

## Synthesis and Study of the Crystal Structure of 2-[(Dipyrrolidin-1-yl) methylene] malononitrile



Wedad M. Al-Adiwish\*, Wedad M. Barag and Mariam S. Saleh

*Department of Chemistry, Faculty of Science, Zawia University, Libya*

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**Abstract:** This study aims to synthesis 2-[(dipyrrolidin-1-yl)methylene] malononitrile 2 and identify its crystal structure by X-ray diffraction analysis. 2-[(dipyrrolidin-1-yl)methylene] malononitrile was prepared by a direct displacement of the methylthio group (SMe) in the 2-[bis(methylthio)methylene] malononitrile 1 with pyrrolidine as cyclic secondary amine by conjugating addition-elimination reaction under reflux conditions for two hours. The compound was obtained in high yield (80%). The structure of compound 2-[(dipyrrolidin-1-yl)methylene] malononitrile 2 was identified by performing X-ray diffraction analysis. Suitable crystals of compound 2 were grown by slow evaporation of methanol solution of the compound. The compound 2 crystallized in an orthorhombic crystal system with a space group of *Pbcn*. In the title compound, the two cyanide groups and the two pyrrolidine rings adopted *trans* configurations across the C2=C3 bond. The bond lengths and angles of the two pyrrolidinyl rings in the compound are within the normal range. The maximum deviation of N5/C2/C3/C4/N5<sup>a</sup>/C4<sup>a</sup> is 0.002(1) around C4, and no deviation has been recorded for the fragment N1/N1<sup>a</sup>/C2/C3 (0.000 (1)°). The dihedral angle between the pyrrolidine ring and N1/N1<sup>a</sup>/C2/C3 is 33.06(8)°, and the dihedral angle between the pyrrolidine ring and N5/C2/C3/C4/N5<sup>a</sup>/C4<sup>a</sup> is 50.57(7)°. The crystal packing is stabilized by two intermolecular and one intramolecular C---H...N hydrogen bonds, which form a one-dimensional polymeric chain along the axis.

**Keywords:** single-crystal X-ray study; crystal structure; orthorhombic crystal system; direct displacement; R factor = 0.040, wR = 0.110.

### INTRODUCTION

Ketene-*N,S*- and -*N,N*-acetals, which are a group of compounds derived from enamines and belong to the acetal chitin family, are very reactive and electronically rich. Ketene acetals are of interest not only because of their unique electronic properties but also for their importance as versatile building blocks in organic synthesis. According to the number and types of compounds, keten *N,S*-acetal is the biggest family in the world of ketene

acetals (Zhang et al., 2016). Having the structural feature of ketene *S,S*-cetals and enamines make ketene *N,S*-acetals versatile and easy to use, particularly in multicomponent and cyclization reactions for the synthesis of various heterocyclic systems and related natural products (Al-Adiwish et al., 2019; Al-Adiwish et al., 2013; Gouda et al., 2010; Khalil et al., 2009; Misra et al., 2007; Zhang et al., 2016). In addition, they; are used as intermediates in dye and are often used in pharmaceutical industries (Al-Adiwish et al.,

\*Corresponding author: Wedad M. Al-Adiwish [w.aladiwish@zu.edu.ly](mailto:w.aladiwish@zu.edu.ly) Department of Chemistry, Faculty of Science, Zawia University, Zawia, Libya.

2019; Al-Adiwish et al., 2013; Al-Afaleq, 2001; El-Saghier et al., 2008; Elgemeie et al., 2007; Gouda et al., 2010; Khalil et al., 2009; Misra et al., 2007). Due to the push-pull alkene skeleton, they are useful reactants and synthetic building blocks for other highly functionalized molecules (Zhang et al., 2016). In spite of the characteristics of ketene acetals and the increasing wealth of information about their synthesis and synthetic applications, there are limited routes to their formation as isolable products (Paris, Schwartz, Sundall, et al., 2021; Paris, Schwartz, & Willand-Charnley, 2021; Zhang et al., 2016). The  $\alpha$ -Cyanoketene-*N,N*-acetals are generally prepared by the direct amination reaction of  $\alpha$ -cyanoketene-*S,S*- and *N,S*-acetals with appropriate amine (Elgemeie, Ali, et al., 2003; Elgemeie, Elghandour, et al., 2003; Elgemeie et al., 2004; Elgemeie et al., 2007; Ma et al., 2009; Sommen et al., 2003; Suryawanshi et al., 2007).

