torsionally excited states were unsuccessful.

The existence of small amounts of trimethylisocyanosilane in equilibrium with the cyano isomer has already been established,[5,11] and similar equilibria have been found for the triethyl and triisopropyl compounds.[16] Recently, evidence has also been presented[17] for the existence of approximately 5% of the isocyanide in trimethylcyanogermane. Booth and Frankiss[11] have observed the N═C stretch in trimethylisocyanosilane at 2095 cm^{-1}. Therefore, one would expect that the N═C stretch in dimethylisocyanosilane should give rise to a band at approximately the same frequency. A weak band at 2097 cm^{-1} was observed in the infrared spectrum of gaseous dimethylcyanosilane that had counterparts in the gas- and liquid-phase Raman spectra at 2094 and 2089 cm^{-1}, respectively. However, a band at 712 cm^{-1} was observed in the infrared spectrum of the gas, and a band at 3308 cm^{-1} was observed in the Raman spectrum of the gas. Furthermore, the relative intensity of the gas-phase Raman bands at 2094 and 3308 cm^{-1} was observed to increase with time. There also appeared in the Raman spectrum of the gas a series of equally spaced lines of low intensity extending from the exciting line for about 130 cm^{-1}. Calculations indicated that these lines were assignable to the rotational transitions of HCN. Therefore, the bands at 3308 and 712 cm^{-1} obviously correspond to the C—H stretch and H—C≡N bend in HCN, which must be a decomposition product of dimethylcyanosilane. The bands observed around 2095 cm^{-1} in the infrared and Raman spectra must, therefore, be assigned to the C≡N stretch in HCN, rather than to the N═C stretch in dimethylisocyanosilane. None of the three fundamental frequencies of HCN were observed in the spectra of the solid, which is as expected, since decomposition should be negligible at such low temperatures. Also, no evidence was found for the existence of the isocyanide during the course of the microwave investigation. However, our results cannot, of course, exclude the presence of the isocyano isomer in very low concentration.

We are currently interested in the barriers to internal rotation of methyl groups in organosilanes. Unfortunately, no barrier information was available from the microwave study on dimethylcyanosilane. Barrier calculations[18] were carried out by assuming that the interaction between the two tops is zero. It was observed for the dimethyldihalosilanes[6] that the two torsions are essentially degenerate. This is in contrast to the isopropyl halides[19] where considerable coupling between the two tops was observed. The assumption of zero interaction between the two tops in dimethylcyanosilane is reasonable on the basis of the increased distance between the two tops and is further supported by earlier observations for the trimethylsilanes.[20] Using an F value of 5.42 cm^{-1} calculated from the proposed structure and a torsional frequency of 206 cm^{-1} one calculates a periodic barrier of 2.8 kcal/mol as an upper limit. This value is in qualitative agreement with the barriers to internal rotation of methyl groups in the dimethylhalosilanes[6] which are in the range 2.07–2.56 kcal/mol. It should be pointed out that the calculation of a valid barrier to internal rotation is dependent on the correct assignment of the torsional modes, and since no data were available for the deuterium-substituted molecule, this assignment is to be considered tentative.

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Registry No. $(CH_3)_2SiHCl$, 1066-35-9; AgCN, 506-64-9; $(CH_3)_2SiHCN$, 18192-18-2.

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Vibrational Spectra and Structure of Biphosphine and Biphosphine-d_4

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Infrared and Raman spectra have been obtained for biphosphine and biphosphine-d_4 in both solid and vapor phases. From the spectral data it is concluded that only one conformer exists in both solid and fluid phases and that this is the gauche conformer. A normal-coordinate analysis has also been performed, in which the 24 vibrational frequencies of the two isotopes have been fitted by 10 force constants to an overall average error of 1.0%. The PED indicates that there is practically no mixing of the normal modes in the P_2H_4 molecule but the P–P stretch and the PD_2 wagging modes are extensively mixed for the P_2D_4 molecule.

Introduction

In recent years compounds of the general formula X_2Y_4 have been the subject of considerable spectroscopic investigation.[1] For the diboron X_2Y_4 molecules the planar (D_{2h}) and staggered (D_{2d}) forms have been shown to be the most stable conformers, whereas for the group 5A (nitrogen, phosphorus, and arsenic) compounds the trans (C_{2h}) and gauche (C_2) forms are the only observed conformations. No cis (C_{2v}) conformers have been observed, apparently due to molecular strains induced by the eclipsed positions of the electron pairs and substituents.

