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4-HYDROXY-2-QUINOLONES 172*. SYNTHESIS AND STRUCTURE OF 4,3'-SPIRO[(6-ALLYL-2-AMINO-5-OXO-5,6-DIHYDRO-4H-PYRANO-[3,2-*c*]QUINOLINE-3-CARBO-NITRILE)-2'-OXINDOLE]

I. V. Ukrainets[1], R. G. Red'kin[1], L. V. Sidorenko[1], and A. V. Turov[2]**

A three-component condensation of 1-allyl-4-hydroxy-2-oxo-1,2-dihydroquinoline, isatin, and malononitrile gave a satisfactory yield of 4,3'-spiro[(6-allyl-2-amino-5-oxo-5,6-dihydro-4H-pyrano-[3,2-c]quinoline-3-carbonitrile)-2'-oxindole], which structure was confirmed by X-ray analysis.

Keywords: 4-hydroxy-2-oxo-1,2-dihydroquinoline, isatin, malononitrile, pyrano[3,2-*c*]quinoline, spiro[indole-3,4'-pyran], X-ray structural analysis.

Derivatives of 4-hydroxy-2-quinolone annelated by the pyran fragment along the *c* edge have long attracted the attention of synthetic chemists and other investigators. Such an interest is primarily because a 2H-pyrano[3,2-*c*]quinolin-5-one ring forms the basis of a series of natural alkaloids isolated from the plant family *Rutaceae,* including veprisine, flindersine, haplamine, paraensidimerine, and vepridimerine [2-5]. Amongst this series there are compounds having photochromic properties [6] that are able to block Ca^{2+} channels in cell membranes [7] or K^+ channels in thymocytes [8].

All of the synthetic methods for building pyrano[3,2-*c*]quinolin-5-one systems are based on the Michael addition of 4-hydroxy-2-quinolones to α,β-unsaturated carbonyl compounds [9-12]. The same principle was used by us in the preparation and study of the previously unreported 4-hydroxy-2-quinolones annelated by a spiro[indole-3,4'-pyran] ring.

Practically the preparation of these types of heterocyclic systems relates to different synthetic schemes, which we have discussed for the structurally closely related 4-hydroxycoumarin. One of the these involves an initial condensation of isatin with malononitrile after which 2-(2-oxoindolin-3-ylidene)malononitrile is introduced into a reaction with 4-hydroxycoumarin [13]. It should be noted immediately that such a method

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** To whom correspondence should be addressed, e-mail: uiv@kharkov.ua.

[1]National University of Pharmacy, Kharkiv 61002, Ukraine.
[2]Taras Shevchenko National University, Kiev 01033, Ukraine, e-mail: nmrlab@uiv.kiev.ua.

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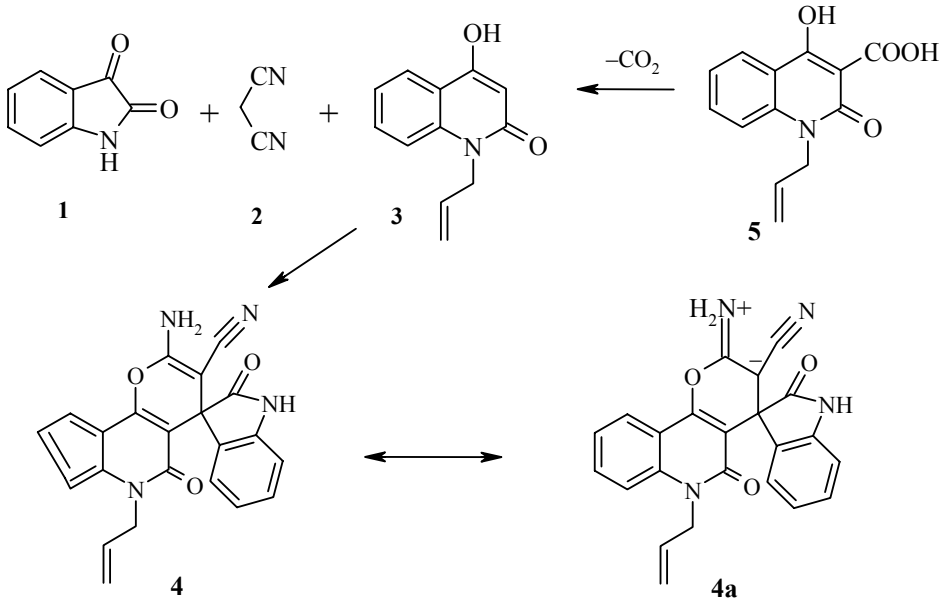
does not give good results and is hence used as a counter synthesis. A somewhat different scheme has some preparative value and consists of a three component condensation in which isatin **1**, malononitrile **2**, and 4-hydroxycoumarin are simultaneously introduced into the reaction and which, moreover, is considerably easier to carry out [13-15].

