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CONVENIENT METHOD FOR SYNTHESIS OF FUNCTIONALLY SUBSTITUTED HEXAHYDROQUINOLINES.
MOLECULAR AND CRYSTAL STRUCTURE OF 4-ISOPROPYL-7,7-DIMETHYL-5-OXO-3-CYANO-2-CYANOMETHYLTHIO-1,4,5,6,7,8-HEXAHYDROQUINOLINE

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We have obtained 2-alkylthio-7,7-dimethyl-5-oxo-4-ethyl(isopropyl)-3-cyano-1,4,5,6,7,8-hexahydroquinolines by reaction of 5,5-dimethylcyclohexane-1,3-dione, propionic (or isobutyric) aldehyde, cyanothioacetamide, and alkyl halides. We have established the structure of 4-isopropyl-7,7-dimethyl-5-oxo-3-cyano-2-cyanomethylthio-1,4,5,6,7,8-hexahydroquinoline by x-ray diffraction.

Functionally substituted hydrogenated 3-cyanopyridine-2(1H)-thiones are of considerable interest from the viewpoint of searching for new biologically active compounds with a broad spectrum of action [1-3]. In this respect, derivatives of hydrogenated quinolinethiones have been much less studied, undoubtedly because of the relatively smaller number of convenient methods for obtaining them. Among such methods the method we proposed earlier in [4, 5] stands out, based on reaction of arylmethylenecyanothioacetamides with dimedone. As a continuation of those studies, in this paper we have studied the reaction of dimedone, aliphatic aldehydes, and cyanothioacetamide in the presence of base, which allowed us to develop convenient methods for synthesis of functionally substituted hexahydroquinolinethiones and to study some of their properties.

On reaction of equimolar amounts of dimedone (I), aliphatic aldehyde (IIa, b), and cyanothioacetamide (III) in the presence of N-methylmorpholine at 20°C in ethanol, apparently the corresponding Knoevenagel condensation products are formed (IVa, b or Va, b), to which under the reaction conditions CH-acid I or III is added according to the Michael addition reaction. The adducts (VIa, b) appearing as a result of these processes undergo cyclocondensation to salts (VIIa, b). Subsequent S-alkylation by alkyl halides (VIIIa-e) yields sulfides (IXa-f) (method A). Acidification of the reaction mass with HCl before addition of halide VIII leads to formation of the substituted hexahydroquinolinethione (X), which confirms formation of salt VIIb in the reaction mixture. Sulfides IXa, b, f are also obtained by reaction of halides VIIIa-c with thione X in alkaline medium (method B).

Thus we improved the method for obtaining compounds IX by carrying out sequential reactions in a single production stage: alkylation of salts VII without isolation of the latter from the reaction mixture. This not only simplifies synthesis of sulfides IX but also increases their yield.

Cyclocondensation of dimedone, aliphatic aldehyde, and malononitrile probably occurs similarly. In this case, pyrans (XVIa, b) are obtained in quantitative yield through a stage of formation of substituted ethylenes (Va, b, XIIIa, b) and adducts (XIVa, b, XVa, b).

The PMR spectra of compounds IX, X, XVI contain signals from the protons of the dimedone moiety, the substituents R, X, Z, and the amino group (Table 1). Also characteristic are chemical shifts of the NH protons in the form of singlets at 9.45-12.43 ppm and the $C_{(4)}-H$ chemical shift in the form of a doublet or a triplet in the 3.33-4.40 ppm region.

