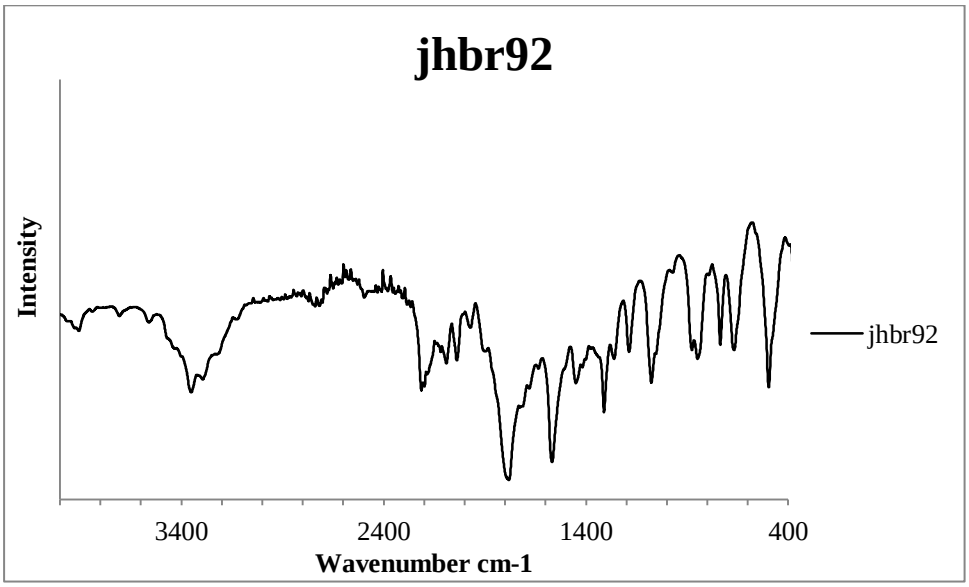
Supporting information for the thesis of Jan Hendrik Blank, University of St Andrews, 2012

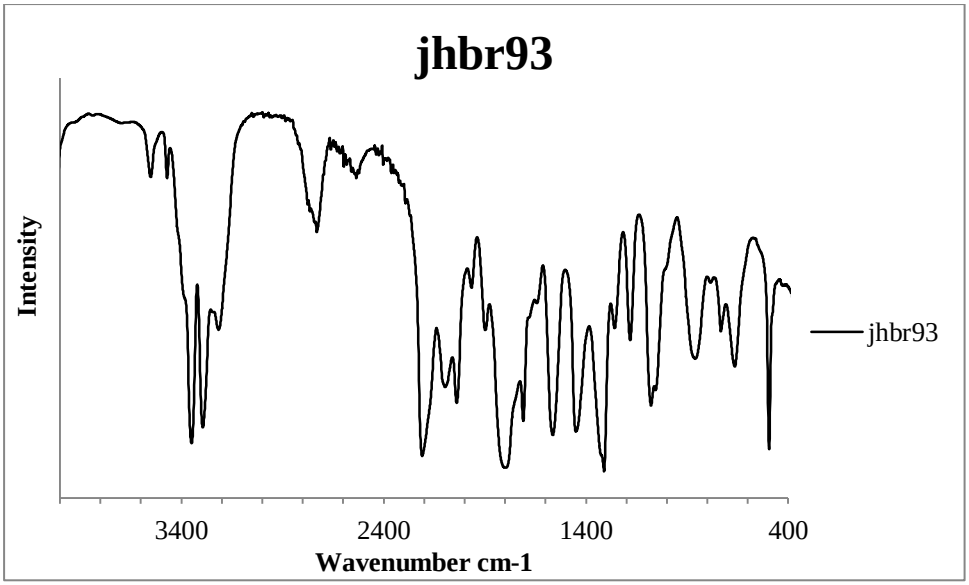
For most of the reactions, IR spectra and NMR spectra were taken of the product liquids:
The assignment of most of the peaks can be done by cross referencing the expected location from the Table of common NMR solvents.[1] The $[PBu_4]X$ shows peaks in the ^1H NMR at δ (ppm): 0.91 (t, P-(C_3H_6-C**H**$_3$)$_3$, 1.46 overlapping signal of P-(CH_2-C**H**$_2$-C_2H_5)$_3$ and P-(C_2H_4-C**H**$_2$-CH_3)$_3$ and 3.36 P-(C**H**$_2$-C_3H_7)$_3$. As discussed above. The compounds with the highest intensities are methanol, ethanol, ethylene glycol, however some signals from these compounds can shift considerably. When the spectra are expanded other peaks become apparent, for instance ethers, however, many overlap making assignment and especially quantification impossible. The hydride regions show multiple signals at times. Their assignment has been discussed in the text. ^{31}P NMR δ (ppm) 34.6.

Jhbr92



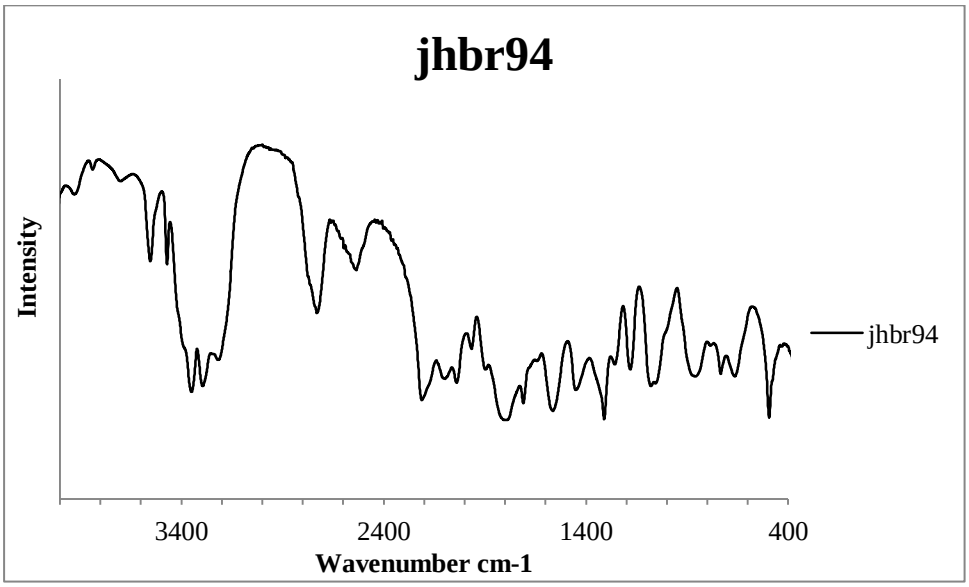
The complete IR spectrum of the liquid product after reaction of reaction jhbr92

Jhbr93



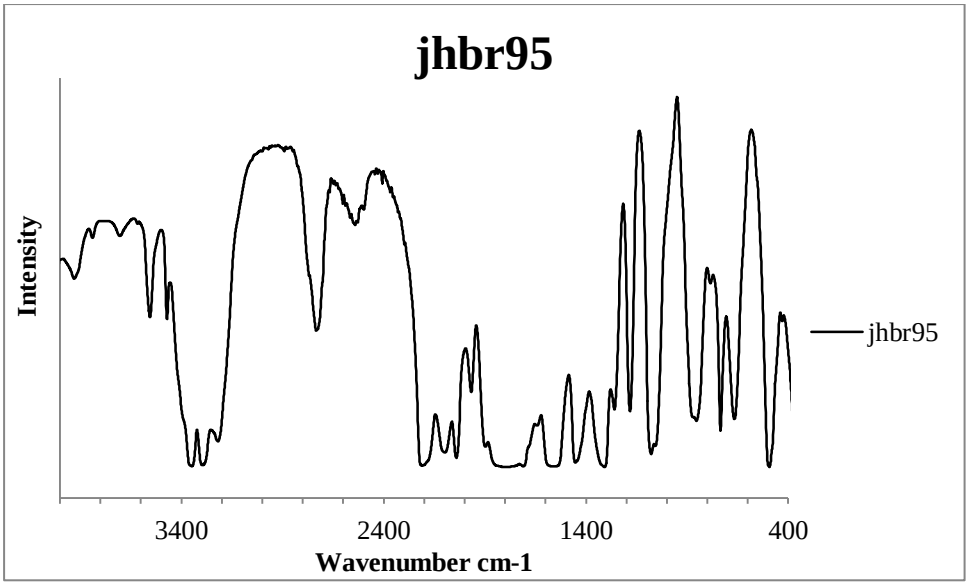
The complete IR spectrum of the liquid product after reaction of reaction jhbr93

Jhbr94



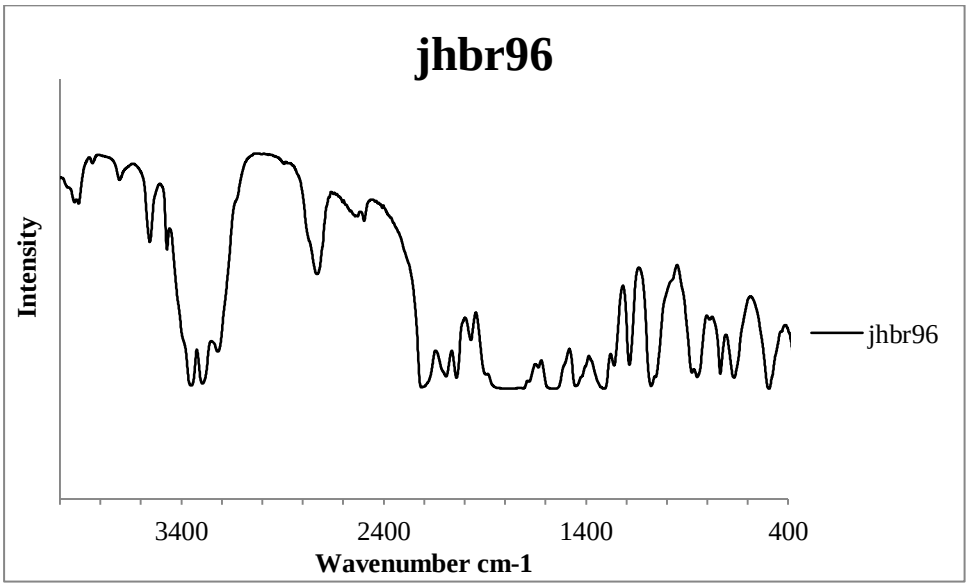
The complete IR spectrum of the liquid product after reaction of reaction jhbr94

Jhbr95



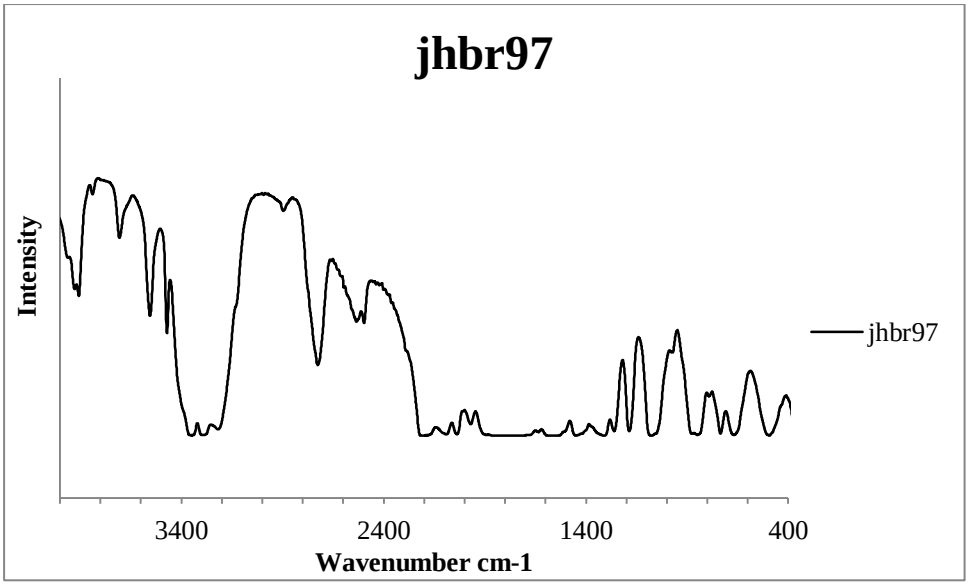
The complete IR spectrum of the liquid product after reaction of reaction jhbr95

Jhbr96



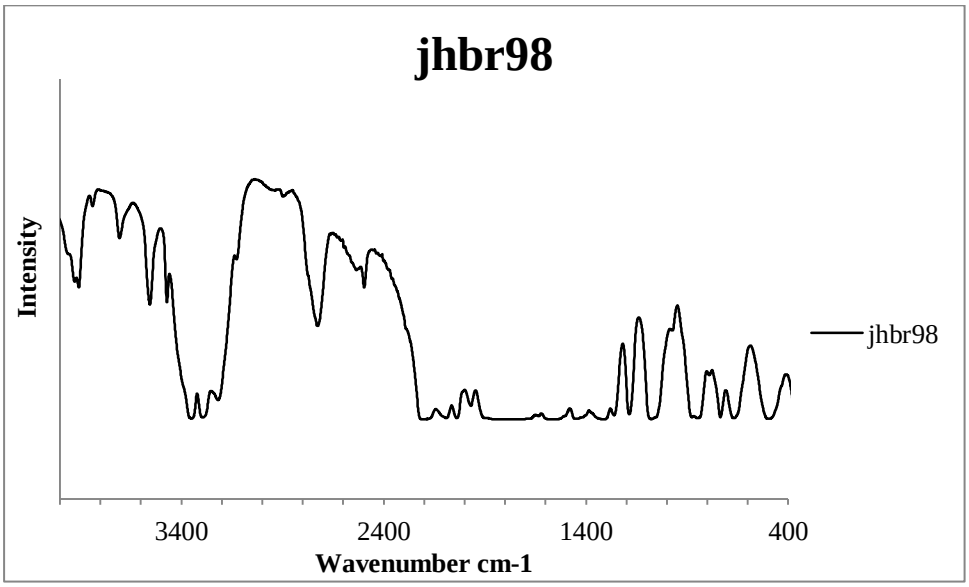
The complete IR spectrum of the liquid product after reaction of reaction jhbr96

Jhbr97



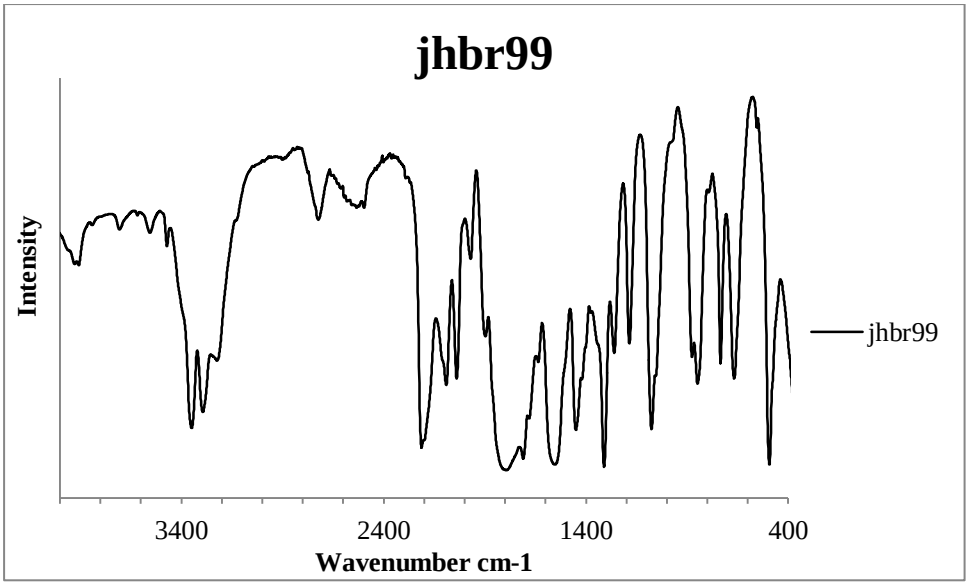
The complete IR spectrum of the liquid product after reaction of reaction jhbr97

Jhbr98



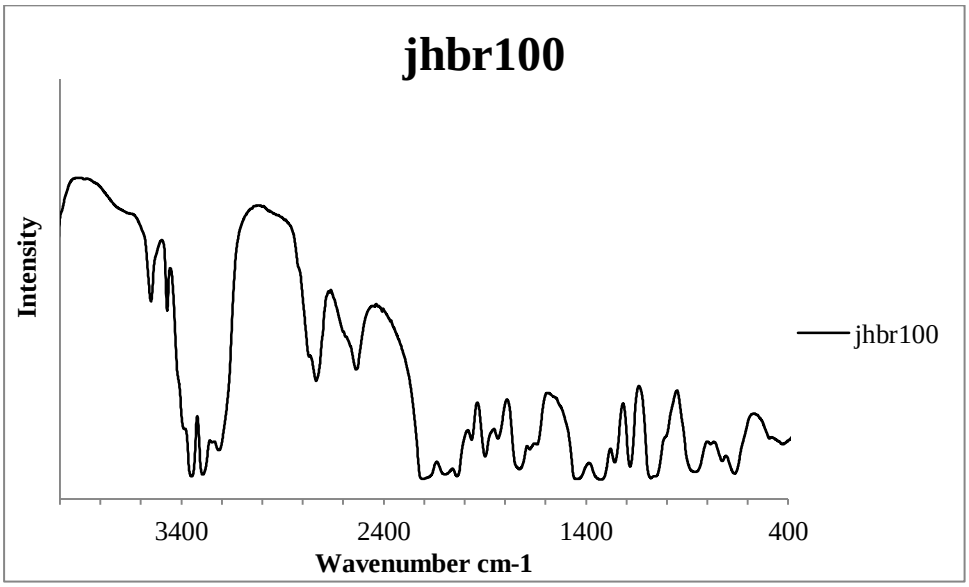
The complete IR spectrum of the liquid product after reaction of reaction jhbr98

Jhbr99



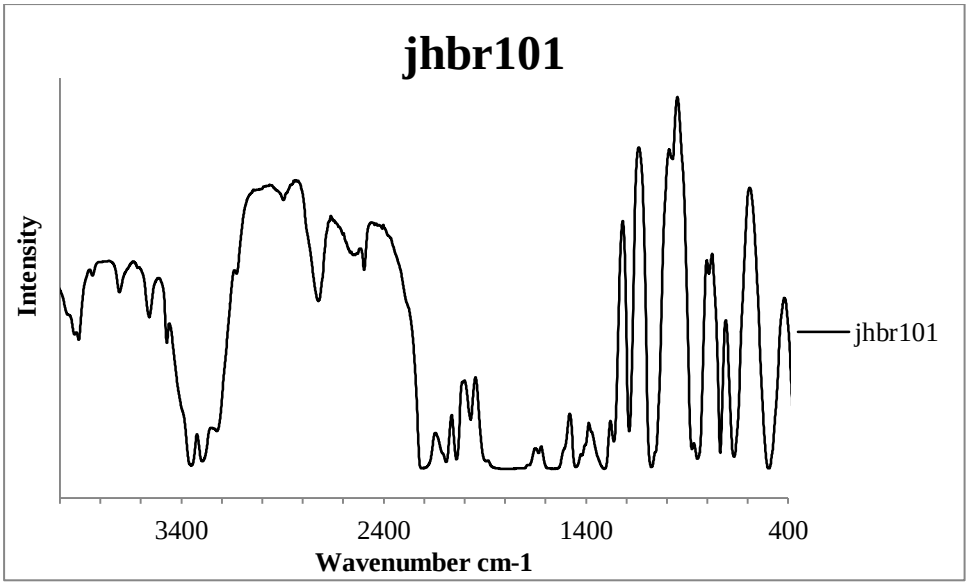
The complete IR spectrum of the liquid product after reaction of reaction jhbr99

Jhbr100



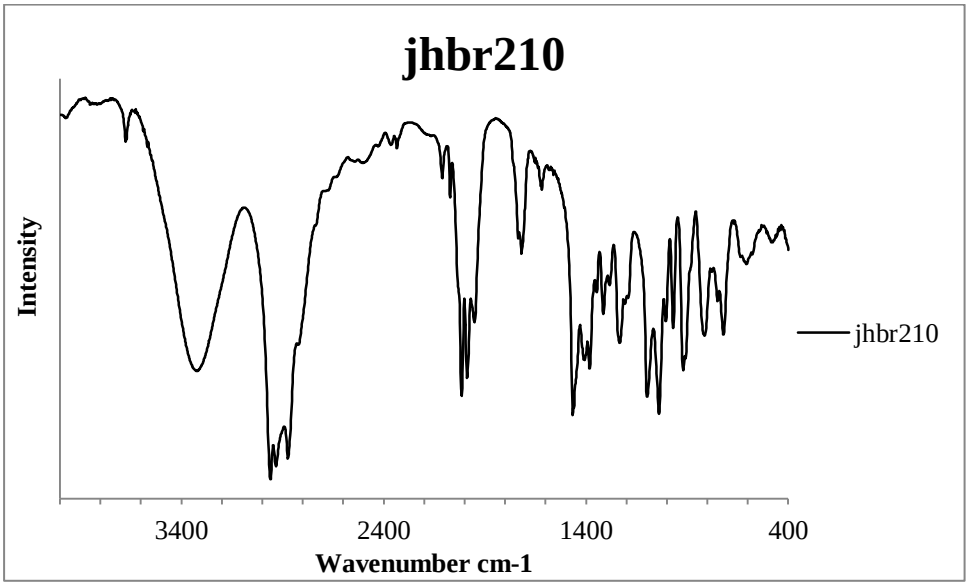
The complete IR spectrum of the liquid product after reaction of reaction jhbr100

Jhbr101

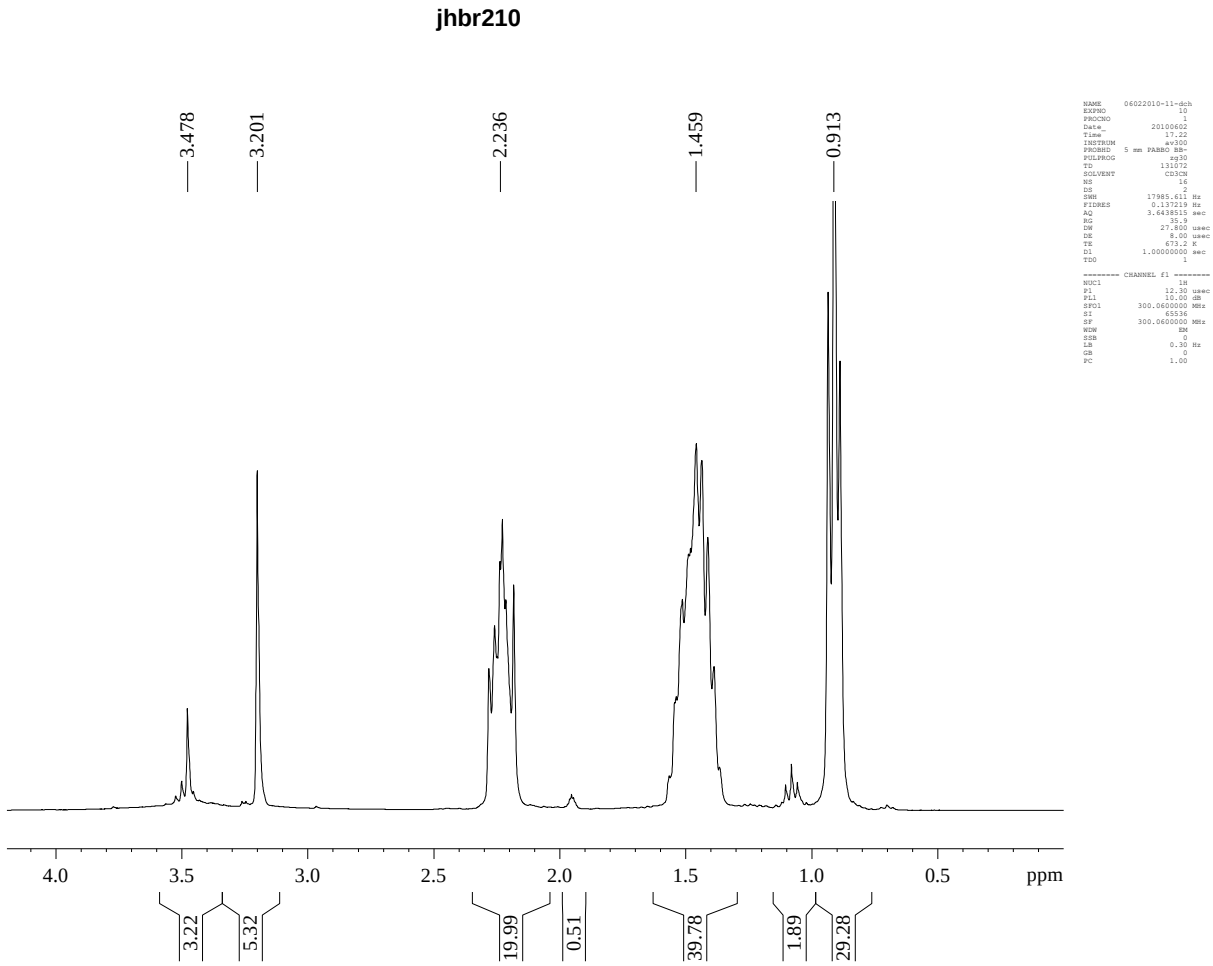


The complete IR spectrum of the liquid product after reaction of reaction jhbr101

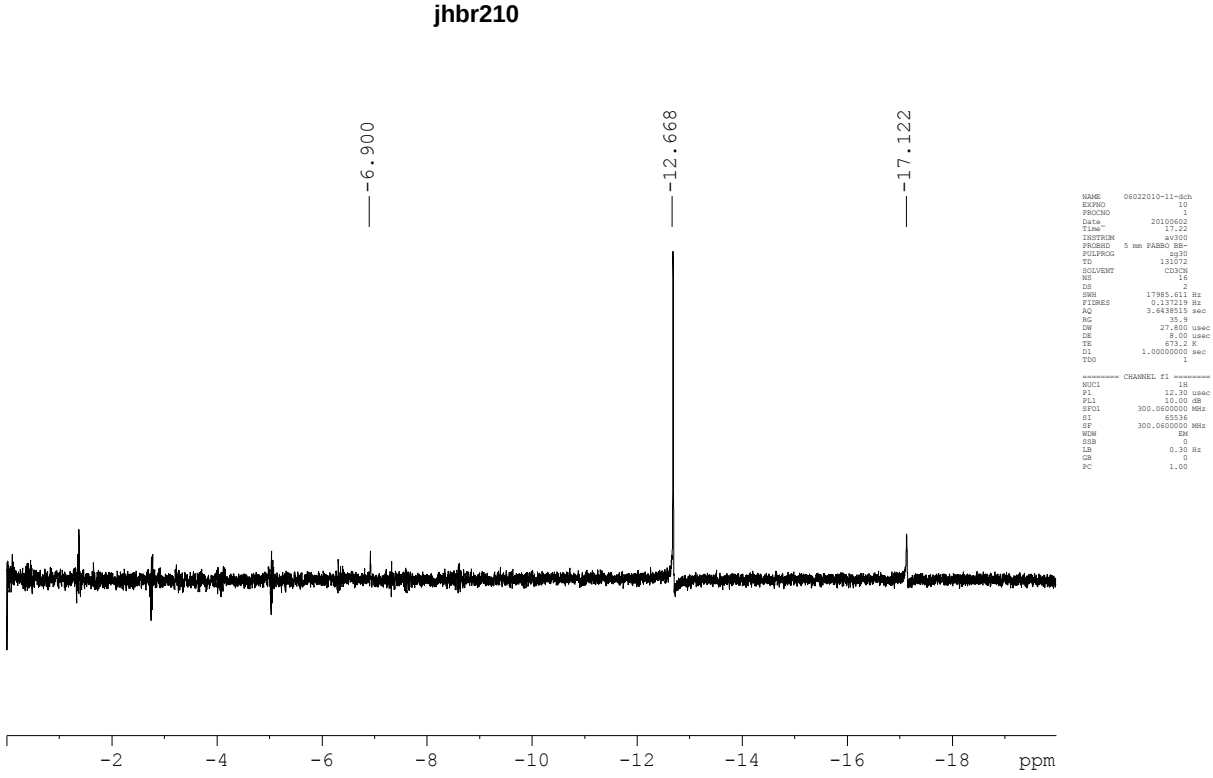
Jhbr210



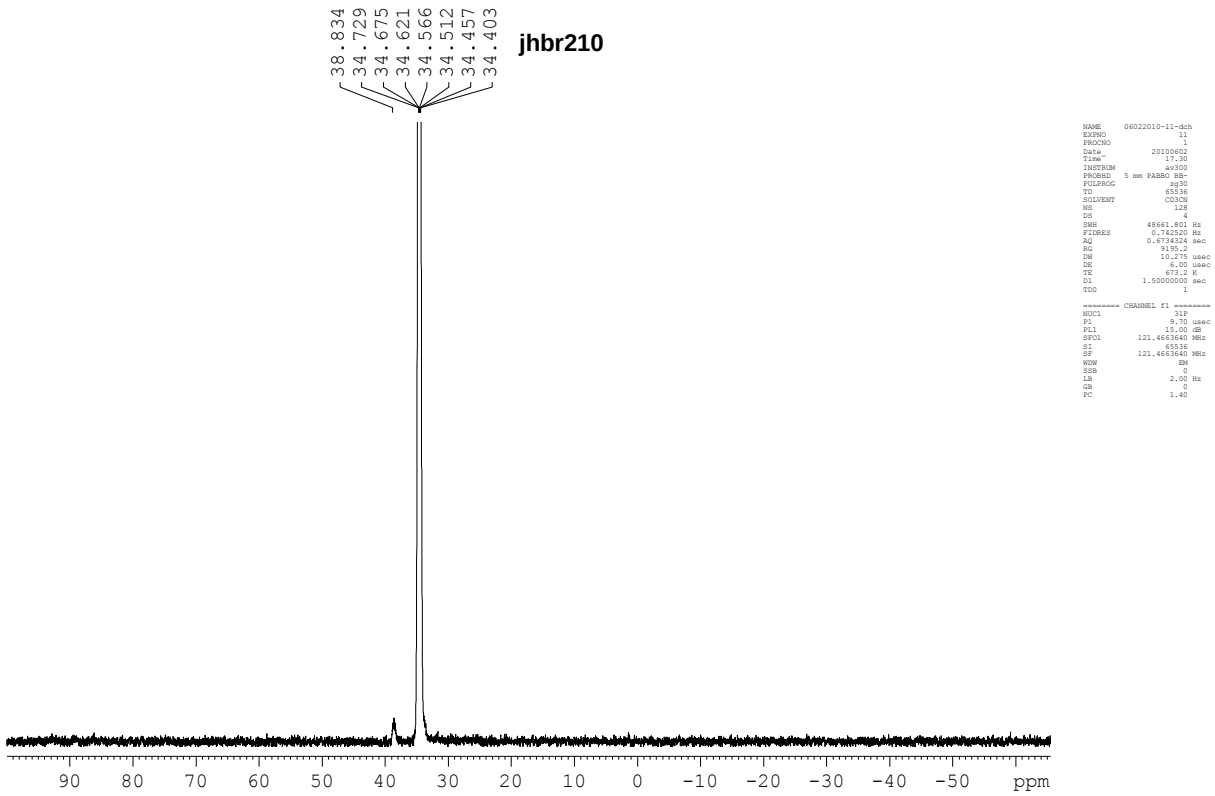
The complete IR spectrum of the liquid product after reaction of reaction jhbr210



The ^{1}H NMR of the liquid product after reaction jhbr210

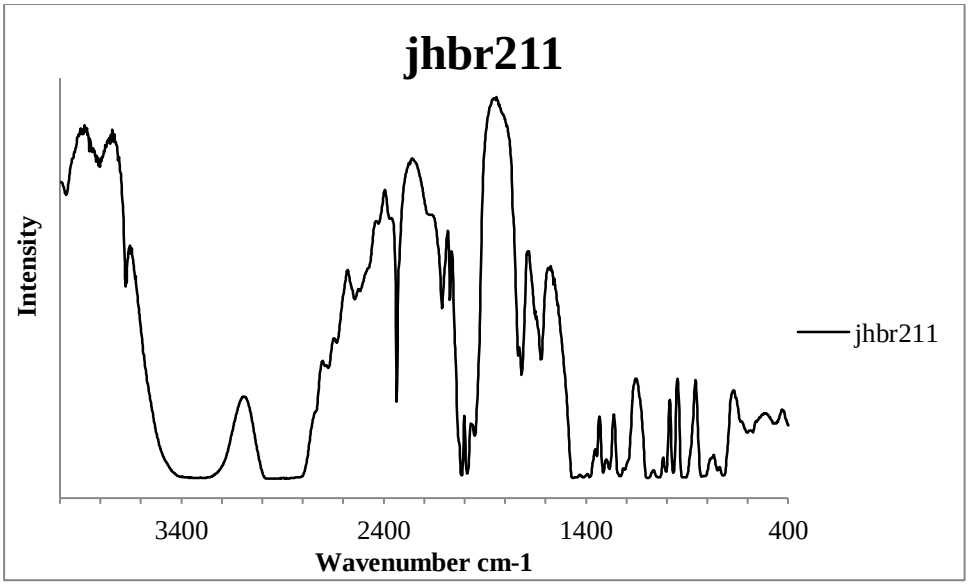


The ^{1}H NMR of the hydride region of the liquid product after reaction jhbr210



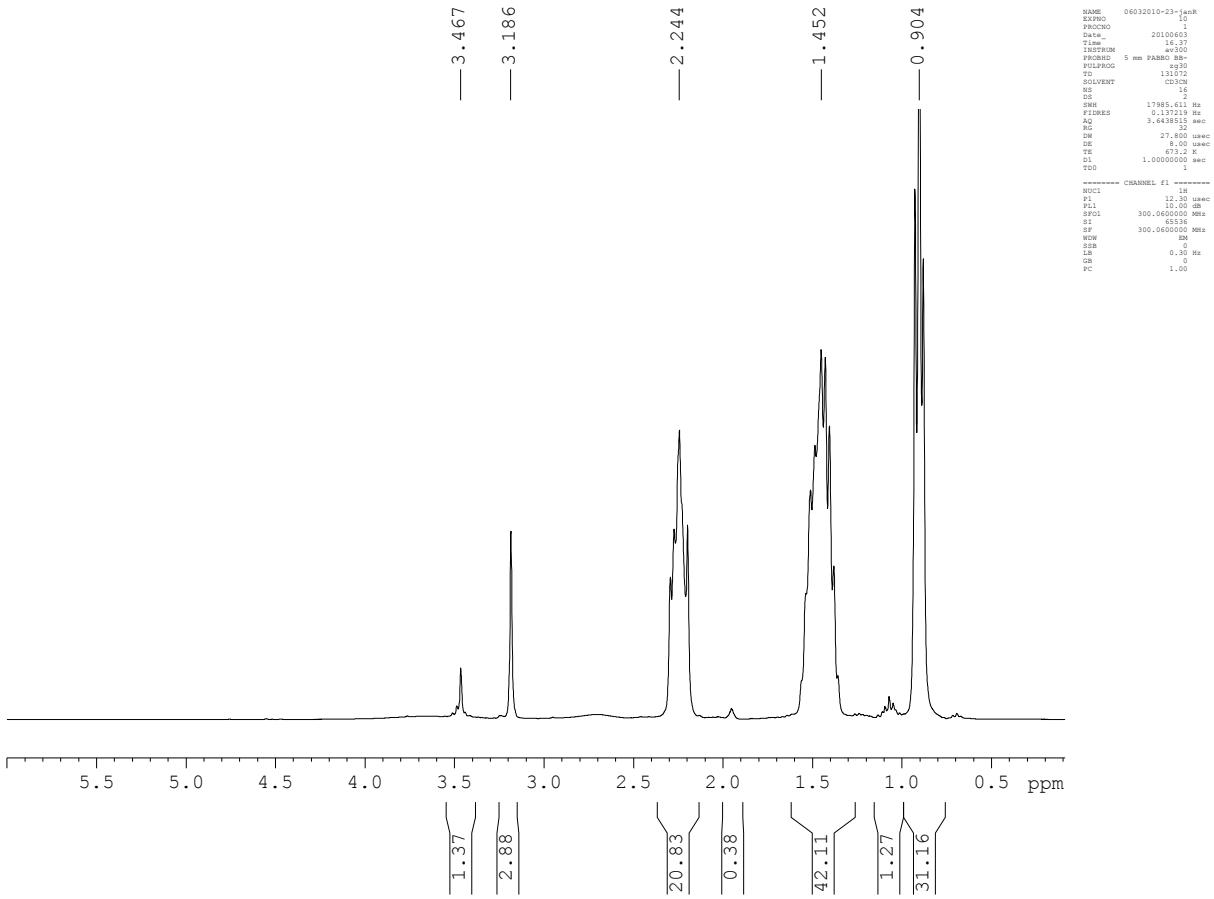
The ^{31}P NMR of the liquid product after reaction jhbr210

Jhbr211

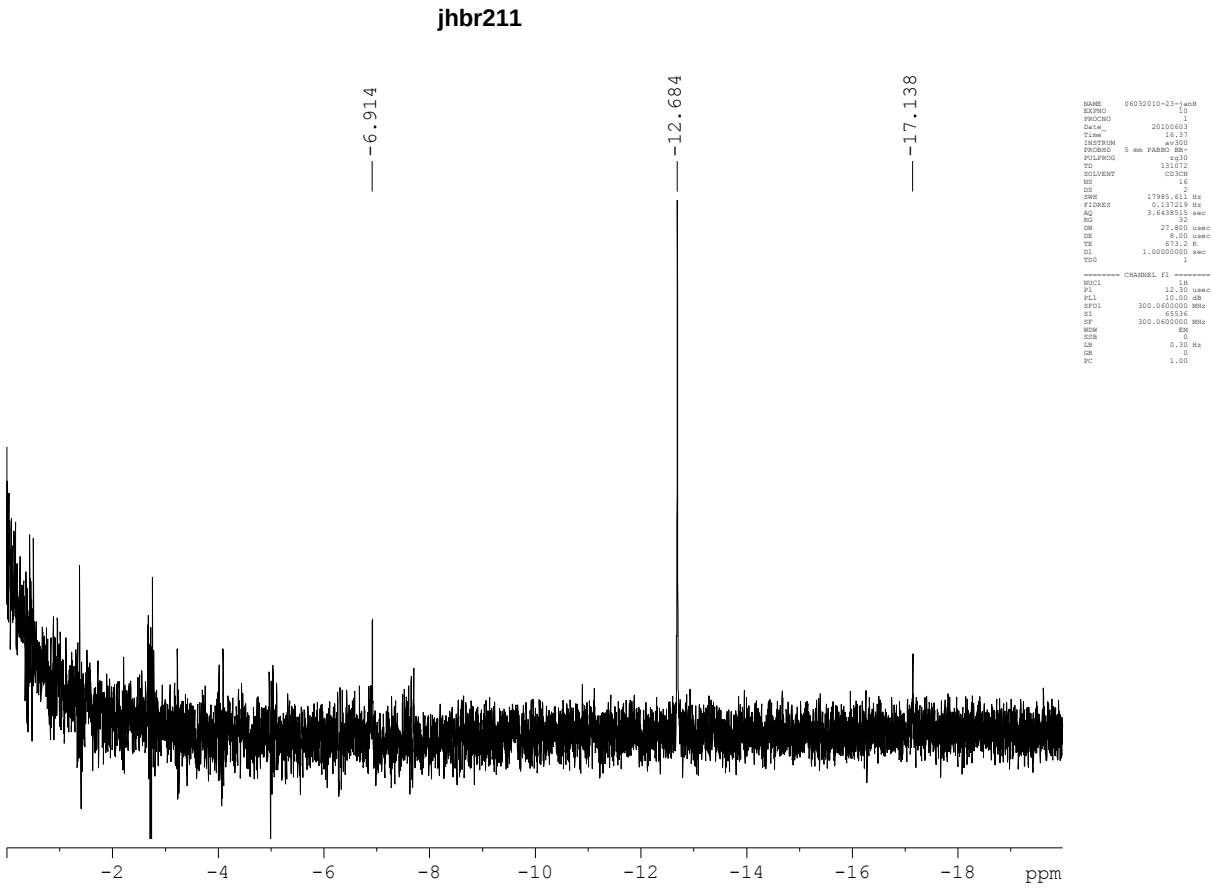


The complete IR spectrum of the liquid product after reaction of reaction jhbr211

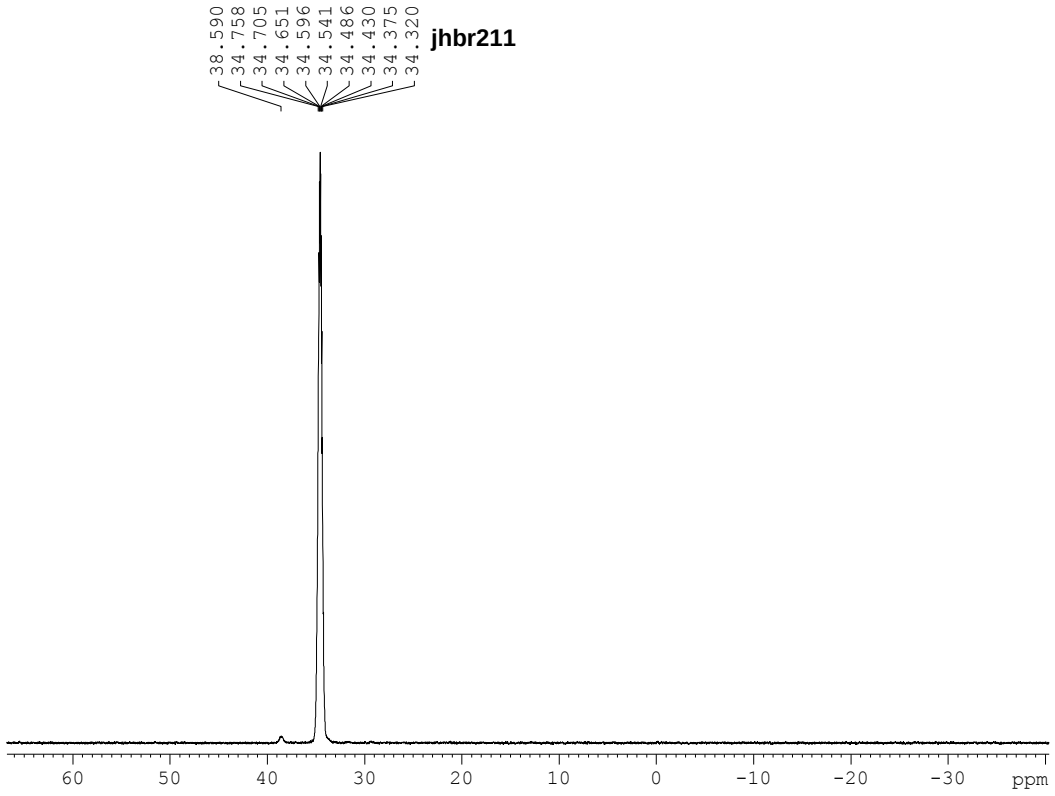
jhbr211



The ^{1}H NMR of the liquid product after reaction jhbr211

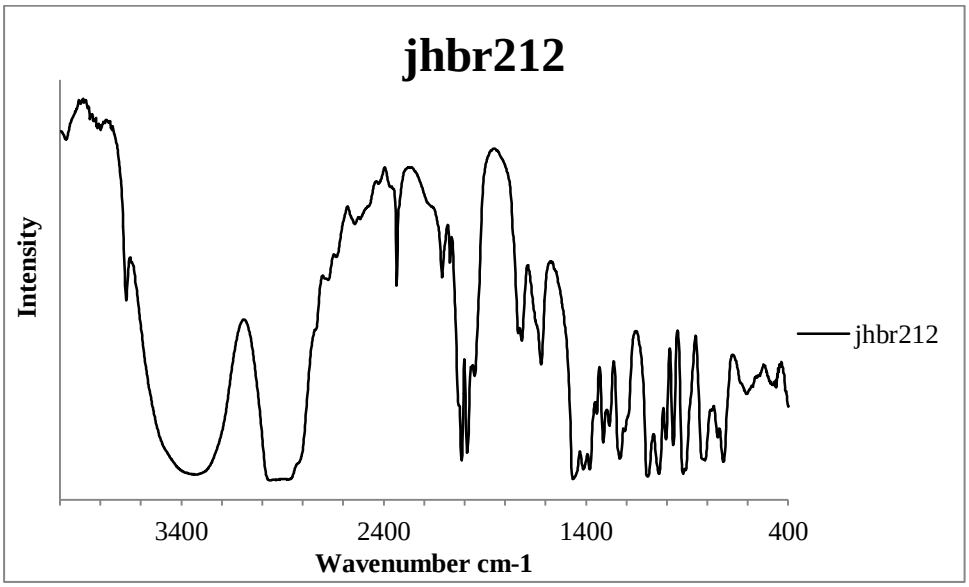


The ^{1}H NMR of the hydride region of the liquid product after reaction jhbr211

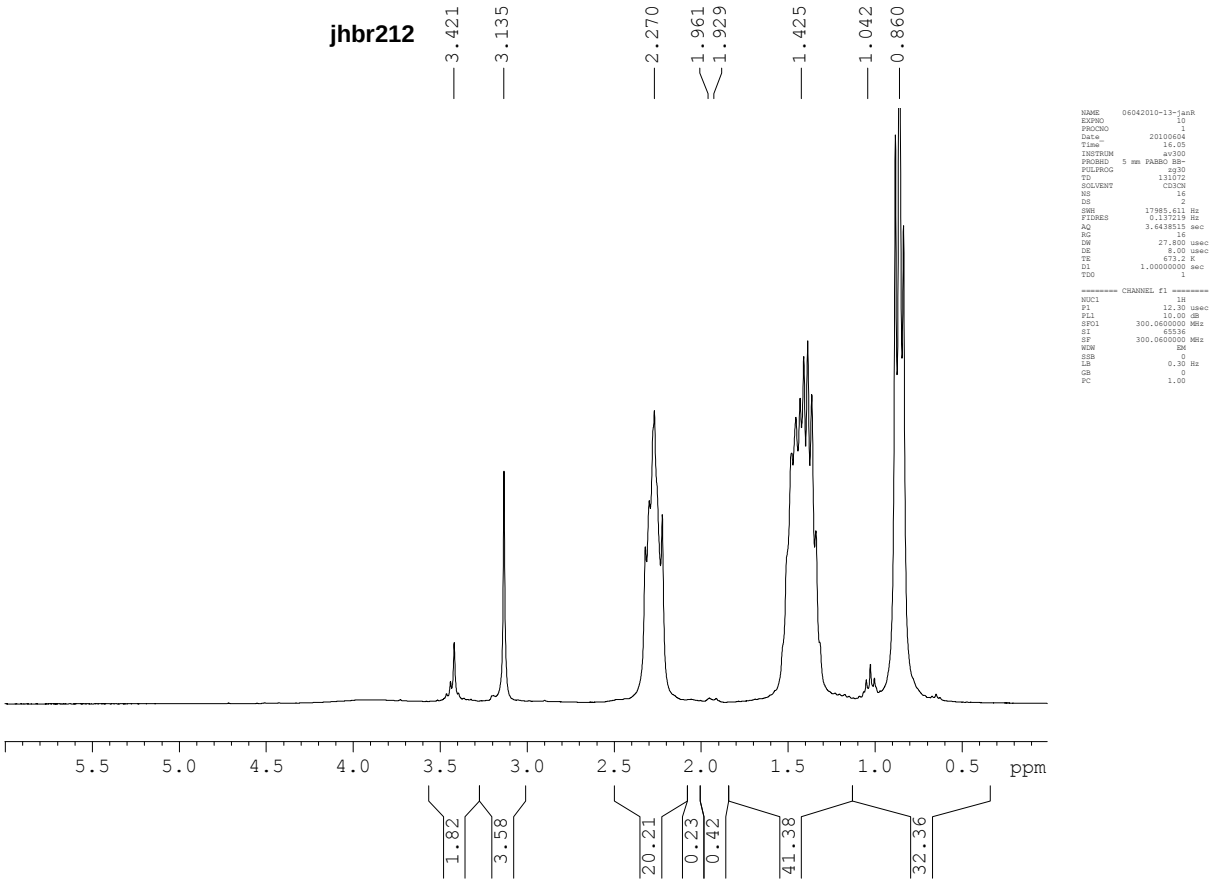


The ^{31}P NMR of the liquid product after reaction jhbr211

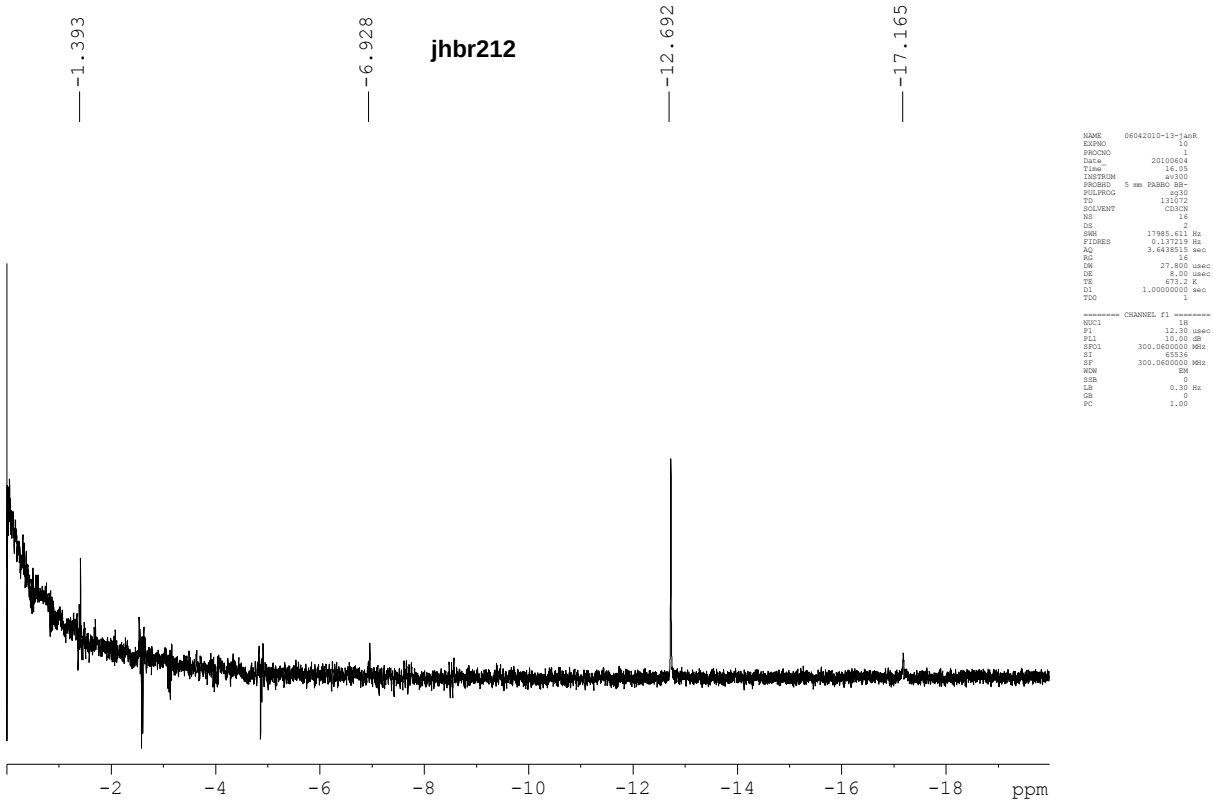
jhbr212



The complete IR spectrum of the liquid product after reaction of reaction jhbr212



