Microwave-assisted synthesis of benzoxazinediones under solvent-free conditions

Juan I. Sarmiento-Sánchez\textsuperscript{a} \textsuperscript{a}, Adrián Ochoa-Terán\textsuperscript{b} \textsuperscript{b}, Lorenzo A. Picos-Corrales\textsuperscript{c}, Lorenzo U. Osuna-Martínez\textsuperscript{c}, Julio Montes-Ávila\textsuperscript{d} and Pedro Bastidas-Bastidas\textsuperscript{d}

\textsuperscript{a}Facultad de Ingeniería Culiacán, Universidad Autónoma de Sinaloa, Culiacán, Mexico; \textsuperscript{b}Centro de Graduados e Investigación en Química, Instituto Tecnológico de Tijuana, Tijuana, Mexico; \textsuperscript{c}Facultad de Ciencias Químico-Biológicas, Universidad Autónoma de Sinaloa, Culiacán, Mexico; \textsuperscript{d}Laboratorio de Análisis de Residuos de Plaguicidas, Centro de Investigación en Alimentación y Desarrollo A.C., Culiacán, Mexico

ABSTRACT

Benzoxazinediones exhibit potential as versatile synthons in the synthesis of wide variety of heterocyclic compounds with biological activity. In this work, an efficient and eco-friendly one-step synthesis of benzoxazine-2,4-diones from phthalic anhydrides derivatives and trimethylsilyl azide using the microwave technique was developed and compared with conventional heating. Microwave irradiation plays a critical role in driving the reaction and providing access to products and/or regioisomers not available from conventional heating. Thus, the regioselectivity of the reaction may be modulated by the irradiation time. Depending on the method employed the benzoxazinediones were isolated with yields in the range of 30–90%.

1. Introduction

Considering their reported biological activities as potent inhibitor of hepatitis C virus (1), butyrylcholinesterase inhibitor (2) and antiallergic (3), antitumor (4), antipsyhotic (5), antileishmanial (6) and antimycobacterial agents (7), benzoxazine-2,4-diones are potential pharmacophores. Also, benzoxazine-2,4-diones are important precursors in the synthesis of several potent compounds as antipsyhotic, anti-inflammatory, bronchodilators and antimicrobial drugs (8). Thus, several paths for the synthesis of benzoxazinediones from anthranilic acid derivatives (9), isatin (10), allenamides (11), carbamoylbenzoic acid (12), 2-hydroxybenzoic acid (13), anacardic acid (14), sodium azide with sulfuric acid (15) and phthalimide (16) have been reported. In recent years, the reaction of trimethylsilyl azide (TMSA) with five-membered heterocyclic anhydride has been extensively used (17). In general, these processes involve several reaction steps and employ expensive, non-commercial or dangerous reagents. Besides the use of non-eco-friendly conventional heating conditions and long reaction times (12–18 hours), some by-products are detected and difficult work-up is necessary to isolate the products. These are some drawbacks for these methodologies. Therefore, new processes are desirable in order to obtain these compounds in better conditions.

On the other hand, the use of microwave technique in organic synthesis is reported in a large number of publications. The elegance of the reaction, high yields, short time span, simplified work-up procedure and eco-
friendly conditions are the main advantages of the method. Using microwaves with a proper power control and temperature is more efficient than conventional heating (18). Thus, the microwave technique was used in the synthesis of some heterocyclic compounds, such as oxadiazoles (19), 1,2,3-triazoles (20), quinolines (21), coumarin (22), tetrahydroquinolines (23), benzofuran-2-carboxamide (24) and sulfonamides (25), among others.

Currently, our research is focused on the development of simple, one-pot synthesis of small heterocyclic compounds. Recently, we reported the synthesis of small heterocyclic compounds with biological activities (26). Herein, we reported an easy one-step synthesis of benzoxazine-2,4-diones under microwave irradiation and eco-friendly conditions.

