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SYNTHESIS AND STRUCTURE OF DICYCLOPENTENOU-RANOCENE, U[C8H6(CH2)3]2

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Allan Zalkin, David H. Templeton, Wayne D. Luke, and Andrew Streitwieser, Jr.

August 1981

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SYNTHESIS AND STRUCTURE OF

DICYCLOPENTENOURANOCENE, $U[C_8H_6(CH_2)_3]_2$

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Abstract

The title compound 1 was synthesized by the reaction of dipotassium bicyclo[6.3.0]undeca-2,4,6-triene-1,8-diide, 3, and UCl₄, and its crystal and molecular structure was determined by single crystal X-ray diffraction. The compound crystallizes in the orthorhombic space group Pbca with 8 molecules in the unit cell with dimensions $\underline{a} = 17.393(8)$ Å, $\underline{b} = 22.468$ (12) Å, $\underline{c} = 8.931$ (4) Å. The uranium atom is located centrally between the two 8-membered rings with bond distances U-C = 2.64 ± 0.03 Å and C-C = 1.40 ± 0.02 Å. The effects of annulation on the physical properties of uranocene are discussed.

Introduction and Results

As part of a continuing study of annulated derivatives^{1,2} of uranocene³ we report the synthesis and X-ray structure determination of bis- π -(cyclopenteno-[8]annulene)uranium(IV) (dicyclopentenouranocene), 1.4 This compound has significance for two reasons. First, it was expected to involve a conformation sufficiently well defined to assist nmr interpretation. Second, it would be the first X-ray structure of an unstrained uranocene in which the ring-U axis is not a C₂ symmetry axis; the position of the uranium was therefore expected to provide a significant indication of the relative roles of covalent and ionic bonding. The title compound was synthesized in a manner similar to dicyclobutenouranocene,² shown in Scheme 1. The bicyclotriene 2 was prepared by ether addition of 1,3-dibromopropane, or the dimethanesulfonate of 1,3-propanediol, to a solution of dilithium cyclooctatrienediide in liquid ammonia, affording distilled yields of 46.8% and 58.4%, respectively. This compound was contaminated by the tricyclic isomer 4; on standing, 2 rearranged completely to 4. Dideprotonation of 2 with potassium amide in THF/liquid ammonia formed a red solution of the dianion, 3, which could be isolated as an impure tan solid. Subsequent reaction of 3 with UCl₄ in THF produced the desired uranocene, 1, in 15.9% yield. The visible spectrum of this green air-sensitive material exhibited the typical uranocene cascade of four principal absorptions at 632, 656, 663 and 680 nm. The pricipal IR absorption bands are compared with those of uranocene in Table I.

The ¹H NMR (toluene- \underline{d}_8) spectrum of <u>1</u> at 30°C showed six sharp well-resolved resonances upfield from TMS, -8.3 ppm (m, 2H), -18.8 ppm (m, 4H), -23.1 ppm (s, 4H), -32.6 ppm (m, 2H), -34.2 ppm (s, 4H), -41.2 ppm (s, 4H), and one resonance downfield from TMS, +24.4 ppm (m, 4H). The ¹³C NMR spectrum (dioxane- \underline{d}_8) at 39°C showed four broad peaks at 308.0, 296.9, 279,0, and 268.5 ppm downfield from TMS and two sharp peaks at 13.4 and -32.5 ppm.

Magnetic susceptibility measurements on the bulk solid from 2.4 to 95.6°K are shown in Fig. 1. Above 20°K the magnetic susceptibility follows the Curie-Weiss Law with C=0.743 \pm 0.005 emu °K mol⁻¹ μ =2.4 \pm 0.1 B.M. and θ =16.6° \pm 0.5°. Below 10°K the magnetic moment was independent of temperature with χ m=2.56 \pm 0.03 x 10⁻² csu/mole. Using a diamagnetic correction of -187 x 10⁻⁶ emu mol⁻¹ the corrected values are C=0.714 \pm 0.005 emu °K mol⁻¹, μ =2.4 \pm 0.1 B.M. and θ =16.1° \pm 0.5°.

The compound crystallizes in the orthorhombic space group Pbca with 8 molecules in the unit cell with dimensions <u>a</u>=17.393(8) Å, <u>b</u>=22.468(12) Å and <u>c</u>=8.931(4) Å. With a molecular weight of 526.46 the calculated density is 2.004 g cm⁻³. The structure was determined by conventional single crystal X-ray methods and was refined by full-matrix least-square to an R factor of 0.04 for 1000 data where $F^2>3\sigma$. Final positional parameters are given in Table II. Tables of calculated positional parameters, and observed structure factors amplitudes are given in the Supplementary Material. Uranium-carbon distances are listed in Table III. The atom numbering is seen in Figures 2 and 3.

Discussion

The title compound exists as discrete molecules in the solid state. All atoms are in the general positions and no symmetry is imposed on the molecule by the space group; however, the molecule does have approximate C, symmetry (see Fig. 3). The uranium atom is centrally sandwiched between the two 8-membered rings with a U-ring distance of 1.92 Å, in good agreement with other uranocenes.^{1,6,7,8} The COT rings of the molecule are rotated about 8° from a staggered configuration (see Fig. 3). In other uranocenes both staggered 11,12 and eclipsed 1,10,11 configurations have been reported. As in ferrocenes, the relative orientation of the rings in uranocene crystals results from crystal packing and not from significant steric interactions between the rings which are 3.85 Å Bond angles and distances for the uranocene part of the apart. molecule are similar to those reported for other uranocenes. 1,6,7,8 The mean planes of carbons, (C_{11}, C_9, C_1, C_8) and (C_{20}, C_{22}, C_{12}) C19), are bent slightly inwards toward the uranium atom from the plane of the 8-membered ring as has been found in dicyclobutenouranocene¹ and in 1,3,5,7,1',3',5',7-octamethyluranocene.⁷ Similar convex distortions in the carbocyclic rings have been observed in ferrocenes, chromacene⁹ and in a variety of organometallic-compounds with planar five, six- and eight-membered rings.¹⁰

Unrealistic bond distances (1.38 to 1.46 Å) and very large and anisotropic thermal parameters in the 5-membered rings indicate that we are observing the mean positions of atoms which have disorder up and down from the mean planes, a situation which is commonplace for aliphatic 5-membered rings.

