collected by filtration and recrystallized from ethanol to yield 10.1 g (89%) of white crystals, mp 250–251°.

Anal. Calcd for $C_{10}H_{24}N_2 \cdot 2HCl$: C, 49.0; H, 10.7; N, 11.4. Found: C, 49.0; H, 10.3; N, 11.4.

Biological Section

Methods and Results.—All of the alkylating agents listed in Table I and II and compounds **22–24** were evaluated as inhibitors of reproduction in our colony of houseflies (*Musca domestica* L.). The method used was previously reported.[1] At 1 wt % concentration in the feed, only those compounds listed in Table III were active. All compounds were mixed dry in the feed.

Table III
Effects of Compounds on the Reproduction of Houseflies

Compd	n	Wt % in feed	No. of flies	% egg batch—Days of oviposition— 1	2	3	4	5	6	7
14	7	1.0	300	39	46	42	66	50	65	49
14	7	0.1	300	76	90	77	81	80	75	82
1	8	1.0	250	..	2	1	.	0		..
1	8	0.1	250	14	10	18	18	38	23	27
1	8	0.01	250	..	77	61	72	70	62	72
15	9	1.0	300	0	0	0	0	0	0	0
15	9	0.1	300	0	..	23	42	41	22	29
16	10	1.0	300	9	16	0	0	0	17	..
16	10	0.1	300	43	73	38	57	50	80	..
17	11	1.0	150	68	44	74	53	74	38	63
Control		...	400	94	94	92	96	94	83	..
Control			250	92	97	90	98	88	91	87

Discussion.—The lack of effect on reproduction of the housefly of all of the bromoacetyl derivatives synthesized agrees with the previously reported results[1] and indicates the necessity of a more active alkylating function before interference with reproduction is achieved. With the aziridinyl derivatives, the necessity of having an NH function on the amide is emphasized by the lack of activity of **19–21**. The necessity of having a bisaziridinyl function is borne out by the lack of activity of **24**. Distance between the aziridine groups is extremely important with regard to activity of these compounds. Maximum activity was found in the C_8–C_9 compounds and dropped off at shorter and longer chain lengths. Investigations are under way in other diamines to better define the distance requirements for maximal chemosterilant activity.

Acknowledgment.—This work was supported by U. S. Public Health Service Grant GM-11491. We wish to thank V. Tovar for assistance with the biological studies.

Insect Chemosterilants. III. 1-Aziridinylphosphine Oxides[1]

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Insect sterilizing activity of 40 variously substituted tri-, di-, and mono(1-aziridinyl)phosphine oxides was correlated with the degree of substitution on the aziridinyl ring and with the type of nonaziridinyl moieties attached to the phosphinylidyne group. All substitutions on the aziridinyl carbon decreased the sterilizing activity of the parent compound. Among the di- and monoaziridinyl compounds, the most effective nonaziridinyl substituents were alkylamino groups, less effective were alkoxy groups, and least effective were aryl groups. In preparing strained aziridinylphosphine oxides, 3-oxa-6-azabicyclo[3.1.0]hexane was synthesized.

Tris(1-aziridinyl)phosphine oxide (tepa) was one of the first chemicals found to effectively sterilize the males of many different insect species.[2] During the years immediately following the discovery, variously substituted aziridinylphosphine oxides were synthesized or obtained from outside sources and then screened for their insect sterilizing activity. In attempts to correlate the structure of substituted phosphine oxides with their chemosterilant activity, two approaches were investigated. One was the introduction of substituents onto one or both carbon atoms of the aziridinyl ring, and the other was the replacement of all aziridinyl moieties with various other groups. All substituted phosphine oxides described in this paper contained at least one aziridinyl group linked to the phosphinylidyne group through its ring nitrogen.

Triaziridinylphosphine Oxides.—The effects of progressive substitution of the hydrogens in tepa by methyl groups have been previously reported.[3] In a recent quantitative study,[4] tepa was found to be 13 times as active in sterilizing male house flies (*Musca domestica* L.) as its C-methyl homolog, metepa. Table I summarizes the sterilizing properties of other tepa analogs used to treat the house fly. Tepa and metepa are included for comparison. Derivation of the values used in grading the sterilizing activity of the compounds tested is discussed in the Experimental Section. Compounds designated by literature references only were synthesized in our laboratory according to published procedures.

