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Synthesis of the novel ionic liquid [*N*-pentylpyridinium]$^+$ [*closo*-$CB_{11}H_{12}$]$^-$ and its usage as a reaction medium in catalytic dehalogenation of aromatic halides†

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The novel, low-melting-point (19 °C) salt [*N*-pentylpyridinium]$^+$ [*closo*-$CB_{11}H_{12}$]$^-$ (2) was synthesized in 93% yield and characterized by elemental analysis, IR spectroscopy, and ^1H, ^{13}C, and ^{11}B NMR spectroscopy. The salt was used as a solvent in several dehalogenation reactions of mono- and poly-chlorides and -bromides, catalyzed by the palladium complexes $PdCl_2(PPh_3)_2$, $PdCl_2$(dppe), and $PdCl_2$(dppf). Complete debromination of C_6Br_6, 1,2,4,5-tetrabromobenzene, $C_{60}Br_8$, and $C_{60}Br_{24}$ was accomplished quite rapidly, whereas dechlorination of 1,2,4-trichlorobenzene proceeded more slowly, but with excellent products selectivity. Copyright © 2003 John Wiley & Sons, Ltd.

KEYWORDS: ionic liquid; polybromofullerenes; polyhalobenzenes; dehalogenation; palladium complexes

INTRODUCTION

Recently, there has been a great deal of interest in developing low-melting-point salts that can function as solvents in organic reactions.[1–3] Such solvents would have the advantage of offering a unique reaction environment, as well as offering practical advantages for distillative product separation and low evaporation rates. The search for suitable benign cation–anion pairs has led to the use of the so-called noncoordinating anions $[ClO_4]^-$, $[SbF_6]^-$, $[CF_3OSO_2]^-$, $[BF_4]^-$, and $[BPh_4]^-$. However, even these have shown reactivity in low dielectric-constant solvents.[4–10] The monoanionic icosahedral cluster 1-carba-*closo*-dodecaborate, (1-) $[CB_{11}H_{12}]^-$, has been shown to be one of the most inert coordinating anions hitherto known.[11] It has no lone pairs of electrons, no basic or nucleophilic sites, is oxidatively stable, and its salts are reasonably soluble in low dielectric-constant solvents.[11] All of these properties make it attractive as a possible anion in ionic liquid solvents.

The melting points of ionic compounds depend on the nature of the cations and anions involved, with packing inefficiency being conducive to lower melting points. As a result, certain cations, such as the imidazolium and N,N'-dialkylimidazolium cations, have been extensively studied.[1–3] Reed and coworkers[12] have described the synthesis of ionic liquids derived from imidazolium salts of carborane anions, with the melting point down to 45 °C. They predicated the possibility of making those room-temperature ionic liquids by suitable modification of corresponding imidazolium cations.[12] Although alkylpyridinium cations were mentioned by the authors, none was included in their studies. We were quite surprised to find that when alkylpyridinium cations were combined with the carborane anion, $[CB_{11}H_{12}]^-$, an extremely low melting-point salt was obtained in high yield. Herein, we report the synthesis and characterization of a new ionic solvent, [*N*-pentylpyridinium]$^+$ [*closo*-$CB_{11}H_{12}$]$^-$, as well as some initial results on the catalytic dehalogenation of polychlorobenzenes, polybromobenzenes and polybromofullerenes in this solvent. The dehalogenation reactions were chosen since their products have been shown to be highly solvent dependent.[13–20]

