Molecular Crystals and Liquid Crystals

ISSN: 1542-1406 (Print) 1563-5287 (Online) Journal homepage: http://www.tandfonline.com/loi/gmcl20

New Approach for Base Catalyzed Eco-Friendly Friedländer Synthesis of Racemic 2,10-Dinitro-8,16-diphenyl-6,7,14,15-tetrahydro-7,15-methanocycloocta[1,2-b:5,6-b']diquinoline-Propanoic Acid Cocrystal: Thermal Properties, Antibacterial Activity, and Self-Assembly

Solhe F. Alshahateet

To cite this article: Solhe F. Alshahateet (2015) New Approach for Base Catalyzed Eco-Friendly Friedländer Synthesis of Racemic 2,10-Dinitro-8,16-diphenyl-6,7,14,15-tetrahydro-7,15-methanocycloocta[1,2-b:5,6-b']diquinoline-Propanoic Acid Cocrystal: Thermal Properties, Antibacterial Activity, and Self-Assembly, Molecular Crystals and Liquid Crystals, 607:1, 169-180, DOI: 10.1080/15421406.2014.927959

To link to this article: http://dx.doi.org/10.1080/15421406.2014.927959

Mol. Cryst. Liq. Cryst., Vol. 607: pp. 169–180, 2015

ISSN: 1542-1406 print/1563-5287 online
DOI: 10.1080/15421406.2014.927959

New Approach for Base Catalyzed Eco-Friendly Friedländer Synthesis of Racemic 2,10-Dinitro-8,16-diphenyl-6,7,14,15-tetrahydro-7,15-methanocycloocta[1,2-*b*:5,6-*b′*]diquinoline-Propanoic Acid Cocrystal: Thermal Properties, Antibacterial Activity, and Self-Assembly

SOLHE F. ALSHAHATEET*

Department of Chemistry, Mutah University, Alkarak, Jordan

Racemic host 2,10-dinitro-8,16-diphenyl-6,7,14,15-tetrahydro-7,15-methanocycloocta[1,2-b:5,6-b′]diquinoline ***6*** *was prepared in high yield as single isomeric product through an ecofriendly friedländer condensation of two equivalents of 2-amino-5-nitrobenzophenone and 1 equiv of bicycle[3.3.1]nonane-3,7-dione in presence of catalytic amount of solid sodium hydroxide (ca. 5%). The versatile host compound* ***6*** *crystallizes from propanoic acid and yielded crystals of the binary inclusion cocrystal (****6****)-propanoic acid (2/3). Self-assembly analysis of the resulted inclusion compound was achieved using X-ray single crystal technique and reported in terms of crystal engineering and supramolecular chemistry. Thermal studies on* $(\mathbf{6})_2\cdot(\text{propanoic acid})_3$ *lattice inclusion compound revealed that it decomposed in two steps; each decomposition step is related to an endotherm. Antibacterial activity of host* ***6*** *proved that it exhibited weak activity against* Micrococus luteus *microorganism causing 10 mm inhibition at 1 mg/disc.*

Keywords 2,10-dinitro-8,16-diphenyl-6,7,14,15-tetrahydro-7,15-methanocycloocta [1,2-b:5,6-b′]diquinoline; binary inclusion cocrystal; eco-friendly friedländer reaction; racemic mixture; X-ray single crystal structure

Introduction

Supramolecular chemistry and green chemistry are considered important branches of science that attracted the attention of chemists in designing new crystalline materials. This is clearly recognized by the awarding of Nobel Prize in chemistry jointly to Charles J Pederson, Donald J Cram, and Jean-Marie Lehn in 1987 for their work in the filed of supramolecular chemistry and crystal engineering which is concerned in structural reactivity and material chemistry of the solid state [1–6]. Two decades later, green chemistry was highly appreciated and recognized by awarding Nobel Prize in chemistry jointly to

*Address correspondence to Solhe F. Alshahateet, Department of Chemistry, Mutah University, P.O. BOX 7, Mutah 61710, Alkarak, Jordan. Tel.: +962-3-2372380; Fax: +962-3-2375540; E-mail: solhe_alshahateet@yahoo.com

Color versions of one or more of the figures in the article can be found online at www.tandfonline.com/gmcl.

Robert H Grubbs, Yves Chauvin, and Richard Schrock in 2005 due to their work in development of the metathesis method in organic synthesis. Currently, supramolecular chemistry attracted chemists to remain dedicated to discovering and developing new and better solvent-free approaches for efficient eco-friendly synthesis of many existing and new compounds [7–8]. Our research group has been interested in developing and synthesizing different types of solid molecules with different functionalities, and testing their ability to form different forms of lattice inclusion compounds that can help in understanding the inclusion phenomena [9–22]. Recently, we were interested in designing, developing, and synthesizing new diquinoline hosts; our interest in this type of compounds is due to the fact that quinolines derivatives are used as drugs for different diseases such as malaria, schizophrenia, and tuberculosis, apart from being studied as supramolecular hosts [23]. In this paper we report a new eco-friendly Friedländer synthesis approach to prepare the racemic host 2,10-dinitro-8,16-diphenyl-6,7,14,15-tetrahydro-7,15-methanocycloocta[1,2-*b*:5,6-*b*′]diquinoline **6** in high yield as single isomeric product (Scheme 1). In addition, host compound **6** formed lattice inclusion cocrystal of the formula $(\mathbf{6})_2\cdot(\text{propanoic acid})_3$ upon direct crystallization from propanoic acid, its solid-state structure was studied by X-ray single crystal technique, analyzed and presented in terms of crystal engineering and supramolecular chemistry. The antibacterial activity of host **6** and thermal properties of $(\mathbf{6})_2\cdot(\text{propanoic acid})_3$ were investigated and reported.

