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Synthesis of Derivatives of *o*-Aminoacetophenone and *o*-Aminobenzyl Alcohol

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Abstract—Oxidation of 2'-hydroxy-8-methylspiro[4*H*-benz-1,3-oxazin-2-one-6,1'-cyclopentane] or *N*-mesyl-2-(cyclopent-1-en-1-yl)-6-methylaniline provided the corresponding ketones. The rearrangement of these ketones oximes under treatment with thionyl chloride gave rise to nitriles of 5-(2-amino-3-methylphenyl)-5-oxopentanoic or 5-(2-methanesulfamido-3-methylphenyl)-5-hydroxypentanoic acids. By heating 5-(2-acetylamido-3-methylphenyl)-5-oxopentanoic acid with LiH in THF 3-(2,8-dimethylquinol-4-on-3-yl)propanoic acid was obtained.

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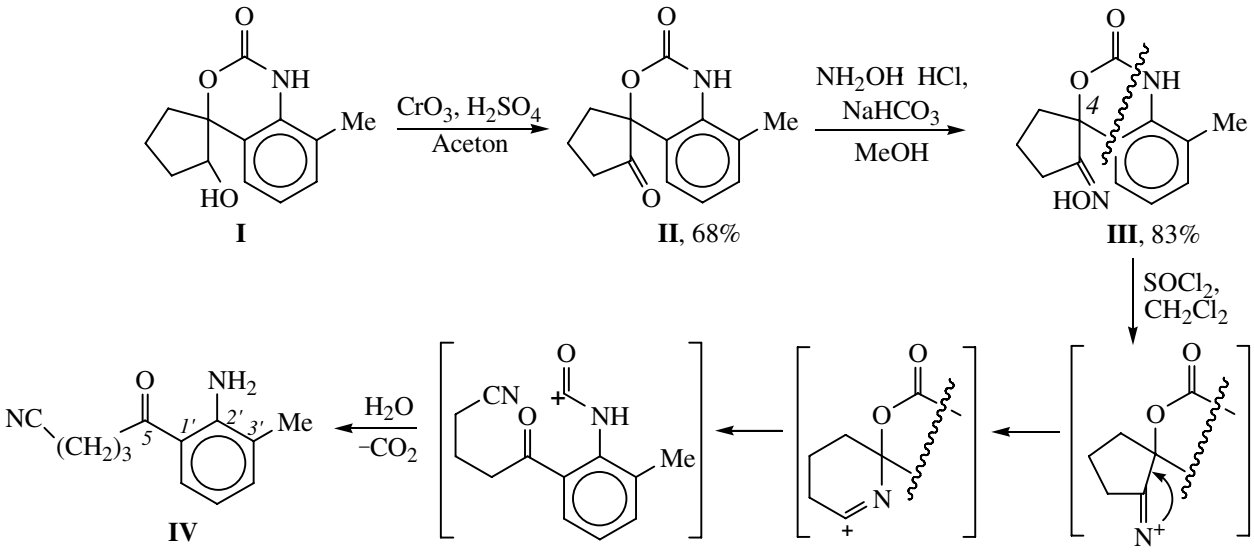
Derivatives of *o*-aminoacetophenone and *o*-amino-benzyl alcohol are used in preparation of substituted indoles [1, 2] and quinolones [3, 4]. We formerly synthesized some representatives of this series by oxidation of *o*-cycloalkenylanilides with hydrogen peroxide or ozone [5]. In extension of these studies we report here on preparation of such ketones and *o*-amino-benzyl alcohol derivatives from 2'-hydroxy-8-methylspiro[4*H*-benz-1-oxazin-2-on-6,1'-cyclopentane] and *N*-mesyl-2-(cyclopent-1-en-1-yl)-aniline. In oxidation of benzoxazinone **I** [6] with CrO_3 ketone **II** was obtained in 68% yield. The reaction of ketone **II** with hydroxylamine gave oxime **III** that treated with $SOCl_2$ was converted into ketonitrile **IV** in a fair yield, although a strong tarring of the reaction mixture occurred.

The ketonitrile structure of the product obtained was confirmed by spectral methods and elemental analysis. In the ^1H NMR spectrum of compound **IV** the methylene groups give rise to two two-proton triplets and one two-proton quintet. In the J-modulated spin-echo ^{13}C NMR

Scheme 1.



spectrum the carbon atoms appear as three signals at 16.6, 20.1, and 36.9 ppm that correspond to the methylene proton signals in the CH-correlation spectrum. The presence of the keto group was proved by the carbonyl carbon signal at 200 ppm.

