

$$\rho = -0.606(\sum\sigma_p^+) + 0.195(\sum E_s) - 1.063 \quad (1)$$

Application of this equation predicts a  $\rho$  value for 2 at 70°C in the range  $-0.84$  to  $-0.90$  in the absence of neighboring group participation.<sup>9</sup>

The reaction of 2 with bromotrichloromethane is straightforward. Hydrogen abstraction occurs exclusively at the benzylic position. The products are the expected dibromides. All kinetic determinations are based on direct competition with mesitylene. They were run in replicate under nitrogen. Conversion to product varied from 10 to 85%. Analyses of reaction mixtures was by GLC. Table I summarizes the relative data obtained corrected to the parent compound.

Table I  
Relative Rates of Secondary Benzylic Hydrogen  
Abstraction from  $\beta$ -(Substituted Phenyl)-Ethyl Bromides

Substituent	Registry no.	$\sigma^+{}^b$	$k_{\text{subst}}/k_{\text{H}}$	No. of runs
<i>p</i> -CH <sub>3</sub> O	14425-64-0	-0.778	4.23 ± 0.21	5
<i>p</i> -CH <sub>3</sub> <sup>a</sup>	6529-51-7	-0.311	1.87 ± 0.26	6
<i>p</i> - <i>t</i> -Bu	56829-61-9	-0.295	1.81 ± 0.16	7
<i>p</i> -F	332-42-3	-0.073	1.08 ± 0.03	6
H	103-63-9	0.000	1.00 ± 0.10	7
<i>p</i> -Cl	6529-53-9	0.114	0.86 ± 0.04	5
<i>m</i> -Cl	16799-05-6	0.399	0.49 ± 0.03	10
<i>m</i> -CF <sub>3</sub>	1997-80-4	0.520	0.33 ± 0.04	7

<sup>a</sup> Corrected for reaction at both benzylic positions. Hydrogen abstraction at the secondary site accounts for 66% of total reactivity. <sup>b</sup> Reference 10.

Optimum correlation was with  $\sigma^+$  parameters. An experimental  $\rho$  value of  $-0.83 \pm 0.02$  was obtained. The correlation coefficient was  $-0.997$ . No systematic deviation from linearity was observed. This is graphically represented in Figure 1.

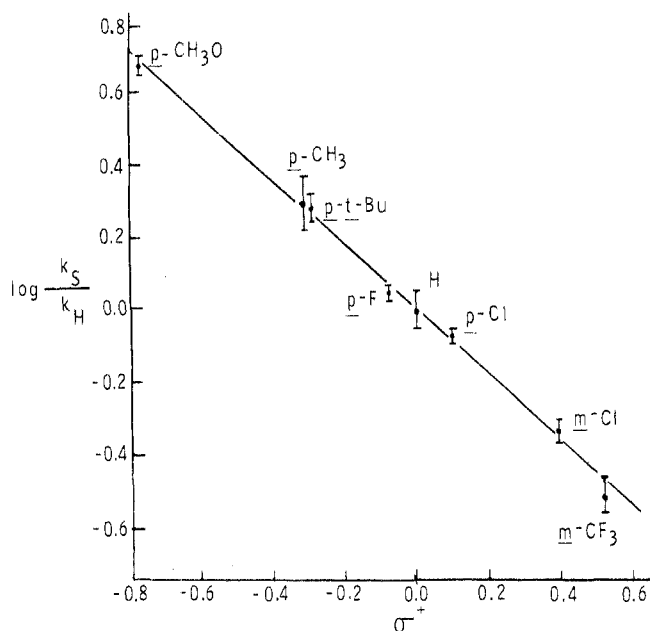


Figure 1. Logarithms of the relative rates of reaction of  $\beta$ -(substituted phenyl)-ethyl bromides vs.  $\sigma^+$  parameters.