This bending appears to be a general feature in sandwich structures where steric interactions between rings is small. Two possible explanations have been offered to account for this bending: 7 1) bending the substituent toward the metal makes each carbon slightly more pyramidal with the π orbital of the $C_n{}^H{}_n$ ring bent inward toward the metal and providing greater directionality for overlap between π orbitals and metal orbitals; 2) contraction in volume of the electron density on the side of the ring adjacent to the highly charged metal ion. Non-bonding interaction between the substituted bond and the more diffuse electron density on the uncomplexed side of the ring would result in an inward bend of the substituent. This latter explanation suggests that the inward bending should be independent of ring size. However, it appears that for 3- and 4-membered rings the substituents bend outward away from the metal.10 Theoretical calculations on d-transition metal compounds attribute this bending to the former explanation and predict an outward bend for 3- and 4-membered ring and an inward bend in the substituent for rings larger than 5.10 This bending of the substituent in toward the metal appears to be a general feature in all substituted uranocenes in the absence of steric effects and may reflect the proposed covalency in ligandmetal bonding in these systems.¹¹

An especially important point in this regard is the central position with respect to the C_8 rings of uranium in all uranocene structures thus far studied. This result is expected for a model involving important covalent ring-metal bonding. If ionic

character dominated we would expect some deviations from C_8^- centrosymmetry, particularly in a case such as the present where the overall structure is so lacking in symmetry. This result makes the present structure an especially significant one.

The spectral properties of 1 show no significant differences from those of other uranocenes. In the visible spectrum the shift in λ_{\max} from that of uranocene, (λ_{\max} 616 nm), is between that of 1,1'-dialkyl substituted uranocenes, (λ_{\max} 610-625 nm), and 1,3,5,7,1',3',5',7'-octamethyluranocene (λ_{\max} 650 nm), in accord with the proposed charge transfer model for the visible spectrum. ^{12,13}

The paramagnetic shifts of the ¹H NMR resonances of 1 are similar to those reported for other substituted uranocenes.¹¹ Due to the paramagnetic center, the endo and exo protons in the α and β positions of the annulated rings are non-equivalent. The three non-equivalent ring proton positions could be assigned to the resonances at -32.1, -34.2 and -41.2 ppm, from the lack of splitting due to J-J coupling and their larger line widths (ca. 30 Hz). The remaining four resonances were partially resolved multiplets and could be easily differentiated into α (+24.4, -18.8 ppm) and (-8.3, -32.6 ppm) sets by integration. Further assignment into endo and exo sets was made in the following manner.

In uranocene¹⁴ and 1,3,5,7,1',3',5',7'-octamethyluranocene¹⁵ the paramagnetic shifts have been factored into contact and pseudo contact components.¹¹ The contact component affords an upfield shift to ring protons, and an alternating pattern of upfield and downfield shifts to substituent protons such that protons on the

 β , δ , etc., carbons are shifted upfield and protons on the α , γ , etc., carbons are shifted downfield, with the magnitude of the contact interaction rapidly diminshing to zero along the substituent chain. Variable temperature studies on uranocene and octamethyl-uranocene have established that the total paramagnetic shift is linear with 1/T. Consequently, the pseudo-contact contribution can be expressed by

$$\Delta_{\text{Pseudo-contact}} = \frac{(\chi_z - 1/2 \chi_x - 1/2 \chi_y)(3 \cos^2\theta - 1)}{3} - \frac{(\chi_z - \chi_y)(\sin^2\theta \cos 2\psi)}{2} (1)$$
Where symmetry requires $\chi_x = \chi_y$, the latter term is zero.¹⁶

For a number of unsymmetrical uranocenes it appears that $\chi_x = \chi_y$ and that the last term in eq. 1 can be generally neglected.¹¹ With this assumption, the sign of the pseudo-contact shift will be controlled by θ . Similar to dicyclobutenouranocene we expect the contact shift of the α_{exo} and α_{endo} protons to be approximately the same; calculation from the observed structure gives $\theta < 54.74^{\circ}$ for the α_{exo} protons and thus, the pseudo-contact shift is downfield. Hence, the α proton resonances are assigned as $\alpha_{exo} + 24.4$ ppm and α_{endo} -18.8 ppm; by analogy, the β -proton resonances are β_{exo} 8.3 ppm and β_{endo} -32.6 ppm.

For ¹³C NMR the contact shift model for uranocene¹⁵ predicts that the contact shifts for carbons should be just opposite those of the corresponding protons. Ring carbons are shifted to low field, and are assigned tentatively to the four resonances at 308.0, 296.9, 297.0, 268.5 ppm. Correspondingly, the α carbon is assigned to the upfield resonance (-32.5 ppm) and the β carbon to the 13.4 ppm resonance.

The similarity of the ¹H and ¹³C resonances in 1 with those of other substituted uranocenes ^{12,13} and the parent compound ¹² itself suggest that the electronic factors giving rise to the paramagnetic shifts are similar in all uranocenes. Further support for this generalization comes from the magnetic susceptability data which shows that the magnetic moment of 1 is equal within experimental error to that of uranocene.^{5,17} Further analysis of the nmr spectra of 1 and related annulated uranocenes, their temperature dependences and dissection into contact and pseudocontact components will be detailed in another paper now in preparation.

