Monatsh Chem (2009) 140:479–483
DOI 10.1007/s00706-008-0076-4

ORIGINAL PAPER

Thermal ring opening of 1,1-dibromo and 1-bromo-2-chloromethylcyclopropanes: observation of a formal debromochlorination

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Received: 23 September 2008 / Accepted: 26 September 2008 / Published online: 22 October 2008

Abstract When the title compounds are thermolyzed in the gas phase under vacuum or in hot quinoline, several products are formed. A predominant product in all cases is a chlorine-free buta-1,3-diene which has been formed by formal debromochlorination, a reaction not previously observed during thermolysis of chlorinated bromocyclopropanes.

Keywords Cyclopropanes · Dehalogenation · Conjugated dienes · Trapping · 4-Phenyl-1,2,4-triazoline-3,5-dione

Introduction

Thermolysis of halogenated cyclopropanes generally results in ring opening and formation of products in a predictable manner through substitution or dehydrohalogenation reactions. The structures of the products depend on the reaction conditions and on the substituents attached to the ring [1–4], a fact which can be illustrated by a few examples. Thus, heating of liquid 1,1-dibromo-2,2-dimethylcyclopropane (**1a**) at approximately 200 °C gives 1,2-dibromo-3-methyl-2-butene as the sole product in 60% yield [5, 6], but when gaseous **1a** passes through a glass tube kept at 500 °C under vacuum, only 2-bromo-3-methylbuta-1,3-diene is obtained in 96% yield [7]. A clean transformation also takes place when 1-chloro-1-fluoro-2-methoxy-2-methylcyclopropane (**1b**) is heated in quinoline above 50 °C; elimination of HCl takes place and furnishes 2-fluoro-3-methoxybuta-1,3-diene only, in 85% yield [8].

But some halocyclopropanes deviate from the established reactivity pattern and give unexpected products. Among these compounds are two gem-dichlorocyclopropanes with a chloromethyl group attached directly to the ring. When these compounds undergo thermal ring opening the main products are still halogenated alkenes, but not those expected from the accepted mechanism for such reactions; instead, products are formed by transformations which also involve rearrangements because of chlorine migration [9]. It is therefore apparent that the chloromethyl substituent significantly affects the reactivity of these chlorocyclopropanes. On this basis we became interested in investigating whether the chloromethyl substituent affects the reactivity pattern of bromo-substituted cyclopropanes in a similar fashion.

Results and discussion

In order to look for such a chloromethyl effect four chloromethyl-substituted bromocyclopropanes were thermolyzed under conditions similar to those employed to trigger **1a** and **1b** to react [5–7]. One of the compounds selected was the chloromethyl analogue to **1a**, viz. 1,1-dibromo-2-chloromethyl-2-methylcyclopropane (**3a**; R=CH_3, X=Br), whereas the others were **3b–3d** (Scheme 1). The gem-dibromides, **3a** and **3b**, were

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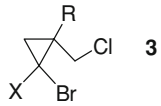


a R = Me, X = Br; **b** R = H, X = Br; **c** R = Me, X = H; **d** R = X = H

Scheme 1

synthesized from the corresponding allyl chlorides under phase-transfer catalysis [10–25] and ultrasound irradiation [26, 27] whereas the corresponding monobromides, **3c** and **3d**, were furnished as *cis–trans* mixtures by treating **3a** and **3b** with tributyltin hydride in diethyl ether at room temperature [21–25, 28, 29]. The monobromides could also be obtained by bromocarbene addition to the allyl chlorides using the method of Martel and Hiriart [30], but the yields were mediocre.

