Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry
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Synthesis of pyrido[2,3-*b*]indoles and pyrimidoindoles *via* Pd-catalyzed amidation and cyclization

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SUPPORTING INFORMATION

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General Information:

The procedure does not require inert atmosphere. All the amide products obtained were purified by column chromatography using silica gel (100-200 mesh). Hexane was used as a co-eluent. ^{1}H and ^{13}C NMR were recorded in 400 and 100 MHz spectrometer respectively. The chemical shifts are reported in ppm downfield to TMS (δ = 0) for ^{1}H NMR and relative to the central $CDCl_3$ resonance (δ = 77.0) for ^{13}C NMR. IR spectra were recorded on FT/IR. Elemental analyses were recorded on Flash EA 1112 analyzer in School of Chemistry, University of Hyderabad. Mass spectra were recorded on either VG7070H mass spectrometer using EI technique or LCMS-2010 mass spectrometer. Melting points were measured in open capillary tubes and are uncorrected.

General procedure for the coupling of 1-benzyl-3-chloro-1*H*-indole-2-carbaldehyde and thiophene-2-carboxamide:

An oven dried Ace Pressure tube with Teflon stir bar was charged with $Pd_2(dba)_3$ (5.7 mg, 1.85 μmol, 2.0 mol % Pd), BINAP (5.0 mg, 0.92 μmol, 0.5 mol %), thiophene-2-carboxamide (28 mg, 0.07 mmol) and base [Cs_2CO_3 (195 mg) or K_3PO_4 (127 mg) or K_2CO_3 (83 mg) or *t*-BuOK (58 mg), 1-benzyl-3-chloro-1H-indole-2-carbaldehyde (0.05g ,0.185 mmol) and *t*-BuOH (2.0 mL) and then capped with a Teflon screw cap and the mixture was heated to 110 ^{o}C with stirring for 8 h. At this point, the reaction mixture was allowed to cool to room temperature, diluted with ethyl acetate, and the resulting solution was filtered through celite, and concentrated under reduced pressure. The crude material was purified by column chromatography on silica gel (eluting with hexanes/ethyl acetate) to give the corresponding coupled product (**1e**).

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General procedure for the coupling of 3-bromo-1-ethyl-1*H*-indole-2-carbaldehyde and benzamide:

An oven dried Ace Pressure tube with Teflon stir bar was charged with $Pd_2(dba)_3$ (1.3 mg, 1.4 µmol, 1.0 mol % Pd), BINAP (0.60 mg, 0.9 µmol, 0.25 mol %), benzamide (28 mg, 0.239 mmol) and base [Cs_2CO_3 (195 mg) or K_3PO_4 (127 mg) or K_2CO_3 (83 mg) or *t*-BuOK (58 mg), 3-bromo-1-ethyl-1H-indole-2-carbaldehyde (0.05g ,0.199 mmol) and toluene (2.0 mL) and then capped with a Teflon screw cap and the mixture was heated to 110 ^oC with stirring for 8 h. At this point, the reaction mixture was allowed to cool to room temperature, diluted with ethyl acetate, and the resulting solution was filtered through celite, and concentrated under reduced pressure. The crude material was purified by column chromatography on silica gel (eluting with hexanes/ethyl acetate) to give the corresponding coupled product (**1a**).

General procedure for the coupling of 2-bromo-1-ethyl-1*H*-indole-3-carbaldehyde and thiophene-2-carboxamide:

An oven dried Ace Pressure tube with Teflon stir bar was charged with $Pd_2(dba)_3$ (0.9 mg, 0.99 µmol, 1.0 mol % Pd), BINAP (0.31 mg, 0.49 µmol, 0.25 mol %), thiophene-2-carboxamide (30 mg, 0.239 mmol) and base [Cs_2CO_3 (195 mg) or K_3PO_4 (127 mg) or K_2CO_3 (83 mg) or *t*-BuOK (58 mg), 2-bromo-1-ethyl-1H-indole-3-carbaldehyde (0.2g ,5.9 µmol) and *t*-BuOH (2.0 mL) and then capped with a Teflon screw cap and the mixture was heated to 110 ^oC with stirring for 8 h. At this point, the reaction mixture was allowed to cool to room temperature, diluted with ethyl acetate, and the resulting solution was filtered through celite, and concentrated under reduced pressure. The crude material was purified by column chromatography on silica gel (eluting with hexanes/ethyl acetate) to give the corresponding coupled product (**1f**).

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General procedure for the coupling of 3-halo-indole-2-carbaldehyde and amide:

An oven dried Ace Pressure tube with Teflon stir bar was charged with $Pd_2(dba)_3$ (if X = Cl, 1.0 mol %, 2.0 mol % Pd, if X = Br, 0.75 mol %, 1.5 mol % Pd), BINAP (0.50 mol %), amide (1.2 equiv) and Cs_2CO_3 (3.0 equiv), 3-halo-indole-2-carbaldehyde (1.0 equiv) and if X = Br, toluene (1.0 mL); X = Cl, *t*-BuOH (2.0 mL) and then capped with a Teflon screw cap and the mixture was heated to 110 ^oC with stirring according to mentioned time in Table 1. At this point, the reaction mixture was allowed to cool to room temperature, diluted with ethyl acetate, and the resulting solution was filtered through celite, and concentrated under reduced pressure. The crude material was purified by column chromatography on silica gel (eluting with hexanes/ethyl acetate) to give the corresponding coupled product.

General procedure for the coupling of 2-halo-3-carbonylindoles and amide:

An oven dried Ace Pressure tube with Teflon stir bar was charged with $Pd_2(dba)_3$ (0.50 mol %, 1.0 mol % Pd), BINAP (0.25 mol %), amide (1.2 equiv) and Cs_2CO_3 (3.0 equiv), 2-halo-3-carbonylindoles (1.0 equiv) and if X = Br, toluene (2.0 mL) ; X = Cl, *t*-BuOH (2.0 mL) and then capped with a Teflon screw cap and the mixture was heated to 110 ^oC with stirring according to mentioned time in Table 1. At this point, the reaction mixture was allowed to cool to room temperature, diluted with ethyl acetate, and the resulting solution was filtered through celite, and concentrated under reduced pressure. The crude material was purified by column chromatography on silica gel (eluting with hexanes/ethyl acetate) to give the corresponding coupled product.

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General procedure for base-promoted cyclization to 2-substitued-4-hydroxy-*α*-carbolines:

An oven dried Ace Pressure tube with Teflon stir bar was charged with *N*-(3-acetyl-1-(substituted)-1*H*-indol-2-yl)amides (1.0 equiv) and *t*-BuOK (5.0 equiv) in THF (6 mL). The pressure tube was then sealed with a Teflon screw-cap and the reaction was placed in a preheated oil bath at 110 °C. The reaction mixture was stirred for according to mentioned time in Table 2 and then removed from the oil bath and allowed to cool to room temperature. The reaction mixture was then diluted with ethyl acetate. The resultant reaction mixture was extracted with EtOAc (20 mL), washed with water (100 mL) and dried over anhydrous Na_2SO_4. The solvent was removed *in vacuo* to obtain the pure product (2**a-2j**). Hexane was added and removed twice more before the product was dried *in vacuo*.

General procedure for the one-pot synthesis of pyrimido[4,5-*b*] & [5,4-*b*]indoles (Method A):

An oven dried Ace Pressure tube with Teflon stir bar was charged with $Pd_2(dba)_3$ (1.3 mg, 1.4 μmol, 1.0 mol % Pd), BINAP (0.18 mg, 2.9 μmol, 1.5 mol %), amide (86 mg, 0.07 mmol) and base [Cs_2CO_3 (195 mg) or K_3PO_4 (127 mg) or K_2CO_3 (83 mg) or *t*-BuOK (58 mg), 3-halo-2-formylindoles or 2-halo-3-carbonylindoles (0.2g ,5.9 μmol) and *t*-BuOH (2.0 mL) and then capped with a Teflon screw cap and the mixture was heated to 110 ^oC with stirring and the reaction was monitored by TLC. When the starting material was completely consumed, the reaction mixture was cooled to <80 ^oC and $HCOONH_4$ (6.0 equiv) in *t*-BuOH (2 mL) was added in one portion. The resulting mixture was heated back to 110 ^oC and the reaction mixture was stirred for according time mentioned in Table 3. When the reaction was complete, reaction

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mixture was allowed to cool to room temperature, diluted with ethyl acetate, and the resulting solution was filtered through celite, and concentrated under reduced pressure. The crude material was purified by column chromatography on silica gel (eluting with hexanes/ethyl acetate) to give the corresponding pyrimido[4,5-*b*] & [5,4-*b*]indole product.

General procedure for the one-pot synthesis of pyrimido[4,5-*b*] & [5,4-*b*]indoles (Method B):

An oven dried Ace Pressure tube with Teflon stir bar was charged with *N*-(3-carbonyl-1-(substituted)-1*H*-indol-2-yl)amides or *N*-(1-(substituted)-2-formyl-1*H*-indol-3-yl)amides (1.0 equiv) and $HCOONH_4$ (6.0 equiv) in *t*-BuOH (6 mL). The pressure tube was then sealed with a Teflon screw-cap and the reaction was placed in a preheated oil bath at 110 °C. The reaction mixture was stirred for according to mentioned time in Table 4 and then removed from the oil bath and allowed to cool to room temperature. The reaction mixture was then diluted with ethyl acetate. The resultant reaction mixture was extracted with EtOAc (20 mL), washed with water (100 mL) and dried over anhydrous Na_2SO_4. The solvent was removed *in vacuo* to obtain the pure product (**3a-3t**). Hexane was added and removed twice more before the product was dried *in vacuo*.

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General procedure for the synthesis of dihydropyrido[4,5-*b*]indoles:

A R.B. flask with Teflon stir bar charged with *N*-(3-formyl-1-(substituted)-1*H*-indol-2-yl)amides (1.0 mmol), nitro styrene (1.0 mmol) and DABCO (0.5 mmol) were mixed under stirring and heated at 70 ^{o}C for 3h. After the reaction was over, the residue was diluted with dichloromethane, adsorbed on silicagel and subjected to column chromatography to obtain the **4a-b** in good yields.

Materials:

The starting materials 3-carbonyl-2-haloindole[1] and 2-formyl-3-haloindole[2] were prepared based on the literature procedures.

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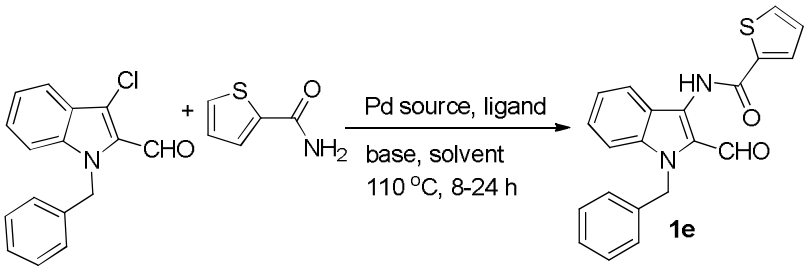
Table S1A Optimization of the Pd-catalyzed cross-coupling of 1-benzyl-3-chloro-1*H*-indole-2-carbaldehyde and thiophene-2-carboxamide [a]



Entry	Pd source-Ligand	Solvent	Base	Time (h)	Yield (%)[a]
1	$Pd_2(dba)_3$ - BINAP	*t*-BuOH	Cs_2CO_3	12	62[b]
2	$Pd_2(dba)_3$ – BINAP	DMF	Cs_2CO_3	24	-
3	$Pd_2(dba)_3$ – BINAP	DMSO	Cs_2CO_3	24	-
4	$Pd_2(dba)_3$ – BINAP	toluene	Cs_2CO_3	24	40[b]
5	$Pd_2(dba)_3$ – BINAP	toluene	K_2CO_3	24	32[b]
6	$Pd_2(dba)_3$ – PPh_3	*t*-BuOH	Cs_2CO_3	24	-
7	$Pd_2(dba)_3$ - Pcy_3	*t*-BuOH	Cs_2CO_3	24	10
8	$Pd(OAc)_2$ - BINAP	toluene	Cs_2CO_3	24	14
9	$Pd(OAc)_2$ - Pcy_3	toluene	Cs_2CO_3	24	trace
10	$PdCl_2$ - Pcy_3	toluene	Cs_2CO_3	24	-
11	$PdCl_2(PPh_3)_2$	toluene	Cs_2CO_3	24	-
12	$Pd_2(dba)_3$ - BINAP	toluene	Cs_2CO_3	20	46[c]
13	$Pd_2(dba)_3$ – BINAP	toluene	K_2CO_3	24	42
14	$Pd_2(dba)_3$ - BINAP	*t*-BuOH	K_2CO_3	10	85[c]
15	**$Pd_2(dba)_3$ - BINAP**	***t*-BuOH**	**Cs_2CO_3**	**8**	**92[c]**
16	$Pd_2(dba)_3$ – BINAP	toluene	Cs_2CO_3	15	60[c]
17	$Pd_2(dba)_3$ – BINAP	*t*-BuOH	K_3PO_4	12	83[c]

Conditions: 1-benzyl-3-chloro-1*H*-indole-2-carbaldehyde (1.0 mmol), thiophene-2-carboxamide (1.2 mmol), Pd (2.0 mol %), ligand (1.0 mol %), base (3.0 mmol), solvent (2.0 mL/mmol), 110 °C, 8-24 h. [a] isolated yields. [b] $Pd_2(dba)_3$ (0.50 mol %), BINAP (0.25 mol %), [c] $Pd_2(dba)_3$ (1.0 mol %), BINAP (0.50 mol %)

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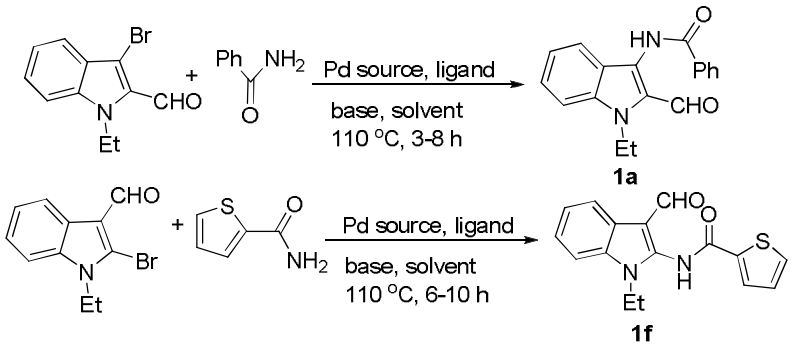
Table S1B Optimization of the Pd-catalyzed cross-coupling of 3-bromo-1-ethyl-1*H*-indole-2-carbaldehyde and benzamide, 2-bromo-1-ethyl-1*H*-indole-3-carbaldehyde and thiophene-2-carboxamide



Entry	Pd source-Ligand	Solvent	Base	Time (h)	Yield (%)[a]
1	$Pd_2(dba)_3$ - BINAP	*t*-BuOH	Cs_2CO_3	24	1a (49); 1f (62)
2	$Pd_2(dba)_3$ – BINAP	THF	Cs_2CO_3	24	1a (18); 1f (27)
3	$Pd_2(dba)_3$ – BINAP	dioxane	Cs_2CO_3	24	1a (15); 1f (21)
4	$Pd_2(dba)_3$ – BINAP	DMF	Cs_2CO_3	24	1a (-); 1f (-)
5	$Pd_2(dba)_3$ – BINAP	DMSO	Cs_2CO_3	24	1a (-); 1f (-)
6	$Pd_2(dba)_3$ - BINAP	dioxane	Cs_2CO_3	24	1a (12); 1f (20)
7	$Pd_2(dba)_3$ - PPh_3	*t*-BuOH	Cs_2CO_3	26	1a (57); 1f (62)
8	$Pd_2(dba)_3$ - Pcy_3	*t*-BuOH	Cs_2CO3	20	1a (57); 1f (62)
9	$Pd_2(dba)_3$ - Pcy_3	toluene	Cs_2CO_3	18	1a (78); 1f (83)
10	$Pd_2(dba)_3$ - BINAP	toluene	Cs_2CO_3	10	1a (81)[b];**1f(94)[b]**
11	**$Pd_2(dba)_3$ - BINAP**	**toluene**	**Cs_2CO_3**	**6**	**1a (90) [c];1f (94)[b]**
12	$Pd_2(dba)_3$ - BINAP	toluene	K_3PO_4	15	1a (51); 1f (64)
13	$Pd_2(dba)_3$ - BINAP	toluene	K_2CO_3	24	1a (56); 1f (65)
14	$Pd(OAc)_2$ - BINAP	toluene	Cs_2CO_3	24	1a (55); 1f (70)
15	$Pd(OAc)_2$ - PPh_3	toluene	K_2CO_3	24	1a (42); 1f (48)
16	$Pd(OAc)_2$ - Pcy_3	toluene	K_2CO_3	24	1a (45); 1f (53)
17	$PdCl_2$ - Pcy_3	toluene	K_2CO_3	24	1a (11); 1f (26)
18	$PdCl_2(PPh_3)_2$	toluene	Cs_2CO_3	24	1a (-); 1f (-)

Conditions: 3-bromo-1-ethyl-1*H*-indole-2-carbaldehyde (1.0 mmol), benzamide (1.2 mmol), Pd (1.5 mol %), ligand (0.75 mol %), 2-bromo-1-ethyl-1*H*-indole-3-carbaldehyde (1.0 mmol), thiophene-2-carboxamide (1.2 mmol), Pd (1 mol %), ligand (0.25 mol %), base (3.0 mmol), solvent (2.0 mL/mmol), 110 °C, 6-24 h. [a] isolated yields. [b] $Pd_2(dba)_3$ (0.50 mol %), BINAP (0.25 mol %), [c] $Pd_2(dba)_3$ (0.75 mol %), BINAP (0.50 mol %)

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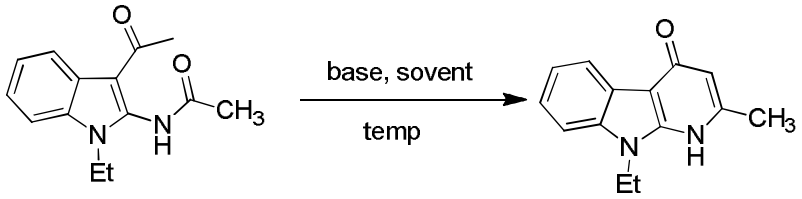
Table S2A Optimization for the synthesis of 9-ethyl-2-methyl-9*H*-pyrido[2,3-*b*]indol-4-ol [a]



Entry	Base	Solvent	Temp (°C)	Time (h)	Yield (%)[b]
1	Cs_2CO_3	*t*-BuOH	110	24	25
2	NaOH	THF	70	24	12
3	NaOH	*t*-BuOH	110	24	16
4	NaOH	DMF	110	24	-
5	NaOH	dioxane	110	24	12
6	NaOEt	toluene	110	24	-
7	Cs_2CO_3	THF	70	24	40
8	NaOMe	dioxane	110	24	-
9	NaOAc	*t*-BuOH	110	24	20
10	*t*-BuOK	MeOH	70	24	47
11	***t*-BuOK**	**THF**	**70**	**6**	**88**
12	*t*-BuOK	dioxane	110	24	42
13	*t*-BuOK	toluene	110	24	13
14	*t*-BuOK	DMF	110	24	20

[a]Conditions: *N*-(3-acetyl-1-ethyl-1*H*-indol-2-yl)acetamide (1.0 mmol) and base (5.0 mmol), solvent (2.0 mL/mmol), 70-110 °C, 6-24 h. [b] Isolated yields.

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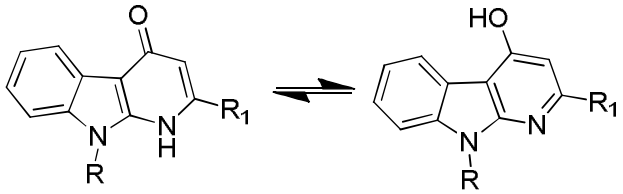
Computational Analysis:

Generally, aliphatic carbonyl compounds which have alpha hydrogens undergo *keto-enol* tautomerism; among them the *keto* form is favored over the *enol* form due to the stabilization of the C=O bond. Though, this common fashion is not obeyed by the cyclic conjugated ketone systems. In this tautomerism, the *enol* form is predominant over *keto* form albeit literature [3]also supports the existence of the *enol* form almost exclusively. This conclusion was achieved by performing electronic structure calculations on the isomers of these molecules.



Keto form enol form

Density functional theory (DFT) hybrid method with the Becke's 3 parameter exchange functional of Lee, Yang and Parr (B3LYP) and 6-31+G(D) (valence double zeta plus polarization functions of d type) basis set is used to optimize the geometries of the two tautomers of the 9-ethyl-2-methyl-1H-pyrido[2,3-b]indol-4(9H)-one. The effect of solvation also studied here. Three types (gas phase, using methanol and tetra hydro furan as solvents with polarizable continuum model (PCM) solvent model) of calculations are done on the two tautomers. All the calculations were done using Gaussian 09 package. It is well recognized that this method and basis set are reliable for organic molecules. The two structures converged to a minimum and both minima were verified by establishing that the matrix of energy second derivatives (Hessian) has only positive Eigen values (all vibrational frequencies real). The optimized keto and enol forms are depicted by **Fig S2a & S2b**. The transition state is verified by the presence of only one large negative vibrational frequency (-1671cm^{-1}). The obtained transition state is depicted in **Fig S2c**.

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The electronic and activation energies of the two tautomers and transition states in the three models of theory are tabulated in Table S2B.

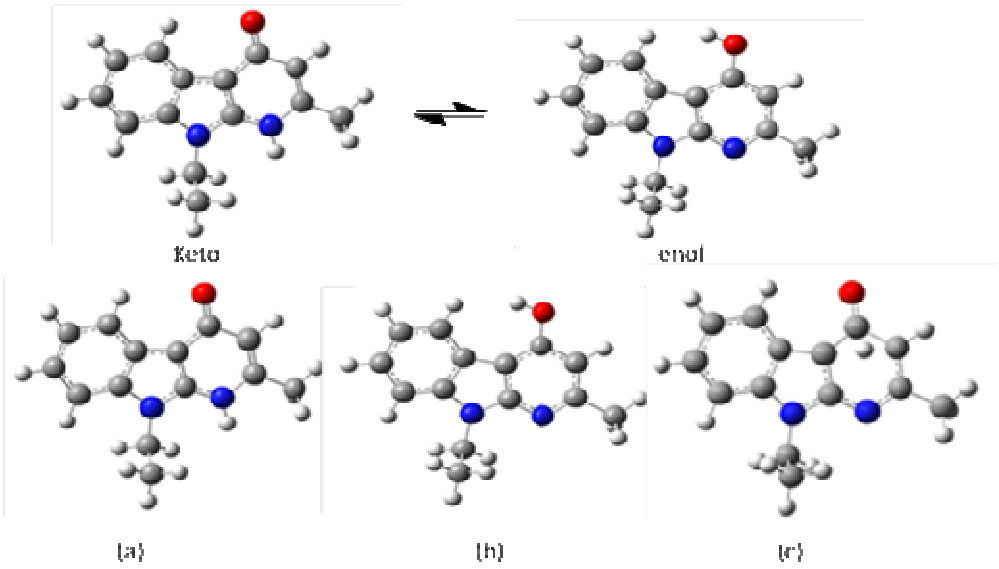

Fig S2. B3LYP and 6-31+G(D) Optimized (a) *keto* form (b) *enol* form (c) transition state

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Table S2B. The total energy of the Keto, Enol and Transition states at B3LYP/6-31+G(D) level of theory in gas phase and solvent

Method	**Keto form** (Hartree)	**Enol form** (Hartree)	**Transition State** (Hartree)	**Activation Energy** (K.cal/mol)
Gas Phase	-726.6992284	-726.7088136	-726.3832852[a]	198.255
Methanol	-726.7225416	-726.7177769	-726.6256917	60.774
THF	-726.8643483	-726.8604681	-726.7665929	61.342

[a] The transition state was optimized with B3LYP method and 6-31g basis set but reactant and product were optimized with B3LYP method and 6-31+G(D) basis. Because the transition state search with 6-31+G(D) is unsuccessful.

From the Table S2B, it can be seen that the relative energy difference between the enol forms to keto form is very small. While the enol form is stable in gas phase than keto form by 6.011 Kcal/mol, the keto form is stable than the enol form by ~3 Kcal/mol in the solvent model. This stabilization can be attributed to the hydrogen bonding by the keto group with methanol. The relative energies are very small enough to be in equilibrium with the other counterpart even at room temperature. But in our synthesis only enol form is predominant than the keto form. So, we optimized the transition state for the conversion, the energies are tabulated in Table S2B. The activation energy for keto-enol tautomerism is 198.255, 60.774, 61.342 Kcal/mol in the gas

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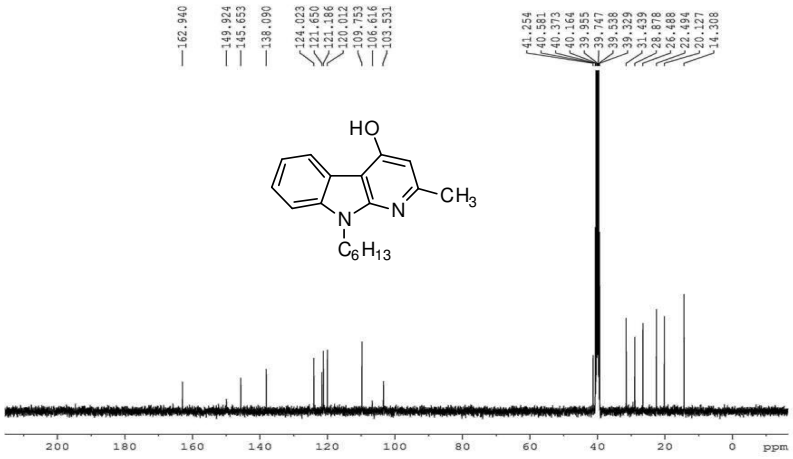
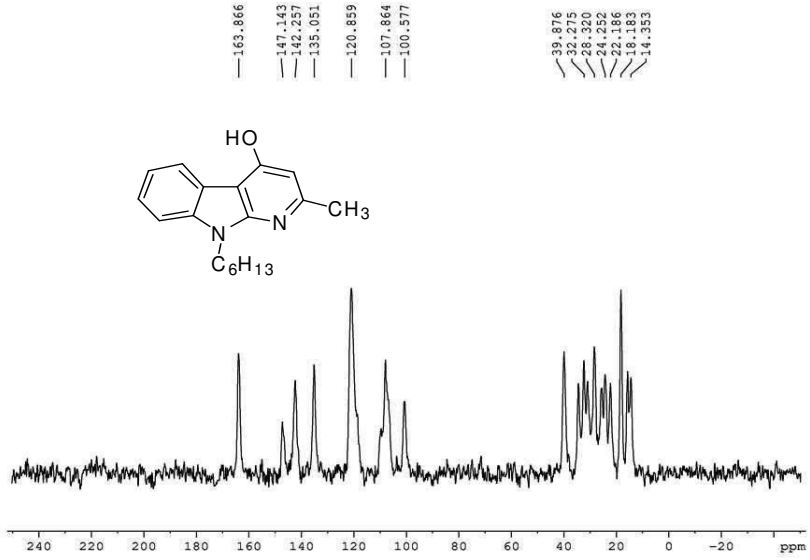
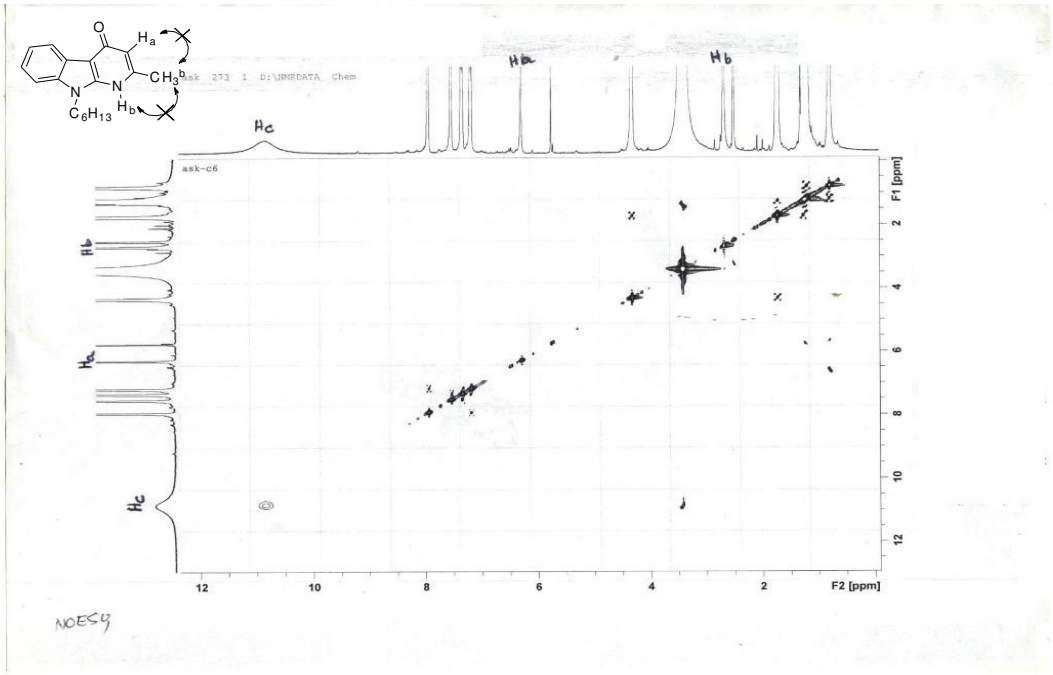
phase and solvent models respectively. These energy barriers cannot be attained with room temperature energy. So, only one form is predominant. The variable temperature (VT-PXRD) spectroscopic studies also support this statement.



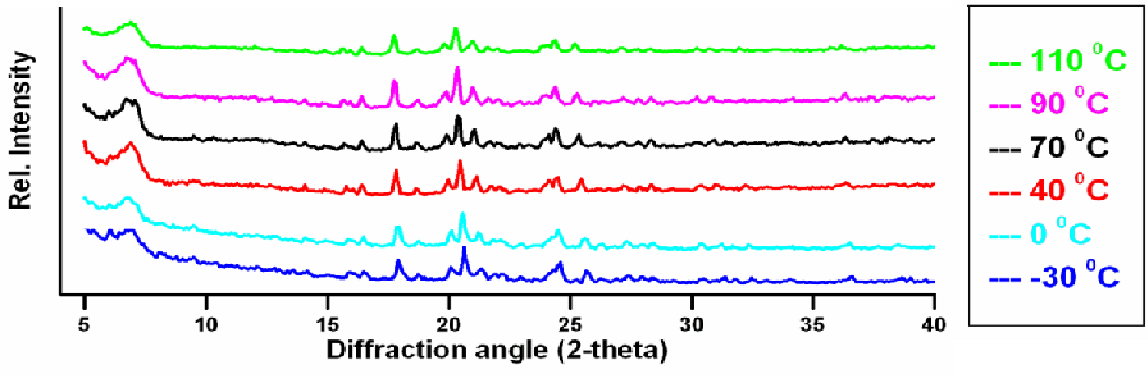
(a)



(b)



(c)



(d)

Figure S3 (a) ^{13}C NMR of 2f in liquid state (DMSO), (b) Solid State ^{13}C NMR, (c) NOESY of 2f (d) VT-PXRD patterns of 2f on -30 °C to 110 °C.

The IR spectrum of the molecule shows a broad peak at $\Delta\upsilon_{max}$ 3410 cm^{-1}. This peak was assigned as an O-H vibration. The peak cannot be assigned for the N-H bond vibrational frequency (~3300 cm^{-1})

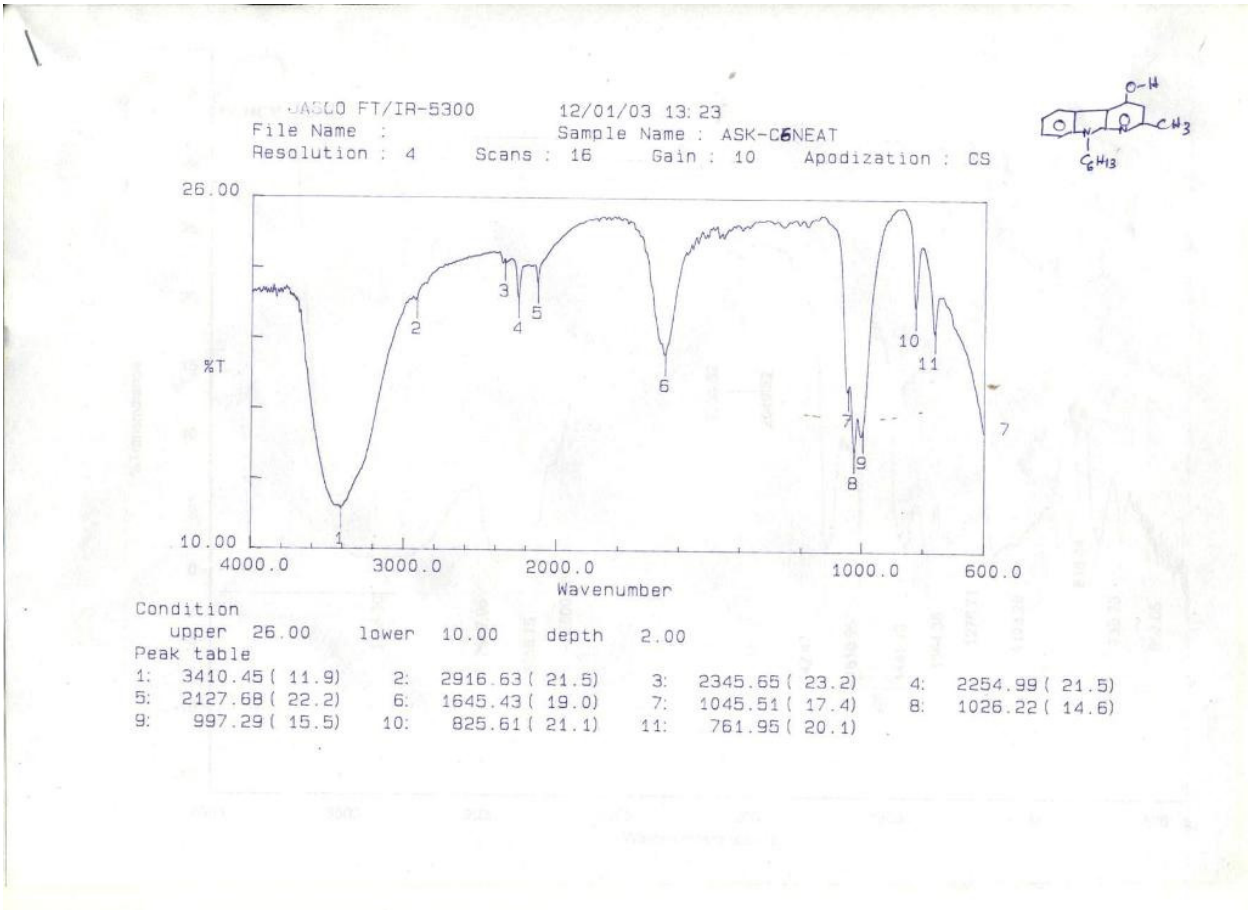


Figure S4. IR Spectrum of **2f**

Although tautomerism is a difficult subject to study in the gas phase, we recorded the GCMS of **2f** which shows a fragment by elimination of OH (M-17). This indicates the presence of a OH group in 2**f**.

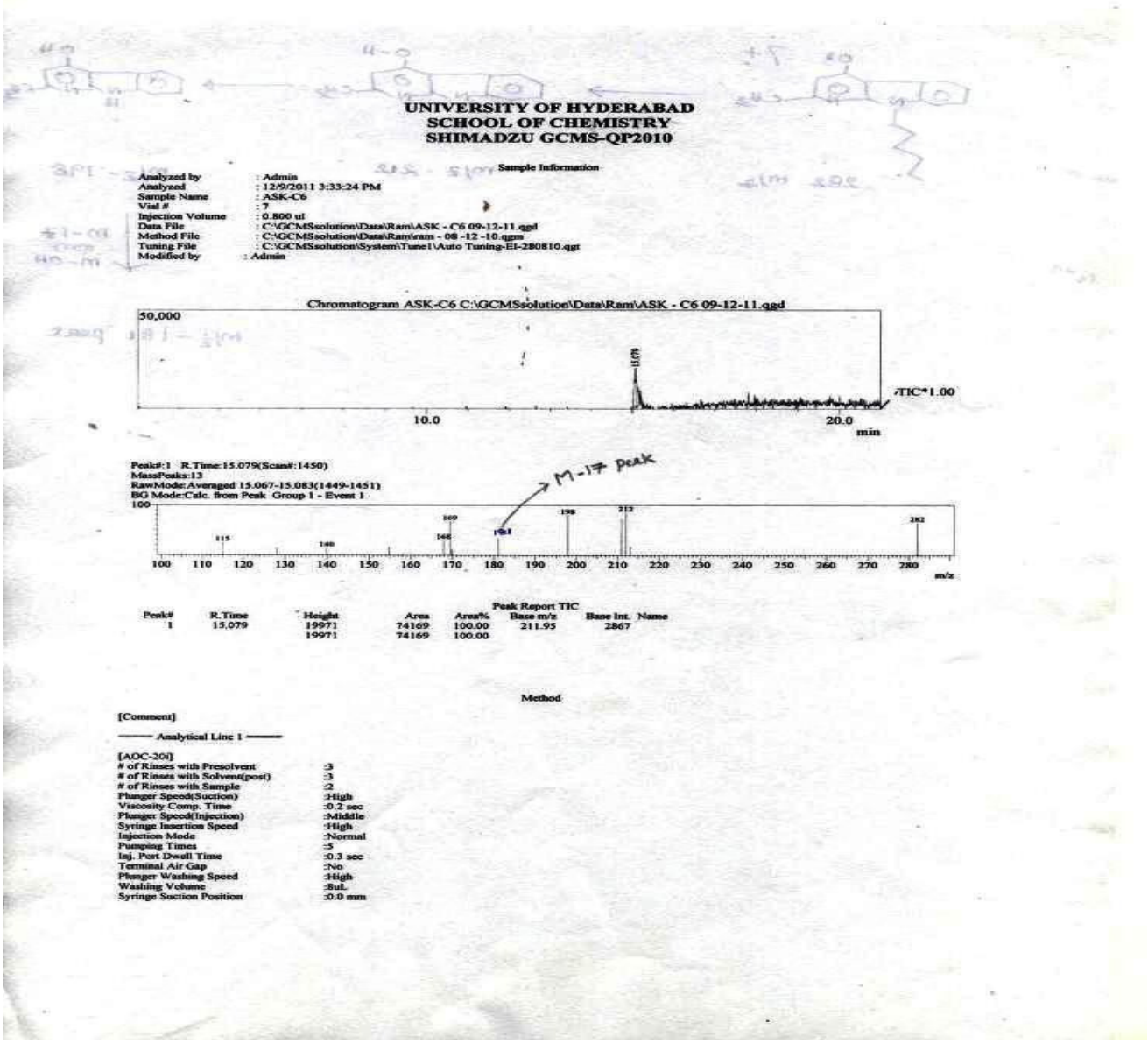
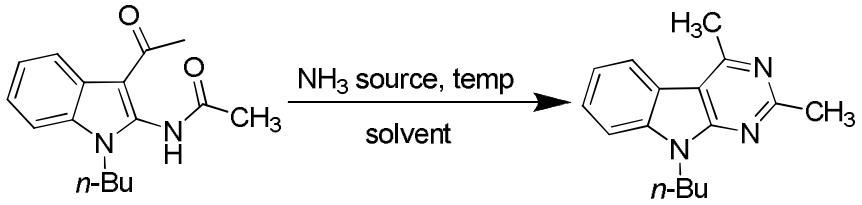


Figure S5. GC-MS of 2f

Table S3A Optimization for the synthesis of 9-butyl-2,4-dimethyl-9*H*-pyrimido[4,5-*b*]indole [a]



Entry	Ammonia Source	Solvent	Temp (°C)	Time (h)	Yield (%)[b]
1	NH_4OH	*t*-BuOH	110	24	45
2	NH_4OH	THF	110	24	-
3	NH_4OH	MeOH	110	24	10
4	NH_4Cl	*t*-BuOH	110	24	12
5	NH_4OAc	*t*-BuOH	110	24	40
6	NH_4OAc	MeOH	80	24	17
7	NH_4OAc	DMF	110	24	-
8	**$HCOONH_4$**	***t*-BuOH**	**110**	**5**	**91**
9	$HCOONH_4$	MeOH	80	24	43
10	$HCOONH_4$	MeCN	110	24	-
11	$HCOONH_4$	DMF	110	24	-
12	$HCOONH_4$	DMSO	110	24	-

[a]Conditions: *N*-(3-acetyl-1-ethyl-1*H*-indol-2-yl)acetamide (1.0 mmol) and NH_3 source (6.0 mmol), solvent (2.0 mL/mmol), 80-110 °C, 5-24 h. [b]Isolated yields.

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Quantum Yield Calculations:

Quantum yield calculations have been done following the equation

$$\frac{\Phi_{sample}}{\Phi_{std.}} = \frac{A_{sample}}{A_{std.}} \times \frac{OD_{std.}}{OD_{sample}} \times \frac{\eta^{2}_{sample}}{\eta^{2}_{std.}}$$

where std. abbreviates for the standard (here quinine sulfate), A is the integrated emission intensity, O.D. stands for the optical density at the excited wavelength and η is the refractive index (for dichloromethane η = 1.424 and for water solutions η = 1.333). The excitations have been performed at iso-O.D. where the absorption curve of the sample intersects the same of quinine sulfate.

References:

(1) (a) Lu, S. C.; Duan, X. Y.; Shi, Z. J.; Li, B.; Ren Y. W.; Zhang, W. *Org. Lett*. **2009**, *11*, 3902-3905. (b) Ketcha, D. M.; Lieurance, B.A.; Homan, D. F. J. *J. Org. Chem.* **1996,** *61,* 6523-6525.

(2) Majo, V. J.; Perumal, P. T. *J. Org. Chem.* **1996,** *61,* 6523-6525.

(3) A. S. Kumar, R. Nagarajan, *Org. Lett.* **2011**, *6*, 1398.

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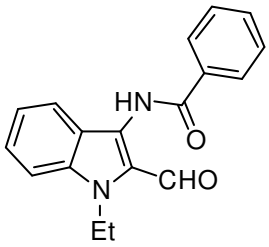
Analytical data for all new compounds

N-(1-Ethyl-2-formyl-1*H*-indol-3-yl)benzamide (1a):



Pale brown solid

mp. 113-115 °C

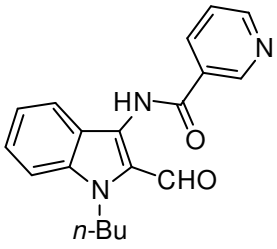
IR (KBr): 3352, 3217, 2920, 1789, 1653, 1519, 1454, 1390, 1222, 1210, 1109, 918, 727 cm^{-1}.

^1H NMR (400 MHz, TMS, $CDCl_3$) δ: 9.87 (s, 1H), 9.62 (s, 1H), 7.94 (d, 2H, J = 8.0 Hz), 7.74 (d, 1H, J = 8.0 Hz), 7.62 (d, 1H, J = 8.0 Hz), 7.47-7.43 (m, 1H), 7.29-7.20 (m, 4H), 3.92 (q, 2H, J = 8.0 Hz), 1.22 (t, 3H, J = 8.0 Hz).

^{13}C NMR (100 MHz, TMS, $CDCl_3$) δ: 189.2, 167.8, 136.2, 136.0, 134.8, 132.5, 132.4, 128.6, 127.8, 124.5, 123.0, 121.7, 121.6, 120.6, 110.4, 106.5, 38.2, 14.5.

LC-MS: *m/z* = 293 (M+H), positive mode; Anal. Calcd for molecular formula $C_{18}H_{16}N_2O_2$; C, 73.95; H, 5.52; N, 9.58 %; found: C, 73.85; H, 5.51; N, 9.45 %.

N-(1-Butyl-2-formyl-1*H*-indol-3-yl)nicotinamide (1b):



Yellow solid

mp. 196-198 °C

IR (KBr): 3329, 3222, 2917, 1792, 1662, 1522, 1460, 1329, 1262, 1209, 1111, 917, 725 cm^{-1}.

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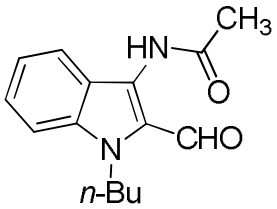
^{1}H NMR (400 MHz, TMS, $CDCl_3$) δ: 10.79 (s, 1H), 10.02 (s, 1H), 9.30 (s, 1H), 8.76 (s, 1H), 8.35 (d, 1H, J = 8.0 Hz), 7.99-7.97 (m, 1H), 7.39-7.28 (m, 4H), 4.11 (t, 2H, J = 8.0 Hz), 1.81-1.74 (m, 2H), 1.30-1.22 (m, 2H), 0.86 (t, 3H, J = 8.0 Hz).

^{13}C NMR (100 MHz, TMS, $CDCl_3$) δ: 184.8, 165.0, 153.0, 149.0, 135.9, 134.5, 132.3, 128.7, 126.9, 125.0, 123.8, 123.4, 123.1, 119.1, 110.8, 44.9, 30.9, 20.1, 13.6.

LC-MS: m/z = 322 (M+H), positive mode; Anal. Calcd for molecular formula $C_{19}H_{19}N_3O_2$; C, 71.01; H, 5.96; N, 13.08 %; found: C, 71.22; H, 5.89; N, 13.15 %.

N-(1-Butyl-2-formyl-1*H*-indol-3-yl)acetamide (1c):



Orange solid

mp. 131-133 °C

IR (KBr): 3317, 3222, 2950, 1795, 1655, 1562, 1498, 1329, 1225, 1209, 1127, 919, 725 cm^{-1}.

^{1}H NMR (400 MHz, TMS, $CDCl_3$) δ: 9.99 (s, 1H), 9.61 (s, 1H), 8.02 (d, 1H, J = 8.0 Hz), 7.33-7.24 (m, 3H), 4.09 (t, 2H, J = 8.0 Hz), 2.28 (s, 3H), 1.79-1.71 (m, 2H), 1.33-1.25 (m, 2H), 0.91 (t, 3H, J = 8.0 Hz).

^{13}C NMR (100 MHz, TMS, $CDCl_3$) δ: 184.6, 170.5, 141.6, 134.3, 124.8, 123.8, 123.4, 123.0, 119.3, 110.7, 107.2, 44.4, 30.9, 20.1, 13.6.

LC-MS: m/z = 259 (M+H), positive mode; Anal. Calcd for molecular formula $C_{15}H_{18}N_2O_2$; C, 69.74; H, 7.02; N, 10.84 %; found: C, 69.81; H, 7.12; N, 10.76 %.

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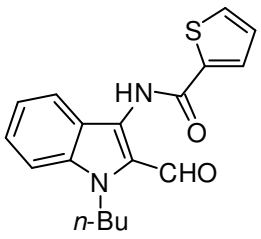
N-(1-Butyl-2-formyl-1*H*-indol-3-yl)thiophene-2-carboxamide (1d):



Pale brown solid

mp. 184-186 °C

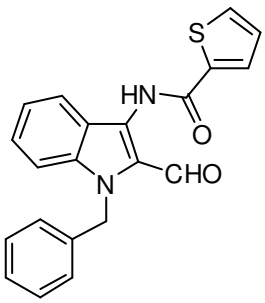
IR (KBr): 3325, 3201, 2927, 1759, 1652, 1572, 1485, 1332, 1262, 1208, 1129, 918, 732 cm^{-1}.

^1H NMR (400 MHz, TMS, $CDCl_3$) δ: 10.66 (s, 1H), 10.12 (s, 1H), 7.93-7.91 (m, 2H), 7.65-7.38 (m, 2H), 7.32-7.29 (m, 2H), 7.19-7.17 (m, 1H), 4.33 (t, 2H, J = 8.0 Hz), 1.85-1.78 (m, 2H), 1.28-1.26 (m, 2H), 0.90 (t, 3H, J = 8.0 Hz).

^{13}C NMR (100 MHz, TMS, $CDCl_3$) δ: 184.3, 160.6, 142.2, 137.5, 134.5, 132.7, 132.5, 130.8, 129.2, 128.3, 125.4, 123.0, 117.7, 111.0, 45.7, 30.8, 20.1, 13.6.

LC-MS: m/z = 327 (M+H), positive mode; Anal. Calcd for molecular formula $C_{18}H_{18}N_2O_2S$; C, 66.23; H, 5.56; N, 8.58 %; found: C, 66.38; H, 5.51; N, 8.49 %.

N-(1-Benzyl-2-formyl-1*H*-indol-3-yl)thiophene-2-carboxamide (1e):



Pale yellow solid

mp. 217-219 °C

IR (KBr): 3259, 3120, 2845, 1763, 1669, 1632, 1529, 1427, 1372, 1209, 1109, 1012, 915, 739 cm^{-1}.

^1H NMR (400 MHz, TMS, $CDCl_3$) δ: 10.62 (s, 1H), 10.22 (s, 1H), 7.95 (d, 1H, J = 8.0 Hz), 7.83-7.82 (m, 1H), 7.64-7.63 (m, 1H), 7.33-7.31 (m, 1H), 7.30-7.24 (m, 5H), 7.18-7.15 (m, 1H), 7.09-7.07 (m, 2H), 5.62 (s, 2H).

^{13}C NMR (100 MHz, TMS, $CDCl_3$) δ: 185.6, 160.7, 142.3, 137.7, 136.0, 134.9, 134.4, 133.7, 133.1, 132.1, 130.8, 128.9, 128.3, 127.7, 126.8, 125.5, 123.2, 117.8, 111.6, 105.2, 49.4.

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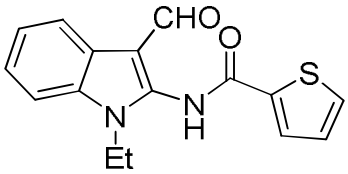
LC-MS: *m/z* = 361 (M+H), positive mode; Anal. Calcd for molecular formula $C_{21}H_{16}N_2O_2S$; C, 69.98; H, 4.47; N, 7.77 %; found: C, 69.71; H, 4.38; N, 7.67 %.

N-(1-Ethyl-3-formyl-1*H*-indol-2-yl)thiophene-2-carboxamide (1f):



Pale yellow solid

mp. 147-149 °C

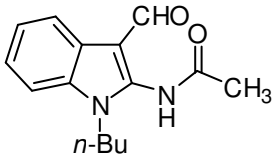
IR (KBr): 3252, 3159, 2876, 1778, 1669, 1612, 1466, 752, 717 cm^{-1}.

^1H NMR (400 MHz, TMS, $CDCl_3$) δ: 10.67 (s, 1H), 10.09 (s, 1H), 7.92-7.91 (m, 2H), 7.61 (d, 1H, *J* = 8.0 Hz), 7.39-7.37 (m, 1H), 7.31-7.13 (m, 3H), 4.30 (q, 2H, *J* = 8.0 Hz), 1.46 (t, 3H, *J* = 8.0 Hz).

^{13}C NMR (100 MHz, TMS, $CDCl_3$) δ: 184.8, 160.8, 141.9, 137.5, 134.4, 134.1, 132.7, 130.9, 128.9, 125.4, 123.1, 123.0, 118.0, 110.8, 40.8, 14.1.

LC-MS: *m/z* = 299 (M+H), positive mode; Anal. Calcd for molecular formula $C_{16}H_{14}N_2O_2S$; C, 64.41; H, 4.73; N, 9.39 %; found: C, 64.32; H, 4.79; N, 9.28 %.

N-(1-Butyl-3-formyl-1*H*-indol-2-yl)acetamide (1g):



Orange solid

mp. 176-178 °C

IR (KBr): 3259, 3160, 2852, 1776, 1670, 1635, 1532, 1479, 1452, 1382, 1210, 1120, 1020, 915, 720 cm^{-1}.

^1H NMR (400 MHz, TMS, $CDCl_3$) δ: 9.99 (s, 1H), 9.61 (s, 1H), 8.02 (d, 1H, *J* = 8.0 Hz), 7.33-7.24 (m, 3H), 4.09 (t, 2H, *J* = 8.0 Hz), 2.28 (s, 3H), 1.79-1.71 (m, 2H), 1.33-1.25 (m, 2H), 0.91 (t, 3H, *J* = 8.0 Hz)

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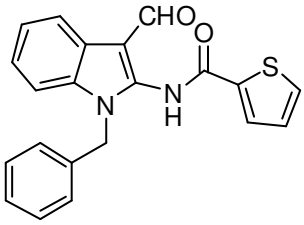
^{13}C NMR (100 MHz, TMS, $CDCl_3$) δ: 184.6, 170.5, 141.6, 134.3, 127.9, 124.8, 123.4, 123.0, 119.3, 110.7, 107.2, 44.4, 30.9, 20.1, 13.6.

LC-MS: *m/z* = 259 (M+H), positive mode; Anal. Calcd for molecular formula $C_{15}H_{18}N_2O_2$; C, 69.74; H, 7.02; N, 10.84 %; found: C, 69.85; H, 7.12; N, 10.76 %.

N-(1-Benzyl-3-formyl-1*H*-indol-2-yl)thiophene-2-carboxamide (1h) :



Yellow solid

mp. 229-231 °C

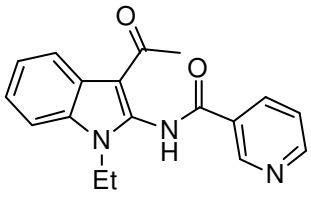
IR (KBr): 3262, 3129, 2852, 1769, 1662, 1635, 1520, 1414, 1375, 1202, 1102, 1010, 919, 735 cm^{-1}.

^{1}H NMR (400 MHz, TMS, $CDCl_3$) δ: 10.60 (s, 1H), 10.20 (s, 1H), 7.95 (d, 1H, *J* = 8.0 Hz), 7.83-7.82 (m, 1H), 7.65-7.63 (m, 1H), 7.31-7.24 (m, 6H), 7.18-7.15 (m, 1H), 7.09-7.07 (m, 2H), 5.62 (s, 2H).

^{13}C NMR (100 MHz, TMS, $CDCl_3$) δ: 185.0, 160.6, 142.4, 137.3, 135.8, 134.9, 132.8, 131.2, 130.9, 128.8, 128.2, 127.9, 127.5, 126.7, 125.4, 123.2, 123.1, 117.5, 111.4, 105.2, 49.5.

LC-MS: *m/z* = 361 (M+H), positive mode; Anal. Calcd for molecular formula $C_{21}H_{16}N_2O_2S$; C, 69.98; H, 4.47; N, 7.77 %; found: C, 69.91; H, 4.52; N, 7.86 %.

N-(3-Acetyl-1-ethyl-1*H*-indol-2-yl)nicotinamide (1i):



Dark brown solid

mp. 116-118 °C

IR (KBr): 3289, 3150, 2929, 2862, 1685, 1624, 1550, 1432, 1427, 1370, 1228, 1110, 1021, 915 cm^{-1}.

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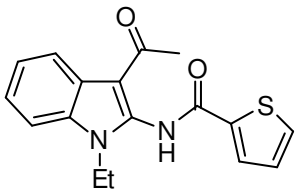
^{1}H NMR (400 MHz, TMS, $CDCl_3$) δ: 11.78 (s, 1H), 9.33 (s, 1H), 8.82 (s, 1H), 8.40-8.38 (m, 1H), 7.82-7.79 (m, 1H), 7.48-7.43 (m, 2H), 7.31-7.29 (m, 2H), 4.35 (q, 2H, J = 8.0 Hz), 2.67 (s, 3H), 1.48 (t, 3H, J = 8.0 Hz).

^{13}C NMR (100 MHz, TMS, $CDCl_3$) δ: 195.8, 164.6, 153.2, 149.4, 142.5, 135.5, 134.3, 129.0, 124.9, 123.6, 122.7, 122.6, 120.0, 110.9, 104.3, 41.0, 30.6, 14.1.

LC-MS: m/z = 306 (M+H), negative mode; Anal. Calcd for molecular formula $C_{18}H_{17}N_3O_2$; C, 70.34; H, 5.58; N, 13.67 %; found: C, 70.45; H, 5.51; N, 13.76 %.

N-(3-Acetyl-1-ethyl-1*H*-indol-2-yl)thiophene-2-carboxamide (1j):



Orange solid

mp. 132-134 °C

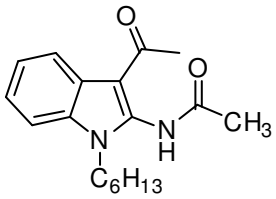
IR (KBr): 3250, 3127, 2919, 2822, 1672, 1632, 1532, 1411, 1389, 1202, 1126, 1029, 957 cm^{-1}.

^{1}H NMR (400 MHz, TMS, $CDCl_3$) δ: 11.78 (s, 1H), 7.94 (d, 1H, J = 8.0 Hz), 7.82-7.80 (m, 1H), 7.64 (d, 1H, J = 8.0 Hz), 7.46-7.44 (m, 1H), 7.32-7.30 (m, 2H), 7.21-7.18 (m, 1H), 4.40 (q, 2H, J = 8.0 Hz), 2.70 (s, 3H), 1.50 (t, 3H, J = 8.0 Hz).

^{13}C NMR (100 MHz, TMS, $CDCl_3$) δ: 195.7, 160.7, 143.1, 138.3, 134.4, 132.5, 130.7, 128.3, 125.0, 122.6, 122.4, 119.9, 110.9, 103.7, 41.2, 30.6, 14.1.

LC-MS: m/z = 313 (M+H), positive mode; Anal. Calcd for molecular formula $C_{17}H_{16}N_2O_2S$; C, 65.36; H, 5.16; N, 8.97 %; found: C, 65.26; H, 5.24; N, 8.91 %.

N-(3-Acetyl-1-hexyl-1*H*-indol-2-yl)acetamide (1k):



Pale yellow solid

mp. 131-133 °C

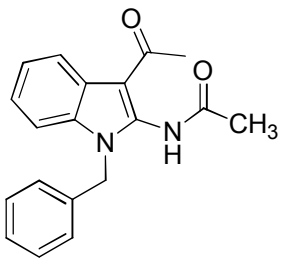
IR (KBr): 3259, 3210, 2912, 2852, 1666, 1614, 1565, 1412, 1327, 1222, 1122, 912, 907 cm^{-1}.

^1H NMR (400 MHz, TMS, $CDCl_3$) δ: 10.46 (s, 1H), 7.82-7.80 (m, 1H), 7.39-7.38 (m, 1H), 7.30-7.27 (m, 2H), 4.24 (t, 2H, J = 8.0 Hz), 2.69 (s, 3H), 2.33 (s, 3H), 1.81-1.78 (m, 2H), 1.33-1.23 (m, 6H), 0.89-0.86 (m, 3H).

^{13}C NMR (100 MHz, TMS, $CDCl_3$) δ: 195.4, 169.9, 142.3, 134.3, 124.9, 122.4, 122.2, 120.0, 110.9, 104.1, 45.6, 31.3, 30.7, 28.6, 26.5, 24.4, 22.5, 13.9.

LC-MS: m/z = 301 (M+H), positive mode; Anal. Calcd for molecular formula $C_{18}H_{24}N_2O_2$; C, 71.97; H, 8.05; N, 9.33 %; found: C, 71.85; H, 8.12; N, 9.45 %.

N-(3-Acetyl-1-benzyl-1*H*-indol-2-yl)acetamide (1l):



Pale purple solid

mp. 223-225 °C

IR (KBr): 3252, 3232, 2910, 2855, 1672, 1634, 1575, 1556, 1477, 1329, 1125, 960, 907 cm^{-1}.

^1H NMR (400 MHz, TMS, $CDCl_3$) δ: 10.45 (s, 1H), 7.85 (d, 1H, J = 8.0 Hz), 7.32-7.19 (m, 6H), 7.03-7.01 (m, 2H), 5.51 (s, 2H), 2.72 (s, 3H), 2.26 (s, 3H).

^{13}C NMR (100 MHz, TMS, $CDCl_3$) δ: 195.5, 170.1, 142.5, 136.1, 134.7, 128.7, 128.1, 127.6, 126.7, 126.5, 124.8, 122.7, 122.5, 120.0, 111.3, 104.5, 49.1, 30.8, 24.3.

LC-MS: m/z = 307 (M+H), positive mode; Anal. Calcd for molecular formula $C_{19}H_{18}N_2O_2$; C, 74.49; H, 5.92; N, 9.14 %; found: C, 74.63; H, 5.88; N, 9.07 %.

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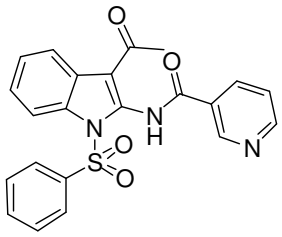
N-(3-acetyl-1-(phenylsulfonyl)-1*H*-indol-2-yl)nicotinamide (1m)(Known compound)



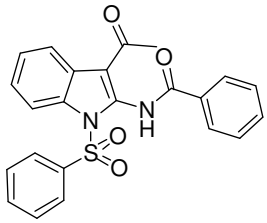
Solid, R_f (8 % EtOAc/hexane)

mp: 171-172 °C

IR (KBr): 3352, 2961, 2926, 2856, 1726, 1668, 1614, 1460, 1373, 1248, 1099, 1043, 800, 744, 696 cm^{-1}.

^1H NMR (400 MHz, TMS, $CDCl_3$) δ: 12.89 (s, 1H), 9.33 (s, 1H,), 8.85 (d, 1H, J = 8.0 Hz), 7.77-7.45 (m, 1H), 7.46-7.43 (m, 2H), 7.41-7.20 (m, 2H), 7.18-7.15 (m, 3H), 7.14-7.05 (m, 1H); 6.86-6.79 (m, 2H), 2.72 (s, 3H). Spectral data matched reported for this compound.

N-(3-acetyl-1-(phenylsulfonyl)-1*H*-indol-2-yl)benzamide(1n): (Known compound)



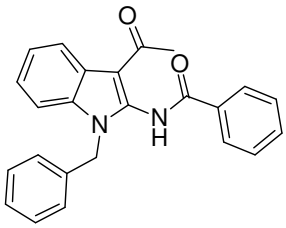
Solid, R_f (6 % EtOAc/hexane)

mp. 167-168 °C

IR (KBr): 3359, 2963, 2936, 2872, 1729, 1668, 1624, 1465, 1473, 1348, 1099, 1043, 810, 745, 699 cm^{-1}.

^1H NMR (400 MHz, TMS, $CDCl_3$) δ: 12.81 (s, 1H), 8.11-8.09 (m, 1H), 7.80-7.69 (m, 1H), 7.67-7.60 (m, 2H), 7.58-7.55 (m, 2H), 7.44-7.35 (m, 2H), 7.32-7.27 (m, 4H), 7.26-7.14 (m, 2H), 2.75 (s, 3H). Spectral data matched reported for this compound.

N-(3-acetyl-1-benzyl-1*H*-indol-2-yl)benzamide (1o): (Known compound)



Solid, R_f (5 % EtOAc/Hexane)
m.p. 217-218 °C

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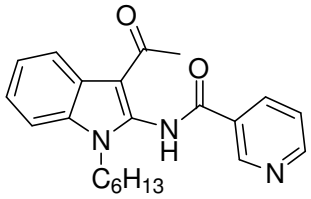
IR (KBr): 3295, 3157, 2991, 2962, 2872, 1689, 1614, 1545, 1462, 1417, 1373, 1271, 1228, 1155, 1107, 1022, 978, 914, 800, 746, 709 cm^{-1}.

^1H NMR (400 MHz, TMS, $CDCl_3$) δ: 11.64 (s, 1H), 8.07-8.05 (m, 2H), 7.89-7.82 (m, 2H), 7.61-7.54 (m, 1H), 7.52-7.40 (m, 2H), 7.36-7.34 (m, 1H), 7.26-7.24 (m, 4H), 7.09-7.07 (m, 2H), 5.66 (s, 2H), 2.75 (s, 3H). Spectral data matched reported for this compound

_N_-(3-acetyl-1-hexyl-1_H_-indol-2-yl)nicotinamide (1p):



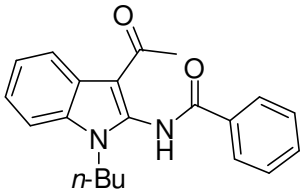
Solid, R_f (5 % EtOAc/hexane)

m.p. 271-272 °C

IR (KBr): 3057, 2959, 2928, 2866, 2247, 1689, 1614, 1554, 1419, 1271, 1228, 1155, 1107, 1022, 912, 748 cm^{-1}.

^1H NMR (400 MHz, TMS, $CDCl_3$) δ: 11.80 (s, 1H), 9.37 (s, 1H), 8.86 (s, 1H), 8.42 (d, 1H, J = 4.0 Hz), 7.84-7.83 (m, 1H), 7.47-7.45 (m, 2H), 7.35-7.31 (m, 1H), 7.29-7.14 (m, 1H), 4.43 (q, 2H, J = 14.2 Hz), 2.70 (s, 3H), 1.29-1.23 (m, 9H), 0.91-0.78 (m, 2H). Spectral data matched reported for this compound.

_N_-(3-acetyl-1-butyl-1_H_-indol-2-yl)benzamide(1q):



Solid, R_f (4 % EtOAc/hexane)

m.p. 212-213 °C

IR (KBr): 3271, 3057, 2961, 2930, 2866, 1685, 1614, 1548, 1462, 1417, 1373, 1271, 1228, 1155, 1107, 1022, 978, 914, 800, 746, 709 cm^{-1}.

^1H NMR (400 MHz, TMS, $CDCl_3$) δ: 11.70 (s, 1H), 8.16-8.14 (m, 2H), 7.85-7.83 (m, 1H), 7.64-7.62 (m, 1H), 7.59-7.55 (m, 1H), 7.47-7.45 (m, 2H), 7.34-7.31 (m, 2H), 4.43 (t, 2H, J =

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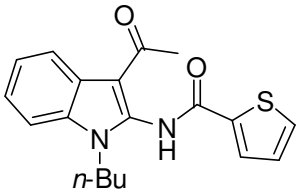
14.8 Hz), 2.75 (s, 3H), 1.85 (m, 2H), 1.33 (m, 2H), 0.93 (s, 3H). Spectral data matched reported[3] for this compound.

_N_-(3-acetyl-1-butyl-1_H_-indol-2-yl)thiophene-2-carboxamide (1r):



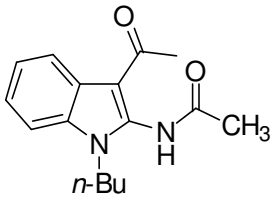
Solid, R_f (5 % EtOAc/hexane)

m.p. 201-202 °C

IR (KBr): 3282, 3059, 2981, 2940, 2866, 1685, 1624, 1558, 1462, 1427, 1373, 1271, 1228, 1165, 1117, 1022, 978, 914, 800, 746, 718 cm^{-1}.

^1H NMR (400 MHz, TMS, $CDCl_3$) δ: 11.83 (s, 1H), 7.96 (d, 1H, J = 4.0 Hz), 7.83-7.81 (m, 1H), 7.65-7.64 (m, 1H), 7.45-7.43 (m, 1H), 7.34-7.32 (m, 2H), 7.20-7.19 (m, 1H) 4.43-4.39 (m, 2H), 2.71 (s, 3H), 1.87-1.80 (m, 2H), 1.33-1.27 (m, 2H), 0.94 (t, 3H, J = 4.0 Hz). Spectral data matched reported[3] for this compound.

_N_-(3-acetyl-1-butyl-1_H_-indol-2-yl)acetamide (1s):



Solid, R_f (8 % EtOAc/hexane)

m.p. 102-103 °C

IR (KBr): 3059, 2959, 2930, 2866, 1689, 1616, 1554, 1462, 1419, 1269, 1155, 1107, 1020, 746 cm^{-1}.

^1H NMR (400 MHz, TMS, $CDCl_3$) δ: 10.48 (s, 1H), 7.82-7.80 (m, 1H), 7.42-7.39 (m, 1H), 7.32-7.28 (m, 2H), 4.26 (t, 2H, J = 14.8 Hz), 2.69 (s, 3H), 2.34 (s, 3H), 1.79 (q, 2H, J = 8.0 Hz), 1.28 (q, 2H, J = 8.0 Hz), 0.93 (s, 3H). Spectral data matched reported[3] for this compound.

_N_-(3-acetyl-1-ethyl-1_H_-indol-2-yl)benzamide (1t):

Solid, R_f (8 % EtOAc/hexane)

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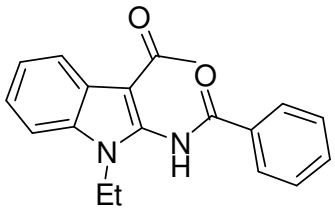
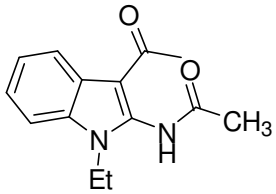


m.p. 97-98 °C

IR (KBr): 3291, 3150, 2960, 2942, 2869, 1689, 1624, 1558, 1473, 1437, 1383, 1291, 1238, 1155, 1127, 1022, 988, 914, 820, 756, 729 cm^{-1}.

^1H NMR (400 MHz, TMS, $CDCl_3$) δ: 11.75 (s, 1H), 8.17-8.15 (m, 2H), 7.85-7.83 (m, 1H), 7.63-7.61 (m, 1H), 7.58-7.54 (m, 2H), 7.49-7.46 (m, 1H), 7.34-7.32 (m, 2H), 4.42 (t, 2H, J = 14.8 Hz), 2.72 (s, 3H), 1.42 (t, 3H, J = 14.4 Hz). Spectral data matched reported[3] for this compound.

_N_-(3-acetyl-1-ethyl-1_H_-indol-2-yl)acetamide (1u): (Known compound)



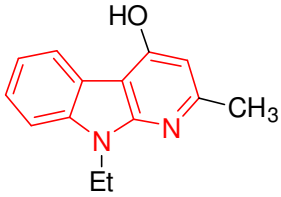
Solid, R_f (5 % EtOAc/hexane)

M.P. 97-98 °C

IR (KBr): 3296, 3057, 2976, 2920, 2862, 1689, 1614, 1545, 1462, 1417, 1267, 1159, 1105, 1022, 800, 748, 702 cm^{-1}.

^1H NMR (400 MHz, TMS, $CDCl_3$) δ: 10.50 (s, 1H), 7.82-7.81 (m, 1H), 7.43-7.42 (m, 1H), 7.32-7.30 (m, 2H), 4.28 (q, 2H, J = 17.2 Hz), 2.69 (s, 3H), 2.34 (s, 3H), 1.46 (t, 3H, J = 4.2 Hz). Spectral data matched reported[3] for this compound.

9-Ethyl-2-methyl-9_H_-pyrido[2,3-_b_]indol-4-ol (2a)



Pale yellow solid

mp. 117-119 °C

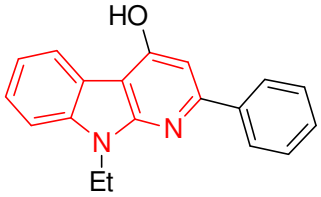
IR (KBr): 3429, 2927, 1658, 1026, 1003, 769, 725, 570, 528 cm^{-1}.

^1H NMR (400 MHz, TMS, DMSO-d_6) δ: 10.88 (s, br, 1H), 7.94 (1H, d, J = 6.16 Hz), 7.54 (d, 1H, J = 8.0 Hz), 7.36-7.33 (m, 1H), 7.20-7.17 (m, 1H), 6.29 (s, 1H), 4.40 (q, 2H, J = 5.68 Hz), 2.68 (s, 3H), 1.29 (t, 3H, J = 5.72 Hz).

^{13}C NMR (100 MHz, TMS, DMSO-d_6) δ: 162.9, 149.5, 145.7, 137.6, 124.0, 121.7, 121.2, 120.0, 109.5,106.8, 103.3, 36.0, 26.0, 14.3.

LC-MS: m/z = 225 (M+H), negative mode; Anal. Calcd for molecular formula $C_{14}H_{14}N_2O$; C, 74.31; H, 6.24; N, 12.38 %; found: C,74.42; H, 6.21; N, 12.28 %.

9-Ethyl-2-phenyl-9*H*-pyrido[2,3-*b*]indol-4-ol (2b)



Yellowish orange solid

mp. 152-154 °C

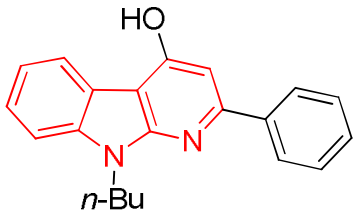
IR (KBr): 3425, 2924, 1635, 1572, 1456, 1379, 1338, 1178, 1109, 1022, 750 cm^{-1}.

^{1}H NMR (400 MHz, TMS, $CDCl_3$) δ: 11.73 (s, br, 1H), 8.24 (d, 1H, J = 6.0 Hz), 8.11 (d, 2H, J = 6.0 Hz), 7.50-7.40 (m, 4H), 7.39-7.32 (m, 1H), 7.31-7.29 (m, 1H), 7.06 (s, 1H), 4.61 (q, 2H, J = 6.0 Hz); 1.52 (t, 3H, J = 5.6 Hz).

^{13}C NMR (100 MHz, TMS, $CDCl_3$) δ: 159.3, 155.3, 153.5, 140.0, 138.6, 128.9, 128.6, 128.5, 128.0, 127.1, 125.2, 122.9, 120.0, 119.7, 108.6, 102.8, 100.3, 36.3, 14.1.

LC-MS: m/z = 289 (M+H), positive mode ; Anal. Calcd for molecular formula $C_{19}H_{16}N_2O$; C, 79.14 ; H, 5.59; N, 9.72 %; found: C,79.24; H, 5.51; N, 9.65 %.

9-Butyl-2-phenyl-9*H*-pyrido[2,3-*b*]indol-4-ol (2c)



Brown solid

mp. 132-134 °C

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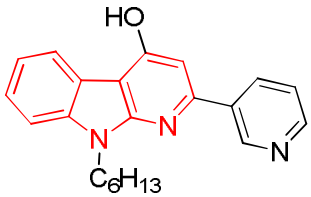
IR (KBr): 3431, 2920, 1635, 1568, 1452, 1362, 1330, 1176, 1100, 1025, 756 cm^{-1}.

^1H NMR (400 MHz, TMS, DMSO-d_6) δ: 11.63 (s, br, 1H), 8.27 (d, 1H, J = 6.16 Hz), 8.00 (d, 2H, J = 4.0 Hz), 7.47-7.40 (m, 4H), 7.36 (d, 1H, J = 5.6 Hz), 7.31-7.28 (m, 1H), 6.98 (s, 1H), 4.53 (t, 2H, J = 8.0 Hz), 1.91-1.96 (m, 2H), 1.47-1.39 (m, 2H), 0.97 (t, 3H, J = 8.0 Hz).

^{13}C NMR (100 MHz, TMS, DMSO-d_6) δ: 160.2, 154.7, 153.4, 139.6, 138.8, 128.9, 128.6, 128.5, 128.0, 127.1, 125.1, 122.8, 120.0, 119.9, 108.9, 103.0, 100.8, 41.4, 31.1, 20.3, 13.8.

LC-MS: m/z = 317 (M+H), positive mode; Anal. Calcd for molecular formula $C_{21}H_{20}N_2O$; C, 79.72; H, 6.37; N, 8.85 %; found: C,79.61; H, 6.31 ; N, 8.79 %.

9-Hexyl-2-(pyridin-3-yl)-9*H*-pyrido[2,3-*b*]indol-4-ol (2d)



Pale yellow solid,

mp. 112-113 °C

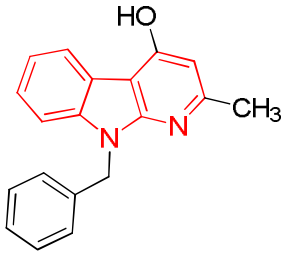
IR (KBr): 3412, 2918, 1572, 1458, 1375, 1340, 1170, 1109, 1024, 771, 750, 694 cm^{-1}.

^1H NMR (400 MHz, TMS, $CDCl_3$) δ: 10.59 (s, br, 1H), 8.45-8.10 (m, 2H), 7.59-7.13 (m, 7H), 4.48 (t, 2H, J = 4.0 Hz), 1.82 (t, 2H, J = 4.0 Hz), 1.32-1.12 (m, 6H), 0.75 (t, 3H, J = 8.0 Hz).

^{13}C NMR (100 MHz, TMS, $CDCl_3$) δ: 161.0, 153.9, 138.8, 134.3, 133.0, 126.8, 125.8, 122.8, 121.4, 120.4, 120.2, 119.8, 119.7, 109.7, 103.0, 100.2, 41.1, 31.1, 28.6, 26.3, 22.4, 14.2.

LC-MS: m/z = 346 (M+H), positive mode; Anal. Calcd for molecular formula $C_{22}H_{23}N_3O$; C, 76.49; H, 6.71; N, 12.16 %; found: C, 76.38; H, 6.63; N, 12.23 %.

9-Benzyl-2-methyl-9*H*-pyrido[2,3-*b*]indol-4-ol (2e)



Yellow color solid

mp. 125-127 °C

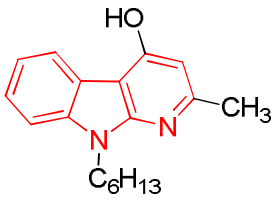
IR (KBr): 3406, 2912, 1560, 1452, 1379, 1336, 1172, 1122, 1022, 770, 732, 654 cm^{-1}.

^1H NMR (400 MHz, TMS, $CDCl_3$) δ: 10.88 (s, br, 1H), 7.95 (d, 1H, J = 7.68 Hz), 7.42 (d, 1H , J = 7.68 Hz), 7.29-7.17 (m, 7H), 6.34 (s, 1H), 5.59 (s, 2H), 2.71 (s, 3H).

^{13}C NMR (100 MHz, TMS, $CDCl_3$) δ: 163.0, 160.4, 150.2, 145.7, 138.1, 138.0, 128.8, 128.6, 127.5, 127.2, 124.1, 121.8, 121.2, 120.3, 110.0, 107.0, 103.5, 44.4, 20.1.

LC-MS: m/z = 289 (M+H), positive mode ; Anal. Calcd for molecular formula $C_{19}H_{16}N_2O$; C, 79.14; H, 5.59; N, 9.72 %; found: C, 79.03; H, 5.51; N, 9.65 %.

9-Hexyl-2-methyl-9*H*-pyrido[2,3-*b*]indol-4-ol (2f)



Pale yellow solid

mp. 122-124 °C

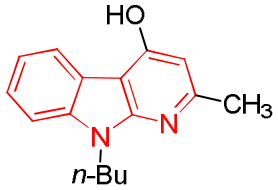
IR (KBr): 3410, 2916, 2345, 2254, 2127, 1645, 1045, 1026, 997, 825, 761 cm^{-1}.

^1H NMR (400 MHz, TMS, DMSO-d_6) δ: 10.88 (s, br, 1H), 7.94 (d, 1H, J = 7.72 Hz), 7.53 (d, 1H, J = 7.72 Hz), 7.34 (t, 1H, J = 8.0 Hz), 7.18 (t, 1H, J = 8.0 Hz), 6.29 (s, 1H), 4.32 (t, 2H, J = 8.0 Hz), 2.68 (s, 3H), 1.75-1.72 (m, 2H), 1.24-1.20 (m, 6H), 0.80 (t, 3H, J = 8.0 Hz).

^{13}C NMR (100 MHz, TMS, DMSO-d_6) δ: 162.9, 149.9, 145.6, 138.0, 124.0, 121.6, 121.1, 120.0, 109.7, 106.6, 103.5, 41.2, 31.4, 28.8, 26.4, 22.4, 20.1, 14.3.

LC-MS: m/z = 283 (M+H), positive mode; Anal. Calcd for molecular formula $C_{18}H_{22}N_2O$; C, 76.56; H, 7.85; N, 9.92 %; found: C, 76.45; H, 7.81; N, 9.88 %.

9-Butyl-2-methyl-9*H*-pyrido[2,3-*b*]indol-4-ol (2g)



Off-white solid

mp. 127-129 °C

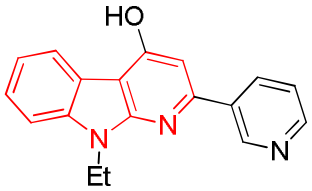
IR (KBr): 3450, 2932, 1552, 1460, 1372, 1372, 1317, 1195, 1120, 1019, 779, 715 cm^{-1}.

^1H NMR (400 MHz, TMS, DMSO-d_6) δ: 10.83 (s, br, 1H), 7.94 (d, 1H, J = 8.0 Hz), 7.55-7.52 (m, 1H), 7.35 (t, 1H, J = 8.0 Hz), 7.18 (t, 1H, J = 8.0 Hz), 6.28 (s, 1H), 4.32 (t, 2H, J = 8.0 Hz), 2.69 (s, 3H), 1.75-1.71 (m, 2H), 1.22-1.19 (m, 2H), 0.81 (t, 3H, J = 8.0 Hz).

^{13}C NMR (100 MHz, TMS, DMSO-d_6) δ: 163.2, 150.4, 146.1, 138.5, 124.2, 121.7, 121.0, 120.0, 110.0, 107.0, 103.3, 41.4, 31.5, 22.7, 20.3, 14.7.

LC-MS: m/z = 255 (M+H), positive mode; Anal. Calcd for molecular formula $C_{16}H_{18}N_2O$; C, 75.56; H, 7.13; N, 11.01 %; found: C, 75.46; H, 7.21; N, 11.12 %.

9-Ethyl-2-(pyridin-3-yl)-9*H*-pyrido[2,3-*b*]indol-4-ol (2h)



Pale yellow solid

mp. 122-124 °C

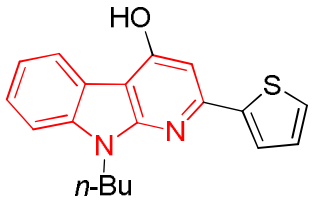
IR (KBr): 3459, 2969, 1585, 1492, 1375, 1332, 1197, 1102, 1095, 785, 715 cm^{-1}.

^1H NMR (400 MHz, TMS, $CDCl_3$) δ: 11.7 (s, br, 1H), 8.22 (d, 1H, J = 8.0 Hz), 8.13-8.08 (m, 2H), 7.47-7.43 (m, 3H), 7.39-7.37 (m, 1H), 7.28-7.26 (m, 1H), 7.04 (s, 1H), 4.59 (q, 2H, J = 8.0 Hz), 1.49 (t, 3H, J = 8.0 Hz).

^{13}C NMR (100 MHz, TMS, $CDCl_3$) δ: 160.2, 155.7, 153.9, 140.0, 138.6, 128.9, 128.6, 128.1, 127.2, 125.3, 122.9, 120.1, 119.8, 108.7, 102.9, 100.4, 36.6, 13.8.

LC-MS: m/z = 290 (M+H), positive mode; Anal. Calcd for molecular formula $C_{18}H_{15}N_3O$; C, 75.69; H, 6.03; N, 13.24 %; found: C, 74.65; H, 5.28; N, 14.43 %.

9-Butyl-2-(thiophen-2-yl)-9*H*-pyrido[2,3-*b*]indol-4-ol (2i)



Yellowish solid

mp. 127-129 °C

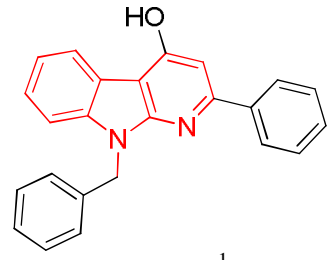
IR (KBr): 3460, 2932, 1565, 1459, 1377, 1362, 1192, 1127, 1090, 779, 717 cm^{-1}.

^1H NMR (400 MHz, TMS, $CDCl_3$) δ: 11.70 (s, br, 1H), 7.92-7.91 (m, 2H), 7.65-7.63 (m, 1H), 7.40-7.29 (m, 5H), 4.33 (t, 2H, J = 8.0 Hz), 1.85-1.78 (m, 2H), 1.28-1.26 (m, 2H), 0.90 (t, 3H, J = 8.0 Hz).

^{13}C NMR (100 MHz, TMS, $CDCl_3$) δ: 160.5, 156.6, 149.5, 142.2, 137.5, 134.5, 132.7, 130.8, 128.3, 125.4, 123.1, 123.0, 117.7, 111.0, 105.2, 45.7, 30.8, 20.1, 13.6.

LC-MS: m/z = 323 (M+H), positive mode; Anal. Calcd for molecular formula $C_{19}H_{18}N_2OS$; C, 70.78; H, 5.63; N, 8.69 %; found: C, 70.65; H, 5.58; N, 8.59 %.

9-Benzyl-2-phenyl-9*H*-pyrido[2,3-*b*]indol-4-ol (2j)



Brown solid

mp. 128-130 °C

IR (KBr): 3452, 2927, 1572, 1445, 1372, 1327, 1190, 1130, 1052, 752, 711 cm^{-1}.

^1H NMR (400 MHz, TMS, $CDCl_3$) δ: 11.15 (s, br, 1H), 8.12 (d, 2H, J = 8.0 Hz), 7.57 (d, 1H, J = 8.0 Hz), 7.46-7.43 (m, 2H), 7.36 (d, 1H, J = 8.0 Hz), 7.20-7.13 (m, 6H), 6.99 (s, 1H), 6.75-6.72 (m, 1H), 6.59 (d, 1H, J = 8.0 Hz), 4.86 (s, 2H).

^{13}C NMR (100 MHz, TMS, $CDCl_3$) δ: 163.2, 160.4, 140.8, 136.1, 134.3, 134.1, 130.5, 130.4, 129.7, 129.0, 128.8, 128.7, 128.6, 128.4, 127.5, 127.3, 127.1, 123.6, 122.3, 122.2, 121.1, 116.8, 108.7, 43.5.

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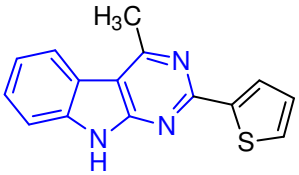
LC-MS: *m/z* = 351 (M+H), positive mode; Anal. Calcd for molecular formula $C_{24}H_{18}N_2O$; C, 82.26; H, 5.18; N, 7.99 %; found: C, 82.45; H, 5.23; N, 7.91 %.

4-Methyl-2-(thiophen-2-yl)-9*H*-pyrimido[4,5-*b*]indole (3a):



Yellow solid

mp: 127-129 °C

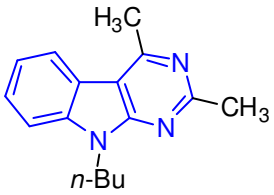
IR (KBr): 3050, 2856, 1919, 1852, 1680, 1570, 1462, 1252, 1170, 1100, 1028, 990, 752 cm^{-1}

^1H NMR (400 MHz, TMS, $CDCl_3$) δ: 11.0 (s, 1H,), 7.89-7.88 (m, 1H), 7.78-7.70 (m, 1H), 7.69-7.29 (m, 3H), 7.24-7.21 (m, 2H,), 2.73 (s, 3H).

^{13}C NMR (100 MHz, TMS, $CDCl_3$) δ: 161.8, 158.4, 144.4, 137.2, 133.0, 132.3, 130.7, 128.4, 123.7, 122.7, 122.3, 119.3, 111.8, 101.0, 29.9.

LC-MS: *m/z* = 265 (M+H), positive mode; Anal. Calcd for molecular formula $C_{15}H_{11}N_3S$; C, 67.90; H, 4.18; N, 15.84 %; found: C, 67.81; H, 4.23; N. 15.76 %.

9-Butyl-2,4-dimethyl-9*H*-pyrimido[4,5-*b*]indole(3b)



Pale purple solid or Pink

mp: 117-119 °C

IR (KBr): 3059, 2926, 2856, 1923, 1886, 1682, 1622, 1574, 1494, 1469, 1408, 1255, 1174, 1111, 1028, 999, 927, 794, 736 cm^{-1}.

^1H NMR (400 MHz, TMS, $CDCl_3$) δ: 8.07 (d, 1H, *J* = 8.0 Hz), 7.53-7.50 (m, 2H), 7.35-7.26 (m, 1H), 4.43 (t, 2H, *J* = 8.0 Hz), 2.95 (s, 3H), 2.82 (s, 3H), 1.89-1.85 (m, 2H), 1.41-1.35 (m, 2H), 0.96 (t, 3H, *J* = 8.0 Hz).

^{13}C NMR (100 MHz, TMS, $CDCl_3$) δ: 163.7, 159.2, 155.8, 139.0, 126.3, 122.5, 120.9, 120.2, 109.7, 109.5, 41.1, 30.8, 29.7, 26.4, 20.2, 13.7.

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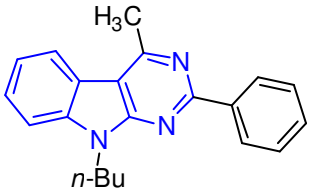
LC-MS: *m/z* = 254 (M+H), positive mode; Anal. Calcd for molecular formula $C_{16}H_{19}N_3$; C, 75.85; H, 7.56; N, 16.59 %; found: C, 75.96; H, 7.52; N, 16.51 %.

9-Butyl-4-methyl-2-phenyl-9*H*-pyrimido[4,5-*b*]indole (3c)



White dirty solid,

mp: 132-134 °C

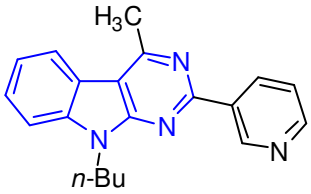
IR (KBr): 2924, 2858, 1672, 1622, 1514, 1346, 1251, 1093, 1022, 906 cm^{-1}

^1H NMR (400 MHz, TMS, $CDCl_3$) δ: 8.64 (d, 2H, *J* = 8.0 Hz), 8.11 (d, 1H, *J* = 8.0 Hz), 7.55-7.35 (m, 6H), 4.55 (t, 2H, *J* = 8.0 Hz), 3.08 (s, 3H,), 2.32-2.29 (m, 2H), 1.96-1.92 (m, 2H), 0.99 (t, 3H, *J* = 8.0 Hz).

^{13}C NMR (100 MHz, TMS, $CDCl_3$) δ: 160.3, 159.4, 145.7, 144.2, 139.7, 130.1, 128.4, 126.7, 122.7, 121.2, 120.2, 118.2, 114.8, 112.7, 110.4, 109.7, 41.2, 31.9, 29.7, 20.2, 14.1.

LC-MS: *m/z* = 316 (M+H), positive mode; Anal. Calcd for molecular formula $C_{21}H_{21}N_3$; C, 79.97; H, 6.71; N, 13.32 %; found: C, 79.85; H, 6.81; N, 13.45 %.

9-Butyl-4-methyl-2-(pyridin-3-yl)-9*H*-pyrimido[4,5-*b*]indole (3d):



White solid

mp: 187-189 °C

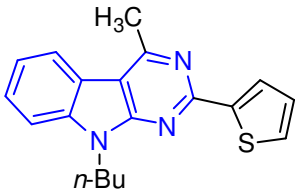
IR (KBr): 2950, 2820, 1635, 1580, 1522, 1490, 1452, 1263, 1091, 850, 750, 712 cm^{-1}

^1H NMR (400 MHz, TMS, $CDCl_3$) δ: 9.82 (s, 1H), 8.87-8.84 (m, 1H), 8.69 (s, 1H), 8.07 (d, 1H, *J* = 8.0 Hz), 7.57-7.49 (m, 2H), 7.44-7.35 (m, 2H), 4.49 (t, 2H, *J* = 4.0 Hz), 3.01 (s, 3H), 1.95-1.87 (m, 2H), 1.45-1.34 (m, 2H), 0.99 (t, 3H, *J* = 8.0 Hz).

^{13}C NMR (100 MHz, TMS, $CDCl_3$) δ: 159.7, 158.0, 155.6, 150.3, 149.9, 139.5, 135.5, 134.3, 126.8, 123.2, 122.7, 121.1, 120.0, 110.9, 109.7, 41.2, 30.8, 23.1, 20.2, 13.7.

LC-MS: *m/z* = 317 (M+H), positive mode; Anal. Calcd for molecular formula $C_{20}H_{20}N_4$; C, 75.92; H, 6.37; N, 17.71 %; found: C, 75.85; H, 6.31; N, 17.65 %.

9-Butyl-4-methyl-2-(thiophen-2-yl)-9H-pyrimido[4,5-b]indole (3e):



Brown colour solid

mp: 131-133 °C

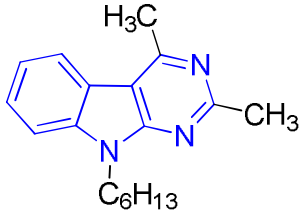
IR (KBr): 2926, 2858, 1620, 1572, 1531, 1494, 1469, 1400, 1263, 1097, 848, 798, 718 cm^{-1}.

^{1}H NMR (400 MHz, TMS, $CDCl_3$) δ: 8.13-8.08 (m, 2H), 7.57-7.46 (m, 3H), 7.39-3.28 (m, 1H), 7.19-7.17 (m, 1H), 4.51 (t, 2H, *J* = 8.0 Hz), 3.02 (s, 3H), 1.91-1.90 (m, 2H), 1.44-1.38 (m, 2H), 1.13 (t, 3H, *J* = 8.0 Hz).

^{13}C NMR (100 MHz, TMS, $CDCl_3$) δ: 159.7, 157.1, 155.6, 144.9, 139.4, 128.7, 128.1, 128.0, 126.4, 122.5, 121.0, 120.4, 110.1, 109.7, 41.0, 30.7, 23.0, 20.1, 13.7.

LC-MS: *m/z* = 322 (M+H), positive mode; Anal. Calcd for molecular formula $C_{19}H_{19}N_3S$; C, 70.99; H, 5.96; N, 13.07 %; found: C, 70.85; H, 5.91; N, 13.18 %.

9-Hexyl-2,4-dimethyl-9*H*-pyrimido[4,5-*b*]indole (3f)



Yellow solid

mp: 102-103 °C

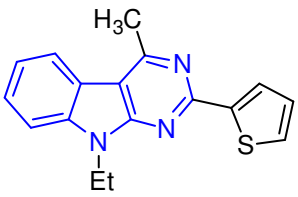
IR (KBr): 3049, 2930, 2866, 1930, 1682, 1622, 1574, 1496, 1469, 1402, 1205, 1018, 993, 823, 792, 742, 711, 435 cm^{-1}.

^{1}H NMR (400 MHz, TMS, $CDCl_3$) δ: 8.05 (d, 1H, J = 8.0 Hz), 7.53-7.47 (m, 2H), 7.37-7.33 (m, 1H), 4.41 (t, 2H, J = 4.0 Hz), 2.97 (s, 3H), 2.82 (s, 3H), 1.88-1.85 (m, 2H), 1.34-1.26 (m, 6H), 0.86 (t, 3H, J = 4.0 Hz).

^{13}C NMR (100 MHz, TMS, $CDCl_3$) δ: 163.5, 159.0, 155.6, 139.6, 126.4, 122.5, 121.0, 120.1, 109.7, 41.3, 31.4, 29.7, 28.6, 26.6, 26.1, 22.6, 22.5, 14.0.

LC-MS: m/z = 282 (M+H), positive mode; Anal. Calcd for molecular formula $C_{18}H_{23}N_3$; C, 76.83; H, 8.24; N, 14.93 %; found: C, 76.65; H, 8.19; N, 14.85 %.

9-Ethyl-4-methyl-2-(thiophen-2-yl)-9H-pyrimido[4,5-*b*]indole (3g):



Off-white solid

mp: 122-124 °C

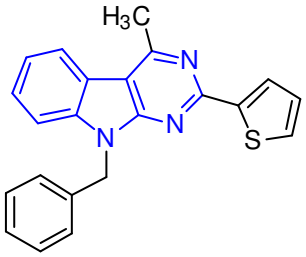
IR (KBr): 2950, 2892, 1526, 1452, 1398, 1256, 1166, 1112, 1035, 856, 795, 722 cm^{-1}.

^{1}H NMR (400 MHz, TMS, $CDCl_3$) δ: 8.07-8.05 (m, 2H), 7.55-7.50 (m, 2H), 7.48-7.14 (m, 3H), 4.51 (q, 2H, J = 8.0 Hz), 2.98 (s, 3H), 1.49 (t, 3H, J = 8.0 Hz).

^{13}C NMR (100 MHz, TMS, $CDCl_3$) δ: 159.8, 157.2, 155.2, 144.8, 139.1, 131.6, 128.7, 128.0, 126.4, 122.5, 121.1, 120.6, 110.4, 109.6, 36.3, 29.7, 14.0.

LC-MS: m/z = 294 (M+H), positive mode; Anal. Calcd for molecular formula $C_{17}H_{15}N_3S$; C, 69.59; H, 5.15; N, 14.32 %; found: C, 69.45; H, 5.21; N, 14.25 %.

