Inhibitors for the decomposition of these flavin nucleotides were studied. EDTA, pyrophosphate, and orthophosphate were found to inhibit decomposition of both FMN and FAD in the homogenate. The minimum concentrations of these inhibitors for 50% or 100% inhibition were obtained.

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UDC 547.544 : 544.621

134. Tsutomu Momose, Yo Ueda, Tatsuo Shoji, and Hiroshige Yano: Organic Analysis. XII.[5] Infrared Spectra of Phenylsulfonyl Derivatives. (2). SO_2-Stretching Frequencies of Benzenesulfonamide Derivatives and CO-Stretching Frequencies of N-Acetylsulfonamide Groups.

(*Pharmaceutical Institute, Medical Faculty, University of Kyushu**)

A few infrared spectral studies on benzenesulfonamide derivatives were reported by Adams, *et al.*,[1] Schreiber,[2] Bellamy,[3] and Baxter, *et al.*,[4] but effect of substitution on the SO_2-stretching frequencies is hardly known.

In this work, infrared spectra of 48 benzenesulfonamide derivatives were measured and the effect of a substituent on the SO_2-stretching frequency is discussed. The CO-stretching frequency of the N-acetylsulfonamide group is also discussed.

Results and Discussion

Nature of the Spectra of SO_2-Stretching Vibrations

Since most of benzenesulfonamide derivatives were sparingly soluble in organic solvents except alcohols, a Nujol mull method was used for all samples in the measurement.

All compounds exhibited very strong absorption bands of an asymmetric (ν_{as}) and symmetric (ν_s) stretching mode of SO_2 group. Both absorption bands appeared as one or two bands, but in general, ν_{as} was more complex than ν_s. Their frequencies are tabulated in Table I.

Similarly as in the case of phenyl alkyl sulfone derivatives[5] all maximum bands, listed in bold-face type in the table, are used in this discussion. The ν_{as} and ν_s of benzenesulfonamide derivatives were in the ranges of 1358~1303 cm^{-1} (7.37~7.68 μ) and of 1173~1130 cm^{-1} (8.53~8.85 μ), respectively, although those of phenyl alkyl sulfone derivatives were in the ranges of 1339~1279 cm^{-1} and 1172~1136 cm^{-1}, respectively. Therefore, the SO_2-frequencies, especially ν_{as}, of benzenesulfonamide derivatives existed in a shorter wavelength region than that of phenyl alkyl sulfone derivatives, as shown in Table II. This shift is reverse of that of CO-frequencies between carbonamides and carbonyl compounds.

The NH_2 group has both mesomeric and inductive effect. In carbonamides +M effect is larger than -I effect and the binding of CO group will be weakened by a resonance form

* Katakasu, Fukuoka (百瀬 勉, 上田 陽, 庄司達雄, 矢野弘重).

1) R. Adams, J. J. Tjepkema: J. Am. Chem. Soc., **70**, 4204(1948).
2) Kurt C. Schreiber: Anal. Chem., **21**, 1168(1949).
3) L. J. Bellamy: "The Infrared Spectra of Complex Molecules," Methuen, 300(1954).
4) J. N. Baxter, J. Cymerman-Craig, J. B. Willis: J. Chem. Soc., **1955**, 669.
5) Part XI: This Bulletin, **6**, 415(1958).

of (Ib). For example, benzamide absorbs at a longer wave-length (1664 cm^{-1}) than acetophenone (1689 cm^{-1}).

TABLE I. Infrared Absorption Bands of SO_2 Group[a)]

(A) R_1-C$_6$H$_4$-SO_2NH_2

Compd. No.	R_1	ν_{as}			ν_s		Mean wave number
(I)	H	**1339**	1315[s]		**1156**		1248
(II)	CH_3	**1331**	1306[s]		1172	**1152**	1242
(III)	NH_2	1341	1324[s]	**1317**	**1148**		1233
(IV)	CN	**1345**			**1166**		1256
(V)	COOH	**1346**	1321		**1160**		1253
(VI)	NH_2CH_2	**1329**	1305		**1156**		1243
(VII)	$HCl \cdot NH_2CH_2$	**1351**	1246[s]	1336	**1160**		1256
(VIII)	$HCl \cdot NH_2(CH_3)CH$	**1333**	1320[s]		**1157**		1245
(IX)	$HCl \cdot CH_3NHCH_2$	1340	**1322**		1170	**1160**	1241
(X)	$HCl \cdot NH_2CH_2CH_2$	**1340**	1327[s]		**1161**		1251
(XI)	$HCl \cdot NH_2(C_3H_7)CH$	**1329**	1316[s]		**1168**		1249
(XII)	$CH_3CONHCH_2$	**1335**	1315		**1151**		1243
(XIII)	$CH_3CONH(CH_3)CH$	**1341**			**1155**		1248
(XIV)	$CH_3(CH_3CO)NCH_2$	**1331**			**1147**		1239
(XV)	$CH_3CONHCH_2CH_2$	**1303**			**1157**		1230
(XVI)	$CH_3CONH(CH_3)CHCH_2CH_2$	**1339**			**1166**		1253
(XVII)	$NH_2CONHCH_2$	**1314**			**1166**		1240
(XVIII)	$(CH_3COO)_2CH$	**1351**			**1160**		1256
(XIX)	HON=CH	**1348**			**1167**	1153	1258

