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The Synthesis and Structure of Triphenylsiloxycyclotriphosphazenes

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ABSTRACT

The triphenylsiloxy-substituted cyclotriphosphazenes, $N_3P_3Cl_5OSiPh_3$, gem-$N_3P_3Cl_4(OSiPh_3)_2$, $N_3P_3(OSiPh_3)_6$, and $N_3P_3(OPh)_5OSiPh_3$, have been prepared. The synthesis of gem-$N_3P_3Cl_4(OSiPh_3)_2$ involves the reaction of $(NPCl_2)_3$ with Ph_3SiONa to form the intermediates gem-$N_3P_3Cl_4(OSiPh_3)_2(ONa)$ and gem-$N_3P_3Cl_4(ONa)_2$, which yield gem-$N_3P_3Cl_4(OSiPh_3)_2$ when treated with Ph_3SiCl. The compounds $N_3P_3Cl_5OSiPh_3$ and $N_3P_3(OSiPh_3)_6$ are formed by the condensation reactions of $N_3P_3Cl_5OBu^n$ and $N_3P_3(OBu^n)_6$, respectively, with Ph_3SiCl. The compound $N_3P_3(OPh)_5OSiPh_3$ is synthesized by the reaction between $N_3P_3(OPh)_5Cl$ and Et_3SiONa to first give the intermediate $N_3P_3(OPh)_5ONa$, which yields $N_3P_3(OPh)_5OSiPh_3$ when reacted with Ph_3SiCl. The structural characterization and properties of these compounds are discussed. The crystal and molecular structure of gem-$N_3P_3Cl_4(OSiPh_3)_2$ has been investigated by single-crystal X-ray diffraction techniques. The crystals are monoclinic with the space group $P2_1/c$ with $a = 16.850(8)$, $b = 12.829(4)$, $c = 18.505(15)$ Å, and $\beta = 101.00(6)°$ with $V = 3927$ Å^3 and $Z = 4$.

INTRODUCTION

In recent years we have explored a variety of methods for the linkage of organosilicon units to small-molecule phosphazene rings and to phosphazene high polymers [1–18]. One of the objectives of this effort has been the synthesis of high polymers that are hybrids of polyphosphazenes and polysiloxanes (silicones) and that combine the special properties of both systems.

The linkage of organosilicon units to small-molecule cyclophosphazenes has two purposes—first, the preparation of "monomers" that can be polymerized by ring-opening methods to macromolecules and, second, the exploration of new reactions that might later be carried out on phosphazene high polymers [19]. Thus, in this second sense, the small-molecule reactions are model processes for the more challenging macromolecular reactions [20].

Virtually all the reactions of this type that we have explored have involved the connection of the organosilicon units to the phosphazene skeleton through P–C–Si, P–N–Si, or P–O–C–Si bonds, rather than through P–O–Si linkages. A considerable body of earlier work [21–24] had provided convincing evidence that P–O–Si bonds are sensitive to cleavage by moisture and other mild reagents and that only rarely do they survive the synthesis processes used for their assembly. For example, Zhivukin and coworkers [24] in 1962 reported that $NaOSiEt_3$ and $NaOSiPh_3$ react with chlorophosphazenes *not* to give the expected species such as $[NP(OSiR_3)_2]_3$, but instead phosphazenes with $P–O^-Na^+$ side units and organosiloxanes. This process closes off an important route to phosphazene-organosilicon hybrids, since the reaction of chlorophosphazenes with alkoxides or aryloxides to give $[NP(OR)_2]_{3,4,\text{ or } n}$ species is probably the most important synthesis pathway in the whole field of phosphazene chemistry [19,25–27]. Other routes to phosphazenes with P–O–Si side

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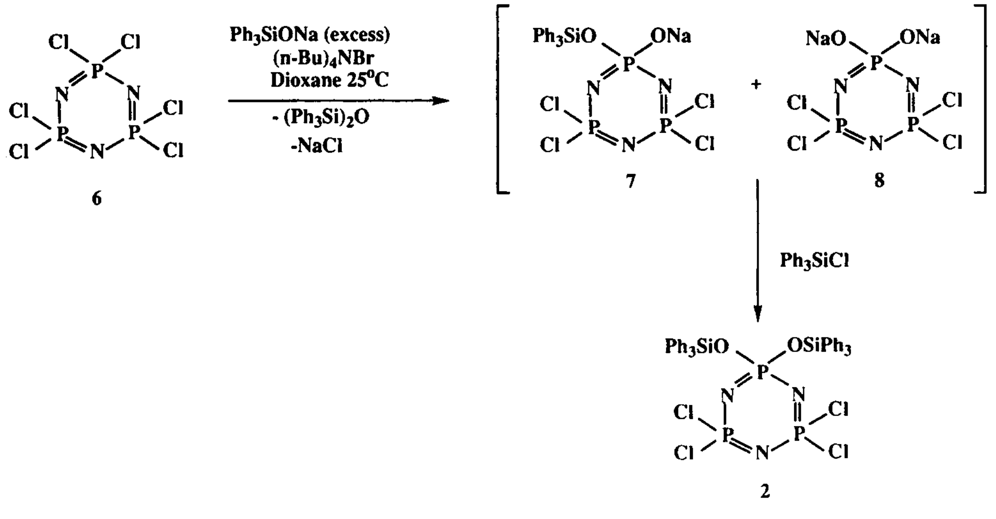

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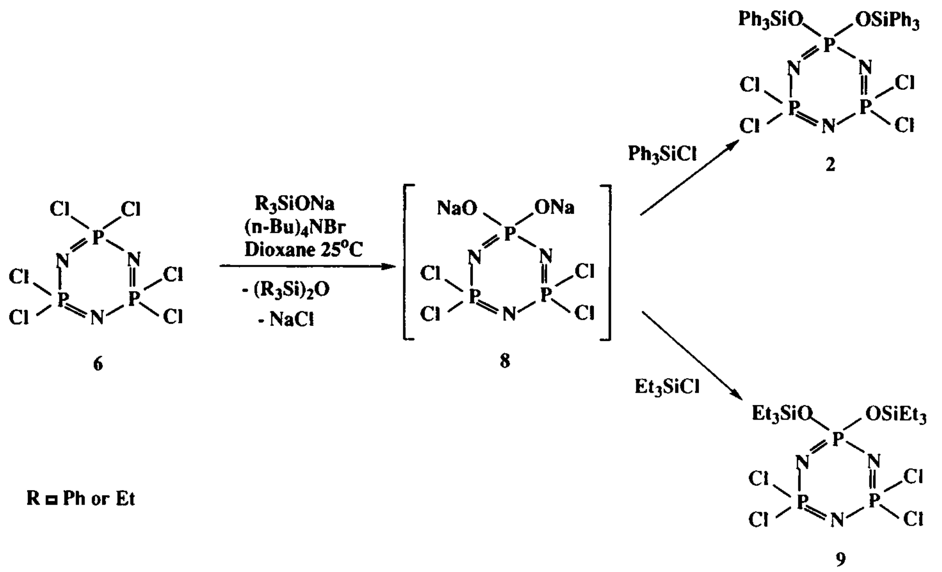


linkages have been developed. For instance, cyclotriphosphazenes with $P{-}O^-Na^+$ groups react with chlorosiloxanes to yield organosilicon-substituted cyclotriphosphazenes [23]. Butoxyphosphazenes react with either chlorosilanes or chlorosiloxanes via a condensation process to yield organosilicon-cyclotriphosphazene species [23,24]. However, these compounds are unstable to hydrolysis in contact with atmospheric moisture. Hydrolytically stable organosiloxy-organophosphazene polymers have been reported by Russian investigators [28], but only when phenylsiloxy and phenoxy groups are attached as side groups to the phosphazene ring.

The hydrolytic stability of phenylsiloxy groups when linked to a phosphazene ring through a P–O–Si linkage has prompted us to devise new methods for the synthesis of arylsiloxy-phosphazenes (1). Two methods were used. The first involves the reaction of a cyclotriphosphazene intermediate bearing $P{-}O^-Na^+$ side groups with Ph_3SiCl to yield the cyclotriphosphazenes *gem*-$N_3P_3Cl_4(OSiPh_3)_2$ (2) and $N_3P_3(OPh)_5OSiPh_3$ (3). The second method makes use of the condensation reaction between butoxycyclotriphosphazenes and Ph_3SiCl at elevated temperatures to yield $N_3P_3Cl_5OSiPh_3$ (4) and $N_3P_3(OSiPh_3)_6$ (5).

The synthesis, characterization, and properties of these triphenylsiloxycyclotriphosphazene species are described, and the X-ray crystal structure of one of these compounds, *gem*-$N_3P_3Cl_4(OSiPh_3)_2$, is presented.

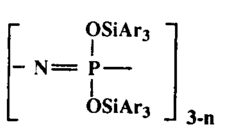
RESULTS AND DISCUSSION

Synthesis of gem-$N_3P_3Cl_4(OSiPh_3)_2$ (2)

The reaction between hexachlorocyclotriphosphazene (6) and an excess of sodium triphenylsiloxide

(4–8 equivalents) in dioxane at 25°C for 24 hours in the presence of $(n\text{-Bu})_4NBr$ yields the intermediate cyclotriphosphazene species *gem*-$N_3P_3Cl_4(OSiPh_3)(ONa)$ (7) and *gem*-$N_3P_3Cl_4(ONa)_2$ (8) (Scheme 1). Intermediate 7 is the main product formed when 4 equivalents of Ph_3SiONa react with $(NPCl_2)_3$. Species 8 predominates when 8 equivalents of Ph_3SiONa react with $(NPCl_2)_3$. Approximately equal quantities of 7 and 8 are formed when 6 equivalents of Ph_3SiONa are used.

The ^{31}P NMR spectra of 7 and 8 were consistent with the proposed structures. The ^{31}P NMR spectrum of 7 is an AB_2 spin pattern (υ_A: −15.8 ppm, υ_B: 15.5 ppm, J_{PNP} = 53.1 Hz) as is the ^{31}P NMR spectrum of 8 (υ_A: −4.1 ppm, υ_B: 13.9 ppm, J_{PNP} = 30.6 Hz). Both 7 and 8 react with Ph_3SiCl to yield *gem*-$N_3P_3Cl_4(OSiPh_3)_2$ (2), which gives rise to a new ^{31}P NMR AB_2 spin pattern (υ_A: −16.6 ppm, υ_B: 20.7 ppm, J_{PNP} = 80.7 Hz). The ^{31}P NMR spectrum of 2 was similar to that of the analogous compound, *gem*-$N_3P_3Cl_4[(OSiMe_2)_xCl)]_2$, formed during the reaction between $(NPCl_2)_3$ and $(OSiMe_2)_3$ at 250°C [23].



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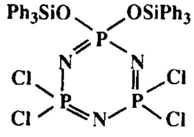

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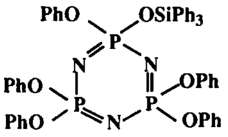

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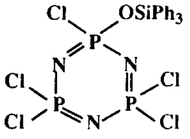

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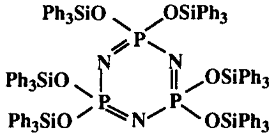
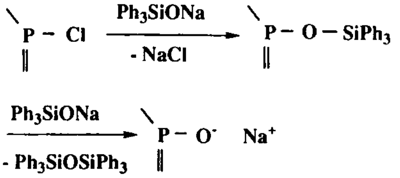


5

The formation of the P–O$^-$Na$^+$ group is believed to first involve the substitution of a P–Cl bond by Ph_3SiONa to generate a P–O–$SiPh_3$ side unit, which reacts further with more Ph_3SiONa to yield the P–O$^-$Na$^+$ functionality and hexaphenyldisiloxane (Equation 1). The liberation and isolation of $Ph_3SiOSiPh_3$ provides evidence for this reaction. When $(NPCl_2)_3$ and Ph_3SiONa react, species 7 is detected in significant quantities before all the $(NPCl_2)_3$ is consumed, as determined by ^{31}P NMR spectroscopy. This indicates that the mechanism of the reaction between $(NPCl_2)_3$ and Ph_3SiONa to yield 7 may be more complex than the simple sequence of reactions shown in Equation 1. Intermediate 7 reacts with Ph_3SiONa to yield 8 and $Ph_3SiOSiPh_3$. The identification of intermediates 7 and 8 is described in the next section.



(1)

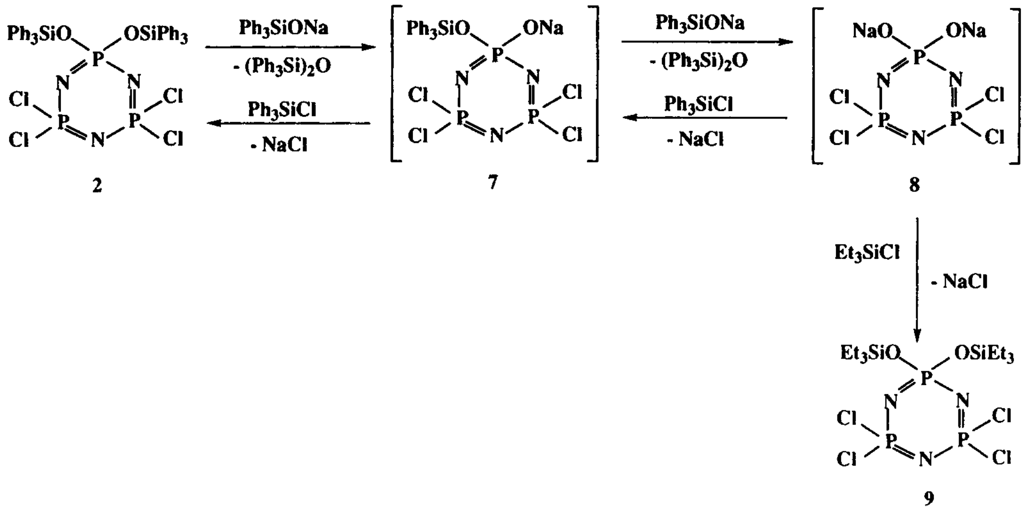
Identification of gem-$N_3P_3Cl_4(OSiPh_3)(ONa)$ (7) and gem-$N_3P_3Cl_4(ONa)_2$ (8)

Two methods were used to identify intermediate species *gem*-$N_3P_3Cl_4(OSiPh_3)(ONa)$ and *gem*-$N_3P_3Cl_4(ONa)_2$. The first method made use of reactions that trapped out one of these intermediates, 8, on treatment with Et_3SiCl and Ph_3SiCl to yield *gem*-$N_3P_3Cl_4(OSiEt_3)_2$ (9) and *gem*-$N_3P_3Cl_4(OSiPh_3)_2$ (2), respectively (Scheme 2). The second involved the generation of intermediate species 7 and 8 by an alternative route—the reaction between *gem*-$N_3P_3Cl_4(OSiPh_3)_2$ and Ph_3SiONa (Scheme 3). Attempts to isolate and characterize 7 and 8 resulted in the decomposition of these species.

The reaction between $(NPCl_2)_3$ and excess Ph_3SiONa or Et_3SiONa in dioxane [with $(n\text{-Bu}_4)NBr$] at 25°C yields 8 (Scheme 2). Species 8 reacts with Ph_3SiCl and Et_3SiCl to give 2 and 9, respectively. It was important to demonstrate that 8, generated from Ph_3SiONa and $(NPCl_2)_3$, reacts with Et_3SiCl to yield *gem*-$N_3P_3Cl_4(OSiEt_3)_2$. This same intermediate, 8, generated from *Et*$_3$SiONa and $(NPCl_2)_3$, reacts with *Ph*$_3$SiCl to yield *gem*-$N_3P_3Cl_4(OSiPh_3)_2$. If 8 were an intermediate that contained an organosilicon side unit, treatment of the intermediate with the chlorosilane species would yield products that contain both phenyl- and ethylsiloxyl side units in the same molecule. Such species were not detected. Species 2 and 9 can only be formed by treatment of an intermediate such as 8 with Ph_3SiCl and Et_3SiCl, respectively.

The compound *gem*-$N_3P_3Cl_4(OSiPh_3)_2$ reacts with 1 equivalent of Ph_3SiONa in dioxane in the presence of $(n\text{-Bu})_4NBr$ at 25°C to yield 7 and

SCHEME 3



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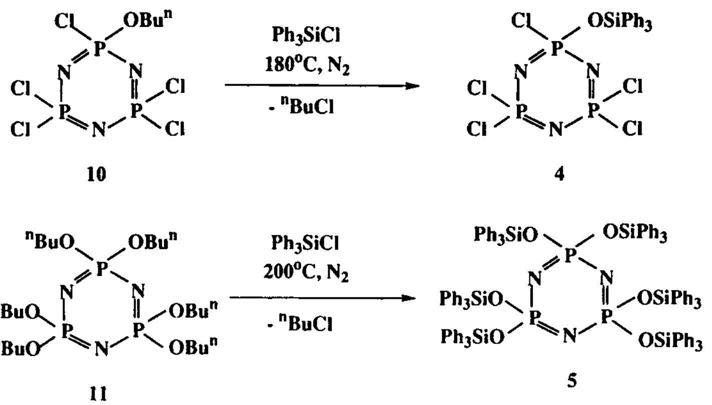


$Ph_3SiOSiPh_3$ (Scheme 3). Species 7 generated from this reaction is identical to 7 formed from $(NPCl_2)_3$ and Ph_3SiONa, as determined by ^{31}P NMR spectroscopy. Further reaction of 7 with excess Ph_3SiONa yields 8. Species 8, formed by the sequence shown in Scheme 3, is identical to 8 formed from $(NPCl_2)_3$ and Ph_3SiONa. Species 8 reacts with Et_3SiCl to yield *gem*-$N_3P_3Cl_4(OSiEt_3)_2$, as described previously. Species 8 also reacts with Ph_3SiCl to reform 7, which yields *gem*-$N_3P_3Cl_4(OSiPh_3)_2$ when treated with more Ph_3SiCl.

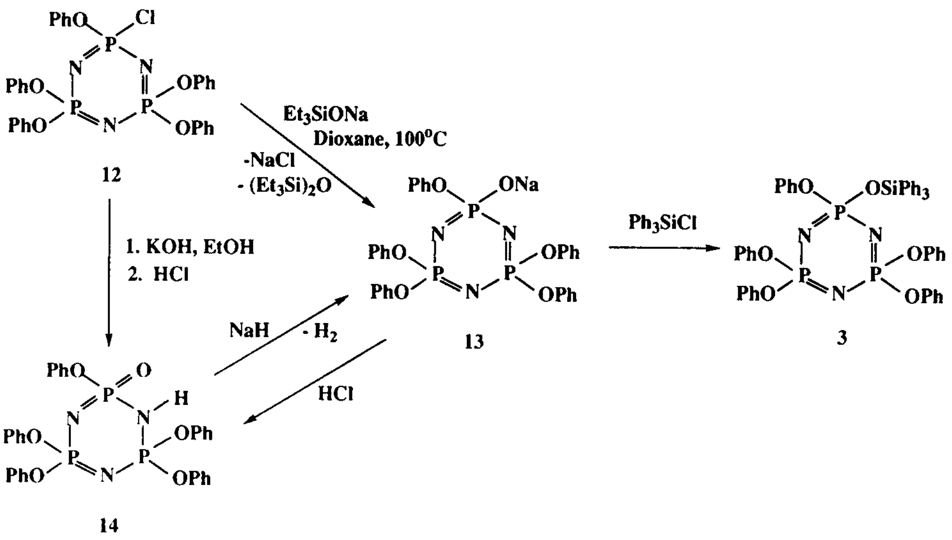
Butoxyphosphazene-Chlorosilane Condensation Reactions: Synthesis of $N_3P_3Cl_5OSiPh_3$ (4) and $N_3P_3(OSiPh_3)_6$ (5)

The triphenylsiloxycyclotriphosphazenes $N_3P_3Cl_5OSiPh_3$ (4) and $N_3P_3(OSiPh_3)_6$ (5) were prepared by the reaction of $N_3P_3Cl_5OBu^n$ (10) and $N_3P_3(OBu^n)_6$ (11), respectively, with Ph_3SiCl at elevated temperatures (Scheme 4). The mechanism of the reaction between butoxyphosphazenes and chlorosilanes is discussed elsewhere [28]. The reactions described here appear to follow the same mechanism.

The reaction between $N_3P_3Cl_5OBu^n$ (10) and an excess of Ph_3SiCl under an atmosphere of dry nitrogen at 180°C yields $N_3P_3Cl_5OSiPh_3$ (4) and eliminates butyl chloride during the condensation reaction. During the course of the reaction, the ^{31}P AB_2 pattern associated with 10 (υ_A: 14.8 ppm, υ_B: 22.4 ppm, J_{PNP} = 63.5 Hz, $CDCl_3$ solvent) disappeared and a new AB_2 spin pattern appeared for 4 (υ_A: 2.9 ppm, υ_B: 22.1 ppm, J_{PNP} = 70.0 Hz). Butyl chloride was identified as a volatile reaction product by infrared spectroscopic comparison with an authentic sample and by a combination of gas chromatography/mass spectrometry.