Introducing one higher alkyl group or phenyl group into each aziridine ring drastically reduced the sterilizing activity (Table I). In this respect, the effects were comparable to those obtained by introducing two or more methyl groups into the ring.[3] The reduced physiological activity of the substituted compounds appeared to be related to their decreased susceptibility to nucleophilic reagents. Simple measurements of

(1) Presented before the Symposium on Chemosterilants of the Division of Agricultural and Food Chemistry, 150th National Meeting of the American Chemical Society, Atlantic City, N. J., Sept 12–17, 1965. Previous paper: A. B. Bořkovec and C. W. Woods, *J. Med. Chem.*, **8**, 545 (1965).

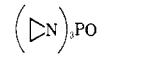
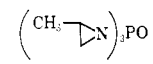
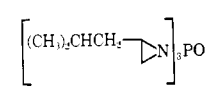
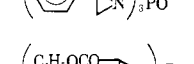
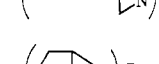
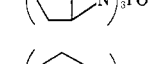
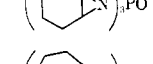
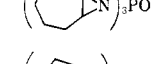
(2) A. B. Bořkovec, *Residue Rev.*, **6**, 87 (1964).

(3) A. B. Bořkovec and C. W. Woods, Advances in Chemistry Series, No. 41, American Chemical Society, Washington, D. C., 1963, p 47.

(4) S. C. Chang and A. B. Bořkovec, *J. Econ. Entomol.*, **57**, 488 (1964).

TABLE I

HOUSE FLY STERILIZING ACTIVITY OF TRIAZIRIDINYLPHOSPHINE OXIDES

Compd	Structure	Graded activity[a]	Source
1		+++	*b*
2		++	*b*
3		–	*c*
4		–	*d*
5		–	*e*
6		–	*e*
7		–	*e*
8		–	*e*
9		–	*e*

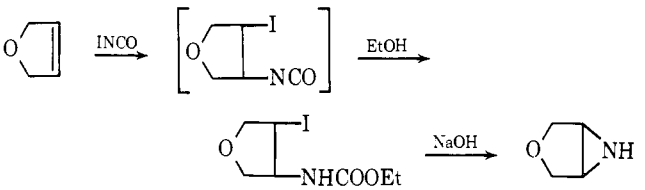
[a] +++, high activity (comparable to or higher than that of tepa); ++, moderate activity (comparable to metepa); +, low but significant activity; and –, insignificant or not detectable activity at any concentration tested. [b] Commercial sources. [c] A. T. Bottini, University of California, Davis, Calif. [d] Interchemical Corp. [e] See Experimental Section.

the stability of tepa and metepa in aqueous solutions[5] supported this proposition. In order to gain further evidence, tepa analogs, prepared so that the aziridine rings contained either electron-withdrawing substituents or cyclic groups, were tested. (The cyclic groups could be expected to impart additional strain to the ring.) For those analogs with electron-withdrawing constituents, preparation of the intermediate basic aziridine proved to be difficult. Only esters of 2-aziridinecarboxylic acid[6] were suitable for synthesis of the tepa analog. Ethyl 2-aziridinecarboxylate was converted with phosphorus oxychloride to the corresponding tris(2-carbethoxy-1-aziridinyl)phosphine oxide, but the sterilizing activity of the latter was very low.

Although the tris(2,3-dimethyl-1-aziridinyl)phosphine oxide[3] was a much weaker chemosterilant than metepa, it was conceivable that if the two alkyl substituents were linked together, thus exerting additional strain on the already strained aziridine ring, the sterilizing activity of the bicyclic derivative might be increased. Cycloalkenimines, previously synthesized,[7] appeared suitable for this purpose.

The treatment of 6-azabicyclo[3.1.0]hexane, 7-azabicyclo[4.1.0]heptane, and 8-azabicyclo[5.1.0]octane, respectively, with phosphorus oxychloride, yielded the expected trisubstituted phosphine oxides (Table I). Unfortunately, none of the compounds demonstrated significant sterilizing activity.

