

Spectroscopic Characterization of (Z)-N-(2-Amino-1,2-Dicyanovinyl) Formimidate via Raman Technique

Jules Seh Noel Guei^{1*}, Lydia Davenport², Aschalew Kassu³, Mebougna Drabo³

¹Department of Physics, Chemistry, and Mathematics, Alabama A&M University, Huntsville, AL, USA

²Center for Educator Preparation & Certification Services, Alabama A&M University, Huntsville, AL, USA

³Department of Mechanical & Civil Engineering and Construction Management, Alabama A&M University, Huntsville, AL, USA

Email: *Jules.guei@aamu.edu

How to cite this paper: Guei, J.S.N., Davenport, L., Kassu, A. and Drabo, M. (2025) Spectroscopic Characterization of (Z)-N-(2-Amino-1,2-Dicyanovinyl) Formimidate via Raman Technique. *Journal of Materials Science and Chemical Engineering*, 13, 47-54. <https://doi.org/10.4236/msce.2025.138004>

Received: June 15, 2025

Accepted: August 17, 2025

Published: August 20, 2025

Copyright © 2025 by author(s) and Scientific Research Publishing Inc. This work is licensed under the Creative Commons Attribution International License (CC BY 4.0).

<http://creativecommons.org/licenses/by/4.0/>



Open Access

Abstract

This study employs Raman spectroscopy for the first time to characterize (Z)-N-(2-amino-1,2-dicyanovinyl) formimidate. Raman, in addition to IR and NMR (acquired and literature), helps confirm the molecular structure of the compound. Additionally, the melting point (mp 132°C - 134°C) data helps confirm the compound's purity. Raman spectra, measured with a 785 nm laser, reveal key vibrational modes, including two peaks at 2242 and 2206 cm⁻¹ (C≡N stretches), two peaks at 1634 and 1591 cm⁻¹ (C=N and C=C stretches), two peaks at 1369 and 1224 cm⁻¹ attributed to (C-O and N-H bands). The acquired FT-IR spectra showed characteristic peaks at 3416, 3304, 2243, 2208, 1635, 1606 cm⁻¹ as reported by IR data of Alves *et al* (1990) [1] and US Patent #US 8,518,901 B2 (2013) [2] at 3309 cm⁻¹ (NH/NH₂ stretches), 2247 cm⁻¹ (C≡N stretches), 2207 cm⁻¹ (CN stretches)~1636 cm⁻¹ (C=N stretches), 1608, 1256 (C-O str.), 810 cm⁻¹ supporting the presence of cyano, formimidate, and amino groups. These findings validated the compound's identity and highlighted the complementary nature of Raman and IR techniques for formimidate analysis.

Keywords

Formimidate, Raman Spectroscopy, IR Spectroscopy, Cyano, Amino Groups

1. Introduction

(Z)-N-(2-amino-1,2-dicyanovinyl) formimidate is a compound of interest due to its potential applications in thin film technologies, such as sensors or electronic materials [3] [4]. Its molecular structure, featuring cyano (C≡N), formimidate

tional signatures, making them ideal candidates for spectroscopic analysis. The $C\equiv N$ stretch typically appears in the 2200 - 2300 cm^{-1} range in both Raman and IR spectra, while $C=N$ stretches are observed around 1600 - 1700 cm^{-1} [6]. Recent studies, such as Monika *et al.* (2020), have employed density functional theory (DFT) to assign vibrational modes in dicyano aromatic conjugated molecules, enhancing the precision of spectral interpretations and validating experimental findings [10].

Computational methods, particularly DFT, have become indispensable in vibrational spectroscopy, enabling the prediction of vibrational frequencies and intensities to support experimental peak assignments [11]. Advances in computational chemistry allow for the simulation of vibrational spectra in complex systems, such as thin films, providing deeper insights into molecular behavior and interactions [11]. This study builds upon the foundational work of [5], using Raman and IR spectroscopy to characterize (Z)-N-(2-amino-1,2-dicyanovinyl) formimidate with potential computational support to refine spectral assignments.

