

Biological mechanisms of action of novel C-10 non-acetal trioxane dimers in prostate cancer cell lines

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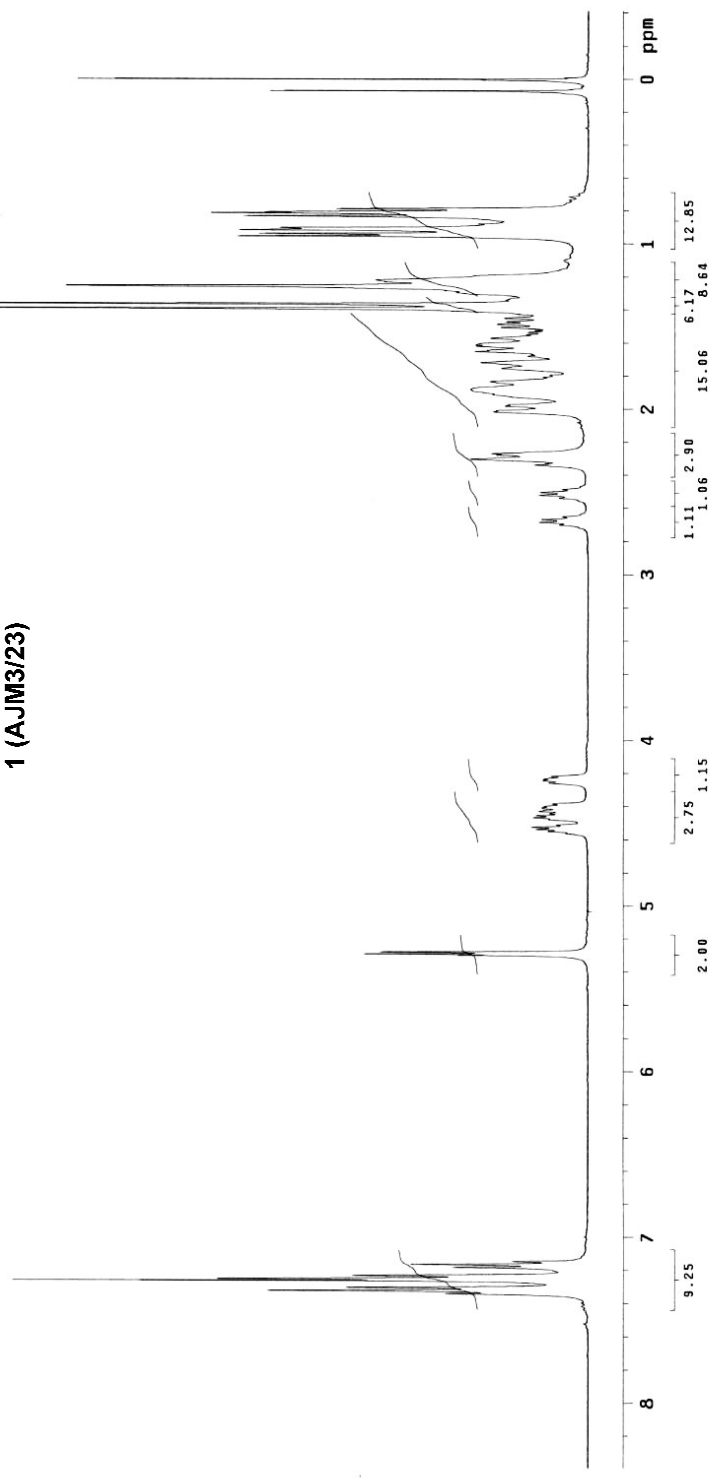
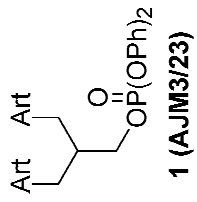
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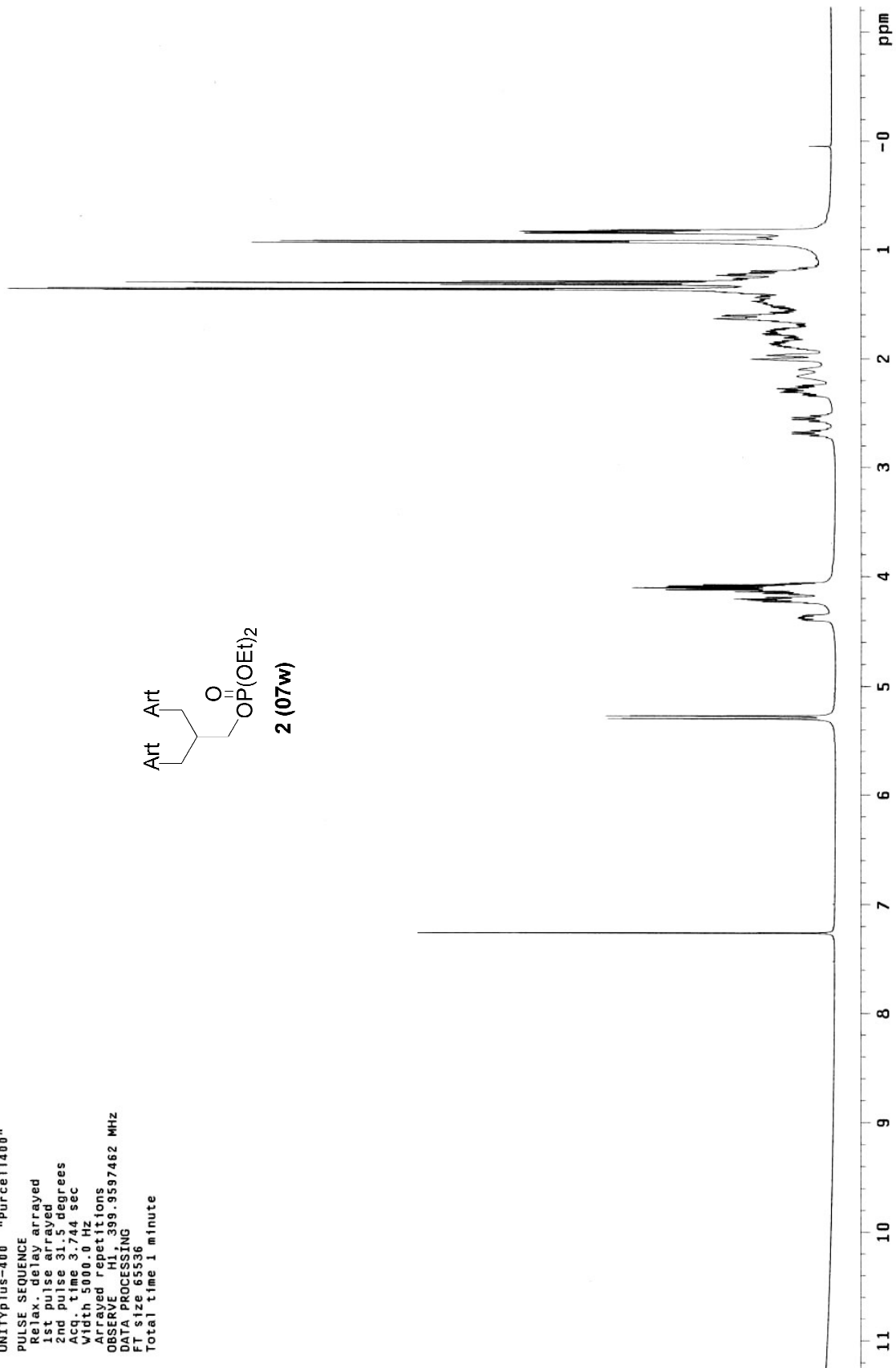
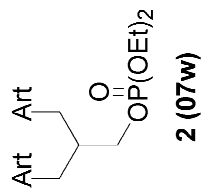
Supporting Information