9-Benzyl-4-methyl-2-(thiophen-2-yl)-9*H*-pyrimido[4,5-*b*]indole (3h)



Brown solid

mp: 115-117 °C

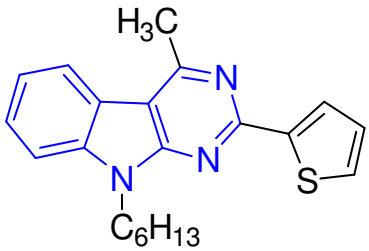
IR (KBr): 2924, 2858, 1570, 1494, 1427, 1398, 1249, 1163, 1151, 1101, 1093, 1020, 844, 796, 711 cm^{-1}.

^1H NMR (400 MHz, TMS, $CDCl_3$) δ: 8.15-8.07 (m, 2H), 7.47-7.17 (m, 10H), 5.70 (s, 2H), 3.04 (s, 3H).

^{13}C NMR (100 MHz, TMS, $CDCl_3$) δ: 160.0, 157.3, 155.7, 144.7, 139.3, 138.2, 136.7, 128.9, 128.7, 128.5, 128.2, 128.0, 127.9, 127.6, 126.5, 123.2, 122.4, 122.2, 121.4, 120.6, 110.2, 45.1.

LC-MS: *m/z* = 356 (M+H), positive mode; Anal. Calcd for molecular formula $C_{22}H_{17}N_3S$; C, 74.34; H, 4.82; N, 11.82 %; found: C, 74.19; H, 4.78; N, 11.75 %.

9-Hexyl-4-methyl-2-(thiophen-2-yl)-9*H*-pyrimido[4,5-*b*]indole (3i)



light yellow solid

mp: 129-131°C

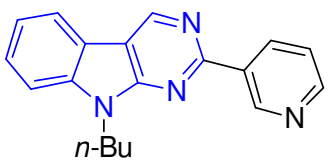
IR (KBr): 3020, 2950, 2828, 1522, 1454, 1422, 1362, 1207, 1160, 1109,1052, 1032, 850, 752 cm^{-1}.

^1H NMR (400 MHz, TMS, $CDCl_3$) δ: 8.89-8.71 (m, 1H), 8.14-8.13 (m, 1H), 7.61-7.28 (m, 5H), 4.63-4.40 (m, 2H), 2.87 (s, 3H), 1.55 -1.46 (m, 5H), 1.31-1.26 (m, 4H), 0.91-0.86 (m, 2H).

^{13}C NMR (100 MHz, TMS, $CDCl_3$) δ: 159.8, 158.2, 155.3, 153.3, 150.5, 150.1, 149.6, 139.2, 135.6, 126.8, 122.8, 121.2, 111.1, 109.6, 41.1, 36.3, 31.6, 30.5, 29.6, 23.1, 13.9.

LC-MS: *m/z* = 350 (M+H), positive mode; Anal. Calcd for molecular formula $C_{21}H_{23}N_3S$; C, 72.17; H, 6.63; N, 12.02 %; found: C, 72.08; H, 6.69; N,12.15 %.

9-Butyl-2-(pyridin-3-yl)-9H-pyrimido[4,5-*b*]indole (3j)



White dirty solid

mp: 74-76 °C

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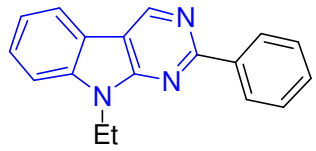
IR (KBr): 3059, 2922, 2852, 1612, 1573, 1459, 1400, 1252, 1160, 1121, 1020, 795, 738 cm^{-1}.

^1H NMR (400 MHz, TMS, $CDCl_3$) δ: 9.83 (s, 1H), 9.34 (s, 1H), 8.87-8.72 (m, 2H), 8.15 (d, 1H, J = 8.0 Hz), 7.60-7.58 (m, 1H), 7.55-7.53 (m, 1H), 7.47-7.44 (m, 1H), 7.41-7.37 (m, 1H), 4.55 (t, 2H, J = 8.0 Hz), 1.99-1.92 (m, 2H), 1.48-1.39 (m, 2H), 1.01 (t, 3H, J = 8.0 Hz).

^{13}C NMR (100 MHz, TMS, $CDCl_3$) δ: 158.7, 155.6, 150.6, 150.0, 148.0, 139.9, 135.5, 134.2, 127.6, 123.4, 121.5, 121.3, 119.2, 112.9, 110.0, 41.2, 30.8, 20.1, 13.7.

LC-MS: *m/z* = 303 (M+H), positive mode; Anal. Calcd for molecular formula $C_{19}H_{18}N_4$; C, 75.47; H, 6.00; N, 18.53 %; found: C,75.54; H, 6.08; N, 18.41 %.

9-Ethyl-2-phenyl-9*H*-pyrimido[4,5-*b*]indole (3k)



Greenish brown solid

mp. 114-116 °C

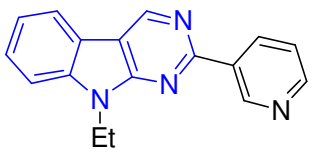
IR (KBr): 3061, 2978, 2930, 1668, 1622, 1581, 1556, 1493, 1467, 1433, 1400, 1359, 1224, 1140, 1091, 1066, 1024, 993, 923, 810, 769, 738, 698, 543, 449, 405 cm^{-1}.

^1H NMR (400 MHz, TMS, $CDCl_3$) δ: 9.33 (s, 1H), 8.65 (m, 2H), 8.13 (d, 1H, J = 7.8 Hz), 7.58-7.50 (m, 5H), 7.39-7.35 (m, 1H), 4.59 (q, 2H, J = 7.2 Hz), 1.54 (t, 3H, J = 7.24 Hz).

^{13}C NMR (100 MHz, TMS, $CDCl_3$) δ: 160.8, 155.4, 148.0, 139.5, 138.7, 132.0, 130.1, 129.2, 128.4, 128.3, 127.3, 121.4, 121.1, 119.5, 112.5, 109.6, 36.2, 13.9.

LC-MS: *m/z* = 275 (M+H), positive mode; Anal. Calcd for molecular formula $C_{18}H_{15}N_3$; C, 79.10; H, 5.53; N, 15.53 %; found: C, 79.21; H, 5.58; N, 15.58 %.

9-Ethyl-2-(pyridin-3-yl)-9*H*-pyrimido[4,5-*b*]indole (3l)



Yellowish orange solid

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mp. 92-94 °C

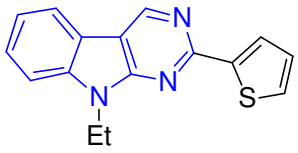
IR (KBr): 3042, 2935, 2920, 1610, 1562, 1552, 1530, 1485, 1475, 1390, 1200, 1132, 1085, 850, 795, 732, 709, 415 cm^{-1}.

^1H NMR (400 MHz, TMS, $CDCl_3$) δ: 9.79 (s, 1H), 9.27 (d, 1H, J = 2.0 Hz), 8.81 (d, 1H, J = 7.84 Hz), 8.68 (s, 1H), 8.08 (d, 1H, J = 7.64 Hz), 7.54 (d, 1H, J = 7.32 Hz), 7.49-7.47 (m, 1H), 7.42-7.39 (m, 1H), 7.36-7.32 (m, 1H), 4.53 (q, 2H, J = 4.0 Hz), 1.50 (t, 3H, J = 8.0 Hz).

^{13}C NMR (100 MHz, TMS, $CDCl_3$) δ: 158.6, 155.1, 149.9, 147.9, 139.5, 135.5, 134.1, 132.8, 123.3, 121.5, 121.3, 119.2, 113.0, 109.7, 108.2, 36.3, 13.9.

LC-MS: m/z = 275 (M+H), positive mode; Anal. Calcd for molecular formula $C_{17}H_{14}N_4$; C, 74.43; H, 5.14; N, 20.42 %; found: C, 74.32; H, 5.21; N, 20.36 %.

9-Ethyl-2-(thiophen-2-yl)-9*H*-pyrimido[4,5-*b*]indole (3m)



White solid

mp. 116-118 °C

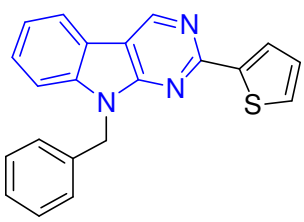
IR (KBr): 3053, 2974, 2928, 1622, 1585, 1554, 1531, 1493, 1469, 1429, 1396, 1221, 1138, 1084, 852, 796, 738, 709, 451, 405 cm^{-1}.

^1H NMR (400 MHz, TMS, $CDCl_3$) δ: 9.21 (s, 1H), 8.14-8.08 (m, 2H), 7.58-7.47 (m, 3H), 7.37-7.33 (m, 1H), 7.20-7.18 (m, 1H), 4.54 (q, 2H, J = 7.2 Hz), 1.52 (t, 3H, J = 7.24 Hz).

^{13}C NMR (100 MHz, TMS, $CDCl_3$) δ: 157.6, 155.1, 147.9, 144.7, 139.4, 128.9, 128.7, 128.2, 128.1, 127.2, 121.2, 119.6, 112.2, 109.6, 36.2, 13.9.

LC-MS: m/z = 280 (M+H),positive mode; Anal. Calcd for molecular formula $C_{16}H_{13}N_3S$; C, 68.79; H, 4.69; N, 15.04 %; found: C, 68.85; H, 4.61; N, 15.11 %.

9-Benzyl-2-(thiophen-2-yl)-9*H*-pyrimido[4,5-*b*]indole(3n)



Pale brown Solid

mp. 92-94 °C

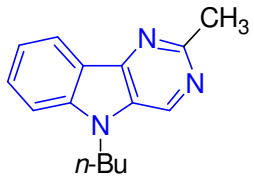
IR (KBr): 3120, 2960, 2952, 1610, 1530, 1512, 1490, 1452, 1350, 1220, 1150, 1082, 847, 752 cm^{-1}.

^1H NMR (400 MHz, TMS, $CDCl_3$) δ: 9.25 (s, 1H), 8.15-8.14 (m, 1H), 8.09 (d, 1H, J = 7.76 Hz); 7.50-7.46 (m, 2H); 7.42-7.36 (m, 3H), 7.34-7.32 (m, 2H), 7.30-7.25 (m, 2H), 7.20-7.18 (m, 1H), 5.69 (s, 2H).

^{13}C NMR (100 MHz, TMS, $CDCl_3$) δ: 157.8, 155.7, 148.1, 144.5, 139.6, 136.5, 131.7, 129.1, 128.8, 128.3, 128.2, 127.7, 127.5, 127.4, 126.4, 121.6, 121.2, 119.7, 112.2, 110.4, 45.1.

LC-MS: *m/z* = 342 (M+H), positive mode; Anal. Calcd for molecular formula $C_{21}H_{15}N_3S$; C, 73.87; H, 4.43; N, 12.31 %; found: C, 73.96; H, 4.38; N, 12.45 %.

5-Butyl-2-methyl-5*H*-pyrimido[5,4-*b*]indole (3o)



Yellow solid

mp. 121-123 °C

IR (KBr): 3050, 2922, 1612, 1525, 1517, 1460, 1412, 1360, 1222, 1166, 1022, 867, 717 cm^{-1}.

^1H NMR (400 MHz, TMS, $CDCl_3$) δ: 9.13 (s, 1H), 8.06 (d, 1H, J = 8.0 Hz); 7.53-7.51 (m, 1H), 7.47-7.45 (m, 1H), 7.34-7.32 (m, 1H), 4.41 (t, 2H, J = 8.0 Hz), 2.8 (s, 3H), 1.88-1.84 (m, 2H), 1.40-1.35 (m, 2H), 0.96 (t, 3H, J = 4.0 Hz).

^{13}C NMR (100 MHz, TMS, $CDCl_3$) δ: 164.2, 155.7, 147.6, 139.3, 127.1, 121.1, 121.0 , 119.3, 111.5, 109.8, 41.1, 30.8, 26.4, 20.2, 13.7.

LC-MS: *m/z* = 240 (M+H), positive mode; Anal. Calcd for molecular formula $C_{15}H_{17}N_3$; C,75.28 ; H, 7.16; N, 17.56 %; found: C, 75.36; H, 7.21; N, 17.45 %.

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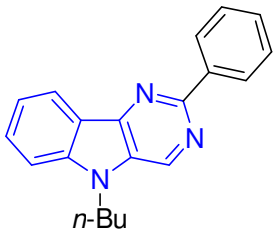
5-Butyl-2-phenyl-5*H*-pyrimido[5,4-*b*]indole (3p)



White solid

mp. 127-129 °C

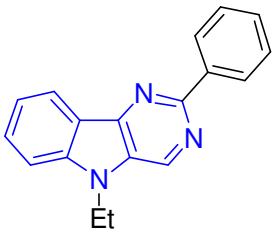
IR (KBr): 3050, 2972, 1602, 1525, 1506, 1422, 1407, 1272, 1238, 852, 817, 752 cm^{-1}.

^1H NMR (400 MHz, TMS, $CDCl_3$) δ: 9.33 (s, 1H), 8.62-8.60 (m, 2H), 8.11 (d, 1H, J = 7.72 Hz), 7.55-7.44 (m, 5H) 7.37-7.33 (m, 1H), 4.53 (t, 2H, J = 7.04 Hz), 1.97-1.89 (m, 2H), 1.45-1.35 (m, 2H), 0.98 (t, 3H, J = 7.32 Hz).

^{13}C NMR (100 MHz, TMS, $CDCl_3$) δ: 160.5, 155.9, 147.6, 139.9, 138.3, 137.3, 135.2, 130.2, 128.5, 128.3, 127.4, 121.4, 121.2, 119.4, 112.3, 109.9, 41.2, 30.8, 20.2, 13.7.

LC-MS: *m/z* = 302 (M+H), positive mode; Anal. Calcd for molecular formula $C_{20}H_{19}N_3$; C, 79.70; H, 6.35; N, 13.94 %; found: C, 79.86; H, 6.31; N, 13.81 %.

5-Ethyl-2-phenyl-5*H*-pyrimido[5,4-*b*]indole (3q)



Pale brown solid

mp. 133-135 °C

IR (KBr): 3046, 2992, 1655, 1585, 1575, 1462, 1417, 1379, 1235, 1216, 859, 792, 717 cm^{-1}.

^1H NMR (400 MHz, TMS, $CDCl_3$) δ: 9.36 (s, 1H), 8.61-8.58 (m, 2H), 8.14-8.12 (m, 1H), 7.61-7.50 (m, 5H), 7.40-7.36 (m, 1H), 4.60 (q, 2H, J = 8.0 Hz), 1.54 (t, 3H, J = 8.0 Hz).

^{13}C NMR (100 MHz, TMS, $CDCl_3$) δ: 160.3, 155.0, 147.1, 139.7, 137.8, 130.4, 128.6, 128.3, 127.7, 121.6, 121.5, 119.4, 116.9, 114.7, 112.5, 109.8, 36.4, 13.9.

LC-MS: *m/z* = 274 (M+H),positive mode; Anal. Calcd for molecular formula $C_{18}H_{15}N_3$; C, 79.10; H, 5.53; N, 15.37 %; found: C, 79.25; H, 5.61; N, 15.26 %.

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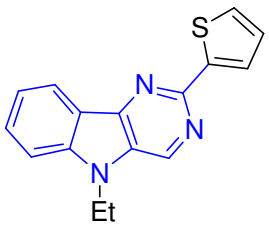
5-Ethyl-2-(thiophen-2-yl)-5*H*-pyrimido[5,4-*b*]indole (3r)



Yellow solid

mp. 129-131 °C

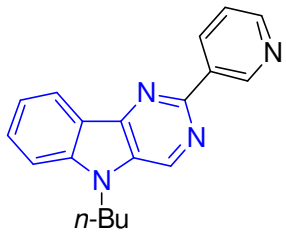
IR (KBr): 2950, 2822, 1652, 1621, 1582, 1557, 1492, 1443, 1372, 1236, 1217, 892, 819, 792, 712cm^{-1}.

^{1}H NMR (400 MHz, TMS, $CDCl_3$) δ: 9.23 (s, 1H), 8.14-8.13 (m, 1H), 8.09 (d, 1H, J = 8.0 Hz), 7.58-7.47 (m, 3H), 7.37-7.34 (m, 1H), 7.20-7.18 (m, 1H), 4.53 (q, 2H, J = 8.0 Hz), 1.52 (t, 3H, J = 8.0 Hz).

^{13}C NMR (100 MHz, TMS, $CDCl_3$) δ: 158.1, 155.5, 148.4, 145.1, 139.6, 139.3, 129.3, 129.1, 128.2, 127.3, 121.4, 119.7, 112.4, 109.8, 36.3, 14.1.

LC-MS: *m/z* = 280 (M+H), positive mode; Anal. Calcd for molecular formula $C_{16}H_{13}N_3S$; C, 68.79; H, 4.69; N, 15.04 %; found: C, 68.62; H, 4.61; N, 15.16 %.

5-Butyl-2-(pyridin-3-yl)-5*H*-pyrimido[5,4-*b*]indole (3s)



White dirty solid

mp. 132-134 °C

IR (KBr): 2962, 2810, 1662, 1600, 1572, 1523, 1485, 1432, 1379, 1252, 1227, 891, 822, 779, 720cm^{-1}.

^{1}H NMR (400 MHz, TMS, $CDCl_3$) δ: 9.31 (s, 1H), 8.85 (d, 2H, J = 8.0 Hz), 8.12 (d, 1H, J = 7.76 Hz), 7.60-7.50 (m, 2H), 7.45-7.34 (m, 3H), 4.52 (t, 2H, J = 7.04 Hz), 1.97-1.90 (m, 2H), 1.46-1.36 (m, 2H), 0.99 (t, 3H, J = 7.04 Hz).

^{13}C NMR (100 MHz, TMS, $CDCl_3$) δ: 164.0, 158.4, 155.6, 150.4, 149.8, 148.0, 139.9, 135.6, 132.6, 122.7, 121.5, 121.3, 119.2, 112.9, 110.0, 41.2, 30.8, 20.2, 13.7.

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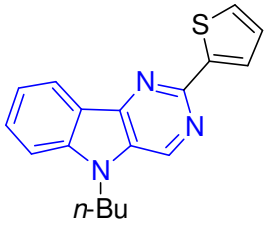
LC-MS: *m/z* = 303 (M+H), positive mode; Anal. Calcd for molecular formula $C_{19}H_{18}N_4$; C, 75.47; H, 6.00; N, 18.53 %; found: C, 75.38; H, 6.12; N, 18.45 %.

5-Butyl-2-(thiophen-2-yl)-5*H*-pyrimido[5,4-*b*]indole (3t)



Purple brown solid

mp. 119-121 °C

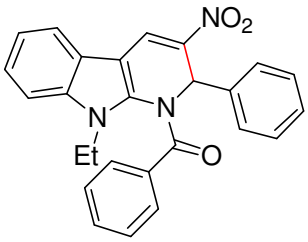
IR (KBr): 3012, 2912, 1672, 1612, 1880, 1532, 1480, 1462, 1395, 1386, 1332, 1289, 1252, 1212, 1192, 896, 852, 779, 715cm^{-1}.

^{1}H NMR (400 MHz, TMS, $CDCl_3$)δ: 9.23 (s, 1H), 8.13-8.09 (m, 2H), 7.58-7.47 (m, 3H), 7.38-7.34 (m, 1H), 7.28-7.18 (m, 1H), 4.51 (t, 2H, *J* = 4.0 Hz), 1.98-1.91 (m, 2H), 1.46-1.37 (m, 2H), 1.02 (t, 3H, *J* = 7.32 Hz).

^{13}C NMR (100 MHz, TMS, $CDCl_3$) δ: 157.5, 155.6, 147.9, 144.7, 139.8, 129.0, 128.1, 128.0, 127.2, 121.3, 121.2, 119.6, 112.0, 109.9, 41.1, 30.7, 20.1, 13.6.

LC-MS: *m/z* = 306 (M-H), negative mode; Anal. Calcd for molecular formula $C_{18}H_{17}N_3S$; C, 75.47; H, 5.57; N, 13.67 %; found: C, 70.25; H, 5.51; N, 13.76 %.

(9-ethyl-3-nitro-2-phenyl-2,9-dihydro-1H-pyrido[2,3-b]indol-1-yl)(phenyl)methanone (4a)



Pale yellow solid

mp. 232-234 °C

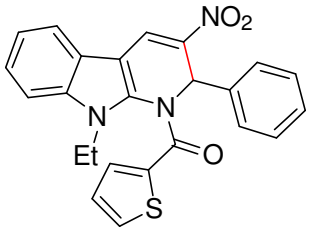
IR (KBr): 3063, 2926, 2854, 1670, 1612, 1508, 1464, 1352, 1269, 1095, 1018, 750, 690 cm^{-1}.

^{1}H NMR (400 MHz, TMS, $CDCl_3$) δ: 8.61 (s, 1H), 7.76-7.74 (m, 1H), 7.53-7.52 (m, 4H), 7.40-7.35 (m, 4H), 7.32-7.19 (m, 5H), 7.16 (s, 1H), 3.63 (q, 2H, *J* = 4.0 Hz), 0.91 (t, 3H, *J* = 7.32 Hz).

^{13}C NMR (100 MHz, TMS, $CDCl_3$) δ: 169.7, 149.3, 139.6, 137.1, 136.4, 135.8, 133.1, 132.1, 128.9, 128.8, 128.7, 128.5, 127.1, 126.9, 126.6, 124.0, 123.4, 122.7, 118.6, 117.2, 112.4, 111.0, 102.1, 58.3, 39.6, 13.6.

LC-MS: *m/z* = 424 (M+H), positive mode; Anal. Calcd for molecular formula $C_{26}H_{21}N_3O_3$; C, 73.74; H, 5.00; N, 9.92 %; found: C,73.65; H,5.00; N, 9.98 %.

(9-Ethyl-3-nitro-2-phenyl-2,9-dihydro-1*H*-pyrido[2,3-*b*]indol-1-yl)(thiophen-2-yl)methanone (4b)



Orange solid

mp. 217-219 °C

IR (KBr): 2916, 1651, 1612, 1527, 1509, 1432, 1272, 1105, 1018 cm^{-1}.

^{1}H NMR (400 MHz, TMS, $CDCl_3$) δ: 8.60 (s, 1H), 7.75-7.74 (m, 1H), 7.58-7.57 (m, 1H), 7.38-7.37 (m, 1H), 7.32-7.30 (m, 2H), 7.29-7.26 (m, 6H), 7.25-7.05 (m, 1H), 6.95-6.93 (m, 1H), 3.69 (q, 2H, *J* = 4.0 Hz), 0.87 (t, 3H, *J* = 4.0 Hz).

^{13}C NMR (100 MHz, TMS, $CDCl_3$) δ: 162.7, 138.6, 137.4, 136.2, 135.8, 135.7, 135.5, 133.4, 133.3, 132.6, 128.8, 127.8, 127.1, 126.7, 126.4, 123.9, 123.6, 122.7, 118.7, 111.0, 102.5, 58.3, 39.5, 13.6.

LC-MS: *m/z* = 428 (M-H), negitive mode; Anal. Calcd for molecular formula $C_{24}H_{19}N_3O_3S$; C, 67.12; H, 4.46; N, 9.78 %; found: C, 67.26; H, 4.51; N, 9.65 %.

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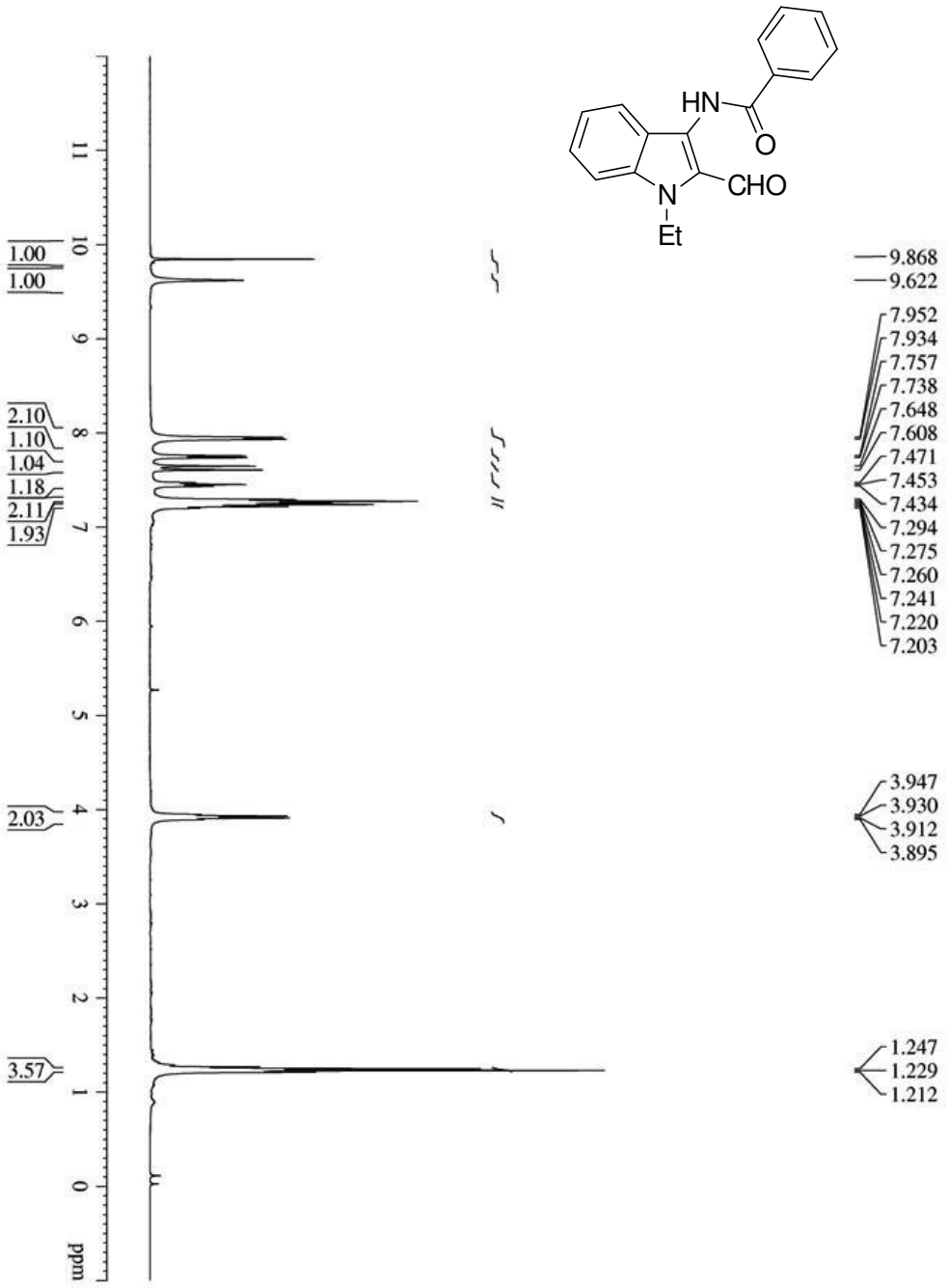
^{1}H NMR of *N*-(1-ethyl-2-formyl-1*H*-indol-3-yl)benzamide (1a):



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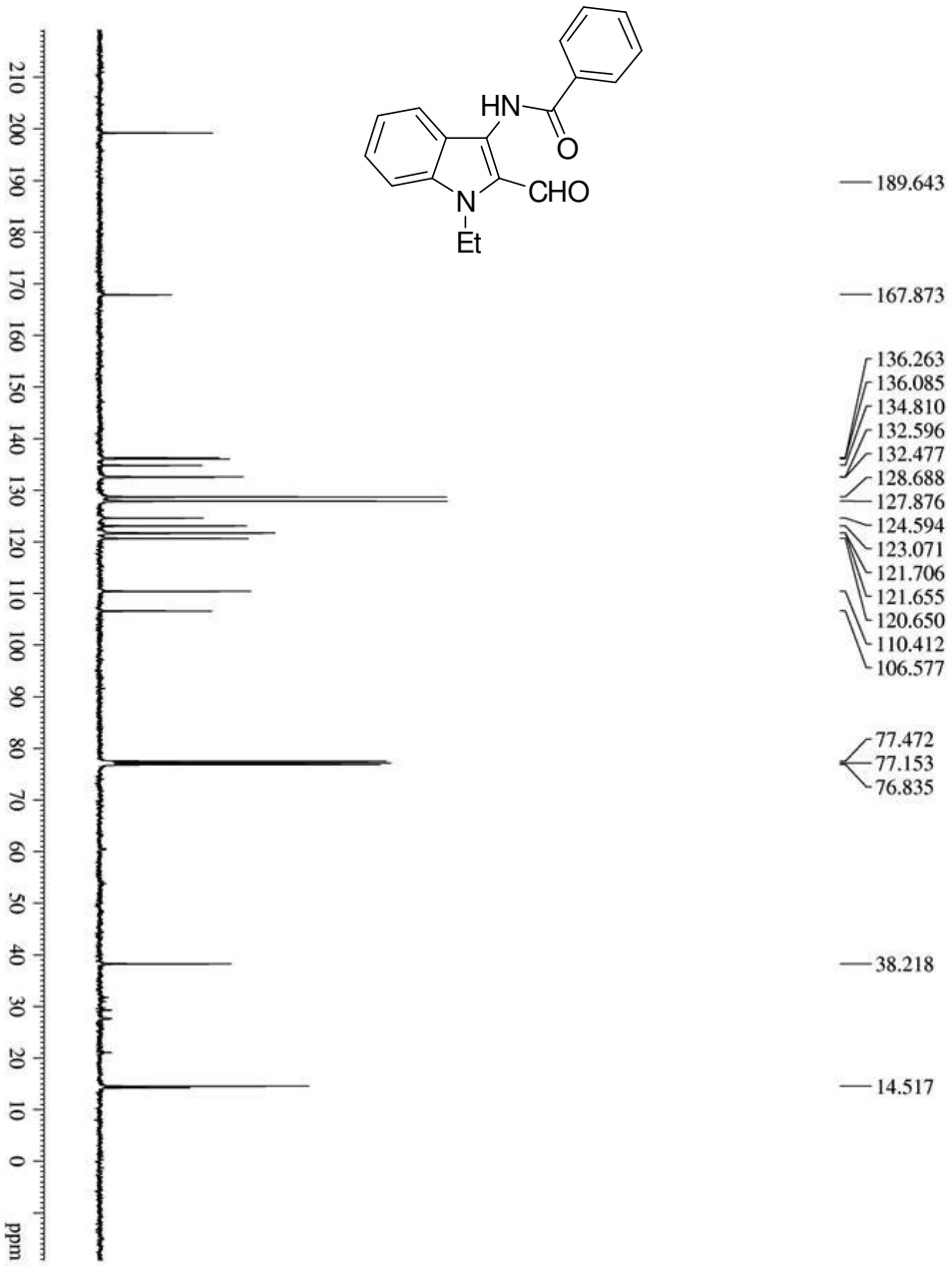
^{13}C NMR of *N*-(1-ethyl-2-formyl-1*H*-indol-3-yl)benzamide (1a):



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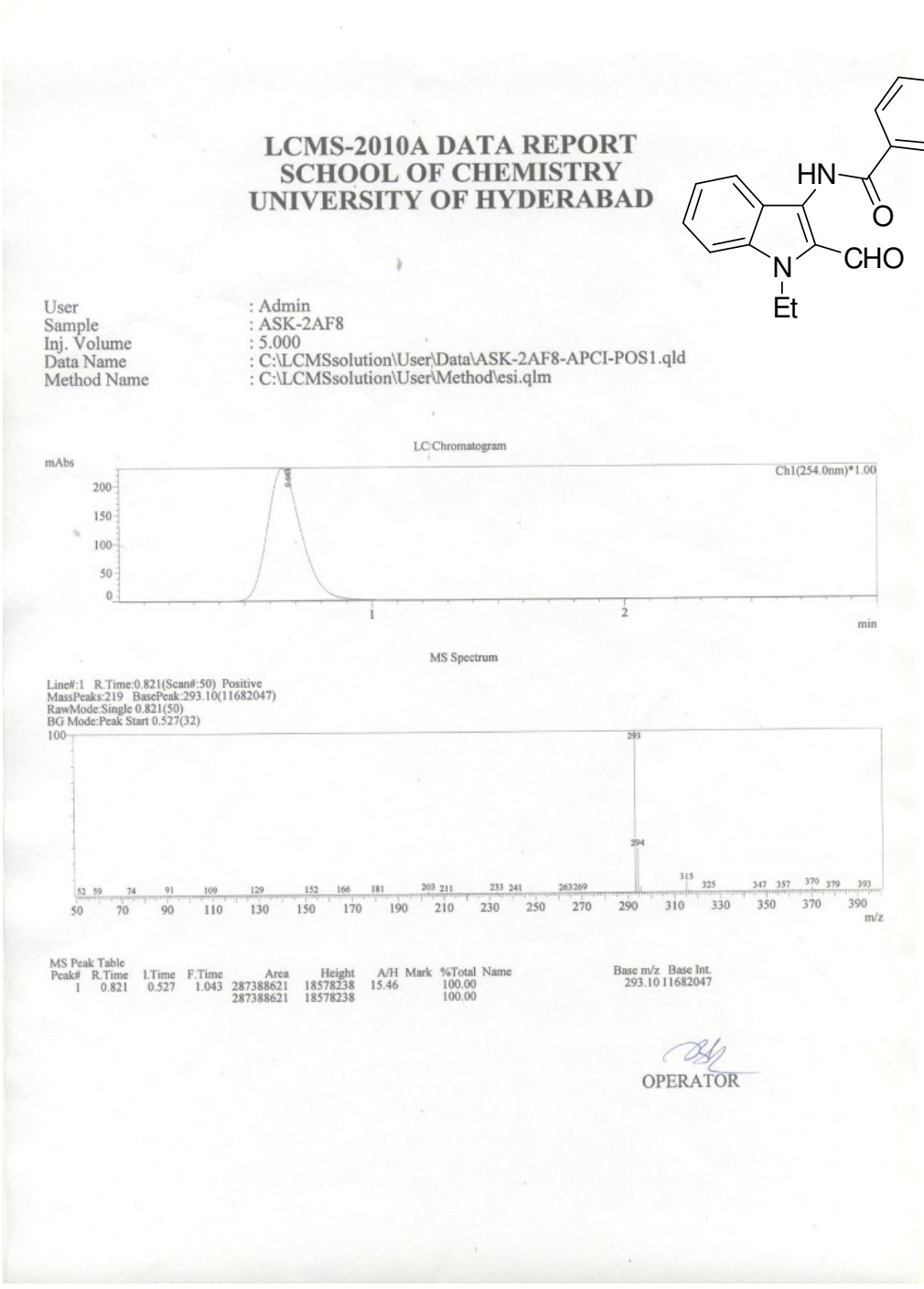
LCMS of *N*-(1-ethyl-2-formyl-1*H*-indol-3-yl)benzamide (1a):



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CHN Analysis of *N*-(1-ethyl-2-formyl-1*H*-indol-3-yl)benzamide (1a):

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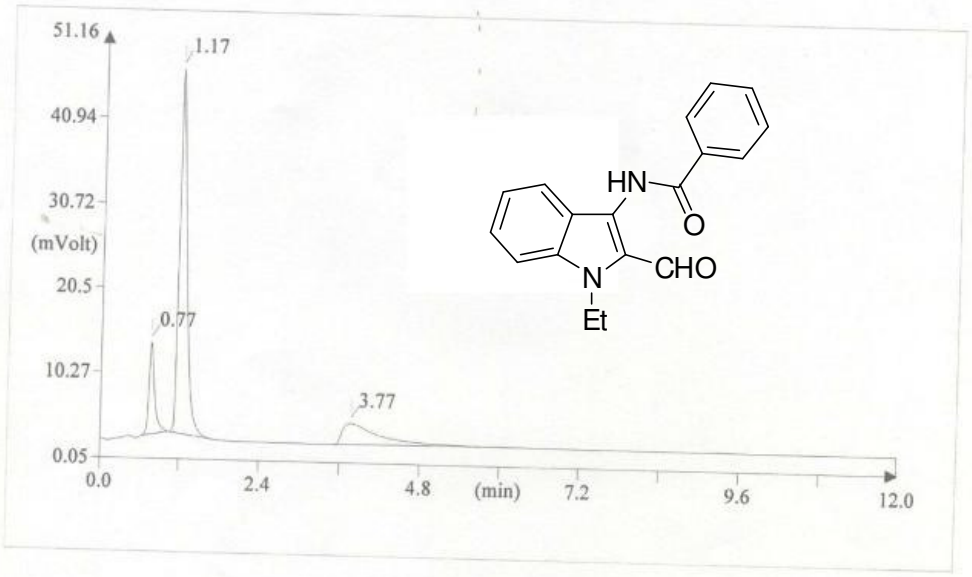
Method filename:	E:\Program Files\Thermo Finnigan\Eager 300 for EA1112\DATA\Sys_data_ex
Sample ID:	ASK-2AF8 (# 158)
Analysis type:	UnkNown
Chromatogram filename:	UNK-30012012-28.dat
Sample weight:	1.126

(1a)



Element Name	Element %	Ret. Time
Nitrogen	9. 45	0. 77
Carbon	73. 85	1. 17
Hydrogen	5. 51	3. 77

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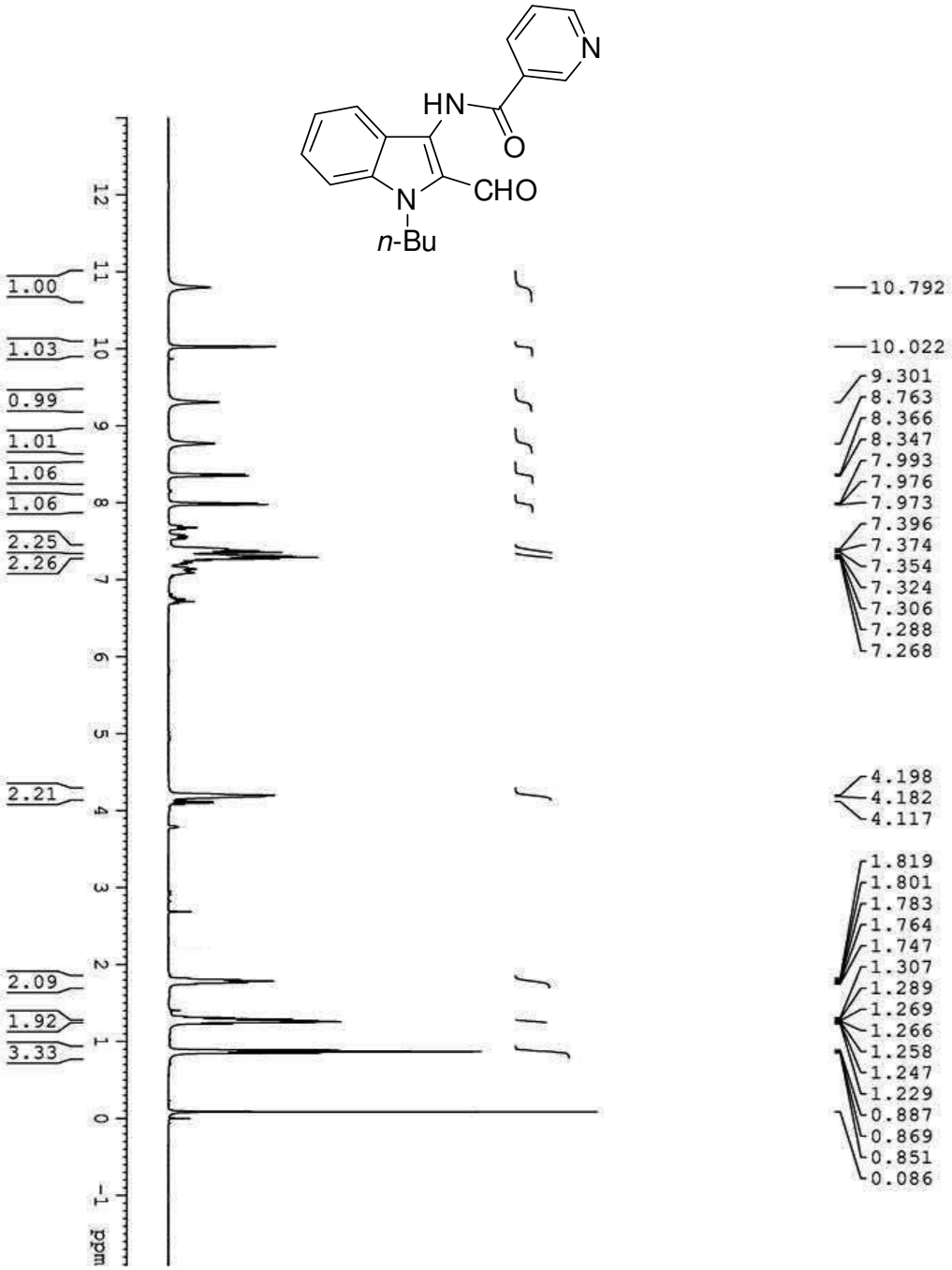
^{1}H NMR of *N*-(1-butyl-2-formyl-1*H*-indol-3-yl)nicotinamide (1b):



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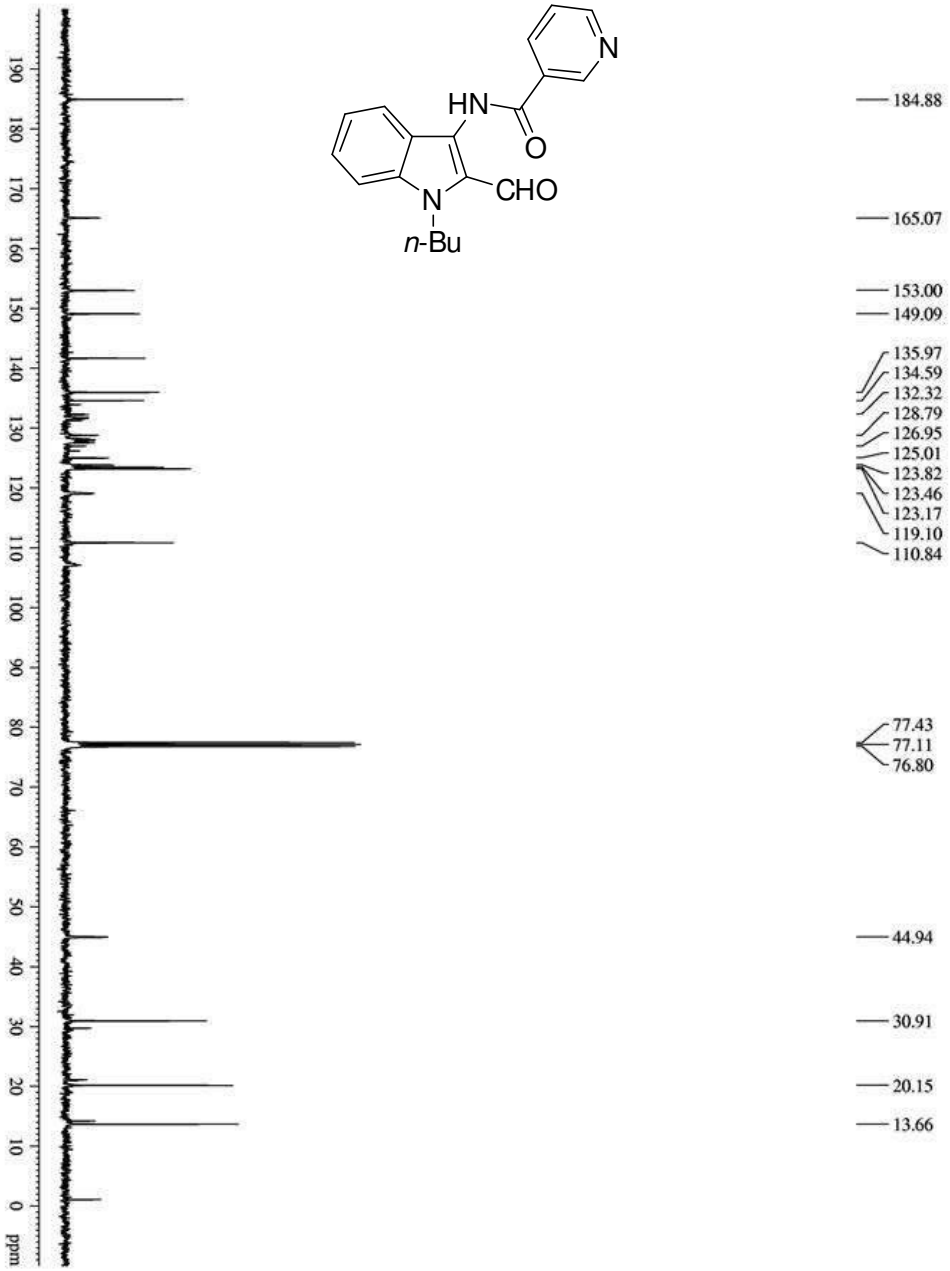
^{13}C NMR of *N*-(1-butyl-2-formyl-1*H*-indol-3-yl)nicotinamide (1b):



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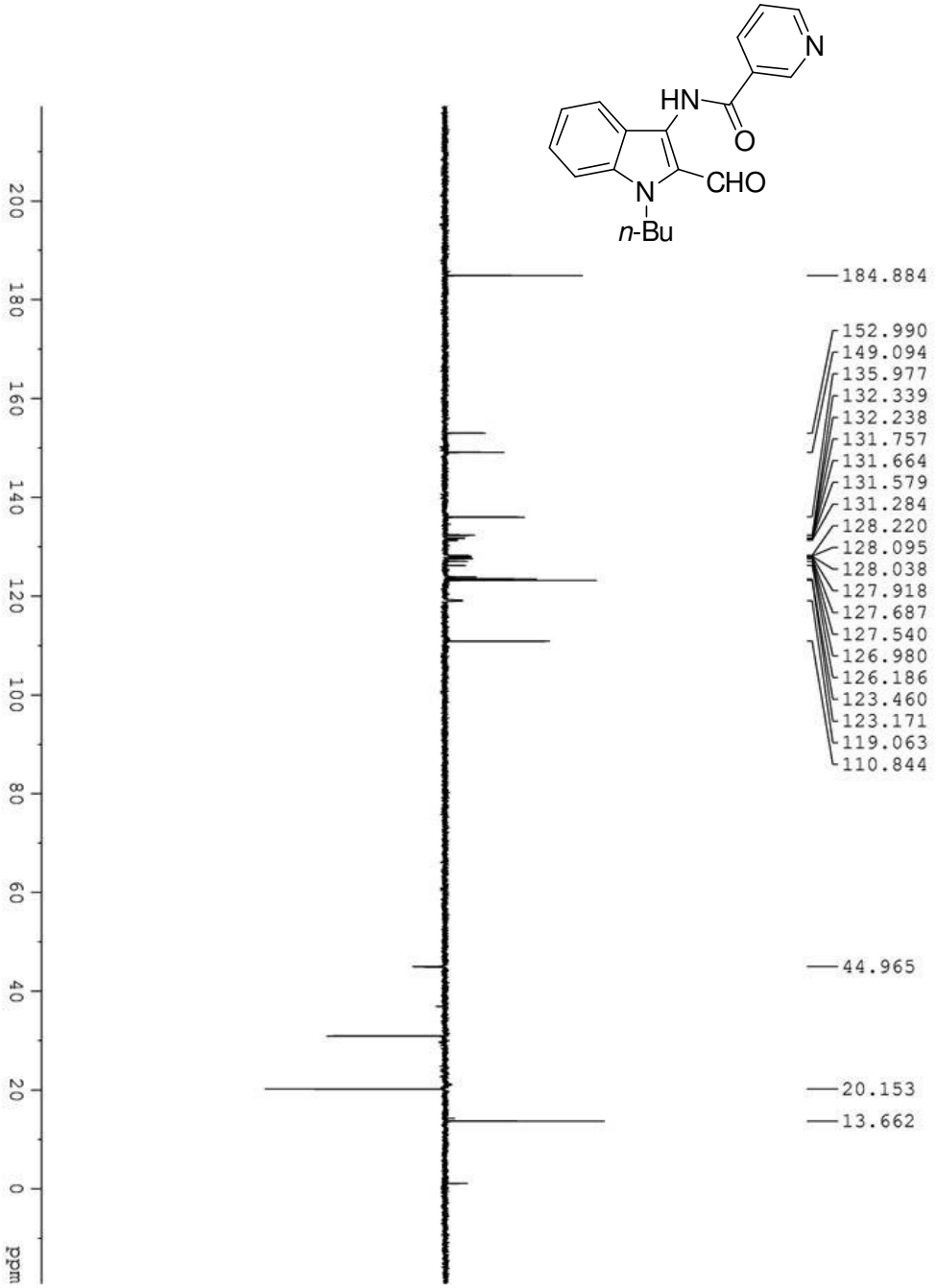
DEPT of *N*-(1-butyl-2-formyl-1*H*-indol-3-yl)nicotinamide (1b):



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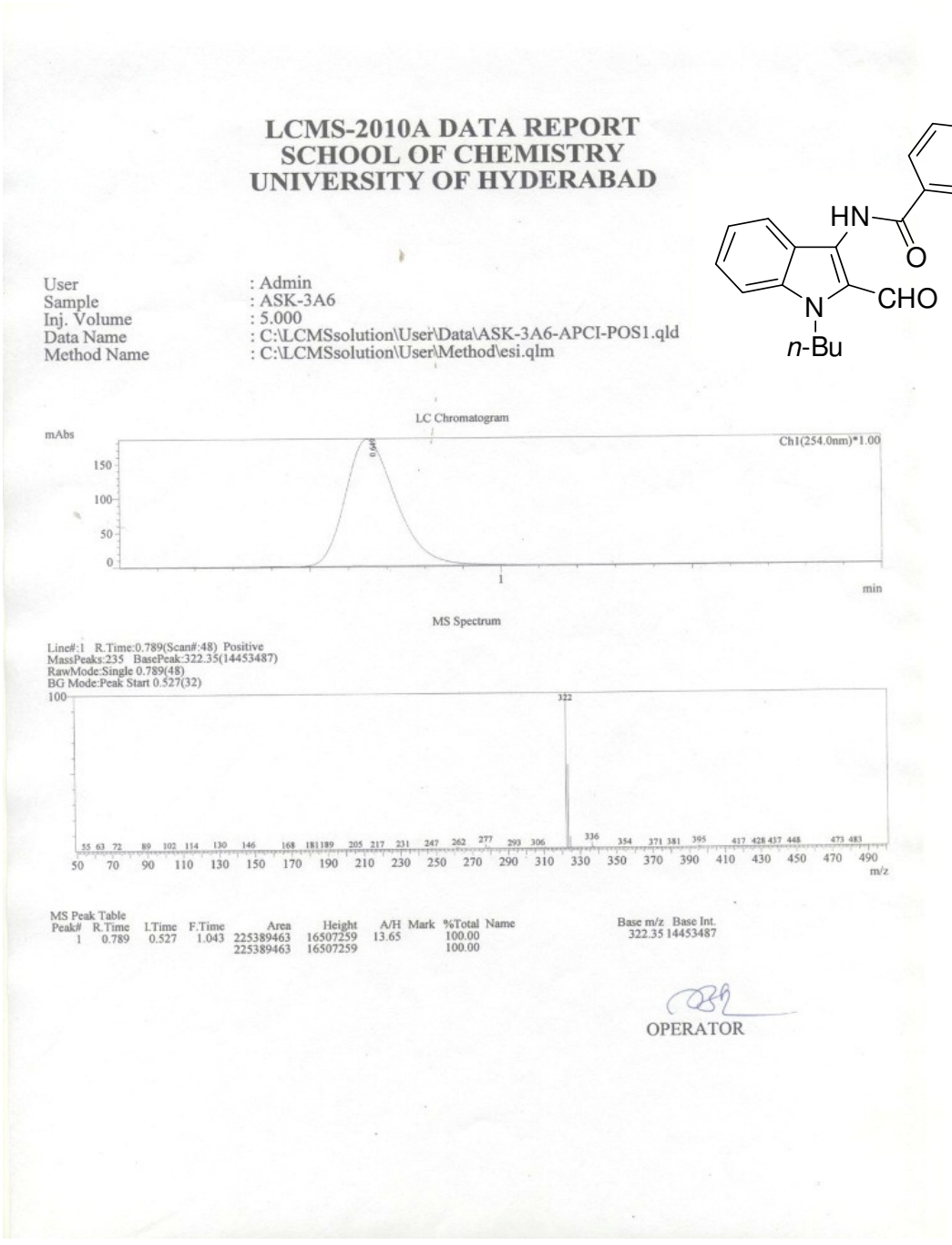
LCMS of *N*-(1-butyl-2-formyl-1*H*-indol-3-yl)nicotinamide (1b):



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User : Admin
Sample : ASK-3A6
Inj. Volume : 5.000
Data Name : C:\LCMSsolution\User\Data\ASK-3A6-APCI-POS1.qld
Method Name : C:\LCMSsolution\User\Method\esi.qlm

LC Chromatogram

MS Spectrum

Line#:1 R.Time:0.789(Scan#:48) Positive
MassPeaks:235 BasePeak:322.35(14453487)
RawMode:Single 0.789(48)
BG Mode:Peak Start 0.527(32)

MS Peak Table

Peak#	R.Time	I.Time	F.Time	Area	Height	A/H	Mark	%Total	Name
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				225389463	16507259			100.00	

Base m/z Base Int.
322.35 14453487

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CHN Analysis of *N*-(1-butyl-2-formyl-1*H*-indol-3-yl)nicotinamide (1b):

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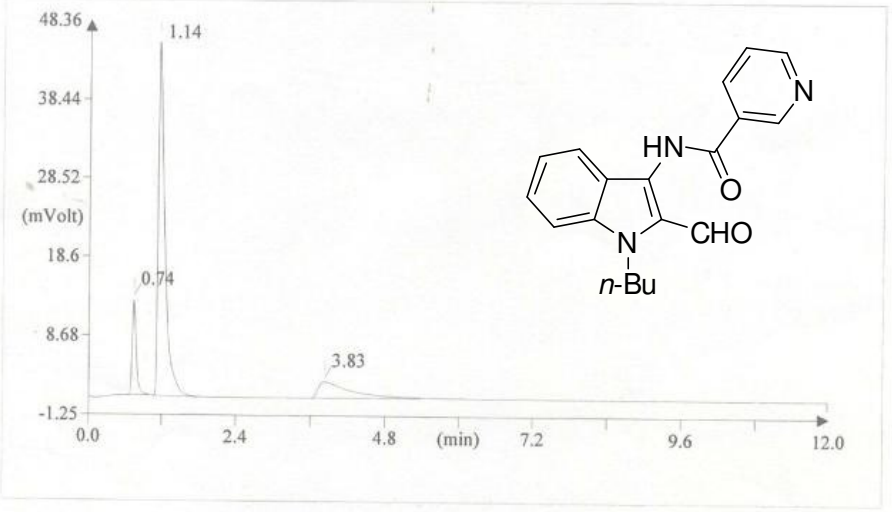
Method filename:	E:\Program Files\Thermo Finnigan\Eager 300 for EA1112\DATA\Sys_data_ex
Sample ID:	ASK-3A6 (# 132)
Analysis type:	UnkNown
Chromatogram filename:	UNK-30012012-2.dat
Sample weight:	1.113



Element Name	Element %	Ret. Time
Nitrogen	13. 15	0. 74
Carbon	71. 22	1. 14
Hydrogen	5. 89	3. 83

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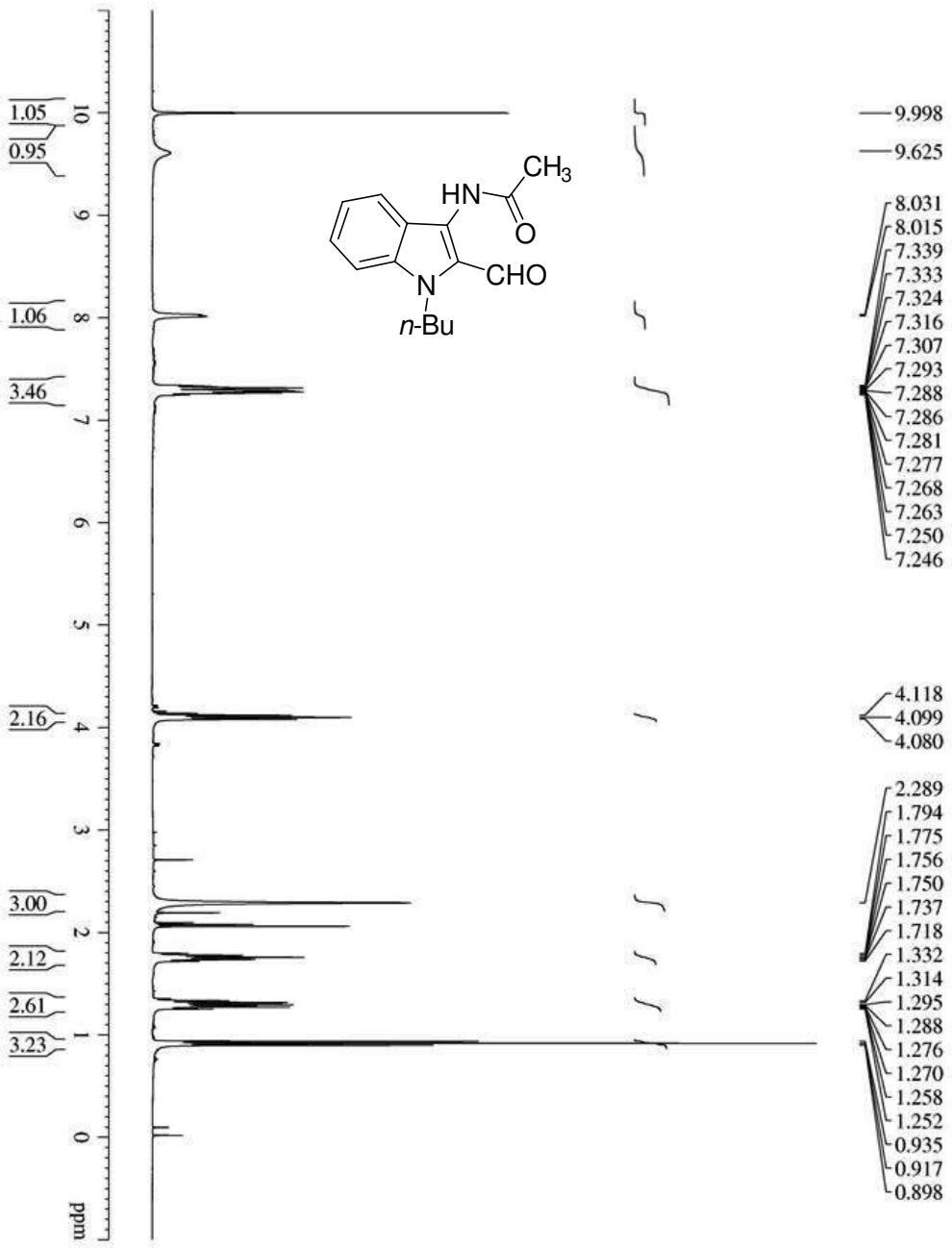
^{1}H NMR of *N*-(1-butyl-2-formyl-1*H*-indol-3-yl)acetamide (1c):



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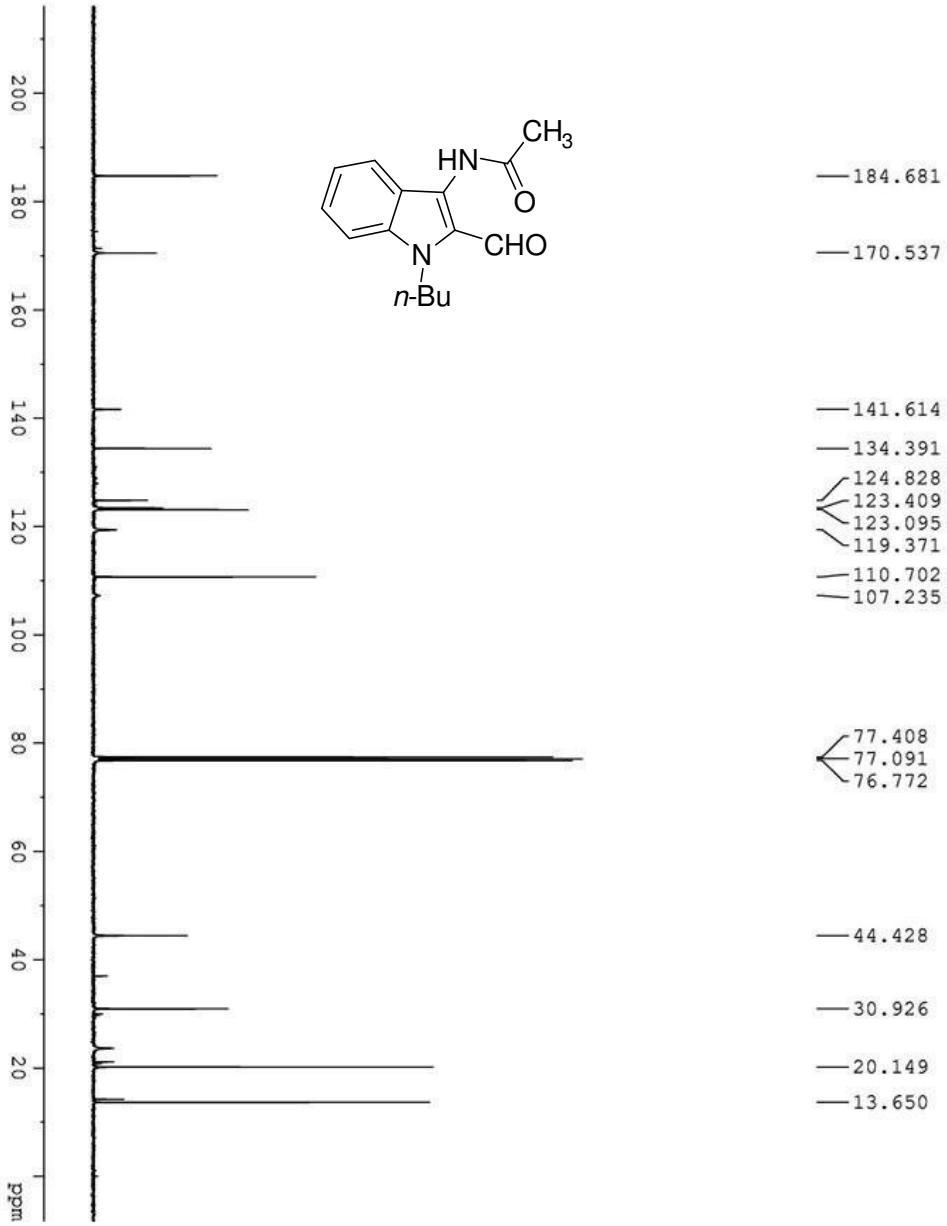
^{13}C NMR of *N*-(1-butyl-2-formyl-1*H*-indol-3-yl)acetamide (1c):



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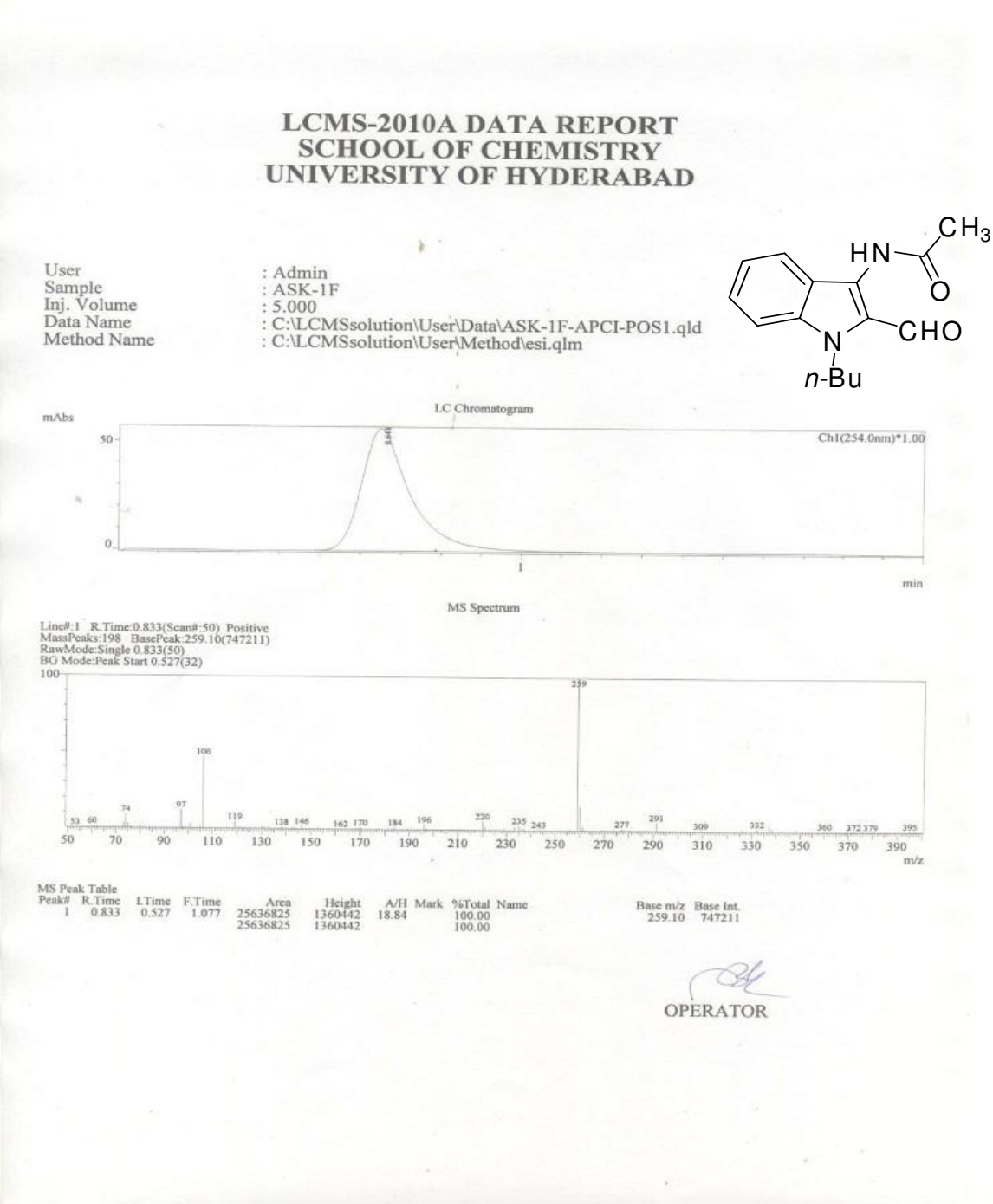
LCMS of *N*-(1-butyl-2-formyl-1*H*-indol-3-yl)acetamide (1c):



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User : Admin
Sample : ASK-1F
Inj. Volume : 5.000
Data Name : C:\LCMSsolution\User\Data\ASK-1F-APCI-POS1.qld
Method Name : C:\LCMSsolution\User\Method\esi.qlm

MS Spectrum

Line#:1 R.Time:0.833(Scan#:50) Positive
MassPeaks:198 BasePeak:259.10(747211)
RawMode:Single 0.833(50)
BG Mode:Peak Start 0.527(32)

MS Peak Table

Peak#	R.Time	I.Time	F.Time	Area	Height	A/H	Mark	%Total	Name	Base m/z	Base Int.
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				25636825	1360442			100.00			

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CHN Analysis of *N*-(1-butyl-2-formyl-1*H*-indol-3-yl)acetamide (1c):

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Method filename:	E:\Program Files\Thermo Finnigan\Eager 300 for EA1112\DATA\Sys_data_ex
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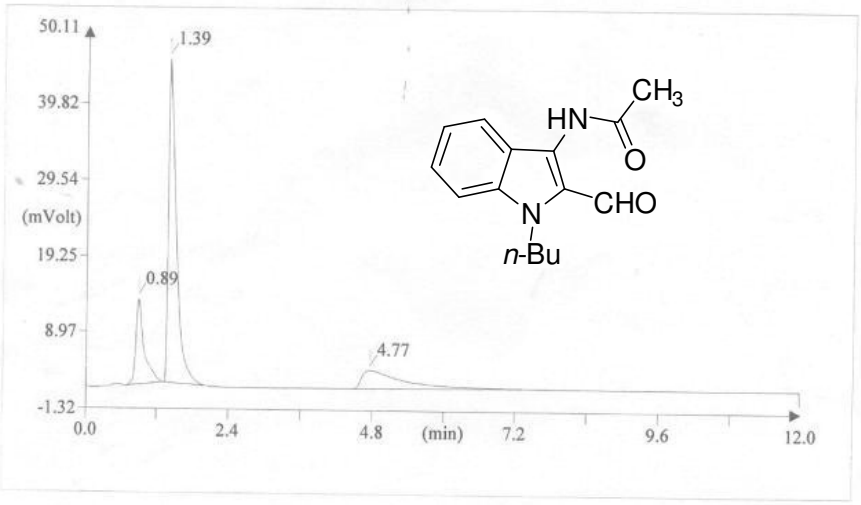
Sample weight:	.986



Element Name	Element %	Ret. Time
Nitrogen	10. 76	0. 89
Carbon	69. 81	1. 39
Hydrogen	7. 12	4. 77

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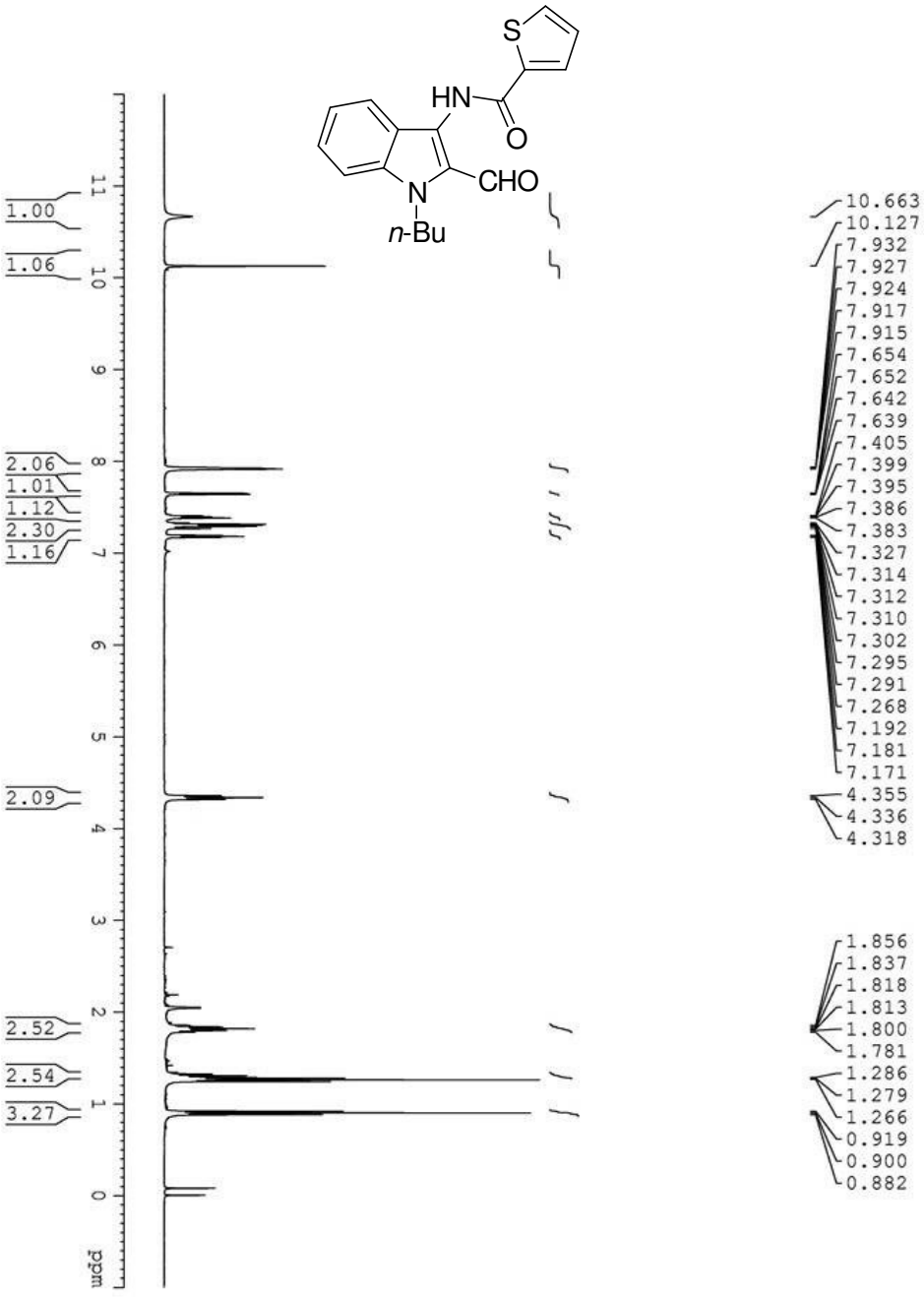
^{1}H NMR of *N*-(1-butyl-2-formyl-1*H*-indol-3-yl)thiophene-2-carboxamide (1d):



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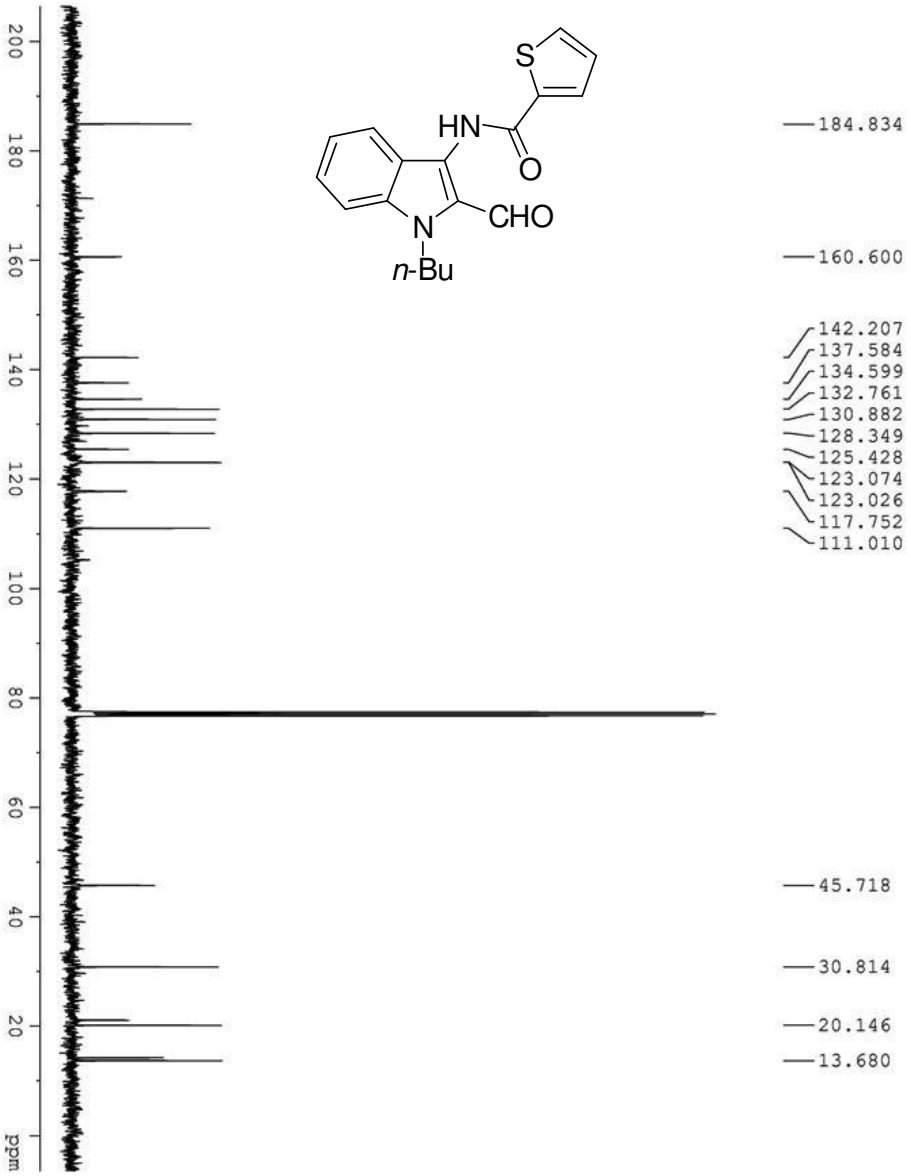
^{13}C NMR of *N*-(1-butyl-2-formyl-1*H*-indol-3-yl)thiophene-2-carboxamide (1d):



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LCMS of *N*-(1-butyl-2-formyl-1*H*-indol-3-yl)thiophene-2-carboxamide (1d):

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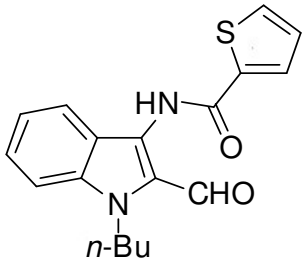
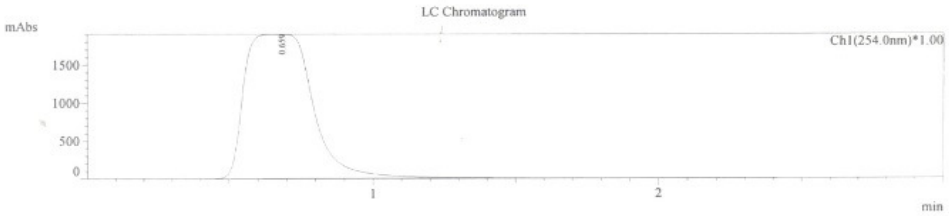
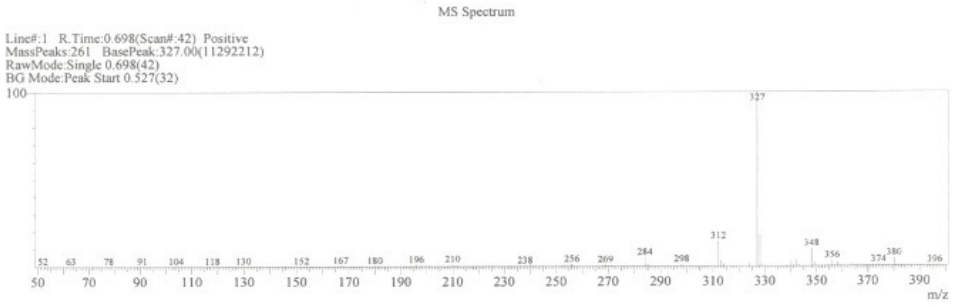


User : Admin
Sample : ASK-3A4
Inj. Volume : 5.000
Data Name : C:\LCMSsolution\User\Data\ASK-3A4-APCI-POS1.qld
Method Name : C:\LCMSsolution\User\Method\esi.qlm





MS Peak Table

Peak#	R.Time	I.Time	F.Time	Area	Height	A/H	Mark	%Total	Name
1	0.698	0.527	1.443	1021214308	40617528	25.14		100.00	
				1021214308	40617528			100.00	

Base m/z Base Int.
327.00 11292212

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CHN of *N*-(1-butyl-2-formyl-1*H*-indol-3-yl)thiophene-2-carboxamide (1d):

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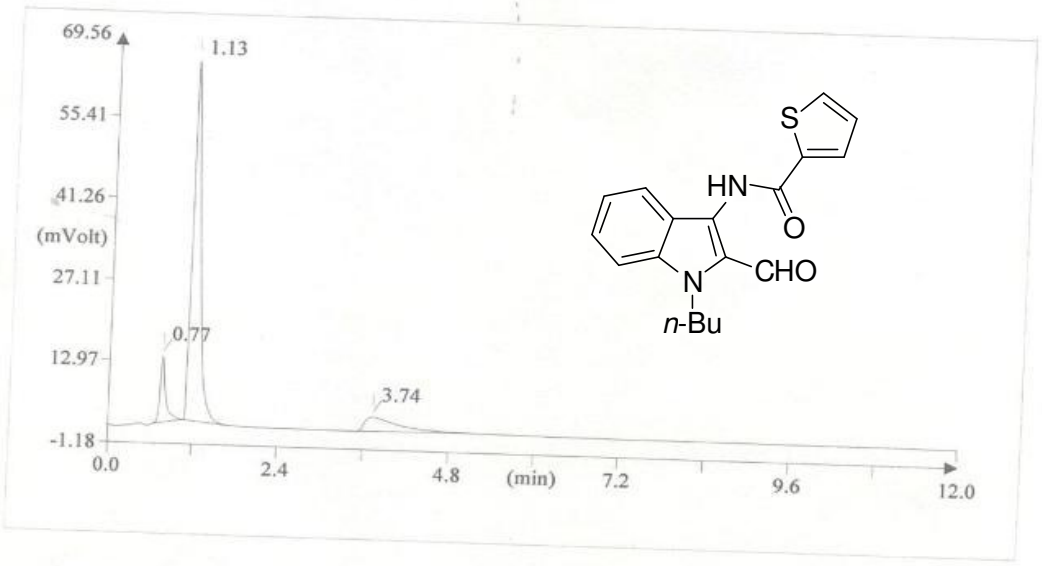
Method filename:	E:\Program Files\Thermo Finnigan\Eager 300 for EA1112\DATA\Sys_data_ex
Sample ID:	ASK-3A4 (# 137)
Analysis type:	UnkNown
Chromatogram filename:	UNK-30012012-7.dat
Sample weight:	1.175



Element Name	Element %	Ret. Time
Nitrogen	8. 49	0. 77
Carbon	66. 38	1. 13
Hydrogen	5. 51	3. 74

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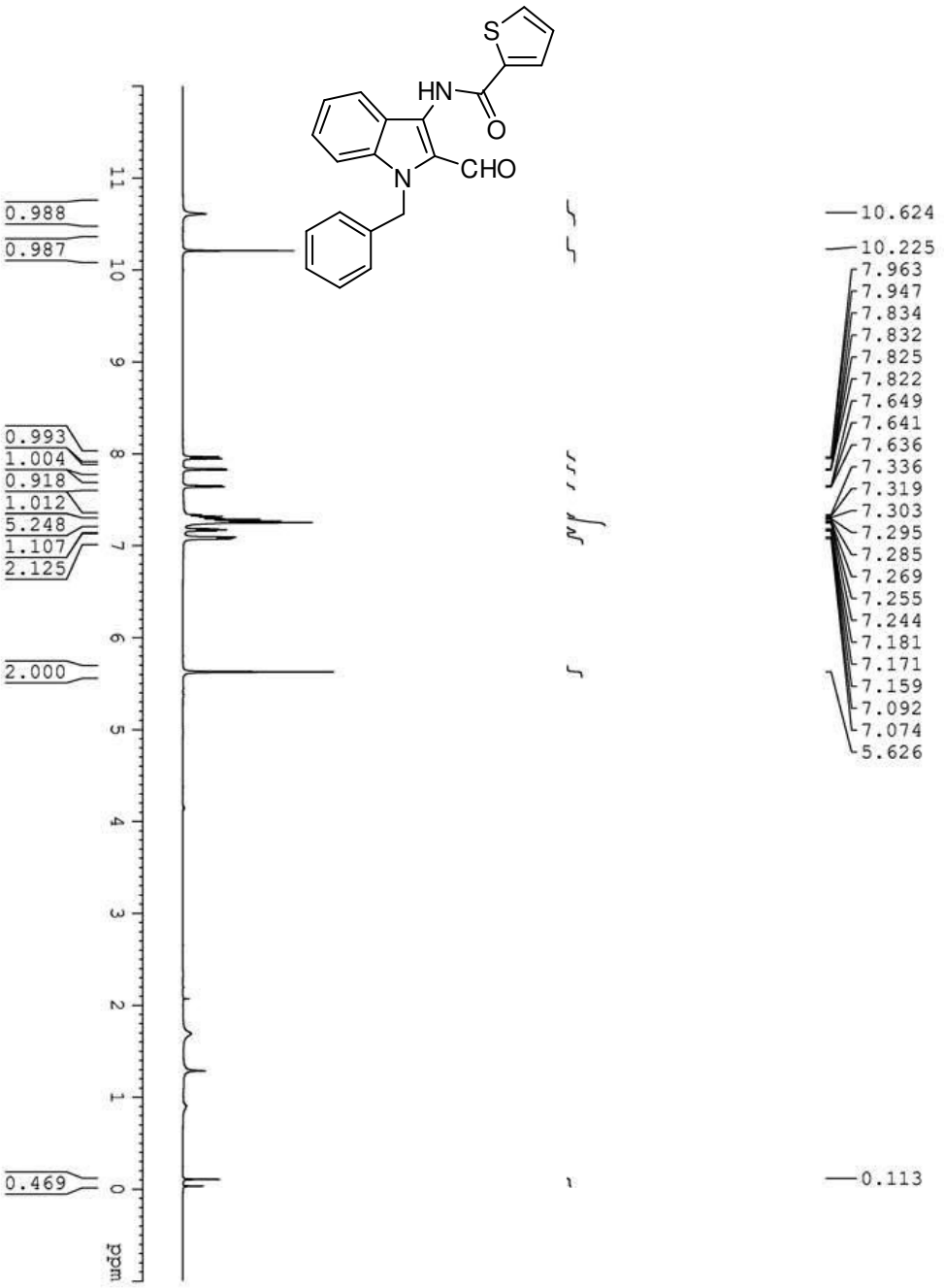
^{1}H NMR of *N*-(1-benzyl-2-formyl-1*H*-indol-3-yl)thiophene-2-carboxamide (1e):



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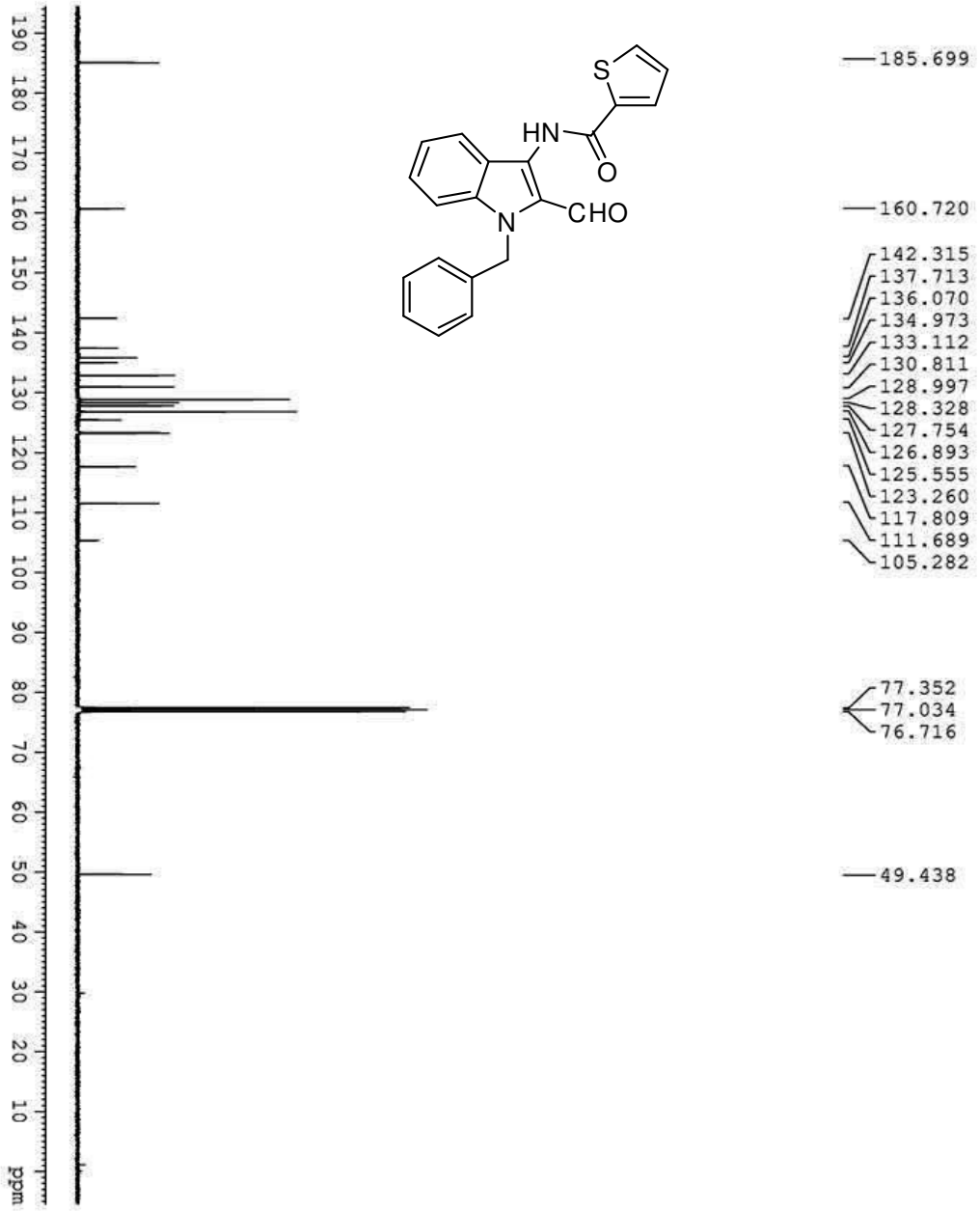
^{13}C NMR of *N*-(1-benzyl-2-formyl-1*H*-indol-3-yl)thiophene-2-carboxamide (1e):



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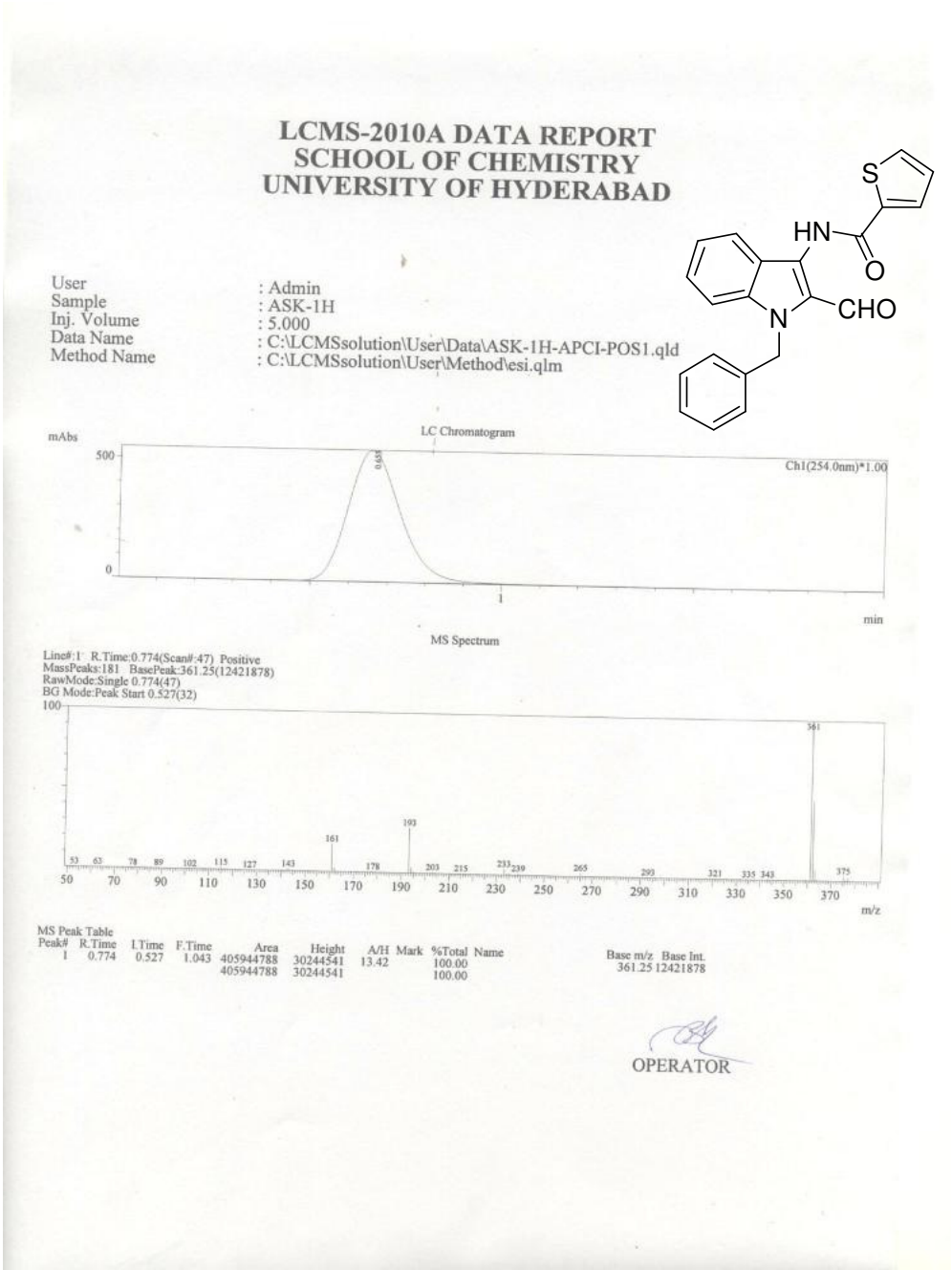
LCMS of *N*-(1-benzyl-2-formyl-1*H*-indol-3-yl)thiophene-2-carboxamide (1e):



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CHN Analysis of *N*-(1-benzyl-2-formyl-1*H*-indol-3-yl)thiophene-2-carboxamide (1e):

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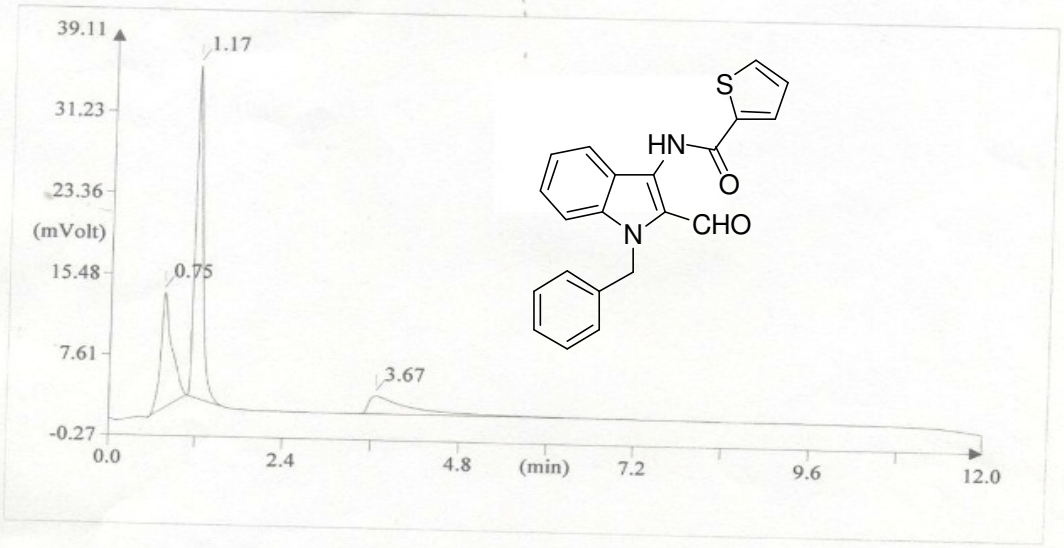
Method filename: E:\Program Files\Thermo Finnigan\Eager 300 for EA1112\DATA\Sys_data_ex
Sample ID: ASK-2AF9 (# 141)
Analysis type: UnkNown
Chromatogram filename: UNK-30012012-11.dat
Sample weight: 1.021



Element Name	Element %	Ret. Time
Nitrogen	7. 67	0. 75
Carbon	69. 71	1. 17
Hydrogen	4. 38	3. 67

