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TWO CRYSTALLINE PENTOFURANOSYL BROMIDES: TRI-*O*-(*p*-NITROBENZOYL)-β-D-RIBOFURANOSYL BROMIDE AND TRI-*O*-(*p*-NITROBENZOYL)-α,β-D-XYLOFURANOSYL BROMIDE*

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ABSTRACT

Methyl α,β-D-ribofuranoside was *p*-nitrobenzoylated to give methyl tri-*O*-(*p*-nitrobenzoyl)-β-D-ribofuranoside (**2**), and this was treated with HBr in acetic acid to give tri-*O*-(*p*-nitrobenzoyl)-β-D-ribofuranosyl bromide (**3**). Bromide **3** could be converted into 2,5-anhydro-3,4,6-tri-*O*-(*p*-nitrobenzoyl)-D-allononitrile (**4**) with $Hg(CN)_2$, or hydrolyzed to 2,3,5-tri-*O*-(*p*-nitrobenzoyl)-D-ribose (**5**). On *p*-nitrobenzoylation, **5** gave tetra-*O*-(*p*-nitrobenzoyl)-β-D-ribofuranose (**6**). The synthesis of tri-*O*-(*p*-nitrobenzoyl)-α,β-D-xylofuranosyl bromide (**11**) started with methyl 3,5-*O*-isopropylidene-β-D-xylofuranoside (**7**), which was *p*-nitrobenzoylated to give ester **8**; this was then hydrolyzed, and the product *p*-nitrobenzoylated to give methyl tri-*O*-(*p*-nitrobenzoyl)-β-D-xylofuranoside (**10**) which, on treatment with HBr in CH_2Cl_2, afforded the desired bromide (**11**). Nucleophilic replacement with $Hg(CN)_2$ afforded 2,5-anhydro-3,4,6-tri-*O*-(*p*-nitrobenzoyl)-D-gulononitrile (**12**).

INTRODUCTION

Pentofuranosyl halides are valuable, synthetic intermediates for the preparation of nucleosides and nucleoside analogs. Crystalline halides are preferred, as they can be purified and stored. This paper describes the synthesis of two crystalline pentofuranosyl bromides, namely, tri-*O*-(*p*-nitrobenzoyl)-β-D-ribofuranosyl bromide and tri-*O*-(*p*-nitrobenzoyl)-D-xylofuranosyl bromide. The latter compound is the first crystalline xylofuranosyl halide to be prepared.

At present, the most widely used D-ribofuranosyl halides are syrups; they are tri-*O*-benzoyl-D-ribofuranosyl chloride[1] and tri-*O*-benzoyl-D-ribofuranosyl bromide[2]. Both are obtained from a crystalline precursor, 1-*O*-acetyl-2,3,5-tri-*O*-benzoylβ-D-ribofuranose[2]. Attempts to prepare crystalline *p*-substituted-benzoyl D-ribofuranosyl

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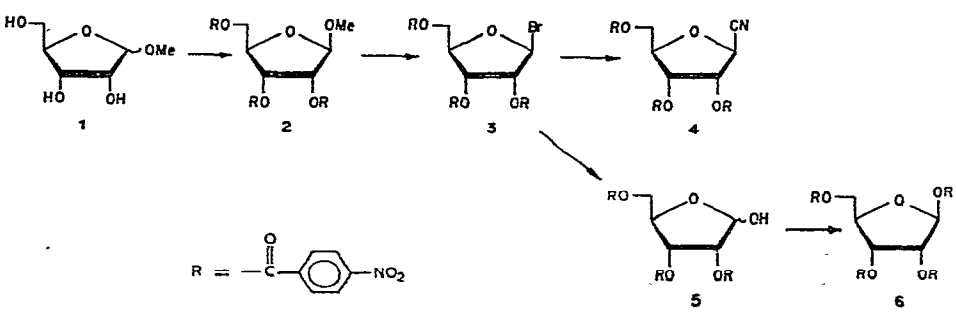
halides, such as the *p*-toluoyl and *p*-anisoyl[3] derivatives, resulted in the formation of syrupy products. Two crystalline D-ribofuranosyl halides have recently been described: tri-*O*-(*p*-chlorobenzoyl)-β-D-ribofuranosyl bromide was obtained by Szeker and Bardos[4] in five steps from methyl D-ribofuranoside, and 2,3-*O*-isopropylidene-5-*O*-trityl-D-ribofuranosyl chloride was prepared by Fox *et al.*[5] for use in reactions where neighboring-group participation is not wanted. Lately, Earl and Townsend[6] have succeeded in crystallizing Todd's tri-*O*-acetyl-β-D-ribofuranosyl chloride[7].

