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A new strategy for the construction of polycycles bearing a nitrogen atom on the ring fusion†

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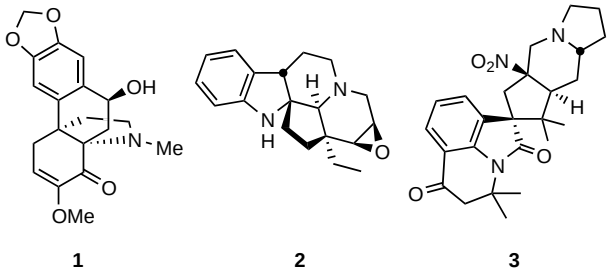
Abstract—Nitroethylenes bearing either a phenylthio or a phenylselenoether group in β-position are efficient synthetic equivalents of nitroacetylene. A [4+2] cycloaddition/carbenoid-mediated elimination of $PhS(e)CH_3$/[4+2] cycloaddition sequence of reactions is shown to produce formal biscycloadducts of nitroacetylene in high yields. The cycloaddition reactions are activated by high-pressure and proceed with high regioselectivities and stereoselectivities.

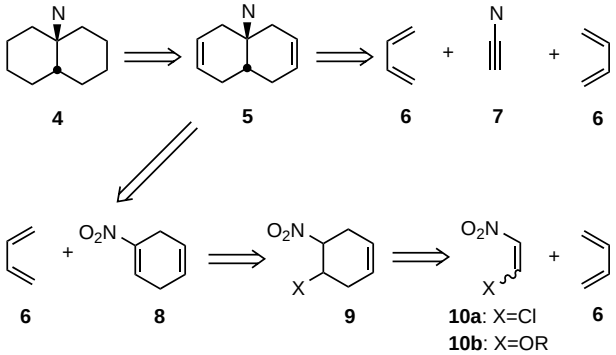
The realm of alkaloids is replete with polycyclic structures featuring a nitrogen atom on a ring junction. Prostephanaberrine (**1**), andrangine (**2**) and cyclopiamine A (**3**) are examples of such molecules (Fig. 1). The presence of a nitrogen on a quaternary carbon constitutes a synthetic difficulty which has been overcome by different ways. Thus, nucleophilic 1,2-shifts (including the Curtius, Hoffmann and Lossen rearrangements) have long been known, as having been the formation of tertiary azides form the corresponding alcohols and bromides.[1] More recently, oxidative methods have formally allowed the replacement of a tertiary hydrogen with an azide group.[2] Finally, [2,3] sigmatropic rearrangements of allylic nitro compounds have been used to generate products with this particular substitution pattern.[3] In the course of a program dedicated to the preparation of some of the above-cited natural products, we developed a new strategy for the construction of polycycles bearing a nitrogen atom at the ring junction, the account of which is reported hereafter.



1 2 3

Figure 1.

Retrosynthetic analysis of bicycle **4**, with the aim of exploiting [4+2] cycloaddition reactions, points the finger to the use of nitrogen-substituted acetylene synthon **7**, for which the best representative would be the unstable nitroacetylene (Scheme 1).[4] Only two examples of sterically hindered nitroalcynes have so far been shown to be stable enough to work with.[4,5] Potential synthetic equivalents of nitroacetylenes have however been reported for several decades and are characterised by the general structure **10**, in which the β-substituent may be either a chlorine atom, a silyl, ether (ester) or amino group, or a thio or selenoether function, as well



Scheme 1.

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† Dedicated to Professor Heinz-Günter Viehe.

