Catalytic Asymmetric Strecker Hydrocyanation of Imines Using $Yb(OTf)_3$-pybox Catalysts

Babak Karimi*, and Aziz Maleki

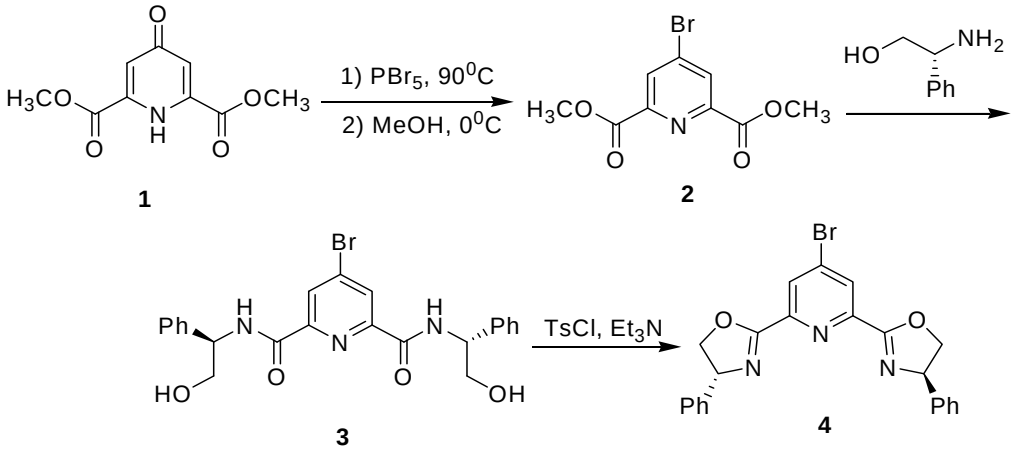
Department of Chemistry, Institute for Advanced Studies in Basic Sciences (IASBS), P.O.Box 45195-1159, Gava Zang, Zanjan, Iran

Table of Contents

1. Experimental Procedure:

1-1. General: ^{1}H-NMR spectra were recorded on commerical instruments (250 MHz). Chemical shifts were reported in ppm from tetramethylsilane with the solvent resonance as the internal standard ($CDCl_3$: δ= 7.26). Spectra are reported as follows: chemical shift (= ppm), multiplicity (s= singlet, d= doublet, t= triplet, q= quartet, m= multiplet), coupling constants (Hz), integration. ^{13}C-NMR spectra were collected on commerical instruments (62.90 MHz) with complete proton decoupling. The enantiomeric excesses were determined by HPLC analysis on CHIRALPAK AD column. Optical rotations were measured on a commerical polarimeter and reported as follows: $[\alpha]_D^T$ (c = g/100 mL, solvent). Reagents obtained from commercial sources were used without further purification.

1-2. Preparation of the Chiral Ligand



The preparation of chiral 4-bromopyridine-2,6-bis(oxazoline) ligand was achieved according to a know procedure reported by Moberg *et al.* (*Org. Lett.* **2003**, *5*, 3663) almost without any modification. The reaction sequences was demonstrated in the above reaction scheme:

Compound 2: m.p. 155-156 °C; ^{1}H NMR (250 MHz, $CDCl_3$): δ = 8.40 (s, 2H), 3.97 (s, 6H); ^{13}C NMR (62.90 MHz, $CDCl_3$): δ =163.97, 149.05, 135.7, 131.27, 53.45.

Compound 3: m.p. 83-85 °C; ^{1}H NMR (250 MHz, $CDCl_3$): δ = 8.50 (d, *J* = 7.5, 2H), 8.36 (s, 2H), 7.27 (m, 10H), 5.16 (m, 2H), 3.92 (d, *J* = 5.0 Hz, 4H), 2.94 (br s, 2H); ^{13}C NMR (62.90 MHz, $CDCl_3$): δ =162.5, 149.8, 138.7, 136.3, 128.8, 128.4, 127.9, 126.6, 66.9, 56.9.

Compound 4: m.p. 195-196 °C; ^{1}H NMR (250 MHz, $CDCl_3$): δ =8.51 (s, 2H), 7.29 (m, 10H), 5.44 (t, *J* = 7.5 Hz, 2H), 4.92 (t, *J* = 7.5 Hz, 2H), 4.42 (t, *J* = 10 Hz, 2H); ^{13}C NMR

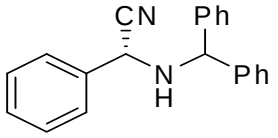
(62.90 MHz, $CDCl_3$): δ =162.5, 147.6, 141.3, 134.0, 129.4, 128.9, 127.9, 126.8, 75.7, 70.3.

1-3. General Procedure for the Preparation of Imine Substrate: the aldehyde (20 mmol) and diphenylmethylamine (20 mmol) and Na_2SO_4 (0.5 g) in dichloromethane (5 mL) were stirred in rt at 22 ^oC. The solution was filtrated and solvent was removed in vacuo. Products could be purified by recrystalyzation (Hexane-ethylacetate).

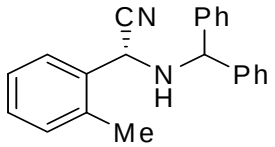
1-4. General Procedure for the Preparation of Ytterbium(III) Triflate Complexe: A 2-dram ovendried vial was charged with a stirbar, $Yb(OTf)_3$ (30 mg, 0.048 mmol), and the corresponding pybox ligand (44 mg , 0.098 mmol) in a dry box. The vial was capped with a septum and removed from the dry box. Dichloromethane (1.0 mL) was added to the vial under an atmosphere of dry Ar. The resulting mixture was stirred vigorously at rt for 1 h until the reaction became homogeneous.

1-5. General Catalytic Procedure: To the resulting complex solution 1 mmol of corresponding imine and 4 mL dichloromethane were added under argon and the resulting reaction mixture were cooled to the desired temperature. After 20 min TMSCN (2 mmol) were added to the flask in one portion and then methanol (2 mmol) was injected via the septum through a drop wise mode. The reaction was maintained at the desired temperature until consumption of imine as monitored by thin layer chromatography. The excess of nucleophile and the solvent were removed in the cold in vacuo and then products were purified by flash chromatography on silica gel.

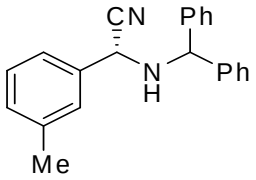
1-6. Characterizations of products:



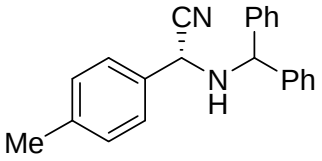
(Benzhydryl-amino)- phenyl-acetonitrile: The crude material was purified by flash chromatography on silica gel (THF/Hexane, 1:40) to afford the product in 92% yield as a white solid. The chromatographed material was determined to be of 97% ee by chiral HPLC analysis [Chiralpak AD, 90-10 *n*-hexane/*i*PrOH, 1.0 mL/min]; m.p. 94-96 ^oC; [α $]_D^{20}$ = 66.25 (c = 0.08 in $CHCl_3$). ^1H NMR (250 MHz, $CDCl_3$): δ = 7.61-7.075 (m, 15 H), 5.27 (s, 1 H), 4.62 (s, 1 H), 2.16 (s, 1 H) ppm. ^{13}C NMR (62.90 MHz, $CDCl_3$): δ =159.7, 142.7, 141.1, 134.9, 129.0, 128.8, 128.0, 127.7, 127.4, 127.3, 127.1, 118.78, 65.6, 52.4 ppm.



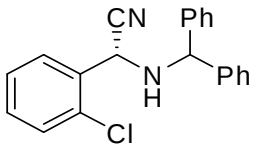
(Benzhydryl-amino)-*o*-tolyl-acetonitrile: The crude material was purified by flash chromatography on silica gel (THF/Hexane, 1:40) to afford the product in 97% yield as a white solid. The chromatographed material was determined to be of 93% ee by chiral HPLC analysis [Chiralpak AD, *n*-hexane/*i*PrOH, 98:2, 1.0 mL/min]; m.p. 106-108 ^{o}C, [α $]_D^{20}$ = 181.25 (c = 0.32 in $CHCl_3$). ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.61-7.22(m, 14 H), 5.31 (s, 1 H), 4.64 (s, 1 H), 2.30 (s, 3 H), 2.02 (s, 1 H; NH) ppm. ^{13}C NMR (62.90 MHz, $CDCl_3$): δ = 142.8, 141.0, 136.5, 133.2, 131.2, 129.2, 128.9, 128.8, 128.1, 127.9, 127.7, 127.5, 127.0, 126.7, 118.8, 65.8, 50.4, 18.9 ppm.



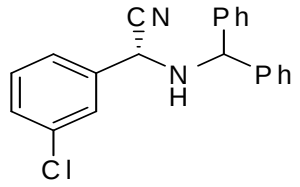
(Benzhydryl-amino)-*m*-tolyl-acetonitrile: The crude material was purified by flash chromatography on silica gel (THF/Hexane, 1:40) to afford the product in 95% yield as a white solid. The chromatographed material was determined to be of 82% ee by chiral HPLC analysis [Chiralpak AD, *n*-hexane/*i*PrOH, 98:2, 1.0 mL/min], m.p. 69-71 ^{o}C; [α $]_D^{20}$ = 56.7 (c = 0.10 in $CHCl_3$). ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.58-7.24 (m, 14 H), 5.28 (s, 1 H), 4.58 (s, 1 H), 2.41 (s, 3 H), 2.15 (s, 1 H) ppm. ^{13}C NMR (62.90 MHz, $CDCl_3$): δ = 142.8, 141.2, 138.9, 134.9, 129.8, 129.0, 128.9, 128.8, 127.9, 127.7, 127.5, 127.2, 124.4, 118.9, 65.6, 52.4, 21.4ppm.



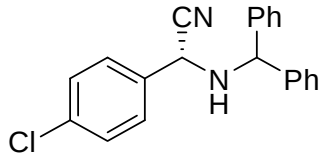
(Benzhydryl-amino)-*p*-tolyl-acetonitrile: The crude material was purified by flash chromatography on silica gel (THF/Hexane, 1:40) to afford the product in 93% yield as a white solid. The chromatographed material was determined to be of 96% ee by chiral HPLC analysis [Chiralpak AD, *n*-hexane/*i*PrOH, 90: 10, 1.0 mL/min], m.p. 104-106 ^{o}C. [α $]_D^{20}$ = 55.0 (c = 0.2 in $CHCl_3$). ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.63-7.24 (m, 14 H), 5.29 (s, 1 H), 4.60 (s, 1 H), 2.41 (s, 3 H), 2.16 (s, 1 H) ppm. ^{13}C NMR (62.90 MHz, $CDCl_3$): δ = 142.8, 141.3, 139.0, 132.1, 129.7, 129.1, 128.8, 127.9, 127.7, 127.5, 127.2, 127.2, 119.0, 65.6, 52.2, 21.2 ppm.



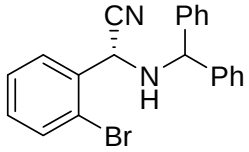
(Benzhydryl-amino)-(2-chloro-phenyl)-acetonitrile: The crude material was purified by flash chromatography on silica gel (THF/Hexane, 1:40) to afford the product in 97% yield as a white solid. The chromatographed material was deter-mined to be of 92% ee by chiral HPLC analysis [Chiralpak AD, *n*-hexane/*i*PrOH, 98:2, 1.0 mL/min]; m.p. 98-100 ^{o}C, $[\alpha]_D^{20}$ = 127.7 (c = 0.18 in $CHCl_3$). ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.59-7.02 (m, 14H), 5.25(s, 1 H), 4.91 (s, 1 H), 2.19 (s, 1 H) ppm. ^{13}C NMR (62.90 MHz, $CDCl_3$): δ = 142.6, 140.7, 133.5,132.8, 130.6, 130.4, 129.3, 128.8, 128.8, 128.0, 127.7, 127.7, 127.6, 127.2, 118.2, 65.7, 50.2 ppm.



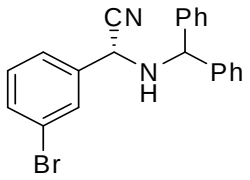
(Benzhydryl-amino)-(3-chloro-phenyl)-acetonitrile: The crude material was purified by flash chromatography on silica gel (THF/Hexane, 1:40) to afford the product in 93% yield as a white solid. The chromatographed material was determined to be of 91% ee by chiral HPLC analysis [Chiralpak AD, *n*-hexane/*i*PrOH, 90: 10, 1.0 mL/min]; m.p. 101-103 ^{o}C, $[\alpha]_D^{20}$ = 38.8 (c = 0.34 in CHCl3). ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.62-7.26 (m, 14H), 5.27(s, 1 H), 4.60 (s, 1 H), 2.20 (s, 1 H) ppm. ^{13}C NMR (62.90 MHz, $CDCl_3$): δ = 142.6, 140.9, 136.8, 135.0, 130.3, 129.3, 129.2, 128.9, 128.1, 127.9, 127.5, 127.2, 125.5 118.3, 65.7, 51.9 ppm.



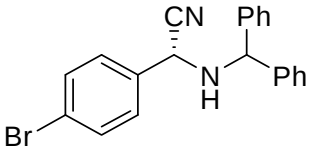
(Benzhydryl-amino)- (4-chloro-phenyl)-acetonitrile: The crude material was purified by flash chromatography on silica gel (THF/Hexane, 1:40) to afford the product in 91% yield as a white solid. The chromatographed material was determined to be of 93% ee by chiral HPLC analysis [Chiralpak AD, *n*-hexane/iPrOH, 90:10, 1.0 mL/min]; m.p. 102-104 ^{o}C, $[\alpha]_D^{20}$ = 34.32 (c = 0.67 in $CHCl_3$). ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.72-7.09 (m, 14 H), 5.26 (s, 1 H), 4.59 (s, 1 H), 2.07 (s, 1 H) ppm. ^{13}C NMR (62.90 MHz, $CDCl_3$): δ =142.5, 140.9, 135.1, 133.4, 129.2, 129.1, 128.9, 128.7, 128.1, 127.8, 127.4, 127.1, 118.4, 65.6, 51.8 ppm.



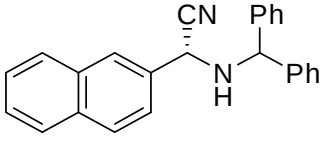
(Benzhydryl-amino)-(2-bromo-phenyl)-acetonitrile: The crude material was purified by flash chromatography on silica gel (THF/Hexane, 1:40) to afford the product in 97% yield as a white solid. The chromatographed material was deter-mined to be of 97% ee by chiral HPLC analysis [Chiralpak AD, *n*-hexane/*i*PrOH, 98:2, 1.0 mL/min]; m.p. 101-103 ^{o}C, $[\alpha]_D^{22}$ = 107.14 (c = 0.28 in $CHCl_3$). ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.90-7.24(m, 14H), 5.23(s, 1 H), 4.89 (s, 1 H), 2.12 (s, 1 H) ppm. ^{13}C NMR (62.90 MHz, $CDCl_3$): δ = 142.6, 140.7, 134.5, 133.8, 130.8, 129.4, 128.8, 128.2, 128.0, 127.9, 127.7, 127.2, 123.4, 118.2, 65.6, 52.5 ppm.



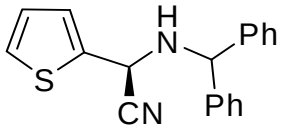
(Benzhydryl-amino)-(3-bromo-phenyl)-acetonitrile: The crude material was purified by flash chromatography on silica gel (THF/Hexane, 1:40) to afford the product in 92% yield as a white solid. The chromatographed material was determined to be of 91% ee by chiral HPLC analysis [Chiralpak AD, *n*-hexane/*i*PrOH, 98:2, 1.0 mL/min]; m.p. 102-104 ^{o}C, $[\alpha]_D^{20}$ = 305 (c = 0.2 in $CHCl_3$). ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.72-7.25 (m, 14 H), 5.25(s, 1 H), 4.58 (s, 1 H), 2.17 (s, 1 H) ppm. ^{13}C NMR (62.90 MHz, $CDCl_3$): δ = 142.5, 140.8, 137.0, 132.3, 130.5, 130.4, 129.1, 128.9, 128.1, 127.8, 127.5, 127.1, 125.93, 123.0, 118.2, 65.7, 51.8 ppm



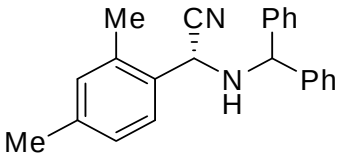
(Benzhydryl-amino)-(4-bromo-phenyl)-acetonitrile: The crude material was purified by flash chromatography on silica gel (THF/Hexane, 1:40) to afford the product in 90% yield as a white solid. The chromatographed material was determined to be of 84% ee by chiral HPLC analysis [Chiralpak AD, *n*-hexane/*i*PrOH, 90:10, 1.0 mL/min], m.p. 99-101 ^{o}C, $[\alpha]_D^{20}$ = 28.46 (c = 0.13 in $CHCl_3$). ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.76-7.22 (m, 14 H), 5.25 (s, 1 H), 4.57 (s, 1 H), 2.17 (s, 1 H) ppm. ^{13}C NMR (62.90 MHz, $CDCl_3$): δ = 142.5, 140.9, 133.9, 132.2, 129.1, 129.0, 128.9, 128.1, 127.8, 127.4, 127.1,123.2, 118.3, 65.6, 51.8 ppm.



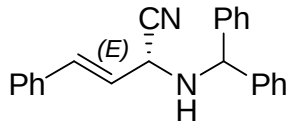
(Benzhydryl-amino)-naphthalen-2-yl-acetonitrile : The crude material was purified by flash chromatography on silica gel (THF/Hexane, 1:40) to afford the product in 90% yield as a white solid. The chromatographed material was determined to be of 80% ee by chiral HPLC analysis [Chiralpak AD, *n*-hexane/*i*PrOH, 90:10, 1.0 mL/min]; m.p. 119-121 ^{o}C, $[\alpha]_D^{20}$ = 7.8 (c = 0.12 in $CHCl_3$). ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.94-7.04 (m, 17 H), 5.36 (s, 1 H), 4.79 (s, 1 H), 2.28(s, 1 H) ppm. ^{13}C NMR (62.90 MHz, $CDCl_3$): δ =142.8, 141.2, 133.4, 133.1, 132.3, 129.2, 129.0, 128.9, 128.2, 128.0, 127.8, 127.6, 127., 126.9, 126.8, 126.4, 124.9, 118.9, 65.8, 52.6 ppm.



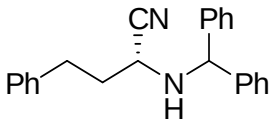
(Benzhydryl-amino)-thiophen- 2-yl-acetonitrile: The crude material was purified by flash chromatography on silica gel (THF/Hexane, 1:40) to afford the product in 85% yield as a white solid. The chromatographed material was determined to be of 92% ee by chiral HPLC analysis [Chiralpak AD, *n*-hexane/*i*PrOH, 90:10, 1.0 mL/min], m.p. 83-85 ^{o}C, $[\alpha]_D^{24}$ = 60.12 (c = 0.4 in $CHCl_3$). ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.63-7.00 (m, 13 H), 5.27 (s, 1 H), 4.80 (s, 1 H), 2.41 (s, 1 H) ppm. ^{13}C NMR (62.90 MHz, $CDCl_3$): δ = 142.5, 140.8, 138.3, 129.1, 128.9, 128.1, 127.9, 127.4, 127.1, 126.95, 126.7, 126.1, 118.2, 65.41, 48.2 ppm.



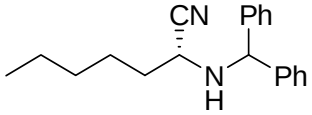
(Benzhydryl-amino)-(2,4-dimethyl-phenyl)-acetonitrile: The crude material was purified by flash chromatography on silica gel (THF/Hexane, 1:40) to afford the product in 97% yield as a white solid. The chromatographed material was determined to be of 95% ee by chiral HPLC analysis [Chiralpak AD, *n*-hexane/*i*PrOH, 90:10, 1.0 mL/min], m.p. 123-125 ^{o}C, $[\alpha]_D^{20}$ = 470 (c = 0.2 in $CHCl_3$). ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.61-7.06 (m, 13 H), 5.29 (s, 1 H), 4.60 (d, *J* = 10 Hz, 1 H), 2.35 (s, 3H), 2.27 (s, 3H), 2.01 (d, *J* = 12.5 Hz, 1 H), ppm. ^{13}C NMR (62.90 MHz, $CDCl_3$): δ = 142.9, 141.1, 139.1, 136.2, 132.0, 130.4, 128.9, 128.7, 128.0, 127.9, 127.6, 127.5, 127.3, 127.0, 118.94, 65.8, 50.1, 21.1, 18.8 ppm. Anal. Calcd for $C_{23}H_{22}N_2$: C, 84.6; H, 6.7; N, 8.6. Found: C, 84.5; H, 6.9; N, 9.0.



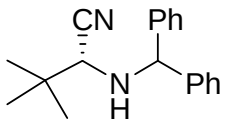
2-(Benzhydryl-amino)-4-phenyl-but-3-enenitrile: The crude material was purified by flash chromatography on silica gel (THF/Hexane, 1:40) to afford the product in 98% yield as a white solid. The chromatographed material was determined to be of 97% ee by chiral HPLC analysis [Chiralpak AD, *n*-hexane/*i*PrOH, 98:2, 1.0 mL/min], m.p. 90-92 ^{o}C, $[\alpha]_D^{20}$ = 16.08 (c = 0.23 in $CHCl_3$). ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.64-7.03 (m, 15 H), 6.91 (d, *J* = 17.5 Hz, 1 H), 6.23 (dd, ^1J = 17.5 ^2J =5 Hz, 1 H), 5.22 (s, 1 H), 4.24 (s, 1 H), 1.01 (d, *J* = 12.5 Hz, 1 H) , ppm. ^{13}C NMR (62.90 MHz, $CDCl_3$): δ = 142.8, 141.0, 135.3, 133.8, 129.0, 128.81, 128.75, 128.6, 127.9, 127.7, 127.4, 127.1, 126.82 122.4, 118.3, 65.4, 50.1 ppm.



2-(Benzhydryl-amino)-4-phenyl-butyronitrile: The crude material was purified by flash chromatography on silica gel (THF/Hexane, 1:50) to afford the product in 86% yield as a white solid. The chromatographed material was determined to be of 73% ee by chiral HPLC analysis [Chiralpak AD, *n*-hexane/*i*PrOH, 98:2, 1.0 mL/min], m.p. 79-80 ^{o}C, $[\alpha]_D^{20}$ = 63.4 (c = 0.54 in $CHCl_3$). ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.55-7.21 (m, 15 H), 5.23 (s, 1 H), 3.47 (s, 1 H), 2.90 (t, *J* = 7.5 Hz, 2 H), 2.15 (q, *J* = 7.5 Hz, 2 H), 1.91(s, 1 H) ppm. ^{13}C NMR (62.90 MHz, $CDCl_3$): δ = 143.3, 141.3, 140.2, 128.95, 128.86, 128.7, 128.5, 127.85, 127.75, 127.6, 127.2, 126.5, 120.2, 65.6, 48.0, 35.4, 31.9 ppm.

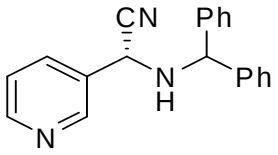


2-(Benzhydryl-amino)-heptanenitrile: The crude material was purified by flash chromatography on silica gel (THF/Hexane, 1:60) to afford the product in 95% yield as oil. The chromatographed material was determined to be of 61% ee by chiral HPLC analysis [Chiralpak AD, *n*-hexane/*i*PrOH, 98:2, 1.0 mL/min], m.p. 99-101 ^{o}C, $[\alpha]_D^{20}$ =35 (c = 0.6 in $CHCl_3$). ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.55-7.27 (m, 10 H), 5.21 (s, 1 H), 3.41 (s, 1 H), 1.81 (m, 3 H), 1.55 (m, 2 H), 1.33(m, 4 H), 0.97(m, 3 H) ppm. ^{13}C NMR (62.90 MHz, $CDCl_3$): δ = 143.6, 141.8, 129.0, 128.8, 127.8, 127.7, 127.6, 127.3, 120.4, 65.7, 48.5, 33.7, 31.3, 25.4, 22.6, 14.2 ppm.

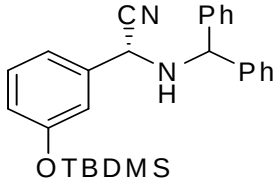


2-(Benzhydryl-amino)-3,3-dimethyl-butyronitrile: The crude material was purified by flash chromatography on silica gel (THF/Hexane, 1:60) to afford the product in 91%

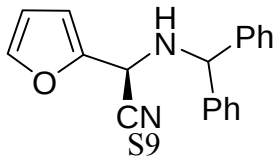
yield as a white solid. The chromatographed material was determined to be of 76% ee by chiral HPLC analysis [Chiralpak AD, *n*-hexane/*i*PrOH, 98:2, 1.0 mL/min]; m.p. 46-48 ^{o}C, $[\alpha]_D^{20}$ = 83.62 (c = 0.41 in $CHCl_3$). ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.54-7.25 (m, 10 H), 5.15 (s, 1 H), 3.11 (s, 1 H), 1.47 (s, 1 H), 1.12 (s, 9H) ppm. ^{13}C NMR (62.90 MHz, $CDCl_3$): δ = 143.6, 141.4, 130.1, 128.84, 127.78, 127.7, 127.5, 127.2, 119.6, 65.8, 59.0, 34.4, 26.4 ppm.



(Benzhydryl-amino)-pyridin- 3-yl-acetonitrile: The crude material was purified by flash chromatography on silica gel (THF/Hexane, 1:30) to afford the product in 57% yield as a yellow solid. The chromatographed material was determined to be of 62% ee by chiral HPLC analysis [Chiralpak AD, *n*-hexane/*i*PrOH, 90:10, 1.0 mL/min], m.p. 115-117 ^{o}C, $[\alpha]_D^{20}$ = 140.2 (c = 0.5 in $CHCl_3$). ^{1}H NMR (250 MHz, $CDCl_3$): δ = 8.76 (s,1H), 8.58 (d, *J* = 5 Hz, 1 H), 7.86 (d, *J* = 7.5 Hz, 1 H), 7.56 (d, *J* = 7.5 Hz, 2 H), 7.45-7.21 (m, 8H), 4.63 (d, *J* = 10 Hz, 1 H), 2.31 (d, *J* = 10 Hz, 1 H) ppm. ^{13}C NMR (62.90 MHz, $CDCl_3$): δ = 150.3, 148.8, 142.4, 140.7, 134.8, 130.8, 129.2, 128.9, 128.1, 127.8, 127.4, 127.0, 123.6, 117.8, 65.7, 50.3 ppm. Anal. Calcd for $C_{20}H_{17}N_3$: C, 80.2; H, 5.68; N, 14.0. Found: C, 79.7; H, 5.8; N, 13.8.

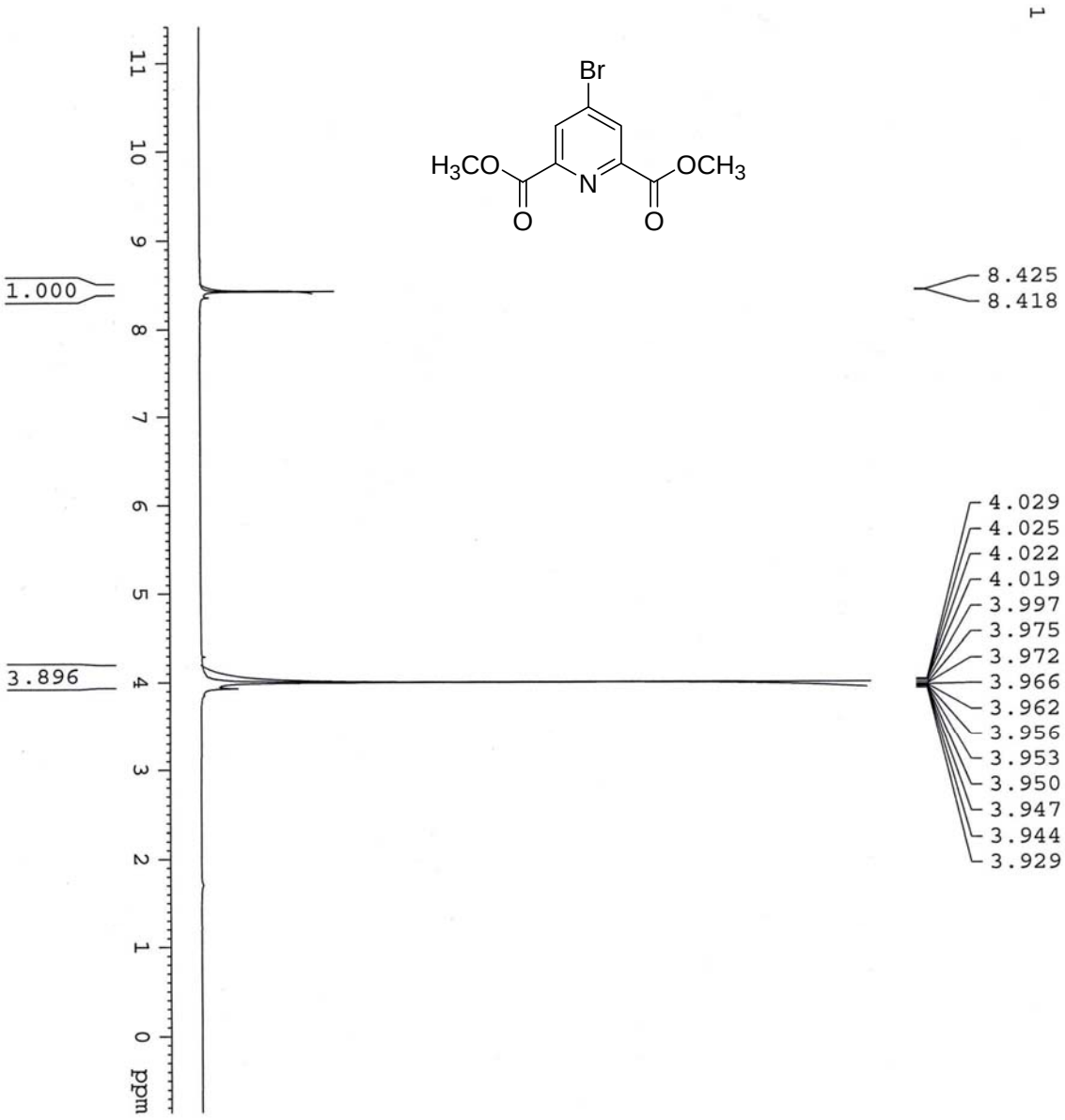


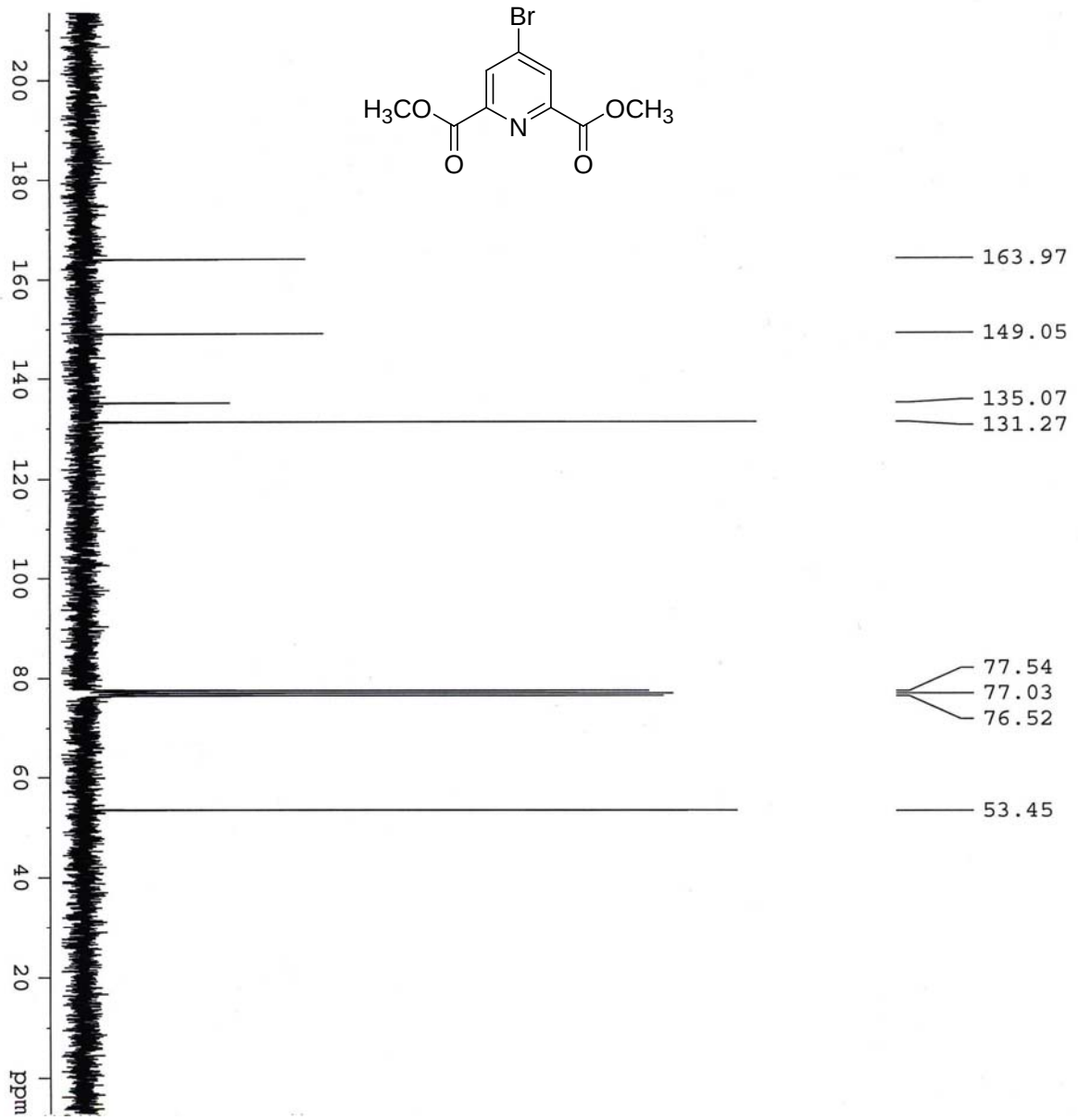
(Benzhydryl-amino)-[3-(tert-butyl-dimethyl-silanyloxy)-phenyl]-acetonitrile: The crude material was purified by flash chromatography on silica gel (THF/Hexane, 1:40) to afford the product in 96% yield as a white solid. The chromatographed material was determined to be of 87% ee by chiral HPLC analysis [Chiralpak AD, *n*-hexane/*i*PrOH, 98:2, 1.0 mL/min], m.p. 89-91 ^{o}C, $[\alpha]_D^{20}$ = 550.3 (c = 0.12 in $CHCl_3$). ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.58-6.84 (m, 14 H), 5.23 (s, 1 H), 4.55 (s, 1 H), 2.18 (s, 1 H), 1.01 (s, 9 H), 0.24(s, 6 H) ppm. ^{13}C NMR (62.90 MHz, $CDCl_3$): δ = 156.2, 142.7, 141.2, 136.4, 130.0, 129.0, 128.8, 127.9, 127.7, 127.4, 127.1, 120.6, 120.0, 119.0, 118.8, 65.5, 52.1, 25.7, 18.3, -4.3 ppm. Anal. Calcd for $C_{27}H_{32}N_2OSi$: C, 75.7; H, 7.47; N, 6.54. Found: C, 76.3; H, 7.6; N, 7.2.

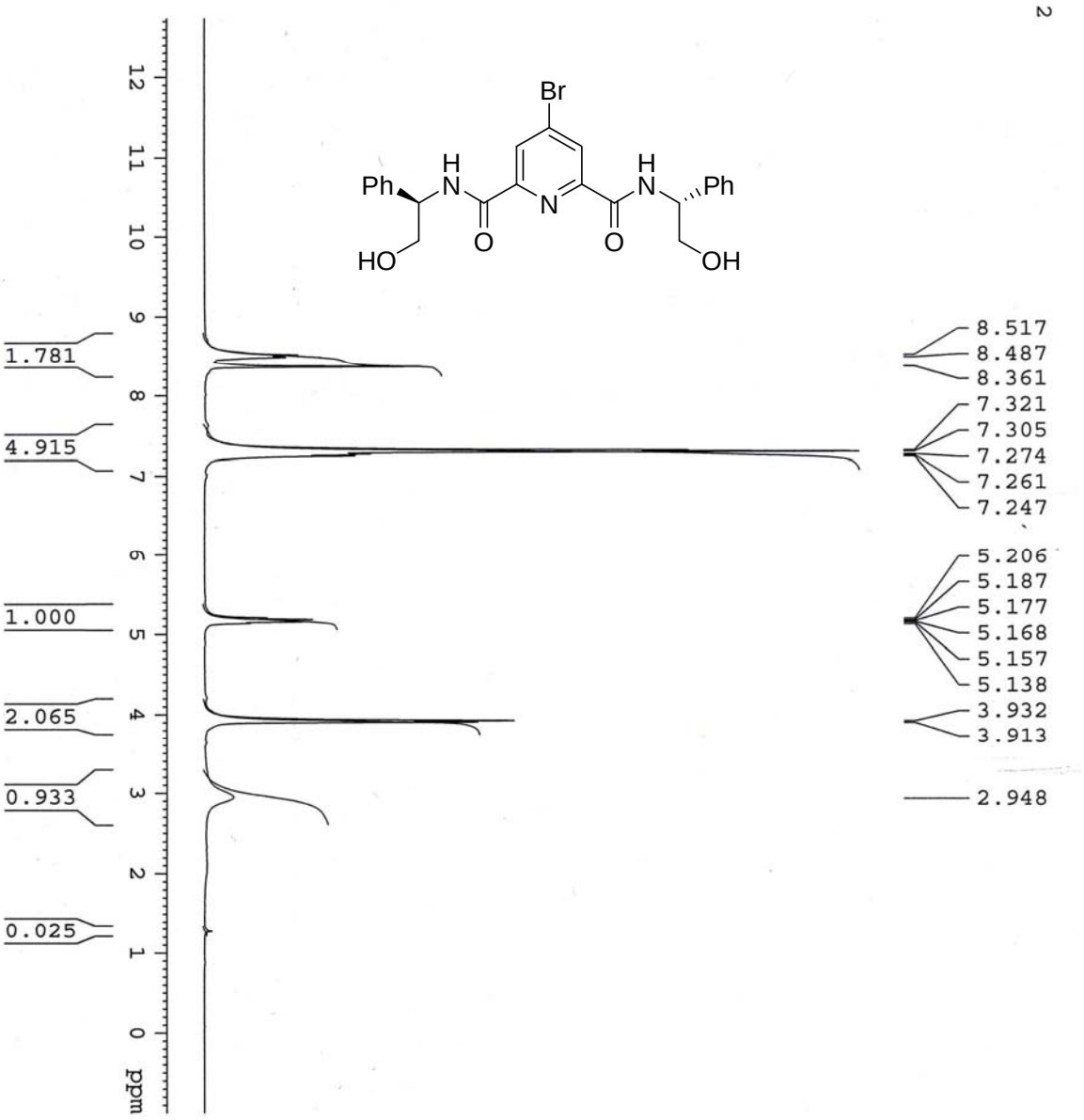


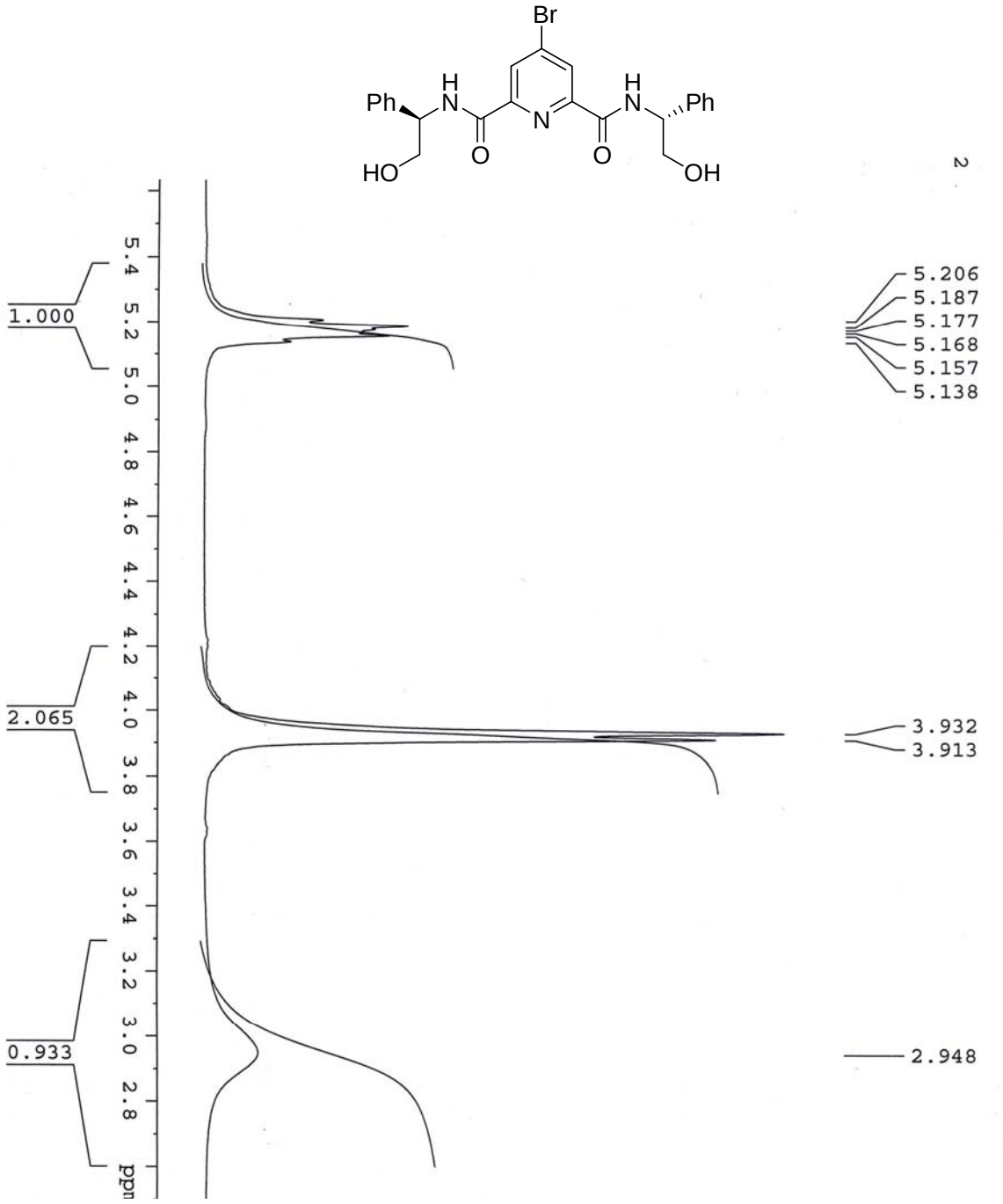
2-(benzhydrylamino)-2-(furan-2-yl)acetonitrile: The crude material was purified by flash chromatography on silica gel (THF/Hexane, 1:40) to afford the product in 50% yield as a white solid. The chromatographed material was determined to be of 45% ee by chiral HPLC analysis [Chiralpak AD, *n*-hexane/iPrOH, 98:2, 1.0 mL/min]; m.p.99-101 ^{o}C, $[\alpha]_D^{20}$ = 8.07 (c = 0.52 in $CHCl_3$). ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.53-7.20 (m, 12 H), 6.39 (dd, ^1J = 17.5 ^2J =1.5 Hz, 1 H), 5.16 (s, 1 H), 4.65 (s, 1 H), 2.33 (s, 1 H) ppm. ^{13}C NMR (62.90 MHz, $CDCl_3$): δ =147.2, 143.6, 142.3, 140.8, 129.0, 128.8, 128.0, 127.8, 127.4, 127.2, 127.0, 110.6, 108.9, 65.1, 49.2 ppm.

