Radical Deoxygenation of Hydroxyl Groups via Phosphites

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Supporting Information

¹H, ¹³C and ³¹P NMR Spectra

STANDARD 1H OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

File: phosphite-steroid

INOVA-500 "N1"

PULSE SEQUENCE

Pulse 41.4 degrees

Acq. time 2.666 sec

Width 3759.8 Hz

32 repetitions

OBSERVE H1, 399.9649457 MHz

DATA PROCESSING

FT size 32768

Total time 1 min, 25 sec

13C OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

File: phosphite-steroid-cl3

INOVA-500 "N1"

PULSE SEQUENCE

Relax. delay 0.900 sec

Pulse 45.0 degrees

Acq. time 1.199 sec

Width 25000.0 Hz

368 repetitions

OBSERVE C13, 100.5712699 MHz

DECOUPLE H1, 399.9669644 MHz

Power 44 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 70 hr, 16 min, 25 sec

PHOSPHORUS OBSERVE
STANDARD PARAMETERS
PHOSPHATE REGION

Pulse Sequence: s2pul

Solvent: CDCl₃
Ambient temperature
File: phosphite-steroid-p31
INOVA-500 "Ni"

PULSE SEQUENCE

Pulse 45.0 degrees
Acq. time 0.800 sec
Width 60015.0 Hz
128 repetitions
OBSERVE P31, 161.9086763 MHz
DECOUPLE H1, 399.9669644 MHz
Power 44 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz
FT size 131072
Total time 1 min, 43 sec

STANDARD 1H OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl₃

Temp. 25.0 C / 298.1 K

File: phosphite-secondary-steroid-side-phosphonate
INOVA-500 "Ni"

PULSE SEQUENCE

Pulse 41.4 degrees

Acq. time 2.666 sec

Width 6000.2 Hz

60 repetitions

OBSERVE H1, 399.9649469 MHz

DATA PROCESSING

FT size 32768

Total time 5 min, 42 sec

PHOSPHORUS OBSERVE
STANDARD PARAMETERS
PHOSPHATE REGION

Pulse Sequence: s2pul

Solvent: CDCl₃

Temp. 25.0 C / 298.1 K

File: phosphite-phosphonate-side-product-p31

INOVA-500 "N1"

PULSE SEQUENCE

Pulse 44.2 degrees

Acq. time 0.800 sec

Width 50000.0 Hz

232 repetitions

OBSERVE P31, 161.9086781 MHz

DECOUPLE H1, 399.9669644 MHz

Power 44 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 2 hr, 52 min, 33 sec

STANDARD 1H OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Temp. 25.0 C / 298.1 K

File: phosphite-phosphonate

INOVA-500 "Ni"

PULSE SEQUENCE

Pulse 41.4 degrees

Acq. time 2.666 sec

Width 6000.2 Hz

32 repetitions

OBSERVE H1, 399.9649454 MHz

DATA PROCESSING

FT size 32768

Total time 5 min, 42 sec

PHOSPHORUS OBSERVE
STANDARD PARAMETERS
PHOSPHATE REGION

Pulse Sequence: s2pul

Solvent: CDCl₃
Temp. 25.0 C / 298.1 K
File: phosphite-phosphonate-p31
INOVA-500 "N1"

PULSE SEQUENCE

Pulse 44.2 degrees
Acq. time 0.800 sec
Width 50000.0 Hz
168 repetitions
OBSERVE P31, 161.9086751 MHz
DECOUPLE H1, 399.9669644 MHz
Power 44 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz
FT size 131072
Total time 2 hr, 52 min, 33 sec

STANDARD 1H OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

File: p-steroid-h

INOVA-500 "Ni"

