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Structural and Biological Characterization of Antimony(V) Polyamines

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ABSTRACT

The condensation of organoantimony(V) dihalides with diamines forming antimony(V) polyamines employing the interfacial technique is described. Structural characterization was accomplished by employing elemental analysis, light-scattering photometry, infrared spectroscopy, control reactions, and mass spectroscopy. The products exhibit mild inhibition to a wide range of bacteria and to HeLa, BHK-21, and L929 cancer-related cell lines.

INTRODUCTION

The synthesis of polymers containing metal atoms in the backbone of the polymer chain is well known. For instance, polyethers, polyesters, polyamidoximes, and polyoximes have been synthesized based on the Lewis acid-base reaction concept [1-3]. Utilizing an interfacial condensation polymerization technique, Carraher and Blaxall synthesized a number of antimony containing polyesters of form 1 [4]. These products were of high molecular weight (typically > 10⁵).

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where R = methyl, ethyl, butyl, phenyl R' = aliphatic and aromatic hydrocarbons X = chloride and bromide

Doak and Freedman [5] synthesized monomeric compounds from the condensation of mono-Lewis bases (in particular monoamines) with a number of organometallic halides (including antimony, 2) [5]

 $R_3 SbX_2 + R'NH_2 - R_3 Sb(NH-R')_2$

2

where R = methyl, ethyl, phenyl

R' = aliphatic and aromatic hydrocarbons

X = chloride and bromide

This was extended by Carraher and Moon to include reaction with polyamines derived from arsenic(V) organometallics and diamines (Form 1 where the metal is arsenic [6]). These products exhibited biological activities against selected bacteria and were soluble in several liquids.

The condensation of organoantimony(V) dihalides with diamines is an extension of the work cited above. Here we describe the initial synthesis of antimony(V) polyamines of Form 3, presenting structural evidence and biological assays related to their potential use as additives in medical and coatings applications.



EXPERIMENTAL

Chemicals

The following chemicals were used without further purification (from Aldrich Chemical Co., Milwaukee, unless noted otherwise):

2,6-Diamino-5-nitropyrimidine; 2,6-diaminoanthraquinone; 2,5dichloro-p-phenylenediamine; 2,5-diaminotoluene sulfate; 2,6-diaminopyridine dihydrochloride; 4,4'-diaminodiphenyl amine sulfate; 1,6diaminohexane; 2,4-diamino-5(3,4-dimethoxybenzil)pyrimidine (Burroughs Wellcome Co., Research Triangle Part, North Carolina); 1,12diaminododecane; 2,6-diamino-8-purinol hemisulfate monohydrate; adenine (Chemalog Chemical Dynamics Corp., South Plainfield, New Jersey); 1,9-diaminononane; 4,6-diamino-2-methyl-5-nitrosopyrimidine (Parish Chemical Co., Provo, Utah); 2,4-diamino-6-hydroxypyrimidine; 4,4'-diaminodiphenyl sulfon (Fluka AG, Buchs SG, Switzerland); 2-methoxy-p-phenylenediamine; 4,4'-methylenedianiline; 4,4'diaminobenzanilide; diaminomaleonitril; Zineb (Pfaltz and Bauer Inc., Stamford, Connecticut); 2,4-diamino-7-hydroxypyrimidine; 4,4'-methylbis(3-nitroaniline); 3.6-diaminoacridine hemisulfate; 1.4-diaminopiperizine; 1,8-diaminooctane; 1,4-diaminobutanone dihydrochloride; 1,3-diamino-2-hydroxypropane; β -(4-aminophenyl)ethyl amine; uracil; histamine dihydrochloride (United States Biochemical Corp., Cleveland, Ohio); 4,4'-diaminooctafluorodiphenyl; triphenylantimony dichloride; trimethylantimony dichloride (Strem Chemicals, Inc., Newburyport, Massachusetts); and tributylantimony.

Polymerization Apparatus and Procedure

For all the polymerization reactions conducted, a Kimex emulsifying jar was used. The jar was placed on a Waring Blendor (Model 1120) whose speed, without load, is 18,000 rpm.

The lid of the jar has one hole in it for a large mouthed funnel through which a large volume of solution can be added in a short period of time so that the rate of addition of a solution to the jar can be held approximately constant.

