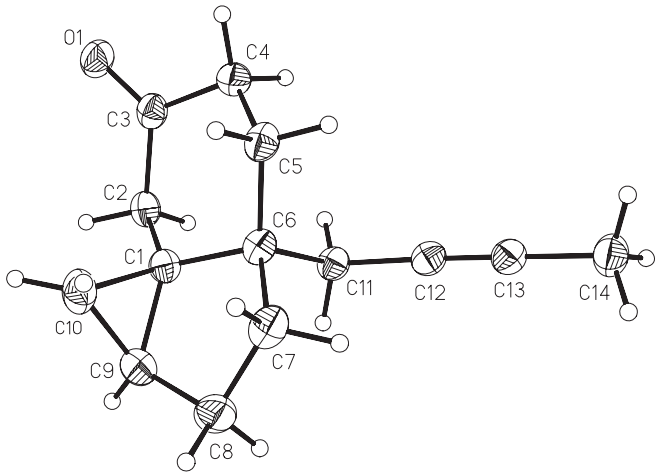
Z. Kristallogr. NCS **221** (2006) 481-482 / **DOI** 10.1524/ncrs.2006.0164

Crystal structure of (1a*SR*,3a*RS*,7a*SR*)-3a-but-2-ynyl-1a,2,3,3a,4,5-hexahydro-1*H*-cyclopropa[*c*]inden-6(7*H*)-one, $C_{14}H_{18}O$

N. T. Tzvetkov[I,II], B. Neumann[III], H.-G. Stammler[III] and J. Mattay*[,I]

[I] Universität Bielefeld, Fakultät für Chemie, Organische Chemie I, Postfach 100131, 33501 Bielefeld, Germany
[II] Universität Bonn, Pharmazeutisches Institut, An der Immenburg 4, 53121 Bonn, Germany
[III] Universität Bielefeld, Fakultät für Chemie, Abteilung für Röntgenstrukturanalyse AC III, Universitätsstr. 25, 33615 Bielefeld, Germany

Received September 8, 2006, accepted and available on-line November 22, 2006; CCDC no. 1267/1887



Abstract

$C_{14}H_{18}O$, monoclinic, $P12_1/c1$ (no. 14), a = 13.7800(4) Å, b = 7.1220(3) Å, c = 11.7670(7) Å, β = 98.640(1)°, V = 1141.7 Å^3, Z = 4, $R_{gt}(F)$ = 0.045, $wR_{ref}(F^2)$ = 0.125, T = 100 K.

Source of material

The title compound has been obtained by cyclopropanization reaction of (7a′*SR*)-7a′-prop-2-ynyl-1′,2′,4′,6′,7′,7a′-hexahydrospiro[1,3-dioxolan-2,5′-indene] with diethyl zinc (1 M solution in *n*-hexane) and freshly distilled diiodomethane in dry diethyl ether in accordance with a separate report [1]. Purification by column chromatography (cyclohexane/ethyl acetate 85:15) and subsequent separation by HPLC (cyclohexane/ethyl acetate 99:1) gave the title product as a colorless oil. Colorless needles, suitable for X-ray structure analysis were obtained after slow evaporation of a minimum of the solvent mixture over night at 273 K and crystallization over a period of several days at 268 K (m.p. 333-334 K).

Discussion

The title compound contains three asymmetric centers and crystallizes in a centrosymmetric space group, that is therefore a racemate of two enantiomeric molecules, which are oriented antiparallelly in the crystal packing. The shortest intramolecular bond length is d(C12—C13) = 1.197(2) Å. The molecules are linked into layers by a combination of π-π interactions of the butynyl substituent (d(C13···C13) = 3.830 Å) and by weak intermolecular C–H···O hydrogen bonds. The shortest intermolecular distances are d(O1···H2B–C2) = 2.501 Å and d(O1···H10B–C10) = 2.530 Å. The cyclopropane unit is connected to the indene basic structure in the asymmetric centers C1 and C9 and it is *trans* to the butynyl substituent in the C6 position.

Table 1. Data collection and handling.

Crystal:	colorless needle, size 0.02 × 0.08 × 0.30 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.72 cm^{-1}
Diffractometer, scan mode:	Nonius KappaCCD, φ/ω
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7865, 2012
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1575
$N(param)_{refined}$:	137
Programs:	SHELXS-97 [2], SHELXL-97 [3], SHELXTL-plus [4]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(2A)	4*e*	0.4785	0.5305	0.0875	0.031
H(2B)	4*e*	0.4342	0.3228	0.0931	0.031
H(4A)	4*e*	0.3716	0.4445	0.3889	0.034
H(4B)	4*e*	0.3614	0.2591	0.3116	0.034
H(5A)	4*e*	0.2069	0.3890	0.2851	0.032
H(5B)	4*e*	0.2483	0.5960	0.2694	0.032
H(7A)	4*e*	0.1351	0.6455	0.0902	0.037
H(7B)	4*e*	0.0933	0.4416	0.0486	0.037
H(8A)	4*e*	0.1642	0.4513	–0.1168	0.044
H(8B)	4*e*	0.1420	0.6717	–0.1038	0.044
H(9)	4*e*	0.3286	0.5909	–0.1087	0.039
H(10A)	4*e*	0.2656	0.8068	0.0838	0.041
H(10B)	4*e*	0.3797	0.8108	0.0522	0.041
H(11A)	4*e*	0.2431	0.2074	0.0073	0.029
H(11B)	4*e*	0.3022	0.1618	0.1319	0.029
H(14A)	4*e*	–0.0129	–0.0251	0.2486	0.051
H(14B)	4*e*	0.0009	–0.1863	0.1576	0.051
H(14C)	4*e*	–0.0622	–0.0030	0.1172	0.051

