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The Total Synthesis of Goniofufurone

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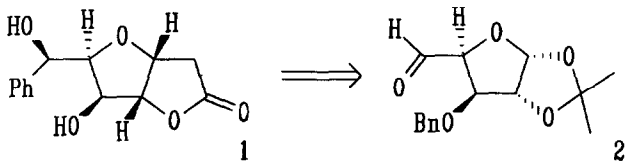
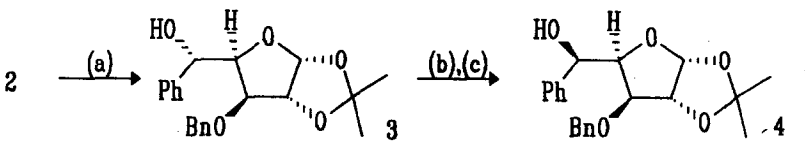
Abstract: The total synthesis of natural (+)-Goniofufurone and related compounds from D-glucose is reported utilising a non-classical Wittig reaction. The factors governing the epimerisation of the tetronate 7 are also discussed.

Goniofufurone, a novel styryllactone isolated from the stem bark of *Goniothalamus giganteus*, and shown to be cytotoxic to human tumour cells,[1] has attracted much recent synthetic attention,[2,3] indeed the total synthesis of its enantiomer by two groups[3] confirmed the absolute configuration of goniofufurone as **1**; this was shortly followed by the total synthesis of **1** by Shing *et al.*[4] We report herein the synthesis[5] of **1** from D-glucose, utilising as the key step a non-classical Wittig cyclisation of a stabilised phosphorane with a butyrolactone.[6] Analysis of **1** indicates that the stereochemistry in the tetrahydrofuran ring is identical to that found in the 1,2-O-isopropylidene-D-xylofuranose derivative **2** and this was chosen as the start point for our synthesis.

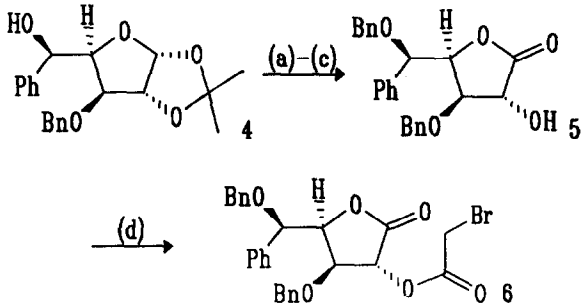


Inch reported[7] that the addition of ethereal phenyl magnesium bromide to **2** (prepared in four steps from D-glucose, 53% overall yield[8]) gave a 78% yield of two alcohols **3** and **4** in a ratio of 14:1 ratio respectively, the minor product **4** possessing the correct stereochemistry for goniofufurone. The reaction proceeds under chelation control and efforts to change the ratio in favour of **4** were unsuccessful (although using phenyl lithium in diethylether gave 3:4 in 2:1 ratio and 60% yield). However access to **4** was possible by oxidation of **3** followed by reduction of the intermediate ketone to give a separable 1:8 mixture in 67% overall yield.



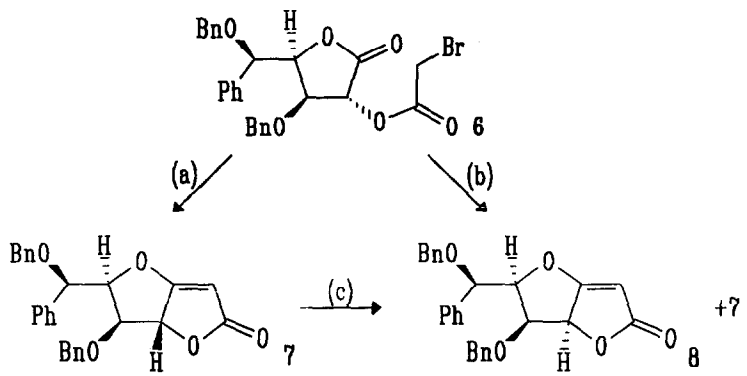
(a) PhMgBr, Et_2O, reflux (78%), 3:4; 16:1 (b) PCC, CH_2Cl_2,
(c) $NaBH_4$, $CeCl_3.7H_2O$, MeOH, -78°C (67%), 3:4; 1:8.

