Enantiospecific syntheses of (R)- and (S)-proline and some derivatives from D-glucono-1,5-lactone

Claudio Mazzini, Letizia Sambri, Henk Regeling, Binne Zwanenburg and Gordon J. F. Chittenden*

a Department of Organic Chemistry, NSR Center for Molecular Structure, Design and Synthesis, The University, Toernooiveld, 6525 ED Nijmegen, The Netherlands
b Groupe ‘Biocatalyse et Chimie Fine,’ Faculté des Sciences de Luminy, Case 901-163, Avenue de Luminy, 13288 Marseille, France
c Dipartimento di Chimica Organica ‘A Mangini’ Università di Bologna, Viale Risorgimento 4, 40136 Bologna, Italy

Carbohydrate-based enantiospecific syntheses of (R)-proline 1 and (S)-proline 2 from the previously reported D-erythro-hexonate ester 9 are described. Azide-substitution reactions on appropriately activated intermediates derived from ester 9, followed by reductive cyclization (H₂/Pd–C), gave the substituted pyrrolidines 14 and 22, which were converted into their corresponding N-Cbz derivatives 16 and 24 in conventional manner. Mild acidic hydrolysis of these, followed by oxidation (sodium metaperiodate), gave the protected prolinals 3 and 4, which on further oxidation (sodium chlorite), followed by catalytic hydrogenolysis, gave the prolines 1 and 2. The N-Cbz-prolino derivatives 5 and 6 are also reported.

Introduction

The stereospecific syntheses of enantiomerically pure compounds, especially those of natural origin, is an active area of modern organic chemistry. The synthesis of prolines and their derivatives seems currently of interest, in particular for the introduction of functional groups and for the application of modern organic chemistry. The synthesis of prolines and their related protected derivatives is an active area of interest.

The stereospecific syntheses of enantiomerically pure compounds 1 and 2 produced racemic mixtures which were unsuccessful. Catalytic hydrogenation of compound 13 over palladium charcoal (10%) proceeded with concomitant ring closure to produce the substituted pyrrolidine 14, which on treatment with toluene-p-sulfonyl chloride (TsCl)–triethylamine gave the crystalline N-toluene-p-sulfonate ester 15. Treatment of compound 14 in aq. ethanol with benzyl chloroformate in the presence of sodium hydrogen carbonate in the usual manner gave the corresponding N-benzylxoyoxazaborinocarbonyl (N-Chz) derivative 16. Reduction of aldehyde 3 with sodium borohydride in methanol produced the corresponding (R)-proline derivative 5.

Oxidation of aldehyde 3 with sodium chlorite, in the presence of 2-methylbut-2-ene as a chlorine scavenger, was rapid and proceeded smoothly to give the protected (R)-proline derivative 7, which on catalytic hydrogenolysis provided pure (R)-proline 1 (Scheme 1).

Treatment of the bromoester 18 with sodium azide in N,N-dimethylformamide (DMF) gave the corresponding azide 19, which on catalytic hydrogenation (H₂/Pd–C) gave the protected (R)-proline derivative 10 and further oxidation (sodium metaperiodate) gave the protected proline 11.

Results and discussion

Treatment of the toluene-p-sulfonate ester 10 with sodium azide in N,N-dimethylformamide (DMF) gave the corresponding pyrrolidine 12. The reaction mixture produced after 18 h indicated the presence of epimeric amines, produced during the initial phase of the displacement reaction, on remaining compound 11 in toluene with diisobutylaluminum hydride (DIBAL) at -78 °C yielded the pure, but unstable aldehyde 13. Attempts to form a stable crystalline phenylhydrzone, or semicarbazone, of compound 13 were unsuccessful. Catalytic hydrogenation of compound 13 over palladium charcoal (10%) proceeded with concomitant ring closure to produce the substituted pyrrolidine 14, which on treatment with toluene-p-sulfonyl chloride (TsCl)–triethylamine gave the crystalline N-toluene-p-sulfonate ester 15. Treatment of compound 14 in aq. ethanol with benzyl chloroformate in the presence of sodium hydrogen carbonate in the usual manner gave the corresponding N-benzylxoyoxazaborinocarbonyl (N-Chz) derivative 16. Hydrolysis (80% aq. acetic acid) of compound 16 gave the diol 17, which on oxidation with aq. sodium metaperiodate in methanol gave the protected (R)-proline derivative 5. Reduction of aldehyde 3 with sodium borohydride in methanol produced the corresponding (R)-proline derivative 5.

