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Didecyldimethylammonium bromide (DDAB): a universal, robust, and highly potent phase-transfer catalyst for diverse organic transformations

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Abstract—Didecyldimethylammonium bromide (DDAB) has been scrutinized in comparison with traditional phase-transfer catalysts in variety of liquid–liquid reactions. It was found to be an exceptionally comprehensive, durable, and highly efficient phase-transfer catalyst (PTC) in a number of representative organic transformations such as C- and N-alkylations, isomerization, esterification, elimination, cyanation, bromination, and oxidation under very mild conditions of temperature and mixing. It was confirmed that DDAB is an exceedingly accessible and concurrently a highly liphophilic phase-transfer catalyst. This unprecedented characteristic renders DDAB to be a multipurpose catalyst that functions effectively both in mass transfer controlled and chemically controlled phase-transfer reactions.

1. Introduction

Phase-transfer reactions are ordinarily classified according to the nature and the solubility of the catalyst, and the number and type of the phases involved in the reacting systems. Most familiar are liquid–liquid (LLPTC) and liquid–solid (SLPTC) reactions. Liquid–solid–liquid (LSLPTC) using e.g., polymer supported phase-transfer catalysts or liquid–liquid–liquid (TLPTC) where the catalyst forms a third liquid phase is less typical.[1–3] A more universal approach to the categorization of phase-transfer systems is based on kinetic criteria.[4] Since all phase-transfer catalyzed reactions have at least one transfer step and one chemical step they can be broadly sorted into two major groups: mass transfer controlled reactions (T-reactions) and chemically controlled reactions (I-reactions). Accordingly, two main mechanisms have been recognized in phase-transfer reactions: (a) the extraction mechanism where anions are rapidly transferred as ion-pairs from aqueous or solid phase into the organic phase where they slowly react with a substrate and (b) the interfacial mechanism,[5] typical in reactions promoted by alkali, where interfacial deprotonation converts an anion (such as a carbanion, oxanion or azanion), which is slowly extracted into the organic phase to swiftly react with an electrophilic substrate. Other processes where the transfer step (or steps) is slow relative to the intrinsic chemical reaction are also classified under the interfacial mechanism. The distinction between these two mechanisms is not always very sharp and in some cases both are operating in tandem. Thus it was argued that the classical cyanide displacement reaction in a liquid–liquid system, known to proceed via the extraction mechanism, operates under mass transfer control when the stirring is not effective, namely below 200 rpm. A slow anion transfer rate can also stem from high interfacial tension between phases, highly hydrated anions or the use of phase-transfer catalysts that are inhibited from approaching the interphase. In general it is well established that the effect of mixing on the observed reaction rate is the best experimental method to determine if a system is transfer or chemically controlled.[6] Thus the latter phase-transfer catalyzed reactions are not affected by stirring above approximately 200 rpm while the transfer controlled reactions will increase in rate as a function of stirring up to 2000 rpm.

Solid–liquid phase-transfer reactions, where the function of the catalyst is to transfer anions from a solid crystalline salt into the organic phase, are usually considered to be transfer controlled under all conditions,[7,8] although some exceptions are known.

Several authors have realized that the nature of the quaternary ammonium phase-transfer catalyst required for transfer controlled reactions is entirely different from the characteristics of the preferred catalyst for reaction controlled processes. The typical, widely spread, accessible catalyst introduced by Makosza[9] in 1966 is triethylbenzylammonium chloride (TEBA), which is very effective and extensively

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used in T-reactions (mainly reactions of organic anions) but perform poorly where the extraction mechanism is dominant.[10] Cetyltrimethylammonium bromide (CTMAB) is also a weak catalyst in I-reactions but quite active in solid–liquid mass transferred controlled PTC reactions.[11]

