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ORIGINAL ARTICLE

Microwave irradiation: A facile, scalable and convenient method for synthesis of *N*-phthaloylamino acids

Hassan M. Al-Hazimi [a,*], Ayman El-Faham [b], Mohamed Ghazzali [a], Khalid Al-Farhan [a]

[a] *Chemistry Department, College of Science, King Saud University, P.O. Box 2455, Riyadh 11451, Saudi Arabia*
[b] *Sabbatical Leave, University of Alexandria, Faculty of Science, Chemistry Department, P.O. Box 426 Ibrahimia, 12321 Alexandria, Egypt*

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X-ray crystallography

Abstract We describe herein a fast and rapid technique for preparation of *N*-phthaloyl amino acids under microwave irradiation. The microwave methodology is rapid, convenient, proceeds under mild conditions, and gives a better yield (81–98%) and high purity of the title compounds. The spectral data as well as the X-ray analysis established the structure of the prepared *N*-protected amino acids.

1. Introduction

Phthalimide derivatives are used as anesthetics (Settimo et al., 1989), DNA cleaving agents (Brana and Ramos, 2001; Brana et al., 2003), tumericidals (Kirhenbaum et al., 1994), optical brighteners (Schellhammer and Schroeder, 1975; Stewart, 1981) and as dyes (Wurthner et al., 2004; Ramachandram and Samanta, 1997). They are used as fluoroprobes (Barros et al., 1997) and have interesting photophysical properties (Arhens et al., 2004; Yan et al., 2005). Pyromellitic dianhydride is an analogous compound, which undergoes facile polymerization reaction with diamines to give amide or imides containing polymers. It also has relevance to nanomaterials (Holman et al., 2003). Compounds analogous to phthalimides self assemble to form nano-tube like structures (Kimizuka et al., 1995; Kishikawa et al., 1999; Barooah et al., 2003). The *N*-phthaloyl derivatives have vast host–guest chemistry (Davies et al., 1984; Jaber et al., 1995; Sandhu and Sandhu, 1995; El-Asmy et al., 1994). The *N*-phthaloylglycine is a simple *N*-protected amino acid and has interesting supramolecular structural features on formation of metal complexes (Abdel-Rahman, 2001). Because of the great importance of amino acids in physiological and pharmacological events, and because biological interactions (Juaristi, 1997) and the important of the *N*-protecting amino in peptide synthesis, it is worthwhile to find a simple, moderate, clean and convenient method for preparation of *N*-phthaloyl amino acid derivatives which are usually prepared by conventional method (Bedair et al., 2006). The present work represented the synthesis of

*Corresponding author. Tel.: +966 1 4657900; fax: +966 1 4657992.
E-mail addresses: mghazzali@ksu.edu.sa (H.M. Al-Hazimi), aelfaham@ksu.edu.sa (A. El-Faham).

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N-phthaloyl amino acids under solventless mild conditions using microwave irradiation as a rapid, convenient method which give excellent yields and high purity of the products than conventional method.

2. Experimental

Melting points are determined on an electrothermal's IA9000 series digital capillary melting point apparatus. IR spectra were run (KBr discs) on Perkin Elmer FT spectrophotometer 1000. ^{1}H and ^{13}C NMR spectra were recorded on a JEOL ECP 400 NMR spectrometer operating at 400 MHz in $CDCl_3$ unless otherwise stated with TMS as internal standard. DEPT and HETCOR experiments were recorded on 500 MHz instrument (Brucker, J.F.B. 288) at King Saud University. Chemical shifts are given in δ ppm and coupling constants (J) are given in Hz. The used Microwave oven is Model No. NN-CD997s/NN-CD987W. Solid structure of **3b** was determined by single-crystal X-ray diffraction spectroscopy.

*2.1. General procedure for the preparation of (**3a**–l)*

A mixture of equimolar amount of phthalic anhydride **1** (10 mmol) and **2a–l** (10 mmol) in small aliquot of glacial acetic acid was placed in a 100 mL beaker covered with a watch glass and was then irradiated microwave (1000 w) for 20 min except for the acid **2k**, it was 15 min. The cold reaction mixture was treated with methanol; the solid product (**3a–l**) was filtered, dried and recrystallized from ethanol.

