

Compounds I-XVII were synthesized by the methods in [7-9], and their purity and individuality were monitored by thin-layer chromatography. The structures of the substances were established on the basis of data from the IR, UV, and PMR spectra.

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#### REACTION OF 2,4,6-TRIALKYL-PYRIMIDINE 1,3-DIOXIDES WITH ELECTROPHILIC REAGENTS

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The nitrosation of 2,4,6-trimethylpyrimidine 1,3-dioxide and the bromination of 2,4,6-trimethyl- and 2-ethyl-4,6-dimethylpyrimidine 1,3-dioxides take place primarily at the methyl groups in the 4 and 6 positions of the heteroring. In the reaction of 2,4,6-trimethylpyrimidine 1,3-dioxide with phosphorus oxychloride chlorine is incorporated in the methyl group in the 2 position of the heteroring, while in the reaction with acetic anhydride an acetoxy group is incorporated in the methyl group in the 2 position and in the 5 position of the heteroring, whereas in the case of tosyl chloride a tosyloxy group is incorporated in the 5 position of the heteroring.

Pyrimidine 1,3-dioxides have relatively recently become accessible [1, 2], and virtually no study has been devoted to their chemical properties [3]. The reaction of N-oxides of alkyl-substituted azines with electrophilic reagents takes place both with retention of the oxygen atom of the N-oxide group, as, for example, in halogenation and nitrosation, and with the loss of the oxygen atom of this group, as, for example, in acetoxylation and halogenation with phosphorus oxychloride [4]. In the present research we studied the reactions of 2,4,6-trialkylpyrimidine 1,3-dioxides (Ia, b) with alkyl nitrites, bromine, phosphorus oxychloride, acetic anhydride, and tosyl chloride.

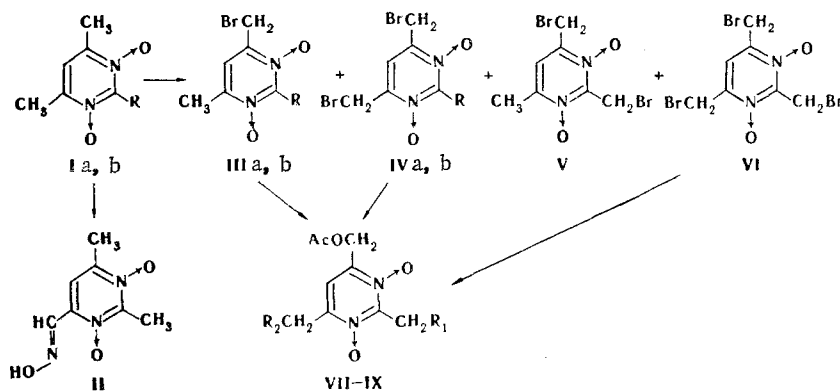
The nitrosation of 1,3-dioxide Ia with ethyl or amyl nitrite in an acidic medium (cf. [5]) led to the formation of 4-oximidomethyl-2,6-dimethylpyrimidine 1,3-dioxide (II). The nonequivalence of the protons of the two methyl groups (2.39 and 2.62 ppm) in the PMR spectrum of II (Table 1) and the coincidence of their chemical shifts with the shifts of the protons of the corresponding methyl groups in starting 1,3-dioxide Ia (2.31 ppm for 4,6-CH<sub>3</sub>, and 2.62 ppm for 2-CH<sub>3</sub> [2]) indicate that nitrosation took place at the methyl group in the 4 position

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of pyrimidine 1,3-dioxide Ia. It may be proposed that the oxime group in II has an E configuration, as in the case of oximes of 2- and 6-formyl-4-phenylpyrimidine 1-oxides [6].

The reaction of 1,3-dioxide Ia with bromine leads to 4-bromomethyl-2,6-dimethyl- (IIIa), 4,6-bis(bromomethyl)-2-methyl- (IVa), 2,4-bis(bromomethyl)-6-methyl- (V), and 2,4,6-tris(bromomethyl)-pyrimidine 1,3-dioxide (VI) (cf. [5, 7]). Of the dibromination products (IVa and V), the principal product is the 4,6-bis(bromomethyl) derivatives (IVa). Only 4-bromomethyl-6-methyl- (IIIb) and 4,6-bis(bromomethyl)-2-ethylpyrimidine 1,3-dioxide (IVb) were isolated from the products of the reaction of 1,3-dioxide Ib with bromine. The structures of bromo derivatives III-VI are in good agreement with the spectral data and the results of elementary analysis of these compounds (Tables 1 and 2). Thus the nonequivalence of the protons of the methyl groups in the PMR spectrum of IIIa made it possible to unambiguously assign the 4-bromomethyl-2,6-dimethylpyrimidine 1,3-dioxide structure to it (Table 1). It follows from the PMR spectra of bis(bromomethyl) derivatives IVa and V and tris(bromomethyl) derivatives VI that the signals of the substituents in the 2 position of pyrimidine 1,3-dioxide are found at weaker field than those of the substituents in the 4 and 6 positions. A bathochromic shift of the absorption maximum as compared with the UV spectra of starting 1,3-dioxides Ia, b is observed in the UV spectra of bromo derivatives III-VI (Table 2).



