mechanism, since the acidic nature of the system at elevated temperatures can catalyze olefin isomerization. Data indicating that olefin isomerization does occur are shown in Table VI. These experiments were

TABLE VI
OLEFIN ISOMERIZATION

Component	Reagent, %	Product, %
4-Methyl-1-pentene	98.3	95.0
4-Methyl-*cis*-2-pentene	1.7	2.4
4-Methyl-*trans*-2-pentene	...	<0.1
Unidentified (not C_6)[a]	...	2.5

[a] Probably from decomposing zinc salt.

made by passing a known olefin sample at a high space velocity over decomposing zinc O,O-diisopropylphosphorodithioate at 155° and analyzing the product by gas chromatography. These results are in contrast with those of Ashford, *et al.*,[7] who contacted 99.1% 4-methyl-1-pentene with the decomposition residue at 60° for 3 hr without olefin isomerization. The difference in rate of isomerization at this unrealistically low decomposition temperature, 95° below the decomposition temperature employed by them, could account for their observation. However, carbonium ion stability may have an influence on the rate of the isomerization step. Also, the alkyl group could undergo rearrangement at this step. It is significant, however, that predominantly *n*-propyl mercaptan was formed from the decomposition of the corresponding zinc *n*-propyl salt in this work, and *n*-butyl mercaptan was formed from the zinc *n*-butyl salt in the work of Luther and Sinha.[6] Finally, it is also significant that Perry[15] found from the decomposition of zinc O,O-di(4-methyl-2-pentyl)phosphorodithioate, 96% of 4-methyl-1-pentene and 4-methyl-2-pentene, the products expected from *cis* elimination, and only 4% of 2-methyl-1-pentene and 2-methyl-2-pentene, the products expected *via* a rearrangement of a carbonium ion.

Conclusions

The dependence of the thermal stability of metal O,O-dialkylphosphorodithioates on variations of both the alkyl group structure and the size of the metal ion suggests that the initial reaction involves an isomerization followed by an intramolecular (*cis*) elimination of olefin.

Acknowledgment.—The authors are especially indebted to Dr. H. Myers for many helpful discussions and to our Analytical Division for assistance in the analyses.

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Bis(phenoxy)phosphinyl as *N*-Blocking Group in Amino Sugar Nucleoside Synthesis[1]

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3,4,6-Tri-*O*-acetyl-2-deoxy-2-*N*-[bis(phenoxy)phosphinyl]amino-α-D-glucosyl bromide (**1**) was condensed with 6-benzamido-9-chloromercuripurine to give the substituted nucleoside (**2a**) and this on *N*-debenzoylation and treatment with ammoniacal benzyl alcohol yielded 9-{2-*N*-[bis(benzyloxy)phosphinyl]amino-2-deoxy-β-D-glucopyranosyl}adenine (**2c**) which was hydrogenated, under mild conditions, to 9-(2-amino-2-deoxy-β-D-glucopyranosyl)adenine (**3**). Condensation of **1** with bis(trimethylsilyl)cytosine gave the substituted nucleoside (**4b**), which upon treatment with ammoniacal benzyl alcohol and mild hydrogenation yielded 1-(2-amino-2-deoxy-β-D-glucopyranosyl)cytosine (**5**).

The nucleosides of amino sugars are usually synthesized by condensing a blocked amino sugar halide with a derivative of a purine or pyrimidine according to the Fischer–Helferich[2] method, as modified by Davoll and Lowy,[3] or Hilbert and Johnson.[4] In the synthesis of nucleosides of 2-amino-2-deoxyaldoses considerable difficulty has been experienced in the selection of an *N*-blocking group which can be easily removed, especially if the neighboring hydroxyl group is configurationally *trans*. In addition, the *N*-blocking group on C-2 should not be strongly participating as otherwise it may interfere with the reactivity of the glycosyl halide in nucleoside formation. In some few cases the *N*-acetyl has been used successfully[5-7] for such syntheses but has not always been removed from the reaction product. Other blocking groups employed have been the *N*-benzyloxycarbonyl,[7] *N*-methoxycarbonyl,[7] and from this laboratory, the *N*-2,4-dinitrophenyl,[8] *N*-benzylsulfonyl,[9] and the *N*-trifluoroacetyl[10] groups have been used for this purpose. The 2,4-dinitrophenyl[8] group gave both anomeric forms of a purine nucleoside, since it shows no tendency to participate at C-1.

