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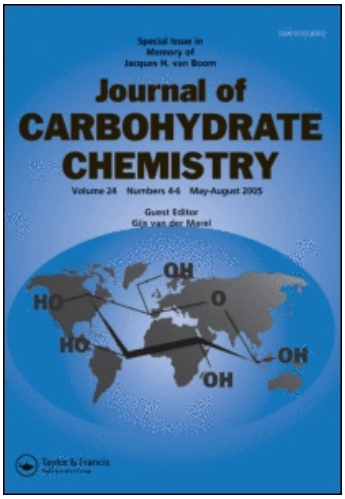

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MAOS of Sugar Phenylosazones and their Derived Pyrazoles and Triazoles

E.S. H. El Ashry[a]; K. F. Atta[a]; S. Aboul-Ela[a]; R. Beldi[a]
[a] Faculty of Science, Department of Chemistry, Alexandria University, Alexandria, Egypt

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MAOS of Sugar Phenylosazones and their Derived Pyrazoles and Triazoles

E. S. H. El Ashry, K. F. Atta, S. Aboul-Ela, and R. Beldi

Faculty of Science, Department of Chemistry, Alexandria University, Alexandria, Egypt

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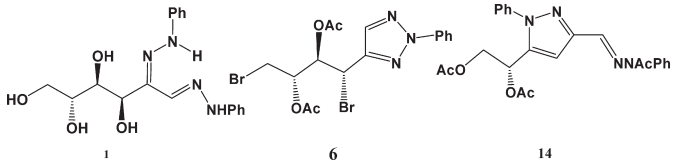
Microwave-assisted organic synthesis (MAOS) has proven to be practical to provide heterocycles from sugar osazones; an efficient method was developed for the characterization of sugars via their osazones **1–4** using microwave irradiation. The microwave-assisted organic synthesis irradiation technique has been applied to convert D-arabino-hexose phenylosazone to 2-phenyl-4-(D-arabinotetrahydroxybutyl-1,2,3-triazole (**5**), which was then oxidized to the corresponding aldehyde whose oxime **9** was transformed to 4-cyano-2-phenyltriazole **10**. The condensation of **7** with thiosemicarbozide gave **10**. Degradation of **1** afforded mesoxaldehyde 1,2-bisphenylhydrazone **11**, which cyclized to 1-phenyl-4-phenylazo-pyrazole (**12**) under acidic conditions. Irradiation of **6** in HBr/AcOH afforded 4-(D-arabino-2′,3′-di-O-acetyl-1′,4′-dibromobutyl)-2-phenyl-2*H*-1,2,3-triazole. The acetylated phenylosazone was converted to furopyridazine **14**. The irradiation of phenylosazone with acetic anhydride in pyridine gave the respective O-acetyl derivative, whereas with boiling acetic anhydride gave the pyrazole **14**, which afforded **15** and **16**.



Keywords Microwave irradiation, Sugar osazones, Phenylosazone, Triazole, Pyrazole

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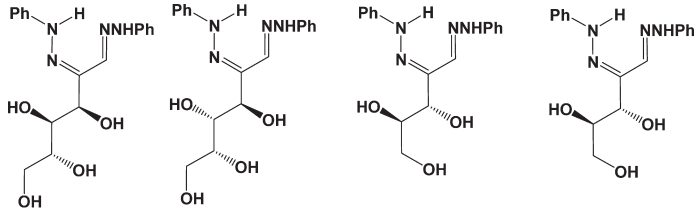
Razika Beldi is on leave from the Ministry of High Education and Scientific Research, Algeria.
Address correspondence to E. S. H. El Ashry, Faculty of Science, Department of Chemistry, Alexandria University, Alexandria, Egypt. E-mail: eelashry60@hotmail.com

INTRODUCTION

The role of carbohydrates as raw materials has received increased interest because of their availability as annually renewable biomasses. The low-molecular-weight carbohydrates are accessible as building blocks for the synthesis of biologically significant chiral natural products[1] and compounds of industrial application profile.[2] The carbohydrate-derived heterocycles have led to the synthesis of a diversity of heterocycles[1–4] and acyclonucleosides.[5,6]

The interest in employing microwave-assisted organic synthesis (MAOS) could lead to low-cost and eco-friendly compounds within the framework of a green chemistry approach, in addition to the enhancement of the regio- and stereoselectivities in the organic reactions. Continuing our efforts on the role of microwave (MW) irradiation in the synthesis of organic compounds,[7,8] and the synthesis of heterocyclic compounds from carbohydrates precursors,[1,3,5,6] this report describes a practical elaboration of sugar phenylosazones and their conversion into various functionalized heterocyclic compounds under MW irradiation.

