Synthesis, structure, and properties of piperidinium 3-(4-chlorophenyl)-2-cyano-3-(2-hydroxy-4,4-dimethyl-6-oxocyclohex-1-enyl)thiopropionimidate

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Condensation of 4-chlorobenzaldehyde with cyanothioacetamide and dimedone in the presence of piperidine afforded piperidinium 3-(4-chlorophenyl)-2-cyano-3-(2-hydroxy-4,4-dimethyl-6-oxocyclohex-1-enyl)thiopropionimidate. This salt was studied by X-ray diffraction analysis and used in the synthesis of the previously unknown partially hydrogenated 2-alkylthioquinolin-2-ones.

Key words: condensation, 4-chlorobenzaldehyde, dimedone, cyanothioacetamide, cyclohexen-1-ol, quinolinones, alkylation, X-ray diffraction analysis.

Previously,[1–4] it has been found that the reactions of arylmethylidenecyanothioacetamides with dimedone or the reactions of aromatic aldehydes with cyanothioacetamide and dimedone in the presence of secondary amines afforded salts which have been described previously as substituted ammonium 2-(1-aryl-2-cyano-2-thiocarbamoylethyl)-5,5-dimethyl-3-oxocyclohex-1-enolates. Cyclization of the latter to form substituted hydrogenated 2-aminobenzo[*b*]pyrans and hexahydroquinoline-2-thiolates was studied. In addition, substituted 9b-hydroxytetrahydroindeno[1,2-*b*]pyridines were synthesized by condensation of 2-arylmethylidene-1,3-indanediones with cyanothioacetamide in the presence of piperidine followed by the reaction with alkyl halides. The conversions of the resulting compounds were studied.[5] Substituted sulfur-containing 3-cyanooctahydroquinolines are unknown.

We synthesized salt **4** by condensation of 4-chlorobenzaldehyde (**1**) with cyanothioacetamide (**2**) and dimedone (**3**) in the presence of piperidine. It appeared that salt **4** is piperidinium 3-(4-chlorophenyl)-2-cyano-3-(2-hydroxy-4,4-dimethyl-6-oxocyclohex-1-enyl)thiopropionimidate (Scheme 1). The molecular and crystal structure of this key intermediate in the synthesis of the previously unknown partially hydrogenated 2-alkylthio-3-cyanoquinolin-5-ones was established by X-ray diffraction analysis.

The principal geometric parameters of molecule **4** are given in Table 1. The overall view of **4** is shown in Fig. 1. Judging from the C(17)—S(1) and C(17)—N(1) bond lengths, salt **4** contains the —C(=NH)—S$^-$ group. The cyclohexene C(8)—C(13) ring is substantially nonplanar. The C(8), C(9), C(10), C(12), and C(13) atoms are coplanar within 0.009 Å. The O(1) and O(2) atoms deviate from this plane by -0.020 and -0.055 Å, respectively, whereas the C(11) atom deviates from this plane by 0.643 Å. The torsion angles (τ) in this ring are as follows:

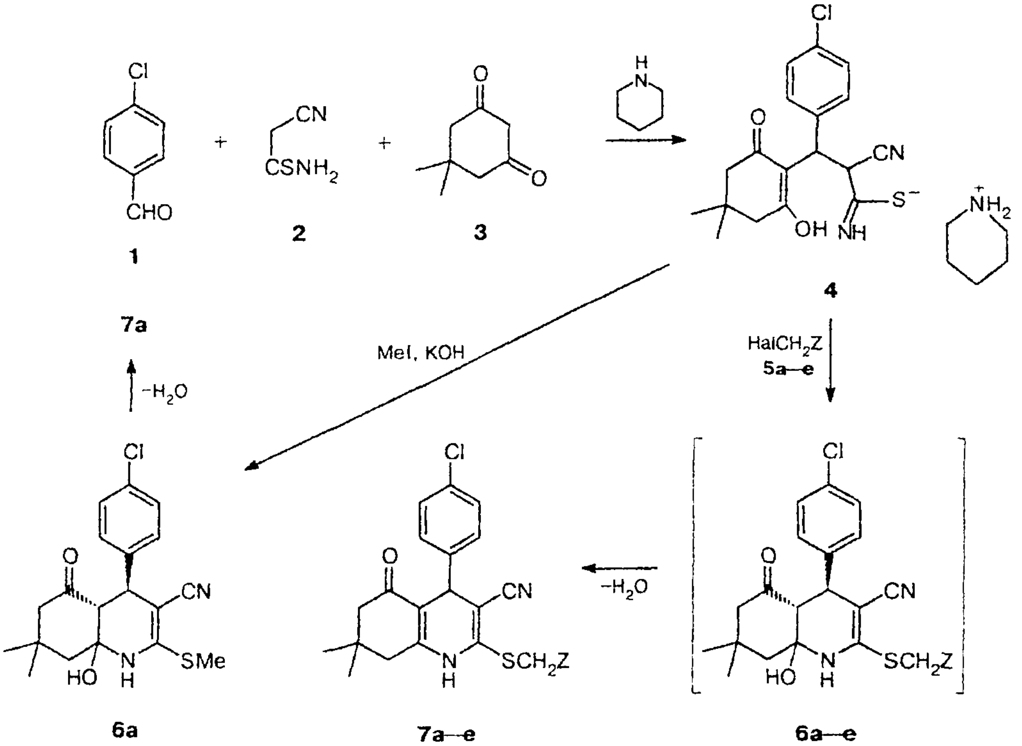
Angle	τ/deg
C(13)—C(8)—C(9)—C(10)	−1.77
C(8)—C(9)—C(10)—C(11)	−26.72
C(9)—C(10)—C(11)—C(12)	51.61
C(10)—C(11)—C(12)—C(13)	−51.31
C(11)—C(12)—C(13)—C(8)	27.33
C(12)—C(13)—C(8)—C(9)	1.03

The modified Cremer—Pople parameters[6] calculated for this ring (S = 0.70, θ = 39.83, ψ = 0.54°) indicate that this ring adopts a slightly distorted half-chair conformation. The O(1)—C(9) (1.263(10) Å), C(9)=C(8) (1.394(11) Å), C(8)—C(13) (1.388(11) Å), and C(13)=O(2) (1.272(9) Å) bond lengths are indicative of a substantial delocalization of the electron density in the O(1)—C(9)=C(8)—C(13)=O(2) moiety due to conjugation.[7] The shortened O(1)...C(1) and O(2)...C(1) contacts (2.838(9) and 2.877(10) Å, respectively; twice the mean value of the van der Waals radii of the O and C atoms is 2.97 Å[8]) lead to an increase in the O(1)—C(9)—C(8) and O(2)—C(13)—C(8) bond angles (123.8(7)° and 122.0(7)°, respectively) compared to the O(1)—C(9)—C(10) and O(2)—C(13)—C(12) bond angles (116.4(7)° and 117.2(7)°, respectively). The remaining geometric parameter of anion **4** have standard values.

In the crystal, molecules **4** are linked in centrosymmetrical dimers through the O(1)...H(1)—N(1) hy-

Translated from *Izvestiya Akademii Nauk. Seriya Khimicheskaya*, No. 4, pp. 733—737, April, 2000.

Scheme 1



Z = H (**a**), Ph (**b**), COOMe (**c**), CH_2=CCMe (**d**), Me (**e**)
Hal = I (**5a**,**e**), Cl (**5b**—**d**)

drogen bonds (O...N, 2.805(9) Å; O...H, 2.15(5) Å; N(1)—H(1), 0.68(6) Å; O(1)...H(1)—N(1), 162(3)°; Fig. 2). In addition, the shortened O(1)...N(3A), O(1)...N(3b), and O(3)...N(2) distances (2.80(2), 2.63(3), and 3.07(1) Å, respectively) suggest that H bonds between the cation and the anion as well as H bonds between the anion and the ethanol molecule of solvation can occur.