The ^{1}H NMR of the liquid product after reaction jhbr212



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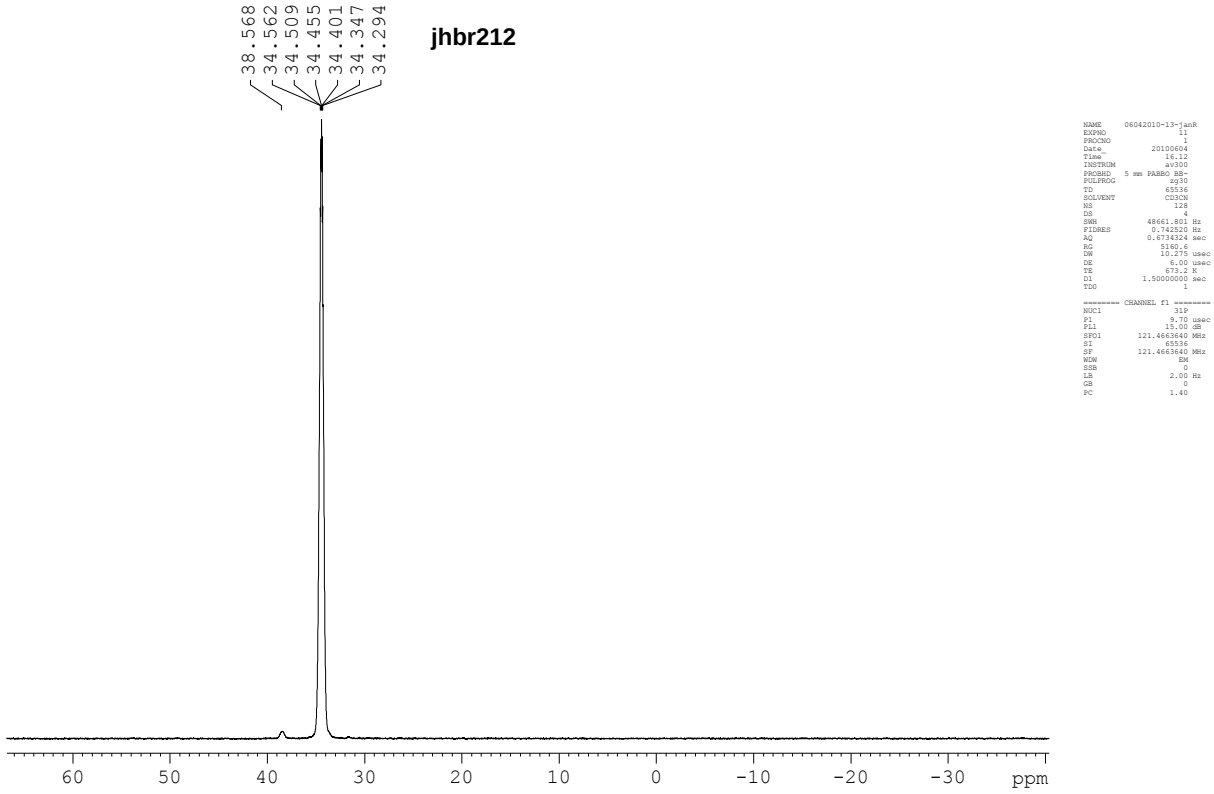
TD 131072
SOLVENT CD3CN
NS 16
DS 2
SWH 17985.611 Hz
FIDRES 0.137219 Hz
AQ 3.6438515 sec
RG 16
DW 27.800 usec
DE 8.00 usec
TE 673.2 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 12.30 usec
PL1 10.00 dB
SFO1 300.0600000 MHz
SI 65536
SF 300.0600000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

The ^{1}H NMR of the hydride region of the liquid product after reaction jhbr212



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PULPROG zg30
TD 65536
SOLVENT CD3CN
NS 128
DS 4
SWH 48661.801 Hz
FIDRES 0.742520 Hz
AQ 0.6734324 sec
RG 5160.6
DW 10.275 usec
DE 6.00 usec
TE 673.2 K
D1 1.50000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 31P
P1 9.70 usec
PL1 15.00 dB
SFO1 121.4663640 MHz
SI 65536
SF 121.4663640 MHz
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SSB 0
LB 2.00 Hz
GB 0
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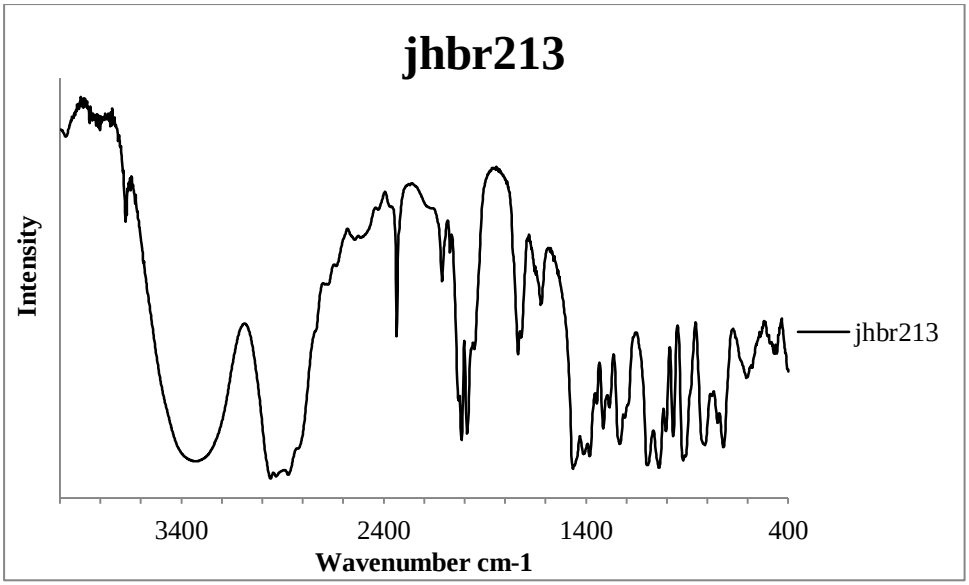
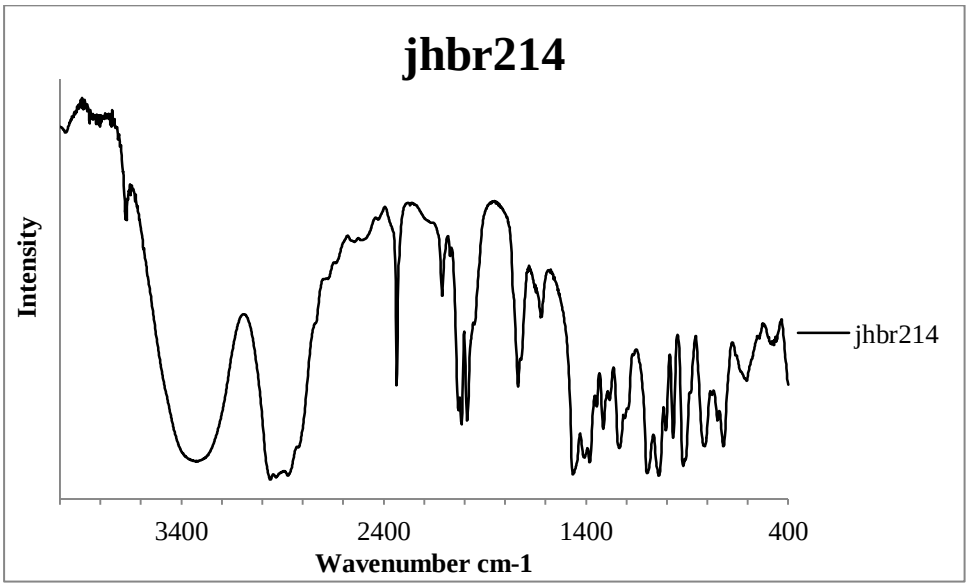
The ^{31}P NMR of the liquid product after reaction jhbr212

Jhbr213



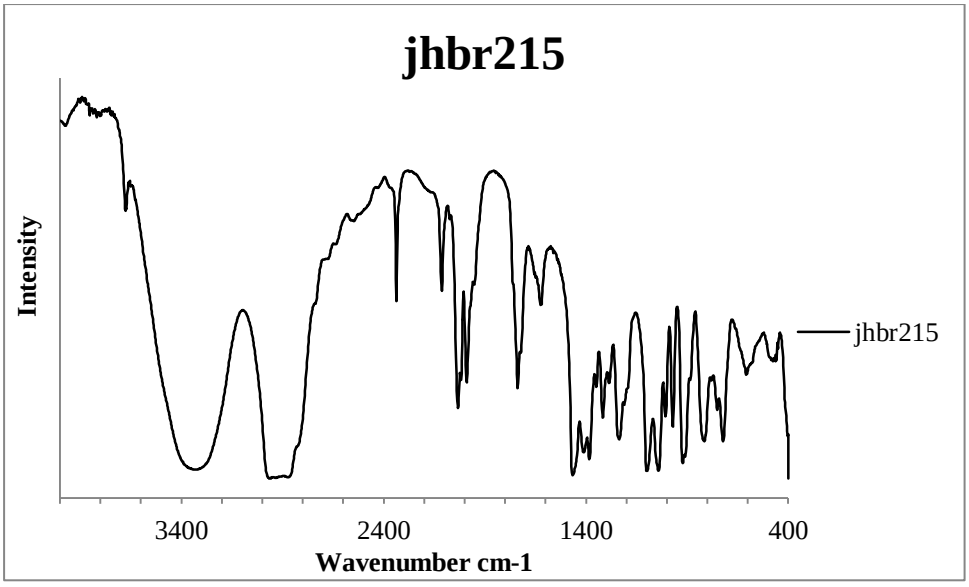
The complete IR spectrum of the liquid product after reaction of reaction jhbr213

Jhbr214



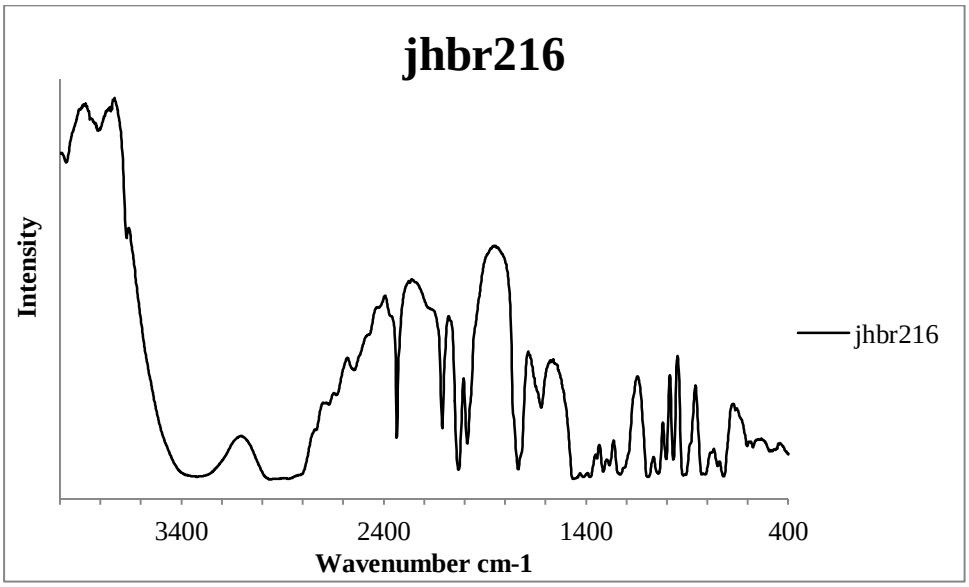
The complete IR spectrum of the liquid product after reaction of reaction jhbr214

Jhbr215

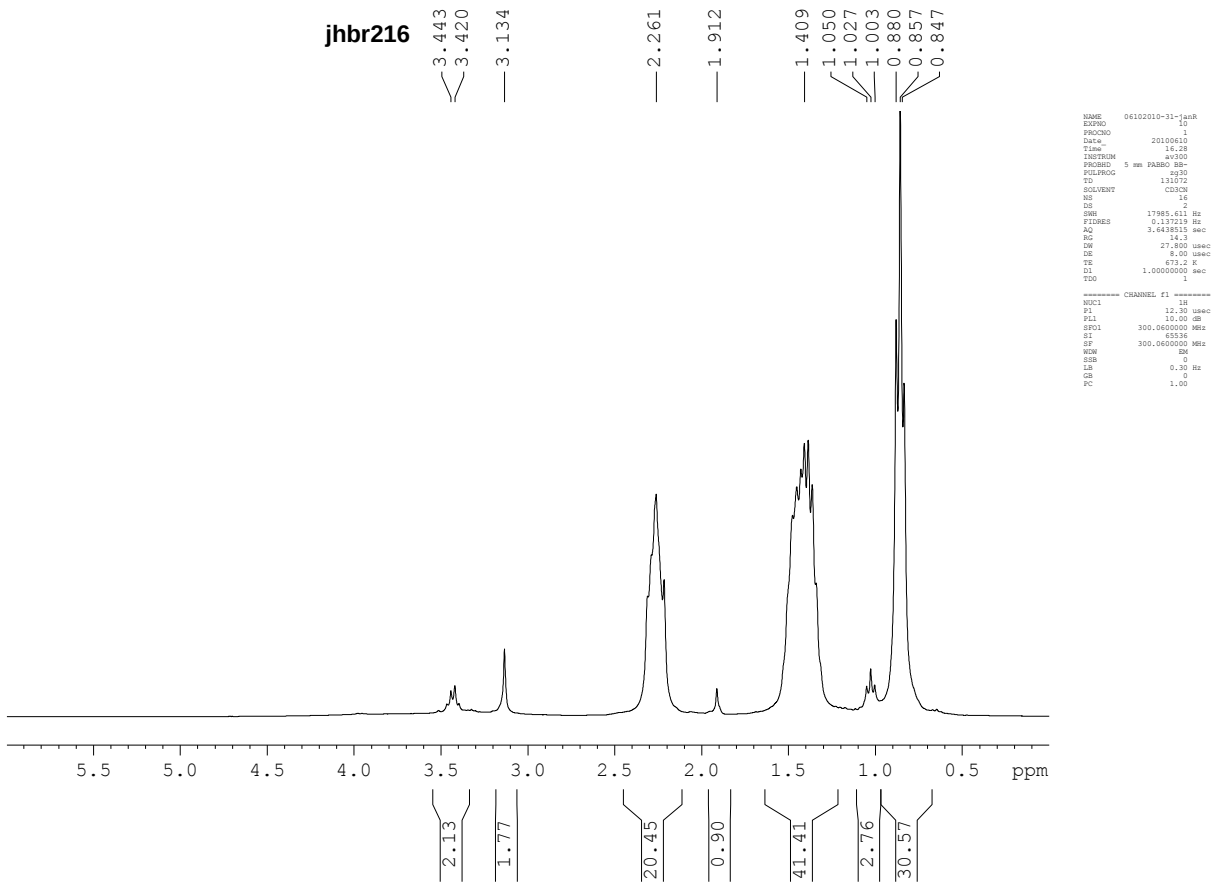


215

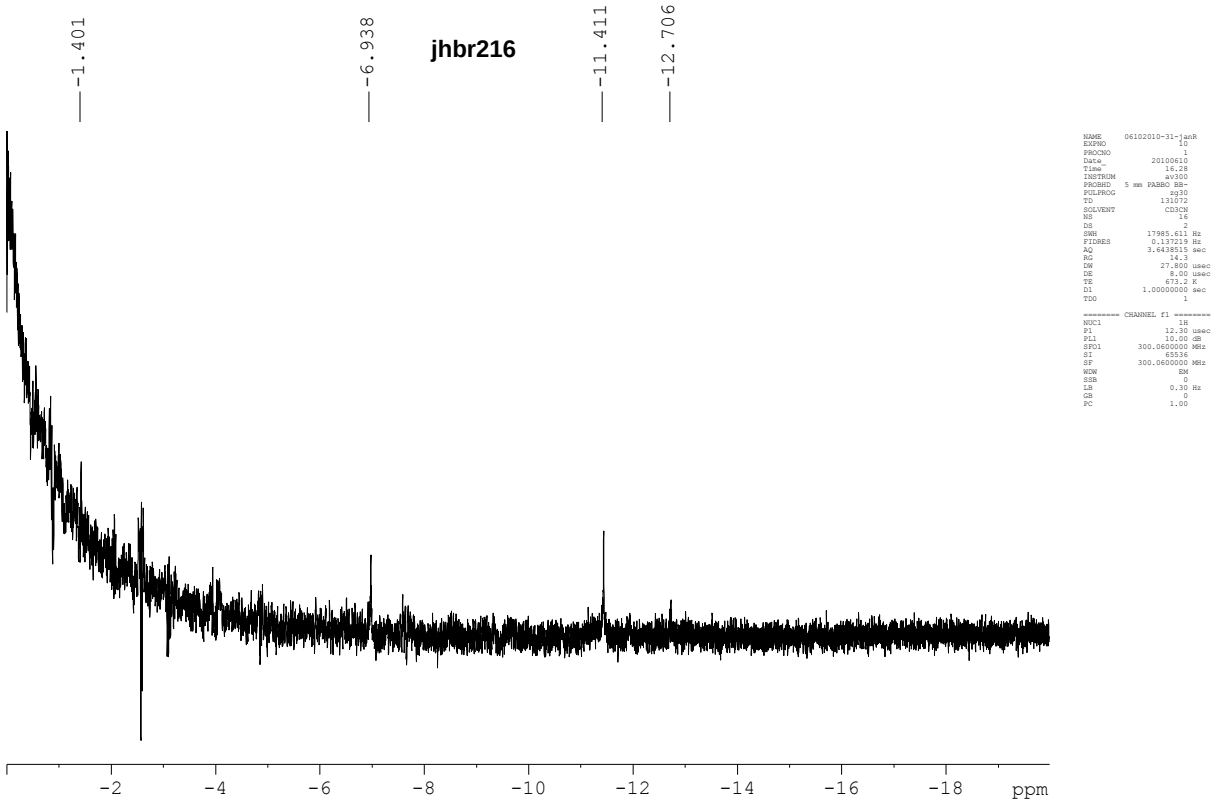
jhbr216



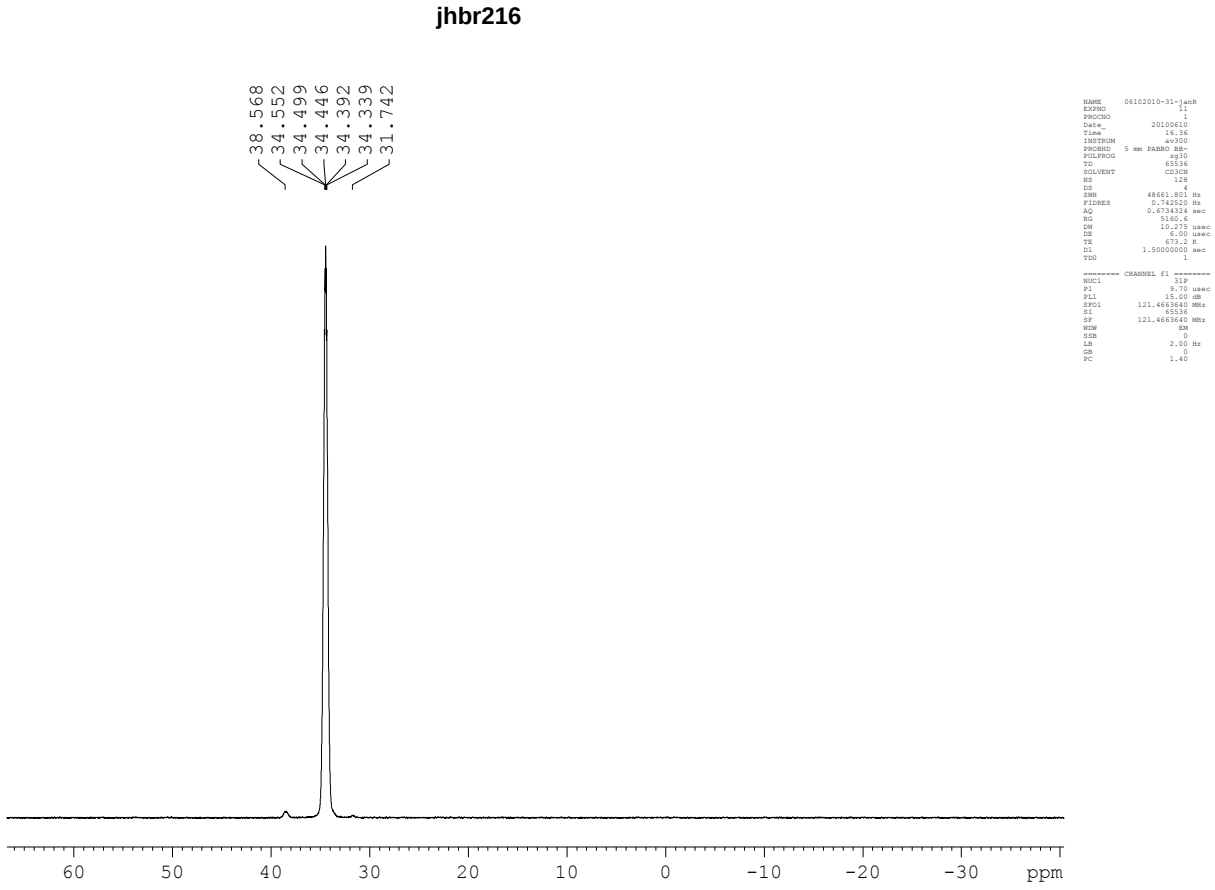
The complete IR spectrum of the liquid product after reaction of reaction jhbr216



The ^{1}H NMR of the liquid product after reaction jhbr216

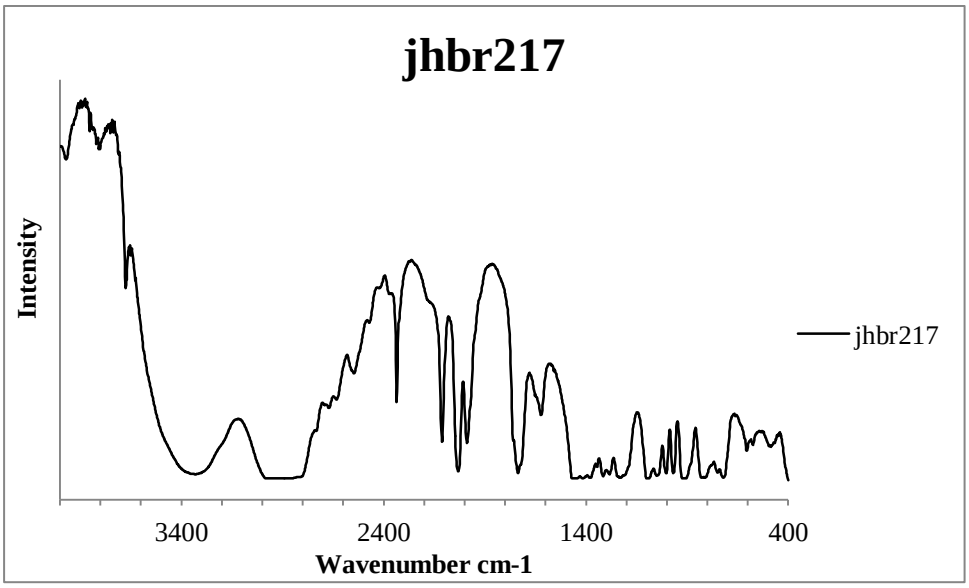


The ^{1}H NMR of the hydride region of the liquid product after reaction jhbr216

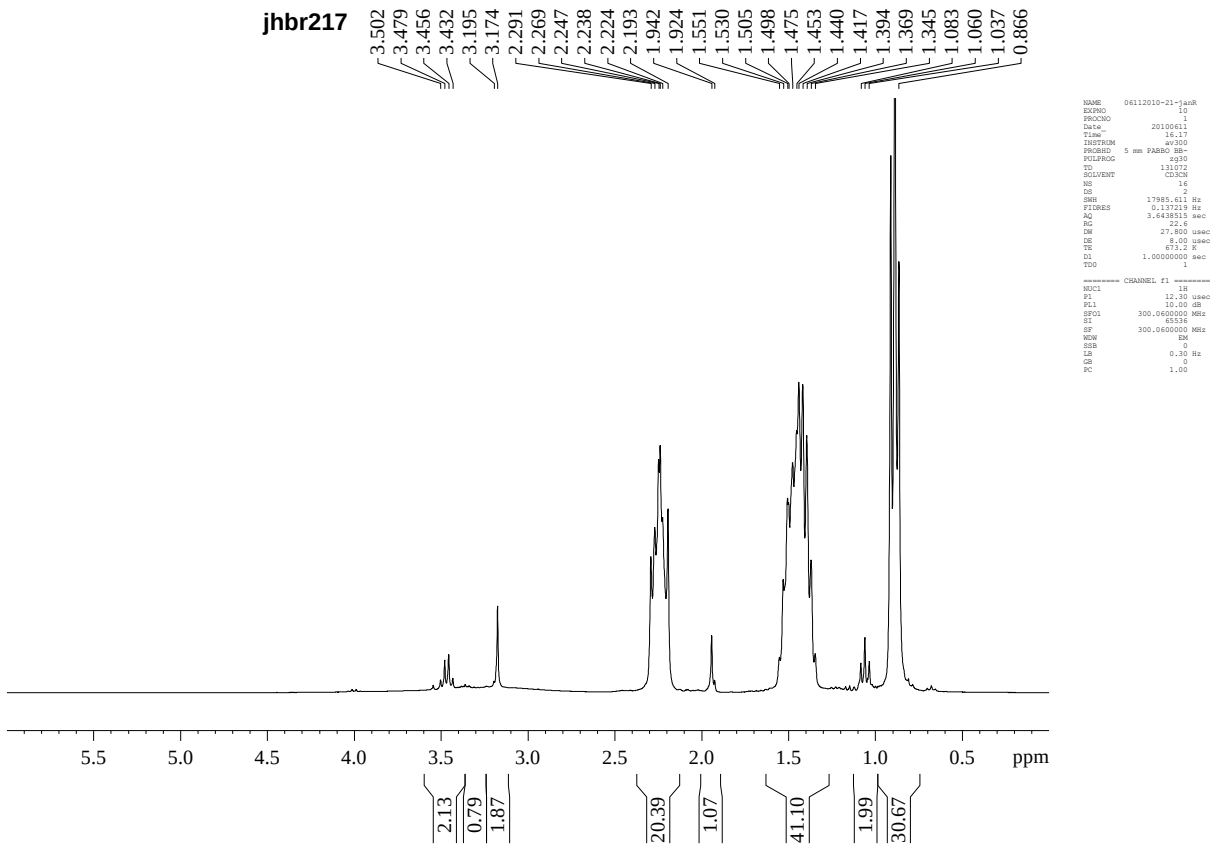


The ^{31}P NMR of the liquid product after reaction jhbr216

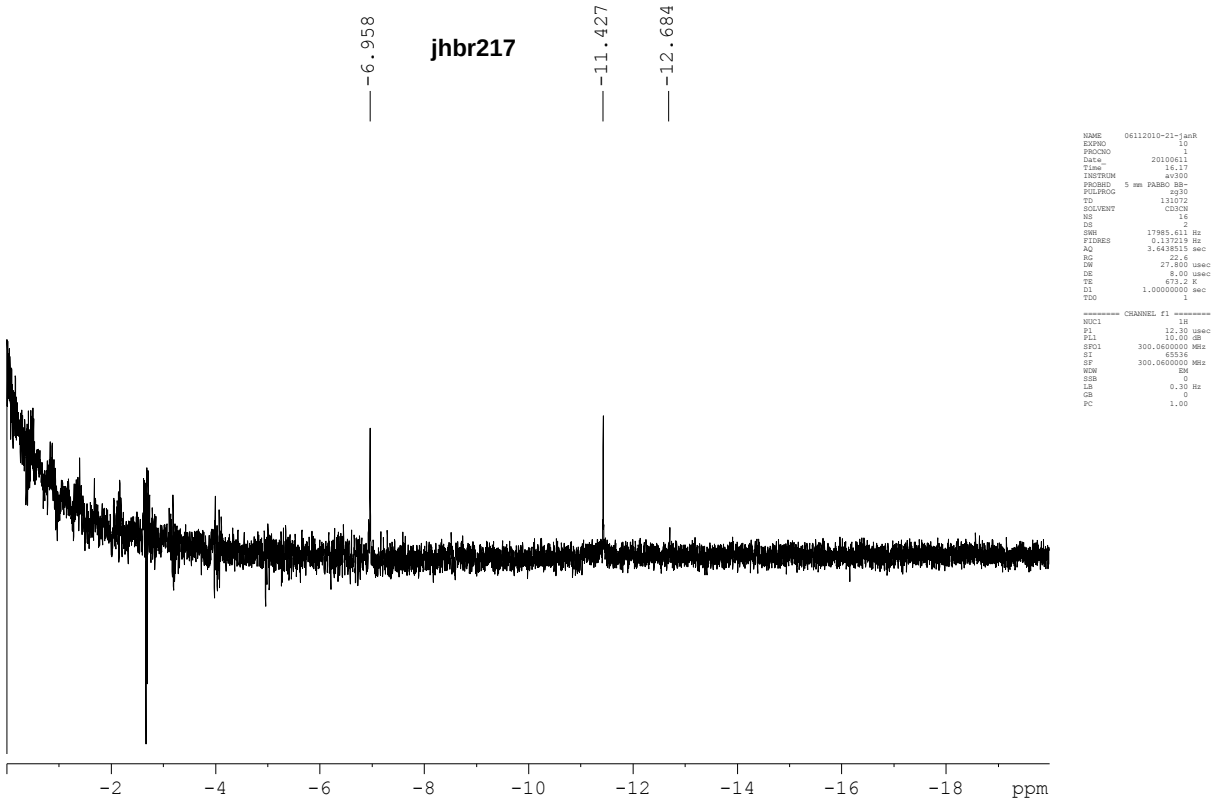
jhbr217



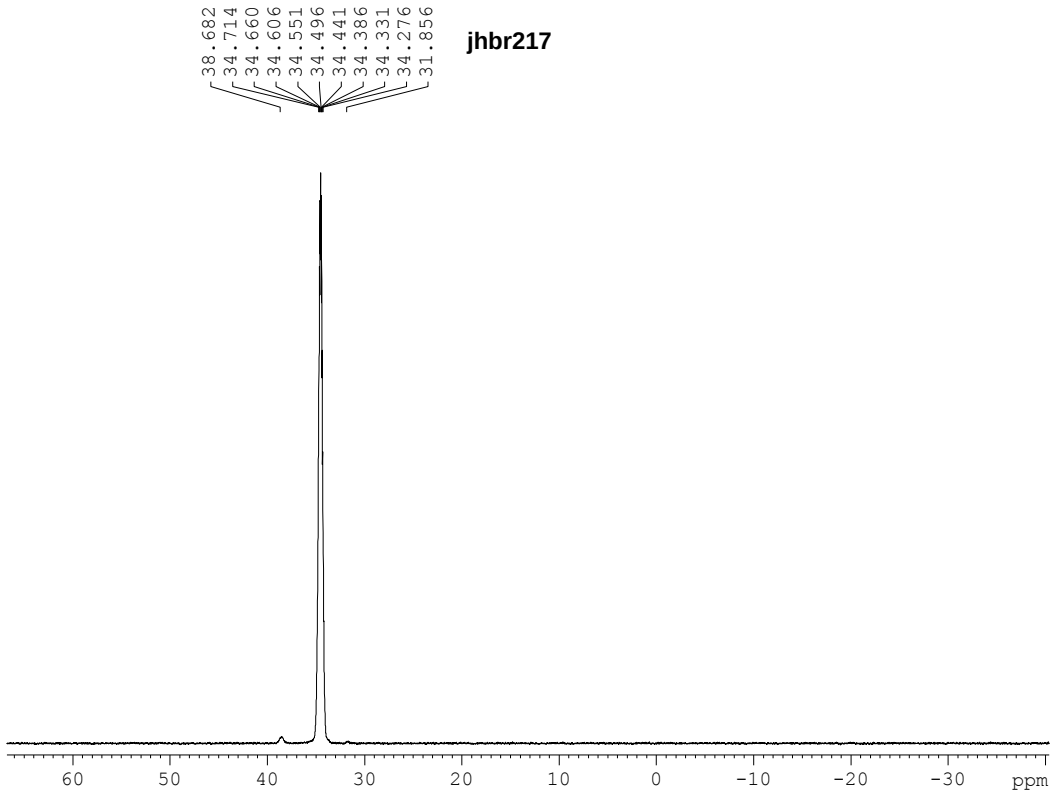
The complete IR spectrum of the liquid product after reaction of reaction jhbr217



The ^{1}H NMR of the liquid product after reaction jhbr217

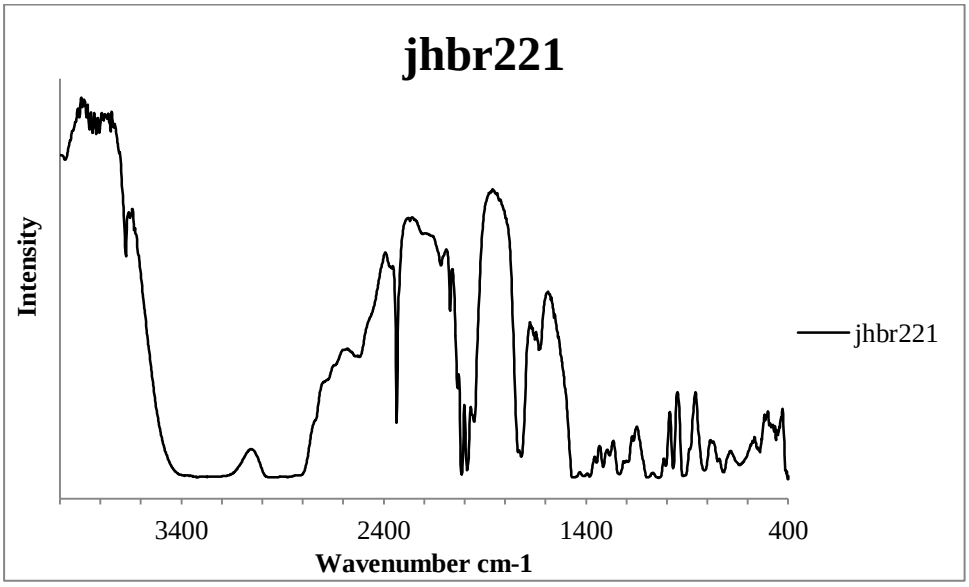


The ^{1}H NMR of the hydride region of the liquid product after reaction jhbr217

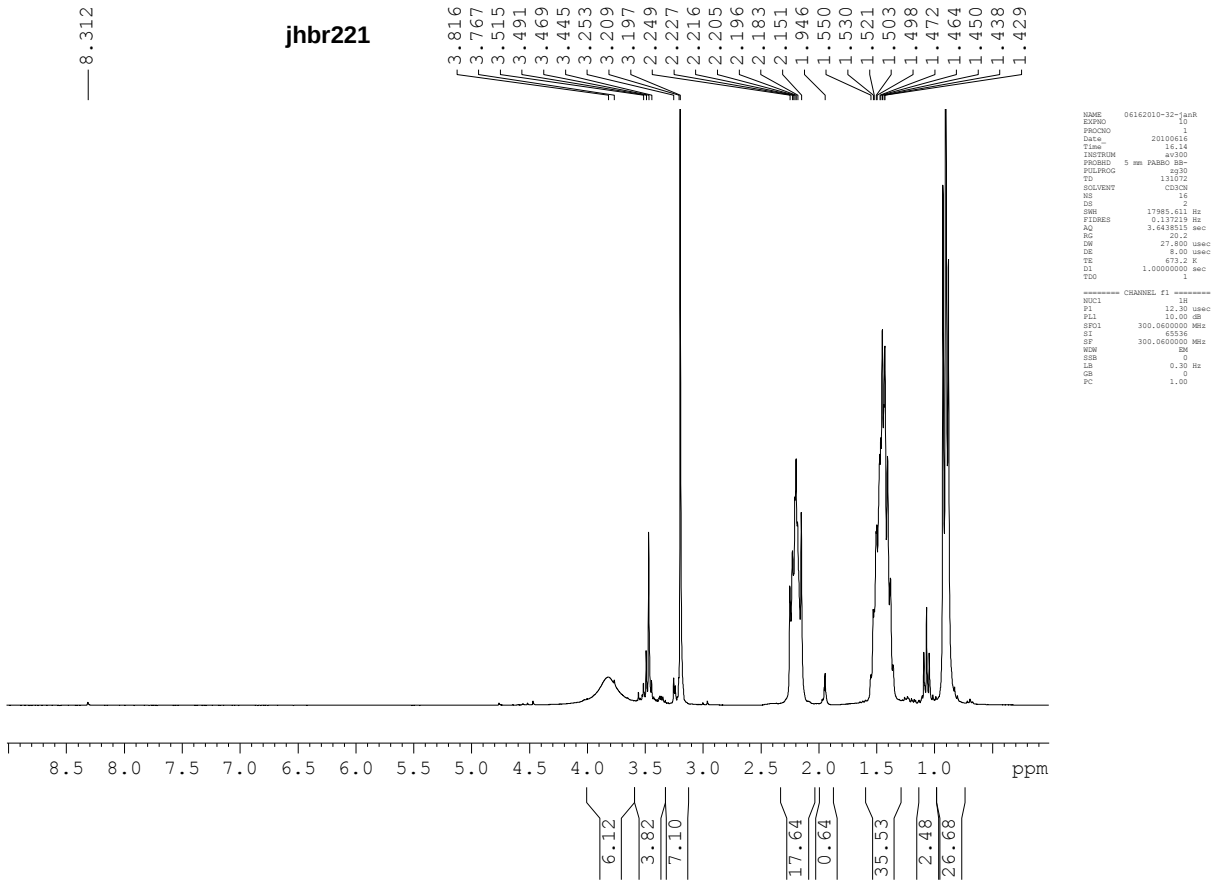


The ^{31}P NMR of the liquid product after reaction jhbr217

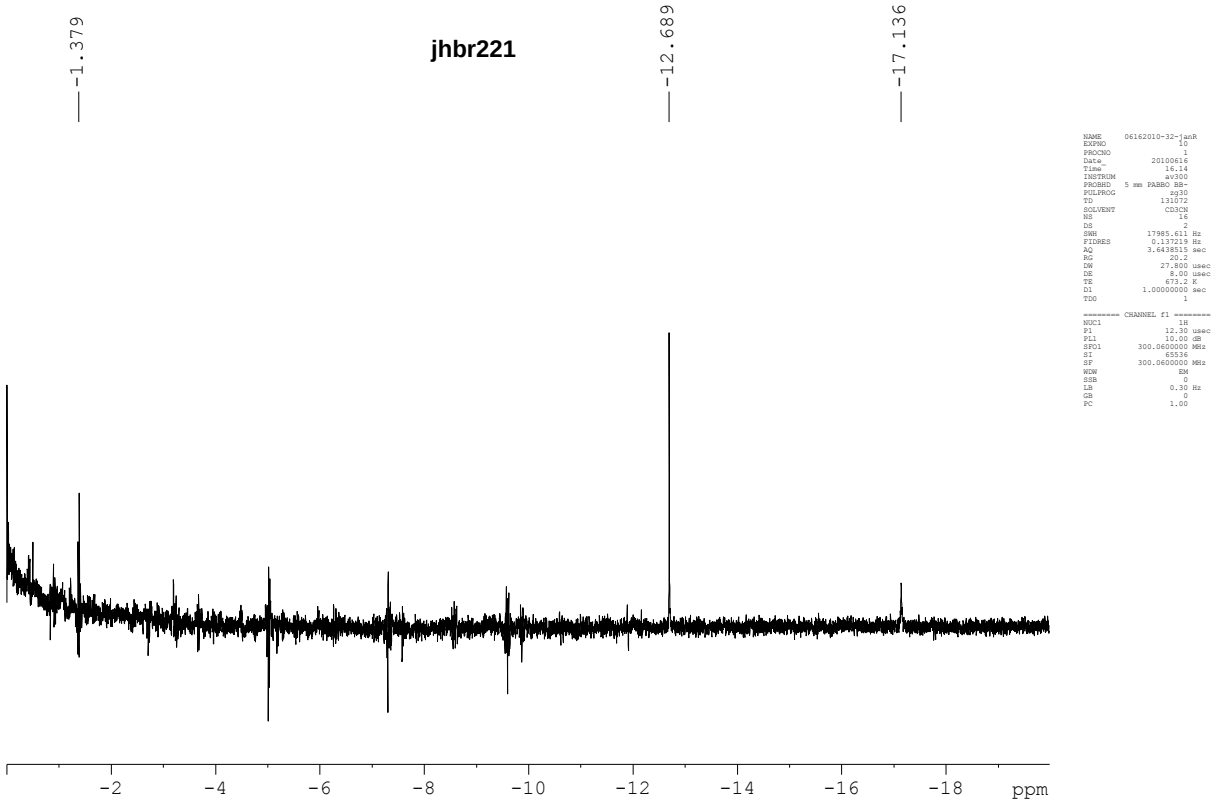
jhbr221



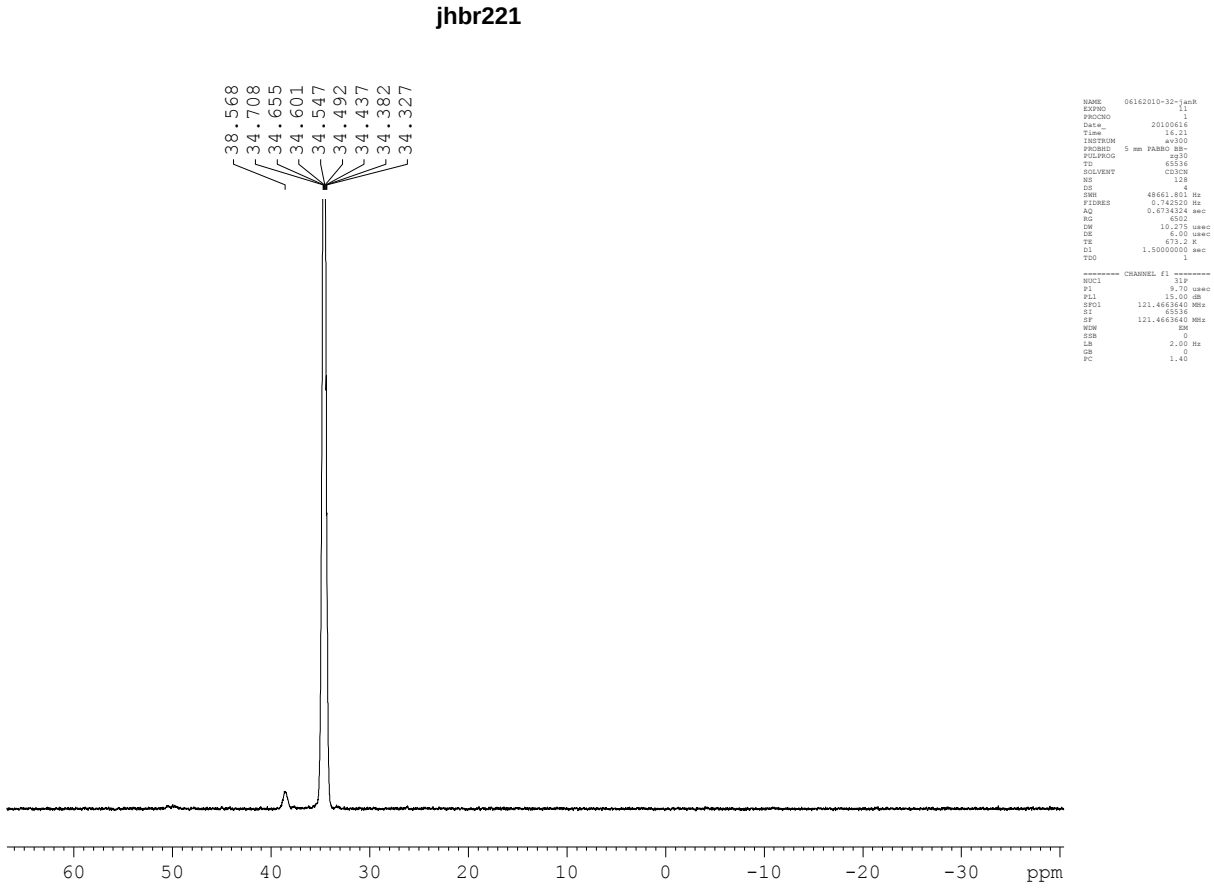
The complete IR spectrum of the liquid product after reaction of reaction jhbr221



The ^{1}H NMR of the liquid product after reaction jhbr221

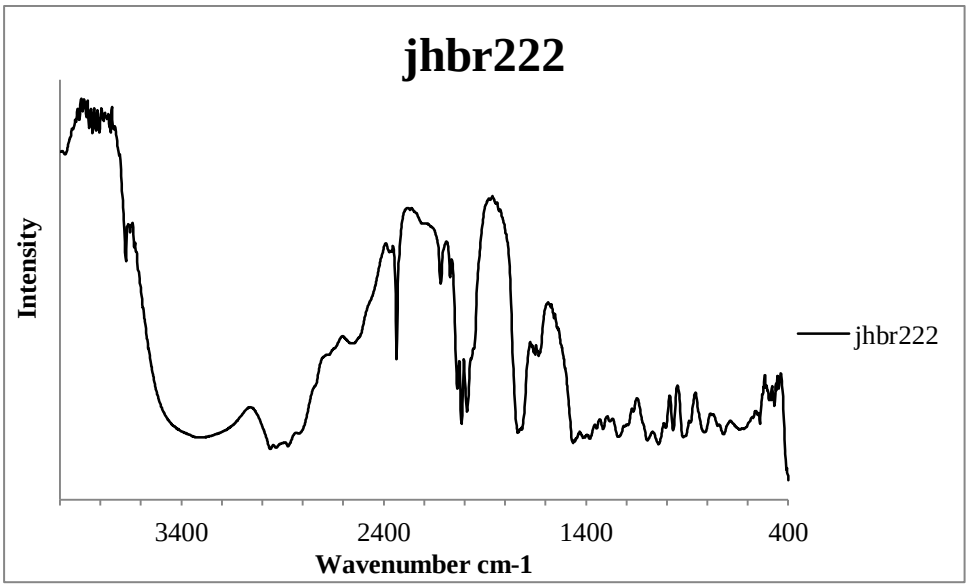


The ^{1}H NMR of the hydride region of the liquid product after reaction jhbr221

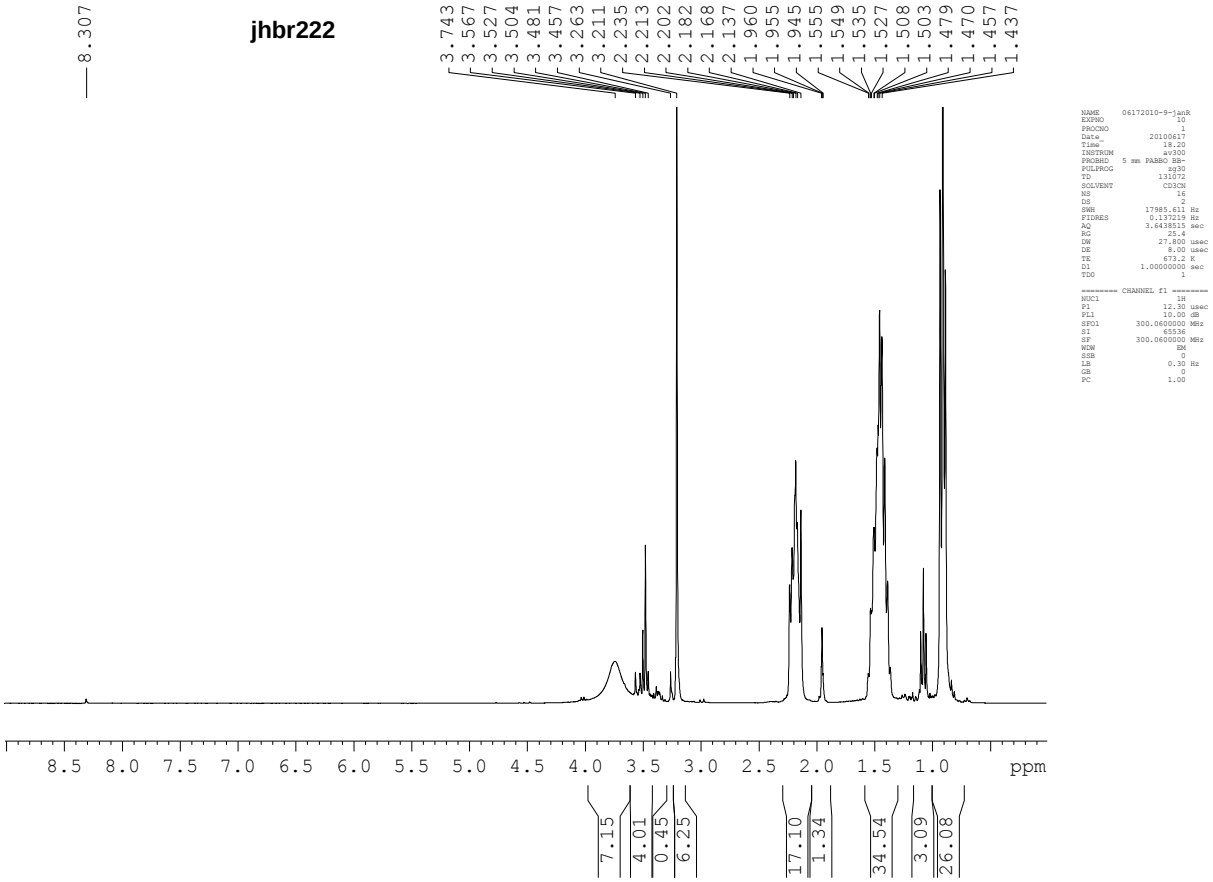


The ^{31}P NMR of the liquid product after reaction jhbr221

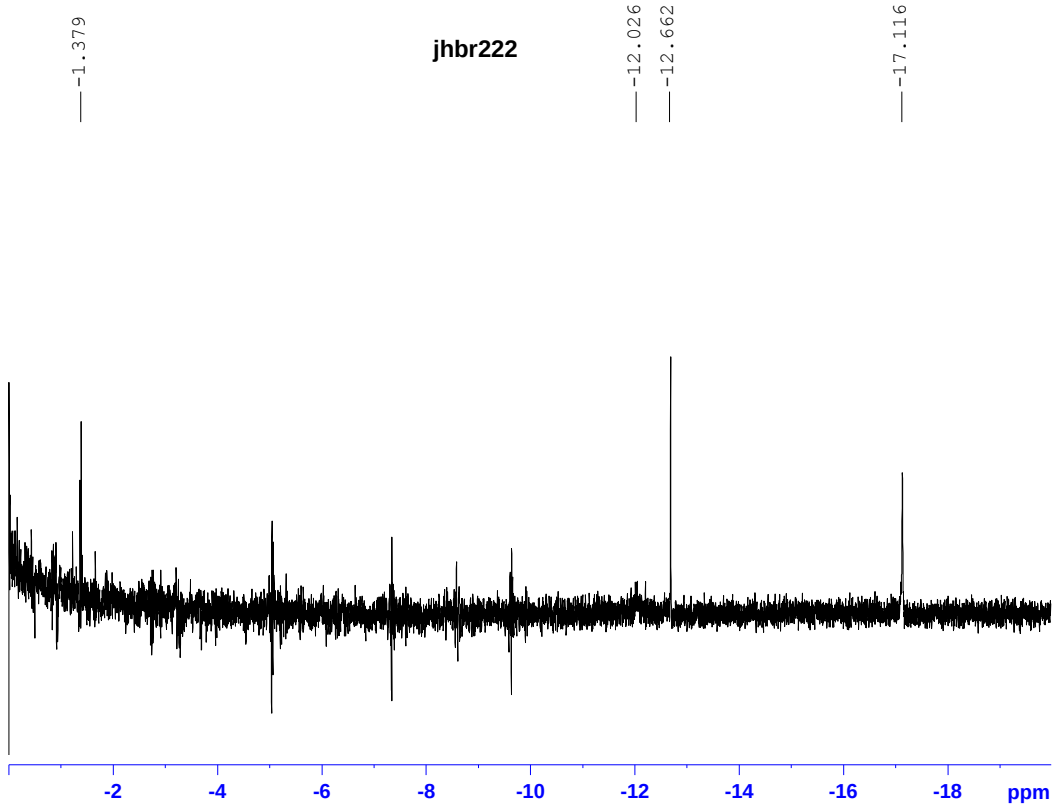
jhbr222



The complete IR spectrum of the liquid product after reaction of reaction jhbr222

