S1 Rigid Core Vinamidinium Salts and Their N,N'-Rotamers

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Table of Contents for Supporting Information:

S3 – ORTEP Drawing of Cation **3a**

S4 - X-Ray Data Collection, Structure Solution and Refinement for **3a**

S5 - Unit Cell with Atomic Skeleton of **3a**

S6 - Table S1, Crystal Data, Data Collection, and Solution and Refinement for **3a**

S7 - Table S2, Atomic Coordinates and Equivalent Isotropic Displacement Parameters for **3a**

S8 - Table S3, Bond Lengths and Angles for **3a**

S9 - Table S4, Anisotropic Displacement Parameters for **3a**

S10 - Table S5, Torsion Angles for **3a**

S11 - Table S6, Hydrogen Coordinates and Isotropic Displacement Parameters for **3a**

S12 - ¹H NMR Spectrum of (Z)-**2a**

S13 - ¹H NMR Spectrum of (Z)-**2b**

S14 - ¹H NMR Spectrum of (E,Z)-**2b**

S15 - ¹H NMR Spectrum of **2f**

S16 - ¹H NMR Spectrum of **3a**

S17 - ¹H NMR Spectrum of **3b**

S18 - ¹H NMR Spectrum of **3b**

S19 - ¹H NMR Spectrum of **3b**

S20 - ¹H NMR Spectrum of **3b**

S21 - ¹H NMR Spectrum of **3d**

S2 Rigid Core Vinamidinium Salts and Their N,N'-Rotamers

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Table of Contents for Supporting Information (continued):

S22 - ^1H NMR Spectrum of **3d**

S23 - ^1H NMR Spectrum of **3d**

S24 - ^1H NMR Spectrum of **3e**

S25 - ^1H NMR Spectrum of **3j**

S26 – Computational Values for Rotamers **3aA** and **3aB**

S3 – ORTEP Drawing for Cation 3a

ORTEP drawing of cation 3a; selected bond lengths (Å), bond angles (deg), and torsion angles (deg): C(1)-C(2), 1.377(6); C(2)-N(1), 1.324(5); C(1)-C(6), 1.382(6); C(6)-N(2), 1.310(6); C(9)-N(1), 1.459(6); C(2)-C(3), 1.501(6); C(2)-C(1)-C(6), 120.4(4); C(1)-C(2)-C(3), 121.2(4); C(1)-C(2)-N(1), 123.1(4); C(2)-N(1)-C(9), 124.8(4); C(3)-C(2)-N(1), 115.7(4); C(1)-C(2)-N(1)-C(9), 1.1(7); C(1)-C(6)-N(2)-C(10), 1.3(8); C(2)-C(1)-C(6)-N(2), 179.6(4); C(2)-C(1)-C(6)-C(5), 1.3(6); C(3)-C(2)-N(1)-C(9), -179.0(4); C(5)-C(6)-N(2)-C(10), 179.7(5). C(6)-C(1)-C(2)-N(1), 177.4(4); C(6)-C(1)-C(2)-C(3), -2.5(6).

DATA COLLECTION

A crystal of the compound was attached to a glass fiber and mounted on the Siemens SMART system for a data collection at 297(2) K. An initial set of cell constants was calculated from reflections harvested from three sets of 20 frames. These initial sets of frames are oriented such that orthogonal wedges of reciprocal space were surveyed. This produces orientation matrices determined from 123 reflections. Final cell constants are calculated from a set of 5414 strong reflections from the actual data collection. Please refer to Table 1 for additional crystal and refinement information.

The data collection technique used for this specimen is generally known as a hemisphere collection. Here a randomly oriented region of reciprocal space is surveyed to the extent of 1.3 hemispheres to a resolution of 0.84 Å. Three major swaths of frames are collected with 0.30° steps in ω . In the event the lattice is triclinic some additional sets of frames are collected to better model the absorption correction.

STRUCTURE SOLUTION AND REFINEMENT

The space group $P2_12_12_1$ was determined based on systematic absences and intensity statistics.¹ A successful direct-methods solution was calculated which provided most non-hydrogen atoms from the E-map. Several full-matrix least squares / difference Fourier cycles were performed which located the remainder of the non-hydrogen atoms. All non-hydrogen atoms were refined with anisotropic displacement parameters unless stated otherwise. All hydrogen atoms were placed in ideal positions and refined as riding atoms with group isotropic displacement parameters.