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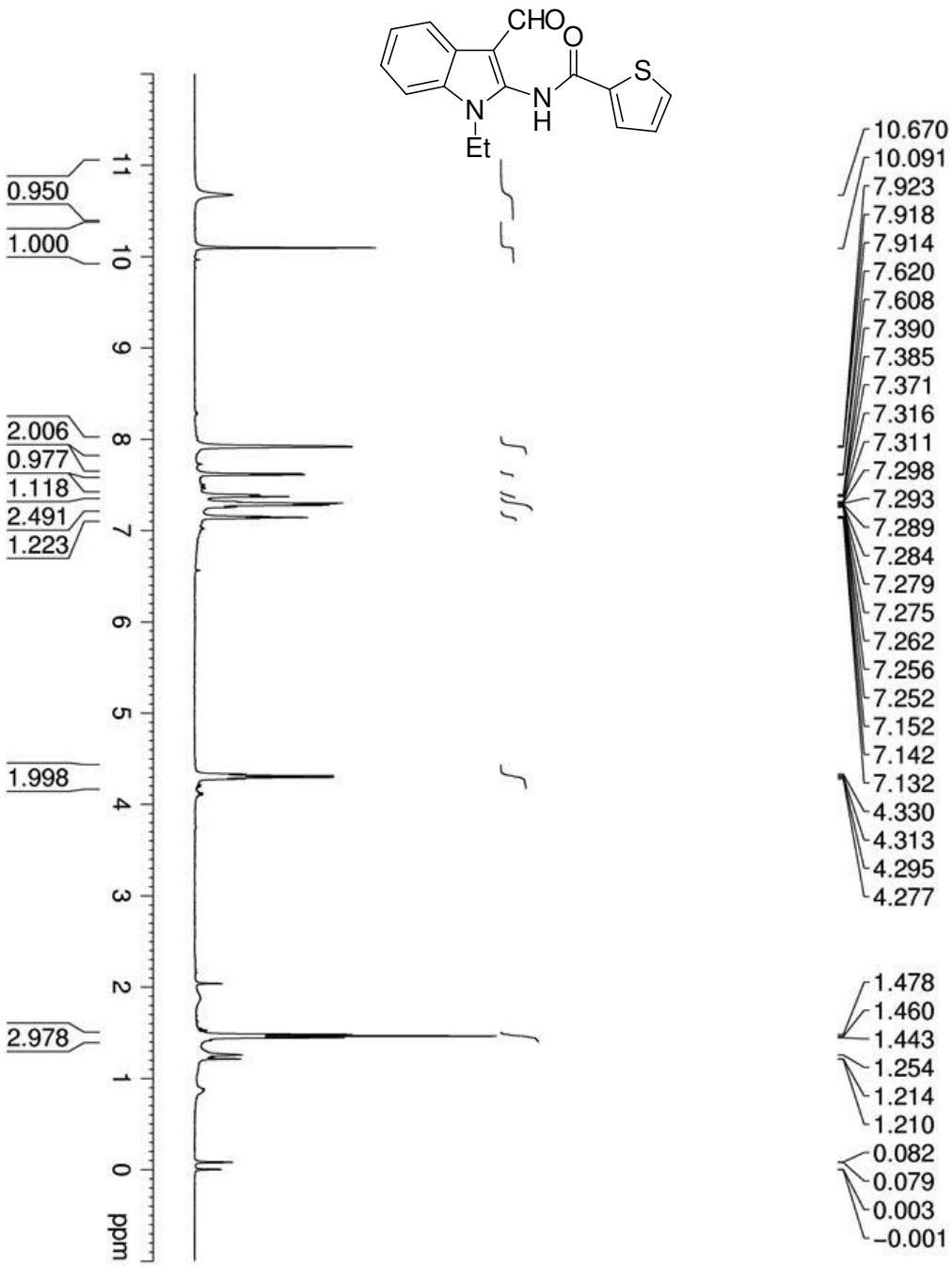
^{1}H NMR of *N*-(1-ethyl-3-formyl-1*H*-indol-2-yl)thiophene-2-carboxamide (1f):



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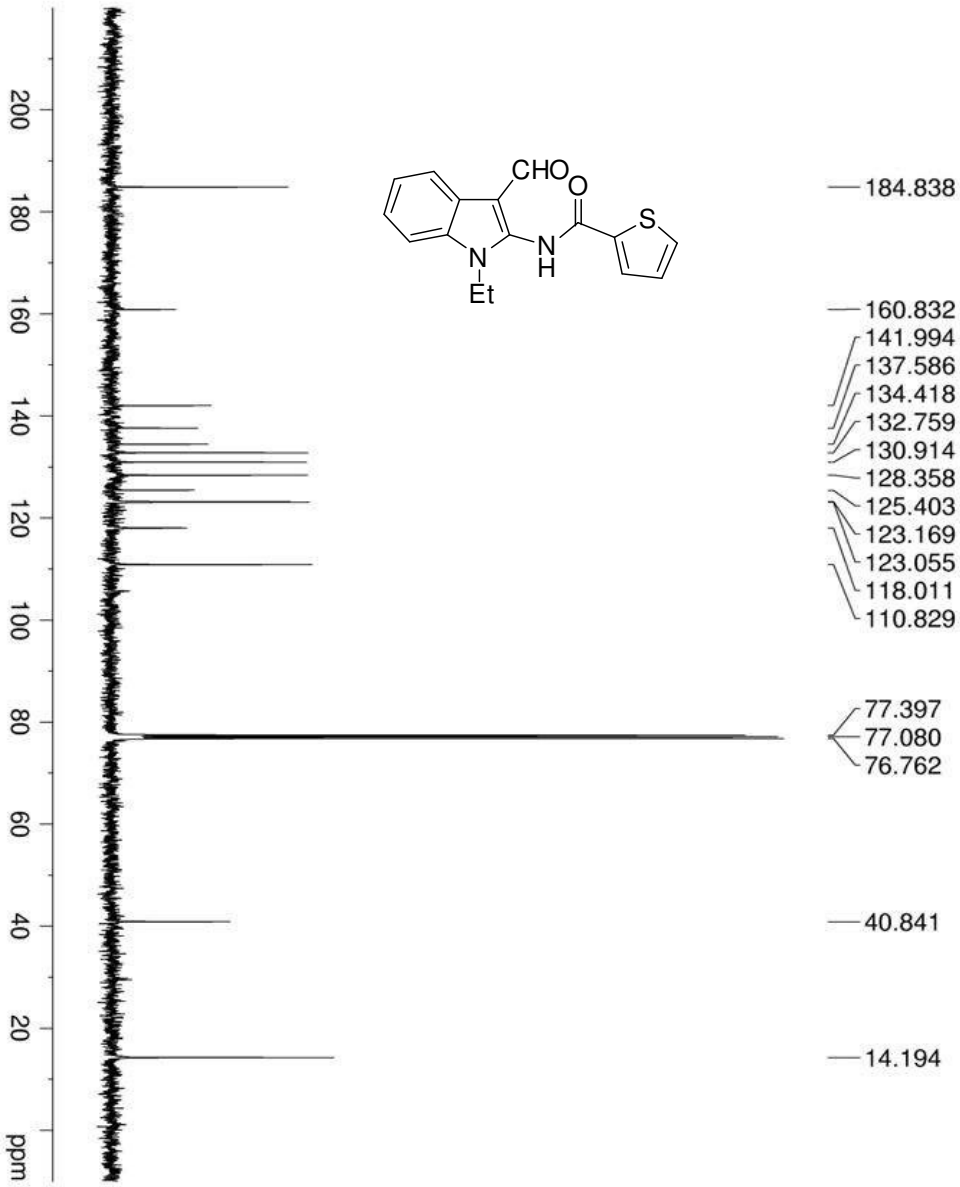
^{13}C NMR of *N*-(1-ethyl-3-formyl-1*H*-indol-2-yl)thiophene-2-carboxamide (1f):



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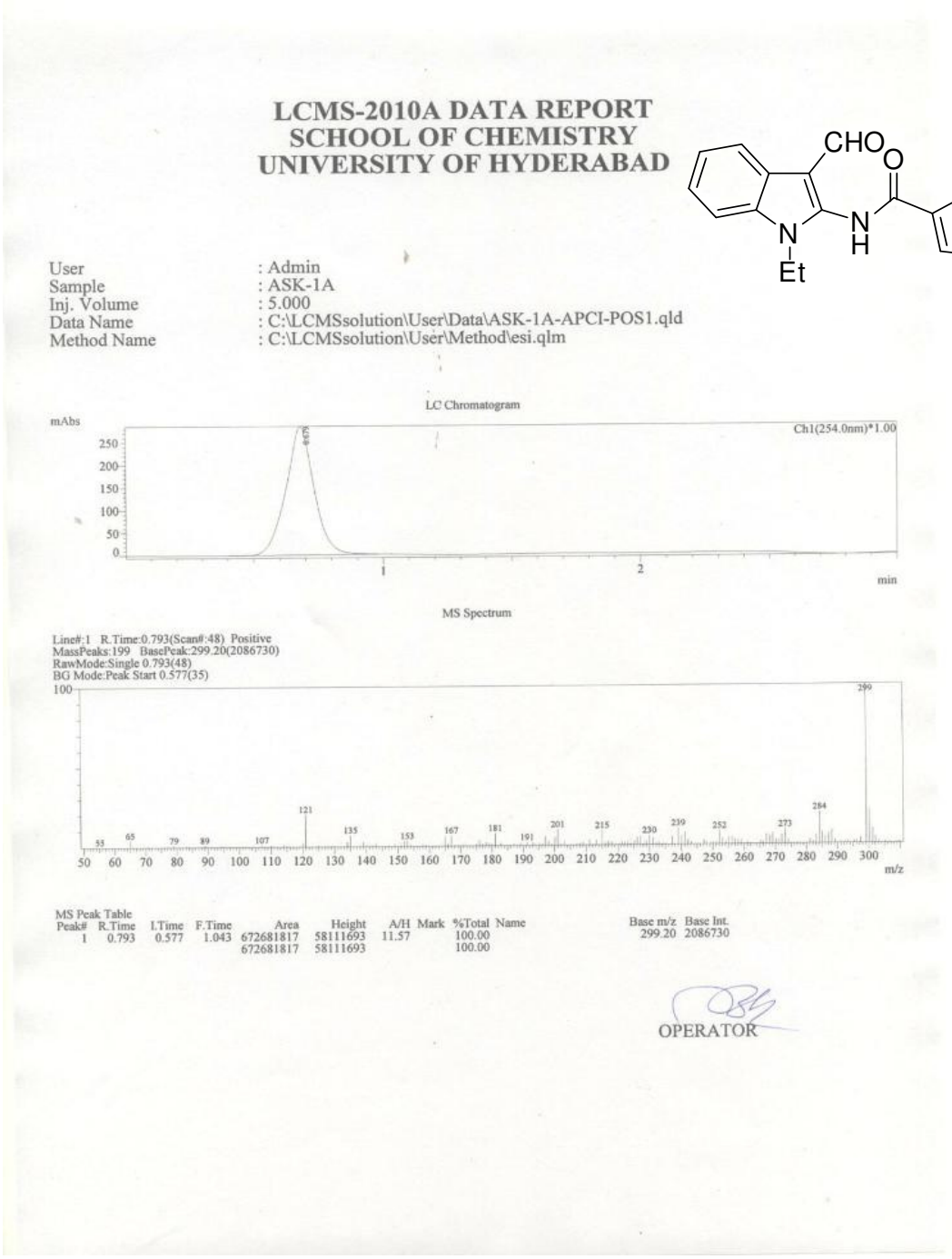
LCMS of *N*-(1-ethyl-3-formyl-1*H*-indol-2-yl)thiophene-2-carboxamide (1f):



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CHN Analysis of *N*-(1-ethyl-3-formyl-1*H*-indol-2-yl)thiophene-2-carboxamide (1f):

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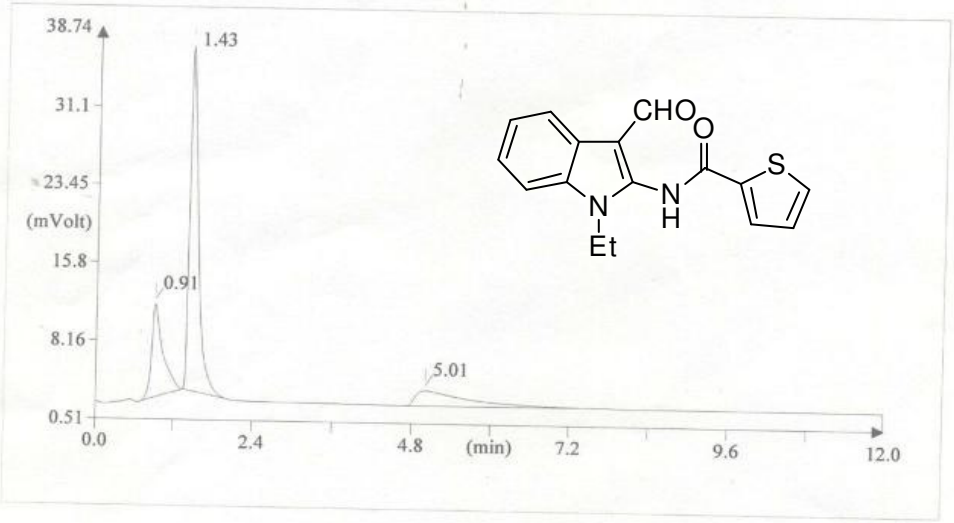
Method filename: E:\Program Files\Thermo Finnigan\Eager 300 for EA1112\DATA\Sys_data_ex
Sample ID: ASK-1A (# 106) → 1f
Analysis type: UnkNown
Chromatogram filename: UNK-24012012-6.dat
Sample weight: .976



Element Name	Element %	Ret. Time
Nitrogen	9. 28	0. 91
Carbon	64. 32	1. 43
Hydrogen	4. 79	5. 01

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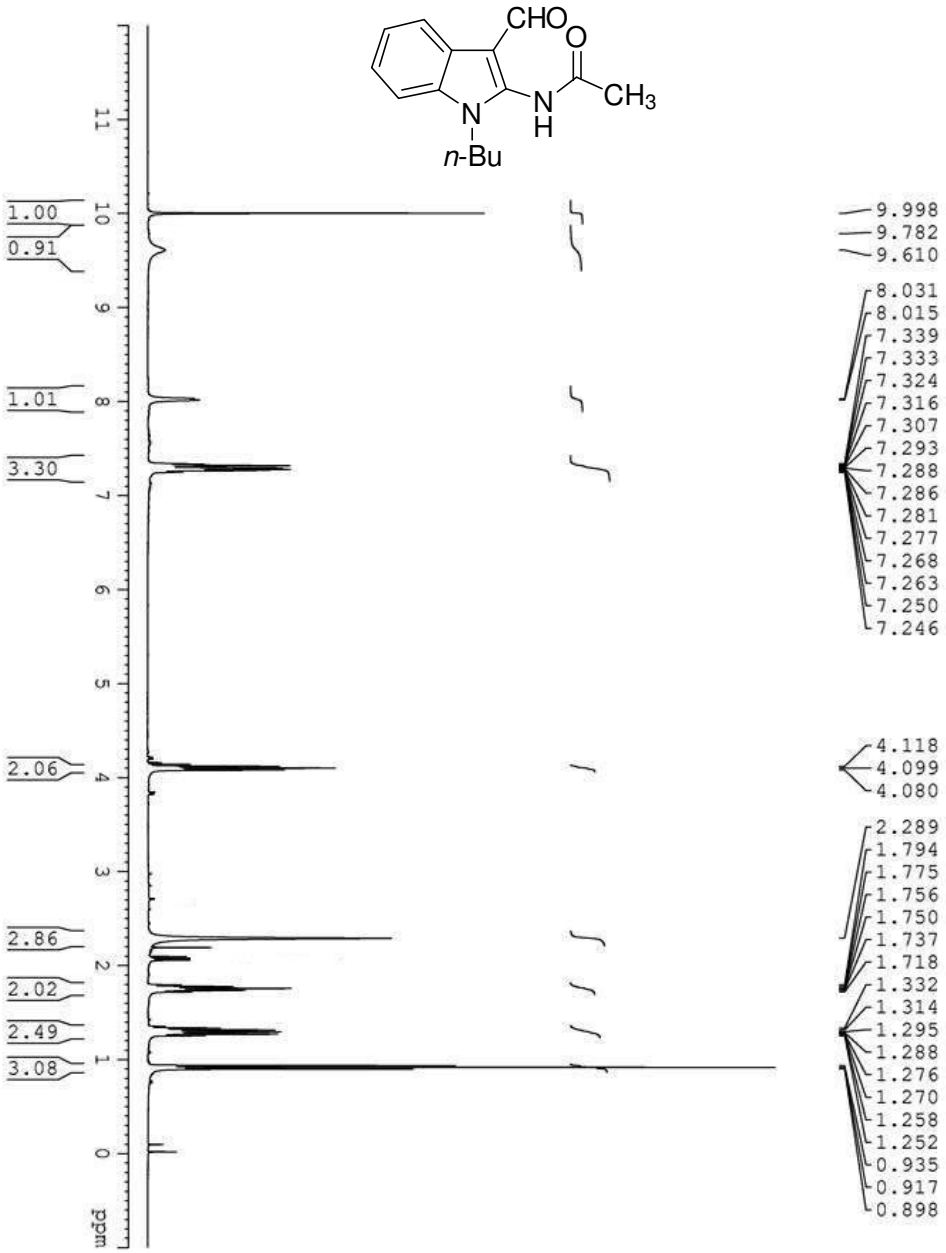
^{1}H NMR of *N*-(1-butyl-3-formyl-1*H*-indol-2-yl)acetamide (1g):



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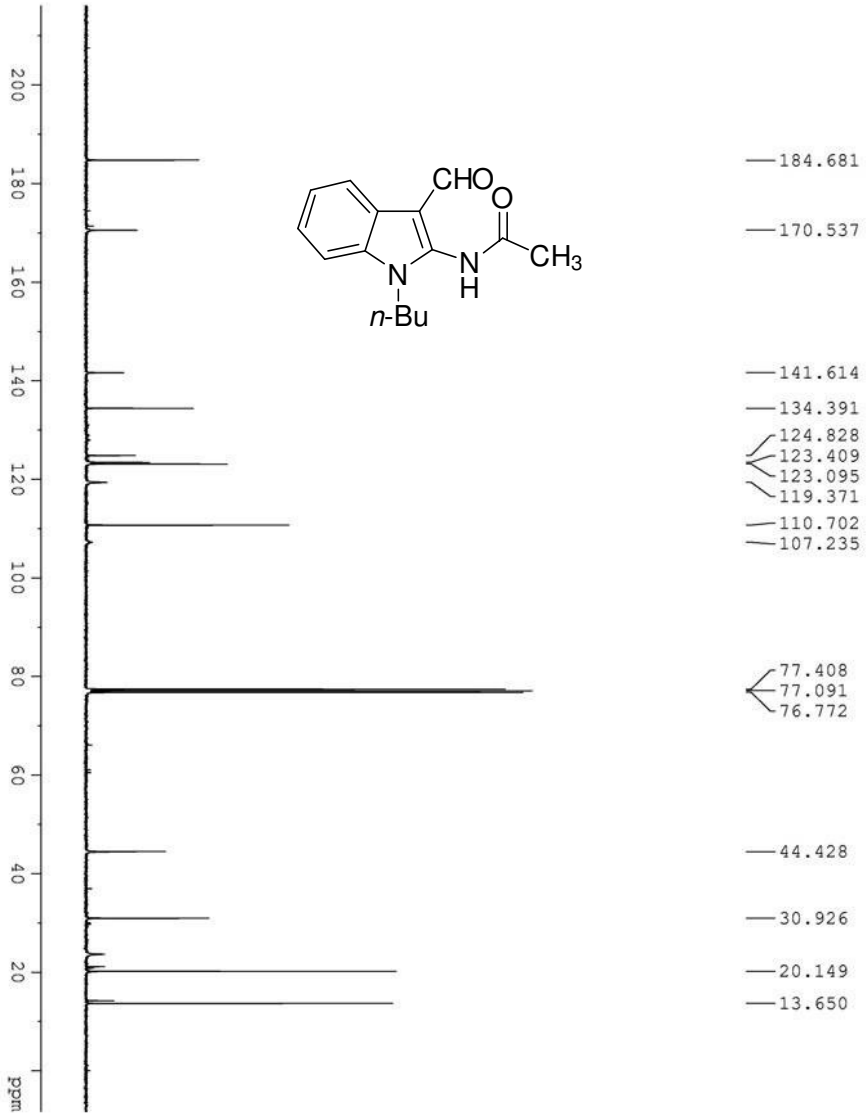
^{13}C NMR of *N*-(1-butyl-3-formyl-1*H*-indol-2-yl)acetamide (1g):



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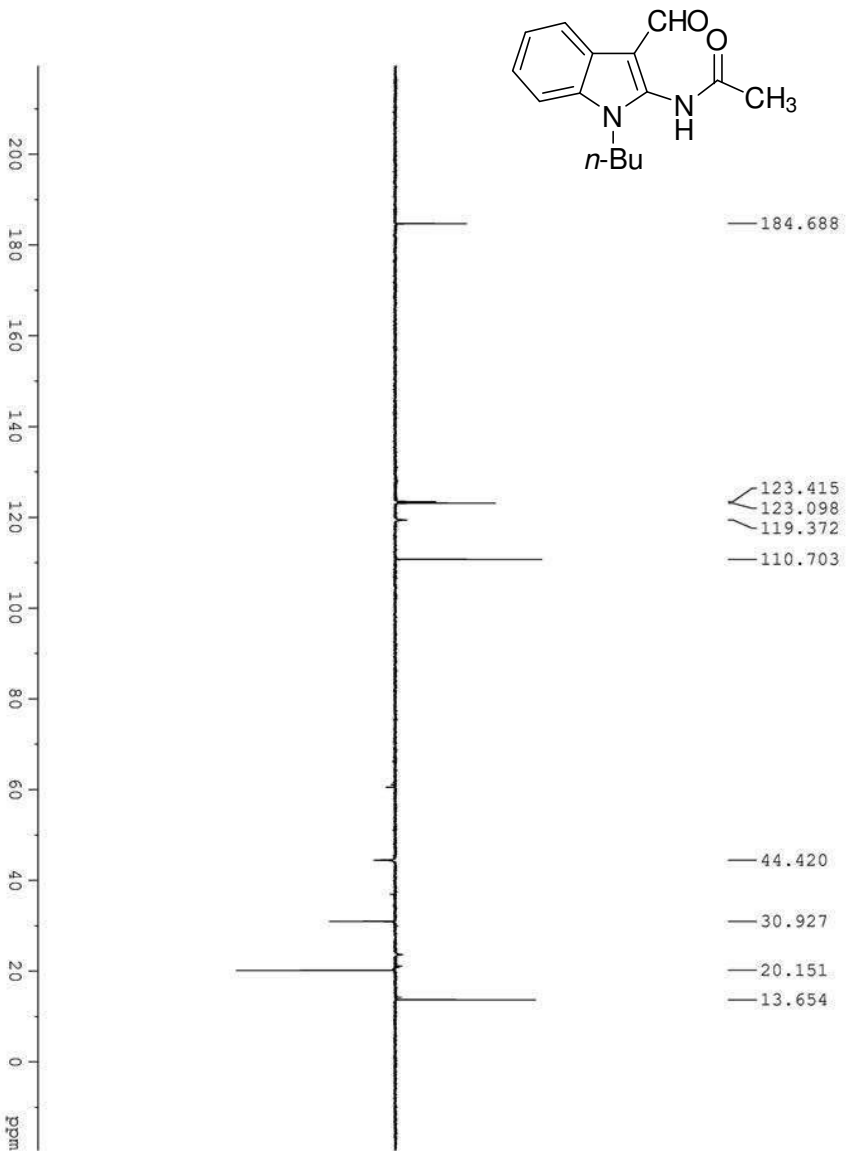
DEPT of *N*-(1-butyl-3-formyl-1*H*-indol-2-yl)acetamide (1g):



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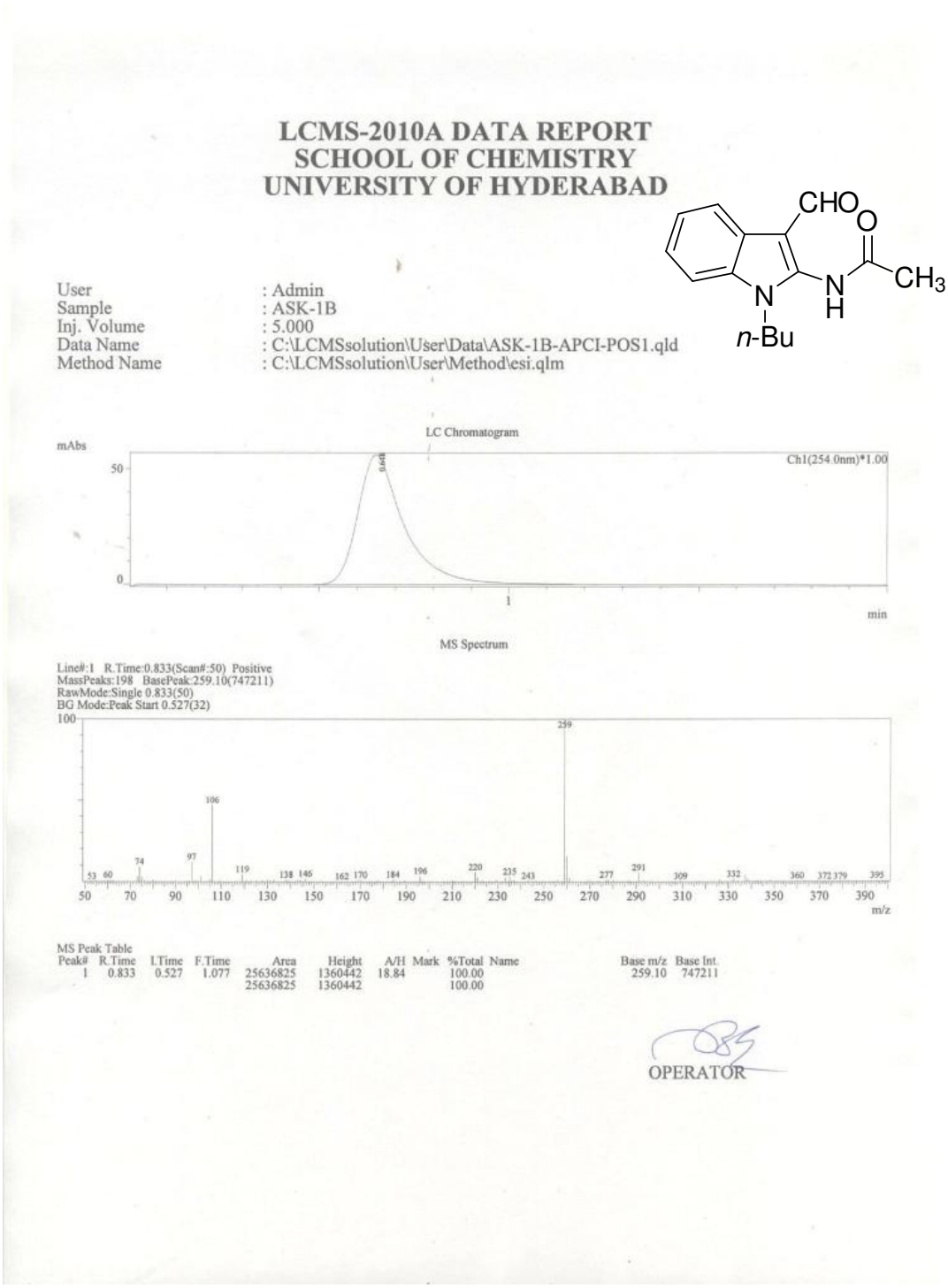
LCMS of *N*-(1-butyl-3-formyl-1*H*-indol-2-yl)acetamide (1g):



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CHN Analysis of *N*-(1-butyl-3-formyl-1*H*-indol-2-yl)acetamide (1g):

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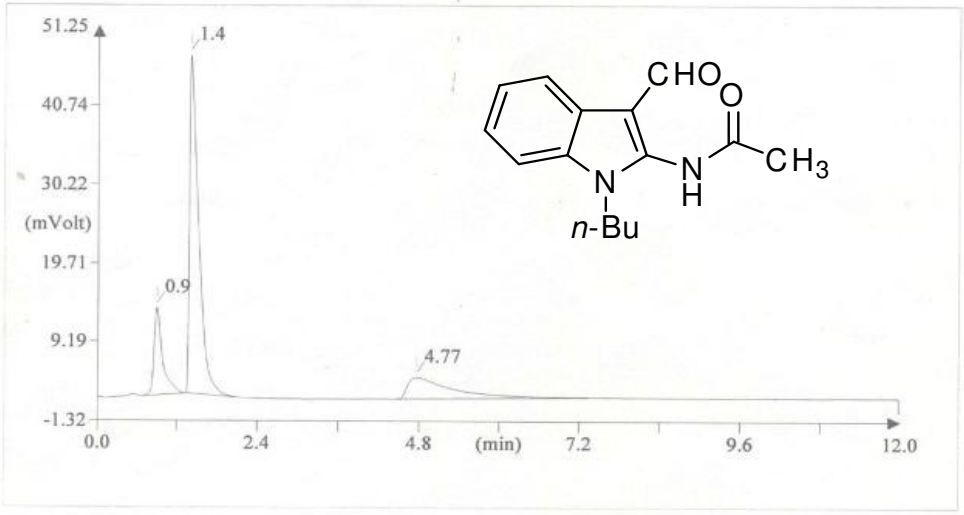
Method filename:	E:\Program Files\Thermo Finnigan\Eager 300 for EA1112\DATA\Sys_data_ex
Sample ID:	ASK-1B (# 114) → 1g ✓
Analysis type:	UnkNown
Chromatogram filename:	UNK-24012012-14.dat
Sample weight:	.991



Element Name	Element %	Ret. Time
Nitrogen	10. 76	0. 90
Carbon	69. 85	1. 40
Hydrogen	7. 12	4. 77

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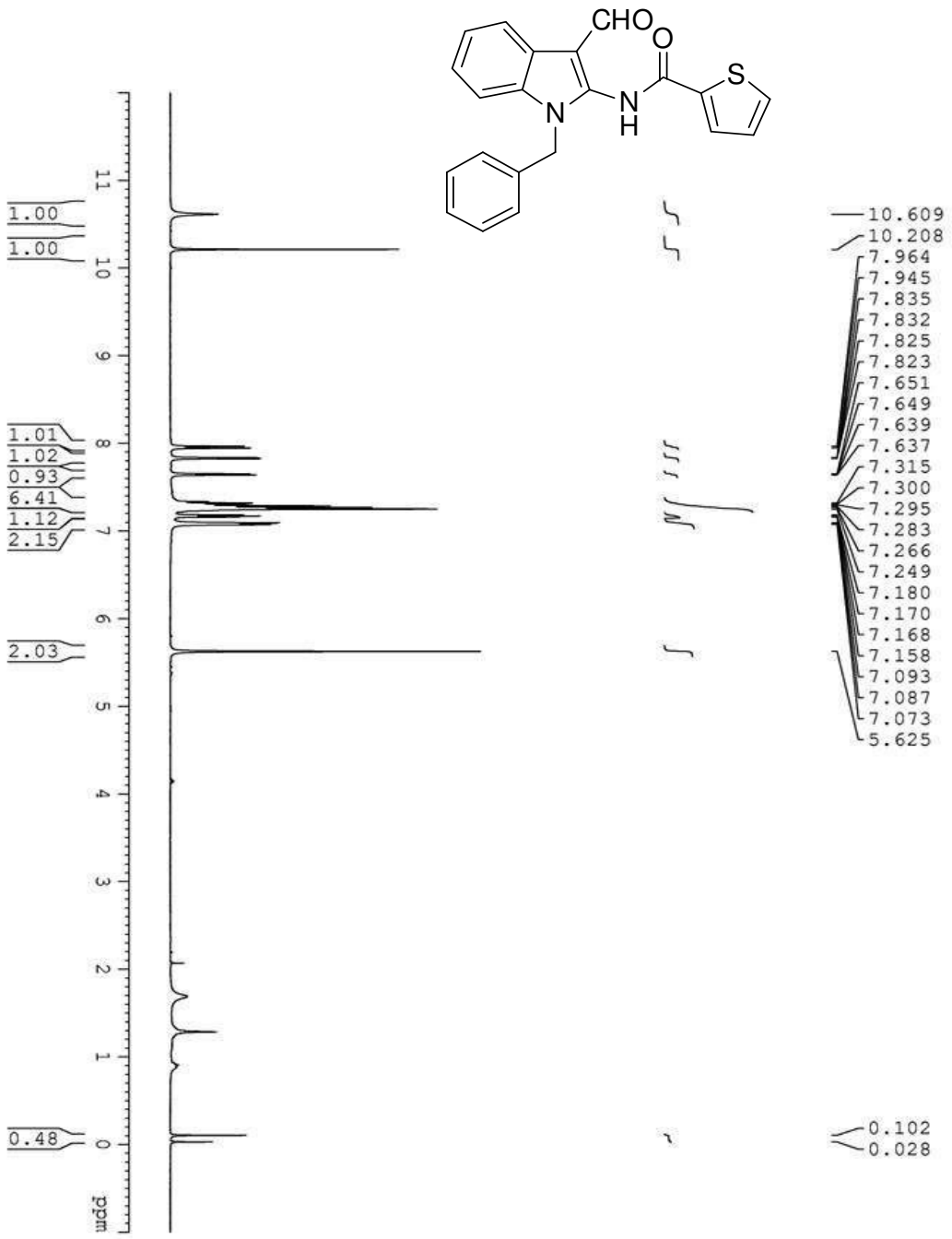
^{1}H NMR of *N*-(1-benzyl-3-formyl-1*H*-indol-2-yl)thiophene-2-carboxamide (1h):



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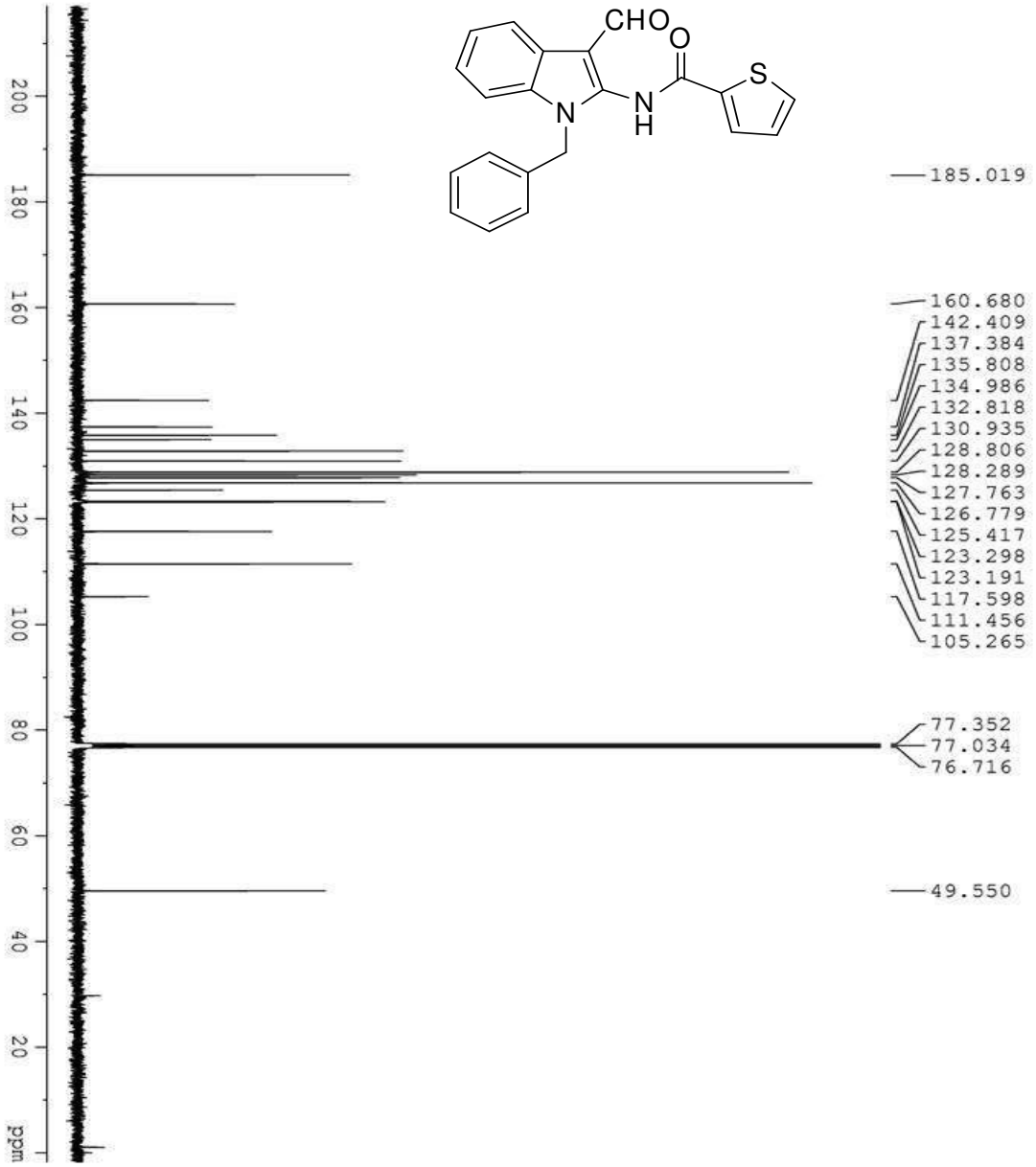
^{13}C NMR of *N*-(1-benzyl-3-formyl-1*H*-indol-2-yl)thiophene-2-carboxamide (1h):



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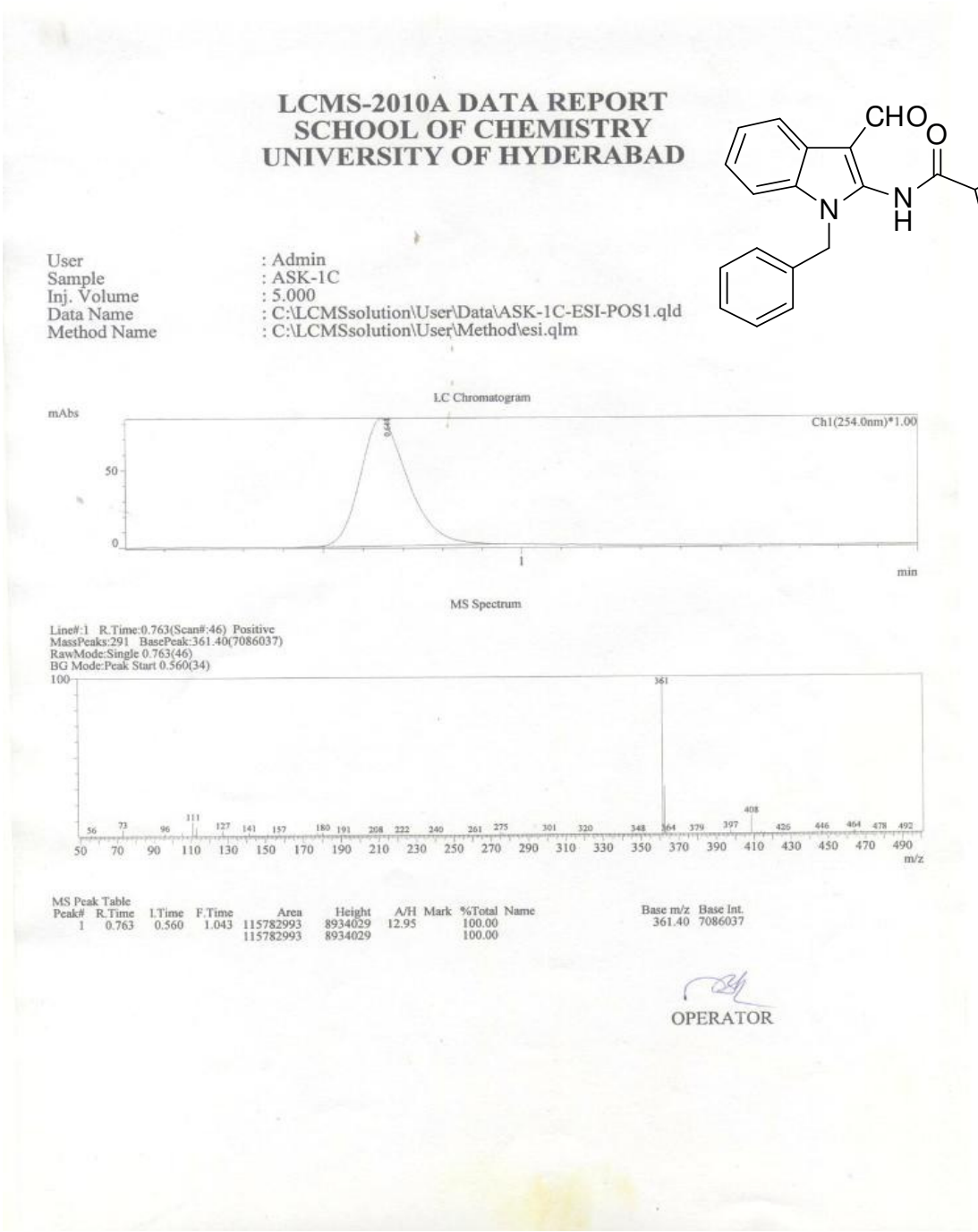
LCMS of *N*-(1-benzyl-3-formyl-1*H*-indol-2-yl)thiophene-2-carboxamide (1h):



LCMS-2010A DATA REPORT
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User : Admin
Sample : ASK-1C
Inj. Volume : 5.000
Data Name : C:\LCMSsolution\User\Data\ASK-1C-ESI-POS1.qld
Method Name : C:\LCMSsolution\User\Method\esi.qlm

LC Chromatogram

MS Spectrum

Line#:1 R.Time:0.763(Scan#:46) Positive
MassPeaks:291 BasePeak:361.40(7086037)
RawMode:Single 0.763(46)
BG Mode:Peak Start 0.560(34)

MS Peak Table

Peak#	R.Time	I.Time	F.Time	Area	Height	A/H	Mark	%Total	Name	Base m/z	Base Int.
1	0.763	0.560	1.043	115782993	8934029	12.95		100.00		361.40	7086037
				115782993	8934029			100.00			

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CHN Analysis of *N*-(1-benzyl-3-formyl-1*H*-indol-2-yl)thiophene-2-carboxamide (1h):

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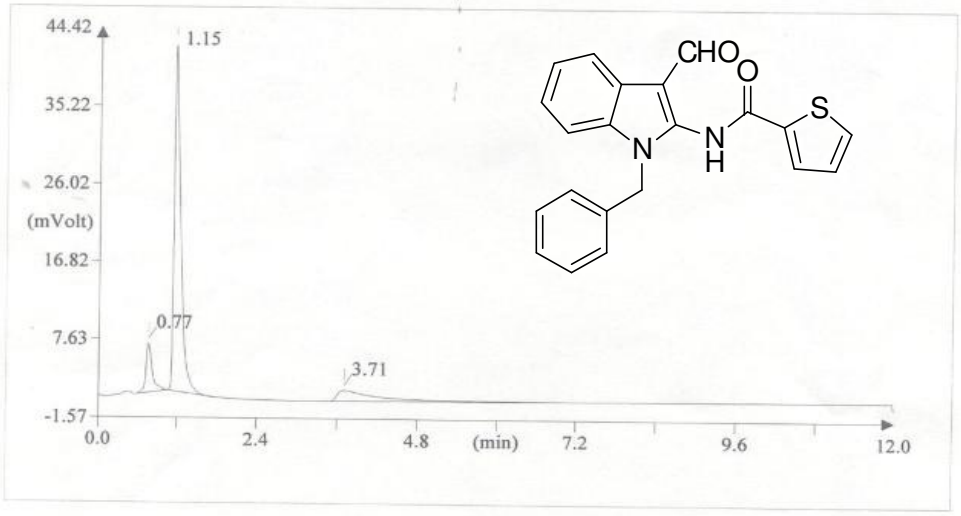
Method filename: E:\Program Files\Thermo Finnigan\Eager 300 for EA1112\DATA\Sys_data_ex
Sample ID: ASK-1H (# 113)
Analysis type: UnkNown
Chromatogram filename: UNK-24012012-13.dat
Sample weight: .983



Element Name	Element %	Ret. Time
Nitrogen	7. 86	0. 77
Carbon	69. 91	1. 15
Hydrogen	4. 52	3. 71

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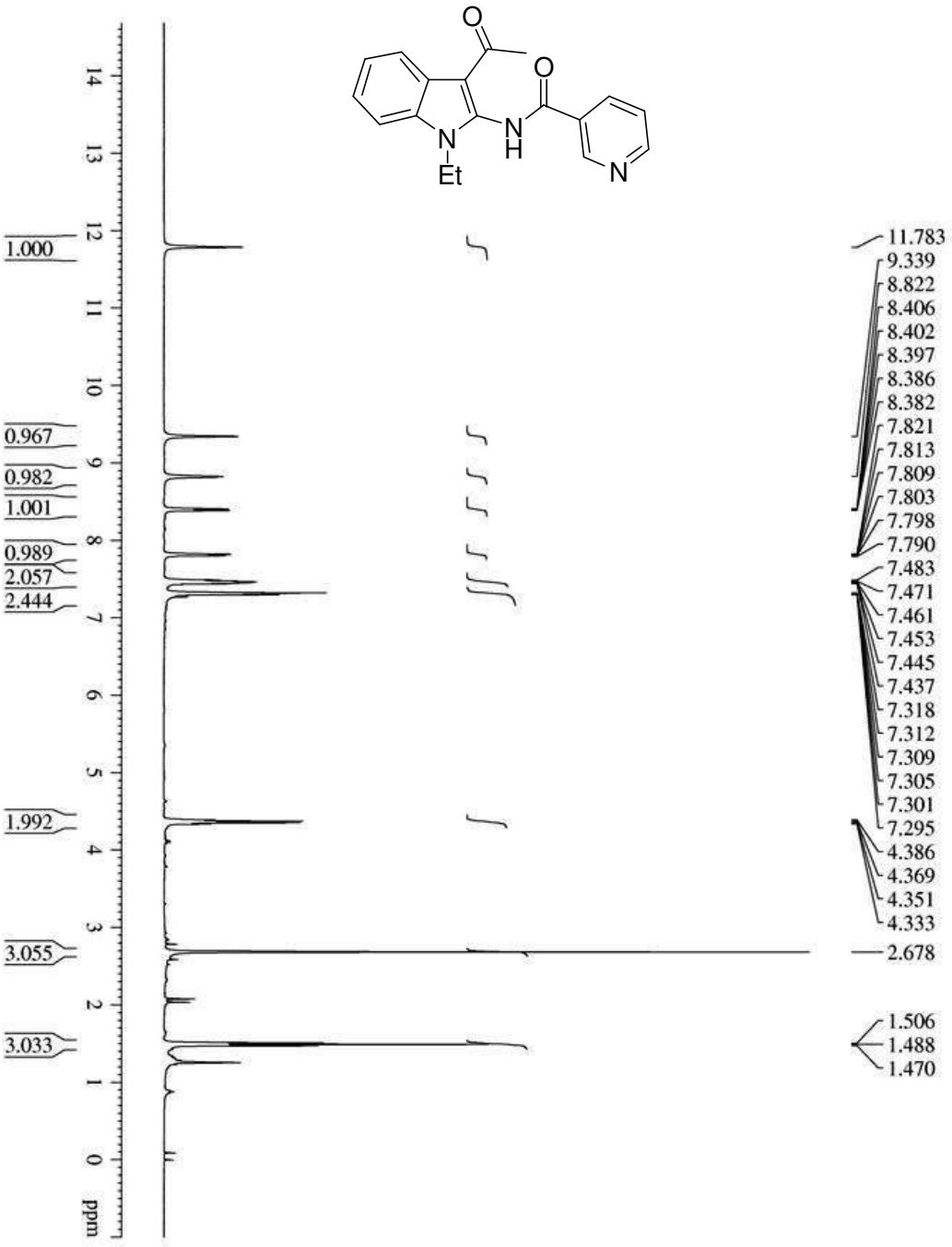
^{1}H NMR of *N*-(3-acetyl-1-ethyl-1*H*-indol-2-yl)nicotinamide (1i):



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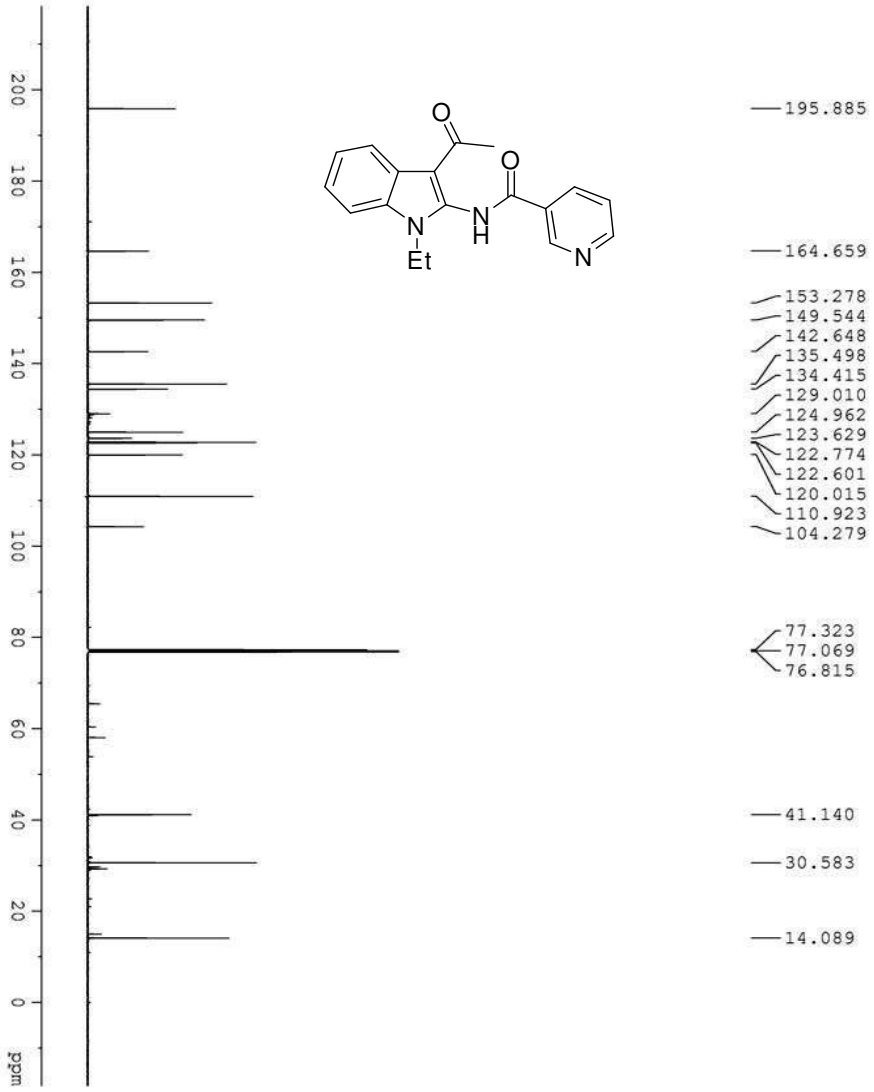
^{13}C NMR of *N*-(3-acetyl-1-ethyl-1*H*-indol-2-yl)nicotinamide (1i):



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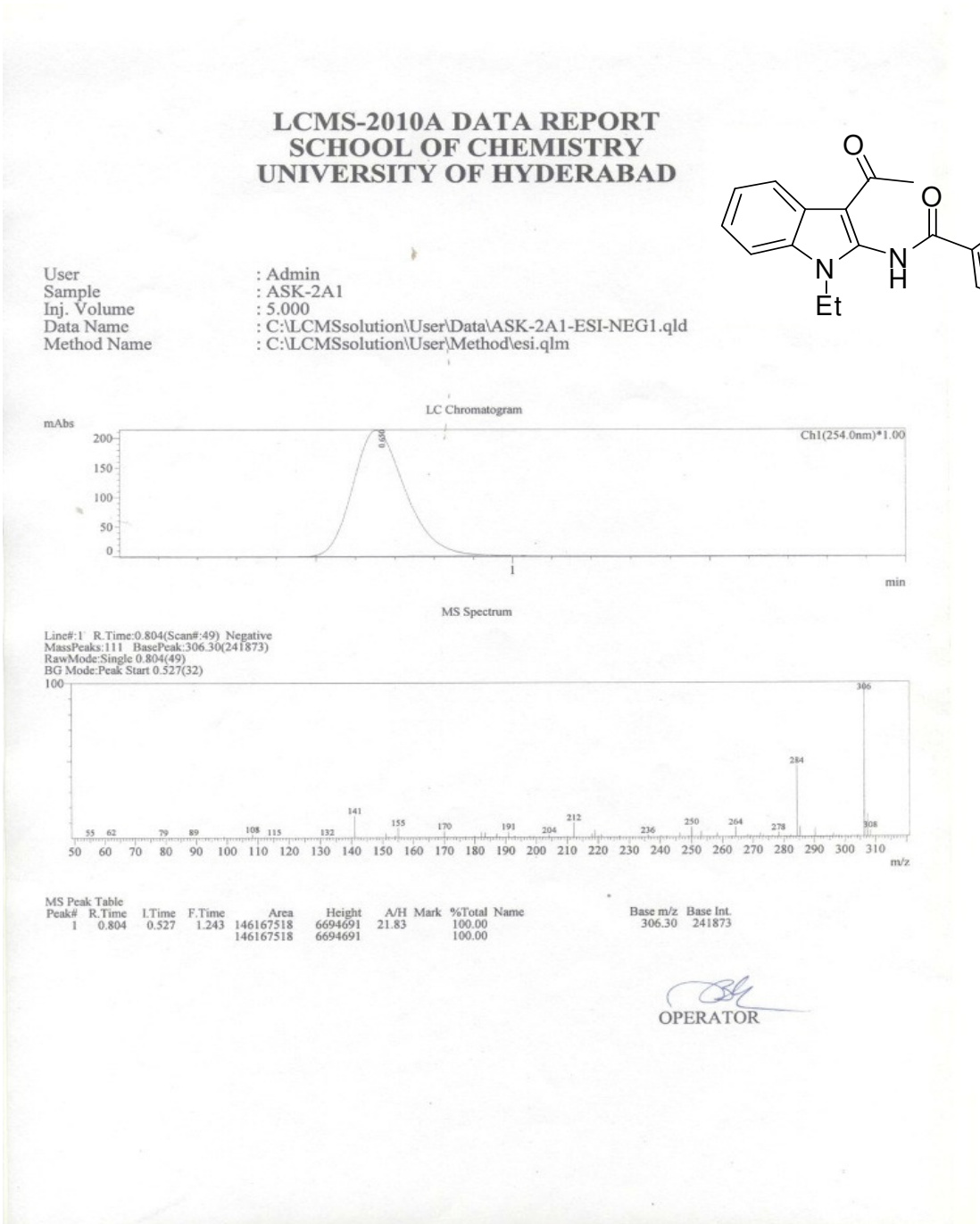
LCMS of *N*-(3-acetyl-1-ethyl-1*H*-indol-2-yl)nicotinamide (1i):



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CHN Analysis of *N*-(3-acetyl-1-ethyl-1*H*-indol-2-yl)nicotinamide (1i):

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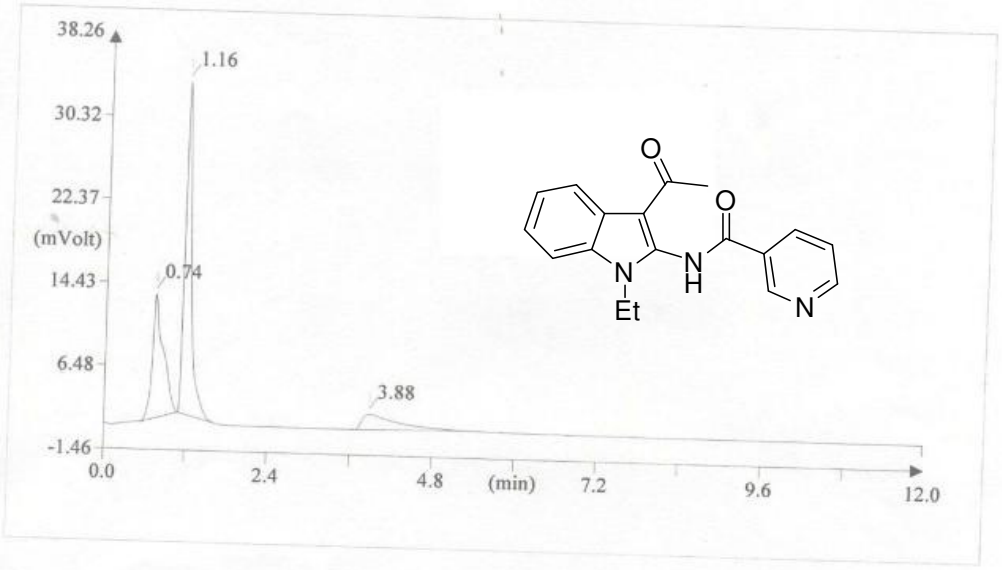
Method filename:	E:\Program Files\Thermo Finnigan\Eager 300 for EA1112\DATA\Sys_data_ex
Sample ID:	ASK-2A1 (# 146)
Analysis type:	UnkNown
Chromatogram filename:	UNK-30012012-16.dat
Sample weight:	1.018



Element Name	Element %	Ret. Time
Nitrogen	13. 76	0. 74
Carbon	70. 45	1. 16
Hydrogen	5. 51	3. 88

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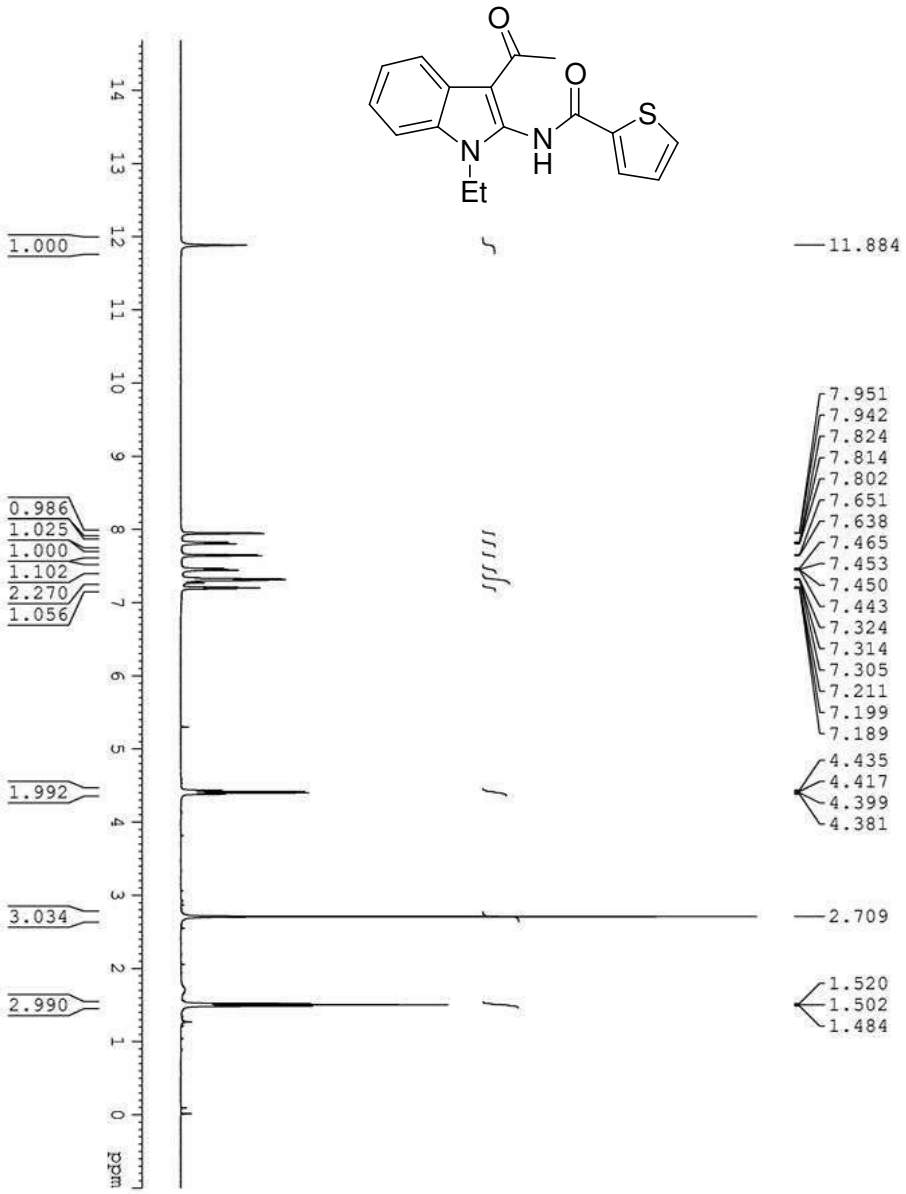
^{1}H NMR of *N*-(3-acetyl-1-ethyl-1*H*-indol-2-yl)thiophene-2-carboxamide (1j):



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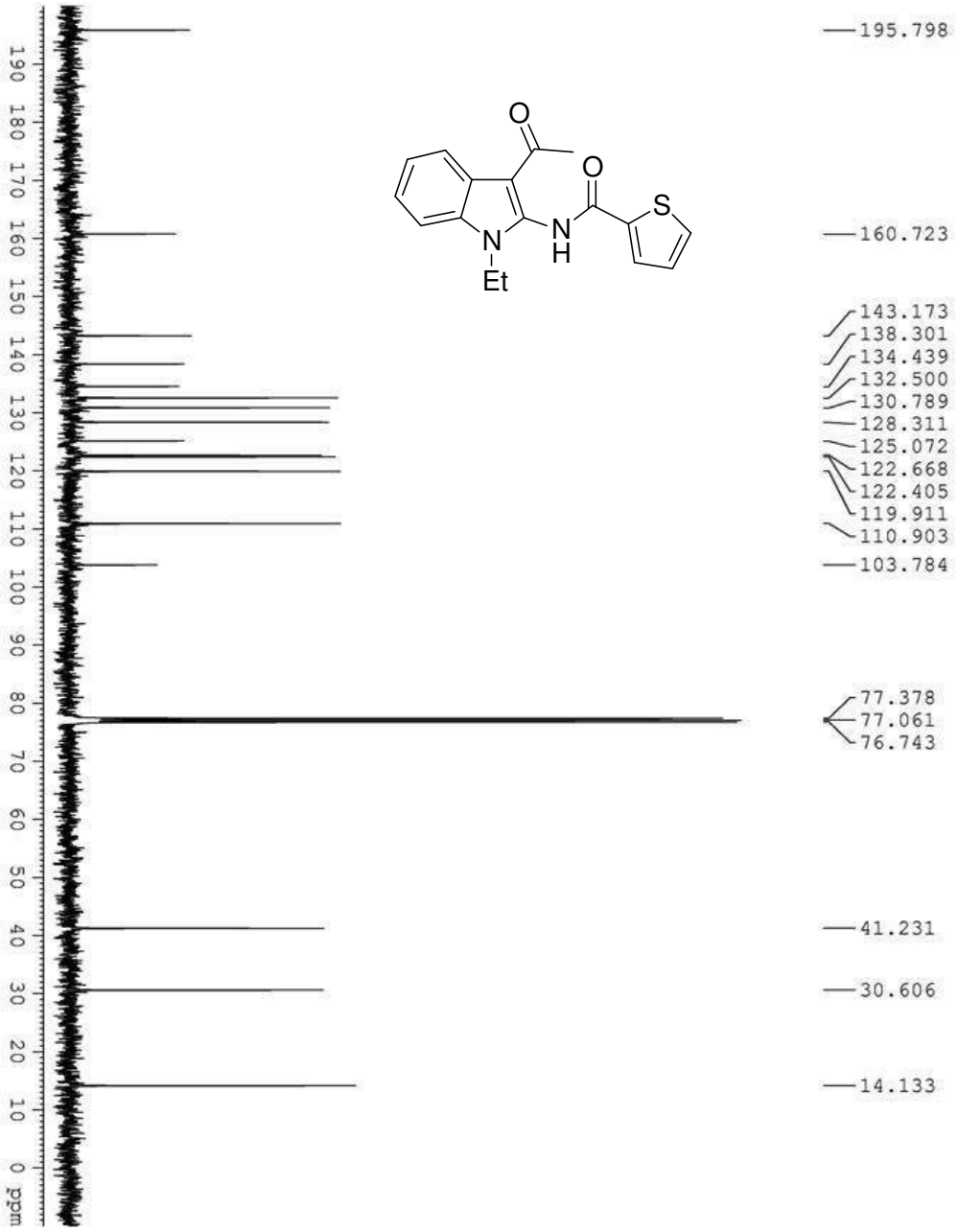
^{13}C NMR of *N*-(3-acetyl-1-ethyl-1*H*-indol-2-yl)thiophene-2-carboxamide (1j):



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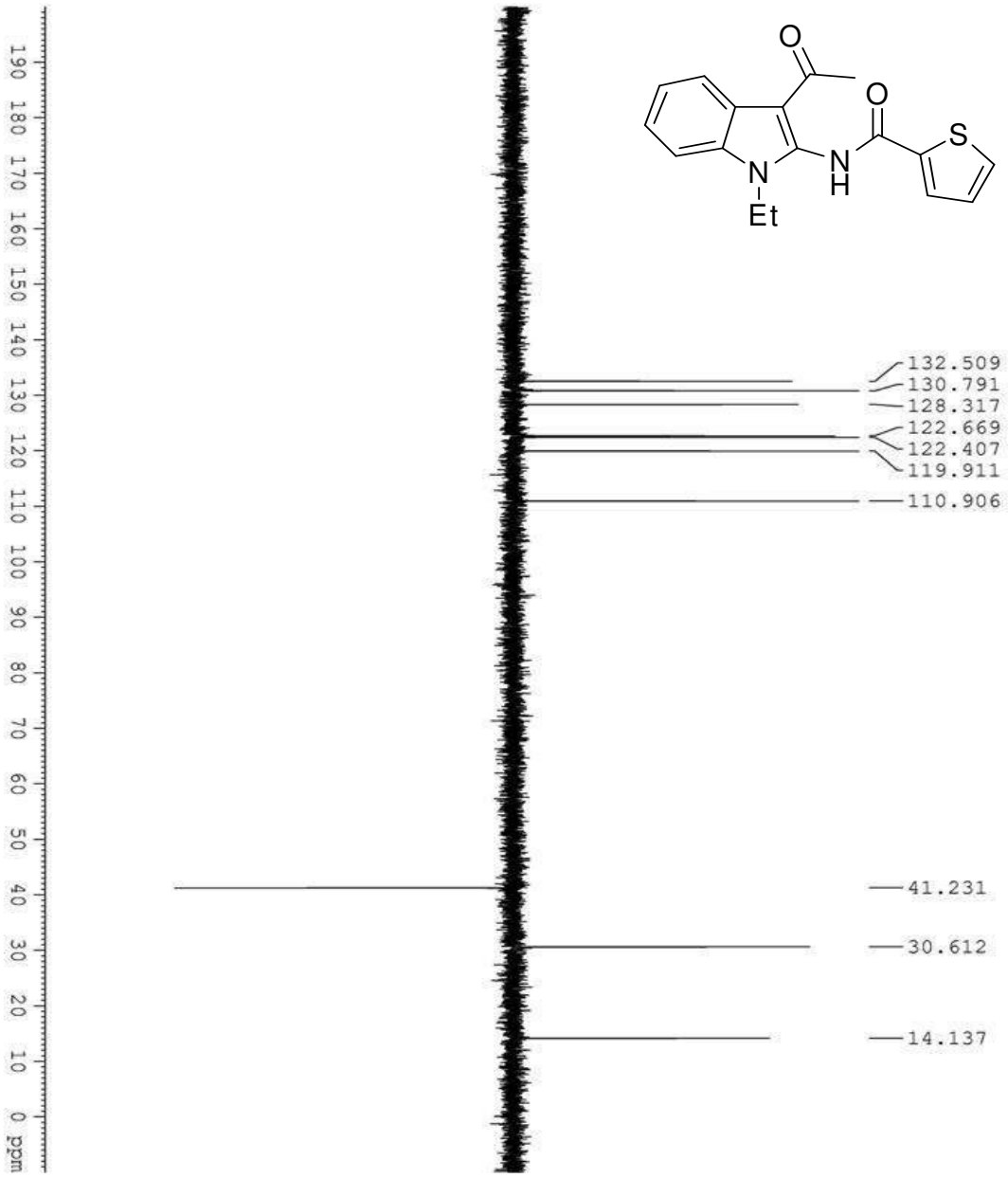
DEPT of *N*-(3-acetyl-1-ethyl-1*H*-indol-2-yl)thiophene-2-carboxamide (1j):



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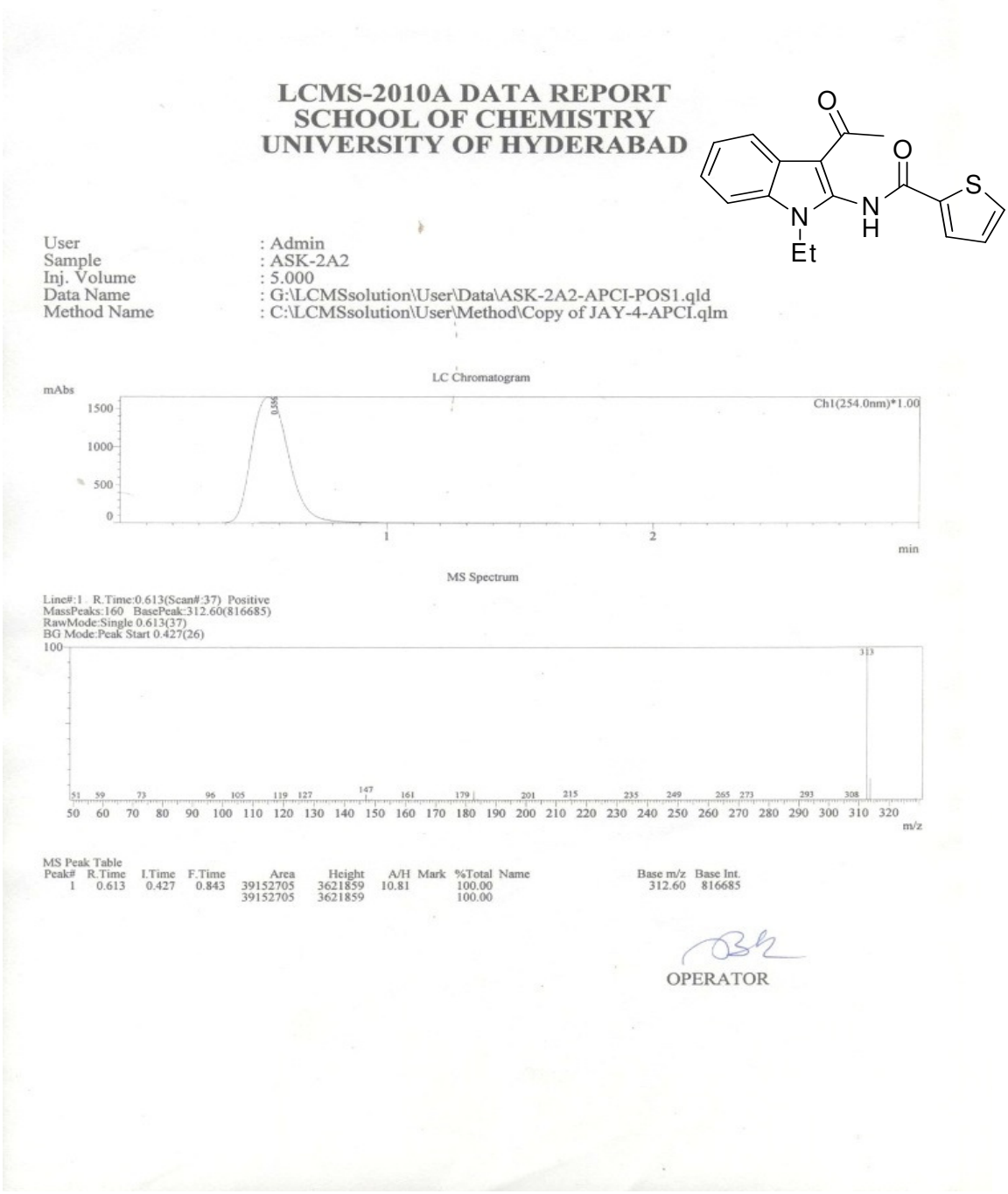
LCMS of *N*-(3-acetyl-1-ethyl-1*H*-indol-2-yl)thiophene-2-carboxamide (1j):



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CHN Analysis of *N*-(3-acetyl-1-ethyl-1*H*-indol-2-yl)thiophene-2-carboxamide (1j):

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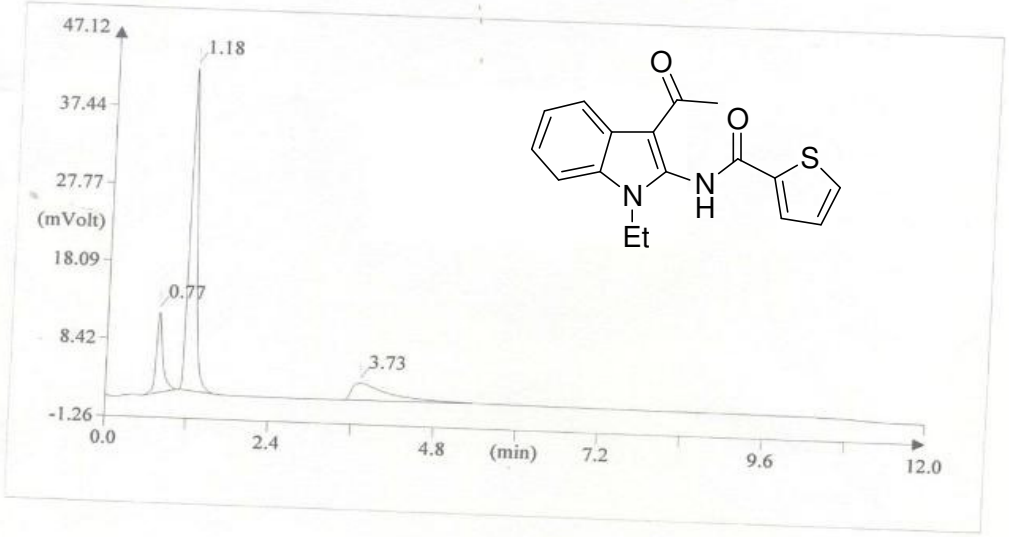
Method filename: E:\Program Files\Thermo Finnigan\Eager 300 for EA1112\DATA\Sys_data_ex
Sample ID: ASK-2A2 (# 157)
Analysis type: UnkNown
Chromatogram filename: UNK-30012012-27.dat
Sample weight: 1.138



Element Name	Element %	Ret. Time
Nitrogen	8. 91	0. 77
Carbon	65. 26	1. 18
Hydrogen	5. 24	3. 73

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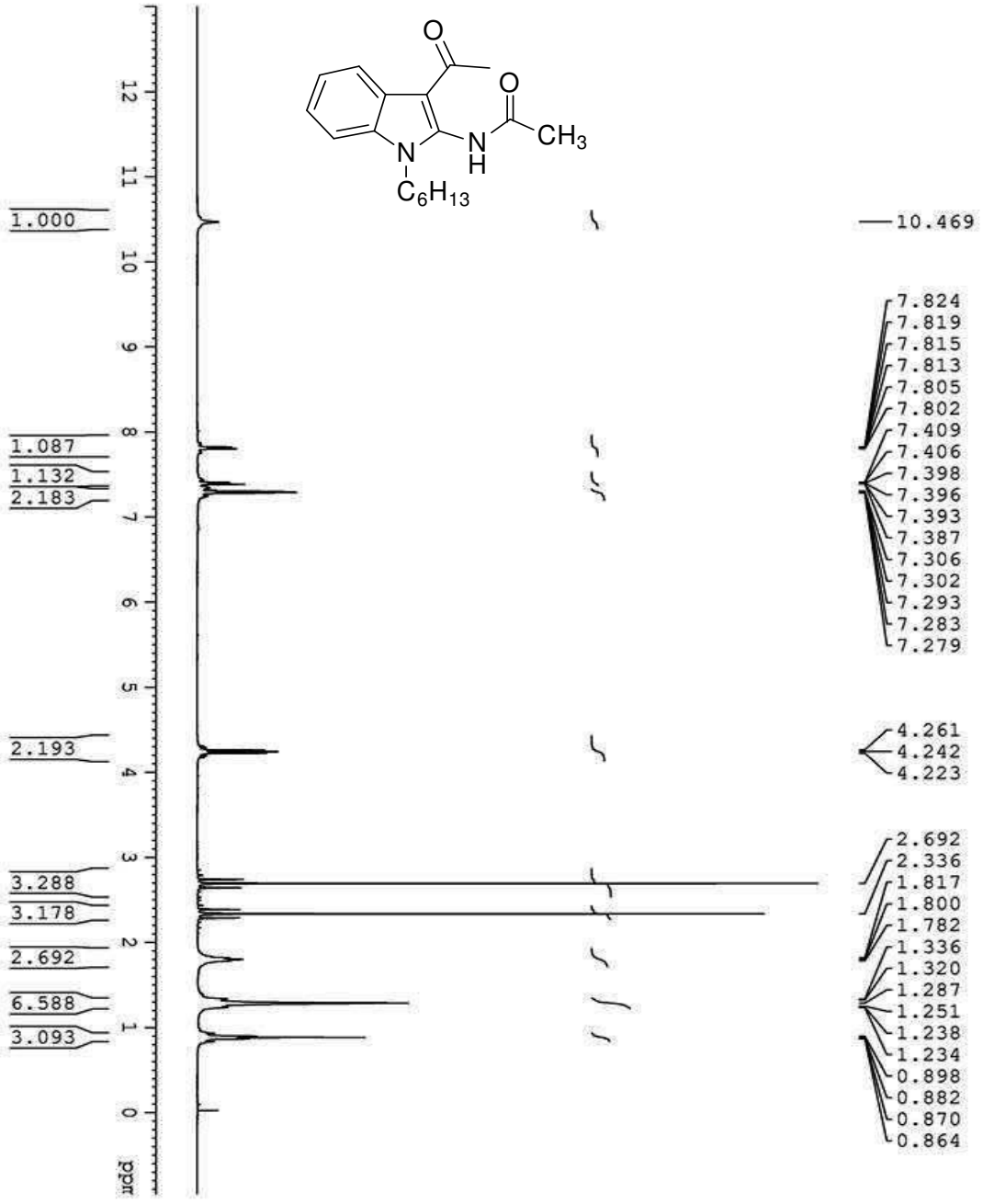
^{1}H NMR of *N*-(3-acetyl-1-hexyl-1*H*-indol-2-yl)acetamide (1k):



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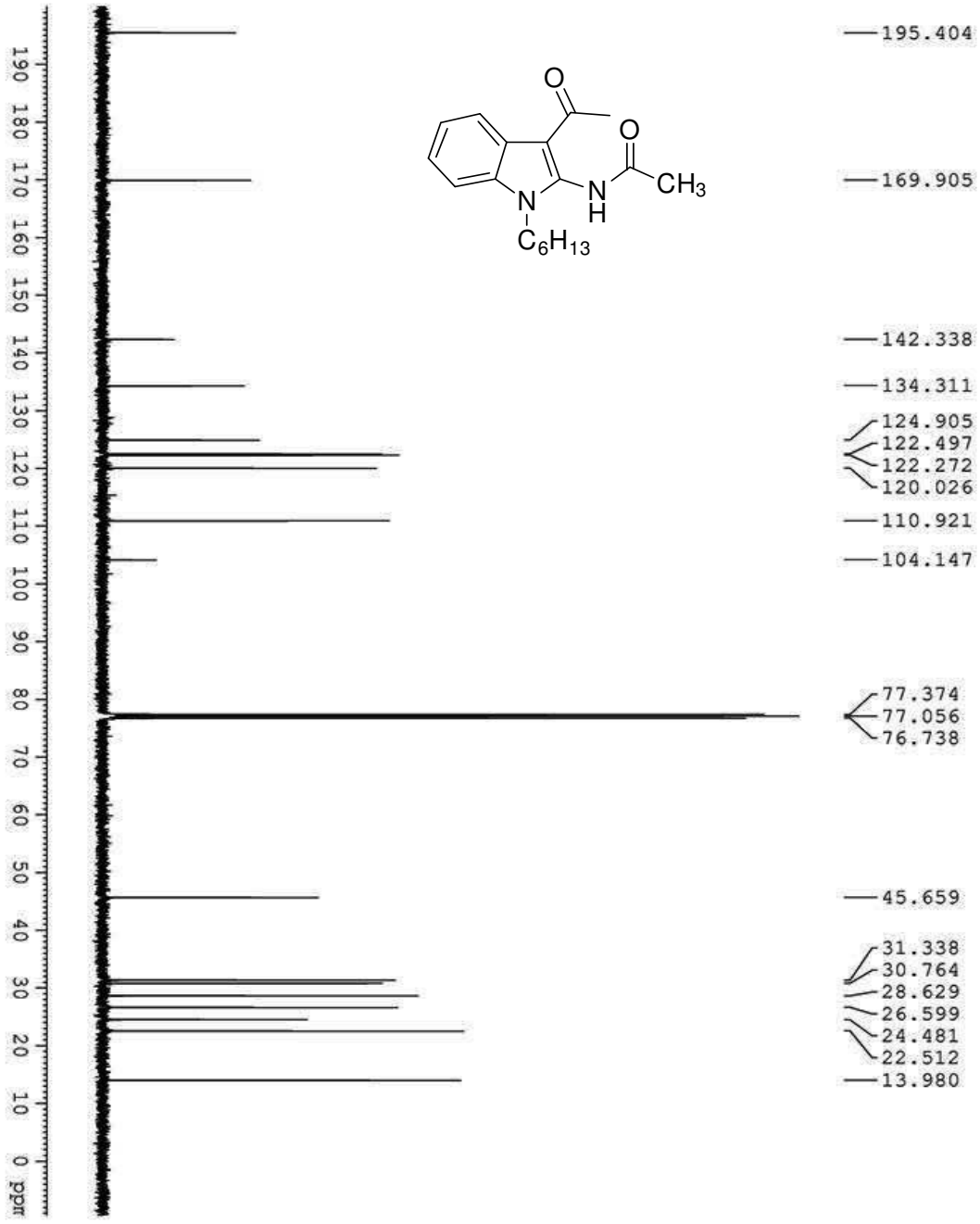
^{13}C NMR of *N*-(3-acetyl-1-hexyl-1*H*-indol-2-yl)acetamide (1k):



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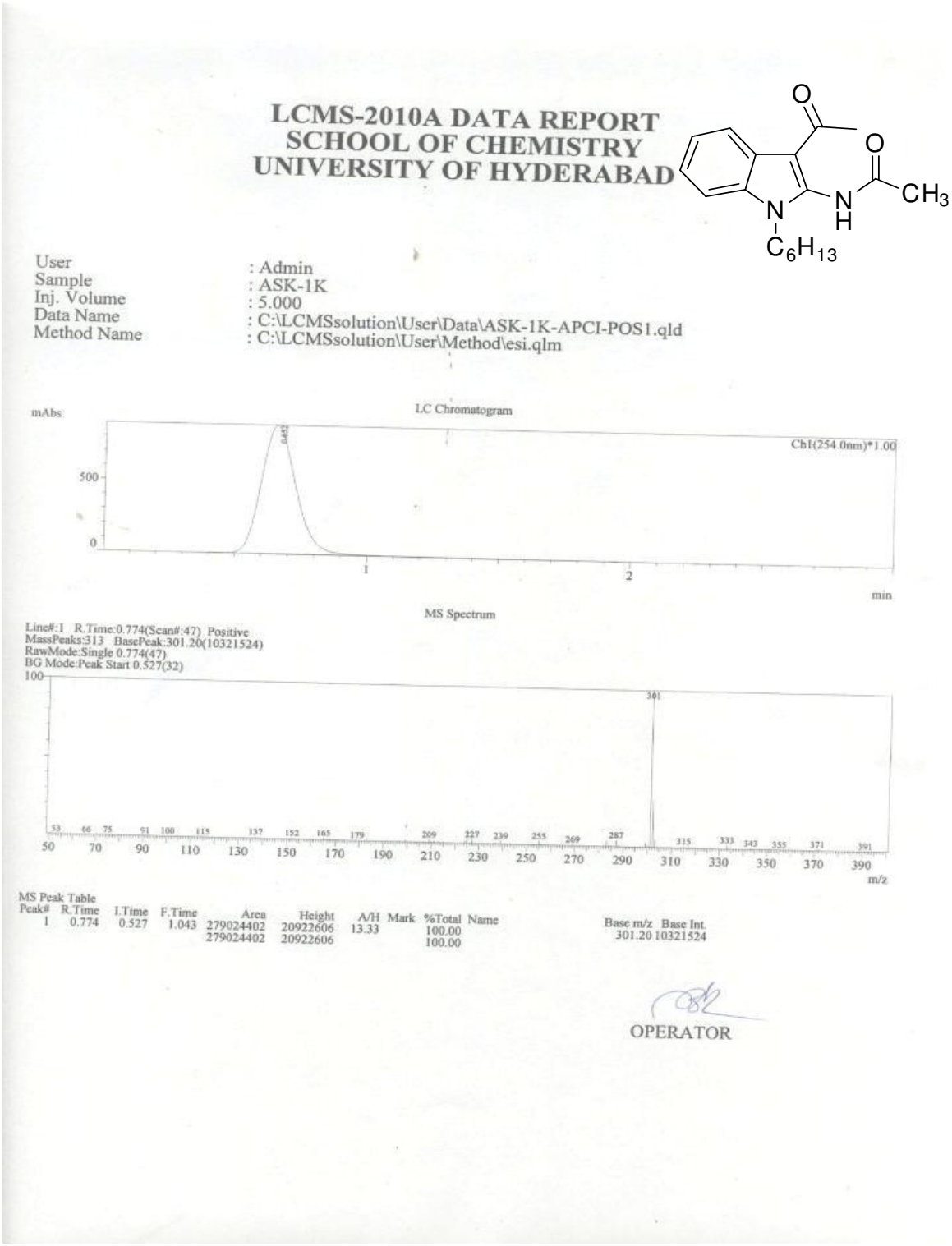
LCMS of *N*-(3-acetyl-1-hexyl-1*H*-indol-2-yl)acetamide (1k):



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CHN Analysis of *N*-(3-acetyl-1-hexyl-1*H*-indol-2-yl)acetamide (1k):

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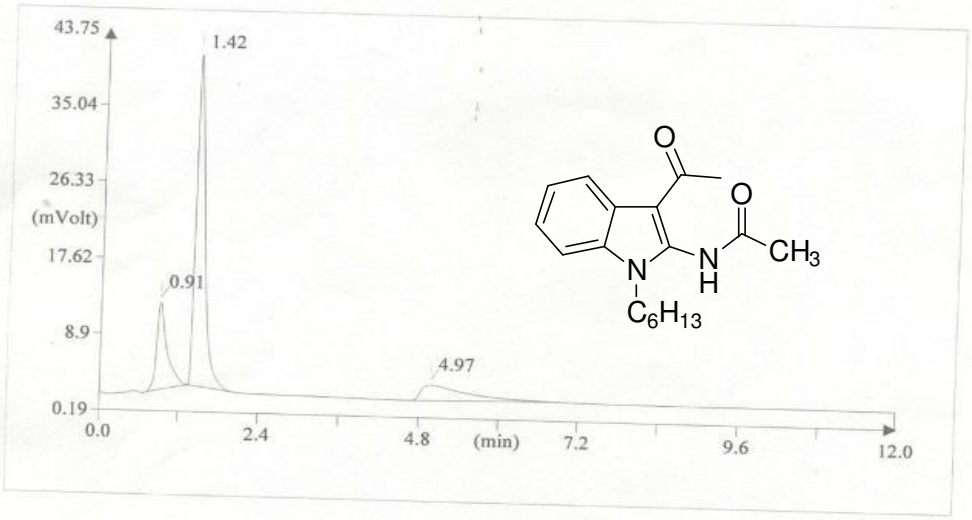
Method filename:	E:\Program Files\Thermo Finnigan\Eager 300 for EA1112\DATA\Sys_data_ex
Sample ID:	ASK-1K (# 107)
Analysis type:	UnkNown
Chromatogram filename:	UNK-24012012-7.dat
Sample weight:	.975



Element Name	Element %	Ret. Time
Nitrogen	9. 45	0. 91
Carbon	71. 85	1. 42
Hydrogen	8. 12	4. 97

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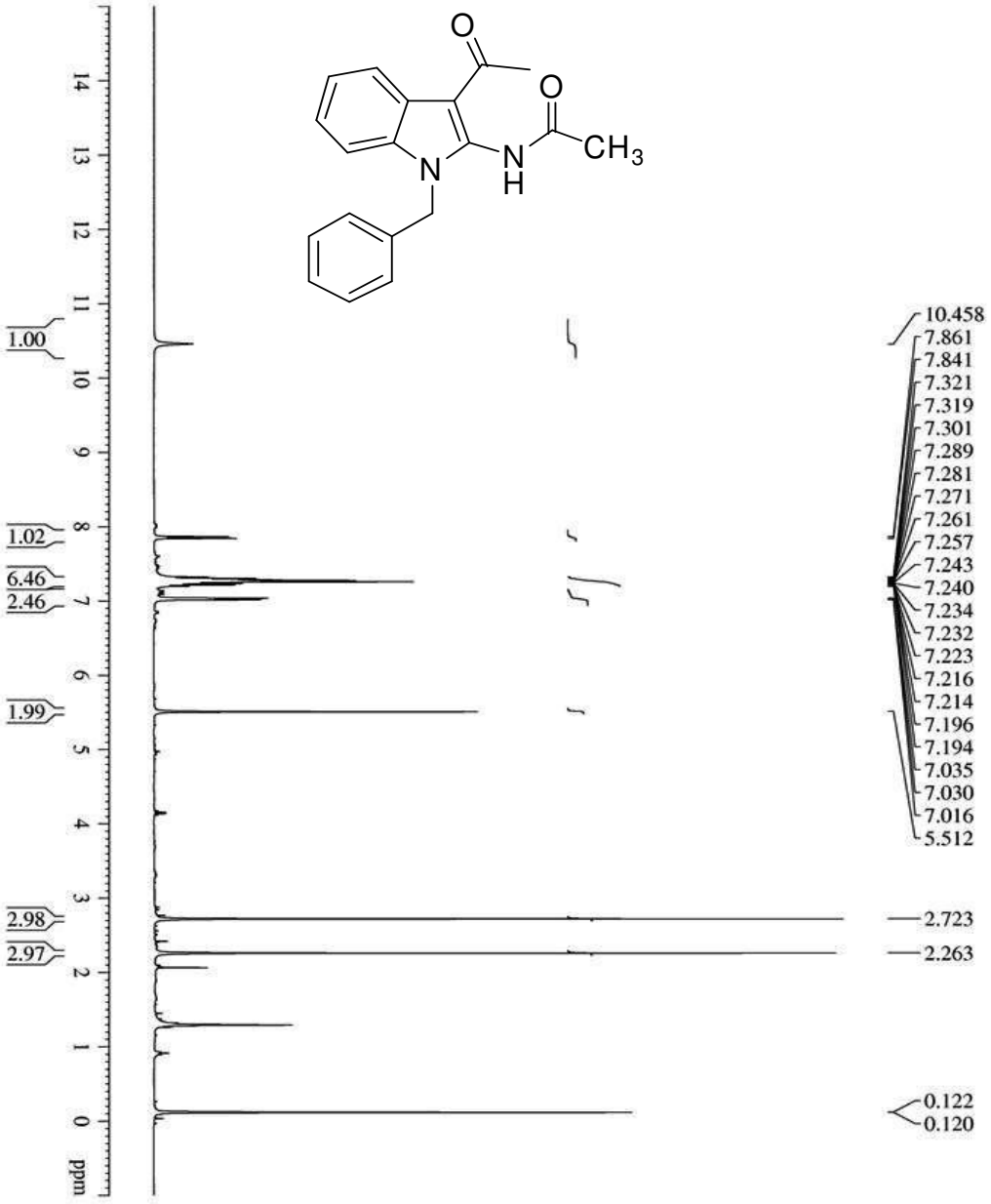
^{1}H NMR of *N*-(3-acetyl-1-benzyl-1*H*-indol-2-yl)acetamide (1l):



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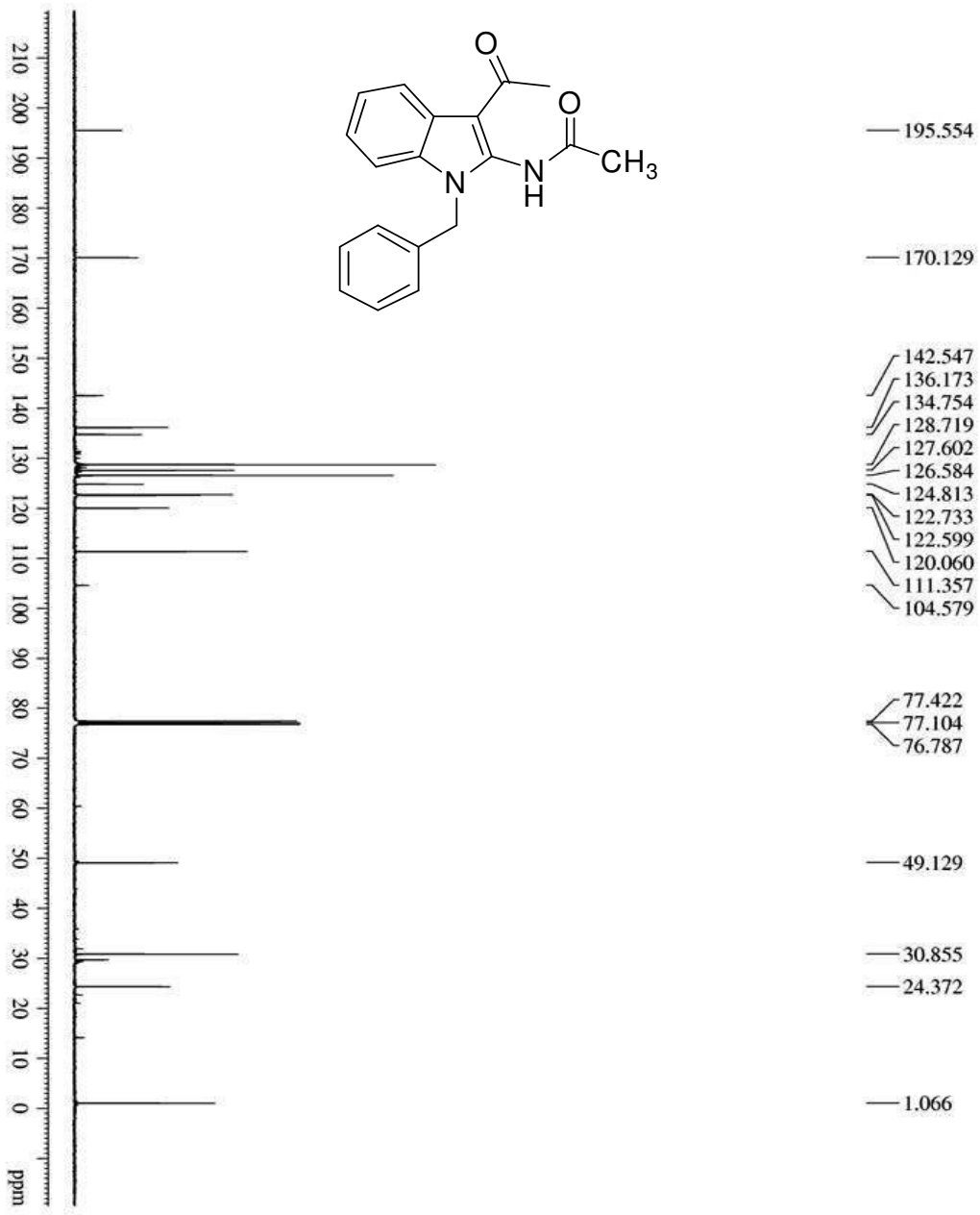
^{13}C NMR of *N*-(3-acetyl-1-benzyl-1*H*-indol-2-yl)acetamide (1l):



