### SUPPORTING INFORMATION

## Stereochemistry of a Second Riolozane and Other Diterpenoids from

### Jatropha dioica

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#### **Table of Contents**

**S1** 

**S2** 

**S3** 

**S4** 

**S5** 

**S6** 

**S7** 

**S8** 

**S9** 

**S10** 

**S11** 

S12

**S13** 

**S14** 

Chromatograms obtained from HPLC analysis for hexane fraction (A) and methylene chloride extract (B) from <i>J. dioica</i> roots	4
Elemental Analysis of 6-epi-Riolozatrione (2)	5
HRESIMS of 6- <i>epi</i> -Riolozatrione (2)	6
Infrared Spectra of 6-epi-Riolozatrione (2)	7
Experimental <sup>1</sup> H NMR (400 MHz) and <sup>13</sup> C NMR (100 MHz) spectra of $1$ and $2$ in CDCl <sub>3</sub> .	8
Comparison of the 1D <sup>1</sup> H NMR spectra of compounds <b>1</b> and <b>2</b> in CD <sub>3</sub> OD at 700 MHz, FIDs processed with Lorentzian-Gaussian apodization	9
Comparison of the ${}^{1}$ H NMR signals of H-12 in compounds <b>1</b> and <b>2</b> at 400 and 750 MHz	13
Comparison of the 1D $^{13}$ C NMR spectra of compounds 1 and 2 in CD <sub>3</sub> OD at 100 MHz.	14
HSQC spectrum (400 MHz, CD <sub>3</sub> OD) of 6- <i>epi</i> -Riolozatrione (2)	15
HMBC spectrum (400 MHz, CD <sub>3</sub> OD) of 6- <i>epi</i> -Riolozatrione (2)	16
NOESY spectrum (400 MHz, CD <sub>3</sub> OD) of 6-epi-Riolozatrione (2)	17
Calculated vs. experimental 1D $^{1}$ H-NMR spectra of compounds 1 and 2 in CD <sub>3</sub> OD at 700 MHz	18
$^{1}$ H NMR HiFSA data for Riolozatrione (1) in CD <sub>3</sub> OD (PERCH .pms file format)	19
<sup>1</sup> H NMR HiFSA data for 6- <i>epi</i> -Riolozatrione (2) in CD <sub>3</sub> OD (PERCH .pms file format)	20

- **S15** <sup>1</sup>H NMR HiFSA data for Riolozatrione (1) in CDCl<sub>3</sub> (PERCH .pms file 21 format)
- **S16** <sup>1</sup>H NMR HiFSA data for 6-*epi*-Riolozatrione (**2**) in CDCl<sub>3</sub> (PERCH .pms 22 file format)

- **S17** Full <sup>1</sup>H NMR  $\delta$  and *J*-correlation maps, termed Quantum Interaction and 23 Linkage Tables (QuILTs), of riolozatrione (1) and 6-*epi*-riolozatrione (2) were achieved by HiFSA processing of the 700 MHz spectra in CDCl<sub>3</sub>.
- **S18** Calculated (red) vs. experimental (blue) and difference (green) 1D <sup>1</sup>H-NMR 24 signals in compounds **1** and **2** in CDCl<sub>3</sub> at 700 MHz.
- **S19** Experimental and calculated IR and VCD spectra at the B3PW91/DGDZVP 25 level of theory for **3** and **4**.

**Figure S1**. Chromatograms obtained from HPLC analysis for hexane fraction (A) and methylene chloride extract (B) from *J. dioica* roots.



HPLC Analysis Method of obtained extracts from J. dioica roots.

HPLC Method. The HPLC analysis of extracts A and B was performed on a Waters liquid chromatograph 1525 linked to a Waters diode array detector 2996, using a Waters AccQ-Tag column and isocratic elution with acetonitrile-water (50:50).

n-Hexane fraction (A): Hydro-methanolic extract of *J. dioica* roots (250 g) at RT, was evaporated at reduced pressure at 40 °C, and subsequent partitioned to obtain fractions of n-hexane, ethyl acetate, and butyl alcohol. DCM extract (B): Dried and powdered roots of *J. dioica* (250 g) were extracted with DCM (3 x 1L) at room temperature to give 3.5 g of concentrated crude extract at reduced pressure.

			nalisis Ele	emental po	or Combu	stión	C,
Investig	ador solicitant	IN e: Dr. Gab	IFORME D oriel E. Cue	E ANÁLISI: evas Gonzá	S lez Bravo		
No. de	Clave de la	Peso	N [%]	C [%]	H [%]	S [%]	Fecha de
39	ISO-1	2.047		76.13	8.21		24-02-2016
Control Se utiliz Thermo = 7.38 %	<b>de calidad:</b> tó el material o Scientific. El pro 5. Los valores te	de referen omedio de óricos son	ncia metior los valores : N = 9.39 <sup>(</sup>	ina como obtenidos %, C = 40.2	estándar es: N = 9.4 5 % y H = 1	de verific 45 %, C = 7.43 %.	ación, marca ⊧ 40.43 % y ⊦
Respon	sable del anális	sis: Q. Ma	ría de la Pa	az Orta Pére	Ξ		
Fecha d	e informe: 7 de	marzo de	2016.				

# Figure S2. Elemental analysis of 6-*epi*-Riolozatrione (2)

#### Figure S3. HRESIMS spectrum of 6-epi-Riolozatrione (2)



	Mass	Intensity	Calc. Mass	Mass Difference (mmu)	Mass Difference (ppm)	Possible Formula	Unsaturation Number
ĺ	315.19650	1168548.88	315.19602	0.48	1.54	12C201H2716O3	7.

Figure S4. Infrared spectrum of 6-*epi*-Riolozatrione (2)





Figure S5. Experimental <sup>1</sup>H NMR (400 MHz) and <sup>13</sup>C NMR (100 MHz) spectra of 1 and 2 in  $CDCl_3$ .

Figure S6. Comparison of the 1D  $^{1}$ H-NMR spectra of compounds 1 and 2 in CD<sub>3</sub>OD at 700 MHz, FIDs processed with Lorentzian-Gaussian apodization.