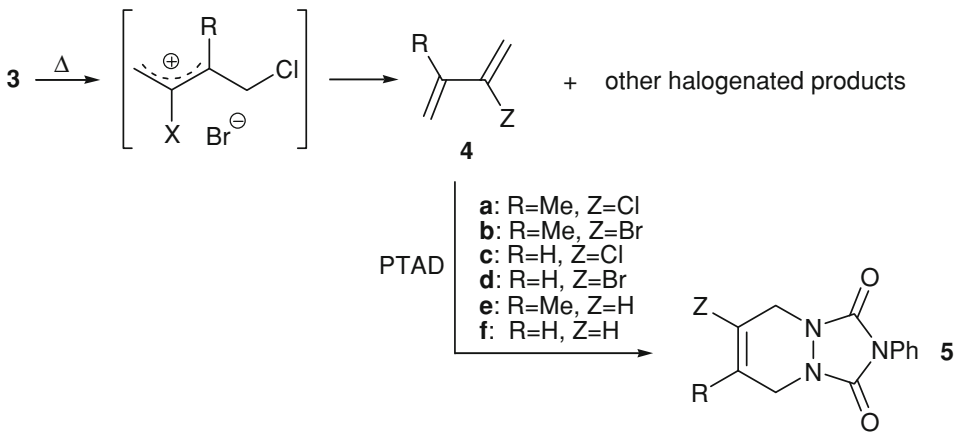
When **3a** was pyrolyzed by flash-vacuum pyrolysis (FVP) at 450–500 °C and in quinoline heated under reflux a large number of products were formed. Two products predominated and based on spectroscopic and spectrometric evidence they seemed to be 2-chloro-3-methylbuta-1,3-diene (**4a**) [5, 6, 31] and 2-bromo-3-methylbuta-1,3-diene (**4b**) [32]. Both compounds were difficult to isolate in the pure form, but when the pyrolysate was treated directly with 4-phenyl-1,2,4-triazoline-3,5-dione (PTAD) at room temperature (until the red colour persisted) both dienes underwent Diels–Alder reaction, and gave the corresponding 3-chloro and 3-bromo-4-methyl-8-phenyl-1,6,8-triazabicyclo[4.3.0]non-3-ene-7,9-dione (**5a** and **5b**) (Scheme 2). The best combined yield of **5a** and **5b** was obtained when **3a** was thermolyzed by FVP, but it is as low as 11% due to loss of dienes during the Diels–Alder reaction.

Pyrolysis of 1,1-dibromo-2-chloromethylcyclopropane (**3b**) gave results similar to those obtained for **3a** except that the product mixture appeared to be less complex in hot quinoline than by FVP. Again, two major products were isolated under both conditions, 2-chlorobuta-1,3-diene (chloroprene) (**4c**) [9, 21] and 2-bromobuta-1,3-diene (bromoprene) (**4d**) [21, 33, 34], and again the yields were sensitive to the reaction temperature. The best combined isolated yield of **4c** and **4d** was 13%. Both dienes could be trapped by PTAD and the products were identified as the corresponding cycloadducts 3-chloro and 3-bromo-8-phenyl-1,6,8-triazabicyclo[4.3.0]-non-3-ene-7,9-dione (**5c** [35] and **5d**; Scheme 2).

Thermolyses of an isomeric mixture of 1-bromo-2-chloromethyl-2-methylcyclopropane (**3c**) gave reaction mixtures, the composition of which varied substantially with the reaction conditions. GC–MS analysis clearly indicated that all products except one contained one or two halogen atoms. Both in hot quinoline and under FVP conditions the main product seemed to be isoprene (**4e**), which was trapped by PTAD and gave 3-methyl-8-phenyl-1,6,8-triazabicyclo[4.3.0]non-3-ene-7,9-dione (**5e**), with physical properties identical with those of an authentic sample of the compound prepared by independent synthesis [36]. The highest yield of **5e** obtained is ca. 8%.

Just as dibromide **3b** reacted similarly to the methylated dibromo analogue **3a** but gave less complex reaction mixtures, monobromide **3d** turned out to react similarly to **3c**, but afforded fewer products both by FVP and in hot quinoline. GC–MS analyses of cold pyrolysates just after pyrolysis clearly indicated that all products except one contained either one or two halogen atoms. The halogen-free compound was the main product under most reaction conditions, and when analyzed spectroscopically and by GC–MS its structure proved to be buta-1,3-diene (**4f**). As expected this diene could be trapped by treatment of the product mixtures with PTAD; this gave 8-phenyl-1,6,8-triazabicyclo[4.3.0]non-3-ene-7,9-dione (**5f**), which had physical properties identical with those of an authentic sample of the compound [33, 34]. The highest yield of **5f** achieved is 30%.

Scheme 2



The results presented above are surprising when the established theory for electrocyclic ring opening of halogenated cyclopropanes is considered [1–4, 37–45]. If the chloromethylated bromocyclopropanes **3** had reacted in accordance with this theory, just like **1a** [5–7, 46], **1b** [8], and other chlorine-free analogues to **3a**–**3d** [46, 47] do under comparable conditions, the reaction mixtures should have consisted of chlorinated allyl bromides and chloro-substituted 2-bromobuta-1,3-dienes (from **3a** and **3b**) or chloro-substituted buta-1,3-dienes (from **3c** and **3d**), i.e. compounds with the same number of halogen atoms or one halogen atom less than the starting materials. From our spectroscopic and GC–MS analyses of the reaction mixtures it is clear that such products are formed, but they are not the main products; instead dienes with two halogen atoms less than the starting material predominate. It is therefore clear that the reactivity of the bromo-substituted cyclopropane ring is significantly modified by replacing a methyl group with a chloromethyl moiety.