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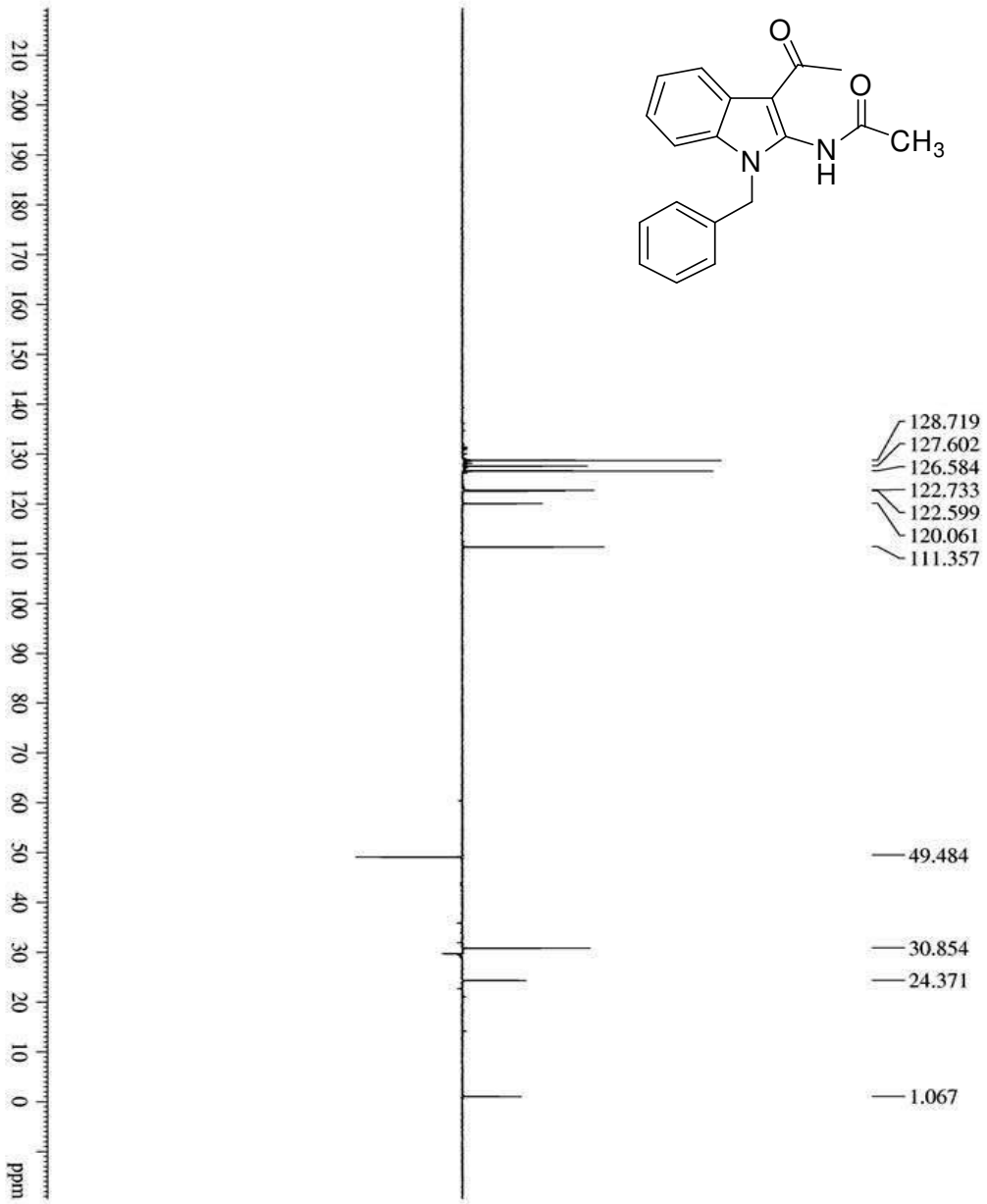
DEPT of *N*-(3-acetyl-1-benzyl-1*H*-indol-2-yl)acetamide (1l):



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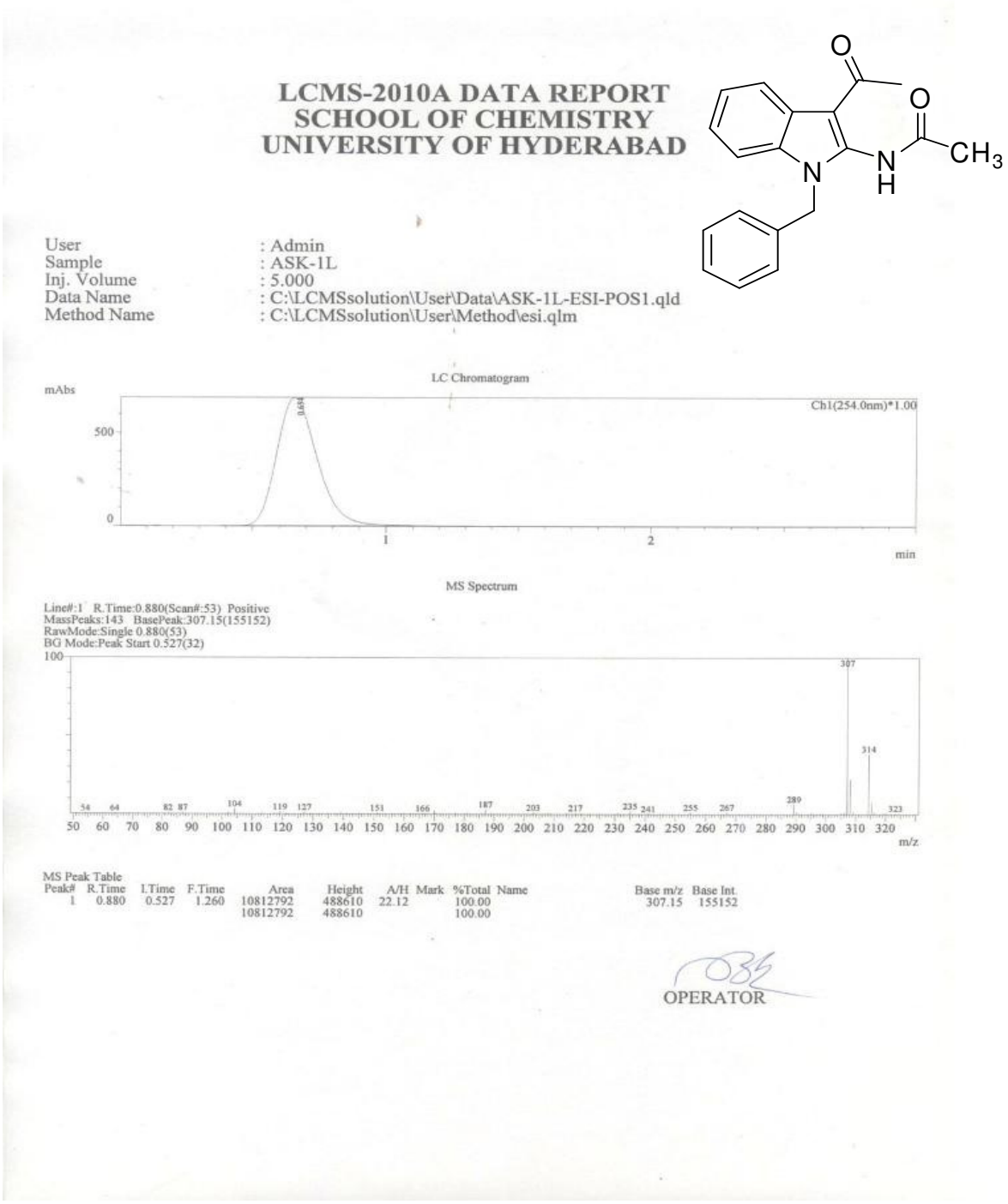
LCMS of *N*-(3-acetyl-1-benzyl-1*H*-indol-2-yl)acetamide (1l):



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CHN Analysis of *N*-(3-acetyl-1-benzyl-1*H*-indol-2-yl)acetamide (1l):

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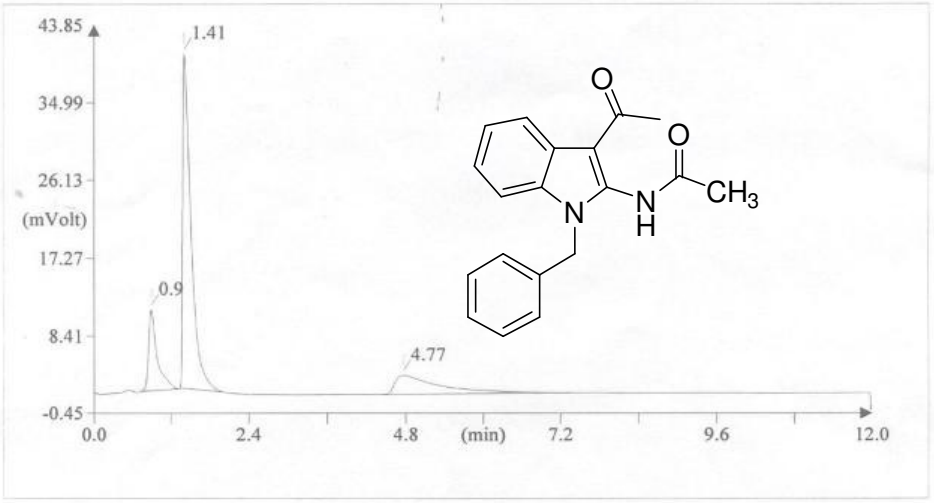
Method filename:	E:\Program Files\Thermo Finnigan\Eager 300 for EA1112\DATA\Sys_data_ex
Sample ID:	ASK-1L (# 109)
Analysis type:	UnkNown
Chromatogram filename:	UNK-24012012-9.dat
Sample weight:	.976



Element Name	Element %	Ret. Time
Nitrogen	9. 07	0. 90
Carbon	74. 63	1. 41
Hydrogen	5. 88	4. 77

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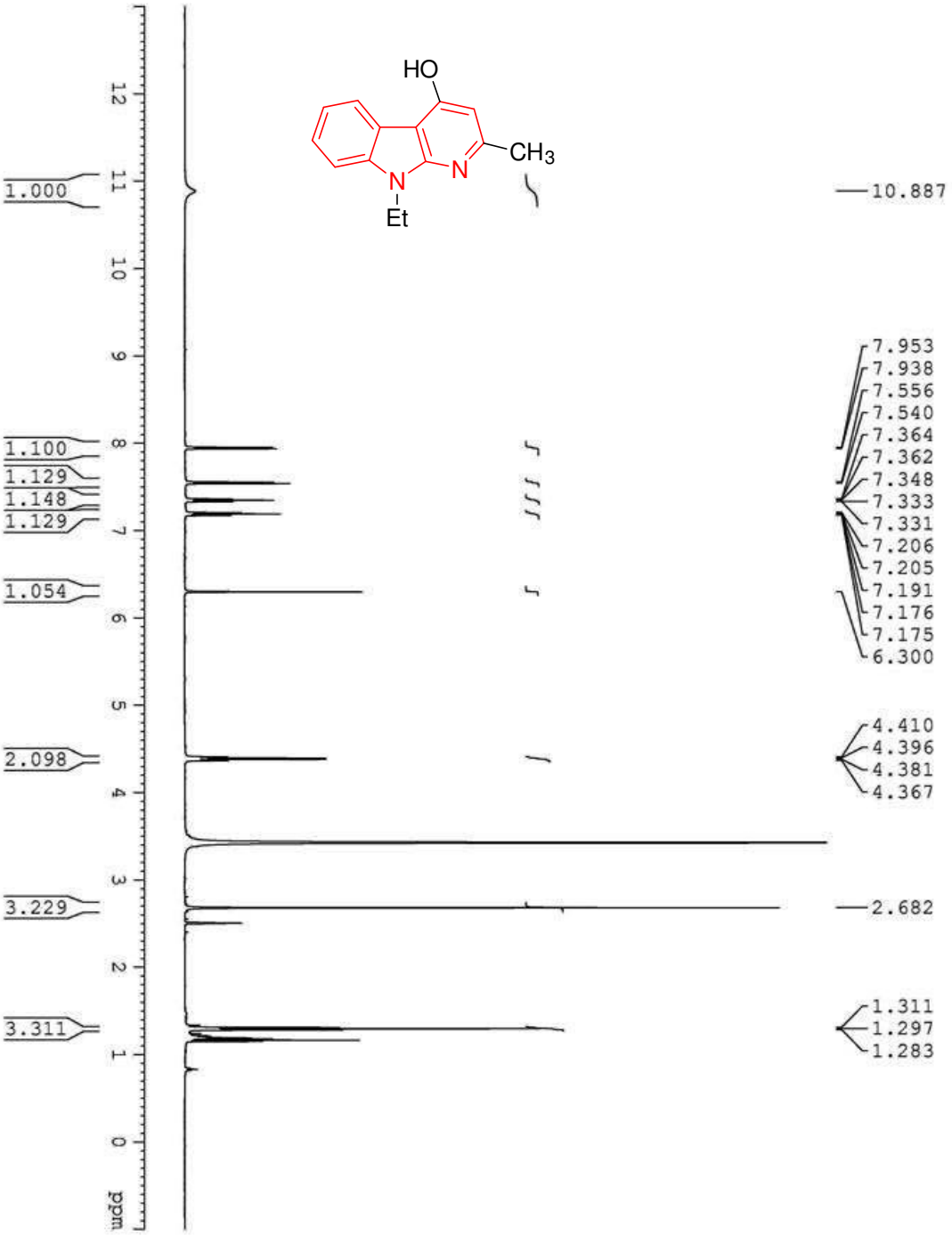
^{1}H NMR of 9-ethyl-2-methyl-9*H*-pyrido[2,3-*b*]indol-4-ol (2a):



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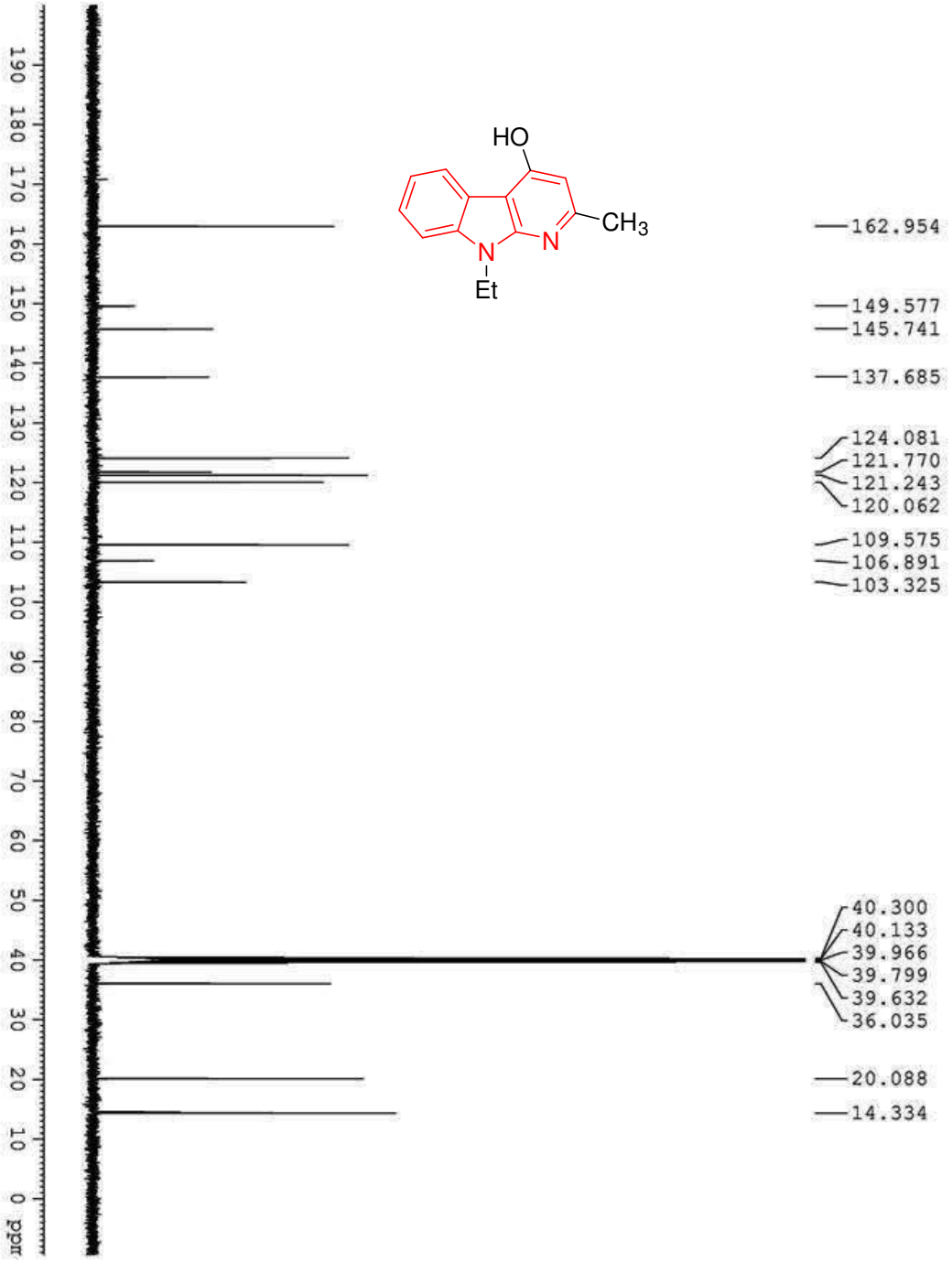
^{13}C NMR of 9-ethyl-2-methyl-9*H*-pyrido[2,3-*b*]indol-4-ol (2a)



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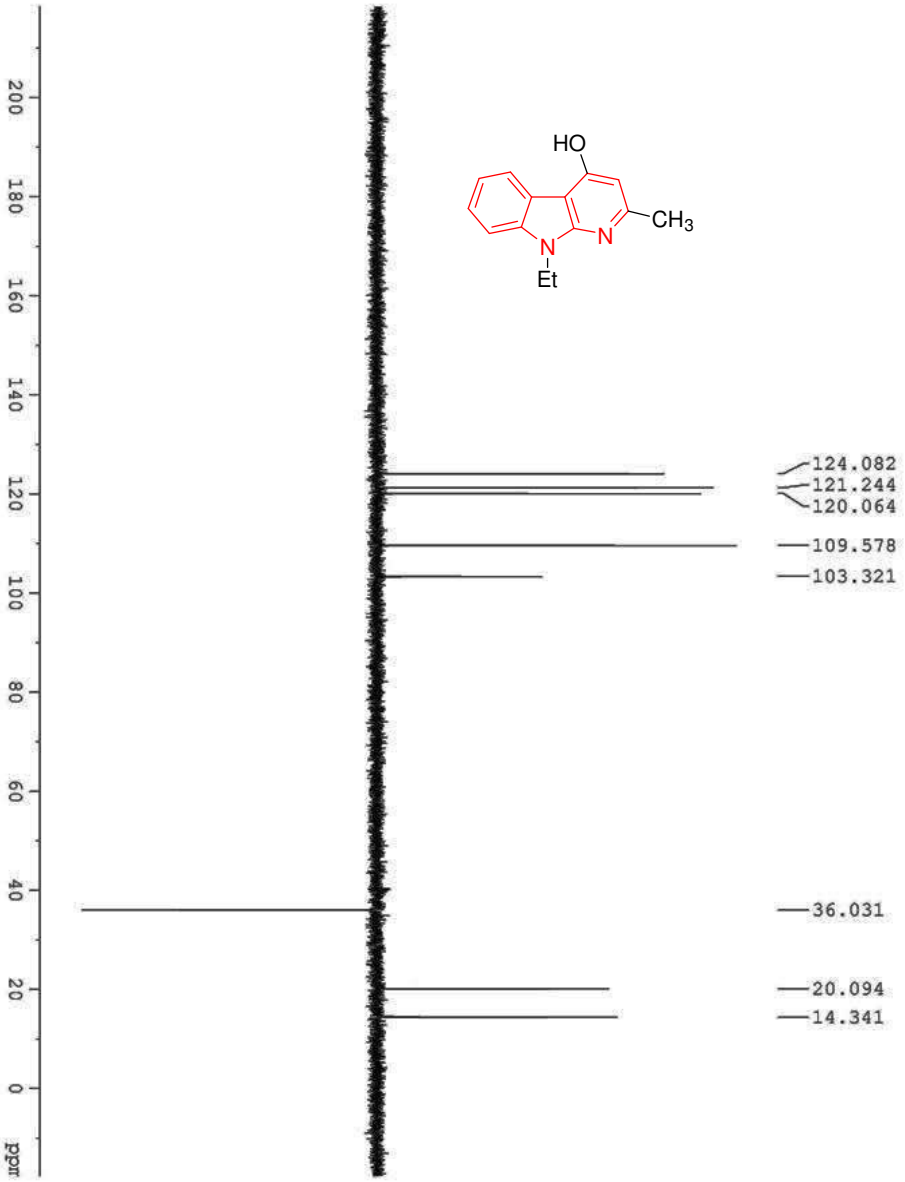
DEPT of 9-ethyl-2-methyl-9*H*-pyrido[2,3-*b*]indol-4-ol (2a)



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LCMS 9-ethyl-2-methyl-9*H*-pyrido[2,3-*b*]indol-4-ol (2a)

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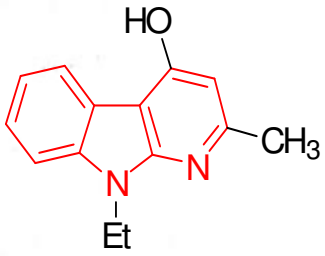
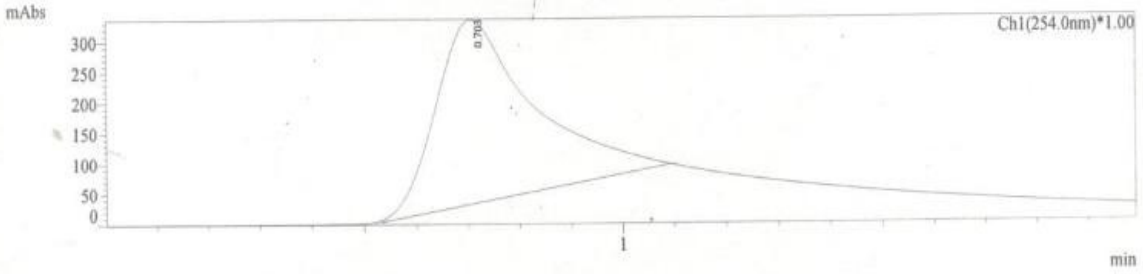


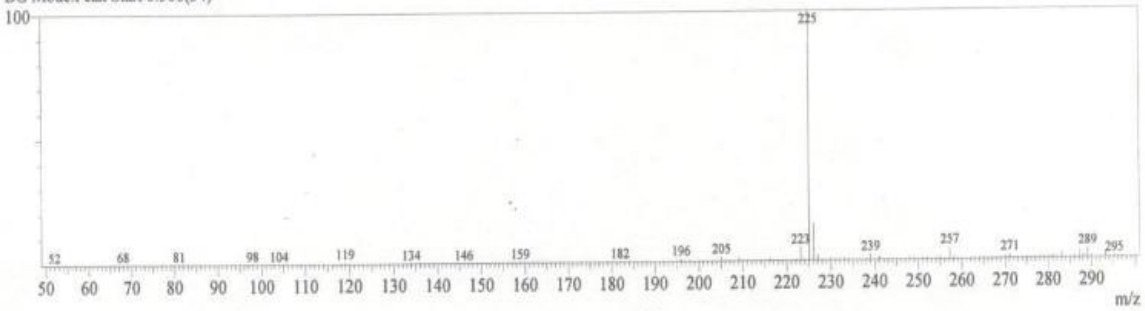
User	: Admin
Sample	: ASK-C2
Inj. Volume	: 5.000
Data Name	: C:\LCMSsolution\User\Data\ASK-C2-APCI-NEG1.qld
Method Name	: C:\LCMSsolution\User\Method\esi.qlm

LC Chromatogram



MS Spectrum

Line#:1 R.Time:0.802(Scan#:48) Negative
MassPeaks:159 BasePeak:225.10(245679)
RawMode:Single 0.802(48)
BG Mode:Peak Start 0.560(34)



MS Peak Table

Peak#	R.Time	I.Time	F.Time	Area	Height	A/H	Mark	%Total	Name	Base m/z	Base Int.
1	0.802	0.560	1.143	34385003	2284097	15.05		100.00		225.10	245679
				34385003	2284097			100.00			

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CHN Analysis of 9-ethyl-2-methyl-9*H*-pyrido[2,3-*b*]indol-4-ol (2a)

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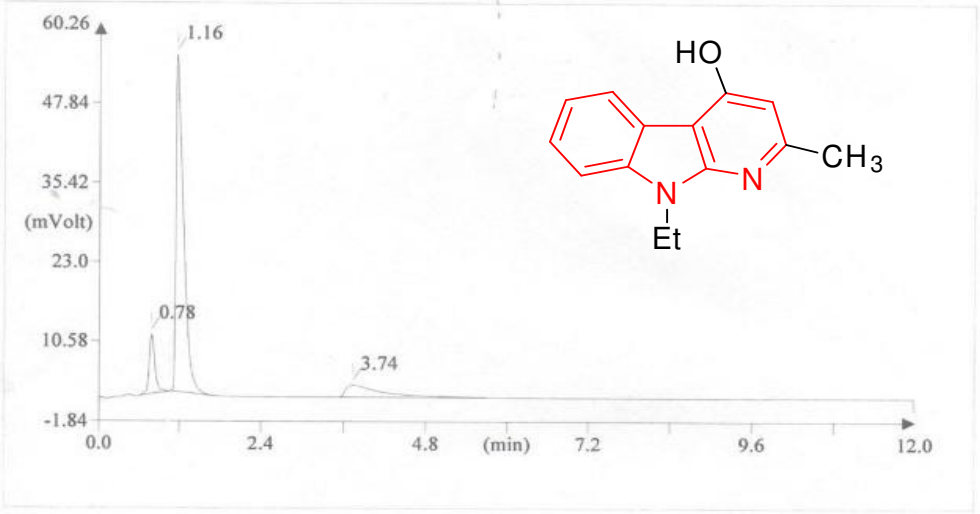
Method filename:	E:\Program Files\Thermo Finnigan\Eager 300 for EA1112\DATA\Sys_data_ex
Sample ID:	ASK-C2 (# 173)
Analysis type:	UnkNown
Chromatogram filename:	UNK-15092011-13.dat
Sample weight:	1.179



Element Name	Element %	Ret. Time
Nitrogen	12. 28	0. 78
Carbon	74. 42	1. 16
Hydrogen	6. 21	3. 74

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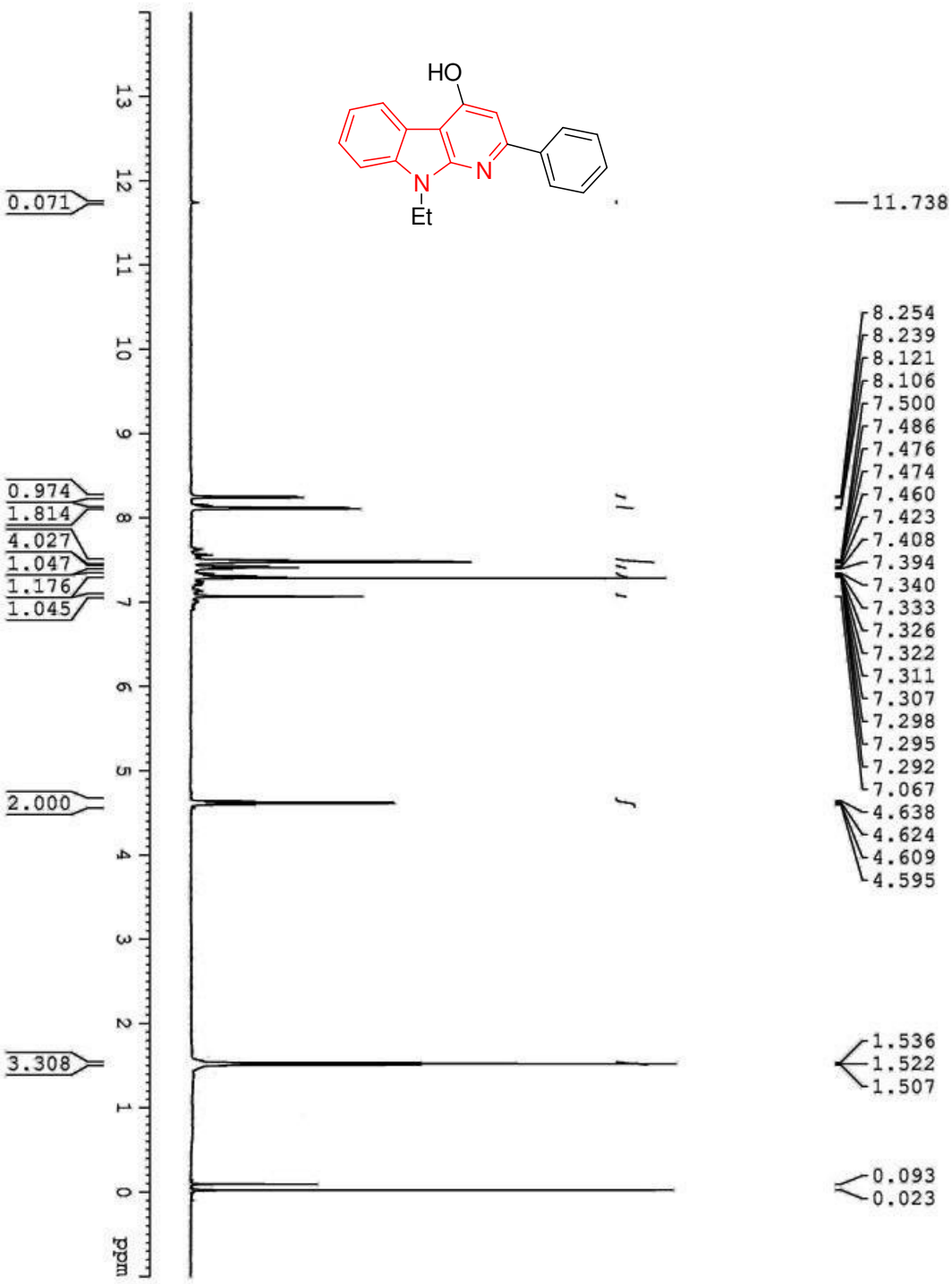
^{1}H NMR of 9-ethyl-2-phenyl-9*H*-pyrido[2,3-*b*]indol-4-ol (2b)



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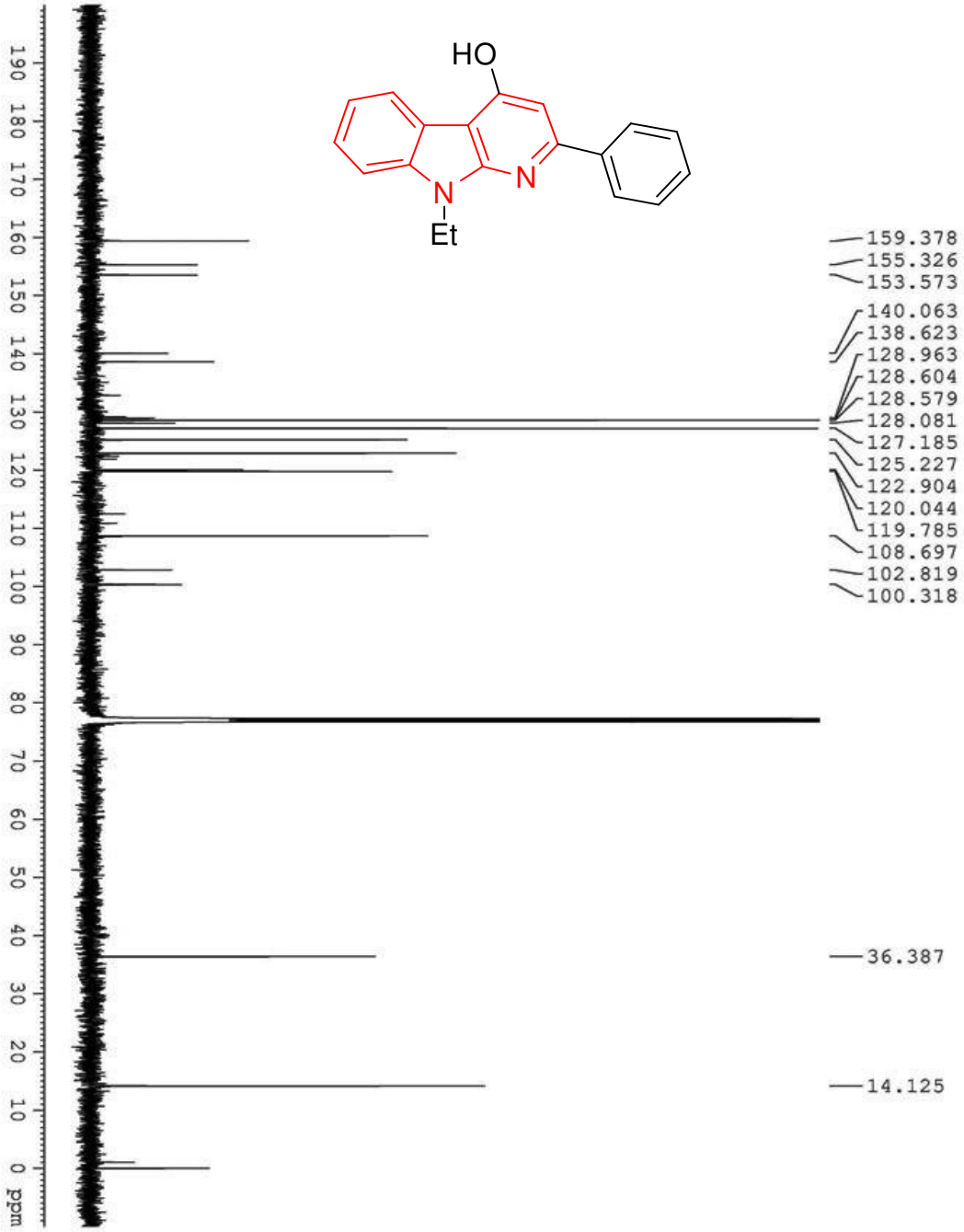
^{13}C NMR of 9-ethyl-2-phenyl-9*H*-pyrido[2,3-*b*]indol-4-ol (2b)



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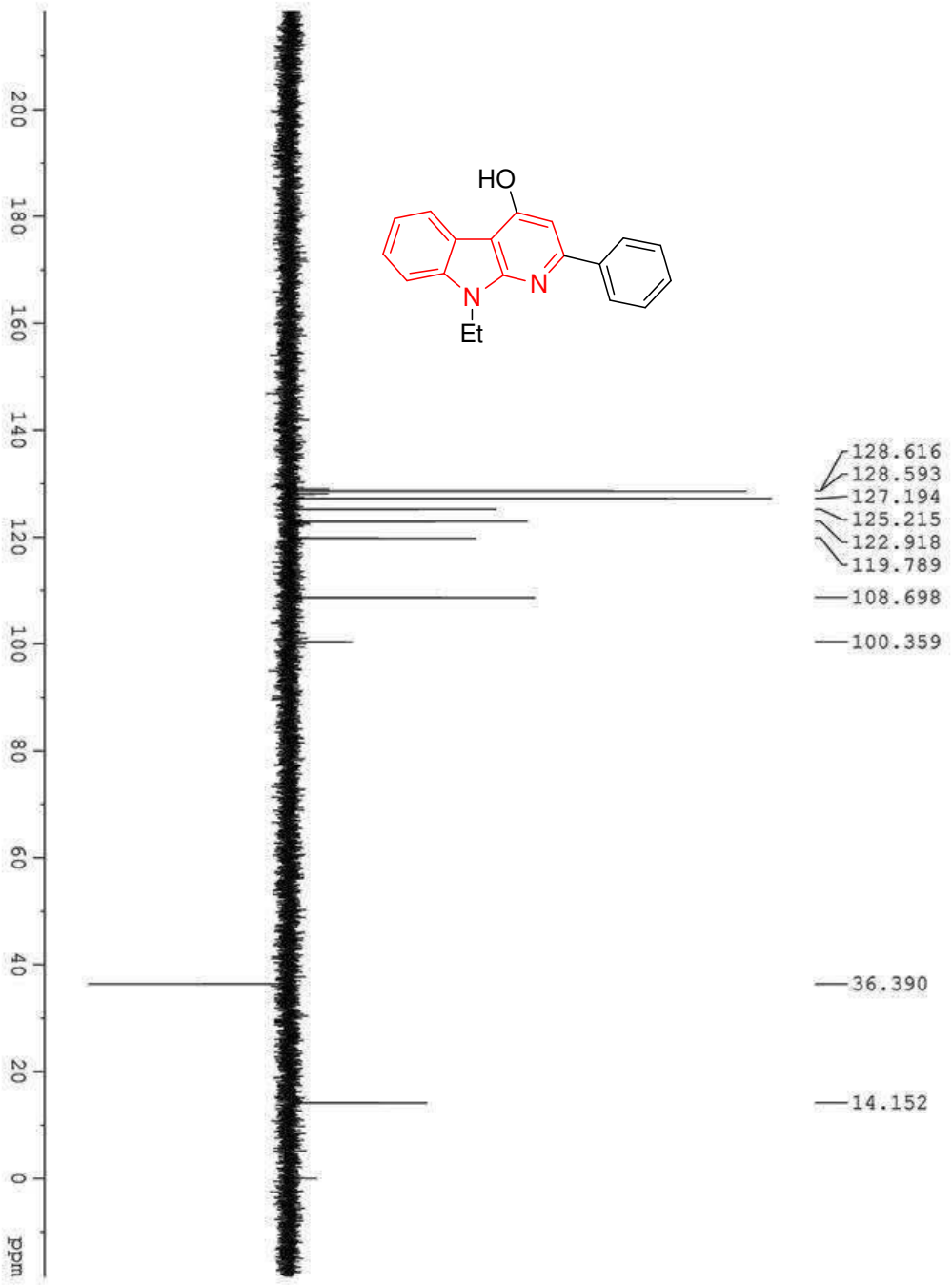
DEPT of 9-ethyl-2-phenyl-9*H*-pyrido[2,3-*b*]indol-4-ol (2b)



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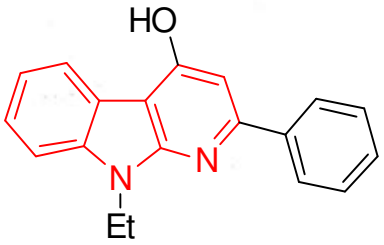
LCMS of 9-ethyl-2-phenyl-9*H*-pyrido[2,3-*b*]indol-4-ol (2b)



LCMS-2010A DATA REPORT
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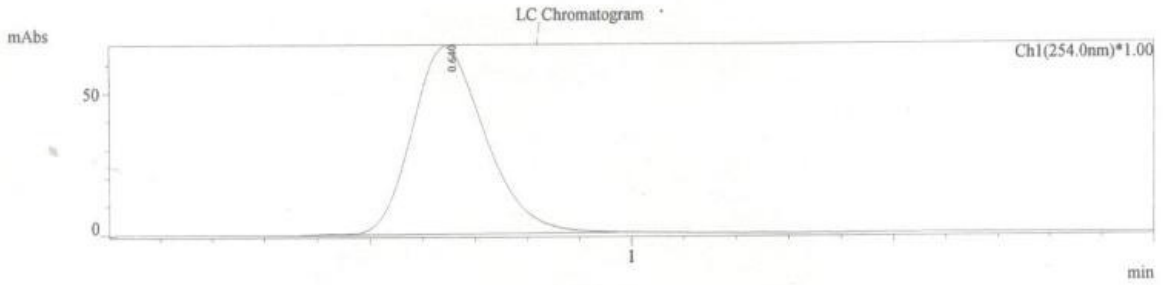
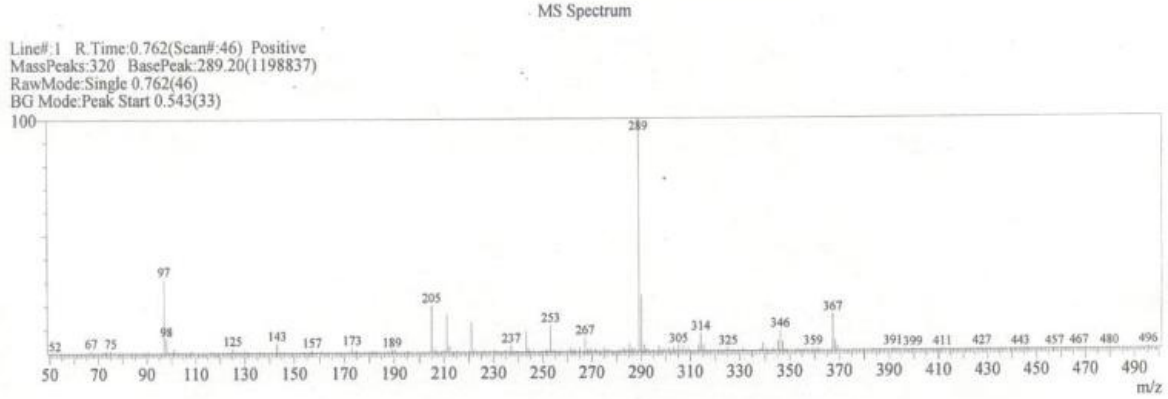
User : Admin
Sample : ASK-C3
Inj. Volume : 1.000
Data Name : C:\LCMSsolution\User\Data\ASK-C3-APCI-POS1.qld
Method Name : C:\LCMSsolution\User\Method\esi.qlm





MS Peak Table

Peak#	R.Time	I.Time	F.Time	Area	Height	A/H	Mark	%Total	Name	Base m/z	Base Int.
1	0.762	0.543	1.043	63878670	4220354	15.13		100.00		289.20	1198837
				63878670	4220354			100.00			

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CHN Analysis of 9-ethyl-2-phenyl-9*H*-pyrido[2,3-*b*]indol-4-ol (2b)

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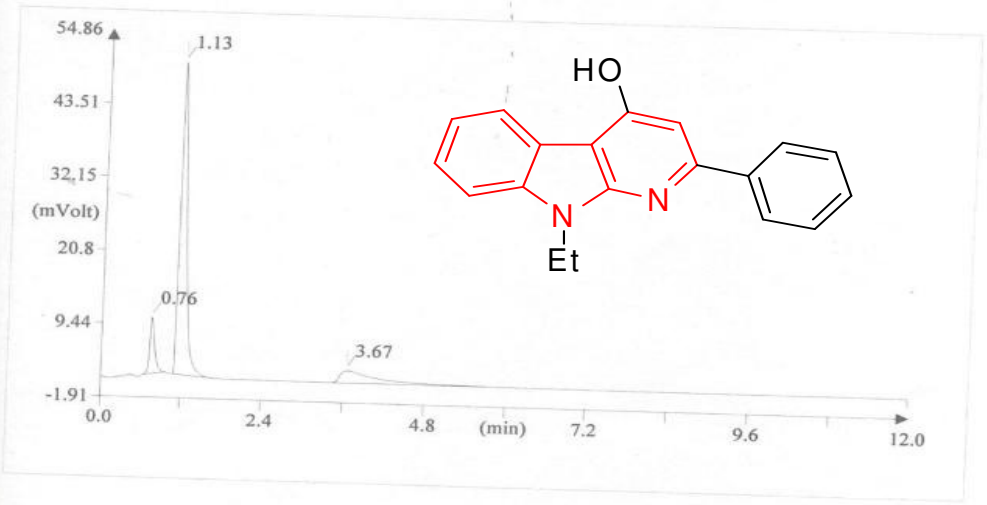
Method filename:	E:\Program Files\Thermo Finnigan\Eager 300 for EA1112\DATA\Sys_data_ex
Sample ID:	ASK-C3 (# 182)
Analysis type:	UnkNown
Chromatogram filename:	UNK-15092011-22.dat
Sample weight:	1.116



Element Name	Element %	Ret. Time
Nitrogen	9. 65	0. 76
Carbon	79. 24	1. 13
Hydrogen	5. 51	3. 67

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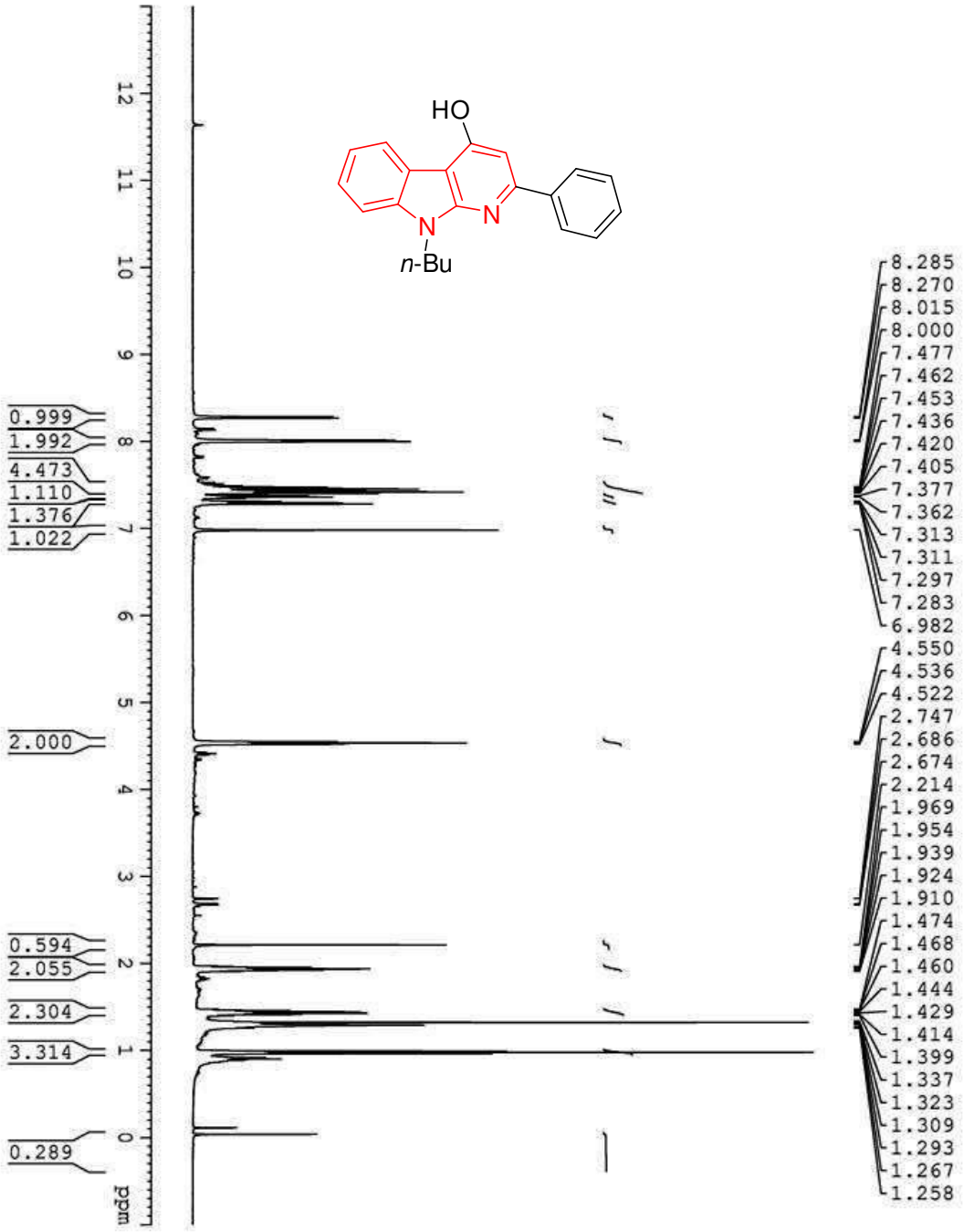
^{1}H NMR of 9-butyl-2-phenyl-9*H*-pyrido[2,3-*b*]indol-4-ol (2c)



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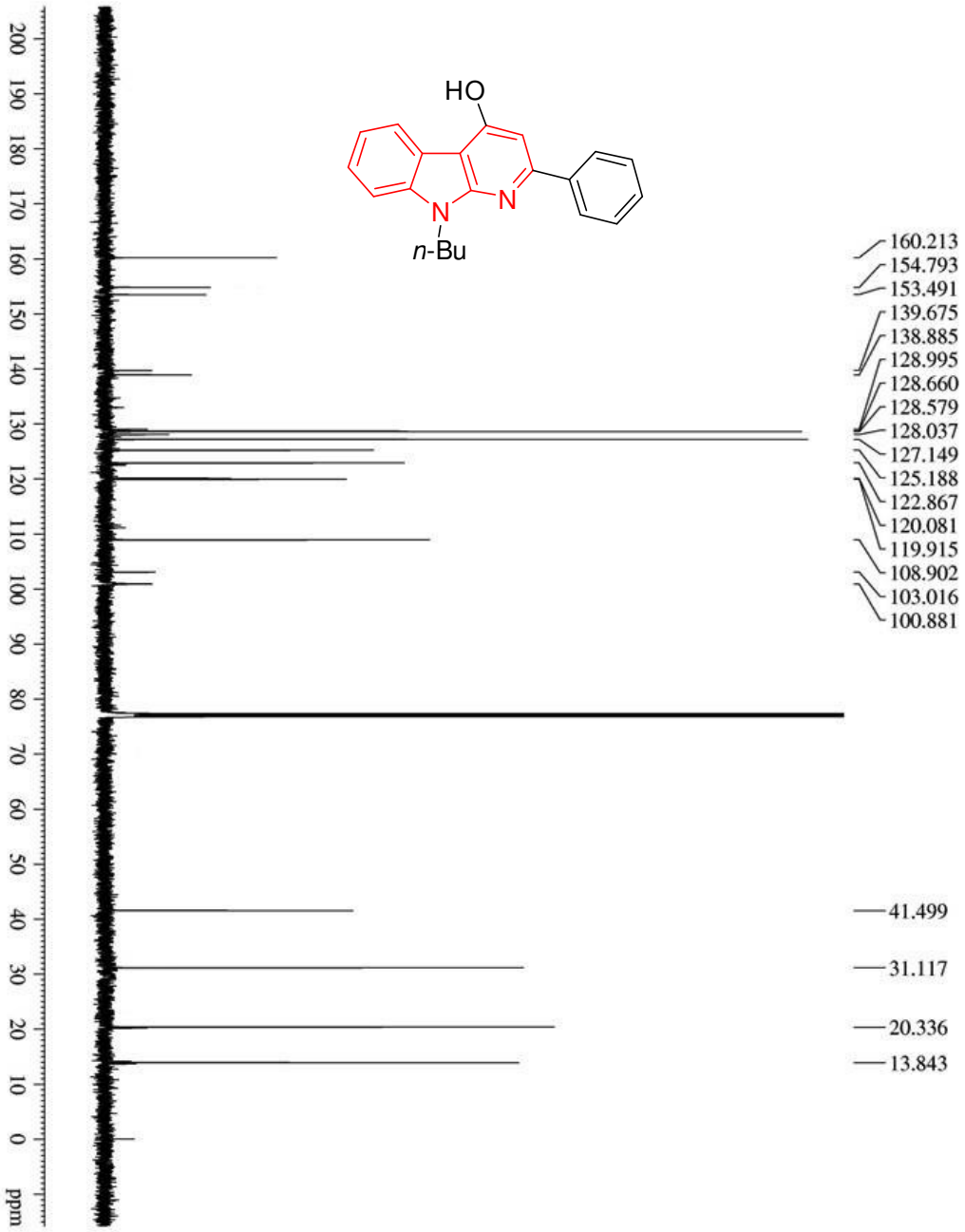
^{13}C NMR of 9-butyl-2-phenyl-9*H*-pyrido[2,3-*b*]indol-4-ol (2c)



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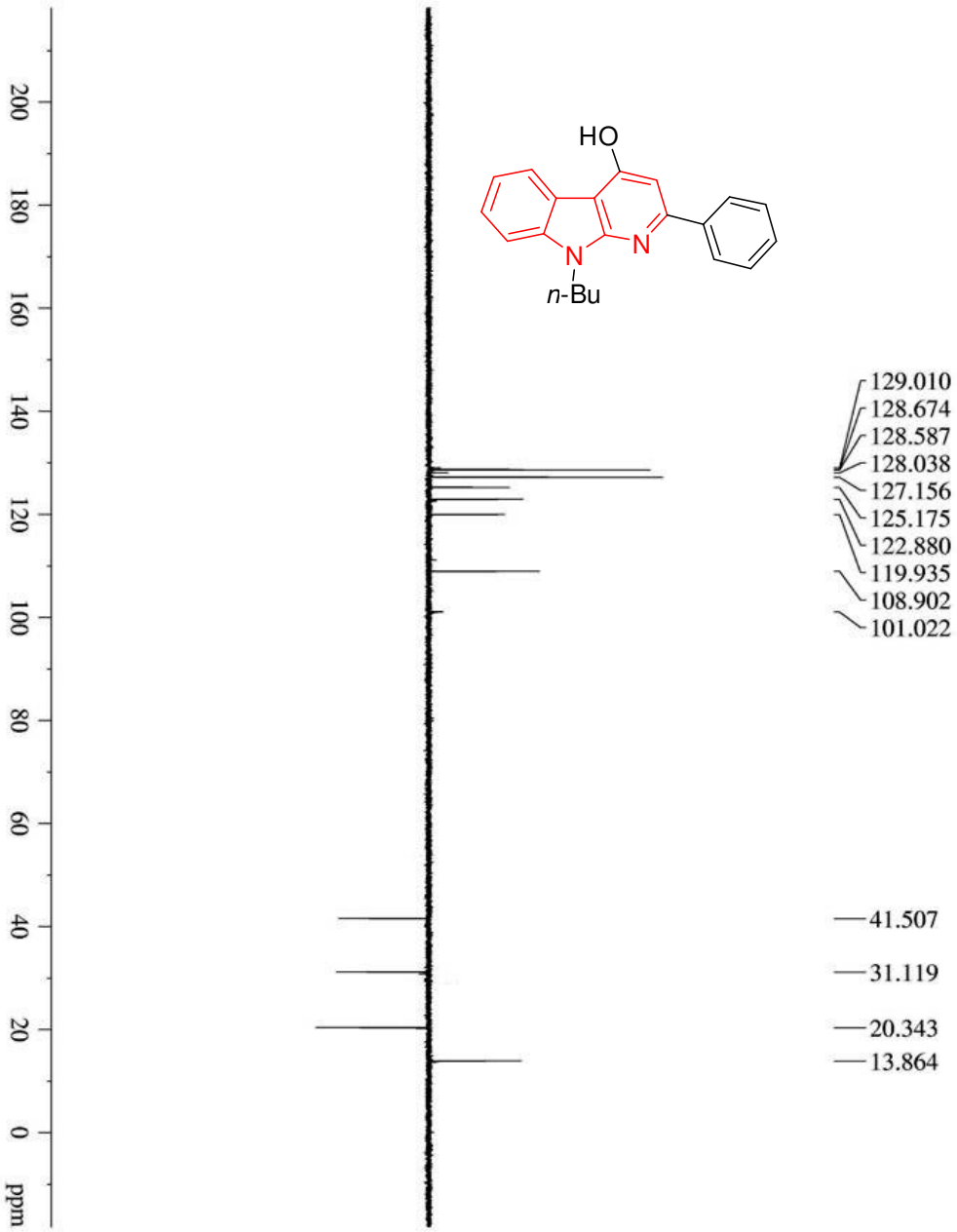
DEPT of 9-butyl-2-phenyl-9*H*-pyrido[2,3-*b*]indol-4-ol (2c)



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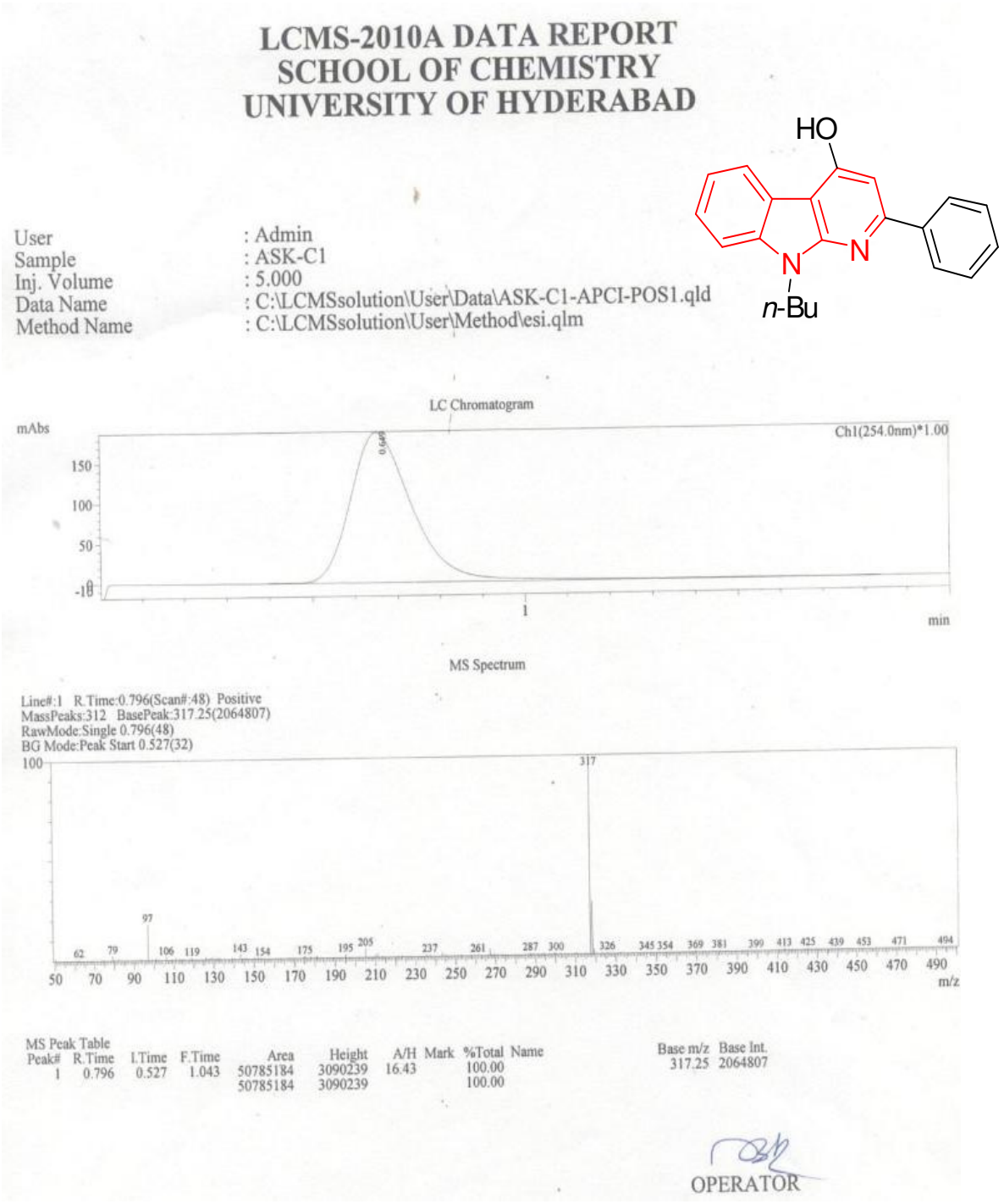
LCMS of 9-butyl-2-phenyl-9*H*-pyrido[2,3-*b*]indol-4-ol (2c)



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CHN Analysis of 9-butyl-2-phenyl-9*H*-pyrido[2,3-*b*]indol-4-ol (2c)

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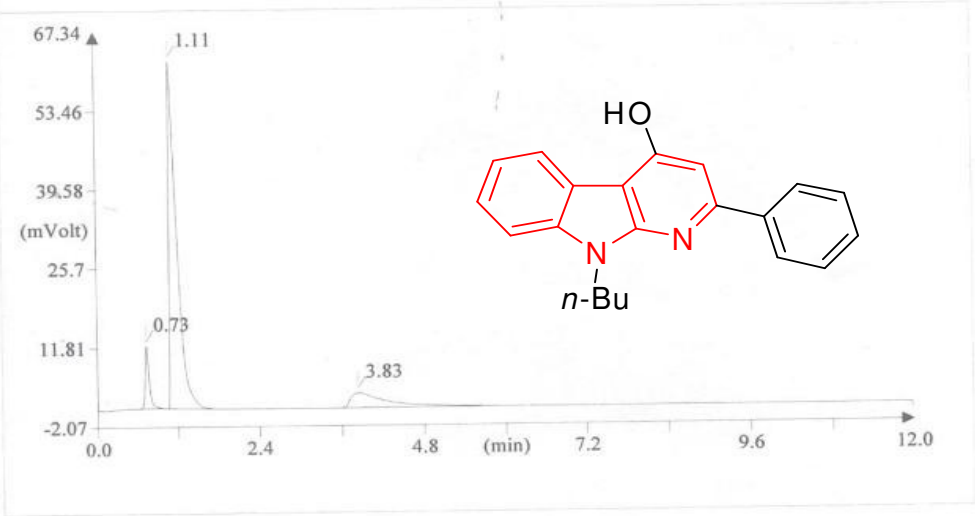
Method filename:	E:\Program Files\Thermo Finnigan\Eager 300 for EA1112\DATA\Sys_data_ex
Sample ID:	ASK-C1 (# 162)
Analysis type:	UnkNown
Chromatogram filename:	UNK-15092011-2.dat
Sample weight:	1.232



Element Name	Element %	Ret. Time
Nitrogen	8. 79	0. 73
Carbon	79. 61	1. 11
Hydrogen	6. 31	3. 83

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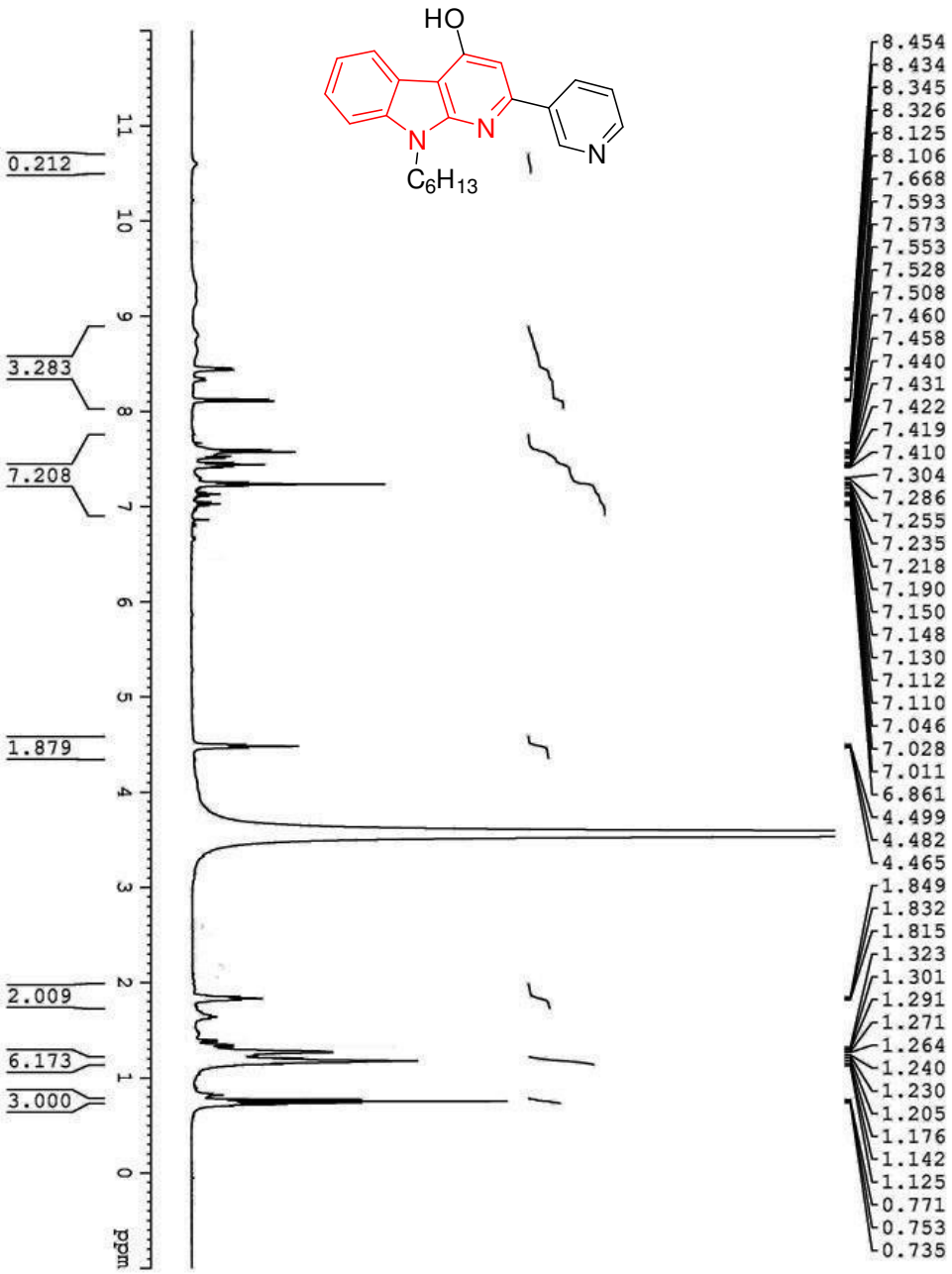
^{1}H NMR of 9-hexyl-2-(pyridin-3-yl)-9*H*-pyrido[2,3-*b*]indol-4-ol (2d)



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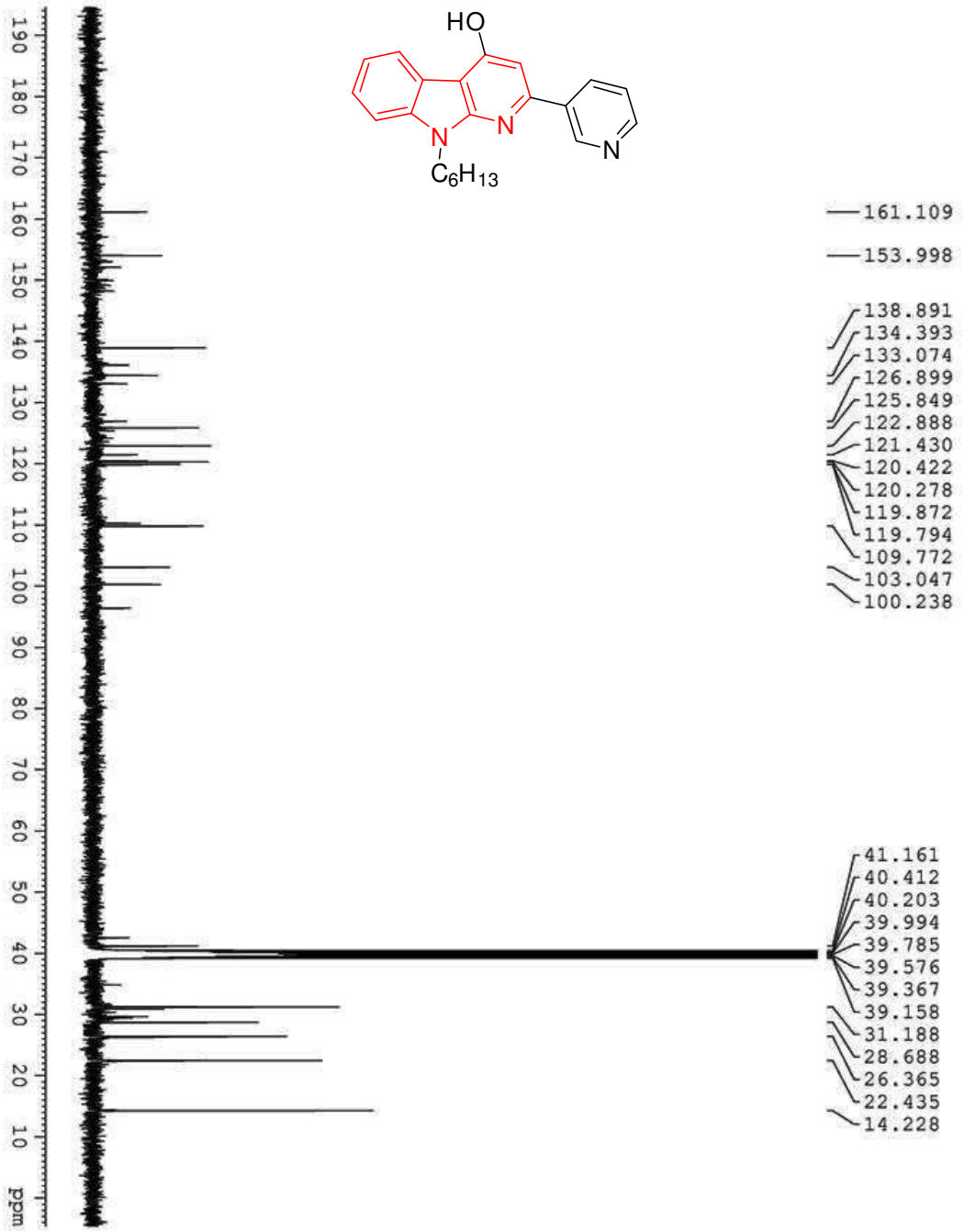
^{13}C NMR of 9-hexyl-2-(pyridin-3-yl)-9*H*-pyrido[2,3-*b*]indol-4-ol (2d)



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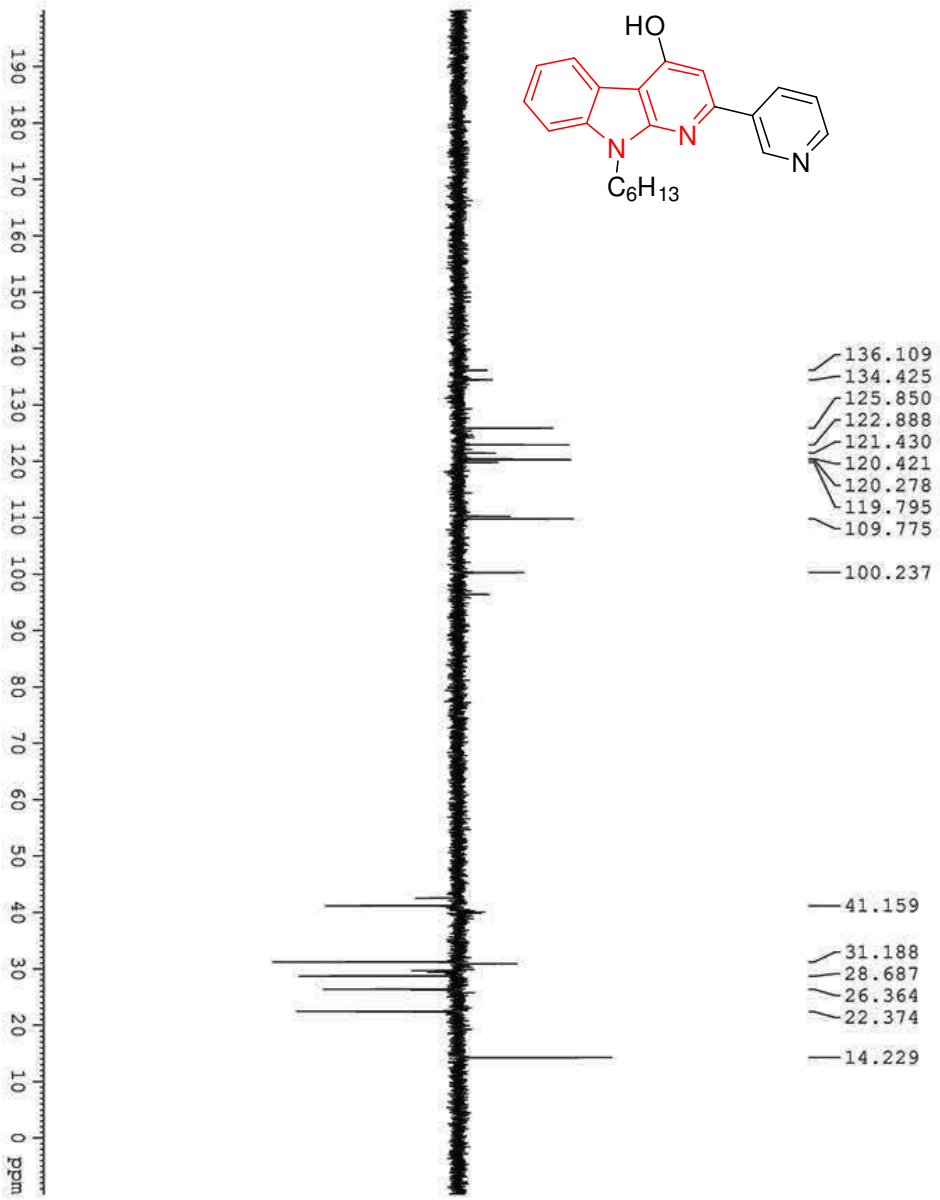
DEPT of 9-hexyl-2-(pyridin-3-yl)-9*H*-pyrido[2,3-*b*]indol-4-ol (2d)



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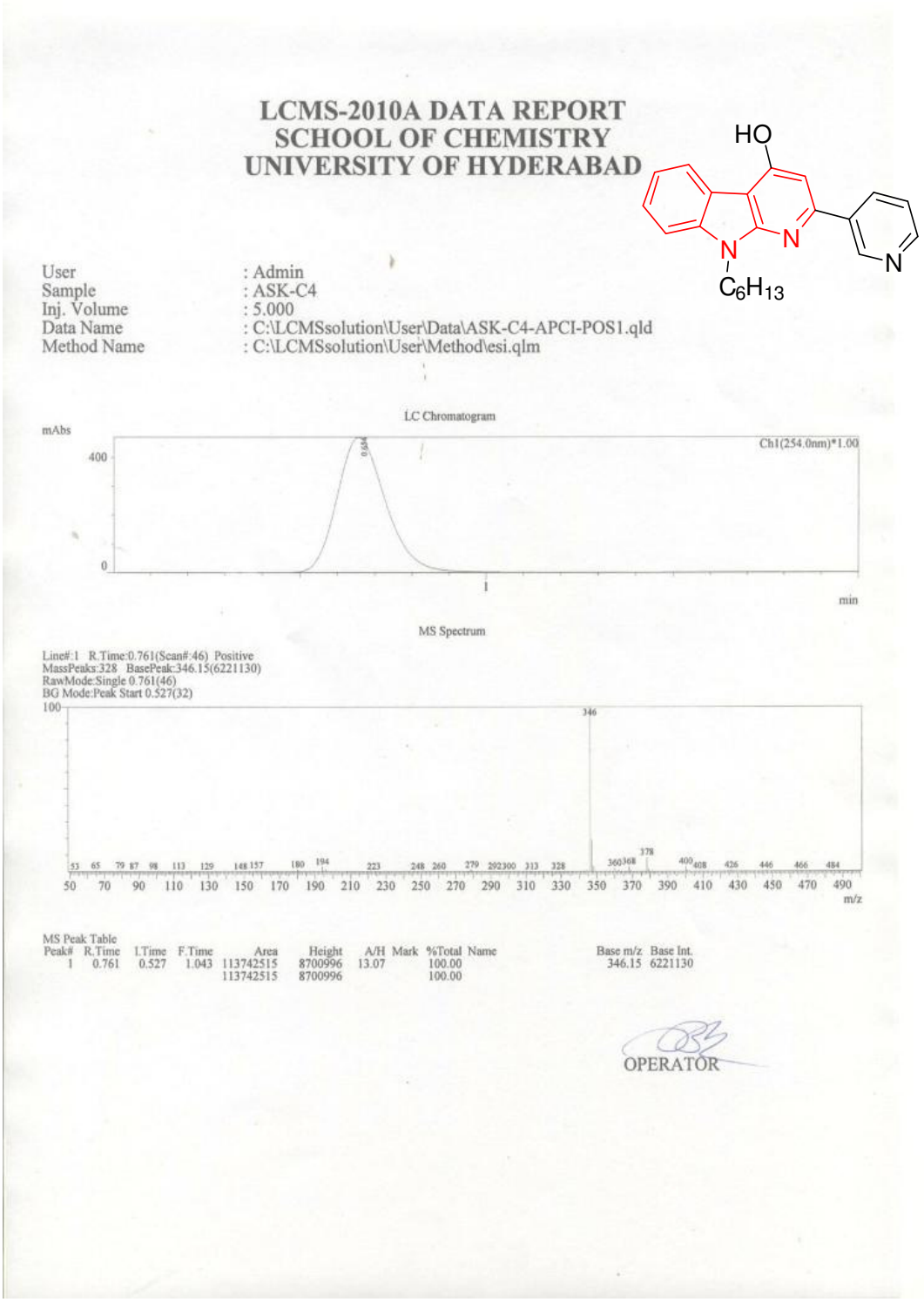
LCMS of 9-hexyl-2-(pyridin-3-yl)-9*H*-pyrido[2,3-*b*]indol-4-ol (2d)



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CHN Analysis of 9-hexyl-2-(pyridin-3-yl)-9*H*-pyrido[2,3-*b*]indol-4-ol (2d)

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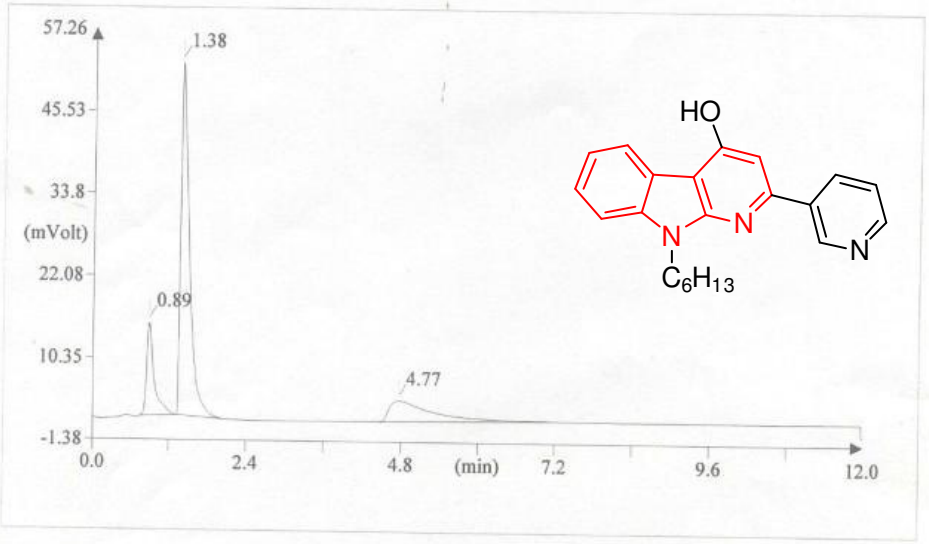
Method filename:	E:\Program Files\Thermo Finnigan\Eager 300 for EA1112\DATA\Sys_data_ex
Sample ID:	ASK-C4 (# 108) → 3d
Analysis type:	UnkNown
Chromatogram filename:	UNK-24012012-8.dat
Sample weight:	1.132



Element Name	Element %	Ret. Time
Nitrogen	12. 23	0. 89
Carbon	76. 38	1. 38
Hydrogen	6. 63	4. 77

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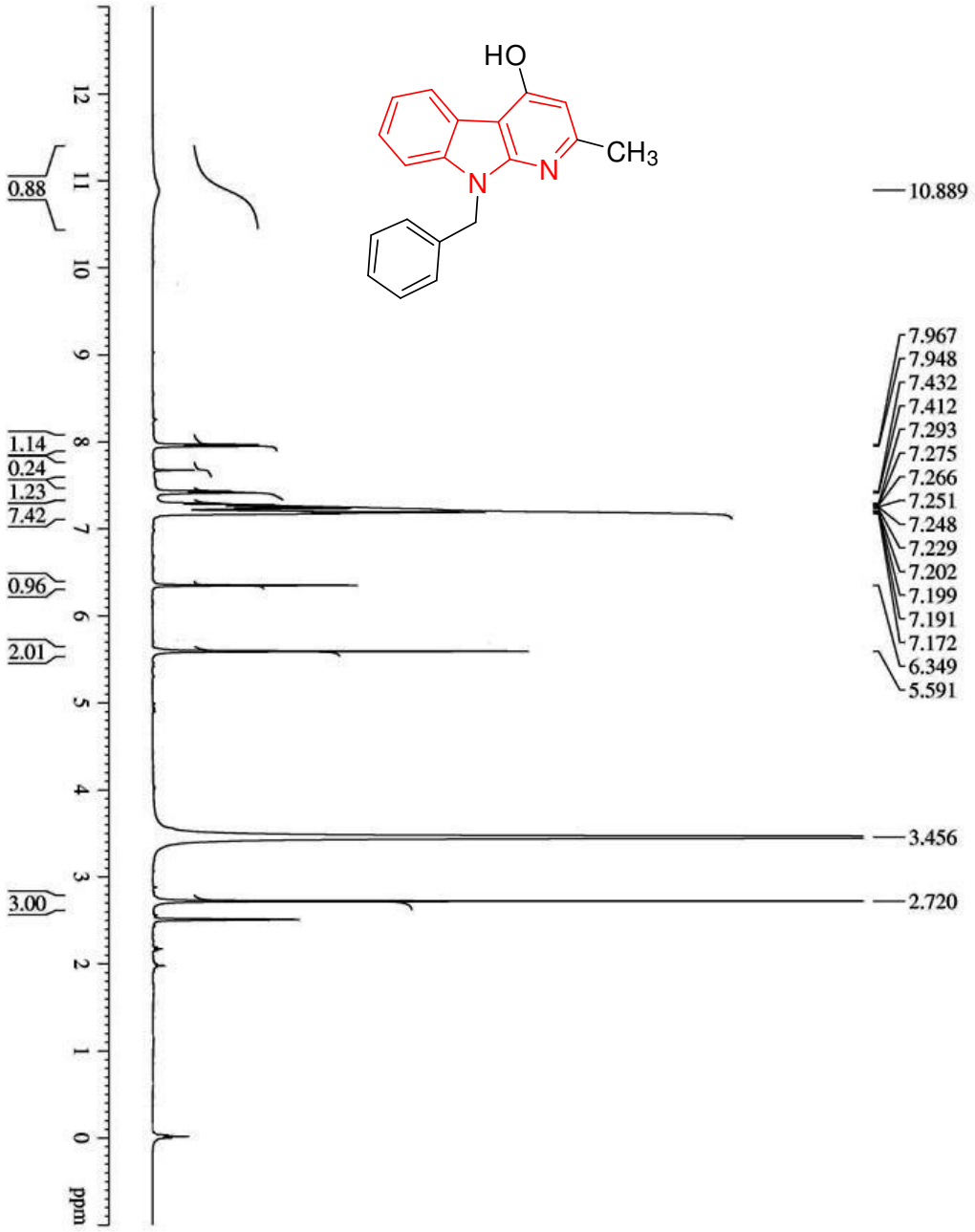
^{1}H NMR of 9-benzyl-2-methyl-9*H*-pyrido[2,3-*b*]indol-4-ol (2e)



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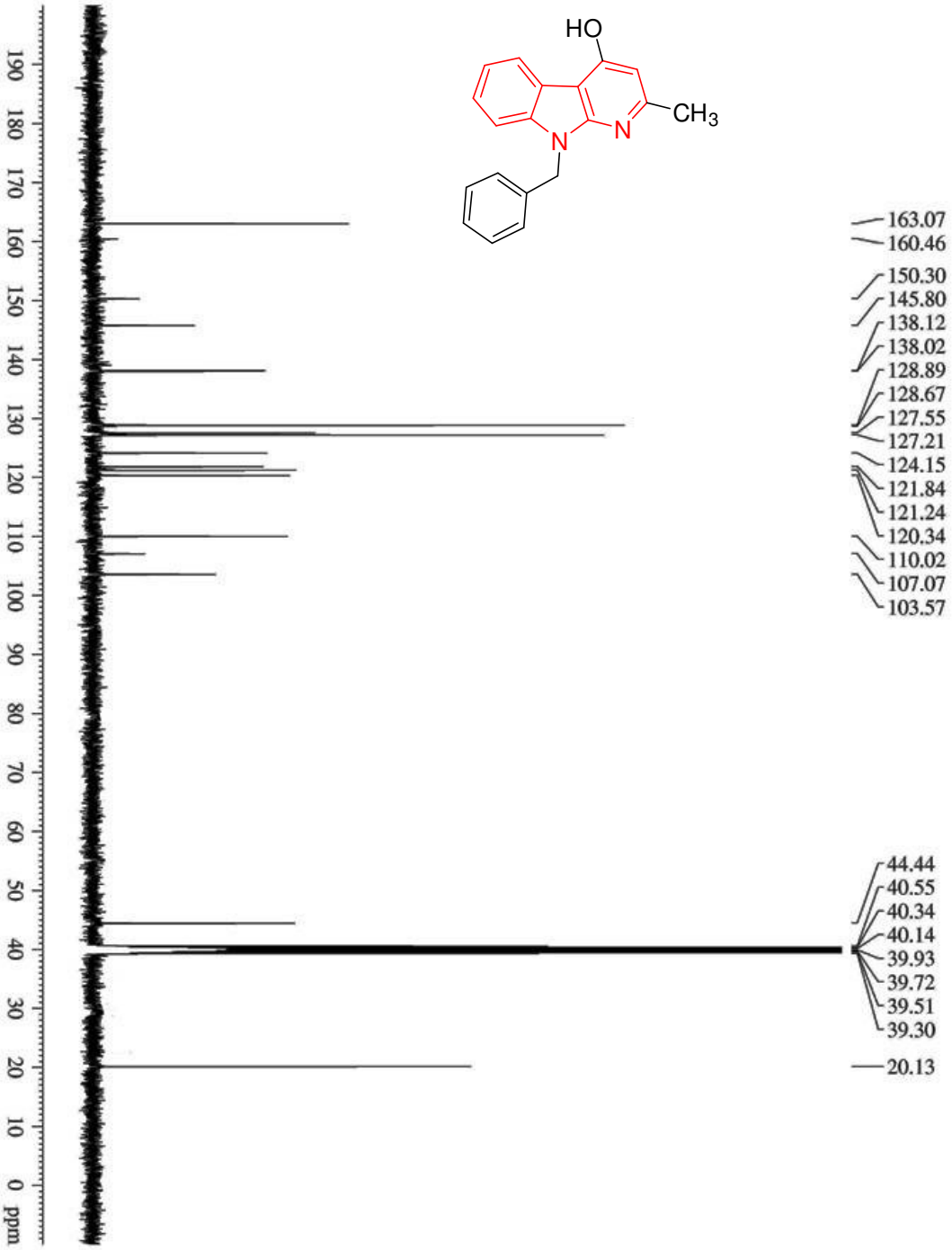
^{13}C NMR of 9-benzyl-2-methyl-9*H*-pyrido[2,3-*b*]indol-4-ol (2e)



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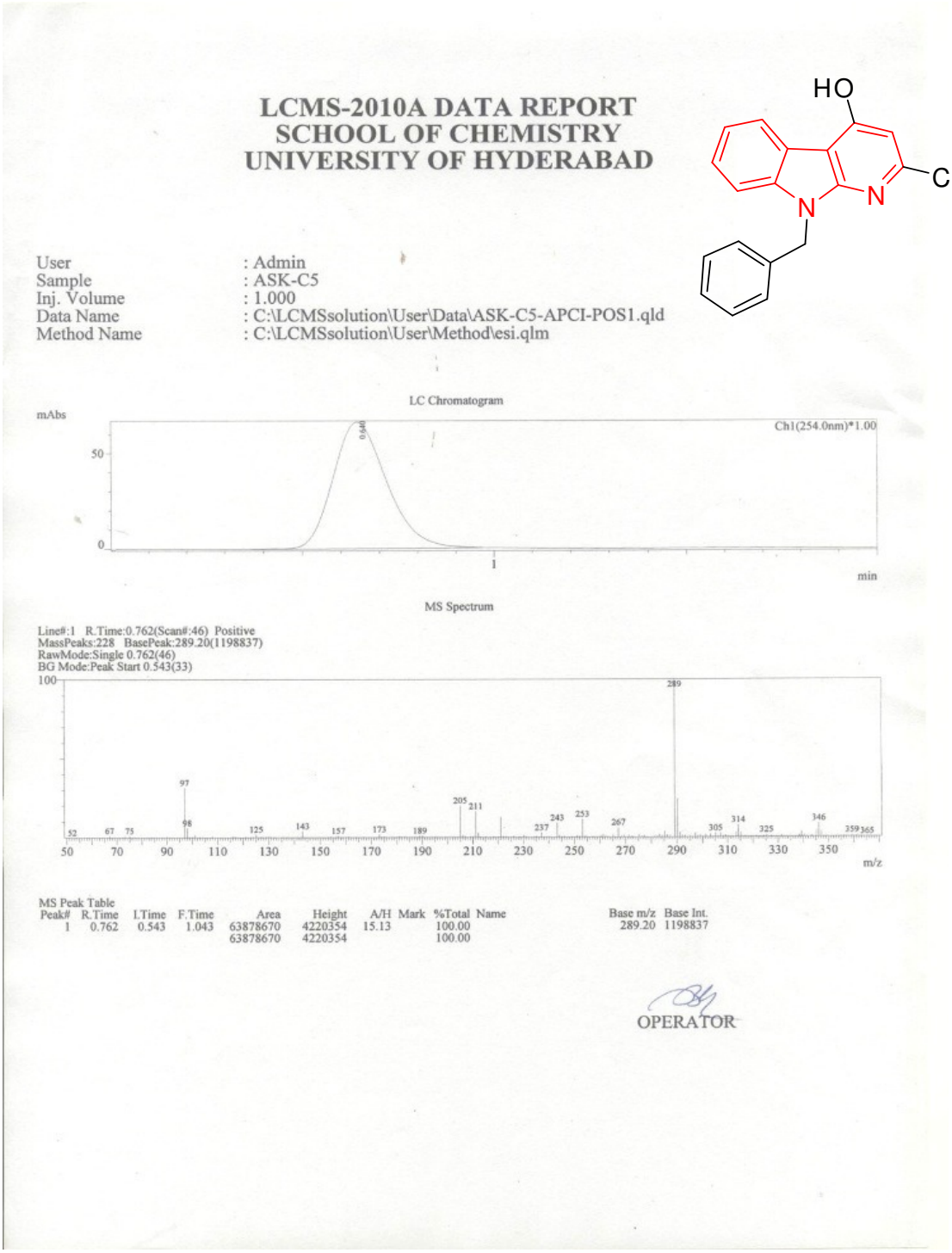
LCMS of 9-benzyl-2-methyl-9*H*-pyrido[2,3-*b*]indol-4-ol (2e)



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CHN Analysis of 9-benzyl-2-methyl-9*H*-pyrido[2,3-*b*]indol-4-ol (2e)

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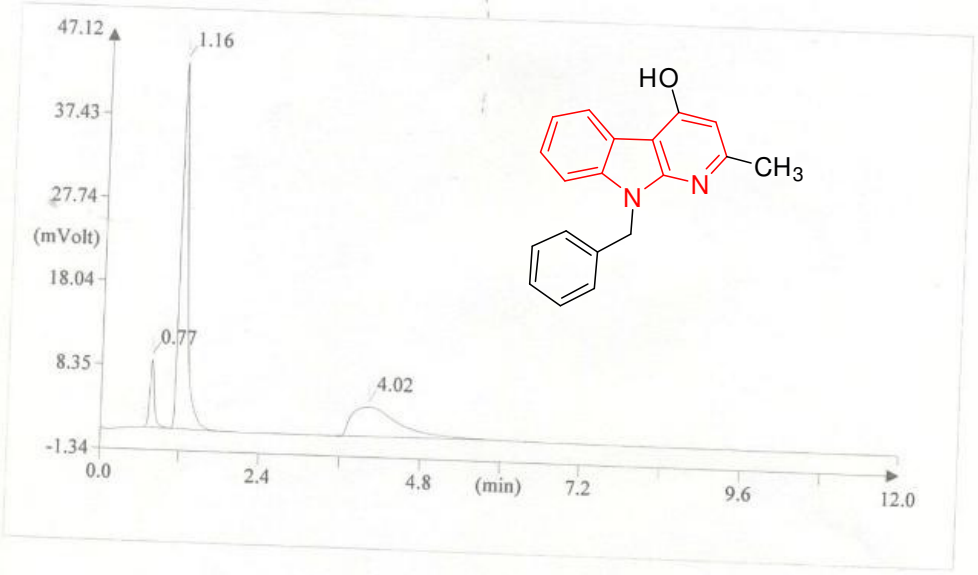
Method filename:	E:\Program Files\Thermo Finnigan\Eager 300 for EA1112\DATA\Sys_data_ex
Sample ID:	ASK-C5 (# 133)
Analysis type:	UnkNown
Chromatogram filename:	UNK-30012012-3.dat
Sample weight:	1.103



Element Name	Element %	Ret. Time
Nitrogen	9. 65	0. 77
Carbon	79. 03	1. 16
Hydrogen	5. 51	4. 02

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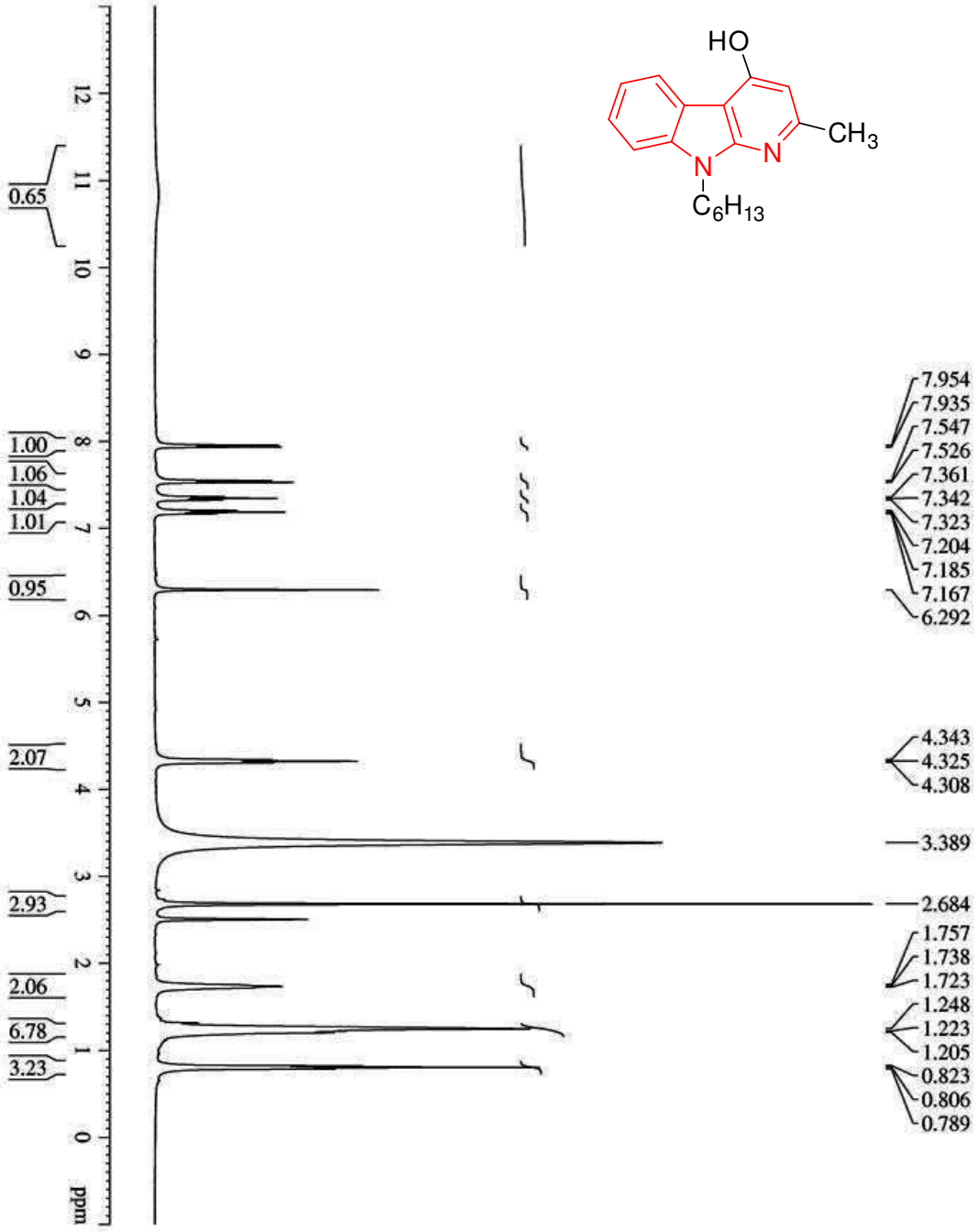
^{1}H NMR of 9-hexyl-2-methyl-9*H*-pyrido[2,3-*b*]indol-4-ol (2f)



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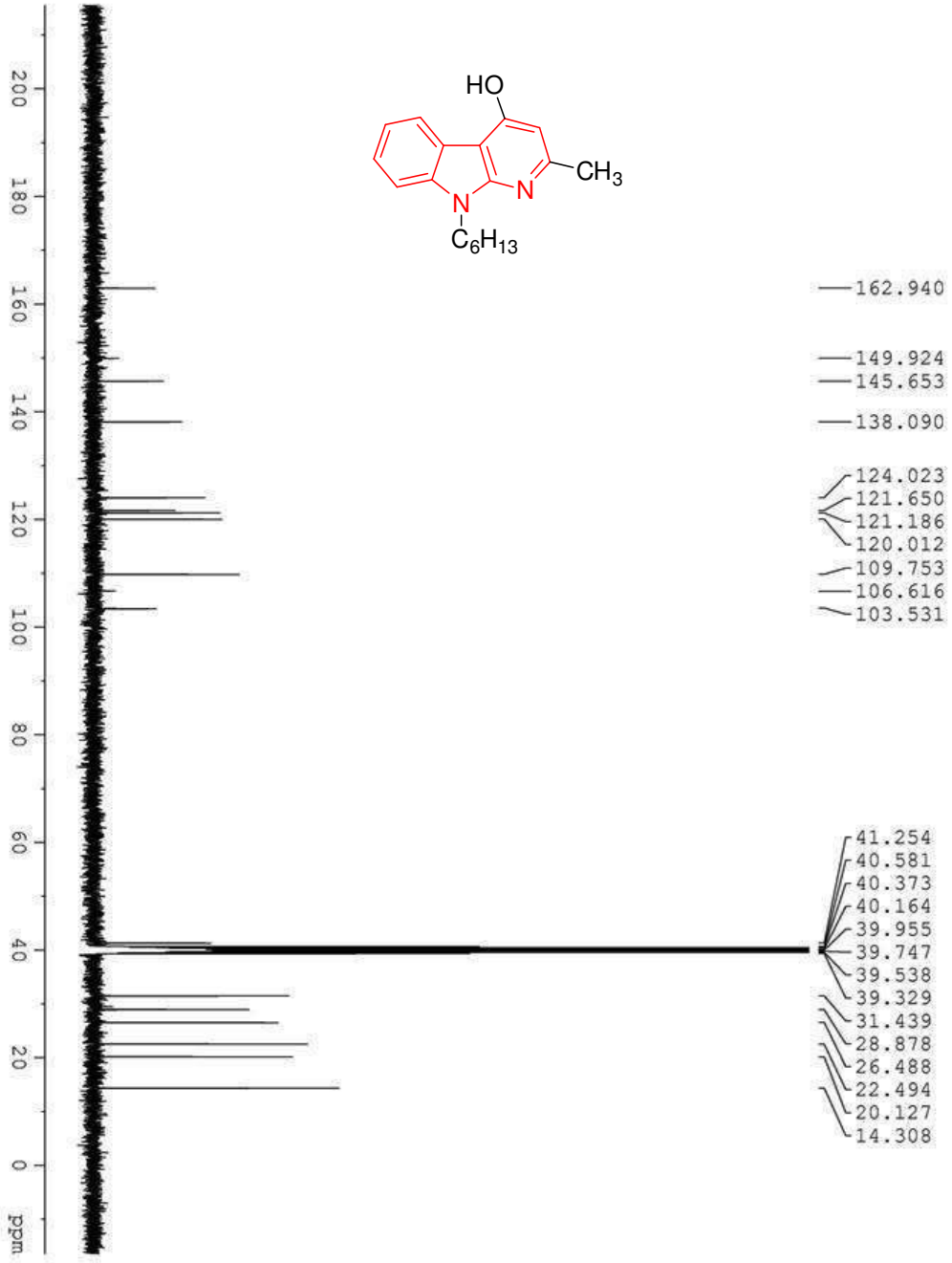
^{13}C NMR of 9-hexyl-2-methyl-9*H*-pyrido[2,3-*b*]indol-4-ol (2f)