2. Experimental design

All reagents were purchased in the highest quality available and were used without further purification. Nuclear Magnetic Resonance of $^1$H and $^{13}$C spectra were recorded on a Varian Mercury 200 MHz or Bruker 400 MHz Spectrometer in DMSO-$d_6$ or CD$_2$OD with tetramethysilane as internal standard, chemical shifts ($\delta$) in ppm and coupling constants ($J$) in Hz. Mass spectra were obtained with a GC Varian Titan 4000 using positive chemical ionization, and the intensities were reported as a relative percentage to the base signal after the corresponding m/z value. Infrared spectra were recorded on a Cary 660 series FTIR-ATR spectrophotometer. Melting points were obtained on a Stuart photometer. Melting points were obtained on a Stuart apparatus model SMP30 and were uncorrected; the value is reported as an average of three separate experiments. All reactions described herein were conducted in Pyrex tubes sealed with a silicon septum in a single-mode microwave reactor (Discover-SP model 909150 equipped with an Explorer 12 hybrid model 909150, with 725 W of maximum power, CEM Corp.) with 100 W of initial power.

2.2. General procedure for the synthesis of benzoxazine-2,4-diones under microwave irradiation

To a phthalic anhydride derivative (1.0 mmol) was added TMSA (1.0 equiv.). The mixture was placed in a microwave reactor vessel (10 mL) and heated at 120°C for 8 minutes, and then cooled to room temperature. The solid was washed with diethyl ether (2 × 3 mL) to obtain the benzoxazine-2,4-diones in high purity.

2.2.1. 2H-3,1-benzoxazine-2,4(1H)-dione (6a)

NMR data are similar to those previously reported (27). White solid. Yield 61%. $^1$H-NMR (400 MHz, CD$_2$OD): $\delta$ 7.94 (1H, dd, $J=7.3, 1.5$ Hz, ArH), 7.65 (1H, td, $J=7.8, 1.6$ Hz, ArH), 7.21 (1H, m, ArH), 7.08 (1H, m, ArH). $^{13}$C-NMR (101 MHz, CD$_2$OD): $\delta$ 161.3, 149.6, 142.8, 138.3, 130.7, 125.2, 116.5, 111.8. GC-MS (CI$^+$) m/z: 164 [M+H]$^+$. 

2.2.2. 2H-pyrido[3,2-d][1,3]oxazine-2,4(1H)-dione (6b)

NMR data are similar to those previously reported (5, 28). White solid. Yield 50%. $^1$H-NMR (400 MHz, CD$_2$OD): $\delta$ 8.60 (1H, dd, $J=4.9, 1.7$ Hz, ArH), 8.22 (1H, dd, $J=8.0, 1.6$ Hz, ArH), 7.54 (1H, dd, $J=8.1, 4.9$ Hz, ArH). $^{13}$C-NMR (101 MHz, CD$_2$OD): $\delta$ 169.6, 168.3, 152.8, 152.2, 139.9, 128.5, 126.6. GC-MS (Cl$^+$) m/z: 165 [M+H]$^+$. 

2.2.3. 2H-pyrazino[2,3-d][1,3]oxazine-2,4(1H)-dione (6c)

NMR data are similar to those previously reported (29). Pale brown solid. Yield 58%. $^1$H-NMR (200 MHz, DMSO-$d_6$): $\delta$ 11.74 (1H, br s, NH), 8.95 (1H, d, $J=4.0$ Hz, ArH), 8.32 (1H, d, $J=4.0$ Hz, ArH). $^{13}$C-NMR (50 MHz, DMSO-$d_6$): $\delta$ 163.5, 153.8, 140.1, 134.3, 132.2, 123.5. GC-MS (Cl$^+$) m/z: 166 [M+H]$^+$. 