We now wish to report the use of bis(phenoxy)-phosphinyl, $PO(OC_6H_5)_2$, as an *N*-blocking group in the synthesis of a purine nucleoside, 9-(2-amino-2-deoxy-β-D-glucopyranosyl)adenine (**3**), and of a pyrimidine nucleoside, 1-(2-amino-2-deoxy-β-D-glucopyranosyl)cytosine (**5**).

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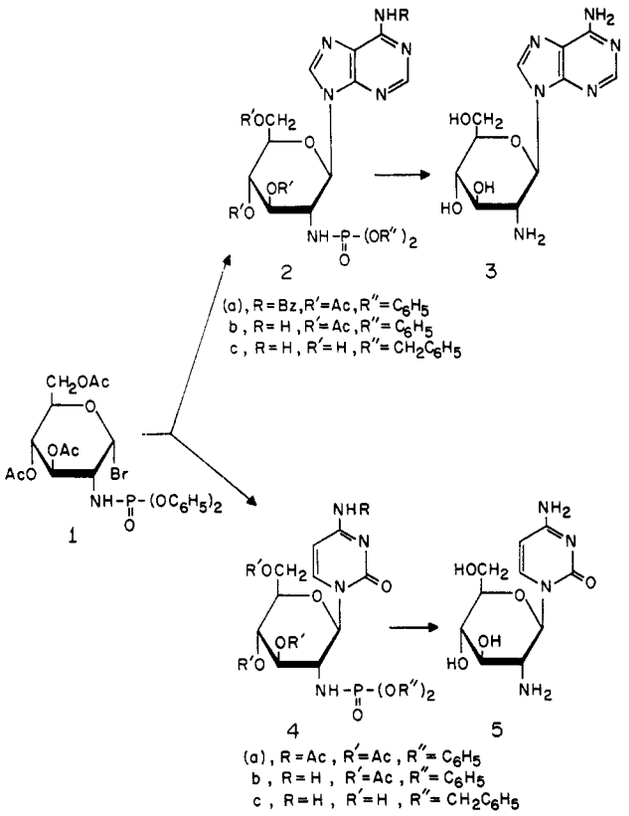
SCHEME I



The *N*-bis(alkoxy)phosphinyl, NH-PO(OR)$_2$, derivatives of amino acids and amino acid esters have been prepared.[11] Methyl 2-amino-2-deoxy-β-D-glucopyranoside was synthesized[12] using bis(phenoxy)phosphinyl as an *N*-protecting group which was subsequently removed by hydrogenation under high pressure in the presence of platinum oxide. Zervas and Konstas[12] also replaced the phenoxy groups in the above derivative by benzyloxy groups with ammoniacal benzyl alcohol. The resulting *N*-derivative could be removed by hydrogenation with a palladium catalyst at a much lower pressure.