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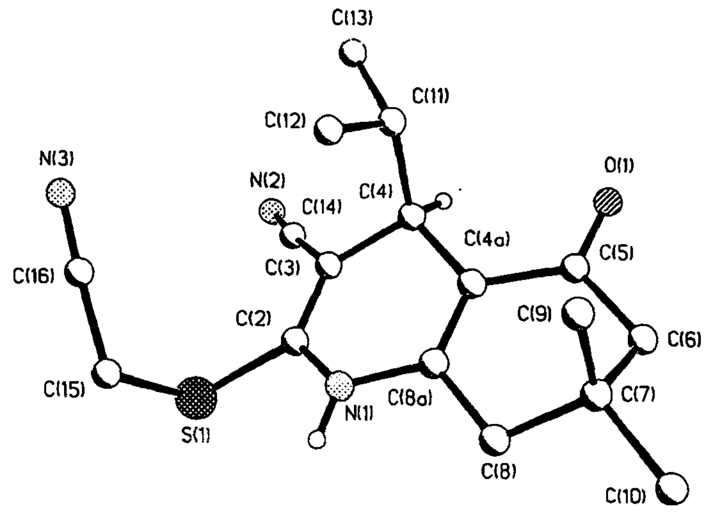
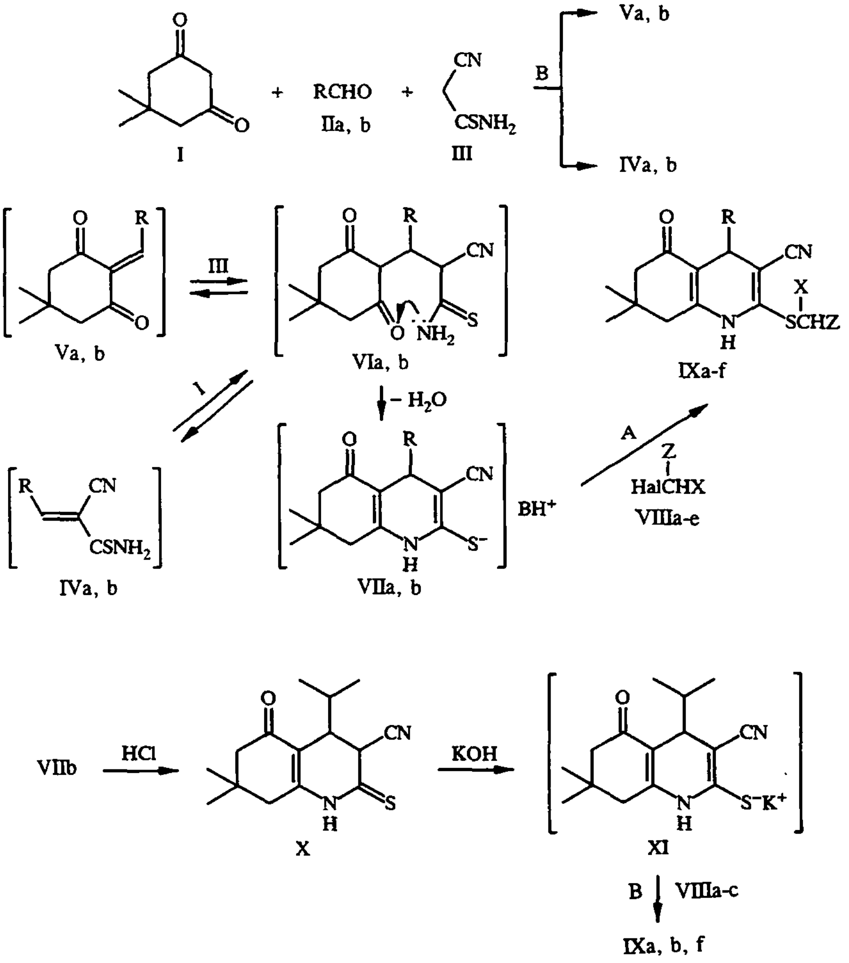


Fig. 1. General view of IXa molecule.

In the IR spectra, we observe absorption bands for stretching vibrations of the conjugated cyano group at 2188-2210 cm^{-1}, the amino group at 3182-3364 cm^{-1}, the C═O group at 1620-1705 cm^{-1}, and also the unconjugated cyano group at 2254 cm^{-1}, which confirms the structure of the products obtained (Table 2).