2.2.4. 8-fluoro-2H-benzo[d][1,3]oxazine-2,4(1H)-dione (6d)

NMR data are similar to those previously reported (30). White solid. Yield 40%. $^1$H-NMR (400 MHz, DMSO-$d_6$): $\delta$ 11.91 (1H, br s, NH), 7.75 (1H, dt, $J=7.8, 1.0$ Hz, ArH), 7.67 (1H, ddd, $J=10.8, 8.1, 1.2$ Hz, ArH), 7.23 (1H, td, $J=8.1, 4.7$ Hz, ArH). $^{13}$C-NMR (101 MHz, CD$_2$OD): $\delta$ 112.84 (d, $J=2.93$ Hz, C), 122.20 (d, $J=16.87$ Hz, CH), 123.33 (d, $J=6.60$ Hz, CH), 124.47 (d, $J=3.67$ Hz, CH), 130.22 (d, $J=14.67$ Hz, C), 146.58 (CO–N), 148.78 (d, $J=247.22$ Hz, CO–F), 158.95 (d, $J=3.67$ Hz, CO–O). GC-MS (Cl$^+$) m/z: 170 [M–CO$_2$+MeOH+H]$^+$. 

2.1. General procedure for the synthesis of benzoxazine-2,4-diones under conventional heating

Phthalic anhydrides (2 mmol) and TMSA (1.05 equiv.) were stirred in tetrahydrofuran (THF) (20 mL) into a Schlenk flask (80 mL) for 17 hours at reflux. The resulting solution was concentrated under vacuum to dryness. The solid formed was washed with diethyl ether (3 × 5 mL) to obtain the benzoxazine-2,4-diones in high purity.
2.2.5. 6,7-dichloro-2H-benzo[d][1,3]oxazine-2,4(1H)-dione (6e)
NMR data are similar to those previously reported (31). White solid. Yield 58%. $^1$H-NMR (400 MHz, DMSO-d$_6$): $\delta$ 11.93 (1H, br s, NH), 8.08 (1H, s, ArH), 7.31 (1H, d, $J = 0.5$ Hz, ArH). $^{13}$C-NMR (101 MHz, DMSO-d$_6$): $\delta$ 158.2, 146.5, 140.8, 138.9, 129.9, 125.4, 116.8, 111.1. GC-MS (CI$^+$) m/z: 220 [M+CO$_2$+MeOH+H]$^+$. 161.1, 149.8, 148.8, 142.8, 140.6, 136.3, 130.4, 126.5, 123.2, 116.4, 113.0, 111.1, 109.1, 36.7, 35.6, 31.6, 31.3. GC-MS (CI$^+$) m/z: 208 [M−CO$_2$+MeOH+H]$^+$.

2.2.6. 5,6,7,8-tetrahydro-2H-benzo[d][1,3]oxazine-2,4(1H)-dione (6f)
NMR data are similar to those previously reported (32). White solid. Yield 53%. $^1$H-NMR (400 MHz, CD$_2$OD): $\delta$ 2.42 (2H, m, CH$_2$), 2.32 (2H, m, CH$_2$), 1.80 (4H, m, 2xCH$_2$). $^{13}$C-NMR (101 MHz, CD$_2$OD): $\delta$ 162.7, 154.9, 150.6, 103.4, 27.1, 22.7, 22.3. GC-MS (CI$^+$) m/z: 156 [M−CO$_2$+MeOH+H]$^+$. 161.7, 159.4, 158.0 (d, $JC$ = 254.6 Hz), 159.9, 159.2, 158.0 (d, $JC$ = 241.4 Hz), 147.0, 146.8, 143.8, 143.6, 132.3, 124.9, 117.6, 117.5, 114.1, 111.7, 111.4, 101.7. GC-MS (CI$^+$) m/z: 170 [M−CO$_2$+MeOH+H]$^+$. 208 [M+H]$^+$, 166 [M−CO$_2$+MeOH+H]$^+$.