**Figure S7.** Comparison of the <sup>1</sup>H-NMR signals of the H-12 proton in compound **1** at 400 and 750 MHz and compound **2** at 750 MHz. Only in **1**, the highly coupled vicinal methylene protons, 2H-13, form an AB pair with very close chemical shifts ( $\Delta\delta$ =0.0014ppm). Therefore, H-12 in **1** gives rise to a higher order resonance pattern with a ddd-like multiplicity that can be readily misinterpreted when working under first-order assumptions. In contrast, as the methylene protons, 2H-13, resonate as relatively distant AM/AX nuclei in **2**, H-12 gives rise to an apparent triplet (pseudo triplet; with two additional long-range couplings). In fact, as the 6-*epi* relationship between **1** and **2** does essentially not affect the geometry of the six-membered ring, its coupling patters have to be identical in both molecules – only the resonances are not, due to the higher order effects caused by the differences in *relative* chemical shifts.



Figure S8. Comparison of the 100 MHz 1D <sup>13</sup>C-NMR spectra of compounds 1 and 2 in CD<sub>3</sub>OD.





Figure S9. HSQC spectrum (400 MHz, CD<sub>3</sub>OD) of 6-*epi*-Riolozatrione (2).



## Figure S10. HMBC spectrum (400 MHz, CD<sub>3</sub>OD) of 6-*epi*-Riolozatrione (2).



### Figure S11. NOESY spectrum (400 MHz, CD<sub>3</sub>OD) of 6-*epi*-Riolozatrione (2).

**Figure S12.** Calculated (red) vs. experimental (blue) and difference (green) 1D <sup>1</sup>H-NMR signal of H-1b in compound 1 in CD<sub>3</sub>OD at 700 MHz.



# **Figure S13**. <sup>1</sup>H NMR HiFSA data for Riolozatrione (1) in CD<sub>3</sub>OD (PERCH .pms file format)

ACTIVE S	SPECIES:1H		
CHEMICAI	_ SHIFTS(PPM):	FIT-INDEX=	= 28.957
PROTON 2	*SPIN=1 SPECI	ES=1H PC	)PULATION= 1.0000
Н7 /1	3.378440	1*1*1 STA	AT=Y PRED= 3.159 RANGE= 0.474 WIDTH= 1.033 RESP= 1.0000 HSQC=C7
H12 /1	1.710480	1*1*1 STA	AT=Y PRED= 1.558 RANGE= 0.411 WIDTH= 0.609 RESP= 1.0000 HSQC=C12
H15 /1	1.939020	1*1*1 STA	AT=Y PRED= 1.832 RANGE= 0.462 WIDTH= 0.876 RESP= 1.0000 HSQC=C15
H6 /1	2.337510	1*1*1 STA	AT=Y PRED= 2.872 RANGE= 0.468 WIDTH= 0.578 RESP= 1.0000 HSQC=C6
H1A /1	2.238770	1*1*1 STA	AT=Y PRED= 1.678 RANGE= 0.451 WIDTH= 0.521 RESP= 1.0000 HSQC=C1
H1B /1	2.563580	1*1*1 STA	AT=Y PRED= 2.328 RANGE= 0.444 WIDTH= 0.585 RESP= 1.0000 HSQC=C1
H2 /1	2.776340	1*1*1 STA	AT=Y PRED= 2.460 RANGE= 0.441 WIDTH= 0.483 RESP= 1.0000 HSQC=C2
H3A /1	1.907940	1*1*1 STA	AT=Y PRED= 2.069 RANGE= 0.391 WIDTH= 0.442 RESP= 1.0000 HSQC=C3
H3B /1	2.462910	1*1*1 STA	AT=Y PRED= 2.460 RANGE= 0.370 WIDTH= 0.506 RESP= 1.0000 HSQC=C3
H19 /1	1.140890	1*1*3 STA	AT=Y PRED= 1.058 RANGE= 0.264 WIDTH= 0.500 RESP= 1.0000 HSQC=C19
H13A/1	3.043140	1*1*1 STA	AT=Y PRED= 3.063 RANGE= 0.407 WIDTH= 0.653 RESP= 1.0000 HSQC=C13