Introducing an additional heteroatom (oxygen) into the cycloalkenimines was also tried. The base, 3-oxa-6-azabicyclo[3.1.0]hexane, which represents a new heterocyclic system, was prepared according to a reaction sequence applied previously to the synthesis of cycloalkenimines.[8]



The structure of the product was confirmed by infrared and nmr spectra (Figure 1). Treatment of the base with phosphorus oxychloride gave tris(3-oxa-6-azabicyclo[3.1.0]hexan-6-yl)phosphine oxide, but its sterilizing activity was again negligible.

It appears then that any substitution on the aziridinyl rings of tepa gives rise to compounds that are less effective in sterilizing insects than the parent compound.

Diaziridinylphosphine Oxides.—Replacement of the entire aziridinyl moiety by another group resulted in compounds of surprisingly high sterilizing activity in spite of the total number of aziridinyl groups per molecule being reduced. Effects of the replacement of one aziridinyl ring with aryl, alkoxy, and substituted-amino groups are indicated in Table II. Several of the listed compounds also bear substituents on the two aziridinyl rings. Choice of compounds was governed by their availability rather than by intention. Most of the compounds were obtained from outside sources or prepared according to published procedures. Fortunately, the sterilizing activities of the parent triaziridinyl compounds were known and thus the relative activities of the derivatives are of significance. The most striking feature of the diaziridines is the high activity of the amino-substituted compounds. Quantitative evaluation[9] of compounds **14–18** revealed that their activity in sterilizing male house flies was higher than that of the parent compound tepa and that **14**, a methylamino derivative, is one of the most potent male house fly sterilants known. Because the aziridinyl moiety is undoubtedly the carrier of sterilizing activity in this type of alkylating agents, one could expect that a reduction in the number of such groups in a molecule of a chemosterilant would always lead to a decrease in sterilizing activity. Although the physical properties of the lower alkylamino derivatives (**14–17**) do not appear to be different from those of tepa (**1**), the findings reported herein accentuate the importance of the carrier portion of the molecule to its physiological activity. In general, the sterilizing activity decreases with the increasing chain length of the alkyl group, the octyl compound (**20**) being only slightly active. Dialkyl substitution on the amino nitrogen (**28–30**) is also effective, particularly when methyl groups are the substituents (**28**).

(5) M. Beroza and A. B. Bořkovec, *J. Med. Chem.*, **7**, 44 (1964).

(6) Hoffmann-LaRoche & Co., British Patent 847,205 (Sept 7, 1960).

(7) (a) O. E. Paris and P. E. Fanta, *J. Am. Chem. Soc.*, **74**, 3007 (1952); (b) P. E. Fanta, *J. Chem. Soc.*, 1441 (1957); (c) P. B. Talukdar and P. E. Fanta, *J. Org. Chem.*, **24**, 555 (1959).

(8) (a) A. Hassner and C. Heathcock, *Tetrahedron*, **20**, 1037 (1964). (b) While this paper was in preparation, the synthesis of 3-oxa-6-azabicyclo-[3.1.0]hexane by the Wenker method was announced; *cf.* P. E. Fanta and E. N. Walsh, *J. Org. Chem.*, **31**, 59 (1966).

(9) S. C. Chang and A. B. Bořkovec, *J. Econ. Entomol.*, in press.