It is apparent that no evidence for participation is found. Unlike aliphatic radicals, radicals such as 1, even with electron-withdrawing groups present, must achieve sufficient stabilization so as to make bromine participation unnecessary under the reaction conditions employed.

## Experimental Section

**Materials.** With the exception of the parent compound, all the  $\beta$ -(substituted phenyl)-ethyl bromides were prepared from the corresponding alcohols by reaction with phosphorous tribromide. All compounds showed physical properties (boiling points and indices of refraction) in agreement with literature values. The NMR spectra of all compounds were as expected and GLC indicated purities in excess of 98%. Bromotrichloromethane, mesitylene, and bromobenzene were purified by distillation prior to use. Purity was again in excess of 98%.

**Kinetic Determinations.** Solutions of substituted phenethyl bromides, mesitylene or (2-bromoethyl)benzene, bromobenzene, and bromotrichloromethane were prepared in the approximate molar ratios of 1:1:0.5:10. Approximately 0.75 ml of the solution was placed in each of several ampules.

The ampules were cooled to dry ice-isopropyl alcohol temperature until the solutions solidified. The ampules were evacuated at 2.0–3.0 mm and flushed several times with nitrogen with intermediate thawing. The ampules were sealed under vacuum and one was reserved for the analysis of the unreacted starting materials. The remainder were placed horizontally just below the surface of mineral oil constant-temperature bath maintained at  $70.0 \pm 0.5^\circ\text{C}$ . The solutions were irradiated with uv light provided by a Sylvania 275-W sun lamp placed 20 cm above the surface of the oil. Reaction times varied from 1 to 3 hr, by which time up to 85% of substituted phenethyl bromides and mesitylene had reacted. The ampules were then cooled and opened. Analysis of the mixtures, both before and after the reaction, was via GLC on a 5% SE-30 on Chromosorb W column.

Conversion of raw data to relative rates involved the use of standard formulas.<sup>11</sup>

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## Reactions Which Relate to the Environmental Mobility of Arsenic and Antimony. I. Quaternization of Trimethylarsine and Trimethylstibine

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Several papers have appeared which discuss the environmental distribution of arsenic<sup>2-8</sup> and antimony.<sup>6-9</sup> However, little attention has been given to the chemical changes which facilitate the movement of these elements from one subsystem of the biosphere to another. One of the more important processes in arsenic mobilization has been shown to be the reduction and methylation of inorganic arsenic compounds by microorganisms to produce trimethyl- and dimethylarsine.<sup>10-12</sup> At this time, it has not been demonstrated that methylstibines are metabolites of microorga-

Table I  
Quaternization of Tertiary Stibines and Arsines in Methanol and Acetonitrile at  $29.5 \pm 1^\circ\text{C}$   
 $\text{R}_3\text{E} + \text{R}'\text{X} \rightarrow (\text{R}_3\text{ER}')^+\text{X}^-$

