Molecular and crystal structures of (6*R*,7*R*,14*S*)-6,14-etheno-7-[(1*R*)-1-hydroxyethyl]- and (6*R*,7*R*,14*S*)-6,14-etheno-7-[(1*S*)-1-hydroxyethyl]-6,7,8,14-tetrahydro-17-nor-17-phenylthebaine

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The crystal structures and absolute configurations of (6*R*,7*R*,14*S*)-6,14-etheno-7-[(1*R*)-1-hydroxyethyl]-6,7,8,14-tetrahydro-17-nor-17-phenylthebaine and (6*R*,7*R*,14*S*)-6,14-etheno-7-[(1*S*)-1-hydroxyethyl]-6,7,8,14-tetrahydro-17-nor-17-phenylthebaine were established by X-ray diffraction analysis.

Key words: morphine alkaloids, thebaine, X-ray diffraction analysis, absolute configuration.

The nature of the substituent at the nitrogen atom of morphine alkaloids is one of the most important factors that exert not only a quantitative but also a qualitative effect on their pharmacological activity.[1] Recently,[2] we have reported the synthesis of a first derivative containing the aromatic substituent at this position. In this work, we established the molecular and crystal structures of two analogous *N*-phenyl-substituted derivatives of morphine alkaloids. It was of interest to study the effect of the aromatic substituent at the nitrogen atom on the conformation of the piperidine ring of the molecule, to reveal the differences in the intramolecular interactions in both diastereomeric molecules, and to establish the absolute configurations at the diastereogenic center.

The absolute configurations of diastereomeric (6*R*,7*R*,14*S*)-6,14-etheno-7-[(1*R*)-1-hydroxyethyl]-6,7,8,14-tetrahydro-17-nor-17-phenylthebaine (**1**) and (6*R*,7*R*,14*S*)-6,14-etheno-7-[(1*S*)-1-hydroxyethyl]-6,7,8,14-tetrahydro-17-nor-17-phenylthebaine (**2**) were established by X-ray diffraction analysis. The standard[3] projections of these diastereomers are shown in Figs. 1 and 2, respectively. The bond lengths and bond angles in the molecules under study are given in Tables 1—4. The familiar absolute configuration of the molecule of natural thebaine was used as the criterion for the validity of the assignment of the configurations of the title molecules.

The molecules under study have structures similar to those of the molecules of morphine alkaloids studied previously.[3—6] The ring *C* adopts a boat conformation. The piperidine ring has a chair conformation with the equatorial orientation of the phenyl substituent.

The *R* isomer (**1**) is characterized by the rather strong intramolecular hydrogen bond O(4)—H(O(4))...O(3) [O(4)...O(3), 2.694(2) Å; O(4)—H(O(4)), 0.91(3) Å; H(O(4))...O(3), 1.87(3) Å; the O(4)—H(O(4))...O(3) angle, 150(2)°], which apparently contributes to stabilization of this isomer.

The *S* isomer (**2**) crystallized with the methanol molecule in a ratio of 1 : 1. In the crystal, molecules **2**

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

Bond	*d*/Å	Bond	*d*/Å
O(1)—C(1)	1.380(2)	C(8)—C(9)	1.535(2)
O(1)—C(12)	1.469(2)	C(8)—C(13)	1.548(2)
O(2)—C(2)	1.375(2)	C(9)—C(10)	1.562(2)
O(2)—C(23)	1.421(2)	C(10)—C(20)	1.533(2)
O(3)—C(22)	1.425(2)	C(10)—C(11)	1.573(2)
O(3)—C(11)	1.436(2)	C(11)—C(19)	1.508(2)
O(4)—C(20)	1.433(2)	C(11)—C(12)	1.546(2)
N(17)—C(24)	1.421(2)	C(12)—C(13)	1.542(2)
N(17)—C(16)	1.468(2)	C(13)—C(14)	1.496(2)
N(17)—C(7)	1.485(2)	C(13)—C(15)	1.533(2)
C(1)—C(14)	1.374(2)	C(15)—C(16)	1.526(2)
C(1)—C(2)	1.393(2)	C(18)—C(19)	1.329(2)
C(2)—C(3)	1.401(3)	C(20)—C(21)	1.519(3)
C(3)—C(4)	1.392(3)	C(24)—C(29)	1.396(2)
C(4)—C(5)	1.401(2)	C(24)—C(25)	1.402(2)
C(5)—C(14)	1.386(2)	C(25)—C(26)	1.386(3)
C(5)—C(6)	1.514(2)	C(26)—C(27)	1.391(3)
C(6)—C(7)	1.561(2)	C(27)—C(28)	1.374(3)
C(7)—C(8)	1.533(2)	C(28)—C(29)	1.398(3)
C(8)—C(18)	1.513(2)		

