

Unexpected formation of dinaphthoaza-17-crown-5 ether containing γ -aminopiperidine subunit

Anh T. Le, Nhung T. Dao, Hieu H. Truong, Thuyen T. Do, Elena I. Polyakova, Ivan V. Lin'ko, Pavel V. Dorovatovskii and Victor N. Khrustalev

The full mechanism of the transformation can be rationalized as below:

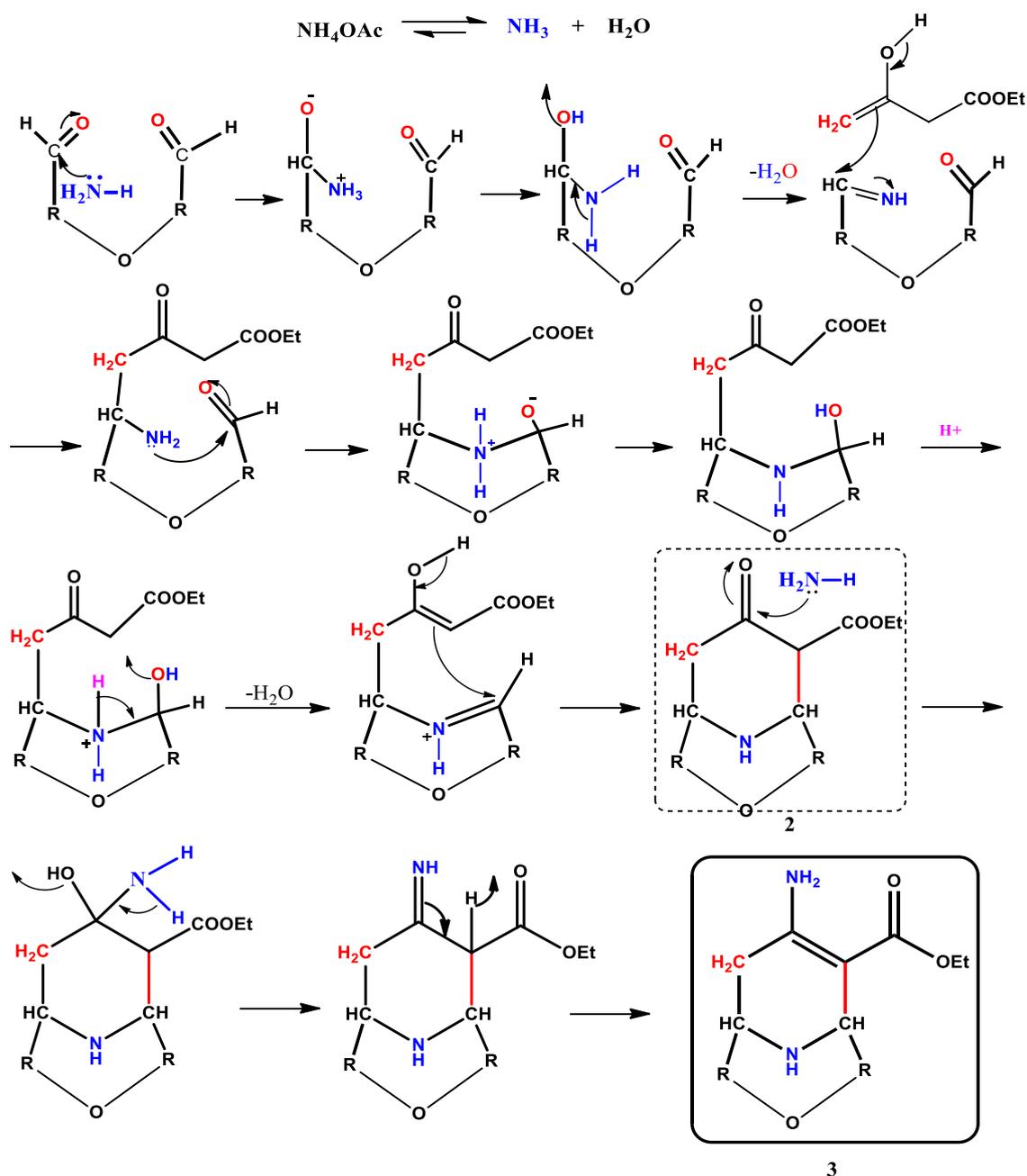


Figure S1. The suggested mechanism of reaction

New compound **3** gave satisfactory spectroscopic and analytical results. Its spectral data including HRMS, ^1H NMR and ^{13}C NMR was consistent with the structure of 2⁴-amino-2³-ethoxycarbonyl-2¹,2²,2⁵,2⁶-tetrahydro-4,7,10,13-tetraoxa-2(2,6)-pyridina-1(2,1),3(1,2)-dinaphthalenacyclotridecaphane.

The molecule of **3** comprises a fused tetracyclic system containing the aza-17-crown-5-ether macrocycle, 1,2,3,6-tetrahydropyridine and two naphthalene rings. The dihedral angles between the basal (C32/C33/C34/C35)-plane of the 1,2,3,6-tetrahydropyridine ring and planes of the two naphthalene rings are 86.76(4) and 71.34(5)°, respectively. The N34 and N36 nitrogen atoms have the trigonal-planar (sum of the bond angles is 358(3)°) and trigonal-pyramidal (sum of the bond angles is 325(2)°) configurations, respectively.

The solvate methanol and water molecules form a robust 1:1:1 H-bonded associate with the molecule **3** (Figures 1 and S2).