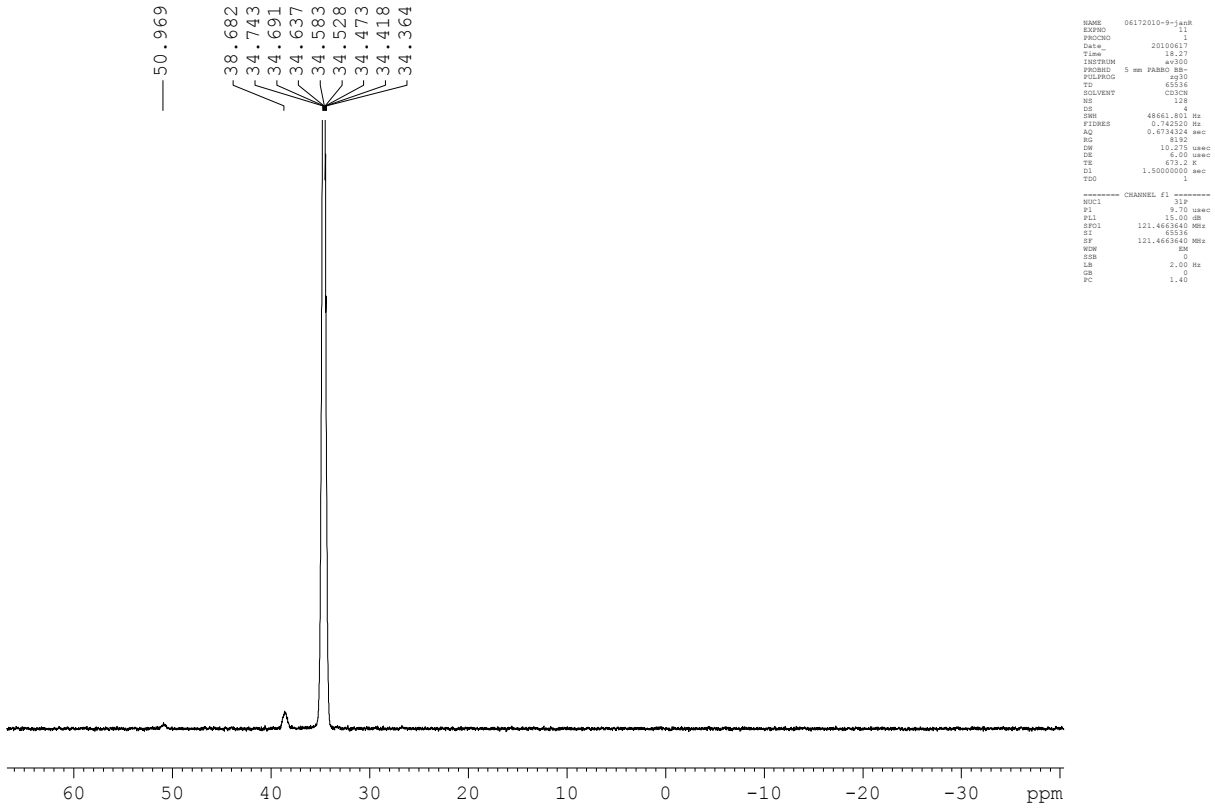


The ^{1}H NMR of the liquid product after reaction jhbr222



The ^{1}H NMR of the hydride region of the liquid product after reaction jhbr222

jhbr222



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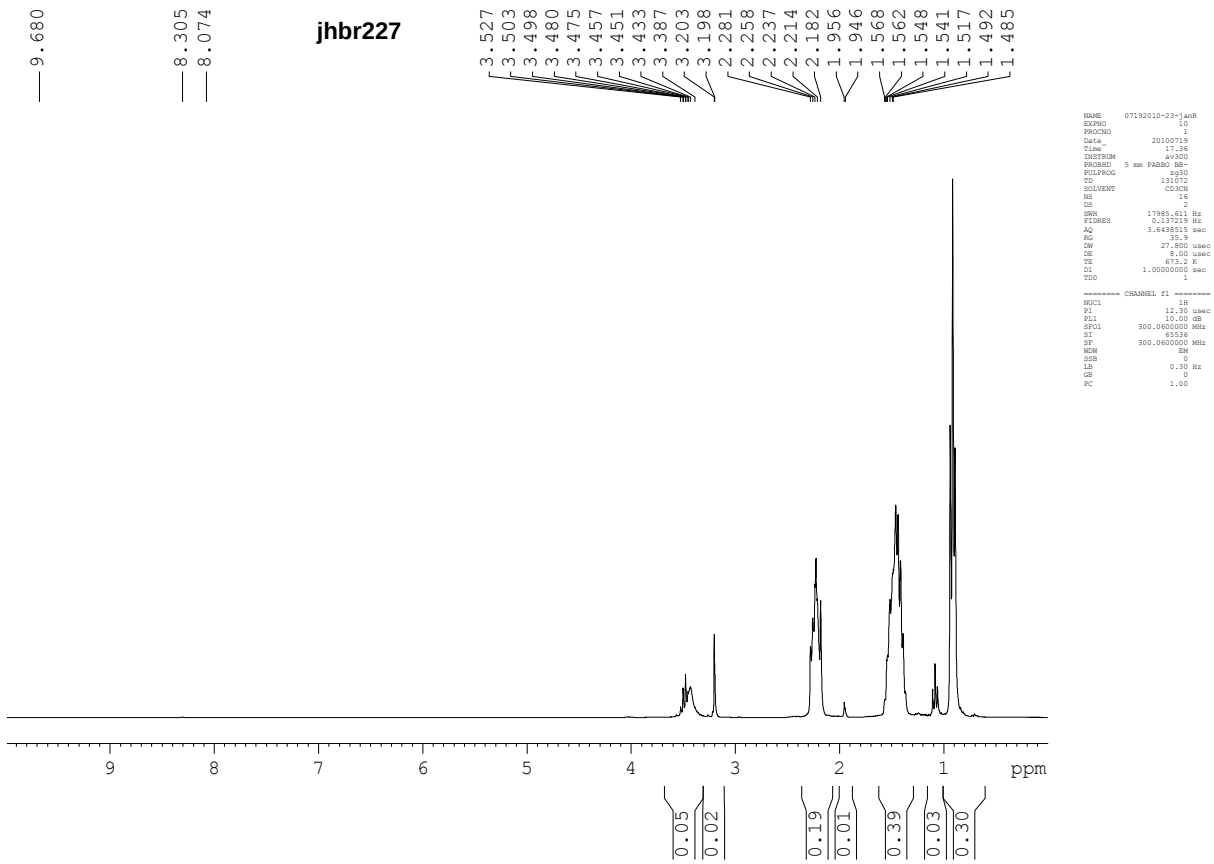
TD 65536
SOLVENT CD3CN
NS 128
DS 4
SWH 48661.801 Hz
FIDRES 0.742520 Hz
AQ 0.6734324 sec
RG 8192
DW 10.275 usec
DE 6.00 usec
TE 673.2 K
D1 1.50000000 sec
TD0 1

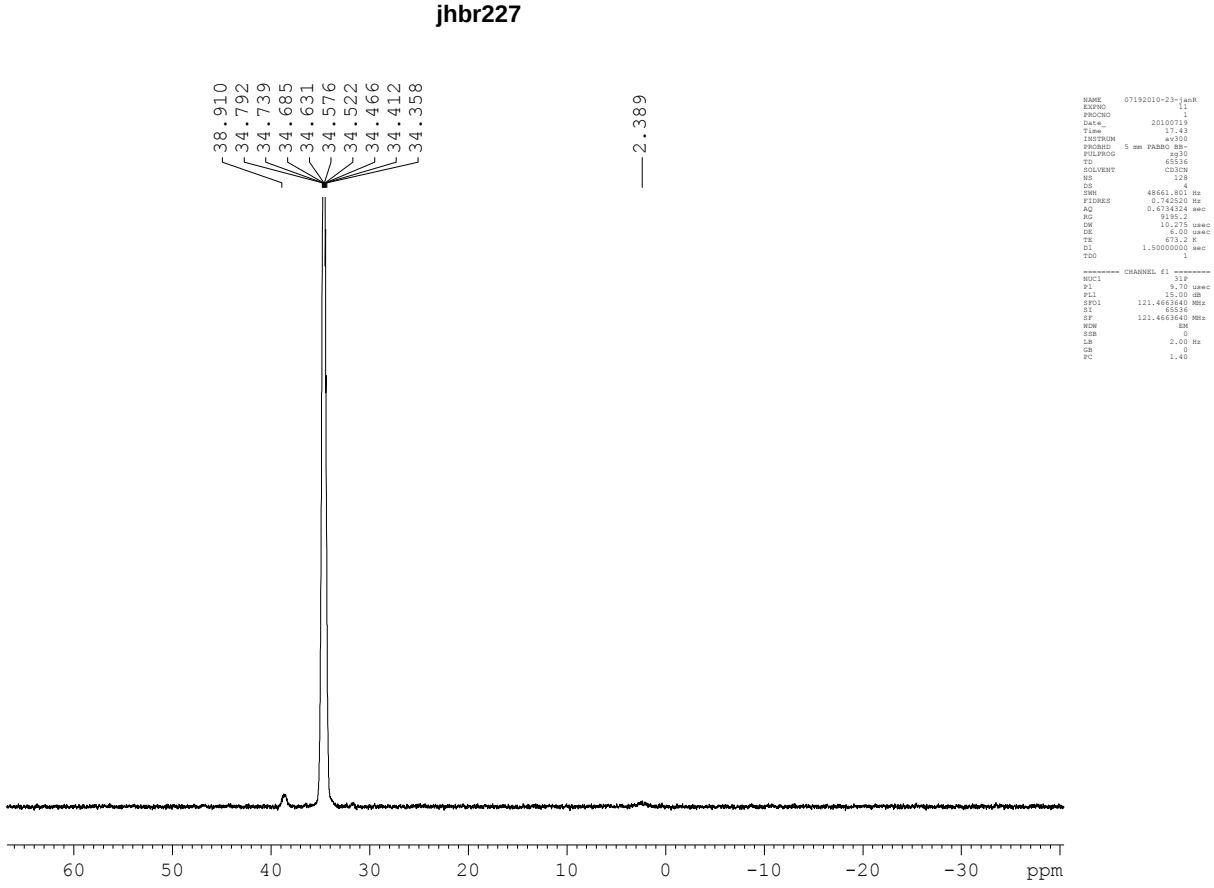
======== CHANNEL f1 ========
NUC1 31P
P1 9.70 usec
PL1 15.00 dB
SFO1 121.4663640 MHz
SI 65536
SF 121.4663640 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.40

The ^{31}P NMR of the liquid product after reaction jhbr222

Jhbr227

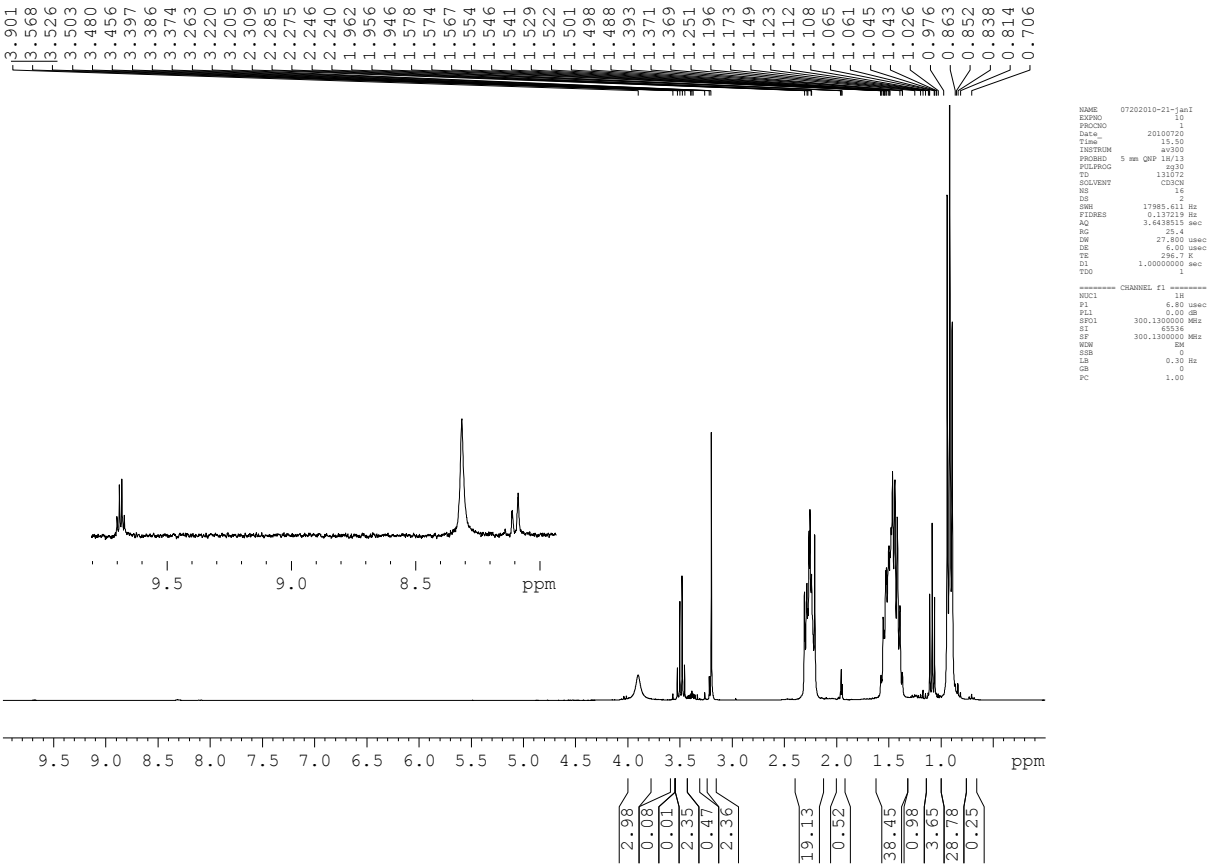


The ^{1}H NMR of the liquid product after reaction jhbr227

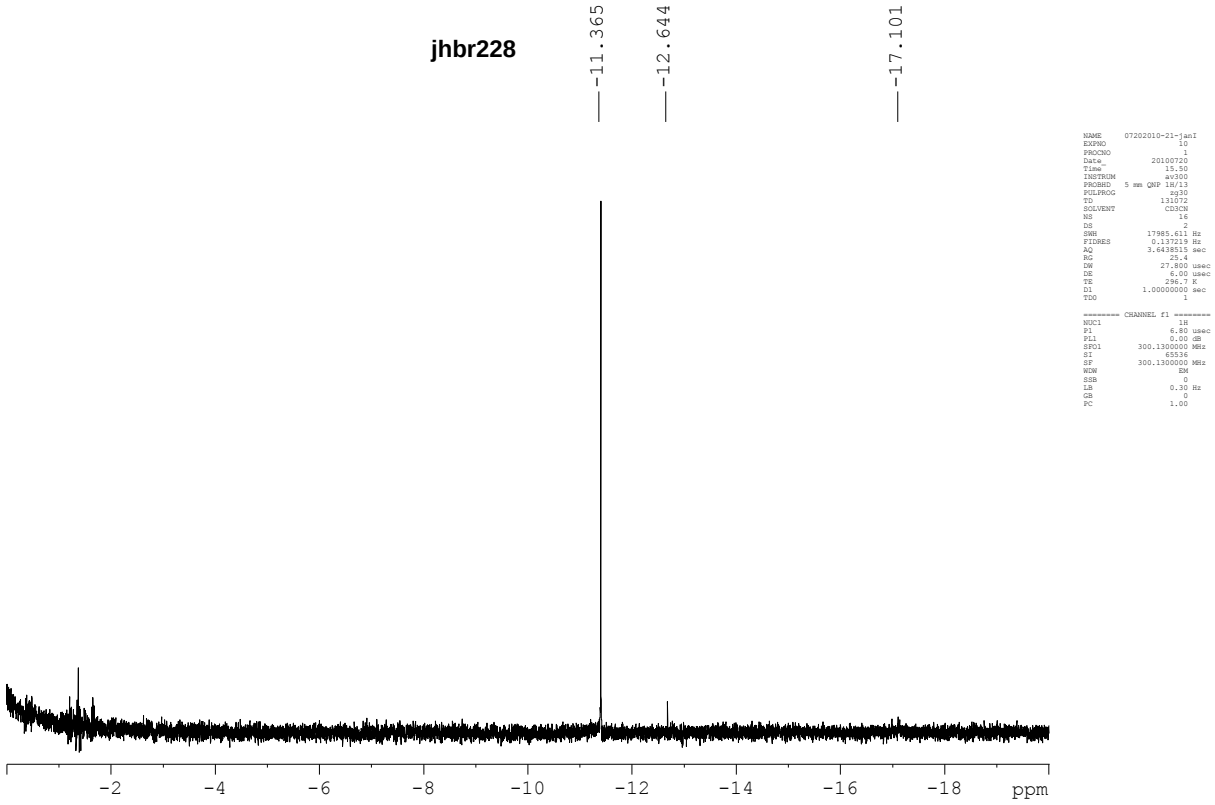


The ^{31}P NMR of the liquid product after reaction jhbr227

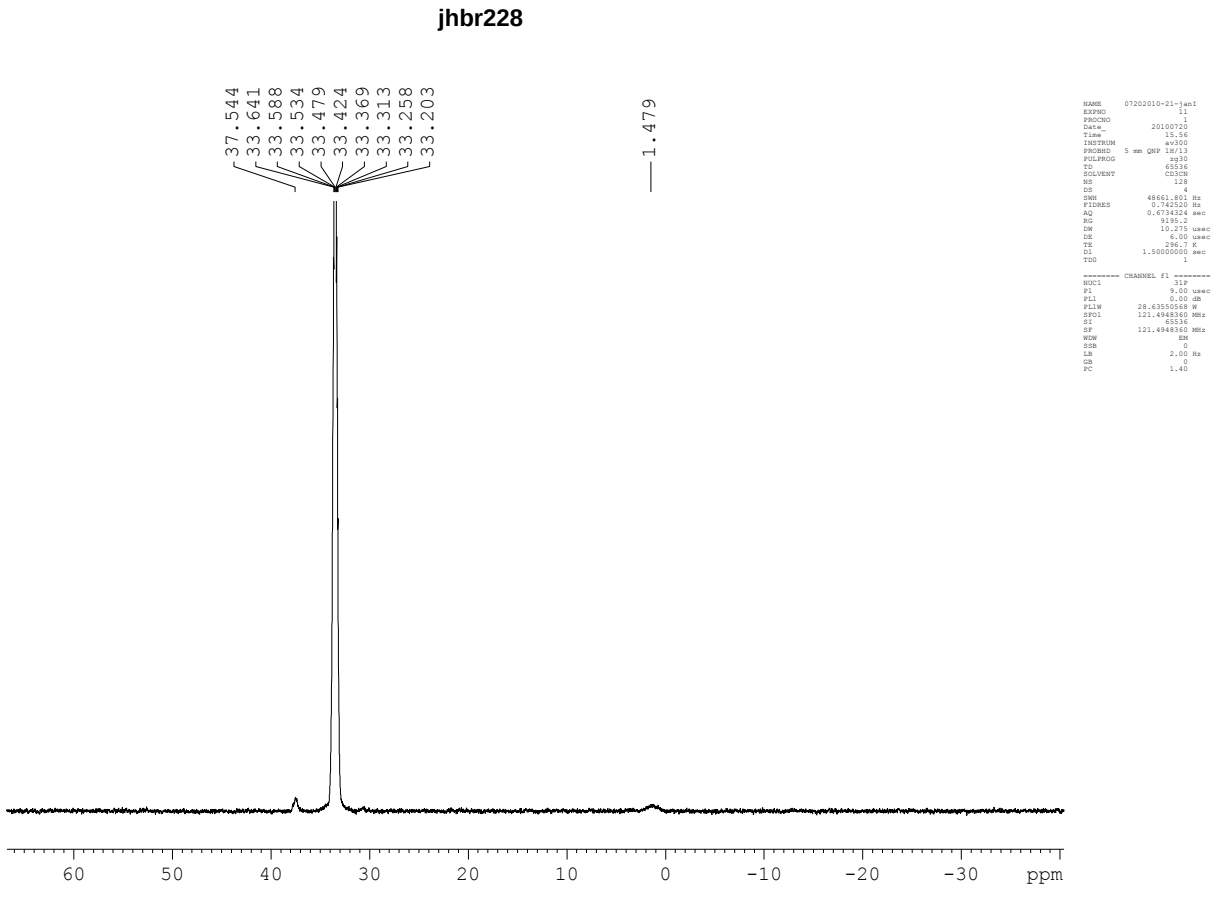
Jhbr228



The ^{1}H NMR of the liquid product after reaction jhbr228

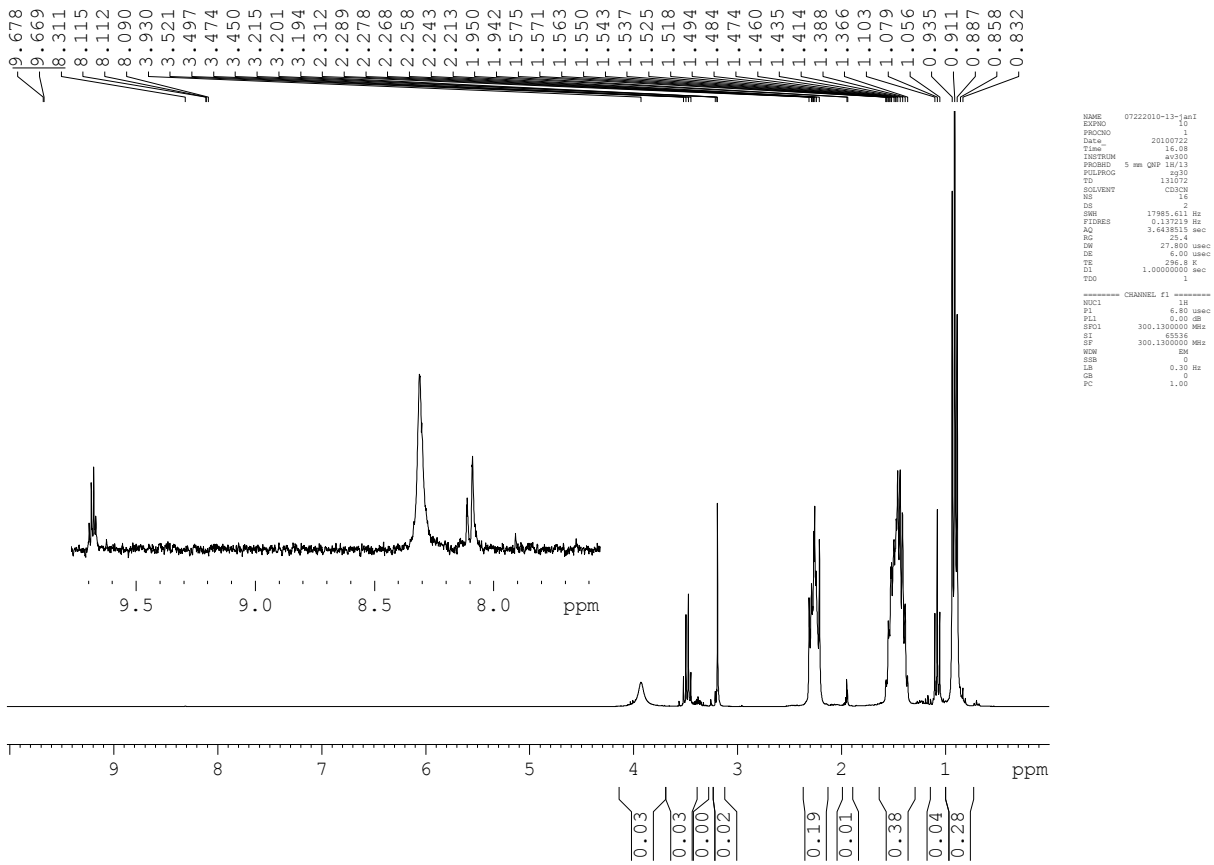


The ^{1}H NMR of the hydride region of the liquid product after reaction jhbr228

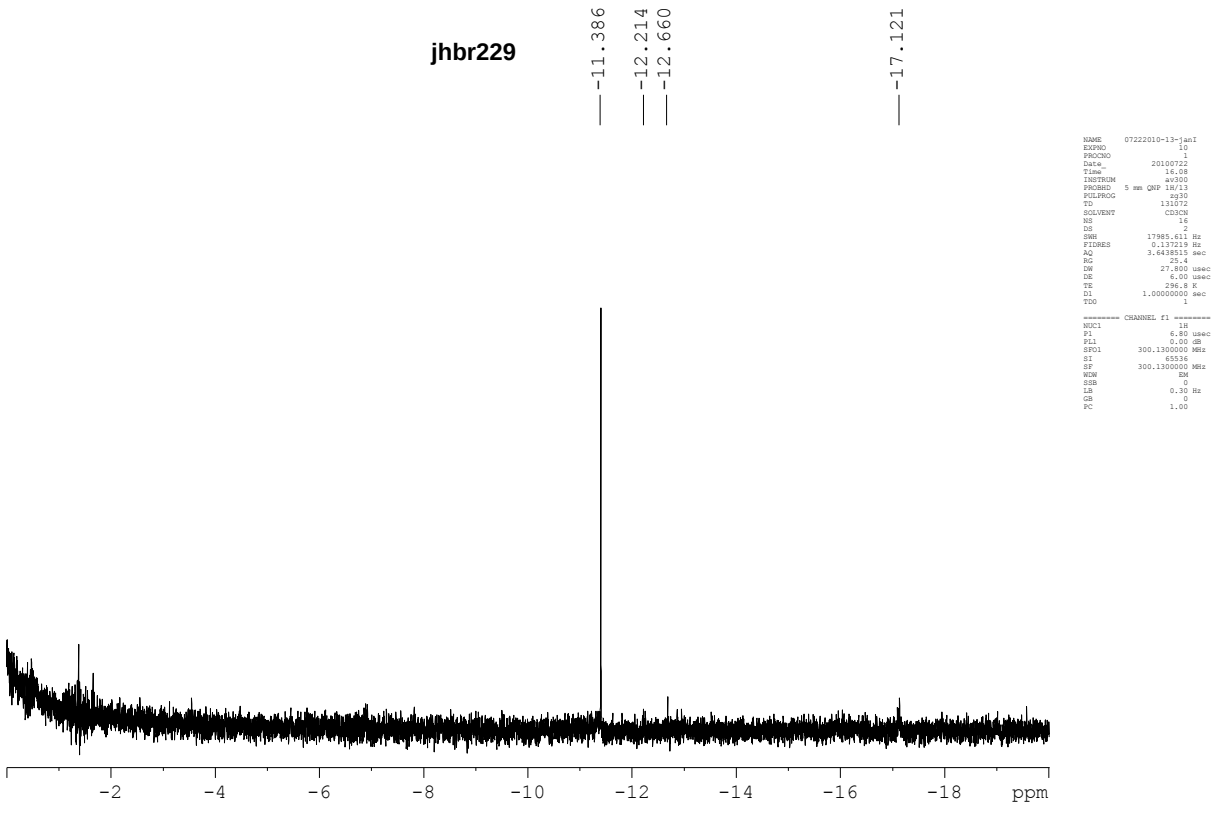


The ^{31}P NMR of the liquid product after reaction jhbr228

Jhbr229

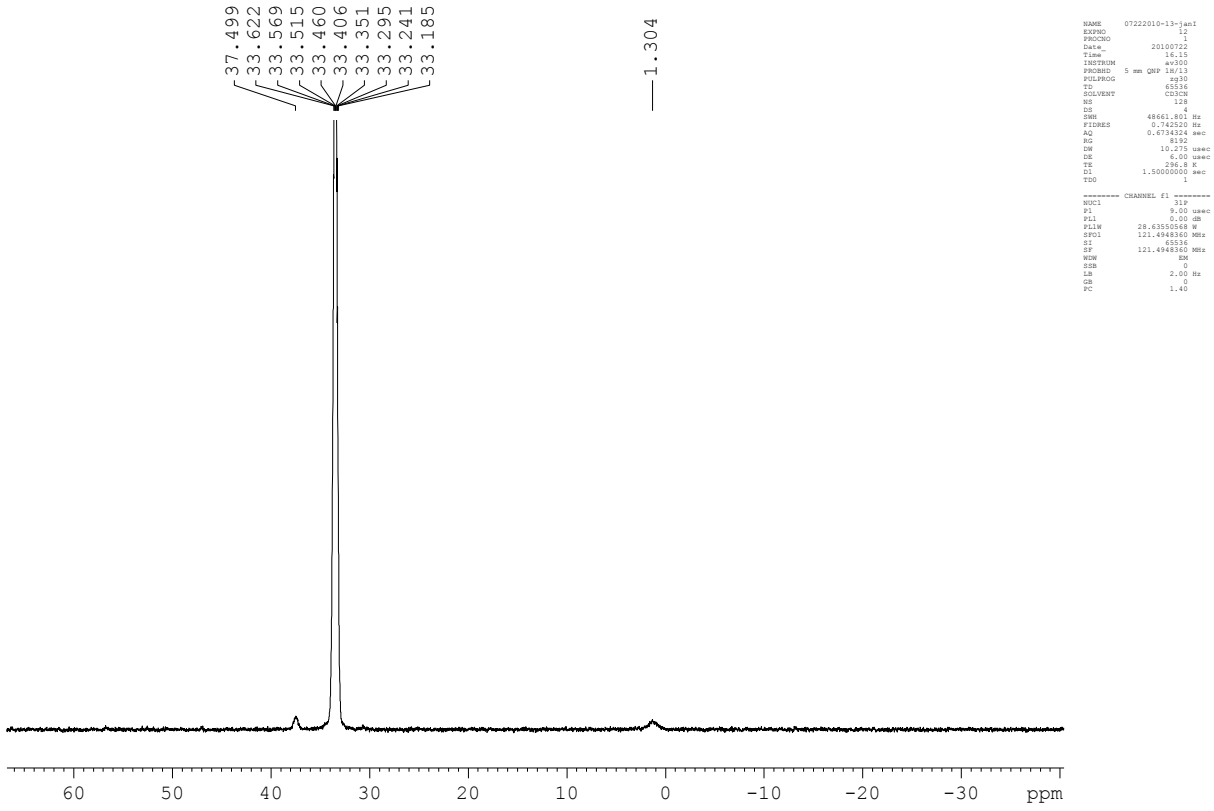


The ^{1}H NMR of the liquid product after reaction jhbr229



The ^{1}H NMR of the hydride region of the liquid product after reaction jhbr229

jhbr229



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PROCNO 1
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Time 16.15
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TD 65536
SOLVENT CDCN3
NS 128
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SWH 48661.801 Hz
FIDRES 0.742520 Hz
AQ 0.6734324 sec
RG 8192
DW 10.275 usec
DE 6.00 usec
TE 296.8 K
D1 1.50000000 sec
TD0 1

======== CHANNEL f1 ========
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P1 9.00 usec
PL1 0.00 dB
PL1W 28.63550568 W
SFO1 121.4948360 MHz
SI 65536
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SSB 0
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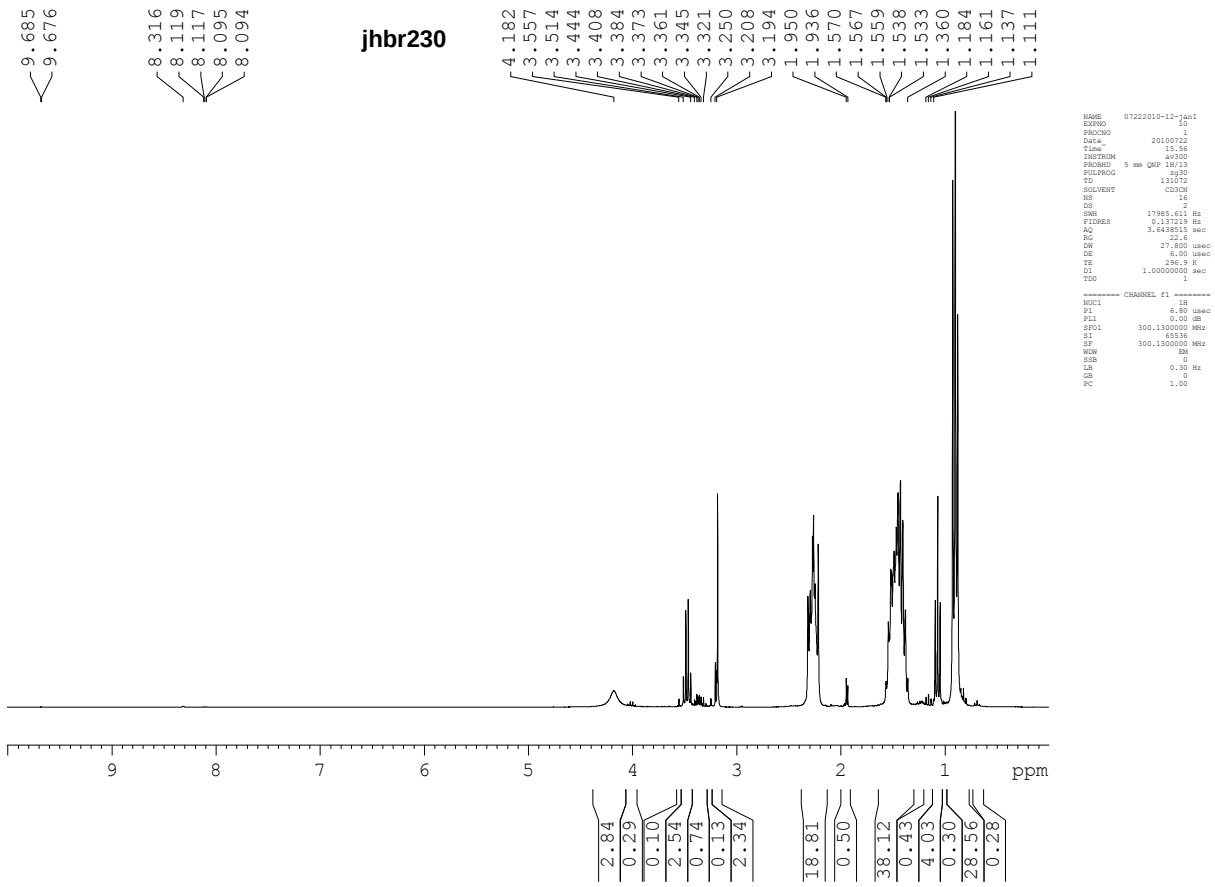
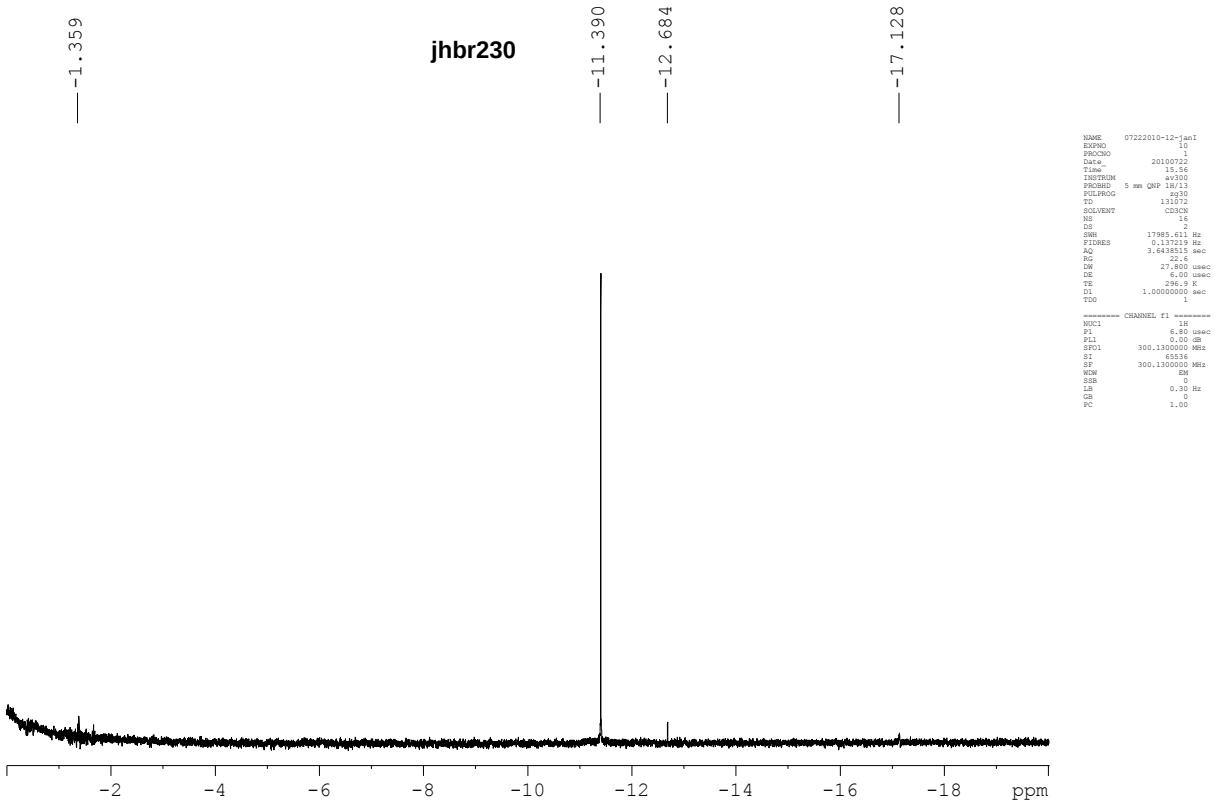
The ^{31}P NMR of the liquid product after reaction jhbr229

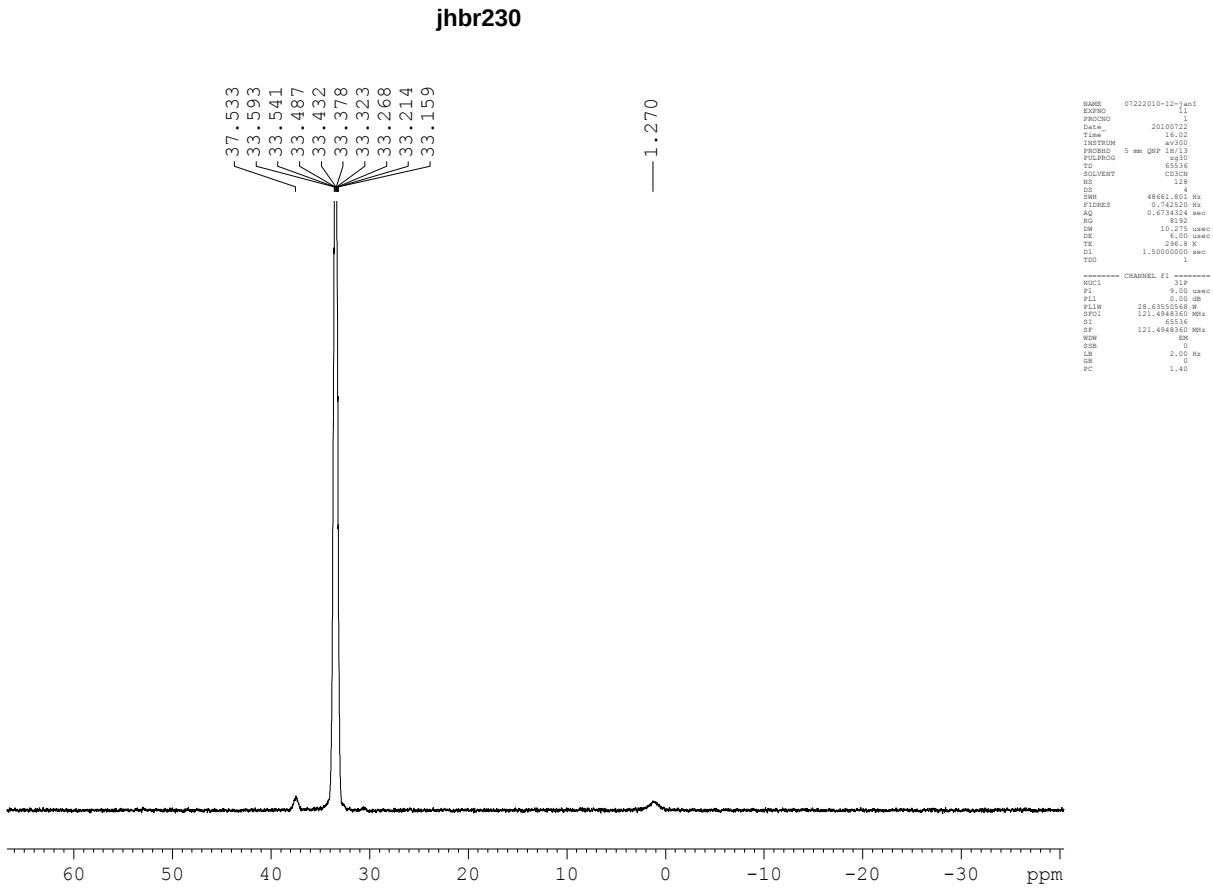
Jhbr230



The ^{1}H NMR of the liquid product after reaction jhbr230

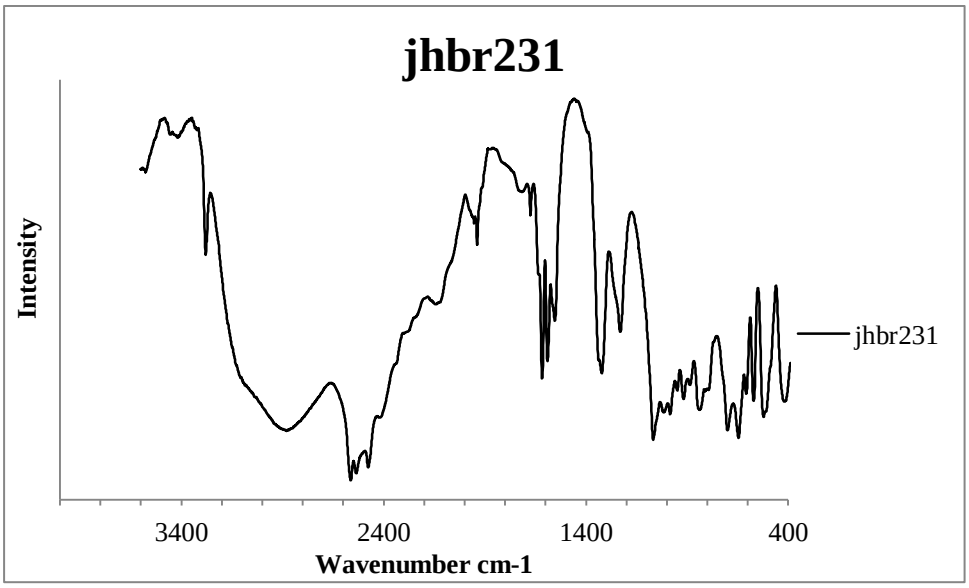


The ^{1}H NMR of the hydride region of the liquid product after reaction jhbr230



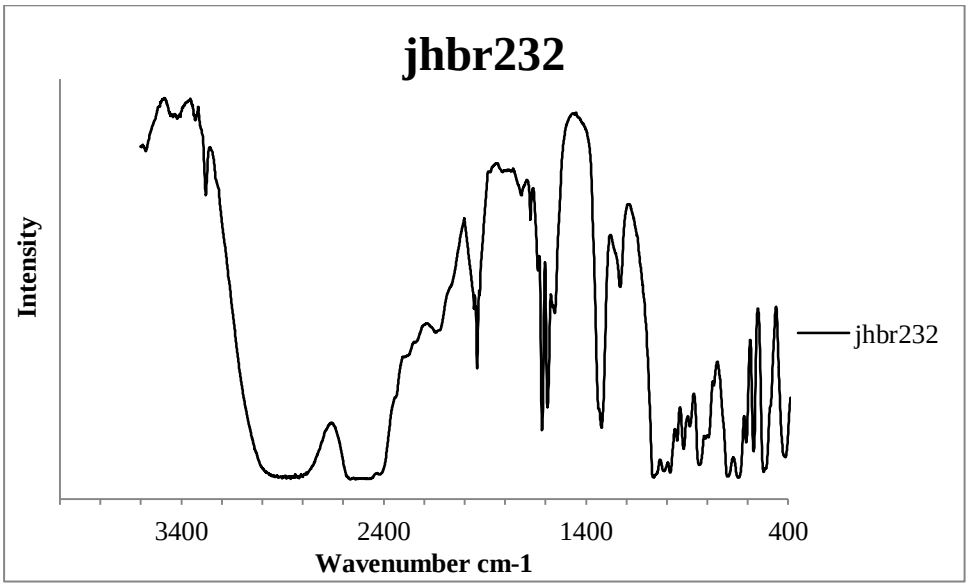
The ^{31}P NMR of the liquid product after reaction jhbr230

jhbr231



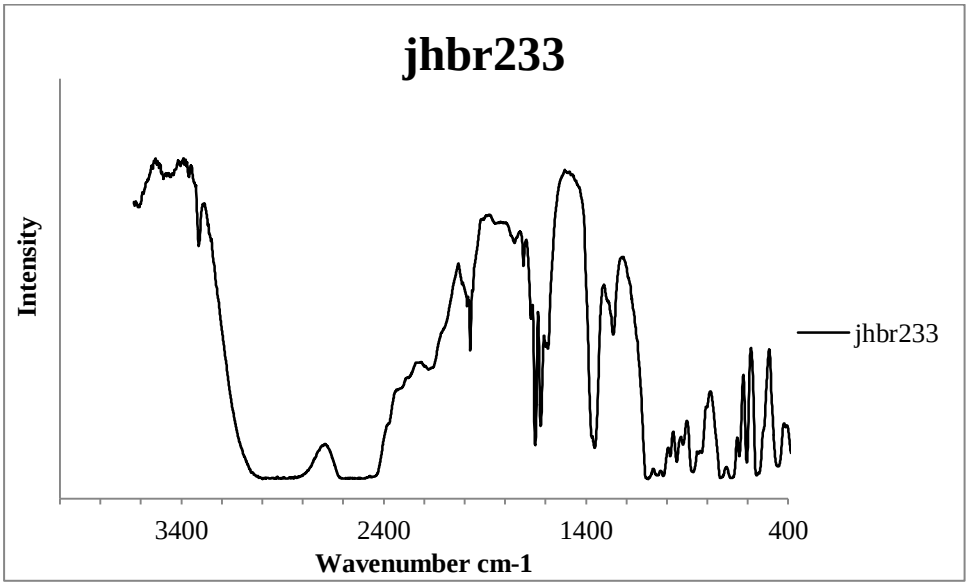
The complete IR spectrum of the liquid product after reaction of reaction jhbr231

jhbr232



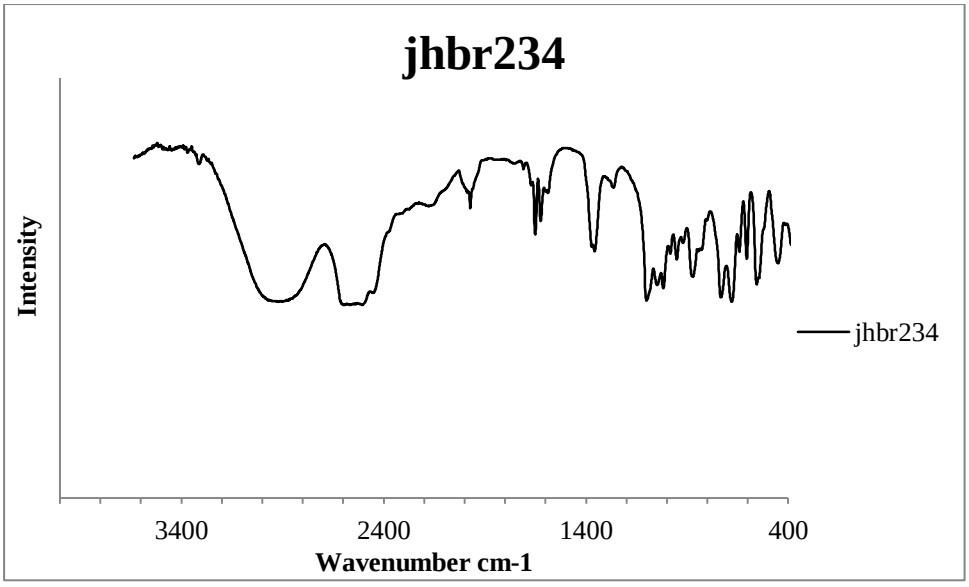
The complete IR spectrum of the liquid product after reaction of reaction jhbr232

jhbr233



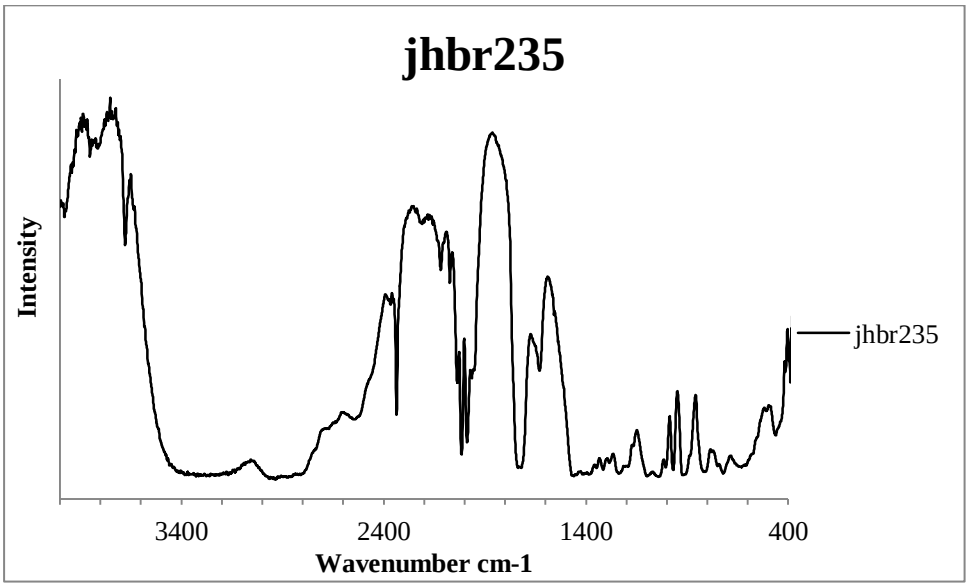
The complete IR spectrum of the liquid product after reaction of reaction jhbr233

jhbr234



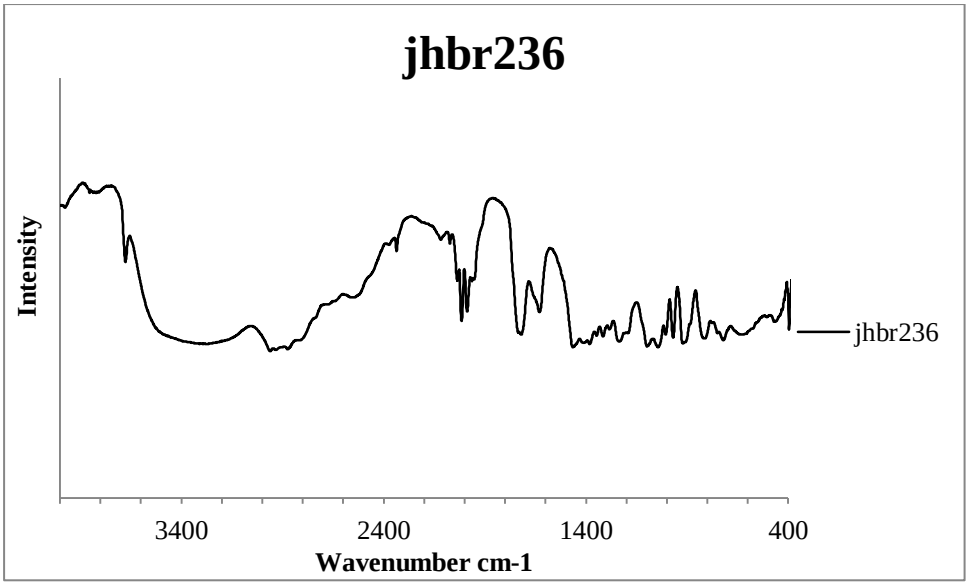
The complete IR spectrum of the liquid product after reaction of reaction jhbr234

