Synthesis and X-ray structural study of 1-adamantylmethyl 2-cyanoacrylate and 1,10-decanediol bis-2-cyanoacrylate

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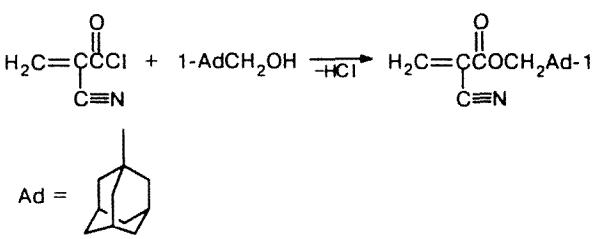
1-Adamantylmethyl 2-cyanoacrylate (**1**) was prepared by the reaction of 2-cyanoacryloyl chloride with 1-adamantylmethanol. 1,10-Decanediol bis-2-cyanoacrylate (**2**) was synthesized by transesterification of methyl 2-cyanoacrylate with 1,10-decanediol. Esters **1** and **2** were studied by X-ray structural analysis.

Key words: 2-cyanoacryloyl chloride; methyl 2-cyanoacrylate, transesterification; 1-adamantylmethyl 2-cyanoacrylate, 1,10-decanediol bis-2-cyanoacrylate, X-ray structural study.

Esters of 2-cyanoacrylic acid have been known for more than 45 years.[1] These esters are widely used in polymer chemistry as a basis for quick-acting single-component cold-setting adhesives.[2] Recently, 2-cyanoacrylates have found use in laboratory practice as highly active reagents for organic synthesis.[3,4]

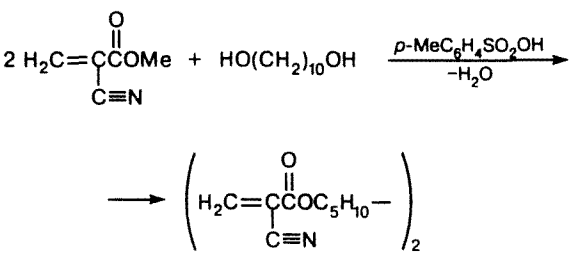
With the aim of obtaining quantitative characteristics relating the reactivities of 2-cyanoacrylates to their structures, we carried out an X-ray structural analysis of compounds of this class. In this work, we report on the synthesis and discuss the results of X-ray structural analysis of 1-adamantylmethyl 2-cyanoacrylate (**1**) and 1,10-decanediol bis-2-cyanoacrylate (**2**). Compound **1** was prepared by the reaction of 2-cyanoacryloyl chloride with 1-adamantylmethanol in analogy to the procedure described previously[5,6] (Scheme 1).

Scheme 1



Ester **1** is a crystalline colorless compound. The ^{1}H NMR spectrum of this compound is typical of esters of 2-cyanoacrylic acid. 1,10-Decanediol bis-2-cyanoacrylate **2** was synthesized by transesterification of methyl 2-cyanoacrylate with 1,10-decanediol in the presence of *p*-toluenesulfonic acid[7] according to Scheme 2.

Scheme 2



A multistage synthesis of bis-2-cyanoacrylate **2** has been reported previously.[8] The properties of the compound obtained (m.p., ^{1}H NMR spectrum) correspond to the data in the literature.[8]

The X-ray structural study demonstrated that molecules **1** and **2** have geometries typical of esters of acrylic acid (Tables 1—4).[9] However, the methylene H atoms show a pronounced acid character, apparently because of the presence of strong electron-withdrawing groups (cyano and ester groups). This accounts for the short intermolecular contacts typical of weak hydrogen bonds that are observed in the crystals of compounds **1** and **2** (Figs. 1 and 2): C—H···O=C for compound **1** [O(1′) (x, $y-1$, z)···C(3), 3.49(1) Å; O(1′) (x, $y-1$, z)···H(3A), 2.4(1) Å; O(1′)···H(3A)—C(3), 167(6)° and O(1)···C(3′), 3.43(1) Å; O(1′)···H(3E), 2.4(1) Å; O(1′)···H(3E)—C(3′), 168(6)° for two crystallographically independent molecules I and II, respectively]; C—H···N=C for com-

† Deceased in 1995.

