was stirred for 12 h and the solvent removed in vacuuo to yield the crude product. Clear crystals of pure **2** were obtained by slow cooling of a hot saturated toluene solution. Anal. Calcd for $GeC_{40}H_{38}N_2O_2$ (**2**): C, 73.72; H, 5.87; N, 4.29 Found: C, 71.60; H, 5.78; N, 3.62. ^1H NMR (C_6D_6, 30 °C): δ 1.51 (s, N*Me*$_2$), 6.8-7.5 (aromatics).

$Sn(OAr')_2(NMe_2)_2$ (3). To a stirred solution of $Sn(NMe_2)_4$ (0.50 g, 1.70 mmol) in benzene (10 cm^3) was added dropwise a solution of 2,6-di-*tert*-butyl-4-methylphenol (HOAr′, 0.75 g, 3.4 mmol) in benzene (10 cm^3). The resulting clear, bright yellow solution was stirred for 12 h before the solvent was removed in vacuuo. The crude yellow product was taken up in the minimum amount of hot hexane and the solution slowly cooled to yield large yellow crystals. Anal. Calcd for $SnC_{34}H_{58}N_2O_2$ (**3**): C, 63.26; H, 9.05; N, 4.34. Found: C, 59.67; H, 9.41; N, 3.89. ^1H NMR (C_6D_6, 30 °C): δ 1.47 (s, *Bu*^{t}), 2.25 (s, 4-*Me*), 2.36 (s, SnN*Me*$_2$, $^2J(^{119}Sn-^1H)$ = 74 Hz), 6.91 (s, *m*-H).

Crystallographic Studies. Selected crystallographic data are collected in Table VIII. The data collection and refinement were carried out by using the routine procedures adopted by the Purdue Chemistry Department Crystallographic Center. A suitable crystal of each of the three compounds was examined under deoxygenated Nujol and mounted in an appropriately sized glass capillary surrounded by epoxy resin. In the case of **3** a 10% loss of intensity of standards was observed during data collection and a linear correction was applied. For the germanium compounds **1b** and **2** no decay was observed and no correction for estimation was applied. For the tin compound **1** an empirical absorption correction was applied. The positions of the hydrogen atoms were calculated by assuming idealized geometry and a carbon-hydrogen bond distance of 0.95 Å. For the methyl groups, one hydrogen was located in a difference Fourier map. This position was idealized, and the remaining positions were calculated. No hydrogen atoms were refined. For compound **2** the other enantiomorph yielded $R = 0.044$ and $R_w = 0.055$ with GOF = 1.788 so that the enantiomorph reported here is correct.

Acknowledgment. We thank the National Science Foundation (Grant CHE-8915573) for support of this research.

Supplementary Material Available: Tables of crystallographic data, thermal parameters, full bond distances and angles, and hydrogen positional and thermal parameters (34 pages); listings of observed and calculated structure factors (57 pages). Ordering information is given on any current masthead page.

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Polymeric Xenon Materials: $(-Xe-O-TeF_4-O-)_n$. Crystal Structure of $HF \cdot HO-TeF_4-O-Xe^+AsF_6^-$

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XeF_2 reacts with *cis*-$(HO)_2TeF_4$ to form a yellow, insoluble polymer $(XeO_2TeF_4)_n$ that is stable to about 80 °C. Reaction of *cis*-$(HO)_2TeF_4$ with $Xe_2F_3^+AsF_6^-$ affords a material of the composition $FXe-O-TeF_4-O-Xe^+AsF_6^-$. Recrystallization of this in anhydrous HF affords a crystalline material $HF \cdot HO-TeF_4-O-Xe^+AsF_6^-$. According to its crystal structure (a = 504.74 (4), b = 758.93 (5), c = 1387.25 (7) pm; α = 90.14 (5), β = 95.74 (5), γ = 99.67 (6)°; $P\bar{1}$; Z = 2) the coordination number of xenon is closer to 1 than in any other known xenon compound.