jhbr235



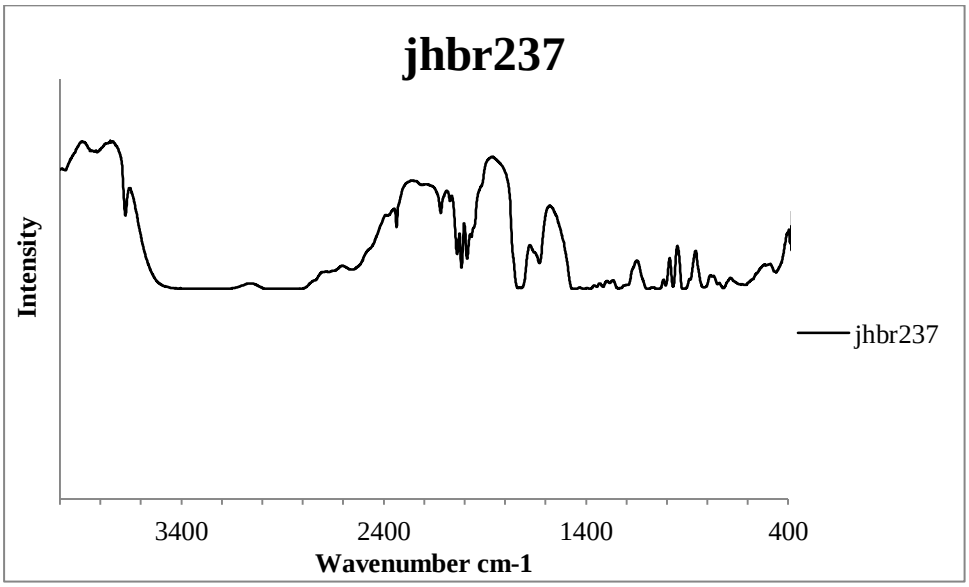
The complete IR spectrum of the liquid product after reaction of reaction jhbr235

jhbr236



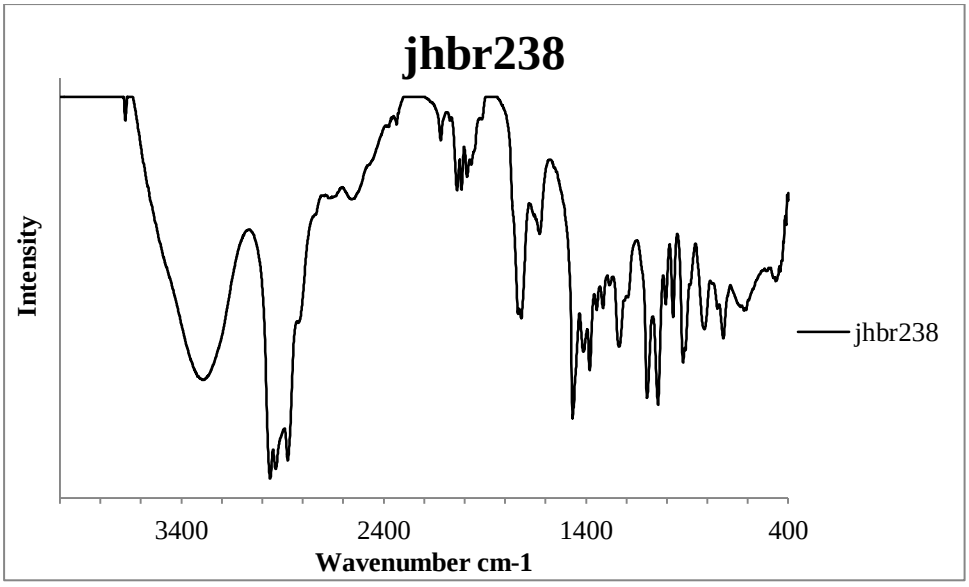
The complete IR spectrum of the liquid product after reaction of reaction jhbr236

jhbr237



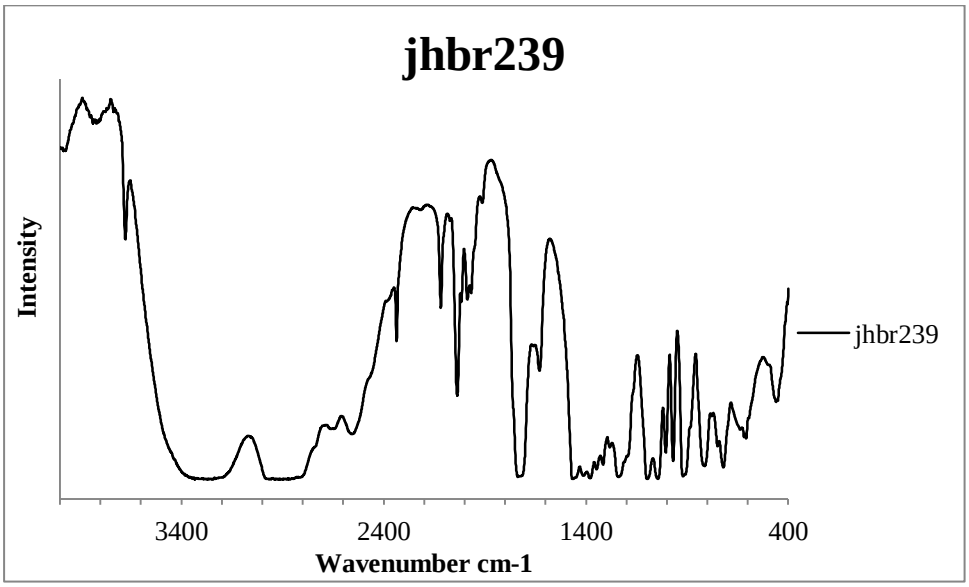
The complete IR spectrum of the liquid product after reaction of reaction jhbr237

jhbr238



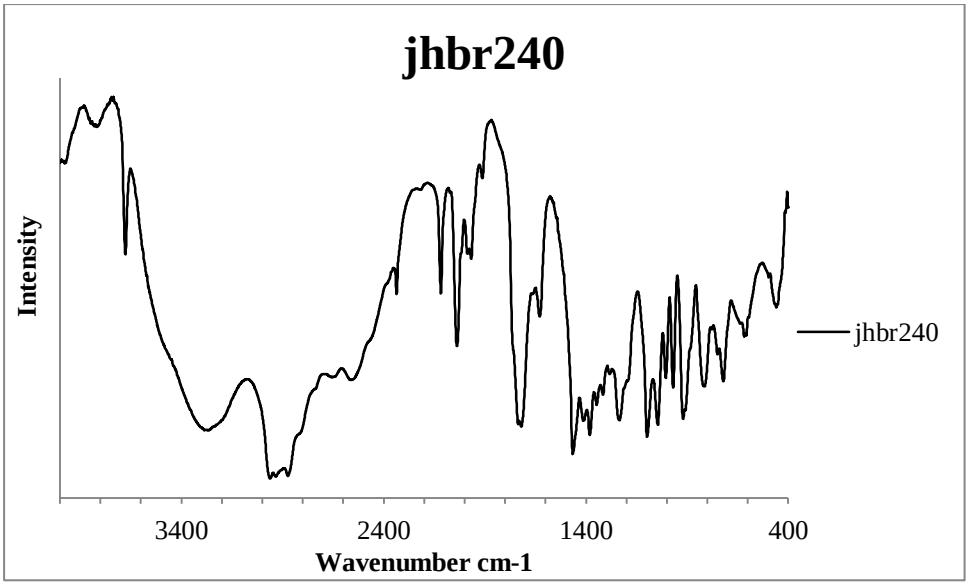
The complete IR spectrum of the liquid product after reaction of reaction jhbr238

jhbr239



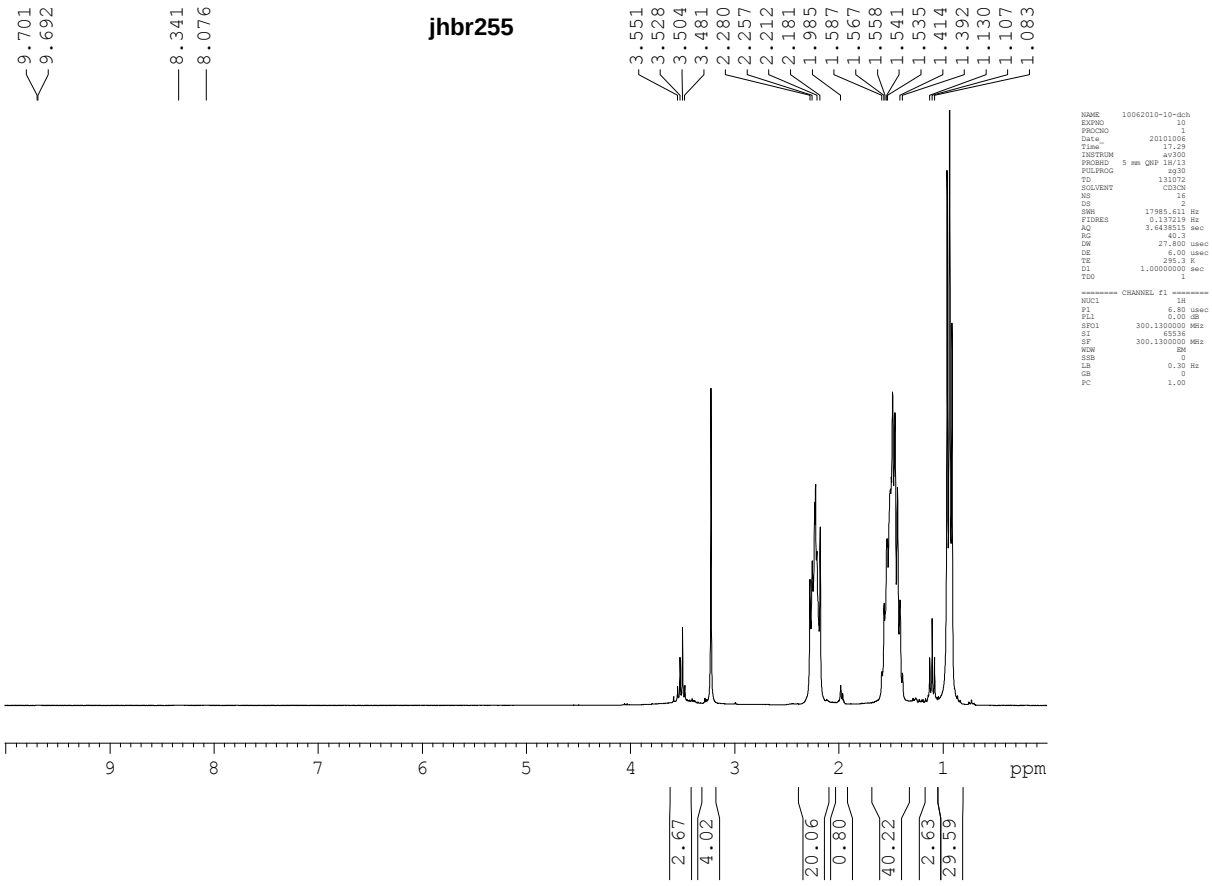
The complete IR spectrum of the liquid product after reaction of reaction jhbr239

jhbr240

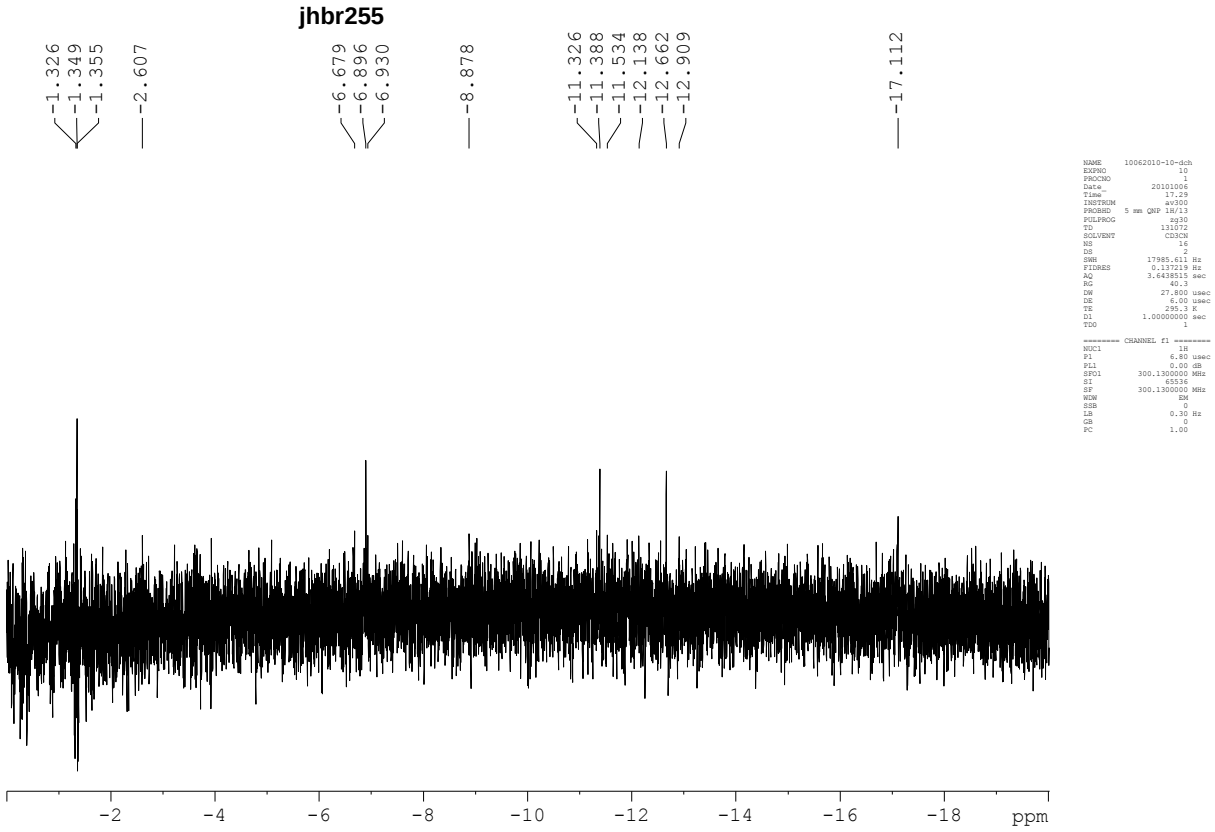


The complete IR spectrum of the liquid product after reaction of reaction jhbr240

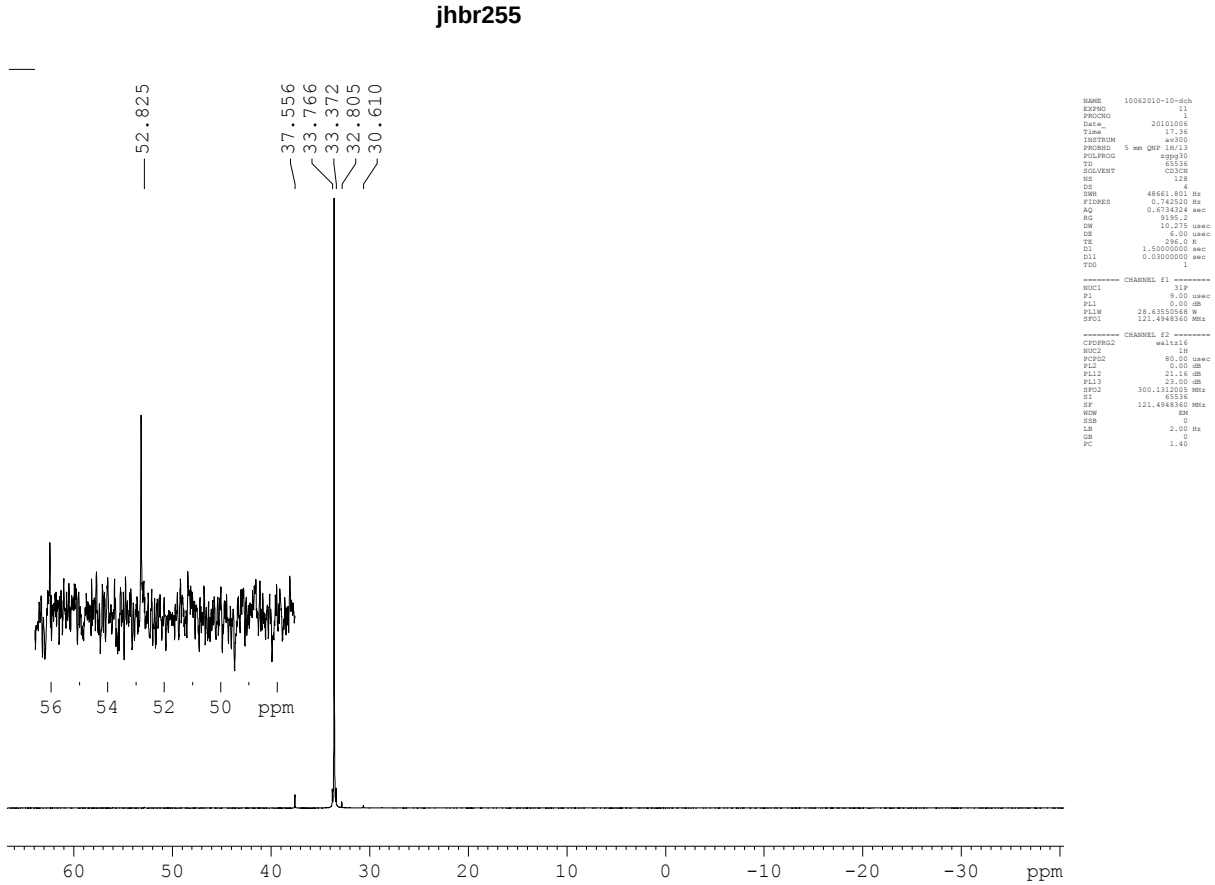
jhbr255



The ^{1}H NMR of the liquid product after reaction jhbr255

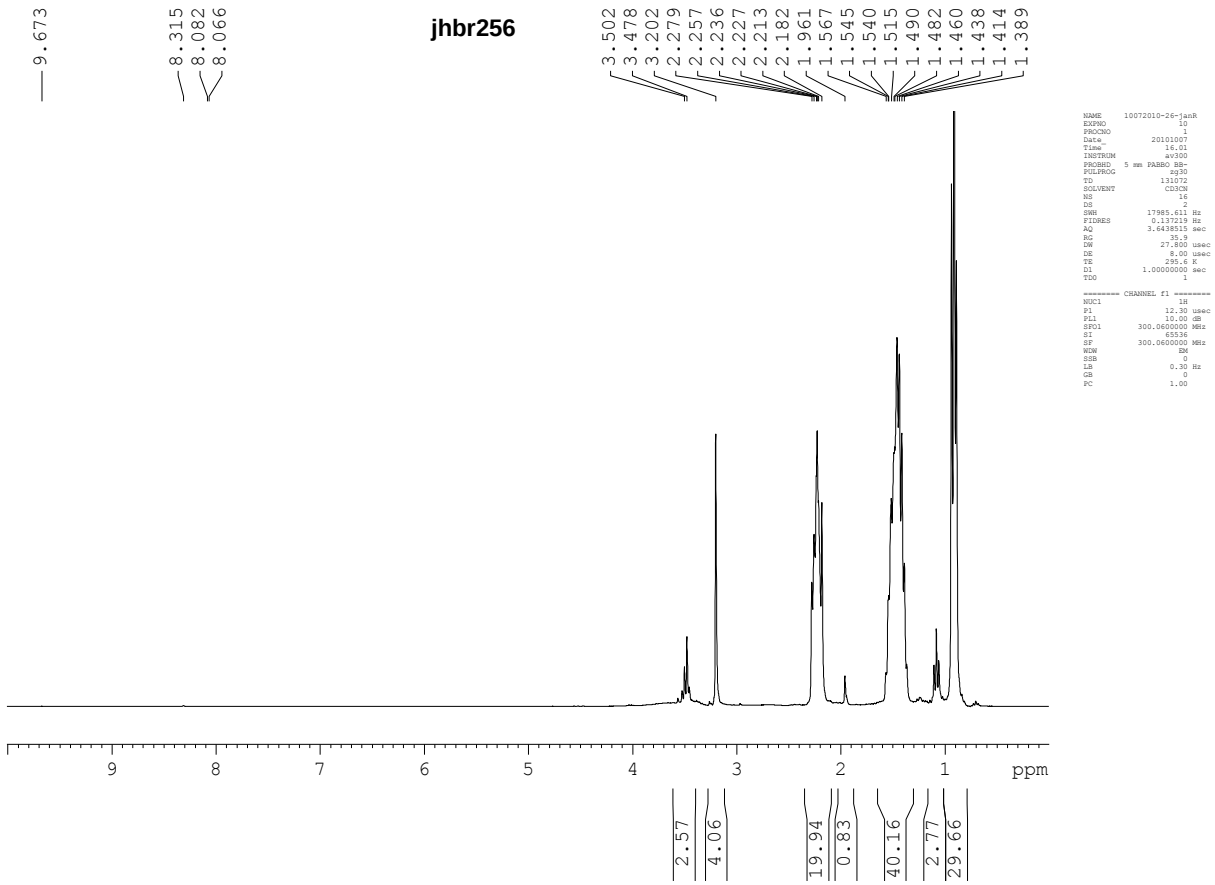


The ^{1}H NMR of the hydride region of the liquid product after reaction jhbr255

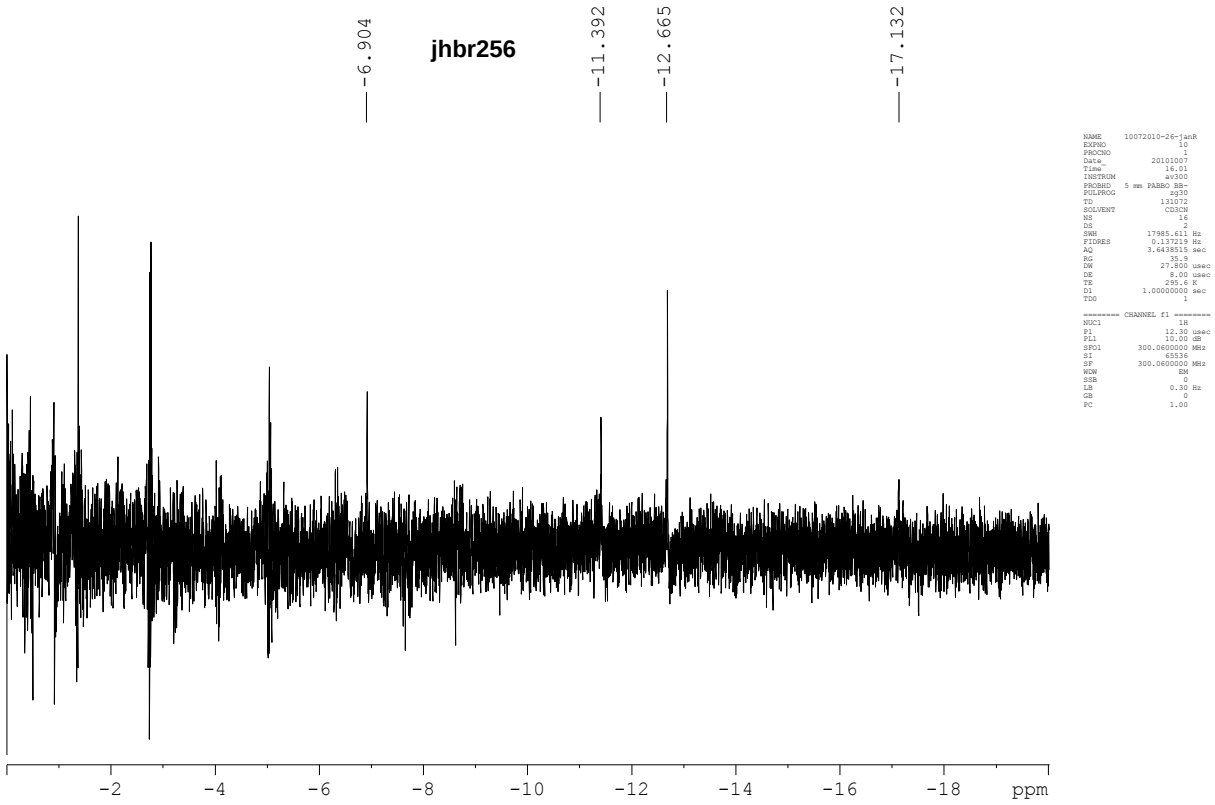


The ^{31}P NMR of the liquid product after reaction jhbr255

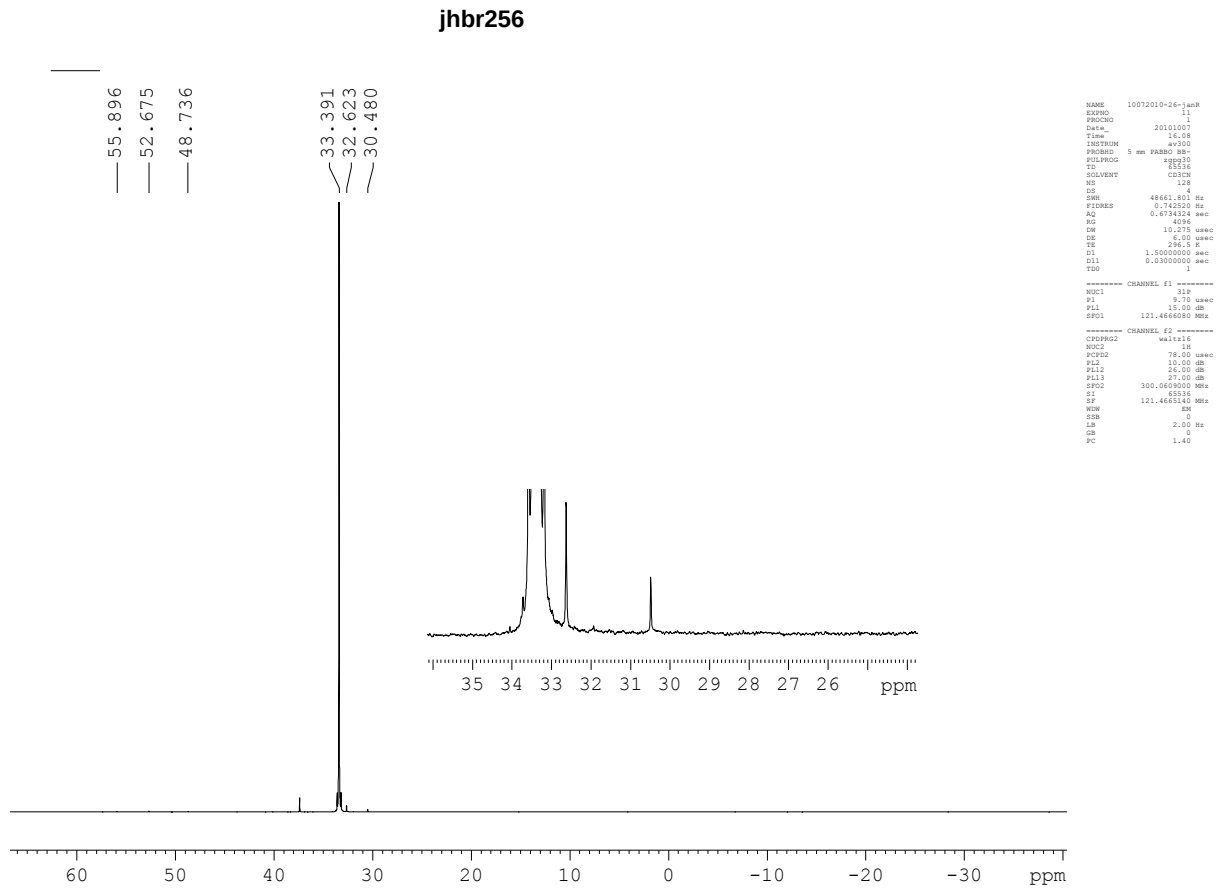
jhbr256



The ^{1}H NMR of the liquid product after reaction jhbr256



The ^{1}H NMR of the hydride region of the liquid product after reaction jhbr256



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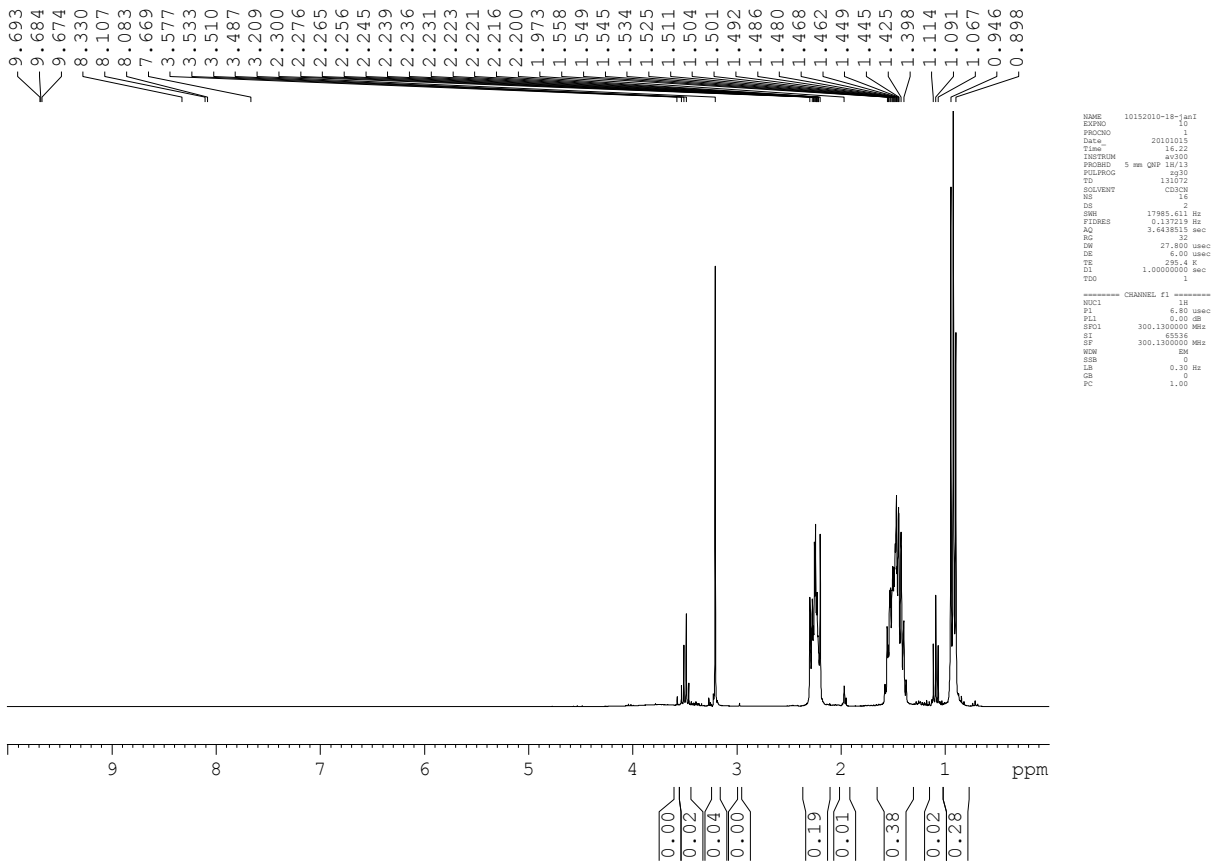
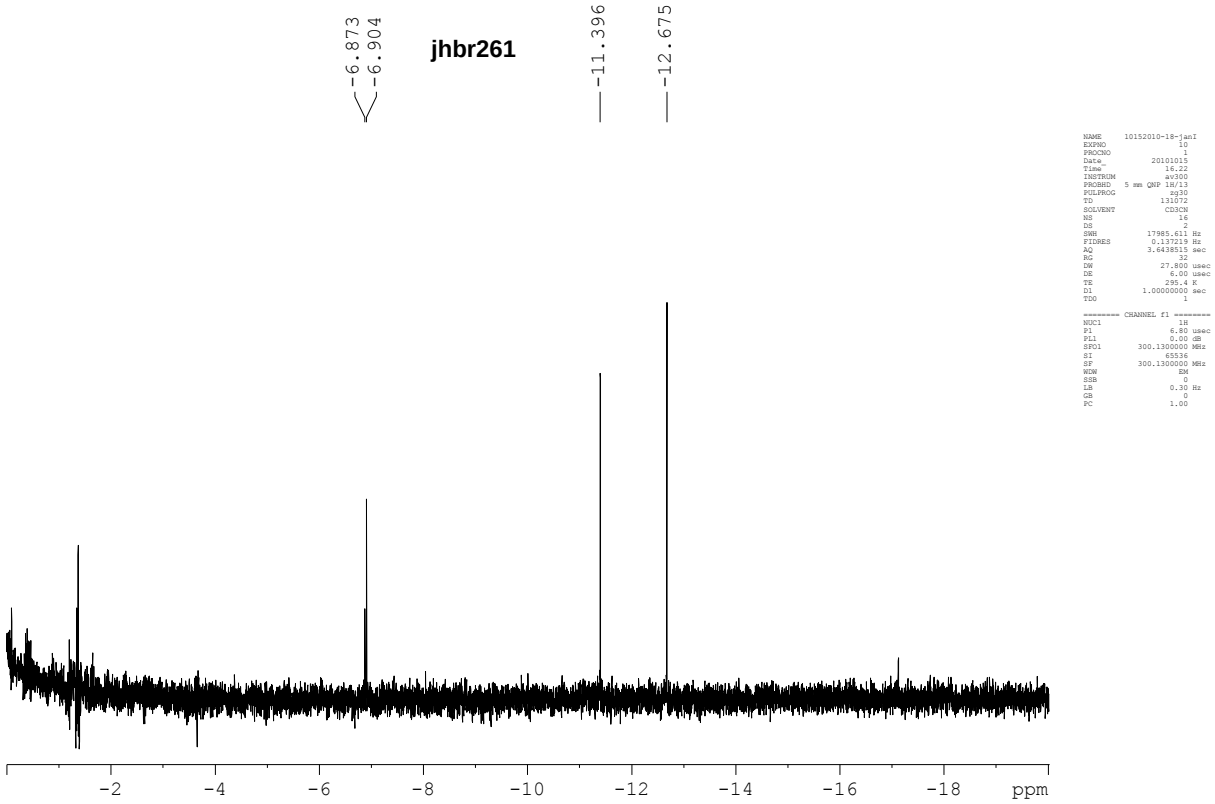
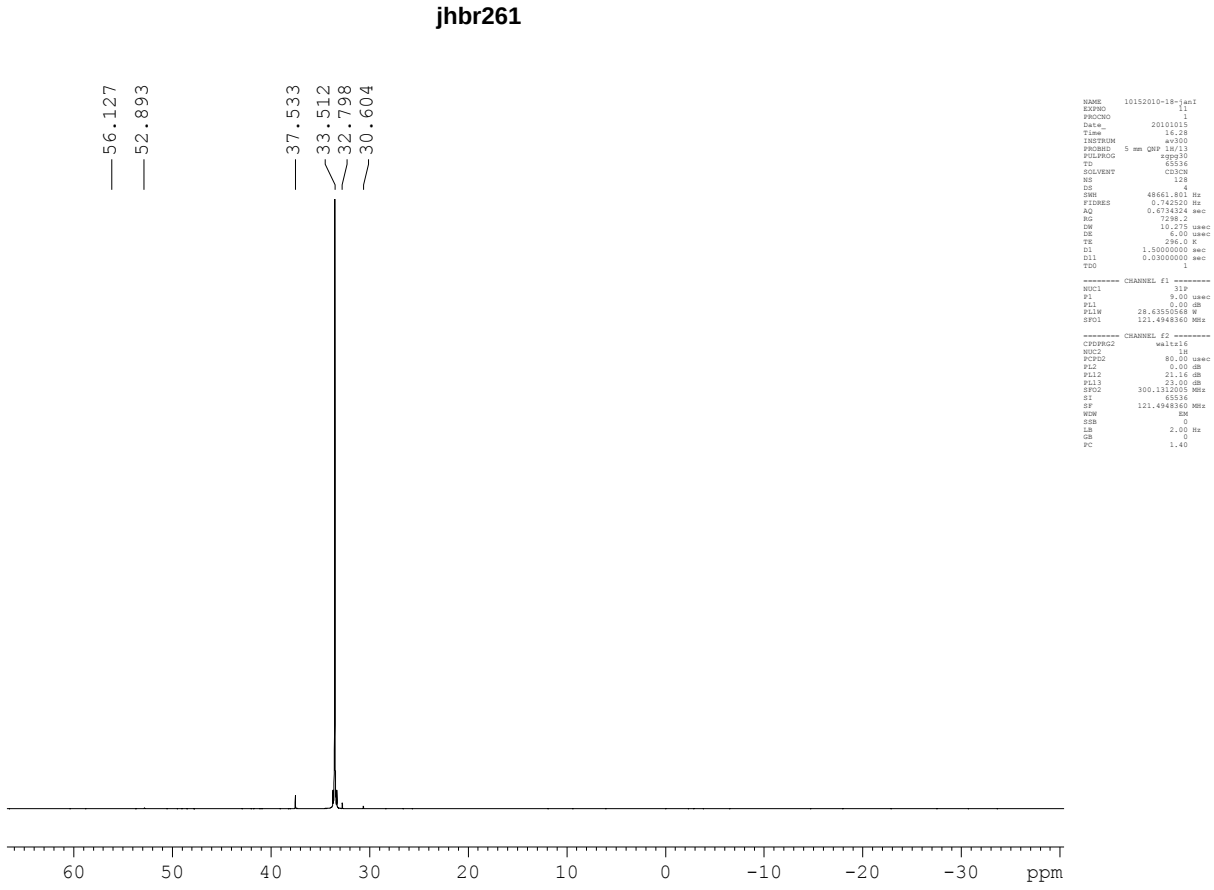


The ^{1}H NMR of the liquid product after reaction jhbr261

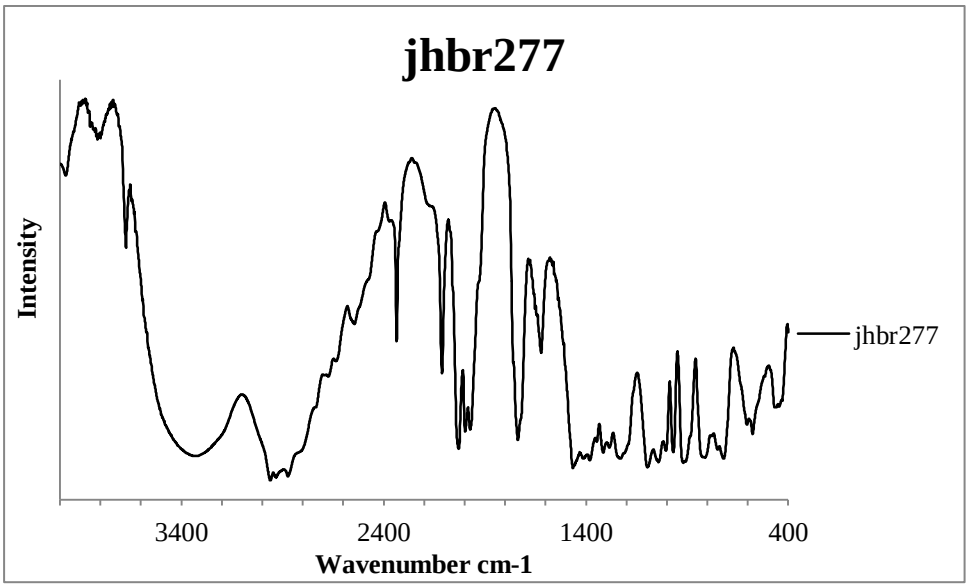


The ^{1}H NMR of the hydride region of the liquid product after reaction jhbr261



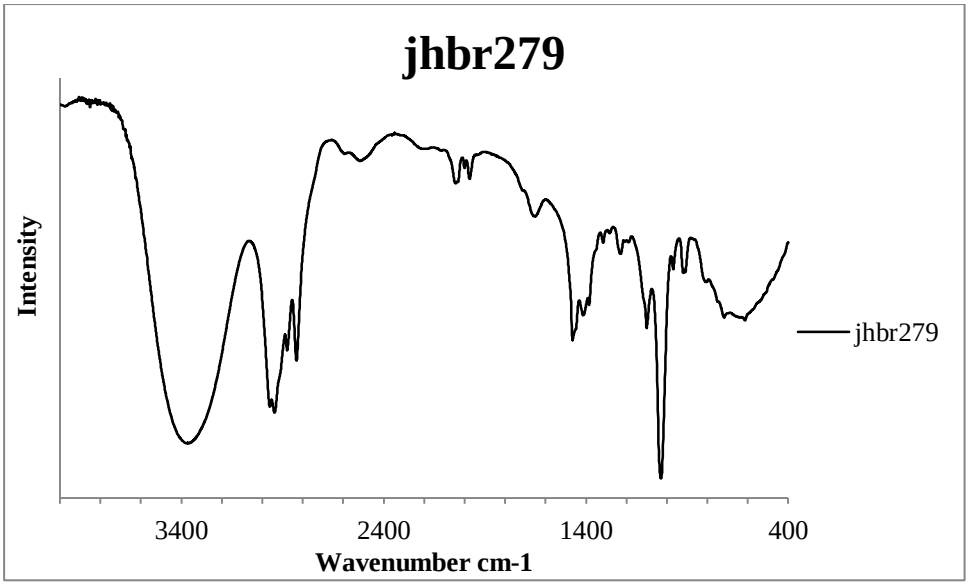
The ^{31}P NMR of the liquid product after reaction jhbr261

jhbr277



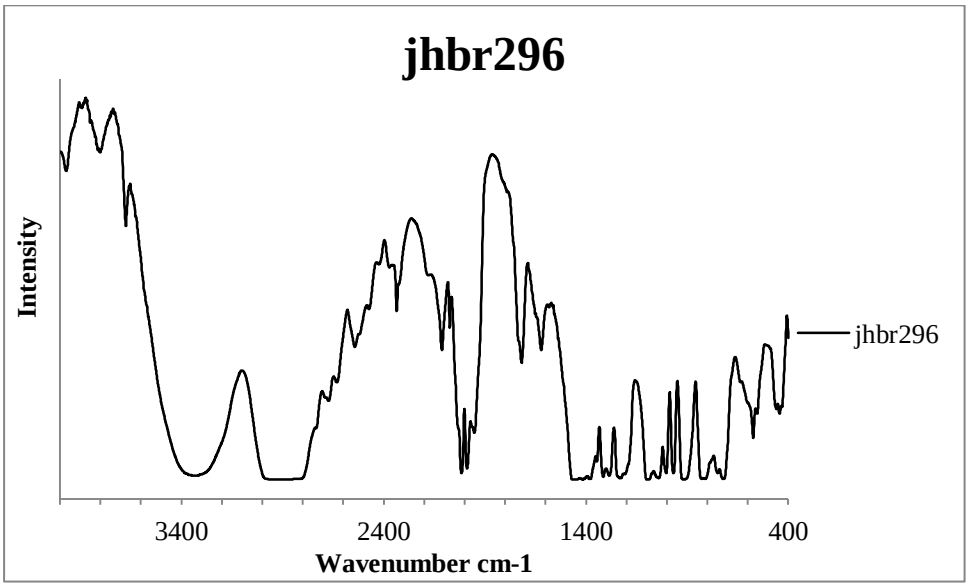
The complete IR spectrum of the liquid product after reaction of reaction jhbr277

jhbr279



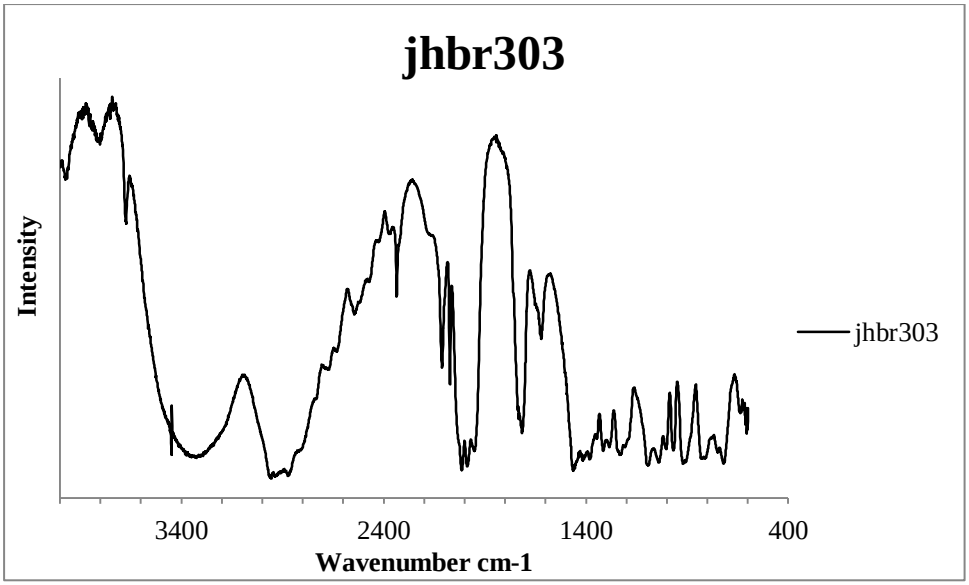
The complete IR spectrum of the liquid product after reaction of reaction jhbr279

jhbr296

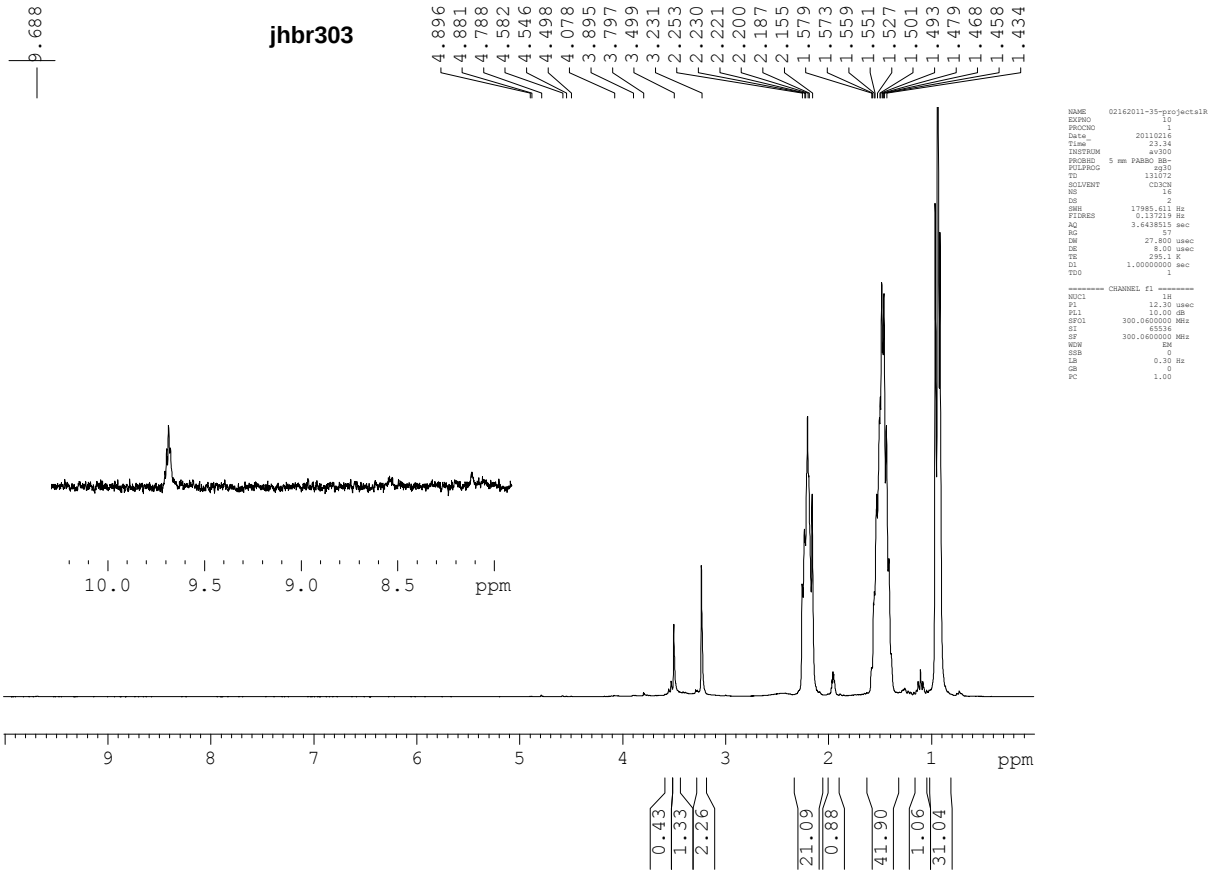


The complete IR spectrum of the liquid product after reaction of reaction jhbr296

