A Study of the Ionic Liquid Mediated Microwave Heating of Organic Solvents

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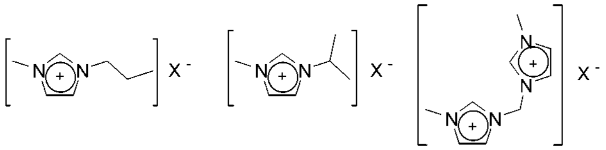
Abstract: The use of ionic liquids as aids for microwave heating of nonpolar solvents has been investigated. We show that hexane and toluene together with solvents such as THF and dioxane can be heated way above their boiling point in sealed vessels using a small quantity of an ionic liquid, thereby allowing them to be used as media for microwave-assisted chemistry. Using the appropriate ionic liquid, the heating can be performed with no contamination of the solvent. To show the applicability of the system, two test reactions have been successfully performed.

The use of microwave ovens as tools for synthetic chemistry is a fast growth area.[1,2] Since the first reports of microwave-assisted synthesis in 1986,[3,4] the technique has been accepted as a method for reducing reaction times often by orders of magnitude and for increasing yields of product compared to conventional methods.[5,6] As a result, this has opened up the possibility of optimizing new reactions in a very short time. A key advantage of modern scientific microwave apparatus is the ability to control reaction conditions very specifically, monitoring temperature, pressure, and reaction times. Several methods have been developed for performing reactions using microwaves including using solvent-free conditions or adsorbing reactants onto inorganic supports such as silicas or clays. If the reaction needs to be carried out in a solvent, the medium needs to have a high dielectric constant (ϵ) in order to take advantage of the microwave heating effect. To this end, solvents such as 1-methyl-2-pyrrolidone (NMP) ($\epsilon = 32.2$), DMSO ($\epsilon = 46.7$), DMF ($\epsilon = 36.7$), and other high boiling polar solvents are often used. Although these are excellent solvents for performing the reaction, the subsequent workup procedure is complicated by the need to remove the solvent at the end of the reaction. In addition, there are situations where it would be desirable to perform the reaction in less polar solvents such as hexane ($\epsilon = 1.88$), toluene ($\epsilon = 2.38$), THF ($\epsilon = 7.58$), and dioxane ($\epsilon = 2.21$). However, with their substantially lower dielectric constants these are clearly less suitable for use in microwave-assisted synthesis. In their microwave-assisted synthesis of thiocarbonyls using a polymer-supported thionating reagent, Ley et al. show that addition of a small quantity of an ionic liquid to a toluene solution can greatly increase the rate and yields of reaction.[7] Ionic liquids made of organic cations and appropriate anions have attracted much recent attention as solvents for chemistry because of the fact that they have melting points close or near to room temperature.[8] It has also been shown that they can be used as reactants.[9] They have negligible vapor pressure and are immiscible with a range of organic solvents meaning that organic products can be easily removed and the ionic liquid can be recycled. From the perspective of microwave chemistry one of the points of key importance is their high polarity and that this is variable depending on the cation and anion so can effectively be tuned to a particular application. Despite this, application of microwave chemistry to reactions using ionic liquids has not been exploited on many occasions.[10]



1 X = I
4 X = PF$_6$
7 X = BF$_4$

2 X = Br
5 X = PF$_6$

3 X = I
6 X = PF$_6$

Figure 1. Ionic liquids used in the studies.

In this note we report the results of our investigations building on the idea of using ionic liquids as aids for microwave heating of nonpolar solvents. We show that hexane and toluene as well as with solvents such as THF and dioxane can be heated way above their boiling point in sealed vessels using a small quantity of an ionic liquid, thereby allowing them to be used as media for microwave-assisted chemistry. Our attention has focused not only on the heating effects but also on studying the contamination, if any, of the parent solvent with the ionic liquid or any decomposition products formed as they are heated. In such cases, there would clearly be a disadvantage to using this route, especially if the decomposition products interact with the reaction mixture to stop the reaction or lead to unwanted byproducts.

