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SYNTHESES OF *p*-AMINOPHENYL α-D-TALOPYRANOSIDE AND 1-THIO α-D-TALOPYRANOSIDE AS ANALOGS OF GLYCOSIDASE SUBSTRATES*

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ABSTRACT

p-Nitrophenyl and *p*-aminophenyl α-D-talopyranoside and 1-thio-α-D-talopyranosides were prepared for studies on specificity of glycosidases. Reaction of α-D-talopyranose pentaacetate with *p*-nitrophenol gave exclusively *p*-nitrophenyl 2,3,4,6-tetra-*O*-acetyl-α-D-talopyranoside (**2**) in 63% yield. A similar reaction with *p*-nitrobenzenethiol afforded the 1-thio analog (**3**) of **2** in 41.8% yield; the *p*-nitrophenyl 2,3,4,6-tetra-*O*-acetyl-1-thio-β-D-talopyranoside (**6**) was also obtained in low yield (6.7%). The two α-D-talosides **2** and **3** were catalytically deacetylated in near-quantitative yields by methanolic sodium methoxide. The *p*-nitrophenyl α-D-talopyranoside (**4**) and 1-thio-α-D-talopyranoside (**5**) were reduced with palladium on barium sulfate catalyst to the corresponding *p*-aminophenyl talosides. The acetylated *p*-nitrophenyl D-talosides **2**, **3**, and **6** were determined, from their 250-MHz n.m.r. spectra, to exist in the $^4C_1$ (D) conformation in chloroform solution.

INTRODUCTION

The requirements for the binding of substrates to glycosidases are generally regarded as fairly stringent with respect to the sugar moiety[1], although considerable freedom exists in the choice of the aglycon[2]. However, some glycosidases are capable of hydrolyzing glycosidic linkages of more than one sugar. A frequently encountered enzyme in this respect is 2-acetamido-2-deoxy-β-D-glucosidase, which hydrolyzes both 2-acetamido-2-deoxy-β-D-glucopyranosides and 2-acetamido-2-deoxy-β-D-galactopyranosides[3]. Furthermore, β-D-glucosidase and β-D-galactosidase from almond emulsin are known to hydrolyze β-D-xylosides and β-L-arabinopyranosides, respectively[4]. More recently, De Prijcker *et al.*[5] have reported that the α-D-mannosidase of *Medicago sativa* can bind to α-D-mannosides and also α-D-glucosides, thus indicating that this enzyme is able to tolerate configurational change at C-2 of its substrate. These observations suggested the need for hitherto unreported aryl glycosides of the

*Part I of a series "Aryl Glycosides of Uncommon Aldohexoses."

uncommon aldohexoses, D-talose, D-idose, D-allose, D-gulose, and D-altrose, in order to understand fully the requirements for the binding of the glycosidases with their substrates. These derivatives are also of potential interest for use as ligands in the affinity purification of glycosidases, in view of the fact that not all glycosidases having the same activity are inhibited by a given ligand, and there is, thus, a need for substitute analogs. For example, only one of the two β-D-galactosidases from bovine testes is retained on the affinity adsorbent containing the ligand *p*-aminophenyl 1-thio-β-D-galactopyranoside, whereas the other is eluted unretarded from the column, indicating the selective affinity of the ligand for one of the two β-D-galactosidases[6]. Furthermore, these derivatives may prove useful in the purification by affinity chromatography of those glycosidases, such as the α-D-mannosidase from *Aspergillus niger*[7] and yeast vacuolar membrane[8], and the β-D-mannosidase[9] from *A. niger*, which are not inhibited by the commonly employed 1-thio analogs of the respective *p*-nitrophenyl D-glycopyranoside substrates.

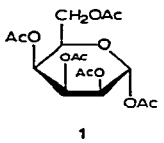
This paper describes the syntheses of *p*-aminophenyl α-D-talopyranoside (**7**) and 1-thio-α-D-talopyranoside (**8**). Of particular interest is the efficacy of **4**, **5**, **7**, and **8** in binding to α-D-mannosidases and α-D-galactosidases, as these derivatives differ from the substrates of these two enzymes in configurations at C-4 and C-2, respectively. This consideration is based upon the observed inhibition[10] of the β-D-mannosidase of human synovial fluid by *p*-nitrophenyl β-D-galactopyranoside, which suggests the possibility that, for the $^4C_1$ (D) conformation, the axial hydroxyl group at C-4 of this derivative effectively substitutes for the axial hydroxyl group at C-2 of the substrate (*p*-nitrophenyl β-D-mannopyranoside) for this enzyme.