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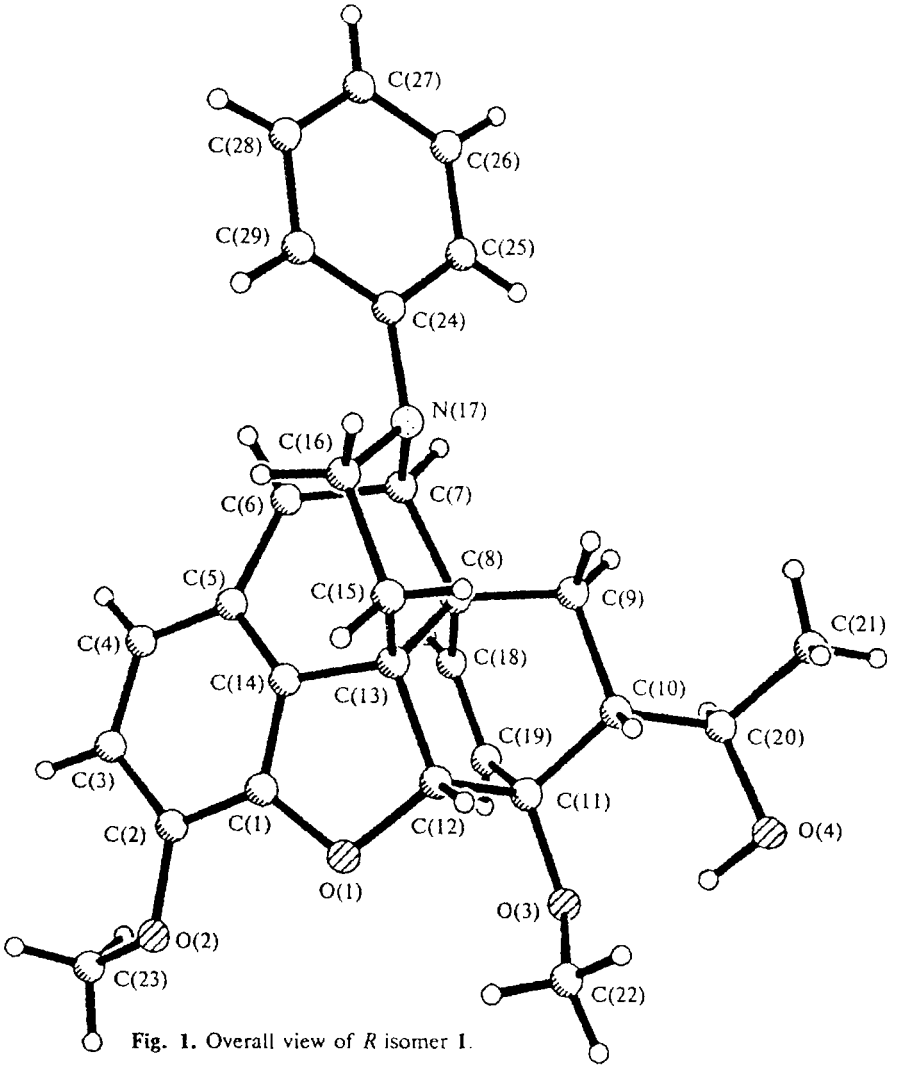

Fig. 1. Overall view of *R* isomer **1**.

