AN UNUSUAL PRODUCT OF THE REACTION OF 1-PHENYL-3-(3,4-DIMETHOXYPHENYL)-3-(2-OXOCYCLOHEXYL)-1-PROPANONE WITH HYDROGEN SULFIDE AND ACIDS: 2α-PHENYL-2,4-ortho-(14,15-DIMETHOXYBENZO)-cis-1-THIADECALIN

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The reaction of 1-phenyl-3-(3,4-dimethoxyphenyl)-3-(2-oxocyclohexyl)-1-propanone with hydrogen sulfide and acids gives an intramolecular rearrangement product, 2α-phenyl-2,4-ortho-(14,15-dimethoxybenzo)-cis-1-thiadecalin in addition to the usual products of disproportionation of intermediate 2-phenyl-4-(3,4-dimethoxyphenyl)-5,6-tetramethylene-4H-thiopyran, namely, 5,6-tetramethylenethiopyrilium salts and 2α-phenyl-4α-(3,4-dimethoxyphenyl)-cis-1-thiadecalin. The configurational and conformational assignments for the sulfides, their sulfoxides, and sulfones were made by ^{13}C NMR spectroscopy.

1-Aryl- and 1,3-diaryl-3-(2-oxocyclohexyl)-1-propanones are converted by the action of hydrogen sulfide in acid media initially to the corresponding 5,6-polymethylene-4H-thiopyrans [1], which then, by the action of a strong mineral or organic acid, undergo disproportionation to give 5,6-polymethylenethiopyrilium salts and 2-thiabicycloalkenes or 2-thiabicycloalkanes [2-4].

We have studied the reaction of 1-phenyl-3-(3,4-dimethoxyphenyl)-3-(2-oxocyclohexyl)-1-propanone (I) at room temperature with hydrogen sulfide and trifluoroacetic acid. In contrast to other "seven-membered" 1,5-diketones, diketone I forms the tetracyclic intramolecular rearrangement product, 2α-phenyl-2,4-ortho-(14,15-dimethoxybenzo)-cis-1-thiadecalin (V) along with the usual reaction products, namely, thiopyrilium trifluoroacetate III and cis-1-thiade-

TABLE 1. Conditions and Products of the Reactions of 1,5-Diketone I with Hydrogen Sulfide and Acids

Reaction conditions			Reaction products (mp, °C)[a]	Yield, g (%)	IV/V or VI/V sulfide ratios in the mixture[b]
starting compound (mmoles)	acid (amount, ml)	solvent (amount, ml)			
I (30)	CF_3COOH (30)	—	III (166—168) IV (142,5—143,5) V (180—181)	7,0 (50) 3,1 (28)	1 : 8
I (40)	70% $HClO_4$ (17,4)	CH_3COOH (75)	V (180—181) VI (145—146) VII (183—184)	5,9 (40) 9,1 (49)	1 : 1
I (30)	BF_3 (30)	CH_3COOH (70)	V (180—181) VI (145—146) VIII (191—193)	4,6 (44) 5,8 (45)	3 : 1
II (12)	CF_3COOH (30)	—	IV (142,5—143,5) V (180—181) (III), VII (183—184)[c]	1,3 (29) 2,8 (52)	5 : 1

[a]Products IV, VI, and VII were identified by mixing melting points with authentic samples [2, 4, 5]. [b]The sulfide ratio in the mixture was determined relative to the ^{13}C NMR spectra of the crude mixtures. [c]Trifluoroacetate III was converted to perchlorate VII.

