Lewis Acid-Promoted Oxidative Addition of Thioimidates to Pd(0)

Indranath Ghosh and Peter A. Jacobi*

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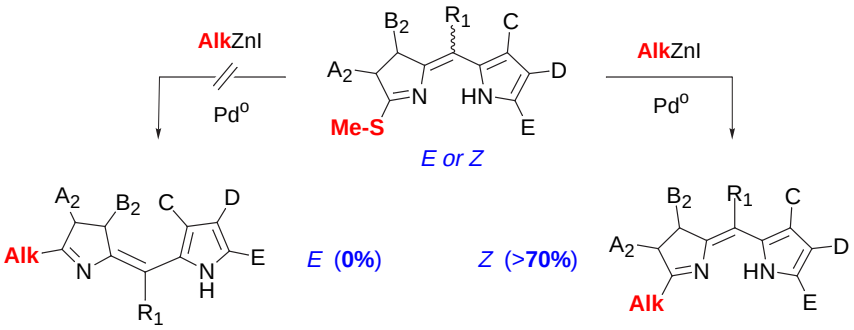
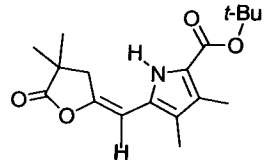
Supporting Information



STANDARD 1H OBSERVE

expi stdih

SAMPLE		DEC. & VT	
date	Nov 15 2001	dfrq	299.961
solvent	CDCl3	dn	H1
file	exp	dpwr	26
ACQUISITION		dof	0
sfrq	299.961	dm	nnn
tn	H1	dmm	c
at	2.504	dmf	200
np	20032	homo	n
sw	4000.0	dseq	
fb	2200	dres	1.0
bs	16	PROCESSING	
tpwr	59	wtfile	
pw	8.7	proc	ft
d1	1.000	fn	not used
tof	0	meth	f
nt	8		
ct	8	werr	
alock	n	wexp	wft
gain	not used	wbs	wft
FLAGS		wnt	
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-508.9		
wp	4000.0		
vs	377		
sc	0		
wc	250		
hzmm	16.00		
is	1113.75		
rfl	2689.6		
rfp	2180.7		
th	20		
ins	100.000		
nm	ph		

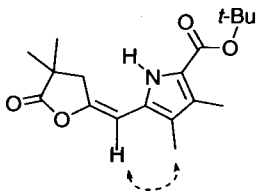


6b-*E*

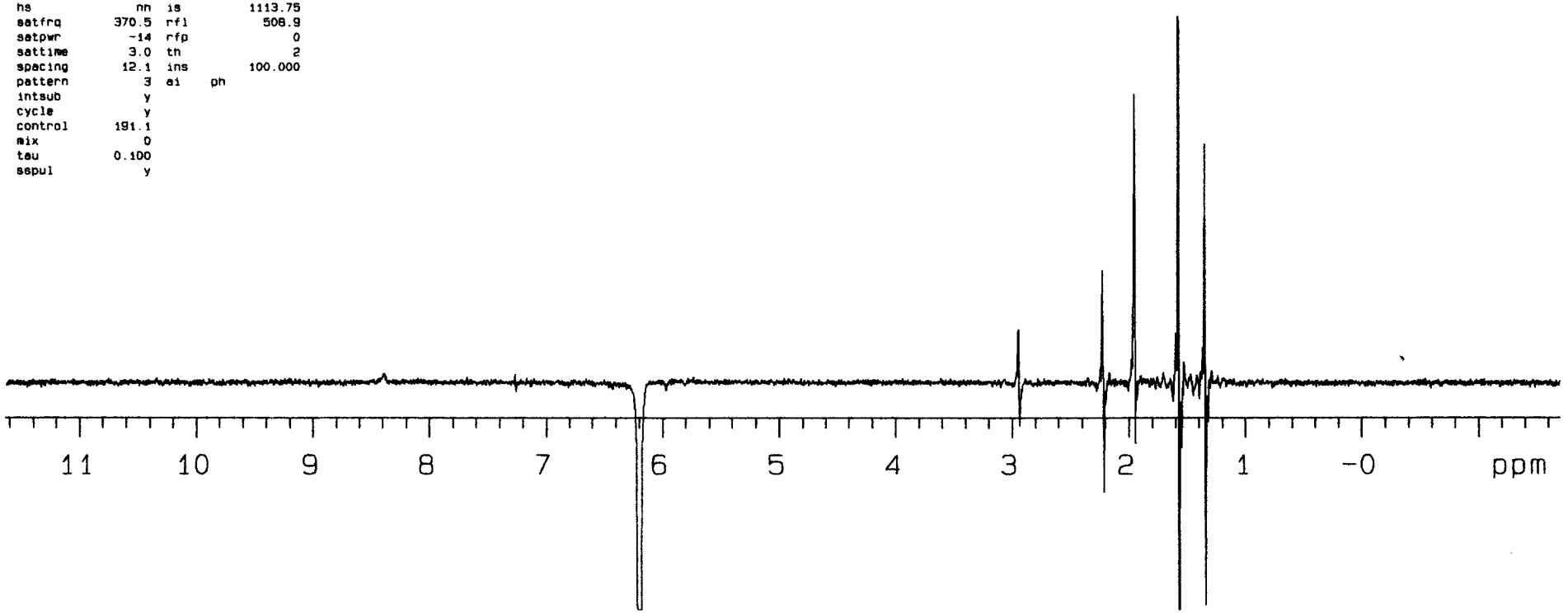
STANDARD 1H OBSERVE

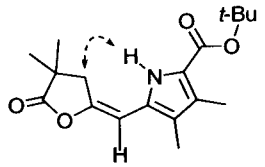
expl cyclenoe

SAMPLE		DEC. & VT	
date	Nov 15 2001	dn	H1
solvent	CDCl3	dof	0
file	exp	dm	nnn
ACQUISITION		dmm	c
sfrq	299.961	dpwr	26
tn	H1	dpwrf	undefined
at	2.504	homo	n
np	20032	PROCESSING	
sw	4000.0	lb	0.40
fb	2200	fn	not used
bs	32	math	f
ss	8		
pw	13.0	werr	ADC_test
d1	2.000	wexp	wft
tof	0	wbs	wft
nt	1024	wnt	
ct	192	DISPLAY	
alock	n	sp	-508.9
gain	48	wp	4000.0
FLAGS		vs	7647
il	n	sc	0
in	n	wc	250
dp	y	hzmm	16.00
hs	nn	is	1113.75
satfrq	370.5	rfl	508.9
satpwr	-14	rfp	0
sattime	3.0	th	2
spacing	12.1	ins	100.000
pattern	3	ai ph	
intsub	y		
cycle	y		
control	191.1		
mix	0		
tau	0.100		
sspul	y		



6b-*E*



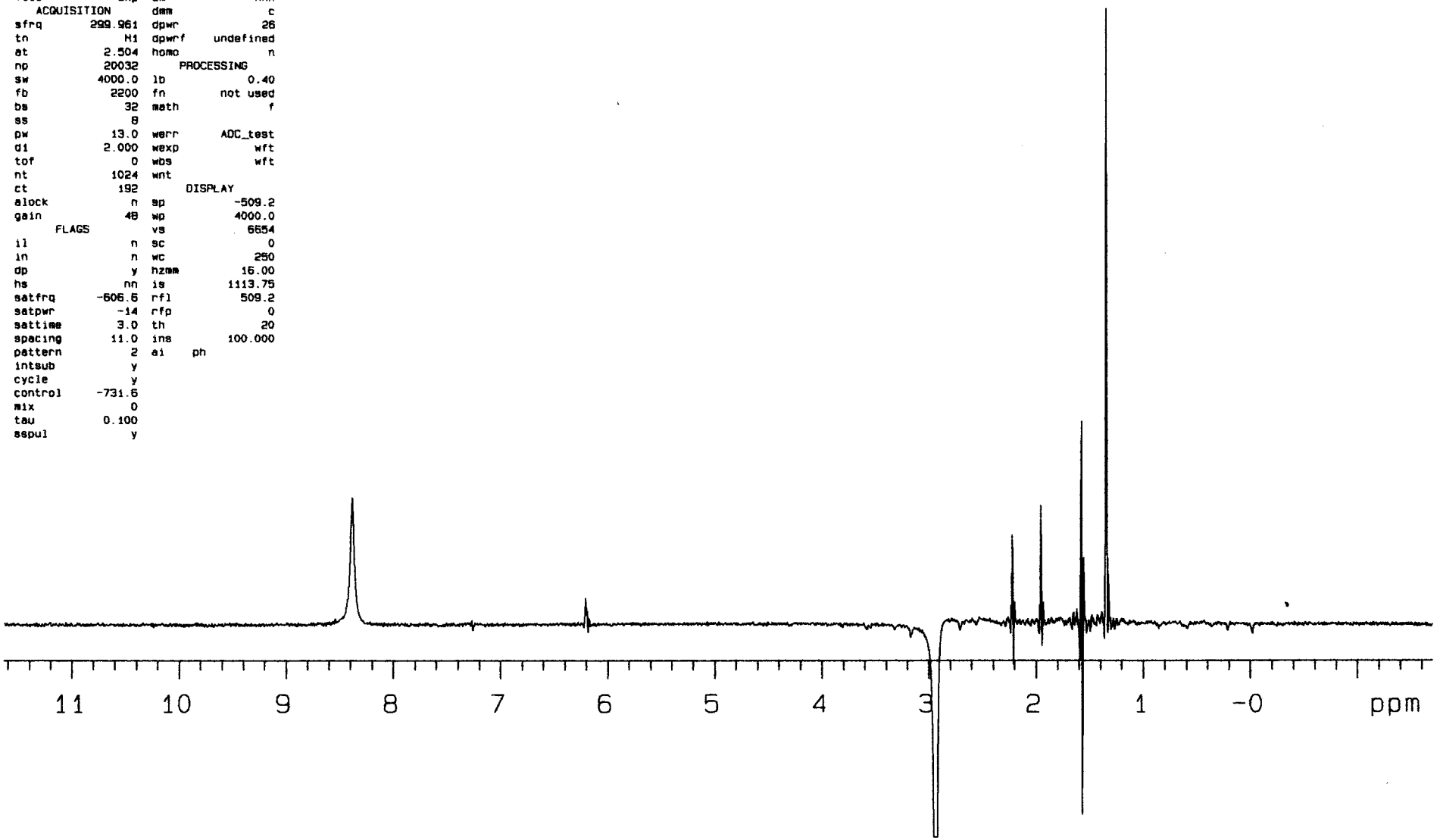


6b-*E*

STANDARD 1H OBSERVE

expi cyclenoe

SAMPLE		DEC. & VT	
date	Nov 15 2001	dn	H1
solvent	CDCl3	dof	0
file	exp	dm	nnn
ACQUISITION		dmm	c
sfrq	299.961	dpwr	26
tn	H1	dpwrf	undefined
at	2.504	homo	n
np	20032	PROCESSING	
sw	4000.0	lb	0.40
fb	2200	fn	not used
bs	32	math	f
ss	8		
pw	13.0	werr	ADC_test
d1	2.000	wexp	wft
tof	0	wbs	wft
nt	1024	wnt	
ct	192	DISPLAY	
alock	n	sp	-509.2
gain	48	wp	4000.0
FLAGS		vs	6654
il	n	sc	0
in	n	wc	250
dp	y	hzmm	16.00
hs	nn	is	1113.75
satfrq	-606.6	rfl	509.2
satpwr	-14	rfp	0
sattime	3.0	th	20
spacing	11.0	ins	100.000
pattern	2	ai ph	
intsub	y		
cycle	y		
control	-731.6		
mix	0		
tau	0.100		
sspul	y		

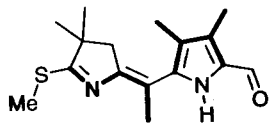


STANDARD PROTON PARAMETERS

exp1 s2pul

SAMPLE		DEC. & VT	
date	Feb 26 2002	dfrq	499.875
solvent	CDCl3	dn	H1
file	exp	dpwr	0
ACQUISITION		dof	0
sfrq	499.875	dm	nnn
tn	H1	dmm	c
at	2.997	dmf	200
np	47936	homo	n
sw	7998.4	dseq	
fb	4400	dres	1.0
bs	8	temp	21.0
tpwr	59	DEC2	
pw	7.7	dfrq2	0
d1	1.000	dn2	
tof	513.1	dpwr2	1
nt	32	dof2	0
ct	32	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	200
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	PROCESSING	
hs	nn	lb	0.05
DISPLAY		wtfile	
sp	-19.6	proc	ft
wp	5026.7	fn	not used
vs	148	math	f
sc	0		
wc	240	werr	
hzmm	20.94	wexp	wft
is	7242.74	wbs	wft
rfl	4625.4	wnt	
rfp	3634.1		
th	7		
ins	1.000		

nm cdc ph

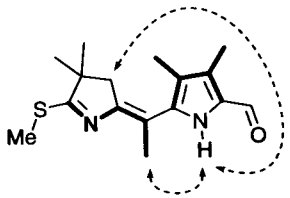


9a-*E*

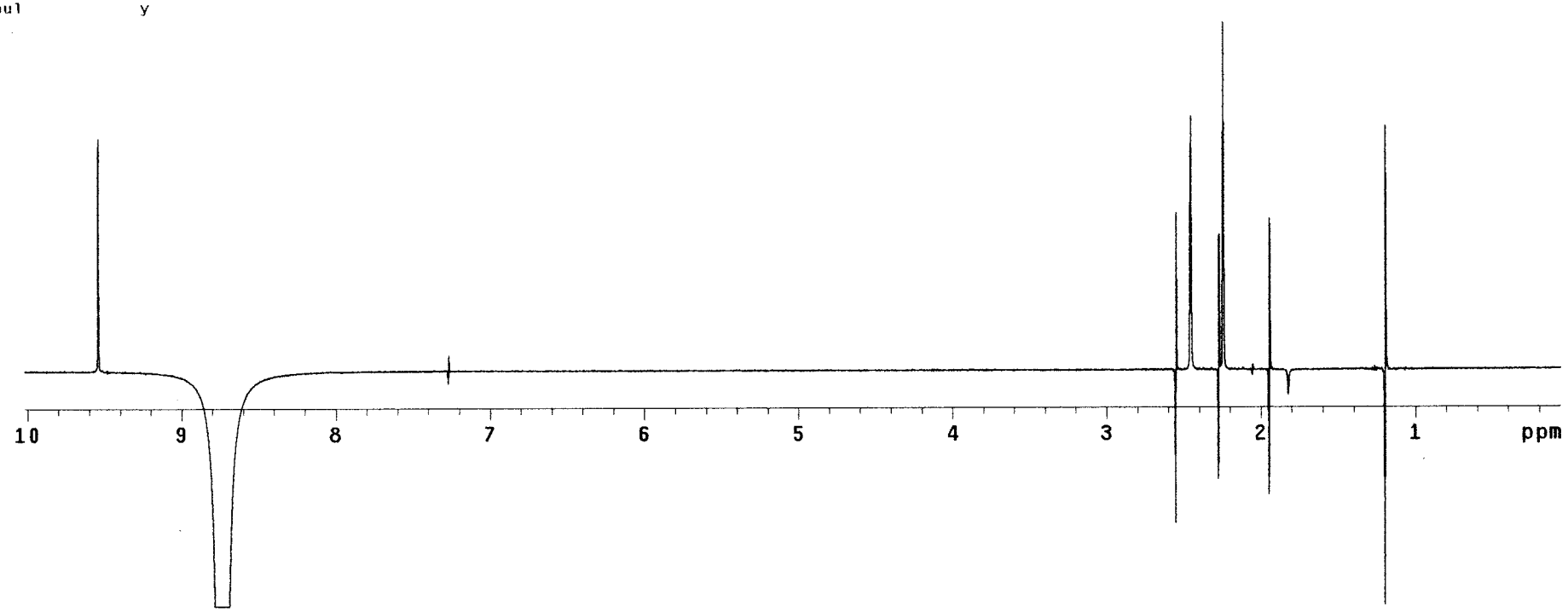
STANDARD PROTON PARAMETERS

exp5 cyclenoe

SAMPLE		DEC. & VT	
date	Feb 26 2002	dn	H1
solvent	CDCl3	dof	0
file	/data/guest/I~	dm	nnn
G500/NOE-NH-E-SMeF~		dmm	c
	ormyl.fid	dpwr	0
ACQUISITION		dpwrf	undefined
sfrq	499.875	homo	n
tn	H1	temp	21.0
at	2.997	PROCESSING	
np	47936	lb	0.33
sw	7998.4	fn	not used
fb	4400	math	f
bs	32		
ss	8	werr	ADC_test
pw	11.6	wexp	wft
d1	2.000	wbs	wft
tof	513.1	wnt	
nt	10000	DISPLAY	
ct	4256	sp	2.9
alock	n	wp	5004.5
gain	48	vs	10000
FLAGS		sc	0
il	n	wc	240
in	n	hzmm	20.85
dp	y	is	7242.74
hs	nn	rfl	991.4
satfrq	1869.4	rfp	0
satpwr	-14	th	7
sattime	3.0	ins	1.000
spacing	50.0	ai cdc ph	
pattern	1		
intsub	y		
cycle	y		
control	1522.5		
mix	0		
tau	0.100		
sspul	y		



