tem. The relative amounts of various metabolites present was determined by scanning in a gas-flow counter.

Cleavage of the glucuronide was carried out in the following fashion. One milliliter of urine was diluted to 4 ml. with *p*H 6.8 0.075 *M* phosphate buffer, and 40 units of Sigma β-glucuronidase was added. Incubation at 38° for 10 hr. gave nearly complete reaction.

Hydroxy Ethynylcyclohexyl Carbamate from Urine. (1) **Rats.**—A 24 hr. urine collection was made from a colony of rats which had received 800 mg. of "cold" ethinamate at an intravenous dose level of 50 mg./kg. To this urine was added that of two additional rats which had received labeled ethinamate. The crude metabolite was removed from the urine by extraction with methylene chloride at *p*H 10. Partial purification resulted from partition chromatography on silica gel employing a *n*-butanol–water system. Further purification was effected by alumina (Woelm) chromatography using benzene–methylene chloride mixtures for elution. The radioactive metabolite so isolated was an oil which could not be crystallized. The infrared spectra were obtained in acetonitrile and in chloroform solution.

Anal. Calcd. for $C_9H_{13}O_3N$: N, 7.65. Found: N, 5.55.

(2) **Humans.**—A total of 25 liters of urine was collected from humans who had received a combined total oral dose of 44 g. of "cold" ethinamate 8 hr. before collection. The extraction and chromatographic procedures were the same as those employed for the rat material. Those fractions eluted from alumina by benzene–methylene chloride mixtures which were shown by infrared analysis to contain both the acetylene group and the carbamate grouping were combined and evaporated to dryness. Trituration of the resulting oil gave a semi-crystalline mass which was filtered and washed with methylene chloride. This procedure yielded 625 mg. of semi-solid material, m.p. 128–132°. An infrared spectrum in chloroform solution was identical to that of the rat metabolite in the same solvent. Two recrystallizations from acetone–petroleum ether gave fine needles, m.p. 135–136°. A 2.9% solution in methanol in a one decimeter tube gave an observed optical rotation of +0.015°. The molecular weight calculated from X-ray crystallographic data was 183 (theory 183). The pure material was insoluble in chloroform but an infrared spectrum in acetonitrile was identical to that of the rate metabolite in the same solvent.

Anal. Calcd. for $C_9H_{13}O_3N$: C, 59.00; H, 7.15; N, 7.65. Found: C, 58.65; H, 7.11; N, 7.22.

Hydroxyethylcyclohexyl Carbamate (IV).—Fifty milligrams (0.27 mmole) of hydroxyethinamate in 12 ml. of ethanol was reduced with hydrogen in 20 mg. of pre-reduced palladium on calcium carbonate catalyst. Uptake ceased in 13 minutes after 0.55 mmole of hydrogen had been consumed. The product was recovered by filtration, evaporation to dryness and two recrystallizations from benzene. The yield was 20 mg. (39%) of crystalline product, m.p. 128–129°. The infrared spectrum in acetonitrile was consistent with the structure.

Anal. Calcd. for $C_9H_{17}O_3N$: N, 7.48. Found: N, 7.29.

Acetoxyethinamate (V).—Fifty milligrams of hydroxyethinamate was dissolved in 5 ml. of pyridine and 1 ml. of acetic anhydride added. The solution was stored at room temperature for 24 hr. Ether was added and the resulting ether solution washed with water. The product was recovered by evaporation to dryness. Recrystallization from benzene gave acetoxyethinamate, m.p. 157–158°. The infrared spectrum ($CHCl_3$) was consistent with this structure.

Anal. Calcd. for $C_{11}H_{15}O_4N$: C, 58.65; H, 6.71; N, 6.22. Found: C, 58.76; H, 6.86; N, 5.94.