The synthesis of $N_3P_3(OSiPh_3)_6$ (5) has been attempted previously [28], but the products obtained were cyclotriphosphazene species with only three-to-five butyl groups replaced by triphenylsilyl groups. Hexabutoxycyclotriphosphazene (11) reacts with an excess of Ph_3SiCl at 200°C in an atmosphere of dry nitrogen to yield $N_3P_3(OSiPh_3)_6$ (5). The reaction was monitored by ^{31}P NMR spectroscopy, which showed that the singlet associated with 11 at 17.8 ppm ($CDCl_3$ solvent) disappeared to yield multiple

SCHEME 5



peaks between +7 and −5 ppm, which indicated the formation of cyclotriphosphazene species that contained both butyl and triphenylsilyl groups. As the reaction proceeded, the multiplets converged to yield a singlet at −3.3 ppm for **5**. Compound **5** was isolated and purified, as described in the Experimental section.

Phenoxycyclotriphosphazene Reactions: Synthesis of $N_3P_3(OPh)_5OSiPh_3$ (3)

Compound **3**, $N_3P_3(OPh)_5OSiPh_3$, was prepared by the reaction sequence shown in Scheme 5. Monochloropentaphenoxycyclotriphosphazene (**12**) reacts with sodium triethylsiloxide in hot dioxane at 100°C for 72 hours to yield the intermediate $N_3P_3(OPh)_5ONa$ (**13**). As the progress of the reaction was monitored by ^{31}P NMR spectroscopy, the spectrum of **12** (υ_A: 22.1 ppm, υ_B: 7.0 ppm, J_{PNP} = 85.6 Hz) diminished slowly while the spectrum of **13** (υ_A: 6.2 ppm, υ_B: 9.2 ppm, J_{PNP} = 75.2 Hz) began to appear. Hexaethyldisiloxane and NaCl were also formed during this reaction, and this indicated the formation of the $P–O^-Na^+$ unit from the P–Cl bond of **12**. Species **13** reacts with Ph_3SiCl to yield **3**, which gives rise to a new ^{31}P NMR AB_2 spin pattern (υ_A: 3.0 ppm, υ_B: 8.7 ppm, J_{PNP} = 96.6 Hz). However, attempts to isolate **3** from the reaction by-products by chromatographic and recrystallization techniques were unsuccessful. An alternative procedure was developed to isolate **3**.

Intermediate **13** was protonated with HCl to yield the phosphazadiene species $N_2P_2(OPh)_4$-NHP(O)(OPh) (**14**), which was purified by chromatographic and recrystallization techniques. Compound **14** was also prepared by the known reaction of **12** with ethanolic potassium hydroxide, followed by protonation with HCl [29]. When **14** reacts with NaH, hydrogen is evolved and intermediate **13** is formed, free from organosilicon by-products. The reaction of **13** with Ph_3SiCl yielded **3**, which was isolated as an oil and was fully characterized, as described in the Experimental section. An alternative synthesis of $N_3P_3(OPh)_5OSiPh_3$, by the condensation reaction between $N_3P_3(OPh)_5(OBu^n)$ and Ph_3SiCl, has been described elsewhere [30].

Structural Characterization and Physical Properties of the Organosiloxycyclotriphosphazenes

The cyclotriphosphazene compounds were characterized by a combination of ^{31}P NMR, ^1H NMR, and infrared spectroscopy, mass spectrometry, and microanalysis. These data are presented in the Experimental section. The microanalytical data are consistent with the proposed structures. The mass spectra for the cyclotriphosphazene compounds showed the expected parent ion peaks. Major fragments were detected for the loss of phenyl (M-77) (species **2**, **3**, **4**, and **14**), phenoxy (M-93) (species **14**), chlorine (M-35) (species **4**), and ethyl (M-29) (species **9**). The correct chlorine isotope pattern was detected when applicable. Mass spectral analysis of compound **5** was not successful.

The ^{31}P NMR spectra of the cyclotriphosphazenes were consistent with the substitution pattern around the phosphazene ring. An AB_2 spin pattern was found for species **2**, **3**, **4**, **9**, and **14**. The PCl_2 chemical shifts for **2**, **4**, and **9** were detected at 23–20 ppm and the $P(OPh)_2$ shift for **3** was detected at 8.3 ppm. The $P(OSIR_3)_2$ resonances in **2** and **9** were shifted downfield and were detected at −16 to −17 ppm. The $P(Cl)(OSiPh_3)$ resonance for **4** and the

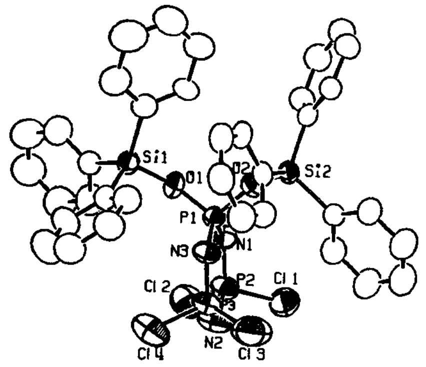


FIGURE 1 ORTEP view of $N_3P_3Cl_4(OSiPh)_3$ (**2**) approximately parallel to the plane of the cyclophosphazene ring.

$P(OPh)(OSiPh_3)$ resonance for **3** were detected at 2.9 and 2.5 ppm, respectively. Coupling constants in the AB_2 spin patterns ranged from 60 to 100 Hz. The $P(OSiPh_3)_2$ resonance for the hexasubstituted compound **5** was detected as a singlet at −3.3 ppm. The ^{31}P NMR spectrum of **14** is highly dependent on the nature of the solvent and has been discussed elsewhere [31].

The ^1H NMR spectra of the triphenylsiloxy and phenoxy-substituted cyclotriphosphazenes (**2–5**, **14**) showed multiplets at δ 7.7–6.6. The quartet and triplet for the ethyl group in **9** were split into complex multiplets by the phosphorus atoms of the phosphazene ring.

The infrared spectra showed mainly two intense vibrations for the P–N segments at 1300–1100 cm^{-1} and the P–O–Si linkage at 1100–1000 cm^{-1}. Other characteristic vibrations were detected for Si–Ph (1430–1400 cm^{-1}), C–C (1590, 1480 cm^{-1}), and P–Cl (600–580, 520–500 cm^{-1}) when applicable.

The triphenylsiloxy species **2**, **4**, and **5** are white crystalline solids while **3** is a viscous oil that resisted crystallization. These species were all stable toward atmospheric moisture but did, however, hydrolyze at the P–O–Si linkage (to yield Ph_3SiOH and insoluble products) in wet solvents or in contact with substrates such as silica gel. By contrast, the *alkyl*siloxycyclotriphosphazene compound *gem*-$N_3P_3Cl_4(OSiEt_3)_2$ (**9**), and others [23,24], hydrolyze more readily in contact with moisture than do the triphenylsiloxy analogues. Thus, it can be stated that, in general, triphenylsiloxycyclotriphosphazenes are more resistant to hydrolysis than their alkyl counterparts, but the hydrolytic instability of the P–O–Si linkage still exists.

Compounds $N_3P_3Cl_5OSiPh_3$ (**4**) and *gem*-$N_3P_3Cl_4(OSiEt_3)_2$ (**9**) are volatile at temperatures near 150°C (0.05 mm Hg) but species *gem*-$N_3P_3Cl_4(OSiPh_3)_2$ (**2**), $N_3P_3(OSiPh_3)_6$ (**5**), and $N_3P_3(OPh)_5OSiPh_3$ (**3**) are not, and these species decompose when heated to higher temperatures. A preliminary study of the thermal stability of compounds **2–5** and **9** indicated that the P–O–Si linkage in these compounds is thermally unstable at temperatures that approach 200°C. For example, when the compound *gem*-$N_3P_3Cl_4(OSiPh_3)_2$ was heated in vacuo at 210°C for 48 hours, Ph_3SiCl volatilized and insoluble cyclotriphosphazene species were formed. This indicated that a condensation process occurs that eliminates Ph_3SiCl and may form P–O–P cross-linkages. In addition, the high temperatures required for the synthesis of **4** and **5** (180 and 200°C, respectively) appear to be responsible for the formation of insoluble decomposition products during these condensation reactions.

The Crystal and Molecular Structure of gem-$N_3P_3Cl_4(OSiPh_3)_2$ (2)

A single-crystal X-ray structure determination was carried out for **2**, since the triphenylsiloxy groups in the molecule represent one of the bulkiest side groups yet attached to a cyclotriphosphazene ring. It was of interest to determine the effect of the large side groups on the bond lengths, bond angles, and planarity of the cyclotriphosphazene ring.

The structure of **2** is shown in Figure 1. All nonhydrogen atoms except the carbon atoms of the phenyl rings have been labeled. In addition, the carbon atoms of the phenyl rings of **2** have been left unshaded for clarity. Bond distances, bond angles, and positional parameters are listed in Tables 1, 2, and 3, respectively. The phosphazene ring was found to be almost planar (χ^2 = 110 for the weighted least-squares plane through the phosphazene ring). Two triphenylsiloxy groups are attached to the phosphazene ring at P(1) and two chlorine atoms are attached to each of the two remaining phosphorus atoms.

The P–N bond lengths in the phosphazene ring of **2** are unequal. The average P–N bond length from P(1) to the adjacent nitrogen atoms N(1) and N(3) is 1.583(4) Å, while the N(1)–P(2)/N(3)–P(3) average bond length is 1.554(5) Å. The P(2)–N(2)/P(3)–N(2) average bond distance is 1.565(10) Å. The alternation of average bond lengths at increased distance from the substituted phosphorus atom is detected in many cyclotriphosphazenes [32,33]. The P–N bonds of **2** follow this trend.

The skeletal phosphorus atoms in cyclic phosphazenes usually occupy a distorted tetrahedral geometry with endocyclic ring angles near 120°, and exocyclic angles near 100°. The exocyclic O(1)–P(1)–O(2) angle is widened to 103.4(2)° due to the steric repulsion of the two triphenylsiloxy groups, and the N(1)–P(1)–N(3) angle is constricted to 115.8(2)° to accommodate hybridization changes. The other N–P–N endocyclic angles, centered at P(2) and P(3), are 119.3(3) and 119.7(3)°, respectively. These angles are similar to the N–P–N angles of 118.4(2)° in $(NPCl_2)_3$ [34]. The exocyclic Cl–P–Cl angles centered at P(2) and P(3) of 100.1(1) and 101.1(1)° do not differ significantly from those reported for $(NPCl_2)_3$ [34].

The P–Cl bonds of **2** have an average length of 1.999(11) Å, with the P(2)–Cl(1) bond distance of 2.014(3) Å being the longest. The P–Cl bond distances in **2** are comparable to the P–Cl bond distances in $(NPCl_2)_3$ [34]. The average P–O bond length of 1.549(7) Å in **2** is slightly shorter than the average P–O bond lengths of 1.582(2) and 1.581(10) Å in $N_3P_3(OPh)_6$ [35] and $N_4P_4(OMe)_8$ [36], respectively.

The bond angles and the bond lengths associated with the triphenylsiloxy groups are unexceptional. The average Si–O bond length of 1.661(1) Å, the average Si–C bond length of 1.860(4) Å, and the average C–C bond length of 1.40(1) Å are similar to the Si–O, Si–C, and C–C bond lengths reported for hexaphenylcyclotrisiloxane [37] and octaphenylcyclotetrasiloxane [38]. The average C–Si–C and C–C–C bond angles of 112(2) and 120(1)°, respectively, were comparable to the same angles of the phenylcyclosiloxane compounds. The P(1)–O(1)–Si(1) and P(1)–O(2)–Si(2) bond angles were 150.5(3) and 143.3(3)°, respectively.

TABLE 1 Bond Lengths (Å) for *gem*-$N_3P_3Cl_4(OSiPh_3)_2$ (**2**)

Cl(1)–P(2)	2.014(3)	C(13)–C(14)	1.368(11)
Cl(2)–P(2)	1.997(3)	C(14)–C(15)	1.387(11)
Cl(3)–P(3)	1.990(3)	C(15)–C(16)	1.447(10)
Cl(4)–P(3)	1.993(3)	C(21)–C(22)	1.405(8)
P(1)–O(1)	1.542(4)	C(21)–C(26)	1.404(8)
P(1)–O(2)	1.555(4)	C(22)–C(23)	1.461(9)
P(1)–N(1)	1.579(5)	C(23)–C(24)	1.367(10)
P(1)–N(3)	1.587(4)	C(24)–C(25)	1.417(11)
P(2)–N(1)	1.549(5)	C(25)–C(26)	1.411(10)
P(2)–N(2)	1.576(6)	C(31)–C(32)	1.398(9)
P(3)–N(2)	1.555(6)	C(31)–C(36)	1.386(8)
P(3)–N(3)	1.559(5)	C(32)–C(33)	1.388(9)
Si(1)–O(1)	1.662(4)	C(33)–C(34)	1.391(10)
Si(1)–C(11)	1.857(6)	C(34)–C(35)	1.377(10)
Si(1)–C(21)	1.855(6)	C(35)–C(36)	1.402(10)
Si(1)–C(31)	1.862(6)	C(41)–C(42)	1.392(8)
Si(2)–O(2)	1.660(4)	C(41)–C(46)	1.387(8)
Si(2)–C(41)	1.867(6)	C(42)–C(43)	1.409(9)
Si(2)–C(51)	1.858(6)	C(43)–C(44)	1.415(10)
Si(2)–C(61)	1.861(6)	C(44)–C(45)	1.366(10)
C(11)–C(12)	1.406(8)	C(45)–C(46)	1.399(9)
C(11)–C(16)	1.392(9)	C(51)–C(52)	1.400(8)
C(12)–C(13)	1.419(9)	C(51)–C(56)	1.407(8)
C(52)–C(53)	1.401(8)		
C(53)–C(54)	1.377(10)		
C(54)–C(55)	1.394(10)		
C(55)–C(56)	1.400(9)		
C(61)–C(62)	1.397(8)		
C(61)–C(66)	1.413(8)		
C(62)–C(63)	1.397(8)		
C(63)–C(64)	1.377(9)		
C(64)–C(65)	1.387(9)		
C(65)–C(66)	1.410(8)		

EXPERIMENTAL

Materials

Hexachlorocyclotriphosphazene was provided by Ethyl Corp. and was purified by recrystallization from hexane and sublimation at 50°C (0.05 mm Hg).

TABLE 2 Bond Angles (°) for *gem*-$N_3P_3Cl_4(OSiPh_3)_2$ (**2**)

O(1)–P(1)–O(2)	103.4(2)	C(21)–Si(1)–C(31)	109.3(3)
O(1)–P(1)–N(1)	108.3(3)	O(2)–Si(2)–C(41)	107.5(2)
O(1)–P(1)–N(3)	111.1(2)	O(2)–Si(2)–C(51)	103.5(2)
O(2)–P(1)–N(1)	107.8(2)	O(2)–Si(2)–C(61)	110.9(2)
O(2)–P(1)–N(3)	109.8(2)	C(41)–Si(2)–C(51)	112.6(2)
N(1)–P(1)–N(3)	115.8(2)	C(41)–Si(2)–C(61)	109.2(3)
Cl(1)–P(2)–Cl(2)	100.1(1)	C(51)–Si(2)–C(61)	112.9(3)
Cl(1)–P(2)–N(1)	108.6(2)	P(1)–O(1)–Si(1)	150.5(3)
Cl(1)–P(2)–N(2)	108.5(2)	P(1)–O(2)–Si(2)	143.3(3)
Cl(2)–P(2)–N(1)	109.2(2)	P(1)–N(1)–P(2)	122.8(3)
Cl(2)–P(2)–N(2)	109.4(2)	P(2)–N(2)–P(3)	119.8(3)
N(1)–P(2)–N(2)	119.3(3)	P(1)–N(3)–P(3)	122.3(3)
Cl(3)–P(3)–Cl(4)	101.1(1)	Si(1)–C(11)–C(12)	119.5(5)
Cl(3)–P(3)–N(2)	108.3(3)	Si(1)–C(11)–C(16)	120.5(5)
Cl(3)–P(3)–N(3)	107.8(2)	C(12)–C(11)–C(16)	120.0(6)
Cl(4)–P(3)–N(2)	108.6(2)	C(11)–C(12)–C(13)	119.5(7)
Cl(4)–P(3)–N(3)	109.7(2)	C(12)–C(13)–C(14)	120.5(7)
N(2)–P(3)–N(3)	119.7(3)	C(13)–C(14)–C(15)	121.4(7)
O(1)–Si(1)–C(11)	105.3(3)	C(14)–C(15)–C(16)	119.0(7)
O(1)–Si(1)–C(21)	111.4(2)	C(11)–C(16)–C(15)	119.6(7)
O(1)–Si(1)–C(31)	104.1(3)	Si(1)–C(21)–C(22)	120.2(5)
C(11)–Si(1)–C(21)	111.5(3)	Si(1)–C(21)–C(26)	120.3(5)
C(11)–Si(1)–C(31)	115.1(3)	C(22)–C(21)–C(26)	119.4(6)
C(21)–C(22)–C(23)	121.0(6)	Si(2)–C(51)–C(56)	121.2(5)
C(22)–C(23)–C(24)	119.5(7)	C(52)–C(51)–C(56)	118.3(5)
C(23)–C(24)–C(25)	120.4(7)	C(51)–C(52)–C(53)	120.6(6)
C(24)–C(25)–C(26)	120.4(7)	C(52)–C(53)–C(54)	120.1(6)
C(21)–C(26)–C(25)	119.2(7)		
Si(1)–C(31)–C(32)	118.4(5)	C(53)–C(54)–C(55)	120.6(6)
Si(1)–C(31)–C(36)	121.7(5)	C(54)–C(55)–C(56)	119.4(7)
C(32)–C(31)–C(36)	119.6(6)	C(51)–C(56)–C(55)	120.8(7)
C(31)–C(32)–C(33)	119.8(7)	Si(2)–C(61)–C(62)	120.3(5)
C(32)–C(33)–C(34)	120.7(7)	Si(2)–C(61)–C(66)	120.7(5)
C(33)–C(34)–C(35)	119.3(7)	C(62)–C(61)–C(66)	119.0(6)
C(34)–C(35)–C(36)	120.6(7)	C(61)–C(62)–C(63)	121.5(6)
C(31)–C(36)–C(35)	119.9(7)	C(62)–C(63)–C(64)	119.5(6)
Si(2)–C(41)–C(42)	122.8(5)	C(63)–C(64)–C(65)	120.2(6)
Si(2)–C(41)–C(46)	118.9(4)	C(64)–C(65)–C(66)	121.2(6)
C(42)–C(41)–C(46)	118.3(6)	C(61)–C(66)–C(65)	118.5(6)
C(41)–C(42)–C(43)	121.6(7)		
C(42)–C(43)–C(44)	118.1(7)		
C(43)–C(44)–C(45)	120.5(7)		
C(44)–C(45)–C(46)	120.2(7)		
C(41)–C(46)–C(45)	121.2(6)		
Si(2)–C(51)–C(52)	120.4(5)		

TABLE 3 Positional Parameters for *gem*-$N_3P_3Cl_4(OSiPh_3)_2$ (2)

Atom	*x*	*y*	*z*
Cl(1)	0.1215(1)	−0.313(2)	0.4083(1)
Cl(2)	0.0457(2)	0.1700(2)	0.3355(1)
Cl(3)	0.0882(2)	0.0763(2)	0.6468(2)
Cl(4)	0.0132(1)	0.2865(2)	0.5905(2)
P(1)	0.2321(1)	0.2282(1)	0.5196(1)
P(2)	0.1094(1)	0.1206(2)	0.4316(1)
P(3)	0.0915(1)	0.1806(2)	0.5679(1)
Si(1)	0.2642(1)	0.4662(2)	0.5225(1)
Si(2)	0.3709(1)	0.1243(2)	0.6249(1)
O(1)	0.2554(3)	0.3402(3)	0.5016(3)
O(2)	0.3150(3)	0.1759(4)	0.5499(3)
N(1)	0.1941(3)	0.1722(5)	0.4450(3)
N(2)	0.0560(4)	0.1294(6)	0.4925(4)
N(3)	0.1782(3)	0.2282(5)	0.5812(3)
C(11)	0.3734(4)	0.4870(6)	0.5596(4)
C(12)	0.4290(4)	0.4075(6)	0.5527(4)
C(13)	0.5122(5)	0.4223(7)	0.5829(5)
C(14)	0.5389(5)	0.5147(8)	0.6156(5)
C(15)	0.4858(6)	0.5950(8)	0.6225(5)
C(16)	0.4004(5)	0.5805(7)	0.5938(5)
C(21)	0.2015(4)	0.5022(6)	0.5909(4)
C(22)	0.2237(5)	0.4690(6)	0.6643(4)
C(23)	0.1776(5)	0.4978(7)	0.7177(4)
C(24)	0.1108(5)	0.5594(8)	0.6975(5)
C(25)	0.0876(5)	0.5943(8)	0.6239(5)
C(26)	0.1327(5)	0.5651(7)	0.5703(5)
C(31)	0.2244(4)	0.5333(6)	0.4338(4)
C(32)	0.1575(5)	0.4902(7)	0.3869(4)
C(33)	0.1224(6)	0.5426(8)	0.3232(5)
C(34)	0.1529(6)	0.6381(8)	0.3054(5)
C(35)	0.2186(6)	0.6806(8)	0.3519(6)
C(36)	0.2542(5)	0.6291(7)	0.4168(5)
C(41)	0.3330(4)	−0.0108(5)	0.6346(4)
C(42)	0.2672(5)	−0.0526(6)	0.5864(5)
C(43)	0.2369(5)	−0.1525(7)	0.5974(5)
C(44)	0.2762(5)	−0.2109(7)	0.6588(5)
C(45)	0.3417(5)	−0.1709(7)	0.7056(5)
C(46)	0.3707(5)	−0.0716(6)	0.6932(4)
C(51)	0.4748(4)	0.1262(5)	0.6048(4)
C(52)	0.5420(4)	0.1126(6)	0.6615(4)
C(53)	0.6201(4)	0.1074(6)	0.6461(5)
C(54)	0.6315(5)	0.1152(6)	0.5746(5)
C(55)	0.5662(5)	0.1317(6)	0.5171(5)
C(56)	0.4879(4)	0.1354(6)	0.5322(4)
C(61)	0.3618(4)	0.2013(5)	0.7081(4)
C(62)	0.3022(4)	0.1769(6)	0.7485(4)
C(63)	0.2925(5)	0.2355(6)	0.8098(4)
C(64)	0.3436(5)	0.3180(6)	0.8321(4)
C(65)	0.4041(5)	0.3429(6)	0.7937(4)
C(66)	0.4146(4)	0.2855(5)	0.7313(4)

Triphenylsilanol and triphenylchlorosilane were obtained from Aldrich and were recrystallized from toluene and hexane, respectively, before use. Triethylsilanol and triethylchlorosilane were obtained from Petrarch Systems, Inc. and were distilled and then stored over molecular sieves before use. Sodium hydride (Aldrich) was washed several times with dry tetrahydrofuran to remove mineral oil and was stored in a dry box before use. Tetrahydrofuran (THF) and 1,4-dioxane were dried over sodium benzophenone ketyl and were distilled in an atmosphere of dry nitrogen before use.