Experiments carried out by us have shown that exchange of 4-hydroxycoumarin for an aza analog, in particular 1-allyl-4-hydroxy-2-oxo-1,2-dihydroquinoline (**3**) does not prove to have any kind of effect on the course of the reaction and thus, it gives 4,3'-spiro[(6-allyl-2-amino-5-oxo-5,6-dihydro-4H-pyrano-[3,2-*c*]quinoline-3-carbonitrile)-2'-oxindole] (**4**).



The 4-hydroxy-2-oxo-1,2-dihydroquinoline-3-carboxylic acids, including the 1-allyl-substituted analog **5**, are very liable to decarboxylation [16], hence can be used in the synthesis of the pyrano-[3,2-*c*]quinolines **4**. It is clear the separation of the intermediate 3H-derivatives **3** is not needed.

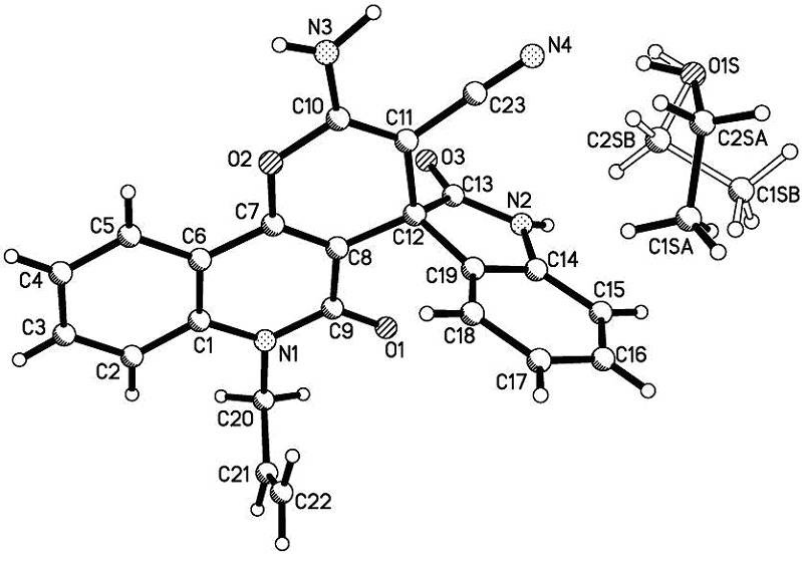


Fig. 1. The crystalline structure of the pyranoquinoline **4** ethanol solvate molecule.

TABLE 1. Bond Lengths (*l*) in the Structure of the Pyranoquinoline **4** Ethanol Solvate Molecule

Bond	*l*, Å	Bond	*l*, Å
N(1)–C(9)	1.388(2)	N(1)–C(1)	1.401(2)
N(1)–C(20)	1.476(2)	N(2)–C(13)	1.353(2)
N(2)–C(14)	1.403(2)	N(3)–C(10)	1.340(2)
N(4)–C(23)	1.157(2)	O(1)–C(9)	1.237(2)
O(2)–C(10)	1.365(2)	O(2)–C(7)	1.386(2)
O(3)–C(13)	1.223(2)	C(1)–C(2)	1.397(2)
C(1)–C(6)	1.412(2)	C(2)–C(3)	1.378(2)
C(3)–C(4)	1.390(2)	C(4)–C(5)	1.372(2)
C(5)–C(6)	1.397(2)	C(6)–C(7)	1.433(2)
C(7)–C(8)	1.350(2)	C(8)–C(9)	1.455(2)
C(8)–C(12)	1.508(2)	C(10)–C(11)	1.359(2)
C(11)–C(23)	1.424(2)	C(11)–C(12)	1.526(2)
C(12)–C(19)	1.519(2)	C(12)–C(13)	1.557(2)
C(14)–C(19)	1.383(2)	C(14)–C(15)	1.384(3)
C(15)–C(16)	1.385(3)	C(16)–C(17)	1.376(3)
C(17)–C(18)	1.382(3)	C(18)–C(19)	1.370(2)
C(20)–C(21)	1.476(3)	C(21)–C(22)	1.274(3)
O(1S)–C(2SA)	1.430(1)	O(1S)–C(2SB)	1.430(1)
C(1SA)–C(2SA)	1.540(1)	C(1SB)–C(2SB)	1.539(1)