We found that heating of compound **4** with halides **5a**—**e** in ethanol over a short period afforded unstable octahydroquinolinones **6**, which were partly converted into dehydrated analogs **7** in the course of the reaction. In these cases, mixtures of products **6** and **7** were formed in different ratios. Subsequent boiling of these mixtures gave individual compounds **7**. The results of physicochemical studies of the latter (see the Experimental section) agree with the data reported for the isostructural analogs of these quinolinones.[4]

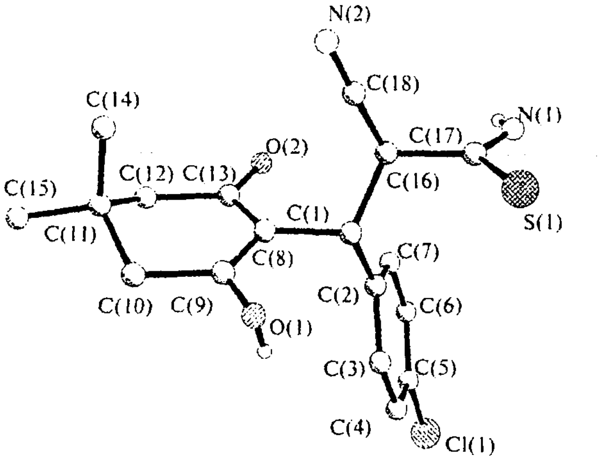


Fig. 1. Overall view of molecule **4**.

Compound **6a** was prepared in high yield by the reaction of salt **4** with iodomethane in the presence of KOH. In the ^1H NMR spectrum of sulfide **6a**, the signals for the H(4) and H(4a) protons are observed as doublets with spin-spin coupling constants of

Table 1. Principal bond lengths (*d*) and bond angles (ω) in molecule **4**

Bond	*d*/Å	Angle	ω/deg
S(1)—C(17)	1.657(9)	C(9)—N(8)—C(13)	120.0(7)
N(1)—C(17)	1.292(11)	C(8)—C(9)—C(10)	119.9(8)
N(2)—C(18)	1.128(12)	C(9)—C(10)—C(11)	115.3(7)
O(1)—C(9)	1.263(10)	C(10)—C(11)—C(12)	107.1(7)
O(2)—C(13)	1.272(9)	C(11)—C(12)—C(13)	113.5(7)
C(8)—C(9)	1.394(11)	C(8)—C(13)—C(12)	120.8(7)
C(8)—C(13)	1.388(11)	S(1)—C(17)—N(1)	123.4(6)
C(9)—C(10)	1.509(10)	S(1)—C(17)—C(16)	121.4(6)
C(10)—C(11)	1.499(12)	N(1)—C(17)—C(16)	115.2(7)
C(11)—C(12)	1.522(11)	N(2)—C(18)—C(16)	176.9(13)
C(12)—C(13)	1.520(11)		
Cl(1)—C(5)	1.743(10)		

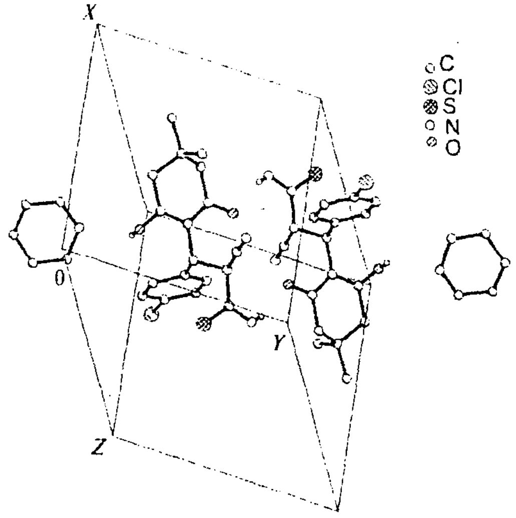


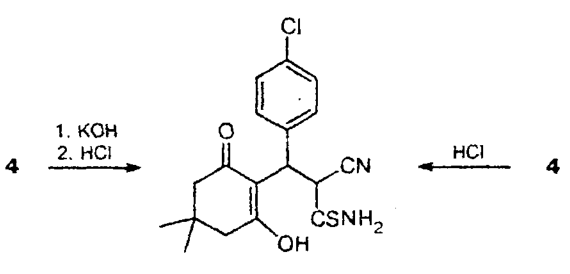
Fig. 2. Crystal packing of compound **4**. Hydrogen bonds are indicated by broken lines.

