A study of two geometric isomers of 1,1'-bi-3cyanocyclohex-2-enylidene

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The preparation, separation, crystal structure and ¹H NMR spectra are reported for the two geometrical isomers of 1,1'-bi-3-cyanocyclohex-2-enylidene. The *E*-isomer crystallized as thin plates in the monoclinic space group $P2_1/n$ with a = 5.3980(5), b = 7.0757(7), c = 15.300(2) Å, $\beta = 94.571(2)^\circ$, and Z = 2. The structure has symmetry C_{2h}. The *Z*-isomer crystallized as needles in the triclinic space group $P\overline{1}$ with a = 7.0790(6), b = 11.3155(9), c = 15.386(1) Å, $\alpha = 104.943^\circ \beta = 90.164(2)^\circ$, $\gamma = 99.494(2)^\circ$, and Z = 4. The compound crystallized with two molecules per asymmetric unit. In C₆D₆, ¹H NMR signals of the 2-vinyl protons appear almost identical. However, in a mixture of C₆D₆ and pyridine- d_5 , the 2-vinyl protons can be distinguished. The structures compare favorably with MM2 calculations.

KEY WORDS: Cyano allyl radical; thermal arrangement; crystal structure; isomer; NMR, MM2.

Introduction

In continuing studies of structural perturbations on the transition region of the Cope rearrangement,¹ cyano is a superior perturbing group due to its small size (more suitable for theoretical calculation than phenyl, for example), little, if any, steric interaction, and good radical-stabilizing ability. In order to interpret the effect of cyano group on the rearrangement² and to predict activation parameters, the magnitude of its stabilization of an allyl radical has to be determined. Access to this quantity can be obtained through thermal interconversion of (E)- and (Z)-1,1'bi-3-cyanocyclohex-2-enylidenes (1). Activation parameters for isomerization of E-1 and Z-1 (Scheme 1) measure the difference in enthalpy and entropy of formation of the planar triene 1 as educt and the orthogonal, singlet diradical as transition state.³



Detailed structures of E-1 and Z-1 are important for both experimental and theoretical analysis. Since E-1 and Z-1 can be separated by crystallization, their structures can be determined by x-ray crystallography. The assignment of the correct stereochemistry of E-1 and Z-1 will allow the isomeric structures of this type of compound (1,1'-bi-3-X-cyclohex-2-enylidenes) generally to be assigned by the relative chemical shift of 2-vinyl protons in ¹H NMR.

Experimental

(E)- and (Z)-1,1'-Bi-3-cyanocyclohex-2-enylidenes

3-Cyano-2-cyclohexen-1-one⁴ (10 g, 0.083 mol), prepared from monoethylene ketal of 1,3-cyclohex-