Experimental

All reactions requiring air-free anhydrous conditions were conducted under an Ar atmosphere or in a Vacuum Atmospheres recirculating glove box. Solvents were distilled from CaH₂ and degassed prior to use. Visible spectra were obtained on a Cary 118 spectrometer, infra-red spectra on a Perkin Elmer 297 spectrometer, ¹H and NMR on Varian T-60 (60 MHz) or the V.C. Berkeley FT-NMR system (180 MHz), and ¹³C NMR on a Brucker TT-23 spectrometer. Mass spectra and elemental analysis were performed by the Analytical Services Laboratory, University of California, Berkeley. Magnetic susceptability measurements were measured on a vibrating-sample magnetometer previously described in the literature.¹⁷

<u>X-ray diffraction</u>. Single crystals suitable for X-ray analysis were grown from hot hexane in an Ar atmosphere glove box. A crystal fragment approximately 0.10 x 0.15 x 0.35 mm was placed on a Picker FACS-I automated diffractometer equipped with graphite monochromated MoKa radiation, (λ =0.70930 Å). The setting angles of 12 manually centered reflections (35° < 20 < 40°) were used to determine the cell parameters by least-squares.

Intensity data were collected using the θ -2 θ scan technique with a scan speed of $2^{\circ}/\min$ on 2θ . Each reflection was scanned from 0.65° before the Ka, peak to 0.65° after the Ka, peak, and backgrounds were counted for 4s at each end of the scan range, offset by 0.5°. The temperature during data collection was 22 ± 1°C. Three standard reflections (6,0,0; 0,0,4; 0,10,0) were measured after every 200th scan. At the beginning of the data collection ω scans of the 400, 060, and 002 reflections showed half-widths of 0.09°, 0.07°, and 0.09° respectively; at the end of the data collection the values were 0.18, 0.12 and 0.11, indicating significant deterioration of the crystal. Although 6043 scans were collected, only the first 3010 were used; the remaining half of the data, based on the declining intensities of the standards, were rejected. The decay was anisotropic with the largest amount recorded by the 600 standard reflection. An isotropic decay correction varying from 0.91 to 1.06 was applied to the 3010 data An absorption correction¹⁸ was estimated and corrections used. between 1.5 and 1.8 were applied to the data. Because the crystal had an irregular shape with indistinct faces, a shape and size

were estimated, and the dimensions were tailored to fit the intensity variations obtained from three azimuthal scans taken after the data collection was concluded. The 3010 scans resulted in 2312 unique data 1000 of which have $F^2 > 3\sigma$.

A three-dimensional Patterson function calculation revealed the uranium position, and subsequent least-squares calculations and Fourier maps revealed all of the carbon atoms. A series of least squares in which the function $\Sigma w (|Fo|-|Fc|)^2 / \Sigma w F_o^2 was$ minimized converged rapidly to the final structure. The expressions that were used in processing the data and estimating the weights are given in the supplementary material; the "ignorance factor", p, was set to 0.06. Scattering factors from Doyle and Turner¹⁹ were used, and anomalous dispersion corrections²⁰ were applied. The positions of all of the hydrogen atoms were estimated and included in the calculations with isotropic temperature factors, but were not refined. Anisotropic thermal parameters were applied to U and all of the carbon atoms.

Because of the low quality of the data, the cyclooctatetraene (COT) ring was not well resolved, and some of the C-C bond distances deviated from the expected values by as much as 0.2 Å. Restraints were imposed on the bond distances in the COT ring and the cyclopenteno group adjacent to the ring in the following manner:²¹ Interatomic distances between selected atoms were introduced into the least-squares calculations and treated as observations; estimated standard deviations of these distances were also introduced and used to calculate the weights. The derivatives of these distances with respect to the positional parameters were calculated by a special patch and these "observations" were not included in the least-squares calculation in the same manner as the observed structure factors. This procedure allows the structure to adjust to the electron density with a flexibility governed by the weighting. The C-C bond distances within the ring were restrained to 1.40 ± 0.02 Å and the C-C bonds from the cyclopento carbon to the ring carbons were restrained to 1.54 ± 0.02 Å.

The discrepancy indices for 1000 data where $F^2 > 3\sigma$ are

 $R = \Sigma ||F_{0}| - |F_{0}|| / \Sigma |F_{0}| = 0.040$ $R_{W} = [\Sigma W |F_{0}| - |F_{0}|)^{2} / \Sigma W |F_{0}|^{2}]^{1/2} = 0.046$

R for all 2332 data is 0.14. The error in an observation of unit weight is 1.07. In the last cycle no parameter changed more than 0.13 σ . The top three peaks in the final difference Fourier map are 1.0 to 1.3 e A⁻³ and are all ripples about the uranium atom.

<u>cis-Bicyclo[6.3.0]undeca-2,4,6-triene</u>, 2. Under an Ar atmosphere, 1.4 g (0.2 mol) of lithium wire (1% sodium) was added to 300 mL of anhydrous liquid ammonia in a 500 mL round bottom flask. To this deep blue solution was added 10.4 g (0.1 mol) of freshly distilled cyclooctatetraene (33°C/14 mm Hg) (BASF), at -40°C, via syringe. The resulting yellow suspension of dianion was stirred for 1.5 hr at -40°C followed by dropwise addition (1 drop every 2 seconds) of 20.2 g (0.1 mol) of 1,3-dibromopropane