PULSE SEQUENCE

Pulse 41.4 degrees

Acq. time 2.666 sec

Width 3759.8 Hz

32 repetitions

OBSERVE H1, 399.9649459 MHz

DATA PROCESSING

FT size 32768

Total time 1 min, 25 sec

STANDARD CARBON PARAMETERS

	INDEX	FREQUENCY	PPM	HEIGHT
Pulse Sequence: s2pul	1	24755.432	196.939	15.7
Solvent: CDC13	2	19559.926	155.607	24.6
Ambient temperature	3	18174.865	144.588	38.1
User: 1-14-87	4	9714.510	77.283	88.3
INOVA-500 "Sn"	5	9682.470	77.028	90.5
PULSE SEQUENCE	6	9650.888	76.777	90.0
Relax. delay 0.700 sec	7	7103.255	56.509	84.3
Pulse 45.3 degrees	8	6940.767	55.216	63.2
Acq. time 1.380 sec	9	5942.494	47.275	66.3
Width 29596.3 Hz	10	5823.030	46.324	40.3
92 repetitions	11	4833.912	38.456	65.6
OBSERVE C13, 125.7610235 MHz	12	4585.373	36.478	33.6
DECOUPLE H1, 499.9067532 MHz	13	4378.029	34.829	68.1
Power 38 dB	14	4247.123	33.787	63.5
continuously on	15	4046.645	32.193	73.1
WALTZ-16 modulated	16	4027.421	32.040	67.5
DATA PROCESSING	17	3652.553	29.057	60.1
Line broadening 0.5 Hz	18	3636.991	28.934	67.3
FT size 131072	19	3413.169	27.153	55.1
Total time 6 hr, 41 min, 52 sec	20	3370.692	26.814	56.4
	21	2763.355	22.143	65.3
	22	2590.658	20.610	69.3
	23	2801.581	15.923	69.2
	24	1530.594	12.176	51.4
	25	-0.000	-0.000	62.7

STANDARD 1H OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Temp. 25.0 C / 298.1 K

File: phosphite-primary-steroid
INOVA-500 "Ni"

PULSE SEQUENCE

Pulse 41.4 degrees

Acq. time 2.666 sec

Width 3630.1 Hz

16 repetitions

OBSERVE H1, 399.9649437 MHz

DATA PROCESSING

FT size 32768

Total time 0 min, 42 sec

¹³C OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: phosphite-primary-steroid-cl3

INNOVA-500 "Ni"

PULSE SEQUENCE

Relax. delay 0.900 sec

Pulse 45.0 degrees

Acq. time 1.199 sec

Width 25000.0 Hz

608 repetitions

OBSERVE C13, 100.5712646 MHz

DECOUPLE H1, 399.9669644 MHz

Power 44 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0

FT size 65536

Total time 70 hr, 16 min, 25 sec

PHOSPHORUS OBSERVE
STANDARD PARAMETERS
PHOSPHATE REGION

Pulse Sequence: s2pul

Solvent: CDCl₃
Temp. 25.0 C / 298.1 K
File: phosphite-primary-steroid-p31
INOVA-500 "N1"

PULSE SEQUENCE

Pulse 44.2 degrees
Acq. time 0.800 sec
Width 50000.0 Hz
128 repetitions
OBSERVE P31, 161.9086781 MHz
DECOUPLE H1, 399.9669644 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 min, 43 sec

STANDARD 1H OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Temp. 25.0 C / 298.1 K

File: phosphite-primary-steroid-reduction-pure
INOVA-500 "Ni"

PULSE SEQUENCE

Pulse 41.4 degrees

Acq. time 2.666 sec

Width 3630.1 Hz

32 repetitions

OBSERVE H1, 399.9649468 MHz

DATA PROCESSING

FT size 32768

Total time 1 min, 25 sec

¹³C OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: phosphite-primary-steroid-reduction-pure-c13
INOVA-500 "N1"

PULSE SEQUENCE

Relax. delay 0.900 sec

Pulse 45.0 degrees

Acq. time 1.199 sec

Width 25000.0 Hz

544 repetitions

OBSERVE C13, 100.5712631 MHz

DECOUPLE H1, 399.9669644 MHz

Power 44 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 70 hr, 16 min, 25 sec

STANDARD 1H OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

File: phosphite-cis-tbutylcyclohexanol

INOVA-500 "Ni"

PULSE SEQUENCE

Pulse 42.1 degrees

Acq. time 3.333 sec

Width 4500.5 Hz

32 repetitions

OBSERVE H1, 300.0732307 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 32768

Total time 1 min, 46 sec

STANDARD CARBON PARAMETERS

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

User: 1-14-87

File: phosphite-cis-tbucyclohexanol-cl3

INOVA-500 "N1"