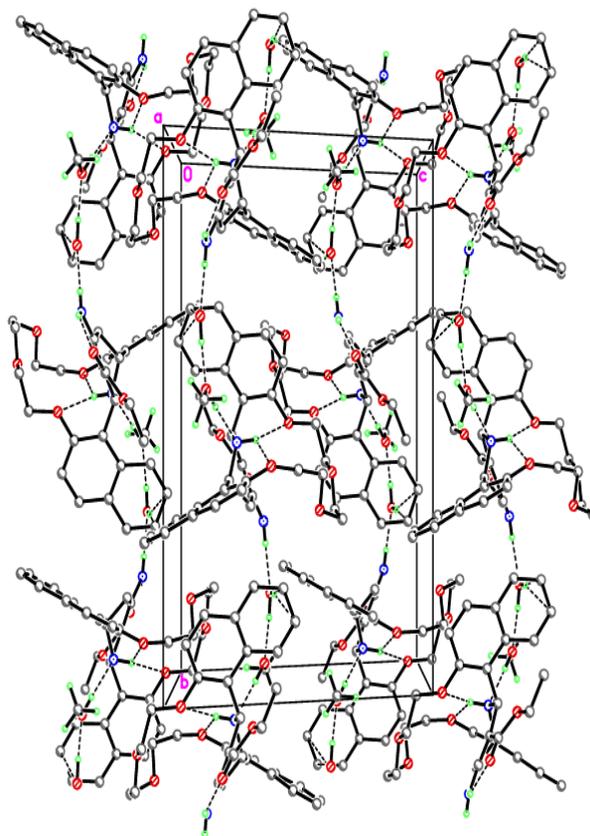


Figure S2. Crystal structure of **3** • CH₃OH • H₂O demonstrating the H-bonded wave-like chains along the crystallographic *b* axis. The intramolecular N—H⋯O and intermolecular C—H⋯O hydrogen bonds are depicted by dotted and dashed lines.

Table S1. Hydrogen bonds for **3** • CH₃OH • H₂O [Å and °].

D*—H...A*	d(D—H)	d(H...A)	d(D...A)	∠ (DHA)
N(34)—H(34A)...O(37)	0.884(15)	2.027(15)	2.7087(13)	133.1(12)
N(34)—H(34B)...O(39)#1	0.890(15)	2.079(15)	2.9667(14)	175.6(13)
N(36)—H(36)...O(12)	0.898(13)	2.358(13)	2.8321(15)	113.0(10)
N(36)—H(36)...O(21)	0.898(13)	2.573(13)	3.1216(13)	120.1(10)
O(39)—H(39D)...O(40)	0.95(2)	1.83(2)	2.7735(13)	173.8(18)
O(40)—H(40)...N(36)	0.858(18)	2.017(18)	2.8708(13)	173.2(16)

* D – proton donor; A – proton acceptor;

Symmetry transformations used to generate equivalent atoms: #1 -x+1/2, y-1/2, -z+3/2

Table S2. Crystal data and structure refinement for **3**

Identification code	3	
Empirical formula	C35 H42 N2 O8	
Formula weight	618.71	
Temperature	100(2) K	
Wavelength	0.78790 Å	
Crystal system	Monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 12.490(3) Å	$\angle = 90^\circ$.
	b = 20.595(4) Å	$\angle = 109.90(3)^\circ$.
	c = 13.061(3) Å	$\angle = 90^\circ$.
Volume	3159.1(14) Å ³	
Z	4	
Density (calculated)	1.301 mg m ⁻³	
Absorption coefficient	0.115 mm ⁻¹	
F(000)	1320	
Crystal size	0.15 x 0.10 x 0.10 mm ³	
Theta range for data collection	2.140 to 31.005°.	
Index ranges	-16<=h<=16, -26<=k<=26, -16<=l<=17	
Reflections collected	27774	
Independent reflections	7317 [R(int) = 0.0276]	
Completeness to theta = 28.212°	98.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.980 and 0.970	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	7317 / 0 / 427	
Goodness-of-fit on F ²	1.051	
Final R indices [for 6533 rflns with I>2σ(I)]	R1 = 0.0366, wR2 = 0.0956	
R indices (all data)	R1 = 0.0409, wR2 = 0.0994	
Extinction coefficient	0.0091(9)	
Largest diff. peak and hole	0.272 and -0.194 e Å ⁻³	

Table S3. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters (Å² $\times 10^3$) for **3**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

Atom	x	y	z	U(eq)
C(1)	2265(1)	3868(1)	7742(1)	22(1)
C(2)	1124(1)	3726(1)	7863(1)	24(1)
C(3)	1081(1)	3412(1)	8827(1)	26(1)
C(4)	2063(1)	3174(1)	9661(1)	30(1)
C(5)	1995(1)	2889(1)	10584(1)	37(1)
C(6)	936(1)	2828(1)	10738(1)	41(1)
C(7)	-33(1)	3027(1)	9944(1)	37(1)
C(8)	3(1)	3310(1)	8962(1)	30(1)
C(9)	-999(1)	3492(1)	8111(1)	34(1)
C(10)	-953(1)	3757(1)	7171(1)	33(1)
C(11)	116(1)	3873(1)	7042(1)	28(1)
O(12)	209(1)	4146(1)	6122(1)	31(1)
C(13)	-739(1)	4086(1)	5118(1)	36(1)
C(14)	-275(1)	3997(1)	4213(1)	38(1)
O(15)	210(1)	3359(1)	4320(1)	38(1)
C(16)	617(1)	3183(1)	3462(1)	40(1)
C(17)	1864(1)	3324(1)	3730(1)	38(1)
O(18)	2031(1)	4006(1)	3843(1)	35(1)
C(19)	3182(1)	4188(1)	4072(1)	32(1)
C(20)	3315(1)	4882(1)	4445(1)	29(1)
O(21)	3196(1)	4898(1)	5499(1)	27(1)