*2.1.1. 2-(1,3-Dioxoisoindolin-2-yl)acetic acid (**3a**)*

White crystals, m.p. 156–157 °C; IR (cm^{-1}): 1733 (C═O), 1665 (C═O, anhydride); ^{1}H NMR: δ 4.32 (s, 2H), 7.86–7.89 (m, 2H), 7.91–7.93 (dd, 2H), 13.27 (s, OH); ^{13}C NMR: δ 39.5, 123.86 (d, 2C), 131.88 (s, 2C), 135.27 (d, 2C), 167.70 (2C═O), 169.37 (C═O).

*2.1.2. 3-(1,3-Dioxoisoindolin-2-yl)propanoic acid (**3b**)*

White crystals, m.p. 110–112 °C; IR (cm^{-1}): 1735 (C═O), 1670 (C═O, anhydride); ^{1}H NMR: δ 2.79 (*t*, J = 7.4 Hz, 2H), 3.99 (*t*, J = 7.4 Hz, 2H), 7.70–7.73 (dd, 2H), 7.82–7.88 (dd, 2H), 12.8 (s, OH); ^{13}C NMR: δ 32.56 (CH_2–N), 33.45 (CH_2), 123.49 (d, 2C), 131.21 (s, 2C), 134.18 (d, 2C), 168.04 (2C═O), 176.1 (C═O).

*2.1.3. 2-(1,3-Dioxoisoindolin-2-yl)propanoic acid (**3c**)*

White crystals, m.p. 106 °C; IR (cm^{-1}): 1740 (C═O), 1668 (C═O, anhydride); ^{1}H NMR: δ 1.67 (d, J = 7.32 Hz, 3H), 4.99 (d, J = 7.32 Hz, 1H), 7.67–7.71 (dd, 2H), 7.79–7.82 (dd, 2H), 10.67 (s, OH); ^{13}C NMR: δ 15.07 (CH_3), 47.32 (CH), 123.67 (d, 2C), 131.78 (s, 2C), 134.36 (d, 2C), 167.50 (2C═O), 175.61 (C═O).

*2.1.4. 2-(1,3-Dioxoisoindolin-2-yl)-3-methylbutanoic acid (**3d**)*

White crystals, m.p. 78 °C; IR (cm^{-1}): 1736 (C═O), 1668 (C═O, anhydride); ^{1}H NMR($CDCl_3$): δ ^{1}H NMR: δ 0.86 & 1.13 (each d, J = 7.3 Hz, 2 × 3H), 2.04 (m, 1H), 4.57 (d, J = 7.4 Hz, 1H), 7.68–7.77 (dd, 2H), 7.81–7.83 (dd, 2H), 12.82 (s, OH); ^{13}C NMR: δ 19.53 & 19.57 (2 × CH_3), 28.42 (CH), 57.94 (CH), 123.66 (d, 2C), 131.59 (s, 2C), 134.48 (d, 2C), 167.91 (2C═O), 174.09 (C═O).

*2.1.5. (DL-)-2-(1,3-dioxoisoindolin-2-yl)-3-methylbutanoic acid (**3e**)*

White crystals, m.p. 84 °C; IR (cm^{-1}): 1736 (C═O), 1665 (C═O, anhydride); ^{1}H NMR($CDCl_3$): δ ^{1}H NMR: δ 0.81 & 0.82 (each d, 2 × 3H (D-isomer), 1.05 & 1.07 (each d, 2 × 3H (L-isomer), 2.51–2.58 (m, 1H), 4.45 (d, J = 8.0 Hz, 1H), 7.88–7.91 (m, 4H), 13.07 (brs, OH); ^{13}C NMR: δ 19.73 & 21.3 (2 × CH_3), 28.42 (CH), 57.42 (CH), 123.95 (d, 2C), 131.46 (s, 2C), 135.41 (d, 2C), 167.99 (2C═O), 170.39 (C═O).

