

**A convenient synthesis of heterocyclic compounds containing
11-oxo-6,11,12,13-tetrahydrodibenzo[*b,g*][1,5]oxazonine fragment**

Victor V. Potapov, Nataliya A. Fetisova, Alexandr V. Nikitin and Alexandre V. Ivachtchenko*

Thin-layer chromatography (TLC) was performed on aluminum sheets precoated with silica gel (Merck, Kieselgel 60 F-254). ¹H NMR spectra were recorded on a Bruker AMX-400 or Varian spectrometer in DMSO-*d*₆ (300 MHz, DMSO-*d*₆+CCl₄, chemical shifts in ppm). High resolution mass-spectra have been recorded using electrospray ionization time-of-flight reflectron experiments (ESI-TOF) on Agilent ESI-TOF mass spectrometer. Samples were electrosprayed into the TOF reflectron analyzer at an ESI voltage of 4000 V and a flow rate of 200 microliters per minute. All solvents and reagents were obtained from commercial sources (Acros Organics, Aldrich, or ChemDiv) and used without purification.

Synthesis of bifunctional reagent 4.

2-(Bromomethyl)benzoic acid methyl ester **1** (0.1 mol) was dissolved in acetonitrile and K₂CO₃ (2 eq.), then salicylaldehyde **2** (1eq.) was added and the reaction mixture was stirred at 80 °C for 5-6 h. On completion, the reaction mixture was cooled to rt and filtered. The solvent was evaporated under reduced pressure. The residue was dissolved in ester, organic layer was washed with water and aq. NaHCO₃ (2%) then the solvent was completely removed *in vacuo*. The obtained residue was used in the following stage without any additional purification. The desired bifunctional aldehyde-ester **3** was obtained in 75% yield.

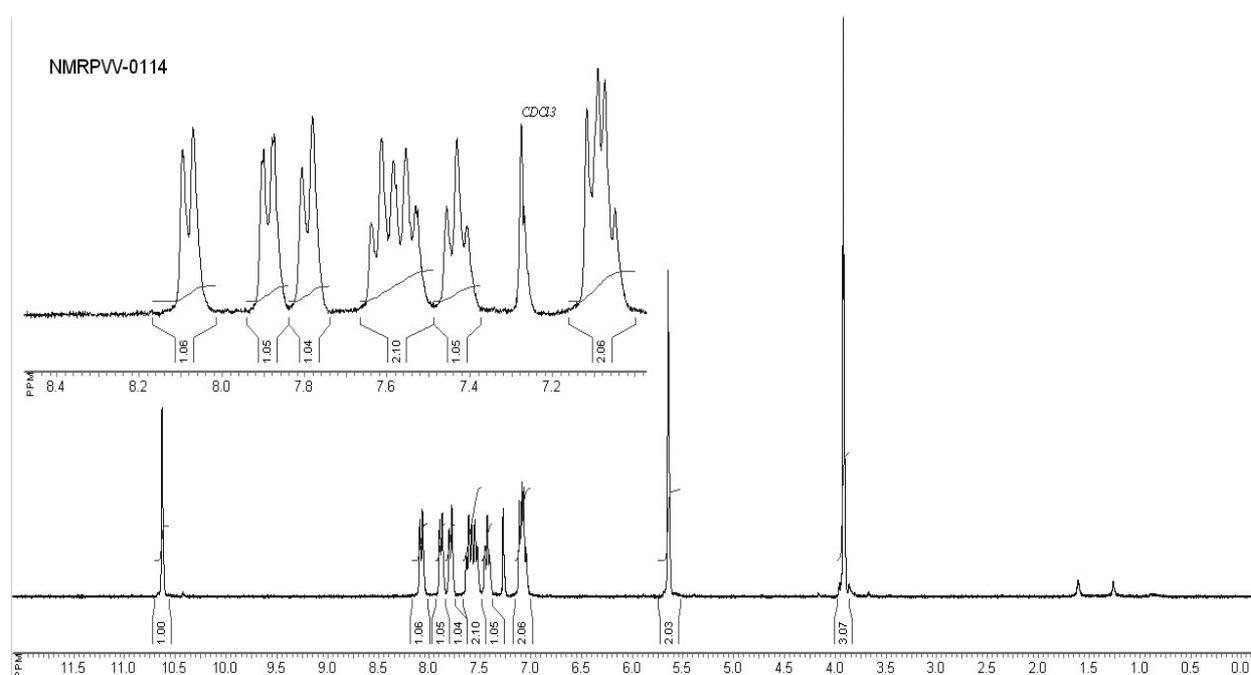
The obtained ester (0.1 mol) was dissolved in MeOH (50%), then KOH (1,2 eq.) was added gradually to the solution. The reaction mixture was stirred at the room temperature for 8-10 h. After the reaction was complete, the solvent was removed *in vacuo*, the obtained residue was dissolved in water. After adding the aq. HCl (5%) the formed precipitate was separated by filtration, washed with water and dried in air. The yield of corresponding aldehyde-acid **4** was approx. 90%.

General procedure for preparation of the target compounds 5a,b.

