

Article

Not peer-reviewed version

Critical Re-Examination of the Synthesis of Adamantyl Hydroperoxide

Ilya Nazarov , Daria Zapravdina , Anna Maximova , [Ilya A. Yakushev](#) , Victor Chapurkin , [Vladimir Burmistrov](#) *

Posted Date: 25 February 2026

doi: 10.20944/preprints202602.1470.v1

Keywords: 1,3-dehydroadamantane; hydroperoxides; hydrogen peroxide; 1-hydroperoxyadamantane; SC XRD



Preprints.org is a free multidisciplinary platform providing preprint service that is dedicated to making early versions of research outputs permanently available and citable. Preprints posted at Preprints.org appear in Web of Science, Crossref, Google Scholar, Scilit, Europe PMC.

Copyright: This open access article is published under a [Creative Commons CC BY 4.0 license](#), which permit the free download, distribution, and reuse, provided that the author and preprint are cited in any reuse.

Disclaimer/Publisher's Note: The statements, opinions, and data contained in all publications are solely those of the individual author(s) and contributor(s) and not of MDPI and/or the editor(s). MDPI and/or the editor(s) disclaim responsibility for any injury to people or property resulting from any ideas, methods, instructions, or products referred to in the content.

Article

Critical Re-Examination of the Synthesis of Adamantyl Hydroperoxide

Ilya Nazarov ¹, Daria Zapravdina ¹, Anna Maksimova ², Ilya A. Yakushev ², Victor Chapurkin ¹ and Vladimir Burmistrov ^{1,*}

¹ Department of Organic Chemistry, Volgograd State Technical University, Volgograd, 400005, Russia

² Laboratory of metal complex catalysis, Kurnakov Institute of General and Inorganic Chemistry, Russian Academy of Sciences, Moscow, 119071, Russia

* Correspondence: crus_himself@mail.ru

Abstract

This study investigates the synthesis of 1-hydroperoxyadamantane addressing discrepancies in prior literature. Our results demonstrate that previous authors were unable to obtain claimed compound under the conditions they described. We confirmed that 1-hydroperoxyadamantane can be synthesized in good yields via the reaction of 1,3-dehydroadamantane with concentrated hydrogen peroxide in DCM. Structure of 1-hydroperoxyadamantane was confirmed by SC XRD analysis. These findings clarify the conditions necessary for successful synthesis of this compound and highlight the importance of comprehensive analytical verification. The study also provides a very detailed, repeatedly verified method for synthesizing 1,3-dehydroadamantane and a description of the necessary laboratory equipment.

Keywords: 1,3-dehydroadamantane; hydroperoxides; hydrogen peroxide; 1-hydroperoxyadamantane; SC XRD

1. Introduction

It is known that 1,3-dehydroadamantane, which belongs to strained propellanes, enters into many addition reactions with various reagents [1–3]. Reactions of compounds containing a multiple bond with hydrogen peroxide in many cases proceed ambiguously, with the formation of a complex mixture of products that cannot always be separated [4]. The closest analogues of such a reaction include the well-studied interaction of carbonyl compounds with hydrogen peroxide, which proceeds with the formation of a complex mixture of peroxide compounds. In this reaction, hydrogen peroxide, as a nucleophilic reagent, reacts at the carbonyl group with the formation of a primary intermediate, which then interacts with the carbonyl group to form a new peroxide. Further reactions occur with the formation of a complex mixture of difficult to separate peroxides [5]. The interaction of unsaturated compounds with hydrogen peroxide is less studied, but in this case, a complex mixture of difficult to separate peroxides is also formed [6].

Isolating individual peroxides from a mixture is a virtually impossible task, as many of them are unstable compounds and decompose during isolation. New approaches to peroxide synthesis, including reactions with hydrogen peroxide, have proven to be successful. These were implemented using carbon tetrachloride or dichloroethane rather than diethyl ether, which is generally accepted for such reactions. Changing the solvent polarity previously allowed us to obtain new hydroxyhydroperoxides based on fluorinated and non-fluorinated carbonyl compounds, both aliphatic, cyclic, and aromatic [7]. The use of diethyl ether as a solvent proved erroneous, as it facilitated the conversion of the initial hydroxyhydroperoxide reaction products into further substitution products due to the formation of hydrogen bonds between the solvent, hydrogen peroxide, and the resulting hydroxyhydroperoxide. An exception are hydroxyhydroperoxides obtained by reacting hydrogen peroxide with perfluorocyclohexanone and perfluorocyclopentanone.