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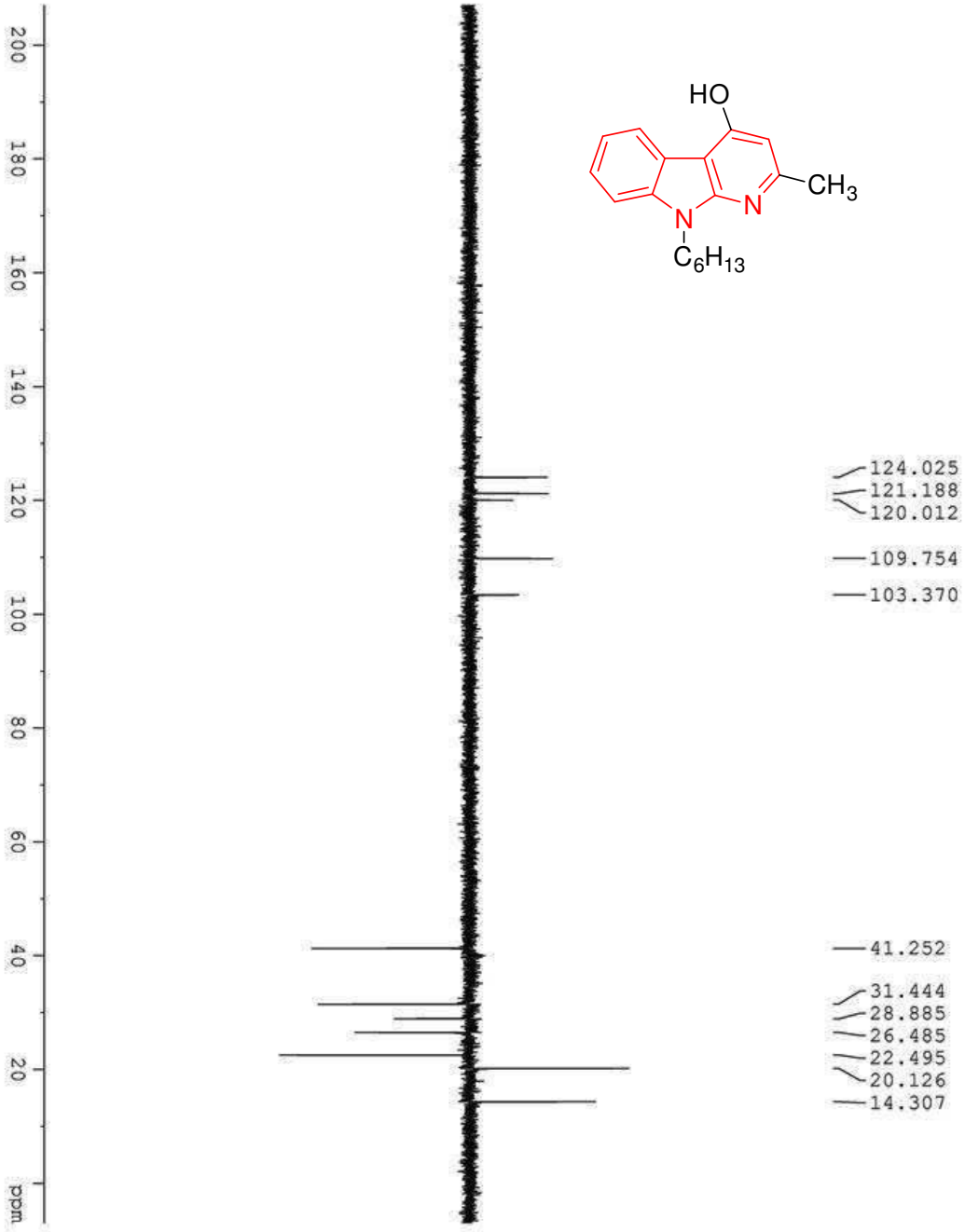
DEPT of 9-hexyl-2-methyl-9*H*-pyrido[2,3-*b*]indol-4-ol (2f)



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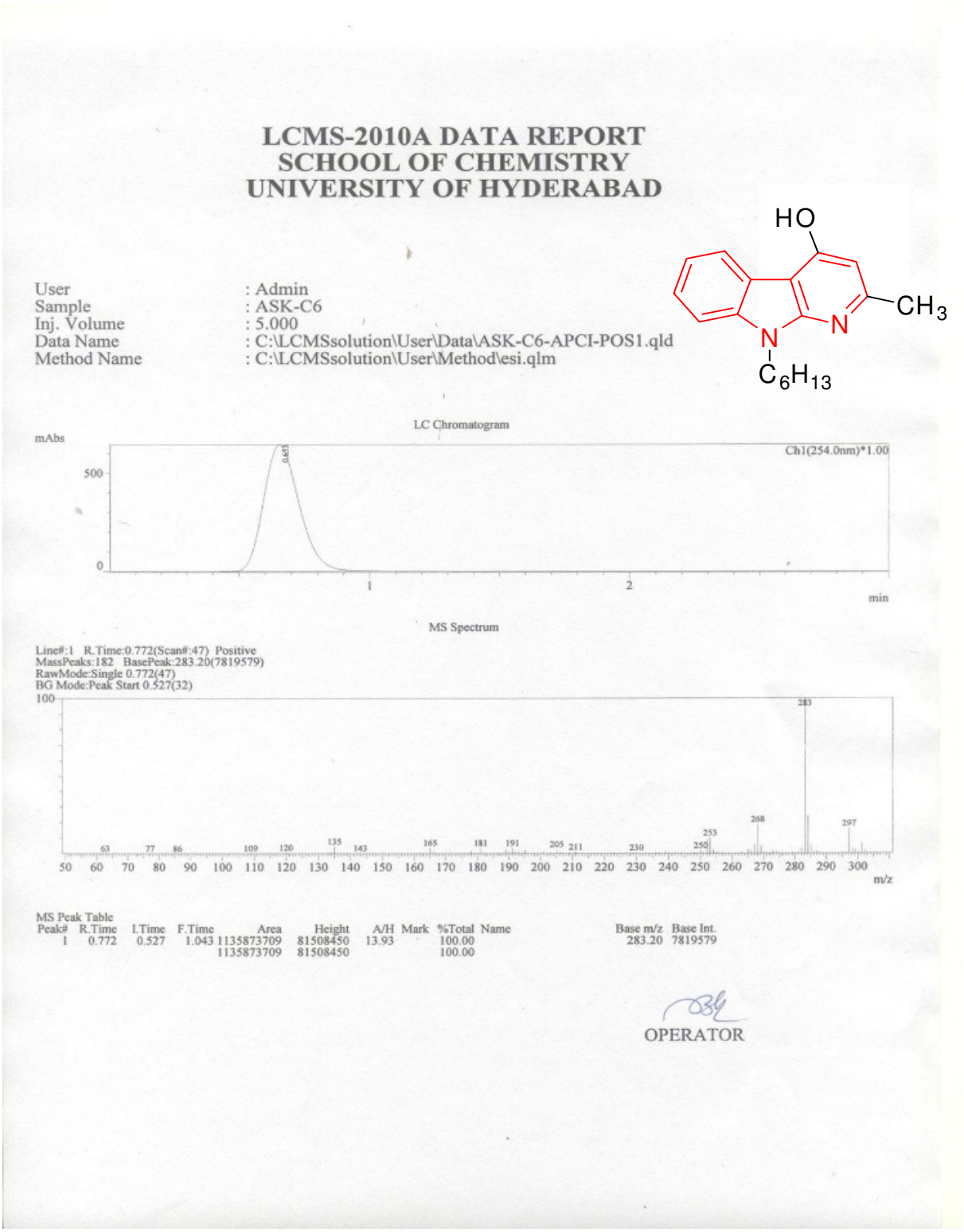
LCMS of 9-hexyl-2-methyl-9*H*-pyrido[2,3-*b*]indol-4-ol (2f)



LCMS-2010A DATA REPORT
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UNIVERSITY OF HYDERABAD

User : Admin
Sample : ASK-C6
Inj. Volume : 5.000
Data Name : C:\LCMSsolution\User\Data\ASK-C6-APCI-POS1.qld
Method Name : C:\LCMSsolution\User\Method\esi.qlm

LC Chromatogram

MS Spectrum

Line#:1 R.Time:0.772(Scan#:47) Positive
MassPeaks:182 BasePeak:283.20(7819579)
RawMode:Single 0.772(47)
BG Mode:Peak Start 0.527(32)

MS Peak Table

Peak#	R.Time	I.Time	F.Time	Area	Height	A/H	Mark	%Total	Name	Base m/z	Base Int.
1	0.772	0.527	1.043	1135873709	81508450	13.93		100.00		283.20	7819579
				1135873709	81508450			100.00			

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CHN Analysis of 9-hexyl-2-methyl-9*H*-pyrido[2,3-*b*]indol-4-ol (2f)

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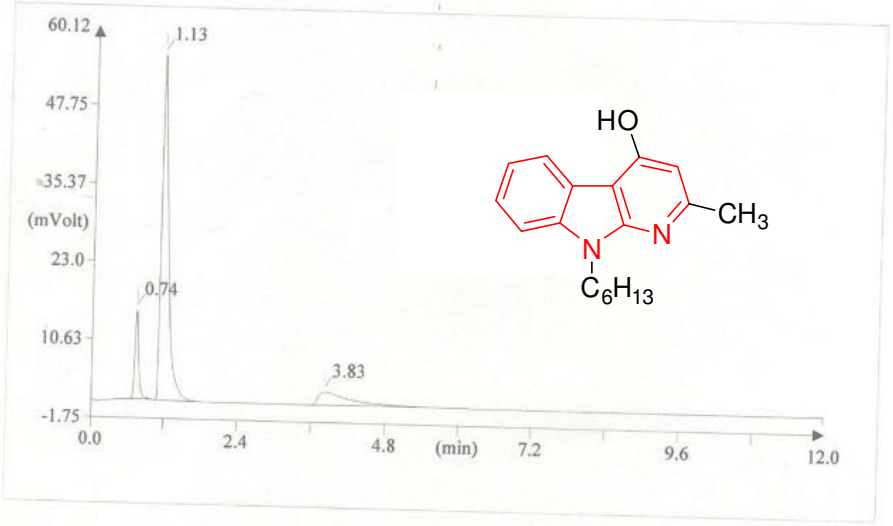
Method filename:	E:\Program Files\Thermo Finnigan\Eager 300 for EA1112\DATA\Sys_data_ex
Sample ID:	ASK-C6 (# 149)
Analysis type:	UnkNown
Chromatogram filename:	UNK-30012012-19.dat
Sample weight:	1.145



Element Name	Element %	Ret. Time
Nitrogen	9. 88	0. 74
Carbon	76. 45	1. 13
Hydrogen	7. 81	3. 83

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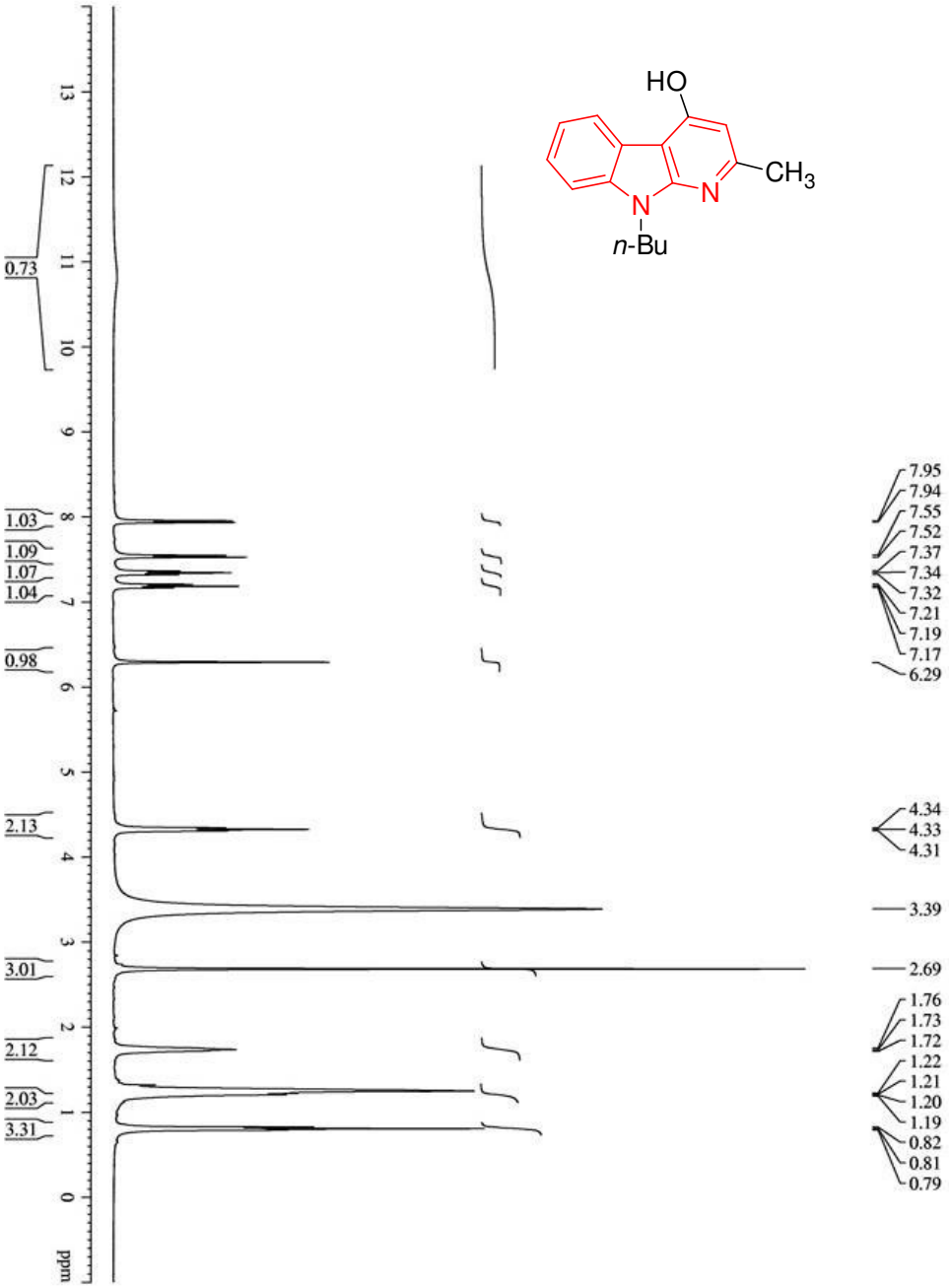
^{1}H NMR of 9-butyl-2-methyl-9*H*-pyrido[2,3-*b*]indol-4-ol (2g)



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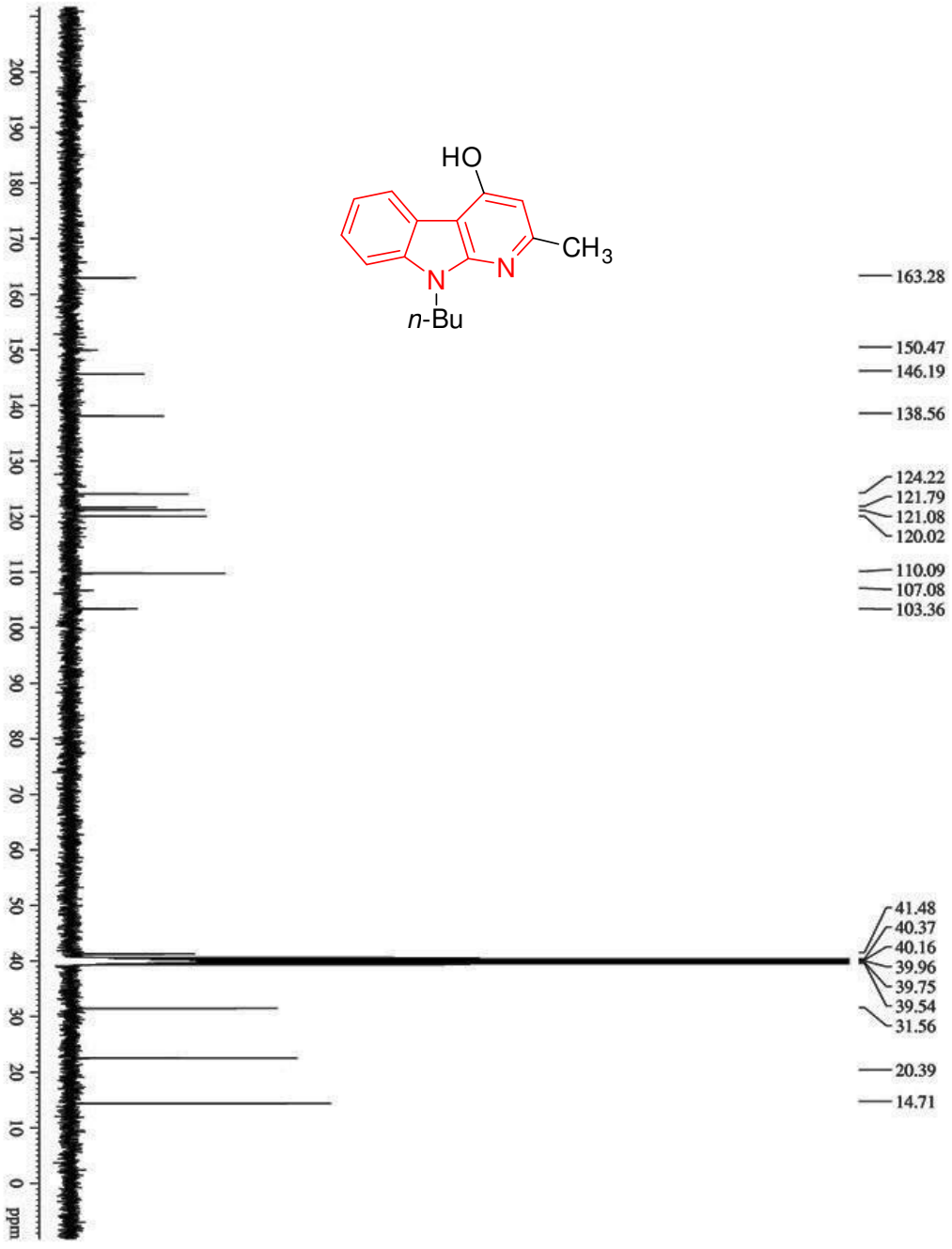
^{13}C NMR of 9-butyl-2-methyl-9*H*-pyrido[2,3-*b*]indol-4-ol (2g)



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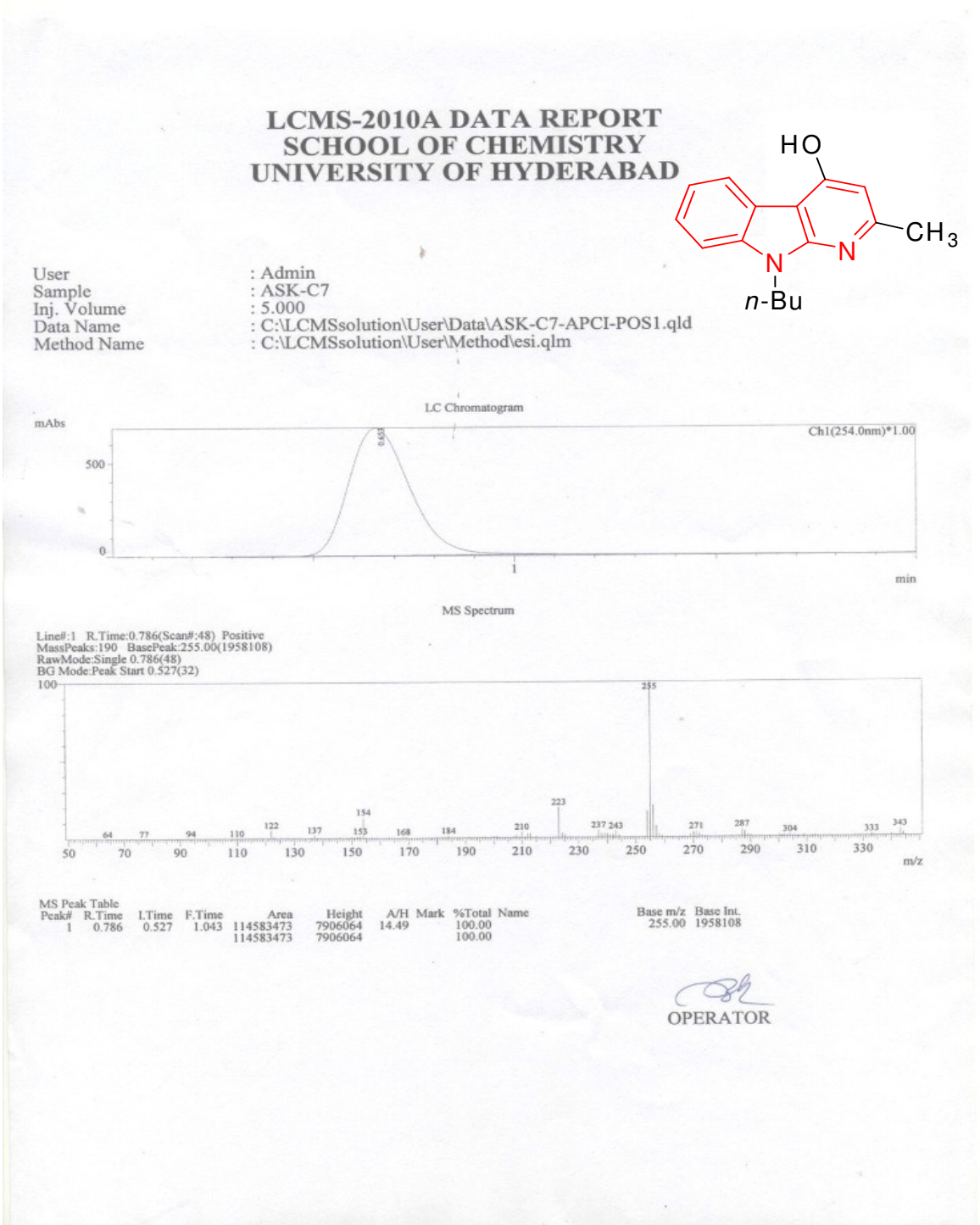
LCMS of 9-butyl-2-methyl-9*H*-pyrido[2,3-*b*]indol-4-ol (2g)



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CHN Analysis of 9-butyl-2-methyl-9*H*-pyrido[2,3-*b*]indol-4-ol (2g)

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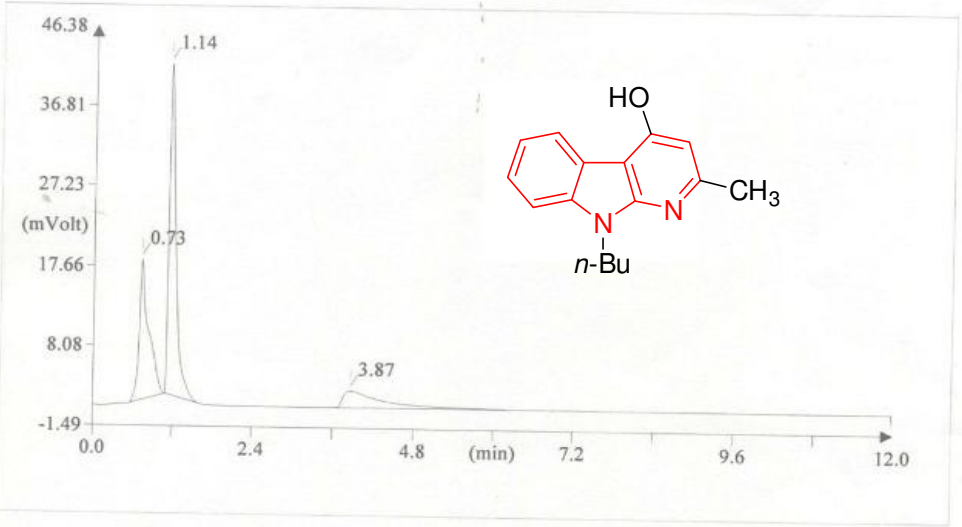
Method filename:	E:\Program Files\Thermo Finnigan\Eager 300 for EA1112\DATA\Sys_data_ex
Sample ID:	ASK-C7 (# 144)
Analysis type:	UnkNown
Chromatogram filename:	UNK-30012012-14.dat
Sample weight:	1.108



Element Name	Element %	Ret. Time
Nitrogen	11. 12	0. 73
Carbon	75. 46	1. 14
Hydrogen	7. 21	3. 87

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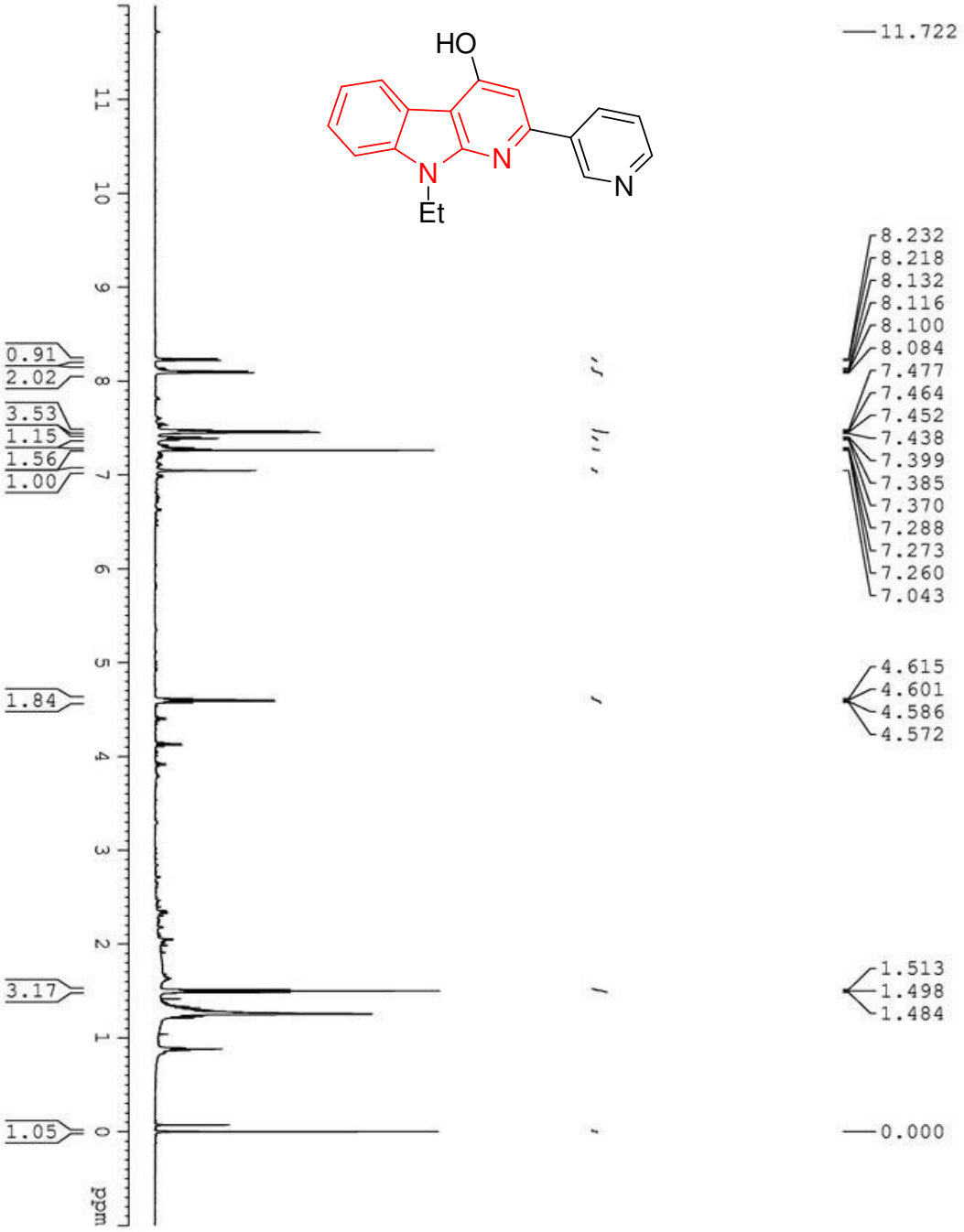
^{1}H NMR of 9-ethyl-2-(pyridin-3-yl)-9*H*-pyrido[2,3-*b*]indol-4-ol (2h)



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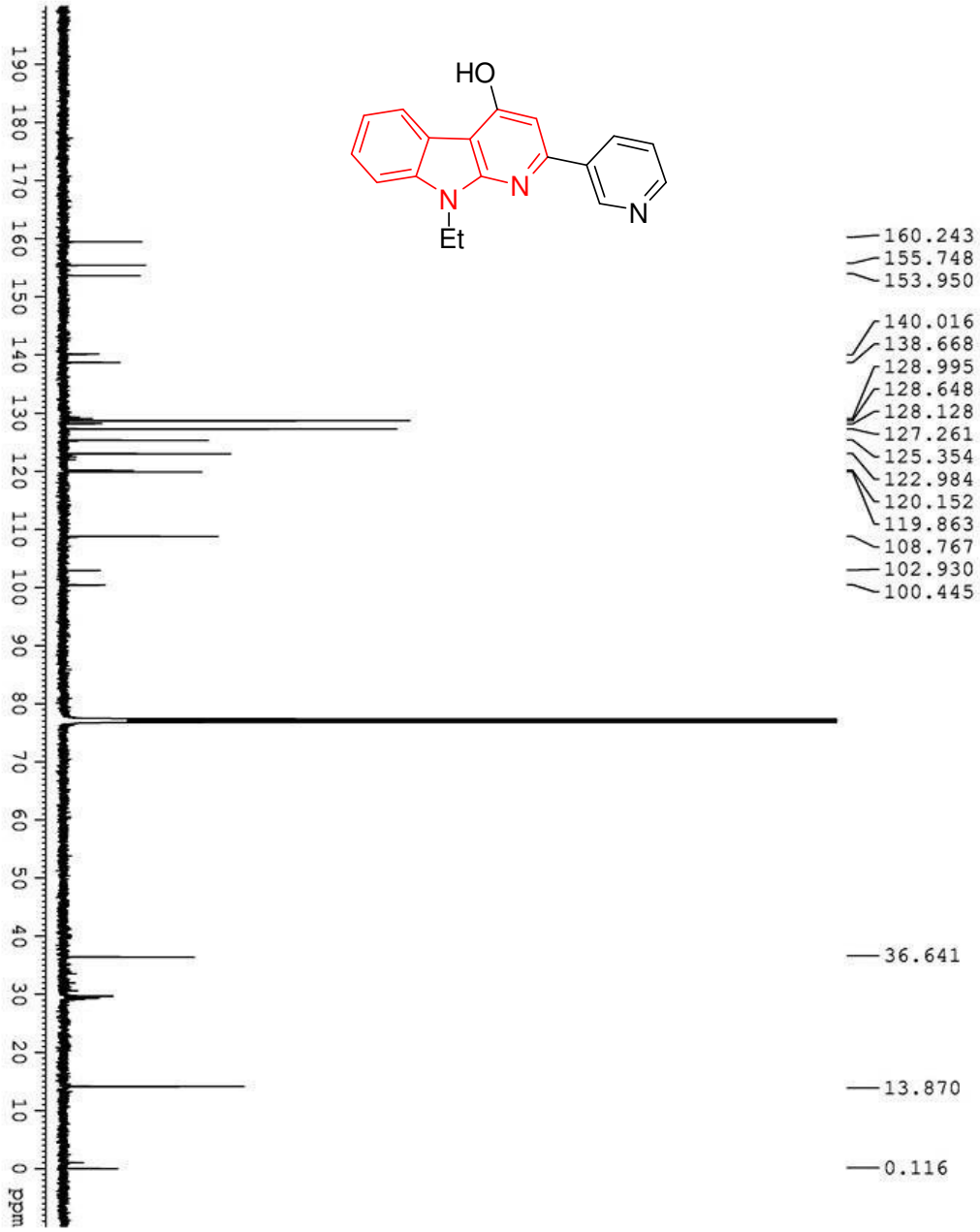
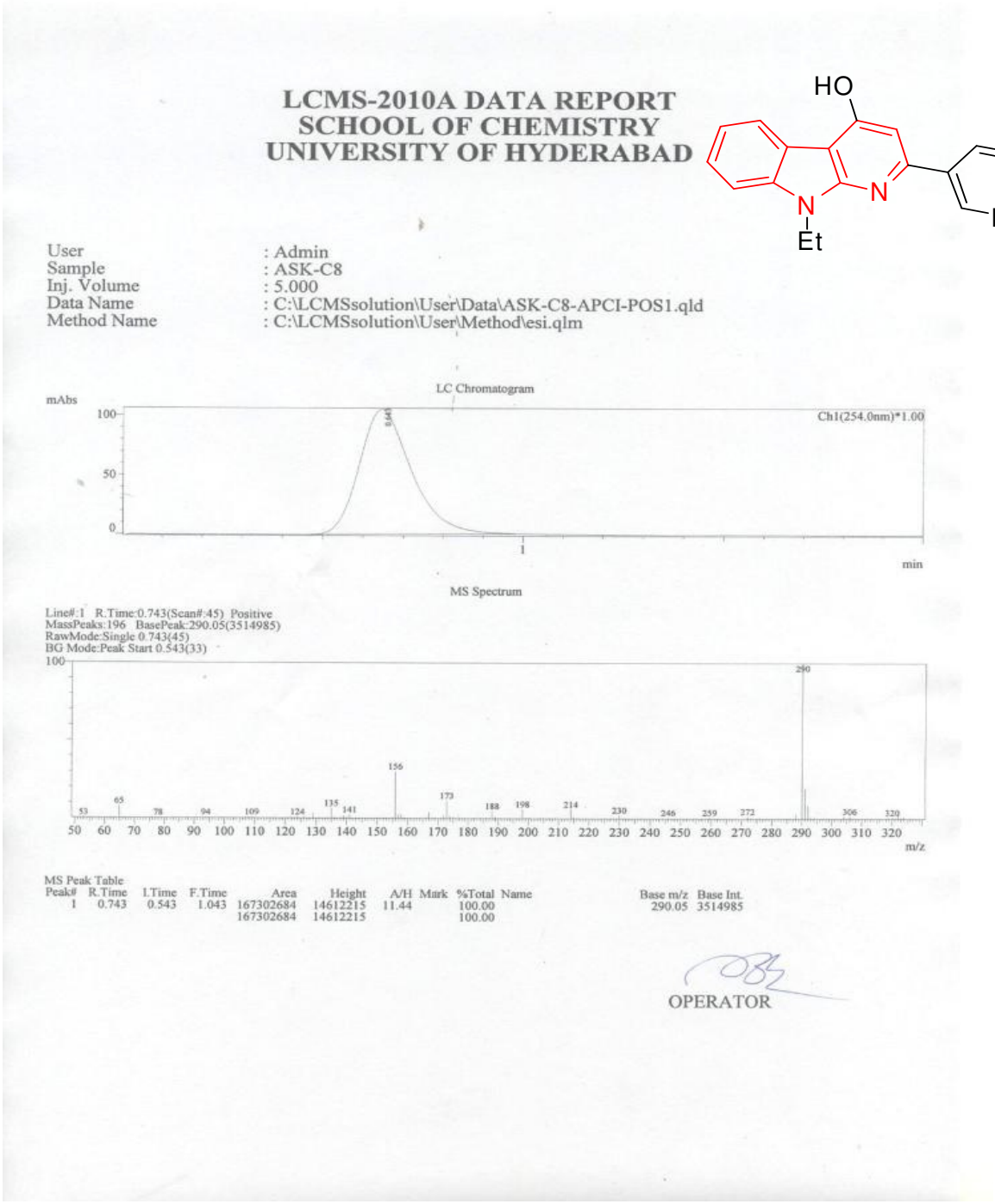
^{13}C NMR of 9-ethyl-2-(pyridin-3-yl)-9*H*-pyrido[2,3-*b*]indol-4-ol (2h)



LCMS of 9-ethyl-2-(pyridin-3-yl)-9*H*-pyrido[2,3-*b*]indol-4-ol (2h)



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CHN Analysis of 9-ethyl-2-(pyridin-3-yl)-9*H*-pyrido[2,3-*b*]indol-4-ol (2h)

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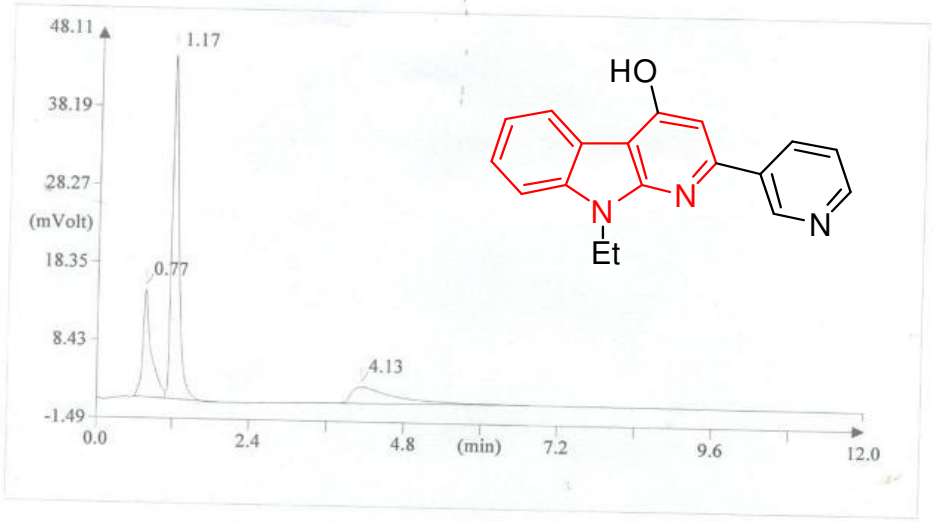
Method filename:	E:\Program Files\Thermo Finnigan\Eager 300 for EA1112\DATA\Sys_data_ex
Sample ID:	ASK-C8 (# 101)
Analysis type:	UnkNown
Chromatogram filename:	UNK-24012012-1.dat
Sample weight:	.978



Element Name	Element %	Ret. Time
Nitrogen	14. 43	0. 77
Carbon	74. 65	1. 17
Hydrogen	5. 28	4. 13

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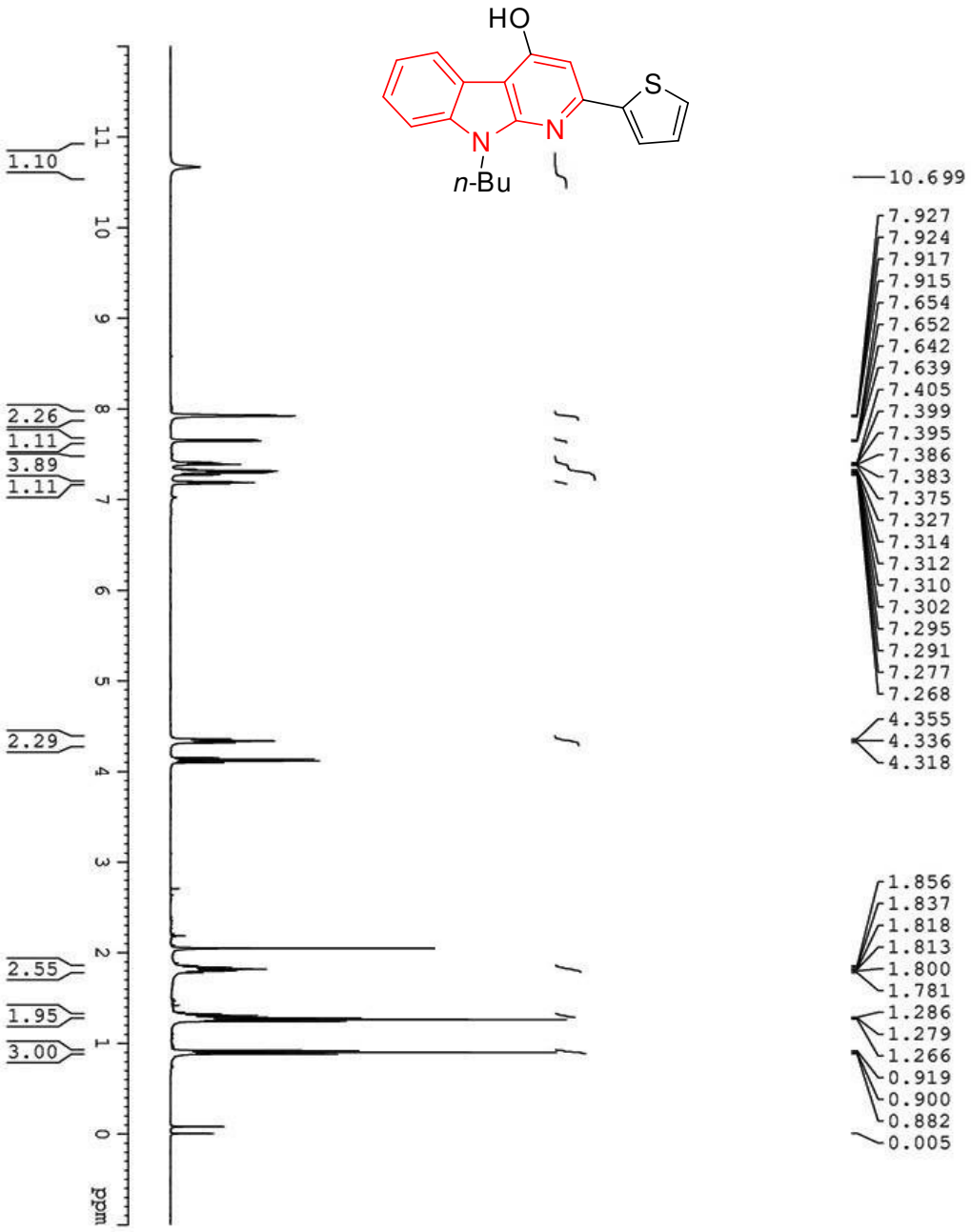
^{1}H NMR of 9-butyl-2-(thiophen-2-yl)-9*H*-pyrido[2,3-*b*]indol-4-ol (2i)



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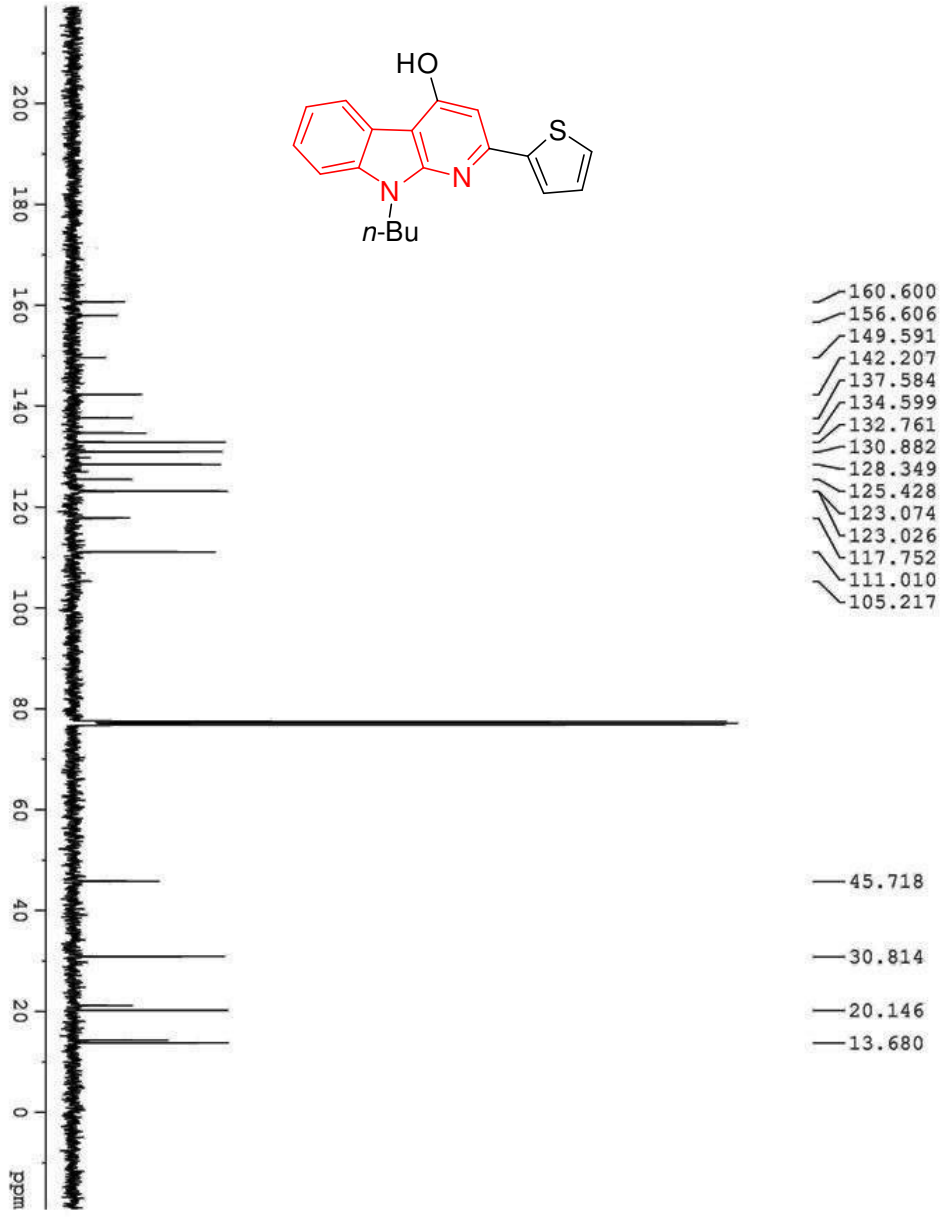
^{13}C NMR of 9-butyl-2-(thiophen-2-yl)-9*H*-pyrido[2,3-*b*]indol-4-ol (2i)



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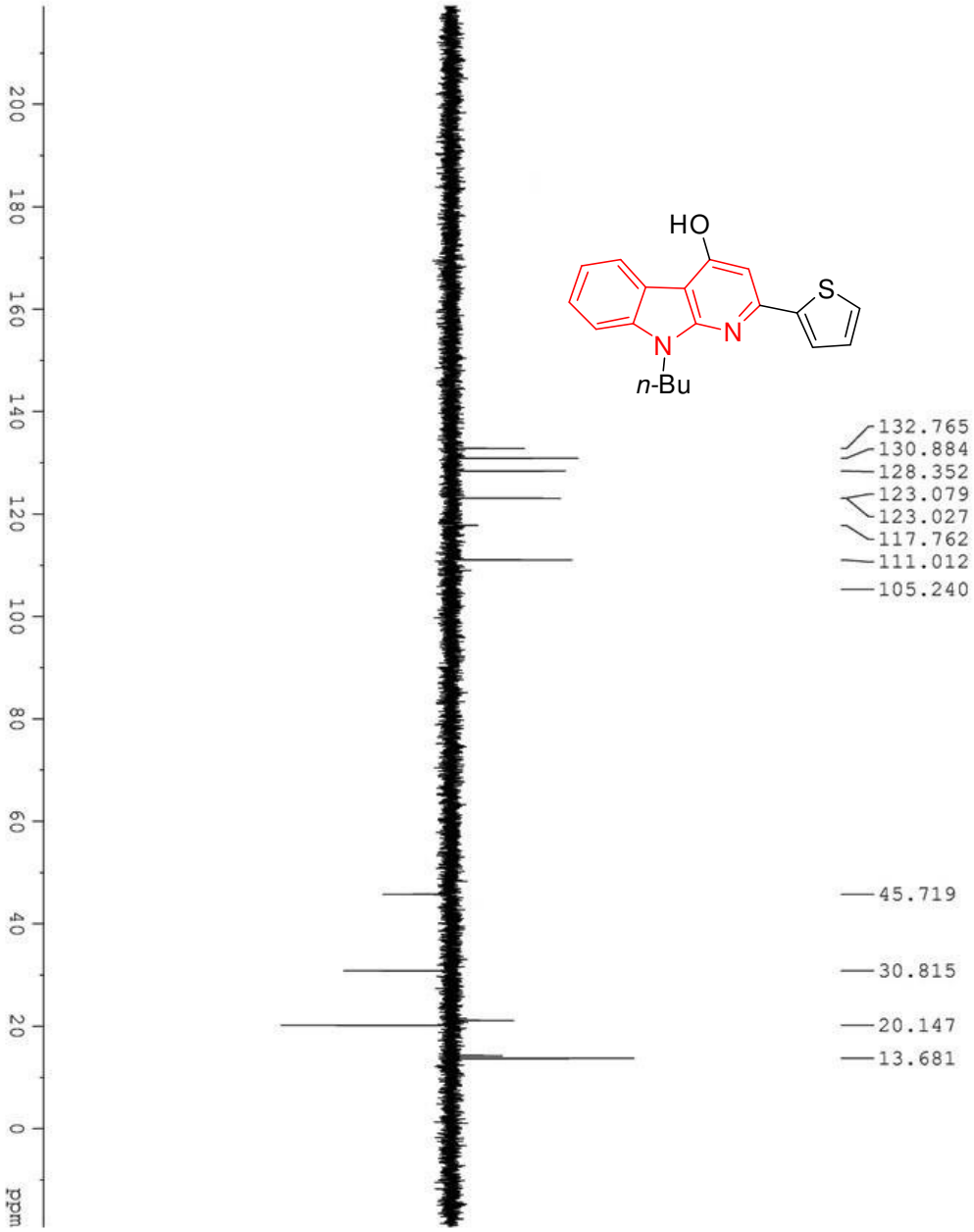
DEPT of 9-butyl-2-(thiophen-2-yl)-9*H*-pyrido[2,3-*b*]indol-4-ol (2i)



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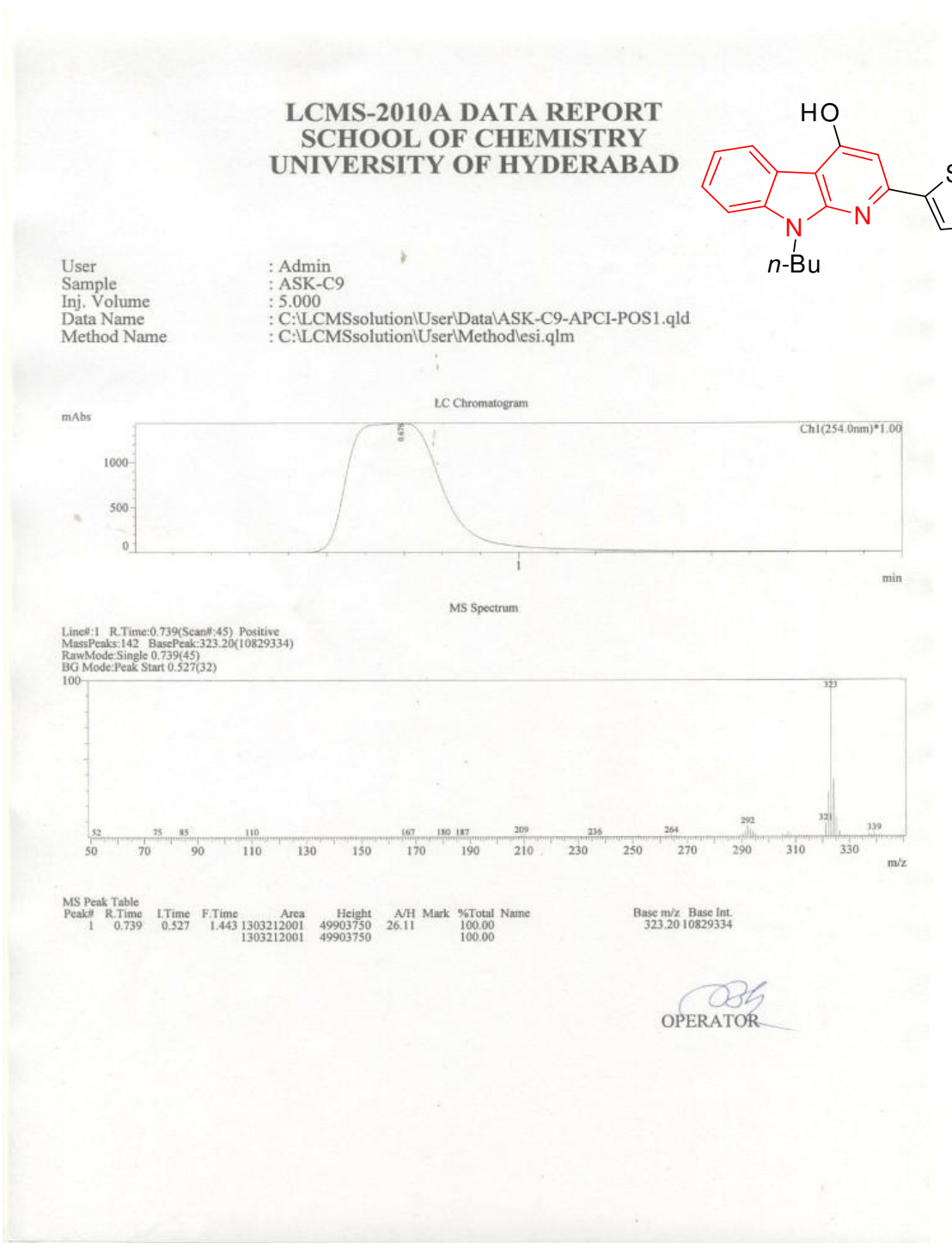
LCMS of 9-butyl-2-phenyl-9*H*-pyrido[2,3-*b*]indol-4-ol (2i)



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CHN Analysis of 9-butyl-2-phenyl-9*H*-pyrido[2,3-*b*]indol-4-ol (2i)

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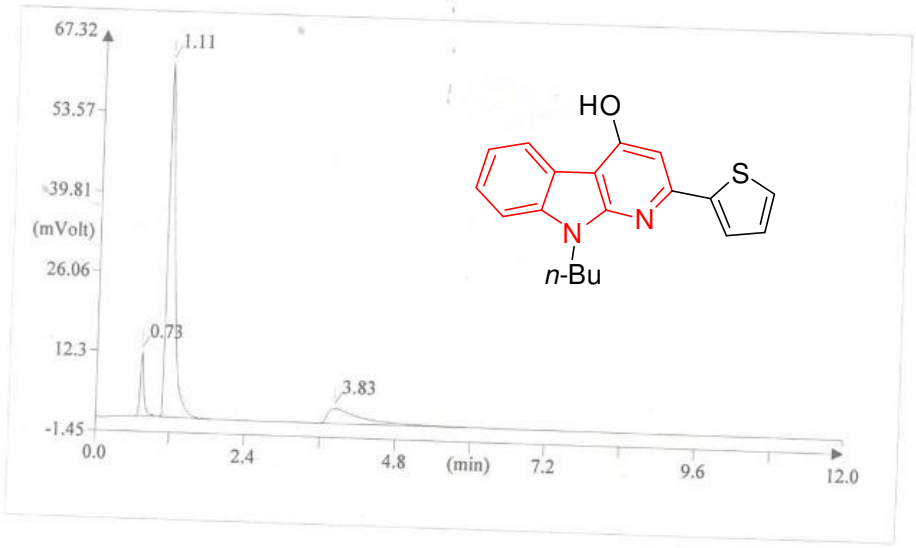
Method filename:	E:\Program Files\Thermo Finnigan\Eager 300 for EA1112\DATA\Sys_data_ex
Sample ID:	ASK-C9 (# 112) → 31
Analysis type:	UnkNown
Chromatogram filename:	UNK-24012012-12.dat
Sample weight:	1.215



Element Name	Element %	Ret. Time
Nitrogen	8. 59	0. 73
Carbon	70. 65	1. 11
Hydrogen	5. 58	3. 83

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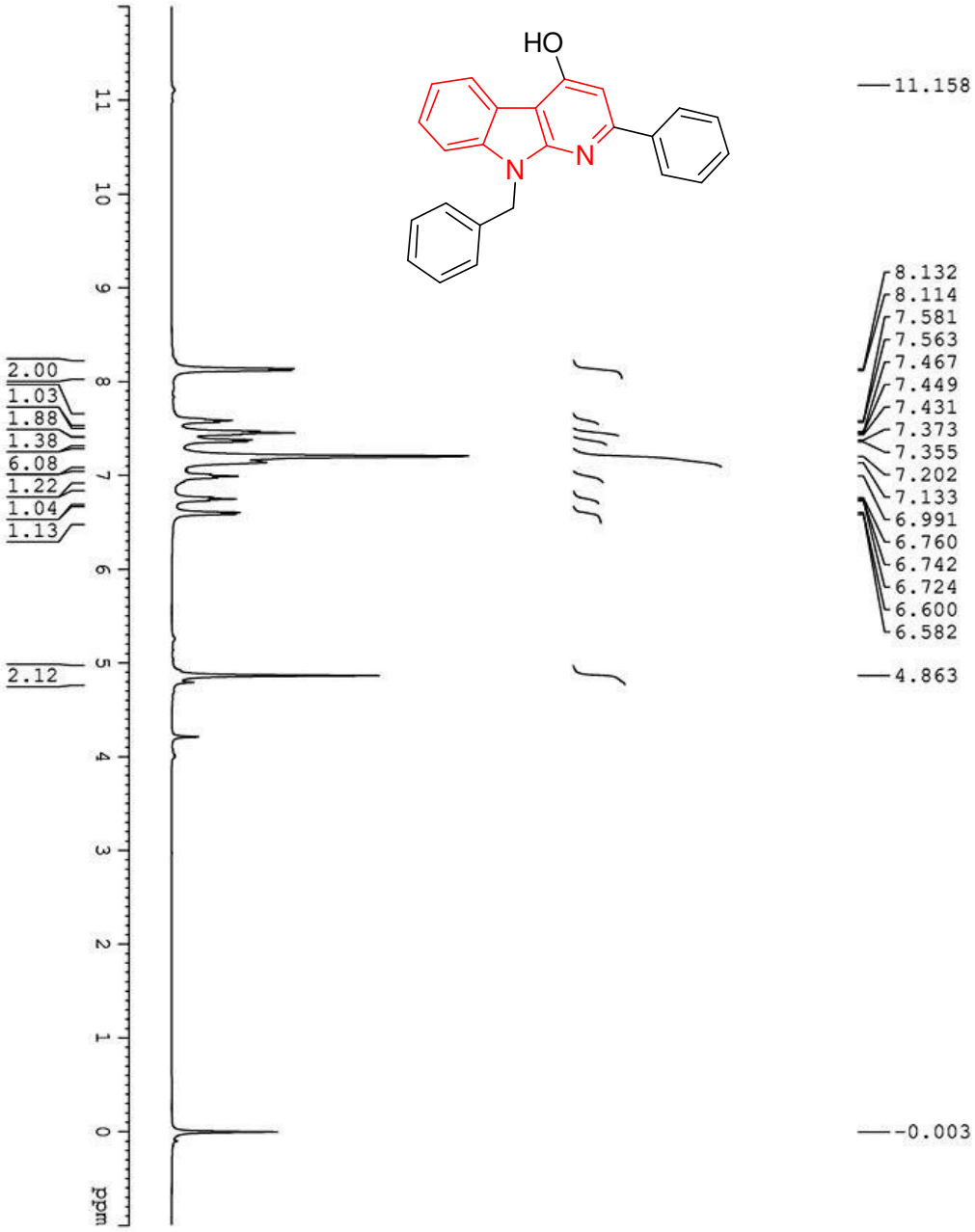
^{1}H NMR of 9-benzyl-2-phenyl-9*H*-pyrido[2,3-*b*]indol-4-ol (2j)



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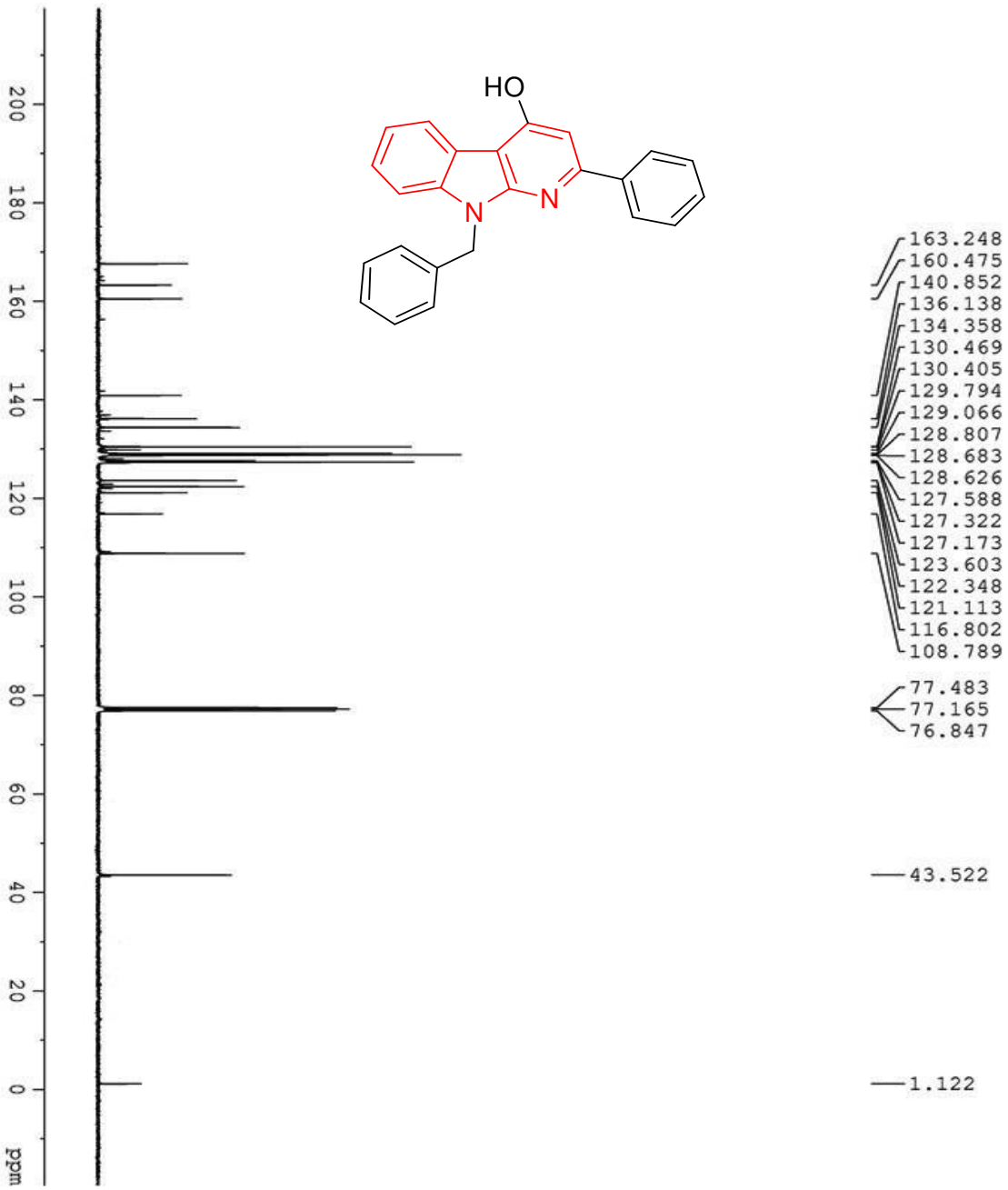
^{13}C NMR of 9-benzyl-2-phenyl-9*H*-pyrido[2,3-*b*]indol-4-ol (2j)



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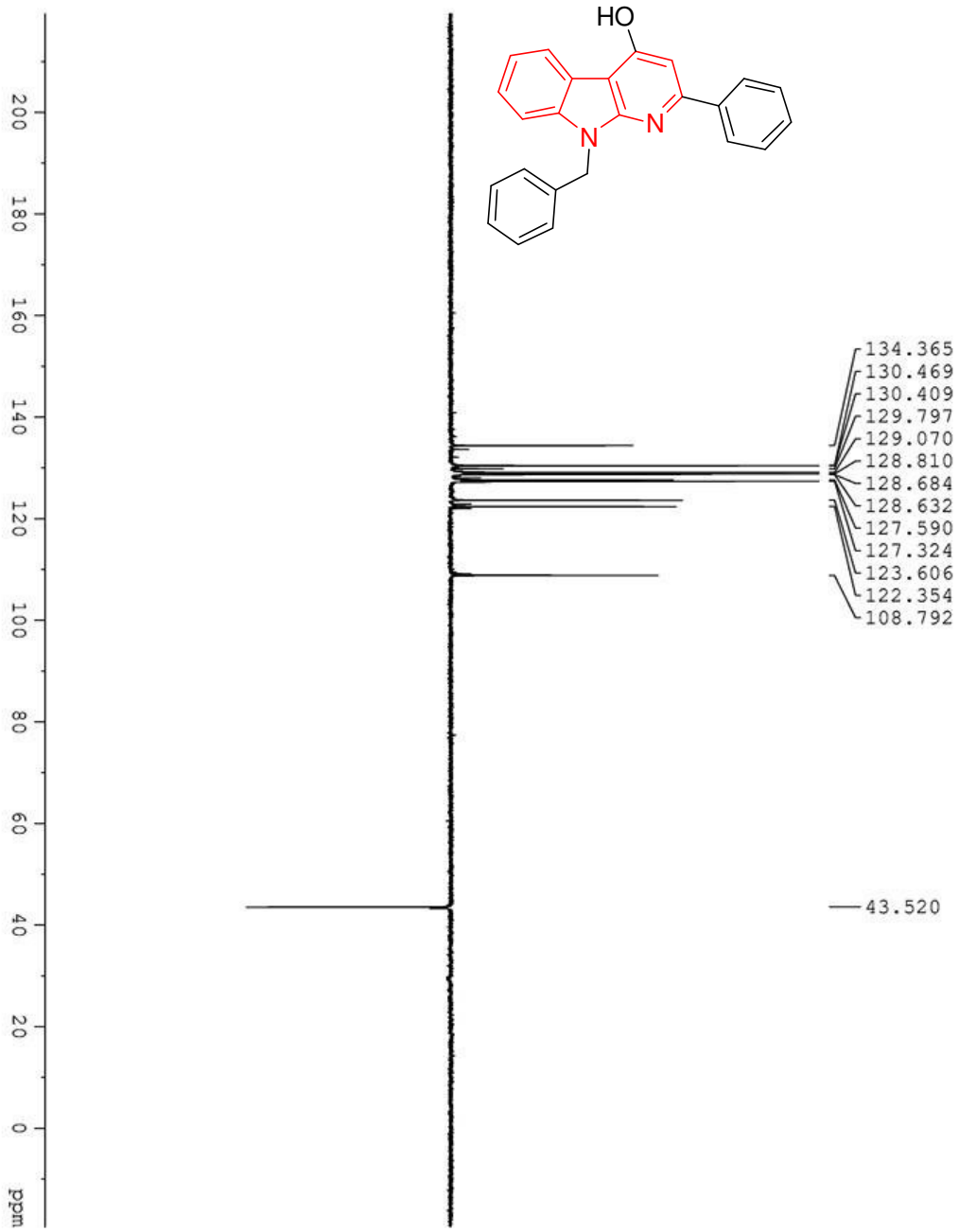
DEPT of 9-benzyl-2-phenyl-9*H*-pyrido[2,3-*b*]indol-4-ol (2j)



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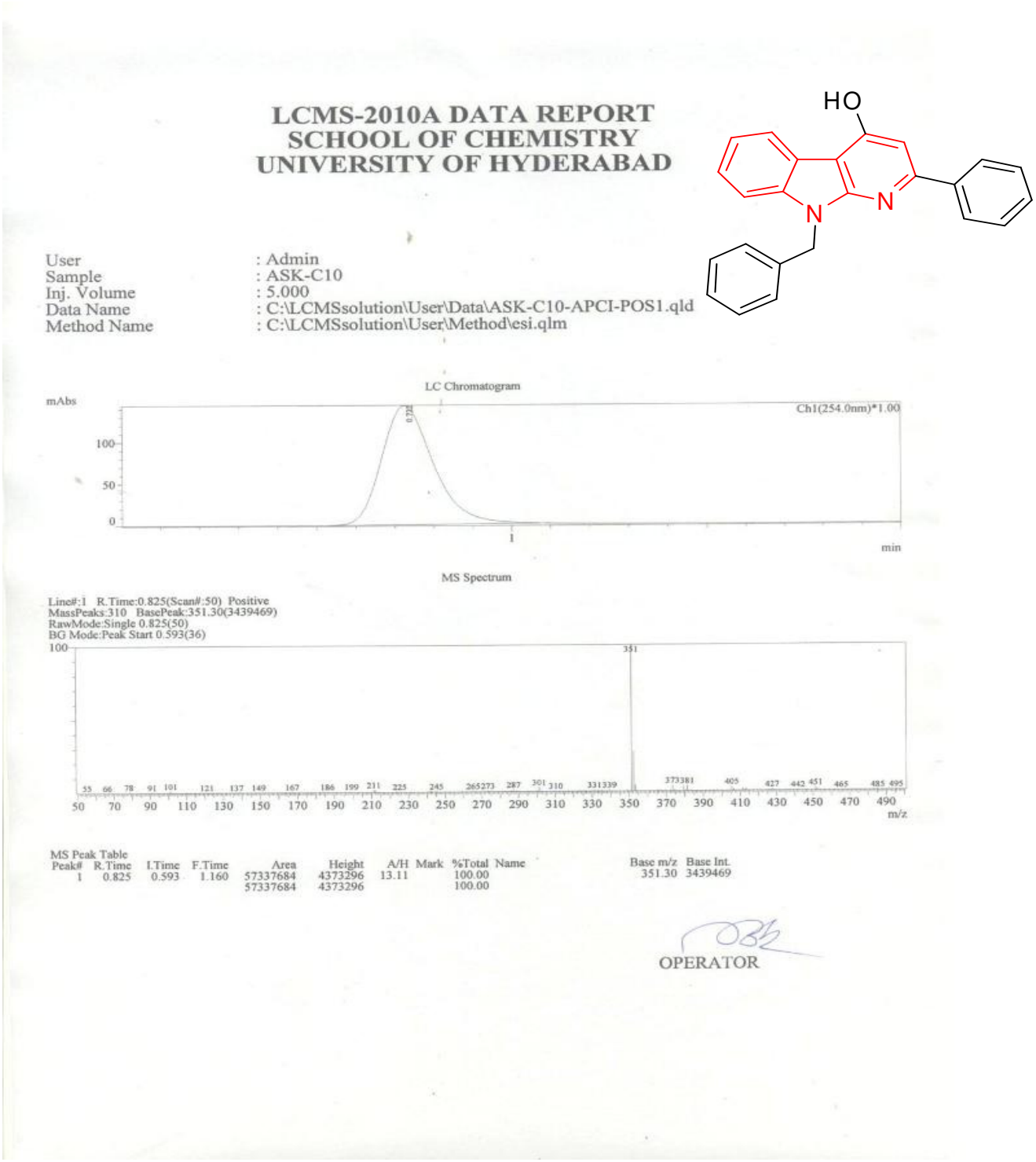
LCMS of 9-benzyl-2-phenyl-9*H*-pyrido[2,3-*b*]indol-4-ol (2j)



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CHN Analysis of 9-benzyl-2-phenyl-9*H*-pyrido[2,3-*b*]indol-4-ol (2j)

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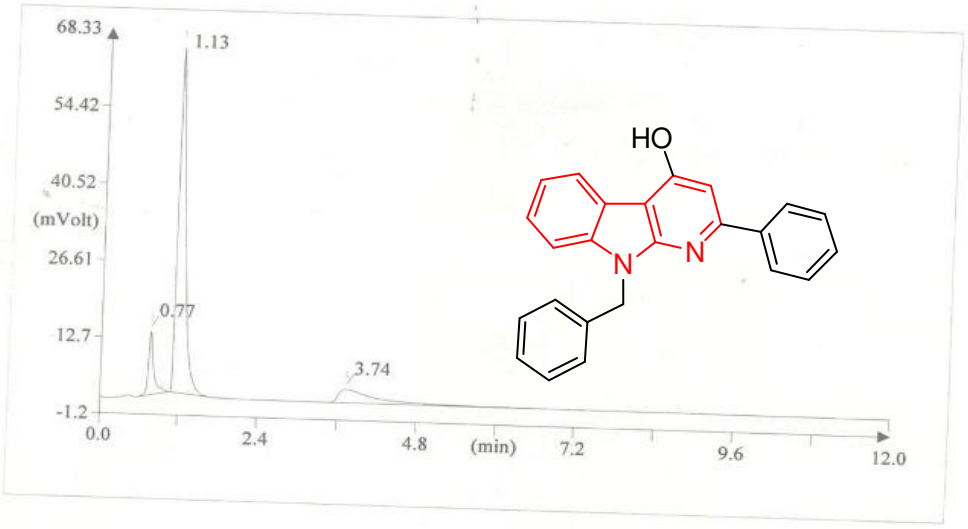
Method filename: E:\Program Files\Thermo Finnigan\Eager 300 for EA1112\DATA\Sys_data_ex
Sample ID: ASK-C10 (# 111) → 2j
Analysis type: UnkNown
Chromatogram filename: UNK-24012012-11.dat
Sample weight: 1.113



Element Name	Element %	Ret. Time
Nitrogen	7. 91	0. 77
Carbon	82. 45	1. 13
Hydrogen	5. 23	3. 74

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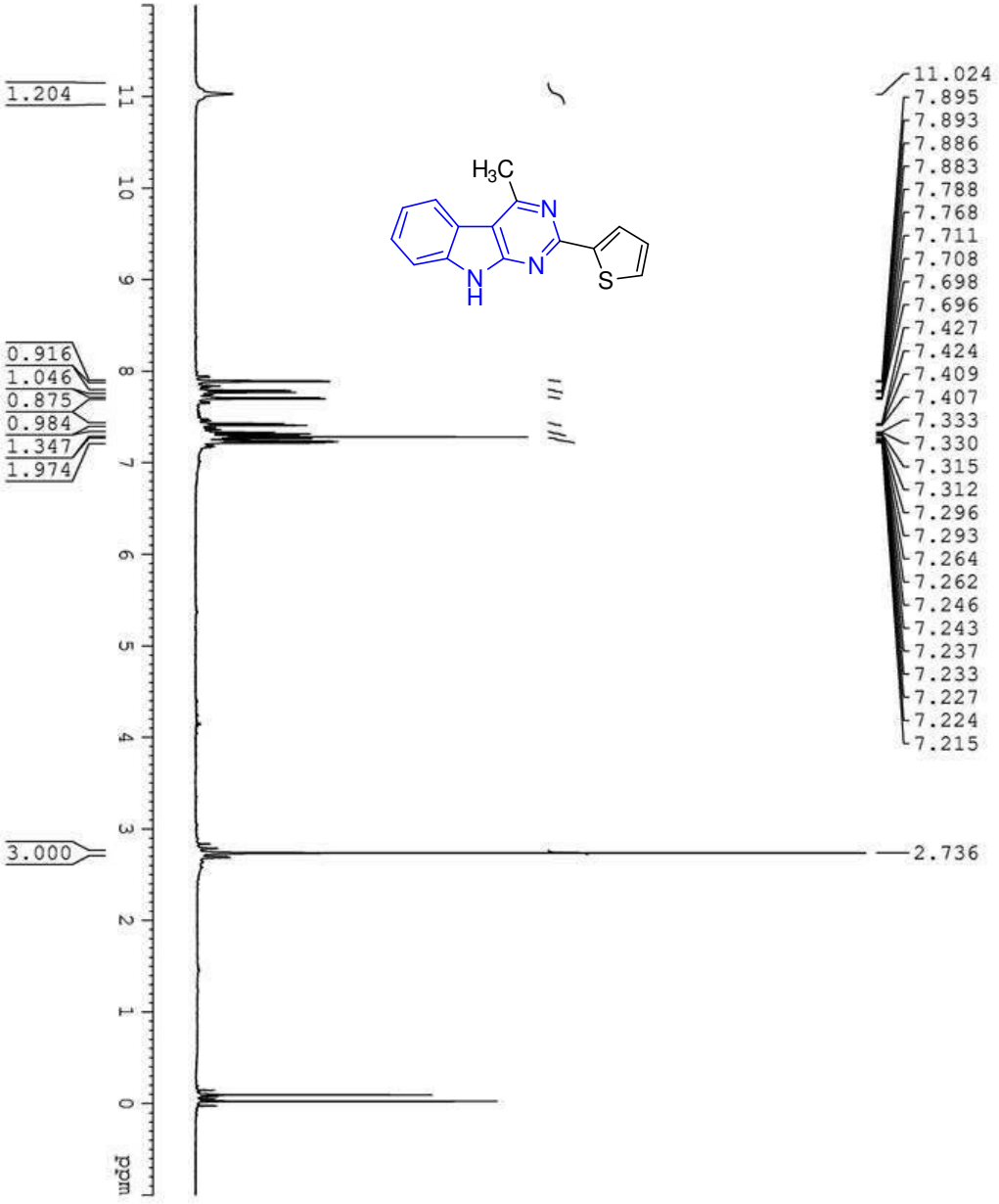
^{1}H NMR of 4-methyl-2-(thiophen-2-yl)-9*H*-pyrimido[4,5-*b*]indole (3a):



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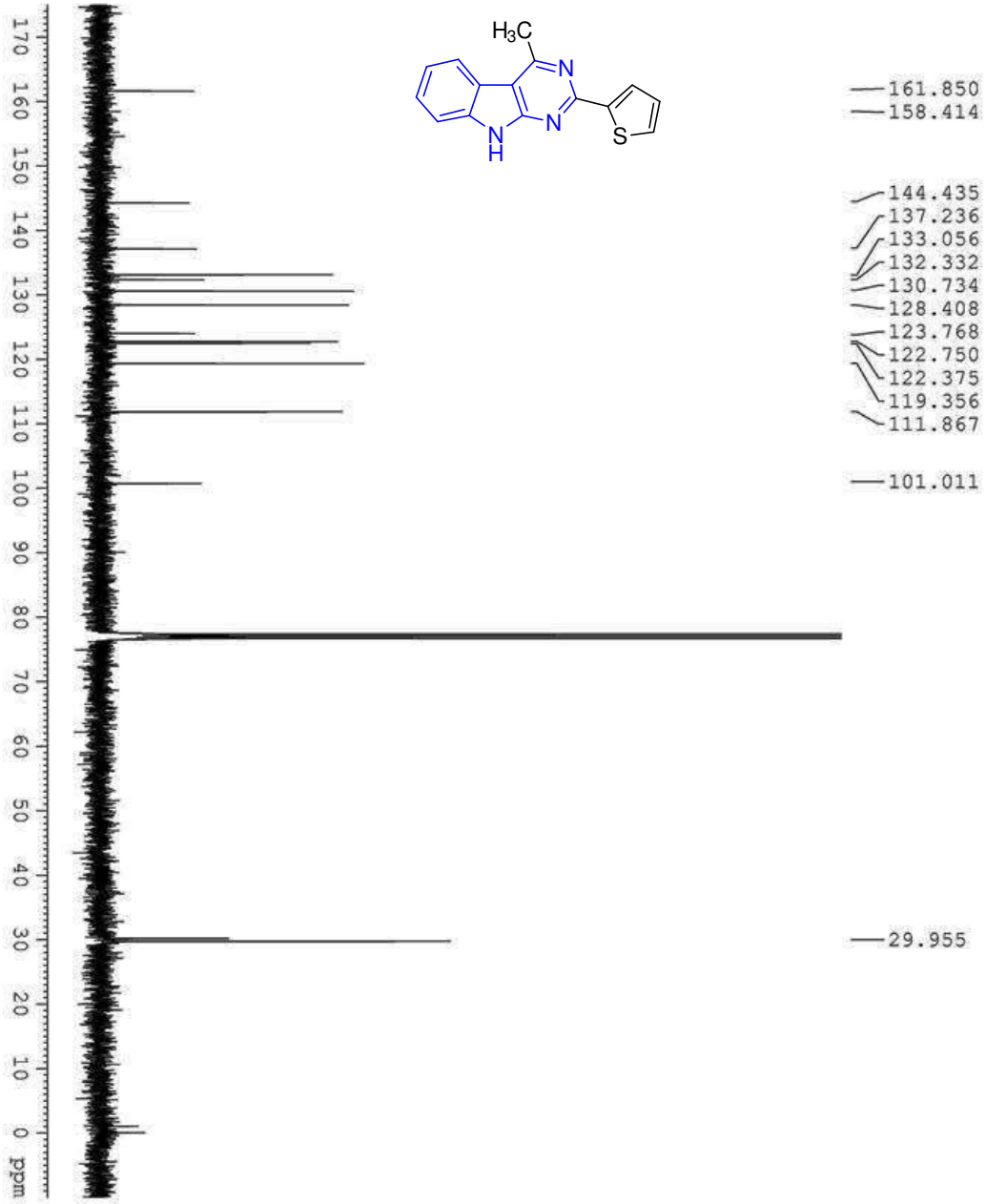
^{13}C NMR of 4-methyl-2-(thiophen-2-yl)-9*H*-pyrimido[4,5-*b*]indole (3a):



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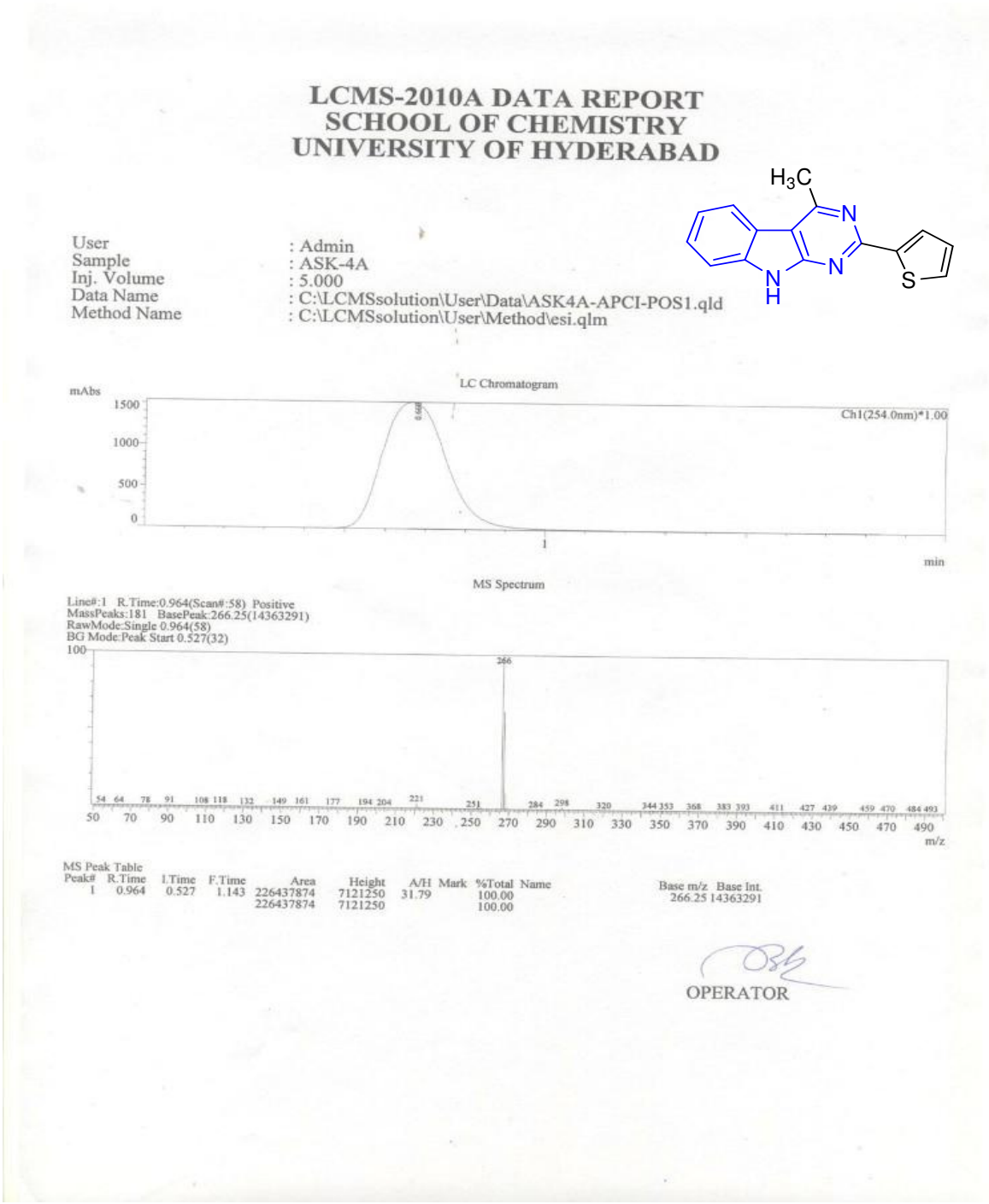
LCMS of 4-methyl-2-(thiophen-2-yl)-9*H*-pyrimido[4,5-*b*]indole (3a):



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CHN Analysis of 4-methyl-2-(thiophen-2-yl)-9*H*-pyrimido[4,5-*b*]indole (3a):

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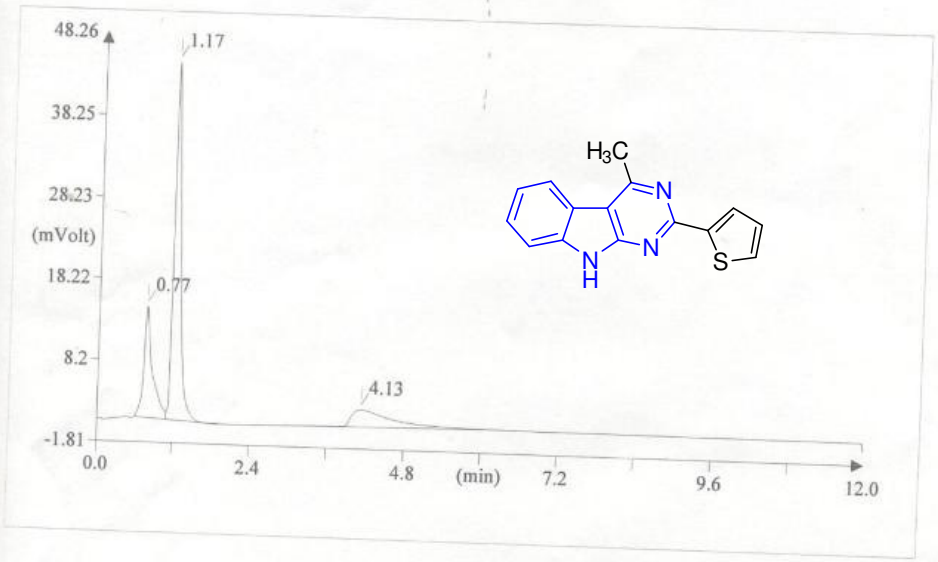
Method filename: E:\Program Files\Thermo Finnigan\Eager 300 for EA1112\DATA\Sys_data_ex
Sample ID: ASK-4A (# 105)
Analysis type: UnkNown
Chromatogram filename: UNK-24012012-5.dat
Sample weight: .988



Element Name	Element %	Ret. Time
Nitrogen	15. 76	0. 77
Carbon	67. 81	1. 17
Hydrogen	4. 23	4. 13

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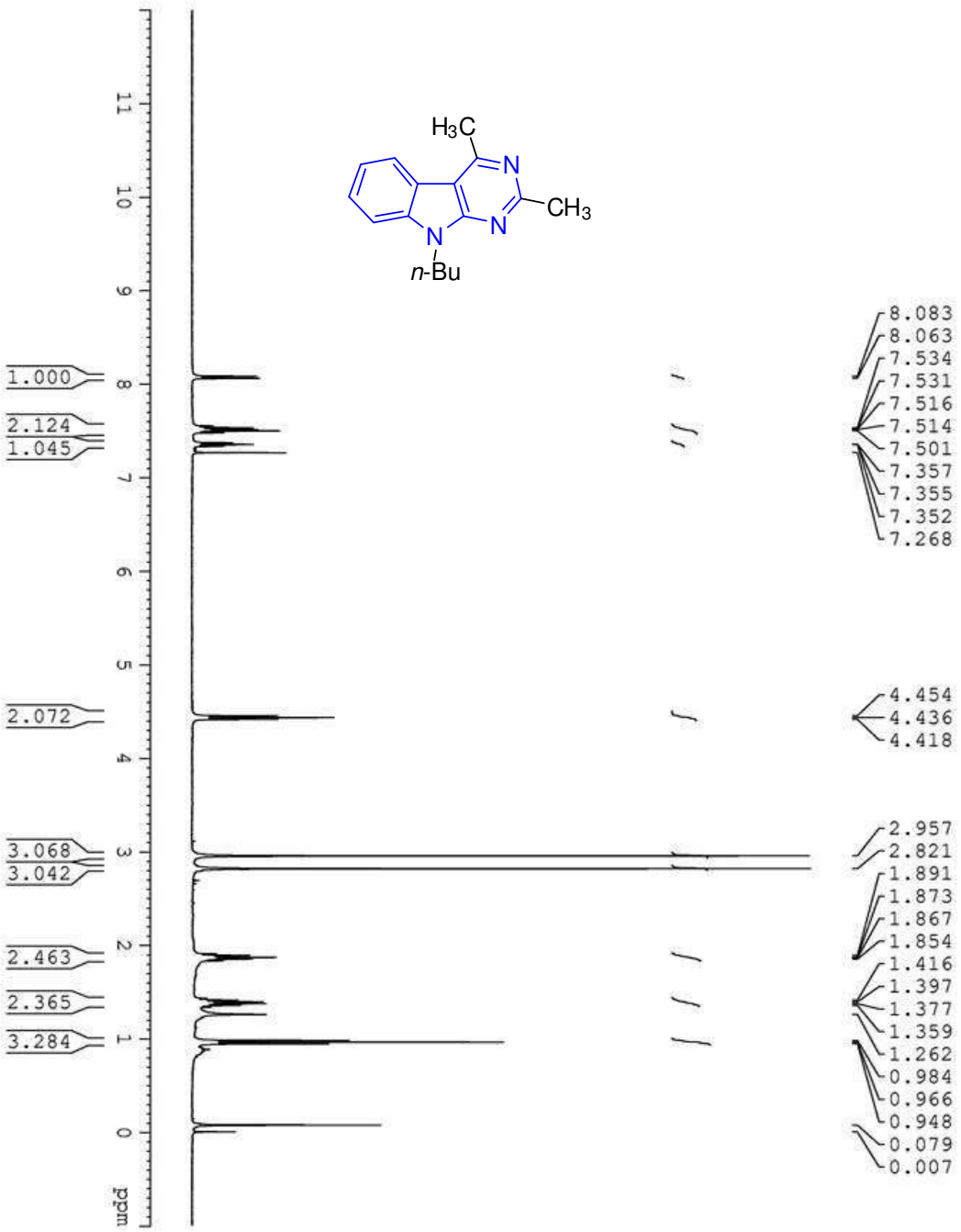
^{1}H NMR of 9-butyl-2,4-dimethyl-9*H*-pyrimido[4,5-*b*]indole (3b):



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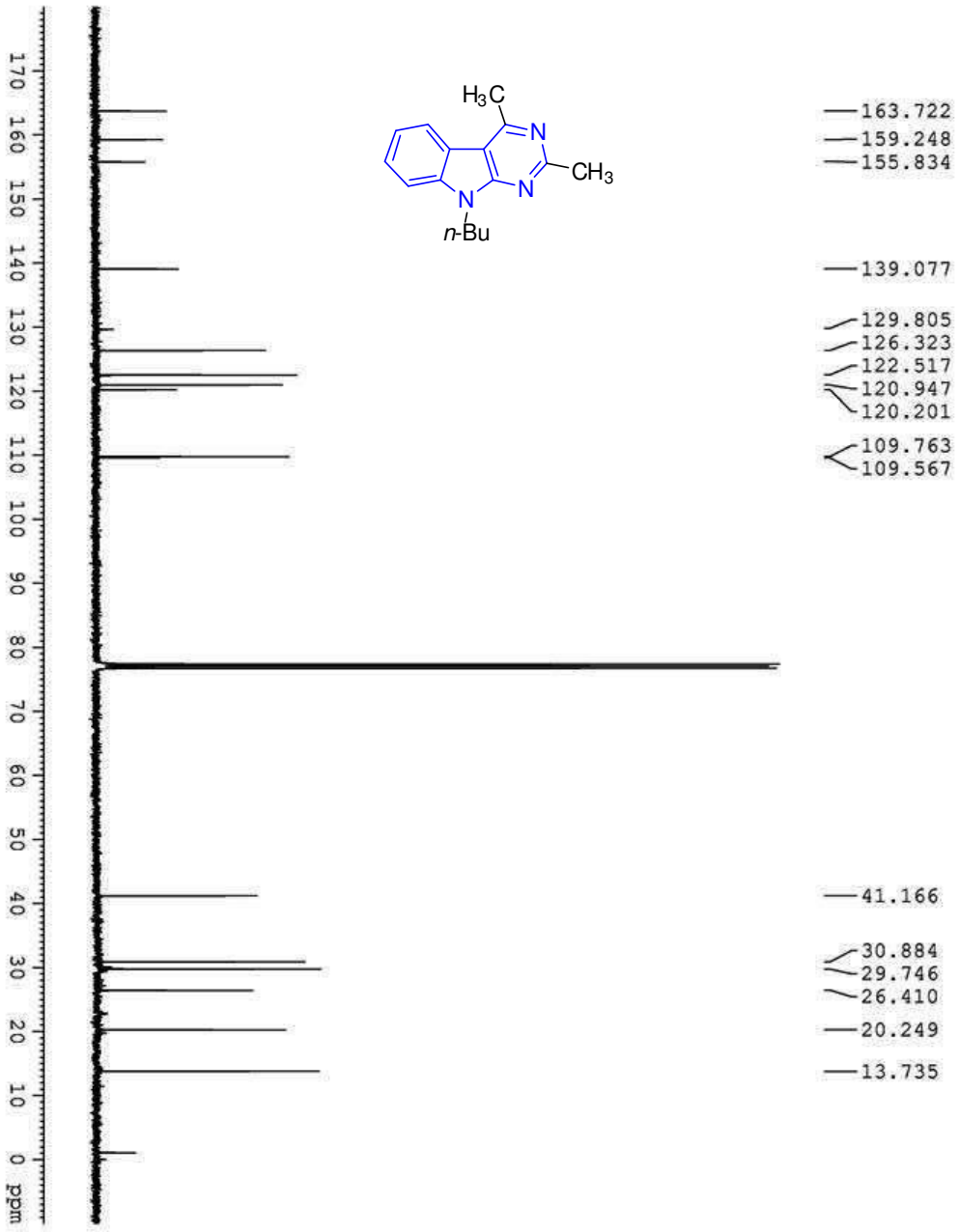
^{13}C NMR of 9-butyl-2,4-dimethyl-9*H*-pyrimido[4,5-*b*]indole (3b):



LCMS of 9-butyl-2,4-dimethyl-9*H*-pyrimido[4,5-*b*]indole (3b):

