Direct Oxidative Amidation of Aldoses by Iodine in Ammonia Water

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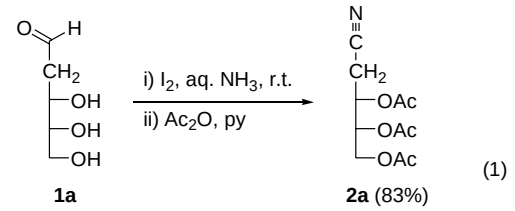
Aldopentoses, aldohexoses and the benzylated derivatives reacted with iodine in ammonia water at room temperature to give their corresponding saccharide amides in high yields. The reactions proceeded with oxidation of the aldose hemiacetals by iodine to generate the saccharide lactone intermediates, which underwent ammonolysis in situ to give the saccharide amides.

Keywords: Aldoses; Amides; Iodine; Ammonia.

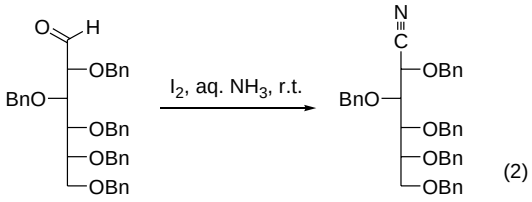
INTRODUCTION

We have recently found a direct method, using iodine in ammonia water, for transformation of aldehydes to nitriles.[1] This transformation often occurs at room temperature within a short period (< 1 h) in an efficient manner. A variety of aldehydes, including aromatic, heterocyclic, aliphatic, conjugated and polyhydroxy aldehydes, have thus been treated with I_2 in aqueous NH_3 to afford their corresponding nitriles with high yields (83-97%). The polyhydroxy aldehydes such as 2-deoxy-D-ribose (**1a**) and 2,3,4,5,6-penta-*O*-benzyl-D-glucose (**1b**) are especially attractive substrates, because they are water-soluble and no protection of the hydroxyl groups is required (Eqs. 1 and 2).

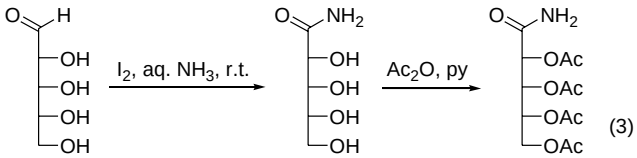
In order to apply this simple, economic and environmentally benign method to carbohydrate chemistry, we investigated further the reactions of aldoses with iodine in ammonia water.



1a **2a** (83%) (1)



1b **2b** (85%) (2)



3a **4a** **5a** (71%) (3)

RESULTS AND DISCUSSION

When D-ribose (1 mmol) was treated with iodine (1.2 mmol) in ammonia water (10 mL of 28% solution), the dark solution became colorless after stirring at room temperature for 6 h. The reaction mixture was concentrated under reduced pressure to give a practically pure product. The ^{13}C NMR spectrum (CD_3OD, 75 MHz) of this product showed five signals at δ 63.0, 69.0, 69.4, 70.8 and 178.6. None of these signals could be attributed to the putative nitrile product, which should exhibit a signal around δ 120 for the cyano group. Instead, the ^{13}C NMR spectrum fit a structure of ribonamide (**4a**)[2] with the amido group displaying at δ 178.6 (Eq. 3). Subsequent peracetylation of this crude product (Ac_2O, pyridine, 25 °C, 6 h) afforded 2,3,4,5-*O*-tetraacetyl-D-ribonamide (**5a**) with full structural characterization.[3]

The aldopentoses and aldohexoses examined in this study all reacted similarly with iodine in ammonia water to

Dedicated to Professor Fa-Ching Chen on the occasion of his ninetieth birthday.