C(22)	3618(1)	5424(1)	6159(1)	24(1)
C(23)	3824(1)	6024(1)	5733(1)	27(1)
C(24)	4198(1)	6544(1)	6414(1)	28(1)
C(25)	4370(1)	6492(1)	7539(1)	25(1)
C(26)	4731(1)	7039(1)	8236(1)	29(1)
C(27)	4896(1)	6997(1)	9326(1)	31(1)
C(28)	4709(1)	6398(1)	9762(1)	28(1)
C(29)	4355(1)	5860(1)	9110(1)	25(1)
C(30)	4172(1)	5884(1)	7972(1)	23(1)
C(31)	3804(1)	5338(1)	7260(1)	22(1)
C(32)	3641(1)	4673(1)	7700(1)	21(1)
C(33)	4249(1)	4124(1)	7324(1)	22(1)
C(34)	3707(1)	3554(1)	6915(1)	23(1)
N(34)	4166(1)	3077(1)	6491(1)	26(1)
C(35)	2538(1)	3407(1)	6948(1)	25(1)
N(36)	2409(1)	4550(1)	7458(1)	22(1)
C(37)	5460(1)	4186(1)	7522(1)	23(1)
O(37)	6042(1)	3842(1)	7147(1)	29(1)
O(38)	5925(1)	4669(1)	8237(1)	26(1)
C(38)	7144(1)	4760(1)	8568(1)	28(1)
C(39)	7390(1)	5417(1)	9099(1)	33(1)
O(39)	2001(1)	6801(1)	8801(1)	42(1)
O(40)	1488(1)	5487(1)	8559(1)	32(1)
C(40)	392(1)	5316(1)	8577(1)	35(1)

Table S4. Bond lengths [Å] and angles [°] for **3**.

C(1)-N(36)	1.4806(12)	C(16)-H(16B)	0.9900
C(1)-C(2)	1.5153(13)	C(17)-O(18)	1.4217(15)
C(1)-C(35)	1.5267(13)	C(17)-H(17A)	0.9900
C(1)-H(1)	1.0000	C(17)-H(17B)	0.9900
C(2)-C(11)	1.3813(15)	O(18)-C(19)	1.4154(14)
C(2)-C(3)	1.4326(15)	C(19)-C(20)	1.5008(16)
C(3)-C(4)	1.4225(17)	C(19)-H(19A)	0.9900
C(3)-C(8)	1.4309(14)	C(19)-H(19B)	0.9900
C(4)-C(5)	1.3692(16)	C(20)-O(21)	1.4340(12)
C(4)-H(4)	0.9500	C(20)-H(20A)	0.9900
C(5)-C(6)	1.4088(18)	C(20)-H(20B)	0.9900
C(5)-H(5)	0.9500	O(21)-C(22)	1.3728(12)
C(6)-C(7)	1.361(2)	C(22)-C(31)	1.3880(14)
C(6)-H(6)	0.9500	C(22)-C(23)	1.4141(14)
C(7)-C(8)	1.4233(17)	C(23)-C(24)	1.3683(15)
C(7)-H(7)	0.9500	C(23)-H(23)	0.9500
C(8)-C(9)	1.4129(18)	C(24)-C(25)	1.4143(15)
C(9)-C(10)	1.3623(18)	C(24)-H(24)	0.9500
C(9)-H(9)	0.9500	C(25)-C(26)	1.4215(15)
C(10)-C(11)	1.4221(15)	C(25)-C(30)	1.4292(14)
C(10)-H(10)	0.9500	C(26)-C(27)	1.3704(16)
C(11)-O(12)	1.3677(14)	C(26)-H(26)	0.9500
O(12)-C(13)	1.4422(14)	C(27)-C(28)	1.4115(16)
C(13)-C(14)	1.4947(18)	C(27)-H(27)	0.9500
C(13)-H(13A)	0.9900	C(28)-C(29)	1.3757(15)
C(13)-H(13B)	0.9900	C(28)-H(28)	0.9500
C(14)-O(15)	1.4337(15)	C(29)-C(30)	1.4261(14)
C(14)-H(14A)	0.9900	C(29)-H(29)	0.9500
C(14)-H(14B)	0.9900	C(30)-C(31)	1.4320(14)
O(15)-C(16)	1.4266(16)	C(31)-C(32)	1.5255(13)
C(16)-C(17)	1.5036(19)	C(32)-N(36)	1.4839(12)
C(16)-H(16A)	0.9900	C(32)-C(33)	1.5335(13)

