The Crystal and Molecular Structure of 9-(*p*-Iodophenyl)-9-azatetracyclo[5.3.1.$0^{2,6}$.$0^{8,10}$]undec-4-ene

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The crystal and molecular structure of 9-(*p*-iodophenyl)-9-azatetracyclo[5.3.1.$0^{2,6}$.$0^{8,10}$]-undec-4-ene ($C_{16}H_{16}IN$) has been determined by single crystal x-ray diffraction methods. The compound crystallizes in space group $P2_1$, (no. 4) with unit cell constants: a = 11.269 (1), b = 7.607 (1), c = 16.714 (1) Å, β = 104.73 (1)°. The structure was solved by the heavy-atom method and refined by block-diagonal least-squares to a final R-value of 0.054 for the 1546 independently measured, statistically significant reflections. The aziridine ring is fused to the norbornyl rather than to the five-membered ring as previously assumed. The nitrogen to phenyl-carbon distance is 1.40_6 Å and indicates a significant amount of double bond character.

Introduction.

Aziridine-type compounds have continued to be of major importance because of their proven efficiency as alkylating agents in the search for effective anticarcinogens (1). Previous to our systematic structural studies of simple aziridine derivatives, each in a different type of bonding situation, practically no general structural information regarding them was available. Our previous efforts had concentrated on simple bicyclic systems (2).

Recently, it came to our attention that the reaction of cyclopentadiene with an azide, when photolyzed, led to the formation of a polycyclic aziridine derivative (3). Two alternative pathways – both leading to rather unusual aziridine derivatives – were possible. As shown in Figure 1, if the cyclopentadiene (I) dimerizes first to II, then adds the azide to form the triazoline (III), the final product, on photolysis, is the tricyclooctane aziridine compound IV. If instead, the azide adds first, the intermediate V is formed; dimerization follows giving VI, and the photolysis product would then be a norbornyl-type aziridine derivative (VII).

Whichever path is followed in the reaction, the resulting product (the polycyclic aziridine compound) would be an unusual aziridine derivative and characteristic of a category for which no structural information was previously available. Furthermore, whichever structure resulted, it would

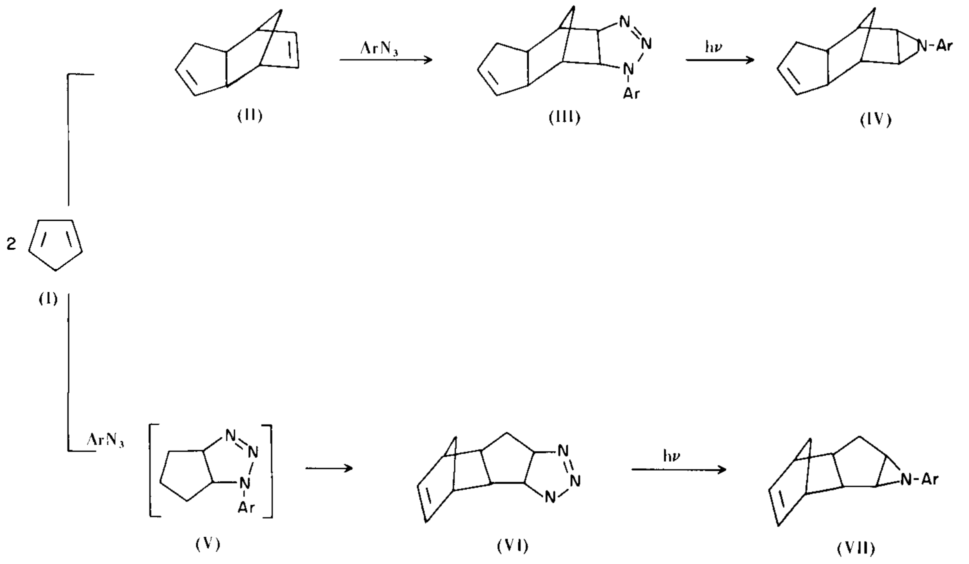

FIGURE 1

shed light on the controversy regarding explanations for the differences in solvolysis rates of norbornyl and tricyclooctane derivatives. A sample of the *p*-iodophenyl derivative of the product (Figure 2) was made available to us and its structure is reported herein. For clarity, the numbering of atoms in this compound will follow the scheme illustrated in Figure 2.