TABLE 2. Valence Angles (ω) in the Pyranoquinoline **4** Ethanol Solvate Molecule

Angle	ω, deg	Angle	ω, deg
C(9)–N(1)–C(1)	123.0(1)	C(9)–N(1)–C(20)	117.3(1)
C(1)–N(1)–C(20)	119.6(1)	C(13)–N(2)–C(14)	112.3(1)
C(10)–O(2)–C(7)	118.9(1)	C(2)–C(1)–N(1)	121.9(1)
C(2)–C(1)–C(6)	119.1(1)	N(1)–C(1)–C(6)	119.1(1)
C(3)–C(2)–C(1)	120.2(2)	C(2)–C(3)–C(4)	121.0(2)
C(5)–C(4)–C(3)	119.3(2)	C(4)–C(5)–C(6)	121.2(2)
C(5)–C(6)–C(1)	119.2(1)	C(5)–C(6)–C(7)	123.1(1)
C(1)–C(6)–C(7)	117.7(1)	C(8)–C(7)–O(2)	122.9(1)
C(8)–C(7)–C(6)	123.2(1)	O(2)–C(7)–C(6)	113.9(1)
C(7)–C(8)–C(9)	119.2(1)	C(7)–C(8)–C(12)	122.9(1)
C(9)–C(8)–C(12)	117.9(1)	O(1)–C(9)–N(1)	120.8(1)
O(1)–C(9)–C(8)	121.4(1)	N(1)–C(9)–C(8)	117.8(1)
N(3)–C(10)–C(11)	126.2(1)	N(3)–C(10)–O(2)	111.9(1)
C(11)–C(10)–O(2)	121.9(1)	C(10)–C(11)–C(23)	119.2(1)
C(10)–C(11)–C(12)	123.5(1)	C(23)–C(11)–C(12)	117.2(1)
C(8)–C(12)–C(19)	114.4(1)	C(8)–C(12)–C(11)	108.6(1)
C(19)–C(12)–C(11)	110.6(1)	C(8)–C(12)–C(13)	112.7(1)
C(19)–C(12)–C(13)	101.1(1)	C(11)–C(12)–C(13)	109.3(1)
O(3)–C(13)–N(2)	127.3(1)	O(3)–C(13)–C(12)	125.3(1)
N(2)–C(13)–C(12)	107.4(1)	C(19)–C(14)–C(15)	122.0(2)
C(19)–C(14)–N(2)	109.3(2)	C(15)–C(14)–N(2)	128.7(2)
C(14)–C(15)–C(16)	117.0(2)	C(17)–C(16)–C(15)	121.3(2)
C(16)–C(17)–C(18)	120.9(2)	C(19)–C(18)–C(17)	118.7(2)
C(18)–C(19)–C(14)	120.1(2)	C(18)–C(19)–C(12)	130.7(1)
C(14)–C(19)–C(12)	109.2(1)	N(1)–C(20)–C(21)	114.1(1)
C(22)–C(21)–C(20)	127.5(2)	N(4)–C(23)–C(11)	178.2(2)
O(1S)–C(2SA)–C(1SA)	113.9(3)	O(1S)–C(2SB)–C(1SB)	98.3(2)

The results of the X-ray analysis carried out have shown the pyrano[3,2-*c*]quinoline **4** is isolated from the reaction mixture as a 1:1 solvate with ethanol (see Fig. 1 and Tables 1 and 2). Moreover, the solvated ethanol molecule is randomized in two positions with equal probability population. The quinolone fragment and the O(1) and C(20) atoms lie in a single plane within the accuracy of 0.02 Å. The 4H-pyran ring occurs in a strongly flattened boat conformation (folding parameters [17]: S = 0.17, θ = 71.9º, ψ = 2.8º). The deviations of atoms O(2) and C(12) from the mean square plane of the remaining ring atoms are -0.07 and -0.14 Å, respectively. The spiro linked fragments are twisted virtually perpendicularly to one another (torsional angle C(7)–C(8)–C(12)–C(19) 113.2(2)º). The non-equivalence of the bonds O(2)–C(7) 1.386(2) and O(2)–C(10) 1.365(2) Å in the dihydropyran ring should be noted and this is also seen in other spiro conjugate pyrans [18]. At the same time, in 4-alkyl- and 4-aryl-substituted derivatives this effect appeared to be non-typical [19, 20].

