Synthesis and Spectral Properties of 5-Methylthio-4*H*-1-(*p*-substituted-phenyl)-3a-(*p*-substituted-phenyl)-9-[(*m*-; and *p*-substituted)-phenoxy]-3a,4-dihydro[1,2,4]oxadiazolo[4,5-*a*][1,5]benzodiazepines

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The preparation of eight novel substituted [1,2,4]oxadiazolo[4,5-*a*][1,5]benzodiazepines which have potentially useful pharmacological properties; by 1,3-cycloaddition of benzonitrile oxides, generated *in situ* from benzohydroxamoyl chloride and triethylamine, to 1,5-benzodiazepine derivatives is described. The structure of all products was corroborated by ir, ^{1}H-nmr, ^{13}C-nmr and ms.

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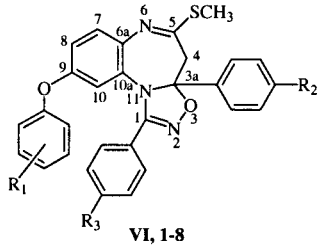
The synthesis of benzodiazepinic derivatives with heterocyclic rings annelated to the "a" [3-6], "c" [7] or "d" [8] sides of the heptatomic system has recently attracted interest of several research.

The fusion of a heterocyclic system to the benzodiazepine ring appears, in fact, especially promising for the synthesis of derivatives with greater activity and specificity, provided that they show similar pharmacological profiles to the benzodiazepines from which they are derived. Recently research has been carried out on substituted [1,2,4]oxadiazolo-[4,5-*a*][1,5]benzodiazepine derivatives [9-10] to confirm its pharmacological activity as an anticonvulsant drug [11]. As a part of a program directed towards the synthesis and spectral property determination of 1,5-benzodiazepine derivatives with possible pharmacological activity, we describe in this report the synthesis of the novel compounds 5-methylthio-4*H*-1-(*p*-substituted-phenyl)-3a-(*p*-substituted-phenyl)-9-[(*m*- and *p*-substituted)-phenoxy]-3a-4-dihydro[1,2,4]oxadiazolo[4,5-*a*][1,5]benzodiazepines, **VI, 1-8** (Scheme 1) as shown in Scheme 2.