3,4,6-Tri-*O*-acetyl-2-deoxy-2-*N*-[bis(phenoxy)phosphinyl]amino-α-D-glucosyl bromide (**1**) was prepared according to Zervas and Konstas[12] and was refluxed in toluene with 6-benzamido-9-chloromercuripurine in the general procedure of Davoll and Lowy[3a] to give in 56% yield, 6-benzamido-9-{3,4,6-tri-*O*-acetyl-2-deoxy-2-*N*-[bis(phenoxy)phosphinyl]amino-β-D-glucosyl}-purine (**2a**). *N*-Debenzoylation at C-6 with picric acid and removal of the picrate anion[13] with an ion-exchange resin yielded 9-{3,4,6-tri-*O*-acetyl-2-deoxy-2-*N*-[bis(phenoxy)phosphinyl]amino-β-D-glucosyl}adenine (**2b**). The bis(phenoxy)phosphinyl group could not be readily removed by hydrogenation, even at higher pressures. Compound **2b** was therefore de-*O*-acetylated and the bis(phenoxy)phosphinyl group was converted to bis(benzyloxy)phosphinyl, PO(OCH$_2$C$_6$H$_5$)$_2$, by prolonged treatment at room temperature with ammoniacal benzyl alcohol.[12] The product, 9-{2-*N*-[bis(benzyloxy)phosphinyl]amino-2-deoxy-β-D-glucopyranosyl}-adenine (**2c**), obtained in good yield, was readily deblocked by hydrogenation at mild hydrogen pressure in the presence of palladium–charcoal catalyst, to give 9-(2-amino-2-deoxy-β-D-glucopyranosyl)adenine (**3**). The desired nucleoside (**3**) had physical constants identifiable with those for the compound prepared previously in this laboratory.[8]

The same *N*-blocking group was used in the preparation of a pyrimidine nucleoside of 2-amino-2-deoxy-D-glucose. Preliminary attempts to prepare 1-(2-amino-2-deoxy β-D-glucopyranosyl)cystosine (**5**) using the same general procedure[3a] as above, by condensing *N*-acetylcytosinemercury[14] with **1**, gave 4-*N*-acetyl-1-{3,4,6-tri-*O*-acetyl-2-deoxy-2-*N*-[bis(phenoxy)phosphinyl]amino-β-D-glucopyranosyl}cytosine (**4a**) in poor yield (8–9%). Use of different reflux times (1–5 hr) or different solvents failed to increase the yield. However, use of bis(trimethylsilyl)cytosine[15] in place of *N*-acetylcytosinemercury, gave 1-{3,4,6-tri-*O*-acetyl-2-deoxy-2-*N*-[bis(phenoxy)phosphinyl]amino-β-D-glucosyl}cytosine (**4b**) in 75% yield. 1-{2-*N*-[Bis(benzyloxy)phosphinyl]amino-2-deoxy-β-D-glucopyranosyl}cytosine (**4c**) was prepared in 83% yield from **4b** as described for the adenine nucleoside. Deblocking of **4c** to give pure 1-(2-amino-2-deoxy-β-D-glucopyranosyl)cytosine (**5**) was achieved in 33% yield using 92% aqueous dioxane or 90% aqueous methanol as solvent and palladium–charcoal catalyst under mild hydrogen pressure (Scheme I). Using a more polar solvent led to increased production of a second product, presumably one where the cytosine moiety was reduced.

The bis(phenoxy)phosphinyl group has thus proved to be a suitable *N*-protecting group in nucleoside synthesis with 2-amino-2-deoxy-D-glucose. Over-all yields of 9-(2-amino-2-deoxy-β-D-glucopyranosyl)adenine (**3**) and 1-(2-amino-2-deoxy-β-D-glucopyranosyl)-cytosine (**5**) from 3,4,6-tri-*O*-acetyl-2-deoxy-*N*-[bis(phenoxy)phosphinyl]amino-α-D-glucosyl bromide (**1**) were 9.3 and 20.5%, respectively.