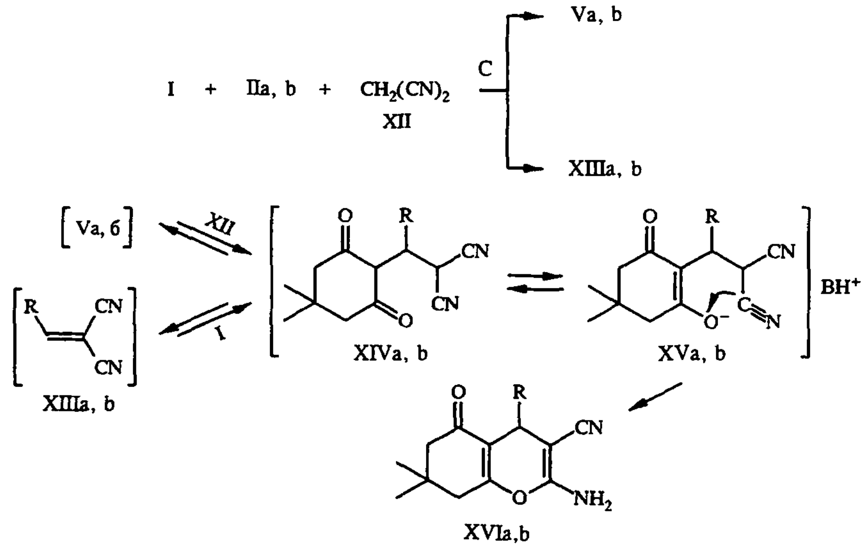


B = N-methylmorpholine; II, IV—VII a R = C_2H_5, b R = $CH(CH_3)_2$; VIII a Hal = Cl, Z = CN, X = H; b Hal = Cl, Z = 4-BrC_6H_4NHCO, X = H; c Hal = Cl, Z = $CONH_2$, X = Ph; d Hal = I, Z = H, X = H; e Hal = Cl, Z = $CONH_2$, X = H; IX a R = $CH(CH_3)_2$, Z = CN, X = H; b R = $CH(CH_3)_2$, Z = 4-BrC_6H_4NHCO, X = H; c R = C_2H_5, Z = $CONH_2$, X = Ph; d R = C_2H_5, Z = H, X = H; e R = C_2H_5, Z = $CONH_2$, X = H; f R = $CH(CH_3)_2$, Z = $CONH_2$, X = H

TABLE 1. PMR Spectra of Synthesized Compounds IX, X, XVI

Compound	Chemical shifts, δ, ppm						
	$C_{(7)}-(CH_3)_2$ s	$C_{(6)}-2H$ s	$C_{(8)}-2H$ s	$C_{(3)}-H$, $C_{(4)}-H$	NH, NH_2 s	R	CHX Z
IXa	1,03	2,38	2,20	3,40 d	9,84	1,65 m 0,75 d 0,85 d	4,22 s
IXb	1,01	2,38	2,16	3,33 d	10,01	1,53 m 1,65 d 1,80 d	7,54 m 10,75 s, 4,00 s
IXc	0,94, 1,01	2,30	2,15	3,35 t	9,52	0,74 t 1,33 m	7,99 д, 7,62 m 4,72 s
IXd	0,98, 1,02	2,33	2,15	3,41 t	9,45	0,75 t 1,34 m	2,48 s
IXe	0,99, 1,03	2,33 d	2,17 d	3,41 t	10,26	0,77 t. 1,35 m	7,55 s, 7,88 s, 3,67 d
IXf	1,03	2,35 d	2,17	3,32 d	10,10, 10,40	0,78 m 1,57 m	3,68 d. 7,57 s, 7,88 s
X	1,04	2,31	2,20	4,40 d 2,90 d	12,43	1,50 m 0,80 d 0,78 d	
XVIa	1,02	2,40	2,24	3,18 t	6,91	0,71 t, 1,50 m	
XVIb	1,03	2,43	2,26 d	3,07 d	6,96	0,67 d 0,92 d 1,73 m	

In order to determine the direction of the cyclocondensation described above and the regioselectivity of alkylation of the hydrogenated quinolinethione X by halides VIII, using x-ray diffraction we unambiguously established the structure of compound IXa. In Fig. 1, we show the general view of the molecule; the bond lengths and bond angles are given in Tables 3 and 4.



