Synthesis of an Octacyclic C$_{60}$ Fragment

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Fragments of buckminsterfullerene (C$_{60}$) include the monumental three compounds corannulene, sumanene, and truxene. These three have served as leading molecules in ongoing research for curved, fused, and π-extended polyaromatic materials. Achieving more structural variations that join the ranks of these three archetypes remains challenging. Herein we report synthesis of an octacyclic hydrocarbon that is an unexplored C$_{60}$-fragment, namely, a 4,11-dihydrodiindeno[7,1,2-ghi:7',1',2'-pqr]chrysene (C$_{28}$H$_{16}$, which we named *Metelykene*). The key to success was solution-compatible synthesis in which double pentagonal rings flank hexagonal ones. This solution-phase approach, coupled with the resulting non-planar π-conjugation, is so straightforward that it offers an entry to a derivative such as a cardo aromatic monomer.

Introduction

Fragments of buckminsterfullerene (C$_{60}$) have played a leading role in material science, because their chemical properties stimulate physics and biology as well as chemistry in diverse potential applications and detailed basic studies.[1] The three most well-known fragments include bowl-shaped hexacyclic "corannulene," heptacyclic "sumanene" and non-bowl-shaped heptacyclic "truxene" and are arguably monumental achievement (Figure 1(a)).[2–4] Although there are other ways to create patterns of fragment, few molecules except the aforementioned three are easily obtainable.[5–8] One continuing point of trouble is the solution-phase symmetric construction of pentagons right besides hexagons.[9–10] About 40 years have passed since fullerene chemistry began, many unsuccessful attempts have been noted towards partial solution-phase synthesis and so major challenges remain.[11]

We explored the solution-phase synthesis of an *unexplored* C$_{60}$-fragment, that is 4,11-dihydrodiindeno[7,1,2-ghi:7',1',2'-pqr]chrysene (**1**, C$_{28}$H$_{16}$) (Figure 1(b)). As the name was lengthy during the course of our study, we named **1** "*Metelyekene*" for a close resemblance to shape of *metelyk* that means a butterfly in
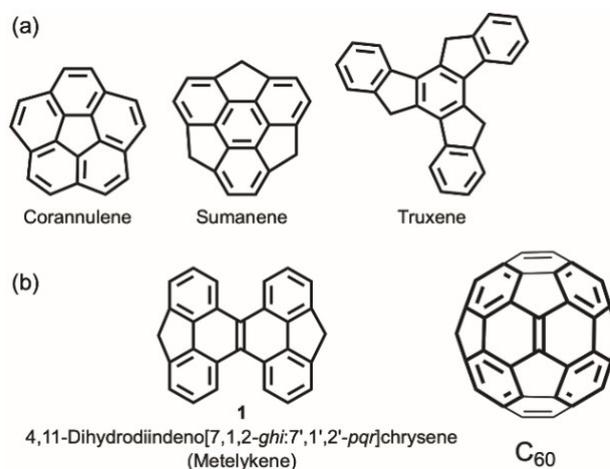

Figure 1. C$_{60}$-fragments: (a) historically monumental molecules, hexacycle "corannulene", heptacycle "sumanene", and heptacycle "truxene", and (b) an unexplored octacycle, 4,11-dihydrodiindeno[7,1,2-ghi:7',1',2'-pqr]chrysene (C$_{28}$H$_{16}$) **1** that is given the nickname "*metelykene*". **1** corresponds to the bold lines in C$_{60}$ illustration.