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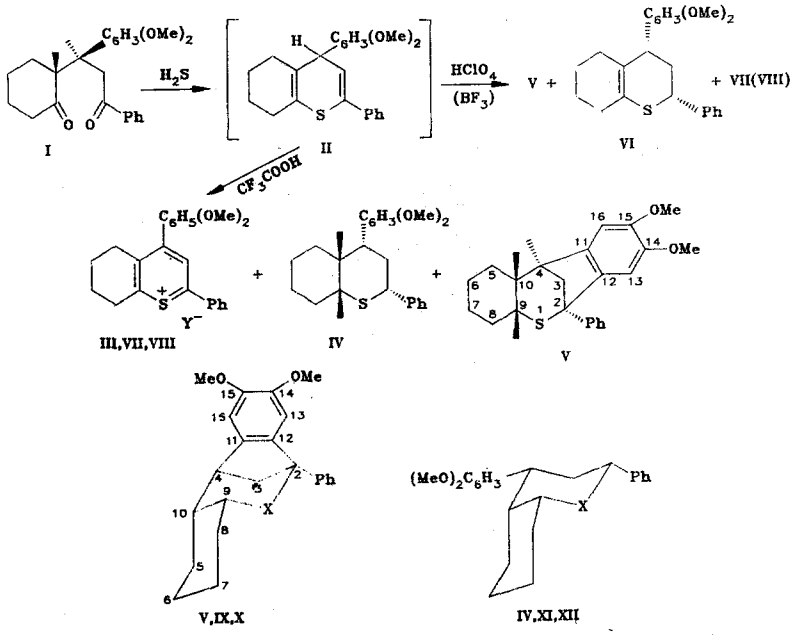
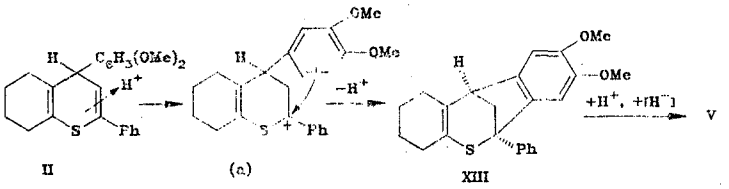
calin IV. The dimethoxyphenyl group in V is bound both to $C_{(2)}$ and $C_{(4)}$ of the heterocycle. This led us also to study the reactions of 1,5-diketone I with hydrogen sulfide and 70% perchloric acid or boron trifluoride etherate. The experimental conditions and reaction products are given in Table 1. In both cases, tetracyclic product V was found in addition to the corresponding perchlorate or tetrafluoroborate VII and VIII and 2-thiabicyclo[4.4.0]-$\Delta^{1,6}$-decene VI. All these compounds are given in Table 2. The configurational and conformational assignments for IV-VI and IX-XII were carried out by ^{13}C NMR spectroscopy (Table 3). Sulfides V and VI were oxidized by hydrogen peroxide to sulfoxides IX and X and sulfones XI and XII, respectively.



III Y = CF_3COO; VII Y = ClO_4; VIII Y = BF_4; IV, V X = S; IX, XI X = SO; X, XII X = SO_2

The formation of trifluoroacetate III and sulfide IV with cis,cis,cis configuration is in accord with the usual concepts concerning the cyclization of 1,5-diketones and 4H-thiopyrans [1] and the mechanism for the disproportionation with acids [3] including the steric specificity of hydride transfer in these systems [5]. Thus, we shall not treat these questions in the present work.

The sterically less hindered double bond ($C_{(2)}$=$C_{(3)}$) is initially protonated in the disproportionation of condensed 4H-thiopyrans with acids [3]. Carbonium ion *a* which is generated in this step may be a hydride ion acceptor. The loss of a hydride ion from a second 5,6-tetramethylene-4H-thiopyran molecule leads to reestablishment of the $C_{(2)}$=$C_{(3)}$ double bond. In our case, this ordinary process is accompanied by the reaction of the electrophilic site in carbonium ion *a* with the dimethoxyphenyl group at $C_{(4)}$. Electrophilic substitution in the aromatic ring apparently leads to intermediate XIII and then to product V due to the reduction of second double bond upon disproportionation.



In our previous work [6], we have established that the heterocycle in 2,4-disubstituted 5,6-polymethylene-4H-thiopyrans is in boat form, while the substituent in the γ-position is pseudoaxial and, thus, close to $C_{(2)}$, which facilitates attack on $C_{(2)}$ upon formation of the carbonium ion. The dimethoxyphenyl group itself is extremely active relative to electrophilic attack, thereby facilitating its reaction with the carbonium site.