| | |
|---|---------|
| ¹ H NMR Spectra..... | S1-S3 |
| ¹³ C NMR Spectra..... | S4-S6 |
| HPLC traces for 1 (AJM3/23) | S7-S8 |
| HPLC traces for 2 (GHP-TM-III-07w) | S9-S10 |
| HPLC traces for 3 (GHP-KB-06) | S11-S12 |

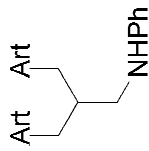
AJM3/23 c01 09/10/02
 Solvent: CDCl3
 Ambient temperature
 UNITYplus-400 "Purcell1400"
 PULSE SEQUENCE
 Relax delay arrayed
 1st pulse arrayed
 2nd pulse 31.5 degrees
 Acq. time 3.744 sec
 Width 10000.0 Hz
 Observed repetitions
 3
 DATA PROCESSING
 FT size 131072
 Total time 3 minutes



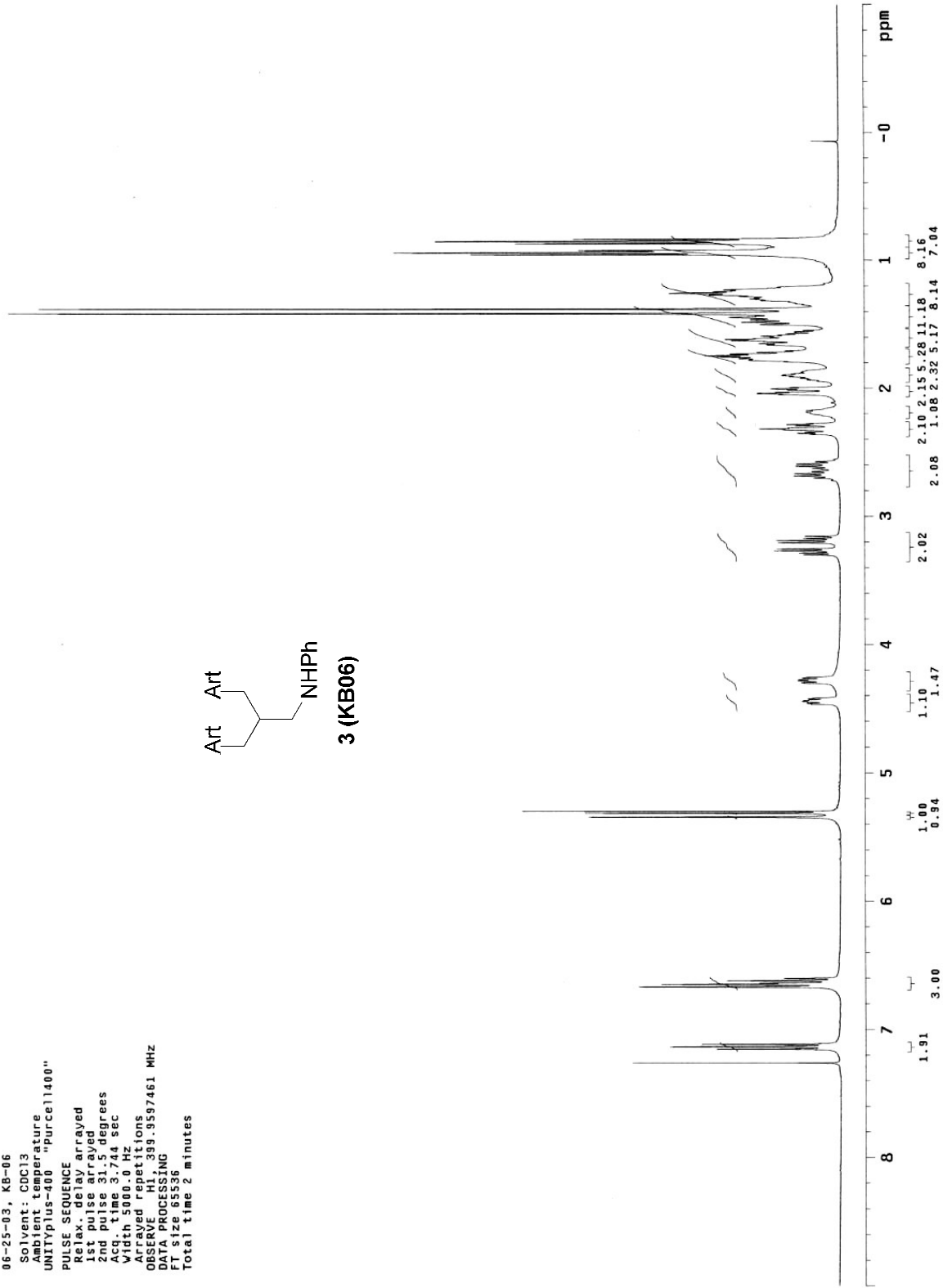
TM-III-07w
062203
Solvent: CDCl3
Ambient temperature
