SHORT PAPER

J. Chem. Research (S), 2003, 204-207

1,3-Dipolar cycloadditions of arylcarbonitrile oxides and diaryl nitrilimines with some 2-arylmethylene-1,3-indanediones; regiochemistry of the reactions†

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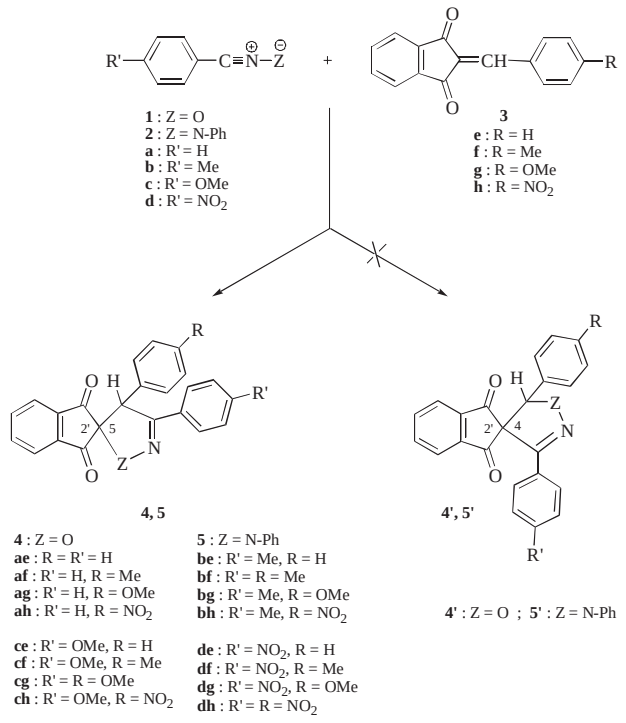
Ring-closure reaction affording spiroisoxazolines and spiropyrazolines *via* a 1,3-dipolar cycloaddition between the title compounds, occurs with high regioselectivity.

Keywords: 1,3-dipolar cycloadditions, arylcarbonitrile oxides, diaryl nitrilimines, 2-arylmethylene-1,3-indanediones

As part of our research[1] into the use of 1,3-dipolar cycloaddition reactions to synthesize five-membered heterocycles, we present here the reactions of aryl carbonitrile oxides **1** and diaryl nitrilimines **2** (generated *in situ* from the corresponding hydroximoyl chlorides and *C*-aryl-*N*-phenylhydrazidoyl chlorides respectively) with some 2-arylmethylene-1,3-indanediones **3** (Scheme 1 and Table 1). Since the dipolarophilic site is exocyclic, we obtained the spirocyclic adducts **4** and **5** (Scheme 1 and Tables 2, 3).



Scheme 1 Formation of spiro-indanediones 4,5.

*Cycloadditions of arylcarbonitrile oxides **1**:* These 1,3-dipoles yielded regioselectively (100%) to the spiro-isoxazolines **4** whatever the nature of the substituents R and R' (Table 1). Yields were moderate (31%) to good (79%).

The ^{1}H NMR data (δ 4-H = 5.22–5.40 ppm) enabled us to determine the structure of regioisomer **4**, spiro[3,4-diaryl-2-isoxazoline-5:2'-1',3'-indanedione], since, in the case of the reverse regioisomer (**4'**), we should observe a chemical shift value higher than 6 ppm for the 5-H proton[2] (Table 1). The ^{13}C NMR data confirmed this result; the chemical shifts of the spiro-carbon atoms (C-5:2') were found between 87.60 and 89.50 ppm, because of the deshielding effect of the oxygen atom.

*Cycloadditions of Diaryl nitrilimines **2**:* These 1,3-dipoles led regioselectively (100%) to spiro-pyrazolines **5** irrespective of the substituents R and R' (Table 2). Yields were good (50%) to very good (90%).

The ^{1}H NMR data did not allow us to distinguish between the two regioisomeric structures **5** or **5'** since chemical shifts of H-5 (in **5**) or H-4 (**5'**) varied between 5.07 and 5.24 ppm. Nor did comparison with the chemical shift values of analogous protons of compounds **6** and **6'** (Fig. 1), obtained by 1,3-dipolar cycloaddition reaction of diphenyl nitrilimine with benzylideneacetone[3], help us to ascertain the regiochemistry.