Therefore, we report here an easy and efficient method to prepare 2-[(dipyrrolidin-1-yl)methylene] malononitrile 2 by direct displacement of the methylthio group (SMe) in the 2-[bis (methylthio) methylene] malononitrile 1 with pyrrolidine by conjugating addition-elimination reaction.

## MATERIALS AND METHODS

All chemicals were of reagent grade. In this study, X-ray diffraction was used. CRYSTAL, SIR92, CAMERON, and ORTEP2 software were used for molecular drawing and preparing the crystallographic data. The following symbols are used; 2-[bis (methylthio) methylene] malononitrile is compound 1; 2-[(dipyrrolidin-1-yl) methylene] malononitrile is compound 2.

**Synthesis of 2-[(dipyrrolidin-1-yl)methylene] malononitrile 2:** Reflux of 2-[bis (methylthio) methylene] malononitrile 1 (1.93 g, 0.01 mol) with pyrrolidine (1.50 ml, 0.02 mol) in (20 ml) of ethanol for 2 hours (Al-Adiwish et al., 2013). The solvent was evaporated in a

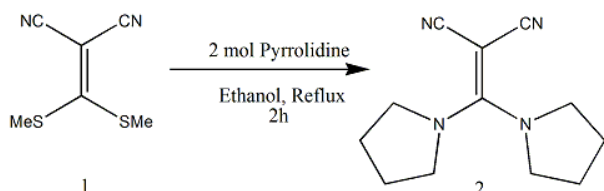
vacuum to obtain a solid crude yellow, which was collected and purified using methanol, and the percentage yield was 80%. Suitable crystals of compound 2 were grown by slow evaporation of the methanolic solution of the compound. The melting point of compound 2 was determined using a hot stage Gallenkamp melting point apparatus, which was 166-168 °C.

**Crystal structure determination:** The data were collected at room temperature with the Bruker SMART APEX CCD spectrometer. The crystal of compound 2 was placed in the cold stream of an Oxford Cryosystems open-flow nitrogen cryostat with a nominal stability of 0.1K. TLC analysis was carried out on silica gel of Merck No. 5545 to monitor the completion of reactions, in which ethyl acetate-methanol (4:1) was used as eluent. The software used for the direct method, least-squares F<sup>2</sup> analysis, molecular drawing, and preparing the crystallographic materials are CRYSTAL, SIR92, CAMERON, and ORTEP2. For further information, see references (Allen et al., 1987; Altomare et al., 1994; Betteridge et al., 2003; Pro, 2010; Watkin et al., 1996).

## RESULT AND DISCUSSION

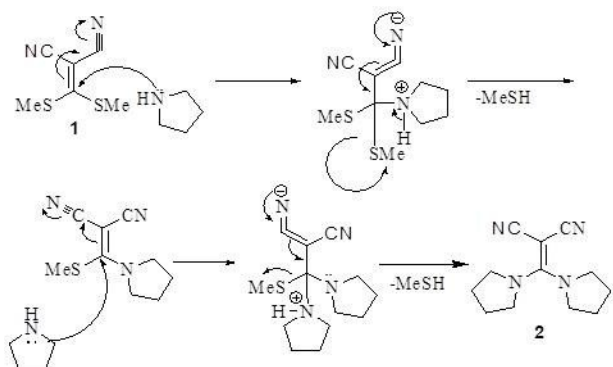
**Synthesis:** The  $\alpha$ -Cyanoketene-*N,N*-acetals are generally prepared by the direct amination reaction of  $\alpha$ -cyanoketene-*S,S*- and *N,S*-acetals with appropriate amine. The crystal structure of 2-[(dipyrrolidin-1-yl)methylene] malononitrile 2, is reported here, which was synthesized by direct displacement of the methylthio group (SMe) in the 2-[bis (methylthio) methylene] malononitrile 1 with cyclic secondary aliphatic amine as pyrrolidine by conjugating addition-elimination reaction in refluxing ethanol for 2 h. The solvent was evaporated in a vacuum to produce an oily viscous crude product that was purified by performing column chromatography with an ethyl acetate and hexane (1:1) eluent. The solvent was concentrated in vacuum to obtain a solid crude product, which was collected

and purified using methanol. The percentage yield was 80%, and this high yield indicates the efficiency of the method used to prepare the target compound 2. Equation 1 illustrates the synthesis method of the target compound 2.