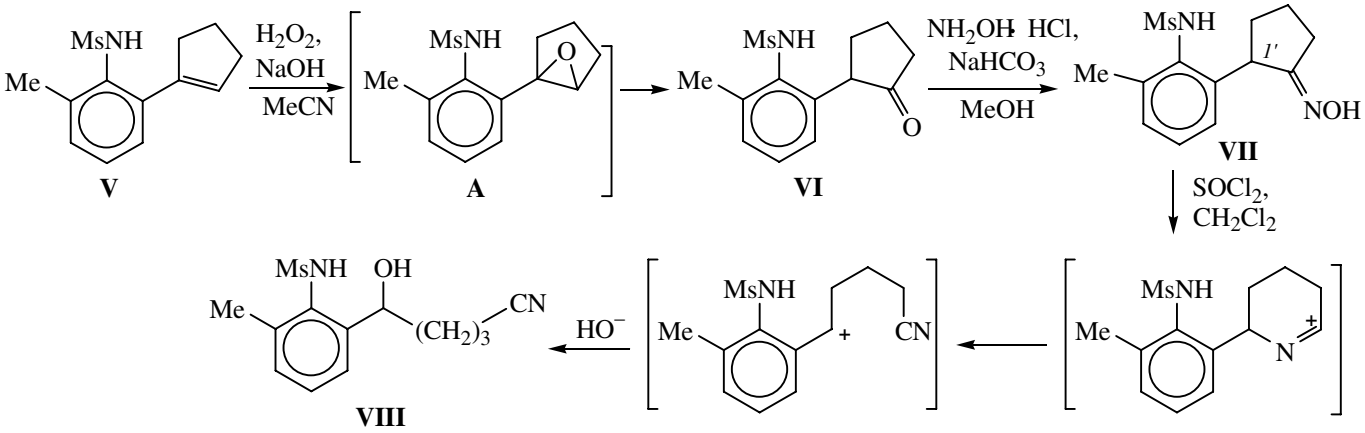
The oxidation of N-mesylate **V** with hydrogen peroxide gave ketone **VI** in 62% yield (Scheme 2). Evidently keto derivative **VI** formed as a result of a rearrangement of epoxide **A** not infrequent in similar systems [6]. The presence of an *ortho*-methyl group favored a single reaction route. We formerly [7] observed formation of four reaction products lacking ketone in oxidation under these conditions of both tosylate and mesylate analogs of compound **V** without the *ortho*-methyl group.

By reaction of ketone **VI** with hydroxylamine oxime **VII** was obtained in 86% yield. By the treatment with $SOCl_2$ oxime **VII** was converted into hydroxynitrile **VIII** in 45% yield. The structure of nitriles **IV** and **VIII** obtained suggests that oximes **III** and **VII** apparently have an *anti*-location of OH group and atoms $C^{1'}$ and C^4 of compounds **III** and **VII** respectively.

