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5-Methyl-2-phenyl-2*H*-1,2,3-diazaarsole in Reaction with Ethyl Diazoacetate

Boris A. Arbuzov, Elena Ya. Zabotina,* and Evelina N. Dianova

A. M. Butlerov Chemical Research Institute at Kazan University, 18, Lenin Str., Kazan 420008, USSR

Igor A. Litvinov,* Victor A. Naumov, Shamil K. Latypov, and Ahat V. Il'asov

A. E. Arbuzov Institute of Organic and Physical Chemistry, 8, Arbuzov Str., Kazan 420083, USSR

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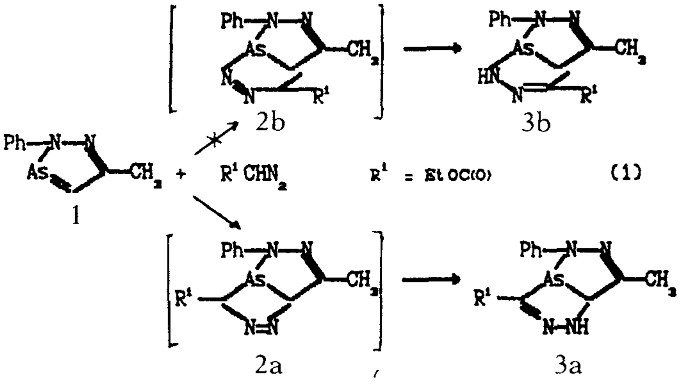
ABSTRACT

5-Methyl-2-phenyl-2H-1,2,3-diazaarsole **1** *reacts with ethyl diazoacetate to form a 1:1 bicyclic product* **3a**, *8-ethoxycarbonyl-4-methyl-2-phenyl-1-arsa-2,3,6,7-tetraazabicyclo[3.3.0]octa-3.7-diene. The latter isomerizes to a two-coordinate arsenic compound* **5**, *3-ethoxycarbonyl-5-[α-(phenylhydrazono)ethyl] 1-1,2,4-diazaarsole. The X-ray crystal structure analyses of both products* **3a** *and* **5** *have been carried out.*

We have compared earlier 2*H*-1.2.3-diazaphospholes [1–5] and 2*H*-1.2.3-diazaarsoles in reactions with some disubstituted diazo-compounds (R_2CN_2, $MePhCN_2$, Me_2CN_2). Monosubstituted diazo-compounds $RCHN_2$ were studied in reactions with diazaphospholes [6–8] but not with 2*H*-1.2.3.-diazaarsoles. However, the 1*H*-1.2.4-diazaarsole reactivity with ethyl diazoacetate was described in Ref. [9]. We wish to report now on the reaction of 2*H*-1.2.3-diazaarsole with ethyl diazoacetate.

5-Methyl-2-phenyl-2*H*-1.2.3-diazaarsole **1** and ethyl diazoacetate yield (Equation 1) two crystalline, yellow 1:1 products with mp 127–128 °C and 233–234 °C, respectively. The mass spectrum of the latter shows a molecular peak with 100%; intensity. The mass spectrum of the first product shows the molecular peak at low inensity (1%) because of redissociation of the substance to the diazaarsole **1** (100%) and a EtOCOĊ=NṄH fragment (14%).



(1)

The IR spectrum of the product of mp 127–128°C exhibits the existence of an NH group (ν 3230 cm^{-1}) indicating a proton shift in the primary adduct **2** (**a** or **b**) to give **3** (**a** or **b**). Its ^{13}C NMR data (Table 1) confirm the structure **3**,, but are not very effective for making a choice between **3a** and its regioisomer **3b**. The ^{1}H NMR spectrum gives evidence for structure **3a**. Double homoresonance at the frequency of the NH signal leads to an increase of the CH signal intensity; this fact shows their proximity. No rapid exchange of protons from the NH group and water present in the solvent (deuteroacetone) was observed. An X-ray crystal structure analysis proved the product to be **3a**, 8-ethoxycarbonyl-4-methyl-2-

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*To whom correspondence should be addressed.