Analytical Techniques

^{31}P NMR spectra were obtained with a Varian CFT-20 NMR spectrometer operated at 32 MHz or a JEOL FX90Q NMR spectrometer operated at 36.2 MHz. ^{31}P NMR chemical shifts are relative to 85% H_3PO_4 at 0 ppm with positive shift values downfield from the reference. ^1H NMR spectra were recorded with the use of a Bruker WP-200 spectrometer operated at 200 MHz. Chemical shifts are relative to tetramethylsilane at $\delta = 0$. ^{13}C NMR (^1H decoupled) spectra were recorded with the use of a Bruker WP-200 spectrometer operated at 50.3 MHz or a JEOL FX90Q spectrometer operated at 22.5 MHz. All spectra are referenced to internal tetramethylsilane at 0 ppm. Infrared spectra were obtained with the use of a Perkin-Elmer 283B grating spectrometer. All reactions were carried out under an atmosphere of dry nitrogen. Elemental analyses were obtained from Galbraith Laboratories, Knoxville, TN.

Synthesis of $N_3P_3Cl_5OBu^n$(10), $N_3P_3(OBu^n)_6$ (11), $N_3P_3(OPh)_5Cl$ (12), *and* $N_3P_3(OPh)_5OH$ (14)

The syntheses of compounds 10 [24,39], 11 [24,40,41], 12 [42,43], and 14 [29] were based on methods described previously.

Synthesis of gem-$N_3P_3Cl_4(OSiPh_3)_2$ (2)

Triphenylsilanol (16.7 g, 0.0604 mol) was dissolved in dry dioxane (100 mL) and was added *cautiously* to a suspension of NaH (1.70 g, 0.078 mol) in dry dioxane (100 mL). After the evolution of hydrogen had ceased, the solution of sodium triphenylsiloxide was filtered under nitrogen to remove excess NaH and added in one portion to a solution of $(NPCl_2)_3$ (5.00 g, 0.0144 mol) and $(n\text{-Bu})_4NBr$ (1.80 g, 0.00558 mol) in dry dioxane (100 mL). The reaction mixture was stirred for 24 hours and during this time a white solid precipitated. Tetraphenylchlorosilane (12.7 g, 0.431 mol) was added to the reaction mixture, which was then stirred at 25°C for 2 hours. The reaction mixture was filtered through a coarse fritted addition funnel under nitrogen atmosphere to remove NaCl and some precipitated $Ph_3SiOSiPh_3$. The solvent was removed from the filtrate under vacuum to yield an oil. Toluene (200 mL) was added to the oil and the solution was filtered to remove residual $Ph_3SiOSiPh_3$. Toluene was removed from the filtrate to yield crude **2**, which was recrystallized several times from heptane to yield pure **2** as a white solid. Yield 3.2 g (27%), m.p. 142–144°C.

Characterization Data for **2**. ^{31}P NMR ($CDCl_3$) AB_2 spin pattern (υ_a: -16.6 ppm, υ_β: 21.0 ppm, J_{PNP} = 80.8 Hz). ^1H NMR δ = 7.55–7.28 (m). IR 1250, 1200 cm^{-1} (vs, PN), 1110–1040 cm^{-1} (vs, POSi). MS calcd. for $C_{36}H_{30}Cl_4N_3O_2P_3Si_2$, 827; found 827 ($Cl_4$ isotope pattern). Found: C, 52.13; H, 3.59; N, 4.97; Si, 6.69.

Synthesis of gem-$N_3P_3Cl_4(OSiEt_3)_2$ (9)

Triethylsilanol (15.2 g, 0.115 mol) was added dropwise *with caution* to sodium (3.45 g, 0.150 mol) in dry dioxane (100 mL). On completion of hydrogen evolution, the dioxane solution of sodium triethylsiloxide was transferred by use of a double-tipped syringe needle to a solution of $(NPCl_2)_3$ (5.00 g, 0.0144 mol) and $(n\text{-Bu})_4NBr$ (1.80 g, 0.00559 mol) in dry dioxane (100 mL). The reaction mixture was stirred at 25°C for 5 hours, after which time ^{31}P NMR spectroscopy indicated that intermediate **8** had formed. Triethylchlorosilane (13.0 g, 0.0863 mol) was added to the reaction mixture, which was stirred at 25°C for 1 hour. Dioxane was removed under vacuum to yield an oil that contained NaCl precipitate. This mixture was distilled at 0.05 mm Hg, which first yielded $Et_3SiOSiEt_3$ and residual Et_3SiCl (50–60°C), then gave **9** (145–150°C) as a clear liquid. Compound **9** was redistilled at 145°C/0.05 mm Hg and a center fraction was collected. Yield 2.0 g (26%).

Characterization Data for **9**. ^{31}P NMR ($CDCl_3$) AB_2 spin pattern (υ_A: -16.4 ppm, υ_B: 20.0 ppm, J_{PNP} = 77.1 Hz). ^1H NMR ($CDCl_3$) $SiCH_2CH_3$, δ = 0.76 (qm) 2H; $SiCH_2CH_3$, δ = 1.00 (tm) 3H. ^{13}C NMR ($CDCl_3$) $SiCH_2CH_3$, υ = 5.06 ppm (d) (J_{POSiC} = 1.2 Hz); $SiCH_2CH_3$, υ = 6.27 (s). IR 1180 cm^{-1} (vs, PN); 1080 cm^{-1} (vs, POSi). MS calcd. for $C_{12}H_{30}C_4N_3O_2P_3Si_2$, 539; found 539 ($Cl_4$ isotope pattern). Elem. anal. calcd. for **9**: C, 26.72; H, 5.62; N, 7.79; Si, 10.42. Found: C, 26.65; H, 5.86; N, 7.46; Si, 9.55.

Synthesis of $N_3P_3Cl_5OSiPh_3$ (4)

Monobutoxypentachlorocyclotriphosphazene (**10**) (5.00 g, 0.0130 mol) and Ph_3SiCl (15.3 g, 0.0519 mol) were heated at 180°C in an atmosphere of dry nitrogen. During the reaction, the ^{31}P NMR AB_2 spin pattern associated with **10** disappeared and a new AB_2 spin pattern appeared for **4** after approximately 20 hours. When cooled, the contents of the reaction vessel were transferred to a vacuum sublimator in a nitrogen-filled dry bag and were heated at 100°C (0.05 mm Hg). Excess Ph_3SiCl sublimed onto a water-cooled cold finger, and *n*-BuCl was collected in a trap at −196°C. The sublimation was recrystallized several times from hot heptane to yield a white solid. Yield 2.4 g (31%); m.p. 121–122°C.

Characterization Data for **4**. ^{31}P NMR ($CDCl_3$) AB_2 spin pattern (υ_A: 2.9 ppm, υ_B: 22.1 ppm, J_{PNP} = 70.0 Hz). ^1H NMR ($CDCl_3$) δ = 7.74–7.38 (m). IR 1195 cm^{-1} (vs, PN), 1070 cm^{-1} (vs, POSi). MS calcd. for $C_{18}H_{15}Cl_5N_3OP_3Si$, 587 ($Cl_5$ isotope pattern). Elem. anal. calcd. for **4**: C, 36.79; H, 2.58; N, 7.15; Si, 4.78. Found: C, 36.95; H, 2.49; N, 7.23; Si, 5.05.

Synthesis of $N_3P_3(OSiPh_3)_6$ (5)

Hexabutoxycyclotriphosphazene (**11**) (1.00 g, 0.00174 mol) and Ph_3SiCl (12.3 g, 0.0417 mol) were stirred together at 200°C in an atmosphere of dry nitrogen for approximately 144 hours. ^{31}P NMR spectroscopy showed that all of **11** had been converted to **5**. When cooled, the contents of the reaction vessel were transferred to a vacuum sublimator in a nitrogen-filled dry bag and then heated at 90°C (0.05 mm Hg) to remove residual Ph_3SiCl and *n*-BuCl as described previously. The sublimation residue was recrystallized twice from a CH_2Cl_2/hexane solvent mixture and once from a toluene/heptane solution to yield **5** as a white solid. Yield 0.3 g (10%), m.p. >300°C.

Characterization Data for **5**. ^{31}P NMR ($CDCl_3$) -3.3 ppm (s). ^1H NMR ($CDCl_3$) δ = 7.70–7.15 (m). IR 1240 cm^{-1} (s, PN), 1050–970 cm^{-1} (vs, POSi). Elem. anal. calcd. for **5**: C, 72.57; H, 5.09; N, 2.35; Si, 9.43. Found: C, 72.76; H, 5.55; N, 2.24; Si, 9.50.

Synthesis of $N_2P_2(OPh)_4NHP(OPh)(O)$ (14) (*via* 12 *and Et_2SiONa*)

Triethylsilanol (12.5 g, 0.0945 mol) was dissolved in dry dioxane and was added dropwise to a suspension of NaH (2.15 g, 0.0896 mol) in dry dioxane (100 mL). After the evolution of hydrogen had ceased, the solution of sodium triethylsiloxide was added to a solution of **12** (8.00 g, 0.0126 mol) in dry dioxane (100 mL). The reaction mixture was then refluxed at 100°C for a period of 52 hours. When cooled to room temperature, dioxane was removed under reduced pressure and the resultant oil was dissolved in ethanol (80 mL). Concentrated HCl (10 mL) was added to the ethanolic solution, which gave a white precipitate. Water (100 mL) was added and the mixture was extracted with 3–100 mL portions of methylene chloride. The organic layer was dried over $MgSO_4$, filtered, and evaporated under reduced pressure to yield an oil. Chromatography on silica gel using 100% methylene chloride eluted hexaethyldisiloxane and residual triethylsilanol. A solvent mixture that consisted of 90% CH_2Cl_2/10% ethanol eluted **14**, which was recrystallized from 95% ethanol. Yield 3.6 g (46%); m.p. 162–164°C (lit. [24] 163–165°C).

Characterization Data for **14**. ^{31}P NMR ($CDCl_3$) AB_2 spin pattern (υ_A: -5.5 ppm, υ_B: 2.7 ppm, J_{PNP} = 53.4 Hz); (dioxane, D_2O lock) AB_2 spin pattern (υ_A: 4.5 ppm, υ_B: -2.9 ppm, J_{PNP} = 61.6 Hz). ^1H NMR ($CDCl_3$) δ = 7.21–6.88 (m), 2.49 (br). IR 1200, 1180 cm^{-1} (vs, PN). MS calcd. for $C_{30}H_{26}N_3O_6P_3$, 617; found, 617. Elem. anal. calcd. for **14**: C, 58.35; H, 4.25; N, 6.81. Found: C, 58.23; H, 4.39; N, 6.67.

Synthesis of $N_3P_3(OPh)_5OSiPh_3$ (**3**)

Compound **14** (3.00 g, 0.0486 mol) was dissolved in dry dioxane (20 mL) and was added to NaH (0.30 g, 0.0125 mol) in dry dioxane (100 mL). The reaction mixture was stirred until the evolution of hydrogen had ceased, then filtered through a medium-porosity fritted addition funnel under an atmosphere of dry nitrogen to remove excess NaH. To the solution of **13** was added Ph_3SiCl (2.86 g, 0.00970 mol), then the reaction mixture was heated to reflux for a period of 12 hours. The reaction mixture was again filtered through a medium-porosity fritted addition funnel under a dry nitrogen atmosphere to remove precipitated NaCl. Solvent was removed from the filtrate under vacuum to yield a viscous oil. The oil was transferred to a vacuum sublimator and heated at 160°C (0.05 mm Hg) until all of the residual Ph_3SiCl sublimed onto the cold finger, which was cooled to −78°C. Compound **3** remained as an oil. Yield 2.8 g (65%).

Characterization Data for **3**. ^{31}P NMR ($CDCl_3$) AB_2 spin pattern (υ_A: 2.5 ppm, υ_B: 8.3 ppm, J_{PNP} = 96.0 Hz). ^1H NMR ($CDCl_3$) δ = 7.59–6.61 (m). IR 1300–1250 cm^{-1} (vs, PN), 1070 cm^{-1} (m, POSi). MS calcd. for $C_{48}H_{40}N_3O_6P_3Si$, 875; found, 875. Elem. anal. calcd. for **3**: C, 65.81; H, 4.61; N, 4.80; Si, 3.21. Found: C, 65.59; H, 4.56; N, 4.45; Si, 3.17.

The Reaction between gem-$N_3P_3Cl_4(OSiPh_3)_2$ (**2**) *and Ph_3SiONa*

A solution of Ph_3SiONa was prepared by the dropwise addition of Ph_3SiOH (3.34 g, 0.0121 mol) in dry dioxane (50 mL) to NaH (0.32 g, 0.0133 mol) in dry dioxane (30 mL). (It was sometimes necessary to gently heat the reaction mixture to initiate the formation of Ph_3SiONa.) On completion of hydrogen evolution, the mixture was filtered under nitrogen to remove excess NaH, then transferred to a graduated addition funnel. Compound **2**, *gem*-$N_3P_3Cl_4(OSiPh_3)$ (2.00 g, 0.00242 mol), was dissolved in dry dioxane (50 mL) and $(n\text{-Bu})_4$, NBr (0.2 g, 0.00062 mol) was added. One equivalent of Ph_3SiONa (ca. 15–20 mL) was added to the solution of **2** and the mixture was stirred for 1 hour at 25°C. ^{31}P NMR spectroscopy showed that **2** was completely converted to intermediate **7**. To the NMR aliquot was added Ph_3SiCl, which converted **7** back to **2**. A portion of the reaction mixture (10 mL) was evaporated to dryness and washed successively with hexane and toluene. The residual white solid was analyzed by thin layer chromatography and gas chromatography, which showed that this compound was $Ph_3SiOSiPh_3$, by retention time comparison with an authentic sample. The remainder of the Ph_3SiONa solution was added to the reaction mixture, which was then stirred at 25°C for 24 hours. ^{31}P NMR spectroscopic analysis indicated that **7** had been completely converted to **8**. Addition of Ph_3SiCl to the NMR aliquot yielded **7**, which gave **2** on addition of more Ph_3SiCl. To another NMR aliquot of **8** was added excess Et_3SiCl, which yielded *gem*-$N_3P_3Cl_4(OSiPh_3)$, as determined by ^{31}P NMR spectroscopic analysis.

X-ray Structure Determination Technique

Crystals of **2** for X-ray diffraction examination were grown from hexane/methylene chloride by slow solvent evaporation at 25°C. A clear crystal, 0.08 × 0.20 × 0.67 mm, was mounted on a glass fiber, which was then transferred to a eucentric goniometer head and attached to an Enraf-Nonius four-circle CAD4 automated diffractometer controlled by a PDP 8A computer. A full rotation orientation photograph was taken with a Polaroid cassette accessory, and 25 reflections of moderate intensity were chosen and centered accurately with the use of an Enraf-Nonius program [44]. These reflections were then used to obtain an orientation matrix for data collection. A monoclinic cell of dimensions a = 16.850(8) Å, b = 12.829(4) Å, c = 18.505(15) Å, and β = 101.00(6)° with V = 3927(7) Å^3 was chosen. The volume found is consistent with Z = 4 and a d(calcd.) = 1.400 g/cm^3. The observed systematic extinctions ($h0l$, $l = 2n + 1$; $0k0$, $k = 2n + 1$) uniquely define the space group $P2_1/c$.