(B) R_1-C$_6$H$_4$-$SO_2NR_2R_3$

Compd. No.	R_1	R_2	R_3	ν_{as}			ν_s		Mean wave number
(XX)	CHO	CH_3	H	**1339**			1175	**1163**	1251
(XXI)	HON=CH	〃	〃	**1333**	1309	1293	**1160**		1247
(XXII)	NH_2CH_2	〃	〃	1333	**1323**	1305	**1161**		1242
(XXIII)	$HCl \cdot NH_2CH_2$	〃	〃	**1331**			**1167**		1249
(XXIV)	CN	$COCH_3$	〃	**1351**	1333[s]		**1156**		1254
(XXV)	COOH	〃	〃	**1358**			**1164**		1261
(XXVI)	$HCl \cdot NH_2CH_2$	〃	〃	**1351**	1323		**1167**		1259
(XXVII)	$CH_3CONH(CH_3)CH$	〃	〃	**1346**	1317		**1167**		1257
(XXVIII)	$CH_3(CH_3CO)NCH_2$	〃	〃	**1350**			**1166**		1258
(XXIX)	$CH_3CONHCH_2$	〃	〃	**1345**			**1164**		1255
(XXX)	$(CH_3CO)_2NCH_2$	〃	〃	**1348**			**1152**		1250
(XXXI)	$CH_3CONHCH_2CH_2$	〃	〃	**1347**			**1168**		1258
(XXXII)	$(CH_3CO)_2NCH_2CH_2$	〃	〃	**1349**			1176	**1164**	1257
(XXXIII)	$CH_3CONHCH_2$[b)]	〃	〃	**1350**			1173	**1165**	1258
(XXXIV)	$(CH_3CO)_2NCH_2$[b)]	〃	〃	**1350**			1173	**1164**	1257
(XXXV)	$CH_3CONHCH_2$	〃	CH_3	1368	**1357**		**1166**		1262
(XXXVI)	$(CH_3CO)_2NCH_2$	〃	〃	**1353**	1338		**1163**		1258
(XXXVII)	$(CH_3COO)_2CH$	〃	〃	**1357**	1342[s]		**1173**	1166[s]	1265
(XXXVIII)	NH_2CH_2		H	**1316**			1147	**1130**	1223
(XXXIX)	$HCl \cdot NH_2CH_2$	〃	〃	1337	**1328**		**1148**	1139	1238

s: shoulder

a) Bold-face type indicates the maximum band

b) In this compound, R_1 is in the position *ortho* to the sulfonamide group

(C) R_1-C$_6$H$_4$-SO_2NH-C$_6$H$_4$-R_4

Compd. No.	R_1	R_4	ν_{as}		ν_s		Mean wave number
(XL)	$HCl \cdot NH_2CH_2$	H	1333^s	**1327**	**1163**	1155^s	1245
(XLI)	NH_2CH_2	CH_3	**1332**		**1153**		1243
(XLII)	$HCl \cdot NH_2CH_2$	〃	**1335**		**1151**		1243
(XLIII)	$HCl \cdot NH_2CH_2$	OC_2H_5	1335	**1329**	**1157**		1243
(XLIV)	NH_2CH_2	OH	**1333**		**1150**		1242
(XLV)	$HCl \cdot NH_2CH_2$	〃	**1316**		**1157**		1237
(XLVI)	NH_2CH_2	COOH	**1325**		**1156**		1241
(XLVII)	$HCl \cdot NH_2CH_2$	〃	**1347**		**1155**		1251
(XLVIII)	$HCl \cdot NH_2CH_2$	NO_2	**1352**		**1163**		1258

On the contrary, in sulfonamides, +M effect on SO_2 group may be smaller than -I effect, and accordingly a contribution of resonance form of (IIb) may increase the force constant of SO_2 group to absorb in a shorter wave-length region.

