Phase-Transfer Catalysis Communications

Achieving Effective Catalyst Separation

New supporting data, conclusions and guidelines for achieving excellent catalyst removal while simultaneously reducing waste stream volume

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Summary: New data have been generated which describe the distribution of three commercial quaternary ammonium (quat) phase-transfer catalysts between five organic "solvents" and two aqueous phases. Examination of these data strongly suggest that effective catalyst separation from the product can be achieved in a variety of cases in which it is desirable to extract the catalyst into water OR have the catalyst remain in the organic phase. It is shown that extraction of the catalyst into water (the most common method of separating catalyst in commercial applications) can be improved by up to three orders of magnitude while using only half of the water washes. In many cases, residual levels of < 1 ppm of water soluble catalysts can be achieved with one or two water washes. In some cases, replacement of an existing catalyst system may be worthwhile to achieve desirable catalyst separation. The data presented here may help prevent operational problems (e.g., undesirable residual levels of catalyst), especially in the development stage of a commercial process which will be locked in by an expensive and rigorous process registration mechanism (e.g., for pharmaceuticals, agricultural chemicals, tires, photographic applications, polymer additives, etc.).

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New Phase-Transfer Catalysis Company Focuses on PTC Process Development

A new company, PTC Organics, Inc., was formed in the 4th quarter of 1998 to use phase-transfer catalysis to develop high-performance low-cost processes for the manufacture of organic chemicals. Organic chemical producers can become more competitive in challenging markets using PTC contract research, consulting, training and/or licensing.

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INTRODUCTION

There are several criteria to be considered when choosing a phase-transfer catalyst for a commercial phase-transfer catalysis (PTC) application. The most important criteria are reactivity, separation of catalyst from the product, availability, the real cost of catalyst and solvent (per kg product produced), method of environmentally acceptable disposal, catalyst stability and toxicity. The first two performance parameters usually considered are reactivity and separation of commercially available catalysts. Few publications describe the effects of both catalyst and solvent on reactivity.[1,2,3] In addition, very little has been published comparing the *distribution* of different phase-transfer catalysts in various solvent systems,[4,5] which is crucial for catalyst separation. Thus, when choosing a phase-transfer catalyst and solvent for a commercial application, industrial process chemists and engineers have to rely on intuition and personal PTC experience (which is often limited to just a few cases at most) to induce high reactivity while also achieving good separation from the product. During the past two years, one of the authors has been approached several times by process chemists and engineers complaining about high residual levels of tetrabutylammonium bromide in their product while generating significant aqueous waste streams. This report is directed at providing useful information to process chemists and engineers for choosing commercially available phase-transfer catalysts to enhance separation as well as reactivity.

Quaternary ammonium, "quat," salts are by far the most common phase-transfer catalysts used in industrial applications. Three commercially available quats will be described in this report: Aliquat® 336[6] (methyl tricaprylyl ammonium chloride: $(C_{8\text{-}10}H_{17\text{-}21})_3NCH_3$ Cl with a C_8:C_{10} ratio of 2:1), Aliquat® 100 (tetrabutyl ammonium bromide) and Aliquat® 175 (methyl tributyl ammonium chloride). Industrial chemists and engineers have found these catalysts to meet the criteria for real world applications (cost effective for reactivity, separation, handling, disposal, etc.) and hence they are used widely in commercial PTC processes. Other phase-transfer catalysts are used in industrial applications, including polyethylene glycols and crown ethers (we intend to study these at a later date).

