# Phase Transfer Agents. I. Transfer of Permanganate, Periodate, and Cyanate Ions from Aqueous to Organic Phases. Preparation of Quaternary Onium Periodates[1]

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## ABSTRACT AND SUMMARY

Several quaternary ammonium and phosphonium halides have been shown to be useful phase transfer agents (PTA) for the efficient transport of permanganate, periodate, and cyanate ions from aqueous to organic phases (benzene and/or methylene chloride). Chemical and spectral methods of analysis were used to measure ion transfer quantitatively and to assess the relative efficiency of PTA.

## INTRODUCTION

The reaction of inorganic ions with organic substrates is often exceedingly slow because of the inability of the reactants to make effective contact on a molecular level. The use of polar hydroxylic solvents to achieve homogeneity is widely employed but preferential solvation of ions may result in low overall reaction rates. The use of highly polar aprotic solvents, such as dimethyl sulfoxide, dimethylformamide, and hexamethylphosphortriamide, has permitted reactions to take place between inorganic and organic species but those solvents often require absolutely anhydrous conditions and their recovery or complete removal is sometimes troublesome and time-consuming. Large increases in the surface area of reactants, exemplified by micellar systems (1), represent another useful way of speeding up reactions but the preparation and, particularly, the breaking of emulsions (2) in order to isolate reaction products can be a formidable task.

A promising approach to reaction rate enhancement between inorganic and organic substances is the use of phase transfer agents (PTA). PTA are compounds, typically although not exclusively quaternary ammonium or phosphonium halides, that transfer inorganic ions from aqueous to organic phases and, for reasons that are not yet clear, cause marked rate accelerations in many cases. This process of ion transfer is sometimes referred to as "ion-pair extraction" (3-5). Although ion transfer has been known for some years (3) it has been only recently that the variables controlling efficient ion transfer have begun to be understood largely through the work of Starks (6,7) and of Herriott and Picker (8). [The subject of phase transfer has been reviewed recently (9,10).]

We have long been interested in reactions of water-soluble inorganic oxidants (2) and nucleophiles (11) with lipids and related compounds. We often encountered considerable difficulty in effecting reaction presumably because of the immiscibility of the reactants. The new knowledge that had become available on PTA prompted us to initiate studies on the efficiency of transfer of several inorganic ions of interest (permanganate, periodate, cyanate) from water to two organic solvents (benzene, methylene chloride) widely used in lipid and phase transfer reactions with approximately ten quaternary and phosphonium halides encompassing a range of structures. In this paper we are reporting simple, accurate, and rapid chemical and spectral methods for quantitatively evaluating the effectiveness of PTA in transferring ions from the aqueous to the organic phases. During the course of our work we prepared and characterized some new quaternary ammonium and phosphonium salts.

## RESULTS AND DISCUSSION

### Extraction of Inorganic Ions from Water to Organic Phases Using PTA

*(a) Permanganate Ion:* When equal volumes of benzene or methylene chloride and ca. 0.1 *N* (0.021 *M*) aqueous potassium permanganate solution are shaken vigorously in a separatory funnel, the organic layer remains colorless. In marked contrast, if certain quaternary ammonium or phosphonium halides in excess are predissolved in the organic solvent, a color inversion occurs yielding "purple benzene" (12) or "purple methylene chloride," the aqueous phase becomes colorless in certain cases, and a quantitative to nearly quantitative transfer of $MnO_4^-$ to the organic phase occurs. Results are summarized in Table I.

The most efficient PTA for $MnO_4^-$ transfer, given in only approximate rank order as some of the differences are small and probably not significant, are tetrabutylphosphonium chloride (Expt. 5) with either benzene or methylene chloride; tetrabutylammonium bromide (Expt. 1), hexadecyltrimethylammonium bromide (Expt. 4), and benzyltriethylammonium chloride (Expt. 3) with methylene chloride but *not* with benzene; and tri(*n*-octyl)-*n*-propylammonium bromide (Expt. 6) with benzene but *not* with methylene chloride. Our results are similar to and confirm those reported earlier by Herriott and Picker (12) for the transport of $MnO_4^-$ from water to benzene, although there are some minor discrepancies.