TABLE 2. Characteristics of Compounds Obtained

Compound	mp, °C	IR spectrum, cm^{-1}	Found, %			Chemical formula	Calculated, %			Yield, %
			C	H	S		C	H	S	
III	166—168	1680—1660 (COO^-), 1600, 1585, 1525, 1495 (C=C), 1270, 1075 (C—O—C)	63,1	5,2	6,9	$C_{25}H_{23}F_3O_4S$	63,0	4,9	6,7	50—52
V	180—181	1605, 1495 (C=C arom. 1265, 1070 (C—O—C)	75,2	7,4	8,7	$C_{23}H_{26}O_2S$	75,4	7,2	8,8	22
VIII	191—193	1600, 1540, 1495 (C=C), 1280, 1030 (C—O—C), 1040 (BF_4^-)	61,4	5,3	7,3	$C_{23}H_{23}BF_4O_2S$	61,4	5,2	7,1	45
IX	172—173	1600, 1500 (C=C arom. 1265, 1070 (C—O—C), 1045 (S—O)	72,6	7,2	8,6	$C_{23}H_{26}O_3S$	72,2	6,9	8,4	71
X	221—222,5	1600, 1500 (C=C arom. 1300, 1130 (S—O), 1270, 1065 (C—O—C)	69,5	6,9	8,4	$C_{23}H_{26}O_4S$	69,3	6,6	8,1	87
XI	156—158	1600, 1500 (C=C arom. 1250, 1020 (O—O—C), 1045 (S—O)	71,6	8,0	8,5	$C_{23}H_{28}O_3S$	71,8	7,3	8,3	73

*The recrystallization solvents were 1:1 ethanol—acetone for sulfide V and sulfone X and 2:1:0.5 hexane—ether—acetone for sulfoxides IX and XI. Salts III and VIII were reprecipitated from chloroform by the addition of ether.

The structure of tetracyclic sulfide V indicates that the reduction of the angular double bond as a result of protonation at $C_{(10)}$ and transfer of a hydride ion from 5,6-tetramethylene-4H-thiopyran II to the carbonium site at $C_{(9)}$ proceeds by cis addition although the approach of the hydride ion donor from the side of the condensed aromatic ring is sterically hindered.*

The formation of thiabicyclo[4.4.0]-$\Delta^{1,6}$-decene VI in addition to the intramolecular condensation product V in the reactions of 1,5-diketone I with hydrogen sulfide and perchloric acid or boron trifluoride etherate supports our mechanism for the formation of V.

The reaction products did not contain 2α-phenyl-2,4-ortho-(14,15-dimethoxybenzo)-trans-1-thiadecalin or 2β-phenyl-2,4-ortho-(14,15-dimethoxybenzo)-cis-1-thiadecalin, which are isomers of sulfide V.

Table 1 also gives the results of the disproportionation of thiopyran II in trifluoroacetic acid. The relative yield of tetracyclic sulfide V in this case is significantly lower than in the reaction of 1,5-diketone I with H_2S/CF_3CO_2H. The higher yield of sulfide V in this experiment indicates that the dimethoxyphenyl group apparently also reacts with carbonium ions directly preceding the formation of 5,6-tetramethylene-4H-thiopyran II from the bicyclic semithioacetal.

There have been reports of the intramolecular rearrangement of 9-benzyl-symm-octahydrothioxanthenes upon the action of hydrogen chloride, perchloric acid, and trifluoroacetic acid to give 3,4-benzo-5,9-7,8-bis(tetramethylene)-6-thiabicyclo[3.3.1]-7-nonene as a result of intramolecular electrophilic reaction of the benzyl group with the carbocation site with reduction of the double bond and formation of disproportionation products [7].

The formation of thiadecalin V in the reactions considered in the present work is the first example of an intramolecular rearrangement during a disproportionation reaction. We should note the finding that intermediate XIII is reduced in this reaction while the angular double bond is retained in dihydro product VI.

The configurational and conformational assignments for 2α-phenyl-4α-(3,4-dimethoxyphenyl)-cis-1-thiadecalin (IV) were carried out in our previous work [5]. The $^{13}C-\{H\}$ and double heteronuclear resonance spectra permitted examination of the change in the multiplicity of two signals in the rearrangement product V relative to thiadecaline IV. The aliphatic part of the spectrum of V shows a singlet at 57.70 ppm while the aromatic part shows a singlet at 134.64 instead of doublets at 48.70 and 119.33 ppm in IV. These data indicate an intramolecular reaction of a dimethoxyphenyl group with one of the α-carbon atoms of the heterocycle.

*Assuming that the transfer of the hydride ion from the donor molecule to the acceptor molecule occurs in a bimolecular complex.

