

NEW SYNTHESIS IN THE ACYL-OXIMINE DERIVATIVES SERIA

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Abstract

This paper is a continuation of our research concerning the obtaining and characterization of other new acyl-oximine derivatives with tricyclic structure, which may become new compounds with pharmacological properties like tricyclic antidepressants.

These substances are O-acyl-oximine derivatives with 5H-dibenzo[a,d]cycloheptatriene structure, whose structures were confirmed by elemental analysis, IR and NMR-spectrometry.

Rezumat

Acest articol reprezintă o continuare a cercetărilor făcute de autori referitoare la sinteza și caracterizarea unor noi acil-oximine cu structură triciclică, cu potențială acțiune farmacologică asemănătoare antidepressivelor triciclice. Compușii nou sintetizați sunt derivați O-acil-oximinici ai 5H-dibenzo[a,d]cicloheptatrienei, a căror structură a fost confirmată prin analiză elementală și spectrală (IR și RMN).

Keywords: oximes, oximines, acylation, dibenzocycloheptatriene, 5H-dibenzo[a,d]cycloheptene.

Introduction

It is well known that the compounds with tricyclic structure, like dibenzocycloheptadiene and dibenzocycloheptatriene derivatives, have certain pharmacological activities: antidepressant effect [5, 10], analgesic properties [8], use for attention deficit hyperactivity disorders (ADHD) [11]; other authors consider these compounds as a new strategy of therapy for functional dyspepsia as the second-step therapy [9]. Researchers have also shown that some tricyclic antidepressant can reduce pain in peripheral neuropathy caused by cancer chemotherapy [12]. This therapeutic effect is similar to the effect produced by gabapentin or the combination between

gabapentin and tramadol in the prophylaxis of paclitaxel-induced neuropathy [13].

Despite these important therapeutic effects, most of this tricyclic antidepressants have side effects, mostly caused by their anticholinergic activity, including: changes in appetite, muscle stiffness, nausea, constipation, tremor, dizziness, urinary retention, blurred vision, changes in sexual function; some rare side effects are: hypotension, tinnitus, seizures, mania, heart block, arrhythmias, lips and mouth ulcers, extrapyramidal symptoms, suicidal thoughts [7].

In this article we present the continuation of our previous research [2, 3, 4] in order to obtain new compounds with tricyclic structure, some O-acyl-oximine derivatives of 5H-dibenzo[a,d]cycloheptatriene, with antidepressant potential, analgesic or anti-inflammatory effects.

Materials and Methods

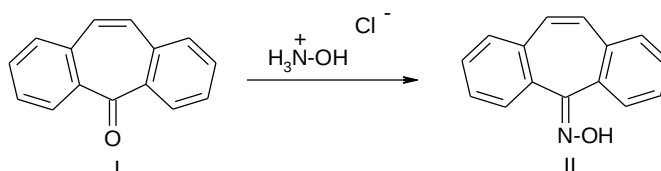
Melting points were measured in open capillary tubes on an Electrothermal 9100 apparatus and are uncorrected. Infrared spectra were recorded on a FT/IR-solid in ATR spectrometer (the signal intensities (height) were denoted by the following abbreviations: w = weak, m = medium, s = strong, v = variable). The NMR spectra were recorded on a Gemini 300BB instrument at room temperature, operating at 300 MHz for ^1H and 75 MHz for ^{13}C . The chemical shifts were recorded in δ units (ppm), relative to residual peak of the deuterated solvent (CDCl_3 and DMSO-d_6). Tetrametylsilane was used as internal standard. The coupling constants values are reported in hertz and the splitting patterns are abbreviated as following: s, singlet; d, doublet; t, triplet; m-multiplet; b, broad. The elemental analyses were performed on a Perkin Elmer CHNS/O Analyser Series II 2400 apparatus and the results were in agreement with the calculated values.

All starting materials and solvents were purchased from commercial suppliers and used without purification unless otherwise noted.

Intermediate synthesis

In order to obtain the new compounds, we performed an acylation reaction of the main intermediate, 5-oximino-5H-dibenzo[a,d]cycloheptene (II), with some acid chlorides.