While the structures of several substituted biphosphine compounds have been well determined by spectroscopic techniques, the structure of the simplest diphosphorus analog, biphosphine (P_2H_4), itself has been the subject of some controversy. In 1956 Nixon[2] obtained the infrared spectra of P_2H_4 and P_2D_4 in both the solid and the vapor phases. From his data he tentatively concluded that biphosphine existed in the gauche from in the vapor phase. Nixon[2] also concluded from his X-ray study that the crystal structure of biphosphine was either $C_2{}^1$ or $C_s{}^1$ with two molecules per primitive cell. Baudler and Schmidt[3,4] obtained the Raman spectra of liquid P_2H_4 and P_2D_4 and concluded that their spectra could be interpreted in terms of either a C_2 or a C_{2h} structure. Liquid biphosphine is particularly photosensitive and these authors observed a number of peaks due to decomposition products. Since the infrared and Raman spectra were obtained for different phases, Frankiss[5] studied both the infrared and Raman spectra of *one* phase of biphosphine—the solid phase. He concluded that biphosphine is in the trans conformation in the solid. More recently both electron diffractions and microwave studies have been performed on biphosphine in the vapor phase. The electron diffraction work[6] claimed a gauche structure with a dihedral angle of 81° from the cis conformation. However, the authors also stated that their results were relatively insensitive to this angle and they could not rule out the possibility of another conformation, a mixture of conformations, or even free rotation about the phosphorus–phosphorus bond. The microwave study[7] positively established the presence of a gauche conformer in the vapor phase and that the dihedral angle was 74° from the cis structure. However, the microwave study could not rule out the presence of a trans conformer due to its zero permanent dipole.

Thus, in view of these inconsistencies and uncertainties, we deemed it interesting and useful to investigate the infrared and Raman spectra of biphosphine and its deuterated analog in order to determine whether or not more than one form is present in the fluid phases and also to ascertain which structure is preferred in the solid state.

Experimental Section

Biphosphine was prepared by the neutral hydrolysis of calcium phosphide (City Chemical Corp. New York, N.Y.) in a modification of the method of Evers and Street.[8] Instead of attaching the hydrolysis apparatus to a vacuum system, the preparation was done in a flow of nitrogen gas in a manner identical with that used in the preparation of phosphine.[9] Upon conclusion of the reaction, the liquid nitrogen trap in the hydrolysis apparatus was attached to a mercury-free, high-vacuum system equipped with greaseless stopcocks. From this point on, all experiment work was performed in the absence of as much light as was feasible, since biphosphine is light sensitive. The phosphine–biphosphine mixture was transferred to the vacuum and passed (without pumping) through a trap held at −160°C (isopentane slush) into a trap at −196°C. This served to remove all traces of PH_3. The material in the −160°C trap was then subjected to a low-temperature fractionation on a vacuum distillation column.[10] This served to remove any H_2O which had been carried through a −30°C bath in the hydrolysis apparatus. Biphosphine-d_4 was prepared by using D_2O in the hydrolysis step.

Infrared spectra were recorded on a Perkin-Elmer Model 621 spectrophotometer whose housing was purged with dry air. Calibration was performed using the tabulated frequencies of Jones and Nadeau.[11] Gas-phase infrared spectra were obtained in a 20-cm cell equipped with CsI windows and solid-phase data were obtained in a low-temperature cell of conventional design. Observed frequencies are expected to be accurate to ± 2 cm^{-1}.

Raman spectra were recorded with a Cary Model 82 spectrophotometer equipped with a CRL 53G argon ion laser. All spectra were obtained using 5145-Å radiation. Spectra of the solids were obtained in a Raman cold cell and gas-phase samples were kept in a standard Raman gas cell while the laser beam was multipassed through the gas. All Raman frequency shifts are believed to be accurate to ± 2 cm^{-1}.

