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Synthesis, Structure, and Antimicrobial Activity of *N*,6-Diaryl-4-methyl-2-oxo-1,2,3,6-tetrahydropyrimidine-5-carboxamides

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Abstract—Reactions of acetoacetic acid *N*-arylamides with aromatic aldehydes and urea led to the formation of *N*,6-diaryl-4-methyl-2-oxo-1,2,3,6-tetrahydropyrimidine-5-carboxamides. Structure and antimicrobial activity of the compounds obtained were examined.

Keywords: Biginelli reaction, acetoacetic acid *N*-arylamides, aromatic aldehydes, urea, antimicrobial activity

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Most of the methods for preparation of pyrimidine derivatives are based on the known Biginelli reaction discovered in 1893 year for the synthesis of pyrimidinones by reacting aromatic aldehyde, urea and ethyl acetoacetate [1–4]. A great interest of the synthetic chemists to the Biginelli condensation made it possible to extend the scope of substrates [5–7]. Use of ketoamides as β-dicarbonyl compounds allowed obtaining pyrimidine 5-carbamoyl derivatives possessing high antihypertensive [8], antitumor [9], tuberculocidal [10], and antimycobacterial [11–13] activity.

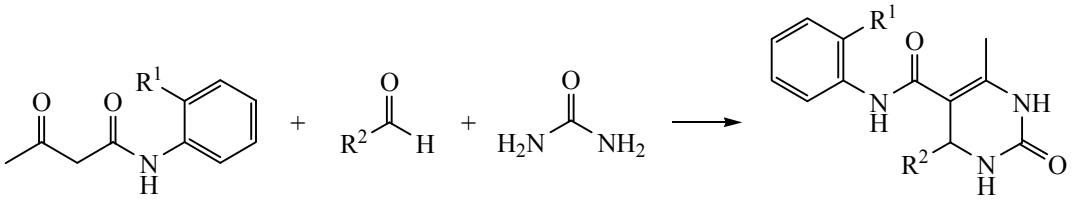
Here we report on the synthesis of previously unknown *N*,6-diaryl-4-methyl-2-oxo-1,2,3,6-tetrahydropyrimidine-5-carboxamides **1**–**11** and the investigation of their structure and antimicrobial activity. The target compounds were obtained by reacting acetoacetic acid *N*-arylamides with aromatic aldehydes and urea using the procedure described in [14].

The reactions proceeded in the solvent-free conditions at 120–150°C for 5–7 min (Scheme 1). Yields of the target compounds were 62–84%.

Compounds **1**–**11** were colorless crystalline substances soluble in DMF, DMSO, soluble at heating in acetic acid and ethanol, insoluble in water.

In the IR spectra of compounds **1**–**11** there were absorption bands due to stretching vibrations of CON (1676–1688 cm^{-1}) and C=O groups (1704–1720 cm^{-1}), as well as N–H (3168–3432 cm^{-1}) and C=C bonds (1600–1620 cm^{-1}).

Scheme 1.



R^1 = H (**1**–**3**), CH_3 (**4**–**7**), CH_3O (**8**, **9**, **10**), Cl (**11**); R^2 = 3-FC_6H_4 (**1**), 2-$NO_2C_6H_4$ (**2**), 4-$C_6H_5C_6H_4$ (**3**), 4-$NO_2C_6H_4$ (**4**), 2-ClC_6H_4 (**5**, **9**), 3-FC_6H_4 (**6**, **10**), 2,5-$(CH_3O)_2C_6H_3$ (**7**), 4-HO-3-$C_2H_5OC_6H_3$ (**8**), 4-$CH_3OC_6H_4$ (**11**).