Solutions of the antimony organometallic in an organic solvent were added to the blender. Rapid stirring was begun and an aqueous solution of a diamine which also contained an added base, sodium hydroxide, NaOH, or triethylamine, Et_3N , or other agent was added. Reaction time was begun when the organic phase was fully added to the stirred aqueous phase.

The products were recovered as precipitates by employing suction filtration and washed with water and the organic liquid employed in the nonaqueous phase.

Instrumentation for Physical and Chemical Characterization

A Perkin-Elmer Model 457 Grating Spectrophotometer was used to obtain infrared spectra of the products. All spectra were taken using potassium bromide pellets. The mass spectral analyses were performed using a DuPont 21-401 MS coupled with a Hewlett-Packard HP-2216C. The elemental analyses were performed using a Perkin-Elmer 240 Elemental Analyzer.

Molecular weight determinations were done using a Brice-Phoenex Model BP-3000 Universal Light-Scattering Photometer. Molecular weight determinations were carried out in the usual manner using 1% polymer solutions with serial dilutions. Refractive index increments, dn/dc, were determined using a Bausch and Lomb Abbe Refractometer Model #3-L.

Preliminary Biological Characterization

Several organisms were chosen to give a wide range of bacterial species. The procedure involved streaking tryptic soy agar (TSA) plates with the organism and then adding a small amount of polymer to a circular area. The plates were then incubated for 24 h at 37° C. After this time period, the plates were checked for inhibition which is indicated by a clear zone on the plate.

Three cell lines were chosen for the cell line studies. Dimethylsulfoxide (DMSO) was the solvent of choice for the polymer since it is not appreciably toxic to man and the polyamines are soluble in it.

The polymer was dissolved in DMSO and the resulting solution filtered using a sintered glass filter. Various concentrations of the polymer were prepared by diluting the stock solution.

Confluent flasks of the cell lines L929 (Mouse Connective Tissue Tumor Line), BHK-21 (Baby Hamster Kidney), and HeLa (Epitheloid Carcinoma, Cervix, Human) were trypsinized, suspended, counted (using a Coulter Counter Model Z_{BI}), and plated onto 24-well Corning

plates. The plates were incubated overnight at 37° C in 5% CO₂ to allow the cells to form a monolayer. Next, Dulbecco's Modified Eagle Medium (DMEM) was aspirated and replaced with 1 mL of the various polymer dilutions (in DMEM). This was then allowed to incubate overnight at 37° C in 5% CO₂. The polymer-containing media was then aspirated and replaced with 1 mL of a solution of DMEM and Neutral Red in 0.85% NaCl (concentration 7.6×10^{-6} g/mL Neutral Red/DMEM). The cells were then allowed to take up the dye (incubate) for 2 h (same conditions). The media was then aspirated and the cells washed twice with 1 mL aliquots of 1:1 Sorensen Citrate Buffer (pH 4.20)/EtOh to develop the full acid color of the dye (about 95% extraction efficiency observed).

The effect of the polymer on the cells was measured from the absorbance of the dye since only live (viable) cells will absorb the dye. This was achieved by measuring the absorbance of the extracted dye at a maximum of 540 nm. From the absorbance of the control and polymer concentrations, the percent live cells was calculated as

absorbance of cell solutions treated with polymer

 $- \times 100 = \%$ viable cells

absorbance of cell solutions without treatment

RESULTS AND DISCUSSION

Historical

As cited in the Introduction section, the condensation of polyoximes with organoantimony dihalides, monoamines with organoantimony dihalides, and diamines with organoarsenic dihalides to form the Lewis acid-base adduct is well established. The condensation of organoantimony dihalides with diamines is a logical extension of these reactions.

Control Reactions

Reactions were attempted excluding either triphenylantimony dichloride, diamine, or sodium hydroxide, with all other reaction conditions held constant. The reactions resulted in clear solutions as compared to the precipitates formed when both monomers and added base are present. This is consistent with the product being derived from more than one of the added ingredients, excluding situations such as single reactant precipitation.