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TABLE 1 ^{1}H and ^{13}C NMR Data of Compounds 3a and 5 in $(CD_3)_2CO$ versus TMS[a]

	C-5	*C-8*	*C-4*	*CH_3*	*CO*	*CH_2*	*$CH_3(R+)$*	*C_i*	*C_o*	*C_m*	*C_p*	*NH*
3a												
δ^1H	5.73 s	—	—	2.25 s	—	4.25 m	1.31 m	6.78 m	7.20 m		7.34 d	9.91 br
δ^{13}C	83.23	142.43	146.43	16.31	165.60	61.27	14.70	149.22	116.37	129.64	120.39	—
5	C-5	C-3	C-6									
δ^1H	—	—	—	2.40 s	—	4.35 m	1.36	6.83 m	7.23 m		7.32 d	8.96 br
δ^{13}C	176.5	[b]	[b]	[b]	61.85	146.27	129.80	146.27	114.27	129.80	120.87	—

[a] For the numbering of the C-position see Figures 1 and 2. [b] The signals are not observed due to small concentration.

phenyl-1-arsa-2,3,6,7-tetraazabicyclo[3,3,0]octa-3,7-diene (Figure 1). X-ray data are discussed below.

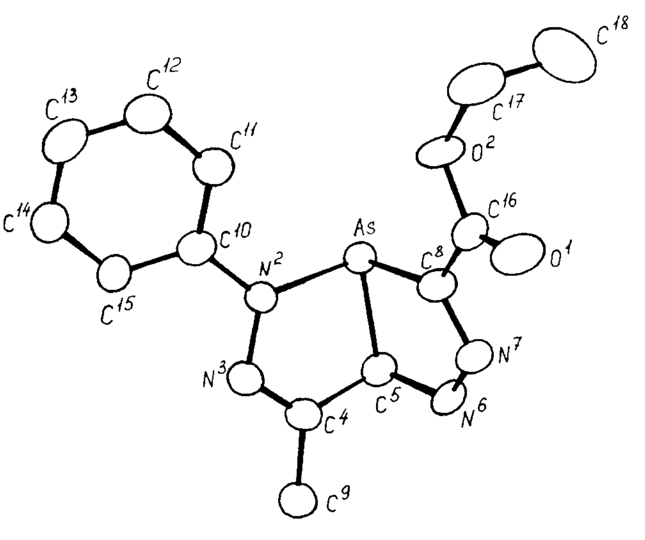
Compound **3a** is stable in $CHCl_3$ or CH_2Cl_2 solution, but a catalytic amount of HCl converts it immediately to the second isomer, mp 233–234 °C. Spectral data of the latter are different from that of **3a**. No NH signal is observed in the ^{1}H-NMR spectrum, which is due to a fast exchange with protons of water (from moist deuteroacetone).

By analogy [6–8] with reactions of 2*H*-1.2.3-diazaphospholes with monosubstituted diazocompound of a two-coordinate arsenic atom: 3-ethoxycarbonyl-5-[α-(phenylhydrazono)ethyl]-1.2.4-**3a** has the structure **4** (Equation 2) resulting from a 1,3 proton shift.

However, an X-ray crystal structure investigation gave an unexpected result and indicated a compound of a two-coordinate arsenic atom: 3-ethoxycarbonyl-5-α[phenylhydrazonolethyl-1.2.4-diazaarsole **5**. It contains a 1-arsa-4-aza-1,3-butadiene fragment of S-trans configuration (Figure 2).

Probably **3a** is converted into **5** via a hydrochloride (Equation 2) with a subsequent cleavage of an As—N bond and elimination of a molecule of HCl.

