

Enhanced One-Pot Microwave Efficient Synthesis of Some Pyrrolidine-2, 5-Diones, Butenamides and their Sulfonamide Derivatives Versus Conventional Thermal Heating Technique

Shadia M. Abdallah

Chemistry Department, University College of Women for Arts, Science, and Education, Ain Shams University, Cairo, Egypt.

DUE TO the highly important medical applications and wide biological activities of pyrrolidines, butenamides, and their sulfonamide derivatives, such as bacteriocides, fungicides, and insecticides, compounds (4-42) were synthesized from condensation of α , β -unsaturated anhydrides (1-3), with the corresponding amines, using either enhancing microwave irradiation heating or the conventional thermal heating technique in order to compare between their efficiency.

Keywords: Microwave, Synthesis, Condensation, Butenamides, Pyrrolidines and Sulfonamides.

The microwave irradiation comparable to conventional thermal heating proves to generate rapid intense heating with consequent significant reduction in reaction time, enhancement of yield and purity of products, in addition to its friendly environmental effect, known as green chemistry. It proves to be a convenient method of heating, comparable to conventional thermal technique. Moreover, some reactions that do not take place by conventional thermal heating technique or give very low yields, can be accomplished in high yields under microwave irradiation technique^(1,2). Amides, imides, and their derivatives are known to have wide medical applications, biological activities, and as starting materials in industry⁽³⁻⁷⁾. The aim of this work is the comparison between the two techniques and to synthesis amides and imides derivatives in high and pure yield.

Results and Discussion

Solvent-free microwave irradiation technique

Solvent-free microwave irradiation of 2-phenylmethylenebutanedioic anhydride (1), 2-(4-methoxyphenylmethylene)butanedioic anhydride (2), or 2-(methylphenylmethylene)butanedioic anhydride (3)^(8,9) with different N-substituted 4-aminobenzenesulfonamides (a-g)⁽¹⁰⁾, gave the corresponding N-(N'-substituted benzenesulfonamido)-3-carboxy-4-aryl-3-butenamide derivatives, and/or N-(N'-substituted benzenesulfonamido)-3-benzylidene-pyrrolidine-2,5-dione derivatives. The microwave irradiation of anhydride (3) with some amines (h-m) gave the corresponding 3-carboxy-3-butenamide derivatives and/or pyrrolidine-2, 5-dione derivatives.

Email: shadiamabdallah@hotmail.com

The results obtained show that under solvent-free microwave irradiation heating of anhydrides (1 and 2) with N-phenyl-4-aminobenzenesulfonamide (a), N-(4-methylphenyl)-4-aminobenzenesulfonamide (b), N-(4-methoxyphenyl)-4-aminobenzenesulfonamide (c), and N-(4-chlorophenyl)-4-aminobenzenesulfonamide (d)⁷ gave pyrrolidine-2,5-diones (4-7, 12-14 and 16), whereas anhydride (3) gave separable mixtures from the corresponding butenamides (21, 23, 25, and 27) and pyrrolidine-2,5-diones (22, 24, 26, and 28). This can be attributed to the steric effect exerted by the methyl group in anhydride (3).

However, microwave irradiation of anhydrides (1-3) with either N-(4-nitrophenyl)-4-aminobenzenesulfonamide (e) or N-benzyl-4-aminobenzenesulfonamide (g), gave the corresponding butenamides (8, 11, 17, 20, 29, and 32). This is ascribed to low nucleophilicity of the amido nitrogen atom towards further intramolecular nucleophilic attack on the carbonyl carbon to give pyrrolidine-2,5-dione, due to the presence of the nitro group, and the steric effect exerted by the sp³ methylene group in the benzyl moiety, respectively.

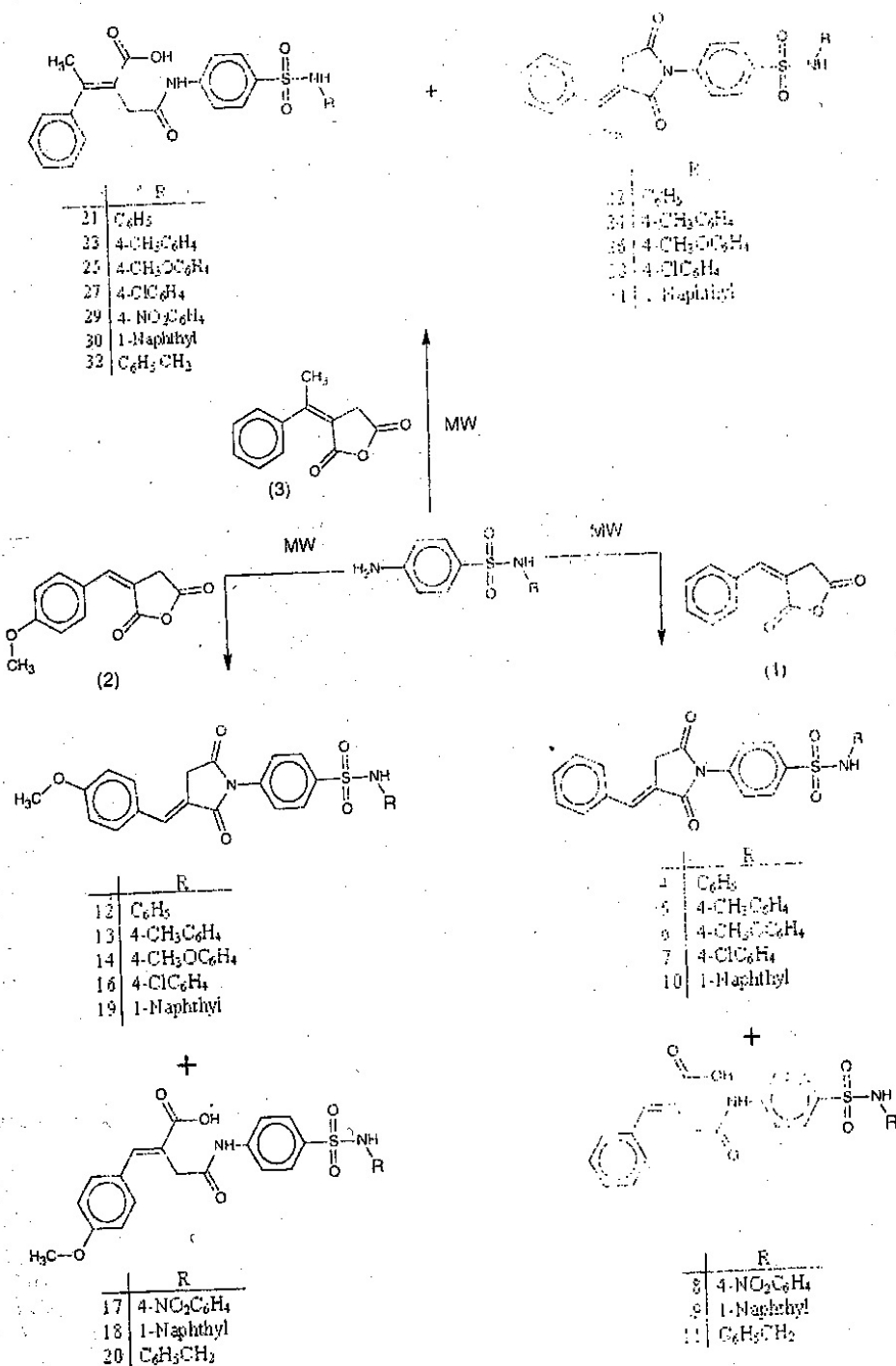
On the other hand, N-(1-naphthyl)-4-aminobenzenesulfonamide (f) gave with anhydrides (1-3), the respective separable mixtures from butenamides (9, 18, and 30) and pyrrolidine-2,5-diones (10, 19, and 31). The formation of the cyclic pyrrolidine-2,5-diones, irrespective to the low reactivity of the amido nitrogen atom in butenamides to form cyclic compounds can be attributed to the coplanarity of the naphthyl ring that facilitates the formation of the cyclic compounds (Scheme 1).