Our starting point was to prepare and screen a range of 1,3-dialkylimidazolium halides (Figure 1 entries **1**, **2**, and **3**) for the heating of organic solvents. The ionic liquids were prepared by literature methods from alkyl halides and *N*-methylimidazole. **1** and **2** were prepared

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Table 1. The Microwave Heating Effects of Adding a Small Quantity of 1 and 2 to Hexane, Toluene, THF, and Dioxane[a]

solvent used	ionic liquid added	temp attained, °C	time taken, s	*T* without ionic liquid, °C[b]	boiling point, °C[c]
hexane	**1**	217	10	46	69
	2	228	15		
toluene	**1**	195	150	109	111
	2	234	130		
THF	**1**	268	70	112	66
	2	242	60		
dioxane	**1**	264	90	76	101
	2	246	90		

[a] Experiments run using a microwave irradiation power of 200 W. [b] Temperature attained during the same microwave irradiation time but without any ionic liquid added. [c] Boiling point of the solvent used (for comparison purposes).

by heating a toluene solution of methyl imidazole and alkyl halide,[11] and **3** was prepared by refluxing diiodomethane and methylimidazole in THF for 2 days.[12] When purified, **1** and **2** are liquids at room temperature while **3** is a solid, melting at 260 °C.

Once prepared and purified, the microwave heating properties of the ionic liquids were investigated in the absence of any other solvent. Reactions were performed in sealed vessels so that any pressure build-up could be monitored. With **1** and **2**, using 100 W irradiation power, the preselected maximum temperature of 300 °C was reached within 15 s. For such rapid heating in common organic solvents, the term microwave flash heating has recently been introduced by Hallberg and co-workers.[13] To avoid the formation of hot-spots, the irradiation of the ionic liquids was accompanied by rapid stirring using a Teflon-coated stirring bar. The fact that ionic liquids have negligible vapor pressure even at elevated temperatures was clearly evidenced in our experiments, no significant pressure rise being noted during the course of the irradiation. These results indicate that ionic liquids may offer an alternative to solvents such as NMP[6] and DMF[14] for flash heating organic compounds. In the case of **3**, using 300 W irradiation power, it took 4 min to reach the preselected maximum temperature of 300 °C. This lengthy time is because the solid first has to start to melt. Once melting has started, the temperature rise is very dramatic, moving from 200 °C to 300 °C within 2 s.

Using **1** and **2**, the microwave heating effects of adding a small quantity of ionic liquid to solutions of hexane, toluene, THF, and dioxane were investigated. The temperatures attained and time taken to reach this point are tabulated in Table 1. As a comparison, temperatures reached in the absence of ionic liquids are also shown. The experiments were run using 0.2 mmol (between 10 and 55 mg) of ionic liquid and 2 mL of solvent. All experiments were run using an irradiation power of 200 W. From the table it is clearly seen that the ionic liquids have a dramatic effect on the heating of all the solvents studied, temperatures well in excess of the boiling points being achieved rapidly. **CAUTION!!**[15] The effects of varying the quantity of ionic liquid used in the solvent heating was investigated. We found that for heating 2 mL of solvent, 0.2 mmol ionic liquid was the optimal amount, any more resulting in too rapid heating and the heating being too slow when using any less.

To determine whether there is any contamination of the solvent by the ionic liquid, the solution was decanted off from the ionic liquid and the solvent removed under vacuum. The flask was then washed with a small volume of $CDCl_3$ and the ^1H NMR of the whole concentrate recorded. In all cases some level of contamination was found, the level being directly correlated to the temperature attained in the microwave heating experiment. Further investigation showed that the contamination was coming from decomposition of the ionic liquids over time at elevated temperatures. We find that although **1** and **2** can be used for microwave heating of solvents to around 160 °C, increasing the temperature much above this leads to the onset of some decomposition with major decomposition occurring above 230 °C. Our studies show that the ionic liquids decompose to give methyl iodide and *n*-propylimidazole in the case of **1**, and methyl bromide and isopropylimidazole in the case of **2**. This decomposition at elevated temperatures is not totally unexpected. Vacuum pyrolysis of 1-methyl-3-alkylimidazolium halides at between 300 and 600 °C leads to a similar decomposition, again forming the corresponding methyl halide and alkylimidazole.[16] In all these cases, the halide ion, X^-, presumably acts as a nucleophile in attacking the cation with the subsequent elimination of MeX.