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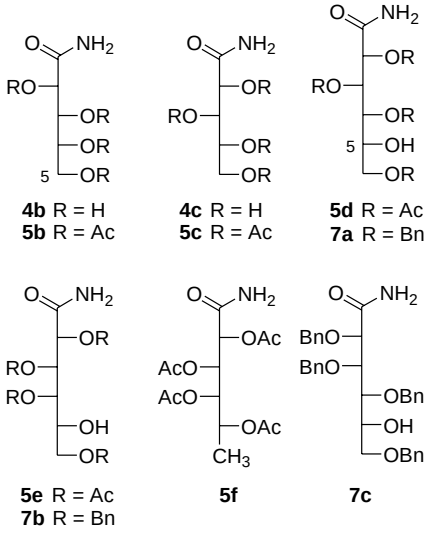
Table 1. Reactions of Aldoses with Iodine in Ammonia Water at Room Temperature, Giving Saccharide Amides

Aldose	Cosolvent	Reaction time (h)	Amide product (yield, %)
D-ribose (**3a**)	–	6	**5a**[a] (71)[b, c]
D-arabinose (**3b**)	–	10	**5b**[a] (62)[b, c]
D-xylose (**3c**)	–	9	**5c**[a] (78)[b, c]
D-glucose (**3d**)	–	3	**5d**[a] (87)[b]
D-galactose (**3e**)	–	3	**5e**[a] (85)[b]
D-fucose (**3f**)	–	2	**5f**[a] (90)[b]
2,3,4,6-tetra-*O*-benzyl-D-glucose (**6a**)	THF	15	**7a** (82)
2,3,4,6-tetra-*O*-benzyl-D-galactose (**6b**)	THF	2	**7b** (85)
2,3,4,6-tetra-*O*-benzyl-D-mannose (**6c**)	THF	7	**7c** (89)

[a] The final product of two steps: (i) reaction with I_2 in aqueous NH_3, and (ii) acetylation with Ac_2O in pyridine.
[b] The overall yield of two steps.
[c] Though the reaction with I_2 in aqueous NH_3 was clean as shown by the ^{13}C NMR analysis, the overall yield decreased due to incomplete acetylation.

afford the corresponding saccharide amides in good yields (Table 1). These aldopentoses and aldohexoses usually exist as the hemiacetal forms by linkage of the C-4 or C-5 hydroxyl groups with the C-1 aldehyde groups. Our current method thus provided a way to modify aldose derivatives, particularly at the C-4 and C-5 positions. For example, a THF solution of 2,3,4,6-tetra-*O*-benzyl-D-glucose (**6a**) was treated with iodine in ammonia water to give 2,3,4,6-tetra-*O*-benzyl-D-gluconamide (**7a**) in 82% yield. The unprotected hydroxyl group at C-5 has been subjected to Moffatt or Swern oxidation to give a saccharide ketoamide as a pivotal precursor for the preparation of glyconolactams, azasugars and L-aldohexoses.[4,5] On the one hand, this ketoamide can be reduced by $NaBH_4$ to give the saccharide amide with 5*S*-chirality that eventually leads to L-aldohexoses.[5] On the other hand, this ketoamide can be converted to *O*-benzylated gluconolactam by consecutive condensation with NH_3/MeOH and reduction with Me_3SiH/BF_3.[4a] Reduction of the *O*-benzylated gluconolactam with $LiAlH_4$, followed by hydrogenolysis of the benzyl groups, has been achieved to afford deoxynojirimycin, an azasugar possessing glucosidase inhibiting activity.[4d]

Saccharide amides can be formed either by hydration of saccharide nitriles,[6] or by ammonolysis of saccharide lactones[4,5] as shown in Fig. 1. In order to discern these two possible reaction pathways, we prepared the saccharide lactone **8a**[4d] and nitrile **9a**.[6d] Lactone **8a** was obtained by Moffatt or Swern oxidation [Me_2SO, $(COCl)_2$] of tetrabenzylglucose **6a**.[4d,5a] On the other hand, **6a** reacted with hydroxylamine to give an oxime, which underwent dehydration on treatment with Ph_3P/CBr_4 to give nitrile **9a**.[6d] Ammonolysis of lactone **8a** in $I_2/NH_{3\ (aq)}$ solution proceeded rapidly (< 20 min), whereas complete hydration of nitrile **9a** under the same con-