How dienes **4a**–**4d**, isoprene (**4e**), and buta-1,3-diene (**4f**) have been formed has yet to be firmly established. The formation of a 2-chlorobuta-1,3-diene and a 2-bromobuta-1,3-diene in the same reaction, **4a** and **4b** from **3a**, and also **4c** and **4d** form **3b**, strongly indicates that at least two mechanisms are involved. Several possibilities can be invoked, particularly for the generation of the chlorine-free dienes. Thus, it is conceivable that the allylic cation formed upon opening of the cyclopropane ring (Scheme 2) undergoes a retro-chloronium addition (facilitated by the bromide ion); this will give dienes by a formal debromochlorination reaction, which has previously not been observed in thermolytic ring opening of halogenated cyclopropanes. Another possibility could be a mechanistic cross-over from an ionic to a radical pathway, which has been observed at higher temperatures with highly fluorinated and chlorinated cyclopropanes [9]. Such a change can be initiated by homolytic cleavage of the cyclopropane ring or the chlorine-methylene bond next to the ring.

Experimental

General remarks

The instrumentation and the analytical methods have been described elsewhere [48] In addition, some of the HRMS analyses were obtained on an Autospec Ultima2000 Micromass spectrometer with EBE geometry. The spectrometer was operated in the EI mode at 70 eV and the samples were injected at an ion source temperature of 170 °C.

*Syntheses of cyclopropanes **3a**–**3d***

1,1-Dibromo-2-chloromethyl-2-methylcyclopropane (**3a**) and 1,1-dibromo-2-chloromethylcyclopropane (**3b**) were synthesized from methallyl chloride and allyl chloride under phase-transfer conditions as described in Refs. [10–25]. Treatment of **3a** and **3b** with tributyltin hydride in diethyl ether at room temperature gave 1-bromo-2-chloromethyl-2-methylcyclopropane (**3c**) and 1-bromo-2-chloromethylcyclopropane (**3d**) in good yields [21–25].

*Thermolyses of **3**; general procedures*

Gas phase

The reactions were carried out with a horizontal electrical furnace (Heraeus RoK/A 4/60) equipped with a quartz tube (length 60 cm, 2.5 cm i.d.), which was packed with Pyrex glass wool containing some anhydrous Na_2CO_3. The pyrolysis temperature was measured using a thermocouple wound around the tube. The pyrolysate was collected in a trap cooled by liquid N_2 or CO_2–acetone, and the product mixtures were thoroughly analyzed (GC, IR, ^{1}H NMR, ^{13}C NMR, MS) before work-up and trapping experiments with 4-phenyl-3*H*-1,2,4-triazole-3,5(4*H*)-dione (PTAD) dissolved in dichloromethane were carried out. PTAD was added until its red colour persisted. The products were subsequently isolated by flash chromatography.

Liquid phase

Thermolyses in quinoline were performed essentially as described elsewhere [31, 49]. The substrate was mixed with three times the amount of quinoline (b.p. 235 °C) under nitrogen and stirred under reflux. The pyrolysate was collected in a cooled flask (liquid N_2 or CO_2–isopropyl alcohol), and when the reaction was over, as indicated by GC analysis, the cooling bath was removed before PTAD in CH_2Cl_2 was added until its red colour persisted. The products were then isolated by flash chromatography.

*Thermolysis of 1,1-dibromo-2-chloromethyl-2-methylcyclopropane (**3a**)*

Dibromide **3a** (2.04 g) was thermolyzed in the gas phase at 450–500 °C/0.8–1.2 mmHg. The cold pyrolysate was treated with PTAD, and the products were isolated by flash chromatography (SiO_2, *n*-hexane–ethyl acetate 7:3). A mixture of several unidentified products (1.39 g; $R_f = 0.80$) and a white solid (0.28 g; $R_f = 0.34$), consisting of a 1:9 mixture of two compounds, were obtained. Flash chromatography was used to isolate pure samples of the two compounds, but several separations were necessary to achieve this goal. The first separation gave a fraction slightly enriched in the minor product and another fraction somewhat

enriched in the major product. Both these fractions were subjected separately to a second flash chromatography elution and similar enrichments took place. This process was repeated until pure samples of the two compounds had been obtained and enabled us to perform structure elucidation by spectroscopy and spectrometry. The minor compound seemed to be 3-chloro-4-methyl-8-phenyl-1,6,8-triazabicyclo-[4.3.0]non-3-ene-7,9-dione (**5a**) whereas the major compound was 3-bromo-4-methyl-8-phenyl-1,6,8-triazabicyclo[4.3.0]non-3-ene-7,9-dione (**5b**).