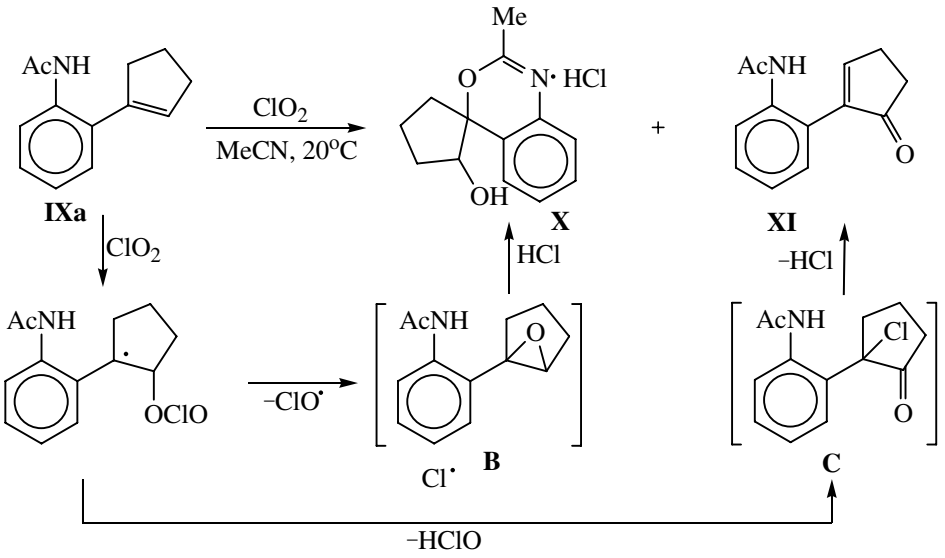
From N-acetylated derivatives of the aminobenzyl alcohol of **VIII** type 2,3-disubstituted indoles may be synthesized [1, 2]. We previously established that it was impossible to obtain from the N-acylated analogs of *N*-mesylate **V** cyclopentanones similar to ketone **VI** by oxidation with the hydrogen peroxide in the system Na_2WO_4–H_3PO_4 or in the presence of alkali, or with dimethyldioxirane. In all instances the reaction led to the formation of benzoxazines or indolines [7, 8]. Aiming at preparation of a ketone type compound with an N-acetyl group we attempted an oxidation of anilide **IXa** with ClO_2. The reaction provided a mixture of benzoxazine **X** and α,β-unsaturated ketone **XI** (Scheme 3). The reaction mixture suffered a considerable tarring. Benzoxazine **X** is apparently a product of the subsequent cyclization of epoxy compound **B**, and ketone **XI** may arise by eliminating HCl from of a chloroderivative **C**. The reaction of ClO_2 with olefins is known to provide both epoxides and ketone type compounds with a halogen attached to the adjacent carbon atom [9]. The composition and structure of compounds **X** and **XI** obtained were con-firmed by spectral methods and elemental analysis. The NMR spectra of benzoxazine hydrochloride are in agree-ment with the spectra of the free base [7]. The ketone structure is proved by the presence in the ^{13}C NMR spec-trum of a peak in the region 210 ppm. The signals of atoms $C^{1'}$ and $C^{2'}$ (145 and 166 ppm) are shifted down-field. All the three signals are close to those in the spec-trum of analogous ketone we have described before [10].

Oxidation of anilides **IXa** and **IXb** [5] with the hydrogen peroxide in formic acid takes the third route giving ketoacids (Scheme 4). Ketoacids **XIIa** and **XIIb** [5] are apparently products of epoxides **D** transformations [6, 11, 12]. The heating of ketocarboxylic acid **XIIb** with lithium hydride in THF gives 4-quinolone-3-carboxylic acid **XIII** in 50% yield; this acid is characterized by a considerably higher melting point (283°C) than the initial ketoacid (145°C). The quinolone structure was proved by spectral methods. In the ^1H NMR spectrum of compound **XIII** only two triplets of methylene groups protons are observed whereas the spectrum of initial compound **XIIb** contains signals of three methylene groups. The other signals in the NMR spectra of quinolone **XIII** are consistent with the suggested structure. In

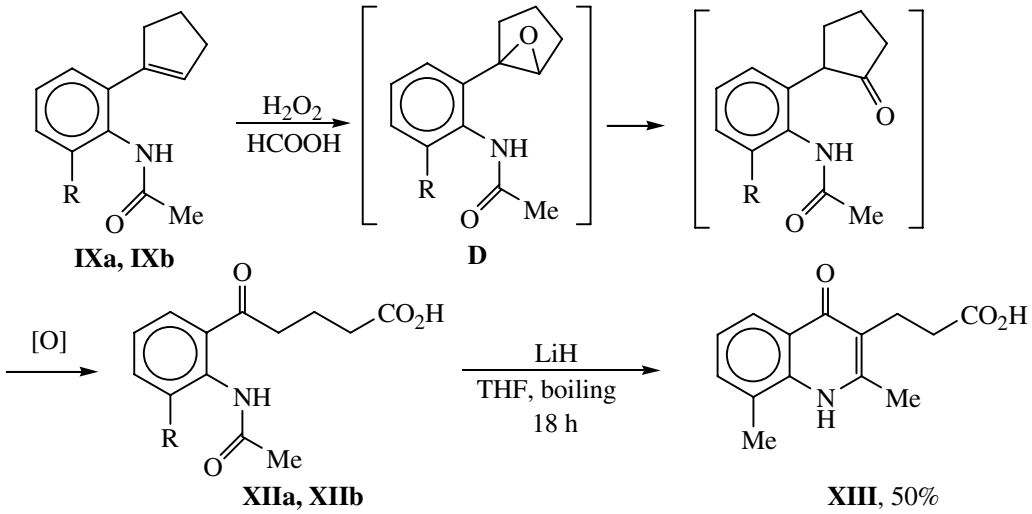
Scheme 2.