RESULTS AND DISCUSSION

Penta-*O*-acetyl-α-D-talopyranose (**1**), prepared from β-D-galactopyranose pentaacetate by the antimony pentachloride procedure of Paulsen[11], was fused[12,13] with *p*-nitrophenol in the presence of anhydrous zinc chloride at 125° to afford crystalline *p*-nitrophenyl 2,3,4,6-tetra-*O*-acetyl-α-D-talopyranoside (**2**) in 62% yield as the exclusive product; no trace of the β anomer was evident. Similarly, fusion of **1** with *p*-nitrobenzenethiol afforded crystalline *p*-nitrophenyl 2,3,4,6-tetra-*O*-acetyl-1-thio-α-D-talopyranoside (**3**) as a major product (41.8%). However, in contrast to the reaction of **1** with *p*-nitrophenol, a small proportion (6.7%) of the β anomer **6** was also isolated. The formation of a proportionately larger amount of the β glycoside has also been observed in the reaction of D-mannopyranose pentaacetate with *p*-nitrobenzenethiol[14], as compared with that from the reaction with *p*-nitrophenol[15]. The anomeric assignments in the *p*-nitrophenyl talosides **2**, **3**, and **6** were made on the basis of Hudson's isorotation rules[16], and confirmed by 250-MHz nuclear magnetic resonance (n.m.r.) spectroscopy (see later).

The derivatives **2** and **3** were deacetylated[17] with catalytic amounts of sodium methoxide in methanol, and crystalline *p*-nitrophenyl α-D-talopyranoside (**4**) and 1-thio-α-D-talopyranoside (**5**) were isolated in nearly quantitative yields. Pressure

hydrogenation[18] of **4** with 5% palladium-on-barium sulfate afforded the crystalline *p*-aminophenyl α-D-talopyranoside (**7**) in 82% yield. However, similar reduction of **5** gave *p*-aminophenyl 1-thio-α-D-talopyranoside (**8**) as a chromatographically homogeneous syrup (R_F 0.48), which was characterized by conversion into *p*-acetamidophenyl 2,3,4,6-tetra-*O*-acetyl-1-thio-α-D-talopyranoside (**9**).



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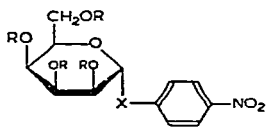
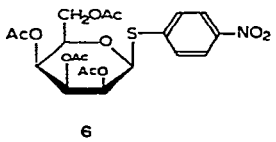


2 X = O, R = Ac
3 X = S, R = Ac
4 X = O, R = H
5 X = S, R = H



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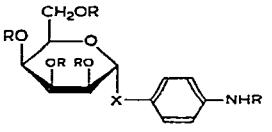

7 X = O, R = H
8 X = S, R = H
9 X = S, R = Ac

The chemical-shift and coupling-constant data from 250-MHz n.m.r. spectra of compounds **2**, **3**, and **6** are listed in Table I. The resonances of the methine protons on the pyranose ring of the three talosides **2** (Fig. 1a), **3**, and **6** were well resolved from each other, and from those of the geminal protons at C-6. For all three derivatives (**2**, **3** and **6**), the H-1 signal appeared as a doublet ($J_{1,2}$ ~1.5 Hz) and the H-3 signal was a triplet ($J_{2,3} = J_{3,4} = 3.7$ Hz). A sextet (doublet of triplets) was present in the spectra of the two α-talosides (**2** and **3**), and this signal was assigned to H-2, which is coupled to H-3 ($J_{2,3}$ 3.7 Hz), H-1 ($J_{1,2}$ ~1.5 Hz), and H-4 ($J_{2,4}$ ~1.2 Hz). The multiplet arising from H-4 of **2** (Fig. 1a) and **3** was resolved only partially, most