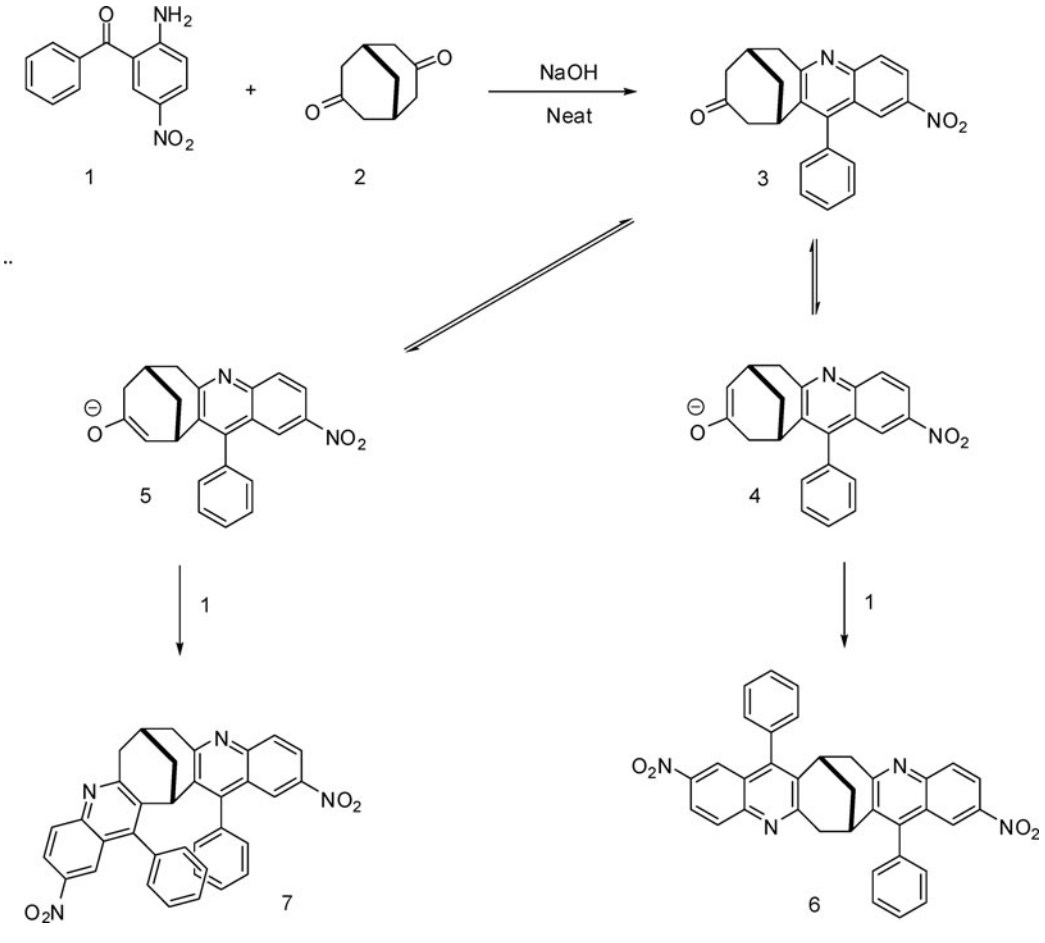
Experimental Details

Materials and Physical Measurements

All chemicals and reagents were obtained commercially and used without further purification. Melting point was measured on a Stuart scientific melting point apparatus in open capillary tubes and is uncorrected. FTIR spectra were measured by direct transmittance by means of the KBr pellet technique and recorded over the range 4000–500 cm^{-1}, on a Maltson 5000 FTIR spectrophotometer. The ^{1}H-NMR and ^{13}C-NMR experiments were conducted on a Bruker 400 MHz. Chemical shifts were referenced to *TMS* as the internal standard and deuterated dimethylsulfoxide (DMSO-d_6) as the solvent. X-ray single crystal structure determination was carried out at Department of Chemistry, National University of Singapore. Thermogravimetric (TGA) and differential scanning calorimetry analysis (DSC) were performed on a TA instrument SDT 2960 with simultaneous TGA-DSC.

Preparation of the 2,10-dinitro-8,16-diphenyl-6,7,14,15-tetrahydro-7,15-methanocycloocta[1,2-b:5,6-b′]diquinoline 6

Eco-friendly selective and clean synthesis of host compound **6** was achieved by using base catalyzed solvent-free approach through Friedländer condensation reaction of 2-amino-5-nitrobenzophenone **1** (1.60 g, 6.6 mmol) and racemic bicycle[3.3.1]nonane-3,7-dione **2** [24] (0.5 g, 3.3 mmol) in presence of catalytic amount of solid sodium hydroxide (*ca*. 5%). The mixture was ground with a mortar and pestle at room temperature until it produced a gummy material which solidified upon standing overnight. The reaction mixture was then reground and tested by TLC technique that confirmed the presence of single isomer. The reaction mixture was poured into HCl (5 mL, 2 M), the solid was collected and dried to give analytically pure host compound **6** in racemic form (90%, mp $> 300^\circ$C, as pale orange solid) as single isomeric Friedländer product; FTIR (KBr) 3099, 2874, 2825, 1643, 1593,



Scheme 1. Synthetic route for eco-friendly Friedländer isomeric product **6.**

1537, 1481, 1442, 1383, 1346, 1232, 1030, 956, 914, 848, 821, 744, 704, 619. Found: C, 74.42; H, 4.31; N, 9.95. ($C_{35}H_{24}N_4O_4$) requires C, 74.46; H, 4.28; N, 9.92%. NMR and HRMS data were in agreement with what was reported previously for the preparation of host compound **6** in refluxing acid-catalyzed alcoholic solution [21, 25–26].