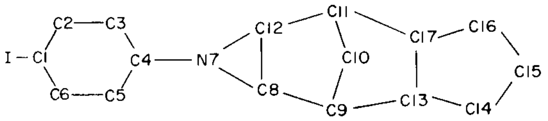

FIGURE 2

Experimental.

A sample of the title compound was kindly furnished to us by Professor Paul Fanta, Illinois Institute of Technology. Beautifully formed cylindrical needles were recrystallized by a solvent exchange technique (4) using chloroform and diethyl ether. A crystal (0.23 mm long and 0.08 mm in diameter) was selected and mounted with the b-axis coincident with *phi* on a G.E. XRD-5 Diffractometer. An investigation of reciprocal space revealed 2/m symmetry with the only extinction being a 2_1 screw axis. Thus at this stage, the possible monoclinic space groups were either $P2_1$ (no. 4) of $P2_1/m$ (no. 11). Least-square lattice constants (listed below with estimated standard deviations) were obtained from 29 carefully measured two-*theta* values using copper radiation under fine conditions (1° take off angle and 0.05° slit).

$a = 11.269 \pm 0.001$ Å
$b = 7.607 \pm 0.001$ Å
$c = 16.714 \pm 0.001$ Å
$\beta = 104.73 \pm 0.01°$

The crystal density measured by flotation techniques is 1.63 g/cc which compares favorably to 1.67 assuming four molecules per unit cell. The crystal was then transferred to a G.E. XRD-490 fully-automated diffractometer and the intensity data were collected by the stationary crystal-stationary counter method using balanced zirconium and yttrium filters with molybdenum Kα-radiation. A total of 2627 independent reflections were measured to a two-*theta* maximum of 50° (d = 0.840Å). Of these, 1546 reflections were considered observed (from statistical tests). The intensities were corrected for absorption as a function of *phi* only (linear absorption coefficient for Mo radiation is 23.2 cm^{-1}). Lorentz-polarization corrections were applied and the corrected intensities were then reduced to structure amplitudes in the usual manner.

Refinement.

A three dimensional Patterson function was calculated to resolve the space-group ambiguity and to locate the heavy atom positions. On the basis of the locations of the large peaks in the Patterson map, the space group $P2_1/m$ was readily eliminated and the remaining choice ($P2_1$) with two crystallographically unique molecules in the asymmetric unit was chosen. The y-coordinate of one of the iodine atoms was arbitrarily chosen to be 0.5, fixing the origin in $P2_1$. The second iodine was placed relative to this origin. Five cycles of isotropic block-diagonal least-squares refinement of the iodine positions yielded an R-value of 0.22. An electron density map phased by the iodines showed the phenyl and aziridine rings clearly, but left some ambiquity as to the remainder of the structure. Additional cycles of least-squares refinement using the iodines and these nine atoms in each molecule led to a value of R = 0.18. A new Fourier map based on these refined positions was calculated and yielded positions for the norbornyl and cyclopentene atoms. All 36 non-hydrogen atoms were refined isotropically by block-diagonal least-squares (using unit weights) to an R-value of 0.11. After conversion to anisotropic temperature factors, five more cycles of similarily weighted anisotropic refinement yielded an R-value = 0.059. The scattering factor table for iodine was corrected for anomalous dispersion with $\Delta f''$ (5) equal to 2.40 electrons. Additional least-squares refinement with both signs for $\Delta f''$ showed no significant difference in either the structure or the R-value. Consequently, all further refinement proceeded without the $\Delta f''$ correction. Examination of the difference electron map phased by the 36 non-hydrogen atoms showed some peaks in those regions where hydrogens were expected. Based on chemical considerations, hydrogen positions were calculated for all carbon atoms except those at the end of the two five-membered rings. Several more cycles of refinement of the 60 atoms (varying only the 36 non-hydrogen atoms) gave a final R-value of 0.052. Table I contains the final values of the coordinates and anisotropic temperature factors for both unique molecules (labelled as "primed" and "unprimed" molecules). Table II lists the calculated hydrogen positions.