Results and Discussion

Figures 1 and 2 show the Raman spectra of gaseous and polycrystalline biphosphine and its deuterated analog while

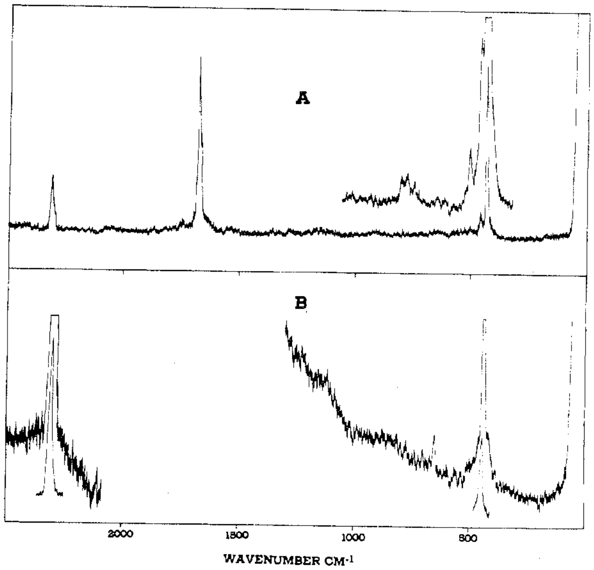

Figure 1. Gas-phase Raman spectra of biphosphine: (A) P_2D_4; (B) P_2H_4.

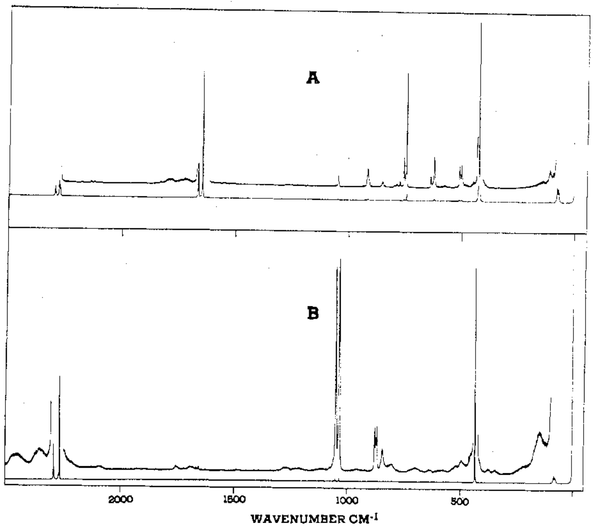

Figure 2. Raman spectra of polycrystalline biphosphine: (A) P_2D_4; (B) P_2H_4.

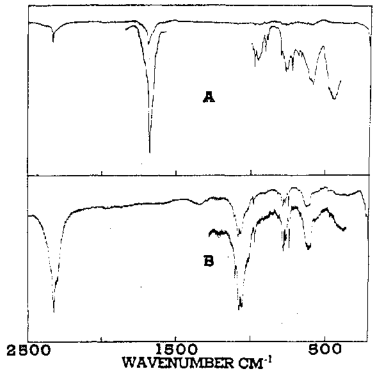

Figure 3. Infrared spectra of gaseous biphosphine: (A) P_2D_4; (B) P_2H_4.

Figures 3 and 4 depict the corresponding infrared spectra of the gaseous and polycrystalline samples. The observed frequencies for P_2H_4 are listed in Table I while those for P_2D_4 are compiled in Table II.

Several important features are readily apparent in the spectral data. For instance, in the Raman spectrum of P_2H_4

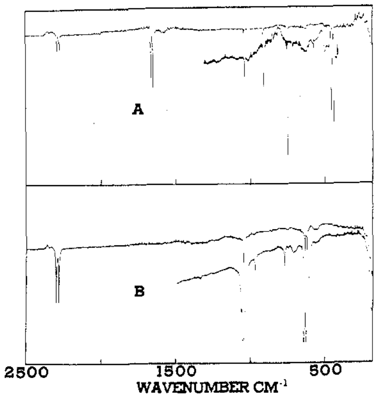

Figure 4. Infrared spectra of polycrystalline biphosphine: (A) P_2D_4; (B) P_2H_4.