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

Angle	ω/deg	Angle	ω/deg	Angle	ω/deg
C(1)—O(1)—C(12)	106.77(12)	C(18)—C(8)—C(9)	105.75(12)	C(15)—C(13)—C(8)	110.98(13)
C(2)—O(2)—C(23)	115.8(2)	C(7)—C(8)—C(9)	115.91(13)	C(12)—C(13)—C(8)	111.74(12)
C(22)—O(3)—C(11)	117.71(13)	C(18)—C(8)—C(13)	107.05(12)	C(1)—C(14)—C(5)	123.6(2)
C(24)—N(17)—C(16)	116.56(13)	C(7)—C(8)—C(13)	105.97(12)	C(1)—C(14)—C(13)	109.62(13)
C(24)—N(17)—C(7)	112.28(12)	C(9)—C(8)—C(13)	108.69(12)	C(5)—C(14)—C(13)	125.0(2)
C(16)—N(17)—C(7)	112.06(13)	C(8)—C(9)—C(10)	109.77(13)	C(16)—C(15)—C(13)	112.03(12)
C(14)—C(1)—O(1)	113.32(14)	C(20)—C(10)—C(9)	109.48(14)	N(17)—C(16)—C(15)	109.62(13)
C(14)—C(1)—C(2)	120.2(2)	C(20)—C(10)—C(11)	114.23(13)	C(19)—C(18)—C(8)	114.82(13)
O(1)—C(1)—C(2)	126.1(2)	C(9)—C(10)—C(11)	108.77(12)	C(18)—C(19)—C(11)	114.80(13)
O(2)—C(2)—C(1)	118.0(2)	O(3)—C(11)—C(19)	106.47(12)	O(4)—C(20)—C(21)	105.7(2)
O(2)—C(2)—C(3)	125.1(2)	O(3)—C(11)—C(12)	113.91(12)	O(4)—C(20)—C(10)	112.9(2)
C(1)—C(2)—C(3)	116.9(2)	C(19)—C(11)—C(12)	108.91(12)	C(21)—C(20)—C(10)	111.7(2)
C(4)—C(3)—C(2)	121.7(2)	O(3)—C(11)—C(10)	113.55(12)	C(29)—C(24)—C(25)	118.4(2)
C(3)—C(4)—C(5)	120.8(2)	C(19)—C(11)—C(10)	109.11(13)	C(29)—C(24)—N(17)	123.5(2)
C(14)—C(5)—C(4)	115.7(2)	C(12)—C(11)—C(10)	104.80(12)	C(25)—C(24)—N(17)	118.10(14)
C(14)—C(5)—C(6)	118.63(14)	O(1)—C(12)—C(13)	107.39(12)	C(26)—C(25)—C(24)	120.8(2)
C(4)—C(5)—C(6)	124.6(2)	O(1)—C(12)—C(11)	112.96(12)	C(25)—C(26)—C(27)	120.4(2)
C(5)—C(6)—C(7)	114.83(13)	C(13)—C(12)—C(11)	108.12(12)	C(28)—C(27)—C(26)	119.2(2)
N(17)—C(7)—C(8)	109.53(12)	C(14)—C(13)—C(15)	113.61(13)	C(27)—C(28)—C(29)	121.2(2)
N(17)—C(7)—C(6)	111.90(13)	C(14)—C(13)—C(12)	101.66(13)	C(24)—C(29)—C(28)	120.0(2)
C(8)—C(7)—C(6)	112.38(13)	C(15)—C(13)—C(12)	113.12(12)		
C(18)—C(8)—C(7)	113.11(12)	C(14)—C(13)—C(8)	105.15(12)		

