NEUROTROPIC AND PSYCHOTROPIC AGENTS. LIX.*

3-METHOXY-11-(3-DIMETHYLAMINOPROPYLIDENE)-6*H*,11*H*-DIBENZO[*b*,*e*]THIEPIN AND SOME RELATED COMPOUNDS

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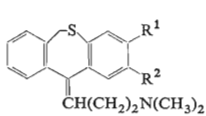
On cyclization of 2-(3-methoxyphenylthiomethyl)benzoic acid (*IV*) a rather low yield of 3-methoxy-6*H*-dibenzo[*b*,*e*]thiepin-11-one (*VI*) was obtained. This was reduced or oxidized to derivatives *VII*—*XI*. Reaction of ketone *VI* with 3-dimethylaminopropylmagnesium chloride gave rise to the tertiary alcohol *XII* which was dehydrated by an acid-catalyzed reaction to a mixture of geometric isomers of olefin *II*, from which one isomer (probably *trans*) was isolated pure. Reaction of *II* with ethyl chloroformate and subsequent hydrolysis effected partial demethylation to the secondary amine *XIV*. Amine *II* as a potential antihistaminic did not meet the expectation.

Antihistamine activity was found in the thymoleptic and antidepressive "prothiadene", *i.e. trans*-11-(3-dimethylaminopropylidene)-6*H*,11*H*-dibenzo[*b*,*e*]thiepin (*I*) and some of its derivatives[1-7]. In the 2-methyl derivative of *I* ("methiadene", *III*) the antihistamine activity was more powerful than the originally sought antireserpine activity and the compound found practical application as an antiallergic agent[1,5,8-12]. From the point of view of our classification[13], substances of the prothiadene series may be viewed as antihistaminics of the diarylmethylene type with a further ring in the molecule, formed by bridging the *o*-positions of the two rings with the —CH_2S— group. On the basis of known relationships between structure and antihistamine activity[13] we could expect an increase of this type of activity by substitution in one of the rings, namely in the *p*-position toward the central diphenylmethane carbon. From the point of view of the 6*H*,11*H*-dibenzo[*b*,*e*]thiepin system this means substitution in position 3. The work of Jucker's[7] and Stach's[14] groups which also dealt with the derivatives of *I*, does not mention 3-substitution derivatives. We described[4] the synthesis of 3-chloro and 3-bromo derivatives of *I*; their antihistamine activity was rather low[6]. Still, we thought it useful to verify this finding by using another substituent, the methoxy group having been chosen on the basis of analogy[13].

For this reason we describe here the synthesis of 3-methoxy-11-(3-dimethylaminopropylidene)-6*H*,11*H*-dibenzo[*b*,*e*]thiepin (*II*) and of some related compounds.



I, $R^1 = R^2 = H$
II, $R^1 = OCH_3$, $R^2 = H$
III, $R^1 = H$, $R^2 = CH_3$

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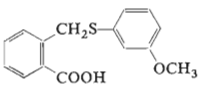
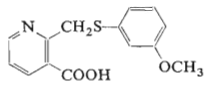
For the synthesis of *II* we used methods analogous to our previous work[1-4,8,9]. By reaction of phthalide[15] with the sodium salt of 3-methoxythiophenol[16] in ethanol we obtained 2-(3-methoxyphenylthiomethyl)benzoic acid (*IV*). Analogously, using 4-azaphthalide[17], we prepared 2-(3-methoxyphenylthiomethyl)nicotinic acid (*V*). While *IV* can be cyclized with polyphosphoric acid[1,2,8] to the desired 3-methoxy-6*H*-dibenzo[*b*,*e*]thiepin-11-one (*VI*) in an approximately 40% yield (the low yield is explained by simultaneously proceeding demethylation), attempts at cyclization of *V* were not successful[18].