Solvent <sup>a</sup>	$\text{R}_3\text{E}^b$	$\text{R}'\text{X}^{a,b}$	Rate constant, $M^{-1}\text{sec}^{-1c}$	$N^d$	% <sup>e</sup>	Days <sup>f</sup>
Methanol	$(\text{CH}_3)_3\text{Sb}^k$	$\text{CH}_3\text{I}^j, m$	$(3.85 \pm 0.06) \times 10^{-4}$	17	99+	$1/3$
Methanol	$(\text{CH}_3)_3\text{Sb}^k$	$\text{CD}_3\text{I}^j, n$	$(3.80 \pm 0.18) \times 10^{-4}$	13	90	$1/3$
Methanol	$(\text{CH}_3)_3\text{Sb}^k$	$\text{CH}_3\text{CH}_2\text{I}^o$	$(9.36 \pm 0.53) \times 10^{-7}$	6	45	7
Methanol	$(\text{CH}_3)_3\text{Sb}^k$	$\text{CH}_3\text{CH}_2\text{Br}^p$	$(1.98 \pm 0.60) \times 10^{-7}$	6	19	6
Methanol	$(\text{CH}_3)_3\text{Sb}^k$	$\text{CH}_3\text{CH}_2\text{CH}_2\text{I}^q$	$(4.41 \pm 0.76) \times 10^{-7}$	6	50	15
Acetonitrile	$(\text{CH}_3)_3\text{Sb}^k$	$\text{CH}_3\text{I}^j$	$(1.14 \pm 0.06) \times 10^{-3}$	7	99+	$1/2, 4$
Acetonitrile	$(\text{CH}_3)_3\text{Sb}^k$	$\text{CH}_3\text{CH}_2\text{I}^j$	$(4.25 \pm 0.27) \times 10^{-6}$	8	76	4
Acetonitrile	$(\text{CH}_3)_3\text{Sb}^k$	$\text{CH}_3\text{CH}_2\text{Br}$	$(1.44 \pm 0.74) \times 10^{-6}$	3	0-13	0-1
Acetonitrile	$(\text{CH}_3)_3\text{Sb}^k$	$\text{CH}_3\text{CH}_2\text{CH}_2\text{I}$	$(3.18 \pm 2.10) \times 10^{-7}$	4	6-21	1-7
Methanol	$(\text{CH}_3)_3\text{As}^l$	$\text{CH}_3\text{I}^j$	$(2.52 \pm 0.19) \times 10^{-6}$	9	58	4
Methanol	$(\text{CH}_3)_3\text{As}^l$	$\text{CH}_3\text{CH}_2\text{I}$	$(1.35 \pm 0.04) \times 10^{-3}$	6	99	$1/2, 4$
Methanol	$(\text{CH}_3)_3\text{As}^l$	$\text{CH}_3\text{CH}_2\text{CH}_2\text{I}$	$(1.26 \pm 0.09) \times 10^{-5}$	8	93	2
Methanol	$(\text{CH}_3)_3\text{As}^l$	$\text{CH}_3\text{CH}_2\text{CH}_2\text{I}$	$(7.34 \pm 0.39) \times 10^{-6}$	8	88	4
Methanol	Pyridine <sup>g</sup>	$\text{CH}_3\text{CH}_2\text{I}$	$1.94 \times 10^{-6}$			
Acetone	Pyridine <sup>g</sup>	$\text{CH}_3\text{CH}_2\text{I}$	$1.0 \times 10^{-5}$			
Acetone	$\text{C}_6\text{H}_5\text{As}(\text{CH}_2\text{CH}_3)_2^h$	$\text{CH}_3\text{I}$	$1.11 \times 10^{-3}$			

<sup>a</sup> Solvents were reagent grade containing only trace amounts of water (<0.1%). They were manipulated only in the inert atmosphere box. Alkyl halides were reagent grade. <sup>b</sup> Typical initial concentrations of  $\text{R}_3\text{E}$  and  $\text{R}'\text{X}$  were 0.5 and 1.0 M, respectively. <sup>c</sup> Standard error in rate is indicated from statistical least-squares fit of rate data. <sup>d</sup> Number of data points. <sup>e</sup> Percent reaction observed. <sup>f</sup> Days reaction observed. <sup>g</sup> From National Bureau of Standards Circular 510 (1951), Table 652.477; reactions at  $25^\circ\text{C}$ . <sup>h</sup> From ref 18; reaction at  $35^\circ\text{C}$ . <sup>i</sup> Reaction seems to drift at long reaction times. This is possibly the result of experimental difficulties, but similar phenomena observed in ref 18 were attributed to approach to equilibrium. <sup>j</sup> Product  $(\text{R}_3\text{ER}')^+\text{X}^-$  precipitates during latter part of reaction. <sup>k</sup> Registry no., 594-10-5. <sup>l</sup> Registry no., 593-88-4. <sup>m</sup> Registry no., 74-88-4. <sup>n</sup> Registry no., 865-50-9. <sup>o</sup> Registry no., 75-03-6. <sup>p</sup> Registry no., 74-96-4. <sup>q</sup> Registry no., 107-08-4.