Scheme 1



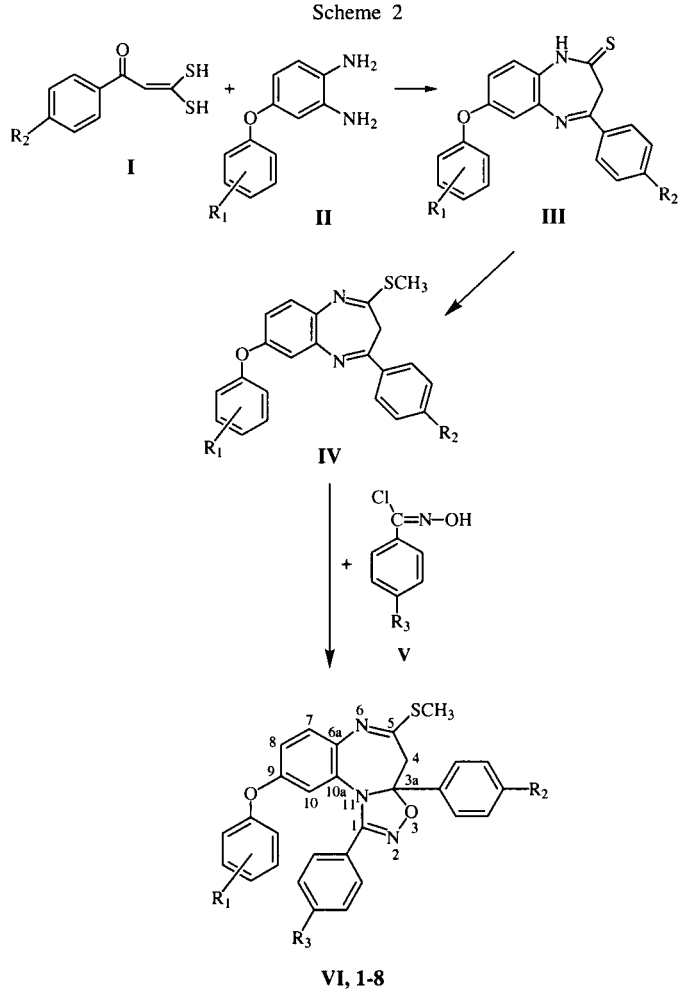
VI, 1-8

	R_1	R_2	R_3
1	H	CH_3	Cl
2	*m*-OCH_3	CH_3	Cl
3	*m*-OCH_3	CH_3	Br
4	*m*-OCH_3	OCH_3	Cl
5	*m*-OCH_3	OCH_3	Br
6	*p*-OCH_3	CH_3	Cl
7	*p*-OCH_3	OCH_3	Cl
8	*p*-OCH_3	OCH_3	Br

The reaction of compounds **I** with **II**, has been performed in anhydrous *ortho*-xylene at reflux for six hours. The 1*H*-1,5-benzodiazepine-2-thiones **III**, have been obtained in 44-65% yield. Treatment of compounds **III**, with sodium hydride and methyl iodide at reflux in anhydrous *ortho*-xylene for four hours afforded the Compounds **IV**, which have been obtained in 78-98%.

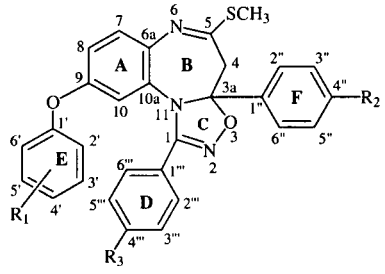
The reactions of compounds **IV** with a slight excess of benzonitrile oxide generated *in situ* from benzohydroxamoyl chloride **V**, and triethylamine, has been performed in

Scheme 2



VI, 1-8

Table 1
^{13}C NMR Spectral Data for Compounds **1-8**



VI, 1-8

Compounds	1	2	3	4	5	6	7	8
R_1	H	*m*-OCH_3	*m*-OCH_3	*m*-OCH_3	*m*-OCH_3	*p*-OCH_3	*p*-OCH_3	*p*-OCH_3
R_2	CH_3	CH_3	CH_3	OCH_3	OCH_3	CH_3	OCH_3	OCH_3
R_3	Cl	Cl	Br	Cl	Br	Cl	Cl	Br
C-1	156.0	155.6	155.7	155.5	156.1	155.1	154.5	155.2
C-3a	111.4	112.3	112.4	112.3	112.3	112.3	111.6	111.2
C-4	44.4	44.1	44.0	44.0	44.5	44.3	44.0	44.4
C-5	170.1	170.5	170.5	170.7	170.5	169.6	170.2	169.5
C-6a	131.4	130.7	130.6	130.7	131.0	130.4	130.7	131.3
C-7	127.4	127.0	127.0	127.0	127.3	128.8	127.0	127.2
C-8	119.7	120.5	120.5	120.4	120.1	118.0	118.3	118.0
C-9	157.0	157.8	158.0	158.1	158.4	156.0	155.7	156.0
C-10	119.8	121.5	121.5	121.4	12 1.0	118.0	118.8	118.0
C-10a	142.4	143.4	143.5	1.43.4	143.4	141.3	142.0	141.1
C-1'	153.8	152.5	152.4	152.4	153.5	149.9	149.8	149.8
C-2'	118.4	103.9	103.8	103.8	104.6	120.2	119.3	120.3
C-3'	129.7	160.4	160.3	160.6	161.0	114.9	114.9	114.8
C-4'	123.6	109.6	109.6	109.6	110.3	160.2	160.1	160.2
C-5'	129.7	130.4	130.2	130.4	130.4	114.9	114.9	114.8
C-6'	118.4	108.6	108.6	108.6	108.6	120.2	119.8	120.3
C-1"	137.2	136.5	136.2	131.3	132.2	137.3	131.4	132.2
$C_{2"}$,$C_{6"}$	125.6	128.7	126.0	127.6	127.2	128.9	127.6	127.0
$C_{3"}$,$C_{5"}$	128.9	126.0	128.9	113.6	113.7	129.0	113.6	113.6
$C_{4"}$	139.1	138.6	138.6	159.8	160.2	139.0	160.1	160.2
$C_{1'''}$	123.9	123.7	124.0	123.8	124.3	124.1	123.8	124.7
$C_{2'''}$,C6	128.7	129.51	131.6	128.7	131.8	129.0	129.4	131.8
$C_{3'''}$,C5	129.1	128.9	129.7	129.5	129.6	129.1	128.8	129.2
$C_{4'''}$	136.6	135.6	124.2	135.4	124.9	136.6	135.6	125.0
R_1	-	55.2	55.3	55.2	55.4	55.7	55.1	55.3
R_2	21.1	20.8	20.7	55.2	55.4	21.2	55.3	55.6