The results obtained from microwave irradiation of anhydride (3) with amines; 4-methylaniline (h), 4-methoxyaniline (i), 4-chloroaniline (j), 4-nitroaniline (k), 1-naphthylamine (l), and benzylamine (m), show that amines (h-k) gave the corresponding pyrrolidine-2,5-diones (34, 36, 38, and 40), whereas amine (l), gave a separable mixture from butenamide (41) and pyrrolidine-2,5-dione (42), and amine (m) gave butenamide (43) as an only product (Scheme 2).

Comparison between the results obtained from reaction of anhydride (3) with N-substituted 4-aminobenzenesulfonamides (b-g) versus amines (h-m); shows that the presence of the benzenesulfonamido moiety decreases the reactivity of the amido nitrogen atom towards intramolecular nucleophilic attack to form the cyclic compounds.

Conventional thermal heating technique

Condensation of anhydrides (1-3) with different 4-amino-N-phenyl-benzenesulfonamide derivatives (a-g) (1:2 mole) in ethanol for at least 4 hr gave the corresponding N-(N'-substituted benzenesulfonamido)-3-carboxy-4-aryl-3-butenamide derivatives, and/or N-(N'-substituted benzenesulfonamido)-3-benzylidene-pyrrolidine-2,5-dione derivatives. Thermal heating of anhydride (3) with amines (h-m) gave the corresponding 3-carboxy-3-butenamide derivatives and/or pyrrolidine-2,5-dione derivatives (Scheme 3).



Scheme 1.

Scheme 2.

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The condensation of anhydrides (1 and 2) with amines (a-c) using conventional thermal heating technique, gave pyrrolidine-2, 5-diones (4-6) and (12-14), respectively. With amine (d), thermal condensation of anhydride (1) gave pyrrolidine-2, 5-dione (7), whereas anhydride (2) gave a separable mixture from butenamide (15) and pyrrolidine-2, 5-dione (16). This can be attributed to the presence of mesomeric effect exerted by the methoxy group in anhydride (2) which decreases the susceptibility of the carbonyl carbon towards the anchimeric attack by the amido nitrogen atom.

On the other hand, the formation of only butenamides (8, 9) and (17, 18), from the thermal heating of anhydrides (1 and 2), respectively, with amines (e-f), can be ascribed to the low nucleophilicity of the amido nitrogen atom in the butenamides (8, 9, 17, and 18), whereas with amine (g), irrespective to the higher nucleophilicity of the amido nitrogen, its reactivity towards cyclization decreases due to the steric effect exerted by the sp^3 methylene group.

However, the formation of butenamides (21, 23, 25, 27, 29, 30, 32, 33, 35, 37, 39, 41, and 43) as only products, resulted from thermal condensation of anhydride (3) with amines (a-m) can be attributed to the steric effect exerted by the tetrahedral sp^3 methyl carbon in anhydride (3), which decreases the susceptibility of the carbonyl carbon to be attacked by the amido nitrogen atom to form the corresponding pyrrolidine-2, 5-dione (Scheme 2)

Experimental

General solvent-free microwave irradiation technique

In an open vessel of the anhydride and N-substituted 4-aminobenzenesulfonamide was dry irradiated from 2-5 min in a microwave oven (1000 watt, 100% power). The reaction progress was monitored by TLC until no more unreacting materials were observed. The reaction mixture was then cooled down to the room temperature and dissolved in chloroform. The chloroform layer was then washed with dilute HCl to get rid of unreacted amines, and then extracted with 10% ice-cold sodium carbonate solution. Acidification of the aqueous layer with ice-cold concentrated hydrochloric acid, precipitated the carboxylic compounds; N-(N'-substituted benzenesulfonamido)-3-carboxy-4-aryl-3-butenamide derivatives. Thoroughly wash of the organic layer with water followed by its dryness over anhydrous sodium sulfate followed by organic solvent distillation gave N-(N'-substituted benzenesulfonamido)-3-substituted pyrrolidine-2, 5-dione derivatives. The products obtained were crystallized from ethanol.

General conventional thermal heating technique

A mixture of α , β -unsaturated anhydride (1-3) and N-substituted 4-aminobenzenesulfonamide (1:2) was heated under reflux for at least 4 hr in ethanol. The reaction progress was monitored by thin layer chromatography (TLC). The reaction mixture was then concentrated and the precipitate formed was filtered and dissolved in chloroform then worked up in the same way given in the *Egypt. J. Chem.* **53**, No. 3 (2010)

solvent-free microwave irradiation technique. Structures of products are confirmed by their spectral analyses, FTIR, ¹HNMR, and MS.

N-(*N'*-Phenylbenzenesulfonamido)-3-benzylidene pyrrolidine-2,5-dione (4)

White crystals, m.p. 195°C, 83% yield in m.w, and 45% in thermal. FTIR (KBr): ν (cm⁻¹) = 3288 (NH), 1775-1704 (2CO; imide), 1372 and 1157 (SO₂; asy. and sym.). MS: m/z = 418 (M⁺, 86.5%, C₂₃H₁₈N₂O₄S), 327 (12.8, C₁₇H₁₃NO₂S), 326 (46.8, C₁₇H₁₂NO₄S) 263 (30, C₁₇H₁₃NO₂), 234(31.4, C₁₆H₁₂NO), 206 (18, C₁₅H₁₂N), 143 (6.5, C₁₀H₇O), 116 (34.5, C₉H₈), 115 (100, C₉H₇), and 97 (5.2, C₄H₃NO₂).

N-[*N'*-(4-Methylphenyl)benzenesulfonamido]-3-benzylidene pyrrolidine-2,5-dione (5)

Yellow crystals, m.p. 238°C, 91% yield in m.w, and 64% in thermal. FTIR (KBr): ν (cm⁻¹) = 3271.5 (NH), 1772-1707 (2CO; imide), 1333 and 1160 (SO₂; asy. and sym.). MS: m/z = 432 (M⁺, 8.3%, C₂₄H₂₀N₂O₄S), 326 (1.6, C₁₇H₁₂NO₂S), 262 (6.5, C₁₃H₁₄N₂O₂S), 116 (40.4, C₉H₈), 115(64, C₉H₇), 107 (8.8, C₇H₉N), 106 (100, C₇H₈N) and 91 (14.5, C₇H₇). ¹HNMR (DMSO-d₆): δ (ppm) = 7.62-7.73 (3H, m; Ph), 7.86-7.93 (2H, m; Ph), 7.61(1H, s; CH Ph), 3.87 (2H, s CH₂CO), 7.06 (2H, d; CH₃Ph), 7.50-7.53 (2H, d; NHPh), 10.28 (1H, s, NH₂SO₂), 7.55-7.58 [(4H, s; NHPhSO₂) and (1H, imp; NHCO)], and 2.15 (3H, s; CH₃Ph).

N-[*N'*-(4-Methoxyphenyl)benzenesulfonamido]-3-benzylidene pyrrolidine-2,5-dione (6)

Pale gray crystals, m.p. 185°C, 92% yield in m.w, and 60% in thermal. FTIR (KBr): ν (cm⁻¹) = 3264 (NH), 1774-1706 (2CO; imide), 1333 and 1160 (SO₂; asy. and sym.). MS: m/z = 448 (M⁺, 48%, C₂₄H₂₀N₂O₅S), 292 (8.7, C₁₇H₁₀NO₂S), 123 (9, C₇H₉NO), 122 (100, C₇H₈NO or C₆H₄NS), 116 (5.8, C₉H₈) and 115 (22.4, C₉H₇). ¹HNMR (DMSO-d₆): δ (ppm) = 7.48-7.59 (3H, m; Ph), 7.70-7.82 (2H, m; Ph), 7.55 (1H, s; CH Ph), 3.87 (2H, s, CH₂CO), 7.59 [(4H, s; NHPhSO₂) and (1H, imp; NHCO)], 10.09 (1H, s, NH₂SO₂), 7.01-7.05 (2H, d; NHPh), 6.81-6.85 (2H, d; PhOCH₃) and 3.68 (3H, s; OCH₃).