Infrared Spectroscopy

Infrared spectral analysis is consistent with a product of Form 3 with the formation of the Sb-N linkage. This linkage is indicated by the presence of a band at about $1100-1200 \text{ cm}^{-1}$ (Table 1).

Bands characteristic of the organoantimony moiety are present.

Sb-N band (cm ⁻¹
1180
1100
1125
1175
1175
1150
1180 1100 1125 1175 1175 1175

TABLE 1. Location of the Antimony-Nitrogen Stretching Band in the Infrared Spectra of Select Condensation Products Derived from the Condensation of Organoantimony(V) Dihalides with Diamines



FIG. 1. The infrared spectrum of the condensation product of triphenylantimony dichloride with 2,6-diamino-5-nitropyrimidine (---) and triphenylantimony dichloride (--).

For instance, for the product from triphenylantimony dichloride and 2.6-diamino-5-nitropyrimidine (Fig. 1), bands characteristic of the antimony-phenyl linkage are present at about 1425-1450 cm⁻¹; bands characteristic of phynel are present at 690 and 725 cm⁻¹. Bands characteristic of the diamine moiety are also present. For instance, infrared spectra of the product of triphenylantimony dichloride with 2,6-diamino-5-nitropyrimidine exhibit a band characteristic of the nitro group stretch at 1600 cm^{-1} and a band characteristic of the carbon-nitro bond at 800 cm^{-1} . The presence of 2,6-diamino-8purinol moiety is evident from the presence of a band at 3100-3500 cm⁻¹ characteristic of oxygen-hydrogen stretching. The presence of the R-O-CH₃ band for the product derived from 2,4-diamino-5-(3,4-dimethoxybenzil)pyrimidine at about 1440-1470 cm⁻¹ indicates the presence of the diamine moiety. The product derived from the condensation of triphenylantimony dichloride with 2,5-dichloro-pphenylenediamine shows a band associated with Cl-CH stretching for an aromatic substituent at 1000 cm^{-1} .

Elemental Analysis

Elemental analyses for carbon, hydrogen, and antimony are in fair agreement with a product of Form 3 (Table 2). Values for nitrogen are in moderate agreement at best. It should be noted that many organometallic condensation polymers and analogous coordination monomeric and polymeric products have difficulty undergoing combustion, leading to poor results for elemental analysis. A combustion catalyst, tungstanyl carbonyl, was utilized in the present study to assist product combustion.

Mass Spectrophotometry

The formal description for the procedure employed is pyroprobe chemical mass spectrophotometry.

Mass spectral data are also in agreement with a product containing units derived from the organoantimony and diamine with fragmentation patters assignable as having originated from both monomeric portions. Further, ion fragments assignable to repeat units are detected (Table 3). For the product from the condensation of triphenylantimony dichloride with 2,6-diamino-5-nitropyrimidine, all of the phenyl unit appear at 77, 78, 79, and 156 biphenyl; all ion fragments are assigned values based on the mass to single charge (m/e ratio). Fragments 199, 200, 275, 277, 354, and 355 are associated with the antimony-phenyl moiety. Fragments characteristic of the presence of the nitropyrimidine appear at 105, 107, 123, 151, 154, and 155. Fragments assignable to the presence of one repeat unit minus selected moieties are found at 291, 293, 295, 332, 353, 355, 367. Thus the pyroprobe-mass spectral results are consistent with a product of Form 3. Results for the additional

TABLE 2. Carbon, Nitrogen, Hydrogen, and Antimony Elemental Analysis for Select Organoantimony(V) Dolyamine Products

Polyamine Frouces								
	ပ %		N %		н %		% Sb	
Diamine ^a	Calculated	Found	Calculated	Found	Calculated	Found	Calculated	Found
4,4' -Diaminodiphenyl- sulfon	53.7	53.2			4.2	4.5		
2,6-Diaminoanthra- quinone	65.0	66.1			4.6	3.0		
4,4' - Methylenedi - aniline	67.8	70.1			4.9	5.1		
Zineb			4.5	6.1	3.0	3.0	20.3	27.6
1,12-Diaminododecane	65.6	60.8					23.2	24.3
Adenine	56.6	60.6			4.1	4.2	24.3	24.6
2,5-Dichloro-p- phenylene diamine			5.3	5.9	3.6	3.3		
c								

^aAll derived from triphenylantimony dichloride.

