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Synthesis of a Novel Tetracyclic Heterocycle : 1-carboxy-13-oxo-[1,2,3]triazolo [1',5':4,3] [1,4]diazepino [6,7-c]chromane, Using Salicylaldehyde as a Starting Material

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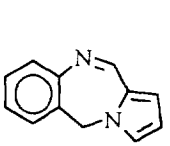
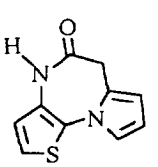
Abstract : A nine-step synthesis of the potentially biologically active title compound, **9**, has been achieved starting from salicylaldehyde and involving two 1,3-dipolar cycloaddition reactions and a lactamisation reaction. The cis configuration of compound **9** resulted from that of compound **4** which was confirmed by X-ray crystallography.

A large number of tricyclic and tetracyclic heterocycles have received much attention due to their biological activity. Structures such as dibenzo[b,e][1,4] diazepine were reported to have significant gastrointestinal activity [1-3]. Fused tricyclic systems involving [1,4]-diazepinic, pyrrolic and benzenic cycles such as **A**[4], [1,4]-diazepinic, thiophenic and benzenic[5], or [1,4]-diazepinic, thiophenic and pyrrolic cycles such as **B** [6], [1,2,3]-triazolo[1,5-a] benzo[1,5] diazepinone C[7] were reported to exhibit interesting CNS activities. The synthesis of fused tetracyclic structures containing benzodiazepine imidazole and pyrrole heterocycles **D**, **E**, has captured the attention of chemists because of their pharmacological activities [8-10].These compounds are reported to display antigastric activity [11] and antiallergic properties [12-15].



A



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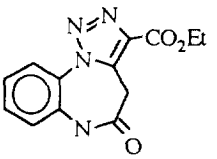

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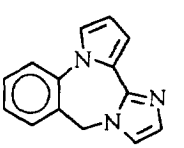
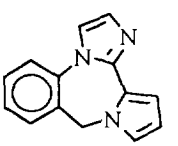
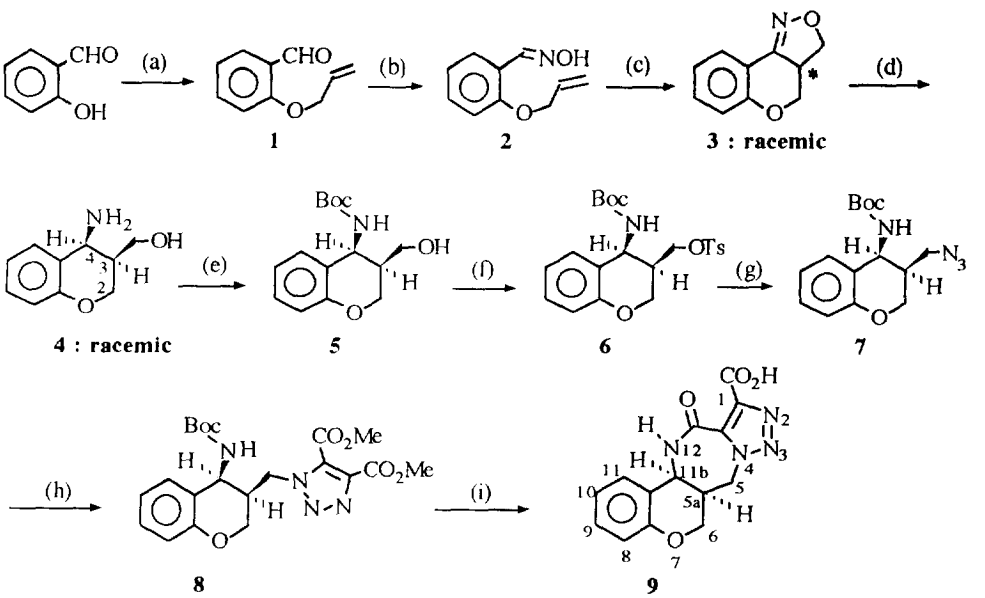


