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NEW DATA ON THE REACTION OF 1,3-DIARYL-3-(2-OXOCYCLOHEXYL)-1-PROPANONES WITH HYDROGEN SULFIDE AND METHANOL

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The stereochemical aspects of the reaction of 1-aryl- and 1,3-diaryl-substituted 3-(2-oxo-cyclohexyl)-1-propanones with hydrogen sulfide and methanol have been discussed. A mechanism is proposed for the formation of trans, trans-1-methoxy-3,5-diaryl-2-thia(oxa)bicyclo-[4.4.0]dec-3-enes.

1-Aryl- and 1,3-diaryl-3-(2-oxocyclohexyl)-1-propanones (I-IV) react with hydrogen sulfide and methanol under conditions of acid catalysis to form bicyclic thioacetals [1]. By analogy with 2-methoxy-2,4-diphenylhexahydrochromene [2] the latter were erroneously considered to be derivatives of 2-methoxyhexahydrothiochromene [1]. Study of the structure of these compounds by PMR [3] and x-ray diffraction [4] showed that they are trans, trans-1-methoxy-3,5-diaryl-2-thiabicyclo[4.4.0]dec-3-enes (VII-X); this caused us to reconsider the reaction mechanism that we had previously proposed [1].

Continuing our work on the bicyclic thioacetals, we have studied the composition and structure of the products of the reaction of the 1,5-diketones I-VI with methanol and hydrogen sulfide at 10-15°C, and have also obtained data on the structure of the starting dicarbonyl compounds. The structural assignments were based on ^{13}C NMR analysis.

It was established that under the conditions described, 1,3-diaryl-3-(2-oxocyclohexyl)-1-propanones (I-IV) form trans-1-methoxy-2-phenyl- (VII) and trans, trans-1-methoxy-3,5-diaryl-2-thiabicyclo[4.4.0]dec-3-enes (VIII-X). The monoaryl-substituted 1,5-diketone I gave, along with trans-thioacetate VII, a small amount of cis-isomer XI. In tests with diaryl substituted 1,5-diketones, cis-thioacetals were not found. Under the same conditions when both hydrogen sulfide and methanol were present in the reaction mixture, 1,5-diketones V and VI reacted with only one of the nucleophiles, viz., methanol, to form trans, trans-1-methoxy-3,5-diaryl-2-oxabicyclo[4.4.0]dec-3-enes (XII, XIII).

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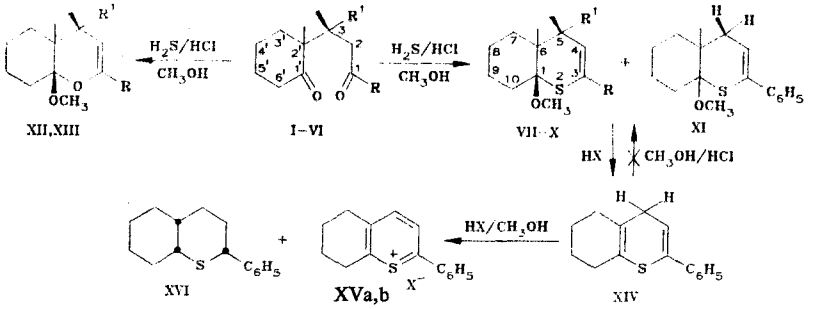
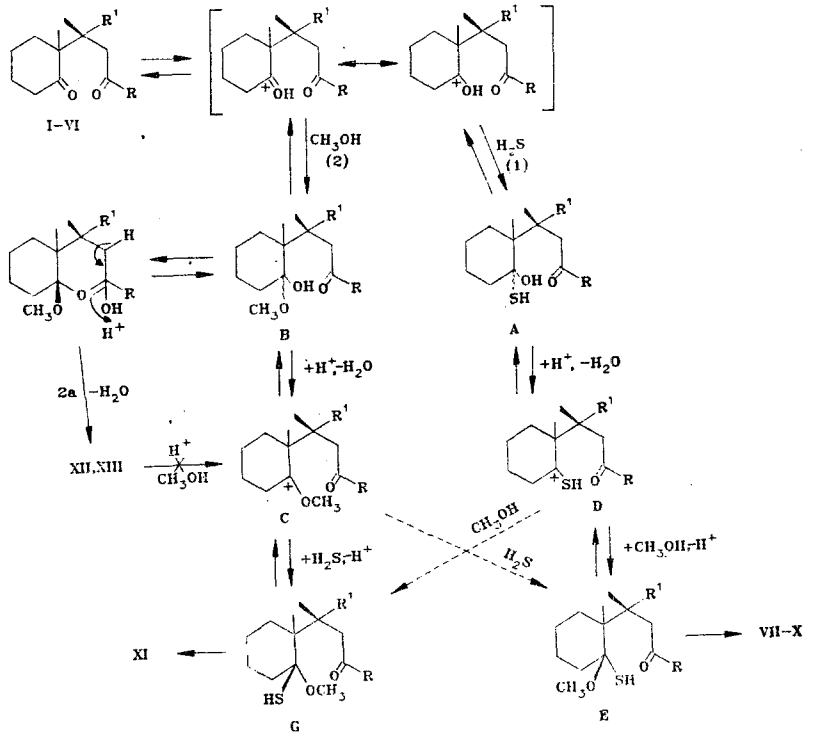