The structure was found as expected. The specimen appears to be a racemic twin that was successfully modeled. The cation is hydrogen bonded to the iodine anion by both NH groups. These hydrogen atoms were placed at chemically reasonable positions at 0.89 Å from the nitrogen.

Data collection and structure solution were conducted at the X-Ray Crystallographic Laboratory, 160 Kolthoff Hall, Chemistry Department, The University of Minnesota. All calculations were performed using SGI INDY R4400-SC or Pentium computers using the SHELXTL V5.0 suite of programs. All publications arising from this report MUST either 1) include Victor G. Young, Jr. as a coauthor or 2) acknowledge both Victor G. Young, Jr. and the X-Ray Crystallographic Laboratory.

¹ SHELXTL-Plus V5.0, Siemens Industrial Automation, Inc., Madison, WI.

Table 1. Crystal data, data collection, and solution and refinement for 98222a.

Crystal Data

Empirical formula	$C_{10}H_{19}IN_2$
Crystal Habit, color	Irregular block, Colorless
Crystal size	0.24 x 0.24 x 0.20 mm
Crystal system	Orthorhombic
Space group	$P2_12_12_1$
	$a = 5.8889(1) \text{ \AA} \quad \alpha = 90^\circ$
	$b = 13.2316(2) \text{ \AA} \quad \beta = 90^\circ$
	$c = 16.8908(1) \text{ \AA} \quad \gamma = 90^\circ$
Volume	$1316.12(3) \text{ \AA}^3$
Z	4
Formula weight	294.17
Density (calculated)	1.485 Mg/m^3
Absorption coefficient	2.400 mm^{-1}
F(000)	584

Data Collection

Diffractionmeter	Siemens SMART Platform CCD
Wavelength	$0.71073 \text{ \AA}$
Temperature	$297(2) \text{ K}$
θ range for data collection	1.96 to 25.16°
Index ranges	$-7 \leq h \leq 7, 0 \leq k \leq 15, 0 \leq l \leq 20$
Reflections collected	6674
Independent reflections	2329 ($R_{\text{int}} = 0.0210$)

Solution and Refinement

System used	SHELXTL-V5.0
Solution	Direct methods
Refinement method	Full-matrix least-squares on F^2
Weighting scheme	$w = [\sigma^2(F_o^2) + (AP)^2 + (BP)]^{-1}$, where $P = (F_o^2 + 2F_c^2)/3$, $A = 0.0308$, and $B = 1.0648$
Absorption correction	SADABS (Sheldrick, 1996)
Max. and min. transmission	1.000 and 0.649
Absolute structure parameter	$0.55(4)$
Data / restraints / parameters	2329 / 0 / 123
R indices ($I > 2\sigma(I)$)	$R1 = 0.0302, wR2 = 0.0734$
R indices (all data)	$R1 = 0.0344, wR2 = 0.0753$
Goodness-of-fit on F^2	1.083
Largest diff. peak and hole	0.763 and $-0.702 \text{ e\AA}^{-3}$

Table 2. Atomic coordinates [$x, 10^4$] and equivalent isotropic displacement parameters [$\text{\AA}^2 \times 10^3$] for 98222a. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)	SOF
I(1)	1731(1)	5568(1)	3530(1)	76(1)	1
C(1)	6312(7)	6110(3)	6498(3)	49(1)	1
C(2)	5454(7)	5839(3)	5770(3)	45(1)	1
C(3)	6479(9)	4991(3)	5301(3)	52(1)	1
C(4)	7577(7)	4180(3)	5823(3)	45(1)	1
C(5)	9164(7)	4711(3)	6402(3)	50(1)	1
C(6)	8093(8)	5576(3)	6828(2)	48(1)	1
C(7)	5765(9)	3593(4)	6272(3)	64(1)	1
C(8)	8935(9)	3466(4)	5300(3)	64(1)	1
N(1)	3698(6)	6298(3)	5438(2)	55(1)	1
C(9)	2473(9)	7149(4)	5783(4)	72(2)	1
N(2)	8974(7)	5807(3)	7516(2)	60(1)	1
C(10)	8188(14)	6624(4)	8022(3)	89(2)	1

Table 3. Bond lengths [Å] and angles [°] for 98222a.