Experimental Section[16]

9-{3,4,6-Tri-*O*-acetyl-2-deoxy-2-*N*-[bis(phenoxy)phosphinyl]-amino-β-D-glucosyl}adenine (2b).—A mixture of 6-benzamido-9-chloromercuripurine[3a] (27.0 g), cadmium carbonate (5.2 g), and Celite (7.3 g) in toluene (400 ml) was azeotropically dried by distillation of approximately 125 ml of the toluene. To the hot suspension was added, with stirring, 3,4,6-tri-*O*-acetyl-2-deoxy-2-*N*-[bis(phenoxy)phosphinyl]amino-α-D-glucosyl bromide[12] (**1**, 12.5 g) and the mixture was refluxed for 5 hr and then kept at room temperature overnight. Solids were filtered and triturated several times with chloroform (total 500 ml). The combined filtrate and washings were evaporated to dryness under diminished

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pressure and redissolved in chloroform. The solution was washed with 30% aqueous potassium iodide and water, dried over anhydrous magnesium sulfate, and concentrated to a small volume. A solid separated after the addition of a small quantity of ether. The yield of crude 6-benzamido-9-{3,4,6-tri-*O*-acetyl-2-deoxy-2-*N*-[bis(phenoxy)phosphinyl]amino-β-D-glucosyl}purine (**2a**) was 8.9 g.

The product was dissolved in 90 ml of warm ethanol. Picric acid (3 g) in 45 ml of ethanol was added and the mixture was refluxed for 1 hr. A yellow solid formed upon cooling the solution and was washed with ether. This picrate was then dissolved in 90% aqueous acetone and stirred with a slight excess of Dowex AG 1-X2 (CO_3^{2-}) resin. The resin was removed by filtration and washed well with acetone. The combined filtrate and washings were concentrated under reduced pressure and yielded 3.5 g (or 25.5% based on **1**) of 9-{3,4,6-tri-*O*-acetyl-2-deoxy-2-N-[bis(phenoxy)phosphinyl]amino-β-D-glucosyl}adenine (**2b**). Compound **2b**, after three recrystallizations from methanol–ether had mp 150–152° with resolidification and remelting at 248–249°, and $[\alpha]^{25}D$ −15 ± 0.5° (*c* 3.2, methanol).

Anal. Calcd for $C_{29}H_{31}N_6O_{10}P$: C, 53.21; H, 4.77; N, 12.84. Found: C, 53.46; H, 4.59; N, 13.02.

9-{2-*N*-[Bis(benzyloxy)phosphinyl]amino-2-deoxy-β-D-glucopyranosyl}adenine (2c).—9-{3,4,6-Tri-*O*-acetyl-2-deoxy-2-*N*-[bis(phenoxy)phosphinyl]amino-β-D-glucosyl}adenine (**2b**, 1.5 g) was dissolved in benzyl alcohol (50 ml) nearly saturated with ammonia at 0°. The solution was kept for 4 days at room temperature, after which it was poured into an excess (500 ml) of ether. The flocculent precipitate was washed several times with ether and was reprecipitated three times by pouring a concentrated methanol solution into an excess of ether. The yield, after crystallization from 95% ethanol, was 1.1 g (86%), mp 195–197° dec with softening at 140–160° and $[\alpha]^{25}D$ −15 ± 1° (*c* 1, methanol).

Anal. Calcd for $C_{25}H_{29}N_6O_7P$: C, 53.95; H, 5.25; N, 15.10. Found: C, 53.55; H, 5.56; N, 15.02.

9-(2-Amino-2-deoxy-β-D-glucopyranosyl)adenine (3).—9-{2-*N*-[Bis(benzyloxy)phosphinyl]amino-2-deoxy-β-D-glucopyranosyl}-adenine (**2c**, 0.2 g) was dissolved in 95% methanol and treated with palladium–charcoal catalyst (0.12 g of 10% catalyst) in the presence of hydrogen (40 psi) for 5 hr at room temperature. The mixture was filtered and the catalyst was washed with 100 ml of water. The combined filtrate and washings were concentrated to 25 ml and passed through a 5-ml bed of Dowex 1 (CO_3^{2-}) resin. The resin was washed with 100 ml of water and the eluate and washings were evaporated to dryness to yield 0.045 g (42%) of crude nucleoside, mp 184–188° dec. After two recrystallizations from absolute ethanol and careful drying under diminished pressure, the crystalline material had mp 186–189° dec and $[\alpha]^{25}D$ −17 ± 2° (*c* 0.7, water) [lit.[7] mp 186–188° dec and $[\alpha]^{23}D$ −17 ± 2° (*c* 0.2, water)].

