5-Hydroxynaphtho[2,1-d]-1,3-oxathiolone-2 (IX) was obtained according to [10] from 1,4-naphthoquinone and thiourea. Light gray crystals, mp 178-180°C (178-180°C [10]).

9-N-Methyl-N-phenylaminobenzo[*a*]phenothiazin-5-one (X). A mixture of 0.2 g (ca. 1 mmole) of 5-hydroxynaphtho[2,1-d]-1,3-oxathiolone-2 and 0.2 g (ca. 1 mmole) of 4-nitroso-N-methyl-N,N'-diphenylamine in 10 ml of glacial acetic acid was heated to boiling for 10 min, cooled, and the resulting precipitate was removed by filtration. Recrystallization from acetone afforded 0.2 g (60%) of compound X, red-violet crystals, λ_{max} 450 nm (log ε 4.28); mp 110-112°C. Found: N 7.3; S 8.4%. $C_{23}H_{16}N_2OS$. Calculated: N 7.6; S 8.7.

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REACTION OF AZIRINES WITH SULFUR NUCLEOPHILES.

1. TREATMENT OF 2,2-DIMETHYL-3-PHENYLAZIRINE WITH β-SUBSTITUTED ETHANETHIOLS

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The reaction of 2,2-dimethyl-3-phenylazirine with β-substituted ethanethiols results in the formation of a previously unknown series of 2-substituted aziridines, namely aziridinylalkylsulfides. Their three-dimensional structures were investigated by x-ray crystallography.

The high reactivity of the C=N bond in 2H-azirines with nucleophilic reagents opens up a wide range of possibilities for the synthesis of diverse nitrogen-containing acyclic and heterocyclic compounds [1]. Up to this time, however, of the large selection of nucleophilic reagents with lone electron pairs, only N-, O-, and P-containing nucleophiles have been reacted with 2H-azirines. Studies of the addition of sulfur-containing nucleophiles, for instance thiols, to the C=N bond in 2H-azirines have not been reported in the literature.

In order to exploit the reactivity of 2,2-dimethyl-3-phenylazirine (I) with functionally substituted thiols, we have investigated its reaction with β-substituted ethanethiols. These reactions lead to the formation of 2-sulfur-containing aziridines, as well as products resulting from their fragmentation and rearrangement.

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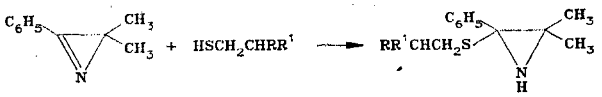
We have established, first of all, that the reaction of azirine I with β-substituted ethanethiols proceeds with the formation of a previously unknown class of 2-substituted aziridines,* namely, the corresponding aziridinylalkylsulfides II-X (Table 1).



I

II-X

II—IX R=H; X R=COONa; II R^1=OH; III $R^1=NH_2\cdot HCl$; IV $R^1=NH_2$; V $R^1=N(CH_3)_2$; VI $R^1=N^+(CH_3)_3$; VII $R^1=NHCH_2COOC_2H_5$; VIII R^1=1-(2-ethoxycarbonyl)-pyrrolidinyl; IX $R^1=NHCH(CH_3)COOC_2H_5$; X $R^1=NHCOCH_3$

The PMR spectra of the sulfides II-X (Table 2) contain signals for the protons of the nonequivalent gem-dimethyl groups at 0.8-1.6 ppm, consistent with cyclic structures for these products. The ^{13}C-NMR spectrum of II shows carbon resonances at 22.5 and 23.3 ppm for the nonequivalent gem-dimethyl groups, as well as singlets due to quaternary carbon atoms at 44.2 and 55.2 ppm. These results confirm the aziridinyl structure for the addition products of thiols and azirine I. The mass spectrum of II displays a peak at 223 for M^+, corresponding to the molecular formula of II. In this way, the combination of physical and spectroscopic analysis of compounds II-X proves that the reaction of azirine I with β-substituted thiols leads to the formation of aziridinylsulfides.