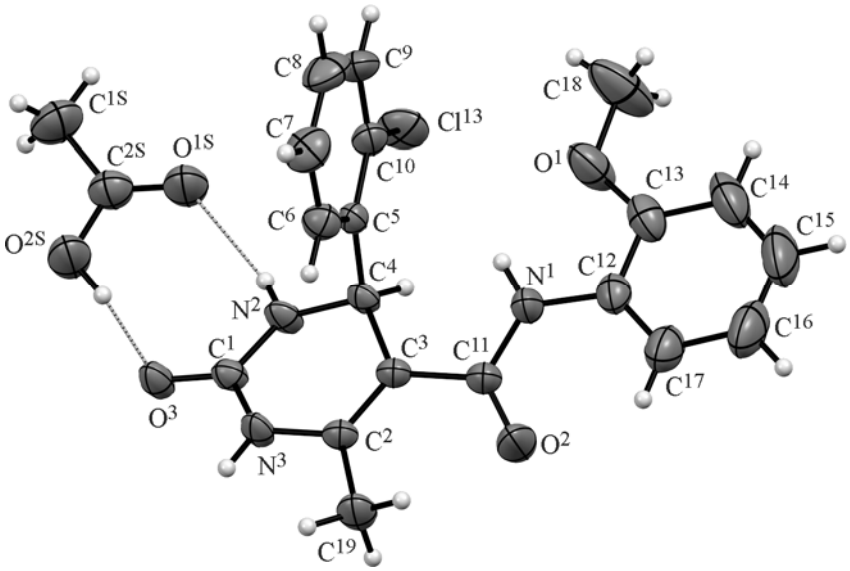


Fig. 1. General view of the molecule of **9** in the crystal.

The ^{1}H NMR spectra of carboxamides **1**–**11** contained the signals of 4-CH_3 (1.84–2.15 ppm), H^6 (5.05–5.87 ppm) and H^1 (6.77–7.80 ppm, $J_{1,6}$ = 1.8–1.9 Hz), amide NH (8.68–9.60 ppm) and 3-NH (7.98–8.79 ppm) protons, as well as the signals of aromatic protons and of substituents at the aromatic ring.

To determine the spatial structure of compound **9** in the crystal, single crystals were grown by a slow evaporation of acetic acid solution that were studied by X-ray diffraction method. Compound **9** crystallizes in a triclinic centrosymmetric space group *P*-1 as a 1 : 1 solvate with acetic acid (Fig. 1). Tetrahydropyrimidine ring has a distorted *boat* conformation. The fragment $N^3C^2C^3C^4$ is planar within 0.01 Å; the C^1 and N^2 atoms are out-of-plane by 0.274 and 0.395 Å, respectively. The chlorophenyl moiety is located in a pseudo-axial position. The fragment $C^3C^{11}O^2N^1C^{12}$ of arylcarbamoyl group, which is planar within 0.02 Å, is turned with respect to methoxyphenyl and dihydropyrimidine rings: the torsion angles $C^{11}N^1C^{12}C^{17}$ and $C^2C^3C^{11}O^2$ are equal to –15.9(4)° and 16.5(4)°. In the crystal molecules of **9** form centrosymmetric dimer associates due to intermolecular hydrogen bond N^3–$H^3\cdots O^3$ [$-x$ + 3, $-y$, $-z$ + 1]. Acetic acid molecule are also stabilized by intermolecular hydrogen bonding. The hydrogen bond parameters are listed in Table 1.

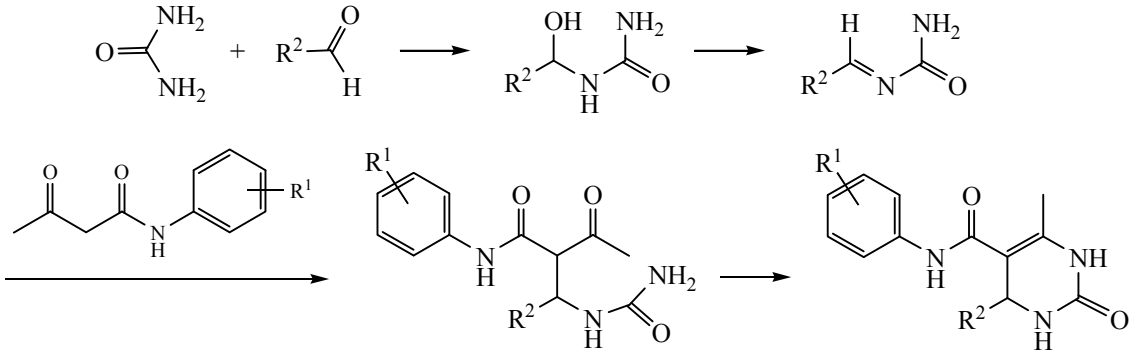
A plausible mechanism of the reaction is shown in Scheme 2. Probably, in the first step urea reacted with aromatic aldehyde to form aminocarbinol which further suffered dehydration. In the second step the reaction of acetoacetic acid *N*-arylamide with *N*-benzylideneurea afforded linear ureide, which underwent cyclization to form desired *N*,6-diaryl-4-methyl-2-oxo-1,2,3,6-tetrahydropyrimidine-5-carboxamide.