The powerful repulsion between the atoms of the allyl substituent and the quinolone fragment (shortened intramolecular contacts H(2)···C(20) 2.53 (the sum of van der Waal radii [21] 2.87), H(2)···H(20a) 2.07 (2.34), H(20a)···C(2) 2.59 (2.87), and H(20b)···O(1) 2.32 Å (2.46 Å)) leads to the lengthening of the N(1)–C(9) bond to 1.388(2) and N(1)–C(1) bond to 1.401(2) when compared with their mean values [22] of 1.353 and 1.371 Å, respectively. The vinyl part of the allyl substituent is placed virtually perpendicularly to the plane of the quinolone ring and this substituent occurs in a conformation close to *syn*-periplanar (torsional angles C(9)–N(1)–C(20)–C(21) 96.1(2)º and N(1)–C(20)–C(21)–C(22) -11.3(3)º, respectively).

The pyrano[3,2-*c*]quinoline **4** molecules are mutually bonded through the bridging ethanol molecules *via* the intermolecular hydrogen bonds N(2)–H(2N)…O(1s)' (-1-*x*, 2-*y*, -*z*) H···O 1.97 Å, N–H···O 175º and O(1s)–H(1sa)···N(4)' H···N 2.10 Å, O–H···N 164º to form dimers. In the crystal the complexes are arranged in infinite chains along the crystallographic (0 1 0) axis with the intermolecular bonds N(3)–H(3Na)···O(1s)' (-*x*, 2-*y*, -*z*) H···O 2.13 Å, N–H···O 165º and N(3)–H(3Nb) ···O(1)' (1+*x*, *y*, *z*) H···O 2.25 Å, N–H···O 158º.

The X-ray analysis carried out not only confirmed the structure of the pyrano[3,2-*c*]quinoline **4** synthesized but further demonstrated its considerable complexity thus presenting a particular interest for NMR investigation. Since the molecule contains two identical aromatic proton spin systems there exists some difficulty in interpretation. The most reliable assignment of these signals can be made by measuring the 2D COSY and NOESY spectra. Hence the COSY spectrum showed that a doublet for one of the aromatic protons having a chemical shift of 7.48 ppm was close in space to the allyl methylene substituent absorbing at 4.72 ppm. From the compound formula it follows that this can only occur in the quinolone fragment found in a *peri* position to the N-allyl substituent. Hence all of the signals spin related to the signal at 7.48 ppm were assigned to the quinolone molecular fragment and could be located by correlation in the COSY spectrum. It was found that all of the aromatic protons in the quinolone ring spin system absorb at a lower field than the signals in the indoline fragment. The cross peak coordinates in the COSY spectrum allow an assignment of all of the proton signals.

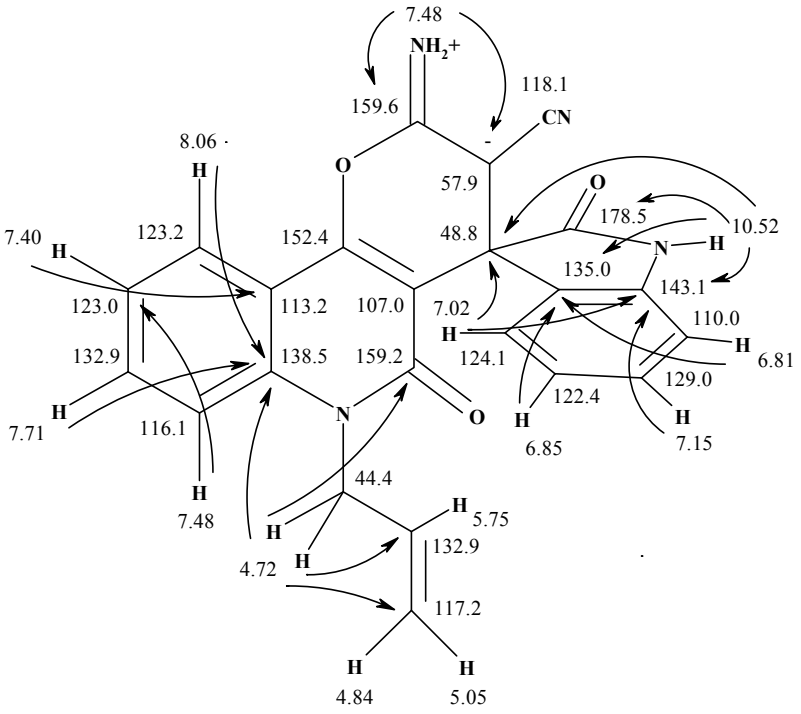
The ^{13}C NMR spectrum of the pyrano[3,2-*c*]quinoline **4** solvate showed the presence of the quaternary spiro atom carbon at 48.8 ppm and a signal for the allyl group methylene signal at 44.4 ppm. The carbon atoms of the ethanol solvate methyl and methylene groups appeared as signals at 19.3 and 56.7 ppm, respectively. Assignment of the signals in the aromatic part of the ^{13}C NMR spectrum can be readily made on the basis of the analysis of cross peaks in the 2D HMQC and HMBC spectra and are given in Table 3. Interpretation of the majority of the quaternary carbon atoms was carried out from the cross peaks due to spin interactions through three chemical bonds. The assignments are given in Scheme 1 with the most important HMBC correlations indicated by arrows.