Table I. Infrared and Raman Frequencies (cm^{-1}) of Biphosphine[a]

Infrared		Raman		
Gas	Solid	Gas	Solid	Assignment
	2299			ν_8
2312			2295	ν_1
	2281	2320		ν_9
			2268	ν_2
1121 Q				PH_3 impurity
1090 R	1065 sh			
1081 Q, A type	1052		1054	ν_{10}
1072 P	1047 sh			
			1037	ν_3
991 Q				
998 Q				PH_3 impurity
			882	ν_{11}
			875	$2\nu_6$
			872	ν_4
801			847	$\nu_5 + \nu_7$
790 Q				
786 Q				
	779			ν_5 + lattice?
757 Q, C type				
~635 b	643	~650	(638)	ν_{12}
	628			ν_5
		438	436	ν_6
			217	ν_7
			155	Lattice
			86	Lattice
			81	Lattice

[a] Abbreviations used: b, broad; sh, shoulder.

Table II. Infrared and Raman Frequencies (cm^{-1}) of Biphosphine-d_4[a]

Infrared		Raman		
Gas	Solid	Gas	Solid	Assignment
	1687 sh			
	1681			ν_8
1688 Q		1690	1670	ν_1
	1657	1686 sh		ν_9
			1650	ν_2
978 Q				PD_2H impurity?
809				PD_3 impurity
~770 b	763		760	ν_{10}
	759 sh	~789		
			750	ν_3
734				PD_3 impurity
~630 b			643	ν_{12}
			627	ν_4
	512		515	ν_6 + lattice
	504		505	ν_6 + lattice
~465 b	468			ν_5
	449			ν_{12}
			428	ν_6
			153	ν_7
			114	Lattice
			83	Lattice
			77	Lattice

[a] Abbreviations used: see Table I.

Table III. Assignment of Low-Frequency Vibrations in Biphosphine and Biphosphine-d_4

Freq, cm^{-1}			
P_2H_4	P_2D_4	Shift factor[a]	Assignment
217	153	1.42	PH_2 torsion
155	114	1.36	Libration
86	83	1.04	Translation
81	77	1.05	Translation

[a] Theoretical shift factors: translations, $(M_D/M_H)^{1/2} = 1.03$; librations, $(I_A{}^D/I_A{}^H)^{1/2} = 1.39$, $(I_B{}^D/I_B{}^H)^{1/2} = 1.06$, $(I_C{}^D/I_C{}^H)^{1/2} = 1.01$.

vapor only one peak is observed in the P–P stretching region at 438 cm^{-1}. Similarly in gaseous P_2D_4, the Raman spectrum shows one intense peak at 428 cm^{-1} with a weak peak at 459 cm^{-1} which may be assigned as the PD_2 wagging motion, which is also observed in the gas-phase infrared spectrum. In the similar compound tetramethylbiphosphine, $(CH_3)_2P$-$P(CH_3)_2$, two peaks are observed for the P–P stretching vibration in the fluid phases.[12,13] In vapor phase $P_2(CH_3)_4$ the two Raman peaks are separated by 31 cm^{-1}. These two peaks have been confidently assigned to the P–P stretch in both the gauche and trans conformations. Since only *one* P–P stretch is observed for both P_2H_4 and P_2D_4, only one conformer must be present, and this, therefore, must be the gauche conformation observed in the microwave spectrum.[7]

Another interesting aspect of the spectra is seen in the infrared and Raman spectra of the solid phase. A number of peaks exist as doublets spaced between 10 and 27 cm^{-1} with an average splitting of approximately 18 cm^{-1}. In his study of P_2H_4, Frankiss[5] assigned these doublets as arising from factor group splitting. We feel, however, that these peaks are due instead to distinct fundamental molecular vibrations of P_2H_4 and P_2D_4 in the gauche from in the solid state. There are several reasons for this conclusion. First of all, factor group splittings in the similar molecules methylphosphine[14] and ethylphosphine[15] are not greater than 4 or 5 cm^{-1} at the most, and we suspect that, in biphosphine, factor group splitting, if observed, should be of this magnitude. Second, a reinvestigation of the spectra of hydrazine and hydrazine-d_4[16] indicates that hydrazine is in the gauche form in the solid phase and thus it seems likely that biphosphine would also prefer the gauche configuration in the solid. Therefore, we feel confident in assigning these doublets to fundamental vibrations of the gauche conformation of biphosphine. Further reasons for assigning the spectra of the solid phase to a C_2 structure are as follows. The fact that both in-phase and out-of-phase motions are seen simultaneously in the Raman or infrared spectra rules out the centrosymmetric C_{2h} structure. Several coincidences are observed between infrared and Raman spectra, and we feel that the lack of further coincidences is due to the inherent weakness of several of the vibrations in either the infrared or Raman spectra. An example of this is seen in the Raman spectrum of P_2H_4. At 638 cm^{-1} a very weak unresolved band is observed which could coincide with either the 643- or 628-cm^{-1} peak observed in the infrared spectrum. One of the more compelling arguments for the gauche structure is seen in Table III. The weak, broad peak at 217 cm^{-1} in the Raman spectrum of solid P_2H_4 is seen to shift to 153 cm^{-1} upon deuteration. We feel that these frequencies are too high for lattice modes, which are assigned in Table III, and the shift factor of 1.42 leads us to assign these peaks to the torsional vibration about the phosphorus–phosphorus bond. Under C_{2h} symmetry the torsional vibration would be forbidden from the Raman spectrum while under C_2 symmetry it is a totally symmetric mode.

