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Chemistry of Diazopolycarbonyl Compounds: VIII.* Synthesis of Nitrogen-containing Heterocycles via Transformations of Substituted 2-diazopentane-1,3,5-triones

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Abstract—Ethyl 5-aryl-2-diazo-3,5-dioxopentanoates and 1,5-diaryl-2-diazopentane-1,3,5-triones are partially enolized in solutions. By O-methylation of enol forms of diazo esters with diazomethane ethyl 5-aryl-2-diazo-5-methoxy-3-oxopent-4-enoates were prepared. Concurrently with the O-methylation the diazo esters undergo heterocyclization into 3,5-disubstituted 4-hydroxypyrazoles which under the reaction condition suffer O- and N-methylation by excess diazomethane. 3,5-Diaroyl-4-hydroxypyrazoles were also obtained from diazopentanetriones but here triethylamine served as the cyclization reagent.

A general procedure was developed formerly for aroylacetylation of diazocarbonyl compounds with aroylketenes generated by thermal decarbonylation of 5-aryl-2,3-dihydrofuran-2,3-diones **Ia–e** [2–5]. Aroylketenes acetylate aroyldizomethanes [2], adamantanoyldiazomethanes [3], ethyl diazoacetate [4], and alkyl 3-diazo-2-oxopropanoates [5] affording respectively 1,3-diaryl-2-diazopentane-1,3,5-triones [2], 1-(1-adamantyl)-5-aryl-2-diazopentane-1,3,5-triones [3], ethyl 5-aryl-2-diazo-3,5-dioxopentanoates [4], and alkyl 6-aryl-3-diazo-2,4,6-trioxohexanoates [5]. It was shown [3–5] that these diazopolycarbonyl compounds treated with triethylamine or primary amines were capable to undergo cyclization affording pyrazole derivatives, and under treatment with triphenylphosphine ethyl 5-aryl-2-diazo-3,5-dioxopentanoates furnished pyridazine derivatives [6, 7]. We carried on the research in the field of cyclization of polycarbonyl diazo compounds proceeding under mild conditions without nitrogen liberation from the diazo group.

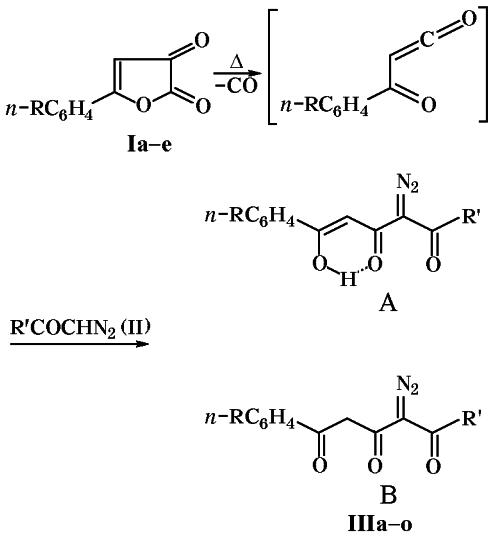
First of all we performed acylation of ethyl diazoacetate and aroyldiazomethanes with aroylketenes by procedures [2–4] and obtained already described compounds **IIIa–h** and previously unknown compounds **IIIi–o**: ethyl 5-aryl-2-diazo-3,5-dioxopentenoates **IIIa–e**, 1,5-diaryl-2-diazopentane-1,3,5-triones **IIIf–o** (Tables 1, 2, Scheme 1).

* Communication VII see [1].

The IR spectra of compounds **IIIi–o** are well consistent with those of diazopentanetriones of similar structure [4].

In the mass spectrum of compound **IIIm** the molecular ion peak is lacking, and appear peaks of the

Scheme 1.



I), R = H (**a**), CH_3 (**b**), CH_3O (**c**), Cl (**d**), Br (**e**); **III**, R' = H_5C_2O, R = H (**a**), CH_3 (**b**), CH_3O (**c**), Cl (**d**), Br (**e**); R' = C_6H_5, R = H (**f**), CH_3 (**g**), Cl (**h**), Br (**i**); R' = 4-$O_2NC_6H_4$, R = H (**j**), CH_3O (**k**), Cl (**l**), Br (**m**); R' =4-$CH_3C_6H_4$, R = CH_3O (**n**); R' = 4-BrC_6H_4, R = CH_3O (**o**).

