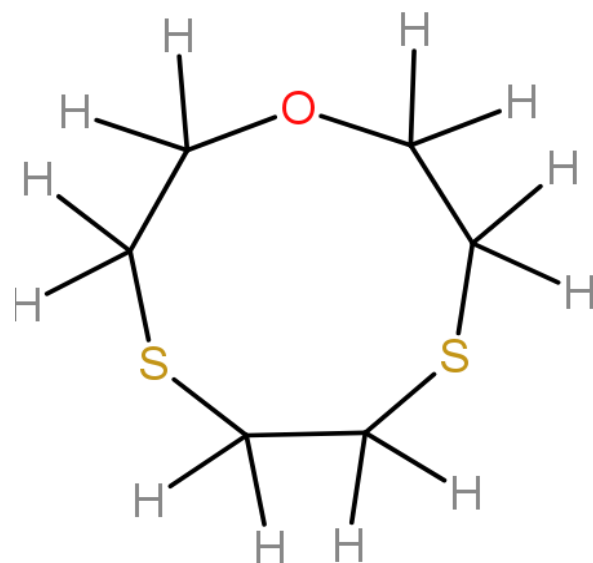




Synthesis and Analysis of 1,4,7-Oxadithionane Heterocyclic Compound



by
Hussam-Addin Ezzo

The 20th Kolmogorov Readings International Scientific School Conference
3-7 May 2020

Introduction

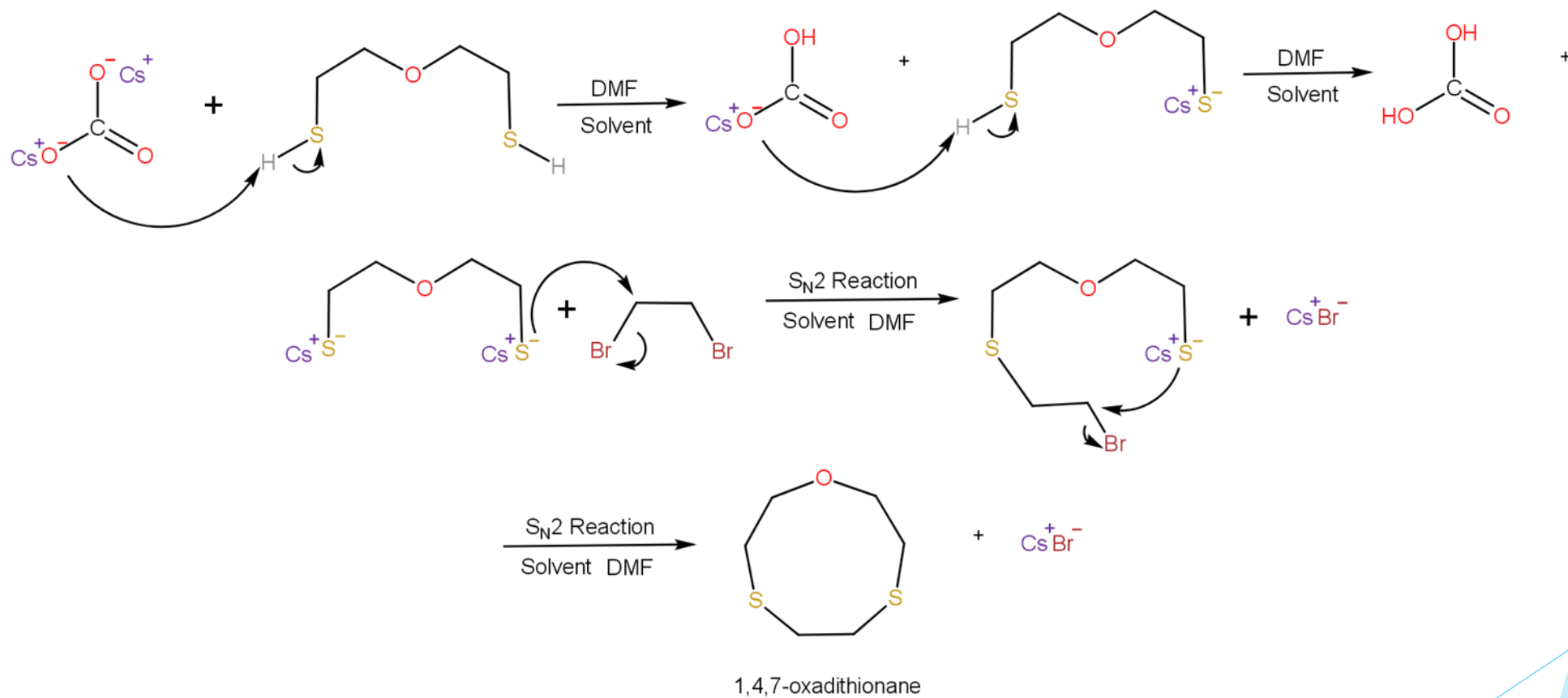
- ▶ Heterocyclic compounds have lots of applications in military, medicine and industrial.
- ▶ Heterocyclic compounds are not applied in metals ions extraction processes.
- ▶ Host-Guest complexing reaction.