UNITYplus-400 "purcell400"
PULSE SEQUENCE
Relax. delay arrayed
1st pulse arrayed
2nd pulse 31.5 degrees
Acq. time 3.744 sec
Width 5000.0 Hz
Arrayed repetitions
OBSERVE H1 399.9597462 MHz
DATA PROCESSING
FT size 65536
Total time 1 minute



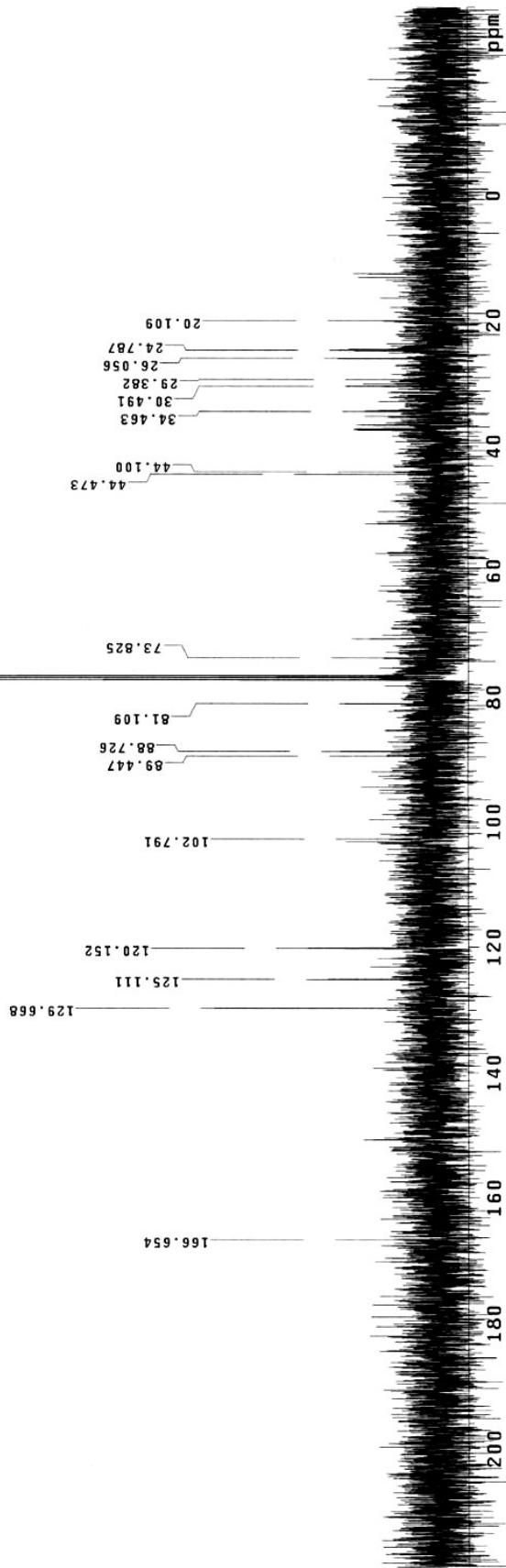
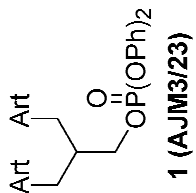
06-25-03, KB-06
 Solvent: CDCl3
 Ambient temperature
 UNITYplus-400 "Purcell1400"
 PULSE_SEQUENCE
 Relax delay arrayed
 1st pulse arrayed
 2nd pulse 31 degrees
 Acq. time 3.744 SEC
 Width 5000.0 Hz
 Arrayed repetitions
 OBSERVE H1, 399.9597461 MHZ
 DATA PROCESSING
 FT size 65536
 Total time 2 minutes