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

Bond	d/Å	Bond	d/Å
O(1)—C(1)	1.388(3)	C(8)—C(18)	1.514(3)
O(1)—C(12)	1.482(3)	C(8)—C(13)	1.550(3)
O(2)—C(2)	1.370(3)	C(8)—C(9)	1.550(3)
O(2)—C(23)	1.426(3)	C(9)—C(10)	1.554(3)
O(3)—C(22)	1.422(4)	C(10)—C(20)	1.546(4)
O(3)—C(11)	1.420(3)	C(10)—C(11)	1.583(3)
O(4)—C(20)	1.424(4)	C(11)—C(19)	1.508(3)
O(5)—C(30)	1.413(5)	C(11)—C(12)	1.547(4)
N(17)—C(24)	1.411(3)	C(12)—C(13)	1.544(3)
N(17)—C(16)	1.463(3)	C(13)—C(14)	1.501(3)
N(17)—C(7)	1.479(3)	C(13)—C(15)	1.536(3)
C(1)—C(14)	1.369(3)	C(15)—C(16)	1.522(4)
C(1)—C(2)	1.388(3)	C(18)—C(19)	1.320(4)
C(2)—C(3)	1.406(4)	C(20)—C(21)	1.518(4)
C(3)—C(4)	1.393(4)	C(24)—C(29)	1.397(4)
C(4)—C(5)	1.406(3)	C(24)—C(25)	1.411(4)
C(5)—C(14)	1.387(3)	C(25)—C(26)	1.388(4)
C(5)—C(6)	1.518(4)	C(26)—C(27)	1.392(4)
C(6)—C(7)	1.578(3)	C(27)—C(28)	1.389(5)
C(7)—C(8)	1.538(3)	C(28)—C(29)	1.396(4)

and molecules of solvation are linked in infinite chains about the 2_1 axis through the intermolecular hydrogen bonds O(4)—H(O(4))...O(5) ($-x$, $0.5+y$, $-z$) [O(4)...O(5), 2.721(5) Å; O(4)—H(O(4)), 1.01(4) Å; H(O(4))...O(5), 1.74(4) Å; the O(4)—H(O(4))...O(5) angle, 164(2)°] and O(5)—H(O(5))...O(1) (x, y, z) [O(5)...O(1), 2.844(5) Å; O(5)—H(O(5)), 0.97(8) Å; H(O(5))...O(1), 1.87(9) Å; the O(5)—H(O(5))...O(1) angle, 177(4)°].

Experimental

Crystals of compound **1*** ($C_{28}H_{31}NO_4$, M = 445.5) are orthorhombic, at −125 °C: a = 8.850(4), b = 12.739(5), c = 19.784(7) Å, V = 2231(2) Å^3, d_{calc} = 1.327 g cm^{-3}, Z = 4, space group $P2_12_12_1$.

Crystals of compound **2*** ($C_{29}H_{35}NO_5$, M = 477.6) are monoclinic, at −125 °C: a = 11.119(3), b = 9.940(3),

* Synthesis of compounds **1** and **2** is reported in Ref. 7.