The weighting scheme for least-squares was changed to $1/\sigma^2$ and the complete refinement was repeated to a final R-value of 0.054. There were no significant differences in the structures obtained from the two weighting schemes. Consequently, the results reported herein are those for the unit weight refinement with no $\Delta f''$ correction for iodine.

Results.

The estimated standard deviations for each of the parameters are given in Table I. The estimated standard

TABLE I

Final Least-Squares Parameters (ESD refers to last digit; $\beta \times 10^4$)

	X	Y	Z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
I	0.3627(1)	0.5000(0)	0.3463(1)	147(2)	198(3)	34(0)	0(3)	24(1)	10(1)
C1	0.3757(18)	0.4786(33)	0.4721(10)	132(23)	205(47)	29(7)	135(37)	42(11)	73(20)
C2	0.4814(20)	0.5325(33)	0.5280(12)	114(26)	222(60)	44(10)	74(32)	4(13)	32(20)
C3	0.4908(19)	0.5163(30)	0.6145(12)	112(23)	137(46)	43(9)	10(29)	10(11)	16(18)
C4	0.3942(15)	0.4358(22)	0.6414(10)	47(16)	88(38)	35(8)	-43(20)	-5(9)	8(13)
C5	0.2890(19)	0.3837(27)	0.5832(11)	101(23)	161(42)	31(8)	-4(27)	26(11)	25(16)
C6	0.2858(18)	0.4088(27)	0.5018(11)	84(21)	161(43)	24(7)	10(24)	-14(10)	7(14)
N7	0.3975(13)	0.4291(21)	0.7273(8)	81(16)	165(38)	22(6)	14(20)	12(9)	18(11)
C8	0.5044(17)	0.3490(25)	0.7823(12)	66(21)	121(39)	40(9)	-25(24)	18(11)	-5(15)
C9	0.5271(18)	0.3910(32)	0.8742(13)	64(21)	256(55)	39(9)	13(29)	3(11)	-19(19)
C10	0.3981(17)	0.4206(27)	0.8791(12)	69(20)	173(48)	46(9)	64(25)	9(11)	-27(17)
C11	0.3452(18)	0.2417(33)	0.8371(13)	60(21)	269(58)	47(11)	25(31)	26(12)	15(21)
C12	0.3913(17)	0.2516(27)	0.7600(11)	69(20)	151(42)	25(8)	25(24)	11(10)	10(15)
C13	0.5565(20)	0.2143(31)	0.9202(11)	97(24)	223(51)	26(8)	-20(30)	22(11)	-9(17)
C14	0.6476(22)	0.0987(36)	0.8977(12)	123(28)	320(65)	25(8)	19(38)	-9(12)	-33(20)
C15	0.5906(25)	-0.0644(32)	0.8657(13)	230(37)	218(61)	39(9)	178(42)	46(15)	38(20)
C16	0.4636(23)	-0.0587(31)	0.8618(11)	200(32)	179(56)	25(8)	-50(36)	6(12)	11(17)
C17	0.4329(17)	0.1148(33)	0.8960(11)	52(20)	305(59)	29(8)	3(29)	21(10)	5(19)
I′	0.8553(1)	0.4682(3)	0.6046(1)	130(2)	184(3)	32(0)	16(3)	19(1)	-12(1)
C1′	0.8602(15)	0.4526(26)	0.4776(10)	61(17)	104(40)	40(8)	23(24)	15(9)	-38(16)
C2′	0.7778(16)	0.3502(26)	0.4245(11)	33(17)	153(41)	34(8)	-48(22)	13(9)	1(15)
C3′	0.7872(18)	0.3303(27)	0.3453(11)	75(22)	158(43)	27(8)	-2(26)	-3(10)	-16(15)
C4′	0.8858(17)	0.4099(24)	0.3198(11)	77(20)	109(36)	34(8)	15(22)	38(10)	11(14)
C5′	0.9640(17)	0.5087(32)	0.3766(12)	81(20)	163(48)	49(9)	20(30)	-2(11)	-54(20)
C6′	0.9598(17)	0.5374(29)	0.4578(11)	61(19)	219(55)	37(8)	-47(26)	5(10)	-49(18)
N7′	0.8883(13)	0.3992(22)	0.2376(9)	58(16)	181(36)	33(7)	1(20)	12(8)	-3(13)
C8′	1.0061(16)	0.3283(26)	0.2216(11)	51(19)	141(40)	34(8)	27(24)	10(10)	9(15)
C9′	1.0286(19)	0.3779(28)	0.1406(12)	84(22)	173(45)	41(9)	-8(26)	35(12)	16(17)
C10′	0.8991(19)	0.3900(31)	0.0826(13)	89(24)	224(53)	49(10)	62(30)	32(13)	6(19)
C11′	0.8599(17)	0.2101(33)	0.1081(11)	40(18)	302(57)	26(8)	-17(29)	3(9)	-7(19)
C12′	0.8924(15)	0.2195(27)	0.2018(10)	35(17)	192(44)	21(7)	-13(23)	7(9)	5(15)
C13′	1.0729(17)	0.2068(32)	0.1112(11)	56(20)	282(57)	29(8)	-46(29)	19(10)	-11(18)
C14′	1.1673(18)	0.0923(30)	0.1694(14)	44(20)	202(50)	67(12)	-1(26)	22(13)	35(21)
C15′	1.1165(21)	-0.0740(25)	0.1768(12)	164(29)	85(39)	48(10)	48(26)	58(14)	-9(15)
C16′	0.9886(23)	-0.0794(23)	0.1299(13)	165(31)	231(61)	44(10)	-46(29)	-14(10)	-26(20)
C17′	0.9542(18)	0.860 (29)	0.0846(10)	95(22)	248(51)	9(6)	-13(28)	13(9)	-16(14)