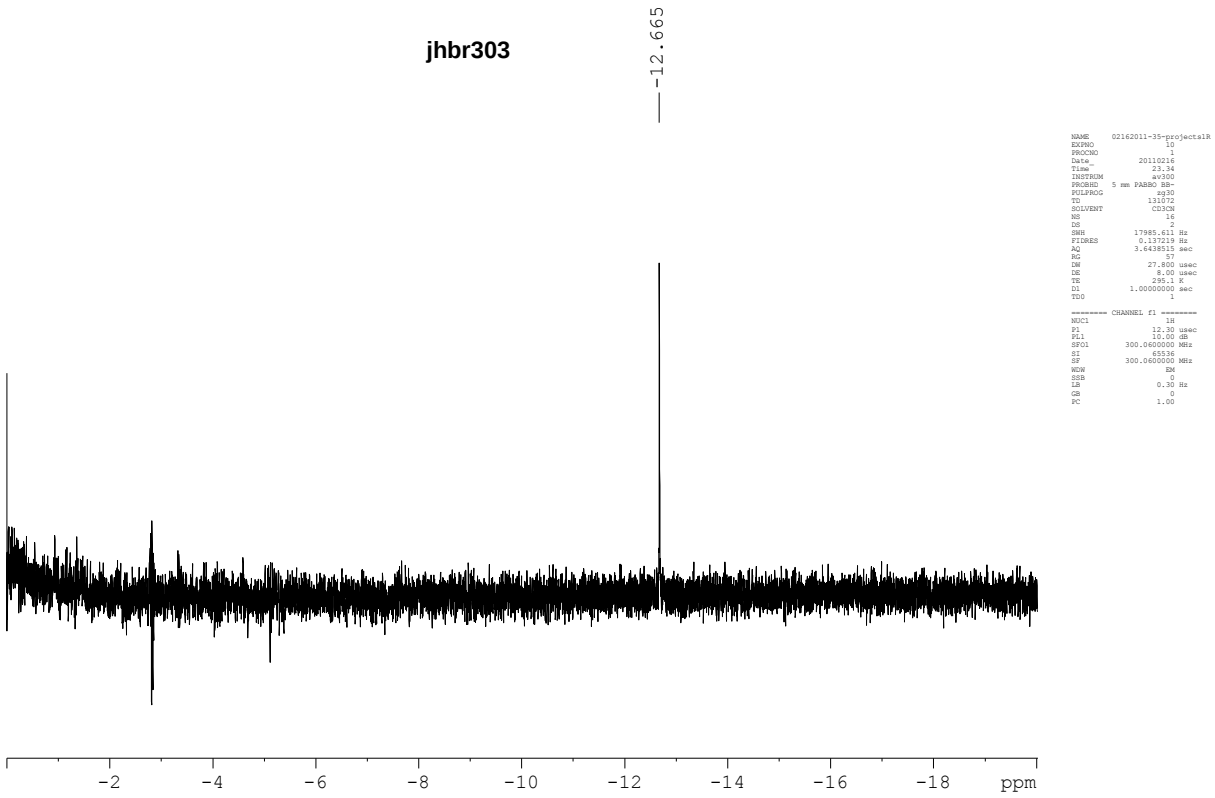
jhbr303



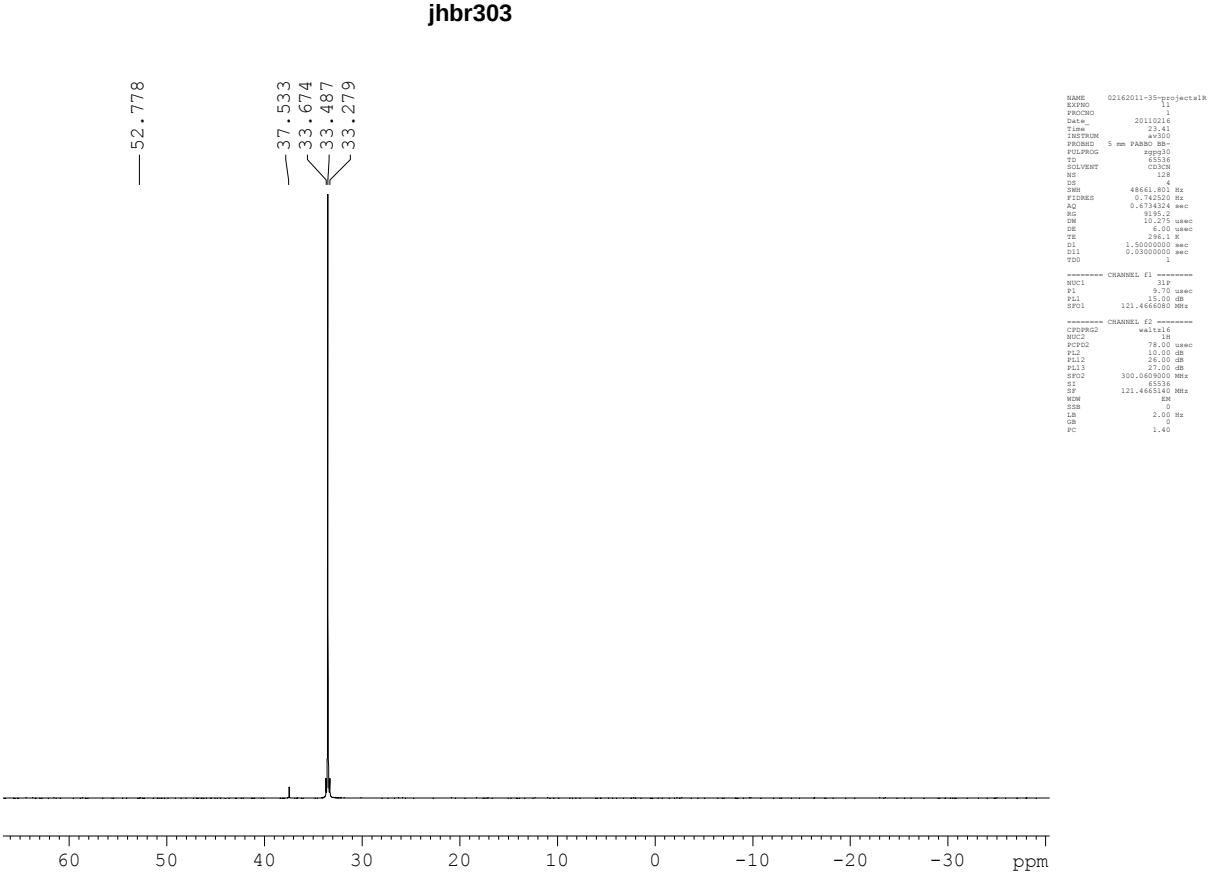
The complete IR spectrum of the liquid product after reaction of reaction jhbr303



The ^{1}H NMR of the liquid product after reaction jhbr303

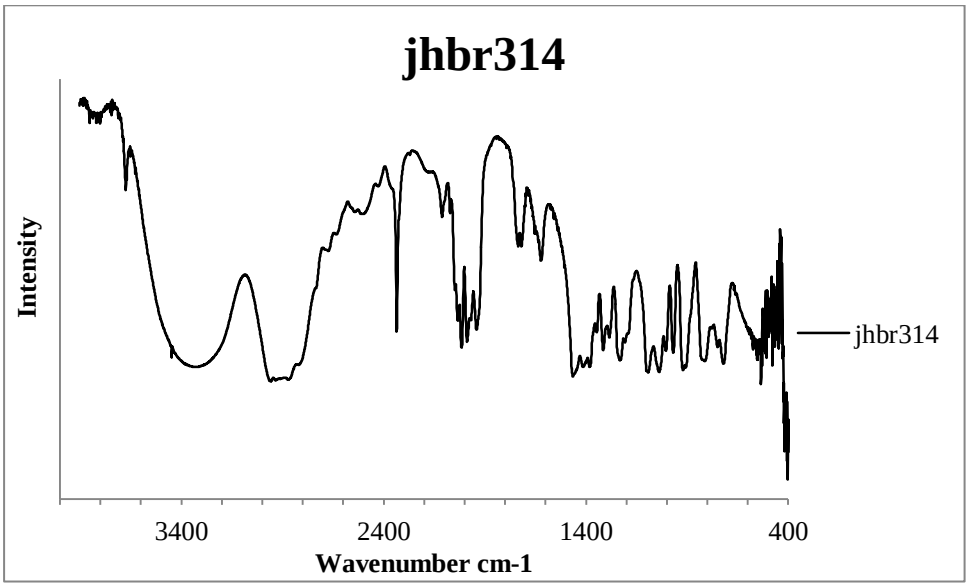


The ^{1}H NMR of the hydride region of the liquid product after reaction jhbr303



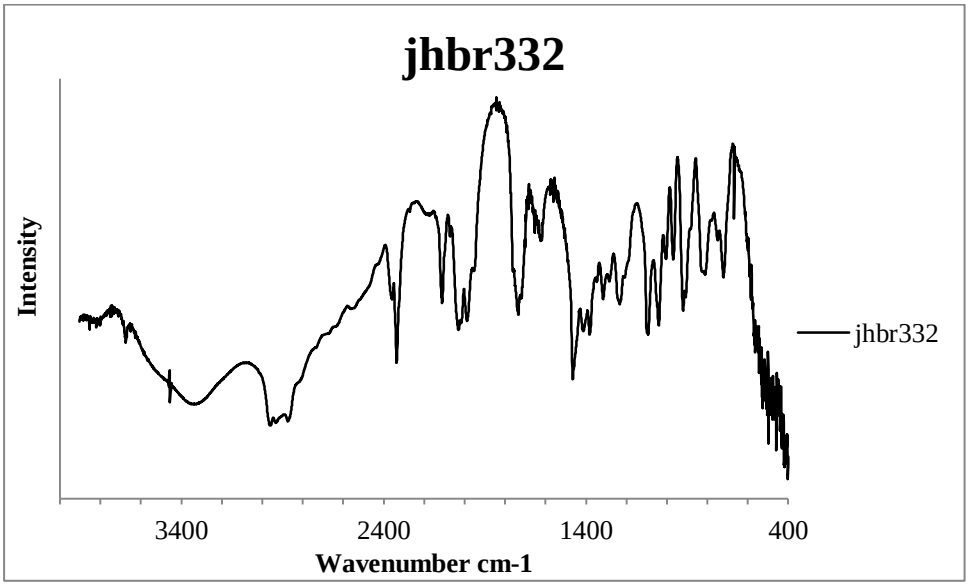
The ^{31}P NMR of the liquid product after reaction jhbr303

jhbr314



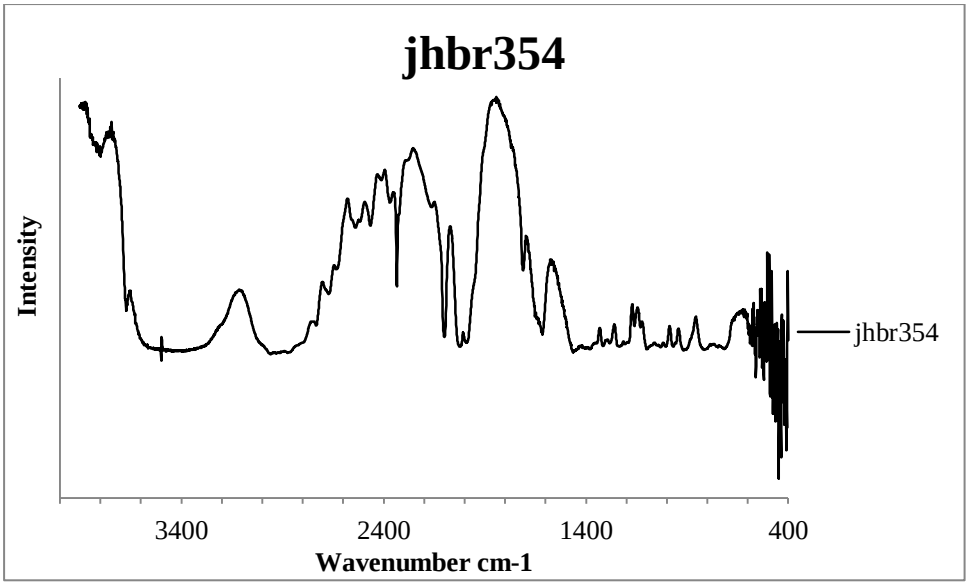
The complete IR spectrum of the liquid product after reaction of reaction jhbr314

jhbr332

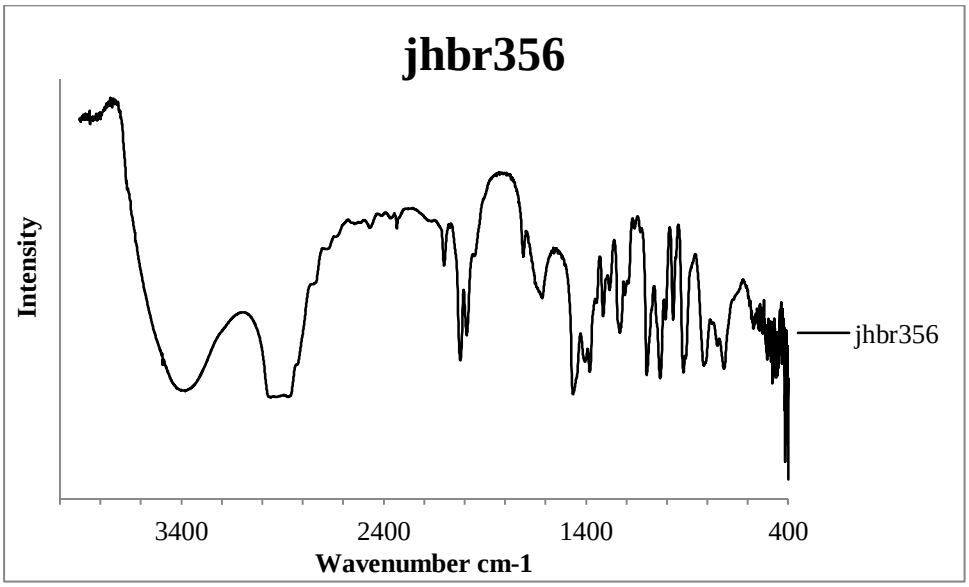


The complete IR spectrum of the liquid product after reaction of reaction jhbr332

jhbr354

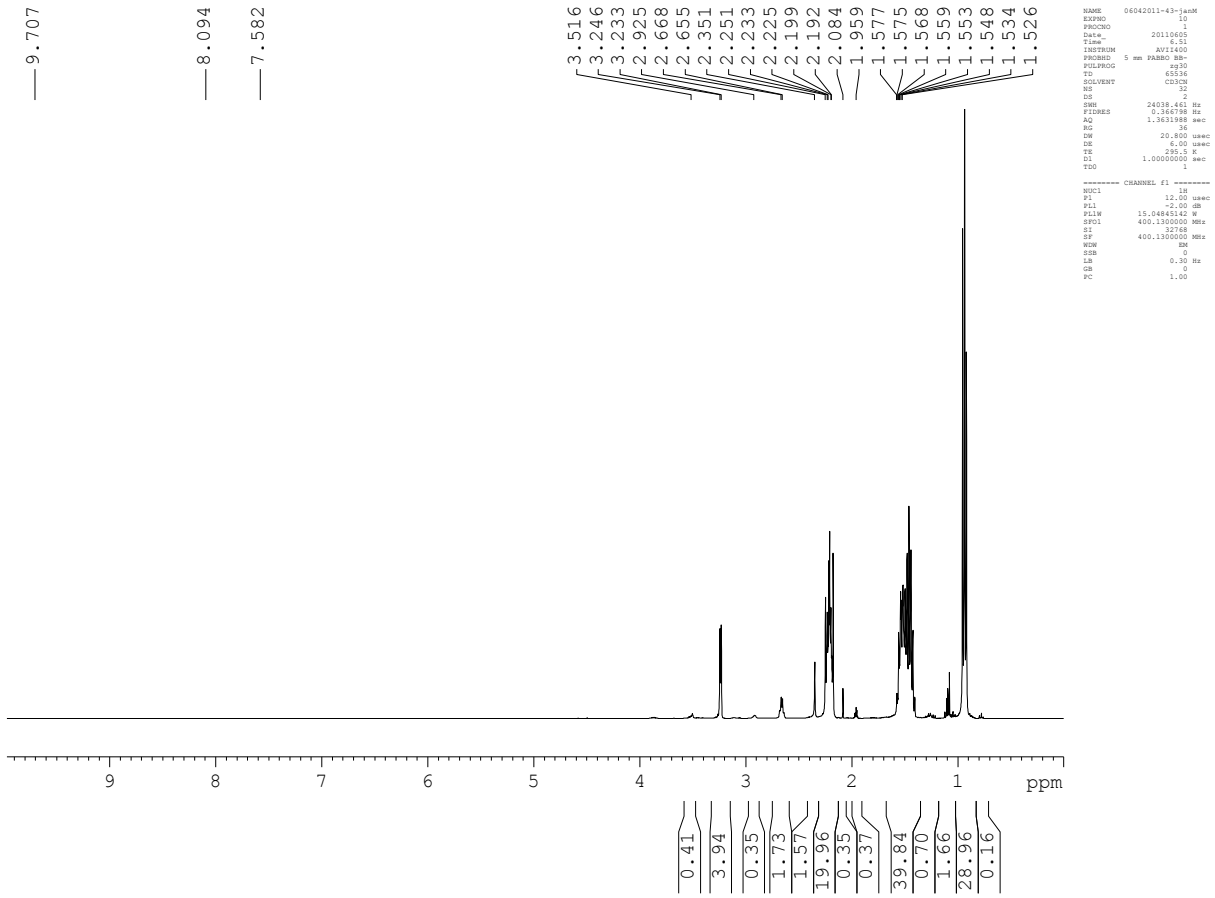


The complete IR spectrum of the liquid product after reaction of reaction jhbr354 jhbr356

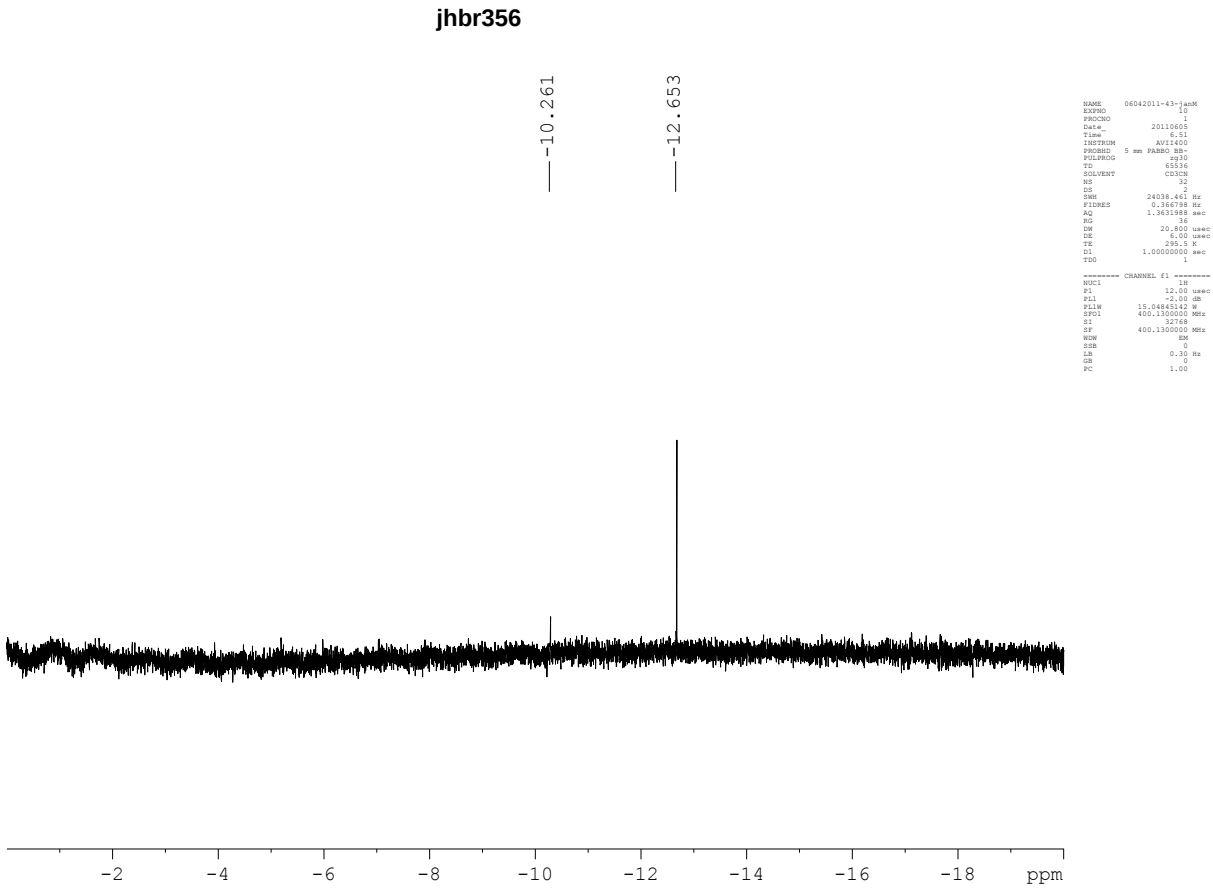


The complete IR spectrum of the liquid product after reaction of reaction jhbr356

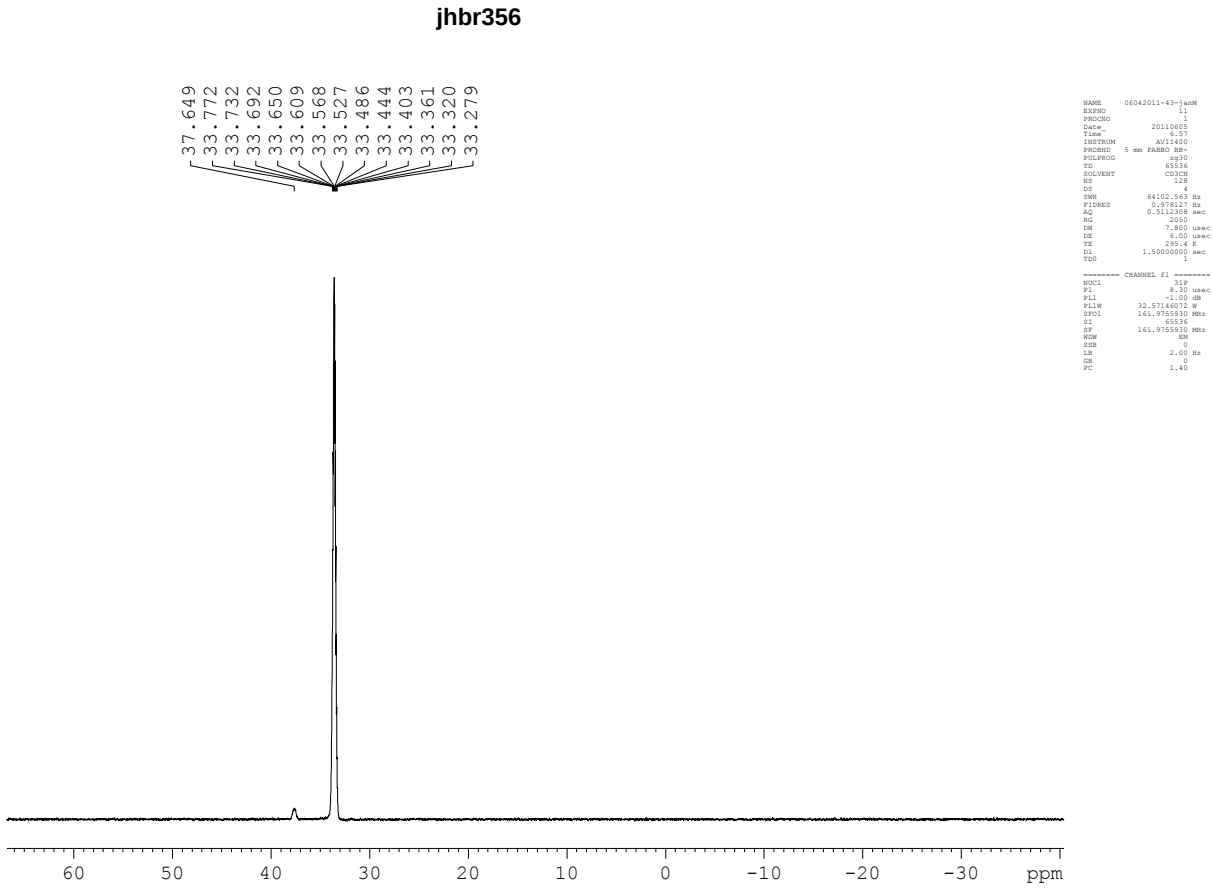
jhbr356



The ^{1}H NMR of the liquid product after reaction jhbr356

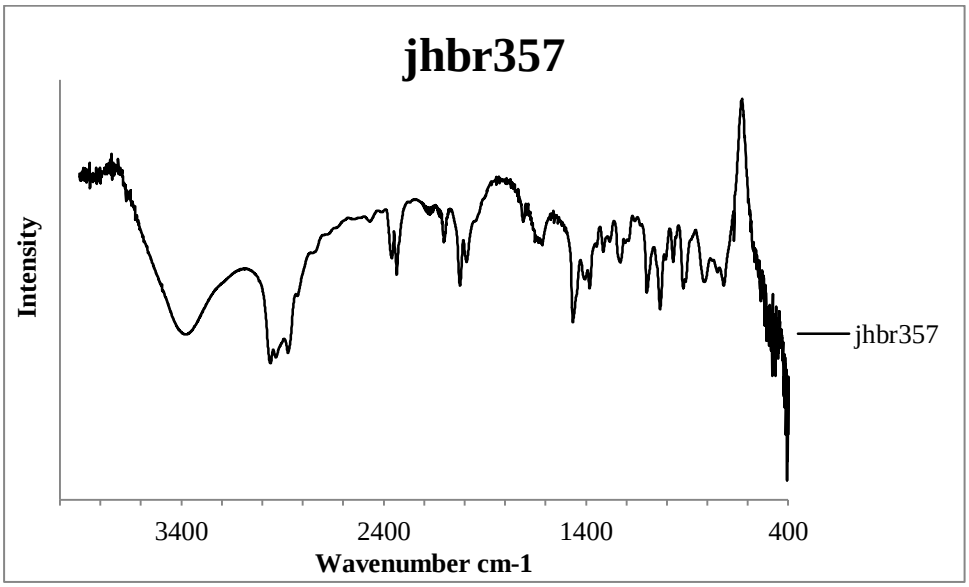


The ^{1}H NMR of the hydride region of the liquid product after reaction jhbr356

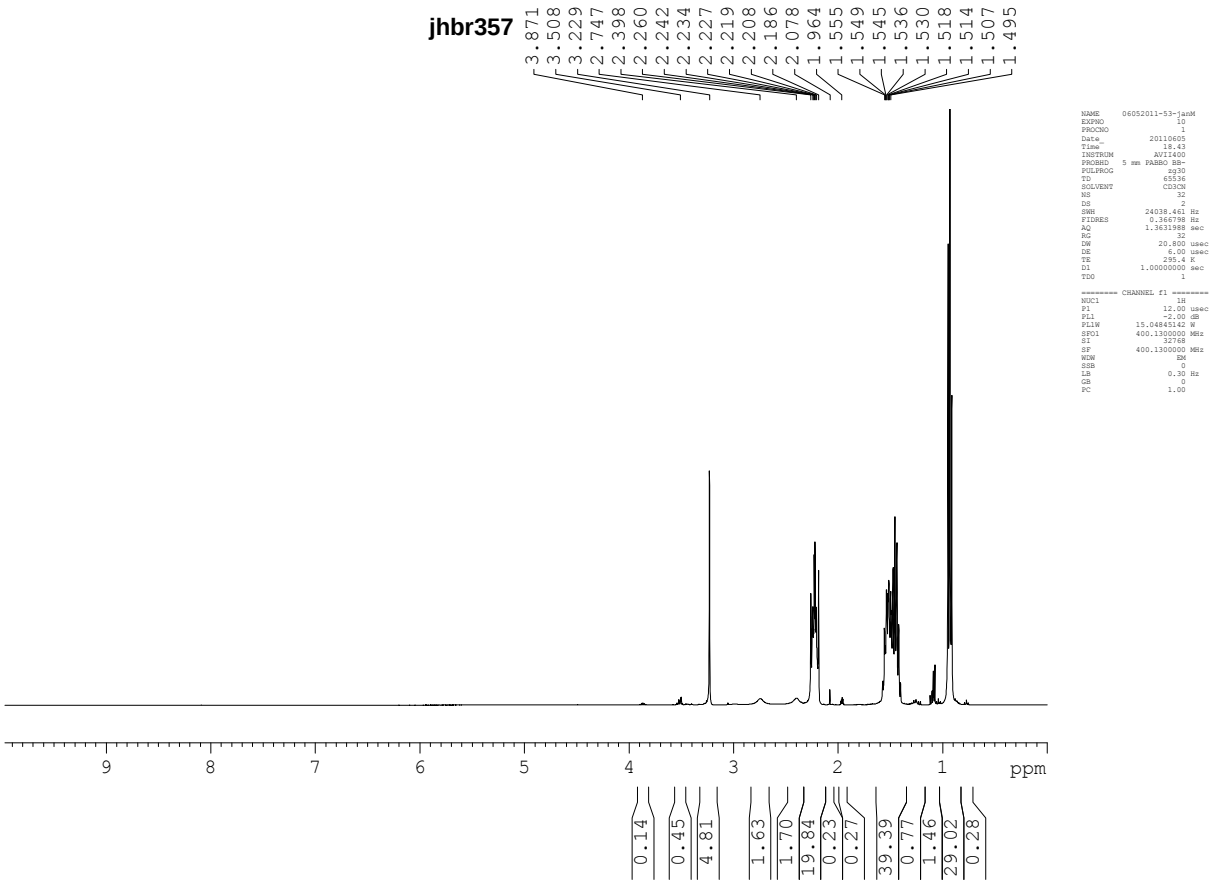


The ^{31}P NMR of the liquid product after reaction jhbr356

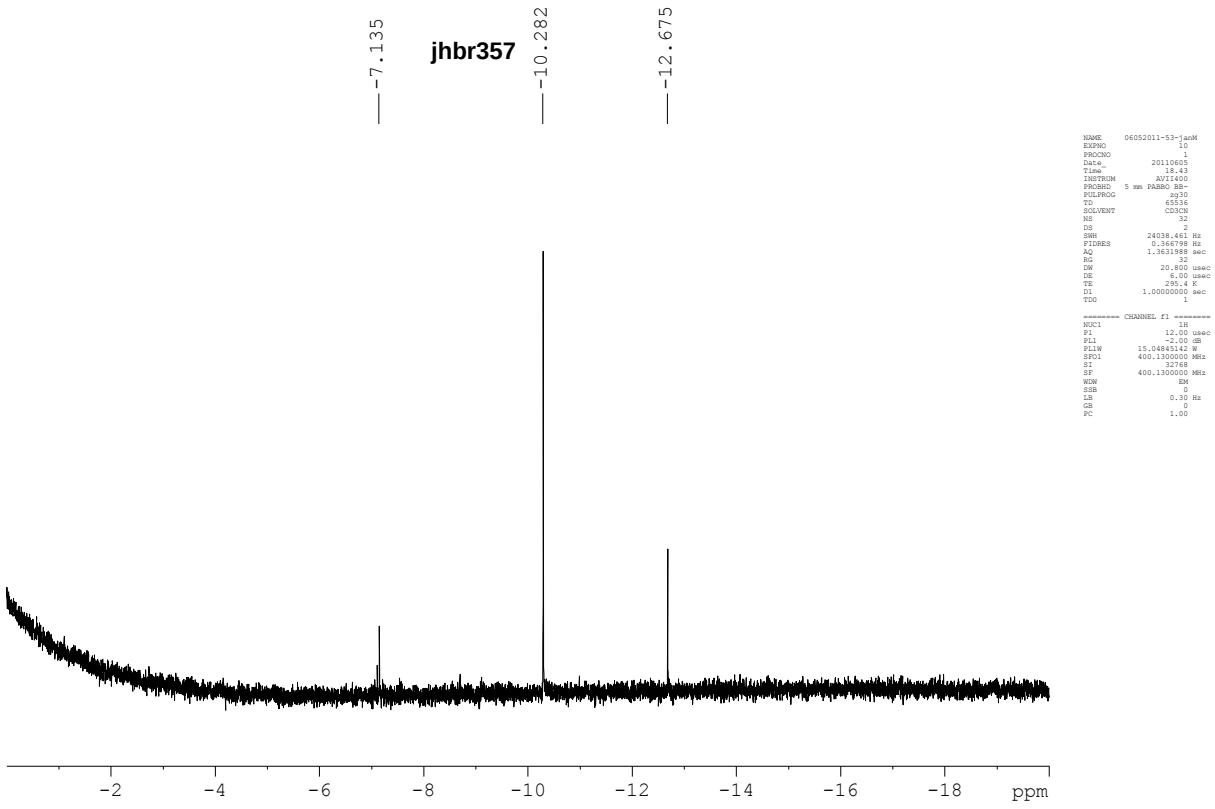
jhbr357



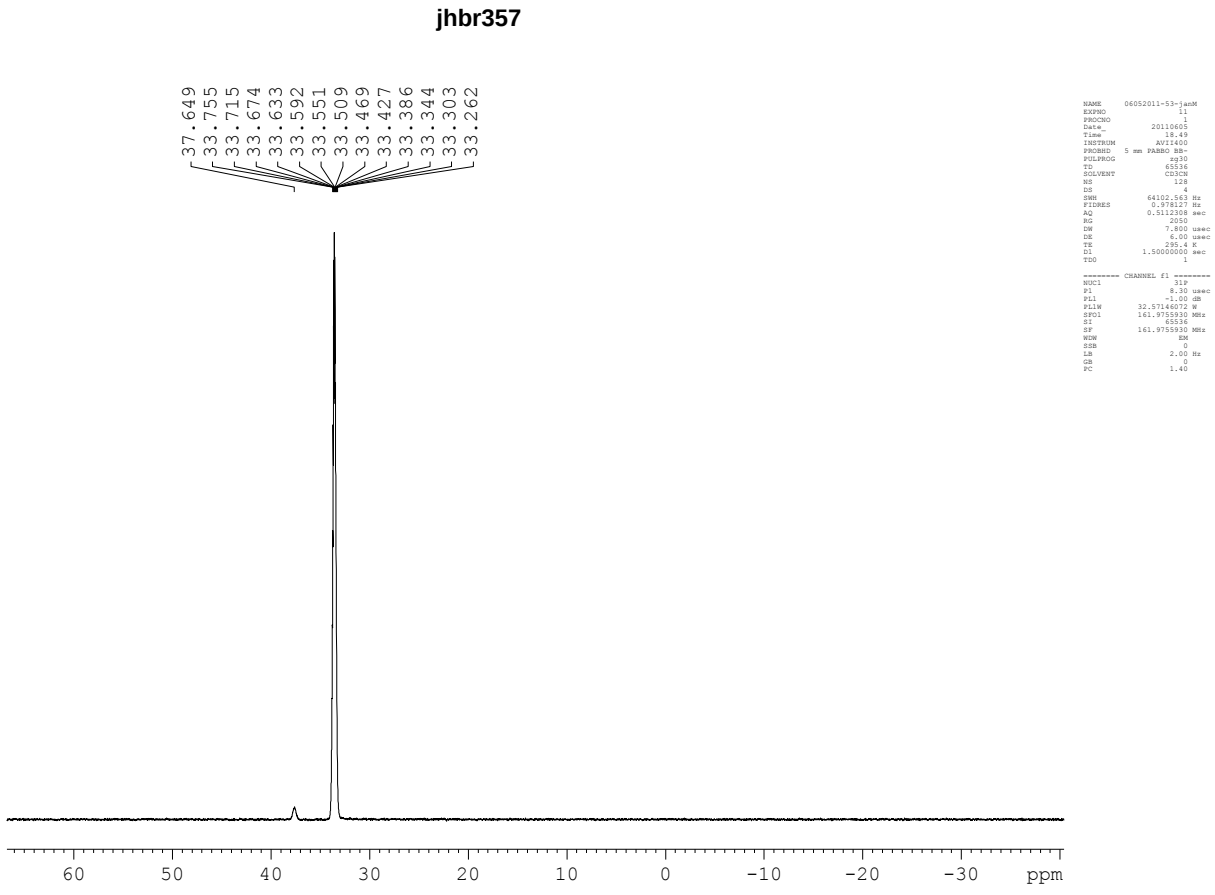
The complete IR spectrum of the liquid product after reaction of reaction jhbr357



The ^{1}H NMR of the liquid product after reaction jhbr357

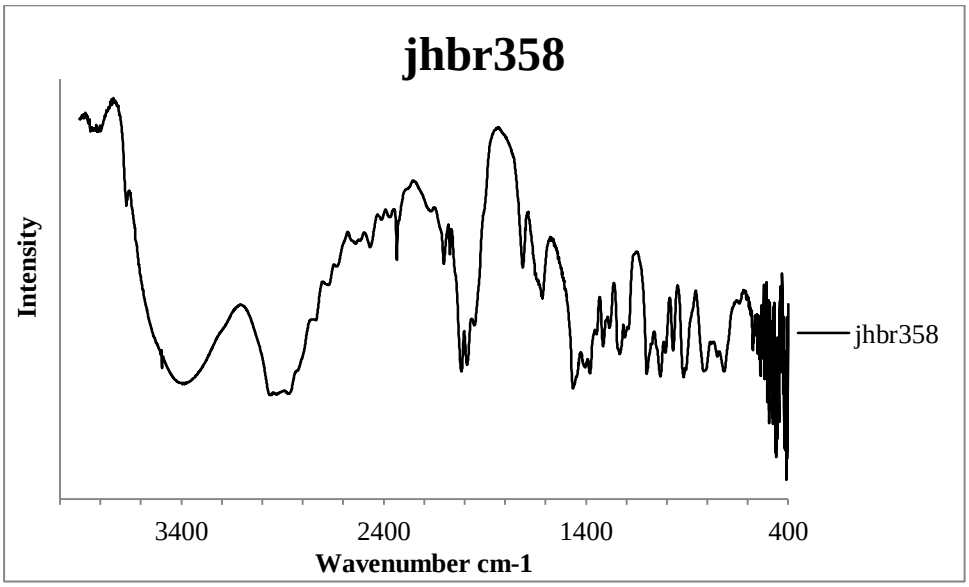


The ^{1}H NMR of the hydride region of the liquid product after reaction jhbr357



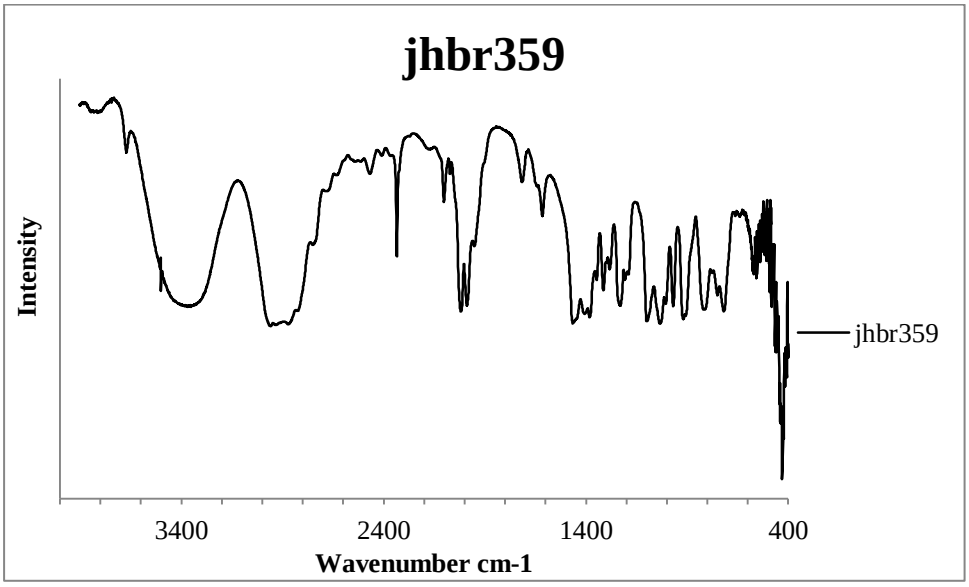
The ^{31}P NMR of the liquid product after reaction jhbr357

jhbr358



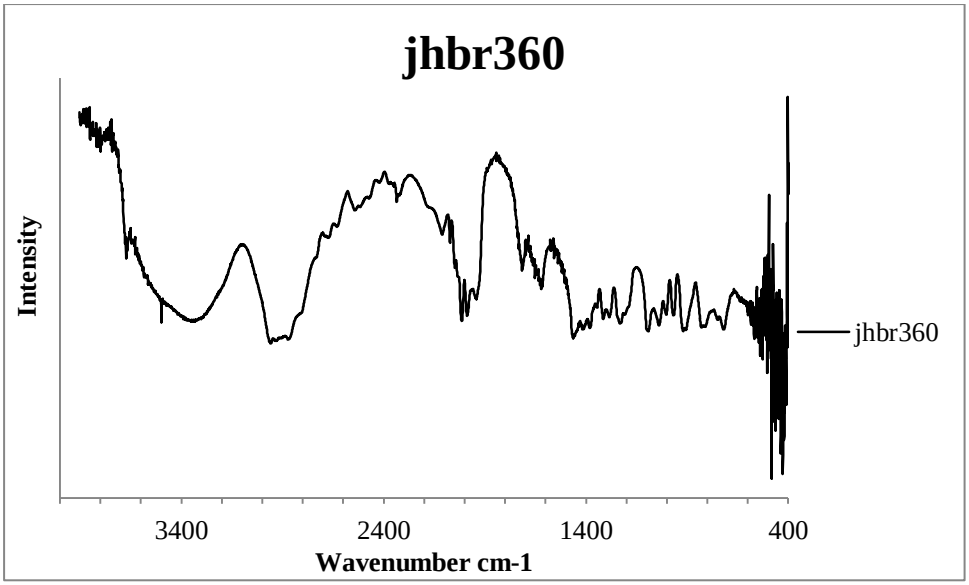
The complete IR spectrum of the liquid product after reaction of reaction jhbr358

jhbr359



The complete IR spectrum of the liquid product after reaction of reaction jhbr359

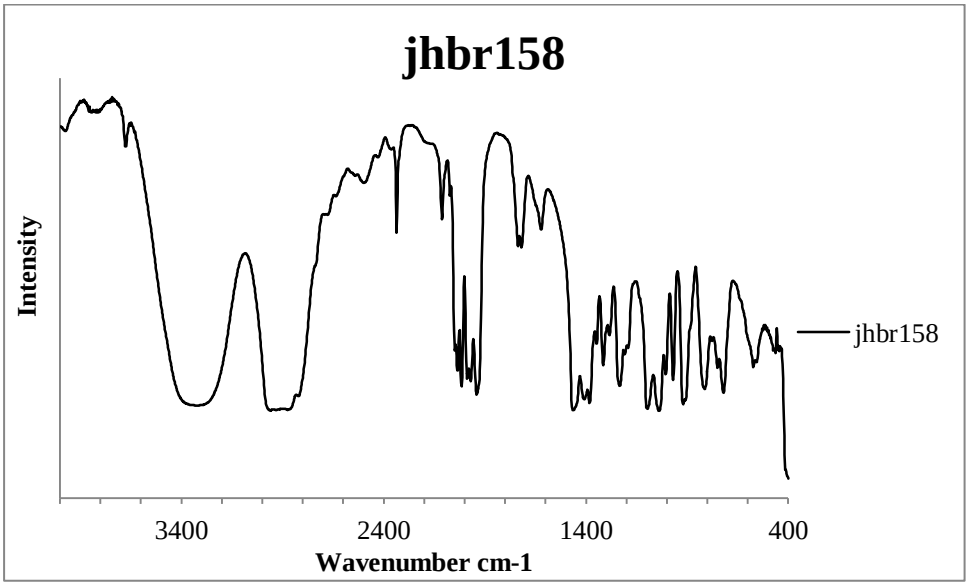
jhbr360



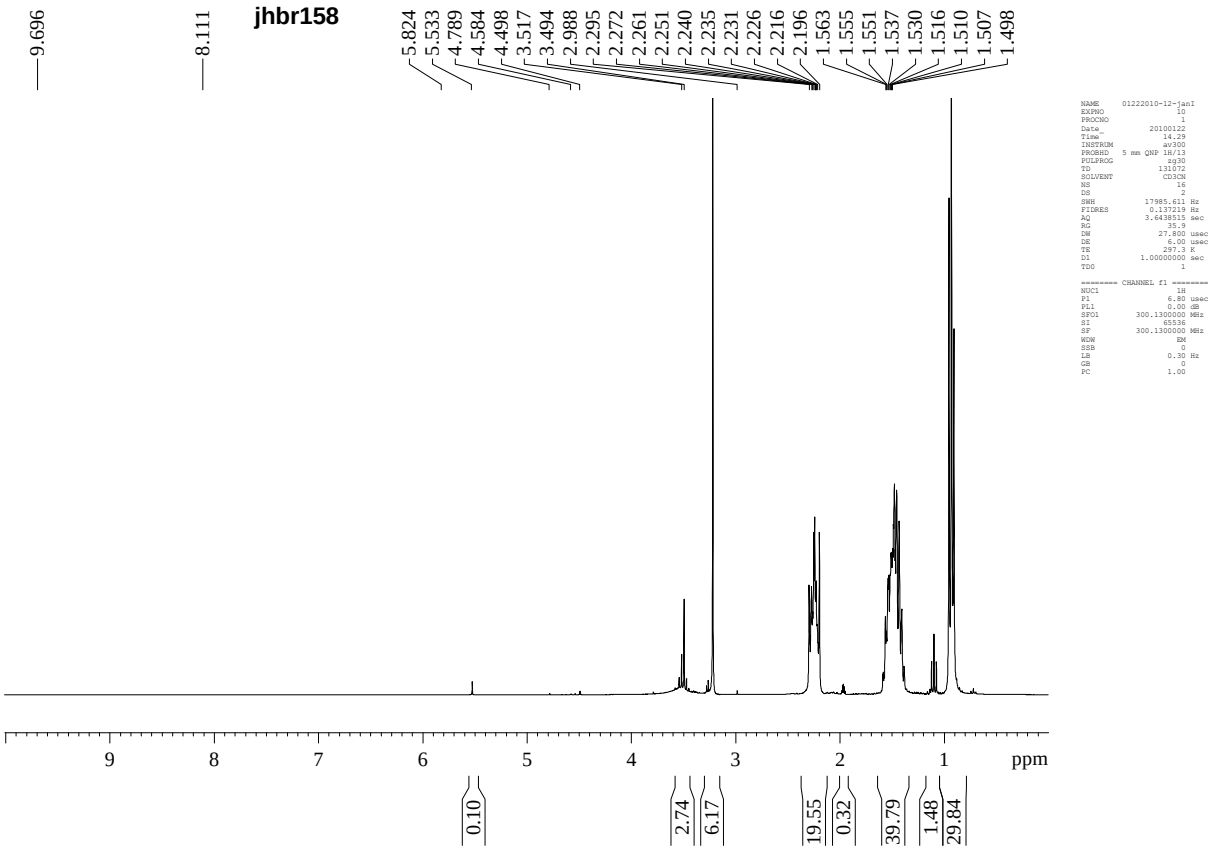
The complete IR spectrum of the liquid product after reaction of reaction jhbr360

Recorded spectra of product liquids after reaction.