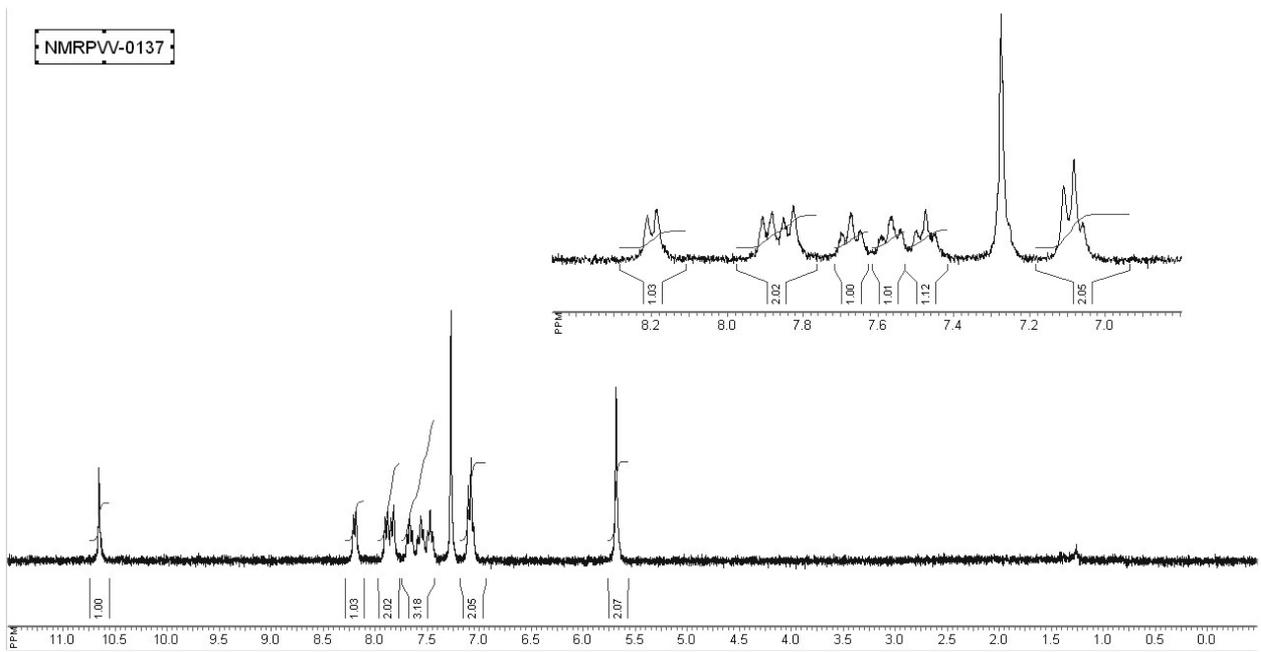
The aldehyde-acid **4** (1.0 mmol) and the corresponding primary amine (1.5 mmol) were dissolved in methanol (3 mL). The solution was kept for 10 min at rt, then the isonitrile (1.5 mmol) was added and the resulting mixture was stirred at 25 °C for 5-10 h. The reaction was followed by TLC (5% MeOH in CH₂Cl₂). On completion, the reaction mixture was cooled to rt, the formed precipitate was filtered off, washed with methanol and, if required, purified by recrystallization from diethyl ether or by chromatography on silica gel, eluting with CH₂Cl₂. The title compounds **5a,b** were obtained as colorless solids in comparatively low yields (28 and 30%, respectively) as the mixture of corresponding enantiomers.

Satisfactory analytical data for compounds synthesized in this work.

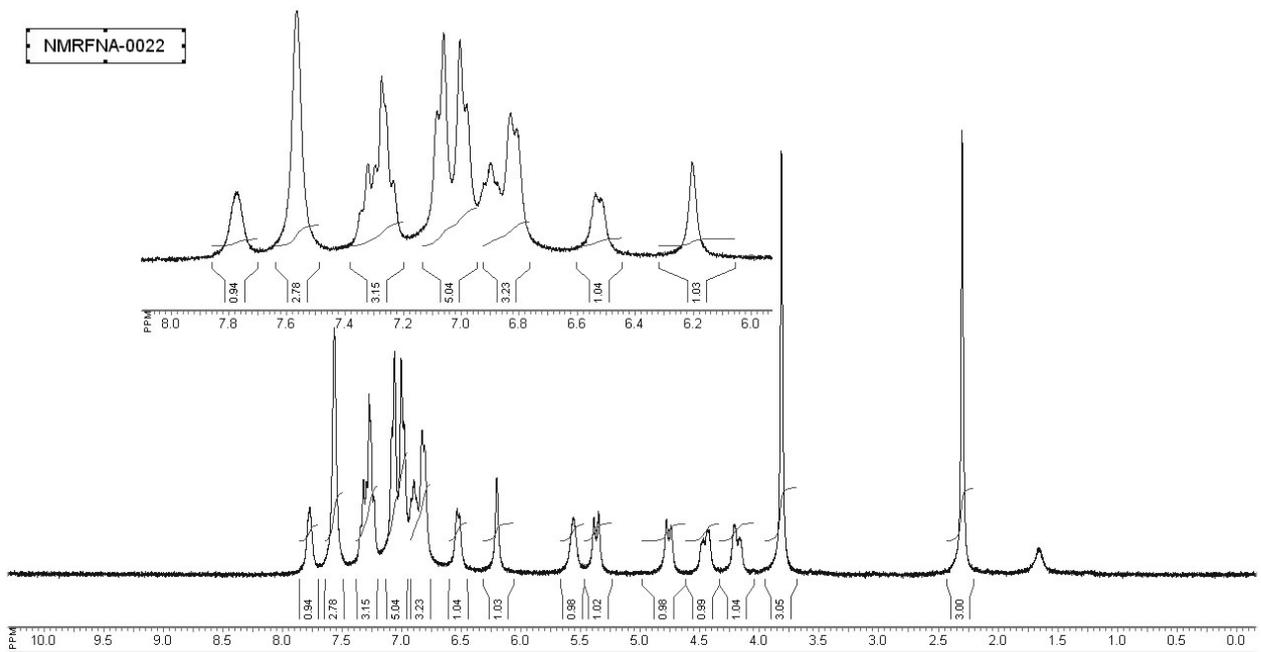
Compound 3:



Compound 4:



Compound 5a:





10550 N. Torrey Pines Road
Mail code: BCC-007
La Jolla, CA 92037
(858) 784-9415
fax: (858) 784-9496

Mass Spectrometry
Request & Analysis Form

Sample ID

C9CW4692

PI: Chemical Diversity Laboratory

Phone: 858-794-4860

Name: caroline williams

Fax: 858-794-4931

Email: cw@chemdiv.com

Requested method: ESI-TOF high-acc

Molecular Formula: C₃₁H₂₈N₂O₄

Mass: 492.2048968 exact: 492.2048968

Scan Range: 100 to 600

Compound type heterocycle Functional groups -ROR, amide

Confidence **confident** Purity **very pure** Toxicity **unknown**

Solution concentration 1 mg/ml Solvent MeOH

Other suitable solvents: H₂O MeOH CHCl₃
Other: _____

Comments + special instructions:

| | Molecular weight | | | |
|------------------|------------------|-------------|-----|-------|
| | expected | observed by | | |
| ESI-TOF high-acc | | MALDI-TOF | ESI | GC/MS |
| MH+ | 493.2122 | 493.2121 | | |
| MNa+ | 515.1941 | | | |
| [M-H]- | 491.1976 | | | |
| [M+Cl]- | 527.1743 | | | |

Exact mass error: 0.1 mmu

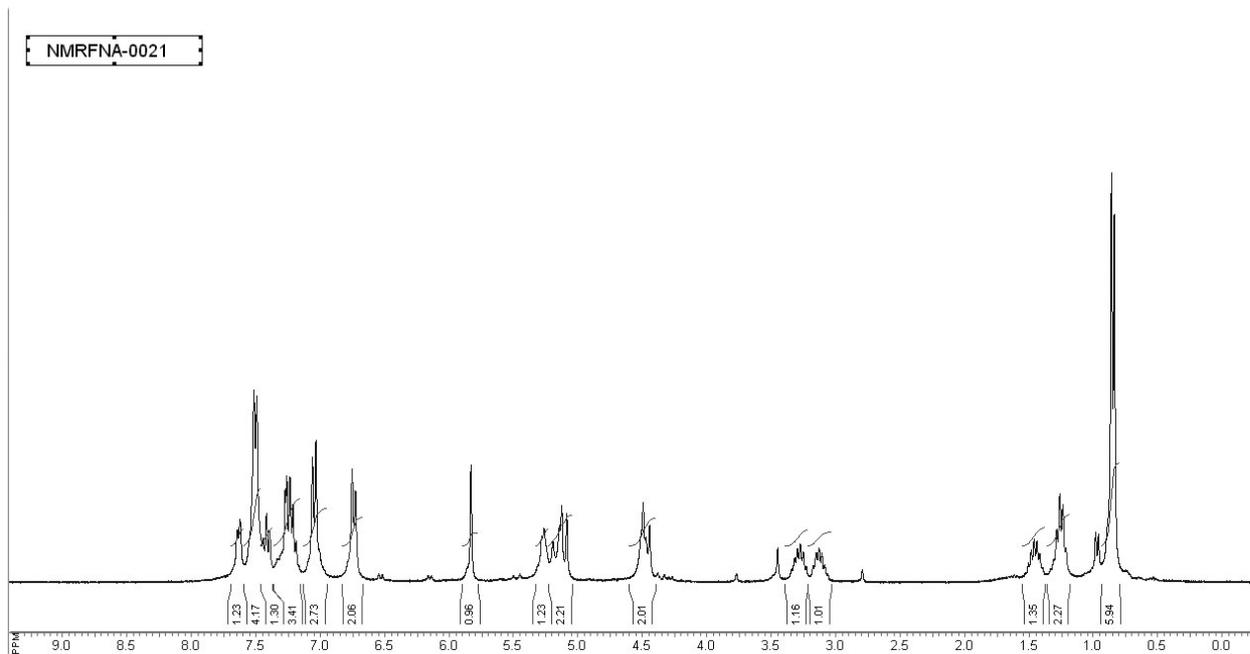
0.2 ppm

Matrix used: DHB α -cyano sinapinic acid THAP Other: _____

Solvent used: MeOH CHCl₃ H₂O Other: _____

Operator: WU

Compound 5b:





THE
SCRIPPS
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INSTITUTE

10550 N. Torrey Pines Road
Mail code: BCC-007
La Jolla, CA 92037
(858) 784-9415
fax: (858) 784-9496

Mass Spectrometry
Request & Analysis Form

Sample ID

C9CW4691

PI: Chemical Diversity Laboratory

Phone: 858-794-4860

Name: caroline williams

Fax: 858-794-4931

Email: cw@chemdiv.com

Requested method: ESI-TOF high-acc

Molecular Formula: C28H29ClN2O3

Mass: 476.1866594 exact: 476.1866594

Scan Range: 100 to 500

Compound type heterocycle Functional groups -Br, Cl, F, -ROR, amide

Confidence confident Purity very pure Toxicity unknown

Solution concentration 1 mg/ml Solvent MeOH

Other suitable solvents: H₂O MeOH CHCl₃
Other: _____

Comments + special instructions:

| | Molecular weight | | | | |
|---------|------------------|------------------|-----------|-----|-------|
| | expected | observed by | | | |
| | | ESI-TOF high-acc | MALDI-TOF | ESI | GC/MS |
| MH+ | 477.1939 | 477.1934 | | | |
| MNa+ | 499.1759 | | | | |
| [M-H]- | 475.1794 | | | | |
| [M+Cl]- | 511.1561 | | | | |
| | | | | | |

Exact mass error: 0.5 mmu

1.0 ppm

Matrix used: DHB α -cyano sinapinic acid THAP Other: _____

Solvent used: MeOH CHCl₃ H₂O Other: _____

Operator: WU

X-Ray crystallographic data for compound 5a:

Empirical formula $C_{31}H_{28}N_2O_4$
Formula weight 492.55
Temperature 120(2) K
Wavelength 0.71073 Å
Crystal system Orthorhombic
Space group Pbcn
Unit cell dimensions $a = 21.819(3)$ Å $\alpha = 90^\circ$
 $b = 9.3072(12)$ Å $\beta = 90^\circ$
 $c = 25.577(3)$ Å $\gamma = 90^\circ$
Volume $5194.2(11)$ Å³
Z 8
Density (calculated) 1.260 Mg/m³
Absorption coefficient 0.084 mm⁻¹
F(000) 2080
Crystal size 0.40 x 0.08 x 0.05 mm³
Theta range for data collection 1.59 to 26.05°
Index ranges $-26 \leq h \leq 26$, $-11 \leq k \leq 11$, $-31 \leq l \leq 30$
Reflections collected 29578
Independent reflections 5089 [R(int) = 0.0816]

| | |
|---|---|
| Completeness to theta = 26.05° | 99.1 % |
| Absorption correction | Semi-empirical from equivalents |
| Max. and min. transmission | 0.993 and 0.969 |
| Refinement method | Full-matrix least-squares on F ² |
| Data / restraints / parameters | 5089 / 0 / 334 |
| Goodness-of-fit on F ² | 1.052 |
| Final R indices [for 3012 rflns with I>2σ(I)] | R1 = 0.0797, wR2 = 0.1770 |
| R indices (all data) | R1 = 0.1330, wR2 = 0.2141 |
| Largest diff. peak and hole | 0.478 and -0.244 e.Å ⁻³ |

Table 1. Atomic coordinates (x 10⁴) and equivalent isotropic displacement parameters (Å² x 10³) for compound **5b**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

| Atom | x | y | z | U(eq) |
|--------|---------|----------|---------|-------|
| C(1) | 1707(1) | -590(4) | 2517(1) | 28(1) |
| C(1A) | 1325(1) | -105(4) | 2118(1) | 26(1) |
| C(2) | 1614(2) | -1915(4) | 2749(1) | 32(1) |
| C(3) | 1134(2) | -2758(4) | 2585(1) | 36(1) |
| C(4) | 747(2) | -2334(4) | 2189(1) | 32(1) |
| C(4A) | 842(1) | -999(4) | 1957(1) | 27(1) |
| O(5) | 478(1) | -597(2) | 1537(1) | 30(1) |
| C(6) | 8(1) | 477(4) | 1644(1) | 29(1) |
| C(6A) | 87(1) | 1727(4) | 1278(1) | 31(1) |
| C(7) | -138(2) | 1606(4) | 769(1) | 39(1) |
| C(8) | -53(2) | 2678(5) | 408(2) | 48(1) |
| C(9) | 260(2) | 3924(5) | 552(2) | 53(1) |
| C(10) | 469(2) | 4097(4) | 1058(2) | 40(1) |
| C(10A) | 387(1) | 2992(4) | 1422(1) | 31(1) |
| C(11) | 598(1) | 3303(3) | 1968(1) | 27(1) |
| O(11) | 381(1) | 4309(3) | 2209(1) | 34(1) |
| N(12) | 1028(1) | 2432(3) | 2181(1) | 22(1) |
| C(13) | 1377(1) | 1354(3) | 1878(1) | 25(1) |
| C(14) | 1196(1) | 2633(3) | 2722(1) | 24(1) |
| C(15) | 910(2) | 1858(4) | 3114(1) | 31(1) |
| C(16) | 1050(2) | 2106(4) | 3634(1) | 37(1) |
| C(17) | 1478(2) | 3141(5) | 3764(1) | 40(1) |
| O(17) | 1647(1) | 3509(3) | 4263(1) | 53(1) |
| C(18) | 1770(2) | 3915(4) | 3368(1) | 37(1) |
| C(19) | 1628(1) | 3671(4) | 2852(1) | 31(1) |
| C(20) | 1354(2) | 2755(6) | 4683(2) | 66(1) |
| C(21) | 2022(1) | 1929(3) | 1739(1) | 21(1) |