*2.1.6. 2-(1,3-Dioxoisoindolin-2-yl)-2-methylpropanoic acid (**3f**)*

White powder, m.p. 114–116 °C; IR (cm^{-1}): 1739(C═O), 1664 (C═O, anhydride); ^{1}H NMR: δ 1.72 (s, 2 × CH_3), 7.78–7.82 (m, 4H), 12.98 (s, OH); ^{13}C NMR: δ 24.77 (2CH_3), 60.28 (s), 123.43 (d, 2C), 131.65 (s, 2C), 135.13 (d, 2C), 168.49 (2C═O), 174.46 (C═O).

*2.1.7. 2-(1,3-Dioxoisoindolin-2-yl)-3-(methylthio)propanoic acid (**3g**)*

off white prisms, m.p. 82–84 °C; IR (cm^{-1}): 1736 (C═O), 1668 (C═O, anhydride); ^{1}H NMR: δ 2.01 (s, CH_3), 2.37 (q, 2H), 2.52 (m, 2H), 4.93 (*t*, CH) 7.88–7.91 (m, 4H), 13.3 (s, OH); ^{13}C NMR: δ 14.95 (CH_3), 28.11, 30.61, 51.12, 123.85 (d, 2C), 131.80 (s, 2C), 135.24 (d, 2C), 168.00 (2C═O), 170.97 (C═O).

*2.1.8. 2-(1,3-Dioxoisoindolin-2-yl)-3-phenylpropanoic acid (**3h**)*

White prisms, m.p. 156–157 °C; IR (cm^{-1}): 1731 (C═O), 1663 (C═O, anhydride); ^{1}H NMR: δ 3.58 (d, J = 8.8 Hz, 2H), 5.22 (*t*, J = 8.8 Hz, 1H), 7.66 (m, 2H), 7.76 (m, 2H), 9.94 (s, OH); ^{13}C NMR: δ 34.35, 53.17, 123.66 (d, 2C), 127.04, 128.7 (d, 2C), 128.9 (d, 2C), 131.51 (s, 2C), 134.29 (d, 2C), 167.53 (2C═O), 174.75 (C═O).

*2.1.9. 2-(1,3-Dioxoisoindolin-2-yl)-3-(4-hydroxyphenyl)-propanoic acid (**3i**)*

White plates, m.p. 257–259 °C; IR (cm^{-1}): 1730 (C═O), 1663 (C═O, anhydride); ^{1}H NMR ($CDCl_3$): δ 3.49 (d, J = 8.8 Hz, 2H), 5.14 (*t*, J = 8.8 Hz, 1H), 7.67 (m, 2H), 7.75 (m, 2H), 10.76, 9.96 (each s, 2 × OH); ^{13}C NMR: δ 33.66, 53.12, 115.56 (d, 2C), 123.64 (d, 2C), 130.10 (d, 2C), 131.51 (s, 2C), 134.27 (d, 2C), 154.47, 167.54 (2C═O), 173.17 (C═O).

*2.1.10. 2-(1,3-Dioxoisoindolin-2-yl)benzoic acid (**3j**)*

White crystals, m.p. 198–200 °C; IR (cm^{-1}): 1709 (C═O), 1658 (C═O, anhydride); ^{1}H NMR: δ 7.56 (d, J = 8.8 Hz, 1H), 8.06 (d, J = 8.8 Hz, 1H), 7.63 (*t*, J = 7.4 Hz, 1H), 7.77 (*t*, J = 7.4 Hz, 1H), 7.91–7.95 (m, 2H), 7.98–8.01 (m, 2H), 13.16 (s, OH); ^{13}C NMR: 121.16 (d, 2C), 132.46 (d, 2C), 126.88, 126.91, 128.31, 128.68, 129.12 (s, C), 129.38 (s, 2C), 130.65, 164.78 (2C═O), 163.76 (C═O).

*2.1.11. 4-(1,3-Dioxoisoindolin-2-yl)benzoic acid (**3k**)*

White plates, m.p. 270–272 °C; IR (cm^{-1}): 1705 (C═O), 1657 (C═O, anhydride); ^{1}H NMR: δ 7.61 (d, J = 8.8 Hz, 2H), 7.90–7.94 (m, 2H), 7.97–8.01 (m, 2H), 8.04 (d, J = 8.8 Hz, 2H), 13.3 (s, OH); ^{13}C NMR: 124.11 (d, 2C), 127.56 (d, 2C), 130.39 (d, 2C), 130.55 (s), 132.05 (s, 2C), 135.39 (d, 2C), 136.38 (s), 167.29 (2C═O), 167.21 (C═O).