Table IV. Observed and Calculated Raman and Infrared Frequencies (cm^{-1}) of Solid Biphosphine

Raman	Infrared	Calcd	Assignment and PED
			P_2H_4
	2299	2308	ν_8 100% S_9
2295		2308	ν_1 100% S_3
	2281	2283	ν_9 100% S_8
2268		2283	ν_2 100% S_2
1054	1052	1056	ν_{10} 93% S_{12}, 6% S_{11}
1037		1040	ν_3 97% S_6, 3% S_5
882		884	ν_{11} 94% S_{11}, 6% S_{12}
872		898	ν_4 96% S_5, 3% S_6
(638)	643	629	ν_{12} 100% S_{10}
	628	613	ν_5 100% S_4
436		438	ν_6 100% S_1
217		216	ν_7 100% S_7
			P_2D_4
	1681	1659	ν_8 100% S_9
1670		1659	ν_1 100% S_3
	1657	1641	ν_9 100% S_8
1650		1641	ν_2 100% S_2
760	762	757	ν_{10} 94% S_{12}, 6% S_{11}
750		746	ν_3 96% S_6, 3% S_5
643		638	ν_{11} 93% S_{11}, 6% S_{12}
627		645	ν_4 89% S_5, 8% S_1, 3% S_6
	468	462	ν_{12} 99% S_{10}
	449	443	ν_5 78% S_4, 22% S_1
428		416	ν_6 70% S_1, 25% S_4, 5% S_5
153		155	ν_7 100% S_7

Finally the assignment of the gauche form in the solid phase follows from the vapor-phase data. For instance the P–P stretch in gaseous P_2H_4 and P_2D_4 falls at 438 and 428 cm^{-1}, respectively. In the sold phase these frequencies shift to 436 and 427 cm^{-1}. It has been shown[1] that fairly large frequency shifts are observed upon solidification as a conformational charge occurs. Since only a very small shift is observed, we conclude that the gauche form observed in the vapor state is also the conformation present in the solid state.

Assignment

On the basis of the well-established assignment of the fundamentals of hydrazine,[17] the P–H bending modes are assigned in the order PH_2 scissors > PH_2 twist > PH_2 wag, although for the C_2 (gauche) structure, where the P–H distances and angles are different for the two hydrogens attached to the same phosphorus, these terms would have little physical meaning. Since the two PH_2 groups are so widely separated (2.219 Å) we feel that they act almost independently of the molecular symmetry and the in-phase and out-of-phase bending motions will differ little in frequency. The assignments of the torsion and the P–P stretch have been discussed in earlier sections. Thus, the assignment of P_2H_4 and P_2D_4 fundamentals on the basis of C_2 symmetry is given in Table IV.

Table V. Internal and Symmetry Coordinates for Biphosphine

Internal coordinate	Description
r	P–P bond length
R_1, R_2	"Inner" P–H bond lengths
Q_1, Q_2	"Outer" P–H bond lengths
α_1, α_2	"Inner P–P–H bond angles
β_1, β_2	"Outer P–P–H bond angles
γ_1, γ_2	H–P–H bond angles
τ	Dihedral torsional angle

Symmetry Coordinates

A Symmetry Block

$S_1 = r$ $S_5 = 1/\sqrt{2}(\beta_1 + \beta_2)$
$S_2 = 1/\sqrt{2}(R_1 + R_2)$ $S_6 = 1/\sqrt{2}(\gamma_1 + \gamma_2)$
$S_3 = 1/\sqrt{2}(Q_1 + Q_2)$ $S_7 = \tau$
$S_4 = 1/\sqrt{2}(\alpha_1 + \alpha_2)$