D-Arabinohexosulose phenylosazone (**1**) is a well-known derivative for the characterization of D-glucose that readily formed upon reaction with phenylhydrazine in the presence of acetic acid. Similarly, the osazone derivatives **2–4** from D-galactose, L-arabinose, and D-xylose have been obtained. Herein we have developed the formation of such osazones under MW irradiation, which proved that this technique can readily lead to the identification of the respective sugars within 2 min of irradiation in a practical way. Moreover, it can be a model experiment in student laboratories.



1 2 3 4

The cyclization of the phenylosazone **1** to 4-(D-arabino-1′,2′,3′,4′-tetrahydroxybutyl)-2-phenyl-2*H*-1,2,3-triazole **(5)**, in 96% yield, was carried out by irradiation of a paste of **1** and copper sulfate using water. Increasing the amount of water gave the same product in 87% yield.

A solution of 4-(D-arabino-1′,2′,3′,4′-tetrahydroxybutyl)-2-phenyl-2H-1,2,3-triazole (**5**) in 33% hydrobromic acid in acetic acid was irradiated for 2 min to afford the new dibromodiacetoxy derivative **6** in 80% yield. Reaction of HBr in acetic acid with alditolyl residues is known[9] to replace one or more of the hydroxyl groups with bromine and to acetylate the others. When the reaction was carried on **5**, a product was obtained whose elemental analysis agreed

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with the molecular formula indicating the introduction of two bromine atoms and acetylating the two other hydroxyls. The two acetyl groups were confirmed from the appearance of two singlets at δ 2.00 and 2.10 ppm in the 500 MHz NMR spectrum. The two doublets of doublet of H-4′ and H-4″ appeared at δ 3.4 and 3.60 ppm, respectively. These peaks are observed at a lower field than that expected for those attached to the OAc group and, similarly, H-1′ appeared at δ 5.40, a lower field than that attached to OAc. Therefore, it can be concluded that the two bromine atoms are attached to C-1′ and C-4′. Moreover, the coupling constant $J_{1',2'} = 4.5$ Hz indicated that inversion of configuration at C-1′ had taken place; otherwise, the coupling constant would be ca. 8 Hz. Moreover, the x-ray analysis of an acyclic analog and the expected mechanism of its formation indicated that an inversion of configuration of the secondary carbon atom would take place during such a reaction.[9]

The degradation of triazolyl-acyclonucleoside **5** by sodium metaperiodate afforded 2-phenyl-1,2,3-triazole-4-carboxaldehyde **(7)**. It was converted, under microwave irradiation for 2 min, to 2-phenyl-1,2,3-triazole carboxaldehyde oxime (**8**) in 88% yield. The oxime, upon irradiation for 3 min, with acetic anhydride supported on bentonite gave 4-cyano-2-phenyl-1,2,3-triazole in 78% yield. The 2-phenyl-1,2,3-triazole-5-carboxaldehyde thiosemicarbazone **(10)** was similarly prepared from the reaction of **7** with thiosemicarbazide upon irradiation for 3 min.

Glycol cleavage of the tetritolyl residue in **1** with sodium metaperiodate gave mesoxaldehyde 1,2-bisphenylhydrazone **(11)**, whose cyclization under MW irradiation required 2 min to afford 1-phenyl-4-phenylazopyrazole **(12)**. Acetylation of **1** was done in the MW oven for 3 min to give tetra-*O*-acetyl-D-arabino-hexosulose phenylosazone **(13)** in 78% yield, which, upon deacetylation with 1.5% aqueous sodium hydroxide under MW irradiation, gave the Percival dianhydro-osazone.[16]

The conversion of D-glucose phenylosazone **(1)** into the respective pyrazole[17] **14** can be done efficiently under microwave irradiation within 5 min to give 87% yield. The microwave irradiation of **14** with hydroxylamine hydrochloride in dry DMSO supported on bentonite for 4 min gave 5-[(1′*S*)-1′,2′-Diacetoxyethyl]-3-cyano-1-phenylpyrazole **(15)** in 73% yield. Hydrazonolysis of **14** with aqueous formaldehyde and acetic acid under MW irradiation gave **16** (Fig. 1).