Anisotropic Temperature Factor of the Form

$$\exp\,[-\beta_{11}\,h^2 + \beta_{22}\,k^2 + \beta_{33}l^2 + \beta_{12}\,2hk + \beta_{13}\,2hl + \beta_{23}\,2kl]$$

deviations in bond lengths range in value from 0.01_5 – 0.03_8 Å. Similarly, the range of estimated standard deviations in bond angles is 1.2-2.1°. The individual distances are summarized in Table III and the individual angles in Table IV for each molecule. Since the two molecules are chemically equivalent (although crystallographically unique) and since each molecule is bisected by a symmetry-independent mirror plane, the study affords at least two (and in most cases, four) independent measurements for each distance and angle. One can thus construct a composite molecule utilizing the average value for each distance and angle. Figure 3 shows such a composite with the average deviation of each of the parameters indicated. The average deviations and the estimated standard devia-

TABLE II

Calculated Hydrogen Positions
(Atom Numbers refer to bonding carbon atom)

Unprimed Molecule

	X	Y	Z
H2	0.557	0.589	0.506
H3	0.569	0.569	0.659
H5	0.211	0.328	0.601
H6	0.205	0.369	0.457
H8	0.598	0.339	0.779
H9	0.587	0.507	0.886
H10′	0.389	0.433	0.940
H10″	0.355	0.532	0.845
H11	0.248	0.231	0.819
H12	0.374	0.152	0.714
H13	0.577	0.259	0.983
H17	0.387	0.109	0.945

Primed Molecule

	X	Y	Z
H2	0.707	0.285	0.445
H3	0.721	0.255	0.303
H5	1.037	0.574	0.356
H6	1.027	0.614	0.501
H8	0.598	0.339	0.779
H9	1.077	0.501	0.147
H10′	0.898	0.405	0.021
H10″	0.848	0.493	0.098
H11	0.764	0.191	0.087
H12	0.872	0.118	0.240
H13	1.101	0.260	0.061
H17	0.916	0.077	0.019

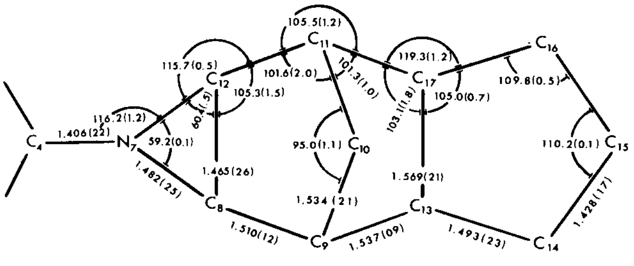

Figure 3 - "Composite" molecule. Average values of distances and angles (with average deviations) given.