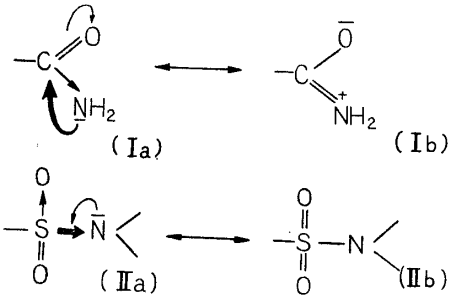


TABLE II. Frequency Shift between Benzenesulfonamide and Phenyl Alkyl Sulfone

R_1-C$_6$H$_4$-SO_2R_2

R_1 \ R_2	NH_2			CH_3			Difference of mean wave number
	ν_{as}	ν_s	Mean wave number	ν_{as}	ν_s	Mean wave number	
H	1339	1156	1248	1285	1143	1214	−34
CH_3	1331	1152	1242	1304	1149	1227	−15
NH_2	1317	1148	1233	1282	1140	1211	−22
CN	1345	1166	1256	1319	1156	1238	−18
$HCl \cdot NH_2CH_2$	1351	1160	1256	1314	1155	1235	−21
$HCl \cdot NH_2(CH_3)CH$	1333	1157	1245	1309	1151	1230	−15
$CH_3CONHCH_2$	1335	1151	1243	1297	1152	1225	−18
$(CH_3COO)_2CH$	1351	1160	1256	1309	1160	1235	−21
HON=CH	1348	1167	1258	1307	1153	1230	−28

TABLE III. Shift of Mean Wave Number by the Substituent R_1

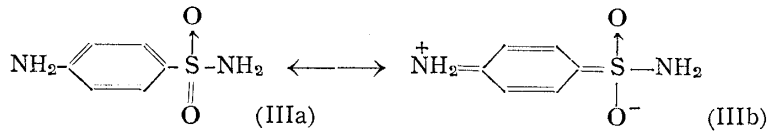
R_1-C$_6$H$_4$-SO_2NH_2

R_1	Mean wave number	Shift	Hammett's σ	δ_p
CN	1256	+ 8	+0.628	+0.30
COOH	1253	+ 5	+0.265	+0.17
H	1248	0	0	0
NH_2CH_2	1243	− 5		−0.03
CH_3	1242	− 6	−0.170	−0.10
NH_2	1233	−15	−0.660	−0.40

Substitution Effect on SO_2 Frequencies

All samples were measured as Nujol mull and a mean frequency $(\nu_{as}+\nu_s)/2$ of the SO_2 group was used in the estimation of substitution effect, which was successfully used in a previous work of this series.

At first, the effects of some substituents (R_1) of R_1—C$_6$H$_4$—SO_2NH_2 were compared with each other. Table III shows that, when benzenesulfonamide is taken as a standard, *p*-NH_2 substituent causes the greatest shift of both frequencies to a longer wave-length region, and *p*-CN causes a shift to a shorter wave-length region. The magnitude of these shifts is approximately linear to Hammett's σ[6] or to the chemical shift parameters of benzene derivatives.[7] It may also be supposed in benzenesulfonamide derivatives that if the electron density of S atom diminishes, a double-bond character of S-O bond will increase, and the force constant of the bond will become larger, absorbing at a shorter wave-length region, and *vice versa*.



The shift of *p*-aminobenzenesulfonamide may be explained by the contribution of its resonance form of (IIIb).

The shift of *p*-cyanobenzenesulfonamide may be explained by the -I effect of *p*-CN group, which will give a stronger double-bond character to the S-O bond, and may cause it to absorb in a shorter wave-length region.

The effect of *p*-CH_3, *p*-CH_2NH_2, and *p*-COOH group is between those of *p*-NH_2 and *p*-CN groups. Substituents other than those discussed have their own effect on the SO_2 bond, and though they cannot be discussed in detail, it may be concluded in general that each substituent has similar effect on the SO_2-frequency in both phenyl alkyl sulfone and benzenesulfonamide derivatives.

