Table VI. Selected Inter- and Intramolecular Nonbonding and Hydrogen-Bonding Contacts (Å) for $[(C_6H_5NH)_2P]_2NC_6H_5$ (**2**)

(a) Intermolecular Contacts			
C(3)-C(13)	3.60 (2)	C(12)-C(14)	3.46 (2)
C(3)-C(14)	3.67 (2)	C(13)-C(12)	3.41 (2)
C(11)-C(13)	3.57 (2)	C(13)-C(11)	3.57 (2)
C(11)-C(43)	3.74 (2)	C(13)-C(13)	3.59 (2)
C(12)-C(13)	3.41 (2)	C(14)-C(12)	3.46 (2)
(b) Intramolecular Contacts			
P(1)-P(2)	2.897 (6)	N(5)-C(31)	3.23 (2)
N(1)-C(31)	3.20 (2)	N(5)-C(32)	3.50 (2)
N(1)-C(36)	3.40 (2)	P(1)-C(2)	3.54 (1)
N(2)-C(31)	3.11 (2)	P(1)-C(6)	3.58 (1)
N(2)-C(32)	3.26 (2)	P(2)-C(2)	3.53 (1)
N(4)-C(31)	3.10 (2)	P(2)-C(6)	
N(4)-C(36)	3.25 (2)		
(c) Hydrogen Bonding Contacts			
O(1)-N(1)	3.04 (1)	O(1)-H(1N1)	2.13 (1)
O(1)-N(2)	3.03 (1)	O(2)-H(2N2)	2.09 (1)

the skeleton conformation is twisted slightly from a pure cisoid, C_{2v}, molecular symmetry pattern. This general conformation has been reported previously for $[(C_6H_5NH)_2P_2(NC_6H_5)_2]_2$-$NC_6H_5$[26] and $[(C_6H_5)_2PN(CH_3)PN(t\text{-}C_4H_9)]_2$[28] in the solid and postulated, based on nuclear magnetic resonance coupling constant data, for bisphosphinoamines in solution.[29]

The structural and bonding parameters for **2** (Table IV) as with **1** are consistent with those of previously studied phenylamido- and/or phenylimidophosphines.[25,26] The anilino groups are planar (planes 1–5) within experimental error. Mean C_6H_5N–P distances of 1.69 Å are identical with the mean exo-$C_6H_5N(H)$–P distances of 1.69 Å. The N–P–N angles in the molecule vary somewhat; mean $C_6H_5N(H)$–P–$N(H)C_6H_5$ angles are 94.8° and mean $C_6H_5N(H)$–P–NC_6H_5 angles are 105.3°. However, the mean of all N–P–N angles is 101.8°, only slightly greater than that of 99.5° observed for the structurally less complex **1**. Comparison of the bond lengths and angles involving N(1) and N(2) with these around N(4) and N(5) suggests that hydrogen bonding has little effect on the conformation of **2**.

Short intramolecular contacts are present between carbon atoms C(31), C(32), and C(36) and nitrogen atoms N(1), N(2), N(4), and N(5) (Table VI). These are consistent with the approximately perpendicular orientation of phenyl ring 3 (plane 3) with respect to the P(1), N(3), P(2) plane (plane 11); dihedral angle = 94°. Examination of intermolecular contacts shows, except for the oxygen atom, C(3) is the only atom in the ether with possibly significant contacts with **2**, i.e., C(3)···C(13) and C(3)···C(14). Phenyl ring 1 is related to itself through the inversion center at 0, $^1/_2$, 0. C(11), C(12), C(13), and C(14) are related by this symmetry operator such that the contacts between them vary from 3.4 to 3.6 Å. These are in the range reported for phenyl stacking interactions.[30] It seems likely that the phenyl stacking has more influence on the configuration of compound **2** than the contacts involving C(3).

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Registry No. **1**, 15159-51-0; **2**·$O(C_2H_5)_2$, 80953-59-9; $C_6H_5NH_2$, 62-53-3; $[(C_2H_5)_2N]_3P$, 2283-11-6.

Supplementary Material Available: Listings of structure factor amplitudes, calculated hydrogen positions and thermal parameters, and thermal parameters for nonhydrogen atoms (12 pages). Ordering information is given on any current masthead page.