TABLE I

CHEMICAL SHIFTS (τ VALUES) AND COUPLING CONSTANTS (Hz) OF ACETYLATED *p*-NITROPHENYL D-TALOSIDES FROM 250-MHz N.M.R. SPECTRA[a]

Compound	*H-1*	*H-2*	*H-3*	*H-4*	*H-5*	*H-6*	*H-6'*	*Acetoxyl*
1-*O*-α (**2**)	4.29 (d) $J_{1,2}$ 1.4 $J_{1,4}$ <0.5	4.68 (sx) $J_{2,3}$ 3.7 $J_{2,4}$ 1.1	4.51 (t) $J_{3,4}$ 3.7	4.59 (pm) $J_{4,5}$ 1.4	5.72 (o) $J_{5,6}$ 5.5 (4.8)[b] $J_{5,6'}$ 7.8 (8.6)[b]	5.81 (q)[b] $J_{6,6'}$ 11.8	5.85 (q)[b]	7.81, 7.84 7.96, 8.08
1-*S*-α (**3**)	4.21 (d) $J_{1,2}$ 1.5 $J_{1,4}$ <0.5	4.68 (sx) $J_{2,3}$ 3.7	4.75 (t) $J_{3,4}$ 3.7	4.60 (pm) $J_{4,5}$ 1.5	5.33 (sx) $J_{5,6}$ 6.4	5.80 (d)	5.80 (d)	7.84, 7.85 7.98, 8.05
1-*S*-β (**6**)	4.88 (d) $J_{1,2}$ 1.4	4.44 (bd) $J_{2,3}$ 3.8	4.85 (t) $J_{3,4}$ 3.8	4.65 (bd) $J_{4,5}$ 1.6	5.95 (o) $J_{5,6}$ 7.2 (8.1)[b] $J_{5,6'}$ 5.6 (5.7)[b]	5.70 (q)[b] $J_{6,6'}$ 11.6	5.76 (q)[b]	7.80, 7.83 7.91, 8.00

[a]Abbreviations: bd = broad doublet, d = doublet, o = octet, pm = partially resolved multiplet, q = quartet, sx = sextet, and t = triplet. [b]Calculated by ABX analyses (ref. 22).