Next, the effect of some substituents attached to the N atom of $-SO_2NH_2$ group was examined. The results are listed in Table IV. It is clear from the Table that electron-accepting group ($COCH_3$) gives a stronger double-bond character to the S-O bond and causes it to absorb in a rather shorter wave-length region. Inversely, electron-donating group (CH_3) makes the S-O bond to have a more single-bond character and to absorb in a longer wave-length region. Above explanation will be confirmed by the effect of six kinds of substituents (R_4) of HCl·NH_2CH_2—C$_6$H$_4$—SO_2NH—C$_6$H$_4$—R_4 as shown in Table V. Electron-donating group (OH) and electron-accepting group (NO_2) show the largest shifts for a longer and for a shorter wave-length region, respectively, when R_4=H is taken as a standard. The order of the shift agrees with Hammett's σ or with the chemical shift parameters of benzene derivatives, and an approximate linear correlation can be seen between them.

CO-Stretching Frequencies of $-SO_2NHCOCH_3$ Group

The amide-I band is known to shift toward a shorter wave-length region in N-arylamide, N-chloroamide,[8] and -CONHCO- compounds.[9] This shift is caused by the electron affinity of aryl, halogen, and CO groups, which will withdraw the N-electron from conjugation with the carbonyl group.

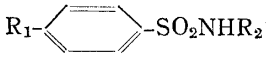
All N-acetylbenzenesulfonamide derivatives measured in solid state had very strong absorption bands in a range of 1720~1691 cm^{-1} (5.81~5.91 μ) (Table VI). These bands must

6) H. H. Jaffé: Chem. Revs., **53**, 191(1953).
7) B. P. Dailey, *et al.*: J. Am. Chem. Soc., **78**, 3043(1956).
8) R. N. Jones, C. Sandorfy: "Chemical Applications of Spectroscopy," Interscience, 525(1956).
9) L. J. Bellamy: "The Infrared Spectra of Complex Molecules," Methuen, 190(1954).

correspond to the amide-I band, and the shift of N-acetylbenzenesulfonamide derivatives, which may be caused by the electronegativity of SO_2 group, reaches an amount of about 40 cm^{-1}.

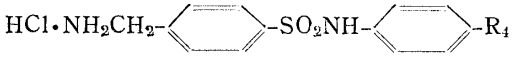
Ishidate and one of the present authors[10] suggested the possibility of a presence of two kinds of diacetates in *p*-aminomethyl- and *p*-aminoethyl-benzenesulfonamide, but, as shown in Table VI, the compounds (XXX), (XXXII), (XXXIV), and (XXXVI) proved to be triacetates which have one weak ν_{NH} band, corresponding to the NH of $-SO_2NH-COCH_3$ group, three amide-I bands, and no amide-II band. The diacetates (XXIX), (XXXI), (XXXIII), and (XXXV) have a strong ν_{NH} and a weak ν_{NH} band in 3-μ region, a very strong amide-II band, and two amide-I bands.

TABLE IV. Shift of Mean Wave Number by Substituent R_2



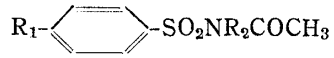
R_1	R_2	Mean wave number	R_2	Mean wave number	Shift
HON=CH	H	1258	CH_3	1247	−11
NH_2CH_2	〃	1243	〃	1242	− 1
$HCl \cdot NH_2CH_2$	〃	1256	〃	1249	− 7
$HCl \cdot NH_2CH_2$	〃	1256	C_6H_5	1245	−11
CN	〃	1256	$COCH_3$	1254	− 2
COOH	〃	1253	〃	1261	+ 8
$HCl \cdot NH_2CH_2$	〃	1256	〃	1259	+ 3
$CH_3CONH(CH_3)CH$	〃	1248	〃	1257	+ 9
$CH_3(CH_3CO)NCH_2$	〃	1239	〃	1258	+19
$CH_3CONHCH_2$	〃	1243	〃	1255	+12
$CH_3CONHCH_2CH_2$	〃	1230	〃	1258	+28

TABLE V. Shift of Mean Wave Number by the Substituent R_4



R_4	Mean wave number	Shift	Hammett's σ	δ_p
NO_2	1258	+13	+0.778	+0.42
COOH	1251	+ 6	+0.265	+0.17
H	1245	0	0	0
CH_3	1243	− 2	−0.170	−0.10
OC_2H_5	1243	− 2	−0.250	
OH	1237	− 8	−0.357	−0.37