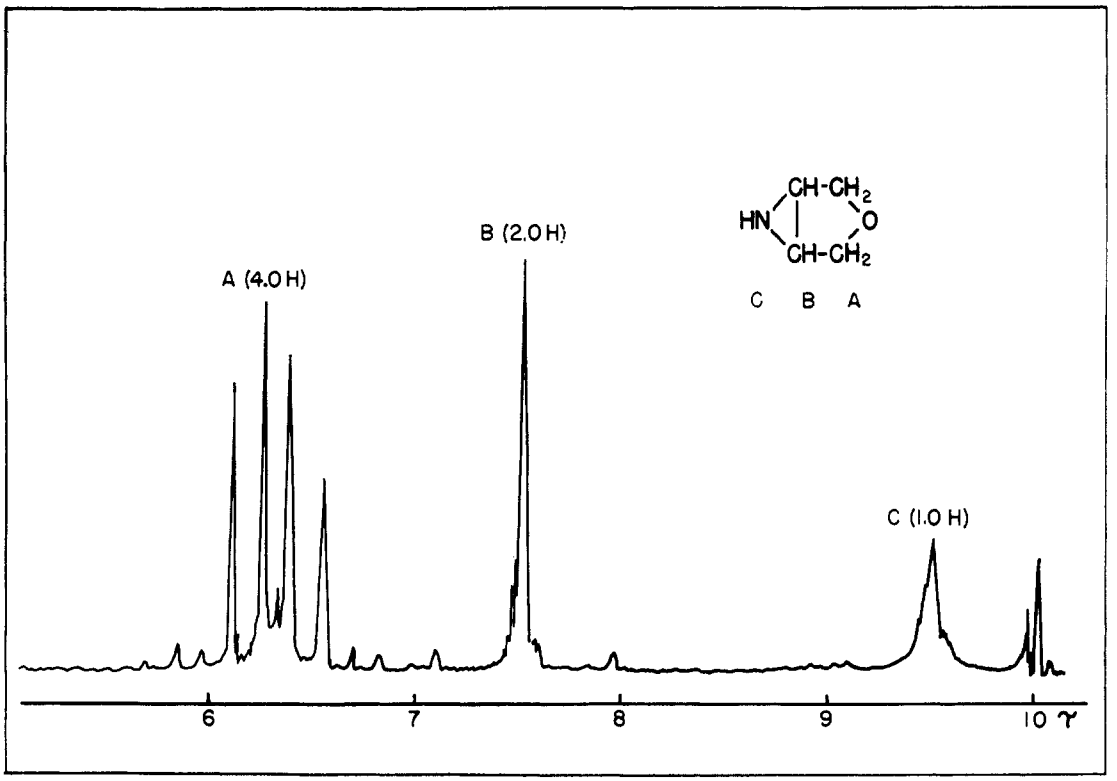


Figure 1.—Nmr spectrum of 3-oxa-6-azabicyclo[3.1.0]hexane in CCl_4 (13%).

More precise determinations,[9] however, indicated a decrease in activity of the dimethyl compound **28** compared with that of the monomethyl compound **14**. Alkyls were not the only substituents to maintain a high activity (**22**, **24**, **26**, and **27**), but they were the only ones giving rise to activities greater than the activity of tepa. Alkoxy (**11**) and aryloxy (**12** and **13**) diaziridines were also active, but substantially less so than the parent triaziridinyl compounds. The aryl group (**10**) proved to be even less active as a substituent than the aryloxy group.

Monoaziridinylphosphine Oxides.—The effects of substituents in the monoaziridinyl series (Table III) paralleled those in the diaziridinyl series. Most of the compounds, with the exception of **38**, had either very low (**33** and **39**) or insignificant activity. Attempts to prepare alkylamino derivatives analogous to **14**, **16**, and **17** were unsuccessful. In spite of several modifications in the experimental procedures only ill-defined polymeric materials were obtained.

Several phosphine sulfides, analogous to the phosphine oxides presented here, were tested.[10] Results substantiated the findings evident in Tables I–III. Unfortunately, the sulfur compounds often exhibited a high toxicity to test insects; their usefulness in structure–activity studies was thus severely limited.

Comparison of the activities of **22** and **23**, **33** and **34**, as well as **38–40**, further substantiates the effects of alkyl substituents on the aziridinyl ring previously observed[3,4] in tepa (**1**) and metepa (**2**).

Results of these tests point to the following generalizations: (a) any substitution on the aziridinyl ring of a chemosterilant reduces the sterilizing activity of the derivative compared with that of the parent compound, and (b) replacing an aziridinyl substituent in a polyaziridinyl chemosterilant does not necessarily reduce the sterilizing activity of the derivative as compared to the parent compound.

(10) Unpublished results.

Experimental Section[11]

Biological Testing.—All compounds listed in Tables I–III were tested at various concentrations as additives to the diet of house flies.[12] Gradation of activities was expressed by a series of plus signs. All feeding tests with house flies were performed by the entomologists of the Entomology Research Division, Agricultural Research Service, U. S. Department of Agriculture, Gainesville, Fla.