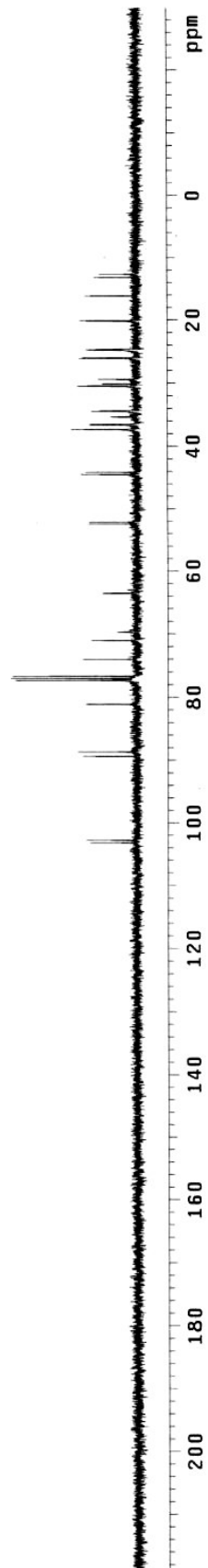
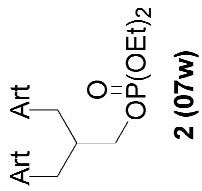
3 (KB06)



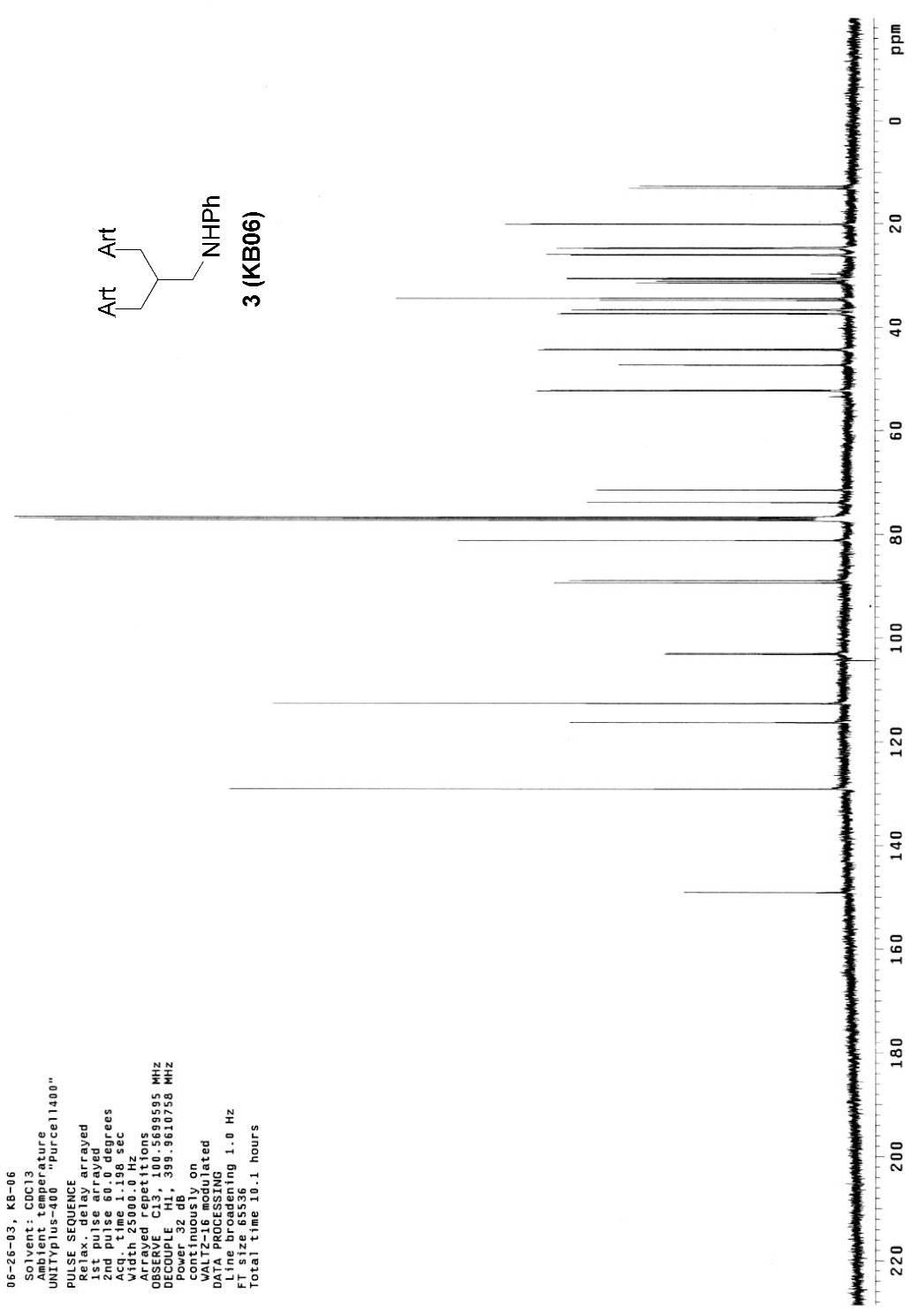
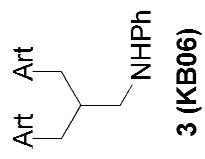
AJM3/23 co1 09/09/02
 Solvent: CDCl3
 Ambient temperature
 UNITYplus-400 "Purcell400"
 PULSE SEQUENCE
 Relax. delay arrayed
 1st pulse arrayed
 2nd pulse 60.0 degrees
 Acq. time 1.199 sec
 Width 25000.0 Hz
 Arrayed repetitions
 OBSERVE C13, 100.4541472 MHZ
 DECOUPLE H1, 399.5005548 MHZ
 Power 32 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 65536
 Total time 16 minutes