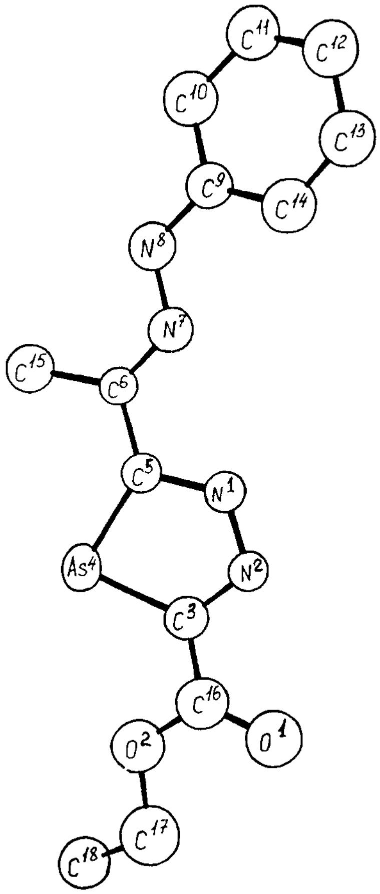
FIGURE 1

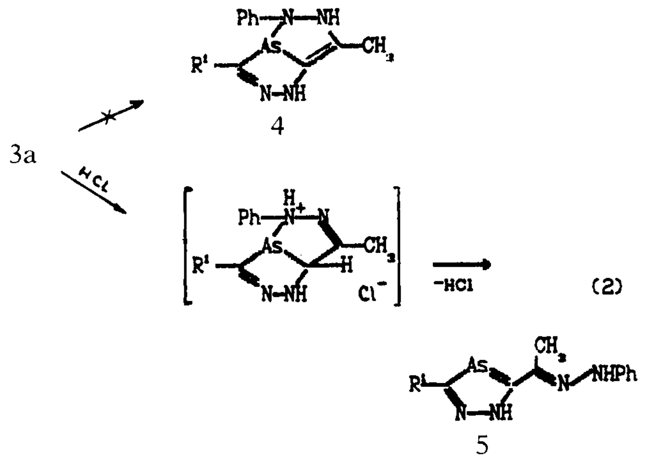


The results of the X-ray structure analysis of both products **3a** and **5** are discussed herein.

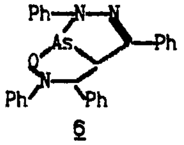
A molecule of **3a** consists of two condensed five-membered heterocycles $AsN^2N^3C^4C^5$ (*1*) and $As\text{-}C^5N^6N^7C^8$ (*2*). They are planar within limits of 0.035(8) Å and 0.100(6) Å, respectively. The dihedral angle between the planes (*1*) and (*2*) is 107.4°. The phenyl ring is almost coplanar with the plane (*1*): the dihedral angle equals 11.0°.

FIGURE 2





The mutual position of arsole rings (*1*) and (*2*) and their conformations in **3a** are not fully the same as those of the other arsenic bicyclo compound **6**, 2,4,6,7-tetraphenyl-1-arsa-2,3,7-triaza-8-oxabicyclo[3.3.0]oct-3-ene described earlier in [11]: the dihedral angle between heterocycles in the latter compound is 80°.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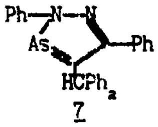
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The conformation of the oxazaarsole ring in **6** is an envelope, the C^5AsON fragment being planar. The geometrical parameters of the same 1.2.3-diazaarsole rings in **3a** and **6** are analogous. Bond distances As—N and As—C are the same within the limits of experimental error. The bond angles N^2AsC^5 are equal 84.4° in both structures and coincide with that found in a two-coordinated arsenic compound **7**: 2.5-diphenyl-4-diphenylmethyl-1.2.3-diazaarsole [12].

X-ray structure analysis of the second product **5** exhibits a planar conformation within limits of 0.02(1) Å. Oxycarbonyl and hydrazone fragments are also planar. The dihedral angle between the planes of the heterocycle and the oxycarbonyl substitutent is 6.2°, and that of the heterocycle and the hydrazone fragment is 13.3°. The length of the $As^4{=}C^5$ bond 1.82(1) Å is the same as that of the As=C bond in **7** and in 4.4′-*bis*-(5-methyl-2-phenyl-1.2.3-diazaarsole) [4] (1.82–1.84 Å). The length of the "single" As^4—C^3 bond 1.89(1) Å is shorter than the same in three-coordinaed arsenic compounds (1.95–1.97 Å) and demonstrates delocalization of electronic density into the five-membered ring.