| | | | | |
|-------|---------|---------|---------|-------|
| O(21) | 2089(1) | 3236(2) | 1700(1) | 30(1) |
| N(21) | 2454(1) | 981(3) | 1639(1) | 27(1) |
| C(22) | 3056(1) | 1434(4) | 1454(1) | 27(1) |
| C(23) | 3091(1) | 1640(4) | 871(1) | 28(1) |
| C(24) | 2841(2) | 650(4) | 527(2) | 42(1) |
| C(25) | 2883(2) | 852(5) | -8(2) | 53(1) |
| C(26) | 3178(2) | 2042(5) | -219(2) | 46(1) |
| C(27) | 3436(2) | 3010(5) | 125(2) | 45(1) |
| C(28) | 3393(2) | 2813(4) | 661(1) | 36(1) |
| C(29) | 3218(2) | 2258(6) | -806(2) | 65(1) |

Table 2. Bond lengths [Å] and angles [°] for compound **5b**.

| | | | |
|-------------------|----------|--------------------|----------|
| C(1)-C(2) | 1.383(5) | C(13)-C(21) | 1.547(4) |
| C(1)-C(1A) | 1.393(4) | C(14)-C(15) | 1.383(5) |
| C(1A)-C(4A) | 1.404(5) | C(14)-C(19) | 1.392(5) |
| C(1A)-C(13) | 1.495(5) | C(15)-C(16) | 1.384(5) |
| C(2)-C(3) | 1.374(5) | C(16)-C(17) | 1.383(6) |
| C(3)-C(4) | 1.377(5) | C(17)-O(17) | 1.373(4) |
| C(4)-C(4A) | 1.392(5) | C(17)-C(18) | 1.395(5) |
| C(4A)-O(5) | 1.388(4) | O(17)-C(20) | 1.432(5) |
| O(5)-C(6) | 1.459(4) | C(18)-C(19) | 1.375(5) |
| C(6)-C(6A) | 1.503(5) | C(21)-O(21) | 1.229(4) |
| C(6A)-C(7) | 1.395(5) | C(21)-N(21) | 1.316(4) |
| C(6A)-C(10A) | 1.396(5) | N(21)-C(22) | 1.459(4) |
| C(7)-C(8) | 1.373(5) | C(22)-C(23) | 1.504(4) |
| C(8)-C(9) | 1.395(6) | C(23)-C(28) | 1.384(5) |
| C(9)-C(10) | 1.382(5) | C(23)-C(24) | 1.386(5) |
| C(10)-C(10A) | 1.400(5) | C(24)-C(25) | 1.385(5) |
| C(10A)-C(11) | 1.497(5) | C(25)-C(26) | 1.390(6) |
| C(11)-O(11) | 1.218(4) | C(26)-C(27) | 1.380(6) |
| C(11)-N(12) | 1.355(4) | C(26)-C(29) | 1.516(5) |
| N(12)-C(14) | 1.442(4) | C(27)-C(28) | 1.387(5) |
| N(12)-C(13) | 1.479(4) | | |
| C(2)-C(1)-C(1A) | 121.0(3) | C(4A)-O(5)-C(6) | 116.2(2) |
| C(1)-C(1A)-C(4A) | 118.1(3) | O(5)-C(6)-C(6A) | 109.5(3) |
| C(1)-C(1A)-C(13) | 123.3(3) | C(7)-C(6A)-C(10A) | 118.7(3) |
| C(4A)-C(1A)-C(13) | 118.4(3) | C(7)-C(6A)-C(6) | 118.5(3) |
| C(3)-C(2)-C(1) | 119.3(3) | C(10A)-C(6A)-C(6) | 122.8(3) |
| C(2)-C(3)-C(4) | 121.9(3) | C(8)-C(7)-C(6A) | 121.4(4) |
| C(3)-C(4)-C(4A) | 118.6(3) | C(7)-C(8)-C(9) | 119.6(4) |
| O(5)-C(4A)-C(4) | 119.0(3) | C(10)-C(9)-C(8) | 120.4(4) |
| O(5)-C(4A)-C(1A) | 119.8(3) | C(9)-C(10)-C(10A) | 119.8(4) |
| C(4)-C(4A)-C(1A) | 121.1(3) | C(6A)-C(10A)-C(10) | 120.2(3) |