The Goal of this Research

- ▶ Improving an effective pathway for synthesizing 1,4,7-Oxadithionane heterocyclic compound with a good yield.

Materials and Methods

1-Synthesis of 1,4,7-Oxadithionane



Materials and Methods

2- Analysis of Product and Equipment

2.1-Analysis with FT-IR

Transmittance spectra were taken on FT-IR spectrophotometer with a range of 400 cm^{-1} to 3150 cm^{-1}

Materials and Methods

2.2. Analysis with Proton Nuclear Magnetic Resonance.

^1H NMR spectra with a range of 0 to 12 ppm were taken on ^1H NMR Spectrophotometer (Bruker 400MHz NMR) of the product (DMF as solvent).

Materials and Methods

2.3. Analysis with ^{13}C Carbon Nuclear Magnetic Resonance.

^{13}C NMR spectra with a range of 0 to 120 ppm were taken on

^{13}C NMR Spectrophotometer (Bruker 400MHz NMR) of the product

(DMF as solvent).

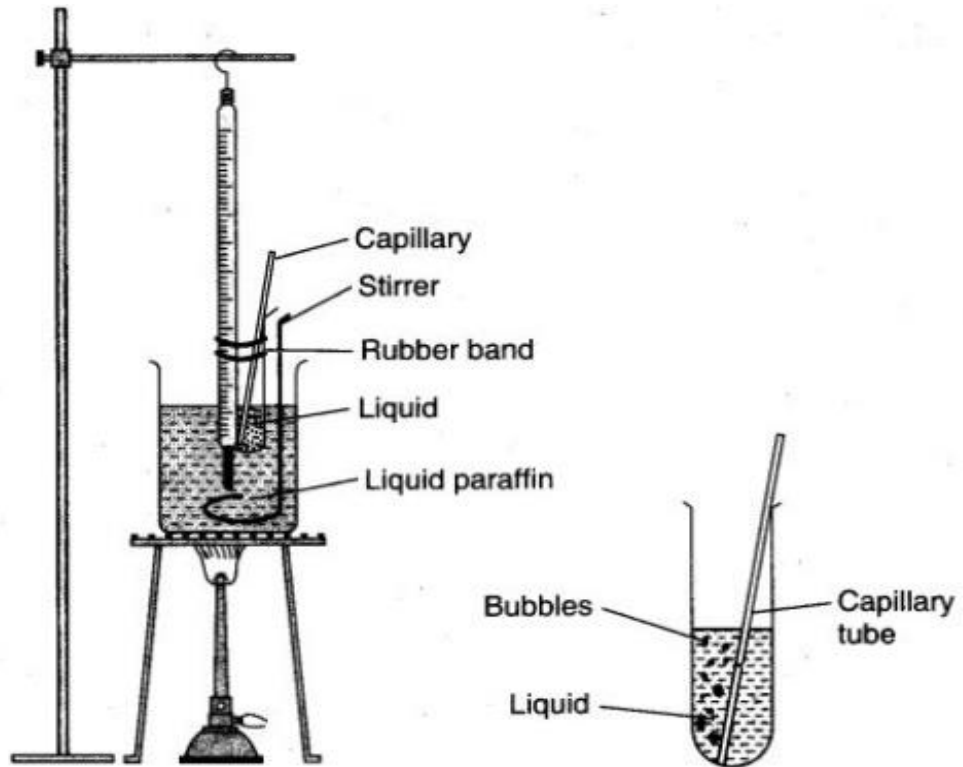
Materials and Methods

2.4. Analysis with LC-MS/MS Mass Spectrometry.

A small quantity of the produced compound was dissolved in *DMF* and directly injected into a Gemini C18 analytical column within an HP 1100 LC system interfaced to an API2000 triple-quadrupole mass spectrometer by an electrospray ionization source operated in positive ion mode.