Solution and Refinement of the Crystal Structures of Lattice Inclusion Cocrystal (6)$_2$·(propanoic acid)$_3$

X-ray quality crystals of the lattice inclusion cocrystal (**6**)$_2$·(propanoic acid)$_3$ were obtained by dissolving 30 mg of solvent-free compound **6** in 1 mL of propanoic acid. Slow evaporation of the solution led to X-ray quality crystals. The single crystal X-ray diffraction experiment was carried out on a Bruker SMART APEX diffractometer equipped with a CCD detector and MoKα sealed tube at 223(2) K. SMART [27] was used for collecting frames data, indexing reflection, and determining lattice parameters. SAINT [27] was used for integrating intensity of reflections and scaling. SADABS [28] was used for absorption correction and SHELXTL [29] for space group and structure determination and least-squares refinements on F^2. All C-H hydrogen atoms were placed in calculated positions for the purpose of structure factor calculation. The hydrogen atoms of the propanoic acid molecules were located from peaks from different map. Crystallographic data (cif) have been deposited with the Cambridge Structural Data Centre, CCDC reference number 721711. See http://www.rsc.org/suppdata/ for crystallographic data in cif format. Copies of the data can be obtained, free of charge, on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK [fax +44(0)-1223-336033 or e-mail deposit@ccdc.cam.ac.uk]. The numerical details, data processing, and refinement of the X-ray structure of the lattice inclusion cocrystal (**6**)$_2$·(propanoic acid)$_3$ are listed in Table 1.

In vitro Antimicrobial Activity

The antimicrobial activity of lattice inclusion host **6** was determined as described earlier [30]. In this study, *Bacillus subtilis* ATCC 6633, *Staphylococcus aureus* ATCC 43300, *Micrococcus luteus* ATCC 10240, *Escherichia coli* ATCC 25922, *Enterobacter aerugenes* ATCC 13048 were used as test microorganisms.

Thermal Analyses; TGA and DSC

Crystals of binary lattice inclusion compound (**6**)$_2$·(propanoic acid)$_3$ were dried and placed in alumina sample pans. In each case, sample masses were 15–20 mg, and was purged using a stream of nitrogen flowing at 200 mL/min. A temperature program was used to carry out the experiments. The temperature started from ambient temperature, isothermal for 10 min, with 10°C/min ramb rate and up to 750°C.

Results and Discussion

Preparation of the 2,10-dinitro-8,16-diphenyl-6,7,14,15-tetrahydro-7,15-methanocycloocta[1,2-b:5,6-b′]diquinoline 6

The racemic dinitrodiphenyldiquinoline host **6** was prepared as outlined in Scheme 1. Eco-friendly Friedländer condensation of 2-amino-5-nitrobenzophenone **1** with racemic bicycle[3.3.1]nonane-3,7-dione **2** in presence of catalytic amount of solid sodium hydroxide

Table 1. Crystallographic parameters for $(\mathbf{6})_2 \cdot (\text{propanoic acid})_3$ lattice inclusion cocrystal

Formula	$(C_{35}H_{24}N_4O_4)_2 \cdot (CH_3CH_2CO_2H)_3$
Temperature	223(2) K
Wavelength	0.71073 Å
Crystal system	Triclinic
Space group	P-1
Unit cell dimensions	$a = 11.8368(9)$ Å, $\alpha = 107.680(2)^\circ$.
	$b = 12.2351(9)$ Å, $\beta = 106.466(2)^\circ$.
	$c = 12.8809(10)$ Å, $\gamma = 92.333(2)^\circ$.
Volume	1688.0(2) $Å^3$
Z	2
Density (calculated)	1.329 Mg/m^3
Absorption coefficient	0.093 mm^{-1}
F(000)	708
Crystal size	$0.36 \times 0.20 \times 0.10$ mm^3
Theta range for data collection	1.75 to 25.00°.
Index ranges	$-14 <= h <= 14$, $-14 <= k <= 14$, $-15 <= l <= 15$
Reflections collected	17650
Independent reflections	5943 [R(int) = 0.0318]
Completeness to theta = 25.00°	99.9%
Absorption correction	Sadabs (Sheldrick 2001)
Max. and min. transmission	0.9908 and 0.9675
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	5943 / 16 / 479
Goodness-of-fit on F^2	1.042
Final R indices [I>2sigma(I)]	$R^1 = 0.0754$, $wR^2 = 0.1825$
R indices (all data)	$R^1 = 0.0957$, $wR^2 = 0.1959$
Largest diff. peak and hole	0.877 and −0.681 e.$Å^{-3}$