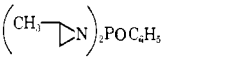
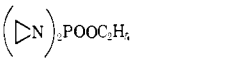
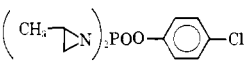
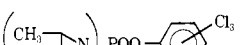
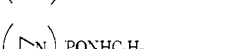
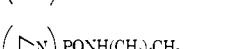
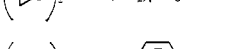
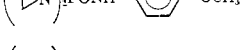
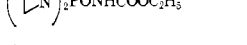
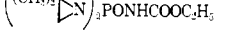
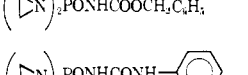
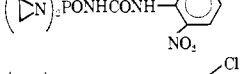
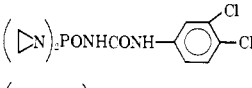
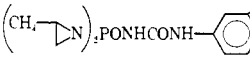
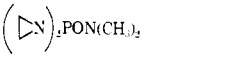
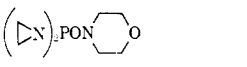
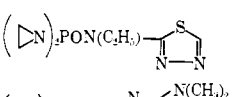
Ethyl 2-Aziridinecarboxylate.—A procedure reported in a patent[6] was followed, but the rate of mixing the reagents proved to be critical. To 500 ml of liquid ammonia was added 31 g of ethyl α-bromoacrylate during a period of 1 hr. If the addition of the acrylate is too rapid it reacts with the intermediate β-amino-α-bromopropionate rather than with NH_3 and the yields of the aziridine are drastically reduced. The present procedure yielded, upon distillation, 11 g (55%) of ethyl 2-aziridinecarboxylate, bp 55–57° (12 mm) [lit.[6] bp 53–54° (11 mm)].

3-Iodotetrahydrofuran-4-ethylcarbamate.—To a solution of iodine isocyanate, prepared by the reaction of 76 g (0.60 mole) of iodine with 50 g (0.33 mole) of freshly prepared silver cyanate in 500 ml of dry ether, was added dropwise 21 g (0.30 mole) of 2,5-dihydrofuran. The temperature was kept at −70° during both reactions and was allowed to rise to room temperature after the addition of dihydrofuran had been completed. Precipitated silver iodide was collected by filtration and the solution was concentrated by distillation under reduced pressure. The resulting isocyanate was not isolated but was converted to the carbamate by treatment with refluxing ethanol for 3 hr. This solution was evaporated to 100 ml, diluted with 600 ml of water, and decolorized with a small quantity of sodium bisulfite. The carbamate was extracted into ether and the ether solution, after drying ($MgSO_4$), was further decolorized by passage through a

(11) All melting and boiling points are corrected.

(12) (a) For detailed experimental procedures, see H. K. Gouck, M. M. Crystal, A. B. Bořkovec, and D. W. Meifert, *J. Econ. Entomol.*, **56**, 506 (1963). (b) For a discussion of concentration effects in oral application of chemosterilants, see C. N. Smith, G. C. LaBrecque, and A. B. Bořkovec, *Ann. Rev. Entomol.*, **9**, 269 (1964).

TABLE II
HOUSE FLY STERILIZING ACTIVITY OF DIAZIRIDINYLPHOSPHINE OXIDES

Compd	Structure	Graded activity[a]	Source
10		+	*b*
11		++	*c*
12		+	*d*
13		+	*d*
14		+++	*e*
15		+++	*f*
16		+++	*e*
17		+++	*g*
18		+++	*f*
19		++	*f*
20		+	*f*
21		+	*h*
22		++	*i*
23		−	*i*
24		++	*i*
25		+	*j*
26		++	*j*
27		++	*j*
28		+++	*g*
29		+	*k*
30		+	*h*
31		++	*g*

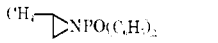
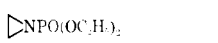
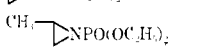
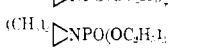
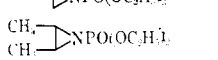
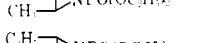
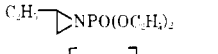
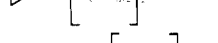
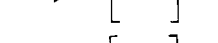
[a] See footnote *a*, Table I. [b] Interchemical Corp. [c] Continental Oil Co. [d] Minnesota Mining and Manufacturing Co. [e] Prepared according to T. Mizuma, Y. Minaki, and S. Toyoshima, *Yakugaku Zasshi*, **81**, 48 (1961). [f] Edwin Kuh, Rutgers University. [g] See Experimental Section. [h] American Cyanamid Co. [i] Armour Pharmaceutical Co. [j] The Squibb Institute. [k] National Institutes of Health.

column of alumina. The crystalline product obtained by evaporation of the ether solution and two recrystallizations from cyclohexane weighed 30 g (35%) and melted at 81–84°.