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Molecular Structure of Gaseous Vanadium Pentafluoride, VF_5

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The molecular structure of VF_5 has been investigated at 30 °C by electron diffraction from the gas. Two models were tested, one based upon an assumed D_{3h} symmetry and one in which all distances were refined as independent parameters without regard for geometrical consistency. The purpose was to explore possible deviations from D_{3h} symmetry implicit in the existence of low-lying polar states discovered from electric field deflection experiments. The results are inconclusive. Although the D_{3h} model gives a slightly poorer fit than does the other, the difference is not statistically significant. At the same time, there are indications in the values of the vibrational amplitudes and in the nonbond distance defects ("shrinkages") that some aspects of the molecular motion are not properly accounted for by either of the models. Such indications are consistent with small deviations from D_{3h} symmetry, e.g., not more than a few degrees in any bond angle. Our distance (r_a) and amplitude (l) values in angstroms with estimated 2σ uncertainties for the D_{3h} model followed by those of the free-distance model are as follows: $r(V-F_{eq}) = 1.708$ (5), 1.701 (7); $r(V-F_{ax}) = 1.734$ (7), 1.747 (13); $r(F_{eq}\cdots F_{ax}) = 2.432$ (3), 2.428 (4); $r(F_{eq}\cdots F_{eq}) = 2.950$ (8), 2.947 (12); $r(F_{ax}\cdots F_{ax}) = 3.463$ (14), 3.471 (17); $l(V-F_{eq}) = l(V-F_{ax}) = 0.051$ (3), 0.047 (2); $l(F_{eq}\cdots F_{ax}) = 0.093$ (5), 0.093 (5); $l(F_{eq}\cdots F_{eq}) = 0.114$ (10), 0.115 (10); $l(F_{ax}\cdots F_{ax}) = 0.068$ (14), 0.067 (14). Results also include the symmetrized quadratic force field used to calculate corrections for the effects of vibrational averaging.

Introduction

Two types of structures have been observed for inorganic pentafluoride molecules in the gas phase, tetragonal pyramidal (C_{4v} symmetry) and trigonal bipyramidal (D_{3h} symmetry). The halogen pentafluorides are of the first type and pentafluorides of atoms from groups 5A and 5B are of the second. From among the latter the structures of PF_5,[1] AsF_5,[2] VF_5,[3] NbF_5,[3a,4] and TaF_5[3a,4] have been measured by electron diffraction. Other structurally related measurements include NMR (for PF_5[5,6] and AsF_5[7]), infrared and Raman spectra (for

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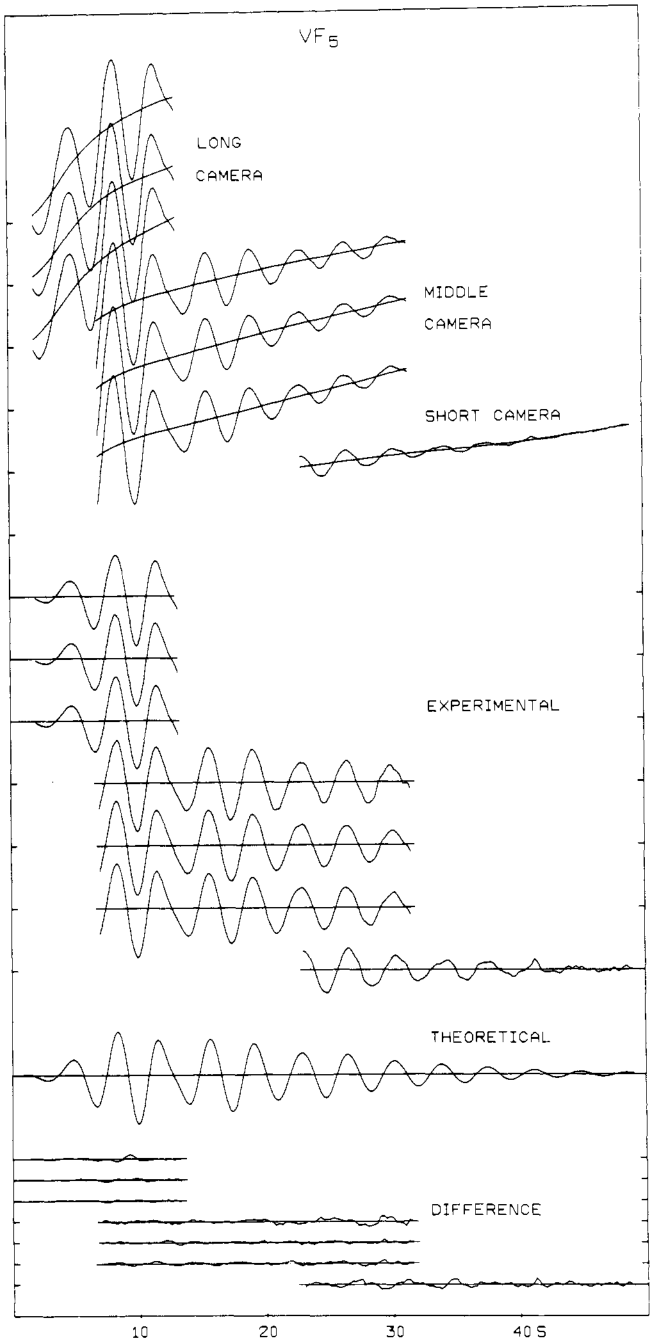