2.2.7. 6,7-dihydro-2H-[1,4]dithiino[2,3-d][1,3]oxazine-2,4(1H)-dione (6g)
Pale yellow solid. Yield 47%. m.p. 200–202°C. $^1$H-NMR (400 MHz, DMSO-d$_6$): $\delta$ 12.07 (brs, 1H), 3.48 (m, 2H), 3.20 (m, 2H). $^{13}$C-NMR (101 MHz, DMSO-d$_6$): $\delta$ 156.4, 149.0, 146.1, 94.7, 26.8, 25.5. FT-IR (ATR): 1749, 1677, 1509. GC-MS (CI$^+$) m/z: 192 [M−CO$_2$+MeOH+H]$^+$. HRMS (EI) calculated for C$_8$H$_8$NO$_2$S$_2$ (M$^+$) 202.9711, found 202.9730.

2.2.8. 4,5-dichloro-2H-1,3-oxazine-2,6(3H)-dione (6h)
NMR data are similar to those previously reported (33). White solid. Yield 33%. $^{13}$C-NMR (101 MHz, CD$_2$OD): $\delta$ 156.8, 147.4, 147.1, 103.5.

2.2.9. 4-methyl-2H-1,3-oxazine-2,6(3H)-dione (6i)
NMR data are similar to those previously reported (33(b), 34). White solid. Yield 30%. $^{1}$H-NMR (400 MHz, CD$_2$OD): $\delta$ 5.48 (1H, q, $J = 1.0$ Hz, CH), 2.17 (3H, d, $J = 1.0$ Hz, CH$_3$). $^{13}$C-NMR (101 MHz, CD$_2$OD): $\delta$ 161.7, 159.4, 150.9, 94.4, 18.6. GC-MS (CI$^+$) m/z: 116 [M−CO$_2$+MeOH+H]$^+$. 161.3, 161.2, 150.5, 149.8, 142.9, 139.4, 135.4, 130.5, 130.1, 126.5, 116.4, 111.5, 109.1, 22.2, 20.7. GC-MS (CI$^+$) m/z: 178 [M+H]$^+$, 166 [M−CO$_2$+MeOH+H]$^+$. 161.1, 149.8, 148.8, 142.8, 140.6, 136.3, 130.4, 126.5, 123.2, 116.4, 113.0, 111.1, 109.1, 36.7, 35.6, 31.6, 31.3. GC-MS (CI$^+$) m/z: 208 [M−CO$_2$+MeOH+H]$^+$. 161.1, 149.8, 148.8, 142.8, 140.6, 136.3, 130.4, 126.5, 123.2, 116.4, 113.0, 111.1, 109.1, 36.7, 35.6, 31.6, 31.3. GC-MS (CI$^+$) m/z: 208 [M−CO$_2$+MeOH+H]$^+$. 161.0, 149.8, 148.8, 142.8, 140.6, 136.3, 130.4, 126.5, 123.2, 116.4, 113.0, 111.1, 109.1, 36.7, 35.6, 31.6, 31.3. GC-MS (CI$^+$) m/z: 208 [M−CO$_2$+MeOH+H]$^+$.
1.6 Hz, ArH), 8.24 (1H, dd, J = 7.8, 1.7 Hz, ArH), 7.62 (1 H, dd, J = 7.8, 4.9 Hz, ArH) for 2H-pyrido[3,2-d][1,3]oxazine-2,4(1H)-dione. GC-MS (Cl⁺) m/z: 153 [M-CO₂+MeOH+H]⁺.

3. Results and discussion

As depicted in Figure 1, the synthesis of the 1H-benzoxazine-2,4-diones involves the formation of the acylazide intermediate 2 mechanistically in the first step, followed by a subsequent Curtius rearrangement to generate an isocyanate 4 and an intramolecular cyclization to provide the benzoxazine-2,4-diones 6.