3. Materials and Experimental Methods

Synthesis of Ethyl (Z)-N-(2-Amino-1,2-dicyanovinyl) formimidate

The titled compound was prepared according to the procedures outlined in our previously published [5] following the modified original procedure of Alves *et al.* [1] and a US patent #US 8,518,901 B2 (2013) [2]. To a solution of commercially available diaminomaleonitrile (3.24 g, 30.1 mmol) in dioxane (50 mL), triethyl orthoformate (4.5 mL, 30.4 mmol) was added. The mixture was heated at reflux for 30 min in a flask fitted with a distillation head, a condenser, and a receiver until 30 mL of ethanol mixed with dioxane was collected. The brown mixture in the distillation flask was cooled overnight at room temperature. Hexanes (50 mL) were added, the flask was swirled to a homogeneous solution, cooled in a cold bath at -13°C for 30 min, and Ether (30 mL) was added. The flask was warmed to 40°C for 10 min. The hot ether solution was filtered and concentrated to yield the titled compound (3.5 g, 21 mmol, 70%). mp $132 - 134^{\circ}\text{C}$ (lit.⁽⁷⁾ $132 - 135^{\circ}\text{C}$); ^1H NMR (CDCl_3) δ 1.26 (t, 3H, CH_3), 4.15 (q, 2H, CH_2), 4.94 (br s, 2H, NH_2 , exchangeable with D_2O), 7.99 (s, 1H, CH). ^{13}C NMR (CDCl_3) δ 36.4, 56.64, 75.59, 77.0, 78.3, 78.4, 144.2.

Sample Preparation

The samples were prepared as a solid compound according to the modified synthesis protocol by Guei *et al.* [5]. The compound was synthesized, and the structure was confirmed via ^1H and ^{13}C NMR, melting point (mp), IR spectroscopy as detailed in the references [1] [2] [5] and Raman spectroscopy.

Raman and IR Spectroscopy

Raman spectra were acquired using a 100 mW, 785 nm laser source with a 60-second integration time and 2-average scans. In addition, the Raman system used a spectral resolution of 6 cm^{-1} and a fiber-coupled laser output of 100 microns, with

a laser power of 100 mW at a working distance of 7 mm. The spectral coverage of the system ranges from 250 to over 2350 cm^{-1} capturing the key vibrational modes of the compound. Hence, the results of this work focus on the characteristic Raman bands of the sample observed within these ranges. Additional details of the Raman system used and the experimental setup are discussed elsewhere [12] [13].

FT-IR spectral data were acquired, and from Alves *et al.* (1990) [1] and US Patent #US 8,518,901 B2 (2013) [2], which report absorption bands characteristic of the compound's functional groups.

4. Results and Discussion

Synthesis, NMR, and Melting Point

The *Ethyl (Z)-N-(2-amino-1,2-dicyanovinyl) formimidate* (Scheme 1) was synthesized according to a modified procedure of Guei *et al.* [5], following the original procedure of Alves *et al.* [1] and the US Patent #US 8,518,901 B2 [2]. It was characterized by melting point and NMR spectroscopy (see methods). Starting with commercially available diaminomaleonitrile and triethylorthoformate, the titled compound was synthesized in a moderate yield (3.5 g, 21 mmol, 70%), with a melting point of 132 - 134 °C (lit.⁽⁷⁾ 132 - 135 °C).

Raman and IR Spectroscopy of Ethyl (Z)-N-(2-amino-1,2-dicyanovinyl) formimidate Data

Raman and IR spectroscopy provide complementary insights due to their sensitivity to different vibrational modes. Raman spectroscopy excels at detecting symmetric stretches, such as the $\text{C}\equiv\text{N}$ stretch at 2208 cm^{-1} , while IR is more sensitive to polar modes, like the NH/NH_2 stretches at 3200–3400 cm^{-1} . The combination of these techniques ensures a comprehensive characterization of the compound's functional groups, confirming its molecular structure. In this study, the Raman bands assignment and the characterization of the compound are primarily based on the acquired data and the literature values [14] [15].

