

Cyclocondensation of [1-Cyano-2-(methylsulfanyl)vinyl]triphenylphosphonium Iodide with Amidine Nucleophiles

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Abstract—The substituted vinylphosphonium salt (*E*)-MeSCH=C(CN)P⁺Ph₃I[−] readily cyclizes under the action of benzamidine and 3-amino-*s*-triazole, but it does not enter cyclocondensation with 2-aminopyridine. The structure of the cyclization product with 3-amino-*s*-triazole was confirmed by its transformation to 7-imino-6-(triphenylphosphoranylidene)-6,7-dihydro-*s*-triazolo[1,5]pyrimidine which was identified by X-ray diffraction. This stabilized ylide and its analogs proved useful starting materials for regioselective syntheses of 2-*R*-4-alkyl-4,7-dihydro-*s*-triazolo[1,5-*a*]pyrimidin-7-ones.

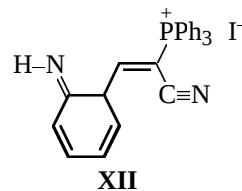
Previously we developed a convenient method for preparing (*E*)-[1-cyano-2-(methylsulfanyl)vinyl]triphenylphosphonium iodide (**I**), a promising multicenter electrophilic reagent for various cyclizations [1]. The most thoroughly studied is its cyclocondensation with hydrazine and its derivatives [2]. In the present work we have studied reactions of compound **I** with a series of amidine nucleophilic substrates. This process leads or may lead to compounds **II–X** (see scheme and Tables 1 and 2).

Treatment of reagent **I** with benzamidine evidently leads to formation not only of (4-amino-2-phenylpyrimidin-5-yl)triphenylphosphonium iodide (**II**), but also of some other compounds, which complicates isolation of the major condensation product pure. Nevertheless, after alkaline dephosphorylation we could isolate about 60% of 4-amino-2-phenylpyrimidine that was prepared previously by another procedure [3], and thus obtained unambiguous evidence for the structure of intermediate phosphonium salt **II**.

We expected that reagent **I** would similarly react with 2-aminopyrimidine. However, we could only obtain compound **III** arising from initial nucleophilic substitution of the methylsulfanyl group. This product underwent no cyclization in the presence of both acids and bases. The structure of compound **III** was confirmed by means of ¹H NMR spectroscopy. In the ¹H NMR spectrum in DMSO-*d*₆, the enamine N–H proton appears as a broad downfield singlet at 12.16 ppm, which may be explained by strong conjugation of the lone electron pair of the exocyclic nitrogen atom with the electron-deficient acyclic π system.

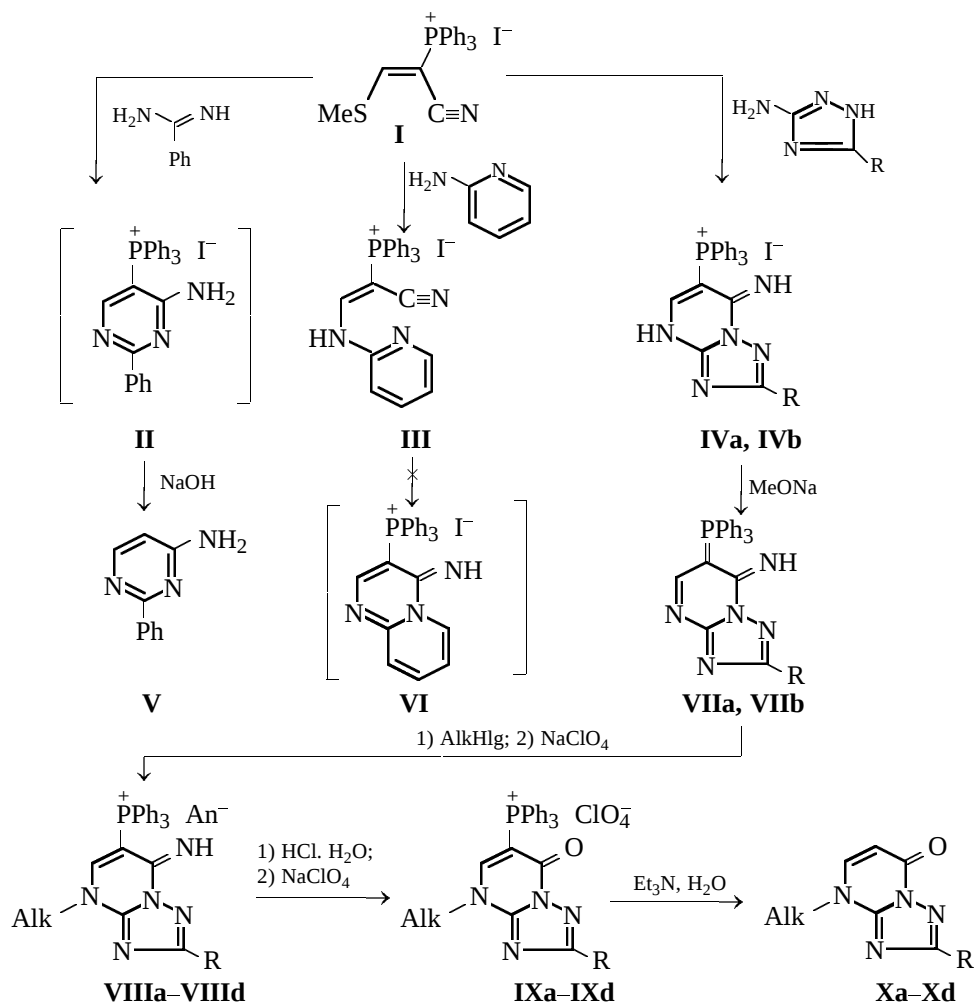
As would be expected, replacement of the 2-pyridyl fragment in **III** by *p*-tolyl does not change significantly the chemical shift of the N–H proton signal [δ_{NH} 11.74 ppm for (1-cyano-2-*p*-toluidino-vinyl)triphenylphosphonium iodide **XI**].