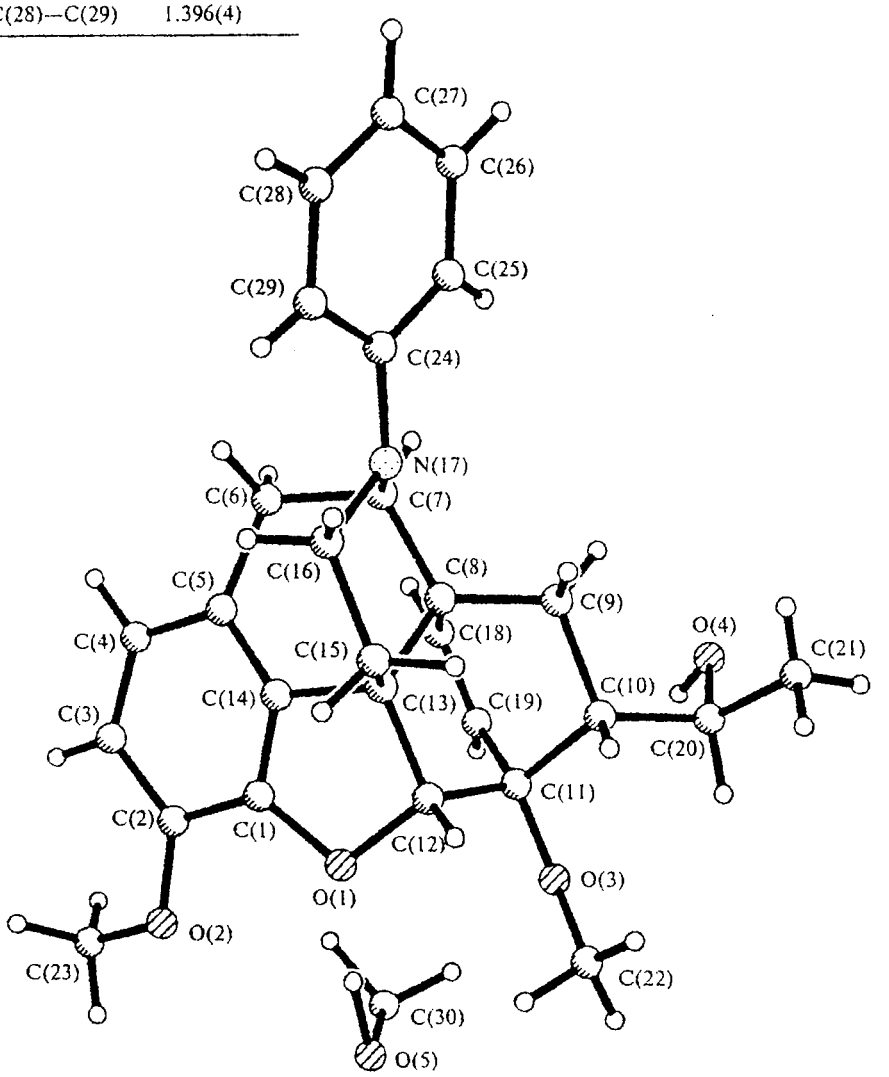

Fig. 2. Overall view of S isomer **2**.

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

Angle	ω/deg	Angle	ω/deg	Angle	ω/deg
C(1)—O(1)—C(12)	106.7(2)	C(18)—C(8)—C(13)	107.1(2)	C(14)—C(13)—C(8)	105.5(2)
C(2)—O(2)—C(23)	117.1(2)	C(7)—C(8)—C(13)	105.8(2)	C(15)—C(13)—C(8)	111.4(2)
C(22)—O(3)—C(11)	117.3(2)	C(18)—C(8)—C(9)	105.5(2)	C(12)—C(13)—C(8)	111.6(2)
C(24)—N(17)—C(16)	117.5(2)	C(7)—C(8)—C(9)	115.8(2)	C(1)—C(14)—C(5)	123.1(2)
C(24)—N(17)—C(7)	117.8(2)	C(13)—C(8)—C(9)	108.9(2)	C(1)—C(14)—C(13)	110.1(2)
C(16)—N(17)—C(7)	113.1(2)	C(8)—C(9)—C(10)	109.0(2)	C(5)—C(14)—C(13)	124.9(2)
C(14)—C(1)—O(1)	113.1(2)	C(20)—C(10)—C(9)	112.2(2)	C(16)—C(15)—C(13)	112.5(2)
C(14)—C(1)—C(2)	121.5(2)	C(20)—C(10)—C(11)	113.7(2)	N(17)—C(16)—C(15)	109.9(2)
O(1)—C(1)—C(2)	125.1(2)	C(9)—C(10)—C(11)	109.6(2)	C(19)—C(18)—C(8)	114.7(2)
O(2)—C(2)—C(1)	117.3(2)	O(3)—C(11)—C(19)	107.1(2)	C(18)—C(19)—C(11)	115.7(2)