A graphite single-crystal incident beam monochromator was used for the data collection with Mo K_α

TABLE 4 Thermal Parameters for *gem*-$N_3P_3Cl_4(OSiPh_3)_2$ (**2**)

Atom	*U(1,1)*	*U(2,2)*	*U(3,3)*	*U(1,2)*	*U(1,3)*	*U(2,3)*
Cl(1)	0.074(1)	0.072(1)	0.095(2)	−0.019(1)	0.003(1)	−0.019(1)
Cl(2)	0.082(2)	0.117(2)	0.080(2)	0.013(2)	−0.025(1)	−0.011(2)
Cl(3)	0.079(1)	0.149(2)	0.136(2)	−0.031(1)	0.026(1)	0.064(2)
Cl(4)	0.070(1)	0.140(2)	0.123(2)	0.033(1)	0.031(1)	−0.021(2)
P(1)	0.0362(9)	0.041(1)	0.043(1)	−0.0037(9)	0.0065(8)	0.0028(9)
P(2)	0.043(1)	0.073(1)	0.060(1)	−0.009(1)	0.001(1)	−0.014(1)
P(3)	0.043(1)	0.077(1)	0.065(1)	−0.005(1)	0.0193(9)	−0.002(1)
Si(1)	0.045(1)	0.040(1)	0.050(1)	0.000(1)	0.0101(9)	0.002(1)
Si(2)	0.039(1)	0.037(1)	0.045(1)	0.0026(9)	0.0069(9)	−0.001(1)
O(1)	0.059(3)	0.034(3)	0.070(3)	0.000(2)	0.020(2)	0.000(2)
O(2)	0.043(3)	0.048(3)	0.054(3)	0.005(2)	0.007(2)	0.004(2)
N(1)	0.051(3)	0.068(4)	0.047(3)	−0.014(3)	0.014(3)	−0.010(3)
N(2)	0.040(3)	0.109(5)	0.085(4)	−0.021(4)	0.015(3)	−0.028(4)
N(3)	0.040(3)	0.067(4)	0.044(3)	−0.010(3)	0.010(3)	−0.011(3)
C(11)	0.049(4)	0.058(5)	0.046(4)	−0.009(4)	0.010(3)	0.008(4)
C(12)	0.036(4)	0.066(5)	0.075(5)	0.005(4)	0.012(4)	0.027(4)
C(13)	0.058(5)	0.090(6)	0.081(6)	−0.006(5)	0.013(4)	0.028(5)
C(14)	0.068(5)	0.116(7)	0.072(5)	−0.000(5)	0.019(4)	0.017(6)
C(15)	0.096(6)	0.120(7)	0.060(5)	−0.044(6)	0.014(5)	−0.028(5)
C(16)	0.079(5)	0.081(6)	0.070(5)	−0.024(5)	0.017(4)	−0.029(5)
C(21)	0.050(4)	0.045(4)	0.051(4)	−0.002(4)	0.009(3)	0.005(4)
C(22)	0.065(5)	0.069(5)	0.063(5)	−0.002(4)	0.017(4)	−0.003(5)
C(23)	0.071(5)	0.087(6)	0.074(5)	0.001(5)	0.026(4)	−0.005(5)
C(24)	0.076(6)	0.092(7)	0.100(6)	0.003(5)	0.025(5)	−0.002(6)
C(25)	0.084(6)	0.109(7)	0.098(6)	0.034(5)	0.025(5)	−0.006(6)
C(26)	0.057(5)	0.076(6)	0.081(5)	0.021(4)	0.016(4)	−0.003(5)
C(31)	0.046(4)	0.049(4)	0.053(4)	0.004(4)	0.010(3)	0.013(4)
C(32)	0.084(6)	0.076(6)	0.051(5)	−0.007(5)	−0.014(5)	0.004(5)
C(33)	0.084(6)	0.102(7)	0.067(5)	−0.004(6)	0.008(5)	0.005(6)
C(34)	0.090(6)	0.111(7)	0.072(6)	−0.010(6)	0.013(5)	0.027(5)
C(35)	0.081(6)	0.104(7)	0.112(7)	−0.030(5)	−0.013(6)	0.051(6)
C(36)	0.073(5)	0.079(6)	0.081(6)	−0.010(5)	−0.006(5)	0.041(5)
C(41)	0.039(3)	0.039(4)	0.050(4)	0.003(3)	0.012(3)	−0.005(4)
C(42)	0.062(5)	0.053(5)	0.078(5)	0.000(4)	0.008(4)	−0.011(4)
C(43)	0.087(6)	0.053(5)	0.112(7)	−0.017(5)	0.034(5)	−0.018(5)
C(44)	0.095(6)	0.056(5)	0.095(6)	0.003(5)	0.039(5)	−0.001(5)
C(45)	0.086(6)	0.058(5)	0.101(6)	−0.007(5)	0.017(5)	0.018(5)
C(46)	0.062(5)	0.048(5)	0.077(5)	−0.003(4)	0.010(4)	0.006(4)
C(51)	0.046(4)	0.029(4)	0.063(4)	−0.002(3)	0.017(3)	−0.006(4)
C(52)	0.042(4)	0.042(4)	0.079(5)	−0.003(4)	0.002(4)	0.001(4)
C(53)	0.048(4)	0.056(5)	0.088(6)	0.003(4)	0.012(4)	−0.006(5)
C(54)	0.060(5)	0.055(5)	0.137(7)	−0.003(4)	0.038(5)	−0.022(5)
C(55)	0.067(5)	0.064(5)	0.102(6)	−0.004(4)	0.035(4)	−0.022(5)
C(56)	0.050(4)	0.053(5)	0.081(5)	−0.003(4)	0.026(4)	−0.011(4)
C(61)	0.044(4)	0.040(4)	0.049(4)	0.012(3)	0.020(3)	0.002(4)
C(62)	0.060(4)	0.061(5)	0.046(4)	−0.002(4)	0.020(3)	0.001(4)
C(63)	0.077(5)	0.071(5)	0.046(4)	0.005(4)	0.020(4)	−0.008(4)
C(64)	0.075(5)	0.068(5)	0.048(4)	0.018(4)	0.008(4)	−0.004(4)
C(65)	0.071(5)	0.052(5)	0.060(5)	0.011(4)	0.006(4)	−0.011(4)
C(66)	0.059(4)	0.035(4)	0.065(5)	0.007(4)	0.002(4)	−0.004(4)

radiation (λ = 0.71703 Å) at room temperature (take-off angle 2.80°). A θ-2θ scan method was used with a variable scan rate ranging from 5°/min for the most reflections to 1°/min for the weak ones. The angular scan width (ω) was variable and amounted to $(0.8 + 0.347 \tan \theta)°$ both below ka_1 and above ka_2. Right and left backgrounds were each scanned for 25% of the total scan time. During data collection, three "standard" reflections were recorded after every 90 minutes of actual X-ray exposure time and were recentered automatically after every 250 reflections to monitor crystal stability and orientation. The standard reflections were used to rescale the data automatically to correct for drift during data collection (drift corrections were random and varied from 0.974 to 1.042). A total of 5136 reflections were collected for $3.0 < 2\theta < 43.2°$; of these, 2972 had intensities with $I > 2\sigma(I)$ and were considered observed. No correction was made for absorption (μ = 5.18 cm^{-1} for Mo K_α).

The nonhydrogen atoms were located by direct methods using the program MULTAN78 [45], and their coordinates were determined by successive least-squares refinements. The hydrogen atoms were located and fixed at their calculated positions (C–H = 0.97 Å) and were assigned isotropic temperature parameters (B = 5.0 Å^2). Refinement of all positional and anisotropic thermal parameters [46] of all nonhydrogen atoms using a non–poisson-type distribution with a weighting factor of 0.07 yielded a final R_1 = 0.0601 and R_2 = 0.0800. Here, $R_1 = \Sigma||F_0| - |F_c||/\Sigma|F_0|$ and $R_2 = [\Sigma w(|F_0| - |F_c|)^2/\Sigma w(F_0)^2]^{1/2}$.

In the final cycle, the maximum shift was 0.00 esd. Neutral atomic scattering factors [47] were used for all atoms and were corrected for anomalous dispersion [48] (both real and imaginary parts). The final difference map was essentially featureless with a maximum electron density of 0.44 e/Å^3. The thermal parameters for the final cycle of refinement are shown in Table 4.

The following data are available as supplementary material: observed and calculated structure factor amplitudes (Table 5); selected weighted least-squares planes and the distance of atoms from those planes (Table 6); and the stereoscopic view of the molecular structure (Figure 2).

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SUPPLEMENTARY MATERIAL

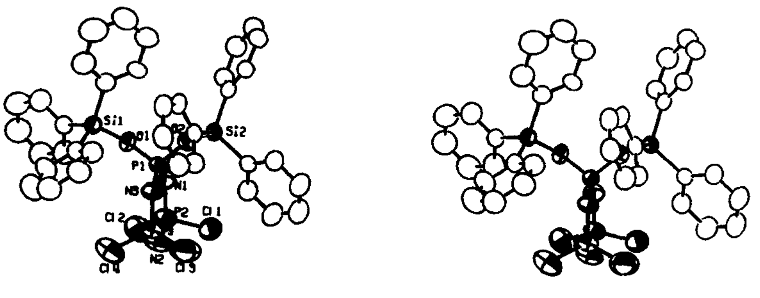

FIGURE 2 Stereoscopic view of the molecular structure of compound **2**.

TABLE 5

A. Equations Defining Least-Squares Planes

Plane 1 through P(1), P(2), P(3), N(1), N(2), N(3):
$-0.3361x + 0.8652y - 0.3722z + 1.6799 = 0$

Plane 2 through P(2), P(3), N(1), N(2), N(3):
$-0.3238x + 0.8707y - 0.3701z + 1.6511 = 0$

Plane 3 through C(11), C(12), C(13), C(14), C(15), C(16):
$0.2979x + 0.3733y - 0.8786z + 5.3079 = 0$

Plane 4 through C(21), C(22), C(23), C(24), C(25), C(26):
$-0.5354x - 0.8165y - 0.2163z + 8.2818 = 0$

Plane 5 through C(31), C(32), C(33), C(34), C(35), C(36):
$0.7251x - 0.4699y - 0.5034z + 5.5560 = 0$

Plane 6 through C(41), C(42), C(43), C(44), C(45), C(46):
$0.7279x - 0.3935y - 0.5616z + 3.9557 = 0$

Plane 7 through C(51), C(52), C(53), C(54), C(55), C(56):
$-0.0696x - 0.9911y - 0.1137z + 3.2659 = 0$

Plane 8 through C(61), C(62), C(63), C(64), C(65), C(66):
$-0.5286x - 0.6148y - 0.5852z + 7.8474 = 0$

TABLE 5 Selected Least-Squares Planes and Distances of Atoms from Planes for *gem*-$N_3P_3Cl_4(OSiPh_3)_2$ (**2**) (continued)

B. Distances of Some Atoms from Plane 1 and Their Estimated Standard Deviations

P(1)	0.003(2)	N(1)	0.011(6)
P(2)	−0.006(2)	N(2)	0.054(7)
P(3)	0.000(2)	N(3)	−0.035(6)

D. Distances (A) of Some Atoms from Plane 3 and Their Estimated Standard Deviations

C(11)	−0.004(7)	C(14)	0.005(9)
C(12)	0.011(7)	C(15)	0.006(9)
C(13)	−0.014(8)	C(16)	−0.004(8)

E. Distances (A) of Some Atoms from Plane 4 and Their Estimated Standard Deviations

C(21)	0.000(7)	C(24)	0.001(10)
C(22)	−0.003(8)	C(25)	−0.005(10)
C(23)	0.003(9)	C(26)	0.003(8)

F. Distances (A) of Some Atoms from Plane 4 and Their Estimated Standard Deviations

C(21)	0.000(7)	C(24)	0.001(10)
C(22)	−0.003(8)	C(25)	−0.005(10)
C(23)	0.003(9)	C(26)	0.003(8)

G. Distances (A) of Some Atoms from Plane 5 and Their Estimated Standard Deviations

C(31)	0.006(7)	C(34)	0.003(10)
C(32)	−0.003(9)	C(35)	0.005(10)
C(33)	−0.003(9)	C(36)	−0.009(9)

H. Distances (A) of Some Atoms from Plane 6 and Their Estimated Standard Deviations

C(41)	−0.011(7)	C(44)	−0.006(9)
C(42)	0.010(8)	C(45)	−0.002(9)
C(43)	0.001(9)	C(46)	0.012(8)

I. Distances (A) of Some Atoms from Plane 7 and Their Estimated Standard Deviations

C(51)	0.004(6)	C(54)	0.015(8)
C(52)	−0.005(7)	C(55)	−0.014(8)
C(53)	−0.003(8)	C(56)	0.003(7)

J. Distances (A) of Some Atoms from Plane 8 and Their Estimated Standard Deviations

C(61)	0.007(7)	C(64)	0.004(8)
C(62)	−0.009(7)	C(65)	−0.004(8)
C(63)	0.003(8)	C(66)	−0.003(7)

K. Chi-Squared Values for the Weighted Least-Squares Planes

Plane 1	110	Plane 5	2
Plane 2	81	Plane 6	7
Plane 3	6	Plane 7	8
Plane 4	1	Plane 8	3

L. Dihedral Angles (degrees) Between Planes

1 and 2	0.8	1 and 6	112.1
1 and 3	56.6	1 and 7	142.3
1 and 4	116.5	1 and 8	22.0
1 and 5	117.6		

TABLE 6 Structure Factor Tables of Fobs and Fcalc for *gem*-$N_3P_3Cl_4(OSiPh_3)_2$ (**2**) (continued)

Values of 10*Fobs and 10*Fcalc

Page 1

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
0	0	4	2075	2154	7	0	4	5	1531	1551	10	0	8	10	218	220	12	1	1	-9	203	203	7
0	0	6	601	567	10	0	4	6	308	302	5	0	8	11	215	194	12	1	1	-8	662	660	11
0	0	8	701	683	11	0	4	7	590	548	11	0	8	12	194	179	14	1	1	-7	576	575	9
0	0	10	264	269	7	0	4	8	100	113	15	0	8	13	140	167	19	1	1	-6	453	346	8
0	0	12	503	510	10	0	4	10	378	416	7	0	8	15	205	244	15	1	1	-5	1863	1734	8
0	0	14	297	309	8	0	4	11	141	155	14	0	9	1	256	256	8	1	1	-4	1122	1038	7
0	1	2	1215	1261	5	0	4	14	210	220	12	0	9	2	322	303	7	1	1	-3	347	295	7
0	1	3	733	804	7	0	4	16	313	309	10	0	9	3	340	303	7	1	1	-2	1171	1131	5
0	1	4	1896	1866	7	0	4	17	279	264	11	0	9	5	223	219	10	1	1	-1	728	794	5
0	1	5	297	375	5	0	4	18	170	162	17	0	9	7	535	569	10	1	1	1	438	476	7
0	1	7	315	318	6	0	5	1	269	268	5	0	9	8	206	199	12	1	1	5	71	22	16
0	1	8	89	94	14	0	5	2	464	438	8	0	9	10	176	196	15	1	1	7	255	242	6
0	1	9	346	360	5	0	5	3	243	199	6	0	9	12	406	371	8	1	1	8	452	578	10
0	1	10	513	529	9	0	5	4	302	281	6	0	10	1	183	174	12	1	1	9	445	442	8
0	1	11	204	171	9	0	5	6	362	366	7	0	10	2	212	219	11	1	1	10	449	437	8
0	1	13	228	227	10	0	5	7	360	343	6	0	10	4	273	308	9	1	1	12	120	152	17
0	1	14	245	231	10	0	5	8	273	254	7	0	10	5	374	389	8	1	1	13	654	642	11
0	1	15	143	174	17	0	5	9	288	270	7	0	10	6	171	183	14	1	1	15	255	265	10
0	1	16	269	271	10	0	5	14	553	557	10	0	10	10	182	186	15	1	1	16	320	320	9
0	1	18	137	142	19	0	5	16	168	146	15	0	10	11	342	363	9	1	2	-17	139	139	17
0	2	0	468	433	7	0	6	0	977	882	11	0	11	1	224	230	11	1	2	-15	127	142	17
0	2	2	271	289	5	0	6	1	463	380	7	0	11	2	418	455	7	1	2	-14	109	144	19
0	2	3	773	737	8	0	6	2	170	105	8	0	11	3	99	154	24	1	2	-12	219	219	9
0	2	4	1315	1312	8	0	6	4	217	203	7	0	11	5	268	270	10	1	2	-11	233	216	8
0	2	5	540	433	9	0	6	5	692	713	11	0	11	8	109	115	23	1	2	-10	122	122	12
0	2	6	466	424	9	0	6	6	100	116	16	0	11	9	301	301	10	1	2	-9	176	192	8
0	2	7	971	895	10	0	6	8	179	165	10	0	12	0	221	262	12	1	2	-8	625	565	10
0	2	8	313	321	6	0	6	9	598	591	11	0	12	2	158	159	16	1	2	-7	411	395	6
0	2	9	828	781	11	0	6	11	166	129	13	0	12	7	262	289	12	1	2	-6	281	271	5
0	2	10	179	147	9	0	6	13	126	121	20	0	13	3	189	203	16	1	2	-5	779	720	9
0	2	11	393	395	7	0	6	15	126	134	21	0	13	4	172	153	17	1	2	-4	286	340	6
0	2	12	248	232	9	0	7	1	254	256	7	1	0	-18	146	156	17	1	2	-3	241	236	5
0	2	13	189	192	11	0	7	2	167	128	9	1	0	-16	233	243	10	1	2	-2	794	790	7
0	2	15	161	168	15	0	7	3	221	204	8	1	0	-14	497	528	10	1	2	-1	1532	1600	6
0	3	1	2833	2793	7	0	7	4	725	736	12	1	0	-12	1089	1066	13	1	2	0	219	254	5
0	3	2	436	454	8	0	7	5	503	530	9	1	0	-10	969	970	12	1	2	1	385	351	7
0	3	3	312	274	6	0	7	6	455	451	8	1	0	-8	478	478	9	1	2	2	1164	1136	7
0	3	4	888	830	9	0	7	7	189	197	11	1	0	-6	104	110	10	1	2	3	1392	1372	7
0	3	5	646	612	10	0	7	8	188	199	11	1	0	-4	246	268	5	1	2	4	1328	1283	8
0	3	6	407	436	7	0	7	10	249	244	10	1	0	-2	1680	1733	5	1	2	5	90	136	13
0	3	7	393	371	7	0	7	11	128	117	19	1	0	2	452	480	7	1	2	6	196	243	7
0	3	8	920	828	11	0	7	12	120	110	21	1	0	4	1365	1313	7	1	2	7	352	432	8
0	3	9	729	696	12	0	8	0	231	221	8	1	0	6	706	677	10	1	2	8	159	186	10
0	3	11	262	247	8	0	8	1	429	385	8	1	0	8	1053	1083	10	1	2	11	261	333	8
0	3	16	172	170	15	0	8	2	403	373	7	1	0	10	1002	995	12	1	2	13	101	141	22
0	4	0	827	710	9	0	8	4	212	218	10	1	0	12	573	571	11	1	2	15	306	327	9
0	4	1	1144	1093	8	0	8	5	183	213	11	1	0	16	359	352	8	1	2	17	138	150	19
0	4	2	194	131	6	0	8	7	651	652	12	1	0	18	115	123	24	1	3	-18	296	263	10
0	4	3	233	191	5	0	8	8	435	449	8	1	1	-15	290	272	9	1	3	-17	228	238	12
0	4	4	310	328	5	0	8	9	354	325	8	1	1	-10	168	145	9	1	3	-16	188	158	13

TABLE 6 Structure Factor Tables of Fobs and Fcalc for *gem*-$N_3P_3Cl_4(OSiPh_3)_2$ (**2**) (continued)