Scheme 3.



Scheme 4.



R = H (**a**), Me (**b**).

the J-modulated spin-echo ^{13}C NMR spectrum appear three signals from atoms C^5, C^6, and C^7, and also 7 singlets corresponding to the quaternary carbons of the aromatic ring, carbonyl, and carboxy groups. In the mass spectrum molecular ion peak was observed with *m/z* 245.

Thus the oxidation of *N*-acyl-2-(1-cyclopenten-1-yl)-anilines led to the formation of 5-(2-amino-3-methylphenyl)-5-oxopentanoic or 5-(2-methanesulfamido-3-methylphenyl)-5-hydroxypentanoic acids, and the heating of 5-(2-acetylamido-3-methylphenyl)-5-oxopentanoic acid with LiH in THF gave 3-(2,8-dimethylquinol-4-on-3-yl)propanoic acid.

EXPERIMENTAL

IR spectra were recorded on a spectrophotometer UR-20. ^1H and ^{13}C NMR spectra were registered on a spectrometer Bruker AM-300 at operating frequencies 300.13 and 75.45 MHz respectively, internal reference TMS. Elemental analysis was carried out on a CHN Analyzer M-185B. For column chromatography was used

silica gel 40/70 μm (Lancaster). Qualitative TLC analysis was performed of Silufol plates Sorbfil PTSKh-AF-V-UV (Join-Stock Co Sorbpolymer, Stavropol), spots were visualized by UV irradiation (λ 254 nm) or by iodine vapor. Mass spectra were taken on a spectrometer MKh 1320 (70 eV). Melting points were measured on a Boëtius heating block.

2'-Oxo-8-methylspiro[4*H*-benz-1-oxazin-2-one-6,1'-cyclopentane] (**II**). To a solution of 2 g (8.58 mmol) of compound **I** in 60 ml of acetone was added 20 ml of Jones' reagent prepared by dissolving 14 g of CrO_3 in 100 ml of water with subsequent addition of 12 ml of concn. H_2SO_4. The mixture was left standing for 15 h. Then $NaHSO_3$ was added till disappearance of the brown color of the upper layer, water-salt layer was separated, acetone was partially evaporated, the residue was diluted with 80 ml of $CHCl_3$, washed with a saturated solution of sodium carbonate till neutral reaction, then with water, dried with $MgSO_4$, and the solvent was evaporated under a reduced pressure. The residue was subjected to column chromatography on silica gel (eluent hexane–EtOAc, 4:1). Yield 1.34 g (68%), mp 209°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 2.02–2.15 m (CH_2), 2.23 s (CH_3), 2.34 q (CH_2, *J* 7.0 Hz), 2.45–2.57 m (CH_2), 6.94 t (H^6, *J* 7.7 Hz), 7.07 d (H^5, *J* 7.4 Hz), 7.14 d (H^7, *J* 7.3 Hz), 9.72 s (NH). ^{13}C NMR spectrum (DMSO-d_6), δ, ppm: 16.9 (CH_3), 17.4, 34.2, 34.7 ($3CH_2$), 85.2 (C^4), 121.8, 122.4, 131.1 (C^5, C^6, C^7), 119.4, 123.2 (C^{4a}, C^8), 134.3 (C^{8a}), 149.9 (C^2), 211.4 (C=O). Found, %: C 67.45; H 5.66; N 6.01. $C_{13}H_{13}NO_3$. Calculated, %: C 67.52; H 5.67; N 6.06.