In the HMBC spectrum correlations were only absent for two carbon atoms at 107.0 and 118.4 ppm. The first of the these corresponds to the bridging C(4a) atom and the second to the carbon of the nitrile group.

A somewhat unusual behavior was seen for the pyrano[3,2-*c*]quinoline **4** when deuterated trifluoroacetic acid was added to its solution in DMSO-d_6. No change that is usually observed in such examples was seen either in the proton or carbon spectrum. Even the active NH and NH_2 protons did not undergo deuterium exchange and remained in the spectrum.

TABLE 3. The Complete List of Heteronuclear $^{1}H–^{13}C$ Correlations Found for the Pyranoquinoline **4** Ethanol Solvate Molecule

^{1}H signals, δ, ppm	Position of the cross peaks in the ^{13}C measurement	
	HMQC	HMBC
10.52	—	178.5; 143.1; 135.0; 48.8
8.06	123.2	152.4; 138.5; 132.9
7.71	132.9	138.5; 116.1; 123.2
7.48	116.1	159.6; 152.4; 123.0; 113.2; 57.9
7.40	123.0	116.1; 113.2
7.15	129.0	143.1; 124.1
7.02	124.1	143.1; 129.0; 48.8
6.85	122.4	135.0; 110.0
6.81	110.0	135.0; 122.4
5.75	132.9	44.4
5.05	117.2	44.4
4.84	117.2	132.9; 44.4
4.72	44.4	159.2; 138.5; 132.9; 117.2



EXPERIMENTAL

The ^{1}H NMR and ^{13}C NMR spectra of the pyranoquinoline **4** solvate, COSY 2D ^{1}H NMR experiments, the NOESY-2D homonuclear Overhauser effect spectroscopy, and the heteronuclear HMQC and HMBC spectra were recorded on a Varian Mercury-400 spectrometer (400 and 100 MHz, respectively). All of the 2D experiments were carried out with gradient selection of useful signals. The mixing times in the pulse sequences were respectively $^{1}J_{CH}$ = 140 and $^{2\text{-}3}J_{CH}$ = 8 Hz. The number of increments in the COSY and HMQC

experiments was 128 and in the HMBC spectrum 400. The mixing time in the NOESY-2D experiment was 500 ms. In all cases the solvent was DMSO-d_6 and the internal standard TMS.

1-Allyl-4-hydroxy-2-oxo-1,2-dihydroquinoline (**3**) and 1-allyl-4-hydroxy-2-oxo-1,2-dihydroquinoline-3-carboxylic acid (**5**) were prepared by the known methods [16] and [23], respectively.