Silver Complex Formation.—The substance to be tested (1 mg.) was dissolved in 1 ml. of 30% aqueous ethanol and a few drops of Tollens reagent added. Ethynylcyclohexanol gave a heavy white precipitate. Ethinamate gave a brown solution and a heavy precipitate. Hydroxyethinamate gave brown solution and no precipitate. Acetoxyethinamate gave a brown solution and a light precipitate.

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INDIANAPOLIS 6, INDIANA

[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY, DUKE UNIVERSITY]

The Structure and Antimicrobial Activity of Some Isothiocyanate Oxides and Sulfides[1]

BY C. K. BRADSHER, F. C. BROWN, E. F. SINCLAIR AND S. T. WEBSTER

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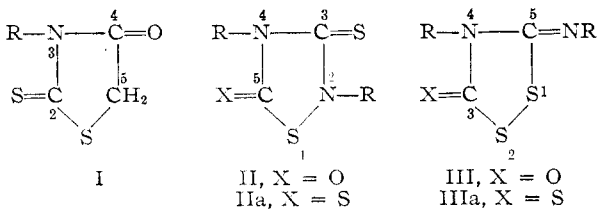
Infrared absorption spectra of the isothiocyanate sulfides lend support to the 4-alkyl(aryl)-5-alkyl(aryl)-imino-1,2,4-dithiazolidine-3-thione structure (IIIa). On the basis of similar evidence, the simple alkyl isothiocyanate oxide bases are most probably 2,4-dialkyl-1,2,4-thiadiazolidine-3-thione-5-ones. The aryl and aralkyl isothiocyanate oxide bases, as well as all of the isothiocyanate oxide salts examined, appear to have the 1,2,4-dithiazolidine structure (III). Tests for antimicrobial activity have been carried out on several isothiocyanate sulfide and oxide derivatives.

It has been shown that 3-substituted rhodanine derivatives (I) possess pronounced antimicrobial activity.[2–4] More recently[5] a systematic study has indicated the advantage of having both the thione group at position 2 and the carbonyl at position 4. The present phase of our work involves



I II, X = O IIa, X = S III, X = O IIIa, X = S

the study of the effect of variations in the nature of the atoms or groups present at positions 1 and 5 of the rhodanine ring. Freund and co-workers[6–8] had

(1) This work was supported by a research grant (E-695(c)) from the National Microbiological Institute of the National Institutes of Health, Public Health Service. Taken in part from the theses submitted by E. Faye Sinclair and Sidney T. Webster in partial fulfillment of the requirements for the degree of Doctor of Philosophy at Duke University, 1956 and 1957, respectively.

(2) G. J. M. van der Kerk, H. C. van Os, G. de Vries and A. K. Sijpesteijn, *Mededel. Landbouwhogeschool en Opzoekingsstas. Staat Gent*, **18**, 402 (1953); *C. A.*, **48**, 316 (1954).

(3) F. C. Brown, C. K. Bradsher, S. M. Bond and R. J. Grantham, *Ind. Eng. Chem.*, **46**, 1508 (1954).

(4) F. C. Brown, C. K. Bradsher, E. C. Morgan, M. Tetenbaum and P. Wilder, Jr., THIS JOURNAL, **78**, 384 (1956).

(5) C. K. Bradsher, F. C. Brown and E. F. Sinclair, *ibid.*, **78**, 6189 (1956).

(6) M. Freund, *Ann.*, **285**, 154 (1895).

(7) M. Freund and E. Asbrand, *ibid.*, **285**, 166 (1895).