*2.1.12. 2-(4-(1,3-Dioxoisoindolin-2-yl)phenyl)acetic acid (**3l**)*
Off white prisms, m.p. 180–181 °C; IR (cm^{-1}): 1718 (C═O), 1662 (C═O, anhydride); ^1H NMR: δ 3.89 (s, CH_2), 7.40–7.49 (m, 4H), 7.77–7.80 (m, 2H), 7.94–7.96 (m, 2H), 9.68 (s, OH); 13C NMR: 123.86 (d, 2C), 126.73 (d, 2C), 130.21 (d, 2C), 130.55(s), 131.81 (s, 2C), 134.50 (d, 2C), (d, 2C), 136.38 (s), 168.50 (2C═O), 168.1 (C═O).

3. Result and discussion

Several parameters have been used to deal with protecting group during peptide-coupling reactions. A key issue is the use of appropriate *N*-protecting groups, such as the carbamate [*tert*-butyloxycarbonyl (Boc, **4**)] (Mckay and Albertson, 1957), benzyloxycarbonyl (Cbz, Z, **5**), (Bergmann and Zervas, 1932), 9-fluorenylmethy-loxcarbonyl (Fmoc, **6**) (Carpino, 1987), or the recent type of base-sensitive amino protecting groups 1,1-dioxobenzo[*b*]thiophene-2-ylmethyloxycarbonyl (Bsmoc,**7**) (Carpino and Philbin, 1999), 2-(*tert*-butylsulfonyl)-2-propyl-oxycarbonyl (Bspoc, **8a**) (Carpino et al., 1999), and 2-methyl-sulfonyl-3-phenyl-1-prop-2-enyloxycarbonyl (Mspoc, **8b**) (Carpino and Mansour, 1999), (Fig. 1). It rarely mentioned about the *N*-Phthaloyl group in peptide synthesis, but it is very useful for other application and synthesis (Burger et al., 1998), because it could be easily removed by aqueous HBr (Hughes and Griffiths, 1997) or hydrazine in ethanol (Chen et al., 1998) and this might not be a suitable condition for longer peptide synthesis.

Although the wide application of microwave-mediated synthesis of organic compounds, only very little work appeared about *N*-phthaloylamino acids. Inspection in the literature cites two works where phthalic anhydride couples with amino acids under microwave-mediated conditions. So, in 1995 Bose et al. (1990) reported that *N*-phthaloylamino acids could be synthesized within few minutes in good yields using microwave irradiation in DMF. Vidal et al. (2000) reported examination of the latter technique for synthesis of phthalimides, among them phthaloylglycine which was obtained in 90 and 81% yields in xylenes and DMF, respectively. However, at the end of irradiation of these syntheses, the products usually extracted with acetone. Here we re-examined this non-thermal methodology in acetic acid for the preparation of *N*-phthaloyl-

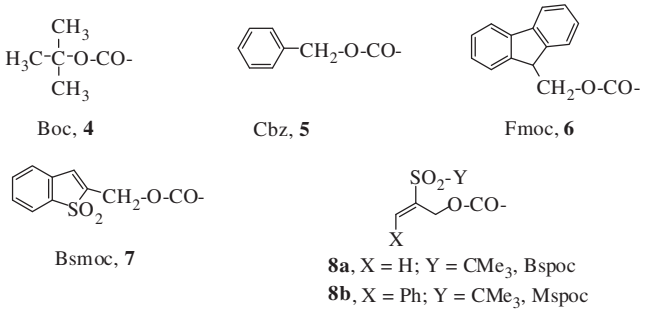
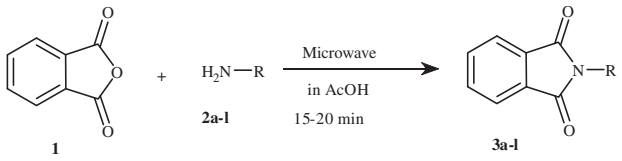


Figure 1 Structure of protecting group.