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Table 1. Bond lengths (d) in the structure of **1**

Bond	d/Å	Bond	d/Å
Molecule I		Molecule II	
O(1)—C(1)	1.19(1)	O(1')—C(1')	1.19(1)
O(2)—C(1)	1.34(1)	O(2')—C(1')	1.34(1)
O(2)—C(4)	1.48(1)	O(2')—C(4')	1.46(1)
N(1)—C(5)	1.14(1)	N(1')—C(5')	1.12(1)
C(1)—C(2)	1.49(1)	C(1')—C(2')	1.47(1)
C(2)—C(3)	1.28(1)	C(2')—C(3')	1.32(1)
C(2)—C(5)	1.45(1)	C(2')—C(5')	1.45(1)
C(4)—C(1A)	1.51(1)	C(4')—C(1B)	1.53(1)
C(1A)—C(2A)	1.53(1)	C(1B)—C(2B)	1.54(1)
C(1A)—C(8A)	1.53(1)	C(1B)—C(8B)	1.54(1)
C(1A)—C(9A)	1.56(1)	C(1B)—C(9B)	1.54(1)
C(2A)—C(3A)	1.56(1)	C(2B)—C(3B)	1.49(1)
C(3A)—C(4A)	1.52(1)	C(3B)—C(4B)	1.54(1)
C(3A)—C(10A)	1.49(1)	C(3B)—C(10B)	1.54(1)
C(4A)—C(5A)	1.54(1)	C(4B)—C(5B)	1.52(1)
C(5A)—C(6A)	1.52(1)	C(5B)—C(6B)	1.53(1)
C(5A)—C(9A)	1.53(1)	C(5B)—C(9B)	1.50(1)
C(6A)—C(7A)	1.51(1)	C(6B)—C(7B)	1.53(1)
C(7A)—C(8A)	1.54(1)	C(7B)—C(8B)	1.53(1)
C(7A)—C(10A)	1.55(1)	C(7B)—C(10B)	1.53(1)

pound **2** [N(1) $(1+x, 1+y, z)$···C(1), 3.380(3) Å; N(1) $(1+x, 1+y, z)$···H(1A), 2.59(2) Å; N(1)···H(1A)—C(1), 138(1)° and N(1) $(-1-x, 2-y, 1-z)$···C(1), 3.477(3) Å; N(1) $(-1-x, 2-y, 1-z)$···H(1B), 2.57(2) Å; N(1)···H(1B)—C(1), 160(1)°]. Under the reaction conditions, these interactions may, apparently, promote the initial coordination of nucleophilic reagents in the vicinity of the reactive groups (coordination of proton-acceptor nucleophiles to the methylene H atoms through hydrogen bonds). Note that the analogous esters of acrylic acid can also form weak C—H...X hydrogen bonds;[10–12] however, it is difficult to estimate the relative acidities of methylene H atoms in derivatives of acrylic and cyanoacrylic acids based on structural data alone. Evidently, the cyano group additionally increases the acidity of the protons of the $=CH_2$ group and thereby enhances the reactivities of compounds **1** and **2**. However, this suggestion calls for more detailed analysis, which we plan to carry out in the future.

Experimental

The ^1H NMR spectra were recorded on a Bruker WP-200SY instrument (200.13 MHz) in C_6D_6 relative to $SiMe_4$.

1-Adamantylmethyl 2-cyanoacrylate (1). A solution of 1-adamantylmethanol (1.66 g, 0.01 mol) in toluene (10 mL) was added to a solution of 2-cyanoacryloyl chloride prepared