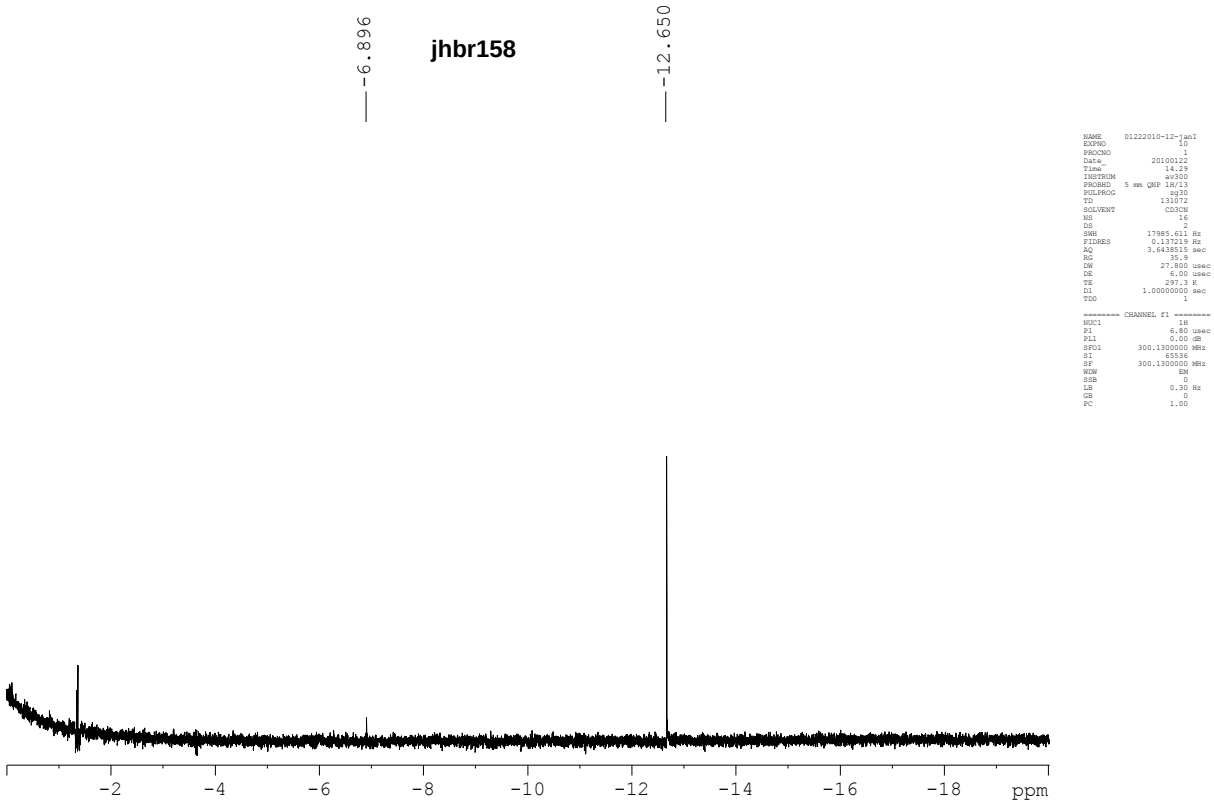
jhbr158



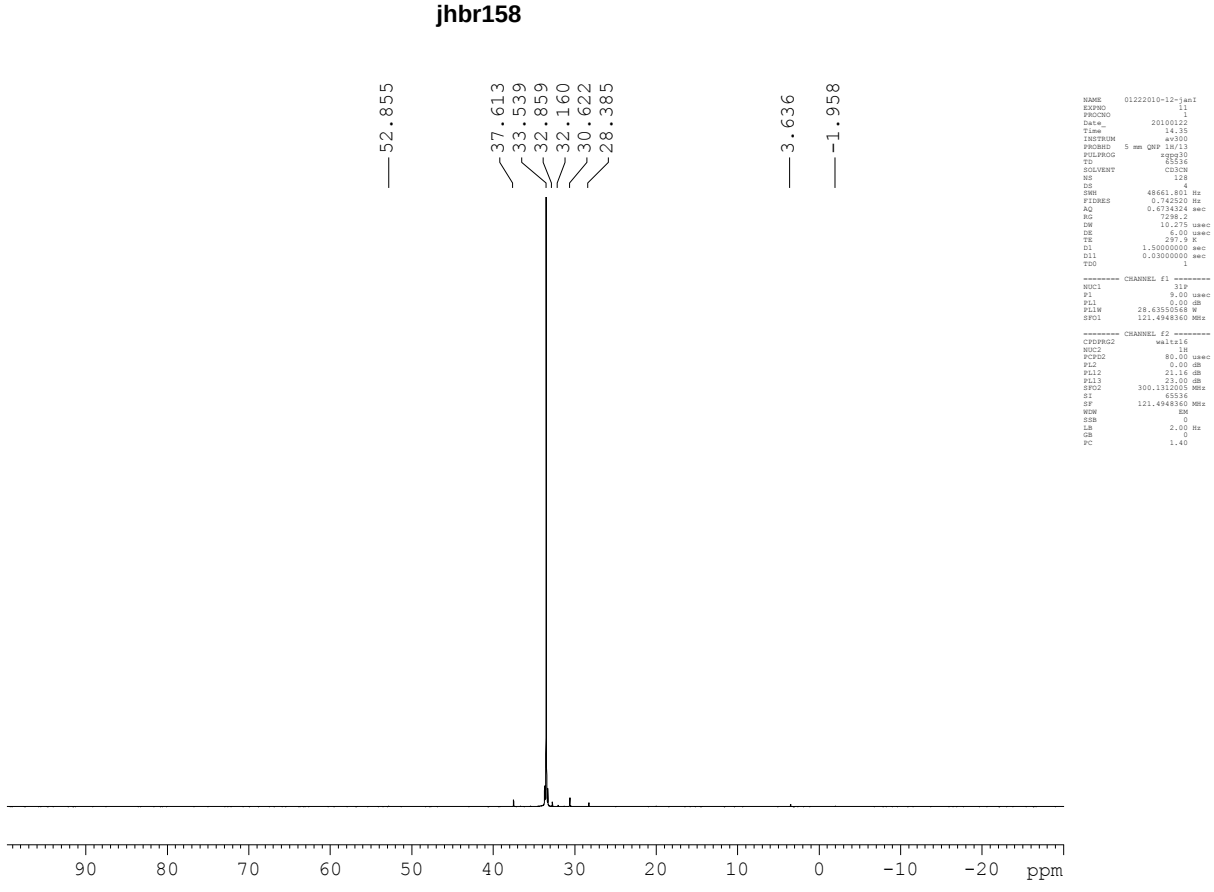
The complete IR spectrum of the liquid product after reaction of reaction jhbr158



The ^{1}H NMR of the liquid product after reaction jhbr158

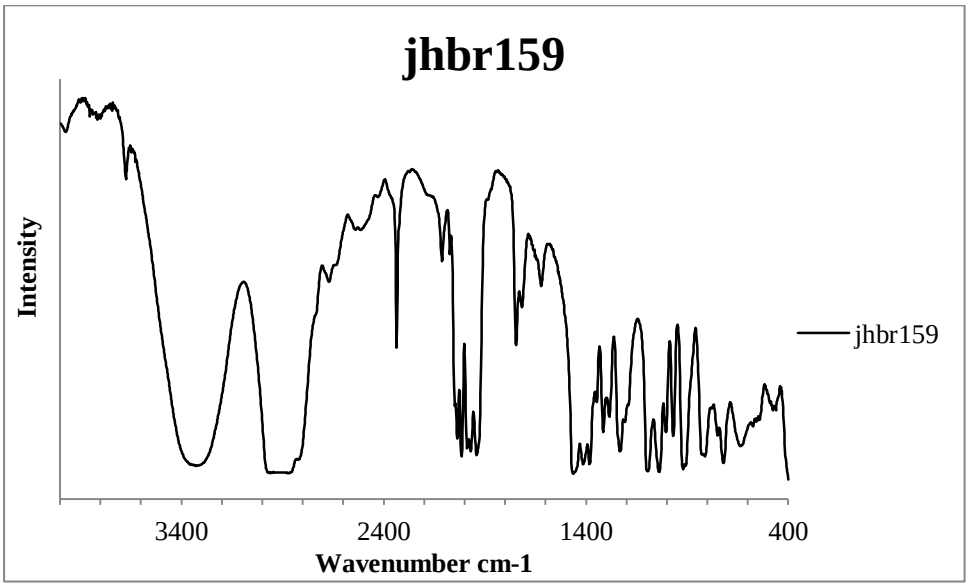


The ^{1}H NMR of the hydride region of the liquid product after reaction jhbr158

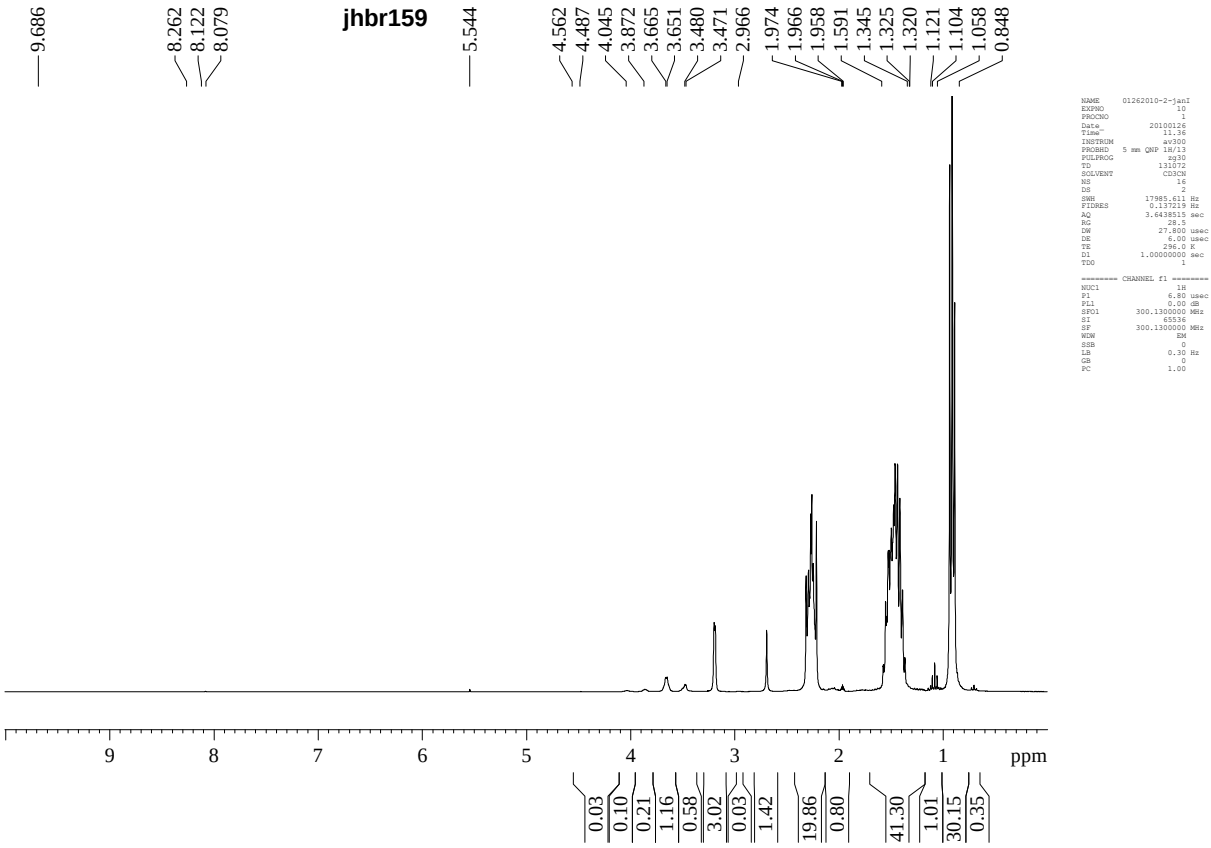


The ^{31}P NMR of the liquid product after reaction jhbr158

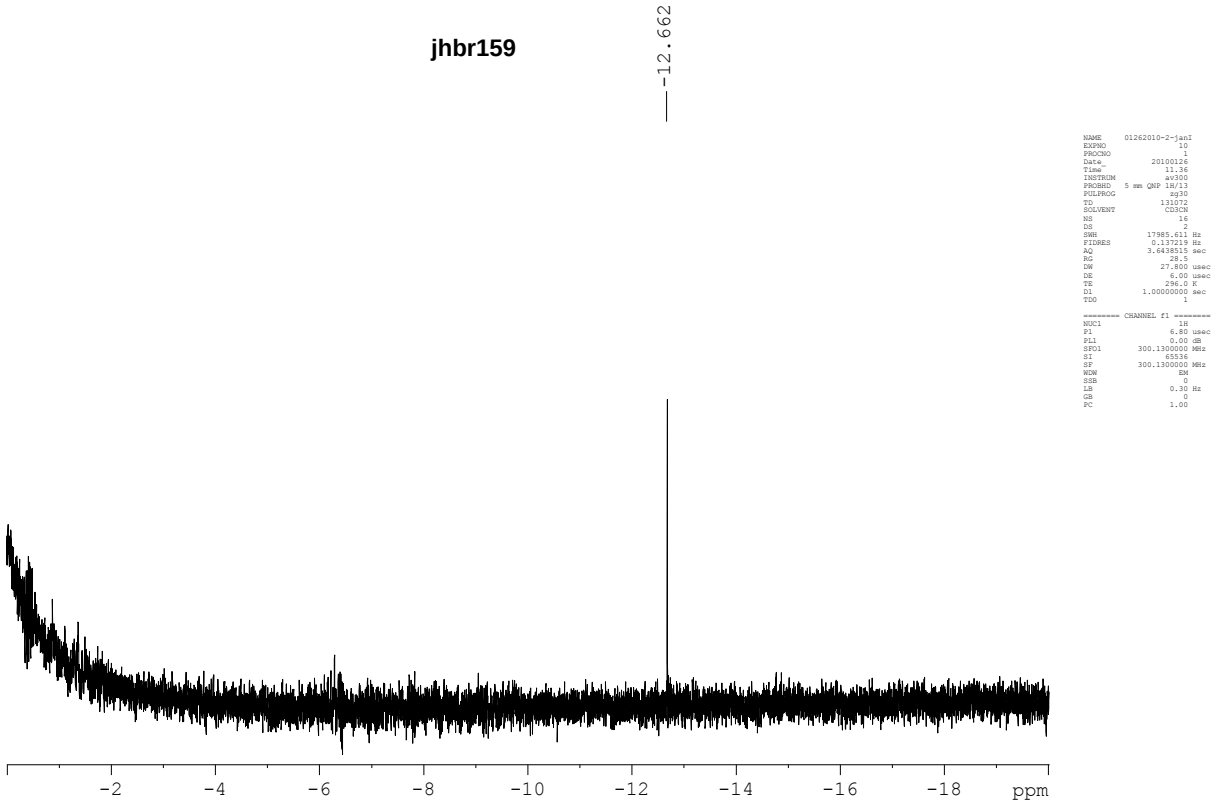
jhbr159



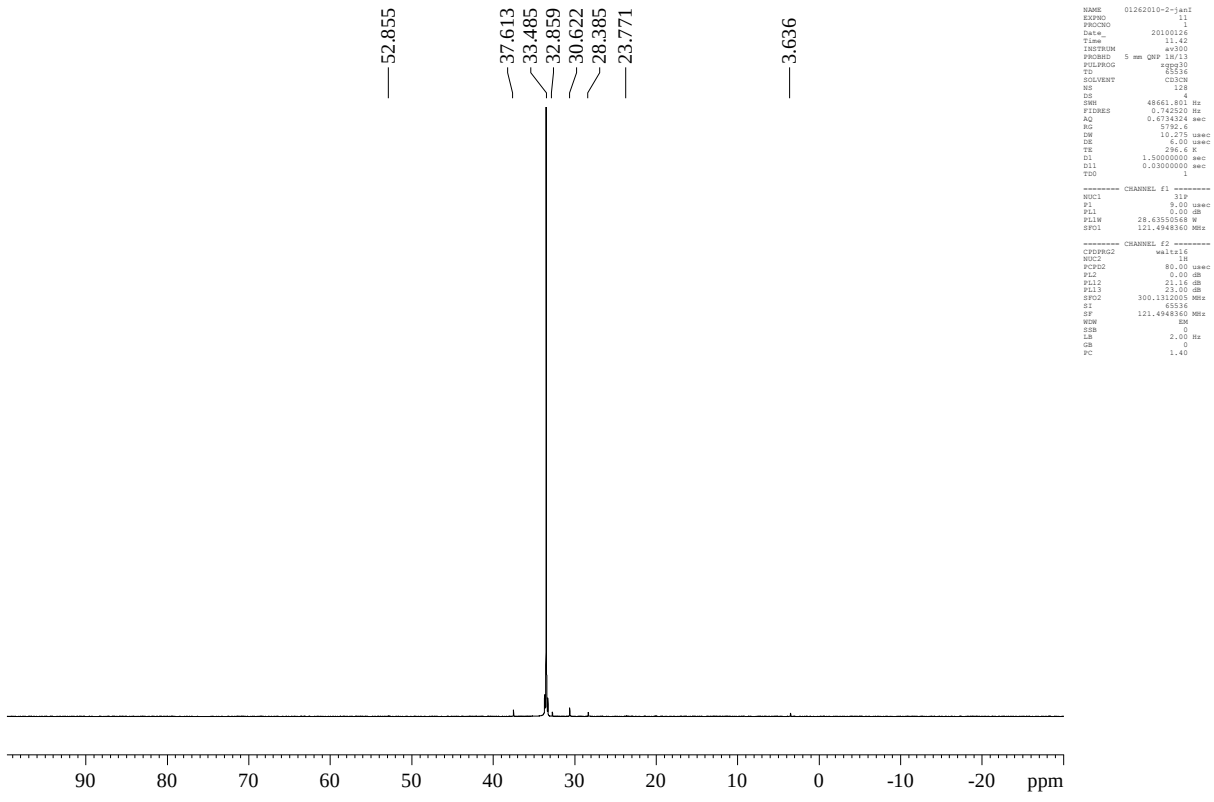
The complete IR spectrum of the liquid product after reaction of reaction jhbr159



The ^{1}H NMR of the liquid product after reaction jhbr159

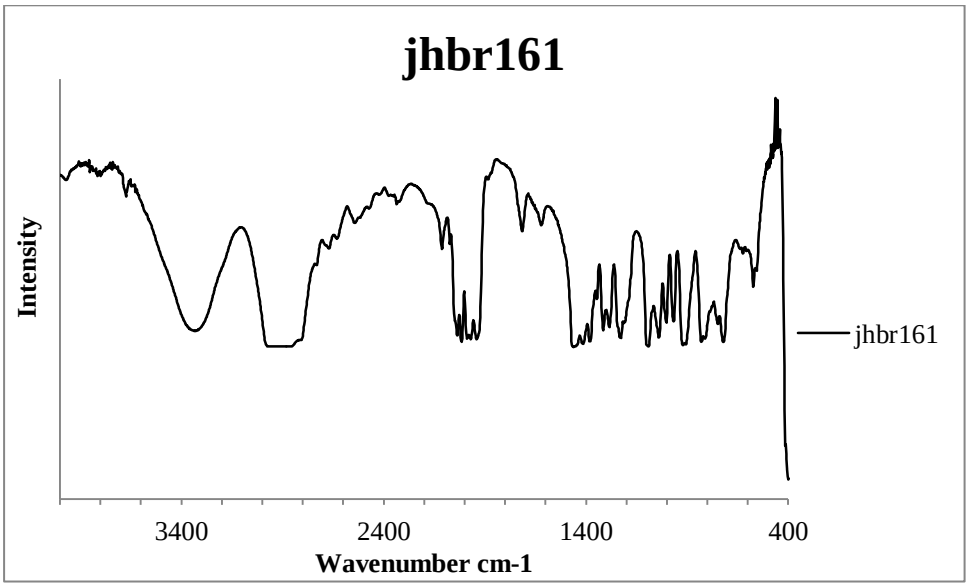


The ^{1}H NMR of the hydride region of the liquid product after reaction jhbr159

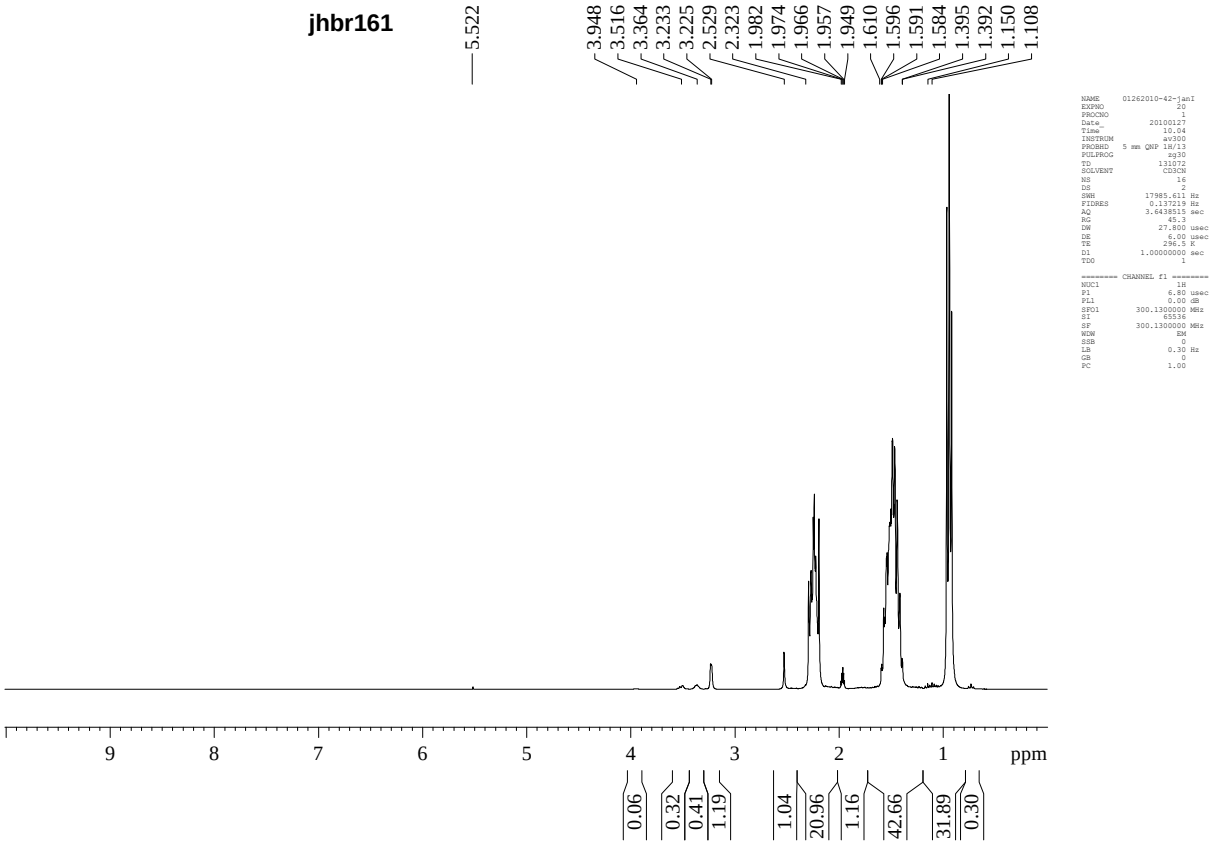


The ^{31}P NMR of the liquid product after reaction jhbr159

jhbr161

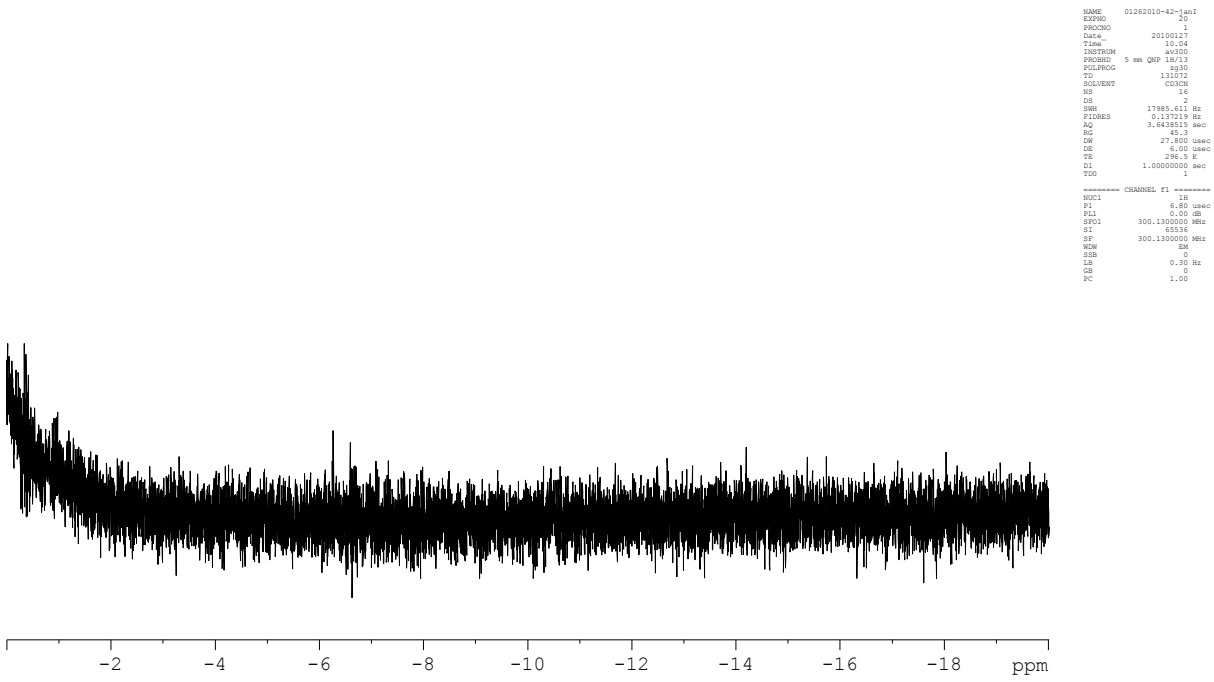


The complete IR spectrum of the liquid product after reaction of reaction jhbr161



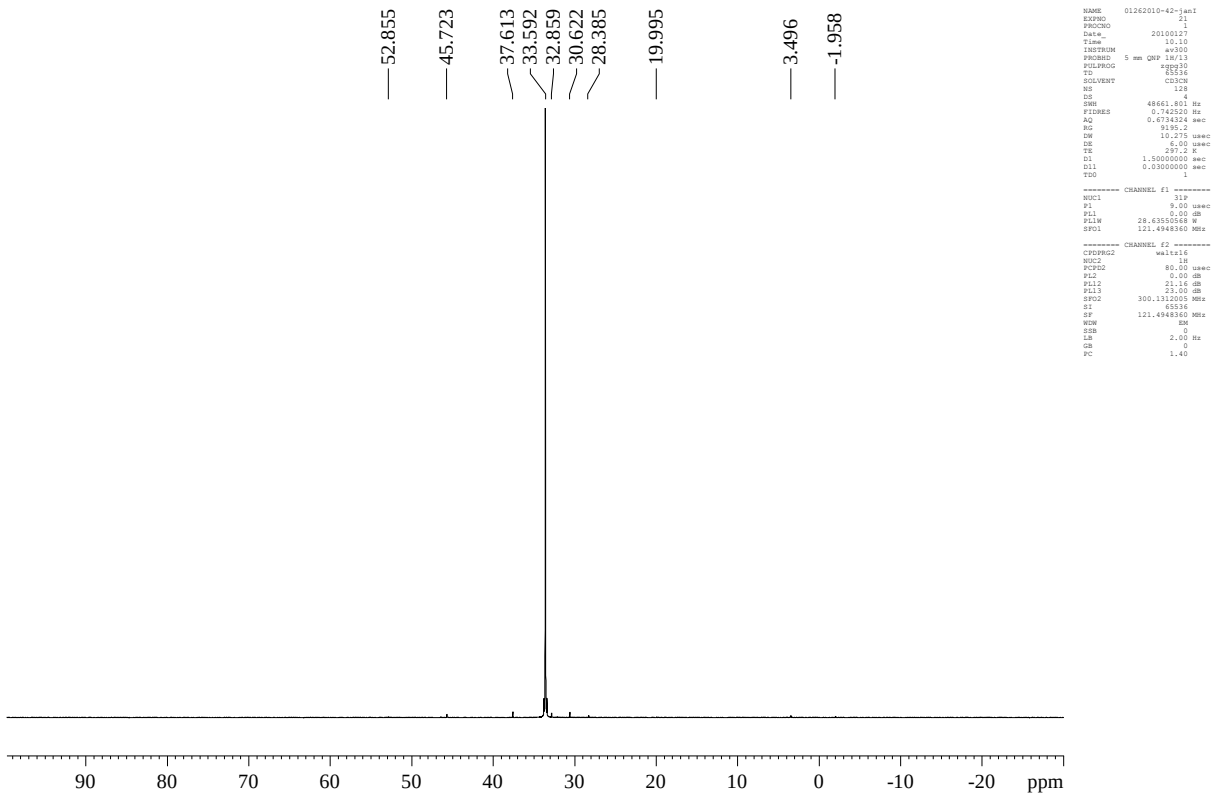
The ^{1}H NMR of the liquid product after reaction jhbr161

jhbr161



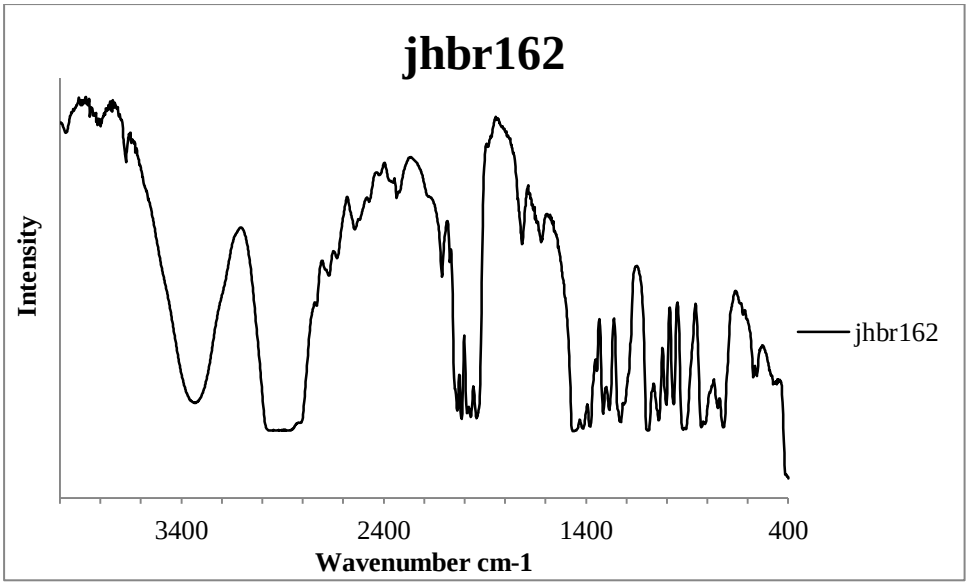
The ^{1}H NMR of the hydride region of the liquid product after reaction jhbr161

jhbr161

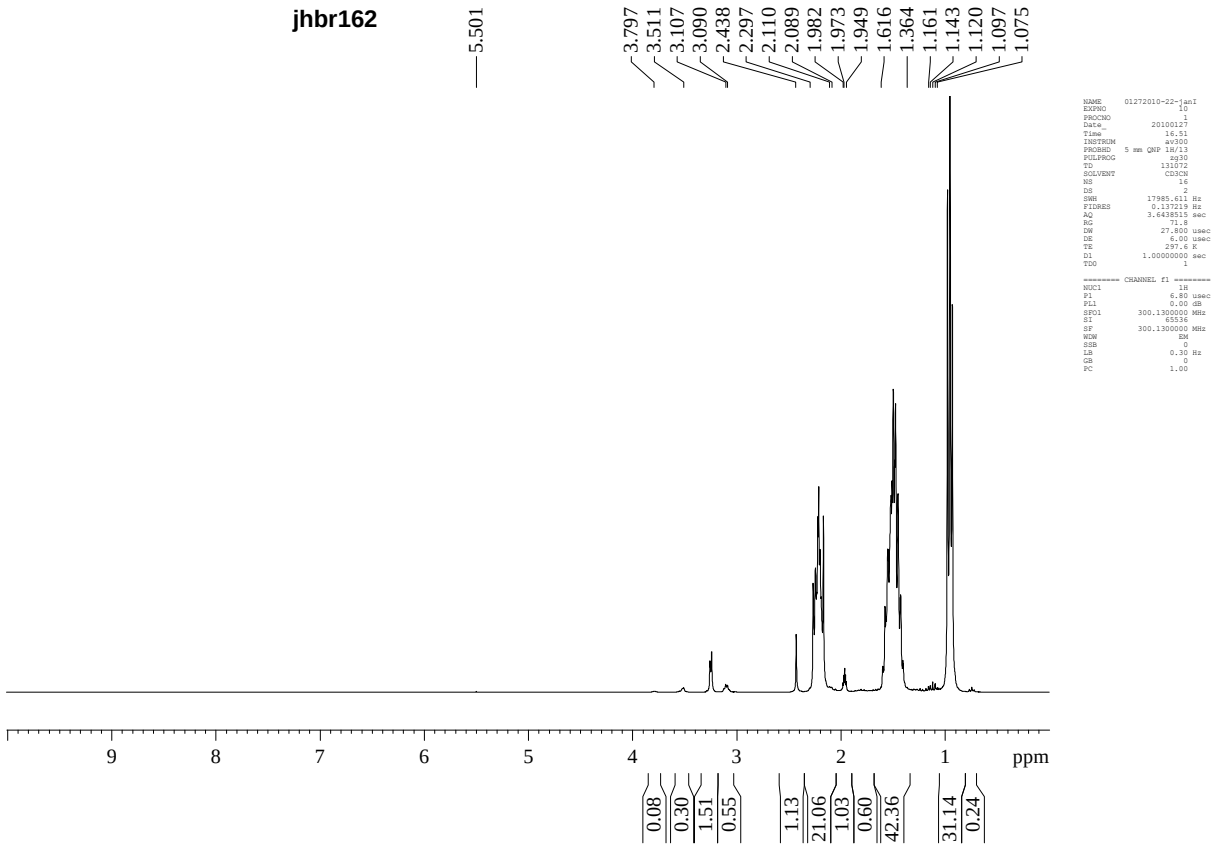


The ^{31}P NMR of the liquid product after reaction jhbr161

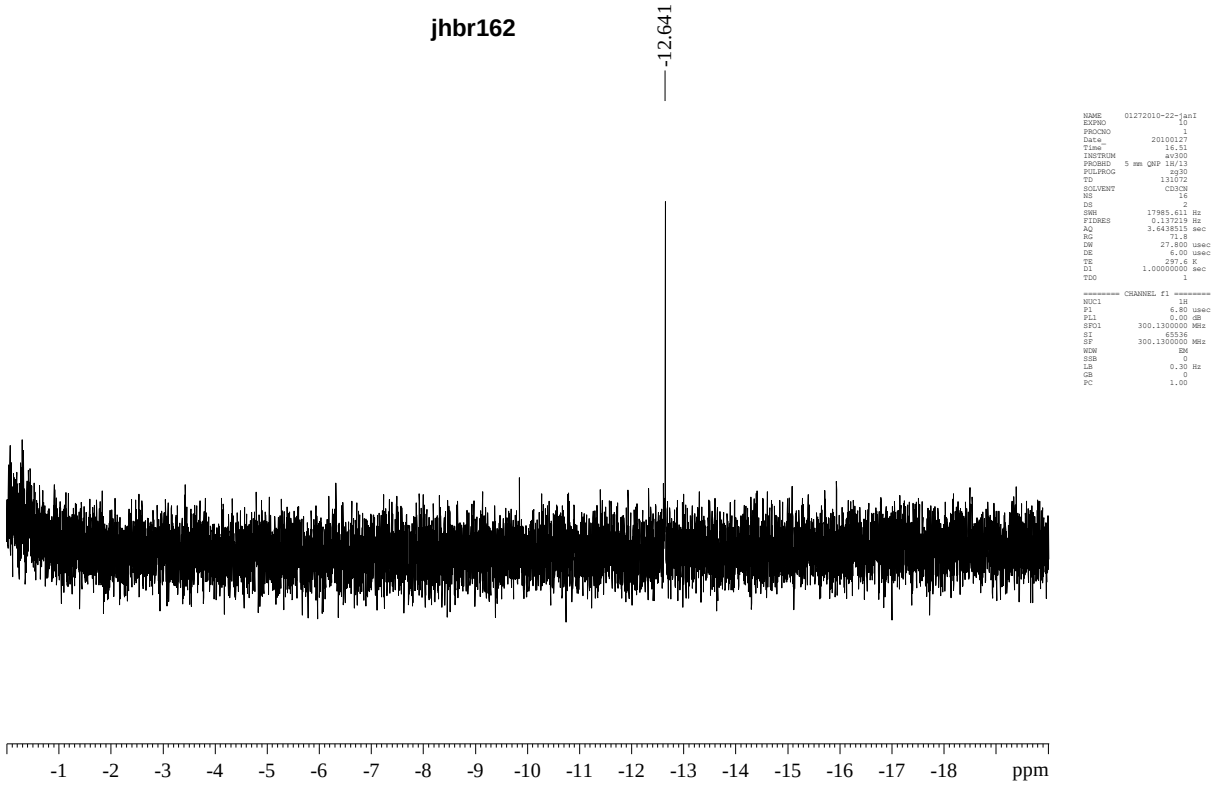
jhbr162



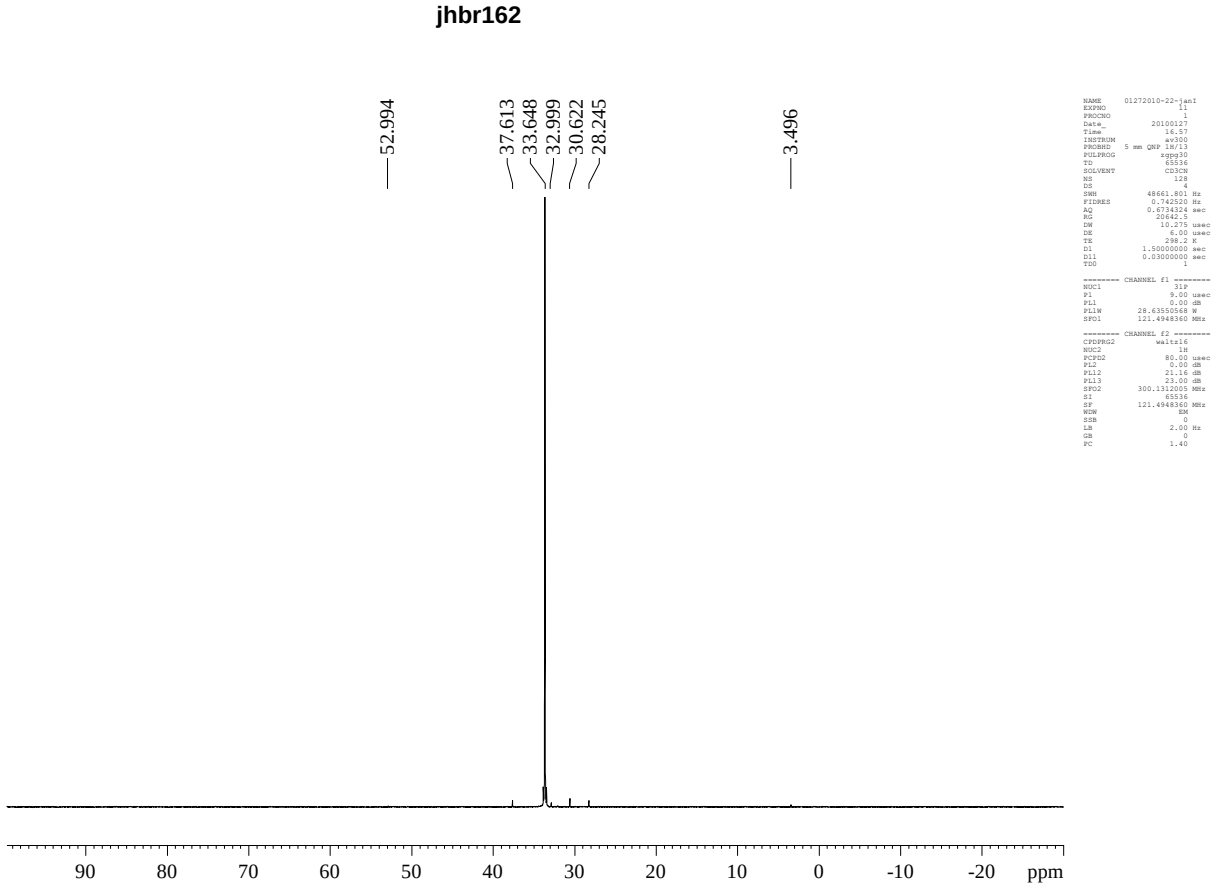
The complete IR spectrum of the liquid product after reaction of reaction jhbr162



The ^{1}H NMR of the liquid product after reaction jhbr162

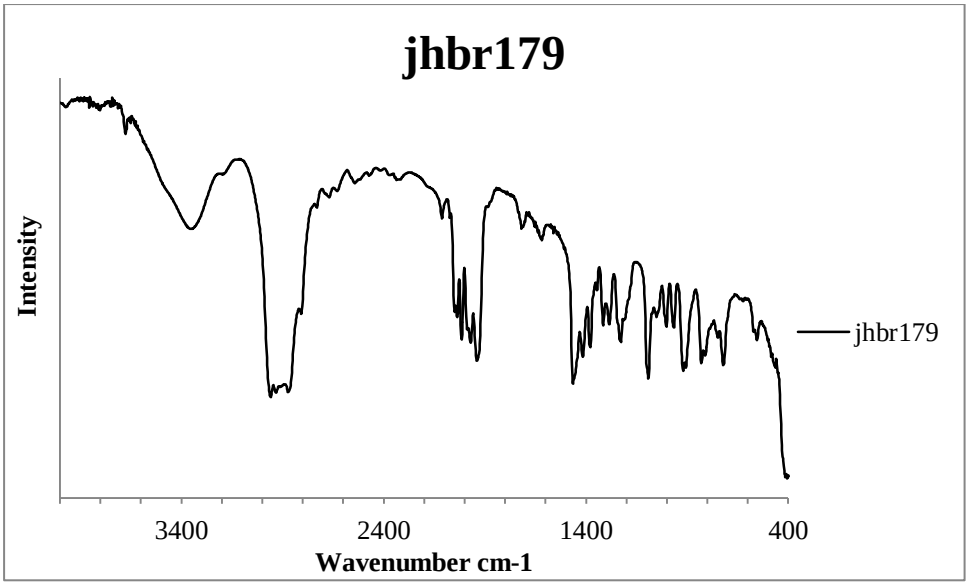


The ^{1}H NMR of the hydride region of the liquid product after reaction jhbr162

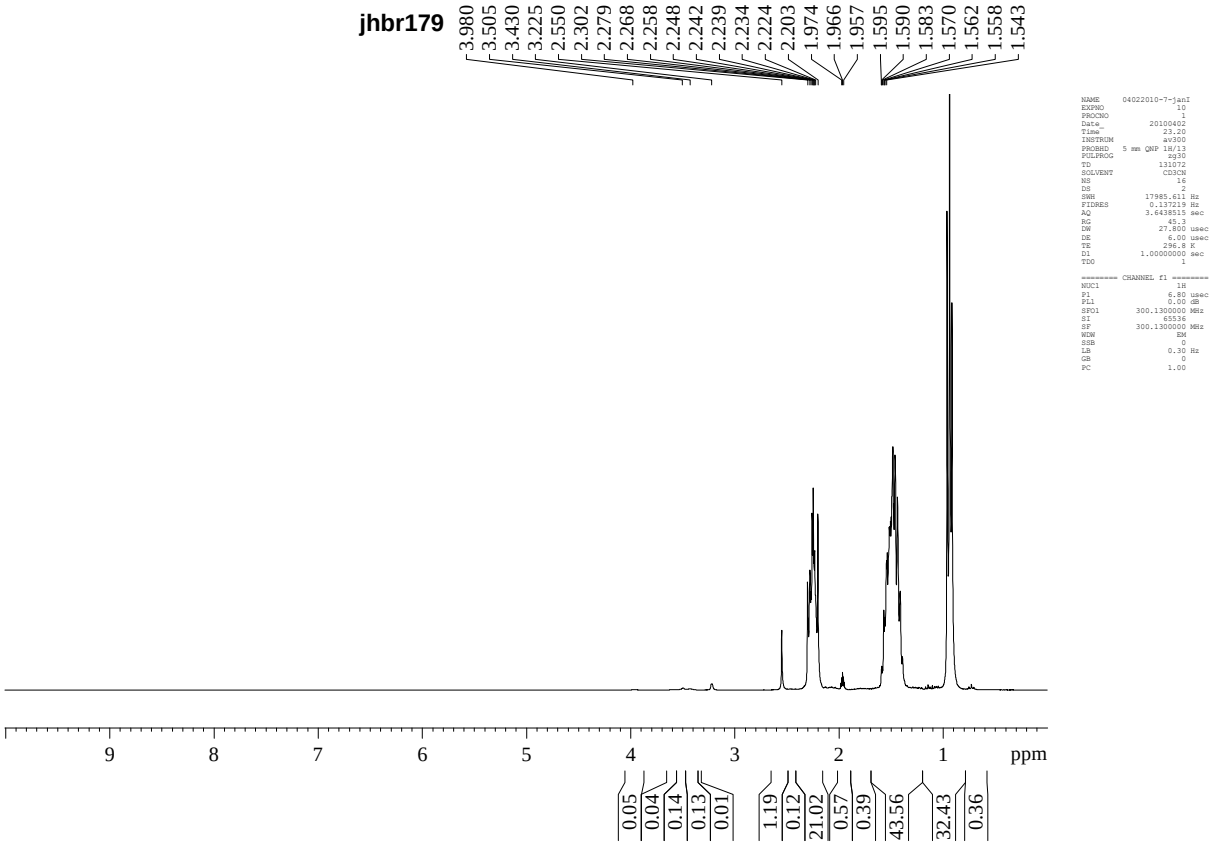


The ^{31}P NMR of the liquid product after reaction jhbr162

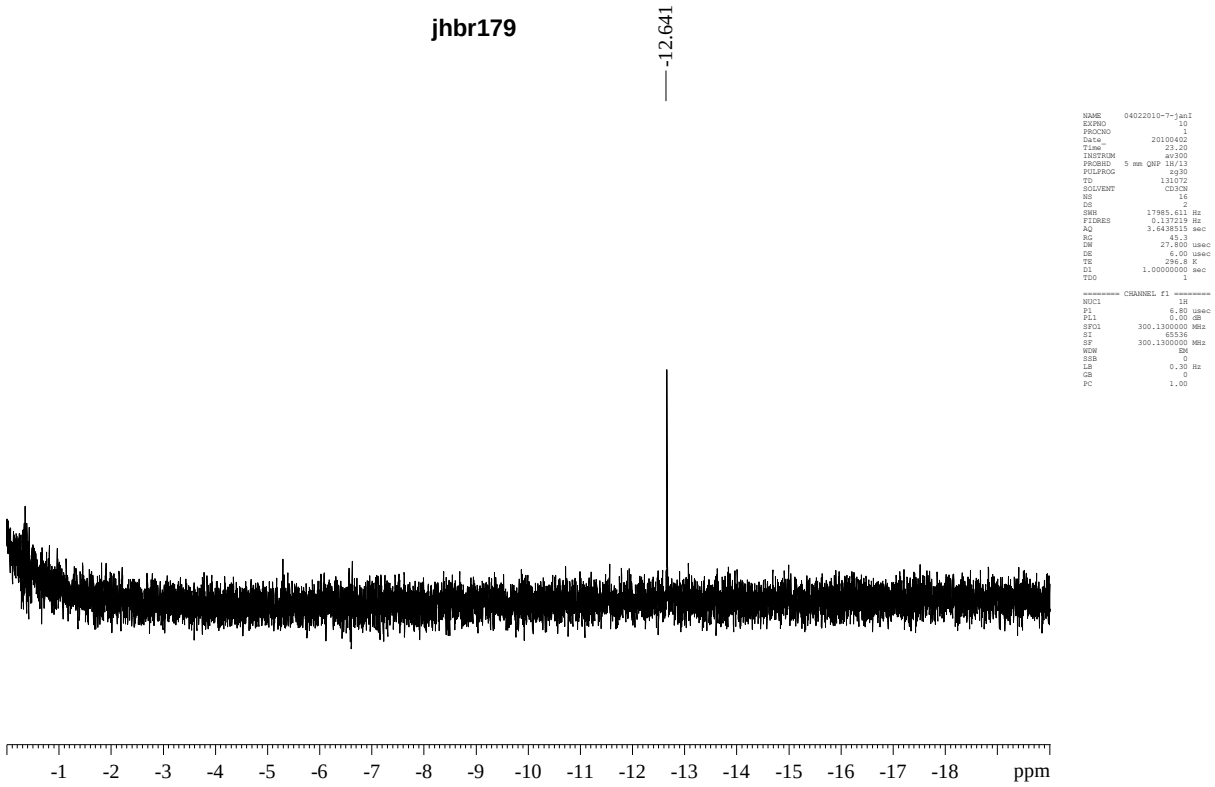
jhbr179



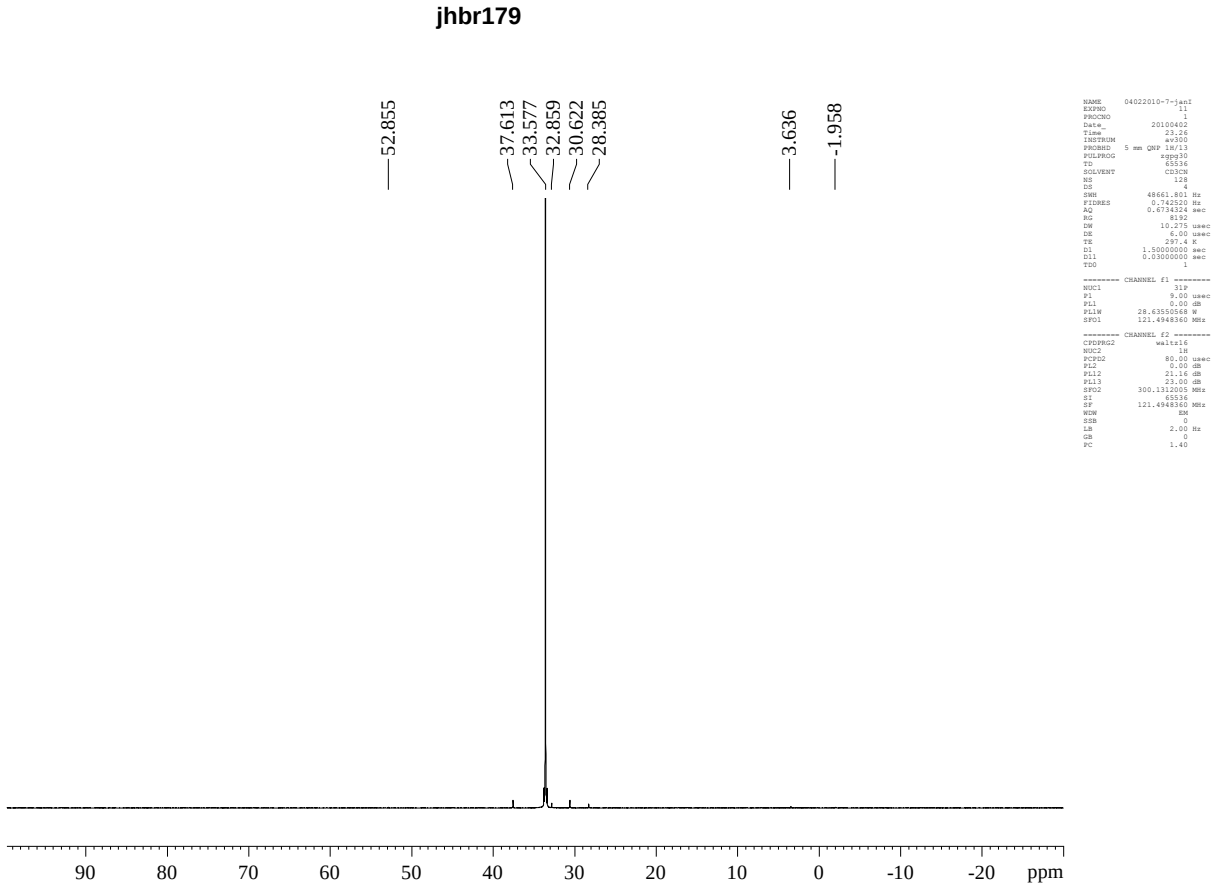
The complete IR spectrum of the liquid product after reaction of reaction jhbr179