Antimicrobial activity of the compounds obtained against bacteria *St. aureus*, *E.coli*, *C. albicans* was determined by serial dilutions of test compound solution in meat-peptone broth and Sabouraud liquid medium. Bacterial load on 1 mL of culture liquid was 250 000 microbial cells. Control and experimental samples were incubated in a heating cabinet at 36–37°C for 18–20 h. Growth of bacterial cultures or inhibition due to the bacteriostatic action of the test compounds was registered. Minimum inhibitory concentrations (MIC, μg/mL), which inhibits the growth of bacterial

Table 1. Hydrogen bonds parameters in molecule of **9**

D–H···A	*d*(D–H), Å	*d*(H···A), Å	*d*(D···A), Å	DHA, deg
N^2–$H^2\cdots O^{1S}$	0.82(2)	2.19(2)	2.981(3)	164(2)
N^3–$H^3\cdots O^3$ [$-x$ + 3, $-y$, $-z$ + 1]	0.80(2)	2.08(2)	2.875(2)	176(2)
O^{2S}–$H^{2S}\cdots O^3$	0.89(4)	1.78(4)	2.657(3)	172(3)

Scheme 2.



cultures, are shown in Table 2. According to the data obtained, compounds **1**–**11** showed weak antimicrobial activity.

EXPERIMENTAL

IR spectra were recorded on a Specord M-80 spectrometer from mulls in mineral oil. ^{1}H NMR spectra were registered on a Bruker 500 spectrometer (500.13 MHz), internal reference TMS. Elemental analysis was performed on a Perkin Elmer 2400 instrument. Melting points were determined on a M-565 apparatus.

X-Ray diffraction study was performed on a Xcalibur Ruby diffractometer equipped with CCD detector by standard method (MoK_α-radiation, ω-scanning) [15]. The extinction was empirically accounted for using SCALE3 ABSPACK algorithm [15]. The structure was solved by the direct method and refined by full-matrix anisotropic approximation for all non-hydrogen atoms. Hydrogen atoms of NH and OH groups were localized from difference electron density synthesis and refined independently in isotropic approximation. The other hydrogen atoms were refined using a *rider* model in isotropic approximation with dependent thermal parameters. All calculations were made using SHELXL [16] and OLEX2 [17] programs.

The crystals of **9** were triclinic, $C_{19}H_{18}ClN_3O_3 \cdot C_2H_4O_2$, space group *P*-1; unit cell parameters at 295(2) K: *a* 7.4500(17)°, *b* 8.8713(16)°, *c* 17.040(3) Å, α 83.915(15), β 85.167(17), γ 72.842(19)°, *V* 1068.3(4) Å^3, d_{calc} 1.3432 g cm^{-3}, μ 0.216 mm^{-1}, *Z* 2. The final refinement parameters: R_1 0.0621, wR_2 0.1365 [for 3449 reflections with $I > 2\sigma(I)$], R_1 0.0948, wR_2 0.1592 (for all 4963 independent reflections), *S* 1.059. The results obtained were deposited in the Cambridge Crystallographicl Data Center (CCDC 1476230).

4-Methyl-*N*-phenyl-6-(3-fluorophenyl)-2-oxo-1,2,3,6-tetrahydropyrimidine-5-carboxamide (1). A mixture of 0.01 mol of acetoacetanilide, 0.01 mol of 3-fluorobenzaldehyde, and 0.01 mol of urea was heated at 120–150°C for 5–7 min until gas evolution completed and the reaction mixture solidified. After cooling, the solid was treated with ethanol; the precipitate was filtered off and recrystallized from ethanol. Yield 2.73 g (84%), mp 248–250°C (C_2H_5OH). IR spectrum, ν, cm^{-1}: 1610 (C=C), 1688 (CON), 1720 (CO), 3215, 3320, 3410 (NH). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 1.96 s (3H, 4-CH_3), 5.28 d (1H, CH, $J_{1,6}$ = 1.8 Hz), 7.20–7.58 m (9H, C_6H_5, FC_6H_4), 7.52 d (1H, 1-NH, $J_{1,6}$ = 1.8 Hz), 7.98 br.s (1H, 3-NH),