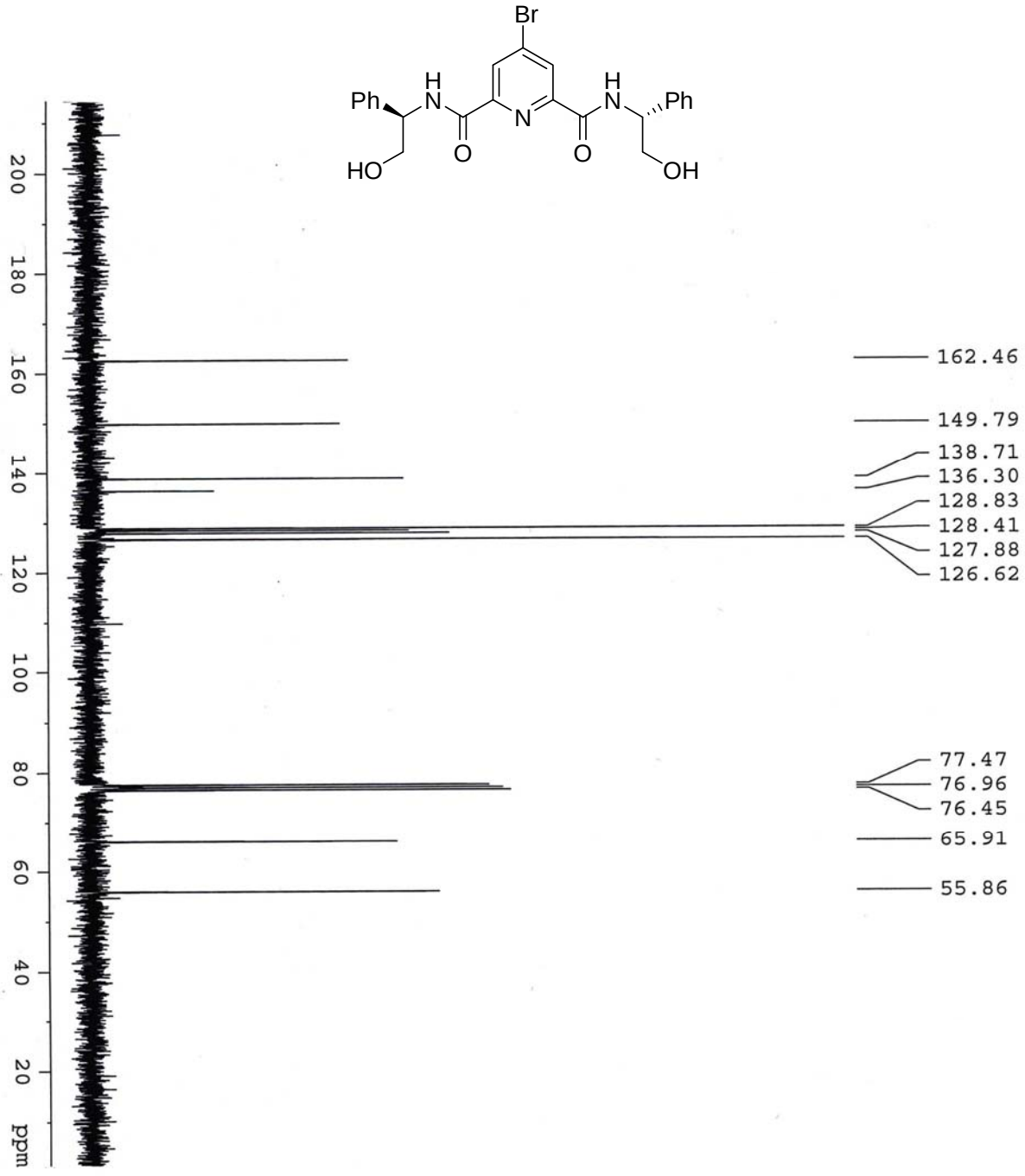
Copy of, ^{1}H , ^{13}C-NMR, HPLC and FT-IR spectra of α-amino nitril derivatives

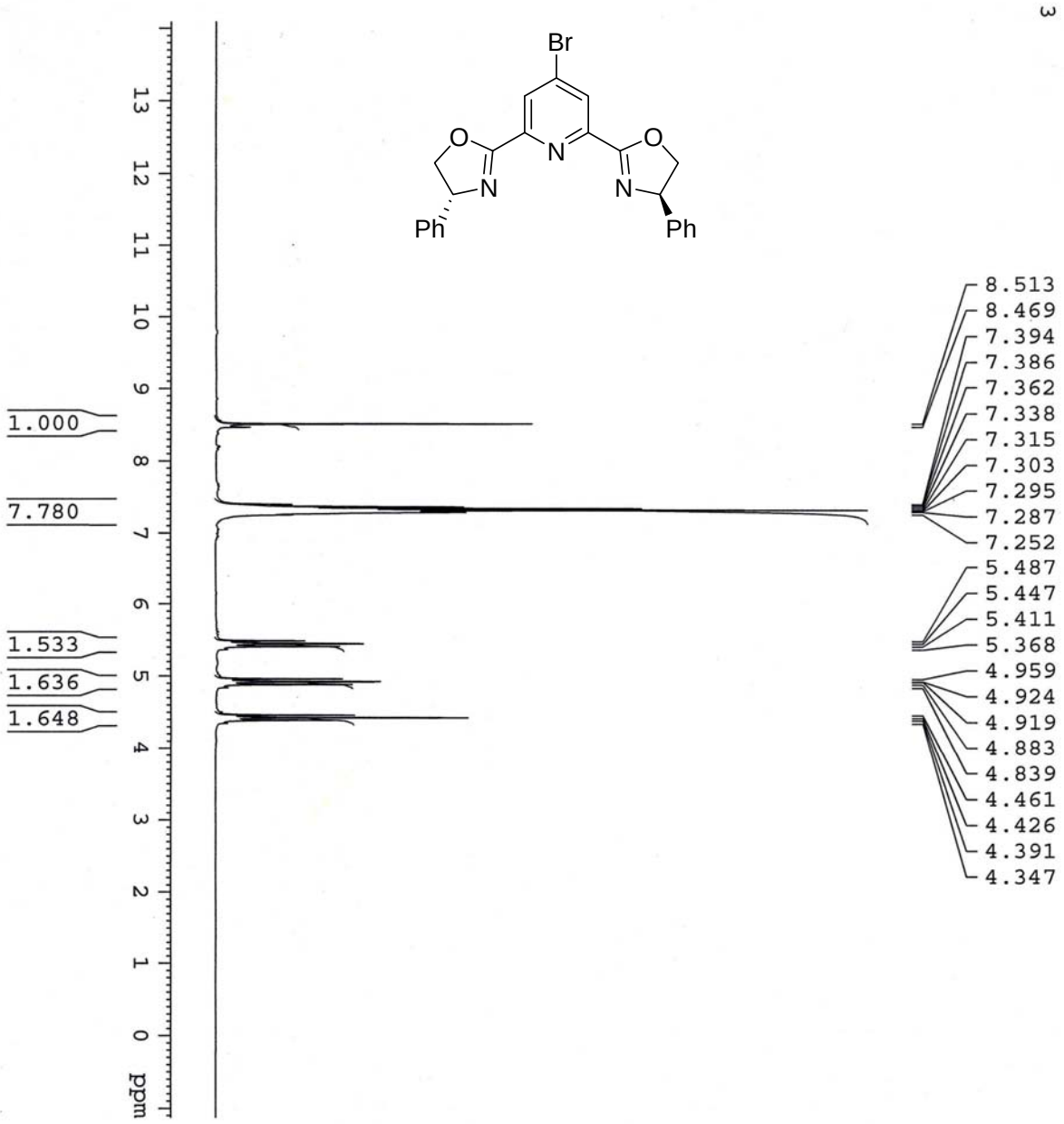


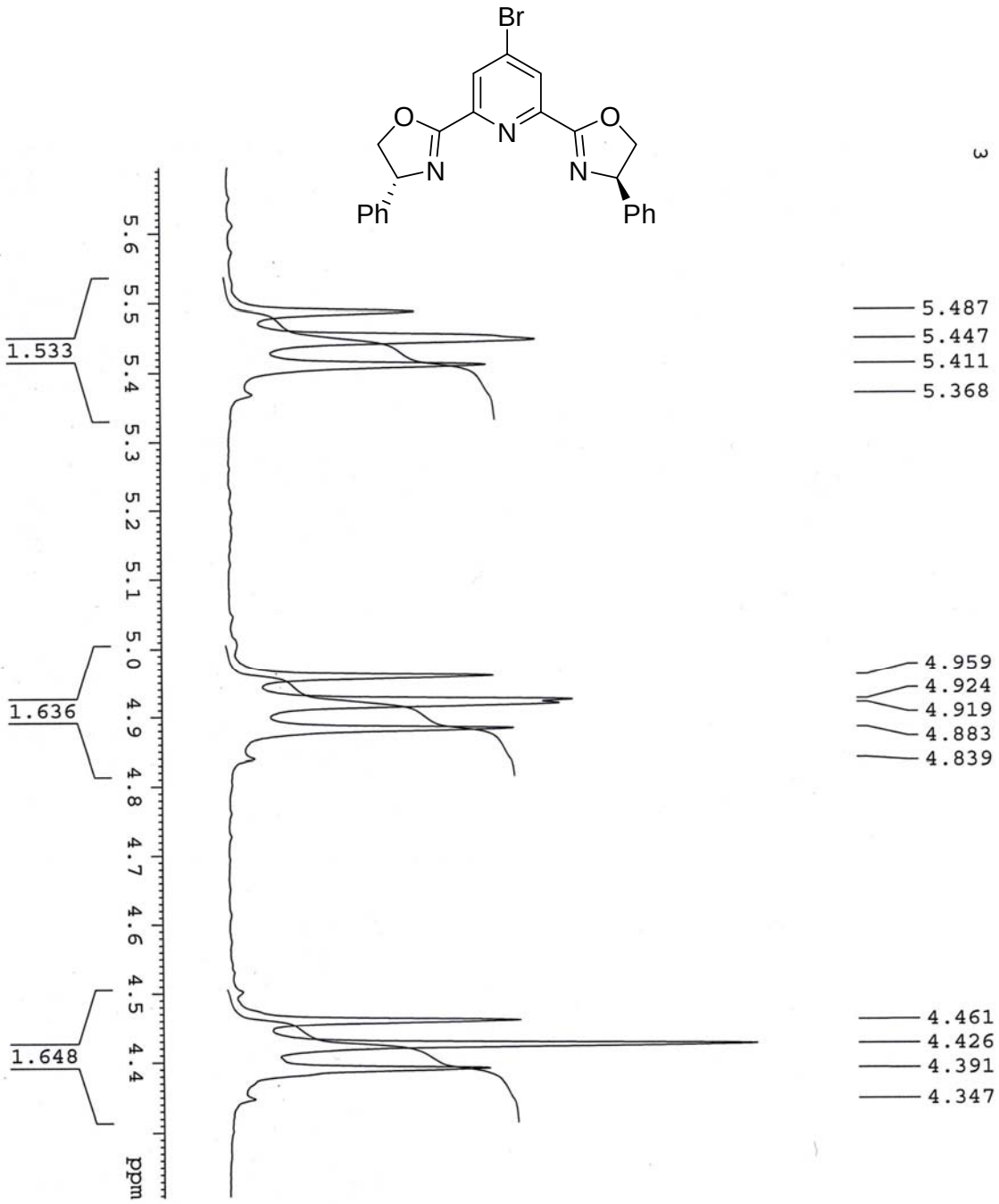

Br
H3CO
N
OCH3
O
O
200
180
160
140
120
100
80
60
40
20
ppm
163.97
149.05
135.07
131.27
77.54
77.03
76.52
53.45

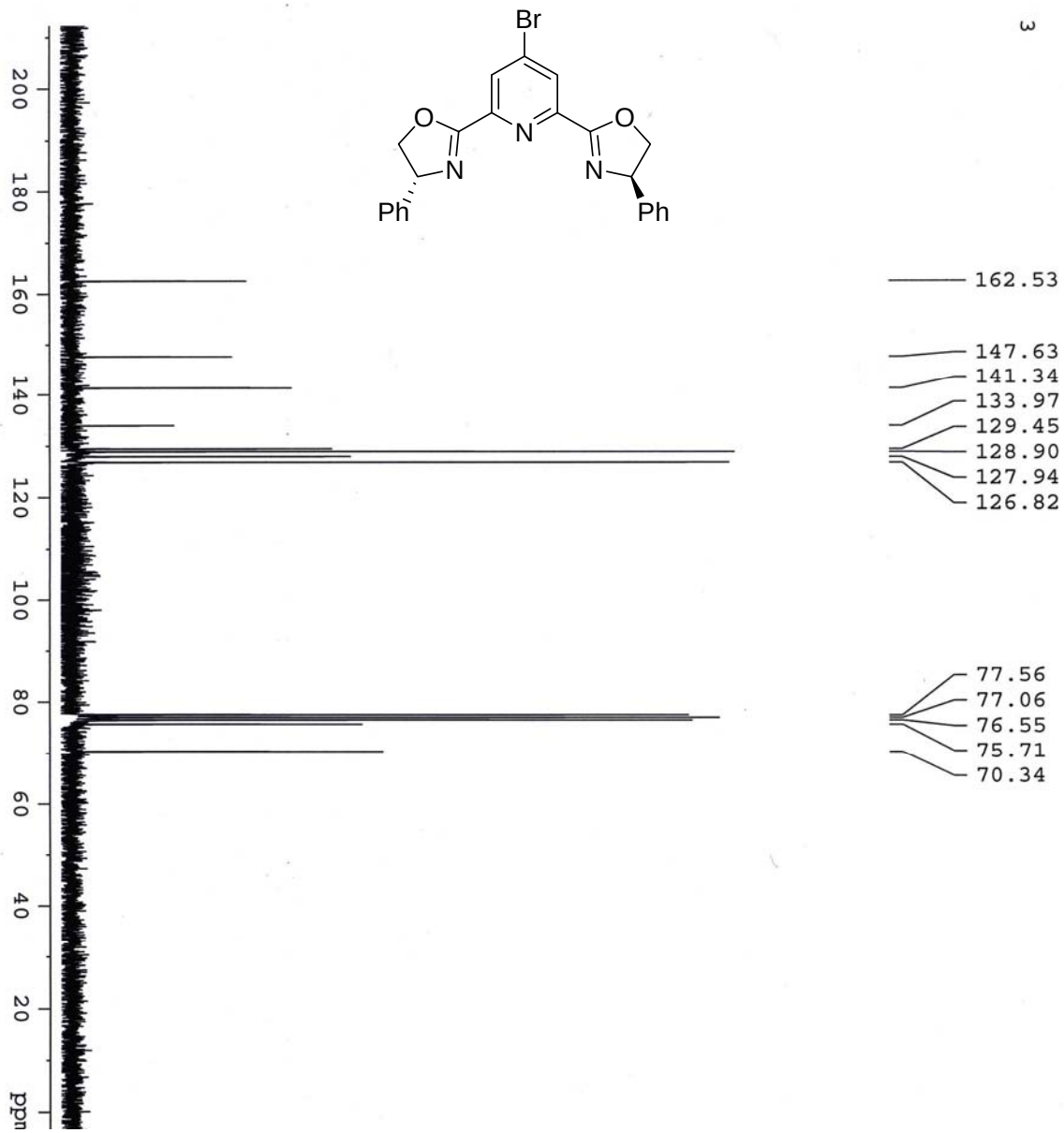

2
Br
H
N
Ph
HO
O
N
O
H
N
Ph
OH
1.781
4.915
1.000
2.065
0.933
0.025
8.517
8.487
8.361
7.321
7.305
7.274
7.261
7.247
5.206
5.187
5.177
5.168
5.157
5.138
3.932
3.913
2.948
ppm

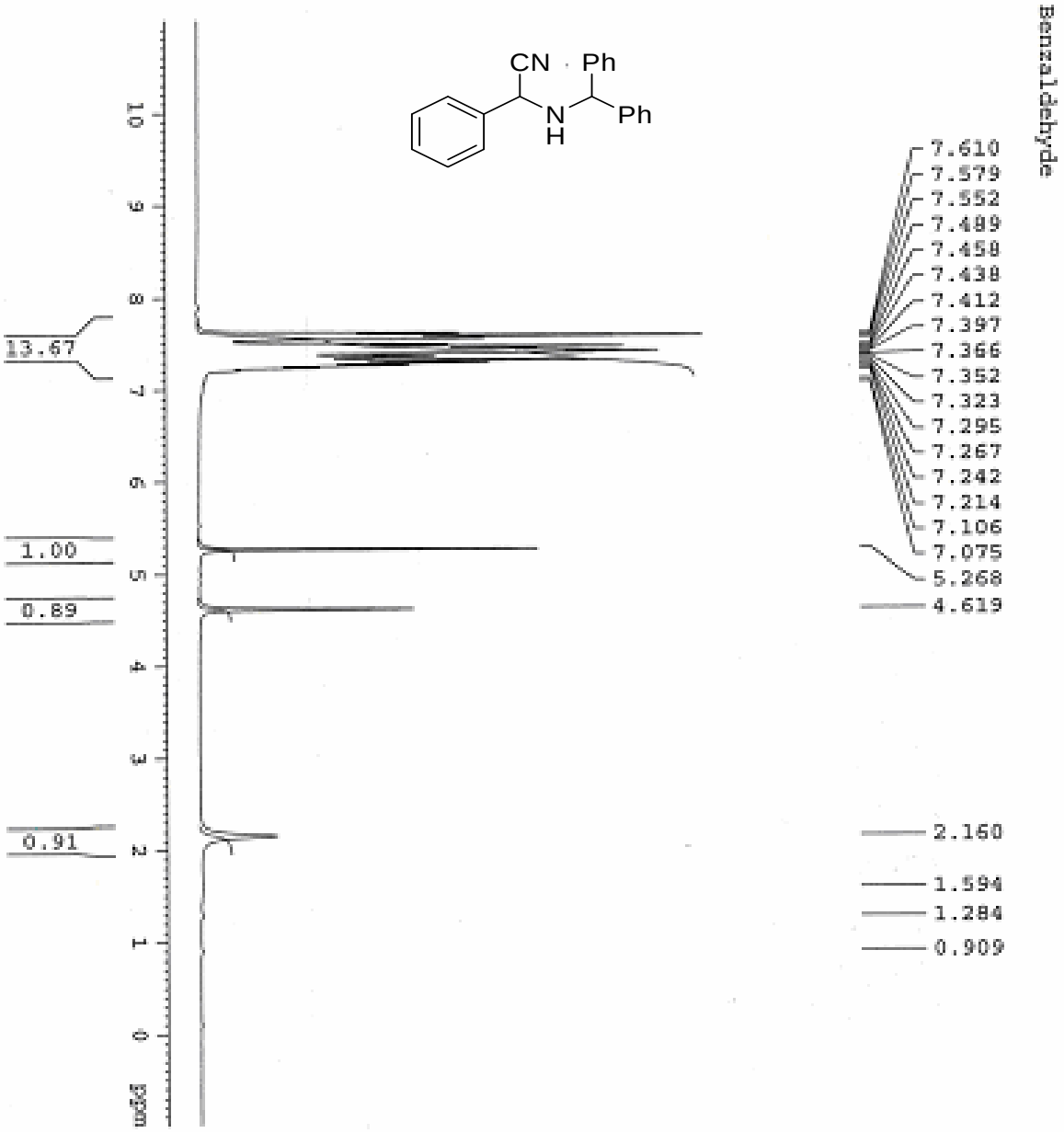

Br
Ph
H
N
N
H
N
Ph
HO
O
O
OH
2
5.206
5.187
5.177
5.168
5.157
5.138
3.932
3.913
2.948
1.000
2.065
0.933
5.4
5.2
5.0
4.8
4.6
4.4
4.2
4.0
3.8
3.6
3.4
3.2
3.0
2.8
ppm

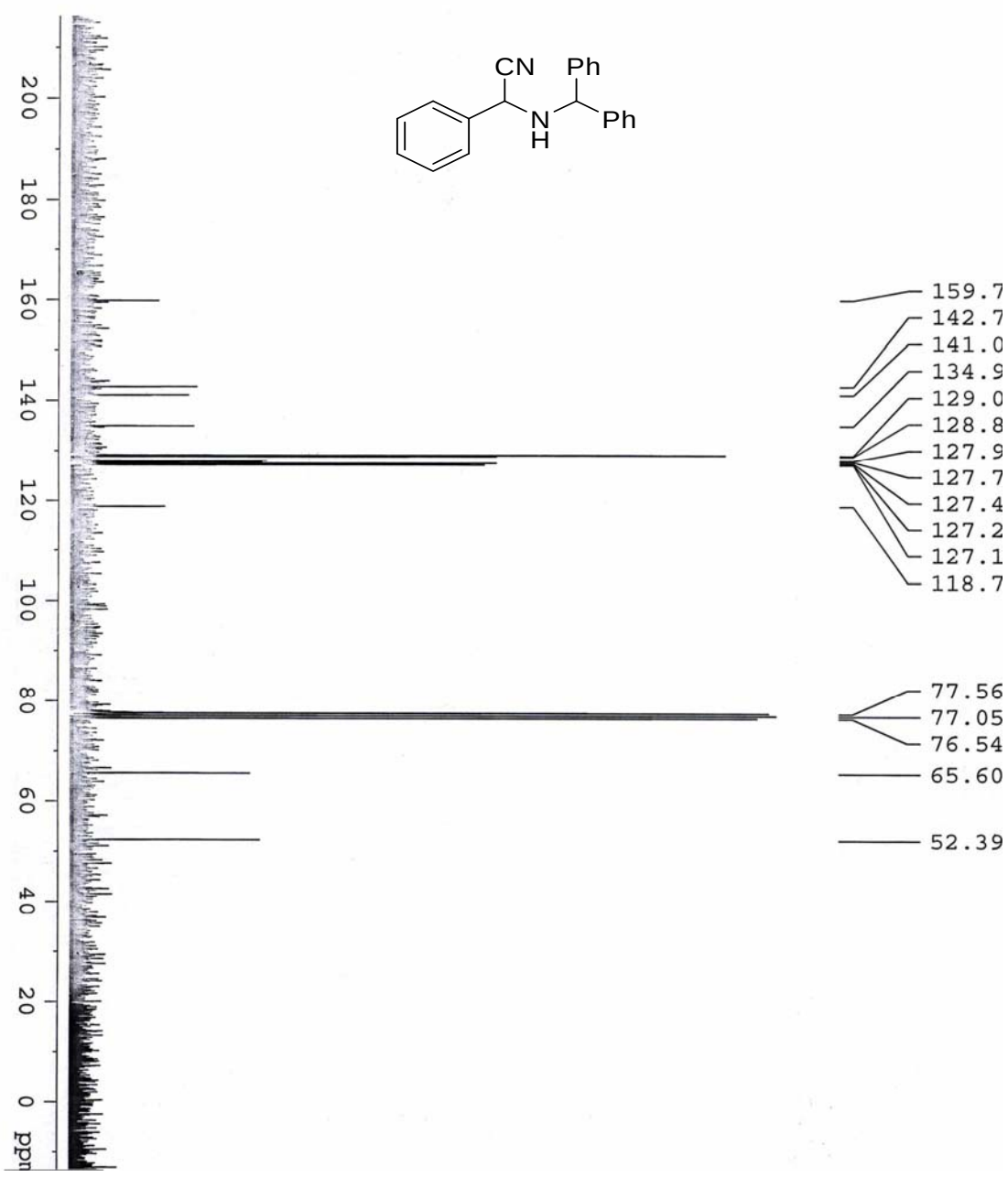

Br
H
N
Ph
HO
O
N
O
H
N
Ph
OH
200
180
160
140
120
100
80
60
40
20
ppm
162.46
149.79
138.71
136.30
128.83
128.41
127.88
126.62
77.47
76.96
76.45
65.91
55.86

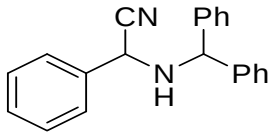

3
Br
O
N
O
N
N
Ph
Ph
8.513
8.469
7.394
7.386
7.362
7.338
7.315
7.303
7.295
7.287
7.252
5.487
5.447
5.411
5.368
4.959
4.924
4.919
4.883
4.839
4.461
4.426
4.391
4.347
1.000
7.780
1.533
1.636
1.648
13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm

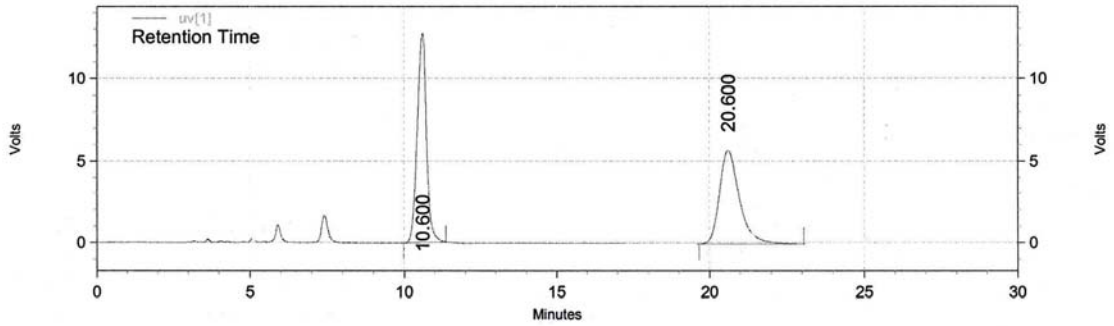

Br
O
N
O
N
N
Ph
Ph
3
5.487
5.447
5.411
5.368
4.959
4.924
4.919
4.883
4.839
4.461
4.426
4.391
4.347
1.533
1.636
1.648
5.6
5.5
5.4
5.3
5.2
5.1
5.0
4.9
4.8
4.7
4.6
4.5
4.4
ppm


Br
O
N
O
N
N
Ph
Ph
3
162.53
147.63
141.34
133.97
129.45
128.90
127.94
126.82
77.56
77.06
76.55
75.71
70.34
200
180
160
140
120
100
80
60
40
20
ppm


CN
Ph
N
H
Ph
Benzaldehyde
7.610
7.579
7.552
7.489
7.458
7.438
7.412
7.397
7.366
7.352
7.323
7.295
7.267
7.242
7.214
7.106
7.075
5.268
4.619
2.160
1.594
1.284
0.909
13.67
1.00
0.89
0.91
10
9
8
7
6
5
4
3
2
1
0
ppm

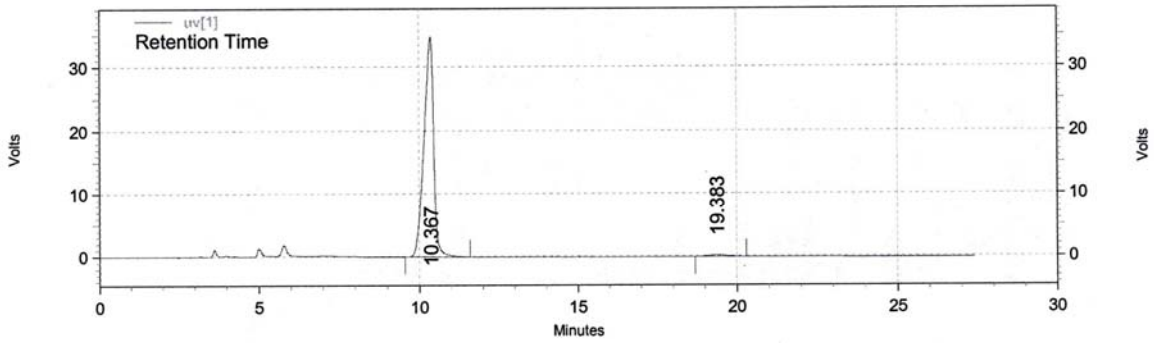

CN
Ph
N
H
Ph
200
180
160
140
120
100
80
60
40
20
0
ppm
159.7
142.7
141.0
134.9
129.0
128.8
127.9
127.7
127.4
127.2
127.1
118.7
77.56
77.05
76.54
65.60
52.39





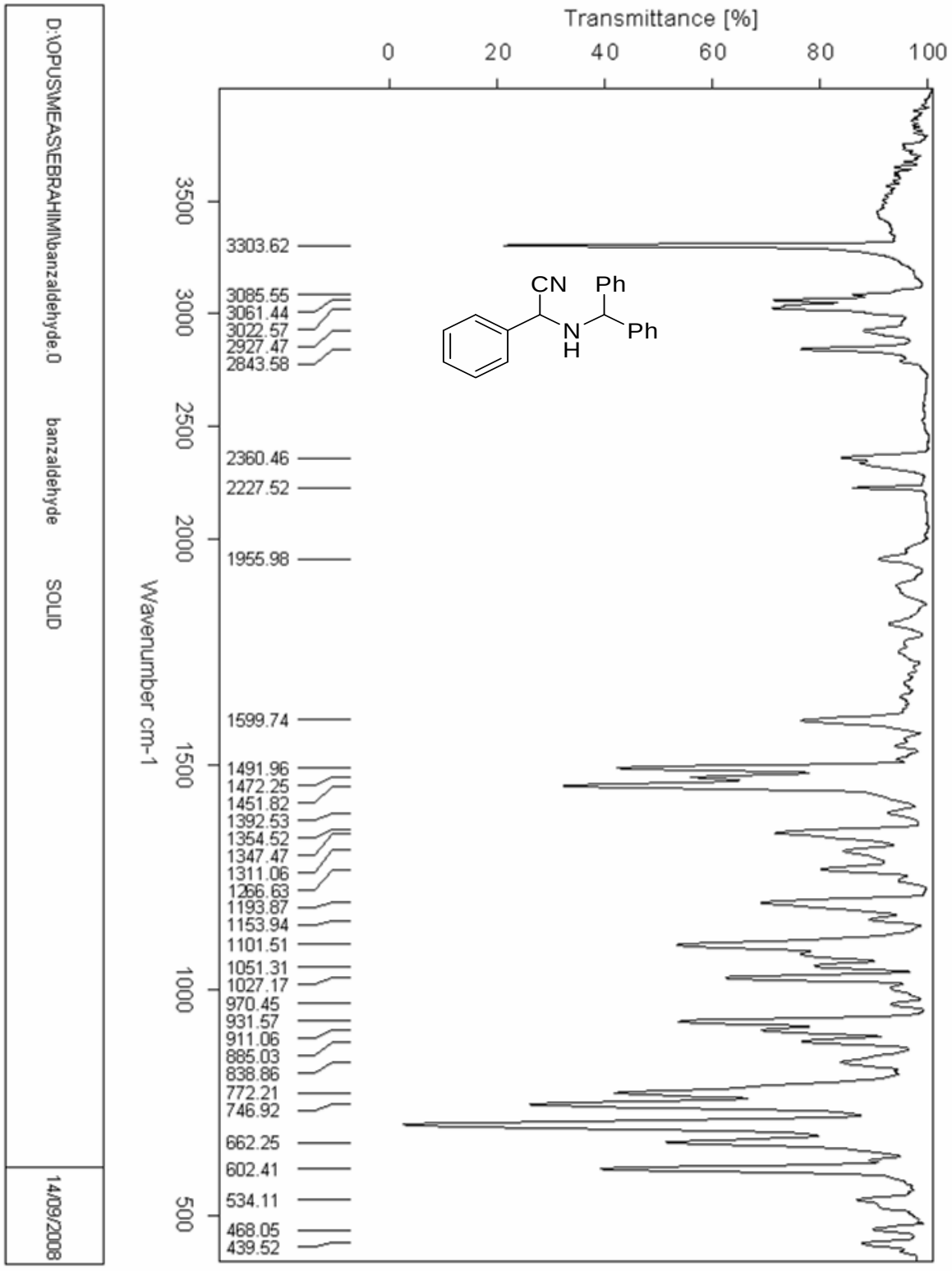
uv[1] Results

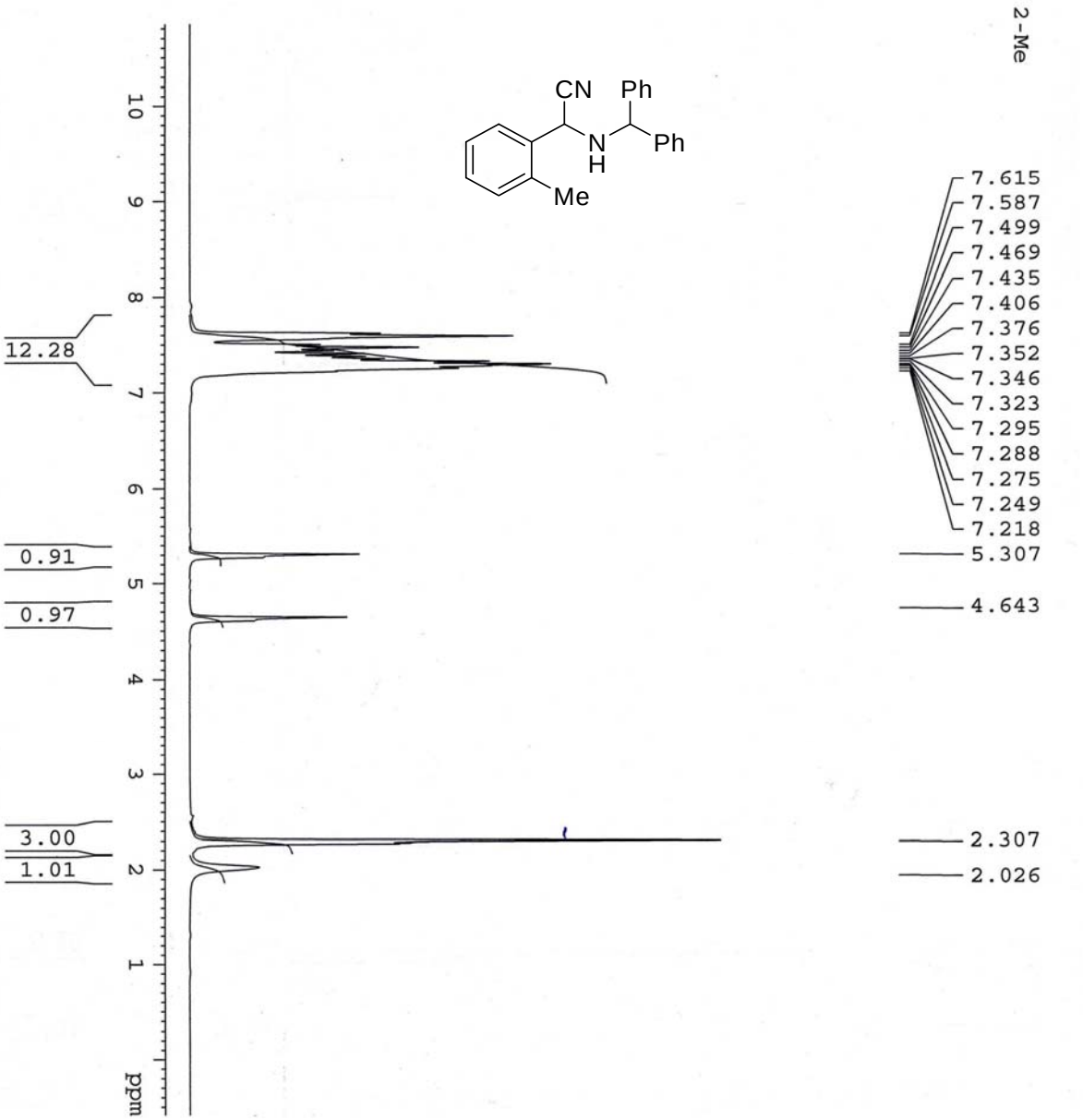
Retention Time	Area	Area %	Height	Height %
10.600	267269	50.95	12757	68.95
20.600	257322	49.05	5744	31.05
Totals	524591	100.00	18501	100.00