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Table 1. Yields, melting points, and IR spectra of compounds **IIIi–o**

Compd. no.	Yield, %	mp, °C	IR spectrum, ν, cm^{-1} (in mineral oil)
IIIi	73.8	96–98	2107 (N_2), 1605, 1696 (C=O)[a]
IIIj	22.4	123–124	2123 (N_2), 1635, 1627 (C=O)
IIIk	32.9	138–140	2128 (N_2), 1653, 1635, 1628 (C=O), 2127 (N_2), 1653, 1635 (C=O)[a]
IIIl	49.4	140–142	2127 (N_2), 1628, 1624 (C=O)
IIIm	63.9	143–145	2125 (N_2), 1628, 1624 (C=O)
IIIn	48.5	117–118	2131 (N_2), 1628, 1624 (C=O)
IIIo	66.0	132–135	2131 (N_2), 1628, 1624 (C=O), 2129 (N_2), 1625, 1602 (C=O)[a]

[a] Spectra registered from KBr pellets.

Table 2. Parameters of ^{1}H NMR spectra of compounds **IIIa, b, d, e, i, m, n** in DMSO-d_6, δ, ppm

Compd. no.	CH_3, CH_2[a]	$COCH_2CO$, s	=CH, s	H arom, m	OH, br.s	Ratio enol–ketone
IIIa	1.31 t (3H, CH_3), 4.31 q (2H, CH_2), 1.15 t (3H, CH_3), 4.15 q (2H, CH_2)	4.62	7.12	7.70	–	53 : 47
IIIb	1.32 t (3H, CH_3), 4.32 q (2H, CH_2), 2.40 s (3H, $\underline{CH}_3C_6H_4$), 1.19 t (3H, CH_3), 4.18 (2H, CH_2), 2.40 s (3H, $\underline{CH}_3C_6H_4$)	4.49	7.08	7.55	15.50	54 : 46
IIId	1.35 t (3H, CH_3), 4.30 q (2H, CH_2), 1.19 t (3H, CH_3), 4.17 q (2H, CH_2)	4.43	7.10	7.75	–	79 : 21
IIIe	1.35 t (3H, CH_3), 4.30 q (2H, CH_2), 1.28 t (3H, CH_3), 4.26 q (2H, CH_2)	4.52	7.19	7.80	15.35	62 : 38
IIIi[b]	–	4.59	7.16	7.75	–	64 : 36
IIIm[b]	–	4.56	7.19	7.80	–	78 : 22
IIIn[b]	2.47 s (3H, $\underline{CH}_3C_6H_4$), 3.86 s (3H, $\underline{CH}_3\underline{O}C_6H_4$), 2.44 s (3H, $\underline{CH}_3C_6H_4$), 3.92 s (3H, $\underline{CH}_3\underline{O}C_6H_4$)	4.51	7.09	7.50	–	76 : 24

[a] Two sets of aliphatic protons signals correspond respectively to enol and keto forms.

[b] The spectra were registered in mixed solvent, DMSO-d_6–CCl_4, 1 : 3.

fragment ions [*m/z* (I_{rel}, %)]: 389/387 (73/92) [$M - N_2$]$^{+\cdot}$, 185/183 (75/88) [BrC_6H_4CO]$^+$, 150 (100) [$O_2NC_6H_4CO$]$^+$. In the mass spectrum of compound **IIIn** also are present peaks of fragment ions [*m/z* (I_{rel}, %)]: 308 (64) [$M - N_2$]$^{+\cdot}$, 280 (18) [$M - N_2$–CO]$^{+\cdot}$, 177 (48) [$CH_3OC_6H_4COCH_2CO$]$^+$, 135 (100) [$CH_3OC_6H_4CO$]$^+$, 119 (62) [$CH_3C_6H_4CO$]$^+$, 91 (43) [$CH_3C_6H_4$]$^+$ in conformity with the assumed structure.

According to ^{1}H NMR spectra described in [3, 4] diazo compounds **IIIa–h** were completely enolized in CCl_4 and CH_2Cl_2 solutions and existed in A form with an intramolecular hydrogen bond of H-chelate type. We registered ^{1}H NMR spectra of compounds **IIIa, b, d, e, i, m, n** and found that in DMSO-d_6 solution both enol (A) and β-diketo (B) forms were present.