3-Chloro-4-methyl-8-phenyl-1,6,8-triazabicyclo-[4.3.0]non-3-ene-7,9-dione (**5a**, $C_{13}H_{12}N_3O_2Cl$)
M.p. 201–202 °C (decomposition). IR (KBr): $\bar{\nu} = 3,061$w, 2,965w, 2,914w, 2,852w, 1,771m, 1,717s, 1,598w, 1,503m, 1,427br, 1,345w, 1,306w, 1,261m, 1,135m, 1,082w, 940w, 913w, 798w, 750w, 690w, 642w cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$, Me_4Si): δ = 1.92-1.94 (3H, m, CH_3), 4.11–4.13 (2H, m, CH_2), 4.21–4.26 (2H, m, CH_2), 7.33–7.55 (5H, m) ppm; ^{13}C NMR (50 MHz, $CDCl_3$, Me_4Si): δ = 16.6 (CH_3), 47.5 (CH_2), 47.8 (CH_2), 119.3 (C), 124.6 (C), 125.2 (2CH), 128.1 (CH), 129.0 (2CH) 130.8 (C), 151.8 (CO), 152.0 (CO) ppm; MS (70 eV): *m/z* (%) = 277 (M^+, 61), 242 (11), 123 (100), 119 (66), 182 (21), 91 (26) and 67 (36); HRMS (EI): *m/z* calcd. for M^+, $C_{13}H_{12}N_3O_2Cl$, 277.0618; found 277.0617.

3-Bromo-4-methyl-8-phenyl-1,6,8-triazabicyclo[4.3.0]non-3-ene-7,9-dione (**5b**, $C_{13}H_{12}N_3O_2Br$)
M.p. 197–198 °C (decomposition). IR (KBr): $\bar{\nu} = 2,955$w, 2,921m, 2,851w, 1,775m, 1,710s, 1,600w, 1,503m, 1,430br, 1,338w, 1,306w, 1,261m, 1,134m, 1,089w, 1,076w, 1,024w, 907w, 792w, 745m, 715w, 687w cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$, Me_4Si): δ = 1.96–1.97 (3H, m, CH_3), 4.13–4.14 (2H, m, CH_2), 4.33–4.36 (2H, m, CH_2), 7.46–7.53 (5H, m) ppm; ^{13}C NMR (100 MHz, $CDCl_3$, Me_4Si): δ = 19.6 (CH_3), 48.1 (CH_2), 49.8 (CH_2), 109.8 (C), 125.3 (2CH), 127.4 (C), 128.2 (CH), 129.1 (2CH), 130.8 (C), 151.7 (CO), 152.1 (CO) ppm; MS (70 eV): *m/z* (%) = 321 (M^+, 30), 242 (3), 207 (11), 123 (100), 119 (51), 91 (24), 67 (53); HRMS (EI): *m/z* calcd. for M^+, $C_{13}H_{12}N_3O_2Br$, 321.0113; found 321.0117.

*Thermolysis of 1,1-dibromo-2-chloromethylcyclopropane (**3b**)*

Dibromide **3b** (201 mg) was thermolyzed in quinoline at 260 °C and a complex product mixture (75.2 mg) was collected. The mixture contained two main components which were formed in essentially equal amounts; these were obtained in 13% total yield according to GC analysis, and seemed to be 2-chlorobuta-1,3-diene (**4c**) [9, 21] and 2-bromobuta-1,3-diene (**4d**) [21, 33, 34] according to ^1H NMR and MS spectra. Treatment with PTAD and subsequent workup gave mixtures of 3-chloro-8-phenyl-1,6,8-triazabicyclo[4.3.0]non-3-ene-7,9-dione (**5c** [37–45]) and 3-bromo-8-phenyl-1,6,8-triazabicyclo[4.3.0]non-3-ene-7,9-dione (**5d**). Based on the spectroscopic and spectrometric properties of these mixtures and those of an authentic sample of **5c** [35], the spectral data of **5d** were obtained.