chloroform under a nitrogen atmosphere at reflux for twenty four hours. The oxadiazolo[4,5-*a*][1,5]benzodiazepines **VI**, **1-8** have been obtained in 11-35% yield.

The infrared spectrum of compounds **1-8** displayed absorptions at 1650-1586 cm^{-1} for C= N stretching; at 1310-1120 cm^{-1} for C-N stretching; at 1211-1028 cm^{-1} for C-O stretching and the corresponding absorptions for aromatic and R-substituents.

In the ^1H nmr spectra the presence of three proton signals at δ 2.42-2.48 singlet were assigned to the methyl protons joined to sulphur (S-CH_3). The presence of two protons signals at δ 3.00-3.10 and 3.23-3.29 doublet was consistent wih the methylene protons of the C-4. The presence of three proton signals at δ 6.60-7.06 multiplet was assigned to the aromatic protons of C-7, C-8 and C-10 of the benzodiazepine framework. The other aromatic protons appeared as a multiplet and AA'BB' system at δ 6.37-7.67 and the signal for the R-substituents.

The ^{13}C nmr spectra of compounds **1-8** are given in Table 1, and the signals were confirmed by using HETCOR, long range HETCOR, COSY and NOESY nmr experiments operating at 500 MHz.

The mass spectra of the compounds **1-8** include ions at m/z ion molecular $[M]^+$; $[M-15]^+$; $[M-47]^+$; $[356+R_1+R_2]^+$; $[326+R_1+R_2]^+$; $[325+R_1+R_2]^+$; $[309+R_1+R_2]^+$; $[298+R_1+R_2]^+$; $[102+R_2]^+$ and $[104+R_2]^+$ the base peak. The mass spectra of the compounds exhibit a stable molecular ion; and the main fragmentation was consistent with the

assigned structures. The proposed fragmentation pathways leading to the formation of a number of important daughter ions have been confirmed of the corresponding parent ion spectra by collision-induced dissociation experiments. The elemental composition of the molecular ion and the principal fragment ion was determined by exact mass measurements.

EXPERIMENTAL

The ir spectra were recorded on a Nicolet Magna TR-750 spectrophotometer. The ^{1}H-nmr spectra were recorded on a Varian Unity 300 spectrometer operating at 300 MHz and the ^{13}C-nmr spectra were recorded on a Varian Uniti Plus-500 spectrometer operating at 500 MHz in deuteriochloroform solution or deuteriodimethyl sulfoxide solution containing tetramethylsilane as the internal standard with chemical shifts δ (ppm) expressed downfield from tetramethylsilane. The mass spectra were measured on a Jeol JMS-AX505 and Jeol MS-SX 102A high resolution mass spectrometer with accurate mass determination of the molecular ion and the principal fragments ions, using the direct inlet system. The spectra were recorded by electron impact at an ionization chamber temperature of 190° and ionizing electron energy of 70 eV.

Compounds **II** and **V** have been prepared following literature methods with modifications [12-14].

General Procedure for the Synthesis of the 2,3-Dihydro-4-(*p*-substituted-phenyl)-7-[(*m*- and *p*-substituted)-phenoxy]-1*H*-1,5-benzodiazepine-2-thiones, **III**.

