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# Thermolysis of 1-(Methylideneamino)-1*H*-pyrrole-2,3-diones. Formation of Pyrazolodioxazines at [4+2]-Cycloaddition of Azomethinimines to Arylcarbaldehydes

**V. E. Zhulanov, M. V. Dmitriev, and A. N. Maslivets***

*Perm State National Reserch University, ul. Bukireva 15, Perm, 614990 Russia*
**e-mail: koh2@psu.ru*

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**Abstract**—4-Acyl-1-[(diphenylmethylidene)amino]-5-methoxycarbonyl-1*H*-pyrrole-2,3-diones generate at the thermolysis 4-acyl-3-(methoxycarbonyl)-1-(diphenylmethylidene)-1*H*-pyrazol-1-ium-5-olates that react with aromatic aldehydes to form methyl 2-aryl-8-acyl-4,4-diphenyl-4*H*-pyrazolo[5,1-*d*][1,3,5]dioxazine-7-carboxylates whose structure is proved by X-ray diffraction analysis.

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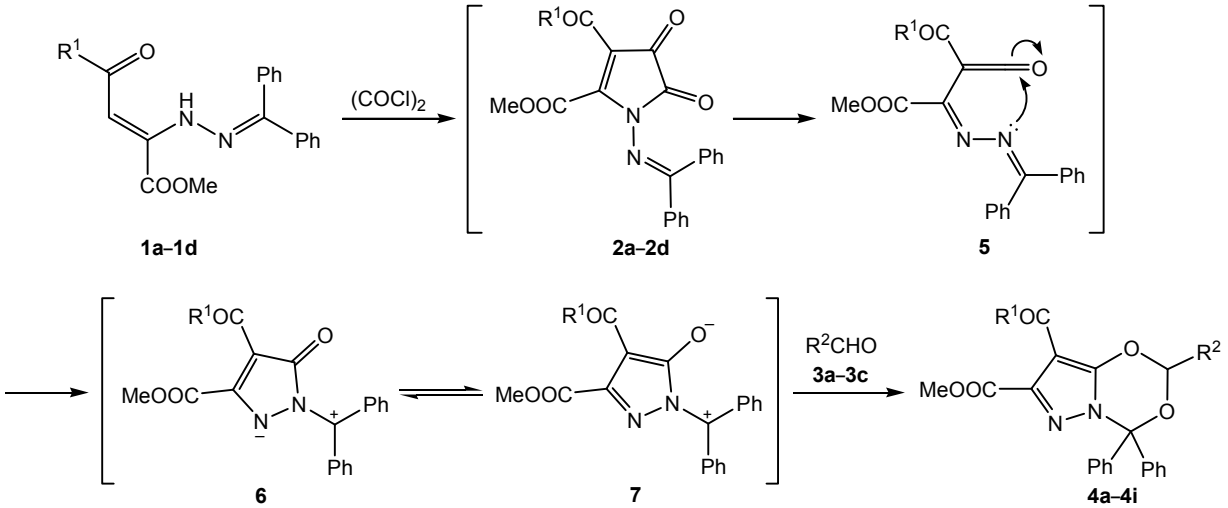
Structural fragments of oxazines and dioxazines are present in a number of synthetic [1–7] and natural [8–10] biologically active compounds exhibiting a tuberculocidal [1, 2], anticonvulsant [3], antimicrobial [4], and other kinds of activity.

We formerly developed a convenient method of synthesis of pyrazolo[5,1-*d*][1,3,5]dioxazines based on the reaction of dipolar [4+2]-cycloaddition of 1,4-dipoles generated *in situ* from 1-(diphenylmethylidene) amino-substituted 1*H*-pyrrole-2,3-diones with 4-bromobenzaldehyde [11]. In order to explore the limitations of the applicability of this reaction we used as dipolarophiles in this study aromatic aldehydes both with electron-donor and electron-acceptor substituents.

The reaction of enhydrazines **1a–1d** with oxalyl chloride in anhydrous chloroform at 60–65°C within 90–100 min leads to the formation of 4-acyl-1-[(diphenylmethylidene)amino]-5-methoxycarbonyl-1*H*-pyrrole-2,3-diones **2a–2d** used in further processes without isolation.