Protection of the C-5-hydroxyl in **4** as a benzyl ether was followed by removal of the acetonide protecting group and bromine oxidation of the resulting hemiacetal to give an α-hydroxy butyrolactone **5**. Bromoacetylation of **5** proceeded smoothly to give **6** in 90% yield suitable for cyclisation.



(a) BnBr, THF, NaH (87%), (b) TFA, H_2O (7:3) (85%),
(c) $Br_2/BaCO_3$, dioxan, H_2O (52%). (d) $BrCOCH_2Br$, Py, Et_2O (90%).

Formation of the phosphonium salt *in situ* followed by base mediated Wittig cyclisation gave the bicyclic tetronic ester **7** in 88% yield. Interestingly if the reaction is performed with a slight excess of base a different product, the bicyclic tetronate **8** in which the C-4 position has epimerised, is formed in 64% yield along with the previously isolated **7** (17%).

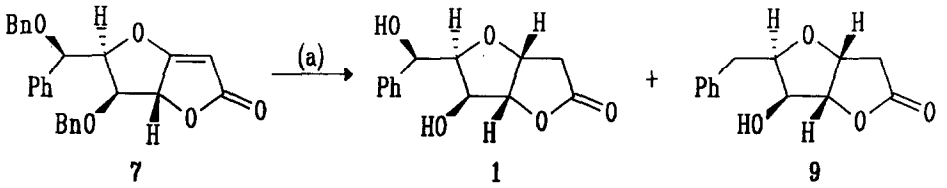


(a) PPh_3, CH_3CN, then DBU, reflux, 30 min (88%), (b) PPh_3, CH_3CN, then excess DBU, reflux, 30 min (81% combined), (c) catalytic DBU, CH_3CN, reflux.

The formation of **8** is interesting insomuch as it would appear to be the thermodynamically less favoured product in which all three substituents are found on the same face of the tetrahydrofuran ring. However on consideration of the nature of the proton source for quenching of the enolate which is likely to be the bulky $DBNH^+$, it is possible that quenching on the face giving rise to the product **7** is the energetically less favourable process due to steric factors experienced by the approaching reagent. This result is supported by the observation that on treatment of a sample of **7** with a catalytic amount of DBN under the cyclisation conditions (conditions (c)) rapid equilibration (<10min) occurs to give a 13:1 mixture of **8** and **7**.

Catalytic hydrogenation of **7** with palladium on charcoal effected removal of both the C3-C4 double bond and the two benzyl protecting groups to give goniofufurone **1** in 58% yield as plates (EtOAc/hexane), m.p. 151-2°C, $[\alpha]_D^{24}$ +8.5° (*c* = 0.8, EtOH), (lit $[\alpha]_D$ +9.0 (*c* = 0.5, EtOH)[1]). A minor product isolated in

22% yield corresponded to 8-deoxygoniofufurone **9** and must originate from a hydrogenolysis reaction (Inch also observed[7] that on prolonged treatment of compound **3** under similar conditions led to hydrogenolysis of the C-5 hydroxyl group to give the corresponding 4-benzylfuranoside).



7 1 9

(a) H_2, 10% Pd on C (58%); (22%)

This synthesis represents a rapid entry (13 steps from D-glucose) to goniofufurone. The easy availability of homochiral butyrolactones[9] together with the ability to control both the nature of the group and the sense of chirality at C-5 in the furanose by Grignard addition, along with the ability to control (by epimerisation) the stereochemistry of C-4 in the bicyclic tetronates will allow preparation of analogues of goniofufurone in which variation at all stereocentres is possible, making this synthetic approach potentially the most flexible to date. The biological activities of compounds prepared within this publication will be reported in due course.

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Experimental

Column chromotography was carried out on Kieselgel (230-400 mesh) with the eluant specified in each case. TLC was conducted on precoated Kieselgel 60 F254 (Art. 5554; Merck) glass plates. All non-aqueous reactions were conducted in oven-dried apparatus under a static atmosphere of argon. Light petroleum refers to the fraction boiling in the range 35-60°C. Dichloromethane, pyridine, diethyl ether, THF and N,N-dimethylformamide (DMF) were dried and distilled before use using standard methods.[10] Chemical shifts are reported as δ values relative to tetramethylsilane as an internal standard. ^{1}H nmr spectra were recorded in deuterochloroform (unless otherwise stated) on a Bruker AC250 spectrometer. IR were recorded as thin films (oils) or as chloroform solutions on a Perkin Elmer 1600 series instrument. Optical rotations were measured from chloroform solutions (unless otherwise stated) using a Perkin Elmer 141 polarimeter. Mass spectra were recorded on a VG Masslab Model 12/253 spectrometer using chemical ionisation (with ammonia as the reagent gas). Accurate mass determinations were recorded on a VG Analytical ZAB-E spectrometer using chemical ionisation (with ammonia as the reagent gas). M.p.s were recorded with a Gallenkamp MF370 apparatus and are uncorrected.