Oxidation of aldehyde 3 with sodium chlorite, in the presence of 2-methylbut-2-ene as a chlorine scavenger, was rapid and proceeded smoothly to give the protected (R)-proline derivative 7, which on catalytic hydrogenolysis provided pure (R)-proline 1 (Scheme 1).

Treatment of the bromoester 18 with sodium azide in DMF did not proceed satisfactorily. Analysis (TLC and GLC) of the reaction mixture produced after 18 h indicated the presence of not only the expected azido derivative 19 but also its diastereomer 11 (vide supra), in the ratio 3:2, indicating that epimerization was occurring during the course of the substitution reaction. This was probably due to nucleophilic attack of bromide ions, produced during the initial phase of the displacement reaction, on remaining compound 18, with subsequent and concurrent replacement of both by azide ions (Scheme 2).

The required compound 19 was obtained successfully by treatment of bromide 18 with a solution of lithium azide in DMF at room temp. for 7 days. Lithium azide is much more
sulphonic esters was also converted into the corresponding unstable aldehyde \( \text{A} \) which on catalytic hydrogenation (palladized charcoal, 10%) yielded the known, commercially available \( \text{B} \), derived from reduction of esters of (S)-proline. It had been implied \(^{16}\) that amino aldehydes derived from chiral amino acids could be difficult to obtain with high optical purity. Compound \( \text{C} \) has not been described hitherto. The two enantiomeric aldehydes \( \text{D} \) and \( \text{E} \) described here probably have very high optical purities in view of their mode of synthesis. These two compounds could be stored (0°C) for appreciable periods of time (2–3 months) without any obvious deterioration or racemisation.

Further oxidation of aldehyde \( \text{F} \) with sodium chlorite in the same manner as described for its enantiomer \( \text{C} \) (vide supra) yielded the known, commercially available \( \text{G} \), which on catalytic hydrogenation (palladized charcoal, 10%) yielded (S)-proline.

The syntheses described illustrate the useful application of the ester \( \text{H} \) as a chiral synthon. Further studies on the use of the aldehydes \( \text{I} \) and \( \text{J} \) as sources of novel chiral ligands is currently in progress.

**Experimental**

Optical rotations were determined with a Perkin-Elmer model 241 automatic polarimeter on 1% solutions in chloroform at 25°C, unless indicated otherwise. TLC on pre-coated plates of silica gel (Merck) was performed with light petroleum-ethyl acetate (1:1). Detection was affected by spraying with 0.1 m KI/2% CrO\(_3\) in 0.05 m H\(_2\)SO\(_4\) and heating at 140°C. Column chromatography and flash-column chromatography were performed on silica gel 60 and 60 H with the solvent mixtures indicated. GLC was performed with a Hewlett-Packard 5890 gas chromatograph; a fused-silica capillary column (25 m) coated with HP-1 cross-linked methyl silicone gumpphase operating at 100–150°C (\( t = 0 \) min, 100°C isothermal; \( t = 5 \) min, 5°C min\(^{-1}\)) and nitrogen as the carrier gas at 2 ml min\(^{-1}\) was used. \(^1\)H NMR spectra were recorded with a Bruker AC 100 (100 MHz) or Bruker AC 300 (300 MHz) spectrometer on solutions in CDCl\(_3\) (internal Me\(_2\)Si) or D\(_2\)O or as indicated. \( \delta \)-Values are given in Hz. \(^{13}\)C NMR spectra were recorded with Bruker AC 100, AC 300 or AM 400 spectrometers operating at 25, 75 and 100.6 MHz respectively on solutions in CDCl\(_3\) (internal Me\(_2\)Si) or D\(_2\)O (external 1,4-dioxane at \( \delta = 67.8\)). Mass spectra were recorded using a double-focus VG 7070E.
spectrometer or a Varian Saturn 2 GC-MS ion-trap system. IR spectra were determined on a Perkin-Elmer 298 spectrometer as indicated. DIBAL was purchased as a 1 M solution in hexane. Light petroleum is the fraction distilled between 60–80 °C.