**1**, **2**, $R^1$ = Ph (**a**), $C_6H_4Me$-4 (**b**), $C_6H_4Cl$-4 (**c**), *t*-Bu (**d**); **3**, $R^2$ = $C_6H_4Br$-4 (**a**), $C_6H_3Cl_2$-2,4 (**b**), Ph (**c**); **4**, $R^2$ = $C_6H_4Br$-4, $R^1$ = Ph (**a**), $C_6H_4Me$-4 (**b**), $C_6H_4Cl$-4 (**c**); $R^2$ = $C_6H_4Cl_2$-2,4, $R^1$ = Ph (**d**), $C_6H_4Me$-4 (**e**), $C_6H_4Cl$-4 (**f**), *t*-Bu (**g**); $R^1$ = $R^2$ = Ph (**h**); $R^1$ = $C_6H_4Me$-4, $R^2$ = Ph (**i**).

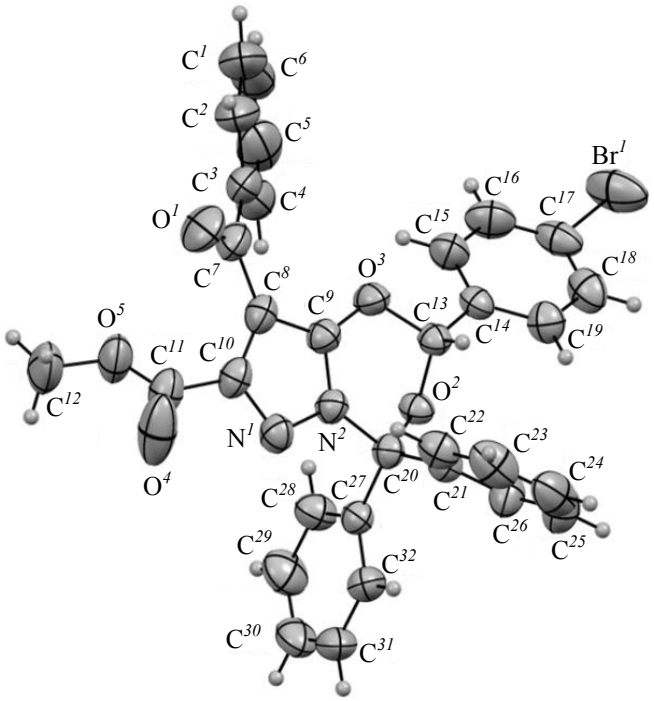



**Fig. 1.** Molecular structure of methyl 8-benzoyl-2-(4-bromophenyl)-4,4-diphenyl-4*H*-pyrazolo[5,1-*d*][1,3,5]-dioxazine-7-carboxylate **4a**. Nonhydrogen atoms are represented by thermal ellipsoids of 50% probability.

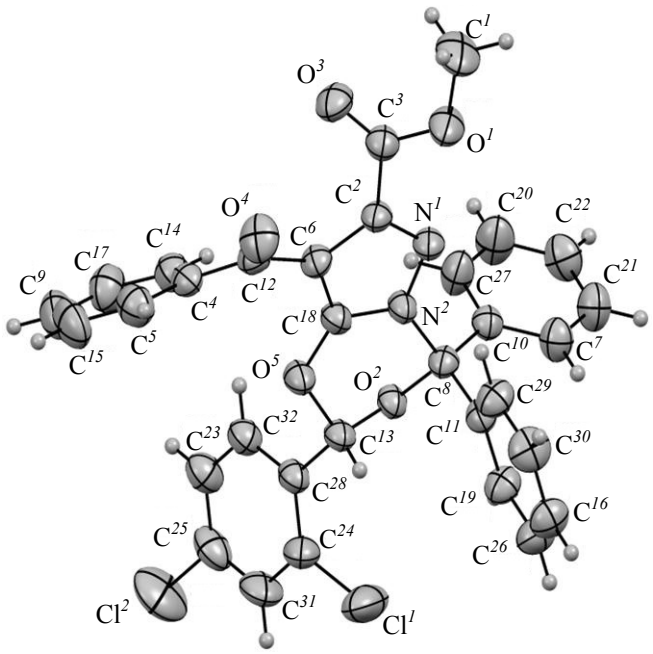



**Fig. 2.** Molecular structure of methyl 8-benzoyl-2-(2,4-dichlorophenyl)-4,4-diphenyl-4*H*-pyrazolo[5,1-*d*][1,3,5]-dioxazine-7-carboxylate **4d**. Nonhydrogen atoms are represented by thermal ellipsoids of 50% probability.