Ion fragment
φ
φφ
Sbø
Sb¢2
Sb¢₃
N
NNN
NO ₂
N
N
N NO ₂
î O J
N ⁻ N ⁻
Repeat unit minus 2¢
Repeat unit minus ϕ , NO ₂
Repeat unit minus 2¢, NO ₂
Repeat unit minus ϕ , NO ₂ , NH

TABLE 3. Major Ion Fragments for the Gases Derived from the Thermal Degradation of the Condensation Product of Triphenylantimony Dichloride with 2,6-Diamino-5-nitropyrimidine^a



^bCIMS (m/e = 60 to 400).

TABLE 4. Major Ion Fragments for the Gases Derived from the Thermal Degradation of the Condensation Product of Triphenylantimony Dichloride with 2,4-Diamino-5(3,4-dimenthoxybenzil)pyrimidine^a

m/e ^b	Ion fragment
79, 78, 77	φ
156	φφ
200, 199, 198	Sbø
275, 277	Sb¢2
352, 353, 354, 355	Sbφ3
231, 229, 228, 227	$\langle \bigcup_{N} \longrightarrow CH_2 \longrightarrow 0 \longrightarrow CH_3$
201, 199, 198	$\langle \bigvee_{N}^{N} \longrightarrow CH_{2} \longrightarrow O^{O-CH_{3}}$
108, 107, 106	С о — сн ₃
81, 80	
92, 93, 94	
152, 151, 150, 147	

(continued)

TABLE 4	(continued)
---------	-------------

m/e ^b	Ion fragment
138, 137, 136	0 ^{- сн} 3 сн3
601	Repeat unit
453	Repeat unit minus
544	Repeat unit minus 2¢CH3
493, 495	Repeat unit minus ϕ , ϕ CH ₃
465	Repeat unit minus ϕ , $2\phi CH_3$
719	Repeat unit plus
	$\stackrel{H}{\sim}$ \swarrow $\stackrel{NH}{\sim}$ $\stackrel{NH}{\sim}$
805	Repeat unit plus
	$\overset{H}{\overset{N}{\longrightarrow}} \overset{N}{\overset{N}{\longrightarrow}} \overset{CH_{2}}{\overset{O}{\longrightarrow}} \overset{O}{\overset{CH_{3}}{\longrightarrow}} \overset{CH_{3}}{\overset{O}{\longrightarrow}} \overset{CH_{3}}{\overset{CH_{3}}{\longrightarrow}} \overset{CH_{3}}{\overset{CH_{3}}{\longrightarrow}} \overset{CH_{3}}{\overset{O}{\longrightarrow}} \overset{CH_{3}}{\overset{CH_{3}}{\longrightarrow}} \overset{CH_{3}}{\overset{CH_{3}}{\overset{CH_{3}}{\longrightarrow}} \overset{CH_{3}}{\overset{CH_{3}}{\longrightarrow}} \overset{CH_{3}}{\overset{CH_{3}}{\longrightarrow}} \overset{CH_{3}}{\overset{CH_{3}}{\overset{CH_{3}}{\longrightarrow}} \overset{CH_{3}}{\overset{CH_{3}}{\overset{CH_{3}}{\longrightarrow}} \overset{CH_{3}}{$
^a Repeat unit	H , N
	$ \xrightarrow{N} \xrightarrow{CH_2} \xrightarrow{O} \xrightarrow{CH_3} $

compounds are given in Tables 4 and 5. These also exhibit the presence of the repeat unit and various fragments of the repeat unit.

Of importance in applications, which may involve extended (high) temperatures, is the presence of fragments containing antimony. While the toxicity of the particular gaseous species is not known, the threshold limiting value, TLV, for antimony in air is 0.5 mg/m^3 and the reported fatal dose of antimony compounds by ingestion is 100-200 mg [7]. Antimony toxicity is reversible and, in cases of acute poisoning, survival for 48 h indicates that recovery is probable [7]. Thus antimony and typical antimony compounds are considered only moderately toxic when compared with other metals such as beryllium (TLV = $2.0 \times 10^{-3} \text{ mg/m}^3$), cadmium (0.1 mg/m^3), chromium (0.1 mg/m^3), and lead (0.15 mg/m^3).