The geometry of substituents in the molecules of **3a** and **5** is normal.

Molecules of **3a** in a crystal are connected in infinite zigzag chains along an axis y by hydrogen bonds N^6—$H \cdots O^{1'}$ ($2 - x$; $\frac{1}{2} + y$; $2 - z$) with parameters: $N^6 \cdots O^{1'}$ 2.895(6) Å, $H \cdots O^1$ 2.22 Å, angle N^6—$H \cdots O^{1'}$ 161°.

Molecules of **5** in a crystal form a complicated three dimensional framework owing to hydrogen bonds N^1—$H \cdots O^{1'}$ ($-x$; $\frac{1}{2} + y$; $\frac{1}{2} - z$) with parameters $N^1 \cdots O^{1'}$ 2.87(2) Å, $H \cdots O^{1'}$ 2.1 Å with angle N^1—$H \cdots O^{1'}$ 128°; and N^8—$H \cdots O^{1''}$ (x; $\frac{1}{2} - y$; $z - \frac{1}{2}$) with parameters $N^8 \cdots O^{1''}$ 3.30(2) Å, $H \cdots O^{1''}$ 2.2 Å with angle N^8 — $H \cdots O^{1''}$ 147°.

EXPERIMENTAL

IR spectra were recorded on a Specord 75 IR spectrometer. NMR spectra were recorded on a Bruker WM 250 spectrometer equipped with an Aspect computer using standard Bruker software. Proton and carbon-13 measurements were made in 5-mm and 10-mm tubes, respectively, at 298 K. Proton spectra recorded at 250.132 MHz were obtained using a spectral width of 2.8 kHz, a 16 K data block, and a 10° pulse. Broadband proton-decoupled ^{13}C-NMR spectra were obtained at 62.9 MHz using a spectral width of 17 kHz, a 16 K data block, a 24° pulse, a 3 ms delay time, and 5000 number of scans. Coupling constants J_{CH} were measured using gated decoupling. Assignment of the signals was carried out on the basis of the analysis of experiments using various methods of homo- and hetero-double resonance and the literature data [10] for analogous structure fragments.

Mass spectra (electronic impact, EI) were recorded on a Hitachi M 80 B instrument, E_a = 12 eV.

X-ray Structure Determination for 3a and 5

A single crystal of **3a** (mp 127–128 °C) was obtained by slow evaporation from a mixture of solvents: petroleum ether/acetonitrile; **5** (mp 233–234 °C) was obtained by recrystallization from acetonitrile/hexane. A summary of the structure determinations is given in Table 2. Atomic coordinates are listed in Tables 3 and 4. Geometrical parameters are given in Tables 5 and 6.

*Reaction of 5-Methyl-2-Phenyl-2*H*-1.2.3-Diazaarsole 1 with Ethyl Diazoacetate*

1g (8.8 mmole) of ethyl diazoacetate was dropped slowly at room temperature into 1.9 g (8.6 mmole) of the diazaarsole **1**. The reaction mixture was heated up to 28 °C. Crystals **3a** were filtered off during 2 d and washed with hot petroleum ether. 2 g (69%) of **3a** was obtained. IR spectrum (ν, cm^{-1}): 433, 490, 570, 597, 616, 660, 680, 730, 750, 790, 820, 837, 855, 1015, 1043, 1065, 1080, 1160, 1208, 1250, 1285, 1310, 1320, 1360, 1430, 1485, 1585, 1655, and 3230.

Mass spectrum (12 eV): m/z (relative intensity, %): 334 (M^+, 1), 306 (1), 249 (M^+-EtOCOC, 1), 233