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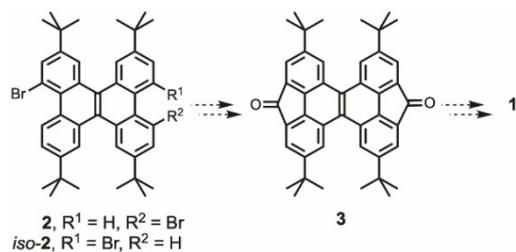📄 Supporting information for this article is available on the WWW under
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Ukrainian. Unlike the three fragments mentioned above, the point group symmetry of octacycle **1** in the ideal planar structure must be D_{2h}. Although **1** seems to be a simple non-bowl-shaped molecule, there is no report of its synthesis: to the best of our knowledge, SciFinder showed no hit on April 2023. Our synthetic strategy focuses on a solution-compatible approach to **1** (Figure 2), wherein the important starting precursor is a soluble hexacyclic dibenzo[g,p]chrysene **2**/iso-**2**. Of interest are: a) the four *tert*-butyl groups provide solubility and b) two bromine atoms provide for synthetic opportunities.[12] This precursor enabled straightforward formation of double pentagons right besides hexagons for giving a key diketone intermediate **3** that easily lead to make a cardo-typed aromatic molecule as well as **1**.

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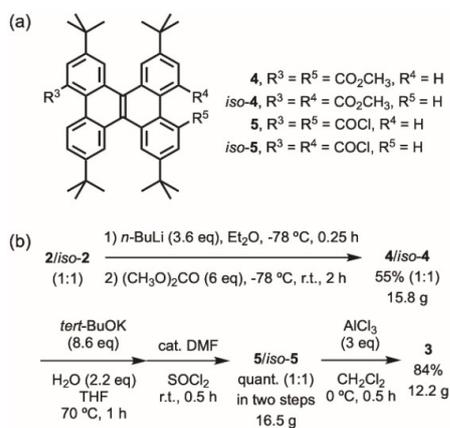**Chemistry Europe**
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Figure 2. Potential scheme to synthesize the octacycle **1** from the starting hexacycle **2**/iso-**2**. A key octacyclic intermediate is **3**.

Scheme 1. Synthesis of octacycle **3**: (a) synthetic intermediates of hexacycles **4**, iso-**4**, **5** and iso-**5**, and (b) reaction paths to diketone **3** from starting precursor of dibenzo[g,p]chrysenes **2**/iso-**2**.
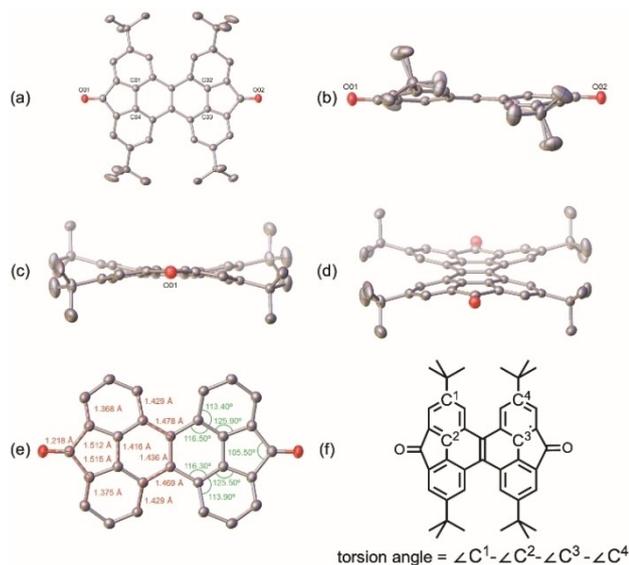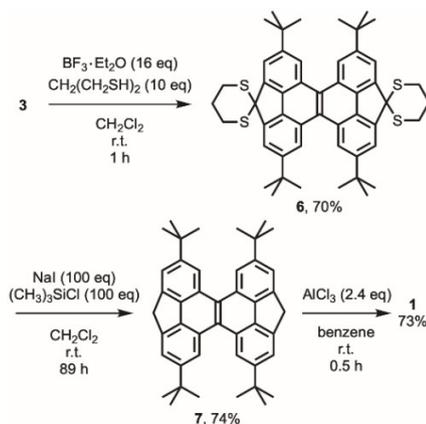

Figure 3. ORTEP drawing of **3** with thermal ellipsoids at the 50% probability level (the hydrogen atoms are omitted for clarity). Red colored-atoms are for oxygens: (a) top view, (b) side view from a *cove* region, (c) side view from a carbonyl moiety, (d) side view from a slanting upper part, (e) top view with a description of selected bond lengths and angles (*tert*-butyl groups are removed for ease of viewing), (f) torsion angle, 20.83(12)°, determined by the four carbon atoms of C^1, C^2, C^3, and C^4.