(Aldrich) in 20mL of ether. The reaction mixture was stirred for 4.5 h at ca. -40°C followed by overnight evaporation of ammonia to afford a red brown solid which was suspended in 200 mL of saturated ammonia chloride and extracted with ether (4x100 mL). The ether extracts were washed with water (3x100 mL) and dried over $MgSO_A$. Removal of solvent followed by vacuum distillation (25°C/0.1 mm Hg) yielded 6.83 g (46.8%) of a clear yellow liquid; mass spectrum parent m/e = 156. The ¹H and ¹³C NMR (CDCl₃) of this material indicated that it was mixture of both the bicyclic (2) and tricyclic (4) valence isomers; bicyclic ¹H NMR: δ 5.80 (s, 6H, vinyl), 2.77 (br m. 1.4H, bridgehead), and 1.65 (complex m, 5.3H cyclopentyl); ¹³C NMR: & 135.4, 127.7, 126.2 (vinyl), 43.5 (bridgehead), 32.3, 19.5 (cyclopentyl); tricyclic ¹H NMR (CDCl₃): δ 5.57 (s, 4H, vinyl), 2.77 and 2.43 (br m, 2H, bridgehead), 1.67 (complex multiplet, 5.3H, cyclopentyl); 13 C NMR: δ 126.6, 120.3 (vinyl), 51.0, 35.6 (bridgehead), 32.9, 24.7 (cyclopentyl). After standing for 4 days at room temperature the material had rearranged completely to the tricyclic isomer, 4.

Following the same procedure but using 23.2 g (0.1 mol) of 5 in 100 mL of THF instead of 1,3-dibromopropane in 10 mL of ether, gave the same product in 58.4% yield.

AgNO₃ adduct of cis-bicyclo[6.3.0]undeca-2,4,6-triene. To a boiling solution of 3.4 g (0.02 mol) of silver nitrate and 15 mL of abs ethanol was added 2.9 g (0.02 mol) of 2. Most of the silver nitrate dissolved upon addition of the hydrocarbon. Cooling of the solutuion is a refrigerator for several hrs afforded

· 13

off-white crystals which were recrystallized from absolute ethanol; mp 135-136°C dec.

Anal. Calcd. for C₁₁H₁₄NO₃Ag: C, 41.79; H, 4.46; N, 4.43. Found: C, 41.48; H, 4.35; N, 4.29.

<u>1,3-Bis(methylsulfonyloxy)propane</u>. To a stirred 0°C solution of 15.22 g (0.2 mol) of 1,3-propanediol (Aldrich), 70 mL (0.5 mol) of triethylamine and 1 L of CH_2Cl_2 in a 2 L round bottom flask was added dropwise 32.5 mL (0.42 mol) of methanesulfonyl chloride (Eastman) over 5 min. The resulting reaction mixture was stirred for 0.5h during which time a white ppt formed. After sequential extractions with 200 mL of ice water, 200 mL of cold 10% HCl, 200 mL of saturated sodium bicarbonate and 200 mL of brine, the organic layer was dried over MgSO₄ and stripped of solvent to afford a white solid which was recrystallized from hot methanol; yield 38.4 g (82.7%); mp 40.5-41.5°C; NMR (CDCl₃): δ 4.37 (t, 4H, -OCH₂-), 3.07 (s, 6H,-CH₃), 2.19 (p, 2H, -CH₂-).

Anal. Calcd. for C₅H₁₂O₆S₂: C, 25.85; H, 5.21; S, 27.61. Found: C, 26.04, H, 5.26, S, 27.42.

Dipotassium Bicyclo[6.3.0]undeca-2,4,6-triene-1,8-diide, 3. Under Ar a suspension of potassium amide in liquid ammonia was prepared by distilling 300 mL of ammonia from a lithium metalammonia solution into a 500 mL round bottom flask containing several mg of anhydrous FeCl₃. Subsequent addition of 2.14 g (0.055 mol) of potassium metal at -40°C afforded a blue solution which was stirred (ca 5 min) until the blue color disappeared indicating formation of the amide. A 4.0 g (0.027 mol) aliquot of 2 was added via syringe and the resulting red brown solution was stirred for 1.5 h at -35°C. The solution was slowly warmed to room temperature and the ammonia was swept out with a steady Ar purge overnight, affording 4.66 g of the crude dianion as a highly air sensitive grey brown solid; NMR (THF- \underline{d}_8): ¹H & 5.67, 1.3, 0.93, broad singlets; ¹³C & 99.9 (quat), 96.9, 89.8, 87.0 (ring), 46.4 (α CH₂), 27.2 (β CH₂). No attempt was mady to purify this material.

Dicyclopentenouranocene, 1. In an Ar atmosphere glove box 2.56 g (0.0068 mol) of UCl₄ in 25 mL of THF was added to a solution of 3.0 g (0.0135 mol) of 3 in 100 mL of THF in a 500 mL round bottom flask, and the resulting green solution was stirred for 18 h. Removal of the solvent by vacuum transfer afforded a green solid which was purified by Soxhlet extraction with hexane; yield 0.57 g (15.9%); mass spectrum, parent peak m/e 526; visible spectrum in hexane, nm ($\epsilon x 10^3$) 632(2.1), 656(0.9), 663(0.9), 680(0.7). The ir spectrum is given in Table I and nmr spectra are discussed in the text.

Acknowledgment

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Supplementary Material Available

Data processing formulas, calculated positional parameters for the hydrogen atoms, anisotropic thermal parameters, carboncarbon distances, a table of selected antles, and a list of observed structure factors (14 pages). Ordering information is given on any current masthead.

Table I. Infrared Spectrum (cm⁻¹) in NUJOL

			· · · ·	
(C ₈ H ₈)	2 ^U ≠	[С ₈ н ₆ (Сн ₂) ₃] ₂ U,	1
		1870	(w)	
1730	(w)	1765	(w)	
		1320	(m)	
1262	(s)	1260	(m)	
1095	(s)*	1090	(m) *	
1018·	(s)*	1020	(m) *	
	·	900	(<u>s</u>)	
7 99	(s)	790	(m)	
	· · ·	745	໌(s)	`л
720	(s)	700	(vs)	

(a) w = weak; m = medium; s = strong; vs = very strong

(b) Refs. 11, 12, 22-25.