Introduction

Substitution chemistry on xenon fluorides, even on the most stable xenon difluoride, is limited to ligands with high electronegativity. Greatest stability has been observed with the oxygen ligands $-OTeF_5$ and $-OSeF_5$, less so with $-O-SO_2F$ and others.[1] Nitrogen ligands are so far limited to the $-N(SO_2F)_2$ and $-N(SO_2CF_3)_2$ groupings and a few members of the *S*-triazine family.[2-4] Compounds that have nitrogen donor functions to xenon such as $[FXe-NCH]^+$ or $[FXe-NC-CH_3]^+$ have been prepared recently also.[5] The bonding to carbon is even more limited. While $Xe(CF_3)_2$ has never been fully characterized,[6] there is now clear evidence for the existence of the $^+Xe-C_6F_5$ cation including a crystal structure.[7,8]

Reaction of XeF_2 with bidentate ligands poses a challenge, since bidentate ligands having enough electronegativity are very limited. The most stabilizing ligand in xenon-oxygen chemistry, $-OTeF_5$, is derived from $HOTeF_5$, which in turn is a member of a series $(HO)_nTeF_{6-n}$. All these compounds $(HO)_nTeF_{6-n}$ for n = 1-5 have been shown to exist,[9-11] but only $HO-TeF_5$ and *cis*- and *trans*-$(HO)_2TeF_4$ have been isolated in pure form.[12,13] Of these, *cis*- and *trans*-$(HO)_2TeF_4$ are here of special interest. *cis*-$(HO)_2TeF_4$ is somewhat more acidic than *trans*-$(HO)_2TeF_4$,[14] and a number of covalent derivatives such as $(ClO)_2TeF_4$, $(BrO)_2TeF_4$, and $[(CH_3)_3SiO]_2TeF_4$ have already been described with both cis and trans configurations.[12] The intermolecular anhydride $F_4TeO_2TeF_4$ and the anion *cis*-$O_2TeF_4^{2-}$ in $Na_2O_2TeF_4$ are also known.[15]

Results

We reported in a short communication that *cis*-$(HO)_2TeF_4$ reacts with XeF_2 with the formation of HF, whereas *trans*-$(HO)_2TeF_4$ does not react under the same conditions, possibly a consequence of its somewhat lower acidity. The product between

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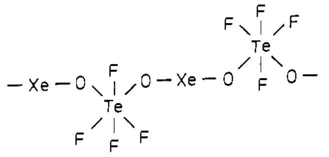
Table I. Crystallographic Data for $HF{\cdot}HO{-}TeF_4{-}O{-}Xe^+AsF_6^-$

$AsF_{11}H_2O_2TeXe$	MW 576.8
$a = 504.74$ (4) pm	$P\bar{1}$ (No 2)
$b = 758.93$ (5) pm	$T = -156$ °C
$c = 1387.25$ (7) pm	$\lambda = 170.69$ pm
$\alpha = 90.14$ (5)°	ρ(calcd) = 3.675 g·cm^{-3}
$\beta = 95.74$ (5)°	$\mu = 94.1$ cm^{-1}
$\gamma = 99.67$ (6)°	$R = 4.2\%$
$V = 521.1 \times 10^6$ pm^3	$R_w = 4.3\%$
$Z = 2$	

Table II. Atomic Parameters for $HF{\cdot}HO{-}TeF_4{-}O{-}Xe^+AsF_6^-$ (Standard Deviations in Parentheses)

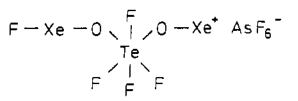
atom	*x*	*y*	*z*
Xe	0.1267 (1)	0.2631 (1)	0.1278 (1)
As	0.3961 (2)	0.2338 (2)	−0.1236 (1)
Te	0.2785 (2)	0.2576 (1)	0.3751 (1)
F	0.0339 (22)	0.7251 (11)	0.3717 (7)
F1	0.3630 (17)	0.0748 (10)	0.3032 (6)
F2	0.4698 (17)	0.1859 (10)	0.4823 (6)
F3	0.5837 (15)	0.3929 (10)	0.3370 (6)
F4	−0.0232 (15)	0.1040 (10)	0.4022 (6)
F11	0.6156 (16)	0.3277 (10)	−0.0269 (6)
F12	0.6276 (16)	0.2954 (11)	−0.2037 (7)
F13	0.1534 (15)	0.1344 (9)	−0.2145 (6)
F14	0.2770 (17)	0.4302 (9)	−0.1398 (6)
F15	0.4990 (16)	0.0318 (10)	−0.1049 (6)
F16	0.1552 (15)	0.1704 (10)	−0.0402 (6)
O1	0.0737 (18)	0.3315 (12)	0.2601 (7)
O2	0.1915 (21)	0.4408 (13)	0.4439 (7)

XeF_2 and *cis*-$(HO)_2TeF_4$ is a faintly yellow solid of the analytical composition $(XeO_2TeF_4)_n$, which is insoluble in all common solvents. It decomposes above 80 °C with evolution of xenon and oxygen, leaving behind a colorless solid that is mostly TeF_4 according to mass spectroscopy. Since the Raman spectrum contains a typical Xe–O stretching vibration at 455.5 cm^{-1} as well as TeF and TeO stretching vibrations (610–778 cm^{-1}), we concluded that the material is a polymer such as



Structural principles require the linearity of the xenon environment and the cis configuration around tellurium.