The first D-xylofuranosyl halide obtained was a chloride, prepared by Baker and Schaub[8] from 2,3,5-tri-*O*-benzoyl-D-xylofuranose and hydrogen chloride. These authors also described tri-*O*-benzoyl-D-xylofuranosyl bromide, later prepared from 2,3,5-tri-*O*-benzoyl-D-xylofuranose by Fox and co-workers[9], who also obtained Baker's chloride from 1-*O*-acetyl-2,3,5-tri-*O*-benzoyl-D-xylofuranose. A number of D-xylofuranosyl halides having good leaving-groups, such as the tosyloxy and mesyloxy groups, on C-3 were prepared by Baker and co-workers[10,11]. Finally, tri-*O*-acetyl-D-xylofuranosyl bromide[12] and chloride[13] were prepared from tetra-*O*-acetyl-D-xylofuranose. All of these D-xylofuranosyl bromides and chlorides, as well as two D-xylofuranosyl fluorides recently described[14,15], were syrups.

DISCUSSION

Tri-*O*-(*p*-nitrobenzoyl)-β-D-ribofuranosyl bromide (**3**), described herein, was prepared in two steps from methyl α,β-D-ribofuranoside (**1**) which was *p*-nitrobenzoylated to give crystalline methyl tri-*O*-(*p*-nitrobenzoyl)-β-D-ribofuranoside (**2**). The n.m.r. spectrum of compound **2** showed a multiplet at δ 8.1 which was assigned to the aromatic protons, and a two-proton multiplet at δ 5.7 assigned to H-2 and H-3. A one-proton singlet at δ 5.2, assigned to the anomeric proton, confirmed that compound **2** existed in the β-D configuration. The signals for H-4, H-5, and H-5′ appeared as a complicated multiplet at δ ~4.7, and the methyl proton appeared as a singlet at δ 3.4.

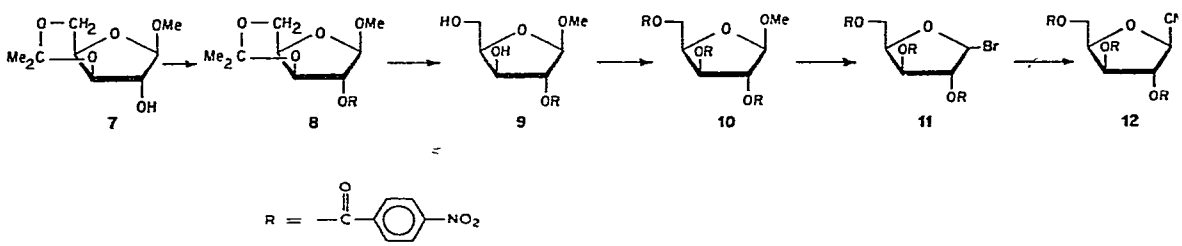
Treatment of compound **2** with hydrogen bromide in acetic acid afforded crystalline 2,3,5-tri-*O*-(*p*-nitrobenzoyl)-β-D-ribofuranosyl bromide (**3**). The n.m.r. spectrum of compound **3** showed the aromatic protons at δ 8.2, and the anomeric



proton as a singlet at δ 6.7, indicative of the β-D confiuration. A two-proton multiplet at δ 6.3, assigned to H-2 and H-3, was followed by a three-proton multiplet at $\delta \sim 5.0$ which was assigned to H-4, H-5, and H-5′. Bromide **3** is a stable compound that can be kept for over six months at 0°. To test its reactivity towards nucleophiles, it was converted into 2,5-anhydro-3,4,6-tri-*O*-(*p*-nitrobenzoyl)-D-allononitrile (**4**) by the action of mercuric cyanide. Compound **4** showed a doublet at δ 5.3 (J 3 Hz) for the proton attached to C-2, which agrees with structure **4**, but does not preclude a *cis* arrangement between H-2 and H-3. To verify the *allo* configuration of nitrile **4**, it was hydrolyzed to the known 2,5-anhydro-D-allonic acid, which was characterized by the formation of 8-β-D-ribofuranosyladenine[16]. In addition, compound **3** was hydrolyzed to 2,3,5-tri-*O*-(*p*-nitrobenzoyl)-D-ribose (**5**) which, on *p*-nitrobenzoylation, gave tetra-*O*-(*p*-nitrobenzoyl)-β-D-ribofuranose (**6**).