TM-III-07w
 062203
 Solvent: CDCl3
 Ambient temperature
 UNITYplus-400 "Purcell1400"
 PULSE SEQUENCE
 Relax. delay arrayed
 1st pulse arrayed
 2nd pulse 60.0 degrees
 Acq. time 1.199 sec
 Width 25000.0 Hz
 Arrayed repetitions
 OBSERVE C13, 100.5699613 MHZ
 DECOUPLE H1, 399.9610758 MHZ
 Power 32 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 65536
 Total time 8 minutes



06-26-93, KB-96
 Solvent: CDCl3
 Ambient temperature
 UNIT: plus-400 "Purcell1400"
 PULSE SEQUENCE
 Relax. delay arrayed
 2nd pulse 50.0 degrees
 Acq. time 1.198 sec
 Width 25000.0 Hz
 Arrayed repetitions
 OBSERVE C13, 100.5689595 MHz
 DECOUPLE H1, 399.9610758 MHz
 LOW PASS FILTER ON
 CONTINUOUSLY
 VALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 65536
 Total time 10.1 hours



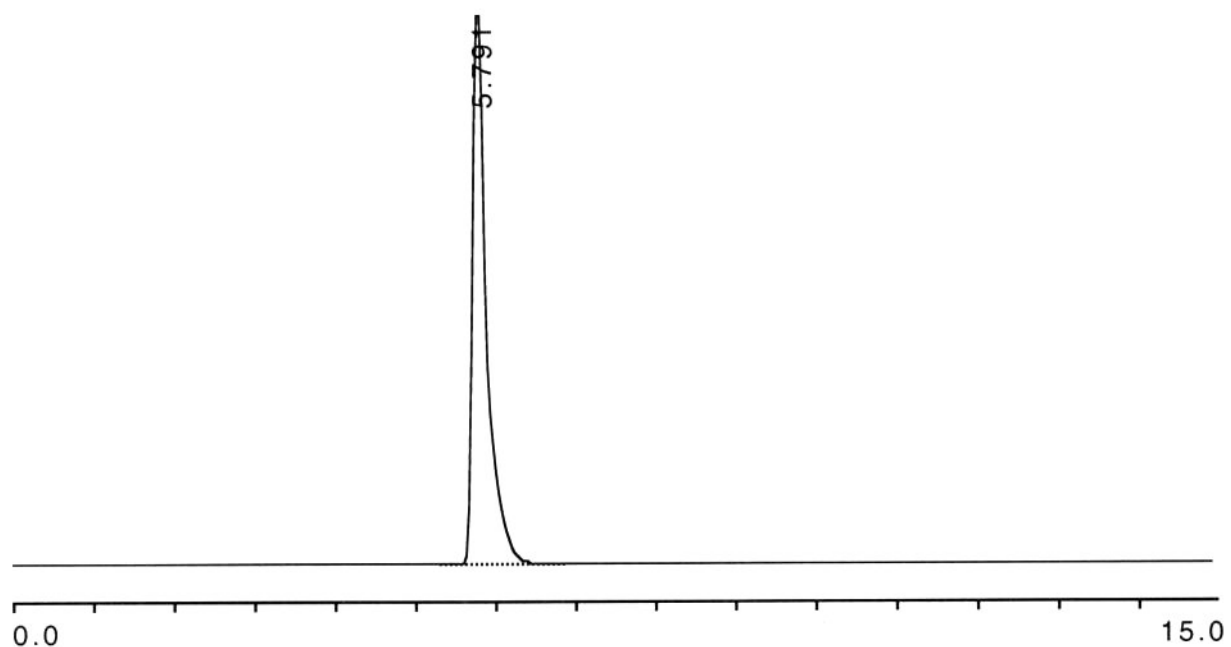
Date: Mon, Jun 19, 2006 8:54 PM

Data: 90_per_cent_EA_in_hex

Sample: 1 (AJM3/23)
New Dynamax Si Semi-Prep (1x25cm)
90% EtOAc in Hex
2.5 mL/min, 0.46 psi