In order to isolate smaller units of this polymer, we have tried to vary the synthetic route. Replacing XeF_2 by $Xe_2F_3^+AsF_6^-$ afforded a compound of the composition $Xe_2FO_2TeF_4AsF_6$. This is a stable yellow solid that is insoluble in chlorofluorocarbons or SO_2FCl, decomposes at −70 °C in SO_2 and at −40 °C in CH_3CN, but dissolves readily in HF. The Raman spectrum of the solid shows a very strong band at 484.5 cm^{-1}, which is assigned as ν_{XeO}, a band at 677 cm^{-1}, attributable to ν_1 of AsF_6^-, and bands at 641, 667, and 762 cm^{-1} (ν_{TeF} and ν_{TeO}). The structure of the material may well be



The only known compound that it may be compared with is $[FXe{-}O{-}SOF{-}O{-}XeF]^+AsF_6^-$. This is formed by reacting $FXe{-}OSO_2F$ with AsF_5.[16,17] Here, however, confirmation of the molecular structure was finally done by X-ray crystallography.[18] Yellow solutions of $FXe{-}O{-}TeF_4{-}O{-}Xe^+AsF_6^-$ in liquid HF show a ^{125}Te NMR spectrum of the A_2B_2 or A_2BC type and a single, broad ^{129}Xe resonance, while the ^{19}F NMR resonance is largely obscured by the intense and broad HF signal. Cooling the solutions in HF afforded yellow platelike crystals that evolve HF at +5 °C, while the remaining solid is ill-defined. The crystal structure (at −156 °C) of this crystalline material is shown in Figure 1; see also Tables I–III. The formula $HF{\cdot}HO{-}TeF_4{-}O{-}Xe^+AsF_6^-$ was clearly established, the homologous compound to the known $F_5Te{-}O{-}Xe^+AsF_6^-$, which has been determined spectroscopically.[19-21]

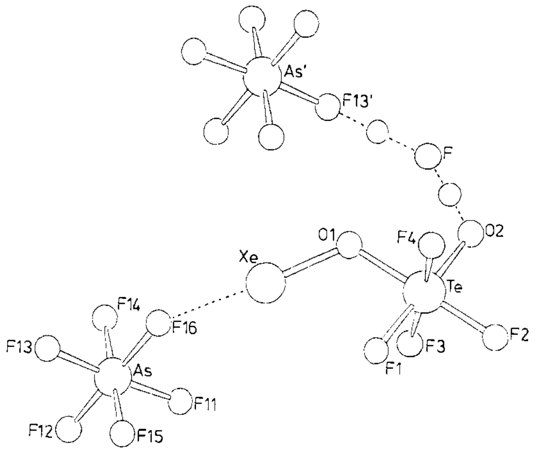


Figure 1. View of $HF{\cdot}HO{-}TeF_4{-}O{-}Xe^+AsF_6^-$. A second AsF_6^- is included to show the bridging through the HF molecule. The proton positions have not been obtained crystallographically but are drawn in assumed positions.

Table III. Bond Distances (pm) and Angles (deg) (Standard Deviations in Parentheses)

Distances			
Xe–F16	245.8 (8)	As–F16	177.1 (7)
Xe–O1	196.2 (9)	Te–F1	183.8 (7)
As–F11	172.2 (8)	Te–F2	182.6 (8)
As–F12	170.2 (8)	Te–F3	182.8 (7)
As–F13	174.2 (7)	Te–F4	182.7 (7)
As–F14	170.6 (7)	Te–O1	194.9 (9)
As–F15	171.2 (7)	Te–O2	182.1 (9)