I, V—VII, XII, XIII R=C_6H_5, II, VIII R=$C_6H_4OCH_3$-4, III, IV, IX, X R=$C_{10}H_7$-β;
I, VII R'=H; II, III, V, VIII, IX, XII R'=C_6H_5, IV, VI, X, XIII R'=$C_6H_4OCH_3$-4

In [1] it was conjectured that the thioacetals originate by addition of methanol to 5,6-tetramethylenethiopyranes such as XIV. But when a methanol solution of 2-phenyl-5,6-tetramethylene-4H-thiopyrane (XIV) [5] was saturated with hydrogen chloride we were unable to find thioacetals VII or XI in the reaction mixture. When the reaction mixture was held a long time at room temperature, there occurred the usual disproportionation of 4-H-thiopyrane XIV to the 5,6-tetramethylenethiopyrylium salt XVa and cis,cis-2-phenyl-1-thiadecalin, XVI [6]. At the same time it was found that under the influence of mineral acid (HCl or $HClO_4$) in methanol or acetic acid, thioacetals VII and XI are converted to the disproportionation products of XIV, viz., salt XVa or XVb and thiadecalin XVI (XIV was identified chromatographically).

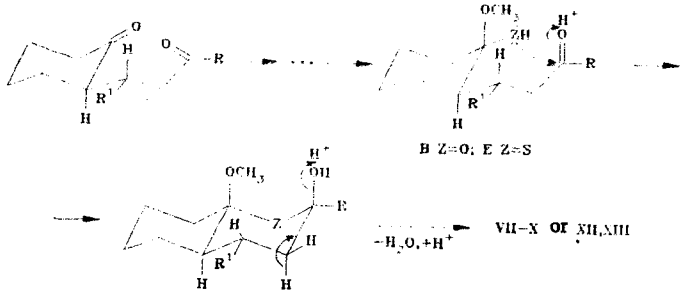
These results confirm that the reaction with nucleophiles (hydrogen sulfide, methanol), proceeds at the more reactive carbonyl of the alicycle. The activated alicyclic carbonyl first reacts with hydrogen sulfide or methanol; then the hydroxyl group in the diastereomeric gem-hydroxythiol (A) or hemiacetal (B) is replaced by methoxy or sulfhydryl groups respectively (routes 1 and 2). The subsequent cyclodehydration of the "semicyclic" thioacetal (E) forms products VII-X with trans,trans configuration.



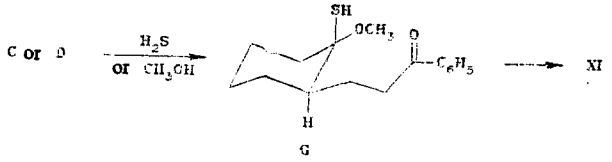
For route (2) we must allow the side reaction of dehydration of the "semicyclic" hemiacetal B to form trans,trans-acetals like XII and XIII. Thus despite the presence in the reaction mixture of both nucleophiles (H_2S, CH_3OH), V and VI form only trans,trans-1-methoxy-3,5-diaryl-2-oxabicyclo[4.4.0]dec-3-enes (XII, XIII) [3, 7]. The explanation must probably be sought in the difference in the rates of the competing reactions (1) and (2), and later (2a). The S-analogs of XII and XIII do not form even when the hydrogen sulfide concentration

per weight of starting diketone is increased; this is done by adding the diketone in portions to methanol presaturated with hydrogen sulfide. Under these conditions XII and XIII are not converted to their S-analogs; possibly the reaction is prevented by their poor solubility in methanol.