C(32)-H(32)	1.0000	C(38)-C(39)	1.5039(15)
C(33)-C(34)	1.3699(14)	C(38)-H(38A)	0.9900
C(33)-C(37)	1.4509(14)	C(38)-H(38B)	0.9900
C(34)-N(34)	1.3483(13)	C(39)-H(39A)	0.9800
C(34)-C(35)	1.5056(14)	C(39)-H(39B)	0.9800
N(34)-H(34A)	0.884(15)	C(39)-H(39C)	0.9800
N(34)-H(34B)	0.890(15)	O(39)-H(39D)	0.95(2)
C(35)-H(35A)	0.9900	O(39)-H(39E)	0.89(2)
C(35)-H(35B)	0.9900	O(40)-C(40)	1.4209(14)
N(36)-H(36)	0.898(13)	O(40)-H(40)	0.858(18)
C(37)-O(37)	1.2294(12)	C(40)-H(40A)	0.9800
C(37)-O(38)	1.3526(12)	C(40)-H(40B)	0.9800
O(38)-C(38)	1.4471(12)	C(40)-H(40C)	0.9800
N(36)-C(1)-C(2)	114.09(8)	C(13)-C(14)-H(14A)	110.2
N(36)-C(1)-C(35)	110.22(8)	O(15)-C(14)-H(14B)	110.2
C(2)-C(1)-C(35)	112.75(8)	C(13)-C(14)-H(14B)	110.2
N(36)-C(1)-H(1)	106.4	H(14A)-C(14)-H(14B)	108.5
C(2)-C(1)-H(1)	106.4	C(16)-O(15)-C(14)	114.09(9)
C(35)-C(1)-H(1)	106.4	O(15)-C(16)-C(17)	112.71(10)
C(11)-C(2)-C(3)	119.00(9)	O(15)-C(16)-H(16A)	109.0
C(11)-C(2)-C(1)	121.15(9)	C(17)-C(16)-H(16A)	109.0
C(3)-C(2)-C(1)	119.82(9)	O(15)-C(16)-H(16B)	109.0
C(4)-C(3)-C(8)	117.22(10)	C(17)-C(16)-H(16B)	109.0
C(4)-C(3)-C(2)	123.22(9)	H(16A)-C(16)-H(16B)	107.8
C(8)-C(3)-C(2)	119.53(10)	O(18)-C(17)-C(16)	108.58(10)
C(5)-C(4)-C(3)	121.71(11)	O(18)-C(17)-H(17A)	110.0
C(5)-C(4)-H(4)	119.1	C(16)-C(17)-H(17A)	110.0
C(3)-C(4)-H(4)	119.1	O(18)-C(17)-H(17B)	110.0
C(4)-C(5)-C(6)	120.44(12)	C(16)-C(17)-H(17B)	110.0
C(4)-C(5)-H(5)	119.8	H(17A)-C(17)-H(17B)	108.4
C(6)-C(5)-H(5)	119.8	C(19)-O(18)-C(17)	112.74(9)
C(7)-C(6)-C(5)	119.93(11)	O(18)-C(19)-C(20)	108.23(9)
C(7)-C(6)-H(6)	120.0	O(18)-C(19)-H(19A)	110.1
C(5)-C(6)-H(6)	120.0	C(20)-C(19)-H(19A)	110.1
C(6)-C(7)-C(8)	121.11(11)	O(18)-C(19)-H(19B)	110.1
C(6)-C(7)-H(7)	119.4	C(20)-C(19)-H(19B)	110.1
C(8)-C(7)-H(7)	119.4	H(19A)-C(19)-H(19B)	108.4
C(9)-C(8)-C(7)	121.78(11)	O(21)-C(20)-C(19)	107.29(9)
C(9)-C(8)-C(3)	118.83(10)	O(21)-C(20)-H(20A)	110.3
C(7)-C(8)-C(3)	119.39(11)	C(19)-C(20)-H(20A)	110.3
C(10)-C(9)-C(8)	121.22(10)	O(21)-C(20)-H(20B)	110.3
C(10)-C(9)-H(9)	119.4	C(19)-C(20)-H(20B)	110.3
C(8)-C(9)-H(9)	119.4	H(20A)-C(20)-H(20B)	108.5
C(9)-C(10)-C(11)	120.20(11)	C(22)-O(21)-C(20)	118.57(8)
C(9)-C(10)-H(10)	119.9	O(21)-C(22)-C(31)	116.31(9)
C(11)-C(10)-H(10)	119.9	O(21)-C(22)-C(23)	121.62(9)
O(12)-C(11)-C(2)	116.43(9)	C(31)-C(22)-C(23)	122.06(9)
O(12)-C(11)-C(10)	122.53(10)	C(24)-C(23)-C(22)	119.67(9)
C(2)-C(11)-C(10)	121.02(10)	C(24)-C(23)-H(23)	120.2
C(11)-O(12)-C(13)	118.28(9)	C(22)-C(23)-H(23)	120.2
O(12)-C(13)-C(14)	108.05(10)	C(23)-C(24)-C(25)	120.94(10)
O(12)-C(13)-H(13A)	110.1	C(23)-C(24)-H(24)	119.5
C(14)-C(13)-H(13A)	110.1	C(25)-C(24)-H(24)	119.5
O(12)-C(13)-H(13B)	110.1	C(24)-C(25)-C(26)	120.55(10)
C(14)-C(13)-H(13B)	110.1	C(24)-C(25)-C(30)	119.43(9)
H(13A)-C(13)-H(13B)	108.4	C(26)-C(25)-C(30)	120.01(10)
O(15)-C(14)-C(13)	107.41(10)		
O(15)-C(14)-H(14A)	110.2		