These addition reactions demonstrate that divalent sulfur compounds are powerful nucleophiles; the addition of thiols to the C=N bond of azirine I is much more facile than the analogous reactions of alcohols, which require basic catalysts and elevated temperatures [3, 4]. In this context, amines react very vigorously with azirine I to give ring-opened derivatives which arise via the isomerization and rearrangement of intermediate 2-aminoaziridines [1].

In this regard it was of interest to study the reaction of azirine I with β-aminoethanethiol (cysteineamine), a bifunctional nucleophile possessing the properties of both amines and thiols. The reaction of β-aminoethanethiol with carbonyl compounds, isothiocyanates, and carbamides gives β-aminoethylthio derivatives as a result of nucleophilic attack by the sulfur atom. In contrast, nitriles and isocyanates react simultaneously with both functional groups of the aminothiol [5].

We have established that treatment of the azirine I with β-aminoethanethiol results in the formation of a single product, the aziridinylalkylsulfide IV, whose structure was determined based on the literature data mentioned above. Since the dissociation of the SH group depends upon the pH of the reaction mixture [5], the reaction of azirine I and β-aminoethanethiol was carried out in alcohol in the presence of base, thus ensuring a high concentration of the thiolate anion and facilitating nucleophilic attack on the C=N bond of azirine I and formation of the sulfide IV.

In order to obtain an aziridine containing a thiocholine moiety in the 2 position, we synthesized the iodide and bromide salts of 2-(3,3-dimethyl-2-phenyl-2-aziridinylthio)ethyltrimethylammonium ion (VI). The sulfide VI was prepared in two ways: 1) nucleophilic attack of β-mercaptoethyltrimethylammonium iodide (or bromide) on azirine I, or 2) direct alkylation of the tertiary amino group in the aziridinylalkylsulfide V with methyl iodide. The direction of alkylation of the sulfide V depends on the ratio of reagents, the temperature of the reaction, and the solvent. If the reaction is carried out in ether at −15 to −20°C with an equimolar amount of the two reagents, the sulfide VI is formed in quantitative yield; the PMR spectrum of VI shows unequivocally a signal for the protons in the trimethylammonium group. If the reaction temperature is increased to 20°C, the PMR spectrum of the product mixture shows, in addition to the resonance signals of the sulfide VI, a singlet for the protons of a $S-CH_3$ group at 1.8 ppm, as well as proton signals for a second ethyltrimethylammonium residue. This spectrum is consistent with a 6:1 mixture of the sulfide VI and (methylthio)ethyltrimethylammonium iodide; the latter arises from alkylation of the sulfide VI with methyl iodide and subsequent decomposition of the sulfonium salt. When the reaction was carried out in alcohol

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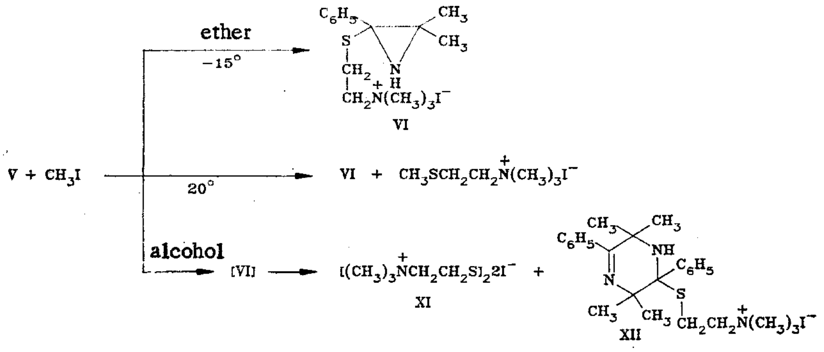
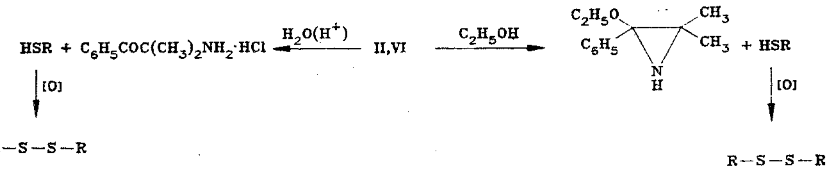
*While this paper was in press, a contribution concerning the synthesis of 1-ethyl- and 1-isopropyl-2-phenylthio-3-phenylaziridines via the complex hydride-promoted reduction of 2-sulfonylaziridines appeared in the literature [2].

as solvent, the alkylation product (sulfide VI) could not be isolated. The product was identified by means of its IR and PMR spectra as a 3:5 mixture of the disulfide XI and a substituted tetrahydropyrazine XII; these products arise via the fragmentation and subsequent reactions of the sulfide VI.