It was concluded that for reactions proceeding via the extraction mechanism, the preferred catalysts were the more organophilic and symmetrical quaternary cations.[12] The latter demonstrate higher extraction coefficients and more reactive nucleophilic anions (due to lower Coulombic interaction energies with the larger cation). Conversely, in transfer controlled reactions the supreme phase-transfer catalysts are the more ‘accessible’ or ‘open faced’ quaternary ammonium cations.[13] The latter carry smaller alkyl groups, and thus readily occupy positions close to the interphase thus creating a higher catalyst concentration at the site of the rate-determining step. Mason et al.[14] have demonstrated the correlation between the surface properties of several quaternary ammonium salts (quats) and their catalytic activity in C-alkylation process, a typical I-reaction. It was concluded that quats that more effectively reduce the surface pressure at the water–organic interphase are also the more potent PT catalysts. It was also proven that the catalytic activity did not directly stem from the reduction in surface pressure. A quantitative empirical parameter q introduced by Halpern[15] can be used to assess the T versus I activity of various quaternary ammonium catalysts. The q value is calculated by adding the reciprocals of the number of carbons on each of the alkyl chains of the ammonium cations. Thus for TEBA $q=1.64$ and for tetrahexylammonium bromide (THAB) $q=0.67$. It is generally accepted that for T-reactions the q-value should be higher than 1. Best results are obtained with catalysts with q value between 1.5 and 2.0. It should be noted, however, that a certain critical level of organophilicity is still required for interfacial reactions. Thus tetramethyl- and tetraethylammonium catalysts are not considered as active T-type catalysts. For I-reactions q should preferably be <1. Despite the high q value of surfactants such as CTAB ($q>3$), these are not useful catalysts due to the formation of emulsions in water–organic mixtures when the latter are present. Another important criterion in phase-transfer catalyst selection is the thermal and chemical stability of the catalyst,[16] and also the ease of its separation from the reaction mixture after the process is completed.[17]

In view of these and other guidelines, most practitioners have chosen tetrabutylammonium bromide (TBAB) ($q=1$) as the default catalyst for exploratory PTC applications. Indeed this catalyst performs satisfactorily in essentially all I- and T- PTC reactions and it is also reasonably stable to alkaline and thermal conditions and swiftly washed away from product mixtures once the reaction is completed. However it is rarely the optimal catalyst for a given process.

We have now recognized that another family of quaternary ammonium salts, represented by the commercially available surfactant didecyldimethylammonium bromide (DDAB), is far more pertinent to the label of ‘universal’ or ‘default’ phase-transfer catalyst. As is demonstrated in this work, DDAB is an excellent PT catalyst in both I- and T-reactions. The group of dialkyldimethylammonium halides has been overlooked in the past by PTC researchers, probably due to high cost and limited availability. This has evidently changed since the syntheses of the latter compounds have been recently dramatically simplified via a procedure based on simple and mild alkylation of dimethylformamide.[18,19]

DDAB is being used in various industrial fields including bio-chemical industries.[20] DDAB has not been previously proposed as a phase-transfer catalyst except for one example in which we have demonstrated its unique activity in an oxidation reaction using aqueous hydrogen peroxide, where it showed superior activities among the PTCs screened.[21]

2. Results and discussion

Prior to our catalytic experiments, we assessed the physiochemical properties of DDAB in comparison with some standard phase-transfer catalysts such as TEBA (Makosza’s catalyst),[22] tricaprylmethylammonium chloride (aliquat 336, Starks’ catalyst),[23] and TBAB (Brandstrom’s catalyst).[24] According to the standard evaluation of quaternary ammonium salts as phase-transfer catalysts, Makosza’s catalyst is unsymmetrical but has a shorter alkyl chain, and hence is an accessible catalyst. Starks’ catalyst possesses one short alkyl chain, and is liphophilic in nature but inferior from the symmetry perspective, and Brandstrom’s catalyst is in perfect symmetry but is only partially accessible due to the masking of C4 alkyl chains. Considering the extraction mechanism, the relative performance of these three catalysts is: Starks’ catalyst>Brandstrom’s catalyst>Makosza’s catalyst, whereas for the interfacial mechanism, the order is: Makosza’s catalyst>Brandstrom’s catalyst>Starks’ catalyst. In comparison with the above, DDAB has a potential advantage in being both highly accessible and highly organophilic at the same time. Initially, the surface pressure was measured for water/air and toluene/air interfaces in the presence of DDAB. It was found to be 2.78 dynes/cm and 2.41 dynes/cm, respectively. These figures are an order of magnitude smaller than the surface pressure reported for other PTCs such as TBAB, TEBA, and aliquat 336[14] signifying that DDAB is a potentially more active catalyst.