These data allow us to exclude formation of isomer **XII** in which the imino group cannot come in conjugation with the exocyclic π system and, what is more, steric factors unfavour intramolecular hydrogen bonding.



The signal of the imino proton of phosphonium salt **XII** can not be observed near 12 ppm, since the respective signal of related compounds **IV** and **VIII** locates near 8 ppm (Table 1).

Unlike 2-aminopyridine which does not enter cyclocondensation with reagent **I**, 3-amino-*s*-triazole and 3-amino-5-phenyl-*s*-triazole, that contains an amidine residue, extremely easily convert into compounds **IV** and **VII** (the latter are formed by HI elimination from **IV**). The structure of one of the stabilized ylides **VIIa** was reliably established by X-ray diffraction analysis. The general view of molecule **VIIa** is given in the figure, principal bond lengths and angles, in Table 3, and atomic coordinates, in Table 4.



$\text{R} = \text{H}$ (**a**, **c**, **d**), C_6H_5 (**b**); $\text{An} = \text{ClO}_4$ (**VIIIa**, **VIIIc**, **VIIIId**), **I** (**VIIIb**); $\text{Alk} = \text{CH}_3$ (**a**, **b**), C_2H_5 (**c**), $\text{CH}_2=\text{CH}-\text{CH}_2$ (**d**); $\text{Hlg} = \text{Br}$, **I**.