(8) M. Freund and G. Bachrach, *ibid.*, **285**, 184 (1895).

prepared three isothiocyanate oxides and three isothiocyanate sulfides which seemed to fall within the scope of our study. These compounds had the empirical formulas $(RNCS)_2O$ and $(RNCS)_2S$, where R = methyl, ethyl and phenyl, and were formulated[6-8] as 2,4-disubstituted-1,2,4-thiadiazolidine-3-thione-5-ones II and 4-alkyl(aryl)-5-alkyl(aryl)-imino-1,2,4-dithiazolidine-3-thiones IIIa, respectively. Freund assigned unrelated structures to the isothiocyanate oxides and sulfides because the latter were also prepared by the reaction of bromine on alkylamine alkyldithiocarbamates whereas alkylthiocarbamates and bromine gave no isothiocyanate oxides. On the basis of further experiments, Hantzsch and Wolvekamp[9] concluded that the dithiazolidine formulas (III and IIIa) represent the correct structure for both the oxides and sulfides. More recently in a survey of the literature in this field, Bambas[10] has suggested that the thiadiazolidine formulations (II and IIa) are correct for both the oxides and sulfides.

In a recent paper[11] dealing with 3-imino-5-alkylmercapto-1,2,4-dithiazole salts as antifungal agents, Allen, Shelton and Van Campen employed as a comparison standard a highly fungistatic compound referred to as "4-methyl-5-methylimino-1,2,4-dithiazolidine-3-one,"[12] which from the reference cited appears to be the methyl isothiocyanate oxide of Freund and Asbrand.[7] Our interest in the isothiocyanate oxides and sulfides was aroused by the uncertainty which seemed to surround the structure of these compounds as well as the possibility that the correct structures might be related to that of the antimicrobial 3-substituted rhodamines I.

A comparison of the alternate structures II and III shows that a decision between them might be made on the basis of the presence or absence of the imino group in the molecule. Since the iminio group is known to cause strong absorption in the 6.02–6.21 μ[13] region of the infrared spectrum, a convenient means is available for establishing the correct structure for both the isothiocyanate sulfides and oxides. As may be seen from Table I all of the isothiocyanate sulfides appear to have the imino group present and therefore must have the 4-alkyl(aryl)-5-alkyl(aryl)-imino-1,2,4-dithiazolidine-3-thione structure (IIIa) endorsed by both Freund[6-8] and Hantzsch.[9]

The isothiocyanate oxides seem to be divided into two well-defined groups, the simple alkyl derivatives which give only very feeble absorptions in 6.02–6.21 μ region, and the aralkyl and aryl isothiocyanate oxides which absorb significantly in the 6.12–6.14 μ range, and usually with greater intensity than in the carbonyl region. On the basis of these observations, the Hantzsch and Wolvekamp[1] formula for the methyl and ethyl isothiocyanate oxides is clearly wrong, and the correct structure in both cases is that of a 2,4-dialkyl-1,2,4-thiadiazolidine-3-thione-5-one (II). The same observations make it appear probable that the aryl and aralkylisothiocyanate oxides can be represented best as 4-aryl(aralkyl)-5-aryl(aralkyl)-imino-1,2,4-dithiazolidine-3-ones III.

A number of isothiocyanate oxides have been prepared as the hydrobromide salts. Determination of the infrared spectra of five of these salts revealed that all show characteristic absorptions in the imino region and can best be represented as 4-alkyl-5-alkylimino-1,2,4-dithiazolidine-3-one salts (III·HBr). This means that the free base V has a different skeletal structure from that of its hydrobromide IV. It is easy to understand that where easy interconversion of this type is possible, the dithiazolidine form III having the basic alkylimino