D



E

On the basis of these reports, and as a continuation of our program directed to the preparation of fused ring species derived from simple starting material, we describe here the synthesis of 1-carboxy-13-oxo-[1,2,3] triazolo [1',5': 4,3] [1,4] diazepino [6,7-c] chromane **9**, in order to verify its potential pharmacological activity. Such a structure may constitute an important building block for the construction of a wide variety of bio-active compounds. It seemed to us unlikely to synthesize compound **9**, a tetracyclic heterocycle by following the classical pathway by a fusion between triazolodiazepine and chromane at its 3,4 side, so we designed an original reaction pathway, where salicylaldehyde served as the starting material. (Scheme 1). Commercially available salicylaldehyde, was O-alkylated with allylbromide to give allylether **1**. Oximation of **1** followed by treatment of **2** with sodium hypochlorite led to a 1,3-dipolar intramolecular cycloaddition involving nitriloxide dipôle formed "in situ" and the double bond of the allyl group [16-18] .



(a) : H_2CCHCH_2Br/ K_2CO_3/ acetone (83%); (b) : H_2NOH,HCl/ NaOH/ H_2O, (87%). (c) : NaOCl/ $CHCl_3$ (83%). (d) : $LiAlH_4$/ Et_2O (95%). (e) : $(Boc)_2O$/ dioxane-H_2O (2:1) (85%). (f) : TsCl/ pyridine (90%). (g) : NaN_3/ DMF (87%). (h) : Dimethyl acetylendicarboxylate (86%). (i) : 4N HCl (95%)

Reductive opening of isoxazoline heterocycle **3** with lithium-aluminium hydride in ether at room temperature[19] afforded amino-alcohol **4** . The proton coupling constant value (J_{H3-H4} = 4.4Hz) is in agreement with a cis configuration; it is close to that reported by Lövgren[20] for the cis configuration of 3-hydroxy-4-isopropylaminochromane (J_{H3-H4} =4.0Hz), as compared to the trans isomer (J_{H3-H4} = 3.5 Hz). The average values of ^3J coupling constants in compound **4** (J_{H2A-H3} = 4.8Hz, J_{H2B-H3} = 6.0Hz, J_{H3-H4} = 4.4Hz) showed that the hetero ring probably adopted a half-chair conformation. This result is in agreement with that of Bolger and his coworkers [21,22] who found a half-chair conformation for a number of substituted flavans and also with that of Cotterill et al.[23] who studied the stereochemistry of 3,4-disubstituted chromans by ^1H nmr spectroscopy. Structure and configuration of compound **4** was further confirmed by X-ray Diffraction. The molecular geometry of **4** is depicted in figure 1.

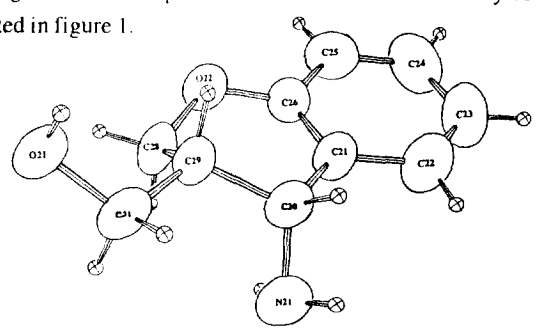


Figure 1 : ORTEP Representation of Compound 4

The perspective view of this molecule corroborates the structure inferred from the spectroscopic evidence. In this structure, the pyran ring had a slightly distorted half-chair form with H_{2A} and H_4 lying in a closely planar orientation. No hydrogen bonding between the amino and hydroxyl group existed, the hydroxyl group being far away from the amino group. Protection of the amino group of **4** by the tert-butoxycarbonyl group (Boc), to get compound **5** , allowed the tosylation and subsequent to the substitution by an azide [24] , **7**. Thus, the stereoselective preparation of amino azide **7** was accomplished in seven steps. 1,3-dipolar cycloaddition involving compound **7** as 1,3-dipole and dimethyl acetylenedicarboxylate as dipolarophile led to triazolic derivative **8**. The structures of compounds **5**, **6**, **7** and **8** were established by means of ^1H and ^{13}C nmr spectroscopy, mass spectrometry and elemental analysis.