C(27)-C(26)-C(25)	121.19(10)	C(34)-C(35)-C(1)	109.74(8)
C(27)-C(26)-H(26)	119.4	C(34)-C(35)-H(35A)	109.7
C(25)-C(26)-H(26)	119.4	C(1)-C(35)-H(35A)	109.7
C(26)-C(27)-C(28)	119.21(10)	C(34)-C(35)-H(35B)	109.7
C(26)-C(27)-H(27)	120.4	C(1)-C(35)-H(35B)	109.7
C(28)-C(27)-H(27)	120.4	H(35A)-C(35)-H(35B)	108.2
C(29)-C(28)-C(27)	121.09(10)	C(1)-N(36)-C(32)	108.30(7)
C(29)-C(28)-H(28)	119.5	C(1)-N(36)-H(36)	108.4(8)
C(27)-C(28)-H(28)	119.5	C(32)-N(36)-H(36)	108.3(8)
C(28)-C(29)-C(30)	121.36(10)	O(37)-C(37)-O(38)	121.75(9)
C(28)-C(29)-H(29)	119.3	O(37)-C(37)-C(33)	126.84(9)
C(30)-C(29)-H(29)	119.3	O(38)-C(37)-C(33)	111.35(8)
C(29)-C(30)-C(25)	117.13(9)	C(37)-O(38)-C(38)	117.49(8)
C(29)-C(30)-C(31)	123.43(9)	O(38)-C(38)-C(39)	106.39(8)
C(25)-C(30)-C(31)	119.45(9)	O(38)-C(38)-H(38A)	110.4
C(22)-C(31)-C(30)	118.41(9)	C(39)-C(38)-H(38A)	110.4
C(22)-C(31)-C(32)	120.57(9)	O(38)-C(38)-H(38B)	110.4
C(30)-C(31)-C(32)	121.01(8)	C(39)-C(38)-H(38B)	110.4
N(36)-C(32)-C(31)	109.52(8)	H(38A)-C(38)-H(38B)	108.6
N(36)-C(32)-C(33)	113.39(8)	C(38)-C(39)-H(39A)	109.5
C(31)-C(32)-C(33)	113.40(8)	C(38)-C(39)-H(39B)	109.5
N(36)-C(32)-H(32)	106.7	H(39A)-C(39)-H(39B)	109.5
C(31)-C(32)-H(32)	106.7	C(38)-C(39)-H(39C)	109.5
C(33)-C(32)-H(32)	106.7	H(39A)-C(39)-H(39C)	109.5
C(34)-C(33)-C(37)	119.49(9)	H(39B)-C(39)-H(39C)	109.5
C(34)-C(33)-C(32)	121.60(9)	H(39D)-O(39)-H(39E)	101.1(16)
C(37)-C(33)-C(32)	118.55(8)	C(40)-O(40)-H(40)	108.5(11)
N(34)-C(34)-C(33)	124.14(9)	O(40)-C(40)-H(40A)	109.5
N(34)-C(34)-C(35)	114.73(9)	O(40)-C(40)-H(40B)	109.5
C(33)-C(34)-C(35)	121.09(9)	H(40A)-C(40)-H(40B)	109.5
C(34)-N(34)-H(34A)	116.4(9)	O(40)-C(40)-H(40C)	109.5
C(34)-N(34)-H(34B)	118.5(9)	H(40A)-C(40)-H(40C)	109.5
H(34A)-N(34)-H(34B)	123.0(13)	H(40B)-C(40)-H(40C)	109.5

Table S5. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **3**. 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(1)	20(1)	25(1)	0(1)	9(1)	1(1)
C(2)	24(1)	20(1)	31(1)	-4(1)	12(1)	-1(1)
C(3)	31(1)	21(1)	31(1)	-7(1)	16(1)	-5(1)
C(4)	34(1)	30(1)	29(1)	-3(1)	12(1)	-9(1)
C(5)	43(1)	40(1)	27(1)	-2(1)	10(1)	-15(1)
C(6)	53(1)	45(1)	29(1)	-7(1)	20(1)	-22(1)
C(7)	45(1)	35(1)	42(1)	-12(1)	28(1)	-15(1)
C(8)	34(1)	24(1)	39(1)	-9(1)	21(1)	-6(1)
C(9)	30(1)	26(1)	51(1)	-6(1)	22(1)	-2(1)
C(10)	25(1)	25(1)	47(1)	-3(1)	11(1)	2(1)
C(11)	26(1)	21(1)	36(1)	-2(1)	10(1)	0(1)
O(12)	24(1)	32(1)	33(1)	4(1)	4(1)	0(1)
C(13)	27(1)	37(1)	37(1)	4(1)	1(1)	2(1)
C(14)	38(1)	36(1)	34(1)	6(1)	3(1)	1(1)
O(15)	47(1)	32(1)	33(1)	2(1)	10(1)	0(1)
C(16)	49(1)	36(1)	32(1)	-6(1)	8(1)	-6(1)
C(17)	47(1)	32(1)	32(1)	-7(1)	8(1)	1(1)
O(18)	33(1)	32(1)	34(1)	-5(1)	4(1)	0(1)
C(19)	33(1)	36(1)	27(1)	-3(1)	9(1)	4(1)

C(20)	30(1)	34(1)	22(1)	1(1)	9(1)	2(1)
O(21)	34(1)	26(1)	22(1)	-1(1)	9(1)	-3(1)
C(22)	21(1)	24(1)	25(1)	0(1)	7(1)	1(1)
C(23)	26(1)	28(1)	25(1)	5(1)	8(1)	1(1)
C(24)	27(1)	24(1)	31(1)	6(1)	9(1)	0(1)
C(25)	22(1)	22(1)	30(1)	2(1)	7(1)	1(1)
C(26)	29(1)	21(1)	35(1)	2(1)	8(1)	-1(1)
C(27)	31(1)	24(1)	35(1)	-5(1)	7(1)	-1(1)
C(28)	29(1)	27(1)	27(1)	-2(1)	7(1)	1(1)
C(29)	25(1)	23(1)	26(1)	0(1)	8(1)	1(1)
C(30)	20(1)	22(1)	26(1)	1(1)	8(1)	2(1)
C(31)	20(1)	21(1)	24(1)	1(1)	7(1)	1(1)
C(32)	21(1)	20(1)	22(1)	0(1)	7(1)	1(1)
C(33)	22(1)	22(1)	22(1)	1(1)	8(1)	2(1)
C(34)	24(1)	23(1)	22(1)	1(1)	7(1)	3(1)
N(34)	26(1)	23(1)	32(1)	-4(1)	12(1)	1(1)
C(35)	24(1)	22(1)	28(1)	-3(1)	10(1)	-1(1)
N(36)	21(1)	20(1)	25(1)	1(1)	8(1)	0(1)
C(37)	25(1)	22(1)	22(1)	2(1)	8(1)	1(1)
O(37)	25(1)	30(1)	35(1)	-6(1)	12(1)	1(1)
O(38)	21(1)	28(1)	28(1)	-5(1)	7(1)	-1(1)
C(38)	21(1)	33(1)	30(1)	-3(1)	7(1)	-1(1)
C(39)	28(1)	34(1)	36(1)	-5(1)	8(1)	-5(1)
O(39)	31(1)	28(1)	71(1)	0(1)	21(1)	2(1)
O(40)	34(1)	27(1)	38(1)	-5(1)	16(1)	0(1)
C(40)	35(1)	31(1)	42(1)	-2(1)	17(1)	1(1)

Table S6. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **3**.

