Aqueous versus Neat Reaction Conditions: The Microwave-Assisted, Selective Conversion of a Fused Anhydride Ring with Amines in the Presence of a Keto Group

Jure Hren, Krištof Kranjc, Slovenko Polanc, Marijan Kočevar*
Faculty of Chemistry and Chemical Technology, University of Ljubljana, Aškerčeva 5, 1000 Ljubljana, Slovenia
Fax +386(1)2419220; E-mail: marijan.kocevar@fkkt.uni-lj.si
Received 23 July 2007; revised 5 November 2007

Abstract: We describe the transformation of fused succinic anhydride derivatives of the bicyclo[2.2.2]oct-2-ene system with various amines under microwave-irradiation conditions. In aqueous suspensions, the reaction of an acetyl-containing derivative yielded chemoselectively succinimide derivatives with the acetyl group remaining unchanged. Conversely, the application of neat reaction conditions with a minor amount of a liquid additive (toluene) yielded succinimide derivatives with the acetyl moiety transformed into an imine group.

Key words: imides, imines, chemoselectivity, green chemistry, microwave synthesis

Within the last decade, great efforts have been made in the field of green chemistry to adopt methods and processes that use less-toxic chemicals, produce smaller amounts of byproducts, and use less energy.1 As part of this green concept, toxic and/or flammable organic solvents are replaced by alternative nontoxic and nonflammable media, or the reactions are carried out without the use of any solvent. Microwave-assisted reactions2 have attracted considerable attention, and many efficient, ecofriendly syntheses3–5 of a variety of organic products have been developed in water3–5c,e,f or as neat4,5a,b,d,e,g,h reactions.

Anhydrides are known to react with primary amines, producing the corresponding amides and imides,6 whereas ketones and aldehydes are transformed to imine-type products.7 Recently, we investigated the transformation of different hydrazines with ketones to give hydrazones,5a,b as well as the reactions of fused anhydrides to produce the corresponding imides.5c,f We also looked into the possible chemoselectivity of the reaction of different hydrazines with bicyclo[2.2.2]oct-2-ene derivative 1, which in addition to two succinic anhydride rings also contains an acetyl group in its structure.5f We anticipated a higher reactivity for one functionality in comparison with the other (the anhydride versus keto moiety) and the eventual chemoselective conversion of compound 1. However, it became clear that this reaction, under the applied conditions, could not be undertaken chemoselectively; the complete transformation with different hydrazines produced the corresponding hydrazono derivatives.

Based on the above results, we decided to perform the reactions of compounds 1 and 5 with a variety of aryl- and alkylamines to explore the green transformations of the bicyclo[2.2.2]oct-2-ene derivatives with the nucleophiles. In our previous work,5c,f we showed that the transformations of the anhydride moieties of the bicyclo[2.2.2]oct-2-ene derivatives with hydrazines to the corresponding fused succinimides were very general and could be applied to a wide variety of hydrazines. Therefore, we investigated the reaction of bicyclo[2.2.2]oct-2-ene derivatives with various amines under green conditions, using microwave (MW) irradiation and water as the reaction medium.

We began with compound 1 (Table 1), which previously5f did not react selectively with hydrazines in water to yield a derivative with an unreacted carbonyl moiety. The reaction of 1 with aniline (2a) (1.2 mmol of aniline/0.5 mmol of 1) was conducted in water at 160 °C, and the 1H NMR spectroscopic analysis of the crude product that was obtained after 60 minutes of MW irradiation showed a complete conversion. The product 3a, containing the unchanged carbonyl moiety, was isolated in high yield (90%) (Table 1, entry 1). The reaction proceeded in the same manner even when 2 mmol of aniline were used. Based on our previous results from reactions using hydrazines, this was not transformation we expected, but it might be explained by the presence of water, which seems to preclude the formation of imines. When analogous aqueous reaction conditions were applied to the transformation of 1 with different amine derivatives 2b–f, products 3b–f, respectively, were obtained in good to high yields (80–90%) (Table 1, entries 2–6). It is worth mentioning that the aqueous reactions with anilines containing halo substituents, 2c–e, did not proceed as smoothly as those with aniline (2a) or 3-methylaniline (2b); however, the yields of 3c–e after crystallization were still relatively good (80–81%) (Table 1, entries 3–5, respectively).