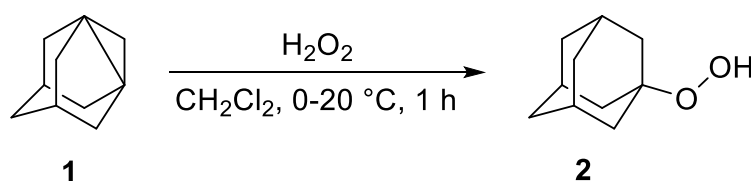
In this reaction, with a lack of hydrogen peroxide, 1,1-dihydroxyperoxides are formed, but they turned out to be unstable and decomposed within 24 hours [8].

Thus, there are virtually no targeted attempts to synthesize 1-hydroperoxyadamantane in the literature. In a number of studies, it is cited as a minor oxidation product of adamantane derivatives, but no data on its isolation or detailed characterization are provided in these articles [9–11]. The interaction of 1,3-dehydroadamantane with hydrogen peroxide, which can lead to the formation of 1-hydroperoxyadamantane, deserves special mention. The authors [12,13] did not pay attention to such key features of reactions involving hydrogen peroxide as the role of the solvent and the effect of temperature, which determine the possibility of peroxide formation. Widely known results of studies on the interaction of hydrogen peroxide with compounds containing multiple bonds were not analyzed, which could have made it possible to avoid a number of errors in the experimental design and to establish the absence of peroxide compounds in the reaction products. The authors of these studies reacted 1,3-dehydroadamantane with hydrogen peroxide in diethyl ether, but the latter readily forms hydrogen bonds with hydrogen peroxide and prevents the formation of 1-hydroperoxyadamantane. This reaction should also have been carried out with an excess of hydrogen peroxide, but the authors, using their own erroneous method of extracting a 30% aqueous hydrogen peroxide solution with diethyl ether, were unable to obtain highly concentrated hydrogen peroxide solutions. They also failed to perform high-quality reactions on the peroxide group in their reaction products. Therefore, the only method for synthesizing 1-hydroperoxyadamantane described in the literature requires critical rethinking.

2. Results and Discussion

First, we extracted an aqueous solution of hydrogen peroxide with absolute diethyl ether using the method of the authors of the patent¹³ and found (by iodometric titration) that the concentration of hydrogen peroxide in the ether solution obtained in this way was $2.8 \pm 0.1\%$. Thus, the described reaction of 1,3-dehydroadamantane with hydrogen peroxide was carried out in the absence of the latter. By repeating the described method, we indeed obtained a product whose heating above 200°C was accompanied by sublimation, which the authors mistakenly took for the decomposition of 1-hydroperoxyadamantane. TLC analysis of this product for the presence of peroxide compounds revealed only one spot $R_f=0.36$, which refers to the residual content of hydrogen peroxide. Spectral analysis confirmed that adamantane is the reaction product under the specified conditions.

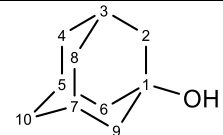
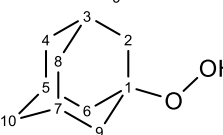
Then we studied the reaction of 1,3-dehydroadamantane (1) with hydrogen peroxide using DCM as a solvent (Scheme 1). We used 90% hydrogen peroxide, to which a solution of 1,3-dehydroadamantane in DCM was added dropwise at $0-5^\circ\text{C}$. After adding the reagents, the reaction mixture was slowly brought to room temperature and stirred for 1 h. The solvent was removed under vacuum. After recrystallization from hexane, a product with a mp of $89-91^\circ\text{C}$ was obtained in 55% yield. TLC carried out in a diethyl ether:cyclohexane = 1:1 system (visualization with KI in CH_3COOH) showed the presence of only one peroxide with $R_f = 0.7$.



Scheme 1. The proposed method for the synthesis of 1-hydroperoxyadamantane (2).