TABLE 5. Major Ion Fragments for the Gases Derived from the Thermal Degradation of the Condensation Product of Triphenylantimony Dichloride with 4,4-Diaminodiphenylsulfon^a

m/e ^b	Ion fragment
79, 78, 77	φ
199, 200	Sbø
275, 277	Sb¢2
354, 355	Sbøs
216	O
508	Repeat unit minus ϕ NH
275	O Repeat unit minus ø, N-ø-S-ø-N O
^a Repeat unit $\begin{pmatrix} \phi & H \\ f & f \end{pmatrix}$ $\begin{pmatrix} s_b - N \\ f \end{pmatrix}$	$- \underbrace{\bigcirc}_{i}^{0} \underbrace{\stackrel{H}{s}}_{i} - \underbrace{\bigcirc}_{i}^{H} \underbrace{\stackrel{H}{s}}_{i} + \underbrace{\bigcirc}_{i} \underbrace{\stackrel{H}{s}}_{i} + \underbrace{\underbrace{O}}_{i} \underbrace{\stackrel{H}{s}}_{i} + \underbrace{O}_{i} \underbrace{O}_{i} + \underbrace{O}_{i} + \underbrace{O}_{i} \underbrace{O}_{i} + \underbrace{O}_{i} \underbrace{O}_{i} + \underbrace{O}_{i} + \underbrace{O}_{i} \underbrace{O}_{i} + \underbrace{O}_{i} +$

^bCIMS (m/e = 60 to 560).

Light-Scattering Photometry

The molecular weight results for selected products appear in Table 6. The products are oligomeric $(\overline{M}_{w} \cong 3 \times 10^{3}, \overline{DP} \cong 10)$ to high polymers $(\overline{M}_{w} \cong 4 \times 10^{6})$.

Product Structure Summary

The presence of both moieties derived from the organoantimony and diamine is indicated by results obtained employing infrared and mass spectroscopy and control reactions. Elemental analyses results are in fair agreement with the proposed structure. The formation of the Sb-N linkage is consistent with previous studies and indicated by infrared spectral results. The formation of a repeat unit of Form 3 is further indicated by the presence of ion fragments assigned as being derived from this repeat unit. The polymeric nature of the product is shown by molecular weight determinations. Thus both the circumstantial and physical evidence are in agreement with products containing repeat units as depicted in Form 3.

BIOLOGICAL ASSAYS

Most antimony-containing products exhibit some toxicity to humans which must be considered when considering applications for antimonycontaining products. Antimony-containing materials are fairly readily available and are known for their use in biological systems. Toxicity can be reduced through complexing the antimony in a polymeric-type compound.

The antimony(V) polyamine products were tested as solids regarding their inhibitory nature toward a variety of bacteria described in Table 7 [8]. Results of this study are found in Table 8. The majority of the compounds exhibit no inhibition against the bacteria studied. However, some of the products show slight or good inhibition. Also, of the products tested, Zineb is the only aliphatic diamine which is a known antifungal, and the polymer derived from it exhibits good inhibition. It must be emphasized that these inhibition studies were conducted on the products in the solid state. Future studies should involve studies with the products solubilized in a suitable liquid such as DMSO.

Solutions of the antimony(V) polyamines were used to study their activity against selected cell lines. The results are tabulated in terms of percent of cells that survived (Tables 9-11). For the cell line BHK-21 (Table 9) the majority of the products show an expected decrease in inhibition in cell growth with a decrease in concentration. A few products showed no dependence on concentration for cell sur-