O(2)—C(2)—C(3)	126.3(2)	O(3)—C(11)—C(12)	114.4(2)	O(4)—C(20)—C(21)	107.3(2)
C(1)—C(2)—C(3)	116.4(2)	C(19)—C(11)—C(12)	108.9(2)	O(4)—C(20)—C(10)	112.7(2)
C(4)—C(3)—C(2)	121.2(2)	O(3)—C(11)—C(10)	114.3(2)	C(21)—C(20)—C(10)	109.8(3)
C(3)—C(4)—C(5)	121.7(2)	C(19)—C(11)—C(10)	109.2(2)	C(29)—C(24)—C(25)	117.7(2)
C(14)—C(5)—C(4)	115.3(2)	C(12)—C(11)—C(10)	102.8(2)	C(29)—C(24)—N(17)	122.3(2)
C(14)—C(5)—C(6)	118.7(2)	O(1)—C(12)—C(13)	107.1(2)	C(25)—C(24)—N(17)	119.8(2)
C(4)—C(5)—C(6)	125.4(2)	O(1)—C(12)—C(11)	113.6(2)	C(26)—C(25)—C(24)	120.9(2)
C(5)—C(6)—C(7)	114.8(2)	C(13)—C(12)—C(11)	108.5(2)	C(25)—C(26)—C(27)	121.2(3)
N(17)—C(7)—C(8)	108.9(2)	C(14)—C(13)—C(15)	113.3(2)	C(26)—C(27)—C(28)	118.1(2)
N(17)—C(7)—C(6)	112.0(2)	C(14)—C(13)—C(12)	101.6(2)	C(27)—C(28)—C(29)	121.5(3)
C(8)—C(7)—C(6)	112.3(2)	C(15)—C(13)—C(12)	112.8(2)	C(24)—C(29)—C(28)	120.6(3)
C(18)—C(8)—C(7)	113.4(2)				