TABLE III

Table of Interatomic Distances (ESD Refers to Last Digits)

	Unprimed Molecule	Primed Molecule
I - C1	2.078 (16)	2.142 (17)
C1 - C2	1.376 (29)	1.355 (26)
C1 - C6	1.345 (28)	1.406 (26)
C2 - C3	1.428 (29)	1.365 (26)
C6 - C5	1.366 (26)	1.387 (27)
C3 - C4	1.418 (27)	1.423 (27)
C5 - C4	1.387 (26)	1.349 (27)
C4 - N7	1.429 (22)	1.384 (23)
N7 - C8	1.451 (24)	1.518 (24)
N7 - C12	1.465 (25)	1.498 (26)
C8 - C12	1.439 (28)	1.491 (26)
C8 - C9	1.526 (29)	1.489 (28)
C12 - C11	1.508 (28)	1.517 (25)
C9 - C10	1.493 (29)	1.536 (30)
C10 - C11	1.578 (32)	1.532 (33)
C9 - C13	1.542 (32)	1.520 (31)
C11 - C17	1.544 (31)	1.546 (30)
C13 - C17	1.546 (30)	1.591 (29)
C13 - C14	1.473 (34)	1.520 (30)
C17 - C16	1.513 (33)	1.469 (31)
C14 - C15	1.436 (35)	1.407 (30)
C16 - C15	1.417 (38)	1.457 (33)

tion are of comparable magnitude for all bonds and angles. Consequently the remaining discussion will focus on the "averaged" molecule.

As previously stated, the molecule, as determined in the crystal study, contains a mirror plane which bisects it on a line running through the iodine, C1, C4, N7, C10 and C15 atoms. Chemically, this should not be the case since the five-membered ring contains one double bond (either C14-C15 or C15-C16). If one looks at the values of these distances ($1.42_8 \pm 0.01_7$ Å), the temperature factors of the atoms involved (especially C15), and refers to the ORTEP drawing (6), figure 4, an explanation for this anomaly is readily apparent. It seems clear that there is orientational disorder at the back end of the molecule accompanied by pronounced thermal anisotropy and that the molecule can pack as easily in the unit cell with the double bond located between C14-C15 as it can with the double-bond located at C15-C16. That, in fact, a double bond does exist in this five-member ring is essentially verified by the planarity of this ring (ESD = 0.011Å) and by the dihedral angle (126°) between this ring and the tricyclooctane ring.

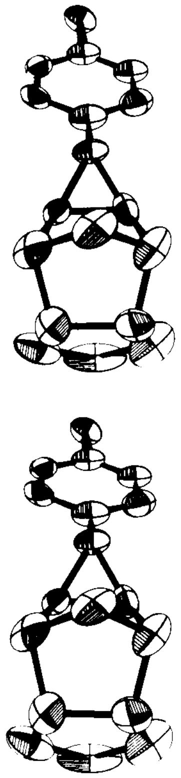

Figure 4 - ORTEP drawing of "unprimed" molecule.

Table V summarizes the least-squares fit and gives the equations of the various planes of interest in this molecule.

A comparison of known molecular features with literature values confirms the estimate of errors. Thus the I-Cl distance of 2.07_8 (16)Å lies in the middle of the range of such values (7) in the literature, and the ICC angles of 119.3 (1.8)° are in conformity with the expected sp^2 hybridization. The phenyl ring distances (1.34_4 (21)Å) and the corresponding angles (119.9 (3.0)°) agree with accepted literature values (1.39_7Å and 120.0°, respectively) (8) although the deviations are often quite large in the individual values, approaching two standard deviations in a number of cases. Such a variation is not unusual when the heavy atom is as large as iodine. Probably a more realistic indication of the level of refinement of the phenyl rings is provided by the least-squares fit of the six atoms, in each case, to a plane. Table V shows that the phenyl planes have ESD values of 0.008 and 0.006Å.