R	yield (%)
a -CH_2COOH	95
b -CH_2CH_2COOH	100
c -$CH(CH_3)COOH$	92
d -CH(isopr.)COOH	81
e (DL-)-CH(isopr.)COOH	89
f -$C(CH_3)_2COOH$	86
g -$CH(CH_2CH_2SCH_3)COOH$	88
h -$CH(CH_2C_6H_5)COOH$	82
i -$CH(CH_2C_6H_4$-*p*-$OH)COOH$	94
j 2-C_6H_4COOH	85
k 4-C_6H_4COOH	83
l-$C_6H_4CH_2COOH$	86

Scheme 1

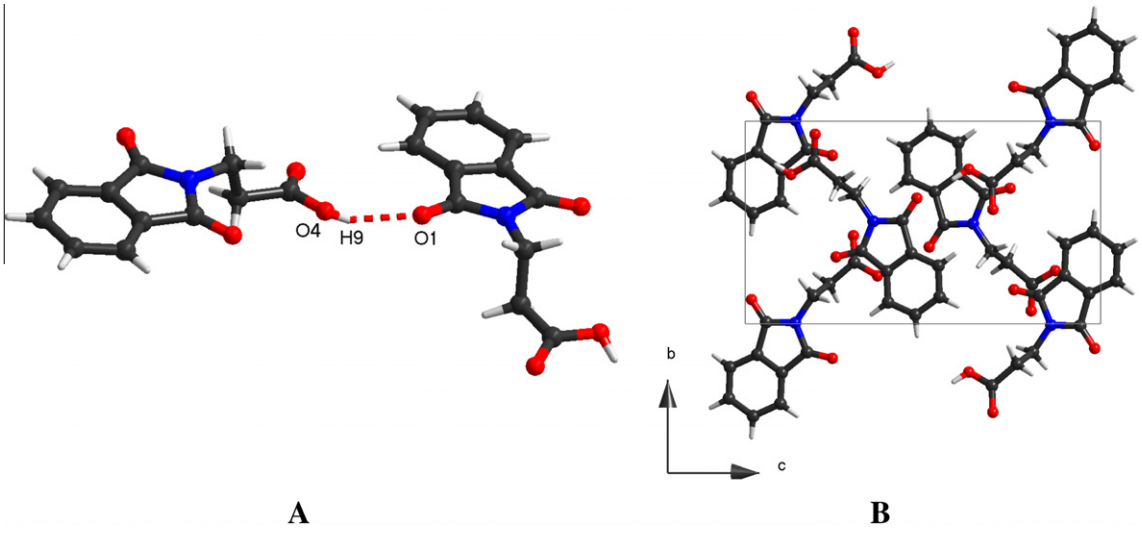


Figure 2 X-ray structure of *N*-phthaloyl-*β*-alanine (**3b**).

Table 1 Crystal data and structure refinement for HF34.

Crystal data	**3b**
Empirical formula	$C_{11}H_9NO_4$
Formula weight	219.19
Temperature (K)	293(2)
Wavelength (Å)	0.71075
Crystal system	Monoclinic
Space group	$P2_1/c$
Unit cell dimensions	
a (Å)	7.101(2)
b (Å)	8.979(2)
c (Å)	15.952(4)
α (°)	90
β (°)	98.839(8)
γ (°)	90
Volume (Å^3)	1005.1(5)
Z	4
Density (calculated) (Mg/^{3})	1.449
Absorption coefficient (mm^{-1})	0.112
F(000)	456
Crystal size (mm^3)	$0.35 \times 0.20 \times 0.20$
Theta range for data collection	3.44–27.48 (°)
Index ranges	$-9 \leqslant h \leqslant 9$, $-11 \leqslant k \leqslant 11$, $-1 \leqslant l \leqslant 20$
Reflections collected	2301
Independent reflections	2301 [R(int) = 0.0000]
Completeness to theta = 27.48°	99.9%
Max. and min. transmission	0.9779 and 0.9618
Refinement method	Full-matrix least-squares on $F2$
Data/restraints/parameters	2301/0/148
Goodness-of-fit on $F2$	1.013
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.1141$, $wR_2 = 0.2637$
R indices (all data)	$R_1 = 0.1517$, $wR_2 = 0.2954$
Largest diff. peak and hole (e Å^{-3})	0.729 and −0.636