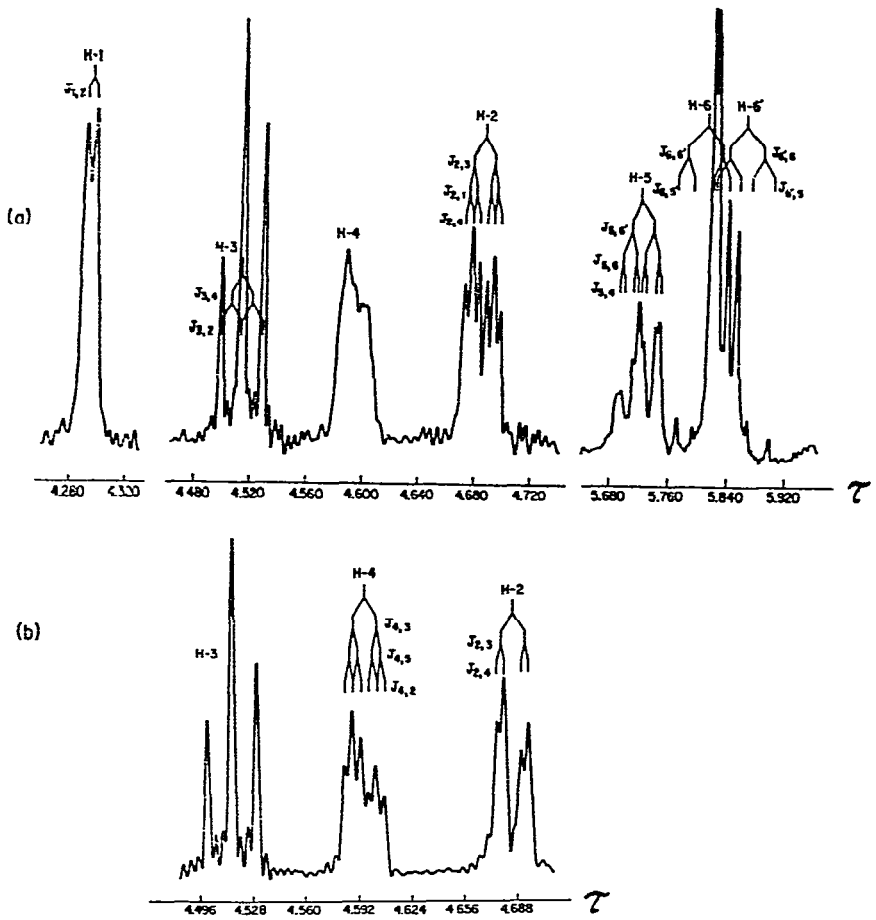

Fig. 1. 250-MHz n.m.r. spectrum of *p*-nitrophenyl 2,3,4,6-tetra-*O*-acetyl-α-D-talopyranoside (**2**) in chloroform-*d* with tetramethylsilane as internal reference: (a) single-resonance spectrum; (b) effect of irradiation of the H-1 resonance.

probably because of coupling of H-4 with H-1, in addition to its coupling with H-2 and H-3. This supposition was confirmed by double-resonance experiments by the frequency-sweep technique. When the H-1 resonance of **2** (Fig. 1b) and **3** was irradiated, the coupling of H-1 with H-2 and H-4 was removed. As expected, this operation caused the collapse of the H-2 signal to a quartet in both **2** (Fig. 1b) and **3**. Also, the H-4 signal of **2** was transformed into a well-defined sextet (doublet of triplets), whereas its definition improved considerably in **3**. Although the precise ^{5}J magnitude (estimated $J_{1,4}$ <0.5 Hz) in compounds **2** and **3** was not determined, it appears to be a common feature of equatorially oriented protons at C-1 and C-4 in hexopyranose rings. Such coupling has been observed thus far in acetylated derivatives of three of four hexopyranoses where it is possible, namely, the α-D-talosides **2** and **3**, α-D-

gulopyranose pentaacetate[19], α-D-idopyranose pentaacetate[20], and *p*-nitrophenyl 2,3,4,6-tetra-*O*-acetyl-α-D-idopyranoside and its 1-thio analog[21]. It is likely that high-resolution n.m.r. spectroscopy will reveal the presence of ^5J couplings in analogous derivatives of the only remaining hexopyranose, namely, α-galactose.

For the 1-thio-β-D-taloside **6**, the H-2 and H-4 resonances appeared, not unexpectedly, as nearly identical signals that could not be distinguished, as for derivatives **2** and **3**, on the basis of the $J_{1,4}$ coupling. However, irradiation of the resonance at τ 4.44 collapsed the H-3 triplet to a doublet, the H-1 doublet to a singlet, and also simplified the signal at τ 4.64 (H-4) to a doublet of rounded peaks, thus establishing the irradiated resonance as that of H-2. The $J_{2,4}$ value in **6** could not be measured precisely, although its existence is proved by the effect on the H-4 resonance of the removal of the H-2–H-4 coupling.

The signal for the C-6 protons of the 1-thio-α-D-taloside **3** appeared as a doublet at τ 5.80, indicating these two protons to be isochronous, whereas the resonances of the geminal protons of the α-D-taloside **2** (Fig. 1a) and 1-thio-β-D-taloside **6** were slightly separated (Table I). The identity of the H-5 signal for the three talosides (**2**, **3**, and **6**) was deduced from the tilt of the H-5 signal and the H-6 signals toward each other, and also on the basis of the $J_{4,5}$ and $J_{5,6(6')}$ coupling constants. Both the observed and calculated[22] (by ABX analysis) $J_{5,6}$ and $J_{5,6'}$ values are listed in Table I. A coupling of 11.6 Hz and 11.8 Hz was observed for the geminal protons of **2** and **6**, respectively, in contrast to the values of 9.6 and 9.5 Hz reported by Streefkerk *et al.* for the geminal protons of per-*O*-trimethylsilyl derivatives of α- and β-D-talopyranose, respectively[23].