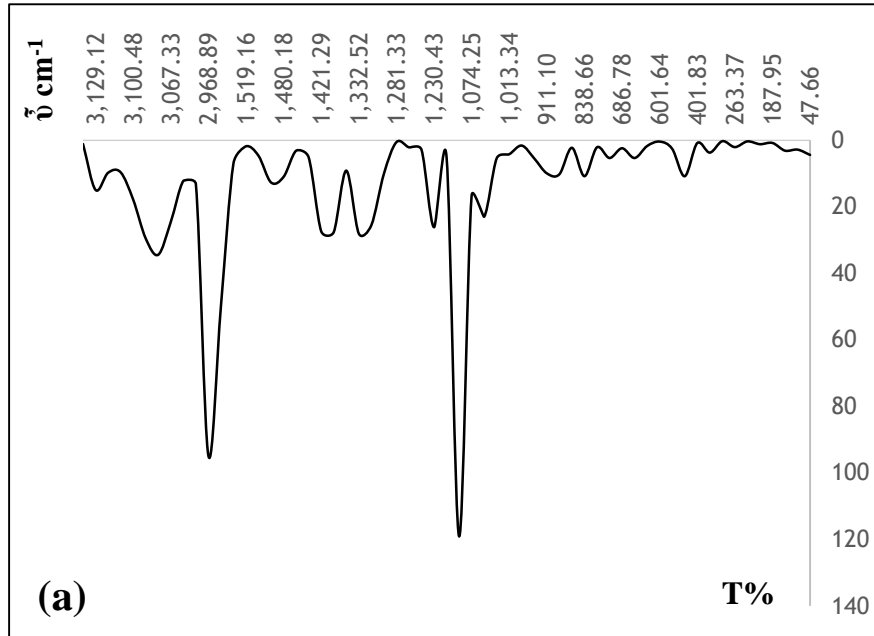
Materials and Methods

2.5. Determination of The Boiling Point.

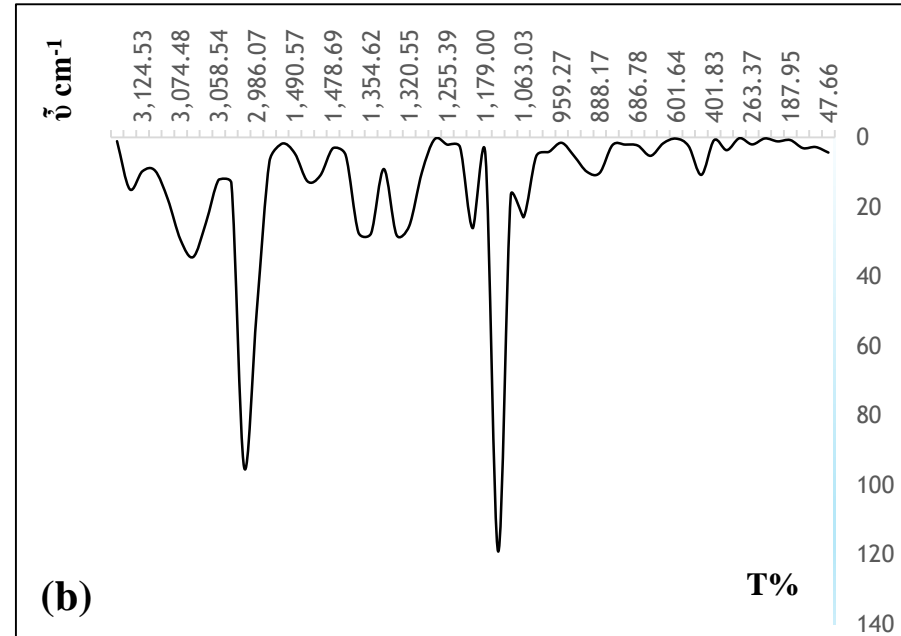


Results and Discussion

1-FT-IR Spectroscopy Analysis Result.