N-[*N'*-(4-Chlorophenyl)benzenesulfonamido]-3-benzylidene pyrrolidine-2,5-dione (7)

Pale yellow crystals, m.p. 203°C, 94 % yield in m.w, and 54% in thermal. FTIR (KBr): ν (cm⁻¹) = 3244 (NH), 1771-1711.5 (2CO; imide), 1373 and 1157 (SO₂; asy. and sym.). MS: m/z = 452 (M⁺, 86.5%, C₂₃H₁₇N₂O₄SCl), 326 (38.9, C₁₇H₁₂NO₄S), 262(100, C₁₇H₁₂NO₂), 206 (12.7, C₁₅H₁₂N), 144 (6.2, C₁₀H₈O), 127 (10, C₆H₆NCl), 126 (32.2, C₆H₅NCl), 125 (3.2, C₆H₄NCl), 116(23.5 C₉H₈) and 115 (55, C₉H₇). ¹HNMR (DMSO-d₆): δ (ppm) = 7.70-7.74 (3H, m; Ph), 7.90-7.94 (2H, m; Ph), 7.59 (1H, s; CH Ph), 3.87 (2H, s, CH₂CO), 7.59-7.63 (2H, d; PhNCO), 7.49-7.52 (2H, d; PhSO₂), 10.60 (1H, s, NH₂SO₂), 7.14-7.18 (2H, d; PhCl) and 7.32-7.36 (2H, d; PhNH).

N-[*N'*-(4-Nitrophenyl)benzenesulfonamido]-3-carboxy-4-phenyl-3-utenamide (8)

Yellow crystals, m.p. 180°C, 94% yield in m.w, and 47% in thermal. FTIR (KBr): ν (cm⁻¹) = 3351-3224.5 (2NH), 3400-2400 (OH; acid), 1707.5(CO; acid), 1648.5(CO; amide), 1331 and 1151.5(SO₂; asy. and sym.). MS: m/z = 481 (M⁺, 0%, C₂₃H₁₉N₃O₇S), 463 (42, C₂₃H₁₇N₃O₆S), 327 (9.3, C₁₇H₁₃NO₄S), 262 (46.2, C₁₂H₁₀NO₃S), 156 (100, C₆H₆NO₂S), 116 (17, C₉H₈), 115 (50.3, C₉H₇), 92 (55.6, C₆H₆N), and 65 (42, HO₂S).

N-[*N'*-(1-Naphthyl)benzenesulfonamido]-3-carboxy-4-phenyl-3-butenamide (9)

White crystals, m.p.150°C, 63 % yield in m.w, and 60% in thermal. FTIR (KBr): ν (cm⁻¹) = 3300-3244 (2NH), 3400-2400 (OH; acid), 1700(CO; acid), 1678.1(CO; amide), 1344 and 1158(SO₂; asy. and sym.). MS: m/z = 468 (M⁺, 100%, C₂₇H₂₂N₂O₅S), 326 (28, C₁₇H₁₂NO₄S), 298 (30, C₁₆H₁₄NO₂S), 143(37.5, C₁₀H₇O), 142 (93, C₁₀H₈N), 117 (9.6, C₉H₉), 116 (15.5 C₉H₈), 115 (74, C₉H₇) and 65 (8.4, HOS₂).

N-[*N'*-(1-Naphthyl)benzenesulfonamido]-3-benzylidene-pyrrolidine-2,5-dione (10)

Brown crystals, m.p. 89°C, 19 % yield in m.w, and 0% in thermal. FTIR (KBr): ν (cm⁻¹) = 3247 (NH), 1770.6-1710 (2CO; imide), 1374 and 1159 (SO₂; asy. and sym.). MS: m/z = 468 (M⁺, 0%, C₂₇H₂₀N₂O₄S), 314 (18.5, C₁₆H₁₄N₂O₃S), 313 (79.5, C₁₆H₁₃N₂O₃S), 298 (7.7, C₁₆H₁₄N₂O₂S), 286(2.5, C₁₅H₁₄N₂O₂S), 285 (10.4, C₁₅H₁₃N₂O₂S), 144 (7, C₁₀H₈O), 143 (5, C₁₀H₇O), 116(64, C₉H₈) and 115 (100, C₉H₇).

N-[*N'*-Benzylbenzenesulfonamido]-3-carboxy-4-phenyl-3-butenamide (11)

White crystals, m.p. 170°C, 88 % yield in m.w, and 61% in thermal. FTIR (KBr): ν (cm⁻¹) = 3332.4-3203.3 (2NH), 3400-2400 (OH; acid), 1682.5(CO; acid), 1634.5(CO; amide), 1538 and 1156.5(SO₂; asy. and sym.). MS: m/z = 450 (M⁺, 20.7%, C₂₄H₂₂N₂O₅S), 116 (26.4, C₉H₈), 115 (39, C₉H₇), 108 (12.7, C₇H₁₀N), 107 (11, C₇H₉N), 106(100, C₇H₈N), 91 (20, C₇H₇), 66 (7.4, H₂O₂S), 65 (19, HO₂S) and 64 (5, O₂S).

¹HNMR (DMSO-d₆): δ (ppm) = 7.77-7.27 (3H, m; Ph), 7.84-7.78 (2H, m; Ph), 7.48 (1H, s; CHPh), 3.57 (2H, s; CH₂CO), 10.53 (1H, s; NHSO₂), 7.78 [4H, s; (4H, s; NHPhSO₂) and NHCO], 8.06-8.00 (1H, t; NHCH₂), 3.94-3.97 (2H, d; CH₂NH), 7.27 (5H, s; PhCH₂) and 12.5 (1H, broad; COOH).

N-[*N'*-Phenylbenzenesulfonamido]-3-(4-methoxybenzylidene)pyrrolidine-2,5-dione(12)

Pale yellow crystals, m.p. 215°C, 91% yield in m.w, and 67% in thermal. FTIR (KBr): ν (cm⁻¹) = 3242.5 (NH), 1767-1702 (2CO; imide), 1380 and 1161(SO₂; asy. and sym.). MS: m/z = 448 (M⁺, 9.1%, C₂₄H₂₀N₂O₅S), 292 (13, C₁₈H₁₄NO₃), 265 (9, C₁₇H₁₅NO), 146(14.4, C₁₀H₁₀O), 145(14, C₁₀H₉O), 131 (12.3, C₉H₇O) and 92(100, C₆H₆N). ¹HNMR (DMSO-d₆): δ (ppm) = 3.84 (3H, s; OCH₃), 7.05-7.09 (2H, d; PhOCH₃), 7.90-7.94 (2H, d; PhCH=), 7.66 (1H, s;

CHPh), 3.2-3.6 (2H, imp.; CH₂CO), 7.70 [4H, s; NPhSO₂], 10.46 (1H, broad; NHSO₂) and 7.14-7.62 (5H, m; Ph).