To avoid problems of contamination by decomposition to alkyl halides and alkyl imidazoles, a range of other ionic liquids were prepared by anion metathesis of **1**, **2**, and **3**. Using literature methods, treatment of **1**, **2**, or **3** in water with HPF_6 leads readily to the corresponding PF_6-coordinated ionic liquids **4**, **5**, and **6**, respectively.[17] Treatment of **1** with $NaBF_4$ leads to the corresponding BF_4 coordinated ionic liquid **7**.[18] The PF_6- and BF_4-coordinated ionic liquids **4**−**7** would be expected to be more thermally stable to decomposition and hence may prove more useful in microwave heating experiments. To assess their potential, again the microwave heating effects of adding a small quantity of ionic liquid to solutions of hexane, toluene, THF, and dioxane were investigated. As with **1** and **2**, dramatic heating effects were observed with **4**, **5**, and **7**. A summary of the data is shown in Table 2. The results show that both **4** and **7** prove useful in microwave heating of solvents, with **4** being more effective. Indeed it was necessary to reduce the microwave power to half that used in the other experiments (i.e., 100 W) in order to stop the solvents reaching temperatures well in excess of 300 °C within a few seconds. There is no contamination[19] of the solvent

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[15] The solvents are heated well above their boiling points so all necessary precautions should be taken when performing such experiments. Vessels designed to withhold elevated pressures must be used. The microwave apparatus used here incorporates a protective metal cage around the microwave vessel in case of explosion. After completion of an experiment, the vessel must be allowed to cool to a temperature below the boiling point of the solvent before removal from the microwave cavity and opening to the atmosphere.

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Table 2. The Microwave Heating Effects of Adding a Small Quantity of 4, 5, and 7 to Hexane, Toluene, THF, and Dioxane[a]

solvent used	ionic liquid added	temp attained, °C	time taken, s	level of contamination
hexane	**4**	279	20	none
	5	90	300	none
	7	192	60	none
toluene	**4**	280	60	none
	5	79	120	none
	7	165	90	contaminated
THF	**4**	231	60	none
	7	95	50	contaminated[b]
dioxane	**4**	149	100	none
	7	184	120	contaminated

[a] Experiments run using a microwave irradiation power of 100 W. [b] **7** is slightly soluble in THF and so cannot totally be removed; so contamination is due to **7** rather than decomposition.

Table 3. The Microwave Heating Effects of Adding a Small Quantity of Premelted and Cooled 5 and 6 to Hexane, Toluene, THF, and Dioxane[a]

solvent used	ionic liquid added	temp attained, °C	time taken, s	level of contamination
hexane	**5**	243	40	none
	6	300	120	none
toluene	**5**	278	50	none
	6	291	120	none
THF	**5**	181	60	contaminated[b]
	6	181	120	none
dioxane	**5**	209	60	contaminated[b]
	6	233	120	none

[a] Experiments run using a microwave irradiation power of 100 W. [b] Contamination is due to trace of **5** in solution rather than decomposition.

when using **4** with any of the solvents screened or when **7** is used with hexane. There is contamination due to decomposition of **7** when used with toluene or dioxane, the extent being much less in the case of the latter. **7** is slightly soluble in THF, so in this case the only source of contamination at the end of the heating experiments is a trace of the parent ionic liquid rather than any decomposition.

When using freshly prepared samples of **5** and **6** for microwave heating experiments, poor results are obtained. This is because they are higher melting ionic liquids (mp s 80 °C and 185 °C, respectively, for **5** and **6**) and, in the presence of the solvents, do not get to a significant temperature at which they can melt. In a related set of experiments we found that if a freshly prepared sample of **5** is heated to melting point on its own in the microwave and then allowed to cool back to room temperature, then it undergoes a phase change and melts at a much lower temperature on heating for a second time. A similar observation was made with **6**.[20] We therefore assessed the heating capabilities of samples of **5** and **6** which had been melted and then cooled before adding the solvent of choice. As shown in Table 3, the effects are significant offering the best microwave heating of all the ionic liquids screened. Again there is little or no decomposition of the ionic liquids and hence little or no contamination of the solvent.

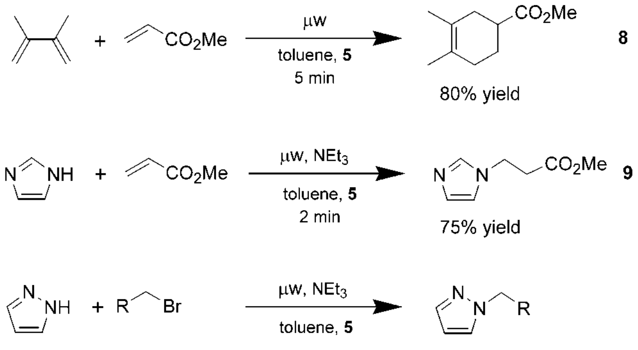

Figure 2. Reactions studied for assessment of the microwave heating method for synthesis.