(*ca*. 5%) gave the diquinoline host **6** as the only detected and isolated product in 90% yield (lit. yield 50% [21]). Zolfigol et al. [31] and Wang et al. [32] reported similar solvent-free approaches for the synthesis of polysubstituted quinolines in which they used iodine and *p*-toluene-sulphonic acid as catalysts, respectively. Rahman et al. [8] reported the use of solid sodium hydroxide as catalyst for facile solvent-free Cliasen-Schmidt reactions of cycloalkanones with substituted benzaldehydes through a grinding technique. Herein, the eco-friendly Friedländer condensation reaction is quite interesting to have further comment: None of the alternative Friedländer isomeric product **7**, which would be expected from condensation of the enolate intermediate **5** with the second 2-amino-5-nitrobenzophenone molecule, was detected. Based on the literature precedent, this outcome was not expected. Aguado et al. [33] modified Friedländer procedure to condense bicycle[3.3.1]nonane-3,7-dione **2** with 2-aminobenzonitrile derivative and the resulted reaction mixture gave substantial proportions of both cyclization products. In our current study, sodium hydroxide enhances the tautomerization of substructure **3** to isomeric enolate intermediates **4** and **5**. As a result, only enolate **4** is condensed with the second molecule of **1**. In addition, the undetected isomeric product **7** is relatively unstable comparing to isomeric product **6** due to the presence of high torsional and steric strain. Similar observations have been rationalized

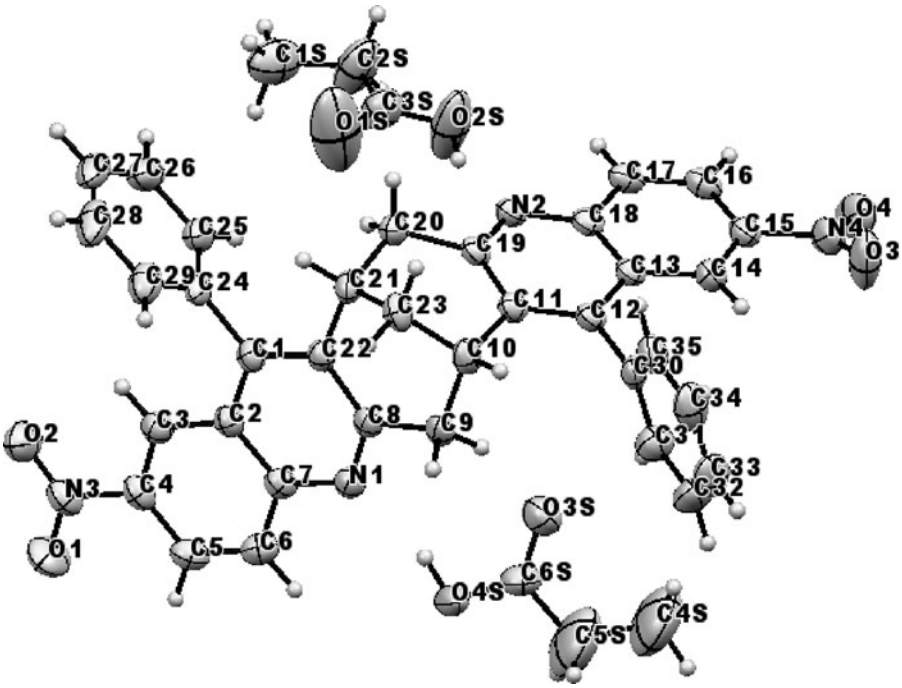


Figure 1. The asymmetric unit of the title structure of cocrystal $(\mathbf{6})_2\cdot$(propanoic acid)$_3$, displacement *ellipsoids* are shown at the 50% probability level. For clarity, only nonhydrogen atoms were labeled.

in our related cases. For example, base catalyzed Friedländer condensation of racemic bicycle[3.3.1]nonane-3,7-dione **2** with 2-aminobenzaldehyde and 2-aminoacetophenone in methanol and ethanol, respectively, was found to yield only one isomeric Friedländer product [12, 34]. The racemic form of host compound **6** is very interesting due to its unusual supramolecular behavior compared to the other diquinolines that we have reported recently [9–22]. It interacted selectively with polar guest molecules [21, 25, 26] which open doors for further screening for new forms of lattice inclusion compounds formed between molecules of **6** and polar molecules of different functionalities.

X-ray Analysis of the Crystal Structure of $(6)_2\cdot$(propanoic acid)$_3$

Crystallization of the isomeric Friedländer product **6** from propanoic acid afforded crystals of the lattice inclusion cocrystal $(\mathbf{6})_2\cdot$(propanoic acid)$_3$ that contains both enantiomers. The racemic title cocrystal, $(\mathbf{6})_2\cdot$(propanoic acid)$_3$ crystallizes in the triclinic space group P-1. The asymmetric unit contains one title molecule with one and a half propanoic acid molecule. The molecular structure of lattice inclusion cocrystal $(\mathbf{6})_2\cdot$(propanoic acid)$_3$ including thermal displacement ellipses with 50% probability is illustrated in Fig. 1. Determination of the function and the structure of any given molecules; including biological ones are determined by hydrogen bonding as in the case of carbohydrates, nucleic acids, and proteins [35]. Propanoic acid is hydrogen bonded to one of the nitrogen atoms of the titled molecule **6** with a contact distance of 2.75 Å (N2 . . . O2S), half of another propanoic acid molecule is disordered into two positions (50:50) through the centre of symmetry, and hydrogen bonded to the other nitrogen atom of the title molecule **6** with a contact distance