3-Bromo-8-phenyl-1,6,8-triazabicyclo[4.3.0]non-3-ene-7,9-dione (**5d**, $C_{12}H_{10}N_3O_2Br$)
IR ($CHCl_3$): $\bar{\nu} = 1,755$w, 1,723s, 1,603m, 630s cm^{-1}; ^1H NMR (250 MHz, $CDCl_3$, Me_4Si): δ = 4.20–4.27 (3H, m), 4.31–4.34 (1H, m), 6.25–6.29 (1H, m, =CH), 7.35–7.54 (5H, m) ppm; ^{13}C NMR (62.5 MHz, $CDCl_3$, Me_4Si): δ = 45.1 (CH_2), 49.6 (CH_2), 113.6 (C), 122.3 (CH), 125.2 (2CH), 128.2 (CH), 129.1 (2CH), 130.8 (C), 151.9 (CO), 152.3 (CO) ppm; MS (EI): *m/z* (%) = 310 (M^+, 9), 309 (M^+, 76), 308 (M^+, 10), 307 (M^+, 80), 228 (9), 134 (9), 132 (93), 119 (93), 109 (100), 91 (34); HRMS (EI): *m/z* calcd. for M^+, $C_{12}H_{10}N_3O_2Br$, 308.99359; found 308.99623.

*Thermolysis of 1-dibromo-2-chloromethyl-2-methylcyclopropane (**3c**)*

Monobromocyclopropane **3c** (217 mg) was thermolyzed in the gas phase at 417 °C/2.6 mmHg. The cold pyrolysate was treated with PTAD, and the products were isolated by flash chromatography (SiO_2, *n*-hexane–ethyl acetate 7:3). Several mixtures of unidentified products were obtained, but in addition 46 mg of contaminated 3-methyl-8-phenyl-1,6,8-triazabicyclo[4.3.0]non-3-ene-7,9-dione (**5e**) was isolated. The IR, ^1H NMR, and ^{13}C NMR spectra of **5e** showed all the peaks exhibited by an authentic sample of the compound, synthesized from isoprene as described by Gillis and Hagarty [36]. Furthermore, the authentic sample co-eluted with **5e** when GC analyses were performed under various conditions, and finally, their mass spectra were identical.

*Thermolysis of 1-dibromo-2-chloromethylcyclopropane (**3d**)*

Monobromocyclopropane **3d** (0.18 g) was thermolyzed in the gas phase at 470 °C/20 mmHg. When all the substrate had passed through the tube a dichloromethane solution of PTAD was added to the pyrolysate until the red colour persisted. Column chromatography (SiO_2, $CHCl_3$) gave 68 mg (30%) 8-phenyl-1,6,8-triazabicyclo[4.3.0]non-3-ene-7,9-dione (**5f**) whose spectroscopic properties were identical with those of an authentic sample of the compound prepared by independent synthesis [33, 34]. M.p. 155–157 °C (Ref. [33] 158–159 °C); MS (70 eV): *m/z* (%) = 229 (M^+, 100), 119 (83), 110 (19), 91 (27), 82 (36),

77 (7), 64 (17), 54 (48); HRMS (EI): *m/z* calcd. for M^+, $C_{12}H_{11}N_3O_2$, 229.085127; found 229.0846.

Acknowledgments Financial support from the University of Tromsø and the University of Bergen is highly appreciated. Thanks are also due to the University of Bergen for a travel grant (to KFAS) and to the University of Tasmania for excellent working conditions (for LKS) during a recent sabbatical stay.

