectrum showed characteristic carbonyl absorption at 1683 cm⁻¹ and C=N absorption at 1640 cm⁻¹. Hence the spectral data support the presence of carbamate group in structure 10. MS (FAB) analysis gave molecular ion peak at 329.26 corresponding to the molecular formula C₂₁H₁₆N₂O₂. All data were consistent with the proposed structure 10 arising through a C to N aryl migration sequence. Structure of 10 was further confirmed on the basis of chemical transformations. Carbamate 10 on hydrolysis using oxalic acid adsorbed on silica gave compound 11 that was characterized on the basis of ¹H NMR, ¹³C NMR, and MS (FAB) data. IR spectrum of compound 11 shows C=O stretch at 1658 cm⁻¹, but the ester carbonyl at 1740 cm⁻¹ was missing indicating the cleavage of ester group on hydrolysis and existence of another carbonyl group. ¹H NMR spectrum exhibited characteristic signal for 13 aromatic hydrogens at δ 8.49-6.60 while the signal corresponding to carbamate methyl proton at δ 3.32 disappeared confirming the cleavage of carbamate group on hydrolysis. ¹³C NMR spectrum exhibited only one carbonyl signal at δ 160.60. FAB-MS analysis gave molecular ion peak at 272.4 which corresponds to the molecular formula C₁₉H₁₃NO. The above spectral characteristics suggest cleavage of ester group on hydrolysis and compound was identified as 5-phenylphenanthridin-6(5H)-one (11) confirming a C to N migration in this case well.
3.3.3 Reaction of \(N\)-(benzylidene)aniline-\(N\)-oxide with Burgess reagent

\(N\)-(benzylidene)aniline-\(N\)-oxide (4a) on reaction with Burgess reagent gave products arising through carbon to nitrogen...
phenyl migration. In the reactions of nitrone 4a, the carbamate intermediates 12a could not be isolated and the corresponding diarylamine 7 was the only isolable product (Scheme 3.4). The product obtained was identified by comparing melting point, TLC and IR spectra with those of authentic sample. Though we could not isolate the carbamate intermediate 12a, generation of diarylamine 7 is consistent with the C to N aryl migration pathway proposed by us. It may be noted that C to N hydrogen migration is an alternative possibility here. In order to verify this, we carried out careful GC-MS analysis of the reaction mixture. GC-MS analysis ruled out aniline generation and hence the C to N hydrogen migration possibility.

\[
\begin{align*}
\text{Ph} & \quad \text{H} \\
\text{C}=\text{N} & \quad \text{Ph} \\
\text{O}^- & \\
\text{Burgess Reagent} & \\
\text{CH}_2\text{Cl}_2 \, \text{(dry)} & \\
\text{RT, 3 h} & \\
4a & \quad 12a & \quad 7 \\
\text{COOMe} & \\
\text{HN Ph} & \\
\text{Hydrolysis} & \\
\text{HN Ph} & \\
12b & \quad 7a
\end{align*}
\]

Scheme 3.4

3.3.4 Reaction of N-(anthracen-9-ylmethylene)aniline-N-oxide with Burgess reagent

Difficulty in isolating intermediate 12a in the above reaction, prompted us to conduct a similar reaction with a different nitrone, N-(anthracen-9-ylmethylene)aniline-N-oxide (4b) and
Burgess reagent. However in this case also the diarylamine 13 (arising through the proposed C to N aryl migration pathway) was the only isolable product (Scheme 3.5).

We explored the possibility of isolating the carbamate intermediate in the reaction of nitrones 4a-b with a cyclic Burgess reagent. It was noted that the corresponding diarylamines 7 and 13 were the only isolable products in these reactions as well (Scheme 3.6). Though we were unsuccessful in isolating the carbamate intermediate, this experiment demonstrated that other variants of Burgess reagent also can initiate C to N aryl migration.