Angles			
F11–As–F12	92.4 (4)	F1–Te–F2	90.3 (3)
F11–As–F13	175.1 (4)	F1–Te–F3	86.2 (4)
F11–As–F14	90.2 (4)	F1–Te–F4	87.4 (4)
F11–As–F15	90.8 (4)	F1–Te–O1	89.2 (3)
F11–As–F16	87.5 (4)	F1–Te–O2	178.7 (4)
F12–As–F13	92.5 (4)	F2–Te–F3	91.1 (4)
F12–As–F14	90.8 (4)	F2–Te–F4	90.5 (4)
F12–As–F15	92.2 (4)	F2–Te–O1	179.4 (3)
F12–As–F16	179.8 (1)	F2–Te–O2	91.0 (4)
F13–As–F14	89.5 (4)	F3–Te–F4	173.4 (4)
F13–As–F15	89.3 (4)	F3–Te–O1	89.1 (4)
F13–As–F16	87.6 (4)	F3–Te–O2	93.6 (4)
F14–As–F15	176.8 (4)	F4–Te–O1	89.2 (4)
F14–As–F16	89.1 (4)	F4–Te–O2	92.8 (4)
F15–As–F16	87.9 (4)	O1–Te–O2	89.6 (4)
Xe–F16–As1	133.0 (4)	F16–Xe–O1	175.6 (3)
Te–O1–Xe	123.3 (4)		

Obviously solvolysis has occurred with HF. The molecule $HF{\cdot}HO{-}TeF_4{-}O{-}Xe^+AsF_6^-$ exhibits a few features that need comment. The shape of the molecule consisting of a *cis*-O_2TeF_4 fragment and of a single coordinated xenon atom is obvious. The latter point is remarkable: Crystal structures of xenon(II) cations are known, e.g. $XeF^+Sb_2F_{11}^-$,[22] $XeF^+AsF_6^-$,[23] and others. In these

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cases xenon is bonded closer to the remaining fluorine atom than in XeF_2, while there is a secondary contact to one of the fluoride atoms from the anion in a linear geometry. It has been established that the longer this "bridging" linear cation–anion contact, the shorter is the bonding between xenon and the covalently bonded fluorine.

$$L{-}Xe^{+}\cdots F{-}XF_5^{-} \qquad L{-}\overset{+}{Xe}\cdots\overset{-}{F}\cdots XF_5 \qquad L{-}Xe{-}F\cdots XF_5$$

The left formula represents the ionic case, the middle the bridging situation, and the right a molecular adduct such as $XeF_2{\cdot}IF_5$.[24] All structurally characterized derivatives of xenon lie within the two extreme bonding situations. $HF{\cdot}HO{-}TeF_4{-}O{-}Xe^+AsF_6^-$ represents the case closely approaching the pure ionic case as in the left formula.

The $Xe{\cdots}F{-}AsF_5$ contact is 245.8 pm, as compared to 234.0 pm in $XeF^+Sb_2F_{11}^-$ and 221.2 ppm in $XeF^+AsF_6^-$. Also, the special As–F bond length of the "bridging" fluorine atom is 177.1 pm, only ≈7 pm longer than the shorter AsF bond lengths. In $XeF^+AsF_6^-$ this difference is about 13.3 pm. On the other hand, the Xe–O bond length is markedly shorter (196.2 pm) than in other xenon(II)–oxygen species (216 pm in $F{-}Xe{-}O{-}SO_2F$),[25] 212 pm in $F_5Se{-}O{-}Xe{-}OSeF_5$).[26]

A closer look shows that the xenon-bonded O–Te bond is slightly longer than the hydrogen-bonded O–Te bond. Obviously the strong participation of the first oxygen in the xenon–oxygen bond decreased its ability for bonding toward tellurium. The HF molecule bridges the tellurium center with another AsF_6^-, since there is a contact between the hydrogen atom and a fluorine atom on arsenic. This special As–F bond is also very little (≈3 pm) longer than the four shorter As–F bonds. The Xe···F contact can therefore be described just a little stronger than this H···F contact.