Therewith the content of the latter depended on the character of R'-substituent and varied from 21 to 47% (Table 2). In the spectra of compounds **IIIa, d, i, m, n** the signal of enol hydroxy proton was not revealed evidently because of its considerable broadening. Just the existence of both forms in solutions of compounds **III** governs the features of their chemical behavior.

It was reported in [3, 4] on intramolecular cyclization of diazoesters **IIIa–d** and diazopentanetriones **IIIf, h** into pyrazole derivatives induced by organic bases, e.g., by triethylamine. We studied reactions of diazo compounds **IIIa–e, i–m, o** with a nucleophile like diazomethane and established that in reaction with diazomethane according to TLC data formed a complex mixture of products whose composition and structure depended on the reaction conditions.

For instance, in reactions of diazoesters **IIIa–e** and diazoketones **IIIi–m, o** with an equimolar amount of dizomethane in ethyl ether at −5 ÷ 0°C from the reaction mixture were isolated respectively 5-aroyl-4-hydroxy-3-ethoxycarbonylpyrazoles **IVa–e** and 3,5-diaroyl-4-hydroxypyrazoles **IVf–k** (Scheme 2).

Table 3. Yields, melting points, IR and ^{1}H NMR spectra of compounds **IVe–k, Va–c, VIa–c**

Compd. no.	Yield, %	mp, °C	IR spectrum, ν, cm^{-1}	^{1}H NMR spectrum, δ, ppm[a]
IVe	67[b]	216–217	3329, 3196 (NH, OH), 1676 ($\underline{CO}OC_2H_5$), 1628 (C=O)	1.42 t (3H, CH_3), 4.40 q (2H, CH_2), 7.68 d (2H, C_6H_4), 7.88 d (2H, C_6H_4), 9.39 s (1H, NH), 14.11 s (1H, OH)
IVf	68	271–273	3200 sh, 3159 (NH, OH), 1632, 1608 (C=O)	7.80 m (9H, C_6H_5, C_6H_4), 9.85 s (0.5H, NH), 10.08 s (0.5H, NH), 14.25 s (1H, OH)
IVg	62	295–296	3322, 3211, 3184 (NH, OH), 1637, 1620, 1611 (C=O)	8.10 m (9H, C_6H_5, C_6H_4), 9.68 s (0.4H, NH), 10.11 s (0.6H, NH), 14.38 s (1H, OH)
IVh	60	272–274	3148 br (NH, OH), 1634, 1600 br (C=O)	3.92 s (3H, CH_3O), 8.20 m (8H, $2C_6H_4$), 9.70 br.s (0.5H, NH), 10.35 br.s (0.5H, NH), 14.30 br.s (1H, OH)
IVi	35	233–235	3210 sh, 3170 (NH, OH), 1638, 1610 (C=O)	8.25 m (8H, $2C_6H_4$), 9.90 br.s (1H, NH), 14.40 br.s (1H, OH)
IVj	38	220–222	3165 br (NH, OH), 1637, 1601 (C=O)	8.25 m (8H, $2C_6H_4$), 9.75 s (0.4H, NH), 9.99 s (0.6H, NH), 14.42 s (1H, OH)
IVk	83	276–277	3210 sh, 3156 br, (NH, OH), 1634, 1602 (C=O)	3.91 s (3H, CH_3), 7.75 m (8H, $2C_6H_4$), 9.87 br.s (0.4H, 10 32 br.s. (0.6H, NH), 14.18 br.s.
Va	50 84[c]	56–57	1712 ($C\underline{OO}C_2H_5$), 1656 (C=O)	1.41 t (3H, $C\underline{H}_3CH_2$), 3.57 s (3H, NCH_3), 4.04 s (3H, CH_3O), 4.34 q (2H, $CH_3C\underline{H}_2$), 7.65m (5H, C_6H_5)
Vb	45, 79[c]	98–100	1712 ($COOC_2H_5$), 1654 (C=O)	–
Vc	48, 87[c]	65–67	1718 ($C\underline{OO}C_2H_5$), 1644 (C=O)	1.31 t (3H, $C\underline{H}_3CH_2$), 2.11 s (3H, $C\underline{H}_3N$), 3.75 s (3H, $C\underline{H}_3OC_6H_4$), 4.05 s (3H, CH_3O), 4.30 q (2H, $CH_3C\underline{H}_2$), 7.00 q (4H, C_6H_4), 7.92 (2H, C_6H_4)
VIa	46	43–45	2136 (N_2), 1720 br (C=O)	–
VIb	39	69–70	2132 (N_2), 1718 br (C=O)	1.18 t (3H, $C\underline{H}_3CH_2$), 2.15 s (3H, $C\underline{H}_3C_6H_4$), 3.78 s (3H, CH_3O), 4.18 q (2H, $CH_3C\underline{H}_2$), 6.68 s (1H, CH), 7.15 q (4H, C_6H_4), 7.55 d (2H, C_6H_4)
VIc	41	79–80	2146 (N_2), 1712 br (C=O)	1.32 t (3H, $C\underline{H}_3CH_2$), 3.88 s (3H, CH_3O), 4.22 q (2H, $CH_3C\underline{H}_2$), 6.78 s (1H, CH), 7.26 d (2H, C_6H_4), 7.56 d (2H, C_6H_4)