9a-*E*

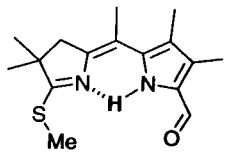


STANDARD PROTON PARAMETERS

exp1 s2pul

SAMPLE		DEC. & VT	
date	Nov 9 2001	dfrq	499.875
solvent	CDCl3	dn	H1
file	exp	dpwr	0
ACQUISITION		dof	0
sfrq	499.875	dm	nnn
tn	H1	dmm	c
at	2.997	dmf	200
np	47936	homo	n
sw	7998.4	dseq	
fb	4400	dres	1.0
bs	8	temp	21.0
tpwr	59	DEC2	
pw	7.7	dfrq2	0
d1	1.000	dn2	
tof	513.1	dpwr2	1
nt	16	dof2	0
ct	16	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	200
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	PROCESSING	
hs	nn	lb	0.05
DISPLAY		wtfile	
sp	-991.1	proc	ft
wp	7998.4	fn	not used
vs	344	math	f
sc	0		
wc	240	werr	
hzmm	33.33	wexp	wft
is	7242.74	wbs	wft
rfl	4625.2	wnt	
rfp	3634.1		
th	7		
ins	1.000		

nm cdc ph



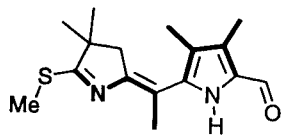
9a-*Z*

STANDARD PROTON PARAMETERS

exp1 s2pul

SAMPLE		DEC. & VT	
date	Feb 25 2002	dfrq	499.876
solvent	CD2Cl2	dn	H1
file	exp	dpwr	0
ACQUISITION		dof	0
sfrq	499.876	dm	nnn
tn	H1	dmm	c
at	2.997	dmf	200
np	47936	homo	n
sw	7998.4	dseq	
fb	4400	dres	1.0
bs	8	temp	21.0
tpwr	59	DEC2	
pw	7.7	dfrq2	0
d1	1.000	dn2	
tof	513.1	dpwr2	1
nt	32	dof2	0
ct	32	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	200
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	PROCESSING	
hs	nn	lb	0.05
DISPLAY		wtfile	
sp	-18.9	proc	ft
wp	5523.7	fn	not used
vs	833	math	f
sc	0		
wc	240	werr	
hzmm	23.02	wexp	wft
is	7242.74	wbs	wft
rfl	3663.0	wnt	
rfp	2659.3		
th	7		
ins	1.000		

nm cdc ph



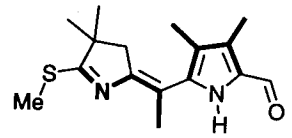
9a-*E*

+ EtZnI (1 equiv.)

STANDARD PROTON PARAMETERS

exp1 s2pul

SAMPLE		DEC. & VT	
date	Feb 25 2002	dfrq	499.876
solvent	CD2Cl2	dn	H1
file	exp	dpwr	0
ACQUISITION		dof	0
sfrq	499.876	dm	nnn
tn	H1	dmm	c
at	2.997	dmf	200
np	47936	homo	n
sw	7998.4	dseq	
fb	4400	dres	1.0
bs	8	temp	21.0
tpwr	59	DEC2	
pw	7.7	dfrq2	0
dl	1.000	dn2	
tof	513.1	dpwr2	1
nt	32	dof2	0
ct	32	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	200
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	PROCESSING	
hs	nn	lb	0.05
DISPLAY		wtfile	
sp	2.8	proc	ft
wp	5481.7	fn	not used
vs	1214	math	f
sc	0		
wc	240	werr	
hzmm	22.84	wexp	wft
is	933185.35	wbs	wft
rfl	1003.6	wnt	
rfp	0		
th	7		
ins	100.000		
nm	ph		



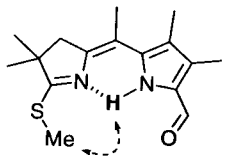
9a-*E*

+ EtZnI (2 equiv.)

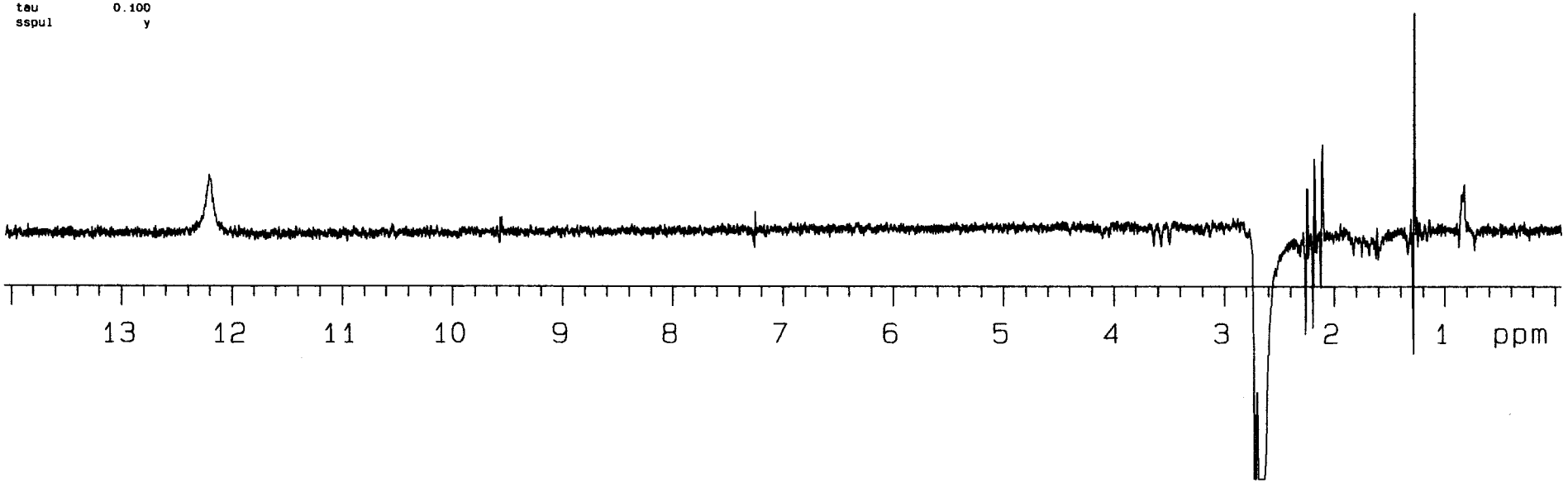
CYCLENOE PARAMETERS

exp5 cyclenoe

SAMPLE		DEC. & VT	
date	Jul 17 2002	dn	H1
solvent	CDCl3	dof	0
file	exp	dm	nnn
ACQUISITION		dmm	c
sfrq	299.962	dpwr	26
tn	H1	dpwrf	undefined
at	2.502	homo	n
np	30016	PROCESSING	
sw	5998.8	lb	0.40
fb	3400	fn	not used
bs	32	math	f
ss	8		
pw	13.0	werr	ADC_test
d1	3.000	wexp	wft
tof	735.4	wbs	wft
nt	1024	wnt	
ct	960	DISPLAY	
alock	n	sp	-15.5
gain	60	wp	4233.2
FLAGS		vs	9214
il	n	sc	0
in	n	wc	250
dp	y	hzmm	16.93
hs	nn	is	1113.75
satfrq	-689.5	rfl	773.1
satpwr	-14	rfp	0
sattime	3.0	th	20
spacing	3.0	ins	100.000
pattern	1	ai ph	
intsub	y		
cycle	y		
control	-1241.1		
mix	0		
tau	0.100		
sspul	y		



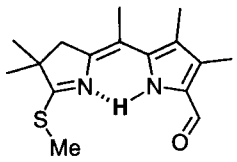
9a-Z



STANDARD PROTON PARAMETERS

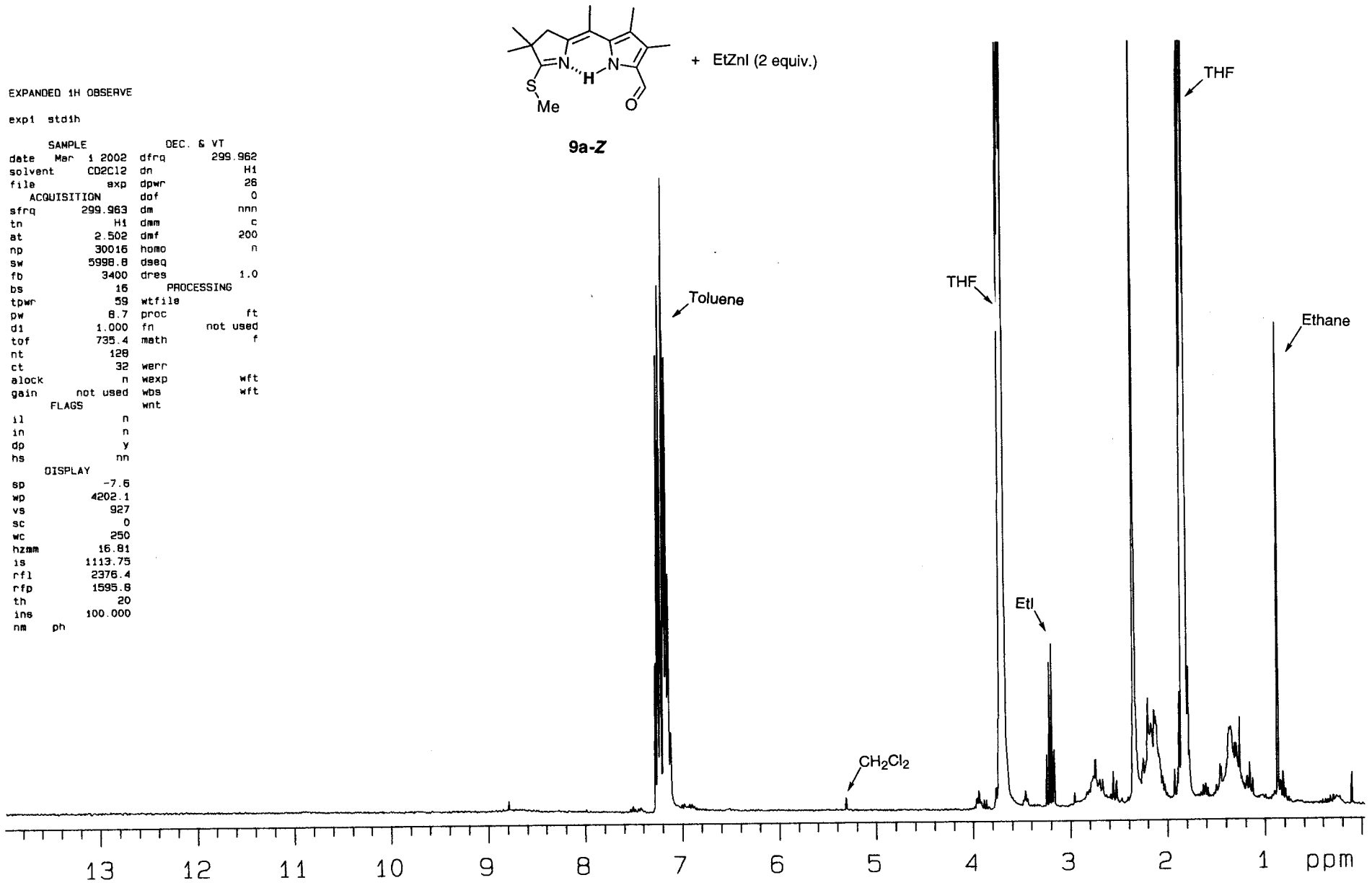
expl s2pul

SAMPLE		DEC. & VT	
date	Feb 25 2002	dfrq	499.876
solvent	CD2Cl2	dn	H1
file	exp	dpwr	0
ACQUISITION		dof	0
sfrq	499.876	dm	nnn
tn	H1	dmm	c
at	2.997	dmf	200
np	47936	homo	n
sw	7998.4	dseq	
fb	4400	dres	1.0
bs	8	temp	21.0
tpwr	59	DEC2	
pw	7.7	dfrq2	0
d1	1.000	dn2	
tof	513.1	dpwr2	1
nt	32	dof2	0
ct	32	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	200
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	PROCESSING	
hs	nn	lb	0.05
DISPLAY		wtfile	
sp	-500.5	proc	ft
wp	7495.1	fn	not used
vs	864	math	f
sc	0		
wc	240	werr	
hzmm	31.23	wexp	wft
is	36981.32	wbs	wft
rfl	1003.6	wnt	
rfp	0		
th	7		
ins	100.000		
nm	ph		



9a-*Z*

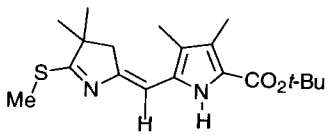
\+ EtZnI (1 equiv.)