With this result in mind, we wondered what the required conditions would be to obtain imine products 4 (Table 1). First, we tried to carry out the above reaction without adding any solvent, but using a large excess of aniline (2a) (5.5 mmol of aniline/0.5 mmol of 1). After MW irradiation for 45 minutes at 180 °C, followed by suitable work-up, we obtained 4a without 3a as a byproduct. Next, we decreased the amount of aniline used to 2 mmol and increased the reaction time to 60 minutes of MW irradiation at 160 °C. We detected the desired product 4a accompa-
nied by product 3a and some other side products in the crude reaction mixture (ratio 4a/3a of 10:1, obtained from the \(^1\)H NMR spectrum). We then tried adding a small amount of toluene (100 mg) to improve the reaction conditions for the formation of 4a. The \(^1\)H NMR spectroscopic analysis of the crude reaction mixture revealed that, using toluene, product 3a was not detected, but other unidentified side products still accompanied 4a. Shorter reaction times (30 min) for both the reaction with and without toluene did not prevent the formation of impurities, even when the reaction was conducted at 120 °C. Furthermore, at 160 °C with an extended reaction time of 120 minutes, and with the use of a larger amount of aniline (4 mmol), impurities were still formed. These facts thus exclude the possibility of the side products being intermediates in the synthesis.

Optimization studies on the reaction of 1 with 4-chloroaniline (2c) showed that toluene was indeed an appropriate choice. After 15 min of MW irradiation at 150 °C and without using any solvent, the ratio of 4c/3c/1 was approximately 2:2:1. Increasing the reaction temperature to 160 °C, leaving the other parameters unchanged, seemed to have a beneficial effect as the ratio of 4c/3c changed to 4:3 (and no substrate 1 remained). However, with the addition of toluene (100 mg), using the same conditions, the ratio of 4c/3c was 3:1 and again no substrate 1 remained; nearly identical results were obtained using butan-1-ol (100 mg) instead of toluene. We used toluene as the additive for these transformations as it is well known that for the synthesis of imines, toluene is often used as the solvent for the removal of water by azotropic distillation. Increasing the reaction temperature to 170 °C or increasing the amount of toluene added (0.5 mL) did not result in better yields of product 4c. Nevertheless, after 75 minutes of MW irradiation at 160 °C and use of the correct workup procedure, product 4c could be isolated in its pure form and in good yield (78%) (Table 1, entry 3).

To find out more about the role of toluene, we again selected the transformation of substrate 1 (0.5 mmol) with 4-chloroaniline (2c) (2 mmol). The amount of toluene was varied and the reaction mixture was analyzed by \(^1\)H NMR spectroscopy after 75 minutes of MW irradiation at 160 °C; in all the reactions, no starting material 1 remained. When we added 50 mg of toluene, the ratio of 4c/3c was around 5:2. Therefore, we decided to increase the amount of toluene, and from the reactions using 100, 150, or 200 mg of toluene, the only product observed was imine 4c. After increasing the amount of toluene to 0.5 mL (432 mg), the predominant product was still imine 4c, but 3c was now also present (ratio 4c/3c of ca. 5:1). An additional increase in the amount of toluene to 1.5 mL resulted in the ratio of 4c/3c changing to approximately 1:0.6.

Next, we investigated the possibility of conducting the above reaction on a larger scale. Using twofold amounts of starting compound 1 (1 mmol) and 4-chloroaniline (2c) (4 mmol), as well as toluene (200 mg), and the same reaction vessel (10 mL) as was used previously, the reaction was again complete in 75 minutes. Imine 4c was isolated as the sole product in 80% yield. With quadruple amounts, the solid material filled the reaction vessel to about two-thirds of its volume, and the monitoring of the temperature revealed an unusual temperature profile (overheating at the beginning). Therefore, we decided to carry out this experiment in a larger closed reaction vessel (heavy-wall Ace pressure tube, 38 mL, Aldrich) and using the CEM Discover ‘open vessel mode’ protocol (without pressure measurement). Again, the reaction finished in 75 minutes and we isolated 4c as the sole product in 83% yield. For a

Table 1  Reactions of Bicyclo[2.2.2]oct-2-ene 1 with Various Amines 2 under Aqueous and Neat Conditions with Microwave Irradiation at 160 °C