XIII—XVI a R = C_2H_5, b R = $CH(CH_3)_2$

In the investigated molecule, the dihydropyridine heterocycle has the boat conformation, the $N_{(1)}$ and $C_{(4)}$ atoms deviate from the plane of the remaining four atoms of the "bottom of the boat" of the heterocycle (planar to accuracy ±0.004 Å) by −0.144 and −0.307 Å respectively, which corresponds to bending of the ring along the lines $C_{(2)}...C_{(8a)}$ by 12.2°, $C_{(3)}...C_{(4a)}$ by 20.3°, $N_{(1)}...C_{(4)}$ by 20.7°. We established such a conformation for this ring in the previously investigated molecules of substituted 1,4-dihydropyridines, which contain an aryl substituent at $C_{(4)}$ [6].

TABLE 2. Characteristics of Synthesized Compounds IXa-f, X, XVIa, b

Compound	Empirical formula	Found % / Calculated %				mp, °C	IR spectra, ν, cm⁻¹			Yield, %, method A/B
		C	H	N	S		C=O	C ≡ N	NH, NH₂	
IXa	$C_{17}H_{21}N_3OS$	64,80 64,73	6,63 6,71	13,40 13,32	10,10 10,16	187 - 189*	1620	2205, 2254	3183	80/75
IXb	$C_{23}H_{26}BrN_3O_2S$	56,44 56,56	5,43 5,37	8,72 8,60	6,49 6,56	242 - 244	1624, 1670	2188	3300, 3195	68/60
IXc	$C_{22}H_{25}N_3O_2S$	66,90 66,81	6,24 6,37	10,55 10,62	8,22 8,11	136 - 138	1610 sh, 1705	2204	3270, 3182	88
IXd	$C_{15}H_{20}N_2OS$	65,11 65,18	7,39 7,29	10,05 10,14	11,68 11,60	192 - 194	1622 sh	2193	3165	90
IXe	$C_{16}H_{21}N_3O_2S$	60,23 60,16	6,55 6,63	13,07 13,16	10,13 10,04	215 - 217*	1620 sh, 1680	2206	3195, 3422	81
IXf	$C_{17}H_{23}N_3O_2S$	61,30 61,23	6,84 6,95	12,55 12,60	9,70 9,62	233 - 235*	1654, 1710	2200	3210, 3425	72/70
X	$C_{15}H_{20}N_2OS$	65,24 65,18	7,21 7,29	10,20 10,14	11,56 11,60	201 - 203	1642	2245	3170	72
XVIa	$C_{14}H_{18}N_2O_2$	68,38 68,27	7,25 7,37	11,49 11,37		174 - 176	1650 sh	2190	3200, 3285, 3364	77
XVIb	$C_{15}H_{20}N_2O_2$	69,33 69,20	7,61 7,74	10,68 10,76		183 - 185	1658 sh	2210 sh	3195, 3308, 3364	85

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*The compound was recrystallized from 1-butanol.

TABLE 3. Bond Lengths *d* (Å) in the IXa Molecule

Bond	*d*/Å	Bond	*d*/Å	Bond	*d*/Å
$S_{(1)}$—$C_{(2)}$	1,768(2)	$C_{(3)}$—$C_{(4)}$	1,521(3)	$C_{(7)}$—$C_{(8)}$	1,527(3)
$S_{(1)}$—$C_{(15)}$	1,824(3)	$C_{(3)}$—$C_{(14)}$	1,427(3)	$C_{(7)}$—$C_{(9)}$	1,530(4)
$O_{(1)}$—$C_{(5)}$	1,232(2)	$C_{(4)}$—$C_{(4a)}$	1,511(3)	$C_{(7)}$—$C_{(10)}$	1,530(4)
$N_{(1)}$—$C_{(2)}$	1,375(3)	$C_{(4)}$—$C_{(11)}$	1,551(3)	$C_{(8)}$—$C_{(8a)}$	1,499(3)
$N_{(1)}$—$C_{(8a)}$	1,375(2)	$C_{(4a)}$—$C_{(5)}$	1,446(3)	$C_{(11)}$—$C_{(12)}$	1,510(4)
$N_{(2)}$—$C_{(14)}$	1,141(3)	$C_{(4a)}$—$C_{(8a)}$	1,354(2)	$C_{(11)}$—$C_{(13)}$	1,524(6)
$N_{(3)}$—$C_{(16)}$	1,135(5)	$C_{(5)}$—$C_{(6)}$	1,504(3)	$C_{(15)}$—$C_{(16)}$	1,447(5)
$C_{(2)}$—$C_{(3)}$	1,352(3)	$C_{(6)}$—$C_{(7)}$	1,527(3)		