2'-Oxo-8-methylspiro[4*H*-benz-1-oxazin-2-one-6,1'-cyclopentane] oxime (**III**). To a solution of 1.73 g (7.49 mmol) of ketone **II** in 40 ml of methanol was added 0.63 g (9 mmol) of $NH_2OH{\cdot}HCl$, and then dropwise was added at stirring a solution of 0.91 g (10.8 mmol) of $NaHCO_3$ in 20 ml of water. The mixture was stirred for 5 h, then diluted with 120 ml of $CHCl_3$, washed with water (30 ml), and dried with $MgSO_4$. On complete evaporation of the solvent the reaction product crystallized. Yield 1.53 g (83%), mp 265–267°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 1.85–2.04 m (CH_2), 2.24 s (CH_3), 2.37–2.65 m ($2CH_2$), 6.91 t (H^6, *J* 7.3 Hz), 7.04 d (H^5, *J* 7.5 Hz), 7.10 d (H^7, *J* 7.1 Hz), 9.67 s (NH), 11.11 s (OH). ^{13}C NMR spectrum (DMSO-d_6), δ, ppm: 17.0 (CH_3), 19.9, 25.4, 37.5 ($3CH_2$), 87.5 (C^4), 121.9, 122.0, 130.4 (C^5, C^6, C^7), 121.7, 122.8 (C^{4a}, C^8), 134.5 (C^{8a}), 150.3 (C^2), 161.8 (C=NOH). Found, %: C 63.47; H 5.76; N 11.29. $C_{13}H_{14}N_2O_3$. Calculated, %: C 63.40; H 5.73; N 11.38.

5-(2-Amino-3-methylphenyl)-5-oxopentanoic acid nitrile (IV). In 5 ml of dichloromethane was dispersed 0.44 g (1.77 mmol) of oxime **III**, and maintaining the temperature of the mixture at 0°C was added dropwise while stirring 0.6 g (5.04 mmol) of $SOCl_2$ in 4 ml of CH_2Cl_2. The stirring at this temperature was continued for 2 h, the a saturated solution of $NaHCO_3$ was added till neutral reaction, the mixture was diluted with 80 ml of CH_2Cl_2, washed with water (30 ml), and dried with $MgSO_4$. On evaporating the solvent in a vacuum we obtained a brown substance subjected to chromatography on silica gel (eluent hexane–EtOAc). We isolated compound **IV** in 0.13 g (35%) yield, R_f 0.4 (hexane–EtOAc, 4:1). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 2.05 q (CH_2, *J* 6.9 Hz), 2.14 s (CH_3), 2.46 t (CH_2, *J* 7.2 Hz), 3.10 t (CH_2, *J* 7.1 Hz), 6.42 br.s (NH_2), 6.58 t (H^5, *J* 7.5 Hz), 7.19 d (H^4, *J* 7.2 Hz), 7.61 d (H^6, *J* 8.2 Hz). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 17.1 (CH_3), 16.6, 20.2, 36.9 ($3CH_2$), 115.1, 128.8, 135.1 (C$^{4'}$, C$^{5'}$, C$^{6'}$), 116.7, 119.6, 123.5 (Ca"N, C^2, C$^{3'}$), 148.9 (C$^{1'}$), 200.3 (C=O). Found, %: C 71.20; H 6.92; N 13.87. $C_{12}H_{14}N_2O$. Calculated, %: C 71.26; H 6.98; N 13.85.

***N*-Methanesulfonyl-2-methyl-6-(2-oxocyclopent-1-yl)aniline** (**VI**). To a solution of 1.25 g (5 mmol) of mesylate **V** in 6 ml of MeCN and 3 ml of MeOH was added 0.51 g (7.5 mmol) of 50% H_2O_2, then 10% solution of KOH till pH 8. The mixture was kept for 5 h, then diluted with 60 ml of $CHCl_3$, washed with 20 ml of 10% $Na_2S_2O_3$ solution, 20 ml of water, and dried with $MgSO_4$. On evaporating the solvent the reaction product was subjected to chromatography on silica gel. Yield 0.83 g (62%), R_f 0.3 (hexane–EtOAc, 3:1). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 2.42 s (CH_3), 2.00–2.52 m ($3CH_2$), 3.01 s (CH_3), 4.08–4.15 m (CH), 7.02 d.d (1H, ArH, J_1 3.2, J_2 6.0 Hz), 7.12–7.20 m (2H, ArH), 7.45 c (NH). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 19.3 (CH_3), 20.8, 29.7, 38.4 ($3CH_2$), 41.8 (CH_3SO_2), 51.7 (CH), 124.8 (C^4), 127.7 (C^3), 129.9 (C^5), 134.1 (C^2), 137.1 (C^1), 137.8 (C^6), 219.8 (C=O). Mass spectrum: *m/z* 267 $[M]^+$. Found, %: C 57.98; H 6.52; N 5.33; S 11.50. $C_{13}H_{17}NO_3S$. Calculated, %: C 58.41; H 6.41; N 5.24; S 11.99.