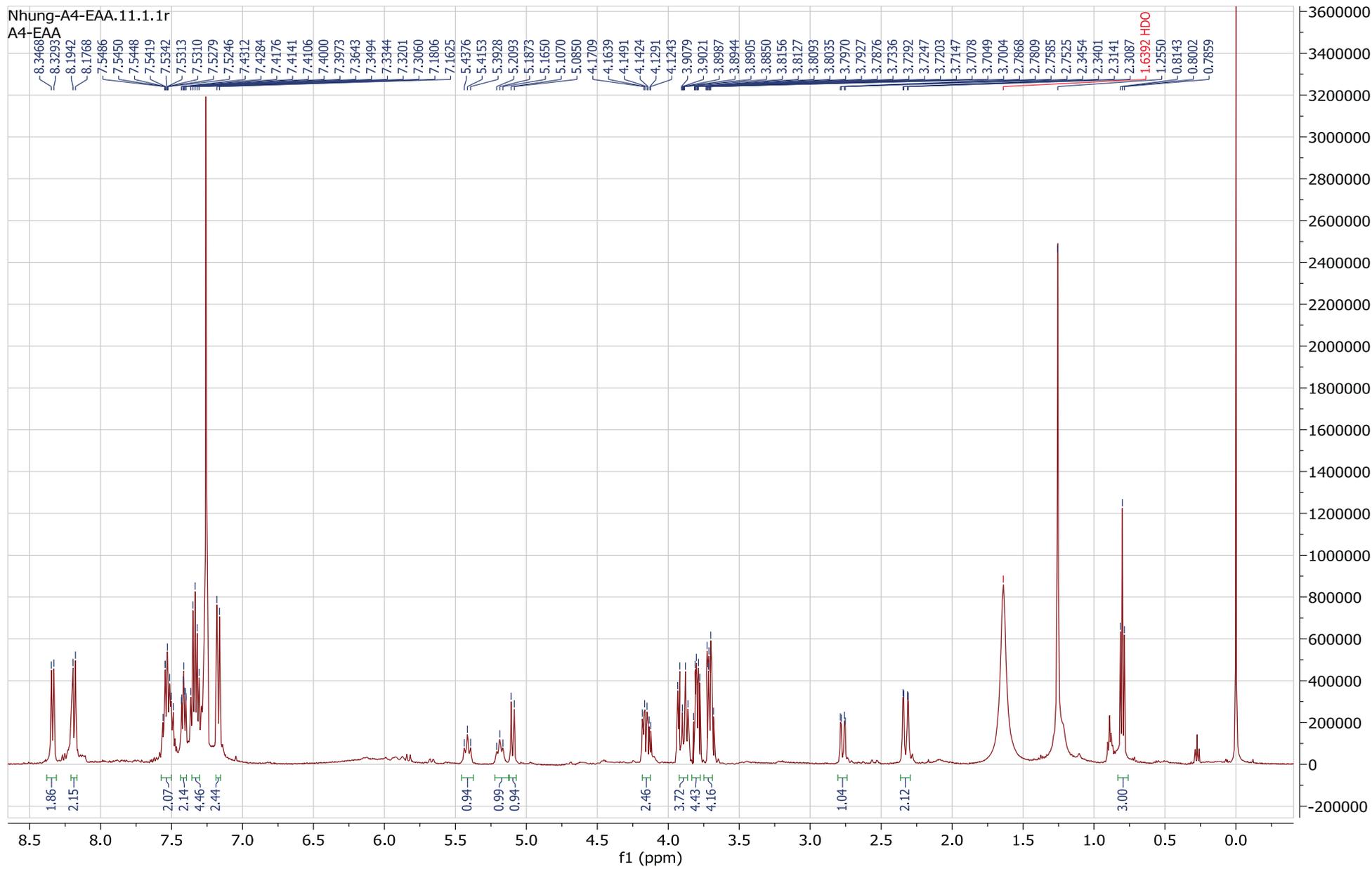
Atom	x	y	z	U(iso)
H(1)	2855	3787	8471	27
H(4)	2786	3213	9576	36
H(5)	2665	2731	11124	45
H(6)	899	2648	11395	49
H(7)	-745	2976	10048	45
H(9)	-1717	3429	8196	41
H(10)	-1638	3864	6599	39
H(13A)	-1219	4481	4991	43
H(13B)	-1214	3708	5156	43
H(14A)	-892	4043	3500	46
H(14B)	314	4329	4260	46
H(16A)	179	3424	2795	48
H(16B)	482	2713	3309	48
H(17A)	2310	3103	4417	46
H(17B)	2124	3160	3142	46
H(19A)	3393	4141	3411	39
H(19B)	3687	3905	4648	39
H(20A)	4073	5049	4488	34
H(20B)	2724	5156	3927	34
H(23)	3704	6066	4978	32
H(24)	4343	6945	6127	33
H(26)	4860	7441	7940	35
H(27)	5133	7366	9782	37
H(28)	4830	6365	10519	34
H(29)	4230	5463	9425	30
H(32)	3995	4697	8511	26
H(34A)	4859(13)	3149(7)	6477(11)	32
H(34B)	3853(12)	2684(7)	6412(11)	32
H(35A)	2508	2952	7181	30
H(35B)	1965	3460	6211	30