The great flexibility in choosing a solvent for a commercial reaction is one of the major strengths of industrial phase-transfer catalysis. Since solvent affects reactivity and catalyst separation, solvent also affects choice of catalyst. Solvents commonly chosen for commercial PTC applications include aromatic hydrocarbons, alkyl halides (despite environmental trends), ketones and "solvent-free PTC." Solvent-free PTC can often be employed when at least one reactant and the product are liquids (at the reaction temperature) or when one of the liquid reactants can be used as a solvent (excess being recycled). For example, many commercial PTC esterifications and etherifications are performed under "solvent-free PTC" conditions. As the reaction progresses, the "solvent" changes from an alkyl halide or alcohol to the ester or ether product. Given the solvents useful for common PTC applications, it would useful to have available data relating to the distribution of commercially available quat salts in a variety of "solvents." The solvents chosen for this study are ethylene dichloride (EDC), methyl isobutyl ketone (MIBK), butyl acetate (BA), anisole ($PhOCH_3$) and chlorobenzene (PhCl).

EXPERIMENTAL AND RESULTS

To a 250 mL Erlenmeyer flask at room temperature were added 0.03 mole quat (12.96 g of Aliquat® 336 or 9.66 g Aliquat® 100 or 9.42 g 75% aqueous Aliquat® 175), 100 g aqueous phase (deionized water or 10% NaCl) and 100 g organic liquid. The two phase systems were mixed, allowed to separate and sampled. The aqueous phases of the Aliquat® 336 experiments and the organic phases of the Aliquat® 175 and Aliquat® 100 experiments were measured for nitrogen. Nitrogen analysis was performed using an Antek Model 703C Chemiluminescent Nitrogen System. The method of analysis involves a high temperature oxidation of the entire sample, converting any chemically bound nitrogen to NO. The NO is contacted with ozone to produce metastable NO_2^*. As the NO_2^* relaxes, it chemiluminesces and this light emission is proportional

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[6] Aliquat is a registered trademark of Cognis Corporation

to the amount of nitrogen in the sample. The distribution of the three quat salts between organic solvents and aqueous phases is shown in Table 1 (two significant digits reported). Calibration curves were run with standards prior to each set of measurements and precision appeared to be within ±5%. The nitrogen values using the chemiluminescence method are thought to be more accurate at the lower concentrations. Thus, the measurements were performed on the phase which had the lower concentration of quat salt, usually the organic phase for Aliquat® 175 and Aliquat® 100, and the aqueous phase for Aliquat® 336. In the case of Aliquat® 100 in ethylene dichloride, very high concentrations of quat salt (greater than 10,000 ppm) were observed in both phases. The Aliquat® 100 values obtained in both phases may be outside the range of

Table 1 *Distribution of Quaternary Ammonium Salts Between Selected Organic and Aqueous Phases*

Quat	Organic Phase	Aqueous Phase	% Quat Salt in Organic Phase	D^{aq}_{org}
Aliquat® 175	EDC	water	0.15%	660
		10% NaCl	0.95%	100
Aliquat® 100	EDC	water	21%	3.8
		10% NaCl	41%	1.5
Aliquat® 336	EDC	water	99.7%	0.0031
		10% NaCl	99.94%	0.00057
Aliquat® 175	MIBK	water	0.38%	260
		10% NaCl	1.4%	71
Aliquat® 100	MIBK	water	7.6%	12
		10% NaCl	33%	2.1
Aliquat® 336	MIBK	water	99.7%	0.0031
		10% NaCl	99.97%	0.00028
Aliquat® 175	butyl acetate	water	0.15%	650
		10% NaCl	0.18%	550
Aliquat® 100	butyl acetate	water	0.42%	240
		10% NaCl	1.4%	70
Aliquat® 336	butyl acetate	water	99.6%	0.0036
		10% NaCl	99.97%	0.00031
Aliquat® 175	anisole	water	0.28%	350
		10% NaCl	0.34%	290
Aliquat® 100	anisole	water	0.36%	270
		10% NaCl	1.4%	73
Aliquat® 336	anisole	water	99.7%	0.0033
		10% NaCl	99.95%	0.00049
Aliquat® 175	chlorobenzene	water	<0.01%	>10,000
		10% NaCl	0.07%	1,500
Aliquat® 100	chlorobenzene	water	0.14%	700
		10% NaCl	0.54%	190
Aliquat® 336	chlorobenzene	water	99.6%	0.0037
		10% NaCl	99.93%	0.00068