Results and Discussion

At the outset of our study, synthesis of key intermediate **3** was elaborated (Scheme 1). The starting **2** and iso-**2** were prepared as a mixture of 1:1 molar ratio, according to our previous report.[13] Solution-phase activation of two bromine atoms in **2**/iso-**2** by organolithium reagent proceeded in 55% yield (15.8 grams) of an ester **4**/iso-**4**. Then, hydrolysis of the ester yielded the corresponding carboxylic acid, which was consecutively followed by transformation into an acid chloride **5**/iso-**5** in quantitative yield (16.5 grams). Finally, the acid chloride underwent $AlCl_3$-mediated intramolecular Friedel-Crafts acylation to yield 84% of diketone **3** (12.2 grams). The molecular structure of **3** was determined by crystallographic analysis, which made apparent its octacycle with contorted pi-conjugations (Figure 3).[14] The octacycle constructs two bridges through two sp^2–carbons of carbonyl substructures (part (a)), and a saddle-shaped topology was also found (part (b), (c) and (d)). Selected bond lengths and angles were depicted in part (e), in which the *cove* space is expanded with the mentioned angles of about 113.40° and 116.50° presumably due to tying aryls with two five-membered rings. The torsion angle of **3** was checked according to the part (f), which exhibited 20.83(12)°. The interlayer distance in packing view was 3.461 Å (Figure S1 in Supporting Information).

Given the opportunity of synthesizing the octacyclic fused-ring, we focused on transformation of **3** to **1** (Scheme 2). The two carbonyl groups of **3** were converted into the dithioketals **6** in 70% yield, and the following reductive desulfurization afforded **7** in 74% yield.[15–17] Finally, aluminum trichloride removed four *tert*-butyl groups, yielding **1** in 73%. All those steps were so compatible with solution-phase that the polycyclic intermediates undertook common transformations under mild reaction conditions.[9,18] Crystallographic analysis of **1** confirms the unique structure (Figure 4).[19] The selected bond lengths and angles of **1** were similar to **3** (part (a)): the angle of 102.54° at the methylene sp^3–carbon of **1** narrowed slightly compared to 105.50° at the carbonyl sp^2–carbon of **3**. The

Scheme 2. Three-step-synthesis of **1** from **3**. Firstly, removal of oxygen atoms of **3** by dithioketal formation gave **6**. Then, the following desulfurization reaction afforded **7**. Finally, the dealkylation of **7** formed **1**.

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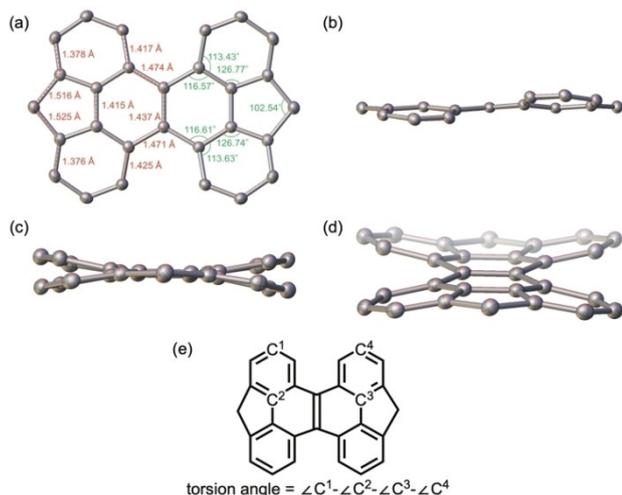European Chemical
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Figure 4. ORTEP drawing of **1** with thermal ellipsoids at the 50% probability level (the hydrogen atoms are omitted for clarity); (a) top view with a description of selected bond lengths and angles, (b) side-view from a *cove* region, (c) side-view from a methylene moiety, (d) side view from a slanting upper part, and (e) torsion angle, 19.89(6)°, determined by the four carbon atoms of C^1, C^2, C^3, and C^4.