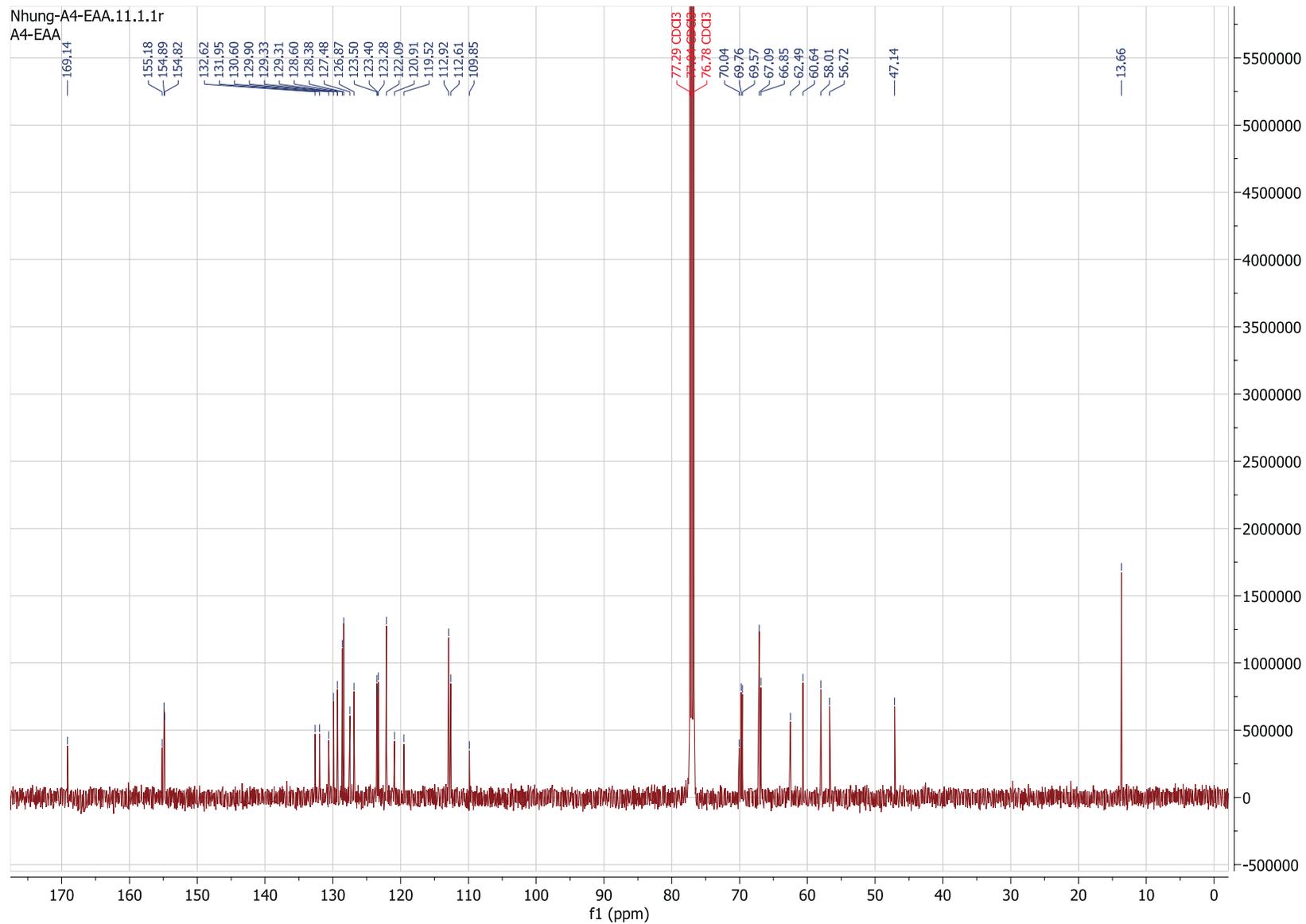
H(36)	2051(11)	4603(6)	6739(11)	27
H(38A)	7398	4742	7928	34
H(38B)	7546	4417	9088	34
H(39A)	6954	5748	8587	50
H(39B)	8205	5511	9304	50
H(39C)	7170	5420	9751	50
H(39D)	1881(16)	6345(10)	8747(15)	64
H(39E)	2753(18)	6822(9)	8939(15)	64
H(40)	1715(14)	5187(8)	8226(14)	49
H(40A)	-157	5324	7834	52
H(40B)	159	5627	9028	52
H(40C)	420	4879	8882	52

Table S7. Torsion angles [°] for **3**.