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EXPERIMENTAL

All synthetic procedures were carried out in an inert atmosphere, using standard Schlenk techniques. Tetrahydrofuran (THF), diethyl ether, hexane, and *N*,*N*,*N*′,*N*′-tetramethyl-1,2-ethylenediamine (TMEDA) were heated over sodium and benzophenone until a dark blue color was obtained, and then distilled under nitrogen just before use. Dichloromethane and chloroform were dried over phosphorus pentoxide and distilled. Pyridine was dried with $NaNH_2$ and distilled. 1-Bromopentane, chlorinated benzenes, brominated benzenes, $PdCl_2(PPh_3)_2$, $PdCl_2$(dppe), and other reagents were obtained from Aldrich. $PdCl_2$(dppf),[21] $C_{60}Br_8$,[22] $C_{60}Br_{24}$,[23,24] and $Cs[B_{11}H_{12}]$[25,26] were synthesized according to literature methods. The ^{1}H, ^{13}C, and ^{11}B NMR spectra were measured using a Bruker 400 spectrometer at 400.1 MHz, 100.6 MHz and 128.4 MHz respectively. IR spectra were measured as KBr pellets using a BIO-RAD spectrophotometer. The elemental analyses for carbon, hydrogen and nitrogen were conducted on a Perkin–Elmer 2400 elemental analyzer. The dynamic viscosity was determined using a CV-100 Caulking Viscometer. Gas chromatography (GC)–Mass spectrometry (MS) (Shimadzu QP 5000) was used to confirm peak identity.

Synthesis of *N*-pentylpyridinium bromide ($[N\text{-pentyl-}C_5H_5N]^+\ Br^-$) (1)

In a process similar to that described in the literature,[27,28] a 3.0 ml (23.89 mmol) sample of 1-bromopentane was added to a 50 ml three-necked round-bottom flask containing 10 ml of dry pyridine, and the mixture was heated to reflux with constant stirring for 4 h. After cooling to room temperature, the volatile components were removed under reduced pressure to give the crude product. After recrystallization from pyridine–ether (1 : 3), 5.22 g (95% yield) of purified salt, $[N\text{-pentyl-}C_5H_5N]^+\ Br^-$ **(1)** was obtained.

^{1}H NMR ($CDCl_3$, ppm): δ 9.34 (m, 2H, 2 N–C_{py}*H*), 8.26 (m, 1H, C_{Py}–H), 7.87 (m, 2H, 2C_{Py}–H), 4.69 (t, 2H, N–C*H*$_2$–), 1.75 (m, 2H, N–C–C*H*$_2$–), 1.04 (m, 4H, N–(C)$_2$–C*H*$_2$–C*H*$_2$–), 0.52 (t, 3H, –C*H*$_3$). ^{13}C NMR ($CDCl_3$, ppm): δ 13.79 (*C*–(C)$_4$–N), 22.07 (C–*C*–(C)$_3$–N), 27.94 ((C)$_2$–*C*–(C)$_2$–N), 31.70 ((C)$_3$–*C*–C–N), 61.81 ((C)$_4$–*C*–N), 128.48 (C_{Py}), 145.18 (C_{Py}); one signal for the pyridine ring does not appear or overlaps with others. Anal. Calc. for $C_{10}H_{16}BrN$ (230.149): C, 52.19; H, 7.01; N, 6.09. Found: C, 52.07; H, 7.06; N, 5. 95%.

Synthesis of $[N\text{-pentylpyridinium}]^+$ [*closo*-$CB_{11}H_{12}]^-$ (2)

$[N\text{-Pentyl-}C_5H_5N]^+\ Br^-$ (0.500 g, 2.17 mmol) dissolved in 10 ml of dry CH_2Cl_2 was added to a solution of $Cs[CB_{11}H_{12}]$ (0.600 g, 2.17 mmol) dissolved in 40 ml of dry methanol. The mixture was stirred at room temperature for 20 h and then all solvents were removed. The resulting viscous residue was dissolved in CH_2Cl_2 and purified by column chromatography (SiO_2), with CH_2Cl_2–diethyl ether (v/v = 4/1) as the movable phase. Removal of the solvent under reduced pressure and drying under high vacuum for 2 days resulted in 0.592 g (93% yield) of $[N\text{-pentylpyridine}]^+$ [*closo*-$CB_{11}H_{12}]^-$ (**2**) isolated as a colorless liquid. The liquid solidifies at 19 °C to give **2** as a colorless waxy solid.