gently curved pi-conjugation takes a saddle-shaped appearance, also observed in **3** (part (b), (c) and (d)). The crystal clarified the torsion angle of **1** as 19.89(6)° (part (e)), in which the angle was comparable to that of **3**. This result suggests that substituent effects of twofold carbonyl groups and fourfold *tert*-butyl moieties sparingly impact on the structural torsion.

With a gram-scale protocol of **3** in hand, other transformations were tried. First transformation through activation of the two carbonyl groups: we focused on synthesis of cardo aromatic molecules (Scheme 3).[21–23] Friedel-Crafts reaction between **3** and anisole occurred in 72% yield to give **8**. The molecular structure of **8** was also determined by crystallographic analysis, which made apparent its double cardo structures (Figure 5).[24] The octacycle **8** spreads its fourfold anisole moieties wide over space, and a contorted polyaromatic
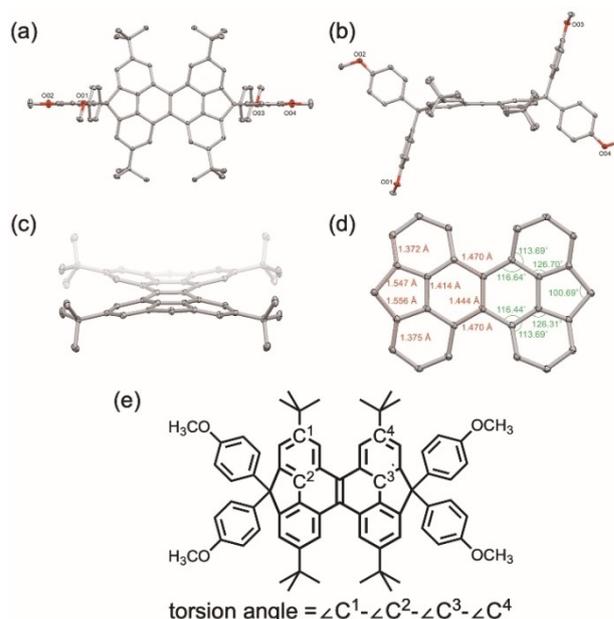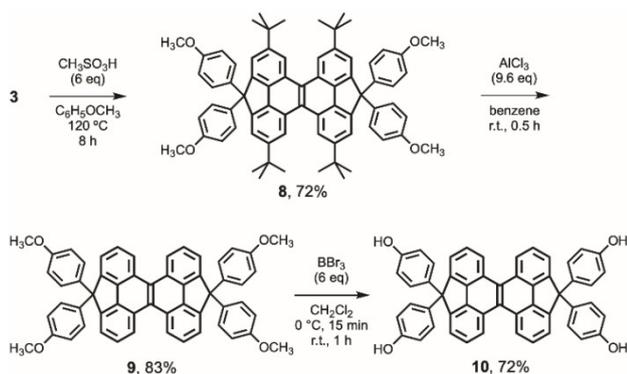

Scheme 3. Access to double cardo-structured molecules **8**, **9**, and **10**. Friedel-Crafts reaction between **3** and anisoles afforded **8**, and the following removal of *tert*-Bu moieties yield **9**, and the final demethylation reaction produced **10**.