The formation of compound XII is characteristic of addition reactions of azirines, in which the initially formed addition product reacts with a second molecule of azirine to form a corresponding tetrahydropyrazine.

The high reactivity of the aziridinylalkylsulfides synthesized in this study is reflected also in their solvolysis reactions. Acidic hydrolysis of the sulfides II and VI proceeds via cleavage of the $S{-}C_{(2)}$ sulfide linkage to generate the original thiol, its disulfide, as well as α-aminoisobutyrophenone hydrochloride; the latter compound offers additional support for the proposed structures for the sulfides II and VI. Solvolytic cleavage of the $S{-}C_{(2)}$ sulfide linkage in VI in alcohol solution results in the formation of the disulfide XI and a 2-alkoxyaziridine. In contrast, the sulfide II does not undergo solvolysis in ethanol solution unless a basic reagent is present, in which case the solvolytic cleavage of the sulfides II and VI generates the corresponding disulfide as well as 2-ethoxy-3,3-dimethyl-2-phenylaziridine.



In order to determine the three-dimensional structure of the aziridinylalkylsulfide, as well as elucidate the possibility of intramolecular hydrogen bond formation, an x-ray crystallographic analysis of the compound II was performed (see Fig. 1). The molecule II possesses the normal conformation and conventional geometry of an aziridine ring. The dihedral angle between the planes of the phenyl and aziridine rings is 56.1°. The hydrogen atom attached to nitrogen lies in the plane formed by the atoms S, $C_{(2)}$, and N (the torsion angle $\angle S{-}C_{(2)}{-}N{-}H(N) = 4.7°$). It follows, therefore, that the lone electron pair on nitrogen is directed away from the $S{-}C_{(2)}$ bond, i.e., the torsion angle is approximately 125°. The crystal structure of II does not show an intramolecular hydrogen bond between the hydroxyl group and the nitrogen atom of the aziridine ring. The crystal packing of the molecule is stabilized by means of intermolecular hydrogen bonds of the type N–H•••O (bond length 2.97 Å; O•••H = 2.15 Å) and O–H•••N (bond length 2.76 Å; H•••N = 1.86 Å). Other intermolecular contacts in the molecule are characterized by distances which do not exceed the sums of the van der Waals radii of the corresponding atoms.

EXPERIMENTAL

IR spectra were recorded on a Specord spectrophotometer in Nujol. PMR spectra were obtained on a Bruker WH-90 spectrometer on 5% solutions with TMS or HMDS as internal standard. The ^{13}C NMR spectrum of the sulfide II was obtained on the Bruker WH-90 spectrometer at 22.63 MHz. Chemical shifts are reported relative to TMS. The mass spectrum of II was recorded on an MS-905 spectrometer at 70 eV. The x-ray structure analysis of II was carried out on a Syn-

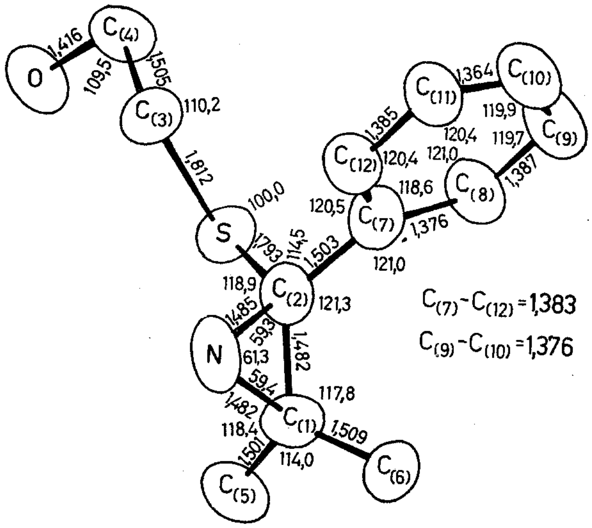


Fig. 1. Three-dimensional structure and geometry of the aziridinyl alkylsulfide II.