TABLE 3. ^{13}C NMR Spectra of IV, V, and IX-XII, δ, ppm

Compound	$C_{(2)}$	$C_{(3)}$	$C_{(3)}$	$C_{(5)}$	$C_{(6)}$	$C_{(7)}$	$C_{(8)}$	$C_{(9)}$	$C_{(10)}$	OCH_3	2-Ph				2,4-Ar or 4-Ar					
											$C_{(1)}$	ortho	meta	para	$C_{(11)}$	$C_{(12)}$	$C_{(13)}$	$C_{(14)}$	$C_{(15)}$	$C_{(16)}$
V	57,70 s	39,20	57,50 d (135)*	24,70	28,60	22,20	30,70	46,63, d (138)*	44,54, d (137)*	55,80	140,47	127,71	127,71	126,60	136,64	134,64	104,84	148,32	148,79	107,28
IX	70,43 s	35,46	49,92 d (131)*	23,71	27,41 †	21,24	27,27†	63,83, d (146)*	46,06, d (139)*	55,74	135,86	128,93	128,14	127,69	136,93	127,91	107,20	148,60	149,33	107,60
X	75,78, s	35,08	51,61, d (130)*	23,33	27,23	20,52 †	19,54†	61,08, d (133)*	45,07 d (138)*	55,85, 55,76	129,63	130,22	127,97	128,51	137,94	128,37	107,60	148,82	150,24	107,79
															$C_{(1)}$	$C_{(6)}$	$C_{(5)}$	$C_{(4)}$	$C_{(3)}$	$C_{(2)}$
IV	48,70	33,50	48,20	21,10	26,50	19,20	31,60	46,50	43,10	55,68, 55,59	142,29	127,12	128,23	126,93	136,88	119,33	110,86	148,48	147,14	111,20
XI	68,93	30,98	46,94	22,11	25,93	20,86	25,65	66,22	46,81	55,66, 55,58	136,77	128,62	128,13	128,05	134,25	118,29	110,76	148,45	148,53	110,76
XII	67,39	31,02	46,67	21,12	25,26	19,16	23,12	60,72	44,94	55,78, 55,67	130,28	129,84	128,38	128,79	134,39	119,30	110,95	147,71	148,74	110,95

*The $^1J_{C-H}$ coupling constants are given in parentheses. †Tentative signal assignment.

TABLE 4. Difference in the Chemical Shifts of the γ-Carbon Atoms in Sulfoxides IX and XI and Sulfones X and XII Relative to the Corresponding Sulfides V and IV, ppm

Atom	IV		V	
	SO	SO_2	SO	SO_2
$C_{(3)}$	−2,52	−2,48	−3,74	−4,12
$C_{(8)}$	−5,95	−8,48	−3,43	−11,16
$C_{(10)}$	+3,61	+1,84	+1,52	+0,53

The complete interpretation of the ^{13}C NMR spectrum of sulfide V became possible due to analysis of two series of sulfide—sulfoxide—sulfone spectra for V, IX, X and IV, XI, XII taking account of the characteristic effects of the sulfinyl and sulfonyl groups in going from sulfides to the corresponding sulfoxides and sulfones. Table 4 gives the γ-effects of the SO and SO_2 groups in IX-XII, which are in good accord with the data for thiadecaline derivatives [10]. The use of the SO and SO_2 group γ-effects in IX-XII permitted the identification of the signals for $C_{(3)}$, $C_{(8)}$, and $C_{(10)}$. The $C_{(8)}$ signals in both series IV → XI → XII and V → IX → X are more shielded than the $C_{(3)}$ signals.

The signals for $C_{(8)}$ and $C_{(10)}$ in sulfides IV and V (Table 3) do not differ significantly, while the $C_{(3)}$ signal in V is shifted downfield by 5.7 ppm, perhaps as a consequence of anisotropy of the condensed dimethoxyphenyl group and the presence of this atom in a five-membered ring. Hence, we have assumed that the intramolecular cyclization proceeds at $C_{(2)}$.

The presence of an upfield signal at 22.20 ppm indicates cis ring fusion according to the accepted criterion for condensed cyclohexanes [11].