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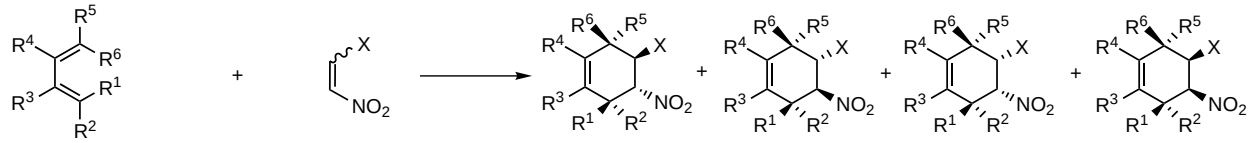
as some of their oxidised analogues (sulfoxide, sulfone).[6] The reactivity of some of these substrates has been successfully exploited in [4+2] cycloaddition processes to deliver the expected cycloadducts. The elimination of the X substituents of compounds **9** would regenerate a nitroalcene whose reactivity could be exploited in a second [4+2] cycloaddition. To the best of our knowledge, the only reported example of a related process involves the interaction of β-chloronitroethylene with a nitrone; thermal loss of HCl from the first cycloadduct induces the formation of two diastereoisomeric biscycloadducts in low yields.[6a] All the other published elimination reactions of substituent X from cycloadducts **9** result in the oxidative production of the corresponding aromatic compounds.[6b,c,f,g]

Thioether **10e** and selenoether **10f** appear to be candidates of choice for our strategy, based on Ono's work as well as the possibility of conducting either base-induced elimination of phenylthiol or phenylselenol, or the oxidation/*syn*-elimination sequence of reactions.

(*E*)-β-Phenylthionitroethylene **10e** has been reported to react satisfactorily with cyclopentadiene **11a** to deliver the expected cycloadduct **12a** (75% isolated yield); however, no reaction occurred with (*E*)-1,3-pentadiene.[6d] In our hands and under similar conditions, this nitroalcene produced **12a** and **13a** in nearly quantitative yield, with a 9:1 diastereoselection in favor of the *endo* isomer (Scheme 2) (Table 1, entry 1). Interaction with cyclohexadiene or 2,3-dimethylbutadiene similarly furnished the corresponding cycloadducts in good yields (entries 2 and 3). The thermal reactions between (*Z*)-β-phenylselenonitroethylene **10f** and **11a** or **11c** proved to be sluggish. Thus heating the mixtures of reactants at 110–120°C for periods of time of one to eleven days resulted in the formation of four cycloadducts in poor yields (entries 4 and 5). Cycloadducts **12** and **13** are believed to arise from in situ base- or acid-catalysed isomerisation of the initial cycloadducts **15** and **14**, respectively.

The highly negative activation volumes characterising the [4+2] cycloaddition processes have generated a large body of results which unambiguously demonstrate the strong acceleration of this type of transformations, when conducted under hyperbar conditions.[7] Compression of a mixture of nitroalcene **10e** and diene **11a** to 12 kbar for 72 h at 25°C resulted in the formation of the same cycloadducts **12a** and **13a** in very good yield (Table 2, entry 1). The stereoselectivity was improved with an *endo*/*exo* ratio of 95/5. Similarly and under a pressure of 16 kbar, cyclohexadiene delivered the adducts in fair yields and with a high diastereoselection (entries 2 and 3). Under a pressure of 12 kbar, the selenoether analogue reacted with **11a** once again more slowly, leading to a 33% yield of all four stereoisomers (see above; entry 4) as well as 54% of unconsumed starting material. Increasing the pressure to 16 kbar, however, furnished an 82% isolated yield of **12**, **13** and **14** (entry 5). Immediately treating this mixture with triethylamine converted **14** into **13**, which was isolated in 76% yield (entry 6). Beneficial activation of the reaction by high pressures was confirmed with other



11a: R^1, R^6=CH_2; R^2=R^3=R^4=R^5=H
11b: R^2=CH_3; R^1=R^3=R^4=R^5=R^6=H
11c: R^1, R^6=CH_2-CH_2; R^2=R^3=R^4=R^5=H
11d: R^3= R^4=CH_3; R^1=R^2=R^5=R^6=H
11e: R^4=CH_3; R^1=R^2=R^3=R^5=R^6=H
11f: R^2=OCH_3; R^4=$OSiMe_3$; R^1=R^3=R^5=R^6=H
11f: R^2, R^3=CH=CH-CH=CH; R^1=R^4=R^5=R^6=H

10e: X=SPh
10f: X=SePh

12 **13** **14** **15**

Scheme 2.

Table 1. Thermal activation of the [4+2] reactions between **10** and **11**

Entry		Diene	Conditions[a]	Yields (%)[b]		Products ratio				**10**[c]
						12	**13**	**14**	**15**	
1	**10e**	**11a**	110°C, 4 h	95	**a**	88	12	–	–	0
2	**10e**	**11c**	130°C, 72 h	75	**b**	80	20	–	–	0
3	**10e**	**11d**	130°C, 24 h	98	**c**	100	–	–	–	0
4	**10f**	**11a**	110°C, 24 h	35	**d**	31	26	34	9	62
5	**10f**	**11c**	120°C, 264 h	16	**e**	–	–	25	75	12

[a] Reaction conditions.
[b] Isolated yields.
[c] Unconsumed dienophile.

Table 2. Hyperbar activation of the [4+2] reactions between **10** and **11**

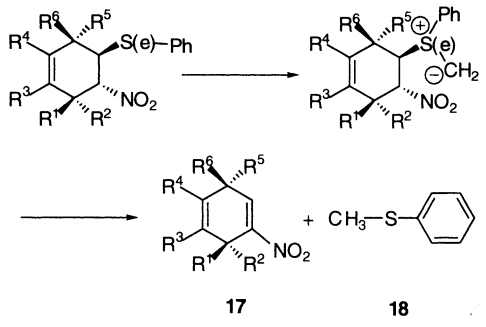
Entry	Substrate	Diene	Conditions	Yields (%)[a]	Ratio of cycloadducts				Recovered starting material **10**
					12	**13**	**14**	**15**	
1	**10e**	**11a**	12 kbar, 25°C, 72 h	82	**a** 95	5	–	–	0
2	**10e**	**11c**	12 kbar, 25°C, 48 h	33	**b** 84	16	–	–	66
3	**10e**	**11c**	16 kbar, 25°C, 24 h	49	**b** 99	1	–	–	0
4	**10f**	**11a**	12 kbar, 50°C, 96 h	33	**d** 25	12	63	–	54
5	**10f**	**11a**	16 kbar, 25°C, 48 h	82	**d** 12	5	83	–	0
6	**10f**	**11a**	16 kbar, 25°C, 48 h, then triethylamine	76	**d** –	100	–	–	0
7	**10f**	**11c**	12 kbar, 50°C, 96 h	65	**e** –	–	46	54	24
8	**10f**	**11c**	16 kbar, 25°C, 24 h	70	**e** –	13	66	21	0
9	**10f**	**11d**	12 kbar, 50°C, 96 h	39	**f** –	–	100	–	20
10	**10f**	**11d**	16 kbar, 25°C, 48 h	92	**f** –	25	75	–	0
11	**10f**	**11e**	12 kbar, 50°C, 96 h	71	**g** –	–	100	–	0
12	**10f**	**11e**	16 kbar, 25°C, 24 h	48	**g** –	50	50	–	12
13	**10f**	**11f**	12 kbar, 50°C, 96 h	96[b]	**h** –	–	–	–	0

[a] Isolated yields.
[b] *p*-Nitrophenol; see text.

dienes. Thus cyclohexadiene, isoprene and 2,3-dimethylbuta-1,3-diene all reacted well at 12 or 16 kbar (entries 7–12). In some cases, increasing the pressure to 16 kbar had a deleterious effect, due to the competitive polymerisation of the diene under those conditions (entry 12). Danishefski's diene **11f** led to the formation of *p*-nitrophenol, resulting from the loss of methanol, phenylselenol and tautomerisation of the ketone (entry 13).

Application of our carbenoid methodology (Scheme 3) to transform cycloadduct **12a** to 2-nitronorbornadiene **17a** failed completely (entry 1, Table 3).[8] Intractable mixtures of compounds were invariably produced, and not a trace of the desired product **17a** could be detected by ^{1}H NMR spectrometry.

Reduction of the carbon–carbon double bond with diimide led to 2-nitro-3-phenylthionorbornane (**19a**), isolated in quantitative yield. Similarly, cycloadduct **12b** was transformed into the corresponding dihydroderivative **19b**. Treatment of **19a** with diethylzinc and trifluoroacetic acid now smoothly produced nitroalcene **17b** in 72% yield. Thus the strain associated with structure **17a** renders the product prone to undergoing polymerisation as well as other side-reactions (Scheme 3). Application of the procedure to cycloadducts **12b**, **12c**, **14f** and **19b** delivered the required nitroalkenes in good yields (Table 3).



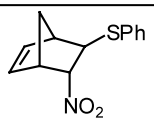
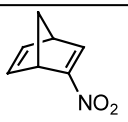
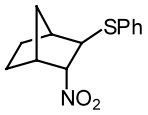
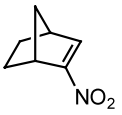
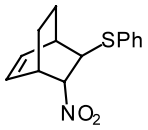
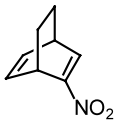
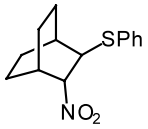
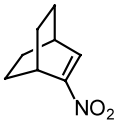
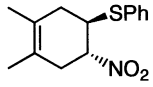
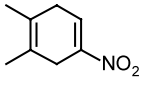
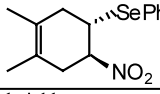
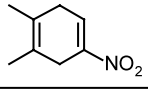
Scheme 3.

The second cycloaddition process was carried out with 2-nitronorborn-2-ene (**17b**) under thermal and hyperbar conditions (Scheme 4, Table 4). Heating at 130°C for several hours is required to attain completion. Under these conditions only cycloadducts **20a** and **20b** could be isolated in high yields (entries 1 and 5); competitive degradation of **17b** or polymerisation of the diene occurred in all other cases, resulting in much lower yields (entries 4, 6 and 10).

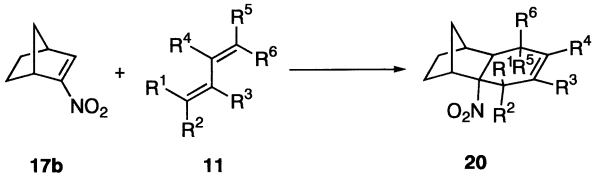
Here again, high-pressure activation proved to be the method of choice to obtain the desired adducts. Thus, for example, while thermal activation of the reactions between **17b** and dienes **11f** or **11g** led to disappointing results, compressing the mixtures of reactants to 12 kbar afforded the products in fair yields (compare entries 6 with 7 and 10 with 11). Even better results were obtained under 16 kbar (entries 8, 9 and 12). It is noteworthy that only one equivalent of Danishefski's diene is necessary to produce the desired adduct in good yield (entry 9).

In each case, a single stereoisomer was produced, demonstrating the high regioselectivity (entries 6–12) and stereoselectivity of the process (entries 1–9). Interaction between the diene and the dienophile occurs exclusively from the less hindered convex face of the latter; there are precedents in the literature for such behavior.[9] In addition, cyclopentadiene and cyclohexadiene are approaching the nitroalkene in an *endo*, less sterically demanding manner (**21**, Fig. 2). The same is true for Danishesky's diene **11f**; in both cases a single diastereoisomer is produced. Assignment of the related stereocentres in **20d** was achieved using 1D and 2D ^{1}H NMR spectrometry techniques. Thus, for example, hydrogens on carbon 5 were coupled with hydrogen 4a

Table 3. Preparation of nitroalkenes **17**

entry	substrate		product		yields (%)[a]
1		**12a**		**17a**	0
2		**19a**		**17b**	72
3		**12b**		**17c**	79
4		**19b**		**17d**	65
5		**12c**		**17e**	49
6		**14f**		**17e**	50

a. isolated yields.



17b **11** **20**

Scheme 4.

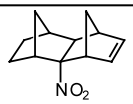
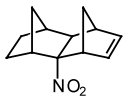
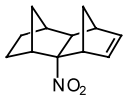
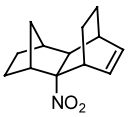
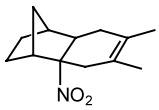
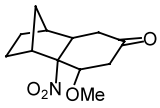
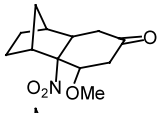
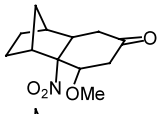
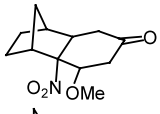
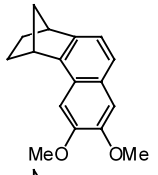
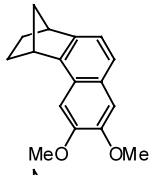
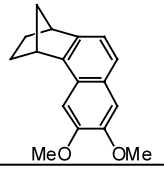
(2.28 and 12.8 Hz, respectively) thereby indicating the depicted regiochemistry (Fig. 2). A strong NOE effect was detected between hydrogen 8 and one of the hydrogen nucleus on carbon 9, indicative of the *cis* relationship between the methoxy and the nitro groups.

In summary, we have shown that nitroethylenes bearing either a phenylthio or a selenoether group in β-position are efficient synthetic equivalents of nitroacetylene. A [4+2] cycloaddition/carbenoid-mediated elimination of $PhS(e)CH_3$/[4+2] cycloaddition sequence of reactions has been developed to produce formal biscycloadducts or nitroacetylene. The cycloaddition processes are activated by high-pressure and are characterised by high regioselectivities and stereoselectivities. The resultant methodology may find application in the synthesis of alkaloids and related natural products. Work is currently in progress to develop these applications.

Supplementary material

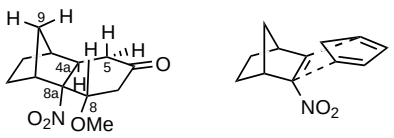
^1H NMR (300 MHz) and ^{13}C NMR (75 MHz) spectra were recorded in deuterated chloroform on a Brucker

Table 4. Cycloaddition between **18a** and dienes **11**

entry	Diene[a]	product		conditions	yields (%)
1	**11a**		**20a**	130°C, 3 hrs	81
2	**11a**		**20a**	12 kbar, 25°C, 96 hrs	70
3	**11a**		**20a**	16 kbar, 25°C, 96 hrs	71
4	**11c**		**20b**	130°C, 2 hrs	9
5	**11d**		**20c**	130°C, 3 hrs	86
6	**11f**		**20d**	130°C, 4 hrs	30
7	**11f**		**20d**	12 kbar, 25°C, 96 hrs	53
8	**11f**		**20d**	16 kbar, 25°C, 96 hrs	75
9	**11f**		**20d**	16 kbar, 25°C, 96 hrs	65 [b]
10	**11g**		**20e**	130°C, 4 hrs	10
11	**11g**		**20e**	12 kbar, 25°C, 96 hrs	44
12	**11g**		**20e**	16 kbar, 25°C, 96 hrs	59

a) 6 equivalents of diene were used. b. Carried out with 1 equivalent of diene.

Avance 300 spectrometer relative to $(CH_3)_4Si$ and $CDCl_3$, respectively. Chemical shifts are expressed in parts per million (ppm). Low- and high-resolution mass spectra were recorded on Unicam ATI Automass and Jeol 500 spectrometers, respectively. IR spectra were



20d **21**

Figure 2.

recorded as chloroform solutions on Perkin–Elmer 16PC FT-IR spectrometers and are expressed in cm^{-1}.

Representative procedure for the thermal reactions

(*E*)-1-Nitro-2-phenylthioethylene (2.0 g, 11.0 mmol), cyclopentadiene (10.0 ml, 7.97 g, 120 mmol), and a 0.1 M solution hydroquinone (1 mL) in tetrahydrofuran (1 mL) are placed in a sealed tube and heated at 110°C for 2 h. The mixture is cooled down to room temperature and the volatiles are evaporated under reduced pressure. Chromatography of the residue and elution with heptane/ethyl acetate (95:5) leads to the isolation of pure, colorless cycloadduct **12**.