**Equation (1).** Synthesis of 2-[(dipyrrolidin-1-yl)methylene] malononitrile 2.

The formation of 2-[(dipyrrolidin-1-yl)methylene] malononitrile can be explained by the proposed mechanism shown in Equation 2 (Elgemeie et al., 1997). The 2-[(dipyrrolidin-1-yl)methylene] malononitrile 2 was produced *via* a nucleophilic attack of the NH group of the pyrrolidine on the  $\beta$ -carbon of the ethylenic bond in compound 1, followed by the elimination of methanethiol. Intermolecular attack by another pyrrolidine with the loss of another methanethiol yielded the corresponding 2-[(dipyrrolidin-1-yl)methylene] malononitrile 2.



**Equation (2).** Mechanism formation of 2-[(dipyrrolidin-1-yl)methylene]malononitrile 2

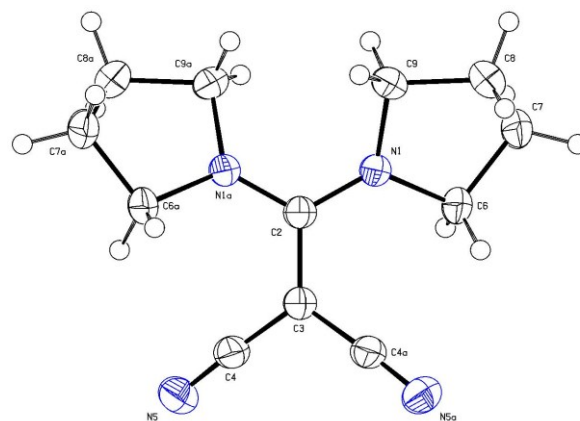
**X-ray crystallography:** The structure of 2-[(dipyrrolidin-1-yl)methylene] malononitrile 2 was identified by x-ray diffraction analysis. Suitable crystals of the compound were grown by slow evaporation of the methanol solution of the compound. Table 1 has a summary of the crystal data and x-ray data

collection, data reduction, and structure refinement results for compound 2.

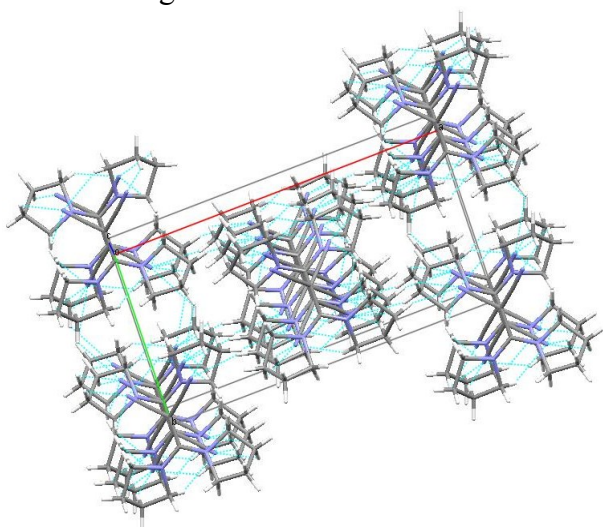
**Table (1).** Crystal data and structure refinement for 2-[(dipyrrolidin-1-yl)methylene]malononitrile 2.

Crystal data (1)	
Chemical formula	C <sub>6</sub> H <sub>8</sub> N <sub>2</sub>
$M_r$	108.14
Crystal system, space group	Orthorhombic, <i>Pbcn</i>
Temperature (K)	150
$a, b, c$ (Å)	16.7024 (5), 8.2383 (3), 8.5041 (3)
$V$ (Å <sup>3</sup> )	1170.15 (7)
$Z$	8
Radiation type	Cu $K\alpha$
$\mu$ (mm <sup>-1</sup> )	0.61
Crystal size (mm)	0.13 × 0.10 × 0.06
Data collection (2)	
Diffractometer	Area
No. of measured, independent and observed [ $I > 2.0\sigma(I)$ ] reflections	12227, 1137, 977
$R_{int}$	0.027
$(\sin \theta/\lambda)_{max}$ (Å <sup>-1</sup> )	0.614
Refinement (3)	
$R[F^2 > 2\sigma(F^2)]$ , $wR(F^2)$ , $S$	0.040, 0.110, 0.94
No. of reflections	1134
No. of parameters	74
$\Delta\rho_{max}$ , $\Delta\rho_{min}$ (e Å <sup>-3</sup> )	0.19, -0.18

The *ORTEP* plot of 2 with the numbering equation is presented in Figure 1. The compound crystallized in orthorhombic crystal system with space group of *Pbcn*



In the title compound, the two cyanide groups and the two pyrrolidine rings adopting *trans* configurations across the C2=C3 bond, as shown in Figure 2.