Further, we have examined the thermal stability of DDAB in comparison with both TBAB and tributylmethylammonium bromide (TBMAB). This was performed using differential scanning calorimetry (DSC). The substances were dissolved in a neutral organic medium (isopropanol) and heated at a rate of 10 °C/min and the thermal events recorded. We established that DDAB decomposed at 255 °C, TBAB at 213 °C, and TBMAB at 206 °C. At a second stage we assessed the thermal stability of these catalysts under basic conditions. This was accomplished using the catalytic isomerization reaction of anethole[25] in the presence of 50% aqueous NaOH (Eq. 1) as a reporter reaction indicative to the presence of an active catalyst.

Table 1 presents the maximum conversions obtained in Eq. 1 at different temperatures in the presence of 8 mol % of TBMAB, TBAB, and DDAB. The table also displays the time required to achieve the maximum conversion. The latter is actually the life span of the particular catalyst under the given conditions, as the reaction stops once the catalyst is totally decomposed. We can safely conclude that DDAB is

Table 1. Study of stability of different phase-transfer catalysts by means of isomerization of 4-propenyl anisole to β-anethole (Eq. 1)[a]

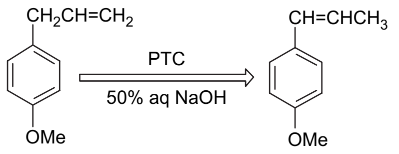
Catalysts[b]	Entry	Temperature (°C)	Time to achieve maximum conversion (min)	Maximum conversion[c] (%)
TBMAB	1	90	45	50
	2	110	20	38
	3	130	10	27
TBAB	1	90	120	28
	2	110	60	22
	3	130	20	18
DDAB	1	90	1080	76
	2	110	1140	67
	3	130	120	52

[a] Reaction condition: 4-propenyl anisole (10 mmol), catalyst (8 mol %), toluene (10 mL), and NaOH (50% w/w, 10 mL).

[b] TBMAB (tributylmethylammonium bromide), TBAB (tetrabutylammonium bromide), and DDAB (didecyldimethylammonium bromide).

[c] Conversion based on gas chromatography analysis with area minimization.

by far more stable than the other two catalysts examined, surviving 120 min at 130 °C versus TBAB that endured for 20 min and TBMAB that degraded after merely 10 min. These experiments were repeated twice with excellent reproducibility.



(1)

We then examined the catalytic activity of DDAB in several illustrative I-type and T-type phase-transfer reactions. All the processes were reported previously using other different quaternary ammonium catalysts. The reactions studied were:

(1) Alkylation of phenylacetonitrile with *n*-butylbromide[26] and of iminostilbene with allyl bromide.[27] Both are typical T-reactions.
(2) Isomerization of 4-propenyl anisole to β-anethole, an I-type reaction.[28]
(3) Oxidation of thiophene 2-carboxaldehyde to thiophene 2-carboxylic acid using hypochlorite as oxidant.[29]
(4) Dehydrobromination of 2-phenylethyl bromide.[30]
(5) Cyanation of benzyl chloride—a classical I-reaction.[31]
(6) Hydrobromination of 1-decanol with aqueous HBr.[32]
(7) Esterification of benzyl chloride with sodium formate,[33] this is also a typical I-reaction.

Reactions 1, 2, and 7 were performed under identical conditions with TBAB, TBMAB, and DDAB as catalysts. Results are presented in Table 2 where the superiority of DDAB in all the three processes is clearly demonstrated. Table 3 displays the experimental conditions and results of the above eight model reactions in the presence of DDAB catalyst. Detailed procedures are given in Section 4. In general, experiments were carried out under mild conditions using a 50 mL reactor (10 mmol scale) equipped with a magnetic stirrer under temperatures of 40–120 °C. The reactions were monitored using gas chromatographic analyses, which were also the basis of calculation for the final conversions. All the tested reactions showed >99% selectivity and very high conversions.