Although tetrabutylammonium bromide (Expt. 1) produces a virtually colorless aqueous phase and a purple benzene solution, ca. 60% of the $MnO_4^-$ is unaccounted for, in contrast with the methylene chloride system. In the benzene-water system both phases contain insoluble material near the interface; this is probably tetrabutylammonium permanganate (see later discussion). Hexadecyltrimethylammonium bromide (Expt. 4) and benzyltriethylammonium chloride (Expt. 3), which are both effective in transferring $MnO_4^-$ to methylene chloride, are almost totally ineffective in the benzene-water system. In addition, ca. 40-80% of $MnO_4^-$ is unaccounted for and a large quantity of precipitate is found in the aqueous but not in the benzene phase. Although tri(*n*-octyl)-*n*-propylammonium bromide (Expt. 6) is moderately effective in transferring $MnO_4^-$ to methylene chloride, approximately 20% of $MnO_4^-$ is unaccounted for (contrast with benzene). Tetraethylammonium bromide (Expt. 2) is totally ineffective in transferring $MnO_4^-$ to benzene which remains colorless but

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TABLE I

Extraction of $MnO_4^-$ from Water into Organic Solvents Using Phase Transfer Agents (PTA)[a]

| Expt. No. | PTA | $MnO_4^-$ Concentration, $\underline{N}$ | | | |
|---|---|---|---|---|---|
| | | $H_2O$-$C_6H_6$ System | | $H_2O$-$CH_2Cl_2$ System | |
| | | $H_2O$ Phase | $C_6H_5$ Phase | $H_2O$ Phase | $CH_2Cl_2$ Phase |
| 1 | $(CH_3CH_2CH_2CH_2)_4\overset{+}{N}\,Br^-$ | $0.003^b$ | $0.042^b$ | 0.001 | $0.100^c$ |
| 2 | $(CH_3CH_2)_4\overset{+}{N}\,Br^-$ | 0.091 | colorless | 0.065 | $0.017^d$ |
| 3 | $C_6H_5CH_2(CH_3CH_2)_3\overset{+}{N}\,Cl^-$ | 0.017 | $0.0004^e$ | 0.010 | $0.094^c$ |
| 4 | $[CH_3(CH_2)_{15}](CH_3)_3\overset{+}{N}\,Br^-$ | 0.061 | $0.002^e$ | 0.001 | $0.105^c$ |
| 5 | $(CH_3CH_2CH_2CH_2)_4\overset{+}{P}\,Cl^-$ | 0.001 | $0.099^c$ | 0.0004 | $0.101^c$ |
| 6 | $[CH_3(CH_2)_7]_3(CH_3CH_2CH_2)\overset{+}{N}\,Br^-$ | 0.001 | $0.096^c$ | 0.0002 | $0.086^d$ |

[a] $C_6H_6$ (25 ml) or $CH_2Cl_2$ containing 0.00075 mol of PTA and 25 ml of 0.105 $\underline{N}$ $KMnO_4$ containing 0.0005 mol of $KMnO_4$ were vigorously shaken. After phase separation, aliquots of both phases were analyzed by iodometry for $MnO_4^-$ content (see Experimental Procedures). (See Reference 12 for additional data.)

[b] Both phases contained insoluble material; ca. 60% of $MnO_4^-$ unaccounted for.

[c] Almost quantitative transfer of $MnO_4^-$ to the organic phase.

[d] Ca. 20% of $MnO_4^-$ unaccounted for.

[e] Precipitate in aqueous phase; ca. 40-80% of $MnO_4^-$ unaccounted for.

TABLE II

Extraction of $IO_4^-$ from Water into Organic Phases Using Phase Transfer Agents (PTA)[a]

| Expt. No. | PTA | $IO_4^-$ Concentration, $\underline{N}$ | |
|---|---|---|---|
| | | $H_2O$ Phase ($H_2O$-$C_6H_6$) | $H_2O$ Phase ($H_2O$-$CH_2Cl_2$) |
| 7 | $(CH_3CH_2CH_2CH_2)_4\overset{+}{N}\,Br^-$ | 0.000 | 0.007 |
| 8 | $(CH_3CH_2CH_2CH_2)_4\overset{+}{P}\,Cl^-$ | 0.000 | 0.000 |
| 9 | $(CH_3CH_2)_4\overset{+}{N}\,Br^-$ | 0.088 | 0.092 |
| 10 | $C_6H_5CH_2(CH_3CH_2)_3\overset{+}{N}\,Cl^-$ | 0.073 | 0.034 |
| 11 | $[CH_3(CH_2)_{15}](CH_3)_3\overset{+}{N}\,Br^-$ | 0.021 | 0.002 |
| 12 | $[CH_3(CH_2)_7]_3(CH_3CH_2CH_2)\overset{+}{N}\,Br^-$ | 0.000 | 0.005 |
| 13 | $[CH_3(CH_2)_4]_4\overset{+}{N}\,I^-$ | -----b | -----b |
| 14 | $[CH_3(CH_2)_3]_4\overset{+}{N}\,I^-$ | -----b | -----b |
| 15 | $(CH_3)_4\overset{+}{N}\,I^-$ | -----b | -----b |