H13B/1	2.838660	1*1*1 STA	AT=Y PRED= 2.343 RANGE= 0.416 WIDTH= 0.956 RESP= 1.0000 HSQC=C13
H16 /1	1.222410	1*1*3 STA	AT=Y PRED= 1.221 RANGE= 0.216 WIDTH= 0.750 RESP= 1.0000 HSQC=C16
H17 /1	0.789750	1*1*3 STA	AT=Y PRED= 0.965 RANGE= 0.332 WIDTH= 0.562 RESP= 1.0000 HSQC=C17
H18 /1	0.967460	1*1*3 STA	AT=Y PRED= 1.183 RANGE= 0.392 WIDTH= 0.676 RESP= 1.0000 HSQC=C18
H20 /1	1.127430	1*1*3 STA	AT=Y PRED= 1.173 RANGE= 0.269 WIDTH= 0.410 RESP= 1.0000 HSQC=C20
COUPLING	CONSTANTS (HZ	): FIT-INDE	X= 0.000
JI0_27	1.0900 J		SIAI-I PRED- J.029 RANGE- J.222
.T10 20	1 6967 T	ил пін 117 цір	STATET TABLE -0.413 RANGE 5.000 STATET DEFDE -0.424 DANGE 5.000
JI0_29 T10_21	1.000/ J		STAT-T FRED0.424 RANGE- J.000
JI0_JI	2.2395 J		STAT-I FRED- 0.000 RANGE- 0.001
JIU_32 T22_24	2.044/ J 7.7001 T	п/ пзв и12 и15	SIAI-I FRED- 0.000 RANGE- 0.001
JZJ_Z4 T22_26	7.7901 0	HI2 HI3	STAT-I FRED- 10.333 RANGE- 2.000
123 30	0.9270 J	U12 U13A	SIAI-I PRED- 9.057 RANGE- 5.000
JZJ_J/	0.9020 J	NIZ NIJA NIS NIJA	SIAI-I PRED- 1.110 RANGE- 5.000
J24_30	-0.2019 J	UIE UID	STAT-I FRED- 0.000 RANGE- 0.001
127 47	-0.3000 J	NE U20	STAT-1 FRED- 0.000 RANGE- 0.001
127 29	0 7750 T	110 1120 116 1117	STAT-1 FRED- 7.052 RANGE- 2.257
127 20	0.7/50 0	110 111A 116 111P	STAT-1 FRED- 0.000 RANGE- 0.001
727 31	0.7439 U	110 111D	STAT-1 FRED- 0.000 RANGE- 0.001
727 22	0.2020 0	ILC ILCD	STAT-1 FRED- 0.000 RANGE- 0.001
129 20	_10 7214 T	110 113B 117 117D	STAT-1 FRED- 0.000 RANGE- 0.001
JZ0_20	6 4 95 4 J	H1A H2	STAT=Y DEED= 1 976 DANGE= 3 218
JZ0_30	2 0396 J	H1A H3A	STAT=Y DEED= 1 737 BANGE= 1 000
.128 32	2.0349 J	H1A H3B	STAT=Y DEED= 2 066 DANGE= 1.000
JT29 30	8 2098 J	H1B H2	STAT=Y PRED= $7 227$ RANGE= 4 500
.T29 31	2 0738 J	H1B H3A	STAT=Y PRED= $2.072$ RANGE= $1.000$
129 32	1 0952 J	H1B H3B	STAT=Y PRED= $1.709$ RANGE= $1.763$
JT30 33	6 9495 J	H2 H19	STAT=Y PRED= $7 051$ RANGE= $1 000$
JT30 31	6 4032 J	H2 H3A	STAT=Y PRED= $3938$ RANGE= $2611$
J30 32	8 4003 J	H2 H3B	STAT=Y PRED= $10$ 316 RANGE= 2 853
J31 32	-15 9028 J	H3A H3B	STAT=Y PRED= $-16$ 751 RANGE= 3 272
J36 37	-18.5145 J	H13A H13B	STAT=Y PRED= -16.154 RANGE= 1.303
CONTROL 8 REF 0.00 2 699.95 0.00 0.00 0.00 0.00 0.000	PARAMETERS: COLVENT = None TERENCE = TMS 0000000 = CONC 98.000 = TEML 0430000 = FIEL 0100000 = Mini 0000000 = Left 0000000 = Righ 0.410 = Defa 0000000 = Data	ENTRATION ERATURE (vo D(1H,MHz), mum line-ir onalizatior frequency t frequency ult line-wi -point reso	<pre>(vol%) )1%, def=1.0%) used to transform shifts to ppms ntensity n criterium (not used) limit(ppm) / limit(ppm) idth(Hz) blution(Hz)</pre>
0.00	0.000 = Gaus 0.000 = Disp 0000000 = Deco	sian contri ersion cont upling free	.bution% :ribution% quency (for DORES only)
CONSTRAI	INTS (in equat	ions X0 = 1	1.0)use no empty lines
END of E	TILE		