TABLE 2 Structure Determination Summary for **3a** and **5** at Room Temperature

	3a	**5**
Empirical formula	$C_{13}H_{13}N_4O_2As$	$C_{13}H_{15}N_4O_2As$
Color, habit	Yellow needlelike crystals	
Crystal size (mm)	0.05 × 0.1 × 0.3	0.2 × 0.1 × 0.05
Crystal system	Monoclinic	Monoclinic
Space group	$P2_1$	$P2_1/c$
Unit cell dimensions[a]	a = 6.222(3) b = 7.469(3) c = 15.502(5) Å β = 99.02(3)°	a = 11.638(8) b = 9.487(9) c = 14.730(6) Å β = 115.47(4)°
Volume	V = 711.5(3) $Å^3$	V = 1468(1) $Å^3$
Z	2	4
Formula weight	334.2	334.2
Density (calcd)	1.56 g/cm^3	1.51 g/cm^3
Absorption coefficient	23.9 cm^{-1}	23.1 cm^{-1}
F(000)	340	680
Diffractometer used	Enraf-Nonius CAD-4	
Radiation	MoK_α (λ = 0.71073 Å)	
2θ range	$4° \leq 2\theta \leq 60°$	$4° \leq 2\theta \leq 44°$
Scan mode	$\omega/(5/3\ \theta)$	
Scan speed	Variable: 2–20/min in θ	
Scan weight	$\omega = 1.35 + 0.35\text{-tg}\theta$	
Standard reflections	Two orientations and two intensities control reflections every 200 reflections	
Index ranges	$-8 \leq h \leq 8$ $0 \leq k \leq 9$ $0 \leq l \leq 20$	$-11 \leq h \leq 8$ $0 \leq k \leq 8$ $0 \leq l \leq 12$
Reflections collected	2304	2304
Observed reflections	1350 ($I \leqq 3\sigma(I)$)	461 ($I \leqq 3\sigma(I)$)
Absorption correction	Empirical correction 12 reflections with X ≦ 80° were measured by azimuthal scan mode	N/A crystals decomposed)
Decay correction	N/A	Linear decay correction
System	SDP-Plus, PDP-11/23	
Solution	Patterson function analysis	
Refinement method	Fuull-matrix least-squares	
Quantity minimized	$\Sigma\ w(F_o - F_C)^2$	
Absolute configuration	Hamilton test ratio [13]: R = 0.03498, wr = 0.0494 for direct structure; R = 0.03725, wR = 0.04751 for inverted; 95% probability for direct structure	N/A
Extinction correction	N/A	N/A
Hydrogen atoms from difference Fourier Maps	Refined isotroically	Not refined, B_{iso} = 4$Å^2$
Weighting scheme	$w = 4\ \lvert F_o\rvert^2/((\sigma(I))^2 + (0.07\ \lvert F_o\rvert^2)^2)^{1/2}$ (non-Poisson contribution)	
Final R-indexes	R = 0.035, wR = 0.045 (1297 reflections, $F^2 \geqq 6\sigma$)	R = 0.047, wR = 0.059 (461 reflections, $F^2 \geqq 3\sigma$)
Goodness of fit	1.1	1.38
Largest Δ/σ	0.28	0.02
Data to parameter ratio	7.2:1	5.12:1
Largest difference peak	0.4 e/$Å^3$	0.5 e/$Å^3$

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[a] Numbers in parentheses are standard deviations.

TABLE 3 Coordinates of Non-Hydrogen Atoms in **3a** and their Equivalent Isotropic Temperature Factors $B_{iso}^{equiv} = 4/3 \sum_{i=1}^{3} \sum_{j=1}^{3} (\mathbf{a}_i\, \mathbf{a}_j)$ $B(i,j)$ (Å^2)