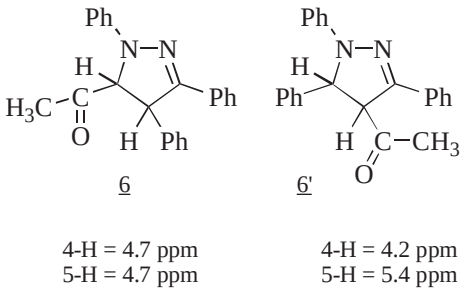


Fig. 1 Chemical shifts of 4-H and 5-H protons of **6** and **6'**

On the other hand, the ^{13}C NMR data were unambiguous: the chemical shift values of spiro-carbon atoms (79.60–80.60 ppm) were in good agreement with structure **5**. In the case of the alternative structure (**5'**), these values should be below 60 ppm.[4]

Elemental analyses and the remaining spectral data (IR, ^{1}H, ^{13}C NMR) were in good agreement with the proposed structures.

In summary: when aryl nitrile oxides or diaryl nitrile imines reacted with activated dipolarophilic alkenes, the predominant or unique resulting cycloadducts bore the substituent of the initial alkene at the 5-position of the isoxazoline or pyrazoline ring. These results are in good agreement with previous work.[1,2]

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† This is a Short Paper, there is therefore no corresponding material in *J Chem. Research (M).*

Table 1 Characteristics of the 2-arylmethylene-1,3-indanediones **3e–f**

Compound	R	M.p. /°C	Yield /%	IR/cm^{-1} $\nu_{C=O}$	$\nu_{C=C\ arom}$	$\nu_{C=C\ olefinic}$	^{1}H NMR/δ ppm
3e	H	153	86	1725,1680	1585	1610	7.87 (s, H vinyl); 7.50–8.63 (m. 9 arom. H)
3f	Me	152	70	1720,1680	1590	1610	2.43 (s, 3H, CH_3); 7.88 (s, H vinyl); 7.22–8.53 (m, 8 arom. H)
3g	OMe	157	75	1720,1680	1585	1610	3.98 (s, 3H, OCH_3); 7.97 (s, H vinyl); 7.03–8.78 (m, 8 arom. H)
3h	NO_2	233	70	1735,1690	1590	1610	8.66 (s, H vinyl); 7.80–8.57 (m, 8 arom. H)

Table 2 Physical and spectroscopic data of the spiro[3,4-diaryl-2-isoxazoline-5:2'-1',3'-indanediones] **4**