Experimental Section

Spectra. Infrared spectra were obtained on a Perkin-Elmer 883 infrared spectrophotometer, either on gases, pressure 1–10 Torr, or on mulls in either halocarbon or mineral oil; the window material most commonly used was KCl. Raman spectra were taken on a Cary 82 instrument using an Ar laser for excitation (Spectra-Physics). Mass spectra were obtained on either a Varian 711 or MAT 112S high-resolution mass spectrometer using a solid inlet probe. Only the most important ions are reported. The mass numbers are given for ^{130}Te. The intensities include all isotopes. The ^{19}F and ^{1}H NMR spectra were recorded on a JEOL FX 90Q nuclear magnetic resonance spectrometer using CCl_3F and $(CH_3)_4Si$, respectively, as external standards. Resonances appearing upfield from CCl_3F in the ^{19}F NMR spectra are designated negative. The ^{125}Te and ^{129}Xe were recorded on a JEOL FX 90Q NMR spectrometer using $(HO)_6Te/H_2O$ and $XeOF_4$ as external standards. Elemental analyses were obtained from Beller Microanalytical Laboratory in Göttingen, West Germany.

Reagents. XeF_2,[27] $Xe_2F_3^+AsF_6^-$,[28] and $(CH_3)_3Si{-}O{-}TeF_5$[29] were prepared according to literature methods. All other reagents were taken from laboratory stock and were purified, where necessary, before use.

Preparation of *cis*-$[(H_3C)_3SiO]_2TeF_4$. Isomerically pure material is formed by the following procedure: A 117.5-g (0.38-mol) sample of $(H_3C)_3Si{-}OTeF_5$, 67.2 g (0.42 mol) of $O[Si(CH_3)_3]_2$, and 0.5 mL of H_2O are refluxed at 100 °C for 132 h. The reflux condensor is held at 30 °C in order not to condense the $(H_3C)_3SiF$ formed in the reaction. Every 24 h another 0.5 mL of H_2O is added. Distillation on a spinning-band column (30-cm length) at normal pressure gives unreacted $[(H_3C)_3Si]_2O$ at 100 °C and then unreacted $(H_3C)_3Si{-}OTeF_5$ at 37–38 °C/100 mbar. The final product at 87 °C/20 mbar is 97.4 g (67% yield) of *cis*-$[(CH_3)_3SiO]_2TeF_4$ as a colorless liquid, mp −10 °C. ^{19}F NMR (neat): a_2b_2 pattern, $\delta_a = -28.0$ ppm, $\delta_b = -35.1$ ppm, $J_{ab} = 161$ Hz, $J(^{125}Te{-}F) = 3391$ Hz. ^{1}H NMR (neat): $\delta = 0.56$ ppm. IR (liquid): 2985 (w), 1765 (w), 1400 (m), 1264 (s), 940 (w), 895 (m), 855 (m), 768 (m), 695 (s), 680 (w), 500 cm^{-1} (vw). Raman (liquid): 2966 (21, dp), 2906 (69, p), 1417 (14, dp), 1263 (10, p), 939 (21, p), 857 (12, p), 766 (19, dp), 695 (6, dp), 683 (78, p), 639 (100, p), 613 (41, p), 495 (71, p), 357 (9, p), 326 (10, p), 311 (13, p), 268 (6, p), 251 (16, p), 226 (3, p), 199 (3, p), 162 cm^{-1} (6, p). Anal. Calcd for $C_6H_{18}Si_2O_2TeF_4$: C, 18.87; H, 4.71; Te, 33.42; F, 19.89. Found: C, 18.60; H, 4.49; Te, 33.02; F, 19.70.

Preparation of *cis*-$(HO)_2TeF_4$. A 25-g (65.5-mmol) sample of *cis*-$[(H_3C)_3Si{-}O]_2TeF_4$ is placed into a Teflon FEP reaction vessel, and 10.5 g (575 mmol) of anhydrous HF is condensed into the reactor by using a stainless steel vacuum line. The mixture is stirred at 50 °C for 48 h. At 0 °C the volatile materials HF and $(H_3C)_3SiF$ are pumped off. The solid residue is sublimed at room temperature under vacuum onto a −5 °C finger. A 14.9-g amount (96% yield) of *cis*-$(HO)_2TeF_4$ is obtained as a colorless, crystalline solid, mp 106 °C. ^{19}F NMR (acetone-d_6): a_2b_2 pattern, $\delta_a = -33.1$ ppm, $\delta_b = -41.8$ ppm, $J_{ab} = 154$ Hz. ^{1}H NMR (acetone-d_6): $\delta = 11.14$ ppm. IR (Nujol): 3600–3490 (m, br), 1625 (w), 1100–1000 (m), 730 (m), 720 (vw), 680 (vw), 650 (vw), 630 (vw), 330 cm^{-1} (m). Raman (solid): 731 (12), 711 (5), 679 (100), 646 (11), 635 (5), 317 (51), 186 cm^{-1} (18). Anal. Calcd for $H_2O_2TeF_4$: H, 0.84; Te, 53.71; F, 31.97. Found: H, 1.17; Te, 51.87; F, 31.2.