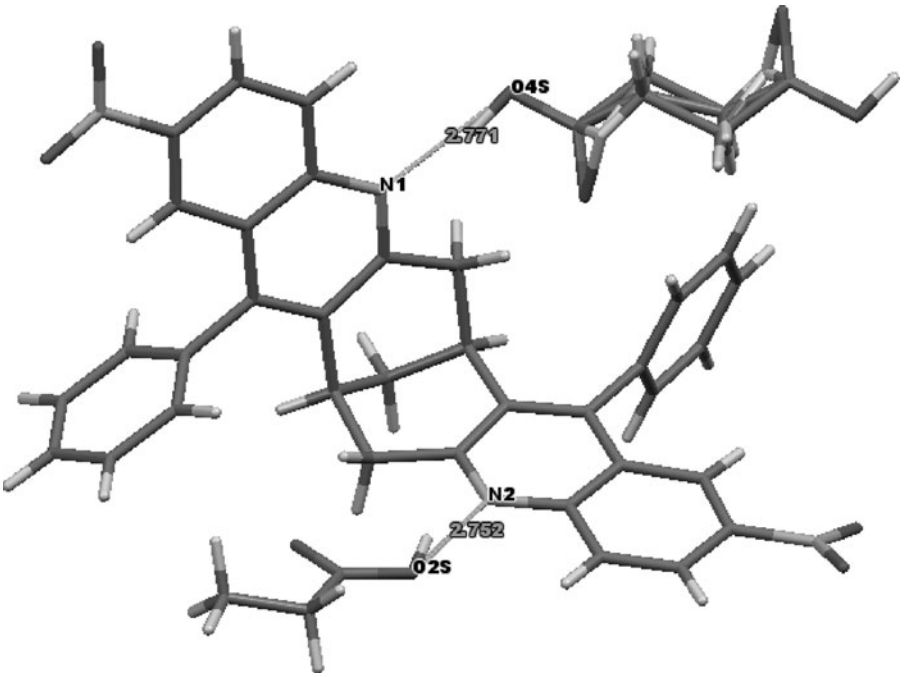


Figure 2. Host–guest interactions between molecules of **6** with two molecules of propanoic acid through different hydrogen bond motifs.

of 2.77 Å (N1 . . . O4S) as shown in Fig. 2. Distances and geometries of different motifs of hydrogen bonds exist in crystal structure of $(\mathbf{6})_2\cdot$(propanoic acid)$_3$ lattice inclusion cocrystal and they are listed in Table 2. It was reported that the C-N-C bond angle in aromatic heterocyclic nitrogen compounds (such as pyridine and its derivatives) is sensitive to protonation, and accordingly, its cationic form exhibits higher bond angle than the corresponding neutral form [36, 37]. The geometries and distances of the pyridine rings existing in host **6** showed that the two C7-N1-C8 and C18-N2-C19 bond angles (Table 3) are within the average literature value for such angle, which is 116±2° [38]. Furthermore, the two C-O bonds of the propanoic acid molecule exhibit different distances (1.18 and 1.33 Å with O-C-O bond angle of 122.6°). These different values proved that the molecules in the binary inclusion cocrystal of **6** are interacted via only strong hydrogen bonds; no proton transfer occurred. In addition, the infrared absorption measurement indicated that the band associated with the carbonyl group of the propanoic acid is observed at 1718 cm^{-1} which proved that only hydrogen-bonded binary compound is formed. In case of proton-transfer, the band of the

Table 2. Hydrogen-bond geometry exist in crystal structure of $(\mathbf{6})_2\cdot$(propanoic acid)$_3$, (Å, °)

D-H . . . A	d(D-H)	d(H . . . A)	d(D . . . A)	<(DHA)
O(2S)-H(2X) . . . N(2)	0.901(19)	0.90(2)	2.751(4)	157(4)
O(4S)-H(4X) . . . N(1)	0.88(2)	1.91(3)	2.771(5)	165(8)

Table 3. Comparison study between the three different lattice inclusion cocrystals formed by racemic host **6**

Acid	Cocrystal formula	N1	N2	(C=O/C−O) distances and angle	C−N−C angles	Crystal system and space group
Formic (I)	$(\mathbf{6})_2\cdot(I)_3\cdot(H_2O)$	N1..O1W (2.88 Å)	N2..O2S (2.66 Å)	1.19/1.30 Å, 126.14° (H-bonded with N2)	118.33° and 119.47°	Orthorhombic *Pbca*
Acetic (II)	$(\mathbf{6})\cdot(II)_2$	N1..O2S (2.70 Å)	N2..O4S (2.75 Å)	1.21/1.29 Å, 123.29° (H-bonded to N1). 1.23/1.28 Å, 116.14° (H-bonded to N2)	118.16° and 119.75°	Triclinic *P-1*
Propanoic (III)	$(\mathbf{6})_2\cdot(III)_3$	N1..O4S (2.77 Å)	N2..O2S (2.75 Å)	1.18/1.33 Å, 122.58° (H-bonded to N1). 1.20/1.24 Å, 120.66° (H-bonded to N2)	118.51° and 118.94°	Triclinic *P-1*