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| Comment. | (T) 1 1/1; 2 | (7) 1 1/1; 2 |
|------------------------------------|--|--|
| Compound | (E)-1,1'-bi-3-cyanocyclohex-2-enylidene | (Z)-1,1'-b1-3-cyanocyclohex-2-enylidene |
| CCDC deposit no. | CCDC-1003/5643 | CCDC-1003/5644 |
| Color/shape | colorless/plate | colorless/needle |
| Empirical formula | $C_{14}H_{14}N_2$ | $C_{14}H_{14}N_2$ |
| Formula weight | 210.27 | 210.27 |
| Temperature | 213(2) K | 213(2) K |
| Crystal system | Monoclinic | Triclinic |
| Space group | $P2_1/n$ | P1 |
| Unit cell dimensions | a = 5.3980(5) Å | a = 7.0790(6) Å |
| | $\alpha = 90^{\circ}$ | $\alpha = 104.943(1)^{\circ}$ |
| (2786 reflections in full | b = 7.0757(7) Å | b = 11.3155(9) Å |
| theta range for ANTI-1) | $\beta = 94.571(2)^{\circ}$ | $\beta = 90.164(2)^{\circ}$ |
| (3106 reflections in full | c = 15.300(2) Å | c = 15.386(1) Å |
| theta range for SYN-1) | $\gamma = 90^{\circ}$ | $\gamma = 99.494(2)^{\circ}$ |
| Volume | 582.5(1) Å ³ | 1173.2(2) Å ³ |
| Z | 2 | 4 |
| Density (calculated) | 1.199 mg/m ³ | 1.191 mg/m ³ |
| Absorption coefficient | 0.072 mm^{-1} | $0.071 \mathrm{mm^{-1}}$ |
| Diffractometer | | Bruker SMART/CCD |
| Radiation/wavelength | | MoK α (graphite monochrom.)/0.71073 Å |
| Theta range for data collection | 2.67 to 24.99° | 1.37 to 28.29° |
| Index ranges | -2 < = h < = 6, -7 < = k | -8 < = h < = 9, -15 < = k |
| C C | < = 8, -18, < = l < = 17 | < = 11, -17, < = l < = 20 |
| Reflections collected | 3293 | 7919 |
| Independent reflections | 1027 [R(int) = 0.0373] | 5489 [R(int) = 0.0335] |
| Data/restraints/parameters | 1027/0/73 | 5489/0/289 |
| Goodness-of-fit on F^2 | 1.002 | 1.112 |
| SHELX-97 weight parameters | 0.0410 | 0.0431 |
| Final R indices $[I > 2\sigma(I)]$ | R1 = 0.0366, wR2 = 0.0786 | R1 = 0.0572, wR2 = 0.1199 |
| <i>R</i> indices (all data) | R1 = 0.0631, wR2 = 0.0847 | R1 = 0.1232, wR2 = 0.1311 |
| Largest diff. peak and hole | 0.101 and $-0.141 \text{ e.}\text{\AA}^{-3}$ | 0.543 and $-0.290 \text{ e.}\text{\AA}^{-3}$ |
| Completeness of data | 100.0% to theta = 24.99° | 94.2% to theta = 28.29° |
| | | |

Table 1. Crystal Data and Structure Refinement

anedione, in 50 mL THF was added to titanium reagent [TiCl₄ (23.6 g, 0.124 mol) was added to 400 mL THF at 0°C, followed by activated zincdust (16.2 g, 0.124 mol) and 10 mL pyridine]. The mixture was heated under reflux for 30 min, then 40 mL of 10% HCl was added dropwise at 0°C.

Table 2. Atomic Coordinates (\times 10⁴) and Equivalent IsotropicDisplacement Parameters (Å² × 10³) for *E*-1

| | x | У | z | $U(eq)^a$ |
|------|----------|----------|---------|-----------|
| N(1) | -1981(3) | 4910(2) | 8809(1) | 61(1) |
| C(1) | 4046(2) | 35(2) | 9677(1) | 31(1) |
| C(2) | 2563(2) | 1726(2) | 9532(1) | 33(1) |
| C(3) | 643(2) | 1854(2) | 8919(1) | 33(1) |
| C(4) | -74(2) | 307(2) | 8278(1) | 40(1) |
| C(5) | 2113(2) | -1005(2) | 8196(1) | 38(1) |
| C(6) | 3266(3) | -1626(2) | 9093(1) | 35(1) |
| C(7) | -798(3) | 3561(2) | 8865(1) | 41(1) |

^{*a*} U(eq) is defined as one-third of the trace of the orthogonalized U^{ij} tensor.