Table 1. ^1H NMR spectral data of the synthesized compounds

Comp. no.	δ , ppm ($\text{DMSO}-d_6$)
III	7.20 d.d (1H, CH arom., $^3J_{\text{HH}}$ 5.1, $^3J_{\text{HH}}$ 5.4 Hz), 7.41 d (1H, CH arom., $^3J_{\text{HH}}$ 8.1 Hz), 7.86–7.99 m (16H, CH arom.), 8.18 d (1H, CH arom., $^3J_{\text{HH}}$ 3.6 Hz), 8.38 d (1H, CH, $^3J_{\text{HP}}$ 12 Hz), 12.16 s (1H, NH)
VIIa	6.85 s (1H, NH), 7.20 d (1H, C^5H , $^3J_{\text{HH}}$ 8.1 Hz), 7.70–7.81 m (15H, $3\text{C}_6\text{H}_5$), 8.10 s (1H, C^2H)
VIIIa	3.69 (3H, CH_3), 7.47 d (1H, C^5H , $^3J_{\text{HP}}$ 12.3 Hz), 7.71–7.87 m (15H, $3\text{C}_6\text{H}_5$), 8.38 s (1H, NH), 8.41 s (1H, C^2H)
IXd	4.94 d (2H, CH_2 , $^3J_{\text{HH}}$ 1.5 Hz), 5.27–5.41 m (2H, CH_2), 5.95 m (1H, $\text{CH}=\text{CH}_2$), 7.79–7.91 m (16H, $3\text{C}_6\text{H}_5$, C^5H), 8.53 s (1H, C^2H)
Xa	3.79 s (3H, CH_3), 6.03 d (1H, C^6H , $^3J_{\text{HH}}$ 7.8 Hz), 8.07 d (1H, C^5H , $^3J_{\text{HH}}$ 8.1 Hz), 8.28 s (1H, C^2H)
Xb	3.84 s (3H, CH_3), 6.04 d (1H, C^6H , $^3J_{\text{HH}}$ 7.8 Hz), 7.53 m (3H, CH arom.), 8.07 d (1H, C^5H , $^3J_{\text{HH}}$ 7.8 Hz), 8.15 m (2H, 2CH arom.)
Xd	4.83 d (2H, CH_2 , $^3J_{\text{HH}}$ 5.7 Hz), 5.29 m (2H, 2CH), 6.00–6.11 m (2H, C^6H , CH), 8.09 d (1H, C^5H , $^3J_{\text{HH}}$ 7.8 Hz), 8.27 s (1H, C^2H)
XI	2.62 s (3H, CH_3), 7.12 d.d (4H, CH arom., $^3J_{\text{HH}}$ 8.4, $^3J_{\text{HH}}$ 8.4 Hz), 7.57 d (1H, CH, $^3J_{\text{HP}}$ 11.1 Hz), 7.83–7.97 m (15H, $3\text{C}_6\text{H}_5$), 11.74 s (1H, NH)

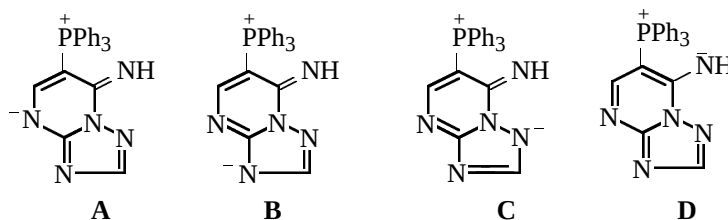
Table 2. Constants, yields, and elemental analyses of the synthesized compounds

Comp. no.	Yield, %	mp, °C (solvent for crystallization)	Found, %			Formula	Calculated, %		
			(Cl) (I)	N	P		(Cl) (I)	N	P
III	73	224–22 (acetone)	(23.75)	7.86	5.84	C ₂₆ H ₂₁ IN ₃ P	(23.79)	7.88	5.81
IVa	80	278–279 (ethanol–acetonitrile, 1 : 1)	(24.28)	13.41	5.94	C ₂₃ H ₁₉ IN ₅ P	(24.25)	13.38	5.92
IVb	55	182–184 (ethanol)	(21.20)	11.53	5.05	C ₂₉ H ₂₃ IN ₅ P	(21.17)	11.68	5.17
VIIa	87	249–251 (acetonitrile)	–	17.77	7.91	C ₂₃ H ₁₈ N ₅ P	–	17.71	7.83
VIIb	78	289–291 (ethanol–acetonitrile, 1 : 1)	–	14.92	6.60	C ₂₉ H ₂₂ N ₅ P ^a	–	14.85	6.57
VIIIa	65	171–173 (ethanol)	6.96	13.80	6.12	C ₂₄ H ₂₁ ClN ₅ O ₄ P	6.95	13.74	6.07
VIIIb	90	294–296 (ethanol–acetonitrile, 2 : 1)	(20.71)	11.48	5.11	C ₃₀ H ₂₅ IN ₅ P	(20.69)	11.42	5.05
VIIIc	58	136–138 (ethanol)	6.73	13.39	5.94	C ₂₅ H ₂₃ ClN ₅ O ₄ P	6.77	13.37	5.91
VIIId	69	195–197 (ethanol)	6.68	13.00	5.81	C ₂₆ H ₂₃ ClN ₅ O ₄ P	6.62	13.07	5.78
IXa	60	190–192 (ethanol–acetonitrile, 2 : 1)	6.98	10.85	5.98	C ₂₄ H ₂₀ ClN ₄ O ₅ P	6.94	10.97	6.06
IXb	62	303–305 (ethanol)	6.12	9.49	5.32	C ₃₀ H ₂₄ ClN ₄ O ₅ P	6.04	9.55	5.28
IXc	36	159–161 (ethanol–acetonitrile, 2 : 1)	6.79	10.70	5.88	C ₂₅ H ₂₂ ClN ₄ O ₅ P	6.75	10.67	5.90
IXd	59	218–219 (ethanol)	6.59	10.40	5.79	C ₂₆ H ₂₂ ClN ₄ O ₅ P	6.60	10.44	5.77
Xa	82	236–238 (ethanol)	–	37.37	–	C ₆ H ₆ N ₄ O	–	37.32	–
Xb	78	248–250 (ethanol)	–	24.82	–	C ₁₂ H ₁₀ N ₄ O ^b	–	24.76	–
Xc	45	143–145 (ethanol–diethyl ether, 2 : 1)	–	34.20	–	C ₇ H ₈ O	–	34.13	–
Xd	51	102–104 (ethanol)	–	31.84	–	C ₈ H ₈ N ₄ O	–	31.80	–
XI	82	198–200 (ethanol–diethyl ether, 1 : 1)	(23.58)	5.10	5.35	C ₂₈ H ₂₄ IN ₂ P	(23.23)	5.13	5.67