12.2 Hz, which is indicative of their *trans*-diaxial arrangement, and, correspondingly, the Ar and CO groups are in *trans*-diequatorial orientations.

Hydroxyoctahydroquinolinone **6a** was dehydrated upon recrystallization from ethanol to form more stable hexahydroquinolinone **7a**.

Treatment of compound **4** with 10% aqueous KOH apparently did not afford the corresponding intramolecular cyclization product, because subsequent treatment of the reaction mixture with hydrochloric acid yielded free Michael adduct **8** (Scheme 2). The latter was also formed by the direct reaction of salt **4** with hydrochloric acid. The ^{1}H NMR spectrum of compound **8** has signals for two diastereomers. The ratio of their integrated intensities is 4 : 1.

Scheme 2



8

Experimental

The IR spectra were recorded on an IKS-29 spectrophotometer in Nujol mulls. The ^{1}H NMR spectra were measured on a Bruker WP-100 SY instrument (100 MHz) in DMSO-d_6 with Me_4Si as the internal standard. The purity of the compounds was monitored by TLC on Silufol UV-254 plates in the 3 : 5 acetone—hexane system.

Piperidinium 3-(4-chlorophenyl)-2-cyano-3-(2-hydroxy-4,4-dimethyl-6-oxocyclohex-1-enyl)thiopropionimidate (4). Cyanothioacetamide (**2**) (2 g, 20 mmol) was added with stirring to a mixture of 4-chlorobenzaldehyde (**1**) (2.81 g, 20 mmol) and piperidine (3 drops) in ethanol (40 mL) at ~20 °C. After 5 min, dimedone (**3**) (2.8 g, 20 mmol) and piperidine (2.47 mL, 25 mmol) were successively added with stirring. After dissolution of the initial reagents, the reaction mixture was filtered through a paper filter and kept for 12 h. The crystalline precipitate that formed was filtered off, washed with ethanol and hexane, and dried at 80 °C for 1 h. Compound **4** was obtained as a solvate with ethanol. The yield was 8.24 g (92%), m.p. 187—189 °C. Found (%): C, 61.51; H, 6.85; N, 9.18; S, 7.24. $C_{23}H_{30}ClN_3O_2S$. Calculated (%): C, 61.66; H, 6.75; N, 9.38; S, 7.16. IR, ν/cm^{-1}: 3150—3270 (NH, OH); 2240 (CN); 1710 (CO). ^{1}H NMR, δ: 0.93 (br.s, 6 H, 2 Me); 1.60 (m, 6 H, $(CH_2)_3$); 1.93 (br.s, 4 H, 2 CH_2 (cyclohexene)); 2.98 (m, 4 H, $N(CH_2)_2$); 5.03 (d, 1 H, C(3)H, ^3J = 11.9 Hz); 5.85 (d, 1 H, C(2)H, ^3J = 11.9 Hz); 7.13 and 7.49 (both d, 4 H, Ar, ^3J = 8.2 Hz); 7.22 (br.s, 1 H, OH or NH); 9.4 (br.s, 1 H, NH or OH).