nisms acting on inorganic antimony compounds, but the extensive similarity of the chemistry of arsenic and antimony gives reason to believe that antimony can be biologically methylated. Methylarsines and -stibines are subject to a number of reactions such as oxidation, quaternization, and complex formation which could facilitate or inhibit their dispersal in the environment. It has recently been reported that environmentally important concentrations of halocarbons ( $\text{CH}_3\text{I}$ ,  $\text{CH}_3\text{Br}$ , and  $\text{CH}_3\text{Cl}$ ) are produced naturally and accumulate in the oceans and atmosphere.<sup>13</sup> In this paper we report quantitative measurements of the rate of quaternization of trimethylstibine and trimethylarsine by alkyl halides in polar solvents.<sup>14-18</sup> Methanol (a protic solvent) and acetonitrile (an aprotic dipolar solvent) were chosen as reaction media because trimethylstibine and trimethylarsine are not soluble enough in water to allow study by the available techniques. It should be possible to extrapolate the results to water by use of appropriate solvent parameters. A typical kinetic run is shown in Figure 1 and the measured rate constants are summarized in Table I.

Several factors can be noted which indicate that the reaction is a bimolecular nucleophilic displacement of halogen from carbon by antimony or arsenic ( $\text{S}_\text{N}2$ ). The reactions are all first order in alkyl halide and first order in stibine or arsine, i.e., second order overall. The orders of reactivity of the alkyl halides,  $\text{CH}_3\text{I} \gg \text{CH}_3\text{CH}_2\text{I} \approx \text{CH}_3\text{CH}_2\text{CH}_2\text{I}$  and  $\text{CH}_3\text{CH}_2\text{I} > \text{CH}_3\text{CH}_2\text{Br}$ , are typical of  $\text{S}_\text{N}2$  type reactions. Also, as expected for an  $\text{S}_\text{N}2$  reaction, there is no appreciable  $\alpha$ -secondary isotope effect in the reaction of  $\text{CH}_3\text{I}$  or  $\text{CD}_3\text{I}$  with trimethylstibine.<sup>19</sup> It should be noted that when  $\text{CD}_3\text{I}$  was treated with trimethylstibine, no  $\text{CH}_3\text{I}$  was observed in the sample. This result confirms the idea that there is no low-energy intermediate which would allow exchange of methyl groups between methyl iodide and trimethylstibine. The data in Table I, including reactions of pyridine taken from other sources, indicate a qualitative nucleophilic reactivity of trimethylarsine and trimethylstibine in the order  $(\text{CH}_3)_3\text{As} > \text{pyridine} \approx (\text{CH}_3)_3\text{Sb}$ .