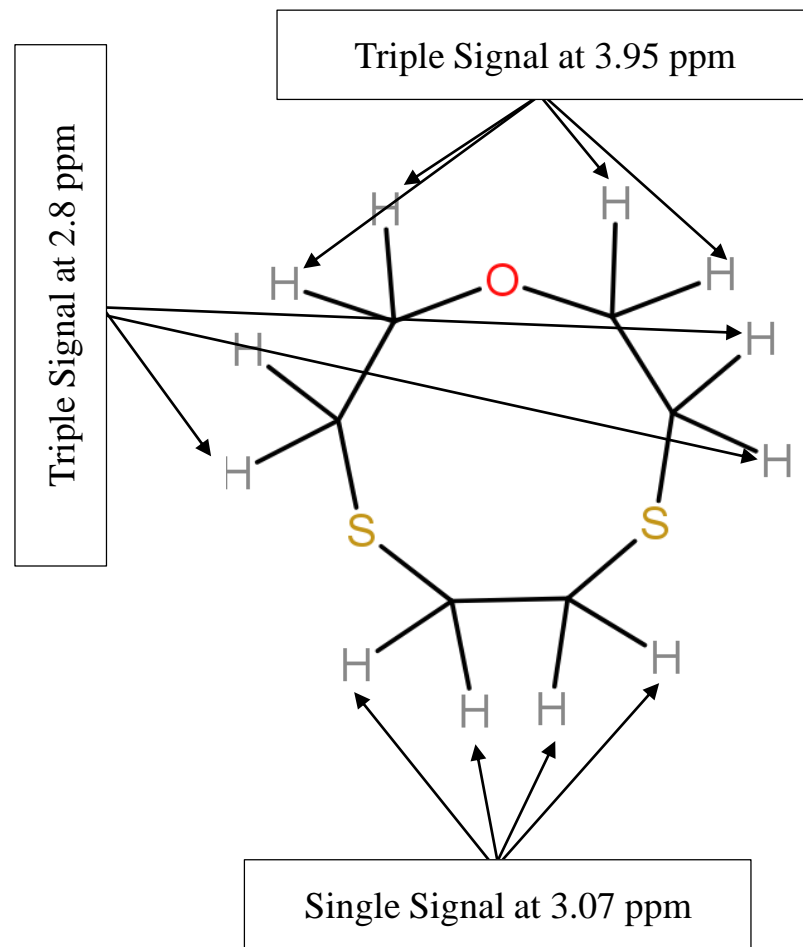
(a) IR spectrum of synthesized compound



(b) standard IR spectrum of 1,4,7-Oxadithionane.

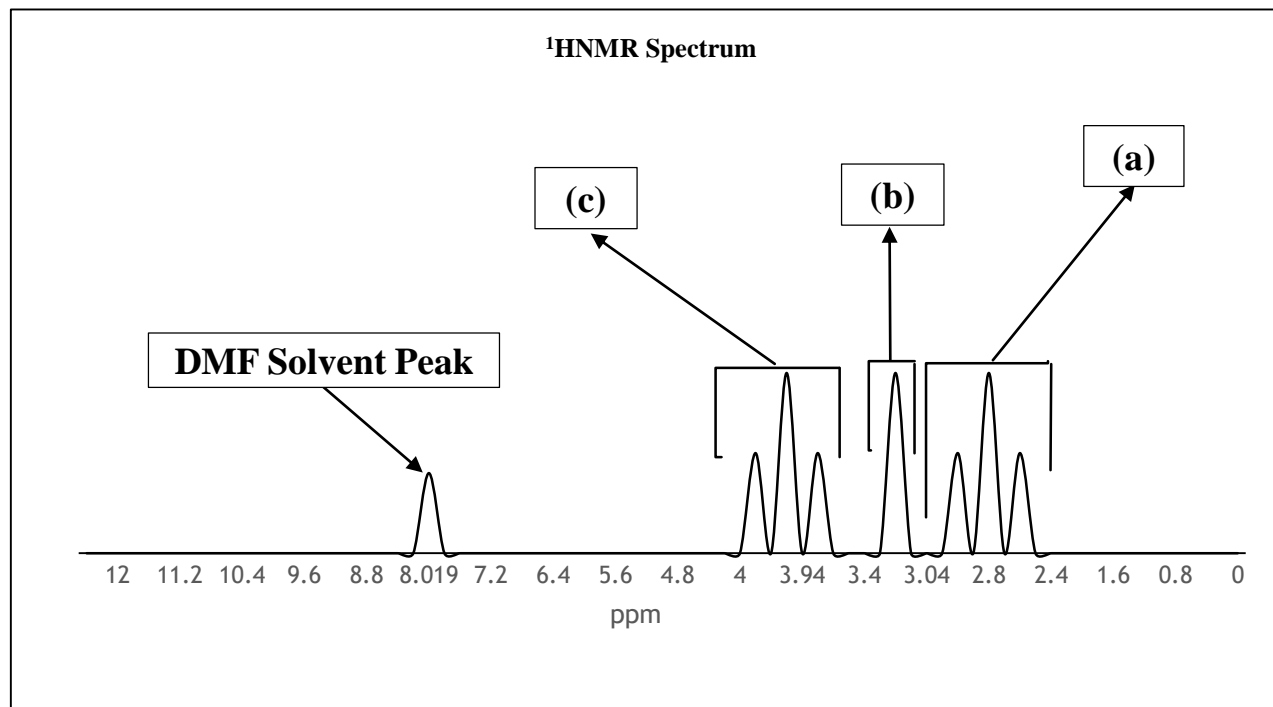
Results and Discussion

2-Expected ^1H NMR signals of the compound.