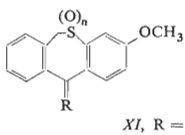
Reduction of ketone *VI* with sodium borohydride in methanol[2] gave rise to the secondary alcohol *VII*. Oxidation of ketone *VI* with hydrogen peroxide in acetic acid at room temperature and at the boiling temperature[2] gave rise to sulfoxide *VIII* and to sulfone *IX*, respectively. Reduction of ketone *VI* with zinc in boiling acetic acid[19] led to a complete removal of oxygen from position 11, resulting in 3-methoxy-6*H*,11*H*-dibenzo[*b*,*e*]thiepin (*X*). Its oxidation with hydrogen peroxide led to sulfoxide *XI*. Reaction of ketone *VI* with 3-dimethylaminopropylmagnesium chloride in tetrahydrofuran[4] gave rise to the tertiary alcohol *XII* which was dehydrated by heating with 10% sulfuric acid. The obtained olefinic base *II* was redistilled and the distillate crystallized for the most part. The crystalline product is homogeneous and, according to its spectrum, it is thought to have the *trans* configuration. The type of its IR spectrum in the range of 800–900 cm^{-1} in carbon disulfide resembles closely that of carbinol *XII*, where no steric interaction of the side chain with aromatic protons in position 1 and 2 can be expected. No further individual product could be isolated from the mother liquors. Heating of the tertiary amine *II* with ethylchloroformate in benzene[4] yielded the oily carbamate *XIII*. Its alkaline hydrolysis led to the secondary amine *XIV* which was isolated as crystalline hydrochloride.



IV



V



VI, R = O, n = 0
VII, R =  , n = 0
VIII, R = O, n = 1
IX, R = O, n = 2
X, R = H_2, n = 0

XI, R = H_2, n = 1
XII, R =  , n = 0
XIII, R = $CH(CH_2)_2NCOOC_2H_5$, n = 0
CH_3
XIV, R = $CH(CH_2)_2NHCH_3$, n = 0

Compound *II* was evaluated pharmacologically for its antihistamine effects. In the histamine aerosol test in guinea pigs upon *p.o.* administration (applied in the form of hydrochloride, the values shown referring to the base) its mean effective dose ED_{50} was 2·6 mg/kg. In the histamine detoxication test in guinea-pigs (histamine was applied subcutaneously at a dose of 20 mg/kg) compound *II* was completely inactive at oral doses of 1 mg/kg, or 10 mg/kg. "Dithiadene"[4,6,18] showed in this arrangement a fully protective effect at a dose of 1 mg/kg. Its ED_{50} was 0·6 mg/kg *p.o.*

The 3-methoxy derivative of prothiadene (*II*) thus showed only a low antihistamine effect in the aerosol test. By this finding one may conclude the study of the effect of 3-substitution in the molecule of *I* on its antihistamine activity. In contrast with other series of antihistaminics, the effect of the above substitution is negative as to the sought activity.

EXPERIMENTAL

The melting points of the preparations were determined in Kofler's block; the samples were dried in the usual way. The UV spectra (in methanol) were recorded in a Unicam SP 700 spectrophotometer, the IR spectra (in Nujol, unless stated otherwise) in a Unicam SP 200 G spectrophotometer and the NMR spectra (in deuteriochloroform, unless stated otherwise) in a ZKR 60 (Zeiss, Jena) spectrometer.

2-(3-Methoxyphenylthiomethyl)benzoic Acid (*IV*)

3-Methoxythiophenol[16] (75 g) was dissolved in a solution of 12·5 g sodium in 180 ml ethanol and, after addition of 71·5 g phthalide[15], the mixture was refluxed for 3 h. After diluting the hot solution with water it was filtered with charcoal and the filtrate was made acid with hydrochloric acid: 104 g (72%), m.p. 103—104°C (aqueous ethanol). NMR spectrum: δ 8·08 (d, 1 H, aromatic proton in the vicinity of carboxyl), 6·50—7·60 (m, 7 H, all the remaining aromatic protons), 4·53 (s, 2 H, CH_2S), 3·66 (s, 3 H, OCH_3), 10·03 (bs, 1 H, disappears on deuteration, COOH). For $C_{15}H_{14}O_3S$ (274·3) calculated: 65·69% C, 5·13% H, 11·69% S; found: 65·12% C, 5·21% H, 11·71% S.