The C4-N distance of 1.40_6 (22)Å is appreciably shortened from the normal C-N single-bond distance and thus indicates appreciable delocalization and double-bond character in this distance. The phenyl-nitrogen angles of 122.4 (2.9)° and the C4-aziridine angles of 116.2 (1.2)° support this assumption.

TABLE IV

Table of Interatomic Angles (ESD)

	Unprimed Molecule	Primed Molecule
I - C1 - C2	119.3 (1.5)	119.6 (1.3)
I - C1 - C6	122.6 (1.3)	115.6 (1.3)
C2 - C1 - C6	118.1 (1.7)	124.4 (1.7)
C1 - C2 - C3	119.3 (2.0)	119.3 (1.7)
C1 - C6 - C5	126.2 (1.8)	112.7 (1.7)
C2 - C3 - C4	119.5 (1.9)	120.3 (1.7)
C6 - C5 - C4	117.4 (1.9)	126.7 (1.9)
C3 - C4 - N7	120.6 (1.6)	119.2 (1.6)
C5 - C4 - N7	119.6 (1.6)	123.9 (1.7)
C3 - C4 - C5	119.3 (1.7)	116.5 (1.7)
C4 - N7 - C8	117.2 (1.4)	115.8 (1.5)
C4 - N7 - C12	114.6 (1.4)	117.4 (1.5)
C8 - N7 - C12	59.1 (1.2)	59.2 (1.2)
N7 - C8 - C12	60.9 (1.3)	59.7 (1.2)
N7 - C12 - C8	60.0 (1.2)	61.1 (1.2)
N7 - C8 - C9	116.7 (1.6)	115.4 (1.6)
N7 - C12 - C11	115.1 (1.7)	115.6 (1.7)
C8 - C12 - C11	106.2 (1.6)	102.9 (1.5)
C12 - C8 - C9	106.2 (1.6)	106.0 (1.6)
C8 - C9 - C10	99.8 (1.6)	103.5 (1.7)
C12 - C11 - C10	99.4 (1.7)	103.5 (1.7)
C8 - C9 - C13	106.2 (1.7)	102.9 (1.6)
C12 - C11 - C17	105.9 (1.6)	106.9 (1.5)
C10 - C9 - C13	101.2 (1.7)	100.7 (1.6)
C10 - C11 - C17	98.5 (1.6)	102.1 (1.6)
C9 - C10 - C11	96.2 (1.6)	93.8 (1.6)
C9 - C13 - C17	103.4 (1.7)	105.3 (1.6)
C11 - C17 - C13	104.4 (1.8)	99.2 (1.6)
C9 - C13 - C14	118.0 (1.8)	121.3 (1.7)
C11 - C17 - C16	118.4 (1.7)	120.1 (1.7)
C13 - C17 - C16	104.4 (1.7)	104.9 (1.7)
C17 - C13 - C14	106.3 (1.9)	104.4 (1.7)
C13 - C14 - C15	109.6 (2.1)	109.8 (1.8)
C17 - C16 - C15	109.6 (1.9)	110.4 (1.9)
C14 - C15 - C16	110.0 (2.1)	110.3 (1.8)

The C-N distances in the aziridine ring (1.48_2 Å) and the C-C distance (1.46_5Å) agree within one standard deviation with the same parameters determined in a microwave study of the free aziridine molecule (9). Similarly, the angles within the aziridine moiety are essentially all 60° as anticipated.

The tricyclooctane nucleus is in very close agreement with a previous study (10) in which such a nucleus is fused to a cyclopropane ring and interestingly enough also agrees

