An unusual way of the reaction of PI₃ with norbornene and norbornadiene

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The iodination of norbornene and norbornadiene with phosphorus triiodide in the presence and in the absence of weak electrophiles (arylsulfenamides or N-chloramines) and with elemental iodine was studied. Depending on the reaction conditions, the reactions resulted in mono- or diiodides of the norbornene and norbornadiene series. All the compounds obtained were characterized by ¹H and ¹³C NMR spectra.

Key words: iodination, phosphorus triiodide, norbornene, norbornadiene.

Unlike the methods of chlorination and bromination of organic compounds that are well developed, the development of preparative methods for iodination always presented a problem for organic chemists since the iodide anion can be readily eliminated and replaced with other nucleophiles. At the same time, it is precisely these properties of alkyl iodides that make them very important compounds for the consequent replacement of the iodine atom with various groups.

Recently we have studied the activation of weak electrophilic reagents with phosphorus halides PHal₃ (Hal = Cl, Br, I) and found that phosphorus trichloride and tribromide form electrophilic complexes with arylsulfenamides,[1] dithiobisamines,[2] and N-chloramines that are capable of the addition at multiple carbon bonds to give β-thio- or β-chlorosubstituted alkyl chlorides or bromides. It seemed promising to use this approach for phosphorus triiodide, the electrophilic activation of which might result in the preparation of the corresponding substituted alkyl iodides. However, the reactions of PI₃ with olefins of the bicyclo[2.2.1]heptane series in the presence of arylsulfenamides and n-chloramines gave unexpected results.

The structure of the products of the reactions under study was established by ¹H and ¹³C NMR spectroscopy. The analysis of the spectra followed by the structural assignment were carried out on the basis of the data on the effect of substituents on the chemical shifts, ¹H—¹H and ¹H—¹³C coupling constants, as well as using homo- and heteronuclear selective double resonance, difference spectroscopy of the nuclear Overhauser effect (NOE),[3] and two-dimensional ¹H—¹H and ¹H—¹³C correlation spectroscopy (COSY).[4] ¹H chemical shifts are given in Table 1, and the ¹³C chemical shifts of all the compounds obtained are given in Tables 2 and 3.

We were able to isolate the expected endo-2-iodo-exo-3-arylthiobicyclo[2.2.1]heptane (1) only in the reactions of ortho- and para-nitrophenylsulfenamides with norbornene in the presence of phosphorus triiodide, while its yield did not exceed 38 %. In addition, the

reaction produces 2-exo-iodonorbornane (2), which becomes the only reaction product when arylsulfenamine is changed to N-chloramine.

The data obtained were compared with the results of iodination of norbornene with phosphorus triiodide in the absence of weak electrophilic additives, as well of its direct iodination with elemental iodine in organic solvent:

1a: Ar = 4-NO₂C₆H₄, 38%
1b: Ar = 2-NO₂C₆H₄, 35%
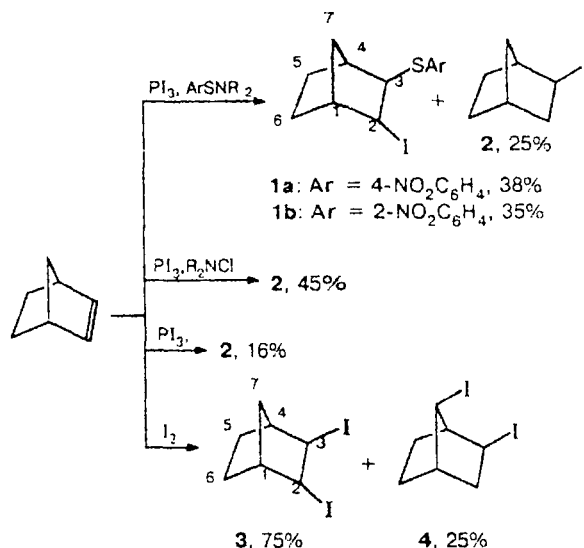
We found that monoiodide 2 is formed in the reaction of norbornene with PI₃ even in the absence of weak electrophilic additives. However, its yield is significantly lower in this case, and the reaction is completed in ~24 h at 20 °C, whereas it is completed in several minutes at room temperature in the presence of ArSNR₂ or R₂NCl. Principally, the formation of the monoiodination product could be attributed to secondary reactions during the purification of the reaction mixture on silica gel; however, the presence of signals of iodide 2 in the