Table 2. Antimicrobial activity of compounds **1**–**11**

Compound	MIC, μm/mL		
	St. aureus	*E. coli*	*Candida albicans*
1	500	500	500
2	1000	1000	500
3	250	250	250
4	500	500	500
5	500	1000	1000
6	500	1000	1000
7	500	1000	1000
8	1000	1000	1000
9	1000	1000	1000
10	250	250	500
11	250	500	500
Dioxidine	62.5–1000	3.9–62.5	–
Chloramine B	500	250	–
Fluconazole	–	–	8–32

8.76 s (1H, NH, amide). Found, %: C 66.33, 66.57; H 4.88, 5.05; N 12.82, 13.05. $C_{18}H_{16}FN_3O_2$. Calculated, %: C 66.45; H 4.96; N 12.92.

Compounds **2**–**11** were prepared similarly.

4-Methyl-*N*-phenyl-6-(2-nitrophenyl)-2-oxo-1,2,3,6-tetrahydropyrimidine-5-carboxamide (2). Yield 2.85 g (81%), mp 234–236°C (C_2H_5OH). IR spectrum, ν, cm^{-1}: 1612 (C=C), 1680 (CON), 1716 (CO), 3200, 3360, 3420 (NH). ^1H NMR spectrum (DMSO-d_6), δ, ppm: 1.99 c (3H, 4-CH_3), 5.87 d (1H, CH, $J_{1,6}$ = 1.9 Hz), 7.15–7.42 m (9H, C_6H_5, $NO_2C_6H_4$), 7.49 d (1H, 1-NH, $J_{1,6}$ = 1.9 Hz), 8.79 br.s (1H, 3-NH), 8.92 s (1H, NH, amide). Found, %: C 61.25, 61.48; H 4.49, 4.66; N 15.78, 16.03. $C_{18}H_{16}N_4O_4$. Calculated, %: C 61.36; H 4.58; N 15.90.

4-Methyl-*N*-phenyl-6-(biphenyl-4-yl)-2-oxo-1,2,3,6-tetrahydropyrimidine-5-carboxamide (3). Yield 2.37 g (62%), mp 254–256°C (C_2H_5OH). ^1H NMR spectrum (DMSO-d_6), δ, ppm: 2.11 s (3H, 4-CH_3), 5.49 d (1H, CH, $J_{1,6}$ = 1.9 Hz), 7.05–7.78 m (14H, C_6H_5, $C_6H_5C_6H_4$), 7.80 d (1H, 1-NH, $J_{1,6}$ = 1.9 Hz), 8.76 br.s (1H, 3-NH), 9.60 s (1H, NH, amide). Found, %: C 75.07, 75.30; H 5.44, 5.61; N 10.85, 11.09. $C_{24}H_{21}N_3O_2$. Calculated, %: C 75.18; H 5.52; N 10.96.

4-Methyl-*N*-2-methylphenyl-6-(4-nitrophenyl)-2-oxo-1,2,3,6-tetrahydropyrimidine-5-carboxamide (4). Yield 2.38 g (65%), mp 233–235°C (C_2H_5OH). IR spectrum, ν, cm^{-1}: 1610 (C=C), 1688 (CON), 1720 (CO), 3200, 3336, 3416 (NH). ^1H NMR spectrum (DMSO-d_6), δ, ppm: 1.90 s (3H, 4-CH_3), 2.10 s (3H, $\underline{CH_3}C_6H_4$), 5.44 d (1H, CH, $J_{1,6}$ = 1.9 Hz), 6.86–8.20 m (8H, $CH_3C_6\underline{H}_4$, $NO_2C_6H_4$), 7.02 d (1H, 1-NH, $J_{1,6}$ = 1.9 Hz), 8.75 br.s (1H, 3-NH), 8.99 s (1H, NH, amide). Found, %: C 62.19, 62.41; H 4.87, 5.04; N 15.16, 15.41. $C_{19}H_{18}N_4O_4$. Calculated, %: C 62.29; H 4.95; N 15.29.