Processing File: 1_2
Sampling Int: 0.1 Seconds

Data:



Analysis: Channel A

| Peak No. | Time | Type | Height(μ V) | Area(μ V-sec) | Area% |
|------------|-------|------|------------------|--------------------|----------------|
| 1 | 5.791 | Err! | 1095519 | <u>11451118</u> | <u>100.000</u> |
| Total Area | | | | 11451118 | 100.000 |

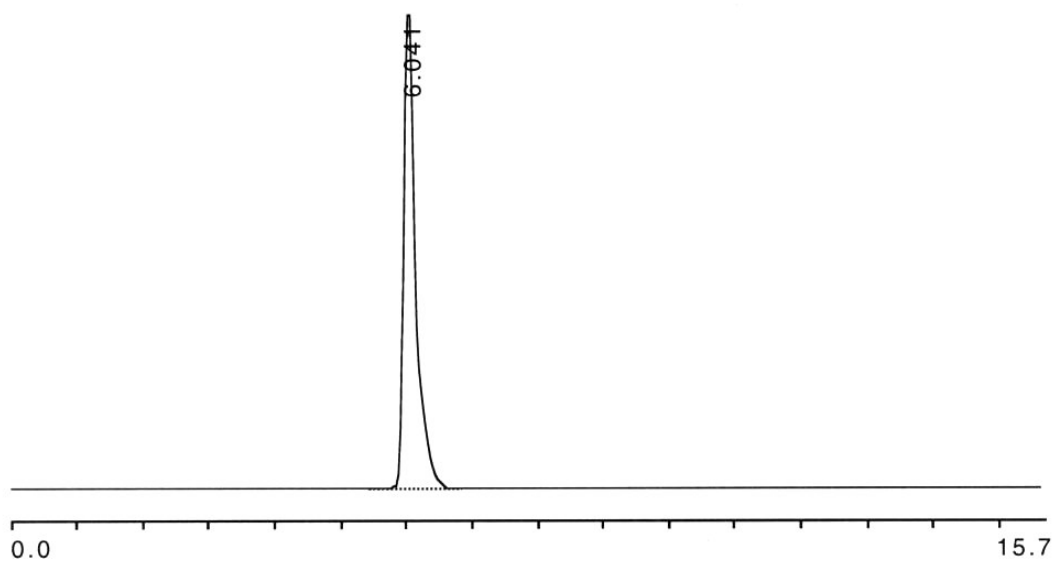
Date: Mon, Jun 19, 2006 9:39 PM

Data: 75_per_cent_EA_in_hex

Sample: 1 (AJM3/23)
New Dynamax Si Semi-Prep (1x25cm)
75% EtOAc in Hex
2.5 mL/min, 0.42 psi

Processing File: 1_1
Sampling Int: 0.1 Seconds

Data:



Analysis: Channel A

| Peak No. | Time | Type | Height(μV) | Area(μV-sec) | Area% |
|------------|-------|------|------------|--------------|---------|
| 1 | 6.041 | N9 | 571570 | 6267906 | 100.000 |
| Total Area | | | | 6267906 | 100.000 |