Table 1. Chemical shifts of ^{1}H nuclei and ^{1}H—^{1}H coupling constants (Hz) for compounds 1—11

Compound	H(1)	H(2)	H(3)	H(4)	H(5)	H(6)	H(7)
1a[a,b]	2.50 ($J_{1,2}= 4.0$)	4.03 ($J_{1,2}= 4.0$, $J_{2,3}= 4.9$)	3.38 ($J_{3,7a}= 2.0$, $J_{2,3}= 4.9$)	2.23	1.62 (endo) 1.72 (exo)	1.72 (endo) 1.89 (exo)	1.38 (anti) ($J_{2,7a}= 2.0$, $J_{7,7}= 10.5$) 1.80 (syn)
1b[c,d]	2.47 ($J_{1,2}= 4.7$)	4.02 ($J_{1,2}= 4.7$, $J_{2,3}= 4.8$)	3.21 ($J_{3,7a}= 2.4$, $J_{2,3}= 4.8$)	2.19	1.43 (endo) 1.68 (exo)	1.68 (endo) 1.90 (exo)	1.32 (anti) ($J_{2,7a}= 2.4$, $J_{7,7}= 10.6$) 1.68 (syn)
2[a]	2.58	4.00 ($J_{2,7a}= 1.8$, $J_{2,3endo}= 3.5$, $J_{2,3exo}= 7.9$)	2.10 (endo) ($J_{2,3}= 7.9$, $J_{3,3}= 15.5$) 2.23 (exo) ($J_{2,3}= 3.5$, $J_{3,3}= 15.5$)	2.24	1.10 (endo) 1.52 (exo)	1.24 (endo) 1.54 (exo)	1.34 (anti) ($J_{2,7a}= 1.8$, $J_{7,7}= 10.5$) 1.89 (syn) ($J_{7,7}= 10.5$)
3[c]	2.38 ($J_{1,2}= 1.6$)	4.52 (exo-HCl) ($J_{1,2}= 1.6$, $J_{2,3}= 3.9$)	3.93 (endo-HCl) ($J_{3,7a}= 2.8$, $J_{2,3}= 3.9$)	2.49	1.33—1.40 (endo) 1.61—1.68 (exo)	1.33—1.40 (endo) 1.61—1.68 (exo)	1.48 (anti) 2.09 (syn)
4[c]	2.69 ($J_{1,6}= 4.2$)	3.84 ($J_{2,7}= 1.2$, $J_{2,3}= 8.3$)	2.16 (endo) ($J_{3,7}= 1.2$, $J_{2,3}= 8.3$, $J_{3,3}= 13.3$) 2.65 (exo)	2.35 ($J_{3,4}= J_{4,5}= 4.2$)	1.29—1.42 (endo) 1.66—1.73 (exo)	1.29—1.42 (endo) 1.66—1.73 (exo)	3.87 ($J_{2,7}= J_{3,7}= 1.2$)
5[a]	3.18	3.61 ($J_{2,7a}= 1.9$, $J_{2,3endo}= 2.7$, $J_{2,3exo}= 7.6$)	1.85 (endo) ($J_{2,3}= 7.6$, $J_{3,3}= 13.1$) 2.17 (exo) ($J_{2,3}= 2.7$, $J_{3,3}= 13.1$)	2.88	6.18 ($J_{1,5}= 2.8$, $J_{5,6}= 5.7$)	5.98 ($J_{4,6}= 2.8$, $J_{5,6}= 5.7$)	1.65 (anti) ($J_{2,7a}= 1.9$, $J_{7,7}= 8.8$) 1.96 (syn) ($J_{7,7}= 8.8$)
6[a]	2.12	3.82	1.55 ($J_{3,5}= 6.0$)	1.32 ($J_{4,5}= 5.9$, $J_{3,4}= 6.0$)	1.08 ($J_{4,5}= 5.9$, $J_{3,5}= 6.0$)	1.26 (exo) ($J_{6,6}= 10.5$) 1.41 (endo) ($J_{6,6}= 10.5$)	1.33 (anti) ($J_{7,7}= 9.4$) 1.93 (syn) ($J_{7,7}= 9.4$)
7[e]	2.05 ($J_{1,6}= 5.3$)	3.10 ($J_{2,3exo}= 3.5$, $J_{2,3endo}= 5.3$)	1.66 (exo) ($J_{2,3}= 3.5$, $J_{3,4}= 5.3$, $J_{3,3}= 10.5$) 1.23 (endo) ($J_{3,3}= 15.2$)	2.05 ($J_{3,4}= 5.3$)	3.10 ($J_{5,6exo}= 3.5$, $J_{5,6endo}= 5.3$)	1.66 (exo) ($J_{5,6}= 3.5$, $J_{1,6}= 5.3$, $J_{6,6}= 10.5$) 1.23 (endo) ($J_{5,6}= 8.0$, $J_{6,6}= 15.2$)	1.82
8[a]	2.30 ($J_{1,7}= 1.5$)	3.73 (exo-HCl)	1.58 ($J_{3,4}= J_{3,5}= 5.2$)	1.35 ($J_{3,4}= J_{4,5}= 5.2$)	1.83 ($J_{3,5}= J_{4,5}= 5.2$)	4.45 (endo-HCl)	1.55 ($J_{1,7}= 1.5$, $J_{7,7}= 11.2$) 2.03 ($J_{4,7}= 1.5$, $J_{7,7}= 11.2$)
9[a]	2.35	3.87 ($J_{2,3}= 1.5$)	1.58 ($J_{2,3}= 1.5$, $J_{3,4}= J_{3,5}= 5.4$)	1.62 ($J_{3,4}= J_{4,5}= 5.4$)	1.58 ($J_{5,6}= 1.5$, $J_{3,5}= J_{4,5}= 5.4$)	3.87	2.18
10[c]	3.17	3.51 ($J_{2,3}= 3.3$)	4.31 ($J_{2,3}= J_{3,4}= 3.3$)	3.12	6.14 ($J = 3.8$, $J_{5,6}= 5.2$)	6.25 ($J = 3.8$, $J_{5,6}= 5.2$)	1.77 (anti) ($J_{7,7}= 9.5$) 2.11 (syn) ($J_{7,7}= 9.5$)
11[c]	3.30	4.09 ($J_{2,7}= 2.2$)	4.09 ($J_{3,7}= 2.2$)	3.30	6.16 ($J = 2.2$)		1.55 (anti) ($J_{7,7}= 9.5$) 2.41 (syn) ($J_{7,7}= 9.5$)