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Triphenylantimony dichloride 1,6-Diar " 2,6-Diar " 2,6-Diar " 2,6-Diar	lem inchestene		
" 2,6-Diar 2,6-Diar 2,6-Diar		36	
	lamtno-8-purinol	39	3.7×10^{3}
" 2,6-Dian	lam inoanth raquinone	40	
	lamino-5-nitropyrimidine	14	
" 2,4-Diar	iam ino-5(3,4-dimethoxybenzil)py rim ldine	21	4.8×10^{3}
" 4,4' - Dia)iam inodiphenylsulfon	18	4.5×10^{3}
" Adenine	ne	6	2.6×10^{3}
" p-Pheny	nylenediamine	12	
" 2-Metho	hoxy-p-phenylenediamine	76	
2,3,5,6-	8-Tetramethyl p-phenylenediamine	8	
" 2,5-Dich	ichloro-p-phenylenediamine	39	4.3×10^{3}
" 2-Nitro-	.o-p-phenylenediamine	23	
". 4,4' Metl	ethylenedianiline	54	
" 4,4' - Dia	01am inobenzani lide	43	
" Zineb		36	
" 2,5-Dich	ichloro-p-phenylenediamine	40	9.3×10^3
Triphenylantimony dibromide 2,6-Dian	iamino-8-purinol	62	1.0×10^{3}
" 4,4' - Dia)iam inodiphenyi	51	1.2×10^3
Trimethylantimony dichloride 2,6-Dlan	lamtno-8-purinol	93	4.4×10^{5}
" 4,4' - Dia) iam i nodipheny lsulfon	40.6	3.7×10^{6}

TABLE 6. Results as a Function of Reactants^a

TABLE 7. Bacteria Studied for the Effect of Antimony(V) Polyamines

Actinobacter calcoaceticus: Gram-negative rod Can be an opportunistic pathogen but is part of the normal flor the skin and mouth Associated with conjunctivities, keratitus, and chronic ear info	ra of ections
Alcaligenes faecalis: Gram-negative rod Part of the normal intestinal flora Has been found to cause urinary tract infections and in debilit: individuals septicemia or meningitis	ated
Branhamella catarrhalis: Gram-negative diplococcus Part of the normal oral and nasopharyngeal flora Has been associated with mucous membrane inflammations, ve discharges, meningitis, and bacterial endocarditis	enereal
Enterobacter aerogenes: Gram-negative rods Part of the normal flora of the intestinal tract Has caused urinary tract infections, endocarditis, pneumonia, bacteremia	and
Escherichia coli: Gram-negative rod Part of the normal intestinal flora Most frequent cause of urinary tract infections May cause cholecystitus, appendicitis, peritonitis, sinusitis, a summer diarrhea	nd
Klebsiella pneumoniae: Gram-negative rod Part of the normal flora of the nose, mouth, and intestines May cause lesions in various parts of the body, pneumonia, ch lung abscess, sinusitis, and upper respiratory infections	ronic
Neisseria mucosa: Gram-negative cocci Part of normal flora of the oronasopharynx Generally nonpathogenic but has been found in rare cases of m	eningitis
Pseudomonas aeruginosa: Gram-negative rod Common inhabitant of soil Frequently found as part of normal flora of the intesting and s Opportunistic pathogen, may infect wounds, contaminates burn draining sinuses and decubitus ulcers, cause urinary tract is tions, eye infections, and meningitis	kin s, nfec-
Staphylococcus epidermis: Gram-positive cocci Generally causes mild infections but has caused septicemis, b endocarditis, and urinary tract infections	acterial
Staphylococcus aureaus: Gram-positive cocci Causes "pimplies," abscesses, impetigo, wound infections, pye cystitis, "food poisoning," pneumonia, meningitis, and enter	elitis, itis

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φ ₃ SbCl ₂ + diamine	ы	coli	Alcal	Staph E	Staph A	Entero	Neiss	Kleb	Bran	Pseu	Actin
4,6-Diamino-2-methyl-5- nitrosopyrimidine	-		-	SI	I	I	I	н	н	-	–
Adenine	z		Z	N	N	N	N	z	z	z	z
2-Nitro-p-phenylenediamine	Z		N	N	N	N	N	Z	N	Z	z
4,4'-Diaminodiphenylsulfon	N		I	Z	N	Z	1	Z	I	CI	SI
4,4' - Diaminobenzanilide	Z		N	z	Z	N	N	N	N	N	N
2,5-Dichloro-p-phenylene- diamine	Z		Z	z	z	Z	z	Z	N	N	Z
2,4-Diamino-5(3,4-dimethoxy- benzil)pyrimidine	Z		N	CI	z	z	z	SI	I	z	N
4,6-Diamino-5-nitroso- pyrimidine	Z		z	z	z	Z	N	Z	N	N	Z
4,4' - Methylenedianiline	z		N	CI	z	N	N	N	I	z	N
2,6-Diamino-8-purinol	z		N	z	Z	Z	N	Z	N	z	z
2,6-Diaminopyridine	z		N	Z	Z	N	N	N	N	z	Z
Zineb	IS		Ι	Ι	SI	IS	1	IS	I	I	I

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 $^{a}N = no$ inhibition; SI = slight inhibition; CI = inhibition but cloudy zone, I = inhibition, clear zone.