Solvate of 4,3'-Spiro[(6-allyl-2-amino-5-oxo-5,6-dihydro-4H-pyrano[3,2-*c*]quinoline-3-carbonitrile)-2'-oxindole] (4) with Ethanol. A mixture of 1-allyl-4-hydroxy-2-oxo-1,2-dihydroquinoline **3** (2.01 g, 0.01 mol), isatin **1** (1.47 g, 0.01 mol), malononitrile **2** (0.66 g, 0.01 mol), and triethanolamine (1.3 ml, 0.01 mol) in ethanol (20 ml) was refluxed for 2 h, cooled, and placed in a freezer at -5ºC for 24 h. The precipitated solvate crystals of the pyranoquinoline **4** with ethanol were filtered off, washed with hot hexane, and dried. Yield 2.56 g (58%); mp 313-315ºC (ethanol). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 10.52 (1H, s, NH); 8.06 (1H, d, *J* = 7.2, H-10); 7.71 (1H, t, *J* = 7.2, H-8); 7.48 (3H, m, H-7 and NH_2); 7.40 (1H, t, *J* = 7.6, H-9); 7.15 (1H, t, *J* = 7.6, H-6' indole); 7.02 (1H, d, *J* = 7.2, H-4' indole); 6.85 (1H, t, *J* = 7.6, H-5' indole); 6.81 (1H, d, *J* = 7.6, H-7' indole); 5.75 (1H, m, C<u>H</u>=CH_2); 5.05 (1H, d, *J* = 10.4, NCH_2CH=C<u>H</u> *cis*); 4.84 (1H, d, *J* = 17.2, NCH_2CH=C<u>H</u> *trans*); 4.72 (2H, d, *J* = 2.8, NCH_2); 4.39 (1H, t, *J* = 5.2, OH ethanol); 3.44 (2H, q, *J* = 5.2, CH_2 ethanol); 1.06 (3H, t, *J* = 7.2, CH_3 ethanol). ^{13}C NMR spectrum, δ, ppm: 178.5 (O=C-2'), 159.6 (H_2NC-2), 159.2 (C-5), 152.4 (C-10B), 143.1 (C-7'A), 138.5 (C-6A), 135.0 (C-3'A), 132.9 (C-8 + NCH_2<u>C</u>H), 129.0 (C-6'), 124.1 (C-4'), 123.2 (C-10), 123.0 (C-9), 122.4 (C-5'), 118.1 (C≡N), 117.2 (NCH_2CH=<u>C</u>H_2), 116.1 (C-7), 113.2 (C-10A), 110.0 (C-7'), 107.0 (C-4A), 57.9 (C-3), 56.7 (CH_3<u>C</u>H_2OH), 48.8 (C-4), 44.4 (NCH_2), 19.3 (<u>C</u>H_3CH_2OH). Found, %: C 67.98; H 5.14; N 12.57. $C_{23}H_{16}N_4O_3$.EtOH. Calculated, %: C 67.86; H 5.01; N 12.66.

When 1-allyl-4-hydroxy-2-oxo-1,2-dihydroquinoline-3-carboxylic acid (**5**) is used as starting material, it is treated as follows. Acid **5** (2.45 g, 0.01 mol) was added in small portions to refluxing DMF (5 ml). The poorly soluble acid rapidly decarboxylated to become the readily soluble 3H-derivative **3**, which was used without separation in the subsequent synthesis as described in the method above.

X-ray Structural Analysis. 1:1 Crystals of the pyranoquinoline **4** with ethanol are triclinic (ethanol). At 20ºC: *a* = 8.665(1), *b* = 10.364(2), *c* = 13.294(2) Å, α = 80.94(1)º, β = 83.52(1)º, γ = 71.69(2)º, *V* = 1116.7(3) Å^3, M_r = 42.47, *Z* = 2, space group $P\bar{1}$, d_{calc} = 1.316 g/cm^3, μ(MoKα) = 0.091 mm^{-1}, *F*(000) = 464. The parameters for the unit cell and intensities of 12,468 reflections (3916 independent with R_{int} = 0.040) were measured on an Xcalibur-3 diffractomer (MoKα radiation, CCD detector, graphite monochromator, ω-scanning to $2\theta_{max}$ = 50º).

The structure was solved by a direct method using the SHELXTL program package [24]. The positions of the hydrogen atoms were revealed from electron density difference synthesis and refined using the "riding" model with $U_{iso} = nU_{eq}$ (*n* = 1.5 for methyl groups and *n* = 1.2 for remaining hydrogen atoms). Hydrogen atoms taking part in the formation of hydrogen bonds were refined isotropically. The structure was refined *via* F^2 full-matrix least-squares analysis in the anisotropic approximation for non-hydrogen atoms to wR_2 = 0.110 for 3831 reflections (R_1 = 0.043 for 1757 reflections with $F > 4\sigma(F)$ and *S* = 0.810). The complete crystallographic information was placed in the Cambridge structural data base (reference CCDC 717536). Interatomic distances and valence angles are given in Tables 1 and 2.

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