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An efficient solventless synthesis of α-aryl-N-[1-methyl-2-(2/4-chlorophenyl)]ethyl nitrones and their antimicrobial activity

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An efficient solventless synthesis of α-aryl-N-[1-methyl-2-(2/4-chlorophenyl)]ethyl nitrones has been achieved in excellent yield. The compounds are characterized by NMR and X-ray studies. The antimicrobial activities of the synthesized compounds have also been investigated.

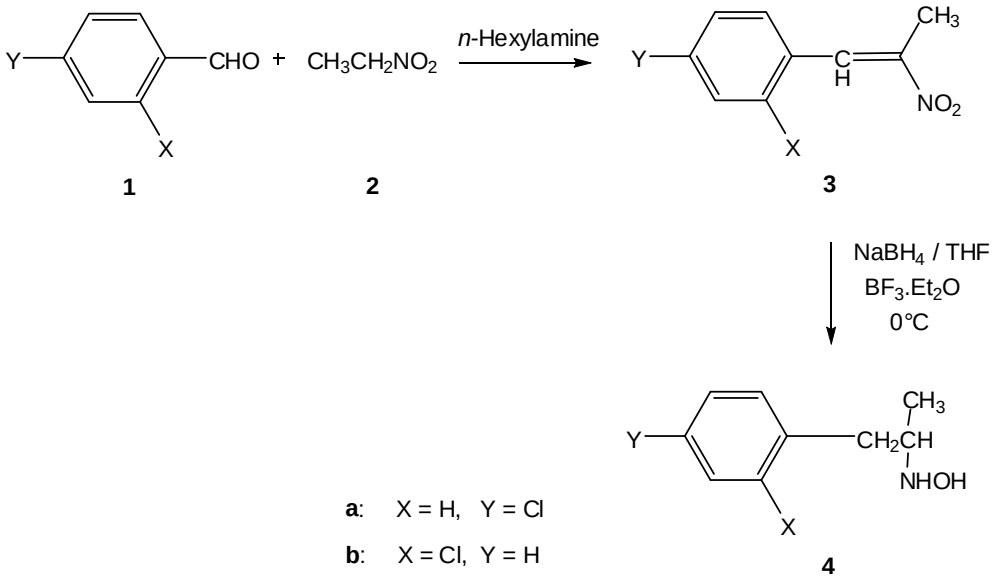
Keywords: Solventless organic reactions, solid state synthesis, green chemical protocol, α-aryl-N-[1-methyl-2-(2/4-chlorophenyl)]ethyl nitrones

The advantages of using solventless reaction[1-3] in developing more benign synthetic protocols include (i) the possibility of formation of compounds of high purity, (ii) the possibility of domino reaction, (iii) fast kinetics, (iv) lower energy usage, (v) use of environmentally benign reagents and procedures to reduce pollution, (vi) simplicity and low equipment cost, (vii) the possibility of avoiding functional group protection-deprotection, (viii) access to new compounds for which the traditional approach fails and (ix) enhanced selectivities without dilution. It has been planned to explore the possibility of preparing heterodienes as potential precursors for Diels-Alder reactions *via* nitrone intermediates. It is proposed to synthesize α-aryl-N-[1-methyl-2-(2/4-chlorophenyl)]-ethyl nitrones by the green chemical route without any solvent. It is so chosen that the reacting materials are solids. The results of the investigation are presented in this article. This class of compounds assumes importance as the antioxidant activity of the β-hydroxy-nitrones has been examined by determination of the index of lipid peroxidation[4]. A set of nitrones constitutes a novel breed of free radical scavenger with increased antioxidant activity over the standard α-phenyl nitrone derivatives[5]. Pyridyl substituted nitrones have been proposed for the treatment of nerve degeneration diseases. It is to be mentioned that a conventional synthesis related to the system studied here with no methyl substituent in the N-alkyl chain and its rearrangement to amide has been studied[6].