Boiling of a solution of 1*H*-pyrrole-2,3-diones **2a–2d**, obtained *in situ*, with aromatic aldehydes **3a–3c** in a 1 : 1 ratio in anhydrous *o*-xylene at 130–140°C within 30–40 min afforded in good yields methyl 2-aryl-8-acyl-4,4-diphenyl-4*H*-pyrazolo[5,1-*d*][1,3,5]-dioxazine-7-carboxylates **4a–4i** whose structure was proved by X-ray diffraction (XRD) analysis of compounds **4a** and **4d**.

Compounds **4a–4i** are colorless crystalline substances readily soluble in DMSO, chloroform, and acetone, insoluble in alkanes and water.

$^1$H NMR spectra of compounds **4a–4i** along with the signals of the protons of aromatic rings and the groups attached to them, of proton signals from a *tert*-butyl group (**4d** and **4g**) contain the singlets of the three protons of the methoxycarbonyl group (3.52–3.75 ppm) and of the methine proton (6.21–6.56 ppm).

According to XRD data compound **4a** (Fig. 1) crystallized in a centrosymmetric space group of the monoclinic crystal system. The pyrazole ring is flat within 0.01 Å. Dioxazine ring is present in the *semichair* conformation, $C^{13}$ and $O^2$ atoms deviate from the plain of the other atoms by 0.32 and –0.36 Å respectively. The angles between the plain of the pyrazole ring and the planar fragments $C^{21}C^{22}C^{23}C^{24}C^{25}C^{26}$ and $C^{27}C^{28}C^{29}C^{30}C^{31}C^{32}$ are 76.6 and 82.4 deg respectively. The multiple bonds in the pyrazole ring are essentially delocalized, the length difference of the formally ordinary bonds $C^9$–$N^2$ [1.350(3) Å], $C^8$–$C^{10}$ [1.413(4) Å] and formally double bonds $C^{10}$=$N^1$ [1.324(4) Å], $C^9$=$C^8$ [1.386(4) Å] does not exceed 0.03 Å. The molecules in the crystal are bound with non-classical intermolecular hydrogen bonds $C^{24}$–$H^{24}$···$O^4$ [$C^{24}$···$H^{24}$ 0.931, $H^{24}$···$O^4$ 2.340, $C^{24}$···$O^4$ 3.253(4) Å] and $C^6$–$H^6$···$O^1$ [$C^6$···$H^6$ 0.929, $H^6$···$O^1$ 2.504, $C^6$···$O^1$ 3.414(8) Å].

According to XRD data compound **4d** (Fig. 2) crystallized in a centrosymmetric space group of the triclinic crystal system. The molecular geometry of compound **4d** is close to the geometry of compound **4a**. The pyrazole ring is planar within 0.01 Å. The dioxazine ring is present in the conformation intermediate between *sofa* and *semichair*. The $O^2$ atom deviates from the plain of the other five atoms by 0.34 Å. In the crystal of compound **4d** several shortened contacts CH···O are present, but these interactions play considerably lesser role in the stabilization of the crystal packing than analogous interactions in the crystal of compound **4a**.

The formation of compounds **4a–4i** occurs apparently due to the thermal decarbonylation of pyrrolediones **2** leading to the generation of hydrazonoylketenes **5** undergoing an intramolecular cyclization with the formation of azomethinimines **6** capable in the absence of capturing agent to dimerize along [3+3] or [4+4] type [11]. In the presence of aromatic aldehydes azomethinimines **6** in the enolate-iminium form **7** enter in the reaction of dipolar [4+2]-cycloaddition with aromatic aldehydes. The use of arylcarbaldehydes with electron-donor substituents like anisic and veratric aldehydes resulted in a sharp decrease in the reaction regioselectivity: the formation in a low yield of pyrazolo[5,1-*d*][1,3,5]dioxazines **4** was confirmed by HPLC-MS method, but individual reaction products were not isolated.