The synthesis of tri-*O*-(*p*-nitrobenzoyl)-D-xylofuranosyl bromide (**11**) started with methyl 3,5-*O*-isopropylidene-β-D-xylofuranoside (**7**), a compound first prepared by Baker *et al.*[17]. Compound **7** was *p*-nitrobenzoylated to give crystalline methyl 3,5-*O*-isopropylidene-2-*O*-(*p*-nitrobenzoyl)-β-D-xylofuranoside (**8**). The n.m.r. spectrum of compound **8** showed the aromatic protons at δ 8.2, followed by a singlet at δ 5.4 assigned to the anomeric proton. A multiplet at δ 5.1, assigned to H-2, was followed by a two-proton multiplet at $\delta \sim 4.4$ assigned to H-3 and H-4. The two protons on C-5, at δ 4.0, were followed by the methoxyl protons at δ 3.5 and the six methyl protons of the isopropylidene group at δ 1.4.

Hydrolysis of the isopropylidene group of compound **8** with 70% acetic acid yielded methyl 2-*O*-(*p*-nitrobenzoyl)-β-D-xylofuranoside (**9**) in crystalline form. This compound was *p*-nitrobenzoylated to give methyl tri-*O*-(*p*-nitrobenzoyl)-β-D-xylofuranoside (**10**) in an overall yield of 85%. (It should be noted that, if compound **7** is hydrolyzed and the product *p*-nitrobenzoylated, the yield of compound **10** is lowered



to less than 10%). When compound **10** was brominated with hydrogen bromide in dichloromethane, it yielded an anomeric mixture of the tri-*O*-(*p*-nitrobenzoyl)-D-xylofuranosyl bromides (**11**) in crystalline form. Integration of the n.m.r. spectrum of the mixed bromides (**11**) showed the α and β anomers to be present in the ratio of 5:2.

To test the reactivity of tri-*O*-(*p*-nitrobenzoyl)-α,β-D-xylofuranosyl bromide (**11**) with nucleophiles, it was treated with mercuric cyanide in nitromethane to give

2,5-anhydro-3,4,6-tri-*O*-(*p*-nitrobenzoyl)-D-gulononitrile (**12**). The n.m.r. spectrum of compound **12** confirmed its *gulo* configuration by showing the proton on C-2 as a singlet at δ 5.6. Compound **12** had a remarkable ability to solvate; it was isolated as both a crystalline, benzene solvate and a crystalline, 1,4-dioxane solvate.

EXPERIMENTAL

Melting points were determined on a Kofler block and are uncorrected. N.m.r. spectra were recorded with a Varian T-60 instrument. Mass spectra were measured with a Varian M-66 instrument by Mr. M. P. Gilles. Infrared spectra were recorded with Perkin–Elmer PE-700 and 621 spectrometers. Elemental analyses were performed by Spang Microanalytical Laboratories of Ann Arbor, Michigan, or by Mr. M. P. Gilles, Department of Chemistry and Chemical Engineering, Michigan Technological University.