Preparation of $(-Xe{-}O{-}TeF_4{-}O-)_n$. A 1-g (5.6-mmol) sample of XeF_2 is weighed into a Teflon FEP reaction vessel. Also 1.4 g (5.9 mmol) of *cis*-$(HO)_2TeF_4$ contained in a smaller FEP tube is placed into the vessel, so that no mixing occurs. In vacuo, 10 mL of anhydrous *n*-$C_4F_9{-}SO_2F$ is condensed at −196 °C into the reaction vessel. The two solid starting materials are mixed by shaking at room temperature. After $^1/_2$ h of stirring, all volatile materials are pumped off. The residue contains 2.0 g (92% yield) of faintly yellow $(-Xe{-}O{-}TeF_4{-}O)_n$, 80 °C dec. Raman (solid): 917 (1), 778.5 (15), 725 (5), 662.5 (12), 610 (13), 455.5 (100), 392.5 (10), 286 (2), 230 (10, sh), 220 (22), 118 cm^{-1} (75). Anal. Calcd for XeO_2TeF_4: Xe, 35.8; Te, 34.8; F, 20.7. Found: Xe, 34.9; Te, 35.1; F, 20.8.

***cis*-$FXeO{-}TeF_4{-}O{-}Xe^+AsF_6^-$.** In a glovebox 3.2 g (6.3 mmol) of $Xe_2F_3^+AsF_6^-$ was weighed into an FEP reactor. Also an Teflon FEP tube containing 1.5 g (6.3 mmol) of *cis*-$(HO)_2TeF_4$ is placed into the reactor as in the experiment above. With use of an all-metal vacuum line and liquid-nitrogen cooling, 15 mL of anhydrous HF is condensed into the reactor. The $Xe_2F_3^+AsF_6^-$ is dissolved in HF at −78 °C, and then, by shaking, the *cis*-$(HO)_2TeF_4$ is mixed with the solution. After being slowly warmed to 0 °C, the mixture is kept at 0 °C until a clear, light yellow solution is obtained. All volatiles are pumped off until no more loss of weight is observed. A 3.8-g amount (86% yield) of a light yellow solid is obtained, mp 89 °C dec. The solid is stable at room temperature; however, it is very moisture sensitive. ^{125}Te NMR (HF): $\delta = -82.5$ ppm, $J(^{125}Te{-}F) = 3502$, 3519 Hz. ^{129}Xe NMR (HF): $\delta = -913$ ppm. Raman (solid): 762 (s), 667 (m), 641 (s), 603 (w), 484.5 (vs), 426.5 (s), 380 (m), 360.5 (m), 317.5 (m), 304 (m), 275.5 (s), 226 (w), 174.5 (s), 144.5 (s), 98.5 (vs), 83.5 cm^{-1} (vs). Anal. Calcd for $Xe_2O_2TeAsF_{11}$: F, 29.6; As, 10.6; Te, 18.1; Xe, 37.2. Found: F, 32.3; As, 11.6; Te, 19.6; Xe, 36.7.

Crystal Structure of $HF{\cdot}HO{-}TeF_4{-}O{-}Xe^+AsF_6^-$. Keeping $FXe{-}O{-}TeF_4{-}O{-}Xe^+AsF_6^-$ for prolonged time in liquid, anhydrous HF at −35 °C afforded orange crystals. Under dry nitrogen a platelike crystal of 0.6 × 0.5 × 0.2 mm size was mounted at a temperature below −100 °C on the tip of a goniometer and cooled to −156 °C. A description of the technique employed for selecting and mounting the crystal can be found in ref 30. The crystal structure was solved with direct methods and refined with SHELX. All atoms were finally refined anisotropically. Hydrogen positions were assumed; see Figure 1. DIFABS was used for an absorption correction. For further experimental data, see Table II. The results of the crystal structure determination are collected in Tables I–III and Figure 1.

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Supplementary Material Available: Listings of full experimental conditions and anisotropic temperature factors (2 pages); a table of observed and calculated structure factors (10 pages). Ordering information is given on any current masthead page.

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