Table 2. Comparison of DDAB with TBAB and TBMAB in catalytic activity as PTC

Reaction	DDAB		TBAB		TBMAB	
	Conv. (%)	Selec. (%)	Conv. (%)	Selec. (%)	Conv. (%)	Selec. (%)
Isomerization[a]	80	—	28	—	50	—
Alkylation[b]	97	100	61	100	73	100
Esterification[c]	100	100	75	100	83	100

[a] Reaction condition: 4-propenyl anisole (10 mmol), catalyst (8 mol %), toluene (10 mL), NaOH (50% w/w, 10 mL), temperature (90 °C), and time (24 h).

[b] Phenylacetonitrile (10 mmol), bromobutane (10 mmol), DDAB (2 mol %), toluene (10 mL), NaOH (50% w/w, 10 mL), temperature (40 °C), and time (2 h).

[c] Benzyl chloride (10 mmol), sodium formate (10 mmol), DDAB (5 mol %), toluene (10 mL), NaOH (50% w/w, 10 mL), temperature (120°), and time (2 h). Conversion and selectivity are based on gas chromatography analysis.

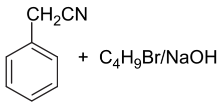
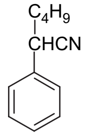
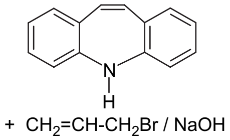
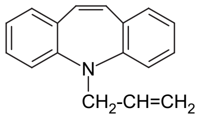
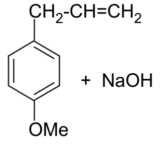
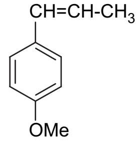
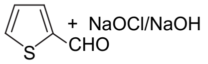
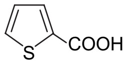
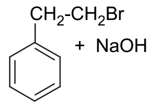
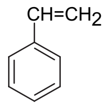
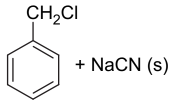
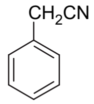
Alkylation of phenylacetonitrile (Entry 1 in Table 3) and iminostilbene (Entry 2 in Table 3) proved that DDAB can effectively catalyze these processes at mild reaction temperature to achieve high conversion and selectivity of products. Phenylacetonitrile and iminostilbene were alkylated to attain 97% and 95% selectivity of products with 2 and 10 mol % DDAB catalysts at 2 and 4 h reaction time, respectively. Further, alkylation of phenylacetonitrile has been screened using DDAB, TBAB, and TBMAB for comparison study, which reveals that DDAB is more active (Table 2) than the other two mentioned PTCs.

The performance of DDAB in an isomerization reaction was tested in 4-propenyl anisole to β-anethole (*p*-methoxy-β-methylstyrene) reaction (Eq. 1, Entry 3 in Table 3). The system was refluxed at 90 °C for 24 h and found to achieve 80% conversion using 8 mol % of DDAB. Further, isomerization was also studied with other quaternary salts (Table 2) where it was found that DDAB is notably more active.

DDAB also showed a good stability and activity in reactions, which took place in the presence of oxidants such as sodium hypochlorite and hydrogen peroxide (Entry 4 in Table 3), where within 1 h, thiophene aldehyde exclusively converted to acid (100% conversion and selectivity) at 45 °C using 1.5 mol % of DDAB catalyst. Entry 5 in Table 3 demonstrates that DDAB catalyst can also be used in elimination reactions to selectively eliminate HBr from 2-phenylethyl bromide by means of sodium hydroxide as base to yield 88% of styrene, with just 1 mol % of catalyst within 4 h of reaction time at 90 °C.

The cyanation reaction of benzyl chloride to obtain acetonitrile (Entry 6 in Table 3) was also performed. In a typical reaction, benzyl chloride was mixed with DDAB catalyst and then kept in a glove box, sodium cyanide added, and the reaction carried out for 1 h and achieved 100% conversion of benzyl chloride and 100% selectivity to

Table 3. Catalytic reactions of didecyldimethylammonium bromide[a]