Anal. Calcd for $C_7H_{12}INO_3$: C, 29.41; H, 4.51; I, 44.39; N, 4.90. Found: C, 29.70; H, 4.27; I, 44.75; N, 5.01.

3-Oxa-6-azabicyclo[3.1.0]hexane.—A solution of 23.7 g (0.083 mole) of 3-iodotetrahydrofuran-4-ethylcarbamate and 14.1 g (0.25 mole) of KOH in 400 ml of water was heated at reflux temperature for 2.5 hr. The solution was saturated with NaCl and continuously extracted with CH_2Cl_2 overnight. Concen-

TABLE III
HOUSE FLY STERILIZING ACTIVITY OF MONOAZIRIDINYLPHOSPHINE OXIDES

Compd	Structure	Graded activity[a]	Source
32		−	*b*
33		+	*b*
34		−	*b*
35		−	*b*
36		−	*b*
37		−	*b*
38		++	*c*
39		+	*d*
40		−	*d*

[a] See footnote *a*, Table I. [b] Interchemical Co. [c] See Experimental Section. [d] P. E. Sonnet and A. B. Bořkovec, unpublished data.

tration of the extract followed by distillation at reduced pressure gave 3.8 g (54%) of the aziridine, bp 48–51° (22 mm), $n^{22}D$ 1.4694.

Anal. Calcd for C_4H_7NO: C, 56.45; H, 8.29; N, 16.46. Found: C, 56.26; H, 8.43; N, 16.20.

Tris(6-azabicyclo[3.1.0]hexan-6-yl)phosphine Oxide (6).—To 1.4 g (0.017 mole) of 6-azabicyclo[3.1.0]hexane[7b] and 1.7 g (0.017 mole) of triethylamine in 25 ml of benzene was added 0.76 g (0.005 mole) of $POCl_3$ in 14 ml of benzene. The reaction mixture was cooled in an ice bath during the addition and then kept at 25° for 3 hr. The precipitated triethylamine hydrochloride was collected by filtration, and the filtrate was evaporated to an oily, crystalline mass. Two recrystallizations from pentane gave 0.9 g (60%) of colorless crystals, mp 105–107°.

Anal. Calcd for $C_{15}H_{24}N_3OP$: C, 61.42; H, 8.25; N, 14.32. Found: C, 61.17; H, 8.34; N, 14.52.

Tris(7-azabicyclo[4.1.0]heptan-7-yl)phosphine oxide (7) was prepared from 7-azabicyclo[4.1.0]heptane[7a] in the same manner as the homolog **6**. The product was obtained in a 30% yield after recrystallization from pentane and melted at 81–83.5°.

Anal. Calcd for $C_{18}H_{30}N_3OP$: C, 64.45; H, 9.01; N, 12.53. Found: C, 64.20; H, 8.82; N, 12.67.

Tris(8-azabicyclo[5.1.0]octan-8-yl)phosphine oxide (8) was prepared from 8-azabicyclo[5.1.0]octane[7c] in the same manner as **6**. The product was obtained in a 60% yield after recrystallization from pentane and melted at 89–90°.

Anal. Calcd for $C_{21}H_{36}N_3OP$: C, 66.81; H, 9.61; N, 11.13. Found: C, 66.67; H, 9.75; N, 11.25.

Tris(3-oxa-6-azabicyclo[3.1.0]hexan-6-yl)phosphine oxide (9) was prepared from 3-oxa-6-azabicyclo[3.1.0]hexane in the same manner as **6**. The product was obtained in 75% yield and melted at 149–150° after recrystallization from ethyl acetate.

Anal. Calcd for $C_{12}H_{18}N_3O_4P$: C, 48.16; H, 6.06; N, 14.04. Found: C, 47.86; H, 6.07; N, 14.12.

Tris(2-carbethoxyaziridinyl)phosphine oxide (5) was prepared from ethyl 2-aziridinecarboxylate in the usual manner. The oily product did not crystallize and could not be distilled. Excess reagents were removed by evaporation at 50° (0.1 mm). According to the quantity of the recovered triethylamine hydrochloride the reaction proceeded to completion.