EXPANDED 1H OBSERVE
exp1 stdih
SAMPLE
date Mar 1 2002
solvent CD2Cl2
file exp
ACQUISITION
sfrq 299.963
tn H1
at 2.502
np 30016
sw 5998.8
fb 3400
bs 16
tpwr 59
pw 8.7
d1 1.000
tof 735.4
nt 128
ct 32
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -7.6
wp 4202.1
vs 927
sc 0
wc 250
hzmm 16.81
is 1113.75
rfl 2376.4
rfp 1595.8
th 20
ins 100.000
nm ph
DEC. & VT
dfrq 299.962
dn H1
dpwr 26
dof 0
dm nnn
dmm c
dmf 200
homo n
dseq
dres 1.0
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp wft
wbs wft
wnt
N
H
N
S
Me
O
+ EtZnI (2 equiv.)
9a-Z
Toluene
THF
THF
EtI
Ethane
CH2Cl2
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm

STANDARD 1H OBSERVE

exp1 std1h

SAMPLE		DEC. & VT	
date	Nov 14 2001	dfrq	299.961
solvent	CDCl3	dn	H1
file	exp	dpwr	26
ACQUISITION		dof	0
sfrq	299.961	dm	nnn
tn	H1	dmm	c
at	2.504	dmf	200
np	20032	homo	n
sw	4000.0	dseq	
fb	2200	dres	1.0
bs	15	PROCESSING	
tpwr	59	wtfile	
pw	8.7	proc	ft
d1	1.000	fn	not used
tof	0	math	f
nt	8		
ct	8	werr	
alock	n	wexp	wft
gain	not used	wbs	wft
FLAGS		wnt	
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-9.9		
wp	3005.8		
vs	131		
sc	0		
wc	250		
hzmm	12.02		
is	1113.75		
rfl	2689.6		
rfp	2180.7		
th	20		
ins	100.000		
nm	ph		

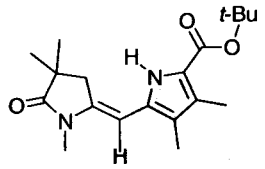


11b-*E*

STANDARD PROTON PARAMETERS

exp1 s2pul

SAMPLE		DEC. & VT	
date	Jul 15 2002	dfrq	499.875
solvent	CDCl3	dn	H1
file	exp	dpwr	0
ACQUISITION		dof	0
sfrq	499.875	dm	nnn
tn	H1	dmm	c
at	2.997	dmf	200
np	47936	homo	n
sw	7998.4	dseq	
fb	4400	dres	1.0
bs	8	temp	21.0
tpwr	59	DEC2	
pw	7.7	dfrq2	0
d1	1.000	dn2	
tof	513.1	dpwr2	1
nt	64	dof2	0
ct	64	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	200
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	PROCESSING	
hs	nn	lb	0.05
DISPLAY		wtfile	
sp	1.1	proc	ft
wp	4997.4	fn	not used
vs	200	math	f
sc	0		
wc	240	werr	
hzmm	20.82	wexp	wft
is	253.88	wbs	wft
rfl	4625.7	wnt	
rfp	3634.1		
th	2		
ins	100.000		
nm	ph		



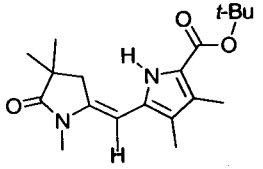
12b-*E*

STANDARD CARBON PARAMETERS

exp1 s2pul

SAMPLE		DEC. & VT	
date	Jul 15 2002	dfrq	499.875
solvent	CDCl3	dn	H1
file	exp	dpwr	41
ACQUISITION		dof	0
sfrq	125.707	dm	nyy
tn	C13	dmm	w
at	1.500	dmf	14815
np	85618	dseq	
sw	28530.7	dres	1.0
fb	15800	homo	n
bs	32	temp	21.0
tpwr	60	DEC2	
pw	11.3	dfrq2	0
d1	0.800	dn2	
d2	0.300	dpwr2	1
tof	1704.8	dof2	0
nt	10000	dm2	n
ct	6272	dmm2	c
alock	n	dmf2	10000
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	PROCESSING	
dp	y	lb	0.50
hs	nn	wtfile	
DISPLAY		proc	ft
sp	-590.9	fn	not used
wp	25689.1	math	f
vs	290		
sc	0	werr	
wc	240	wexp	wft
hzmm	107.04	wbs	wft
is	500.00	wnt	
rfl	10337.0		
rfp	9707.3		
th	68		
ins	1.000		

nm cdc ph

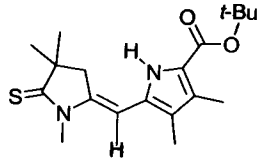


12b-*E*

STANDARD PROTON PARAMETERS

exp1 s2pul

SAMPLE		DEC. & VT	
date	Jul 16 2002	dfrq	499.875
solvent	CDCl3	dn	H1
file	exp	dpwr	0
ACQUISITION		dof	0
sfrq	499.875	dm	nnn
tn	H1	dmm	c
at	2.997	dmf	200
np	47936	homo	n
sw	7998.4	dseq	
fb	4400	dres	1.0
bs	8	temp	21.0
tpwr	59	DEC2	
pw	7.7	dfrq2	0
d1	1.000	dn2	
tof	513.1	dpwr2	1
nt	64	dof2	0
ct	64	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	200
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	PROCESSING	
hs	nn	lb	0.05
DISPLAY		wtfile	
sp	-9.8	proc	ft
wp	4997.4	fn	not used
vs	200	math	f
sc	0		
wc	240	werr	
hzmm	20.82	wexp	wft
is	557.54	wbs	wft
rfl	4625.9	wnt	
rfp	3634.1		
th	7		
ins	100.000		
nm	ph		



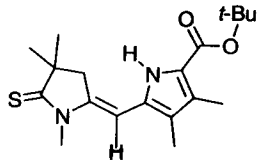
13b-*E*

STANDARD CARBON PARAMETERS

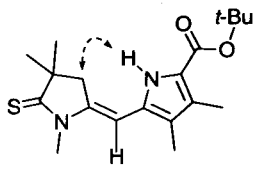
exp1 s2pul

SAMPLE		DEC. & VT	
date	Jul 16 2002	dfrq	499.875
solvent	CDCl3	dn	H1
file	exp	dpwr	41
ACQUISITION		dof	0
sfrq	125.707	dm	nyy
tn	C13	dmm	w
at	1.500	dmf	14815
np	85618	dseq	
sw	28530.7	dres	1.0
fb	15800	homo	n
bs	32	temp	21.0
tpwr	60	DEC2	
pw	11.3	dfrq2	0
d1	0.800	dn2	
d2	0.300	dpwr2	1
tof	1704.8	dof2	0
nt	10000	dm2	n
ct	5536	dmm2	c
alock	n	dmf2	10000
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	PROCESSING	
dp	y	lb	0.50
hs	nn	wtfile	
DISPLAY		proc	ft
sp	-5.8	fn	not used
wp	27635.2	math	f
vs	227		
sc	0	werr	
wc	240	wexp	wft
hzmm	115.15	wbs	wft
is	500.00	wnt	
rfl	10335.6		
rfp	9707.3		
th	68		
ins	1.000		

nm cdc ph



13b-*E*

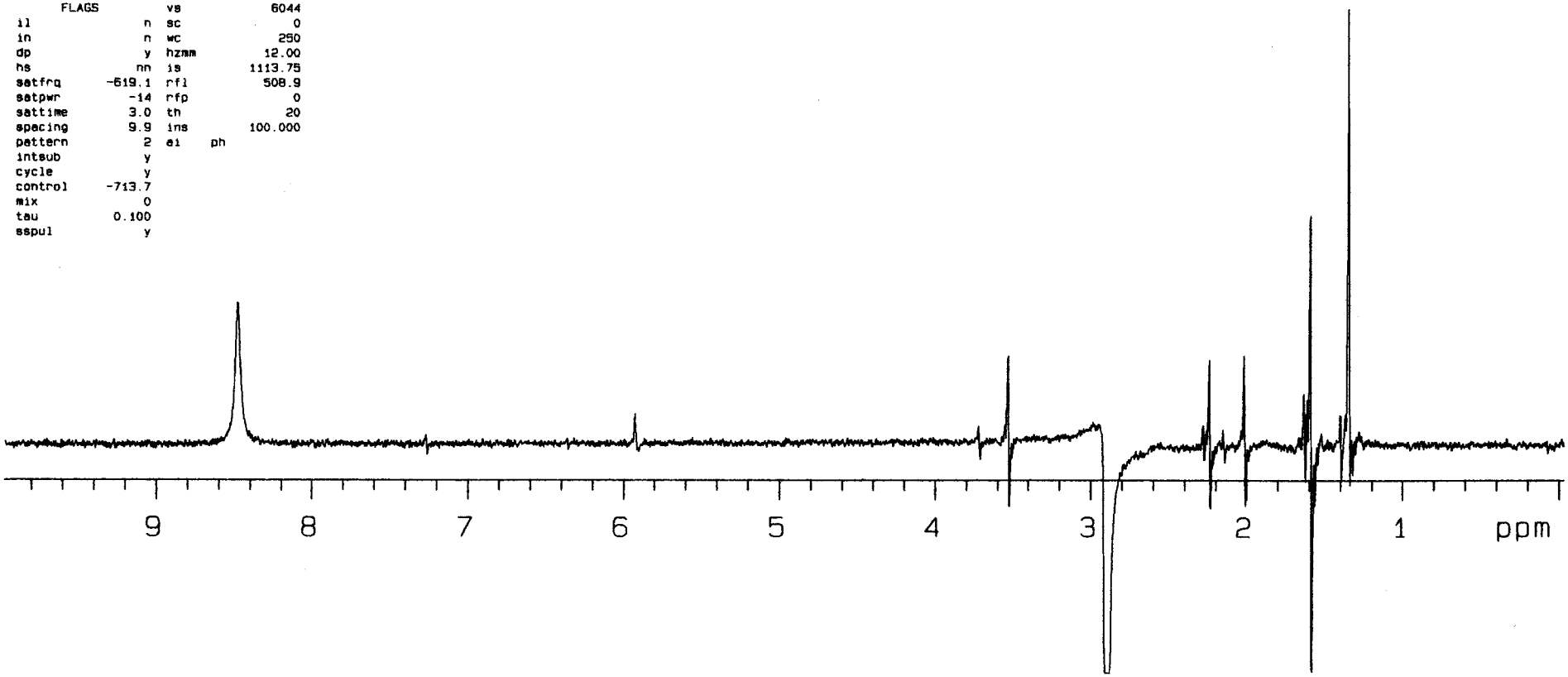


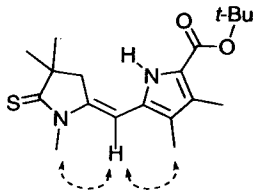
13b-*E*

STANDARD 1H OBSERVE

expi cyclenoe

SAMPLE		DEC. & VT	
date	Nov 9 2001	dn	H1
solvent	CDCl3	dof	0
file	exp	dm	nnn
ACQUISITION		dmm	c
sfrq	299.981	dpwr	26
tn	H1	dpwrf	undefined
at	2.504	homo	n
np	20032	PROCESSING	
sw	4000.0	lb	0.40
fb	2200	fn	not used
bs	32	math	f
ss	8		
pw	13.0	werr	ADC_test
d1	2.000	wexp	wft
tof	0	wbs	wft
nt	1024	wnt	
ct	96	DISPLAY	
alock	n	sp	-9.6
gain	52	wp	3001.2
FLAGS		vs	6044
il	n	sc	0
in	n	wc	250
dp	y	hzmm	12.00
hs	nn	is	1113.75
satfrq	-619.1	rfl	508.9
satpwr	-14	rfp	0
sattime	3.0	th	20
spacing	9.9	ins	100.000
pattern	2	ai ph	
intsub	y		
cycle	y		
control	-713.7		
mix	0		
tau	0.100		
sspul	y		



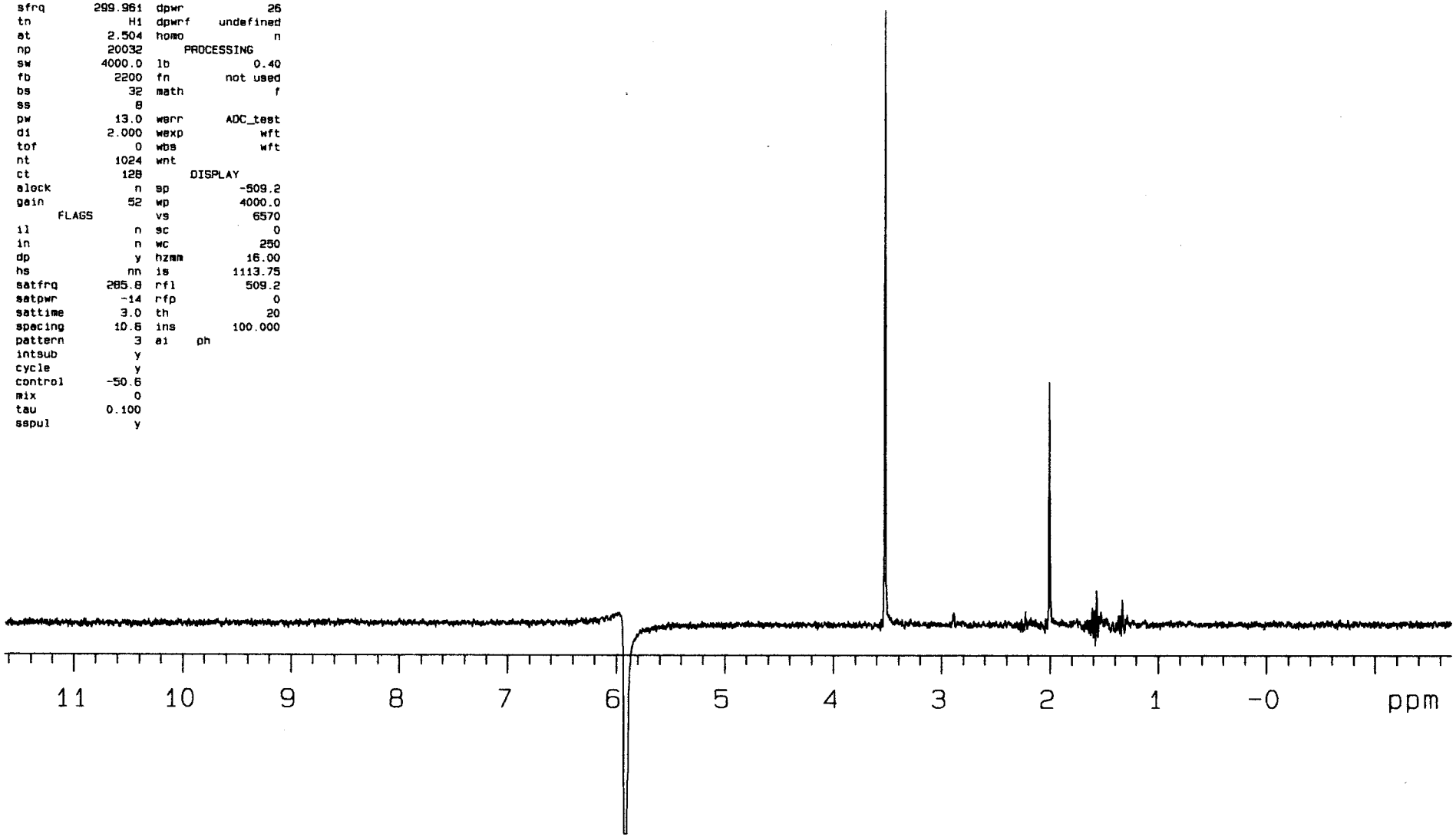


13*b*-*E*

STANDARD 1H OBSERVE

expl cyclenoe

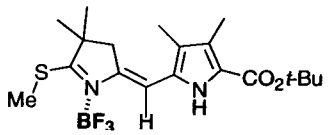
SAMPLE		DEC. & VT	
date	Nov 8 2001	dn	H1
solvent	CDCl3	dof	0
file	exp	dm	nnn
ACQUISITION		dmm	c
sfrq	299.961	dpwr	26
tn	H1	dpwrf	undefined
at	2.504	homo	n
np	20032	PROCESSING	
sw	4000.0	lb	0.40
fb	2200	fn	not used
bs	32	math	f
ss	8		
pw	13.0	werr	ADC_test
d1	2.000	wexp	wft
tof	0	wbs	wft
nt	1024	wnt	
ct	128	DISPLAY	
alock	n	sp	-509.2
gain	52	wp	4000.0
FLAGS		vs	6570
il	n	sc	0
in	n	wc	250
dp	y	hzmm	16.00
hs	nn	is	1113.75
satfrq	285.8	rfl	509.2
satpwr	-14	rfp	0
sattime	3.0	th	20
spacing	10.6	ins	100.000
pattern	3	ai ph	
intsub	y		
cycle	y		
control	-50.6		
mix	0		
tau	0.100		
sspul	y		