^a Found, %: C 73.89; H 4.74. Calculated, %: C 73.87; H 4.70. ^b Found, %: C 63.74; H 4.43. Calculated, %: C 63.71; H 4.46.

The central bicyclic system N^{1–4}C^{1–5} is practically planar: The deviations of atoms from the mean plane are no larger than 0.065 and 0.13 Å. The dihedral angle between the six-membered ring N¹N²C^{1–4} and the five-membered ring N^{2–4}C³C⁵ is as small as 3.9%. The geometric parameters of the bicyclic system suggest significant delocalization of electron density [4, 5]. The P¹ and N⁵ atoms deviate from this plane

by 0.215 and 0.096 Å, respectively (the exocyclic P¹–C¹ and N⁵–C⁴ bonds form angles of 4.9° and 2.7° with the bicyclic system). The P¹...C¹ distance is 1.756(3) Å, which allows compound **VIIa** to be classed with superstabilized phosphonium ylides (cf. [6, 7]). Therefore, along with the nonpolar phosphino-methylene structure depicted in the scheme, betaine structures **A–D** play an important role.



In spite of their clearly defined mesomeric nature, ylide betaines **VII** are quite selectively alkylated, which has made possible the transformation sequence **VII** → **VIII** → **IX** → **X**. One of the final products of these transformations, 4-methyl-4,7-dihydro-s-triazolo-[1,5a]pyrimidin-7-one (**Xa**), was prepared previously by an independent procedure [8].

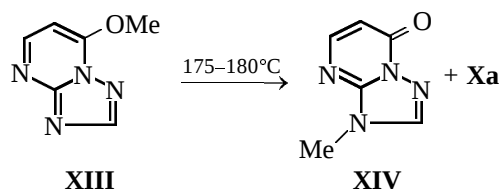
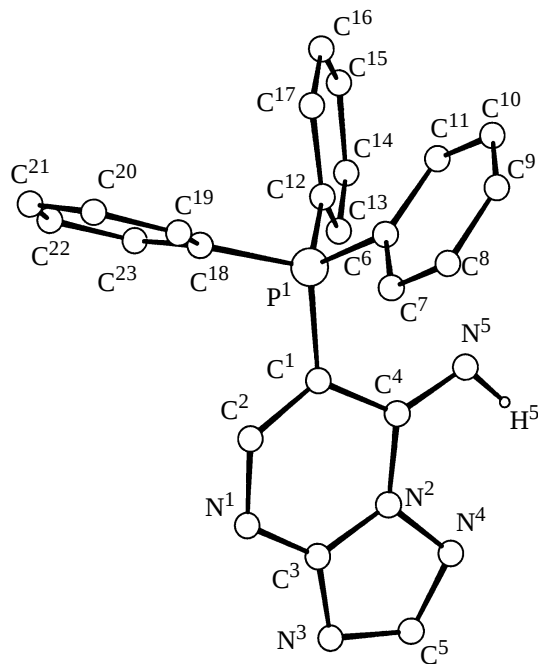