The oxime (II) was obtained by condensation of dibenzosuberone (5H-dibenzo [a,d]cyclohepten-5-one) (I) with hydroxylamine hydrochloride (Figure 1).

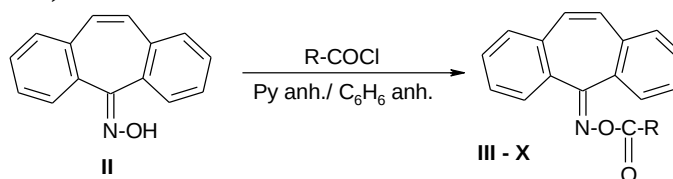
**Figure 1.**

Synthesis of the intermediate compound II

In a round bottom flask equipped with condenser, stirrer and dropping funnel were added 70 mL ethanol, 15 g dibenzosuberone (0.072 moles) and 17 g sodium hydroxide (0.425 moles). The mixture was stirred until the complete dissolution of the compounds. A solution of 7.5 g hydroxylamine hydrochloride (0.107 moles) in 65 mL ethanol was drop wise added. The mixture was refluxed for 6 hours. After the mixture was cooled to room temperature, 55 mL concentrated hydrochloric acid and 120 mL water were added. During this process the temperature was maintained under 20 °C. We obtained a precipitate which was filtered, washed with water and then dried at 75 °C, resulting 14.4 g crude oxime (II), which was recrystallized from toluene (90% yield; m.p. 184-185 °C).

Final compounds synthesis

The new O-acyl-oximines (III – X) were obtained by treating the oxime (II) with substituted aromatic acid chlorides in anhydrous benzene (C_6H_6 anh.), using anhydrous pyridine (Py anh.) as hydracid acceptor agent [1, 6] (Figure 2).

**Figure 2.**

Synthesis of the new O-acyl-oximes (III–X)

The general method of synthesis is the following:

0.0026 moles of oxime II were dissolved in 15 mL anhydrous benzene; gradually were added 0.0026 moles of each corresponding acid chlorides in 15 mL anhydrous benzene and 0.0026 moles anhydrous pyridine. A white precipitate (pyridinium hydrochloride) immediately appeared. The reaction mixture was refluxed for 3 hours and afterwards was filtered. The organic phase was evaporated to dryness to give the final crude compound. All the new acyl-oximines were recrystallized from isopropanol.

Results and Discussion

Following the above general method of synthesis we obtained eight new acyl-oximines. The compounds are solid, crystalline, white, or light yellow substances. The structures were confirmed by elemental analysis, IR and NMR spectra. Chemical structures of the new compounds, molecular formula and molecular mass are presented in Table I.

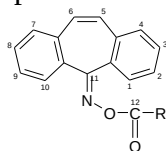


Table I
Characterization of the new acyl-oximine derivatives

Compound	R	Molecular formula	Molecular mass
III		C ₂₃ H ₁₄ F ₃ NO ₂	393.36
IV		C ₂₃ H ₁₄ F ₃ NO ₂	393.36
V		C ₂₃ H ₁₄ F ₃ NO ₂	393.36
VI		C ₂₃ H ₁₃ F ₄ NO ₂	411.35
VII		C ₂₃ H ₁₃ F ₄ NO ₂	411.35
VIII		C ₂₃ H ₁₃ F ₄ NO ₂	411.35
IX		C ₂₃ H ₁₃ F ₄ NO ₂	411.35
X		C ₂₃ H ₁₃ F ₄ NO ₂	411.35

In the following are presented elemental composition, the melting point (m.p.), reaction yield and spectral data for the compounds III-X.

In the following are presented elemental composition, the melting point (m.p.), reaction yield and spectral data for the compounds III-X.

Compound III: **O-(2-Trifluoromethyl-benzoyl)-5-oximino-5H-dibenzo[a,d]cycloheptene**

Elemental analysis: Calculated: C 70.23%, H 3.59%, N 3.56%; Found C 70.45%, H 3.36%, N 3.70%; m.p. 117-118 °C; yield 81%.