A mixture of 0.024 mole of 3,3-dimercapto-1-(*p*-substituted-phenyl)-2-propen-1-one, **I**, 0.024 mole of 3,4-diaminophenyl-R_1-phenyl ether, **II**, in 150 ml of dry *ortho*-xylene was heated at reflux for six hours. After cooling, the crystals were collected, and washed with hexane to yield the compounds **III**, with 46-60% yield.

General Procedure for the Synthesis of the 2-Methylthio-3*H*-4-(*p*-substituted-phenyl)-7-[(*m*- and *p*-substituted)-phenoxy]-1,5-benzodiazepines, **IV**.

A mixture of 0.005 mole of 1*H*-1,5-benzodiazepine-2-thiones, **III**, 0.015 mole of sodium hydride in 150 ml of dry *ortho*-xylene was heated at reflux for one hour. After the reaction mixture was cooled at room temperature, subsequently was added dropwise over a few minutes 0.014 mole of methyl iodide and the reflux continued for one hour. The reaction mixture was cooled to room temperature, filtered and the organic solution was dried with sodium sulphate, filtered and evaporated *in vacuo* to yield a semisolid; the compounds **IV**, with 78-98% yield.

General Procedure for the Synthesis of 5-Methylthio-4*H*-1-(*p*-substituted-phenyl)-3a-(*p*-substituted-phenyl)-9-[(*m*- and *p*-substituted)-phenoxy]-3a-4-dihydro-[1,2,4]-oxadiazolo[4,5-*a*][1,5]-benzodiazepines, **VI**, **1-8**.

To a stirred solution of 2-methylthio-3*H*-4-(*p*-substituted-phenyl)-7-[(*m*- and *p*-substituted)-phenoxy]-1,5-benzodiazepines, **IV**, (0.1 mole) in chloroform (5.0 ml), a solution of thiethylamine (0.2 mole) in the same solvent (3.0 ml) was added dropwise over a few minutes. The mixture was kept under nitrogen atmosphere at reflux for 30-45 minutes. Subsequently was added dropwise a solution of benzohydroxamoyl chloride derivatives, **V**, (0.2 mole) in chloroform (5.0 ml) and the reaction mixture was stirred and heated at reflux for 24 hours, followed by cooling to room temperature and the organic solution was dried (sodium sulfate) and evaporated *in vacuo* to yield a solid. The residual solid was purified on a silica gel chromatography column and elution with hexane-ethyl acetate (98:2) to yield the compounds **VI**, **1-8** (11-35%).

5-Methylthio-4*H*-1-(*p*-chlorophenyl)-3a-(*p*-methylphenyl)-9-phenoxy-3a,4-dihydro-[1,2,4]-oxadiazolo[4,5-*a*][1,5] benzodiazepine (**1**).

This compound was obtained as yellowish semisolid in 15% yield: ir (chloroform): ν C= N 1637, C-N 1320 and 1308, C-O 1123 and 1028 cm^{-1}; ^{1}H nmr (chloroform): δ 2.35 (s, 3H, $C_{4''}$-CH_3), 2.47 (s, 3H, S-CH_3), 3.01 and 3.27 (d, 2H, J = 14.1 Hz, 4-H), 6.72 (d, 1H, J = 2.7 Hz, 10-H), 6.82 (d, 2H, J = 7.8 Hz, 2'-H, 6'-H), 6.95 (d, d, 1H, J = 2.7, 8.7 Hz, 8-H), 7.06 (d, 1H, J = 8.7 Hz, 7-H), 7.09 (t, 1H, J = 7.5 Hz, 4'-H), 7.17 and 7.33 (AA'BB'; 4H, J = 8.9 Hz, phenyl protons of "D" ring), 7.20 and 7.50 (AA'BB'; 4H, J = 8.1 Hz, phenyl protons of "F" ring), 7.22 (t, 1H, J = 7.6, Hz, 5'-H), 7.28 (t, 1H, J = 7.6 Hz, 3'-H), ms: m/z 525 (M^+), 527 $[M+2]^+$, 529 $[M+4]^+$.

Anal. Calcd. for $C_{30}H_{24}ClN_3O_2S$: C, 68.49; H, 4.60; N, 7.99. Found: C, 68.38; H, 4.68; N, 7.92.