Atom	*x*	*y*	*z*	B_{iso}^{equiv}
As	0.89765(7)	1	0.77544(3)	2.333(7)
O^1	0.7646(8)	0.4881(8)	0.8663(3)	4.04(9)
O^2	0.6462(7)	0.6329(7)	0.7415(3)	3.49(9)
N^2	1.1657(8)	0.9585(8)	0.7363(3)	3.1(1)
N^3	1.3509(6)	1.015(1)	0.7917(3)	2.64(8)
N^8	1.0444(8)	0.9602(7)	0.9515(3)	2.9(1)
N^7	0.9535(8)	0.8055(8)	0.9295(3)	2.71(9)
C^4	1.3120(9)	1.0743(9)	0.8647(4)	2.6(1)
C^3	1.0810(9)	1.0851(8)	0.8832(4)	2.5(1)
C^8	0.8757(9)	0.7884(9)	0.8464(4)	2.7(1)
C^9	1.493(1)	1.136(1)	0.9330(4)	3.6(1)
C^{10}	1.185(1)	0.9122(8)	0.6508(4)	2.5(1)
C^{11}	1.012(1)	0.824(1)	0.5987(4)	3.2(1)
C^{12}	1.028(1)	0.776(1)	0.5139(5)	4.0(2)
C^{13}	1.214(1)	0.813(1)	0.4797(4)	4.5(2)
C^{14}	1.386(1)	0.899(1)	0.5304(4)	4.1(2)
C^{15}	1.3708(9)	0.9498(9)	0.6145(4)	3.2(1)
C^{16}	0.7592(9)	0.6194(8)	0.8222(3)	2.5(1)
C^{17}	0.503(1)	0.481(1)	0.7116(4)	4.4(1)
C^{18}	0.296(1)	0.481(1)	0.7486(6)	5.9(2)

TABLE 4 Coordinates of Non-Hydrogen Atoms of Molecule **5** and their Isotropic Temperature Factors B_{iso} (Å^2)

Atom	*x*	*y*	*z*	B_{iso}
As^4	0.2261(2)	0.0153(2)	0.1275(1)	4.30(4)
O^1	0.297(1)	−0.171(1)	0.3169(9)	5.8(3)
O^2	0.126(1)	−0.129(1)	0.350(1)	6.5(4)
N^1	0.023(1)	0.150(1)	0.1203(9)	3.3(3)
N^2	0.051(1)	0.069(1)	0.1989(1)	3.3(3)
N^7	−0.037(1)	0.307(1)	−0.0480(9)	3.9(3)
N^8	−0.068(1)	0.397(2)	−0.124(1)	4.2(3)
C^3	0.146(1)	−0.008(2)	0.214(1)	3.9(4)
C^5	0.102(1)	0.147(2)	0.072(1)	3.3(4)
C^6	0.071(1)	0.243(2)	−0.012(1)	3.0(4)
C^9	−0.182(1)	0.464(2)	−0.162(1)	4.0(4)
C^{10}	−0.201(2)	0.574(2)	−0.232(1)	5.6(5)
C^{11}	−0.320(2)	0.643(2)	−0.269(1)	5.1(5)
C^{12}	−0.414(2)	0.614(2)	−0.242(1)	5.6(5)
C^{13}	−0.390(1)	0.507(2)	−0.173(1)	5.6(4)
C^{14}	−0.281(2)	0.430(2)	−0.134(1)	5.5(5)
C^{15}	0.164(1)	0.244(2)	−0.057(1)	4.7(4)
C^{16}	0.189(2)	−0.103(2)	0.298(1)	5.6(5)
C^{17}	0.342(2)	−0.277(2)	0.400(2)	7.5(6)
C^{18}	0.476(2)	−0.308(3)	0.428(2)	6.0(8)
$C^{18.2}$	0.456(4)	−0.219(6)	0.467(4)	4(1)

(M^+-$EtOCOCNH_2$, 1), 220 (M^+-$EtO_2C_2N_2H$≡diazaarsole **1**, 100), 219 (diazaarsole 1-1, 7), 166 (3), 152 (11), 145 (40), 114 ($EtOCO{-}\dot{C}{=}N{-}\dot{N}H$, 14), 105 (4), 91 (4), 77 (28), 69 (13), 29 (C_2H_5, 1). Anal. calcd for $C_{13}H_{15}N_4O_2As$: N 16.75. Found: N 16.60.