Compound	R	R'	M.p. /°C	Yield /%	IR/cm^{-1} $\nu_{C=O}$	$\nu_{C=N}$	$\nu_{C=Carom}$	^{1}H NMR/^{13}C NMR/δ ppm
4ae	H	H	234	75	1753, 1718	1605	1594	5.31 (s, 1H, H^4); 6.72–8.30 (m, 14 arom. H) 55.25 (OMe); 63.30 (C^4); 88.95 ($C^{5:2'}$); 192.95 (C=O); 196.00 (C=O)
4be	H	Me	250	79	1737, 1705	1608	1585	2.24 (s, 3H, Me); 5.27 (s, 1H, H^4); 6.80–8.65 (m, 13 arom. H) 21.15 (Me); 62.70 (C^4); 88.05 ($C^{5:2'}$); 192.15 (C=O); 195.20 (C=O)
4ce	H	OMe	240	62	1739, 1706	1606	1585	3.76 (s, 3H, OMe); 5.26 (s, 1H, H^4); 6.76–8.10 (m, 13 arom. H) 55.25 (OMe); 63.30 (C^4); 88.95 ($C^{5:2'}$); 192.95 (C=O); 196.00 (C=O)
4de	H	NO_2	232	32	1759, 1724	1610	1574	5.39 (s, 1H, H^4); 6.98–8.12 (m, 13 arom. H) 62.20 (C^4); 89.40 ($C^{5:2'}$); 192.05 (C=O); 195.15 (C=O)
4af	Me	H	228	70	1755, 1720	1608	1591	2.24 (s, 3H, Me); 5.26 (s, 1H, H^4); 6.84–8.78 (m, 13 arom. H) 21.20 (Me); 62.20 (C^4); 88.95 ($C^{5:2'}$); 192.35 (C=O); 195.10 (C=O)
4bf	Me	Me	263	63	1755, 1726	1608	1597	2.21 (s, 3H, Me); 2.23 (s, 3H, Me); 5.23 (s, 1H, H^4); 6.82–8.20 (m, 12 arom. H) 21.15 (Me); 62.30 (C^4); 88.95 ($C^{5:2'}$); 192.30 (C=O); 195.20 (C=O)
4cf	Me	OMe	234	65	1754, 1716	1608	1596	2.24 (s, 3H, Me); 3.77 (s, 3H, OMe); 5.22 (s, 1H, H^4); 6.65–8.09 (m, 13 arom. H) 21.15 (Me); 55.25 (OMe); 63.05 (C^4); 88.95 ($C^{5:2'}$); 192.00 (C=O); 195.00 (C=O).
4df	Me	NO_2	228	45	1754, 1717	1600	1595	2.28 (s, 3H, Me); 5.29 (s, 1H, H^4); 6.89–8.20 (m, 12 arom. H) 21.15 (Me); 62.05 (C^4); 89.45 ($C^{5:2'}$); 192.15 (C=O); 195.30 (C=O)
4ag	OMe	H	240	80	1711, 1679	1590	1595	3.76 (s, 3H, OMe); 5.36 (s, 1H, H^4); 6.79–8.00 (m, 13 arom. H) 55.15 (OMe); 61.20 (C^4); 87.85 ($C^{5:2'}$); 192.25 (C=O); 195.00 (C=O)
4bg	OMe	Me	222	70	1735, 1723	1610	1595	2.24 (s, 3H, Me); 3.76 (s, 3H, OMe); 5.34 (s, 1H, H^4); 6.75–8.07 (m, 12 arom. H) 21.15 (Me); 55.10 (OMe); 61.05 (C^4); 89.05 ($C^{5:2'}$); 192.20 (C=O); 195.25 (C=O)
4cg	OMe	OMe	174	75	1752, 1723	1604	1599	3.71 (s, 3H, OMe); 3.76 (s, 3H, OMe); 5.22 (s, 1H, H^4); 6.65–8.09 (m, 12 arom. H) 55.10 (OMe); 55.25 (OMe); 62.85 (C^4); 89.05 ($C^{5:2'}$); 193.10 (C=O); 196.10 (C=O)
4dg	OMe	NO_2	231	55	1757, 1726	1610	1589	3.78 (s, 3H, OMe); 5.30 (s, 1H, H^4); 6.78–8.19 (m, 12 arom. H) 55.15 (OMe); 61.80 (C^4); 89.45 ($C^{5:2'}$); 192.25 (C=O); 195.35 (C=O)
4ah	NO_2	H	200	52	1752, 1714	1600	1585	5.38 (s, 1H, H^4); 7.22–8.15 (m, 13 arom. H) 61.20 (C^4); 87.85 ($C^{5:2'}$); 192.25 (C=O); 195.00 (C=O).
4bh	NO_2	Me	219	31	1754, 1719	1600	1591	2.25 (s, 3H, Me); 5.36 (s, 1H, H^4); 7.23–8.10 (m, 12 arom. H) 21.15 (Me); 60.70 (C^4); 87.70 ($C^{5:2'}$); 192.15 (C=O); 195.10 (C=O)
4ch	NO_2	OMe	225	64	1755, 1720	1608	1591	3.78 (s, 3H, OMe); 5.34 (s, 1H, H^4); 6.79–8.15 (m, 12 arom. H) 55.35 (OMe); 61.45 (C^4); 87.65 ($C^{5:2'}$); 192.20 (C=O); 195.20 (C=O)
4dh	NO_2	NO_2	180	43	1753, 1716	1604	1594	5.40 (s, 1H, H^4); 7.20–8.19 (m, 12 arom. H) 60.40 (C^4); 88.20 ($C^{5:2'}$); 192.15 (C=O); 195.10 (C=O)

Experimental

Melting points were determined on a Kofler bank. IR spectra were recorded from KBr dispersions (5°/$_{oo}$) with a Perkin-Elmer197 spectrometer; only structurally significant bands (ν) are reported. NMR spectra were recorded in $CDCl_3$ with a Bruker-Spectrospin AC 250 spectrometer operating at 250 MHz for ^{1}H and 62.9 MHz for ^{13}C (compounds **3** and products **5**). Elemental analysis were performed by the Centre de Microanalyses of Claude Bernard University (Lyon I, Vernaison); the results are listed in Table 3.