[a] Solutions of compounds **IVe–k, Va** in DMSO-d_6–CCl_4, 1:3, of compounds **Vc, VIb** in DMSO-d_6, of compound **VIc** in $CDCl_3$.
[b] The reported yield corresponds to cyclization of the respective pentanetrione effected by triethylamine.
[c] Yield of reaction product formed from the corresponding pyrazole and diazomethane.

Compounds **IVa–k** were isolated from the reaction mixture in amounts not exceeding 10% from the theoretical quantity for even at the equimolar ratio of the reagents the reaction took several routes. The previously unknown pyrazoles **IVe–k** were also obtained by an independent synthesis treating diazo compounds **IIIe, i–m, o** with triethylamine along procedure [4] (Table 3).

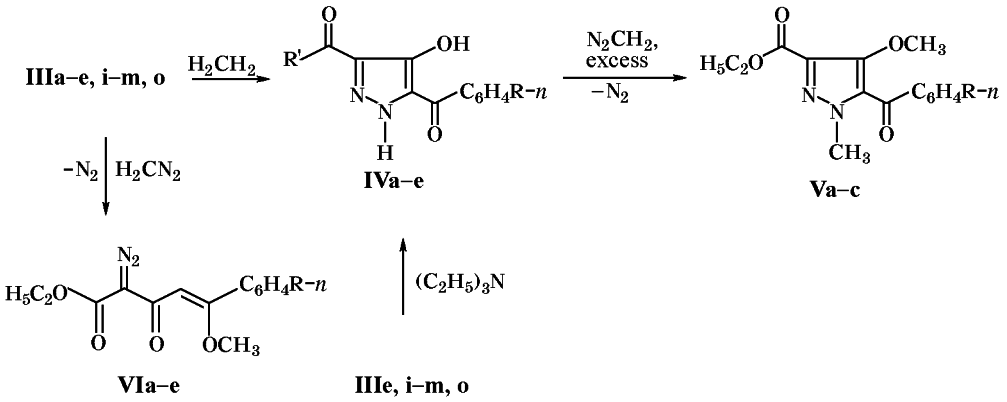
In the IR spectra recorded from mulls of compounds **IVe–k** in mineral oil are present broad absorption bands or plateau at 3148–3329 cm^{-1} characteristic of the NH bonds in the heterocycle and a broadened absorption band of the hydroxy group in the region 3148–3184 cm^{-1}. In the spectra of compounds **IVh, j** a single joint broad band appeared respectively in the region 3148 and 3165 cm^{-1}. The absorption bands of carbonyl groups of the aroyl substituents are present in the regions 1628–1638 and 1600–1611 cm^{-1}. The ester carbonyl gives rise to an absorption band in the IR spectrum of compound **IVe** at 1676 cm^{-1}.

In the mass spectrum of pyrazole **IVj** unlike the mass spectrum of initial diazopentanetrione **IIIm** the molecular peak is observed with *m/z* 415/417 (12/10) $[M]^{+\cdot}$ and fragment ions (*m/z*, I_{rel}, %): 183/185 (100/95) $[BrC_6H_4CO]^+$, 155/157 (48/42) $[BrC_6H_4]^+$, 150 (79) $[O_2NC_6H_4CO]^+$.

According to ^{1}H NMR spectra compounds **IVf–k** exist in solution as two tautomers. As a result the signals of NH and OH protons in the spectrum of compound **IVi** are broadened, and the protons of NH group in the spectra of compounds **IVf–h, j, k** appear as two singlets.

The data of ^{1}H NMR spectra, the shift of absorption bands of enol hydroxy and carbonyl groups in

Scheme 2.