^{1}H NMR ($CDCl_3$, ppm): δ 8.98 (m, 2H, 2N–C_{py}*H*), 8.20 (m, 1H, C_{Py}–H), 7.76 (m, 2H, 2 C_{Py}–H), 4.49 (t, 2H, N–C*H*$_2$–), 2.96 (s, 1H, B–C*H*), 1.65 (m, 2H, N–C–C*H*$_2$–), 0.95 (m, 4H, N–(C)$_2$–C*H*$_2$–C*H*$_2$–), 0.45 (t, 3H, –C*H*$_3$), 0.44–3.20 (m, 11H, 11 BH). ^{13}C NMR ($CDCl_3$, ppm): δ 13.37 (*C*–(C)$_4$–N), 21.52 (C–*C*–(C)$_3$–N), 27.41 ((C)$_2$–*C*–(C)$_2$–N), 31.01 ((C)$_3$–*C*–C–N), 52.82 (C–B), 61.34 ((C)$_4$–*C*–N), 128.13 (C_{Py}), 144.29 (C_{Py}), 145.32 (C_{Py}). IR (KBr, cm^{-1}): 2955 (s, m), 2571 (s, s, ν_{B-H}), 1404 (m, br), 1250 (vs, s), 1091 (s, m), 840 (vs, br), 799 (s, s), 758 (s, s), 687 (m, s), 630 (w, s). Dynamic viscosity (20 °C): = 92 cP (0.01 g cm^{-1} s^{-1}, estimated error ±2%). Anal. Calc. for $C_{11}H_{28}B_{11}N$ (293.273): C, 45.05; H, 9.62; N, 4.78. Found: C, 49.97; H, 9.59; N, 4.73%.

Dehalogenation procedure

In a typical reaction, a 1.0 mmol sample of the halogenated substrate (0.1 mmol was used for $C_{60}Br_8$ and $C_{60}Br_{24}$) was put in 10 ml of the ionic liquid, containing the particular palladium catalyst (0.5–1.0 mol% of the substrate). The resulting mixture was then dissolved in THF to obtain a clear solution; all volatile components were removed under reduced pressure. After 10 min of additional stirring, TMEDA (1.0 ml, 6.7 mmol) was slowly added with continual stirring, followed by the careful addition of $NaBH_4$ to give a 2.5 : 1 molar ratio of borohydride-to-substrate (78.0–570.0 mg, 2.0–15.0 mmol). The suspension was stirred at room temperature under atmospheric pressure. Samples were withdrawn periodically, diluted with 10.0 ml of THF, or CS_2 in the cases of $C_{60}Br_8$ and $C_{60}Br_{24}$, and then analyzed by GC (HP 5890 series II). GC conditions: column HP1, SiO_2, 25 m × 0.32 mm × 0.52 µm. GC program: hold at 32 °C for 5 min; heat at 15 °C min^{-1} to 260 °C, hold for 1 min, the injector at 200 °C and the flame ionization detector at 250 °C. The peak identity was confirmed by GC–MS.

RESULTS AND DISCUSSION

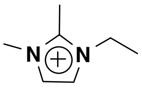
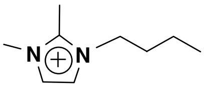
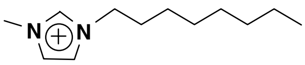
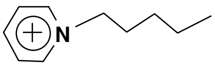
Ionic liquid preparation

N-Pentylpyridinium bromide (**1**) was prepared in good yield (95%) by refluxing 1-bromopentane in dry pyridine, following a slight modification of the general method described by Levenson.[27] The metathesis reaction of **1** with cesium carborane, Cs[*closo*-$CB_{11}H_{12}$], in CH_2Cl_2, followed by column chromatographic purification afforded the *N*-pentylpyridinium carborane (**2**) in 93% yield. The syntheses reactions are essentially quantitative, with the lower yield for **2** being due to loss by absorption on the SiO_2 during column chromatography.

The ^{1}H and ^{13}C NMR spectra of **1** and **2**, as well as their elemental analyses, are consistent with their formulations as given in the Experimental section. There is a general upfield shift in both the ^1H NMR ($\Delta\delta = 0.06-0.36$ ppm) and ^{13}C NMR ($\Delta\delta = 0.42-0.89$ ppm) spectra in going from **1** to **2**. The ^{11}B NMR spectrum of **2** shows the expected three resonances in a 1 : 5 : 5 peak area ratio for the CB_{11} cage. Other than the strong ν_{B-H} absorption at 2571 cm^{-1}, the IR spectrum of **2** shows no noteworthy features and is presented for purposes of qualitative analysis.