Anal. Calcd for $C_{11}H_{16}N_6O_4$: C, 44.59; H, 5.44; N, 28.37. Found: C, 44.20; H, 5.06; N, 28.60.

1-{3,4,6-Tri-*O*-acetyl-2-deoxy-2-*N*-[bis(phenoxy)phosphinyl]-amino-β-D-glucosyl}cytosine (4b). Method A. With *N*-Acetylcytosinemercury.—Attempts to prepare 4-*N*-acetyl-1-{3,4,6-tri-*O*-acetyl-2-dexoy-2-*N*-[bis(phenoxy)phosphinyl]amino-β-D-glucosyl}cytosine (**4a**) by refluxing **1** with *N*-acetylcytosinemercury[14] were made using different reflux times (1–5 hr) in either benzene or toluene. The glycosyl bromide (**1**) was added in two equal portions, the second after 0.5 to 1 hr. The yields of **4a** in all trials were similar. A typical condensation follows.

A mixture of *N*-acetylcytosinemercury[14] (0.44 g) in toluene (30 ml) was azeotropically dried by distillation of approximately one-fourth of the solvent. 3,4,6-Tri-*O*-acetyl-2-deoxy-2-*N*-[bis(phenoxy)phosphinyl]amino-α-D-glucosyl bromide (**1**, 0.75 g) was added with stirring, and the mixture was refluxed for 1 hr. An equal quantity (0.75 g) of the bromide (2 equiv total) was again added and the mixture was refluxed for an additional 1–4 hr. The reaction mixture was cooled and treated with 100 ml of petroleum ether (bp 30–60°), and the precipitate so formed was extracted with chloroform. The chloroform solution was washed with 30% aqueous potassium iodide and water, then dried over sodium sulfate and evaporated to dryness. Attempts to isolate the desired nucleoside **4b** by treatment of the residue with picric acid in boiling ethanol followed by depicration with ion-exchange resin, as described above, resulted in a mixture of products in a yield of 0.10–0.12 g from which **4b** was isolated in approximately 60% yield or 8–9% yield based on the *N*-acetylcytosinemercury used.

Method B. With Bis(trimethylsilyl)cytosine.—3,4,6-Tri-*O*-acetyl-2-deoxy-2-*N*-[bis(phenoxy)phosphinyl]amino-α-D-glucosyl bromide (**1**, 3.0 g) was added over a period of 15 min to a stirred and gently heated (short of reflux) mixture of 1.20 g of bis(trimethylsilyl)cytosine[15] in 130 ml of dry benzene. The mixture was then refluxed for 2 hr. The white precipitate which formed was filtered and added to 70% aqueous ethanol containing 1.0 g of sodium hydrogen carbonate. After heating for 15 min at 60°, the solution was evaporated to near dryness under diminished pressure. A chloroform extract of the mixture was dried over sodium sulfate and evaporated to yield 2.37 g (75%) of crude product, mp 260° dec with softening at 155–170°, and $[\alpha]^{22}D$ +12 ± 1° (*c* 3.2, methanol).

The product formed a well-defined, yellow, crystalline picrate, mp 144–146° and $[\alpha]^{21}D$ +51 ± 2° (*c* 1, acetone).

Anal. Calcd for $C_{34}H_{34}N_7O_{18}P$: C, 47.50; H, 3.99; N, 11.41. Found: C, 47.11; H, 3.96; N, 11.60.

Depication in the previously described manner gave material with mp 260° dec with softening at 153–165° and $[\alpha]^{20}D$ +12 ± 0.5° (*c* 3.5, methanol).