The described reaction is an example of the synthesis of difficultly available substituted pyrazolo-[5,1-*d*][1,3,5]dioxazines with several functional substituents in the pyrazole and dioxazine rings.

## EXPERIMENTAL

IR spectra were recorded on a spectrophotometer Perkin Elmer Spectrum Two from mulls in mineral oil. $^1$H and $^{13}$C NMR spectra were registered on spectrometers Bruker DRX 400 or Bruker Avance III HD 400 [operating frequencies 400 ($^1$H) and 100 ($^{13}$C) MHz], internal reference HMDS. Elemental analysis was carried out on an analyzer vario Micro cube. For optimization of reaction conditions method HPLC-MS was used: column Acquity UPLC BEH C18 1.7 μm, mobile phases acetonitrile–water, flow rate 0.6 mL/min, detector ESI MS Xevo TQD. The homogeneity of compounds synthesized was confirmed by TLC on Silufol plates, eluents benzene–ethyl acetate, 5 : 1, ethyl acetate, development in iodine vapor.

**Methyl 8-benzoyl-2-(4-bromophenyl)-4,4-diphenyl-4*H*-pyrazolo[5,1-*d*][1,3,5]dioxazine-7-carboxylate (4a).** To a solution of 1.0 mmol of compound **1a** in 5 mL of anhydrous chloroform was added 1.0 mmol of oxalyl chloride, the mixture was boiled for 90 min, then 5 mL of anhydrous *o*-xylene was added, and chloroform was distilled off. The solution was stirred at 130–140°C (while boiling) for 10 min, then 1.0 mmol of *p*-bromobenzaldehyde was added, and the boiling was continued for 15 min more. The solution was cooled, evaporated in a vacuum, the formed precipitate was recrystallized from acetone. Yield 68%, mp 167–170°C (decomp.). IR spectrum, ν, cm$^{-1}$: 1734, 1638, 1603, 1553. $^1$H NMR spectrum ($CDCl_3$), δ, ppm: 3.57 s (3H, COOMe), 6.21 s (1H, C$^2$H), 7.31–7.57 m (17H$_{arom}$), 7.83–7.87 d.t (2H$_{arom}$, *J* 8.5, 1.6 Hz). $^{13}$C NMR spectrum ($CDCl_3$), δ, ppm: 52.3 (MeOCO), 95.6, 96.6, 104.3, 124.7, 127.3 (2C), 128.1 (2C), 128.4 (2C), 128.4 (2C), 128.6 (2C), 129.1 (2C), 129.3 (2C), 130.0, 130.2, 132.0 (2C), 132.8, 132.9, 138.6, 138.7, 139.0, 142.8, 150.6, 162.3 (MeOCO), 187.9 (ArCO). Found, %: C 64.61; H 3.78; N 4.67. $C_{32}H_{23}BrN_2O_5$. Calculated, %: C 64.55; H 3.89; N 4.70.

Compounds **4b–4i** were similarly prepared.

**Methyl 2-(4-bromophenyl)-8-(4-methylbenzoyl)-4,4-diphenyl-4*H*-pyrazolo[5,1-*d*][1,3,5]dioxazine-7-carboxylate (4b).** Yield 66%, mp 187–190°C (acetone, decomp.). IR spectrum, ν, cm$^{-1}$: 1734, 1638, 1603, 1553. $^1$H NMR spectrum ($CDCl_3$), δ, ppm: 2.41 s (3H, Me), 3.60 s (3H, MeOCO), 6.20 s (1H, C$^2$H), 7.23 d (2H$_{arom}$, *J* 7.9 Hz), 7.31–7.50 m (12H$_{arom}$), 7.52 d (2H$_{arom}$, *J* 8.4 Hz), 7.76 d (2H$_{arom}$, *J* 8.1 Hz). $^{13}$C NMR spectrum ($CDCl_3$), δ, ppm: 21.8 (Me), 52.2 (MeOCO), 95.6, 96.6, 104.5, 124.7, 127.4 (2C), 128.2 (2C), 128.3 (2C), 128.6 (2C), 129.1 (2C), 129.1 (2C), 129.5 (2C), 130.0, 130.2, 132.0 (2C), 133.0, 136.2, 138.8, 138.7, 139.2, 142.7, 143.8, 150.3, 162.3 (MeOCO), 187.6 (ArCO). Found, %: C 64.77; H 4.29; N 4.56. $C_{33}H_{25}BrN_2O_5$. Calculated, %: C 65.03; H 4.13; N 4.60.