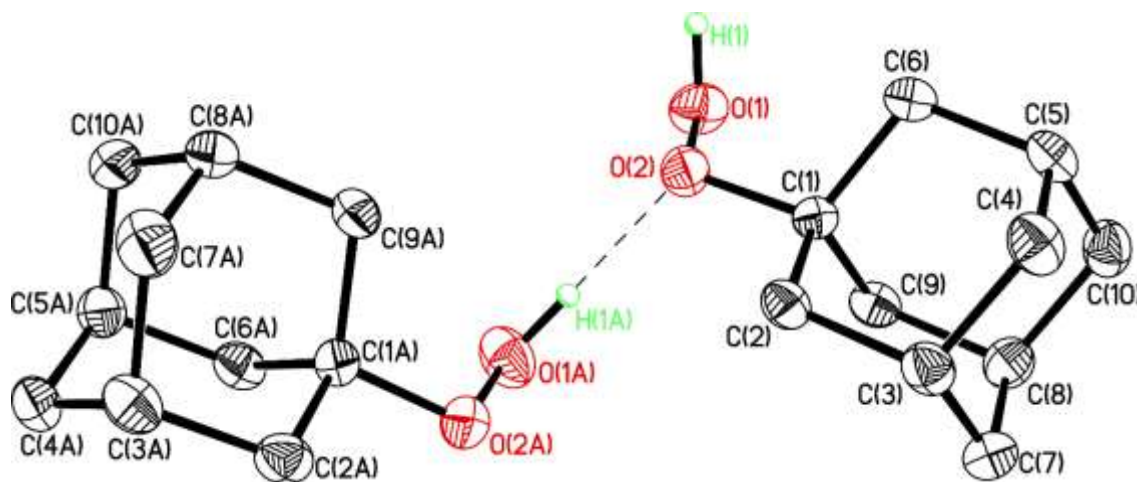
Under GC-MS conditions, 1-hydroperoxyadamantane (2) decomposes to form 1-hydroxyadamantane. Therefore, we compared the NMR spectra of the obtained product and commercially available 1-hydroxyadamantane. A comparison of the chemical shifts in the ^1H and ^{13}C NMR spectra of compound 2 and 1-hydroxyadamantane is presented in Table 1.

Table 1. Comparison of chemical shifts in NMR spectra for 1-hydroperoxyadamantane (**2**) and 1-hydroxyadamantane.

	¹ H				¹³ C			
	4,8,10 CH ₂	2,6,9 CH ₂	3,5,7 CH	OH	4,8,10 CH ₂	2,6,9 CH ₂	3,5,7 CH	1 C-O
	1.51-1.56 m	1.58 d 2.9 Hz	2.03 br.s	4.29 br.s	30.5	36.4	45.7	66.4
	1.54-1.61 m	1.68 d 3.0 Hz	2.10 br.s	10.59 br.s	34.9	41.2	45.1	82.8

By slow evaporation of a solution of 1-hydroperoxyadamantane in a mixture of dichloromethane and ethanol (1:1) at 4°C, crystals suitable for study by SC XRD analysis were obtained. This finally confirmed that we had obtained compound **2**.

According to the SC XRD data, **2** crystallizes in the monoclinic *P2₁* space group with two crystallographically independent molecules in an asymmetric unit (Figure 1). Both molecules have similar molecular geometry with slightly different interatomic distances and angles, within the experimental error (Tables S2 and S3). At the same time, all bond lengths and angles are within normal range for adamantane moiety, and more noticeable feature of the molecule is hydroperoxy group, forming intermolecular hydrogen bonds with neighboring molecules (Figures 1 and 2).

**Figure 1.** The molecular structure and atom numbering scheme of 1-hydroperoxyadamantane (**2**), with displacement ellipsoids drawn at the 50% probability level. C–H hydrogen atoms are omitted for clarity.

The C–O (1.449(2)–1.451(2) Å) and O–O (1.469(2) Å) distances are varying within values that are close to the mean C–O and O–O bond lengths (1.43 Å and 1.46 Å, respectively) for H–O–O–C groups deposited in Cambridge Structural Database (274 entries). It should be noted that difference between dihedral angles C(1)–O(2)–O(1)–H(1) 101(2)° and C(1A)–O(2A)–O(1A)–H(1A) 97(3)° cannot be considered statistically significant in that case. Each molecule of **2** takes part in the formation of two intermolecular hydrogen bonds, being a donor in the case of hydrogen atoms H(1), H(1A) and an acceptor by oxygen atoms O(2), O(2A), and such interactions can be classified as a classic type H-bonds based on geometrical parameters (Table S4).