Table 2. Bond angles (ω) in the structure of **1**

Angle	ω/deg	Angle	ω/deg
Molecule I		Molecule II	
C(1)—O(2)—C(4)	119.8(6)	C(1')—O(2')—C(4')	118.5(6)
O(1)—C(1)—O(2)	125.2(8)	O(1')—C(1')—O(2')	124.0(8)
O(1)—C(1)—C(2)	124.1(7)	O(1')—C(1')—C(2')	124.4(8)
O(2)—C(1)—C(2)	110.6(7)	O(2')—C(1')—C(2')	111.6(7)
C(1)—C(2)—C(3)	124.6(7)	C(1')—C(2')—C(3')	125.1(8)
C(1)—C(2)—C(5)	114.6(8)	C(1')—C(2')—C(5')	115.5(8)
C(3)—C(2)—C(5)	120.6(9)	C(3')—C(2')—C(5')	119.5(9)
O(2)—C(4)—C(1A)	110.0(6)	O(2')—C(4')—C(1B)	107.5(6)
N(1)—C(5)—C(2)	177.2(9)	N(1')—C(5')—C(2')	177.9(9)
C(4)—C(1A)—C(2A)	112.1(6)	C(4')—C(1B)—C(2B)	111.5(6)
C(4)—C(1A)—C(8A)	110.3(6)	C(4')—C(1B)—C(8B)	113.1(6)
C(2A)—C(1A)—C(8A)	110.2(6)	C(2B)—C(1B)—C(8B)	108.1(6)
C(4)—C(1A)—C(9A)	106.2(6)	C(4')—C(1B)—C(9B)	109.0(6)
C(2A)—C(1A)—C(9A)	109.0(6)	C(2B)—C(1B)—C(9B)	107.4(6)
C(8A)—C(1A)—C(9A)	108.9(6)	C(8B)—C(1B)—C(9B)	107.6(6)
C(1A)—C(2A)—C(3A)	109.7(6)	C(1B)—C(2B)—C(3B)	112.2(7)
C(2A)—C(3A)—C(4A)	109.0(7)	C(2B)—C(3B)—C(4B)	108.0(7)
C(2A)—C(3A)—C(10A)	109.1(7)	C(2B)—C(3B)—C(10B)	110.4(7)
C(4A)—C(3A)—C(10A)	110.1(6)	C(4B)—C(3B)—C(10B)	109.0(7)
C(3A)—C(4A)—C(5A)	108.8(7)	C(3B)—C(4B)—C(5B)	109.3(7)
C(4A)—C(5A)—C(6A)	108.9(7)	C(4B)—C(5B)—C(6B)	109.4(8)
C(4A)—C(5A)—C(9A)	111.0(7)	C(4B)—C(5B)—C(9B)	110.5(8)
C(6A)—C(5A)—C(9A)	110.9(6)	C(6B)—C(5B)—C(9B)	109.1(7)
C(5A)—C(6A)—C(7A)	108.8(6)	C(5B)—C(6B)—C(7B)	110.4(7)
C(6A)—C(7A)—C(8A)	110.1(7)	C(6B)—C(7B)—C(8B)	107.9(7)
C(6A)—C(7A)—C(10A)	110.1(7)	C(6B)—C(7B)—C(10B)	108.7(7)
C(8A)—C(7A)—C(10A)	108.3(6)	C(8B)—C(7B)—C(10B)	109.4(7)
C(1A)—C(8A)—C(7A)	109.1(6)	C(1B)—C(8B)—C(7B)	111.8(6)
C(1A)—C(9A)—C(5A)	108.0(6)	C(1B)—C(9B)—C(5B)	111.1(7)
C(3A)—C(10A)—C(7A)	110.2(7)	C(3B)—C(10B)—C(7B)	109.9(7)

Table 3. Bond lengths (d) in the structure of **2**

Bond	d/Å	Bond	d/Å
O(1)—C(1)	1.202(3)	C(2)—C(4)	1.442(3)
O(2)—C(1)	1.337(3)	C(5)—C(6)	1.504(3)
O(2)—C(5)	1.461(3)	C(6)—C(7)	1.529(3)
N(1)—C(4)	1.138(3)	C(7)—C(8)	1.522(3)
C(1)—C(2)	1.495(3)	C(8)—C(9)	1.529(3)
C(2)—C(3)	1.328(3)	C(9)—C(9A)	1.525(5)