* Correspondence author (e-mail: oc1jm@uni-bielefeld.de)

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	4*e*	0.52014(9)	0.5224(2)	0.3023(1)	0.0272(7)	0.0333(7)	0.0374(7)	–0.0054(5)	–0.0036(5)	0.0033(5)
C(1)	4*e*	0.3269(1)	0.5289(2)	0.0734(1)	0.0261(9)	0.0214(8)	0.0253(8)	–0.0006(7)	0.0033(7)	0.0005(7)
C(2)	4*e*	0.4282(1)	0.4544(2)	0.1182(1)	0.0239(9)	0.0251(8)	0.0301(9)	–0.0019(7)	0.0071(7)	0.0010(7)
C(3)	4*e*	0.4436(1)	0.4648(2)	0.2483(1)	0.0238(9)	0.0198(8)	0.0314(9)	0.0009(7)	0.0015(7)	0.0017(7)
C(4)	4*e*	0.3601(1)	0.3980(3)	0.3087(1)	0.0293(9)	0.0308(9)	0.0241(8)	–0.0035(7)	0.0048(7)	–0.0013(7)
C(5)	4*e*	0.2572(1)	0.4616(2)	0.2521(1)	0.0253(9)	0.0267(9)	0.0303(9)	–0.0022(7)	0.0081(7)	–0.0069(7)
C(6)	4*e*	0.2424(1)	0.4337(2)	0.1217(1)	0.0214(8)	0.0222(8)	0.0274(8)	0.0010(6)	0.0036(6)	–0.0036(7)
C(7)	4*e*	0.1508(1)	0.5263(2)	0.0535(2)	0.0253(9)	0.0250(9)	0.041(1)	0.0032(7)	–0.0001(7)	–0.0024(7)
C(8)	4*e*	0.1786(1)	0.5628(3)	–0.0668(2)	0.037(1)	0.031(1)	0.038(1)	0.0023(8)	–0.0054(8)	0.0056(8)
C(9)	4*e*	0.2885(1)	0.6028(3)	–0.0450(2)	0.036(1)	0.0295(9)	0.0319(9)	–0.0015(8)	0.0015(7)	0.0062(8)
C(10)	4*e*	0.3183(1)	0.7386(2)	0.0524(2)	0.034(1)	0.0229(9)	0.045(1)	–0.0025(8)	0.0022(8)	0.0040(8)
C(11)	4*e*	0.2420(1)	0.2209(2)	0.0908(1)	0.0243(8)	0.0234(9)	0.0259(8)	–0.0002(7)	0.0043(6)	–0.0027(7)
C(12)	4*e*	0.1559(1)	0.1215(2)	0.1209(1)	0.0273(9)	0.0234(8)	0.0259(8)	0.0006(7)	0.0032(6)	–0.0039(7)
C(13)	4*e*	0.0840(1)	0.0432(2)	0.1426(1)	0.0271(9)	0.0270(9)	0.0271(8)	–0.0006(7)	0.0033(7)	–0.0020(7)
C(14)	4*e*	–0.0049(1)	–0.0506(3)	0.1687(2)	0.029(1)	0.036(1)	0.038(1)	–0.0062(8)	0.0063(7)	–0.0011(8)

Acknowledgments. We are grateful to the Volkswagen-Stiftung, Hannover, to the Deutsche Forschungsgemeinschaft (DFG) and the Fonds der Chemischen Industrie for financial support. N.T.T. thanks Dr. Harald Euler (Mineralogisch-Petrologisches Institut, University of Bonn) for his support during the preparation of this work.

References

1. Tzvetkov, N. T.: Photoreaktionen tricyclischer Cyclopropylketone – Aufbau von Polyquinanen und analoger Ringsysteme. Dissertation, University of Bielefeld, Germany 2005.
2. Sheldrick, G. M.: SHELXS-97. Program for the Solution of Crystal Structures. University of Göttingen, Germany 1997.
3. Sheldrick, G. M.: SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.
4. Sheldrick, G. M.: SHELXTL-plus. Structure Determination Software Suite. Release 4.1. Siemens Analytical Systems, Madison, Wisconsin, USA 1991.