Table 5. Coordinates ($\times 10^4$) and isotropic equivalent (isotropic for H atoms) temperature factors of the atoms in the structure of **1**

Atom	*x*	*y*	*z*	*U*	Atom	*x*	*y*	*z*	*U*
O(1)	−408(1)	−5756(1)	−1290(1)	23(1)	C(29)	−5768(2)	−10102(1)	−1007(1)	24(1)
O(2)	1796(2)	−6242(1)	−2297(1)	30(1)	H(O(4))	−3698(31)	−2961(21)	−801(13)	38(7)
O(3)	−2102(1)	−3766(1)	−1135(1)	22(1)	H(3)	883(26)	−7913(18)	−2984(12)	28(6)
O(4)	−4592(2)	−2912(1)	−577(1)	39(1)	H(4)	−1523(29)	−8695(20)	−2942(12)	36(6)
N(17)	−5167(2)	−8201(1)	−964(1)	20(1)	H(61)	−4130(25)	−8922(17)	−2050(10)	23(5)
C(1)	−485(2)	−6614(1)	−1714(1)	21(1)	H(62)	−4361(24)	−8017(16)	−2560(11)	20(5)
C(2)	525(2)	−6854(1)	−2231(1)	24(1)	H(7)	−5912(24)	−7456(15)	−1774(10)	16(5)
C(3)	136(2)	−7689(1)	−2658(1)	25(1)	H(91)	−6170(27)	−5655(18)	−1127(11)	25(5)
C(4)	−1262(2)	−8186(1)	−2614(1)	24(1)	H(92)	−5542(24)	−6234(17)	−490(11)	25(5)
C(5)	−2297(2)	−7912(1)	−2108(1)	21(1)	H(10)	−3962(25)	−4868(16)	−255(11)	21(5)
C(6)	−3963(2)	−8181(1)	−2121(1)	22(1)	H(12)	−1628(25)	−5614(18)	−460(11)	27(6)
C(7)	−4945(2)	−7580(1)	−1592(1)	18(1)	H(151)	−3472(24)	−7110(16)	−106(11)	23(5)
C(8)	−4284(2)	−6503(1)	−1413(1)	16(1)	H(152)	−1881(28)	−7609(19)	−283(11)	32(6)
C(9)	−5226(2)	−5831(1)	−926(1)	19(1)	H(161)	−3954(29)	−8804(17)	−213(12)	31(6)
C(10)	−4315(2)	−4830(1)	−722(1)	18(1)	H(162)	−3083(25)	−8912(17)	−915(10)	20(5)
C(11)	−2850(2)	−4765(1)	−1170(1)	17(1)	H(18)	−4301(24)	−6024(17)	−2460(11)	22(5)
C(12)	−1863(2)	−5697(1)	−934(1)	18(1)	H(19)	−3025(24)	−4447(16)	−2248(10)	20(5)
C(13)	−2727(2)	−6722(1)	−1085(1)	17(1)	H(20)	−5703(24)	−3753(16)	−1224(11)	21(5)
C(14)	−1787(2)	−7189(1)	−1637(1)	19(1)	H(211)	−7303(30)	−3318(20)	−304(13)	37(7)
C(15)	−2891(2)	−7435(1)	−465(1)	21(1)	H(212)	−7448(34)	−4503(22)	−452(14)	50(8)
C(16)	−3729(2)	−8449(1)	−631(1)	22(1)	H(213)	−6303(28)	−4049(18)	194(13)	34(6)
C(18)	−4004(2)	−5821(1)	−2027(1)	19(1)	H(221)	−664(28)	−2893(20)	−630(12)	32(6)
C(19)	−3276(2)	−4929(1)	−1900(1)	19(1)	H(222)	−1527(29)	−3639(18)	−150(13)	34(6)
C(20)	−5341(2)	−3863(1)	−771(1)	25(1)	H(223)	−206(26)	−4079(20)	−636(11)	27(6)
C(21)	−6701(2)	−3956(2)	−306(1)	36(1)	H(231)	3040(27)	−6940(19)	−3009(11)	28(6)
C(22)	−1043(3)	−3607(2)	−603(1)	33(1)	H(232)	1758(29)	−6097(20)	−3311(12)	35(6)
C(23)	2507(2)	−6263(2)	−2940(1)	37(1)	H(233)	3285(30)	−5751(20)	−2904(12)	37(6)
C(24)	−6189(2)	−9049(1)	−1058(1)	20(1)	H(25)	−7955(28)	−8113(19)	−1230(11)	30(6)
C(25)	−7694(2)	−8812(1)	−1218(1)	24(1)	H(26)	−9746(32)	−9415(20)	−1430(13)	42(7)
C(26)	−8733(2)	−9605(2)	−1337(1)	30(1)	H(27)	−9024(30)	−11129(22)	−1342(13)	42(7)
C(27)	−8301(3)	−10652(2)	−1292(1)	33(1)	H(28)	−6505(31)	−11543(22)	−1088(13)	40(7)
C(28)	−6835(3)	−10892(1)	−1124(1)	29(1)	H(29)	−4783(27)	−10317(18)	−913(11)	24(5)