Compound **2** is an air-stable, waxy solid that melts at 19 °C to give a colorless liquid. Table 1 lists the melting point of **2**, along with those of several polyalkyl imidazolium salts of the [*closo*-$CB_{11}H_{12}$]$^-$ anion.[12] The lower melting point of **2** is somewhat surprising in view of the 70 °C value reported for the 1-octyl-2-methylimidazolium salt. As seen in Table 1, the melting points of the solids are as much a function of the shape of the ions as of their overall size. This demonstrates that, for equivalent charges, melting points depend on the packing efficiency of the ions, of which the size is just one of several important factors. The reactions described in this report for the preparation of **2** are general ones that should be applicable to the syntheses of a series of *N*-alkylpyridinium salts of carborane anions containing different alkyl groups and exhibiting a span of melting points suitable for reactions at different temperatures. Such an array of ionic liquids would be a useful complement to the imidazolium series.

Table 1. Melting points of different cations with [*close*-$CB_{11}H_{12}$]$^-$

Cations	M.p. (°C)
	156[a]
	129[a]
	122[a]
	70[a]
	19

[a] See Ref. 12.

Dehalogenation reactions

Compound **2** was used as the solvent for a number of dehalogenation reactions catalyzed by $PdCl_2(PPh_3)_2$, $PdCl_2$(dppe) and $PdCl_2$(dppf). In the runs, the catalysts to polyhalide substrate molar ratios were fixed initially at 1%, 0.5% and 0.5% respectively for the different catalysts. It was found that the particular catalyst dissolved only slowly in the ionic liquid. In order to hasten solubility, the substrate, catalyst and ionic liquid were added to THF, affording a clear solution. Once this was accomplished the THF could be removed, and the base (TMEDA) and reductant ($NaBH_4$) were added to commence the dehalogenation reactions. The reaction stoichiometries used in our studies were essentially the same as those reported for similar reactions in organic solvents.[16,17] The progress of the reactions was monitored by removing and analyzing aliquots every 20 min. The results are shown in Tables 2 and 3 and in Figures 1 and 2.

Debromination

The brominated substrates chosen for study were hexabromobenzene (C_6Br_6) 1,2,4,5-tetrabromobenzene ($C_6H_2Br_4$), $C_{60}Br_8$, and $C_{60}Br_{24}$. The results, listed in Table 2, show that, in all cases, complete debromination was accomplished in less than 10 h at room temperature. For any substrate, the catalyst efficiency decreased in the order $PdCl_2$(dppf) > $PdCl_2$(dppe) > $PdCl_2(PPh_3)_2$. In general, for the same catalyst the C_{60}-based substrates had 100% conversion times that were about twice those of the benzene-based substrates with the longest found for $C_{60}Br_{24}$. Figure 1 shows plots of percentage conversion of $C_{60}Br_{24}$ as a function of time for each catalyst. The plots clearly show the increased efficiency of the two bidentate catalysts, $PdCl_2$(dppe) and

Table 2. Debromination data in liquid [*N*-pentylpyridinium]$^+$ [*closo*-$CB_{11}H_{12}$]$^-$ (**2**)

	100% conversion time[a] (h) and [TOF][b]			
Polybromide	$PdCl_2(PPh_3)_2$ (1%)[c]	$PdCl_2$(dppe) (0.5%)[c]	$PdCl_2$(dppf) (0.5%)[c]	Products (mol%)
C_6Br_6	4.8 [21]	3.4 [59]	2.6 [77]	Benzene (100)
1,2,4,5-Tetrabromobenzene	4.1 [24]	2.8 [71]	2.2 [91]	Benzene (100)
$C_{60}Br_8$	8.4 [12]	6.8 [29]	5.7 [35]	C_{60} (100)
$C_{60}Br_{24}$	9.3 [11]	7.4 [27]	6.2 [32]	C_{60} (100)

[a] The 100% conversion time is based on GC analysis; the actual time might be a few minutes earlier due to sample withdrawal and preparation times.
[b] Turnover frequency.
[c] The catalyst concentration is defined as [catalyst mole]/[substrate mole] × 100%.

