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The reactions of 3,7-dimethylenebicyclo[3.3.1]nonane, norbornadiene and *cis*,*cis*-1,5-cyclooctadiene with pentafluoro-λ^6-sulfanyl chloride

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ABSTRACT

Radical transannular cyclizations of the non-conjugated dienes, such as 3,7-dimethylenebicyclo[3.3.1]-nonane and norbornadiene with SF_5Cl upon UV-irradiation led to the corresponding SF_5-substituted 3,7-noradamantane and nortricyclanes with high yields. Radical reaction of *cis*,*cis*-1,5-cyclooctadiene with SF_5Cl led to a product of SF_5Cl addition to one of the diene double bonds either UV-irradiation or triethylborane were used for radical initiation.

1. Introduction

Introduction of a SF_5 group into organic compounds has been known for over 50 years ago, but the number of SF_5-containing compounds is limited by SF_5 sources or building blocks. Compounds containing SF_5 group possess unique properties due to the low surface energy, high chemical resistance, thermal stability, high electronegativity and lipophilicity [1–6]. These properties could lead to potential biological activity of SF_5-based compounds. A number of patents describe the properties of SF_5-derivatives as fungicides, herbicides and insecticides [4,7,8]. An SF_5 analogue of the insecticide Fipronil [9] has a significantly higher activity than the corresponding CF_3 compound.

Important and convenient methods for the addition of the SF_5 group into organic compounds are based on radical addition of SF_5Cl to unsaturated substrates using thermolysis [10], UV-irradiation [5] and $(CH_3CH_2)_3B$ as a low-temperature initiator [11–12]. The prepared addition products are widely used for the synthesis of various classes of SF_5-based compounds (alcohols, aldehydes, ketones, acids, aromatic, alkenes, acetylenes, hetero-cycles); but only a relatively few SF_5-carbocycle compounds with norbornane cage have been synthesized [5,13].

Among the alkenes, the non-conjugated polyenes are of special interest because their transformations lead to various biologically active polycyclic compounds [14,15]. Polycycles with adamantyl and noradamantyl moieties are often used as building blocks for new drugs with various types of physiological activity [16–19]. Derivatives including nortricyclane and norbornane bridged cycles are structurally similar to natural terpenes. A number of such polycycles show activities for the treatment or the prevention of neurologic diseases [20–23].

Convenient and selective preparations of functional derivatives of adamantane and noradamantane are based on transannular cyclizations of bicyclo[3.3.1]nonane dienes with electrophilic [24–28] or radical agents [29–31]. The transannular cyclizations of norbornadiene with electrophilic and radical reagents [32–36] are used for selective preparation of nortricyclane derivatives. In contrast to norbornadiene reactions, the selectivity of *cis*,*cis*-1,5-cyclooctadiene reactions and structure of the formed products depend strongly on the nature of the reagents, solvents and the presence of catalysts or initiators [35–39]. Products of the addition to one double bond along with the cyclization products to bicyclo[3.3.0]octane derivatives are characteristic for radical reactions of *cis*,*cis*-1,5-cyclooctadiene [35–38].

To the best of our knowledge no reactions of non-conjugated dienes with SF_5Hlg (Hlg = Cl, Br) leading to cyclization products have been reported. The purpose of this work was to examine the

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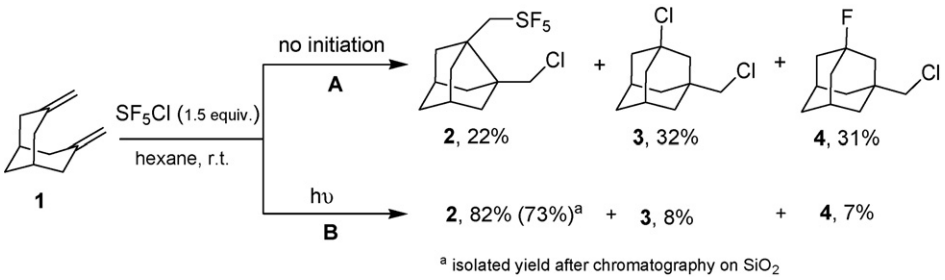


Scheme 1. The yields were determined by NMR and GS–MS.

effectiveness of electrophilic SF_5 radicals in reactions of non-conjugated dienes with different cyclization possibilities, such as 3,7-dimethylenebicyclo[3.3.1]nonane, norbornadiene and *cis*,*cis*-1,5-cyclooctadiene.

2. Results and discussion

2.1. Transannular cyclization 3,7-dimethylenebicyclo[3.3.1]nonane with SF_5Cl

Cyclization of 3,7-dimethylenebicyclo[3.3.1]nonane (**1**) with SF_5Cl in dry *n*-hexane proceeded for 3 h at ambient temperature without any special initiation and gave a mixture of three products **2**–**4**, in similar yields: the desired SF_5-substituted noradamantane **2** and two adamantane derivatives, **3** as Cl_2 addition product and **4** as a "Cl–F" addition product (Scheme 1, path **A**).

The ^1H NMR spectrum of product **2** shows two characteristic signals, a singlet of the CH_2Cl group at 3.55 ppm and a pentet for the CH_2SF_5 group at 3.94 ppm (J = 9.0 Hz). In the ^{19}F NMR spectrum of **2** the typical appearance of the AB_4-spin system for the SF_5-substituent was observed with nine lines for the apical fluorine atom (A-part) at 85.1 ppm and a doublet of multiplets of the basal fluorines (B_4-part) at 69.4 ppm (J = 143.0 Hz). The characteristic signals of adamantanes **3** and **4** in NMR spectra {^1H NMR: **3** (s, 3.28 ppm CH_2Cl), **4** (s, 3.32 ppm CH_2Cl) and ^{19}F NMR **4** (s, −133.8 ppm the fluorine atom in the bridgehead position)} are in agreement with those reported for **3** [40] and **4** [28].

It has been previously shown, that transannular cyclization of bicyclo[3.3.1]nonane dienes with radical agents (CCl_4/AIBN [29], C_6H_5SH, CH_3PhSO_2Hlg (Hlg = Cl, Br) [30]) and with polyfluoroalkyl radicals [31] gives mainly noradamantane derivatives. Using the DFT method, we have established that formation of noradamantane derivatives at cyclization of diene **1** with electrophilic trifluoromethyl radical corresponds to kinetic control [31]. This allowed us to assign of noradamantane product **2** forming through the radical mechanism. We consider, that formation of radicals without special initiation of reaction diene **1** with SF_5Cl (Scheme 1, pathway A), proceeds through the reagents interaction in accordance to olefin-induced homolysis [41]. The latter can be proved by a considerable increased yield of the SF_5-product **2** by carrying out the reaction of **1** with SF_5Cl upon irradiation with a Hg lamp in quartz apparatus (Scheme 1, path **B** and Fig. 1).

The formation of products **3** and **4** with the adamantane framework points to a polar mechanism of the electrophilic transannular cyclization of diene **1** with molecular chlorine and "F–Cl" accordingly (Fig. 1). This mechanism is well known for many Ad_E reactions of bicyclo[3.3.1]nonane dienes with electrophilic agents [24–28]. We suppose, that molecular chlorine was formed by recombination of chlorine atoms or reaction of $Cl^•$ and SF_5Cl. Participation of the molecular chlorine in the formation of product **3** is confirmed by the data from paper [42], in which dichloride **3** was obtained by dry chlorine passing through a solution of diene **1** in CCl_4. In contrary, cyclization of diene **1** with a chlorine atom and a $^•CCl_3$ radical obtained by homolysis of CCl_4 with AIBN [29] gives mostly a chlorononoradamantane derivative as the major product. The formation of "F–Cl" addition products in reactions of SF_5Cl [10] with olefins has been well known. This is supported by DFT computation that showed that the SF_5Cl molecule dissociates easier to the interhalogen FCl and SF_4 than to the radical products $^•SF_5$ and $Cl^•$ or $^•SF_4Cl$ and $F^•$ [43].

So, carrying out the reaction of **1** with SF_5Cl upon UV-irradiation (Scheme 1, way **B**) is an effective route for increasing the yield of product **2**, and for decreasing the yields of **3** and **4** in the electrophilic reactions.

2.2. The transannular cyclization of norbornadiene with SF_5Cl

Norbornadiene **5** and SF_5Cl upon irradiation with a Hg lamp in quartz apparatus led to formation of cyclization products **6** and **7** in high yields (Scheme 2).

Products of the "F–Cl" addition were not observed in the ^{19}F NMR spectra. Nortricyclane **6** was separated from the reaction mixtures by silica gel column chromatography. The formation of the product **7** was confirmed by ^1H and ^{19}F NMR spectra. It is well known from the literature that proton signals H–C^5 for *endo*-3-halogen,*exo*-5 substituted nortricyclanes are downfield shifted relative to H–C^5 signals of *exo*-3-halogen,*exo*-5 isomers [33,35,36]. In the ^1H NMR spectrum of **7** the pentet of the H–C^5 proton signal (4.54 ppm, J = 8 Hz) is downfield shifted relative to H–C^5 proton signal of **6** (3.81 ppm, J = 8 Hz). Moreover, the ^1H NMR spectra of product **6** and **7** show the characteristic singlets of the C^3HCl group protons, at 3.88 and 3.99 ppm respectively. In the ^{19}F NMR spectra of **6** and **7** the typical appearance of the AB_4-spin system for the

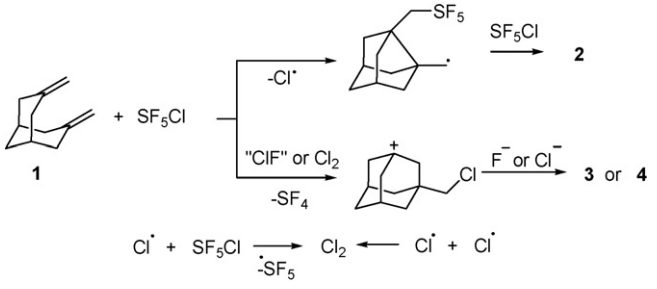
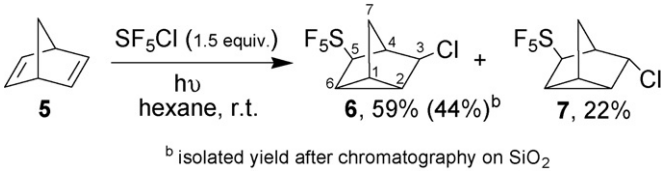
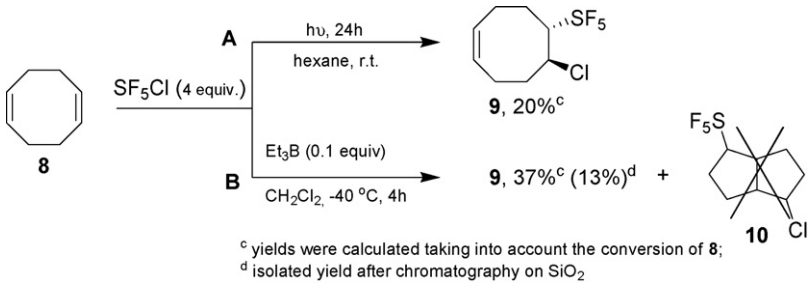


Fig. 1. The cyclization mechanism of 3,7-dimethylene[3.3.1]nonane (**1**) with SF_5Cl without initiation.



Scheme 2. The yields were determined by NMR and GS–MS.



Scheme 3. The yields were determined by NMR and GS–MS.

SF_5-substituent were observed with nine lines for the apical fluorine atom (A-part, at 83.2 ppm for **6** and 84.4 ppm for **7**), and the doublets of multiplets of the basal fluorines (B_4-part, at 59.1 ppm with J = 144.9 Hz for **6**, and at 61.8 ppm with J = 144.9 Hz for **7**).

It should be pointed out that the ratio of *exo*-3,*exo*-5 substituted nortricyclanes **6** and *endo*-3,*exo*-5 substituted nortricyclanes **7** (**6**/**7** = 2.7) in the reaction of diene **5** with SF_5Cl (Scheme 2) is higher than for most of the well known radical reactions of norbornadiene, e.g. *exo*-3,*exo*-5/*endo*-3,*exo*-5 product ratios lie in the range of ~0.94–1.3 for reactions of **5** with perfluoroalkylhalides (chloro, iodo) [32,36], $HlgCCl_3$ (Hlg = Cl, Br) [35] or RSO_2Cl [35]. Probably, the sterically demanding SF_5 group increases the stereoselectivity of radical addition reaction to norbornadiene and leads to the high ratio of the isomers **6**/**7** = 2.7.

2.3. The reaction of *cis*,*cis*-1,5-cyclooctadiene with SF_5Cl

The reaction of *cis*,*cis*-1,5-cyclooctadiene (**8**) with SF_5Cl upon UV-irradiation could not be completed for 24 h even when a four-fold excess of the reagent was used. Conversion of this reaction was 60%. The complicated mixture of products was observed in the ^{19}F and ^1H NMR spectra (Scheme 3, path **A**). From the four SF_5-substituted products (^{19}F NMR) the main product **9** originates from the SF_5Cl addition to the one double bond of the starting diene (^{19}F NMR: B_4-part at 54.1 ppm with J = 143.1 Hz, nine lines of A-part, at 85.8 ppm). The SF_5Cl (four-fold excess) addition reaction to diene **8** was rather more effective (93% conversion of **8**) using triethylborane (0.1 equiv.) for radical initiation in CH_2Cl_2 solution at −40 °C, this method was proposed by Dolbier and co-workers for improving the addition reaction of SF_5Cl to alkenes and alkynes [11,12]. Only the SF_5-product **9** was observed in ^{19}F NMR of the complicated reaction mixture (Scheme 3, path **B**). There was no SF_5-substituted cyclization product **10**, and in the ^{19}F NMR of the reaction mixture multiplets of C–F products were observed (range −169 to 177 ppm for comparison [39]). The use of a 1.5-fold excess of SF_5Cl in the paths **A** and **B** (Scheme 3) led to lower conversions (~30–40%) of *cis*,*cis*-1,5-cyclooctadiene, the latter was observed in ^1H NMR of the corresponding reaction mixtures after the reactions.

The ^1H NMR spectrum of product **9** shows a broad singlet of the CHCl group at 5.12 ppm, a multiplet of the $CHSF_5$ group at 4.31 ppm (J = 9 Hz) and two multiplets of HC=CH group at 5.63 and 5.90 ppm.

Similar to our data with an excess of sulfur chloride pentafluoride, only one equivalent reacts with butadiene [10]. Furthermore, products of the addition to the only one double bond of the diene **8** along with cyclization products were reported in the literature [35–39]. It might be supposed that the addition of the second equivalent of the reagent and especially a bulky radical $^{\bullet}SF_5$ is hindered by steric interference with the substituents in the cyclooctene ring.

So, we see the opposite tendencies of the dienes **1**, **3** and **8** to cyclization and it is consistent with empiric Baldwin rules. According to them both 6- and 7-*endo*-*exo*-*trig*-cyclizations are favorable, but neither of 3- and 5-*endo*-*exo*-*trig*-cyclizations are favorable [44]. The physical sense of these rules lies in the stereo-electronic requirements to the transitional cyclization state. According to the Fukui's orbital mixing rule [45], the favorable steric arrangement for transannular cyclization is not only spatial closeness of the two π-bonds, but also their position in the same plane. Thus, UV and photo-electronic spectra data prove interaction of double bonds and splitting of π-levels for the dienes **1** [46] and **3** [47], that favors their transannular cyclization. On the contrary, the conformational mobility of the diene **8** proved by unlimited number of conformations and in particular twist-boat form for its close analogue 1,7-dibromo-*cis*,*cis*-cycloocta-1,5-diene determined by X-Ray structural analysis [48] hampers transannular cyclization of *cis*,*cis*-1,5-cyclooctadiene.

3. Conclusion

We have studied the radical transannular cyclizations abilities of non-conjugated dienes, such as 3,7-dimethylenebicyclo[3.3.1]-nonane, norbornadiene and *cis*,*cis*-1,5-cyclooctadiene with SF_5Cl. It has been shown that the use of UV-radiation for the reactions of the dienes **1** and **5** with SF_5Cl is an efficient route to increase yields of the radical cyclization products. The reaction of *cis*,*cis*-1,5-cyclooctadiene with SF_5Cl is less selective and leads mainly to the formation of the product of SF_5Cl addition to one double bond in low yields, using triethylborane (0.1 equiv.) for radical initiation was rather more effective than UV-irradiation.

4. Experimental

The ^1H (200.13 MHz), ^{13}C (50.32 MHz) and ^{19}F (188.31 MHz) NMR spectra were recorded on a Bruker DPX-200 spectrometer using $CDCl_3$ as solvent and TMS or CCl_3F as internal standards. MS (EI) and HRMS spectra were obtained on a Varian MAT CH7A instrument at 70 eV. All reagents from commercial suppliers were used without further purification. 3,7-Dimethylenebicyclo[3.3.1]-nonane (**1**) was prepared by the known procedure (Ref. [29]). All reactions were carried out under the inert atmosphere (nitrogen) and monitored by TLC and ^{19}F NMR spectroscopy.

4.1. Reactions dienes 1, 5, 8 with SF_5Cl upon UV-irradiation: general procedure

A mixture of dienes (0.012 mol) and SF_5Cl (0.018 mol, and 0.048 mol for diene **8**) in anhydrous *n*-hexane (20 ml) contained in a quartz tube were irradiated for 3 h (24 h for diene **8**) with a high-pressure Hg vapour lamp at ambient temperature. Volatiles were removed by evaporation giving a light yellow oil of mixtures of the corresponding products. The pure products were isolated by column chromatography on silica gel using *n*-hexane or pentane as an eluent.

Reaction diene **1** with SF_5Cl without initiation was carried out in a similar manner but without UV-irradiation and in a Pyrex flask.

*4.1.1. 3-Chloromethyl-7-[(pentafluoro-λ^6-sulfanyl)methyl]tricyclo[3.3.1.0^{3,7}]nonane (**2**, 73%)*

Colorless oil; ^{1}H NMR ($CDCl_3$): δ 1.62 (m, 2H, norAd), 1.67 (m, 2H, norAd), 1.84 (m, 4H, norAd), 1.97 (m, 2H, norAd), 2.29 (m, 2H, norAd), 3.55 (s, 2H, CH_2Cl), 3.94 (p, 2H, CH_2SF_5, J = 9 Hz); ^{13}C NMR ($CDCl_3$): δ 34.2 (s, C-9), 35.6 (s, C-1,5), 47.8 (s, C-2,4), 49.6 (p, J = 1.9 Hz, C-6,8), 50.1 (s, CH_2Cl), 50.2 (p, J = 1 Hz, C-3), 55.4 (p, J = 1.6 Hz, C-7), 78.9 (p, J = 9.9 Hz, CH_2SF_5); ^{19}F NMR ($CDCl_3$): δ 69.4 (dm, J = 143 Hz, B_4-part), 85.1 (9 lines, A-part); MS (EI) m/z (%): 275 $[M-Cl]^+$ (3%), 185 $[M+2-SF_5]^+$ (30%), 183 $[M-SF_5]^+$ (100%), 147 $[M-SF_5-HCl]^+$ (20%); HRMS for $[M-Cl]^+$ ($C_{11}H_{16}F_5S$): calculated 275.0893, found 275.0888; for $[M-SF_5]^+$ ($C_{11}H_{16}Cl$): calculated 183.0941, found 183.0936.

*4.1.2. 3-exo-Chloro,5-exo-(pentafluoro-λ^6-sulfanyl)tricyclo[2.2.1.0^{2,6}]heptane (**6**, 44%)*

Colorless oil; ^{1}H NMR ($CDCl_3$): δ 1.77 (m, 2H), 1.97 (m, 1H), 2.12 (d, 1H, H-7, J_{AB} = 10 Hz), 2.24 (d, 1H, H-7, J_{AB} = 10 Hz), 2.65 (s, 1H), 3.81 (p, 1H, $CHSF_5$, J = 8 Hz), 3.88 (s, CHCl); ^{13}C NMR ($CDCl_3$): δ 13.9 (s), 20.7 (p, J = 4.3 Hz), 21.2 (p, J = 1.2 Hz), 29.2 (p, J = 0.8 Hz), 42.1 (p, J = 2.8 Hz), 61.3 (p, J = 3.1 Hz, CHCl), 86.2 (pd, J = 12.4 Hz, 1.2 Hz, $CHSF_5$); ^{19}F NMR ($CDCl_3$): δ 59.1 (dm, J = 144.9 Hz, B_4-part), 83.2 (9 lines, A-part); MS (EI) m/z (%): 256 $[M+2]^+$ (13%), 254 $[M]^+$ (28%), 129 $[M+2-SF_5]^+$ (20%), 127 $[M-SF_5]^+$ (62%), 91 $[M-SF_5-HCl]^+$ (100%); Anal. Calcd. for $C_7H_8ClF_5S$: C, 33.0; H, 3.2; Cl, 13.9; S, 12.6. Found: C, 33.0; H, 3.1; Cl, 13.8; S, 12.5.

*4.1.2.1. 3-endo-Chloro,5-exo-(pentafluoro-λ^6-sulfanyl)tricyclo[2.2.1.0^{2,6}]heptane (**7**).* Colorless oil; ^{1}H NMR ($CDCl_3$): δ 1.60–1.75 (m, 3H), 1.96 (m, 1H), 2.13 (d, 1H, H-7, J_{AB} = 10 Hz), 2.56 (s, 1H), 3.99 (s, CHCl), 4.54 (p, 1H, $CHSF_5$, J = 8 Hz); ^{19}F NMR ($CDCl_3$): δ 61.8 (dm, J = 144.9 Hz, B_4-part), 84.4 (9 lines, A-part).

*4.1.3. trans-5-Chloro-6-(pentafluoro-λ^6-sulfanyl)cyclooctene (**9**)*

Colorless oil; ^{1}H NMR ($CDCl_3$): δ 1.75–2.85 (m, 8H), 4.31 (m, 1H, $CHSF_5$, J = 9 Hz), 5.12 (br.s, 1H, CHCl), 5.63 (m, 1H), 5.90 (m, 1H); ^{13}C NMR ($CDCl_3$): δ 21.9 (s), 23.1 (p, J = 1.6 Hz), 29.5 (p, J = 3.7 Hz), 36.9 (s), 61.1 (p, J = 4.7 Hz, CHCl), 89.4 (pd, J = 7.7 Hz, 1 Hz, $CHSF_5$), 128.6 (s), 132.2 (s); ^{19}F NMR ($CDCl_3$): δ 54.1 (dm, J = 143.1 Hz, B_4-part), 85.8 (9 lines, A-part); MS (EI) m/z (%): 272 $[M+2]^+$ (8%), 270 $[M]^+$ (21%), 143 $[M-SF_5]^+$ (8%), 107 $[M-SF_5-HCl]^+$ (61%), 79 $[M-SF_5-HCl-C_2H_4]^+$ (100%); Anal. Calcd. for $C_8H_{12}ClF_5S$: C, 35.5; H, 3.5; Cl, 13.1; S, 11.9. Found: C, 35.4; H, 3.5; Cl, 13.0; S, 11.8.

*4.2. Reaction diene **8** with SF_5Cl using triethylborane*

Into a three-necked flask equipped with a dry ice reflux condenser and a nitrogen inlet diene **8** (3 g, 0.0277 mol) in anhydrous *n*-hexane (40 ml) was added and cooled to −40 °C. Then SF_5Cl (18.2 g, 0.112 mol, 4 equiv.) was condensed to the solution. The solution was stirred at −40 °C and Et_3B (2.8 ml, 1 M in *n*-hexane) was added slowly using a syringe. The solution was vigorously stirred for 4 h at −30 °C, and then the mixture was warmed up to room temperature. The mixture was hydrolyzed with aqueous $NaHCO_3$ (10%) and the organic layer dried over $NaSO_4$. The solvent was removed, and the purity product **9** (0.98 g, 13% yield) was isolated by column chromatography on silica gel using pentane as an eluent.

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