Table 4. Bond angles (ω) in the structure of **2**

Angle	ω/deg	Angle	ω/deg
C(1)—O(2)—C(5)	117.1(2)	O(2)—C(5)—C(6)	106.9(2)
C(1)—C(2)—C(3)	121.1(2)	C(5)—C(6)—C(7)	111.8(2)
C(1)—C(2)—C(4)	118.0(2)	C(6)—C(7)—C(8)	112.7(2)
C(3)—C(2)—C(4)	120.9(2)	C(7)—C(8)—C(9)	113.5(2)
O(1)—C(1)—O(2)	125.5(2)	C(8)—C(9)—C(9A)	113.4(2)
O(1)—C(1)—C(2)	124.1(2)	N(1)—C(4)—C(2)	179.5(2)
O(2)—C(1)—C(2)	110.4(2)		

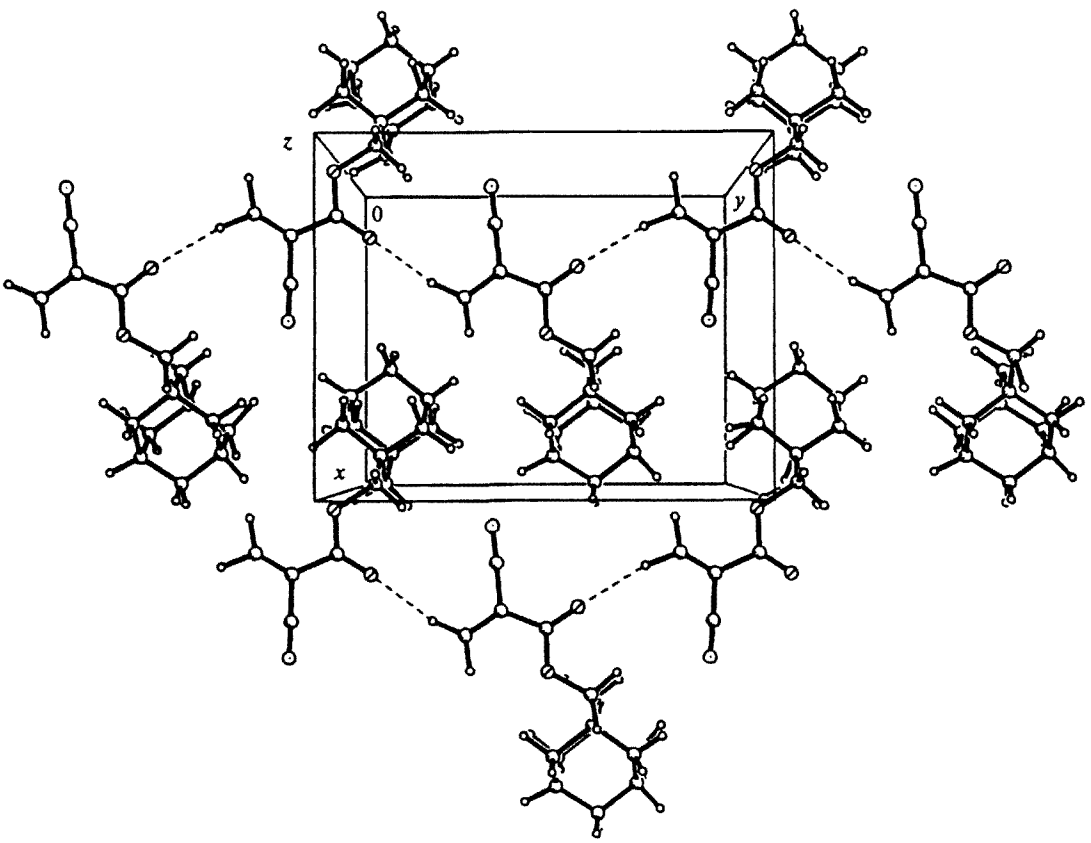

Fig. 1. Overall view of the monolayer (001) of the crystal packing of compound **1**. The short contacts typical of weak hydrogen bonds are indicated by the dot-and-dash lines.