Methyl 2,3,5-tri-O-p-*nitrobenzoyl-β*-D-*ribofuranoside* (**2**). — To a solution of D-ribose (12.5 g) in anhydrous methanol (250 ml) was added anhydrous methanol (25 ml) containing hydrogen chloride (1.11 g), the solution was kept for 90 min at room temperature, and then the reaction was quenched with pyridine (25 ml). The solution was evaporated to a yellow syrup which was treated with pyridine (25 ml), and the evaporation repeated. A solution of the yellow syrup (**1**) in pyridine (300 ml) and chloroform (100 ml) was stirred and cooled to 0°, and *p*-nitrobenzoyl chloride (50 g) was added in portions during 90 min below 10°; the mixture was kept for 15 h at room temperature, and then cooled to 0°. Ice (300 g) was added, and the aqueous layer was extracted with two 25-ml portions of dichloromethane. The extracts were combined, and evaporated under diminished pressure to remove most of the pyridine. The solid residue was dissolved in dichloromethane (500 ml), and the solution successively washed with a saturated solution of sodium hydrogen carbonate (4 × 200 ml), water (4 × 200 ml), ice-cold 1.5M sulfuric acid (2 × 150 ml), and water (2 × 200 ml), dried (anhydrous magnesium sulfate), and evaporated to dryness under diminished pressure. The solid product was triturated with anhydrous ether, and the resulting crystals were recrystallized from acetone–petroleum ether; yield 44.5 g (86%), m.p. 169.5–170°, $[\alpha]_D^{20}$ +79.7° (*c* 1.38, chloroform); mass-spectral data: *m/e* (55) 56, 57 (72), 67 (26), 69 (66), 71 (46), 81 (26), 83 (100), 85 (31), 95 (43), 97 (46), 109 (49), 111 (41), 125 (26), 127 (19), 149 (76), 167 (24), and 279 (3).

Anal. Calc. for $C_{27}H_{21}N_3O_{14}$: C, 53.04; H, 3.46; N, 6.87. Found: C, 53.08; H, 3.45; N, 6.66.

2,3,5-Tri-O-(*p-nitrobenzoyl*)-*β*-D-*ribofuranosyl bromide* (**3**). — A solution of compound **2** (7.25 g) in anhydrous dichloromethane (40 ml) was cooled to 0°, and treated with acetyl bromide (5 ml) and a saturated solution of hydrogen bromide in acetic acid [75 ml, purified by treating it with $B(OAc)_3$ according to the method prescribed by Jolley[18]]. The mixture was kept for 5 days at 4° and then evaporated under diminished pressure to an orange syrup. Slow co-evaporation with 25-ml portions of dry benzene, and then with dry benzene (20 ml) plus dry toluene (20 ml),

afforded the bromide as light-yellow crystals. These were washed with anhydrous diethyl ether, dried overnight under high vacuum (yield 6.19 g, 79%), and recrystallized from dichloromethane in needles, m.p. 100–105°, $[\alpha]_D^{20}$ +55.4° (*c* 1.58, chloroform); mass-spectral data: *m/e* 51 (28), 55 (35), 57 (81), 65 (21), 67 (17), 69 (26), 71 (58), 78 (100), 79 (18), 81 (27), 83 (51), 84 (72), 85 (47), 86 (58), 91 (72), 92 (44), 95 (39), 96 (17), 97 (74), 109 (47), 111 (44), 121 (21), 123 (21), 137 (5), 150 (79), and 167 (10).

Anal. Calc. for $C_{26}H_{18}BrN_3O_{13}$: C, 47.29; H, 2.75; N, 6.36. Found: C, 47.15; H, 2.74; N, 6.36.

2,5-Anhydro-3,4,6-tri-O-(p-*nitrobenzoyl*)-D-*allononitrile* (**4**). — A suspension of mercuric cyanide (20 g; dried for 5 h at 90° under high vacuum) in nitromethane (75 ml) was distilled with benzene (20 ml), and 25 ml of distillate was collected. The suspension was then cooled to 5°, compound **3** (10 g) was added, and the mixture was stirred for 65 h at room temperature and then filtered into a mixture of cold, aqueous M potassium bromide (200 ml) and methanol (40 ml) to remove the mercury salts. The mixture was stirred for 30 min, the suspension filtered, and the filtrate extracted with ten 40-ml portions of dichloromethane; the extracts were combined, successively washed with three 40-ml portions of M potassium bromide and water (50 ml), dried (anhydrous magnesium sulfate), and evaporated under diminished pressure to afford a syrup which crystallized (1.29 g, 14%) from chloroform–carbon tetrachloride in needles, m.p. 105–108°; mass-spectral data: *m/e* 55 (86), 56 (26), 57 (92), 67 (40), 69 (70), 71 (94), 81 (40), 83 (100), 85 (70), 95 (66), 97 (86), 109 (50), 111 (64), 123 (26), and 125 (24).

Anal. Calc. for $C_{24}H_{18}N_4O_{13}$: C, 53.47; H, 2.99; N, 9.24. Found: C, 53.34; H, 3.09; N, 9.14.