(c) very broad

Table II. Positional Parameters^a

Atom	<u>`x</u>	<u>y</u>	Z
C(1)	.047(2)	.163(1)	.159(3)
C(2)	.077 (2)	.122(1)	.264(3)
C(3)	.147(2)	.109(2)	.329(4)
C(4)	.218(2)	.138(1)	.309(3)
C(5)	.254(2)	.182(2)	.225(4)
C(6)	. 228 (2)	. 220 (2)	.115(4)
C(7)	.153 (2)	.2305(9)	.061(3)
C(8)	.079(2)	.209(1)	.074(3)
C(9)	.014(2)	.230(1)	032(3)
C(10)	051(3)	. 205 (3)	.029(6)
C(11)	038(2)	.152(2)	.122(6)
C(12)	. 285(1)	.098(1)	160(3)
C(13)	, 222 (2)	.116(1)	245(3)
C(14)	,142(2)	.111(2)	258(4)
C(15)	.099(2)	.067(2)	184(4)
C(16)	.105(2)	.022(1)	078(4)
C(17)	. 168 (2)	003(1)	004(3)
C(18)	. 245(1)	.014(1)	.016(2)
C(19)	. 294 (1)	.054(1)	052(2)
C(20)	.378(1)	.058(2)	.005(3)
C(21)	. 407 (2)	.111(2)	061(5 <u>)</u>
C(22)	.361(2)	.132(1)	182(4)
U	.17237 (5)	.11510(4)	.0369(1)

^aHere and in the following tables the number in parenthesis is the estimated standard deviation in the least significant figure. The estimated standard deviations are a result of least squares refinement on a model in which C-C distances were restrained as described in the text. Hydrogen atoms were included but not refined.

Table III. <u>Uranium-Carbon Distances</u> (A)

U	-	C(1)	2.66(3)
		C(2)	2.63(3)
		C(3)	2.65(4)
		C(4)	2.60(3)
:		C(5)	2.66(3)
		C(6)	2.64(3)
		C(7)	2.62(2)
		C(8)	2.68(2)
		C(12)	2.66(2)
		C(13)	2.66(3)
		C(14)	2.68(4)
		C(15)	2.58(3)
		C(16)	2.61(3)
		C(17)	2.67(2)
		C(18)	2.61(2)
		C(19)	2.66(2)







XBL 817-10783





dicyclopentenouranocene, 1.



XBL 7811-12981

Figure 2. Ortep ball and spoke view of the molecule.

Hydrogen atoms are omitted for clarity.



the cyclooctetraene rings. Hydrogen atoms omitted.

SYNTHESIS AND STRUCTURE OF

DICYCLOPENTENOURANOCENE, U[C₈H₆(CH₂)₃]₂

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Lawrence Berkeley Laboratory

and

Department of Chemistry, University of California Berkeley, California 94720

SUPPLEMENTARY MATERIAL

Table IV. Thermal Parameters^{a,b}

				•		
Atom	B11	B22	B33	B12	B13	B23
: 1'	· .	· · · · · · · · · · · · · · · · · · ·	•			
C(1)	7.0(19)	6.4(18)	6.3(18)	2.0(16)	1.3(15)	-1.7(15)
C(2)	10.8(24)	7.4(28)	7.3(22)	4.9(19)	4.7(17)	1.1(19)
C(3)	23.9(57)	3.4(19)	5.4(21)	3.6(25)	4.3(30)	.3(15)
C(4)	26.1(63)	4.7(21)	2.6(18)	8.2(28)	-5.8(27)	-1.2(15)
C(5)	6.9(21)	12.4(32)	11.1(34)	5.8(23)	-7.2(23)	-8.1(24)
C(6)	3.5(15)	16.5(38)	13.0(35)	-2.2(20)	3.8(21)	-10.9(28)
C(7) ^C	19.0(36)	1.1(10)	8.7(22)	5(16)	8.1(27)	-3.1(11)
C(8)	7.4(18)	3.3(13)	3.7(17)	1.9(12)	3(13)	6(10)
C(9)	9.1(20)	7.7(20)	9.8(28)	4.4(18)	2.3(22)	1.3(17)
C(10)	9.0(28)	17.1(42)	18.8(45)	2.4(29)	-7.7(30)	-5.6(36)
C(11)	4.7(21)	12.1(28)	25.7(52)	2.3(20)	7.2(26)	3(32)
C(12)	4.9(14)	6.2(17)	4.4(15)	-1.9(12)	3.4(13)	5(12)
C(13)	14.3(33)	6.4(18)	5.2(19)	6.6(23)	2.0(22)	4(15)
C(14)	21.9(56)	12.3(33)	2.7(17)	12.6(41)	1.9(25)	.2(18)
C(15)	8.4(26)	15.6(43)	8.2(30)	8.7(28)	-6.0(24)	-4.7(24)
C(16)	6.0(20)	6.5(22)	15.3(43)	-2.3(15)	2.5(23)	-5.0(22)
C(17)	12.6(24)	2.5(18)	9.3(25)	-1.4(18)	5.2(28)	-1.2(11)
C(18)	5.9(15)	4.8(13)	2.6(13)	.9(12)	.3(12)	1.2(9)
C(19)	7.2(15)	4.7(12)	1.8(12)	0(11)	.6(11)	7(11)
C(20)	4.6(17)	15.1(29)	7.6(24)	.9(18)	-2.6(15)	1.6(19)
C(21)	7.8(22)	21.7(47)	10.8(31)	-7.6(27)	3.4(22)	1.2(31)
C(22)	13.6(32)	6.9(20)	8.9(24)	-1.2(19)	3.4(23)	-2.7(17)
U	5.63(4)	3.44(3)	3.64(4)	1.47(5)	18(5)	60(4)

^aThe anisotropic temperature factor has the form $\exp(-.25(B_{11}H^2a^{*2} + 2B_{12}hka^{*}b^{*} + . . .))$. ^bHydrogen isotropic thermal parameters of 8.0 Å² for H(1)-H(12) and 12.0 Å² for H(13)-H(24) were assigned but not refined.