TABLE 1. Physicochemical Characteristics of the Aziridinylalkylsulfides II-X

Compound	bp [mp], °C (pressure, hPa)	IR spectrum ν, cm^{-1}	Found, %				Molecular formula	Calc., %				Yield, %
			C	H	N	S		C	H	N	S	
II	[108—110]	3210	64,3	7,5	6,1	14,0	$C_{12}H_{17}NOS$	64,6	7,6	6,3	14,3	94
III	[217—219]	1495, 1575, 1600, 2600, 3230	64,5	7,8	12,4	14,1	$C_{12}H_{18}N_2S \cdot HCl$	64,8	8,1	12,6	14,4	55
IV	[90—91]	3280, 3370	64,6	7,9	12,3	14,3	$C_{12}H_{18}N_2S$	64,8	8,1	12,6	14,4	62
V	97—98 (4)	3220	66,9	8,6	11,1	12,5	$C_{14}H_{22}N_2S$	67,2	8,8	11,2	12,8	82
VI	[84—85]	3200	45,7	6,1	6,8	8,0	$C_{15}H_{25}N_2IS$	45,9	6,4	7,1	8,2	71
VII	83—85 (4)	1740, 3300	62,1	7,6	9,2	10,4	$C_{16}H_{24}N_2O_2S$	62,3	7,9	9,3	10,6	48
VIII	89—91 (4)	1735, 3290	65,3	7,8	7,8	8,9	$C_{19}H_{28}N_2O_2S$	65,5	8,0	8,0	9,2	55
IX	110—111 (4)	1735, 3330	63,1	7,8	8,4	9,7	$C_{17}H_{26}N_2O_2S$	63,3	8,1	8,7	9,9	35
X	[146—148]	1400, 1540, 1610, 1655, 1670, 3300, 3370	54,2	5,4	8,3	9,5	$C_{15}H_{19}N_2O_3NaS$	54,5	5,7	8,5	9,7	78

TABLE 2. PMR Spectral Parameters for the Aziridinylalkylsulfides II-X

Compound[a]	R	R^1	δ, ppm						
			CH_2	CH	R	R^1	NH	CH_3	C_6H_5
II	H	OH	2,54	3,63	3,63	2,22	2,22	0,89; 1,63	7,34
III	H	$NH_2 \cdot HCl$	2,81	3,05	3,05	8,27	1,24	0,76; 1,56	7,38
IV	H	NH_2	2,45	2,71	2,71	1,38	1,38	0,85; 1,65	7,29
V	H	$N(CH_3)_2$	2,33	2,33	2,33	2,06	1,4	0,8; 1,6	7,2; 7,4
VI	H	$N(CH_3)_3$	2,89	3,16	3,16	2,93	—	0,91; 1,62	7,47
VII	H	$NHCH_2COOC_2H_5$	2,49	2,68	2,68	b	2,34÷2,98	0,86; 1,65	7,27; 7,44
VIII	H	1-(2-Ethoxycarbonyl)-pyrrolidinyl	2,44	3,02	3,02	c	1,31	0,8; 1,61	7,2; 7,4
IX	H	$NHCH(CH_3)COOC_2H_5$	2,55	2,8	2,8	d	2,4÷3,1	0,86; 1,65	7,26; 7,43
X	COONa	$NHCOCH_3$	2,89	4,33	—	2,03	2,03	0,87; 1,59	7,34

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[a] The solvent for compounds II, IV, V, VII-IX was $CDCl_3$; for III, DMSO-d_6; for X, D_2O.

[b] δ_{NH} 2.34-2.98; δ_{CH_2} 3.32; δ_{OCH_2} 4.19; δ_{CH_3} 1.26 ppm.

[c] $\delta_{CH, \alpha\text{-}CH_2}$ 2.3-2.9; $\delta_{\beta,\gamma\text{-}CH_2}$ 1.83; δ_{OCH_2} 4.09; δ_{CH_3} 1.2 ppm.

