Chemistry of Heterocyclic Compounds **2018**, *54*(3), 339–343

(Aza)indolizines and ethyl propiolate: [8+2] and [1,10] cyclizations

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SUPPLEMENTARY INFORMATION

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Table 1. Crystallographic characteristics, experimental data and refinement of structures **2, 6, 9.**

Compound	**2**	**6**	**9**
CCDC	1828872	1828871	1828873
Molecular formula	$C_{17}H_{19}NO_2$	$C_{11}H_{13}N_2I$	$C_{22}H_{23}NO_4$
Syngony, space group, *Z*	Моноклинная, $P2_1/m$, 2	Моноклинная, $P2_1/c$, 4	Моноклинная, $P2_1/m$, 2
a, b, c, Å	9.7564(7), 7.2290(4), 10.7519(8)	8.0389(3), 12.9386(4), 11.5216(3)	10.3431(10), 7.1094(6), 13.1029(14)
α, β, λ, deg	90.0, 100.800(6), 90.0	90.0, 99.394(2), 90.0	90.0, 96.732(8), 90.0
V, Å^3	744.89(9)	1182.31(7)	956.86(16)
D_{calc}, g/cm^3	1.201	1.686	1.261
Radiation; λ, Å	Cu*K*α, 1.5418		
μ, cm^{-1}	0.623	20.990	0.707
T, K	295(2)		
Sample size, mm	0.1 × 0.1 × 0.1	0.2 × 0.2 × 0.2	0.2 × 0.2 × 0.2
Diffractometer	Stoe STADI VARI Pilatus100K		
Scanning type	Rotation		
Accounting for absorption; T_{min}, T_{max}	*DIFABS*; 0.8826, 0.9693	*DIFABS*; 0.9213, 0.9372	*DIFABS*; 0.8759, 0.9317
θ_{max}, deg	71.659	72.919	70.785
Limits *h, k, l*	$-11 \le h \le 11$, $-8 \le k \le 5$, $-13 \le l \le 13$	$-9 \le h \le 9$, $-16 \le k \le 10$, $-14 \le l \le 14$	$-12 \le h \le 12$, $-8 \le k \le 6$, $0 \le l \le 16$
Number of reflections: measured/ independent (N_1), R_{int}/ c $I>2\sigma(I)$ (N_2)	3692 / 1551 0.0612 / 1051	12944 / 2332, 0.0541 / 2079	11093 / 1929 0.0283 / 1299
Method of refinement	Full matrix method of least squares by F^2		
Number of parameters	131	130	182
Accounting for extinction	Нет		
R_1/wR_2 by N_1	0.0703 / 0.1028	0.0464 / 0.1016	0.0565 / 0.0569
R_1/wR_2 by N_2	0.0472 / 0.0949	0.0440 / 0.0997	0.0343 / 0.0521
S	0.905	0.999	0.859
$\Delta\rho_{min}$ / $\Delta\rho_{max}$	-0.175 / 0.165	-0.935 / 0.843	-0.145 / 0.118
Programs	*X-AREA* (STOE & CIE, 2015); *SHELXS97* (Sheldrick, 2008); *SHELXL-2013* (Sheldrick, 2013); *Ortep-3* (Farrugia, 2012); *WinGX* (Farrugia, 2012)		

X-AREA: Stoe & Cie (2015). *X-AREA.* Stoe & Cie, Darmstadt, Germany.

SHELXS-97: Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112-122.

WinGX: Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849-854.

Ortep-3: Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849-854.

SHELXL-2013: Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3-8.