# **Figure S14**. <sup>1</sup>H NMR HiFSA data for 6-*epi*-Riolozatrione (**2**) in CD<sub>3</sub>OD (PERCH .pms file format)

ACTIVE SP	PECIES:1H			
CHEMICAL	SHIFTS (PPM) :	FIT-INDEX=	14.469	
PROTON 2*	SPIN=1 SPECIE	ES=1H PO	PULATION= 1.0000	
H7A /1	4.432000	1*1*1 STA	T=Y PRED= 3.575 RANGE= 0.470 WIDTH= 1.653 RESP= 1.0000 HSQC:	=C7
H12A/1	1.701460	1*1*1 STA	T=Y PRED= 1.508 RANGE= 0.411 WIDTH= 1.014 RESP= 1.0000 HSQC:	=C12
H15A/1	1.967950	1*1*1 STA	T=Y PRED= 1.809 RANGE= 0.459 WIDTH= 0.946 RESP= 1.0000 HSQC:	=C15
H6A /1	3.056290	1*1*1 STA	T=Y PRED= 3.203 RANGE= 0.435 WIDTH= 0.650 RESP= 1.0000 HSQC:	=C6
H1A /1	2.098740	1*1*1 STA	T=Y PRED= 2.012 RANGE= 0.455 WIDTH= 0.811 RESP= 1.0000 HSQC:	=C1
H1B /1	2.592310	1*1*1 STA	T=Y PRED= 2.186 RANGE= 0.432 WIDTH= 0.829 RESP= 1.0000 HSQC:	=C1
H2A /1	2.784900	1*1*1 STA	T=Y PRED= 2.317 RANGE= 0.415 WIDTH= 0.376 RESP= 1.0000 HSQC:	=C2
H3A /1	1.936890	1*1*1 STA	T=Y PRED= 1.990 RANGE= 0.387 WIDTH= 0.495 RESP= 1.0000 HSQC:	=C3
НЗВ /1	2.509160	1*1*1 STA	T=Y PRED= 2.566 RANGE= 0.380 WIDTH= 0.449 RESP= 1.0000 HSQC:	=C3
Н19 /1	1.138690	1*1*3 STA	T=Y PRED= 1.063 RANGE= 0.230 WIDTH= 0.522 RESP= 1.0000 HSQC:	=C19
H13A/1	3.349960	1*1*1 STA	T=Y PRED= 3.077 RANGE= 0.424 WIDTH= 1.410 RESP= 1.0000 HSQC:	=C13
H13B/1	2.848410	1*1*1 STA	T=Y PRED= 2.336 RANGE= 0.438 WIDTH= 1.286 RESP= 1.0000 HSQC:	=C13
H17 /1	0.906450	1*1*3 STA	T=Y PRED= 1.222 RANGE= 0.216 WIDTH= 1.158 RESP= 1.0000 HSQC:	=C17
Н16 /1	0.733900	1*1*3 STA	T=Y PRED= 0.881 RANGE= 0.351 WIDTH= 0.971 RESP= 1.0000 HSQC:	=C16
H18 /1	1.207750	1*1*3 STA	T=Y PRED= 1.255 RANGE= 0.414 WIDTH= 0.486 RESP= 1.0000 HSQC:	=C18
H20 /1	0.989150	1*1*3 STA	T=Y PRED= 1.169 RANGE= 0.359 WIDTH= 0.567 RESP= 1.0000 HSQC:	=C20
COUPLING J10_27 J10 28	CONSTANTS(HZ) 6.1386 J 1.3117 J	: FIT-INDE H7A H6A H7A H1A	X= 0.000 STAT=Y PRED= 7.606 RANGE= 3.326 STAT=Y PRED= -0.351 RANGE= 5.000	
J10 <sup>2</sup> 9	1.6114 J	H7A H1B	STAT=Y PRED= -0.432 RANGE= 5.000	
J10 31	2.2248 J	Н7А НЗА	STAT=Y PRED= 0.000 RANGE= 0.001	
J10 <sup>32</sup>	2.7617 J	Н7А НЗВ	STAT=Y PRED= 0.000 RANGE= 0.001	
J10_30	0.5000 J	H7A H2A	STAT=Y PRED= 0.000 RANGE= 0.001	
J10_44	-0.4145 J	H7A H18	STAT=Y PRED= 0.000 RANGE= 0.001	
J23_24	7.9358 J	H12A H15A	STAT=Y PRED= 10.333 RANGE= 2.000	
J23_37	0.6424 J	H12A H13B	STAT=Y PRED= 0.269 RANGE= 5.000	
J23_36	8.4397 J	H12A H13A	STAT=Y PRED= 8.850 RANGE= 5.000	
J27_47	7.7129 J	H6A H2O	STAT=Y PRED= 7.032 RANGE= 2.237	
J27_28	0.9992 J	H6A HIA	STAT=Y PRED= 0.000 RANGE= 0.001	
JZI_Z9	0.9352 J	HOA HIB	STATEY PREDE 0.000 RANGEE 0.001	
JZ7_JZ	0.2720 0	HUA HID	STAT-I PRED- 0.000 RANGE- 0.001	
JZ7_30	-18 5844 J	HIA HIR	STAT-Y PRED15 884 PANCE- 2 632	
JT28 30	6 3365 J	H1A H2A	STAT=Y PRED= $7.062$ RANGE= $4.500$	
J28_31	1.9488 J	HIA H3A	STAT=Y PRED= 1.716 RANGE= 1.758	
J28 32	2.0035 J	H1A H3B	STAT=Y PRED= 2.072 RANGE= 1.000	
J29 30	8.2174 J	H1B H2A	STAT=Y PRED= 7.210 RANGE= 4.500	
J29 31	2.0716 J	H1B H3A	STAT=Y PRED= 2.069 RANGE= 1.000	
J29 32	1.1656 J	H1B H3B	STAT=Y PRED= 1.750 RANGE= 1.000	
J30 33	6.9555 J	H2A H19	STAT=Y PRED= 7.051 RANGE= 1.000	
J30 31	6.3205 J	Н2А НЗА	STAT=Y PRED= 7.100 RANGE= 2.288	
J30 <sup>32</sup>	8.3159 J	H2A H3B	STAT=Y PRED= 10.313 RANGE= 2.855	
J31 <sup>32</sup>	-15.8538 J	НЗА НЗВ	STAT=Y PRED= -16.750 RANGE= 3.273	
J36_37	-16.9872 J	H13A H13B	STAT=Y PRED= -16.158 RANGE= 1.302	
CONTROL F SC REFE 0.000 29 699.954 0.001 0.001 0.000 0.000 0.000 0.000 0.000	PARAMETERS: DIVENT = None RENCE = TMS 000000 = CONCH 8.000 = TEMP 30000 = FIELI 00000 = Diago 000000 = Left 000000 = Left 000000 = Data 0.000 = Gauss 0.000 = Dispo 000000 = Decou	ENTRATION ( ERATURE (vo D(1H, MHz), num line-in onalization frequency frequency ilt line-wi point reso sian contri arsion cont upling freq	<pre>vol%) 1%, def=1.0%) used to transform shifts to ppms tensity criterium (not used) limit(ppm) limit(ppm) dth(Hz) lution(Hz) bution% ribution% uency (for DORES only) 0) use percents lines;</pre>	
END of FI	LE	10115 AU = 1	.v,use no empty iines:	