In addition, analysis of the theoretical chemical shifts of the alicyclic carbon atoms for conformations A and B or 2α-phenyl-cis-1-thiadecalin [5] and the chemical shifts of $C_{(5)}$, $C_{(6)}$, $C_{(7)}$, and $C_{(8)}$ in IV and V indicates that the cis-1-thiadecalin system is in conformation A in both IV and V (Table 5).

The orientation of the sulfinyl groups in IX and XI is equatorial. The ^{13}C NMR spectra of the sulfoxides and sulfones of 2-aryl- and 2,4-diaryl-cis-1-thiadecalins will be considered in a subsequent communication.

EXPERIMENTAL

The IR spectra were taken on a UR-20 spectrometer in vaseline oil and hexachlorobutadiene. The ^1H and ^{13}C NMR spectra were taken on a Varian FT-80A fourier-transform spectrometer using HMDS (for ^1H) and $CDCl_3$ solvent (for ^{13}C) as internal standards. The ^{13}C NMR spectra were taken with broad-field suppression of the spin—spin coupling of the ^{13}C and ^1H nuclei with incomplete proton decoupling. The spectra for V, IX, and X were taken with gated decoupling with retention of the $^1J_{C-H}$ values. The data of various workers [5, 8, 9] were taken to calculate the chemical shifts of some alicyclic and aryl group carbon atoms with subsequent comparison of the experimental and theoretical parameters.

The reaction course and product purity were monitored by thin-layer chromatography on Silufol UV-254 plates with 6:1 hexane—ether as the eluent. Sulfides IV and V or V and VI were separated by preparative column chromatography on alumina with hexane as the eluent.

The characteristics of the compounds synthesized for the first time are given in Tables 2 and 3.

Reaction of 1-Phenyl-3-(3,4-dimethoxyphenyl)-3-(2-oxocyclohexyl)-1-propanone (I) with Hydrogen Sulfide and Trifluoroacetic Acid. A sample of 30 ml absolute trifluoroacetic acid was saturated with hydrogen sulfide for 1 h at 20-25°C. A sample of 10.96 g (30 mmoles) diketone I was added in portions over 1 h and saturation with hydrogen sulfide was continued for an additional 3 h. The reaction mixture was maintained for three days until the intermediate 4H-thiopyran had completely disappeared and then repeatedly extracted with a total of 350 ml hexane. The extract was washed with water and dried over $MgSO_4$. Partial evaporation of the hexane in vacuum gave crystallization of sulfide V, mp 180-181°C (from 1:2 ethanol—acetone) in 22% yield. Complete evaporation of the hexane from the residue gave a 1:1

TABLE 5. Comparison of Theoretical[a] and Experimental Chemical Shifts of Alicyclic Carbon Atoms in IV and V, ppm

Compound	Configuration and confirmation	Nature of the data	$C_{(5)}$	$C_{(6)}$	$C_{(7)}$	$C_{(8)}$
2α-phenyl-cis-1-thiadecalin[a]	cis-A	calc.	24,40	26,69	20,88	31,86
	cis-B	calc.	34,22	19,61	28,28	27,34
IV	cis-A	exp.	21,10	26,50	19,20	31,60
V	cis-A	exp.	24,70	28,60	22,20	30,70

[a]Data for the theoretical spectra of 2α-phenyl-cis-1-thiadecalin from our previous work [5] are given for comparison.

mixture of sulfides IV and V in 6% yield. Trifluoroacetate II crystallized upon dilution of the acid mother liquor of the reaction mixture by ether, mp 166-168°C (from chloroform–ether).

Reaction of 1,5-Diketone I with Hydrogen Sulfide and Boron Trifluoride Etherate. A sample of 70 ml glacial acetic acid was saturated with hydrogen sulfide for 1 h at 20°C and then 30 mmoles 1,5-diketone I was added in small portions along with the dropwise addition of 30 ml boron trifluoride etherate over 1.5 h. The reaction mixture was maintained at room temperature for 72 h and diluted with 350 ml ether. Tetrafluoroborate VIII was filtered off, mp 191-193°C (from chloroform–ether) in 45% yield. The mother liquor was washed with water and dried over $MgSO_4$. Removal of the solvent and chromatography permitted the separation of V and VI in 12 and 33% yield, respectively.

Reaction of 1,5-Diketone I with Hydrogen Sulfide and Perchloric Acid. The reaction was carried out as in our previous work [2]. The experimental conditions and results are given in Table 1. Perchlorate VII and a mixture of sulfides V and VI were obtained.