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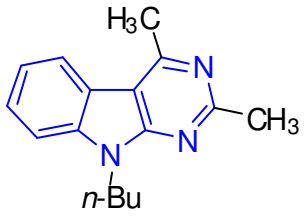
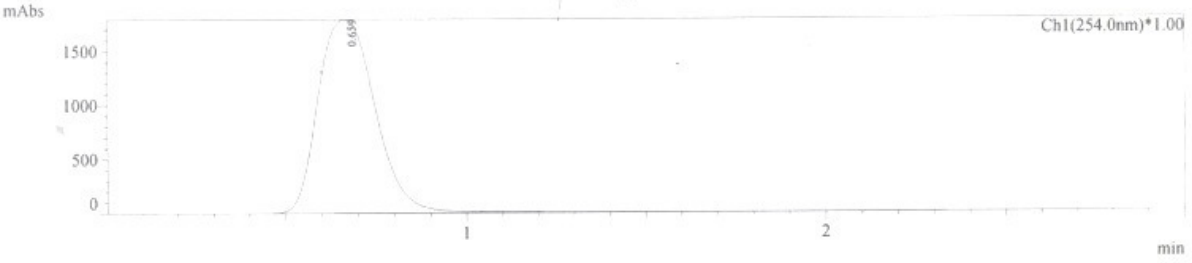


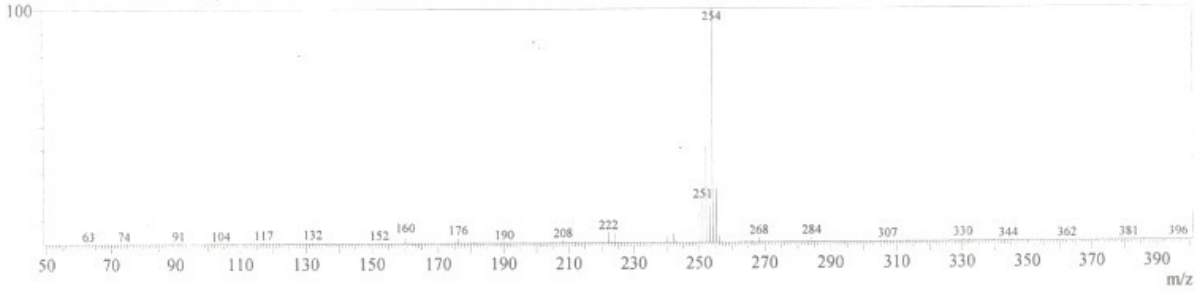
User : Admin
Sample : ASK-P2
Inj. Volume : 5.000
Data Name : C:\LCMSsolution\User\Data\ASK-P2-APCI-POS1.qld
Method Name : C:\LCMSsolution\User\Method\esi.qlm

LC Chromatogram



MS Spectrum

Line#:1 R.Time:0.768(Scan#:46) Positive
MassPeaks:309 BasePeak:254.15(10330512)
RawMode:Single 0.768(46)
BG Mode:Peak Start 0.527(32)



MS Peak Table

Peak#	R.Time	I.Time	F.Time	Area	Height	A/H	Mark	%Total	Name	Base m/z	Base Int.
1	0.768	0.527	1.043	498070894	33091159	15.05		100.00		254.15	10330512
				498070894	33091159			100.00			

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CHN Analysis of 9-butyl-2,4-dimethyl-9*H*-pyrimido[4,5-*b*]indole (3b):

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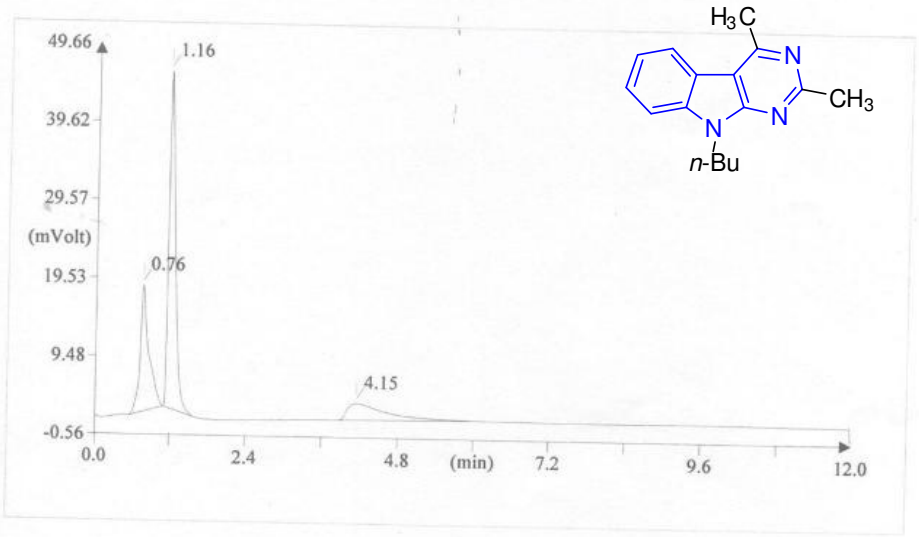
Method filename:	E:\Program Files\Thermo Finnigan\Eager 300 for EA1112\DATA\Sys_data_ex
Sample ID:	ASK-P2 (# 183)
Analysis type:	UnkNown
Chromatogram filename:	UNK-15092011-23.dat
Sample weight:	1.103



Element Name	Element %	Ret. Time
Nitrogen	16. 51	0. 76
Carbon	75. 96	1. 16
Hydrogen	7. 52	4. 15

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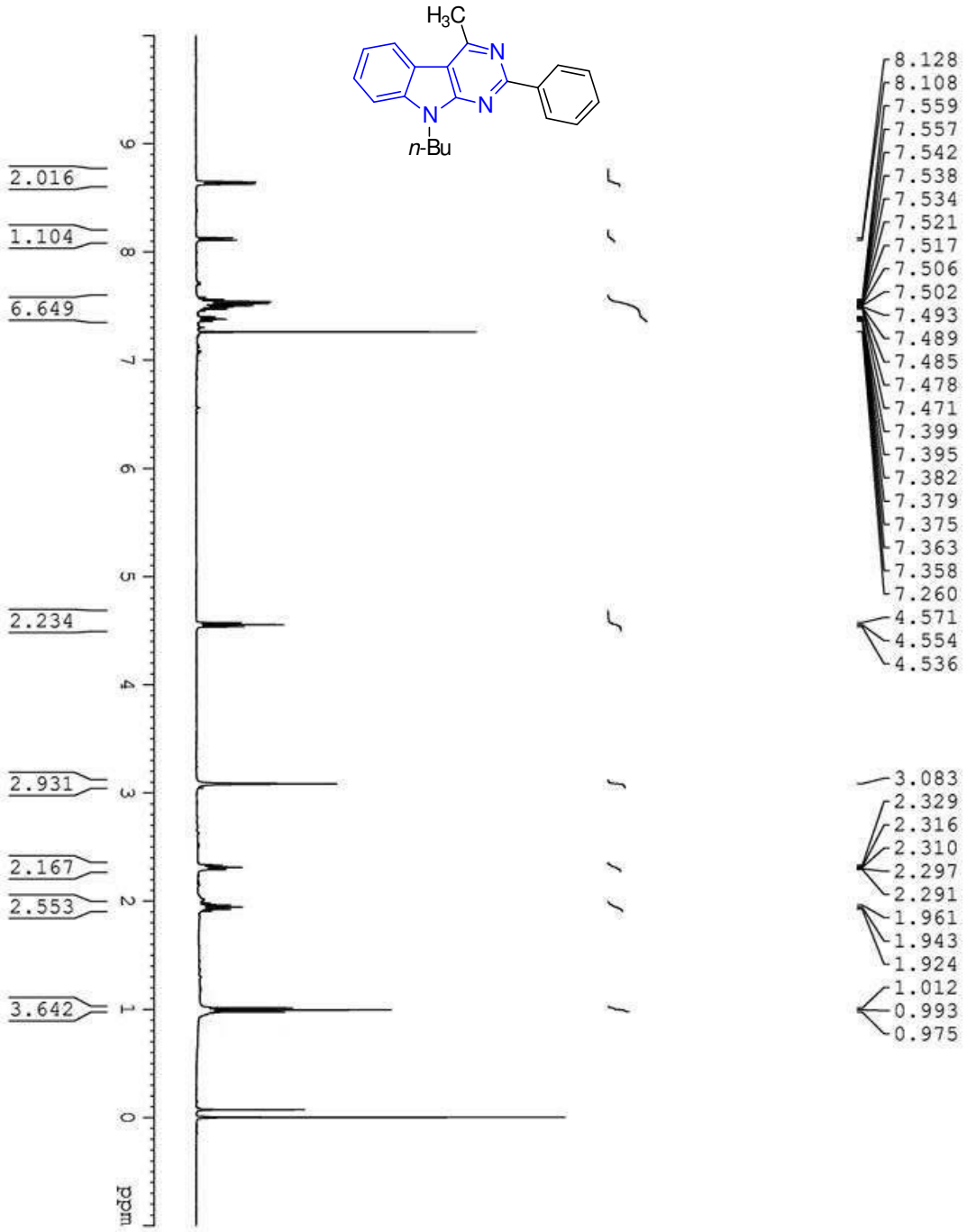
^{1}H NMR of 9-butyl-4-methyl-2-phenyl-9*H*-pyrimido[4,5-*b*]indole (3c)



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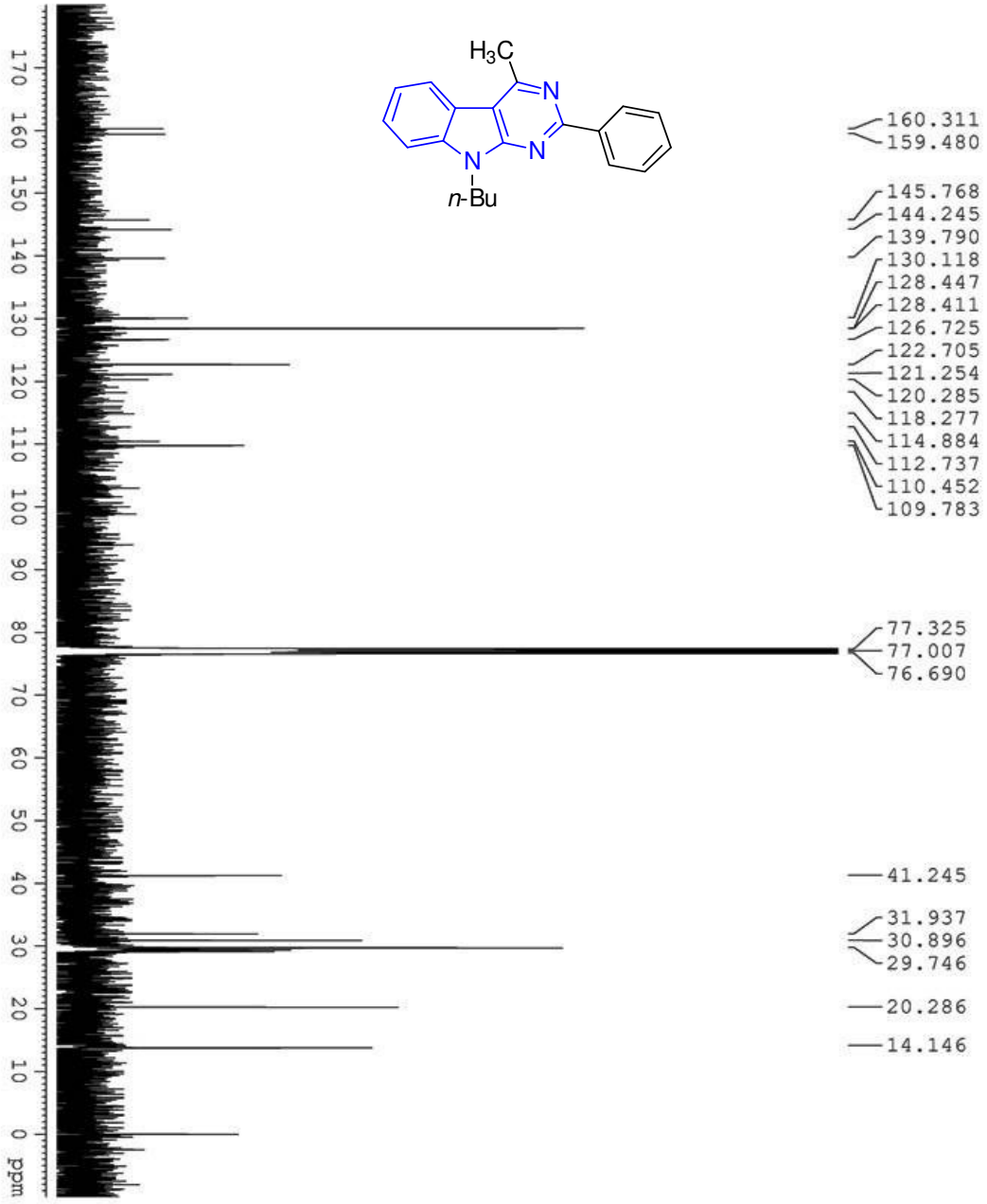
^{13}C NMR of 9-butyl-4-methyl-2-phenyl-9*H*-pyrimido[4,5-*b*]indole (3c)



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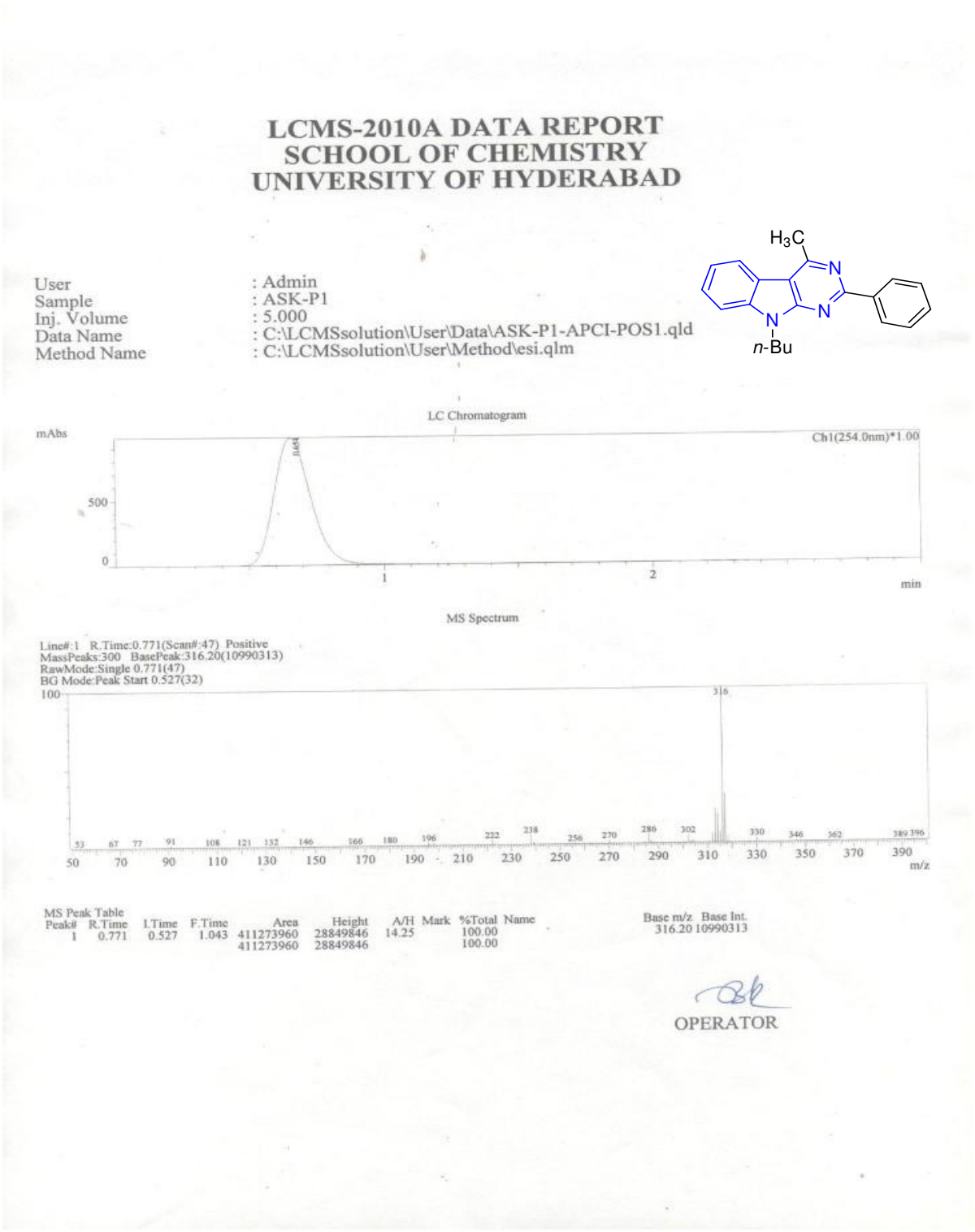
LCMS of 9-butyl-4-methyl-2-phenyl-9*H*-pyrimido[4,5-*b*]indole (3c)



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CHN Analysis of 9-butyl-4-methyl-2-phenyl-9*H*-pyrimido[4,5-*b*]indole (3c)

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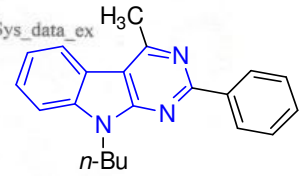
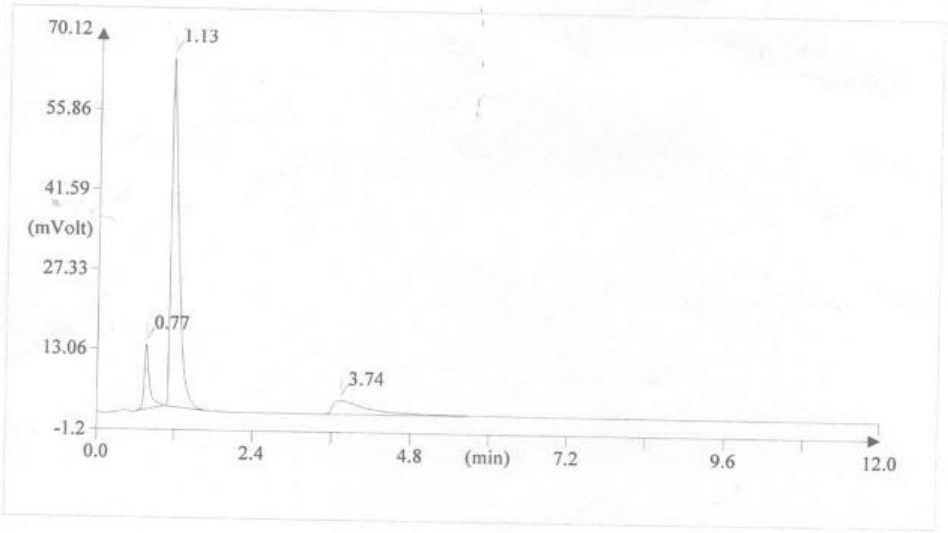
Method filename:	E:\Program Files\Thermo Finnigan\Eager 300 for EA1112\DATA\Sys_data_ex
Sample ID:	ASK-P1 (# 169)
Analysis type:	UnkNown
Chromatogram filename:	UNK-15092011-9.dat
Sample weight:	1265





Element Name	Element %	Ret. Time
Nitrogen	13. 45	0. 77
Carbon	79. 85	1. 13
Hydrogen	6. 81	3. 74

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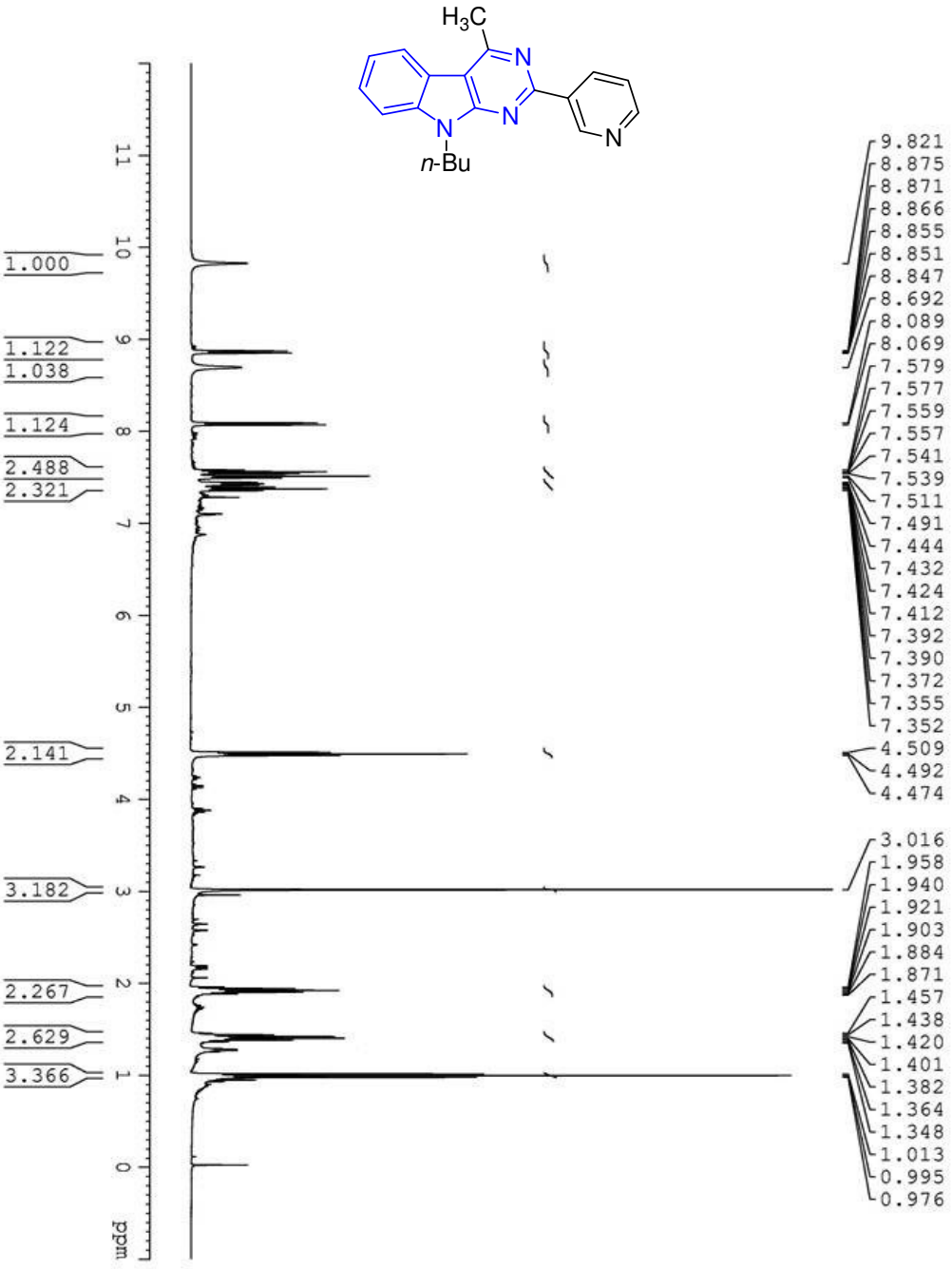
^{1}H NMR of 9-butyl-4-methyl-2-(pyridin-3-yl)-9*H*-pyrimido[4,5-*b*]indole (3d):



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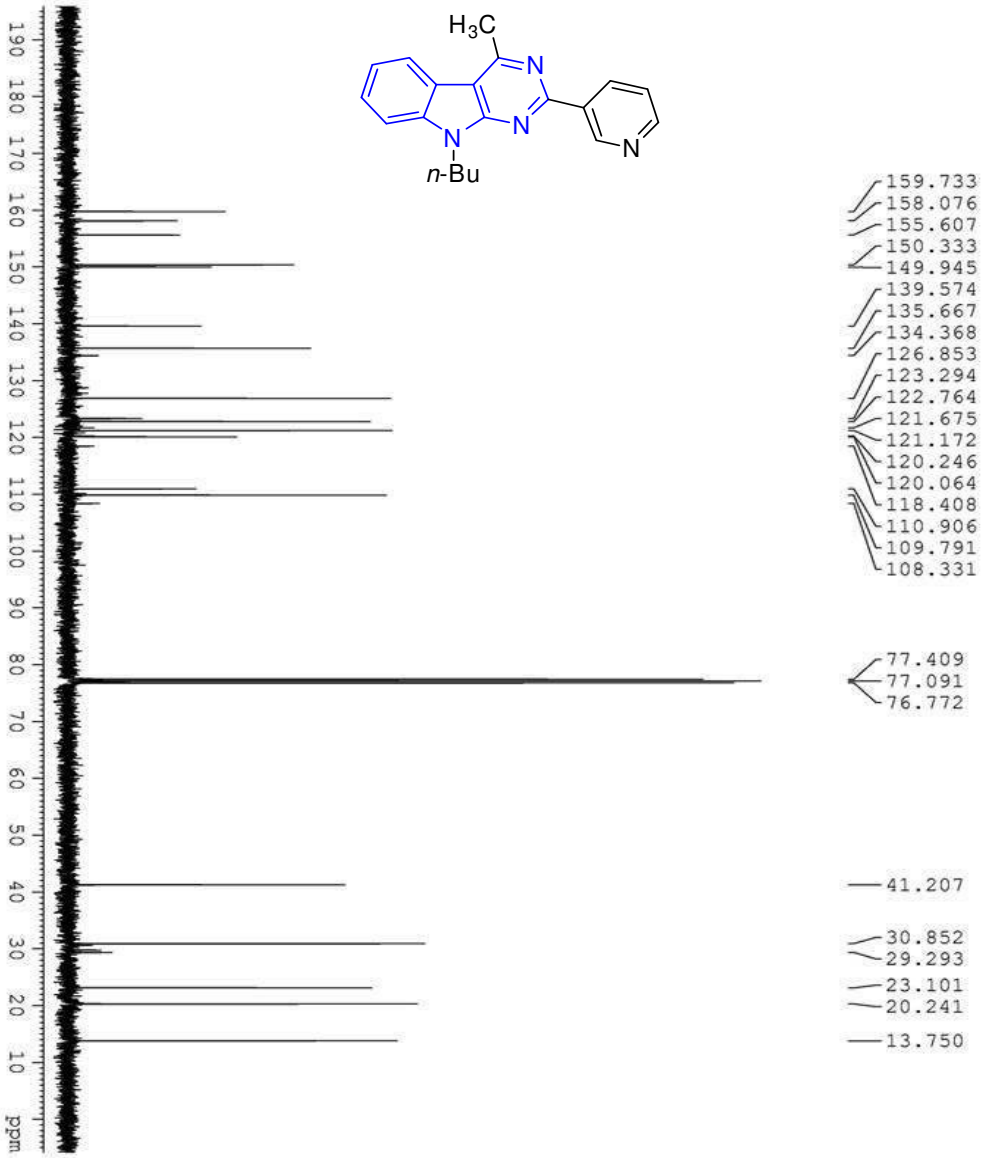
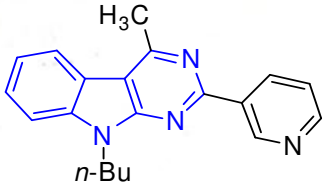
^{13}C NMR of 9-butyl-4-methyl-2-(pyridin-3-yl)-9*H*-pyrimido[4,5-*b*]indole (3d):



LCMS of 9-butyl-4-methyl-2-(pyridin-3-yl)-9*H*-pyrimido[4,5-*b*]indole (3d):



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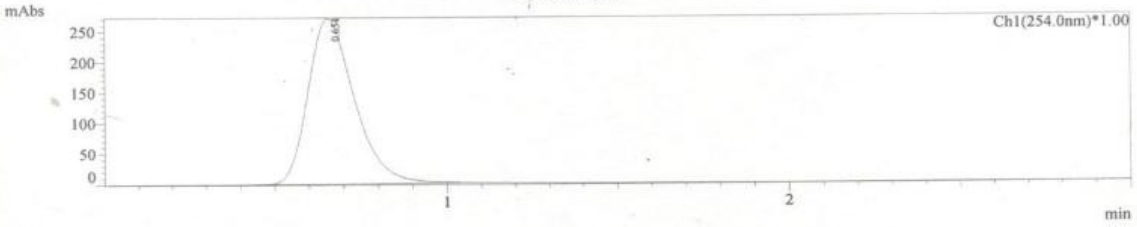
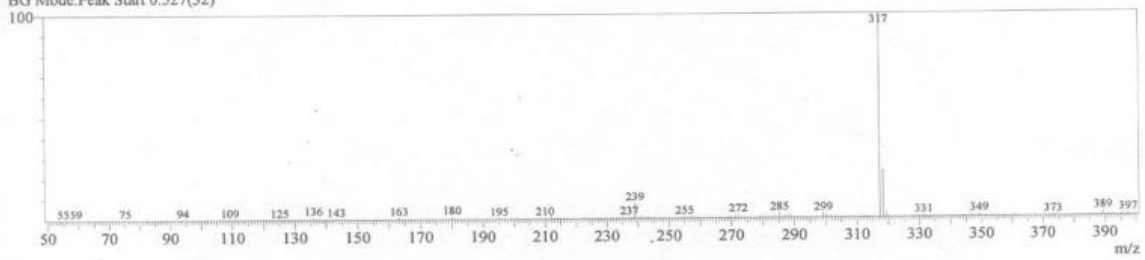
User	: Admin
Sample	: ASK-P14
Inj. Volume	: 5.000
Data Name	: C:\LCMSsolution\User\Data\ASK-P14-APCI-POS1.qld
Method Name	: C:\LCMSsolution\User\Method\esi.qlm

LC Chromatogram



MS Spectrum

Line#:1 R.Time:0.782(Scan#:47) Positive
MassPeaks:295 BasePeak:317.25(3738704)
RawMode:Single 0.782(47)
BG Mode:Peak Start 0.527(32)



MS Peak Table

Peak#	R.Time	I.Time	F.Time	Area	Height	A/H	Mark	%Total	Name
1	0.782	0.527	1.043	113002504	10009999	11.28		100.00	
				113002504	10009999			100.00	

Base m/z	Base Int.
317.25	3738704

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CHN Analysis of 9-butyl-4-methyl-2-(pyridin-3-yl)-9*H*-pyrimido[4,5-*b*]indole (3d):

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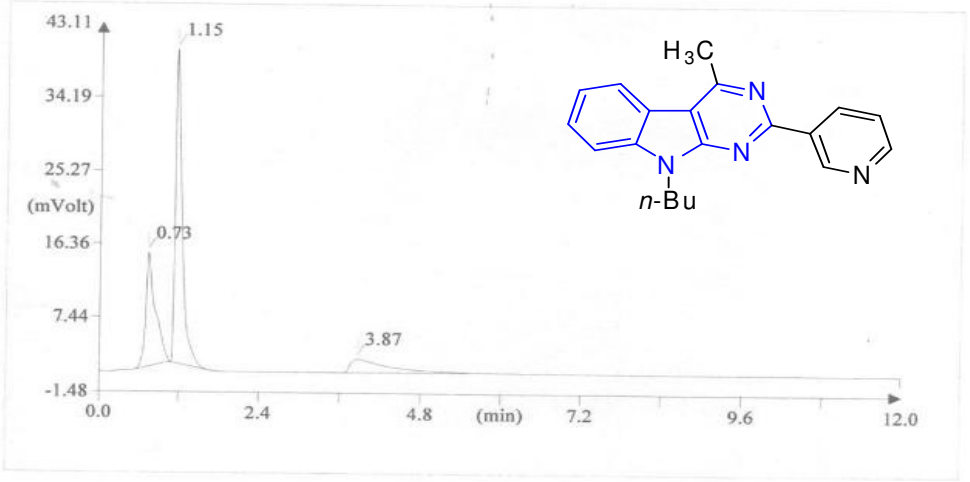
Method filename:	E:\Program Files\Thermo Finnigan\Eager 300 for EA1112\DATA\Sys_data_ex
Sample ID:	ASK-P14 (# 170)
Analysis type:	UnkNown
Chromatogram filename:	UNK-15092011-10.dat
Sample weight:	1.003



Element Name	Element %	Ret. Time
Nitrogen	17. 65	0. 73
Carbon	75. 85	1. 15
Hydrogen	6. 31	3. 87

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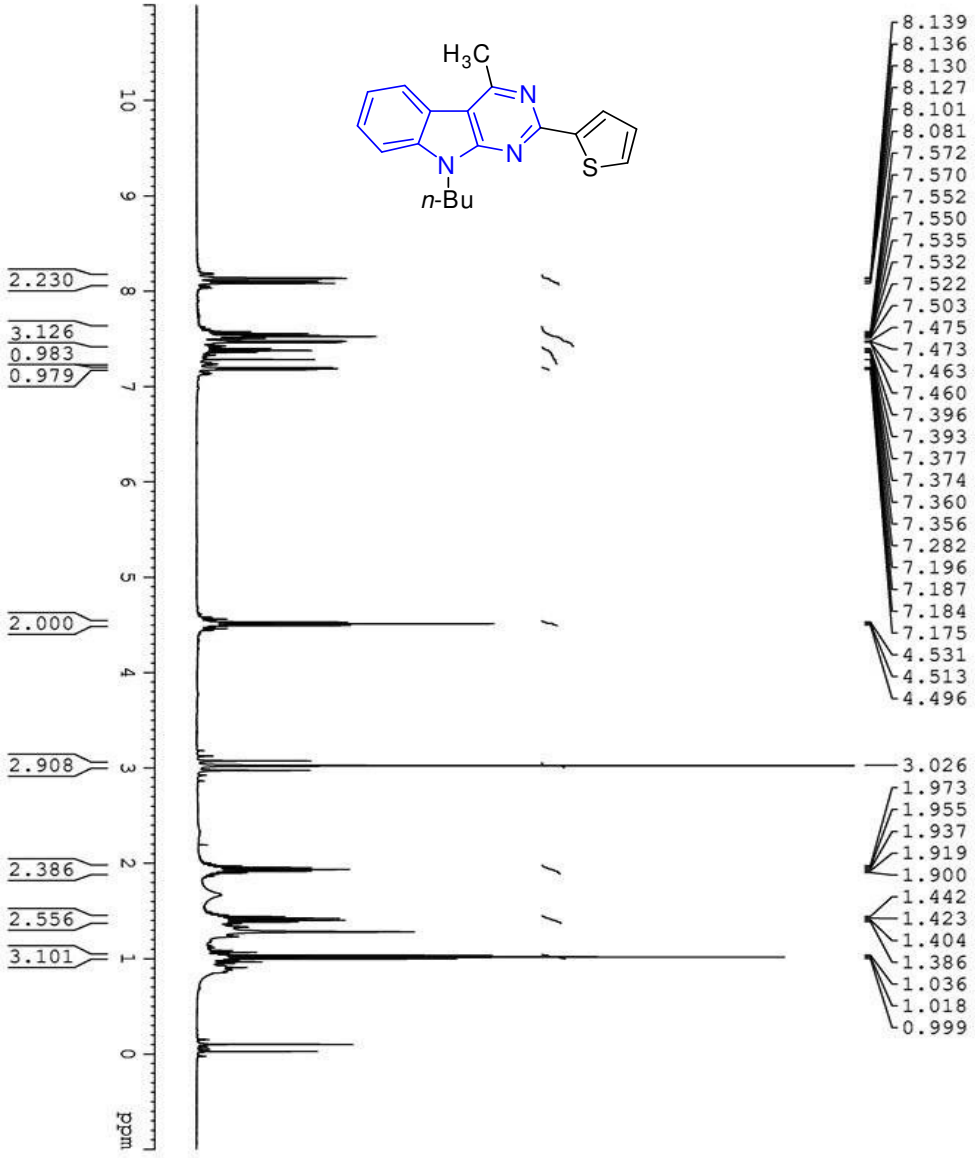
^{1}H NMR of 9-butyl-4-methyl-2-(thiophen-2-yl)-9H-pyrimido[4,5-*b*]indole (3e):



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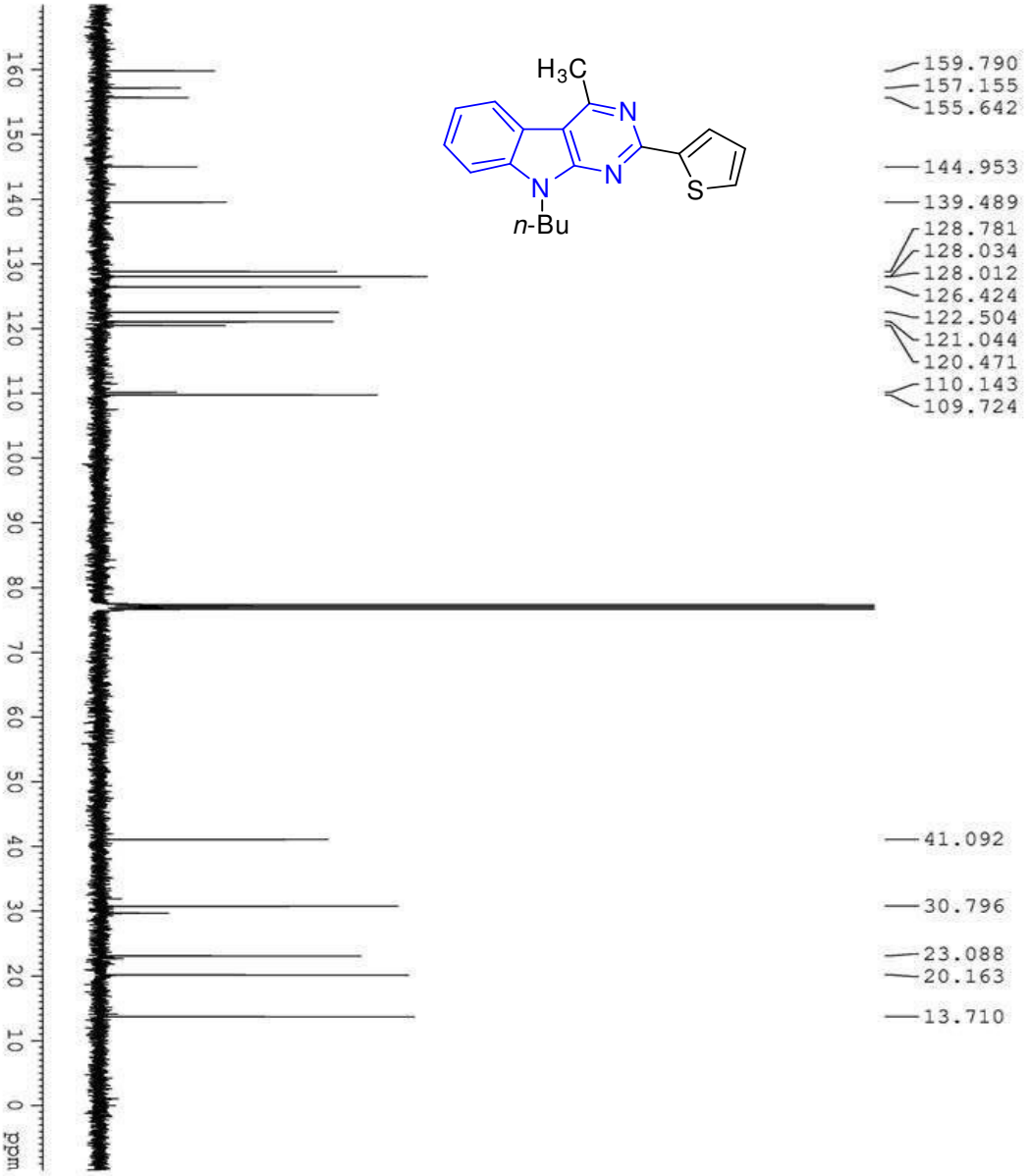
^{13}C NMR of 9-butyl-4-methyl-2-(thiophen-2-yl)-9H-pyrimido[4,5-*b*]indole (3e):



LCMS of 9-butyl-4-methyl-2-(thiophen-2-yl)-9H-pyrimido[4,5-*b*]indole (3e):

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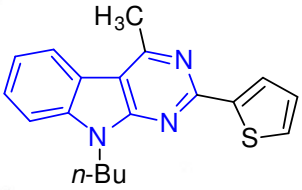
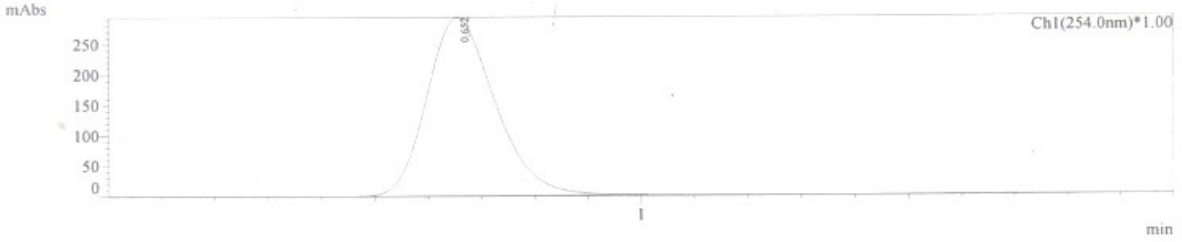


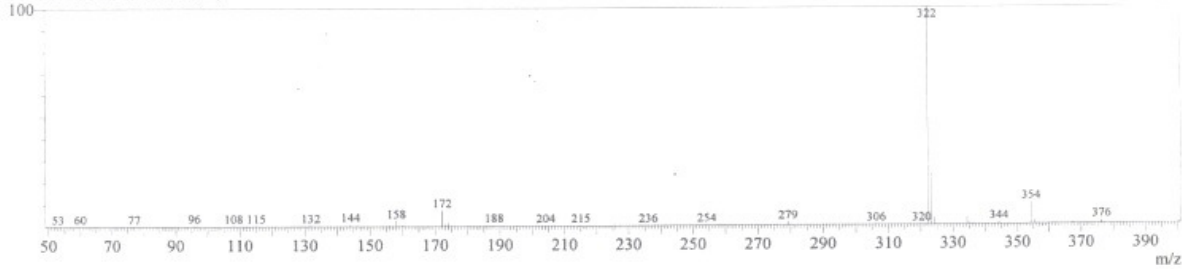
User : Admin
Sample : ASK-P8
Inj. Volume : 5.000
Data Name : C:\LCMSsolution\User\Data\ASK-P8-APCI-POS1.qld
Method Name : C:\LCMSsolution\User\Method\esi.qlm

LC Chromatogram



MS Spectrum

Line#:1 R.Time:0.792(Scan#:48) Positive
MassPeaks:213 BasePeak:322.25(8637410)
RawMode:Single 0.792(48)
BG Mode:Peak Start 0.560(34)



MS Peak Table

Peak#	R.Time	I.Time	F.Time	Area	Height	A/H	Mark	%Total	Name	Base m/z	Base Int.
1	0.792	0.560	1.043	134769268	9596863	14.04		100.00		322.25	8637410
				134769268	9596863			100.00			

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CHN Analysis of 9-butyl-4-methyl-2-(thiophen-2-yl)-9*H*-pyrimido[4,5-*b*]indole (3e):

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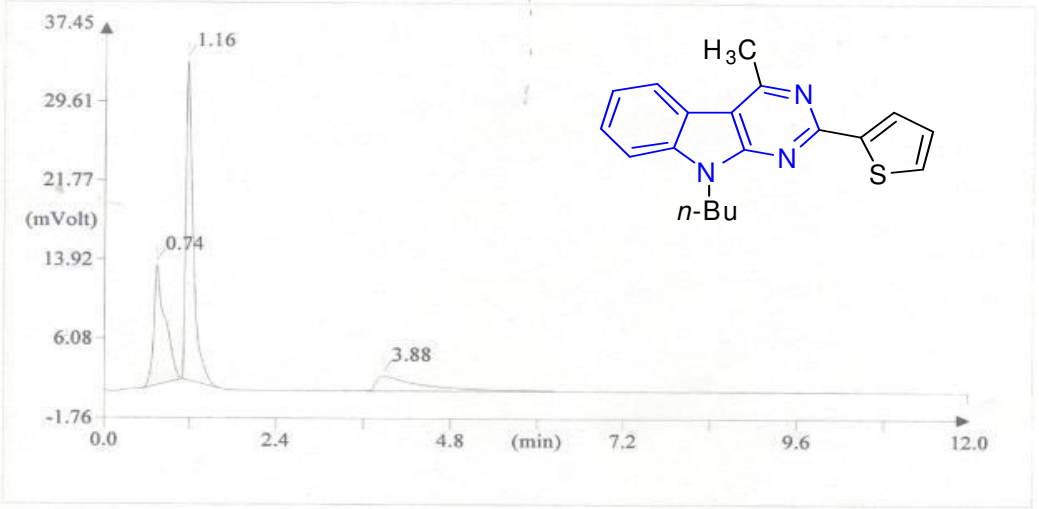
Method filename:	E:\Program Files\Thermo Finnigan\Eager 300 for EA1112\DATA\Sys_data_ex
Sample ID:	ASK-P8 (# 179)
Analysis type:	UnkNown
Chromatogram filename:	UNK-15092011-19.dat
Sample weight:	.997



Element Name	Element %	Ret. Time
Nitrogen	13. 18	0. 74
Carbon	70. 85	1. 16
Hydrogen	5. 91	3. 88

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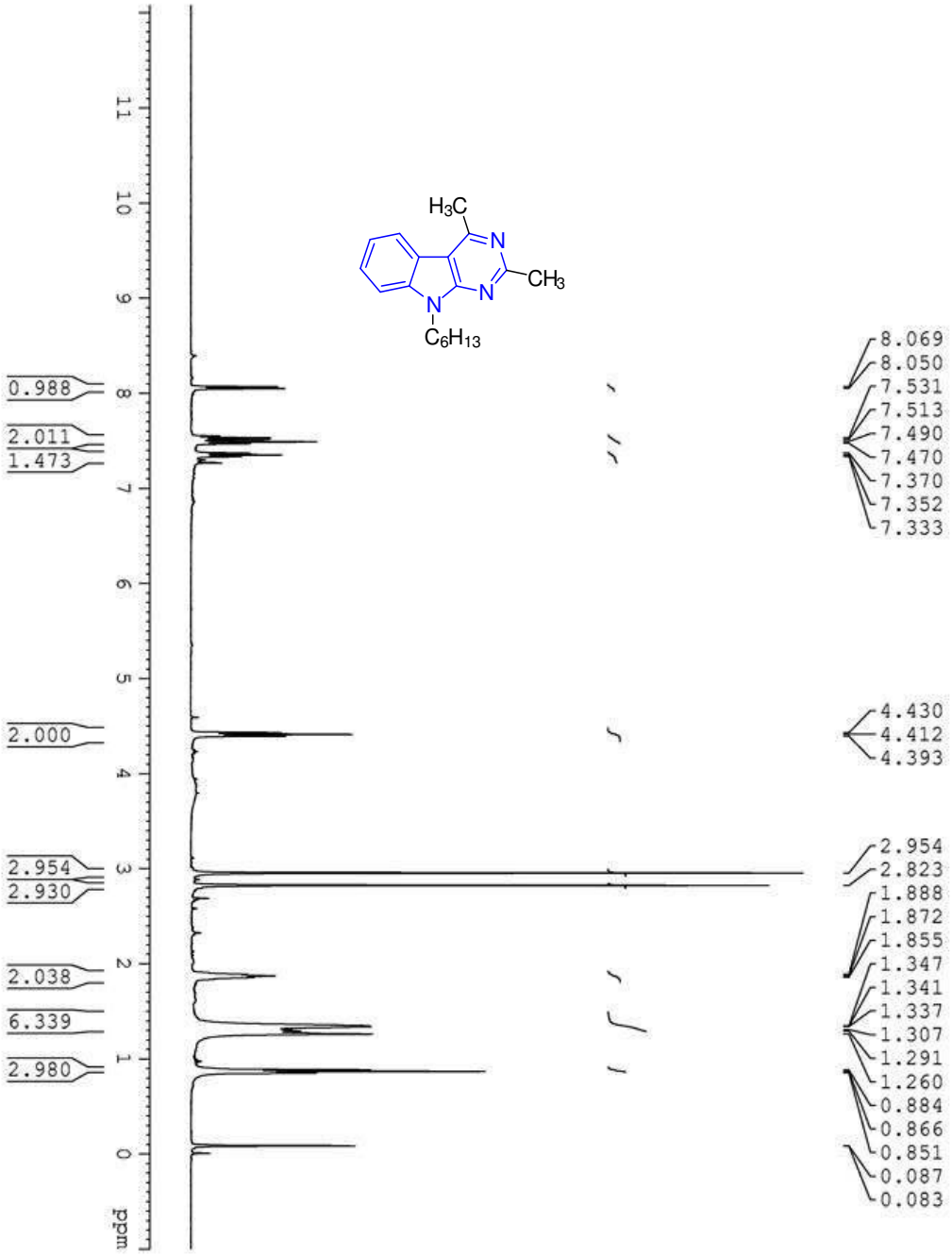
^{1}H NMR of 9-hexyl-2,4-dimethyl-9*H*-pyrimido[4,5-*b*]indole (3f)



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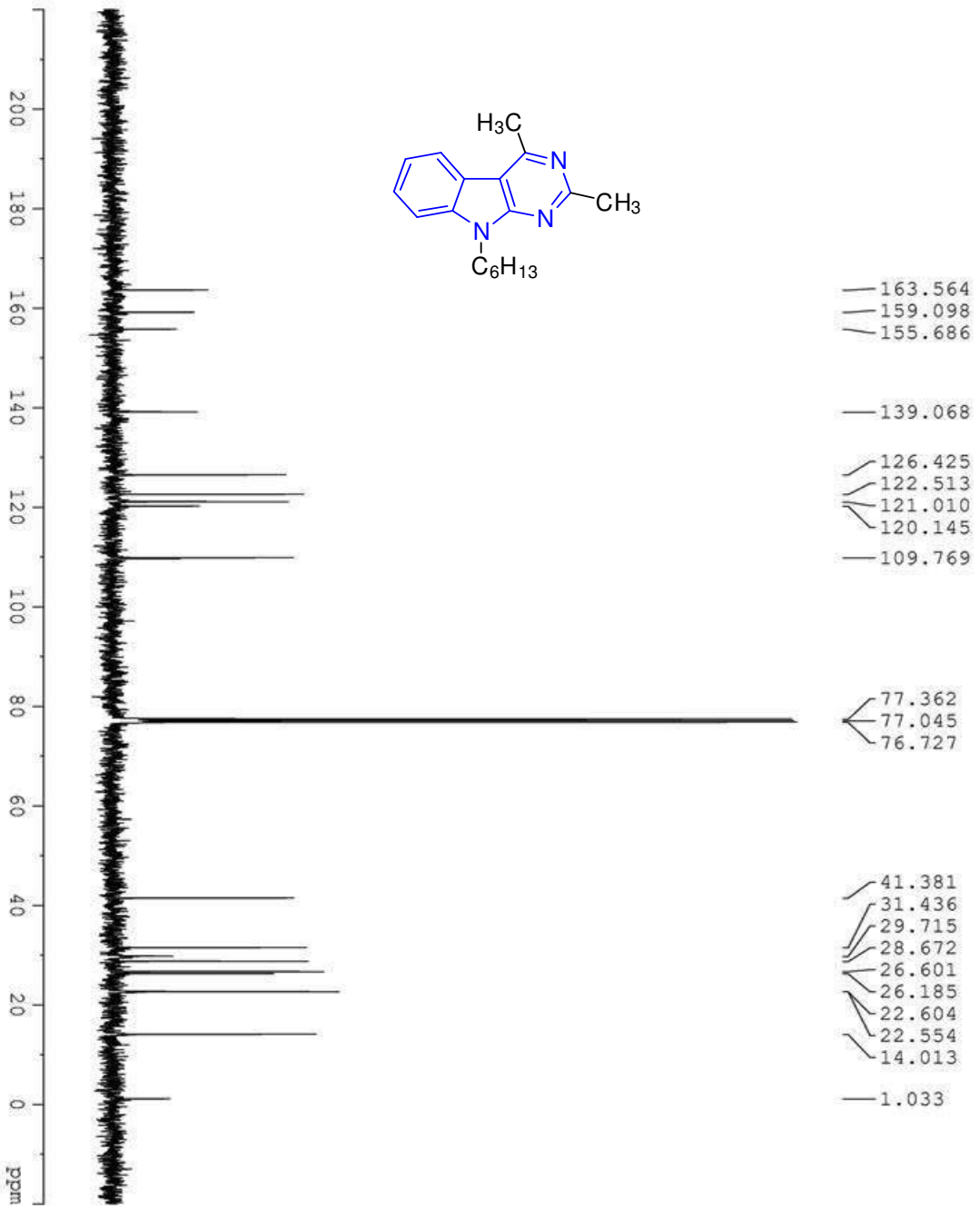
^{13}C NMR of 9-hexyl-2,4-dimethyl-9*H*-pyrimido[4,5-*b*]indole (3f)



LCMS of 9-hexyl-2,4-dimethyl-9*H*-pyrimido[4,5-*b*]indole (3f)

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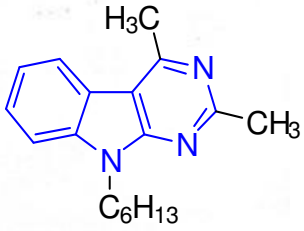
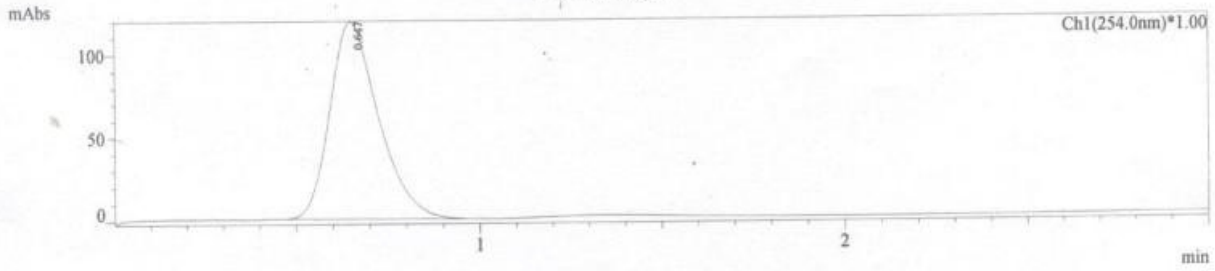


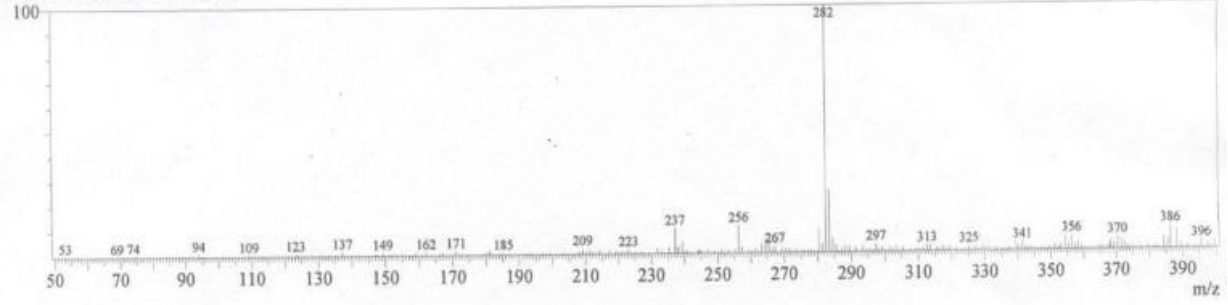
User	: Admin
Sample	: ASK-P12
Inj. Volume	: 5.000
Data Name	: C:\LCMSsolution\User\Data\ASK-P12-APCI-POS1.qld
Method Name	: C:\LCMSsolution\User\Method\esi.qlm

LC Chromatogram



MS Spectrum

Line#:1 R.Time:0.775(Scan#:47) Positive
MassPeaks:295 BasePeak:282.40(481570)
RawMode:Single 0.775(47)
BG Mode:Peak Start 0.527(32)



MS Peak Table

Peak#	R.Time	I.Time	F.Time	Area	Height	A/H	Mark	%Total	Name	Base m/z	Base Int.
1	0.775	0.527	1.043	51135640	4196187	12.18		100.00		282.40	481570
				51135640	4196187			100.00			

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CHN Analysis of 9-hexyl-2,4-dimethyl-9*H*-pyrimido[4,5-*b*]indole (3f)

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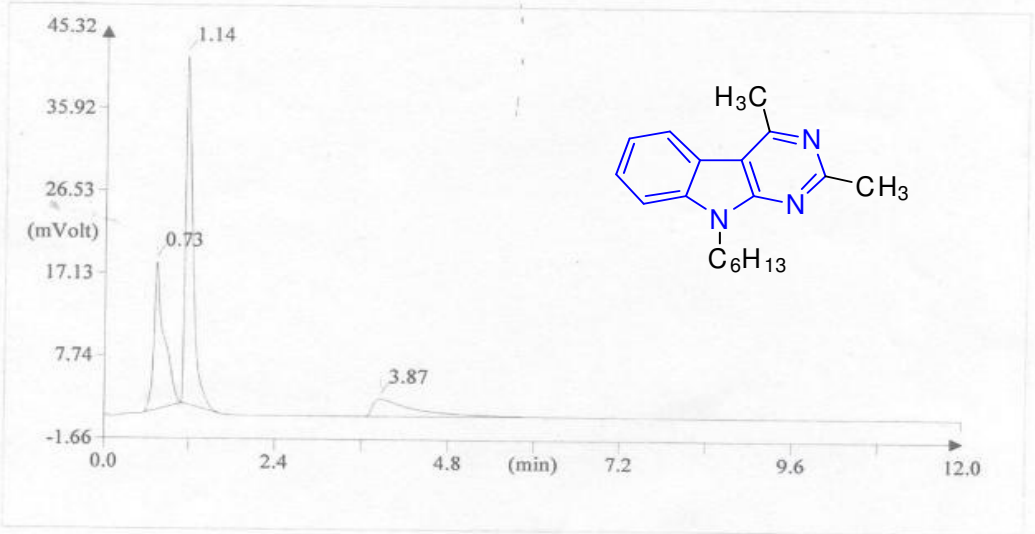
Method filename:	E:\Program Files\Thermo Finnigan\Eager 300 for EA1112\DATA\Sys_data_ex
Sample ID:	ASK-P12 (# 178)
Analysis type:	UnkNown
Chromatogram filename:	UNK-15092011-18.dat
Sample weight:	1.052



Element Name	Element %	Ret. Time
Nitrogen	14. 85	0. 73
Carbon	76. 65	1. 14
Hydrogen	8. 19	3. 87

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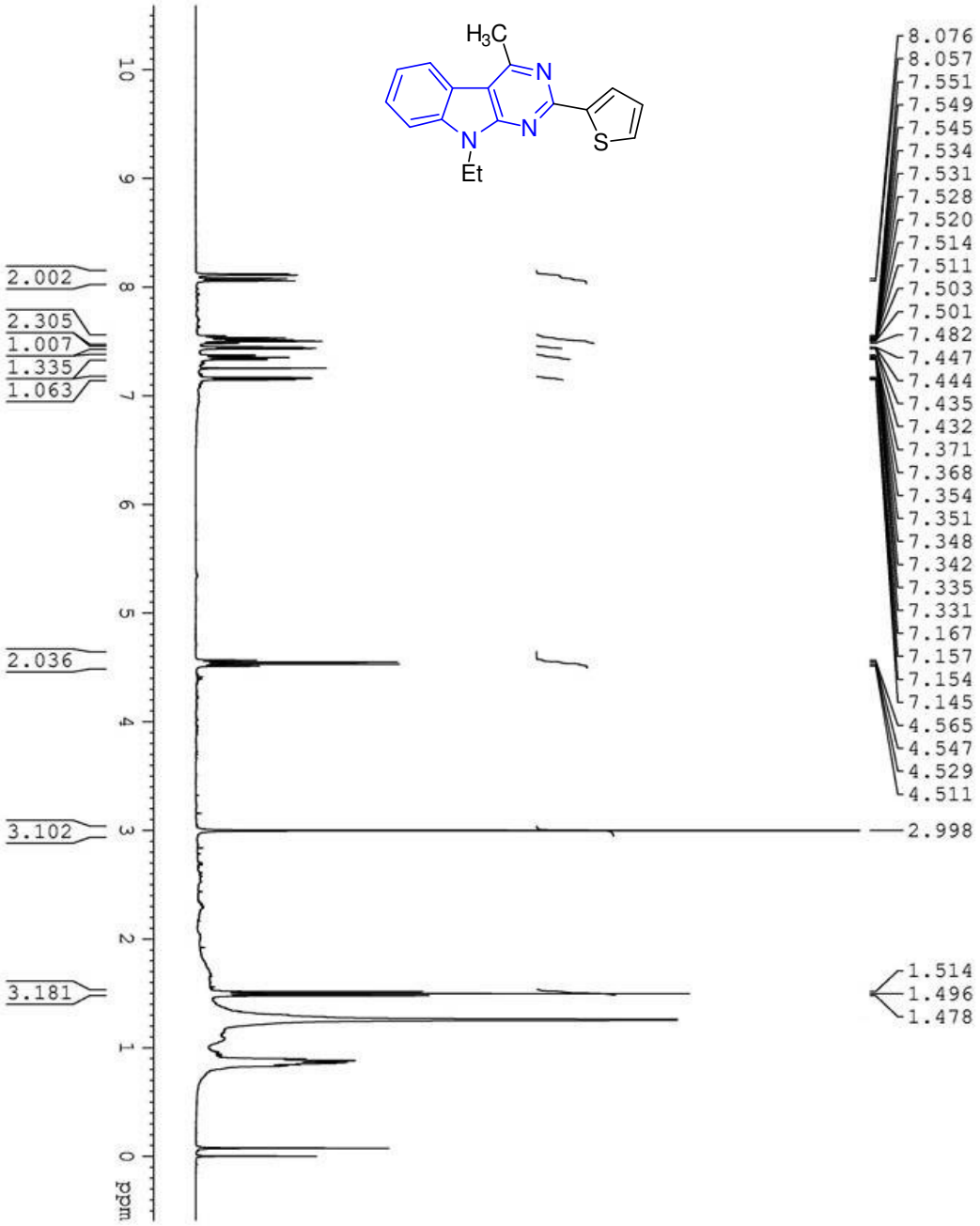
^{1}H NMR of 9-ethyl-4-methyl-2-(thiophen-2-yl)-9*H*-pyrimido[4,5-*b*]indole (3g):



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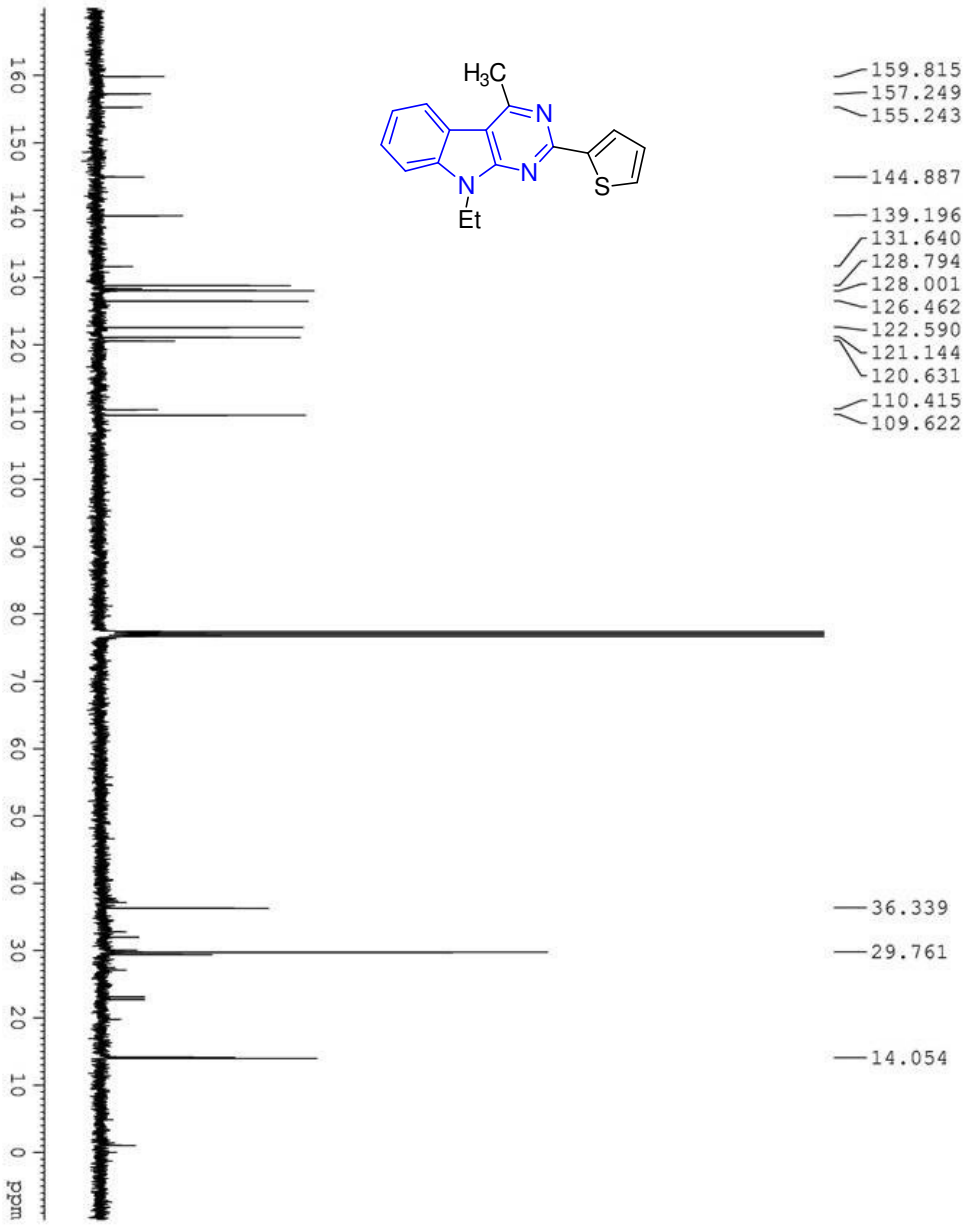
^{13}C NMR of 9-ethyl-4-methyl-2-(thiophen-2-yl)-9*H*-pyrimido[4,5-*b*]indole (3g):



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LCMS of 9-ethyl-4-methyl-2-(thiophen-2-yl)-9*H*-pyrimido[4,5-*b*]indole (3g):

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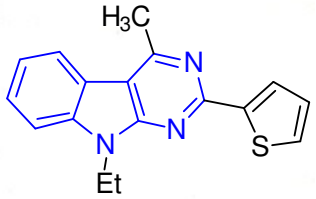
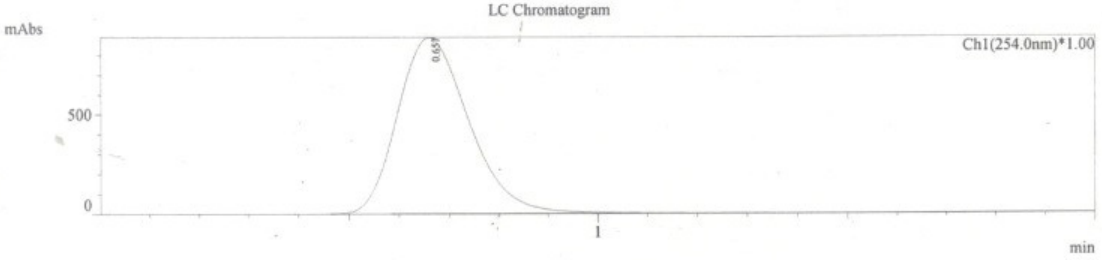


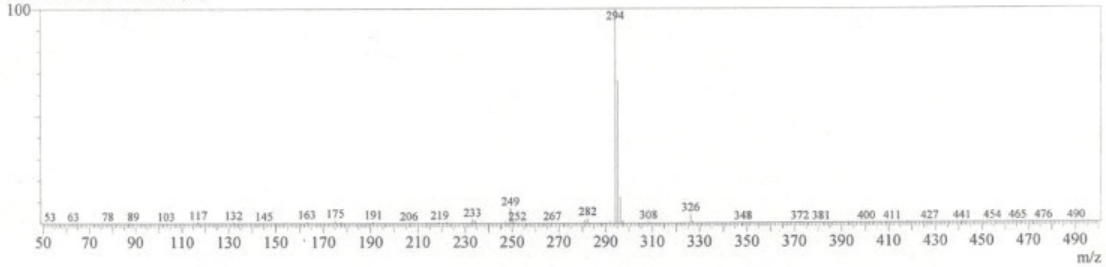
User : Admin
Sample : ASK-P5
Inj. Volume : 5.000
Data Name : C:\LCMSsolution\User\Data\ASK-P5-APCI-POS1.qld
Method Name : C:\LCMSsolution\User\Method\esi.qlm

LC Chromatogram



MS Spectrum

Line#:1 R.Time:0.732(Scan#:44) Positive
MassPeaks:194 BasePeak:294.25(14284222)
RawMode:Single 0.732(44)
BG Mode:Peak Start 0.527(32)



MS Peak Table

Peak#	R.Time	I.Time	F.Time	Area	Height	A/H	Mark	%Total	Name	Base m/z	Base Int.
1	0.732	0.527	1.043	266252521	14540830	18.31		100.00		294.25	14284222
				266252521	14540830			100.00			

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CHN Analysis of 9-ethyl-4-methyl-2-(thiophen-2-yl)-9*H*-pyrimido[4,5-*b*]indole (3g):

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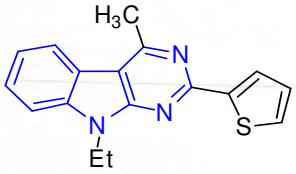
Method filename: E:\Program Files\Thermo Finnigan\Eager 300 for EA1112\DATA\Sys_data_ex
Sample ID: ASK-P5 (# 164)
Analysis type: UnkNown
Chromatogram filename: UNK-15092011-4.dat
Sample weight: 1.271



Element Name	Element %	Ret. Time
Nitrogen	14. 25	0. 78
Carbon	69. 45	1. 15
Hydrogen	5. 21	4. 19

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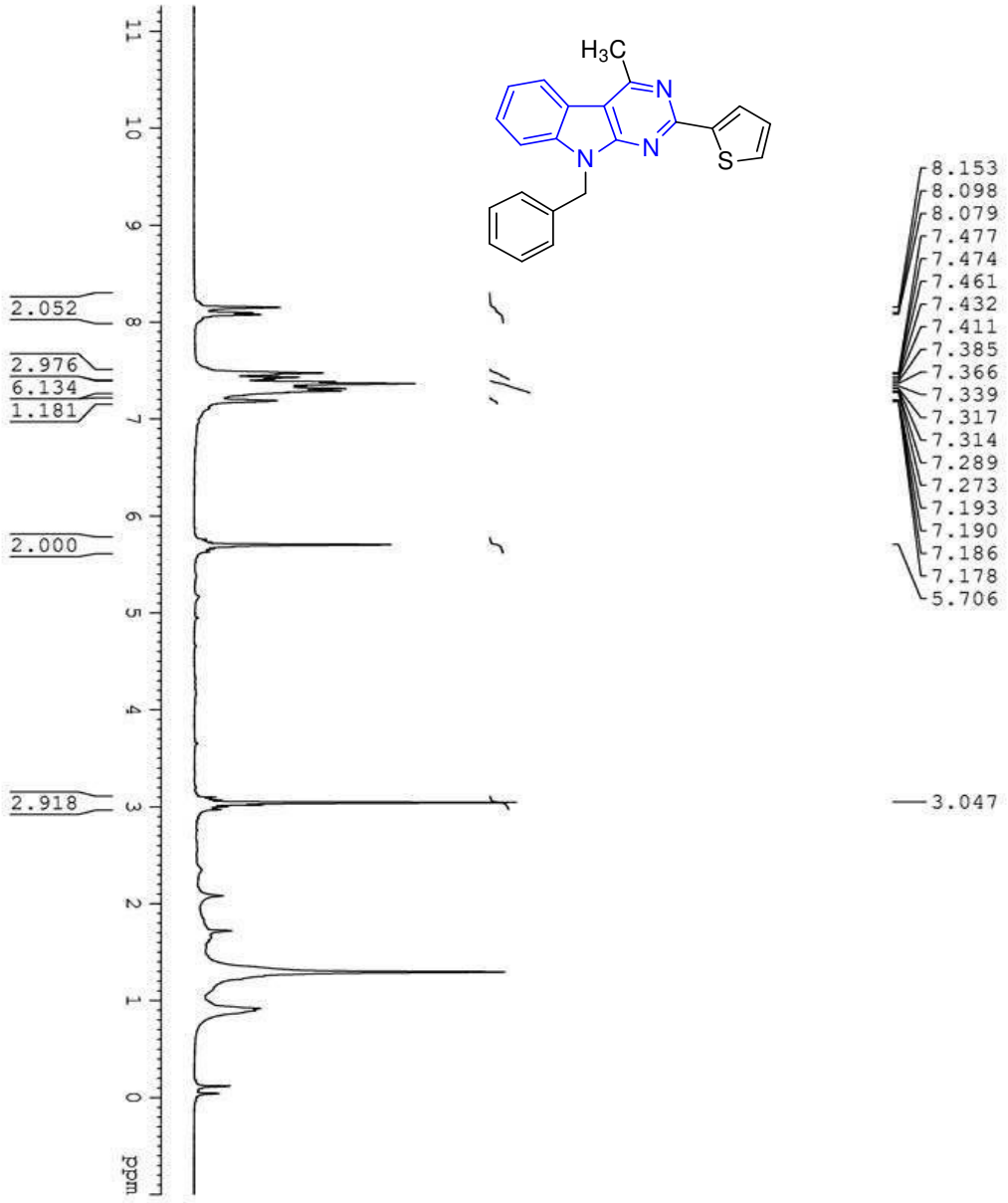
^{1}H NMR of 9-benzyl-4-methyl-2-(thiophen-2-yl)-9*H*-pyrimido[4,5-*b*]indole (3h)



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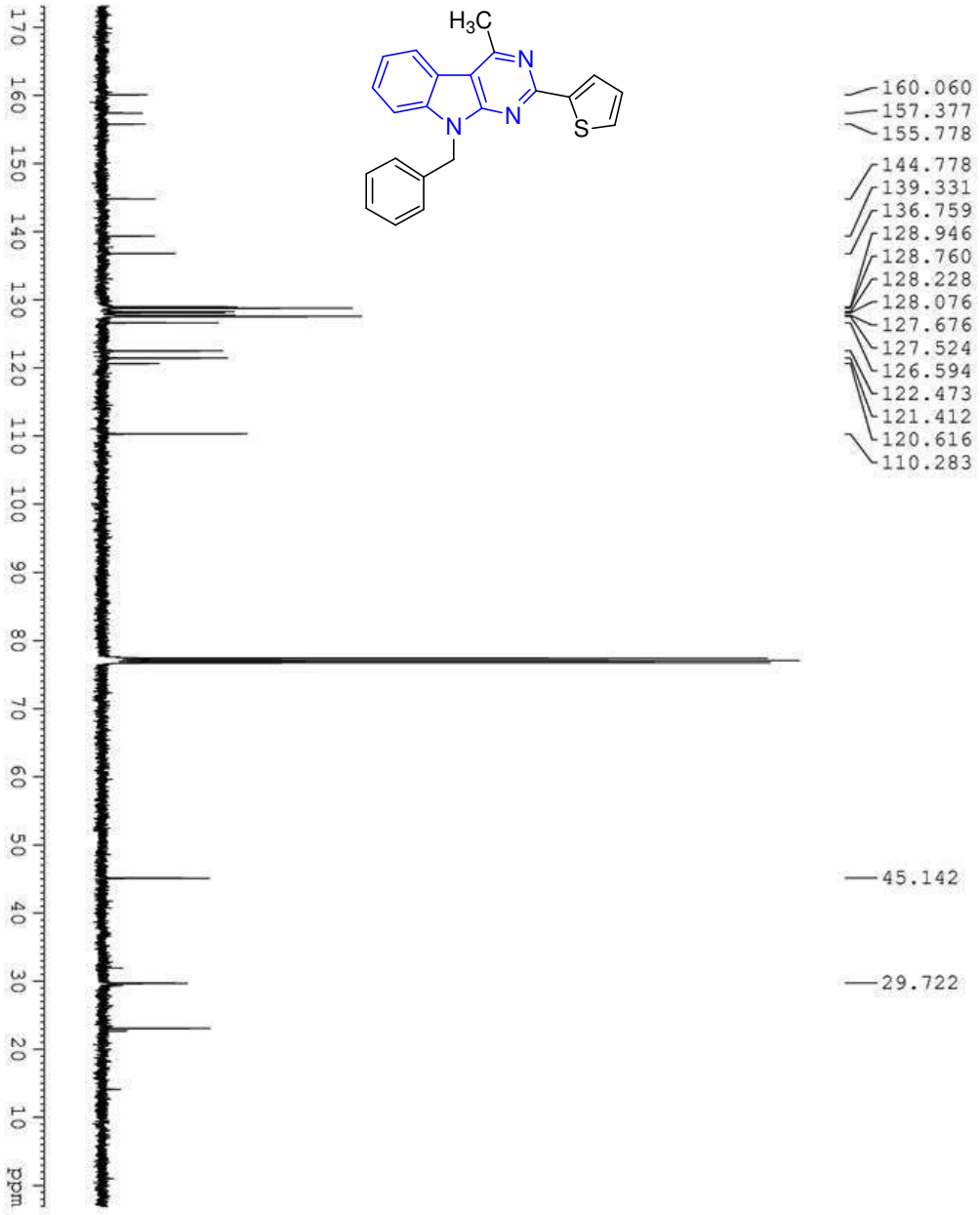
^{13}C NMR of 9-benzyl-4-methyl-2-(thiophen-2-yl)-9*H*-pyrimido[4,5-*b*]indole (3h)



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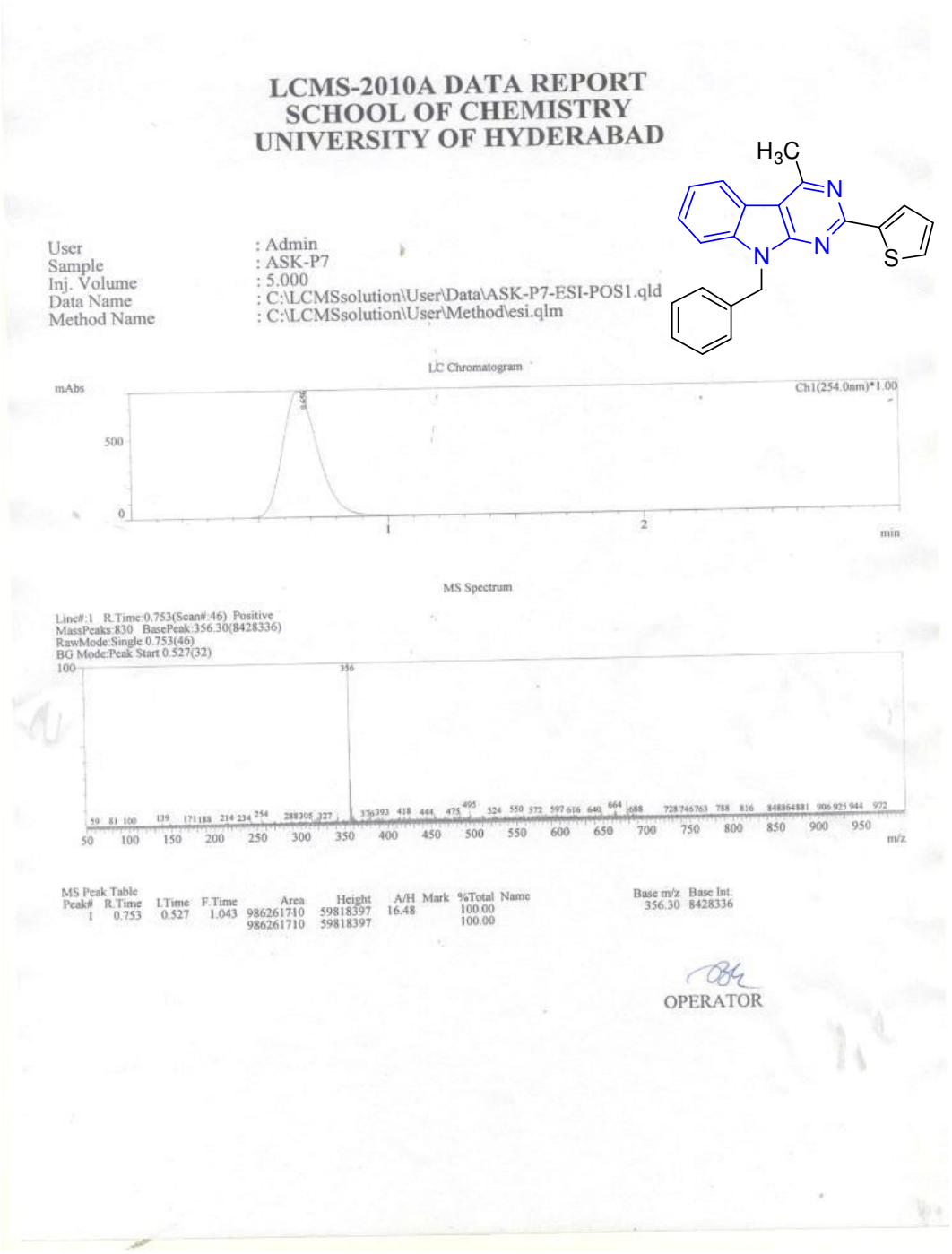
LCMS of 9-benzyl-4-methyl-2-(thiophen-2-yl)-9*H*-pyrimido[4,5-*b*]indole (3h)



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CHN Analysis of 9-benzyl-4-methyl-2-(thiophen-2-yl)-9*H*-pyrimido[4,5-*b*]indole (3h)

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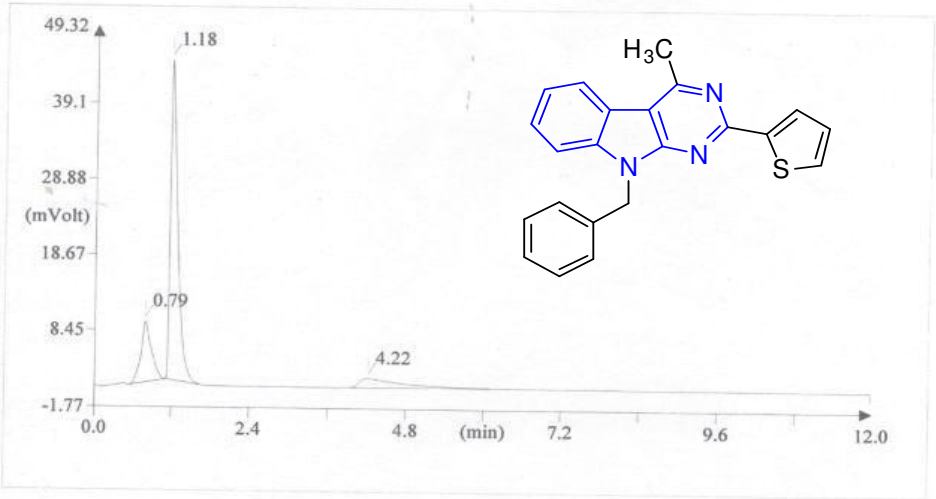
Method filename:	E:\Program Files\Thermo Finnigan\Eager 300 for EA1112\DATA\Sys_data_ex
Sample ID:	ASK-P7 (# 165)
Analysis type:	UnkNown
Chromatogram filename:	UNK-15092011-5.dat
Sample weight:	1.108



Element Name	Element %	Ret. Time
Nitrogen	11. 75	0. 79
Carbon	74. 19	1. 18
Hydrogen	4. 78	4. 22

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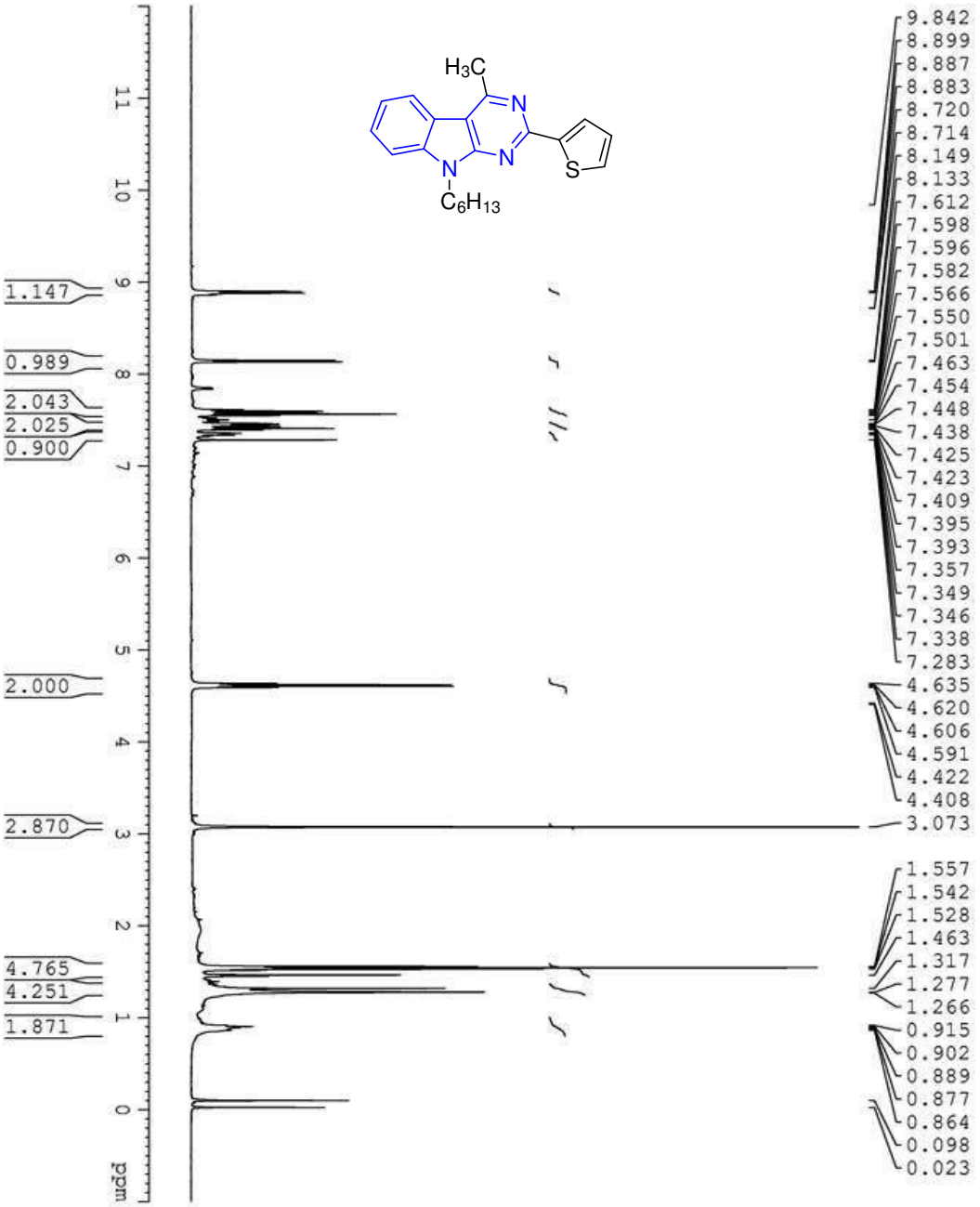
^{1}H NMR of 9-hexyl-4-methyl-2-(thiophen-2-yl)-9*H*-pyrimido[4,5-*b*]indole (3i)



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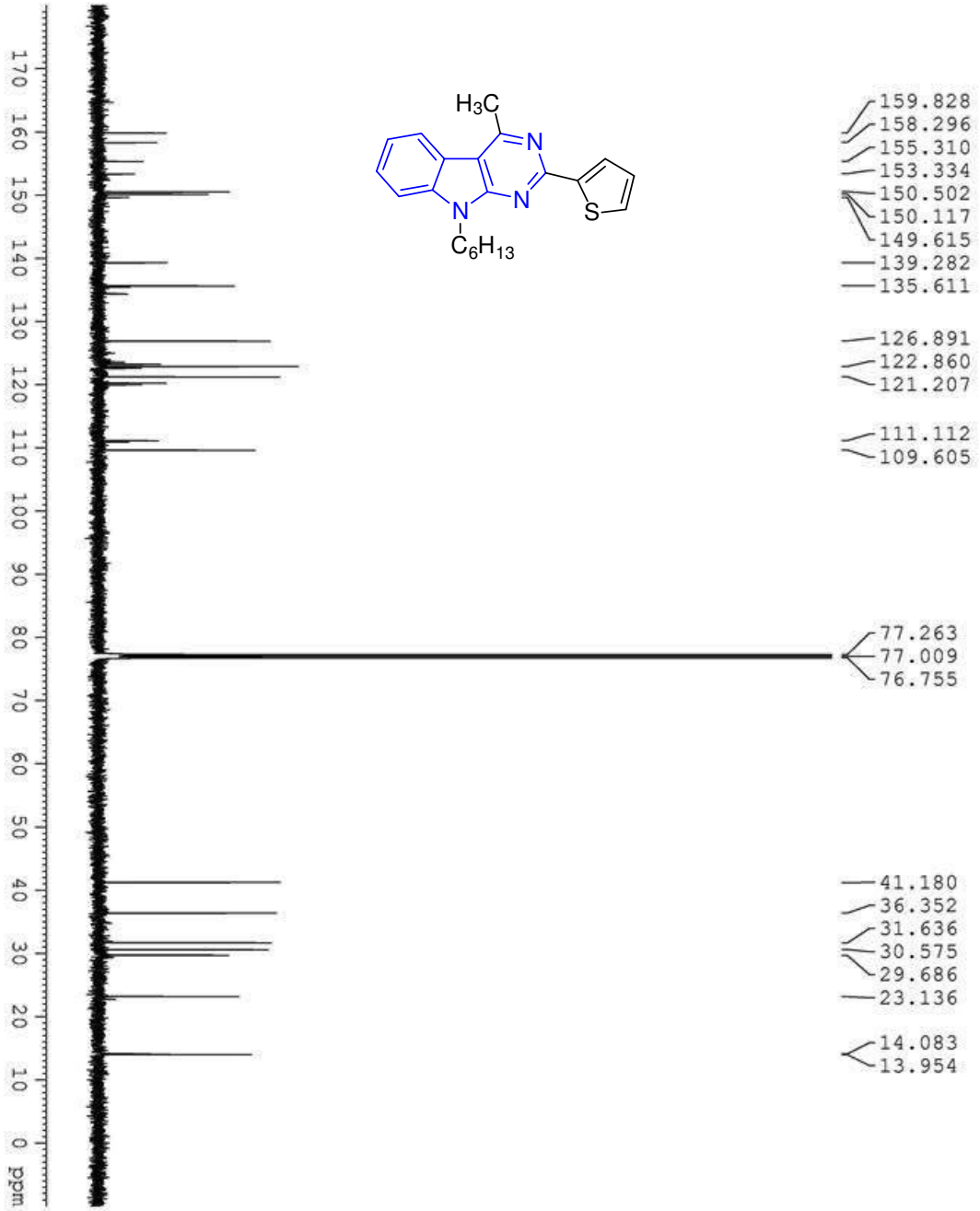
^{13}C NMR of 9-hexyl-4-methyl-2-(thiophen-2-yl)-9*H*-pyrimido[4,5-*b*]indole (3i)



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LCMS of 9-hexyl-4-methyl-2-(thiophen-2-yl)-9*H*-pyrimido[4,5-*b*]indole (3i)

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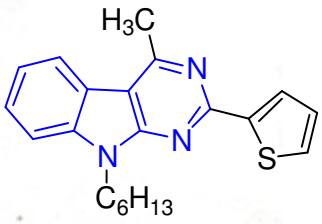
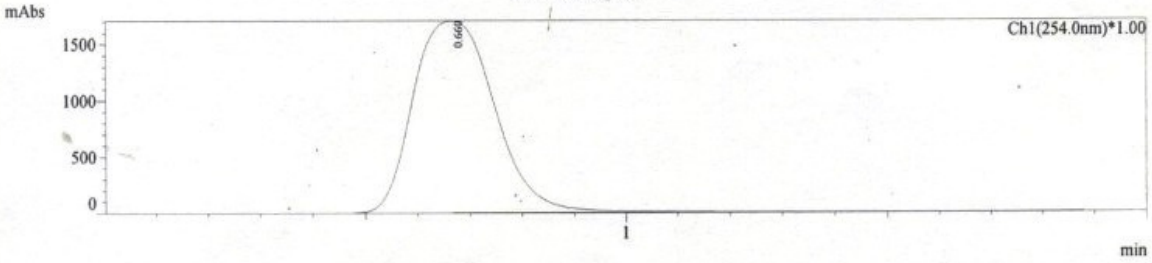


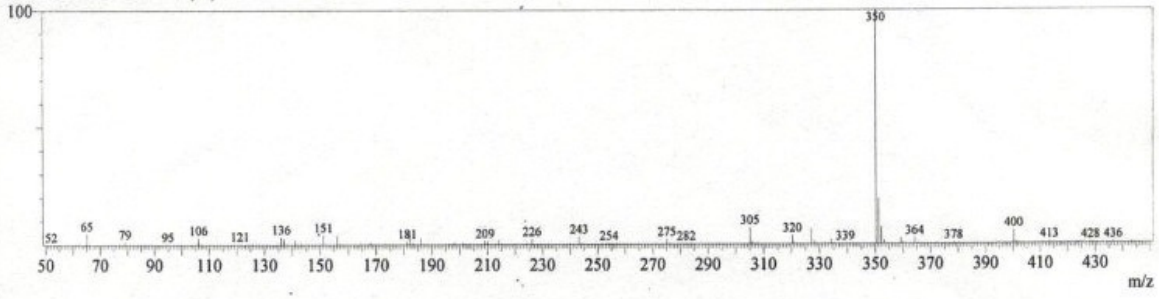
User : Admin
Sample : ASK-P3
Inj. Volume : 5.000
Data Name : C:\LCMSsolution\User\Data\ASK-P3-APCI-POS1.qld
Method Name : C:\LCMSsolution\User\Method\esi.qlm

LC Chromatogram



MS Spectrum

Line#:1 R.Time:0.790(Scan#:48) Positive
MassPeaks:302 BasePeak:350.20(3689540)
RawMode:Single 0.790(48)
BG Mode:Peak Start 0.593(36)



MS Peak Table

Peak#	R.Time	I.Time	F.Time	Area	Height	A/H	Mark	%Total	Name	Base m/z	Base Int.
1	0.790	0.593	1.010	98001247	7661108	12.79		100.00		350.20	3689540
				98001247	7661108			100.00			

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CHN Analysis of 9-hexyl-4-methyl-2-(thiophen-2-yl)-9*H*-pyrimido[4,5-*b*]indole (3i)

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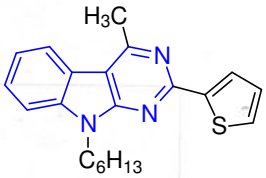
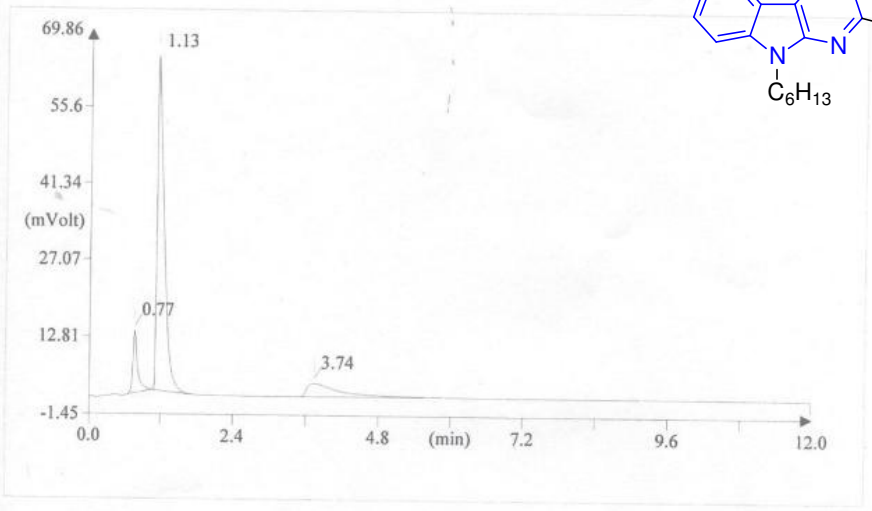
UNIVERSITY OF HYDERABAD

Method filename:	E:\Program Files\Thermo Finnigan\Eager 300 for EA1112\DATA\Sys_data_ex
Sample ID:	ASK-P3 (# 161)
Analysis type:	UnkNown
Chromatogram filename:	UNK-15092011-1.daf
Sample weight:	1.281





Element Name	Element %	Ret. Time
Nitrogen	12. 15	0. 77
Carbon	72. 08	1. 13
Hydrogen	6. 69	3. 74