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2µg/mL 49.6 45.9 66.4 79.5 58.4 47.0 52.1 0 0 Results of the Effect of Selected Antimony(V) Polyamines on the Cell Line BHK-21^a 3 µg/mL 44.5 50.4 33.6 49.3 51.6 40.7 58.1 0 0 5 µg/mL 36.5 15.7 45.3 51.3 46.2 30.3 50.4 0 0 5 µg/mL 61.5 29.9 52.4 10.3 51.356.1 48.1 0 0 6 µg/mL 20.2 19.7 67.2 79.5 33.9 44.7 55.3 0 0 2,4-Diamino-5(3,4-dimethoxybenzil)-2,5-Dichloro-p-phenylenediamine 2-Nitro-p-phenylenediamine 2,6-Diaminoanthraguinone 4.6-Diamino-2-methyl-5-4,4'-Diaminobenzanilide 2,6-Diamino-8-purinol 2,6-Diaminopyridine nitrosopyrimidine ϕ_3 SbCl₂ + diamine TABLE 9. pyrimidine Adenine

^aValues are in % viable cells.

ANTIMONY(V) POLYAMINES

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e L929 ^a	, 2,µg/1
the Cell Line	3 µg/mL
Polyamines on	3 μg/mL
ed Antimony(V)	5 μg/mL
Results of the Effect of Selecte	6 µg/mL
TABLE 10.	Cl ₂ + diamine