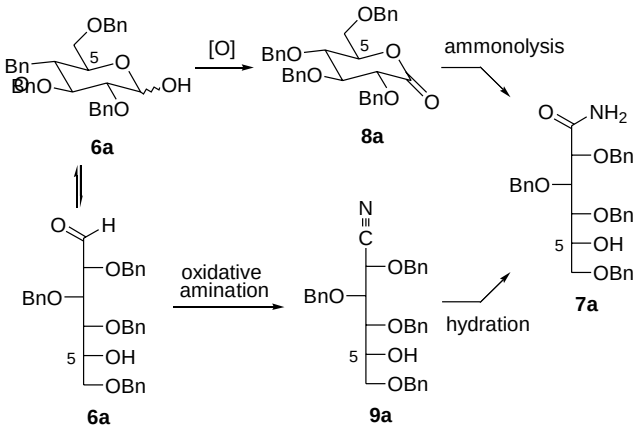


Fig. 1. Two possible reaction pathways for the conversion of aldoses to saccharide amides.

ditions required a prolonged period (18 h).[7] On the other hand, 2,3,4,6-tetrabenzyloxy-5-oxohexanenitrile, the ketonitrile prepared by Swern oxidation of **9a**, did not show any apparent hydration product on stirring in $I_2/NH_{3\,(aq)}$ solution for 24 h.[7]

It is known that aldoses can be oxidized to aldonolactones by using Br_2 or I_2 as the oxidizing agents.[8] For example, *N*-acetylglucosamine and *N*-acetylgalactosamine are oxidized by bromine water to give saccharide lactones.[8a,b] Perbenzyl-maltotriose and maltotetrose react with I_2 in alkaline conditions (KOH, MeOH, 40 °C, 50 min) to give the corresponding lactones.[8c] Peracetylmannose reacts sluggishly (10 days at room temperature) with *N*-iodosuccinimide in CH_2Cl_2 solution to produce mannono-1,5-lactone.[8d] On the basis of these reports and our experimental results, formation of saccharide amides was attributed to ammonolysis of the intermediate lactones. The aldopentoses and aldohexoses used in this study tended to exist as cyclic hemiacetals, which could be oxidized by I_2 (or INH_2)[9] in ammonia water to give saccharide lactones.

This reaction mode for conversion of aldoses to saccharide amides via the intermediacy of aldolactones is different from that found in our previous study[1] for conversion of common aldehydes to nitriles. Except for 2-deoxy-D-ribose, the aldehydes used in our previous study[1] are not in hemiacetal forms. The aldehyde groups may easily condense with NH_3 to form aldimines, which are subsequently oxidized by iodine to give *N*-iodo aldimine intermediates.[10] These *N*-iodo aldimine intermediates may also be formed by condensation of the aldehydes with INH_2. Elimination of HI from these intermediates in ammonia solution would occur rapidly to give the corresponding nitrile products. It is unclear why the reaction of 2-deoxy-D-ribose with $I_2/NH_{3\,(aq)}$ gives a nitrile product, but not the corresponding amide.

In summary, our current method provides a convenient way to convert aldopentoses, aldohexoses and their derivatives to the corresponding saccharide amides by a direct oxidative amidation in $I_2/NH_{3\,(aq)}$. This method shows distinct advantageous features over the previously reported two-step methods:[5,6] (i) The transformation is realized in a simple one-pot procedure, without primary preparations of the intermediary saccharide lactones via Moffatt or Swern oxidations; (ii) Iodine is utilized as a convenient and mild oxidizing agent; (iii) Ammonia water (readily available) is used instead of liquid ammonia; (iv) No organic solvent is required in the reactions with water-soluble aldoses; (v) Protection of the hydroxyl groups in aldoses is not mandatory in this transformation; and (vi) Practically pure saccharide amides are obtained in high yields by extraction with ether or simply by removal of volatiles from the reaction mixture. The products of D-sugar amides are applicable to the preparations of biologically active azasugars and L-sugars.[4,5] As the amide group can be transformed into other functional groups, for example via Hofmann rearrangement to an amino group, the saccharide amides are potentially used as chiral precursors in organic synthesis.