Interestingly, the glycosidic oxygen and sulfur atoms in the two α-talosides **2** and **3** affect differently the chemical shifts of the *syn*-diaxial protons at C-3 and C-5 (Table I). For example, comparison of the 1-thio-β-D-taloside **6** with the α anomer **3**, reveals that H-3 is deshielded by 0.1 p.p.m., whereas the other *syn*-diaxial proton, at C-5, is deshielded by 0.62 p.p.m. When the sulfur atom of the thio-α-D-taloside **3** is replaced by oxygen (compound **2**), the axial proton at C-3 is shifted downfield by 0.25 p.p.m. and the axial proton at C-5 is moved upfield by 0.39 p.p.m. As both H-3 and H-5 should experience nearly equal deshielding by sufur in **3**, and oxygen in **2**, the observed inconsistency is possibly due to the location of H-5 in the shielding zone of the aromatic ring in its favored orientation. At the same time such an orientation, may also account for the nonequivalence of the geminal protons of **2**.

Axial acetoxymethyl groups are reported[24] to resonate between τ 7.81 and 7.85, as compared with those of equatorial acetoxymethyl groups which generally resonate between τ 7.89 and 7.98; equatorially oriented, primary acetoxymethyl signals may appear to slightly higher field at τ 7.98–8.02. In accordance with this classification, the two low-field resonances between τ 7.80 and 7.85 in **2**, **3**, and **6** are assigned to the axial acetoxyl groups at C-2 and C-4. Because the equatorial primary acetoxymethyl group may resonate at a frequency outside the range already given[24], no specific assignments are made for the signals at τ 7.91 and 8.00.

The foregoing n.m.r. evidence supports the $^4C_1$(D) conformation for the

talosides **2**, **3**, and **6**. Streefkerk *et al.* have determined that trimethylsilyl 2,3,4,6-tetrakis-*O*-(trimethylsilyl)-α- and -β-D-talopyranosides also adopt the $^4C_1$(D) conformation[23].

EXPERIMENTAL

General methods. — Melting points were determined with a Fisher–Johns apparatus and are uncorrected. Infrared spectra were recorded with a Perkin–Elmer Model 257 spectrophotometer. Optical rotations were measured at room temperature in a 1-dm cell with a Perkin–Elmer Model 241 automatic polarimeter. The R_F values were determined by t.l.c. on plates coated with silica gel G having a layer thickness of approximately 0.25 mm. The silica gel G for t.l.c. was purchased from Sigma Chemical Co., St. Louis, Missouri. The solvents employed for t.l.c. were 5:1 benzene–ethyl acetate and 3:1:0.2 ethyl acetate–acetic acid–water for acetylated and nonacetylated derivatives, respectively. Detection of aromatic compounds on t.l.c. plates was effected by iodine vapor. Subsequently, plates were sprayed with 5% (v/v) methanolic sulfuric acid and charred to reveal carbohydrate derivatives. Column chromatography was performed on Hi-Flosil, 60–200 mesh, purchased from Applied Science Laboratories, State College, Pennsylvania. *p*-Nitrobenzenethiol (Aldrich Chemical Co., Cedar Knolls, N.J.) was of 80+% purity. Evaporations were performed under diminished pressure at <40°. The 250-MHz n.m.r. spectra were recorded at the N.M.R. Facility for Biomedical Studies, Carnegie-Mellon University, Pittsburgh, Pennsylvania. The chemical shifts and coupling constants were determined in chloroform-*d* with tetramethylsilane as the internal reference. Elementary analyses were performed by Galbraith Laboratories, Knoxville, Tennessee.