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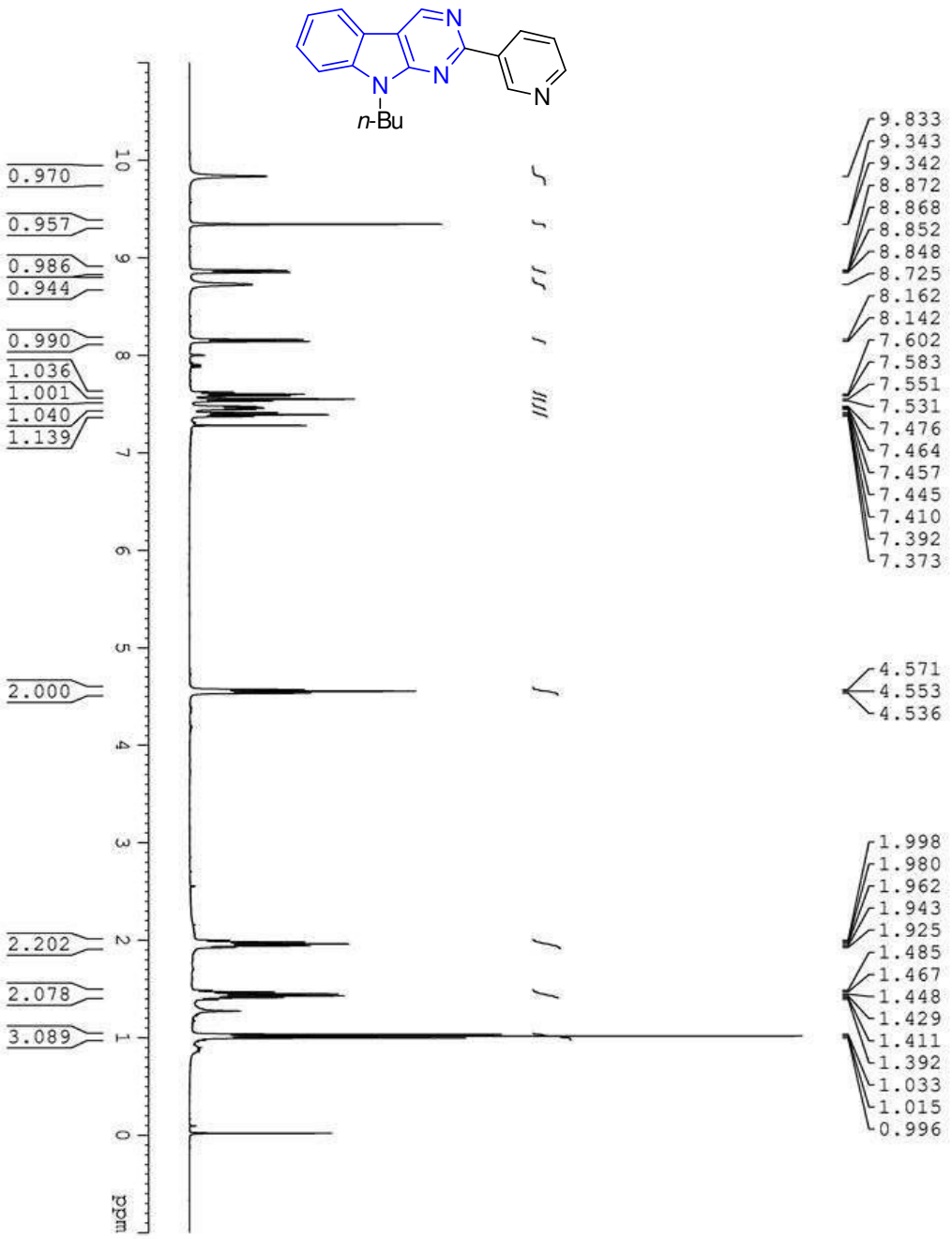
^{1}H NMR of 9-butyl-2-(pyridin-3-yl)-9H-pyrimido[4,5-*b*]indole (3j)



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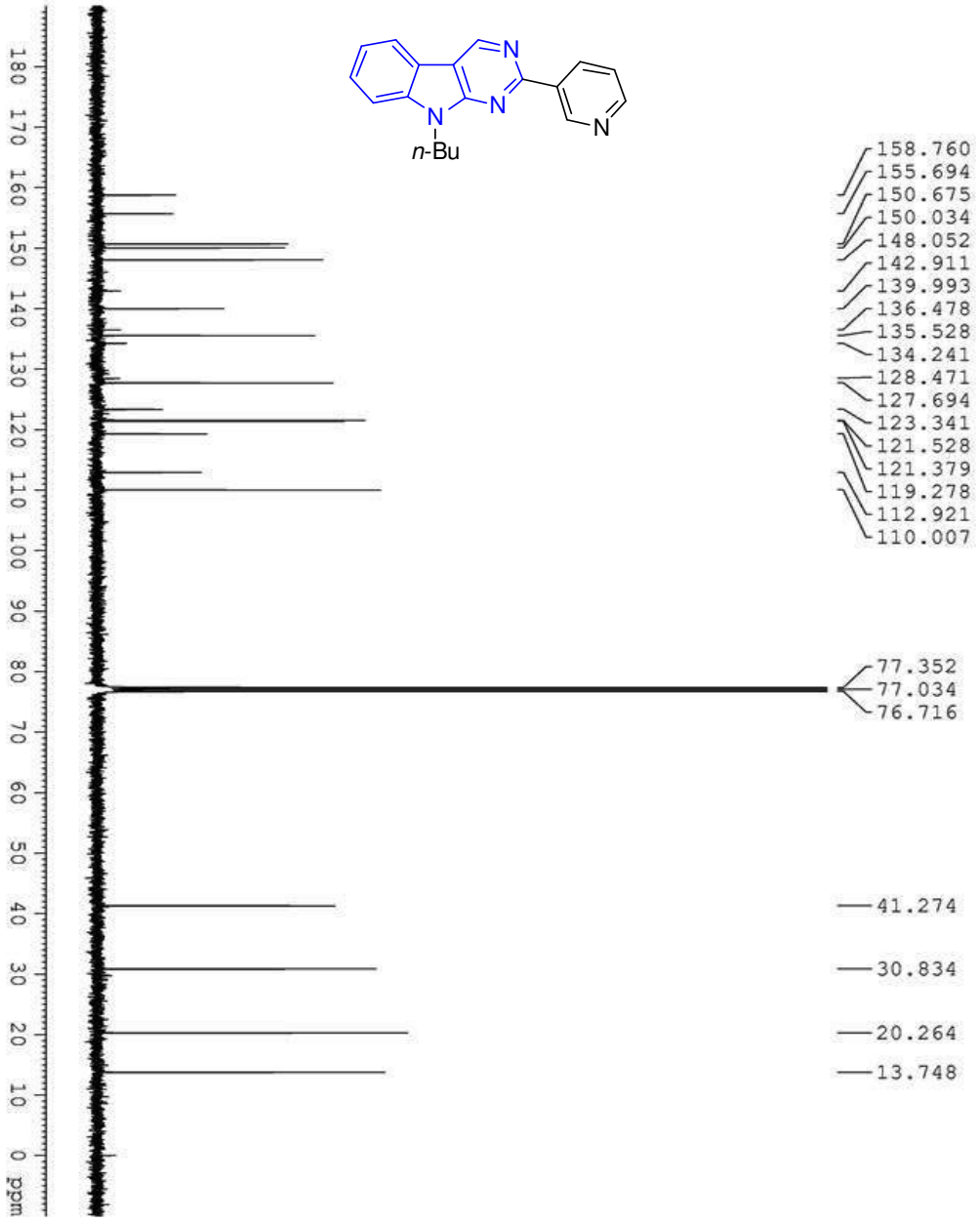
^{13}C NMR of 9-Butyl-2-(pyridin-3-yl)-9H-pyrimido[4,5-*b*]indole (3j)



LCMS of 9-Butyl-2-(pyridin-3-yl)-9H-pyrimido[4,5-*b*]indole (3j)

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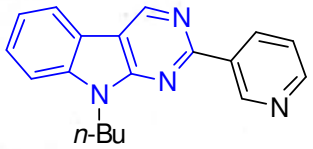
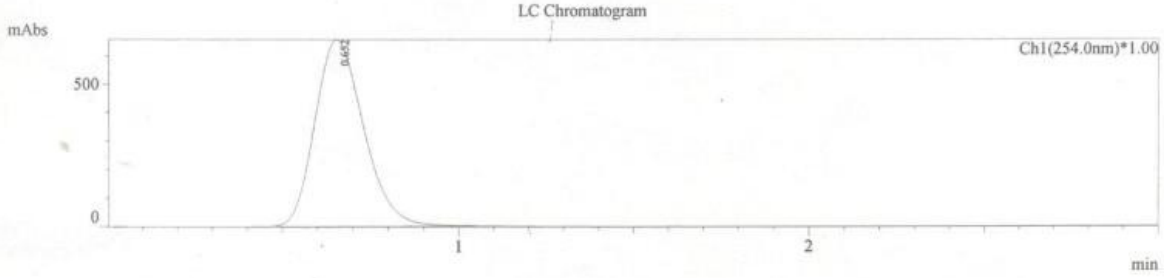


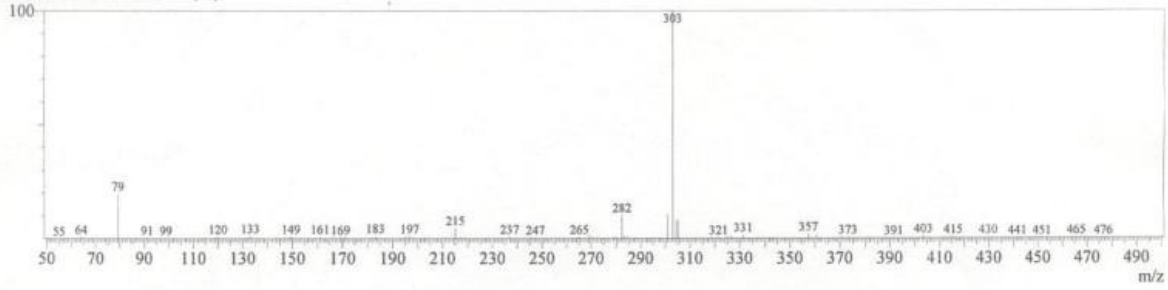
User : Admin
Sample : ASK-P11
Inj. Volume : 5.000
Data Name : C:\LCMSsolution\User\Data\ASK-P11-APCI-POS1.qld
Method Name : C:\LCMSsolution\User\Method\esi.qlm

LC Chromatogram



MS Spectrum

Line#:1 R.Time:0.796(Scan#:48) Positive
MassPeaks:284 BasePeak:302.95(8259348)
RawMode:Single 0.796(48)
BG Mode:Peak Start 0.527(32)



MS Peak Table

Peak#	R.Time	I.Time	F.Time	Area	Height	A/H	Mark	%Total	Name	Base m/z	Base Int.
1	0.796	0.527	1.043	221616904	14792901	14.98		100.00		302.95	8259348
				221616904	14792901			100.00			

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CHN Analysis of 9-Butyl-2-(pyridin-3-yl)-9H-pyrimido[4,5-*b*]indole (3j)

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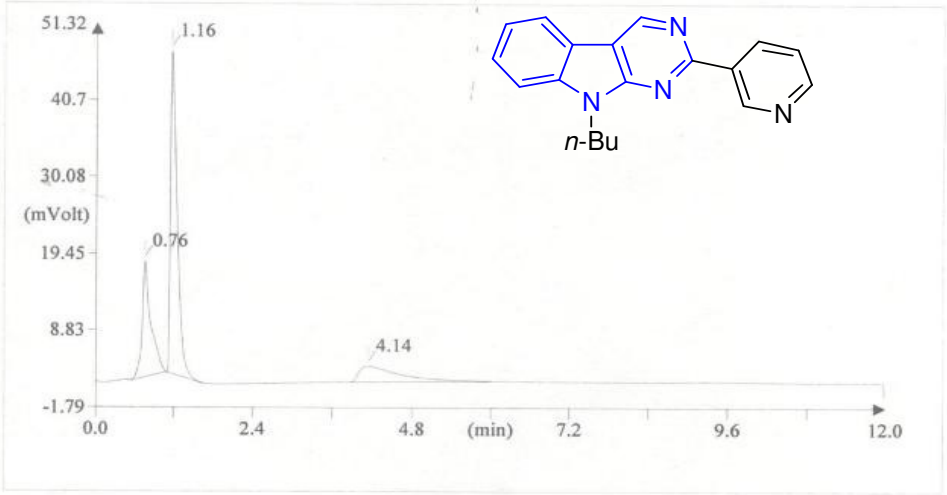
Method filename:	E:\Program Files\Thermo Finnigan\Eager 300 for EA1112\DATA\Sys_data_ex
Sample ID:	ASK-P11 (# 180)
Analysis type:	UnkNown
Chromatogram filename:	UNK-15092011-20.dat
Sample weight:	1.107



Element Name	Element %	Ret. Time
Nitrogen	18. 41	0. 76
Carbon	75. 54	1. 16
Hydrogen	6. 08	4. 14

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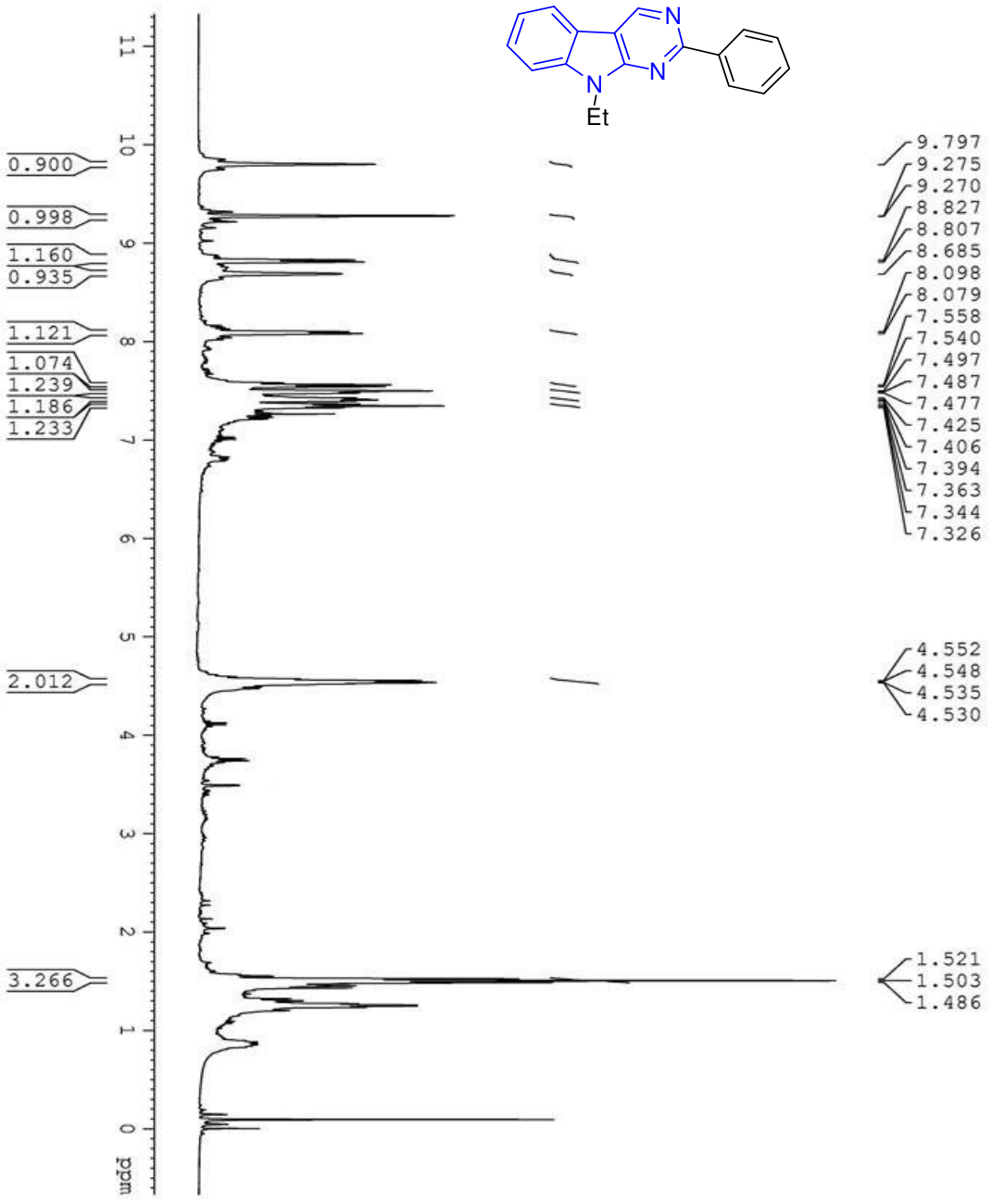
^{1}H NMR of 9-ethyl-2-phenyl-9*H*-pyrimido[4,5-*b*]indole (3k)



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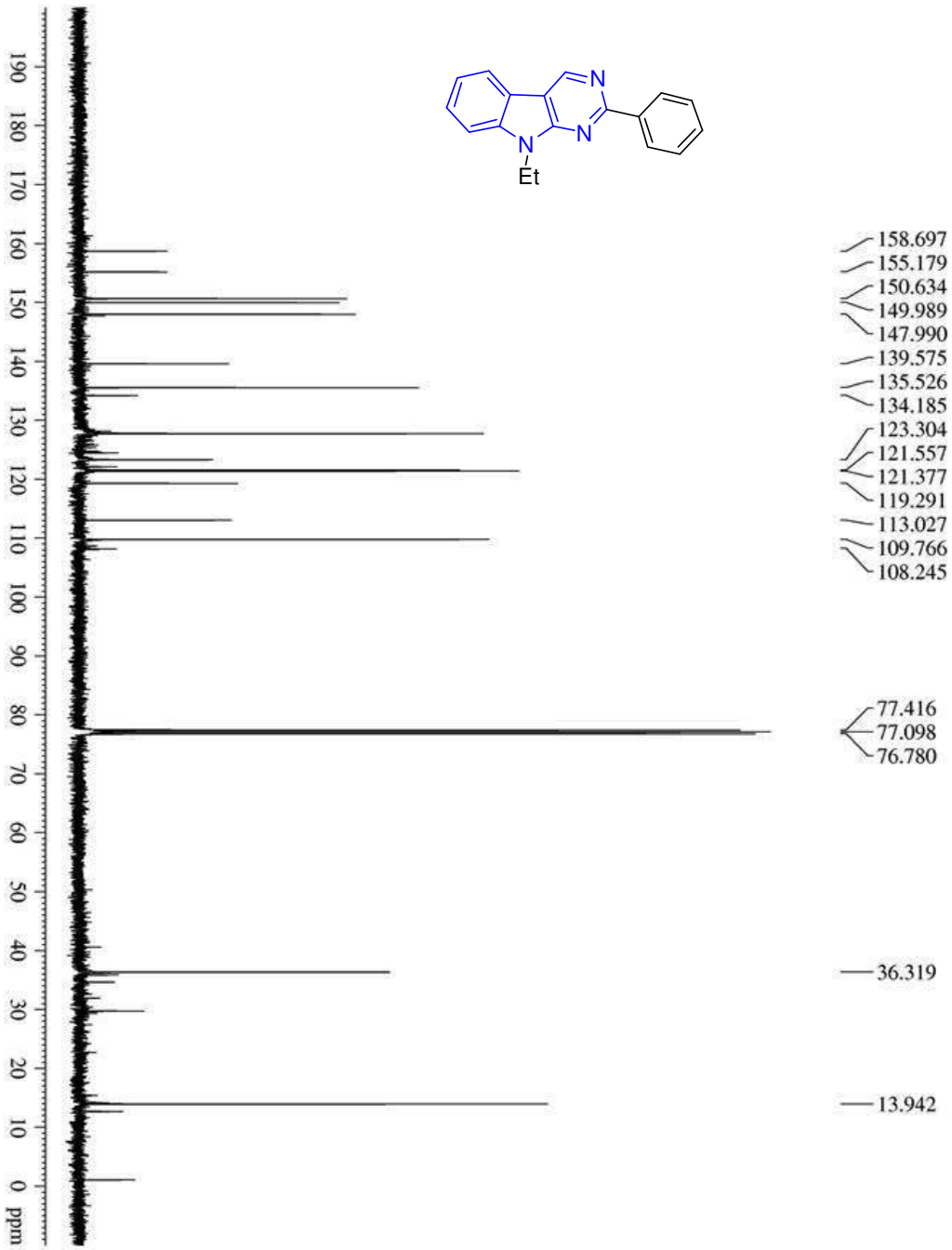
^{13}C NMR of 9-ethyl-2-phenyl-9*H*-pyrimido[4,5-*b*]indole (3k)



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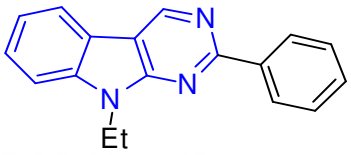
LCMS of 9-ethyl-2-phenyl-9*H*-pyrimido[4,5-*b*]indole (3k)



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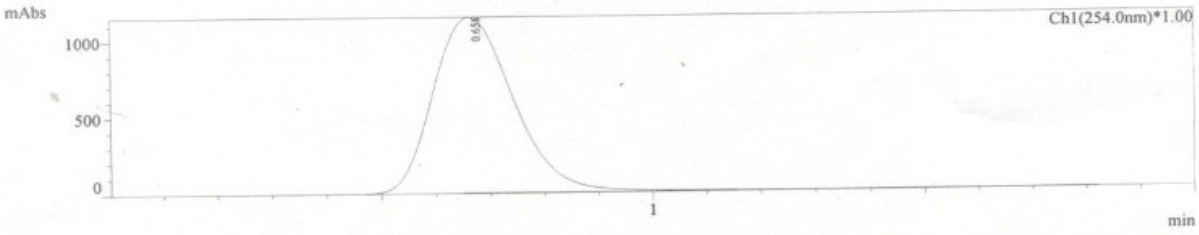
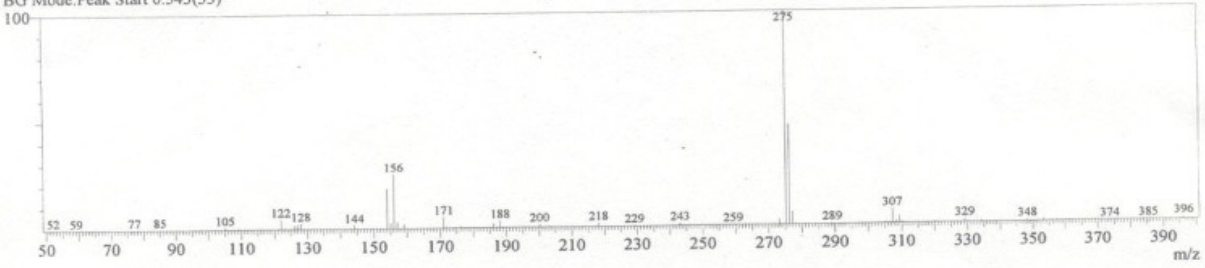
User	: Admin
Sample	: ASK-P4
Inj. Volume	: 5.000
Data Name	: C:\LCMSsolution\User\Data\ASK-P4-APCI-POS1.qld
Method Name	: C:\LCMSsolution\User\Method\esi.qlm

LC Chromatogram



MS Spectrum

Line#:1 R.Time:0.805(Scan#:49) Positive
MassPeaks:214 BasePeak:275.15(12970499)
RawMode:Single 0.805(49)
BG Mode:Peak Start 0.543(33)



MS Peak Table

Peak#	R.Time	I.Time	F.Time	Area	Height	A/H	Mark	%Total	Name
1	0.805	0.543	1.144	496106707	29001617	17.10		100.00	
				496106707	29001617			100.00	

Base m/z	Base Int.
275.15	12970499

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CHN Analysis of 9-ethyl-2-phenyl-9*H*-pyrimido[4,5-*b*]indole (3k)

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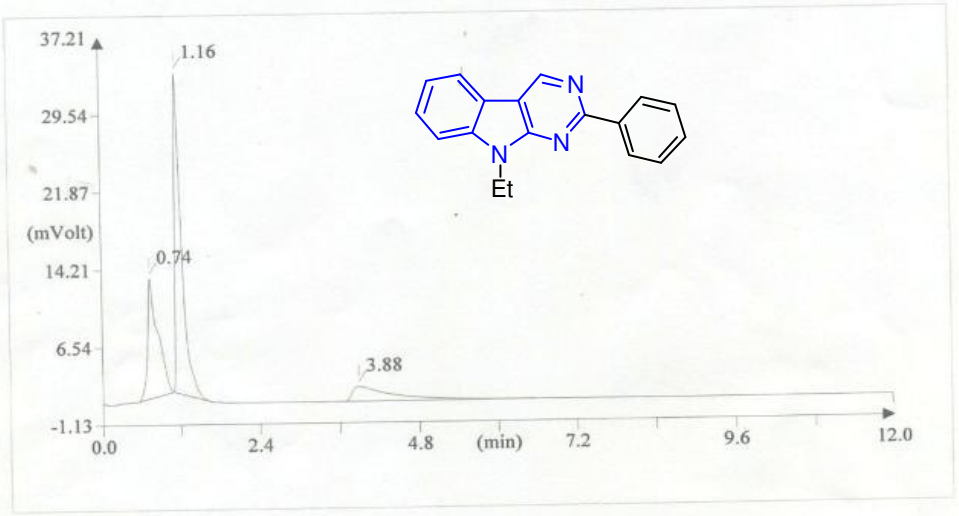
Method filename:	C:\Program Files\Thermo Finnigan\Eager 300 for EA1112\DATA\Sys_data_ex
Sample ID:	ASK-P4 (# 61)
Analysis type:	UnkNown
Chromatogram filename:	UNK-02022012-11.dat
Sample weight:	.975



Element Name	Element %	Ret. Time
Nitrogen	15. 25	0. 74
Carbon	79. 21	1. 16
Hydrogen	5. 58	3. 88

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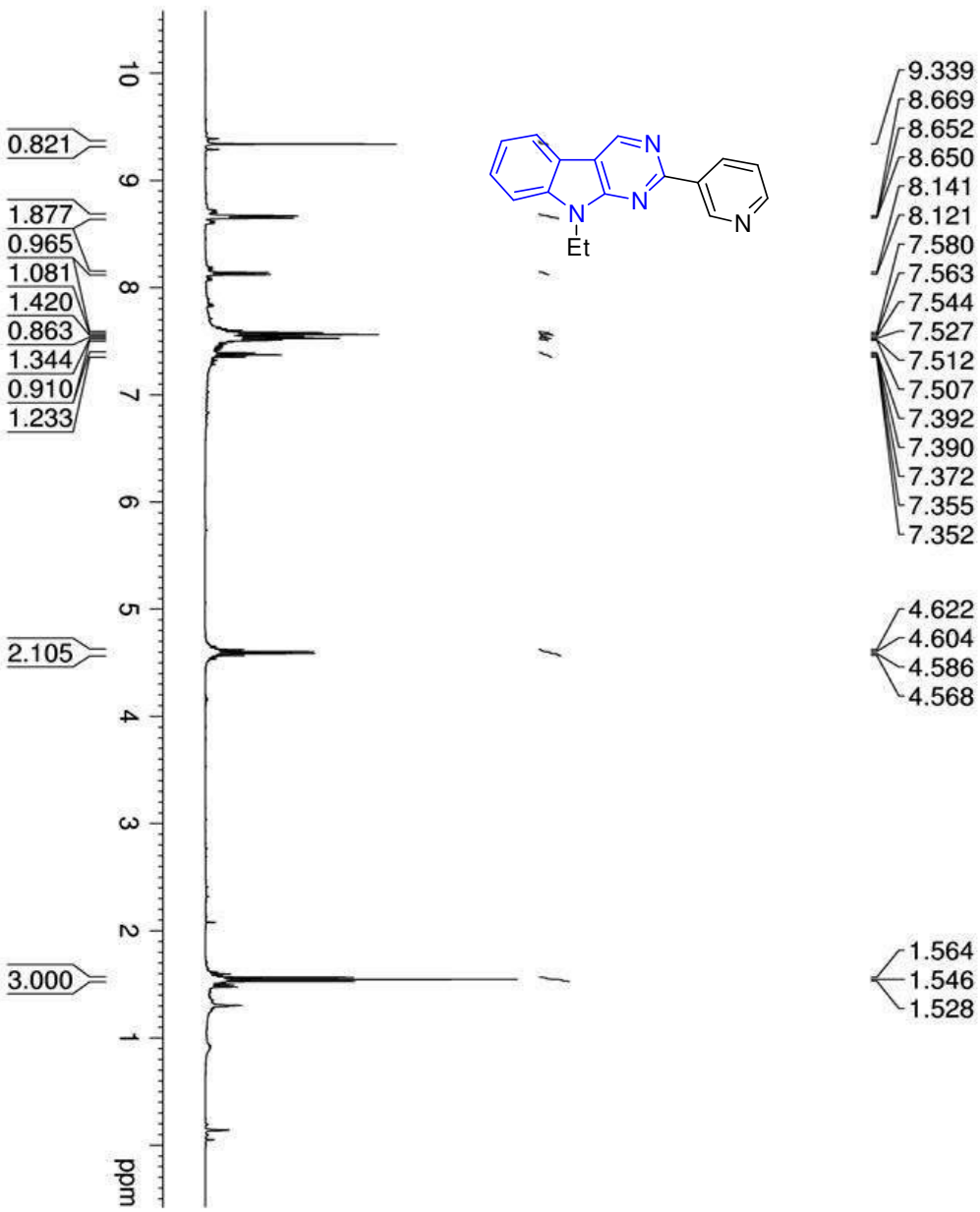
^{1}H NMR of 9-ethyl-2-(pyridin-3-yl)-9*H*-pyrimido[4,5-*b*]indole (3l)



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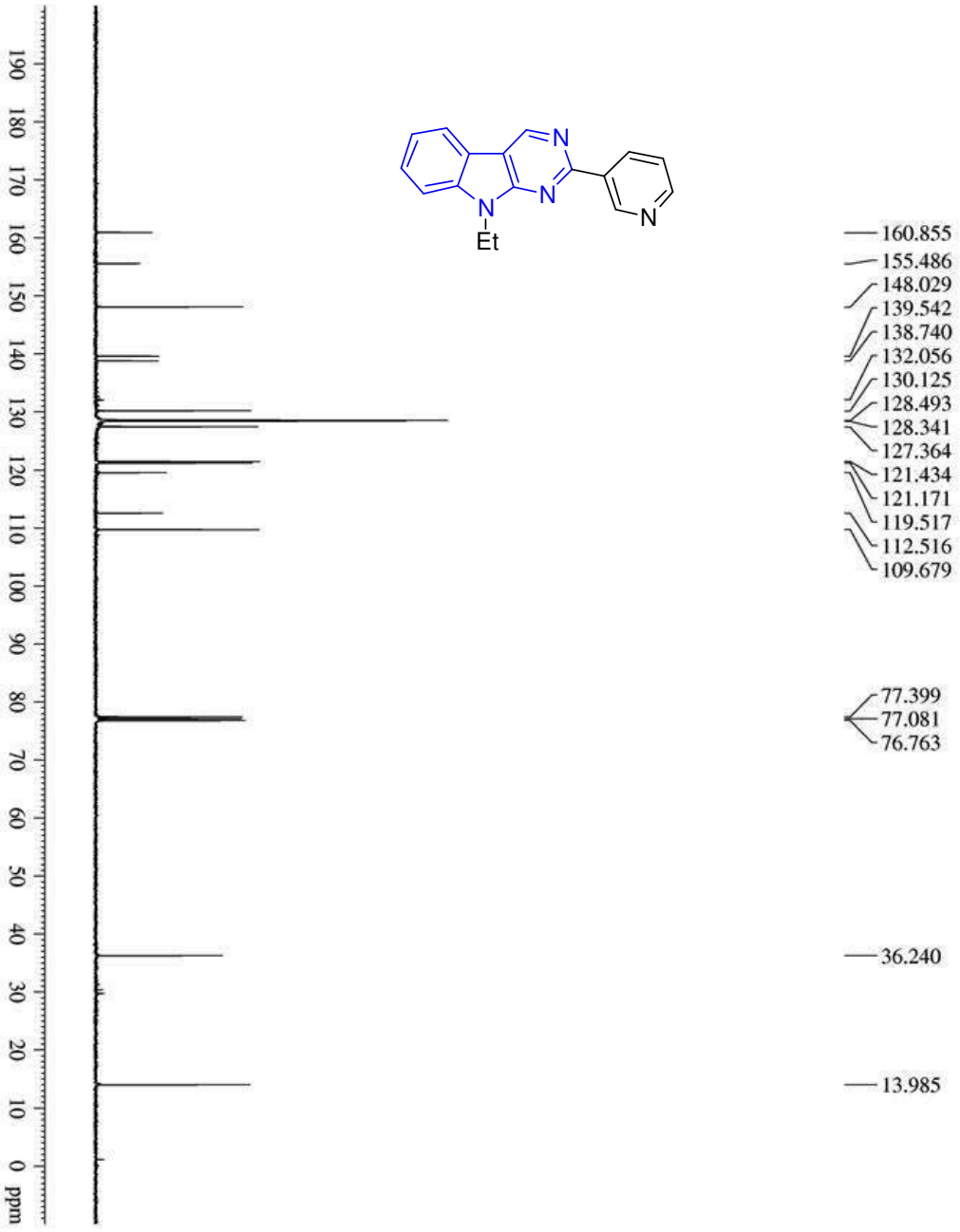
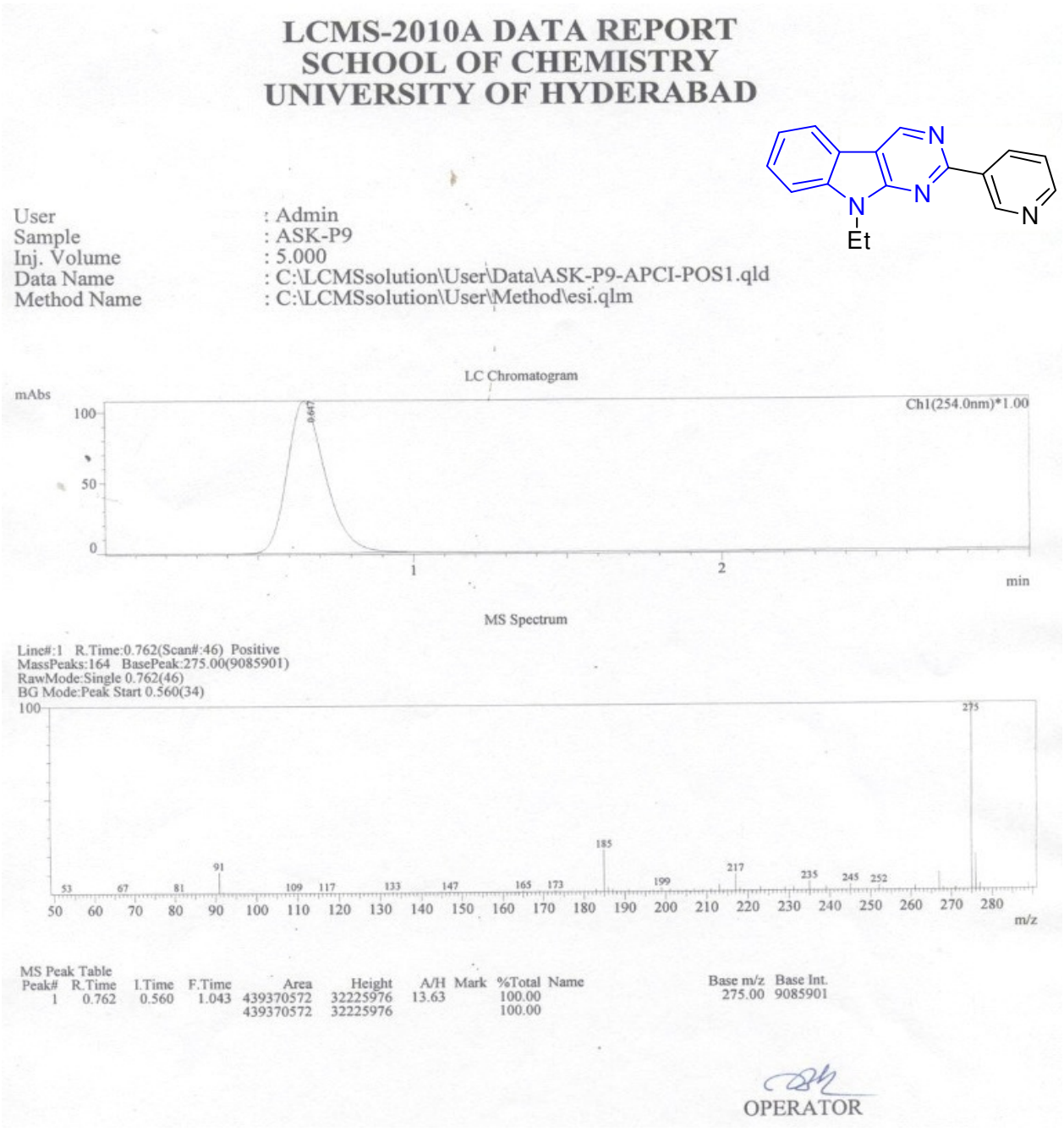
^{13}C NMR of 9-ethyl-2-(pyridin-3-yl)-9*H*-pyrimido[4,5-*b*]indole (3l)



LCMS of 9-ethyl-2-(pyridin-3-yl)-9*H*-pyrimido[4,5-*b*]indole (3l)



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CHN Analysis of 9-ethyl-2-(pyridin-3-yl)-9*H*-pyrimido[4,5-*b*]indole (3l)

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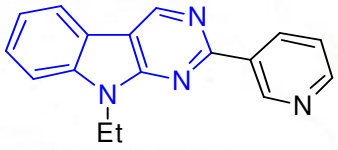
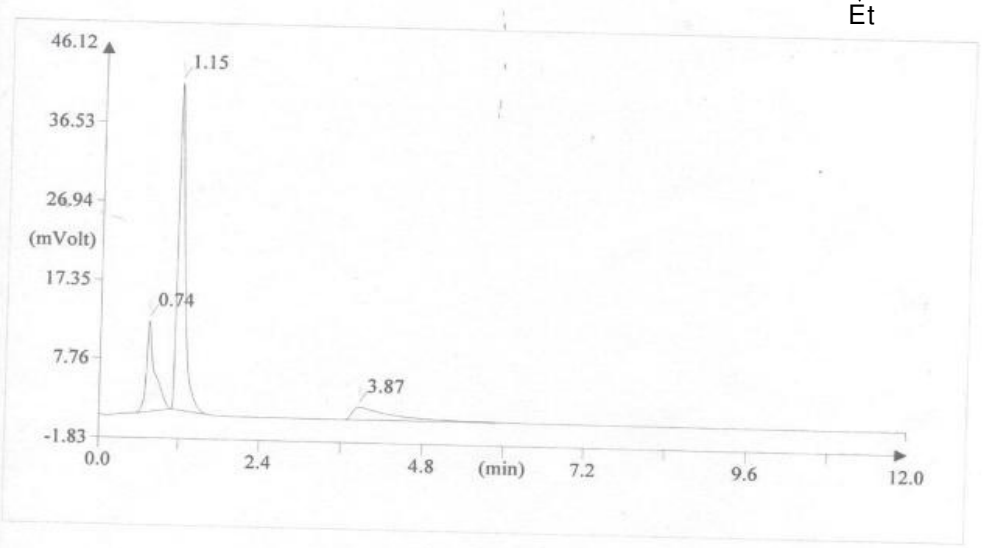
Method filename:	E:\Program Files\Thermo Finnigan\Eager 300 for EA1112\DATA\Sys_data_ex
Sample ID:	ASK-P9 (# 184)
Analysis type:	UnkNown
Chromatogram filename:	UNK-15092011-24.dat
Sample weight:	1.065





Element Name	Element %	Ret. Time
Nitrogen	20. 36	0. 74
Carbon	74. 32	1. 15
Hydrogen	5. 21	3. 87

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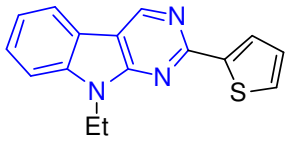
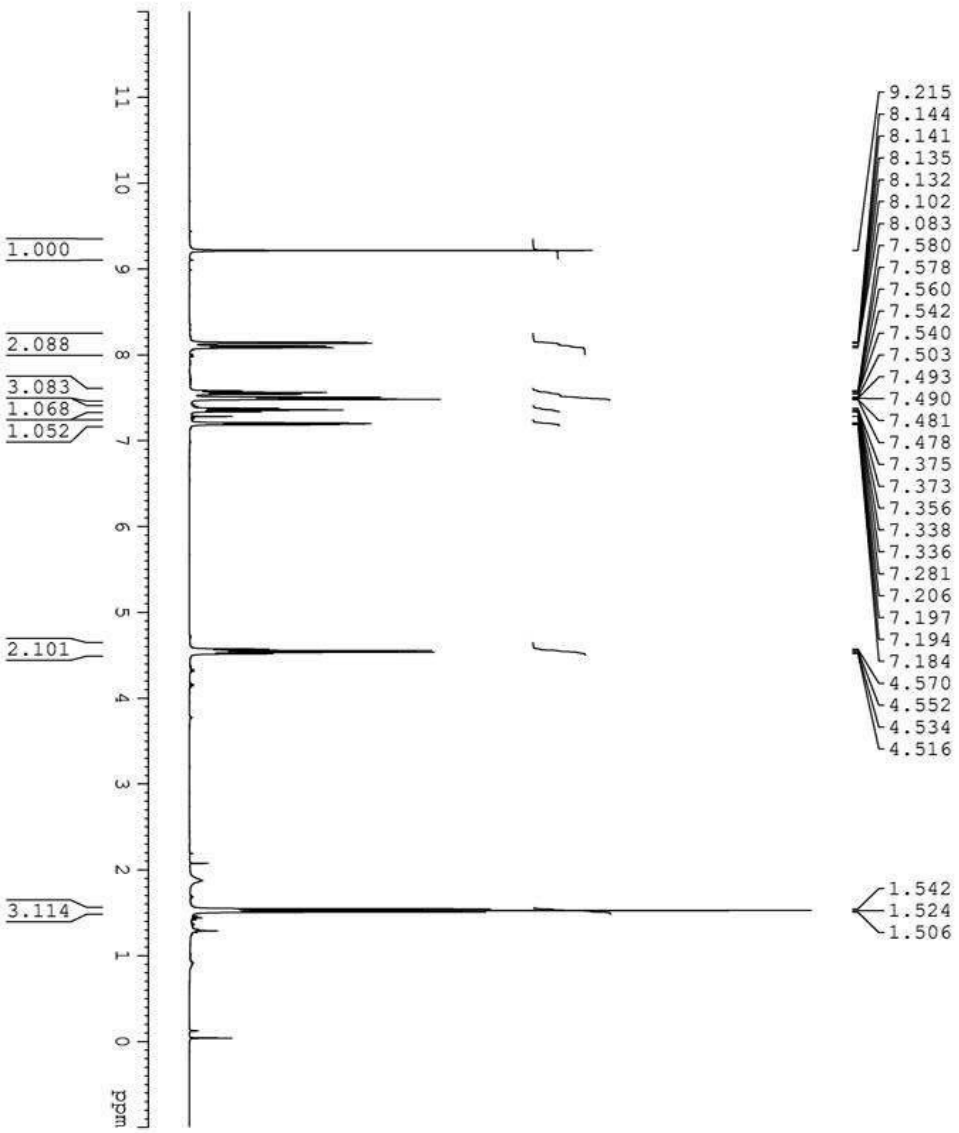
^{1}H NMR of 9-ethyl-2-(thiophen-2-yl)-9*H*-pyrimido[4,5-*b*]indole (3m)





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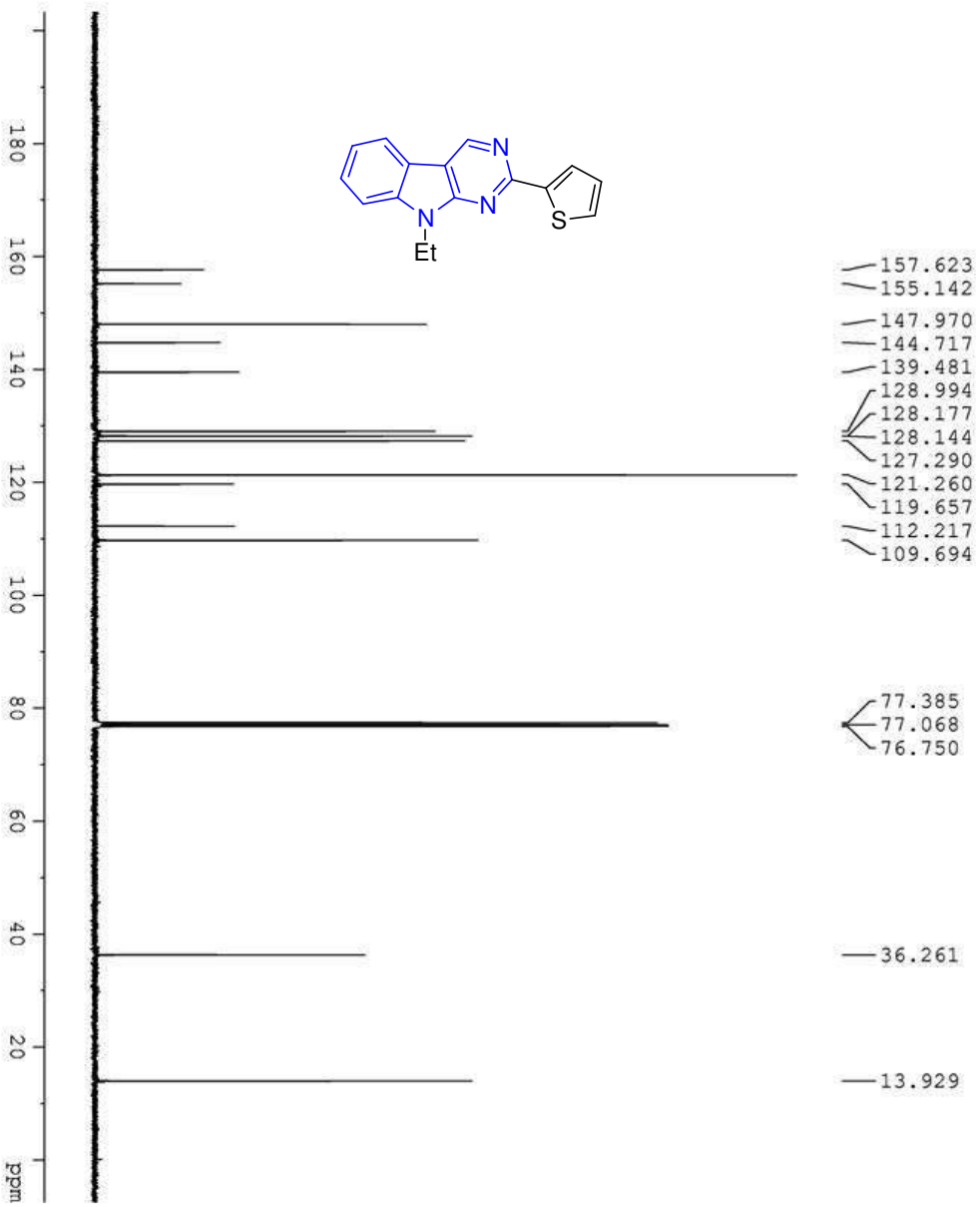
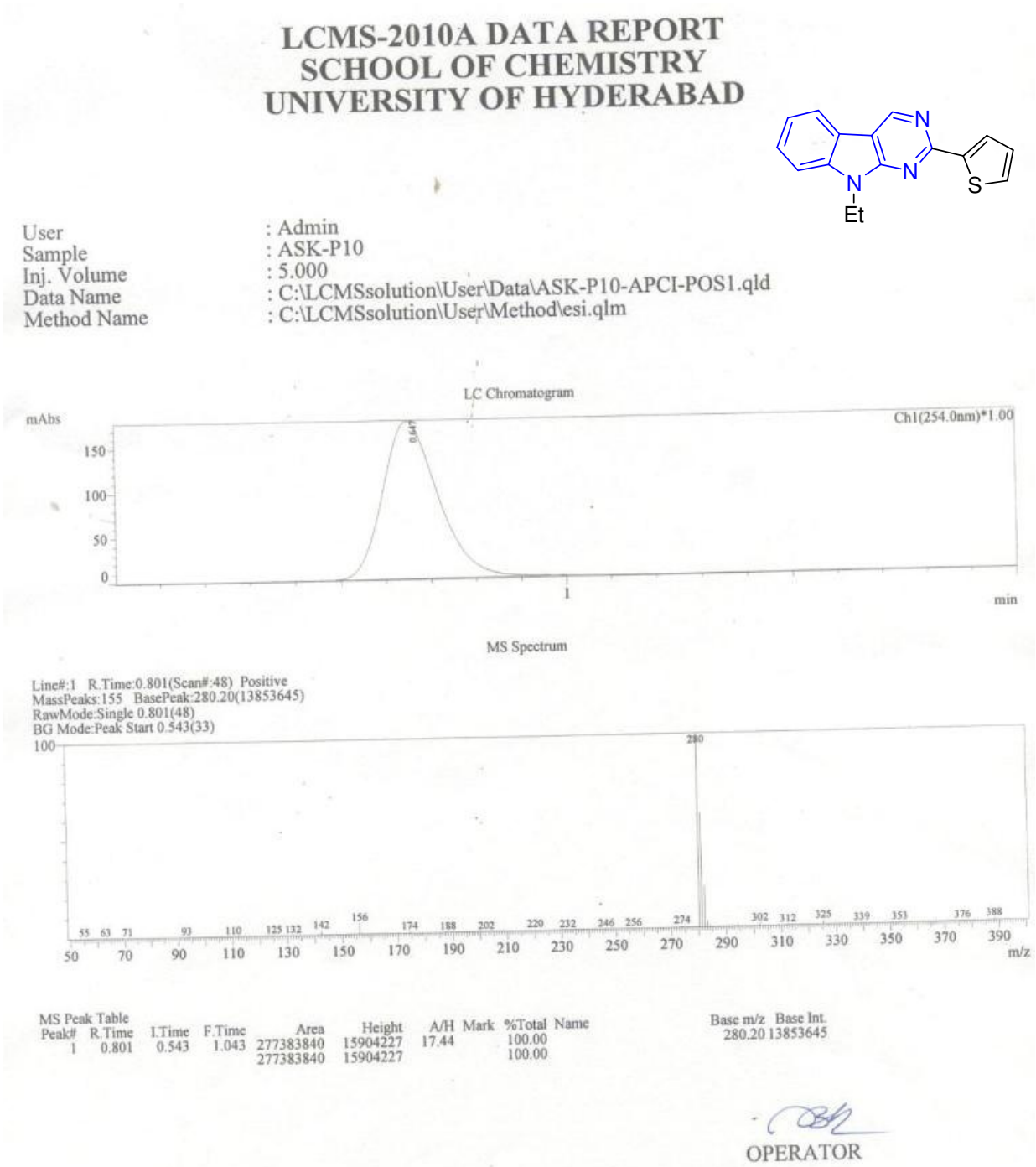
^{13}C NMR of 9-ethyl-2-(thiophen-2-yl)-9*H*-pyrimido[4,5-*b*]indole (3m)



LCMS of 9-ethyl-2-(thiophen-2-yl)-9*H*-pyrimido[4,5-*b*]indole (3m)



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CHN Analysis of 9-ethyl-2-(thiophen-2-yl)-9*H*-pyrimido[4,5-*b*]indole (3m)

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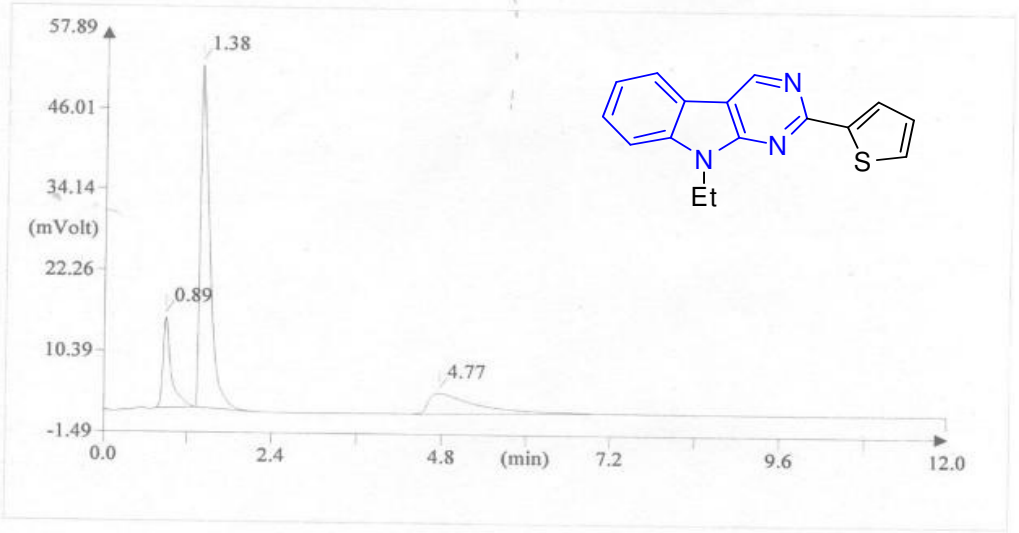
Method filename:	E:\Program Files\Thermo Finnigan\Eager 300 for EA1112\DATA\Sys_data_ex
Sample ID:	ASK-P10 (# 163)
Analysis type:	UnkNown
Chromatogram filename:	UNK-15092011-3.dat
Sample weight:	1.195



Element Name	Element %	Ret. Time
Nitrogen	15. 11	0. 89
Carbon	68. 85	1. 38
Hydrogen	4. 61	4. 77

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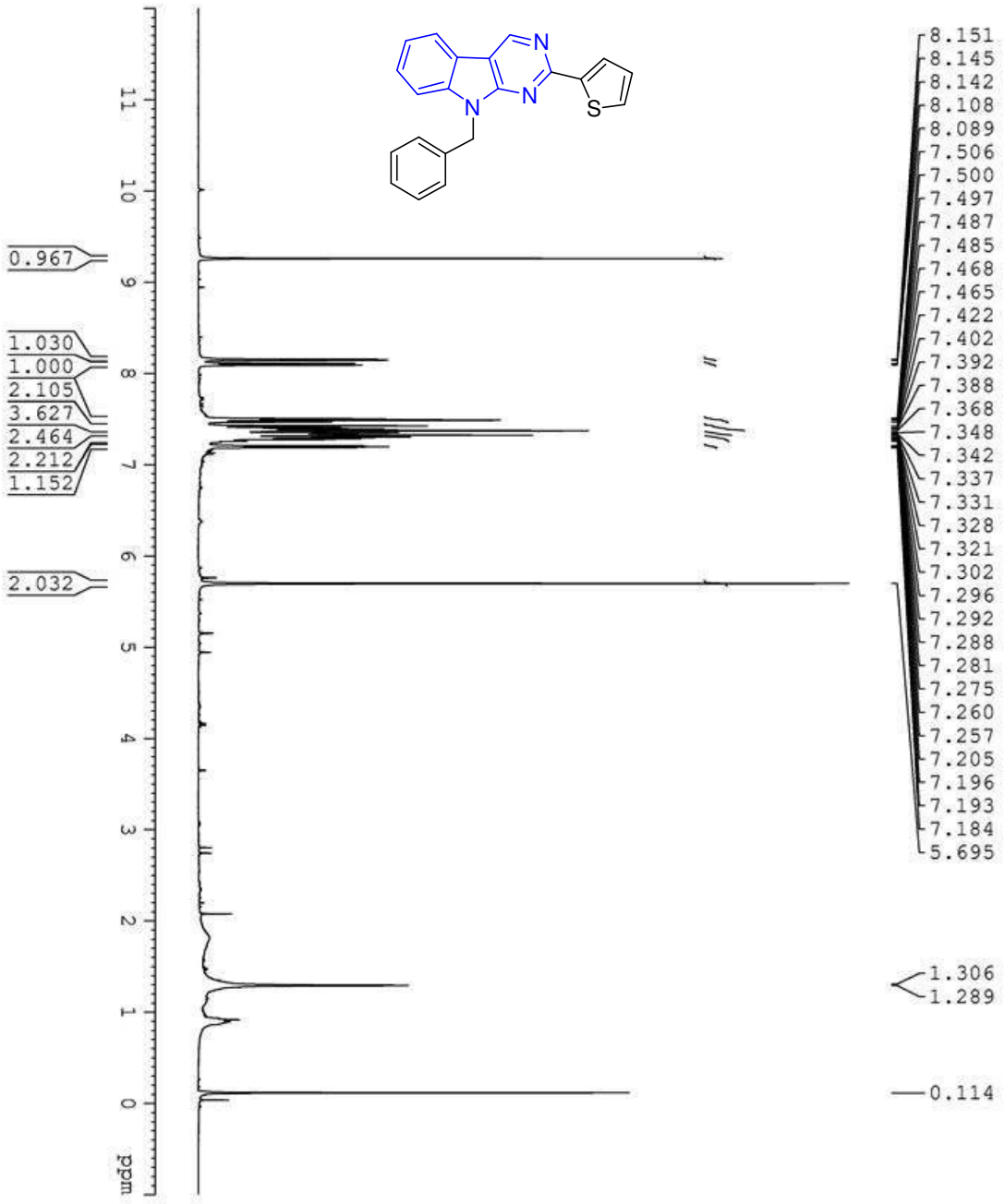
^{1}H NMR of 9-benzyl-2-(thiophen-2-yl)-9*H*-pyrimido[4,5-*b*]indole (3n)



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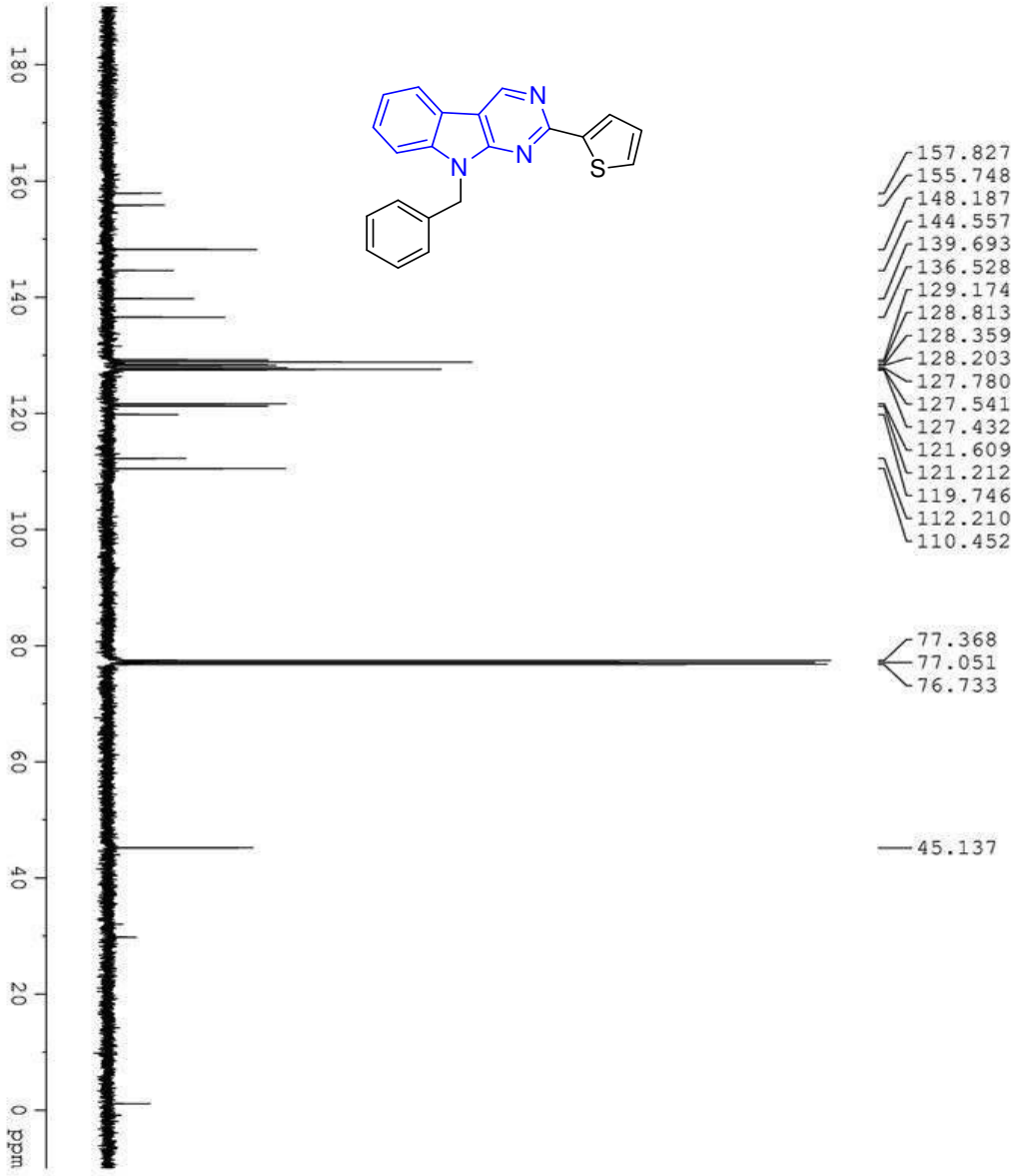
^{13}C NMR of 9-benzyl-2-(thiophen-2-yl)-9*H*-pyrimido[4,5-*b*]indole (3n)



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LCMS of 9-benzyl-2-(thiophen-2-yl)-9*H*-pyrimido[4,5-*b*]indole (3n)

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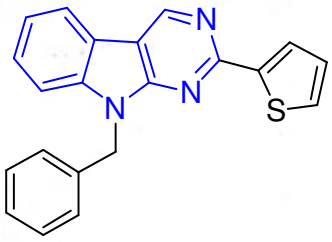
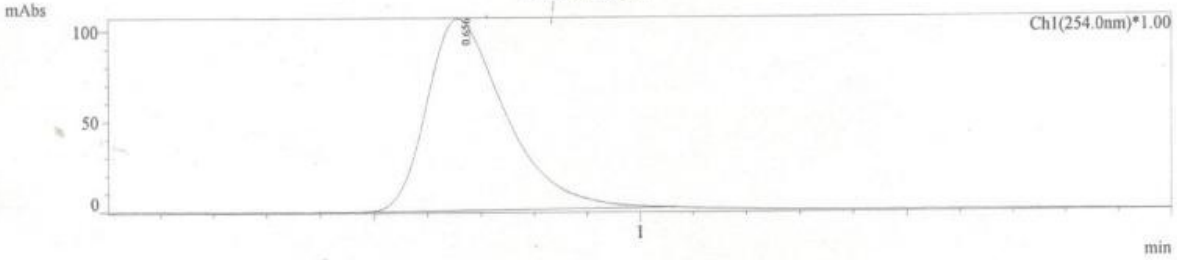


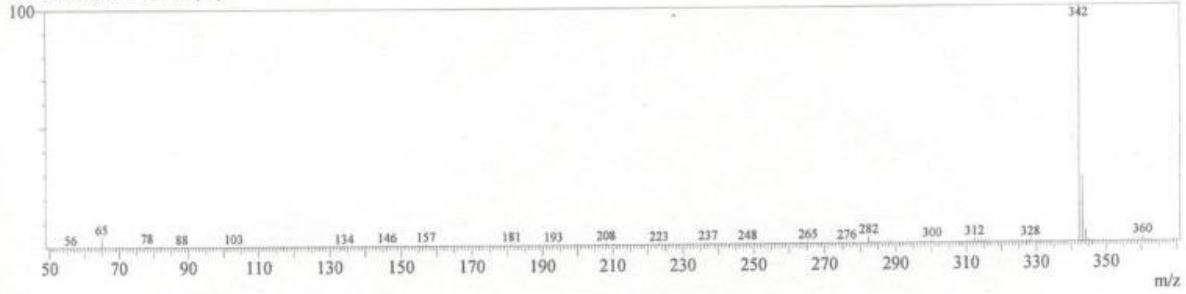
User : Admin
Sample : ASK-P13
Inj. Volume : 5.000
Data Name : C:\LCMSsolution\User\Data\ASK-P13-APCI-POS1.qld
Method Name : C:\LCMSsolution\User\Method\esi.qlm

LC Chromatogram



MS Spectrum

Line#:1 R.Time:0.779(Scan#:47) Positive
MassPeaks:181 BasePeak:342.30(3765285)
RawMode:Single 0.779(47)
BG Mode:Peak Start 0.527(32)



MS Peak Table

Peak#	R.Time	I.Time	F.Time	Area	Height	A/H	Mark	%Total	Name	Base m/z	Base Int.
1	0.779	0.527	1.060	55846893	3495512	15.97		100.00		342.30	3765285
				55846893	3495512			100.00			

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CHN Analysis of 9-benzyl-2-(thiophen-2-yl)-9*H*-pyrimido[4,5-*b*]indole (3n)

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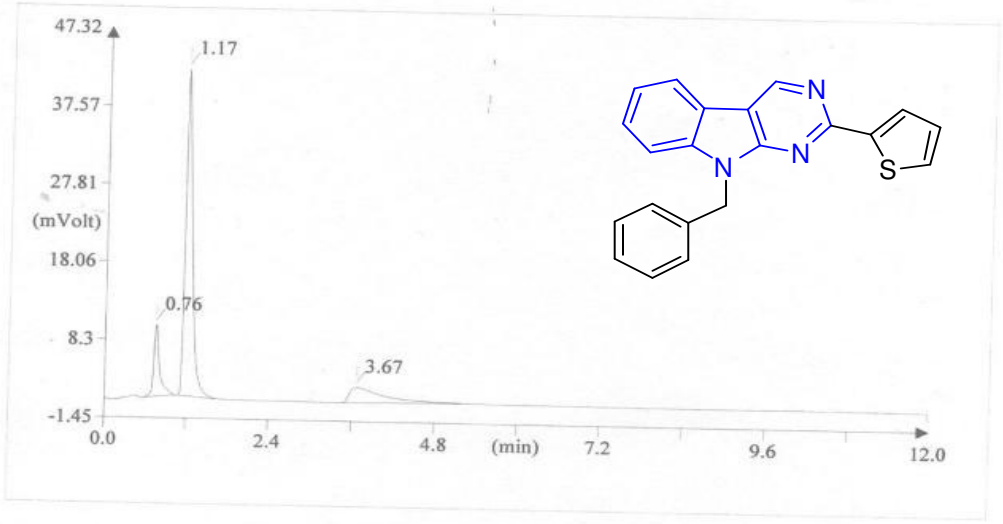
Method filename:	E:\Program Files\Thermo Finnigan\Eager 300 for EA1112\DATA\Sys_data_ex
Sample ID:	ASK-P13 (# 185)
Analysis type:	UnkNown
Chromatogram filename:	UNK-15092011-25.dat
Sample weight:	1.071



Element Name	Element %	Ret. Time
Nitrogen	12. 45	0. 76
Carbon	73. 96	1. 17
Hydrogen	4. 38	3. 67

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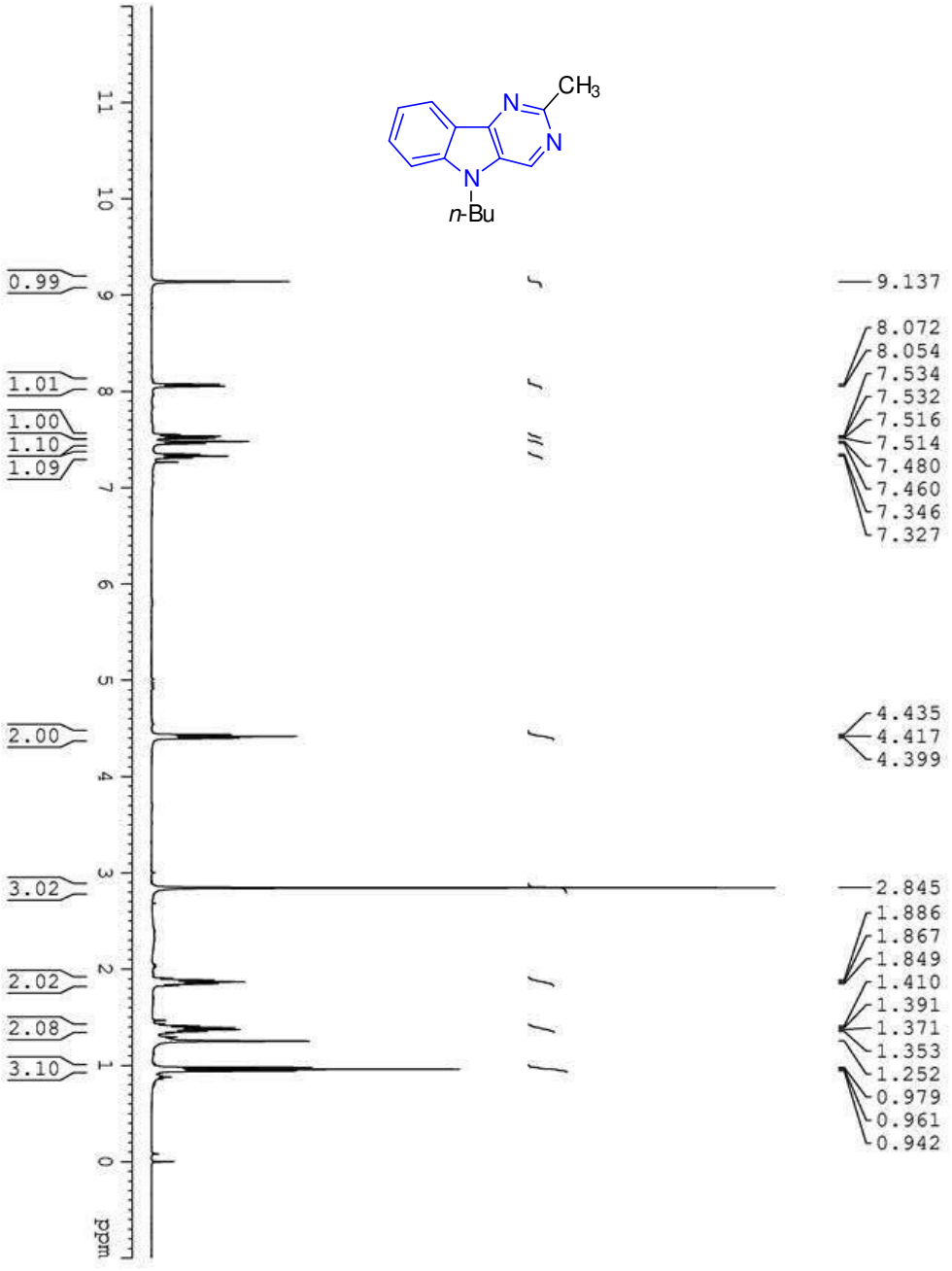
^{1}H NMR of 5-butyl-2-methyl-5*H*-pyrimido[5,4-*b*]indole (3o)



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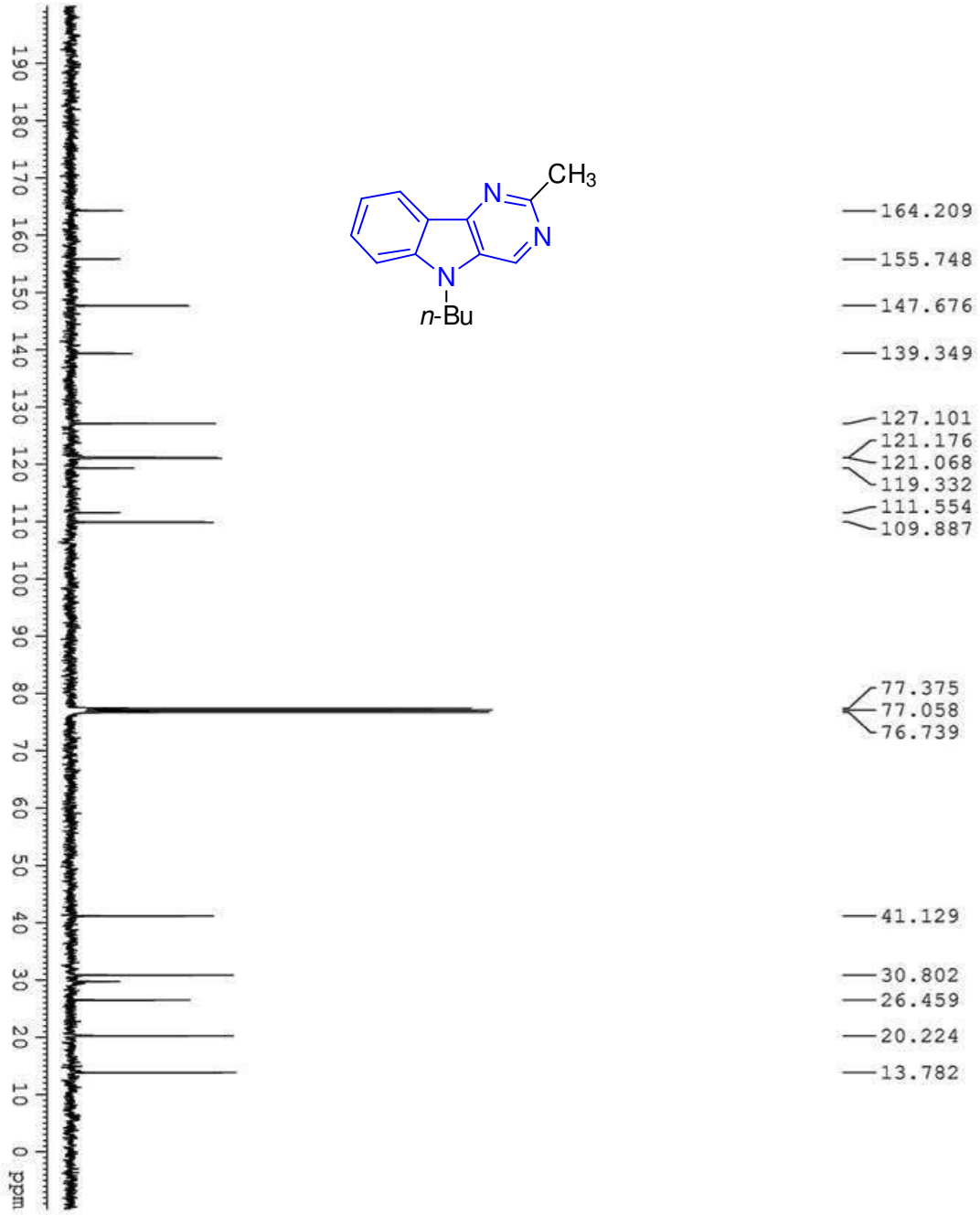
^{13}C NMR of 5-butyl-2-methyl-5*H*-pyrimido[5,4-*b*]indole (3o)



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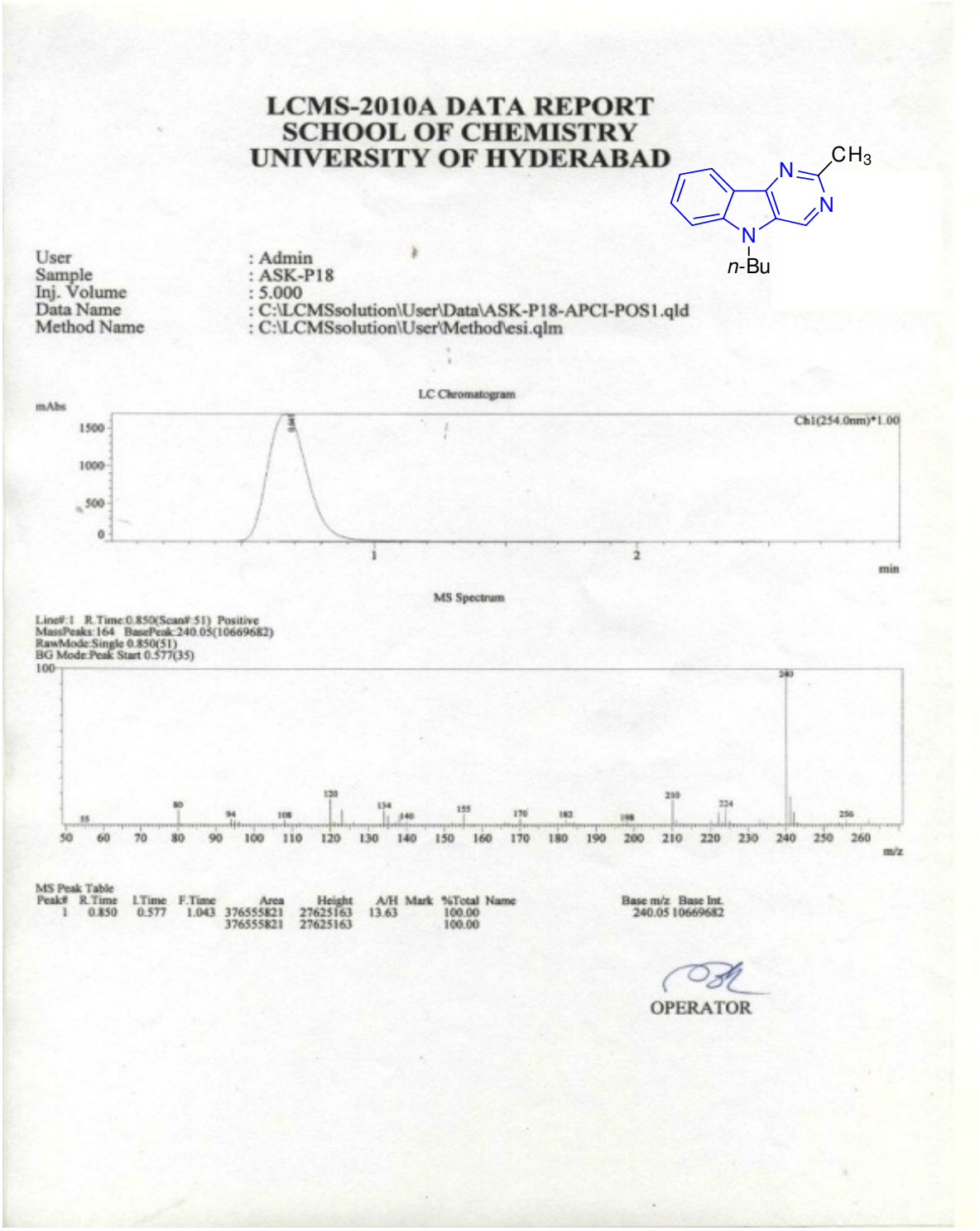
LCMS of 5-butyl-2-methyl-5*H*-pyrimido[5,4-*b*]indole (3o)



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CHN Analysis of 5-butyl-2-methyl-5*H*-pyrimido[5,4-*b*]indole (3o)

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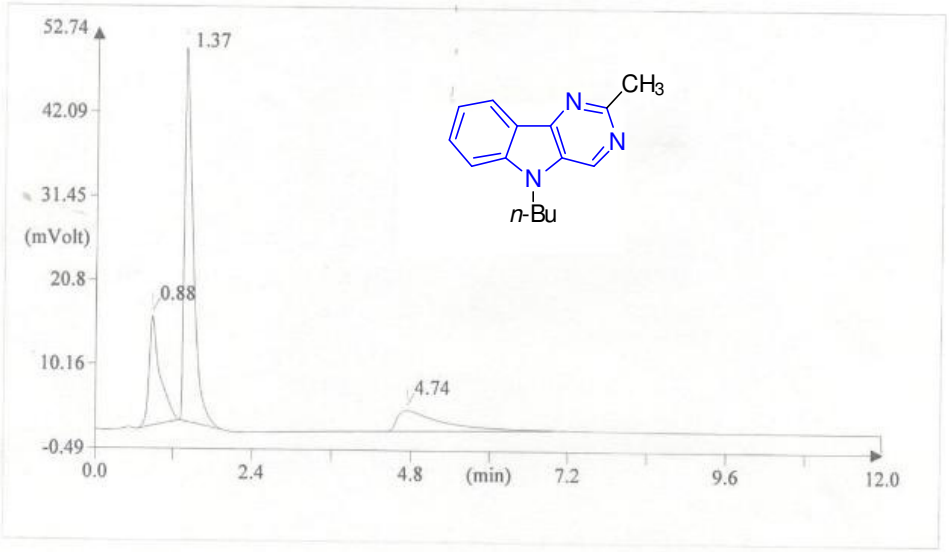
Method filename:	E:\Program Files\Thermo Finnigan\Eager 300 for EA1112\DATA\Sys_data_ex
Sample ID:	ASK-P18 (# 143)
Analysis type:	UnkNown
Chromatogram filename:	UNK-30012012-13.dat
Sample weight:	1.116



Element Name	Element %	Ret. Time
Nitrogen	17. 45	0. 88
Carbon	75. 36	1. 37
Hydrogen	7. 21	4. 74

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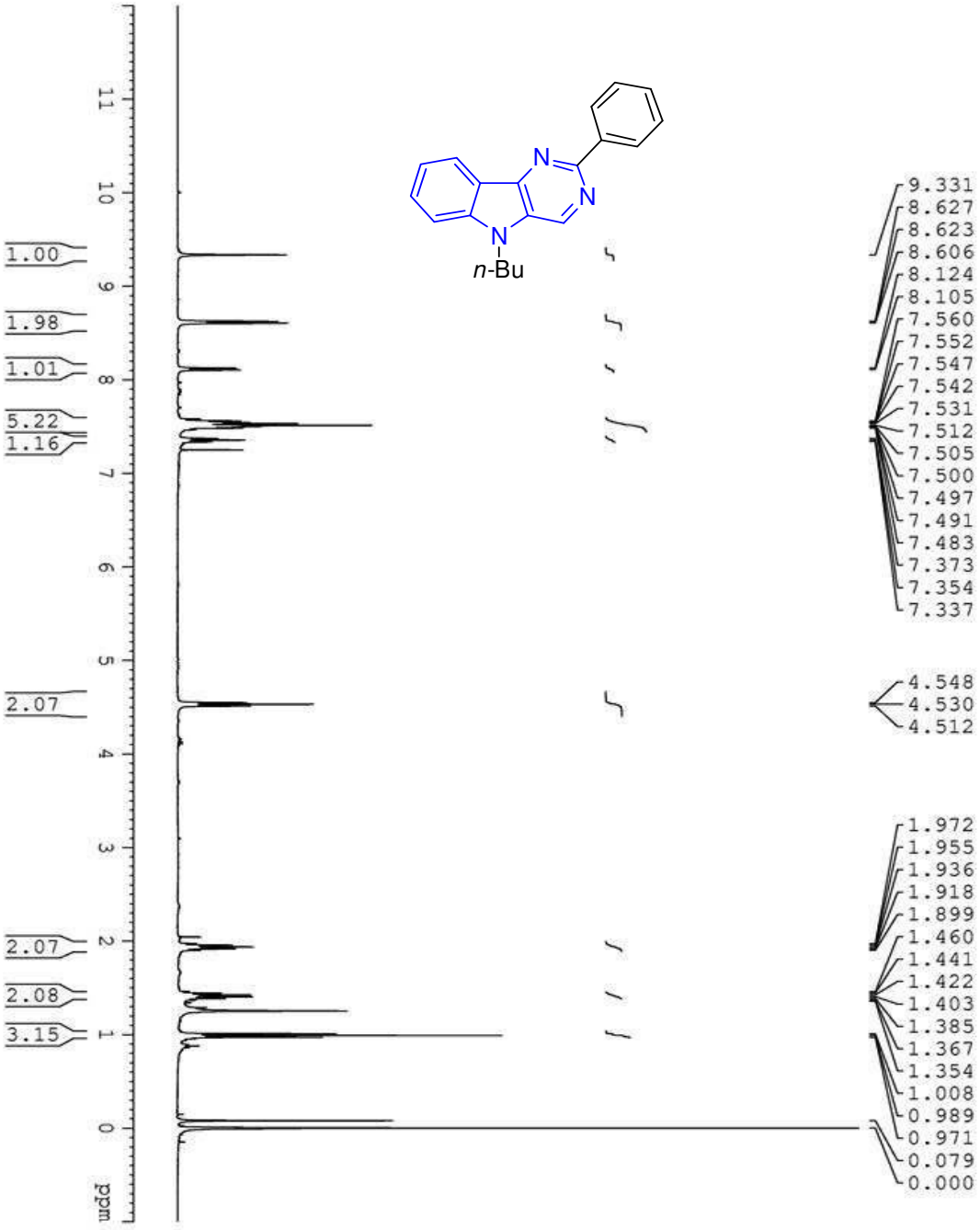
^{1}H NMR of 5-butyl-2-phenyl-5*H*-pyrimido[5,4-*b*]indole (3p)



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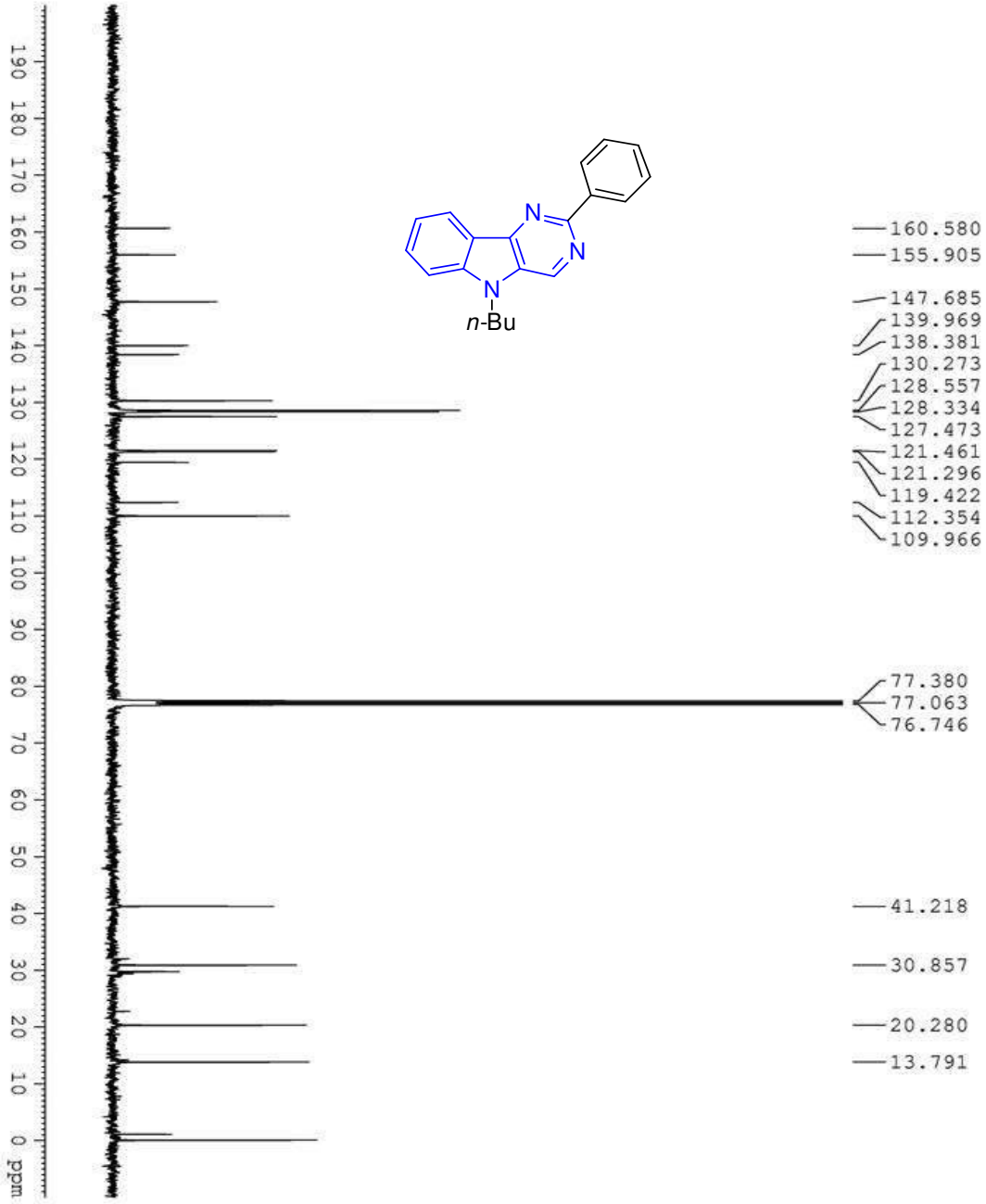
^{13}C NMR of 5-butyl-2-phenyl-5*H*-pyrimido[5,4-*b*]indole (3p)



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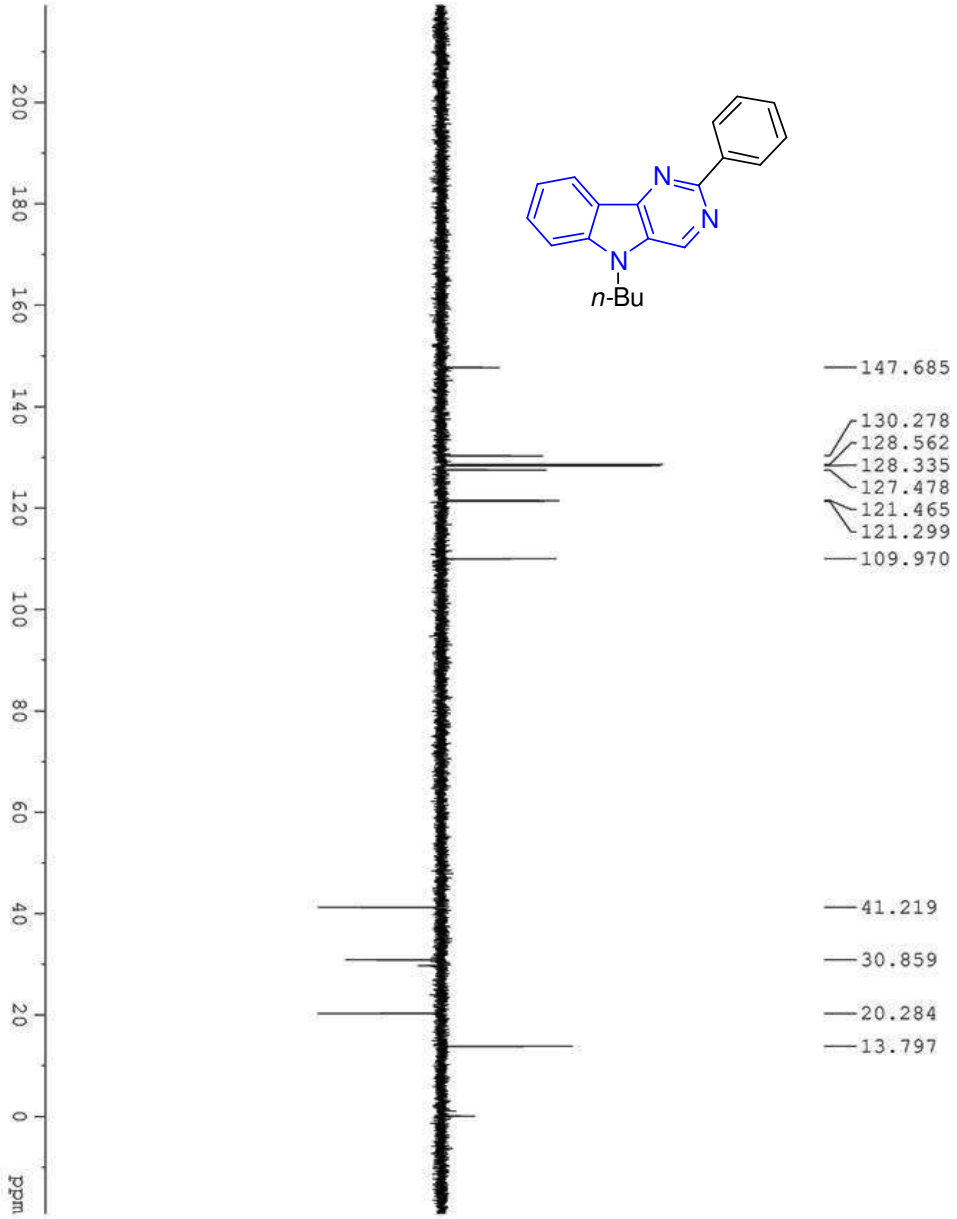
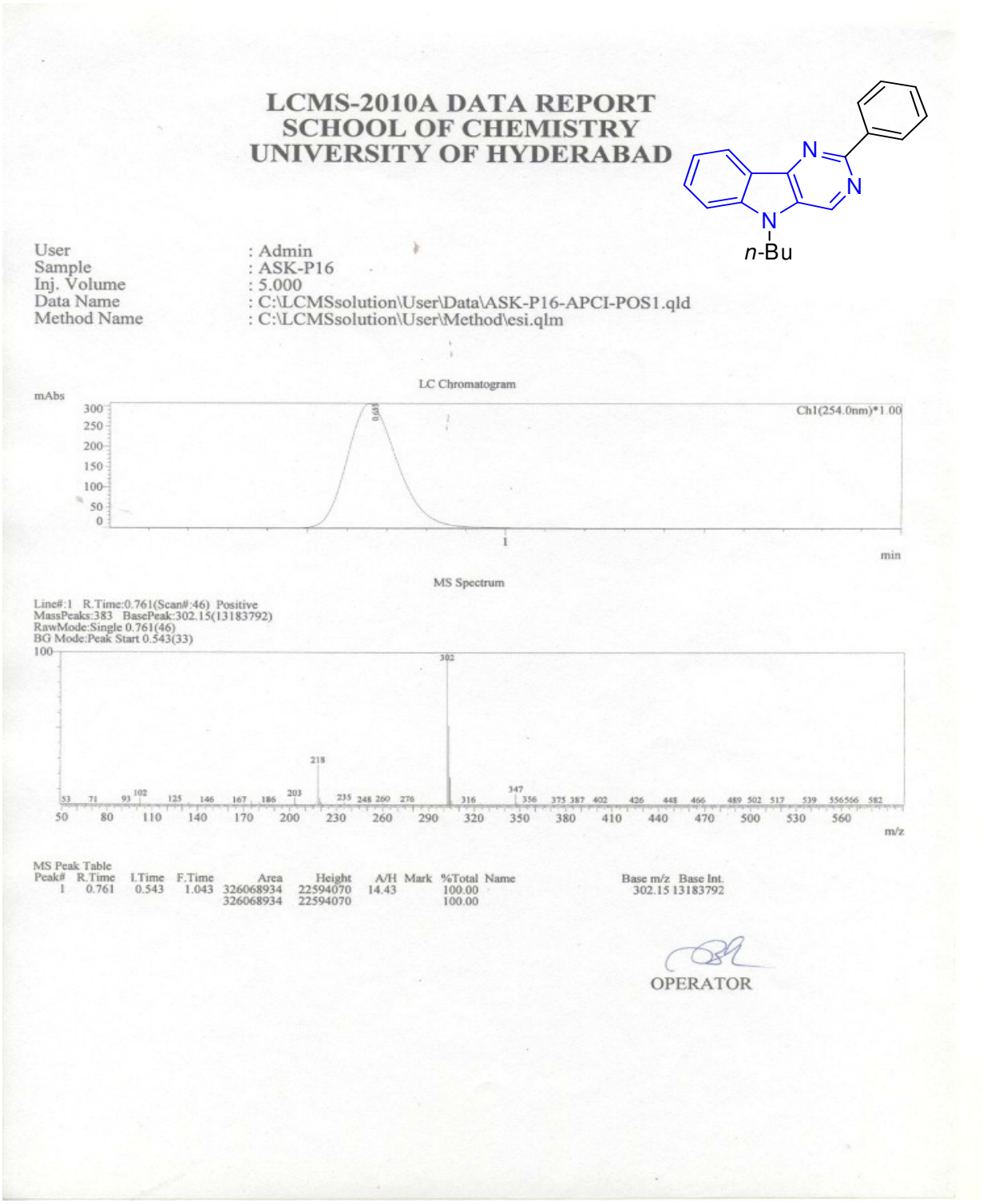
DEPT of 5-butyl-2-phenyl-5*H*-pyrimido[5,4-*b*]indole(3p)



LCMS of 5-butyl-2-phenyl-5*H*-pyrimido[5,4-*b*]indole(3p)



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CHN Analysis of 5-butyl-2-phenyl-5*H*-pyrimido[5,4-*b*]indole(3p)