C(1)-C(2)	1.377(6)	C(1)-C(6)	1.382(6)
C(2)-N(1)	1.324(5)	C(2)-C(3)	1.501(6)
C(3)-C(4)	1.532(6)	C(4)-C(8)	1.520(6)
C(4)-C(7)	1.522(6)	C(4)-C(5)	1.524(6)
C(5)-C(6)	1.492(6)	C(6)-N(2)	1.310(6)
N(1)-C(9)	1.459(6)	N(2)-C(10)	1.453(6)
<hr/>			
C(2)-C(1)-C(6)	120.4(4)	N(1)-C(2)-C(1)	123.1(4)
N(1)-C(2)-C(3)	115.7(4)	C(1)-C(2)-C(3)	121.2(4)
C(2)-C(3)-C(4)	112.9(4)	C(8)-C(4)-C(7)	110.0(4)
C(8)-C(4)-C(5)	109.7(4)	C(7)-C(4)-C(5)	110.2(4)
C(8)-C(4)-C(3)	108.8(4)	C(7)-C(4)-C(3)	110.4(4)
C(5)-C(4)-C(3)	107.8(4)	C(6)-C(5)-C(4)	113.8(4)
N(2)-C(6)-C(1)	122.6(4)	N(2)-C(6)-C(5)	116.1(4)
C(1)-C(6)-C(5)	121.2(4)	C(2)-N(1)-C(9)	124.8(4)
C(6)-N(2)-C(10)	124.7(4)		

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters [$\text{\AA}^2 \times 10^3$] for 98222a. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [(ha)^2 U_{11} + \dots + 2hka b U_{12}]$

	U11	U22	U33	U23	U13	U12
I (1)	72 (1)	104 (1)	53 (1)	-11 (1)	-1 (1)	19 (1)
C (1)	51 (2)	46 (2)	50 (2)	-8 (2)	1 (2)	5 (2)
C (2)	44 (2)	45 (2)	48 (2)	7 (2)	-1 (2)	-1 (2)
C (3)	55 (3)	60 (3)	40 (2)	-2 (2)	-2 (2)	5 (2)
C (4)	50 (2)	46 (2)	40 (2)	-2 (2)	0 (2)	1 (2)
C (5)	48 (2)	49 (2)	52 (3)	-4 (2)	-6 (2)	7 (2)
C (6)	51 (2)	46 (2)	46 (2)	-2 (2)	-4 (2)	-4 (2)
C (7)	73 (3)	49 (3)	70 (4)	-2 (2)	10 (3)	-12 (2)
C (8)	79 (4)	58 (3)	55 (3)	-9 (2)	7 (2)	16 (3)
N (1)	50 (2)	55 (2)	60 (2)	7 (2)	-9 (2)	2 (2)
C (9)	60 (3)	55 (3)	101 (5)	8 (3)	-10 (3)	13 (2)
N (2)	71 (3)	55 (2)	53 (2)	-14 (2)	-18 (2)	7 (2)
C (10)	124 (5)	83 (4)	61 (3)	-28 (3)	-19 (4)	14 (4)

Table 5. Torsion angles [$^{\circ}$] for 98222a.

C(6)-C(1)-C(2)-N(1)	177.4(4)	C(6)-C(1)-C(2)-C(3)	-2.5(6)
N(1)-C(2)-C(3)-C(4)	-151.0(4)	C(1)-C(2)-C(3)-C(4)	28.9(6)
C(2)-C(3)-C(4)-C(8)	-169.5(4)	C(2)-C(3)-C(4)-C(7)	69.8(5)
C(2)-C(3)-C(4)-C(5)	-50.6(5)	C(8)-C(4)-C(5)-C(6)	168.2(4)
C(7)-C(4)-C(5)-C(6)	-70.6(5)	C(3)-C(4)-C(5)-C(6)	49.8(5)
C(2)-C(1)-C(6)-N(2)	179.6(4)	C(2)-C(1)-C(6)-C(5)	1.3(6)
C(4)-C(5)-C(6)-N(2)	154.7(4)	C(4)-C(5)-C(6)-C(1)	-26.8(6)
C(1)-C(2)-N(1)-C(9)	1.1(7)	C(3)-C(2)-N(1)-C(9)	-179.0(4)
C(1)-C(6)-N(2)-C(10)	1.3(8)	C(5)-C(6)-N(2)-C(10)	179.7(5)