A mixture of **5** and **3a** (0.2 g) gradually crystallized from the reaction mass and was isolated during 25 d and washed with petroleum ether/acetonitrile. The total yield of **3a** and **5** was 76.3%. IR spectrum of **5** (ν, cm^{-1}): 430, 470, 500, 540, 600, 610, 690, 745, 760, 780, 820, 830, 866, 880, 1015, 1045,

TABLE 5 Bond Angles ω (°) in **3a** and **5**

3a		**5**	
Angle	ω	Angle	ω
N^2AsC^5	84.4(2)	$C^3As^4C^5$	81.7(7)
N^2AsC^8	101.1(3)	$C^{18}O^2C^{17}$	117(2)
C^5AsC^8	82.3(3)	$N^1N^2C^3$	111(1)
$C^{18}O^2C^{17}$	116.0(6)	$N^2N^1C^5$	119(1)
AsN^2N^3	116.2(4)	$N^8N^7C^8$	121(1)
AsN^2C^{10}	123.9(4)	$N^7N^8C^9$	121(1)
$N^3N^2C^{10}$	118.5(5)	$As^4C^5N^1$	111(1)
$N^2N^3C^4$	113.6(4)	$As^4C^5C^6$	132(1)
$N^7N^8C^9$	119.1(5)	$N^1C^5C^6$	117(1)
$N^6N^7C^8$	114.9(5)	$As^4C^3N^2$	117(1)
$N^3C^4C^5$	120.4(5)	$As^4C^3C^{18}$	124(1)
$N^3C^4C^9$	120.6(5)	$N^2C^3C^{16}$	118(1)
$C^5C^4C^9$	119.0(5)	$N^7C^8C^5$	119(2)
AsC^5N^6	105.9(4)	$N^7C^8C^{15}$	125(2)
AsC^5C^4	105.3(4)	$C^5C^6C^{15}$	115(1)
$N^6C^5C^4$	111.4(5)	$O^1C^{18}O^2$	121(2)
AsC^8N^7	115.4(5)	$O^1C^{16}C^3$	123(2)
AsC^8C^{16}	128.9(4)	$O^2C^{16}C^3$	116(2)
$N^7C^8C^{16}$	115.2(5)		
$O^1C^{16}O^2$	124.1(6)		
$O^1C^{16}C^8$	126.1(6)		
$O^2C^{16}C^8$	109.8(6)		

TABLE 6 Bond Distances d (Å) in **3a** and **5**

3a		**5**	
Bond	d	Bond	d
$As—N^2$	1.888(5)	$As^4—C^5$	1.82(1)
$As—C^3$	1.974(6)	$As^4—C^3$	1.89(1)
$As—C^8$	1.942(6)	$O^1—C^{16}$	1.30(2)
$O^1—C^{16}$	1.192(9)	$O^2—C^{18}$	1.33(2)
$O^2—C^{16}$	1.339(7)	$O^2—C^{17}$	1.49(2)
$O^2—C^{17}$	1.47(1)	$N^1—N^2$	1.31(2)
$N^2—N^3$	1.389(7)	$N^2—C^3$	1.26(2)
$N^2—C^{10}$	1.394(7)	$N^1—C^5$	1.38(2)
$N^3—C^4$	1.274(8)	$N^7—N^6$	1.33(2)
$N^6—N^7$	1.308(7)	$N^7—C^6$	1.29(2)
$N^6—C^5$	1.456(8)	$N^8—C^9$	1.36(2)
$N^7—C^8$	1.309(7)	$C^8—C^6$	1.45(2)
$C^4—C^5$	1.510(8)	$C^3—C^{18}$	1.44(2)
$C^4—C^9$	1.493(8)	$C^6—C^{15}$	1.50(2)
$C^8—C^{18}$	1.475(9)		

1095, 1160, 1255, 1288, 1340, 1395, 1442, 1490, 1555, 1595, 1680, 3260, and 3360.

Mass spectrum, m/z (relative intensity, %): 334 (M^+, 100), 287 (52), 260 (M^+-EtOCOH, 43), 234 (6), 220 (diazaarsole **1**, 2), 196 (4), 182 (3), 166 (55), 155 (6), 140 (4), 128 (9), 115 (9), 114 (4), 102 (19), 92 (53), 77 (77), and 65 (81). Anal. calcd. for $C_{13}H_{15}N_4O_2As$: N 16.75. Found: N 17.00.

SUPPLEMENTARY MATERIAL AVAILABLE

Expanded tables of bond distances, bond angles, refined displacement parameter expressions (betas), positional parameters, and 10^{*} F_{obs} plus F_{calc} are available and can be obtained from the authors designated by an asterisk.

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