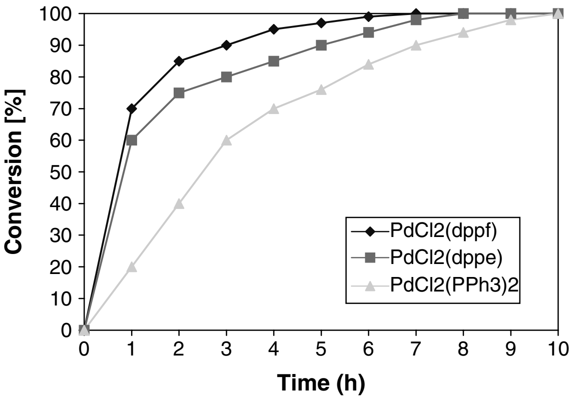


Figure 1. Percentage debromination of $C_{60}Br_{24}$ versus time in ionic liquid for different catalysts.

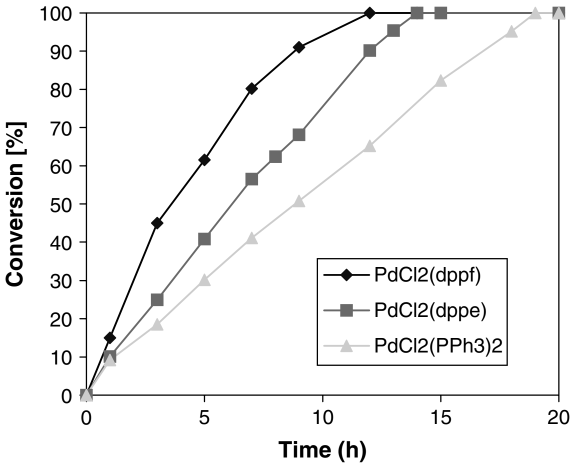


Figure 2. Dechlorination of 1,2,4-trichlorobenzene by different catalysts.

Table 3. Product distribution in the dechlorination of 1,2,4-trichlorobenzene

Solvent	Catalyst	1,2-DCB[a] (%)	1,3-DCB[a] (%)	1,4-DCB[a] (%)
THF[b]	$PdCl_2$(dppf)	40	20	40
CH_3CN[b]	$PdCl_2$(dppf)	50	20	30
DMF[b]	$PdCl_2$(dppf)	70	15	15
2[c]	$PdCl_2(PPh_3)_2$	86	10	4
2[c]	$PdCl_2$(dppe)	92[c]	5	3
2[c]	$PdCl_2$(dppf)	95	4	1

[a] DCB : dichlorobenzene.
[b] Ref. 17.
[c] This work.

$PdCl_2$(dppf), compared with their diphosphine analog. It should be noted that, in the absence of catalyst, all of the dehalogenation reactions proceeded quite slowly.