Table 3. Principal bond lengths (*d*, Å) and bond angles (ω, deg) in molecule **VIIa**

Bond	<i>d</i>	Angle	ω
P ¹ –C ¹	1.756(3)	C ¹ P ¹ C ⁶	111.19(12)
P ¹ –C ⁶	1.809(2)	C ¹ P ¹ C ¹²	113.99(12)
P ¹ –C ¹²	1.802(2)	C ⁶ P ¹ C ¹²	109.63(12)
P ¹ –C ¹⁸	1.806(3)	C ¹ P ¹ C ¹⁸	108.11(12)
N ¹ –C ²	1.328(3)	C ⁶ P ¹ C ¹⁸	109.43(12)
N ¹ –C ³	1.352(3)	C ¹² P ¹ C ¹⁸	104.19(13)
N ² –N ⁴	1.371(3)	C ² N ¹ C ³	113.9(2)
N ² –C ³	1.370(3)	N ⁴ N ² C ³	110.4(2)
N ² –C ⁴	1.408(3)	C ³ N ² C ⁴	125.4(2)
N ³ –C ³	1.330(3)	C ³ N ³ C ⁵	102.1(2)
N ³ –C ⁵	1.355(4)	N ² N ⁴ C ⁵	100.5(2)
N ⁴ –C ⁵	1.316(3)	C ⁴ N ⁵ H ⁵	108.1(18)
N ⁵ –C ⁴	1.280(3)	C ² C ¹ C ⁴	120.1(2)
C ¹ –C ²	1.401(3)	N ¹ C ² C ¹	126.7(2)
C ¹ –C ⁴	1.439(3)	N ¹ C ³ N ²	123.1(2)
N ⁵ –H ⁵	0.91(3)	N ² C ³ N ³	109.3(2)
		N ² C ⁴ C ¹	110.5(2)
		N ³ C ⁵ N ⁴	117.7(2)

The yield of compound **Xa** in that case did not exceed 50%, and, moreover, the product should be separated from isomer **XIV** by column chromatography. The application scope of the thermal rearrangement **XIII** → **X** cannot be wide, and, therefore, the use of stabilized phosphonium ylide betaines **VII** for regioselective synthesis 2-R-4-alkyl(alkenyl)-4,7-di-

General view of molecule **VIIa**.

hydro-*s*-triazolo[1,5-*a*]pyrimidin-7-ones **X** is of preparative value. It is quite possible that this approach can be extended to prepare such complex analogs of compounds **X** as acylnucleosides.

EXPERIMENTAL

The ¹H NMR spectra were obtained on a Varian VXR-300 spectrometer for (CD₃)₂SO solutions against internal TMS.

X-ray diffraction study of a single crystal (0.50 × 0.52 × 0.53 mm) of compound **VIIa** was carried out at room temperature on an Enraft–Nonius CAD-4 automatic four-circle diffractometer (MoK_α radiation; 2θ/ω 1.2; θ_{max} 27°; index ranges 0 < *h* < 13, 0 < *k* < 19, and –17 < *l* < 17). A total of 2731 reflections was collected, 2402 of which were unique (*R*_{int} 0.019). Crystals of compound **I** are monoclinic, *a* 12.880(2), *b* 9.344(5), *c* 16.478(14) Å, β 96.24(6)°, *V* 1971(2) Å³, *M* 395.41, *Z* 4, *d*_{calc} 1.33 g cm^{–3}, μ 1.53 cm^{–1}, and space group *P*2₁/*c* (no. 14). The structure was solved by the direct method and refined by anisotropic least squares using the CRYSTALS program package [9]. Absorption was included by means of azimuthal scanning [10]. Refining was carried out by 1893 reflections with *I* > 3σ(*I*) (266 refined parameters, 7.1 reflections per one parameter). All hydrogen atoms were obtained from difference synthesis and included in the calculations with fixed positional and thermal parameters (H⁵ was refined isotropically). The Chebyshev weight scheme [11] with five parameters, 1.19, 0.77, 1.13, 0.26, and 0.32, was used the refinement. The final values of divergence factors were *R* 0.033 and *R*_w 0.035, *GOF* 1.153. The residual electron density from the differential Fourier series is 0.15 and –0.20 e Å^{–3}.