| | |
|--------------------|----------|
| C(6A)-C(10A)-C(11) | 123.6(3) |
| C(10)-C(10A)-C(11) | 116.1(3) |
| O(11)-C(11)-N(12) | 121.7(3) |
| O(11)-C(11)-C(10A) | 120.0(3) |
| N(12)-C(11)-C(10A) | 118.2(3) |
| C(11)-N(12)-C(14) | 119.0(3) |
| C(11)-N(12)-C(13) | 123.4(3) |
| C(14)-N(12)-C(13) | 117.4(2) |
| N(12)-C(13)-C(1A) | 111.2(2) |
| N(12)-C(13)-C(21) | 110.8(3) |
| C(1A)-C(13)-C(21) | 118.6(3) |
| C(15)-C(14)-C(19) | 119.6(3) |
| C(15)-C(14)-N(12) | 120.9(3) |
| C(19)-C(14)-N(12) | 119.5(3) |
| C(14)-C(15)-C(16) | 120.7(3) |
| C(17)-C(16)-C(15) | 119.7(3) |
| O(17)-C(17)-C(16) | 125.3(3) |
| O(17)-C(17)-C(18) | 115.0(4) |
| C(16)-C(17)-C(18) | 119.6(3) |
| C(17)-O(17)-C(20) | 117.1(3) |
| C(19)-C(18)-C(17) | 120.5(3) |
| C(18)-C(19)-C(14) | 119.8(3) |
| O(21)-C(21)-N(21) | 124.2(3) |
| O(21)-C(21)-C(13) | 118.0(3) |
| N(21)-C(21)-C(13) | 117.6(3) |
| C(21)-N(21)-C(22) | 120.9(3) |
| N(21)-C(22)-C(23) | 113.8(3) |
| C(28)-C(23)-C(24) | 117.7(3) |
| C(28)-C(23)-C(22) | 120.6(3) |
| C(24)-C(23)-C(22) | 121.6(3) |
| C(25)-C(24)-C(23) | 120.8(4) |
| C(24)-C(25)-C(26) | 121.5(4) |
| C(27)-C(26)-C(25) | 117.5(4) |
| C(27)-C(26)-C(29) | 121.4(4) |
| C(25)-C(26)-C(29) | 121.1(4) |
| C(26)-C(27)-C(28) | 121.1(4) |
| C(23)-C(28)-C(27) | 121.4(4) |

Table 3. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for compound **5b**. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

| Atom | U ¹¹ | U ²² | U ³³ | U ²³ | U ¹³ | U ¹² |
|--------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| C(1) | 22(2) | 29(2) | 34(2) | -2(2) | 2(1) | 2(1) |
| C(1A) | 23(2) | 25(2) | 30(2) | -1(1) | 4(1) | 1(1) |
| C(2) | 31(2) | 36(2) | 30(2) | 5(2) | 5(1) | 4(2) |
| C(3) | 30(2) | 37(2) | 41(2) | 10(2) | 15(2) | 3(2) |
| C(4) | 23(2) | 28(2) | 46(2) | 2(2) | 12(2) | -2(1) |
| C(4A) | 23(2) | 28(2) | 32(2) | -1(2) | 2(1) | 2(1) |
| O(5) | 23(1) | 29(1) | 38(1) | -4(1) | -1(1) | 3(1) |
| C(6) | 21(2) | 29(2) | 36(2) | -4(2) | 1(1) | -1(1) |
| C(6A) | 25(2) | 34(2) | 34(2) | 0(2) | 1(1) | 6(2) |
| C(7) | 33(2) | 46(2) | 39(2) | 0(2) | -8(2) | -1(2) |
| C(8) | 43(2) | 69(3) | 32(2) | 11(2) | -13(2) | -3(2) |
| C(9) | 52(2) | 61(3) | 45(2) | 26(2) | -14(2) | -5(2) |
| C(10) | 35(2) | 40(2) | 46(2) | 15(2) | -8(2) | -2(2) |
| C(10A) | 20(2) | 35(2) | 37(2) | 4(2) | -2(1) | 4(1) |
| C(11) | 23(2) | 21(2) | 36(2) | 3(2) | 3(1) | 2(1) |
| O(11) | 30(1) | 27(1) | 45(2) | -1(1) | 0(1) | 4(1) |
| N(12) | 19(1) | 21(1) | 25(1) | -2(1) | 1(1) | 1(1) |
| C(13) | 21(2) | 27(2) | 27(2) | -2(1) | 0(1) | -1(1) |
| C(14) | 20(2) | 27(2) | 27(2) | 1(1) | -2(1) | 5(1) |
| C(15) | 27(2) | 31(2) | 35(2) | 2(2) | 2(1) | 2(2) |
| C(16) | 33(2) | 48(2) | 31(2) | 4(2) | 7(2) | 7(2) |
| C(17) | 36(2) | 52(3) | 31(2) | -1(2) | -3(2) | 10(2) |
| O(17) | 55(2) | 74(2) | 31(2) | -4(1) | -4(1) | 2(2) |
| C(18) | 32(2) | 40(2) | 38(2) | -3(2) | -3(2) | 0(2) |
| C(19) | 25(2) | 31(2) | 37(2) | -1(2) | -2(1) | 4(2) |
| C(20) | 83(3) | 90(4) | 26(2) | 6(2) | 4(2) | 3(3) |
| C(21) | 19(2) | 19(2) | 25(2) | 2(1) | -3(1) | -3(1) |
| O(21) | 26(1) | 22(1) | 40(1) | 3(1) | 3(1) | 1(1) |
| N(21) | 21(1) | 24(2) | 36(2) | 3(1) | 1(1) | -4(1) |
| C(22) | 21(2) | 27(2) | 33(2) | 3(1) | 2(1) | -1(1) |
| C(23) | 20(2) | 32(2) | 32(2) | 1(2) | 1(1) | 3(1) |
| C(24) | 42(2) | 47(2) | 36(2) | 0(2) | -2(2) | -12(2) |
| C(25) | 48(2) | 71(3) | 38(2) | -6(2) | -4(2) | -16(2) |
| C(26) | 31(2) | 72(3) | 34(2) | 4(2) | -1(2) | 0(2) |
| C(27) | 36(2) | 59(3) | 41(2) | 15(2) | 9(2) | -1(2) |
| C(28) | 32(2) | 41(2) | 35(2) | 4(2) | 5(2) | -2(2) |
| C(29) | 52(3) | 112(4) | 30(2) | 7(2) | 1(2) | -12(3) |