Entry	Reaction	Reactants	Products[j]	Conversion[k] (%)	Selectivity[k] (%)
1[b]	Alkylation			97	100
2[c]	Alkylation			95	100
3[d]	Isomerization			80 cis+trans	—
4[e]	Oxidation			100	100
5[f]	Elimination			88	100
6[g]	Cyanation			100	100
7[h]	Bromination	$C_{10}H_{21}OH+HBr/H_2O$	$C_{10}H_{21}Br$	100	100
8[i]	Esterification	$C_6H_5CH_2Cl+HCOONa$	$HCOOCH_2C_6H_5$	100	100

[a] Reactions were conducted in a batch reactor under liquid phase condition.
[b] Phenylacetonitrile (10 mmol), bromobutane (10 mmol), DDAB (2 mol %), NaOH (50% w/w, 10 mL), toluene (10 mL), temperature (40 °C), and time (2 h).
[c] Iminostilbene (10 mmol), 3-bromopropene (10 mmol), DDAB (10 mol %), NaOH (50% w/w, 10 mL), toluene (10 mL), temperature (55 °C), and time (4 h).
[d] 4-Allylanisole (10 mmol), DDAB (8 mol %), NaOH (50% w/w, 10 mL), toluene (10 mL), temperature (90 °C), and time (24 h).
[e] Thiophene aldehyde (10 mmol), sodium hypochlorite (10 mmol), DDAB (1.5 mol %), NaOH (50% w/w, 10 mL), toluene (10 mL), temperature (45 °C), and time (1 h).
[f] 2-Phenylethyl bromide (10 mmol), DDAB (1 mol %), NaOH (50% w/w, 10 mL), toluene (10 mL), temperature (90 °C), and time (4 h).
[g] Benzyl chloride (10 mmol), sodium cyanide (10 mmol), DDAB (1.5 mol %), toluene (10 mL), temperature (105 °C), and time (1 h).
[h] 1-Decanol (10 mmol), hydrogen bromide (10 mmol), DDAB (10 mol %), hydrogen peroxide (30%, 3 mL), NaOH (50% w/w, 10 mL), toluene (10 mL), temperature (120 °C), and time (16 h).
[i] Benzyl chloride (10 mmol), sodium formate (10 mmol), DDAB (5 mol %), NaOH (50% w/w, 10 mL), toluene (10 mL), temperature (120°), and time (2 h).
[j] Products are identified by GC–MS or by comparison with authentic samples.
[k] Conversion and selectivity of products are based on gas chromatography analysis.

phenylacetonitrile. The stability of DDAB under strongly acidic conditions was checked in a hydrohalogenation reaction by means of hydrogen bromide (48% aqueous solution) and achieved 100% conversion of 1-decanol to 1-bromodecane after 16 h of heating at 120 °C reflux temperature (Entry 7 in Table 1). Esterification of benzyl chloride using sodium formate as esterification agent achieved 100% conversion of benzyl chloride, in 2 h of reaction time using 5 mol % of DDAB catalyst. Further, this reaction was also studied and evaluated in comparison with TBAB and TBMAB, which again confirms the superiority of DDAB (Table 2).

The excellent high conversions obtained, across the board, for this diversified group of PTC reactions are indeed remarkable and as far as we know unprecedented. It is evident that DDAB functions extraordinarily at different PTC mechanisms under very mild condition and retains its activity for long periods of time even in highly basic conditions.

Since DDAB is sparingly soluble in water (100 mg DDAB in 30 mL water) it can be extracted and removed from organic reaction mixtures by repeated washing with water.

3. Conclusions

DDAB is ascertained as a general purpose and universal phase-transfer catalyst that performs very effectively in a variety of highly diversified liquid–liquid reactions. This novel quaternary ammonium salt was shown to operate effectively both via the extraction mechanism and the interfacial mechanism, and in addition was realized to be exceptionally stable in relatively high temperatures and high pH.

4. Experimental

4.1. Materials and instruments

Didecyldimethylammonium bromide (DDAB) was purchased from Aldrich and characterized for its chemical and physical properties prior to catalytic experiments. Other chemicals (reagents and solvents) were purchased from commercial firms (>99% pure) and used without further purification. The reaction mixtures were analyzed with a gas chromatograph (HP 5890) equipped with a flame ionization detector and a capillary column (5% cross-linked phenyl methyl silicone gum, 0.2×50 m). The products were also compared with authentic samples wherever possible.