STANDARD PROTON PARAMETERS

exp1 s2pul

SAMPLE		DEC. & VT	
date	Feb 24 2002	dfrq	499.876
solvent	CD2Cl2	dn	H1
file	exp	dpwr	0
ACQUISITION		dof	0
sfrq	499.876	dm	nnn
tn	H1	dmm	c
at	2.997	dmf	200
np	47936	homo	n
sw	7998.4	dseq	
fb	4400	dres	1.0
bs	8	temp	21.0
tpwr	59	DEC2	
pw	7.7	dfrq2	0
d1	1.000	dn2	
tof	513.1	dpwr2	1
nt	128	dof2	0
ct	80	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	200
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	PROCESSING	
hs	nn	lb	0.05
DISPLAY		wtfile	
sp	-512.5	proc	ft
wp	7505.8	fn	not used
vs	182	math	f
sc	0		
wc	240	werr	
hzmm	31.27	wexp	wft
is	181.00	wbs	wft
rfl	3664.2	wnt	
rfp	2659.3		
th	7		
ins	100.000		
nm	ph		

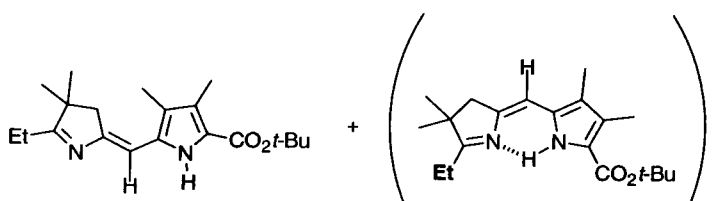


17b-*E*

STANDARD PROTON PARAMETERS

exp1 s2pul

SAMPLE		DEC. & VT	
date	Jul 23 2002	dfrq	499.875
solvent	CDCl3	dn	H1
file	exp	dpwr	0
ACQUISITION		dof	0
sfrq	499.875	dm	nnn
tn	H1	dmm	c
at	2.997	dmf	200
np	47936	homo	n
sw	7998.4	dseq	
fb	4400	dres	1.0
bs	8	temp	21.0
tpwr	59	DEC2	
pw	7.7	dfrq2	0
d1	1.000	dn2	
tof	513.1	dpwr2	1
nt	500	dof2	0
ct	72	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	200
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	PROCESSING	
hs	nn	lb	0.05
DISPLAY		wtfile	
sp	-20.6	proc	ft
wp	6023.1	fn	not used
vs	200	math	f
sc	0		
wc	240	werr	
hzmm	25.10	wexp	wft
is	2927.78	wbs	wft
rfl	4625.7	wnt	
rfp	3634.1		
th	1		
ins	100.000		
nm	ph		



18b-*E*

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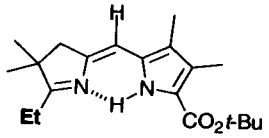
18b-*Z*

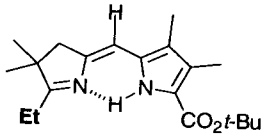


18b-Z

STANDARD 1H OBSERVE

exp1 stdih

SAMPLE		DEC. & VT	
date	Jul 30 2002	dfrq	299.961
solvent	CDCl3	dn	H1
file	/data1/guest/~	dpwr	26
	Z-meso-H-ethylbuty~	dof	0
	lester.fid	dm	nnn
ACQUISITION		dmm	c
sfrq	299.961	dmf	200
tn	H1	homo	n
at	2.504	dseq	
np	20032	dres	1.0
sw	4000.0	PROCESSING	
fb	2200	wtfile	
bs	16	proc	ft
tpwr	59	fn	not used
pw	8.7	math	f
d1	1.000		
tof	0	werr	
nt	500	wexp	wft
ct	160	wbs	wft
alock	n	wnt	
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-11.6		
wp	3491.9		
vs	225		
sc	0		
wc	250		
hzmm	13.97		
is	3814.70		
rfl	2689.6		
rfp	2180.7		
th	20		
ins	400.000		
nm	ph		