**Methyl 2-(4-bromophenyl)-4,4-diphenyl-8-(4-chlorobenzoyl)-4*H*-pyrazolo[5,1-*d*][1,3,5]dioxazine-7-carboxylate (4c).** Yield 69%, mp 170–172°C (acetone, decomp.). IR spectrum, ν, cm$^{-1}$: 1733, 1641, 1588, 1552. $^1$H NMR spectrum ($CDCl_3$), δ, ppm: 3.63 s (3H, MeOCO), 6.21 s (1H, C$^2$H), 7.31–7.57 m (16H$_{arom}$), 7.79 d (2H$_{arom}$, *J* 8.4 Hz). $^{13}$C NMR spectrum ($CDCl_3$), δ, ppm: 52.3 (MeOCO), 95.8, 96.7, 104.1, 124.9, 127.3 (2C), 128.1 (2C), 128.4 (2C), 128.6 (2C), 128.8 (2C), 129.2 (2C), 130.1, 130.2, 130.7 (2C), 132.1 (2C), 132.7, 137.1, 138.6, 139.0, 139.4, 142.7, 150.6, 162.2 (MeOCO), 190.4 (ArCO). Found, %: C 60.81; H 3.53; N 4.43. $C_{32}H_{22}ClBrN_2O_5$. Calculated, %: C 61.02; H 3.52; N 4.45.

**Methyl 8-benzoyl-2-(2,4-dichlorophenyl)-4,4-diphenyl-4*H*-pyrazolo[5,1-*d*][1,3,5]dioxazine-7-carboxylate (4d).** Yield 60%, mp 172–174°C (acetone, decomp.). IR spectrum, ν, cm$^{-1}$: 1733, 1646, 1596, 1588. $^1$H NMR spectrum (DMSO-$d_6$), δ, ppm: 3.57 s (3H, MeOCO), 6.56 s (1H, C$^2$H), 7.33–7.66 m (14H$_{arom}$), 7.73 d (1H$_{arom}$, *J* 2.0 Hz), 7.88 d (2H$_{arom}$, *J*

7.3 Hz), 7.92 d (1$H_{arom}$, *J* 8.4 Hz). Found, %: C 65.55; H 3.75; N 4.75. $C_{32}H_{22}Cl_2N_2O_5$. Calculated, %: C 65.65; H 3.79; N 4.79.

**Methyl 2-(2,4-dichlorophenyl)-8-(4-methylbenzoyl)-4,4-diphenyl-4*H*-pyrazolo[5,1-*d*][1,3,5]dioxazine-7-carboxylate (4e).** Yield 63%, mp 203–206°C (acetone, decomp.). IR spectrum, ν, $cm^{-1}$: 1732, 1642, 1604, 1556. $^1H$ NMR spectrum (DMSO-$d_6$), δ, ppm: 2.37 s (3H, Me), 3.57 s (3H, MeOCO), 6.55 s (1H, $C^2H$), 7.31–7.46 m (8$H_{arom}$), 7.56–7.63 m (4$H_{arom}$), 7.71–7.75 m (2$H_{arom}$), 7.77 d (2$H_{arom}$, *J* 8.1 Hz), 7.91 d (1$H_{arom}$, *J* 8.5 Hz). Found, %: C 66.05; H 4.02; N 4.64. $C_{33}H_{24}Cl_2N_2O_5$. Calculated, %: C 66.12; H 4.04; N 4.67.