Aryl nitrile oxides and Diaryl nitrilimines were generated following the methods of previous literature reports.[1,5,6]

Preparation of 2-arylmethylene-1,3-indanediones (**3**): These compounds were obtained by an adaptation of a published procedure:[7] *General procedure*: A mixture of 1,3-indanedione (20 mmol) and the arylaldehyde (20 mmol) in toluene (50 ml) was refluxed during 24 hours with a catalytic amount of *p*-toluenesulfonic acid (Dean-Stark apparatus). The reaction mixture was poured into water (100ml) and extracted with ether (3×50ml). The organic phase was washed with water (3×50ml) and dried over anhydrous Na_2SO_4. The solvents were evaporated off and the residue dissolved and crystallised from an ethanol : toluene mixture (50 : 50, 50 ml) (Table 1).

Cycloaddition reactions of aryl nitrile oxides (**1**) *with 2-arylmethylene-1,3-indanediones* (**3**). *Spiro-compounds* **4**. *General procedure*: The enedione **3** (5 mmol) was dissolved in 20 ml of toluene in a 100 ml Erlenmeyer flask. The α-chlorooxime of the appropriate arylaldehyde (precursor to the 1,3-dipole **1**)[2,5] (5 mmol) was added, and to the magnetically stirred mixture anhydrous triethylamine (2 ml) was added dropwise. After refluxing for 24 h the precipitated triethylamine hydrochloride was filtered off and the solvent evaporated. The residue was dissolved in a toluene : ethanol (50 : 50) mixture from which the product **4** crystallised. For physical and spectroscopic properties see Table 2. Analytical data are listed in Table 4.

Cycloaddition reactions of diaryl nitrile imines **2** *with 2-arylmethylene-1,3-indanediones* (**3**). *Spiro-compounds* **5**. *General procedure*: The enedione **3** (5 mmol) was dissolved in 20 ml of toluene in a 100 ml Erlenmeyer flask. Then the α-chlorophenylhydrazone of the arylaldehyde (precursor to the 1,3-dipole **2**)[1,6] (5 mmol) was added, and to the magnetically stirred mixture anhydrous triethylamine (2 ml) was added dropwise. After refluxing for 24 h the precipitated triethylamine hydrochloride was filtered off and the solvent evaporated. The residue was dissolved in a toluene : ethanol (50 : 50) mixture from which the product **4** crystallised. For physical and spectroscopic properties see Table 3. Analytical data are listed in Table 4.

Table 3 Physical and spectroscopic data of the spiro[1,3,4-triaryl-2-pyrazoline-5:2'-1',3'-indanediones] **5**