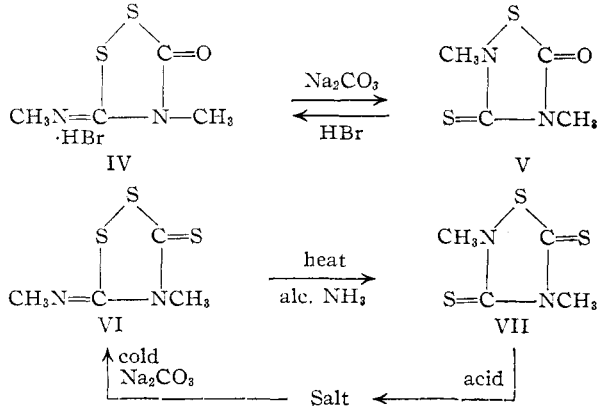


TABLE I

INFRARED ABSORPTION OF ISOTHIOCYANATE SULFIDES AND OXIDES IN THE CARBONYL AND IMINO REGIONS

Isothiocyanate sulfides	M.p. sample, °C.	Absorption peaks,[a] μ, in regions Carbonyl	Imino
CH_3[b]	83.5–84[c]	...	6.09S
C_2H_5	29–30[d]	...	6.11S
C_6H_5	157.5–158.5[e]	...	6.13S
Oxides			
CH_3	104.5–105.5[f]	5.84S	....
C_2H_5[g]	41–42[h]	5.87S	....
$C_6H_5(CH_2)_2$	98–100	5.84S	6.12M
$C_6H_5CH_2$	90–91	5.87S	6.13S
C_6H_5[g]	116–118[i]	5.85S	6.14S
$4ClC_6H_4$	133–135	5.83S	6.12S
$4BrC_6H_4$	143–144	5.83S	6.14S
Hydrobromide salts of oxides			
CH_3[i]	227–229	5.85S	6.16S
C_2H_5[g]	201–203	5.77S	6.13S
C_4H_9[g]	159–160	5.78S	6.23S
$CH_3O(CH_2)_2$[g]	142–144.5	5.78S	6.23S
$C_6H_5(CH_2)_2$[g]	168.5–170.5	5.91S	6.13S

[a] Only the peak of maximum intensity in the region is given, the intensity being indicated by the abbreviations S (strong), M (medium), and W (weak). [b] Except as noted all spectra were determined in carbon tetrachloride solution. [c] Lit.[8] m.p. 86°. [d] Lit.[8] m.p. 29.5°. [e] Lit.[8] m.p. 154–156°. [f] Lit.[7] m.p. 108°. [g] Spectrum determined in chloroform solution. [h] Lit.[8] m.p. 45°; 42°, E. Sell, *Ber.*, **6**, 322 (1873). Lit.[8] m.p. 117–118°. [i] Spectrum determined by the potassium bromide plate method.

(9) A. Hantzsch and M. Wolvekamp, *Ann.*, **331**, 278 (1904).

(10) L. L. Bambas, "Five-membered Heterocyclic Compounds with Nitrogen and Sulfur or Nitrogen, Sulfur and Oxygen," Interscience Publishers, Inc., New York, N. Y., 1952, pp. 35–80.

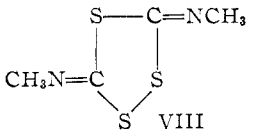
(11) R. E. Allen, R. S. Shelton and M. G. Van Campen, THIS JOURNAL, **76**, 1158 (1954).

(12) It appears implicit from reference 11 that the discovery of the antifungal activity of this compound is due to Gregory and Johnson, W. A. Gregory, Ph.D. thesis, Cornell University, 1947.

(13) F. A. Miller in Gilman's "Organic Chemistry—An Advanced preatise," Vol. III, John Wiley and Sons, Inc., New York, N. Y., 1953, T. 145.

group present would be the one most stable in the presence of acid.

A close analogy is to be found in the behavior of methyl isothiocyanate sulfide, 4-methyl-5-methylimino-1,2,4-dithiazolidine-3-thione (VI), which unlike its homologs and congeners undergoes rearrangement when heated in alcohol containing a few drops of ammonia, and yields a base to which Hantzsch and Wolvekamp[9] have assigned the structure VII. The infrared absorption spectrum of the rearrangement product shows no imino group present, and obviously eliminates the alternate 1,2,4-



VIII

trithiolane structure VIII proposed by Freund.[6] It is interesting that when the rearrangement product VII is converted to the salt and then treated with cold sodium carbonate solution, it gives back the unrearranged base VI.