The substituted β-methyl-β-nitrostyrenes **3**, the precursors for the present synthesis, were prepared by the condensation of appropriate aldehydes **1** with nitroethane **2** by reported methods[7]. The diboranes, generated *in situ* by the addition of BF_3-etherate to sodium borohydride, effected the chemoselective reduction of nitrostyrenes to the corresponding saturated hydroxylamines **4** (Ref. 8). The condition of the reaction is very important, as any variation in the conditions and concentration of the reagent may lead to complete reduction to amines (**Scheme I**).

The hydroxylamines prepared **4** are solids and their structures have been confirmed by NMR spectra. The methylene hydrogens appear at δ 2.58 and 2.86 as neat doublet of doublets with coupling constants of 13.2 and 6.9 Hz in **4a**. The methyl signal appears as doublet at δ 1.07 (J = 6.6 Hz). The methine hydrogen appears at δ 3.17 as a sextet. The methyl carbon appears at δ 17.3, the methylene carbon at δ 39.1 and the methine carbon at δ 58.3.

The addition of the hydroxylamines **4a** or **4b** to different solid arylaldehydes **5** has been carried out under solventless condition. An equimolar mixture of



Scheme I

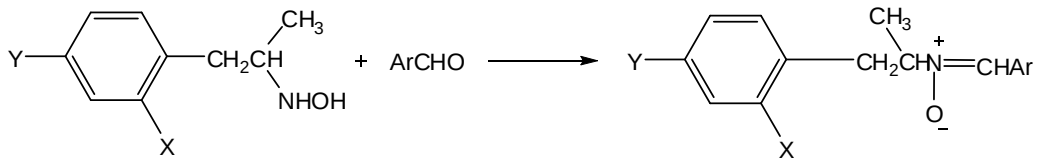
4a/4b with **5** was ground in a mortar and left at RT. The mixture on grinding becomes viscous in some cases and remains as solid in others. The progress of the reaction was monitored by TLC from time to time and the product was recrystallised after completion of the reaction from ethanol. It is noticed that the reaction gets completed in an hour or two (**Scheme II**). The yield in all the cases is quite good, more than 80%, giving only one product, α-aryl-N-[1-methyl-2-(2/4-chlorophenyl)]ethyl nitrone. The physical, analytical and spectral characterization data for all the synthesized compounds, **5a-q**, are presented in **Table I**.

To study the effect of temperature on the course of the reaction, the ground mixture was kept immediately at 70°C and the progress of the reaction was monitored by TLC. Though there is no appreciable increase in the yield, there is tremendous decrease in reaction time, most of the reactions getting completed in less than an hour. It should be noted that heating the reaction mixture has not led to any decomposition. The results of this solvent-free synthesis of nitrones at elevated temperature are summarized in **Table II**, indicating the yields and time taken for the completion of the reaction.

The observed reaction time in the above condensation reaction under thermal condition is nevertheless relatively high. It is well known that microwave irradiation greatly reduces the reaction time of organic reactions, especially for condensation reactions. Hence it was decided to effect the present reaction under microwave and thus a well ground mixture of aldehyde and hydroxylamine was irradiated under 480 W (2450 MHz) radiation. As expected, the reaction time was greatly reduced taking just minutes with almost quantitative conversion to nitrones. The results are summarized in **Table II**.