[d] δ_{NH} 2.34-3.10; δ_{CH} 3.37; δ_{CH_3CH} 1.32; δ_{OCH_2} 4.19; δ_{CH_3} 1.3 ppm.

TABLE 3. Atomic Coordinates in a Molecule of Aziridinylalkylsulfide II[a]

Atom	*x*	*y*	*z*
S	0,42095 (6)	0,3876 (1)	0,35526 (3)
O	0,3333 (2)	0,7091 (3)	0,4094 (1)
N	0,3671 (2)	0,0402 (4)	0,3733 (1)
$C_{(4)}$	0,4204 (3)	0,6284 (5)	0,4355 (2)
$C_{(3)}$	0,4137 (3)	0,4376 (5)	0,4271 (1)
$C_{(1)}$	0,4036 (2)	0,0487 (4)	0,3161 (1)
$C_{(2)}$	0,4526 (2)	0,1636 (4)	0,3588 (1)
$C_{(5)}$	0,3278 (3)	0,1163 (6)	0,2741 (2)
$C_{(6)}$	0,4667 (3)	−0,1057 (6)	0,2979 (2)
$C_{(7)}$	0,5599 (2)	0,1248 (4)	0,3830 (1)
$C_{(8)}$	0,5688 (3)	0,0479 (5)	0,4331 (1)
$C_{(9)}$	0,6678 (3)	0,0176 (5)	0,4563 (2)
$C_{(10)}$	0,7587 (3)	0,0621 (6)	0,4284 (2)
$C_{(11)}$	0,7510 (3)	0,1365 (6)	0,3784 (2)
$C_{(12)}$	0,6522 (3)	0,1697 (5)	0,3557 (2)
$H_{(2)}$	0,676 (2)	−0,040 (4)	0,493 (1)
$H_{(4)}$	0,813 (3)	0,168 (5)	0,361 (2)
$H_{(5)}$	0,647 (3)	0,223 (5)	0,320 (2)
$H_{(6)}$	0,344 (3)	0,394 (5)	0,441 (1)
$H_{(7)}$	0,473 (2)	0,383 (4)	0,445 (1)
$H_{(8)}$	0,416 (3)	0,654 (4)	0,475 (2)
$H_{(9)}$	0,490 (3)	0,669 (5)	0,420 (1)
$H_{(1)}$	0,508 (3)	0,017 (4)	0,455 (1)
$H_{(3)}$	0,829 (3)	0,031 (6)	0,443 (2)
$H_{(10)}$	0,514 (3)	−0,147 (4)	0,324 (1)
$H_{(11)}$	0,512 (3)	−0,074 (5)	0,267 (2)
$H_{(12)}$	0,422 (3)	−0,203 (6)	0,288 (2)
$H_{(13)}$	0,282 (3)	0,027 (4)	0,263 (1)
$H_{(14)}$	0,370 (3)	0,152 (5)	0,239 (2)
$H_{(15)}$	0,288 (3)	0,214 (5)	0,285 (1)
$H_{(16)}$	0,309 (3)	0,098 (4)	0,376 (1)
$H_{(17)}$	0,352 (3)	0,821 (5)	0,401 (2)

[a] The values for the anisotropic thermal parameters may be obtained from the authors.

tex $P2_1$ automatic diffractometer. Cell constants for $C_{12}H_{17}NOS$ are: rhombohedral, a = 12.548 (2), b = 7.797 (1), c = 24.606 (5) Å, V = 2407.4 (5) Å, M = 223.36, d_{calc} = 1.23 g/cm^3, μ (CuK$_\alpha$) = 21.1 cm^{-1}, Z = 8, space group P_{bca}. The intensities of 1463 independent reflections were measured by $\theta/2\theta$ scan up to $2\theta_{max}$ = 150° (graphite monochromator, CuK$_\alpha$ irradiation). In the calculations 1271 reflections with $I \geq 2\sigma$ were used. The structure was solved using direct methods. The structure was refined by the method of least squares with anisotropic thermal parameters for the nonhydrogen atoms and isotropic thermal parameters for the hydrogen atoms. The final R factor was 0.039. The standard deviations for the interatomic distances and valence angles did not exceed 0.005 Å and 0.4°, respectively. The atomic coordinates are listed in Table 3.