[a] See Table I, footnote a, for the procedure used; $NaIO_4$ solution was used in place of $KMnO_4$. The original concentration of $NaIO_4$ in the aqueous phase was 0.105 $\underline{N}$ (0.05 $\underline{M}$).

[b] Quaternary ammonium iodides could not be studied as they liberated iodine when benzene or methylene chloride solutions were mixed with aqueous $NaIO_4$.

it transfers some $MnO_4^-$ to methylene chloride; 20% of $MnO_4^-$ remains unaccounted for in the latter case. The failure of tetraethylammonium bromide to transfer $MnO_4^-$ to the organic phases probably reflects the higher water-solubility of the ion-pair thus resulting in an unfavorable partition coefficient.

*(b) Periodate Ion:* Study of the transfer of $IO_4^-$ from the aqueous phase to benzene or methylene chloride was conducted in the same manner as described for $MnO_4^-$. Iodometric analysis for $IO_4^-$ could be conducted reproducibly only on the aqueous phase. The change in concentration of $IO_4^-$ in the aqueous phase before and after mixing was used as the criterion of phase transfer effectiveness. Table II summarizes the results.

PTA that are about equally effective in transferring $IO_4^-$ from the aqueous to the organic phases are tetrabutylphosphonium chloride (Expt. 8), tetrabutylammonium bromide (Expt. 7) and tri(*n*-octyl)-*n*-propylammonium bromide (Expt. 12) with *both* benzene and methylene chloride; and hexadecyltrimethylammonium bromide (Expt. 11) with methylene chloride but *not* with benzene.

Tetraethylammonium bromide (Expt. 9) is almost totally ineffective in transferring $IO_4^-$ to either benzene or methylene chloride (see Table I, Expt. 2 for a similar result in $MnO_4^-$ transfer). Benzyltriethylammonium chloride (Expt. 10) is also relatively ineffective with both benzene and methylene chloride.

*(c) Cyanate Ion:* Transfer of $\bar{O}CN$ from aqueous solution to methylene chloride was determined by infrared spectroscopic examination of the aqueous phase before and after shaking with a methylene chloride solution of various PTA using calcium fluoride cells. The characteristic absorption of $\bar{O}CN$ at 2180 $cm^{-1}$ permits the construction of a standard Beer-Lambert plot; linearity is obtained over the range 0-1 $\underline{M}$ in a plot of concentration of $\bar{O}CN$ against the logarithm of relative transmittance at 2180 $cm^{-1}$ (log $T_{H_2O}/T_{solution}$). Table III summarizes the results.

Even with the most effective PTA, tetrabutylphosphonium chloride (Expt. 6), only 60-65% of the $\bar{O}CN$ is transferred to the methylene chloride phase. This result is in contrast with our experience with $MnO_4^-$ and $IO_4^-$ which could be quantitatively transported to organic phases with certain PTA (Tables I, II). Even with a 50% molar excess of phosphonium chloride over $\bar{O}CN$ (Expt. 16b) only a slight increase is observed in the quantity of $\bar{O}CN$ transferred from the aqueous to the organic phase, in comparison with equimolar quantities of PTA and $\bar{O}CN$ (Expt. 16a).