[a] CD$_2$Cl$_2$. [b] H—Ar: 7.39 ($J = 8.9$, 2 H), 8.13 ($J = 8.9$, 2 H). [c] CDCl$_3$. [d] H—Ar: 7.35 ($J = 7.8$), 7.39, 7.75 (2 H). [e] C$_6$D$_6$.

Table 2. [13]C chemical shifts (ppm) for compounds 1—11

Com-pound	C(1)	C(2)	C(3)	C(4)	C(5)	C(6)	C(7)
1a[a]	45.72	35.00	60.13	43.88	34.44	28.91	28.06
1b[b]	45.23	34.39	59.30	43.11	33.60	28.71	28.10
2[a]	48.63	30.68	45.63	38.65	29.03	28.77	36.61
3[b]	46.71	42.00	38.54	48.58	27.63	28.99	34.53
4[b]	49.32	28.45	40.07	44.82	23.67	18.62	25.70
5[a]	53.43	24.96	39.62	43.51	138.53	133.80	46.61
6[a]	38.33	35.01	19.79	13.39	13.39	31.02	32.58
7[a]	49.41	26.50	43.77	49.41	26.50	43.77	34.41
8[a]	45.77	32.05	23.09	15.05	21.18	28.70	31.49
9[a]	45.37	25.81	22.81	14.05	22.81	25.81	31.84
10[b]	52.97	32.99	37.66	51.46	138.02	135.46	44.83
11[b]	54.82	33.89	33.89	54.82	136.50	136.50	44.31

[a]CD_2Cl_2. [b]$CDCl_3$.

Table 3. [13]C chemical shifts for the aro-matic cycle atoms of compounds 1a,b

Com-pound	C$_a$	C$_b$	C$_c$	C$_d$	C$_e$	C$_f$
1a	146.82	128.03	124.32	145.84	124.32	128.03
1b	146.64	126.15	133.20	128.40	124.81	136.60

^{1}H NMR spectrum of the reaction mixture before the separation on SiO_2 makes this assumption unlikely.

One can expect the formation of elementary iodine in the disproportionation of PI_3

$$2 PI_3 \rightleftharpoons P_2I_4 + I_2$$
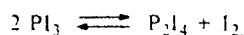
but the iodination with elemental iodine results in the expected 2,3- (3) and 2,7-diiodides (4); this permits us to reject the hypothesis about participation of elemental iodine in the iodination with phosphorus triiodide.