TABLE 4. Bond Angles ω (degrees) in IXa Molecule

Angle	ω	Angle	ω
$C_{(2)}$—$S_{(1)}$—$C_{(15)}$	99,4(1)	$C_{(5)}$—$C_{(6)}$—$C_{(7)}$	114,4(2)
$C_{(2)}$—$N_{(1)}$—$C_{(8a)}$	121,0(2)	$C_{(6)}$—$C_{(7)}$—$C_{(8)}$	107,6(2)
$S_{(1)}$—$C_{(2)}$—$N_{(1)}$	115,9(1)	$C_{(6)}$—$C_{(7)}$—$C_{(9)}$	109,7(2)
$S_{(1)}$—$C_{(2)}$—$C_{(3)}$	123,6(2)	$C_{(8)}$—$C_{(7)}$—$C_{(9)}$	110,9(2)
$N_{(1)}$—$C_{(2)}$—$C_{(3)}$	120,3(2)	$C_{(6)}$—$C_{(7)}$—$C_{(10)}$	110,2(2)
$C_{(2)}$—$C_{(3)}$—$C_{(4)}$	121,7(2)	$C_{(8)}$—$C_{(7)}$—$C_{(10)}$	109,3(2)
$C_{(2)}$—$C_{(3)}$—$C_{(14)}$	120,3(2)	$C_{(9)}$—$C_{(7)}$—$C_{(10)}$	109,1(2)
$C_{(4)}$—$C_{(3)}$—$C_{(14)}$	117,9(2)	$C_{(7)}$—$C_{(8)}$—$C_{(8a)}$	113,3(2)
$C_{(3)}$—$C_{(4)}$—$C_{(4a)}$	108,6(2)	$N_{(1)}$—$C_{(8a)}$—$C_{(4a)}$	120,1(2)
$C_{(3)}$—$C_{(4)}$—$C_{(11)}$	113,9(2)	$N_{(1)}$—$C_{(8a)}$—$C_{(8)}$	115,9(2)
$C_{(4a)}$—$C_{(4)}$—$C_{(11)}$	112,1(2)	$C_{(4a)}$—$C_{(8a)}$—$C_{(8)}$	124,0(2)
$C_{(4)}$—$C_{(4a)}$—$C_{(5)}$	119,0(2)	$C_{(4)}$—$C_{(11)}$—$C_{(12)}$	112,9(2)
$C_{(4)}$—$C_{(4a)}$—$C_{(8a)}$	121,9(2)	$C_{(4)}$—$C_{(11)}$—$C_{(13)}$	111,6(2)
$C_{(5)}$—$C_{(4a)}$—$C_{(8a)}$	119,1(2)	$C_{(12)}$—$C_{(11)}$—$C_{(13)}$	111,3(3)
$O_{(1)}$—$C_{(5)}$—$C_{(4a)}$	120,5(2)	$N_{(2)}$—$C_{(14)}$—$C_{(3)}$	174,4(2)
$O_{(1)}$—$C_{(5)}$—$C_{(6)}$	120,7(2)	$S_{(1)}$—$C_{(15)}$—$C_{(16)}$	112,1(2)
$C_{(4a)}$—$C_{(5)}$—$C_{(6)}$	118,7(2)	$N_{(3)}$—$C_{(16)}$—$C_{(15)}$	177,8(4)

The cyclohexenone ring has the conformation of a distorted half-chair: the $C_{(6)}$ and $C_{(7)}$ atoms deviate on different sides from the moiety $C_{(8)}$, $C_{(8a)}$, $C_{(4a)}$, $C_{(5)}$ (planar to accuracy ±0.015 Å) by 0.062 and -0.605 Å respectively, and the dihedral angle between the considered planar moieties of the molecule is equal to 12.0°, i.e., the bicyclic system is slightly flattened (see Fig. 1).