2-Dimethylaminoethanethiol was synthesized according to [8]. bp 91-93°C. PMR spectrum ($CDCl_3$): 2.56 (4H, m, CH_2), 2.24 [6H, s, $N(CH_3)_2$], 1.69 ppm (1H, s, SH).

N-(2-Mercaptoethyl)proline ethyl ester was synthesized according to [9]. bp 89-91°C (4 torr). PMR spectrum ($CDCl_3$): 4.24 (2H, q, $O-CH_2$), 3.23-2.95 (3H, m, 5-H and 2-H), 2.71 (4H, m, $N-C_2H_4$), 1.95 (5H, m, 3,4-H and SH), 1.33 ppm (3H, t, CH_3).

(3,3-Dimethyl-2-phenylaziridinyl-2)-2-hydroxyethylsulfide (II). To a solution of 4.74 g (0.03 mole) of azirine I [10] in 30 ml of ethyl alcohol was added dropwise 2.34 g (0.03 mole) of 2-mercaptoethanol. The solution was stirred 10 h at room temperature, the ethanol was evaporated, and the residue was recrystallized from ether. Yield 6.3 g (94%).

Compounds III, VI, and X were prepared in an analogous manner and were purified by washing twice with 20 ml of ether.

(3,3-Dimethyl-2-phenylaziridinyl-2)-2-aminoethylsulfide (IV). To a suspension of 3.41 g (0.03 mole) of 2-aminoethanethiol hydrochloride in 20 ml of ethyl alcohol were added dropwise and simultaneously 86 ml of a 4% ethanolic solution of sodium ethoxide and a solution of 4.74 g (0.03 mole) of azirine I in 10 ml of ethyl alcohol. The mixture was stirred 10 h at room temperature and the NaCl precipitate was removed by filtration. The filtrate was concentrated, and the residue was recrystallized from ether. Yield 4.1 g (62%).

(3,3-Dimethyl-2-phenylaziridinyl-2)-2-dimethylaminoethylsulfide (V). A mixture of 4.74 g (0.03 mole) of azirine I and 3.15 g (0.03 mole) of 2-dimethylaminoethanethiol was maintained in an ampule at 70°C for 7 h and then distilled. Yield 6.17 g (82%).

Sulfides VII-IX were prepared in an analogous manner.

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PYRROLES FROM KETOXIMES AND ACETYLENE.

29.* SYNTHESIS OF ALKYLPYRROLES FROM DIALKYLKETOXIMES AND DICHLOROETHANE BY REACTION WITH KOH—DMSO

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UDC 547.742.07

Treatment of dialkylketoximes with dichloroethane and KOH—DMSO results in the formation of alkylpyrroles in 31-61% yields. In addition, the corresponding 1-vinylpyrroles are formed simultaneously in yields up to 18%. The corresponding 1,2-bis(alkylideneiminoxy)ethanes are obtained as side products in the reaction; they result from nucleophilic displacement of chlorine atoms by oximate anions.

We have previously established that 1,2-dihaloethanes may be used in place of acetylene in the synthesis of 2,3-disubstituted pyrroles such as 4,5,6,7-tetrahydroindole and its 1-vinylderivative [2]. Later this reaction variation was expanded to include the oximes of 3-acylindoles [3]. Further publications on the use of dialkylketoximes in this general reaction did not provide complete synthetic details.

In the present paper we report the experimental details for the synthesis of 2,3-dialkyl substituted pyrroles from the reaction of symmetrical and unsymmetrical dialkylketoximes with dichloroethane and KOH—DMSO. (Formula, top, following page.)

The dependence of the yields of pyrroles II and III, as well as of the side products, 1,2-bis(alkylideneiminoxy)ethanes IV, on the reaction conditions and structures of the ketoximes is presented in Table 1. The reaction is sensitive to changes in the reaction temperature, the concentration of KOH and the method of its introduction into the reaction mixture, and also to the presence of water in amounts ranging from 0.5-5%. The structure of the ketoxime also influences the yield of pyrroles. Higher total yields of pyrroles are obtained when the mole ratio of ketoxime:dichloroethane:KOH is on the order of 1:(2-3):7-10, the temp-

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*For Communication No. 28, see [1].

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