TABLE V

Least Squares Planes and Dihedral Angles

- Planes -

Label	Plane	Atoms Involved	Equation of Plane	E.S.D. (Å)
	Phenyl			
a	(unprimed)	C1, C2, C3, C4, C5, C6	$-.4209p + .9067q + .0271r = 2.553$	0.008
a′	(primed)	C1′, C2′, C3′, C4′, C5′, C6′	$-.5020p + .8140q - .2922r = 3.311$	0.006
	Aziridine			
b	(unprimed)	N, C8, C12	$-.4746p + .3933q + .7875r = 9.886$	-----
b′	(primed)	N′, C8′, C12′	$+.0034p - .3833q + .9236r = 2.399$	-----
	Tricyclo octane system			
c	(unprimed)	C8, C9, C11, C12	$-.4554p + .8607q - .2276r = 1.677$	.009
c′	(primed)	C8′, C9′, C11′, C12′	$-.5766p + .8147q - .0613r = -4.170$	.018
d	(unprimed)	C9, C11, C13, C17	$-.4784p + .3772q + .7930r = 11.274$	.009
d′	(primed)	C9′, C11′, C13′, C17′	$-.0210p - .3715q + .9282r = .822$	.016
e	Bridgehead (primed)	C9, C10, C11	$-.0223p - .4431q + .8962r = 11.325$	-----
e′	Bridgehead (unprimed)	C9′, C10′, C11′	$-.5397p + .4213q + .7288r = -3.086$	-----
	Cyclopentene Ring			
f	(unprimed)	C13, C14, C15, C16, C17	$-.0356p - .3981q + .9166r = 12.910$	.011
f′	(primed)	C13′, C14′, C15′, C16′, C17′	$-.5246p + .3503q + .7759r = -4.174$	.012

- Dihedral Angles -

Planes Involved (Letters Refer to Above)	Angle
Phenyl - Aziridine	
a b	125.3°
a′ b′	126.0°
Aziridine - Tricyclic System	
b c	112.0°
b′ c′	111.8°
Bridgehead - Remainder of Tricyclic System	
e c	127.9°
e′ c′	125.7°
e d	124.0°
e′ d′	122.1°

quite closely with a second study involving a norbornene nucleus (11). The distances within the tricyclooctane moiety average to $1.53_9 \pm 0.01_8$ Å. If one excludes the bridgehead carbon (C10), the remaining angles average to 104.5 (1.8)°. This value lies between the previously reported values of 104.0 (1.5)° found for tricyclooctane (10) and the 104.8 (2.0)° found for norbornene (11).

More surprisingly, the bridgehead carbon (C10) also mimics both previously mentioned structures in its molecular parameters. Its angles of fusion with the base ring (101.4° ± 1.50°) are experimentally identical to the similar angles in the tricyclooctane (101°) and to those in the norbornene (100°). The angle at the bridgehead position (95° ± 1.1°) is actually closer to that in the norbornene (96°) although the differences are not statistically significant. These results would thus present grave doubts about the correctness of the explanations advanced for the differences in the rates of solvolysis between substituted tricyclooctanes and norbornene compounds. Such explanations for this rate difference (1:10) (11) have generally assumed that in the norbornenes, the geometry of the bridgehead position is altered, and the bridgehead angle opened up to facilitate formation of the transition state (12). In actuality, the plane containing the bridgehead carbon (defined by C_9, C_{10}, C_{11}) forms essentially equivalent dihedral angles (125.1°, 123.7°) with the planes defined by the four atoms on either side and thus is in no way uiquely situated.

The parameters in the five-membered ring, with the exception of the disordering about atom C15, are those to be anticipated. Thus the distance of ($1.49_3 \pm 0.02_3$Å) and the angles at the fusion points of 105.0 ± 0.7° are comparable to those found in cyclopentene oxide (13). The planarity of this five-membered ring (ESD = 0.011Å) further enhances this similarity.

Knowing now that the compound is the tricyclooctane derivative and not the norbornyl derivative, the correct mechanism of formation is obvious. Namely, the dimerization proceeds first, followed by the addition of the azide to form the triazoline which, upon photolysis, gives this product. The product, incidently, is that which had been predicted as the most likely one (14).

The packing within the unit cell is consistent with that estimated on the basis of Van der Waal contact distances. There are only three I-C distances less than 4.0Å (I-C8. . . . 3.95Å,, I-C15. . . . 3.75Å, I′-C6. . . . 3.90Å); no C-C contacts less than 3.5Å, and only two H-H contacts less than 2.5Å (H13-H11′. . . . 2.45Å, H11′-H10′. . . . 2.35Å).

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