N(36)-C(1)-C(2)-C(11)	54.08(12)	C(24)-C(25)-C(26)-C(27)	179.69(10)
C(35)-C(1)-C(2)-C(11)	-72.65(12)	C(30)-C(25)-C(26)-C(27)	0.07(16)
N(36)-C(1)-C(2)-C(3)	-128.12(9)	C(25)-C(26)-C(27)-C(28)	0.34(16)
C(35)-C(1)-C(2)-C(3)	105.15(10)	C(26)-C(27)-C(28)-C(29)	-0.62(16)
C(11)-C(2)-C(3)-C(4)	172.57(10)	C(27)-C(28)-C(29)-C(30)	0.48(16)
C(1)-C(2)-C(3)-C(4)	-5.28(15)	C(28)-C(29)-C(30)-C(25)	-0.07(14)
C(11)-C(2)-C(3)-C(8)	-5.44(14)	C(28)-C(29)-C(30)-C(31)	179.99(9)
C(1)-C(2)-C(3)-C(8)	176.71(9)	C(24)-C(25)-C(30)-C(29)	-179.83(9)
C(8)-C(3)-C(4)-C(5)	-3.36(16)	C(26)-C(25)-C(30)-C(29)	-0.20(14)
C(2)-C(3)-C(4)-C(5)	178.59(10)	C(24)-C(25)-C(30)-C(31)	0.11(14)
C(3)-C(4)-C(5)-C(6)	-0.52(18)	C(26)-C(25)-C(30)-C(31)	179.74(9)
C(4)-C(5)-C(6)-C(7)	2.94(19)	O(21)-C(22)-C(31)-C(30)	-175.92(8)
C(5)-C(6)-C(7)-C(8)	-1.30(18)	C(23)-C(22)-C(31)-C(30)	2.58(14)
C(6)-C(7)-C(8)-C(9)	177.09(11)	O(21)-C(22)-C(31)-C(32)	5.26(13)
C(6)-C(7)-C(8)-C(3)	-2.67(17)	C(23)-C(22)-C(31)-C(32)	-176.24(9)
C(4)-C(3)-C(8)-C(9)	-174.88(10)	C(29)-C(30)-C(31)-C(22)	177.99(9)
C(2)-C(3)-C(8)-C(9)	3.24(15)	C(25)-C(30)-C(31)-C(22)	-1.95(13)
C(4)-C(3)-C(8)-C(7)	4.89(15)	C(29)-C(30)-C(31)-C(32)	-3.20(14)
C(2)-C(3)-C(8)-C(7)	-176.99(9)	C(25)-C(30)-C(31)-C(32)	176.86(8)
C(7)-C(8)-C(9)-C(10)	-179.32(10)	C(22)-C(31)-C(32)-N(36)	-78.31(11)
C(3)-C(8)-C(9)-C(10)	0.44(16)	C(30)-C(31)-C(32)-N(36)	102.90(10)
C(8)-C(9)-C(10)-C(11)	-1.93(17)	C(22)-C(31)-C(32)-C(33)	49.45(12)
C(3)-C(2)-C(11)-O(12)	-177.27(9)	C(30)-C(31)-C(32)-C(33)	-129.34(9)
C(1)-C(2)-C(11)-O(12)	0.55(14)	N(36)-C(32)-C(33)-C(34)	-6.56(13)
C(3)-C(2)-C(11)-C(10)	4.03(15)	C(31)-C(32)-C(33)-C(34)	-132.28(10)
C(1)-C(2)-C(11)-C(10)	-178.14(9)	N(36)-C(32)-C(33)-C(37)	-179.58(8)
C(9)-C(10)-C(11)-O(12)	-178.98(10)	C(31)-C(32)-C(33)-C(37)	54.69(11)
C(9)-C(10)-C(11)-C(2)	-0.37(16)	C(37)-C(33)-C(34)-N(34)	-12.65(15)
C(2)-C(11)-O(12)-C(13)	156.87(10)	C(32)-C(33)-C(34)-N(34)	174.40(9)
C(10)-C(11)-O(12)-C(13)	-24.46(15)	C(37)-C(33)-C(34)-C(35)	165.13(9)
C(11)-O(12)-C(13)-C(14)	-143.17(10)	C(32)-C(33)-C(34)-C(35)	-7.83(14)
O(12)-C(13)-C(14)-O(15)	71.26(12)	N(34)-C(34)-C(35)-C(1)	162.38(8)
C(13)-C(14)-O(15)-C(16)	175.63(10)	C(33)-C(34)-C(35)-C(1)	-15.59(13)
C(14)-O(15)-C(16)-C(17)	93.97(13)	N(36)-C(1)-C(35)-C(34)	54.26(11)
O(15)-C(16)-C(17)-O(18)	-64.42(13)	C(2)-C(1)-C(35)-C(34)	-176.97(8)
C(16)-C(17)-O(18)-C(19)	-178.60(9)	C(2)-C(1)-N(36)-C(32)	162.56(8)
C(17)-O(18)-C(19)-C(20)	-166.22(9)	C(35)-C(1)-N(36)-C(32)	-69.41(10)
O(18)-C(19)-C(20)-O(21)	69.77(11)	C(31)-C(32)-N(36)-C(1)	171.53(8)
C(19)-C(20)-O(21)-C(22)	160.24(9)	C(33)-C(32)-N(36)-C(1)	43.76(10)
C(20)-O(21)-C(22)-C(31)	-160.95(9)	C(34)-C(33)-C(37)-O(37)	17.76(15)
C(20)-O(21)-C(22)-C(23)	20.54(13)	C(32)-C(33)-C(37)-O(37)	-169.06(9)
O(21)-C(22)-C(23)-C(24)	177.12(9)	C(34)-C(33)-C(37)-O(38)	-159.55(9)
C(31)-C(22)-C(23)-C(24)	-1.30(15)	C(32)-C(33)-C(37)-O(38)	13.62(12)
C(22)-C(23)-C(24)-C(25)	-0.65(15)	O(37)-C(37)-O(38)-C(38)	-1.46(14)
C(23)-C(24)-C(25)-C(26)	-178.42(10)	C(33)-C(37)-O(38)-C(38)	176.02(8)
C(23)-C(24)-C(25)-C(30)	1.21(15)	C(37)-O(38)-C(38)-C(39)	165.06(9)

Nhung-A4-EAA.11.1.1r
A4-EAA





A4_ACETOACETATE_180420155405 #585 RT: 3.59 AV: 1 NL: 3.48E7

T: ITMS + c ESI Full ms [50.00-1000.00]