N-[*N'*-(4-Methylphenyl)benzenesulfonamido]-3-(4-methoxybenzylidene) pyrrolidine-2,5-dione (13)

Yellow crystals, m.p. 220°C, 93% yield in m.w, and 70% in thermal. FTIR (KBr): ν (cm⁻¹) = 3276.3 (NH), 1772.5-1706 (2CO; imide), 1375 and 1161 (SO₂; asy and sym.). MS: m/z = 462 (M⁺, 0%, C₂₅H₂₂N₂O₅S), 246 (6.5, C₁₃H₁₂NO₂S), 216 (3.2, C₁₂H₁₀NO₃), 146 (12, C₁₀H₁₀O), 145 (6.5, C₁₀H₉O), 132 (33.6, C₉H₈O), 115 (18.4, C₉H₇), 105 (100, C₇H₇N) and 104 (97, C₇H₆N). ¹HNMR (DMSO-d₆): δ (ppm) = 3.83 (3H, s; OCH₃), 7.04-7.09 (2H, d; PhOCH₃), 7.86-7.90 (2H, d; PhCH=), 7.59 (1H, s; CHPh), 3.2-3.6 (2H, imp.; CH₂CO), 7.55 (4H, s; (NPhSO₂), 10.27 (1H, s; NHSO₂), 7.04-7.09 [4H,s; CH₃PhNH and 2.20 (3H, s; PhCH₃).

N-[*N'*-(4-Methoxyphenyl)benzenesulfonamido]-3-(4-methoxybenzylidene) pyrrolidine-2,5-dione (14)

Pale yellow crystals, m.p. 175°C, 95% yield in m.w, and 71% in thermal. FTIR (KBr): ν (cm⁻¹) = 3281 (NH), 1769-1706 (2CO; imide), 1377 and 1164 (SO₂; asy. and sym.). MS: m/z = 476 (M⁺, 46%, C₂₅H₂₂N₂O₆S), 292 (9.1, C₁₈H₁₄NO₃), 146 (5.4, C₁₀H₁₀O), 145 (9.8, C₁₀H₉O), 131 (4, C₉H₇O), 123 (9, C₇H₉NO), 122 (100, C₇H₈NO), 106 (2.6, C₆H₄NO), 103 (6.3, C₈H₇), and 65 (2, HOS₂).

N-[*N'*-(4-Chlorophenyl)benzenesulfonamido]-3-carboxy-4-(4-methoxyphenyl)-3-butenamide (15)

White crystals, m.p. 179°C, 0% yield in m.w, and 47% in thermal. FTIR (KBr): ν (cm⁻¹) = 3261-3193 (2NH), 3400-2400 (OH; acid), 1690 (CO, acid), 1667.2 (CO; amide), 1336 and 1157.5 (SO₂; asy. and sym.). MS: m/z = 500 (M⁺, 0%, C₂₄H₂₁N₂O₆SCl), 327 (3.2, C₁₆H₁₁N₂O₄S), 282 (44, C₁₂H₁₁N₂O₂SCl), 218 (23.5, C₁₂H₁₀O₄), 217 (4.2, C₁₂H₁₁NO₃), 191 (3.3, C₁₁H₁₁O₃), 156 (100, C₆H₆NO₂S), 146 (54, C₁₀H₁₀O) and 126 (11.8, C₆H₅NCl). ¹HNMR (DMSO-d₆): δ (ppm) = 3.77 (3H, s; OCH₃), 7.08-7.11 (2H, d; PhOCH₃), 7.47-7.43 (2H, d; PhCH=), 3.55 (2H, s; CH₂CO), 10.35 (1H, s; NHCO), 7.77 [(4H, s; NPhSO₂), and (1H, imp.; CHPh)], 10.55 (1H, s; NHSO₂), 6.98-7.02 (2H, d; PhNH) and 7.28-7.32 (2H, d; PhCl).

N-[*N'*-(4-Chlorophenyl)benzenesulfonamido]-3-(4-methoxybenzylidene) pyrrolidine-2,5-dione (16)

Yellow crystals, m.p. 230°C, 86% yield in m.w, and 30% in thermal. FTIR (KBr): ν (cm⁻¹) = 3254 (NH), 1768-1708 (2CO; imide), 1377 and 1161 (SO₂; asy. and sym.). MS: m/z = 482 (M⁺, 65.4%, C₂₄H₁₉N₂O₅SCl), 356 (39.1, C₁₈H₁₄NO₅S), 293 (27.6, C₁₈H₁₅NO₃), 292 (100, C₁₈H₁₄NO₃), 264 (33.7, C₁₂H₇NO₂), 146 (25.4, C₁₀H₁₀O), 145 (21.3, C₁₀H₉O), 131 (21.7, C₉H₇O), 126 (31, C₆H₅NCl) and 115 (6.5, C₉H₇). ¹HNMR (DMSO-d₆): δ (ppm) = 3.83 (3H, s; OCH₃), 7.13-7.17 (2H, d; PhOCH₃), 7.57-7.62 (2H, d; PhCH=), 7.57-7.69 (1H,

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imp.; CHPh), 3.2-3.6 (2H, imp.; CH_2CO), 7.89 (2H, d; PhN), 7.93 (2H, d; PhSO_2), 10.60 (1H, s; NHSO_2), 7.05-7.09 (2H,d; PhCl) and 7.32-7.36 (2H, d; PhNH).

N-[*N'*-(4-Nitrophenyl)benzenesulfonamido]-3-carboxy-4-(4-methoxyphenyl)-3-butenamide (17)

Yellowish brown crystals, m.p. 150 °C, 91% yield in m.w, and 43% in thermal. FTIR (KBr): ν (cm^{-1}) = 3295-3255 (2NH), 3400-2400 (OH, acid), 1690(CO; acid), 1672.3 (CO; amide), 1347 and 1160(SO_2 ; asy. and sym.). MS: m/z = 511(M^+ , 0%, $\text{C}_{24}\text{H}_{21}\text{N}_3\text{O}_8\text{S}$), 493 (18.6, $\text{C}_{24}\text{H}_{19}\text{N}_3\text{O}_7\text{S}$), 293 (43.8, $\text{C}_{12}\text{H}_{11}\text{N}_3\text{O}_4\text{S}$), 218(23.4, $\text{C}_{12}\text{H}_{10}\text{O}_4$), 156 (100, $\text{C}_6\text{H}_6\text{NO}_2\text{S}$), 146 (28, $\text{C}_{19}\text{H}_{10}\text{O}$), 139 (6.6, $\text{C}_6\text{H}_7\text{N}_2\text{O}_2$), 131 (8, $\text{C}_9\text{H}_7\text{O}$), 115 (6.3, C_9H_7) and 92 (94.8, $\text{C}_6\text{H}_6\text{N}$).

N-[*N'*-(1-Naphthyl) benzenesulfonamido] -3-carboxy-4- (4-methoxyphenyl)-3-butenamide (18)

Pale brown crystals, m.p. 170°C, 64% yield in m.w, and 68% in thermal. FTIR (KBr): ν (cm^{-1}) =3244-3203 (2NH), 3400-2400 (OH; acid), 1717(CO; acid), 1667(CO; amide), 1257 and 1176.8(SO_2 ; asy. and sym.). MS: m/z = 516 (M^+ , 0%, $\text{C}_{28}\text{H}_{24}\text{N}_2\text{O}_6\text{S}$), 499(22.3, $\text{C}_{28}\text{H}_{23}\text{N}_2\text{O}_5\text{S}$), 498 (17, $\text{C}_{28}\text{H}_{22}\text{N}_2\text{O}_5\text{S}$), 218 (65, $\text{C}_{12}\text{H}_{10}\text{O}_4$), 191 (58.8, $\text{C}_{11}\text{H}_{11}\text{O}_3$), 147 (40, $\text{C}_{10}\text{H}_{11}\text{O}$), 146 (100, $\text{C}_{10}\text{H}_{10}\text{O}$), 143 (38.1 $\text{C}_{10}\text{H}_9\text{N}$), 131 (47.5, $\text{C}_9\text{H}_7\text{O}$), 115 (15.3, C_9H_7) and 92 (11.3, $\text{C}_6\text{H}_6\text{N}$).