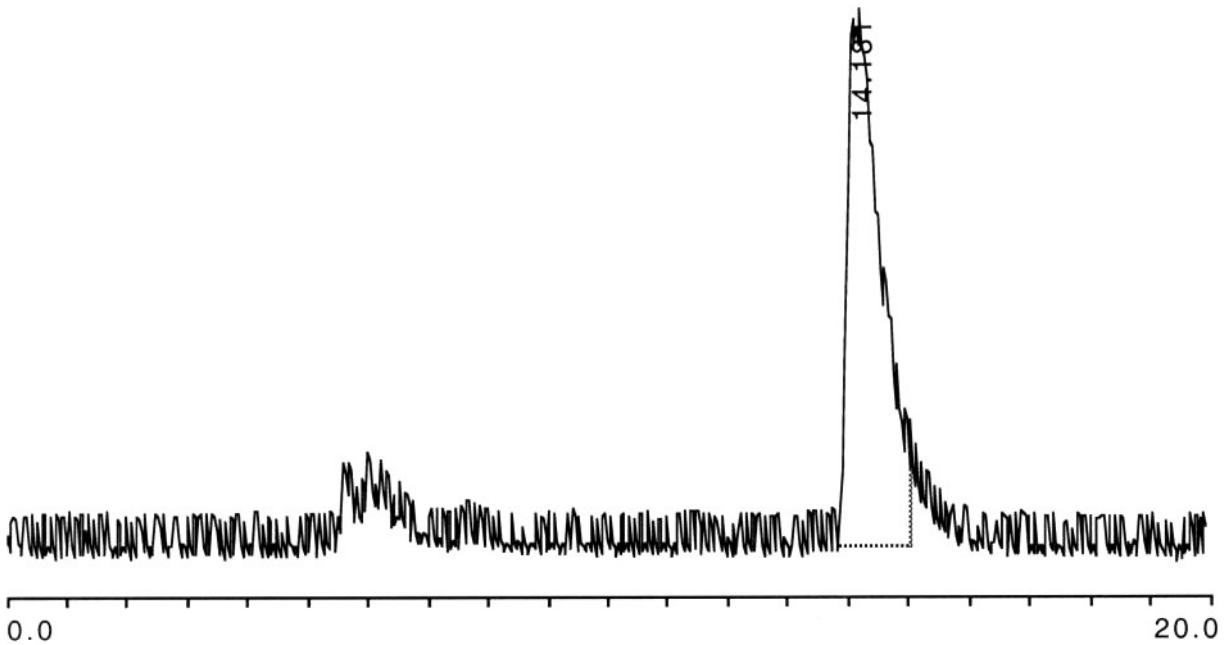
Date: Mon, Jun 19, 2006 10:50 PM

Data: 75_per_cent_trace

Sample: 2 (07w)
New Dynamax Si Semi-Prep (1x25cm)
75% EtOAc in Hex
2.5 mL/min, 0.43 psi

Processing File: 2_1
Sampling Int: 0.1 Seconds

Data:



Analysis: Channel A

| Peak No. | Time | Type | Height(μV) | Area($\mu\text{V}\cdot\text{sec}$) | Area% |
|------------|--------|------|-------------------------|--------------------------------------|---------|
| 1 | 14.181 | N1 | 10856 | 394729 | 100.000 |
| Total Area | | | | 394729 | 100.000 |

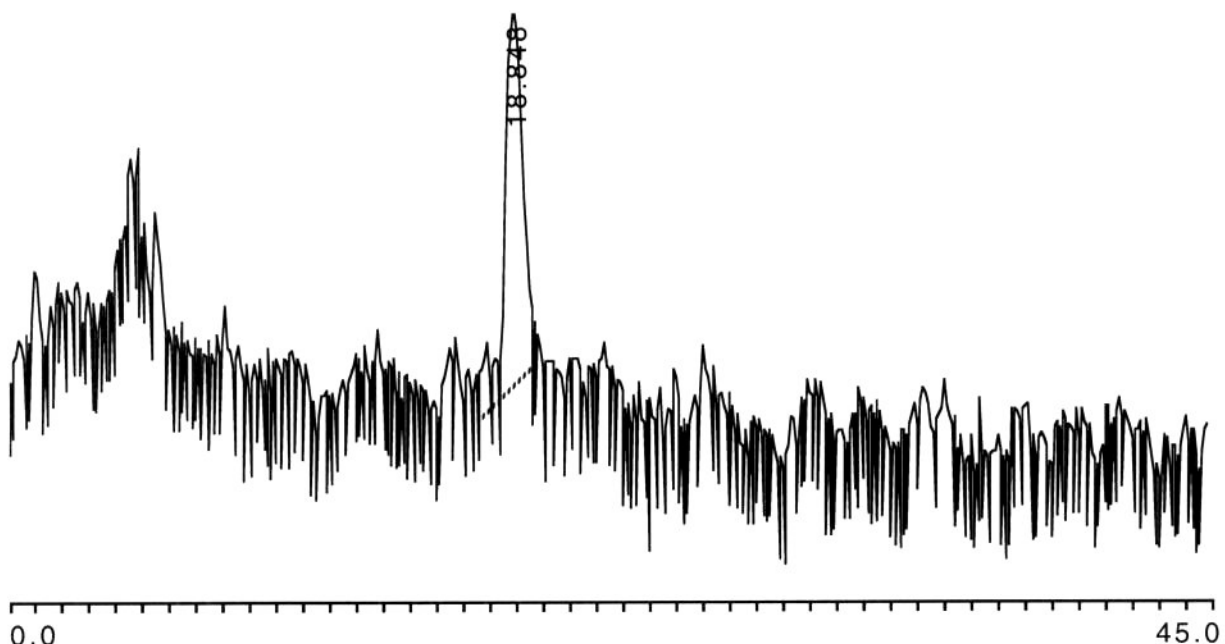
Date: Sat, Jun 28, 2003 7:10 PM

Data: TM-III-07w-062803d

Sample: 2 (07w)
Silica semi-prep column;
60% EtOAc in hexanes
Flow =3.0 mL/min, P = 0.30 kpsi
UV detector @ 254 nm

Processing File: 2_2
Sampling Int: 0.1 Seconds

Data:



Analysis: Channel A

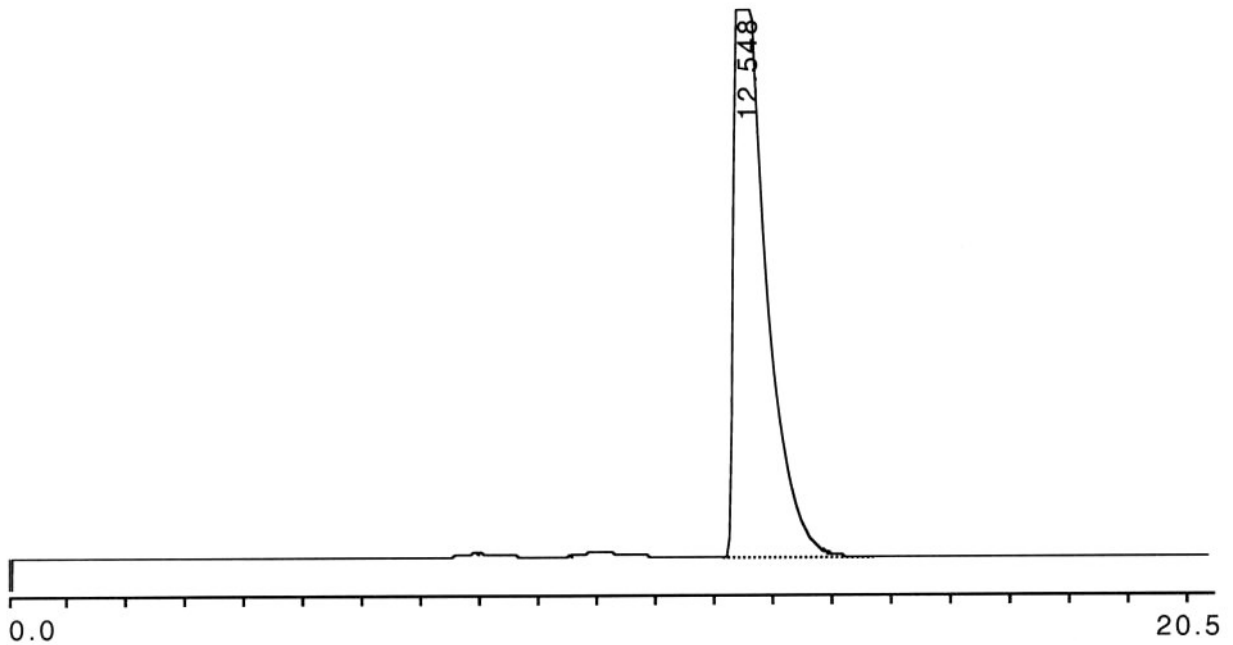
| Peak No. | Time | Type | Height(μ V) | Area(μ V-sec) | Area% |
|------------|--------|------|------------------|--------------------|----------------|
| 1 | 18.848 | N | 3286 | <u>116043</u> | <u>100.000</u> |
| Total Area | | | | 116043 | 100.000 |

Date: Mon, Jun 26, 2006 4:38 PM
Data: 20_per_cent_trace

Sample: 3 (KBO6)
Dynamax 1x25 cm
20% EtOAc/hexanes
2.5 mL/min, 0.24 kpsi, 270 nm

Processing File:
Sampling Int: 0.1 Seconds

Data:



Analysis: Channel A

| Peak No. | Time | Type | Height(μ V) | Area(μ V-sec) | Area% |
|------------|--------|------|------------------|--------------------|----------------|
| 1 | 12.548 | Err! | 1193546 | <u>44299336</u> | <u>100.000</u> |
| Total Area | | | | 44299336 | 100.000 |

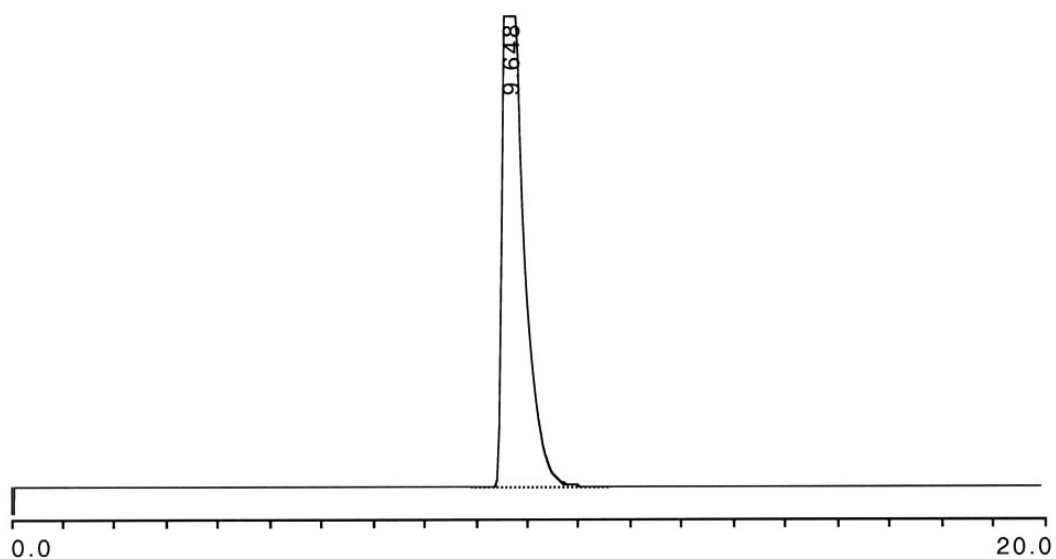
Date: Mon, Jun 26, 2006 5:09 PM

Data: 25_per_cent_trace

Sample: 3 (KBO6)
Dynamax 1x25 cm
25% EtOAc/hexanes
2.5 mL/min, 0.23 kpsi, 270 nm

Processing File:
Sampling Int: 0.1 Seconds

Data:



Analysis: Channel A

| Peak No. | Time | Type | Height(μV) | Area(μV -sec) | Area% |
|------------|-------|------|-------------------|---------------------|----------------|
| 1 | 9.648 | Err! | 1193822 | <u>33853708</u> | <u>100.000</u> |
| Total Area | | | | 33853708 | 100.000 |