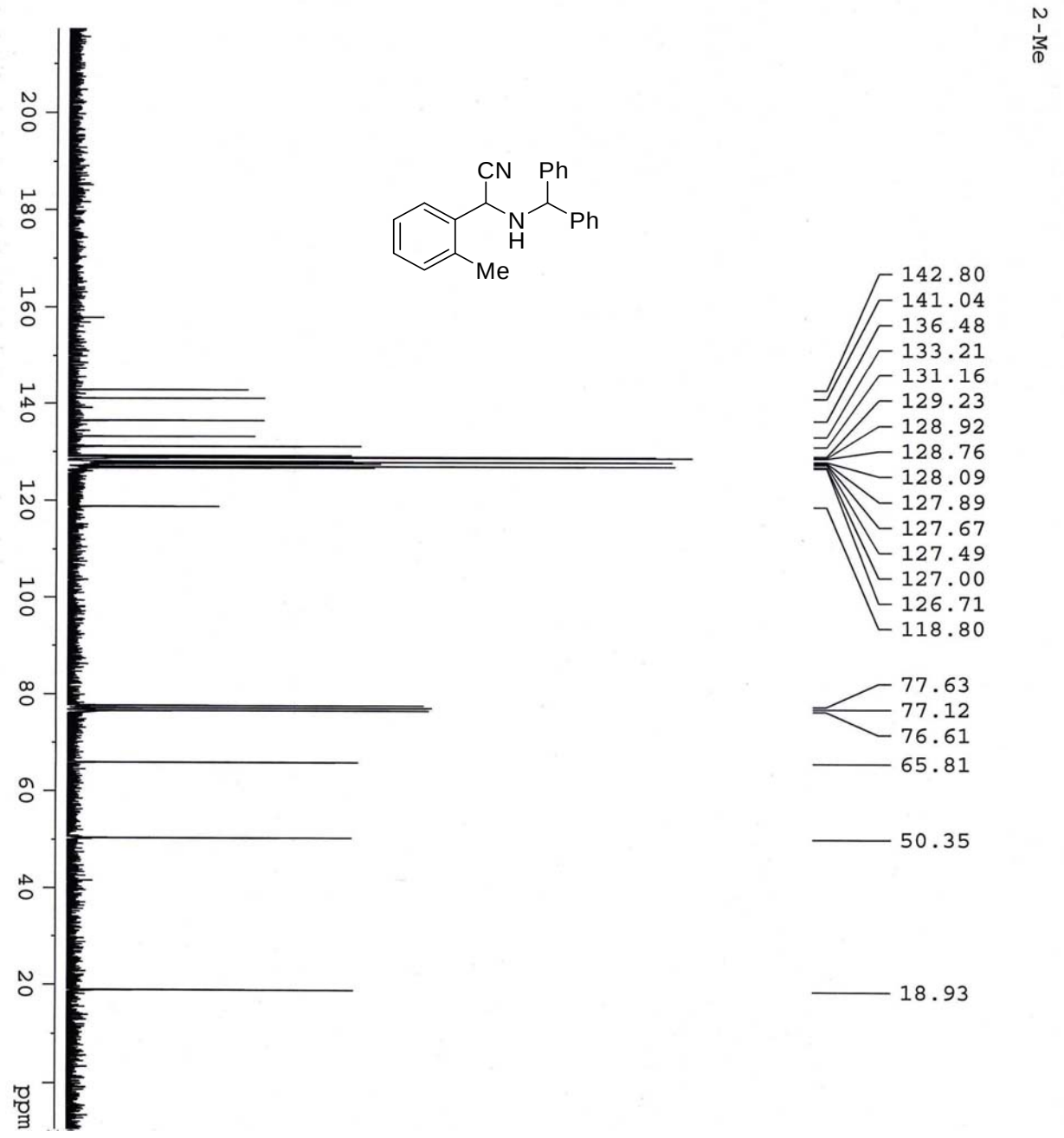


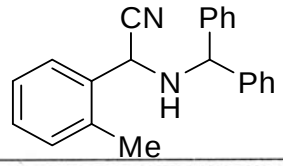
uv[1] Results

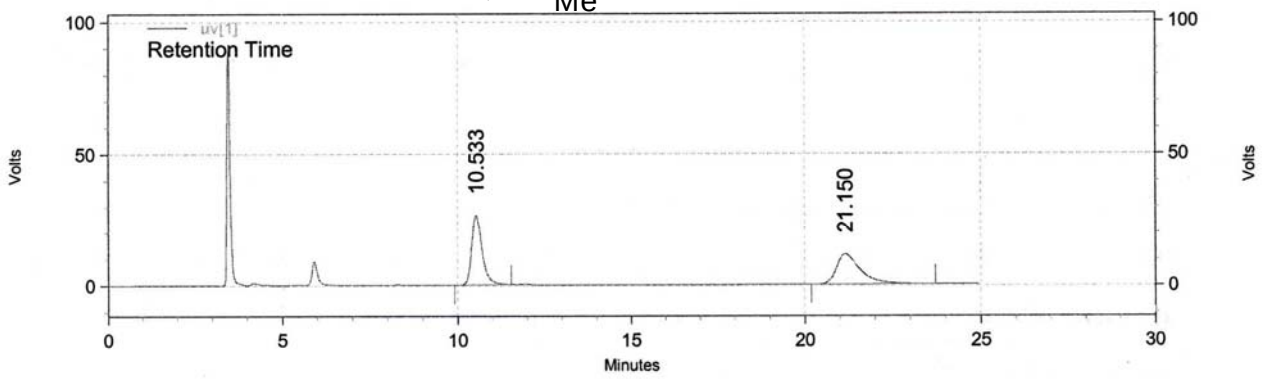
Retention Time	Area	Area %	Height	Height %
10.367	739747	98.60	34836	99.22
19.383	10500	1.40	275	0.78
Totals	750247	100.00	35111	100.00


D:\OPUS\MEAS\EBRAHIM\banzaldehyde.0
banzaldehyde
SOLID
14/09/2008
Transmittance [%]
0
20
40
60
80
100
Wavenumber cm-1
3500
3000
2500
2000
1500
1000
500
3303.62
3085.55
3061.44
3022.57
2927.47
2843.58
2360.46
2227.52
1955.98
1599.74
1491.96
1472.25
1451.82
1392.53
1354.52
1347.47
1311.06
1266.63
1193.87
1153.94
1101.51
1051.31
1027.17
970.45
931.57
911.06
885.03
838.86
772.21
746.92
662.25
602.41
534.11
468.05
439.52
CN
Ph
N
H
Ph


CN
Ph
N
H
Ph
Me
2-Me
7.615
7.587
7.499
7.469
7.435
7.406
7.376
7.352
7.346
7.323
7.295
7.288
7.275
7.249
7.218
5.307
4.643
2.307
2.026
12.28
0.91
0.97
3.00
1.01
ppm

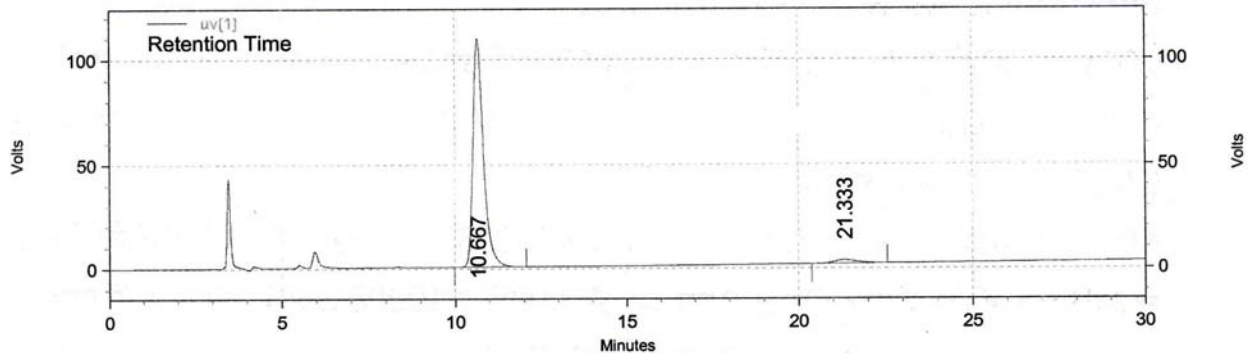

2-Me
CN
Ph
N
H
Ph
Me
142.80
141.04
136.48
133.21
131.16
129.23
128.92
128.76
128.09
127.89
127.67
127.49
127.00
126.71
118.80
77.63
77.12
76.61
65.81
50.35
18.93
200
180
160
140
120
100
80
60
40
20
ppm





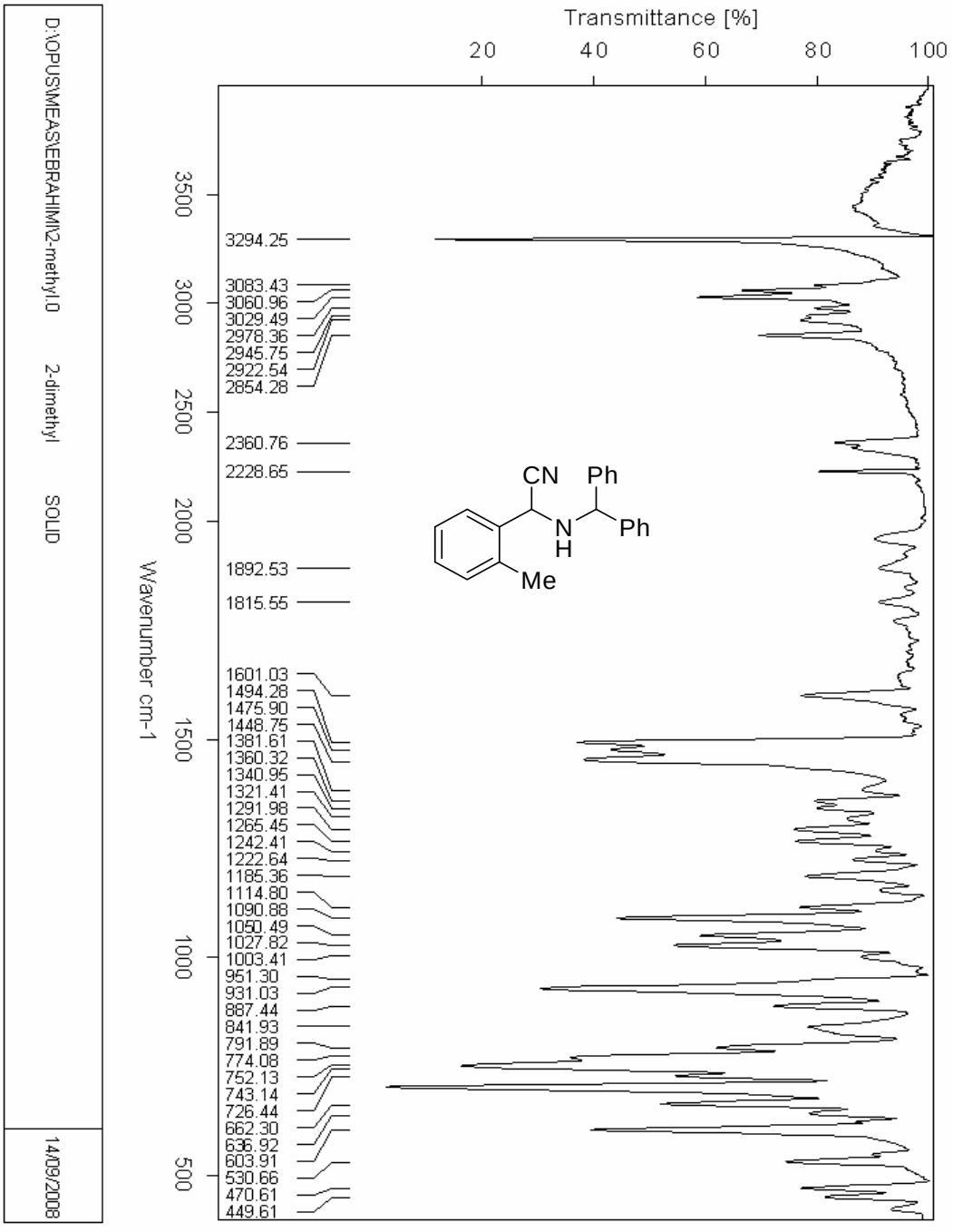
uv[1] Results

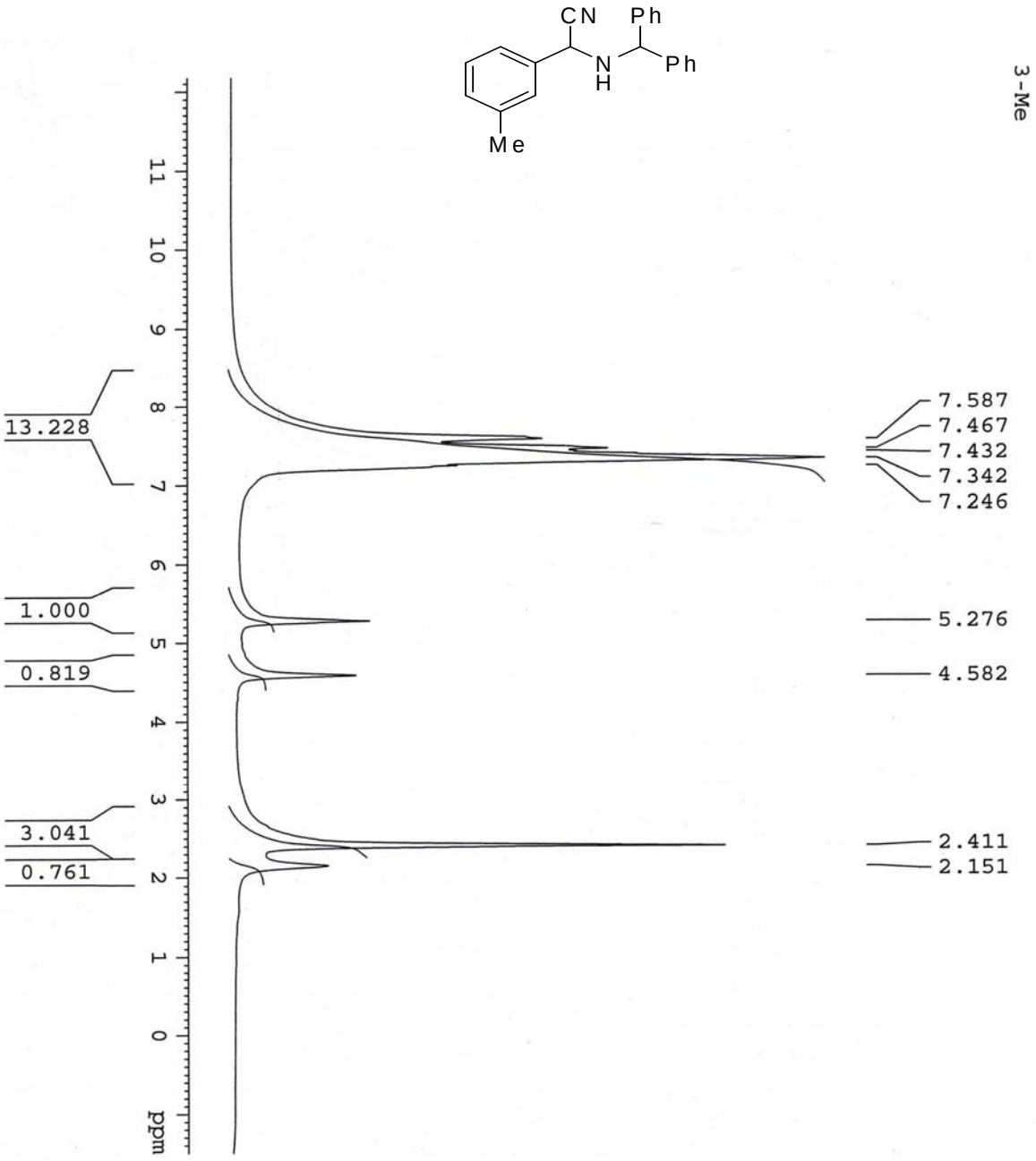
Retention Time	Area	Area %	Height	Height %
10.533	549351	50.69	26457	70.00
21.150	534444	49.31	11341	30.00
Totals	1083795	100.00	37798	100.00

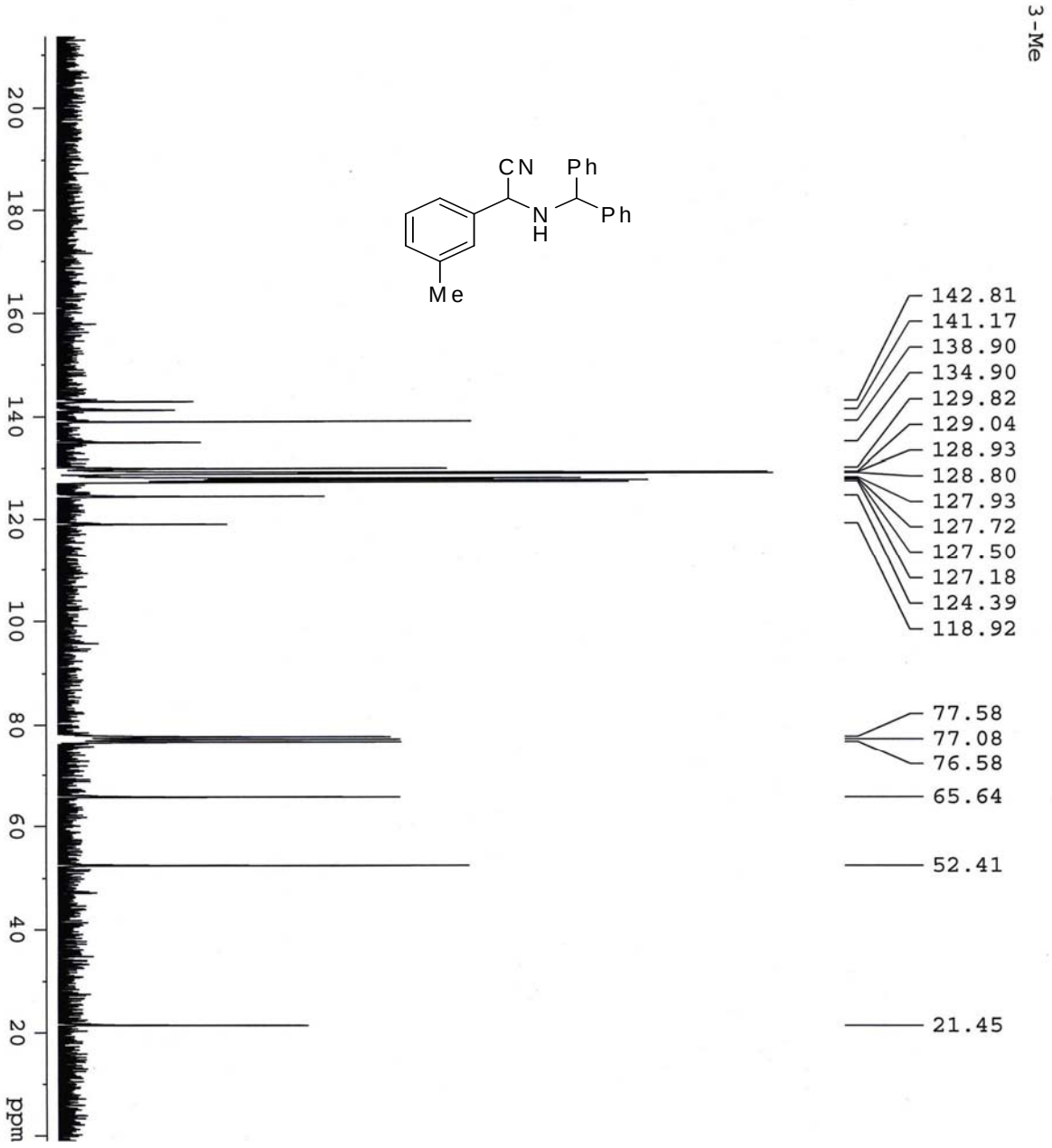


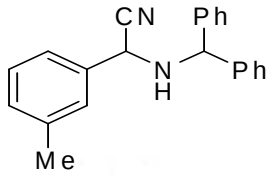
uv[1] Results

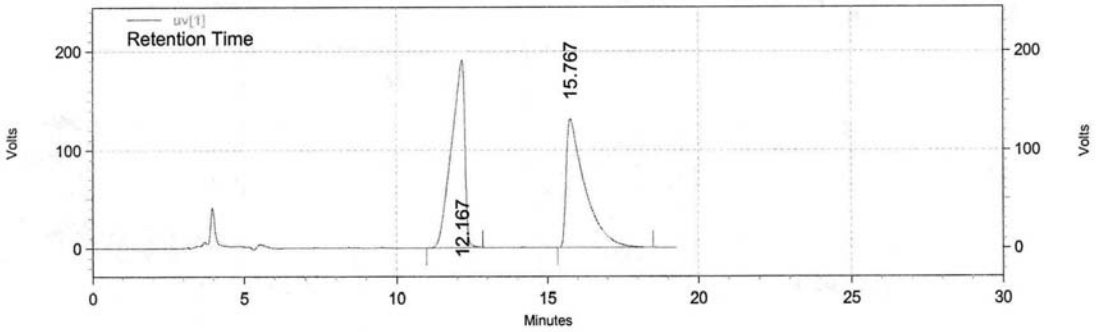
Retention Time	Area	Area %	Height	Height %
10.667	2314169	96.88	109299	98.49
21.333	74631	3.12	1675	1.51
Totals	2388800	100.00	110974	100.00


D:\OPUS\MEAS\EBRAHIMI\2-methyl.0
2-dimethyl
SOLID
14/09/2008
Transmittance [%]
20
40
60
80
100
Wavenumber cm-1
3500
3000
2500
2000
1500
1000
500
3294.25
3083.43
3060.96
3029.49
2978.36
2945.75
2922.54
2854.28
2360.76
2228.65
1892.53
1815.55
1601.03
1494.28
1475.90
1448.75
1381.61
1360.32
1340.95
1321.41
1291.98
1265.45
1242.41
1222.64
1185.36
1114.80
1090.88
1050.49
1027.82
1003.41
951.30
931.03
887.44
841.93
791.89
774.08
752.13
743.14
726.44
662.30
636.92
603.91
530.66
470.61
449.61
CN
Ph
N
H
Ph
Me


CN
Ph
N
H
Ph
Me
3-Me
7.587
7.467
7.432
7.342
7.246
5.276
4.582
2.411
2.151
13.228
1.000
0.819
3.041
0.761
ppm

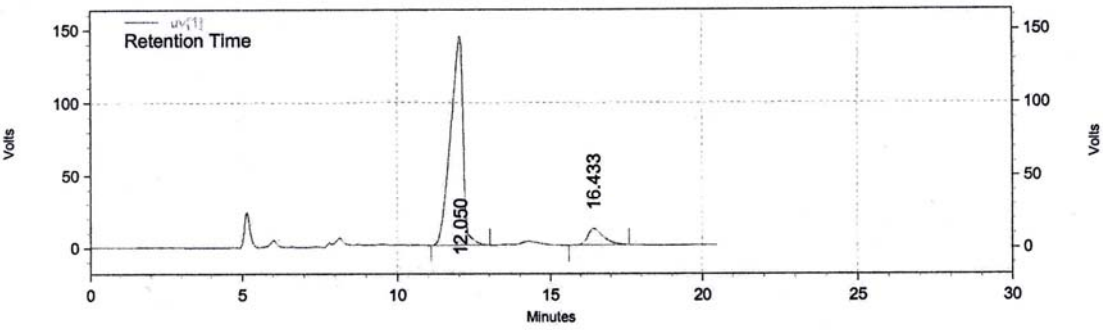

3-Me
CN
Ph
N
H
Ph
Me
142.81
141.17
138.90
134.90
129.82
129.04
128.93
128.80
127.93
127.72
127.50
127.18
124.39
118.92
77.58
77.08
76.58
65.64
52.41
21.45
200
180
160
140
120
100
80
60
40
20
ppm





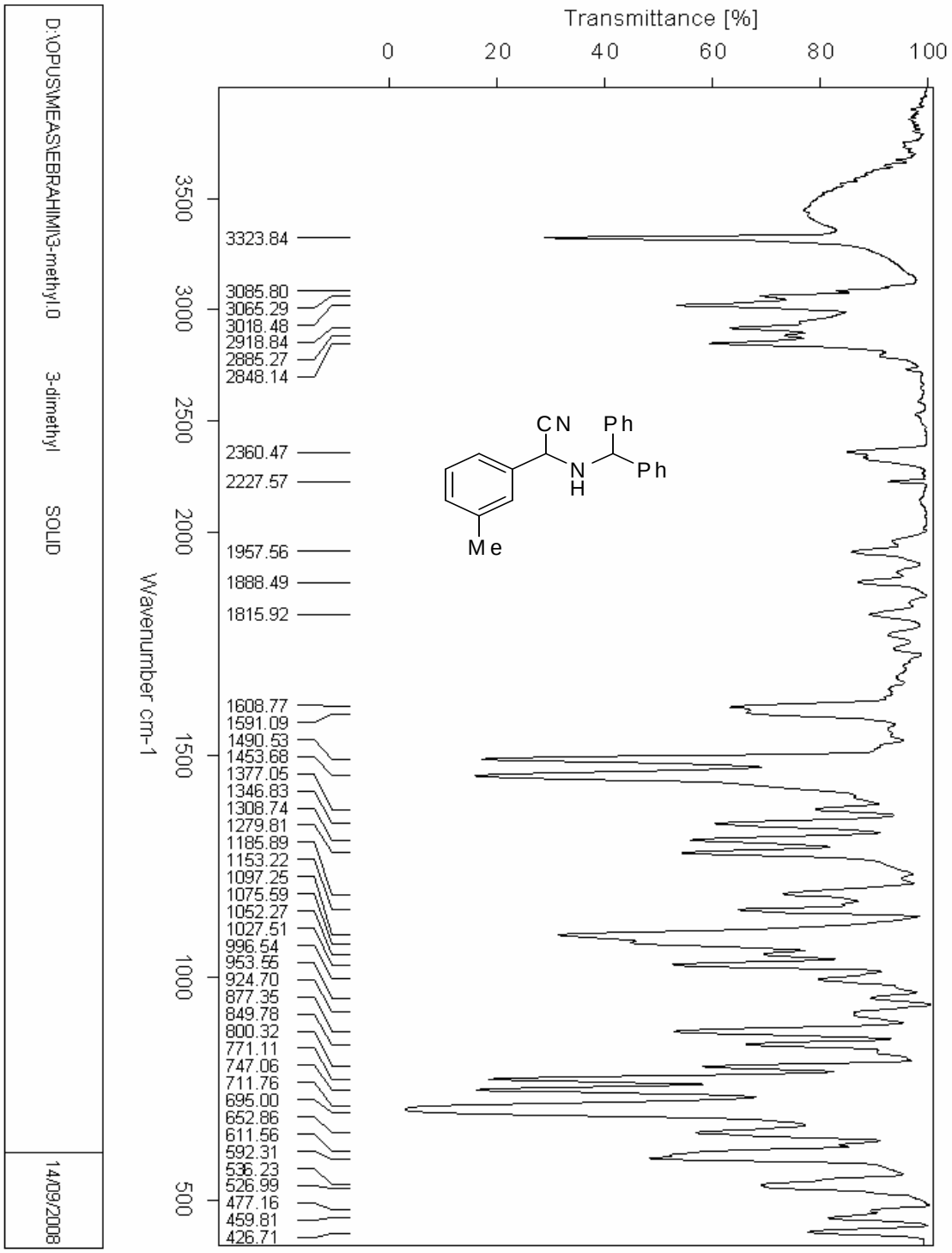
uv[1] Results

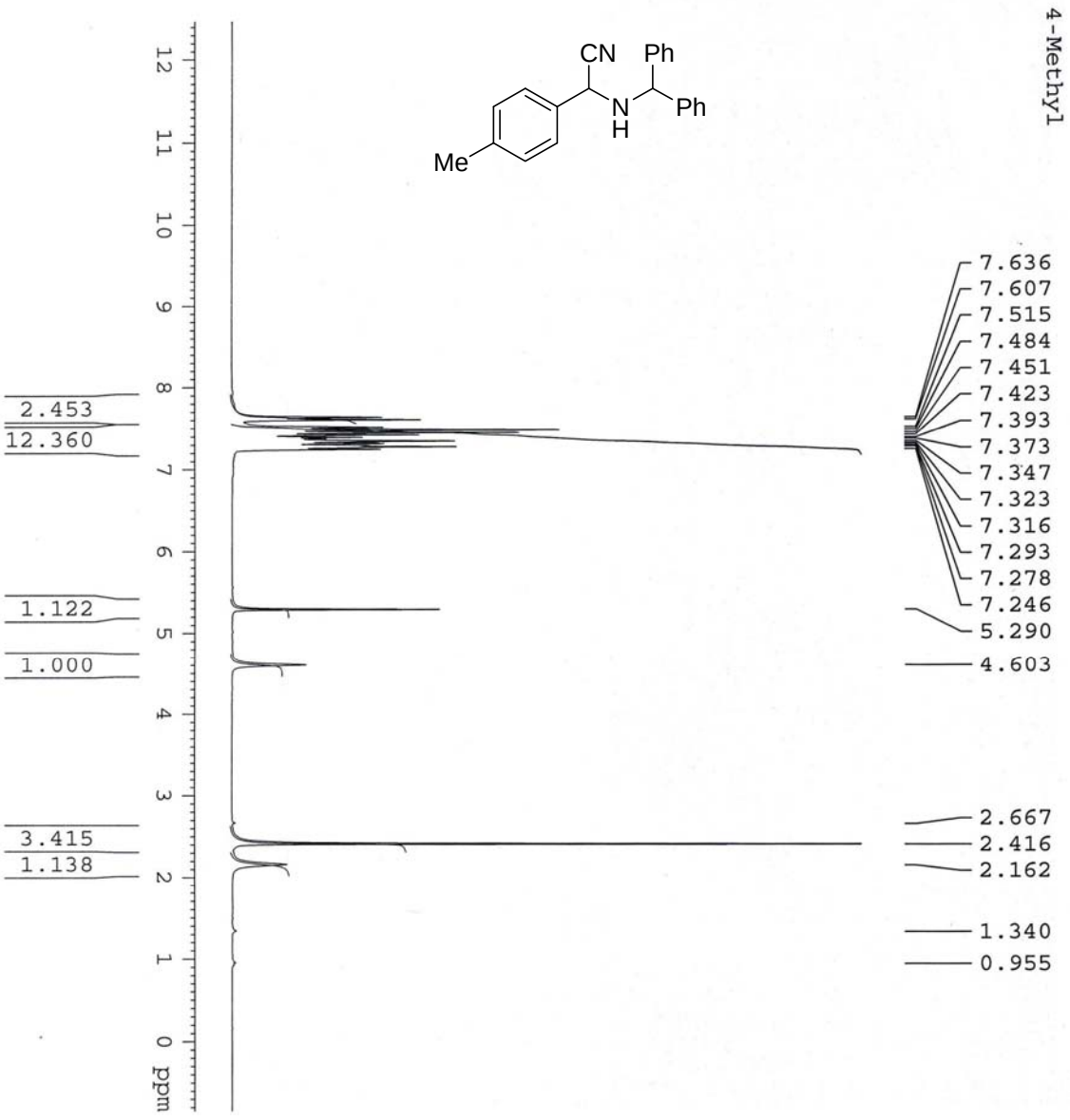
Retention Time	Area	Area %	Height	Height %
12.167	5849027	50.73	190286	59.19
15.767	5681656	49.27	131191	40.81
Totals	11530683	100.00	321477	100.00

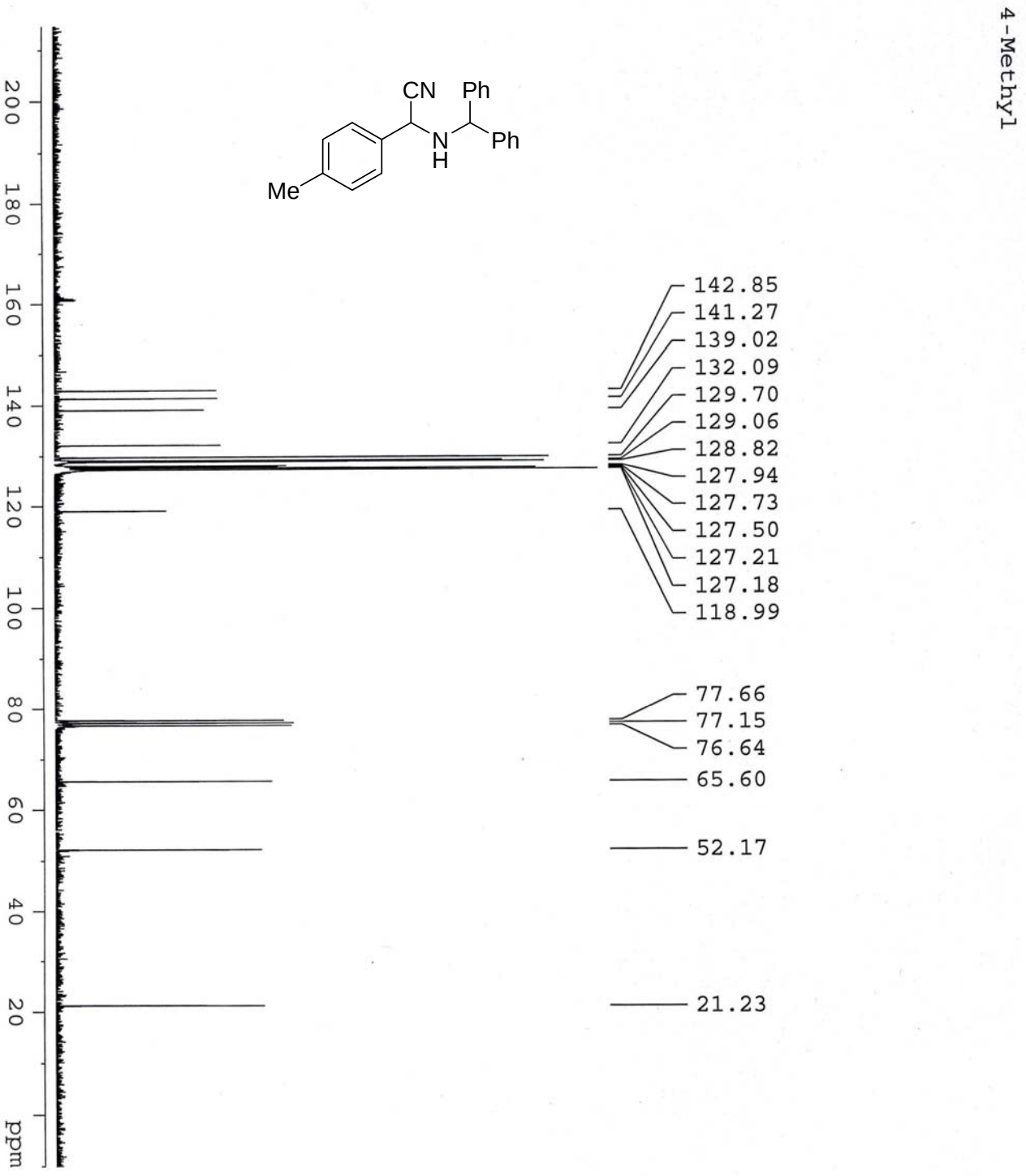


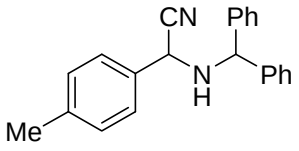
uv[1] Results

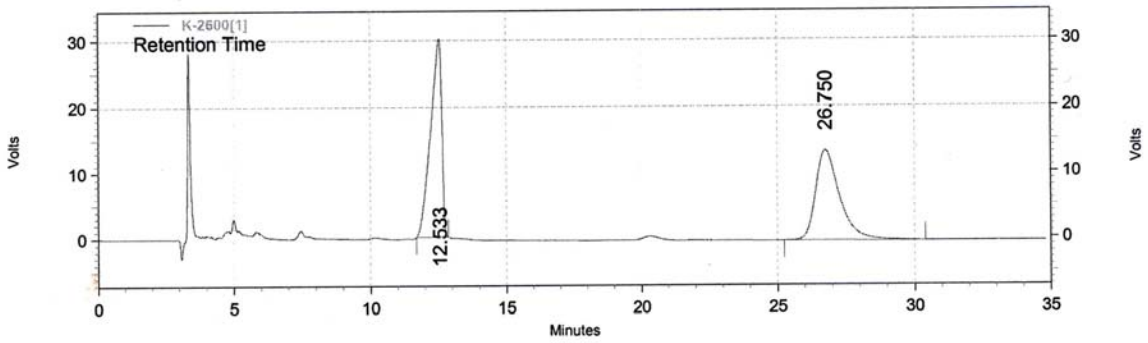
Retention Time	Area	Area %	Height	Height %
12.050	4094635	91.05	143897	92.71
16.433	402437	8.95	11307	7.29
Totals	4497072	100.00	155204	100.00


D:\OPUS\MEAS\EBRAHIMI\3-methyl.0
3-dimethyl
SOLID
14/09/2008
Transmittance [%]
0
20
40
60
80
100
Wavenumber cm-1
3500
3000
2500
2000
1500
1000
500
3323.84
3085.80
3065.29
3018.48
2918.84
2885.27
2848.14
2360.47
2227.57
1957.56
1888.49
1815.92
1608.77
1591.09
1490.53
1453.68
1377.05
1346.83
1308.74
1279.81
1185.89
1153.22
1097.25
1075.59
1052.27
1027.51
996.54
953.55
924.70
877.35
849.78
800.32
771.11
747.06
711.76
695.00
652.86
611.56
592.31
536.23
526.99
477.16
459.81
426.71
CN
Ph
N
H
Ph
Me


4-Methyl
CN
Ph
N
H
Ph
Me
7.636
7.607
7.515
7.484
7.451
7.423
7.393
7.373
7.347
7.323
7.316
7.293
7.278
7.246
5.290
4.603
2.667
2.416
2.162
1.340
0.955
2.453
12.360
1.122
1.000
3.415
1.138
ppm

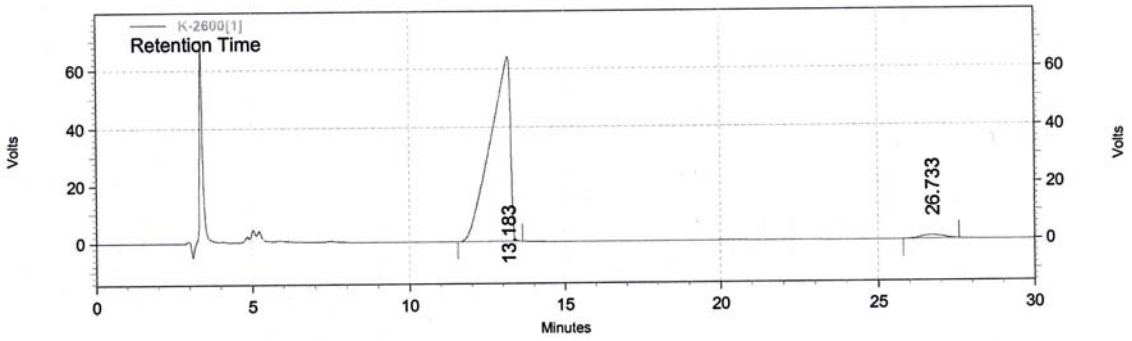

4-Methyl
CN
Ph
N
H
Ph
Me
142.85
141.27
139.02
132.09
129.70
129.06
128.82
127.94
127.73
127.50
127.21
127.18
118.99
77.66
77.15
76.64
65.60
52.17
21.23
200
180
160
140
120
100
80
60
40
20
ppm