The orientation of the isopropyl substituent at the $C_{(4)}$ atom relative to the "bottom of the boat" of the heterocycle in other moieties of the molecule is such that the number of constrained intramolecular nonvalence contacts is minimum ($C_{(13)}\ldots C_{(14)}$ 3.224(3) Å; twice the van der Waals radius of the C atom, 3.40 Å [7]). However, this eliminates the possibility of free rotation about the $C_{(4)}\ldots C_{(11)}$ bond.

The spatial arrangement of the S-cyanomethylene moiety relative to the "bottom of the boat" of the heterocycle is characterized by the values of the torsional angles: $C_{(3)}C_{(2)}S_{(1)}C_{(15)}$ −111.4°C, $C_{(2)}S_{(1)}C_{(15)}C_{(16)}$ 70.1°, $S_{(1)}C_{(15)}C_{(16)}N_{(3)}$ −88.8°.

The bond lengths and bond angles in the investigated molecule are comparable with those we established earlier in 1,4-dihydropyridines [8] and with the standard values in [9].

In the crystal, the intermolecular hydrogen bonds $N_{(1)}-H_{(1)}\ldots O_{(1)}$ (x, $-0.5 - y$, $-0.5+z$) ($N_{(1)}\ldots O_{(1)}$ 2.775(3), $N_{(1)}\ldots H_{(1)}$ 0.85(2), $H_{(1)}\ldots O_{(1)}$ 1.95(2) Å, angle $N_{(1)}-H_{(1)}\ldots O_{(1)}$ 166(1)°) join the IXa molecules into infinite chains along the z axis.

EXPERIMENTAL

The PMR spectra were taken on a Bruker WP-100 SY (100 MHz) in DMSO-D_6 (internal standard TMS). The IR spectra were taken on an IKS-29 spectrophotometer in Vaseline oil. The course of the reaction and the purity of the compounds were monitored using TLC on Silufol UV-254 plates; eluent, acetone−hexane (3:5).

TABLE 5. Atomic Coordinates ($\times 10^4$; for H, $\times 10^9$) in IXa Molecule

Atom	x	y	z
$S_{(1)}$	3194(1)	1089(1)	450(1)
$O_{(1)}$	3342(2)	-3111(2)	3967(1)
$N_{(1)}$	3126(2)	-1303(2)	897(1)
$N_{(2)}$	2863(3)	2148(2)	3015(2)
$N_{(3)}$	114(3)	1515(3)	612(3)
$C_{(2)}$	2990(2)	-138(2)	1263(1)
$C_{(3)}$	2787(2)	23(2)	2206(1)
$C_{(4)}$	2589(2)	-1062(2)	2872(1)
$C_{(4a)}$	3129(2)	-2193(2)	2453(1)
$C_{(5)}$	3462(2)	-3195(2)	3091(1)
$C_{(6)}$	3981(2)	-4327(2)	2668(2)
$C_{(7)}$	3584(2)	-4598(2)	1628(1)
$C_{(8)}$	3761(2)	-3432(2)	1037(2)
$C_{(8a)}$	3311(2)	-2295(2)	1499(1)
$C_{(9)}$	2332(3)	-4971(3)	1595(2)
$C_{(10)}$	4283(3)	-5647(3)	1218(2)
$C_{(11)}$	1323(2)	-1277(3)	3084(2)
$C_{(12)}$	589(3)	-1485(4)	2181(3)
$C_{(13)}$	855(3)	-244(5)	3696(3)
$C_{(14)}$	2810(2)	1228(2)	2618(2)
$C_{(15)}$	1898(3)	935(3)	-305(2)
$C_{(16)}$	899(3)	1281(3)	203(2)
$H_{(1)}$	326(2)	-137(2)	31(1)
$H_{(4)}$	296(2)	-87(2)	347(1)
$H_{(61)}$	383(2)	-500(2)	308(2)
$H_{(62)}$	474(2)	-420(2)	268(2)
$H_{(81)}$	348(2)	-349(2)	42(2)
$H_{(82)}$	455(2)	-331(2)	93(2)
$H_{(91)}$	220(2)	-569(3)	197(2)
$H_{(92)}$	184(2)	-428(2)	188(2)
$H_{(93)}$	211(2)	-510(3)	89(2)
$H_{(101)}$	420(2)	-636(2)	160(2)
$H_{(102)}$	505(3)	-539(3)	118(2)
$H_{(103)}$	406(2)	-583(2)	57(2)
$H_{(11)}$	129(2)	-199(2)	346(2)
$H_{(121)}$	89(2)	-209(3)	171(2)
$H_{(122)}$	57(2)	-73(3)	178(2)
$H_{(123)}$	-4(3)	-173(3)	235(2)
$H_{(131)}$	133(3)	-5(3)	426(3)
$H_{(132)}$	19(3)	-53(3)	397(2)
$H_{(133)}$	76(3)	46(3)	334(2)
$H_{(151)}$	176(2)	7(2)	-51(2)
$H_{(152)}$	201(2)	144(2)	-79(2)