4-(4-Chlorophenyl)-3-cyano-8a-hydroxy-7,7-dimethyl-2-methylthio-1,4,4a,5,6,7,8,8a-octahydroquinolin-5-one (6a). A 10% aqueous solution of KOH (2.8 mL, 5 mmol) was added with stirring to a suspension of salt **4** (2.24 g, 5 mmol) in 80% ethanol (30 mL). After 5 min, iodomethane (**5a**) (0.31 mL, 5 mmol) was added. After 20 min, the precipitate that formed was filtered off and washed with ethanol and hexane. The yield of compound **6a** was 1.41 g (75%), m.p. 285—287 °C. Found (%): C, 60.42; H, 5.43; N, 7.54; S, 8.34. $C_{19}H_{21}ClN_2O_2S$. Calculated (%): C, 60.55; H, 5.62; N, 7.43; S, 8.51. IR, ν/cm^{-1}: 3270—3390 (NH, OH); 2195 (CN); 1725, 1740 (CO). ^{1}H NMR, δ: 0.9 and 1.03 (both s, 6 H, 2 Me); 1.87 and 2.25 (both d, 2 H, $C(8)H_2$, ^2J = 13 Hz); 2.46 (s, 2 H, $C(6)H_2$); 2.53 (s, 3 H, SMe); 3.01 (d, 1 H, C(4a)H, ^3J = 12.2 Hz); 3.37 (d, 1 H, C(4)H, ^3J = 12.2 Hz); 5.75 (s, 1 H, OH); 7.18 and 7.41 (both d, 4 H, Ar, ^3J = 8.1 Hz); 7.73 (s, 1 H, NH).

4-(4-Chlorophenyl)-3-cyano-7,7-dimethyl-2-methylthio-1,4,5,6,7,8-hexahydroquinolin-5-one (7a). ***A.*** A mixture of salt **4** (2.24 g, 5 mmol) and iodomethane (**5a**) (0.31 mL, 5 mmol) in 80% ethanol (30 mL) was refluxed for 10 min and filtered through a paper filter. After 12 h, the precipitate that formed upon storage (~20 °C) was filtered off and washed with ethanol and hexane.

B. Compound **6a** (0.75 g, 2 mmol) was recrystallized from ethanol (15 mL). After 12 h, the precipitate that formed was filtered off and washed with hexane.

The yields of product **7a** prepared according to procedures ***A*** and ***B*** were 1.38 g (77%) and 0.64 g (89%), respectively, m.p. 281—283 °C. Found (%): C, 63.42; H, 5.44; N, 7.57; S, 8.99. $C_{19}H_{19}ClN_2OS$. Calculated (%): C, 63.59; H, 5.34; N, 7.81; S, 8.93. IR, ν/cm^{-1}: 3265—3381 (NH); 2190 (CN); 1740, 1753 (CO). ^{1}H NMR, δ: 0.9 and 1.03 (both s, 6 H, 2 Me); 2.03 and 2.32 (both d, 2 H, $C(8)H_2$, ^2J = 13.3 Hz); 2.45 (s, 2 H, $C(6)H_2$); 2.52 (s, 3 H, SMe); 4.50 (s, 1 H, C(4)H); 7.19 and 7.43 (both d, 4 H, Ar, ^3J = 8.2 Hz); 9.66 (s, 1 H, NH).

Compounds **7b—e** were prepared analogously to **7a** according to method ***A*** with the use of halides **5b—e**, respectively.

2-Benzylthio-4-(4-chlorophenyl)-3-cyano-7,7-dimethyl-1,4,5,6,7,8-hexahydroquinolin-5-one (7b). The yield was 1.68 g (77%), m.p. 174—176 °C. Found (%): C, 69.14; H, 5.56; N, 6.21; S, 7.45. $C_{25}H_{23}ClN_2OS$. Calculated (%): C, 69.03; H, 5.33; N, 6.44; S, 7.37. IR, ν/cm^{-1}: 3240 (NH); 2200 (CN); 1620 (CO). ^{1}H NMR, δ: 0.92 and 1.02 (both s, 6 H, 2 Me); 2.05 and 2.22 (both d, 2 H, $C(8)H_2$, ^2J = 16.5 Hz); 2.45 (s, 2 H, $C(6)H_2$); 4.30 (s, 2 H, SCH_2); 4.38 (s, 1 H, C(4)H); 7.00 (d, 2 H, Ar, ^3J = 8.2 Hz); 7.30 (m, 7 H, 2 Ar); 9.80 (s, 1 H, NH).