¹H-NMR (CDCl₃, δ ppm, *J* Hz, T=298K): 7.83(m, 1H, H-1); 7.70(m, 1H, H-15); 7.62÷7.33(m, 10H, H-arom); 7.00(d, 1H, H-6 or H-5, syst. AB, 12.4); 6.95(d, 1H, H-5 or H-6, syst. AB, 12.4).

The heteronuclear correlation spectrum revealed that the proton at δ = 7.83 ppm is linked to C15. The spectral attribution for C15 is relieved because it has a multiplicity feature (1:3:3:1), given by the presence of three vicinal fluorine atoms, which is achieved by scalar coupling *J* (3F-C¹⁵) = 5.2 Hz.

¹³C-NMR (CDCl₃, δ ppm, T=298K): 165.21(C-11); 164.14(C-12); 134.33(Cq); 133.23(Cq); 129.81(q, C-13, *J*)= 0.6 Hz); 129.50(Cq); 128.85(q, C-14, *J*(3F-C¹⁴)=30.2 Hz); 122.11(q, CF₃, *J*(3F-C)= 273.7 Hz); 131.71(q, C-18, *J*(3F-C¹⁸)=1.1 Hz); 131.36(CH); 130.67(CH); 130.17(CH); 130.14(CH); 129.75(CH); 129.38(CH); 129.10(CH); 128.98(CH); 128.97(CH); 128.24(CH); 128.19(C-1); 127.76(CH); 126.74(q, C-15, *J*(3F-C¹⁵)= 5.2 Hz).

FT-IR (solid in ATR, ν cm⁻¹): 3067w; 3023w; 1768vs; 1604w; 1587w; 1331w; 1311s; 1281m; 1241s; 1154s; 1125vs; 1080s; 1031s; 974s; 901w; 876w; 852m; 798m; 762s; 720m; 688w; 642w.

Compound IV: **O-(3-Trifluoromethyl-benzoyl)-5-oximino-5H-dibenzo[a,d]cycloheptene**

Elemental analysis: Calculated: C 70.23%, H 3.59%, N 3.56%; Found C 70.02%, H 3.42%, N 3.78%; m.p. 145-146 °C; yield 78%.

¹H-NMR (CDCl₃, δ ppm, *J* Hz, T=298K): 8.11÷8.07(m, 2H, H-18, H-16); 7.84(dd, 1H, H-1, 1.4, 7.8); 7.79(bd, 1H, H-16, 8.0); 7.70(m, 1H, H-10); 7.58÷7.43(m, 7H, H-arom); 7.02(d, 1H, H-6 or H-5, syst. AB, 12.1); 6.97(d, 1H, H-5 or H-6, syst. AB, 12.1).

¹³C-NMR (CDCl₃, δ ppm, T=298K): 164.89(C-11); 162.26(C-12); 134.36(Cq); 133.42(Cq); 133.20(Cq); 131.26(q, C-15, *J*(3F-C¹⁵)=33.0 Hz); 129.78(Cq); 129.56(Cq); 123.51(q, CF₃, *J*(3F-C)= 272.4 Hz); 132.97(q, C-14, ³*J*(3F-C¹⁴)=1.6 Hz); 130.87(CH); 130.13(CH); 129.85(CH); 129.74(CH); 129.66(CH); 129.56(CH); 129.49(CH); 129.23(CH);

129.08(CH); 129.03(CH); 128.40(CH); 128.25(CH); 127.62(CH); 126.62(q, C-16, $^3J(3F-C^{16})=3.9$ Hz).

FT-IR (solid in ATR, ν cm^{-1}): 3054w; 3027w; 1754vs; 1607w; 1590w; 1437w; 1333vs; 1223vs; 1170s; 1103vs; 1062vs; 979s; 885m; 869m; 819w; 793m; 763s; 743m; 710w; 677m; 645w.

Compound V: **O-(4-Trifluoromethyl-benzoyl)-5-oximino-5H-dibenzo[a,d]cycloheptene**

Elemental analysis: Calculated: C 70.23%, H 3.59%, N 3.56%; Found C 70.39%, H 3.48%, N 3.68%; m.p. 168-169 °C; yield 77.5%.