Values of 10*Fobs and 10*Fcalc Page 2

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
1	3	-15	263	247	10	1	4	12	142	138	15	1	7	-12	163	153	15	1	9	-2	328	360	7
1	3	-13	637	604	12	1	4	13	160	143	15	1	7	-11	226	203	11	1	9	-1	271	243	8
1	3	-12	297	292	7	1	4	14	547	621	9	1	7	-10	128	136	17	1	9	0	165	153	12
1	3	-11	356	348	6	1	4	15	191	153	14	1	7	-9	173	180	12	1	9	1	222	201	9
1	3	-10	674	638	12	1	4	16	389	403	9	1	7	-8	223	220	9	1	9	3	235	233	9
1	3	-9	628	600	10	1	5	-17	292	257	10	1	7	-7	604	623	10	1	9	5	468	499	7
1	3	-8	454	418	8	1	5	-16	240	230	11	1	7	-6	357	333	6	1	9	6	475	449	9
1	3	-7	82	63	14	1	5	-14	271	259	10	1	7	-5	234	230	8	1	9	7	406	390	7
1	3	-6	813	750	10	1	5	-13	242	229	10	1	7	-2	499	518	8	1	9	8	115	129	21
1	3	-5	1120	1055	9	1	5	-12	207	194	10	1	7	-1	323	309	5	1	9	9	301	283	9
1	3	-4	1153	1074	8	1	5	-11	229	216	9	1	7	2	789	784	12	1	9	11	290	304	10
1	3	-3	456	531	8	1	5	-7	353	324	6	1	7	3	157	158	10	1	9	12	116	89	22
1	3	-2	701	710	9	1	5	-6	1020	1053	11	1	7	4	852	894	13	1	9	13	165	174	17
1	3	-1	582	545	9	1	5	-5	369	323	6	1	7	5	244	225	8	1	10	-11	120	90	20
1	3	0	1374	1323	7	1	5	-3	194	194	7	1	7	6	151	148	12	1	10	-10	98	43	24
1	3	1	1292	1219	7	1	5	-2	1133	1144	10	1	7	7	178	192	11	1	10	-3	403	405	7
1	3	2	1012	919	8	1	5	0	357	359	6	1	7	8	226	215	10	1	10	-1	177	189	13
1	3	3	228	256	6	1	5	2	1143	1103	10	1	7	9	169	183	13	1	10	0	152	132	14
1	3	4	1365	1301	9	1	5	3	492	458	8	1	7	10	267	273	10	1	10	1	291	309	8
1	3	5	226	215	6	1	5	4	649	644	11	1	7	11	239	237	11	1	10	3	177	176	13
1	3	6	288	310	5	1	5	5	296	315	6	1	7	13	218	225	12	1	10	4	232	201	11
1	3	8	463	520	9	1	5	6	150	136	10	1	7	14	141	170	20	1	10	9	314	311	9
1	3	9	549	651	10	1	5	8	464	509	8	1	7	15	226	233	13	1	10	10	204	177	13
1	3	10	285	300	7	1	5	10	97	132	21	1	8	-12	137	150	18	1	11	-10	150	145	18
1	3	11	220	273	10	1	5	11	109	105	19	1	8	-11	294	296	9	1	11	-3	155	153	16
1	3	12	159	215	13	1	5	16	248	239	12	1	8	-10	449	451	9	1	11	-2	168	149	15
1	3	13	119	138	19	1	5	17	108	71	26	1	8	-9	153	171	15	1	11	-1	113	95	20
1	4	-17	156	155	17	1	6	-17	142	148	20	1	8	-6	391	415	8	1	11	1	213	210	12
1	4	-16	207	186	12	1	6	-14	178	166	14	1	8	-5	233	207	8	1	11	2	174	166	14
1	4	-14	209	231	12	1	6	-13	183	169	13	1	8	-4	290	278	7	1	11	5	135	140	10
1	4	-13	128	131	16	1	6	-9	252	258	8	1	8	-3	749	737	12	1	12	-6	171	167	15
1	4	-12	165	170	12	1	6	-8	404	409	8	1	8	-2	248	241	7	1	12	-3	212	226	12
1	4	-10	391	377	7	1	6	-5	83	85	17	1	8	-1	677	684	12	1	12	-1	130	105	19
1	4	-9	339	316	6	1	6	-4	115	126	13	1	8	0	431	455	7	1	12	0	125	101	19
1	4	-8	671	719	11	1	6	-3	451	479	9	1	8	1	585	593	10	1	12	1	229	223	12
1	4	-7	209	169	7	1	6	-2	161	142	9	1	8	3	250	232	8	1	12	4	255	238	11
1	4	-6	519	472	9	1	6	-1	986	939	11	1	8	4	508	514	9	1	12	7	159	170	18
1	4	-4	193	205	6	1	6	0	274	287	6	1	8	6	182	190	11	1	12	8	331	311	10
1	4	-3	951	938	9	1	6	1	317	310	6	1	8	7	552	573	9	1	13	2	204	217	13
1	4	-2	757	668	9	1	6	2	242	273	7	1	8	8	317	318	8	1	13	3	208	199	13
1	4	-1	1777	1728	8	1	6	3	1038	1053	11	1	8	9	648	656	11	2	0	-18	139	130	16
1	4	1	373	331	7	1	6	4	364	349	7	1	9	-11	360	358	9	2	0	-16	236	231	10
1	4	3	276	231	5	1	6	5	497	459	9	1	9	-10	105	124	23	2	0	-14	344	345	7
1	4	4	880	830	10	1	6	6	321	298	6	1	9	-9	176	184	14	2	0	-12	112	109	15
1	4	5	213	247	7	1	6	7	149	131	12	1	9	-8	417	401	7	2	0	-10	302	287	6
1	4	6	1258	1213	10	1	6	8	109	66	17	1	9	-7	117	86	19	2	0	-8	370	268	6
1	4	7	313	333	6	1	6	9	97	88	21	1	9	-6	304	303	8	2	0	-6	712	684	9
1	4	8	84	66	19	1	6	12	358	351	8	1	9	-5	123	118	17	2	0	-4	1869	1968	7
1	4	9	219	245	8	1	7	-16	135	121	19	1	9	-4	287	314	8	2	0	-2	1460	1529	6
1	4	10	291	357	8	1	7	-15	221	235	13	1	9	-3	311	292	8	2	0	0	740	690	6

```
Values of 10*Fobs and 10*Fcalc                                                                                  Page  3

H  K  L   Fobs  Fcalc  SigF      H  K  L   Fobs  Fcalc  SigF      H  K  L   Fobs  Fcalc  SigF      H  K  L   Fobs  Fcalc  SigF
-  -  -   ----  -----  ----      -  -  -   ----  -----  ----      -  -  -   ----  -----  ----      -  -  -   ----  -----  ----

2  0  2    312    334     5      2  2  1   2210   2114     6      2  4 -7     80    132    17      2  6  0    659    611    11
2  0  4    889    909     8      2  2  2    498    535     8      2  4 -6    656    563    11      2  6  1    204    233     8
2  0  8    306    332     6      2  2  3    591    551    10      2  4 -5    481    445     9      2  6  2    438    447     7
2  0 12    141    132    15      2  2  4   1294   1360     8      2  4 -3    756    758    10      2  6  3    388    369     6
2  0 14    266    263    10      2  2  5    237    245     7      2  4 -2    550    470     9      2  6  5     88     58    17
2  0 18    312    290    10      2  2  6    313    383     7      2  4 -1    756    752     9      2  6  7    507    523     8
2  1-18    152    130    16      2  2  7    309    335     8      2  4  0   1722   1598     8      2  6  8    199    170    10
2  1-17    150    160    15      2  2  9    592    733    11      2  4  1    889    816     9      2  6  9    337    323     7
2  1-16    124    114    18      2  2 10    318    372     6      2  4  2    571    597     9      2  6 10    175    185    13
2  1-14    475    496     9      2  2 11    215    248     9      2  4  3    150    136     8      2  6 11    369    385     8
2  1-13    293    290     7      2  2 13    363    409     8      2  4  4    404    399     7      2  6 13    334    376     9
2  1-12    748    730    12      2  2 15    210    216    13      2  4  5    512    488    10      2  7-15    201    176    13
2  1-10    167    162     9      2  2 16    203    217    13      2  4  6    866    843    11      2  7-14     97     74    24
2  1 -9    191    215     7      2  3-16    174    155    14      2  4  7    353    403     8      2  7-13    187    189    13
2  1 -8    157    165     8      2  3-15    441    457     7      2  4  9    191    215    10      2  7-12    265    245    10
2  1 -7   1193   1128     9      2  3-13    382    331     6      2  4 13    127    144    20      2  7-11    359    376     8
2  1 -5    778    757     9      2  3-12    122    140    15      2  4 14    207    208    13      2  7 -8    700    688    11
2  1 -4    719    657     8      2  3-11    118    111    13      2  4 15    210    255    13      2  7 -7    275    289     7
2  1 -3    113    141     8      2  3-10    420    385     7      2  5-17    334    283     9      2  7 -6    293    289     7
2  1 -2    840    780     6      2  3 -9    529    535    10      2  5-13    219    224    11      2  7 -5    228    243     8
2  1 -1    353    371     6      2  3 -8    360    345     7      2  5-10    449    451     8      2  7 -4    438    467     9
2  1  0    590    596     6      2  3 -7    356    325     7      2  5 -9    115    112    14      2  7 -3    186    173     9
2  1  1   1418   1483     6      2  3 -6    347    309     6      2  5 -8   1042   1000    12      2  7 -1    728    730    12
2  1  2   1637   1646     6      2  3 -5    354    332     7      2  5 -7    128    148    11      2  7  0    534    496    10
2  1  4     71     24    16      2  3 -4    134     84     9      2  5 -6    792    796    11      2  7  1   1231   1205    11
2  1  5   1151   1216     9      2  3 -3    357    403     7      2  5 -5    378    369     7      2  7  2    439    419     8
2  1  6    268    287     6      2  3 -2    254    317     5      2  5 -4    275    291     5      2  7  3    739    690    12
2  1  7    104    108    12      2  3 -1     77     46    14      2  5 -3    196    222     7      2  7  4     88    113    19
2  1  8   1130   1132    11      2  3  0    139    130     8      2  5 -2    639    607    11      2  7  7    314    339     7
2  1  9    439    486     9      2  3  1    408    330     7      2  5 -1    654    626    10      2  7  8    134    147    15
2  1 10    570    551    10      2  3  2    646    605     9      2  5  0    397    441     7      2  7  9    445    464     8
2  1 11    312    312     7      2  3  3    368    396     8      2  5  2    396    347     6      2  7 10    127     83    18
2  1 12    366    360     7      2  3  4    325    318     6      2  5  3    364    385     6      2  7 11    187    198    14
2  1 13    127    136    18      2  3  5    232    212     6      2  5  4    719    747    11      2  7 12    314    333    10
2  1 14    115     92    22      2  3  7    535    669    10      2  5  5    355    305     5      2  8-15    144    117    18
2  1 16    183    193    14      2  3  8    167    258    10      2  5  8    287    266     7      2  8-14    127    129    20
2  2-18    144    146    17      2  3  9    229    299     8      2  5 10    390    405     7      2  8-13    290    279    10
2  2-15    163    196    14      2  3 11    160    190    12      2  5 12    622    627    11      2  8-12    153    138    16
2  2-13    511    498     9      2  3 12    149    171    14      2  5 14    256    262    11      2  8-11    300    291    10
2  2-12    413    412     8      2  3 14    253    271    11      2  5 15    134    174    20      2  8-10    136    133    17
2  2-11    126    119    13      2  3 15    187    148    14      2  5 17    161    179    19      2  8 -8    132    112    15
2  2-10    299    317     6      2  4-18    264    253    11      2  6-11    115    137    18      2  8 -7    139    149    15
2  2 -9   1100   1057    11      2  4-16    211    190    12      2  6 -8    482    479     8      2  8 -6    380    417     8
2  2 -8    583    544    10      2  4-15    138     81    16      2  6 -7     85     56    18      2  8 -5    438    457     8
2  2 -7    301    304     5      2  4-14    208    197    12      2  6 -6    434    427     8      2  8 -4    194    201    10
2  2 -6    622    566    10      2  4-13    596    576    10      2  6 -5    120    162    12      2  8 -3    401    376     7
2  2 -4   1010    931     8      2  4-11    477    484     8      2  6 -4    230    254     7      2  8 -2    292    305     7
2  2 -3   1083    995     7      2  4-10    144    155    12      2  6 -3    220    193     7      2  8 -1    436    399     8
2  2 -2    704    658     8      2  4 -9    422    422     8      2  6 -2    734    665    11      2  8  2    434    453     7
2  2 -1   1025   1046     7      2  4 -8    138    145    11      2  6 -1    149     95     9      2  8  3    582    587    10
```

TABLE 6 Structure Factor Tables of Fobs and Fcalc for *gem*-$N_3P_3Cl_4(OSiPh_3)_2$ (**2**) (continued)

Values of 10*Fobs and 10*Fcalc

Page 4

H	K	L	Fobs	Fcalc	SigF
2	8	4	467	453	7
2	8	5	705	715	12
2	8	6	277	276	9
2	8	7	274	254	9
2	8	11	213	208	12
2	8	12	222	204	12
2	8	13	162	147	17
2	8	14	268	262	12
2	9	-14	206	197	13
2	9	-10	129	134	18
2	9	-8	188	182	13
2	9	-7	162	151	14
2	9	-6	362	341	7
2	9	-5	115	69	17
2	9	-4	515	528	10
2	9	-3	371	381	7
2	9	-2	109	149	17
2	9	-1	324	315	7
2	9	0	335	367	7
2	9	1	230	246	9
2	9	2	208	195	10
2	9	5	453	471	10
2	9	6	178	166	14
2	9	7	470	500	8
2	9	8	153	162	17
2	9	13	220	227	14
2	10	-11	125	96	19
2	10	-9	196	173	13
2	10	-7	168	153	15
2	10	-5	262	259	10
2	10	-3	312	303	8
2	10	-2	270	288	9
2	10	-1	105	134	22
2	10	0	264	303	9
2	10	2	220	216	11
2	10	3	313	331	9
2	10	4	121	85	19
2	10	5	205	209	12
2	10	10	184	148	15
2	11	-8	215	188	12
2	11	-4	296	237	9
2	11	-3	206	220	13
2	11	0	168	150	14
2	11	3	317	319	9
2	11	7	163	138	16
2	11	9	136	141	21
2	11	10	120	140	24
2	12	-8	171	167	16
2	12	-7	173	157	16
2	12	-4	104	36	24
2	12	-2	264	262	11
2	12	2	183	204	15
2	12	3	161	128	16
2	12	6	115	145	24
2	12	7	159	129	17
2	13	-1	154	170	18
2	13	0	110	87	24
2	13	1	370	333	9
2	13	2	147	176	20
2	13	3	283	263	12
3	0	-16	102	86	21
3	0	-14	877	937	15
3	0	-12	987	933	13
3	0	-10	340	342	6
3	0	-8	411	430	7
3	0	-6	337	336	5
3	0	-4	187	146	6
3	0	-2	1030	1059	7
3	0	0	249	230	6
3	0	2	903	908	8
3	0	4	252	320	5
3	0	6	1254	1257	10
3	0	8	847	854	12
3	0	10	216	210	9
3	0	12	549	512	10
3	0	14	689	715	12
3	0	16	214	216	13
3	1	-18	178	145	13
3	1	-17	150	153	15
3	1	-16	156	149	15
3	1	-15	135	116	17
3	1	-14	644	651	11
3	1	-13	134	115	14
3	1	-12	370	352	7
3	1	-11	249	266	7
3	1	-10	619	678	10
3	1	-9	854	943	11
3	1	-7	440	524	8
3	1	-6	377	392	6
3	1	-5	645	618	9
3	1	-4	2525	2479	7
3	1	-3	2700	2554	7
3	1	-2	377	427	6
3	1	-1	134	118	7
3	1	0	1030	1018	7
3	1	1	1225	1107	7
3	1	2	696	709	8
3	1	3	567	571	8
3	1	5	967	962	9
3	1	6	752	861	10
3	1	8	866	898	12
3	1	9	475	505	9
3	1	10	107	123	16
3	1	11	558	610	9
3	1	12	138	141	16
3	1	13	160	166	15
3	1	15	174	164	15
3	1	18	184	174	16
3	2	-16	135	141	17
3	2	-15	508	490	9
3	2	-13	627	592	10
3	2	-12	264	257	8
3	2	-11	355	299	7
3	2	-10	210	194	8
3	2	-8	841	843	11
3	2	-7	504	435	9
3	2	-6	432	425	7
3	2	-5	563	485	9
3	2	-4	277	275	5
3	2	-3	797	761	8
3	2	-2	307	363	5
3	2	-1	1692	1680	7
3	2	0	1824	1790	7
3	2	1	1216	1158	7
3	2	2	949	878	8
3	2	3	769	703	9
3	2	4	954	1032	9
3	2	5	347	390	7
3	2	6	340	410	6
3	2	7	124	72	12
3	2	8	285	333	6
3	2	10	344	396	7
3	2	13	223	241	12
3	2	14	215	198	12
3	2	17	213	227	14
3	3	-18	259	228	11
3	3	-15	295	258	9
3	3	-14	300	295	9
3	3	-13	611	598	10
3	3	-11	317	312	7
3	3	-10	409	426	7
3	3	-9	756	764	12
3	3	-8	194	196	8
3	3	-7	181	191	8
3	3	-6	123	150	10
3	3	-5	1105	1076	9
3	3	-3	300	292	6
3	3	-2	715	682	9
3	3	-1	1318	1214	8
3	3	0	74	54	15
3	3	1	764	664	9
3	3	2	733	605	9
3	3	3	691	602	10
3	3	5	288	261	5
3	3	6	166	147	9
3	3	7	838	864	12
3	3	8	326	389	6
3	3	9	243	298	8
3	3	12	158	189	15
3	3	13	289	353	9
3	3	15	140	187	19
3	3	16	205	217	14
3	4	-15	345	363	8
3	4	-14	248	231	10
3	4	-13	353	345	8
3	4	-12	494	499	7
3	4	-10	655	633	12
3	4	-9	438	471	8
3	4	-8	618	619	10
3	4	-7	425	410	7
3	4	-6	550	458	8
3	4	-5	486	442	10
3	4	-4	139	100	9
3	4	-2	432	383	7
3	4	-1	174	169	7
3	4	0	304	285	5
3	4	1	158	164	8
3	4	2	697	651	10
3	4	3	188	209	7
3	4	4	619	536	10
3	4	5	218	233	7
3	4	6	342	357	6
3	4	7	475	534	10
3	4	9	106	118	17
3	4	10	310	337	8
3	4	11	337	368	8
3	4	12	419	439	8
3	4	14	195	218	14
3	4	17	140	158	22
3	5	-16	156	168	17
3	5	-15	147	122	16
3	5	-13	185	169	13
3	5	-12	267	243	9
3	5	-11	308	268	8
3	5	-10	459	429	8
3	5	-9	508	489	9
3	5	-8	164	146	10
3	5	-7	195	187	8
3	5	-6	128	128	12
3	5	-5	1095	1054	11

Values of 10*Fobs and 10*Fcalc Page 5

H	K	L	Fobs	Fcalc	SigF
3	5	-4	634	633	11
3	5	-1	433	445	8
3	5	0	1384	1353	10
3	5	1	77	23	16
3	5	2	580	623	10
3	5	3	872	889	11
3	5	4	385	346	7
3	5	8	370	422	7
3	5	9	129	108	16
3	5	10	174	187	13
3	5	13	190	206	14
3	5	14	119	147	23
3	5	15	107	119	25
3	6	-12	123	137	19
3	6	-10	416	406	7
3	6	-9	512	516	9
3	6	-7	89	104	18
3	6	-6	515	482	9
3	6	-5	457	463	8
3	6	-4	706	738	12
3	6	-3	621	617	11
3	6	-2	529	494	8
3	6	-1	210	212	7
3	6	0	326	318	6
3	6	1	700	726	12
3	6	2	596	543	10
3	6	3	949	920	12
3	6	4	345	360	7
3	6	5	377	383	7
3	6	7	439	433	9
3	6	8	100	124	20
3	6	9	134	169	16
3	6	10	215	235	11
3	6	12	215	228	12
3	6	15	186	208	15
3	7	-14	143	135	17
3	7	-13	152	163	17
3	7	-11	304	294	9
3	7	-10	269	221	9
3	7	-9	423	442	8
3	7	-8	315	315	7
3	7	-6	431	456	8
3	7	-4	543	542	9
3	7	-3	181	152	9
3	7	-2	194	162	9
3	7	-1	228	245	7
3	7	1	116	149	14
3	7	2	735	693	13
3	7	3	279	275	7
3	7	5	252	254	8
3	7	6	466	434	8
3	7	7	235	230	10
3	7	8	102	115	20
3	7	11	252	260	11
3	8	-9	194	201	12
3	8	-8	222	230	11
3	8	-7	300	256	8
3	8	-6	289	257	8
3	8	-5	528	527	9
3	8	-4	574	551	9
3	8	-3	373	409	7
3	8	-2	229	230	8
3	8	-1	521	529	10
3	8	0	309	321	7
3	8	1	343	333	7
3	8	2	339	340	7
3	8	3	326	323	7
3	8	5	603	617	11
3	8	6	290	311	9
3	8	7	273	306	9
3	8	9	187	185	14
3	8	10	193	216	14
3	8	12	257	248	11
3	8	14	205	155	15
3	9	-11	174	161	15
3	9	-6	163	181	14
3	9	-5	518	519	9
3	9	-4	192	184	11
3	9	-3	233	264	9
3	9	-2	116	121	17
3	9	-1	183	177	11
3	9	0	340	323	7
3	9	2	244	247	9
3	9	3	173	165	12
3	9	4	307	330	9
3	9	5	153	141	16
3	9	6	410	435	7
3	9	7	446	444	8
3	9	8	114	58	21
3	9	9	364	373	8
3	9	11	172	157	16
3	9	12	121	96	25
3	10	-6	179	122	13
3	10	-5	299	316	9
3	10	-3	174	176	14
3	10	-2	117	162	20
3	10	-1	255	259	10
3	10	3	262	277	10
3	10	5	253	242	11
3	10	11	193	190	15
3	11	-9	122	139	23
3	11	-6	150	144	17
3	11	-5	260	273	10
3	11	-4	108	80	22
3	11	-3	121	149	20
3	11	-2	155	128	15
3	11	-1	331	342	9
3	11	1	228	224	11
3	11	2	147	155	17
3	11	5	275	249	10
3	11	9	218	199	14
3	12	-6	185	193	15
3	12	-4	232	221	12
3	12	-3	210	196	13
3	12	3	238	240	12
3	12	4	129	109	21
3	12	5	343	342	9
3	12	6	174	153	16
3	12	7	128	121	22
3	13	0	156	168	18
4	0	-16	404	393	7
4	0	-14	858	912	13
4	0	-12	136	167	13
4	0	-10	556	604	8
4	0	-8	1164	1210	10
4	0	-6	758	732	10
4	0	-2	2302	2258	7
4	0	0	624	694	9
4	0	2	761	741	9
4	0	4	704	713	10
4	0	6	1066	1009	10
4	0	8	600	612	11
4	0	10	118	114	15
4	0	12	591	616	9
4	0	16	507	462	9
4	1	-15	123	180	19
4	1	-14	359	358	7
4	1	-13	485	457	8
4	1	-11	251	254	7
4	1	-10	306	300	6
4	1	-9	78	67	17
4	1	-8	177	212	8
4	1	-6	108	123	11
4	1	-5	709	698	9
4	1	-4	1240	1160	8
4	1	-3	81	133	14
4	1	-1	1005	1024	8
4	1	0	1305	1305	7
4	1	1	2006	2081	8
4	1	2	76	93	15
4	1	3	345	250	6
4	1	4	325	329	6
4	1	5	414	438	7
4	1	6	902	872	11
4	1	7	137	95	11
4	1	8	221	241	8
4	1	9	217	245	9
4	1	10	245	239	9
4	1	11	141	162	15
4	1	12	591	588	11
4	1	14	325	323	9
4	1	15	202	182	13
4	1	16	223	243	13
4	1	17	107	96	26
4	2	-18	186	173	14
4	2	-17	232	222	11
4	2	-16	133	117	17
4	2	-15	190	204	12
4	2	-14	218	207	10
4	2	-13	273	263	8
4	2	-11	330	311	6
4	2	-9	505	497	8
4	2	-8	259	249	6
4	2	-6	465	412	8
4	2	-5	126	124	9
4	2	-4	272	242	6
4	2	-3	1321	1248	8
4	2	-1	1649	1648	8
4	2	0	1446	1382	8
4	2	2	671	646	9
4	2	3	1709	1608	9
4	2	4	1022	952	10
4	2	5	483	517	10
4	2	6	246	306	6
4	2	7	771	817	12
4	2	8	443	466	8
4	2	9	455	543	9
4	2	10	420	420	9
4	2	12	380	369	7
4	2	13	199	212	13
4	2	14	132	101	19
4	2	15	152	139	17
4	2	17	133	110	21
4	3	-17	166	176	15
4	3	-16	117	85	20
4	3	-15	749	736	12
4	3	-14	243	237	10
4	3	-13	472	454	9
4	3	-12	381	368	7
4	3	-10	314	330	6