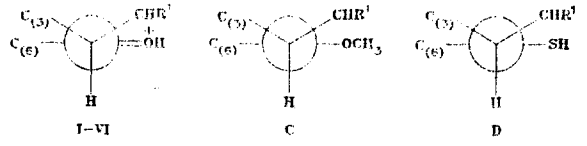
Consideration of the stereochemical features of the formation of thioacetals VII-XI and acetals XII and XIII presents well known difficulties because the intermediates can not be isolated due to their high reactivity. Aside from the structure of the final products, we can form an opinion on "semicyclic" hemiacetal B and hemithioacetal E, the structures of which must be most favorable in configuration and conformation for the formation of the final acetals XII and XIII or the thioacetals VII-X. The same can be said of intermediate G from which cis-isomer XI forms.



The diagrammed trans-diequatorial disposition of the reacting groups ought to promote the intramolecular cyclization of intermediates B or E to form XII and XIII or VII-X with the trans,trans-configuration. In intermediate G the axial-equatorial disposition of the reacting groups should give cis-isomers like XI.



Heterocyclization of intermediate G to cis-thioacetal XI is possible only in the absence of a second aryl group (R^1 = H), i.e., when the acyclic segment at $C(_2)$ in the alicycle has sufficient conformational mobility. The lower yield of XI than of VII, 1:7, and the absence of cis-isomers in tests with diaryl-substituted 1,5-diketones confirm this assumption.



It is known [8] that in 2-methylcyclohexanone the projection angle between the equatorial methyl and the carbonyl is 15°. The stereochemistry of the reaction of a nucleophilic compound at the carbonyl of a cyclic ketone has been thoroughly studied [8]. Usually the nucleophile adds at the less shielded side of the carbonyl bond. In the 1,5-diketones I-VI the equatorial ortho-substituent and the carbonyl oxygen are close to a shielded position; this can easily be seen when Dreiding models or Newman projections are constructed. As the projections show, the stereo-chemical conditions for attack on protonated 1,5-diketones and the intermediate ions C and D should be identical. The bulky and conformationally "rigid" ortho-substituent should screen off equatorial attack. Apparently the steric factor favors axial attack by the nucleophile, e.g., methanol, which also determines the formation of acetals XII and XIII of trans,trans configuration (if reaction with methanol is faster than with hydrogen sulfide). It must be supposed that intermediate E probably arises by axial attack by methanol on cation D; equatorial attack by hydrogen sulfide on cation C is sterically hindered.

As a result of axial attack cation C should form intermediate G which cannot undergo intramolecular cyclization in the presence of a second aryl substituent R^1, as confirmed by experiment. Indeed, in this case cis-thioacetals do not form. The same intermediate G can be obtained by equatorial attack by methanol on cation D; but if we consider that thanks to the anomeric effect the methoxyl tends to occupy an axial position, then the D → G conversion is more probable than the C → G conversion. Thus, in view of the evident steric unfavorability it is not possible to completely exclude equatorial attack during the C → E and D → G conversions from consideration.

Formation of cation C can be initiated by cleavage of bicyclic acetals such as XII and XIII in the presence of acid in a polar solvent; in the absence of other nucleophiles we can expect reaction with hydrogen sulfide. As noted above, in methanol XII and XIII could not be converted to the respective thioacetals. The reaction of acetals like XII and XIII with hydrogen sulfide in acetic acid will be reported separately.

Tables 1 and 2 show the experimental ^{13}C NMR spectra of the starting 1,5-diketones I, II, IV-VI, and their reaction products with hydrogen sulfide and methanol, VII, VIII, X-XIII. Spectral lines were assigned by means of off-resonance spectra, by comparison of integrated intensity and multiplet character of lines, and by comparison with calculated chemical shifts.*

As Table 1 shows, the spectra of the 1,5-diketones I, II, IV-VI are generally similar. The aliphatic and alicyclic parts of the spectrum have ten sharp signals that are evidence for their conformational and configurational homogeneity. Of the two possible isomers (threo and erythro), the energetically more favored threo form is realized, with a substituent in the equatorial position. 1,3-Axial interactions destabilize the conformer with a bulky substituent in axial position. It is known that even the methyl in 2-methylhexanone occupies the equatorial position. The spectrum of III could not be obtained because of its poor solubility in $CDCl_3$. The chemical shift of the alicyclic carbonyl carbon in diketones (see Table 1) is not substantially different from that in cyclohexanone ($\delta C{=}O$ 208.9 ppm), whereas the alkylaromatic carbonyl carbon appears in a stronger field, especially when the aryl segment contains an electron donor group, as in II. The observed changes in the carbonyl carbon signals also confirm the proposed mechanism of the reaction of the 1,5-diketones with hydrogen sulfide and methanol.