3.3.5 Conclusions

On the basis of the results obtained in the reaction of classic Burgess reagent with different nitrones and a novel cyclic variant of Burgess reagent with nitrones, we demonstrated that the novel C to N aryl migration in the Burgess reagent–nitrone reaction is a general reaction as well. Another striking feature of this
rearrangement is the remarkable migratory aptitude observed here. In the case of 2 and 3, migratory aptitude cannot be ascertained. However, with 4a, b, the aryl group migrates preferentially. Observed migratory aptitude can be explained in two different ways: i) it is the more electron rich group that migrates; ii) it is the syn group that migrates. Since only limited data is available at this stage, any conclusion made on this regard at best will be half-baked. Detailed analysis of migratory aptitude is presented in Chapter 4 of this thesis.

A plausible mechanism for the rearrangement can be proposed on the basis of available experimental evidences. Migration of the aryl group to the electron deficient nitrogen is the key step in the overall transformation. Such migrations are possible via different intermediates. Involvement of a cyclic intermediate promotes migration of the more electron rich aromatic ring. Conversely, migratory aptitude in a Beckmann type rearrangement should be controlled by geometrical constraints with the anti group migrating preferentially. Two possible mechanisms for the observed C to N aryl migration are presented in Scheme 3.7.

We have taken cues from available literature while presenting the two mechanistic possibilities. In Burgess reagent mediated dehydration of alcohols, the reagent first ionizes at low temperature in non-polar solvents to provide tight ion pairs,\textsuperscript{31-33} which then react with alcohol. In a similar way here also Burgess reagent undergoes ionization with the elimination of triethylamine part leaving a positive charge on sulphur. Then an attack of the
oxygen centre on dipole to sulphur followed with concomitant formation of C-N bond leads to a 1,2,3,5-oxathiadiazolidine intermediate $5b$. Subsequent elimination of the SO$_3$ group$^5$ with concomitant C to N aryl migration gives the carbamate product (Scheme 3.6). Loss of SO$_3$ from $5b$ generates a nitrenium ion intermediate setting the stage for a carbon to electron deficient nitrogen migration. Needless to mention, the more electron rich entity will migrate preferentially. Isolation of triethylamine-sulphur trioxide complex in certain cases endorses credence to this proposal.

Alternatively, a Beckmann type mechanism can also be proposed for the Burgess reagent mediated rearrangement of nitrones. Herein, nitrone attacks Burgess reagent in a nucleophilic fashion as in the earlier case to give the open-chain intermediate $5a$. Intermediate $5a$ has the right structural features to undergo Beckmann rearrangement such as an efficient nucleofuge as
N-substituent and an *anti* group that is set to migrate. However, migratory aptitude in this case should be controlled by stereoelectronic factors and only the *anti* group can migrate. On the contrary, in the case of aldonitrone-Burgess reagent reaction, we observed exclusive migration of the *syn* group. Hence, a Beckmann type rearrangement involving the open-chain intermediate 5a is improbable in this case. Intermediate 5a at best will serve as a precursor to 1,2,3,5-oxathiadiazolidine intermediate 5b (Scheme 3.7). Based on these considerations, we endorse the mechanism involving intermediate 5b to account for the observed C to N aryl migration with the more electron rich group migrating preferentially. Exclusive migration of the *syn* group, thus, is just a fortuitous event. A more detailed investigation of migratory aptitude in the nitrone-Burgess reagent reaction is presented in Chapter 4 of this thesis.

Hydrolysis of carbamate intermediates also provided interesting results. Generally, carbamates during hydrolysis are first converted to carbamic acid which then decarboxylates to afford the corresponding amines. Similarly, alkylidenecarbamates are expected to undergo hydrolysis to imines that might undergo further hydrolysis to give the corresponding ammonia derivative and carbonyl compound. But in the hydrolysis of Compound 6, C=N and ester group remain intact and only C-N bond is cleaved on hydrolysis and the products obtained are diphenylamine and benzoylcarbamate. On the other hand, hydrolysis of compound 10 apparently follows the expected hydrolysis pathway of carbamate.
The observed dichotomy, however, is easily explainable on the basis of the mechanism presented in Scheme 3.8. In the case of 10, intermediate 10b undergoes C-N bond cleavage to give a stable phenanthridinone product 11. Thus, both 6 and 10 undergo hydrolysis through the same mechanism; but with difference preference for CN bond cleavage. Product stability control is operating here. Furthermore, this type of hydrolysis occurs only under acidic conditions. Both 6 and 10 are inert towards bases.