Methyl 4-azido-2,3,4-trIDEOxy-5,6-O-isopropylidenedioxy-threo-hexonate ([4R,5S]-methyl 4-azido-5,6-(isopropylidenedioxy)-hexanolate) 11

A stirred mixture of compound 10 (2.4 g, 6.65 mmol) and sodium azide (1.28 g, 19.7 mmol) in DMF (50 ml) was heated for 12 h at 100 °C, cooled and then treated with ice-water (200 ml). The mixture was extracted with diethyl ether (2 × 150 ml) and the combined extracts were washed successively with saturated aq. sodium chloride (2 × 20 ml), water (2 × 20 ml), sodium azide (1.28 g, 19.7 mmol) in DMF (50 ml) was heated (2 × 150 ml) and the combined extracts were washed successively with saturated aq. sodium chloride (2 × 20 ml) and water (2 × 150 ml) and the combined extracts were washed successively with saturated aq. sodium chloride (2 × 20 ml) and water (2 × 150 ml), dried (MgSO₄), and concentrated in vacuo. Column chromatography (ethyl acetate–light petroleum, 3:1) of the resultant material gave aldehyde 13 (1.04 g, 82%) as an oil. NMR (CDCl₃, 400 MHz, δ ppm): 3.28 (m, 1 H, H-4), 2.50 (m, 2 H, H-2), 1.78 (m, 2 H, H₃-3) and 1.48 and 1.37 (2 H, each 3 H, CMe₂); δ_{f} (CDCl₃) 173.08, 78.49, 66.37, 62.73, 51.78, 30.32, 26.34, 25.9 and 25.17; m/z (KBr/cm²): 228 (M⁺ – 15, 11%), 216 (3.7), 130 (11.7), 101 (81), 87 (13), 59 (21) and 43 (100); v_{max}(neat)/cm⁻¹: 2880, 2710, 2100 and 1710.

(5R,4'S)-5-(2′,2′-Dimethyl-1′,3′-dioxolan-4′-yl)pyrrolidin-2-one 12

A solution of compound 11 (0.604 g, 2.49 mmol) in methanol (20 ml) was treated with palladized charcoal (10%; 60 mg) and was then hydrogenated (1 atm) at room temp. The inorganic material was removed by filtration and washed with methanol (20 ml), and the combined filtrate and washings were concentrated in vacuo to give an oil which crystallized on storage (48 h). Recrystallization (diisopropyl ether–dichloromethane) gave pure lactam 12 (0.23 g, 63%), mp 102–104 °C; [α]_D = 54 (Found: C, 58.27; H, 8.11; N, 7.45. C₉H₇N₃O₄ requires C, 58.36; H, 8.16; N, 7.56%); δ_{f}(100 MHz; CDCl₃) 6.48 (br s, 1 H, NH), 4.03–3.67 (m, 4 H, H-4′, -5 and H-5′), 2.3–1.95 (m, 4 H, H₂-2 and -4) and 1.42 and 1.33 (2 s, each 3 H, CMe₂); v_{max}(KBr)/cm⁻¹: 13260, 1690 and 1650.

(4R,5S)-4-Azido-5,6-(isopropylidenedioxy)hexanolate 13

A solution of DIBAL (7.1 ml) was added dropwise to a stirred, cooled (−78 °C) solution of compound 11 (1.44 g, 5.9 mmol) in light petroleum–toluene (25 ml, 1:1) maintained under nitrogen. The mixture was stirred for a further 1 h at the same temperature, treated with sodium sulfite decarhydrate (1.5 g), diluted with dichloromethane (25 ml) and stirred at room temp. for 1 h. The mixture was then treated with anhydrous sodium sulfate, and filtered, the inorganic material was washed with dichloromethane, and the combined filtrate and washings were washed with water (2 × 20 ml), dried (NaSO₄), and concentrated in vacuo. Column chromatography (light petroleum–ethyl acetate, 1:1) of the resultant material gave aldehyde 13 (1.04 g, 82%) as an oil, [α]_D = 104; δ_{f}(CDCl₃) 9.81 (s, 1 H, CHO), 4.10 (m, 2 H, H-5, H-6′), 3.82 (dd, J 6 and 8, 1 H, H₆-3), 2.32 (m, 1 H, H-4), 2.68 (m, 2 H, H₂-2), 1.84–1.70 (m, 2 H, H₂-3) and 1.47 and 1.38 (2 H, each 3 H, CMe₂); δ_{f}(CDCl₃) 200.73, 110.12, 78.53, 66.33, 62.71, 40.22, 26.32, 25.13 and 23.01; m/z: 186 (M⁺ + 1 – 28, 2.8%), 101 (100), 82 (44), 55 (12) and 43 (97); v_{max}(neat)/cm⁻¹: 2880, 2710, 2100 and 1710.