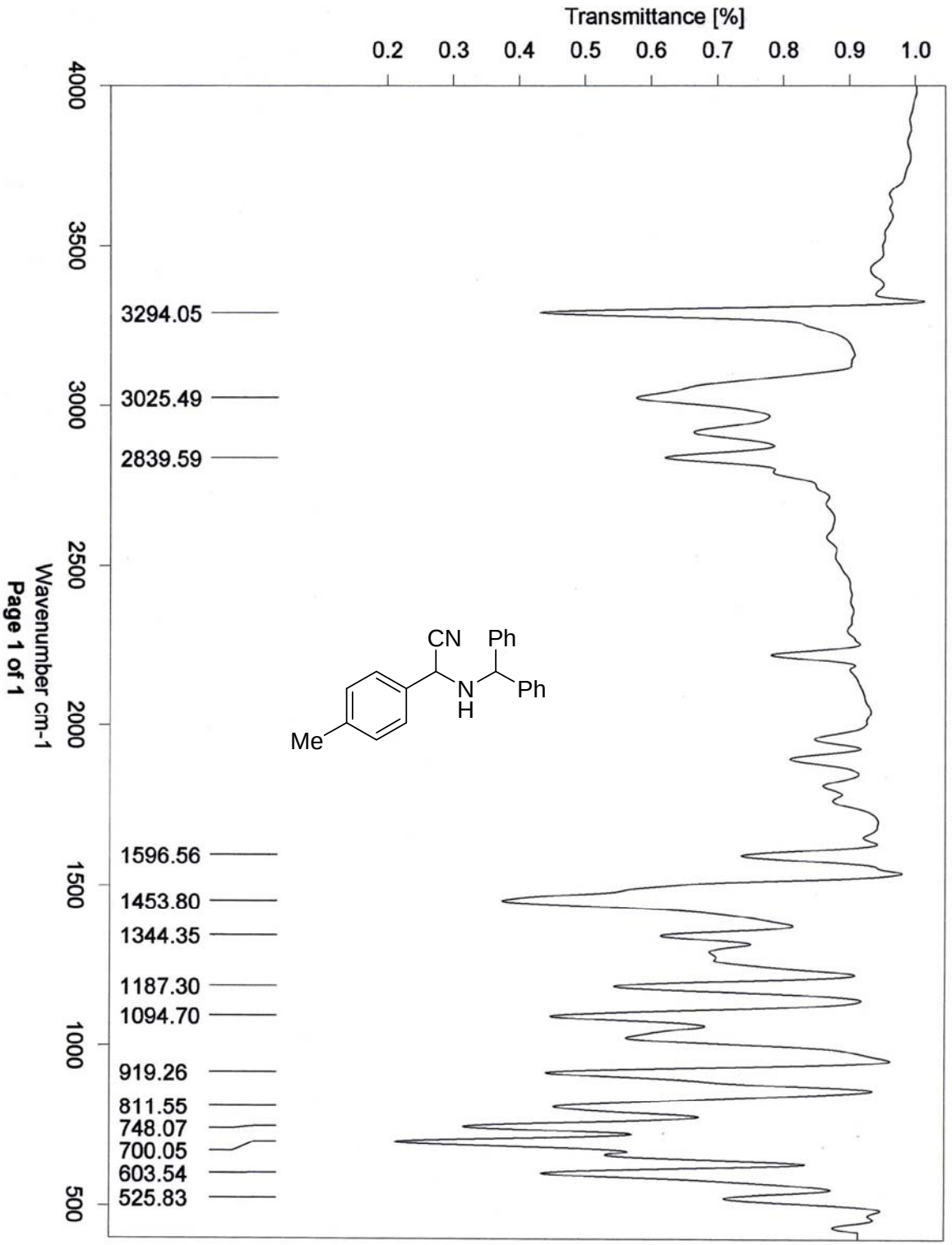
K-2600[1] Results

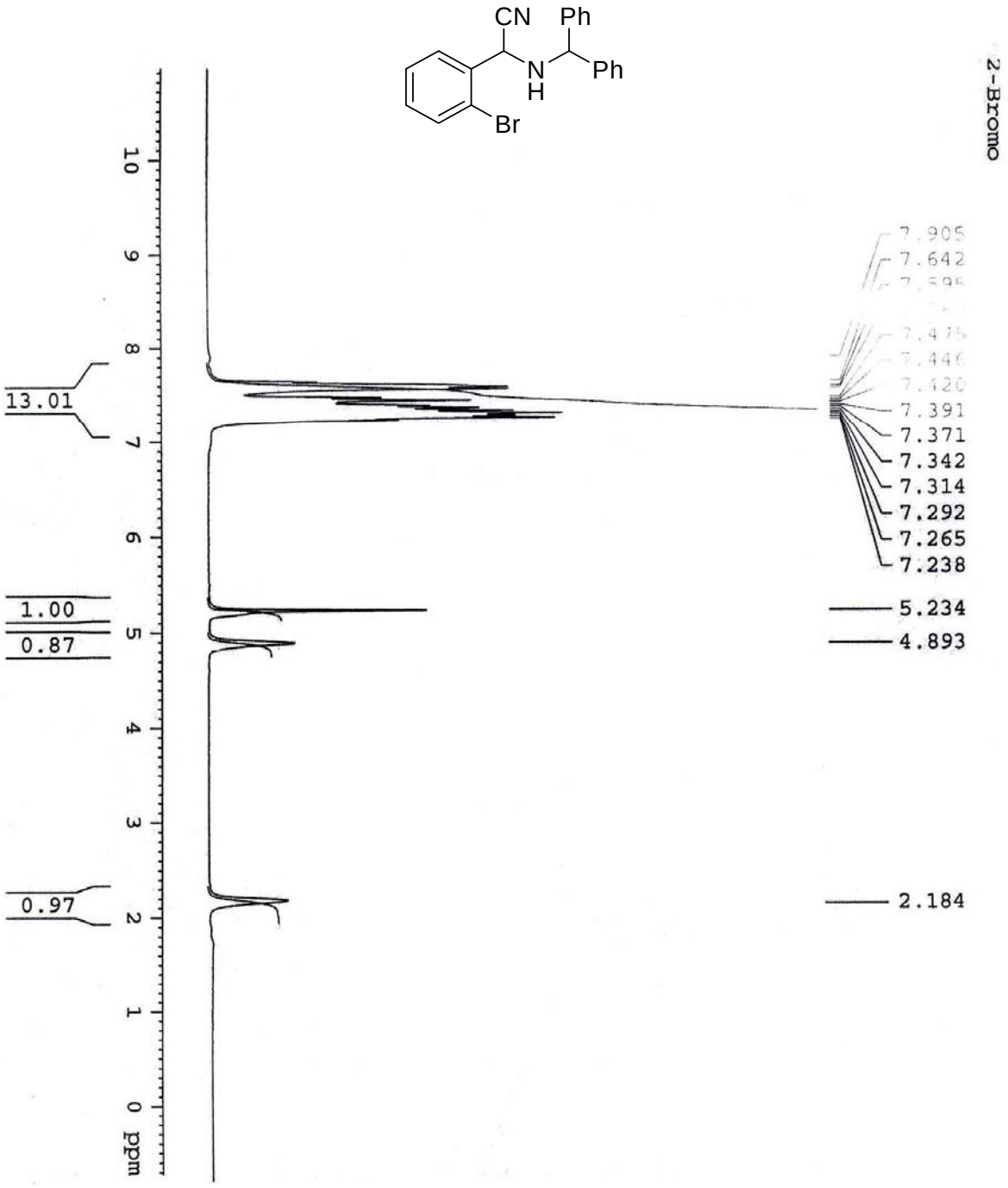
Retention Time	Area	Area %	Height	Height %
12.533	854191	50.64	29973	68.58
26.750	832562	49.36	13735	31.42
Totals	1686753	100.00	43708	100.00

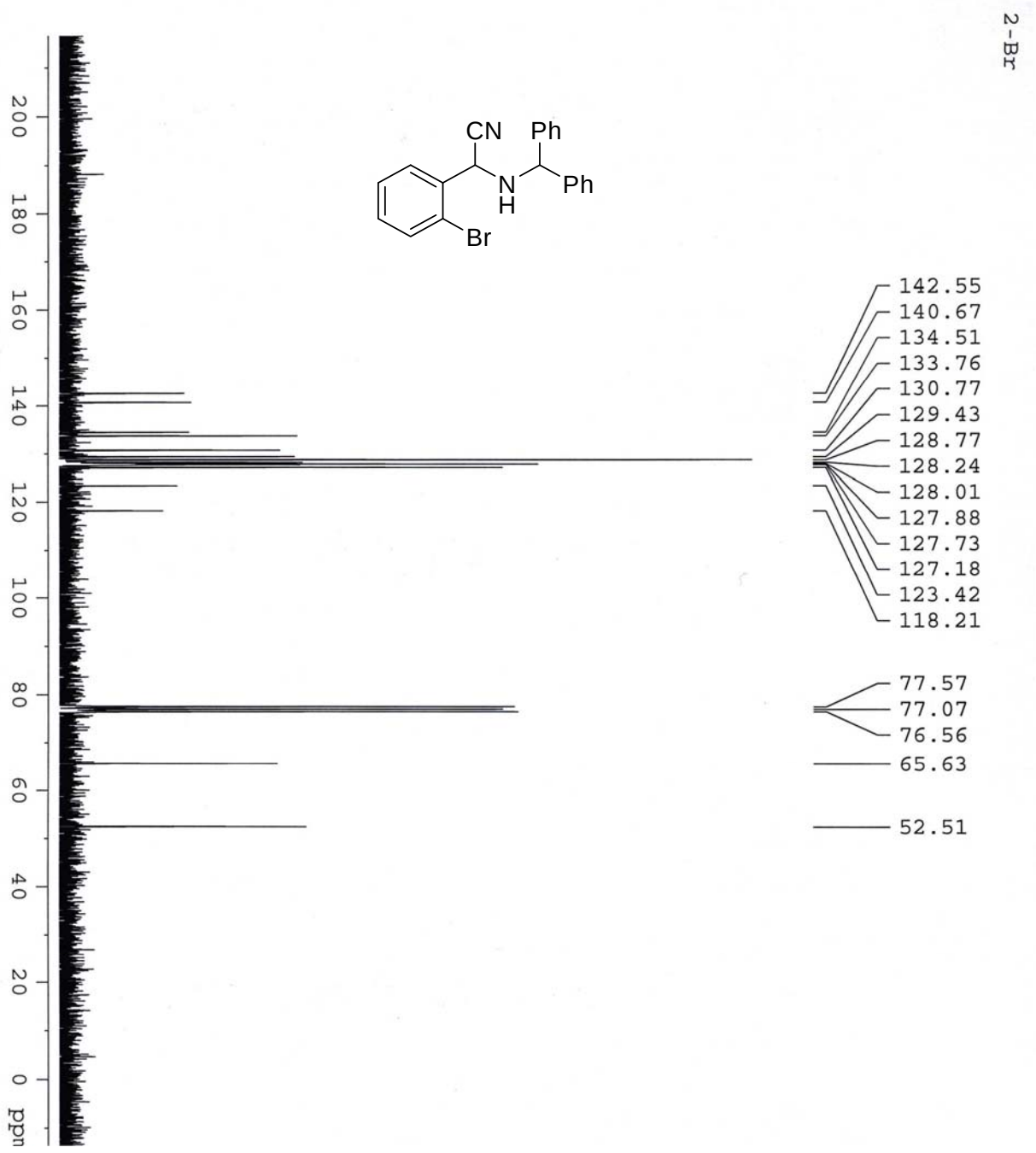


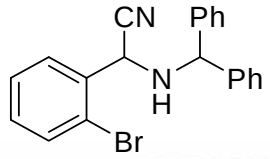
K-2600[1] Results

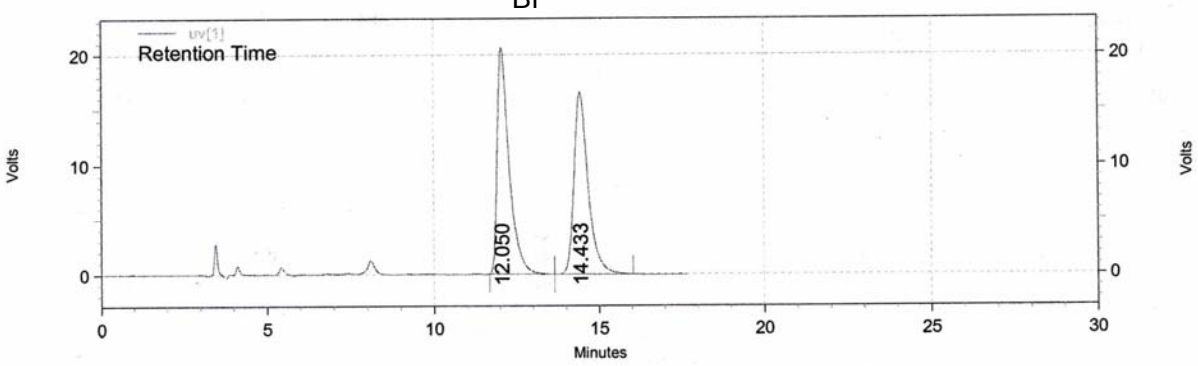
Retention Time	Area	Area %	Height	Height %
13.183	2862210	98.00	64090	98.20
26.733	58342	2.00	1173	1.80
Totals	2920552	100.00	65263	100.00


Transmittance [%]
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1.0
4000
3500
3000
2500
2000
1500
1000
500
Wavenumber cm-1
Page 1 of 1
3294.05
3025.49
2839.59
1596.56
1453.80
1344.35
1187.30
1094.70
919.26
811.55
748.07
700.05
603.54
525.83
CN
Ph
N
H
Ph
Me


CN
Ph
N
H
Ph
Br
2-Bromo
7.905
7.642
7.475
7.446
7.420
7.391
7.371
7.342
7.314
7.292
7.265
7.238
5.234
4.893
2.184
13.01
1.00
0.87
0.97
ppm

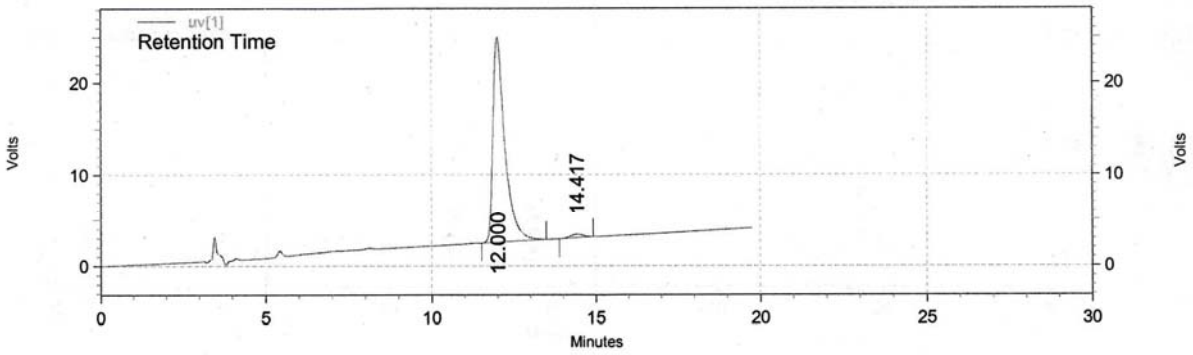

2-Br
CN
Ph
N
H
Ph
Br
142.55
140.67
134.51
133.76
130.77
129.43
128.77
128.24
128.01
127.88
127.73
127.18
123.42
118.21
77.57
77.07
76.56
65.63
52.51
200
180
160
140
120
100
80
60
40
20
0
ppm





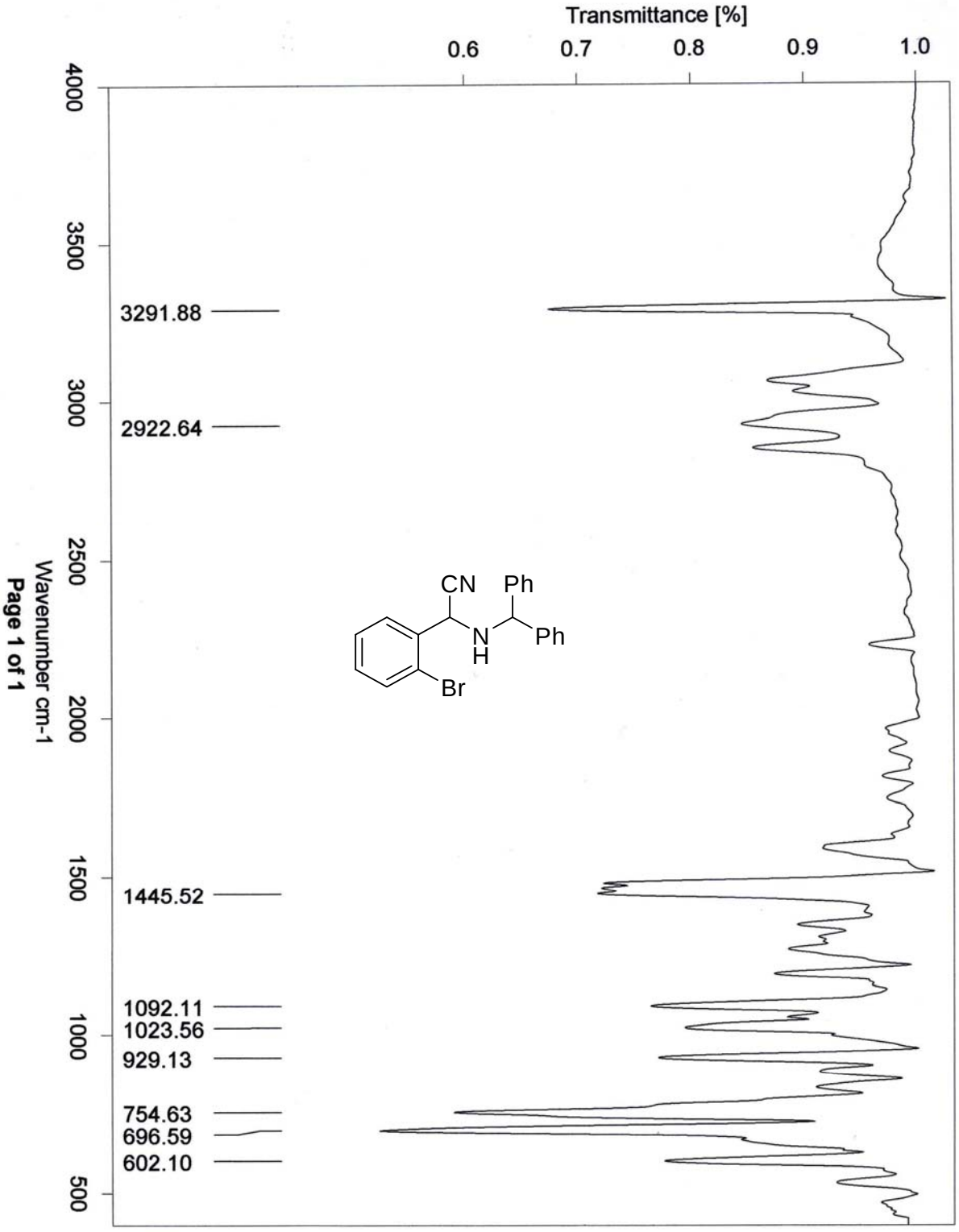
uv[1] Results

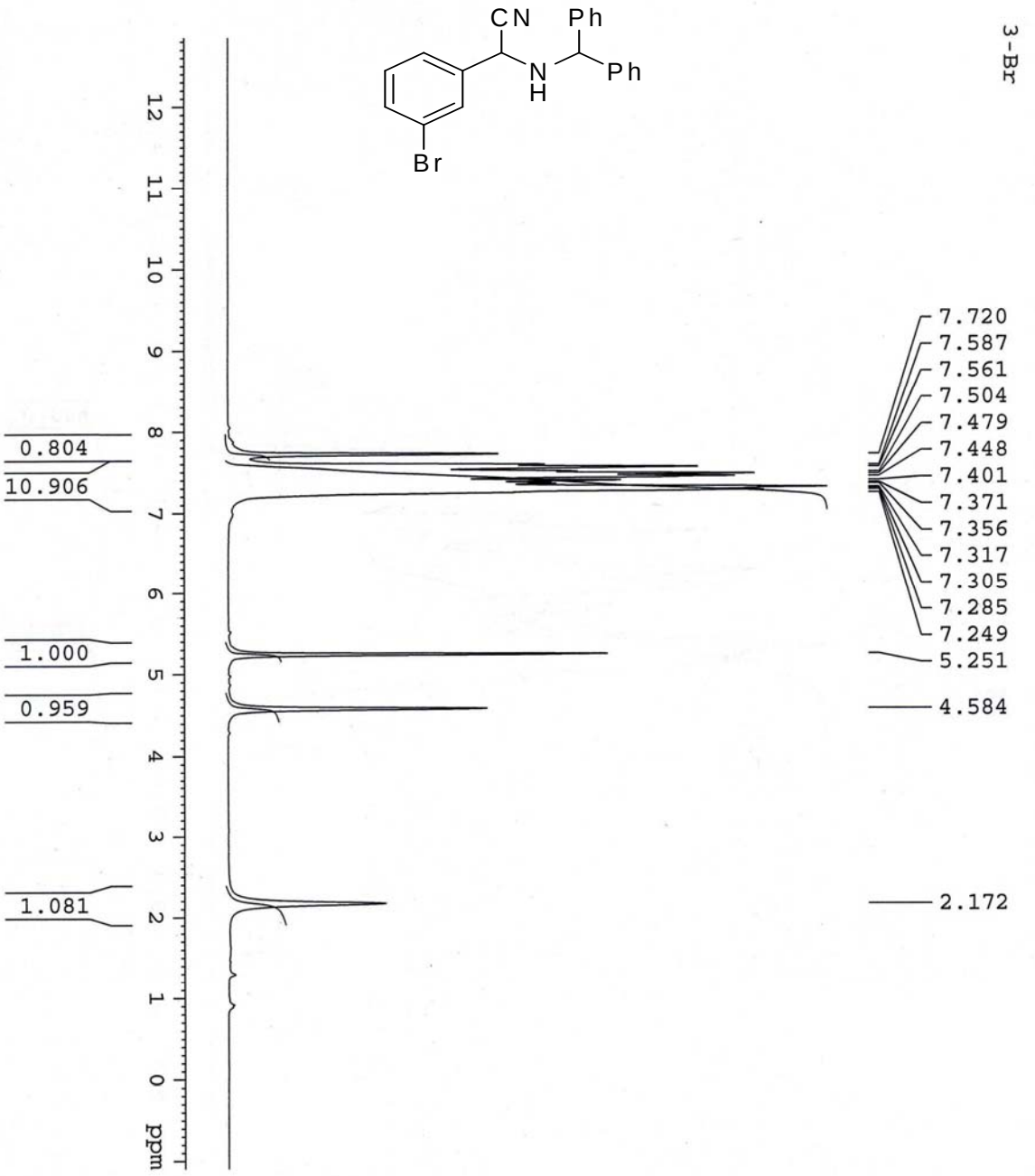
Retention Time	Area	Area %	Height	Height %
12.050	515148	51.01	20620	55.49
14.433	494827	48.99	16541	44.51
Totals	1009975	100.00	37161	100.00

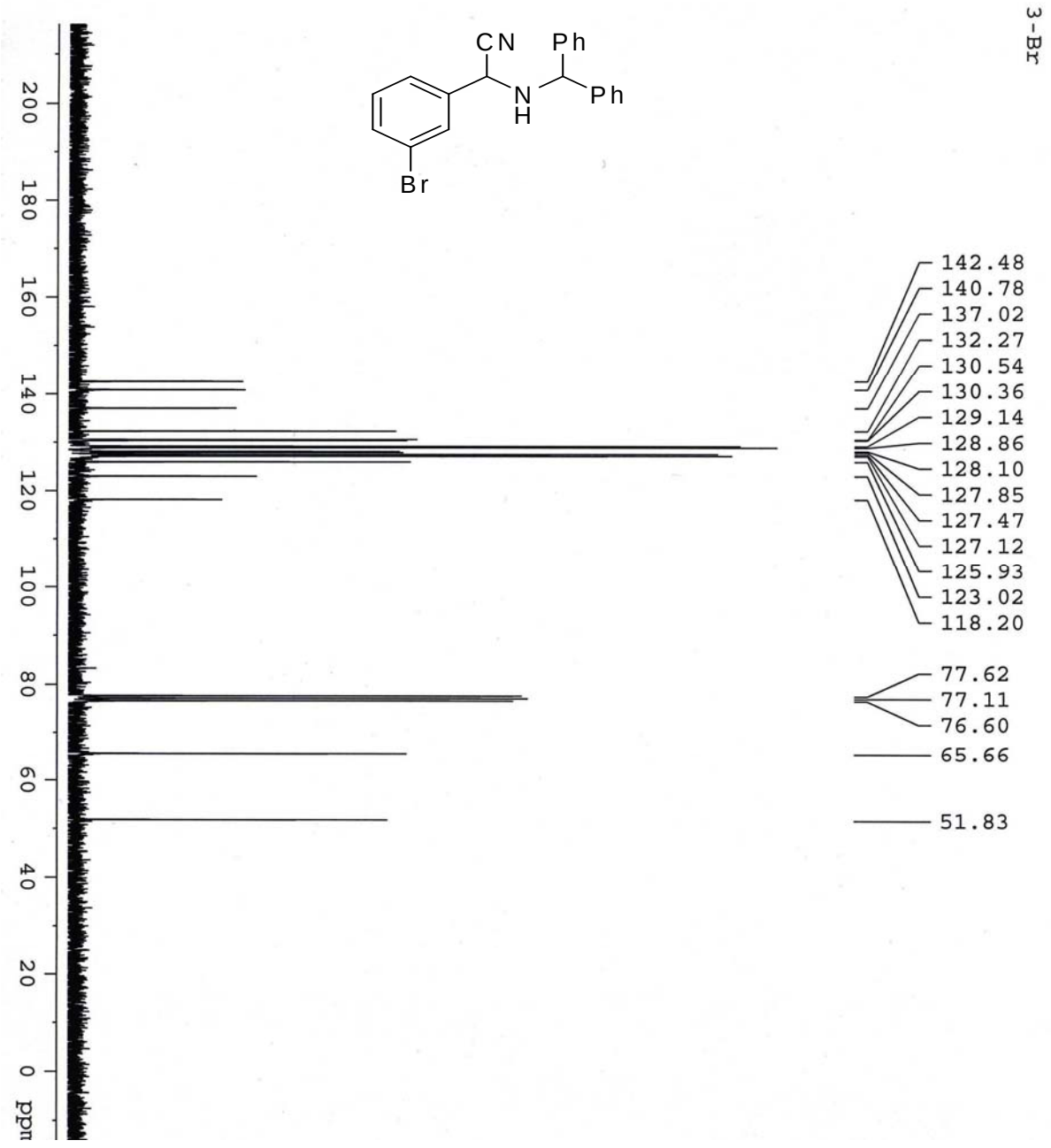


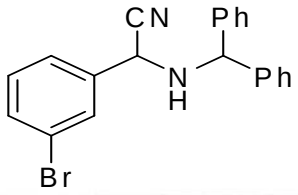
uv[1] Results

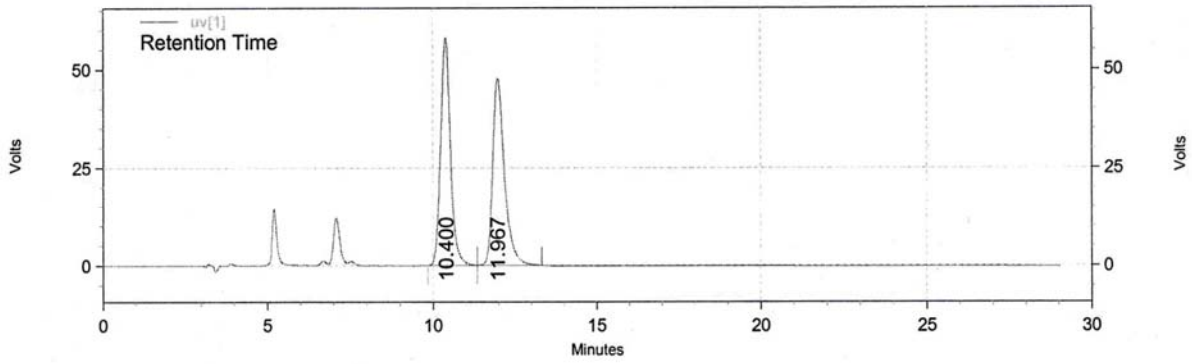
Retention Time	Area	Area %	Height	Height %
12.000	565423	98.28	22434	98.36
14.417	9881	1.72	373	1.64
Totals	575304	100.00	22807	100.00


Transmittance [%]
0.6
0.7
0.8
0.9
1.0
4000
3500
3000
2500
2000
1500
1000
500
Wavenumber cm-1
Page 1 of 1
3291.88
2922.64
1445.52
1092.11
1023.56
929.13
754.63
696.59
602.10
CN
Ph
N
H
Ph
Br


CN
Ph
N
H
Ph
Br
3-Br
7.720
7.587
7.561
7.504
7.479
7.448
7.401
7.371
7.356
7.317
7.305
7.285
7.249
5.251
4.584
2.172
0.804
10.906
1.000
0.959
1.081
ppm

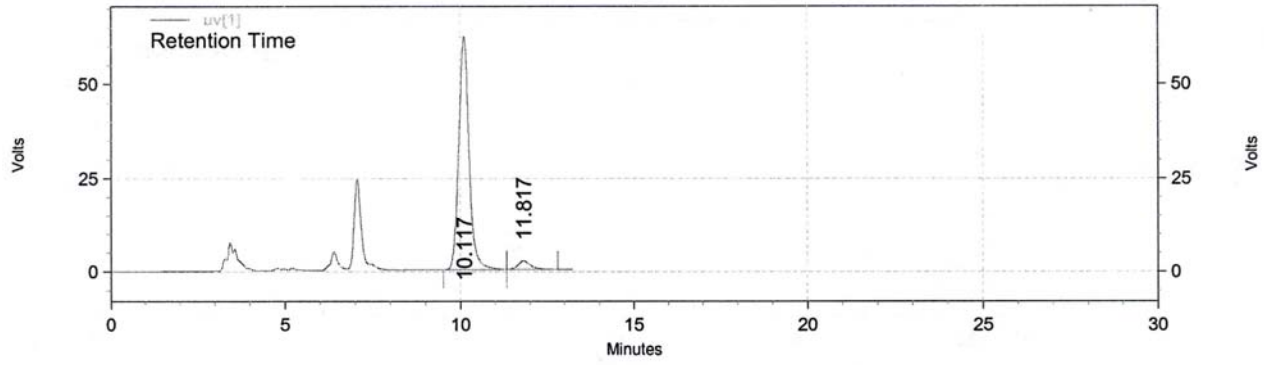

3-Br
CN
Ph
N
H
Ph
Br
142.48
140.78
137.02
132.27
130.54
130.36
129.14
128.86
128.10
127.85
127.47
127.12
125.93
123.02
118.20
77.62
77.11
76.60
65.66
51.83
200
180
160
140
120
100
80
60
40
20
0
ppm





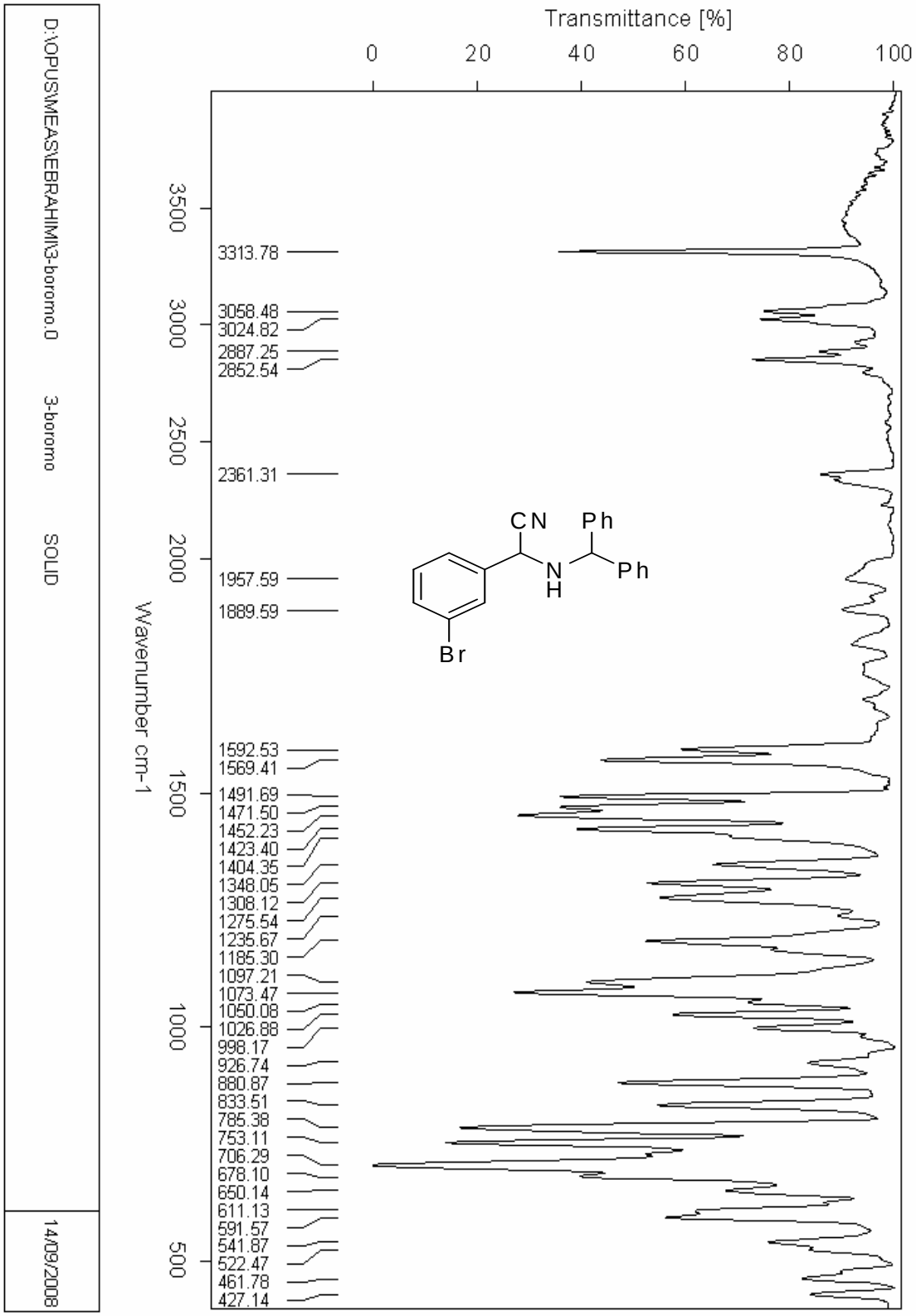
uv[1] Results

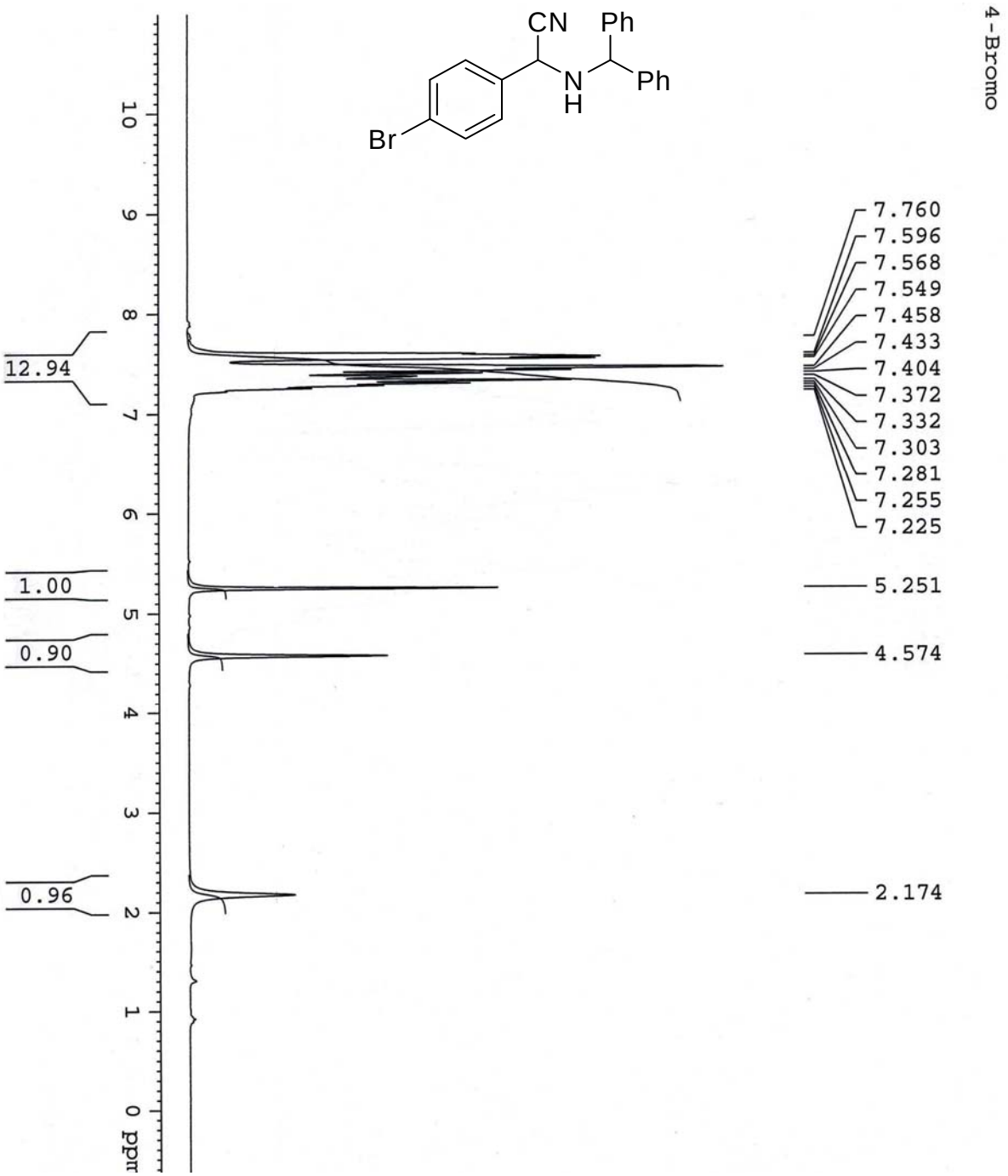
Retention Time	Area	Area %	Height	Height %
10.400	1208186	50.27	58081	54.90
11.967	1195071	49.73	47718	45.10
Totals	2403257	100.00	105799	100.00