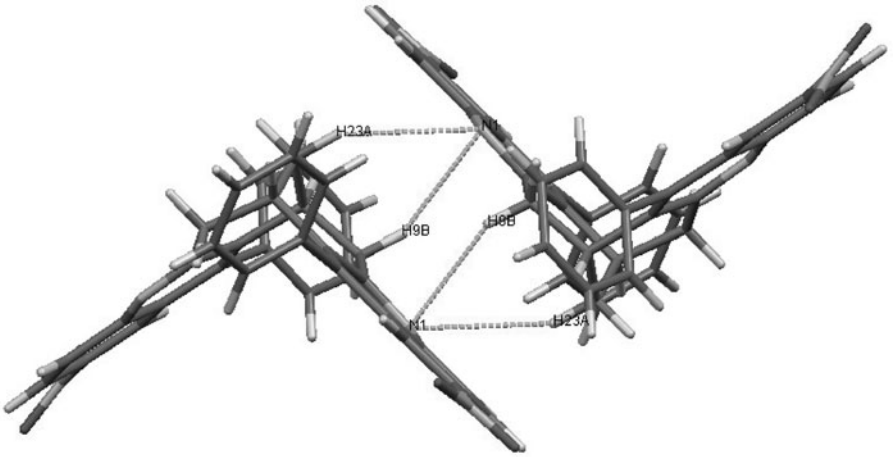


Figure 3. Host–host interactions between two opposite enantiomers of **6** which yielded a centrosymmetric dimer.

carboxylate group should be shifted to a lower wave number ($\sim$1540 cm^{-1}) which was not occurred. Previously, we reported the inclusion behavior of host **6** with small monoprotic organic acids; formic acid [25] and acetic acid [26]. Both acids gave lattice inclusion cocrystal with different stoichiometry, geometries, and supramolecular motif interactions, Table 3 shows a comparison between these properties. The difference in the hydrogen bond distances might be due to the difference in the pKa values for the three acids. Since the acetic acid and propanoic acid are very close in the pKa value (4.75 and 4.87, respectively), they exhibit relatively shorter hydrogen bond distances compared to those existing in case of the relatively stronger acid, formic acid (pKa = 3.75). All supramolecular architectures of the lattice inclusion cocrystal $(\mathbf{6})_2\cdot$(propanoic acid)$_3$ involve extensive N . . . H, N . . . N, N . . . O, O . . . O, and O . . . H noncovalent interactions. Opposite enantiomers of host **6** are bifurcated through double H9B . . . N1 . . . H23A interaction with contact distances of 2.80 and 2.91 Å, respectively, to produce centrosymmetric dimer (Fig. 3). The shortest contact distance between nitrogen atoms of molecules of **6** with opposite chirality is 4.36 Å and it occurred as double N1 . . . N3 interaction. Nonhydrogen atoms, such as oxygen and nitrogen interacted together in form of N4 . . . O1 (3.00 Å) and O1 . . . O4 (3.00 Å) interactions between molecules of **6** with same symmetry. Nitro group of one molecule of **6** is interacting through one of its oxygen atom (O1) with an aromatic hydrogen atom (H16) of another molecule of **6** with contact distance of 2.81 Å. Disordered propanoic acid molecule is bifurcated through its O3S nitrogen with two aromatic hydrogen atoms (H16 and H17) of host molecule **6** with contact distances of 2.60 and 2.68 Å as shown in Fig. 4. The crystal packing of the inclusion cocrystal $(\mathbf{6})_2\cdot$(propanoic acid)$_3$ is illustrated in Fig. 5.

In vitro Antimicrobial Activity of Lattice Inclusion Host 6

Lattice inclusion host **6** exhibited weak activity against *Micrococcus luteus* microorganism causing 10 mm inhibition at 1 mg/disc. The MIC for compound **6** against the susceptible bacterial strain is found to be 0.5 mg/mL with bacterio static effect. Despite that lattice inclusion host **6** is lipophilic; its molecular size may render its permeability through the outer layers of the cell wall.