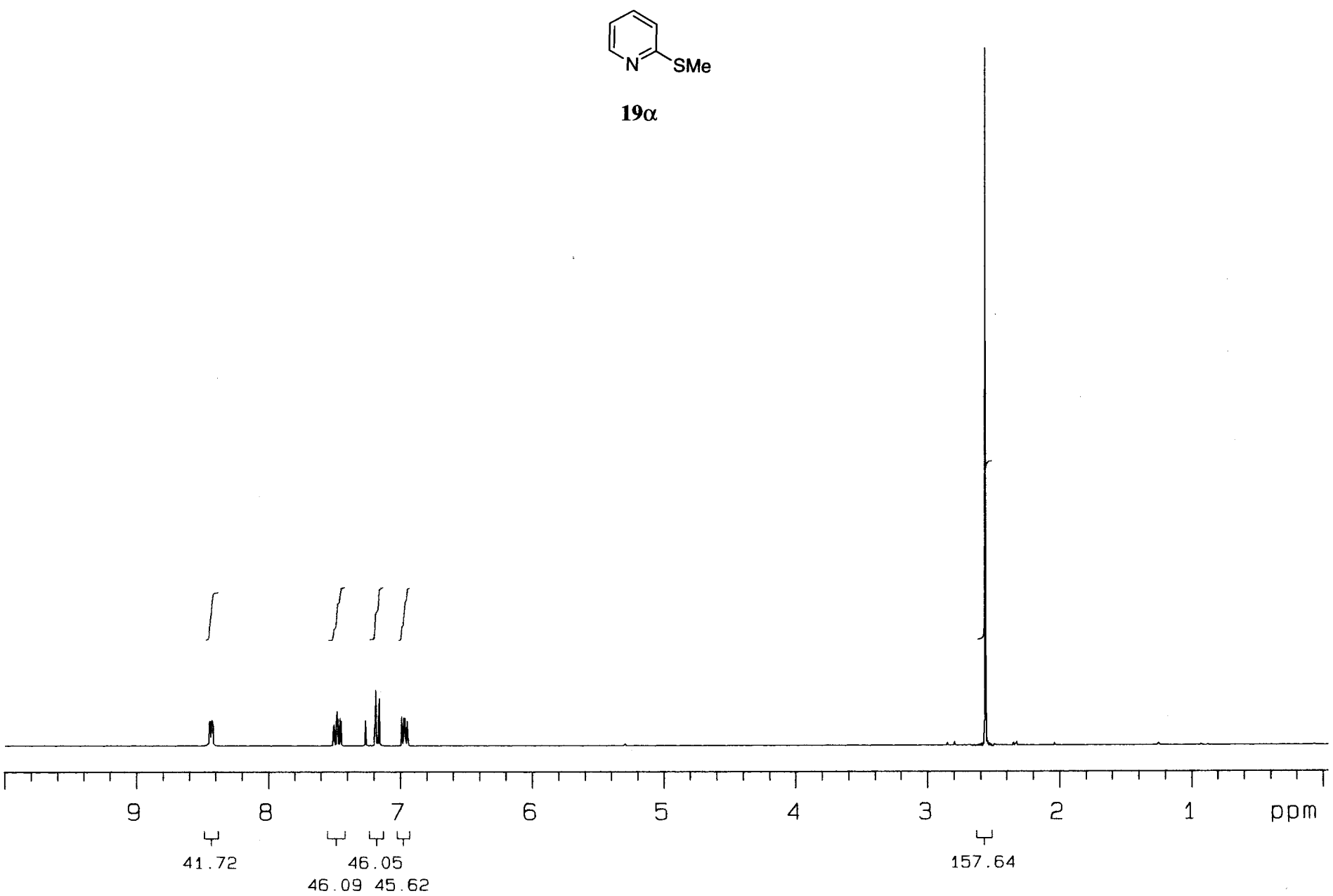


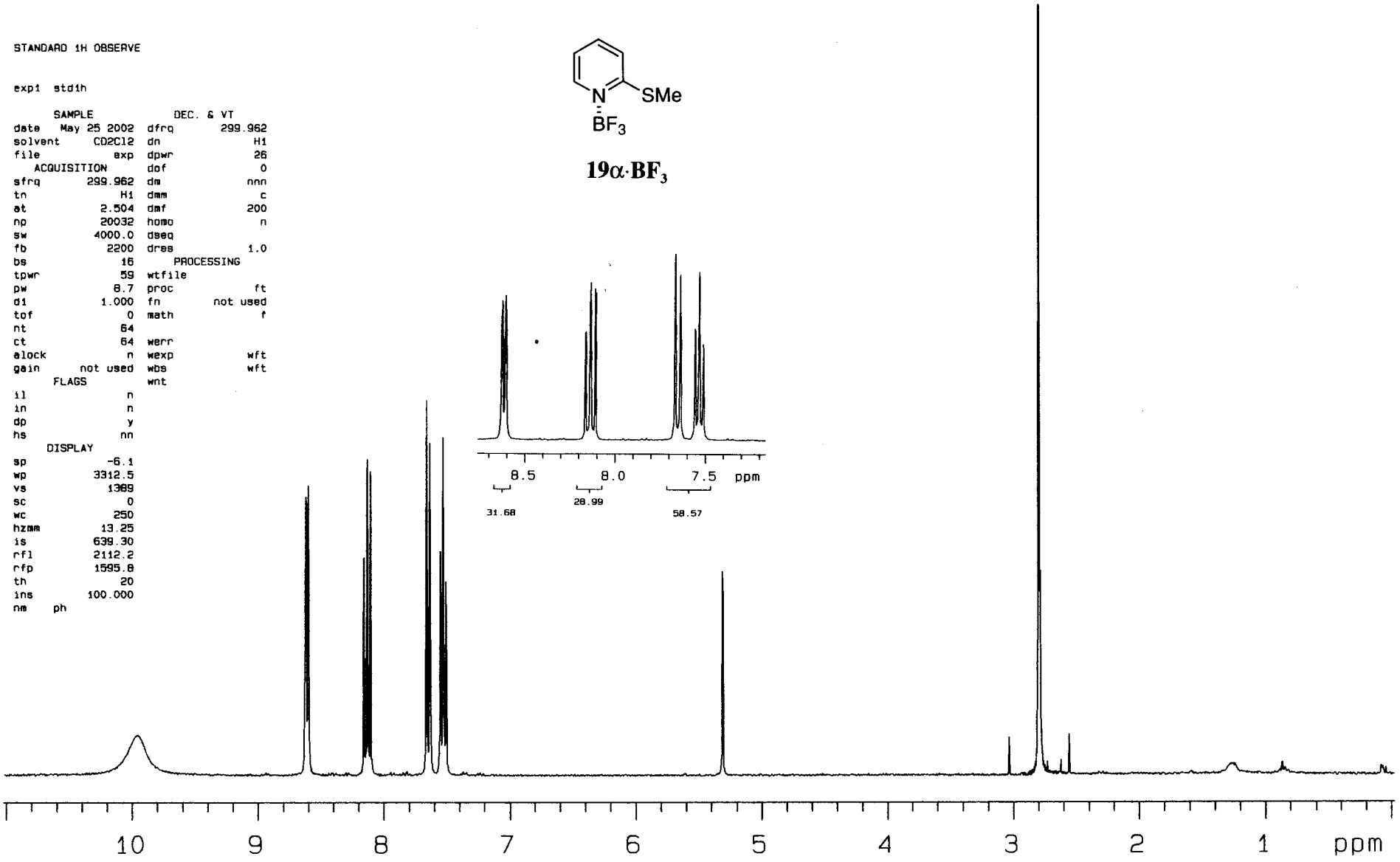
18b-*Z*

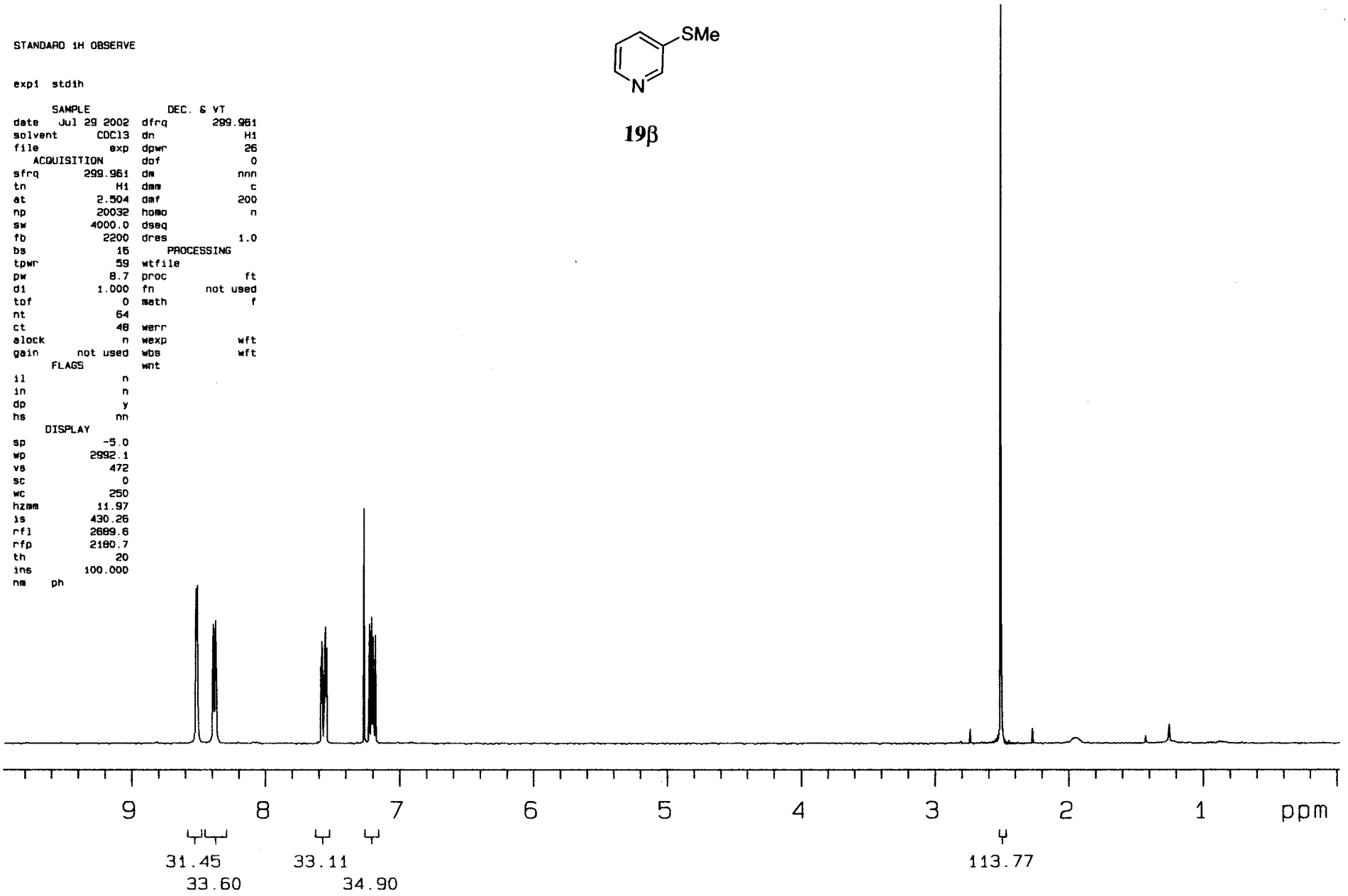
13C OBSERVE

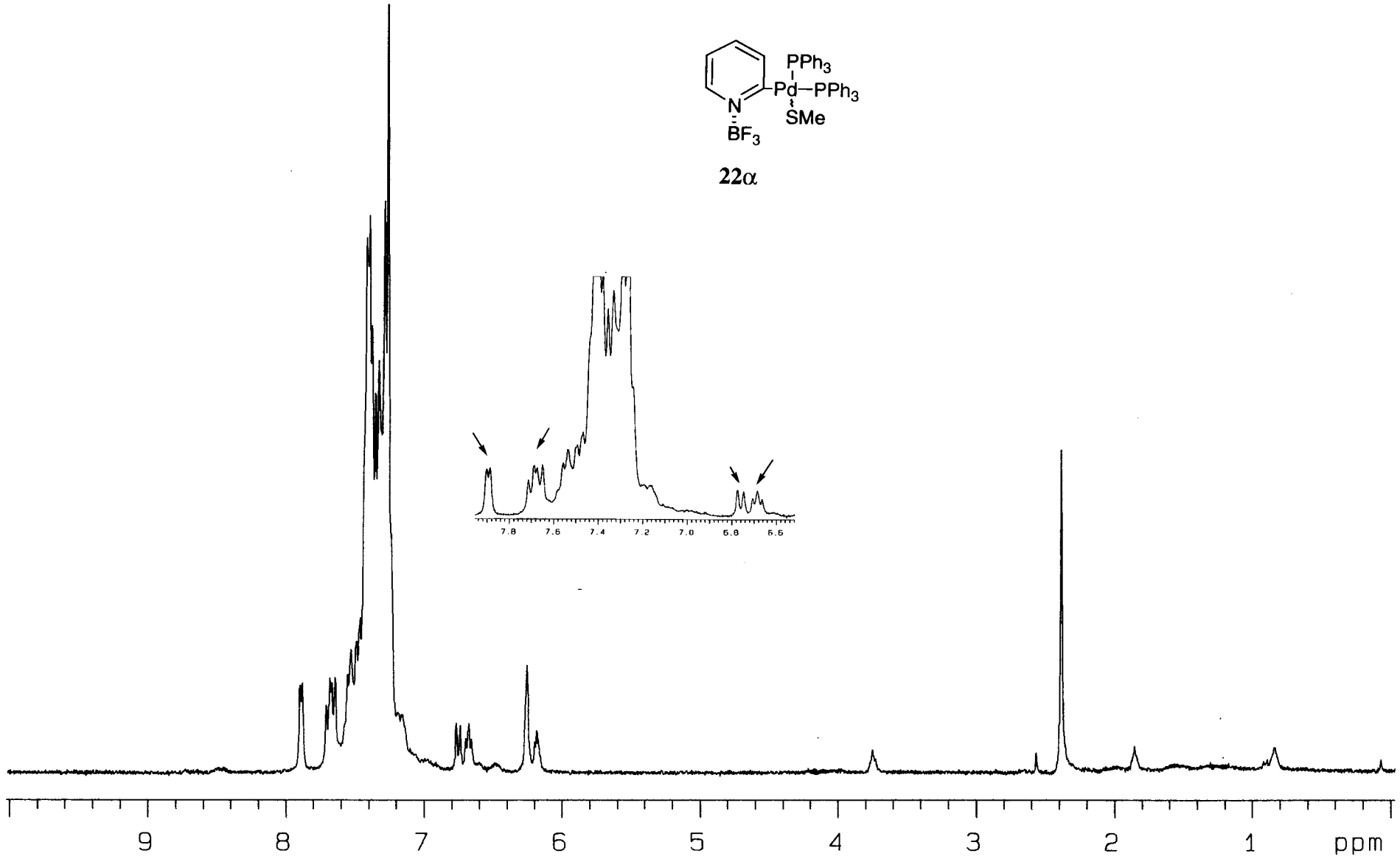
exp1 std13c

SAMPLE		DEC. & VT	
date	Jul 31 2002	dfrq	299.961
solvent	CDCl3	dn	H1
file	exp	dpwr	39
ACQUISITION		dof	0
sfrq	75.433	dm	nyy
tn	C13	dmm	w
at	1.201	dmf	8548
np	43328	dseq	
sw	18042.4	dres	1.0
fb	10000	homo	n
bs	64	PROCESSING	
tpwr	57	lb	0.10
pw	7.0	wtfile	
d1	0.700	proc	ft
d2	0.300	fn	not used
tof	961.8	math	f
nt	100000		
ct	13760	werr	
alock	n	wexp	wft
gain	not used	wbs	wft
FLAGS		wnt	
il	n		
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-475.7		
wp	15603.1		
vs	242		
sc	0		
wc	250		
hzmm	62.41		
is	500.00		
rfl	499.4		
rfp	0		
th	15		
ins	100.000		
nm cdc ph			


N
SMe
19α
9
8
7
6
5
4
3
2
1
ppm
41.72
46.05
46.09
45.62
157.64


STANDARD 1H OBSERVE
exp1 std1h
SAMPLE
date May 25 2002
solvent CD2Cl2
file exp
ACQUISITION
sfrq 299.962
tn H1
at 2.504
np 20032
sw 4000.0
fb 2200
bs 16
tpwr 59
pw 8.7
d1 1.000
tof 0
nt 64
ct 64
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -6.1
wp 3312.5
vs 1389
sc 0
wc 250
hzmm 13.25
is 639.30
rfl 2112.2
rfp 1595.8
th 20
ins 100.000
nm ph
DEC. & VT
dfrq 299.962
dn H1
dpwr 26
dof 0
dm nnn
dmm c
dmf 200
homo n
dseq
dres 1.0
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp wft
wbs wft
wnt
N
SMe
BF3
19α·BF3
8.5
8.0
7.5
ppm
31.68
28.99
58.57
10
9
8
7
6
5
4
3
2
1
ppm


STANDARD 1H OBSERVE
expi stdih
SAMPLE
date Jul 29 2002
solvent CDCl3
file exp
ACQUISITION
sfrq 299.961
tn H1
at 2.504
np 20032
sw 4000.0
fb 2200
bs 16
tpwr 59
pw 8.7
d1 1.000
tof 0
nt 64
ct 48
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -5.0
wp 2992.1
vs 472
sc 0
wc 250
hzmm 11.97
is 430.26
rfl 2689.6
rfp 2180.7
th 20
ins 100.000
nm ph
DEC. & VT
dfrq 299.961
dn H1
dpwr 26
dof 0
dm nnn
dmm c
dmf 200
homo n
dseq
dres 1.0
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp wft
wbs wft
wnt
SMe
N
19β
9
8
7
6
5
4
3
2
1
ppm
31.45
33.60
33.11
34.90
113.77

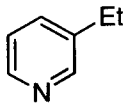


22α

STANDARD PROTON PARAMETERS

exp1 s2pul

SAMPLE		DEC. & VT	
date	Jul 15 2002	dfrq	499.875
solvent	CDCl3	dn	H1
file	exp	dpwr	0
ACQUISITION		dof	0
sfrq	499.875	dm	nnn
tn	H1	dmm	c
at	2.997	dmf	200
np	47936	homo	n
sw	7998.4	dseq	
fb	4400	dres	1.0
bs	8	temp	21.0
tpwr	59	DEC2	
pw	7.7	dfrq2	0
d1	1.000	dn2	
tof	513.1	dpwr2	1
nt	64	dof2	0
ct	40	dm2	n
alock	n	dmm2	c
gain	not used	dmf2	200
FLAGS		dseq2	
il	n	dres2	1.0
in	n	homo2	n
dp	y	PROCESSING	
hs	nn	lb	0.05
DISPLAY		wtfile	
sp	-8.6	proc	ft
wp	4993.5	fn	not used
vs	195	math	f
sc	0		
wc	240	werr	
hzmm	20.81	wexp	wft
is	396.15	wbs	wft
rfl	4625.9	wnt	
rfp	3634.1		
th	7		
ins	100.000		
nm	ph		



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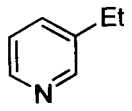
STANDARD CARBON PARAMETERS

exp1 s2pul

SAMPLE		DEC. & VT	
date	Jul 15 2002	dfrq	499.875
solvent	CDCl3	dn	H1
file	exp	dpwr	41
ACQUISITION		dof	0
sfrq	125.707	dm	nyy
tn	C13	dmm	w
at	1.500	dmf	14815
np	85618	dseq	
sw	28530.7	dres	1.0
fb	15800	homo	n
bs	32	temp	21.0
tpwr	60	DEC2	
pw	11.3	dfrq2	0
d1	0.800	dn2	
d2	0.300	dpwr2	1
tof	1704.8	dof2	0
nt	10000	dm2	n
ct	1632	dmm2	c
alock	n	dmf2	10000
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	PROCESSING	
dp	y	lb	0.50
hs	nn	wtfile	
DISPLAY		proc	ft
sp	-591.8	fn	not used
wp	25685.2	math	f
vs	148		
sc	0	werr	
wc	240	wexp	wft
hzmm	107.02	wbs	wft
is	500.00	wnt	
rfl	10337.8		
rfp	9707.3		
th	68		
ins	1.000		
nm cdc ph			



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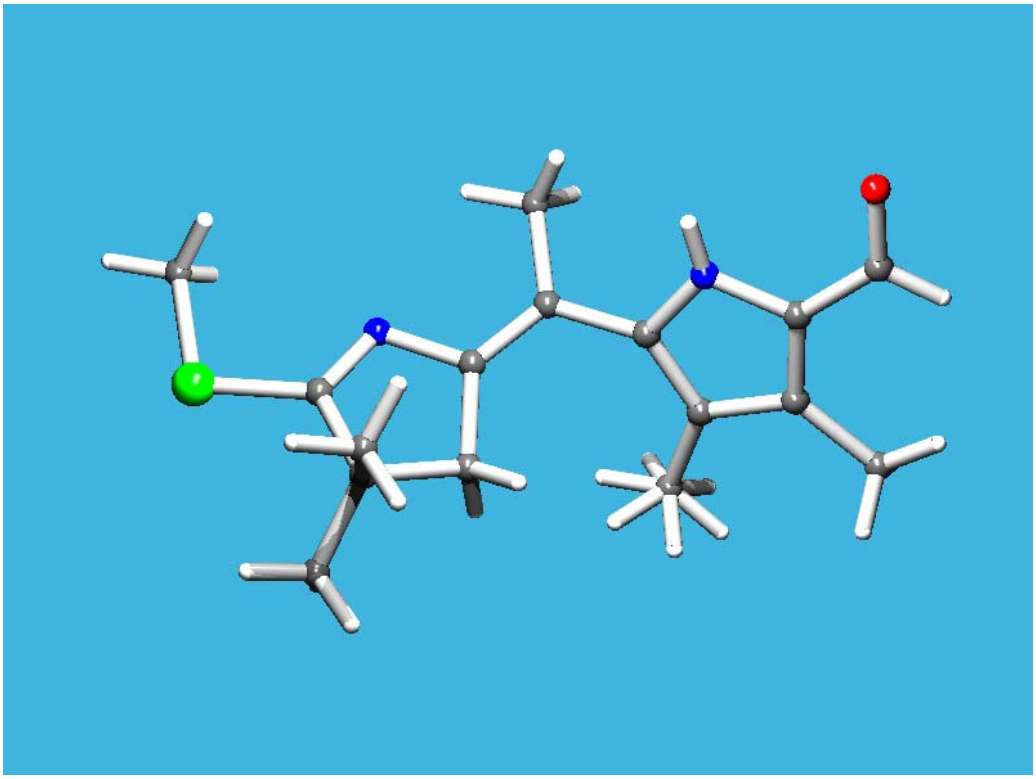
REFERENCE NUMBER: **02006**

CRYSTAL STRUCTURE REPORT

$C_{16}\ H_{22}\ N_2\ O\ S$

Report prepared for: I. Ghosh/P. Jacobi, Darmouth College, NH

8th January 2002



Neil R. Brooks

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Department of Chemistry

University of Minnesota

207 Pleasant St. S.E.

Minneapolis, MN 55455

Data collection

A crystal (approximate dimensions 0.44 x 0.40 x 0.36 mm^3) was placed onto the tip of a 0.1 mm diameter glass capillary and mounted on a Bruker CCD area detector diffractometer for a data collection at 173(2) K. A preliminary set of cell constants was calculated from reflections harvested from three sets of 20 frames. These initial sets of frames were oriented such that orthogonal wedges of reciprocal space were surveyed. This produced initial orientation matrices determined from 167 reflections. The data collection was carried out using MoKα radiation (graphite monochromator) with a frame time of 10 seconds and a detector distance of 4.9 cm. A randomly oriented region of reciprocal space was surveyed to the extent of 1.5 hemisphere and to a resolution of 0.84 Å. Three major sections of frames were collected with 0.30º steps in ω at 3 different ϕ settings and a detector position of -28º in 2θ. The intensity data were corrected for absorption and decay (SADABS).[1] Final cell constants were calculated from the xyz centroids of 2791 strong reflections from the actual data collection after integration (SAINT 6.01, 1999).[2] Please refer to Table 1 for additional crystal and refinement information.

Structure solution and refinement

The structure was solved using SIR92[3] and refined using SHELXL-97.[4] The space group $P2_1/c$ was determined based on systematic absences and intensity statistics. A direct-methods solution was calculated which provided most non-hydrogen atoms from the E-map. Full-matrix least squares / difference Fourier cycles were performed which located the remaining non-hydrogen atoms. All non-hydrogen atoms were refined with anisotropic displacement parameters. Most hydrogen atoms were placed in ideal positions and refined as riding atoms with relative isotropic displacement parameters. One methyl group was found to be disordered over two positions at 60° from each other. The hydrogen atom on the pyrrole N atom was found in the difference map and allowed to refine freely. The final full matrix least squares refinement converged to R1 = 0.0443 and wR2 = 0.1309 (F^2, all data). The program PLATON[5] was used for checking the structure.

Structure description

The structure is similar to the one suggested but the ester group appears to have been converted into an aldehyde.

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

[1] An empirical correction for absorption anisotropy, R. Blessing, *Acta Cryst.* **A51**, 33-38 (1995).

[2] SAINT V6.1, Bruker Analytical X-Ray Systems, Madison, WI.

[3] SIR92, A. Altomare, G. Cascarano, C. Giacovazzo, A. Gualardi, *J. Appl. Cryst.* **26**, 343-350 (1993).

[4] SHELXTL-Plus V5.10, Bruker Analytical X-Ray Systems, Madison, WI (1998).

[5] A.L. Spek, *Acta. Cryst.* **A46**, C34 (1990). PLATON, A Multipurpose Crystallographic Tool, Utrecht University, Utrecht, The Netherlands, A. L. Spek (2000).