As all the reactions involved in this work to obtain compounds **5**, **6**, **7** and **8** could not affect the configuration of the two asymmetric centers, we believed that these compounds maintained the same cis configuration. However, they did not maintain exactely the same half-chair conformation because of the different values of $^3J_{H2\text{-}H3}$ (**5** : $J_{H2A\text{-}H3}$ = 11.5Hz, $J_{H2B\text{-}H3}$ = 3.2Hz; **7** : $J_{H2A\text{-}H3}$ = 9.2Hz, $J_{H2B\text{-}H3}$ = 1.8Hz; **8** : $J_{H2A\text{-}H3}$ = 9.6Hz, $J_{H2B\text{-}H3}$ = 4.3Hz). In ^1H nmr spectrum of compound **5** we noted a deshielded signal due to the hydroxyl proton (δ : 4.33ppm, dd, $J_{H\alpha A\text{-}OH}$ = 3Hz, $J_{H\alpha B\text{-}OH}$ = 9Hz); in comparison with the same proton signal of compound **4** (δ : 3.01ppm, s); (all the NMR spectrums were taken in the same conditions: the same concentration in $CDCl_3$ at room temperature) . The hydroxylic proton in **5** is probably involved in hydrogen bonding interaction with the nitrogen atom and is coupled with adjacent protons $H_{\alpha A}$ and $H_{\alpha B}$. In the infra-red spectrum, the sharp stretching band between 3600 and 3400 cm^{-1} due to the O-H group was unaffected by dilution; this corroborated the presence of an intramolecular hydrogen bonding in compound **5**. The nitrogen proton is coupled with H_4 ($J_{H4\text{-}NH}$ ~ 8Hz) in the four compounds, **5**, **6**, **7** and **8**; these constant values gave a dihedral angle H_4-C_4-N-H of about 20°, this could be explained by a position of the tert-butoxycarbonyl group where the N-Boc bond adopted an opposite position to the C_3-C_4 bond that avoided steric hindrance with the α-methylene substituent.

Removal of the protecting group in acidic medium and concomitant lactamisation of the aminoester provided 1-carboxy-13-oxo-[1,2,3] triazolo [1',5':4,3] [1,4] diazepine [6,7-c] chromane **9** in 31% overall yield. Infra-red, NMR, mass spectrometry and elemental analysis of compound **9** are in agreement with its structure.This compound has now been submitted for biological tests to evaluate its activity.

Our synthesis of **7** is concise (7 steps) and efficient (38% overall yield) enough to encourage us in the synthesis of other fused tetracyclic heterocycles. Employing disubstituted chromane **7** as the key intermediate, we are carrying out short syntheses of a variety of tetracyclic heterocycles, using mono and disubstituted acetylenes. The results are encouraging and will be published later.

EXPERIMENTAL

Melting points were measured in capillary tube using a Büchi 510 apparatus, and are uncorrected. Infra-red spectra were recorded as potassium bromide pellets on a Perkin-Elmer 1760 X spectrophotometer. ^1H-nmr spectra were obtained on a Bruker AC 250 and AM 300 spectrometers with tetramethylsilane as an internal standard. High resolution mass spectra were performed by direct ionisation (EI at 70 eV) on a Hewlett Packard 5989 A apparatus. Elemental analysis were performed at the Centre de Microanalyse du CNRS at Lyon (France). The progress of the reactions was monitored by tlc and the purity of each compound was checked by

analytical thin-layer chromatography (tlc) on silica gel plates with F-254 indicator, visualisation was accomplished by uv light and iodine.