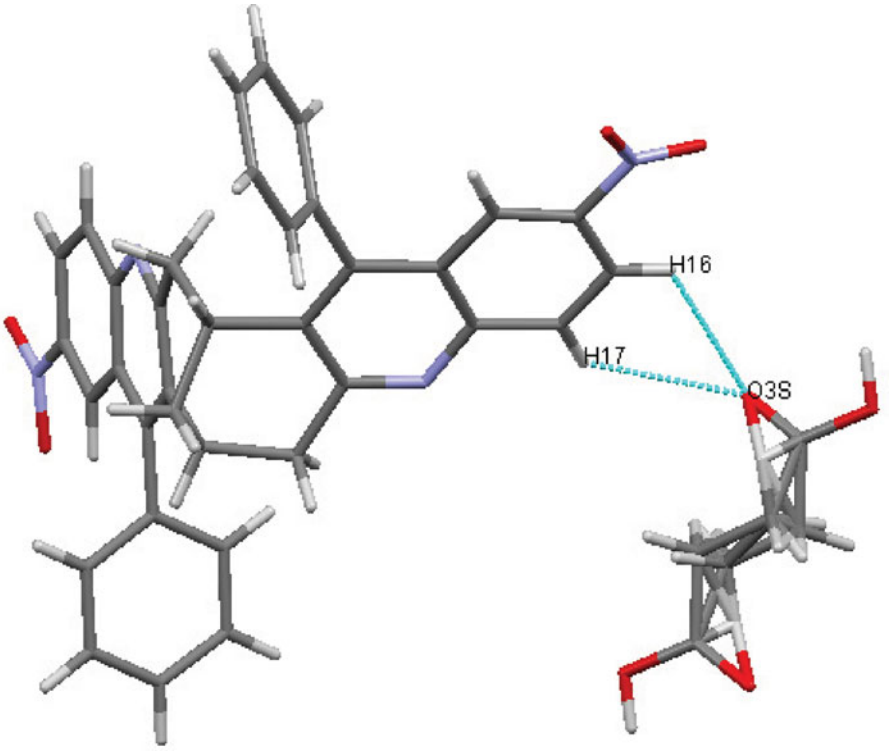


Figure 4. Host–guest interactions between disordered propanoic acid molecule and host **6**.

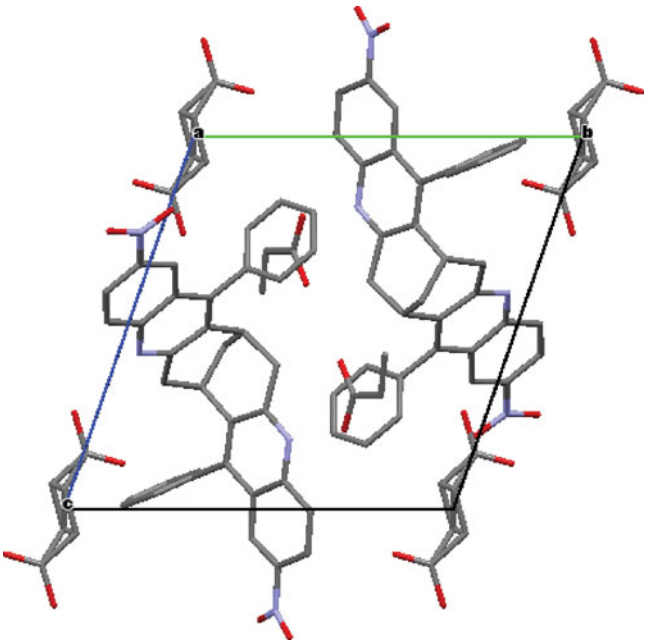


Figure 5. Crystal packing of lattice inclusion cocrystal $(\mathbf{6})_2$·(propanoic acid)$_3$; all hydrogen atoms were omitted for clarity.

Thermal Properties; TGA and DSC Studies on Binary Lattice Inclusion (6)$_2$·(propanoic acid)$_3$

The thermal stability and decomposition profile of the binary lattice inclusion (**6**)$_2$·(propanoic acid)$_3$ is examined by performing TGA and DSC studies. The (**6**)$_2$·(propanoic acid)$_3$ lattice inclusion compound decomposed in two steps. The measured mass loss from the inclusion compound was 16.70% and in agreement with the calculated value (16.45%). In DSC, each decomposition step is related to an endotherm with onset temperature of 135–137.5°C which is lower than that of the parent molecules, compound **6** and propanoic acid individually. However, due to the intricacy of the decomposition, comparison of the onset temperature with the boiling point of the guest was not possible.

Conclusions

The versatile host **6** was prepared through new eco-friendly solvent-free Friedländer approach with clean and high yield outcome compared to the reported procedure which was conducted in refluxing acidic alcoholic solution. Compound **6** proved again to be an interesting molecule that formed a binary lattice inclusion cocrystal with propanoic acid. The solid state behavior of the resulted cocrystal was studied using X-ray crystallography technique and analyzed in terms of crystal engineering and supramolecular chemistry. All supramolecular architectures of the lattice inclusion cocrystal (**6**)$_2$·(propanoic acid)$_3$ involve extensive N . . . H, N . . . N, N . . . O, O . . . O, and O . . . H noncovalent interactions and can be used to rationalize the stability of resulted cocrystal. Antibacterial activity of host **6** proved that it exhibited weak activity against *Micrococcus luteus* microorganism causing 10 mm inhibition at 1 mg/disc. Thermal studies on (**6**)$_2$·(propanoic acid)$_3$ lattice inclusion compound revealed that it decomposed in two steps; each decomposition step is related to an endotherm.

Acknowledgments

The author thanks Mutah University (Jordan), Institute of Chemical and Engineering Sciences (Singapore) and the National University of Singapore (Singapore) for conducting the experiments needed to complete this work. Many thanks are due to Dr. Wael A. Al-Zereini (Department of Biological Science, Mutah University, Jordan) for his help in performing the antibacterial tests.

References

[1] Lehn, J.-M. (1978). *Pure Appl. Chem.*, *50*, 871.
[2] Lehn, J.-M. (1995). *Supramolecular Chemistry: Concepts and Perspectives*, Wiley-VCH: Weinheim, ISBN 978-3527293117
[3] Steed, J. W., & Atwood, J. L. (2000). *Supramolecular Chemistry*, Wiley: Chichester.
[4] Schalley, C. A. (2007). *Analytical Methods in Supramolecular Chemistry*, Wiley-VCH: Weinheim.
[5] Himaja, M., Poppy, D., & Asif, K. (2011). *Int. J. Res. Ayurveda Pharm.*, *2*(4), 1079.
[6] Desiraju, G. R. (1989). *Crystal Engineering: The Design of Organic Solids*, Elsevier: Amsterdam.
[7] Patil, S., Jadhav, S. D., & Patil, U. P. (2012). *Arch. Appl. Sci. Res.*, *4*(2), 1074.
[8] Motiur Rahman, A. F. M., Ali, R., Jahng, Y., & Kadi, A. A. (2012). *Molecules*, *17*, 571.