Table 4. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for compound **5b**.

| Atom | x | y | z | U(iso) |
|--------|------|-------|------|--------|
| H(1A) | 2036 | -2 | 2632 | 34 |
| H(2A) | 1879 | -2238 | 3019 | 39 |
| H(3A) | 1067 | -3659 | 2751 | 43 |
| H(4A) | 423 | -2939 | 2075 | 39 |
| H(6A) | 43 | 811 | 2010 | 35 |
| H(6B) | -404 | 49 | 1596 | 35 |
| H(7A) | -355 | 763 | 671 | 47 |
| H(8A) | -206 | 2572 | 62 | 57 |
| H(9A) | 329 | 4659 | 301 | 63 |
| H(10A) | 668 | 4962 | 1158 | 48 |
| H(13A) | 1157 | 1280 | 1536 | 30 |
| H(15A) | 616 | 1148 | 3025 | 37 |
| H(16A) | 852 | 1567 | 3900 | 45 |
| H(18A) | 2069 | 4616 | 3457 | 44 |
| H(19A) | 1825 | 4210 | 2585 | 37 |
| H(20A) | 1513 | 3108 | 5018 | 99 |
| H(20B) | 1440 | 1725 | 4651 | 99 |
| H(20C) | 911 | 2916 | 4666 | 99 |
| H(21) | 2352 | 46 | 1691 | 33 |
| H(22A) | 3363 | 705 | 1559 | 32 |
| H(22B) | 3166 | 2349 | 1627 | 32 |
| H(24A) | 2639 | -177 | 660 | 50 |
| H(25A) | 2705 | 163 | -236 | 63 |
| H(27A) | 3646 | 3826 | -8 | 55 |
| H(28A) | 3575 | 3497 | 889 | 43 |
| H(29A) | 3445 | 3143 | -880 | 97 |
| H(29B) | 2804 | 2330 | -952 | 97 |
| H(29C) | 3431 | 1440 | -964 | 97 |

Table 5. Torsion angles [$^\circ$] for compound **5b**.

| Atomic Indications | | | | Torsion angles |
|--------------------|-------|-------|-------|----------------|
| C(2) | C(1) | C(1A) | C(4A) | 0.0(5) |
| C(2) | C(1) | C(1A) | C(13) | 175.7(3) |
| C(1A) | C(1) | C(2) | C(3) | 0.5(5) |
| C(1) | C(2) | C(3) | C(4) | 1.2(5) |
| C(2) | C(3) | C(4) | C(4A) | 1.3(5) |
| C(3) | C(4) | C(4A) | O(5) | 176.3(3) |
| C(3) | C(4) | C(4A) | C(1A) | 0.8(5) |
| C(1) | C(1A) | C(4A) | O(5) | 175.6(3) |