The ^{1}H and ^{13}C NMR spectral features of compounds **5a-q** are given in **Table I**. The $–CH_2–CH–$ pattern of nitrone is substantially different from the starting material, hydroxylamine. For **5c**, C-(4-dimethylaminophenyl)-N-1-methyl-2-(4-chlorophenyl)-ethyl nitrone, the methylene hydrogens CH_a and CH_b appear at δ 2.79 and 3.37 as doublets of doublets, the former with coupling constants 13.5 and 4.5 Hz and the latter with 13.5 and 9.0 Hz. The difference in the chemical shift position of diastereotopic hydrogen is just 0.28 ppm in the hydroxylamine, but it is 0.42 ppm in the nitrone. The coupling pattern is also different for the nitrone and the hydroxylamine. The methine hydrogen has a coupling of 4.5 Hz and 9.0 Hz with the vicinal hydrogens in the nitrones, whereas both the methylenic hydrogens have the same coupling constant with the methine hydrogen in the hydroxylamine. The methine hydrogen has also experienced a downfield shift of 0.8 ppm compared to the hydroxylamine and this appears as a multiplet at δ 4.0. almost 1 ppm deshilded compared to hydroxylamine. (delete the colored portion) The methyl doublet (J = 6.6 Hz) appears at δ 1.53. The coupling connectivities are well established by H–H COSY spectrum.

The methine carbon in the ^{13}C NMR spectrum of **5c** appears at δ 72.96 and the methylene carbon at δ 40.06. The methyl carbon appears at δ 19.5. The



4 **5**

a: X = H; Y = Cl; Ar = 4-Chlorophenyl
b: X = H; Y = Cl; Ar = 4-Nitrophenyl
c: X = H; Y = Cl; Ar = 4-N,N-Dimethylaminophenyl
d: X = H; Y = Cl; Ar = 2-Phenylthiophenyl
e: X = H; Y = Cl; Ar = 2-Hydroxy-1-naphthyl
f: X = H; Y = Cl; Ar = 4-Phenylthiophenyl
g: X = H; Y = Cl; Ar = 3,4-Methylenedioxyphenyl
h: X = Cl; Y = H; Ar = 4-Chlorophenyl
i: X = Cl; Y = H; Ar = 4-Nitrophenyl
j: X = Cl; Y = H; Ar = 2-Phenylthiophenyl
k: X = Cl; Y = H; Ar = 2-Hydroxy-1-naphthyl
l: X = Cl; Y = H; Ar = 3,4-Methylenedioxyphenyl
m: X = Cl; Y = H; Ar = 2-Methoxyphenyl
n: X = Cl; Y = H; Ar = 4-N,N-Dimethylaminophenyl
o: X = Cl; Y = H; Ar = 4-Hydroxyphenyl
p: X = Cl; Y = H; Ar = 4-Phenylthiophenyl
q: X = Cl; Y = H; Ar = 3,4-Dimethoxyphenyl

Scheme II

Table I — Melting point, analytical and spectral characterization data of nitrones **5a-q**