Dechlorination

In contrast to the debromination reactions, the dechlorination of the polychlorobenzenes can produce different products, depending on the choice of catalyst, reductant, base and solvent. As with the debromination reactions, the base TMEDA and the reductant $NaBH_4$ were used in the dechlorination studies, along with the catalysts $PdCl_2(PPh_3)_2$, $PdCl_2$(dppe), and $PdCl_2$(dppf). Chlorobenzene and 1,2,4-trichlorobenzene were chosen as substrates. The results are shown in Figure 2 and Table 3. Chlorobenzene was found to dechlorinate only slowly with any catalyst, with complete conversion to benzene being achieved after 35 h, 27 h, and 21 h for the catalysts $PdCl_2(PPh_3)_2$, $PdCl_2$(dppe), and $PdCl_2$(dppf) respectively. This order of catalytic efficiency is the same as found for the polybromides (see Table 2). On the other hand, dechlorination of 1,2,4-trichlorobenzene to dichlorobenzene proceeds more rapidly, with 100% conversion being achieved in less than 20 h, even for the poorest catalyst ($PdCl_2(PPh_3)_2$). The increase in rate with degree of chlorination agrees with the results of Hor and coworkers,[17] who studied the dechlorination of polychlorides in several organic solvents, using TMEDA and $NaBH_4$ in the presence of the $PdCl_2$(dppf) catalyst. However, the ionic liquid solvent seems to enhance dechlorination. In the organic solvents, chlorobenzene was reported to be essentially unreactive,[16,17] whereas, in **2** complete dechlorination could be achieved in 21 h. The dechlorination of 1,2,4-trichlorobenzene could lead to any one of three isomers: 1,2-dichlorobenzene (ortho isomer), 1,3-dichlorobenzene (meta isomer), or 1,4-dichlorobenzene (para isomer). Table 3 lists the product distributions found for the different catalysts, as well as those reported by Lassová *et al.*,[17] who studied the $PdCl_2$(dppf)-catalyzed dechlorination of 1,2,4-trichlorobenzene in THF, CH_3CN and *N*,*N*-dimethylformamide using TMEDA and $NaBH_4$. As can be seen from Table 3, the isomer distribution in the ionic liquid solvent is ortho $\gg$ meta $>$ para for any catalyst, with the greatest differentiation (95 : 4 : 1) found for $PdCl_2$(dppf). These results differ significantly from those reported in THF, in which the order was ortho $=$ para $>$ meta (40 : 40 : 20); other solvents gave different ratios, as can be seen from Table 3.[17]

The most striking aspect of Table 3 is the influence the solvent has on the isomer distribution of the dechlorinated product. The most regiospecific dechlorination is found in the ionic liquid (**2**), in which the elimination was almost exclusively from the 4-position of the trichlorobenzene. Although the substitution mechanism in the ionic liquid has not been determined, there is no reason to assume that it differs greatly from that of other palladium-catalyzed dehalogenations in which oxidative addition/reductive elimination of the arylhalide to an *in situ* generated palladium(0) is the critical step in determining both the rate and the product distribution.[8–20] If this is the case, it is not readily apparent why the 4-position is almost exclusively the one where that adds to the palladium(0), especially in view of the other results listed in Table 3. For the dechlorination of 1,2,4-trichlorobenzene catalyzed by a $PdCl_2$ that was anchored to the polymer poly(*N*-vinyl)-2pyrrolidone, Zhang *et al.*[13] found an isomer distribution of the dichlorobenzene products of 61% 1,2-, 16% 1,3-, and 9% 1,4-, with the remainder being composed of more highly dechlorinated benzenes. The authors attributed this preference for dechlorination at the 4-position to steric effects. It may well be that the large solvent particles in the ionic liquid solvent (**2**) imposes a more stringent steric requirement than found in the smaller solvents. The detailed mechanism of dehalogenation in the ionic liquid solvent is currently being studied in our laboratories.

Recycle process

All three of the highly active catalysts can be repeatedly reused without separation from the ionic liquid. The recycling process can be performed by distillation of volatile products at the end of the reaction. The system can be recycled at least seven times before the turnover frequency changes noticeably. The catalysts must be redissolved in either $CHCl_3$ or CH_2Cl_2 and filtered through Celite to remove the NaCl or NaBr when it is saturated with these inorganic salts. If this is not done, then fine salt particles increase the reaction time by blocking effective diffusion of reagents in the ionic liquid. The catalytic system was still active even after 1 month storage under an inert atmosphere.

CONCLUSION

We have synthesized and characterized a new ionic liquid with a low melting point of 19 °C from the combination of a derivated *N*-alkylpyridinium cation and carborane anion. This ionic liquid was used as a solvent for dehalogenation reactions of 1,2,4-trichlorobenzene to give almost exclusive dechlorination at the 4-position. In general, the reaction times in this solvent are much shorter than those found in organic solvents. This catalytic system is highly active and quite robust. Our future research is concentrated on studying the mechanism of this catalytic system and exploring the practical application of this new medium in dehalogenation and other reactions.

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