Figure 1. Intensity curves. The experimental curves are (upper) s^4I_t superposed on the final backgrounds (the curves are magnified 5 times to show better the molecular part of the scattering) and (lower) s^4I_t minus backgrounds times s. The theoretical curve is for model 1. Difference curves are experimental minus theoretical.

PF_5,[8-10] AsF_5,[8-10] and VF_5[8,9,11]), and electric field deflection (for all five[12]).

The diffraction work revealed that the axial bonds in PF_5, AsF_5, NbF_5,[4] and TaF_5[4] are longer than the equatorial ones in accord with prediction based on the well-known valence shell electron pair repulsion (VSEPR) theory.[13] It is also the case for VF_5, as shown in an unpublished electron-diffraction investigation[14a] and in preliminary work some years ago in this laboratory. Recently, we decided to take up the VF_5 problem again. Accurate measurements of the bond lengths were of course desirable, but our principal interest centered on the detection of a possible distortion of the molecule from its nominal D_{3h} symmetry. Evidence for such a distortion is implicit in the results of the electric deflection experiments, which find low-lying polar states in VF_5 but none in the other D_{3h} pentafluorides. Also, the vibrational spectrum of VF_5 differs from those of PF_5 and AsF_5 in ways that are not inconsistent with structural differences. We knew from our preliminary work that any distortion was surely small, and on this account alone its detection would be difficult. The problem is further complicated, however, by the Berry inversion[15] that the D_{3h} pentafluorides are inferred to undergo according to interpretations of NMR and spectroscopic data. Although direct evidence of this process has not been found in the diffraction data from any of these molecules, it might be detectable in VF_5 in view of the other differences VF_5 exhibits.

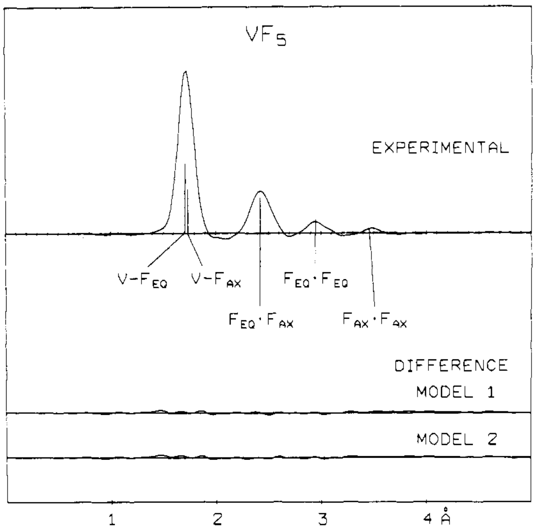

Figure 2. Experimental radial distribution curve and differences, experimental minus theoretical, for the two models tested, $B = 0.0009$ Å^{-2}. Vertical lines indicate positions of r_a distances in model 1; the lengths of the lines are proportional to the weights of the distances.