In this regard, the synthesis of benzoxazine-2,4-diones compounds started with the optimization of reaction conditions. For this purpose, the synthesis of product 6a using THF as solvent under microwave irradiation was studied. First, in accordance with conventional thermal conditions, the temperature was set at 80°C and then, varying the time of the reaction from 4 to 20 minutes, the reactions were monitoring by TLC until the reaction of the phthalic anhydride was complete. It was found that compound 6a is formed with poor yield. Therefore, the temperature was increased to 100°C and 120°C at same reaction time. From these experiments, the optimal conditions were established at 120°C and 8 minutes, reaching a total conversion of the phthalic anhydride. The mixture reaction was evaporated at vacuum and the product was precipitated with diethyl ether, to get the corresponding benzoxazine-2,4-dione. Subsequently, the reaction with different solvents using optimal reaction conditions in the synthesis of compound 6a was performed (Table 1).

Due to the fast heating in microwave irradiation, halogenated solvents of low molecular weight (e.g. DCM or chloroform) were not used; an explosion risk in the formation of di- or tri-azido compounds is always present. Moreover, it has been reported that the use of TMSA under microwave irradiation is safe to handling (40).

The best options for conventional heating and microwave irradiation were THF and solvent-free condition. Although in the solvent-free condition yields were lower, the work-up was easier. Due to our interest in green chemistry, solvent-free conditions were mainly exploited. In this approach, all the products were precipitated by the addition of diethyl ether, avoiding the need to purify the final compounds via other means (i.e. column chromatography).

Washburne (41) reported that oxazine-2,6-diones were obtained as silylated products. However, silylated compounds were not detected in this work. Based on the observed results, similar yields under conventional heating and microwave irradiation were obtained (Table 2).

Chakravorty et al. (42) reported the synthesis of benzoxazinediones on alumina and silica powder in 10 minutes at 540 W using a domestic microwave oven with moderate yield. Interestingly, in solvent-free conditions benzoxazinediones were not obtained. These

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Table 1. Solvents study of compound 6a under µW irradiation.

| Entry | Solvent      | Yield (%) |
|-------|--------------|-----------|
| 1     | Neat         | 61        |
| 2     | H₂O          | 22        |
| 3     | THF          | 90        |
| 4     | 1,4-dioxane  | 13        |
| 5     | Benzene      | 33        |
| 6     | Acetonitrile | 78        |
types of experiments are conducted with an increased risk to the user, heating efficiency can vary drastically between different positions of the load when small loads are heated, and the yields are usually more difficult to optimize and reproduce (43). Our study is more reproducible because a single-mode microwave reactor was used. Furthermore, this study has shown a lower time reaction and potency, and eco-friendly conditions with moderate yield in the synthesis of benzoxazinediones derivatives.

In the other hand, Nagasaka (44) reported isomeric mixture of benzoxazinediones from the reaction of non-symmetric phthalic anhydrides with TMSA. We observed this same effect for phthalic anhydrides substituted in 3- or 4-position (Table 3) due to that the azide nucleophilic attack can take place on either of the anhydride carboxyls. The regioselectivity of the benzoxazine-2,4-diones resulted from a combination of electronic and steric effects, as reported by McMillan (45).

The regioselectivity in solvent-free conditions may be modulated by the irradiation time (Table 3). When reaction was performed at 8 minutes 6k was obtained as a mixture of 6- and 7-isomer with a regioisomeric ratio of 49:51, respectively. When the reaction was performed at 30 minutes the isomeric ratio changed to 38:62. Similar result was observed with 6l, at 8 minutes the isomeric ratio was 28:72 and at 30 minutes isomeric ratio changed to 33:67 for 6- and 7-isomer.