<table>
<thead>
<tr>
<th>Entry</th>
<th>Amine 2 (R₁)</th>
<th>Time (min)</th>
<th>Aqueous product 3</th>
<th>Yield* (%)</th>
<th>Time (min)</th>
<th>Neat product 4</th>
<th>Yield* (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>2a</td>
<td>60</td>
<td>3a</td>
<td>90</td>
<td>60</td>
<td>4a</td>
<td>75</td>
</tr>
<tr>
<td>2</td>
<td>2b</td>
<td>60</td>
<td>3b</td>
<td>90</td>
<td>60</td>
<td>4b</td>
<td>70</td>
</tr>
<tr>
<td>3</td>
<td>2c</td>
<td>75</td>
<td>3c</td>
<td>81b</td>
<td>75</td>
<td>4c</td>
<td>78</td>
</tr>
<tr>
<td>4</td>
<td>2d</td>
<td>75</td>
<td>3d</td>
<td>80b</td>
<td>120</td>
<td>4d</td>
<td>72</td>
</tr>
<tr>
<td>5</td>
<td>2e</td>
<td>90</td>
<td>3e</td>
<td>81b</td>
<td>120</td>
<td>4e</td>
<td>71</td>
</tr>
<tr>
<td>6</td>
<td>2f</td>
<td>60</td>
<td>3f</td>
<td>90</td>
<td>–</td>
<td>–</td>
<td>–</td>
</tr>
</tbody>
</table>

* Yield of isolated product.

After crystallization (MeOH–H₂O).

Further scale-up procedure, the use of an open vessel microwave setup and the eventual removal of water that is formed during the reaction process could also be considered.

In addition to the supposed primary function of the liquid additive, i.e. the rinsing of the volatile amines from the upper parts of the reaction vessel, thus enabling greater conversions with a smaller excess of 2, it seems that it has another important role. With small amounts of toluene (100 mg), it might be possible that water, eliminated during the reaction, is relatively quickly evaporated and deposited on the uppermost, coldest parts of the reaction vessel. 4-Chloroaniline (2e), with a higher boiling point (232 °C) than water, is presumably deposited slightly lower down in the vessel; from there it can be effectively rinsed by toluene, whereas the water probably remains mostly on the uppermost part of the vessel, where it is unavailable for the possible hydrolysis of the imine produced. When the amount of toluene is increased, the reaction takes place in solution and toluene can effectively rinse both the amine and water from the upper parts of the vessel back into the reaction mixture. Water can, thus, at least partially prevent the formation of the imine or eventually hydrolyze the formed imine and, therefore, 3c is also observed as one of the products.

In addition to aniline (2a) and 4-chloroaniline (2e), the reaction runs chemoselectively under conditions analogous to those described above [neat, 160 °C, toluene (100 mg)] with other substituted anilines to give the corresponding imine products, e.g. 4b, 4d, and 4e (Table 1, entries 2, 4, and 5, respectively).

Both sets of conditions complement each other nicely and products 3 or 4 can be obtained with complete selectivity under azeotropic or neat reaction conditions, respectively.

The aqueous reaction conditions can also be extended to the transformation of substituted fused succinic anhydride derivatives of bicyclo[2.2.2]oct-2-ene 5a–d with a variety of nonconjugated amines 2f–h. The reactions take place readily in water, yielding the corresponding products 6a–e (Table 2). However, an attempt to use such nonconjugated amines 2f–h for the reaction of 1 yielding products similar to 4 was unfortunately not successful. This was confirmed by the mass spectra of the crude reaction mixtures; analysis using the 1H NMR spectra was not possible because of the large number of overlapping signals. The fact that imine-type products 4 were not formed could be attributed to the lower stability of the imine moiety when an aliphatic residue is bound to the imine nitrogen. It is a well-known fact that aromatic groups stabilize the imine functionality, thus preventing its hydrolysis back to the starting amine and ketone. It is also well known that imines can exist as both the syn- and anti-isomers, but in our cases the imines formed are most probably in only one form as their 1H NMR spectra show only one set of signals. We suppose that they exist as anti-isomers because such an isomer would be less sterically crowded with the aromatic and sterically demanding bicyclo[2.2.2]oct-2-ene groups further apart.