EXPERIMENTAL SECTION

Representative Procedure for the Reactions of Saccharides with Iodine in Ammonia Water

CAUTION: It is known that iodine reacts with ammonia water under certain conditions to give a black powder of nitrogen triiodide monoamine ($NI_3{\cdot}NH_3$).[8b] The dry powder explodes readily by mechanical shock, heat or irradiation. Although we did not have any incidents when handling the reactants in this study, one should avoid using excess reagent.

A solution of 2,3,4,6-tetra-*O*-benzyl-D-glucose (1 mmol) and iodine (1.2 mmol) in ammonia water (10 mL of 28% solution) and THF (1 mL) was stirred at room temperature for the indicated time as monitored by TLC analyses. The dark solution became colorless at the end of reaction. The reaction mixture was charged with aqueous $Na_2S_2O_3$ (0.5 mL of 5% solution), followed by extraction with Et_2O (2 × 15 mL), to give a practically pure product of 2,3,4,6-tetra-*O*-benzyl-D-gluconamide in 82% yield. For the water-soluble substrates, such as D-ribose, the cosolvent (THF) is not needed in the reaction. After the aldose was completely consumed, the reaction mixture was concentrated under reduced pressure, instead of extraction with Et_2O, to give exclusively the desired D-ribonamide[2] (**4a**) as shown by the ^{13}C NMR analysis. Peracetylation of **4a** with Ac_2O in pyridine was carried out to give 2,3,4,5-*O*-tetraacetyl-D-ribonamide (**5a**) for full characterization.[3]

All the products **4a-c**, **5a-f** and **7a-c** are known compounds.

D-Ribonamide[2] (4a)

^{13}C NMR (CD_3OD, 75 MHz) δ 63.0, 69.0, 69.4, 70.8, 178.6.

D-Arabinamide[2] (4b)

^{13}C NMR (D_2O, 75 MHz) δ 64.7, 72.2, 72.4, 73.0, 180.0.

D-Xylonamide[2] (4c)

^{13}C NMR (D_2O, 75 MHz) δ 63.8, 70.8, 73.8, 74.1,

180.0.

2,3,4,5-Tetra-*O*-acetyl-D-ribonamide[3] (5a)

$[\alpha]_D^{25}$ -32.6 (*c* 2.0, $CHCl_3$), lit.[3] -35.5 ($CHCl_3$). ^{1}H NMR ($CDCl_3$, 200 MHz) δ 2.04 (3H, s), 2.06 (3H, s), 2.08 (3H, s), 2.10 (3H, s), 3.84 (1H, dd, *J* = 11.7, 7.4 Hz), 4.27 (1H, dd, *J* = 11.7, 5.1 Hz), 5.21-5.35 (2H, m), 5.71 (1H, dd, *J* = 10.3, 2.1 Hz), 5.90 (1H, br s, NH), 5.98 (1H, br s, NH). ^{13}C NMR ($CDCl_3$, 50 MHz) δ 20.1, 20.2, 20.3, 20.5, 61.4, 68.1, 71.8, 72.0, 167.9, 169.1, 169.5, 169.6, 170.1.