When 3-fluorophthalic anhydride was used under conventional heating only 8-isomer 6d was detected, while under microwave irradiation the regioisomeric ratio for 5- and 8-isomer was 75:25 (compound 6m). It is clear that the conventional heating condition favors in each case thermodynamic regioisomers. On the other hand, as microwave accelerates the reaction reaching to activation energy in less time than conventional heating, the regioisomeric ratio could be affected purely by thermal/kinetic factors. This is more evident in the experiments made without solvent in a time reaction of 8 minutes. In particular, 6b and 6i were isolated as

### Table 2. Yields of benzoxazine-2,4-diones under conventional heating and μW irradiation conditions.

| Product | R1, R2, X | C.H. | μW<sub>ref</sub>, 8 min | μW<sub>μW</sub>, 8 min | μW<sub>μW</sub>, 30 min |
|---------|-----------|------|----------------|--------------------|-----------------|
| 6a      | R1 = H, R2 = H, X = CH | 79:21 (66) | 65:35 (50) | 52:48 (51) | 28:72 |
| 6b      | R1 = H, R2 = H, X = N | 49:51 (61) | 54:46 (80) | 49:51 (50) | 38:62 |
| 6c      | R1 = F, R2 = H, X = CH | 80:20 (56) | 53:47 (52) | 28:72 (48) | 33:67 |
| 6d      | R1 = tert-butyl, R2 = H, X = CH | 0:100 (40) | 78:22 (65) | 75:25 (53) | 85:15 |

Notes: C.H. – conventional heating; nf – compound was not isolated.

### Table 3. Isomers ratio of benzoxazinediones synthesized.

| Product | R1, R2, X | C.H. | μW<sub>μW</sub>, 8 min | μW<sub>μW</sub>, 8 min | μW<sub>μW</sub>, 30 min |
|---------|-----------|------|----------------|--------------------|-----------------|
| 6j      | R1 = tert-butyl, R2 = H, X = CH | 79:21 (66) | 65:35 (50) | 52:48 (51) | 28:72 |
| 6k      | R1 = H, R2 = H, X = CH | 49:51 (61) | 54:46 (80) | 49:51 (50) | 38:62 |
| 6l      | R1 = F, R2 = H, X = CH | 80:20 (56) | 53:47 (52) | 28:72 (48) | 33:67 |
| 6m      | R1 = H, R2 = F, X = CH | 0:100 (40) | 78:22 (65) | 75:25 (53) | 85:15 |
| 6n      | R1 = H, R2 = H, X = N | 52:48 (91) | 100:0 (90) | 100:0 (50) | 69:31 |

Notes: C.H. – conventional heating.

<sup>a</sup> Isomers ratio was obtained by 1H-NMR of isolated mixtures. The isolated yields of isomeric mixtures are given in bracket.

<sup>b</sup> The isomers mixture was separated by the chromatographic column.
pure regiosomeric products using neat microwave conditions.

The reaction between citraconic anhydride and TMSA under conventional heating provided a 70:30 isomeric ratio for 4- and 5-isomers (46). Under solvent-free microwave conditions only 4-isomer was generated at 8 minutes (compound 6i).

In summary, the synthesis of benzoxazine-2,4-diones by microwave irradiation from phthalic anhydrides and TMSA has important advantages: (i) lower reaction time than conventional heating (8 minutes vs. 17 hours) and (ii) products are obtained in better or comparable yields. This methodology is amenable for a combinatorial chemistry approach, and it could be used to prepare targeted libraries to identify compounds that exhibit potential as versatile synthons in the synthesis of wide variety of heterocyclic compounds with potential biological activity.

4. Conclusion

In this work we reported a convenient and easy microwave-assisted solvent-free synthesis approach for benzoxazine-2,4-diones from phthalic anhydrides. This approach affords desired products in moderate to good yields quickly, using a simple work-up procedure, avoiding the need to purify the final products. Also, it was found that the microwave irradiation dramatically reduces the reaction time from hours to several minutes, which is an important factor on the viability of new synthetic methods. Also under microwave conditions it was possible to access products that in conventional heating are not obtained. Further, the microwave irradiation plays a critical role in driving the reaction providing access to a regioisomer not available from conventional heating.

Disclosure statement

No potential conflict of interest was reported by the authors.

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ORCiD

Juan I. Sarmiento-Sánchez  http://orcid.org/0000-0001-8961-4436
Adrián Ochoa-Terán  http://orcid.org/0000-0002-3746-3960

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