TABLE 6 Structure Factor Tables of Fobs and Fcalc for *gem*-$N_3P_3Cl_4(OSiPh_3)_2$ (**2**) (continued)

Values of 10*Fobs and 10*Fcalc

Page 6

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
4	3	-9	270	239	6	4	5	-13	319	295	8	4	7	-10	561	562	9	4	9	7	99	61	24
4	3	-8	450	428	8	4	5	-12	587	597	11	4	7	-9	142	166	15	4	9	8	298	292	9
4	3	-7	808	777	11	4	5	-11	426	414	7	4	7	-8	406	404	8	4	9	10	173	182	16
4	3	-6	599	572	10	4	5	-10	240	270	9	4	7	-7	187	211	10	4	9	12	140	52	19
4	3	-5	1224	1204	9	4	5	-9	402	355	6	4	7	-4	629	649	11	4	10	-11	178	159	18
4	3	-4	199	171	6	4	5	-8	317	312	7	4	7	-3	356	299	6	4	10	-9	115	132	21
4	3	-3	199	212	6	4	5	-7	216	212	8	4	7	-2	563	500	9	4	10	-7	238	217	10
4	3	-2	594	686	10	4	5	-6	273	281	6	4	7	-1	447	431	7	4	10	-6	214	217	12
4	3	-1	361	335	7	4	5	-5	693	683	12	4	7	0	358	343	7	4	10	-5	212	201	12
4	3	0	560	597	9	4	5	-4	164	186	9	4	7	4	352	380	8	4	10	-4	192	198	13
4	3	1	175	130	7	4	5	-3	353	355	5	4	7	5	142	143	14	4	10	-2	167	159	14
4	3	2	123	56	9	4	5	-2	293	303	6	4	7	6	189	205	11	4	10	-1	174	140	13
4	3	3	263	198	5	4	5	0	786	802	11	4	7	7	158	170	14	4	10	0	310	336	9
4	3	4	116	108	11	4	5	1	176	131	8	4	7	8	230	247	10	4	10	1	367	351	7
4	3	5	715	673	11	4	5	3	165	133	9	4	7	9	180	188	14	4	10	2	323	328	8
4	3	6	993	975	11	4	5	4	106	114	14	4	7	10	235	244	11	4	10	5	232	221	11
4	3	7	518	559	10	4	5	5	544	513	9	4	7	14	183	166	16	4	10	7	186	171	13
4	3	10	378	417	8	4	5	7	444	441	8	4	8	-13	252	255	11	4	10	11	161	176	17
4	3	13	124	158	20	4	5	8	463	446	9	4	8	-12	209	175	12	4	11	-8	249	238	11
4	3	15	220	208	13	4	5	10	505	531	10	4	8	-11	331	355	9	4	11	-3	189	205	12
4	3	17	256	245	12	4	5	11	203	180	12	4	8	-10	232	228	11	4	11	-2	167	168	14
4	4	-18	120	125	22	4	5	13	246	272	11	4	8	-9	210	217	12	4	11	-1	109	111	22
4	4	-15	304	316	9	4	5	15	199	201	14	4	8	-7	266	238	9	4	11	1	118	106	19
4	4	-13	243	223	10	4	6	-15	257	218	11	4	8	-6	125	126	16	4	11	3	255	276	11
4	4	-12	145	163	14	4	6	-14	228	232	11	4	8	-5	235	236	9	4	11	4	264	253	10
4	4	-11	237	236	8	4	6	-11	352	357	7	4	8	-4	134	148	14	4	11	5	496	485	9
4	4	-9	490	445	9	4	6	-9	490	477	8	4	8	-3	321	338	7	4	12	-6	137	89	17
4	4	-7	377	338	6	4	6	-7	150	136	11	4	8	-2	408	412	8	4	12	-5	145	164	17
4	4	-6	221	223	7	4	6	-6	784	802	13	4	8	2	189	210	11	4	12	-4	135	93	18
4	4	-5	211	217	7	4	6	-5	566	550	9	4	8	3	357	323	7	4	12	-1	198	209	13
4	4	-4	380	360	6	4	6	-4	702	681	11	4	8	4	733	739	12	4	12	4	151	175	19
4	4	-3	122	142	10	4	6	-3	335	336	6	4	8	5	426	418	7	4	12	6	121	107	23
4	4	-2	240	222	6	4	6	-2	179	136	9	4	8	6	377	409	7	4	13	-1	358	377	10
4	4	-1	174	161	7	4	6	-1	163	154	9	4	8	7	153	133	15	5	0	-18	131	97	18
4	4	0	837	810	10	4	6	0	105	99	14	4	8	8	379	352	8	5	0	-16	119	141	19
4	4	1	365	283	7	4	6	1	819	831	12	4	8	9	231	215	11	5	0	-14	444	485	8
4	4	2	343	351	7	4	6	2	146	136	10	4	8	13	244	198	12	5	0	-12	293	319	7
4	4	3	444	428	8	4	6	3	931	937	12	4	9	-14	172	180	16	5	0	-10	483	488	8
4	4	4	414	351	8	4	6	4	685	656	12	4	9	-13	140	135	19	5	0	-8	429	444	7
4	4	5	232	228	7	4	6	5	545	538	10	4	9	-9	108	133	22	5	0	-4	1309	1333	8
4	4	6	606	553	11	4	6	6	158	182	12	4	9	-8	323	306	9	5	0	0	697	717	9
4	4	7	235	236	8	4	6	7	188	185	11	4	9	-7	263	271	10	5	0	2	446	433	7
4	4	10	117	100	17	4	6	8	208	187	10	4	9	-6	266	259	9	5	0	4	718	726	10
4	4	11	339	335	8	4	6	9	482	462	7	4	9	-5	151	158	14	5	0	8	264	246	7
4	4	13	395	406	8	4	6	10	245	252	10	4	9	-4	124	140	17	5	0	12	610	605	10
4	4	14	179	194	15	4	6	12	153	157	16	4	9	-2	166	189	12	5	0	14	395	405	8
4	4	15	111	116	24	4	6	13	164	166	15	4	9	3	638	632	11	5	0	16	245	246	12
4	4	16	192	185	15	4	6	14	226	258	13	4	9	4	256	269	10	5	1	-18	185	199	14
4	5	-17	252	217	11	4	6	15	223	202	13	4	9	5	848	854	14	5	1	-16	250	242	10
4	5	-16	147	152	17	4	7	-11	151	148	16	4	9	6	403	392	7	5	1	-15	128	120	18

Values of 10*Fobs and 10*Fcalc

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H	K	L	Fobs	Fcalc	SigF
5	1	-14	161	166	14
5	1	-13	507	542	8
5	1	-11	526	550	9
5	1	-9	267	285	6
5	1	-8	313	299	5
5	1	-6	1029	976	10
5	1	-5	1577	1468	9
5	1	-4	1106	956	9
5	1	-3	819	736	9
5	1	-2	200	184	6
5	1	-1	1158	1148	8
5	1	0	913	886	9
5	1	1	673	694	10
5	1	2	777	820	10
5	1	3	700	615	10
5	1	4	556	520	10
5	1	5	202	192	7
5	1	6	368	385	6
5	1	7	1073	1071	12
5	1	8	127	117	13
5	1	9	880	882	14
5	1	10	336	326	7
5	1	11	274	304	9
5	1	12	385	375	8
5	1	13	244	241	11
5	2	-17	233	237	12
5	2	-16	179	179	13
5	2	-15	217	242	12
5	2	-14	105	97	22
5	2	-13	99	45	19
5	2	-11	90	109	19
5	2	-10	463	456	7
5	2	-8	450	479	7
5	2	-7	210	215	7
5	2	-5	97	58	12
5	2	-4	268	283	5
5	2	-3	151	132	8
5	2	-2	159	157	7
5	2	-1	717	601	9
5	2	0	923	945	9
5	2	1	1030	964	9
5	2	2	617	596	10
5	2	3	404	407	7
5	2	4	499	505	9
5	2	5	262	185	6
5	2	7	124	110	13
5	2	8	380	397	8
5	2	9	178	180	11
5	2	10	144	154	15
5	2	11	249	243	10
5	2	12	147	152	17
5	2	13	209	213	13
5	2	16	218	213	14
5	3	-18	176	149	16
5	3	-14	577	568	9
5	3	-13	122	98	17
5	3	-12	517	524	9
5	3	-9	798	815	13
5	3	-8	434	378	8
5	3	-7	512	514	9
5	3	-6	259	204	6
5	3	-5	212	142	7
5	3	-4	191	191	7
5	3	-3	268	289	5
5	3	-2	313	270	5
5	3	-1	596	600	10
5	3	0	282	275	5
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5	3	2	590	579	10
5	3	3	583	532	10
5	3	4	331	303	5
5	3	5	215	246	8
5	3	6	165	126	9
5	3	7	160	130	11
5	3	8	233	184	8
5	3	9	115	138	17
5	3	12	312	328	9
5	3	13	257	255	11
5	3	14	360	366	9
5	3	15	135	100	20
5	3	16	149	141	19
5	4	-18	168	167	17
5	4	-15	196	180	13
5	4	-14	472	462	7
5	4	-12	733	723	14
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5	4	-10	302	313	7
5	4	-9	429	433	7
5	4	-8	188	172	9
5	4	-7	181	154	9
5	4	-6	596	543	11
5	4	-5	1312	1294	10
5	4	-4	154	156	9
5	4	-3	677	598	11
5	4	-2	621	679	11
5	4	-1	130	154	10
5	4	0	646	583	11
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5	4	2	219	212	7
5	4	3	153	145	10
5	4	4	241	244	7
5	4	5	286	283	7
5	4	6	391	371	7
5	4	7	257	259	8
5	4	8	631	625	11
5	4	9	125	150	17
5	4	10	442	418	8
5	4	11	162	170	16
5	4	12	112	107	23
5	4	14	140	142	20
5	5	-15	164	175	16
5	5	-12	117	109	19
5	5	-11	179	190	12
5	5	-10	141	132	14
5	5	-8	231	236	8
5	5	-7	296	297	6
5	5	-6	548	553	10
5	5	-5	590	575	10
5	5	-4	318	276	6
5	5	-3	487	448	8
5	5	-2	184	186	8
5	5	-1	615	605	11
5	5	0	477	534	8
5	5	1	302	300	6
5	5	2	398	406	7
5	5	3	415	440	7
5	5	4	125	97	13
5	5	5	589	571	11
5	5	6	466	492	8
5	5	8	234	227	10
5	5	9	306	313	8
5	5	11	123	66	20
5	5	12	161	182	16
5	6	-17	289	261	11
5	6	-15	129	100	19
5	6	-13	435	441	8
5	6	-11	522	517	10
5	6	-10	239	231	10
5	6	-9	239	241	9
5	6	-8	360	359	6
5	6	-7	228	231	8
5	6	-6	214	217	8
5	6	-5	97	85	16
5	6	-4	194	166	8
5	6	-3	573	571	10
5	6	-2	639	646	11
5	6	-1	523	508	9
5	6	1	495	473	8
5	6	2	165	160	10
5	6	3	629	620	11
5	6	4	364	393	7
5	6	5	403	407	7
5	6	6	312	303	7
5	6	7	435	415	9
5	6	8	231	221	10
5	6	9	300	305	9
5	6	10	100	92	24
5	6	11	279	257	10
5	6	12	161	188	16
5	6	13	221	204	13
5	6	14	130	104	21
5	6	15	216	201	14
5	7	-16	121	112	23
5	7	-15	129	96	20
5	7	-11	307	327	9
5	7	-10	146	171	16
5	7	-9	388	387	7
5	7	-6	375	360	6
5	7	-4	479	470	6
5	7	-3	300	306	7
5	7	-2	728	721	12
5	7	-1	210	228	9
5	7	0	485	517	9
5	7	2	556	578	9
5	7	3	127	104	14
5	7	4	809	800	14
5	7	5	312	311	8
5	7	7	363	378	7
5	7	8	121	118	19
5	7	9	397	391	8
5	7	12	121	94	21
5	7	13	130	76	21
5	8	-15	132	102	21
5	8	-11	236	230	11
5	8	-9	338	344	8
5	8	-8	116	125	20
5	8	-7	330	389	8
5	8	-6	237	256	9
5	8	-4	166	171	12
5	8	-3	165	155	12
5	8	-2	98	91	18
5	8	0	412	393	10
5	8	1	334	351	7
5	8	2	214	225	10
5	8	3	731	754	13
5	8	5	585	617	11
5	8	6	292	288	9
5	8	7	415	423	8
5	8	8	111	94	22
5	9	-12	212	199	13

TABLE 6 Structure Factor Tables of Fobs and Fcalc for *gem*-$N_3P_3Cl_4(OSiPh_3)_2$ (**2**) (continued)

Values of 10*Fobs and 10*Fcalc Page 8

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
5	9	-10	253	267	11	6	0	8	706	748	12	6	2	7	710	709	12	6	5	-13	311	302	9
5	9	-9	131	96	19	6	0	12	603	607	12	6	2	8	327	329	7	6	5	-12	478	489	8
5	9	-8	106	75	22	6	1	-18	246	249	12	6	2	9	255	252	9	6	5	-11	152	129	14
5	9	-7	610	578	9	6	1	-17	239	224	11	6	2	10	408	381	7	6	5	-10	586	568	11
5	9	-6	199	224	13	6	1	-13	342	359	7	6	2	12	100	95	24	6	5	-9	133	116	14
5	9	-4	164	161	13	6	1	-12	89	102	20	6	2	14	105	80	24	6	5	-8	272	236	7
5	9	0	160	164	13	6	1	-11	208	208	9	6	2	15	155	169	18	6	5	-7	212	225	9
5	9	2	256	244	9	6	1	-10	476	486	7	6	2	16	299	313	11	6	5	-6	439	456	8
5	9	3	145	162	16	6	1	-8	947	976	11	6	3	-16	167	150	14	6	5	-5	552	528	10
5	9	4	331	335	8	6	1	-7	261	205	6	6	3	-15	149	152	16	6	5	-3	438	460	8
5	9	5	181	181	14	6	1	-6	910	936	10	6	3	-13	379	367	7	6	5	-2	516	544	10
5	9	6	279	276	10	6	1	-5	702	758	10	6	3	-12	223	201	10	6	5	-1	645	667	11
5	9	7	149	137	16	6	1	-4	461	498	8	6	3	-9	404	408	8	6	5	0	194	203	8
5	9	8	166	150	15	6	1	-3	633	693	10	6	3	-8	374	367	6	6	5	1	468	464	8
5	10	-11	155	177	18	6	1	-2	264	288	5	6	3	-7	286	301	6	6	5	2	172	155	9
5	10	-9	200	183	13	6	1	-1	169	182	7	6	3	-6	254	238	6	6	5	3	196	197	9
5	10	-7	321	317	9	6	1	0	789	789	10	6	3	-5	458	420	9	6	5	5	265	278	7
5	10	-6	131	133	19	6	1	1	138	125	9	6	3	-4	393	430	6	6	5	6	798	786	12
5	10	-5	136	194	18	6	1	2	225	198	6	6	3	-3	322	300	5	6	5	7	260	254	9
5	10	-1	151	153	16	6	1	3	229	207	6	6	3	-2	77	92	16	6	5	8	588	611	11
5	10	0	115	111	20	6	1	4	86	96	15	6	3	-1	503	526	9	6	5	9	168	176	13
5	10	1	245	229	10	6	1	5	183	137	9	6	3	0	146	146	8	6	5	10	299	306	9
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5	10	7	113	115	22	6	1	7	331	360	6	6	3	4	201	219	8	6	6	-14	149	163	18
5	10	9	236	229	13	6	1	8	281	280	7	6	3	5	296	266	6	6	6	-12	255	261	11
5	10	10	171	160	17	6	1	11	296	305	9	6	3	6	302	281	7	6	6	-11	150	159	16
5	11	-5	177	195	15	6	1	14	164	172	16	6	3	7	321	324	7	6	6	-10	219	226	10
5	11	-3	152	145	16	6	1	15	177	146	16	6	3	11	344	326	8	6	6	-9	249	215	9
5	11	-1	228	231	11	6	1	16	178	169	16	6	3	13	143	152	18	6	6	-8	235	264	9
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5	12	-6	188	191	14	6	2	-11	233	212	8	6	4	-8	242	241	8	6	6	0	214	214	8
5	12	-1	118	72	21	6	2	-10	394	385	8	6	4	-7	643	640	12	6	6	1	315	310	6
5	12	2	150	164	19	6	2	-9	224	197	8	6	4	-6	181	194	9	6	6	2	140	101	12
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5	12	5	302	307	10	6	2	-7	924	973	11	6	4	-4	361	412	7	6	6	4	141	118	13
6	0	-18	161	183	15	6	2	-6	183	199	8	6	4	-3	242	206	6	6	6	5	199	215	10
6	0	-16	126	125	18	6	2	-5	133	109	9	6	4	-2	462	466	8	6	6	6	547	526	9
6	0	-14	306	326	8	6	2	-4	281	292	5	6	4	0	382	415	6	6	6	7	221	198	10
6	0	-10	250	232	7	6	2	-3	457	355	7	6	4	1	246	247	6	6	6	8	489	500	10
6	0	-8	1245	1192	11	6	2	-2	469	486	8	6	4	2	336	376	7	6	6	9	135	105	18
6	0	-6	1140	1092	10	6	2	-1	229	241	6	6	4	3	1117	1091	11	6	6	10	221	211	12
6	0	-4	438	393	8	6	2	0	362	336	6	6	4	4	534	527	10	6	6	11	234	225	12
6	0	-2	287	330	5	6	2	1	134	119	9	6	4	5	191	191	9	6	6	12	169	176	16
6	0	0	1254	1181	9	6	2	2	824	789	10	6	4	6	488	495	8	6	6	13	149	175	19
6	0	2	1543	1552	9	6	2	4	320	344	5	6	4	9	196	218	11	6	7	-12	135	145	18
6	0	4	895	867	11	6	2	5	762	756	12	6	4	10	110	103	20	6	7	-11	145	114	16
6	0	6	383	333	6	6	2	6	479	460	9	6	5	-15	326	352	9	6	7	-8	627	638	10

```
Values of 10*Fobs and 10*Fcalc                                                                                    Page  9

H  K  L   Fobs  Fcalc  SigF    H  K  L   Fobs  Fcalc  SigF    H  K  L   Fobs  Fcalc  SigF    H  K  L   Fobs  Fcalc  SigF
-  -  -   ----  -----  ----    -  -  -   ----  -----  ----    -  -  -   ----  -----  ----    -  -  -   ----  -----  ----

6  7 -7    153    148    13    6 10 -9    327    319     9    7  1  8    308    346     7    7  3  9    259    279     9
6  7 -6    165    186    12    6 10 -7    198    164    12    7  1 10    275    275     9    7  3 10    427    410     7
6  7 -5    358    322     6    6 10 -2    200    211    12    7  1 12    202    206    13    7  3 12    305    298     9
6  7 -3    270    267     8    6 10 -1    130    103    18    7  1 14    158    131    17    7  3 13    122    114    20
6  7 -2    187    203    10    6 10  0    199    234    13    7  2-15    306    297     9    7  4-17    228    213    12
6  7 -1    263    278     8    6 10  1    450    460     8    7  2-13    346    328     7    7  4-15    137    110    17
6  7  0    153    152    12    6 10  3    233    240    11    7  2-12    121    180    17    7  4-14    142    136    17
6  7  1    369    372     6    6 10  8    134    125    20    7  2-11    354    373     7    7  4-12    137    140    16
6  7  2    487    491     8    6 11 -3    246    257    11    7  2 -9    339    346     6    7  4-11    224    227     9
6  7  4    563    550    10    6 11 -1    148    152    17    7  2 -7    279    233     6    7  4 -9    790    797    14
6  7  5    222    222    10    6 11  1    155    178    17    7  2 -6    329    334     5    7  4 -7    221    210     8
6  7  6    270    250     9    6 11  2    135    170    19    7  2 -5    296    342     5    7  4 -6    633    641    11
6  7  7    625    628    11    6 11  3    296    319    10    7  2 -4    235    254     6    7  4 -5    597    629    11
6  7  8    478    473    10    6 11  4    177    164    16    7  2 -3    928    926    10    7  4 -4   1102   1049    11
6  7 12    332    303    10    6 11  5    230    237    13    7  2 -2    484    503     8    7  4 -3    201    229     8
6  8-15    238    209    13    6 12  0    129    124    22    7  2 -1    455    362     9    7  4 -2    123     99    11
6  8-13    224    243    13    6 12  2    215    222    14    7  2  1    365    364     6    7  4  0    622    663    11
6  8-12    205    211    13    6 12  3    109    124    25    7  2  2    430    445     7    7  4  2    259    225     6
6  8-11    126    148    20    7  0-18    354    316     9    7  2  3    148    153    10    7  4  3    629    631    11
6  8 -9    200    165    13    7  0-16    234    218    11    7  2  4    472    504     9    7  4  4    170    189    10
6  8 -8    239    247    10    7  0-14    408    412     8    7  2  5    429    490     9    7  4  5    266    263     7
6  8 -7    248    268    10    7  0 -8   1238   1222    11    7  2  6    154    134    11    7  4  6    360    379     7
6  8 -5    323    323     7    7  0 -6    682    603    10    7  2  7    535    548    10    7  4  7    492    445     9
6  8 -3    295    285     8    7  0 -4   1469   1329    10    7  2  9    120    152    17    7  4  8    369    378     7
6  8 -2    520    490     8    7  0 -2    480    490     8    7  2 10    218    211    11    7  4  9    105    118    21
6  8 -1    132    116    15    7  0  0    942    894    10    7  2 11    120    122    20    7  4 10    224    222    11
6  8  0    169    147    12    7  0  2    316    284     7    7  2 15    264    260    11    7  4 11    127    157    20
6  8  1    300    298     8    7  0  6    444    498     8    7  3-17    167    138    15    7  5-16    257    240    11
6  8  2    656    646    11    7  0  8    725    743    13    7  3-16    195    197    13    7  5-14    276    296    10
6  8  3    200    216    11    7  0 10    695    637    11    7  3-15    112    163    21    7  5-10    246    243     9
6  8  4    439    441     7    7  0 12    402    376     8    7  3-14    179    159    13    7  5 -9    229    206     9
6  8  7    140    160    18    7  1-18    145    150    18    7  3-13    389    385     7    7  5 -8    129    118    14
6  8  9    207    180    13    7  1-14    420    399     7    7  3-12    359    356     7    7  5 -6    321    286     6
6  8 11    228    237    13    7  1-13    266    258     9    7  3-11    295    270     7    7  5 -5    602    578    11
6  9-12    151    186    18    7  1-11    404    435     7    7  3 -8    245    257     7    7  5 -4    425    453     9
6  9-11    148    130    18    7  1-10     84    116    20    7  3 -7    485    465     8    7  5 -3    480    464     8
6  9-10    144    144    18    7  1 -9    146    131    11    7  3 -6    653    658    11    7  5 -2    184    202     9
6  9 -9    202    207    12    7  1 -7    441    464     8    7  3 -5    658    629    11    7  5  2    124    105    13
6  9 -8    230    234    12    7  1 -6    380    381     7    7  3 -4    501    478     9    7  5  4    154    160    11
6  9 -5    338    364     8    7  1 -5    216    218     7    7  3 -3    647    675    10    7  5  6    748    725    13
6  9 -3    183    162    12    7  1 -4    673    639    11    7  3 -2    441    398     8    7  5  7    330    322     8
6  9  0    195    158    12    7  1 -3    773    722    10    7  3 -1   1375   1343    10    7  5  8    451    446     8
6  9  1    421    411     8    7  1 -1    582    537     9    7  3  0    100     97    13    7  5  9    259    318    11
6  9  3    424    389     7    7  1  0    222    202     6    7  3  1    502    518     8    7  5 10    403    388     8
6  9  4    194    183    12    7  1  1    565    614    10    7  3  2    298    325     6    7  5 11    318    317     9
6  9  5    280    280    10    7  1  2    375    396     7    7  3  3    930    908    11    7  6-15    239    243    12
6  9  7    228    252    12    7  1  3    309    301     5    7  3  5     94    104    17    7  6-13    336    321     9
6  9  9    195    217    14    7  1  4    586    581    10    7  3  6    402    404     7    7  6-10    319    311     8
6  9 10    135    143    20    7  1  5    941    932    12    7  3  7    454    482    10    7  6 -8    397    386     7
6 10-11    117    149    24    7  1  7    188    187    10    7  3  8    306    296     7    7  6 -6    384    368     7
```