N-[*N'*-(1-Naphthyl)benzenesulfonamido]-3-(4-methoxybenzylidene) pyrrolidine -2,5-dione (19)

Reddish brown crystals, m.p. 226°C, 26% yield in m.w, and 0% in thermal. FTIR (KBr): ν (cm^{-1}) =3281(NH), 1704-1706 (2CO; imide), 1599 and 1126(SO_2 ; asy. and sym.). MS: m/z = 498 (M^+ , 87.6%, $\text{C}_{28}\text{H}_{22}\text{N}_2\text{O}_5\text{S}$), 146 (12.6, $\text{C}_{10}\text{H}_{10}\text{O}$), 145 (9.1, $\text{C}_{10}\text{H}_9\text{O}$), 143 (24.3, $\text{C}_{10}\text{H}_7\text{O}$ or $\text{C}_{10}\text{H}_9\text{N}$), 142(100, $\text{C}_{10}\text{H}_8\text{N}$), 122(6.2, $\text{C}_6\text{H}_4\text{NS}$), 115(53.3, C_9H_7) and 103 (9, C_8H_7).

N-[*N'*-Benzylbenzenesulfonamido]-3-carboxy-4-(4-methoxyphenyl)-3-butenamide (20)

Gray crystals, m.p. 190°C, 88% yield in m.w, and 27% in thermal. FTIR (KBr): ν (cm^{-1}) =3371-3260(2NH), 3400-2400 (OH; acid), 1711(CO; acid), 1680 (CO; amide), 1323 and 1154(SO_2 ; asy. and sym.). MS: m/z = 480 (M^+ , 9.1%, $\text{C}_{25}\text{H}_{24}\text{N}_2\text{O}_6\text{S}$), 293 (15, $\text{C}_{18}\text{H}_{15}\text{NO}_3$), 262 (12, $\text{C}_{13}\text{H}_{14}\text{N}_2\text{O}_2\text{S}$), 191 (10, $\text{C}_{11}\text{H}_{11}\text{O}_3$), 156 (15 $\text{C}_6\text{H}_6\text{NO}_2\text{S}$), 146 (56.5, $\text{C}_{10}\text{H}_{10}\text{O}$), 145(39.4, $\text{C}_{10}\text{H}_9\text{O}$), 106 (71.7, $\text{C}_7\text{H}_8\text{N}$), 91 (84, C_7H_7) and 77 (100, C_6H_5). ^1H NMR (DMSO- d_6): δ (ppm) = 3.79 (3H, s; OCH_3), 7.00-7.04 (2H, d; PhOCH_3), 7.47-7.50 (2H, d; PhCH=), 3.59 (2H, s; CH_2CO), 10.58 (1H, s; NHCO), 7.79 [4H, s; NHPhSO_2] and (1H, imp., PhCH=), 8.02 (1H, t; NHSO_2), 3.97 (2H, d; CH_2NH) and 7.28 (5H, s; PhCH_2).

N-(*N'*-Phenylbenzenesulfonamido)-3-carboxy-4-methyl-4-phenyl-3-butenamide (21)

White crystals, m.p. 162°C, 60% yield in m.w, and 20% in thermal. FTIR (KBr): ν (cm^{-1}) = 3300-3250(2NH), 3400-2400 (OH; acid), 1690(CO; acid), 1688 (CO; amide), 1337 and 1157(SO_2 ; asy and sym.). MS: m/z = 450 (M^+ , 0%, $\text{C}_{24}\text{H}_{22}\text{N}_2\text{O}_5\text{S}$), 433 (40, $\text{C}_{24}\text{H}_{21}\text{N}_2\text{O}_4\text{S}$), 432 (100, $\text{C}_{24}\text{H}_{20}\text{N}_2\text{O}_4\text{S}$), 340 (37.3, *Egypt. J. Chem.* **53**, No. 3 (2010)

$C_{18}H_{14}NO_4S$), 277 (0, 4 $C_{18}H_{15}NO_2$), 276 (62.7, $C_{18}H_{14}NO_2$), 248(30, $C_{12}H_{12}N_2O_2S$), 156 (28, $C_6H_6NO_2S$), 130 (23.2, $C_{10}H_{10}$), 115 (28.4, C_9H_7) and 92 (40.4, C_6H_6N). 1H NMR (DMSO- d_6): δ (ppm) = 7.66 (5H, s; Ph), 2.36 (3H, s; $PhC(CH_3)=C$), 3.19 (2H, s; CH_2CO), 10.39 (1H, s; $NHOSO_2$), 7.36 (4H, s; $NPhSO_2$), 10.16 (1H, s; $NHCO$), 7.05-7.23 (5H, m; $NHPh$) and 12.56 (1H, broad; COOH).

N-(N'-Phenylbenzenesulfonamido)-3-phenylethylidene-pyrrolidine-2, 5-dione (22)

Pale yellow crystals, m.p. 136°C, 20% yield in m.w, and 0% in thermal. FTIR (KBr): ν (cm^{-1}) = 3248.3(NH), 1766-1707 (2CO; imide), 1377 and 1159 (SO_2 ; asy. and sym.). MS: m/z = 432 (M^+ , 100%, $C_{24}H_{20}N_2O_4S$), 340 (47, $C_{18}H_{14}NO_4S$), 276 (93.8, $C_{18}H_{14}NO_2$), 248(29.6, $C_{12}H_{12}N_2O_2S$), 233 (10, $C_{12}H_{11}NO_2S$), 130 (29, $C_{10}H_{10}$), 129 (43.8, $C_{10}H_9$), 115 (32.4, C_9H_7) and 92 (44, C_6H_6N). 1H NMR (DMSO- d_6): δ (ppm) = 7.52-7.66 (3H, m; Ph), 7.89-7.93 (2H, m; Ph), 2.34 (3H, s; $PhC(CH_3)=C$), 2.63 (2H, s; CH_2CO), 7.44 (4H, s; $NPhSO_2$), 10.44 (1H, s; $NHOSO_2$) and 7.05-7.32 (5H, m; Ph).

N-[N'-(4-Methylphenyl)benzenesulfonamido]-3-carboxy-4-methyl-4-phenyl-3-butenamide (23)

White crystals, m.p. 210°C, 67% yield in m.w, and 22% in thermal. FTIR (KBr): ν (cm^{-1}) = 3300-3255(2NH), 3400-2400 (OH; acid), 1705(CO; acid), 1688.5 (CO; amide), 1331 and 1157(SO_2 ; asy. and sym.). MS: m/z = 464 (M^+ , 0%, $C_{25}H_{24}N_2O_5S$), 447 (80.7, $C_{25}H_{23}N_2O_4S$), 446 (79, $C_{25}H_{22}N_2O_4S$), 277 (15.3, $C_{18}H_{15}NO_2$), 276(33, $C_{18}H_{14}NO_2$), 130(8, $C_{10}H_{10}$), 115 (22.5, C_9H_7), 107 (6.5, C_7H_9N), 106(100, C_7H_8N) and 91 (2.2, C_7H_7). 1H NMR (DMSO- d_6): δ (ppm) = 7.63 (5H, s; Ph), 2.35 (3H, s; $PhC(CH_3)=C$), 3.18 (2H, s; CH_2CO), 10.24 (1H, s; $NHOSO_2$), 7.36 (4H, s; $NHPhSO_2$), 9.99 (1H, s; $NHCO$), 7.24 (2H,d; $NHPh$), 7.00 (2H,d; CH_3Ph) and 2.18 (3H, s; CH_3Ph).