It appears that a major difference between the isothiocyanate oxides and sulfides lies in the stability of the dithiazolidine forms III and IIIa. While the salts in both series must exist in this form, the simple oxide salts when treated with even cold sodium carbonate solution yield a base which rearranges rapidly to the thiadiazolidine form II. In the isothiocyanate sulfide series the dithiazolidine form IIIa appears quite stable, and deliberate efforts to bring about rearrangement have succeeded only with the first member of the series (IIIa, X = CH_3). The failure of even the ethyl homolog (IIIa, X = C_2H_5) to undergo this reaction suggests that the larger groups offer some type of steric impediment to the rearrangement. It may well be that it is the size of the substituent groups which confers stability on the dithiazolidine form (III) of the aryl and aralkyl isothiocyanate oxides.

A total of nine isothiocyanate oxide salts have been synthesized and tested for antimicrobial activity (Table II). Seven of these formed an homologous series (III·HBr, R = $CH_3 \rightarrow C_7H_{15}$).

TABLE II

ANTIMICROBIAL ACTIVITY OF THE HYDROBROMIDE SALTS OF SOME ISOTHIOCYANATE OXIDES (III·HBr)

R	*A. niger* % inhibition at 250 p.p.m.	Lowest concn. giving 100% inhibition, p.p.m. *A. niger*	*B. subtilis*	*E. coli*
CH_3	100	25[a]	25	50
C_2H_5	100	50[b]	25	100
C_3H_7	100	25[c]	25	250
C_4H_9	100	25[d]	25	>250
C_5H_{11}	54	>250	< 25	>250
C_6H_{13}	49	>250	>250	>250
C_7H_{15}	16	>250	>250	>250
$CH_3O(CH_2)_2$	45	>250	100	>250
$C_6H_5(CH_2)_2$	68	>250	250	>250

[a] Inhibition at 10 p.p.m., −10%. [b] Inhibition at 25 p.p.m., 27%. [c] Inhibition at 10 p.p.m., 46%. [d] Inhibition at 10 p.p.m., 59%.

It may be seen that increasing the number of carbon atoms in the chain stepwise up to R = heptyl causes a sudden decrease in activity toward *Aspergillus niger* as one goes from butyl to amyl. No effect on the activity toward *B. subtilis* was observed with alkyl groups up to amyl, but there was a rapid decrease in activity toward *E. coli*. Also there was a sudden decrease in activity for *B. subtilis* when going from amyl to hexyl. It is of interest that the 2-methoxyethyl group is less effective than the butyl group in promoting activity toward *B. subtilis*.

In Table III are recorded the results of the antimicrobial activity tests on some free bases, both isothiocyanate oxides and sulfides. The most potent compound in this group is methyl isothiocyanate oxide which has been reported[14] to possess high activity toward several microörganisms. One of our continuing interests[3,5,15,16] has been the comparison of oxygen and sulfur as promoters of antimicrobial activity. From Table III a comparison of the activity of the structurally analogous rearranged methyl isothiocyanate sulfide (IIa, R = CH_3) and the methyl isothiocyanate oxide (II, R = CH_3) as well as the phenyl isothiocyanate sulfide (IIIa, R = C_6H_5) with the comparable oxide (III, R = C_6H_5) reveals that, in this system, replacement of oxygen by sulfur results in a decrease in antimicrobial activity.

TABLE III

ANTIMICROBIAL ACTIVITY OF ISOTHIOCYANATE SULFIDES AND OXIDES

Sulfides R	Probable formula	*A. niger* % inhibition at 250 p.p.m.	Lowest concn. giving 100% inhibition *A. niger*	*B. subtilis*	*E. coli*
CH_3	IIIa	100	100[a]	250	250
CH_3 (rearranged)	IIa	100	100[b]	>250	>250
C_2H_5	IIIa	100	100[c]	100	>250
C_6H_5	IIIa	−26	>250	>250	>250
Oxides					
CH_3	II	100	25[d]	25	100
$C_6H_5CH_2$	III	4	>250	>250	>250
C_6H_5	III	45	>250	100	>250
4-ClC_6H_4	III	31	>250	25	>250
4-BrC_6H_4	III	33	>250	50	>250

[a] Inhibition at 50 p.p.m., 26%. [b] Inhibition at 50 p.p.m., 23%. [c] Inhibition at 50 p.p.m., 8%. [d] Inhibition at 10 p.p.m., 10%.