Table 3. Bond Lengths [Å] and Angles $[\circ]$ for E-1^{*a*}

| N(1) - C(7) | 1.1474(18) |
|--------------------|------------|
| C(1) - C(1) # 1 | 1.370(3) |
| C(1) - C(2) | 1.4466(17) |
| C(1) - C(6) | 1.5161(18) |
| C(2) - C(3) | 1.3446(18) |
| C(3) - C(7) | 1.436(2) |
| C(3) - C(4) | 1.4991(19) |
| C(4) - C(5) | 1.5146(19) |
| C(5) - C(6) | 1.5255(19) |
| C(1)#1-C(1)-C(2) | 121.04(15) |
| C(1)#1-C(1)-C(6) | 123.69(15) |
| C(2) - C(1) - C(6) | 115.24(11) |
| C(3) - C(2) - C(1) | 123.31(12) |
| C(2) - C(3) - C(7) | 118.74(13) |
| C(2) - C(3) - C(4) | 123.45(12) |
| C(7) - C(3) - C(4) | 117.81(11) |
| C(3) - C(4) - C(5) | 109.86(11) |
| C(4) - C(5) - (6) | 111.49(12) |
| C(1) - C(6) - C(5) | 112.41(11) |
| N(1) - C(7) - C(3) | 178.68(16) |

^{*a*} Symmetry transformations used to generate equivalent atoms: #1 - x + 1, -y, -z + 2.

Two gemometrical isomers

Table 4. Atomic Coordinates (\times 10⁴) and Equivalent IsotropicDisplacement Parameters (Å² × 10³) for Z-1

| | X | у | z | $U(eq)^a$ |
|--------|----------|----------|----------|-----------|
| N(1) | 2371(3) | 3841(2) | 6901(1) | 62(1) |
| N(2) | 2308(3) | 10388(2) | 5672(1) | 78(1) |
| C(1) | 2615(2) | 5100(2) | 4110(1) | 34(1) |
| C(2) | 2489(2) | 4978(2) | 5022(1) | 36(1) |
| C(3) | 2457(2) | 3897(2) | 5234(1) | 37(1) |
| C(4) | 2473(3) | 2685(2) | 4542(1) | 48(1) |
| C(5) | 1779(3) | 2761(2) | 3632(1) | 49(1) |
| C(6) | 2800(3) | 3923(2) | 3400(1) | 44(1) |
| C(7) | 2408(3) | 3879(2) | 6165(2) | 45(1) |
| C(8) | 2632(3) | 6204(2) | 3899(1) | 33(1) |
| C(9) | 2515(2) | 7342(2) | 4581(1) | 36(1) |
| C(10) | 2459(3) | 8427(2) | 4392(1) | 40(1) |
| C(11) | 2529(3) | 8582(2) | 3453(1) | 54(1) |
| C(12) | 3460(4) | 7590(2) | 2862(2) | 64(1) |
| C(13) | 2730(3) | 6309(2) | 2944(1) | 45(1) |
| C(14) | 2374(3) | 9499(2) | 5110(2) | 51(1) |
| N(1A) | -1581(3) | 8843(2) | -1140(1) | 63(1) |
| N(2A) | 6475(3) | 12032(2) | 3030(1) | 62(1) |
| C(1A) | 4057(3) | 7623(2) | -30(1) | 39(1) |
| C(2A) | 2531(3) | 8232(2) | -235(1) | 38(1) |
| C(3A) | 1100(3) | 7670(2) | -844(1) | 41(1) |
| C(4A) | 855(4) | 6336(2) | -1360(2) | 66(1) |
| C(5A) | 1991(4) | 5644(2) | -896(2) | 79(1) |
| C(6A) | 4023(3) | 6331(2) | -641(2) | 60(1) |
| C(7A) | -354(3) | 8345(2) | -1005(1) | 46(1) |
| C(8A) | 5407(3) | 8160(2) | 657(1) | 38(1) |
| C(9A) | 5387(3) | 9373(2) | 1265(1) | 37(1) |
| C(10A) | 6671(3) | 9882(2) | 1962(1) | 38(1) |
| C(11A) | 8285(3) | 9274(2) | 2172(1) | 49(1) |
| C(12A) | 8740(3) | 8357(2) | 1328(1) | 54(1) |
| C(13A) | 6960(3) | 7483(2) | 863(1) | 51(1) |
| C(14A) | 6519(3) | 11074(2) | 2549(1) | 45(1) |

^{*a*} U(eq) is defined as one-third of the trace of the orthogonalized U^{ij} tensor.