3-O-Benzyl-1,2-O-isopropylidene-5-C-phenyl-α-D-*gluco*-pentafuranose (4).

PCC (3.45 g 16.0 mmol) was added in one portion to a vigorously stirred cooled (0°C) solution of **3** (2.69 g, 7.56 mmol)[7] in dichloromethane (20 ml). After 16 hrs ether (100 ml) was added and the mixture filtered through a silica pad which was throughly washed with further portions of ether (5 x 25 ml). Evaporation of the solvents and redissolution in methanol (30 ml) was followed by the addition of cerium chloride heptahydrate (3.40 g, 9.07 mmol), cooling (-78 °C) and treatment with sodium borohydride (343 mg, 9.07 mmol). After stirring to room temperature (24 hrs) the solution was extracted with ethyl acetate (3 x 50 ml), the extracts dried ($MgSO_4$) and evaporated. Chromatography of the residue (18% ethyl acetate in petrol)

gave **4** (1.65 g, 60%, Rf = 0.23, $[\alpha]_D^{24}$ -77° (*c* = 4.02,), (lit $[\alpha]_D$ -76° (*c* = 1.0)[7].) and recovered **3** (0.20 g, 7%, Rf = 0.12) as oils.

Data for **4**: **^{1}H nmr**; δ 1.32 (3H, s, Me), 1.48 (3H, s, Me), 4.04 (1H, d, J = 3.2 Hz, CH), 4.34 (1H, dd, J = 6.5, 3.2 Hz, CH), 4.49 (1H, d, J = 11.5 Hz, CH), 4.63 (1H, d, J = 3.8 Hz, CH), 4.69 (1H, d, J = 11.5 Hz, CH), 5.09 (1H, d, J = 6.5 Hz, CH), 6.02 (1H, d, J = 3.8 Hz, CH), 7.26-7.43 (10H, m, Ph). **^{13}C nmr**; δ 26.17 (Me), 26.69 (Me), 71.86 (CH), 72.18 (CH_2), 81.73, 82.58, 82.63, 105.12 (all CH), 111.57 (C), 126.16-129.39 (Ph CH's), 136.82, 141.41 (Ph C's). **IR** *ν* max 3488 (OH) 3062, 3031, 2988 (CH).

3,5-Di-O-benzyl-1,2-O-isopropylidene-5-C-phenyl-α-D-*gluco*-pentafuranose.

Sodium hydride (260 mg of a 60% dispersion in oil, 6.5 mmol) was added to a cooled (0°C), stirred solution of **4** (1.543 g, 4.33 mmol) in THF (20 ml). After stirring for 10 min sufficient DMF (10 ml approx) was added to dissolve any precipitates; this was followed by the addition of benzyl bromide (1.43 g, 5.63 mmol). On completion of the reaction (4 hrs as indicated by t.l.c.) saturated ammonium chloride solution (25 ml) was added and the reaction extracted with ether (4 x 50 ml). The extracts were washed with saturated lithium bromide solution (2 x 25ml), dried ($MgSO_4$) and evaporated; chromatography (8% ethyl acetate in petrol, Rf = 0.17) gave 3,5-Di-O-benzyl-1,2-O-isopropylidene-5-C-phenyl-α-D-*gluco*-pentafuranose (1.682 g, 3.77 mmol, 87%, $[\alpha]_D^{24}$ -43.6° (*c* = 1.27)) as a solid (m. p. 83-85 °C).