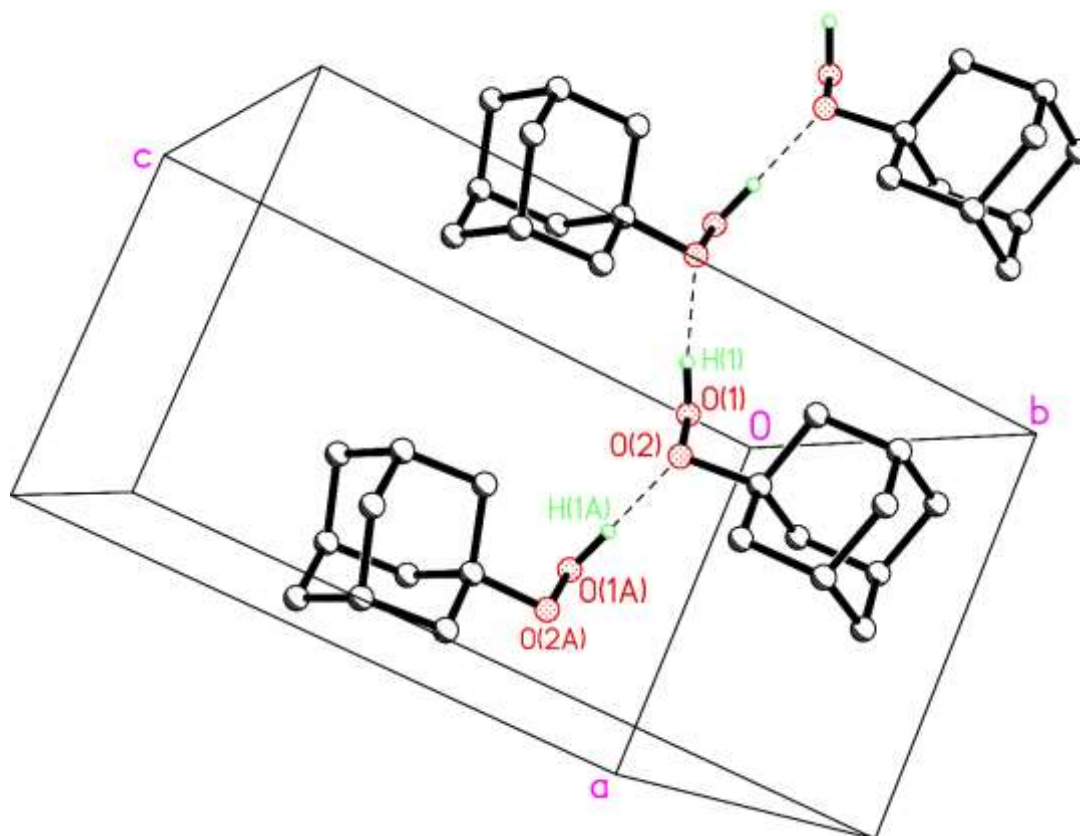


Figure 2. Hydrogen bonding between molecules of 1-hydroperoxoadamantane (**2**).

Crystal structure packing analysis showed that molecules of **2** forms infinite chains *via* hydrogen bonding, which in turn interact with each other through weak Van der Waals interactions (Figure 3) as expected.

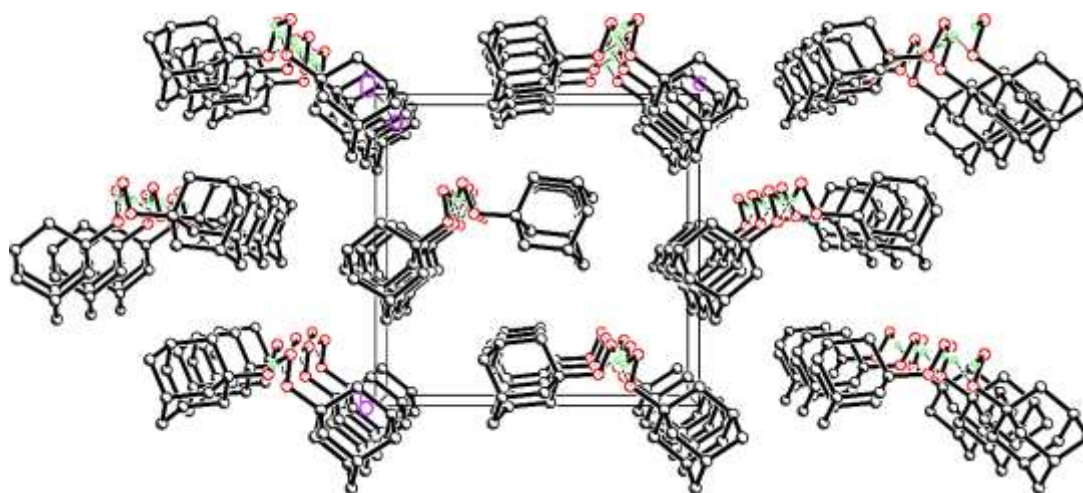
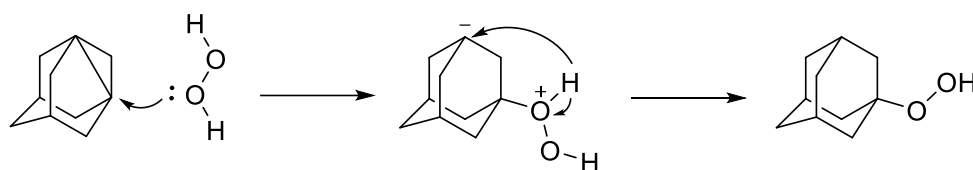