Compd	m.p. (°C)	Observed % (Calcd) C	H	N	^{1}H NMR ($CDCl_3$, δ, ppm)	^{13}C NMR ($CDCl_3$, δ, ppm)
5a	158-60	62.20 (62.36	4.67 4.87	4.70 4.55)	1.50 (d, *J* = 6.6 Hz, 3H), 3.20 (dd, *J* = 13.5, 9.0 Hz, 1H), 2.70 (dd, *J* = 13.5, 4.5 Hz, 1H), 3.95 (m, 1H), 7.00 (s, 1H), 7.15 (d, 2H), 7.20 (AB quartet, 4H), 7.90 (d, 2H)	19.1, 40.2, 74.4, 127.2, 129.0, 129.1, 129.3, 130.1, 131.4, 132.5, 134.5, 136.9
5b	100-02	60.40 (60.29	4.60 4.71	8.90 8.79)	1.50 (d, *J* = 6.6 Hz, 3H), 3.20 (dd, *J* = 13.5, 9.0 Hz, 1H), 2.75 (dd, *J* = 13.5, 4.5 Hz, 1H), 4.10 (m, 1H), 7.00 (s, 1H), 7.22 (AB quartet, 4H), 8.01 (AA′, BB′ pattern, 4H)	19.2, 40.1, 74.2, 124.0, 129.0, 129.2, 129.9, 131.0, 132.4, 135.0, 136. 9, 148.2
5c	121	68.00 (68.26	6.50 6.64	8.90 8.85)	1.53 (d, *J* = 6.6 Hz, 3H), 3.37 (dd, *J* = 13.5, 9.0 Hz, 1H), 2.79 (dd, *J* = 13.5, 4.5 Hz, 1H), 4.00 (m, 1H), 6.45 (d, 2H), 7.00 (s, 1H), 7.24 (AB quartet, 4H), 7.90 (d, 2H)	19.3, 40.1, 74.7, 113.1, 118.2, 129.1, 129.5, 129.7, 131.5, 132.5, 136.5, 144.2
5d	Liquid	69.00 (69.21	5.50 5.24	4.30 4.19)	1.35 (d, *J* = 6.6 Hz, 3H), 3.15 (dd, *J* = 13.5, 9.0 Hz, 1H), 2.70 (dd, *J* = 13.5, 4.5 Hz, 1H), 3.03, (s, 6H), 3.90 (m, 1H), 6.91-7.32 (m, 8H), 7.00 (s, 1H), 7.20 (AB quartet, 4H), 9.12 (d, 1H)	19.3, 39.2, 40.3, 74.1, 127.1, 127.2, 129.1, 129.2, 129.6, 129.8, 129.9, 130.1, 131.1, 131.5, 132.0, 133.1, 132.1, 136.8
5e	182	70.50 (70.70	5.00 5.30	4.40 4.12)	1.50 (d, *J* = 6.6 Hz, 3H), 3.15 (dd, *J* = 13.5, 9.0 Hz, 1H), 2.80 (dd, *J* = 13.5, 4.5 Hz, 1H), 4.30 (m, 1H), 7.00 (s, 1H), 7.21 (AB quartet, 4H), 6.81-7.60 (m, 6H), 10.5 (s, 1H)	19.1, 40.2, 74.2, 109.1, 118.2, 124.0, 126.2, 128.3, 129.4, 129.7, 130.4, 130.8, 131.0, 131.5, 133.0, 134.4, 136.3, 153.5
5f	115	69.00 (69.21	5.50 5.24	4.30 4.19)	1.45 (d, *J* = 6.6 Hz, 3H), 3.30 (dd, *J* = 13.5, 9.0 Hz, 1H), 2.75 (dd, *J* = 13.5, 4.5 Hz, 1H), 4.00 (m, 1H), 7.00 (s, 1H), 7.22 (AB quartet, 4H), 7.20 (m, 7H), 7.90 (d, 2H)	19.1, 40.1, 74.3, 127.3, 127.6, 129.2, 129.4, 129.6, 129.8, 131.2, 131.4, 131.5, 131.6,* 132.6, 133.7
5g	Liquid	64.40 (64.26	5.10 5.04	4.30 4.41)	1.45 (d, *J* = 6.6 Hz, 3H), 3.25 (dd, *J* = 13.5, 9.0 Hz, 1H), 2.70 (dd, *J* = 13.5, 4.5 Hz, 1H), 3.95 (m, 1H), 5.80 (s, 2H), 6.70 (d, 1H), 7.00 (s, 1H), 7.23 (AB quartet, 4H), 7.25 (dd, 1H), 7.90 (d, 1H)	19.2, 40.1, 74.2, 91.3, 115.2, 115.7, 122.3, 122.7, 129.4, 129.9, 131.5, 132.2, 133.8, 136.9

— *Contd*

Table I — Melting point, analytical and spectral characterization data of nitrones **5a-q** — *Contd*