 $^{\rm C}$ C(7) thermal tensor calculates non-positive definite.

		•	
H(1)	.0376	. 0969	. 3019
H(2)	.1485	.0756	.3938
н(3)	2554	.1212	.3757
H(4)	.3070	.1877	.2478
н(5)	.2664	.2427	.066
H(6)	.1535	.2631	0079
H(7)	.2394	.1424	3217
H(8)	.1154	.138	3197
Н(9)	.0465	.0692	2161
H(10)	.0576	.0038	0509
H(11)	.1561	0391	.0441
H(12)	.2692	0078	.0949
H(13)	.0222	.2159	1315
H(14)	.0099	.272	0334
H(15)	0841	.1930	051
Н(16)	076	. 2337	.0885
H(17)	069	.1523	.2094
н(18)	0453	.1162	.0687
Н(19)	.4072	.0244	0276
н(20)	.3793	.0602	.1107
H(21)	.4578	.104	0972
н(22)	.4092	.1414	.014
н(23)	.3527	.1736	1759
н(24)	.3833	.1223	2756

Table V. Calculated Positional Parameters for the Hydrogen Atoms in Dicyclopentenouranocene.

C(1)	C(2)	1.41(2)
C(2)	C(3)	1.40(2)
C(3)	C(4)	1.40(2)
C(4)	C(5)	1.39(2)
C(5)	C(6)	1.38(2)
C(6)	C(7)	1.40(2)
C(7)	C(8)	1.38(2)
C(8)	C(1)	1.40(2)
C(12)	C(13)	1.39(2)
C(13)	C(14)	1.40(2)
C(14)	C(15)	1.40(2)
C(15)	C(16)	1.39(2)
C(16)	C(17)	1.39(2)
C(17)	C(18)	1.40(2)
C(18)	C(19)	1.39(2)
C(19)	C(20)	1.40(2)
C(8)	C(9)	1.55(2)
C(9)	C(10)	1.38(5)
C(10)	C(11)	1,46(6)
C(11)	C(1)	1.53(2)
C(19)	C(20)	1.54(2)
C(20)	C(21)	1.43(5)
C(21)	C(22)	1.43(5)
C(22)	C(12)	1.53(2)

. . معرض برجانه اراره

^aThese distances are the result of a restrained structural refinement and are thus prejudiced.

Table VIL Selected Angles (deg.)

	C(1)	- '	U -	C(2)		31.0(5)
	C(2)	_	u -	C(3)		30.7(5)
	C(3)	-	υ -	C(4)		31.0(5)
	C(4)	-	u -	C(5)		30.6(5)
	C(5)	. –	ບ່ -	C(6)		30.2(5)
	C(6)	_ 1	υ· -	C(7)		31.0(5)
. •	C(7)	-	u -	C(8)		30.1(4)
	C(8)	-	u -	C(1)		.30.3(4)
	C(12)) –	υ –	C(13)		30.4(5)
	C(13) –	υ –	C(14)		30.4(5)
	C(14)) –	υ -	C(15)		30.8(5)
	C(15) –	U -	C(16)		31.1(5)
	C(16) –	u -	C(17)		30.5(4)
	C(17)	U -	C(18)		30.7(4)
	C(18) -	u -	C(19)		30.5(4)
	C(19) –	u -	C(12)		30.6(5)
C(8)	_	C(1)		C(2)		134(3)
C(1)		C(2)	_	C(3)		138(4)
C(2)	-	C(2)	_	C(A)		1 20 (4)
C(3)	-	C(4)		C(5)		1/2(2)
C(4)	-	C(5)	·	C(5)		133(3) T43(3)
C(5)	_	C(6)		C(7)		131(3)
C(6)	_	C(7)	-	C(8)		140(3)
C(7)	_	C(8)		C(0)		133(3)
C(19)	· _	C(12)	-	C(13)		133(2)
C(12)	· _	C(13)	-	C(14)		143(3)
C(13)	·	C(14)	_	C(15)		124(4)
C(14)	-	C(15)	-	C(16)		142(3)
C(15)	-	C(16)		C(1.7)		132(3)
C(16)	-	C(17)	-	C(18)		135(2)
C(17)	-	C(18)	-	C(19)		135(2)
C(18)	-	C(19)	-	C(12)		134(2)
C(2)	-	C(1)	·	C(11)		113(3)
C(8)	-	C(1)	-	C(11)		113(3)
C(1)	-	C(11)	-	C(10)		98(3)
C(11)	-	C(10)	-	C(9)		115(4)
C(10)	-	C(19)	• • -	C(8)		104(3)
C(1)	-	C(8)	·	C(9)		105(2)
C(7)	-	C(8)	 .	C(9)		122(3)
C(13)		C(12)	-	C(22)		118(3)
C(19)	-	C(12)	-	C(22)	•	110(2)
C(12)	-	C(22)	-	C(21)		104(3)
C(22)	-	C(21)	-	C(20)		112(3)
C(21)	-	C(20)	-	C(19)	••	105(3)
C(20)		C(19)	-	C(12)		107(2)
C(20)	-	C(19)	-	C(18)		119(3)