To show that these ionic liquids can be used to heat solvents for extended times, samples of hexane and toluene containing **4** was heated to 300 °C and held at this temperature for 30 min. Analysis of the solvent at the end of the heating showed no contamination.

Ley et al. have already demonstrated the potential of the ionic liquid heating method in synthesis,[7] but to expand on this we have screened three further reactions to show the applicability of the methodology (Figure 2). The first reaction chosen was the Diels−Alder reaction between 2,3-dimethylbutadiene and methyl acrylate to give the [4 + 2] adduct **8**.[21] This reaction is traditionally performed in toluene or xylene and takes between 18 and 24 h to reach completion, giving yields of **8** varying from 9 to 90% depending on the solvent used and the temperature at which the reaction is run.[22,23,24] Even when using catalysts such as $AlCl_3$, the reaction still takes 5 h to reach 73% yield.[25] Using a mixture of toluene and **5** we were able to prepare **8** in 5 min in 80% yield, this offering a significant rate enhancement over the conventional methods. During the reaction, a microwave power of 100 W was used and the temperature of the mixture held at 200 °C. The NMR spectrum of the crude product shows that the ionic liquid used does not decompose during the reaction or contaminate the product mixture. In a control experiment, the reaction was repeated in the absence of **5**, and it was found that after the same time (5 min at 100 W microwave power) there was no product formed. Interestingly this same reaction has been reported previously using microwave irradiation with xylene as solvent, but the time taken to reach the same level of product yield is well in excess of 3 h, presumably because the reaction mixture was heated only to 95 °C.[26,27]

The second reaction chosen for study was the base-catalyzed Michael addition of methyl acrylate to imidazole to yield **9**. We chose this reaction, as we thought it would be a test for whether the ionic liquid was indeed acting solely as a heater in the reaction rather than

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(19) Experiments suggest that NMR is sensitive to very low levels of contaminants so is a useful assay tool.

(20) Such phase changes have also been noted with other higher melting point ionic liquids. See for example: Larsen, A. S.; Holbrey, J. D.; Tham, F. S.; Reed, C. A. *J. Am. Chem. Soc.* **2000**, *122*, 7264.

(21) It should be noted that rate acceleration in other Diels−Alder reactions via microwave irradiation has been observed before, but the synthetic procedure is often not as convenient as presented here. For examples, see refs 1a and 1b.

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becoming chemically involved, as there would be scope for reaction between the ionic liquid and the base or acrylate during the course of the reaction. This reaction has already been performed using microwave irradiation, using basic clays (Li^+ and Cs^+ montmorillonites) as catalysts.[28] We replaced the clays by triethylamine and found that using a mixture of toluene and **5** we were able to prepare **9** in 2 min in an isolated yield of 75%. During the reaction, a microwave power of 100 W was used and the temperature of the mixture held at 200 °C. Again, the NMR spectrum of the crude product mixture shows that the ionic liquid used does not decompose during the reaction or contaminate the product mixture. This result is interesting in that it shows that we could not only improve reaction times and yields over the conventional methods (no product observed after this time) but also over the previously reported microwave method (yields of 40−50% being obtained after 3 min irradiation). In a control experiment, the reaction was repeated first in the absence of **5** and toluene and second in the absence of toluene, and in both cases it was found that after the same time (2 min at 100 W microwave power) there was no product formed.

The third reaction studied was the alkylation of pyrazoles with alkyl halides. This again is a reaction that has been studied previously using microwave irradiation.[29] We find that this reaction is not possible using our experimental protocol, the aryl halide reacting with the ionic liquid at the elevated temperatures used in the reaction. Although we have not managed to characterize the reaction products, it is clear to see that all the ionic liquid is destroyed since the biphasic starting mixture (solvent and ionic liquid) becomes a monophasic mixture after just a few seconds of microwave irradiation. This shows the limitations of our protocol; it not being possible to undertake reactions which use or generate nucleophiles such as halide ions. Although this removes a number of possible reactions from the synthetic chemist's portfolio, the success of the other reactions does show the potential of the technique to preparative chemistry.