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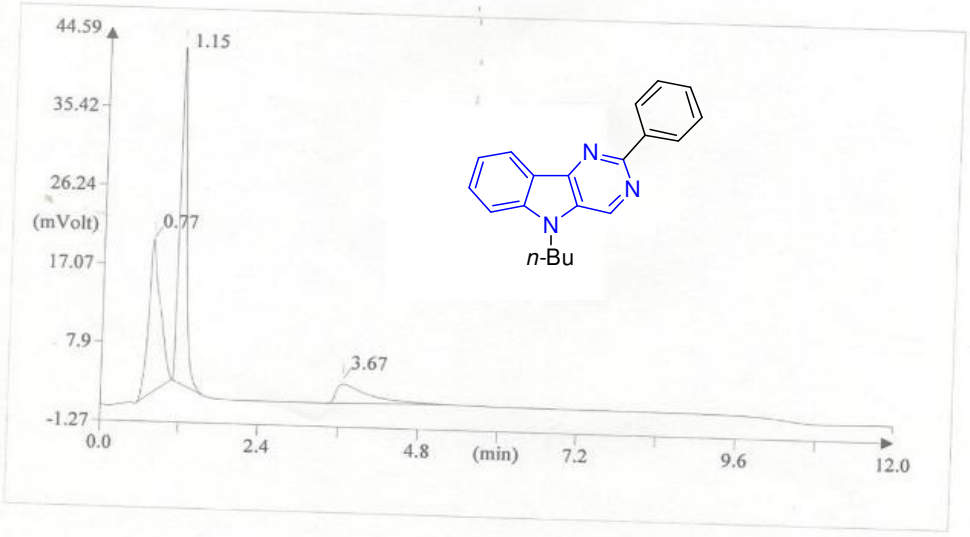
Method filename:	E:\Program Files\Thermo Finnigan\Eager 300 for EA1112\DATA\Sys_data_ex
Sample ID:	ASK-P16 (# 151)
Analysis type:	UnkNown
Chromatogram filename:	UNK-30012012-21.dat
Sample weight:	1.116



Element Name	Element %	Ret. Time
Nitrogen	13. 81	0. 77
Carbon	79. 86	1. 15
Hydrogen	6. 31	3. 67

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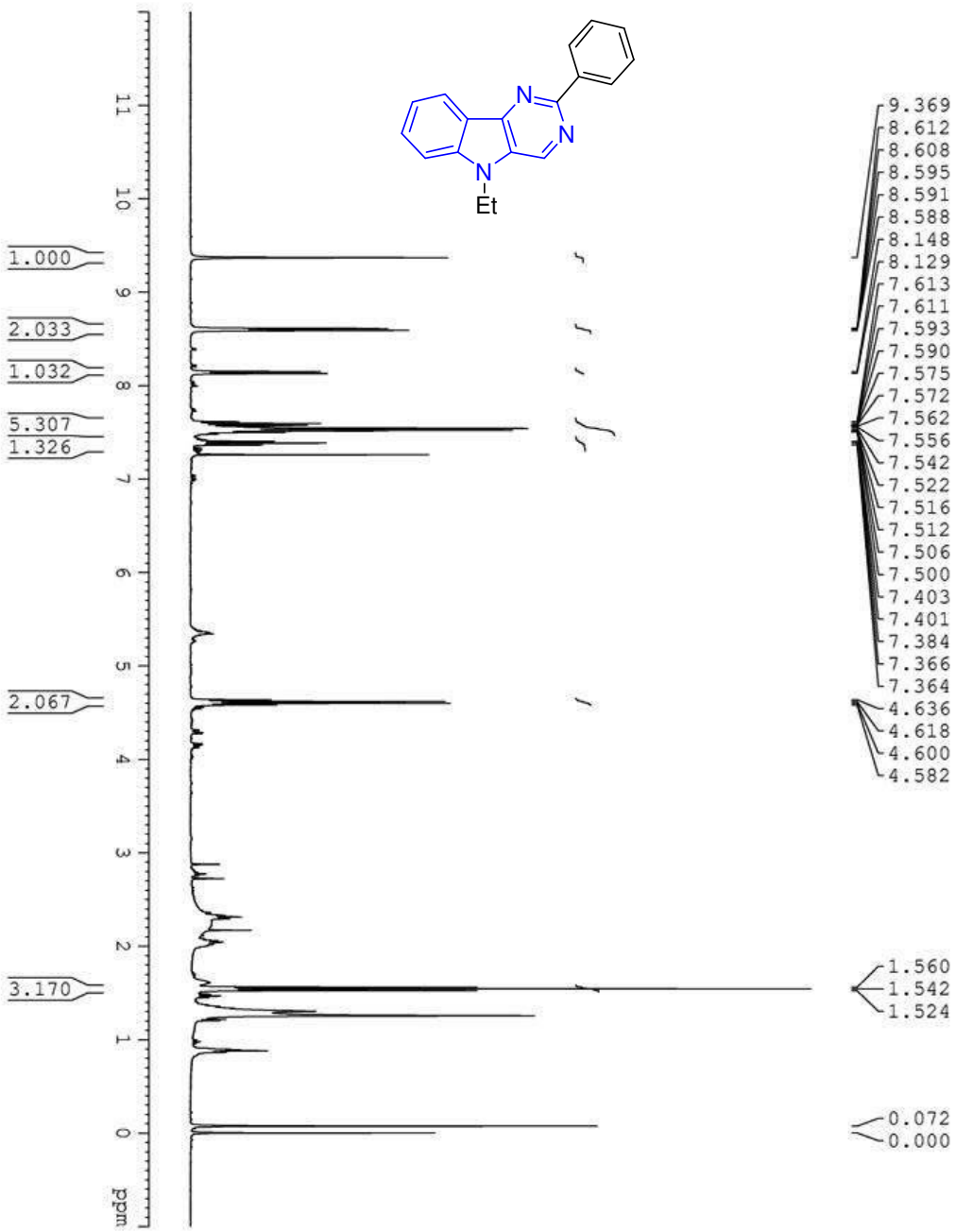
^{1}H NMR of 5-ethyl-2-phenyl-5*H*-pyrimido[5,4-*b*]indole (3q)



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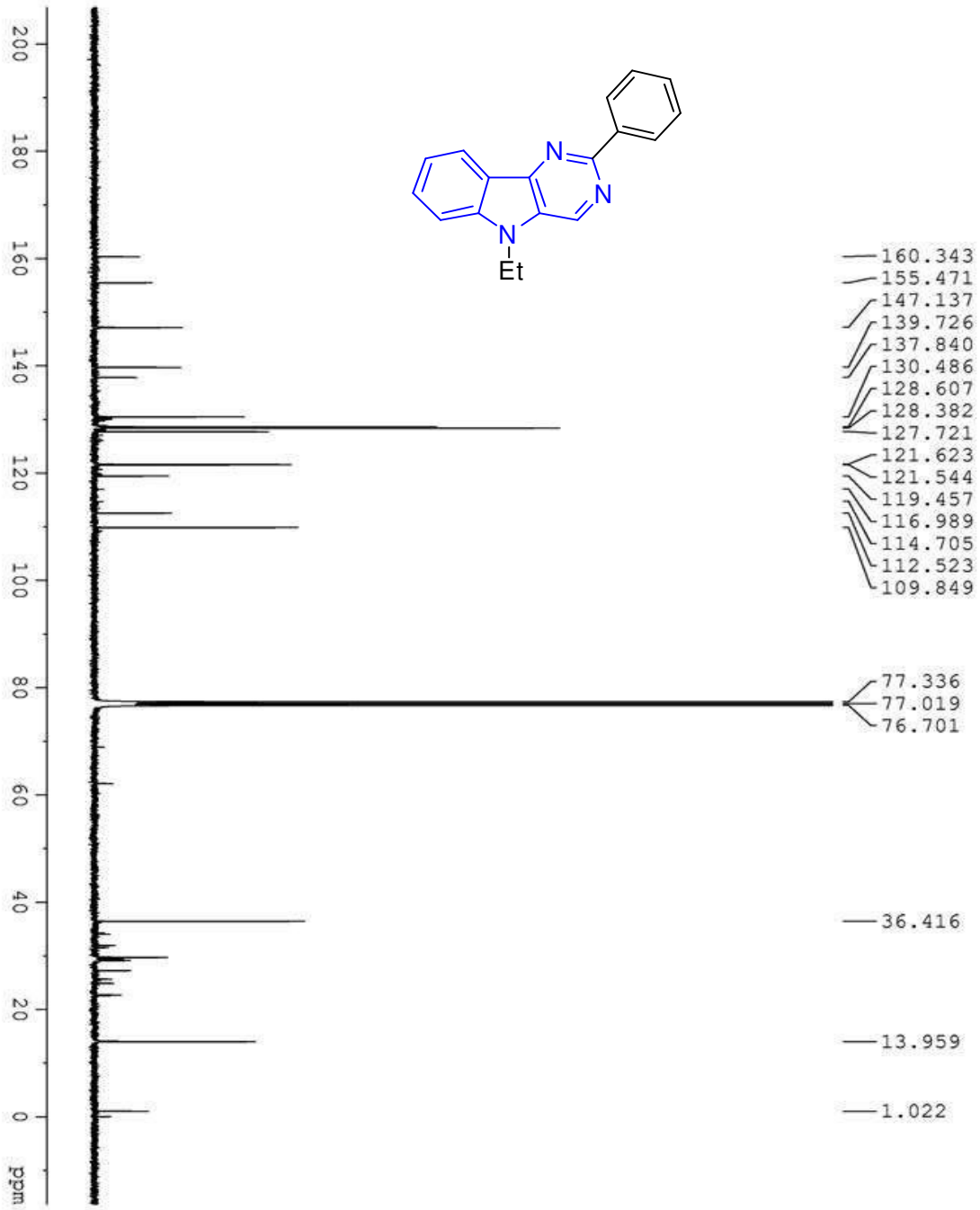
^{13}C NMR of 5-ethyl-2-phenyl-5*H*-pyrimido[5,4-*b*]indole (3q)



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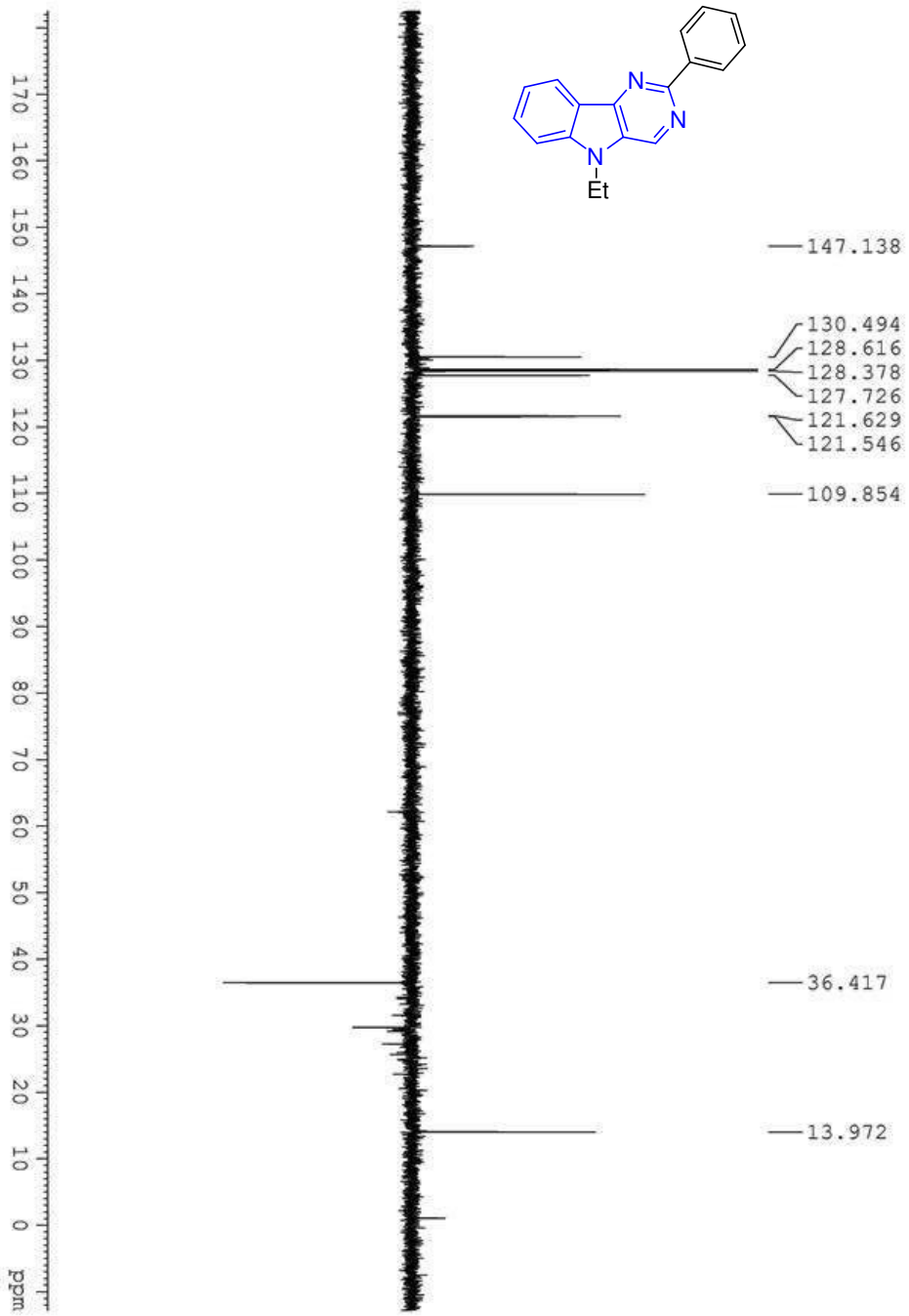
DEPT of 5-ethyl-2-phenyl-5*H*-pyrimido[5,4-*b*]indole (3q)



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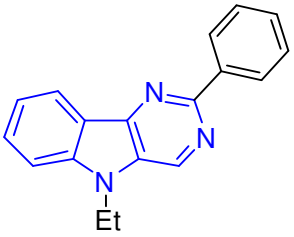
LCMS of 5-ethyl-2-phenyl-5*H*-pyrimido[5,4-*b*]indole (3q)



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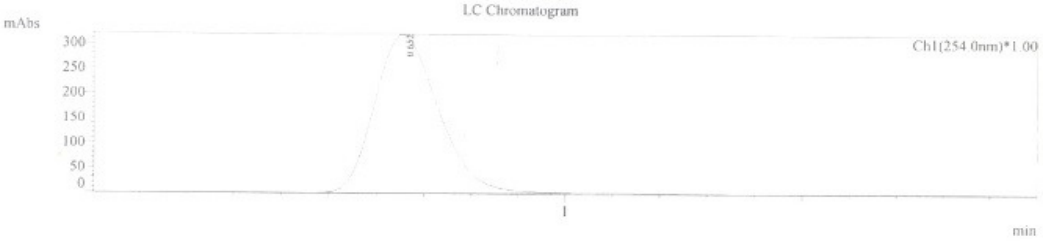
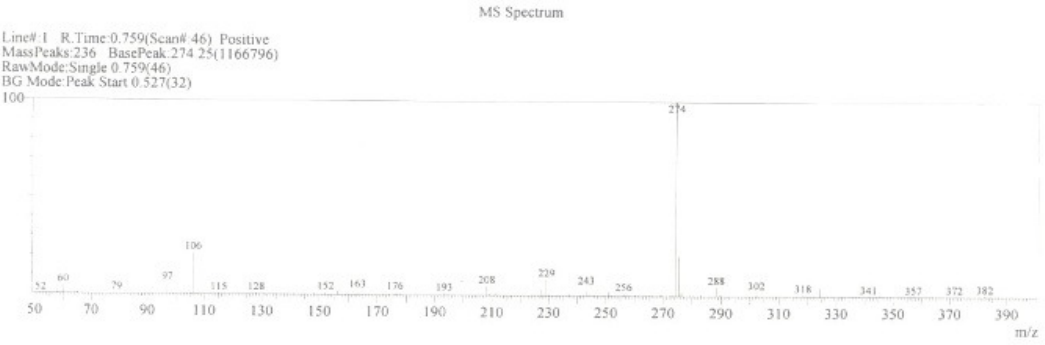
User : Admin
Sample : ASK-P15
Inj. Volume : 5.000
Data Name : C:\LCMSsolution\User\Data\ASK-P15-APCI-POS1.qld
Method Name : C:\LCMSsolution\User\Method\esi.qlm





MS Peak Table

Peak#	R.Time	I.Time	F.Time	Area	Height	A/H	Mark	%Total	Name	Base m/z	Base Int.
1	0.759	0.527	1.043	24019201	1811380	13.26		100.00		274.25	1166796
				24019201	1811380			100.00			

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CHN Analysis of 5-ethyl-2-phenyl-5*H*-pyrimido[5,4-*b*]indole (3q)

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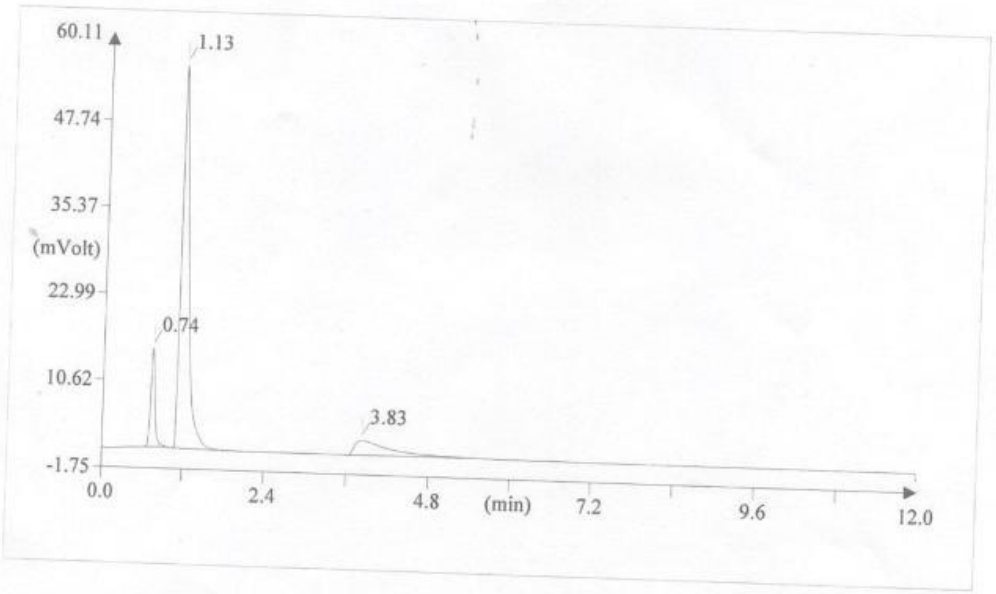
Method filename:	E:\Program Files\Thermo Finnigan\Eager 300 for EA1112\DATA\Sys_data_ex
Sample ID:	ASK-P15 (# 135)
Analysis type:	UnkNown
Chromatogram filename:	UNK-30012012-5.dat
Sample weight:	1.126



Element Name	Element %	Ret. Time
Nitrogen	15. 26	0. 74
Carbon	79. 25	1. 13
Hydrogen	5. 61	3. 83

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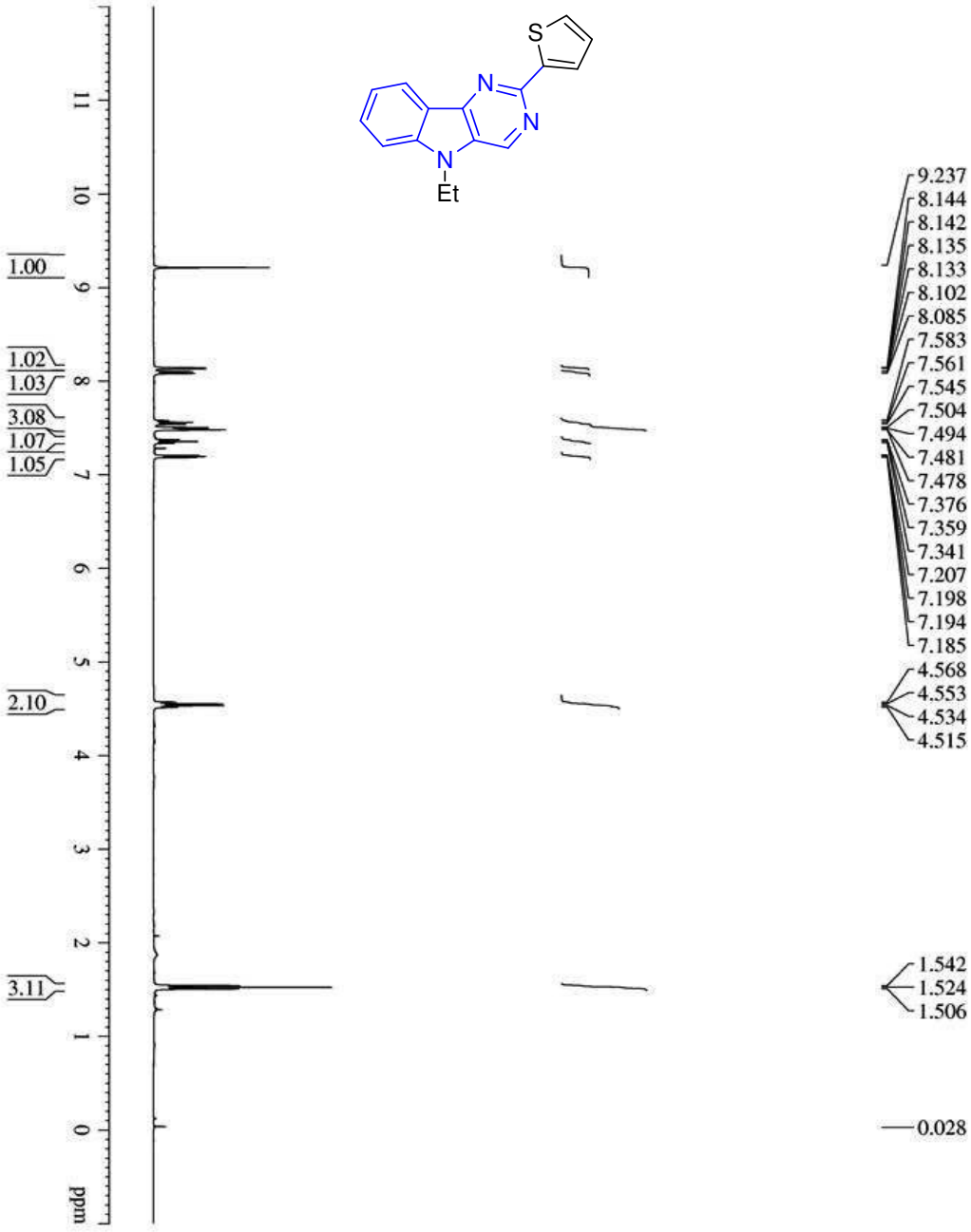
^{1}H NMR of 5-ethyl-2-(thiophen-2-yl)-5*H*-pyrimido[5,4-*b*]indole (3r)



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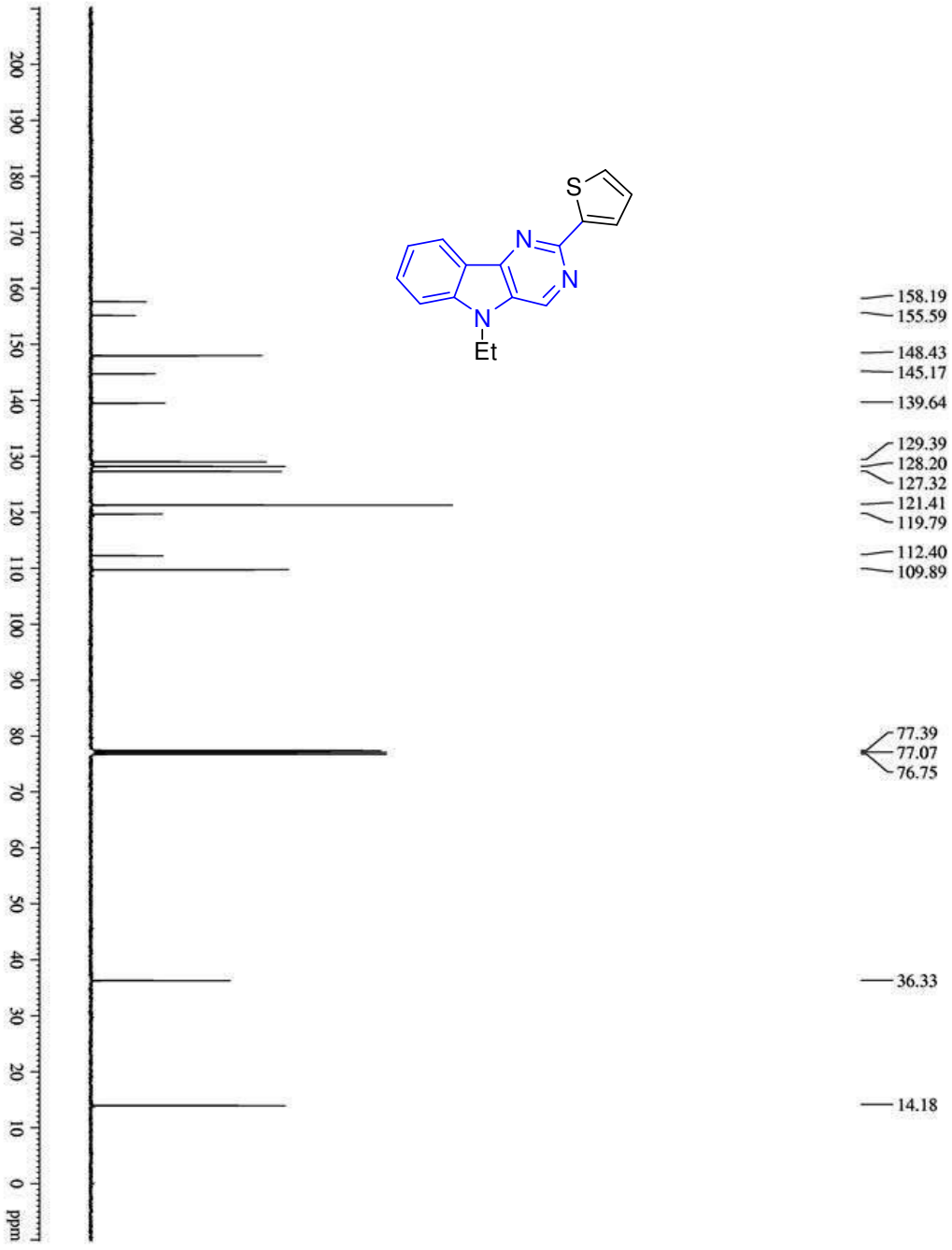
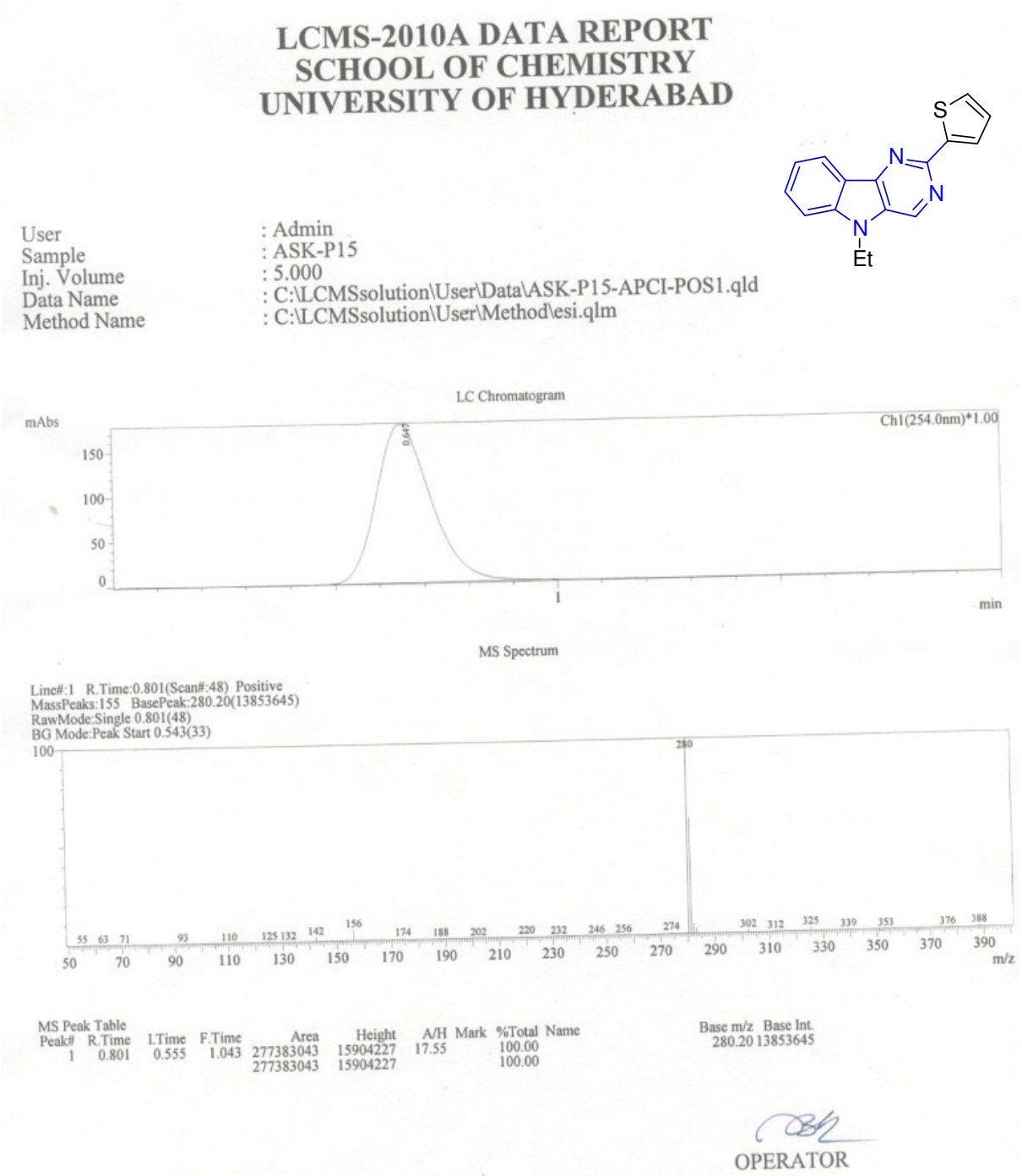
^{13}C NMR of 5-ethyl-2-(thiophen-2-yl)-5*H*-pyrimido[5,4-*b*]indole (3r)



LCMS of 5-ethyl-2-(thiophen-2-yl)-5*H*-pyrimido[5,4-*b*]indole (3r)



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CHN Analysis of 5-ethyl-2-(thiophen-2-yl)-5*H*-pyrimido[5,4-*b*]indole (3r)

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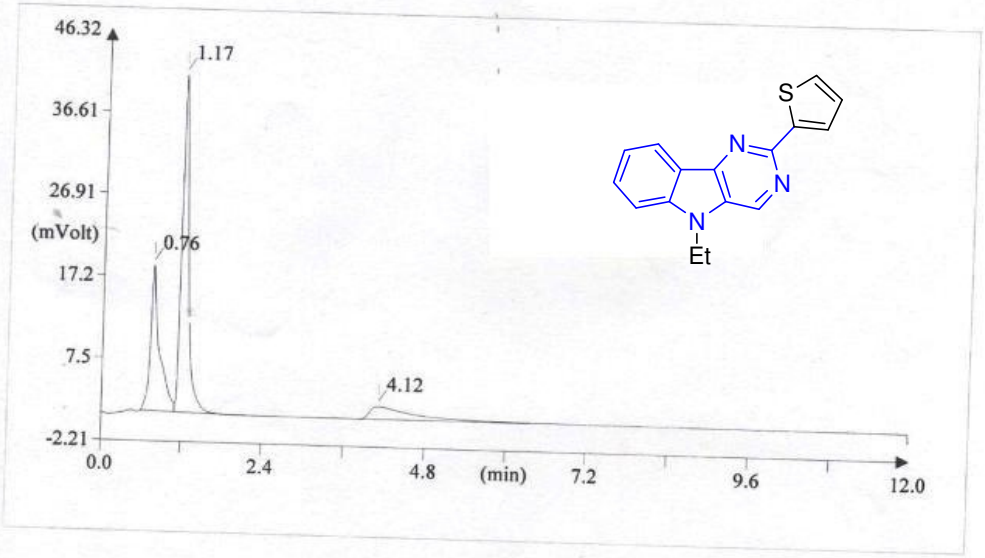
Method filename:	E:\Program Files\Thermo Finnigan\Eager 300 for EA1112\DATA\Sys_data_ex
Sample ID:	ASK-P20 (# 145)
Analysis type:	UnkNown
Chromatogram filename:	UNK-30012012-15.dat
Sample weight:	1.113



Element Name	Element %	Ret. Time
Nitrogen	15. 16	0. 76
Carbon	68. 62	1. 17
Hydrogen	4. 61	4. 12

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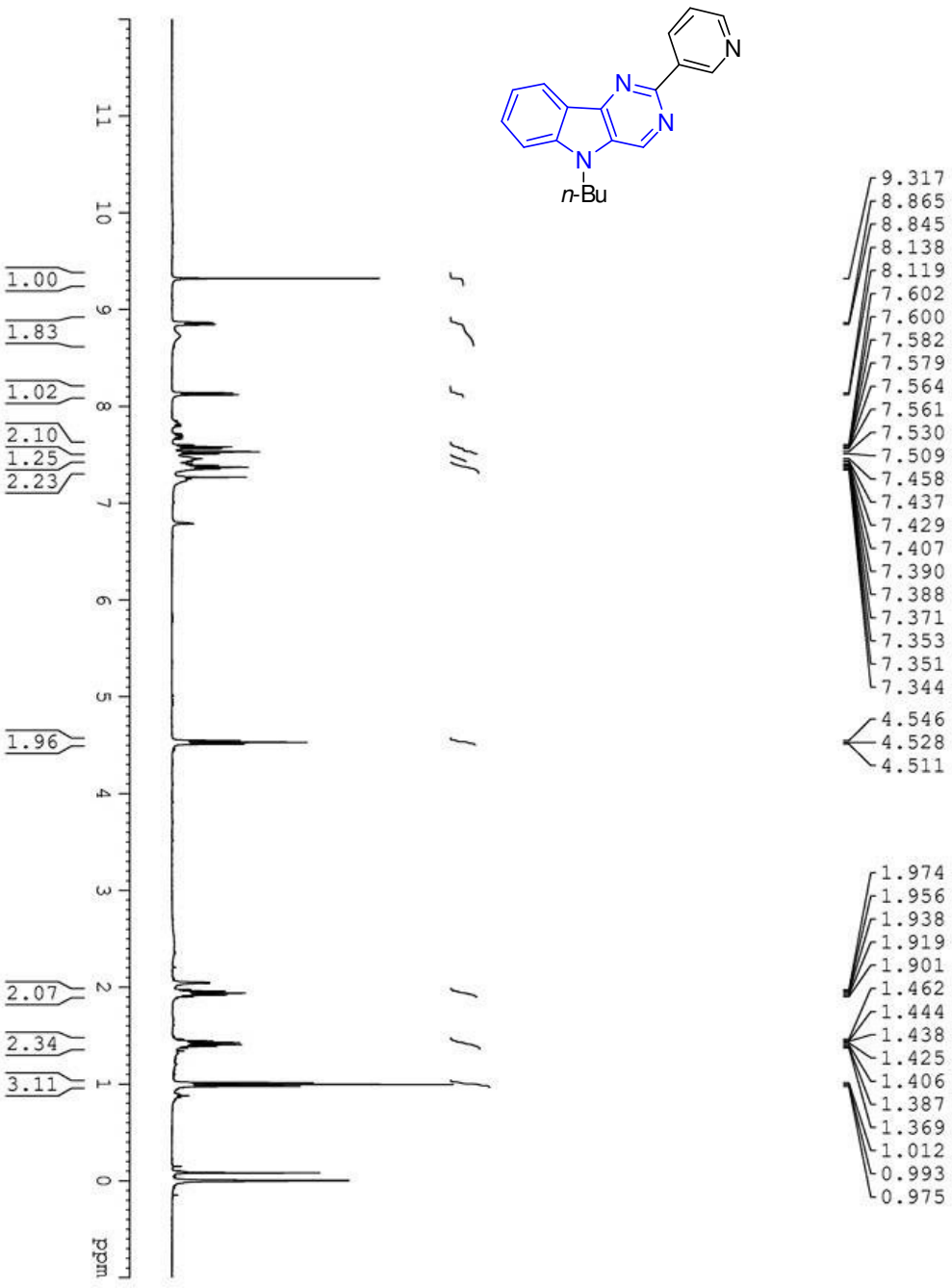
^{1}H NMR of 5-butyl-2-(pyridin-3-yl)-5*H*-pyrimido[5,4-*b*]indole (3s)



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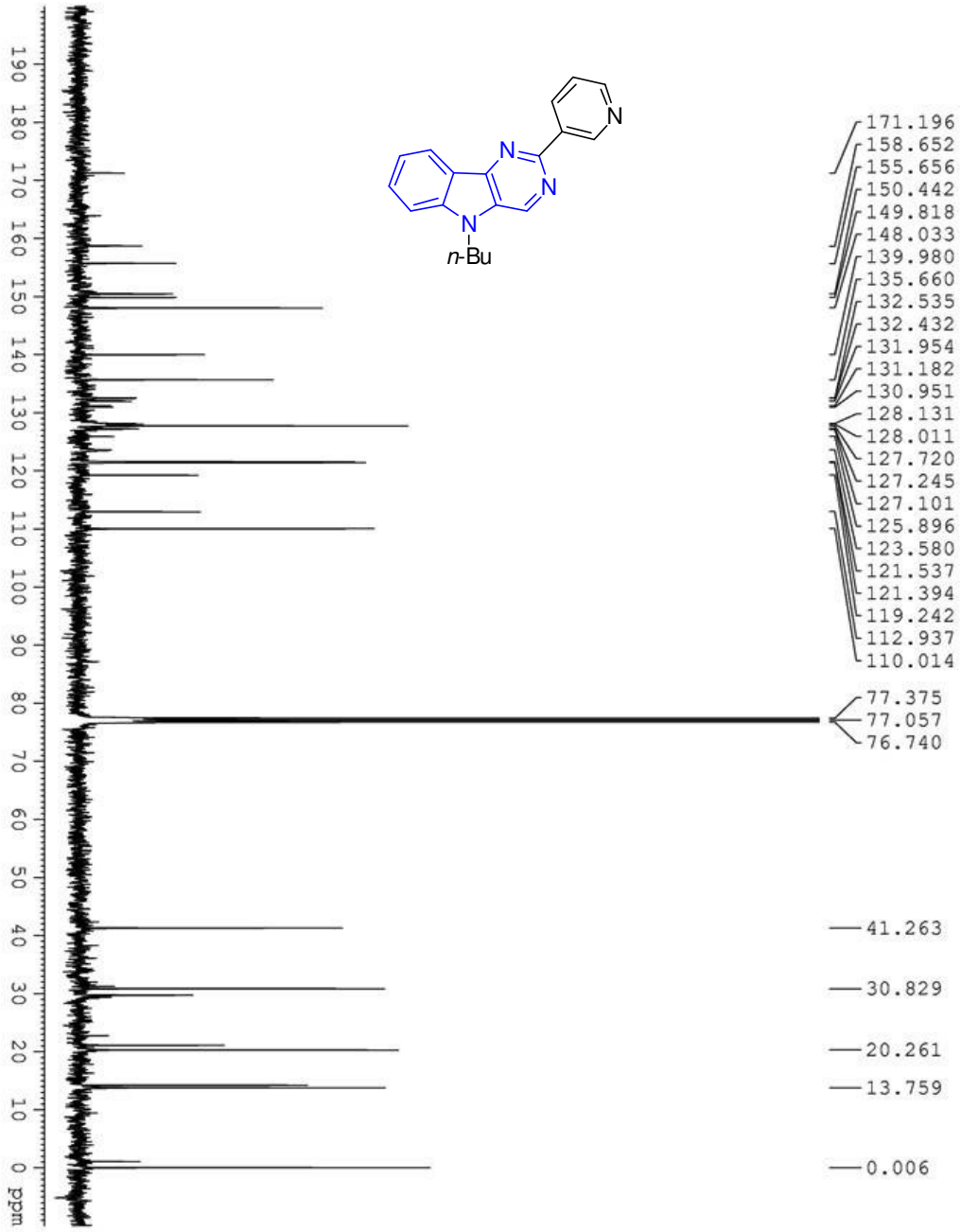
^{13}C NMR of 5-butyl-2-(pyridin-3-yl)-5*H*-pyrimido[5,4-*b*]indole (3s)



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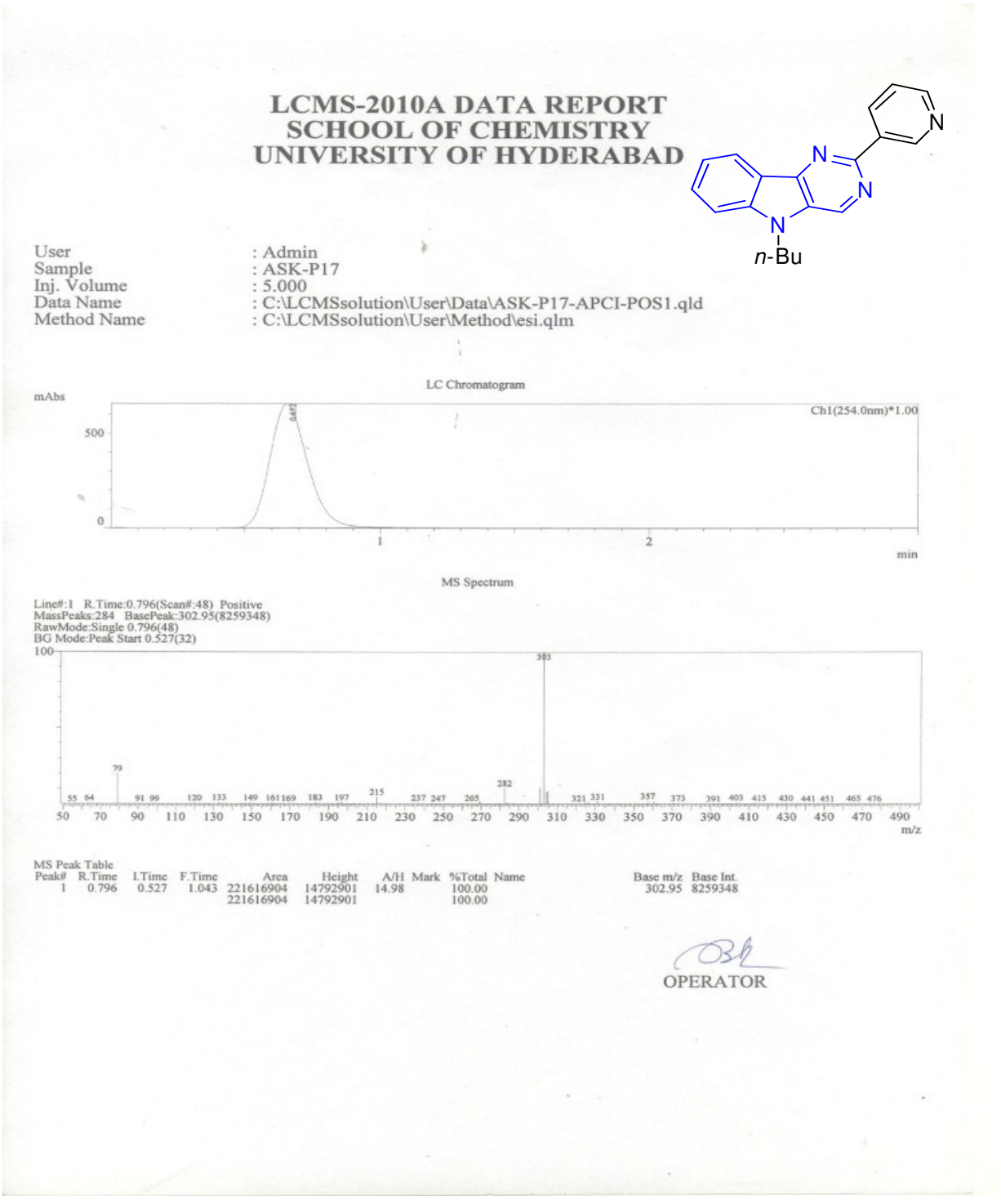
LCMS of 5-butyl-2-(pyridin-3-yl)-5*H*-pyrimido[5,4-*b*]indole (3s)



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CHN Analysis of 5-butyl-2-(pyridin-3-yl)-5*H*-pyrimido[5,4-*b*]indole (3s)

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Method filename:	E:\Program Files\Thermo Finnigan\Eager 300 for EA1112\DATA\Sys_data_ex
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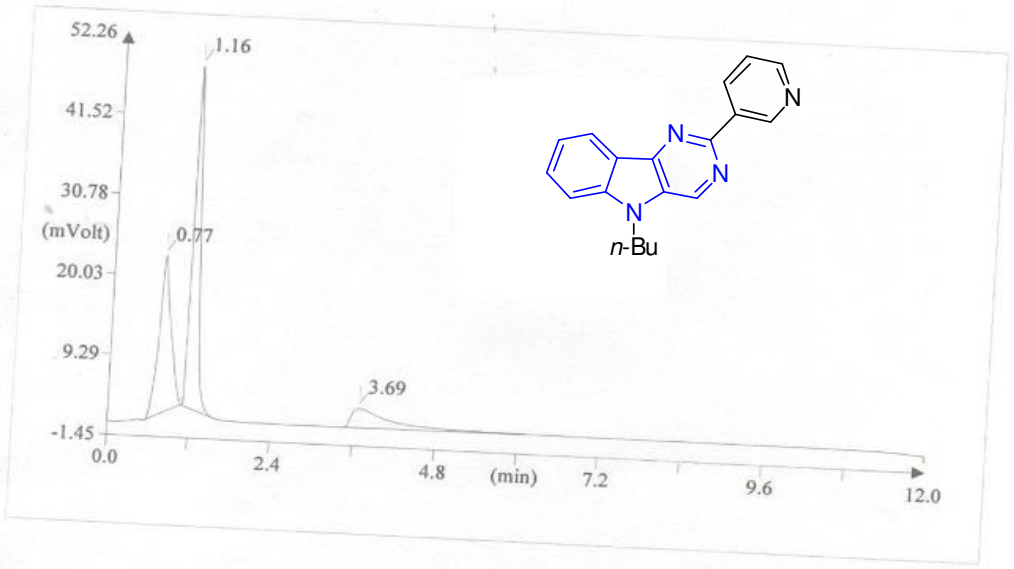
Analysis type:	UnkNown
Chromatogram filename:	UNK-30012012-20.dat
Sample weight:	1.143



Element Name	Element %	Ret. Time
Nitrogen	18. 45	0. 77
Carbon	75. 38	1. 16
Hydrogen	6. 12	3. 69

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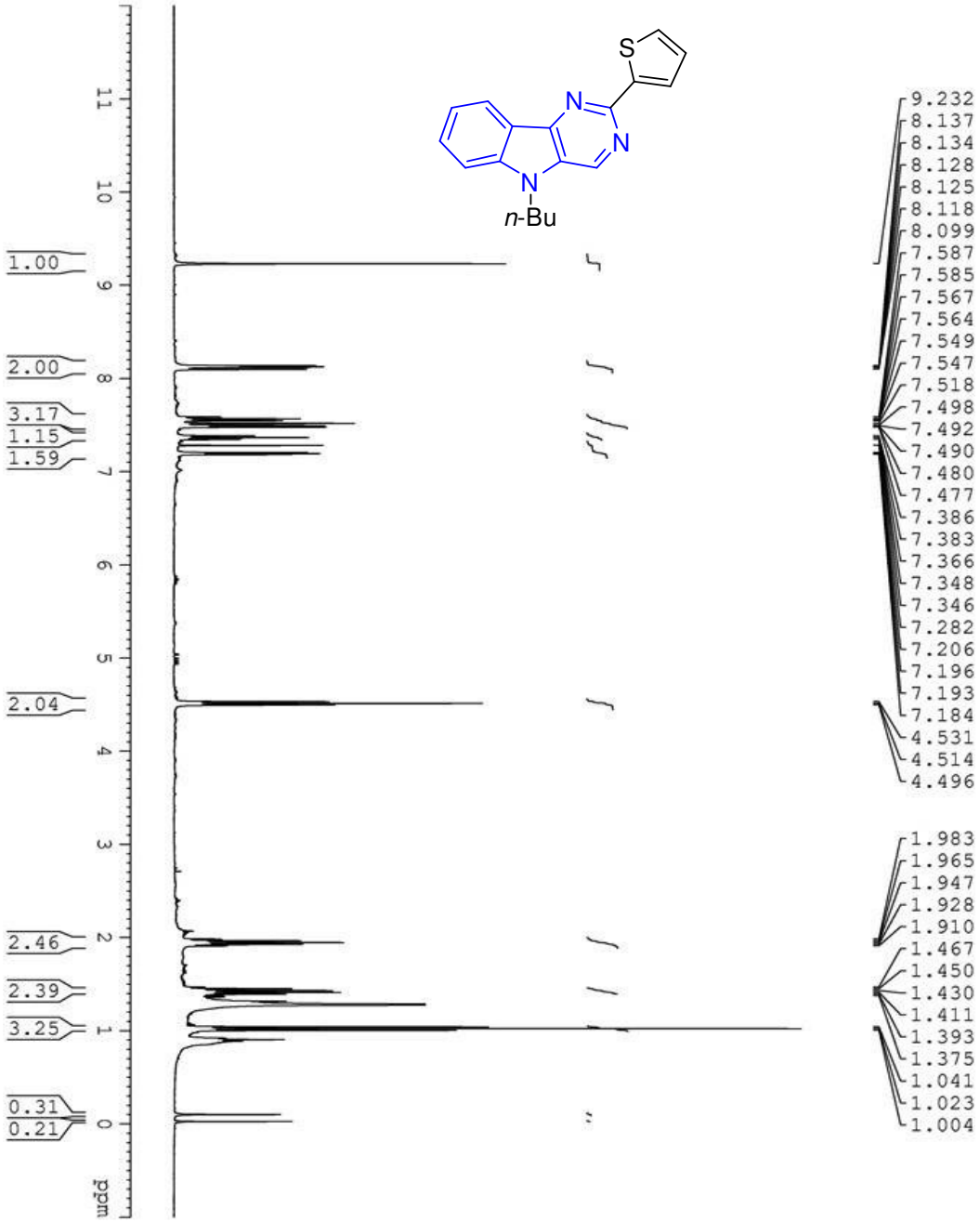
^{1}H NMR of 5-butyl-2-(thiophen-2-yl)-5*H*-pyrimido[5,4-*b*]indole (3t)



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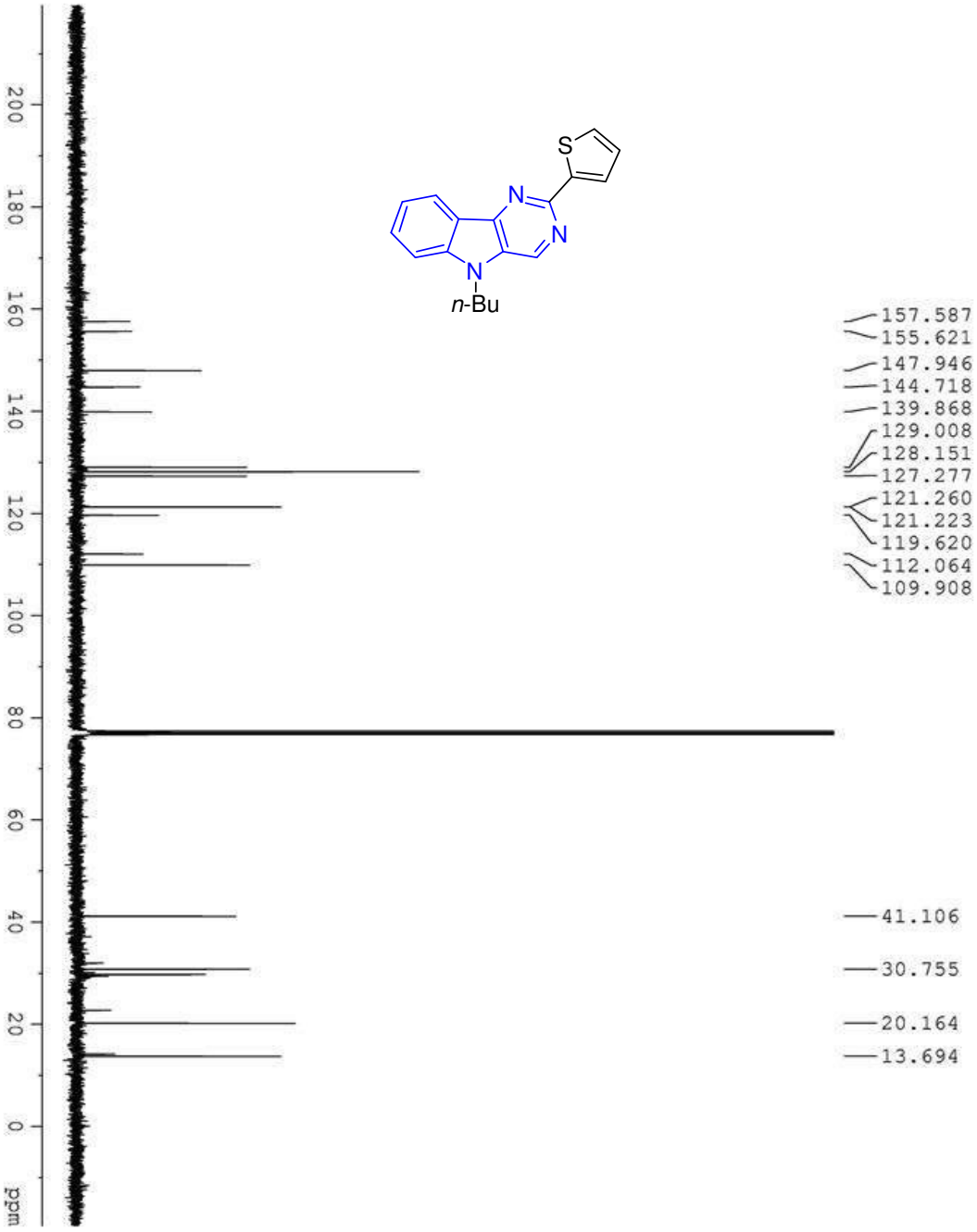
^{13}C NMR of 5-butyl-2-(thiophen-2-yl)-5*H*-pyrimido[5,4-*b*]indole (3t)



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LCMS of 5-butyl-2-(thiophen-2-yl)-5*H*-pyrimido[5,4-*b*]indole (3t)

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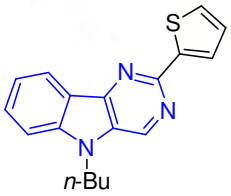

User : Admin
Sample : ASK-4T
Inj. Volume : 5.000
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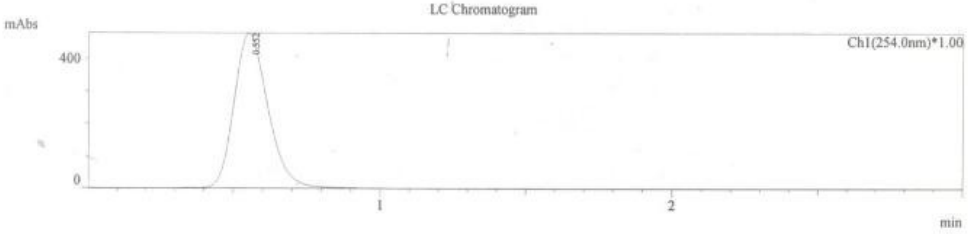
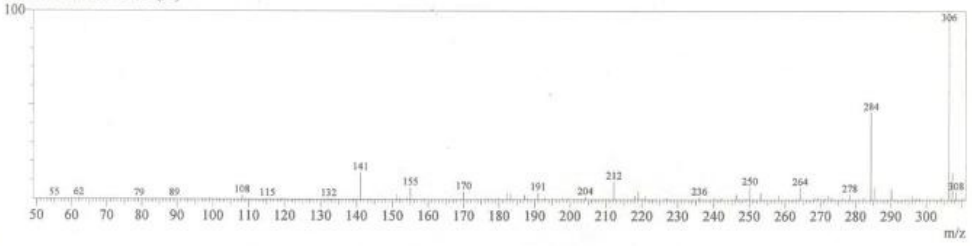
LC Chromatogram



MS Spectrum

Line#:1 R.Time:0.804(Scan#:49) Negative
MassPeaks:111 BasePeak:306.30(241873)
RawMode:Single 0.804(49)
BG Mode:Peak Start 0.527(32)



MS Peak Table

Peak#	R.Time	I.Time	F.Time	Area	Height	A/H	Mark	%Total	Name	Base m/z	Base Int.
1	0.804	0.527	1.243	146167518	6694691	21.83		100.00		306.30	241873
				146167518	6694691			100.00			

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CHN Analysis of 5-butyl-2-(thiophen-2-yl)-5*H*-pyrimido[5,4-*b*]indole (3t)

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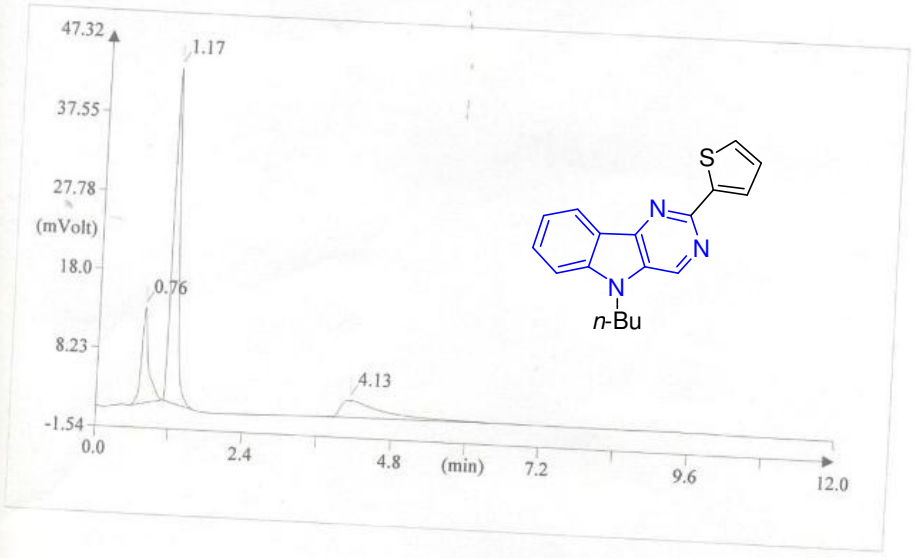
Method filename:	E:\Program Files\Thermo Finnigan\Eager 300 for EA1112\DATA\Sys_data_ex
Sample ID:	ASK-4T (# 102)
Analysis type:	UnkNown
Chromatogram filename:	UNK-24012012-2.dat
Sample weight:	.981



Element Name	Element %	Ret. Time
Nitrogen	13. 76	0. 76
Carbon	70. 25	1. 17
Hydrogen	5. 51	4. 13

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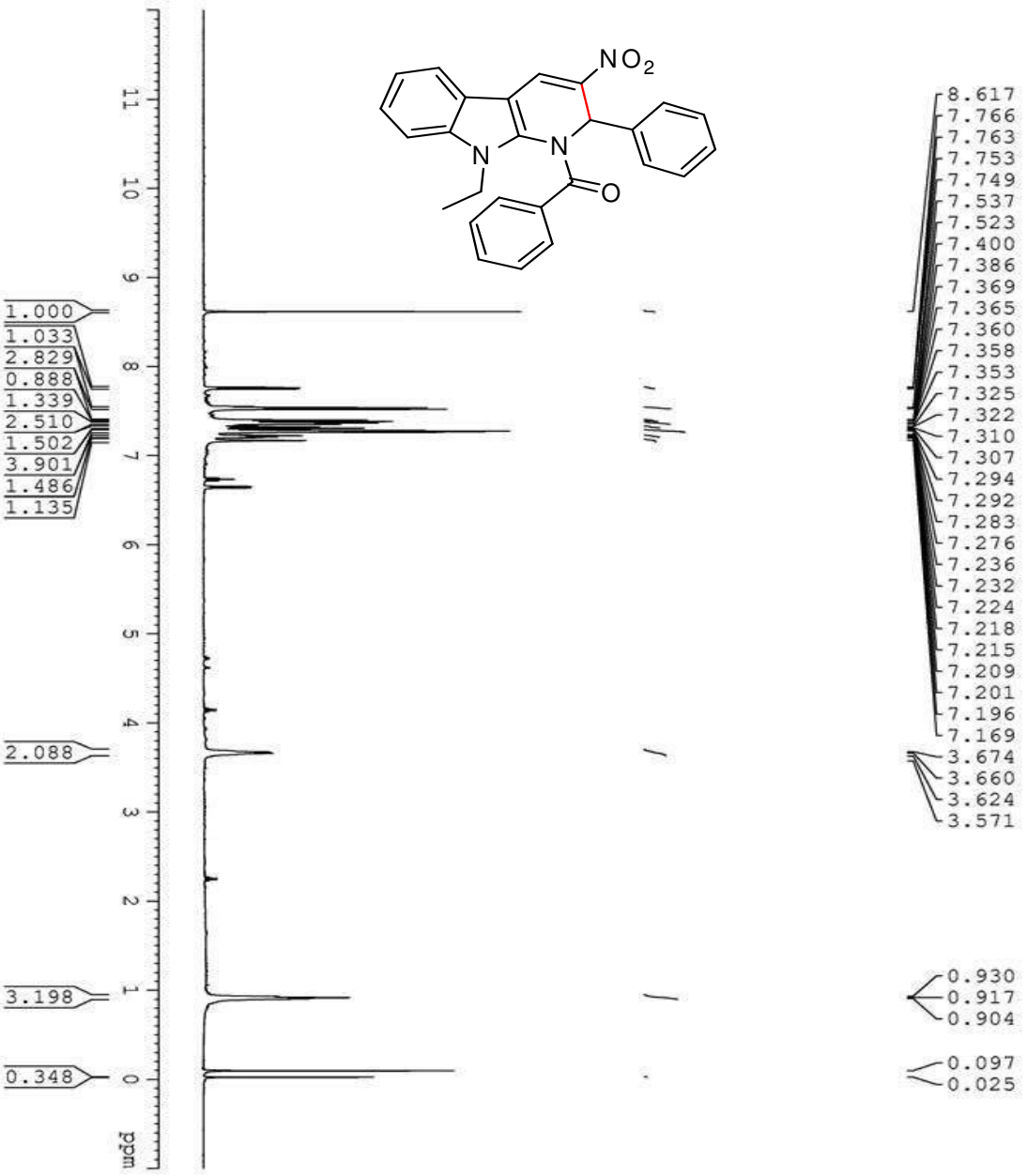
^{1}H NMR of (9-ethyl-3-nitro-2-phenyl-2,9-dihydro-1*H*-pyrido[2,3-b]indol-1-yl)(phenyl)methanone (4a)



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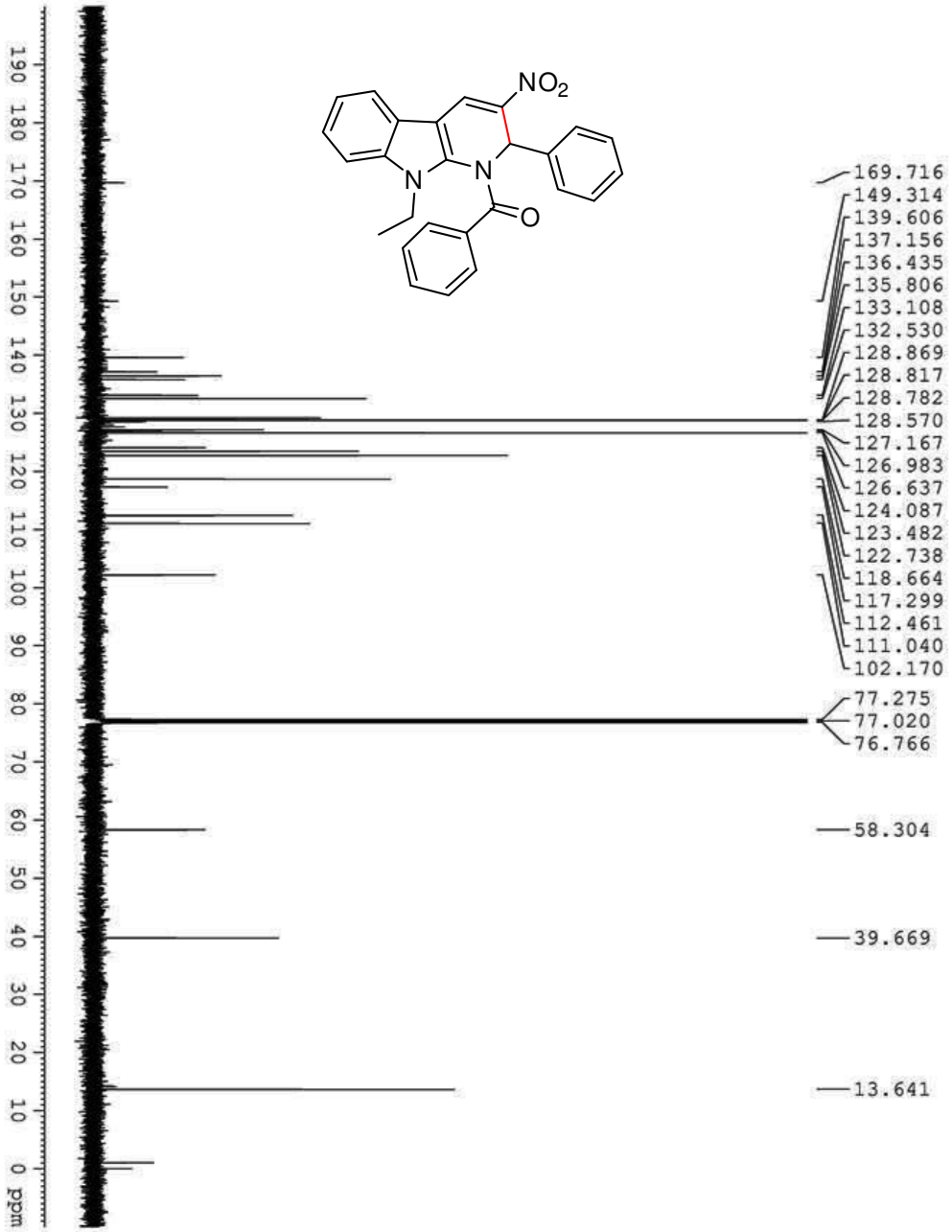
^{13}C NMR of (9-ethyl-3-nitro-2-phenyl-2,9-dihydro-1*H*-pyrido[2,3-b]indol-1-yl)(phenyl)methanone (4a)



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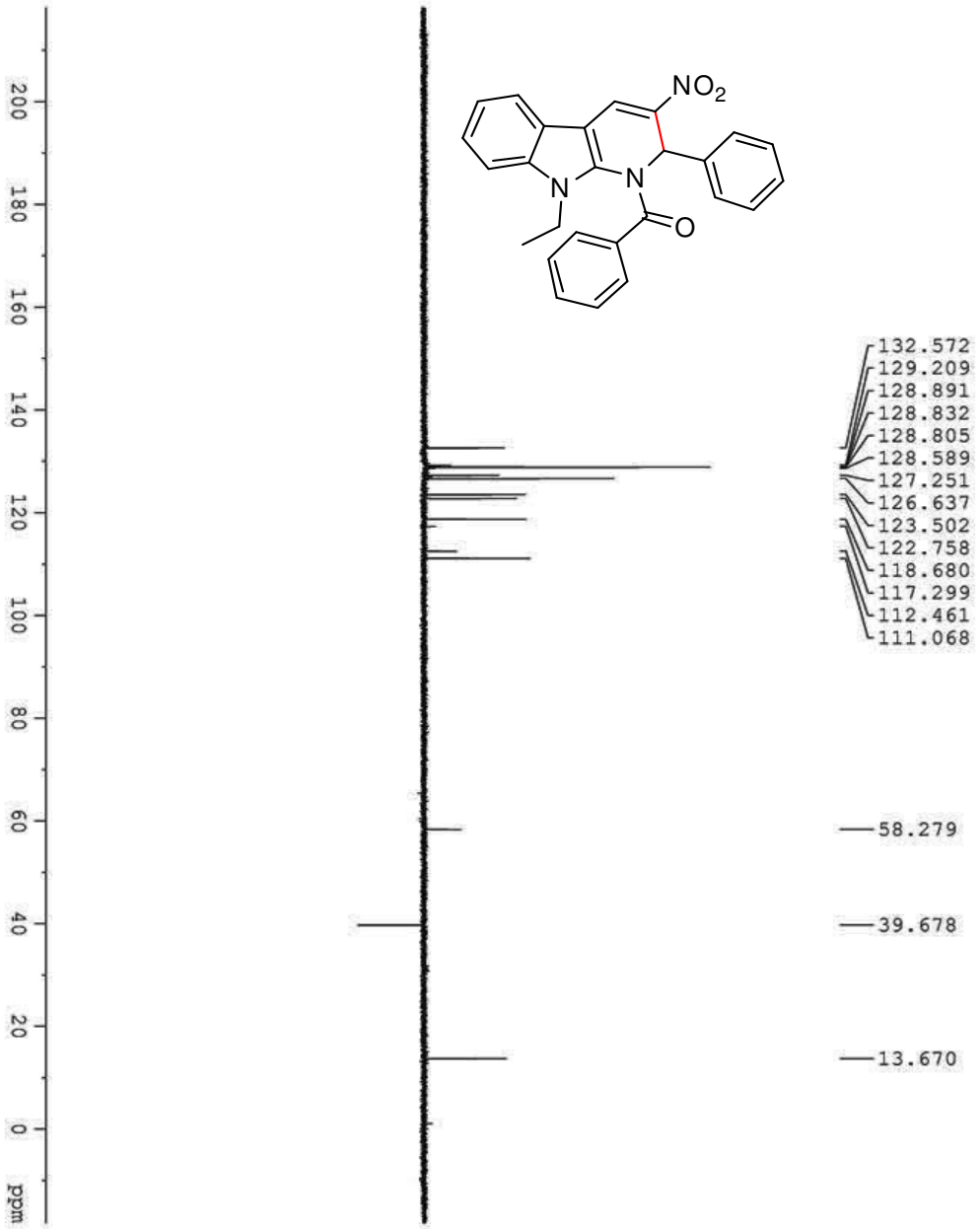
DEPT of (9-ethyl-3-nitro-2-phenyl-2,9-dihydro-1*H*-pyrido[2,3-b]indol-1-yl)(phenyl)methanone (4a)



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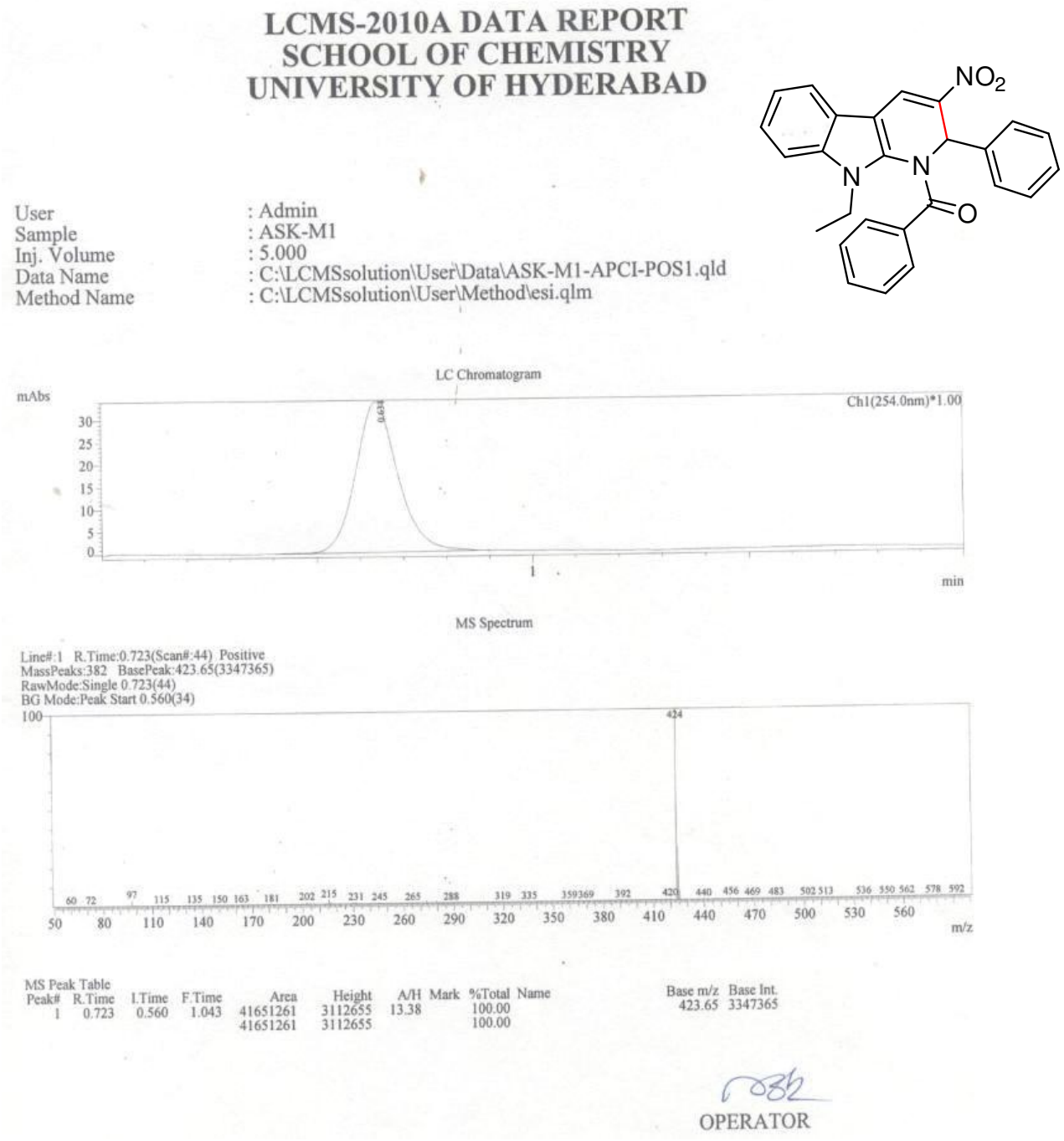
LCMS of (9-ethyl-3-nitro-2-phenyl-2,9-dihydro-1*H*-pyrido[2,3-b]indol-1-yl)(phenyl)methanone (4a)



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CHN Analysis of (9-ethyl-3-nitro-2-phenyl-2,9-dihydro-1*H*-pyrido[2,3-b]indol-1-yl)(phenyl)methanone (4a)

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Method filename:	E:\Program Files\Thermo Finnigan\Eager 300 for EA1112\DATA\Sys_data_ex
Sample ID:	ASK-M1 (# 167)
Analysis type:	UnkNown
Chromatogram filename:	UNK-15092011-7.dat
Sample weight:	.989

Element Name	Element %	Ret. Time
Nitrogen	9. 98	0. 77
Carbon	73. 65	1. 18
Hydrogen	5. 06	3. 70

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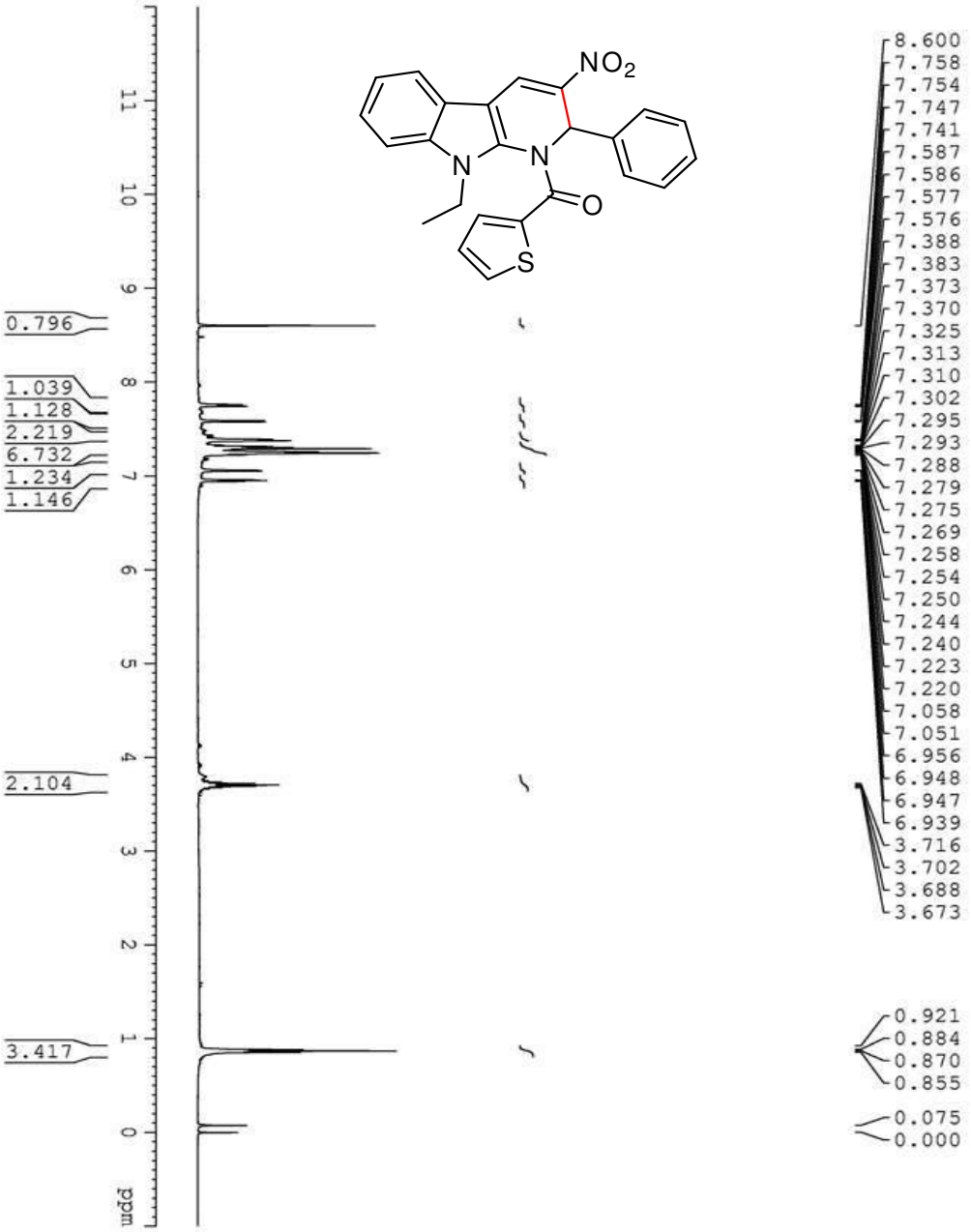
^{1}H NMR of (9-ethyl-3-nitro-2-phenyl-2,9-dihydro-1*H*-pyrido[2,3-*b*]indol-1-yl)(thiophen-2-yl)methanone (4b)



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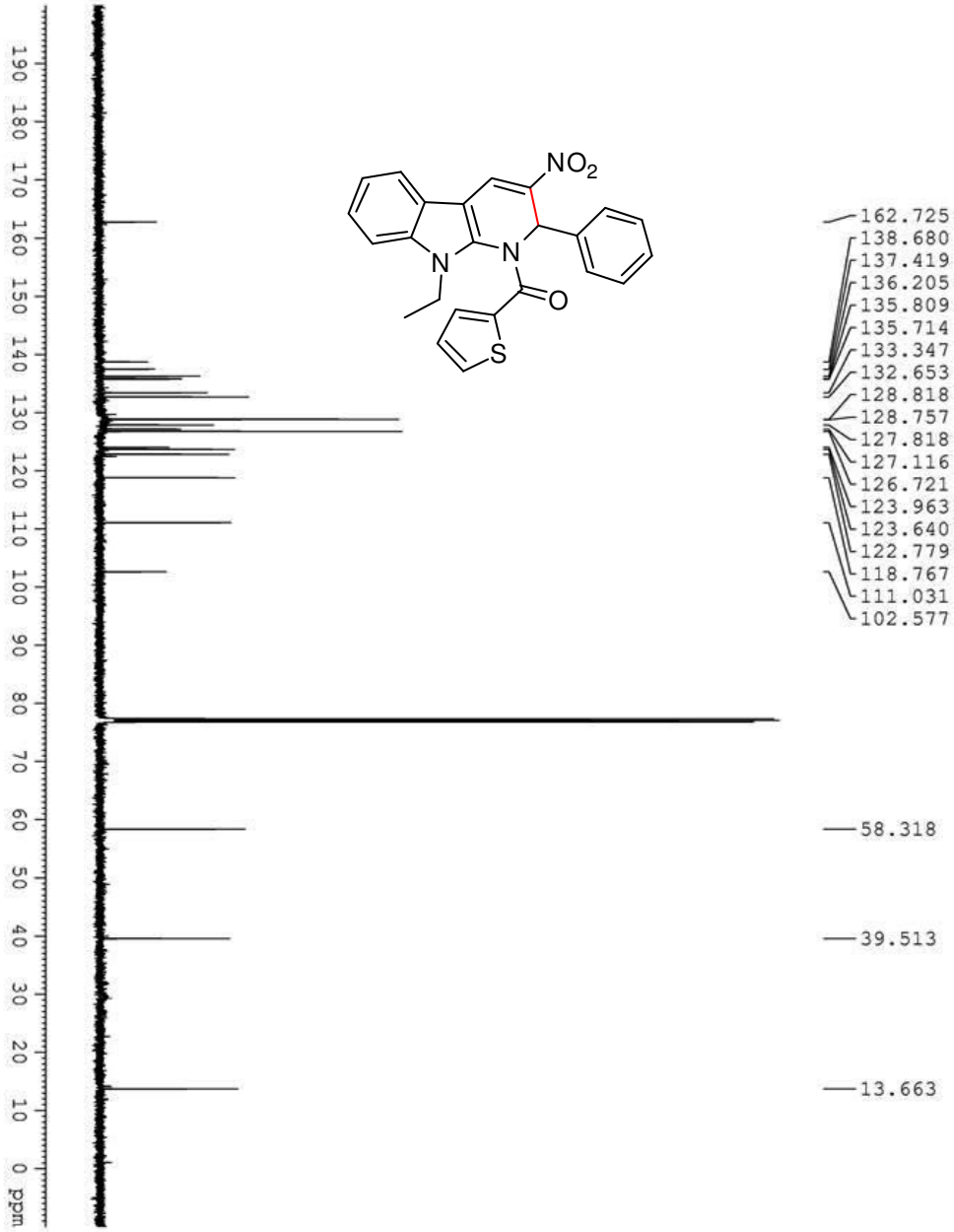
^{13}C NMR of (9-ethyl-3-nitro-2-phenyl-2,9-dihydro-1*H*-pyrido[2,3-*b*]indol-1-yl)(thiophen-2-yl)methanone (4b)



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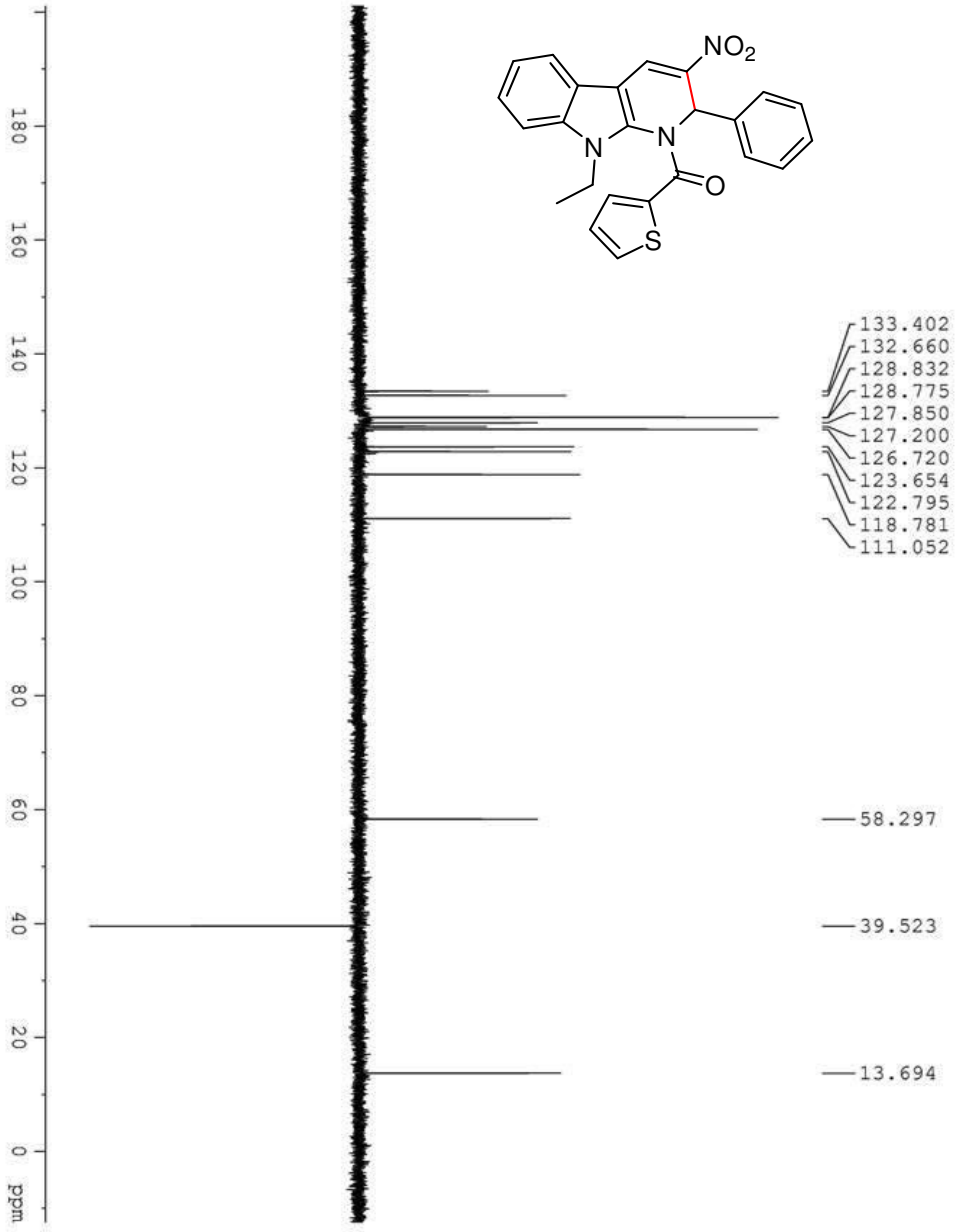
DEPT of (9-ethyl-3-nitro-2-phenyl-2,9-dihydro-1*H*-pyrido[2,3-*b*]indol-1-yl)(thiophen-2-yl)methanone (4b)



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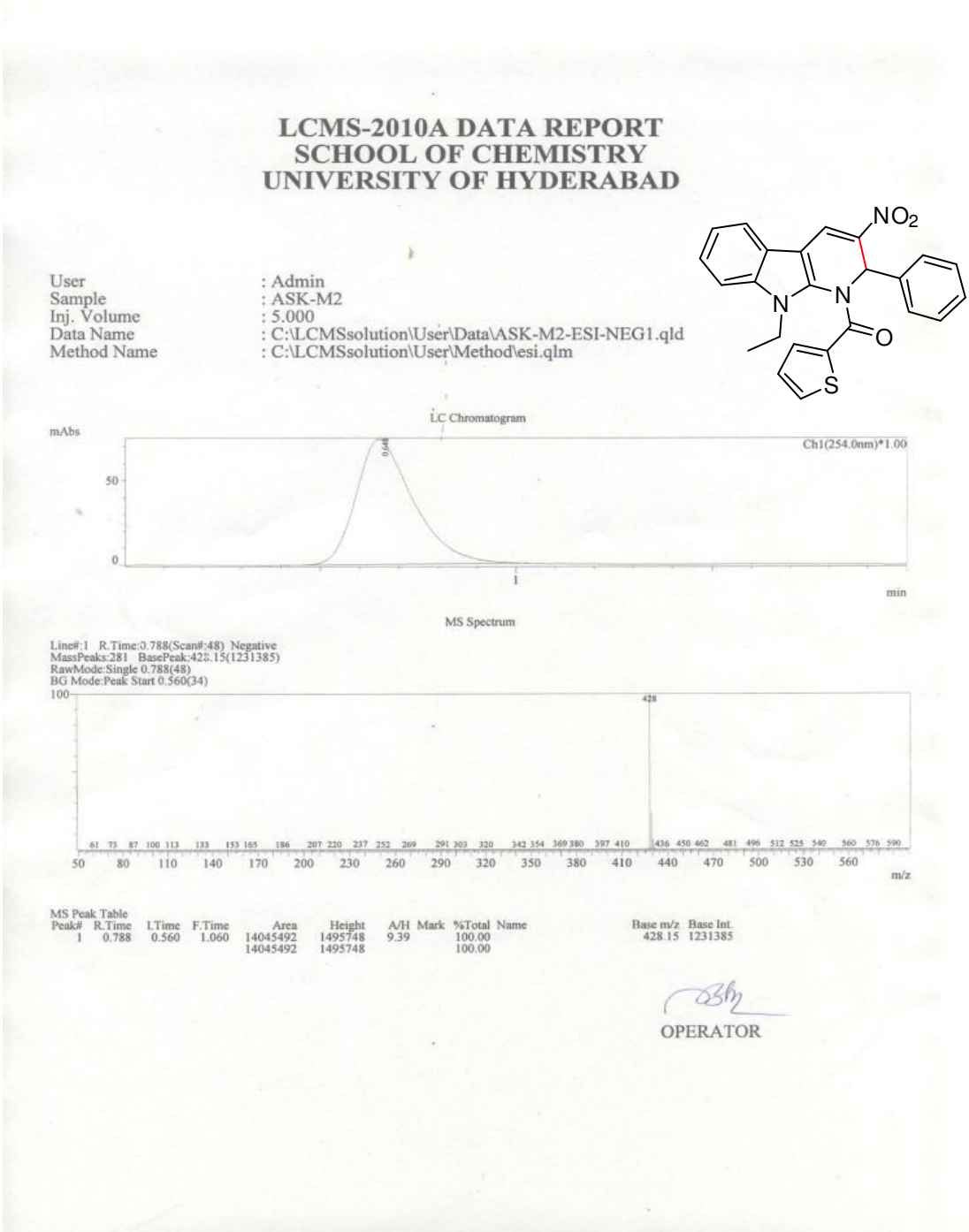
LCMS of (9-Ethyl-3-nitro-2-phenyl-2,9-dihydro-1*H*-pyrido[2,3-*b*]indol-1-yl)(thiophen-2-yl)methanone (4b)



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CHN Analysis of (9-Ethyl-3-nitro-2-phenyl-2,9-dihydro-1*H*-pyrido[2,3-*b*]indol-1-yl)(thiophen-2-yl)methanone (4b)

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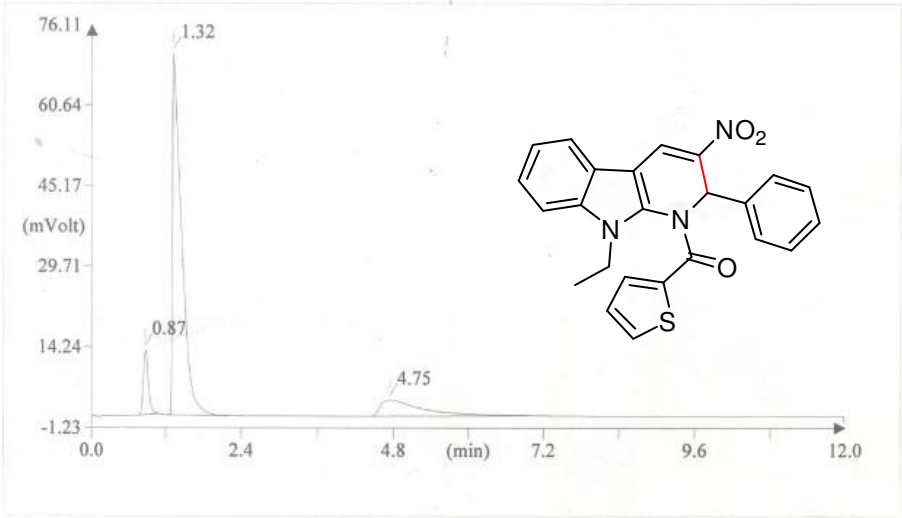
Method filename:	C:\Program Files\Thermo Finnigan\Eager 300 for EA1112\DATA\Sys_data_ex
Sample ID:	ASK-M2 (# 96)
Analysis type:	UnkNown
Chromatogram filename:	UNK-24012012-1.dat
Sample weight:	1.427



Element Name	Element %	Ret. Time
Nitrogen	9. 65	0. 87
Carbon	67. 26	1. 32
Hydrogen	4. 51	4. 75

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Computational Outputs

For optimized Keto form geometry:

Atomic Number	Co-ordinates X	Y	Z
6	1.580309	0.404257	-0.174170
6	1.135106	-0.936472	-0.008198
6	2.082680	-1.965577	0.100368
6	3.439201	-1.640837	0.047311
6	3.860219	-0.307220	-0.111848
6	2.936054	0.735316	-0.223939
6	-0.652397	0.447981	-0.156599
6	-0.307984	-0.895853	0.003261
1	1.757118	-2.993220	0.222828
1	4.183113	-2.428789	0.131349
1	4.922327	-0.079566	-0.146757
1	3.271784	1.761541	-0.340489
7	0.460217	1.241836	-0.274267
6	0.490568	2.697440	-0.416097
1	1.368820	2.945184	-1.019123
1	-0.377932	3.001474	-1.010715
6	0.526828	3.442670	0.922128
1	1.410663	3.160294	1.503391

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1	0.565134	4.522708	0.740796
1	-0.362624	3.228156	1.524726
6	-1.334281	-1.903363	0.135153
1	-2.169205	1.858666	-0.303866
6	-2.693258	-1.366545	0.082978
6	-2.977197	-0.038520	-0.074047
7	-1.946120	0.877726	-0.194134
8	-1.099611	-3.128775	0.282599
6	-4.365663	0.533549	-0.130365
1	-4.519625	1.278971	0.660087
1	-4.551827	1.025975	-1.093316
1	-5.107237	-0.257822	-0.002911
1	-3.513412	-2.071253	0.176395

For optimized Enol form geometry:

Atomic Number	Co-ordinates X	Y	Z
6	1.551417	0.396505	-0.174771
6	1.091332	-0.943544	-0.010437
6	2.032492	-1.979435	0.093029
6	3.393835	-1.677168	0.035074
6	3.827678	-0.349179	-0.126464
6	2.915270	0.702297	-0.232117

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6	-0.698975	0.505649	-0.155629
6	-0.354807	-0.867088	0.001384
1	1.728933	-3.016369	0.217957
1	4.124962	-2.476606	0.116149
1	4.892175	-0.134383	-0.167988
1	3.261685	1.724316	-0.352227
7	0.460426	1.253062	-0.266158
6	0.512487	2.705215	-0.412385
1	1.318083	2.944116	-1.115150
1	-0.429371	3.008886	-0.875161
6	0.715045	3.439981	0.916760
1	1.651714	3.141550	1.400026
1	0.750951	4.521363	0.740312
1	-0.110624	3.231280	1.605376
6	-1.427516	-1.757094	0.119615
1	-0.349563	-3.352709	0.286281
6	-2.728523	-1.241979	0.076779
6	-2.936764	0.135886	-0.083294
7	-1.926300	1.022340	-0.199250
8	-1.286977	-3.103499	0.275083
6	-4.333347	0.703975	-0.134182
1	-4.479040	1.437063	0.668412

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1	-4.499983	1.227104	-1.083649
1	-5.091222	-0.078079	-0.031517
1	-3.569510	-1.921483	0.168197

For optimized Transition state geometry:

Atomic Number	Co-ordinates X	Y	Z
6	1.549569	0.373936	-0.193892
6	1.087344	-0.962198	-0.099379
6	2.002266	-2.017158	-0.059138
6	3.361595	-1.726349	-0.126084
6	3.805737	-0.398800	-0.235272
6	2.911581	0.668655	-0.270372
6	-0.697441	0.507366	-0.112897
6	-0.360555	-0.895431	-0.082859
1	1.655816	-3.040924	0.023648
1	4.087268	-2.531731	-0.093329
1	4.870325	-0.196218	-0.288881
1	3.271633	1.688112	-0.347468
7	0.459102	1.243620	-0.198286
6	0.519677	2.702655	-0.284448
1	1.295591	2.963257	-1.008189
1	-0.433370	3.032881	-0.697066

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6	0.781928	3.372001	1.064429
1	1.728691	3.043763	1.499408
1	0.827153	4.456773	0.936948
1	-0.016973	3.146358	1.774299
6	-1.408970	-1.823423	0.396240
1	-1.426126	-1.267975	-1.102371
6	-2.676040	-1.274580	-0.156557
6	-2.911750	0.120602	-0.195056
7	-1.921090	1.021424	-0.120896
8	-1.254335	-2.920414	0.907428
6	-4.309216	0.650635	-0.363101
1	-4.982940	-0.107722	-0.764663
1	-4.708729	0.967924	0.606199
1	-4.316672	1.521678	-1.020516
1	-3.517408	-1.955641	-0.232844