Compound	R	R'	M.p. /ºC	Tield /%	IR/cm^{-1} $\nu_{C=O}$	$\nu_{C=N}$	$\nu_{C=Carom}$	^{1}H NMR/^{13}C NMR/δ ppm
5ae	H	H	136	86	1754, 1715	1595	1591	5.11 (s, 1H, H^4); 6.00–8.04 (m, 19 arom. H) 64.00 (C^4); 80.50 (C$^{5:2'}$); 194.50 (C=O); 197.50 (C=O).
5be	H	Me	142	70	1754, 1710	1595	1585	2.29 (s, 3H, Me); 5.16 (s, 1H, H^4); 6.60–8.10 (m, 18 arom. H) 21.10 (Me); 64.10 (C^4); 80.50 (C$^{5:2'}$); 194.60 (C=O); 197.60 (C=O)
5ce	H	OMe	162	80	1745, 1710	1665	1595	3.77 (s, 3H, OMe); 5.14 (s, 1H, H^4); 6.74–8.10 (m, 18 arom. H) 55.00 (OMe); 64.30 (C^4); 80.30 (C$^{5:2'}$); 194.50 (C=O); 197.60 (C=O)
5de	H	NO_2	216	84	1745, 1710	1590	1574	5.17 (s, 1H, H^4); 6.69–8.11 (m, 18 arom. H) 63.50 (C^4); 80.60 (C$^{5:2'}$); 193.60 (C=O); 196.60 (C=O)
5af	Me	H	146	85	1745, 1715	1595	1591	2.22 (s, 3H, Me); 5.13 (s, 1H, H^4); 6.74–8.16 (m, 18 arom. H) 21.00 (Me); 63.60 (C^4); 80.30 (C$^{5:2'}$); 194.30 (C=O); 197.40 (C=O)
5bf	Me	Me	196	60	1745, 1715	1597	1592	2.13 (s, 3H, Me); 2.19 (s, 3H, Me); 5.07 (s, 1H, H^4); 6.69–7.83 (m, 17 arom. H) 21.20 (Me); 21.40 (Me); 64.20 (C^4); 80.60 (C$^{5:2'}$); 194.70 (C=O); 197.70 (C=O)
5cf	Me	OMe	162	68	1745, 1715	1596	1592	2.17 (s, 3H, Me); 3.69 (s, 3H, OMe); 5.07 (s, 1H, H^4); 6.67–7.90 (m, 18 arom. H) 21.20 (Me); 55.30 (OMe); 64.30 (C^4); 80.50 (C$^{5:2'}$); 194.80 (C=O); 197.90 (C=O)
5df	Me	NO_2	256	80	1750, 1715	1595	1595	2.24 (s, 3H, Me); 5.15 (s, 1H, H^4); 6.68–8.12 (m, 17 arom. H) 21.00 (Me); 63.20 (C^4); 80.50 (C$^{5:2'}$); 193.60 (C=O); 196.70 (C=O)
5ag	OMe	H	196	90	1755, 1720	1600	1565	3.71 (s, 3H, OMe); 5.14 (s, 1H, H^4); 6.61–8.09 (m, 18 arom. H) 54.90 (OMe); 63.70 (C^4); 80.30 (C$^{5:2'}$); 194.50 (C=O); 197.50 (C=O)
5bg	OMe	Me	234	74	1755, 1720	1600	1595	2.29 (s, 3H, Me); 3.70 (s, 3H, OMe); 5.12 (s, 1H, H^4); 6.61–8.09 (m, 17 arom. H) 21.20 (Me); 54.90 (OMe); 63.70 (C^4); 80.30 (C$^{5:2'}$); 194.60 (C=O); 197.60 (C=O)
5cg	OMe	OMe	212	80	1755, 1720	1600	1596	3.71 (s, 3H, OMe); 3.76 (s, 3H, OMe); 5.22 (s, 1H, H^4); 6.74–7.50 (m, 17 arom. H) 54.90 (OMe); 55.00 (OMe); 63.70 (C^4); 80.40 (C$^{5:2'}$); 194.70 (C=O); 197.80(C=O)
5dg	OMe	NO_2	250	71	1755, 1720	1590	1586	3.72 (s, 3H, OMe); 5.15 (s, 1H, H^4); 6.68–8.12 (m, 17 arom. H) 54.90 (OMe); 62.90 (C^4); 80.50 (C$^{5:2'}$); 193.80 (C=O); 196.80 (C=O)
5ah	NO_2	H	254	51	1750, 1715	1590	1585	5.24 (s, 1H, H^4); 6.93–8.03 (m, 18 arom. H) 62.80 (C^4); 79.70 (C$^{5:2'}$); 194.10 (C=O); 196.80 (C=O)
5bh	NO_2	Me	258	50	1745, 1715	1600	1591	2.30 (s, 3H, Me); 5.20 (s, 1H, H^4); 6.91–8.04 (m, 17 arom. H) 21.20 (Me); 62.90 (C^4); 79.60 (C$^{5:2'}$); 194.30 (C=O); 196.40 (C=O)
5ch	NO_2	OMe	196	50	1745, 1715	1600	1591	3.65 (s, 3H, OMe); 5.13 (s, 1H, H^4); 6.67–8.04 (m, 17 arom. H) 55.40 (OMe); 63.10 (C^4); 79.80 (C$^{5:2'}$); 194.30 (C=O); 196.90 (C=O)
5dh	NO_2	NO_2	>264	50	1750, 1715	1595	1591	5.24 (s, 1H, H^4); 6.69–8.14 (m, 17 arom. H) 62.10 (C^4); 79.90 (C$^{5:2'}$); 193.40 (C=O); 196.00 (C=O)