Raman spectra of Ethyl (Z)-N-(2-amino-1,2-dicyanovinyl) formimidate Data

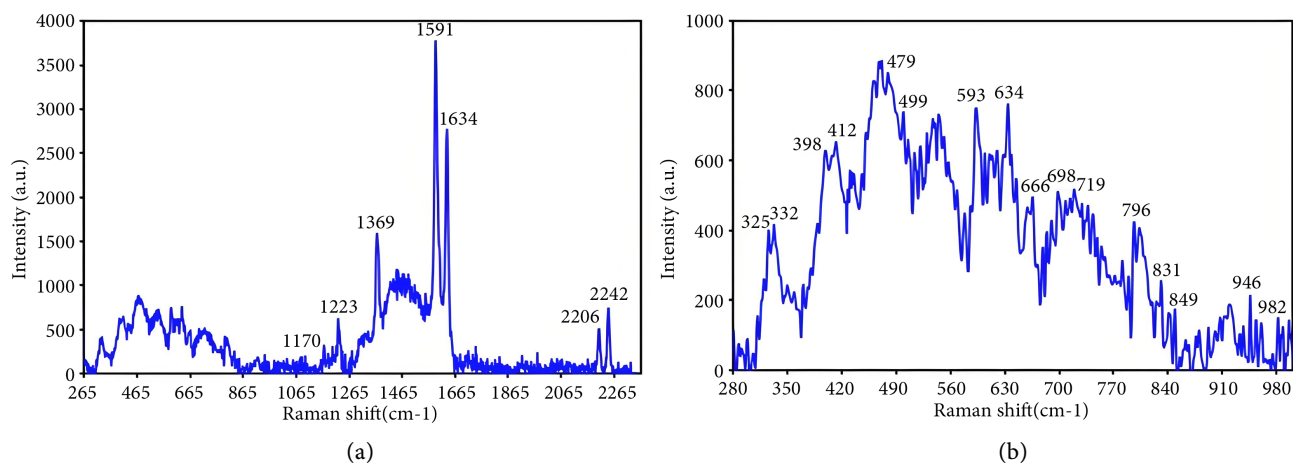


Figure 1. (a) Raman spectra of Ethyl (Z)-N-(2-amino-1,2-dicyanovinyl) formimidate captured in the 265 - 2365 cm^{-1} region; (b) Raman spectra of Ethyl (Z)-N-(2-amino-1,2-dicyanovinyl) formimidate captured in the 280 - 1000 cm^{-1} region.

Table 1. Major Raman peaks for the compound and band assignments.

Wavenumber (cm ⁻¹)	Assignment
1224	C-O and N-H
1370	C-O and N-H
1591	C=N and C=C
1633	C=N and C=C
2205	C≡N
2243	C≡N

The peaks at 1223 and 1369 cm⁻¹ are assigned to C-O and N-H consistent with the formimidate group. The higher intensity peaks in the compound suggest a possible crystalline structure. The strong peaks at 1591 and 1634 cm⁻¹ are assigned to the C=N and C=C stretching vibration, consistent with the conjugate system. This assignment aligns with literature values for C=N stretches in conjugated systems, typically observed in the 1475 - 1700 cm⁻¹ range [15]. The peak at 2205 and 2243 cm⁻¹ corresponds to the C≡N stretching vibration, typical for cyano groups in organic nitriles, where the C≡N stretch appears around 2200 - 2300 cm⁻¹ (Smith, 2019 [11]). This peak's consistency across both samples supports the presence of cyano groups.

Figure 1(a) shows the Raman measurement of the Ethyl (Z)-N-(2-amino-1,2-dicyanovinyl) formimidate sample observed within the spectral coverage of the system. As shown in the figure, the Raman bands located at 1223, 1369, 1591, 1634, 2206, and 2242 cm⁻¹ are very distinct as compared with the other multiple weak bands. The Raman band assignments of these major bands corresponding to the vibrational modes of the compound's functional groups are summarized in **Table 1**. To enhance the resolution of the weak bands captured, the region from 280 - 1000 cm⁻¹ is separately plotted (**Figure 1(b)**).

As mentioned above, the Raman spectroscopic characterization of the Ethyl (Z)-N-(2-amino-1,2-dicyanovinyl) formimidate primarily focuses on the Raman bands observed within the 250 to 2350 cm⁻¹ region. The result revealed several weak, medium, and prominent bands (**Figure 1(a)** and **Figure 1(b)**), indicating the vibrational modes of the functional groups. In general, ethyl functional groups symmetric CH₃ deformation vibrations, CH₂ twisting vibrations, and CH₂ rocking vibrational Raman bands are located in the 1360 - 1390 cm⁻¹, 1200 - 1290 cm⁻¹, and 1060 - 1190 cm⁻¹ regions, respectively [14]. Accordingly, the peak observed at 1369 cm⁻¹ is assigned to symmetric CH₃ deformation vibration, which is within the typical region for ethyl functional groups. The Raman peak observed at 1223 cm⁻¹ indicates the CH₂ wagging vibrations in the formimidate sample, which is within the most common spectral region (1215 - 1285 cm⁻¹) for the wagging vibration mode of this functional group.