***N*-Methanesulfonyl-2-methyl-6-(2-oxocyclopent-1-yl)aniline oxime (VII)** was obtained similarly to compound **III** from 2.0 g (7.49 mmol) of ketone **VI** and 0.63 g (9 mmol) of hydroxylamine hydrochloride. On evaporating the solvent the oily residue was recrystallized from CCl_4. Yield 1.81 g (86%), R_f 0.2 (hexane–EtOAc, 3:1). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.73–1.84 m (CH_2), 2.05 s (CH_3), 2.32–2.82 m ($2CH_2$), 2.98 s (CH_3SO_2), 4.07–4.13 m (CH), 7.02 d (1H, ArH, *J* 7.4 Hz), 7.04 d (1H, ArH, *J* 5.9 Hz), 7.17 d (H^4, *J* 7.6 Hz), 8.08 s

(NH), 8.61 br.s (OH). ^{13}C NMR spec-trum ($CDCl_3$), δ, ppm: 19.3, 42.3 ($2CH_3$), 22.9, 28.2, 35.2 ($3CH_2$), 44.7 (CH), 126.2 (C^4), 127.7 (C^5), 129.3 (C^3), 133.0 (C^2), 137.5 (C^6), 141.1 (C^1), 170.3 (C=NOH). Found, %: C 54.98; H 6.51; N 9.85; S 11.39. $C_{13}H_{18}N_2O_3S$. Calculated, %: C 55.30; H 6.43; N 9.92; S 11.35.

5-(2-Methanesulfamido-3-methylphenyl)-5-γθdpOqCθpentanoic acid nitrile (VIII) was prepared similarly to compound **IV** from 1.77 mmol of oxime **VII**. After chromatography on silica gel (hexane–EtOAc) yield was 0.22 g (45%), R_f 0.3 (hexane–EtOAc, 3:1). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.73–2.27 m ($3CH_2$), 2.00–2.23 br.s (OH), 2.40 s (CH_3), 3.05 s (CH_3SO_2), 5.57 d.d (1H, CHOH, J_1 4.7, J_2 8.9 Hz), 6.86 s (NH), 7.23 d (1H, ArH, J 6.4 Hz), 7.30 t (1H, H^4, J 7.8 Hz), 7.47 d (1H, ArH, J 7.4 Hz). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 18.9, 41.0 ($2CH_3$), 16.4, 22.9, 37.6 ($3CH_2$), 57.5 (CHOH), 119.5 (C≡N), 125.9 (C^4), 128.7 (C^5), 130.6 (C^2), 131.1 (C^3), 136.7 (C^6), 141.1 (C^1). Found, %: C 55.38; H 6.40; N 9.88; S 11.42. $C_{13}H_{18}N_2O_3S$. Calculated, %: C 55.30; H 6.43; N 9.92; S 11.35.

Anilide IXa oxidation with chlorine dioxide. To a solution of 0.1 g (0.5 mmol) of anilide **IX** in 10 ml of MeCN was added 0.5 mmol of ClO_2 in 20 ml of MeCN at 20°C. The reaction progress was monitored by TLC, then the solvent was evaporated. The dark residue was subjected to column chromatography on silica gel.

2'-Hydroxy-2-methylspiro[(4*H*-benz-1,3-oxazine)-6,1'-cyclopentane] hydrochloride (X). Yield 0.041 g (32%), R_f 0.3 (MεOH–EtOAc, 1:3). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 1.62–2.28 m ($3CH_2$), 2.52 s (CH_3), 4.21 t ($H^{2'}$, J 5.0 Hz), 4.31 br.s (OH), 7.36–7.47 m (4H, ArH). ^{13}C NMR spectrum (DMSO-d_6), δ, ppm: 19.8 (CH_3), 19.3, 31.3, 35.8 ($3CH_2$), 77.1 (CHOH), 97.2 (C^4), 117.1, 127.0, 128.3, 129.5 (C^5, C^6, C^7, C^8), 121.6, 128.8 (C^{8a}, C^{4a}), 170.1 (C^2). Found, %: C 61.60; H 6.30; Cl 14.02; N 5.49. $C_{13}H_{16}ClNO_2$. Calculated, %: C 61.54; H 6.36; Cl 13.97; N 5.52.