Data: **^{1}H nmr**; δ 1.33 (3H, s, Me), 1.48 (3H, s, Me), 4.30-4.83 (8H, m, 4 CH's, 2 CH_2's), 5.93 (1H, d, J = 3.7 Hz, CH), 7.27-7.55 (15H, m, Ph). **^{13}C nmr**; δ 26.18 (Me), 26.66 (Me), 70.20, 72.31 (2 CH_2'), 78.15, 81.75, 82.04, 82.80, 104.92 (all CH), 111.40 (C), 127.40-128.33 (Ph CH's), 137.67, 138.07, 139.26 (Ph C's). **IR**; *ν* max 3087, 3063, 3031, 2988, (CH). **MS**; 464 (12%, M+NH_4^+), 447 (10%, M+H^+), 339 (100%, M^+-BnO). **HRMS**; found 464.2437, $C_{28}H_{34}O_5N$ requires 464.2437.

3,5-Di-O-benzyl-5-C-phenyl-D-*gluco*-pento-1,4-lactone (5).

The previously prepared 3,5-Di-O-benzyl-1,2-O-isopropylidene-5-C-phenyl-α-D-*gluco*-pentofuranose (1.533 g, 3.43 mmol) was dissolved in a trifloroacetic acid/water mixture (7:3, 10 ml) and stirred for 4 hours. The solution was evaporated and the residue subjected to chromatography (40% ethyl acetate in petrol, Rf = 0.4) to give the intermediate hemiacetal (1.19g, 2.93mmol, 85%) as a mixture of anomers. **^{1}H nmr**; δ 2.42 (2H, br s, OH's), 3.40-4.70 (9H, m, 5 CH's, 2 CH_2's), 7.05-7.48 (15H, m, 3 Ph's). Redissolution of this product (1.18 g, 2.90 mmol) in a dioxane/water mixture (2:1, 6 ml) was followed by the addition of finely powdered barium carbonate (1.45 g, 7.32 mmol). After cooling (0°C), bromine (0.3 ml, 940 mg, 5.86 mmol) was added and the resultant solution stirred in the dark at room temperature for 36 hours. After addition of saturated sodium thiosulphate (15 ml) the solution was filtered through a pad of celite which was washed with ethyl acetate (3 x 50 ml); the separated aqueous layer was further extracted with ethyl acetate (2 x 50 ml). These extracts were combined, dried ($MgSO_4$), evaporated and subjected to chromatography (20% ethyl acetate in petrol Rf = 0.21) to give **5** (614.2 mg, 1.52 mmol, 52%, (69% based on recovered starting material), $[\alpha]_D^{24}$ +8.1° (*c* = 0.938,)) as an oil and recovered hemiacetal (290mg, 25%).

Data for **5**: **^{1}H nmr**; δ 2.04 (1H, d, J = 0.4 Hz, OH) 3.88 (1H, dd, J = 7.0, 0.4 Hz, CH), 4.23-4.61 (5H, m, CH, 2 CH_2's), 4.85 (1H, d, J = 3.6 Hz, CH), 4.92 (1H, dd, J = 7.4, 3.6 Hz, CH), 7.19-7.44 (15H, m, Ph). **^{13}C nmr**; δ 71.03 (CH_2), 71.66 (CH), 72.84 (CH_2), 78.80, 88.54, 81.59, (all CH's), 127.94-129.02 (Ph CH's), 136.22, 137.17, 137.71 (Ph C's), 175.16, (C=O). **IR**; *ν* max 3444 (OH), 3087, 3063, 3031, (CH), 1787 (C=O).**MS**; 422 (100%, M+NH_4^+), 405 (15%, M+H^+), 297 (60%, M^+-BnO). **HRMS**; found 422.1967, $C_{25}H_{28}O_5N$ requires 422.1967.

2-Bromoacetyl-3,5-di-O-benzyl-5-C-phenyl-D-*gluco*-penta-1,4-lactone (6).

Pyridine (142 mg, 1.80 mmol) and bromoacetyl bromide (380 mg, 1.88 mmol) were added sequentially to a cooled (0°C), stirred solution of **5** (692.8 mg, 1.713 mmol) in dry ether (10 ml). After 12 hours at room temperature the reaction was quenched by the addition of saturated copper sulphate solution (20 ml) and then extracted with ether (3 x 20 ml). Drying ($MgSO_4$), evaporation and chromatography (15% ethyl acetate in petrol, Rf = 0.18) of the extracts gave **6** (815.0 mg, 1.55 mmol, 90%, $[\alpha]_D^{24}$ +25.6° (*c* = 0.324)) as an oil.