$^1\text{H-NMR}$ (CDCl_3 , δ ppm, J Hz, $T=298\text{K}$): 7.97(d, 2H, H-15, H-17, 8.2); 7.83(m, 1H, H-1); 7.66(d, 2H, H-14, H-18, 8.2); 7.57÷7.42(m, 7H, H-arom); 7.02(d, 1H, H-6 or H-5, syst. AB, 12.1); 6.97(d, 1H, H-5 or H-6, syst. AB, 12.1).

$^{13}\text{C-NMR}$ (CDCl_3 , δ ppm, $T=298\text{K}$): 164.90(C-11); 162.52(C-12); 134.68(q, C-16, $J(3F-C^{16})=32.6$ Hz); 134.34(Cq); 133.48(Cq); 132.21(Cq); 131.97(q, C-13, $J(3F-C^{13})=1.4$ Hz); 129.83(Cq); 123.50(q, CF_3 , $J(3F-C)=272.9$ Hz); 130.90(C-5 or C-6); 130.10(C-14, C-18); 130.10(C-5 or C-6); 129.86(CH); 129.65(CH); 129.45(CH); 129.07(CH); 129.06(CH); 128.31(CH); 128.23(CH); 127.63(CH); 125.54(q, C-15, C-17, $^3J(3F-C^{15-17})=3.7$ Hz).

FT-IR (solid in ATR, ν cm^{-1}): 3063w; 1754vs; 1589w; 1409w; 1326s; 1254s; 1237s; 1177m; 1157m; 1116s; 1081vs; 1065vs; 1014m; 976s; 854m; 798m; 765s; 721w; 693m; 677w.

Compound VI: **O-(2-Fluoro-4-trifluoromethyl-benzoyl)-5-oximino-5H-dibenzo[a,d]cycloheptene**

Elemental analysis: Calculated: C 67.16%, H 3.19%, N 3.41%; Found C 67.38%, H 3.03%, N 3.46%; m.p. 133-134 °C; yield 83%.

$^1\text{H-NMR}$ (CDCl_3 , δ ppm, J Hz, $T=298\text{K}$): 7.95(t, 1H, H-18, $^3J(\text{H}^{17}-\text{H}^{18})=^4J(\text{F}^{14}-\text{H}^{18})=7.4$ Hz); 7.82(m, 1H, H-1); 7.67(m, 1H, H-10); 7.53÷7.41(m, 8H, H-arom); 7.37(bd, 1H, H-15, $^3J(\text{F}-\text{H}^{15})=10.1$ Hz); 7.00(d, 1H, H-6 or H-5, syst. AB, 12.1); 6.96(d, 1H, H-5 or H-6, syst. AB, 12.1).

$^{13}\text{C-NMR}$ (CDCl_3 , δ ppm, $T=298\text{K}$): 165.24(C-11); 161.25(d, C-14, $J(\text{F}-\text{C}^{14})=263.5$ Hz); 160.81(d, C-12, $^3J(\text{F}-\text{C}^{11})=4.1$ Hz); 136.34(qd, C-16, $J(3F-C^{16})=33.6$ Hz, $J(\text{F}-\text{C}^{16})=8.0$ Hz); 134.37(Cq); 133.23(Cq); 133.11(Cq); 129.77(Cq); 124.39(d, C-13, $J(\text{F}-\text{C}^{13})=2.6$ Hz); 122.56(qd, CF_3 , $J(3F-C)=277.3$ Hz, $^4J(\text{F}-\text{CF}_3)=2.6$ Hz); 133.22(CH); 133.20(d, C-18, $J(\text{F}-\text{C}^{18})=1.8$ Hz); 130.70(C-5 or C-6); 129.84(CH); 129.55(CH); 129.11(CH); 129.02(CH); 129.00(CH); 128.69(d, C-10, $J(\text{F}-\text{C}^3)=2.0$ Hz); 128.23(CH); 127.74(CH); 120.94(qv, C-17, $^3J(3F-C^{17})=^4J(\text{F}-\text{C}^{17})=3.9$ Hz); 114.62(dq, C-15, $^2J(\text{F}-\text{C}^{15})=25.6$ Hz, $^3J(3F-C^{15})=3.9$ Hz).

It should be noted the coupling in the space between fluorine atom in position 14 and the pair proton - carbon in position 10. The relationship resulting from the carbon and proton spectra, as well as the two-dimensional homo and heteronuclear correlations.