N-[N'-(4-Methylphenyl)benzenesulfonamido]-3-phenylethylidene-pyrrolidine-2,5-dione (24)

Yellow crystals, m.p. 130°C, 25% yield in m.w, and 0% in thermal. FTIR (KBr): ν (cm^{-1}) = 3250(NH), 1767-1705 (2CO; imide), 1378 and 1158 (SO_2 ; asy. and sym.). MS: m/z = 446 (M^+ , 90.8%, $C_{25}H_{22}N_2O_4S$), 277 (20, $C_{18}H_{15}NO_2$), 276 (28.5, $C_{18}H_{14}NO_2$), 340(9.8, $C_{18}H_{14}NO_4S$), 130 (12, $C_{10}H_{10}$), 115 (23, C_9H_7), 107 (13, C_7H_9N), 106 (100, C_7H_8N), 102 (3, C_8H_6) and 79 (30, HNO_2S). 1H NMR (DMSO- d_6): δ (ppm) = 7.49-7.64 (3H, m; Ph), 7.78-7.89 (2H, m; Ph), 2.36 (3H, s; $PhC(CH_3)=C$), 2.6 (2H, s; CH_2CO), 7.44 (4H, s; $NPhSO_2$), 10.28 (1H, s; $NHOSO_2$), 7.00 [4H,s; $NHPhCH_3$], and 2.13 (3H, s; CH_3Ph).

N-[N'-(4-Methoxyphenyl) benzenesulfonamido]-3-carboxy-4-methyl-4-phenyl- 3-butenamide (25)

Pale yellow crystals, m.p. 130°C, 52% yield in m.w, and 72% in thermal. FTIR (KBr): ν (cm^{-1}) = 3300-3243(2NH), 3400-2400 (OH; acid), 1705(CO; acid), 1665.5 (CO; amide), 1333 and 1158(SO_2 ; asy. and sym.). MS: m/z = 480(M^+ , 2.3%, $C_{25}H_{24}N_2O_6S$), 278 (33, $C_{13}H_{13}N_2O_3S$), 156 (4.3, C_6H_6NO), 123 (20.4,

C₇H₉NO), 131 (1, C₁₀H₁₁), 122(100, C₆H₄NS or C₇H₈NO), 108 (9, C₇H₈O), 92 (10.3, C₆H₆N) and 79 (4, HNO₂S). ¹HNMR (DMSO-d₆): δ (ppm) = 7.61 (5H, s; Ph), 2.26 (3H, s; PhC(CH₃)=C), 3.17 (2H, s; CH₂CO), 10.21 (1H, s; NHSO₂), 7.36 (4H, s; NHP_hSO₂), 9.78 (1H, s; NHCO), 6.94(2H, d; PhNH), 6.81 (2H, d; PhOCH₃) and 3.67(3H, s; OCH₃).

N-[N'-(4-Methoxyphenyl) benzenesulfonamido]-3- phenylethylidene pyrrolidine - 2,5-dione (26)

Yellow crystals, m.p. 178°C, 40% yield in m.w, and 0% in thermal. FTIR (KBr): ν (cm⁻¹) = 3254(NH), 1767-1705 (2CO; imide), 1376 and 1160(SO₂; asy. and sym.). MS: m/z = 462 (M⁺, 35%, C₂₅H₂₂N₂O₅S), 130 (4, C₁₀H₁₀), 129 (5.1, C₁₀H₉), 123 (8.1, C₇H₉NO), 122 (100, C₇H₈O or C₆H₄NS), 115(8, C₉H₇), 102 (2, C₈H₆) and 96 (1.9, C₄H₂NO₂). ¹HNMR (DMSO-d₆): δ (ppm) = 7.33-7.55 (3H, m; Ph), 7.80-7.85 (2H, m; Ph), 2.33 (3H, s; PhC(CH₃)=C), 2.62 (2H, s; CH₂CO), 7.44 (4H, s; NPhSO₂), 10.08 (1H, s; NHSO₂), 7.01-7.06 (2H, d; PhNH), 6.81-6.86 (2H, d; PhOCH₃) and 3.68 (3H, s; OCH₃).

N-[N'-(4-Chlorophenyl) benzenesulfonamido]-3-carboxy-4-methyl-4-phenyl- 3-butenamide (27)

White crystals, 200°C, 30% yield in yield in m.w, and 37% in thermal. FTIR (KBr): ν (cm⁻¹) = 3300-3243(2NH), 3400-2400 (OH; acid), 1705(CO; acid), 1665.5 (CO; amide), 1333 and 1158(SO₂; asy. and sym.). MS: m/z = 484 (M⁺, 5%, C₂₄H₂₁N₂O₅SCl) 466 (100, C₂₄H₁₉N₂O₄SCl), 340 (55.5, C₁₈H₁₄NO₄S), 277 (19, C₁₈H₁₅NO₂), 276 (99.4, C₁₈H₁₄NO₂), 220 (6.6, C₁₆H₁₄N), 157 (9.6, C₁₁H₉O), 126 (46, C₆H₅NCl), 129 (25.6, C₁₀H₉), 115 (18.8, C₉H₇) and 79 (2, HNO₂S).

N-[N'-(4-Chlorophenyl) benzenesulfonamido]-3-phenylethylidene pyrrolidine-2,5-dione (28)

Pale yellow crystals, m.p. 110°C, 63% yield in m.w, and 37% in thermal. FTIR (KBr): ν (cm⁻¹) = 3241(NH), 1766-1706 (2CO; imide), 1375 and 1160(SO₂; asy. and sym.). MS: m/z = 466 (M⁺, 100%, C₂₄H₁₉N₂O₄SCl), 340 (55.5, C₁₈H₁₄NO₄S), 277 (19, C₁₈H₁₅NO₂), 276 (99.4, C₁₈H₁₄NO₂), 220 (6.6, C₁₆H₁₄N), 157 (9.6, C₁₁H₉O), 126 (46, C₆H₅NCl), 129 (25.6, C₁₀H₉), 115 (18.8, C₉H₇) and 79 (2, HNO₂S).

N-[N'-(4-Nitrophenyl) benzenesulfonamido]-3-carboxy-4-methyl-4-phenyl-3-butenamide (29)

Pale yellow crystals, m.p. 119°C, 79% yield in m.w, and 61% in thermal. FTIR (KBr): ν (cm⁻¹) = 3360-3355(2NH), 3400-2400 (OH; acid), 1700(CO; acid), 1690 (CO; amide), 1344 and 1160(SO₂; asy. and sym.). MS: m/z = 495 (M⁺, 1.8%, C₂₄H₂₁N₃O₇S), 294 (4, C₁₈H₁₇NO₃), 293 (19, C₁₂H₁₁N₃O₄S), 292 (2.5, C₁₂H₁₀N₃O₄S), 202(9.2, C₁₂H₁₀O₃), 156(100, C₆H₆NO₂S), 137(0.9, C₆H₅N₂O₂), 131 (3, C₁₀H₁₁), 130 (13, C₁₀H₁₀), 115 (16.3, C₉H₇) and 92 (87, C₆H₆N).

N-[*N'*-(1-Naphthyl) benzenesulfonamido]-3-carboxy-4-methyl-4-phenyl-3-butenamide (30)

Pale violet crystals, m.p. 129°C, 53% yield in m.w, and 41% in thermal. FTIR (KBr): ν (cm⁻¹) = 3300-3248 (2NH), 3400-2400 (OH; acid), 1700(CO; acid), 1698 (CO; amide), 1399.9 and 1159 (SO₂; asy. and sym.). MS: m/z = 500(M⁺, 0%, C₂₈H₂₄N₂O₅S), 328 (1.3, C₁₇H₁₆N₂O₃S), 327(4, C₁₇H₁₅N₂O₃S), 298 (12.7, C₁₆H₁₄N₂O₂S), 297 (2.4, C₁₆H₁₃N₂O₂S), 143 (45, C₁₀H₉N), 142 (27.7, C₁₀H₈N), 130 (65, C₁₀H₁₀), 129 (92, C₁₀H₉), 115 (100, C₉H₇) and 79 (4, HNO₂S). ¹HNMR (DMSO-d₆): δ (ppm) = 7.75-7.87 (3H,m; Ph), 8.07(2H, m; Ph), 2.36 (3H, s; PhC(CH₃)=C), 3.19 (2H, s; CH₂CO), 10.49 (1H, s; NH₂SO₂), 7.44 (4H, s; NHPhSO₂), 10.20 (1H, s; NHCO) and 7.80-7.10 (7H, m; C₈H₇).