2-allyloxybenzaldehyde 1

A solution of salicylaldehyde (22g, 0.18 mole), allylbromide (22.4g, 0.185 mole) and potassium carbonate (22g, 0.217 mole) in dry acetone (40 ml) was refluxed for 3 hours. At the completion of the reaction, the resulting mixture was cooled to room temperature and poured into water (200 ml). The aqueous phase was extracted with ether (200 ml) (or dichloromethane) and the extract washed with 5% NaOH solution and water. The organic phase was dried over $CaCl_2$ and concentrated under reduced pressure. The crude product was distilled under reduced pressure to get compound **1** (18.26g, 83%); b.p. = 113°C/12mm; ^1H nmr ($CDCl_3$) δ ppm 4.73 (dd, 2H, J: 5.0, 1.6 Hz), 5.14-5.40 (m,2H), δ 5.40-5.90 (m,1H), 6.70-8.00 (m,4H), 10.62 (s,1H); ms, m/z: 162 ($M^{+\cdot}$).

2-allyloxybenzaldoxime 2

A solution of sodium hydroxide (14g, 0.35 mole) in water (40 ml) was cooled to 0°C and compound **1** (18g, 0.11 mole) was slowly added under stirring. Hydroxylamine hydrochloride (15.18g, 0.22 mole) was slowly added to the stirred mixture. After the addition, the mixture was allowed to warm to room temperature. After standing one hour, it was cooled in an ice bath. The crystals which deposited were filtered and recrystallized from hexane to yield 15.6g (87%) of yellow crystals of 2-allyloxybenzaldoxime **2**. m.p. = 37°C; ir (KBr), ν cm^{-1}: 1610, 3260; ^1H nmr ($CDCl_3$): δppm 4.56 (dd, J: 5.0, 1.6Hz, 2H), 5.15-5.55 (m,2H), 5.80-6.31 (m,1H), 6.80-7.80 (m,4H), 9.01 (s,1H); ms, m/z: 177 (M^+); (found: C,67.57; H,6.23. Calcd. for $C_{10}H_{11}NO_2$: C, 67.79; H, 6.21%).

Δ^1-isoxazolino[3,4-c]chromane 3

Aldoxime **2** (15.5g, 0.088 mole) was dissolved by magnetical stirring in chloroform (150 ml). A commercial solution of sodium hypochlorite (150 ml) was added dropwise over a period of 45 min. During the addition the temperature was maintained at -5°C to -10°C. Upon completion of the reaction as determined by tlc, the mixture was allowed to warm to room temperature under stirring during 15 to 20 min. The mixture was washed several times with water until the aqueous washing were at a neutral pH; the organic phase was then dried over sodium sulfate, evaporated to dryness. The crude product was recrystallized from water-ethanol (60-40) to yield 12.86g (83%) of white crystals of **3**. m.p. = 63°C; ir (KBr), ν cm^{-1}: 1625; ^1H nmr ($CDCl_3$): δ ppm 3.75-4.90 (m,5H), 6.80-7.40 (m,3H), 7.60-7.80 (m,1H); ^{13}C nmr ($CDCl_3$): δ ppm 70.63 (C_3), 45.62 (C_{3a}), 69.25 (C_4), 155.58 (C_{5a}), 117.40 (C_6), 132.45 (C_7), 121.60 (C_8), 125.60 (C_9), 113.04 (C_{9a}), 152.76 (C_{9b}); ms, m/z: 175 ($M^{+\cdot}$); (found.: C,68.56; H,5.19. Calcd. for $C_{10}H_9NO_2$: C, 68.63; H,5.14%).

4-amino-3-hydroxymethylchromane 4

A mixture of lithium aluminium hydride (3.7g, 0.97 mole) and dry ether (250 ml) was cooled to 0°C in a two necked flask under an inert atmosphere. An etheral solution (150 ml) of **3** (12.8g, 0.073 mole) was added dropwise through an isobar dropping funnel over a period of about 1 hour. At the end of the addition, the mixture was stirred at room temperature for 8 hours. The resulting solution was hydrolyzed and the etheral phase separated, dried over sodium sulfate and evaporated to dryness. The crude product was recrystallized

from ether and yielded 12.16g (95%) of colorless crystals of **4**. m.p. = 71°C; ^{1}H nmr ($CDCl_3$): δ ppm 2.12 (m,1H), 3.01 (s,3H), 3.81 (ddd, 2H; J: 11.3, 4.8, 6.0 Hz), 4.06 (d, 1H; J: 4.4 Hz), 4.11-4.20 (m,2H), 6.75-7.20 (m,4H); ^{13}C nmr ($CDCl_3$): δ ppm 38.88(C_3), 47.82 (C_4), 61.58 (Cα), 63.77 (C_2), 126.28 (C_{4a}), 153.78 (C_{8a}); ms, m/z: 179 ($M^{+\cdot}$); (found. : C,67.13; H,7.17. Calcd. for $C_{10}H_{13}NO_2$: C,67.04; H,7.26%).