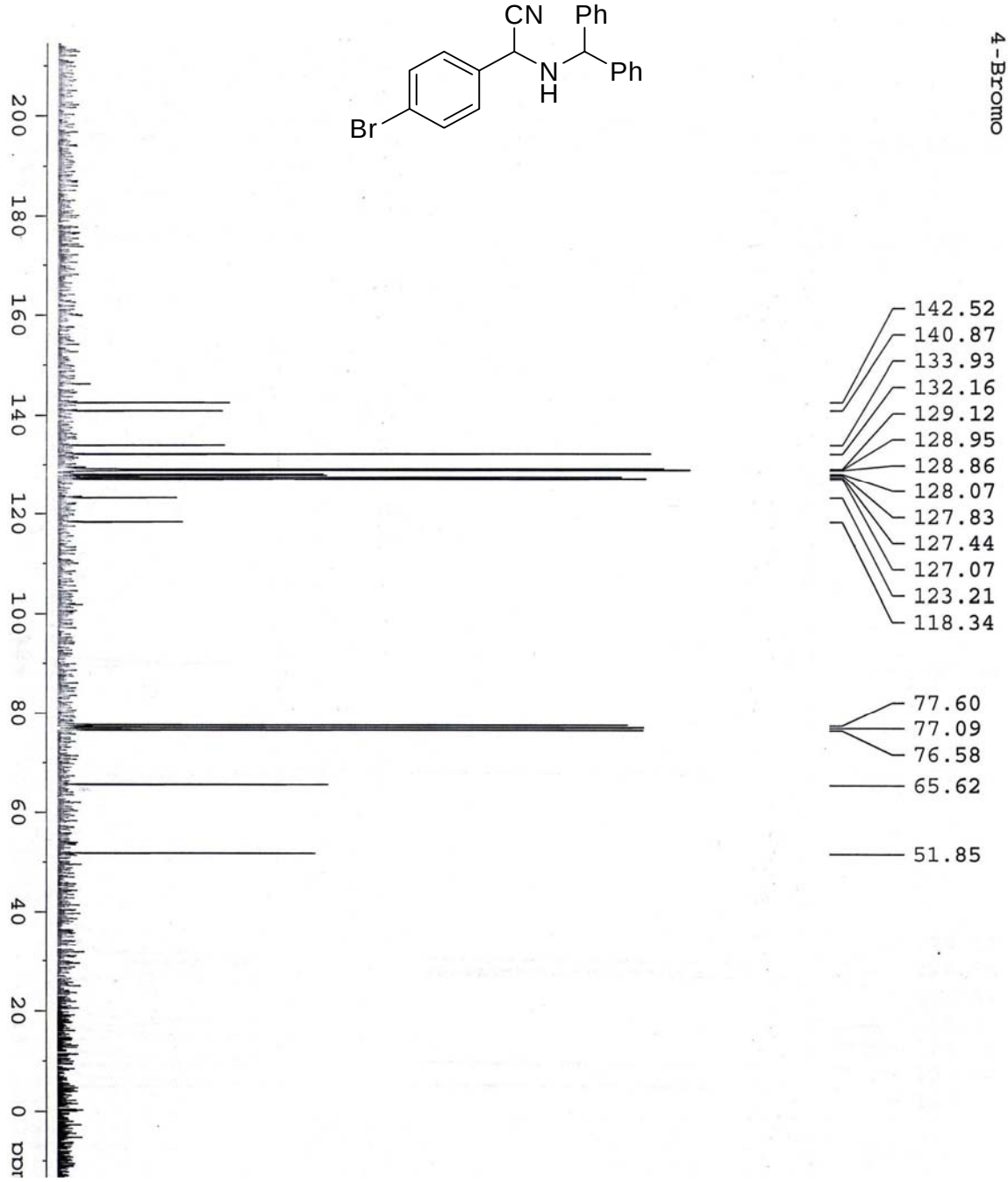


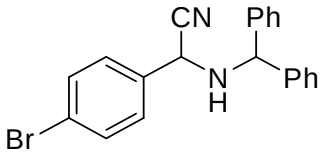
uv[1] Results

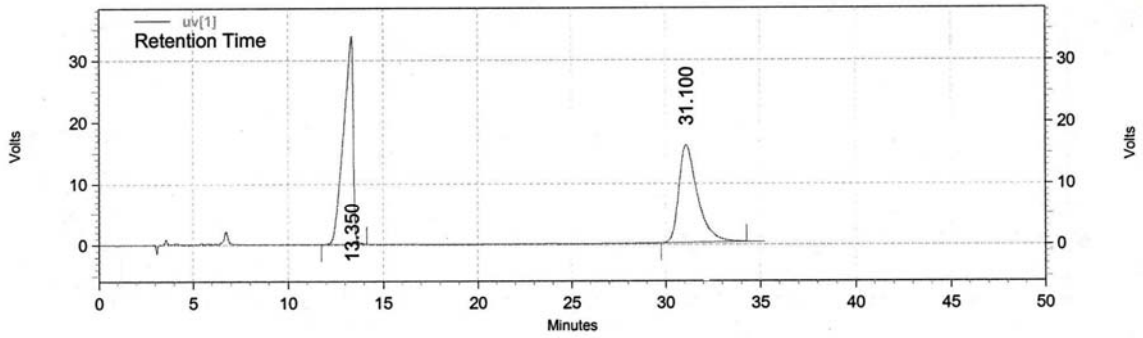
Retention Time	Area	Area %	Height	Height %
10.117	1281213	95.68	62311	96.47
11.817	57886	4.32	2278	3.53
Totals	1339099	100.00	64589	100.00


Transmittance [%]
0
20
40
60
80
100
Wavenumber cm-1
3500
3000
2500
2000
1500
1000
500
3313.78
3058.48
3024.82
2887.25
2852.54
2361.31
1957.59
1889.59
1592.53
1569.41
1491.69
1471.50
1452.23
1423.40
1404.35
1348.05
1308.12
1275.54
1235.67
1185.30
1097.21
1073.47
1050.08
1026.88
998.17
926.74
880.87
833.51
785.38
753.11
706.29
678.10
650.14
611.13
591.57
541.87
522.47
461.78
427.14
CN
Ph
N
H
Ph
Br
D:\OPUS\MEAS\EBRAHIMI\3-boromo.0
3-boromo
SOLID
14/09/2008


CN
Ph
N
H
Ph
Br
4-Bromo
7.760
7.596
7.568
7.549
7.458
7.433
7.404
7.372
7.332
7.303
7.281
7.255
7.225
5.251
4.574
2.174
12.94
1.00
0.90
0.96
10
9
8
7
6
5
4
3
2
1
0
ppm

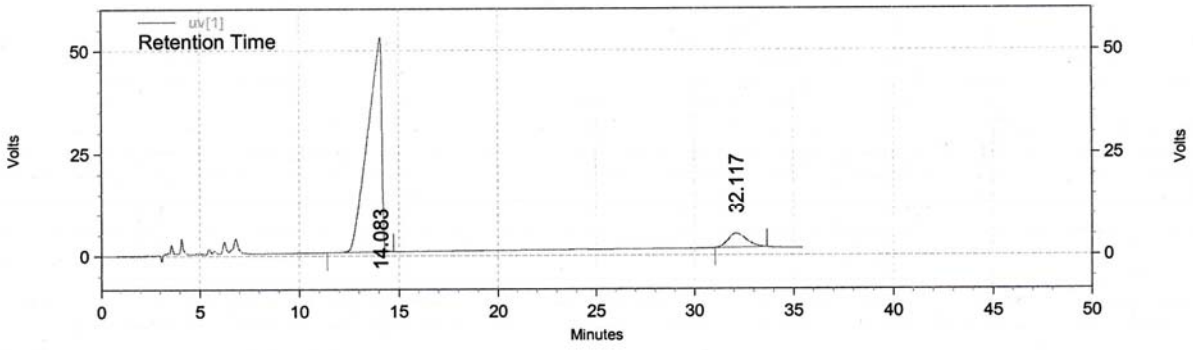

CN
Ph
N
H
Ph
Br
4-Bromo
142.52
140.87
133.93
132.16
129.12
128.95
128.86
128.07
127.83
127.44
127.07
123.21
118.34
77.60
77.09
76.58
65.62
51.85
200
180
160
140
120
100
80
60
40
20
0
PPM





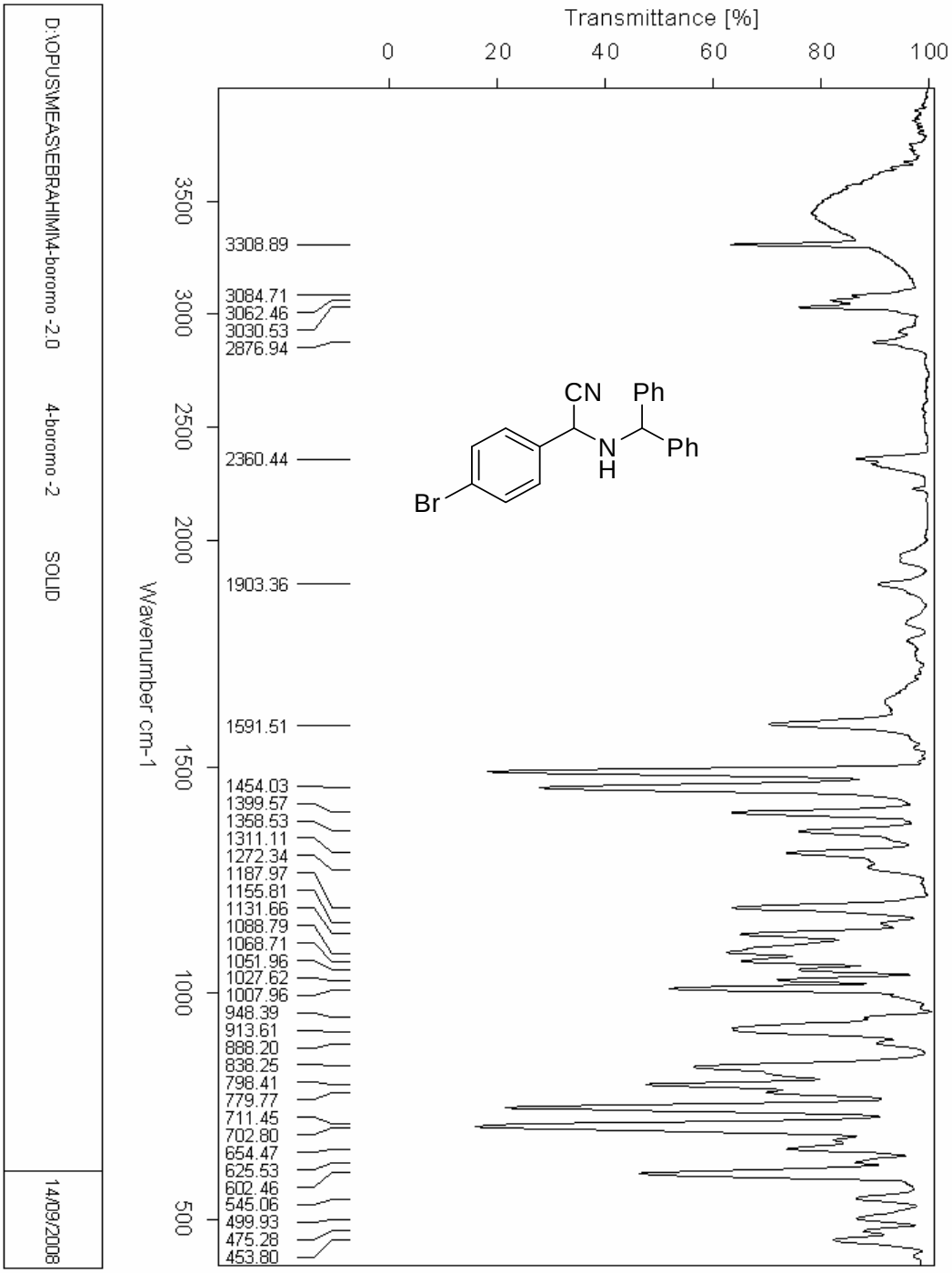
uv[1] Results

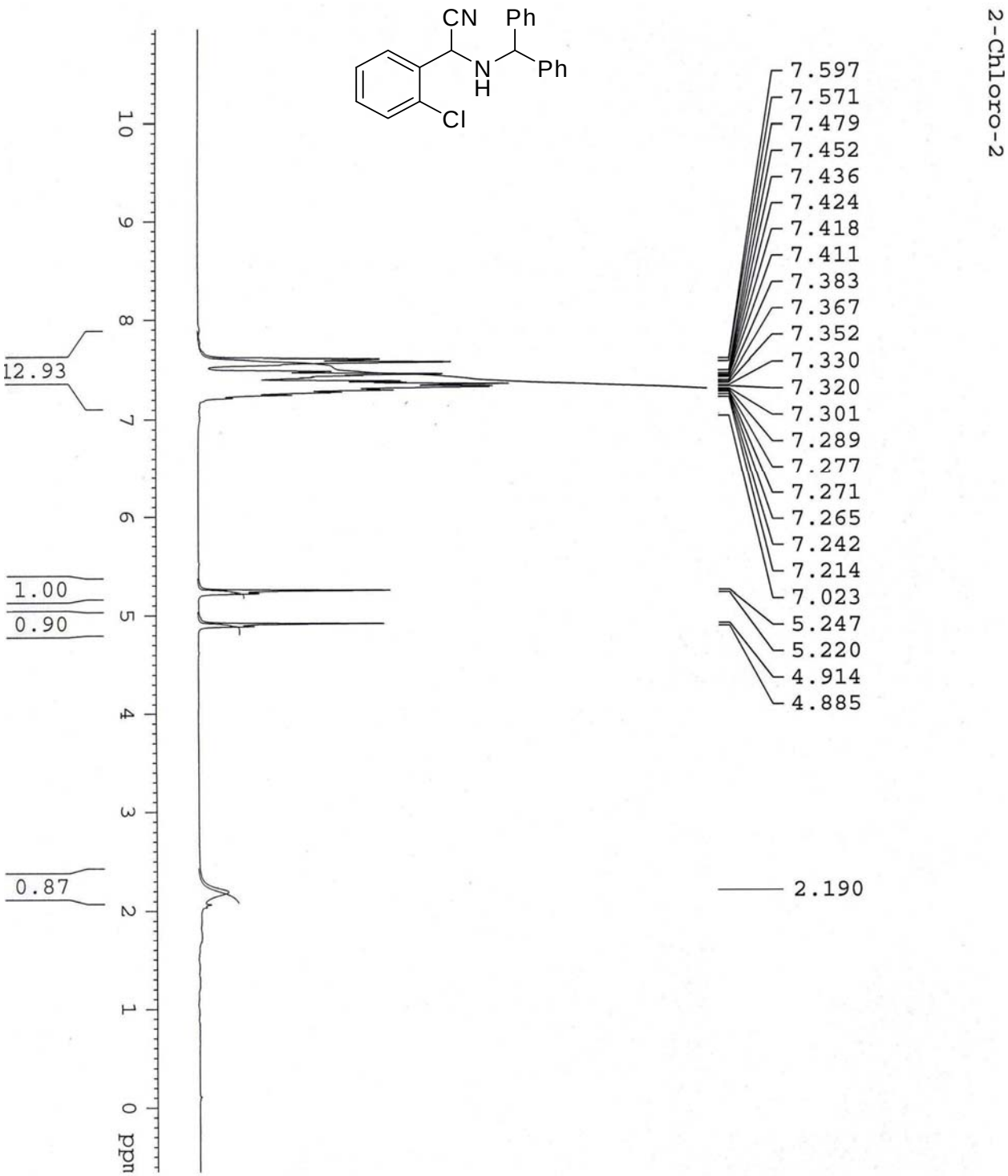
Retention Time	Area	Area %	Height	Height %
13.350	1198028	52.48	33836	67.99
31.100	1084626	47.52	15931	32.01
Totals	2282654	100.00	49767	100.00

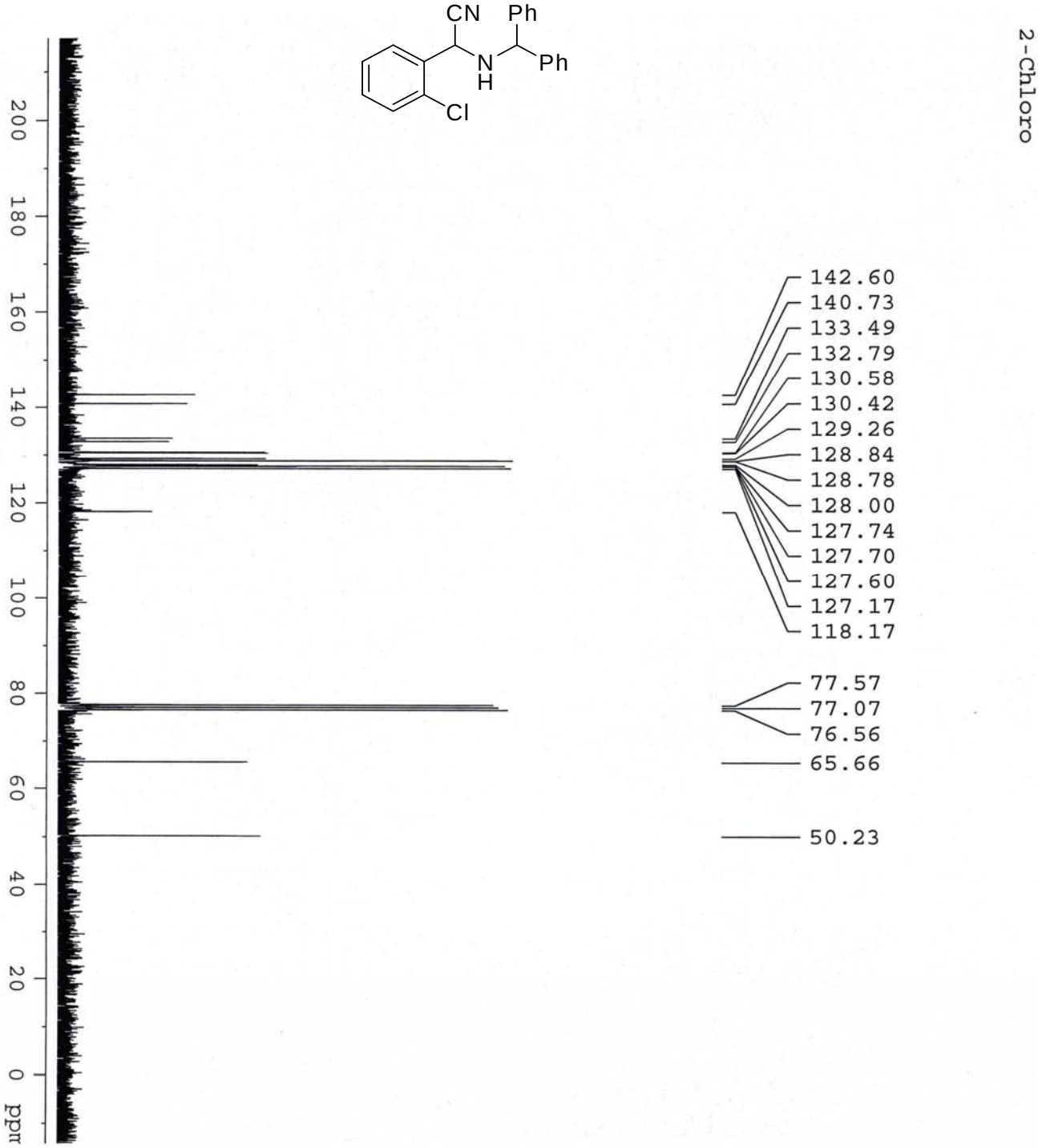


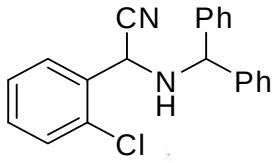
uv[1] Results

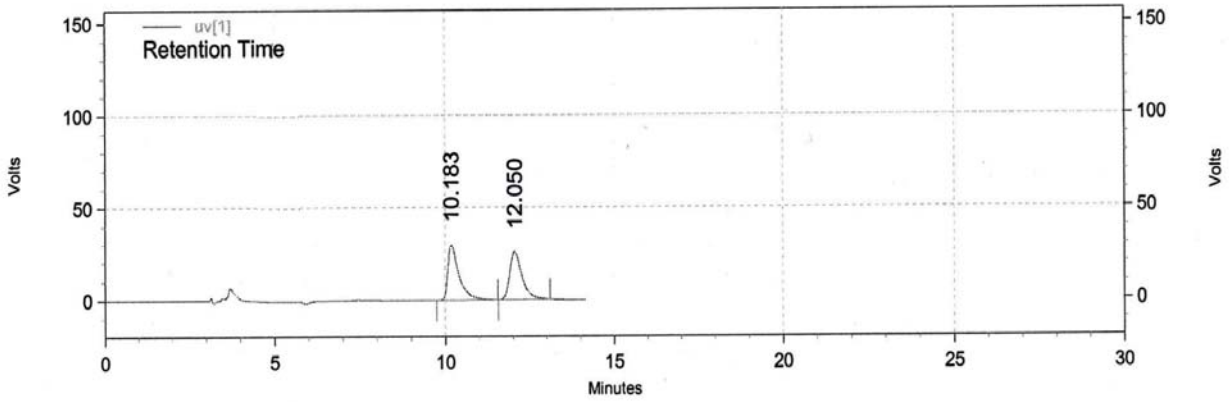
Retention Time	Area	Area %	Height	Height %
14.083	2561967	91.98	52302	93.92
32.117	223466	8.02	3387	6.08
Totals	2785433	100.00	55689	100.00


D:\OPUS\MEAS\EBRAHIM\4-boromo -2.0
4-boromo -2
SOLID
14/09/2008
Transmittance [%]
0
20
40
60
80
100
Wavenumber cm-1
3500
3000
2500
2000
1500
1000
500
3308.89
3084.71
3062.46
3030.53
2876.94
2360.44
1903.36
1591.51
1454.03
1399.57
1358.53
1311.11
1272.34
1187.97
1155.81
1131.66
1088.79
1068.71
1051.96
1027.62
1007.96
948.39
913.61
888.20
838.25
798.41
779.77
711.45
702.80
654.47
625.53
602.46
545.06
499.93
475.28
453.80
CN
Ph
N
H
Ph
Br


2-Chloro-2
CN
Ph
N
H
Ph
Cl
7.597
7.571
7.479
7.452
7.436
7.424
7.418
7.411
7.383
7.367
7.352
7.330
7.320
7.301
7.289
7.277
7.271
7.265
7.242
7.214
7.023
5.247
5.220
4.914
4.885
2.190
12.93
1.00
0.90
0.87
10
9
8
7
6
5
4
3
2
1
0
ppm

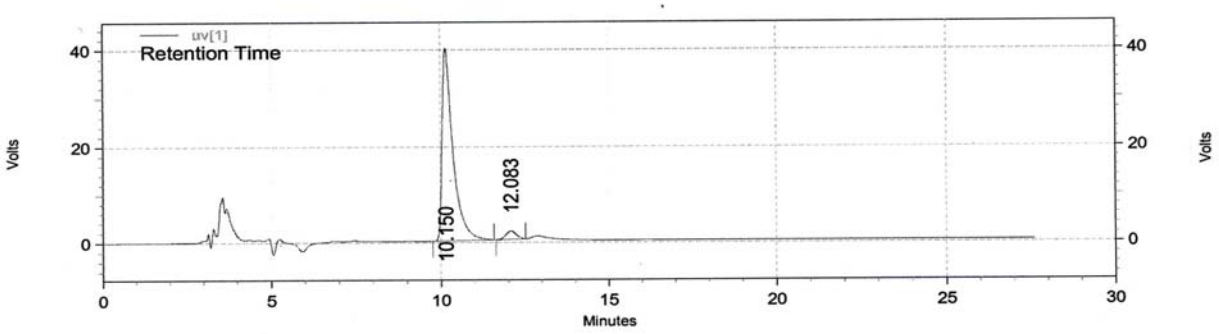

CN
Ph
N
H
Ph
Cl
2-Chloro
142.60
140.73
133.49
132.79
130.58
130.42
129.26
128.84
128.78
128.00
127.74
127.70
127.60
127.17
118.17
77.57
77.07
76.56
65.66
50.23
200
180
160
140
120
100
80
60
40
20
0
ppm





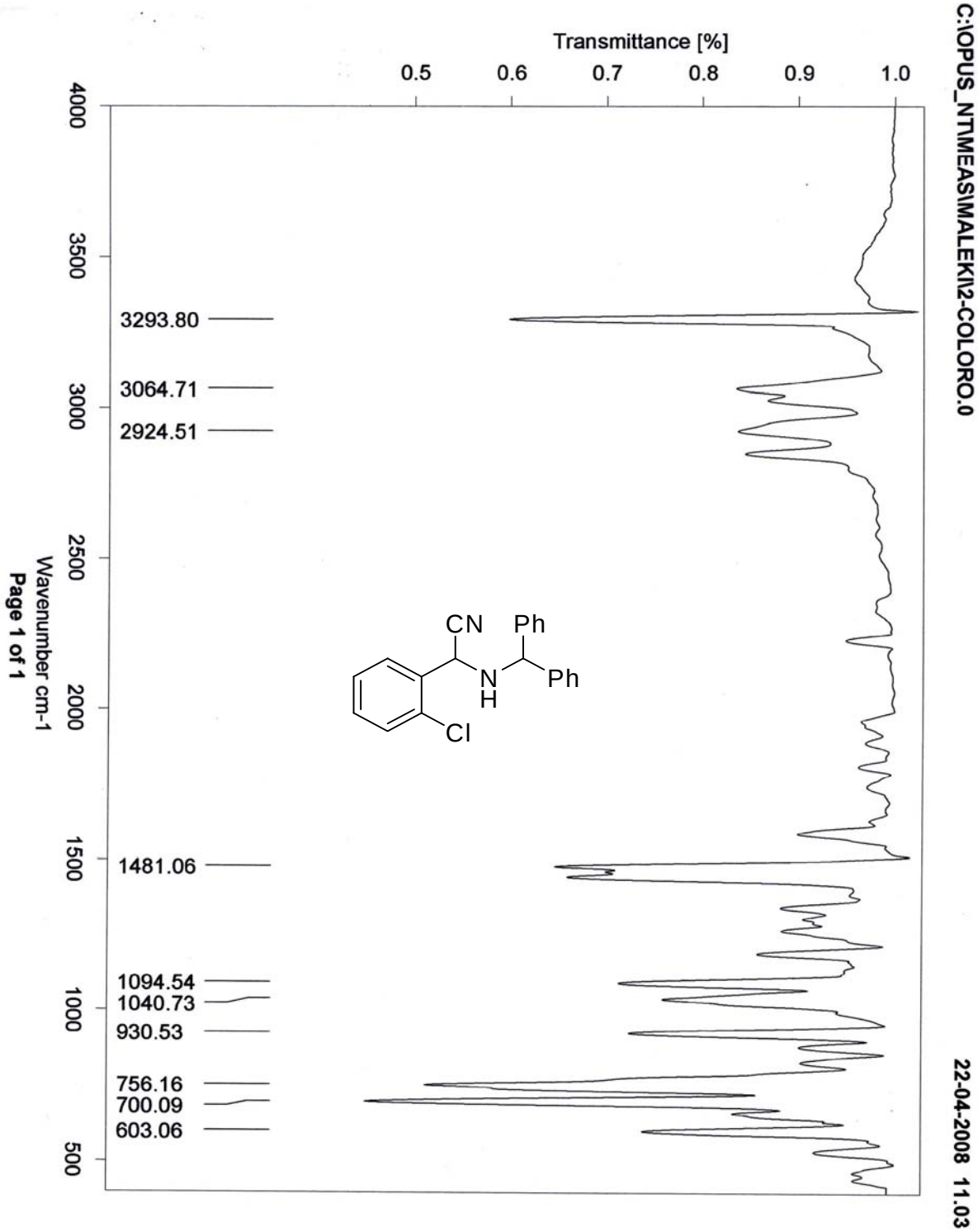
uv[1] Results

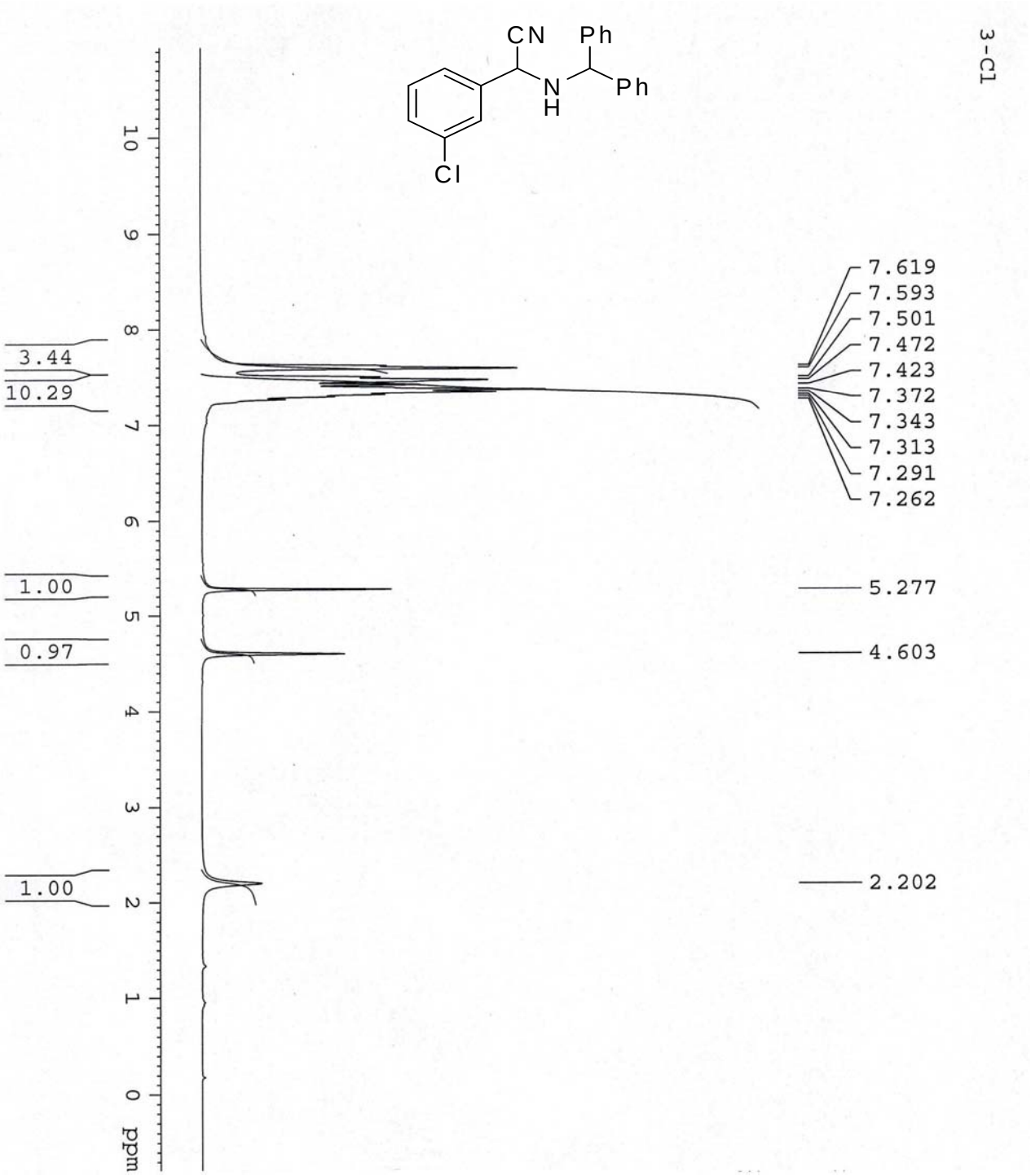
Retention Time	Area	Area %	Height	Height %
10.183	666025	50.93	29822	53.78
12.050	641590	49.07	25634	46.22
Totals	1307615	100.00	55456	100.00

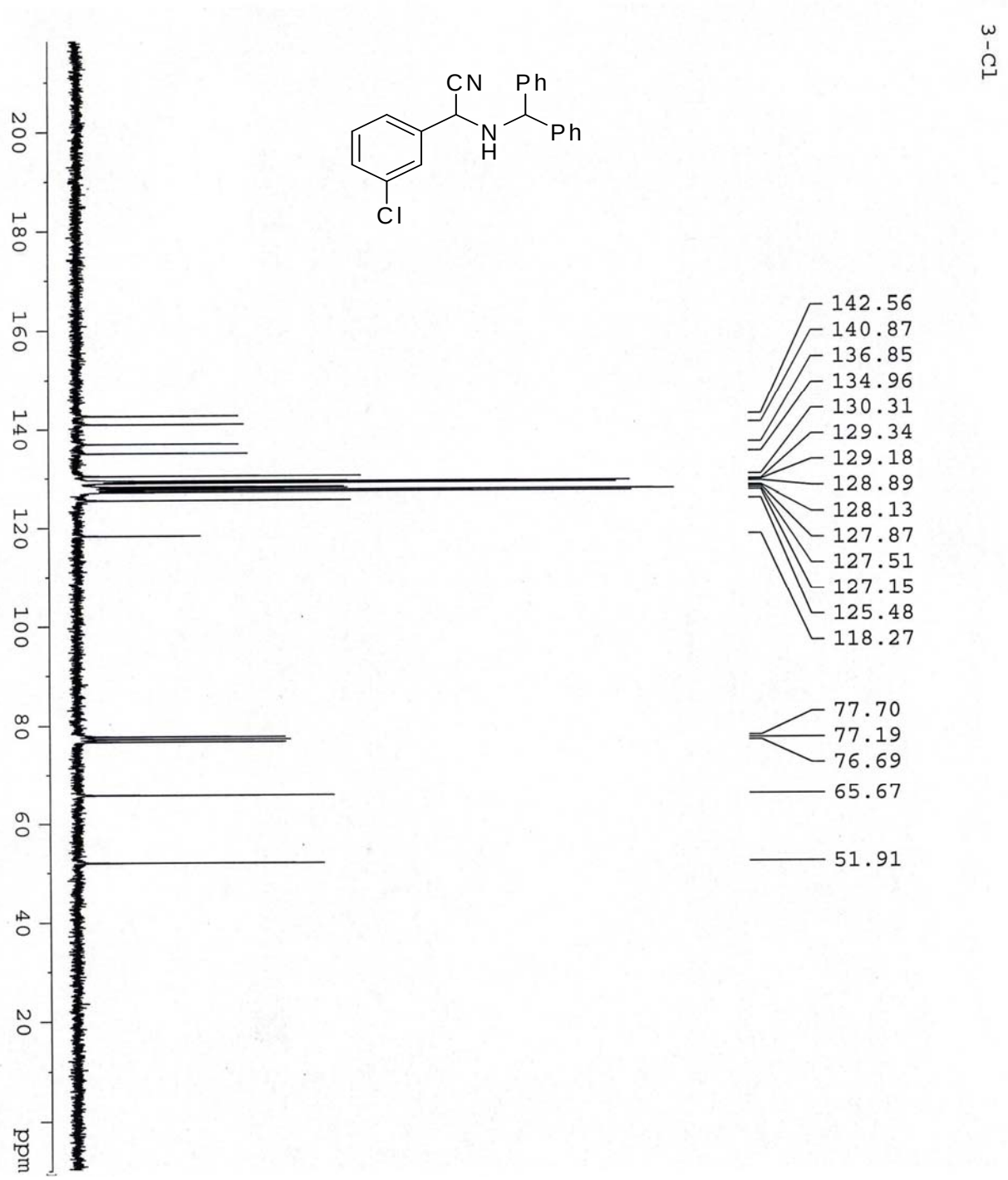


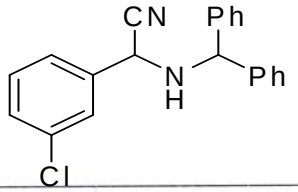
uv[1] Results

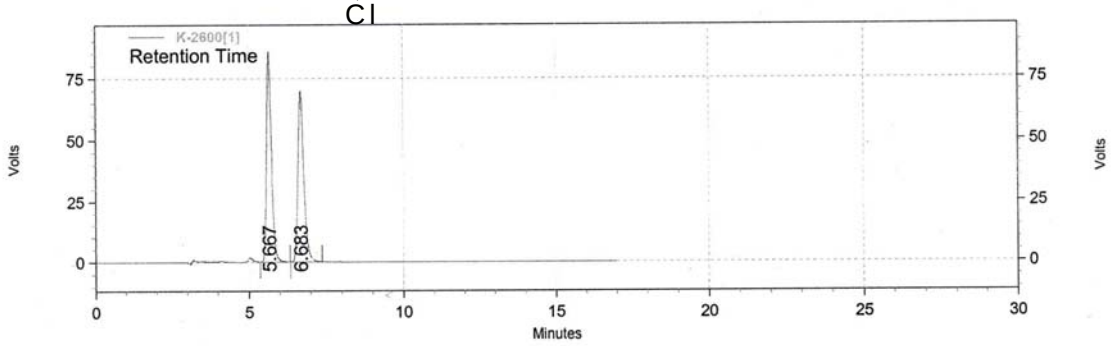
Retention Time	Area	Area %	Height	Height %
10.150	893380	96.08	39925	95.94
12.083	36446	3.92	1689	4.06
Totals	929826	100.00	41614	100.00


Transmittance [%]
0.5
0.6
0.7
0.8
0.9
1.0
4000
3500
3000
2500
2000
1500
1000
500
Wavenumber cm-1
3293.80
3064.71
2924.51
1481.06
1094.54
1040.73
930.53
756.16
700.09
603.06
CN
Ph
N
H
Ph
Cl
C:\OPUS_NT\MEAS\MALEK\I2-COLORO.0
22-04-2008 11.03
Page 1 of 1


CN
Ph
N
H
Ph
Cl
3-Cl
7.619
7.593
7.501
7.472
7.423
7.372
7.343
7.313
7.291
7.262
5.277
4.603
2.202
3.44
10.29
1.00
0.97
1.00
ppm

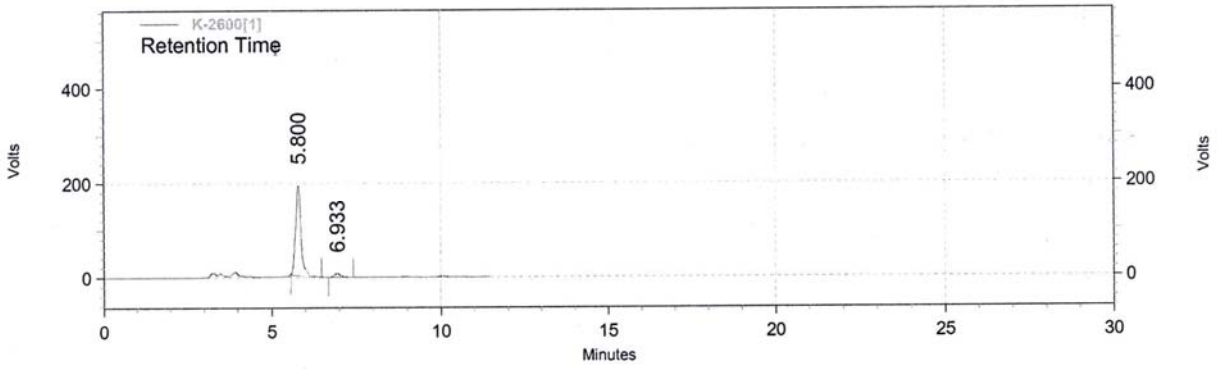

3-Cl
CN
Ph
N
H
Ph
Cl
200
180
160
140
120
100
80
60
40
20
ppm
142.56
140.87
136.85
134.96
130.31
129.34
129.18
128.89
128.13
127.87
127.51
127.15
125.48
118.27
77.70
77.19
76.69
65.67
51.91