Scheme 3.8

3.4 Experimental Section

3.4.1 General Techniques

General experimental techniques and instruments used are described in the experimental section of Chapter 2.

Yields reported are for compounds separated and purified in analytically pure form.

Required nitrones and Burgess reagent were prepared using the reported procedure as detailed in the experimental section of Chapter 2. All the reactions were carried out under nitrogen atmosphere.
3.4.2 General Procedure for Reaction of Nitrones 2 and 3 with Burgess reagent

Three equivalents of Burgess reagent were added under nitrogen to a well stirred solution of nitrone in dry dichloromethane at room temperature and the stirring was continued for 3h. The progress of the reaction was monitored by TLC using ethyl acetate:hexane (8:92). The intermediate carbamate product was isolated from the reaction mixture by adding hexane. Addition of hexane to this reaction mixture gave two layers - a brown coloured bottom layer containing the decomposition products of Burgess reagent and the upper layer containing the precipitated carbamate product which was carefully decanted. The filtrate was allowed to settle and washed repeatedly with hexane to get the colorless precipitate of carbamate in pure form it was further characterized by IR, $^1$H NMR, $^{13}$C NMR, MS (FAB) analysis. Triethylamine-sulphur trioxide complex separated as colourless needles could be isolated under carefully controlled conditions.

3.4.3 Reaction of Nitrone 4a with Burgess Reagent.

Nitrone 4a was dissolved in dry dichloromethane and after purging the reaction mixture with N$_2$, three equivalent Burgess reagent were added and mechanically stirred for 3h. at room temperature. Column chromatography (silica) of reaction mixture using hexane-ethyl acetate (9:1) gave diphenylamine (68% yield). The product obtained was identified by comparing melting point, TLC and IR spectra with those of authentic sample.$^{34}$ GC-MS data indicates exclusive formation of a single amine product with
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retention time 13.02, major peak at 169 corresponds to diphenylamine.

3.4.4 Reaction of Nitrone 4b with Burgess Reagent.

Nitrone 4b was stirred with three equivalent Burgess reagent for 3h. at room temperature. Column chromatography (silica) of reaction mixture using hexane-ethyl acetate (17:3) gave diarylamine 13 (60% yield). The product obtained was identified by melting point, TLC and IR spectra, ^1^H NMR and ESI (MS) data.

3.4.5 Reaction of Nitrones 4a and 4b with Cyclic Burgess Reagent (CBR)

Insitu generated CBR^{30} was reacted with 3 equivalents of nitrones 4a,b for 3h. at room temperature. The solvent removed under reduced pressure, and the residue was purified by column chromatography on silica gel using a mixture of hexane-ethyl acetate (9:1) as eluent gave diarylamine 7 and 13. We repeated the reaction of nitrones 4a, b with cyclic Burgess reagent with a view to isolate the carbamate intermediate. However, the corresponding diarylamines 7 and 13 were the only isolable products in these reactions as well.

3.4.6 Hydrolysis of Carbamate 6

Hydrolysis of carbamate 6 was achieved by acid medium like dilute HCl. After hydrolysis, excess acid was neutralized with sodium bicarbonate solution and the products were isolated by solvent extraction using hexane.
3.4.7 Hydrolysis of Carbamate 10

Hydrolysis of carbamate 10 was achieved by acid medium like oxalic acid adsorbed on silica gel. After hydrolysis, excess acid was neutralized with sodium bicarbonate solution and the products were isolated by solvent extraction.

3.4.8 Spectral and Analytical Data of Novel Compounds

3.4.8.1 Compound 6

Yield 2.60 g (78%); mp 172 °C

IR ν_{max} (KBr): 1713, 1616, 1577, 1490, 1372, 1236, 1195, 1118 cm^{-1}

^{1}H NMR (500 MHz, CDCl₃): δ 7.41-7.39(m, 2H), 7.26-7.20(m, 7H), 7.14-7.10(m, 6H), 3.4(s, 3H)

^{13}C NMR (125 MHz, CDCl₃): δ 163.44, 162.58, 144.25, 134.49, 130.10, 129.04, 128.76, 128.29, 127.51, 126.16, 52.74