In addition, the bands within the 880 - 1000 cm⁻¹ region exhibit rocking vibration of the CH₃ functional group in the compound. The results of the Raman

measurement show weak Raman peaks located at 880 and 996 cm^{-1} , indicating the presence of this mode of vibration of the functional group in the compound. The weak bands in the region 575 - 730 cm^{-1} are due to the C-H stretching vibrations. Hence, the weak bands shown in **Figure 1**, captured at 593, 634, 666, 698, and 719 cm^{-1} , exhibit the C-H stretching vibrations in the compound. The C-H in-plane deformations are characterized by the bands located at 996 and 1223 cm^{-1} , which are within the 990 - 1290 cm^{-1} region common to both the in-plane deformation of C-H and C-O bond stretching vibrations.

The C=C stretching vibrations commonly take place in the 1430 - 1625 cm^{-1} region. Hence, the sharp and strong Raman peak at 1591 cm^{-1} is assigned to the stretching vibration of this bond in the formimidate compound. The band at 1369 cm^{-1} , which overlaps with the CH deformations, and the weak band at 996 cm^{-1} are also characteristic stretching vibrations of the double bond. Similarly, the weak Raman peak located at 1170 cm^{-1} is the characteristic C-N stretching band of amines, and the bands at 2206 and 2242 cm^{-1} are assigned to $\text{C}\equiv\text{N}$ stretching vibration of the bond in the formimidate structure, which is within the common region for this bond (2000 - 2250 cm^{-1}). The weak Raman peak located at 398 cm^{-1} is assigned to C-C \equiv N deformation vibration, which commonly exhibit peaks in the 340 - 390 cm^{-1} region, and the weak bands at 412 and 471 cm^{-1} are assigned to the in-plane bending of the C-CN bond in the compound, which is generally in the 380 - 430 cm^{-1} region of the spectrum.

FT-IR (Fourier-Transform Infrared Spectroscopy) Data

The acquired FT-IR spectra on a *Nicolet Is10* Fourier-Transform Infrared Spectroscopy (FTIR) showed major peaks at 3416, 3304, 2243, 2208, 1635, 1606 cm^{-1} which were corroborated by the literature data [2] IR (KBr) at 3309 cm^{-1} (N-H stretches), 2247 cm^{-1} ($\text{C}\equiv\text{N}$ stretches), 2207 cm^{-1} ($\text{C}\equiv\text{N}$ stretches), and ~1636 cm^{-1} (C=N stretches), 1608, 1256 cm^{-1} (C-O stretches), 810 cm^{-1} . These bands confirm the presence of amino, cyano, and formimidate functional groups, complementing the Raman findings. The $\text{C}\equiv\text{N}$ stretch, being a symmetric vibration, is strong in Raman but also detectable in IR, while the NH/NH₂ stretches are more prominent in IR due to their polar nature.

5. Conclusion

Raman spectroscopy was employed in this study to confirm the chemical structure of (Z)-N-(2-amino-1,2-dicyanovinyl) formimidate. The Raman spectra of the formimidate, acquired for the first time in this study, were consistent with the vibrational modes of the functional group associated with the compound as reported in the literature. The acquired FT-IR data, and the literature [2] support these findings, highlighting the presence of key functional groups. The complementary nature of Raman and IR techniques enhances the reliability of the characterization. The assignments of Raman peaks were validated through comparison with literature data and complementary IR spectra [1] [2] [14]. The C=N and $\text{C}\equiv\text{N}$ stretches are consistent with expected frequencies for conjugated systems

and nitriles, respectively [14] [15]. Future work could involve computational modeling, such as density functional theory (DFT), to refine peak assignments and explore the effects of structural variations on spectral features.

Acknowledgement

The authors gratefully acknowledge the support of Alabama A&M University for providing laboratory facilities and resources essential for this research. Special thanks are extended to the Department of Mechanical & Civil Engineering and Construction Management, the Department of Physics, Chemistry, and Mathematics, and the Center for Educator Preparation & Certification Services at Alabama A&M University for their continued encouragement and support.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