EDC = ethylene dichloride MIBK = methyl isobutyl ketone

accurate measurement. In addition, alkyl halides, such as ethylene dichloride, may interfere with organic phase nitrogen measurement by this method. Therefore, in the cases of Aliquat® 175 and Aliquat® 100 in ethylene dichloride, the organic phases generated were extracted twice *more* with water and these additional water extracts of the organic phases were measured for quat salt content. In the case of Aliquat® 175 in ethylene dichloride, reliable low values were obtained for the aqueous extractions of the organic phase. In the case of Aliquat® 100, very high quat salt values were obtained, which confirmed that much Aliquat® 100 was in both phases (i.e., Aliquat® 100 distributes extensively into both phases). Thus, for Aliquat® 100 the direct measurements on the organic phases were used and are shown in Table 1. In all cases of double extraction of samples of Aliquat® 175 from ethylene dichloride into fresh water, the final water extraction contained no detectable nitrogen, indicating that all of the quat salt was extracted prior to the final extraction.

The distribution of the quat salts between the organic solvents and 10% NaCl was generated to simulate (imperfectly) the reaction matrix at the end of a PTC reaction, since most PTC systems contain significant amounts of salt (e.g., from excess reagents and leaving groups) in the aqueous phase during the reaction. Future studies will be conducted with much higher salt concentration (e.g., 30% NaCl or 50% NaOH, if possible) to more representatively reflect typical PTC conditions. The salt level of 10% NaCl was chosen based upon analytical limitations at this time.

Calculations: The weight of quat salt per weight of sample phase was converted to actual total weight of quat salt in the phase sampled, corrected for molecular weight and phase density. The actual weight of the quat salt in a sampled phase was compared to the total initial charge of quat salt to obtain "% quat salt in the sampled phase." For Aliquat® 175 and Aliquat® 100, the calculation of "% quat salt in the organic phase" was performed directly on organic phase measurements. The "% quat salt in the organic phase" for Aliquat® 336 was calculated from the aqueous phase measurements (100% - %Aliquat® 336 in the aqueous phase).

A quantitative parameter was defined to describe the distribution of quat salts between an organic phase and an aqueous phase. This distribution parameter, "D^{aq}_{org}," reflects the ratio of the amount of quat salt which distributes in 100 g of aqueous phase relative to the amount of the quat salt which distributes in 100 g of organic phase, of a two phase organic- aqueous system. Thus, the as D^{aq}_{org} increases, it becomes easier to wash the quat salt into water and the residual level of quat salt in the organic phase decreases. Note that the ratio is based on weight of quat salt per weight of liquid phase (not per volume).

D^{aq}_{org} was calculated from the "% in the organic phase" using the assumptions: [1] all of the quaternary ammonium salt was soluble in the two phase system, [2] none of the quaternary ammonium salts formed a third phase (this was not thoroughly verified, it was assumed), [3] for Aliquat® 175 and Aliquat® 100, the amount of quat salt in the aqueous phase was the difference between the amount of quat salt initially charged to the system and the amount of quat salt measured in the organic phase; for Aliquat® 336, the amount of quat salt in the organic phase was the difference between the amount of Aliquat® 336 initially charged to the system and the amount of Aliquat® 336 measured in the aqueous phase.

DISCUSSION

Quat Salt Distribution

As expected, Aliquat® 175 (13 carbons) distributes more into water from the solvents shown, relative to Aliquat® 100 (16 carbons). Also as expected, Aliquat® 336 (weighted average of 26 carbons) distributes > 99% into all solvents examined in the presence of fresh water and > 99.9% in the presence of 10% NaCl. These qualitative conclusions are not surprising, however the actual values are very useful as shown below.