4-Methyl-*N*-2-methylphenyl-6-(2-chlorophenyl)-2-oxo-1,2,3,6-tetrahydropyrimidine-5-carboxamide (5). Yield 2.27 g (64%), mp 226–228°C (C_2H_5OH). ^1H NMR spectrum (DMSO-d_6), δ, ppm: 1.84 s (3H, 4-CH_3), 2.08 s (3H, $\underline{CH_3}C_6H_4$), 5.70 d (1H, CH, $J_{1,6}$ = 1.8 Hz), 6.98–7.29 m (8H, $CH_3C_6\underline{H}_4$, ClC_6H_4), 7.37 d (1H, 1-NH, $J_{1,6}$ = 1.8 Hz), 8.60 br.s (1H, 3-NH), 8.87 s (1H, NH, amide). Found, %: C 64.03, 64.27; H 5.02, 5.19; N 11.68, 11.93. $C_{19}H_{18}ClN_3O_2$. Calculated, %: C 64.14; H 5.10; N 11.81.

4-Methyl-*N*-2-methylphenyl-6-(3-fluorophenyl)-2-oxo-1,2,3,6-tetrahydropyrimidine-5-carboxamide (6). Yield 2.57 g (76%), mp 217–219°C (C_2H_5OH). IR spectrum, ν, cm^{-1}: 1620 (C=C), 1676 (CON), 1712 (CO), 3210, 3240, 3416 (NH). ^1H NMR spectrum (DMSO-d_6), δ, ppm: 1.90 s (3H, 4-CH_3), 2.10 s (3H, $\underline{CH_3}C_6H_4$), 5.35 d (1H, CH, $J_{1,6}$ = 1.9 Hz), 6.96–7.35 m (8H, $CH_3C_6\underline{H}_4$, FC_6H_4), 7.51 d (1H, 1-NH, $J_{1,6}$ = 1.9 Hz), 8.64 br.s (1H, 3-NH), 8.92 s (1H, NH, amide). Found, %: C 67.13, 67.36; H 5.26, 5.42; N 12.27, 12.51. $C_{19}H_{18}FN_3O_2$. Calculated, %: C 67.25; H 5.35; N 12.38.

4-Methyl-*N*-2-methylphenyl-6-(2,5-dimethoxyphenyl)-2-oxo-1,2,3,6-tetrahydropyrimidine-5-carboxamide (7). Yield 2.36 g (62%), mp 202–204°C (C_2H_5OH). ^1H NMR spectrum (DMSO-d_6), δ, ppm: 1.91 s (3H, 4-CH_3), 2.12 s (3H, $\underline{CH_3}C_6H_4$), 3.65 s and 3.68 s [6H, $(\underline{CH_3}O)_2C_6H_3$], 5.56 d (1H, CH, $J_{1,6}$ = 1.9 Hz), 6.66–7.08 m [7H, $CH_3C_6\underline{H}_4$, $(CH_3O)_2C_6\underline{H}_3$], 6.77 d (1H, 1-NH, $J_{1,6}$ = 1.9 Hz), 8.53 br.s (1H, 3-NH), 8.68 s (1H, NH, amide). Found, %: C 66.01, 66.24; H 6.00, 6.17; N 10.90, 11.15. $C_{21}H_{23}N_3O_4$. Calculated, %: C 66.13; H 6.08; N 11.02.

4-Methyl-*N*-2-methoxyphenyl-6-(4-hydroxy-3-ethoxyphenyl)-2-oxo-1,2,3,6-tetrahydropyrimidine-5-carboxamide (8). Yield 2.74 g (69%), mp 220–222°C (C_2H_5OH). IR spectrum, ν, cm^{-1}: 1600 (C=C), 1680 (CON), 1710 (CO), 3150 (OH), 3176, 3392, 3424 (NH). ^1H NMR spectrum (DMSO-d_6), δ, ppm: 1.28 t (3H, $\underline{CH_3}CH_2O$ J = 6.0 Hz), 2.14 s (3H, 4-CH_3), 3.61 s (3H, $\underline{CH_3}OC_6H_4$), 3.89 q (2H, $CH_3\underline{CH_2}O$, J = 6.0 Hz), 5.05 d (1H, CH, $J_{1,6}$ = 1.8 Hz), 6.70–7.84 m (7H, $CH_3OC_6\underline{H}_4$, $HO\text{-}C_2H_5OC_6\underline{H}_3$), 7.37 d (1H, 1-NH, $J_{1,6}$ = 1.8 Hz), 7.98 br.s (1H, 3-NH), 8.68 s (1H, NH, amide), 8.81 s (1H, OH). Found, %: C 63.36, 63.60; H 5.75, 5.93; N 10.46, 10.70. $C_{21}H_{23}N_3O_5$. Calculated, %: C 63.47; H 5.83; N 10.57.