I a R=CH<sub>3</sub>; b R=CH<sub>2</sub>CH<sub>3</sub>; VII R<sub>1</sub>=R<sub>2</sub>=H; VIII R<sub>1</sub>=H, R<sub>2</sub>=CH<sub>3</sub>COO; IX R<sub>1</sub>=R<sub>2</sub>=CH<sub>3</sub>COO

The corresponding acetoxy methyl derivatives VII-IX are formed in the reaction of bromomethyl derivatives IIIa, IVa, and VI with potassium acetate in the presence of a catalytic amount of 18-crown-6 in acetonitrile. It should be noted that the singlet of the protons of the acetoxy group in the 4 or 4,6 positions of pyrimidine 1,3-dioxide is found a weaker field than the singlet of the same group in the 2 position. This is possibly explained by the shielding effect of two N-oxide groups on the acetoxy group that is remote from the heteroring.

The available data on the reactions of pyrimidine N-oxides with acid anhydrides and their derivatives are limited [3, 8-10]. In the case of N-monoxides these reactions proceed with the formation of  $\alpha$ -functional derivatives of pyrimidine [8-10]; however, the reaction of 4-phenylpyrimidine 1,3-dioxide with acetic anhydride leads to the  $\beta$ -hydroxy derivative rather than to the  $\alpha$ -hydroxy derivative [3]. When 1,3-dioxide Ia is heated with phosphorus oxychloride in chloroform, it is converted to X with the composition C<sub>7</sub>H<sub>7</sub>ClN<sub>2</sub>O, which corresponds to splitting out of one N-oxide oxygen atom and replacement of a hydrogen atom in starting 1,3-dioxide Ia by chlorine. Singlets of protons of two methyl groups at 2.49 ppm, of a methylene group at 4.95 ppm, and of an aromatic proton (5-H) at 7.17 ppm are observed in the PMR spectrum of X (Table 1). However, these data were not sufficient to enable one to choose among three isomeric structures for chloromethylpyrimidine 1-oxide (Xa-c). A comparison of the measured dipole moment ( $\mu_{\text{exp}}$  4.56 D) with the calculated data for Xa-c ( $\mu$  4.94, 1.64, and 2.76 D, respectively) made it possible to assign the 2-chloromethyl-4,6-dimethylpyrimidine 1-oxide structure (Xa) to X.

In contrast to phosphorus oxychloride, the reaction of 1,3-dioxide Ia with acetic anhydride leads to the formation of two acetoxy derivatives XI and XII. Intense bands at 1740 and 1770 cm<sup>-1</sup>, respectively, which correspond to the stretching vibrations of a C=O bond, are observed in the IR spectra of XI and XII. Data from the PMR spectrum of XI (Table 1) made it possible to assume that this compound has the 2-acetoxy methyl-4,6-dimethylpyrimidine 1-oxide structure. In fact, the reaction of 2-chloromethyl-4,6-dimethylpyrimidine 1-oxide (Xa) with

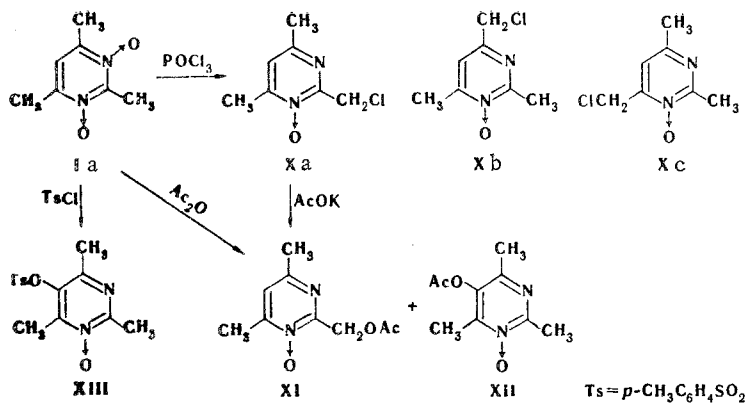
TABLE 1. PMR Spectra\* ( $\delta$ , ppm) of Pyrimidine 1,3-Dioxides and Pyrimidine 1-Oxides (I-XI)