Experimental

Isothiocyanates.—The alkyl and aralkyl isothiocyanates were prepared by the action of ethyl chlorocarbonate on the appropriate N-alkyl dithiocarbamate, essentially as described earlier.[17] The only new compound was 2-methoxyethyl isothiocyanate, which was prepared in 65% yield, b.p. 88.5–92° (23.5 mm.), $n^{24.5}D$ 1.5010.

Anal. Calcd. for C_4H_7NOS: C, 41.00; H, 6.02; N, 11.96. Found[18]: C, 41.26; H, 6.08; N, 11.92.

The *p*-halophenyl isothiocyanates were prepared by known methods.[19]

(14) Allen, Shelton and Van Campen (ref. 11) offer data which would seem to indicate that toward *C. albicans* and *M. canis* the methyl isothiocyanate oxide is at least *comparable* to gliotoxin in activity.

(15) C. K. Bradsher, F. C. Brown and R. J. Grantham, THIS JOURNAL, **76**, 114 (1954).

(16) F. C. Brown, C. K. Bradsher and S. W. Chilton, *J. Org. Chem.*, **21**, 1269 (1956).

(17) M. L. Moore and F. S. Crossley, "Organic Syntheses," Coll. Vol. III, John Wiley and Sons, Inc., New York, N. Y., 1955, p. 599.

(18) All analyses were done by Galbraith Laboratories.

(19) F. B. Dains, R. Q. Brewster and C. P. Olander, "Organic Syntheses," Coll. Vol. I, John Wiley and Sons, Inc., New York, N. Y., 1941, p. 447. We are indebted to Mr. P. E. Shaw for carrying out these preparations for us.

TABLE IV

SYNTHESIS OF ISOTHIOCYANATE OXIDE DERIVATIVES

R	M.p., °C.	Yield, %	Formula	Analyses, % Calcd. C	Calcd. H	Calcd. N	Found C	Found H	Found N
			Salts						
C_3H_7[a]	183–184	45	$C_8H_{15}Br_2N_2OS_2$	32.10	5.05	9.36	32.08	5.15	9.19
C_4H_9	159–160	50	$C_{10}H_{19}BrN_2OS_2$	36.69	5.85	8.56	36.70	6.23	8.64
C_5H_{11}	164.5–166.5	44	$C_{12}H_{23}BrN_2OS_2$	40.56	6.52	7.88	40.36	6.40	7.53
C_6H_{13}	160.5–161	39	$C_{14}H_{27}BrN_2OS_2$	43.85	7.10	7.31	44.04	7.12	6.99
C_7H_{15}	159.5–161	49	$C_{16}H_{31}BrN_2OS_2$	46.67	7.59	6.81	46.90	7.46	6.44
$CH_3O(CH_2)_2$	143–144.5	38	$C_8H_{15}BrN_2O_3S_2$	29.00	4.56	8.46	28.90	4.70	8.79
$C_6H_5(CH_2)_2$	168.5–170.5	58	$C_{18}H_{19}BrN_2OS_2 \cdot H_2O$	48.98	4.80	6.34	49.32	5.12	6.23
			Bases						
$C_6H_5CH_2$[b,c]	90–91	38	$C_{16}H_{14}N_2OS_2$	61.12	4.49		60.86	4.49	
4-BrC_6H_4[a,b,c]	141–143	87	$C_{14}H_8Br_2N_2OS_2$	37.85	1.82	6.31	37.52	1.99	6.34
4-ClC_6H_4[a,b,c]	134–135.5	93	$C_{14}H_8Cl_2N_2OS_2 \cdot {}^1/_2C_2H_6O$	47.62	2.93	7.41	47.67	2.89	7.67
$C_6H_5(CH_2)_2$[b,c]	98–100	..[d]	$C_{18}H_{18}N_2OS_2$	63.13	5.30	8.18	62.63	5.41	8.05

[a] The bromine addition product crystallized directly from the reaction mixture. [b] Free base. [c] Crystallized from alcohol as the free base. [d] A small amount of this compound separated from the mother liquor obtained upon recrystallization of the hydrobromide from alcohol.