Results and Discussion

2-Proton NMR spectroscopy analysis result.



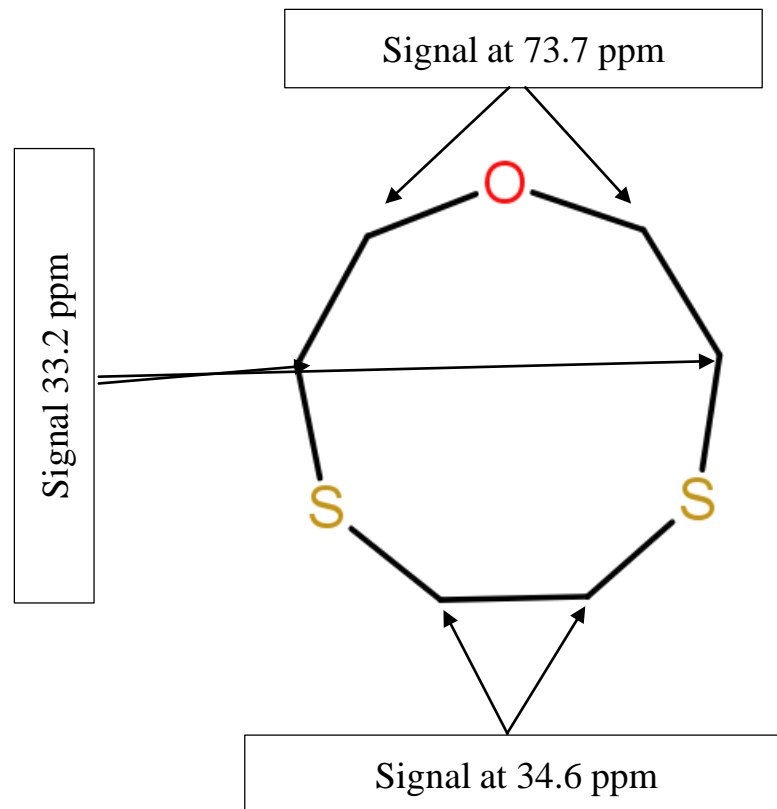
(a) triplet signal 2.8 ppm [$\text{CH}_2\text{-C}(\text{H}_2)\text{-S}$]

(b) single signal 3.07 ppm [$\text{-S-C}(\text{H}_2)\text{-C}(\text{H}_2)\text{-S-}$]

(c) triplet signal 3.95 ppm [$\text{-O-C}(\text{H}_2)\text{-CH}_2\text{-}$]