Compound	R <sub>2</sub>	R <sub>4</sub>	R <sub>6</sub>	5-H
Ib	CH <sub>2</sub> † 3,49   CH <sub>3</sub> 1,31	CH <sub>3</sub> 2,51	CH <sub>3</sub> 2,51	7,24
II	CH <sub>3</sub> 2,62	CH 8,41    NOH 12,28	CH <sub>3</sub> 2,39	7,77
IIIa	CH <sub>3</sub> 2,66	CH <sub>2</sub> Br 4,72	CH <sub>3</sub> 2,37	7,84
IIIb	CH <sub>2</sub> † 3,41   CH <sub>3</sub> 1,27	CH <sub>2</sub> Br 4,61	CH <sub>3</sub> 2,49	7,32
IVa	CH <sub>3</sub> 2,66	CH <sub>2</sub> Br 4,74	CH <sub>2</sub> Br 4,74	8,09
IVb	CH <sub>2</sub> † 3,41   CH <sub>3</sub> 1,27	CH <sub>2</sub> Br 4,64	CH <sub>2</sub> Br 4,64	7,66
V	CH <sub>2</sub> Br 4,92	CH <sub>2</sub> Br 4,76	CH <sub>3</sub> 2,41	8,01
VI	CH <sub>2</sub> Br 4,90	CH <sub>2</sub> Br 4,77	CH <sub>2</sub> Br 4,77	8,24
VII	CH <sub>3</sub> 2,86	CH <sub>2</sub> 5,32   OCOCH <sub>3</sub> 2,17	CH <sub>3</sub> 2,52	7,29
VIII	CH <sub>3</sub> 2,82	CH <sub>2</sub> 5,31   OCOCH <sub>3</sub> 2,17	CH <sub>2</sub> 5,31   OCOCH <sub>3</sub> 2,17	7,29
IX	CH <sub>2</sub> 5,66   OCOCH <sub>3</sub> 2,09	CH <sub>2</sub> 5,31   OCOCH <sub>3</sub> 2,19	CH <sub>2</sub> 5,31   OCOCH <sub>3</sub> 2,19	7,40
Xa	CH <sub>2</sub> Cl 4,95	CH <sub>3</sub> 2,49	CH <sub>3</sub> 2,49	7,17
XI	CH <sub>2</sub> 5,24   OCOCH <sub>3</sub> 2,12	CH <sub>3</sub> 2,37  (2,39)	CH <sub>3</sub> 2,39  (2,37)	6,99

\*The PMR spectra of Ib, IIIb, IVb, and VII-Xa in CDCl<sub>3</sub>, of II, IIIa, IVa, V, and VI in (CD<sub>3</sub>)<sub>2</sub>SO, and of XI in CCl<sub>4</sub> were recorded.

†J = 7.5 Hz.

potassium acetate in the presence of 18-crown-6 leads to XI in quantitative yield. It follows from the PMR spectrum of XII (in CCl<sub>4</sub>), in which only singlets of protons of four methyl group [2.23 (6H), 2.30 (3H), and 2.57 ppm (3H)] are observed, that this compound has the 5-acetoxy-2,4,6-trimethylpyrimidine 1-oxide structure.



In the case of the reaction of 1,3-dioxide Ia with tosyl chloride the principal product is 5-tosyloxy-2,4,6-trimethylpyrimidine 1-oxide (XIII). Singlets of protons of four methyl groups (2.12, 2.21, 2.50, and 2.56 ppm) and an AB quartet of four protons of a phenyl group (2H<sub>m</sub> at 7.46 ppm, 2H<sub>o</sub> at 7.87 ppm, J<sub>AB</sub> = 8.5 Hz) are observed in the PMR spectrum of XIII (in CCl<sub>4</sub>).