TABLE 6 Structure Factor Tables of Fobs and Fcalc for *gem*-$N_3P_3Cl_4(OSiPh_3)_2$ (**2**) (continued)

Values of 10*Fobs and 10*Fcalc

Page 10

H	K	L	Fobs	Fcalc	SigF
7	6	-5	161	144	11
7	6	-3	177	204	10
7	6	0	200	158	9
7	6	1	437	472	8
7	6	3	182	188	11
7	6	4	297	271	7
7	6	5	465	487	8
7	6	6	170	160	13
7	6	7	386	409	7
7	6	8	538	531	9
7	6	9	242	237	11
7	6	12	215	210	13
7	7	-14	181	211	15
7	7	-13	156	169	17
7	7	-12	195	174	13
7	7	-11	202	196	13
7	7	-10	363	357	8
7	7	-8	345	356	7
7	7	-6	156	161	13
7	7	-5	245	260	9
7	7	-4	212	215	9
7	7	-3	320	300	7
7	7	-2	332	338	7
7	7	-1	239	227	9
7	7	0	331	343	7
7	7	4	261	302	9
7	7	7	325	311	9
7	7	9	130	103	18
7	7	12	283	244	11
7	8	-14	166	134	16
7	8	-12	181	181	14
7	8	-11	275	282	10
7	8	-10	282	274	10
7	8	-8	396	431	8
7	8	-7	278	304	9
7	8	-6	160	217	14
7	8	-4	253	246	9
7	8	-2	110	109	19
7	8	-1	217	181	10
7	8	1	884	897	15
7	8	2	557	580	10
7	8	3	576	590	10
7	8	4	168	138	14
7	8	5	192	176	13
7	8	10	214	245	14
7	9	-13	117	106	25
7	9	-11	315	296	10
7	9	-10	122	85	21
7	9	-9	373	385	8
7	9	-7	192	199	13
7	9	-6	266	253	10
7	9	-4	250	225	10
7	9	-3	228	254	11
7	9	-2	115	78	20
7	9	0	164	175	14
7	9	1	276	256	10
7	9	3	207	218	13
7	9	5	150	87	16
7	9	6	130	154	20
7	10	-11	266	263	12
7	10	-9	166	194	18
7	10	-4	156	144	15
7	10	0	141	181	17
7	10	1	265	288	11
7	10	7	164	177	18
7	11	-9	364	359	9
7	11	-7	118	96	22
7	11	-4	104	102	24
7	11	-1	112	94	22
7	11	0	118	137	22
7	11	1	144	166	19
7	11	3	129	133	21
7	11	5	157	133	17
7	12	-5	120	127	23
7	12	-4	162	191	18
8	0	-18	200	212	14
8	0	-16	137	153	18
8	0	-14	323	319	8
8	0	-10	326	298	7
8	0	-8	270	283	7
8	0	-6	298	341	6
8	0	-4	253	273	6
8	0	-2	337	332	5
8	0	0	1344	1379	10
8	0	2	1180	1194	11
8	0	4	1091	1050	12
8	0	6	455	434	8
8	0	8	438	383	7
8	0	10	724	703	13
8	0	12	117	120	22
8	1	-17	106	102	22
8	1	-16	159	145	15
8	1	-15	125	153	20
8	1	-14	243	249	11
8	1	-12	339	340	7
8	1	-11	218	222	9
8	1	-10	105	50	16
8	1	-8	103	103	15
8	1	-7	541	497	8
8	1	-5	786	771	12
8	1	-3	796	775	11
8	1	-2	1078	1102	10
8	1	-1	492	502	9
8	1	0	134	165	11
8	1	2	163	144	9
8	1	3	82	119	18
8	1	4	169	190	9
8	1	5	591	596	10
8	1	7	168	142	12
8	1	8	267	286	9
8	1	9	490	509	10
8	1	10	272	281	10
8	1	12	284	289	10
8	1	15	189	160	15
8	2	-18	139	134	19
8	2	-16	307	288	9
8	2	-12	394	399	7
8	2	-11	142	113	13
8	2	-10	117	128	16
8	2	-8	482	433	9
8	2	-7	133	184	12
8	2	-6	601	588	11
8	2	-5	874	854	12
8	2	-4	385	410	7
8	2	-3	271	248	6
8	2	-2	134	72	10
8	2	-1	1107	1049	11
8	2	0	625	614	11
8	2	1	793	770	12
8	2	2	204	133	8
8	2	3	195	214	8
8	2	4	468	476	8
8	2	6	801	780	13
8	2	8	472	469	8
8	2	9	323	313	8
8	2	10	132	158	19
8	2	11	340	327	8
8	2	13	126	125	21
8	2	14	125	156	22
8	3	-13	305	291	9
8	3	-11	318	327	8
8	3	-9	314	324	7
8	3	-8	115	85	14
8	3	-7	247	278	7
8	3	-6	796	742	12
8	3	-5	91	61	16
8	3	-4	468	436	8
8	3	-3	366	334	7
8	3	-2	215	235	7
8	3	-1	602	595	11
8	3	1	81	134	18
8	3	2	309	340	6
8	3	3	162	187	10
8	3	4	500	522	9
8	3	6	88	20	20
8	3	10	181	162	14
8	3	11	314	283	9
8	4	-17	115	99	23
8	4	-16	339	294	9
8	4	-14	152	166	16
8	4	-13	224	229	12
8	4	-12	231	231	11
8	4	-11	317	349	8
8	4	-10	126	131	16
8	4	-9	211	191	9
8	4	-8	264	238	8
8	4	-7	181	200	10
8	4	-6	224	241	8
8	4	-5	371	363	7
8	4	-4	433	428	8
8	4	-2	526	522	10
8	4	-1	163	179	9
8	4	1	123	131	13
8	4	2	99	52	16
8	4	3	463	474	8
8	4	4	650	636	11
8	4	6	474	442	9
8	4	7	708	630	13
8	4	9	230	217	11
8	4	12	118	95	21
8	4	14	178	142	16
8	5	-17	359	346	10
8	5	-15	272	275	11
8	5	-14	301	267	9
8	5	-13	169	201	15
8	5	-12	294	296	9
8	5	-11	129	115	18
8	5	-9	399	412	7
8	5	-8	407	399	7
8	5	-6	665	659	11
8	5	-5	131	126	13
8	5	-4	275	273	7
8	5	-3	258	264	7
8	5	-2	195	205	9
8	5	-1	679	673	12
8	5	1	179	206	10
8	5	2	262	278	8
8	5	3	143	147	13
8	5	4	362	378	7
8	5	5	536	525	10

Values of 10*Fobs and 10*Fcalc

Page 11

H	K	L	Fobs	Fcalc	SigF
8	5	6	146	172	14
8	5	7	236	230	10
8	5	8	244	233	11
8	5	9	335	342	8
8	5	11	191	181	13
8	6	-15	139	140	19
8	6	-14	111	127	22
8	6	-13	138	127	18
8	6	-12	184	177	13
8	6	-11	140	116	17
8	6	-10	388	383	7
8	6	-9	191	192	11
8	6	-8	418	418	7
8	6	-5	417	414	7
8	6	-4	118	144	15
8	6	-3	146	146	12
8	6	-2	142	121	13
8	6	-1	232	243	9
8	6	0	374	354	6
8	6	1	441	451	6
8	6	2	228	242	9
8	6	3	266	267	8
8	6	4	163	162	13
8	6	5	174	154	12
8	6	8	108	121	23
8	6	9	289	308	10
8	6	10	151	126	17
8	6	11	189	188	14
8	6	12	126	127	22
8	7	-14	220	195	12
8	7	-10	283	300	9
8	7	-7	257	228	9
8	7	-5	402	413	7
8	7	-3	109	80	18
8	7	-1	145	165	14
8	7	0	301	317	8
8	7	2	317	309	8
8	7	3	456	464	8
8	7	4	284	267	9
8	7	5	697	725	12
8	7	6	363	337	8
8	7	7	143	121	17
8	7	8	104	187	23
8	7	9	122	82	20
8	7	10	168	159	16
8	8	-14	163	143	17
8	8	-12	399	407	8
8	8	-11	180	161	15
8	8	-10	462	474	9
8	8	-9	377	369	8
8	8	-7	370	367	8
8	8	-5	303	316	9
8	8	-4	194	218	12
8	8	-3	417	413	8
8	8	-2	168	156	13
8	8	0	302	282	9
8	8	1	126	129	19
8	8	2	166	176	15
8	8	3	175	188	14
8	8	4	174	197	14
8	8	6	264	280	10
8	8	8	114	79	22
8	8	9	205	200	14
8	9	-11	348	328	9
8	9	-10	236	247	12
8	9	-9	142	177	18
8	9	-8	202	194	13
8	9	-4	167	157	15
8	9	-3	295	268	9
8	9	-2	292	312	9
8	9	-1	541	527	9
8	9	1	155	234	16
8	9	2	403	402	8
8	9	3	120	79	19
8	9	4	200	199	13
8	9	5	168	173	15
8	9	9	182	170	16
8	10	-9	356	363	9
8	10	-7	107	157	24
8	10	-4	157	158	16
8	10	-1	305	297	9
8	10	0	200	220	13
8	10	2	133	132	18
8	10	5	233	222	12
8	10	7	178	185	16
8	11	-8	177	137	15
8	11	-1	169	198	16
8	11	1	136	105	18
8	11	2	341	348	9
9	0	-16	166	162	15
9	0	-12	787	814	14
9	0	-10	532	530	8
9	0	-8	377	374	8
9	0	-6	218	244	8
9	0	-4	186	196	8
9	0	0	444	465	8
9	0	2	775	793	13
9	0	4	204	206	9
9	0	6	620	623	10
9	0	8	421	416	8
9	0	10	611	627	12
9	0	12	193	203	13
9	0	14	295	291	11
9	1	-18	101	12	24
9	1	-17	140	142	19
9	1	-16	209	200	12
9	1	-15	164	165	15
9	1	-14	411	413	8
9	1	-12	296	290	8
9	1	-11	396	404	7
9	1	-10	457	456	7
9	1	-8	427	447	7
9	1	-7	517	523	9
9	1	-6	471	447	8
9	1	-5	229	254	7
9	1	-3	206	202	8
9	1	-2	115	127	13
9	1	-1	482	449	8
9	1	0	278	270	6
9	1	1	335	352	6
9	1	2	414	403	7
9	1	3	392	433	7
9	1	5	119	157	15
9	1	6	648	641	11
9	1	8	781	810	14
9	1	9	249	247	10
9	1	10	222	240	11
9	1	11	250	236	11
9	2	-15	241	238	11
9	2	-13	523	517	10
9	2	-12	129	147	18
9	2	-11	237	232	9
9	2	-10	308	311	7
9	2	-9	191	210	10
9	2	-7	115	126	15
9	2	-6	360	383	8
9	2	-4	347	383	6
9	2	-3	177	158	9
9	2	-2	905	927	12
9	2	-1	328	374	6
9	2	1	420	412	7
9	2	2	142	162	11
9	2	3	505	486	9
9	2	4	529	541	11
9	2	6	270	281	8
9	2	7	515	506	9
9	2	8	408	403	7
9	2	9	216	204	12
9	2	12	296	294	10
9	3	-17	176	170	16
9	3	-16	338	325	9
9	3	-15	156	163	16
9	3	-14	100	110	24
9	3	-11	656	694	12
9	3	-8	365	390	7
9	3	-7	353	373	7
9	3	-6	156	166	11
9	3	-5	161	167	10
9	3	-4	476	488	9
9	3	-3	219	242	8
9	3	-2	305	296	6
9	3	-1	250	242	7
9	3	0	357	359	6
9	3	1	79	101	19
9	3	2	290	275	7
9	3	3	367	339	6
9	3	4	90	147	19
9	3	6	262	240	9
9	3	7	343	384	8
9	3	9	353	359	8
9	3	10	224	203	11
9	3	11	321	310	9
9	3	12	190	191	14
9	4	-17	165	161	17
9	4	-11	149	149	16
9	4	-8	842	903	14
9	4	-7	295	303	7
9	4	-6	603	602	11
9	4	-5	410	414	7
9	4	-4	320	327	6
9	4	-3	355	365	6
9	4	0	357	387	7
9	4	1	149	127	11
9	4	3	385	389	6
9	4	4	168	183	12
9	4	5	260	255	9
9	4	6	161	139	14
9	4	7	620	623	12
9	4	9	310	302	9
9	4	10	293	319	10
9	4	13	109	79	26
9	5	-15	221	199	12
9	5	-13	201	209	13
9	5	-11	129	145	19
9	5	-10	99	59	23
9	5	-9	381	374	7
9	5	-7	357	362	7
9	5	-4	200	207	10
9	5	-3	279	299	7
9	5	-2	204	210	9

TABLE 6 Structure Factor Tables of Fobs and Fcalc for *gem*-$N_3P_3Cl_4(OSiPh_3)_2$ (**2**) (continued)

Values of 10*Fobs and 10*Fcalc

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H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
9	5	-1	86	81	20	9	8	-5	328	331	8	10	1	1	680	681	12	10	3	6	259	246	9
9	5	1	289	291	8	9	8	-4	200	177	12	10	1	2	348	321	6	10	3	7	398	413	8
9	5	2	630	593	9	9	8	-3	618	639	11	10	1	3	840	793	13	10	3	8	253	264	11
9	5	3	645	666	12	9	8	-2	364	332	8	10	1	4	207	192	10	10	3	12	155	121	17
9	5	4	592	573	10	9	8	-1	445	452	9	10	1	5	179	166	11	10	4	-12	314	334	9
9	5	5	353	356	7	9	8	1	217	212	12	10	1	7	494	490	9	10	4	-11	207	237	12
9	5	6	196	192	13	9	8	9	277	266	11	10	1	9	368	369	8	10	4	-10	639	665	12
9	5	7	142	141	17	9	9	-11	123	114	21	10	1	10	282	270	10	10	4	-9	178	171	13
9	5	8	319	317	9	9	9	-9	142	160	18	10	1	11	170	158	15	10	4	-8	259	264	9
9	5	9	191	204	13	9	9	-8	217	212	12	10	2	-16	206	219	13	10	4	-7	443	463	8
9	5	11	170	167	17	9	9	-6	151	151	16	10	2	-14	185	175	14	10	4	-6	296	294	8
9	6	-15	108	132	26	9	9	-5	315	312	9	10	2	-12	272	282	10	10	4	-4	275	275	7
9	6	-13	144	147	18	9	9	-4	192	186	13	10	2	-11	148	115	15	10	4	-3	111	135	16
9	6	-10	127	118	19	9	9	-3	229	191	11	10	2	-10	186	192	12	10	4	-1	109	131	16
9	6	-9	347	334	8	9	9	-2	322	357	9	10	2	-9	467	506	7	10	4	2	476	452	9
9	6	-6	418	426	8	9	9	-1	209	204	12	10	2	-7	265	244	8	10	4	6	326	347	8
9	6	-5	193	213	11	9	9	0	287	310	9	10	2	-5	621	670	12	10	4	7	415	426	8
9	6	-4	384	412	7	9	9	3	121	136	20	10	2	-4	500	470	9	10	4	10	117	129	22
9	6	-3	481	476	8	9	10	-4	106	32	23	10	2	-3	168	207	10	10	4	11	341	316	10
9	6	-2	109	96	17	9	10	-1	335	338	9	10	2	-2	442	470	8	10	5	-13	214	227	12
9	6	-1	779	755	13	9	10	3	164	150	16	10	2	-1	562	547	9	10	5	-11	174	162	14
9	6	1	305	316	8	9	11	-5	206	197	14	10	2	1	90	77	19	10	5	-10	224	232	11
9	6	3	465	459	9	9	11	-3	254	244	12	10	2	3	227	201	9	10	5	-9	463	477	9
9	6	4	266	288	9	9	11	-2	114	120	24	10	2	4	285	298	8	10	5	-8	593	606	10
9	6	5	721	737	15	9	11	-1	137	142	21	10	2	5	221	238	10	10	5	-7	353	359	7
9	6	6	256	247	10	9	11	0	202	198	14	10	2	6	631	622	11	10	5	-6	437	461	7
9	7	-12	484	489	8	10	0	-14	270	266	10	10	2	7	134	109	17	10	5	-3	353	359	7
9	7	-11	113	81	21	10	0	-12	190	182	12	10	2	8	123	82	19	10	5	5	300	295	9
9	7	-10	413	429	7	10	0	-10	342	339	7	10	2	9	209	213	12	10	5	7	170	197	16
9	7	-9	150	155	16	10	0	-8	514	525	9	10	2	10	235	250	11	10	5	9	163	146	15
9	7	-8	196	217	13	10	0	-6	955	923	13	10	2	12	321	317	10	10	5	11	155	129	17
9	7	-7	573	599	11	10	0	-4	471	492	8	10	2	13	291	278	11	10	6	-13	193	202	13
9	7	-6	602	583	9	10	0	-2	676	660	12	10	3	-15	224	212	12	10	6	-12	168	122	15
9	7	-5	338	333	8	10	0	0	198	212	9	10	3	-14	172	124	14	10	6	-10	190	173	13
9	7	-3	246	237	9	10	0	2	708	680	11	10	3	-13	262	266	10	10	6	-9	172	156	14
9	7	-2	539	518	9	10	0	4	204	191	10	10	3	-10	228	224	10	10	6	-8	256	259	10
9	7	-1	240	251	9	10	0	6	1087	1064	14	10	3	-9	337	334	7	10	6	-7	379	382	8
9	7	0	131	126	16	10	0	8	549	534	9	10	3	-8	771	771	13	10	6	-6	628	660	11
9	7	1	98	99	21	10	0	10	322	273	9	10	3	-7	683	646	12	10	6	-5	278	288	9
9	7	2	329	345	8	10	1	-15	155	161	16	10	3	-6	278	264	8	10	6	-1	145	160	15
9	7	3	391	416	7	10	1	-14	234	234	11	10	3	-5	349	319	6	10	6	1	388	411	8
9	7	4	279	273	9	10	1	-12	341	348	8	10	3	-4	207	226	9	10	6	2	189	200	13
9	7	5	97	78	24	10	1	-10	202	180	10	10	3	-3	167	189	11	10	6	3	347	361	8
9	7	6	205	216	12	10	1	-9	161	162	12	10	3	-1	199	210	9	10	6	4	325	292	9
9	7	8	154	174	17	10	1	-7	234	268	8	10	3	0	539	502	9	10	6	5	295	290	9
9	7	10	320	290	10	10	1	-6	205	214	9	10	3	1	328	306	7	10	6	7	448	450	8
9	8	-10	190	208	14	10	1	-5	121	128	13	10	3	2	544	543	9	10	6	9	157	122	17
9	8	-8	171	179	14	10	1	-4	102	153	16	10	3	3	455	474	9	10	7	-11	127	148	21
9	8	-7	214	212	11	10	1	-2	153	158	10	10	3	4	334	324	7	10	7	-10	180	187	14
9	8	-6	135	149	18	10	1	-1	126	115	12	10	3	5	127	136	17	10	7	-9	175	189	15