Figure 3. Fragment of the crystal packing of 1-hydroperoxoadamantane (**2**). Stick-and-ball model projected *along* the crystallographic *a*-axis. C–H hydrogen atoms are omitted for clarity.

When the reaction time of 1,3-dehydroadamantane with hydrogen peroxide is increased to 3 hours, the yield of 1-hydroperoxyadamantane decreases to 2%. According to TLC data, the same two peroxides remain in the reaction mixture: 1-hydroperoxyadamantane ($R_f = 0.7$) and hydrogen peroxide ($R_f = 0.36$). Thus, it can be concluded that when the synthesis time is increased to 3 hours, 1-hydroperoxyadamantane decomposes and is converted to adamantane.

Since the reaction proceeds quite rapidly at low temperatures, we assume an ionic mechanism (Scheme 2).



Scheme 2. Proposed mechanism of interaction of **1** with hydrogen peroxide.

Due to the specific shape of the propellane bond in compound **1**, two nodal carbon atoms are exposed to attack by a nucleophile. In the first step, the oxygen electron pair attacks one of the nodal atoms (either one, as they are equivalent). Both electrons from the propellane bond are then localized on the second nodal atom. In the second step, the positively charged oxygen atom takes the electrons from the O-H bond, and the corresponding hydrogen atom moves to the negatively charged nodal carbon atom, forming compound **2**.

4. Materials and Methods

The structure of the obtained compounds was confirmed using ^1H and ^{13}C NMR spectroscopy on a Bruker DPX 300 spectrometer (300 MHz, Bruker, USA) in DMSO-*d*₆ solvent. NMR spectra were calibrated using residual solvent signals. Mass spectra were recorded on an Agilent MS 5977b chromatograph/mass spectrometer (Agilent Technologies, Inc., USA) with electron impact ionization (EI). High-resolution mass spectra were obtained on Maxis MicroTOF II time-of-flight mass spectrometer system (Bruker Daltonics Inc., Germany). The purity of the obtained compounds was determined on an HPLC-UV system (Jasco PU 980 pump, Gilson 159 detector), Dr. Maisch Reprisil-Pur C18-AQ column (150x4 mm, 3 μm). Purification of the obtained compounds was performed on a Büchi Pure C-815 Flash flash chromatograph (Büchi Labortechnik AG, Switzerland) using FlashPure cartridges. Melting points were measured on a Büchi M-565 (Büchi Labortechnik AG, Switzerland) and the average values of three independent experiments were recorded.

1,3-Dibromoadamantane was synthesized as described in literature [14]. Commercially available 30% hydrogen peroxide was concentrated to 90% in rotary evaporator under reduced pressure and calibrated iodometrically. Other reagents were commercially available and were used without additional purification. Solvents were dried using generally known methods.

Synthetic Procedures

Preparation of a catalyst for the synthesis of 1,3-dehydroadamantane. A 50 ml flat-bottom conical flask was charged with 20 ml of anhydrous THF, 2.0 g of finely chopped lithium metal, and 150 μl of MePh₂SiCl. The flask was purged with argon, sealed with parafilm, and stirred magnetically. The catalyst prepared in this manner can be used the next day and stored for 3 months without loss of activity.