Thus the direction of the reaction of 1,3-dioxide Ia with electrophilic reagents, which proceeds with splitting out of the oxygen atom of the N-oxide group, depends on the type of

TABLE 2. Characteristics of the Synthesized Compounds (I-XIII)

Compound	mp, °C	UV spectrum, $\lambda_{\max}$ , nm (log $\epsilon$ )	Found, %				Empirical formula	Calculated, %				Yield, %
			C	H	Br	N		C	H	Br	N	
Ib	133-135	258 (4,46), 276 sh (4,09), 333 (2,83)	57,1	7,3	—	16,7	$C_8H_{12}N_2O_2$	57,1	7,2	—	16,7	38
II	220 (dec.)	276 (4,34), 322 (4,15)	45,9	5,2	—	22,6	$C_7H_9N_3O_3$	45,9	5,0	—	22,9	59
IIIa	149-152	266 (4,43), 295 (4,06), 349 (2,99)	35,9	3,8	34,7	11,7	$C_7H_9BrN_2O_2$	36,1	3,9	34,3	12,0	20
IIIb	122-124	267 (4,41), 292 (4,03), 348 (2,94)	38,5	4,5	32,4	11,1	$C_8H_{11}BrN_2O_2$	38,9	4,5	32,3	11,3	28
IVa	145-146	274 (4,42), 304 (4,04), 366 (3,07)	27,1	2,7	51,3	8,7	$C_7H_8Br_2N_2O_2$	27,0	2,6	51,2	9,0	19
IVb	152-154	274 (4,43), 305 (4,05), 364 (3,04)	29,5	3,2	49,0	8,5	$C_8H_{10}Br_2N_2O_2$	29,5	3,1	49,0	8,6	20
V	155-157	276 (4,28), 297 sh (3,96), 374 (2,80)	26,9	2,8	51,4	8,8	$C_7H_8Br_2N_2O_2$	27,0	2,6	51,2	9,0	6
VI	188 (dec.)	282 (4,43), 303 sh (4,13), 382 (2,78)	21,9	1,9	61,1	6,9	$C_7H_7Br_3N_2O_2$	21,5	1,8	61,3	7,2	8
VII · H <sub>2</sub> O	55-56	258 (4,39), 276 sh (4,02), 336 (2,78)	47,0	5,9	—	12,1	$C_9H_{12}N_2O_4 \cdot H_2O$	47,0	6,1	—	12,2	38
VIII	84-86	262 (4,44), 280 sh (4,09), 343 (2,91)	48,5	5,4	—	10,0	$C_{11}H_{14}N_2O_6$	48,9	5,2	—	10,4	87
IX	107-108	267 (4,45), 282 sh (4,18), 366 (2,88)	47,6	4,9	—	8,7	$C_{13}H_{16}N_2O_8$	47,6	4,9	—	8,5	84
Xa	116-118	269 (4,04), 298 sh (3,41)	48,6	5,2	20,5†	15,8	$C_7H_6ClN_2O$	48,7	5,3	20,5†	16,2	22
XI	99-100	264 (4,00), 290 sh (3,50)	55,4	6,3	—	14,5	$C_9H_9N_2O_3$	55,1	6,2	—	14,3	19
XII	105-106	263 (4,03), 306 (3,66)	54,7	6,2	—	14,3	$C_9H_{12}N_2O_3$	55,1	6,2	—	14,3	14
XIII	94-95	267 (4,03), 311 (3,64)	54,6	5,2	10,4‡	9,0	$C_{14}H_{16}N_2O_4S$	54,5	5,2	10,4‡	9,1	40

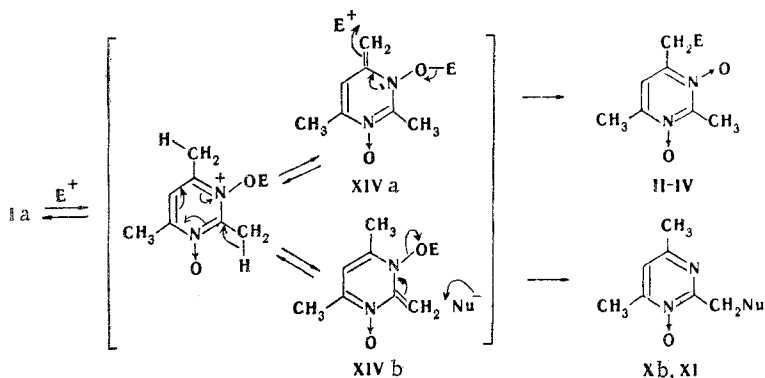
\*The compounds were recrystallized: Ib and Xa-XIII from hexane, II from alcohol, IIIa, IVa, V, and VI from chloroform-ethyl acetate, IIb from ether, and VII-IX from ethyl acetate.