The dark brown solution was filtered through a bed of celite. The filtrate was added saturated NaCl aqueous solution and extracted with diethyl ether several times. The ether solution was washed with 5% NaHCO₃ solution, dried over MgSO₄, and concentrated. The residual orange oil was passed through a silica gel column using 1:1 petroleum/ CH₂Cl₂ as eluting solvents. The elute was concentrated to a solid, which was dissolved in hot ethyl acetate and crystallized at 0° C. Pale-yellow crystals were obtained (E-1) as plates. To the mother liquid was gradually added petroleum ether and pale yellow needle crystals were obtained (Z-1). E-1: mp, 259.5°C; ¹H NMR (10:1 benzene- d_6 /pyridine- d_5), δ 1.03 (tt, J = 5.8 Hz, J = 6.2 Hz, 4H), 1.57 (t, J= 6.2 Hz, 4H), 1.74 (t, J = 5.8 Hz, 4H), 6.67 (s, 2H); MS (EI) m/e 210 (M, 100). Z-1: mp, 171.5°C;

¹H NMR (10:1 benzene- d_6 /pyridine- d_5), δ 1.07 (tt, J = 5.9 Hz, J = 6.2 Hz, 4H), 1.60 (t, J = 6.2 Hz, 4H), 1.75 (t, J = 5.9 Hz, 4H), 6.77 (s, 2H); MS (EI) m/e 210 (M, 100).

X-ray crystallographic analysis

Data were collected using a Bruker SMART CCD based diffractometer equipped with an LT-2 low-temperature apparatus operating at 213 K. Suitable colorless crystals were chosen and mounted on a glass fiber using grease. Data were measured using omega scans of 0.3° per frame for 30 s, such that a hemisphere was collected. A total of 1271 frames were collected with a maximum resolution of 0.75 Å. The first 50 frames were recollected at the end of data collection to monitor for decay. Cell parameters were retrieved using SMART⁵ software and refined using SAINT on all observed reflections. Data reduction was performed using the SAINT software,⁶ which corrects for Lp and decay. Data for Z-1 was limited to 0.85 Å since greater than 80% of the data integrated to less than $2\sigma I$. The structures were solved by the direct method using the SHELXS-97⁷ program and refined by least squares method on F^2 , SHELXL-97,8 incorporated in SHELXTL-PC V 5.10.9

The structures were solved in the space group $P2_1/n(\#14)$ for *E*-1 and *P*-1 (#2) for *Z*-1 as determined by analysis of systematic absences. All nonhydrogen atoms are refined anisotropically. Hydrogens were calculated by geometrical methods and refined as a riding model. The crystals used for the diffraction studies showed no decomposition during data collection. Final refinement values are given in Table 1.

Discussion

1,1'-Bi-3-cyanocyclohex-2-enylidene was synthesized by application of the Mukaiyama-Tyrlic-McMurry reaction¹⁰ to 3-cyano-2-cyclohexen-1-one, which was prepared by hydrolyzing the cyanohydrin formed by reaction of NaCN with the monoethylene ketal of 1,3-hexanedione obtained from 1,3-cyclohexanedione and ethylene glycol (Scheme 2). The resulting mixture of *E*-1 and *Z*-1 can be separated by crystallization. Less soluble *E*-1 crystallized first as plates (mp 259.5°C) from ethyl acetate, and then *Z*-1 was obtained as needles (mp 171.5°C) from the mother liquid by gradual addition of petroleum ether.