p-*Nitrophenyl 2,3,4,6-tetra*-O-*acetyl*-α-D-*talopyranoside* (**2**). — 1,2,3,4,6-Penta-*O*-acetyl-α-D-talopyranose (1) was prepared from β-D-galactopyranose pentaacetate by acetoxonium-ion rearrangement essentially as described by Paulsen[11], except that 2,3,4,6-tetra-*O*-acetyl-β-D-galactopyranosyl chloride was prepared by the aluminum chloride procedure[25]. A mixture of α-D-talopyranose pentaacetate (**1**, 2.0 g) and *p*-nitrophenol (2.0 g) was fused[12,13] for 45 min at 125° in an evacuated flask in the presence of anhydrous zinc chloride (0.47 g) in 2 ml of 19:1 acetic acid–acetic anhydride. The dark-brown syrup was dissolved in chloroform (30 ml), and the solution was washed successively with cold water (3 × 30 ml), cold, M sodium hydroxide (4 × 30 ml), and cold water (5 × 30 ml). After drying with anhydrous sodium sulfate, the chloroform solution was evaporated. The residual syrup crystallized from methanol (10 ml) to give 2.41 g (62.9%) of **1**, m.p. 159–161°. One recrystallization from methanol furnished the analytical sample, m.p. 163–165°, $[\alpha]_D$ +116.0° (*c* 0.31, chloroform); R_F 0.38; ν_{max}^{KBr} 1750 and 1737 (C=O), 1610, 1593, and 1495 (aromatic), 1520 and 1349 (NO_2), 1230 (acetate C–O–C), 868, 858, 792, 758, 742 (shoulder), and 738 cm^{-1}; n.m.r.: see Table I and Fig. 1.

Anal. Calc. for $C_{20}H_{23}NO_{12}$: C, 51.18; H, 4.94; N, 2.98. Found: C, 51.37; H, 5.05; N, 3.03.

p-Nitrophenyl 2,3,4,6-tetra-O-acetyl-1-thio-α- (**3**) *and -β-D-talopyranoside* (**6**). — α-D-Talopyranose pentaacetate[11] (**1**, 2.0 g) was fused[12,13] with *p*-nitrobenzenethiol (2.0 g) for 30 min as already described for **2**. The dark-brown mixture was then extracted with chloroform (50 ml) and filtered to remove a small amount of an insoluble solid. The filtrate was washed with cold water (3 × 30 ml), cold, saturated sodium hydrogencarbonate solution (3 × 30 ml), and cold water (4 × 30 ml), and dried with anhydrous sodium sulfate. The yellow, syrupy residue obtained after evaporation of the chloroform solution was taken up in benzene (5 ml), and filtered to remove 879 mg of yellow crystals, mainly of **3**, m.p. 144–150°. Both the crystals and the filtrate were separately chromatographed on columns of silica gel. The columns were washed with benzene to remove by-products arising from *p*-nitrobenzenethiol and then the desired glycosides **3** and **6** were eluted with 10:1 benzene–ethyl acetate. Fractions were monitored by t.l.c., and those rich in the β-glycoside **6** were pooled and rechromatographed twice to give pure **6**. In this way, 1.04 g (41.8%) of the α anomer (**3**) and 0.167 g (6.7%) of the β anomer (**6**) were obtained.

An analytical sample of the α anomer **3**, m.p. 162–163°, was prepared by recrystallization from methanol; $[\alpha]_D$ +162.4° (*c* 0.25, chloroform); R_F 0.38; ν_{max}^{KBr} 1750 and 1735 (C=O), 1595 and 1578 (aromatic), 1510 and 1340 (NO_2), 1260, 1230, and 1210 (acetate C–O–C), 858 (C–N), 845 (*p*-disubstituted phenyl), and 748 cm^{-1} (C–N–O); n.m.r.: see Table I.

Anal. Calc. for $C_{20}H_{23}NO_{11}S$: C, 49.48; H, 4.78; N, 2.89. Found: C, 49.51; H, 4.82; N, 2.95.

Recrystallization from methanol afforded the analytical sample of the β anomer **6**, m.p. 165–167°, $[\alpha]_D$ −90.4° (*c* 0.31, chloroform); R_F 0.33; ν_{max}^{KBr} 1745 and 1735 (C=O), 1598 and 1580 (aromatic), 1518 and 1340 (NO_2), 1240–1215 (acetate C–O–C), 855, 773, 748, 735, and 725 cm^{-1}; n.m.r.: see Table I.