The differences in distribution behavior of Aliquat® 175 and Aliquat® 100 between aqueous and organic phases appears to fall into two categories. In all cases, Aliquat® 175 distributes into the organic solvent from fresh water at < 0.4%. In contrast, Aliquat® 100 distributes into the organic solvent from fresh water at < 0.4% only from relatively low polarity solvents such as chlorobenzene (ε = 5.6), butyl acetate (ε ~ 5-6) and anisole (ε = 4.3). In moderately polar solvents, such as MIBK (ε = 13) and EDC (ε = 10), Aliquat® 100 distributes into the organic phase from fresh water 7.6% and 21%, respectively, which is 20 and 140 times more, respectively, than Aliquat® 175. Depending on the solvent, both Aliquat®

175 and Aliquat® 100 distribute 20% more to 4 times more into fresh water than into 10% NaCl. These differences between the distribution behaviors of Aliquat® 175 and Aliquat® 100, have significant ramifications on catalyst separation.

One key objective of this study is to provide data to industrial chemists/engineers to estimate [1] the expected volumes of aqueous waste streams and [2] residual levels of quat salts in the product phase, when extracting quat salts such as Aliquat® 175 and Aliquat® 100 from the organic phase/product into aqueous washes. Following is an example of how the data can be used to perform such estimations. In this hypothetical example, we assume: [1] at the end of a reaction the aqueous phase is similar in solubility properties to 10% NaCl, [2] the organic phase is similar in solubility properties to MIBK, [3] the weights of organic phase and aqueous phase are equal and [4] the total amount of quat salt in the system is 10,000 ppm (1 wt%). Under such conditions there would be 140 ppm Aliquat® 175 or 3300 ppm Aliquat® 100 in the MIBK phase. Assuming these organic phases were washed twice with fresh water in weights equal to the weight of the organic phase, then Table 2 shows the calculated amount of residual catalyst in the organic phase. An important observation with practical ramifications is that in this hypothetical MIBK system, one could perform only *half of the water wash* (reducing aqueous waste stream volume) and still *achieve more than an order of magnitude less residual quat salt* using Aliquat® 175 compared to using Aliquat® 100. Using two fresh water washes in this MIBK system, the residual level of Aliquat® 175 may be *four orders of magnitude* less than for Aliquat® 100 and would be undetectable by commonly known analytical methods.

Similar calculations are shown in Table 3 for an ethylene dichloride hypothetical system. Table 3 shows a more dramatic advantage in favor of Aliquat® 175 in the ethylene dichloride system vs the MIBK system. In fact, removal of Aliquat® 100 from ethylene dichloride is likely to require very extensive water washing and should generate significant volumes of aqueous waste stream. Even in ethylene dichloride systems in which Aliquat® 175 does not induce as much reactivity as Aliquat® 100, it still may be worthwhile to consider using larger quantities of Aliquat® 175 in place of Aliquat® 100, just to obtain better separation with comparable reactivity.

Similar calculations are shown in Table 4 for a butyl acetate hypothetical system. These data may be useful when considering the separation characteristics of a PTC system at the end of a solvent-free esterification. Table 4 shows a less dramatic advantage in favor of Aliquat® 175 in the butyl acetate system vs the MIBK system (though still a 50-fold difference in catalyst residue in butyl acetate after one fresh water wash).

One may ask how can Aliquat® 175 be an effective catalyst in MIBK, for example, if only 1.4% of the quat salt distributes into the organic phase in the presence of 10% NaCl. The answer is that most PTC systems work at very high ionic strength, and Aliquat® 175 in particular, excels in PTC reactions using 50% NaOH. At these ionic strengths, even the most hydrophilic quat salts are salted out of the aqueous phase. As noted above, attempts will be made in the future to measure D^{aq}_{org} at significantly higher ionic strengths to be more representative of real PTC systems. Note that in some cases with high ionic strength, a third phase can be formed (e.g., with Bu_4N^+ salts[7,8,9]) and very high reactivity may be observed. The possibility of the formation of a third phase would greatly complicate the measurements of D^{aq}_{org} and any resulting interpretations. Such systems are rarely reported but we should be aware of the possibility.