In conclusion, the present microwave protocol provides an efficient, simple, and rapid method for the synthesis of pyrazole and triazole derivatives from carbohydrates as renewable biomass. Efficient and rapid characterizations of sugars as their osazones have been carried out in a practical manner by using MW irradiation. In addition to the reduced time of reaction, the yields could be improved (Table 1) in an economical and eco-friendly manner, and thus green chemistry can be achieved.

EXPERIMENTAL

Melting points were determined on a Mel-temp apparatus and were uncorrected, and are given in Table 1 with the authentic melting points and

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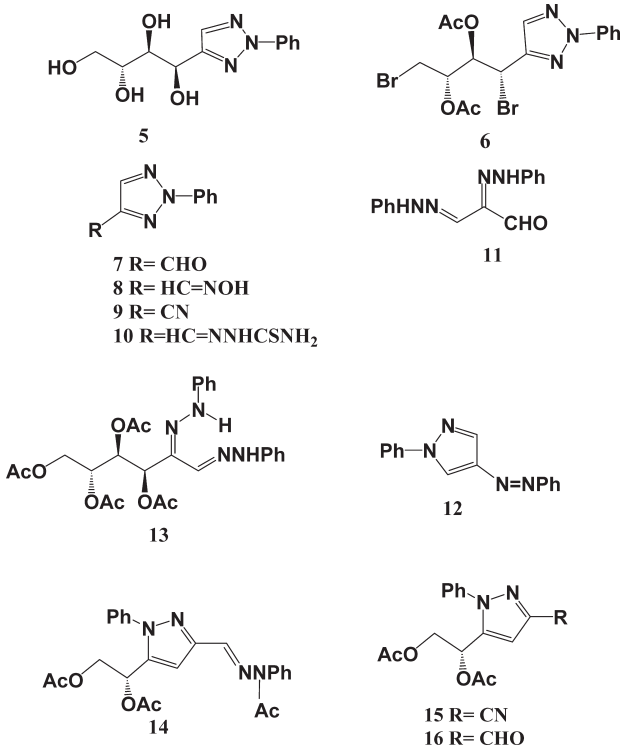

Figure 1: Structure of the prepered compounds.

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literature references. TLC was performed on Baker–Flex silica gel 1B-F plates using n-hexane/ethyl acetate (H/E) as developing solvents and the spots were detected by their characteristic colors and by UV light absorption. Irradiation with MW was done in a domestic microwave oven E.M. 230 M (800-watt output power). The reactions were done in a closed Teflon vessel supported in a tray and placed, at the center of the plate, inside the oven; the scale was adjusted on defrost. ^{1}H NMR and ^{13}C NMR spectra were recorded on a JEOL spectrometer (500 MHz). The chemical shifts are expressed on δ-scale using Me_4Si as a standard, and coupling constant values are given in Hz. The microanalyses were done at the microanalytical unit at Cairo University.

Preparation of Sugar Phenylosazones[12] 1–4 General Procedure

To a solution of sugar (0.3 g) in water (3 mL), phenylhydrazine (0.5 mL) and two drops of glacial acetic acid were added, the mixture was irradiated for 2 min, and the yellow crystalline products were collected, washed successively with water, and dried (Table 1).

Table 1: Comparison of results under microwave (MW) and conventional method (CM).

Comp. No.	Time		Yield (%)		mp°C
	MW (min)	CM (h)	MW	CM	Found/reported
1	2	2	91	87[12]	200/202[10,12]
2	2	0.3	94		203/201[12]
3	2	0.1	88		161–162/164[12]
4	2	0.1	93		168–169/166[12]
5	0.5	2	96	67[13]	198/195–196[13]
6	2		80		100–102
7	3	24	89	84[13]	66/68–69[13]
8	2		88		140
9	3		78		86–88
10	3		84		189–190
11	2	0.25	84	85[14]	194–196/198[14], 198–200[14]
12	2	0.3	90		122–123/122–123[14]
13	3		86		121–124/115–117[15]
14	5	0.7	87	77[11]	130–131/131[17], 132–133[11]
15	4	2	56	67[11]	Syrup
16	6	5	73	86[11]	Syrup