4-Methyl-*N*-2-methoxyphenyl-6-(2-chlorophenyl)-2-oxo-1,2,3,6-tetrahydropyrimidine-5-carboxamide (9). Yield 2.60 g (70%), mp 236–238°C (C_2H_5OH). IR spectrum, ν, cm^{-1}: 1620 (C=C), 1688 (CON), 1720 (CO), 3208, 3416, 3432 (NH). ^1H NMR spectrum (DMSO-d_6), δ, ppm: 2.19 s (3H, 4-CH_3), 3.64 s (3H, $\underline{CH_3}OC_6H_4$), 5.60 d (1H, CH, $J_{1,6}$ = 1.8 Hz), 6.62–7.34 m (8H, $CH_3OC_6\underline{H}_4$, ClC_6H_4), 7.78 d (1H, 1-NH, $J_{1,6}$ = 1.8 Hz), 7.85 br.s (1H, 3-NH), 8.82 s (1H, NH, amide). Found, %: C 61.26, 61.50; H 4.81, 4.98; N 11.17, 11.42. $C_{19}H_{18}ClN_3O_3$. Calculated, %: C 61.38; H 4.88; N 11.30.

4-Methyl-*N*-2-methoxyphenyl-6-(3-fluorophenyl)-2-oxo-1,2,3,6-tetrahydropyrimidine-5-carboxamide (10). Yield 2.63 g (74%), mp 196–198°C (C_2H_5OH).

IR spectrum, ν, cm^{-1}: 1608 (C=C), 1680 (CON), 1712 (CO), 3168, 3232, 3416 (NH). ^1H NMR spectrum (DMSO-d_6), δ, ppm: 2.15 s (3H, 4-CH_3), 3.67 s (3H, $\underline{CH_3}OC_6H_4$), 5.28 d (1H, CH, $J_{1,6}$ = 1.9 Hz), 6.84–7.74 m (8H, $CH_3OC_6\underline{H_4}$, FC_6H_4), 7.70 d (1H, 1-NH, $J_{1,6}$ = 1.9 Hz), 8.32 br.s (1H, 3-NH), 8.76 s (1H, NH, amide). Found, %: C 64.11, 64.34; H 5.02, 5.18; N 11.70, 11.95. $C_{19}H_{18}FN_3O_3$. Calculated, %: C 64.22; H 5.11; N 11.82.

4-Methyl-*N*-2-chlorophenyl-6-(4-methoxyphenyl)-2-oxo-1,2,3,6-tetrahydropyrimidine-5-carboxamide (11). Yield 2.49 g (67%), mp 213–215°C (C_2H_5OH). IR spectrum, ν, cm^{-1}: 1600 (C=C), 1688 (CON), 1704 (CO), 3168, 3250, 3400 (NH). ^1H NMR spectrum (DMSO-d_6), δ, ppm: 2.13 s (3H, 4-CH_3), 3.67 s (3H, $\underline{CH_3}OC_6H_4$), 5.24 d (1H, CH, $J_{1,6}$ = 1.8 Hz), 6.77–7.40 m (8H, $CH_3OC_6\underline{H_4}$, ClC_6H_4), 7.47 d (1H, 1-NH, $J_{1,6}$ = 1.8 Hz), 8.67 br.s (1H, 3-NH), 8.77 s (1H, NH, amide). Found, %: C 61.27, 61.51; H 4.80, 4.97; N 11.19, 11.43. $C_{19}H_{18}ClN_3O_3$. Calculated, %: C 61.38; H 4.88; N 11.30.

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