**Figure (2).** Crystal packing of 2-[(dipyrrolidin-1-yl)methylene] malononitrile 2 viewed down the *c* axis

The bond lengths and angles of the two pyrrolidinyl rings in the compound 2 are within the normal range (Allen et al., 1987). Selected bond distances, angles and torsion angles, are presented in Table 2. The bond length of C2=C3 [1.429(2) Å] is longer than the average [C=C (1.34 Å)] and indicates the single C–C bond character (1.455 Å).

In addition, the bond length of C2–N1 [1.3400(13) Å] and the bond length of C3–C4 [1.4148(16) Å] are shorter than the normal [C–N (1.47 Å)] and the average [C–C (1.455 Å)], respectively. This is probably due to the strong  $\pi$ -delocalization between the C2 and nitrogen atoms which is give rise to a decrease in the bond length of the C–N and an increase in the bond length of the C=C and became highly polarized.

The mean planes data for compound 2 and the dihedral angles between the planes, are listed in Table 3. The fragments N1/N1<sup>a</sup>/C2/C3 and N5/C2/C3/C4/N5a/C4a are both planar. The maximum deviation of N5/C2/C3/C4/N5a/C4a is 0.002(1) around C4, and no deviation has been recorded for

the fragment N1/N1<sup>a</sup>/C2/C3 [0.000 (1)°]. The dihedral angle between the two planes is 27.80(5)°. The pyrrolidine ring N1/ C6/ C7/ C8/C9 is almost planar with a maximum deviation of 0.272(2)° around C8.

**Table (2).** Selected geometric parameters (Å, °) of the 2-[(dipyrrolidin-1-yl)methylene] malononitrile 2.

Bond	Bond length [Å]
N(1)–C(2)	1.3400(13)
N(1)–C(9)	1.4822(18)
C(2)–C(3)	1.429(2)
C(3)–C(4)	1.4148(16)
C(7)–C(8)	1.519(2)
N(1)–C(6)	1.4854(16)
N(5)–C(4)	1.1529(19)
C(3)–C(4)	1.4148(16)
C(6)–C(7)	1.522(2)
C(8)–C(9)	1.524(2)
C(2)–N(1)–C(6)	123.93(11)
C(2)–N(1)–C(9)	125.63(11)
C(6)–N(1)–C(9)	110.44(10)
N(1)–C(2)–C(3)	119.62(8)
N(1)–C(2)–N(1) <sup>a</sup>	120.76(14)
C(3)–C(2)–N(1) <sup>a</sup>	119.62(8)
C(2)–C(3)–C(4)	122.98(8)
C(2)–C(3)–C(4) <sup>a</sup>	122.98(8)
C(4)–C(3)–C(4) <sup>a</sup>	114.05(14)
N(5)–C(4)–C(3)	177.02(15)
C(2)–N(1)–C(9)–C(8)	162.79(12)
N(1)–C(2)–C(3)–C(4) <sup>a</sup>	27.70(9)
C(6)–N(1)–C(2)–C(3)	25.68(14)
C(6)–N(1)–C(2)–N(1)	154.32(11)
C(9)–N(1)–C(2)–C(3)	154.40(11)
C(9)–N(1)–C(2)–N(1) <sup>a</sup>	25.60(16)
C(2)–N(1)–C(6)–C(7)	169.68(11)
C(2)–N(1)–C(9)–C(8)	162.79(12)
N(1)–C(2)–C(3)–C(4) <sup>a</sup>	27.70(9)
C(6)–N(1)–C(2)–C(3)	25.68(14)

The dihedral angle between the pyrrolidine ring and N1/N1<sup>a</sup>/C2/C3 is 33.06(8)°, and the dihedral angle between the pyrrolidine ring and N5/C2/C3/C4/N5a/C4a is 50.57(7)°.

**Table (3).** Plane deviations and angles [°] between selected planes in the structure of 2.

Plane	Atom with greatest deviation	Angle between planes
N1/N1a/C3/C3(i)	0.000 (1)	i & ii = 27.80(5)
N5/C2/C3/C4/N5a/C4a(i)	C4; 0.002(1)	i & iii = 33.06(8)
N1/C6/C7/C8/C9(iii)	C8; 0.272(2)	ii & iii = 50.57(7)

The hydrogen bonding geometry of compound 2 is listed in Table 4. An intramolecular C(6) H(62) N(5) hydrogen bond has been observed in the structure of the compound. The C—H...N hydrogen bonds further stabilize the packing down the *c* direction, Figure 2.