5-Methylthio-4*H*-1-(*p*-chlorophenyl)-3a-(*p*-methylphenyl)-9-(*m*-methoxyphenoxy)-3a,4-dihydro[1,2,4]oxadiazolo[4,5-*a*][1,5]-benzodiazepine (**2**).

This compound was obtained as yellow semisolid in 14% yield; ir (chloroform): ν C= N 1650, C-N 1320 and 1310, C-O 1120 and 1030 cm^{-1}; ^{1}H nmr (chloroform): δ 2.35 (s, 3H, $C_{4''}$-CH_3), 2.44 (s, 3H, S-CH_3), 3.10 and 3.27 (d, 2H, J = 14.4 Hz, 4-H), 3.70 (s, 3H, $C_{3''}$-OCH_3), 6.37 (d, d, 1H, J = 1.8, 7.8 Hz, 4'-H), 6.38 (d, 1H, J = 1.5 Hz, 2'-H), 6.69 (d, d; 1H, J = 2.4, 7.4 Hz, 6'-H), 6.96 (d, d, 1H, J = 2.5, 8.6 Hz, 8-H), 7.02 (d, 1H, J = 8.7 Hz, 7-H), 7.06 (d, 1H, J = 2.7 Hz, 10-H), 7.22 and 7.62 (AA'BB', 4H, J = 8.1 Hz, phenyl protons of "F" ring), 7.23 (t, 1H, J = 7.7 Hz, 5'-H), 7.41 (s, 4H, phenyl protons of "D" ring); ms: m/z 555 (M^+), 557 $[M+2]^+$, 559 $[M+4]^+$.

Anal. Calcd. for $C_{31}H_{26}ClN_3O_3S$: C, 66.96; H, 4.71; N, 7.56. Found: C, 66.88; H, 4.80; N, 7.49.

5-Methylthio-4*H*-1-(*p*-bromophenyl)-3a-(*p*-methylphenyl)-9-(*m*-methoxyphenoxy)-3a,4-dihydro[1,2,4]oxodiazolo[4,5-*a*][1,5]-benzodiazepine (**3**).

This compound was obtained as yellowish needles in 31% yield, mp 59°; ir (chloroform): ν C= N 1590, C-N 1260 and 1200, C-O 1191 and 1138 cm^{-1}; ^{1}H nmr (deuterio dimethyl sulfoxide): δ 2.31 (s, 3H, $C_{4''}$-CH_3), 2.44 (s, 3H, S-CH_3), 3.10 and 3.29 (d, 2H, J = 14.2 Hz, 4-H), 3.71 (s, 3H, $C_{3''}$-OCH_3), 6.37 (d, d, 1H, J = 1.8, 7.7 Hz, 4'-H), 6.38 (d, 1H, J = 1.5 Hz, 2'-H), 6.69 (d, d, 1H, J = 2.4, 7.4 Hz, 6'-H), 6.96 (d, d, 1H, J = 2.6, 8.5 Hz, 8-H), 6.99 (d, 1H, J = 8.6 Hz, 7-H), 7.06 (d, 1H, J = 2.7 Hz, 10-H), 7.22 and 7.62 (AA'BB', 4H, J = 8.1 Hz, phenyl protons of "F" ring); 7.23 (t, 1H, J = 7.6 Hz, 5'-H), 7.33 and 7.56 (AA'BB', 4H, J = 8.7 Hz, phenyl protons of "D" ring); ms: m/z 599 (M^+), 601 $[M+2]^+$, 603 $[M+4]^+$.

Anal. Calcd. for: $C_{31}H_{26}BrN_3O_3S$: C, 62.00; H, 4.36; N, 7.00. Found: C, 61.88; H, 4.42; N, 7.09.

5-Methylthio-4*H*-1-(*p*-chlorophenyl)-3a-(*p*-methoxyphenyl)-9-(*m*-methoxyphenoxy)-3a,4-dihydro[1,2,4]oxadiazolo[4,5-*a*]-[1,5]benzodiazepine (**4**).