FT-IR (solid in ATR, ν cm^{-1}): 3065w; 1750s; 1588w; 1503w; 1426m; 1332s; 1310m; 1267w; 1240m; 1215s; 1171s; 1126vs; 1092m; 1071s; 1040m; 976m; 918m; 902m; 877m; 846m; 798m; 773s; 745w; 722w; 694m.

Compound VII: **O-(2-Fluoro-3-trifluoromethyl-benzoyl)-5-oximino-5H-dibenzo[a,d]cycloheptene**

Elemental analysis: Calculated: C 67.16%, H 3.19%, N 3.41%; Found C 67.42%, H 3.36%, N 3.28%; m.p. 164-165 °C; yield 89%.

$^1\text{H-NMR}$ (CDCl_3 , δ ppm, J Hz, $T=298\text{K}$): 8.01(td, 1H, H-18, $^3J(\text{H}^{17}-\text{H}^{18})=^4J(\text{F}^{14}-\text{H}^{18})=8.0$ Hz, $^4J(\text{H}^{16}-\text{H}^{18})=2.0$ Hz); 7.82(dd, 1H, H-1, 1.4, 7.4); 7.76(bd, 1H, H-16, 8.0); 7.70(m, 1H, H-10); 7.41÷7.53(m, 6H, H-arom); 7.28(t, 1H, H-17, 8.0); 7.00(d, 1H, H-5 or H-6, syst. AB, 12.4); 6.96(d, 1H, H-5 or H-6, syst. AB, 12.4).

$^{13}\text{C-NMR}$ (CDCl_3 , δ ppm, $T=298\text{K}$): 165.18(C-11); 160.85(d, C-12, $J(\text{F}-\text{C}^{11})=3.5$ Hz); 158.97(dq, C-14, $J(\text{F}-\text{C}^{14})=270.9$ Hz, $^3J(\text{F}-\text{C}^{14})=2.1$ Hz); 134.37(Cq); 133.14(Cq); 133.08(Cq); 131.15(qd, C-16, $J(3\text{F}-\text{C}^{16})=4.5$ Hz, $J(\text{F}^{14}-\text{C}^{16})=2.3$ Hz); 129.66(Cq); 119.86(qd, C-15, $J(3\text{F}-\text{C}^{15})=33.6$ Hz, $J(\text{F}-\text{C}^{15})=13.3$ Hz); 119.14(d, C-13, $J(\text{F}-\text{C}^{13})=10.4$ Hz); 136.07(dq, C-18, $^3J(\text{F}-\text{C}^{18})=1.5$ Hz, $J(3\text{F}-\text{C}^{18})=0.7$ Hz); 130.64(C-5 or C-6); 130.23(C-6 or C-5); 129.84(CH); 129.60(CH); 129.11(CH); 128.99(CH); 128.98(CH); 128.78(d, C-10, $J(\text{F}^{14}-\text{C}^{10})=2.4$ Hz); 128.26(C-1); 127.73(CH); 123.99(CH); 123.94(CH).

FT-IR (solid in ATR, ν cm^{-1}): 3105w; 3066w; 3029w; 1740s; 1610m; 1594m; 1464s; 1331s; 1271s; 1238m; 1196s; 1171m; 1157m; 1119vs; 1097m; 1075s; 1059s; 980m; 948w; 886w; 875w; 860m; 845w; 793m; 764m; 752s; 737m; 712w; 700w; 676m.

Compound VIII: **O-(2-Fluoro-6-trifluoromethyl-benzoyl)-5-oximino-5H-dibenzo[a,d]cycloheptene**

Elemental analysis: Calculated: C 67.16%, H 3.19%, N 3.41%; Found C 67.40%, H 3.04%, N 3.58%; m.p. 135-136 °C; yield 78.5%.

$^1\text{H-NMR}$ (CDCl_3 , δ ppm, J Hz, $T=298\text{K}$): 7.80(m, 1H, H-1); 7.56÷7.34(m, 9H, H-arom); 7.30(td, 1H, H-15, 8.2 1.6); 6.99(d, 1H, H-5 or H-6, syst. AB, 12.2); 6.95(d, 1H, H-5 or H-6, syst. AB, 12.2).