[9] Alshahateet, S. F., Bishop, R., Craig, D. C., & Scudder, M. L. (2011). *Cryst. Growth Des.*, *11*, 4474.
[10] Alshahateet, S. F., Rahman, A. N. M. M., Bishop, R., Craig, D. C., & Scudder, M. L. (2002). *Cryst. Eng. Comm.*, *4*(97), 585.
[11] Alshahateet, S. F., Bishop, R., Craig, D. C., & Scudder, M. L. (2001). *Cryst. Eng. Comm.*, *3*(48), 225.
[12] Alshahateet, S. F., Bishop, R., Craig, D. C., & Scudder, M. L. (2004). *Cryst. Growth Des.*, *4*, 837.
[13] Ghalib, R. M., Hashim, R., Alshahateet, S. F., Mehdi, S. H., Sulaiman, O., *et al.* (2012). *J. Chem. Crystallogr.*, *42*, 783.
[14] R Ghalib, R. M., Hashim, R., Alshahateet, S. F., Mehdi, S. H., Sulaiman, O., *et al.* (2011). *J. Mol. Struct.*, *1005*, 152.
[15] Bishop, R., Scudder, M. L., Craig, D. C., Rahman, A. N. M. M., & Alshahateet, S. F. (2005). *Mol. Cryst. Liq. Cryst.*, *440*, 173.
[16] Alshahateet, S. F., Bishop, R., Craig, D. C., & Scudder, M. L. (2001). *Cryst. Eng. Comm.*, *3*(25), 107.
[17] Alshahateet, S. F., Bishop, R., Craig, D. C., & Scudder, M. L. (2010). *Cryst. Growth Des.*, *10*, 1842.
[18] Alshahateet, S. F., Bishop, R., Scudder, M. L., Hu, C. Y., Lau, E. H. E., *et al.* (2005). *Cryst. Eng. Comm.*, *7*, 139.
[19] Alshahateet, S. F., Bishop, R., Craig, D. C., & Scudder, M. L. (2001). *Cryst. Eng. Comm.*, *3*(55), 264.
[20] Alshahateet, S. F., Bishop, R., Craig, D. C., Kooli, F., & Scudder, M. L. (2008). *Cryst. Eng. Comm.*, *10*, 297.
[21] Alshahateet, S. F., Ong, T. T., Bishop, R., Kooli, F., & Messali, M. (2006). *Cryst. Growth Des.*, *6*(7), 1676.
[22] Alshahateet, S. F., Bishop, R., Craig, D. C., & Scudder, M. L. (2003). *Cryst. Eng. Comm.*, *5*(74), 417.
[23] Kalita, D., Deka, H., Samanta, S. S., Guchait, S., & Baruah, J. B. (2011). *J. Mol. Struct.*, *990*, 183.
[24] Bertz, S. H. (1985). *J. Org Chem.*, *50*, 3585.
[25] Alshahateet, S. F. (2011). *J. Chem. Crystallogr.*, *41*, 570
[26] Alshahateet, S. F. (2011). *Jordan J. Chem.*, *6*(1), 21.
[27] SMART and SAINT. (1996). *Software Reference Manual*, Version 4.0, Siemens Energy and Automation, Inc., Analytical Instrumentation, Madison, WI, USA.
[28] Sheldrick, M. G. (1996). *SADABS Software for empirical absorption correction*, University of Göttingen, Germany.
[29] SHELXTL (1996). *Reference Manuals*, Version 5.03, Semens Energy and Automation, Inc., Analytical Instrumentation, Madison, WI, USA.
[30] Alshahateet, S. F., Al-Zereini, W. A., & Alghezawi, N. M. (2011). *J. Chem. Crystallogr.*, *41*, 1807.
[31] Zolfigol, M. A., Salehi, P., Ghaderi, A., & Shiri, M. (2007). *J. Chinese Chem. Soc.*, *54*, 267.
[32] Jia, C.-S., Zhang, Z., Tu, S.-J., & Wang, G.-W. (2006). *Org. Biomol. Chem.*, *4*, 104.
[33] Aguado, F., Badıa, A., Banos, J. E., Bosch, F., Bozzo, C., *et al.* (1994). *Eur. J. Med. Chem.*, *29*, 205.
[34] Alshahateet, S. F., Alghezawi, N. M., & Bishop, R. (2011). *Jordan J. Chem.*, *6*(2), 139.
[35] Desiraju, G. R., & Steiner, T. (1999). *The Weak Hydrogen Bond in Structural Chemistry and Biology*, Oxford Science Publications: Oxford.
[36] Boenigk, D., & Mootz, J. (1998). *J. Am. Chem. Soc.*, *110*, 2135.
[37] Cowan, J. A., Howard, J. A. K., Mclntyre, G. J., Lo, SM-F., & Williams, I. D. (2003). *Acta Crystallogr. B*, *59*, 794.
[38] Bis, J. A., & Zaworotko, M. J. (2005). *Cryst. Growth Des.*, *5*(3), 1169.