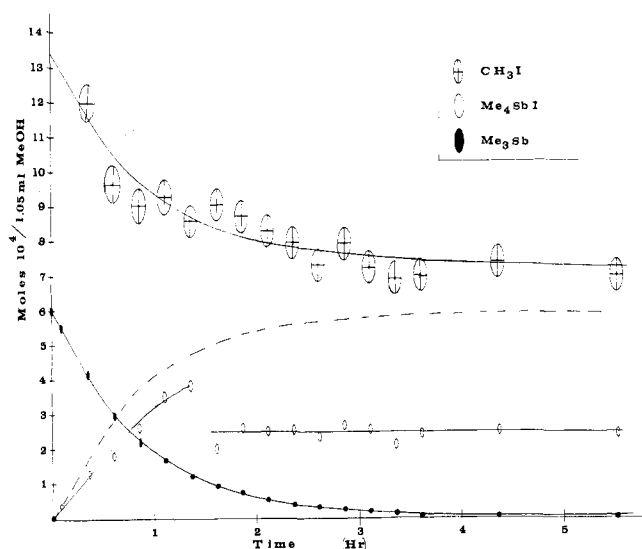


Figure 1. Plot of moles of trimethylstibine (●), methyl iodide (⊕), and tetramethylstibonium iodide (○) contained in 1.05 ml of methanol solution as a function of time. The dashed line represents the concentration of tetramethylstibonium iodide expected if no precipitation occurred. Note that tetramethylstibonium iodide supersaturates before reaching equilibrium concentration (0.24 M). The error in each measurement of trimethylstibine or tetramethylstibonium iodide is approximately 3%. Error bars representing approximately 6% error are used for methyl iodide because its signal is interfered with by a  $^{13}\text{C}$ - $^1\text{H}$  satellite of the methanol solvent.

Concerning the solvent effect on the reaction of trimethylstibine with methyl iodide, note that the reaction is very slow, requiring weeks to reach completion,<sup>14</sup> in diethyl ether. The more polar solvents greatly increase the rate of quaternization. In addition, Kosower<sup>20</sup> has suggested that there are specific interactions between nitrile solvents and halogen leaving groups which account for the enhanced rate of quaternization in acetonitrile relative to methanol

even though the solvents have similar dielectric constants. The reactions of primary environmental interest occur in water. There are a number of solvent parameters ( $Y$ ,  $\Omega$ ,  $Z$ ,  $S$ ,  $E_T$ ,  $\delta$ )<sup>21</sup> which could be used to correlate reaction rates in various solvents. Kosower<sup>22</sup> has used  $Z$  to correlate the rates of SN2 reactions in a series of protic solvents. We can write  $\log k_2 = AZ + C$ , where  $A \approx 0.025$  in protic solvents and  $C$  is a constant which depends only on the nucleophile and substrate. Using our results for trimethylstibine and trimethylarsine reacting with methyl iodide in methanol ( $Z$  value 83.6), the  $C$ 's for the reactions are  $-5.5$  and  $-5.0$ , respectively. The  $Z$  value of water is 94.6. Thus, the rate constant for reaction of  $(\text{CH}_3)_3\text{Sb}$  and  $(\text{CH}_3)_3\text{As}$  with  $\text{CH}_3\text{I}$  in water can be estimated to be  $7 \times 10^{-4}$  and  $3 \times 10^{-3} \text{ M}^{-1} \text{ sec}^{-1}$ , respectively. A similar approach can be applied to the other reactions in Table I. The result is that the reactions are expected to be about twice as fast in water as in methanol.

### Experimental Section

Trimethylantimony dibromide was prepared by the method described by Doak et al.<sup>23</sup> It was recrystallized from acetone. Solutions of trimethylstibine for kinetic studies were prepared in a recirculating inert atmosphere ( $\text{N}_2$ ) box by refluxing  $(\text{CH}_3)_3\text{SbBr}_2$  with a twofold excess of 30-mesh zinc in the solvent of choice (methanol or acetonitrile) for 1 hr, followed by vacuum distillation of the solution. The solutions were standardized by adding an excess of methyl iodide to a weighed sample of each solution and weighing the tetramethylstibonium iodide isolated after removal of solvent.

Trimethylarsine was obtained from a commercial source. Solutions of trimethylarsine for kinetic studies were prepared by weight using conventional vacuum line techniques.