Figure 5. ORTEP drawing of **8** with thermal ellipsoids at the 50% probability level (the hydrogen atoms are omitted for clarity): (a) top view, (b) side-view from a *cove* region, (c) side view from a slanting upper part (for ease of viewing, anisole groups are removed and substructures in the foreground are colored darker), (d) top view with a description of selected angles and bond-lengths, and (e) torsion angle, 20.03(11)°, determined by the four carbon atoms of C^1, C^2, C^3, and C^4.

hydrocarbon with the shape of a saddle was observed (part (a), (b) and (c)). The angle of 100.69° at the quaternary carbon of **8** narrowed as compared to that of 102.54° at the carbonyl sp^2–carbon of **1** (part (d)). The dihedral angle of **8** according to the part (e) displayed 20.03(11)°, which was comparable to the angles of **1** and **3**. The saddle-shape turned out to be common to three octacycles **1**, **3**, and **8**. Then, removal of *tert*-butyl groups of **8** produced tetra-methoxy **9** in 83% yield, and **9** was finally transformed into tetrol **10** in 78% yield.

DFT calculations were performed to obtain information on the frontier orbitals of the unsubstituted **1**, diketone **3**, and cardo-structured **8** at the B3LYP/6-31G(d,p) level of theory.[20] Structural optimization of **1** and **3** was performed with C_{2h} symmetry, and **8** was performed with C_i symmetry (Figure S4). The obtained structures were almost reproduced in the X-ray crystal structures in the bond lengths and torsion angles at the cove region (Figure S4). The bond length of the C—Ar bond from the carbonyl carbon atom in **3** obtained in the calculation is shorter than the corresponding lengths obtained from the X-ray crystal structure, likely to be due to the packing. Their frontier orbitals mappings and their energies and HOMO-LUMO energy gaps are summarized in Figure 6. The HOMO and LUMO orbitals of **1** and **8** are similar (part (a) and (c)) and both originate from the dibenzo[*g,p*]chrysene backbone. On the other hand, the LUMO of **3** is strongly influenced by the carbonyl group, whereas the HOMO is derived from the dibenzo[*g,p*]chrysene skeleton (part (b)). Both HOMO and LUMO energies of **3** were lower than those of **1** due to the electron-

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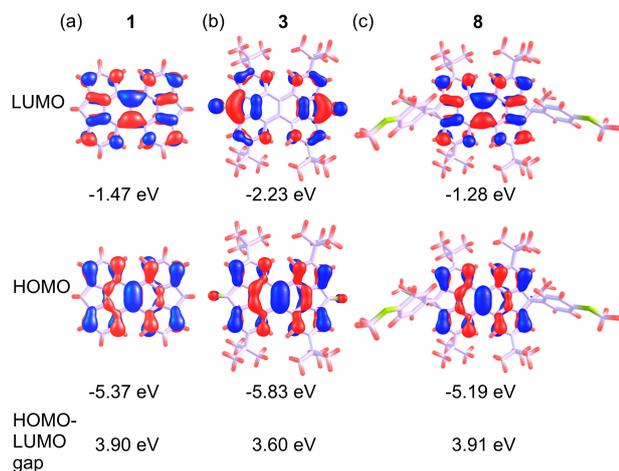Chemistry
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Figure 6. Frontier orbitals mappings and the energies for (a) **1**, (b) **3**, and (c) **8** calculated at the B3LYP/6-31G(d,p) level of theory.
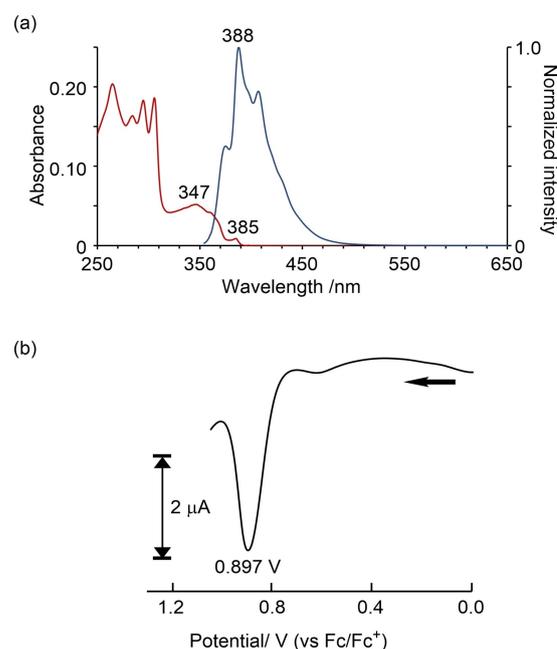