Symmetry transformations used to generate equivalent atoms:

Table 6. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 98222a.

	x	y	z	U(eq)	SOF
H(1B)	5689(7)	6655(3)	6769(3)	59	1
H(3A)	7617(9)	5264(3)	4945(3)	62	1
H(3B)	5305(9)	4680(3)	4980(3)	62	1
H(5A)	9693(7)	4222(3)	6789(3)	60	1
H(5B)	10479(7)	4956(3)	6115(3)	60	1
H(7A)	4672(35)	3329(24)	5905(4)	96	1
H(7B)	5018(44)	4035(8)	6640(16)	96	1
H(7C)	6461(12)	3045(17)	6555(18)	96	1
H(8A)	7914(10)	3068(20)	4985(16)	96	1
H(8B)	9839(49)	3028(20)	5626(3)	96	1
H(8C)	9910(48)	3852(4)	4959(16)	96	1
H(1A)	3247(6)	6078(3)	4985(2)	66	1
H(9A)	1208(44)	7322(21)	5450(14)	108	1
H(9B)	3473(22)	7720(11)	5826(23)	108	1
H(9C)	1930(62)	6967(12)	6299(11)	108	1
H(2A)	10099(7)	5450(3)	7681(2)	72	1
H(10A)	9064(60)	6633(25)	8501(14)	134	1
H(10B)	6615(28)	6522(21)	8148(25)	134	1
H(10C)	8366(81)	7257(5)	7751(12)	134	1

Current Data Parameters
NAME yd-1-96a-30
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 990206
Time 9.58
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 4464.286 Hz
FIDRES 0.137855 Hz
AQ 3.6270580 sec
RG 64
DM 112.000 us
DE 160.00 us
TE 300.0 K
D1 0.5000000 sec
P1 8.50 us
DE 160.00 us
SF01 300.1312405 MHz
NUCLEUS 1H

F2 - Processing parameters

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WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 20.00

1D NMR plot parameters

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F1 3421.62 Hz
F2P -0.172 ppm
F2 -51.60 Hz
PPMCH 0.52602 ppm
HZCM 157.87378 Hz

Current Data Parameters
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EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
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Time 16.27
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PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 4164.286 Hz
FIDRES 0.137855 Hz
AQ 3.6270580 s
RG 64
DM 112.000 us
DE 160.00 us
TE 300.0 K
D1 0.5000000 s
P1 8.50 us
DE 160.00 us
SF01 300.1312405 MHz
NUCLEUS 1H
F2 - Processing parameters
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WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 20.00
1D NMR plot parameters
CX 22.00 cm
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F1 2624.86 Hz
F2P -0.248 ps
F2 -74.56 Hz
PPMCH 0.40883 ps
HZCN 122.70187 Hz

Current Data Parameters
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EXPNO 1
PROCNO 1

F2 - Acquisition Parameters:

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PROBHD 5 mm QNP 1H
PULPROG zg30
TO 32384
SOLVENT CDCl3
NS 8
DS 0
SWH 4464.286 Hz
FIDRES 0.137855 Hz
AQ 3.6270580 sec
RG 64
DW 112.000 usec
DE 150.00 usec
TE 300.0 K
D1 0.50000000 sec
P1 8.50 usec
DE 160.00 usec
SF01 300.1312405 MHz
NUCLEUS 1H

F2 - Processing parameters

SI 16384
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WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 20.00

1D NMR plot parameters

CX 22.00 cm
F1P 9.250 ppm
F1 2776.20 Hz
F2P -0.250 ppm
F2 -75.03 Hz
PPMCH 0.43182 ppm
HZCM 129.60158 Hz