In conclusion, the demonstrated methods were successful for the transformation of the fused anhydride moiety of 1 and 5a–d, and also of a carbonyl group in 1, with aniline (2a) and its derivatives 2b–h in a selective manner. The reaction of 1 gave two types of product, imines 4 and compounds with an unreacted acetyl group 3, depending on the reaction conditions used (neat or aqueous, respectively). The use of water as a liquid medium or neat reaction conditions and microwave irradiation as the energy source render this synthesis ecofriendly, and these reaction conditions concomitantly simplify the isolation of the products to give them in good to high yields.

Melting points were determined on a Kofler micro hot stage and are uncorrected. 1H NMR spectra were recorded with a Bruker Avance DPX 300 spectrometer at 29 °C using TMS as an internal standard. 13C NMR spectra were recorded on the same instrument and were

Table 2 Reactions of Various Bicyclo[2.2.2]oct-2-enes 5 with Amines 2 under Aqueous Conditions with Microwave Irradiation at 160 °C

<table>
<thead>
<tr>
<th>Entry</th>
<th>Substrate 5</th>
<th>Amine 2</th>
<th>Time (min)</th>
<th>Product 6</th>
<th>Yielda (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>5a</td>
<td>R2</td>
<td>R1NH2</td>
<td>2f</td>
<td>3-pyridylmethyl</td>
</tr>
<tr>
<td>2</td>
<td>5b</td>
<td>Me</td>
<td>4-MeOC6H4</td>
<td>H</td>
<td>2f</td>
</tr>
<tr>
<td>3</td>
<td>5c</td>
<td>2-furyl</td>
<td>H</td>
<td>H</td>
<td>2f</td>
</tr>
<tr>
<td>4</td>
<td>5d</td>
<td>Ph</td>
<td>H</td>
<td>H</td>
<td>2g</td>
</tr>
<tr>
<td>5</td>
<td>5b</td>
<td>Me</td>
<td>4-MeOC6H4</td>
<td>H</td>
<td>2h</td>
</tr>
</tbody>
</table>

a Yield of isolated product.
referred against the central line of the solvent signal (DMSO-d$_6$, \( \delta = 39.5 \) ppm). Infrared spectra were obtained with a BioRad FTS 3000MX spectrophotometer using KBr pellets for all products. Mass spectra were recorded with a VG-Analytical Auto Spec Q instrument. Elemental analyses were performed with a Perkin Elmer 2400 Series II CHNS/O Analyzer. Thin-layer chromatography was carried out on Fluka silica gel TLC cards. The starting compounds 1 and 5a–d were prepared according to the published procedures, with all other reagents and solvents used as received from commercial suppliers. The reactions were conducted in air using a focused MW unit (Discover by CEM Corporation, Matthews, NC). The machine had a continuous, focused microwave power delivery system with an operator-selectable power output of 0–300 W. Reactions were performed in 10-mL glass vessels sealed with a septum. The pressure was controlled by a load cell connected to the vessel via the septum. The temperature of the contents of the vessel was monitored using a calibrated IR temperature controller mounted under the vessel. All the mixtures were stirred with a Teflon-coated magnetic stir bar. Temperature, pressure, and power profiles were recorded and commercially available software provided by the manufacturer of the MW unit.

**Imides 3a–f and 6a–e Using Aqueous Conditions; General Procedure**

A mixture of fused succinic anhydride derivative 1 (211.5 mg, 0.5 mmol) or 5a–d (0.5 mmol) and amine 2a–h (1.2 mmol) in distilled H$_2$O (2 mL) was irradiated using the focused MW equipment for a period of 50–90 min (see Table 1 and 2). The temperature was set to 160 °C; the power to 170 W, and the ramp time to 5 min. After the reaction was complete, the mixture was cooled, and the precipitated solid was filtered off and washed with distilled H$_2$O (1–3 mL).

**Imines 4a–e Using Neat Conditions; General Procedure**

A mixture of fused succinic anhydride 1 (211.5 mg, 0.5 mmol), amine 2a–e (2 mmol), and toluene (dried and stored over Na, 100 mg) was irradiated using the focused MW equipment for a period of 60–120 min (see Table 1). The temperature was set to 160 °C, the power to 170 W, and the ramp time to 5 min. After the reaction was complete, the mixture was cooled and EtOH–H$_2$O (3:1, 1–2 mL) was added. The suspension was gently heated, thoroughly triturated, and cooled; the precipitated solid was filtered off and washed with EtOH–H$_2$O (3:1, 0.5–1 mL).