Some equations of interest:

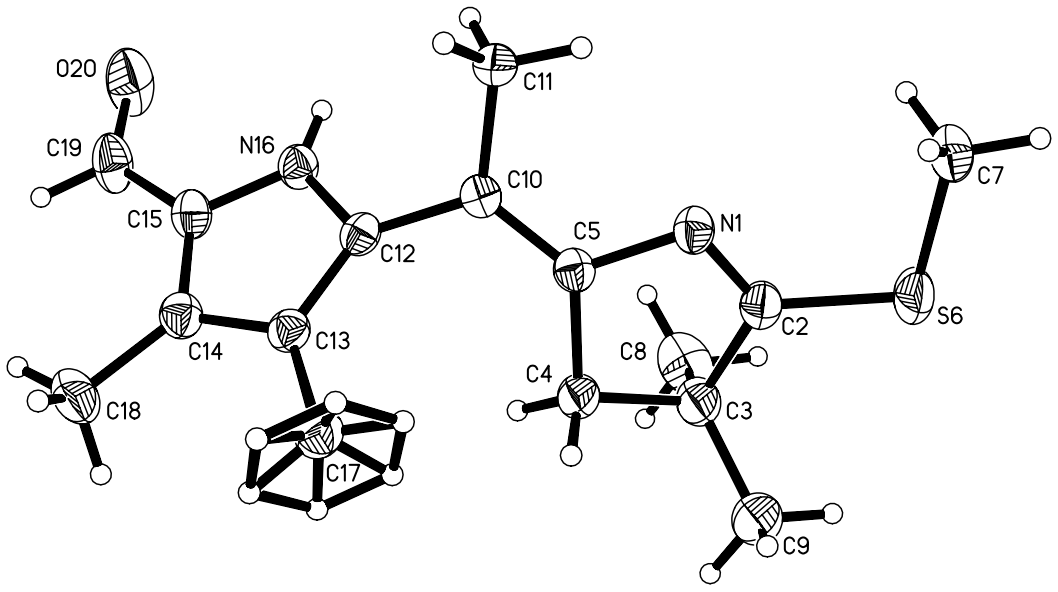
$$R_{int} = \Sigma\, |F_o^2 - < F_o^2 >| \,/\, \Sigma |F_o{}^2|$$

$$R_1 = \Sigma ||F_o| - |F_c|| \,/\, \Sigma |F_o|$$

$$wR2 = [\Sigma [w(F_o^2 - F_c{}^2)^2] \,/\, \Sigma\, [w(F_o^2)^2]]^{1/2}$$

$$\text{where } w = q \,/\, [\sigma^2 (F_o^2) + (a*P)^2 + b*P + d + e*\sin(\theta)]$$

$$\text{GooF} = S = [\Sigma [w(F_o^2 - F_c{}^2)^2] \,/\, (n-p)]^{1/2}$$



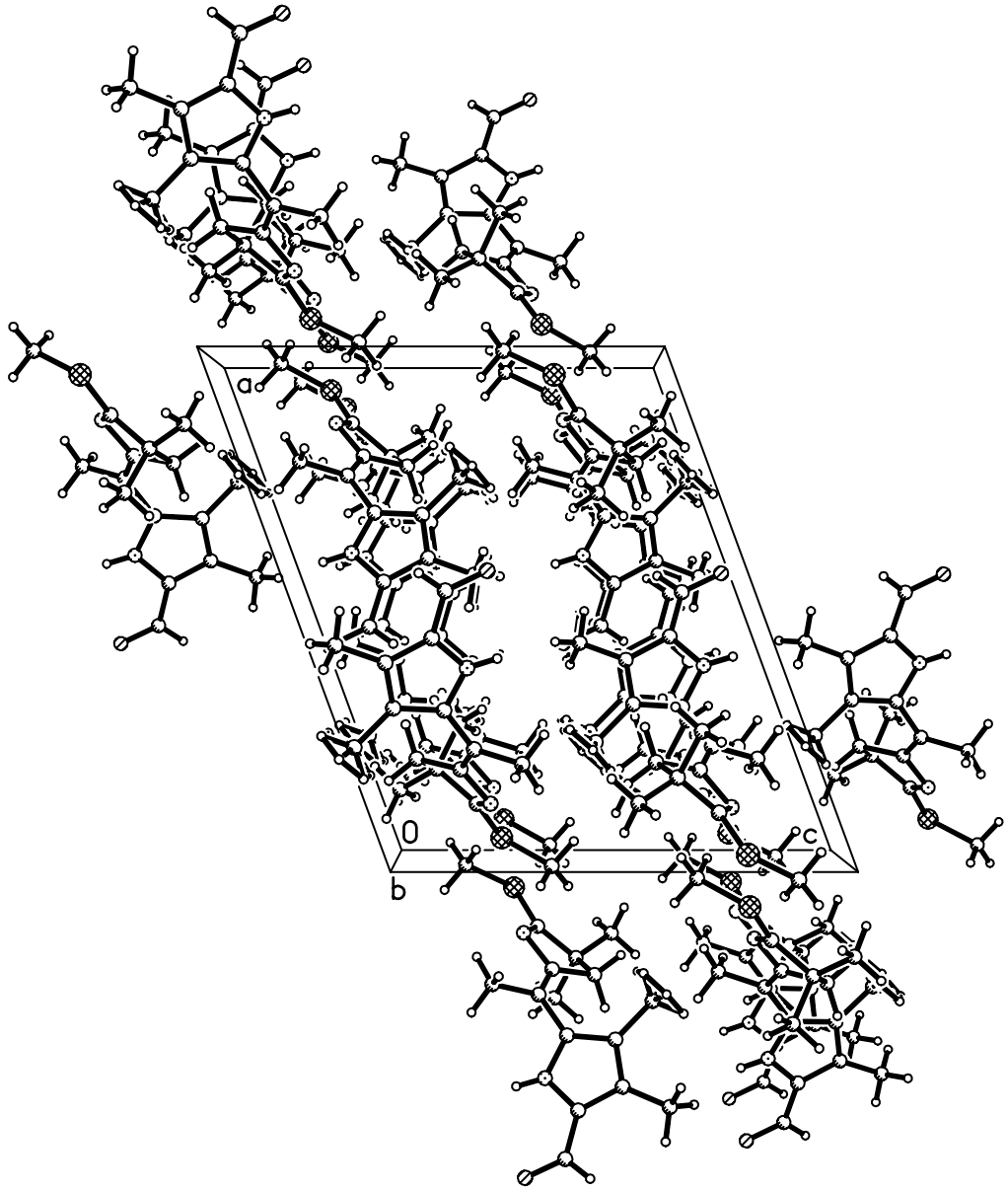

a
0
c
b

Table 1. Crystal data and structure refinement for 02006.

Identification code	02006	
Empirical formula	$C_{16}\ H_{22}\ N_2\ O\ S$	
Formula weight	290.42	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a= 15.576(3) Å	α= 90°
	b= 8.4535(15) Å	β= 110.222(3)°
	c= 13.024(2) Å	γ = 90°
Volume	1609.2(5) Å^3	
Z	4	
Density (calculated)	1.199 Mg/m^3	
Absorption coefficient	0.199 mm^{-1}	
F(000)	624	
Crystal color, morphology	pale, block	
Crystal size	0.44 x 0.40 x 0.36 mm^3	
Theta range for data collection	1.39 to 27.53°.	
Index ranges	-20 = h = 20, -10 = k = 10, -16 = l = 15	
Reflections collected	14039	
Independent reflections	3664 [R(int) = 0.028]	
Observed reflections	2948	
Completeness to theta = 27.53°	99.2%	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.918 and 0.843	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3664 / 0 / 188	
Goodness-of-fit on F^2	1.052	
Final R indices [I>2sigma(I)]	R1 = 0.0443, wR2 = 0.1212	
R indices (all data)	R1 = 0.0556, wR2 = 0.1309	
Largest diff. peak and hole	0.411 and -0.281 e.Å^{-3}	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for 02006. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
N1	1351(1)	1513(2)	2712(1)	26(1)
C2	1301(1)	72(2)	2383(1)	30(1)
C3	1954(1)	-401(2)	1792(2)	36(1)
C4	2188(1)	1273(2)	1493(2)	30(1)
C5	2018(1)	2281(2)	2362(1)	24(1)
S6	596(1)	-1368(1)	2621(1)	43(1)
C7	100(1)	-369(2)	3492(2)	36(1)
C9	1524(2)	-1464(2)	804(2)	57(1)
C10	2423(1)	3630(2)	2829(1)	22(1)
C8	2808(2)	-1167(2)	2618(2)	51(1)
C11	2171(1)	4429(2)	3721(1)	27(1)
C12	3153(1)	4394(2)	2524(1)	23(1)
C13	3199(1)	4842(2)	1511(1)	25(1)
C14	4031(1)	5653(2)	1714(1)	28(1)
C15	4479(1)	5672(2)	2843(1)	28(1)
N16	3930(1)	4907(2)	3321(1)	26(1)
C17	2480(1)	4598(2)	410(1)	33(1)
C18	4352(1)	6389(3)	862(2)	43(1)
C19	5329(1)	6373(2)	3475(2)	37(1)
O20	5691(1)	6307(2)	4472(1)	52(1)

Table 3. Bond lengths [Å] and angles [°] for 02006.

N(1)-C(2)	1.285(2)	C(10)-C(11)	1.508(2)
N(1)-C(5)	1.4270(19)	C(12)-N(16)	1.364(2)
C(2)-C(3)	1.526(2)	C(12)-C(13)	1.398(2)
C(2)-S(6)	1.7369(17)	C(13)-C(14)	1.409(2)
C(3)-C(9)	1.523(3)	C(13)-C(17)	1.497(2)
C(3)-C(8)	1.535(3)	C(14)-C(15)	1.394(2)
C(3)-C(4)	1.545(2)	C(14)-C(18)	1.500(2)
C(4)-C(5)	1.512(2)	C(15)-N(16)	1.379(2)
C(5)-C(10)	1.343(2)	C(15)-C(19)	1.424(2)
S(6)-C(7)	1.7911(19)	C(19)-O(20)	1.225(2)
C(10)-C(12)	1.476(2)		

C(2)-N(1)-C(5)	106.99(13)	C(5)-C(10)-C(11)	121.46(14)
N(1)-C(2)-C(3)	116.23(14)	C(12)-C(10)-C(11)	116.20(13)
N(1)-C(2)-S(6)	125.08(13)	N(16)-C(12)-C(13)	108.06(13)
C(3)-C(2)-S(6)	118.62(12)	N(16)-C(12)-C(10)	119.84(14)
C(9)-C(3)-C(2)	113.93(17)	C(13)-C(12)-C(10)	131.92(14)
C(9)-C(3)-C(8)	111.41(18)	C(12)-C(13)-C(14)	107.41(14)
C(2)-C(3)-C(8)	108.92(16)	C(12)-C(13)-C(17)	126.94(14)
C(9)-C(3)-C(4)	113.61(16)	C(14)-C(13)-C(17)	125.57(15)
C(2)-C(3)-C(4)	98.30(13)	C(15)-C(14)-C(13)	107.19(14)
C(8)-C(3)-C(4)	109.91(16)	C(15)-C(14)-C(18)	127.07(15)
C(5)-C(4)-C(3)	102.10(13)	C(13)-C(14)-C(18)	125.71(16)
C(10)-C(5)-N(1)	121.46(14)	N(16)-C(15)-C(14)	107.96(14)
C(10)-C(5)-C(4)	129.89(14)	N(16)-C(15)-C(19)	122.15(16)
N(1)-C(5)-C(4)	108.54(12)	C(14)-C(15)-C(19)	129.85(16)
C(2)-S(6)-C(7)	102.73(8)	C(12)-N(16)-C(15)	109.37(14)
C(5)-C(10)-C(12)	122.33(14)	O(20)-C(19)-C(15)	126.05(17)

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for 02006. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U_{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U_{12}\ ]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
N1	22(1)	24(1)	31(1)	0(1)	10(1)	-1(1)
C2	31(1)	27(1)	34(1)	-1(1)	13(1)	-4(1)
C3	47(1)	24(1)	46(1)	-5(1)	28(1)	-4(1)
C4	36(1)	24(1)	36(1)	-5(1)	19(1)	-4(1)
C5	22(1)	24(1)	26(1)	1(1)	9(1)	1(1)
S6	51(1)	29(1)	59(1)	-8(1)	32(1)	-15(1)
C7	30(1)	36(1)	43(1)	2(1)	16(1)	-5(1)
C9	89(2)	36(1)	62(1)	-21(1)	49(1)	-24(1)
C10	20(1)	23(1)	23(1)	3(1)	7(1)	2(1)
C8	56(1)	38(1)	72(2)	11(1)	38(1)	16(1)
C11	28(1)	27(1)	27(1)	-3(1)	12(1)	-2(1)
C12	21(1)	20(1)	27(1)	-1(1)	7(1)	1(1)
C13	26(1)	22(1)	27(1)	-2(1)	10(1)	0(1)
C14	27(1)	26(1)	32(1)	0(1)	12(1)	0(1)
C15	24(1)	27(1)	34(1)	3(1)	10(1)	-1(1)
N16	22(1)	28(1)	25(1)	1(1)	6(1)	-3(1)
C17	37(1)	31(1)	28(1)	0(1)	7(1)	-5(1)
C18	43(1)	51(1)	39(1)	3(1)	21(1)	-11(1)
C19	25(1)	41(1)	41(1)	10(1)	6(1)	-8(1)
O20	36(1)	66(1)	40(1)	15(1)	-4(1)	-19(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for 02006.

	x	y	z	U(eq)
H4A	2833	1342	1533	36
H4B	1783	1584	750	36
H7A	-319	-1088	3678	53
H7B	-240	560	3112	53
H7C	585	-33	4164	53
H9A	1350	-2475	1045	85
H9B	1966	-1654	434	85
H9C	979	-948	296	85
H8A	3084	-436	3227	76
H8B	3250	-1405	2257	76
H8C	2634	-2147	2897	76
H11A	1684	3826	3860	40
H11B	1955	5505	3491	40
H11C	2709	4474	4391	40
H17A	2694	5030	-157	49
H17B	1917	5140	384	49
H17C	2358	3464	283	49
H17D	1952	4060	497	49
H17E	2730	3949	-44	49
H17F	2288	5626	57	49
H18A	4963	6841	1214	64
H18B	3926	7226	481	64
H18C	4377	5580	334	64
H19A	5645	6945	3088	45
H16A	4101(14)	4720(20)	4006(18)	34(5)

Table 6. Torsion angles [°] for 02006.