Compd	m.p.	Observed % (Calcd)			^{1}H NMR	^{13}C NMR
	(°C)	C	H	N	($CDCl_3$, δ, ppm)	($CDCl_3$, δ, ppm)
5h	105	62.10 (62.36	4.90 4.87	4.40 4.55)	1.58 (d, *J* = 6.6 Hz, 3H), 3.13 (dd, *J* = 13.5, 4.8 Hz, 1H), 3.34 (dd, *J* = 13.5, 9.0 Hz, 1H), 4.30 (m, 1H), 7.00 (s, 1H), 7.10-7.30 (m, 4H), 7.31 (d, *J* = 8.4 Hz, 2H), 8.08 (d, *J* = 8.4 Hz, 2H)	18.7, 38.4, 71.7, 126.9, 128.5, 128.6, 128.7, 129.4, 129.7, 131.7, 132.2, 133.6, 135.2, 135.5
5i	145	60.50 (60.29	4.50 4.71	8.60 8.79)	1.60 (d, *J* = 6.6 Hz, 3H), 3.16 (dd, *J* = 13.5, 5.1 Hz, 1H), 3.33 (dd, *J* = 13.5, 9.3 Hz, 1H), 4.43 (m, 1H), 7.10-7.38 (m, 4H), 7.22 (s, 1H), 8.20 (d, *J* = 9 Hz, 2H), 8.28 (d, *J* = 9 Hz, 2H)	19.2, 39.0, 72.9, 124.1, 124.7, 127.4, 129.1, 129.9, 130.9, 131.8, 132.0, 134.0, 136.3, 147.9
5j	Liquid	69.00 (69.21	5.50 5.24	4.40 4.19)	1.40 (d, *J* = 6.6 Hz, 3H), 3.03 (dd, *J* = 13.5, 5.4 Hz, 1H), 3.26 (dd, *J* = 13.5, 9.0 Hz, 1H), 4.24 (m, 1H), 7.00-7.35 (m, 12H), 7.79 (s, 1H), 9.30 (d, *J* = 7.8 Hz, 1H)	18.1, 38.0, 71.6, 125.7, 125.9, 127.9, 128.2, 128.3, 128.5, 128.7, 128.9, 129.9, 130.2, 131.1, 131.4, 132.3, 133.2, 134.3, 134.6, 136.0
5k	120	70.50 (70.70	5.50 5.30	3.90 4.12)	1.68 (d, *J* = 6.3 Hz, 3H), 3.20 (dd, *J* = 13.5, 3.3 Hz, 1H), 3.35 (dd, *J* = 13.5, 10.5 Hz, 1H), 4.60 (m, 1H), 7.00-7.50 (m, 8H), 7.70 (d, *J* = 7.2 Hz, 1H), 7.80 (s, 1H), 7.80 (d, *J* = 9.0 Hz, 1H), 12.10 (s, 1H)	18.9, 38.4, 70.2, 107.8, 120.8, 122.0, 123.5, 127.3, 127.3, 127.9, 128.7, 128.7, 129.4, 131.8, 132.4, 133.4, 134.3, 134.7, 138.4, 160.9
5l	65	64.00 (64.20	5.30 5.04	4.65 4.41)	1.65 (d, *J* = 6.6 Hz, 3H), 3.12 (dd, *J* =13.5, 5.1 Hz, 1H), 3.35 (dd, *J* = 13.5, 8.7 Hz, 1H), 4.24 (m, 1H), 5.94 (s, 2H), 6.80 (d, *J* = 8.1 Hz, 1H), 6.98 (s, 1H), 7.00-7.40 (m, 5H), 8.06 (d, *J* = 1.5 Hz, 1H)	18.6, 38.2, 71.1, 101.3, 108.1, 108.3, 124.1, 124.7, 126.8, 128.3, 129.2, 131.6, 132.9, 133.6, 135.3, 147.3, 148.6