Data for **6**: **^{1}H nmr**; δ 3.82 (2H, s, CH_2), 4.32-4.93 (7H, m 3 CH's, 2 CH_2's), 5.28 (1H, d, J = 6.0 Hz, CH), 7.21-7.53 (15H, m, Ph). **^{13}C nmr**; δ 24.64, 71.18, 72.79 (3 CH_2's), 73.13, 77.63, 78.57, 81.72, (all CH's), 127.83-128.69 (Ph CH's), 136.14, 136.54, 137.35 (Ph C's), 165.72, 169.26, (C=O's). **IR**; *ν* max 3063, 3031, (CH), 1800, 1752 (C=O).

6R,7R,8S-7-Benzyloxy-1,5-dioxa-6-(R-benzyloxybenzyl)-2-oxobicyclo[3.3.0.^{4,8}]oct-3-ene (7).

Triphenylphosphine (176 mg, 0.67 mmol) was added to a stirred solution of **6** (293.3 mg, 0.56 mmol) in dry acetonitrile (5 ml) and the resulting solution stirred under an argon atmosphere at 40°C until TLC indicated full conversion to the phosphonium salt (2 hours). At this point the reaction was cooled (0°C) and DBU (84.0 mg, 0.55 mmol) was added; after stirring for 5 minutes to effect formation of the phosphorane the reaction was heated at reflux for 30 minutes whereupon it was cooled, diluted with ether (50 ml) and filtered through a short (3 cm) silica column which was washed with further ether (3 x 20 ml). Evaporation and chromatography (30% ether in petrol, Rf = 0.15) of the filtrate gave **7** (210.2 mg, 0.492 mmol, 88%, $[\alpha]_D^{24}$ +52.7° (*c* = 0.23)) as an oil.

Data for **7**: **^{1}H nmr**; δ 4.26 (3H, m, 3 CH's) 4.44 (1H, d, J = 11.6 Hz, CH), 4.51 (1H, d, J = 11.6 Hz, CH), 4.65 (1H, d, J = 11.7 Hz, CH), 4.85 (1H, d, J = 2.3Hz, CH), 4.96 (1H, s, vinyl CH), 5.29 (1H, m, CH), 7.22-7.43 (15H, m, Ph). **^{13}C nmr**; δ 70.93, 72.80 (2 CH_2's), 78.72, 78.69, 81.29, 88.37, 92.75, (all CH's), 127.54-129.23 (Ph CH's), 135.52, 137.25, 137.46 (Ph C's), 174.00 (C=O), 182.50 (C). **IR**; *ν* max 3060, 3048, 3036, (CH), 1772 (C=O), 1655 (C=C). **MS**; 446 (10%, M+NH_4^+), 429 (100%, M+H^+), 321 (5%, M^+-BnO). **HRMS**; found 429.1702, $C_{27}H_{25}O_5$ requires 429.1702.

6R,7R,8R-7-Benzyloxy-1,5-dioxa-6-(R-benzyloxybenzyl)-2-oxobicyclo[3.3.0.^{4,8}]oct-3-ene (8).

Triphenylphosphine (525 mg, 2.00 mmol) was added to a stirred solution of **6** (873.3 mg, 1.66 mmol) in dry acetonitrile (15 ml) and the resulting solution stirred under an argon atmosphere at 40°C until TLC indicated full conversion to the phosphonium salt (2 hours). At this point the reaction was cooled (0°C), DBU (255.5 mg, 1.68 mmol) was added and the reaction immediately brought to reflux and kept there for 30 minutes whereupon it was cooled, diluted with ether (100 ml) and filtered through a short (3 cm) silica column which was washed with further ether (3 x 40 ml). Evaporation and chromatography (40% ether in petrol) of the residue gave the previously prepared **7** (120.0 mg, 17%) and **8** (456.4 mg, 64%, Rf = 0.15, $[\alpha]_D^{24}$ +75.9° (*c* = 1.03)) as a solid (Mp 48-49°C).