This compound was obtained as yellowish needles in 35% yield, mp 50°; ir (chloroform): ν C= N 1590, C-N 1193 and 1180, C-O

1186 and 1140 cm^{-1}; ^1H nmr (deuterio dimethyl sulfoxide): δ 2.44 (s, 3H, S-CH_3), 3.10 and 3.28 (d, 2H, J = 14.4 Hz, 4-H), 3.70 (s, 3H, C_3-OCH_3), 3.76 (s, 3H, C_4"-OCH_3), 6.37 (d, d, 1H, J = 1.8, 7.7 Hz, 4'-H), 6.38 (d, 1H, J = 1.5 Hz, 2'-H), 6.69 (d, d, 1H, J = 1.8, 7.5 Hz, 6'-H), 6.95 and 7.67 (AA'BB', 4H, J = 8.7 Hz, phenyl protons of "F" ring), 6.97 (d, d, 1H, J = 2.1, 8.4 Hz, 8-H), 7.00 (d, 1H, J = 8.7 Hz, 7-H), 7.04 (d, 1H, J = 2.4 Hz, 10-H), 7.22 (t, 1H, J = 7.5 Hz, 5'-H), 7.41 and 7.61 (s, 4H, phenyl protons of "D" ring); ms: m/z 571 (M^+), 573 $[M+2]^+$, 575 $[M+4]^+$.

Anal. Calcd. for $C_{31}H_{26}ClN_3O_4S$: C, 65.08; H, 4.58; N, 7.35. Found: C, 65.17; H, 4.51; N, 7.26.

5-Methylthio-4*H*-1-(*p*-bromophenyl)-3a-(*p*-methoxyphenyl)-9-(*m*-methoxyphenoxy)-3a,4-dihydro[1,2,4]oxadiazolo[4,5-*a*]-[1,5]benzodiazepine (**5**).

This compound was obtained as yellowish needles in 18% yield, mp 70°; ir (chloroform): ν C= N 1586, C-N 1254 and 1183, C-O 1141 and 1101 cm^{-1}; ^1H nmr (chloroform): δ 2.48 (s, 3H, S-CH_3), 3.01 and 3.25 (d, 2H, J = 14.4 Hz, 4-H), 3.78 (s, 3H, C_3-OCH_3), 3.82 (s, 3H, C_4"-OCH_3), 6.39 (d, d, 1H, J = 1.6, 7.6 Hz, 4'-H), 6.40 (d, 1H, J = 1.5 Hz, 2'-H), 6.64 (d, d, 1H, J = 2.4, 7.4 Hz, 6'-H), 6.75 (d, 1H, J = 2.7 Hz, 10-H), 6.90 and 7.54 (AA'BB', 4H, J = 9.3 Hz, phenyl protons of "F" ring), 6.96 (d, d, 1H, J = 2.7, 8.7 Hz, 8-H), 7.05 (d, 1H, J = 8.4, 7-H), 7.18 (t, 1H, J = 8.7, 5'-H), 7.26 and 7.41 (AA'BB', 4H, J = 8.4 Hz, phenyl protons of "D" ring); ms: m/z 615 (M^+), 617 $[M+2]^+$, 619 $[M+4]^+$.

Anal. Calcd. for $C_{31}H_{26}BrN_3O_4S$: C, 60.39; H, 4.25; N, 6.82. Found: C, 60.30; H, 4.31; N, 6.72.

5-Methylthio-4*H*-1-(*p*-chlorophenyl)-3a-(*p*-methylphenyl)-9-(*p*-methoxyphenoxy)-3a,4-dihydro[1,2,4]oxadiazolo[4,5-*a*][1,5]-benzodiazepine (**6**).