N-[*N'*-(1-Naphthyl) benzenesulfonamido]-3-phenylethylidene-pyrrolidine-2,5-dione (31)

Violet crystals, m.p. 98°C, 15% yield in m.w, and 0% in thermal. FTIR (KBr): ν (cm⁻¹) = 3251(NH), 1766.5-1706 (2CO; imide), 1372and 1159(SO₂; asy. and sym.). MS: m/z = 482 (M⁺, 0%, C₂₈H₂₂N₂O₄S), 328 (14.1, C₁₇H₁₆N₂O₃S), 327 (64.7, C₁₇H₁₅N₂O₃S), 298 (13.5, C₁₆H₁₄N₂O₂S), 297 (4, C₁₆H₁₃N₂O₂S), 202(23.8, C₁₂H₁₀O₃), 143 (13, C₁₀H₉N), 142 (11.4, C₁₀H₈), 130 (90.3, C₁₀H₁₀), 129 (100, C₁₀H₉) and 115 (88, C₉H₇). ¹HNMR (DMSO-d₆): δ (ppm) = 7.80-7.88 (3H, m; Ph), 8.07 (2H, m; Ph), 2.34 (3H, s; PhC(CH₃)=C), 3.01 (2H, s; CH₂CO), 7.45 (4H, s; NPhSO₂), 10.40 (1H, s; NH₂SO₂) and 7.17-7.62 (7H, m; C₈H₇).

N-(*N'*-Benzylbenzenesulfonamido)-3-carboxy-4-methyl-4-phenyl-3-butenamide (32)

White crystals, m.p. 153°C, 85% yield in m.w, and 57% in thermal. FTIR (KBr): ν (cm⁻¹) = 3321-3267(2NH), 3400-2400 (OH; acid), 1700(CO; acid), 1680 (CO; amide), 1328 and 1157(SO₂; asy. and sym.). MS: m/z = 464 (M⁺, 0%, C₂₅H₂₄N₂O₅S), 446 (22.4, C₂₅H₂₂N₂O₄S), 262 (32, C₁₃H₁₄N₂O₂S), 202 (36.5, C₁₂H₁₀O₃), 157(9.2, C₁₁H₉O), 156(15.2, C₆H₆NO₂S), 130 (41.4, C₁₀H₁₀), 129 (74, C₁₀H₉), 115 (46, C₉H₇), 106 (100, C₇H₈N), 92 (66.4, C₆H₆N) and 79 (14, HNO₂S).

N-(4-Methylphenyl)-3-carboxy-4-methyl-4-phenyl-3-butenamide (33)

White crystals, m.p. 162°C, 0% yield in m.w, and 26% in thermal. FTIR (KBr): ν (cm⁻¹) = 3300 (NH), 3100-2500 (OH acid), 1680 (CO, acid), and 1650 (CO, amide). MS: m/z = 309 (M⁺, 25%, C₁₉H₁₉NO₃), 291 (22, C₁₉H₁₇NO₂), 202 (30, C₁₂H₁₀O₃), 130 (25, C₁₀H₁₀), 129 (33, C₁₀H₉), and 107 (100, C₇H₉N).

N-(4-Methylphenyl)-3-phenylethylidene-pyrrolidine-2, 5-dione (34)

Pale brown crystals, m.p. 118°C, 93% yield in m.w, and 0% in thermal. FTIR (KBr): ν (cm⁻¹) = 1756.8-1699 (2CO; imide). MS: m/z = 307 (M⁺, 100%, C₁₉H₁₇NO₃), 292 (2.6, C₁₈H₁₄NO₃), 202 (1.7, C₁₂H₁₂NO₂), 149 (33, C₈H₇NO₂), 130 (91.2, C₁₀H₁₀), 129 (75, C₁₀H₉), 115 (53, C₉H₇) and 108 (16, C₇H₈O).

N-(4-Methoxyphenyl)-3-carboxy-4-methyl-4-phenyl-3-butenamide (35)

Gray crystals, m.p. 172°C, 0% yield in m.w, and 45% in thermal. FTIR (KBr): ν (cm⁻¹) = 3298 (NH), 3100-2500 (OH acid), 1698 (CO, acid), and 1663

(CO, amide). MS: $m/z = 325$ (M^+ , 8%, $C_{19}H_{19}NO_4$), 307 (8%, $C_{19}H_{17}NO_3$), 202 (13%, $C_{12}H_{10}O_3$), 130 (31%, $C_{10}H_{10}$), 129 (53%, $C_{10}H_9$), and 123 (100%, C_7H_9NO). 1H NMR (DMSO- d_6): δ (ppm) = 7.31-7.43 (5H, m; Ph), 2.33 (3H, s; $PhC(\underline{CH_3})=C$), 3.13 (2H, s; $\underline{CH_2}CO$), 9.57 (1H, s; $NH\underline{CO}$), 7.23-7.25 (2H, d; $NHPh$), 6.82 – 6.85 (2H, d; $PhO\underline{CH_3}$), and 3.70 (3H, s; $O\underline{CH_3}$).

N-(4-Methoxyphenyl)-3-phenylethylidene pyrrolidine-2, 5-dione (36)

Gray crystals, m.p. 144°C, 97% yield in m.w, and 0% in thermal. FTIR (KBr): ν (cm^{-1}) = 1756.8-1699 (2CO; imide). MS: $m/z = 307$ (M^+ , 100%, $C_{19}H_{17}NO_3$), 292 (2.6, $C_{18}H_{14}NO_3$), 202 (1.7, $C_{12}H_{12}NO_2$), 149 (33, $C_8H_7NO_2$), 130 (91.2, $C_{10}H_{10}$), 129 (75, $C_{10}H_9$), 115 (53, C_9H_7) and 108 (16, C_7H_8O).

N-(4-Chlorophenyl)-3-carboxy-4-methyl-4-phenyl-3-butenamide (37)

White crystals, m.p. 176°C, 0% yield in m.w, and 49% in thermal. FTIR (KBr): ν (cm^{-1}) = 3290 (NH), 3100-2500 (OH; acid), 1695 (CO; acid) and 1660 (CO; amid). MS: $m/z = 329$ (M^+ , 4%, $C_{18}H_{16}NO_3Cl$), 311 (6%, $C_{18}H_{14}NO_2Cl$), 202 (9%, $C_{12}H_{10}O_3$), 130 (27%, $C_{10}H_{10}$), 129 (64%, $C_{10}H_9$), and 127 (100% C_6H_6NCl).

N-(4-Chlorophenyl)-3-phenylethylidene pyrrolidine-2, 5-dione (38)

Brown crystals, m.p. 146°C, 80% yield in m.w, and 0% in thermal. FTIR (KBr): ν (cm^{-1}) = 1755-1704 (2CO; imide). MS: $m/z = 311$ (M^+ , 68.7%, $C_{18}H_{14}NO_2Cl$), 158 (9.2, $C_{11}H_{10}O$), 131 (12.5, $C_{10}H_{11}$), 130 (100, $C_{10}H_{10}$), 129 (83.2, $C_{10}H_9$), 127 (19, C_6H_6NCl), 125 (7.8, C_6H_4NCl), 115 (70.2, C_9H_7), 111 (5.3, C_6H_4Cl) and 102 (8, C_8H_6).