**N-(9-Acetyl-2,6-bis(4-chlorophenyl)-4,8-etheno-8-methyl-1,3,5,7-tetraoxododecahydropyrrolo[3,4-f][isoindol-4-yl]benzamide (3c)**

Yield: 261.5 mg (81%); off-white powder; mp 290–292 °C (MeOH–H$_2$O).

**IR (KBr):** 3414 (br), 1775, 1720, 1648, 1536, 1495 cm$^{-1}$.

**1$^1$H NMR (300 MHz, DMSO-d$_6$):** \( \delta = 1.95 \) (s, 3 H, Me), 2.25 (s, 3 H, Me), 2.31 (s, 6 H, 2 Me), 3.36 (d, \( J = 8.3 \) Hz, 2 H, H-7a, H-8a), 4.54 (d, \( J = 8.3 \) Hz, 2 H, H-3a, H-4a), 7.14 and 7.56 (AA’XX’, \( J = 8.7 \) Hz, 8 H, 2 × 4-CIC$_6$H$_4$), 7.35 (s, 1 H, H-10), 7.45–7.57 (m, 3 H, Ph), 7.83–7.89 (m, 2 H, Ph), 8.82 (s, 1 H, NH).

**1$^3$C NMR (75.5 MHz, DMSO-d$_6$):** \( \delta = 17.7, 28.2, 41.3, 43.6, 49.0, 57.9, 127.1, 127.4, 128.1, 128.7, 129.1, 131.2, 131.9, 135.4, 137.6, 138.5, 143.7, 173.4, 174.6, 197.0.

**MS (EI):** \( m/z \% = 601 (3) [M]^+$, 69 (100).**

Anal. Calcd for C$_{36}$H$_{31}$N$_3$O$_6$: C, 71.87; H, 5.19; N, 6.98. Found: C, 71.55; H, 5.25; N, 6.96.
Yield: 292 mg (78%); white powder; mp 268–271 °C (MeOH–H₂O).

Yield: 244 mg (75%); white crystals; mp 259–261 °C (MeOH–H₂O).

Anal. Calcd for C₃₄H₂₉N₅O₆: C, 67.65; H, 4.84; N, 11.60. Found: C, 67.3; H, 4.85; N, 11.53.

N-[4-(8-ethoxy-8-methyl-1,3,5,7-tetraoxo-2,6-diphenyl-9-1H isoindolylmethyl)ethyl]dodecahydropyrrolo[3,4-f]isoindol-4-yl]benzamide (4a)

Yield: 244 mg (75%); white crystals; mp 259–261 °C (MeOH–H₂O).

IR (KBr): 3470 (br), 1772, 1716, 1642, 1542, 1491 cm⁻¹.

Anal. Calcd for C₄₀H₂₉F₃N₄O₅·0.5H₂O: C, 67.51; H, 4.25; N, 7.87. Found: C, 67.4; H, 4.52; N, 7.87.

IR (KBr): 3289 (br), 1769, 1705, 1682, 1659, 1600, 1548 cm⁻¹.

IR (KBr): 3462 (br), 1773, 1713, 1633, 1550, 1491 cm⁻¹.

N-[4-(8-ethoxy-8-methyl-1,3,5,7-tetraoxo-2,6-di-3-tolyl-9-1H isoindolylmethyl)ethyl]dodecahydropyrrolo[3,4-f]isoindol-4-yl]benzamide (4b)

Yield: 242 mg (70%); white powder; mp 277–279 °C (EtOH).

IR (KBr): 3462 (br), 1773, 1713, 1633, 1550, 1491 cm⁻¹.

IR (KBr): 3356, 1768, 1713 (br), 1663, 1595, 1495 cm⁻¹.

N-[4-(8-ethoxy-8-methyl-1,3,5,7-tetraoxo-2,6-di-3-tolyl-9-1H isoindolylmethyl)ethyl]dodecahydropyrrolo[3,4-f]isoindol-4-yl]benzamide (4c)

Yield: 251 mg (71%); white crystals; mp 284–286 °C (MeOH–H₂O).