3-hydroxymethyl-4-N-tertiobutoxycarbonylaminochromane 5

To a solution of aminoalcohol **4** (12.1g, 0.067 mole) in dioxane-water (2-1) cooled to 0°C, was added di-tert-butyldicarbonate (16.75g, 0.083 mole).The solution was stirred at 25°C for 1 hour. After concentration under vacuum, the residue was dissolved in ether and washed with water. The organic phase was dried over sodium sulfate and concentrated to dryness. The crude product was recrystallized from hexane-ether (8-2) to yield 10.28g (85%) of white crystals of **5**. m.p. = 137°C; ^{1}H nmr ($CDCl_3$): δ ppm 1.46 (s,9H), 2.38 (m,1H), 3.35-3.60 (m,2H; J: 12.0, 5.4 Hz), 3.65 (t,1H; J: 11.5 Hz), 4.00 (ddd, 1H; J: 11.5, 3.2, 2.4 Hz), 4.33 (dd, 1H; J: 9.0, 3.0 Hz), 4.80-5.10 (m,2H), 6.78-7.26 (m,4H); ^{13}C nmr ($CDCl_3$): δ ppm 28.31 (Boc), 40.70 (C_3), 45.71(C_4), 59.43(Cα), 63.05 (C_2), 80.68 [C (Boc)], 154.58 (C_{8a}), 156.70 [C=O(Boc)]; ms, m/z: 279 ($M^{+\cdot}$); (found.: C,64.60; H,7.59. Calcd. for $C_{15}H_{21}NO_4$: C,64.52; H,7.53%).

3-methyltosylate-4-N-tertiobutoxycarbonylaminochromane 6

A mixture of protected aminoalcohol **5** (10.2g, 0.036 mole) and pyridine (15 ml) was stirred until complete dissolution, then cooled to -5°C. Tosylchloride (7.23g, 0.038 mole) was slowly added. The mixture was stirred at room temperature for 2 hours. At the end of the reaction, a water acidified was added to the reaction mixture until neutral pH and the solution extracted with dichloromethane. The organic extract was concentrated to dryness and the crude product was recrystallized from ethanol to yield 9.18g (90%) of compound **6**. m.p. = 160°C; ^{1}H nmr ($CDCl_3$): δ ppm 1.43 (s,9H), 2.43 (s,3H), 2.51 (m,1H), 3.90 (m,2H),4.30 (m,2H), 4.69 (dd,1H; J: 8.2, 3.8 Hz), 4.90 (d,1H; J: 8.2 Hz), 6.73-7.76 (m,8H); ^{13}C nmr ($CDCl_3$): δ ppm 21.60 [CH_3(Ts)], 28.23 [CH_3(Boc)], 36.98 (C_3), 46.19 (C_4), 67.72 (C_2), 63.75 (C_2), 80.15 [C(Boc)], 121.10 (C4a), 154.14 (C_{8a}), 155.29 [C=O(Boc)], 144.96 (C_{SO2}); ms, m/z: 433 ($M^{+\cdot}$); (found.: C,60.83; H,6.27. Calcd. for $C_{22}H_{27}NO_6S$: C,60.97; H,6.27%).