V, R = H_5C_2O, R = H (**a**), CH_3 (**b**), CH_3O (**c**), Cl (**d**), Br (**e**); R' = C_6H_5, R = Br (**f**); R' = 4-$O_2NC_6H_4$, R = H (**g**), CH_3O (**h**), Cl (**i**), Br (**j**); R' = 4-BrC_6H_4, R = CH_3O (**k**); **V**, R = H (**a**), CH_3 (**b**), CH_3O (**c**); **VI**, R = H (**a**), CH_3 (**b**), Cl (**c**).

the IR spectra to the lower frequency region, and the presence of low-frequency absorption bands of NH group indicate that compounds **IVe–k** exist in forms stabilized by intramolecular hydrogen bonds whose character and strength depend on substituents R and R'. Compounds **IVa–d** (R' = C_2H_5O) have a structure of ethyl 3-pyrazolecarboxylates [4], but in [4] has been assumed an existence of a single hydrogen bond between a carbonyl and an enol hydroxy group. However the shift to lower frequencies in the IR spectra of compounds **IV** of the absorption of both carbonyl groups suggests that both carbonyls are involved into hydrogen bonds with the enol hydroxyl and NH group of the heterocycle respectively. This assumption is supported by the chemical transformations of compounds **IV**.

Thus the reaction of diazoesters **IIIa–c** with excess diazomethane gave rise to 5-aroyl-1-methyl-4-methoxy-3-ethoxycarbonylpyrazoles **Va–c**. Compounds **Va–c** are products of exhaustive methylation of substances **IVa–c**, and they were also prepared by direct methylation with diazomethane of pyrazoles **IVa–c** (Table 3). In the IR spectra of pyrazoles **Va–c** the absorption bands of both carbonyls are shifted to high-frequency region compared to the corresponding bands in the spectra of initial compounds **IVa–c**.

The cyclization mechanism of diazo compounds **III** into pyrazole derivatives **IV** when treated with diazomethane is likely to follow the same pattern as that described in [4] for reaction of enol form A with triethylamine. However in the former case the reaction is catalyzed by trace amounts of alkali retained in diazomethane prepared by decomposition of methylnitrosourea [8]. Thus suggestion was supported by the experiment where the reaction of diazoesters **IIIa, b, d** was carried out with a distilled ether solution of diazomethane. Thus we suppressed pyrazoles **IVa, b, d** formation and succeeded in isolation from the reaction mixture of ethyl 5-aryl-2-diazo-5-methoxy-3-oxopentenoates **VIa–c** in 39–46% yield (Table 3). It should be noted that diazoesters **VIa–c** arise in trace amounts also in reaction of compounds **IIIa, b, d** with undistilled solution of diazomethane (TLC data).

The O-methylation of diazoesters **III** and their heterocyclization into pyrazoles **IV** are concurrent processes: diazoesters **VIa, b** do not undergo cyclization into pyrazoles **IVa, b** under treatment with diazomethane.

EXPERIMENTAL

IR spectra were recorded on spectrophotometers Specord M-80 or FSM-1201 from mulls in mineral oil. ^{1}H NMR spectra were registered on spectrometer Bruker WR-80-SY (80 MHz), internal reference HMDS, solvents $CHCl_3$, DMSO-d_6. Mass spectra were measured on Varian MAT-311A instrument in the following mode: emission current 1000mA, ionizing electrons energy 70 eV, vaporizer temperature 120–150°C, source temperature 200°C. The reaction progress was monitored, purity and homogeneity of compounds synthesized was checked by TLC on Silufol UV-254 plates, eluent ether–benzene–acetone, 10 : 9 : 1, development in iodine vapor. The elemental analyses are given in Table 4.