Table 6. Coordinates ($\times 10^4$) and isotropic equivalent (isotropic for H atoms) temperature factors of the atoms in the structure of **2**

Atom	x	y	z	U	Atom	x	y	z	U
O(1)	1573(2)	5320(2)	185(1)	24(1)	H(O(4))	763(35)	10028(45)	−2538(38)	43(10)
O(2)	2514(2)	5042(2)	2737(2)	32(1)	H(O(5))	348(65)	5687(76)	1008(63)	106(21)
O(3)	−125(2)	7428(2)	−1194(2)	30(1)	H(3)	4983(33)	5316(44)	3265(35)	40(9)
O(4)	780(2)	9587(2)	−3335(2)	40(1)	H(4)	6230(29)	5834(33)	1924(26)	20(7)
O(5)	−305(2)	5912(3)	1401(2)	40(1)	H(61)	5837(27)	7082(34)	−246(26)	18(7)
N(17)	4587(2)	5419(2)	−2727(2)	22(1)	H(62)	6166(29)	5656(36)	−616(28)	26(8)
C(1)	2833(2)	5252(3)	767(2)	22(1)	H(7)	4891(24)	7292(30)	−2178(24)	9(6)
C(2)	3330(2)	5205(3)	2024(2)	24(1)	H(91)	2375(25)	6480(31)	−3693(25)	14(6)
C(3)	4625(2)	5389(3)	2444(2)	26(1)	H(92)	2540(29)	8047(35)	−3292(28)	23(7)
C(4)	5347(2)	5678(3)	1636(2)	24(1)	H(10)	542(27)	6373(34)	−3251(26)	18(7)
C(5)	4830(2)	5714(3)	365(2)	22(1)	H(12)	932(31)	4939(36)	−1605(31)	28(8)
C(6)	5469(2)	6271(3)	−565(2)	23(1)	H(151)	2806(27)	3402(35)	−1671(27)	22(7)
C(7)	4575(2)	6542(2)	−1873(2)	19(1)	H(152)	2187(35)	4419(42)	−2956(35)	43(10)
C(8)	3225(2)	6802(2)	−1823(2)	19(1)	H(161)	4198(29)	3429(37)	−2865(29)	26(8)
C(9)	2294(2)	7146(3)	−3061(2)	22(1)	H(162)	4814(28)	3871(32)	−1599(27)	17(7)
C(10)	952(2)	7148(3)	−2888(2)	24(1)	H(18)	3807(30)	8540(38)	−560(30)	28(8)
C(11)	1012(2)	7095(3)	−1476(2)	22(1)	H(19)	1818(28)	8770(36)	−257(28)	24(8)
C(12)	1477(2)	5648(3)	−1114(2)	20(1)	H(20)	−687(29)	8355(34)	−3363(27)	23(7)
C(13)	2793(2)	5494(2)	−1311(2)	18(1)	H(211)	598(37)	8054(39)	−5210(33)	35(9)
C(14)	3577(2)	5385(2)	−19(2)	19(1)	H(212)	−674(48)	7158(61)	−5174(45)	72(14)
C(15)	2909(2)	4270(3)	−2102(2)	22(1)	H(213)	−575(42)	8848(54)	−5303(42)	60(13)
C(16)	4203(2)	4137(3)	−2306(2)	24(1)	H(221)	−1080(33)	5651(41)	−1192(34)	35(9)
C(18)	3105(2)	7932(2)	−964(2)	20(1)	H(222)	−1349(37)	6318(49)	−2470(40)	50(11)
C(19)	1979(2)	8082(3)	−801(2)	23(1)	H(223)	−1825(39)	6957(47)	−1402(36)	49(11)
C(20)	161(2)	8324(3)	−3574(2)	33(1)	H(231)	3354(35)	6177(42)	4252(33)	41(10)
C(21)	−134(3)	8084(4)	−4943(3)	39(1)	H(232)	2259(31)	5200(40)	4366(31)	32(8)
C(22)	−1139(3)	6524(4)	−1598(3)	39(1)	H(233)	3667(31)	4591(38)	4352(31)	28(8)
C(23)	2990(3)	5238(3)	4021(2)	34(1)	H(25)	5448(32)	7320(39)	−3873(31)	30(8)
C(24)	5552(2)	5392(3)	−3328(2)	22(1)	H(26)	6797(31)	7321(38)	−5123(31)	29(8)
C(25)	5805(2)	6548(3)	−3947(2)	23(1)	H(27)	7918(30)	5256(39)	−5243(30)	31(8)
C(26)	6670(2)	6511(3)	−4638(2)	27(1)	H(28)	7455(38)	3347(48)	−4161(37)	45(10)
C(27)	7307(3)	5332(3)	−4751(2)	31(1)	H(29)	6071(32)	3368(40)	−3057(32)	33(9)
C(28)	7070(3)	4196(3)	−4133(3)	35(1)	H(301)	−254(41)	7545(47)	2447(41)	55(12)
C(29)	6218(3)	4221(3)	−3419(3)	29(1)	H(302)	−162(48)	7861(60)	997(51)	71(15)
C(30)	101(4)	7233(4)	1761(3)	50(1)	H(303)	1060(51)	7575(60)	2213(49)	77(16)

c = 11.320(3) Å, β = 104.96(2)°, V = 1208.7(6) Å^3, d_{calc} = 1.312 g cm^{-3}, Z = 2, space group $P2_1$.

The unit cell parameters and intensities of 4009 and 3018 (for compounds **1** and **2**, respectively) independent reflections were measured on an automated four-circle Siemens P3/PC diffractometer (Mo-Kα radiation, graphite monochromator, θ/2θ scanning technique to θ_{max} = 28°). The structures were solved by direct methods, which allowed us to reveal all nonhydrogen atoms. Then the structures were refined by the full-matrix least-squares method with anisotropic temperature factors for nonhydrogen atoms. The positions of all hydrogen atoms were located from difference Fourier syntheses and refined isotropically. The final values of the R factors were as follows: R = 0.038 using 3085 reflections with $I > 2\sigma$, R_w= 0.088 based on all 3682 independent reflections for compound **1** and R = 0.039 using 2575 reflections with $I > 2\sigma$, R_w= 0.099 based on all 2716 independent reflections for compound **2**. All calculations were carried out using the SHELXTL PLUS program package[8] (PC version). The atomic coordinates and isotropic equivalent (isotropic for H atoms) temperature factors are listed in Tables 5 and 6.

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