Experimental Section

The VF_5 sample (<98%) was obtained from ROC/RIC, Research Organic/Inorganic Chemical Corp., and was used without further purification. Diffraction photographs were made in the Oregon State apparatus with an r^3 sector at a temperature of 30 °C with Kodak projector slide (medium) plates. Exposures were made for 20–480 s. Ambient pressure in the apparatus during the experiments was (3.0×10^{-6})–(2.0×10^{-5}) torr. Nozzle-to-plate distances were 749.08 ("long camera"), 299.80 ("middle camera"), and 119.68 nm ("short camera"). The electron wavelength was 0.057 36–0.057 42 Å calibrated in separate experiments from CO_2 diffraction patterns ($r_a(CO) = 1.1646$ Å, $r_a(OO) = 2.3244$ Å). Three plates from the long, three from the middle, and one from the short camera distance were selected for structure analysis.

The procedures for obtaining the total scattered intensity distributions $s^4(I_t(s))$, the molecular intensities $s(I_m(s))$, and the radial

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Table I. Symmetry Force Constants and Wavenumbers for VF_5[a]

							obsd[b]	calcd
a_1	F_{11}	V-F_{eq}	5.374	0.700		ν_1	718	718
	F_{22}	V-F_{ax}		4.534		ν_2	608	608
a_2	F_{33}	V-F_{ax}	4.541	1.000		ν_3	784	784
	F_{44}	α(ax)		1.161		ν_4	331	331
e′	F_{55}	V-F_{eq}	4.514	0.400	0.500	ν_5	810	810
	F_{66}	α(eq)		0.289	−0.100	ν_6	282	282
	F_{77}	α(ax)			0.980	ν_7	109	140[c]
e″	F_{88}	α(ax)	1.061			ν_8	336	336

[a] Units for force constants are aJ $Å^{-2}$ for stretching and aJ rad^{-2} for bending, with corresponding units for interaction constants. Units for wavenumbers are cm^{-1}. [b] Reference 8. [c] The value for ν_7 was intentionally calculated higher than the observed one; see ref 24.

distribution curves $rD(r)$ have been described.[16] The values of the electron-scattering amplitudes (and the phases required for other calculations) were taken from ref 17. Figure 1 shows curves of the total intensities and backgrounds. These data are available as supplementary material. Figure 2 shows the final experimental radial distribution curve calculated with the convergence factor B equal to 0.0009 $Å^2$.

Structure Analysis

Description of Models. The positions of the peaks of the radial distribution curve reveal immediately that any distortion of the molecule from D_{3h} symmetry, as is suggested by the electric field deflection experiments, must be very small: these positions are consistent with that symmetry after account has been taken of the normally encountered effects of harmonic vibration (shrinkage). We first investigated a model based on an assumed D_{3h} symmetry and distances of the geometrically consistent r_α type taking the average of the bond lengths, $\langle V{-}F\rangle = [3r(V{-}F_{eq}) + 2r(V{-}F_{ax})]/5$, and their difference, $\Delta(V{-}F) = r(V{-}F_{ax}) - r(V{-}F_{eq})$, as parameters. The vibrational data necessary for the conversion of the r_α distances to the r_a used in the diffraction equations ($r_a = r_\alpha + K + \delta r - l^2/r_\alpha$) were calculated from the quadratic force field described below. We also tested a model with no symmetry constraint, i.e., one in which the five distances were treated as independent parameters, in order to obtain an estimate of the experimental shrinkage. For each of these models the amplitudes associated with the nonbond distances could be refined without constraints. However, the correlations among the bond lengths and their amplitudes required that the difference between the latter be assigned. We set the difference to zero in accordance with the calculated values. Anharmonicity corrections were included for the bond lengths only; the κ values obtained from the usual diatomic molecule approximation[18] were 1.14 × 10^{-6} Å for both V-F_{eq} and V-F_{ax}.