Likewise, the hypothesis that the product of the addition of phosphorus triiodide to the double carbon-carbon bond (similar to that in the reaction with phosphorus tribromide: PBr_3 + >C=C< → Br—C—C—PBr_2, see Refs. 5, 6) is the intermediate in this reaction cannot explain satisfactorily the results of the iodination with PI_3, since the C—P bond is very strong and there are no conditions for its hydrolysis in the course of the process.

The spatial location of substituents in adducts 1a and 1b is unambiguously determined by the coupling con-stants of the α-protons of HCS groups and HCI groups, which have characteristic values in the rigid norbornane skeleton.[7] For example, the $J_{2,3}$ = 4.9 Hz value for 1a corresponds to the *trans*-configuration of these protons, which appear in the spectrum at 3.38 and 4.03 ppm, respectively. The relatively large vicinal constant $J_{1,2}$ (4.0 Hz) of the proton of the HCI group indicates its *exo*-orientation. Similar interaction with the other pro-ton located at the head of the bridge (H-4) is not observed for the *endo*-protons of the HCS group, but a

typical W-coupling constant appears for the *anti*-H(7) and H—CS protons (2.0 and 2.4 Hz). Similar interac-tions are also typical of 1b (see Table 1).

The *exo* configuration of the iodine atom in the product 2 was determined by the [1]H—^{1}H COSY method.

trans-Diiodonorbornane (3) is the main product of the iodination of norbornene with elemental iodine. Its stabil-ity, which is unusual for an 1,2-cycloalkane diiodide, may result from the fact that its two iodine atoms cannot form the antiperiplanar conformation that substantially im-pedes the elimination. The coupling constants of the α-protons (see Table 1) also served as key parameters for the determination of the stereochemistry of product 3.

In addition to diiodide 3, a certain amount of the product of the Wagner—Meerwein rearrangement (4) with a *syn*-configuration of the iodine substituents is formed in the reaction of norbornene with I_2. The structure of the product 4 is confirmed by the coupling constant of the *anti*-H(7) and *endo*-H(3) protons, which equals 1.2 Hz.

However, the reactions of norbornadiene with phos-phorus triiodide under conditions similar to those for norbornene give a set of products of other type. The formation of iodosulfenylation products is not observed at all even when the reaction is carried out in the presence of arylsulfenamides; insead, the reaction af-fords a mixture of hydroiodination products (5—7) in a high yield:
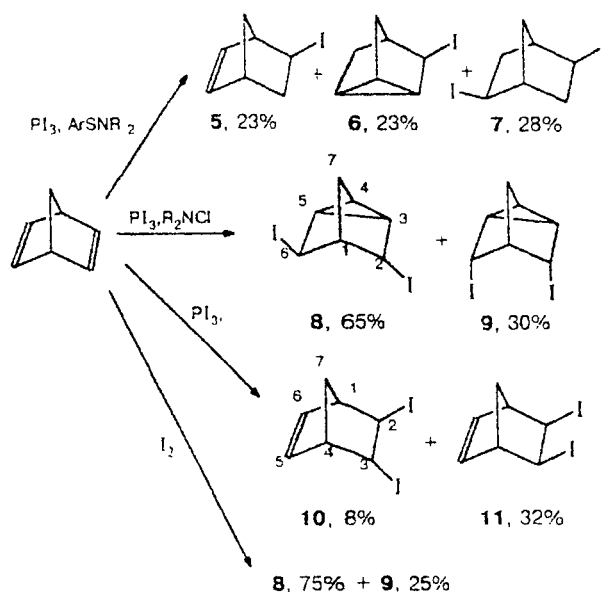
The addition of *N,N*-diethylchloramine to the reac-tion mixture results in products of other structure, namely, diiodides of nortricyclane structure 8 and 9, similar to the products of the addition of elemental iodine. The unusual formation of product 9 of di-*endo*-configuration of the substituents and the absence of di-*exo*-iodide are noteworthy. These can be explained by

the *endo*-attack of the electrophile, which is not typical of such systems.[8] As a rule, this process results in products of the attack of a nucleophile at the position adjacent to the electrophile (of 10 type) rather than nortricyclenes, since the nucleophilic attack at the p-electron pair of the double bond on the front side of the iodonium cation is extremely unfavorable.[8] When the iodination is carried out with PI₃ in the absence of other electrophiles, diiodides 10 and 11, the products of the addition at only one of the two multiple bonds of norbornadiene, are formed. In addition, the ¹H NMR spectrum of the reaction mixture indicates the presence of at least three iodides of nortricyclane structure, which were impossible to isolate in individual forms because of their low total yield (not higher than 5 %).