In many industrial applications, it is advantageous to purify the product by distillation, recrystallization or extraction of the organic product (e.g., carboxylate salts) into water. In such cases, one could greatly minimize (nearly eliminate) contamination of aqueous waste streams with quat salt, by choosing a quaternary ammonium phase-transfer catalyst which distributes nearly quantitatively into the organic phase. It may be seen from the data in Table 1 that Aliquat® 336 distributes nearly quantitatively into polar and non-polar organic solvents, especially in the presence of

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Table 2 *Aqueous Waste Streams Generated in Separating Aliquat® 175 and Aliquat® 100 from MIBK by Extraction Into Water (initial total [quat salt] = 10,000 ppm)*

% Quat Salt in MIBK in contact with	Aliquat® 175	Aliquat® 100
10% NaCl	140 ppm	3300 ppm
after 1st water wash	0.53 ppm	250 ppm
after 2nd water wash	*2 ppb*	19 ppm

Table 3 *Aqueous Waste Streams Generated in Separating Aliquat® 175 and Aliquat® 100 from EDC by Extraction Into Water (initial total [quat salt] = 10,000 ppm)*

% Quat Salt in EDC in contact with	Aliquat® 175	Aliquat® 100
10% NaCl	95 ppm	4100 ppm
after 1st water wash	0.14 ppm	860 ppm
after 2nd water wash	*0.2 ppb*	180 ppm

Table 4 *Aqueous Waste Streams Generated in Separating Aliquat® 175 and Aliquat® 100 from Butyl Acetate by Extraction Into Water (initial total [quat salt] = 10,000 ppm)*

% Quat Salt in Butyl Acetate in contact with	Aliquat® 175	Aliquat® 100
10% NaCl	18 ppm	140 ppm
after 1st water wash	*32 ppb*	0.59 ppm
after 2nd water wash	*0.06 ppb*	*2 ppb*

high ionic strength of the aqueous phase. Aliquat® 336 distributes 99.7% into toluene in a toluene/water system and 99.96% into toluene in a toluene/10% NaCl system. Indeed, in many industrial applications, Aliquat® 336 is used when the product is distilled, recrystallized or extracted into water. In such cases, it is usually economically feasible to incinerate the still bottoms or other organic stream containing the Aliquat® 336. Aliquat® 336 is the most organophilic large volume phase-transfer catalyst and is advantageous when very high distribution into the organic phase is required to enhance reactivity and catalyst separation.

Reactivity

The pKa guidelines[10] suggest that base-promoted reactions of substrates with acidity in the pKa range 16-23 should work well with "accessible" quats with a "q value" in the range 1.3-2.0. Aliquat® 175 has a q value of 1.75 (hence its name) which suggests it may be suitable for performing base-promoted reactions with

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substrates in the pKa range of 16-23. We recently published the superior reactivity of the C-alkylation of phenylacetonitrile (pKa ~16) in the presence of Aliquat® 175 vs Aliquat® 100.[11] The data in the current paper show that Aliquat® 175 extracts into water much better than Aliquat® 100 from several common PTC solvents. Based on the pKa guidelines and the results reported here, it is now recommended that when base-promoted reactions are being performed on substrates in the pKa range of 16-23, Aliquat® 175 should be screened as the catalyst since it is likely to induce higher reactivity as well as better catalyst separation. This recommendation is emphasized because chemists usually choose tetrabutylammonium bromide (TBAB) *FIRST* when developing new processes and syntheses, since TBAB usually works and can be separated by extraction into water.

There are several publications which compare Aliquat® 336 with tetrabutylammonium bromide and show that the Aliquat® 336 induces higher reactivity. In these cases, the quat salt can be separated by distillation, recrystallization, extraction into water or extraction into a non-polar solvent (proposed method of separation shown in parentheses): solvent-free esterification[12] (distillation of product), etherification[13] (distillation of product), S-alkylation[14] (distillation of product), hydrolysis[15] (extraction of product into water), polymerization[16] (extraction of catalyst into non-polar organic solvent), oxidation[17] (recrystallization of product). In these cases, it may be advantageous to keep the quat salt in the organic phase to avoid the presence of quat salt in the aqueous waste stream and/or it may be possible to recycle the quat salt. Therefore, in these cases, Aliquat® 336 would provide both more favorable reactivity and more favorable separation relative to Aliquat® 100.