Quadratic Force Field. The complete set of fundamental frequencies[8] as well as the results of several force field[8,19-22] and root mean square amplitude[19,20,23] calculations have been reported for VF_5. Our interest in the force field arose mainly from our need for the quantities K (perpendicular amplitude corrections) and δr (centrifugal distortions) used in converting r_α distances to the other types. The force field we adopted is given in Table I. It differs from those proposed by other authors mainly in the values of the bending constants in the e′ block and in the values of several of the off-diagonal constants. For example, the axial bending constant F_{77} has a value larger than previously published values and comparable to those of the other axial bends F_{44} and F_{88}. Also, the equatorial bending constant F_{66} has a value that is smaller than previous values and considerably smaller than the axial bending constants. Most of the off-diagonal constants have magnitudes somewhat larger than those proposed before. These changes are consistent with recent results for other trigonal-bipyramidal molecules.[24] These recent results have suggested also, for reasons connected to the Berry inversion, that the wavenumber of the inversion mode should be increased in order to give agreement between the observed and calculated $X_{eq}\cdots X_{eq}$ amplitudes. This increase for VF_5 was taken to be about 30 cm^{-1}. Except for this, the force field of Table I reproduces the observed wave numbers exactly.

Refinement Results. The structure was refined by least-squares adjustment of the theoretical intensity curve to the seven experimental curves[25] (Figure 1) with a unit weight matrix. The results are given in Table II. The agreement between corresponding r_a values for the two models is reasonably good, but the differences that do exist raise the question as to whether a model of D_{3h} symmetry is an adequate description of the structure. A conservative interpretation of the results of Hamilton's test[26] suggests that model 1 may not be rejected in favor of model 2 at the 5% level usually regarded as significant, and we therefore conclude that the electron-diffraction results are consistent with D_{3h} symmetry for the molecule. The two models of Table II suggest the range of parameter space compatible with our data. The correlation matrix for model 1 is given in Table III; that for model 2 is in the supplementary material.

Discussion

The most interesting question about the VF_5 structure concerns the matters of molecular symmetry and of the Berry-type inversion, which, through angle deformations, exchanges axial and equatorial bonds while preserving C_{2v} symmetry for the molecule. There are several points about the diffraction results that bear on these matters, as follows. First, it is clear that any deviation from D_{3h} symmetry such as is implied by the electric deflection experiments[12] must be small—probably not larger than a few degrees in any bond angle. Second, any Berry-type inversion process in VF_5 cannot involve a smooth, large amplitude motion (in the classical sense) because there is no obvious indication of molecules distributed throughout intermediate positions along the inversion coordinate. Third, although the diffraction data are consistent with D_{3h} symmetry for the molecule as mentioned in the preceding section, the amplitude values give some suggestion of molecular motion not properly accounted for by either of the two models. Specifically, the calculated amplitude values in Table II are, except for $l(F_{eq}\cdots F_{eq})$, smaller than the experimental ones. (The possible significance of this third point is weakened a bit by the amplitude measurements for VF_5 obtained in Thompson and Bartell's unpublished investigation[14b] where the values for the bonds and for $F_{ax}\cdots F_{ax}$ appear to be in slightly better agreement with the calculated values than ours.) The last point concerns the shrinkages (Table II), which may also be interpreted as indicating dy-

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Table II. Structural Results for VF_5[a]

	model 1 (D_{3h})[b]				model 2[c]		
	r_α	r_g	r_a	l	r_a	l	l(calcd)
⟨V–F⟩[d]	1.715	1.720	1.718 (2)		1.719 (2)		
Δ(V–F)[d]	0.028	0.027	0.027 (11)		0.046 (20)		
V–F_{eq}	1.704	1.709	1.708 (5)	0.051 (3)	1.701 (7)	0.047 (2)	0.0430
V–F_{ax}	1.732	1.736	1.734 (7)	0.051	1.747 (13)	0.047	0.0430
$F_{eq}\cdots F_{ax}$	2.429	2.435	2.432 (3)	0.093 (5)	2.428 (4)	0.093 (5)	0.0809
$F_{eq}\cdots F_{eq}$	2.951	2.955	2.950 (8)	0.114 (10)	2.947 (12)	0.115 (10)	0.1145
$F_{ax}\cdots F_{ax}$	3.463	3.465	3.463 (14)	0.068 (14)	3.471 (17)	0.067 (14)	0.0557
R[e]	0.077				0.076		
adjusted vibrational cor (shrinkages)[f]							
$F_{eq}\cdots F_{ax}$			0.002		0.010 (6)		
$F_{eq}\cdots F_{eq}$			0.007		−0.002 (17)		
$F_{ax}\cdots F_{ax}$			0.005		0.022 (31)		