by SbCl ₂ + diamine $6 \mu g/mL$ $5 \mu g/mL$ $3 \mu g/mL$ $3 \mu g/mL$ $3 \mu g/mL$ $18.6 m trosopyrimidine 10.8 4.2 16.4 18.6 16.7 46.4 30.0 25.0 25.0 25.0 25.0 25.14 80.8 46.2 25.0 25.14 10.8 47.5 51.4 80.8 46.2 25.0 25.0 25.0 25.0 25.0 25.0 25.0 25$						
$1, 6$ -Diamino-2-methyl-5- nitrosopyrimidine 10.8 4.2 16.4 18.6 n itrosopyrimidine 16.7 46.4 30.0 25.0 2 -Nitro-p-phenylenediamine 47.5 51.4 80.8 46.2 2 -Nitro-p-phenylenediamine 47.5 51.4 80.8 46.2 $1, 4^{+}$ -Diaminobenzanilide 56.9 49.4 47.2 63.6 $2, 5$ -Dichloro-p-phenylenediamine 56.1 53.9 49.2 46.4 $2, 4$ -Diamino- $5(3, 4$ -dimethoxybenzil)- 64.4 44.2 66.2 55.0 $2, 6$ -Diamino-nomulting 22.8 22.8 55.0 42.5 $2, 6$ -Diamino- 8 -purtinol 61.9 53.1 48.4 48.4	b ₃ SbCl ₂ + diamine	6 µg/mL	5 µg/mL	3 μg/mL	3 µg/mL	2 µg/mĽ
Adenine 16.7 46.4 30.0 25.0 $-Nitro-p-phenylenediamine47.551.480.846.24.4^{-}-Diaminobenzanilide56.949.447.263.67.5-Dichloro-p-phenylenediamine56.153.949.246.47.4-Diamino-5(3,4-dimethoxybenzil)-64.444.266.255.07.6-Diamino-8-purinoidine22.822.855.042.57.6-Diamino-8-purinoi61.953.148.448.4$	l,6-Diamino-2-methyl-5- nitrosopyrimidine	10.8	4.2	16.4	18.6	50.6
3. Nitro-p-phenylenediamine 47.5 51.4 80.8 46.2 4. Diaminobenzanilide 56.9 49.4 47.2 63.6 5. Dichloro-p-phenylenediamine 56.1 53.9 49.2 46.4 4. Diamino-5(3,4-dimethoxybenzil)- 54.4 44.2 66.2 55.0 9. F-Diamino-5(3,4-dimethoxybenzil)- 54.4 44.2 66.2 55.0 7. F-Diamino-6(3,4-dimethoxybenzil)- 54.4 44.2 66.2 55.0 7. F-Diamino-8(1,9) 53.1 48.4 48.4 7. F-Diamino-8-purtinol 61.9 53.1 48.4 48.4	Adenine	16.7	46.4	30.0	25.0	70.6
1,4'-Diaminobenzanilide 56.9 49.4 47.2 63.6 3,5-Dichloro-p-phenylenediamine 56.1 53.9 49.2 46.4 3,4-Diamino-5(3,4-dimethoxybenzil)- 64.4 44.2 66.2 55.0 pyrimidine 22.8 22.8 55.0 42.5 ,6-Diamino-8-purinole 61.9 53.1 48.4	}-Nitro-p-phenylenediamine	47.5	51.4	80.8	46.2	70.6
1,5-Dichloro-p-phenylenediamine 56.1 53.9 49.2 46.4 1,4-Diamino-5(3,4-dimethoxybenzil)- 64.4 44.2 66.2 55.0 pyrimidine 22.8 22.8 55.0 42.5 c,6-Diamino-8-purtinol 61.9 53.1 48.4 48.4	i,4' -Diaminobenzanilide	56.9	49.4	47.2	63.6	71.4
1,4-Diamino-5(3,4-dimethoxybenzil)- 64.4 44.2 66.2 55.0 pyrimidine 64.4 44.2 66.2 55.0 1,6-Diaminoanthraquinone 22.8 22.8 55.0 42.5 2,6-Diamino-8-purinol 61.9 53.1 48.4 48.4	t, 5-Dichloro-p-phenylenediamine	56.1	53.9	49.2	46.4	73.9
, 6-Diaminoanthraquinone 22.8 22.8 55.0 42.5 , 6-Diamino-8-purinol 61.9 53.1 48.4 48.4	, 4-Diamino-5(3,4-dimethoxybenzil)- pyrimidine	64.4	44.2	66.2	55.0	80.3
, 6-Diamino-8-purinol 61.9 53.1 48.4 48.4	,,6-Diaminoanthraquinone	22.8	22.8	55.0	42.5	71.2
	t, 6-Diamino-8-purinol	61.9	53.1	48.4	48.4	57.3
7,6-Diaminopyridine 43.4 81.4 50.8 63.9	t, 6-Diaminopyridine	43.4	81.4	50.8	63.9	66.7

^aAll values are in % viable.

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¢₃SbCl₂ + diamine	6 µg/mL	5 µg/mL	4 μg/mL	3 μg/mL	2 µg/mL
4, 6-Diamino-2-methyl-5- nitrosopyrimidine	55.7	38.2	25.7	29.3	67.9
Adenine	15.4	32.9	55.0	64.3	52.8
2-Nitro-p-phenylenediamine	74.3	74.3	75.7	90.0	87.9
4,4' - Diam inobenzanilide	89.3	81.1	83.6	78.6	88.6
2,5-Dichloro-p-phenylenediamine	85.4	70.0	72.5	98.2	92.9
2,4-Diamino-5(3,4-dimethoxybenzil)- pyrimidine	76.8	68.2	68.9	68.2	83.6
2,6-Diaminoanthraquinone	18.9	42.9	92.1	55.0	74.6
2,6-Diamino-8-purinol	77.9	0	63.8	79.6	83.2
2,6-Diaminopyridine	57.1	0	63.9	78.9	72.9

^aAll values are in % viable cells.

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vival. These same findings hold true for results with the cell lines L929 and HeLa (Tables 10 and 11, respectively).

These biological analyses are meant to only briefly evaluate the potential inhibitory nature of the compounds. As noted, all of the antimony-containing compounds exhibited inhibition to some degree. Applications requiring some biological inhibition might be a use for these compounds as typical application and additives to coatings, cloth, and wood products.

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