4-(4-Chlorophenyl)-3-cyano-7,7-dimethyl-2-methoxycarbonylmethylthio-1,4,5,6,7,8-hexahydroquinolin-5-one (7c). The yield was 1.42 g (68%), m.p. 182—184 °C. Found (%): C, 60.77; H, 4.89; N, 6.92; S, 7.77. $C_{21}H_{21}ClN_2O_3S$. Calculated (%): C, 60.50; H, 5.08; N, 6.72; S, 7.69. IR, ν/cm^{-1}: 3245 (NH); 2192 (CN); 1750 (CO). ^{1}H NMR, δ: 0.91 and 1.03 (both s, 6 H, 2 Me); 2.06 and 2.28 (both d, 2 H, C(8)H_2, ^2J = 16.6 Hz); 2.43 (s, 2 H, C(6)H_2); 3.62 (s, 3 H, OMe); 3.97 (s, 2 H, SCH_2); 4.52 (s, 1 H, C(4)H); 7.20 and 7.38 (both d, 4 H, Ar, ^3J = 8.1 Hz); 9.81 (s, 1 H, NH).

4-(4-Chlorophenyl)-3-cyano-7,7-dimethyl-2-metallylthio-1,4,5,6,7,8-hexahydroquinolin-5-one (7d). The yield was 1.34 g (67%), m.p. 237—239 °C. Found (%): C, 66.34; H, 5.62; N, 6.93; S, 7.79. $C_{22}H_{23}ClN_2OS$. Calculated (%): C, 66.23; H, 5.81; N, 7.02; S, 8.04. IR, ν/cm^{-1}: 3252 (NH); 2190 (CN); 1620 (CO). ^{1}H NMR, δ: 0.92 and 1.01 (both s, 6 H, 2 Me); 1.80 (s, 3 H, Me); 2.03 and 2.27 (both d, 2 H, C(8)H_2, ^2J = 16.4 Hz); 2.44 (s, 2 H, C(6)H_2); 3.54 and 3.81 (both d, 2 H, SCH_2, ^2J = 13 Hz); 4.49 (s, 1 H, C(4)H); 4.81 (d, 2 H, CH_2=C, ^2J = 6.5 Hz); 7.18, 7.38 (both d, 4 H, Ar, ^3J = 8.2 Hz); 9.72 (s, 1 H, NH).

4-(4-Chlorophenyl)-3-cyano-2-ethylthio-7,7-dimethyl-1,4,5,6,7,8-hexahydroquinolin-5-one (7e). The yield was 1.04 g (56%), m.p. 240—242 °C. Found (%): C, 64.56; H, 5.63; N, 7.15; S, 8.91. $C_{20}H_{21}ClN_2OS$. Calculated (%): C, 64.42; H, 5.68; N, 7.51; S, 8.60. IR, ν/cm^{-1}: 3180 (NH); 2203 (CN); 1620 (CO). ^{1}H NMR, δ: 0.91 and 1.02 (both s, 6 H, 2 Me); 1.19 (t, 3 H, Me, ^3J = 6.7 Hz); 2.05 and 2.31 (both d, 2 H, C(8)H_2, ^2J = 16.2 Hz); 2.46 (s, 2 H, C(6)H_2); 3.03 (m, 2 H, SCH_2); 4.51 (s, 1 H, C(4)H); 7.19 and 7.40 (both d, 4 H, Ar, ^3J = 8.2 Hz); 9.74 (s, 1 H, NH).

3-(4-Chlorophenyl)-2-cyano-3-(2-hydroxy-4,4-dimethyl-6-oxocyclohex-1-enyl)thiopropionamide (8). *A.* A 10% aqueous solution of KOH (1.12 mL, 2 mmol) was added with stirring to a suspension of salt **4** (0.9 g, 2 mmol) in ethanol (15 mL). After 10 min, 10% HCl was added to the reaction mixture to pH 3. After 1 h, the precipitate that formed was filtered off and washed with ethanol and hexane.