TABLE III

Extraction of $\bar{O}CN$ from Water into Methylene Chloride[a]

| Expt. No. | PTA | KOCN Conc. in Aqueous Phase, M | % Initial KOCN Transferred to $CH_2Cl_2$ Phase |
|---|---|---|---|
| 16a | $(CH_3CH_2CH_2CH_2)_4\overset{+}{P}\ Cl^{-b}$ | 0.20 | 60 |
| 16b | $(CH_3CH_2CH_2CH_2)_4\overset{+}{P}\ Cl^{-c}$ | 0.18 | 64 |
| 17 | $(CH_3CH_2CH_2CH_2)_4\overset{+}{N}Br^-$ | 0.35 | 30 |
| 18 | $C_6H_5CH_2(C_2H_5)_3\overset{+}{N}Cl^-$ | 0.38 | 24 |
| 19 | $[CH_3(CH_2)_7]_3CH_3CH_2CH_2\overset{+}{N}Br^-$ | 0.42 | 16 |
| 20 | $n$-$C_{16}H_{33}(CH_3)_3\overset{+}{N}Br^-$ | 0.42 | 16 |
| 21 | $[CH_3(CH_2)_4]_4\overset{+}{N}I^-$ | 0.46 | 8 |

[a] Initial concentration of aqueous KOCN solution, 0.5 M. Equal volumes of $CH_2Cl_2$ (10 ml) *containing* Phase Transfer Agents(PTA) (5 mmol) and KOCN solution (10 ml containing 5 mmol) were shaken vigorously. After separation of the layers, the aqueous phase was examined by infrared spectroscopy at 2180 $cm^{-1}$ using calcium fluoride cells.

[b] Equimolar quantites of PTA:KOCN.

[c] 50% molar excess of PTA:KOCN.

### Preparation of Quaternary Ammonium and Phosphonium Permanganates and Periodates

The observation that higher reaction rates are obtained when greater than catalytic quantities of PTA to substrate are used (6-8) prompted us to study the preparation and characterization of quaternary onium permanganates and periodates with the object of using these new PTA in stoichiometric quantities as high speed oxidizing agents in organic media.

When equal volumes of 0.1 M aqueous solutions of tetrabutylammonium bromide and potassium permanganate are mixed immediate precipitation of a purple solid, assumed to be tetrabutylammonium permanganate, occurs. After cooling in ice, filtering and vacuum drying, a 65-70% yield of an unstable highly reactive purple solid is obtained which immediately begins to turn brown. Storage under high vacuum over phosphorus pentoxide slows down but does not completely inhibit the production of a brown product ($MnO_2$?). Analysis of freshly prepared material for oxidizing capacity by iodometry yields values ranging from 70-95% of that calculated and the values decrease with time. On heating [CAUTION: MILLIGRAM QUANTITIES ONLY] the tetrabutylammonium permanganate decomposes explosively or with ignition at about 100-105 C. Similar results are obtained with tetrabutylphosphonium permanganate which also decomposes at about 100 C. Triphenylarsonium permanganate is also unstable and decomposes slowly at room temperature and explosively at 108 C (13).

In contrast ammonium and phosphonium periodates are easily prepared usually in good to excellent yields by mixing equimolar solutions of aqueous sodium periodate and quaternary onium chlorides or bromides (but *not* iodides) dissolved in water, methyl acetate-water or aqueous ethanol as required (Table IV). The chilled (O C) reaction mixtures are filtered and the white precipitates are vacuum dried (50-99% yields). These reaction products are virtually analytically pure (iodometric analysis) without further treatment and their melting points are either identical with or only slightly lower than those of the recrystallized analytical samples (25-80% yields). NMR spectra are consistent with proposed structures.

Quaternary onium periodates are stable in the dark but slowly turn yellow on exposure to diffuse light. They are not significantly hygroscopic but they were dried under vacuum at 30-60 C prior to analysis. With the exception of tetraphenylphosphonium periodate which is quite insoluble, the periodates listed in Table IV are slightly soluble in methylene chloride, chloroform, benzene and dimethyl sulfoxide.

### Miscellaneous

Reaction of aqueous tetrabutylammonium bromide with an equimolar quantity of aqueous potassium dichromate yielded easily filtered organge crystals which turned black within a few hours; no further work was conducted on onium dichromates.

Sodium iodate did not yield a precipitate of quaternary ammonium iodate on treatment with tetrabutylammonium bromide.

## EXPERIMENTAL PROCEDURES

### Starting Materials

PTA were purchased from Aldrich Alfa Inorganics, (Milwaukee, WI); Columbia (Columbia, SC); Eastman, (Rochester, NY); and/or Fluka AG (Hauppauge, NY). Sodium periodate, potassium permanganate, and potassium cyanate were the purest reagent grades. Best grades of benzene and methylene chloride were dried and fractionally distilled before use (15).