Table 2 Atomic coordinates (×104) and equivalent isotropic displacement parameters (Å$^2 \times 10^3$) for HF34. U(eq.) is defined as one third of the trace of the orthogonalized U^{IJ} tensor.

	x	y	z	U (eq.)
O(1)	2663(6)	6207(4)	4827(2)	56(1)
N(1)	3153(6)	5057(4)	3595(2)	42(1)
C(2)	2276(7)	2867(6)	5427(3)	48(1)
O(2)	3369(6)	3310(4)	2553(2)	65(1)
C(1)	2224(8)	1332(6)	5464(3)	59(1)
O(3)	1500(7)	9354(4)	1962(2)	73(1)
C(6)	2466(9)	457(6)	4778(3)	62(2)
O(4)	2291(6)	7370(4)	1281(2)	60(1)
C(5)	2778(8)	1072(5)	4014(3)	55(1)
C(4)	2837(7)	2599(5)	3978(3)	43(1)
C(3)	2581(6)	3489(5)	4669(3)	38(1)
C(7)	2781(6)	5084(5)	4418(3)	41(1)
C(8)	3154(7)	3607(5)	3268(3)	44(1)
C(9)	3398(7)	6380(5)	3084(3)	48(1)
C(10)	1521(8)	6969(6)	2641(3)	55(1)
C(11)	1782(7)	8040(5)	1940(3)	46(1)

Table 3 Anisotropic displacement parameters (Å$^2 \times 10^3$) for HF34. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2\ a^{*2}U^{11} + \cdots + 2\ h\ k\ a^*\ b^*\ U^{12}]$.

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	84(3)	44(2)	41(2)	−7(1)	14(2)	−5(2)
N(1)	52(2)	41(2)	33(2)	5(2)	10(2)	−1(2)
C(2)	54(3)	59(3)	31(2)	3(2)	8(2)	−4(2)
O(2)	99(3)	65(2)	33(2)	0(2)	22(2)	16(2)
C(1)	74(4)	57(3)	46(3)	17(2)	10(3)	−9(3)
O(3)	125(4)	41(2)	59(2)	6(2)	31(2)	15(2)
C(6)	80(4)	38(3)	66(3)	11(2)	4(3)	−4(3)
O(4)	94(3)	46(2)	42(2)	4(2)	20(2)	7(2)
C(5)	77(4)	39(2)	49(3)	3(2)	9(2)	6(2)
C(4)	47(3)	47(2)	34(2)	3(2)	6(2)	4(2)
C(3)	40(2)	43(2)	32(2)	3(2)	4(2)	0(2)
C(7)	42(2)	45(2)	34(2)	2(2)	2(2)	−2(2)
C(8)	49(3)	47(3)	37(2)	6(2)	9(2)	8(2)
C(9)	55(3)	47(3)	41(2)	12(2)	7(2)	−4(2)
C(10)	61(3)	54(3)	52(3)	17(2)	16(2)	2(2)
C(11)	49(3)	48(3)	41(2)	7(2)	6(2)	1(2)

Table 4 Hydrogen coordinates (×10^4) and isotropic displacement parameters (Å$^2 \times 10^3$) for HF34.

	x	y	z	U (eq.)
H(2)	2112	3453	5891	58
H(1)	2020	875	5966	71
H(6)	2418	−573	4829	75
H(9)	2247	7963	887	89
H(5)	2938	482	3551	66
H(4)	4201	6131	2665	58
H(3)	4036	7151	3449	58
H(7)	864	7472	3051	66
H(8)	732	6143	2406	66

glycine and other ten representatives. Therefore, we have applied this procedure for the synthesis of target compounds which proved to give excellent yields and in short time reactions (15–20 min), while using conventional method takes 10–14 h reflux in acetic acid or DMF. The synthetic pathway depicted in Scheme 1 outlines the chemistry of the present study. Full characterization of the synthesized compounds was based on the spectral data, particularly ^{1}H and ^{13}C NMR. The assignments of various protons and carbons were achieved by running DEPT-135 and HETCOR Experiments. Thus, IR spectra of **3a–l** exhibited two stretching bands in the regions 1658–1670 cm^{-1} and 1705–1740 cm^{-1}, attributed to the amidic carbonyl and acidic one, respectively.