Anal. Calc. for $C_{20}H_{23}NO_{11}S$: C, 49.48; H, 4.78; N, 2.89. Found: C, 49.60; H, 4.84; N, 2.93.

p-Nitrophenyl α-D-talopyranoside (**4**). — Compound **2** (292 mg) was deacetylated[17] with 2.3 ml of 0.02M methanolic sodium methoxide. After 5 h at room temperature, methanol (5 ml) was added to dissolve the crystals of **4**, and the solution was neutralized with methanol-washed Dowex-50 (H^+) resin. Filtration, followed by evaporation, left a colorless, crystalline residue, which was triturated with anhydrous ether and filtered; yield, 181 mg (96.8%); m.p. 170–175°, $[\alpha]_D$ +187.4° (*c* 0.22 methanol); R_F 0.73; ν_{max}^{KBr} 3500–3200 (OH), 1612, 1593, and 1494 (aromatic), 1510, and 1340 (NO_2), 868, 852, 802, 790, 670 (shoulder), and 755 cm^{-1}.

Anal. Calc. for $C_{12}H_{15}NO_8$: C, 47.84; H, 5.02; N, 4.65. Found: C, 47.78; H, 5.09; N, 4.71.

p-Nitrophenyl 1-thio-α-D-talopyranoside (**5**). — Compound **3** (292 mg) was deacetylated as already described for **4**. The yield of crystalline **5** was 189 mg (99.5%); m.p. 178–180°, $[\alpha]_D$ +330.9° (*c* 0.25, methanol); R_F 0.74; ν_{max}^{KBr} 3500–3300 (OH), 1598 and 1580 (aromatic), 1510 and 1335 (NO_2), 860, 843, 828, 800, and 744 cm^{-1}.

Anal. Calc. for $C_{12}H_{15}NO_7S$: C, 45.42; H, 4.76; N, 4.41. Found: C, 45.34; H, 4.81; N, 4.42.

p-*Aminophenyl* α-D-*talopyranoside* (**7**). — Compound **4** (150 mg) was dissolved in methanol (50 ml) and hydrogenated over 5% palladium on barium sulfate (0.100 g) at an initial pressure of 50 lb.in.$^{-2}$ for 18 h. The catalyst was removed by filtration through Celite, and the filtrate was evaporated to dryness. Crystallization of the residue from methanol afforded cream-colored crystals of **7**; yield, 111 mg (82.2%); m.p. 180–182°, $[\alpha]_D$ +151.0° (*c* 0.25, methanol); R_F 0.17; ν_{max}^{KBr} 3500–3300 (OH, NH), 1630 (NH_2), 1510 (aromatic), 860, 847, 832, 812, and 790 cm^{-1}.

Anal. Calc. for $C_{12}H_{17}NO_6$: C, 53.13; H, 6.32; N, 5.16. Found: C, 52.97; H, 6.45; N, 5.06.

p-*Acetamidophenyl 2,3,4,6-tetra*-O-*acetyl-1-thio*-α-D-*talopyranoside* (**9**). — Hydrogenation of compound **5** (150 mg) as described for **7** afforded a chromatographically homogeneous syrup of *p*-aminophenyl 1-thio-α-D-talopyranoside (**8**) which could not be induced to crystallize; yield 144 mg (106%); R_F 0.48. The syrupy **8** was acetylated overnight at room temperature with a mixture of pyridine (3 ml) and acetic anhydride (1 ml). The solvents were removed by evaporation of toluene from the product to obtain a yellow syrup (250 mg, 106% from **5**) which was homogeneous except for the presence of traces of four components. The syrup was dissolved in methanol and decolorized with Nuchar. Concentration of the decolorized solution gave **9** as an almost colorless syrup (208 mg, 88.5% from **5**), $[\alpha]_D$ +112.6° (*c* 0.33, chloroform). Crystallization from 2-propanol–petroleum ether (low boiling) afforded 165 mg (70.2% from **5**) of analytically pure **9** as colorless crystals, m.p. 145–147°, $[\alpha]_D$ +125.9° (*c* 0.23, chloroform); R_F 0.05; ν_{max}^{KBr} 3350 (NH), 1745 (ester C=O), 1680 (amide, type I band), 1592 and 1492 (aromatic), 1525 (amide, type II band), 1250 (shoulder) and 1230 (acetate C–O–C), 835, and 760 cm^{-1}.

Anal. Calc. for $C_{22}H_{27}NO_{10}S$: C, 53.11; H, 5.47; N, 2.82. Found: C, 52.98; H, 5.43; N, 2.81.

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