The set and the structures of the iodination products, as well as the reaction conditions (low temperature, polar solvent), allow us to assume the electrophilic nature of the addition in all of the cases except PI₃. However, the pathways for the formation of compounds 2 and 7 in the absence of reactive hydrogen remain unclear.

Monoiodides 5 and 6 were isolated by chromatography and characterized as a mixture. The ¹H—¹H COSY spectrum of 5 shows strong H(5)—H(6), H(2)—H(3), and H(3)—H(4) interactions and slightly less manifested H(5)—H(4), H(6)—H(1), and H(2)—*anti*-H(7) interactions. At the same time, the absence of the H(1)—H(2) interaction allows one to reject the possible structure with the proton at the substituent in the *exo*-position. Structure 5 is additionally confirmed by the NOE experiment. Irradiation of the H—CI proton (3.61 ppm) results in an increase of the integral intensity of the H(1) (3.18 ppm), *endo*-H(3) (1.85 ppm), and H(6) (5.98 ppm) signals and does not change the multiplet signal assigned to the *syn*-H(7) proton (1.96 ppm).

The iodide 6 of nortricyclane structure displays ¹H and ¹³C NMR spectral parameters typical of similar structures.[9,10] The *exo*-configuration of the substituent relative to the C(7) atom is proved by NOE: a noticeable effect was observed for the H(6) proton (1.41 ppm) and was absent for the H(7) protons (1.33 and 1.93 ppm) when the H—CI proton (3.82 ppm) was irradiated.

The NMR spectra of di-*exo*-iodide 7 consist of few lines (four signals in the ¹³C NMR spectrum and six signals in the ¹H NMR spectrum), which confirms the high symmetry of the system. The *endo*-configuration was assigned to the HCI protons (3.10 ppm), since no vicinal interactions between them and the H(1) and H(4) protons (2.05 ppm) located at the head of the bridge were observed.

Diiodonortricyclanes 8 and 9 were isolated and characterized as mixtures of isomers. The chemical shifts at the substituents corresponded to the correlations given in Ref. 9. The signals in the ¹H NMR spectrum of 8 were assigned on the basis of the NOE experiments: the perturbation of the *exo*-H—CI proton (3.73 ppm) resulted in an increase in the integral intensity of the *syn*-

oriented (with respect to the above proton) H(7) proton (1.55 ppm). However, in the case of the irradiation of the *endo*-H—CI atom (4.45 ppm), the signal of the H(7) proton remains unchanged, while an increase in the integral intensities of the signals of the H(1) and H(5) protons is observed.

Di-*endo*-iodonortricyclane 9, which is stereoisomeric to compound 8, is characterized by isochronic signals of the H—CI protons (H(2) and H(6)) and the corresponding carbon atoms (C(2) and C(6)) due to its symmetry. It is noteworthy that due to the steric effects of the two iodine atoms, the two carbon α-atoms (25.81 ppm) in compound 9 are by 3—5 ppm more shielded than in isomer 8 (28.70 and 32.05 ppm). The *exo*-configuration of protons at the substituents was determined on the basis of the ¹H—¹H COSY of a mixture of 8 and 9: the cross-peaks of the H—CI protons of nortricyclane 9 are similar to those of the *exo*-H—CI protons of compound 8.

Diiodides 10 and 11, which are the main products of the reaction of norbornadiene with PI₃ in the presence of *N*-chloramine, were isolated in individual forms after two-stage chromatography of the reaction mixture.

The *trans*-configuration of the substituents in diiodide 10 follows from the lower value of the $J_{2,3}$ coupling constant (3 Hz) and from the presence of two non-equivalent signals of olefin protons. The *cis*-di-*exo*-configuration of diiodide 11 is proved by the presence of the sole signal of olefin protons (6.16 ppm) and one signal of H—CI protons (4.09 ppm), as well as by the appearance of a W-coupling constant (2.2 Hz) for the H—CI and *anti*-H(7) protons.

Thus, the reaction of olefins of the bicyclo[2.2.1]heptane series with phosphorus triiodide in the presence of weak electrophiles opens up the possibility for the preparation of various mono- and diiodides with norbornane, norbornene, and nortricyclane structures that are impossible to synthesize by direct iodination of these olefins. The mechanisms of the reactions described above require thorough studies.