5m	Liquid	67.50 (67.22	6.10 5.93	4.30 4.61	1.55 (d, *J* = 6.6 Hz, 3H), 3.13 (dd, *J* = 13.5, 5.1 Hz, 1H), 3.39 (dd, *J* = 13.5, 8.7 Hz, 1H), 3.74 (s, 3H), 4.33 (m, 1H), 6.80 (d, *J* = 8.1 Hz, 1H), 7.00-7.50 (m, 6H), 7.58 (s, 1H), 9.20 (dd, *J* = 7.8, 1.5 Hz, 1H)	18.8, 38.2, 71.7, 55.4, 109.6, 119.3, 120.5, 126.8, 127.8, 128.1, 128.6, 129.2, 131.1, 131.6, 133.7, 135.4, 156.7
5n	105	68.00 (68.26	6.90 6.64	8.60 8.85	1.55 (d, *J* = 6.3 Hz, 3H), 3.08, (s, 6H), 3.12 (dd, *J* = 13.5, 4.8 Hz, 1H), 3.38 (dd, *J* = 13.5, 8.7 Hz, 1H), 4.20 (m, 1H), 6.65 (d, *J* = 8.7 Hz, 2H), 6.93 (s, 1H), 7.05 (t, *J* = 7.2 Hz, 1H), 7.12 (t, *J* = 7.2 Hz, 1H), 7.24 d, *J* = 7.2 Hz, 1H), 7.34 (d, *J* = 7.2 Hz, 1H), 8.01 (d, *J* = 8.7 Hz, 2H)	18.6, 38.1, 70.3, 39.9, 111.0, 118.3, 126.3, 128.1, 129.2, 129.7, 130.4, 131.8, 133.6, 133.7, 135.7
5o	160	66.00 (66.33	5.80 5.52	4.50 4.83)	1.52 (d, *J* = 6.3 Hz, 3H), 3.11 (dd, *J* = 13.5, 5.1 Hz, 1H), 3.32 (dd, *J* = 13.5, 8.7 Hz, 1H), 3.35 (b, 1H), 4.26 (m, 1H), 6.80 (d, *J* = 8.7 Hz, 2H), 7.09 (s, 1H), 7.15-7.40 (m, 4H), 7.90 (d, *J* = 8.7 Hz, 2H)	17.8, 37.2, 69.7, 114.6, 120.9, 126.0, 127.5, 128.4, 130.0, 130.8, 132.7, 132.9, 134.5, 158.6
5p	85	69.50 (69.21	5.50 5.24	3.99 4.19)	1.55 (d, *J* = 6.6 Hz, 3H), 3.12 (dd, *J* = 13.5, 5.1 Hz, 1H), 3.34 (dd, *J* = 13.5, 8.7 Hz, 1H), 4.28 (m, 1H), 7.02 (s, 1H), 7.00-7.40 (m, 11H), 8.02 (d, *J* = 8.7 Hz, 2H)	18.7, 38.3, 71.4, 126.9, 127.9, 128.2, 128.3, 128.7, 128.9, 129.3, 131.6, 132.0, 132.5, 132.7, 133.5, 133.6, 135.2, 139.5
5q	128	64.99 (64.77	5.60 5.99	4.50 4.19)	1.55 (d, *J* = 6.0 Hz, 3H), 3.14 (dd, *J* = 13.5, 4.8 Hz, 1H), 3.36 (dd, *J* = 13.5, 9.0 Hz, 1H), 3.84 (s, 3H), 3.90 (s, 3H), 4.26 (m, 1H), 6.80 (d, *J* = 8.4 Hz, 1H), 7.00 (s, 1H), 7.20-7.40 (m, 5H), 8.36 (s, 1H)	18.2, 37.8, 70.6, 110.0, 110.4, 122.5, 123.1, 126.4, 127.9, 128.8, 131.2, 132.9, 133.2, 134.9, 147.7, 149.9, 55.3, 55.6