[1-Cyano-2-(2-pyridylamino)vinyl]triphenylphosphonium iodide III. A mixture of 0.01 mol of phosphonium salt **I** and 0.01 mol of 2-aminopyridine in 30 ml of acetonitrile was refluxed for 2 h, left for 1 h at 20–25°C, the solvent was removed in a vacuum, and the residue was purified by crystallization.

[2-R-7-Imino-4,7-dihydro-*s*-triazolo[1,5-*a*]pyrimidin-6-yl]triphenylphosphonium iodides IVa and IVb. A mixture of 0.01 mol of phosphonium salt **I** and 0.01 mol of amino-*s*-triazole or 3-amino-5-phenyl-*s*-triazole in 40 ml of acetonitrile was refluxed for 5 h and left for 12 h at 20–25°C. The precipitate was filtered off and purified by crystallization.

4-Amino-2-phenylpyridine (V). To a solution of 0.005 mol of phosphonium salt **I** in 30 ml of methanol, 0.005 mol of benzamidine and a solution of 0.01 mol of sodium hydroxide in 5 ml of methanol

was added. The resulting mixture was left for 10 days at 20–25°C, the methanol was removed in a vacuum, the residue was washed with 20 ml of water, and the precipitate was purified by crystallization from ethanol. Yield 60%, mp 134–136°C, what agrees with data reported in [3].

7-Imino-6-triphenylphosphoranylidene-6,7-dihydro-s-triazolo[1,5-*a*]pyrimidine (VIIa). To a suspension of 0.01 mol of compound **IVa** or **IVb** in 30 ml of methanol, 0.01 mol of sodium methylate was added. The resulting mixture was heated at 40–50°C to dissolve the precipitate, and the solution was left for 1 h at 20–26°C. Water, 20 ml, was then added, and the mixture was left for 1 h. A precipitate formed and was filtered off and purified by crystallisation.

7-Imino-2-phenyl-6-triphenylphosphoranylidene-6,7-dihydro-s-triazolo[1,5-*a*]pyrimidine (VIIb) was prepared analogously to compound **VIIa**, but after heating at 40–50°C for 10–15 min and cooling to 20–25°C, the precipitate was filtered off and purified by crystallization.

[7-Imino-4-methyl(ethyl)-4,7-dihydro-s-triazolo[1,5-*a*]pyrimidin-6-yl]triphenylphosphonium perchlorates VIIIa and VIIIc. A mixture of 0.01 mol of compound **VIIa** and 10 ml of methyl or ethyl iodide was left for 12 h at 20–25°C. Volatile substances were removed in a vacuum. The oily residue was dissolved in 30 ml of ethanol, and 20 ml of the saturated water solution of sodium perchlorate was added. The precipitate was filtered off, washed with water, and purified by crystallization.

[7-Imino-4-methyl-2-phenyl-4,7-dihydro-s-triazolo[1,5-*a*]pyrimidin-6-yl]triphenylphosphonium iodide (VIIIb) was prepared similarly to compounds **VIIIa** and **VIIIc**, but the residue after removal of methyl iodide was treated with 30 ml of ethanol, and the precipitate was filtered off and purified by crystallization.

[4-Allyl-7-imino-4,7-dihydro-s-triazolo[1,5-*a*]pyrimidin-6-yl]triphenylphosphonium perchlorate (VIIId). To a solution of 0.01 mol of compound **VIIa** in 30 ml of dichloromethane, 0.012 mol of allyl bromide was added. The resulting mixture was left at 20–25°C for 12 h, the solvent was removed in a vacuum, the residue was dissolved in 20 ml of ethanol, 20 ml of saturated aqueous sodium perchlorate was added. The precipitate was filtered off, washed with water, and purified by crystallization.