4-Alkyl(aryl)-5-alkyl(aryl)imino-1,2,4-dithiazolidine-3-one hydrobromides (III·HBr) were prepared essentially as described by Freund, *et al.*[7,8] In a 200-ml. round-bottomed flask equipped with stirrer, condenser and dropping funnel, a solution containing 0.085 mole of the isothiocyanate in 25 ml. of chloroform and 5 ml. of 95% ethanol was stirred while a solution containing 10 ml. of bromine in 10 ml. of chloroform was added over a period of about 30 minutes. Upon addition of the bromine the solution refluxed spontaneously, with a considerable quantity of hydrogen bromide being evolved near the end of the reaction. Stirring was continued for ten minutes longer, and then the reaction mixture was worked up.

Depending upon whether the bromine addition product separated readily or not, one of several techniques was used in isolating the product as the hydrobromide. (a) In the preparations where the addition compound crystallized either directly or after concentration and cooling of the reaction mixture, the product was collected and refluxed on the steam-bath with 35–50 ml. of ethanol until a yellow-gold solution was obtained (evolution of hydrogen bromide), which upon cooling deposited crystals of the hydrobromide. Shaking the alcoholic solution with ether was often an effective means of purifying and precipitating the hydrobromide. (b) When the addition compound did not separate, the reaction mixture was refluxed with 50 ml. of ethanol until the evolution of hydrogen bromide ceased, and then concentrated *in vacuo* on the steam-bath to effect removal of chloroform. Additional ethanol was put in and, upon cooling the alcoholic solution, the hydrobromide separated. The hydrobromide was recrystallized from ethanol.[20] In some cases the reaction mixture was concentrated initially to a red oil by distillation of most of the chloroform and bromine before refluxing the addition product with alcohol. Details concerning yields and properties may be found in Table IV.

4-Alkyl(aryl)-5-alkyl(aryl)imino-1,2,4-dithiazolidine-3-thiones (IIIa) were prepared as described by Freund, *et al.*[7,8]; the phenyl compound was prepared by the method of Friedmann and Gattermann.[21] The samples used for spectroscopic measurements[22] had the following melting points: methyl, m.p. 83.5–84° (lit.[7] 86°); ethyl, m.p. 29–30° (lit.[8] 29.5°); phenyl, m.p. 157.5–158.5° (lit. 154–156°[8] and 154°[21]). The rearranged methyl compound 2,4-dimethyl-1,2,4-thiadiazolidine-3,5-dithione (IIa, R = CH_3), melted at 123–124° (lit.[7] 120°) and showed no absorption in the 6.02–6.21 μ region.

Absorption Spectra.—The infrared absorption spectra were determined in carbon tetrachloride or chloroform solution using matched 1-mm. sodium chloride cells. A Perkin-Elmer double-beam spectrophotometer was used for the measurements.

Antimicrobial Testing.—The testing methods were the same as those described earlier.[4,23]

DURHAM, NORTH CAROLINA

(20) Recrystallization of the hydrobromide salts of the aryl- and aralkylisothiocyanate oxides from alcohol tended to convert them to the free bases.

(21) A. Friedmann and L. Gattermann, *Ber.*, **25**, 3526 (1892).

(22) These spectra were obtained in carbon tetrachloride.

(23) We are indebted to Mrs. Marilena Ferguson and to Mrs. Dorcus Clarke for carrying out these tests.