$$I = C - (t_{c}/2t_{b})(B_{1}+B_{2})$$

$$\sigma(B) = Max[(t_{c}/2t_{b})(B_{1}+B_{2})^{\frac{1}{2}}, (t_{c}/2t_{b})|B_{1}-B_{2}|]$$

$$\sigma(I) = [0 + \sigma^{2}(B)]^{\frac{1}{2}}$$

$$F^{2} = (D \cdot A/Lp)I$$

$$\sigma(F^{2}) = (D \cdot A/Lp)\sigma(I)$$

$$F^{2}_{a} = \Sigma F^{2}/n$$

$$\sigma(F^{2}_{a}) = [\Sigma \sigma^{2}(F^{2})]^{\frac{1}{2}}/n \qquad \text{When } S(F^{2}_{a}) > 4\sigma(F^{2}_{a}), \sigma(F^{2}_{a}) \text{ is replaced by } S(F^{2}_{a}).$$

$$S(F^{2}_{a}) = [\Sigma|F^{2}-F^{2}_{a}|^{2}/n(n-1)]^{\frac{1}{2}}$$

$$\sigma(F^{2}_{o}) = [\sigma^{2}(F^{2}_{a}) + (pF^{2}_{a})^{2} + q^{2}]^{\frac{1}{2}}$$

$$F_{o} = (F^{2}_{a})^{\frac{1}{2}}$$

$$\sigma(F) = F_{o} - [F^{2}_{a} - \sigma(F^{2}_{o})]^{\frac{1}{2}} \text{ when } \sigma(F^{2}_{o}) \leq F^{2}_{a} \text{ or } [\sigma(F^{2}_{a})]^{\frac{1}{2}} \text{ when } \sigma(F^{2}_{a}) > F^{2}_{a}$$

$$Lp = [\cos^{2}2\theta_{m} + \cos^{2}2\theta]/[\sin 2\theta (1 + \cos^{2}2\theta_{m})]$$

$$wtg = t/\sigma^{2}(F)$$

C = counts recorded during a scan

- I = individual raw intensity, background removed.
- $t_{c} = scan count time$
- t_b = background count time
- B₁ = individual background count
- σ(B) = estimated standard deviation of the total background count
- F = structure factor
- D = decay correction; an empirically applied correction obtained from the fluctuations of the standard reflections.
- A = absorption correction
- Lp = Lorentz and polarization corrections

- $\theta_{m} = monochromater angle$
- θ = crystal diffraction angle
- S = scatter
- a = average
- q = additional uncertainty that affects the weak intensities
- p = estimate of non-statistical
 errors
- wtg = weighting factors in least squares

OBSERVED STRUCTURE FACTORS, STANDARD DEVIATIONS, AND DIFFERENCES (ALL X 2.02 UC(22)H(24) F(0,0,0) = 3801

FOB AND FCA ARE THE OBSERVED AND CALCULATED STRUCTURE FACTORS. SG = ESTIMATED STANDARD DEVIATION OF FOB. DEL = /FOB/ - /FCA/. * INDICATES ZERO WEIGHTED DATA.