B Symmetry Block

$S_8 = 1/\sqrt{2}(R_1 - R_2)$ $S_{11} = 1/\sqrt{2}(\beta_1 - \beta_2)$
$S_9 = 1/\sqrt{2}(Q_1 - Q_2)$ $S_{12} = 1/\sqrt{2}(\gamma_1 - \gamma_2)$
$S_{10} = 1/\sqrt{2}(\alpha_1 - \alpha_2)$

Table VI. Force Constants and Their Descriptions for Biphosphine

Force const	Value,[a] mdyn/Å	Description
K_r	1.898 ± 0.09	P–P str
K_R	2.996 ± 0.03	P–H str
K_Q	3.062 ± 0.03	P–H str
H_α	0.441 ± 0.01	P–P–H angle bend
H_β	0.865 ± 0.01	P–P–H angle bend
H_γ	0.662 ± 0.01	H–P–H angle bend
H_τ	0.0132 ± 0.001	Torsion
$F_{\beta\beta}$	−0.06 ± 0.01	(P–P–H)–(P–P–H) interaction
$F_{\gamma\gamma}$	−0.007 ± 0.007	(H–P–H)–(H–P–H) interaction
$F_{r\alpha}$	0.12 ± 0.03	(P–P)–(P–P–H) interaction

[a] Bending coordinates weighted by 1 Å.

Normal-Coordinate Analysis

To our knowledge no previous normal-coordinate calculations have been reported for biphosphine. The structure of biphosphine used in the normal-coordinate analysis was that determined by the microwave study.[7] Internal and symmetry coordinates used in the normal-coordinate procedure are defined in Table V. Schachtschneider's perturbation program[18] was used to adjust the force constants for the best fit to the observed frequencies. The final force field comprised of ten force constants is listed in Table VI. From these force constants, the calculated frequencies and potential energy distribution listed in Table IV were obtained. Since the dispersions for all the force constants were small, we feel confident in our assignment and choice of force constants. The frequency fit was to within an average error of 8 cm^{-1} for P_2H_4 and 10 cm^{-1} for P_2D_4, resulting in an overall fit for both molecules of 9 cm^{-1} or 1.0%. In addition to the 10 force constants listed in Table VI, interaction force constants F_{RQ}, F_{RR}, F_{QQ}, $F_{\alpha\alpha}$, $F_{\alpha\beta}$, $F_{\alpha\gamma}$, and $F_{\beta\gamma}$ were also considered. However, their absolute values were all less than 0.01 mdyn/Å and the frequency fit was not improved appreciably by their inclusion. Thus, these force constants were not retained in the final force field.

It is readily apparent that all the motions are reasonably pure with the exception of the PD_2 wag of A symmetry and the P–P stretch in P_2D_4. We feel that these vibrations mix only because of the nearness in frequency of these two totally symmetric modes. No such mixing is observed in P_2H_4. If force constant $F_{\gamma\gamma}$ is left out of the force field, the fit worsens to 1.1% and the deformational modes are no longer ordered. Thus, we feel that, though small, $F_{\gamma\gamma}$ is real and significant. The two P–H stretching force constants consistently converged to the values shown in Table VI. However, it is not clear whether this difference is significant. The two P–H bond lengths were found to be different by the microwave study[7] and, therefore, the force constants should also be different. The microwave study[7] also found the "inner" and "outer" P–P–H bond angles to be different. This is manifested in the two P–P–H force constants, one of which (the "outer") is almost twice the other. Since the two "inner" protons are considerably closer to each other than the "outer" protons, we feel that they might repel each other in some fashion, so as to lower the "inner" P–P–H angle bending force constant.

Conclusions

The spectroscopic evidence, now, all points to the fact that biphosphine exists in the gauche conformation in all phases. This can be rationalized on the basis of the "gauche effect".[19] Using the "gauche effect" one would predict the stable structure to be that which has the maximum number of gauche interactions between adjacent electron pairs and polar bonds. Wolfe[19] has further pointed out that when both electron pairs and polar bonds are present, the observed structure depends not only on the number of gauche interactions but also upon the distance between the interacting groups. With the long (2.219 Å) phosphorus–phosphorus bond we feel that the hydrogen atoms are too far apart to exert any influence on the molecular structure and it is the dominating effect of the lone electron pairs that determines the structure of biphosphine. In the solid phase, at least, these results are contrary to the structures found for other P_2Y_4 molecules but are, however, consistent with the X-ray crystal study performed by Nixon.[2]

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Registry No. P_2H_4, 13445-50-6; P_2D_4, 39863-38-2.