(2R,4’S)-2-(2′,2′-Dimethyl-1′,3′-dioxolan-4′-yl)pyrrolidin-2-one 14

A solution of compound 13 (0.442 g, 2.07 mmol) in methanol (25 ml) was treated with palladized charcoal (10%; 45 mg) and was then hydrogenated (1 atm) at room temp. for 7 h. The inorganic material was removed by filtration and washed with methanol (10 ml). The combined filtrate and washings were concentrated in vacuo at 45 °C to give the pyrrolidone 14 (0.34 g, 96%). A portion of the product (172 mg) was distilled in vacuo (Kügelrohr, 80 °C/0.25 mbar) to give pure compound 14 (130 mg, 76%); [α]_D = +9 (Found: C, 62.84; H, 10.41; N, 7.36. C₁₁H₁₈N₃O₄ requires C, 63.12; H, 8.01%; N, 7.18%; δ_{f}(CDCl₃) 3.99 (m, 2 H, H-5), 3.64 (m, 1 H, H-4′), 3.09–2.89 (m, 3 H, H₂), 2.10 (br s, 1 H, NH), 1.85–1.67 (m, 3 H, CH₃) and 1.42 and 1.36 (2 H, each 3 H, CMe₂); δ_{f}(CDCl₃) 109.13, 79.40, 67.11, 60.95, 46.30, 27.43, 26.77, 25.37 and 25.24; m/z: 172 (M⁺ + 1, 8.4%), 156 (3.3), 70 (100) and 43 (31); v_{max}(film)/cm⁻¹: 3330, 2940, 2850 and 1690.

A portion of the above material (98.4 mg, 0.575 mmol) in methanol (2 ml) containing trimethylamine (0.163 ml, 1.17 mmol) was treated with TsCl (134 mg, 0.702 mmol), set aside at room temp. for 4 h, and processed in the usual manner. Recrystallization (light petroleum) of the resultant crude crystalline material (151 mg) gave the N-toluene-p-sulfonate 15 (125 mg, 67%), mp 95–97 °C; [α]_D = 86 (Found: C, 58.67; H, 6.83; N, 4.31; S, 9.78. C₁₅H₁₈N₃O₃S requires C, 59.05; H, 7.12; N, 4.30; S, 9.85%); δ_{f}(CDCl₃) 7.72 and 7.32 (2 d, each 3 H, ArH), 4.21 (q, J 6.3, 1 H, H-4′), 4.13 (dd, J 8.5 and 6.3, 1 H, H-5′), 3.98 (dd, J 8.5 and 6.3, 1 H, H-5′), 3.72 (m, 1 H, H-2), 3.41 (m, 1 H, H-5), 3.18 (m, 1 H, H₆-5), 2.43 (s, 3 H, C₂H₃Me), 1.90

† 1 bar = 10⁵ Pa.
(2R,4'S)-N-Benzoylcarbonyl-2-(2',2'-dimethyl-1',3'-dioxolan-4'-yl)pyrrolidine 16

Treatment of a stirred solution of free amine 14 (336 mg, 1.97 mmol) in 50% aq. ethanol (25 ml), containing sodium hydrogen carbonate (336 mg), with benzyl chlororormate (0.45 ml, 3.2 mmol) followed by processing in the usual manner and column chromatography (hexane-ethyl acetate, 3:1) of the resulting material gave compound 16 (491 mg, 82%), \[^{1}H\]NMR and \[^{13}C\]NMR were as described above for compound 14 gave the N-toluene sulfonate (109 mg, 57%), mp 71–72.5 °C (from light petroleum); \[^{1}J\]NMR and \[^{13}J\]NMR were as described above for compound 14.