†Shoulders are indicated by sh.

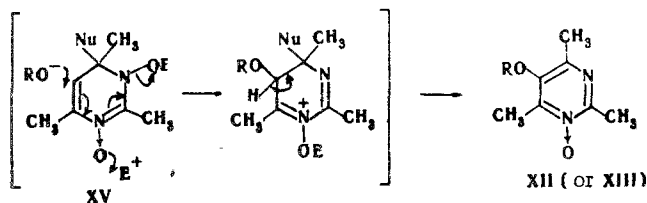
‡The experimentally found and calculated values for Cl (Xa) and S (XIII) are presented.

reagent used: A halogen is introduced in the methyl group in the 2 position of the heteroring in the reaction with phosphorus oxychloride, acetoxy groups are introduced in the methyl group in the 2 position and in the 5 position of the heteroring in the reaction with acetic anhydride, and a tosyl group is introduced in the 5 position of the heteroring in the case of tosyl chloride.

It might be assumed [4] that the reaction of 1,3-dioxides Ia,b with electrophilic reagents, which proceeds with both the retention and loss of the oxygen atom of an N-oxide group, passes through the intermediate formation of methylidenepyrimidines XIVa,b. However, 4(or 6)-methylidenepyrimidine XIVa is evidently less deactivated relative to electrophilic attack than 2-methylidenepyrimidine XIVb. Pyrimidine 1,3-dioxides II-IV with substituted alkyl groups in the 4 position of the heteroring are primarily formed in the bromination and nitrosation of 1,3-dioxides Ia, b, whereas 2-halomethyl- and 2-acetoxymethylpyrimidine 1-oxides Xa and XI are primarily formed in the reaction with phosphorus oxychloride and acetic anhydride. In the latter case 2-methylidenepyrimidine XIVb undergoes nucleophilic attack by the chloride ion or the acetoxy anion rather than electrophilic attack.



Several schemes have been proposed to explain the formation of  $\beta$ -hydroxy derivatives in the azine series [4]; in analogy with these schemes it may be proposed that the formation of  $\beta$ -hydroxy derivatives XII and XIII may proceed through both intermediate 1,6-dihydropyrimidines XV and methylidenepyrimidines XIV with subsequent incorporation of an acyloxy group in the  $\beta$  position of the pyrimidine ring via a type of allylic substitution of sigmatropic shift of this group from the nitrogen atom.



The tendency to form  $\beta$ -hydroxy derivatives in the reaction of 1,3-dioxides or pyrimidines with acylating reagents is possibly associated with additional activation of the  $\beta$  position of the heteroring by a second N-oxide group. The possibility that activation of the  $\beta$  position may be intensified by the addition of the electrophile to the second N-oxide group of the dihydropyrimidine or methylidenepyrimidine is also not excluded.

#### EXPERIMENTAL

The IR spectra were recorded with a UR-20 spectrometer. The UV spectra of solutions of the compounds in alcohol were recorded with a Specord UV-vis spectrophotometer. The PMR spectra were recorded with a Varian A-56-60A spectrometer with hexamethyldisiloxane as the internal standard. The experimental dipole moments were calculated by the Guggenheim-Smith method [11]. The dielectric permeabilities of solutions in benzene were determined at 25°C by the heterodyne method with a Tangens apparatus, and the refractive indexes were determined with an Abbe refractometer. The theoretical dipole moments for Xa-c were calculated via a  $\mu_{N=O}$  1.62 D [12],  $\mu_{Csp^2-C1}$  1.87 D [13],  $\mu_{CH_3}$  0.37 D,  $\mu_{Csp^2-Csp^2}$  0.70 D [13]. 18-Crown-6 was produced in the chemical pilot plant of the Novosibirsk Institute of Organic Chemistry of the Siberian Branch of the Academy of Sciences of the USSR was used in the research.

4,6-Dimethyl-2-ethylpyrimidine 1,3-Dioxide (Ib). A 3.0-g (0.023 mole) sample of N-(4-oximido-2-pentyl)hydroxylamine [14] was added with stirring at a rate proportionate to the rate at which it dissolved to a solution of 1.44 g (0.025 mole) of propionaldehyde in 15 ml of alcohol. After 2 h, the solvent was evaporated, dioxane was added, and the mixture was evaporated again. The residue was dissolved in 200 ml of dioxane, and a suspension of 17.8 g (0.205 mole) of active manganese dioxide in 50 ml of dioxane [2] was added with stirring. After 1 h, the suspension was filtered, the filtrate was evaporated, the residue was triturated in ether, and the precipitated Ib was removed by filtration. The yield was 1.48 g.