2,3,4,5-Tetra-*O*-acetyl-D-arabonamide[6a,6c] (5b)

$[\alpha]_D^{25}$ +21.2 (*c* 3.0, $CHCl_3$), lit.[6a] +24.3 ($CHCl_3$). IR (KBr) 3454, 1750, 1699, 1374 cm^{-1}. ^{1}H NMR ($CDCl_3$, 200 MHz) δ 1.97 (3H, s), 1.98 (3H, s), 2.01 (3H, s), 2.11 (3H, s), 4.02 (1H, dd, *J* = 12.7, 4.9 Hz), 4.16 (1H, dd, *J* = 12.7, 2.7 Hz), 5.08 (1H, m), 5.35 (1H, d, *J* = 2.4 Hz), 5.62 (1H, dd, *J* = 9.0, 2.4 Hz), 6.27 (1H, br s), 6.58 (1H, br s). ^{13}C NMR ($CDCl_3$, 50 MHz) δ 20.3, 20.4, 20.5, 20.6, 61.6, 67.8, 68.9, 70.8, 169.0, 169.4, 169.5, 169.7, 170.5.

2,3,4,5-Tetra-*O*-acetyl-D-xylonamide[6a,6c] (5c)

IR (KBr) 3449, 1750, 1699, 1375 cm^{-1}. ^{1}H NMR ($CDCl_3$, 200 MHz) δ 1.95 (3H, s), 1.97 (3H, s), 2.02 (3H, s), 2.13 (3H, s), 3.96 (1H, dd, *J* = 12.1, 6.0 Hz), 4.24 (1H, dd, *J* = 12.1, 4.3 Hz), 5.19-5.27 (2H, m), 5.51 (1H, dd, *J* = 6.1, 4.1 Hz), 6.47 (1H, br s), 6.55 (1H, br s). ^{13}C NMR ($CDCl_3$, 50 MHz) δ 20.2, 20.3, 20.4, 20.5, 61.7, 69.1, 69.4, 71.2, 169.0, 169.4, 169.5, 169.9, 170.4.

2,3,4,5,6-Penta-*O*-acetyl-D-gluconamide[6a,6b] (5d)

Mp 181-183 °C, lit.[6b] 184-185 °C. $[\alpha]_D^{25}$ +26.1 (*c* 1.25, $CHCl_3$), lit.[6b] +23.6 ($CHCl_3$). IR (KBr) 3414, 1758, 1699, 1377 cm^{-1}. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 2.01 (3H, s), 2.03 (3H, s), 2.04 (3H, s), 2.09 (3H, s), 2.20 (3H, s), 4.12 (1H, dd, *J* = 12.3, 5.6 Hz), 4.33 (1H, dd, *J* = 12.3, 3.9 Hz), 5.05 (1H, m), 5.32 (1H, d, *J* = 5.1 Hz), 5.46 (1H, dd, *J* = 6.2, 5.1 Hz), 5.65 (1H, t, *J* = 5.1 Hz), 5.86 (1H, br s), 6.16 (1H, br s). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 20.2, 20.4 (2 ×), 20.6 (2 ×), 20.7, 61.6, 68.8, 69.4, 71.2, 168.6, 169.3, 169.6, 169.9 (2 ×), 170.7. HRMS calcd for $C_{16}H_{24}NO_{11}$ (M + H) 406.1349; found 406.1330.

2,3,4,5,6-Penta-*O*-acetyl-D-galactonamide[6a,6b] (5e)

Mp 169-170 °C, lit.[6a] 166-167 °C. $[\alpha]_D^{25}$ +25.6 (*c* 1.8, $CHCl_3$), lit.[6a] +27 ($CHCl_3$). IR (KBr) 3441, 1750, 1679, 1378 cm^{-1}. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.99 (3H, s), 2.03 (3H, s), 2.04 (3H, s), 2.05 (3H, s), 2.15 (3H, s), 3.80 (1H, dd, *J* = 11.6, 7.3 Hz), 4.22 (1H, dd, *J* = 11.6, 5.1 Hz), 5.19-5.22 (2H, m), 5.29 (1H, dd, *J* = 9.9, 1.9 Hz), 5.64 (1H, dd, *J* = 10.0, 1.9 Hz), 6.10 (1H, br s), 6.33 (1H, br s). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 20.3, 20.4, 20.5, 20.6, 20.7, 62.0, 67.3, 67.4, 67.8, 70.7, 168.9, 169.3, 169.6, 169.7, 170.3, 170.4. HRMS calcd for $C_{16}H_{24}NO_{11}$ (M + H) 406.1349; found 406.1332.