In conclusion, we have shown that hexane and toluene together with solvents such as THF and dioxane can be heated way above their boiling point in sealed vessels using a small quantity of an ionic liquid, thereby allowing them to be used as media for microwave-assisted chemistry. With careful choice of ionic liquid, contamination of the solvent can be avoided. In the cases of the solid ionic liquids studied we find that if they are melted and allowed to cool they undergo a phase change and melt at a much lower temperature on heating for a second time and can be very powerful microwave heating agents. We have shown the potential of this heating method to synthesis and believe that it could offer significant advantages to preparative chemists attempting to improve rates and yields of reactions best run in nonpolar solvents.

Experimental Section

General. Microwave experiments were conducted using a CEM Discover Synthesis Unit (CEM Corp., Matthews, NC).[30] The machine consists of a continuous focused microwave power delivery system with operator selectable power output from 0 to 300 W. Reactions were performed in glass vessels (capacity 10 mL) sealed with a septum. The pressure is controlled by a load cell connected to the vessel via a 14-gauge needle which penetrates just below the septum surface. The temperature of the contents of the vessel was monitored using a calibrated infrared temperature control mounted under the reaction vessel. Dried solvents were used in every case. All experiments were performed using a stirring option whereby the contents of the vessel are stirred by means of a rotating magnetic plate located below the floor of the microwave cavity and a Teflon-coated magnetic stir bar in the vessel. All chemicals were reagent grade and used as purchased. The ^1H and $^{31}P\{^1H\}$ NMR spectra were recorded at 250 MHz and 293 K and referenced to TMS and H_3PO_4 respectively.

Representative Procedure for Study of the Microwave Heating Effects of Ionic Liquids. A small quantity of ionic liquid (20−50 mg) was placed in a microwave vessel containing a Teflon-coated magnetic stirrer bar. To this was added 2 mL of dried solvent of choice. The vessel was then sealed with a septum and the experiment undertaken. For safety, the maximum pressure and temperature were set to 200 bar and 300 °C, respectively. A microwave irradiation power of either 100 or 200 W was used for the experiments depending on the ionic liquid used. The power used was governed by the results of experiments heating the ionic liquid alone, the power being ramped up in increments until a suitable and controllable rate of heating was observed.

Preparation of 8. To a microwave vessel containing 2 mL of toluene and **5** (55 mg) was added 2,3-dimethylbutadiene (0.23 mL, 2 mmol) and methyl acrylate (0.18 mL, 2 mmol). A Teflon-coated magnetic stirrer bar was added and the vessel sealed. The sample was irradiated for 5 min using a power of 100 W. For safety, the maximum pressure was set to 200 bar. After being cooled to room temperature, the vessel was opened and the toluene layer removed using a pipet. The ionic liquid layer was washed with toluene, and these washings were combined with the original toluene layer. Removal of the solvent resulted in a light yellow oil, the NMR spectrum of which showed product but no evidence for ionic liquid or its decomposition products. A yield of **8** of 80% was obtained, the product being chracterized by comparison of ^1H and ^{13}C NMR data with that in the literature.[31]

Preparation of 9. To a microwave vessel containing 2 mL of toluene and **5** (55 mg) were added imidazole (135 mg, 2 mmol), methyl acrylate (0.18 mL, 2 mmol), and triethylamine (0.28 mL, 2 mmol). A Teflon-coated magnetic stirrer bar was added and the vessel sealed. The sample was irradiated for 2 min using a power of 100 W. For safety, the maximum pressure was set to 200 bar. After cooling to room temperature, the vessel was opened and the toluene layer removed using a pipet. The ionic liquid layer was washed with toluene, and these washings were combined with the original toluene layer. Removal of the solvent resulted in a light yellow residue, the NMR of which showed a mixture of starting materials and product but no evidence for ionic liquid or its decomposition products. Washing the yellow residue with acetone led to a light yellow oil, characterized as **9** by comparison of ^1H and ^{13}C NMR data with that in the literature (150 mg, 75%).[32]

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Supporting Information Available: Syntheses and spectroscopic data for the ionic liquids used in these studies (**1**−**7**). This material is available free of charge via the Internet at http://pubs.acs.org.

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