2-(3-Methoxyphenylthiomethyl)nicotinic Acid (*V*)

Similarly to the previous case, 4·0 g 3-methoxythiophenol and 3·85 g 4-azaphthalide[17] yielded 5·5 g (68%) crude product which was purified by crystallization from a mixture of benzene and light petroleum, m.p. 149—150°C. NMR spectrum (CD_3SOCD_3): δ 8·64 (dd, J = 5·0; 2·0Hz, 1 H, aromatic proton in the vicinity of the pyridine N), 8·29 (dd, J = 8·0; 2·0 Hz, 1 H, aromatic proton in the vicinity of carboxyl), 7·40 (q, J = 8·0; 5·0 Hz, 1 H, the remaining aromatic proton of the pyridine ring), 6·50—7·25 (m, 3 H, aromatic protons of the benzene ring in positions 4, 5, 6), 6·96 (d, J = 2·0 Hz, 1 H, aromatic proton of the benzene ring in position 2), about 7·00 (1 H, COOH), 4·65 (s, 2 H, CH_2S), 3·69 (s, 3 H, OCH_3). For $C_{14}H_{13}NO_3S$ (275·3) calculated: 61·07% C, 4·75% H, 5·09% N, 11·65% S; found: 60·94% C, 4·64% H, 4·96% N, 11·82% S.

3-Methoxy-6*H*-dibenzo[*b*,*e*]thiepin-11-one (*VI*)

Acid *IV* (116·5 g) was added to 500 g polyphosphoric acid at 120°C and the mixture was heated for 4 h at 120—125°C. It was then poured into 2 kg ice, extracted with benzene, the extract washed with 5% KOH and evaporated. From the residue (80 g), crystallization from benzene and light petroleum yielded a total of 38·8 g (36%) product, m.p. 100—101°C. UV spectrum:

λ_{max} 235 nm (log ε 3·70), inflexion, 244 nm infl. (4·23), 254 nm (4·30), 293 nm (4·09). IR spectrum: 740 (1,2-C_6H_4), 828, 859, 881 (1,2,4-C_6H_3), 1030, 1060, 1229 and 1286 (Ar—O—R), 1584 (Ar), 1645 cm^{-1} (Ar—CO—Ar). For $C_{15}H_{12}O_2S$ (256·3) calculated: 70·29% C, 4·72% H, 12·51% S; found: 70·29% C, 4·78% H, 12·73% S.

3-Methoxy-6*H*,11*H*-dibenzo[*b*,*e*]thiepin-11-ol (*VII*)

$NaBH_4$ (0·6 g) was added in parts to a suspension of 2·6 g ketone *VI* in 30 ml methanol, and, after subsiding of the reaction, it was refluxed for 20 min. Methanol was then evaporated, the residue decomposed with water and extracted with chloroform. Treatment of the extract yielded 2·5 g (95%) product, m.p. 124—125°C (benzene–light petroleum). UV spectrum λ_{max} 220 nm (log ε 4·39), 253 nm infl. (3·84), 290 nm (3·30), 297 nm (3·27). IR spectrum: 738 and 774 (1,2-C_6H_4), 796, 828, 860 and 875 (1,2,4-C_6H_3), 1040 (CHOH), 1230 (Ar—O—R), 1600 (Ar), 3570 cm^{-1} (OH). NMR spectrum: δ 6·95—7·65 (m, 5 H, aromatic protons in positions 1, 7, 8, 9, 10), 6·56 (m, 2 H, aromatic protons in positions 2 and 4), 6·05 (s, 2 H, after deuteration 1 H, CHOH), 4·46 (s, 2 H, CH_2S), 3·64 (s, 3 H, OCH_3). For $C_{15}H_{14}O_2S$ (258·3) calculated: 69·74% C, 5·46% H, 12·41% S; found: 69·72% C, 5·53% H, 12·18% S.