# **Figure S15**. <sup>1</sup>H NMR HiFSA data for Riolozatrione (1) in CDCl<sub>3</sub> (PERCH .pms file format)

ACTIVE S	PECIES:1H				
CHEMICAL	SHIFTS(PE	PM):			
PROTON	2*SPIN=	1 SPECI	ES=1H	POPULA	TION(Y) = 0.80740
Н7	/ 1 3.	.126458	1*1*1	STAT=Y	PRED= 3.125 RANGE= 0.073 WIDTH(Y)= 1.324 RESP(Y)= 1.0000
H12	/1 1.	.614077	1*1*1	STAT=Y	PRED= 1.614 RANGE= $0.073$ WIDTH(Y) = $0.892$ RESP(Y) = 1.0000
H15	/1 1.	.883196	1*1*1	STAT=Y	PRED= 1.883 RANGE= 0.073 WIDTH(Y)= 0.646 RESP(Y)= 1.0000
H6	/1 2.	.456355	1*1*1	STAT=Y	PRED= 2.457 RANGE= 0.073 WIDTH(Y) = 1.023 RESP(Y) = 1.0000
HIA	/1 2.	.310340	1*1*1	STAT=Y	PRED= 2.310 RANGE= $0.073$ WIDTH(Y) = $0.878$ RESP(Y) = $1.0000$
HIB	/ 1 2.	.485253	1 * 1 * 1	STAT=Y	PRED= 2.483 RANGE= $0.073$ WIDTH(Y) = $0.726$ RESP(Y) = $1.0000$
HZ	/ 1 2.	./53404	1 ~ 1 ~ 1	STAT=1	PRED= 2.755 RANGE= $0.073$ WIDTH(Y) = $0.940$ RESP(Y) = 1.0000
HJA	/ 1 1.	.901866	1*1*1	STAT=1	PRED= 1.961 RANGE= $0.073$ WIDTH(Y) = $0.907$ RESP(Y) = 1.0000
пэр 110	/ 1 2.	153530	1*1*3	SIAI-I CTAT-V	PRED- 2.497 RANGE- 0.073 WIDIH(I) - 0.910 RESP(I) - 1.0000 DPED- 1.155 $PANCE$ 0.073 WIDTH(V) - 0.996 $PESP(V)$ - 1.0000
п13л u13л	/ 1 1.	002150	1*1*1	SIAI-I CTAT-V	PRED- 1.155 RANGE- 0.075 WIDIH(I)- 0.000 RESP(I)- 1.0000
H13B	/ 1 2.	884841	1 * 1 * 1	STAT-I STAT-V	PRED = 2.800 RANGE = 0.073 WIDTH(1) = 0.928 RESP(1) = 1.0000 PRED = 2.878 PANCE = 0.073 WIDTH(Y) = 1.124 PESP(Y) = 1.0000
H16	/ 1 2.	235937	1*1*3	STAT-I STAT-V	PRED = 2.070 RANGE = 0.073 WIDTH(1) = 1.124 RESP(1) = 1.0000 PRED = 1.235 PANCE = 0.073 WIDTH(Y) = 1.143 PESP(Y) = 1.0000
H17	/ 1 0	852886	1*1*3	STAT-I STAT-V	PRED= 0.853 PANGE= 0.073 WIDTH(Y) = 0.933 PESP(Y) = 1.0000
н18	/ 1 1	088124	1*1*3	STAT=1 STAT=Y	PRED= 1 089 RANGE= 0.073 WIDTH(Y) = 1 031 RESP(Y) = 1 0000
H20	/ 1 1	157132	1*1*3	STAT=1 STAT=Y	PRED= 1.000 KANGE= 0.073 WIDTH(Y) = 0.846 RESP(Y) = 1.0000 PRED= 1.157 RANGE= 0.073 WIDTH(Y) = 0.846 RESP(Y) = 1.0000
112.0	/ 1 1.	.13/132	1.1.2	SIAI-I	PRED- 1.137 RANGE- 0.075 WIDIN(1)- 0.040 RESP(1)- 1.0000
COUPLING	CONSTANTS	S(H7) ·			
.T10 27	2 000	асти асти	7	н6	STAT=Y PRED= 1 896 RANGE= 0 100
T10 29	1 375	70 0 11 70 T 11	7	110 111 h	STAT-1 TRED- 1.000 RANGE- 0.100
T10 20	1 597	79 0 II 71 TU	7	U1D	STAT-1 FRED- 1.557 RANGE- 0.100
T10 31	2 201	/1 U II 10 T II	7	1110	STAT-1 FRED- 1.007 RANGE- 0.100
.T10 32	2 846	сэ он сэ ти	7	H3B	STAT=Y PRED= 2 845 PANCE= 0 100
.123 24	7 682	ол 21 ти	12	H15	STAT=Y PRED= 7 790 PANCE= 0 100
.123 36	8 012	21 он 27 лн	12	H13A	STAT=Y PRED= 7 928 RANGE= 0 100
.123 37	0.645	52 .тн	12	H13B	STAT=Y PRED= 0.963 RANGE= 0.100
.124 36	-0 474	18. тн	15	H13A	STAT=Y PRED= -0 202 RANGE= 0 100
124 37	-0 120	10 он 16 лн	15	H13B	STAT=Y PRED= -0 500 RANGE= 0 100
127 28	0.120	31 .тн	6	HIA	STAT=Y PRED= $0.775$ RANGE= $0.100$
127 29	0 633	35 .ТН	6	HIB	STAT=Y PRED= 0 746 RANGE= 0 100
JZ7 31	0 008	30 лн	6	нза	STAT=Y PRED= 0 203 RANGE= 0 100
J27 32	0.067	73 ЈН	6	H3B	STAT=Y PRED= $0.088$ RANGE= $0.100$
J27 47	7.459	99 ЈН	6	H20	STAT=Y PRED= $7.464$ RANGE= $0.100$
J28 29	-18.561	L1 ЈН	1A	H1B	STAT=Y PRED= -18.721 RANGE= 0.100
J28 30	6.649	99 ЈН	1A	Н2	STAT=Y PRED= 6.495 RANGE= 0.100
J28 31	2.045	53 ЈН	1A	НЗА	STAT=Y PRED= 2.040 RANGE= 0.100
J28 32	2.047	72 ЈН	1A	НЗВ	STAT=Y PRED= 2.035 RANGE= 0.100
J29 30	8.276	59 ЈН	1B	Н2	STAT=Y PRED= 8.210 RANGE= 0.100
J29 <sup>31</sup>	2.136	58 JH	1B	НЗА	STAT=Y PRED= 2.072 RANGE= 1.000
J29 32	1.019	97 ЈН	1в	НЗВ	STAT=Y PRED= 1.095 RANGE= 0.100
J30_31	6.604	44 ЈН	2	H3A	STAT=Y PRED= 6.403 RANGE= 0.100
J30 <u>3</u> 2	8.407	73 ЈН	2	НЗВ	STAT=Y PRED= 8.400 RANGE= 0.100
J30_33	6.971	L6 JH	2	H19	STAT=Y PRED= 6.949 RANGE= 0.100
J31_32	-16.039	98 JH	3A	НЗВ	STAT=Y PRED= -15.903 RANGE= 0.100
J36_37	-18.164	40 ЈН	13A	H13B	STAT=Y PRED= -18.515 RANGE= 0.100
CONTROL S REF 0.00	PARAMETERS colvent = TERENCE = T 1.000 = 1000000 =	S: none (d IMS Concent Minimum	ef. 99% ration line-i	vol%, de	ed) lef=1.0%)
0.00	100000 =	Diagona	lizatio	on criter:	ium (not in use)
699.95	432200 =	FIELD(1	H,MHz),	used to	transform shifts to ppms
16.23	558651 =	Leit ir	equency	(ppm)	
-3.90	528483 =	Right f	requenc	y (ppm)	
	0.000 =	Acquisi	tion ti	me (s, fo	Or QMILS)
0 050	0.39/ =	Line-wi	ath (fo	or modes l	D, P & T, U=use defaults)
0.053	52602 = 52600	Data-po	Int res	olucion	(RZ)
	0 000 -	Diesers	ion act	-use dela tributi-	auto IIOM INF)
0 00	0.000 =	Decourd	ing fro		for DORES)
0.00		Decoupt	THÀ TLE	чиенсу (.	TOT DORED)
CONSTRAI	NTS (in eq	quations	X0 = 1	.0)use	e no empty lines

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### Figure S16. <sup>1</sup>H NMR HiFSA data for 6-epi-Riolozatrione (2) in CDCl<sub>3</sub> (PERCH .pms file format)