### Analytical Procedures

$MnO_4^-$ in aqueous or solvent phases was determined iodometrically: (a) aqueous solutions (1-10 ml depending on $MnO_4^-$ concentration) were dissolved in 2 N acetic acid (5-30 ml) and aqueous potassium iodide (5 ml of a 30% solution) was added. Liberated iodine was titrated with 0.02 N sodium thiosulfate using starch indicator; (b) benzene or methylene chloride solutions were analyzed similarly except that acetone (30 ml) was added before the potassium iodide to obtain a homogeneous solution.

$IO_4^-$ in aqueous solutions was determined iodometrically (16).

Potassium cyanate was determined in aqueous solutions by infrared spectroscopy using $CaF_2$ cells and measuring transmittance at 2180 $cm^{-1}$. A Beer-Lambert plot of potassium cyanate concentration against log $(T_{H_2O}/T_{solution})$ was linear from 0-1 M.

### Phase Transfer Studies

(a) *$MnO_4^-$*. Table I, footnote a, gives the procedure. Aliquots of the clear aqueous and organic phases were analyzed iodometrically. Precipitation at the interface was ignored thus accounting for the failure to obtain a satisfactory material balance for $MnO_4^-$ in some of the experiments. When precipitation was absent, >95% of $MnO_4^-$ was accounted for. (b) *$IO_4^-$*. Table II, footnote a, gives the procedures. Only the aqueous phase gave reproducible

TABLE IV

Quaternary Ammonium and Phosphonium Periodates[a]

| | | | Elemental Analysis, % | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | Calculated | | | | Found | | | |
| Compound | MP, °C[b] | Yield,[c] % | C | H | N or P | I | C | H | N or P | I |
| $(CH_3CH_2CH_2CH_2)_4\overset{+}{N}\,\overset{-}{I}O_4$[d,e] | 178<br>182 ($C_2H_5OH$) | 50 | 44.3 | 8.3 | 3.2 | 29.3 | 44.0 | 8.0 | 3.3 | 29.1 |
| $(C_2H_5)_3C_6H_5CH_2\overset{+}{N}\,\overset{-}{I}O_4$[f,g] | 148<br>149 (1:1<br>($C_2H_5OH:H_2O$) | 80 | 40.7 | 5.7 | 3.7 | 33.1 | 40.8 | 5.6 | 3.8 | 32.9 |
| $(CH_3)_3$-$\underline{n}$-$C_{16}H_{33}\overset{+}{N}\,\overset{-}{I}O_4$[d,h] | 130-5<br>134-6(1:1<br>($C_2H_5OH:H_2O$ | 80 | 48.0 | 8.8 | 3.0 | 26.7 | 48.2 | 8.8 | 3.0 | 26.8 |
| ($\underline{n}$-$C_8H_{17})_3CH_3CH_2CH_2\overset{+}{N}\overset{-}{I}O_4$[d,i] | 76<br>79-80 (1:1<br>$C_2H_5OH:H_2O$ | 90 | 55.2 | 9.9 | 2.4 | 21.6 | 55.4 | 9.8 | 2.4 | 21.9 |
| $(C_2H_5)_4\overset{+}{N}\,\overset{-}{I}O_4$[j] | 176-7<br>($\underline{t}$-$C_4H_9OH$) | 82 | --- | --- | --- | --- | --- | --- | --- | --- |
| $(CH_3CH_2CH_2CH_2)_4\overset{+}{P}\,\overset{-}{I}O_4$[e,f] | 179<br>180 ($C_2H_5OH$) | 50 | 42.7 | 8.0 | 6.9 | 28.2 | 42.5 | 7.8 | 6.7 | 28.0 |
| $(C_6H_5)_4\overset{+}{P}\,\overset{-}{I}O_4$[d,f] | 288<br>289 ($CH_3OH$) | 97 | 54.3 | 3.8 | 5.9 | 24.0 | 54.1 | 3.7 | 6.0 | 24.2 |
| $(C_6H_5)_3C_6H_5CH_2\overset{+}{P}\overset{-}{I}O_4$[e,f] | 164-174<br>164 | 99 | 55.2 | 4.0 | 5.7 | 23.4 | 55.2 | 4.2 | 5.9 | 23.4 |
| $(C_6H_5)_3CH_3\overset{+}{P}\overset{-}{I}O_4$[d,e] | 185-6<br>186-7 ($C_2H_5OH$) | 96 | 48.7 | 3.9 | 6.6 | 27.1 | 49.0 | 3.9 | 6.6 | 27.1 |
| $(CH_3CH_2CH_2)_3$-$\underline{n}$-$C_{16}H_{33}\overset{+}{P}\,\overset{-}{I}O_4$[d,e] | 38<br>41-2[k] | 90 | 54.4 | 9.7 | 5.0 | 20.6 | 54.6 | 9.5 | 5.2 | 20.4 |