Table 2.1. Specific interatomic distances *d* (A) in the structure **2.**

Bond	*d*	Bond	*d*
N1 C9	1.361(2)	N1 C5	1.361(2)
N1 C2	1.371(2)	C2 C11	1.394(3)
C2 C3	1.439(3)	C3 C4	1.386(3)
C3 C31	1.512(2)	C4 C5	1.432(3)
C5 C6	1.381(3)	C6 C7	1.383(3)
C7 C8	1.399(3)	C8 C9	1.391(3)
C9 C10	1.427(2)	C10 C11	1.408(3)
C10 C12	1.446(3)	C12 O12	1.205(2)
C12 O13	1.345(2)	O13 C14	1.457(2)
C14 C15	1.502(3)	C31 C33a	1.5221(13)
C31 C33	1.5221(13)	C31 C32	1.5221(13)

Symmetric transformation (a): *x*, 1/2-*y*, *z*

Table 2.2. Specific bond angles ω (deg.) in the structure **2.**

Angle	ω	Angle	ω
C9 N1 C5	131.39(15)	C9 N1 C2	114.55(15)
C5 N1 C2	114.06(15)	N1 C2 C11	104.27(15)
N1 C2 C3	104.77(14)	C11 C2 C3	150.96(15)
C4 C3 C2	107.58(15)	C4 C3 C31	127.38(16)
C2 C3 C31	125.05(15)	C3 C4 C5	109.23(16)
N1 C5 C6	114.10(18)	N1 C5 C4	104.36(14)
C6 C5 C4	141.54(17)	C5 C6 C7	118.26(19)
C6 C7 C8	124.68(17)	C9 C8 C7	117.76(18)
N1 C9 C8	113.79(16)	N1 C9 C10	104.36(14)
C8 C9 C10	141.84(17)	C11 C10 C9	107.28(15)
C11 C10 C12	124.58(15)	C9 C10 C12	128.13(16)
C2 C11 C10	109.54(14)	O12 C12 O13	123.19(17)
O12 C12 C10	125.12(17)	O13 C12 C10	111.68(15)
C12 O13 C14	116.96(14)	O13 C14 C15	105.75(14)
C3 C31 C33a	108.59(9)	C3 C31 C33	108.59(9)
C33 C31 C33a	111.32(18)	C3 C31 C32	110.21(12)
C33a C31 C32	109.07(10)	C33 C31 C32	109.07(10)

Symmetric transformation (a): *x*, 1/2-*y*, *z*

Table 3.1. Specific interatomic distances *d* (A) in the structure **6.**

Bond	*d*	Bond	*d*
I9 C9	2.085(4)	N1 C2	1.375(4)
N1 C9	1.392(5)	N1 C5	1.390(5)
C2 C3	1.372(5)	C3 N4	1.371(5)
C3 C31	1.518(5)	N4 C5	1.336(5)
C5 C6	1.395(5)	C6 C7	1.340(6)
C7 C8	1.419(6)	C8 C9	1.343(5)
C31 C34	1.527(6)	C31 C33	1.530(6)
C31 C32	1.533(6)		

Table 3.2. Specific bond angles ω (deg.) in the structure **6.**

Angle	ω	Angle	ω
C2 N1 C9	131.7(3)	C2 N1 C5	106.9(3)
C9 N1 C5	121.5(3)	C3 C2 N1	106.5(3)
C2 C3 N4	110.1(3)	C2 C3 C31	129.4(3)
N4 C3 C31	120.5(3)	C5 N4 C3	106.5(3)
N4 C5 N1	110.0(3)	N4 C5 C6	130.9(4)
N1 C5 C6	119.1(3)	C7 C6 C5	119.9(4)
C6 C7 C8	120.0(3)	C9 C8 C7	121.7(4)
C8 C9 N1	117.8(3)	C8 C9 I9	123.0(3)
N1 C9 I9	119.2(3)	C3 C31 C34	108.5(3)
C3 C31 C33	109.2(4)	C34 C31 C33	110.9(4)
C3 C31 C32	108.9(3)	C34 C31 C32	109.0(4)
C33 C31 C32	110.5(4)		