**Methyl 2-(2,4-dichlorophenyl)-4,4-diphenyl-8-(4-chlorobenzoyl)-4*H*-pyrazolo[5,1-*d*][1,3,5]dioxazine-7-carboxylate (4f).** Yield 60%, mp 175–177°C (acetone, decomp.). IR spectrum, ν, $cm^{-1}$: 1732, 1644, 1588, 1556. $^1H$ NMR spectrum (DMSO-$d_6$), δ, ppm: 3.60 s (3H, MeOCO), 6.58 s (1H, $C^2H$), 7.35–7.48 m (7$H_{arom}$), 7.55–7.62 m (5$H_{arom}$), 7.65 d.d (1$H_{arom}$, *J* 8.4, 1.9 Hz), 7.73 d (1$H_{arom}$, *J* 2.0 Hz), 7.90 d (2$H_{arom}$, *J* 8.6 Hz), 7.94 d (1$H_{arom}$, *J* 8.5 Hz). Found, %: C 61.94; H 3.39; N 4.50. $C_{32}H_{21}Cl_3N_2O_5$. Calculated, %: C 62.00; H 3.41; N 4.52.

**Methyl 8-(2,2-dimethylpropanoyl)-2-(2,4-dichlorophenyl)-4,4-diphenyl-4*H*-pyrazolo[5,1-*d*][1,3,5]dioxazine-7-carboxylate (4g).** Yield 62%, mp 156–158°C (acetone, decomp.). IR spectrum, ν, $cm^{-1}$: 1730, 1640, 1600, 1556. $^1H$ NMR spectrum (DMSO-$d_6$), δ, ppm: 1.16 s (9H, $Me_3C$), 3.75 s (3H, MeOCO), 6.57 s (1H, $C^2H$), 7.26–7.49 m (7$H_{arom}$), 7.54–7.62 m (3$H_{arom}$), 7.67 d.d (1$H_{arom}$, *J* 8.5, 1.9 Hz), 7.78 d (1$H_{arom}$, *J* 1.9 Hz), 7.96 d (1$H_{arom}$, *J* 8.5 Hz). $^{13}C$ NMR spectrum (DMSO-$d_6$), δ, ppm: 25.8 (3C, Me, $\underline{Me}_3C$), 43.9 ($Me_3\underline{C}$), 52.2 (<u>Me</u>OCO), 93.4, 96.0, 104.0, 127.0 (2C), 128.0 (2C), 128.3 (3C), 128.9 (2C), 129.7, 129.8, 130.0, 130.2 (2C), 133.1, 136.5, 137.7, 138.4, 141.6, 146.2, 162.0 (MeO<u>C</u>O), 201.9 ($Me_3C\underline{C}$O). Found, %: C 63.63; H 4.61; N 4.92. $C_{30}H_{26}Cl_2N_2O_5$. Calculated, %: C 63.72; H 4.63; N 4.95.

**Methyl 8-benzoyl-2,4,4-triphenyl-4*H*-pyrazolo-[5,1-*d*][1,3,5]dioxazine-7-carboxylate (4h).** Yield 63%, mp 160–162°C (acetone, decomp.). IR spectrum, ν, $cm^{-1}$: 1731, 1649, 1597, 1572. $^1H$ NMR spectrum ($CDCl_3$), δ, ppm: 3.59 s (3H, MeOCO), 6.27 s (1H, $C^2H$), 7.35–7.56 m (18$H_{arom}$), 7.85–7.89 m (2$H_{arom}$). $^{13}C$ NMR spectrum ($CDCl_3$), δ, ppm: 52.2 (<u>Me</u>OCO), 96.3, 96.5, 104.3, 126.4 (2C), 127.4 (2C), 128.3 (2C), 128.4 (2C), 128.6 (2C), 128.7 (2C), 129.1 (2C), 129.3 (2C), 129.9, 130.0, 130.4, 132.8, 133.9, 138.8, 138.9, 139.3, 142.9, 150.8, 162.4 (MeO<u>C</u>O), 187.9 (Ar<u>C</u>O). Found, %: C 74.36; H 4.55; N 5.40. $C_{32}H_{24}N_2O_5$. Calculated, %: C 74.41; H 4.68; N 5.42.