Methyl 4-azido-2,3,4-trideoxy-5,6-O-isopropylidene-2-erythro-hexonate ([4S,5S]-methyl-4-azido-5,6-(isopropylidenedioxy)hexanate) 19

A stirred solution of compound 18 (2.85 g, 10.2 mmol) in dry DMF (24 ml) was treated with lithium azide (2.45 g, 50 mmol) and then was set aside at room temp. for 7 days. The mixture was treated with a further portion of diethyl ether (100 ml) and the combined ether layers were washed successively with saturated aq. sodium chloride and water, dried (Na$_2$SO$_4$), and concentrated in vacuo. Flash column chromatography (hexane-ethyl acetate, 3:1) of the resultant residue gave pure (GLC) compound 19 (158 mg, 65%) as an oil, \[^{1}H\]NMR and \[^{13}C\]NMR as described above for compound 14. Compound 19 was recrystallized from diisopropyl ether–methanol (8:1) to give 19 (0.206 g, 59%), mp 125–126 °C (from diisopropyl ether–dichloromethane); \[^{1}J\]NMR and \[^{13}J\]NMR as described above for compound 14.

(5S,4'S)-5-(2',2'-Dimethyl-1',3'-dioxolan-4'-yl)pyrrolidin-2-one 20

Reductive cyclization of compound 19 (0.458 g, 1.89 mmol) in the manner described above for compound 11 gave the lactam 20 (0.206 g, 59%), mp 125–126 °C (from diisopropyl ether–dichloromethane); \[^{1}J\]NMR and \[^{13}J\]NMR as described above for compound 14.

(4S,5S)-4-Azido-5,6-(isopropylidenedioxy)hexan-2-one 21

A solution of compound 19 (1.26 g, 5.19 mmol) was treated with DIBAL (6.23 ml) and processed as described above. Column chromatography (light petroleum–ethyl acetate, 3:1) of the resulting material gave the aldehyde 21 (0.85 g, 77%), \[^{1}J\]NMR and \[^{13}J\]NMR as described above for compound 14. Compound 21 was recrystallized from diisopropyl ether–methanol (8:1) to give 21 (0.206 g, 59%), mp 125–126 °C (from diisopropyl ether–dichloromethane); \[^{1}J\]NMR and \[^{13}J\]NMR as described above for compound 14.

(2S,4'S)-2-(2',2'-Dimethyl-1',3'-dioxolan-4'-yl)pyrrolidine 22

A solution of compound 22 (0.206 g, 1.89 mmol) in methanol (20 ml) was hydrogenated (1 atm) in the presence of palladized charcoal (10%; 60 mg) for 6 h and was then processed as described above. Distillation in vacuo (Kugelrohr, 80 °C/0.5 mbar) of the material gave pure compound 22 (0.322 g, 62.5%), \[^{1}J\]NMR and \[^{13}J\]NMR as described above for compound 14.
A stirred solution of compound 17 (405 mg, 1.52 mmol) in a mixture of water (12 ml) and methanol (6 ml) was treated with sodium metaperiodate (325 mg, 1.52 mmol) and set aside in the dark for 2 h. The mixture was extracted with dichloromethane (2 × 20 ml) and the combined extracts were washed with water (20 ml), dried (Na₂SO₄) and concentrated in vacuo. Column chromatography (light petroleum-ethyl acetate, 3:1) of the residue gave the aldehyde 3 (280 mg, 79%), δH (CDCl₃) 9.59 (d, 0.5 H, J 1.6, CHO), 9.49 (d, J = 2.3, 0.5 H, CHO), 7.32 (5 H, PhH), 5.16 (2 H, PhCH₂), 4.30 (m, 0.5 H, H-2), 4.20 (m, 0.5 H, H-3), 3.56 (m, 2 H, H-5), 2.05 (m, 2 H, H-5) and 1.92 (m, 2 H, H-4); δC(CDCl₃) 199.98, 155.23 and 154.37 (1 C), 136.35 + 136.12 (1 C), 128.85, 128.39, 127.99, 67.13, 65.15 + 64.76 (1 C), 47.12 + 46.59 (1 C), 27.67 + 26.47 (1 C) and 24.37 + 23.60 (1 C); m/z 204 (M⁺ – 29, 3.15%), 160 (18.92), 91 (100) and 65 (12.60); v_max(neat/cm⁻¹) 2978, 2880, 1735 and 1694.