Figure 7. (a) Absorption (red line) and emission (blue line) spectra of **1** (10 μM in CH_2Cl_2, excitation wavelength: 347 nm), (b) square wave voltammogram of **1** in CH_2Cl_2 (1.0 mM) including 50 mM NBu_4BF_4 as a supporting electrolyte under argon at 25 °C (working electrode: Pt).

withdrawing nature of the carbonyl groups. In particular, the LUMO of **3** was significantly decreased by 0.76 eV compared to **1** (HOMO was reduced by 0.46 eV), thereby reducing the HOMO-LUMO gap (3.60 eV).

Our previously reported structures of DBC derivatives, including a silicon-bridged one, have a twisted naphthalene moiety with D_2 symmetry (twisted form).[12] On the other hand, the X-ray crystal structures of the newly synthesized compounds **1**, **3**, and **8** showed that the two phenanthrene moieties are curved in opposite directions (saddled form), and the symmetry of **1** and **3** is approximately C_{2h} (the symmetry of **8** is approximately C_i). To investigate this difference, we performed structural optimization of compounds **1** and **3** for structures with D_2 and C_{2h} symmetry by DFT calculation and compared their energies. The results revealed that the twisted forms with D_2 symmetry were slightly more stable (~1.9 kcal/mol) than the saddled forms with C_{2h} symmetry for **1** and **3** (Figure S4 and S5, and Table S1). Therefore, the saddled forms obtained in the X-rays may be due to intermolecular interactions in packing.

The spectroscopic properties of **1** were investigated using UV/Vis absorption and photoluminescence measurements (Figure 7). In the absorption spectrum, a broad absorption around 350 nm and a peak at 385 nm were observed. In the emission spectrum, the peaks with the vibrational structure were observed ($\lambda_{max} = 388$ nm), which is considered to be due to the right skeleton. The absolute photoluminescence quantum yield was 8%.

The electrochemical properties of compound **1** were investigated. The voltammograms exhibited irreversible redox processes at slow scan rates (50–500 mV/s) (Figure S6(a)-(d)). Decomposition at benzylic positions is considered to occur under the conditions. The redox process was almost reversible when the scan rate increased to 1000 mV/s (Figure S6(e)). The oxidation potential obtained from the square wave to voltammogram was 0.897 V (vs Fc/Fc$^+$, Figure 7(b)).

Conclusions

In conclusion, the straightforward synthesis of a C_{60}-fragmentary *metelykene* **1** has been achieved. The formation of two five-membered rings and crystallographic characterization of the octacycles stand alone in the area of synthetic organic chemistry. Our results suggest the following three salient features: One, the key precursor **3** was built properly through productive solution-phase protocols, in which the intramolecular double cyclization plays a vital role. Two, **3** was transformed into a C_{60}-fragmentary hydrocarbon **1** that was analyzed with crystallographic technique revealing the same saddle-shaped topology as **3**. Three, DFT calculations of **1**, **3**, and **8** revealed the information on frontier orbitals. The clear differences between those HOMO/LUMO levels were observed. These results emphasize the relevance of the octacyclic architecture to design new and potent polyaromatic materials unachievable heretofore: Particularly, there are many more variations of chemical transformations to try on solution-processable **3**. In these endeavors we look forward to reporting on synthetic and material advantages of the peacefully sounded *metelykene*.