Values of 10*Fobs and 10*Fcalc

H	K	L	Fobs	Fcalc	SigF
10	7	-8	174	184	14
10	7	-7	270	246	10
10	7	-6	161	148	15
10	7	-5	141	128	17
10	7	-4	281	289	10
10	7	-2	791	792	14
10	7	-1	403	422	7
10	7	0	446	460	8
10	7	2	111	60	21
10	7	3	127	141	19
10	7	7	174	186	15
10	7	9	288	289	11
10	8	-11	169	144	16
10	8	-7	151	171	16
10	8	-5	219	210	12
10	8	-4	129	128	19
10	8	-3	173	154	14
10	8	-2	135	171	18
10	8	-1	107	113	23
10	8	0	144	110	16
10	8	1	205	209	12
10	8	3	152	149	16
10	8	4	203	174	12
10	8	5	162	163	16
10	8	8	227	257	13
10	9	-10	149	182	18
10	9	-6	239	257	11
10	9	-4	182	183	14
10	9	-3	184	199	14
10	9	-1	267	294	10
10	9	0	405	410	8
10	9	1	131	138	19
10	9	2	311	298	9
10	10	-5	244	226	11
10	10	-3	421	409	8
10	10	-2	211	218	13
10	10	-1	258	246	11
10	10	1	145	148	18
10	10	3	175	165	15
11	0	-14	255	250	10
11	0	-12	120	96	19
11	0	-8	230	262	9
11	0	-6	650	651	11
11	0	-4	733	731	12
11	0	0	457	472	9
11	0	2	234	241	9
11	0	4	155	192	13
11	0	8	134	78	17
11	0	12	334	294	9
11	1	-13	299	292	9
11	1	-11	145	185	16
11	1	-10	344	333	7
11	1	-9	93	83	21
11	1	-8	137	137	14
11	1	-7	114	125	16
11	1	-6	202	185	10
11	1	-5	393	389	7
11	1	-4	189	204	10
11	1	-3	875	874	13
11	1	-2	504	514	9
11	1	-1	173	157	10
11	1	0	213	225	9
11	1	1	360	321	6
11	1	2	441	473	7
11	1	4	257	249	9
11	1	5	227	259	10
11	1	6	642	625	11
11	1	7	311	306	9
11	1	8	296	294	9
11	1	9	449	432	8
11	1	10	220	212	12
11	1	11	209	204	13
11	1	12	152	165	18
11	2	-16	109	111	23
11	2	-14	302	302	9
11	2	-13	144	156	17
11	2	-12	236	257	10
11	2	-8	479	470	9
11	2	-6	756	748	13
11	2	-5	471	493	9
11	2	-4	269	261	7
11	2	-3	131	116	13
11	2	-2	206	181	9
11	2	0	189	197	10
11	2	1	899	929	14
11	2	2	233	243	9
11	2	3	271	264	8
11	2	4	554	551	9
11	2	5	565	606	11
11	2	6	415	405	9
11	2	7	141	158	17
11	2	8	273	280	10
11	2	11	236	228	12
11	3	-14	111	70	22
11	3	-13	122	100	19
11	3	-12	98	144	24
11	3	-11	130	144	17
11	3	-10	632	626	11
11	3	-9	168	178	14
11	3	-8	483	486	7
11	3	-7	448	445	8
11	3	-5	437	445	7
11	3	-4	173	172	11
11	3	-1	289	285	7
11	3	0	134	117	13
11	3	2	172	177	12
11	3	5	112	97	19
11	3	6	137	123	17
11	3	8	382	374	8
11	3	10	318	289	9
11	3	11	166	167	16
11	4	-12	319	325	9
11	4	-10	255	230	10
11	4	-9	425	426	7
11	4	-8	273	265	9
11	4	-5	237	211	9
11	4	-4	300	311	8
11	4	-1	246	265	9
11	4	0	190	174	11
11	4	6	157	152	15
11	4	8	129	135	19
11	4	9	155	157	16
11	4	10	310	298	10
11	5	-11	237	202	11
11	5	-6	232	242	10
11	5	-3	160	186	13
11	5	-1	417	439	7
11	5	0	566	576	10
11	5	1	381	389	7
11	5	2	447	446	7
11	5	3	304	305	8
11	5	4	180	143	13
11	5	6	265	255	10
11	5	7	375	406	8
11	6	-14	121	112	21
11	6	-13	229	244	13
11	6	-11	233	219	11
11	6	-9	172	190	15
11	6	-7	166	191	14
11	6	-5	312	331	9
11	6	-4	468	474	9
11	6	-3	272	266	9
11	6	-2	421	435	8
11	6	0	95	48	23
11	6	1	440	437	7
11	6	2	287	272	9
11	6	3	260	274	10
11	6	4	149	181	16
11	6	5	286	277	10
11	6	6	251	228	11
11	6	7	160	112	15
11	6	9	217	206	13
11	7	-11	270	247	10
11	7	-10	299	321	10
11	7	-9	195	210	13
11	7	-8	150	153	17
11	7	-6	303	324	9
11	7	-3	152	179	17
11	7	-1	146	148	16
11	7	0	176	177	14
11	7	2	276	264	10
11	7	3	194	193	13
11	7	4	97	72	23
11	7	6	329	299	9
11	7	7	130	122	19
11	7	8	190	180	15
11	8	-10	135	171	20
11	8	-9	170	161	15
11	8	-7	174	167	13
11	8	-6	282	241	9
11	8	-5	271	286	10
11	8	-4	227	226	11
11	8	-1	179	195	14
11	8	0	105	117	22
11	8	1	183	187	14
11	8	4	235	260	11
11	8	7	199	217	15
11	9	-3	332	339	9
11	9	4	132	134	20
11	10	0	178	178	15
12	0	-8	115	170	18
12	0	-6	109	94	17
12	0	-2	612	587	11
12	0	4	622	628	12
12	0	8	230	216	11
12	1	-14	98	76	23
12	1	-12	166	171	14
12	1	-11	217	211	12
12	1	-10	289	279	9
12	1	-9	259	280	10
12	1	-8	372	397	8
12	1	-7	193	195	11
12	1	-6	916	954	14
12	1	-5	452	474	9
12	1	-4	771	798	13
12	1	-3	360	359	7
12	1	-1	361	373	7
12	1	0	172	180	11
12	1	1	162	171	12
12	1	2	240	242	9

TABLE 6 Structure Factor Tables of Fobs and Fcalc for *gem*-$N_3P_3Cl_4(OSiPh_3)_2$ (**2**) (continued)

Values of 10*Fobs and 10*Fcalc

Page 14

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
12	1	3	312	322	8	12	4	-5	413	419	7	12	9	2	214	200	13	13	3	5	435	431	8
12	1	4	140	137	16	12	4	-4	184	188	11	13	0	-14	220	198	13	13	3	7	261	239	11
12	1	5	462	460	7	12	4	-3	210	207	10	13	0	-8	308	334	9	13	3	8	241	244	12
12	1	8	153	167	16	12	4	-2	240	267	9	13	0	-6	202	198	12	13	4	-10	128	101	19
12	1	10	251	266	12	12	4	0	245	230	10	13	0	-2	345	372	7	13	4	-8	310	315	9
12	2	-16	144	74	18	12	4	2	197	221	12	13	0	2	459	456	7	13	4	-7	256	252	10
12	2	-15	149	123	17	12	4	3	459	467	9	13	0	4	261	252	10	13	4	-6	107	114	21
12	2	-14	105	35	22	12	4	4	437	433	9	13	0	8	148	142	17	13	4	-5	107	114	22
12	2	-13	132	123	18	12	4	7	110	109	21	13	1	-12	320	329	9	13	4	-4	283	292	9
12	2	-11	115	103	21	12	4	8	332	351	9	13	1	-11	340	344	8	13	4	-3	324	335	8
12	2	-10	269	254	10	12	5	-13	222	239	12	13	1	-8	484	523	9	13	4	-2	449	460	8
12	2	-9	202	251	12	12	5	-12	138	170	18	13	1	-7	308	318	9	13	4	-1	140	163	17
12	2	-8	686	692	11	12	5	-11	291	276	9	13	1	-6	426	401	9	13	4	3	252	252	10
12	2	-7	505	502	9	12	5	-10	267	266	10	13	1	-5	318	339	8	13	4	5	98	80	24
12	2	-6	735	767	13	12	5	-9	318	312	9	13	1	-4	136	118	15	13	5	-13	167	172	16
12	2	-5	309	310	8	12	5	-8	169	147	14	13	1	-3	377	384	7	13	5	-8	239	214	11
12	2	-4	240	239	9	12	5	-5	317	311	8	13	1	-2	152	154	14	13	5	-7	165	177	14
12	2	-3	267	269	8	12	5	-4	150	204	15	13	1	0	371	380	8	13	5	-5	148	171	16
12	2	-2	313	303	7	12	5	-3	191	196	12	13	1	2	807	787	14	13	5	-4	152	170	16
12	2	-1	135	138	14	12	5	-1	299	302	9	13	1	3	158	137	15	13	5	-3	748	700	12
12	2	0	296	307	8	12	5	1	360	353	8	13	1	4	152	144	16	13	5	-2	134	152	18
12	2	1	283	320	8	12	5	5	212	214	12	13	1	5	209	204	12	13	5	0	135	114	16
12	2	2	246	227	9	12	5	9	129	105	20	13	1	6	155	117	15	13	5	2	286	272	9
12	2	3	222	206	11	12	6	-13	179	139	15	13	1	7	217	185	12	13	5	3	240	227	10
12	2	5	211	206	11	12	6	-12	104	7	22	13	1	9	168	149	16	13	5	4	131	125	18
12	2	6	165	135	14	12	6	-10	165	192	15	13	2	-13	146	159	17	13	5	5	204	198	12
12	2	7	154	112	16	12	6	-9	122	97	19	13	2	-11	204	205	12	13	6	-11	315	312	10
12	2	9	163	170	15	12	6	-6	205	197	12	13	2	-9	481	514	9	13	6	-10	158	139	17
12	2	10	413	364	8	12	6	-5	354	334	8	13	2	-7	509	498	8	13	6	-9	313	280	9
12	2	11	329	273	10	12	6	-4	240	225	10	13	2	-5	259	243	10	13	6	-8	118	123	21
12	3	-11	201	218	13	12	6	-3	321	311	9	13	2	-4	559	552	10	13	6	-6	156	143	15
12	3	-10	191	197	13	12	6	-1	441	415	8	13	2	-3	383	384	7	13	6	-5	142	165	17
12	3	-9	97	97	23	12	6	1	239	239	11	13	2	-2	577	604	10	13	6	-4	113	127	21
12	3	-7	294	293	9	12	6	5	171	143	14	13	2	-1	227	210	11	13	6	-1	107	89	21
12	3	-4	274	262	8	12	7	-9	139	127	17	13	2	0	386	398	7	13	6	0	219	252	12
12	3	-3	118	88	16	12	7	-7	150	147	16	13	2	1	579	579	10	13	6	3	219	231	12
12	3	-2	537	561	9	12	7	-6	227	246	11	13	2	2	276	273	9	13	6	6	342	325	9
12	3	-1	134	161	15	12	7	-2	234	226	11	13	2	3	408	406	7	13	7	-10	223	254	13
12	3	0	346	329	7	12	7	-1	252	281	10	13	2	6	121	130	20	13	7	-8	120	101	21
12	3	1	148	154	14	12	7	0	129	133	18	13	2	7	163	160	15	13	7	-5	127	129	20
12	3	3	408	402	7	12	7	2	268	260	10	13	3	-14	247	210	12	13	7	-3	130	110	19
12	3	5	117	100	19	12	7	3	129	120	19	13	3	-12	340	340	9	13	7	-2	278	304	10
12	3	6	284	296	10	12	7	4	164	150	15	13	3	-11	120	122	21	13	7	-1	143	141	17
12	3	7	144	161	18	12	7	7	236	233	12	13	3	-10	116	133	21	13	7	0	260	252	10
12	3	8	118	135	21	12	8	-5	139	120	16	13	3	-7	351	361	8	13	7	1	222	205	12
12	3	9	129	97	19	12	8	-2	329	340	9	13	3	-6	401	414	7	13	7	4	152	120	17
12	4	-12	109	160	22	12	8	0	237	241	11	13	3	-4	414	411	7	13	7	5	149	137	18
12	4	-10	259	251	10	12	8	4	315	296	10	13	3	-2	169	175	14	13	8	-7	108	138	24
12	4	-8	126	51	17	12	9	-2	191	190	14	13	3	1	164	187	14	13	8	-6	257	258	12
12	4	-7	387	388	7	12	9	-1	114	143	23	13	3	4	119	70	19	13	8	-4	238	230	11

Values of 10*Fobs and 10*Fcalc Page 15

H	K	L	Fobs	Fcalc	SigF
13	8	-2	274	266	10
13	8	0	175	124	15
13	8	1	147	150	18
13	8	2	174	145	16
13	8	3	139	105	19
13	9	-3	169	153	16
14	0	-14	129	150	21
14	0	-12	229	217	12
14	0	-10	157	169	15
14	0	-4	236	258	11
14	0	-2	374	383	8
14	0	0	268	246	10
14	0	2	682	660	13
14	1	-11	158	138	15
14	1	-9	391	390	8
14	1	-7	247	223	10
14	1	-6	239	251	11
14	1	-5	343	348	8
14	1	-4	153	151	16
14	1	-3	319	332	9
14	1	-1	115	111	21
14	1	0	336	342	9
14	1	1	412	426	8
14	1	2	353	352	9
14	1	3	199	206	13
14	1	6	122	149	22
14	1	7	136	130	19
14	2	-14	131	121	21
14	2	-11	197	219	14
14	2	-10	248	231	11
14	2	-7	168	164	15
14	2	-6	293	303	9
14	2	-4	371	407	8
14	2	0	108	58	21
14	2	1	148	140	17
14	2	2	308	330	9
14	2	3	176	134	14
14	2	4	271	263	10
14	2	6	161	120	16
14	3	-13	201	202	15
14	3	-11	150	169	18
14	3	-9	194	206	13
14	3	-7	347	338	8
14	3	-6	215	225	11
14	3	-4	521	557	9
14	3	-2	462	457	7
14	3	0	187	180	13
14	3	1	276	276	10
14	3	2	173	147	15
14	3	4	202	201	13
14	3	6	105	89	24
14	4	-12	284	290	11
14	4	-8	202	186	13
14	4	-7	209	197	12
14	4	-5	156	185	16
14	4	-3	219	207	12
14	4	-1	123	85	19
14	4	2	129	143	18
14	4	6	213	181	13
14	5	-10	120	156	22
14	5	-5	438	432	8
14	5	-4	123	102	20
14	5	-2	184	175	13
14	5	0	289	301	9
14	5	1	140	126	16
14	6	-10	288	278	10
14	6	-8	145	147	18
14	6	-6	103	117	25
14	6	-5	261	263	11
14	6	-3	568	570	10
14	6	-1	448	430	8
14	6	0	236	235	11
14	6	1	205	198	13
14	6	2	135	141	18
14	7	-7	186	168	15
14	7	-6	250	267	12
14	7	-4	106	93	24
14	7	-3	182	187	15
14	7	2	130	112	20
15	0	-4	210	195	12
15	0	-2	386	379	8
15	0	0	373	368	8
15	0	2	518	452	8
15	0	4	138	104	18
15	1	-11	139	132	19
15	1	-10	188	199	15
15	1	-9	145	133	18
15	1	-2	147	137	17
15	1	1	443	427	8
15	1	2	207	224	13
15	1	3	281	277	10
15	1	4	159	129	16
15	1	5	120	104	22
15	1	6	164	181	18
15	2	-11	144	149	19
15	2	-10	152	125	18
15	2	-9	270	268	11
15	2	-8	231	214	12
15	2	-7	166	175	15
15	2	-2	265	278	10
15	2	0	357	339	8
15	2	2	282	271	10
15	2	4	193	167	14
15	2	6	167	180	17
15	3	-12	146	149	19
15	3	-10	173	174	16
15	3	-8	147	133	18
15	3	-7	330	329	9
15	3	-5	123	103	19
15	3	-4	122	117	20
15	3	-1	213	209	12
15	3	1	274	256	10
15	3	2	161	165	16
15	3	3	126	82	20
15	3	5	233	228	13
15	4	-9	178	185	15
15	4	-6	317	300	9
15	4	-5	383	382	8
15	4	-4	373	366	8
15	4	-3	218	219	12
15	4	0	244	230	11
15	4	1	291	282	10
15	4	2	146	151	18
15	4	4	183	142	15
15	5	-5	151	138	17
15	5	-3	252	264	11
15	5	-1	118	98	21
15	5	3	138	170	20
15	6	-7	183	168	15
15	6	-6	288	279	10
15	6	-4	119	140	23
15	6	-3	138	157	20
15	6	-1	162	158	17
16	0	-10	197	205	14
16	0	-4	337	335	9
16	0	-2	433	409	8
16	0	4	146	157	19
16	1	-11	123	147	23
16	1	-5	163	160	16
16	1	-2	154	121	16
16	1	0	283	303	10
16	1	4	138	105	20
16	2	-10	253	239	12
16	2	-9	149	174	18
16	2	-4	279	269	10
16	2	-1	367	372	8
16	2	0	140	113	18
16	2	1	198	203	14
16	3	-8	128	112	20
16	3	-7	311	320	10
16	3	-6	273	270	11
16	3	-4	133	110	18
16	3	2	120	98	23
16	4	-8	238	232	12
16	4	-6	314	304	10
16	4	-5	149	141	18
16	4	-3	190	194	15
16	4	0	115	108	23
16	5	-5	230	230	12
16	5	-4	220	176	13
16	5	-3	146	124	19
16	5	0	161	113	17
17	0	-6	119	81	21
17	0	-4	201	203	14
17	0	-2	237	218	12
17	1	-8	120	126	22
17	1	-3	159	142	17
17	1	-1	221	203	13
17	2	-7	160	185	18
17	2	-2	182	182	15
17	2	0	227	217	12
17	3	-4	133	127	21