methine carbon has suffered a deshielding of nearly 17 ppm compared to the **4a**. The azomethine carbon appears at δ 136.2 as evidenced by the C–H COSY spectrum. This carbon is relatively shielded compared to azomethine carbons of diaryl nitrones. The HMBC spectrum is useful in confirming the quarternary carbons of the aryl ring. Thus, unambiguous assignment of all the carbons and hydrogens has been achieved by a combined 2D analysis.

The single crystal X-ray analysis[9] has been carried out on the synthesized nitrone, **5n**. The ORTEP diagram and the packing diagram of **5n** are given in **Figure 1** and **Figure 2** respectively. The crystal data is presented in **Table III**.

Table II — Reaction time and yield under different conditions for the formation of **5**

Compd	At RT		At 70°C		Under microwave irradiation	
	Time taken (Hr)	Yield (%)	Time taken (min)	Yield (%)	Time taken (min)	Yield (%)
5a	1.25	84	20	94	4	~ 100
5b	2.5	88	35	91	6	~ 100
5c	8.0	82	45	88	10	~ 100
5d	10.0	81	60	84	10	~ 100
5e	9.0	80	50	85	8	~ 100
5f	2.5	84	20	89	6	~ 100
5g	8.0	81	50	88	10	~ 100
5h	1.0	85	20	90	2	~ 100
5i	2.0	80	30	95	4	~ 100
5j	4.0	83	60	85	8	~ 100
5k	8.0	85	60	90	10	~ 100
5l	2.0	80	30	95	4	~ 100
5m	2.5	85	40	90	4	~ 100
5n	2.5	85	40	85	4	~ 100
5o	1.0	80	30	90	4	~ 100
5p	1.0	85	30	90	4	~ 100
5q	1.5	80	50	85	4	~ 100

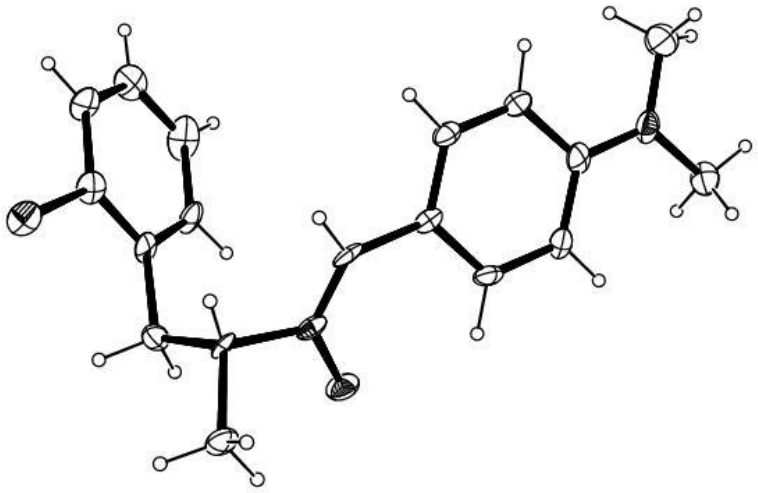


Figure 1 — ORTEP diagram of [2-(2-chlorophenyl)-1-methylethyl] [(Z)-(4-N,N-dimethylaminophenyl)methylidene] ammoniumolate, **5n**

Nitrones are found to exhibit antibacterial activity[10-12]. Hence, the antibacterial susceptibility testing on the synthesised nitrones **5** has been carried out at two different concentrations *viz*., 25 μg and 50 μg against *E Coli, Pseudomonas, Salmonella* and *S aureus*. Most of the nitrones **5** exhibited remarkable activity. Much change has not been observed at higher concentrations of 50 μg in the inhibition zone diameter. This indicates that 25 μg of the test sample is sufficient to inhibit the growth of the bacteria. The activity has been studied with ampicillin as the control sample. **5n** is active against all the four microorganisms tested. **5o** is active against *E Coli* and *S aureus* alone, while **5l** is active against *Pseudomonas* and *Salmonella* alone.