Experimental Section

Synthesis of 4,11-dihydrodiindeno[7,1,2-*ghi*:7′,1′,2′-*pqr*]chrysene (**1**): Under an argon atmosphere, to a suspension of **7** (1.4 g, 2.4 mmol) in dry benzene (38 mL) was added aluminum chloride (770 mg, 5.8 mmol). After stirred at room temperature for 0.5 h, the reaction mixture was quenched with H_2O (60 mL) at 0 °C. The aqueous layer was extracted with CH_2Cl_2 (30 mL x 3), and combined

organic phases were washed with brine (60 mL), dried over Na_2SO_4, filtered, and concentrated *in vacuo* to give 1.2 g of crude products. Purification by short-plugged silica-gel column chromatography (hexane/CH_2Cl_2, 9:1) gave 624 mg (73%) of **1** as white (colorless) solid materials. Rf value 0.35 (hexane/toluene, 9:1); M.p. 268–274 °C; ^1H NMR (400 MHz, $CDCl_3$) 9.11 (dd, $J = 6.4$ Hz, 6.4 Hz, 4H, H-2), 7.85–7.81 (m, 8H, H-1, H-3), 4.47 (s, 4H, CH_2) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) 142.1 (C-3a), 138.7 (C-3a^1), 129.1 (C-14b), 128.2 (C-14c), 127.7 (C-2), 124.6 (C-3), 122.1 (C-1), 37.8 (CH_2) ppm; MS (DART-TOFMS) *m/z*: 353 [MH]$^+$; IR (neat) 2923, 1494, 1442, 1418, 1393, 1085, 1027, 937, 821, 767, 708, 619, 475 cm^{-1}; HRMS (DART-TOF) calcd. for $C_{28}H_{17}$: 353.1325 [MH]$^+$, found: 353.1314; Anal. Calcd. for $C_{28}H_{16}$; C, 95.42; H, 4.58. Found: C, 95.43; H, 4.43.

Crystallographic data: Deposition Numbers 2207864 (for **1**), 2160146 (for **3**), and 2129619 (for **8**) contain the supplementary crystallographic data for this paper. These data are provided free of charge by the joint Cambridge Crystallographic Data Centre and Fachinformationszentrum Karlsruhe Access Structures service.

Conflict of Interests

The authors declare no conflict of interest.

Data Availability Statement

The data that support the findings of this study are available in the supplementary material of this article.

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[14] The single crystal of **3** was prepared by slow evaporation of CH_2Cl_2/hexane (1 mL/2 mL) solution of the sample (3 mg), and packing view is Figure S1 in Supporting Information, CCDC-2160146. Monoclinic, space group P 1 2/c 1, colorless, $a = 13.8253(3)$ Å, $b = 6.6719(2)$ Å, $c = 19.4490(5)$ Å, $\alpha = 90°$, $\beta = 90.696(2)°$, $\gamma = 90°$, $V = 1793.86(8)$ Å^3, $Z = 2$, $T = 93$ K, $d_{calcd.} = 1.277$ g cm^{-3}, μ(Mo-$K\alpha$) = 1.914 mm^{-1}, $R_1 = 0.0733$, $wR_2 = 0.2156$, GOF = 1.067.

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[19] The single crystal of **1** was prepared by slow evaporation of CH_3CN (6 mL) solution of the sample (3 mg), and packing view is Figure S2 in Supporting Information, CCDC-2207864 (for **1**). Monoclinic, space group P 1 21/c 1, colorless, $a = 7.1544(3)$ Å, $b = 13.1937(5)$ Å, $c = 8.8116(4)$ Å, $\alpha = 90°$, $\beta = 104.091(4)°$, $\gamma = 90°$, $V = 806.73(6)$ Å^3, $Z = 4$, $T = 93$ K, $d_{calcd.} = 1.451$ g cm^{-3}, μ(Mo-$K\alpha$) = 0.627 mm^{-1}, $R_1 = 0.0450$, $wR_2 = 0.1329$, GOF = 1.134.