Crystals of Compound IXa are monoclinic, at 20°C: $a = 11.797(4)$, $b = 10.860(2)$, $c = 13.871(4)$ Å, $\beta = 92.38(2)°$, $V = 1776(1)$ Å^3, $d_{calc} = 1.185$ g/cm^3, $Z = 4$, space group $P2_1/c$. The cell parameters and the intensities of 4207 independent reflections were measured on a Siemens P3/PC four-circle automatic diffractometer (λMoKα, graphite monochromator, $\theta/2\theta$ scanning to $\theta_{max} = 27°$). The structure was deciphered by the direct method, determining all the nonhydrogen atoms, and refined by full-matrix least-squares in the anisotropic approximation for the nonhydrogen atoms from 2778 reflections with $I > 3\sigma(I)$. All the hydrogen atoms were objectively determined by Fourier difference syntheses and refined isotropically. The final values of the R factors were $R = 0.043$, $R_w = 0.043$ ($S = 0.616$). All the calculations were done using the SHELXTL PLUS program [9] (PC version). The coordinates of the atoms are given in Table 5 (the thermal parameters of the atoms can be obtained from the authors).

2-Alkylthio-7,7-dimethyl-5-oxo-4-R-3-cyano-1,4,5,6,7,8-hexahydroquinolines (IXa-f). A. A mixture of 1.40 g (10 mmoles) dimedone I, 10 mmoles aldehyde IIa, b, 1.00 g (10 mmoles) 2-cyanothioacetamide III, and 1.2 ml N-methylmorpholine in 20 ml ethanol was stirred at 20°C for 4 h. Then 10 mmoles alkyl halide VIIIa-e was added and this was stirred for 3 h. The precipitate formed was filtered off and washed successively with water, ethanol, and hexane. Obtained: compounds IXa-f (Tables 1, 2).

B. A 5.6 ml portion of 10% aqueous KOH solution and 10 mmoles alkyl halide VIIIa-c were added with stirring to a solution of 2.76 g (10 mmoles) thione X in 8 ml DMF. Then this was stirred at 25°C for 3 h. The precipitate formed was separated and then washed successively with water, ethanol, and hexane. Obtained: compounds IXa, b, f, similar in melting point and IR spectra to the compounds synthesized by method A.

4-Isopropyl-7,7-dimethyl-5-oxo-3-cyano-3,4,5,6,7,8-hexahydroquinoline-2-(1H)-thione (X). A mixture of 1.40 g (10 mmoles) dimedone I, 0.72 g (10 mmoles) aldehyde IIb, 1.00 g (10 mmoles) 2-cyanothioacetamide III, and 1.2 ml N-methylmorpholine in 20 ml ethanol was stirred at 25°C for 4 h. Then 4.5 ml 10% solution of hydrochloric acid was added and it was stirred for 30 min. The precipitate formed was filtered off and washed with ethanol. Obtained: compound X (Tables 1, 2).

2-Amino-4-R-7,7-dimethyl-5-oxo-3-cyano-5,6,7,8-tetrahydro-4H-benzo[b]pyrans (XVIa, b). A mixture of 1.40 g (10 mmoles) dimedone I, 10 mmoles aldehyde IIa, b, 0.66 g (10 mmoles) malonodinitrile XII, and 0.1 ml N-methylmorpholine in 20 ml ethanol was stirred at 25°C for 5 h. The precipitate formed was filtered off and washed with ethanol. Obtained: compounds XVIa, b (Tables 1, 2).

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