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Esterification of Chrysotile–Asbestos by Allyl Alcohol

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In this paper, a method is developed for grafting organic allylic ester groups onto the surface of hydrolyzed or chlorinated chrysotile. It is shown that the degree of esterification increases gradually in relation to the degree of hydrolysis. It is possible to obtain in this way a sheet-like organomineral polymer.

Introduction

Phyllosilicates like chrysotile, which possess a basic Mg(OH) surface, are known to be relatively inert. In particular, they do not respond to the action of silane coupling agents which are so effective with acidic silanols.[1] However, the octahedral layer of phyllosilicate can be eliminated by acid leaching. This treatment, which preserves the crystalline structure of the siloxanic planes, generates on the surface a very reactive acidic layer.

When this hydrolysis is carried out in the presence of halosilane or disiloxane, a condensation reaction takes place and a kind of sheetlike organomineral polymer is produced.[2] The basis for the approach was provided by Lentz,[3] who applied this type of synthesis to monomeric minerals like olivine, sodalite, or natrolite.

In this article a procedure to synthesize another polymeric organomineral material is described. This was obtained by allowing hydrolyzed or chlorinated chrysotile to react with allyl alcohol.

Experimental Section

General Information. The chrysotile–asbestos used in this work is grade 4 fibers from the western part of Canada. Their physical properties and their composition are reported in Table I.

Hydrolysis. The fibers were hydrolyzed in a 1:1 solution of concentrated hydrochloric acid (12 *N*) in 2-propanol, as described in a previous publication.[4] The hydrolyzed fibers were carefully washed with 2-propanol dispersed in benzene, and freeze-dried under vacuum. After elimination of the benzene, the widely open fibers were dried under vacuum at 60° for 24 hr. The degree of hydrolysis was expressed as the fraction (%) of the original magnesium which was eliminated.

Esterification. The reaction of esterification was carried out in a 500-ml vessel. Five grams of hydrolyzed chrysotile was refluxed for 4 hr under mild agitation with a mixture of 100 ml of pyridine and 100 ml of allyl alcohol (reagent grade). After cooling, the esterified fibers were separated from the solution by filtration. The esterified phyllosilicates were then extracted with ethyl ether in a Soxhlet apparatus for 4 hr to remove traces of unreacted reagents. The product was finally dried for 24 hr at room temperature under a pressure of 10^{-2} Torr and for 48 hr at 60° under 10^{-1} Torr.

Table I. Physicochemical Characteristics of the Fibers

A. Virgin chrysotile: Chemical analysis		A. Virgin chrysotile: Specific surface	B. Hydrolyzed chrysotile: Hydrolysis deg, %	B. Hydrolyzed chrysotile: Specific surface[5]
SiO_2	41.08%	BET, N_2[5] 14.38 m^2/g	20	65
Al_2O_3	1.01%		38	125
Fe_2O_3	2.63%		49	200
MgO + CaO	41.24%		62	285
Heat loss	14.53%		70	360
	100.49%		78	425

Table II. Esterification of Chrysotile–Asbestos

Degree of hydrolysis, %	Hydrolyzed fibers: % C	Hydrolyzed fibers: mequiv of OR/100 g[a]	Hydrolyzed fibers: No. of OR/100 nm^2 [b]	Chlorinated fibers: % C	Chlorinated fibers: mequiv of OR/100 g[a]	Chlorinated fibers: No. of OR/100 nm^2 [b]
20	1.3	37	335	1.9	54	515
38	2.6	75	362	2.9	85	402
49	3.1	91	276	4.9	148	443
62	4.9	148	312	6.9	216	458
70	5.4	164	274	7.4	233	390
78	7.6	241	342	9.2	300	425

[a] Milliequivalents of ester groups/100 g of inorganic substrate.
[b] Number of ester groups/100 nm^2.

Chlorination. In a second series of experiments, we submitted the hydrolyzed fiber to chlorination with thionyl chloride before carrying