**Methyl 8-(4-methylbenzoyl)-2,4,4-triphenyl-4*H*-pyrazolo[5,1-d][1,3,5]dioxazine-7-carboxylate (4i).** Yield 38%, mp 173–176°C (acetone, decomp.). IR spectrum, ν, $cm^{-1}$: 1739, 1636, 1605, 1557. $^1H$ NMR spectrum ($CDCl_3$), δ, ppm: 2.41 s (3H, Me), 3.61 s (3H, MeOCO), 6.26 s (1H, $C^2H$), 7.24 d (2$H_{arom}$, *J* 7.9 Hz), 7.35–7.55 m (15$H_{arom}$), 7.78 d (2$H_{arom}$, *J* 8.1 Hz). $^{13}C$ NMR spectrum ($CDCl_3$), δ, ppm: 21.8 (Me), 52.2 (<u>Me</u>OCO), 96.3, 96.5, 104.5, 126.5 (2C), 127.4 (2C), 128.3 (2C), 128.6 (2C), 128.7 (2C), 129.0 (2C), 129.1 (2C), 129.5 (2C), 129.9, 130.0, 130.3, 133.9, 136.2, 138.9, 139.3, 142.7, 143.7, 150.6, 162.4 (MeO<u>C</u>O), 187.7 (Ar<u>C</u>O). Found, %: C 74.54; H 4.81; N 5.25. $C_{33}H_{26}N_2O_5$. Calculated, %: C 74.70; H 4.94; N 5.28.

**X-ray diffraction analysis** of compounds **4a** and **4d** was carried out on an automatic four-circle diffractometer Xcalibur R (Agilent Technologies) by a standard procedure [Mo$K_\alpha$-radiation, 295(2) K, ω-scanning, scan step 1°] applying program package CrysAlisPro [12]. The extinction was accounted for empirically using the algorithm SCALE3 ABSPACK [12]. The structures were solved by the direct method using the program SHELXS-97 and refined applying the program SHELXL-97 [13] with respect to $F^2$ in an anisotropic approximation for nonhydrogen atoms (hydrogen atoms were included in the refinement in the *rider* model in an isotropic approximation with dependent thermal parameters).

For analysis of compound **4a** was used a fragment of a colorless crystal of the size 0.4 × 0.3 × 0.25 mm. Crystal system monoclinic, *a* 11.941(3), *b* 18.538(3), *c* 12.5422(17) Å, β 91.318(17)°, space group $P2_1/c$, *Z* 4. The completeness of data collection for θ<26.00° 99.9%. Final refinement parameters are as follows: $R_1$ 0.0617, $wR_2$ 0.1737 [for 3861 reflections with $I>2\sigma(I)$], $R_1$ 0.1113, $wR_2$ 0.2028 (for all 6761 independent reflections, $R_{int}$ 0.0386), *GOOF* 1.112, Δρ 0.726/–1.186 ēÅ$^{-3}$.

For analysis of compound **4d** was used a colorless crystal of the size 0.1 × 0.1 × 0.07 mm. Crystal system triclinic, *a* 7.0550(11), *b* 10.5792(17), *c* 19.621(3) Å, α 86.223(13), β 88.169(12), γ 73.157(14)°, space group *P*–1, *Z* 2. The completeness of data collection

for $\theta < 26.00°$, 99.9%. Final refinement parameters are as follows: $R_1$ 0.0611, $wR_2$ 0.1229 [for 2879 reflections with $I > 2\sigma(I)$], $R_1$ 0.1618, $wR_2$ 0.1697 (for all 6604 independent reflections, $R_{int}$ 0.0570), *GOOF* 0.964, $\Delta\rho$ 0.317/–0.315 ē Å$^{-3}$.

The results of structural experiments are deposited to Cambridge Crystallographic Data Centre under the numbers CCDC 1457142 (**4a**) and CCDC 1531814 (**4d**). These data are available free at the address www.ccdc.cam.ac.uk.

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