Anal. Calcd for $C_{15}H_{24}N_3O_7P$: C, 46.27; H, 6.21; N, 10.79. Found: C, 46.48; H, 6.35; N, 10.72.

Bis(1-aziridinyl)isopropylaminophosphine Oxide (17).[13]—A mixture of 38 g (0.40 mole) of isopropylamine hydrochloride and 90 ml of $POCl_3$ was heated at reflux for 20 hr. Excess $POCl_3$ was removed by evaporation under reduced pressure and the residue was recrystallized from cyclohexane to give 52 g (75%) of dichloroisopropylaminophosphine oxide, mp 70–71°. To 15 g (0.35 mole) of ethylenimine and 30 g (0.30 mole) of triethylamine in 200 ml of benzene was added dropwise 24.6 g (0.14 mole) of the dichloroisopropylaminophosphine oxide in 50 ml of benzene. The reaction mixture was cooled in an ice bath during the addition and then held at 25° for 1 hr. The precipitated triethylamine hydrochloride was removed by filtration, and the filtrate was evaporated to a crystalline solid. After two recrystallizations from benzene, 13 g (48%) of product, mp 74–76°, was obtained.

Anal. Calcd for $C_9H_{16}N_3OP$: C, 44.44; H, 8.52; N, 22.21. Found: C, 44.49; H, 8.70; N, 22.40.

Bis(1-aziridinyl)dimethylaminophosphine oxide (28) was prepared from dichlorodimethylaminophosphine oxide[14] in the same manner as **17**; bp 60–63° (0.1 mm).

Anal. Calcd for $C_6H_{14}N_3OP$: C, 41.14; H, 8.06; N, 23.99. Found: C, 41.27; H, 8.22; N, 23.75.

1-Aziridinylbis(dimethylamino)phosphine oxide (38) was prepared from chlorobis(dimethylamino)phosphine oxide[14] in the same manner as **17**. A 75% yield of product, bp 52–54° (0.1 mm), was obtained.

Anal. Calcd for $C_6H_{16}N_3OP$: C, 40.67; H, 9.10; N, 23.71. Found: C, 40.41; H, 8.99; N, 23.51.

Dichloro[4,6-bis(dimethylamino)-*s*-triazin-2-yl]aminophosphine Oxide.—A mixture of 20 g (0.11 mole) of 4,6-bis(dimethylamino)-2-amino-*s*-triazine and 150 ml of $POCl_3$ was heated at reflux temperature for 20 hr. The excess $POCl_3$ was removed by distillation under reduced pressure and the resulting white precipitate was washed with chloroform and collected by filtration. The product was insoluble in all common solvents and could not be recrystallized: it weighed 25 g (75%) and melted at 240–255°.

Anal. Calcd for $C_7H_{13}Cl_2N_6OP$: C, 28.11; H, 4.38; Cl, 23.71; N, 28.10; P, 10.36. Found: C, 27.87; H, 4.60; Cl, 23.35; N, 27.84; P, 10.52.

Bis(1-aziridinyl)[4,6-bis(dimethylamino)-*s*-triazin-2-ylamino]phosphine Oxide (31).—To 4.3 g (0.10 mole) of ethylenimine and 8.4 g (0.083 mole) of triethylamine in 100 ml of benzene was slowly added 10 g (0.033 mole) of dichloro[4,6-bis(dimethylamino)-*s*-triazin-2-ylamino]phosphine oxide. The reaction mixture was stirred and cooled in an ice bath during the addition and then kept at 25° overnight. The precipitated triethylamine hydrochloride was removed by filtration and the solvent was evaporated under reduced pressure. The product, after recrystallization from benzene, weighed 4.5 g (43%) and melted at 147–149°.

Anal. Calcd for $C_{11}H_{21}N_8OP$: C, 42.30; H, 6.77; N, 35.87. Found: C, 42.11; H, 6.95; N, 35.58.

(13) To maintain uniformity, all phosphinylidyne compounds were named as substituted phosphine oxides.

(14) J. E. Gardiner and B. A. Kilby, *J. Chem. Soc.*, 1769 (1960).