[4-Methyl(ethyl, allyl)-7-oxo-4,7-dihydro-s-triazolo[1,5-*a*]pyrimidin-6-yl]triphenylphosphonium perchlorates (IXa, IXc, IXd). A mixture of 0.05 mol of compounds **VIIIa**, **VIIIc**, or **VIIIId** and 25 ml of

Table 4. Atomic coordinates and equivalent isotropic thermal parameters U_{eq} (Å²) in molecule **VIIa**

Atom	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
P ¹	0.23578(4)	0.23950(6)	0.35316(4)	0.0357
N ¹	0.41912(15)	−0.0217(2)	0.23695(13)	0.0500
N ²	0.26635(14)	0.0313(2)	0.14596(11)	0.0426
N ³	0.37557(18)	−0.1269(2)	0.10430(14)	0.0585
N ⁴	0.22170(17)	−0.0086(2)	0.07000(12)	0.0553
N ⁵	0.13657(17)	0.1980(3)	0.17729(14)	0.0564
C ¹	0.28188(17)	0.1359(2)	0.27572(14)	0.0377
C ²	0.37756(18)	0.0637(3)	0.28911(15)	0.0448
C ³	0.35851(18)	−0.0403(3)	0.16546(15)	0.0444
C ⁴	0.22092(18)	0.1281(2)	0.19727(14)	0.0405
C ⁵	0.2908(2)	−0.1023(3)	0.04993(16)	0.0608
C ⁶	0.23297(17)	0.4275(2)	0.32692(13)	0.0363
C ⁷	0.32072(18)	0.4847(3)	0.29639(15)	0.0449
C ⁸	0.3235(2)	0.6298(3)	0.27780(17)	0.0545
C ⁹	0.2397(2)	0.7157(3)	0.28912(17)	0.0547
C ¹⁰	0.1517(2)	0.6589(3)	0.31797(17)	0.0570
C ¹¹	0.14788(18)	0.5153(3)	0.33699(15)	0.0470
C ¹²	0.10949(17)	0.1853(2)	0.37968(13)	0.0383
C ¹³	0.0543(2)	0.0722(3)	0.34082(15)	0.0509
C ¹⁴	0.0418(2)	0.0343(3)	0.36531(19)	0.0672
C ¹⁵	0.0821(2)	0.1091(4)	0.4264(2)	0.0729
C ¹⁶	0.0268(2)	0.2190(3)	0.46573(17)	0.0640
C ¹⁷	0.06935(18)	0.2560(3)	0.44356(15)	0.0484
C ¹⁸	0.32218(17)	0.2124(2)	0.44578(13)	0.0391
C ¹⁹	0.3966(2)	0.3101(3)	0.47511(16)	0.0514
C ²⁰	0.4564(2)	0.2849(3)	0.54865(17)	0.0602
C ²¹	0.4433(2)	0.1648(3)	0.59284(15)	0.0591
C ²²	0.3721(3)	0.0631(4)	0.56201(19)	0.0807
C ²³	0.3118(2)	0.0868(3)	0.48884(18)	0.0696
H ⁵	0.112(2)	0.174(3)	0.1255(18)	0.071(9)

conc. HCl was heated for 5 h at 90–100°C and then poured into an ice water. The precipitate was filtered off and purified by crystallization.

[4-Methyl-7-oxo-2-phenyl-4,7-dihydro-s-triazolo[1,5-*a*]pyrimidin-6-yl]triphenylphosphonium perchlorate (IXb) was prepared similarly to compounds **IXa**, **IXc**, and **IXd**, but after heating with HCl the product was dissolved in 15 ml of acetonitrile, and 20 ml of saturated aqueous sodium perchlorate was added. The precipitate was filtered off, washed with water, and purified by crystallization.

4-Methyl-4,7-dihydro-s-triazolo[1,5-*a*]pyrimidin-7-one (Xa). A mixture of 0.006 mol of compound **IXa**, 20 ml of acetonitrile, 0.006 mol, and 1 ml of water was refluxed for 4–5 h, the solvent was removed in a vacuum, and the solid residue was treated

with 10 ml of ethanol. The precipitate was filtered off and purified by crystallization.

4-Methyl-2-phenyl-4,7-dihydro-s-triazolo[1,5-a]pyrimidin-7-one (Xb) was prepared similarly to compound **Xa**.

4-Ethyl(allyl)-4,7-dihydro-s-triazolo[1,5-a]pyrimidin-7-ones Xc and Xd were prepared similarly to compounds **Xa** and **Xb** but isolated by column chromatography on silica gel, eluent chloroform–methanol, 100:2.

(1-Cyano-2-*p*-toluidinovinyl)triphenylphosphonium iodide (XI). A mixture of 0.01 mol of phosphonium salt **I** and 0.01 mol of *p*-toluidine in 30 ml of acetonitrile was refluxed for 2 h, left for 1 h at 20–25°C, and the precipitate was filtered off and purified by crystallization.

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