TABLE VI. Amide-I and -II, and ν_{NH} band of



Compd. No.	R_1	m.p. (°C)	R_2	ν_{NH}		Amide-I band			Amide-II band
(XXIV)	CN		H				1712		
(XXV)	COOH		〃				1686[b]		
(XXVI)	$HCl \cdot NH_2CH_2$		〃				1691		
(XXVII)	$CH_3CONH(CH_3)CH$		〃				1709	1647	
(XXVIII)	$CH_3(CH_3CO)NCH_2$		〃				1709	1610	
(XXIX)	$CH_3CONHCH_2$	214	〃	3413	3106		1703	1641	1550
(XXX)	$(CH_3CO)_2NCH_2$	196	〃		3197	1725	1704	1674	
(XXXI)	$CH_3CONHCH_2CH_2$	192	〃	3430	3132		1703	1646	1558
(XXXII)	$(CH_3CO)_2NCH_2CH_2$	145～146	〃		3135	1727[s]	1718	1658	
(XXXIII)	$CH_3CONHCH_2$[a]	216～218	〃	3376	3067		1697	1639	1559
(XXXIV)	$(CH_3CO)_2NCH_2$[a]	146～148	〃		3150	1733[s]	1720	1657	

10) M. Ishidate, T. Momose: Yakugaku Zasshi, **67**, 214(1947).

(XXXV)	$CH_3CONHCH_2$	110～111	CH_3	3344	3115	1692	1631	1556
(XXXVI)	$(CH_3CO)_2NCH_2$	83～85	〃			1706[b]	1687	
(XXXVII)	$(CH_3COO)_2CH$		〃			1692[b]		

a) *ortho* compound b) overlapped bands s: shoulder

The estimation of acetyl groups by the Kögl's semi-micro method agrees with these results and therefore the former theory should be abandoned.

The authors are indebted to Mr. H. Matsui for the measurement of infrared spectra and wish to express their gratitude to Miss S. Tada and Mr. M. Shirōzu for the microanalyses.

Experimental

Infrared spectra were measured with a Koken Model DS-201 recording infrared spectrophotometer using NaCl prism.

4-(3-Acetamido-3-methylpropyl) benzenesulfonamide (XVI)—(3-Amino-3-methylpropyl)benzene was acetylated with Ac_2O. Distillation under reduced pressure gave pale yellow oily substance of $b.p_5$ 168～170°. Sulfonation of this acetate with chlorosulfonic acid and amination of the oily sulfonyl chloride with 28% NH_4OH gave crystals which were recrystallized from dil. EtOH to colorless plates, m.p. 187～188°. *Anal.* Calcd. for $C_{12}H_{18}O_3N_2S$: N, 10.74. Found: N, 11.24.

N-Acetyl-4-cyanobenzenesulfonamide (XXIV)—(IV) was acetylated with Ac_2O and the product was recrystallized from water to colorless needles, m.p. 207～209°. *Anal.* Calcd. for $C_9H_8O_3N_2S$: N, 12.50. Found: N, 12.35.

4-(N-Methylacetamidomethyl) benzenesulfonamide (XIV)—Sulfonation of N-methyl-N-benzylacetamide with chlorosulfonic acid and amination of the oily sulfonyl chloride with 28% NH_4OH gave crystals, which were recrystallized from AcOEt and dried over P_2O_5. Colorless plates, m.p. 162～163°. *Anal.* Calcd. for $C_{10}H_{14}O_3N_2S$: N, 11.56. Found: N, 11.89.

N-Acetyl-4-(N-methylacetamidomethyl) benzenesulfonamide (XXVIII)—(XIV) was acetylated with Ac_2O and was recrystallized from water. Colorless plates, m.p. 233°. *Anal.* Calcd. for $C_{10}H_{16}O_4N_2S$: N, 9.86. Found: N, 9.91.

2-Aminomethylbenzenesulfonamide Di- and Tri-acetates (XXXIII and XXXIV)—2.5 g. of 2-aminomethylbenzenesulfonamide was refluxed for 2 hrs. with 10 cc. of Ac_2O and 2.5 g. of AcONa. After cool, the reaction mixture was poured into water and extracted successively with ether and AcOEt. Ethereal solution gave crystals on evaporation which were recrystallized from EtOH to colorless prisms, m.p. 146～148°. *Anal.* Calcd. for $C_{13}H_{16}O_5N_2S$: N, 8.97. Found: N, 8.53.

AcOEt solution gave crystals on evaporation which were recrystallized from EtOH to colorless prisms, m.p. 216～218°. *Anal.* Calcd. for $C_{11}H_{14}O_4N_2S$: N, 10.37. Found: N, 9.87.

Estimation of Acetyl Group—The Kögl's semimicro method was used and the results are shown in Table VII.

TABLE VII.