2,3,5-Tri-O-(p-*nitrobenzoyl*)-D-*ribofuranose* (**5**). — A solution of compound **3** (3 g) in acetone (25 ml) plus dichloromethane (25 ml) was treated with a suspension of silver carbonate (1.25 g) in water (5 ml), with vigorous stirring. The mixture was kept for 90 min at 5°, and filtered, and the filtrate evaporated under diminished pressure to syrupy **5** (1.76 g, 65%); it crystallized from nitromethane in needles, m.p. 177–178°; mass-spectral data: *m/e* 51 (33), 53 (13), 65 (94), 75 (55), 76 (45), 81 (26), 92 (16), 103 (17), 104 (50), 109 (19), 121 (86), 137 (30), 150 (100), 151 (22), and 167 (100).

Anal. Calc. for $C_{26}H_{19}N_3O_{14}\cdot 0.5H_2O$: C, 51.09; H, 3.32; N, 6.93. Found: C, 51.30; H, 3.15; N, 6.83.

1,2,3,5-Tetra-O-(p-*nitrobenzoyl*)-β-D-*ribofuranose* (**6**). — Powdered compound **5** (2.00 g) was dissolved in chloroform (10 ml) plus pyridine (30 ml), and the solution cooled to 5°, and stirred. *p*-Nitrobenzoyl chloride (0.65 g) was added slowly during 1 h below 8°, and the mixture was kept for 5 h at 15°, cooled to 5°, and treated with ice (30 g). The aqueous layer was separated, and extracted with two 10-ml portions of dichloromethane, and the extracts were combined with the organic layer and evaporated under diminished pressure to a syrup which was dissolved in dichloromethane (50 ml). The solution was successively washed with two 15-ml portions of an ice-cold, saturated solution of sodium hydrogen carbonate, two 15-ml portions

of ice-cold water, ice-cold 1.5M sulfuric acid (10 ml), and two 15-ml portions of ice-cold water, dried (anhydrous magnesium sulfate), and evaporated to a syrup which crystallized (0.5 g, 20%). Compound **6** had m.p. 199–201°; mass-spectral data: *m/e* 51 (71), 57 (6), 65 (42), 74 (13), 75 (19), 67 (20), 81 (8), 84 (10), 104 (28), 121 (49), 135 (10), 150 (100), 151 (11), 167 (97), and 412 (2).

Anal. Calc. for $C_{33}H_{22}N_4O_{17}$: C, 53.09; H, 2.97; N, 7.50. Found: C, 52.58; H, 2.97; N, 7.42.

Methyl 3,5-O-isopropylidene-2-O-(p-nitrobenzoyl)-β-D-xylofuranoside (**8**). — A solution of methyl 3,5-*O*-isopropylidene-β-D-xylofuranoside[17] (**7**, 42.9 g) in pyridine (250 ml) was cooled in an ice bath, *p*-nitrobenzoyl chloride (43.1 g) was added, and the mixture was kept for 30 min in the ice bath, and then stirred for 24 h at room temperature. Ice (400 g) was added, and the mixture kept for 1 h at room temperature to hydrolyze the excess of *p*-nitrobenzoyl chloride. Dichloromethane (500 ml) was added, and the suspension was filtered to remove the *p*-nitrobenzoic acid that separated. The filtrate was successively washed with water (250 ml) and a cold, saturated solution of sodium hydrogen carbonate (3 × 500 ml), dried (anhydrous magnesium sulfate), and evaporated to dryness under diminished pressure at 40°. Toluene (3 × 100 ml) was added to and evaporated from the residue, to remove the last traces of pyridine, and the product was recrystallized from methanol (66.2 g, 89.2%), m.p. 94–95°; mass-spectral data: *m/e* 338 (55), 322 (.6), 252 (4), 233 (4), 150 (100), and 104 (12).

Anal. Calc. for $C_{16}H_{19}NO_8$: C, 54.39; H, 5.42; N, 3.96. Found: C, 54.27; H, 5.35; N, 3.87.