References

- [1] Alves, M.J., Booth, B.L. and Proenç, M.F.J.R.P. (1990) Synthesis of 5-Amino-4-(Cyanoformimidoyl)-1h-Imidazole: A Reactive Intermediate for the Synthesis of 6-Carbamoyl-1,2-Dihydropurines and 6-Carbamoyl purines. *Journal of the Chemical Society, Perkin Transactions 1*, No. 6, 1705-1712. <https://doi.org/10.1039/p19900001705>
- [2] Hosmane, R.S., Raman, V. and Kumar, R. (2013) Fused Diimidazodiazepine Compounds and Methods of Use and Manufacture Thereof. Google Patents: 2013, US Patent: 8,518,901 B2.
- [3] Amna, B. and Ozturk, T. (2025) Organic Field-Effect Transistor-Based Sensors: Recent Progress, Challenges and Future Outlook. *Journal of Materials Chemistry C*, **13**, 8354-8424. <https://doi.org/10.1039/d4tc04265d>
- [4] Yang, J., Zhao, Z., Wang, S., Guo, Y. and Liu, Y. (2018) Insight into High-Performance Conjugated Polymers for Organic Field-Effect Transistors. *Chem*, **4**, 2748-2785. <https://doi.org/10.1016/j.chempr.2018.08.005>
- [5] Guei, J. and Hosmane, R.S. (2005) Synthesis of a Novel Tricyclic Nucleobase Analogue Containing a Diimidazodiazepine Nucleus with Potential Selectivity. Abstracts of Papers, *230th ACS National Meeting*, Washington, DC, American Chemical Society, 28 August-1 September 2005.
- [6] Alves, M.J., Booth, B.L., Freitas, A.P. and Proença, M.F.J.R.P. (1992) (z)-N⁶-(2-Amino-1,2-Dicyanovinyl)Formamidrazone: A Precursor in the Synthesis of 1,5-Diaminoimidazoles and 6-Carbamoyl-1,2-Dihydropurines. *Journal of the Chemical Society, Perkin Transactions 1*, No. 7, 913-917. <https://doi.org/10.1039/p19920000913>
- [7] Booth, B.L., Costa, F.A.T., Mahmood, Z., Pritchard, R.G. and Proença, M.F. (1999) Synthesis of (Z)-N-(2-Amino-1,2-Dicyanovinyl)Formamide O-Alkyloximes and a Study of Their Cyclization in the Presence of Base. *Journal of the Chemical Society, Perkin Transactions 1*, No. 13, 1853-1858. <https://doi.org/10.1039/a901332f>
- [8] Larkin, P. (2011) *Infrared and Raman Spectroscopy: Principles and Spectral Interpretation*. Elsevier.
- [9] Furukawa, Y., Seto, K., Nakajima, K., Itoh, Y., Eguchi, J., Sugiyama, T., *et al.* (2012) Infrared and Raman Spectroscopy of Organic Thin Films Used for Electronic Devices. *Vibrational Spectroscopy*, **60**, 5-9.

- <https://doi.org/10.1016/j.vibspec.2011.10.012>
- [10] Monika, Inaoka, S., Iwata, K. and Saha, S. (2020) Towards the Critical Understanding of Selected Vibrational Features in Biologically Important Dicyano Aromatic Conjugated Molecules: Importance of Electron Donating/Withdrawal Groups and Geometry Associated with Dicyano Group. *Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy*, **224**, Article ID: 117419. <https://doi.org/10.1016/j.saa.2019.117419>
- [11] Smith, B.C. (2019) Organic Nitrogen Compounds IV: Nitriles. *Spectroscopy*, **34**, 18-21, 44. <https://www.spectroscopyonline.com/view/organic-nitrogen-compounds-iv-nitriles>
- [12] Farley, C., Kassu, A., Bose, N., Jackson-Davis, A., Boateng, J., Ruffin, P., *et al.* (2016) Short Distance Standoff Raman Detection of Extra Virgin Olive Oil Adulterated with Canola and Grapeseed Oils. *Applied Spectroscopy*, **71**, 1340-1347. <https://doi.org/10.1177/0003702816681796>
- [13] Robinson, P.D., Kassu, A., Sharma, A., Kukhtareva, T., Farley, C.W., Smith, C., *et al.* (2013) Surface-Enhanced Raman Spectroscopy Scattering from Gold-Coated Ceramic Nanopore Substrates: Effect of Nanopore Size. *Journal of Nanophotonics*, **7**, Article ID: 073592. <https://doi.org/10.1117/1.jnp.7.073592>
- [14] Socrates, G. (2001) *Infrared and Raman Characteristic Group Frequencies*. 3rd Edition, John Wiley.
- [15] Silverstein, R.M., Webster, F.X. and Kiemle, D.J. (2005) *Spectrometric Identification of Organic Compounds*. 7th Edition, John Wiley & Sons.