K FOI	3 SG	DEL	K	FOB	SG	DEL	K	F06	SS	DEL	K	F08	SG	DEL	K	F08	SG	DEL
HoL	2 0	• 0	0	689	-22	-5	2	235	19	15	22	98	30	-27*	2	241	9	2
2 11	115	=22*	2	127	9	-14	i.	û	95	-60-	23	55	57	147	3	69	14	29.
4135	61	-12	ū	680	17	17	6	197	16	-8		1.12	1.	. 3	- L	73	15	-0.2
5 44	14	15	6	125	12	-1	A	102	27	284	· , '	271	Ē	1	5	104	12	ר ד
64184	76	26	6	470	15	- 2	Ŭ	سانىتى ت.لىت	4.		5	740	22	4 1.	ć	407	20	
40 621		40	4 0	770	4.4	-0		1964	46) <u>1</u> 36	2	112	23	14 7	2	193	60	-0
10 624	13	13	10	620	44	11	4	434	10	20	3	221	ç	3		50	52	0
12 64	20	6	12	263	15	4	2:	1258	40	34	4	230	5	12	8	73	15	204
14 468	15	-1	14	213	11	5	3	485	13	11	5	280	S	4	- 5	7.9	17	•6*
16 329	3 12	-1	16	228	12	- 4	4	366	12	18	6	664	19	3	10	130	11	-8
18 521	27	17	18	181	15	-19	- 5	103	6	4	7	133	7	11	11	52	54	48.
20 128	17	3	20	83	33	4*	-6	634	20	-3	8	174	· 7	- 2	12	122	11	-4
22 314	13	12	22	137	22	2	7	107	7	2	9	431	14	1	13	82	18	25*
24 (73	-8*	۲	1,L=	ם פ	, 5	8	428	13	12	10	463	15	-17	14	130	11	1
H, L:	2	. 1	2	588	22	-5	9	495	16	5	11	0	42	-24 , *	15	47	63	46*
2 32	7 10	12	4	146	10	24	10	655	20	-9	12	245	10	-8	16	71	23	-16-
4 349	5 12	18	6	481	16	-32	11	22	39	-22*	13	217	ç	- 5	17	72	33	-10-
6 1 6	5 7	-6	8	302	11	2	12	6 N N	13	-4	14	250	ē	= 15	1.8	1 0 1	16	7
8 36	9	21	10	389	13	-8	13	230	- A	-12	15	26	4.8	74	19	<u> </u>	71	-94
10 142	A A	6	12	310	12	-4	14	321	14	-13	16	258	10		20	96	1 0	17.*
12 84		_1 < ¥	4 /.	224	14	2	15	54	24	- 4 7 4	47	194	4 0	5	20			
14 70	. 37	-14	16	204	12	c	46	700	17	-17	49	171	20	5	ิก์	196-	4 6	, U
16 440		1. 1.	4.8	405	20	-104	47	408	4.7	- 22	40	210	50	マルマル	4	400	~ ~	-0 -
40 4			46	202	40	-46	4.0	720	40	- 4 4	72	044	26		-	202	14	0 70
20 77		-4.95	20	242	10	-10	10	190	10	-11	24	211	11	404	2	34	23	<u>ئ</u> ت
20 10	33	-10-			ູ ບຸ 		73	94	12	2	21	94	27	127	<u> </u>	217	. 9	-1
22 (00	• f ?	U U	134	13	2	23	515	14	- 4	22	រូវ	55	-39-	4	431	14	-4
<u></u>		A 14	~	~		~ e. 34	~ .			-				A	~	A	~ ~	
24 68	83	6*	2	0	55	-60*	21	128	17	•7	23	41	70	-15*	5	89	25	-15*
24 65 H,L=	83	6* , 2	2	0 87	55 23	-60* -16*	21 22	128	17 63	-7 -48₹	23 H	41 1063	70	-15*	5 6	89 165	25 9	-15*
24 68 H,L= 01110	83 0 34	6* 2 0	246	0 87 50	55 23 58	-60* -16* 24*	21 22 23	128 0 82	17 63 23	-7 -48* -3*	23 H C	41 1,L3 745	70 1, 2E	-15 [#] 4 17	5 6 7	89 165 217	25 9 11	-15* -6 -7
24 65 H,L 01110 2 201	83 0 34 7	6* 2 0 7	2 4 6 8	0 87 50 86	55 23 58 25	-60* -16* 24* -28*	21 22 23 24	128 0 82 189	17 63 23 12	-7 -48* -3* -25	23 H C 1	41 •L≥ 745 277	70 1, 2E 10	-15 [#] 4 17 13	5678	89 165 217 372	25 9 11 13	-15* -6 -7 -14
24 68 H,L= 01110 2 201 4 253	83 0 34 7 8	6* 2 0 7 -6	2 4 6 8 10	0 87 50 86 0	55 23 58 25 62	-60* -16* 24* -28* -13*	21 22 23 24	128 0 82 189 1,L=	17 63 23 12 1,	-7 -48* -3* -25 2	23 H C 1 2	41 1.5 745 277 30	70 1; 2E 10 41	-15* 4 17 13 -9*	56789	89 165 217 372 12	25 9 11 13 68	-15* -6 -7 -14 -33*
24 65 H,L= 01110 2 201 4 253 6 325	83 0 34 7 8 8	6* 2 0 7 -6 8	2 4 6 10 12	0 87 50 86 0	55 28 55 25 55 25 64	-60* -16* 24* -28* -13* -46*	21 22 23 24	128 82 189 1,L= 286	17 63 23 12 10	-7 -48* -3* -25 -25 5	23 H C 1 2 3	41 •L≇ 745 277 30 301	70 2€ 10 41 10	-15* 4 17 13 -9* 7	567 89 10	89 165 217 372 12 267	25 9 11 13 68 10	-15* -6 -7 -14 -33* 2
24 68 H,L= 01110 2 201 4 253 6 328 8 690	83 0 34 7 8 8 10 23	6* 2 7 -6 8 -1	2 4 6 8 10 12 14	0 87 50 86 0 55	5285244 666	-60* -16* 24* -28* -28* -13* -46* 42*	21 22 23 24 0 1	128 82 189 1,L= 286 335	17 63 23 12 10 11	-7 -48* -3* -25 2 5 12	23 C 1 2 3 4	41 1,L 745 277 30 301 632	70 2E 10 41 10 21	-15* 4 17 13 -9* 7 19	567 89 10 11	89 165 217 372 12 267 202	25 9 11 13 68 10	-15* -6 -7 -14 -33* 2 -3
24 63 H,L= 01110 2 201 4 253 6 323 8 690 10 419	83 0 34 7 8 10 23 15	6* 2 7 -6 8 -1 6	2 4 6 10 12 14 16	0 87 50 86 0 55 13	5538525 52525 52525 525 525 525 525 525 525	-60* -16* 24* -28* -13* -46* 42* -46* -46*	21 22 23 24 0 1 2	128 82 189 1,L= 286 335 159	17 63 23 12 10 11 6	-7 -48* -3* -25 25 5 12 2	23 H C 1 2 3 4 5	41 , L= 745 277 301 632 86	70 2E 10 41 10 21 10	-15# 4 17 13 -9# 7 19 5	56789 10112	89 165 217 372 12 267 202 224	25 9 11 13 68 10 10	•15* •6 •7 •14 •33* •3 •13
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3	723	52	-31	9	153	7	-7 '	17	0	80	-26*	10	198	28	-11	8	179	15	-28
4	625	39	-18	10	177	8	7	18	0	81-	1014	11	٥	59	-36*	1	327	11	-1
5	339	22	- 8	11	518	15	-3	19	142	15	-15	12	132	18	4	2	0	60	-45*
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15	460	15	-11	21	119	18	11	7	81	18	-10*	4	50	64	-15*	12	0	47	-358
16	181	73	-68*	22	115	22	-23*	8	123	9	-1	5	61	30	49#	13	40	50	34*
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19	77	35	-20*	- 7	458	15	2	4	432	14	-11	11	546	17	7	19	183	12	17
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1	1° F 🖬	3,	, 5	15	115	81	424	7	43	23	20*	14	38	64	-7*	2	68	27	81*
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14	93	25	614	8	234	9	14	17	0	61	- 9*	12	6	63	-15*	9	Q	44	-19÷
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-4	0	67	-20*	5	360	12	-4	15	0	60	-384	3	0	6 <u>5</u>	-414	5	24	42	-39*
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