Methyl 2,3,5-tri-O-(p-nitrobenzoyl)-β-D-xylofuranoside (**10**). — A solution of compound **8** in 70% acetic acid (23.0 ml) was kept for 2 h at 50°, evaporated to dryness under diminished pressure at 50°, and the residue treated by adding and evaporating toluene (25 ml) and then ethanol (15 ml). A solution of the resultant, greenish syrup (**9**) in pyridine (125 ml) was cooled in an ice bath, and treated with *p*-nitrobenzoyl chloride (11.6 g); the mixture was stirred, under anhydrous conditions, for 30 min in an ice bath and for 20 h at room temperature. Ice (100 g) was added, the mixture was kept until the ice had melted, and dichloromethane (125 ml) was added. The suspension was filtered, and the filtrate was transferred to a separatory funnel. The aqueous layer was removed, and the dichloromethane layer was successively washed with a saturated, aqueous solution of sodium hydrogen carbonate (3 × 150 ml) and water (150 ml), dried (anhydrous magnesium sulfate), and evaporated to dryness. The residue was dissolved in the minimal volume of boiling acetone, and petroleum ether was added until the ratio of acetone to petroleum ether was 2:3. The resulting crystals were filtered off, and recrystallized twice from acetone–petroleum ether; yield, 15.3 g of chromatographically pure crystals, m.p. 144°. The mother liquors afforded another 1.2 g of crystals (overall yield, 95.3%); mass-spectral data: *m/e* 611 (.01), 580 (.85), 431 (.9), 412 (4), 386 (6), 371 (4), 300 (8), 277 (2), 167 (2), 150 (100), 134 (6), 120 (4), and 104 (8).

Anal. Calc. for $C_{27}H_{21}N_3O_{14}$: C, 53.04; H, 3.56; N, 6.78. Found: C, 53.01; H, 3.32; N, 6.81.

2,3,5-Tri-O-(p-*nitrobenzoyl*)-α,β-D-*xylofuranosyl bromide* (**11**). — A solution of compound **10** (3 g) in dry dichloromethane (60 ml) presaturated with hydrogen bromide was kept for 24 h at 0–3° under anhydrous conditions. The mixture was evaporated under diminished pressure at 40°, and then dry toluene (3 × 25 ml) was added to and evaporated from the product. The resulting solid was crystallized from chloroform–hexane (yield 2.2 g, 68%). Recrystallization from chloroform–hexane gave an analytical sample of the anomers of the bromide; mass-spectral data: *m/e* 412 (4), 411 (4), 326 (1), 324 (1), 280 (10), 262 (2), 245 (4), 167 (80), 150 (100), 121 (97), 104 (26), 82 (15), 21 (28), and 80 (17).

Anal. Calc. for $C_{26}H_{18}BrN_3O_{13}$: C, 47.29; H, 2.75; Br, 12.10; N, 6.36. Found: C, 47.26; H, 2.72; Br, 12.14; N, 6.41.

2,5-Anhydro-3,4,6-tri-O-(p-*nitrobenzoyl*)-D-*gulononitrile* (**12**). — A suspension of 2,3,5-tri-*O*-*p*-nitrobenzoyl-D-xylofuranosyl bromide (6.5 g) and mercuric cyanide (4.8 g) in dry nitromethane (65 ml) was stirred for 44 h at room temperature, the inorganic matter was removed by filtration, and the filtrate was added to a stirred solution of cold M potassium bromide (500 ml) in methanol (75 ml). The resulting mixture was extracted with dichloromethane (5 × 100 ml), and the extracts were combined, successively washed with 2.0M potassium bromide (2 × 150 ml) and water (250 ml), dried (anhydrous magnesium sulfate), and evaporated under diminished pressure at 40° to a yellow syrup. Compound **12** crystallized from benzene–ethyl acetate in solvated needles (yield 1.3 g, 22.0%), m.p. 211–217°; mass-spectral data: *m/e* 290 (2), 272 (4), 167 (46), 150 (100), 121 (39), and 104 (26).

Anal. Calc. for $C_{27}H_{18}N_4O_{13}\cdot C_6H_6$: C, 53.47; H, 2.99; N, 9.24. Found: C, 53.15; H, 3.04; N, 8.82.

Recrystallization from 1,4-dioxane gave the 1,4-dioxane solvate, m.p. 210–212°.

Anal. Calc. for $C_{27}H_{18}N_4O_{13}\cdot C_4H_8O_2$: C, 53.61; H, 3.77; N, 8.07. Found: C, 53.49; H, 3.80; N, 8.00.

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