The structures of VII-XIII were previously established by us [3] on the basis of PMR spectra, while x-ray diffraction data were also obtained for VIII [4]. These compounds have trans,trans,trans configuration [3, 4]. Table 2 shows the ^{13}C NMR spectra of thioacetals VII, VIII, X, and XI and acetals XII and XIII. The problem was to make configurational assignments for the isomers of VII-XI, using the spectral parameters of VII-XIII of known configurations. As Table 2 shows, the spectra of thioacetals VII, VIII, X, and XI and acetals XII and XIII are similar, and for the oxygen analogs there is a noticeable weak-field shift of the heterocyclic α-carbon $C(_1)$ and $C(_3)$ signals of 12 and 6 ppm respectively, due to the presence of the highly electronegative heteroatom. At the same time the signals of $C(_4)$ and $C(_6)$ which are located in γ-position to oxygen undergo a strong-field shift.

In going from trans,trans-1-methoxy-3-phenyl-2-thiabicyclo[4.4.0]dec-z-ene (VII) to its isomer IX, the ^{13}C NMR spectrum does not show any change of location or multiplet character of the phenyl or methoxy signals. The location of the double bond is also retained, as evidenced by the vinyl proton signals in the PMR spectra of VII and XI (see Experimental). These facts show that isomers VII and XI apparently differ in the way the rings are joined. In both cases the heterocycle has the semi-chair conformation, but the nature of the binary doublet of the vinyl proton differs. In trans isomer VII this doublet is deformed because of remote interaction with the axial proton at $C(_6)$, whereas in cis isomer XI the vinyl proton signal is a distinct binary doublet with $^3J_{45}$ = 6.36 and 2.82 Hz. In trans isomer VII, as in VIII, for which x-ray diffraction analysis is given, the alicycle has the chair conformation. In cis isomer XI the alicycle conformation has changed to the twist form. The presumption of twist form accounts for the γ-effects of the axial bonds (~2 ppm) that affect the $C(_8)$ and $C(_4)$ shifts, and for the absence of an effect of axial CH_3O on the $C(_9)$ signal.

*Calculations were carried out with allowance for known parameters of ^{13}C chemical shifts of cis- and trans-1-thiadecalins [9], increments of aryl substituents in cis-1-thiadecalins [10], and ^{13}C chemical shifts in substituted benzene [12].

TABLE 1. ^{13}C Chemical Shifts of 3-(2-Oxocyclohexyl)-1-propanones I, II, IV-VI*

Compound	mp, deg C (from ethanol)	$C_{(1')}$	$C_{(2')}$	$C_{(3')}$	$C_{(4')}$	$C_{(5')}$	$C_{(6')}$	$C_{(1)}$	$C_{(2)}$	$C_{(3)}$	R	R^1
I	52—53	211,51	49,19	33,66	24,28	27,28	41,32	199,01	35,49	23,75	136,20; 127,75; 127,25; 132,07	—
II	135—136	213,47	55,68	32,21	23,87	28,35	42,10	197,13	43,67	42,12	129,96; 130,28; 113,42; 163,06 (55,20)	141,92; 128,24; 128,24; 126,,38
IV	137—138	213,32	55,72	32,19	23,89	28,26	42,07	198,54	44,21	40,37	135,13; 133,59; 132,21; 129,63; 129,32; 129,20; 127,93; 127,88; 126,28; 123,71	134,08; 128,97; 113,57; 157,99 (54,72)
V	148—149	213,15	55,62	32,34	23,92	28,26	42,11	198,52	44,02	40,96	136,99; 128,23; 127,94; 132,54	141,91; 128,23; 128,23; 126,38
VI	145—146	213,44	55,81	32,18	23,91	28,31	42,09	198,75	44,16	40,18	136,93; 128,24; 127,97; 132,56	133,74; 129,05; 113,67; 157,98 (54,93)

*For R and R^1 are shown the chemical shifts of quaternary, ortho-, meta, and para- ring carbons, respectively; parentheses indicate methoxy signals of naphthyl substituent not assigned.