The ^{1}H NMR of the liquid product after reaction jhbr179

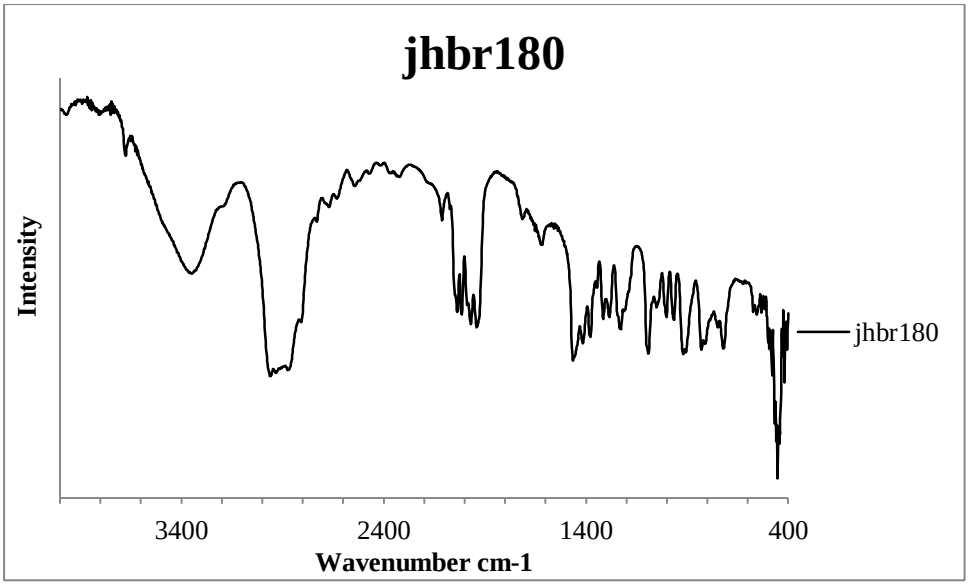


The ^{1}H NMR of the hydride region of the liquid product after reaction jhbr179

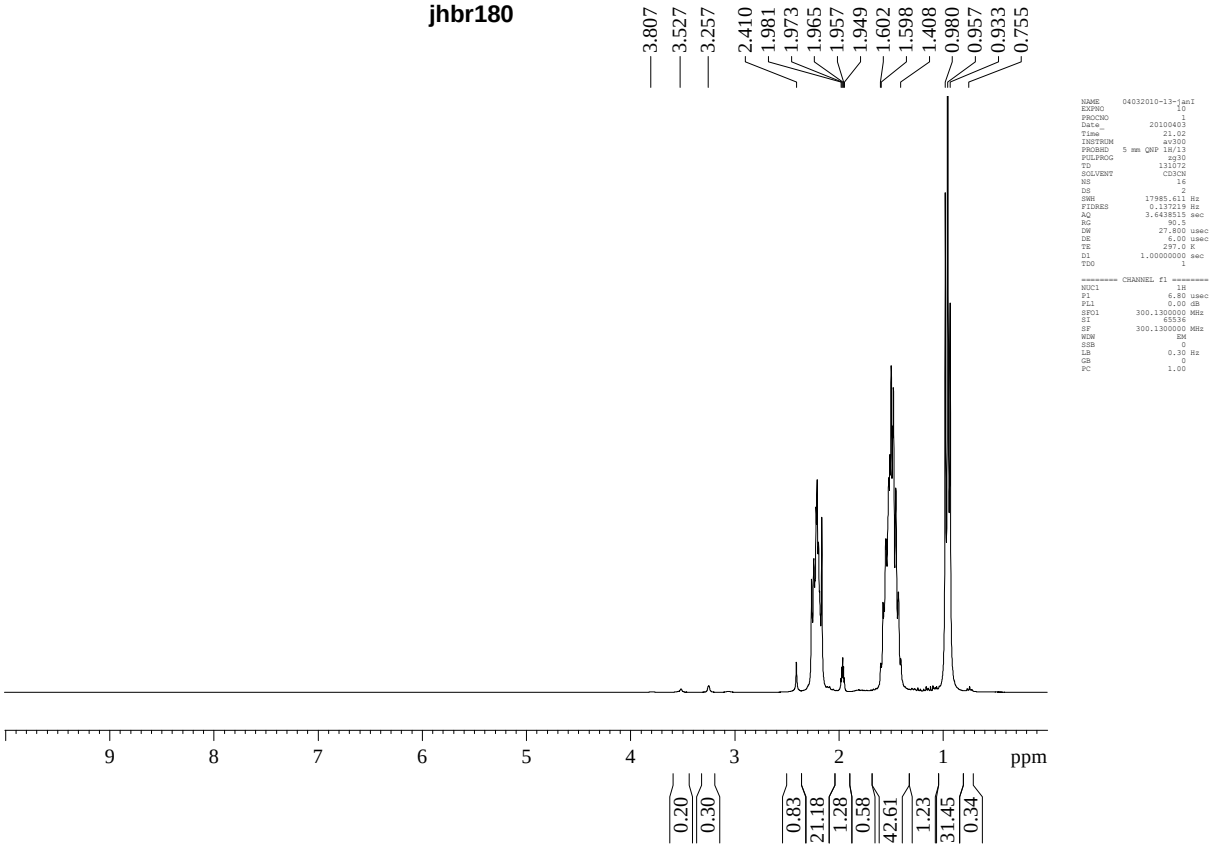


The ^{31}P NMR of the liquid product after reaction jhbr179

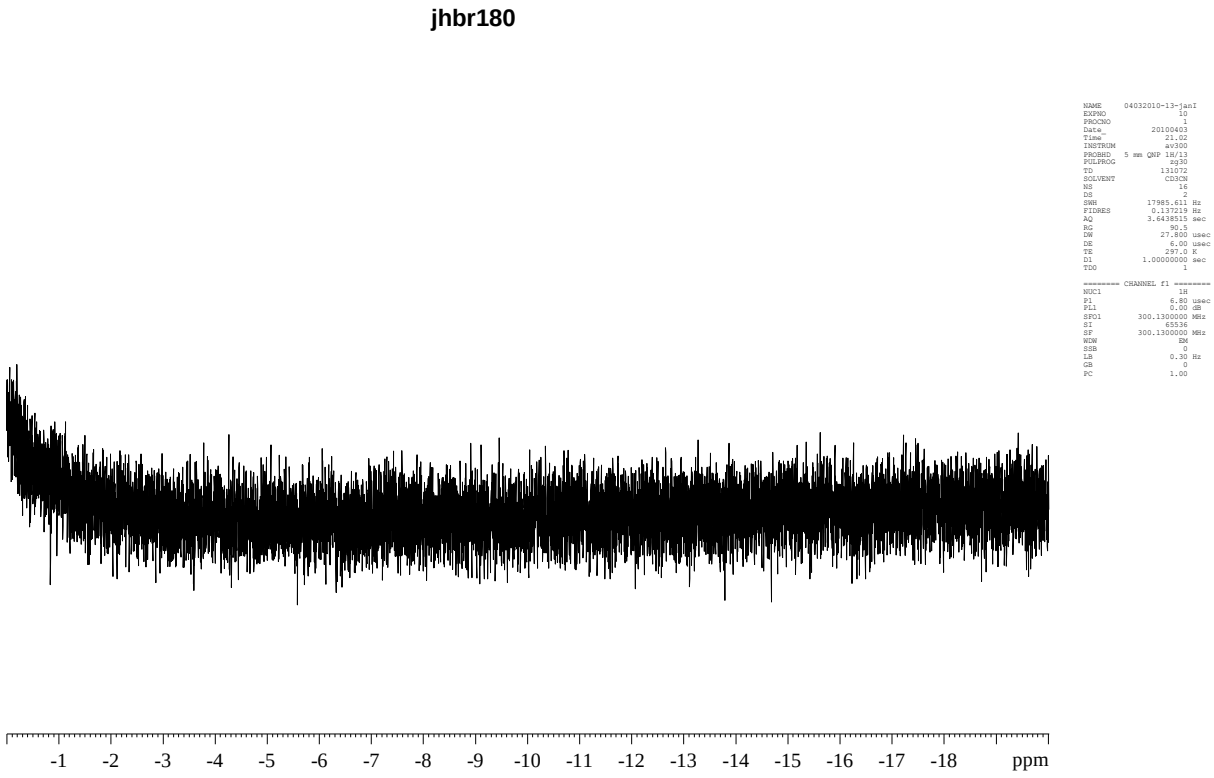
jhbr180



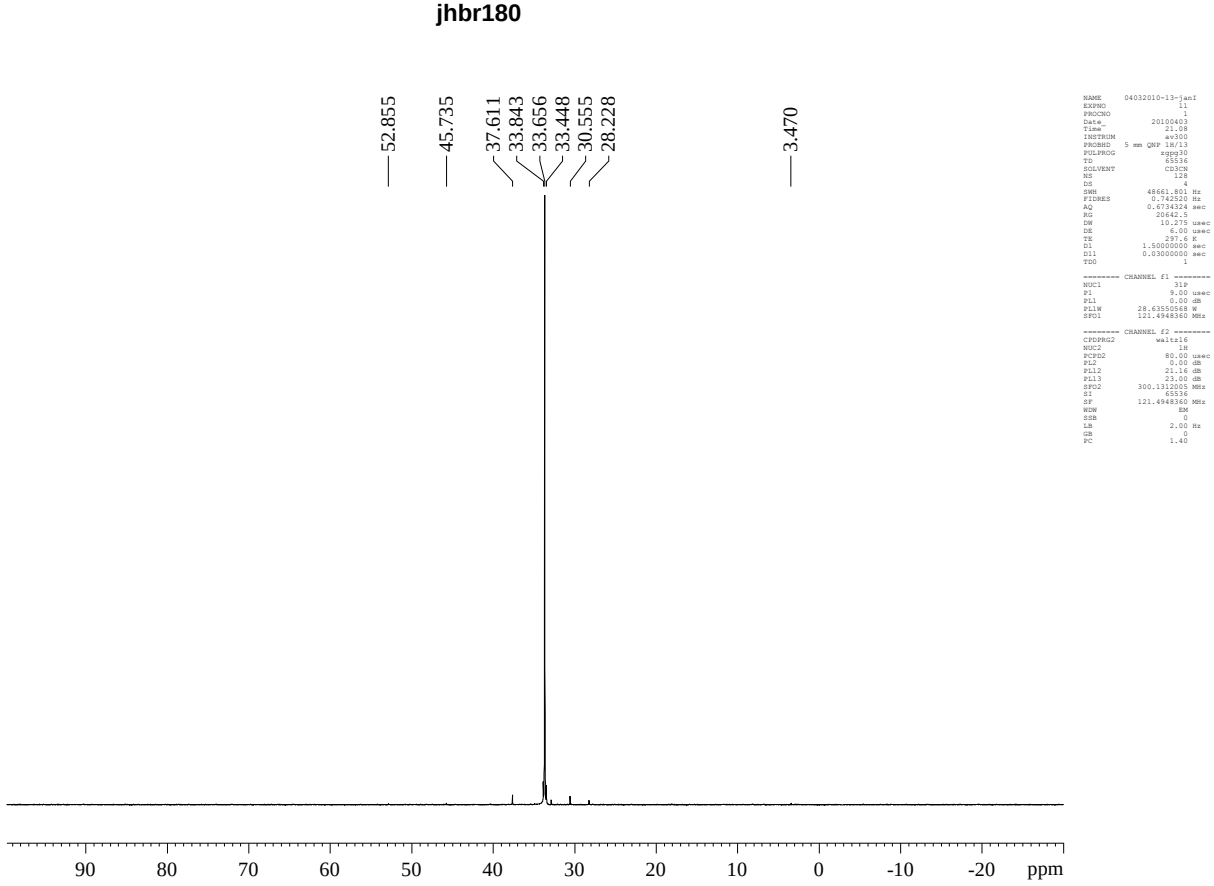
The complete IR spectrum of the liquid product after reaction of reaction jhbr180



The ^{1}H NMR of the liquid product after reaction jhbr180

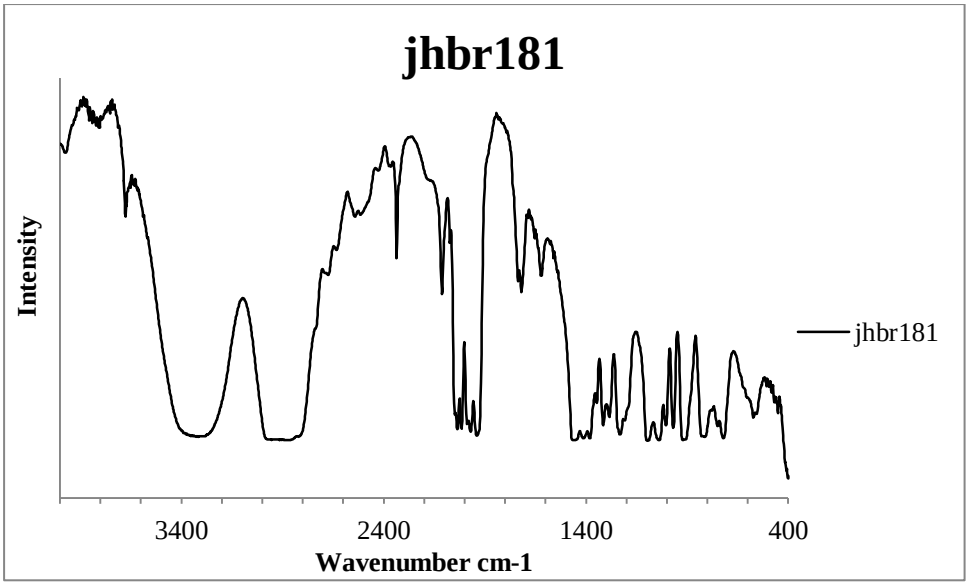


The ^{1}H NMR of the hydride region of the liquid product after reaction jhbr180



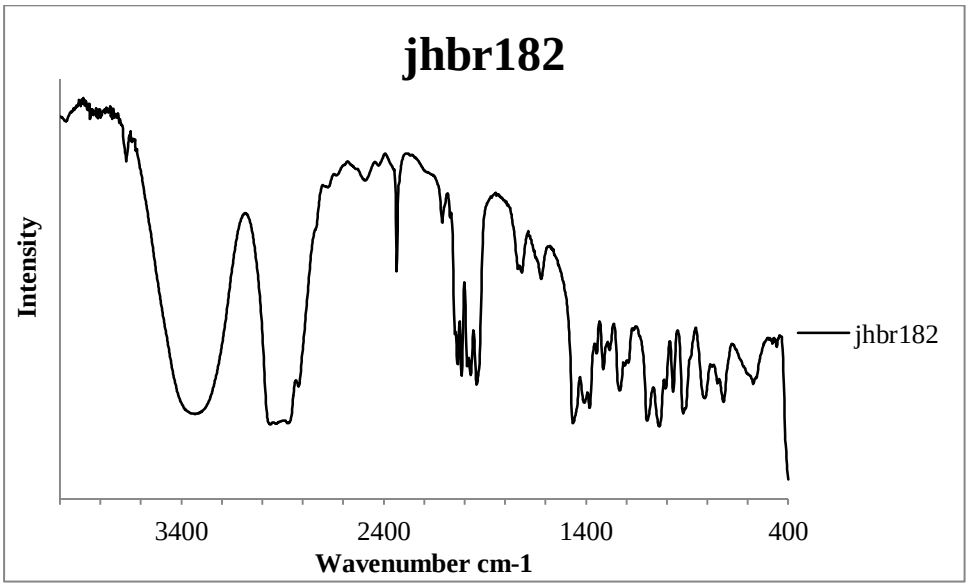
The ^{31}P NMR of the liquid product after reaction jhbr180

jhbr181



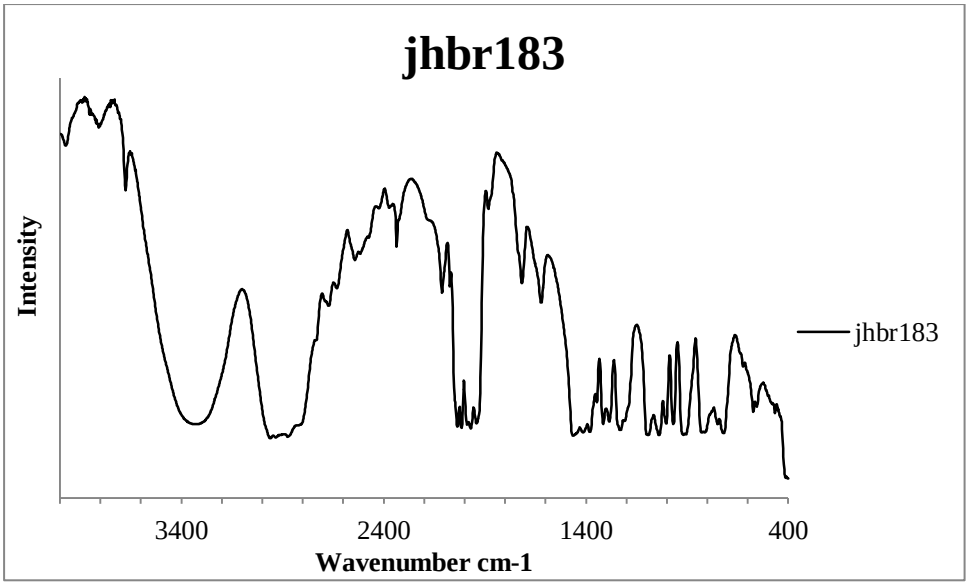
The complete IR spectrum of the liquid product after reaction of reaction jhbr181

jhbr182



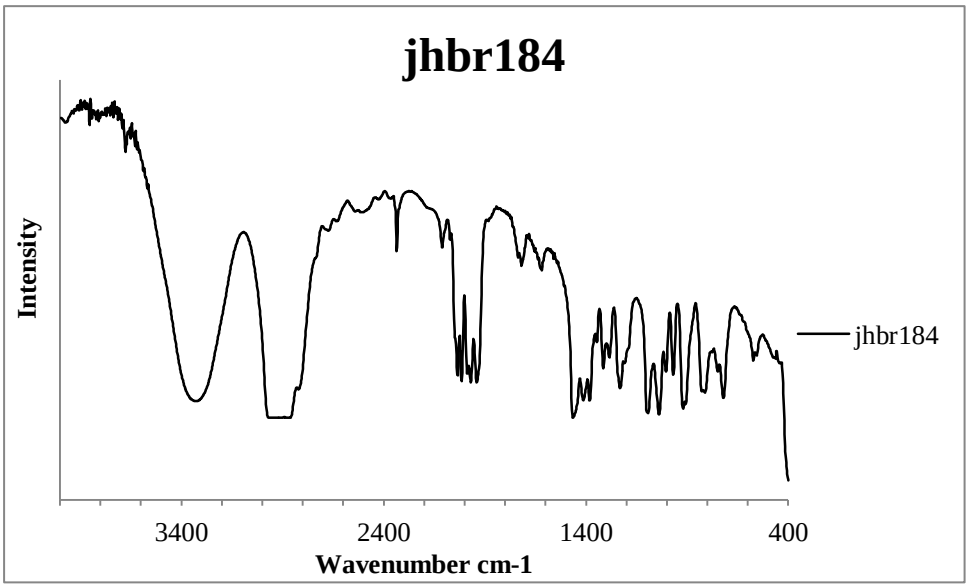
The complete IR spectrum of the liquid product after reaction of reaction jhbr182

jhbr183

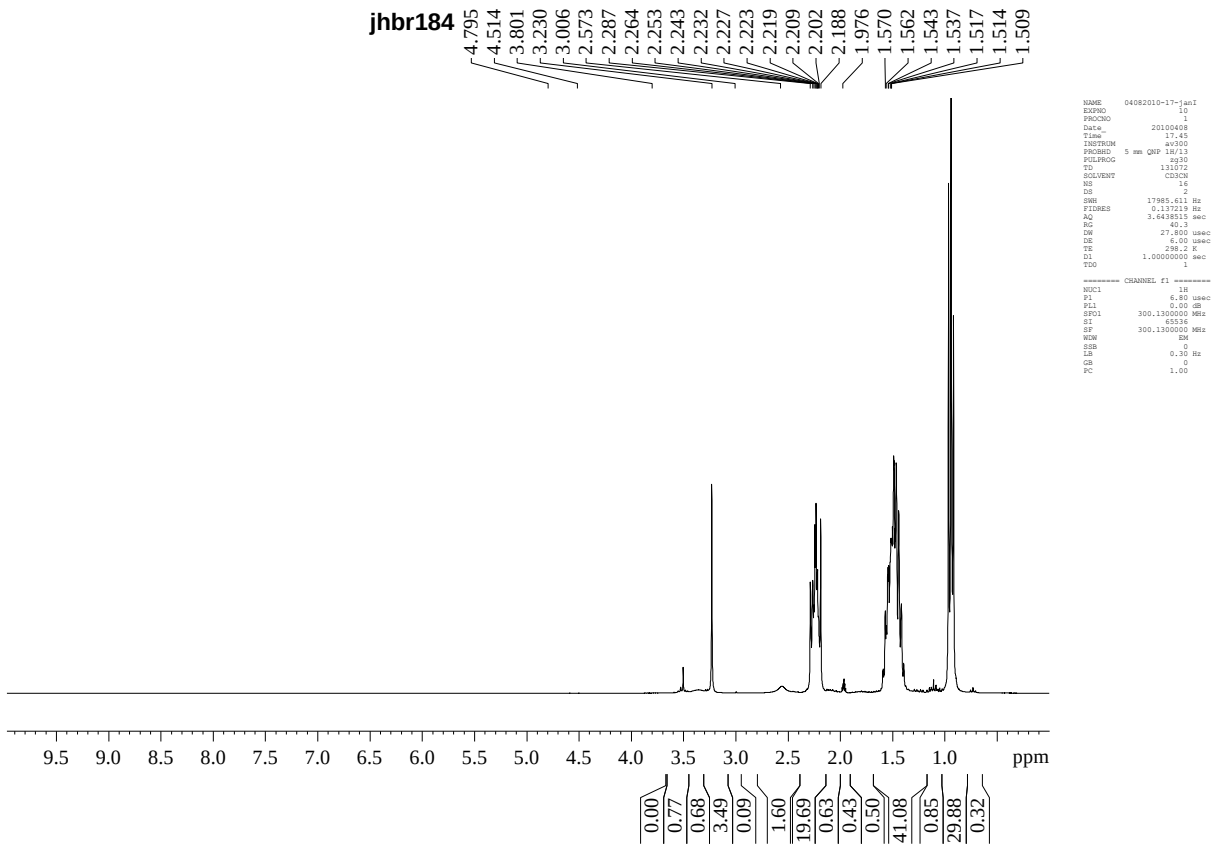


The complete IR spectrum of the liquid product after reaction of reaction jhbr183

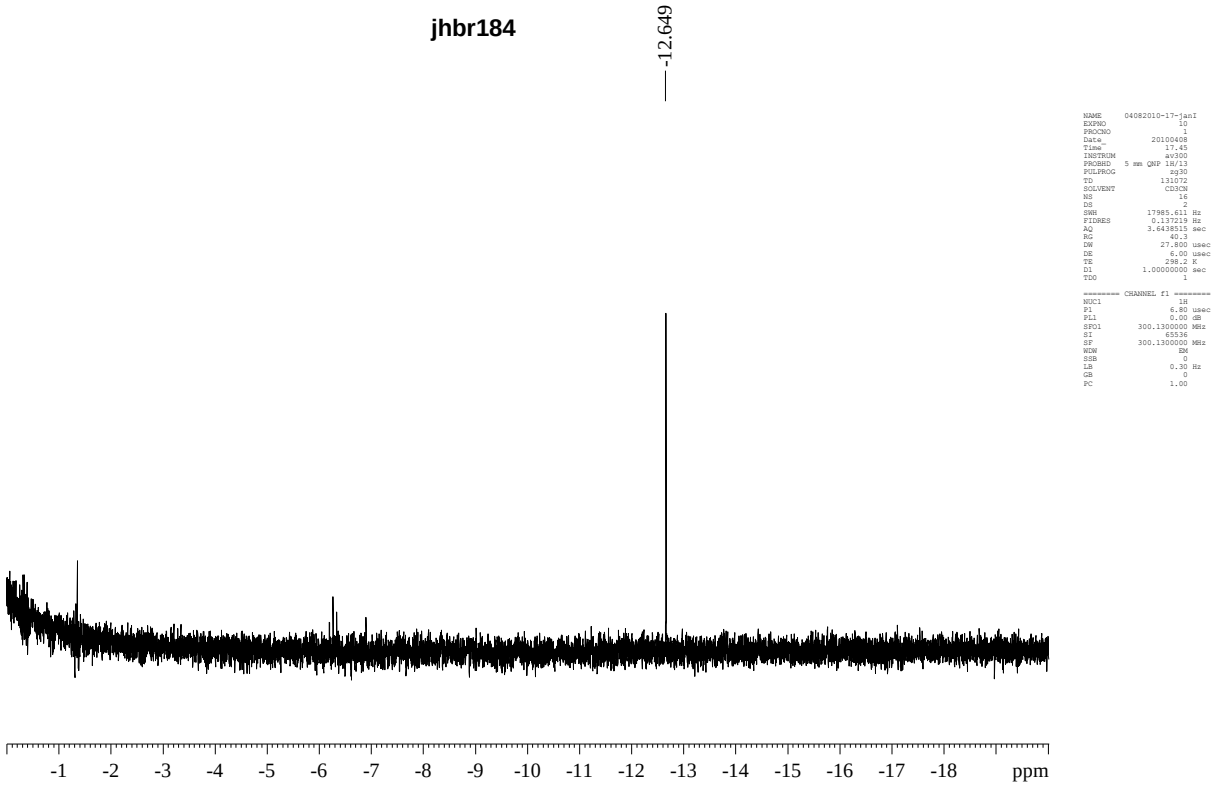
jhbr184



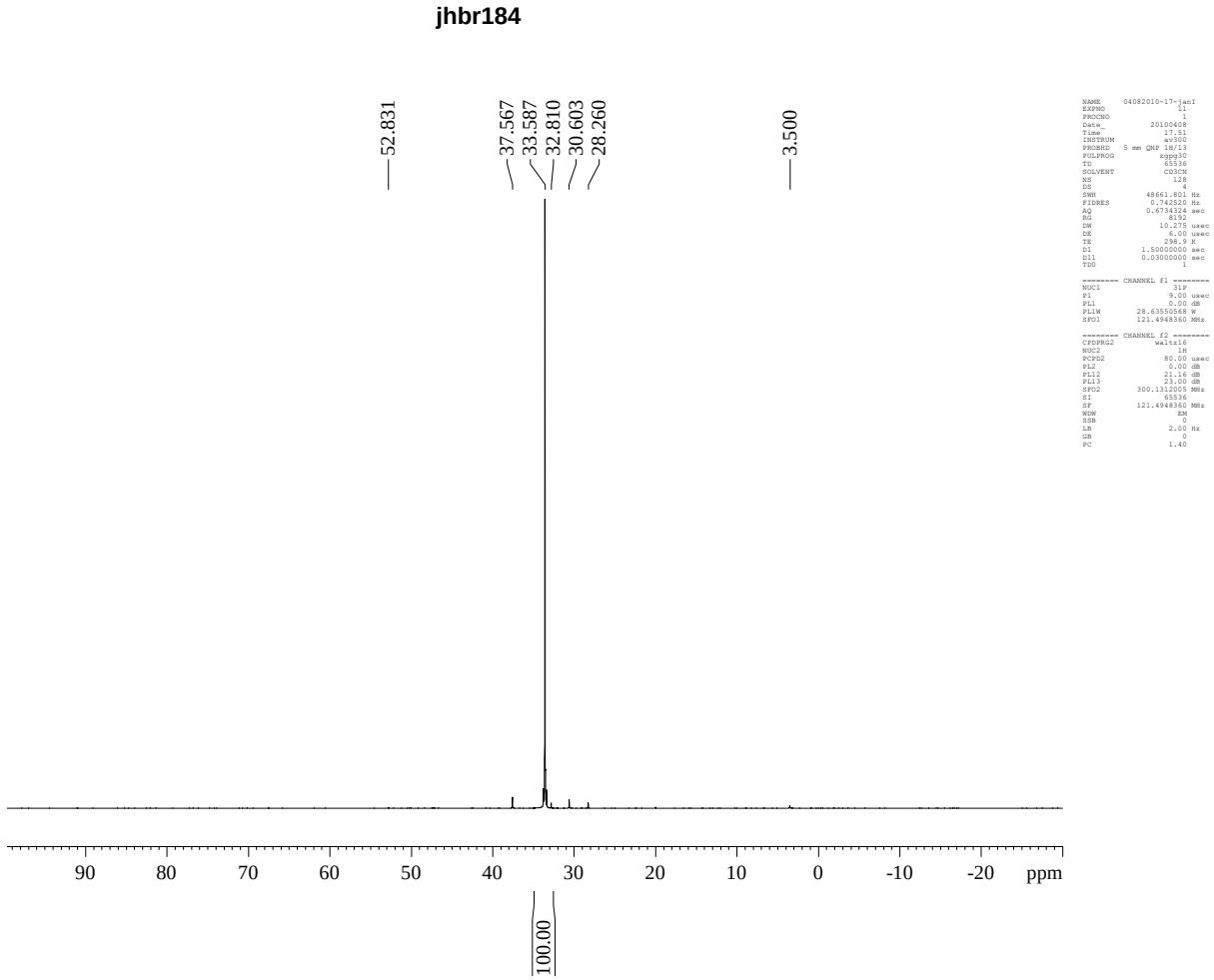
The complete IR spectrum of the liquid product after reaction of reaction jhbr184



The ^{1}H NMR of the liquid product after reaction jhbr184

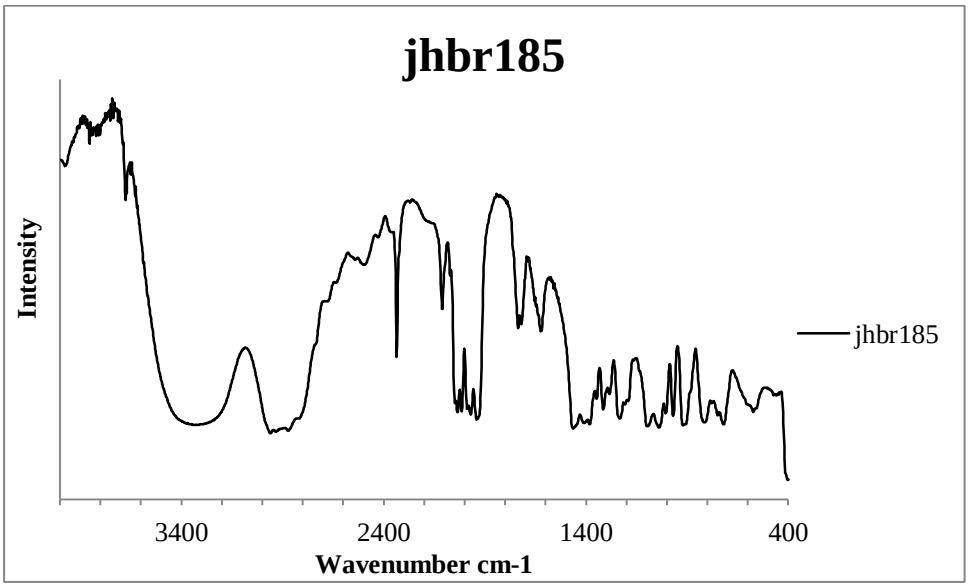


The ^{1}H NMR of the hydride region of the liquid product after reaction jhbr184

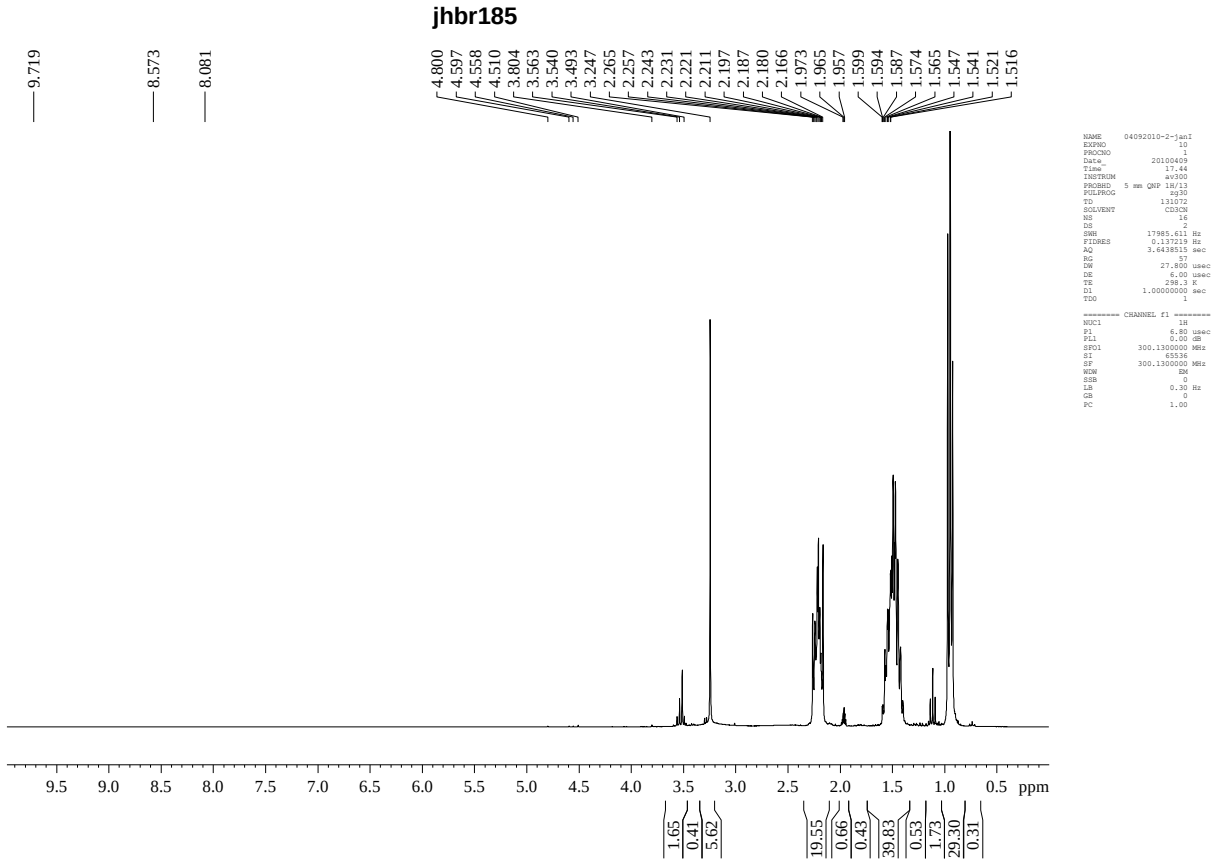


The ^{31}P NMR of the liquid product after reaction jhbr 184

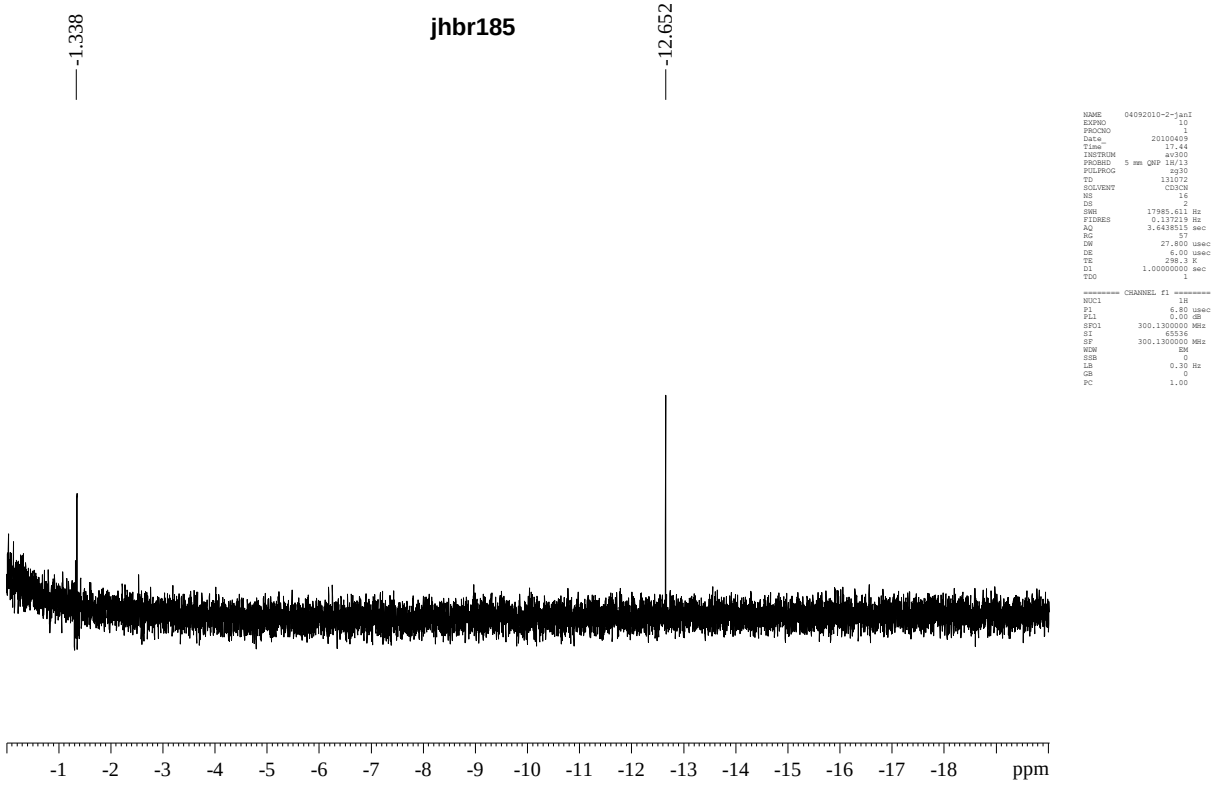
jhbr185



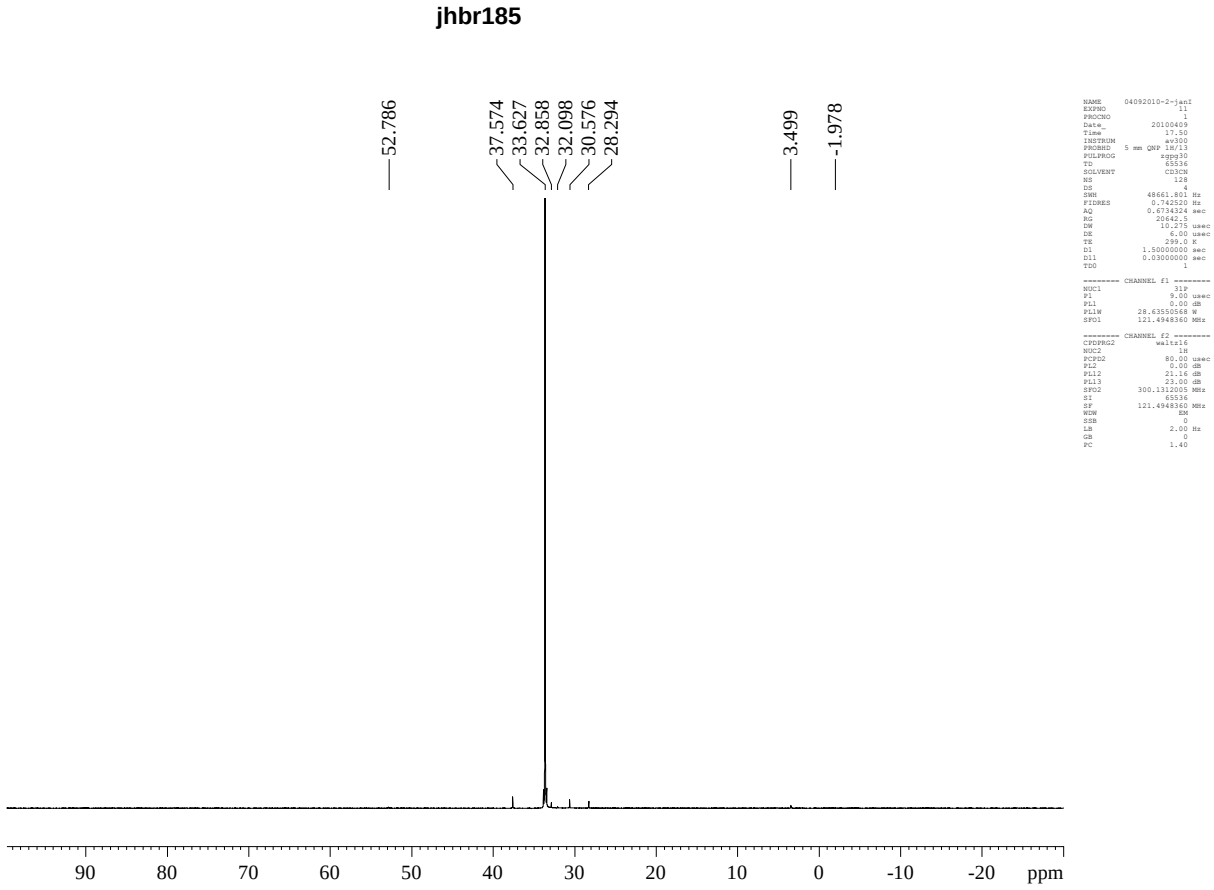
The complete IR spectrum of the liquid product after reaction of reaction jhbr185



The ^{1}H NMR of the liquid product after reaction jhbr185

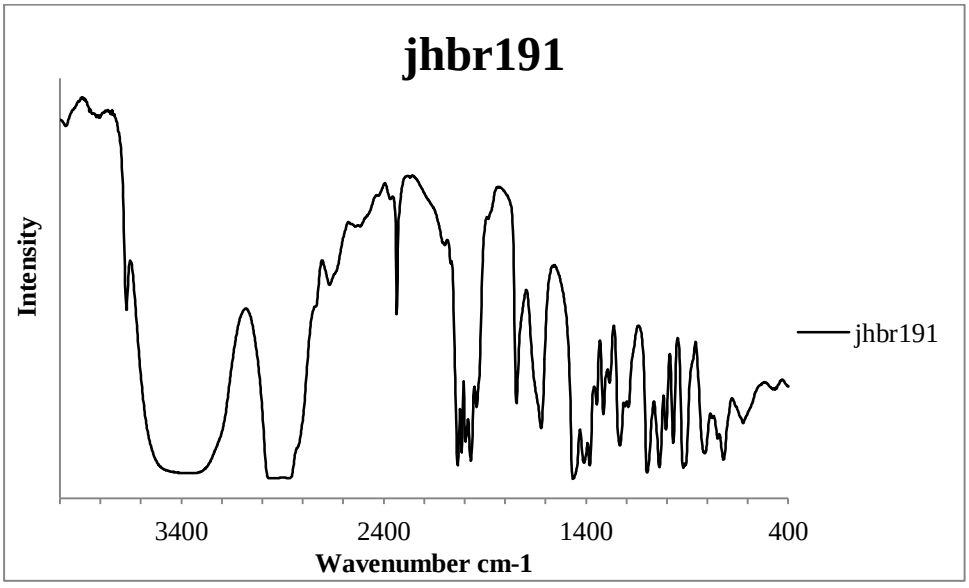


The ^{1}H NMR of the hydride region of the liquid product after reaction jhbr185

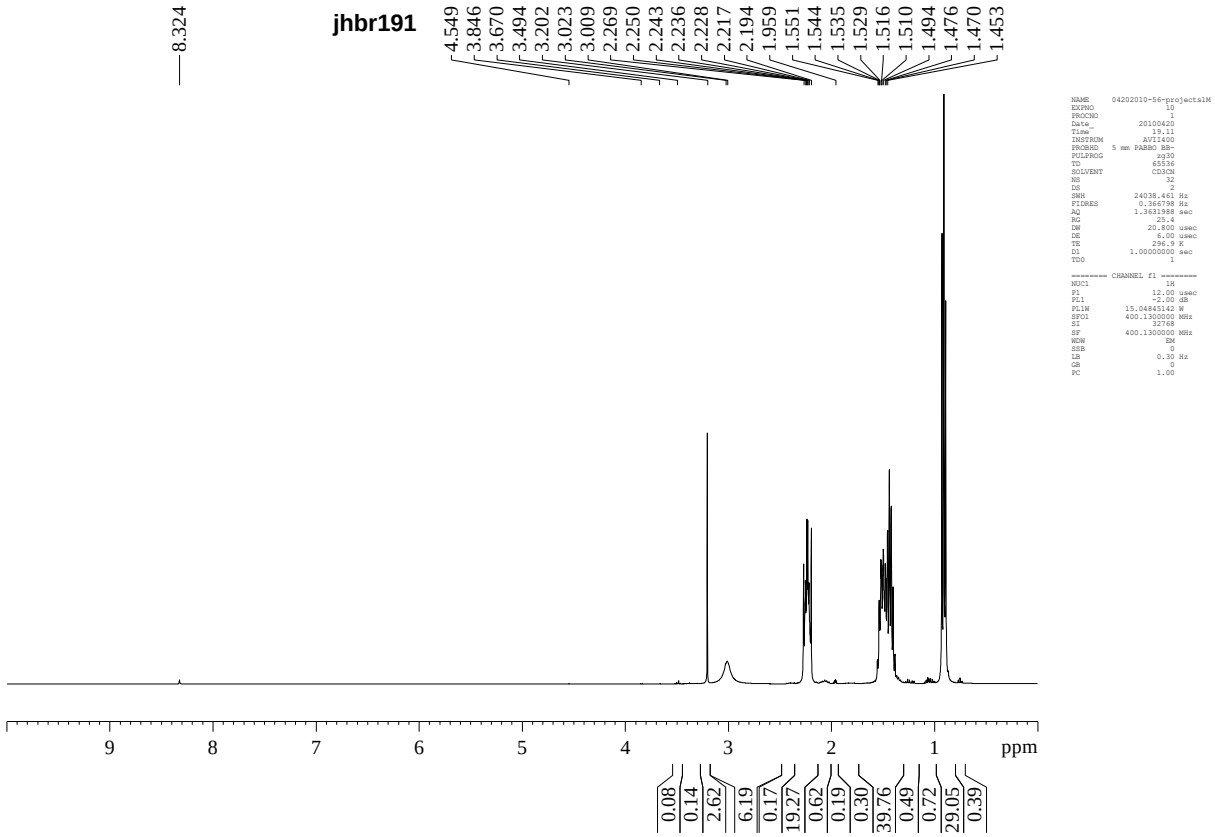


The ^{31}P NMR of the liquid product after reaction jhbr185

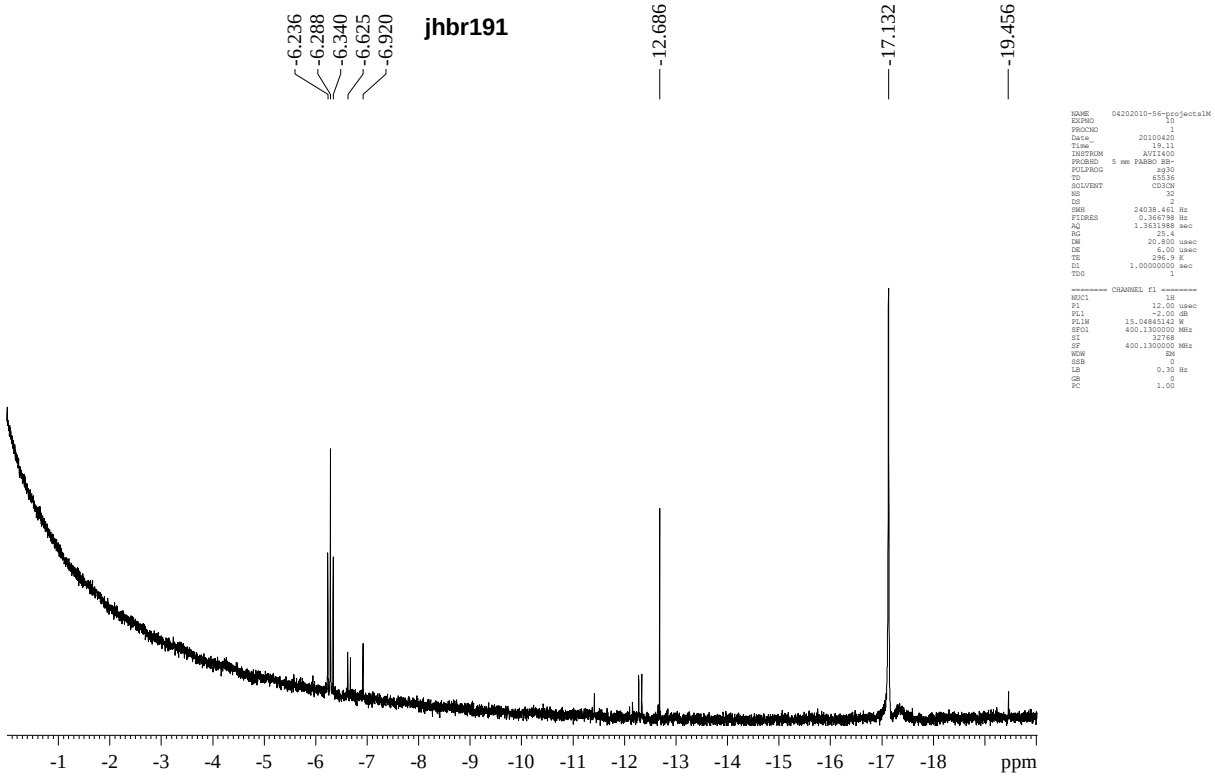
jhbr191



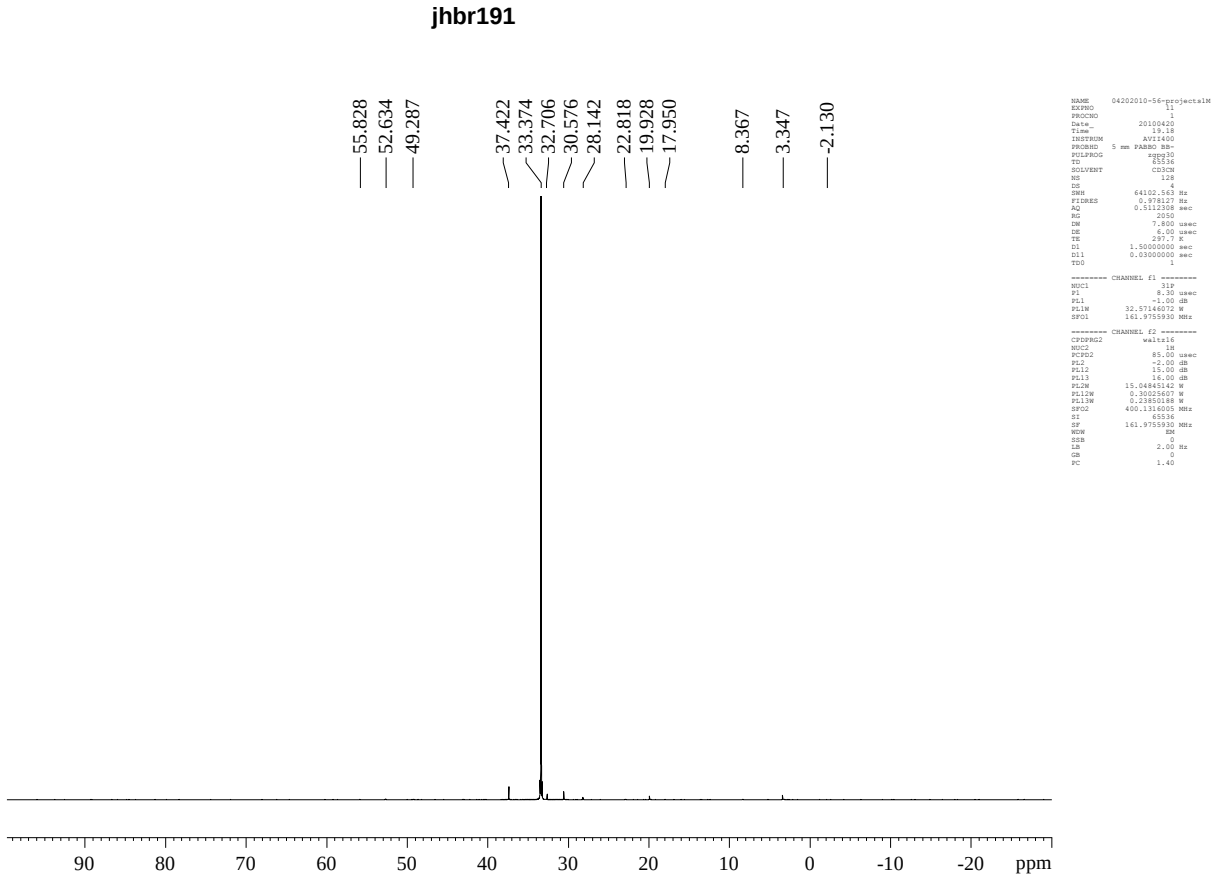
The complete IR spectrum of the liquid product after reaction of reaction jhbr191



The ^{1}H NMR of the liquid product after reaction jhbr191

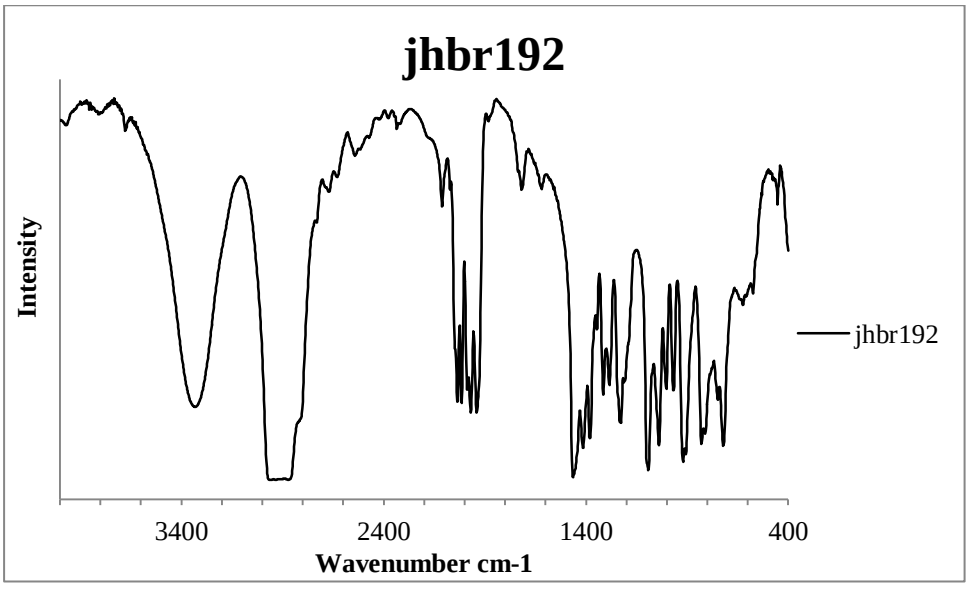


The ^{1}H NMR of the hydride region of the liquid product after reaction jhbr191



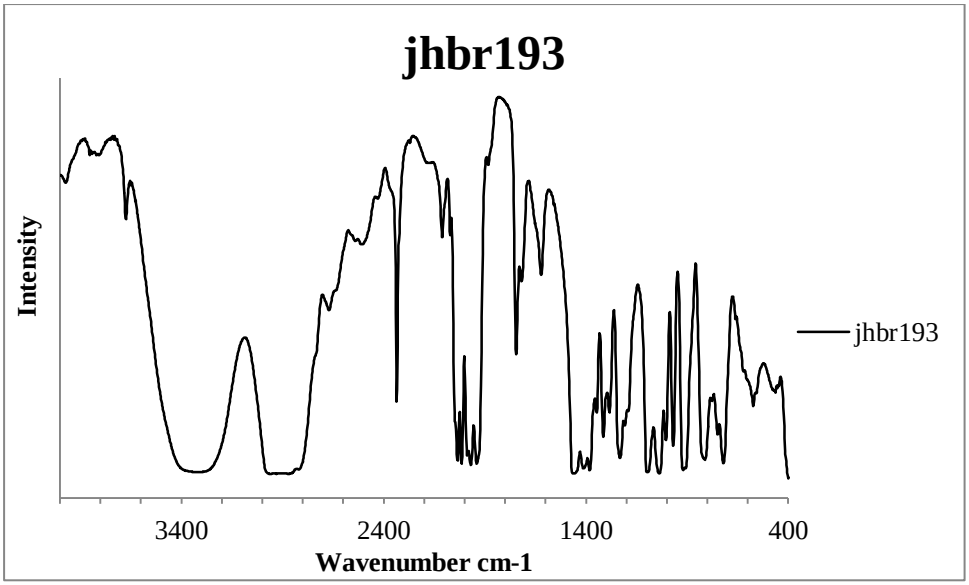
The ^{31}P NMR of the liquid product after reaction jhbr191

jhbr192

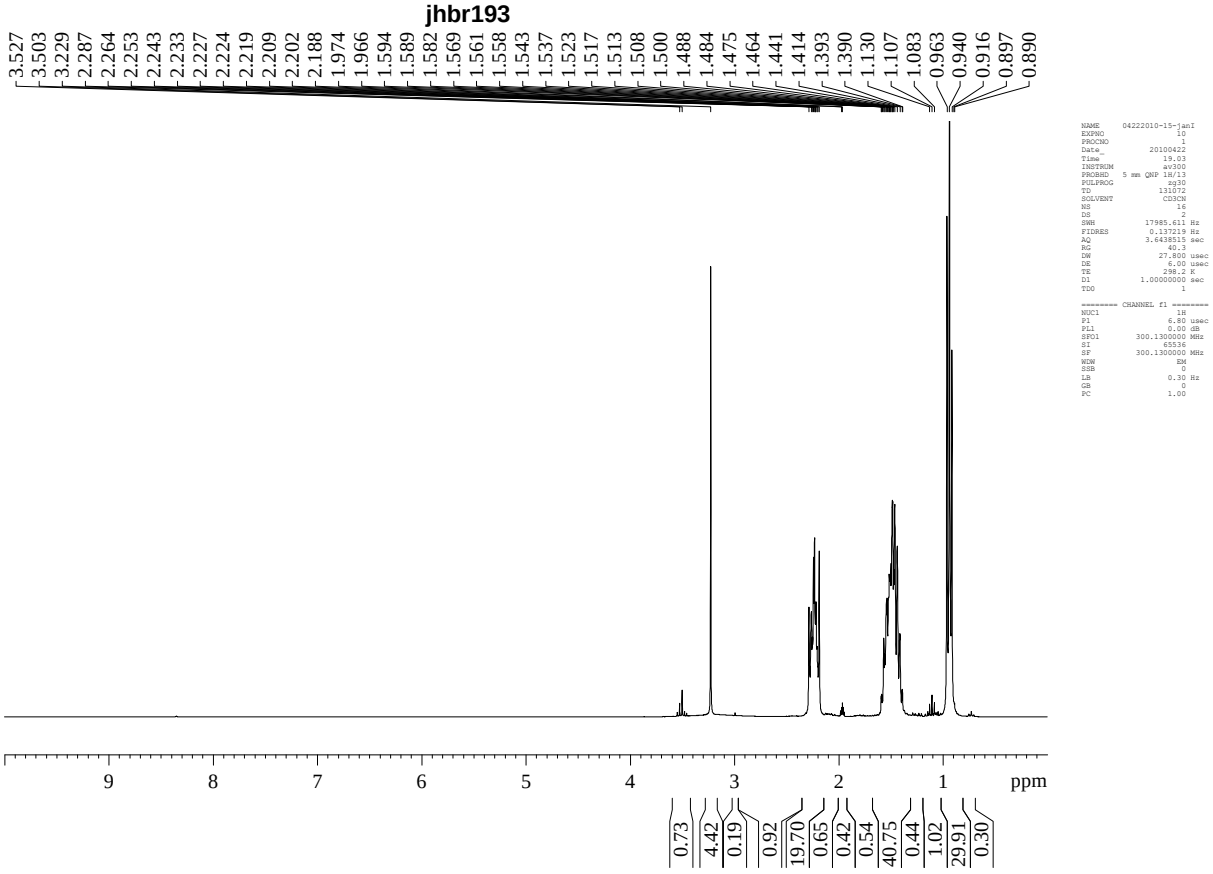


The complete IR spectrum of the liquid product after reaction of reaction jhbr192