^{1}H and ^{13}C NMR of **3a–l** confirmed their structures, and the assignments of all protons and carbons (see experimental) were done by the help of HETCOR experiments while the assignment of the aliphatic protons in **3a–h** was further confirmed by DEPT-135. The used method proved to be safe in term of racemization. This was confirmed by the synthesis of authentic sample from *N*-phthaloyl-(L)-valine and *N*-phthaloyl-(DL)-valine. The ^{1}H NMR not detected any of the D-valine in the pure sample of L-isomer (L-at δ 1.05 & 1.07 ppm and D-at δ 0.81 & 0.82 ppm). The structures of the prepared phthalimides were unambiguously verified by single X-ray crystallography of *N*-phthaloyl-β-alanine (**3b**) (Fig. 2). The *N*-phthaloyl glycine generally crystallizes in a monomeric form with a molecule of water and it has an extended hydrogen bonded

structure. Recently the dimeric structure was confirmed by X-ray crystallography (Barooah et al., 2006). From the X-ray crystallography of *N*-phthaloyl-*β*-alanine (**3b**) it was observed that (**3b**) exist as a dimer due to the H-bond between the OH of the carboxyl group and the carbonyl group of the second phthaloyl group (–CO–O4-H–O1═C–, Fig. 2A) (Tables 1–4). The dimeric step-like structure with two phthaloyl units disposed in a *trans* manner should be able to accommodate planar molecules to form guest-host compound (Fig. 2B).

4. Conclusion

Microwave irradiation is safe in term of racemization, fast and rapid technique for preparation of *N*-phthaloyl amino acids. The X-ray crystallography established the structure of the prepared *N*-protected amino acids and most of the *N*-phthaloyl amino acids exist as a dimer. Further investigation for the x-ray crystallography and the biological activity of the prepared compounds are under investigation in our laboratory.