Synthesis of 1,3-dehydroadamantane (1). 5.5 g of 1,3-dibromoadamantane was added to a pre-prepared catalyst. The flask was purged with argon, sealed with parafilm, and stirred on a magnetic stirrer for 3 h. After this time, the reaction mixture was decanted into a round-bottomed flask, which also contained boiling stones. The flask was placed in an oil bath, and a sublimation vessel was placed on top, connected to a vacuum. The vacuum was set to 0.1 atm, and the bath temperature to 90°C. After 15 min, boiling of the solvent ceased. The bath temperature was then raised to 130°C, and maximum vacuum was set. Ice was then added to the sublimation vessel and maintained for 15 min. Precipitation of 1,3-dehydroadamantane was observed on the surface of the sublimation vessel. The vacuum was released, the remaining ice was removed, and the sublimation vessel was transferred to another 50 ml flask containing 20 ml of DCM. The DCM was brought to a boil and the 1,3-

dehydroadamantane was washed off. Obtained 2.0 g (80% yield) as white crystalline solid. Mass spectrum, m/z ($I_{rel.}$, %): 134 (62%, $[M]^+$), 119 (50%), 105 (30%), 91 (85%), 79 (100%).

Laboratory setup for the synthesis of 1,3-dehydroadamantane (**1**) is presented in the Supplementary materials (Figure S8).

Synthesis of 1-hydroperoxyadamantane (2). The reactor was charged with 1.5 g (0.045 mol) of 90% hydrogen peroxide solution and cooled to 0-5 °C. Under argon atmosphere, 2.0 g (0.01 mol) of **1** dissolved in 10 ml of DCM were added dropwise with stirring. After mixing the reagents, the temperature was raised to room temperature and stirred for 1 hour. The solvent was removed under vacuum. Obtained 1.1 g (55% yield) as white crystalline substance. M.p. 89-91 °C. TLC in the system diethyl ether:cyclohexane = 1:1 R_f = 0.7 (H_2O_2 in this system R_f = 0.36). Under GC-MS conditions, it decomposes to 1-hydroxyadamantane. NMR 1H (DMSO- d_6), δ , ppm: 1.54-1.61 m (6H, Ad), 1.68 d (3H, J 3.0 Hz, Ad), 2.10 br.s (3H, Ad), 10.60 br.s (1H, OOH). NMR ^{13}C (DMSO- d_6), δ , ppm: 34.9 (3C), 41.2 (3C), 45.1 (3C), 82.8 (1C, C-O). HRMS-ESI m/z calculated for $[M + Na]^+ C_{10}H_{16}NaO_2^+ = 191.1043$, found 191.1040.

X-Ray Crystallography

The single-crystal X-ray data for **2** was collected using Bruker D8 Venture Photon II four-circle diffractometer (Bruker, Billerica, MA, USA) in φ and ω -scan mode (Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å) at the Center for Collective Use of the Kurnakov Institute RAS (Moscow, Russia). The raw data was indexed and integrated with the APEX3 program suite [15]. Experimental intensities were corrected for absorption effects using SADABS [16]. The crystal structure was solved by direct methods [17] and refined by the full-matrix least-squares on F^2 [18] using OLEX2 structural data visualization and analysis program suite [19]. All non-hydrogen atoms were refined with anisotropic thermal parameters. The C-H and O-H hydrogen atoms were located in difference-Fourier maps and refined isotropically. The crystallographic data and structure refinement details table for **2** were prepared using pyCIFer [20] and given in the Table S1.

Crystal data for **2**: $C_{10}H_{16}O_2$, $M = 168.23$, $P2_1$, $a = 6.4481(3)$ Å, $b = 11.5705(4)$ Å, $c = 11.9634(4)$ Å, $\beta = 90.4120(15)^\circ$, $V = 892.54(6)$ Å³, $Z = 4$, $d_{calc} = 1.252$ g/cm³. Colourless prism single crystal with dimensions $0.19 \times 0.18 \times 0.14$ mm was selected and intensities of 14961 reflections were collected ($\mu = 0.085$ mm⁻¹, $\theta_{max} = 30.517^\circ$). After merging of equivalence reflections and absorption corrections, 5432 independent reflections ($R_{int} = 0.0350$) were used for the structure solution and refinement. Final R factors $R_1 = 0.0429$ [for 4384 reflections with $F^2 > 2\sigma(F^2)$], $wR_2 = 0.0993$ (for all reflections), $S = 1.024$, and largest diff. peak and hole are 0.209 and -0.181 e/Å³, respectively.