Data for **8**: **^{1}H nmr**; δ 4.28 (1H, d, J = 11.0 Hz, CH) 4.42 (1H, d, J = 11.0 Hz, CH), 4.63 (3H, m, 3 CH's), 4.84 (1H, d, J = 10.8 Hz, CH), 4.92 (1H, d, J = 10.8 Hz, CH), 4.04 (1H, s, vinyl CH), 5.08 (1H, d, J = 3.0 Hz, CH), 7.19-7.47 (15H, m, Ph). **^{13}C nmr**; δ 70.30, 73.80 (2 CH_2's), 74.86, 76.27, 80.33, 86.96, 93.99, (all CH's), 127.33-128.68 (Ph CH's), 136.52, 137.13, 137.20 (Ph C's), 174.68 (C=O), 182.56 (C). **IR**; *ν* max 3063, 3031, (CH), 1773 (C=O), 1659 (C=C). **MS**; 429 (30%, M+H^+). **HRMS**; found 429.1702, $C_{27}H_{25}O_5$ requires 429.1702. **Microanalysis;** found C = 75.28, H = 5.44; $C_{27}H_{24}O_5$ requires C = 75.67, H = 5.65.

Goniofufurone (1) and 8-deoxygoniofufurone (9) (4R,6R,7R,8S-6-benzyl-1,5-dioxa-7-hydroxy-2-oxobicyclo[3.3.0.^{4,8}]octane).

Palladium on charcoal (10%, 75 mg) was added to a stirred solution of **7** (135 mg) in ethyl acetate (2 ml) the reaction vessel was flushed with hydrogen gas and then stirring continued for 1 hour. At this point the solvent was removed and replaced by ethyl alcohol (3 ml) the reaction vessel was again flushed with hydrogen gas and stirring continued for a further 15 hours. Filtration through a silica pad followed by chromatography (60% ethyl acetate in petrol) gave two products; **goniofufurone 1** (46mg, 58%, Rf = 0.24) as plates (EtOAc/hexane), (m.p. 151-2°C, $[\alpha]_D^{24}$ +8.5° {*c* = 0.8, EtOH}, lit $[\alpha]_D$ +9.0 (*c* = 0.5, EtOH)[1]) and **8-deoxygoniofufurone 9** (16mg, 22%, Rf = 0.31, $[\alpha]_D^{24}$ +37° (*c* = 0.14)) as a solid (m.pt. 101-103°C).

Data for **1**: **^{1}H nmr**; δ 2.66 (1H, dd, J = 18.8, 1.4 Hz CH) 2.73 (1H, dd, J = 18.8, 5.4 Hz, CH), 2.85 (1H, s, OH), 4.08 (1H, dd, J = 2.6, 4.9 Hz, CH), 4.20 (1H, br d, J = 0.5 Hz, OH), 4.40 (1H, d, J = 2.0 Hz CH), 4.85 (1H, d, J = 4.2 Hz CH), 5.10 (1H, m, CH), 5.16 (1H, d, J = 4.9 Hz CH), 7.33-7.43 (5H, m, Ph). **^{13}C nmr**; δ 36.07 (CH_2), 73.60, 74.52, 77.35, 82.95, 87.40 (all CH's), 125.85, 128.52, 128.85 (Ph CH,s), 138.77, (Ph C), 175.20 (C=O). **IR**; 3350 (OH), 1788 (C=O). **MS**; 268 (100%, M+NH_4^+), 250 (15%, M^+), 233 (18%, M^+-OH). **HRMS**; found 268.1185, $C_{13}H_{18}O_5N$ requires 268.1185. **Microanalysis**; found C = 62.18, H = 5.45; $C_{13}H_{14}O_5$ requires C = 62.39, H = 5.64.

Data for **9**: **^{1}H nmr**; δ 2.63 (1H, dd, J = 18.9, 1.2 Hz CH) 2.74 (1H, dd, J = 18.9, 6.1 Hz, CH), 2.95 (1H, dd, J = 13.8, 7.2 Hz CH) 3.02 (1H, dd, J = 13.8, 6.8 Hz, CH), 3.51 (1H, s, OH), 4.15 (2H, m, 2 CH's), 4.88 (1H, d, J =4.7 Hz, CH), 4.98 (1H, ddd, J = 4.7, 6.1, 1.2 Hz CH), 7.05-7.35 (5H, m, Ph). **^{13}C nmr**; δ 34.30, 35.86 (2 CH_2's), 74.09, 76.16, 81.47, 87.62, (all CH's), 126.76, 128.69, 129.04 (Ph CH's), 137.28 (Ph C), 175.57 (C=O). **IR**; 3460 (OH), 1787 (C=O). **MS**; 252 (100%, M+NH_4^+), 235 (10%, M+H^+). **HRMS**; found 252.1236, $C_{13}H_{18}O_4N$ requires 252.1236.

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