$^{13}\text{C-NMR}$ (CDCl_3 , δ ppm, $T=298\text{K}$): 165.22(C-11); 161.17(bd, C-12, $^3J(\text{F}^{14}-\text{C}^{12})=5.8$ Hz); 159.46(d, C-14, $J(\text{F}-\text{C}^{14})=252.2$ Hz); 134.36(Cq); 133.19(Cq); 132.98(Cq); 129.71(qd, C-18, $J(3\text{F}-\text{C}^{18})=32.9$ Hz, $J(\text{F}^{14}-$

C^{18})=3.3 Hz); 129.57(Cq); 122.49(qd, CF_3 , $J(3F-C)$ =274.7 Hz, $J(F-CF_3)$ =2.9 Hz); 119.30(dq, C-13, $J(F^{14}-C^{13})$ =23.1 Hz, $J(3F-C^{13})$ =2.9 Hz); 132.00(d, C-16, $J(F^{14}-C^{16})$ =8.6 Hz); 130.55(C-5 or C-6); 130.24(C-6 or C-5); 129.79(CH); 129.43(CH); 129.06(CH); 128.97(CH); 128.95(CH); 128.35(CH); 128.17(C-1); 127.84(CH); 122.06(qd, C-17, $J(3F-C^{17})$ =4.6 Hz, $^4J(F^{14}-C^{17})$ =3.5 Hz); 119.65(d, C-15, $J(F-C^{15})$ =22.2 Hz).

C12 signal is *broadened doublet*. The doublet is due to the coupling with the fluorine at position 14, and the expansion is made by coupling 4 links, with a small constant, between C12 and the fluorine atoms from trifluoromethyl.

The signal from δ = 128.34 ppm is expanded due to the coupling in space with the fluorine atom or atoms in positions 14 and 18. In the previous examples this signal was attributed to C10 and the effect was felt that stems from coupling through space with F representing the proof of this assertion; in support of this statement comes also the Hetcor proof that C10 is linked to H10, which is in turn coupled to F14.

FT-IR (solid in ATR, ν cm^{-1}): 3117w; 3069w; 2981w; 1766vs; 1611m; 1588m; 1482w; 1462m; 1312s; 1261vs; 1234s; 1174vs; 1141s; 1124vs; 1088s; 1056m; 1042m; 975m; 912m; 879w; 843m; 802s; 776m; 765m; 750m; 724m; 695w; 673w.

Compound IX: **O-(2-Fluoro-5-trifluoromethyl-benzoyl)-5-oximino-5H-dibenzo[a,d]cycloheptene**

Elemental analysis: Calculated: C 67.16%, H 3.19%, N 3.41%; Found C 67.32%, H 3.29%, N 3.26%; m.p. 163-164 °C; yield 82%.

1H -NMR ($CDCl_3$, δ ppm, J Hz, T=298K): 8.09(dd, 1H, H-18, $J(H^{16}-H^{18})$ = 2.2 Hz, $J(F^{14}-H^{18})$ =6.3 Hz); 7.82(m, 1H, H-1); 7.77(m, 1H, H-16); 7.69(m, 1H, H-10); 7.54÷7.41(m, 6H, H-arom); 7.23(t, 1H, H-15, $J(F^{14}-H^{15})=J(H^{16}-H^{15})$ =9.3 Hz); 7.01(d, 1H, H-6 or H-5, syst. AB, 12.6); 6.97(d, 1H, H-5 or H-6, syst. AB, 12.6).