**Table (4).** Hydrogen-bonding geometry (Å, °) for 2

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C(6)—H(62)...N(5) <sup>a</sup>	1.02	2.62	3.3958(19)	134
C(6)—H(61)...N(5) <sup>i, b</sup>	1.02	2.61	3.239(2)	120
C(9)—H(92)...N(5) <sup>ii, b</sup>	1.02	2.61	3.494(2)	145

<sup>a</sup> Intramolecular, <sup>b</sup> Intermolecular; Symmetry codes: (i) 1-x, 1-y, 1-z; (ii) x, 1-y, 1/2+z

## CONCLUSION

In summary, we have successfully synthesized and studied the crystal structure of 2-[(dipyrrolidin-1-yl) methylene] malononitrile 2. The percentage yield was 80%. The crystal data, X-ray data collection, data reduction, and structure refinement results of compounds were presented in this paper. The compound crystallized in an orthorhombic crystal system with a space group of *Pbcn*. In the title compound, the two cyanide groups and the two pyrrolidine rings adopted *trans* configurations across the C2=C3 bond. The crystal packing is stabilized by two intermolecular and one intramolecular C---H...N hydrogen bond, which form a one-dimensional polymeric chain along the axis.

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## تحضير ودراسة التركيب البلوري للمركب 2- [ثنائي بيروليدين-1-يل ميثيلين] مالونونتريل

وداد ميلاد الأديوش<sup>\*</sup>، وداد محمد برق ومريم صالح صالح

قسم الكيمياء، كلية العلوم، جامعة الزاوية، الزاوية- ليبيا

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**المستخلص:** تهدف هذه الدراسة تحضير 2- [ثنائي بيروليدين-1-يل ميثيلين] مالونونتريل 2، ودراسة تركيبه البلوري بواسطة الأشعة السينية. 2- [ثنائي بيروليدين-1-يل ميثيلين] مالونونتريل 2 تم تحضيره عن طريق الاستبدال المباشر لمجموعة الميثيل ثايو (SMe) في المركب 2- [ثنائي (ميثيل ثايو) ميثيلين] مالونونتريل 1 مع بيروليدين كأمين أليفاتي ثانوي عن طريق تفاعل الإضافة والحذف المتعاقب تحت التقطير المرتد لمدة ساعتين. تم الحصول على المركب بإنتاجية عالية (80%). تم تحديد هيكل المركب 2 عن طريق إجراء تحليل حيود الأشعة السينية. تم الحصول على البلورات المناسبة للمركب 2 عن طريق التبخر البطيء للمحلول الميثانولي للمركب حيث تبلور المركب في النظام البلوري المعيني القائم مع المجموعة الفراغية Pbcn. في الشكل البلوري للمركب، مجموعتي السيانيد، وحلقتين بيروليدين تشكل الهيئة الفراغية ترانس عبر رابطة  $C_2=C_3$ ، أطوال، وزوايا الروابط بين حلقتين بيروليدين في المركب 2  $C_{12}H_{16}N_4$  كانت ضمن أطوال الروابط الطبيعية. أقصى انحراف لـ  $N5 / C2 / C3 / C4 / N5^a$  هو  $C4^a$  (1) 0.002 حول  $C4$  ولم يتم تسجيل أي انحراف للجزء  $[0.000(1)^\circ]$   $N1 / N1^a / C2 / C3$ . الزاوية ثنائية السطوح بين حلقة البيروليدين والمستوى  $N1 / N1^a / C2 / C3$  هي  $33.06 (8)^\circ$ ، بينما الزاوية ثنائية السطوح بين حلقة البيروليدين، والمستوى  $N5 / C2 / C3 / C4 / N5^a / C4^a$  هي  $50.57 (7)^\circ$ . البنية البلورية للمركب مرتبطة بواسطة رابطتين هيدروجينيتين بين الجزيئات وواحدة داخل الجزيئية H...N--C، والتي تشكل سلسلة بوليمرية أحادية البعد على طول المحور.

**الكلمات المفتاحية:** دراسة الأشعة السينية أحادية البلورة، التركيب البلوري، النظام البلوري المعيني، الاستبدال المباشر، عامل  $0.110 = wR$ ،  $0.040 = R$

<sup>\*</sup> وداد ميلاد الأديوش [w.aladiwish@zu.edu.ly](mailto:w.aladiwish@zu.edu.ly) قسم الكيمياء، كلية العلوم، جامعة الزاوية، الزاوية- ليبيا.