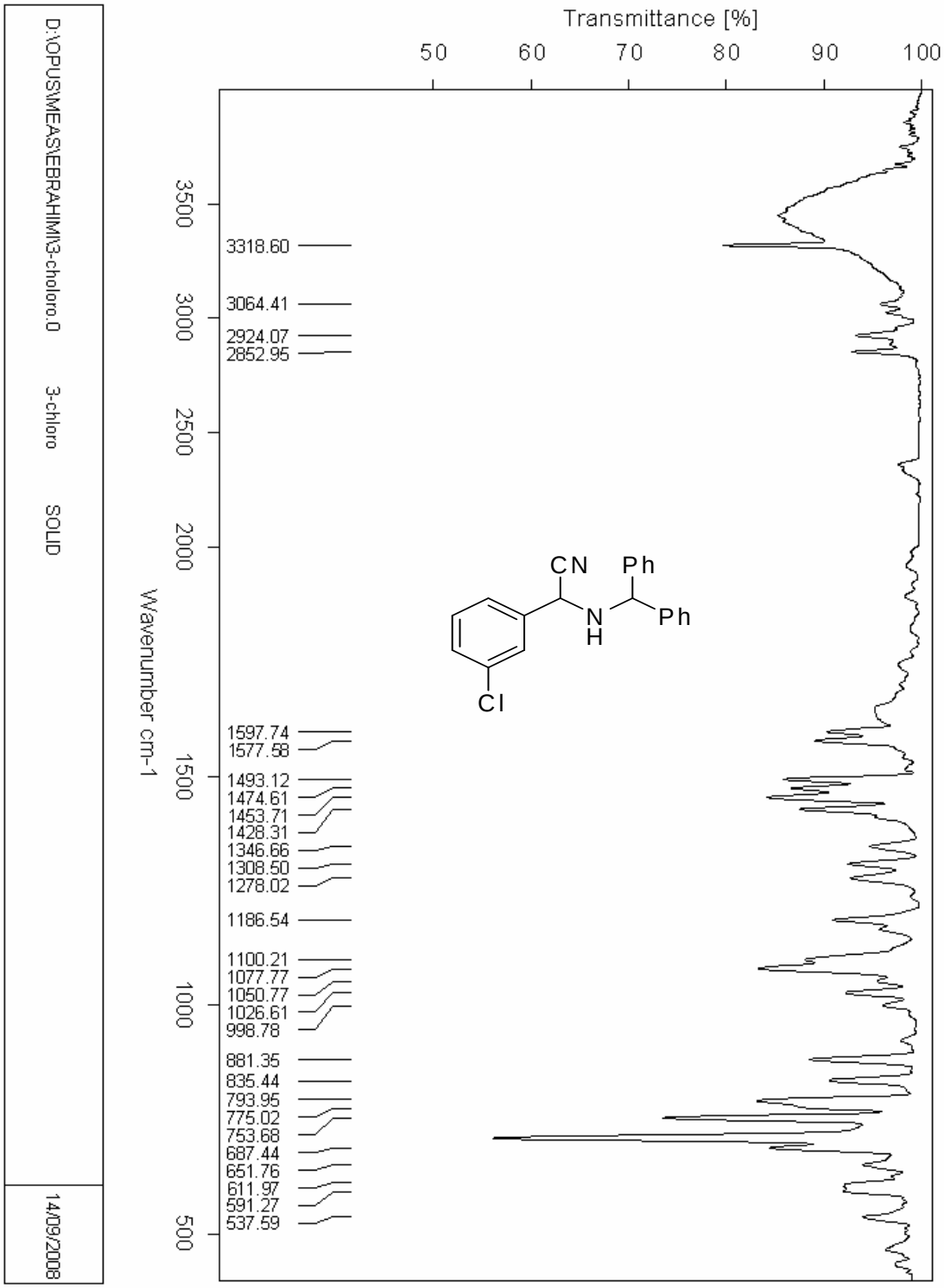
K-2600[1] Results

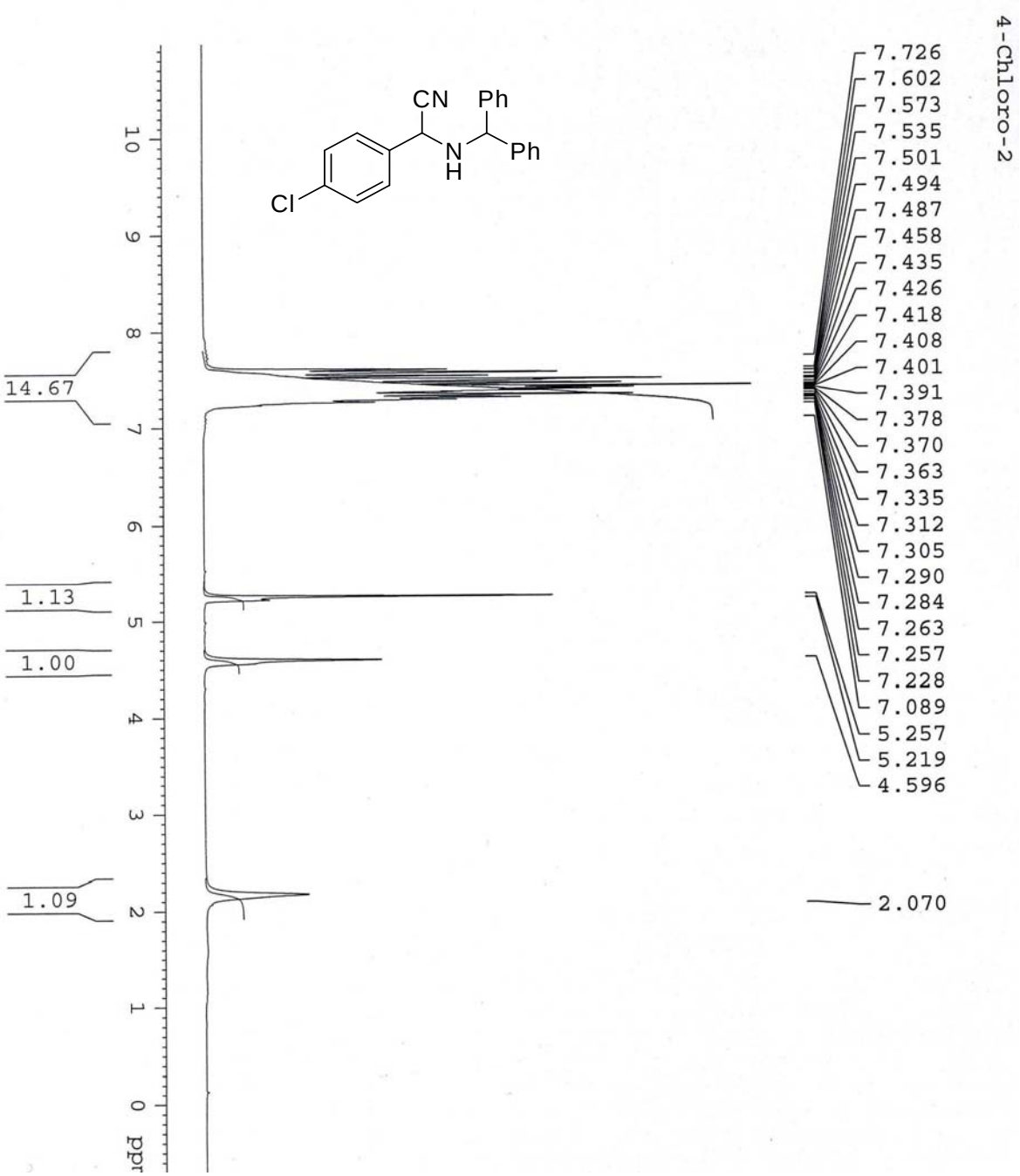
Retention Time	Area	Area %	Height	Height %
5.667	940203	50.53	85613	55.08
6.683	920319	49.47	69810	44.92
Totals	1860522	100.00	155423	100.00

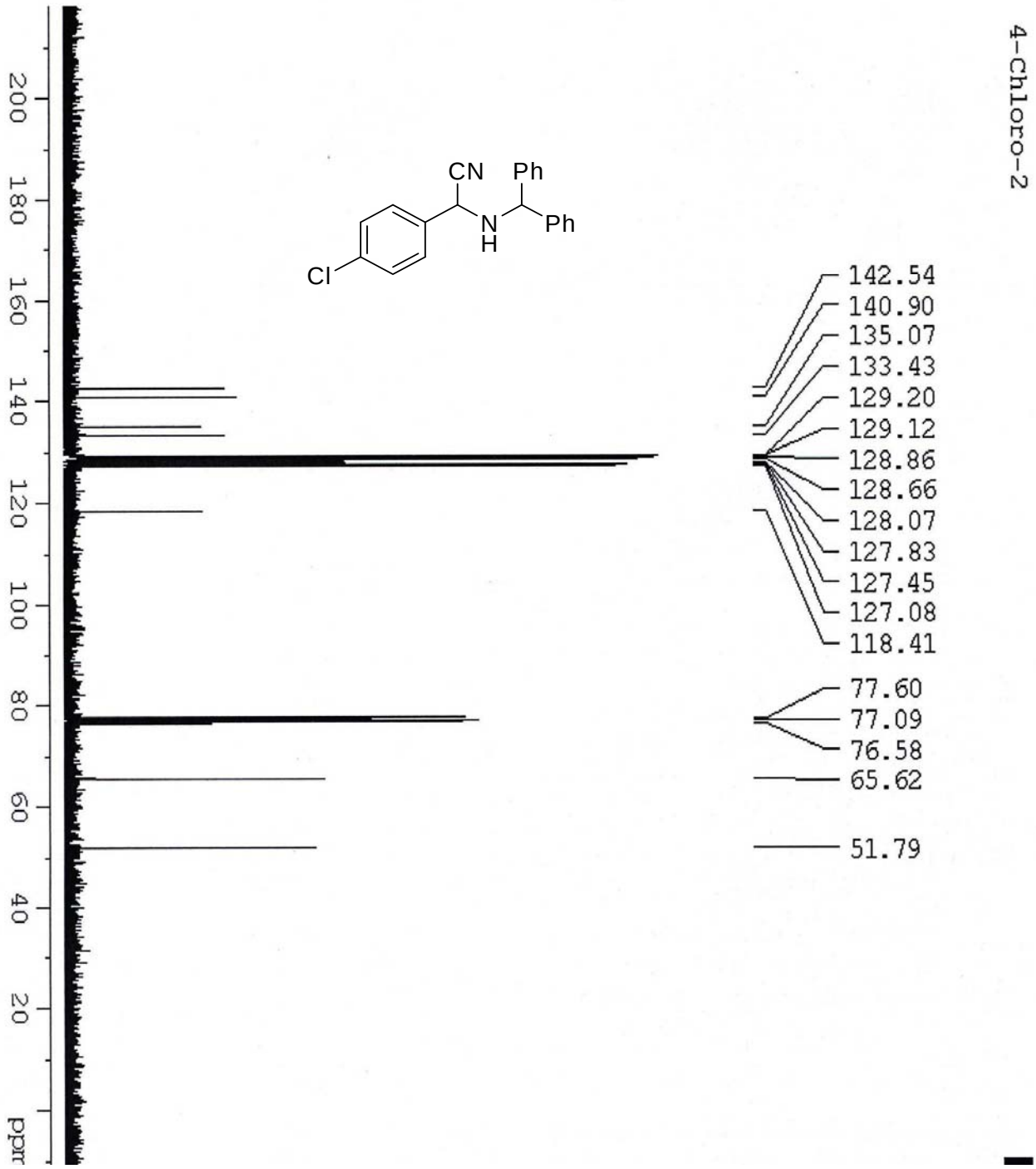


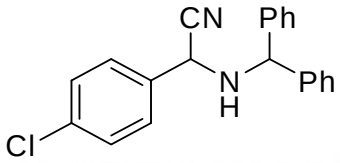
K-2600[1] Results

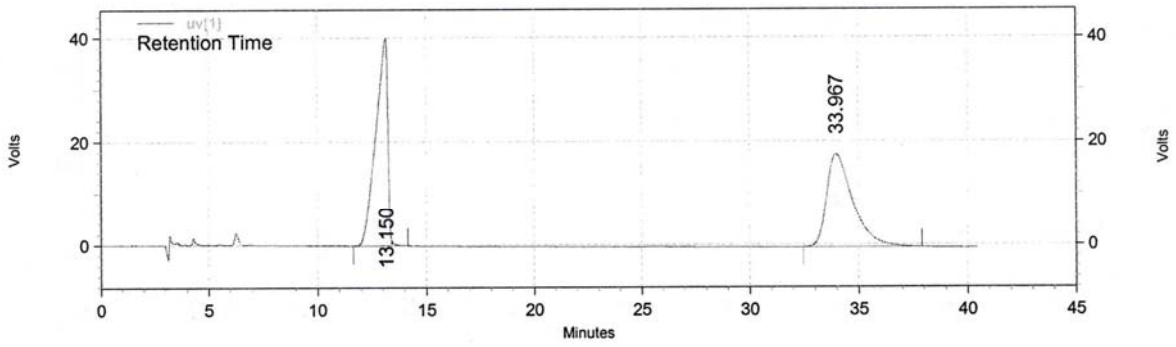
Retention Time	Area	Area %	Height	Height %
5.800	2310694	95.69	190599	96.12
6.933	104084	4.31	7703	3.88
Totals	2414778	100.00	198302	100.00


D:\OPUS\MEAS\EBRAHIM\3-choloro.0
3-chloro
SOLID
14/09/2008
Transmittance [%]
50
60
70
80
90
100
Wavenumber cm-1
3500
3000
2500
2000
1500
1000
500
3318.60
3064.41
2924.07
2852.95
1597.74
1577.58
1493.12
1474.61
1453.71
1428.31
1346.66
1308.50
1278.02
1186.54
1100.21
1077.77
1050.77
1026.61
998.78
881.35
835.44
793.95
775.02
753.68
687.44
651.76
611.97
591.27
537.59
CN
Ph
N
H
Ph
Cl


4-Chloro-2
7.726
7.602
7.573
7.535
7.501
7.494
7.487
7.458
7.435
7.426
7.418
7.408
7.401
7.391
7.378
7.370
7.363
7.335
7.312
7.305
7.290
7.284
7.263
7.257
7.228
7.089
5.257
5.219
4.596
2.070
CN
Ph
N
H
Ph
Cl
14.67
1.13
1.00
1.09
10
9
8
7
6
5
4
3
2
1
0
ppm

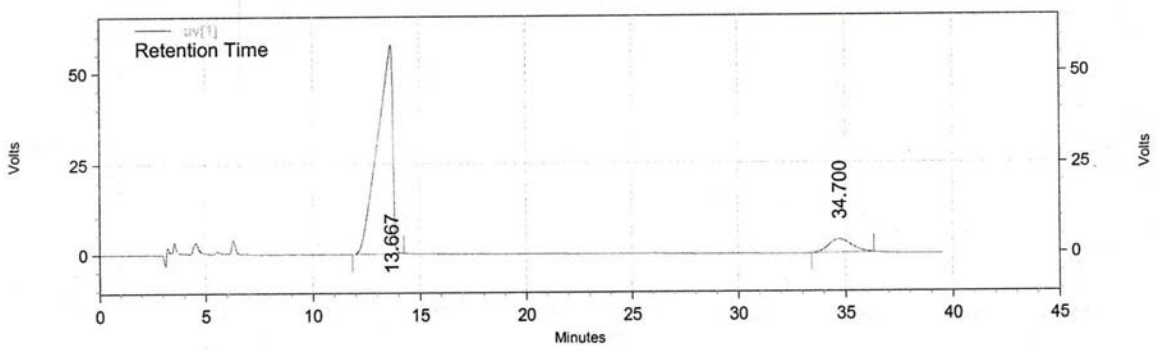

4-Chloro-2
CN
Ph
N
H
Ph
Cl
142.54
140.90
135.07
133.43
129.20
129.12
128.86
128.66
128.07
127.83
127.45
127.08
118.41
77.60
77.09
76.58
65.62
51.79
200
180
160
140
120
100
80
60
40
20
ppm





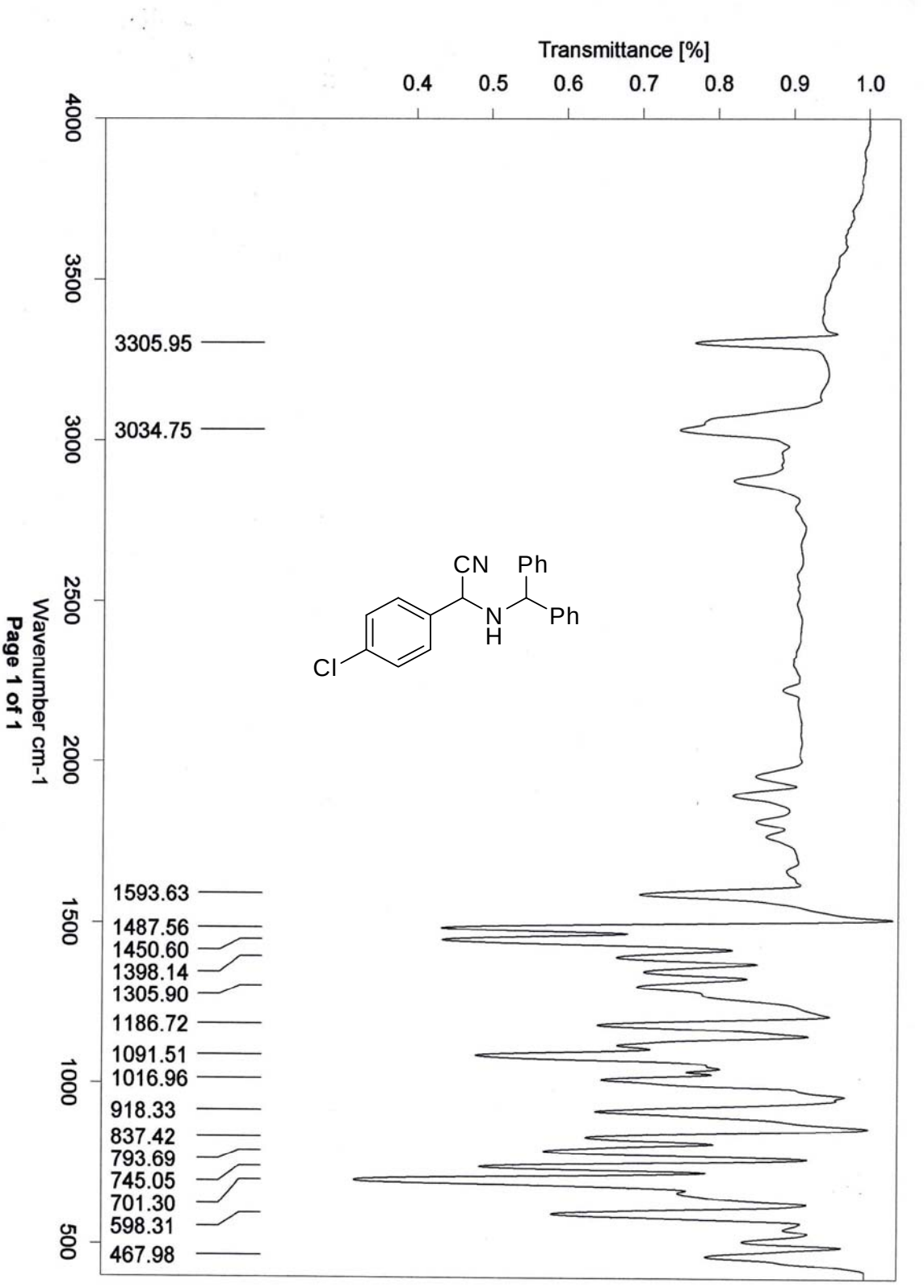
uv[1] Results

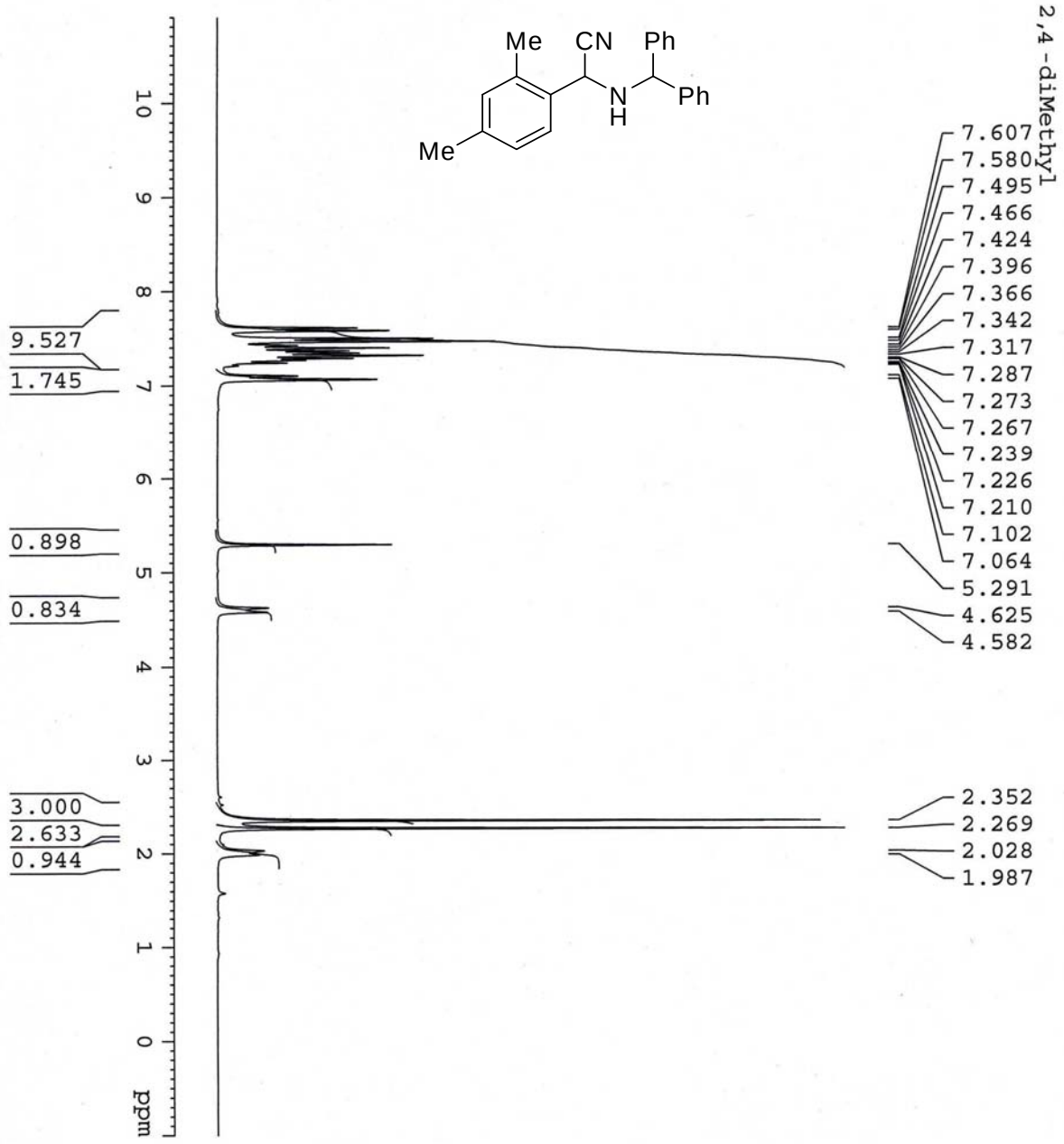
Retention Time	Area	Area %	Height	Height %
13.150	1538758	50.72	40120	69.03
33.967	1494797	49.28	17999	30.97
Totals	3033555	100.00	58119	100.00

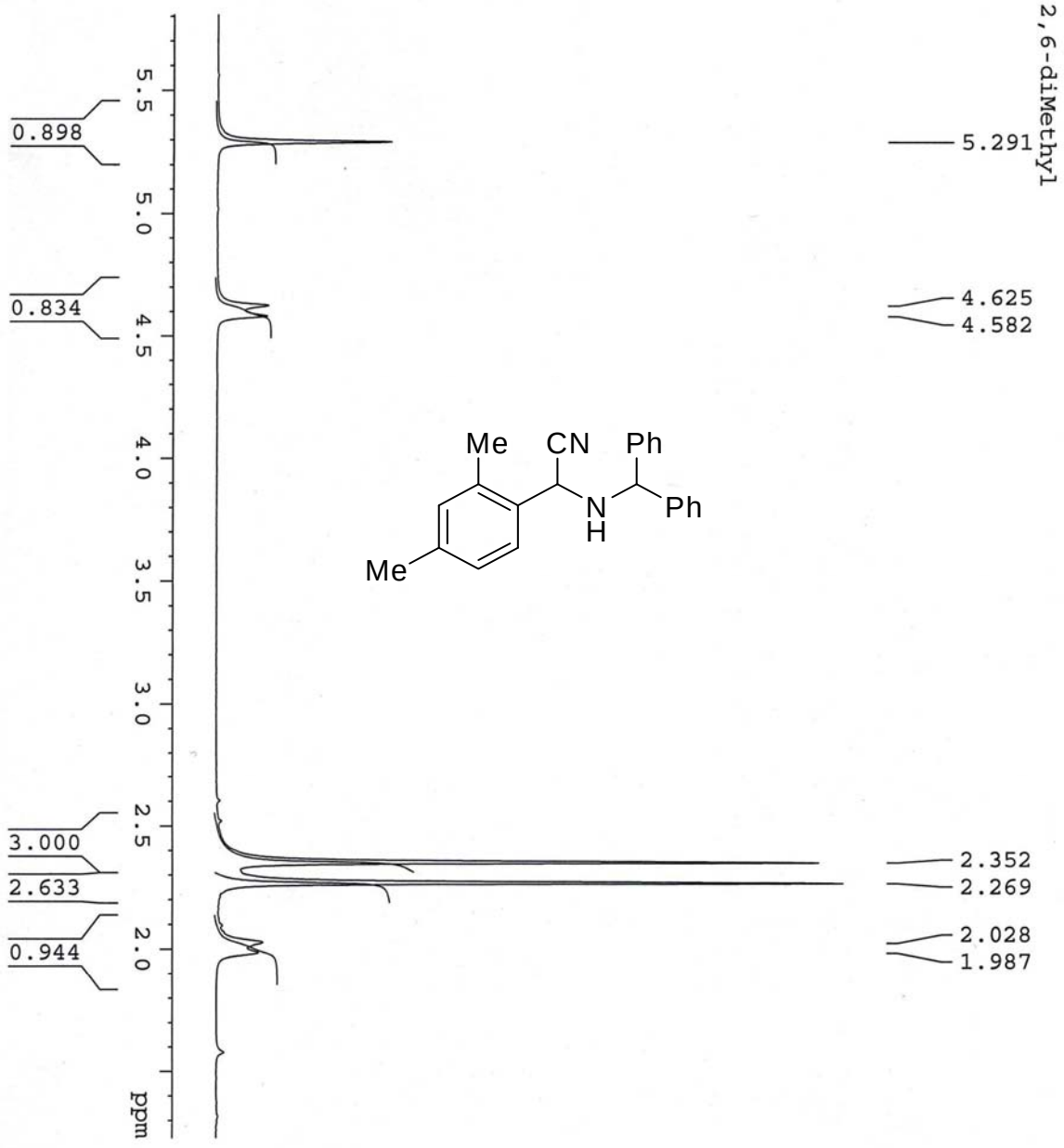


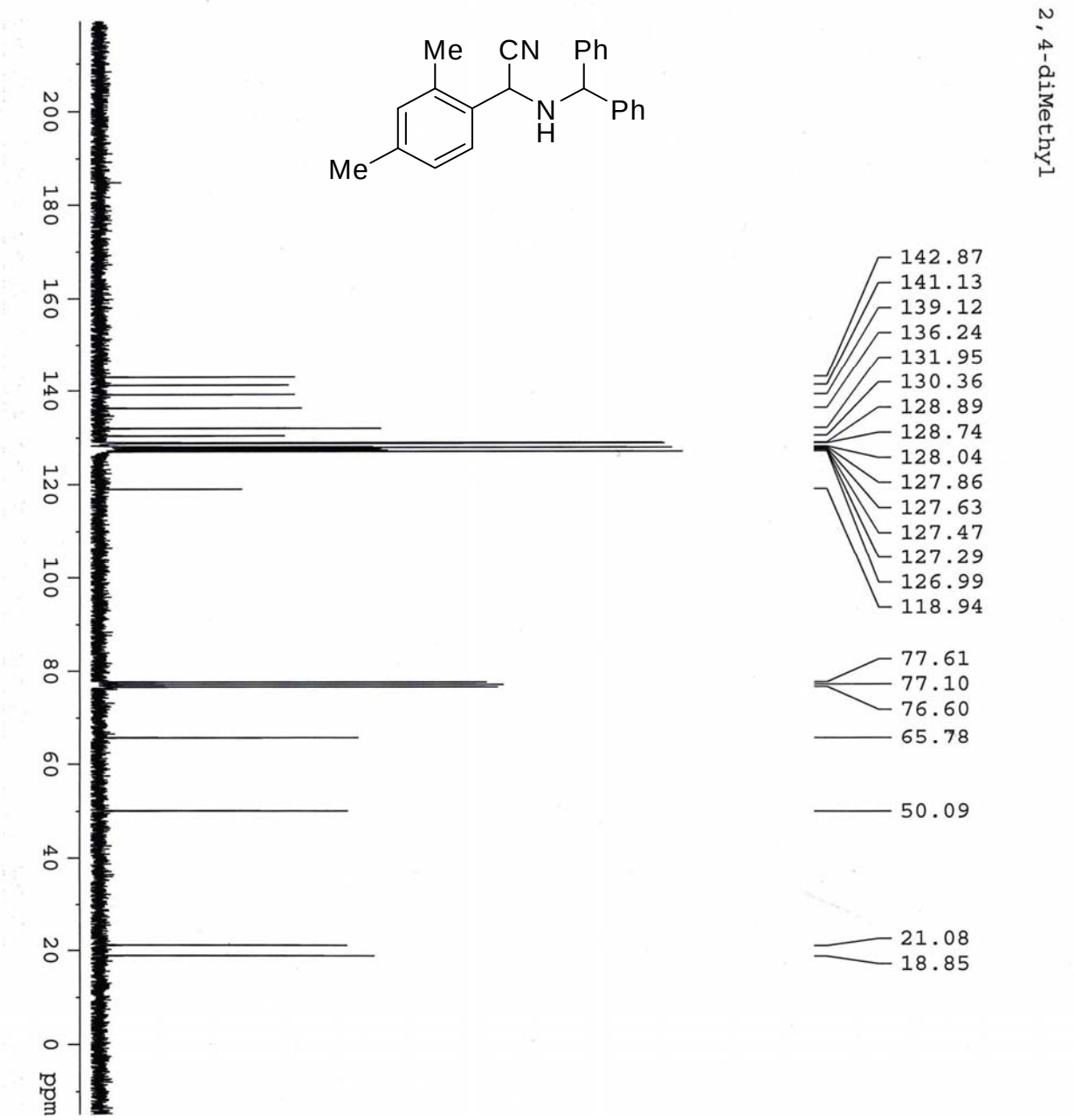
uv[1] Results

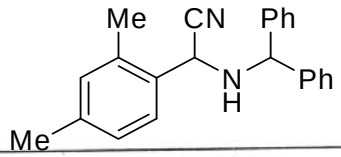
Retention Time	Area	Area %	Height	Height %
13.667	2815236	91.25	57620	94.05
34.700	270056	8.75	3644	5.95
Totals	3085292	100.00	61264	100.00

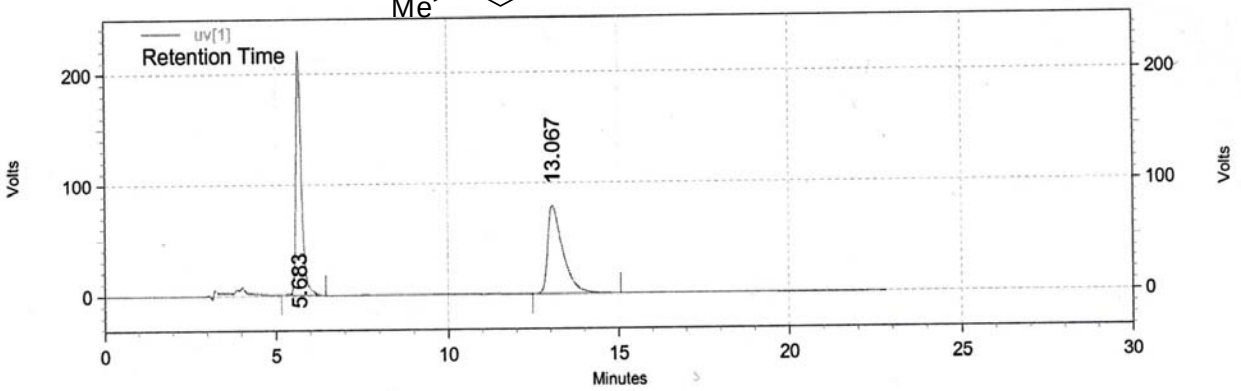

Transmittance [%]
0.4
0.5
0.6
0.7
0.8
0.9
1.0
4000
3500
3000
2500
2000
1500
1000
500
Wavenumber cm-1
Page 1 of 1
3305.95
3034.75
1593.63
1487.56
1450.60
1398.14
1305.90
1186.72
1091.51
1016.96
918.33
837.42
793.69
745.05
701.30
598.31
467.98
CN
Ph
N
H
Ph
Cl


Me
CN
Ph
N
H
Ph
Me
2,4-diMethyl
7.607
7.580
7.495
7.466
7.424
7.396
7.366
7.342
7.317
7.287
7.273
7.267
7.239
7.226
7.210
7.102
7.064
5.291
4.625
4.582
2.352
2.269
2.028
1.987
9.527
1.745
0.898
0.834
3.000
2.633
0.944
ppm


2,6-diMethyl
5.291
4.625
4.582
2.352
2.269
2.028
1.987
0.898
0.834
3.000
2.633
0.944
Me
CN
Ph
N
H
Ph
Me
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
ppm

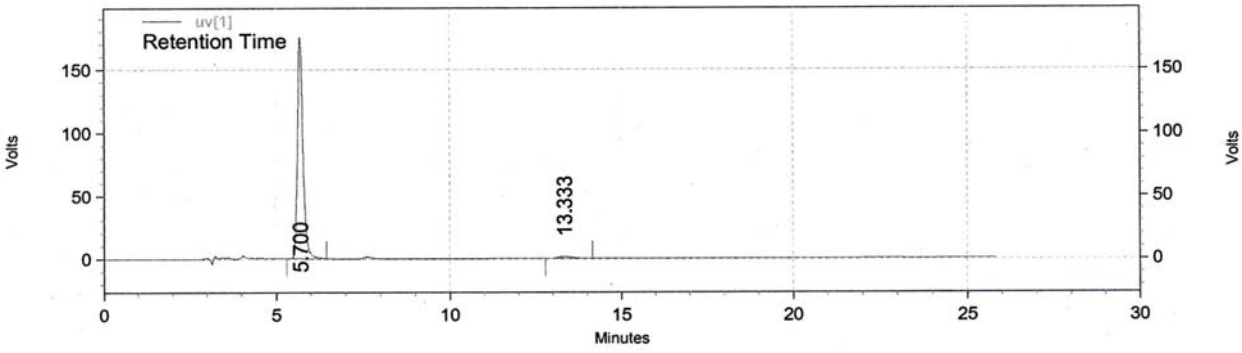

Me
CN
Ph
N
H
Ph
Me
2,4-diMethyl
142.87
141.13
139.12
136.24
131.95
130.36
128.89
128.74
128.04
127.86
127.63
127.47
127.29
126.99
118.94
77.61
77.10
76.60
65.78
50.09
21.08
18.85
200
180
160
140
120
100
80
60
40
20
0
ppm





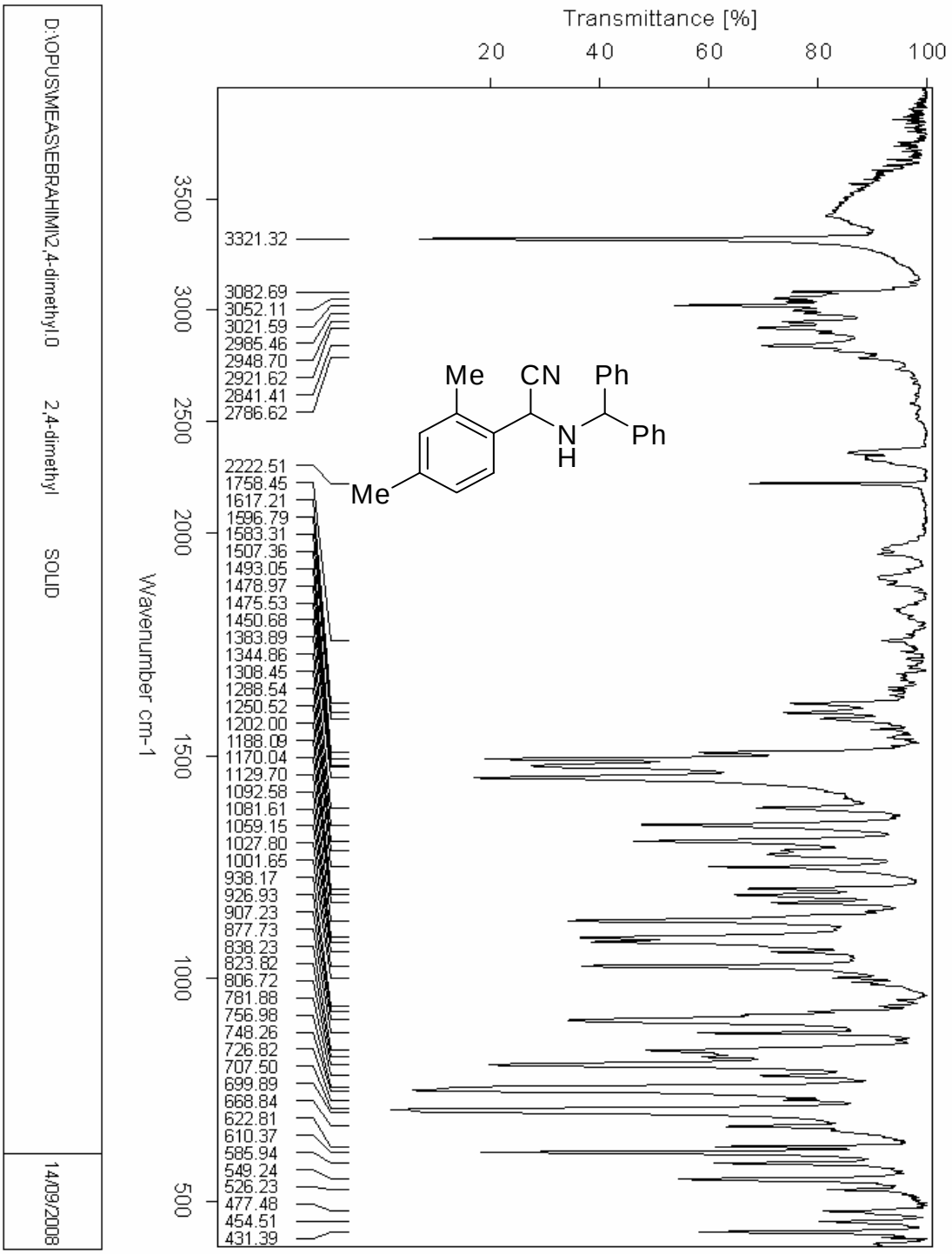
uv[1] Results

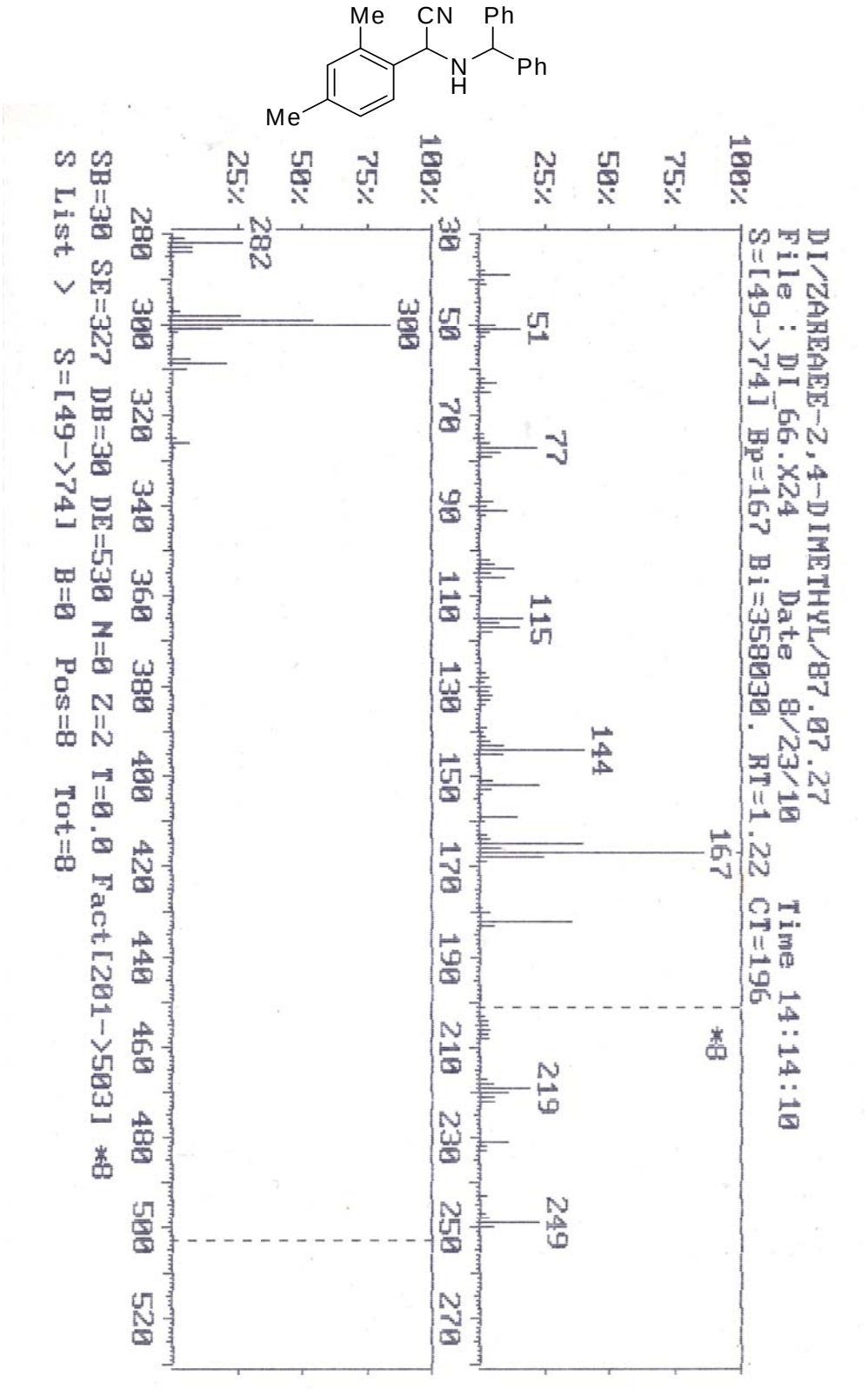
Retention Time	Area	Area %	Height	Height %
5.683	2404489	49.85	220226	73.47
13.067	2419097	50.15	79516	26.53
Totals	4823586	100.00	299742	100.00