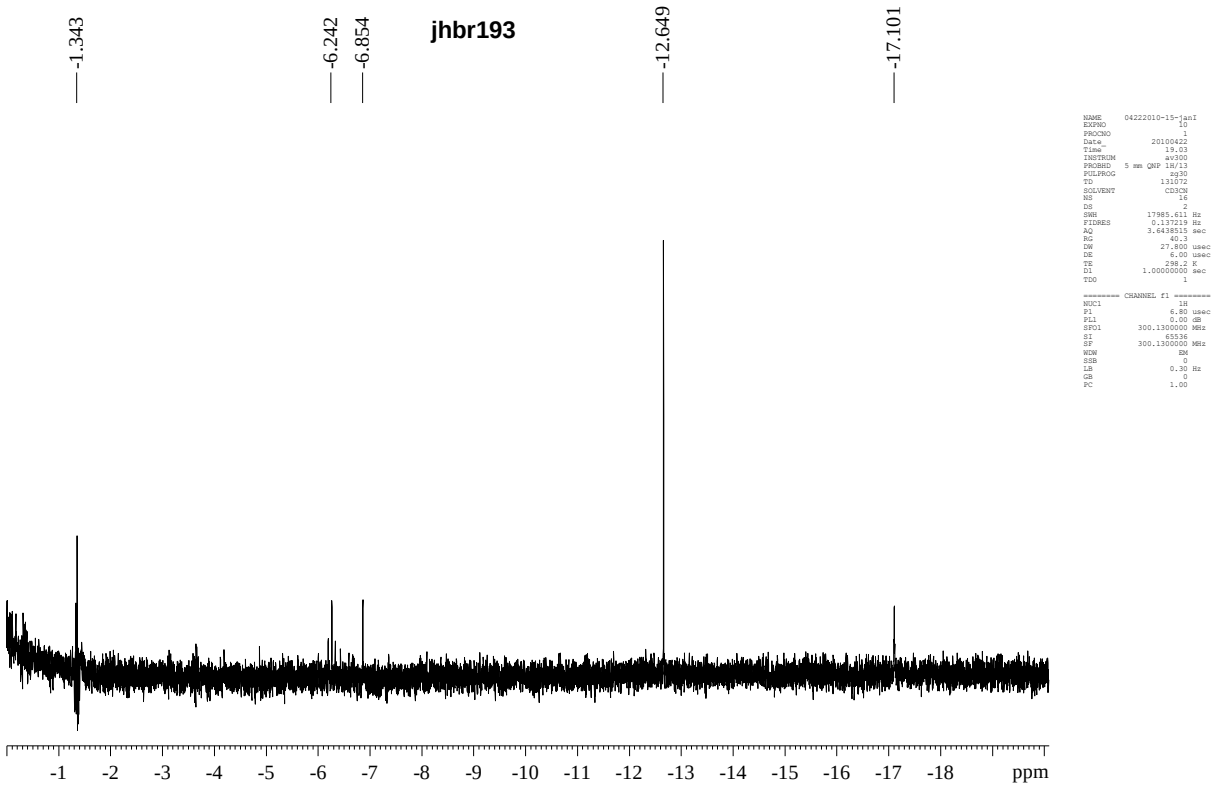
jhbr193



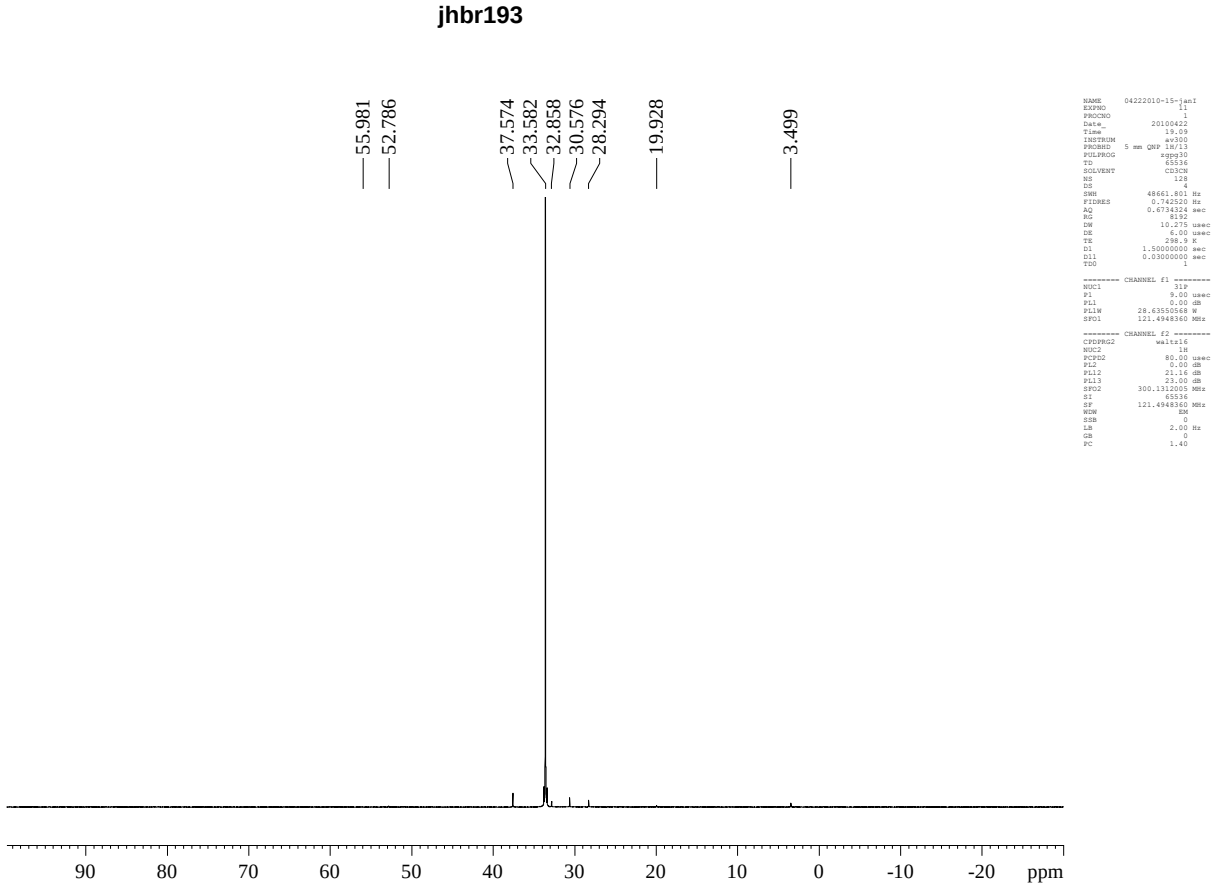
The complete IR spectrum of the liquid product after reaction of reaction jhbr193



The ^{1}H NMR of the liquid product after reaction jhbr193

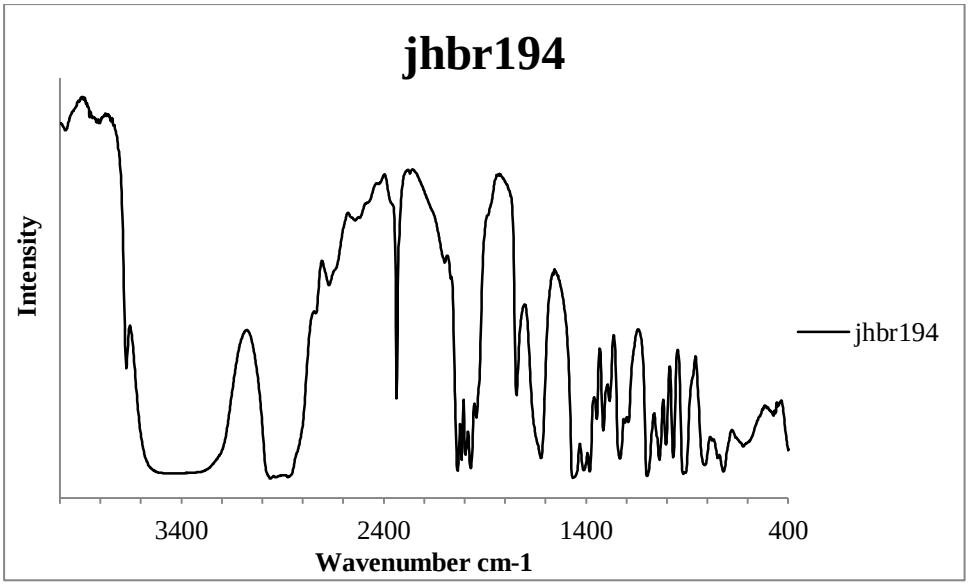


The ^{1}H NMR of the hydride region of the liquid product after reaction jhbr193

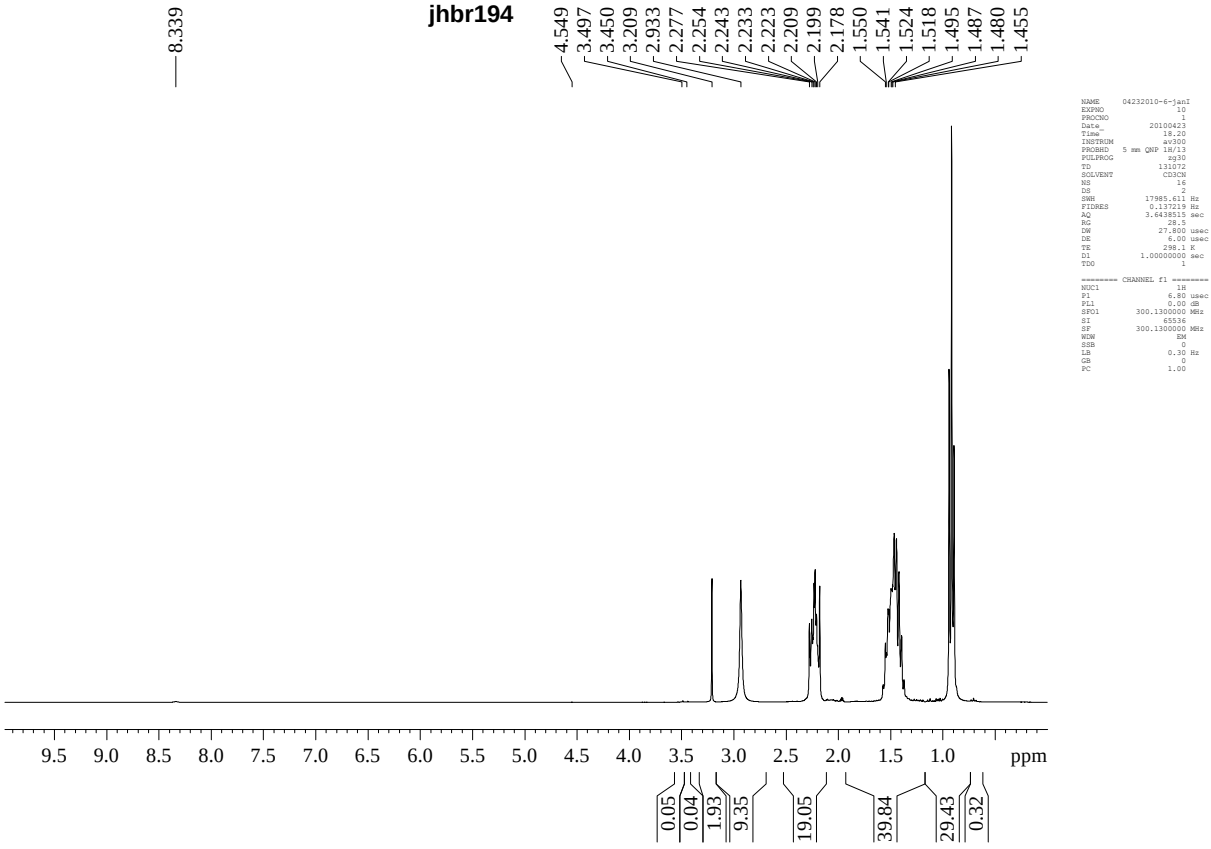


The ^{31}P NMR of the liquid product after reaction jhbr193

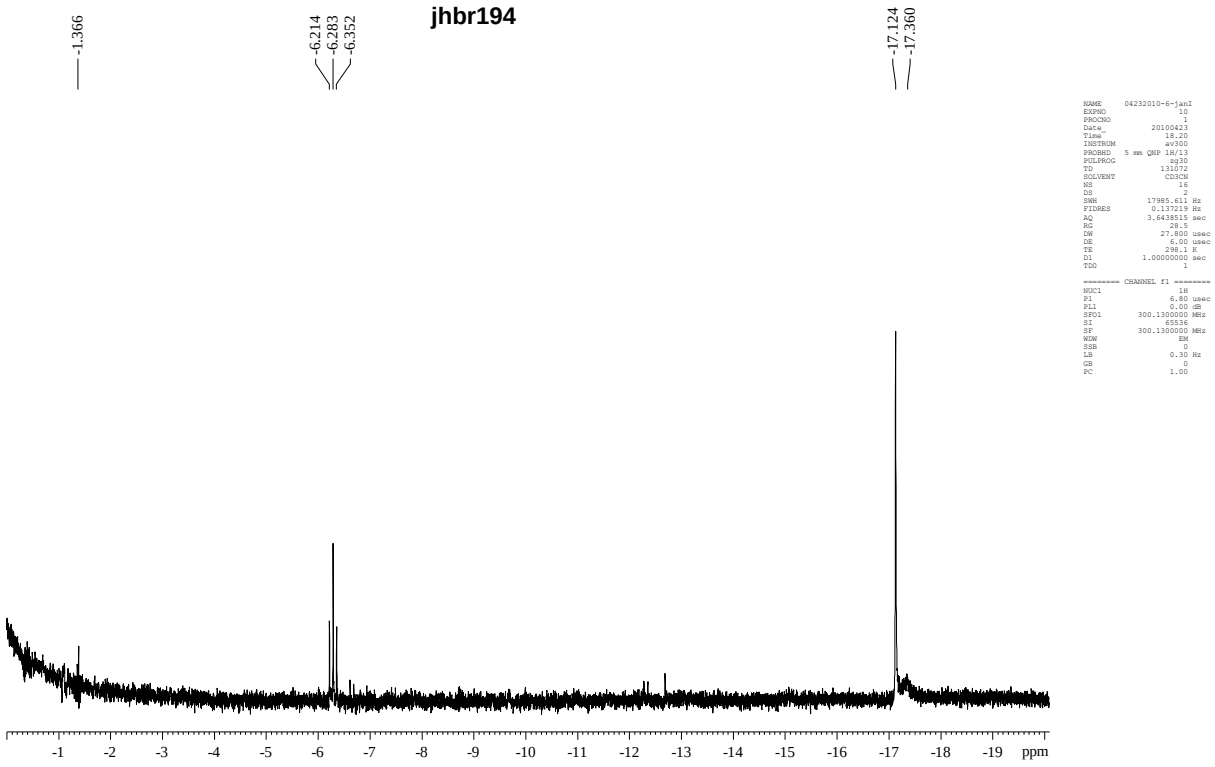
jhbr194



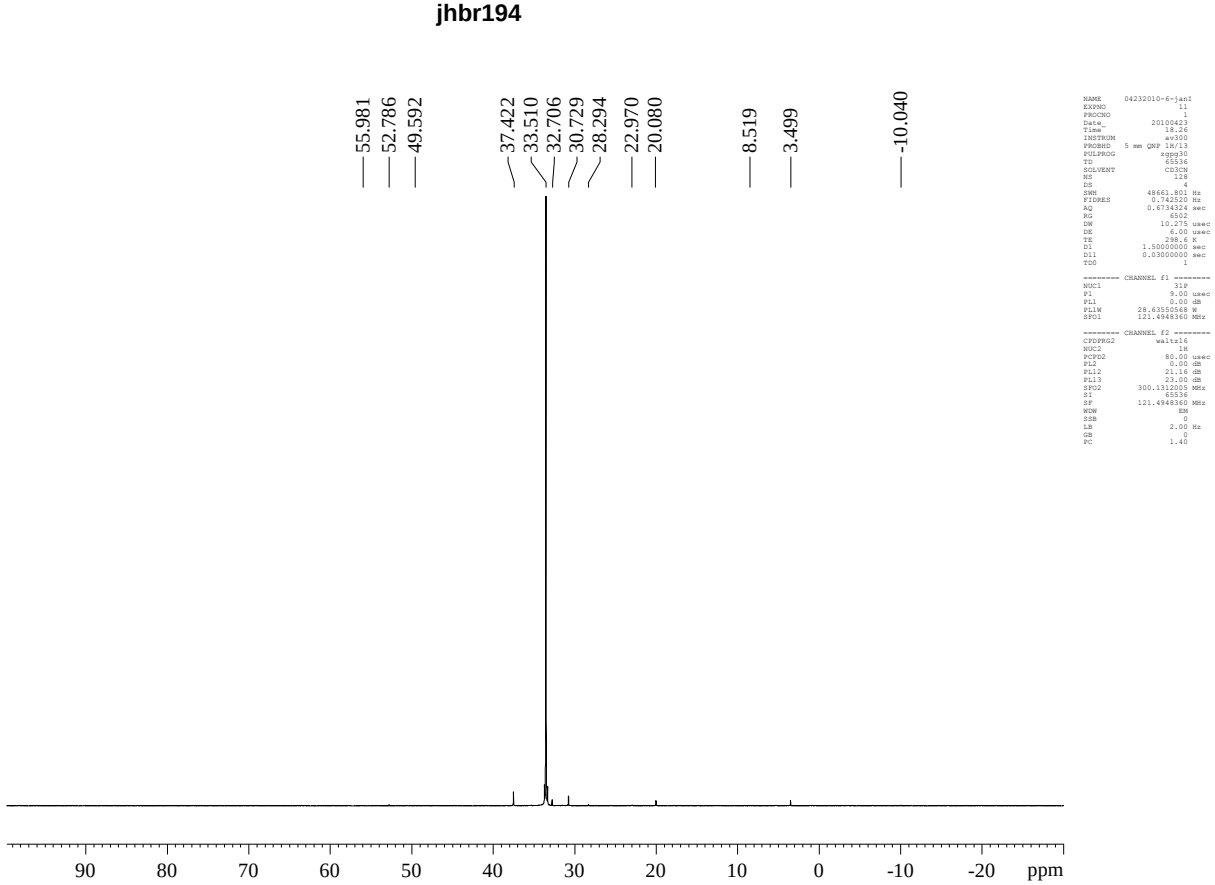
The complete IR spectrum of the liquid product after reaction of reaction jhbr194



The ^{1}H NMR of the liquid product after reaction jhbr194

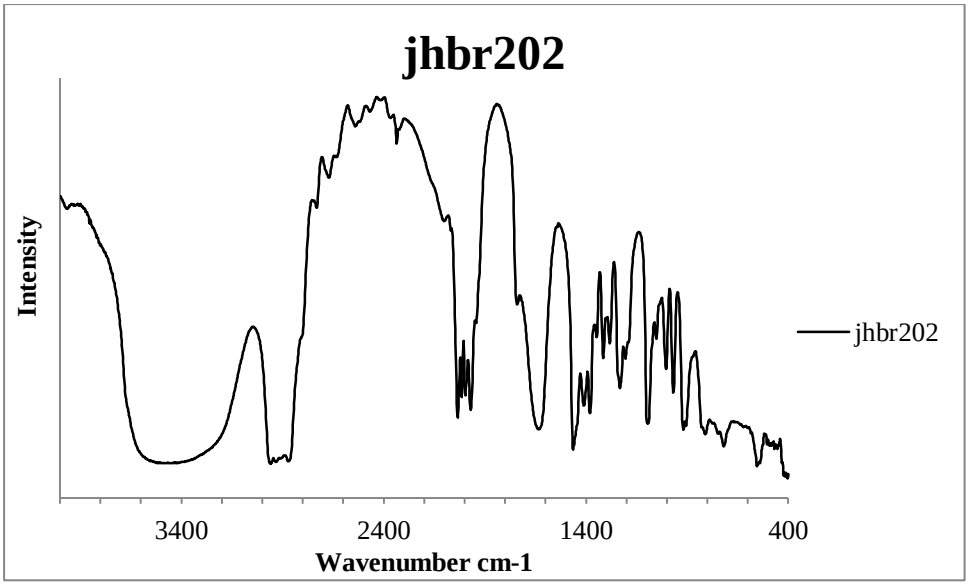


The ^{1}H NMR of the hydride region of the liquid product after reaction jhbr194

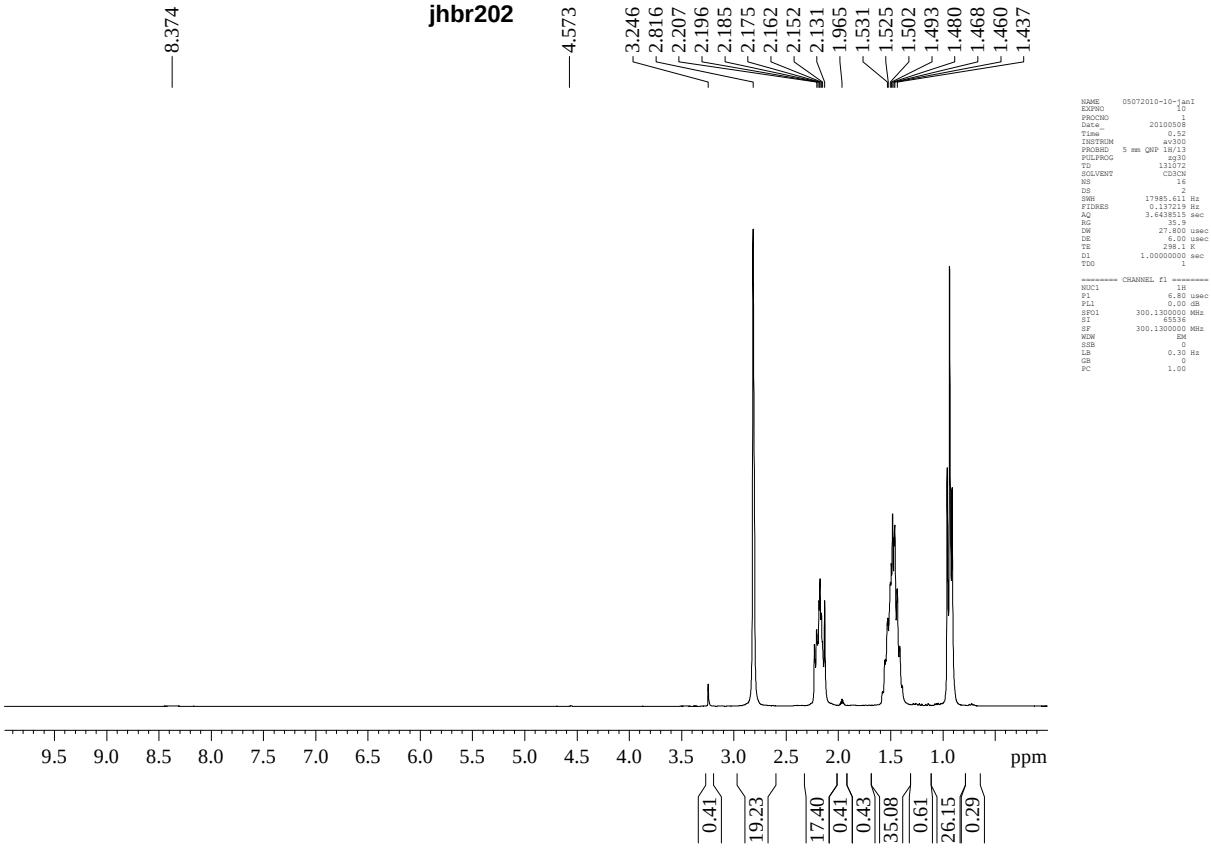


The ^{31}P NMR of the liquid product after reaction jhbr194

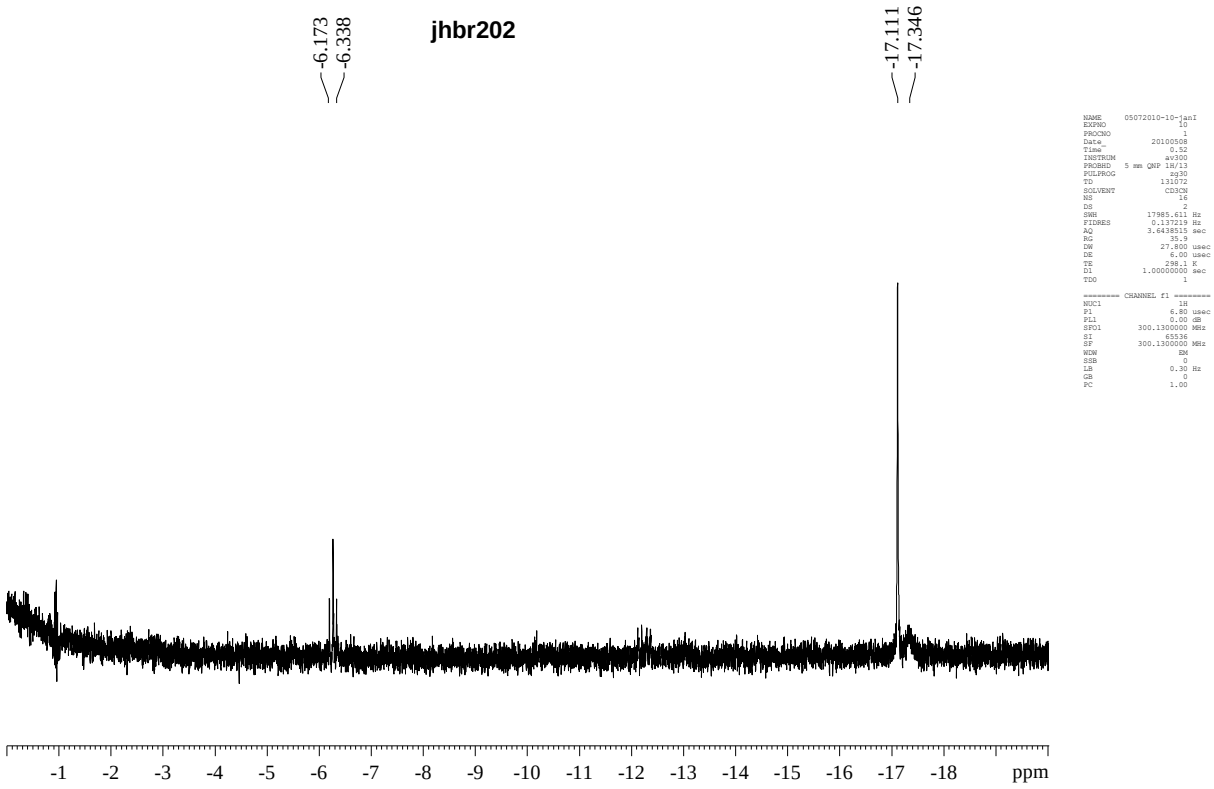
jhbr202



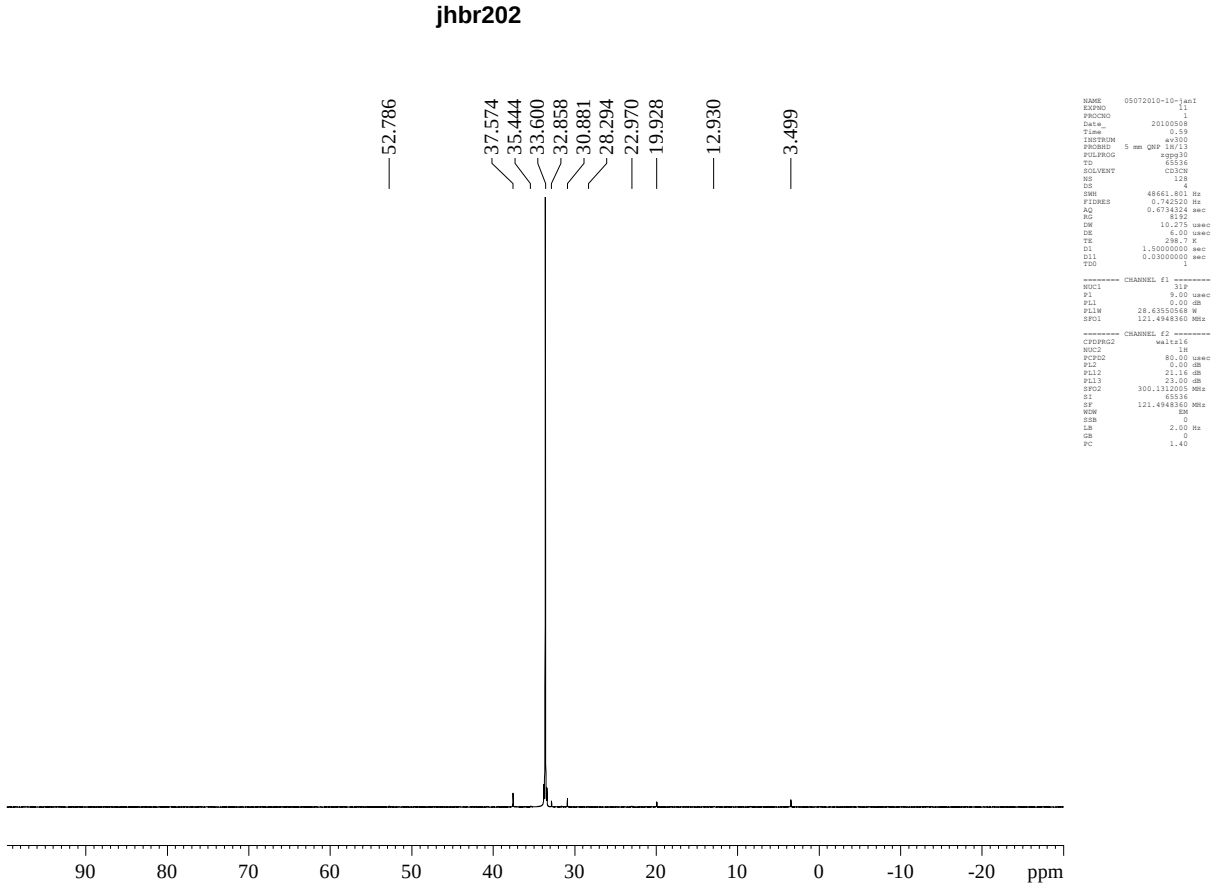
The complete IR spectrum of the liquid product after reaction of reaction jhbr202



The ^{1}H NMR of the liquid product after reaction jhbr202

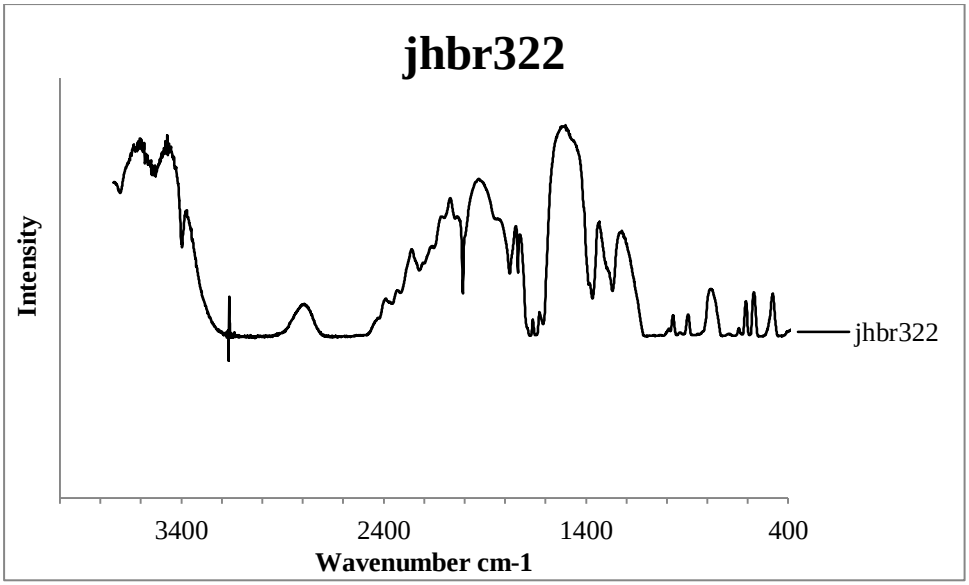


The ^{1}H NMR of the hydride region of the liquid product after reaction jhbr202



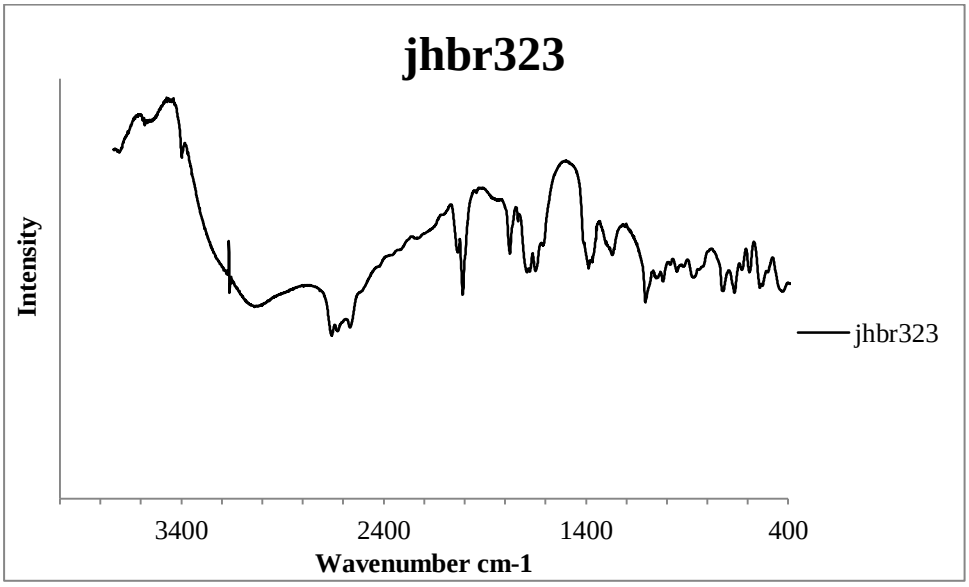
The ^{31}P NMR of the liquid product after reaction jhbr202

jhbr322



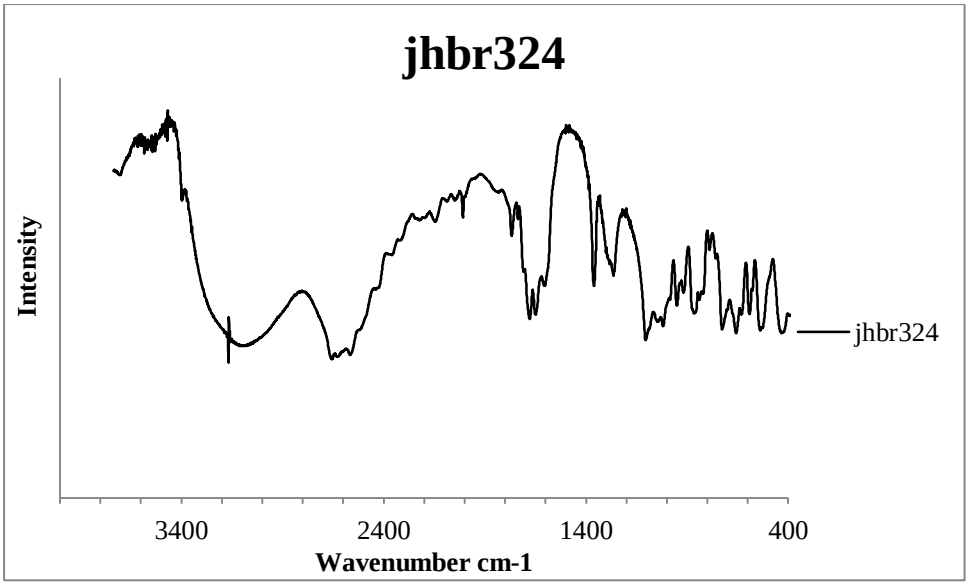
The complete IR spectrum of the liquid product after reaction of reaction jhbr322

jhbr323



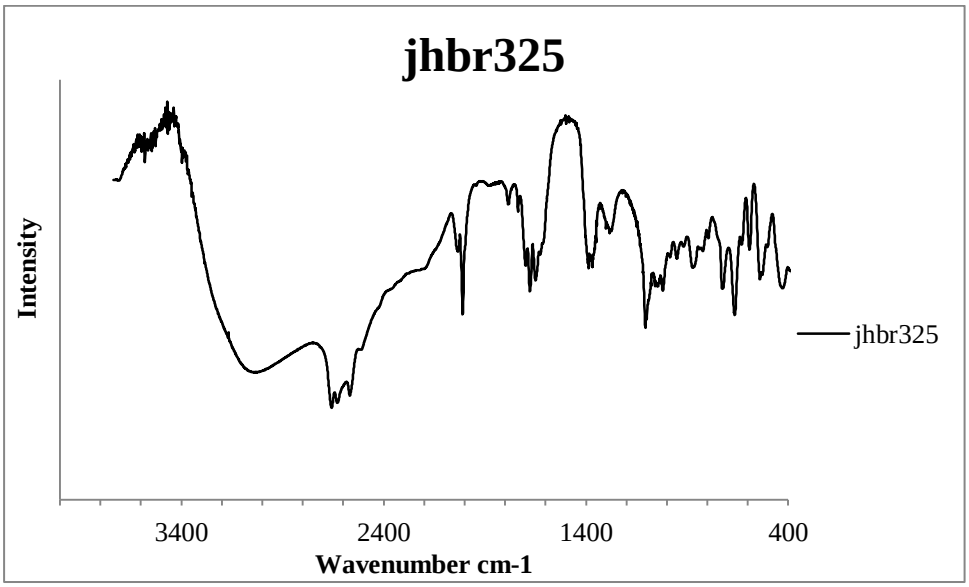
The complete IR spectrum of the liquid product after reaction of reaction jhbr323

jhbr324



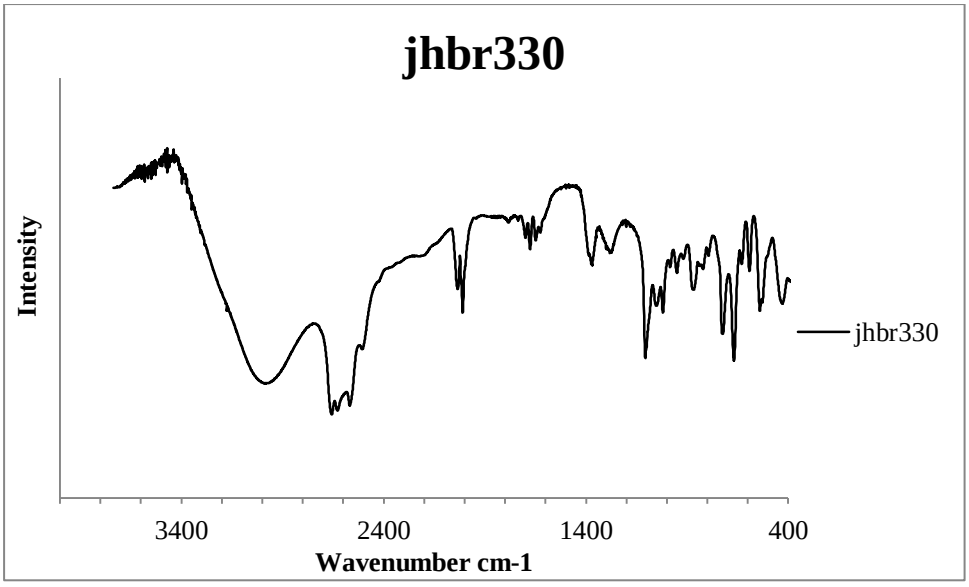
The complete IR spectrum of the liquid product after reaction of reaction jhbr324

jhbr325



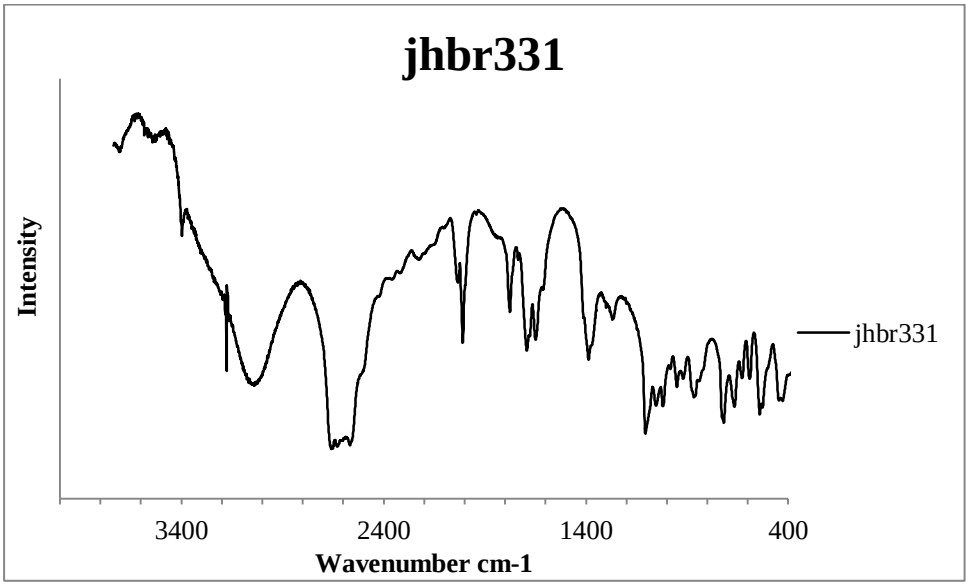
The complete IR spectrum of the liquid product after reaction of reaction jhbr325

jhbr330



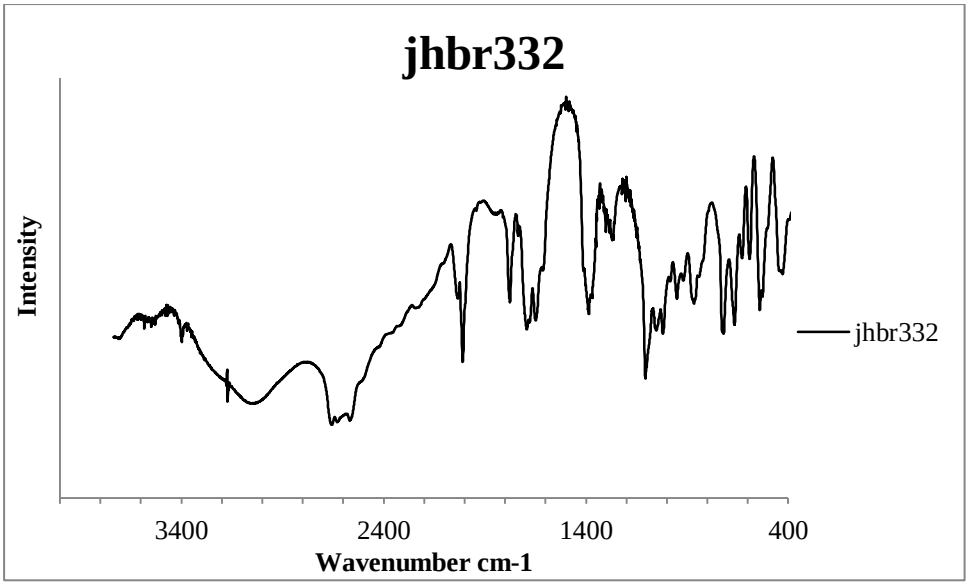
The complete IR spectrum of the liquid product after reaction of reaction jhbr330

jhbr331



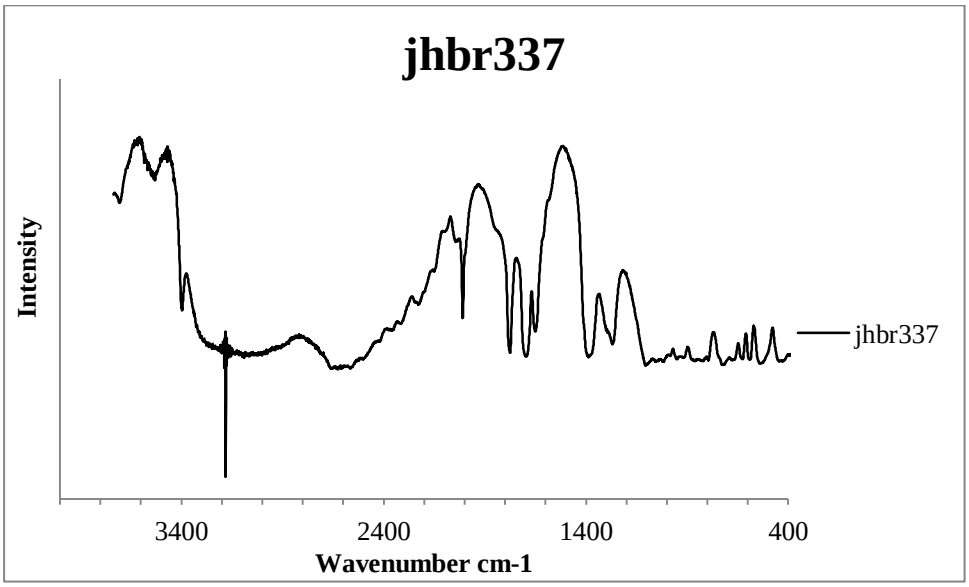
The complete IR spectrum of the liquid product after reaction of reaction jhbr331

jhbr332



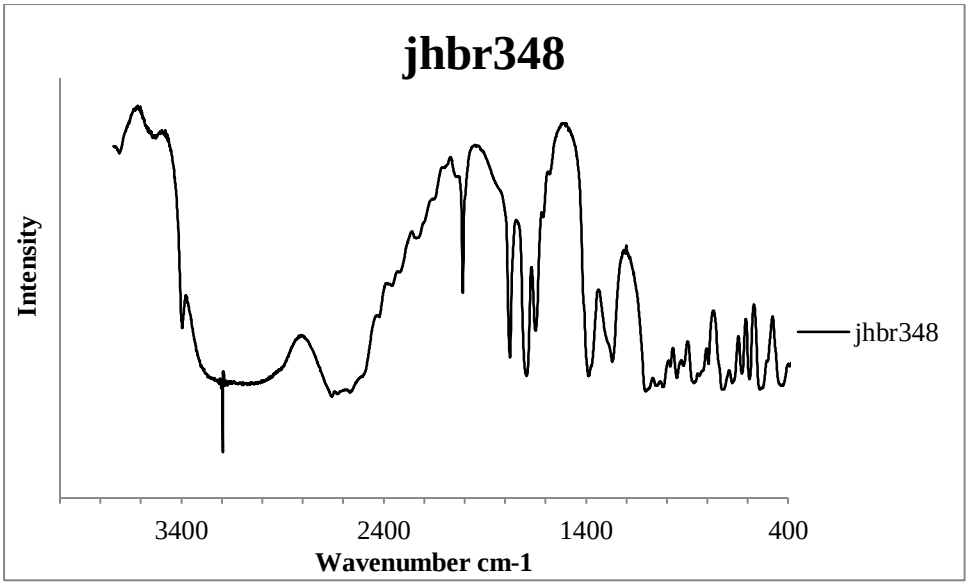
The complete IR spectrum of the liquid product after reaction of reaction jhbr332

jhbr337



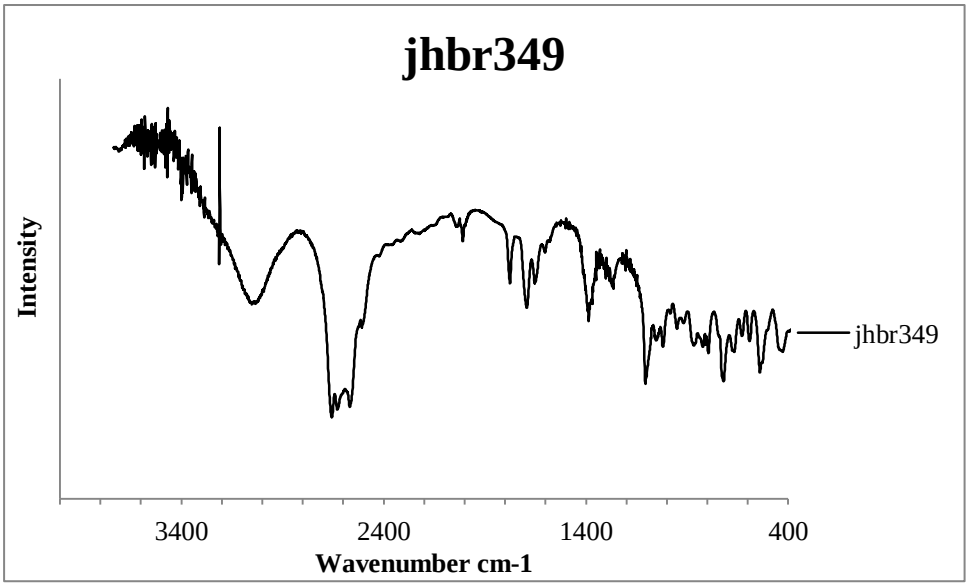
The complete IR spectrum of the liquid product after reaction of reaction jhbr337

jhbr348



The complete IR spectrum of the liquid product after reaction of reaction jhbr248

jhbr349



The complete IR spectrum of the liquid product after reaction of reaction jhbr349

References

1. H. E. Gottlieb, V. Kotlyar and A. Nudelman, *The Journal of Organic Chemistry*, 1997, **62**, 7512-7515.