References

1. Wendisch D (1971) Carbocyclische Dreiring-Verbindungen. Methoden der Organischen Chemie (Houben-Weyl), Band IV/3. Thieme, Stuttgart, pp 615–632
2. Boche G, Walborsky HM (1990) In: S Patai S, Rappoport Z (eds) Cyclopropane-derived reactive intermediates, Chap 3. Wiley, Chichester
3. Jendralla H (1997) In: de Meijere A (Ed) Carbocyclic three-membered ring compounds, methods of organic chemistry (Houben–Weyl) vol E 17c. Thieme, Stuttgart, pp 2313–2387
4. Robinson GC (1968) J Org Chem 33:607
5. Sandler SR (1968) Chem Ind (London) 1481
6. Sandler SR (1967) J Org Chem 32:3876
7. Werstiuk NH, Roy CD (2001) Tetrahedron Lett 42:3255
8. Patrick TB, Rogers J, Gorrell K (2002) Org Lett 4:3155
9. Fields R, Haszeldine RN, Peter D (1969) J Chem Soc (C) 165
10. Makosza M, Wawrzyniewicz M (1969) Tetrahedron Lett 4659
11. Baird MS, Baxter AGW, Devlin BRJ, Searle RJG (1979) J Chem Soc, Chem Commun 210
12. Al-Dulayymi A, Li X, Neuenschwander M (2000) Helv Chim Acta 83:571
13. Hirao T, Nagata S, Agawa T (1985) Tetrahedron Lett 26:5795
14. Dueker A, Szeimies G (1985) Tetrahedron Lett 26:3555
15. Arct J, Skattebøl L (1982) Acta Chem Scand Ser B B36:593
16. Baird MS (1981) J Chem Res (S) 352
17. Labeish NN, Kharicheva EM, Mandel'shtam TV, Kostikov RR (1978) Zh Org Khim 14:818
18. Labeish NN, Kharicheva EM, Mandel'shtam TV, Kostikov RR (1978) Chem Abstr 89:23809
19. Skattebøl L, Nilsen NO, Myhren F (1986) Acta Chem Scand Ser B B40:782
20. Nilsen NO, Skattebøl L, Baird S, Buxton SR, Slowey PD (1984) Tetrahedron Lett 25:2887
21. Pettersen A, Jørgensen E, Sydnes LK (1990) Acta Chem Scand 44:603
22. D'yachenko AI, Abramova NM, Zotova SV, Nesmeyanova OA, Bragin OV (1985) Izv Akad Nauk SSSR, Ser Khim 2043
23. D'yachenko AI, Abramova NM, Zotova SV, Nesmeyanova OA, Bragin OV (1986) Chem Abstr 104:168022
24. D'yachenko AI, Korneva OS, Abramova NM, Nefedov OM (1984) Izv Akad Nauk SSSR, Ser Khim 2818
25. D'yachenko AI, Korneva OS, Abramova NM, Nefedov OM (1985) Chem Abstr 102:166343
26. Xu L, Brinker UH (1998) Preparation of 7,7- dibromobicyclo[4.1.0]heptane. In: Luche J-L (ed) Synthetic organic sonochemistry. Plenum Press, New York, p 344
27. Brinker UH, Lin G, Xu L, Smith WB, Mieusset J-L (2007) J Org Chem 72:8434
28. Seyferth D, Yamazaki H, Alleston DL (1963) J Org Chem 28:703
29. Sydnes LK (1978) Acta Chem Scand B B32: 47
30. Martel B, Hiriart JM (1972) Synthesis 201
31. Baird MS, Mahli BS, Sheppard L (1990) J Chem Soc, Perkin Trans 1:1881
32. Baird MS, Hussain HH (1985) J Chem Res (S) 183
33. Jensen F, Foote CS (1989) J Am Chem Soc 109:6376
34. Cookson RC, Gilani SSH, Stevens DR (1967) J Chem Soc (C) 1905
35. Sauer J, Schröder B (1967) Chem Ber 100:678
36. Gillis BT, Hagarty JD (1967) J Org Chem 32:330
37. DePuy CH, Schnack LG, Hausser JW, Wiedemann W (1965) J Am Chem Soc 87:4006
38. Cristol SJ, Sequeira RM, DePuy CH (1965) J Am Chem Soc 87:4007
39. Ledlie DB, MacLean S (1969) J Org Chem 34:1123
40. Parham WE, Yong KS (1970) J Org Chem 35:683
41. Hausser JW, Uchic JT (1972) J Org Chem 37:4087
42. Sydnes LK, Skattebøl L, Chapleo CB, Leppard DG, Svanholt KL, Dreiding AS (1975) Helv Chim Acta 58:2061
43. Sydnes LK, Hemmingsen TH (1985) Acta Chem Scand B B39:93
44. Faza ON, Lopez CS, Alvarez R, de Lera AR (2004) Org Lett 6:905
45. Faza ON, Lopez CS, Alvarez R, de Lera AR (2004) J Org Chem 69:9002
46. Asahara T, Ono K, Tanaka K (1971) Bull Chem Soc Jpn 44:1130
47. Sliwinski WF, Su TM, PvR Schleyer (1972) J Am Chem Soc 94:133
48. Sydnes LK, Alnes KFS, Natalia Erdogan (2005) Monatshefte für Chemie 136:1737
49. Ando T, Hosaka H, Yamanaka H, Funasaka W (1969) Bull Chem Soc Jpn 42:2013