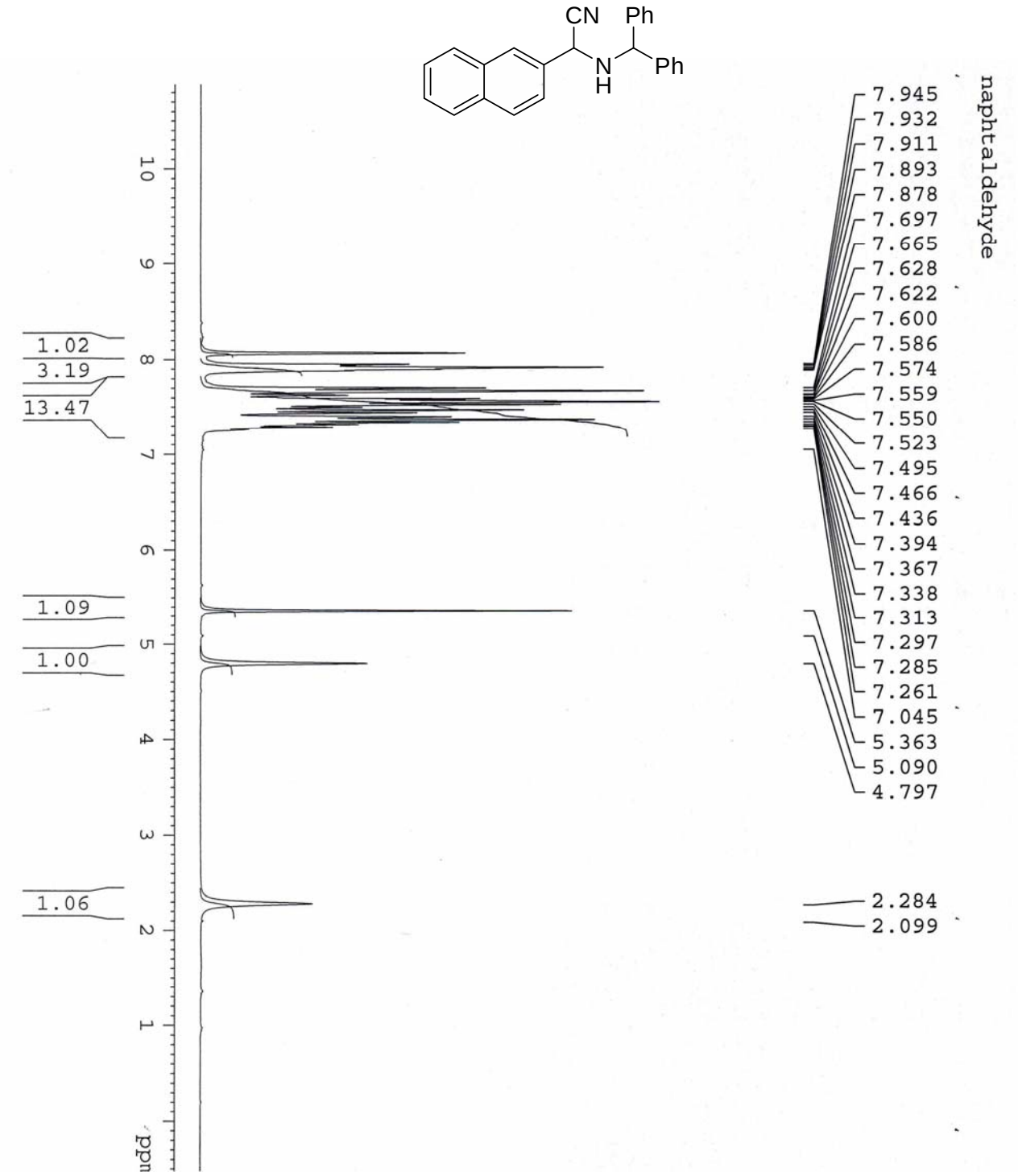


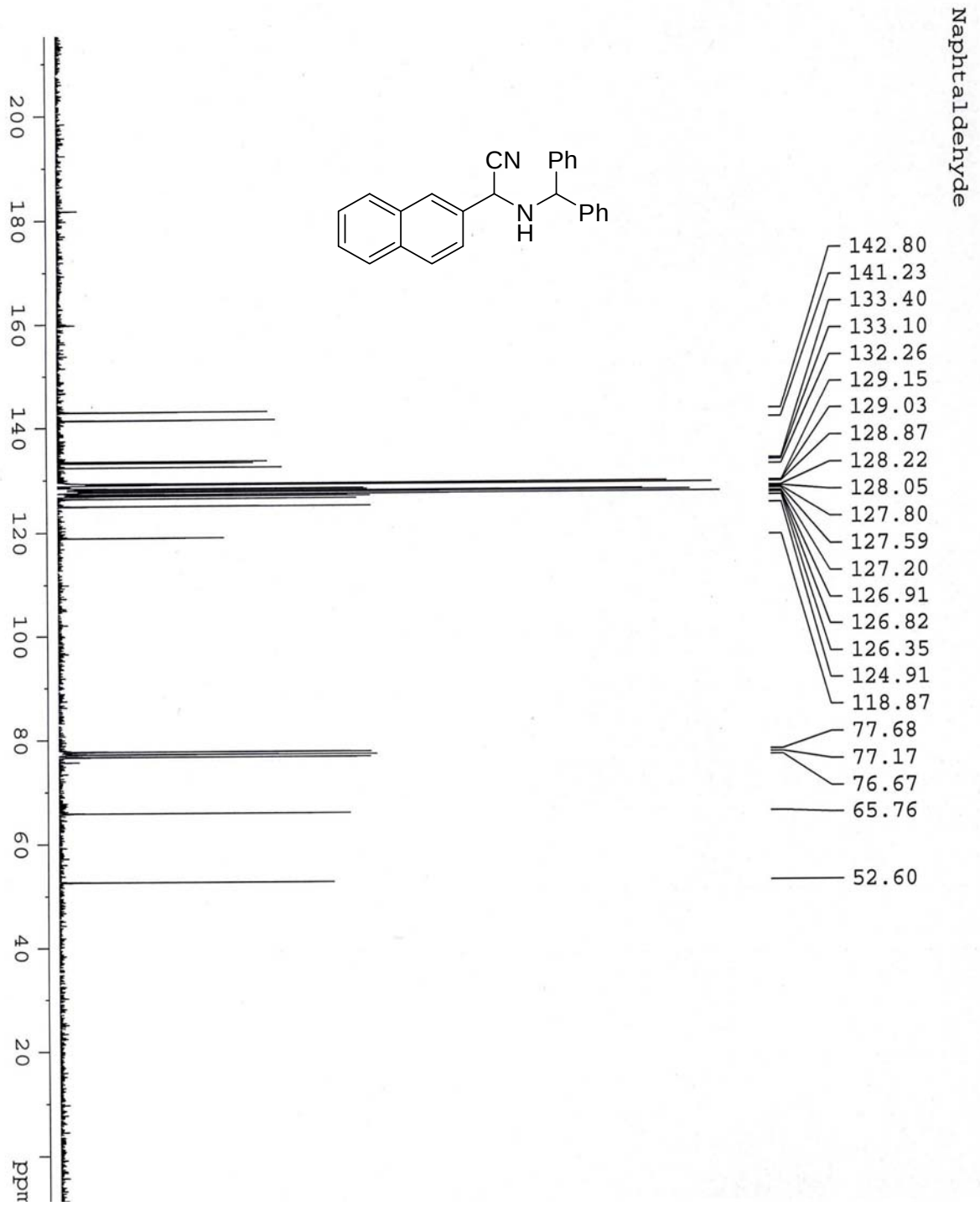
uv[1] Results

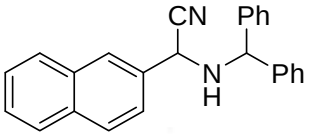
Retention Time	Area	Area %	Height	Height %
5.700	1909732	97.99	175390	99.18
13.333	39271	2.01	1444	0.82
Totals	1949003	100.00	176834	100.00

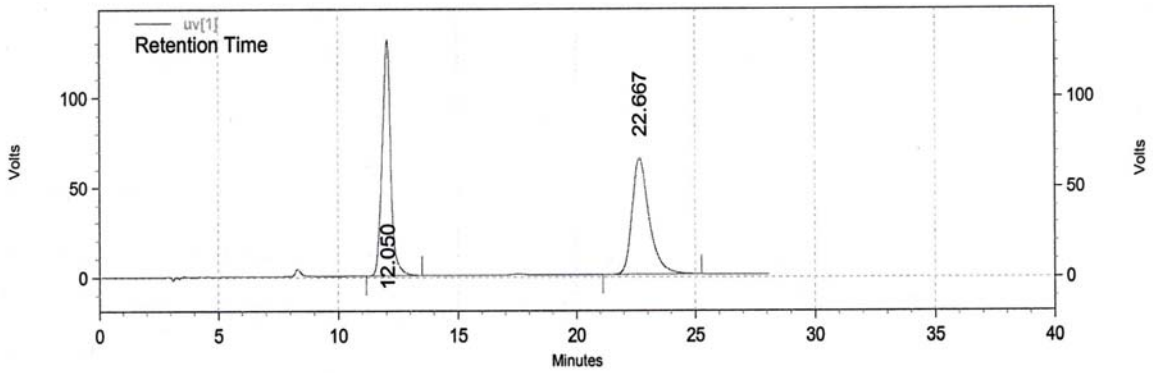

D:\OPUS\MEAS\EBRAHIMI\2,4-dimethyl.0
2,4-dimethyl
SOLID
14/09/2008
Transmittance [%]
20
40
60
80
100
Wavenumber cm-1
3500
3000
2500
2000
1500
1000
500
3321.32
3082.69
3052.11
3021.59
2985.46
2948.70
2921.62
2841.41
2786.62
2222.51
1758.45
1617.21
1596.79
1583.31
1507.36
1493.05
1478.97
1475.53
1450.68
1383.89
1344.86
1308.45
1288.54
1250.52
1202.00
1188.09
1170.04
1129.70
1092.58
1081.61
1059.15
1027.80
1001.65
938.17
926.93
907.23
877.73
838.23
823.82
806.72
781.88
756.98
748.26
726.82
707.50
699.89
668.84
622.81
610.37
585.94
549.24
526.23
477.48
454.51
431.39
Me
CN
Ph
N
H
Ph
Me


Me
CN
Ph
N
H
Ph
Me
DI/ZAREAEE-2,4-DIMETHYL/87.07.27
File : DI_66.X24 Date 8/23/10 Time 14:14:10
S=[49->74] Bp=167 Bi=358030. RT=1.22 CT=196
100%
75%
50%
25%
51
77
115
144
167
*8
219
249
282
300
30 50 70 90 110 130 150 170 190 210 230 250 270
280 300 320 340 360 380 400 420 440 460 480 500 520
SB=30 SE=327 DB=30 DE=530 N=0 Z=2 T=0.0 Fact[201->503] *8
S List > S=[49->74] B=0 Pos=8 Tot=8


CN
Ph
N
H
Ph
naphtaldehyde
7.945
7.932
7.911
7.893
7.878
7.697
7.665
7.628
7.622
7.600
7.586
7.574
7.559
7.550
7.523
7.495
7.466
7.436
7.394
7.367
7.338
7.313
7.297
7.285
7.261
7.045
5.363
5.090
4.797
2.284
2.099
1.02
3.19
13.47
1.09
1.00
1.06
10
9
8
7
6
5
4
3
2
1
ppm

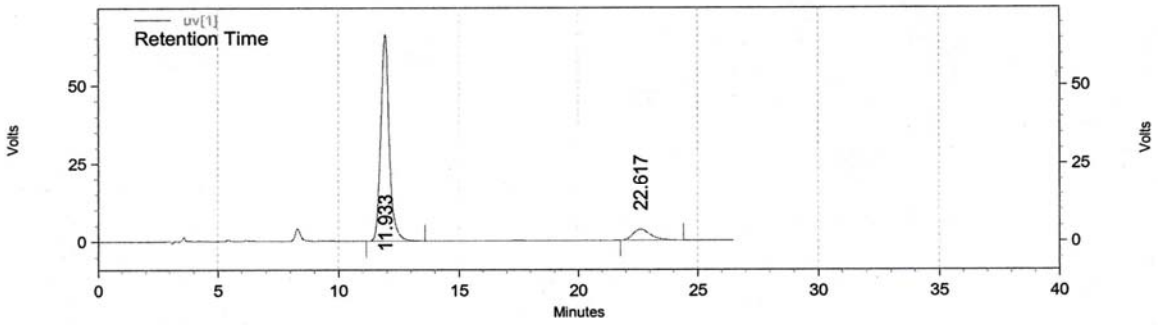

Naphtaldehyde
CN
Ph
N
H
Ph
142.80
141.23
133.40
133.10
132.26
129.15
129.03
128.87
128.22
128.05
127.80
127.59
127.20
126.91
126.82
126.35
124.91
118.87
77.68
77.17
76.67
65.76
52.60
200
180
160
140
120
100
80
60
40
20
ppm





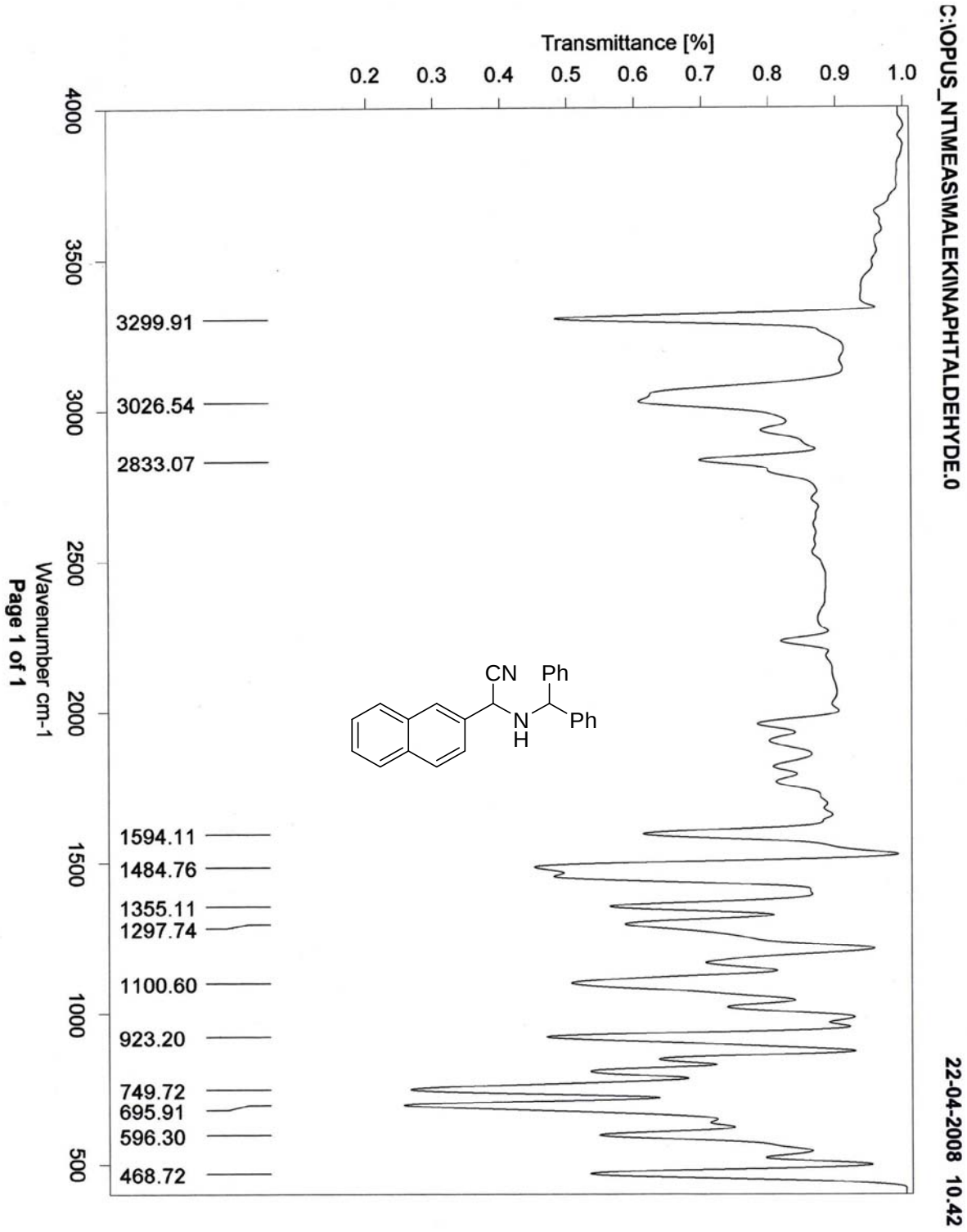
uv[1] Results

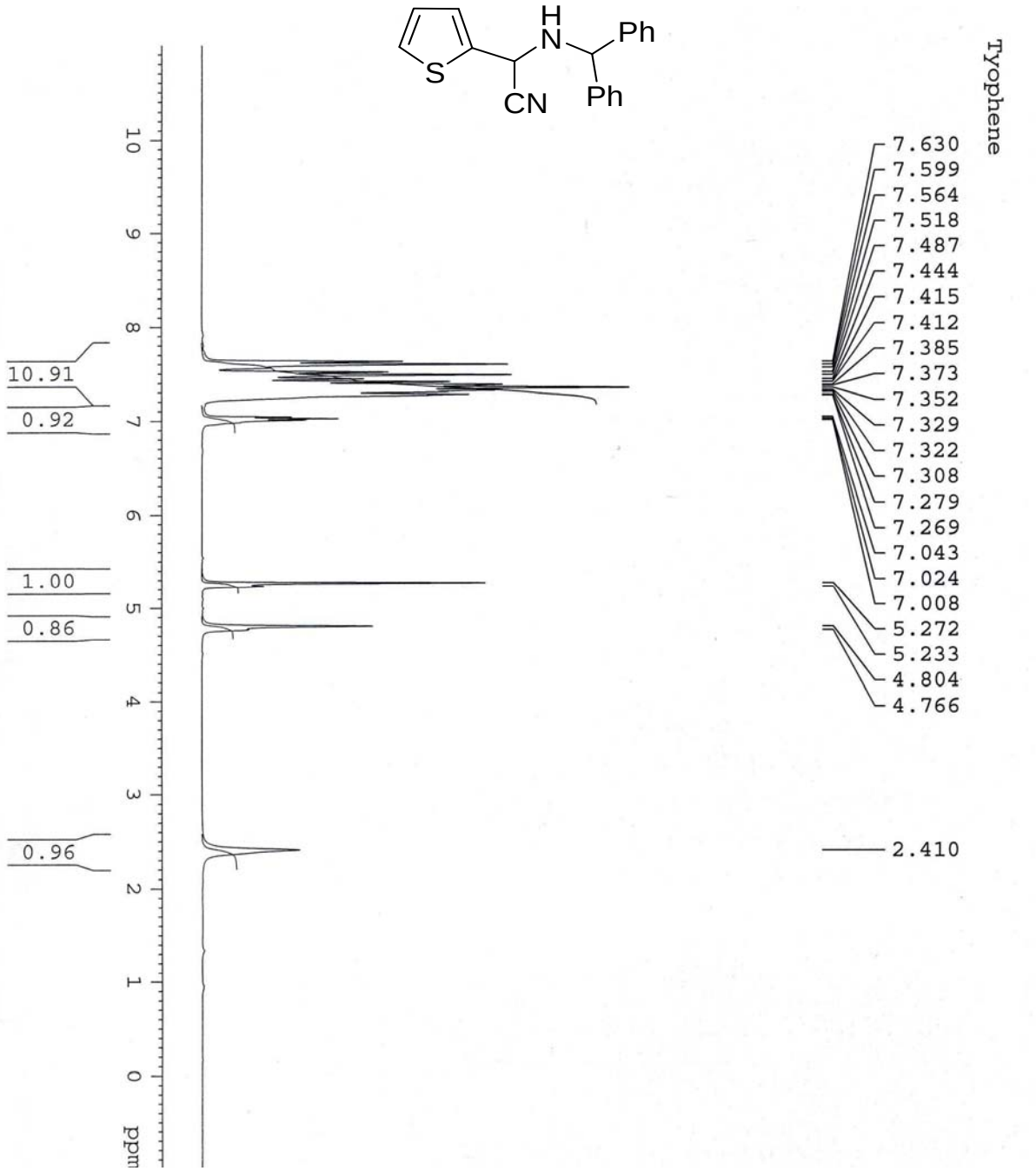
Retention Time	Area	Area %	Height	Height %
12.050	3249517	50.16	131220	66.90
22.667	3228331	49.84	64930	33.10
Totals	6477848	100.00	196150	100.00

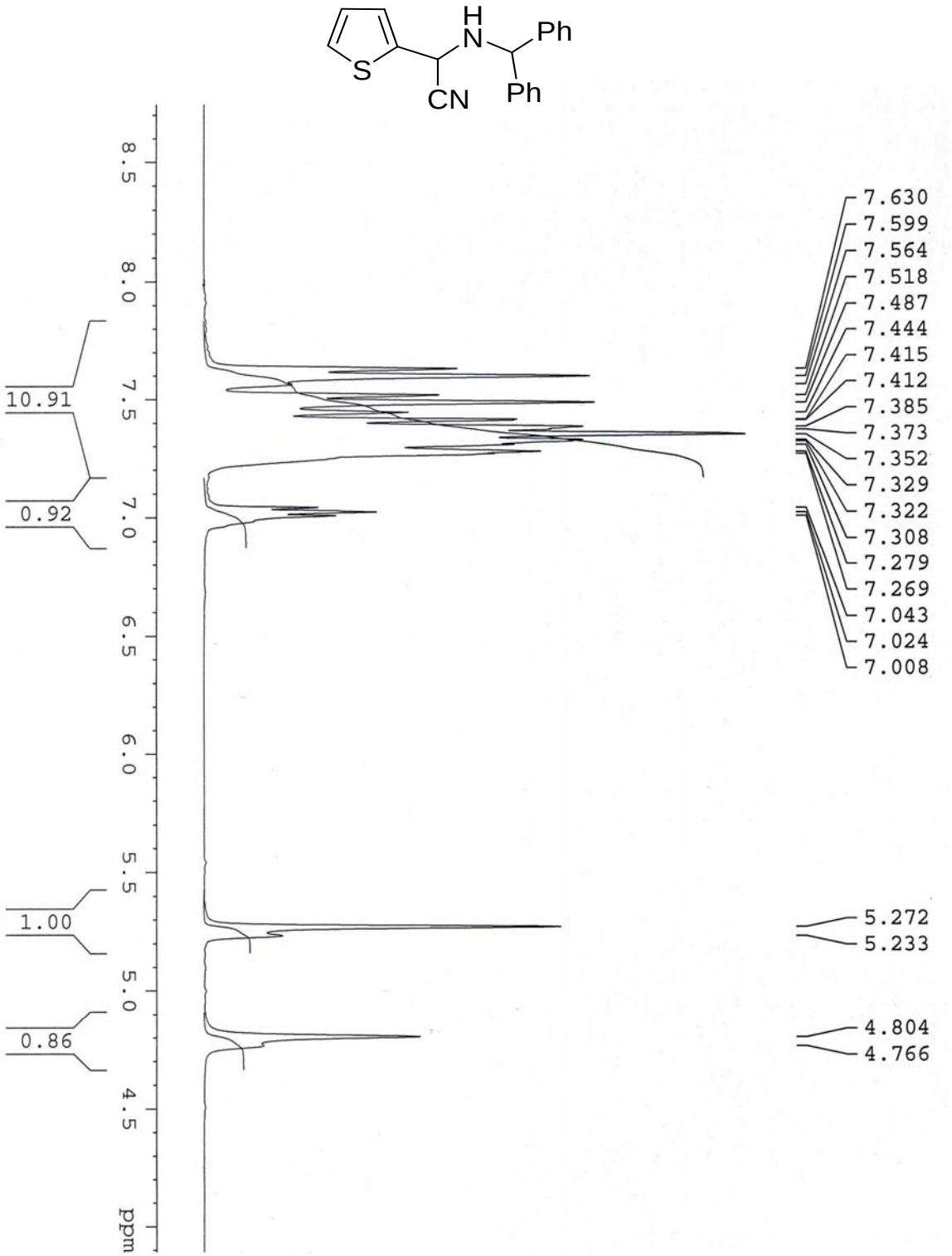


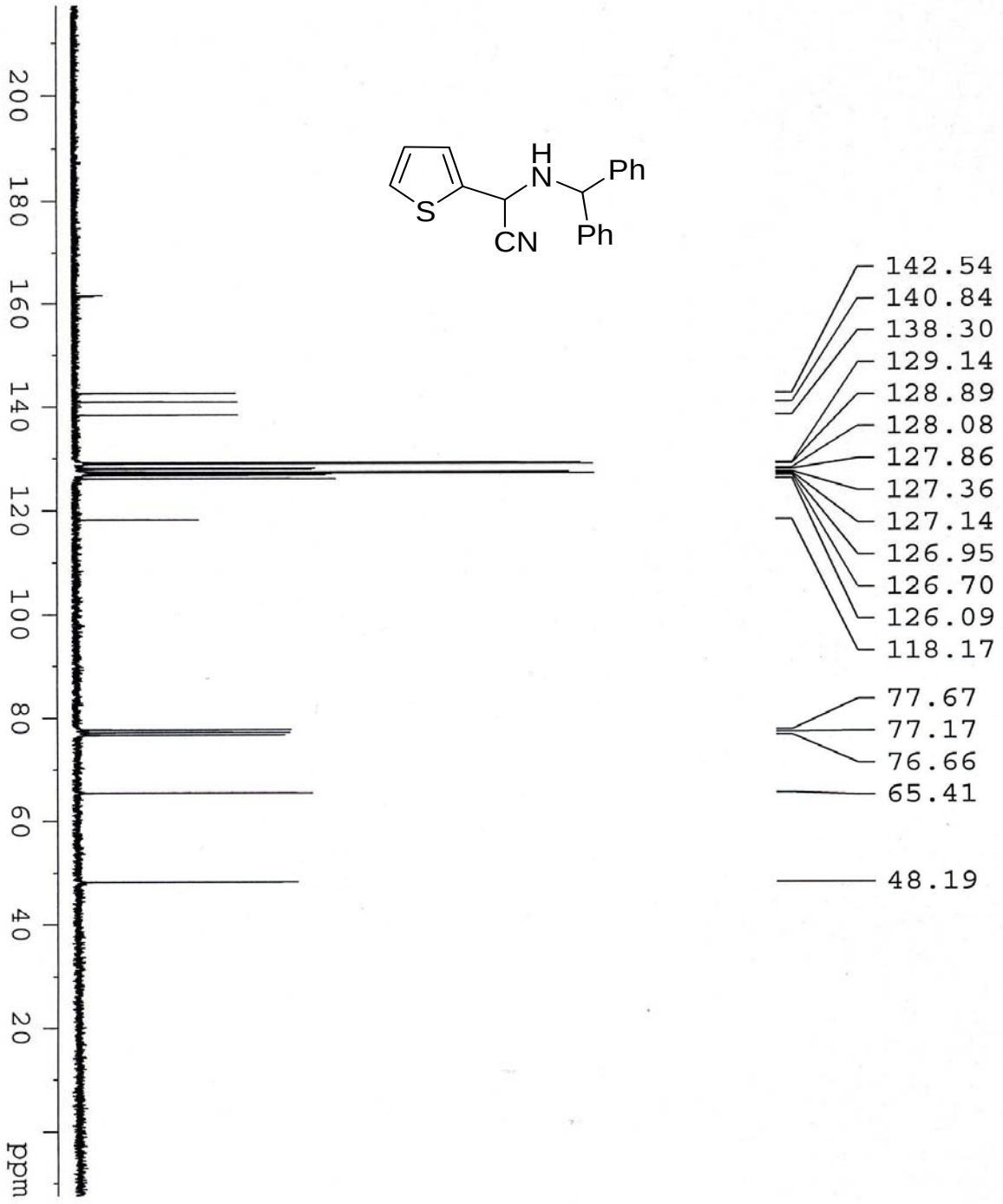
uv[1] Results

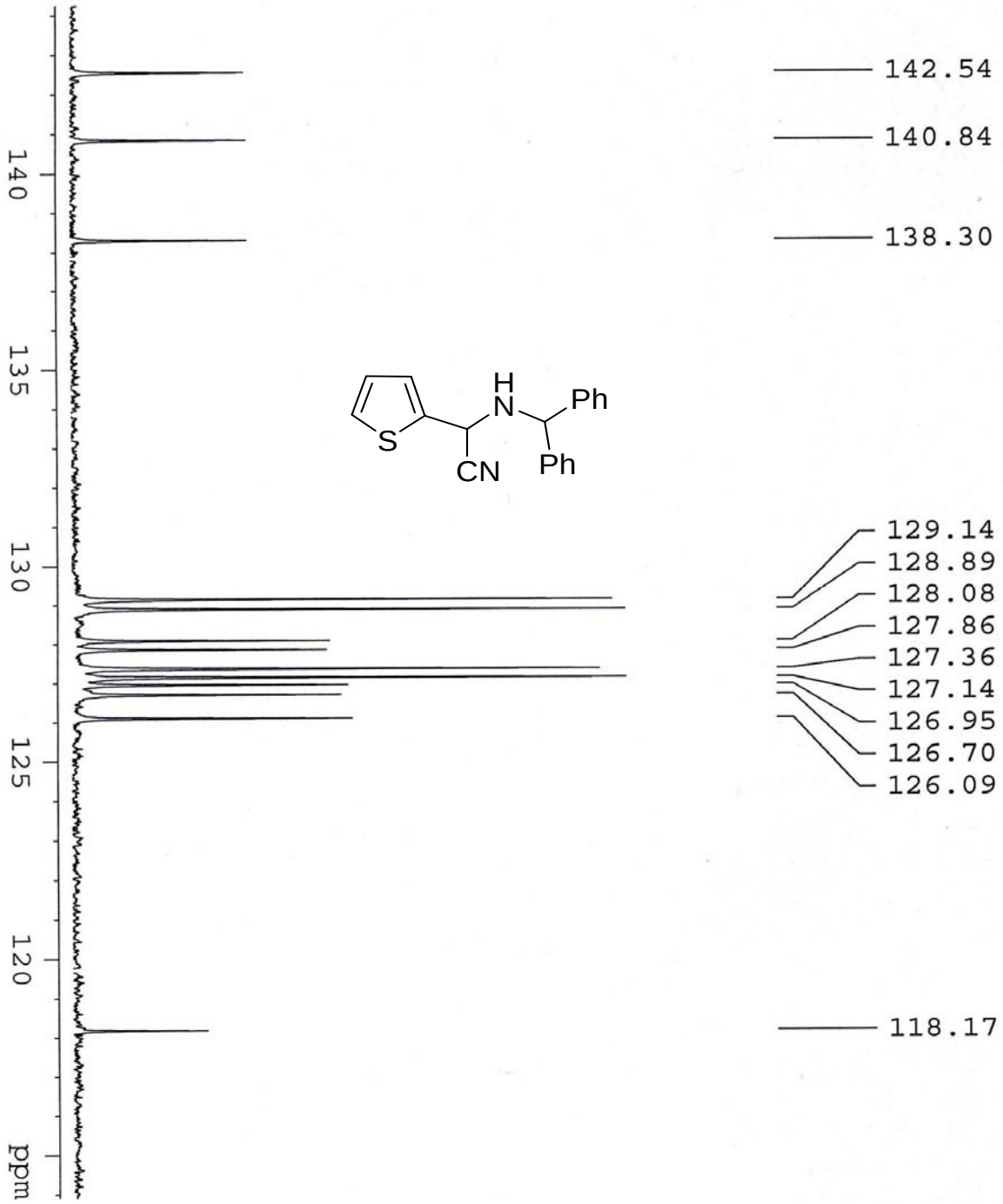
Retention Time	Area	Area %	Height	Height %
11.933	1614768	90.05	66144	94.97
22.617	178413	9.95	3504	5.03
Totals	1793181	100.00	69648	100.00

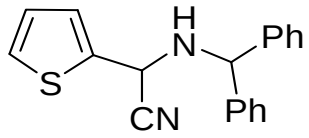

C:\OPUS_NT\MEAS\MALEKI\NAPHTALDEHYDE.0
Transmittance [%]
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1.0
4000
3500
3000
2500
2000
1500
1000
500
Wavenumber cm-1
Page 1 of 1
3299.91
3026.54
2833.07
1594.11
1484.76
1355.11
1297.74
1100.60
923.20
749.72
695.91
596.30
468.72
CN
Ph
N
H
Ph
22-04-2008 10.42

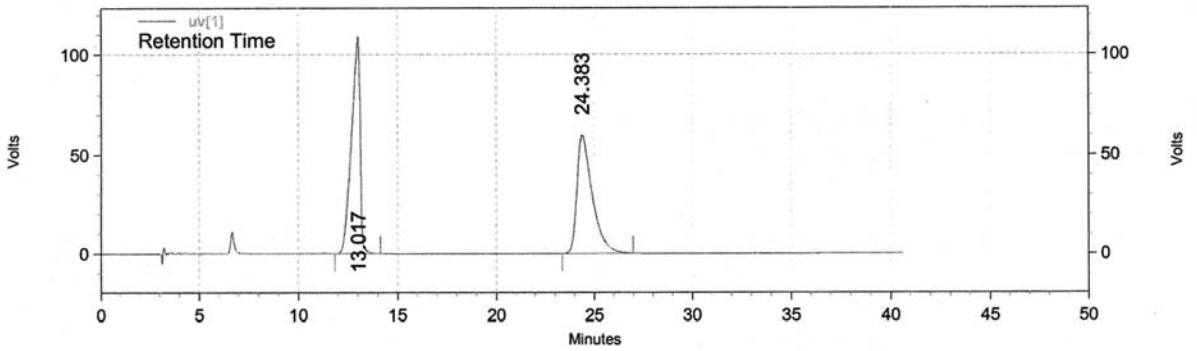

H
N
Ph
S
CN
Ph
Tyophene
7.630
7.599
7.564
7.518
7.487
7.444
7.415
7.412
7.385
7.373
7.352
7.329
7.322
7.308
7.279
7.269
7.043
7.024
7.008
5.272
5.233
4.804
4.766
2.410
10.91
0.92
1.00
0.86
0.96
10
9
8
7
6
5
4
3
2
1
0
ppm


H
N
Ph
S
CN
Ph
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
ppm
10.91
0.92
1.00
0.86
7.630
7.599
7.564
7.518
7.487
7.444
7.415
7.412
7.385
7.373
7.352
7.329
7.322
7.308
7.279
7.269
7.043
7.024
7.008
5.272
5.233
4.804
4.766


200
180
160
140
120
100
80
60
40
20
ppm
S
H
N
Ph
CN
Ph
142.54
140.84
138.30
129.14
128.89
128.08
127.86
127.36
127.14
126.95
126.70
126.09
118.17
77.67
77.17
76.66
65.41
48.19

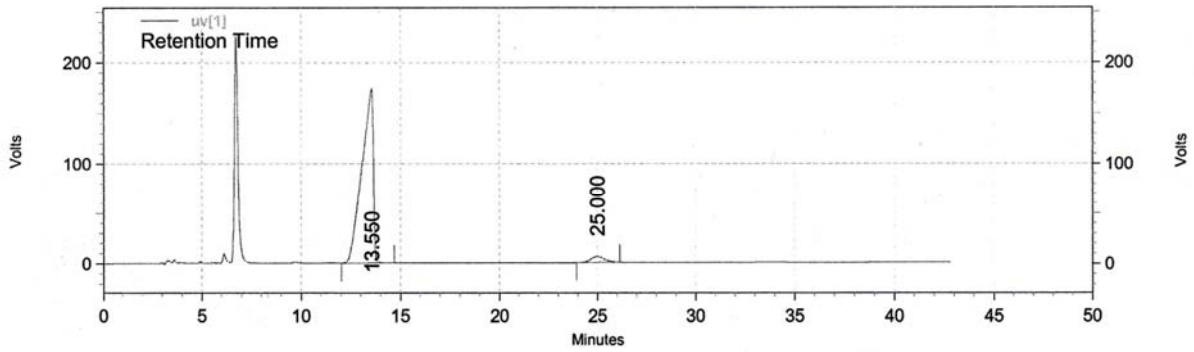

142.54
140.84
138.30
129.14
128.89
128.08
127.86
127.36
127.14
126.95
126.70
126.09
118.17
140
135
130
125
120
ppm
H
N
Ph
Ph
S
CN