Anal. Calcd for $C_{28}H_{31}N_4O_{11}P$: C, 53.33; H, 4.96; N, 8.89. Found: C, 53.46; H, 4.97; N, 8.91.

1-{2-*N*-[Bis(benzyloxy)phosphinyl]amino-2-deoxy-β-D-glucopyranosyl}cytosine (4c).—1-{3,4,6-Tri-*O*-acetyl-2-deoxy-2-*N*-[bis(phenoxy)phosphinyl]amino-β-D-glucopyranosyl}cytosine (**4b**, 2.7 g) was dissolved in 50 ml of benzyl alcohol nearly saturated with ammonia at 0°, and the solution was kept for 48 hr at room temperature, after which it was heated for 15 min at 60° under water aspirator vacuum to remove excess ammonia. The precipitate obtained on pouring the solution into 500 ml of ether was dissolved in 20 ml of methanol and reprecipitated in ether. The yield was 1.9 g (83%), mp 212–215° dec. After two crystallizations from 95% methanol, the product had mp 213–215° dec and $[\alpha]^{22}D$ +44 ± 2° (*c* 0.5, water).

Anal. Calcd for $C_{24}H_{29}N_4O_8P$: C, 54.13; H, 5.49; N, 10.52. Found: C, 54.33; H, 5.61; N, 10.24.

1-(2-Amino-2-deoxy-β-D-glucopyranosyl)cytosine (5).—1-{2-*N*-[bis(benzyloxy)phosphinyl]amino-2-deoxy-β-D-glucopyranosyl}-cytosine (**4c**, 1.0 g) was dissolved in 92% aqueous dioxane and treated with 0.6 g of 10% palladium–charcoal catalyst in the presence of hydrogen (30 psi) for 4 hr at room temperature. The solution was filtered and the catalyst was washed with 120 ml of water. The combined filtrate and washings were concentrated to 20 ml and passed through 15 ml of Dowex 1 (CO_3^{2-}) ion-exchange resin. The resin was then washed with 100 ml of water and the combined eluate and washings were evaporated to yield 0.28 g (55%) of hydrogenated material. Thin layer chromatography, using methanol developer and sulfuric acid indicator, showed a major spot at R_f 0.3 (A) and two minor ones at R_f 0.6 (B) and R_f 0.8. Use of a more polar solvent (higher percentage of water) for hydrogenation yielded more of substance B. A portion of the hydrogenated product (0.20 g) was separated by thin layer chromatography (five 200 × 200 × 1 mm silica gel G plates, methanol as developer and ultraviolet indication) to give 0.12 g of A (60%), mp 215.5–219° dec, and 0.025 g of B. Substance A (**5**) was crystallized from methanol–ether–chloroform and dried: mp 217–219° dec with swelling at 170–190°, $[\alpha]^{25}D$ +33 ± 1° (*c* 1.2, water), $\lambda_{max}^{0.1\ N\ HCl}$ 212 and 275 mμ, $[\alpha]^{25}D$ +51.5 ± 2° (as sulfate, *c* 0.5, water) [lit.[7] $\lambda_{max}^{0.1\ N\ HCl}$ 210 and 274.5 mμ, $[\alpha]^{25}D$ +50° (*c* 1, water) for the sulfate dihydrate].

Anal. Calcd for $C_{10}H_{16}N_4O_5$: C, 44.12; H, 5.88; N, 20.60. Found: C, 43.97; H, 5.81; N, 20.60.

Owing to the small amount available, substance B was not further investigated but its ultraviolet spectrum (0.1 *N* hydrochloric acid) showed one band at 208 mμ with the band at 275 mμ absent.

Registry No.—**2b**, 7482-55-5; **2c**, 7482-56-6; **3**, 3768-20-5; **4b**, 7491-77-2; picrate, 7540-82-1; **4c**, 7482-57-7; **5**, 7474-51-3. **2a**, 7539-96-0.

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