Treatment of compound 25 (234 mg, 0.83 mmol) with sodium metaperiodate (189 mg, 0.83 mmol) as described above yielded aldehyde 4 (165 mg, 80%); δH (CDCl₃) 7.65 [lit.36] δH (MeOH); δC(CDCl₃) 9.58 (d, J = 1.7, 0.5 H, CHO), 9.48 (d, 0.5 H, J = 2.3, CHO), 7.32 (5 H, PhH), 5.16 (2 H, PhCH₂), 4.28 (m, 0.5 H, H-2), 4.19 (m, 0.5 H, H-3), 3.55 (m, 2 H, H-5), 2.05 (m, 2 H, H-5) and 1.92 (m, 2 H, H-4); δC(CDCl₃) 199.88, 155.24 + 154.37 (1 C), 136.37, 128.40, 127.93, 67.14, 65.17 + 64.77 (1 C), 47.20 + 46.67 (1 C), 27.70 + 26.49 (1 C) and 24.39 + 23.62 (1 C); m/z 204 (M⁺ – 29, 3.67%), 160 (19.80), 91 (100) and 65 (12.62); v_max(neat/cm⁻¹) 2978, 2860, 1720 and 1690.

A solution of compound 7 (131 mg, 0.53 mmol) in methanol (10 ml) was treated with palladium on charcoal (10%; 13 mg) and the mixture was hydrogenated (1 atm) for 5 h. The insoluble material was removed by filtration, then washed with methanol, and the combined filtrate and washings were concentrated in vacuo to give compound 1 (61 mg, 96%), mp 220°C (from EtOH); δH (CDCl₃) 8.09 (water); δC(CDCl₃) 8.45 (1 C), 7.52 (H-3), 7.32 (H-8), 5.52 (m, 1 H, H-2), 3.52 (m, 2 H, H-5), 2.19 (m, 2 H, H-3) and 1.96 (m, 2 H, H-4); δC(CDCl₃) 178.01 + 175.99 (1 C), 156.15 + 154.34 (1 C), 136.46 + 136.17 (1 C), 128.51, 128.39, 128.17, 127.96, 127.89 + 127.67 (1 C), 67.64 + 67.10 (1 C), 59.37 + 58.56 (1 C), 46.09 + 46.71 (1 C), 34.98 + 34.87 (1 C) and 24.30 + 23.45 (1 C); m/z 249 (M⁺ – 1.84%), 160 (13.82), 114 (32.49), 91 (100), 70 (12.45), 65 (12.40) and 39 (5.19); v_max(KBr)/cm⁻¹ 3010, 2980, 2835 and 1780.

Treatment of compound 2 (140 mg) in a mixture of 2-propanol (8 ml) and methanol, and the combined extracts were dried (Na₂SO₄) and concentrated in vacuo to give compound 2 (144 mg, 99%), mp 224°C (from EtOH); δH (CDCl₃) 8.46 (1 C), 7.52 (H-3), 7.32 (H-8), 5.52 (m, 1 H, H-2), 3.52 (m, 2 H, H-5), 2.19 (m, 2 H, H-3) and 1.96 (m, 2 H, H-4); δC(CDCl₃) 178.09 + 175.93 (1 C), 156.00 + 154.36 (1 C), 136.45 + 136.21 (1 C), 128.50, 128.38, 128.14, 127.94, 127.87 + 127.66 (1 C), 67.57 + 67.14 (1 C), 59.32 + 58.57 (1 C), 46.89 + 46.67 (1 C), 30.87 + 29.15 (1 C) and 24.27 + 23.43 (1 C); m/z 249 (M⁺ – 2.67%), 160 (18.25), 114 (36.77), 91 (100), 70 (17.48), 65 (17.10) and 39 (12.37); v_max(KBr)/cm⁻¹ 3020, 2980, 2860, 2880 and 1730.

Hydrogenolysis of compound 8 (131 mg, 0.53 mmol) in the presence of palladium charcoal (10%; 13 mg) as described above for compound 7 gave title compound 2 (66 mg, 98%), mp 224°C (from EtOH); δH (CDCl₃) –83 (water); δC(CDCl₃) 3.89 (m, 1 H, H-2), 3.16 (m, 2 H, H-3) and 2.30 –1.96 (m, 4 H, H-3 and –4); δC(CDCl₃) 177.35, 63.97, 48.81, 31.72 and 26.58; v_max(KBr)/cm⁻¹ 3080, 2985, 2940, 2860, 2651, 2480, 1655, 1420 and 1362.

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