[20] Density functional theory (DFT) calculations were performed at B3LYP/6-31G(d,p) using the Gaussian 16 suite program (G16RevC.01); see, M. J. Frisch, G. W. Trucks, H. B. Schlegel, G. E. Scuseria, M. A. Robb, J. R. Cheeseman, G. Scalmani, V. Barone, G. A. Petersson, H. Nakatsuji, X. Li, M. Caricato, A. V. Marenich, J. Bloino, B. G. Janesko, R. Gomperts, B. Mennucci, H. P. Hratchian, J. V. Ortiz, A. F. Izmaylov, J. L. Sonnenberg, D. Williams-Young, F. Ding, F. Lipparini, F. Egidi, J. Goings, B. Peng, A. Petrone, T. Henderson, D. Ranasinghe, V. G. Zakrzewski, J. Gao, N. Rega, G. Zheng, W. Liang, M. Hada, M. Ehara, K. Toyota, R. Fukuda, J. Hasegawa, M. Ishida, T. Nakajima, Y. Honda, O. Kitao, H. Nakai, T. Vreven, K. Throssell, J. A. Montgomery, Jr., J. E. Peralta, F. Ogliaro, M. J. Bearpark, J. J. Heyd, E. N. Brothers, K. N. Kudin, V. N. Staroverov, T. A. Keith, R. Kobayashi, J. Normand, K. Raghavachari, A. P. Rendell, J. C. Burant, S. S. Iyengar, J. Tomasi, M. Cossi, J. M. Millam, M. Klene, C. Adamo, R. Cammi, J. W. Ochterski, R. L. Martin, K. Morokuma, O. Farkas, J. B. Foresman, D. J. Fox, Gaussian, Inc., Wallingford CT, 2019.

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[24] The single crystal of **8** was prepared by slow evaporation of CH_2Cl_2/CH_3CN (1 mL/1 mL) solution of the sample (3 mg), and packing view is Figure S3 in Supporting Information, CCDC-2129619. Orthorhombic, space group P −1, colorless, $a = 11.2861(4)$ Å, $b = 11.5074(4)$ Å, $c = 12.4187(4)$ Å, $\alpha = 76.728(3)°$, $\beta = 75.229(3)°$, $\gamma = 80.630(3)°$, $V = 1508.56(9)$ Å^3, $Z = 2$, $T = 93$ K, $d_{calcd.} = 1.289$ g cm^{-3}, μ(Mo-$K\alpha$) = 2.179 mm^{-1}, $R_1 = 0.0721$, $wR_2 = 0.2178$, GOF = 1.086.

RESEARCH ARTICLE

"Metelykene"   **C60 fragment**
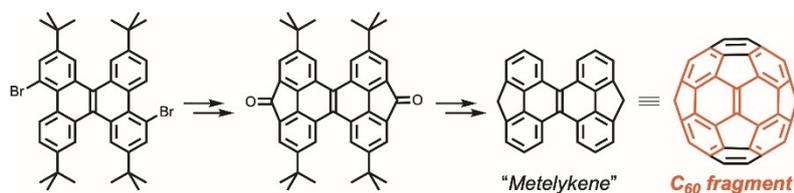
Synthesis of an unexplored C60-fragmentary octacycle (C28H16; 4,11-dihydrodiindeno[7,1,2-*ghi*:7′,1′,2′-*pqr*]chrysene), which we named *Metelykene* (derived from "metelyk" that means butterfly in Ukrainian) is described. The key precursor is a solution-processable hexacycle having fourfold *tert*-butyls and twofold bromines. The bromines were substituted with carbonyls, and the following cyclization yielded the C60-fragment core, unattainable so far.

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Synthesis of an Octacyclic C60 Fragment