PULSE SEQUENCE

Relax. delay 0.700 sec

Pulse 27.2 degrees

Acq. time 1.300 sec

Width 29996.3 Hz

256 repetitions

OBSERVE C13, 125.7010245 MHz

DECOUPLE H1, 499.9067532 MHz

Power 32 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz

FT size 131072

Total time 6 hr, 41 min, 52 sec

PHOSPHORUS OBSERVE
STANDARD PARAMETERS
PHOSPHATE REGION

Pulse Sequence: s2pul

Solvent: CDCl₃

Temp. 25.0 C / 298.1 K

File: phosphite-cis-tbutylcyclohexanol-p31

INOVA-500 "N1"

PULSE SEQUENCE

Pulse 44.2 degrees

Acq. time 0.800 sec

Width 50000.0 Hz

84 repetitions

OBSERVE P31, 161.9086874 MHz

DECOUPLE H1, 399.9669644 MHz

Power 44 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 1 min, 43 sec

-0.114

STANDARD 1H OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Temp. 25.0 C / 298.1 K

File: phosphite-3-octanol

INOVA-500 "Ni"

PULSE SEQUENCE

Pulse 41.4 degrees

Acq. time 2.666 sec

Width 3630.1 Hz

16 repetitions

OBSERVE H1, 399.9649508 MHz

DATA PROCESSING

FT size 32768

Total time 0 min, 42 sec

13C OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: phosphite-3-octanol-p31
INOVA-500 "Ni"

PULSE SEQUENCE

Relax. delay 0.900 sec
Pulse 45.0 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
288 repetitions

OBSERVE C13, 100.5712654 MHz

DECOUPLE H1, 399.9669644 MHz

Power 44 dB

continuously on
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz
FT size 65536
Total time 70 hr, 16 min, 25 sec

PHOSPHORUS OBSERVE
STANDARD PARAMETERS
PHOSPHATE REGION

INDEX	FREQUENCY	PPM	HEIGHT
1	22785.534	140.731	94.3
2	22780.194	140.698	83.9

Pulse Sequence: s2pul

Solvent: CDCl₃
Temp. 25.0 C / 298.1 K
File: phosphite-3-octanol-cl3
INOVA-500 "Ni"

PULSE SEQUENCE

Pulse 44.2 degrees
Acq. time 0.800 sec
Width 50000.0 Hz
128 repetitions
OBSERVE P31, 161.9086766 MHz
DECOUPLE H1, 399.9669644 MHz
Power 44 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz
FT size 131072
Total time 1 min, 43 sec

STANDARD 1H OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Temp. 25.0 C / 298.1 K

File: phosphite-secondary-chain-alcohol-alpha-methyl
INOVA-500 "Ni"

PULSE SEQUENCE

Pulse 41.4 degrees

Acq. time 2.666 sec

Width 3630.1 Hz

32 repetitions

OBSERVE H1, 399.9649561 MHz

DATA PROCESSING

FT size 32768

Total time 1 min, 25 sec

PHOSPHORUS OBSERVE
STANDARD PARAMETERS
PHOSPHATE REGION

Pulse Sequence: s2pul

Solvent: CDCl3

Temp. 25.0 C / 298.1 K

File: phosphite-secondary-alcohol-chain-alpha-methyl-p31

INOVA-500 "Ni"

PULSE SEQUENCE

Pulse 44.2 degrees

Acq. time 0.800 sec

Width 60015.0 Hz

104 repetitions

OBSERVE P31, 161.9086854 MHz

DECOUPLE H1, 399.9669644 MHz

Power 44 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 2 hr, 52 min, 33 sec

STANDARD CARBON PARAMETERS

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: phosphite-sugar

INOVA-500 "Ni"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 44.2 degrees

Acq. time 2.500 sec

Width 4671.8 Hz

8 repetitions

OBSERVE H1, 499.9042537 MHz

DATA PROCESSING

FT size 32768

Total time 18 min, 42 sec

PHOSPHORUS OBSERVE
STANDARD PARAMETERS
PHOSPHATE REGION

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: phosphite-sugar-p31

INOVA-500 "Ni"

PULSE SEQUENCE

Pulse 45.0 degrees

Acq. time 0.800 sec

Width 60015.0 Hz

128 repetitions

OBSERVE P31, 161.9086047 MHz

DECOUPLE H1, 399.9669644 MHz

Power 44 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 1 min, 43 sec

STANDARD 1H OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

File: p-sugar-h

INOVA-500 "N1"