3-Methoxy-6*H*-dibenzo[*b*,*e*]thiepin-11-one 5-Oxide (*VIII*)

30% H_2O_2 (1 ml) was added to a mixture of 15 ml acetic acid and 2·6 g ketone *VI* and the mixture was stirred occasionally for 48 h at room temperature. The solution formed was diluted with water and the precipitate was filtered: 2·4 g (87%), m.p. 161—162°C (ethanol). UV spectrum: λ_{max} 230 nm infl. (log ε 4·18), 263 nm (3·97), 3·01 nm (4·09). IR spectrum: 768 (1,2-C_6H_4), 793, 845 and 872 (1,2,4-C_6H_3), 1030 (SO), 1260 (Ar—O—R), 1585 (Ar), 1636 cm^{-1} (ArCOAr). For $C_{15}H_{12}O_3S$ (272·3) calculated: 66·16% C, 4·44% H, 11·77% S; found: 65·88% C, 4·50% H, 11·63% S.

3-Methoxy-6*H*-dibenzo[*b*,*e*]thiepin-11-one 5,5-Dioxide (*IX*)

A mixture of 2·6 g ketone *VI*, 15 ml acetic acid and 4·6 ml 30% H_2O_2 was refluxed for 3 h. After cooling, it was diluted with water and, after standing overnight, the precipitated product was filtered; 2·1 g (72%), m.p. 134—135°C (ethanol). UV spectrum: λ_{max} 232 nm (log ε 4·07), 296 nm (4·05). IR spectrum: 750 (1,2-C_6H_4), 788, 843, 868 and 895 (1,2,4-C_6H_3), 1125 (SO_2), 1270 (Ar—O—C), 1315 (SO_2), 1600 (Ar), 1650 cm^{-1} (ArCOAr). For $C_{15}H_{12}O_4S$ (288·3) calculated: 62·50% C, 4·20% H, 11·10% S; found: 62·08% C, 4·09% H, 11·00% S.

3-Methoxy-6*H*,11*H*-dibenzo[*b*,*e*]thiepin (*X*)

Zinc powder (12·6 g) was added in parts under stirring at 50°C to a mixture of 4·0 g ketone *VI* and 120 ml acetic acid. The mixture was refluxed for 8 h. After cooling, it was filtered, the filtrate partly evaporated and the remainder was diluted with water. The separated oil was left to stand in the refrigerator whereupon it crystallized to a nonhomogeneous product which was filtered (3·5 g). According to thin-layer chromatography on alumina it was characterized as a mixture of two compounds from which the desired product was isolated (0·6 g) by chromatography on a column of Al_2O_3, eluting with a mixture of benzene and light petroleum 2 : 3; m.p. 82—83°C (benzene–light petroleum). NMR spectrum: δ 7·24 (s, 4 H, aromatic protons in positions 7, 8, 9, 10), 7·05 (d, 1 H, aromatic proton in position 1), 6·61 (s, 1 H, aromatic proton in position 4), 6·55 (m, 1 H, aromatic proton in position 2), 4·22 (s, 2 H, $ArCH_2Ar$), 4·02 (s, 2 H, CH_2S), 3·64 (s, 3 H, OCH_3). For $C_{15}H_{14}OS$ (242·3) calculated: 74·34% C, 5·82% H, 13·23% S; found: 74·20% C, 5·75% H, 13·05% S.