N-(4-Nitrophenyl)-3-carboxy-4-methyl-4-phenyl-3-butenamide (39)

Yellow crystals, m.p. 182°C, 0% yield in m.w, and 42% in thermal. FTIR (KBr): ν (cm^{-1}) = 3291 (NH), 3100-2500 (OH; acid), 1684 (CO; acid) and 1640 (CO; amid). MS: $m/z = 340$ (M^+ , 0%, $C_{18}H_{16}N_2O_5$), 322 (29%, $C_{18}H_{14}N_2O_4$), 202 (21%, $C_{12}H_{10}O_3$), 138 (54%, $C_6H_6N_2O_2$), and 129 (100%, $C_{10}H_9$).

N-(4-Nitrophenyl)-3-phenylethylidene pyrrolidine-2,5-dione (40)

Brown crystals, m.p. 150°C, 89% yield in m.w, and 0% in thermal. FTIR (KBr): ν (cm^{-1}) = 1754.4-1703 (2CO; imide). MS: $m/z = 322$ (M^+ , 5%, $C_{18}H_{14}N_2O_4$), 202 (7, $C_{12}H_{12}NO_2$), 158 (6.6, $C_{11}H_{10}O$), 157 (11.2, $C_{11}H_9O$), 136 (2.3, $C_6H_4N_2O_2$), 130 (100, $C_{10}H_{10}$), 129 (84.6, $C_{10}H_9$), 122 (2.3, $C_6H_4NO_2$), and 115 (85, C_9H_7).

N-(1-Naphthyl)-3-carboxy-4-methyl-4-phenyl-3-butenamide (41)

Pale violet crystals, m.p. 180°C, 19% yield in m.w, and 35% in thermal. FTIR (KBr): ν (cm^{-1}) = 3270 (NH), 3100-2500 (OH; acid), 1700(CO; acid) and 1640 (CO; amid). MS: $m/z = 345$ (M^+ , 5%, $C_{22}H_{19}NO_3$), 327 (3%, $C_{22}H_{17}NO_2$), 202 (5%, $C_{12}H_{10}O_3$), 143 (100%, $C_{10}H_9N$), 130 (15%, $C_{10}H_{10}$), and 129 (27%, $C_{10}H_9$).

N-(1-Naphthyl)-3-phenylethylidene pyrrolidine-2, 5-dione (42)

Dark gray crystals, m.p. 135°C, 71% yield in m.w, and 0% in thermal. FTIR (KBr): ν (cm^{-1}) = 1762-1703 (2CO; imide). MS: $m/z = 327$ (M^+ , 96.4%, $C_{22}H_{17}NO_2$), 270 (4.1, $C_{20}H_{16}N$), 202 (2, $C_{12}H_{12}NO_2$), 169 (25.5, $C_{11}H_7NO$), 157 *Egypt. J. Chem.* **53**, No. 3 (2010)

(9.5, C₁₁H₉O), 158 (7.6, C₁₁H₁₀O), 131 (7, C₁₀H₁₁), 130 (100, C₁₀H₁₀)129 (64, C₁₀H₉), 128 (46, C₁₀H₈), 127 (23, C₁₀H₇) and 115 (53, C₉H₇).

N-Benzyl-3-carboxy-4-methyl-4-phenyl-3-butenamide (43)

White crystals, m.p. 142°C, 92% yield in m.w, and 71% in thermal. FTIR (KBr): ν (cm⁻¹) = 3263 (NH), 3100-2500 (OH; acid), 1700(CO; acid) and 1640 (CO; amid). MS: m/z = 309 (6%, C₁₉H₁₉NO₃), 291 (20% C₁₉H₁₇NO₂), 202(23.8, C₁₂H₁₀O₃), 143 (13, C₁₀H₉N), 142 (11.4, C₁₀H₈), 130 (90.3, C₁₀H₁₀), 202 (9, C₁₂H₁₀O₃) 130 (35, C₁₀H₁₀), 129 (46, C₁₀H₉), 107 (18, C₇H₉N), and 91 (100, C₇H₇).¹HNMR (DMSO-d₆): δ (ppm) = 7.13-7.40 (10H, m; 2 Ph), 2.2 (3H, s; CH₃), 2.9 (2H, s; CH₂CO), 8.99 (1H, s; NHCO), and 4.24 – 4.26 (2H, d, CH₂NH).

Conclusion

- In the present work, the comparison shows that the microwave irradiation furnishes the formation of the products in excellent yields, shorter time, enhances cyclization, significantly improves the reaction products quantitatively, qualitatively, economically, and environmentally (green chemistry), much more than thermal heating. Moreover, microwave irradiation reactions show regiospecific property, more than thermal ones.

- The results obtained from microwave irradiation of N-substituted 4-aminobenzenesulfonamides with α , β -unsaturated anhydrides show low reactivity than the corresponding non-sulfonated amines. This low reactivity can be attributed to the presence of bezenesulfonamido group.

General Remarks

Microwave irradiation was carried out in a Galanz Microwave Oven, WP1000AP30-2, Chemistry Department, University College of Women for Arts, Science and Education, Ain Shams University. Spectral measurements were carried out at Micro Analytical Centre, Faculty of Science, Cairo University using:

- (a) FTIR PERKIN-ELMER-1430 infrared spectrophotometer; IR spectra.
- (b) GCMS QP 1000 EX Shimaedzy; MS spectra.
- (c) Varian Gemmi (200 MHz); ¹HNMR spectra.

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استخدام كفاءة أشعة الميكروويف فى تشييد بعض البيروليدينات ٢-،
٥- دايون والبيوتينيמידات ومشتقاتهم من السالفوناميد فى وعاء
واحد ومقارنتها بالطريقة التقليدية بالتسخين الحرارى

شادية محمود عبدالله

قسم الكيمياء - كلية النبات للآداب والعلوم والتربية - جامعة عين شمس - القاهرة -
مصر .

تم تشييد بعض البيروليدينات ٢- ، ٥ - دايون والبيوتينيמידات ومشتقاتهم من السالفوناميد نظرا لأهميتهم فى التطبيقات الطبية الواسعة ونشاطهم البيولوجى كمبيدات للبكتريا والفطريات وكمبيدات حشرية ، وذلك بإستخدام طريقة آمنة اقتصادية وصديقة للبيئة (الكيمياء الخضراء) . الهدف من هذا البحث تشييد المركبات (٤-٤) عن طريق تكاثف الأنهيدريدات غير المشبعة فى الوضع ألفا- بيتا (٣-١) مع بعض الأمينات بإستخدام أشعة الميكروويف ومقارنة كفاءتها مع الطريقة التقليديه بالتسخين الحرارى ، وقد أوضحت المقارنة أن تميز طريقة استخدام أشعة الميكروويف على الطريقة التقليدية من حيث قصر مدة إتمام التفاعل بالإضافة إلى تحسن واضح جدا فى الناتج كما وكيفا كما أنها أثبتت قدرتها على انتقائية الناتج ولها دورا كبيرا فى تكوين نواتج حلقية لم يمكن تكوينها بطريقة التسخين التقليدية .

وقد أثبتت مقارنة نتائج تكاثف الأنهيدريدات غير المشبعة فى الوضع ألفا - بيتا (٣-١) مع N - مشتقات ٤-أمينو بنزين سالفوناميد أو الأمينات العادية أن التكاثف مع N - مشتقات ٤-أمينو بنزين سالفوناميد أقل نشاطا من الأمينات العادية وأرجع ذلك إلى وجود مجموعة البنزين فى N - مشتقات ٤-أمينو بنزين سالفوناميد .

ولقد تم اثبات تركيب المركبات الناتجة عن طريق التحليل الطيفى (طيف الكتلة - الأشعة تحت الحمراء والرنين النووى المغناطيسى) .