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EXPNO 11
PROCNO 1

F2 - Acquisition Parameters

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SOLVENT CDCl3
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SWH 4132.231 Hz
FIDRES 0.252211 Hz
AQ 1.9925140 sec
RG 715
DM 121.000 use
DE 151.25 use
TE 300.0 K
D1 0.50000000 sec
P1 8.50 use
SFO1 300.1319180 MHz
NUCLEUS 1H

F2 - Processing parameters

SI 16384
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WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 20.00

1D NMR plot parameters

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F1 3383.49 Hz
F2P 6.133 ppm
F2 1840.68 Hz
PPMCM 0.21419 ppm/
HZCM 64.26380 Hz/c

Current Data Parameters
 NAME SW-1-B4-41
 EXPNO 11
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20000315
 Time 14.19
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 PROBHD 5 mm QNP 1H
 PULPROG zg30
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 SOLVENT COC13
 NS 16
 DS 0
 SWH 4132.231 Hz
 FIDRES 0.252211 Hz
 AQ 1.9825140 sec
 RG 715
 DW 121.000 usec
 DE 151.25 usec
 TE 300.0 K
 D1 0.5000000 sec
 P1 8.50 usec
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 NUCLEUS 1H

F2 - Processing parameters
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 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 20.00

1D NMR plot parameters
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 F1 998.39 Hz
 F2P 2.201 ppm
 F2 660.64 Hz
 PPMCM 0.04689 ppm
 HZCM 14.07294 Hz/

3.002
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2.689
2.595

ppm

Current Data Parameters
 NAME SW-1-B4-41
 EXPNO 11
 PROCNO 1

F2 - Acquisition Parameters
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 PROBHD 5 mm QNP 1H
 PULPROG zg30
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 SOLVENT CDCl3
 NS 16
 DS 0
 SMH 4132.231 Hz
 FIDRES 0.252211 Hz
 AQ 1.9825140 sec
 RG 715
 DW 121.000 usec
 DE 151.25 usec
 TE 300.0 K
 D1 0.50000000 sec
 P1 8.50 usec
 SF01 300.1315180 MHz
 NUCLEUS 1H

F2 - Processing parameters
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 SF 300.1299918 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 20.00

1D NMR plot parameters
 CX 24.00 cm
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 F1 523.04 Hz
 F2P -0.091 ppm
 F2 -27.37 Hz
 PPMCM 0.07641 ppm
 HZCM 22.93368 Hz/

1.06

1.54
1.52

ppm

ppm 1.6 1.4 1.2 1.0 0.8 0.6 0.4 0.2 0.0

Current Data Parameters
 NAME SW-1-89-34
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 PROCNO 1

F2 - Acquisition Parameters
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 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TO 16384
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 4132.231 Hz
 FIDRES 0.252211 Hz
 AQ 1.9825140 sec
 RG 715
 DM 121.000 usec
 DE 151.25 usec
 TE 300.2 K
 D1 0.50000000 sec
 P1 8.50 usec
 SF01 300.1319180 MHz
 NUCLEUS 1H

F2 - Processing parameters
 SI 16384
 SF 300.1299971 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 20.00

1D NMR plot parameters
 CX 24.00 cm
 FIP 9.769 ppm
 F1 2931.98 Hz
 F2 1710.24 Hz
 PPMCM 0.16961 ppm/
 HZCM 50.90582 Hz/c

7.290
 7.315
 7.327

ppm

Current Data Parameters
 NAME SW-1-B9-34
 EXPNO 26
 PROCNO 1

F2 - Acquisition Parameters
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 Time 20.05
 INSTRUM arx300
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TD 64000
 SOLVENT CDCl3
 NS 120
 DS 0
 SWH 4132.231 Hz
 FIDRES 0.064566 Hz
 AQ 7.7440500 sec
 RG 715
 DW 121.000 usec
 DE 151.25 usec
 TE 300.0 K
 D1 0.5000000 sec
 P1 8.50 usec
 SF01 300.1319180 MHz
 NUCLEUS 1H