This compound was obtained as yellowish semisolid in 19% yield; ir (chloroform): ν C= N 1595, C-N 1218 and 1120, C-O 1180 and 1100 cm^{-1}; ^1H nmr (chloroform): δ 2.36 (s, 3H, C_4"-CH_3), 2.45 (s, 3H, S-CH_3), 3.00 and 3.23 (d, 2H, J = 13.8 Hz, 4-H), 3.81 (s, 3H, C_4'-OCH_3), 6.60 (d, 1H, J = 2.7 Hz, 10-H), 6.80 (s, 4H, phenyl protons of "E" ring), 6.87 (d, d, 1H, J = 2.6, 8.6 Hz, 8-H), 7.02 (d, 1H, J = 8.7 Hz, 7-H), 7.18 and 7.50 (AA'BB', 4H, J = 8.7 Hz, phenyl protons of "F" ring), 7.23 and 7.32 (AA'BB', 4H, J = 8.7 Hz, phenyl protons of "D" ring); ms: m/z 555 (M^+), 557 $[M+2]^+$, 559 $[M+4]^+$.

Anal. Calcd. for $C_{31}H_{26}ClN_3O_3S$: C, 66.96; H, 4.71; N, 7.56. Found: C, 67.05; H, 4.64; N, 7.62.

5-Methylthio-4*H*-1-(*p*-chlorophenyl)-3a-(*p*-methoxyphenyl)-9-(*p*-methoxyphenoxy)-3a,4-dihydro[1,2,4]oxadiazolo[4,5-*a*][1,5]-benzodiazepine (**7**).

This compound was obtained as yellowish needles in 11% yield, mp 90°; ir (chloroform): ν C= N 1605, C-N 1280 and 1211, C-O 1177 and 1031 cm^{-1}; ^1H nmr (deuterio dimethyl sulfoxide): δ 2.42 (s, 3H, S-CH_3), 3.09 and 3.27 (d, 2H, J = 14.7 Hz, 4-H), 3.74 (s, 3H, C_4'.OCH_3), 3.77 (s, 3H, C_4"-OCH_3), 6.81 and 6.89 (AA'BB', 4H, phenyl protons of "E" ring), 6.82 (d, 1H, J = 2.7 Hz, 10-H), 6.88 (d, d, 1H, J = 2.4, 8.7 Hz, 8-H), 6.95 and 7.63 (AA'BB', 4H, J = 9.0 Hz, phenyl protons of "F" ring), 6.98 (d, 1H, J = 8.4 Hz, 7-H), 7.38 and 7.44 (AA'BB', 4H, J = 8.7 Hz, phenyl protons of "D" ring); ms: m/z 571 (M^+), 573 $[M+2]^+$, 575 $[M+4]^+$.

Anal. Calcd. for $C_{31}H_{26}ClN_3O_4S$: C, 65.08; H, 4.58; N, 7.35. Found: C, 65.00; H, 4.65; N, 7.29.

5-Methylthio-4*H*-1-(*p*-bromophenyl)-3a-(*p*-methoxyphenyl)-9-(*p*-methoxyphenoxy)-3a,4-dihydro[1,2,4]oxadiazolo[4,5-*a*][1,5]-benzodiazepine (**8**).

This compound was obtained as yellowish needles in 12% yield, mp 35°; ir (chloroform): ν C= N 1610, C-N 1319 and 1203, C-O 1211 and 1100 cm^{-1}; ^1H nmr (chloroform): δ 2.45 (s, 3H, S-CH_3), 3.02 and 3.24 (d, 2H, J = 14.1 Hz, 4-H). 3.81 (s, 6H, C_4'-OCH_3 and C_4"-OCH_3), 6.56 (d, 1H, J = 2.6 Hz, 10-H), 6.80 (s, 4H, phenyl protons of "E" ring), 6.87 (d, d, 1H, J = 2.4, 8.6 Hz, 8-H), 6.89 and 7.53 (AA'BB', 4H, J = 9.0 Hz, phenyl protons of "F" ring), 7.02 (d, 1H, J = 9.0 Hz, 7-H), 7.26 and 7.39 (AA'BB', 4H, J = 8.7 Hz, phenyl protons of "D" ring); ms: m/z 615 (M^+), 617 $[M+2]^+$, 619 $[M+4]^+$.

Anal. Calcd. for $C_{31}H_{26}BrN_3O_4S$: C, 60.39; H, 4.25; N, 6.82. Found: C, 60.47; H, 4.20; N, 6.74.

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[1] Author to whom correspondece should be addressed.

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