C5-N1-C2-C3	2.3(2)	C11-C10-C12-N16	45.23(19)
C5-N1-C2-S6	179.01(13)	C5-C10-C12-C13	52.2(2)
N1-C2-C3-C9	-139.15(18)	C11-C10-C12-C13	-129.31(17)
S6-C2-C3-C9	43.9(2)	N16-C12-C13-C14	-0.21(17)
N1-C2-C3-C8	95.79(19)	C10-C12-C13-C14	174.81(15)
S6-C2-C3-C8	-81.15(18)	N16-C12-C13-C17	-176.94(15)
N1-C2-C3-C4	-18.7(2)	C10-C12-C13-C17	-1.9(3)
S6-C2-C3-C4	164.40(13)	C12-C13-C14-C15	0.51(18)
C9-C3-C4-C5	145.94(17)	C17-C13-C14-C15	177.30(15)
C2-C3-C4-C5	25.20(17)	C12-C13-C14-C18	-177.99(16)
C8-C3-C4-C5	-88.46(17)	C17-C13-C14-C18	-1.2(3)
C2-N1-C5-C10	-160.37(15)	C13-C14-C15-N16	-0.63(18)
C2-N1-C5-C4	16.21(18)	C18-C14-C15-N16	177.85(16)
C3-C4-C5-C10	149.13(17)	C13-C14-C15-C19	-178.22(17)
C3-C4-C5-N1	-27.07(17)	C18-C14-C15-C19	0.3(3)
N1-C2-S6-C7	-5.09(18)	C13-C12-N16-C15	-0.18(17)
C3-C2-S6-C7	171.56(14)	C10-C12-N16-C15	-175.91(13)
N1-C5-C10-C12	176.40(13)	C14-C15-N16-C12	0.51(18)
C4-C5-C10-C12	0.6(3)	C19-C15-N16-C12	178.33(15)
N1-C5-C10-C11	-2.1(2)	N16-C15-C19-O20	4.5(3)
C4-C5-C10-C11	-177.84(15)	C14-C15-C19-O20	-178.17(19)
C5-C10-C12-N16	-133.30(16)		

Table 7. Hydrogen bonds for 02006 [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N16-H16A...O20#1	0.85(2)	2.08(2)	2.914(2)	164.0(19)

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

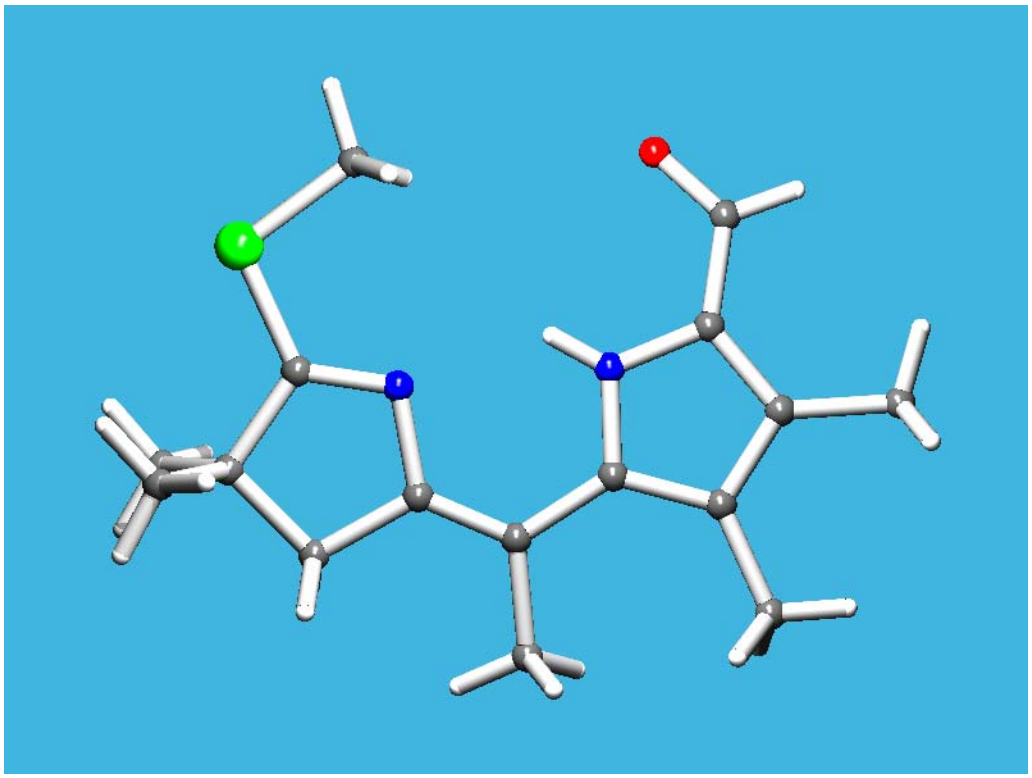
REFERENCE NUMBER: **02057**

CRYSTAL STRUCTURE REPORT

$C_{32}\ H_{44}\ N_4\ O_2\ S_2$

Report prepared for: W. Roberts/P. Jacobi, Dartmouth College.

26th February 2002



Neil R. Brooks

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Department of Chemistry

University of Minnesota

207 Pleasant St. S.E.

Minneapolis, MN 55455

Data collection

A crystal (approximate dimensions 0.30 x 0.26 x 0.20 mm^3) was placed onto the tip of a 0.1 mm diameter glass capillary and mounted on a Siemens CCD area detector diffractometer for a data collection at 173(2) K. A preliminary set of cell constants was calculated from reflections harvested from three sets of 20 frames. These initial sets of frames were oriented such that orthogonal wedges of reciprocal space were surveyed. This produced initial orientation matrices determined from 64 reflections. The data collection was carried out using MoKα radiation (graphite monochromator) with a frame time of 10 seconds and a detector distance of 4.9 cm. The complete sphere of reciprocal space was surveyed to a resolution of 0.84 Å. Four major sections of frames were collected with 0.30º steps in ω at 3 different ϕ settings and a detector position of -28º in 2θ. The intensity data were corrected for absorption and decay (SADABS).[1] Final cell constants were calculated from the xyz centroids of 2629 strong reflections from the actual data collection after integration (SAINT 6.01, 1999).[2] Please refer to Table 1 for additional crystal and refinement information.

Structure solution and refinement

The structure was solved using SIR92[3] and refined using SHELXL-97.[4] The space group *P*-1 was determined based on intensity statistics. A direct-methods solution was calculated which provided most non-hydrogen atoms from the E-map. Full-matrix least squares / difference Fourier cycles were performed which located the remaining non-hydrogen atoms. All non-hydrogen atoms were refined with anisotropic displacement parameters. All hydrogen atoms connected to carbon were placed in ideal positions and refined as riding atoms with relative isotropic displacement parameters. The hydrogen atoms connected to nitrogen were found in the difference map and allowed to refine freely. The final full matrix least squares refinement converged to R1 = 0.0348 and wR2 = 0.0955 (F^2, all data). The program PLATON[5] was used for checking the structure.

Structure description

The structure is the one suggested. There are two molecules in the asymmetric unit.

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

[1] An empirical correction for absorption anisotropy, R. Blessing, *Acta Cryst.* **A51**, 33-38 (1995).

[2] SAINT V6.1, Bruker Analytical X-Ray Systems, Madison, WI.

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[5] A.L. Spek, *Acta. Cryst.* **A46**, C34 (1990). PLATON, A Multipurpose Crystallographic Tool, Utrecht University, Utrecht, The Netherlands, A. L. Spek (2000).

Some equations of interest:

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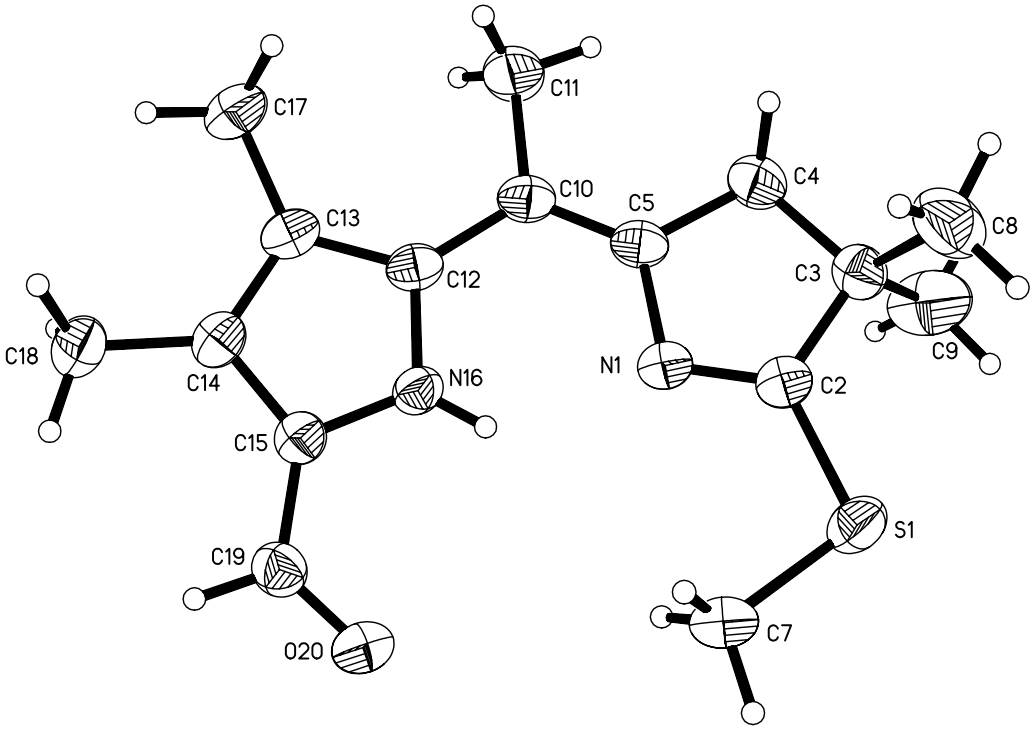
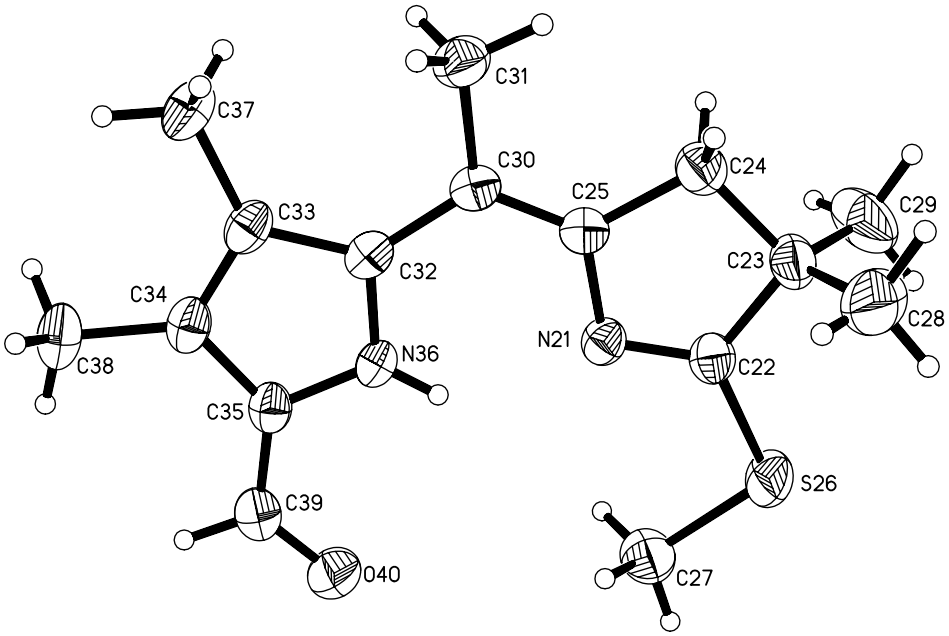
$$R_1 = \Sigma ||F_o| - |F_c|| \,/\, \Sigma |F_o|$$

$$wR2 = [\Sigma[w(F_o^2 - F_c^2)^2] \,/\, \Sigma\, [w(F_o^2)^2]]^{1/2}$$

$$\text{where } w = q \,/\, [\sigma^2 (F_o^2) + (a*P)^2 + b*P + d + e*\sin(\theta)]$$

$$\text{GooF} = S = [\Sigma[w(F_o^2 - F_c^2)^2] \,/\, (n-p)]^{1/2}$$

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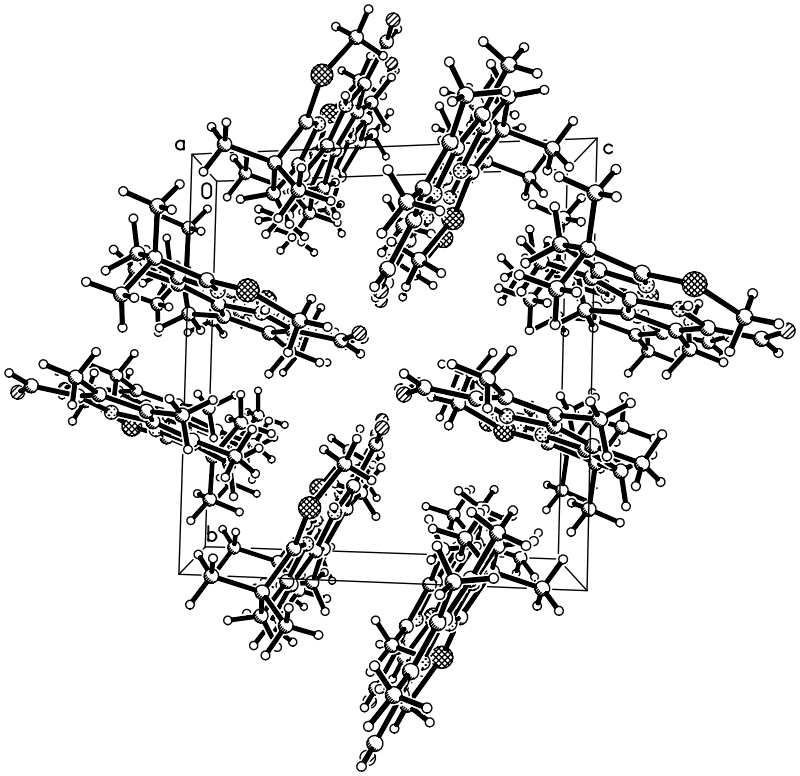
a
c
O
b

Table 1. Crystal data and structure refinement for 02057.

Identification code	02057	
Empirical formula	$C_{32}\ H_{44}\ N_4\ O_2\ S_2$	
Formula weight	580.83	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	*P*-1	
Unit cell dimensions	*a*= 11.9434(12) Å	α= 89.733(2)°
	b= 11.9627(12) Å	β= 62.709(2)°
	c= 12.4894(13) Å	γ = 86.435(2)°
Volume	1582.2(3) $Å^3$	
Z	2	
Density (calculated)	1.219 Mg/m^3	
Absorption coefficient	0.203 mm^{-1}	
F(000)	624	
Crystal color, morphology	yellow, block	
Crystal size	0.30 x 0.26 x 0.20 mm^3	
Theta range for data collection	1.71 to 25.07°.	
Index ranges	-14 = *h* = 14, -14 = *k* = 14, -14 = *l* = 14	
Reflections collected	11902	
Independent reflections	5587 [*R*(int) = 0.018]	
Observed reflections	4428	
Completeness to theta = 25.07°	99.5%	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.946 and 0.895	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5587 / 0 / 382	
Goodness-of-fit on F^2	1.047	
Final *R* indices [*I*>2sigma(*I*)]	*R*1 = 0.0348, *wR*2 = 0.0886	
R indices (all data)	*R*1 = 0.0488, *wR*2 = 0.0955	
Extinction coefficient	0.0035(9)	
Largest diff. peak and hole	0.228 and -0.178 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for 02057. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
S1	4164(1)	3392(1)	1795(1)	43(1)
N1	2479(1)	3347(1)	954(1)	33(1)
C2	3635(2)	3136(1)	746(2)	32(1)
C3	4538(2)	2646(1)	-495(2)	37(1)
C4	3618(2)	2499(2)	-1025(2)	42(1)
C5	2358(2)	3020(1)	-79(2)	34(1)
C7	2770(2)	4046(2)	3016(2)	53(1)
C8	5513(2)	3482(2)	-1201(2)	66(1)
C9	5174(3)	1526(2)	-408(2)	75(1)
C10	1266(2)	3142(1)	-153(2)	35(1)
C11	1258(2)	2764(2)	-1302(2)	52(1)
C12	75(2)	3602(1)	826(2)	32(1)
C13	-1122(2)	3871(1)	895(2)	34(1)
C14	-1931(2)	4265(1)	2077(2)	35(1)
C15	-1235(2)	4219(1)	2722(2)	32(1)
N16	-34(1)	3817(1)	1946(1)	32(1)
C17	-1536(2)	3794(2)	-77(2)	46(1)
C18	-3292(2)	4675(2)	2552(2)	44(1)
C19	-1588(2)	4462(1)	3963(2)	37(1)
O20	-889(1)	4297(1)	4429(1)	49(1)
N21	1467(1)	722(1)	6776(1)	30(1)
C22	290(2)	681(1)	7063(2)	33(1)
C23	-366(2)	-308(2)	7809(2)	39(1)
C24	758(2)	-955(2)	7872(2)	39(1)
C25	1844(2)	-213(1)	7271(1)	30(1)
S26	-559(1)	1682(1)	6670(1)	48(1)
C27	608(2)	2655(2)	5852(2)	50(1)
C28	-1368(2)	97(2)	9061(2)	70(1)
C29	-927(2)	-998(2)	7168(3)	71(1)
C30	3011(2)	-380(1)	7191(1)	30(1)
C31	3305(2)	-1400(2)	7757(2)	41(1)

C32	4009(2)	393(1)	6595(1)	29(1)
C33	5269(2)	371(1)	6413(2)	33(1)
C34	5773(2)	1356(2)	5834(2)	33(1)
C35	4842(2)	1963(1)	5652(1)	31(1)
N36	3795(1)	1356(1)	6118(1)	29(1)
C37	6009(2)	-536(2)	6712(2)	45(1)
C38	7089(2)	1701(2)	5463(2)	46(1)
C39	4855(2)	2998(2)	5087(2)	38(1)
O40	3980(1)	3429(1)	4934(1)	47(1)

Table 3. Bond lengths [Å] and angles [°] for 02057.