2α-Phenyl-4α-(3,4-dimethoxyphenyl)-cis-1-thiadecalin 1-Oxide (XI). A sample of 0.6 g (1.63 mmole) sulfide IV was dissolved in 21 ml glacial acetic acid and 1.65 mmole 30% hydrogen peroxide was added dropwise. The mixture was left for 24 h at room temperature and then poured onto chopped ice. Sulfoxide XI was filtered off.

2α-Phenyl-2,4-ortho-(14,15-dimethoxybenzo)-cis-1-thiadecalin 1-oxide (IX) was obtained according to the procedure described above.

2α-Phenyl-4α-(3,4-dimethoxyphenyl)-cis-1-thiadecaline 1,1-dioxide (XII) and 2α-phenyl-2,4-ortho-(14,15-dimethoxybenzo)-cis-1-thiadecalin 1,1-dioxide (X) were obtained from the corresponding sulfides by oxidation with excess hydrogen peroxide as described in our previous work [12]. Sulfone XII was obtained in 90% yield, mp 166-167°C (from ethanol). A mixed probe of this sample with an authentic sample gave an undepressed melting point.

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REACTIONS OF AZIRINES WITH SULFUR NUCLEOPHILES.

4.* TREATMENT OF 2H-AZIRINE WITH MERCAPTOSUBSTITUTED ACIDS. REACTIONS OF AZIRIDINYL ALKYL SULFIDES WITH CARBOXYLIC ACIDS AND ACYL CHLORIDE DERIVATIVES

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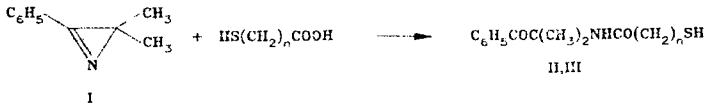
Treatment of 2H-azirines with mercaptosubstituted acids and their derivatives leads to β-ketoamides and 2-aziridinyl alkyl sulfides, respectively. 2-Aziridinyl alkyl sulfides, in turn, react with carboxylic acids to give β-ketoamides and substituted ethanethiol derivatives. Acylation of 2-aziridinyl alkyl sulfides with acyl halides generates a variety of products, depending on the reaction conditions; either products derived from cleavage and isomerization of the aziridinyl ring or (1-acylaziridinyl-2) alkyl sulfides are obtained.

Electrophilic addition of carboxylic acids to the C=N bond of 2H-azirines gives the corresponding β-ketoamides as a result of isomerization and 1,2-cleavage of the aziridine ring in the initially formed 2-acyloxyaziridine derivatives [2]. At the same time, 2,2-dimethyl-3-phenylazirine (I) reacts with β-substituted ethanethiols to give a new type of functional aziridine derivative, namely, aziridinyl alkyl sulfides [3].

It was of interest to us to study the reactions of azirine (I) with mercaptosubstituted acids, i.e., bifunctional reagents which should be capable of entering into both nucleophilic and electrophilic addition reactions to C=N bond of azirine (I). We have found that reaction of azirine I with mercaptoacetic and mercaptopropionic acids occurs at the carboxyl group to generate the corresponding α-(mercaptoacylamino)isobutyrophenones II and III:



I

II n = 1; III n = 2

The formation of products II and III from the reactions of azirine I with mercaptosubstituted acids should be anticipated based on a comparison of the ionization constants of these acids (pK_a 3.68; 10.40 and 4.32; 10.47, respectively) with those of unsubstituted carboxylic acids, which are known to react with 2H-azirines to give the corresponding β-ketoamides.

In cases where protonation of azirine ring and subsequent nucleophilic addition of a carboxylate anion are impossible, such as, for instance, during esterification or salt formation, the mercapto group of the carboxylic acid is the only reactive site, and as a result, nucleophilic addition of the mercapto group to the C=N bond of azirine I occurs, and the corresponding aziridinyl alkyl sulfides are formed, in analogy with the results reported in [3]. For instance, treatment of azirine I with the ethyl ester of mercaptoacetic acid for the sodium salts of N-acetylcysteine or cysteine yields the aziridinyl alkyl sulfides IV-VI as the only reaction products (Table 1):

*For communication 3, see [1].

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