5. Conclusions

Thus, our results demonstrate that the authors of previous studies were unable to obtain either 1-hydroperoxyadamantane or diadamantyl peroxide under the conditions they described. This finding is supported by the absence of quantitative data and by TLC analysis, which did not indicate the presence of claimed peroxide compounds. The 1H NMR signal reported by them as a broad singlet at 7.5 ppm likely corresponds to residual hydrogen peroxide. In contrast, the spectra of 1-hydroperoxyadamantane crystals, obtained by us, display a broad singlet for the hydroperoxide group proton at a chemical shift of 10.60 ppm. We have confirmed that 1-hydroperoxyadamantane can be obtained in good yields via the reaction of 1,3-dehydroadamantane with concentrated hydrogen peroxide in dichloromethane.

Supplementary Materials: The following supporting information can be downloaded at the website of this paper posted on Preprints.org, Figure S1. Chromatogram of the reaction mass containing 1,3-dehydroadamantane (95%, retention time 6.086 min) with 4% of adamantane impurity (retention time 6.607 min). Figure S2. Mass-spectra of 1,3-dehydroadamantane (**1**). Figure S3. NMR 1H of 1-hydroperoxyadamantane (**2**). Figure S4. NMR ^{13}C of 1-hydroperoxyadamantane (**2**). Figure S5. HRMS (ESI) of 1-hydroperoxyadamantane (**2**). Figure S6. NMR 1H of adamantan-1-ol (AdOH). Figure S7. NMR ^{13}C of adamantan-1-ol (AdOH). Figure S8.

Laboratory setup for the synthesis of 1,3-dehydroadamantane. Table S1. Crystallographic data and structure refinement parameters for **2**. Table S2. Selected bond lengths for **2**. Table S3. Selected angles for **2**. Table S4. Hydrogen bonds for **2** [Å and angles].

Author Contributions: Conceptualization, V.C.; investigation, I.N., D.Z., I.Y.; writing, review, and editing, I.Y., V.B.; project administration, V.B. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: The data underlying this study are available in the published article and its Supporting Information.

Accession Codes: Deposition Number 2487732 contain the supplementary crystallographic data for this paper. These data are provided free of charge by The Cambridge Crystallographic Data Centre: ccdc.cam.ac.uk/structures. The corresponding CIF files are also available as Supporting Information.

Acknowledgments: This study was carried out using the facilities of joint center at MIREA Russian Technological University.

Conflicts of Interest: The authors declare no conflicts of interest.

References

1. No, B. I.; Butov, G. M.; Mokhov, V. M.; Parshin, G. Yu. Preparation of α -Adamantyl-Substituted Aliphatic Ketones. *Russ. J. Org. Chem.* **2002**, *38* (2), 295–296. <https://doi.org/10.1023/A:1015598607917>
2. Mokhov, V. M.; Butov, G. M.; Novakov, I. A. 1,3-Dehydroadamantane and Its Derivatives: A Versatile Synthetic Platform for the Preparation of Functional Compounds with a Cage Structure. A Review. *Dokl. Chem.* **2023**, *511* (1), 149–180. <https://doi.org/10.1134/S0012500823600566>
3. Butov, G. M.; Mokhov, V. M.; Burmistrov, V. V.; Saad, K. R.; Pitushkin, D. A. Reactions of 1,3-Dehydroadamantane with Inorganic Oxygen-Free Acids. *Russ. J. Org. Chem.* **2014**, *50* (9), 1276–1278. <https://doi.org/10.1134/S1070428014090073>
4. Bitjukov, O. V.; Serdyuchenko, P. Y.; Kirillov, A. S.; Nikishin, G. I.; Vil', V. A.; Terent'ev, A. O. Advances in Radical Peroxidation with Hydroperoxides. *Beilstein J. Org. Chem.* **2024**, *20*, 2959–3006. <https://doi.org/10.3762/bjoc.20.249>
5. Targhan, H.; Evans, P.; Bahrami, K. A Review of the Role of Hydrogen Peroxide in Organic Transformations. *J. Ind. Eng. Chem.* **2021**, *104*, 295–332. <https://doi.org/10.1016/j.jiec.2021.08.024>
6. Terent'ev, A. O.; Boyarinova, K. A.; Nikishin, G. I. Oxidation of Alkenes with Hydrogen Peroxide, Catalyzed by Boron Trifluoride. Synthesis of Vicinal Methoxyalkanols. *Russ. J. Gen. Chem.* **2008**, *78* (4), 592–596. <https://doi.org/10.1134/S1070363208040130>
7. Chapurkin, V. V.; Rakhimov, A. I.; Chapurkin, S. V. Synthesis and Features of Thermolysis of Fluorine-Containing Aromatic 1-Hydroxy-1-Hydroperoxides and 1,1'-Dihydroperoxides. *Russ. J. Gen. Chem.* **2009**, *79* (2), 242–245. <https://doi.org/10.1134/S1070363209020121>
8. Rakhimov, A.I.; Volynskaya, E.M.; Chapurkin, V.V.; Alekseenko, A.N.; Ilchenko, A.Ya. On the interaction of perfluorocyclic ketones with hydrogen peroxide. *J. Org. Chem. USSR* **1985**, *21* (3), 656–657 (in Russian).
9. Barton, D. H. R.; Halley, F.; Ozbalik, N.; Schmitt, M.; Young, E.; Balavoine, G. Functionalization of Saturated Hydrocarbons. 14. Further Studies on the Mechanism of Gif-Type Systems. *J. Am. Chem. Soc.* **1989**, *111* (18), 7144–7149. <https://doi.org/10.1021/ja00200a037>
10. Mella, M.; Freccero, M.; Albini, A. Photoinduced SET for the Functionalization of Alkanes. *J. Chem. Soc., Chem. Commun.* **1995**, No. 1, 41. <https://doi.org/10.1039/C39950000041>
11. Turitsyna, E. A.; Gritsenko, O. N.; Shteinman, A. A. Effect of the Hydrogen Peroxide Concentration in Stereospecific Oxidation of Alkanes by Models of Non-Heme Oxygenases. *Kinet. Catal.* **2007**, *48* (1), 53–59. <https://doi.org/10.1134/S0023158407010089>