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4-(D-*arabino*-1′,2′,3′,4′-Tetrahydroxybutyl)-2-phenyl-2*H*-1,2,3-triazole[13] (5)

The reaction was carried out by making a paste of D-glucose phenylosazone (0.3 g, 0.84 mmol) and copper sulfate (0.3 g) in water (1 mL), then irradiated for 1 min. The product was extracted with water to give **5** in 96% yield. The reaction was repeated by using a suspension of D-glucose phenylosazone (**1**) (0.3 g, 0.87 mmol) in water (4 mL) and copper sulfate (0.3 g); the mixture was irradiated for 30 sec in the microwave oven, then filtered while hot. The filtrate was concentrated to a small volume and left to cool. The product was recrystallized from water to give colorless needles, R_f 0.35 (E).

4-(D-*allo*-2′,3′-Di-*O*-acetyl-1′,4′-dibromo-butyl)-2-phenyl-2*H*-1,2,3-triazole (6)

A suspension of **5** (0.25 g, 0.94 mmol) in 33% hydrobromic acid in acetic acid (2 mL) was subjected to MW for 2 min. Ice water was added and the product was crystallized from ethanol to afford colorless crystals, R_f 0.67 (H/E 3/1). ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.04, 2.10 (2 s, 6H, 2 × CH_3CO), 3.41 (dd, 1H, $J_{4',4''} = 11.5$ Hz, $J_{4',3'} = 6.1$ Hz, H-4′), 3.60 (dd, 1H, $J_{4'',4'} = 11.5$ Hz, $J_{4'',3'} = 3.8$ Hz, H-4″), 5.17–5.18 (m, 1H, H-3′), 5.40 (d, 1H, $J_{1',2'} = 4.5$ Hz, H-1′), 5.82 (dd, 1H, $J_{2',1'} = 4.5$ Hz, $J_{2',3'} = 5.3$ Hz, H-2′), 7.34–7.49, 7.87–8.03 (2 m, 6H, Ar-H). ^{13}C NMR ($CDCl_3$): $\delta_c = 20.7$, 20.8 (2 × CH_3CO), 29.8 (C-3′),

40.2 (C-2′), 71.1 (C-4′), 73.5 (C-1′), 118.9–146.2 (C-aromatic), 169.2, 169.3 (2 × CO). Anal Calcd. for $C_{16}H_{17}N_3O_4Br_2$ (475.13): C, 40.45; H, 3.61; N, 8.84; Br, 33.63. Found C, 40.20; H, 3.64; N, 9.11; Br, 33.53.

2-Phenyl-1,2,3-triazole-4-carboxaldehyde[13] (7)

A solution of **5** (0.2 g, 0.75 mmol) in ethanol (4 mL) was treated with sodium meta-periodate (1.7 mmol) in water (3 mL). The mixture was irradiated for 2 min, and the product was then recrystallized from ethanol, R_f 0.74 (H/E 5/1).

4-Cyano-2-phenyl-1,2,3-triazole (9)

A mixture of **7** (0.13 g, 0.75 mmol), hydroxylamine hydrochloride (0.06 g, 0.86 mmol), and sodium acetate (0.06 g, 0.90 mmol) in ethanol (4 mL) was irradiated for 2 min. Water (10 mL) was added and the product, 2-phenyl-1,2,3-triazole-4-carboxaldehyde oxime (**8**), was crystallized from ethanol, R_f 0.35 (H/E 5/1). ^{1}H NMR ($CDCl_3$) $\delta = 7.35–7.52$, 8.06–8.11 (2 m, 7H, CH, H-Ar), 9.55 (brs, 1H, OH, D_2O exchangeable). A suspension of which (0.1 g) with bentonite (0.1 g) in acetic anhydride (3 mL) was irradiated for 3 min, then poured onto ice water, and the product was washed with water and crystallized from ethanol, R_f 0.71 (H/E, 5:1). ^{1}H NMR (500 MHz, $CDCl_3$) $\delta = 7.24–7.40$, 8.01–8.13 (2 m, 7H, CH, H-Ar). Anal Calcd. for $C_9H_6N_4$ (170.17): C 63.52; H, 3.55; N, 32.92. Found C, 63.89; H, 3.30; N, 32.63.