ACTIVE SE	PECIES:1H											
CHEMICAL	SHIFTS (PPM) :											
PROTON	2*SPIN= 1 S	PEC	IES=1	H POPU	ULATION	(Y)=	0.91072	2				
H7A / 1	4.21927	2	1*1*1	STAT=Y	PRED=	4.222	RANGE=	0.073	WIDTH(Y) =	3.500	RESP(Y) =	1.0000
H12A / 1	1.60787	7	1*1*1	STAT=Y	PRED=	1.607	RANGE=	0.073	WIDTH(Y) =	0.629	RESP(Y) =	1.0000
H15A / 1	1.86476	3	1*1*1	STAT=Y	PRED=	1.862	RANGE=	0.073	WIDTH(Y) =	0.946	RESP(Y) =	1.0000
H6A / 1	2.92129	3	1*1*1	STAT=Y	PRED=	2.920	RANGE=	0.073	WIDTH(Y) =	0.718	RESP(Y) =	1.0000
H1A / 1	2.17449	9	1*1*1	STAT=Y	PRED=	2.172	RANGE=	0.073	WIDTH(Y) =	3.107	RESP(Y) =	1.0000
H1B / 1	2,47941	8	1*1*1	STAT=Y	PRED=	2.481	RANGE=	0.073	WTDTH(Y) =	1.775	RESP(Y) =	1.0000
H2A / 1	2 75799	8	1*1*1	STAT=Y	PRED=	2 757	RANGE=	0 073	WTDTH(Y) =	0 554	BESP(Y) =	1 0000
u2x / 1	2 00212	6	1*1*1	CTAT-Y	DDED-	2 002	DANCE-	0 073		0.576	DECD(V) -	1 0000
HOA / 1	2.00212	2	1+1+1	SIAI-I	PRED-	2.002	DANCE-	0.073	WIDIH(I)-	0.570	RESP(I) =	1 0000
	. 2.54061	2	1 ~ 1 ~ 2	STAT-1	PRED-	1 150	RANGE-	0.073		0.605	RESP(1) =	1 0000
	2 10151	9 C	1 - 1 - 1	STAT-1	PRED-	2 102	RANGE-	0.073		1 400	RESP(1) =	1 0000
HIJA / I	. 3.12151	0	1.1.1.1	STAT=1	PRED=	3.123	RANGE=	0.073	WIDTH(Y) =	1.498	RESP(I) =	1.0000
HI3B / 1	2.93131	2	T×T×T	STAT=Y	PRED=	2.929	RANGE=	0.073	WIDTH(Y) =	1.868	RESP(Y) =	1.0000
H17B / 1	1.01960	3	1*1*3	STAT=Y	PRED=	1.019	RANGE=	0.073	WIDTH(Y) =	2.914	RESP(Y) =	1.0000
H16B / 1	0.80410	9	1*1*3	STAT=Y	PRED=	0.804	RANGE=	0.073	WIDTH(Y) =	2.037	RESP(Y) =	1.0000
H18B / 1	1.22654	2	1*1*3	STAT=Y	PRED=	1.225	RANGE=	0.073	WIDTH(Y) =	0.780	RESP(Y) =	1.0000
н20в / 1	1.07022	4	1*1*3	STAT=Y	PRED=	1.069	RANGE=	0.073	WIDTH(Y) =	0.619	RESP(Y) =	1.0000
COUPLING	CONSTANTS (HZ	):										
J10_27	6.3191	J	H7A	H6A	STAT=Y	PRED=	= 7.604	RANGE=	= 3.320			
J48	1.4989	J	H7A	H1A	STAT=Y	PRED=	= -0.500	) RANGE	E= 1.000			
J49	1.5066	J	H7A	H1B	STAT=Y	PRED=	= -0.500	) RANGE	E= 1.000			
J45	2.1803	J	H7A	нза	STAT=Y	PRED=	= 2.000	RANGE=	= 1.000			
J46	2.8238	J	H7A	нзв	STAT=Y	PRED=	= 2.000	RANGE=	= 1.000			
20	-0.3510	J	H7A	H18B	STAT=Y							
J23 24	7.8141	J	H12A	H15A	STAT=Y	PRED=	9.000	RANGE=	= 2.000			
J23 36	8.3722	J	H12A	H13A	STAT=Y	PRED=	= 8.821	RANGE:	= 5.000			
.123 37	0 6381	.т	H12A	H13B	STAT=Y	PRED=	= 0 280	BANGE:	= 5 000			
18	0 8346	.т	H63	H1 A	STAT-Y		- 0.280	DANCE-	= 5.000			
19	0 8728	.т	HEA	H1B	STAT-I STAT-V	DDED-	- 0.200	DANCE-	= 5.000			
107 47	7 7524	- -	UCN	H20B	CENTER STATES	DDED-	- 7 022	DANCE-	- 2 220			
720 20	-10 4677	-	110A 111 A	H20B	STAT-I	PRED-	-15032	CANGE-	- 2.230			
JZ6_Z9	-10.40//	J -	п1А 111 р	HID NO.	STAT-1	PRED-	15.94	DANCE-	JE 2.300			
J28_30	6.5099	5	HIA	HZA	STAT=1	PRED=	= 7.071	RANGE	= 4.500			
J28_31	2.13/4	J -	HIA	HJA	STAT=Y	PRED=	= 1./16	RANGE:	= 1.750			
J28_32	2.0492	J	HIA	нзв	STAT=Y	PRED=	= 2.0/2	RANGE=	= 1.000			
J29_30	8.2393	J	HIB	H2A	STAT=Y	PRED=	= 7.215	RANGE=	= 4.500			
J29_31	2.2020	J	HIB	H3A	STAT=Y	PRED=	= 2.069	RANGE=	= 1.000			
J29_32	1.1195	J	н1в	нзв	STAT=Y	PRED=	= 1.749	RANGE=	= 1.000			
J30_31	6.4856	J	H2A	НЗА	STAT=Y	PRED=	= 7.096	RANGE=	= 2.280			
J30_32	8.3419	J	H2A	нзв	STAT=Y	PRED=	= 10.031	L RANGE	E= 3.200			
J30_33	6.9456	J	H2A	н19в	STAT=Y	PRED=	= 7.056	RANGE=	= 1.000			
J31_32	-16.0054	J	нза	нзв	STAT=Y	PRED=	= -16.80	)2 RANG	GE= 3.250			
J36_37	-16.9517	J	H13A	H13B	STAT=Y	PRED=	= -15.50	)9 RANG	GE= 1.330			
_												
CONTROL E	ARAMETERS:											
Sc	lvent = non	e (	def.	99% enri	ched)							
REFE	RENCE = TMS											
	1.000 = Con	cen	tratio	on (vol%	, def=1	.0%)						
0.001	00000 = Min	imu	m line	e-intens:	itv							
0 001	00000 = Dia	aon	aliza	tion crit	terium	(not ir	1150)					
690 054	32200 = ETE	י ח.ד	1H MP	7) 11004	+0 + +	sform	shifta	to pro	ng			
16 224	96007 = 10f	+ +	reque	c,, useu			5111105	co ppi				
_3 005	$01106 - D^2 -$	ບ 1 h+	from	ncy (ppm)	, m)							
-3.905	0 000 - 3-3		itio-	time (ppi	"fan ^							
	0.000 = Acq	uis	TETON	LIME (S	, IOT QI	лтьз)						

0.000 - Addustrin time (s, for QMLS) 0.495 = Line-width (for modes D, P & T, 0=use defaults) 0.053778627 = Data-point resolution (Hz) 142.439 = GAUSSIAN (%, 0=use default from INF) 0.000 = Dispersion contribution (%, 0=use default from INF) 0.00000000 = Decoupling frequency (for DORES)

CONSTRAINTS (in equations X0 = 1.0)...use no empty lines

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**Figure S17**. Full <sup>1</sup>H NMR  $\delta$  and *J*-correlation maps, termed Quantum Interaction and Linkage Tables (QuILTs), of riolozatrione (1) and 6-*epi*-riolozatrione (2) were achieved by HiFSA processing of the 700 MHz spectra in CDCl<sub>3</sub>. Multiplicities within parentheses are due to couplings of  $\leq 1$  Hz. Couplings with absolute value of  $\leq 0.10$  Hz are given as " $\phi$ ".