***N*-Acetyl-2-(5-oxocyclopent-1-en-1-yl)-aniline (XI)**. Yield 0.036 g (34%), R_f 0.3 (EtOAc). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 2.09 s (CH_3), 2.63–2.69 m (CH_2), 2.81–2.85 m (CH_2), 7.15 t (H^4, J 7.5 Hz), 7.23 d (H^6, J 6.3 Hz), 7.36 t (H^5, J 6.9 Hz), 7.77–7.82 m (2H, H^3, $H^{2'}$), 9.04 s (NH). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 24.2 (CH_3), 27.7, 35.7 ($2CH_2$), 125.2, 125.3, 129.4, 129.8 (C^3, C^4, C^5, C^6), 125.4, 135.1 (C^1, C^2), 145.5 ($C^{1'}$), 166.0 ($C^{2'}$), 168.9 (C=O), 210.3 ($C^{5'}$=O). Found, %: C 72.48; H 6.11; N 6.43. $C_{13}H_{13}NO_2$. Calculated, %: C 72.54; H 6.09; N 6.51.

5-(2-Methylcarboxyamidophenyl)-5-oxopentanoic acid (XIIa). To a solution of 0.47 g (2.33 mmol) of anilide **IXa** in 0.6 ml of 90% formic acid was added 0.6 g (8.82 mmol) of 50% hydrogen peroxide maintaining the reaction temperature below 40°C. The reaction mixture was left standing for 24 h at room temperature, then diluted with 60 ml of ethyl acetate, the solution was washed with water (2×20 ml), and dried with $MgSO_4$. On evaporating the solvent the reaction product was recrystallized from CH_2Cl_2. Yield 0.33 g (57%), mp 123–125°C (CH_2Cl_2). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 2.09 q (CH_2, J 7.1 Hz), 2.25 s (CH_3), 2.50 t (CH_2, J 7.1 Hz), 3.15 t (CH_2, J 7.1 Hz), 7.10 d.t ($H^{4'}$, J_1 1.0, J_2 8.0 Hz), 7.55 d.t ($H^{5'}$, J_1 1.0, J_2 8.4 Hz), 7.91 d ($H^{3'}$, J 8.0 Hz), 8.75 d ($H^{6'}$, J 8.4 Hz), 11.70 (NH), 12.05 (COOH). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 19.1 (C^3), 25.5 (CH_3), 32.9 (C^2), 38.8 (C^4), 120.9 ($C^{3'}$), 121.4 ($C^{1'}$), 122.4 ($C^{5'}$), 130.6 ($C^{6'}$), 135.1 ($C^{4'}$), 140.8 ($C^{2'}$), 169.8 (CONH), 178.5 (COOH), 203.8 (C^5). Found, %: C 62.33; H 6.12; N 5.39. $C_{13}H_{15}NO_4$. Calculated, %: C 62.64; H 6.07 N 5.62.

3-(2,8-Dimethylquinol-4-on-3-yl)propanoic acid (XIII). In 30 ml of THF was dissolved at boiling 65 mg (0.25 mmol) of ketoacid **XIIb**. To the solution obtained was added at heating 30 mg of LiH, colorless precipitate separated. The reaction mixture was heated at reflux for 18 h, then cooled to room temperature, 1 ml of water was added, then 10 ml of 5% solution of HCl. The separated precipitate was filtered off. Yield 31 mg (50%), mp 283°C. ^{1}H NMR spectrum (DMTA-d_7), δ, ppm: 2.50 t (CH_2, J 7.5 Hz), 2.60 s (CH_3), 2.90 t (CH_2, J 7.5 Hz), 7.20 d.t (H^6, J 7.0 Hz), 7.50 d, 8.10 d (H^5, H^7, J 7.0 Hz). ^{13}C NMR spectrum (DMF-d_7), δ, ppm: 17.8, 18.0 ($2CH_3$), 22.2 (CH_2), 34.2 (CH_2), 119.1 (C^3), 122.8, 123.7, 132.6 (C^5, C^6, C^7), 124.6, 126.9, 139.1, 148.4 (C^2, C^{4a}, C^8, C^{8a}), 176.4 (CO_2H), 176.9 (C^4). Mass spectrum, *m/z*: 245 $[M]^+$, 227 $[M - H_2O]^+$, 200 $[M - CO_2H]^{2+}$, 185 (max) $[M - H_2O - CH_2CO]^{2+}$ and/or $[M - CO_2H - CH_3]^+$.

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