1,5-Diaryl-2-diazopentane-1,3,5-triones IIIi–o. A solution of 0.01 mol of 5-aryl-2,3-dihydrofuran-2,3-dione **Ia–e** and 0.01 mol of diazo compound **II** in

Table 4. Elemental analyses of compounds **IIIi–o, IVe–k, V–VIa–c**

Compd. no.	Found, %				Formula	Calculated, %			
	C	H	Hlg	N		C	H	Hlg	N
IIIi	54.91	3.11	21.58	7.62	$C_{17}H_{11}BrN_2O_3$	55.0	2.99	21.53	7.55
IIIj	60.62	3.20	–	12.33	$C_{17}H_{11}N_3O_5$	60.54	3.29	–	12.46
IIIk	58.71	3.69	–	11.48	$C_{18}H_{13}N_3O_6$	58.86	3.57	–	11.44
IIIl	54.80	2.79	9.50	11.46	$C_{17}H_{10}ClN_3O_5$	54.93	2.71	9.54	11.3
IIIm	49.14	2.32	19.31	10.01	$C_{17}H_{10}BrN_3O_5$	49.06	2.42	19.20	10.09
IIIn	67.83	4.70	–	8.21	$C_{19}H_{16}N_2O_4$	67.85	4.79	–	8.33
IIIo	53.97	3.38	19.98	6.91	$C_{18}H_{13}BrN_2O_4$	53.88	3.26	19.91	6.98
IVe	46.19	3.16	23.41	8.38	$C_{13}H_{11}BrN_2O_4$	46.04	3.27	23.56	8.26
IVf	55.18	2.16	21.57	8.19	$C_{17}H_{11}BrN_2O_3$	55.0	2.99	21.53	7.55
IVg	60.59	3.17	–	12.30	$C_{17}H_{11}N_3O_5$	60.54	3.29	–	12.46
IVh	58.75	3.49	–	11.32	$C_{18}H_{13}N_3O_6$	58.86	3.57	–	11.44
IVi	55.0	2.79	9.47	11.38	$C_{17}H_{10}ClH_3O_5$	54.93	2.71	9.54	11.30
IVj	49.13	2.31	19.39	10.00	$C_{17}H_{10}BrN_3O_5$	49.06	2.42	19.20	10.09
IVk	53.99	3.31	19.80	6.81	$C_{18}H_{13}BrN_2O_4$	53.88	3.26	19.91	6.98
Va	62.58	6.99	–	9.90	$C_{15}H_{16}N_2O_4$	62.49	7.06	–	9.72
Vb	63.69	6.05	–	9.09	$C_{16}H_{18}N_2O_4$	63.57	6.00	–	9.27
Vc	60.29	5.79	–	8.71	$C_{16}H_{18}N_2O_5$	60.37	5.70	–	8.80
VIa	61.40	5.08	–	10.12	$C_{14}H_{14}N_2O_4$	61.31	5.14	–	10.21
VIb	62.37	5.70	–	9.59	$C_{15}H_{16}Cl_2O_4$	62.49	5.59	–	9.72
VIc	54.39	4.42	11.60	9.00	$C_{14}H_{13}ClN_2O_4$	54.47	4.24	11.48	9.07

40 ml of anhydrous benzene or CCl_4 was heated at reflux for 2 h. The solvent was evaporated, the residue was recrystallized from hexane (compound **IIIi**), acetonitrile (compounds **IIIj–m**), or ethanol (compounds **IIIn, o**).

5-Aroyl-4-hydroxy-3-ethoxycarbonylpyrazoles IVa–e, 3,5-diaroyl-4-hydroxypyrazoles IVf–k. A solution of 0.01 mol of diazo compound **III** and 0.01 mol of triethylamine in 50 ml of ethanol was kept for 48 h at 20–25°C. The precipitate was filtered off and recrystllized from ethanol or acetone.

5-Aroyl-1-methyl-4-methoxy-3-ethoxycarbonylpyrazoles Va–c. (a) To a solution of 0.01 mol of diazoester **IIIa** in 30 ml of chloroform at –5÷0°C while stirring was added a solution of 0.04 mol of diazomethane in 60 ml of ether, and the mixture was maintained for 24 h at –5÷0°C. The solvent was evaporated, the residue was recrystallized from methanol.

(b) To a suspension of 0.01 mol of compound **IVa** in 30 ml of toluene at –5÷0°C while stirring was added a solution of 0.03 mol of diazomethane in 45 ml of ether, and the mixture was stirred till complete dissolution of the precipitate. The solvent was evaporated, the residue recrystallized.

Ethyl 5-aryl-2-diazo-5-methoxy-3-oxopentenoates VIa–c. To a solution of 0.01 mol of diazoester **IIIa** or **IIIb, c** in 15 ml of anhydrous acetone at –5÷0°C while stirring was added a solution of 0.03 mol of diazomethane in 45 ml of ether that had been preliminary dried over KOH pellets and distilled. In 8 h the solvent was evaporated, and the residue recrystallized from methanol.

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