12. Son, V.V.; Ivashchenko, S.P.; Son, T.V. Syntheses based on 1,3-dehydroadamantane. *J. Gen. Chem. USSR (English Translation)*, **1990**, *60*, 710–711.
13. Butov, G.M.; Mokhov, V.M., Ledenev, S.M.; Terent'ev, A.O. Method of producing 1-adamantylhydroperoxide. Patent RU 2471780 C1, 25 November 2011. (In Russian).
14. Sosonyuk, S. E.; Peshich, A.; Tutushkina, A. V.; Khlevin, D. A.; Lozinskaya, N. A.; Gracheva, Y. A.; Glazunova, V. A.; Osolodkin, D. I.; Semenova, M. N.; Semenov, V. V.; Palyulin, V. A.; Proskurnina, M. V.; Shtil, A. A.; Zefirov, N. S. Synthesis and Cytotoxicity of Novel Simplified Eleutherobin Analogues as Potential Antitumour Agents. *Org. Biomol. Chem.* **2019**, *17* (10), 2792–2797. <https://doi.org/10.1039/c8ob02915f>
15. APEX3, SAINT and SADABS. Bruker AXS Inc.: Madison, WI, USA, 2016.
16. Krause, L.; Herbst-Irmer, R.; Sheldrick, G. M.; Stalke, D. Comparison of Silver and Molybdenum Microfocus X-Ray Sources for Single-Crystal Structure Determination. *J. Appl. Crystallogr.* **2015**, *48* (1), 3–10. <https://doi.org/10.1107/s1600576714022985>
17. Sheldrick, G. M. SHELXT– Integrated Space-Group and Crystal-Structure Determination. *Acta Crystallogr. A Found. Adv.* **2015**, *71* (1), 3–8. <https://doi.org/10.1107/S2053273314026370>
18. Sheldrick, G. M. Crystal Structure Refinement with SHELXL. *Acta Crystallogr. C Struct. Chem.* **2015**, *71* (1), 3–8. <https://doi.org/10.1107/S2053229614024218>
19. Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H. OLEX2: A Complete Structure Solution, Refinement and Analysis Program. *J. Appl. Crystallogr.* **2009**, *42* (2), 339–341. <https://doi.org/10.1107/s0021889808042726>
20. Chernyavskiy, D. (2026). pyCIFer - automated CIF edit and ESI-style report to .docx files (Version v2026/1) [Computer software]. <https://doi.org/10.5281/zenodo.18148748>

Disclaimer/Publisher's Note: The statements, opinions and data contained in all publications are solely those of the individual author(s) and contributor(s) and not of MDPI and/or the editor(s). MDPI and/or the editor(s) disclaim responsibility for any injury to people or property resulting from any ideas, methods, instructions or products referred to in the content.