[a] Prepared by mixing $NaIO_4$ (5 mmol) in 10 ml of $H_2O$ with quaternary ammonium or phosphonium halides (5 mmol) in 2.5-15 ml of $H_2O$, methyl acetate: $H_2O$, 50:50, or ethanol:water, 70:30, as indicated in the footnotes referring to specific compounds (footnotes e,g-i).

[b] Melting points of crude products are given above those of analytically pure compounds. Recrystallizing solvent in parentheses.

[c] Crude products were virtually analytically pure.

[d] Prepared from quaternary bromide.

[e] Quaternary halide was dissolved in 10-15 ml of $H_2O$.

[f] Prepared from quaternary chloride.

[g] Quaternary halide was dissolved in 2.5 ml of $H_2O$.

[h] Quaternary halide was dissolved in 10 ml of 50:50 methyl acetate:$H_2O$.

[i] Quaternary halide was dissolved in 15 ml of 70:30 ethanol:$H_2O$.

[j] Reference 14. This compound was not prepared by us but is tabulated for comparison purposes only.

[k] The crude product was purified by dissolution in ethanol followed by precipitation by $H_2O$.

iodometric analytical results. (c) *OCN.* Table III, footnote a, gives the procedure. Only the aqueous phase gave satisfactory and reproducible spectral analyses for $\overline{O}CN$. There was a rough correlation, however, between the quantity of $\overline{O}CN$ calculated to be transferred to the organic phase from the aqueous phase and the intensity of the 2180 $cm^{-1}$ peak seen in the methylene chloride solutions.

## Preparation of Quaternary Onium Permanganates and Periodates

(a) *Permanganates:* Aqueous potassium permanganate (25 ml, 0.1 $\underline{M}$) was added dropwise with stirring to aqueous tetrabutylammonium bromide or tetrabutylphosphonium chloride (25 ml, 0.1 $\underline{M}$) at room temperature. A purple precipitate formed almost immediately. The reaction solution was cooled to 0 C and filtered as rapidly as possible. The purple solid, assumed to be tetrabutylammonium or phosphonium permanganate, was dried under vacuum (65-70% yield). The products were unstable and turned brown almost immediately. At ca. 100-105 C they decomposed rapidly and sometimes explosively with ignition (CAUTION). Equivalent weights determined iodometrically on fresh materials indicated a $MnO_4^-$ content that ranged from 70-95% of that calculated, and the values decreased with time.

An alternate but less convenient procedure was to freeze-dry benzene solutions obtained in the phase transfer studies reported in Table I. The residues were similar in properties to those just described.

(b) *Periodates (Table IV):* The preparation of tetrabutylammonium periodate is typical. Tetrabutylammonium bromide (1.61 g, 5 mmol) was dissolved in water (10 ml) and an aqueous solution (10 ml) of sodium periodate (1.07 g, 5 mmol) was added with stirring; precipitation occurred almost immediately. The reaction mixture was cooled to 0 C and the precipitate of tetrabutylammonium periodate was filtered and dried under vacuum; the crude product, mp 178 C, weighed 1.06 g (50% yield). It was virtually pure; recrystallization from ethanol yielded white needles, mp 182 C, of analytically pure compound.

This procedure, with modifications noted in the footnotes of Table IV, was used to prepare all the periodates. Yields ranged from 50-99% but no attempt was made to optimize yields. All the periodates were stable and nonhygroscopic for all proactical purposes. Their nuclear magnetic resonance (NMR) spectra, taken in $CDCl_3$ or $(CD_3)_2SO$, differed only slightly from those of the starting quaternary halides and were not particularly useful for diagnostic purposes. The only exceptions were methyl or methylene protons attached to nitrogen or phosphorus whose NMR signals were shifted to slightly higher field (ca. 0.3 ppm). Most of the periodates were slightly soluble in methylene chloride, chloroform, benzene and dimethyl sulfoxide, and relatively insoluble in cold water.

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