^{13}C -NMR ($CDCl_3$, δ ppm, T=298K): 165.22(C-11); 163.49(dq, C-14, $J(F^{14}-C^{14})$ =267.2 Hz, $J(3F-C^{14})$ =1.4 Hz); 160.23(d, C-12, $J(F^{14}-C^{12})$ =4.1 Hz); 134.37(Cq); 133.22(Cq); 133.10(Cq); 131.77(dq, C-16, $^3J(F^{14}-C^{16})$ =10.1 Hz, $^3J(3F-C^{16})$ =3.5 Hz); 130.72(C-5 or C-6); 130.19(C-5 or C-6); 129.85(CH); 129.71(Cq); 129.57(CH); 129.19(CH); 129.04(CH); 128.99(CH); 128.63(d, C-10, $J(F^{14}-C^{10})$ =1.7 Hz); 128.22(CH); 127.71(CH); 126.92(qd, C-17, $J(3F-C^{17})$ = 34.1 Hz, $^4J(F^{14}-C^{17})$ =4.0 Hz); 123.11(q, CF_3 , $J(3F-C)$ = 272.5 Hz); 118.14(d, C-13, $J(F^{14}-C^{13})$ =11.7 Hz); 118.01(d, C-15, $J(F^{14}-C^{15})$ =23.6 Hz).

FT-IR (solid in ATR, ν cm^{-1}): 3069w; 3027w; 1762vs; 1616m; 1595m; 1501m; 1336vs; 1272m; 1245m; 1212vs; 1172m; 1108vs; 1079vs;

1037vs; 978s; 916w; 896s; 873m; 844m; 792m; 765vs; 739w; 714w; 678w; 617m.

Compound X: **O-(4-Fluoro-2-trifluoromethyl-benzoyl)-5-oximino-5H-dibenzo[a,d]cycloheptene**

Elemental analysis: Calculated: C 67.16%, H 3.19%, N 3.41%; Found C 67.02%, H 3.42%, N 3.28%; m.p. 153-154 °C; yield 82.5%.

¹H-NMR (CDCl₃, δ ppm, J Hz, T=298K): 7.83(m, 1H, H-1); 7.65(dd, 1H, H-18, $J(\text{F}^{16}\text{-H}^{18})=5.8$ Hz, $J(\text{H}^{17}\text{-H}^{18})=8.4$ Hz); 7.55÷7.35(m, 7H, H-arom); 7.40(m, 1H, H-15); 7.23(td, 1H, H-17, $J(\text{H}^{15}\text{-H}^{17})=2.7$ Hz, 8.4); 7.01(d, 1H, H-6 or H-5, syst. AB, 12.1); 6.96(d, 1H, H-5 or H-6, syst. AB, 12.1).

¹³C-NMR (CDCl₃, δ ppm, T=298K): 165.36(C-11); 162.91(C-12); 163.63(d, C-16, $J(\text{F}^{16}\text{-C}^{16})=254.6$ Hz); 134.31(Cq); 133.26(Cq); 133.12(Cq); 133.12(d, C-18, $J(\text{F}^{16}\text{-C}^{18})=8.8$ Hz); 131.85(qd, C-14, $J(3\text{F-C}^{14})=34.2$ Hz, $^3J(\text{F}^{16}\text{-C}^{14})=8.3$ Hz); 130.73(C-5 or C-6); 130.12(C-5 or C-6); 129.81(CH); 129.73(Cq); 129.46(CH); 129.17(CH); 129.02(C-1); 129.00(CH); 128.14(CH); 128.12(CH); 127.75(CH); 125.54(C-13); 122.19(qd, CF₃, $J(3\text{F-C})=274.0$ Hz, $^4J(\text{F}^{16}\text{-CF}_3)=2.5$ Hz); 118.71(d, C-17, $J(\text{F}^{16}\text{-C}^{17})=21.1$ Hz); 114.95(dq, C-15, $J(\text{F}^{16}\text{-C}^{15})=25.4$ Hz, $J(3\text{F-C}^{15})=5.5$ Hz).

FT-IR (solid in ATR, ν cm⁻¹): 3080w; 3060w; 3025w; 2973w; 1764vs; 1591m; 1494w; 1418m; 1291m; 1266m; 1232s; 1168s; 1148vs; 1130s; 1078m; 1034s; 972m; 894s; 849m; 801m; 773s; 765m; 727w; 699m.

Conclusions

We have synthesized a series of new acyl-oximines, compounds with dibenzocycloheptatriene structure, by acylation of 5-oximino-5H-dibenzo[a,d]cycloheptene with various aromatic carboxylic acid chlorides. The new compounds were characterized by elemental analysis and spectrometric methods (IR, ¹H-NMR and ¹³C-NMR).

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