IR (KBr): 3465 (br), 1774, 1713, 1670, 1605, 1545, 1480 cm⁻¹.

N-[4-(8-ethoxy-8-methyl-1,3,5,7-tetraoxo-2,6-di-3-tolyl-9-1H isoindolylmethyl)ethyl]dodecahydropyrrolo[3,4-f]isoindol-4-yl]benzamide (4d)

Yield: 292 mg (78%); white powder; mp 268–271 °C (MeOH–H₂O).

IR (KBr): 3470 (br), 1772, 1716, 1642, 1542, 1491 cm⁻¹.

N-[2,6-Bis(4-chlorophenyl)-9-1H 4-chlorophenyl]liminoethyl]-4-ethoxy-8-methyl-1,3,5,7-tetraoxodecachrysindro[3,4-f]isoindol-4-yl]benzamide (6a)

Yield: 270 mg (85%); white powder; mp 259–262 °C (EtOH).

IR (KBr): 3456 (br), 1765, 1700, 1648, 1529 cm⁻¹.

N-[2,6-Bis(4-chlorophenyl)-9-1H 4-chlorophenyl]liminoethyl]-4-ethoxy-8-methyl-1,3,5,7-tetraoxodecachrysindro[3,4-f]isoindol-4-yl]benzamide (6b)

Yield: 307 mg (72%); white powder; mp 313–316 °C (MeOH–H₂O).

MS (ESI+): m/z = 753 [M + H⁺].

Anal. Calcd for C₃₀H₂₆Cl₂N₄O₄·H₂O: C, 43.86; H, 3.42; N, 7.19. Found: C, 43.5; H, 3.5; N, 7.2.

N-[2,6-Bis(4-chlorophenyl)-9-1H 4-chlorophenyl]liminoethyl]-4-ethoxy-8-methyl-1,3,5,7-tetraoxodecachrysindro[3,4-f]isoindol-4-yl]benzamide (6c)

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Yield: 307 mg (72%); white powder; mp 313–316 °C (MeOH–H₂O).

MS (ESI+): m/z = 753 [M + H⁺].

Anal. Calcd for C₃₀H₂₆Cl₂N₄O₄·H₂O: C, 43.86; H, 3.42; N, 7.19. Found: C, 43.5; H, 3.5; N, 7.2.
1 H, NH), 8.33 (dd, J = 7.2 Hz, 2 H, 2 H-7a), 8.42 (d, J = 8.1 Hz, 2 H, H-7a, H-8a), 4.43 (s, 4 H, 2 CH2), 4.52 (d, J = 4.8 Hz, 2 H, H-3a, H-4a), 6.40–6.47 (m, 2 H, 2 H of furyl), 6.51 (AB, J = 9.0 Hz, 2 H, H-9, H-10), 7.31 (dd, J = 4.8 Hz, 2 H, 2 H-5’), 7.46–7.58 (m, 5 H, Ph, 2 H-4’), 7.84–7.90 (m, 2 H, Ph), 8.36 (d, J = 1.8 Hz, 2 H, 2 H-2’), 8.45 (dd, J1 = 1.5 Hz, J2 = 4.8 Hz, 2 H, H-6’), 8.83 (s, 1 H, NH).

13C NMR (75.5 MHz, DMSO-d6): δ = 39.1, 42.7, 43.4, 47.9, 58.0, 108.0, 110.5, 123.5, 127.6, 128.1, 129.4, 131.1, 131.6, 132.4, 135.1, 135.6, 142.2, 148.60, 148.61, 151.5, 168.2, 173.9, 174.3.

MS (ESI+): m/z = 614 [M + H]+, 426.

Anal. Calcld for C39H31N3O5·0.5H2O: C, 74.27; H, 5.11; N, 6.66.

Acknowledgment
We thank the Ministry of Higher Education, Science, and Technology of the Republic of Slovenia as well as the Slovenian Research Agency for financial support (P1-0230-0103, J1-6693-0103, and 1007-0310067). Dr. B. Kralj and Dr. D. Žigon (Center for Mass Spectrometry, Jožef Stefan Institute, Ljubljana, Slovenia) are gratefully acknowledged for mass spectrometry measurements.

References


(3) For selected microwave-assisted reactions in water, see:

(4) For recent selected microwave-assisted neat reactions, see:


