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TRANSANNULAR OXYGEN PARTICIPATION IN HALOFLUORINATION REACTIONS OF 9-OXABICYCLO[6 1 0]NON-4-ENE [1]

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SUMMARY

The transannular O-heterocyclization of 9-oxabicyclo-[6 1 0]non-4-ene in halofluorination reactions using N halo-succinimides and triethylamine tris-hydrofluoride or Olah's reagent, respectively, yields endo,endo-2-halo-6-fluoro-9-oxa-bicyclo[3 3 1]nonane as the main product and endo,endo-2-halo-5-fluoro-9-oxabicyclo[4 2 1]nonane as the minor compound by halonium assisted epoxide ring participation

INTRODUCTION

Halofluorinations of unsaturated compounds allow the introduction of fluoride ion under much milder conditions than direct hydrofluorination Recently we have shown, that the combination of N-halosuccinimides and triethylamine tris-hydrofluoride ($Et_3N/3HF$) is a convenient, mild and selective reagent for halofluorination of alkenes The reactions proceed stereospecifically as anti 1,2-additions [2] However, when reacting with medium sized carbocyclic 1,5-dienes [3] or norbornadiene [4], transannular participation of the second double bond is obtained (transannular π-participation), similar to other polar electrophilic reactions in such compounds [5]

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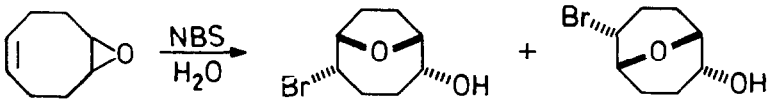
On the other hand the transannular participation of oxygen functions (transannular O-heterocyclization) was found, for example in halohydroxylations and haloalkoxylations of cycloocta-1,5-diene leading to mixtures of endo,endo-2,6-dihalo-9-oxabicyclo[3.3.1]- and endo,endo-2,5-dihalo-9-oxabicyclo-[4.2.1]nonanes [6,7] and in the bromohydroxylation of 9-oxabicyclo[6.1.0]non-4-ene, yielding a mixture of endo-5-bromo-9-oxabicyclo[4.2.1]- and endo-6-bromo-9-oxabicyclo[3.3.1]-nonan-2-endo-ol [8].



Recently formation of the above-mentioned isomeric dihalo-9-oxabicyclononanes was reported also for the reaction of 9-oxabicyclo[6.1.0]non-4-ene with bromine or iodine, respectively, in carbon tetrachloride or acetonitrile [9] and more recently the transannular participation of the epoxide ring in iodinations of 10-oxabicyclo[7.1.0]dec-4-enes and 11-oxabicyclo[8.1.0]undec-5-enes was described [10].

We wish to report herein the halofluorination of 9-oxabicyclo[6.1.0]non-4-ene using the combination of an N-halosuccinimide and triethylamine tris-hydrofluoride or Olah's reagent, respectively.

RESULTS and DISCUSSION

The reaction of 9-oxabicyclo[6.1.0]non-4-ene (1) with one equivalent of N-chloro-, N-bromo- or N-iodosuccinimide, respectively, in the presence of an excess of $Et_3N/3HF$ in methylene chloride at ambient temperature gives, after 3-5 h, in each case a mixture of two isomeric halofluoroethers in good yield. These products were separated by column chromatography and the structures were established mainly by ^1H NMR and ^{13}C NMR studies (cf. Tables 1 and 2) to be endo,endo-2-halo-6-fluoro-9-oxabicyclo[3.3.1]nonane (2) and endo,endo-2-halo-5-fluoro-9-oxabicyclo[4.2.1]nonane (3) in a ratio assigned in the scheme

TABLE 1

^1H NMR data of endo,endo-2-halo-6-fluoro-9-oxabicyclo[3.3.1]nonanes (2) and endo,endo-2-halo-5-fluoro-9-oxabicyclo[4.2.1]nonanes (3)

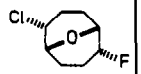
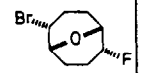
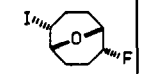
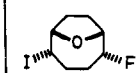
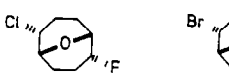
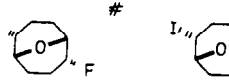
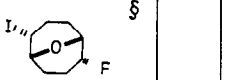
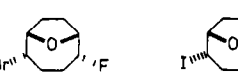
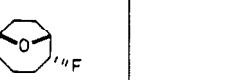
δ [ppm] / J [Hz] / compound	H_6	$^2J_{HF}$	$^3J_{H_{6a}H_{7a}}$	$^3J_{H_{6a}H_{7e}}$	$^3J_{H_{6a}H_{5e}}$	H_2	$^3J_{H_{2a}H_{3a}}$	$^3J_{H_{2a}H_{3e}}$	$^3J_{H_{2a}H_{1e}}$	H_5 (J)	H_1 (J)	other H
	4.84	48.5	10.4	7.0	5.2	4.27	10.5	7.8	5.2	3.99 (5.8)	3.87 (5.5)	2.37 (1H) 2.20-1.85
	4.86	47.8	10.4	6.0	5.0	4.42	12.6	7.1	5.4	4.04 -	3.92 (5.5)	2.51 (1H) 2.40-1.95
	4.87	50.4	10.1	6.1	5.8	4.59	13.1	5.5	5.2	4.10 (5.9)	3.94 (5.4)	2.65 (1H) 2.60-1.80
	H_5	$^2J_{HF}$	$^3J_{H_{5a}H_{4a}}$	$^3J_{H_{5a}H_{4e}}$	$^3J_{H_{5a}H_{6e}}$	H_2	$^3J_{H_{2a}H_{3a}}$	$^3J_{H_{2a}H_{3e}}$	$^3J_{H_{2a}H_{1e}}$	H_6	H_1	other H
	4.91	49.2	7.6	3.6	3.6	4.04	9.1	4.5	4.5	4.58 ($W_{1/2}$=33 Hz)		2.40-1.80
	4.93	49.2	7.5	3.8	3.8	4.13	10.5	4.4	4.4	4.62 ($W_{1/2}$=24 Hz)		2.45-1.80
	4.96	49.8	7.4	3.6	3.6	4.24	11.2	5.7	4.5	4.71 ($W_{1/2}$=18 Hz)		2.40-1.80

TABLE 2

^{19}F NMR and ^{13}C NMR data of endo,endo-2-halo-6-fluoro-9-oxabicyclo[3 3 1]nonanes (2) and endo,endo-2-halo-5-fluoro-9-oxabicyclo[4 2 1]nonanes (3)

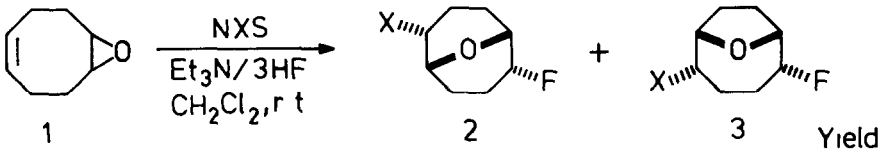
Compound			 #	 §				
^{19}F (δ, J)		183 0 52	182 9 52	184 6 51		184 1 52	184 5 52	186 8 52
^{13}C (δ, J)	C_6	88 5 177 9	87 3 178 8	88 5 179 0	C_5	91 6 176 0	90 5 176 6	91 8 176 6
	C_1	69 6 -	68 5 1 1	70 5 1 1	C_1	82 6 -	81 8 -	84 5 -
	C_5	67 0 25 0	65 8 24 4	67 0 24 3	C_6	78 2 26 0	77 3 25 7	78 9 25 5
	C_2	57 6 -	49 3 -	29 7* -	C_2	60 3 -	52 2 -	34 2* -
	C_3	29 7 -	29 8 -	32 8 -	C_4	28 7 23 1	29 2 22 2	32 7 21 6
	C_7	26 0 20 1	24 9 19 5	26 0 16 8	C_8	28 1 -	27 7 1 9	30 6 1 0
	C_4	23 7 -	23 8 -	26 2 -	C_3	25 3 8 6	24 7 8 6	26 4 9 4
	C_8	22 0 9 0	21 8 8 7	24 3 8 6	C_7	24 8 -	24 2 -	26 3 -

solid mp 60 5 - 61°C

§ solid mp 51 - 52 C

* assigned by a DEPT experiment

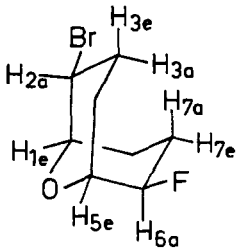
(determined by ^{19}F NMR spectroscopy) The product ratio changes little with the reaction conditions e g , for X=Br after 24 h the ratio has changed only to 78 22



a	X = Cl	71	29	92%
b	X = Br	75	25	95%
c	X = I	74	26	85%

Two examples of structural elucidation follow Endo,endo-2-bromo-6-fluoro-9-oxabicyclo[3 3 1]nonane (2b) was separated from its isomer (3b) by column chromatography Sublimation gave an analytical pure sample (m p 60-60 5 °C) In its mass spectrum compound (2b) gives a molecular ion at m/z 222 From this ion H_2O, F or HF are eliminated in small amounts The main fragmentation process is loss of a bromo-radical (m/z 143) followed by HF elimination (m/z 123) and dehydration (m/z 105) In its ^1H NMR spectrum the characteristic doublet of a quintet belonging to proton H_6 in the neighbourhood of a fluorine substituent appears at δ = 4 86 ppm with J_{HF} = 47 8 Hz The trans-stereochemistry between fluorine and the ether bridge follows from an aa coupling between H_6 and H_{7a} (J = 10 4 Hz) and two ae couplings between H_6 and H_{7e} (J = 6 Hz) or H_6 and H_5 (J = 5 Hz), respectively Analogously the quintet of H_2 in the neighbourhood of the bromine substituent at δ = 4 42 ppm shows an aa coupling to H_{3a} (J = 12 6 Hz) and two ae couplings to H_{3e} and H_{1e} (J = 7 1 Hz and 5 4 Hz, respectively) This is consistent with the equatorial conformation of bromine and therefore a trans-stereochemistry related to the ether bridge The signals of the two equatorial protons adjacent to the ether bridge appear at δ = 4 04 ppm (H_{5e}) and δ = 3 92 ppm (H_{1e}), respectively, as small multiplets (each $W_{1/2}$ = 15 Hz) Double irradiation experiments were used to assign signals The vicinal coupling constants between F and H_{5e} should be near zero, as in 3-deoxy-3-fluoro-D-glucopyrano-

sides bearing an equatorial fluorine atom [11] Other coupling constants J_{H5eH4a} and J_{H5eH6a} have been found to be 5 Hz and J_{H5eH4e} is about 0 5 Hz The same signal shape is found for H_{1e}

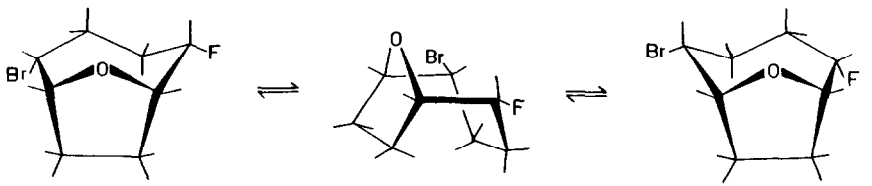


2b

This structure is supported by the ^{19}F and the ^{13}C NMR data (cf Table 2) The most downfield-shifted signal in the ^{1}H-decoupled ^{13}C NMR spectrum, δ = 87 3 ppm ($^{1}J_{CF}$ = 178 8 Hz), belongs to C_6 The neighbours of the CHF group appear at δ = 65 8 ppm ($^{2}J_{CF}$ = 24 4 Hz) (C_5) and δ = 24 9 ppm ($^{2}J_{CF}$ = 19 5 Hz) (C_7) The signal of carbon 1 in the δ-position to the fluorine substituent at δ = 68 5 ppm is a doublet ($^{4}J_{CF}$ = 1 1 Hz) This coupling over four bonds is only possible when the two atoms are in a W conformation This is realized only in the case when fluorine has an equatorial conformation The singlet of C_2 is found at δ = 49 3 ppm Carbon 8, located in the γ-position to the fluoro-substituent, appears at δ = 21 8 ppm ($^{3}J_{CF}$ = 8 7 Hz) The remaining singlets at δ = 29 8 and δ = 23 8 ppm are assigned to C_3 and C_4, respectively

The other isomer, endo,endo-2-bromo-5-fluoro-9-oxabicyclo-[4 2 1]nonane (3b), was separated as an 85% pure oily liquid The mass spectrum of this sample is very similar to that of the isomer (2b), only the intensities of the peaks are different In its ^{1}H NMR spectrum the doublet for H_5 appears at δ = 4 93 ppm (J_{HF} = 49 2 Hz) The trans-stereochemistry of the fluorine substituent in relation to the ether bridge is established from the large coupling constant between H_5 and H_{4a} (J = 7 5 Hz) and two smaller couplings between H_5 and H_{4e} or H_{6e}, respectively (both J = 3 8 Hz) By way of analogy the quintet of H_2, adjacent to bromine, at δ = 4 13 ppm couples with H_{3a} (J = 10 5 Hz), with H_{3e} and H_{1e} (both J = 4 4 Hz)

All coupling constants are smaller than the related ones in (2b), depending on 1) the conformation of the oxacycloheptane ring segment itself, and 11) from the fact that there exists an equilibrium of two alternative chair-like forms A and C (as shown in the scheme), which equilibrate fast at room temperature, perhaps passing an intermediary conformer B (cf [12]) Consequently, all coupling constants represent mixed values from conformer A (equatorial fluorine and quasi-axial bromine) and C (equatorial bromine and quasi-axial fluorine)

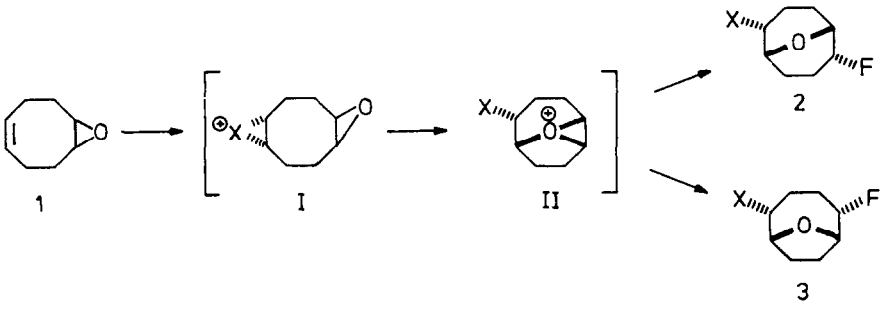


A B C

Moreover, in compounds (3) the coupling constants J_{H5H4} are generally much smaller than the related J_{H2H3} (cf Table 1) This seems to be not only caused by the stronger electronegativity of the fluorine substituent, compared to the other halogens,but is perhaps caused as well by the relative amount of A and C in the equilibrium Thus, conformer C with an aa coupling between H_2 and H_{3a} should be favoured over A

The ^{13}C chemical shifts and CF coupling constants (cf Table 2), however, are in good agreement with the proposed structure

Products (2) and (3) should be formed by the following reaction mechanism

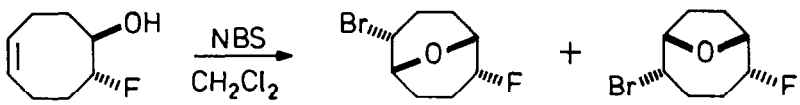


1 I II 2 3

First, the electrophile attacks the double bond of (1) producing a more or less bridged [13] halonium ion I (cf. also [7]). By transannular participation of the epoxide oxygen an oxonium ion II is formed, which is opened by the fluoride anion under formation of mainly 9-oxabicyclo[3.3.1]nonane compounds (2) and a lesser amount of isomers (3).

In contrast to our former findings in halohydroxylation or haloalkoxylation of cycloocta-1,5-diene [7] the product ratio in these reactions is not depending from the employed electrophile. The ratio should be determined mostly by the difference in the thermodynamic stability of the bicyclic systems and perhaps by differences in steric hindrance to fluoride attack (cf. [10]).

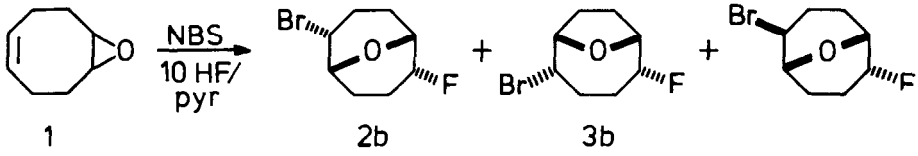
A mixture of products (2b) and (3b) can be obtained as well by treatment of trans-2-fluorocyclooct-5-en-1-ol (4) with NBS in methylene chloride. The products are formed here in a 93:7 ratio (^{19}F NMR) in quantitative yield.



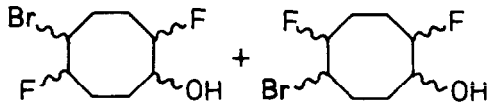
4 2b 3b

The fluoro alcohol (4) was synthesized from (1) by ring opening of the epoxide using $Et_3N/3HF$ at 60 °C for 24 h [14]. At room temperature (1) does not react with this reagent while, with the more acidic Olah's reagent at 25 °C for 90 min, ring opening and transannular π-participation produces two epimeric 6-fluoro-cis-bicyclo[3.3.0]octan-2-ols [14].

However, reaction of (1) with NBS and Olah's reagent in methylene chloride for 3 h at room temperature is less selective and yields five products. The main compounds are again (2b) and (3b), which are formed in a 7:3 ratio (43% and 18%, integrals in ^{19}F NMR). The third product (5) (8% of the mixture) was isolated in 88% purity by column chromatography, while (6) and (7) (ratio 55:45; 31% of the mixture) were obtained as a mixture.



1 2b 3b



6 7

From spectroscopic data it can be concluded that (5) is an epimer of (2b) We assign the structure of exo-2-bromo-endo-6-fluoro-9-oxabicyclo[3 3 1]nonane (5) from the following facts The mass spectrum of the product differs only in peak intensities from that of epimer (2b) In its ^{19}F NMR spectrum a signal appears at δ = 183 3 ppm (J_{HF} = 49 4 Hz) The doublet of a multiplet for H_6 appears in the 200 MHz ^1H NMR spectrum at δ = 4 87 ppm (J_{HF} = 49 2 Hz) The coupling pattern is similar to that of (2b) (J_{H6aH7a} = 10 6 Hz, $J_{H6aH7e} \sim J_{H6aH5e} \sim$ 6 Hz) The signal of H_2 is found at δ = 4 33 ppm as a very poorly resolved small multiplet ($W_{1/2}$ = 12 Hz) which should, therefore, be an equatorial one The multiplets for H_1 and H_5 are not separated and appear between δ = 4 22 and 4 05 ppm The methylene protons are located between δ = 2 4 and 1 7 ppm

In the ^1H decoupled ^{13}C NMR spectrum carbon 6 is found at δ = 88 0 ppm ($^1J_{CF}$ = 179 2 Hz), C_5 at δ = 67 6 ppm ($^2J_{CF}$ = 24 3 Hz), and C_2 at δ = 52 2 ppm For C_1 a doublet is found at δ = 71 6 ppm ($^4J_{CF}$ = 1 1 Hz), which is consistent with an equatorial fluorine and a W-coupling over four bonds (cf compound (2b)) The chemical shifts and CF coupling constants of the other carbon atoms (δ = 28 6, C_3, 26 9, $^3J_{CF}$ = 8 5 Hz, C_8, 25 0, $^2J_{CF}$ = 19 7 Hz, C_7, 18 2 ppm, C_4) are in good agreement with the proposed structure

However, a concise explanation for the mechanism of formation of (5) cannot be given

Compounds (6) and (7) could not be isolated pure They are formed by simple bromofluorination of the double bond and subsequent opening of the epoxide ring by the fluorinating agent The structure of isomeric bromo-difluoro-cyclooctanols was deduced from the spectroscopic data In the mass spectrum the molecular ion does not appear At m/z 241 the M^+-1 peak appears with 10^{-2}% relative intensity Furthermore, fragment ion peaks are found at m/z 224 (M^+-H_2O, 0 2%), 222 (M^+-HF, 0 1%), 204 (M^+-H_2O-HF, 0 1%), 196 (M^+-C_2H_3F, 0 2%), 178 (M^+ -C_2H_3F-H_2O, 0 6%) and 143 (M^+-Br , 17%) The base peak is m/z 41 In its IR spectrum (CCl_4) strong absorptions are found at 3592 cm^{-1} (intramolecular hydrogen bridge), 3430 cm^{-1} (br, intermolecular associated OH), 1096, 1078, 1058 and 1025 cm^{-1} (C-O) In the ^1H decoupled ^{19}F-NMR spectrum singlets with the same integration appear at δ = 159 1 and 171 6 ppm or 159 8 and 173 7 ppm, respectively In the ^1H NMR spectrum a broad multiplet between δ = 5 2 and 4 5 ppm integrates for four protons A broad singlet appears at δ = 3 1 ppm for one OH-proton and the methylene protons are located between δ = 2 5 and 1 5 ppm

EXPERIMENTAL

Mass spectra were recorded on a VG 12-250 apparatus using 70 eV ionization energy IR spectra were measured in solution (CCl_4) on a Perkin Elmer 297 spectrometer ^1H and ^{13}C NMR spectra were obtained in $CDCl_3$ on a Bruker AC 200 (200 13 MHz or 50 32 MHz, resp) on a Bruker AM 300 (300 133 MHz or 75 469 MHz, resp) and some 350 MHz ^1H NMR spectra on a Cameca 350 Signals are given in δ values (ppm) relative to TMS as internal standard ^{19}F NMR spectra were recorded on a Bruker WP 80 (75 38 MHz) spectrometer in $CDCl_3$ Signals are described in φ values (ppm) relative to $CFCl_3$ as internal standard

General Procedure for Halofluorinations

A stirred mixture of 9-oxabicyclo[6 1 0]non-4-ene (1) (0 62 g, 5 mmol) (or 0 72 g of the alcohol 4), $Et_3N/3HF$ (5 ml, 25 mmol) (or 5 ml Olah's reagent) and methylene chloride (10 ml) (or 10 ml dry ether) is treated with the respective N-halosuccinimide (5 5 mmol) at 0°C After 15 min the ice bath is removed and the mixture is stirred for an additional 3 h at ambient temperature Then, the mixture is poured into ice water (50 ml), neutralized with aqueous 28% ammonia and extracted three times with methylene chloride or ether (25 ml) The combined extracts are washed with 0 1 N hydrochloric acid and with 5% aqueous sodium hydrogen carbonate, and dried with sodium sulfate After evaporation of the solvent the product ratio is determined by ^{19}F NMR spectroscopy and the mixture is separated by column chromatography (20 g Kieselgel 60, Merck, 20 cm column, light petroleum ether/diethylether 50 1)

Reaction of 9-oxabicyclo[6 1 0]non-4-ene with NBS and water

A stirred solution of 9-oxabicyclo[6 1 0]non-4-ene (1) (6 2 g, 50 mmol) in dioxan (100 ml), water (30 ml) and catalytic amounts of conc sulfuric acid is treated with NBS (8 9 g, 50 mmol) within 30 min at 10°C The mixture is stirred for an additional 3 h at ambient temperature Usual work up yields a 63 37 mixture of endo-6-bromo-9-oxabicyclo[3 3 1]-nonan-2-endo-ol and endo-5-bromo-9-oxabicyclo[4 2 1]nonan-2-endo-ol in 62% combined yield, b p 120°C at 0 9 mm Hg, n_D^{20} 1 5450, m p 29-30°C Analysis Found C, 43 54, H, 5 84, Br, 36 37% $C_8H_{13}BrO_2$ requires C, 43 46, H, 5 93, Br, 36 14% m/z 220 (M^+, 5%), 141 (M-Br), 140 (M-HBr), 123 (M-Br $-H_2O$) λ_{max} (film) ν (OH) 3624, ν(C-OH) 1083, ν (C-O-C) 1052 cm^{-1}

Endo-6-bromo-9-oxabicyclo[3 3 1]nonan-2-endo-ol

δ_H 4 44 (1H, J_{H6aH7a} 12 6, J_{H6aH7e} 6 1, J_{H6aH5e} 5 7 Hz), 4 06 (1H, J_{H2aH3a} 11 4, J_{H2aH3e} 5 7, J_{H2aH1e} 5 7 Hz), 3 91 (t, 1H, J 5 7 Hz), 3 85 (t, 1H, J 5 8 Hz), 2 5-1 7 (m, 9H, $-CH_2-$ and -OH)

δ_C 69 6 (2C), 68 2 (C-1,C-2,C-5), 50 8 (C-6), 31 2 (C-7), 28 3 (C-3), 24 5 (C-8), 23 2 (C-4)

Endo-5-bromo-9-oxabicyclo[4 2 1]nonan-2-endo-ol

δ_H 4 60-4 50 (m, 2H, >CHBr and >CHOH), 4 25-4 15 (m, 2H, 2x >CH-O-), 2 5-1 7 (m, 9H, $-CH_2-$ and -OH)

δ_C 81 9, 80 9 (C-1,C-6), 70 8 (C-2), 54 1 (C-5), 31 8 (C-4), 29 6 (C-3), 26 6 (C-7), 24 7 (C-8)

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