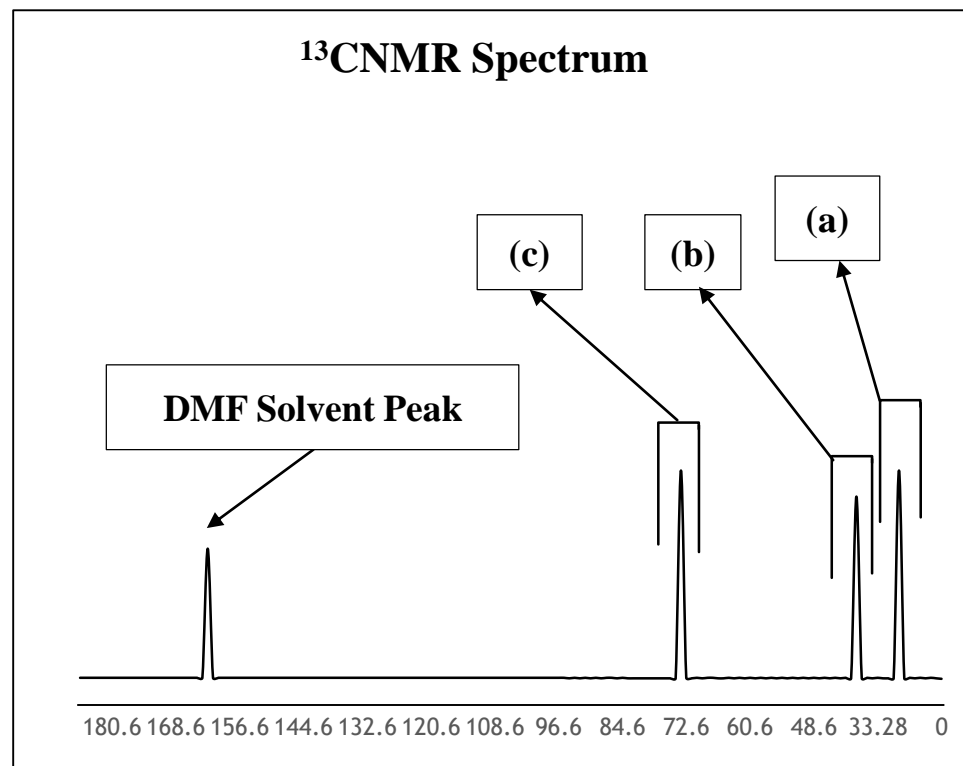
Results and Discussion

3-Expected ^{13}C NMR signals of the compound.



Results and Discussion

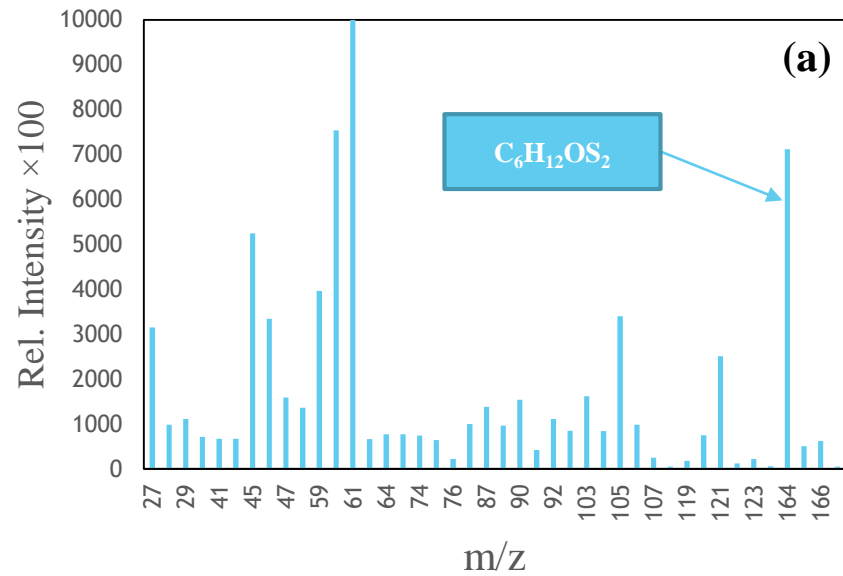
3-¹³C NMR Spectroscopy Analysis Result.



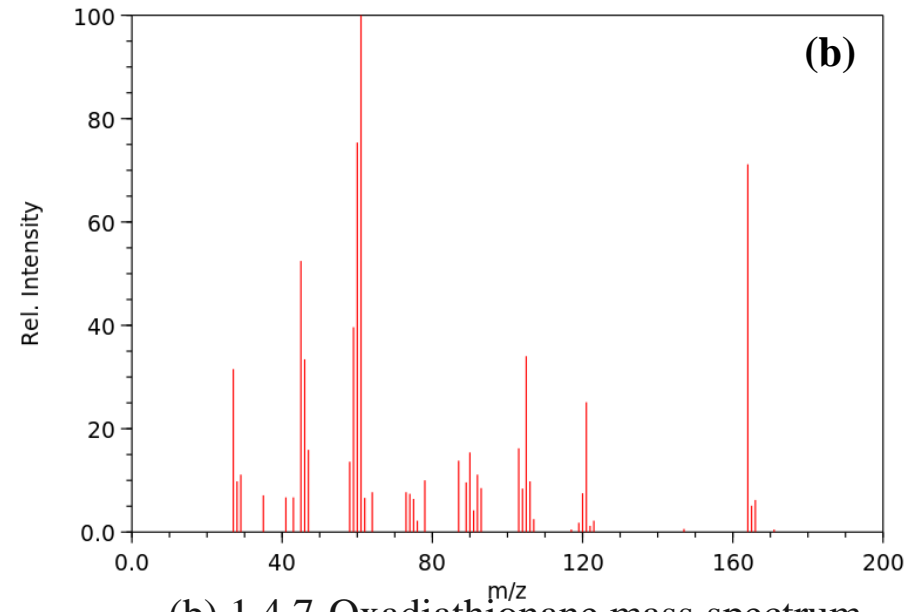
- (a) signal 33.2 ppm [S-(C)H₂-(C)H₂-S] (b) signal 34.6 ppm [(C)H₂-S-]
(c) signal 73.7 ppm [-O-(C)H₂-]

Results and Discussion

4-LC/MS Mass Spectroscopy Analysis Result.