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References

Abdel-Rahman, L.H., 2001. Transition Met. Chem. 26, 412.
Arhens, M.J., Sinks, L.E., Rybtchinski, B., Liu, W., Jones, B.A., Giamo, J.M., Gusev, A.V., Goshe, A.J., Tiede, D.M., Wasielewski, M.R., 2004. J. Am. Chem. Soc. 126, 8284.
Barooah, N., Sarma, R.J., Baruah, J.B., 2003. Cryst. Growth Des. 3, 639.
Barooah, N., Sarma, R.J., Batsanov, A.S.R.J., Baruah, J.B., 2006. J. Mol. Struct. 791, 122.
Barros, T.C., Brochsztain, S., Toscano, V.G., Filho, P.B., Politi, M.J., Photochem, J., 1997. Photobiol. A: Chem. 111, 97.
Bedair, A.H., Ali, F.M., El-Agrody, A.H., Eid, F.A., El-Nassag, M.A.A., El-Sherbeny, G., 2006. Acta Pharm. 56, 273.
Bergmann, M., Zervas, L., 1932. Ber. Dtsch. Chem. Ges. 65, 1192.
Bose, A.K., Manhas, M.S., Ghosh, M., Raju, V.S., Tabei, K., Urbanczyk-lipkowska, Z., 1990. Herocycles 30, 741.
Brana, M.F., Cacho, M., Ramos, A., Domingues, M.T., Pozuelo, J.M., Abradelo, C., Rey-Stolle, M.F., Yuste, M., Carrasco, C., Bailly, C., 2003. Org. Biomol. Chem. 1, 648.
Brana, M.F., Ramos, A., 2001. Curr. Med. Chem. 1, 237.
Burger, K., Mutze, K., Hollweek, W., Koksch, B., 1998. Tetrahedron 54, 5915.
Carpino, L.A., 1987. Acc. Chem. Res. 20, 401.
Carpino, L.A., Mansour, E.M.E., 1999. J. Org. Chem. 64, 8399.
Carpino, L.A., Philbin, M., 1999. J. Org. Chem. 64, 4315.
Carpino, L.A., Philbin, M., Ismail, M., Truran, G.A., Mansour, E.M.E., Iguchi, S., Ionescu, D., El-Faham, A., Riemer, C., Warrass, R., Weiss, M.S., 1999. J. Am. Chem. Soc. 119, 9915.
Chen, C., Wilcoxen, K., McCarthy, J.R., 1998. Tetrahedron Lett. 39, 8229.
Davies, J.E.D., Finocchiaro, P., Herbstien, F.H., Atwood, J.L., Davies, J.E.D., MacNicol, D.D. (Eds.), 1984. Inclusion Compounds, vol. 2. Academic Press, New York, pp. 407–453.
El-Asmy, A.A., Hafez, M.A., Saad, E.M., Taha, F.I., 1994. Transition. Met. Chem. 19, 603.
Holman, M.W., Liu, R., Adams, D.M., 2003. J. Am. Chem. Soc. 125, 12649.
Hughes, J.M., Griffiths, D.V., 1997. Tetrahedron 53, 17815.
Jaber, F., Charbonnier, F., Faure, R., 1995. Acta Crystallogr., Sect. C: Cryst. Struct. Commun. C51, 1765.
Juaristi, E., 1997. An. QuõÂm. Int. Ed. 93, 135.
Kimizuka, N., Kawasaki, T., Hirata, K., Kunitake, T., 1995. J. Am. Chem. Soc. 117, 6360.
Kirhenbaum, M.R., Chen, S.F., Behrens, C.H., Papp, L.M., Stafford, M.M., Sun, J.-H., Behrens, D.L., Fredericks J.R., Polkus, S.T., Sipple, P., Patten, A.D., Dexter, D., Seitz, S.P., Gross, J.L., 1994. Cancer Res. 54, 2199.
Kishikawa, K., Tsubokura, S., Kohmoto, S., Yamamoto, M., Yamguchi, K., 1999. J. Org. Chem. 64, 7568.
McKay, F.C., Albertson, N.F., 1957. J. Am. Chem. Soc. 79, 4686.
Ramachandram, B., Samanta, A., 1997. Chem. Commun. 1037.
Sandhu, G.K., Sandhu Jr., S.S., 1995. Synth. React. Inorg. Met.-Org. Chem. 25, 93.
Schellhammer, C.W., Schroeder, J., 1975. Angew. Chem., Int. Ed. Engl. 14, 665.
Settimo, A.S., Primotiore, G., Ferrarini, P.L., Barili, P.L., Tellini, N., Bianchini, P., 1989. Eur. J. Med. Chem. 24, 263.
Stewart, W.W., 1981. Nature 292, 17.
Vidal, T., Alain, P., Loupy, A., Gedye, R.N., 2000. Tetrahedron 56, 5473.
Wurthner, F., Chen, Z., Hoeben, F.J.M., Osswald, P., You, C.-C., Jonkheijm, P., Herrikhuyzen, J.V., Schenning, A.P.H., van der Schoot, P.P.A.M., Meijer, E.W., Beckers, E.H.A., Meskers, S.C.J., Janssen, R.A.J., 2004. J. Am. Chem. Soc. 126, 10611.
Yan, P., Chowdhury, A., Holman, M.W., Adams, D.M., 2005. J. Phys. Chem. 109, 724.

Further reading

Barooah, N., Karmakar, A., Sarma, R.J., Baruah, J.B., 2006. Inorg. Chem. Commun. 9, 1251.
Carpino, L.A., Ismail, M., Truran, G.A., Mansour, E.M.E., Iguchi, S., Ionescu, D., El-Faham, A., Riemer, C., Warrass, R., 1999. J. Org. Chem. 64, 4324.
Deka, K., Barooah, N., Sarma, R.J., Baruah, J., 2007. J. Mol. Struct. 827, 44.
Eliel, E., Wilen, S.H., Mander, L.N., 1994. Stereochemistry of Organic Compounds. Wiley, New York.
Juaristi, E., 1991. Introduction to Stereochemistry and Conformational Analysis. Wiley, New York.
SHELXTL Reference Manual, Version 5.1, Brucher AXS, Analytical X-ray system, Inc., Madision, WI, 1997.