Table 3.3. Specific dihedral angles φ (deg.) in the structure **6.**

Angle	φ	Angle	φ
C9 N1 C2 C3	-178.9(3)	C5 N1 C2 C3	0.1(3)
N1 C2 C3 N4	-0.6(4)	N1 C2 C3 C31	178.5(3)
C2 C3 N4 C5	0.8(4)	C31 C3 N4 C5	-178.3(3)
C3 N4 C5 N1	-0.8(3)	C3 N4 C5 C6	178.8(3)
C2 N1 C5 N4	0.4(3)	C9 N1 C5 N4	179.6(3)
C2 N1 C5 C6	-179.2(3)	C9 N1 C5 C6	0.0(5)
N4 C5 C6 C7	-179.1(3)	N1 C5 C6 C7	0.3(5)
C5 C6 C7 C8	-0.2(6)	C6 C7 C8 C9	-0.2(6)
C7 C8 C9 N1	0.5(5)	C7 C8 C9 I9	179.5(3)
C2 N1 C9 C8	178.5(3)	C5 N1 C9 C8	-0.4(5)
C2 N1 C9 I9	-0.5(5)	C5 N1 C9 I9	-179.4(2)
C2 C3 C31 C34	-123.4(4)	N4 C3 C31 C34	55.6(4)
C2 C3 C31 C33	-2.5(5)	N4 C3 C31 C33	176.4(4)
C2 C3 C31 C32	118.2(4)	N4 C3 C31 C32	-62.9(4)

Table 4.1. Specific interatomic distances *d* (A) in the structure **9.**

Bond	*d*	Bond	*d*
O14 C14	1.1893(17)	O15 C14	1.3341(17)
O15 C16	1.4452(10)	O18 C18	1.2055(16)
O19 C18	1.3199(16)	O19 C20	1.4459(11)
N1 C5	1.3657(16)	N1 C2	1.3671(16)
N1 C9	1.3675(16)	C2 C11	1.4152(18)
C2 C3	1.4597(18)	C3 C4	1.3703(19)
C3 C31	1.5110(18)	C4 C5	1.4353(19)
C5 C6	1.3701(18)	C6 C7	1.405(2)
C7 C8	1.383(2)	C8 C9	1.3816(18)
C9 C10	1.4158(18)	C10 C12	1.4175(19)
C10 C11	1.4337(17)	C11 C18	1.4663(18)
C12 C13	1.1908(18)	C13 C14	1.435(2)
C16 C17	1.4777(7)	C20 C21	1.5014(9)
C31 C32a	1.5227(12)	C31 C32	1.5227(12)
C31 C33	1.5524(14)		

Symmetric transformation (a): *x*, 3/2-*y*, *z*

Table 4.2. Specific bond angles ω (deg.) in the structure **9.**

Angle	ω	Angle	ω
C14 O15 C16	117.00(10)	C18 O19 C20	117.70(9)
C5 N1 C2	114.99(11)	C5 N1 C9	129.59(11)
C2 N1 C9	115.42(11)	N1 C2 C11	104.22(11)
N1 C2 C3	103.83(11)	C11 C2 C3	151.95(12)
C4 C3 C2	107.57(12)	C4 C3 C31	124.90(13)
C2 C3 C31	127.53(12)	C3 C4 C5	110.10(12)
N1 C5 C6	114.70(13)	N1 C5 C4	103.51(11)
C6 C5 C4	141.79(14)	C5 C6 C7	119.18(14)
C8 C7 C6	122.72(14)	C9 C8 C7	119.20(14)
N1 C9 C8	114.61(13)	N1 C9 C10	104.22(11)
C8 C9 C10	141.16(14)	C9 C10 C12	122.45(12)
C9 C10 C11	108.03(12)	C12 C10 C11	129.51(13)
C2 C11 C10	108.10(11)	C2 C11 C18	127.84(12)
C10 C11 C18	124.06(12)	C13 C12 C10	175.35(15)
C12 C13 C14	176.52(16)	O14 C14 O15	123.34(14)
O14 C14 C13	125.49(15)	O15 C14 C13	111.18(13)
O15 C16 C17	106.70(5)	O18 C18 O19	122.67(13)
O18 C18 C11	125.47(13)	O19 C18 C11	111.87(11)
O19 C20 C21	105.92(5)	C3 C31 C32a	109.60(7)
C3 C31 C32	109.60(7)	C32 C31 C32a	110.36(9)
C3 C31 C33	109.83(10)	C32a C31 C33	108.71(7)
C32 C31 C33	108.71(7)		

Symmetric transformation (a): *x*, 3/2-*y*, *z*