3-Methoxy-6*H*,11*H*-dibenzo[*b*,*e*]thiepin 5-Oxide (*XI*)

A mixture of 3·5 ml acetic acid, 0·6 g *X* and 0·24 ml 30% H_2O_2 was left for 48 h at room temperature. Filtration removed a small amount of precipitate and the filtrate was diluted with water. On standing and cooling, the desired product precipitated, was filtered and recrystallized from ethanol; m.p. 129–131°C. IR spectrum (KBr): 759 (1,2-C_6H_4), 821 and 880 (1,2,4-C_6H_3), 1027 (Ar—SO), 1245 and 1295 (Ar—O—R), 1490 and 1598 cm^{-1} (Ar). NMR spectrum: δ 7·00–7·50 (m, 6 H, aromatic protons in positions 1, 4, 7, 8, 9, 10), 6·88 (dd, J = 9·0; 2·0 Hz, 1 H, aromatic proton in position 2), 3·60–4·80 (m, 4 H, $ArCH_2Ar$ and CH_2S), 3·82 (s, 3 H, OCH_3). For $C_{15}H_{14}O_2S$ (258·3) calculated: 69·74% C, 5·46% H, 12·41% S; found: 69·31% C, 5·49% H, 12·38% S.

3-Methoxy-11-(3-dimethylaminopropyl)-6*H*,11*H*-dibenzo[*b*,*e*]thiepin-11-ol (*XII*)

A solution of 12·75 g ketone *VI* in 30 ml tetrahydrofuran was added dropwise under stirring and external cooling with ice to a solution of 3-dimethylaminopropylmagnesium chloride[4] (from 12·0 g 3-dimethylaminopropyl chloride, 2·4 g Mg in 30 ml tetrahydrofuran). The mixture was stirred for 1 h at room temperature and then decomposed with a solution of 15 g NH_4Cl in 100 ml water. It was extracted with chloroform, the extract was dried with $MgSO_4$ and evaporated. Crystallization of the residue from a mixture of benzene and light petroleum yielded a total of 14·2 g (84%) product melting at 128–129°C. IR spectrum: 760 (1,2-C_6H_4), 810, 860 and 870 (1,2,4-C_6H_3), 1045 and 1050 (OH), 1230 (Ar—O—R), 1560, 1645 (Ar), 2625 (OH in hydrogen bond), 2780 cm^{-1} (N—CH_3). The region from 700 to 900 cm^{-1} was examined in detail (CS_2): 716, 749, 762 (4 H), 801, 812, 835, 853 (2 H), 878 and 889 cm^{-1} (1 H). NMR spectrum: δ 7·00–8·12 (m, 5 H, aromatic protons in positions 1, 7, 8, 9, 10), 6·45–6·80 (m, 2 H, aromatic protons in positions 2 and 4), 4·65 and 3·78 (2 d, 2 H, CH_2S), 3·68 (s, 3 H, OCH_3), 2·08 (s, 6 H, CH_3NCH_3), 1·30–2·50 (m, 7 H, 3 CH_2 and OH). For $C_{20}H_{25}NO_2S$ (343·5) calculated: 69·93% C, 7·34% H, 4·08% N, 9·33% S; found: 70·16% C, 7·37% H, 3·98% N, 9·45% S.

3-Methoxy-11-(3-dimethylaminopropylidene)-6*H*,11*H*-dibenzo[*b*,*e*]thiepin (*II*)

A solution of 30 g amino alcohol *XII* in 280 ml 10% sulfuric acid was refluxed for 15 min. After filtration with charcoal, the cooled filtrate was made alkaline with aqueous ammonia and the base was isolated by extraction with chloroform. An oily mixture of the geometric isomers was obtained in a theoretical yield. A part of the base was redistilled *in vacuo*; b.p. 184°C/0·5 Torr. The distillate crystallized almost completely and the product was once recrystallized from dioxane to a constant m.p. of 84–86°C. UV spectrum: λ_{max} 239 nm (log ε 4·39), 279 nm (3·89). IR spectrum (CS_2): 718, 757, 763 (4 H), 799, 810, 835, 853 (2 H), 882 (1 H), 1040 and 1228 (Ar—O—R), 1598 cm^{-1} (Ar). The form of the spectrum, particularly in the region of extraplanar vibrations of two adjacent aromatic protons, is identical with the corresponding region of the spectrum of carbinol *XII*, whereas a *trans*-configuration for *II* is assumed. NMR spectrum: δ 7·00–7·40 (m, 5 H, aromatic protons in positions 1, 7, 8, 9, 10), 6·58 (dd, J = 9·0; 3·0 Hz, 1 H, aromatic proton in position 2), 6·40 (d, 1 H, aromatic proton in position 4), 5·75 (t, J = 7·0 Hz, 1 H, =CH), 4·90 and 3·28 (d, J = 14·0 Hz, 2 H, CH_2S), 3·63 (s, 3 H, OCH_3), about 2·15 (m, 4 H, CH_2CH_2), 1·09 (s, 6 H, CH_3NCH_3). For $C_{20}H_{23}NOS$ (325·4) calculated: 73·82% C, 7·12% H, 4·30% N, 9·84% S; found: 73·65% C, 7·04% H, 4·33% N, 9·85% S.