. 1	м	ы	1h(B)	$1_2(\alpha)$	2	2h/B)	$2\alpha(\alpha)$	6	7	11	12	$12_{2}(\alpha)$	12h/B)	16	17	10	10	20
δ⊥	101	п	ID(b)	14(u)	2	SD(b)	Sa(u)	-	/		12	15a(u)	13D(b)	10	1/	10	19	20
2.3103	dddd(d	) 1b(β)	d	2	3	4	4	5	4	/	8	/	/	9	9	6	4	6
2.4853	ddddd(d	) 1a(α)	-18.56	d	3	4	4	5	4	7	8	7	7	9	9	6	4	6
2.7534	ddqdd	2	6.65	8.28	d	3	3	6	5	8	9	8	8	10	10	7	3	7
1.9619	ddddd	3b(β)	2.05	2.14	6.60	d	2	5	5	8	9	8	8	10	10	7	4	6
2.4949	ddddd	3a(α)	2.05	1.02	8.41	-16.04	d	5	5	8	9	8	8	10	10	7	4	6
2.4564	qd(dd)	6	0.76	0.63	Ø	Ø	Ø	d	3	6	7	6	6	8	8	5	7	3
3.1265	ddddd	7	1.38	1.59	Ø	2.29	2.85	2.01	d	5	6	5	5	7	7	4	6	4
1.8832	d(dd)	11	Ø	Ø	Ø	Ø	Ø	Ø	Ø	d	3	4	4	4	4	5	9	7
1.6141	dd(d)	12	Ø	Ø	ø	Ø	ø	ø	Ø	7.68	d	3	3	4	4	6	10	8
2.8822	dd(d)	13a(α)	Ø	Ø	Ø	ø	ø	Ø	Ø	-0.47	8.01	d	2	5	5	5	9	7
2.8848	d(dd)	13b(β)	Ø	ø	ø	ø	ø	Ø	Ø	-0.12	0.65	-18.16	d	5	5	5	9	7
1.2359	s	16	Ø	ø	ø	ø	ø	ø	ø	ø	ø	ø	ø	d	4	7	11	9
0.8529	s	17	Ø	ø	Ø	ø	Ø	ø	Ø	Ø	Ø	Ø	Ø	Ø	d	7	11	9
1.0881	s	18	Ø	ø	ø	ø	Ø	Ø	Ø	Ø	Ø	Ø	Ø	Ø	Ø	d	8	6
1.1535	d	19	ø	ø	6.972	ø	ø	ø	Ø	ø	ø	ø	ø	Ø	ø	Ø	d	8
1.1571	d	20	Ø	ø	ø	ø	ø	7.46	ø	ø	ø	Ø	Ø	Ø	ø	ø	Ø	d
													-					1
2 م	М	н	1h(B)	10/01	h	21- (0)	2-1-1	~	_			12 1	1 1 21 10	10				
0 -			10(p)	1a(α)	Z	3D(B)	3a(α)	6	/	11	12	13a(α	) 13b(B	10	17	18	19	20
2.1745	dddd(d)	1b(β)	d	1a(α) 2	3	3D(β) 4	3a(α) 4	5	4	11 7	12 8	13a(α 7	7 13b	9	17 9	18 6	19 4	20 6
2.1745 2.4794	ddddd(d) ddddd(d)	1b(β) 1a(α)	d -18.47	2 d	3	3D(B) 4 4	3a(α) 4 4	5 5	7 4 4	11 7 7	12 8 8	13a(α 7 7	) 13b(β 7 7	9	17 9 9	18 6 6	19 4 4	20 6 6
2.1745 2.4794 2.7580	ddddd(d) ddddd(d) ddqdd	1b(β) 1a(α) 2	d -18.47 6.51	1a(α) 2 d 8.24	2 3 3 d	3D(B) 4 4 3	3a(α) 4 4 3	5 5 6	7 4 4 5	11 7 7 8	12 8 8 9	13a(α 7 7 8	) 130(B) 7 7 8	9 9 9 10	17 9 9 10	18 6 6 7	19 4 4 3	20 6 6 7
2.1745 2.4794 2.7580 2.0021	ddddd(d) ddddd(d) ddqdd ddqdd	1b(β) 1a(α) 2 3b(β)	d -18.47 6.51 2.14	1a(α) 2 d 8.24 2.20	2 3 3 d 6.486	3D(β) 4 4 3 d	3a(α) 4 4 3 2	5 5 6 5	7 4 4 5 5	11 7 7 8 8	12 8 8 9 9	13a(α 7 7 8 8	) 13D(B) 7 7 8 8	9 9 10 10	17 9 9 10 10	18 6 7 7	19 4 4 3 4	20 6 6 7 6
2.1745 2.4794 2.7580 2.0021 2.5406	ddddd(d) ddddd(d) ddqdd ddqdd ddddd	1b(β) 1a(α) 2 3b(β) 3a(α)	d -18.47 6.51 2.14 2.05	12(0) 2 d 8.24 2.20 1.12	2 3 3 d 6.486 8.34	30(β) 4 4 3 d -16.01	3a(α) 4 4 3 2 d	6 5 6 5 5 5	7 4 4 5 5 5 5	11 7 7 8 8 8 8	12 8 9 9 9	13a(α 7 7 8 8 8 8	) 130(B) 7 7 8 8 8 8	9 9 10 10 10	17 9 9 10 10 10	18 6 7 7 7 7	19 4 4 3 4 4	20 6 7 6 6
2.1745 2.4794 2.7580 2.0021 2.5406 2.9213	(d) (d) (d) (d) (d) (d) (d) (d) (d) (d)	1b(β) 1a(α) 2 3b(β) 3a(α) 6	c           -18.47           6.51           2.14           2.05           0.83	Ia(Q) 2 d 8.24 2.20 1.12 0.87	2 3 3 6.486 8.34 Ø	3D(β) 4 4 3 3 d -16.01 Ø	33((2) 4 4 3 2 d Ø	6 5 6 5 5 5 d	7 4 5 5 5 5 3	11 7 7 8 8 8 8 8	12 8 9 9 9 9 7	1.3a(α 7 7 8 8 8 8 8 6	13b(b)           7           7           8           8           8           8           6	16           9           10           10           10           8	17 9 9 10 10 10 8	18 6 7 7 7 7 5	19 4 3 3 4 4 4 7	20 6 7 6 6 6 3
2.1745 2.4794 2.7580 2.0021 2.5406 2.9213 4.2193	ddddddd (d) (dddddd) (dddddd (ddddd (ddddd) (ddddd) (ddddd(d)	1b(β) 1a(α) 2 3b(β) 3a(α) 6 7	13(p) d -18.47 6.51 2.14 2.05 0.83 1.50	13(Q) 2 d 8.24 2.20 1.12 0.87 1.51	2 3 3 6.486 8.34 Ø Ø	3D(β) 4 4 3 3 d -16,01 Ø 2.18	33(Q) 4 4 3 3 2 d 2.82	6 5 5 6 5 5 5 3 4	/ 4 5 5 5 5 3 d	11 7 8 8 8 8 8 6 5	12 8 9 9 9 9 7 6	13a(α 7 7 8 8 8 8 8 8 8 6 5	) 130(β) 7 7 8 8 8 8 8 8 8 6 5	9 9 10 10 10 8 7	17 9 9 10 10 10 8 7	18       6       7       7       7       5       4	<ul> <li>19</li> <li>4</li> <li>4</li> <li>3</li> <li>4</li> <li>4</li> <li>4</li> <li>7</li> <li>6</li> </ul>	20 6 7 6 6 6 3 3