S(1)-C(2)	1.7321(17)	N(21)-C(22)	1.286(2)
S(1)-C(7)	1.797(2)	N(21)-C(25)	1.421(2)
N(1)-C(2)	1.291(2)	C(22)-C(23)	1.523(2)
N(1)-C(5)	1.420(2)	C(22)-S(26)	1.7334(17)
C(2)-C(3)	1.518(2)	C(23)-C(28)	1.526(3)
C(3)-C(8)	1.526(3)	C(23)-C(29)	1.529(3)
C(3)-C(9)	1.527(3)	C(23)-C(24)	1.540(2)
C(3)-C(4)	1.538(2)	C(24)-C(25)	1.510(2)
C(4)-C(5)	1.520(2)	C(25)-C(30)	1.353(2)
C(5)-C(10)	1.348(2)	S(26)-C(27)	1.796(2)
C(10)-C(12)	1.461(2)	C(30)-C(32)	1.465(2)
C(10)-C(11)	1.511(2)	C(30)-C(31)	1.510(2)
C(12)-N(16)	1.369(2)	C(32)-N(36)	1.359(2)
C(12)-C(13)	1.410(2)	C(32)-C(33)	1.414(2)
C(13)-C(14)	1.406(2)	C(33)-C(34)	1.398(2)
C(13)-C(17)	1.510(2)	C(33)-C(37)	1.509(2)
C(14)-C(15)	1.397(2)	C(34)-C(35)	1.395(2)
C(14)-C(18)	1.501(2)	C(34)-C(38)	1.506(2)
C(15)-N(16)	1.368(2)	C(35)-N(36)	1.368(2)
C(15)-C(19)	1.433(2)	C(35)-C(39)	1.420(2)
C(19)-O(20)	1.224(2)	C(39)-O(40)	1.228(2)

C(2)-S(1)-C(7)	101.72(9)	C(10)-C(5)-N(1)	123.50(15)
C(2)-N(1)-C(5)	108.89(14)	C(10)-C(5)-C(4)	127.18(15)
N(1)-C(2)-C(3)	116.19(15)	N(1)-C(5)-C(4)	109.29(14)
N(1)-C(2)-S(1)	123.20(13)	C(5)-C(10)-C(12)	123.77(15)
C(3)-C(2)-S(1)	120.61(13)	C(5)-C(10)-C(11)	118.58(16)
C(2)-C(3)-C(8)	110.00(15)	C(12)-C(10)-C(11)	117.64(15)
C(2)-C(3)-C(9)	111.28(15)	N(16)-C(12)-C(13)	106.75(15)
C(8)-C(3)-C(9)	111.19(19)	N(16)-C(12)-C(10)	120.99(15)
C(2)-C(3)-C(4)	100.57(14)	C(13)-C(12)-C(10)	132.23(15)
C(8)-C(3)-C(4)	111.64(16)	C(14)-C(13)-C(12)	107.48(14)
C(9)-C(3)-C(4)	111.72(16)	C(14)-C(13)-C(17)	123.35(16)
C(5)-C(4)-C(3)	104.75(14)	C(12)-C(13)-C(17)	129.16(16)

C(15)-C(14)-C(13)	107.63(15)	C(30)-C(25)-C(24)	126.95(15)
C(15)-C(14)-C(18)	126.31(16)	N(21)-C(25)-C(24)	109.59(13)
C(13)-C(14)-C(18)	126.05(16)	C(22)-S(26)-C(27)	102.35(8)
N(16)-C(15)-C(14)	107.27(15)	C(25)-C(30)-C(32)	123.24(14)
N(16)-C(15)-C(19)	120.92(15)	C(25)-C(30)-C(31)	118.91(15)
C(14)-C(15)-C(19)	131.75(16)	C(32)-C(30)-C(31)	117.84(14)
C(15)-N(16)-C(12)	110.86(14)	N(36)-C(32)-C(33)	106.92(14)
O(20)-C(19)-C(15)	124.45(17)	N(36)-C(32)-C(30)	121.14(14)
C(22)-N(21)-C(25)	108.72(14)	C(33)-C(32)-C(30)	131.92(15)
N(21)-C(22)-C(23)	116.28(15)	C(34)-C(33)-C(32)	107.22(14)
N(21)-C(22)-S(26)	123.84(13)	C(34)-C(33)-C(37)	123.88(15)
C(23)-C(22)-S(26)	119.86(12)	C(32)-C(33)-C(37)	128.85(16)
C(22)-C(23)-C(28)	110.29(16)	C(35)-C(34)-C(33)	107.88(14)
C(22)-C(23)-C(29)	111.33(15)	C(35)-C(34)-C(38)	125.82(16)
C(28)-C(23)-C(29)	111.55(18)	C(33)-C(34)-C(38)	126.31(16)
C(22)-C(23)-C(24)	100.28(13)	N(36)-C(35)-C(34)	107.13(15)
C(28)-C(23)-C(24)	111.11(16)	N(36)-C(35)-C(39)	121.34(15)
C(29)-C(23)-C(24)	111.79(16)	C(34)-C(35)-C(39)	131.52(16)
C(25)-C(24)-C(23)	104.80(14)	C(32)-N(36)-C(35)	110.85(14)
C(30)-C(25)-N(21)	123.46(15)	O(40)-C(39)-C(35)	125.42(16)

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for 02057. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U_{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U_{12}\ ]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
S1	46(1)	47(1)	46(1)	0(1)	-30(1)	2(1)
N1	38(1)	32(1)	30(1)	1(1)	-18(1)	-3(1)
C2	40(1)	25(1)	35(1)	5(1)	-19(1)	-3(1)
C3	38(1)	33(1)	37(1)	0(1)	-15(1)	3(1)
C4	44(1)	42(1)	36(1)	-5(1)	-15(1)	1(1)
C5	42(1)	31(1)	29(1)	-2(1)	-16(1)	-4(1)
C7	55(1)	71(1)	36(1)	-6(1)	-25(1)	-2(1)
C8	57(1)	83(2)	47(1)	1(1)	-12(1)	-28(1)
C9	102(2)	61(2)	68(2)	-19(1)	-49(2)	44(1)
C10	42(1)	35(1)	30(1)	-2(1)	-18(1)	-5(1)
C11	49(1)	72(1)	40(1)	-16(1)	-23(1)	-3(1)
C12	40(1)	29(1)	32(1)	2(1)	-20(1)	-8(1)
C13	44(1)	29(1)	37(1)	4(1)	-25(1)	-7(1)
C14	39(1)	28(1)	42(1)	4(1)	-23(1)	-4(1)
C15	35(1)	28(1)	35(1)	1(1)	-18(1)	-1(1)
N16	33(1)	33(1)	33(1)	-2(1)	-19(1)	-1(1)
C17	56(1)	47(1)	49(1)	1(1)	-36(1)	-2(1)
C18	43(1)	44(1)	51(1)	2(1)	-27(1)	2(1)
C19	38(1)	36(1)	37(1)	-4(1)	-16(1)	0(1)
O20	51(1)	62(1)	41(1)	-6(1)	-28(1)	3(1)
N21	30(1)	33(1)	31(1)	0(1)	-17(1)	-2(1)
C22	33(1)	37(1)	34(1)	0(1)	-19(1)	-4(1)
C23	32(1)	43(1)	44(1)	7(1)	-19(1)	-9(1)
C24	38(1)	39(1)	41(1)	6(1)	-18(1)	-7(1)
C25	34(1)	30(1)	27(1)	1(1)	-14(1)	-1(1)
S26	37(1)	54(1)	66(1)	16(1)	-33(1)	-5(1)
C27	52(1)	48(1)	60(1)	20(1)	-35(1)	-9(1)
C28	49(1)	65(2)	62(2)	19(1)	1(1)	6(1)
C29	83(2)	61(1)	109(2)	28(1)	-74(2)	-35(1)
C30	34(1)	29(1)	28(1)	-1(1)	-16(1)	1(1)
C31	40(1)	40(1)	44(1)	7(1)	-20(1)	2(1)

C32	31(1)	33(1)	25(1)	-5(1)	-15(1)	4(1)
C33	30(1)	42(1)	29(1)	-5(1)	-16(1)	5(1)
C34	28(1)	45(1)	28(1)	-7(1)	-14(1)	0(1)
C35	27(1)	38(1)	28(1)	-3(1)	-12(1)	-3(1)
N36	24(1)	34(1)	30(1)	0(1)	-14(1)	1(1)
C37	37(1)	55(1)	51(1)	3(1)	-27(1)	5(1)
C38	31(1)	62(1)	47(1)	-6(1)	-20(1)	-4(1)
C39	34(1)	41(1)	38(1)	0(1)	-16(1)	-6(1)
O40	45(1)	44(1)	55(1)	7(1)	-27(1)	0(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for 02057.

	x	y	z	U(eq)
H4A	3896	2889	-1797	50
H4B	3559	1696	-1164	50
H7A	2533	4753	2750	79
H7B	2939	4194	3697	79
H7C	2078	3547	3265	79
H8A	5081	4207	-1194	99
H8B	6018	3205	-2034	99
H8C	6068	3570	-827	99
H9A	5704	1644	-14	113
H9B	5700	1203	-1219	113
H9C	4527	1012	65	113
H11A	1164	3420	-1733	79
H11B	550	2292	-1105	79
H11C	2053	2334	-1812	79
H17A	-1578	3005	-254	69
H17B	-926	4141	-808	69
H17C	-2370	4185	200	69
H18A	-3609	4980	3374	66
H18B	-3788	4051	2558	66
H18C	-3367	5262	2034	66
H19A	-2422	4772	4457	45
H1	604(17)	3761(14)	2065(16)	35(5)
H21	3109(18)	1525(15)	6128(16)	36(5)
H24A	572	-1083	8720	46
H24B	952	-1689	7439	46
H27A	909	2977	6385	75
H27B	233	3255	5556	75
H27C	1319	2263	5167	75
H28A	-983	552	9437	104
H28B	-1721	-551	9559	104

H28C	-2042	549	8990	104
H29A	-1591	-540	7087	107
H29B	-1287	-1658	7641	107
H29C	-262	-1239	6367	107
H31A	2562	-1843	8122	62
H31B	3530	-1164	8380	62
H31C	4014	-1857	7137	62
H37A	6885	-338	6405	68
H37B	5992	-1248	6337	68
H37C	5631	-611	7588	68
H38A	7254	2343	4934	68
H38B	7702	1075	5032	68
H38C	7169	1910	6181	68
H39A	5602	3390	4803	45

Table 6. Hydrogen bonds for 02057 [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N16-H1...N1	0.839(18)	2.051(18)	2.692(2)	132.7(16)
N36-H21...N21	0.825(19)	2.048(18)	2.6721(19)	132.0(16)

Table 7. Torsion angles [°] for 02057.

C5-N1-C2-C3	1.51(19)	C18-C14-C15-N16	-178.69(16)
C5-N1-C2-S1	-178.83(11)	C13-C14-C15-C19	-176.52(17)
C7-S1-C2-N1	-3.69(17)	C18-C14-C15-C19	4.3(3)
C7-S1-C2-C3	175.96(14)	C14-C15-N16-C12	-0.01(19)
N1-C2-C3-C8	113.35(18)	C19-C15-N16-C12	177.42(15)
S1-C2-C3-C8	-66.32(19)	C13-C12-N16-C15	-0.50(18)
N1-C2-C3-C9	-122.96(19)	C10-C12-N16-C15	-178.65(14)
S1-C2-C3-C9	57.4(2)	N16-C15-C19-O20	-2.9(3)
N1-C2-C3-C4	-4.49(19)	C14-C15-C19-O20	173.84(18)
S1-C2-C3-C4	175.83(12)	C25-N21-C22-C23	-0.5(2)
C2-C3-C4-C5	5.24(17)	C25-N21-C22-S26	178.44(12)
C8-C3-C4-C5	-111.39(17)	N21-C22-C23-C28	114.11(19)
C9-C3-C4-C5	123.39(18)	S26-C22-C23-C28	-64.88(19)
C2-N1-C5-C10	-179.36(16)	N21-C22-C23-C29	-121.51(19)
C2-N1-C5-C4	2.33(18)	S26-C22-C23-C29	59.5(2)
C3-C4-C5-C10	176.78(17)	N21-C22-C23-C24	-3.1(2)
C3-C4-C5-N1	-4.99(18)	S26-C22-C23-C24	177.91(12)
N1-C5-C10-C12	-1.3(3)	C22-C23-C24-C25	5.05(17)
C4-C5-C10-C12	176.72(16)	C28-C23-C24-C25	-111.55(17)
N1-C5-C10-C11	179.88(16)	C29-C23-C24-C25	123.13(17)
C4-C5-C10-C11	-2.1(3)	C22-N21-C25-C30	-176.10(16)
C5-C10-C12-N16	-7.9(3)	C22-N21-C25-C24	4.12(18)
C11-C10-C12-N16	170.92(16)	C23-C24-C25-C30	174.34(16)
C5-C10-C12-C13	174.47(18)	C23-C24-C25-N21	-5.89(18)
C11-C10-C12-C13	-6.7(3)	N21-C22-S26-C27	-0.29(18)
N16-C12-C13-C14	0.80(18)	C23-C22-S26-C27	178.62(14)
C10-C12-C13-C14	178.66(17)	N21-C25-C30-C32	0.7(3)
N16-C12-C13-C17	179.95(16)	C24-C25-C30-C32	-179.56(15)
C10-C12-C13-C17	-2.2(3)	N21-C25-C30-C31	179.68(14)
C12-C13-C14-C15	-0.81(19)	C24-C25-C30-C31	-0.6(3)
C17-C13-C14-C15	179.98(16)	C25-C30-C32-N36	2.2(2)
C12-C13-C14-C18	178.39(16)	C31-C30-C32-N36	-176.77(14)
C17-C13-C14-C18	-0.8(3)	C25-C30-C32-C33	-179.74(17)
C13-C14-C15-N16	0.51(18)	C31-C30-C32-C33	1.3(3)

N36-C32-C33-C34	0.96(18)	C38-C34-C35-N36	179.83(16)
C30-C32-C33-C34	-177.30(16)	C33-C34-C35-C39	-178.30(17)
N36-C32-C33-C37	-176.53(16)	C38-C34-C35-C39	1.3(3)
C30-C32-C33-C37	5.2(3)	C33-C32-N36-C35	-0.84(18)
C32-C33-C34-C35	-0.73(18)	C30-C32-N36-C35	177.64(14)
C37-C33-C34-C35	176.91(16)	C34-C35-N36-C32	0.39(18)
C32-C33-C34-C38	179.67(16)	C39-C35-N36-C32	179.10(15)
C37-C33-C34-C38	-2.7(3)	N36-C35-C39-O40	-1.8(3)
C33-C34-C35-N36	0.23(18)	C34-C35-C39-O40	176.57(18)