Table 5. Bond Lengths [Å] and Angles $[\circ]$ for Z-1^{*a*}

| N(1) - C(7) | 1.145(2) | N(1A) - C(7A) | 1.156(2) |
|-----------------------|------------|--------------------------|------------|
| N(2) - C(14) | 1.153(2) | N(2A) - C(14A) | 1.149(2) |
| C(1) - C(8) | 1.367(2) | C(1A) - C(8A) | 1.367(3) |
| C(1) - C(2) | 1.446(3) | C(1A) - C(2A) | 1.449(3) |
| C(1) - C(6) | 1.514(2) | C(1A) - C(6A) | 1.517(2) |
| C(2) - C(3) | 1.342(3) | C(2A) - C(3A) | 1.335(3) |
| C(3) - C(7) | 1.438(3) | C(3A) - C(7A) | 1.435(3) |
| C(3) - C(4) | 1.505(2) | C(3A) - C(4A) | 1.495(3) |
| C(4) - C(5) | 1.510(3) | C(4A) - C(5A) | 1.504(3) |
| C(5) - C(6) | 1.519(3) | C(5A) - C(6A) | 1.516(3) |
| C(8) - C(9) | 1.451(2) | C(8A) - C(9A) | 1.451(2) |
| C(8) - C(13) | 1.506(3) | C(8A) - C(13A) | 1.515(3) |
| C(9) - C(10) | 1.340(3) | C(9A) - C(10A) | 1.346(2) |
| C(10) - C(14) | 1.425(3) | C(10A) - C(14A) | 1.438(3) |
| C(10) - C(11) | 1.500(3) | C(10A) - C(11A) | 1.501(3) |
| C(11) - C(12) | 1.497(3) | C(11A) - C(12A) | 1.511(3) |
| C(12) - C(13) | 1.494(3) | C(12A) - C(13A) | 1.516(3) |
| C(8) - C(1) - C(2) | 122.87(17) | C(8A) - C(1A) - C(2A) | 123.07(16) |
| C(8) - C(1) - C(6) | 121.83(17) | C(8A) - C(1A) - C(6A) | 122.80(18) |
| C(2) - C(1) - C(6) | 115.27(16) | C(2A) - C(1A) - C(6A) | 114.13(17) |
| C(3) - C(2) - C(1) | 123.26(18) | C(3A) - C(2A) - C(1A) | 123.37(17) |
| C(2) - C(3) - C(7) | 119.11(18) | C(2A) - C(3A) - C(7A) | 119.68(17) |
| C(2) - C(3) - C(4) | 123.23(19) | C(2A) - C(3A) - C(4A) | 123.87(19) |
| C(7) - C(3) - C(4) | 117.66(17) | C(7A) - C(3A) - C(4A) | 116.40(18) |
| C(3) - C(4) - C(5) | 110.11(17) | C(3A) - C(4A) - C(5A) | 110.11(18) |
| C(4) - C(5) - C(6) | 111.48(16) | C(4A) - C(5A) - C(6A) | 111.7(2) |
| C(1) - C(6) - C(5) | 112.64(16) | C(1A) - C(6A) - C(5A) | 111.72(17) |
| N(1) - C(7) - C(3) | 178.6(2) | N(1A) - C(7A) - C(3A) | 177.1(2) |
| C(1) - C(8) - C(9) | 122.03(17) | C(1A) - C(8A) - C(9A) | 122.68(17) |
| C(1) - C(8) - C(13) | 122.13(16) | C(1A) - C(8A) - C(13A) | 121.58(16) |
| C(9) - C(8) - C(13) | 115.84(17) | C(9A) - C(8A) - C(13A) | 115.65(17) |
| C(10) - C(9) - C(8) | 123.38(18) | C(10A) - C(9A) - C(8) | 122.71(17) |
| C(9) - C(10) - C(14) | 119.22(19) | C(9A) - C(10A) - C(14A) | 119.26(17) |
| C(9) - C(10) - C(11) | 123.06(18) | C(9A) - C(10A) - C(11A) | 123.71(17) |
| C(14) - C(10) - C(11) | 117.70(18) | C(14A) - C(10A) - C(11A) | 117.03(17) |
| C(12) - C(11) - C(10) | 109.91(17) | C(10A) - C(11A) - C(12A) | 109.41(17) |
| C(13) - C(12) - C(11) | 114.56(19) | C(11A) - C(12A) - C(13A) | 112.06(17) |
| C(12) - C(13) - C(8) | 114.21(17) | C(8A) - C(13A) - C(12A) | 112.82(17) |
| N(2) - C(14) - C(10) | 177.8(2) | N(2A) - C(14A) - C(10A) | 177.2(2) |

^a Symmetry transformations used to generate equivalent atoms.