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2-Phenyl-1,2,3-triazole-5-carboxaldehyde Thiosemicarbazone (10)

To a solution of compound **7** (0.1 g, 0.58 mmol) in ethanol (3 mL) was added thiosemicarbazide (0.08 g, 1.11 mmol) and one drop of acetic acid. The mixture was irradiated for 3 min and the product was recrystallized from ethanol, R_f 0.4 (H/E 2/1). ^{1}H NMR (500 MHz, $CDCl_3$) $\delta = 1.56$ (s, 2H, NH_2), 7.38–7.52, 8.00–8.11 (2 m, 7H, CH, H-Ar), 9.23 (brs, 1H, NH). Anal Calcd. for $C_{10}H_{10}N_6S$ (246.29): C 48.77; H, 4.09; N, 34.12; S, 13.02. Found C, 48.49; H, 4.30; N, 34.43; S, 12.83.

Mesoxaldehyde 1,2-bis(phenylhydrazone)[14] (11)

A suspension of D-arabino-hexosulose phenylosazone (0.2 g, 0.56 mmol) in ethanol (4 mL) was treated with sodium metaperiodate (1.7 mmol) in water (3 mL) and irradiated for 2 min. The product was crystallized from ethanol, R_f 0.63 (H/E, 3:1).

1-Phenyl-4-phenylazo-pyrazole[14] (12)

A solution of compound **11** (0.08 g, 0.30 mmol) in ethanol (2 mL) and one drop of hydrochloric acid was subjected to MW irradiation for 2 min. The product that separated out was recrystallized from ethanol as yellow plates, R_f 0.80 (H/E, 4:1).

Tetra-*O*-acetyl-D-*arabino*-hexosulose phenylosazone[15,16] (13)

D-*arabino*-Hexesulose phenylosazone (0.2 g, 0.56 mmol) was treated with pyridine (1.5 mL) and acetic anhydride (1.5 mL). The mixture was irradiated by MW for 3 min. Crushed ice was added, and the product was washed with water and crystallized from dilute ethanol to give yellow crystals of **13**, R_f 0.5 (H/E, 2:1). Compound **13** (0.15 g, 0.3 mmol) was dissolved in acetone (3 mL). A 1.5% sodium hydroxide solution (4 mL) was added then irradiated for 2 min. The product was filtered and crystallized from ethanol to give the Percival dianhydro-osazone.

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5-((1′*S*)-1′,2′-Diacetoxyethyl)-1-phenylpyrazole 3-carboxaldehyde (N-acetyl-N-phenyl)hydrazone[17] (14)

A mixture of **1** (0.30 g, 0.84 mmol) in acetic anhydride (4 mL) was irradiated for 5 min. Then ice and saturated sodium bicarbonate solution were added and the product was extracted with methylene chloride (2 × 20 mL). The extract was washed with saturated sodium bicarbonate solution and water. Drying ($Na_2 SO_4$) and removal of the solvent gave a syrup, which crystallized from ethanol to give **14** as colorless needles, R_f 0.42 (H/E 1/1).

5-((1′*S*)-1′,2′-Diacetoxyethyl)- 3-cyano-1-phenylpyrazole[11] (15)

A mixture of pyrazole **14** (0.2 g, 0.44 mmol) and bentonite (0.2 g) in dry DMSO (2 mL) was treated with hydroxylamine hydrochloride (0.04 g, 0.58 mmol) and then irradiated for 4 min. The mixture was poured onto ice water saturated with sodium bicarbonate, washed with water, and dried with sodium sulphate. The residue after filtration and concentration to remove solvent, which was purified by column chromatography using hexane/ethyl acetate (hexane:EtOAc 2/1) as eluent, gave **15** as a syrup, R_f 0.5 (H/E 1/1).

5-((1′*S*)-1′,2′-Diacetoxyethyl)-1-phenylpyrazole-3-carboxaldehyde[11] (16)

To a solution of **14** (0.1 g, 0.22 mmol) in ethanol (1 mL) was added 35–38% aqueous solution of formaldehyde (1.5 mL) and acetic acid (0.05 mL), which was then irradiated for 6 min. The mixture was made slightly basic by addition of ice-cold saturated sodium bicarbonate solution. The product was extracted with ether and the organic layer was washed with water, dried with sodium sulphate, and evaporated to give a residue, which was purified by column chromatography using n-hexane/ethyl acetate (4/1) as eluent to give **16** as a syrup.

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