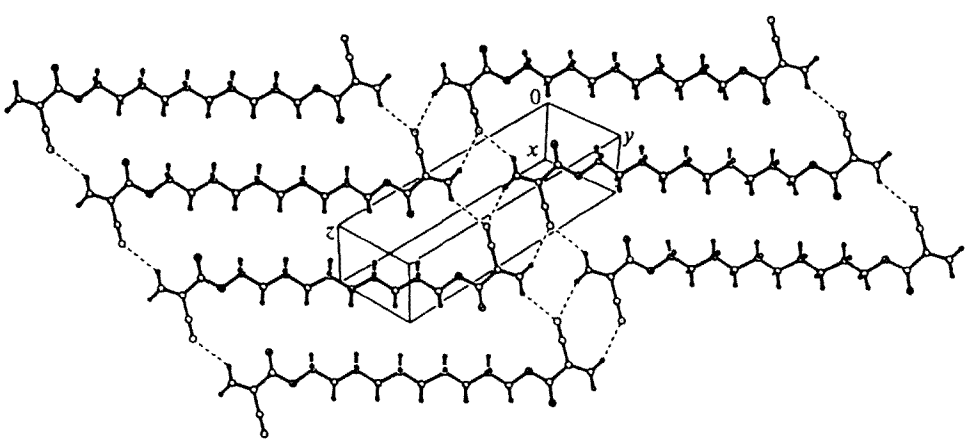

Fig. 2. Overall view of the monolayer ($\bar{4}31$) of the crystal packing of compound **2**. The short contacts typical of weak hydrogen bonds are indicated by the dot-and-dash lines.

Table 5. Coordinates ($\times 10^4$) and equivalent isotropic thermal parameters ($U_{eq} \times 10^3$) of nonhydrogen atoms in the structure of **1**

Atom	x	y	z	U_{eq}/Å^2
		Molecule I		
O(1)	2351(6)	836(5)	6672(3)	77(3)
O(2)	428(5)	11(4)	6835(3)	58(2)
N(1)	4822(7)	−1110(9)	6595(5)	100(4)
C(1)	1734(8)	58(7)	6768(4)	46(3)
C(2)	2321(7)	−1007(7)	6804(4)	47(3)
C(3)	1688(10)	−1836(6)	6957(4)	70(4)
C(4)	−355(8)	987(6)	6834(4)	56(3)
C(5)	3726(9)	−1042(9)	6691(4)	63(3)
C(1A)	−1167(7)	1052(5)	6216(4)	41(3)
C(2A)	−315(6)	1116(7)	5601(3)	46(3)
C(3A)	−1205(7)	1214(6)	4983(4)	49(3)
C(4A)	−2016(8)	2209(6)	5036(5)	61(3)
C(5A)	−2858(8)	2155(6)	5663(4)	57(4)
C(6A)	−3782(7)	1229(7)	5612(4)	55(3)
C(7A)	−2977(8)	242(6)	5559(4)	52(3)
C(8A)	−2098(7)	118(6)	6171(4)	46(3)
C(9A)	−1994(9)	2069(5)	6274(4)	54(3)
C(10A)	−2084(8)	289(6)	4946(4)	60(3)
		Molecule II		
O(1′)	3278(6)	5760(5)	7053(4)	91(3)
O(2′)	5227(5)	5045(4)	6808(3)	65(2)
N(1′)	908(8)	3754(8)	6944(5)	94(4)
C(1′)	3927(9)	5013(7)	6920(4)	55(3)
C(2′)	3413(8)	3944(7)	6870(4)	54(3)
C(3′)	4136(10)	3099(6)	6780(4)	71(4)
C(4′)	5898(8)	6051(6)	6852(4)	58(3)
C(5′)	2003(10)	3846(8)	6921(5)	67(4)
C(1B)	6924(7)	6084(6)	6304(3)	43(3)
C(2B)	6273(8)	6142(7)	5620(4)	53(3)
C(3B)	7261(9)	6208(7)	5079(4)	64(3)
C(4B)	8076(10)	7210(7)	5182(5)	78(4)
C(5B)	8757(9)	7159(6)	5846(5)	69(4)
C(6B)	9669(8)	6206(7)	5864(5)	76(4)
C(7B)	8879(9)	5201(6)	5764(4)	59(3)
C(8B)	7855(8)	5136(6)	6310(4)	51(3)
C(9B)	7768(7)	7069(6)	6386(4)	59(4)
C(10B)	8184(9)	5257(6)	5097(4)	57(3)