TABLE 2. ^{13}C Chemical Shifts of 1-Methoxy-2-thiabicyclo[4.4.0]dec-3-enes (VII, VIII, X, XI) and trans,trans-1-Methoxy-2-phenyl-5-(4-methoxyphenyl)-2-oxabicyclo[4.4.0]dec-3-ene (XIII)*

Compound	mp,† deg C	Configuration	$C_{(1)}$	$C_{(3)}$	$C_{(4)}$	$C_{(5)}$	$C_{(6)}$	$C_{(7)}$	$C_{(8)}$	$C_{(9)}$	$C_{(10)}$	R	R^1
VII	85—86	*trans*	86,41 (48,91)	130,74	119,71	29,39	41,20	28,80	25,87	21,34	33,24	140,18; 125,91; 127,96; 127,28	—
VIII	66—67	*trans, trans*	87,17 (49,10)	129,24	123,21	46,16	48,11	25,78	26,81	21,32	33,36	132,35; 127,18; 113,41; 159,16 (54,91)	144,51; 128,65; 128,24; 126,24
X	165—166	*trans, trans*	87,51 (49,33)	128,01	124,83	45,50	48,35	25,86	26,87	21,39	33,45	136,34; 133,18; 132,87; 127,67; 127,38; 126,01; 125,93; 125,71; 125,71; 124,37	137,12; 129,64; 113,79; 158,16 (55,08)
XI	63—64	*cis*	89,44 (48,77)	129,77	117,77	28,61	39,35	29,91	23,97	25,43	35,30	140,18; 125,91; 128,01; 127,33	—
XIII	135—136	*trans, trans*	99,39 (47,89)	146,67	103,98	40,63	46,74	25,39	26,60	22,49	31,02	136,98; 124,17; 127,92; 127,59	135,44; 129,25; 113,52; 158,05 (54,01)

*Designation of aryl and methoxy groups same as in Table 1.
†Solvent for VII, VIII, XI: ethanol; for X: petroleum ether; for XIII: benzene.

EXPERIMENTAL

^1H and ^{13}C NMR spectra were recorded on a Varian FT-80A Fourier spectrometer at 30°C in $CDCl_3$. ^{13}C NMR spectra were calibrated with the signals of a secondary standard (solvent). In the PMR spectra the internal standard was HMDS. UV spectra were recorded on a SF-4A spectrometer; IR spectra, on a UR-20 spectrometer. The course of the reaction and the purity of the products were monitored by TLC on Silufol UV-254 plates (10:1 cyclohexane: ether).

Stereoisomeric trans- (VII) and cis-1-methoxy-3-phenyl-2-thiabicyclo[4.4.0]dec-3-ene (XI). A suspension of 50 mmole of diketone I [13] in 60 ml of abs. methanol was saturated with hydrogen sulfide at 10-15° for 1.5 h. Then dry hydrogen chloride was passed through the reaction mixture for 10 min, and saturation with hydrogen sulfide was continued another 4 h until the starting ketone had completely disappeared. The reaction products were extracted with petroleum ether, and the extract was washed with water and dried with magnesium sulfate. The solvent was removed in vacuum and from the oily residue there partly crystallized 6.5 g of trans-VII, mp 85-86°C (from ethanol) [1, 3]. UV spectrum in hexane, λ_{max}, nm (log ε): 198 (4.5); 223 (4.1); 271 (3.7). IR spectrum (mineral oil): 1165, 1095, 1080, 1065 cm^{-1} (C–O–C). PMR spectrum: 7.50-7.16 (5H, m, aromatic protons), 5.98 (1H, d.d, vinyl proton), 3.32 (3H, s, OCH_3), 2.15 (2H, m), 2.34 (1H, m, 6-H), 1.44 ppm (8H, alicyclic protons).