[a] In angstroms. Values in parentheses are estimated 2σ. [b] Uncertainties for r_α and r_g estimated to be the same as for r_a. [c] Model specified by five independent distances. [d] See text for definitions of these parameters. [e] $R = [\Sigma_i w_i \Delta_i^2 / \Sigma w_i (s_i(I_i(s)))^2]^{1/2}$ where $\Delta_i = s_i(I_i(s)_{\text{obsd}}) - s_i(I_i(s)_{\text{calcd}})$. [f] Nonbond distance calculated from bond lengths with assumed D_{3h} symmetry, minus observed nonbond distances.

Table III. Correlation Matrix (×100) for Model 1

	⟨r(F–F)⟩	Δr(V–F)	l(V–F)	$l(F_{eq}\cdots F_{ax})$	$l(F_{eq}\cdots F_{eq})$	$l(F_{ax}\cdots F_{ax})$
	100	16	−13	7	−2	−1
		100	−85	−5	4	2
			100	16	<1	1
				100	−14	−2
					100	−10
σ[a]	0.022	0.38	0.056	0.10	0.31	0.49

[a] Standard deviations (×100) from least squares; all values in the table are in angstroms.

namic effects not implicit in the models. Shrinkages, as the footnote to the table indicates, are distance defects. They arise from molecular vibration, which causes an average interatomic *distance* to be different from the distance between the average *positions* of an atomic pair: in linear CO_2, for example, the bending vibrations result in an O···O distance less than twice the bond length. Shrinkages may be calculated from molecular force fields and the molecular structure with assumed symmetry. In some cases, including VF_5, experimental measures are obtained. Thus, of the two sets of shrinkages in Table II, that for model 1 may be termed calculated (i.e., predicted from the force field) and that for model 2 experimental because the distances were refined as independent parameters. The calculated shrinkage values for $F_{eq}\cdots F_{eq}$ and $F_{ax}\cdots F_{ax}$ are very sensitive functions of the parameter Δ(V–F) whereas that for $F_{ax}\cdots F_{eq}$ is less so. For example, with Δ(V–F) = 0.032 Å, an insignificant change from that shown for model 2, the experimental shrinkages become 0.007, 0.007, and 0.004 Å for $F_{eq}\cdots F_{ax}$, $F_{eq}\cdots F_{eq}$, and $F_{ax}\cdots F_{ax}$, respectively. As a set these values are in much better agreement with the calculated ones, but the $F_{eq}\cdots F_{ax}$ value is still too large. In summary, the diffraction results are consistent with D_{3h} symmetry for the VF_5 molecule, but small deviations correponding to a lower equilibrium symmetry cannot be ruled out, and there is weak evidence in both the amplitude and shrinkage values that suggests that such small deviations may quite possibly be present.

The interatomic distances we find in VF_5 are, with the exception of $F_{ax}\cdots F_{ax}$, in good agreement with those from the earlier investigations[3] (⟨V–F⟩ = 1.71 Å, $F_{eq}\cdots F_{ax}$ = 2.43 Å, $F_{eq}\cdots F_{eq}$ = 2.94 Å, $F_{ax}\cdots F_{ax}$ = 3.41 Å). Our result for Δ(V–F), particularly that from model 2, is also in good agreement with the value 0.036 ± 0.010 Å obtained by Thompson and Bartell[14b] in their unpublished work. As is usual in compounds with several electronegative ligands, the bond lengths are considerably shorter than normal, in this case about 0.06–0.08 Å after correction for electronegativity difference. The relative lengths are as expected from VSEPR theory.

Acknowledgment. We are grateful to the National Science Foundation for support of this work (Grant CHE 78-04258).

Registry No. VF_5, 7783-72-4.

Supplementary Material Available: Tables of total intensities and final backgrounds from the seven plates used in the refinements and the correlation matrix for the parameters used for model 2 (Tables IV–VII) (13 pages). Ordering information is given on any current masthead page.