FAB-MS: m/z calculated for C₂₁H₁₈N₂O₂: 330.36 (M⁺); found: m/z 331.17 (M⁺+1)

Elemental analysis calculated for C₂₁H₁₈N₂O₂: C, 76.34; H, 5.49; N, 8.48; found: C, 74.55; H, 3.98; N, 9.57.
3.4.8.2 Compound 8

Yield 1.6 g (64%); mp 116 °C

IR $\nu_{\text{max}}$ (KBr): 3278, 1778, 1751, 1529, 1209, 1018, 702 cm$^{-1}$

$^1$H NMR (500 MHz, CDCl$_3$): $\delta$ 8.09(1H, s), 7.83-7.81(2H, td), 7.61-7.58(1H, tt), 7.50-7.47(2H, m), 3.87(3H, s)

FAB-MS: m/z calculated for C$_9$H$_9$NO$_3$: 179.16 ($M^+$); found: m/z 180.12 ($M^+$+1)

Elemental analysis calculated for C$_9$H$_9$NO$_3$: C, 60.33; H, 5.06; N, 7.82, O, 26.79%; found: C, 61.05; H, 3.08; N, 5.96, O, 24.32%

3.4.8.3 Compound 10

Yield 2.68 g (82%); mp 149 °C

IR $\nu_{\text{max}}$ (KBr): 3055, 2949, 1683, 1640, 1488, 1357, 1191, 1119, 1096 cm$^{-1}$

$^1$H NMR (500 MHz, CDCl$_3$): $\delta$ 8.45-8.43(dd, 1H), 8.30-8.27(dd, 1H), 8.26-8.25(t, 1H), 7.78-7.74(m, 1H), 7.62-7.58(dt, 2H), 7.57-7.53(m, 2H), 7.42-7.41(m, 2H), 7.27-7.24(m, 2H), 6.58-6.56(m, 1H), 3.32(s, 3H)

$^{13}$C NMR (125 MHz, CDCl$_3$): $\delta$ 160.04, 148.53, 139.01, 138.16, 132.81, 132.52, 130.57, 130.21, 129.88, 129.30, 129.23, 129.16, 128.32, 125.32, 123.13, 122.84, 121.81, 119.70, 117.00, 52.26

FAB-MS: m/z calculated for C$_{21}$H$_{16}$N$_2$O$_2$: 328.35 ($M^+$); found: m/z 329.26 ($M^+$+1)

Elemental analysis calculated for C$_{21}$H$_{16}$N$_2$O$_2$: C, 76.81; H, 4.91; N, 8.53; found: C, 73.23; H, 5.07; N, 7.68
3.4.8.4 Compound \( \text{II} \)

Yield 1.97 g (73%); mp 202 °C

**IR** \( \nu_{\text{max}} \) (KBr): 3066, 1658, 1604, 1486, 1324, 1261, 810, 747 cm\(^{-1}\)

**\(^1\)H NMR** (500 MHz, CDCl\(_3\)) \( \delta \) 8.49-8.47 (q, 1H), 8.26-8.22 (q, 1H), 8.22-8.21 (t, 1H), 7.74-7.71 (dt, 1H), 7.55-7.51 (m, 3H), 7.47-7.44 (m, 1H), 7.26-7.25 (t, 1H), 7.24 (d, 1H), 7.23-7.20 (m, 2H), 6.62-6.60 (m, 1H)

**\(^13\)C NMR** (125 MHz, CDCl\(_3\)): 160.69, 138.14, 137.26, 132.98, 131.81, 129.18, 128.06, 127.99, 127.76, 127.11, 124.83, 121.96, 121.63, 120.76, 118.00, 116.00

**FAB-MS**: \( m/z \) calculated for C\(_{19}\)H\(_{13}\)NO: 271.30 (\( M^+ \)); found: \( m/z \) 272.40 (\( M^++1 \))

 Elemental analysis calculated for C\(_{21}\)H\(_{16}\)N\(_2\)O\(_2\): C, 84.11; H, 4.83; N, 5.16; found: C, 81.92; H, 5.12; N, 3.76
3.5. References