The crystallized mixture of isomers VII and XI was chromatographed on an aluminum oxide column (25 cm × 2.5 cm, eluent 300 ml heptane). There were obtained 3 mg of trans-VII (total yield 72%) and 1.3 g (10%) of cis-isomer. Bright orange crystals, mp 63-64° (from ethanol). UV spectrum (in hexane): λ_{max}, nm (log ε): 198 (4.60); 223 (4.20), 271 (3.75). IR spectrum (mineral oil): 1165, 1095, 1080, 1065 cm^{-1} (C–O–C). PMR spectrum: 7.52-7.17 (5 H, m, aromatic protons), 5.94 (1H, d.d, vinyl proton), $^3J_{4,5}$ 2.78 and 4.31 Hz), 3.36 (3H, s, OCH_3), 2.81 (1H, d.q, 6-H, J = 18.54 Hz), 2.11 (2H, m), 1.56 ppm (8H, alicyclic protons). Found: C 73.9; H 7.5; S 12.4%. $C_{16}H_{20}OS$. Calculated C 73.8; H 7.7; S 12.3%.

trans,trans-Thioacetals VIII-X (Table 2) were obtained by the reaction of 1,5-diketones II-IV* with hydrogen sulfide and obtained by the procedure of [3]. After the removal of the crystalline thioacetals from the reaction mixture, only insignificant amounts of the starting 1,5-diketones were found, along with the trans,trans compounds VIII-X.

trans,trans-Acetals XII and XIII were obtained from V and VI under analogous conditions in 78-89% yield; XII, mp 170-171° [3].

Action of Methanol and Hydrogen Chloride on 2-Phenyl-5,6-tetramethylene-4H-thiopyrane (XIV). A suspension of 5 mmole of XIV [5] in 10 ml of dry methanol was saturated for 20 min at 8-10°C with hydrogen chloride and held at that temperature for 24 h. The starting sulfide, 0.4 g (~35%), was filtered off; mp 49-50°C (from ethanol) [5]. Dilution of the mother liquor with ether gave 0.35 g (26%) of 2-phenyl-5,6-tetramethylenethiopyrilium chloride (XVa), mp 101-103°C [6]. The starting thiopyrane XIV and 2-phenyl-1-thiadecalin (XVI) [6] were found in the filtrate by chromatography. The bicyclic thioacetals VII and XI were not detected in the reaction products.

Reactions of trans- (VII) and cis-3-Phenyl-2-thiobicyclo[4.4.0]dec-3-ene (XI) with Perchloric Acid. From 15 mmole of trans-thioacetal VII in 16 ml of glacial acetic acid and 8 ml of 70% perchloric acid there were obtained, as described in [6], 2.85 g (60%) of XVb perchlorate, mp 161-162°C (from acetic acid–ether mixture) and 0.96% (28%) of cis,cis-2-phenyl-1-thiadecalin (XVI), mp 58-59° (from ethanol–acetone mixture) [5, 6].

Similarly, 10 mmole of cis-thioacetal XI yielded XVb perchlorate (56%) and thiadecalin XVI (23%).

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*1,5-Diketones were obtained by the Michael reaction from the respective chalcones and cyclohexanone, as described in [2].

†The ^{13}C NMR spectrum of IX could not be obtained because of its poor solubility in $CDCl_3$.

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SYNTHESIS OF 2-AMINO-1-AZIRINES AND THEIR REACTIONS WITH CARBOXYLIC ACIDS

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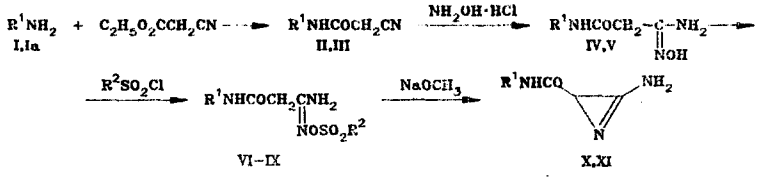
R. S. Él'kinson

UDC 547.717'466.22.07

A method was developed for the synthesis of 2-amino-1-azirines under the conditions of a modified Neber reaction. Their reactivities with respect to mono- and dicarboxylic acids and thiocarboxylic acids were investigated.

In recent years a great deal of attention has been directed to the synthesis and study of the reactivities of functionally substituted 1-azirines [1, 2]. Investigations of addition reactions in the 2-alkylamino-1-azirine series that expand the possibilities of the synthesis of acyclic and heterocyclic nitrogen-containing compounds that are difficult to obtain are of particular interest [3-9]. However, the reactions of 2-amino-1-azirines that contain a primary amino group have not been studied at all.

In order to study the reactivities of 2-amino-1-azirines with a primary amino group we synthesized 2-amino-3-phenylcarbamoyl-1-azirines under the conditions of a modified Neber reaction. As the starting compounds we used phenylcarbamoylacetamidoximes IV and V, which were obtained by the reaction of 2-cyanoacetanilides II and III with hydroxylamine in ethanol.



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