Surface pressure measurements were performed by Du Noy ring method using a Lauda Tensiometer. Using the 'prevented ruptured' technique, in which the interface is not broken during the measurements, readings were taken every 5 min until a stable value was obtained. For this measurement, 100 mg of DDAB was dissolved in 20 mL toluene/ 20 mL water separately and measured.

4.2. Catalytic reactions

4.2.1. Alkylation of phenylacetonitrile and iminostilbene. Alkylation of phenylacetonitrile and iminostilbene was done with 1-bromobutane and 3-bromopropene, respectively, using a 1:1 molar ratio of reactants. In a typical run, phenylacetonitrile (10 mmol) was taken in a 50 mL Schlenk flask and 2 mol % of DDAB was added followed by the addition of 10 mL of toluene. Then 1-bromobutane (10 mmol) was added followed by the addition of 10 mL of NaOH (50% w/w in water). The reaction mixture was magnetically stirred and heated to the required temperature (40 °C) in a preheated oil bath for 2 h. The same procedure was followed for the alkylation of iminostilbene using the required amounts of substrates and catalyst (see Entry 2 of Table 3).

4.2.2. Isomerization of 4-propenyl anisole. Isomerization of 4-propenyl anisole was executed using toluene as solvent. In a typical procedure, 4-propenyl anisole (10 mmol) was taken in a 50 mL Schlenk flask and 8 mol % of DDAB followed by the addition of 10 mL of toluene and 10 mL of NaOH (50% w/w in water). The reaction mixture was stirred at 90 °C over a preheated oil bath for 24 h.

4.2.3. Oxidation of thiophene 2-aldehyde. Oxidation of thiophene 2-aldehyde was performed in a 50 mL Schlenk flask using 1.5 mol % of DDAB in 10 mL toluene. In a representative course of action, thiophene aldehyde (10 mmol) was mixed with 10 mmol of sodium hypochlorite and 10 mL of NaOH (50% w/w in water), and the whole system was stirred at 45 °C for 1 h.

4.2.4. Elimination of HBr from 2-phenylethyl bromide. Elimination of HBr from 2-phenylethyl bromide to obtain styrene was carried out using DDAB catalyst. In a typical run, 2-phenylethyl bromide (10 mmol) was added with 1 mol % of DDAB in toluene followed by the addition of 10 mL of NaOH (50% w/w). The reaction mixture was stirred at 90 °C in a preheated oil bath for 4 h.

4.2.5. Cyanation of benzyl chloride. Cyanation of benzyl chloride was performed using 1.5 mol % of DDAB. In a typical procedure, benzyl chloride (10 mmol) was mixed with 10 mmol of sodium cyanide followed by the addition of 10 mL toluene and NaOH (50% w/w) and the whole system was subjected to heating at 105 °C for 1 h under stirring. Precautionary measures were made to avoid the leakage of cyanide vapor during reaction. The reaction has to be done in a glove box using a water condenser connected to batch reactor to prevent the loss of volatile cyanide vapor from the mixture.

4.2.6. Hydrobromination of 1-decanol. Hydrobromination of 1-decanol was performed in a 50 mL Schlenk flask using 10 mol % of DDAB. In a representative procedure, 1-decanol (10 mmol) was mixed with 10 mmol of hydrogen bromide in water and 3 mL of 30% hydrogen peroxide added. The whole system was stirred at 120 °C for 16 h.

4.2.7. Esterification of benzyl chloride with sodium formate. Benzyl chloride (10 mmol) was mixed with sodium formate (10 mmol), 5 mol % of DDAB, 10 mL of NaOH (50% w/w), and 10 mL toluene were added and the reaction mixture was magnetically stirred and heated to the required temperature (120 °C) in a preheated oil bath for 2 h.

4.2.8. General work up procedure. The progress of reactions was monitored by GC. After completion of the reaction, the reaction mixture was stirred and washed with 5×35 mL of water to remove DDAB and NaOH. The organic layer was dried over $MgSO_4$ and was concentrated under reduced pressure to give a dried compound as product. The products were analyzed with an analytical tool, gas chromatograph, identified by GC–MS, injecting authentic samples, and by ^1H NMR.

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