Table 6. Coordinates ($\times 10^3$) of hydrogen atoms in the structure of **1**

Atom	x	y	z	Atom	x	y	z
	Molecule I				Molecule II		
H(3A)	208	−265	693	H(3D)	518	322	682
H(3B)	64	−195	701	H(3E)	370	233	676
H(4A)	34	161	684	H(4E)	525	668	679
H(4B)	−66	105	742	H(4F)	683	617	722
H(2A)	20	33	558	H(2C)	568	677	564
H(2B)	39	166	560	H(2D)	569	547	560
H(3C)	−33	102	455	H(3F)	662	631	466
H(4C)	−151	288	502	H(4G)	884	733	487
H(4D)	−264	225	459	H(4H)	718	781	508
H(5A)	−343	277	563	H(5B)	952	772	588
H(6A)	−443	104	601	H(6C)	1031	622	549
H(6B)	−423	128	521	H(6D)	1017	615	624
H(7A)	−356	−44	555	H(7B)	936	445	573
H(8A)	−143	−56	622	H(8C)	723	444	633
H(8B)	−282	21	658	H(8D)	820	512	679
H(9A)	−138	275	625	H(9C)	722	775	635
H(9B)	−283	184	665	H(9D)	816	725	679
H(10A)	−275	30	462	H(10C)	748	459	504
H(10B)	−195	−43	491	H(10D)	868	532	457

from 2-cyanoacrylic acid (0.97 g, 0.01 mol) according to the procedure described previously.[13] The solution was stirred at 20 °C for 3 days, and then the solvent was removed *in vacuo*. The residue was crystallized from a toluene—heptane mixture. The yield of colorless crystals of **1** was 41%, m.p. 60—61 °C. ^{1}H NMR, δ: 1.6—2.2 (m, 15 H, Ad); 4.1 (s, 2 H, CH_2O); 5.15, 6.46 (both s, 2 H, $=CH_2$). Found (%): C, 73.41; H, 8.08; N, 5.46. $C_{15}H_{19}NO_2$. Calculated (%): C, 73.44; H, 7.80; N, 5.71.

1,10-Decanediol bis-2-cyanoacrylate (2). A solution of methyl 2-cyanoacrylate (0.3 mol) and 1,10-decanediol (0.15 mol) was added to an anhydrous cold solution of *p*-toluenesulfonic acid in *o*-xylene (azeotropic distillation of xylene from a 0.05 *M* solution of *p*-toluenesulfonic acid hydrate) under an atmosphere of anhydrous sulfur dioxide (the total volume of the solution was 0.5 L). A mixture of xylene and methanol was distilled off over a period of 1 h with intense stirring of the solution under a stream of SO_2, and an equivalent amount of xylene was added. After the solution was cooled, its liquid phase was decanted, and then the solvent was removed *in vacuo* (to 3/4 volume). An equivalent amount of heptane was added, and the mixture was cooled to −20 °C. After one day, the residue was crystallized from hot octane. The yield of bis-2-cyanoacrylate **2** was 20—25%, m.p. 75 °C. ^{1}H NMR, δ: 0.9—1.4 (m, 16 H, CH_2); 3.98 (t, 4 H, CH_2O); 5.5, 6.45 (both s, 2 H, —CH=).