The reactions of trimethylstibine and trimethylarsine with alkyl halides were observed by an NMR technique using a commercial 60-MHz instrument. In an inert atmosphere box, approximately 1 ml of the typically 0.5 M stibine or arsine solution was transferred into a tared thin-wall NMR tube which was then stoppered with a rubber septum. The exact quantity of solution was determined by weight. After allowing the sample to equilibrate thermally in the NMR probe, about 0.15 ml of a mixture of the desired alkyl halide and hexamethyldisilane (prepared and stored under nitrogen; typical mole ratio 10:1) was injected through the septum to initiate the reaction. The exact amount of  $\text{RX}-(\text{CH}_3)_6\text{Si}_2$  solution added was determined by weight at the completion of the reaction. In most kinetic runs, only the signals for the stibine or arsine and the standard,  $(\text{CH}_3)_6\text{Si}_2$ , were observed using 50-Hz sweep width and changing the offset as necessary to put both signals in the scan. The areas of the two signals were measured ( $\pm 3\%$ ) with a planimeter and related to the moles of reactant and standard. Concentrations were calculated by relating the liquid level in the sample tube to the volume of the reacting solution. For the slow reactions which required days to reach a reasonable extent of reaction, the sample tubes were stored in a bath regulated to the probe temperature,  $29.5 \pm 1^\circ\text{C}$ . The concentration vs. time data was fitted by computer to a second-order rate equation by the method of least squares.

In the course of this study the chemical shifts of trimethylstibine ( $\delta$  0.73), trimethylarsine ( $\delta$  0.91), and tetramethylstibonium iodide ( $\delta$  1.58 low concentration,  $\delta$  1.66 saturated) were determined in methanol relative to internal hexamethyldisilane ( $\delta$  0.04).<sup>24</sup> The signal for tetramethylstibonium iodide is quite broad ( $W_{1/2} \approx 4.5$  Hz) owing to unresolved Sb-C-H coupling. The signal for trimethylstibine is not detectably broadened ( $W_{1/2} \approx 0.7$  Hz) because in an asymmetric electric field the antimony quadrupole successfully relaxes the Sb-C-H coupling. In addition, it was noted that the maximum solubility of  $(\text{CH}_3)_4\text{SbI}$  in methanol is 0.24 M and the maximum solubility of  $\text{CH}_3\text{CH}_2\text{Sb}(\text{CH}_3)_3\text{I}$  in acetonitrile is about 0.2 M.

**Acknowledgment.** The assistance of Mr. Richard Thompson in recording the NMR spectra is appreciated.

**Registry No.**—Tetramethylstibonium iodide, 2185-78-6; tetra-(methyl- $d_3$ )stibonium iodide, 56929-85-2; ethyltrimethylstibonium iodide, 56929-86-3; ethyltrimethylstibonium bromide, 56929-87-4; trimethylpropylstibonium iodide, 56929-88-5; tetramethylarsonium iodide, 5814-20-0; ethyltrimethylarsonium iodide, 56929-89-6;

trimethylpropylarsonium iodide, 56929-90-9; trimethylantimony dibromide, 5835-64-3.

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### Applications of Surfactants to Synthetic Organic Chemistry

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Synthetic chemists are frequently faced with the problem of reacting a water-insoluble organic compound with a water-soluble reagent (hydroxide, permanganate, formate, periodate, hypohalite, hydrogen peroxide, hydrobromic acid, hydrazine, etc.)<sup>2</sup>. Several methods are available for countering this difficulty. The organic phase can be stirred rapidly with the aqueous phase; agitation promotes interfacial contact between the two reactants. An alternative procedure utilizes a cosolvent such as dioxane or ethanol in the water. Although a water-cosolvent mixture does not usually dissolve all the organic reactant, the hope is that at least a small portion of the reactant will enter the watery solvent. As the compound is consumed, more of it is supplied from the organic layer. Dipolar aprotic solvents,<sup>3</sup> crown ethers,<sup>4</sup> and phase transfer catalysts<sup>5</sup> are also commonly used in synthesis; they function by dissolving or solubilizing ionic reagents in organic phases. In the present article we assess the value of surfactants in several two-phase reactions. Surfactants disperse organic liquids in water; this could conceivably generate higher yields and shorter reaction times. Surfactants also form micelles which are capable of catalyzing organic reactions.<sup>6</sup> Yet neither "emulsion catalysis" nor micellar catalysis by surfactants has been exploited to any degree in synthetic organic chemistry.<sup>7</sup>