2.1745 2.4794 2.7580 2.0021 2.5406 2.9213 4.2193 1.8648	() () () () () () () () () () () () () (	1b(β) 1a(α) 2 3b(β) 3a(α) 6 7 11	<ul> <li>IS(p)</li> <li>d</li> <li>-18.47</li> <li>6.51</li> <li>2.14</li> <li>2.05</li> <li>0.83</li> <li>1.50</li> <li>Ø</li> </ul>	13(Q) 2 d 8.24 2.20 1.12 0.87 1.51 Ø	2 3 3 6.486 8.34 Ø Ø	3D(β) 4 4 3 3 d -16.01 Ø 2.18 Ø	3a(a) 4 4 3 2 d 2.82 Ø	6 5 6 5 5 5 6 2 5 6 3 2 6.32	/ 4 5 5 5 3 d Ø	11 7 8 8 8 8 8 6 5 4	12 8 9 9 9 9 9 7 6 3	1.3a(α 7 7 8 8 8 8 8 8 6 5 5 4	13b(β)       7       7       8       8       8       8       6       5       4	16       9       9       10       10       10       10       7       4	17 9 9 10 10 10 8 7 4	18 6 7 7 7 5 4 5 4 5	19 4 3 3 4 4 4 7 7 6 9	20 6 7 6 6 6 3 3 4 7
2.1745 2.4794 2.7580 2.0021 2.5406 2.9213 4.2193 1.8648 1.6079	() () () () () () () () () () () () () (	1b(β) 1a(α) 2 3b(β) 3a(α) 6 7 11 12	d -18.47 6.51 2.14 2.05 0.83 1.50 Ø Ø	13(Q) 2 d 8.24 2.20 1.12 0.87 1.51 Ø Ø	2 3 3 6.486 8.34 Ø Ø Ø	3D(j5) 4 4 3 d 3 d -16.01 Ø 2.18 Ø Ø	3a(a) 4 4 3 2 d Ø 2.82 Ø Ø	6 5 5 6 5 5 4 6.32 Ø	<ul> <li>7</li> <li>4</li> <li>5</li> <li>5</li> <li>5</li> <li>3</li> <li>d</li> <li>Ø</li> <li>Ø</li> </ul>	11 7 8 8 8 8 8 6 5 5 d	12 8 9 9 9 9 9 7 6 3 3	13a(α 7 7 8 8 8 8 8 8 8 8 8 8 8 8 6 5 4 3	13b(β)       13b(β)       7       7       8       8       8       8       6       5       4       3	16       9       9       10       10       10       10       10       4	17 9 9 10 10 10 8 7 4 4	18       6       7       7       7       4       5       6	<ul> <li>19</li> <li>4</li> <li>4</li> <li>3</li> <li>4</li> <li>4</li> <li>4</li> <li>7</li> <li>6</li> <li>9</li> <li>10</li> </ul>	20 6 7 6 6 3 3 4 7 8
2.1745 2.4794 2.7580 2.0021 2.5406 2.9213 4.2193 1.8648 1.6079 3.1215	(b)bbbb (b)bbbb (b)bbbb (b)bbb (b)bbbb (b)bbbb (b)bbbb (b)bbbb (b)bbbb (b)bbbbb (b)bbbbb (b)bbbbb (b)bbbbb (b)bbbbb)	<ol> <li>1b(β)</li> <li>1a(α)</li> <li>2</li> <li>3b(β)</li> <li>3a(α)</li> <li>6</li> <li>7</li> <li>11</li> <li>12</li> <li>13a(α)</li> </ol>	d -18.47 6.51 2.14 2.05 0.83 1.50 Ø Ø	13(Q) 2 d 8.24 2.20 1.12 0.87 1.51 Ø Ø Ø	2 3 3 6 4 8 3 4 8 3 4 Ø 0 0 0 0 0 0 0 0 0	3D(j3) 4 4 3 3 4 3 4 3 4 3 4 3 4 2 16,01 0 2.18 Ø 0 Ø Ø Ø Ø Ø Ø Ø Ø Ø Ø Ø Ø Ø	3a(α) 4 4 3 3 2 2 8 2 8 2 8 2 8 2 8 2 8 2 8 2 8 2	6 5 5 6 6 5 5 6 3 6 3 0 0 0 0 0 0	<ul> <li>4</li> <li>4</li> <li>5</li> <li>5</li> <li>5</li> <li>3</li> <li>d</li> <li>Ø</li> <li>Ø</li> <li>Ø</li> <li>Ø</li> <li>Ø</li> <li>Ø</li> </ul>	11 7 7 8 8 8 8 8 8 8 8 8 8 6 5 5 d 7.81 Ø	12 8 8 9 9 9 9 9 9 7 6 3 3 4 8.37	13a(α 7 7 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8	<ul> <li>13b(β)</li> <li>13b(β)</li> <li>7</li> <li>7</li> <li>8</li> <li>8</li> <li>8</li> <li>8</li> <li>6</li> <li>5</li> <li>4</li> <li>3</li> <li>2</li> </ul>	16       9       9       10	17 9 9 10 10 10 10 8 7 4 4 4 5	18       6       7       7       7       5       4       5       6       5       6       5	19 4 3 3 4 4 7 7 6 9 9 10 9	20 6 7 6 6 3 3 4 7 8 7
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