PULSE SEQUENCE

Pulse 41.4 degrees

Acq. time 2.666 sec

Width 3759.8 Hz

16 repetitions

OBSERVE H1, 399.9649473 MHz

DATA PROCESSING

FT size 32768

Total time 1 min, 25 sec

13C OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

INOVA-400 "Zr"

PULSE SEQUENCE

Relax. delay 0.900 sec

Pulse 45.0 degrees

Acq. time 1.199 sec

Width 25000.0 Hz

912 repetitions

OBSERVE C13, 100.5712646 MHz

DECOUPLE H1, 399.9669644 MHz

Power 44 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 70 hr, 16 min, 25 sec

INDEX	FREQUENCY	PPM	HEIGHT
1	16732.536	166.375	11.2
2	13389.996	133.139	59.1
3	13051.241	129.771	100.6
4	12910.092	128.368	94.2
5	11200.293	111.367	25.1
6	10635.701	105.753	29.2
7	8074.435	80.286	26.8
8	7778.405	77.342	146.5
9	7746.361	77.024	151.2
10	7714.316	76.705	144.3
11	7625.050	75.817	39.6
12	6565.294	65.280	35.0
13	3563.036	35.428	48.5
14	2690.970	26.757	42.4
15	2629.933	26.150	37.5
16	0.000	0.000	23.2

STANDARD 1H OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

File: phosphite-admantanol

INOVA-500 "Ni"

PULSE SEQUENCE

Pulse 42.1 degrees

Acq. time 3.333 sec

Width 4500.5 Hz

32 repetitions

OBSERVE H1, 300.0732296 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 32768

Total time 1 min, 46 sec

¹³C OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl₃
Ambient temperature
File: phosphite-admantanol-cl3
INOVA-500 "Ni"

PULSE SEQUENCE

Relax. delay 0.900 sec

Pulse 45.0 degrees

Acq. time 1.199 sec

Width 25000.0 Hz

336 repetitions

OBSERVE C13, 100.5712676 MHz

DECOUPLE H1, 399.9669644 MHz

Power 44 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 70 hr, 16 min, 25 sec

STANDARD 1H OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

File: phosphite-admantanol-p31

INOVA-500 "Ni"

PULSE SEQUENCE

Pulse 45.0 degrees

Acq. time 1.069 sec

Width 59880.2 Hz

64 repetitions

OBSERVE P31, 121.4717937 MHz

DECOUPLE H1, 300.0746975 MHz

Power 45 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz

FT size 65536

Total time 3 hr, 35 min, 31 sec

STANDARD 1H OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

File: phosphite-tertiary-steroid

INOVA-500 "Ni"

PULSE SEQUENCE

Pulse 41.4 degrees

Acq. time 2.666 sec

Width 3630.1 Hz

16 repetitions

OBSERVE H1, 399.9649444 MHz

DATA PROCESSING

FT size 32768

Total time 0 min, 42 sec

PHOSPHORUS OBSERVE
STANDARD PARAMETERS
PHOSPHATE REGION
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: phosphite-tertiary-steroid-p31
INOVA-500 "N1"

PULSE SEQUENCE
Pulse 44.2 degrees
Acq. time 0.800 sec
Width 50000.0 Hz
128 repetitions
OBSERVE P31, 161.9086874 MHz
DECOUPLE H1, 399.9669644 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 min, 43 sec

STANDARD 1H OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

File: phosphite-tertiary-steroid-reduction
INOVA-500 "Ni"

PULSE SEQUENCE

Pulse 41.4 degrees

Acq. time 2.666 sec

Width 3630.1 Hz

16 repetitions

OBSERVE H1, 399.9649413 MHz

DATA PROCESSING

FT size 32768

Total time 0 min, 42 sec

13C OBSERVE
 99.59
 Pulse Sequence: s2pul
 Solvent: CDCl3
 Temp. 25.0 C / 298.1 K
 File: phosphite-steroid-tertiary-reduced-c13
 INOVA-500 "Ni"
 PULSE SEQUENCE
 Relax. delay 0.900 sec
 Pulse 45.0 degrees
 Acq. time 1.199 sec
 Width 25000.0 Hz
 192 repetitions
 OBSERVE C13, 100.5712623 MHz
 DECOUPLE H1, 399.9669644 MHz
 Power 44 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 65536
 Total time 70 hr, 16 min, 25 sec