Experimental

Reaction of PI₃ with norbornene and norbornadiene. A solution of an olefin (3 mmol) was added to a solution of phosphorus triiodide (0.82 g, 2 mmol) in anhydrous methylene dichloride at −70 °C with stirring. After 0.5 h the temperature of the reaction mixture was gradually increased to ~20 °C, the reaction mixture was filtered through a silica gel column (*h* = 5 cm), the solvent was evaporated *in vacuo*, and the residue was chromatographed.

Reaction of PI₃ with norbornene and norbornadiene in the presence of ArSNR₂ or R₂NCl. A solution of *N*-(*o*-nitrophenyl-thio)- or *N*-(*p*-nitrophenylthio)morpholine (0.48 g, 2 mmol) or *N,N*-diethylchloramine (0.21 g, 2 mmol) in anhydrous methylene dichloride was added to a solution of phosphorus triiodide (0.82 g, 2 mmol) in the same solvent with stirring at −70 °C. A solution of olefin (3 mmol) was added after 10 min at the same temperature. After 0.5 h the temperature of the reaction mixture was gradually increased to ~20 °C, the reaction mixture was filtered through a silica gel column (*h* = 5 cm), the solvent was evaporated *in vacuo*, and the residue was chromatographed.

Reaction of I₂ with norbornene and norbornadiene. A solution of an olefin (3 mmol) was added to a solution of iodine (0.51 g, 2 mmol) in anhydrous methylene dichloride with stirring at −20 °C. The temperature of the reaction mixture was increased to ~20 °C, the mixture was stirred until the color of the I₂ disappeared, the solvent was evaporated *in vacuo*, and the residue was chromatographed.

endo-2-Iodo-*exo*-3-(*p*-7-nitrophenylthio)bicyclo[2.2.1]heptane (**1a**), *R*f 0.60. Found (%): C, 42.01; H, 3.94. C₁₃H₁₄NO₂IS. Calculated (%): C, 41.61; H, 3.76.

endo-2-Iodo-*exo*-3-(*o*-nitrophenylthio)bicyclo[2.2.1]heptane (**1b**), *R*f 0.60. Found (%): C, 41.07; H, 3.95. C₁₃H₁₄NO₂IS. Calculated (%): C, 41.61; H, 3.76.

exo-2-Iodobicyclo[2.2.1]heptane (**2**), *R*f 0.93. Found (%): C, 37.09; H, 4.94. C₇H₁₁I. Calculated (%): C, 37.86; H, 4.99.

trans-2,3-Diiodobicyclo[2.2.1]heptane (**3**), *R*f 0.76. Found (%): C, 24.01; H, 3.00. C₇H₁₀I₂. Calculated (%): C, 24.16; H, 2.70.

exo-*syn*-2,7-Diiodobicyclo[2.2.1]heptane (**4**), *R*f 0.38. For a **3** + **4** mixture, found (%): C, 23.82; H, 2.46. C₇H₁₀I₂. Calculated (%): C, 24.16; H, 2.70.

exo-2-Iodobicyclo[2.2.1]hept-5-ene (**5**), *R*f 0.78.

exo-2-Iodotricyclo[2.2.1.0³,⁵]heptane (**6**), *R*f 0.78. For a **5** + **6** mixture, found (%): C, 37.98; H, 3.97. C₇H₉I. Calculated (%): C, 38.21; H, 4.12.

exo,exo-2,5-Diiodobicyclo[2.2.1]heptane (**7**), *R*f 0.71. Found (%): C, 24.36; H, 2.96. C₇H₁₀I₂. Calculated (%): C, 24.16; H, 2.90.

exo,endo-2,6-Diiodotricyclo[2.2.1.0³,⁵]heptane (**8**), *R*f 0.68.
endo,endo-2,6-Diiodotricyclo[2.2.1.0³,⁵]heptane (**9**), *R*f 0.68. For a **8** + **9** mixture, found (%): C, 23.94; H, 2.09. C₇H₈I₂. Calculated (%): C, 24.30; H, 2.33.

exo,endo-2,3-Diiodobicyclo[2.2.1]hept-5-ene (**10**), *R*f 0.74. Found (%): C, 24.53; H, 2.51. C₇H₈I₂. Calculated (%): C, 24.30; H, 2.33.

exo,exo-2,3-Diiodobicyclo[2.2.1]hept-5-ene (**11**), *R*f 0.78. Found (%): C, 24.58; H, 2.57. C₇H₈I₂. Calculated (%): C, 24.30; H, 2.33.

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