Hydrochloride(*monohydrate*), m.p. 113–114°C (ethanol–ether or water). IR spectrum (KBr): 1490 and 2661 (NH^+), 3440 cm^{-1} (H_2O). For $C_{20}H_{26}ClNO_2S$ (379·9) calculated: 63·23% C, 6·89% H, 9·33% Cl, 3·68% N, 8·44% S; found: 63·28% C, 6·61% H, 9·68% Cl, 3·72% N, 8·76% S.

3-Methoxy-11-(3-methylaminopropylidene)-6*H*,11*H*-dibenzo[*b*,*e*]thiepin (*XIV*)

A solution of 11·0 g base *II* in 40 ml benzene was added dropwise over a period of 45 min to a solution of 4·75 g ethyl chloroformate in 20 ml benzene at 75°C. The mixture was refluxed under stirring for 1·5 h, cooled, washed with water and 10% H_2SO_4, dried and filtered with charcoal. Evaporation yielded 10·0 g (77%) oily carbamate *XIII*. A mixture of 9·3 g crude carbamate, 7·45 g KOH and 9 ml ethanol was refluxed for 2 h at 125—130°C. After cooling, it was diluted with water and extracted with benzene. The benzene solution was shaken with 50 ml 10% HCl, whereupon the hydrochloride precipitated: 7·7 g (85%), m.p. 248—250°C (methanol). UV spectrum: λ_{max} 239 nm (log ε 4·37), 279 nm (3·86). IR spectrum: 760 (1,2-C_6H_4), 800 and 858 (1,2,4-C_6H_3), 1226 (Ar—O—R), 1590 (Ar), 1482, 2720 and 2760 cm^{-1} (NH_2^+). NMR spectrum (CD_3SOCD_3): δ 7·10—7·60 (m, 5 H, aromatic protons in positions 1, 7, 8, 9, 10), 7·72 (dd, J = 9·0; 2·5 Hz, 1 H, aromatic proton in position 2), 6·59 (d, J = 2·5 Hz, 1 H, aromatic proton in position 4), 5·91 (t, J = 7·0 Hz, 1 H, =CH), 4·80 (d, J = 14·0 Hz) and 3·66 (d, J = 14·0 Hz, together 2 H, CH_2S), 3·70 (s, 3 H, OCH_3), 2·96 (t, J = 8·0 Hz, 2 H, CH_2N), 2·47 (s, 3 H, N—CH_3), about 2·40 (m, 2 H, =C—CH_2), 9·28 (bs, 1 H, NH). For $C_{19}H_{22}ClNOS$ (347·9) calculated: 65·60% C, 6·37% H, 10·19% Cl, 4·02% N, 9·21% S; found: 65·45% C, 6·58% H, 10·05% Cl, 4·00% N, 9·18% S.

The antihistamine effect of II *was estimated under the direction of Dr J. Metyš in the pharmacological department of this institute. The NMR spectra were measured and interpreted by Dr B. Kakáč and Dr J. Holubek at the physico-chemical department of this institute. The analyses were performed by Mr K. Havel, Mrs V. Šmídová and Mrs J. Komancová at the analytical department of this institute.*

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