B. A 10% HCl solution was added with stirring to a suspension of salt **4** (0.9 g, 2 mmol) in ethanol (15 mL). After 1 h, the precipitate that formed was filtered off and washed with ethanol and hexane.

The yields of product **8** prepared according to methods ***A*** and ***B*** were 0.6 g (82%) and 0.52 g (72%), respectively, m.p. 158—160 °C. Found (%): C, 59.77; H, 5.64; N, 7.47; S, 8.63. $C_{18}H_{19}ClN_2O_2S$. Calculated (%): C, 59.58; H, 5.28; N, 7.72; S, 8.84. IR, ν/cm^{-1}: 3270—3430 (NH_2, OH); 2258 (CN); 1645 (CO). ^{1}H NMR, δ: 0.95 and 1.03 (both br.s, 6 H, 2 Me); 2.07—2.49 (m, 4 H, 2 CH_2 (cyclohexene)); 4.85 (minor isomer) and 5.03 (major isomer) (both d, 1 H, C(1)H, ^3J = 8.5 Hz); 5.29 (minor isomer) and 5.41 (major isomer) (both d, 1 H, C(2)H, ^3J = 8.5 Hz); 7.12, 7.36 (major isomer), 7.23, and 7.43 (minor isomer) (all d, 4 H, Ar, ^3J = 8.2 Hz); 9.20, 9.66 (minor isomer), 9.51, and 9.55 (major isomer) (all br.s, 2 H, NH_2); 11.09 (br.s, 1 H, OH).

X-ray diffraction study of a single crystal of compound 4 of linear dimensions of 0.22×0.31×0.50 mm was carried out on an automated four-circle Enraf-Nonius CAD-4 diffractometer (λ(Mo-Kα) radiation, graphite monochromator, the ratio of the scan rates $\omega/2\theta = 1.2$, $\theta_{max} = 22°$, the segment of the reflection sphere $0 \le h \le 11$, $-12 \le k \le 10$, $-12 \le l \le 11$) at ~20 °C. A total of 5225 reflections were measured of which 3228 reflections were independent (the *R* factor of merging was 0.009). The crystals of compound **4** are triclinic, a = 10.959(2) Å, b = 11.480(2) Å, c = 11.576(2) Å, α = 92.22(3)°, β = 98.87(3)°, γ = 112.76(3)°, V = 1319.1(4) Å^3, Z = 2, d_{calc} = 1.24 g cm^{-3}, μ = 0.25 mm^{-1}, $F(000)$ = 528, space group $P1$ (No. 2). The structure was solved by the direct method and refined anisotropically by the full-matrix least-squares method with the use of the SHELXS and SHELXL-93 program packages.[9,10] The refinement was carried out with the use of 1771 reflections with $I > 2\sigma(I)$ (327 refinable parameters, 5.42 reflections per parameter, the weighting scheme $\omega = 1/[\sigma^2(F_0^2) + (0.120P)^2 + 1.242P]$, where $P = (F_0^2 + 2F_c^2)/3$, the ratio of the maximum/average shift to the error in the last cycle was 0.159/0.021). A correction for anomalous scattering was applied. Absorption in the crystal was ignored. The positions of the H atoms in the piperidinium cation and in the ethanol molecule of solvation were calculated geometrically. The remaining H atoms were located from the difference electron density synthesis. All H atoms were included in the calculations with fixed positional and thermal parameters, and only the H(1) and H(10) atoms bound to the N(1) and O(1) atoms, respectively, were refined isotropically. The final value of the *R* factor ($R_1(F)$) was 0.0804, GOF = 1.358. The residual positive and negative electron densities in the difference Fourier series were 0.52 and −0.38 e A^{-3}, respectively. It should be noted that both the piperidinium cation and the ethanol molecule of solvation are substantially disordered, which is responsible for the rather low accuracy of the determination of the geometric parameters and the high thermal parameters. The atomic coordinates were deposited with the Cambridge Structural Database.

This work was financially supported by the Russian Foundation for Basic Research (Project No. 99-03-32965).

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Received October 11, 1999