It should be noted that there are PTC applications in which Aliquat® 100 is probably the best commercially available catalyst. Such cases exist when the catalyst must be separated by extraction into water (e.g., the product cannot be distilled, recrystallized, etc.) AND Aliquat® 175 does not work. Other cases are those rare systems, as noted above, which are fortunate enough to form a third phase with Aliquat® 100[18] and enjoy particularly high reactivity.

CONCLUSION

In summary, in many cases it is possible to simultaneously achieve excellent catalyst separation and high reactivity in PTC systems by choosing an appropriate phase-transfer catalyst (and solvent). As a first pass guideline, one should use Aliquat® 175 when an “accessible quat” is best (e.g., many alkylations and condensations of substrates with a pKa of 16-23) AND when it is desired to separate the catalyst from the product by extracting the catalyst into water. One should choose Aliquat® 336 when an “organophilic quat” is best (many nucleophilic substitutions, oxidations, etc.) AND when it is desired to separate the catalyst from the product by distilling the product, recrystallizing the product, extracting the product into water or extracting the catalyst into a non-polar solvent in which the product is not soluble. One should choose Aliquat® 100 when Aliquat® 175 does not work and it is still required to separate the catalyst from the product by extracting the catalyst into water. One should also choose Aliquat® 100 when a third phase rich in this catalyst can be formed (this usually requires a lot of development resource to discover/optimize).

It should be noted that most chemists consider Aliquat® 100 and Aliquat® 336 to be the classical general purpose phase-transfer catalysts. It is true that these catalysts induce high reactivity in most PTC reactions and they can be effectively separated for the product by either aqueous or organic workup procedures as described above. However, better reactivity and separation may be achieved in some cases by appropriate choice of quat salt and solvent. In particular, the authors believe (and have seen evidence in industrial applications) that the performance of many commercial PTC processes can be significantly

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improved after evaluating the use of Aliquat® 175 in place of Aliquat® 100.

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New PTC Company

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The industry-wide driving force of achieving reduced cost of manufacture is driving the demand for the services of PTC Organics. A combination of two fundamental factors must be recognized to understand why PTC Organics is well positioned to meet customer needs. First, phase-transfer catalysis technology does in fact reduce the cost of manufacture of many organic chemicals by achieving high yield, reduced cycle time, solvent-free or reduced-solvent processes, better selectivity, replacement or reduction of expensive and/or hazardous raw materials, reduction of unit operations, ability to work with water sensitive reagents and other benefits. Second, process development and scale up departments at most companies do not have the time, and sometimes, the expertise to develop these highly favorable PTC processes. Customers realize multiple simultaneous benefits by outsourcing to PTC Organics: [1] customers benefit from the inherent advantages PTC has to offer, primarily lower cost and higher quality, [2] customers do not have to invest their already constrained R&D resource to achieve the benefits of PTC, [3] overall development cycle time is greatly reduced since PTC Organics has the expertise, focus and equipment to develop PTC processes.

PTC Organics offers the most benefit to customers whose processes/products can really benefit from phase-transfer catalysis, which is the unique competitive advantage of PTC Organics. The reactions which are most amenable to achieving highly favorable process performance using PTC include etherification, esterification, N-alkylation, C-alkylation, S-alkylation, dehydrohalogenation, aldol condensation, Michael addition, reactions with HCl/HBr, nucleophilic displacements using cyanide, fluoride, iodide and many other inorganic/organic nucleophiles.

PTC Organics welcomes inquiries for improving or developing proceses for which phase-transfer catalysis may be able to offer significant benefit.