2,3,4,5-Tetra-*O*-acetyl-D-fuconamide[11] (5f)

^{1}H NMR (DMSO-d_6, 400 MHz) δ 1.05 (3H, d, *J* = 6.5 Hz), 1.95 (3H, s), 1.96 (3H, s), 2.06 (3H, s), 2.07 (3H, s), 4.99 (1H, m), 5.07 (1H, dd, *J* = 9.8, 2.0 Hz), 5.51 (1H, dd, *J* = 9.8, 2.0 Hz), 7.32 (1H, br s), 5.79 (1H, br s). ^{13}C NMR (DMSO-d_6, 75 MHz) δ 16.0, 20.3 (2 ×), 20.6, 20.9, 66.6, 68.2, 70.0, 70.6, 168.1, 168.6, 169.6 (2 ×), 169.8.

2,3,4,6-Tetra-*O*-benzyl-D-gluconamide[4a,4d] (7a)

^{1}H NMR ($CDCl_3$, 300 MHz) δ 3.60 (1H, dd, *J* = 9.9, 5.2 Hz), 3.67 (1H, dd, *J* = 9.9, 3.0 Hz), 3.88 (1H, dd, *J* = 7.3, 5.6 Hz), 3.94 (1H, m), 4.10 (1H, dd, *J* = 5.0, 3.3 Hz), 4.26 (1H, d, *J* = 3.3 Hz), 4.48-4.67 (5H, m), 4.70 (1H, d, *J* = 8.2 Hz), 4.74 (1H, d, *J* = 8.2 Hz), 5.92 (1H, br s), 6.63 (1H, br s), 7.23-7.43 (20H, m). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 71.1, 71.3, 73.3, 73.7, 74.1, 75.2, 77.7, 79.7, 80.5, 127.6, 127.6, 127.7, 127.8, 127.9, 128.2, 128.3, 128.4, 128.5, 136.7, 137.7, 138.0, 138.2, 174.2.

2,3,4,6-Tetra-*O*-benzyl-D-galactonamide[4d] (7b)

^{1}H NMR ($CDCl_3$, 300 MHz) δ 3.53 (1H, dd, *J* = 9.3, 6.4 Hz), 3.60 (1H, dd, *J* = 9.3, 6.4 Hz), 3.90 (1H, dd, *J* = 8.0, 1.2 Hz), 4.16 (1H, dd, *J* = 7.3, 5.4 Hz), 4.20 (2H, s), 4.35-4.72 (8H, m), 5.93 (1H, br s), 6.64 (1H, br s), 7.18-7.34 (20H, m). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 69.2, 71.4, 73.1, 73.2, 73.6, 75.0, 77.2, 79.4, 79.8, 127.4, 127.7, 127.8, 128.0, 128.1, 128.3, 128.6, 136.7, 137.8, 137.9, 13.8.0, 174.5.

2,3,4,6-Tetra-*O*-benzyl-D-mannonamide[4d] (7c)

^{1}H NMR ($CDCl_3$, 300 MHz) δ 3.64-3.68 (2H, m), 3.88 (1H, dd, *J* = 7.1, 5.7 Hz), 4.02 (1H, m), 4.13 (1H, dd, *J* = 5.5, 3.8 Hz), 4.33 (1H, d, *J* = 3.8 Hz), 4.49 (1H, dd, *J* = 5.7, 3.3 Hz), 4.50-4.54 (4H, m), 4.57-4.68 (2H, m), 4.72 (2H, s), 5.73 (1H, br s), 6.59 (1H, br s), 7.17-7.39 (20H, m). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 70.9, 71.2, 72.7, 73.4, 74.4, 74.6, 78.9, 80.0, 81.2, 127.6, 127.7, 127.8, 127.9, 128.0, 128.3, 128.4, 128.5, 137.1, 138.1, 138.3, 173.4.

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