F2 - Processing parameters
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 SF 300.1299971 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 20.00

1D NMR plot parameters
 CX 24.00 cm
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 F1 963.87 Hz
 F2P 1.947 ppm
 F2 584.42 Hz
 PPMCM 0.05268 ppm
 HZCM 15.81034 Hz

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 2.778
 2.622
 2.524

Current Data Parameters
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EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
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PROBHD 5 mm Multinu
PULPROG zg30
TO 16384
SOLVENT COC13
NS 16
DS 0
SMH 3906.250 Hz
FIDRES 0.238419 Hz
AQ 2.0972021 sec
RG 1430
DM 128.000 usec
DE 160.00 usec
TE 300.0 K
D1 0.5000000 sec
P1 8.50 usec
DE 160.00 usec
SF01 300.1318608 MHz
NUCLEUS 1H

F2 - Processing parameters
SI 16384
SF 300.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 20.00

1D NMR plot parameters
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F1P 12.700 ppm
F1 3811.65 Hz
F2P -0.300 ppm
F2 -90.04 Hz
PPHCH 0.54167 ppm/
HZCM 162.57042 Hz/c

SW-1-48-39

Current Data Parameters
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EXPNO 1
PROCNO 1

F2 - Acquisition Parameters:

Date_ 990206
Time 11.13
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PULPROG zgpg30
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NS 8
DS 0
SWH 4464.286 Hz
FIDRES 0.137855 Hz
AQ 3.6270580 sec
RG 64
DE 112.000 us
TE 150.00 us
TE 300.0 K
D1 0.50000000 sec
P1 8.50 us
DE 160.00 us
SFO1 300.1312405 MHz
NUCLEUS 1H

F2 - Processing parameters
SI 16384
SF 300.1299693 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 20.00

1D NMR plot parameters
CX 22.00 cm
F1P 8.746 ppb
F1 2624.86 Hz
F2P -0.248 ppb
F2 -74.58 Hz
PPMCH 0.40883 ppb
HZCM 122.70187 Hz

2.540
2.139
2.111
2.099
2.087
2.061
1.164

3.724
3.703
3.682
3.606
3.585
3.564

5.065

ppm

S26 – Computational Values for Rotamers **3aA** and **3aB**

PC Spartan Version 1.06 Ab-Initio Program Hartree-Fock 3-21G* at 298 K

Category	Rotamer 3aA	Rotamer 3aB	Difference
HF Point Group	C1	C1	
Potential Energy	-495.7193483 a.u.	-495.7167811 a.u.	0.0025672 a.u., 1.611 kcal/mol
Zero Pt. Vibr. E	191.101 kcal/mol	191.137 kcal/mol	0.026 kcal/mol
Translat. Enthalpy	0.889 kcal/mol	0.889 kcal/mol	none
Rotat. Enthalpy	0.889 kcal/mol	0.889 kcal/mol	none
Vibrat. Enthalpy	197.268 kcal/mol	197.223 kcal/mol	0.045 kcal/mol
Translat. Entropy	41.249 cal/mol.K	41.249 cal/mol.K	none
Rotat. Entropy	31.064 cal/mol.K	31.085 cal/mol.K	0.021 cal/mol.K
Vibrat. Entropy	36.965 cal/mol.K	35.542 cal/mol.K	1.423 cal/mol.K
Degeneracy	none	two equivalent forms	1.378 cal/mol.K

For **3aB** → **3aA**, the relevant thermodynamic changes at 298 K are computed to be:

$$\Delta H^0_{(298\text{ K})} = (-1.611 - 0.026 + 0.045) \text{ kcal/mol} = -1.630 \text{ kcal/mol}$$

$$\Delta S^0_{(298\text{ K})} = (-0.021 + 1.423 - 1.378) \text{ cal/mol.K} = 0.024 \text{ cal/mol.K}$$

$$\Delta G^0_{(298\text{ K})} = -1.630 \text{ kcal/mol} - 298\text{K}(0.024)\text{cal/mol.K} = -1.637 \text{ kcal/mol}$$

$K_{\text{eq}} = 16$, the proportions being **94% 3aA** and **6% 3aB**