Table 4 Analytical data of the spiro compounds **4** and **5**

Compound	Molecular formula	Found/calc./% C	H	N	Compound	Molecular formula	Found/calc. /% C	H	N
4ae	$C_{23}H_{15}NO_3$	78.35	4.36	3.92	**5ae**	$C_{29}H_{20}N_2O_2$	81.42	4.74	6.53
		78.17	4.28	3.96			81.29	4.70	6.54
4be	$C_{24}H_{17}NO_3$	78.53	4.49	3.90	**5be**	$C_{30}H_{22}N_2O_2$	81.37	5.02	6.33
		78.46	4.66	3.81			81.43	5.01	6.33
4ce	$C_{24}H_{17}NO_4$	75.02	4.54	3.67	**5ce**	$C_{30}H_{22}N_2O_3$	78.85	4.85	6.09
		75.19	4.47	3.65			78.59	4.84	6.11
4de	$C_{23}H_{14}N_2O_5$	69.41	3.62	7.08	**5de**	$C_{29}H_{19}N_3O_4$	73.69	4.13	8.85
		69.34	3.54	7.03			73.56	4.04	8.87
4af	$C_{24}H_{17}NO_3$	78.59	4.61	3.93	**5af**	$C_{30}H_{22}N_2O_2$	81.62	5.13	6.45
		78.46	4.66	3.81			81.43	5.01	6.33
4bf	$C_{25}H_{19}NO_3$	78.67	5.11	3.64	**5bf**	$C_{31}H_{24}N_2O_2$	81.42	5.24	6.28
		78.72	5.02	3.67			81.56	5.30	6.14
4cf	$C_{25}H_{19}NO_4$	75.46	4.96	3.53	**5cf**	$C_{31}H_{24}N_2O_3$	78.97	5.22	5.87
		75.55	4.82	3.52			78.79	5.12	5.93
4df	$C_{24}H_{16}N_2O_5$	70.03	3.82	6.84	**5df**	$C_{30}H_{21}N_3O_4$	73.63	4.39	8.71
		69.90	3.91	6.79			73.91	4.34	8.62
4ag	$C_{24}H_{17}NO_4$	75.07	4.53	3.70	**5ag**	$C_{30}H_{22}N_2O_3$	78.77	4.81	6.15
		75.19	4.47	3.65			78.59	4.84	6.11
4bg	$C_{25}H_{19}NO_4$	75.29	4.91	3.51	**5bg**	$C_{31}H_{24}N_2O_3$	78.58	5.08	6.02
		75.55	4.82	3.52			78.79	5.12	5.93
4cg	$C_{25}H_{19}NO_5$	72.73	4.69	3.44	**5cg**	$C_{31}H_{24}N_2O_4$	76.32	4.99	5.73
		72.63	4.63	3.39			76.21	4.95	5.73
4dg	$C_{24}H_{16}N_2O_6$	67.14	3.79	6.63	**5dg**	$C_{30}H_{21}N_3O_5$	71.81	4.33	8.29
		67.29	3.76	6.54			71.56	4.20	8.35
4ah	$C_{23}H_{14}N_2O_5$	69.24	3.66	6.92	**5ah**	$C_{29}H_{19}N_3O_4$	73.62	4.08	8.83
		69.34	3.54	7.03			73.56	4.04	8.87
4bh	$C_{24}H_{16}N_2O_5$	69.71	3.97	6.84	**5bh**	$C_{30}H_{21}N_3O_4$	73.82	4.31	8.63
		69.90	3.91	6.79			73.9	4.34	8.62
4ch	$C_{24}H_{16}N_2O_6$	67.07	3.70	6.62	**5ch**	$C_{30}H_{21}N_3O_5$	71.66	4.23	8.29
		67.29	3.76	6.54			71.56	4.20	8.35
4dh	$C_{23}H_{13}N_3O_7$	62.55	2.90	9.51	**5dh**	$C_{29}H_{18}N_4O_6$	67.27	3.59	10.83
		62.31	2.96	9.48			67.18	3.50	10.81

Received 27 May 2003; accepted 3 December 2002
Paper 2/1412

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