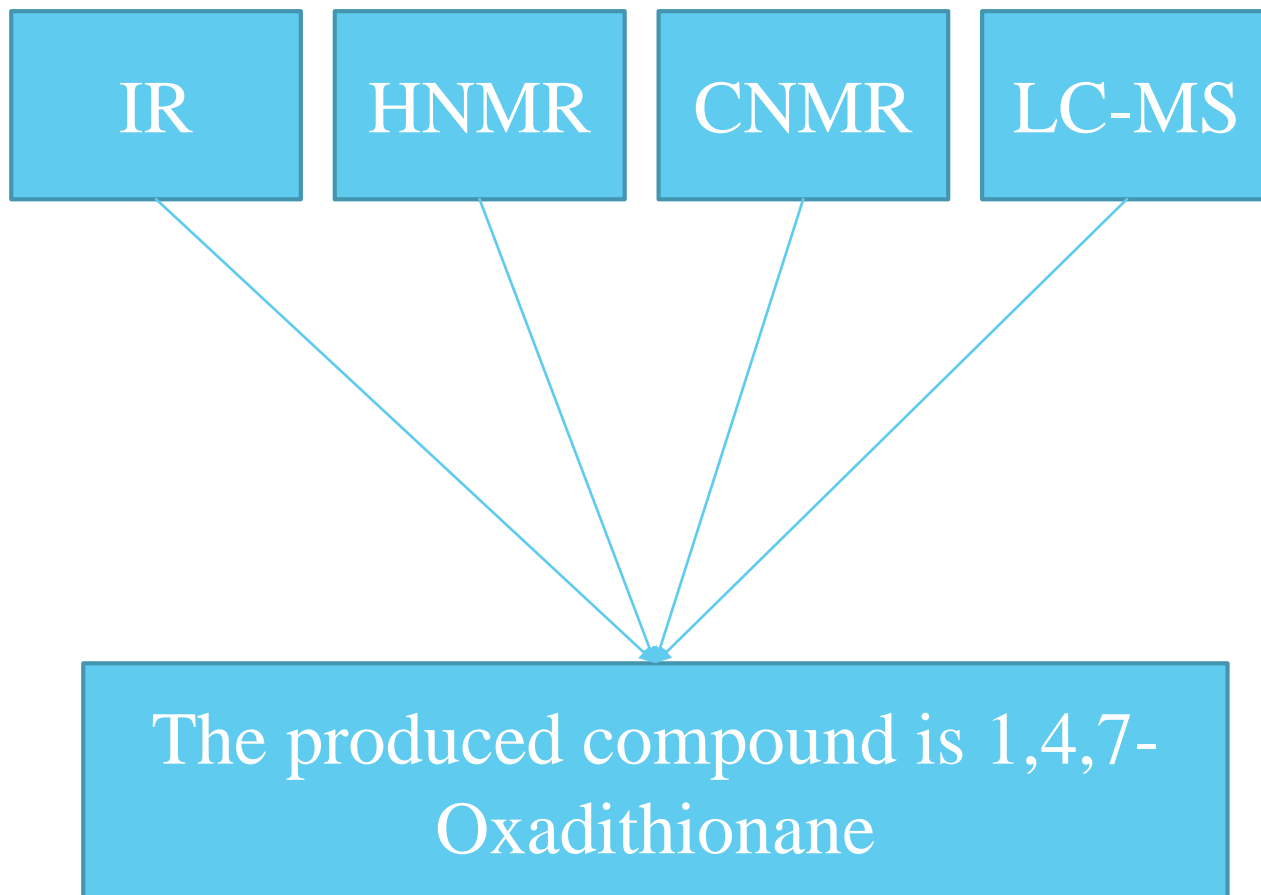


(a) Synthesized compound's mass spectrum.



(b) 1,4,7-Oxadiathionane mass spectrum.