Experimental Section

Melting points are uncorrected. NMR spectra were recorded on a Bruker 300 MHz (UltraShield) NMR

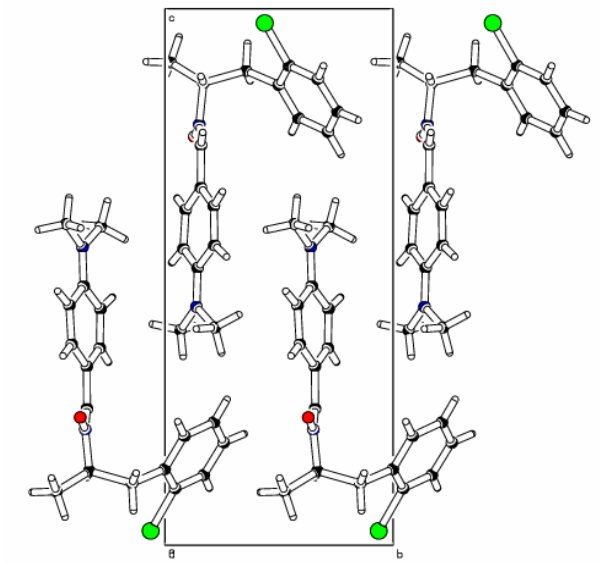


Figure 2 — Packing diagram of [2-(2-chlorophenyl)-1-methylethyl][(Z)-(4-N,N-dimethylamino-phenyl)methylidene] ammoniumolate, **5n**

spectrometer at RT using $CDCl_3$ as solvent with TMS as internal standard. The single crystal X-ray data were collected on a Nonius MACH3 kappa diffractometer with MoK_α radiation (λ = 0.71069 Å). The structure was solved by direct methods from SHELXS-97 and refined by full matrix least squares on F^2 by SHELXL-97. Crystallographic data (excluding structure factors) for the structure in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication number CCDC 648746 for **5n**. Copies of the data can be obtained, free of charge, by contacting The Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK (e-mail: data_request@ccdc.cam.ac.uk; fax: +44 1223 336033)

General procedure for the preparation of α-aryl-N-[1-methyl-2-(2/4-chlorophenyl)]ethyl nitrones, 5

N-Hydroxy-1-methyl-2-(2/4-chlorophenyl)ethanamine, **4** (0.001 mole), prepared by a method similar to that reported in literature,[7,8] and a solid arylaldehyde (0.001 mole) were mixed and ground well in a mortar and left at RT. The progress of the reaction was monitored by TLC. The nitrone obtained was purified by recrystallization from ethanol. The same experiment was performed in an air oven at 70°C and also under microwave dielectric heating.

Antibacterial studies on the synthesised nitrones 5

In the present investigation disk diffusion method has been employed to determine the antibacterial activity. The organism is inoculated evenly into an agar plate to obtain a confluent lawn of growth. Disks impregnated with antimicrobial agents are then placed into the agar. After a suitable incubation period, the plate is examined for zones of inhibited growth around the disk. Depending on the zone's size, the organism is reported to be susceptible, intermediate or resistant to the antimicrobial agent.

Table III — Crystal data and structural refinement for **5n**

Empirical formula	$C_{18}H_{21}ClN_2O$
Formula weight	316.82
Temperature	293 (2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	P 21
Unit cell dimensions	a = 6 (4) Å; α = 90°
	b = 8 (8) Å; β = 93 (9)°
	c = 19 (11) Å; γ = 90°
Volume	831 (1153) $Å^3$
Z	2
Density (calculated)	1.266 mg m^{-3}
Absorption coefficient	0.233 mm^{-1}
F(000)	336
Crystal size	0.25 × 0.16 × 0.17 mm
Theta range for data collection	2.16 to 24.98°
Index ranges	0<=h<=6, -1<=k<=9, -22<=l<=22
Reflections collected	1998
Independent reflections	1812 [R_{int} = 0.0797]
Absorption correction	Psi-scans
Max. and min. transmission	0.9639 and 0.4809
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	1812 / 1 / 202
Goodness-of-fit on F^2	1.346
Final R indices [I>2σ(I)]	R1 = 0.1347, wR2 = 0.3262
R indices (all data)	R1 = 0.2097, wR2 = 0.3764
Largest diff. peak and hole	1.331 and -0.885 e. $Å^{-3}$

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