General procedure for the hyperbar reactions

The requisite nitroalcene (**10e** or **10f**, 0.5 mmol), the diene (6.0 mmol), and a 0.1 M solution hydroquinone (0.1 mL) in dichloromethane (1 mL) are placed in a 2 mL high pressure reactor. Compression to the required pressure and temperatures is achieved for the time needed (see Tables 1–4). Release of the pressure and work-up as above afford the desired product.

*Analytical data for compounds **12a**, **19a**, **17b** and **20a***

Compound **12a**: ^{1}H NMR δ 7.50–7.40 (m, 2H); 7.38–7.23 (m, 3H); 6.44 (dd, $^3J=5.64$, 3.00 Hz, 1H); 6.04 (dd, $^3J=5.64$–2.64 Hz, 1H); 4.87 (t, $^3J=4.76$ Hz, 1H); 3.77 (t, $^3J=3.00$ Hz, 1H); 3.58 (m, 1H); 2.99 (s, 1H); 2.01 (d, $^2J=9.81$ Hz, 1H); 1.77 (d, $^2J=9.81$ Hz, 1H). ^{13}C NMR δ 139.42; 134.62; 133.58; 131.86; 129.70; 127.98; 91.89; 50.32; 49.33; 48.57; 46.92. IR ν 2986, 1548, 1372, 738, 714, 692. Anal. calcd for $C_{13}H_{13}NO_2S$: C, 63.14; H, 5.30; N, 5.66; S, 12.96. Found: C, 63.00; H, 5.41; N, 5.92; S, 13.04%.

Compound **19a**: ^{1}H NMR δ 7.46–7.36 (m, 2H); 7.35–7.21 (m, 3H); 4.69 (m, 1H); 3.97 (dd, $^3J=3.39$, 3.03 Hz, 1H); 2.97 (m, 1H); 2.42 (d, $^2J=4.53$ Hz, 1H); 1.99 (d, $^2J=10.53$ Hz, 1H); 1.84–1.68 (m, 1H); 1.62–1.38 (m, 3H); 1.36–1.20 (m, 1H). ^{13}C NMR δ 134.39; 131.62; 129.67; 127.79; 94.29; 51.58; 43.90; 43.62; 37.08; 28.47; 22.81. Anal. calcd for $C_{13}H_{15}NO_2S$: C, 62.62; H, 6.06; N, 5.62; S, 12.86. Found: C, 62.38; H, 6.09; N, 5.75; S, 12.92%.

Compound **17b**: ^{1}H NMR δ 7.01 (d, $^3J=7.40$ Hz, 1H); 3.49 (m, 1H); 3.15 (m, 1H); 2.00–1.79 (m, 2H); 1.69 (d, $^3J=4.89$ Hz, 1H); 1.49–1.12 (m, 2H,). ^{13}C NMR δ 141.27; 48.15; 43.65; 42.11; 25.52; 24.70, quaternary carbon undetected. These data are identical to those reported in the literature. See: Corey, E. J.; Estreicher, H. *J. Am. Chem. Soc.* **1978**, *100*, 6294–6295.

Compound **20a**: ^{1}H NMR δ 6.22 (dd, $^3J=5.28$, 3.00 Hz, 1H); 6.14 (m, $^3J=5.28$, 3.75 Hz, 1H); 3.47 (m, 1H); 3.04 (m, 1H); 2.94 (m, 1H); 2.70 (m, 1H); 2.40 (dd,, $^2J=8.28$, $^3J=2.25$ Hz, 1H); 2.18 (d, $^3J=1.51$ Hz, 1H); 1.70–1.13 (m, 5H); 1.12–1.00 (m, 1H); 0.91 (dd, $^2J=10.92$, $^3J=1.11$ Hz, 1H). ^{13}C NMR δ 140.03; 134.51; 108.33; 55.04; 52.44; 51.61; 47.38; 44.76; 39.13; 37.64; 30.31; 27.45. IR ν 2968, 1724, 1528, 1458, 1356, 756. Anal. calcd for $C_{12}H_{15}NO_2$: C, 70.22; H, 7.37; N. 6.82. Found: C, 69.84; H, 7.28; N. 6.91%.

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