4-Oximidomethyl-2,6-dimethylpyrimidine 1,3-Dioxide (II). A 0.1-g (0.65 mmole) sample of Ia was added at 0°C to a solution of 0.07 g (1.92 mmole) of HCl in 3 ml of anhydrous alcohol, after which a solution of 0.1 g (1.33 mmole) of ethyl nitrite in 3 ml of anhydrous alcohol was added, and the mixture was stirred at 0°C for 2 h. The precipitated II was removed by filtration and washed with alcohol. The yield was 0.07 g.

Compound II was similarly obtained in 34% yield by nitrosation of Ia with amyl nitrite in methanol.

Bromoethyl Derivatives of Pyrimidine 1,3-Dioxides (III-VI). A solution of 0.27 g (1.69 mmole) of bromine in 3 ml of chloroform was added with stirring at a rate proportionate to the rate at which the solution became colorless to a solution of 0.2 g (1.3 mmole) of Ia in 5 ml of chloroform, after which the mixture was diluted with chloroform and washed with an 8% solution of sodium carbonate and with water saturated with sodium chloride. The chloroform solution was dried over magnesium sulfate and evaporated, and the residue was chromatographed with a column filled with silica gel (elution with methylene chloride) to give 40 mg of VI, 23 mg of V, 77 mg of IVa, and 60 mg of IIIa, respectively.

Compounds IVb and IIIb, respectively, were similarly isolated by bromination of 1,3-dioxide Ib but in the presence of a catalytic amount of concentrated sulfuric acid and by chromatography of the reaction with a column filled with silica gel (elution with chloroform).

Acetoxymethylpyrimidine 1,3-Dioxides (VII-IX). A catalytic amount of 18-crown-6 was added to a suspension of 0.29 g (3 mmole) of potassium acetate in 10 ml of acetonitrile. After 15 min, a 0.35 g (1.5 mmole) sample of IIIa was added in small portions, and the suspension was stirred for 5 h (with chromatographic monitoring). The precipitate was removed by filtration, the solvent was evaporated, and the residue was chromatographed with a column filled with silica gel (elution with chloroform). This procedure yielded VII as an oil, which crystallized when it was triturated in ether. The yield was 0.13 g.

Compounds VIII and IX were similarly obtained using an appropriate excess of potassium acetate and reaction times of 3 and 2 h, respectively. For the isolation of VIII and IX, the solvent was evaporated, and the residue was triturated in ether.

2-Chloromethyl-4,6-dimethylpyrimidine 1-Oxide (Xa). A solution of 0.71 ml (7.8 mmole) of phosphorus oxychloride in 10 ml of chloroform was added to a solution of 1.0 g (6.5 mmole) of Ia in 10 ml of chloroform, and the mixture was refluxed for 3 h (with chromatographic monitoring). It was then diluted with chloroform and washed with an 8% solution of sodium bicarbonate and water saturated with sodium chloride. The chloroform solution was dried over magnesium sulfate and evaporated, and the residue was chromatographed with a column filled with silica gel (elution with chloroform) to give 0.25 g of Xa.

Acetoxypyrimidine 1-Oxide (XI, XII). A 1.67-g (16.4 mmole) sample of acetic anhydride was added to a solution of 0.50 g (3.24 mmole) of Ia in 5 ml of chloroform, and the mixture was refluxed for 4 h (with chromatographic monitoring). The solvent was evaporated, and the residue was chromatographed with a column filled with silica gel (elution with chloroform) to give 0.09 g of XII and 0.12 g of XI, respectively.

Compound XI was obtained by the reaction of Xa with potassium acetate by the method presented above. After 6 days, the precipitate was removed by filtration, the solvent was evaporated, the residue was triturated in ether, and the precipitated XI was removed by filtration. The product was obtained in 99% yield.

5-Tosyloxy-2,4,6-trimethylpyrimidine 1-Oxide (XIII). A solution of 0.31 g (2.0 mmole) of Ia and 0.57 g (3.0 mmole) of tosyl chloride in 3 ml of chloroform was refluxed for 3 h (with chromatographic monitoring), after which the mixture was diluted with chloroform and washed with 8% sodium bicarbonate solution and water. The chloroform solution was dried over magnesium sulfate and evaporated, and the residue was treated with ether. The XIII (0.25 g) was removed by filtration.

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