| | | | | |
|--------|--------|--------|--------|----------|
| C(13) | C(1A) | C(4A) | O(5) | 8.5(4) |
| C(1) | C(1A) | C(4A) | C(4) | 0.1(5) |
| C(13) | C(1A) | C(4A) | C(4) | 176.0(3) |
| C(4) | C(4A) | O(5) | C(6) | 105.8(3) |
| C(1A) | C(4A) | O(5) | C(6) | 78.7(4) |
| C(4A) | O(5) | C(6) | C(6A) | 124.4(3) |
| O(5) | C(6) | C(6A) | C(7) | 80.1(4) |
| O(5) | C(6) | C(6A) | C(10A) | 98.5(4) |
| C(10A) | C(6A) | C(7) | C(8) | 2.1(5) |
| C(6) | C(6A) | C(7) | C(8) | 176.6(3) |
| C(6A) | C(7) | C(8) | C(9) | 0.7(6) |
| C(7) | C(8) | C(9) | C(10) | 1.6(6) |
| C(8) | C(9) | C(10) | C(10A) | 2.4(6) |
| C(7) | C(6A) | C(10A) | C(10) | 1.3(5) |
| C(6) | C(6A) | C(10A) | C(10) | 177.3(3) |
| C(7) | C(6A) | C(10A) | C(11) | 174.3(3) |
| C(6) | C(6A) | C(10A) | C(11) | 7.0(5) |
| C(9) | C(10) | C(10A) | C(6A) | 0.9(5) |
| C(9) | C(10) | C(10A) | C(11) | 176.8(3) |
| C(6A) | C(10A) | C(11) | O(11) | 116.5(4) |
| C(10) | C(10A) | C(11) | O(11) | 59.3(4) |
| C(6A) | C(10A) | C(11) | N(12) | 62.8(4) |
| C(10) | C(10A) | C(11) | N(12) | 121.4(3) |
| O(11) | C(11) | N(12) | C(14) | 5.1(4) |
| C(10A) | C(11) | N(12) | C(14) | 174.3(3) |
| O(11) | C(11) | N(12) | C(13) | 169.6(3) |
| C(10A) | C(11) | N(12) | C(13) | 11.1(4) |
| C(11) | N(12) | C(13) | C(1A) | 126.0(3) |
| C(14) | N(12) | C(13) | C(1A) | 59.3(3) |
| C(11) | N(12) | C(13) | C(21) | 99.9(3) |
| C(14) | N(12) | C(13) | C(21) | 74.8(3) |
| C(1) | C(1A) | C(13) | N(12) | 85.9(4) |
| C(4A) | C(1A) | C(13) | N(12) | 89.8(3) |
| C(1) | C(1A) | C(13) | C(21) | 44.3(4) |
| C(4A) | C(1A) | C(13) | C(21) | 140.1(3) |
| C(11) | N(12) | C(14) | C(15) | 93.4(4) |
| C(13) | N(12) | C(14) | C(15) | 91.7(3) |
| C(11) | N(12) | C(14) | C(19) | 83.4(4) |
| C(13) | N(12) | C(14) | C(19) | 91.5(3) |
| C(19) | C(14) | C(15) | C(16) | 0.1(5) |
| N(12) | C(14) | C(15) | C(16) | 176.7(3) |
| C(14) | C(15) | C(16) | C(17) | 0.1(5) |
| C(15) | C(16) | C(17) | O(17) | 178.6(3) |

| | | | | |
|-------|-------|-------|-------|----------|
| C(15) | C(16) | C(17) | C(18) | 0.7(5) |
| C(16) | C(17) | O(17) | C(20) | 0.1(6) |
| C(18) | C(17) | O(17) | C(20) | 179.3(4) |
| O(17) | C(17) | C(18) | C(19) | 178.3(3) |
| C(16) | C(17) | C(18) | C(19) | 1.0(5) |
| C(17) | C(18) | C(19) | C(14) | 0.8(5) |
| C(15) | C(14) | C(19) | C(18) | 0.2(5) |
| N(12) | C(14) | C(19) | C(18) | 177.0(3) |
| N(12) | C(13) | C(21) | O(21) | 27.2(4) |
| C(1A) | C(13) | C(21) | O(21) | 157.6(3) |
| N(12) | C(13) | C(21) | N(21) | 156.6(3) |
| C(1A) | C(13) | C(21) | N(21) | 26.2(4) |
| O(21) | C(21) | N(21) | C(22) | 2.1(5) |
| C(13) | C(21) | N(21) | C(22) | 173.8(3) |
| C(21) | N(21) | C(22) | C(23) | 85.2(4) |
| N(21) | C(22) | C(23) | C(28) | 137.2(3) |
| N(21) | C(22) | C(23) | C(24) | 44.9(4) |
| C(28) | C(23) | C(24) | C(25) | 1.4(6) |
| C(22) | C(23) | C(24) | C(25) | 179.5(3) |
| C(23) | C(24) | C(25) | C(26) | 0.5(6) |
| C(24) | C(25) | C(26) | C(27) | 0.8(6) |
| C(24) | C(25) | C(26) | C(29) | 179.7(4) |
| C(25) | C(26) | C(27) | C(28) | 1.0(6) |
| C(29) | C(26) | C(27) | C(28) | 179.5(4) |
| C(24) | C(23) | C(28) | C(27) | 1.2(5) |
| C(22) | C(23) | C(28) | C(27) | 179.2(3) |
| C(26) | C(27) | C(28) | C(23) | 0.0(6) |

Table 6. Hydrogen bonds for compound **5b** [\AA and $^\circ$].

| D-H...A | d(D-H) | d(H...A) | d(D...A) | \angle (DHA) |
|-----------------------|--------|----------|----------|----------------|
| N(21)-H(21)...O(21)#1 | 0.91 | 2.08 | 2.747(3) | 129 |

Symmetry transformations used to generate equivalent atoms:

#1 $-x+1/2, y-1/2, z$