Conclusion



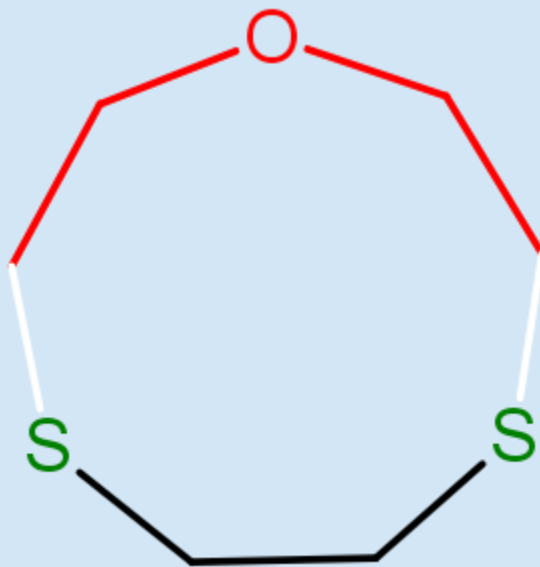
References

- ▶ Wils, Eric R. J., Gas Chromatography/Mass Spectrometry in Analysis of Chemicals Related to the Chemical Weapons Convention, John Wiley & Sons Ltd, UK, ISBN:0-470-84756-5, 2005, p. 205.
- ▶ Charles M. Marson, Chapter Two - Saturated Heterocycles with Applications in Medicinal, Academic Press, Volume 121, 2017, pp. 13-33.
- ▶ Bhuwan B. Mishra, Dhananjay Kumar, Amrita Mishra, Prabhu P. Mohapatra & Vinod K. Tiwari, Chapter 2 - Cyclo-Release Strategy in Solid-Phase Combinatorial Synthesis of Heterocyclic Skeletons, Academic Press, Volume 107, 2012, p. 42.
- ▶ Kyba, Evan P., Helgeson, Roger C., Madan et al., Host-guest complexation. 1. Concept and illustration, Journal of the American Chemical Society, 99, 8, 2002, pp. 2564-2571.
- ▶ Peter R. Griffiths & James A. de Haseth, Fourier Transform Infrared Spectrometry 2nd ed., John Wiley & Sons Ltd, Canada, 2006, p. 251.

- ▶ John McMurry, Organic Chemistry 9th ed., Cengage brain, UK, ISBN-10: 978-1-305-08048-5, 2016, pp. 386-391.
- ▶ Bruice, Paula Yurkanis, Organic Chemistry 8th ed., University of California, Santa Barbara, Pearson Education, ISBN-13: 978-0-13-404228-2, 2016, pp. 657-661.
- ▶ Jonathan Clayden, Nick Greeves & Stuart Warren, Organic Chemistry, Oxford University Press, ISBN-13: 978-0-19-927029-3, 2012, pp. 46-51.
- ▶ O'Dowd, L. & Perkin, F. Mollwo, Determination of boiling points of very small quantities of liquids, Journal of Transactions of the Faraday Society 'The Royal Society of Chemistry', 1908, pp. 95-98.
- ▶ J.S. Bradshaw, J.Y. Hui, B.L. Haymore, J.J. Christensen, R.M. Izatt, Macrocyclic Polyether Sulfide Syntheses, Journal of Heterocycle Chemistry, Vol. 10 1973, No. 1.

- ▶ G.J. Grant, D.F. Galas, M.W. Jones K.D. Loveday, W.T. Pennington, G.L. Schimek, C.T. Eagle, D.G. VanDerveer, Platinum Group Metal Complexes of Macrocyclic Oxathiaethers. The Crystal Structures of 18S4O2, [Pt(9S2O)2] (PF6)2·2CH3NO2, [Pt(18S4O2)](PF6)2, [Pd(9S2O)2](PF6)2·2CH3CN, Journal of Inorganic Chemistry, American Chemical Society, Vol. 37, 1998, pp. 5299-5305.
- ▶ F. Teixidor, A.M. Romerosa, J. Rius, C. Miravittles, J. Casabo, C. Vinas, E. Sanchez, Macrocycles incorporating closo- and nido-carbaborane cages: molecular structure of 1,2-(3'-oxapentane-1',5'-dithiolato-SS')-1',2'-dicarba-closo dodecaborane, Journal of the Chemical Society, Dalton Transactions, The Royal Society of Chemistry, 1990, pp. 525-529.
- ▶ P.J. Linstrom and W.G. Mallard, Eds., NIST Chemistry WebBook, NIST Standard Reference Database Number 69, National Institute of Standards and Technology, Gaithersburg MD, 20899..

Thanks for your listening



1,4,7-Oxadithionane