3-methylazide-4-N-tert-butoxycarbonylaminochromane 7

Sodium azide (2.96, 0.045 mole) was added under magnetic stirring to a solution of compound **6** (9.7g, 0.021 mole) in dimethylformamide (250 ml). The mixture was heated to 70°C in a water bath for 2 hours, filtered and water (300 ml) was added to the filtrate. A white precipitate appeared. It was recovered by filtration and recrystallized from ethanol to yield 7.92g (87%) of compound **7**. m.p. = 101°C; ^{1}H nmr ($CDCl_3$): δ ppm 1.45 (s,9H), 2.40 (m,1H), 3.24 (dd, 1H; J: 12.5, 9.3 Hz), 3.65 (dd, 1H; J: 5.0, Hz), 3.91 (dd, 1H; J: 11.2, 9.2 Hz), 4.25 (dd,1H; J: 9.2, 1.8 Hz), 4.80 (d,1H; J:8.1 Hz), 4.95 (dd, 1H; J: 4.0, 8.1 Hz), 6.79-7.23 (m,4H); ^{13}C nmr ($CDCl_3$): δ ppm 28.37 (CH_3 Boc), 37.23 (C_3), 46.83 (C_4), 49.31 (Cα), 64.61 (C_2), 80.17 (C Boc), 121.63 (C_{4a}), 154.23 (C_{8a}), 155.47 (C=O); ms, m/z: 304 ($M^{+\cdot}$); (found.: C, 59.09; H, 6.67. Calcd. for $C_{15}H_{20}N_4O_3$: C, 59.21; H, 6.58%).

3-(4',5'-dimethylcarboxylate[1,2,3]triazolylmethyl)-4-N-tert-butoxycarbonylaminochromane 8

A mixture of azide **7** (7.90g, 0.026 mole) and dimethyl acetylenedicarboxylate (5.20g, 0.036 mole) was stirred at room temperature for 2 hours. The reaction mixture was filtered and the product was recrystallized from methanol to yield 7.50g (95%) of compound **8**. m.p. = 170°C; ^{1}H nmr ($CDCl_3$): δ ppm 1.48 (s,9H), 2.87 (m,1H), 3.98 and 3.99 (2s,6H), 3.92-4.14 (m,2H), 4.58 (dd,1H; J: 14.1, 4.3 Hz), 5.13 (dd,1H; J: 4.3, 8.3 Hz), 6.79-7.28 (m,4H); ^{13}C nmr δ ppm 28.26 (CH_3 Boc), 37.93 (C_2), 47.16 (C_4), 48.51 (Cα), 52.69 and 53.46 ($2CH_3O$), 64.21 (C_2), 80.32 (C Boc), 130.14 ($C_{5'}$), 140.17 ($C_{4'}$), 153.95 (C_{8a}), 155.45 (C=O Boc), 158.72 and 160.34 (C=O ester); ms, m/z: 446 ($M^{+\cdot}$); (found. C, 56.69; H, 5.71. Calcd. for $C_{21}H_{26}N_4O_7$: C, 56.50; H, 5.83%).

1-carboxy-13-oxo[1,2,3]triazolo[1',5':4,3][1,4]diazepino[6,7-c]chromane 9

A mixture of compound **8** (7.4g, 0.0166 mole) and 4N HCl(100 ml) was stirred under reflux for 30 min. Compound **8** was completely dissolved after 10 min. and a new precipitate was formed during the 10 following min. corresponding to compond **9**. The crude product obtained was recrystallized from DMSO-H_2O (1-1) to get **9** as a white powder 7.0g (95%). m.p. > 260°C; ^{1}H nmr ($DMSOd_6$): δ ppm 2.94 (m,1H°, 4.18-4.23 (m, 2H), 4.58 (d,1H; J: 4.0 Hz), 4.94 (ddd, 2H; J: 13.9, 7.5,6.8 Hz), 6.86-7.38 (m, 4H), 8.48 (br s, 2H); ^{13}C nmr ($DMSOd_6$) δ ppm 36.42 (C_{5a}), 45.73 (C_{11b}), 46.05 (C_5), 62.33 (C_6), 117.52 (C_{11a}), 133.23 (C_1), 140.43 (C_{13a}), 153.72 (C_{7a}), 159.60 (CO_2H), 160.60 (C_{13}); ms, m/z: 300 ($M^{+\cdot}$); (found. C, 55.73; H, 4.23. Calcd. for $C_{14}H_{12}N_4O_4$: C, 56.00; H, 4.00%).

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