Fig. 1. Drawing at 50% ellipsoids showing E-1.



Fig. 2. Drawing showing both molecules of Z-1 drawn at 50% ellipsoids.



Scheme 2. Synthesis of E-1 and Z-2.

E-1 crystallized in the monoclinic space group $P2_1/n$ with the molecule sitting on an inversion center of the cell. Atomic positions are given in Table 2, while bond distances and angles are shown in Table 3. The structure of *E*-1 is shown in Fig. 1. The molecule has C_{2h} symmetry. *Z*-1 crystallized in the space group P1 (#2) and has two molecules per asymmetric unit. Atomic positions are given in Table 4. The bond distances and angles are shown in Table 5. Figure 2 shows *Z*-1 with thermal ellipsoids with its established structure.

In *E*-1, the angle of twist along the central bond is $0^{\circ} (\angle C_2 - C_1 - C_{1A} - C_{2A} = -180^{\circ})$. This is also true of one of the molecule in the unit cell $(\angle C_2 - C_1 - C_8 - C_9 = -0.2^{\circ})$ and very nearly so of the other $(\angle C_{2A} - C_{1A} - C_{8A} - C_{9A} = 2.5^{\circ})$. The other dihedral angles define derivation of the triene system from coplanarity are all close to 180° : *E*-1, $\angle C_{1A} - C_1 - C_2 - C_3 = -178.7^{\circ}$; *Z*-1, $\angle C_8 - C_1 - C_2 - C_3 = 179.5^{\circ}$, $\angle C_1 - C_8 - C_9 - C_{10} = -177.5^{\circ}$; $(Z-1)_A$, $\angle C_{8A} - C_{1A} - C_{2A} - C_{3A} = -173.1^{\circ}$, $\angle C_{1A} - C_{1A} - C_{2A} - C_{$

The structures formed by MM2 calculation are confirmed by X-ray structures. Interannular interactions of hydrogen atoms are noted in Figs. 3 and 4.



Fig. 3. Drawing showing the intermolecular interactions of hydrogen atoms (Å) for *E*-**1.** Those on parenthesis are those from MM2 calculations.



Fig. 4. Drawing showing the intermolecular interactions of hydrogen atoms (\AA) for Z-1. Those in parenthesis are those from MM2 calculations.

The distances calculated by MM2 program (in parentheses) compare favorably with the X-ray distances.

The ¹H NMR spectra of E-1 and Z-1 in benzene d_6 are almost identical. However, in a mixture of benzene- d_6 and pyridine- d_5 (10:1), two isomers can be distinguished by the chemical shifts of 2-vinylprotons (6.67 ppm for E-1 and 6.77 ppm for Z-1). Based on ¹H NMR assignment of E-1 and Z-1 (helped by X-ray structures), it is now possible to speculate isomeric assignment for the other compounds with the same type. The isomer that has a greater value of chemical shift for the 2-vinyl hydrogen is assigned to the Z-isomer and smaller value the E-isomer. The speculation has been examined against the similar types of compounds previously studied in this laboratory and appeared correct so far (for example, 1,1'bicyclohex-2-enylidene¹ in benzene- d_6 : E-isomer, 6.59 ppm, Z-isomer, 6.77 ppm; 1,1'-bi-3-phenylcyclohex-2-envlidene³ in benzene- d_6 : *E*-isomer, 7.10 ppm, Z-isomer, 7.50 ppm).

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