Sample	(XXIX)	(XXX)	(XXXI)	(XXXII)
m.p. (°C)	214	196	192	145～146
Molecular weight	270.31	312.35	284.33	326.37
Weight taken (mg.)	37.3	43.4	31.1	38.8
0.05*N* NaOH consumed (cc.)	5.54	8.31	4.06	6.80
No. of acetyl group	2.008	2.99	1.85	2.87

Summary

Infrared spectra of 48 benzenesulfonamide derivatives were measured and substituent effect on the SO_2-stretching frequencies was discussed, comparing with those of phenyl sulfone derivatives. The SO_2 frequencies, especially ν_{as}, of benzenesulfonamide derivatives proved to be in a shorter wave-length region than that of phenyl sulfone derivatives. Electron-donating or -accepting groups attached to the phenyl ring or directly to the sulfonamide group shifted the SO_2 frequencies to a longer or a shorter wave-length region, respectively.

The CO streching frequency of N-acetylsulfonamide group showed a large shift to a shorter wave-length region.

Synthesis of some benzenesulfonamide derivatives was also described.

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UDC 547.853'789

135. **Shoji Inoue**: Studies on Pyrimidine Derivatives. VII.[1] Synthesis of Thiazolo[5,4-*d*]pyrimidines and Related Compounds. (7)

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In Part III[2] of this series, it was shown that 2-methyl-5-chlorothiazolo[5,4-*d*]pyrimidine was obtained from 2-chloro-4-mercapto-5-aminopyrimidine (I) by the action of acetic anhydride. Since then two kinds of thiazolo[5,4-*d*]pyrimidine derivatives have been prepared from (I). 5-Chlorothiazolo[5,4-*d*]pyrimidine (II) was obtained in a good yield by the treatment of (I) with ethyl orthoformate and 2-hydroxy-5-chlorothiazolo[5,4-*d*]pyrimidine (VI) was prepared by heating (I) with phosgene in dioxane.

The chlorine in (II) is reactive and by the action of sodium ethoxide, sodium ethanethioxide or sodium phenoxide, (II) was converted into 5-ethoxythiazolo[5,4-*d*]pyrimidine (III), 5-ethylthio-thiazolo[5,4-*d*]pyrimidine (IV), and 5-phenoxythiazolo[5,4-*d*]pyrimidine (V), respectively. In these operations, however, a small amount of alkali-soluble by-product was formed in each case. The products thus obtained were assumed to be formed by the cleavage of the C-S bond in (II).

The reactivity of the chlorine in (VI) was decreased owing to the strong influence of the 2-hydroxyl substituent and the condensation product (VII) was obtained from (VI) by refluxing with sodium ethanethioxide for 16 hours under conditions similar to the formation of (IV) from (II). Compound (VII) was also produced by the treatment of 2-hydroxy-5-mercapto-thiazolo[5,4-*d*]pyrimidine (IX) with ethyl bromide and (IX) was prepared from 2,4-dimercapto-5-aminopyrimidine (VIII)[3] and phosgene.

It has already been shown in part IV[3] of this series that 2,5-dimercaptothiazolo[5,4-*d*]-pyrimidine (X) may be prepared from (VIII) and potassium methylxanthate, and that (X) could be converted into the corresponding diethylthio derivative in the usual manner.

On the other hand, when only one mole of ethyl bromide was allowed to react with the dimercapto compound (X), a smooth reaction occurred and the monoethylthio compound, 2-mercapto-5-ethylthio-thiazolo[5,4-*d*]pyrimidine (XI) was obtained. In order to determine the position of substitution in (XI), the remaining group in (XI) was oxidized to the corresponding hydroxyl group by the addition of hydrogen peroxide to the sodium salt of (XI), and the resulting product was found to be identical with 2-hydroxy-5-ethylthio-thiazolo[5,4-*d*]pyrimidine (VII) obtained by the above-mentioned process.

Similarly, the reaction of 2,7-dimercaptothiazolo[5,4-*d*]pyrimidine (XIII),[1] prepared from (XII) by the action of one mole of ethyl bromide, afforded the 7-substituted monoethylthio compound (XIV), and this was oxidized to 2-hydroxy-7-ethylthio-thiazolo[5,4-*d*]pyrimidine (XV) by a method identical to the reaction of (XI) with hydrogen peroxide.

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1) Part VI: This Bulletin, **6**, 352(1958).

2) Part III: *Ibid.*, **6**, 343(1958).

3) Part IV: *Ibid.*, **6**, 346(1958).