X-ray structural study of compounds 1 and 2. Crystals of **1** ($C_{15}H_{19}NO_2$, M = 245.3) are orthorhombic, at −100 °C: a = 10.185(3) Å, b = 12.814(4) Å, c = 20.368(6) Å, V = 2658(1) Å^3, Z = 8, d_{calc} = 1.226 g cm^{-3}, space group $P2_12_12_1$. The unit cell parameters and intensities of 3158 reflections were measured on an automated four-circle Siemens P3/PC diffractometer (T = 173 K, Mo-Kα radiation, graphite monochromator, θ/2θ-scanning technique, θ_{max} = 25°). The structure was solved by the direct method and refined by the full-matrix least-squares method in anisotropic approximation for nonhydrogen atoms. Hydrogen atoms were located from the difference Fourier map and included in the refinement with fixed positional (the riding model) and thermal (U_{iso} = 0.05 Å^2) parameters. The final R factors were as follows: R = 0.058 and R_w = 0.062 using 1196 independent reflections with $I > 2.5\sigma(I)$. All calculations were carried out on an IBM PC/AT-386 computer using the SHELXTL PLUS program package. Coordinates and equivalent isotropic thermal parameters of nonhydrogen atoms are given in Table 5; coordinates of hydrogen atoms are listed in Table 6.

Crystals of **2** ($C_{18}H_{24}N_2O_4$, M = 332.4) are triclinic; at −100 °C: a = 4.618(2) Å, b = 5.679(2) Å, c = 17.305(5) Å, α = 92.37(2)°, β = 93.36(2)°, g = 98.12(2)°, V = 447.9(2) Å^3, Z = 1 (the molecule occupies a special position, *i.e.*, is located in the inversion center), d_{calc} = 1.232 g cm^{-3}, space group $P\bar{1}$. The unit cell parameters and intensities of 2438 reflections were measured on an automated four-circle Siemens P3/PC diffractometer (T = 173 K, Mo-Kα radiation, graphite monochromator, θ/2θ-scanning technique, θ_{max} = 25°). The structure was solved by the direct method and refined by the full-matrix least-squares method in anisotropic approximation

Table 7. Coordinates ($\times 10^4$) and equivalent isotropic thermal parameters ($U_{eq} \times 10^3$) of nonhydrogen atoms in the structure of **2**

Atom	*x*	*y*	*z*	U_{eq}/Å^2
O(1)	3525(4)	11376(3)	7150(1)	39(1)
O(2)	297(3)	7992(3)	7135(1)	34(1)
N(1)	−4891(5)	7595(4)	5687(1)	45(1)
C(1)	20(7)	12795(4)	5882(2)	44(1)
C(2)	−526(5)	10715(4)	6207(1)	29(1)
C(3)	1362(5)	10113(4)	6881(1)	29(1)
C(4)	−2961(5)	8964(4)	5918(1)	31(1)
C(5)	2006(5)	7074(4)	7763(1)	35(1)
C(6)	−75(5)	5314(4)	8161(1)	31(1)
C(7)	1520(5)	4065(4)	8788(1)	30(1)
C(8)	−531(5)	2221(4)	9182(1)	31(1)
C(9)	1034(5)	904(4)	9796(1)	31(1)

Table 8. Coordinates ($\times 10^3$) and isotropic thermal parameters ($U_{iso} \times 10^3$) of hydrogen atoms in the structure of **2**

Атом	*x*	*y*	*z*	U_{iso}/Å^2
H(3A)	179(5)	1382(4)	608(1)	36(7)
H(3B)	−124(6)	1313(5)	546(2)	46(7)
H(5A)	365(5)	637(4)	755(1)	28(6)
H(5B)	283(6)	838(5)	810(2)	47(7)
H(6A)	−158(5)	610(4)	836(1)	31(6)
H(6B)	−118(5)	419(4)	778(2)	40(7)
H(7A)	310(5)	333(4)	860(1)	22(5)
H(7B)	252(6)	524(5)	917(2)	47(7)
H(8A)	−210(5)	297(4)	940(1)	28(6)
H(8B)	−156(5)	110(4)	879(1)	36(6)
H(9A)	256(5)	9(4)	956(1)	28(6)
H(9B)	209(6)	207(5)	1019(2)	49(7)

for nonhydrogen atoms. Hydrogen atoms were located from the difference Fourier map and refined isotropically. The final *R* factors were as follows: $R = 0.038$ and $R_w = 0.041$ using 1120 independent reflections with $I > 3\sigma(I)$. All calculations were carried out on an IBM PC/AT-386 computer using the SHELXTL PLUS program package. Atomic coordinates and isotropic thermal parameters (equivalent thermal parameters for nonhydrogen atoms) are given in Tables 7 and 8.

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