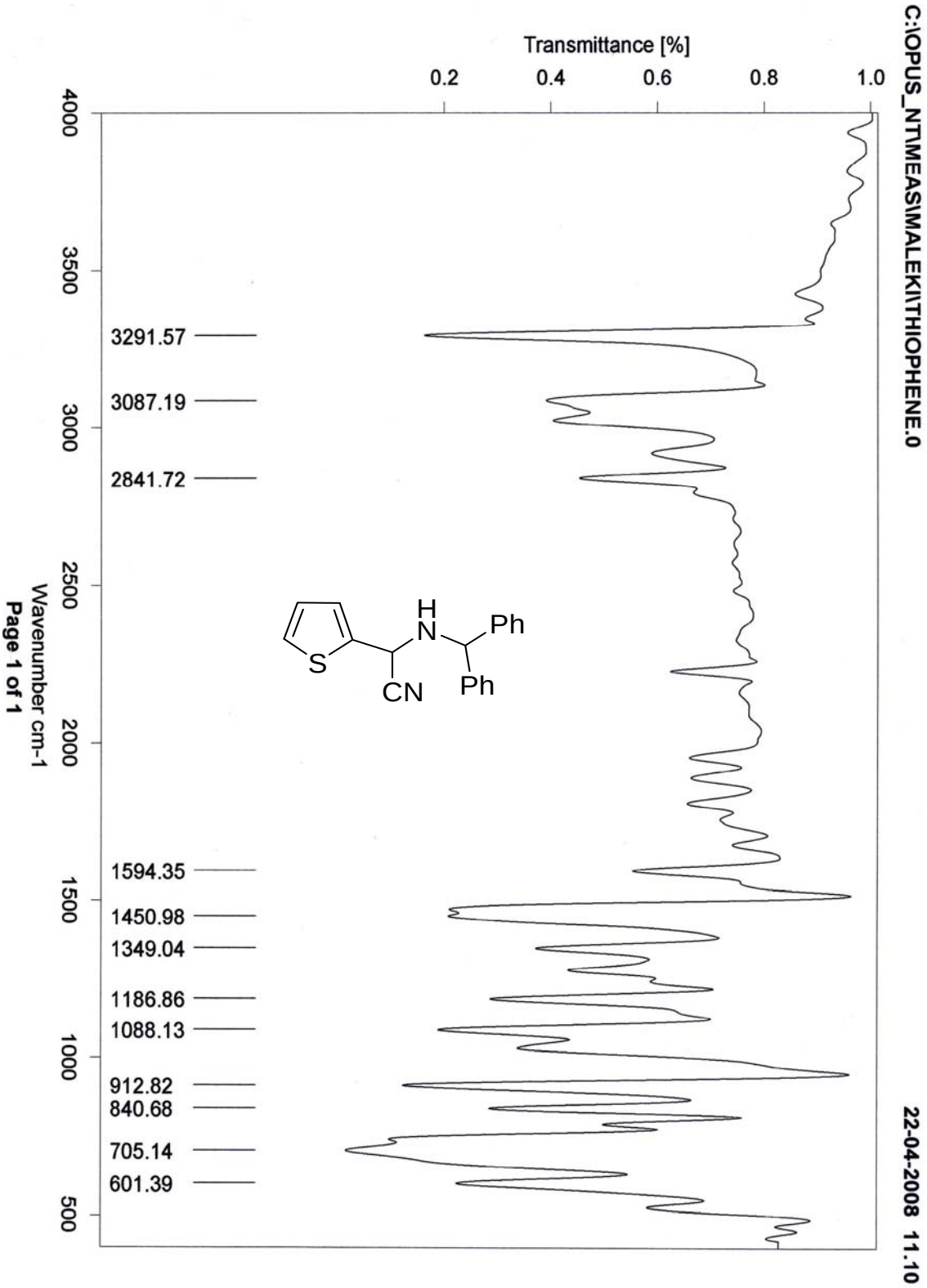
uv[1] Results

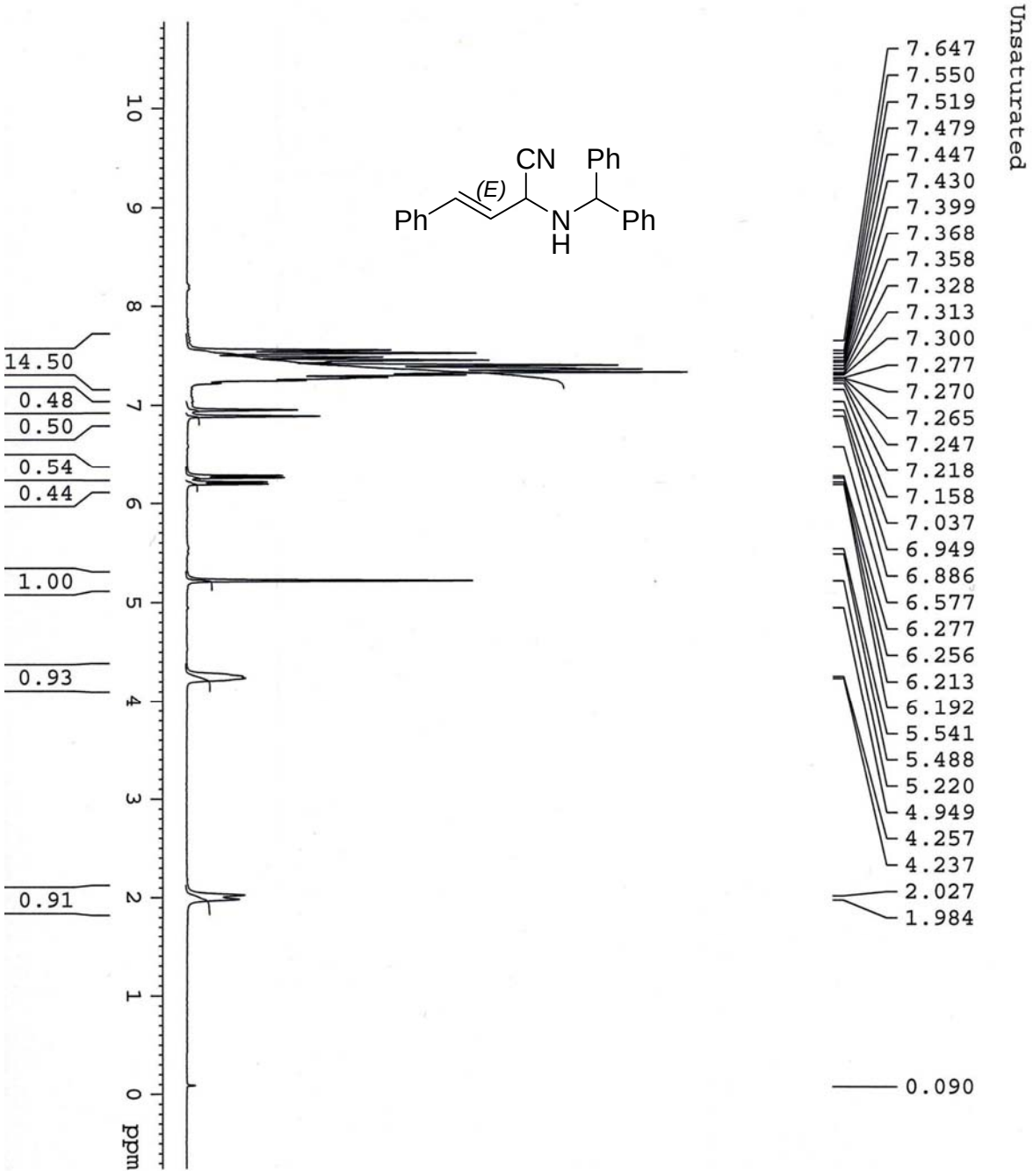
Retention Time	Area	Area %	Height	Height %
13.017	3304310	50.45	109150	64.62
24.383	3245890	49.55	59772	35.38
Totals	6550200	100.00	168922	100.00

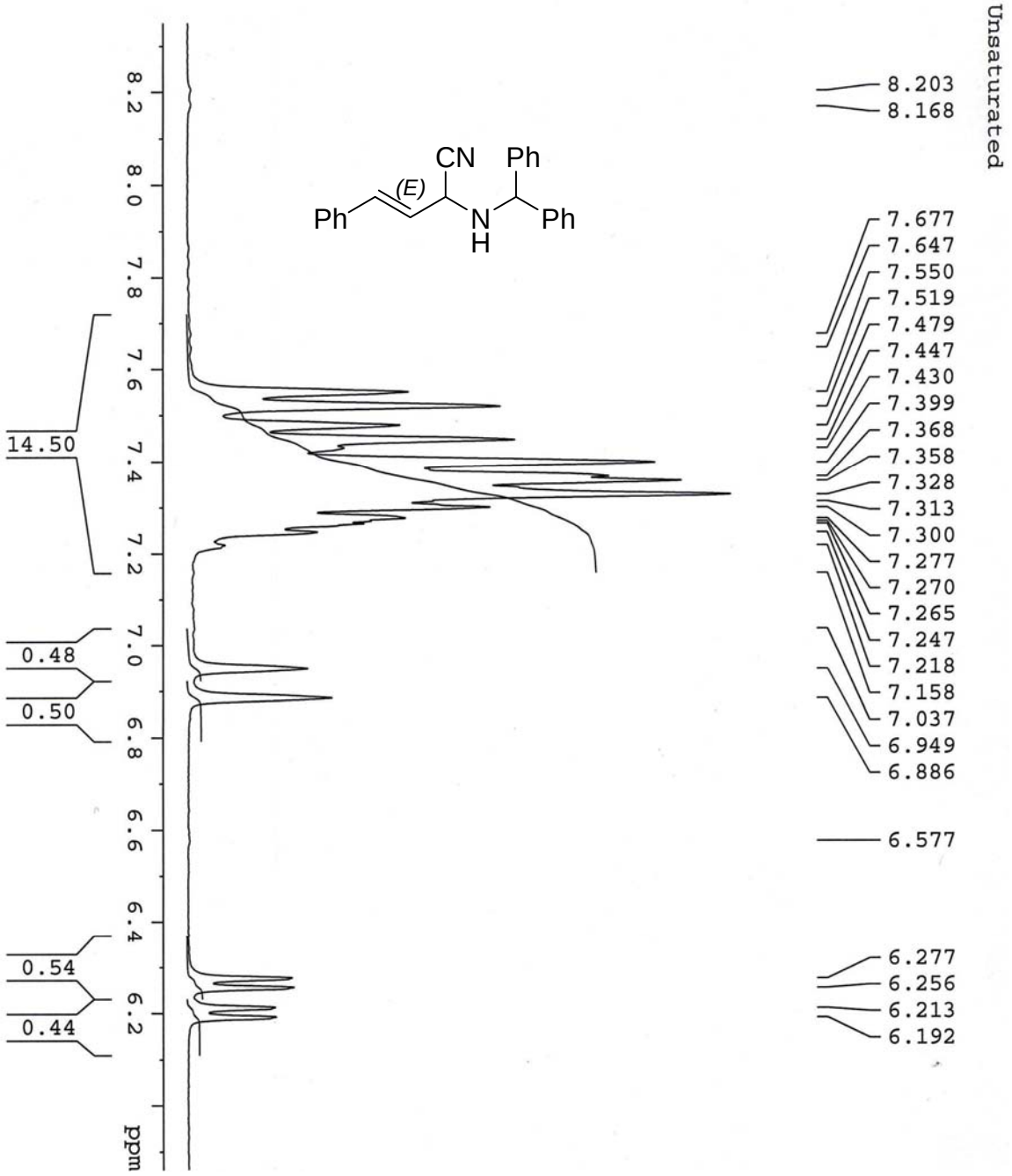


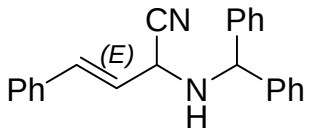
uv[1] Results

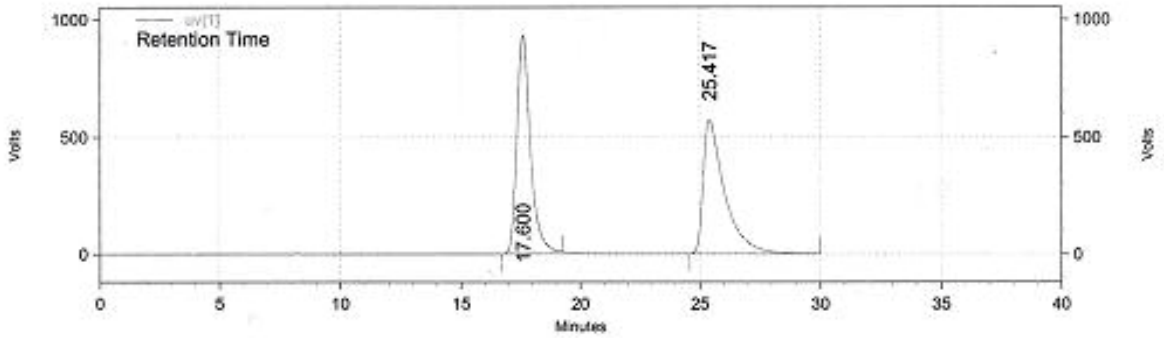
Retention Time	Area	Area %	Height	Height %
13.550	6964558	96.05	173753	96.70
25.000	286588	3.95	5932	3.30
Totals	7251146	100.00	179685	100.00


C:\OPUS_NT\MEAS\MALEKI\THIOPHENE.0
Transmittance [%]
0.2
0.4
0.6
0.8
1.0
4000
3500
3000
2500
2000
1500
1000
500
Wavenumber cm-1
Page 1 of 1
3291.57
3087.19
2841.72
1594.35
1450.98
1349.04
1186.86
1088.13
912.82
840.68
705.14
601.39
H
N
Ph
Ph
S
CN
22-04-2008 11.10


CN
Ph
(E)
Ph
N
H
Ph
14.50
0.48
0.50
0.54
0.44
1.00
0.93
0.91
ppm
Unsaturated
7.647
7.550
7.519
7.479
7.447
7.430
7.399
7.368
7.358
7.328
7.313
7.300
7.277
7.270
7.265
7.247
7.218
7.158
7.037
6.949
6.886
6.577
6.277
6.256
6.213
6.192
5.541
5.488
5.220
4.949
4.257
4.237
2.027
1.984
0.090

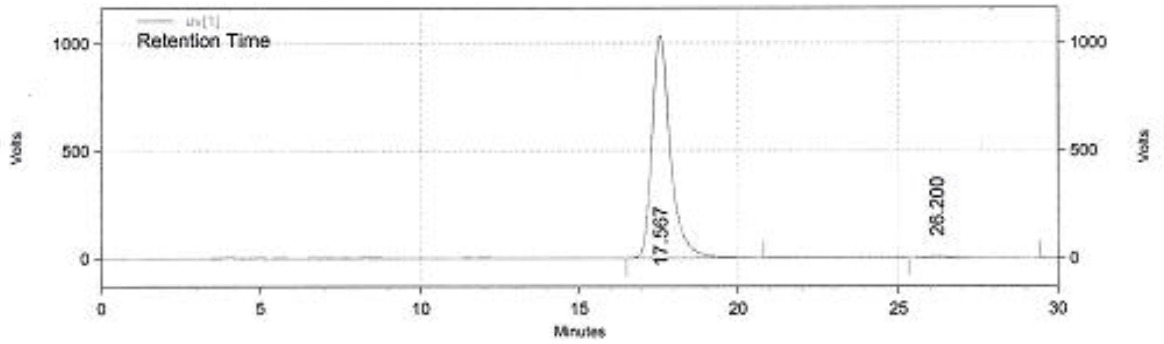

Unsaturated
8.203
8.168
7.677
7.647
7.550
7.519
7.479
7.447
7.430
7.399
7.368
7.358
7.328
7.313
7.300
7.277
7.270
7.265
7.247
7.218
7.158
7.037
6.949
6.886
6.577
6.277
6.256
6.213
6.192
14.50
0.48
0.50
0.54
0.44
8.2
8.0
7.8
7.6
7.4
7.2
7.0
6.8
6.6
6.4
6.2
ppm
CN
Ph
(E)
Ph
N
H
Ph





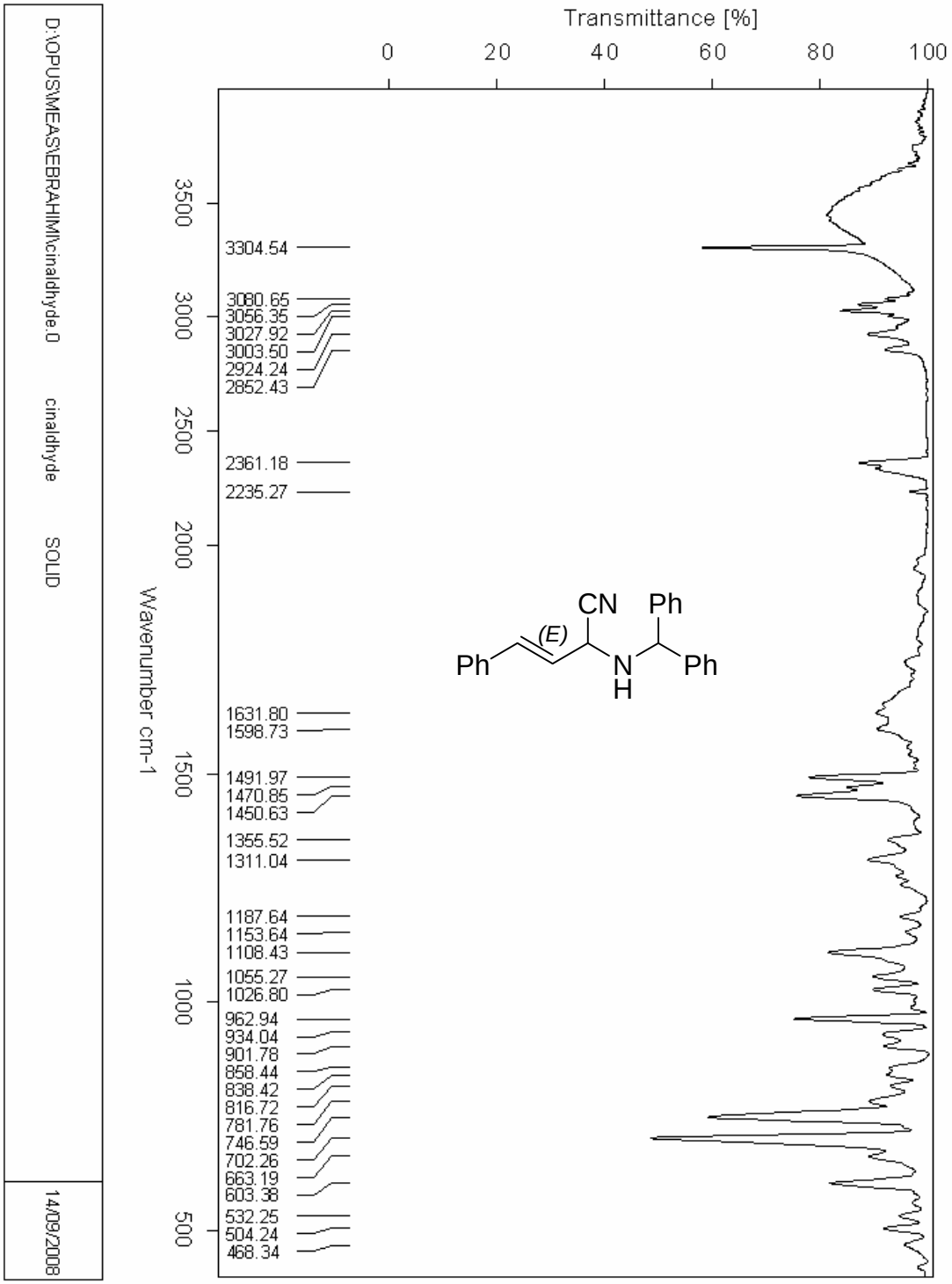
uv[1] Results

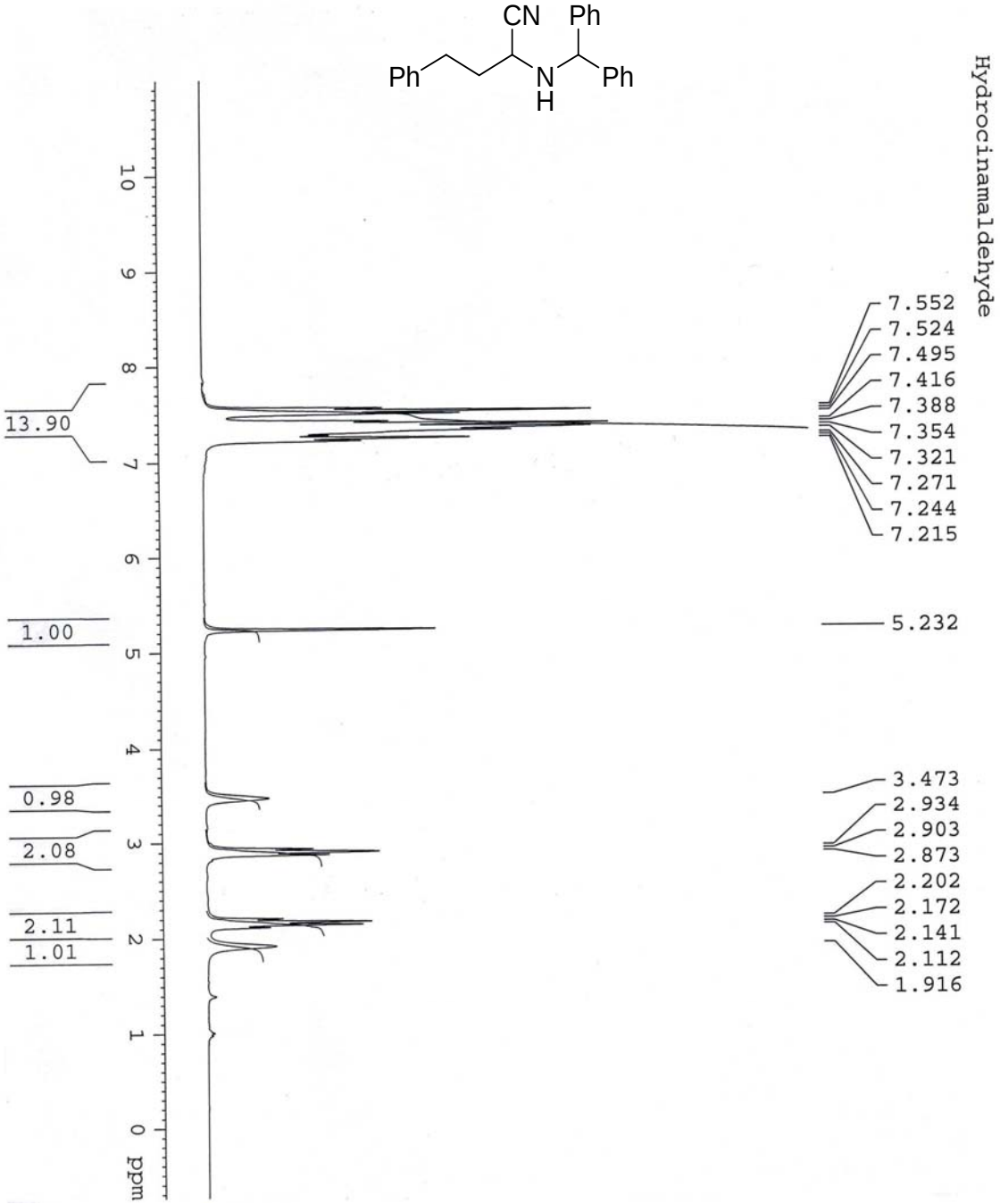
Retention Time	Area	Area %	Height	Height %
17.600	36533190	50.85	931955	61.97
25.417	35304911	49.15	571991	38.03
Totals	71838101	100.00	1503946	100.00

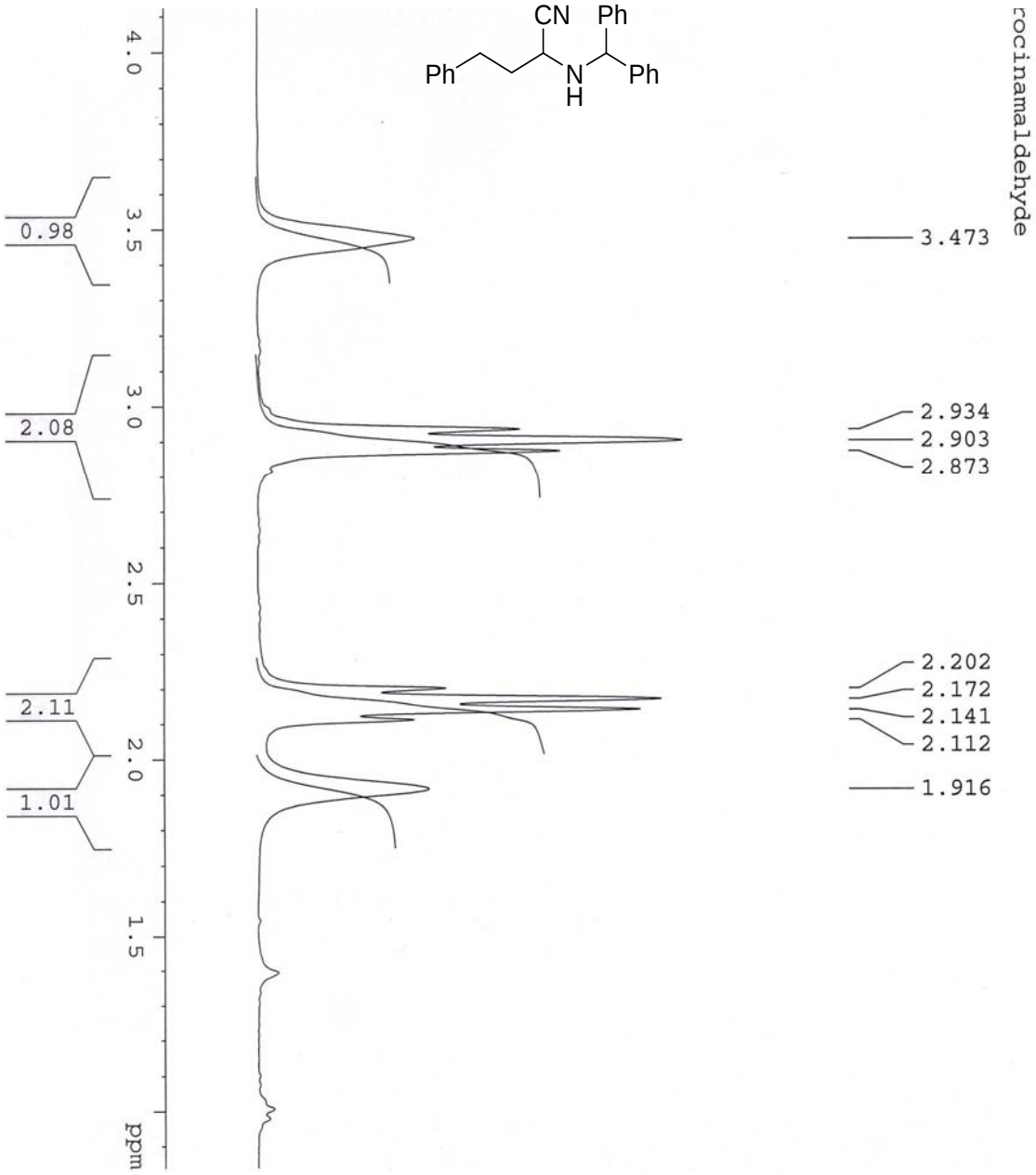


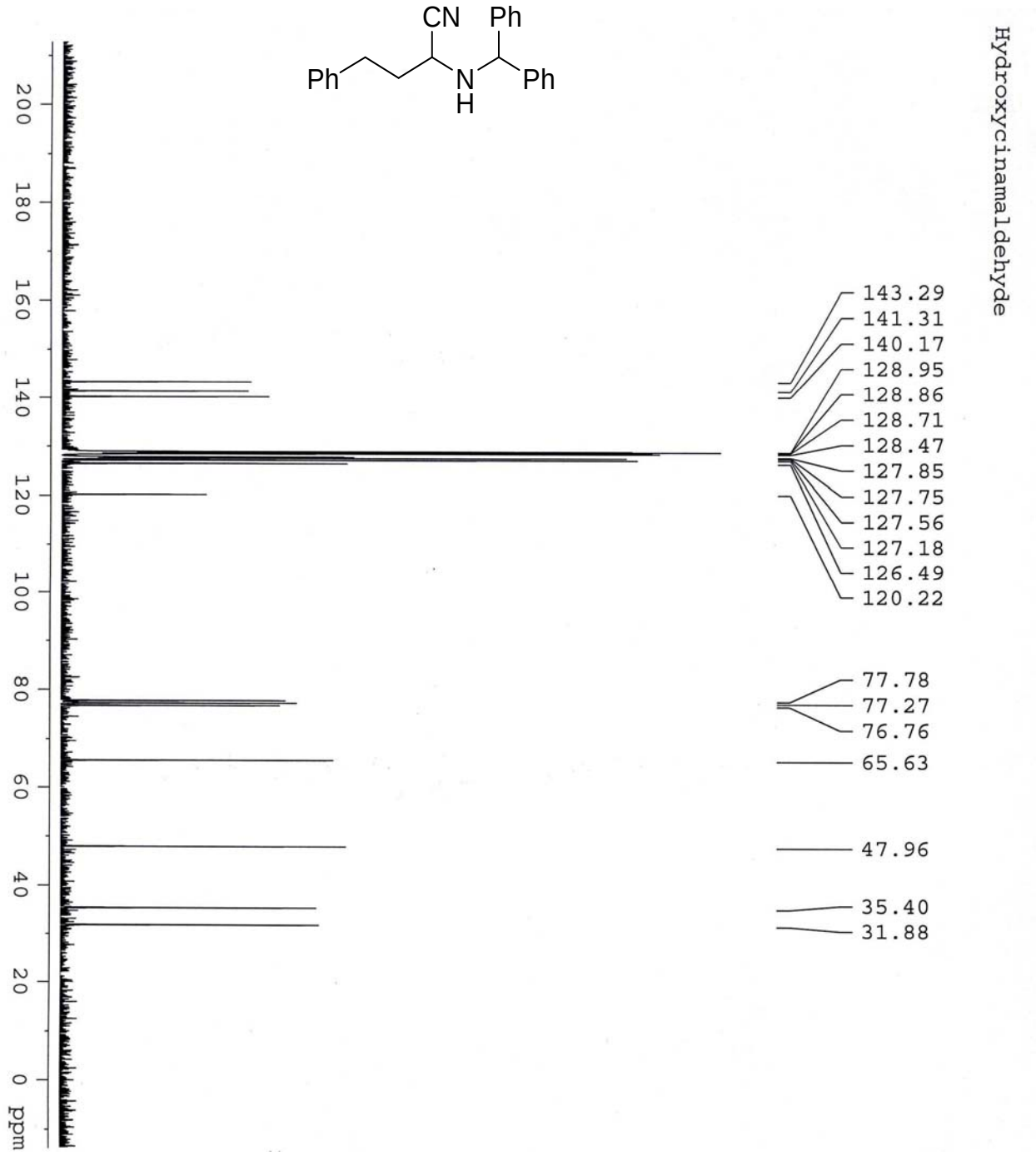
uv[1] Results

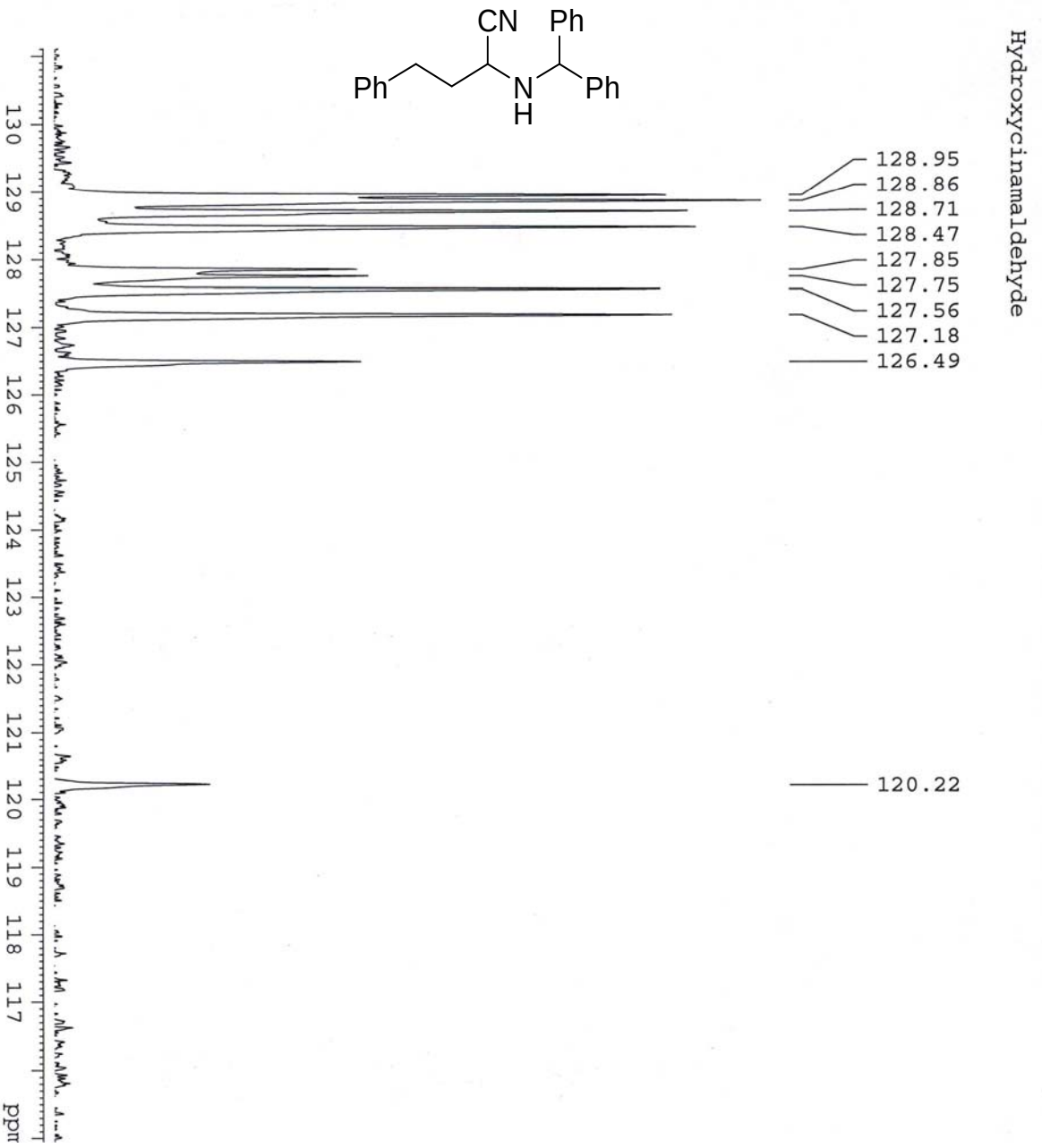
Retention Time	Area	Area %	Height	Height %
17.567	41785244	98.96	1032644	99.36
26.200	439235	1.04	6647	0.64
Totals	42224479	100.00	1039291	100.00

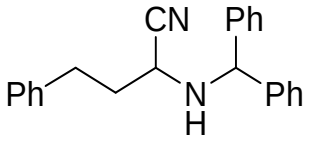

D:\OPUS\MEAS\EBRAHIM\cinaldhyde.0
cinaldhyde
SOLID
14/09/2008
Transmittance [%]
0
20
40
60
80
100
Wavenumber cm-1
3500
3000
2500
2000
1500
1000
500
3304.54
3080.65
3056.35
3027.92
3003.50
2924.24
2852.43
2361.18
2235.27
1631.80
1598.73
1491.97
1470.85
1450.63
1355.52
1311.04
1187.64
1153.64
1108.43
1055.27
1026.80
962.94
934.04
901.78
858.44
838.42
816.72
781.76
746.59
702.26
663.19
603.38
532.25
504.24
468.34
CN
Ph
(E)
Ph
N
H
Ph

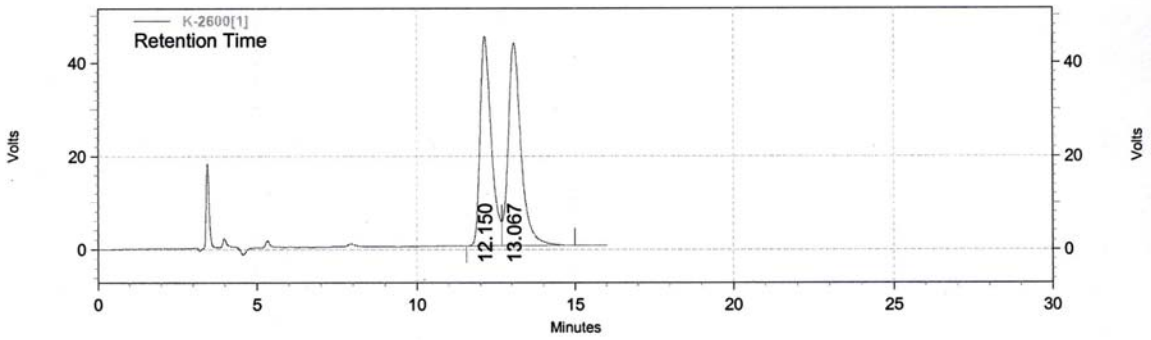

CN
Ph
Ph
N
H
Ph
Hydrocinnamaldehyde
7.552
7.524
7.495
7.416
7.388
7.354
7.321
7.271
7.244
7.215
5.232
3.473
2.934
2.903
2.873
2.202
2.172
2.141
2.112
1.916
13.90
1.00
0.98
2.08
2.11
1.01
ppm


CN
Ph
Ph
N
H
Ph
rocinamaldehyde
4.0
3.5
3.0
2.5
2.0
1.5
ppm
0.98
2.08
2.11
1.01
3.473
2.934
2.903
2.873
2.202
2.172
2.141
2.112
1.916


CN
Ph
Ph
N
H
Ph
Hydroxycinamaldehyde
143.29
141.31
140.17
128.95
128.86
128.71
128.47
127.85
127.75
127.56
127.18
126.49
120.22
77.78
77.27
76.76
65.63
47.96
35.40
31.88
200
180
160
140
120
100
80
60
40
20
0
ppm

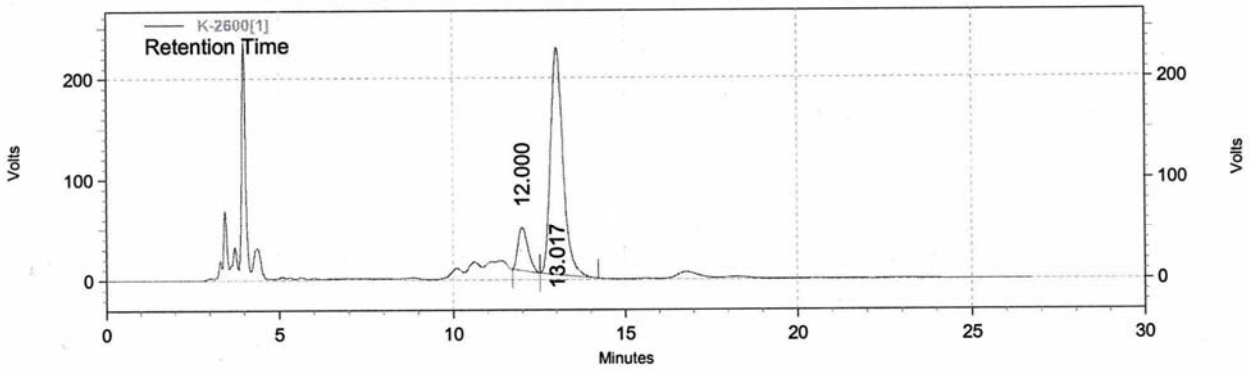

CN
Ph
Ph
N
H
Ph
Hydroxycinamaldehyde
128.95
128.86
128.71
128.47
127.85
127.75
127.56
127.18
126.49
120.22
130
129
128
127
126
125
124
123
122
121
120
119
118
117
ppm





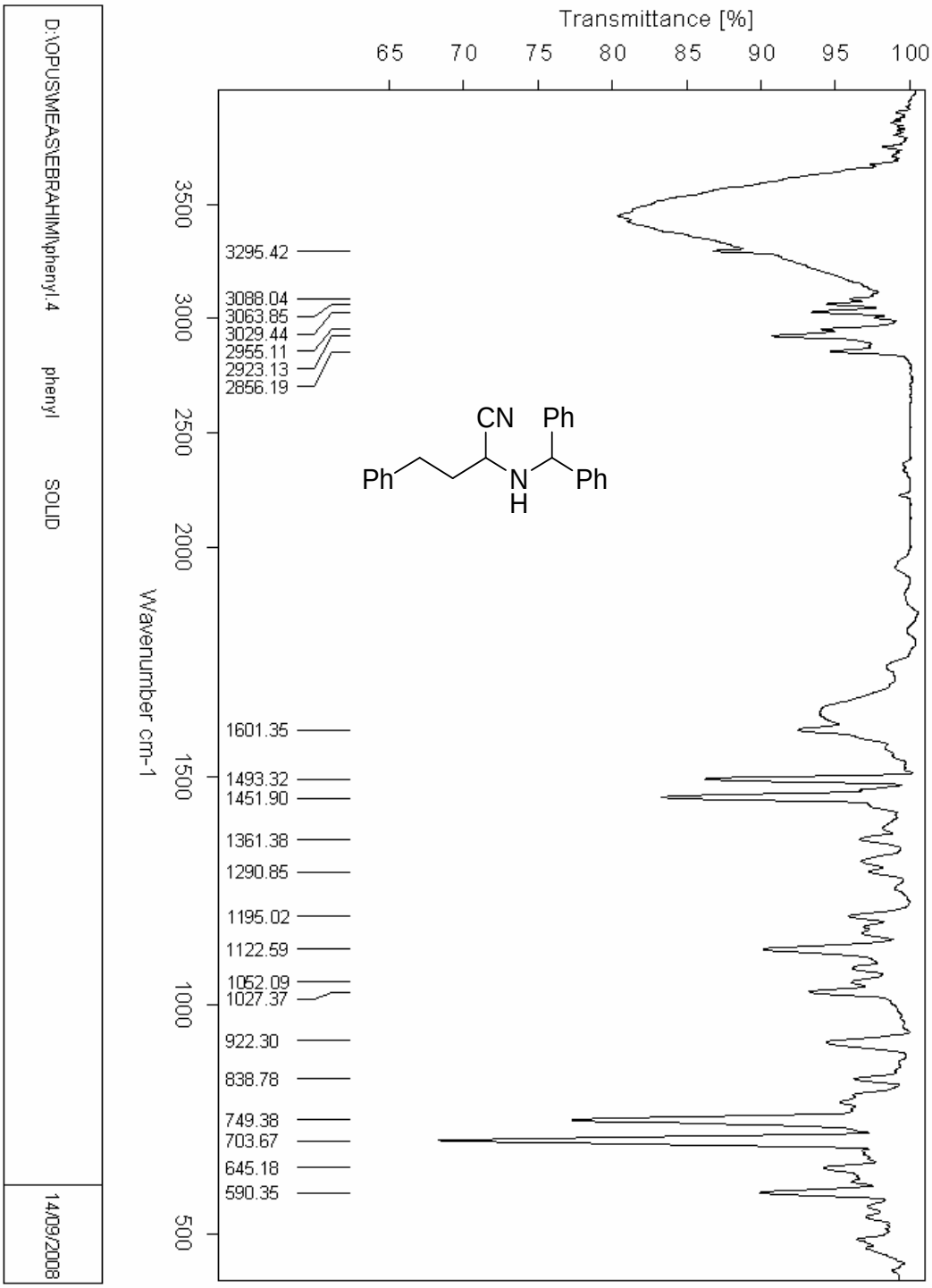
K-2600[1] Results

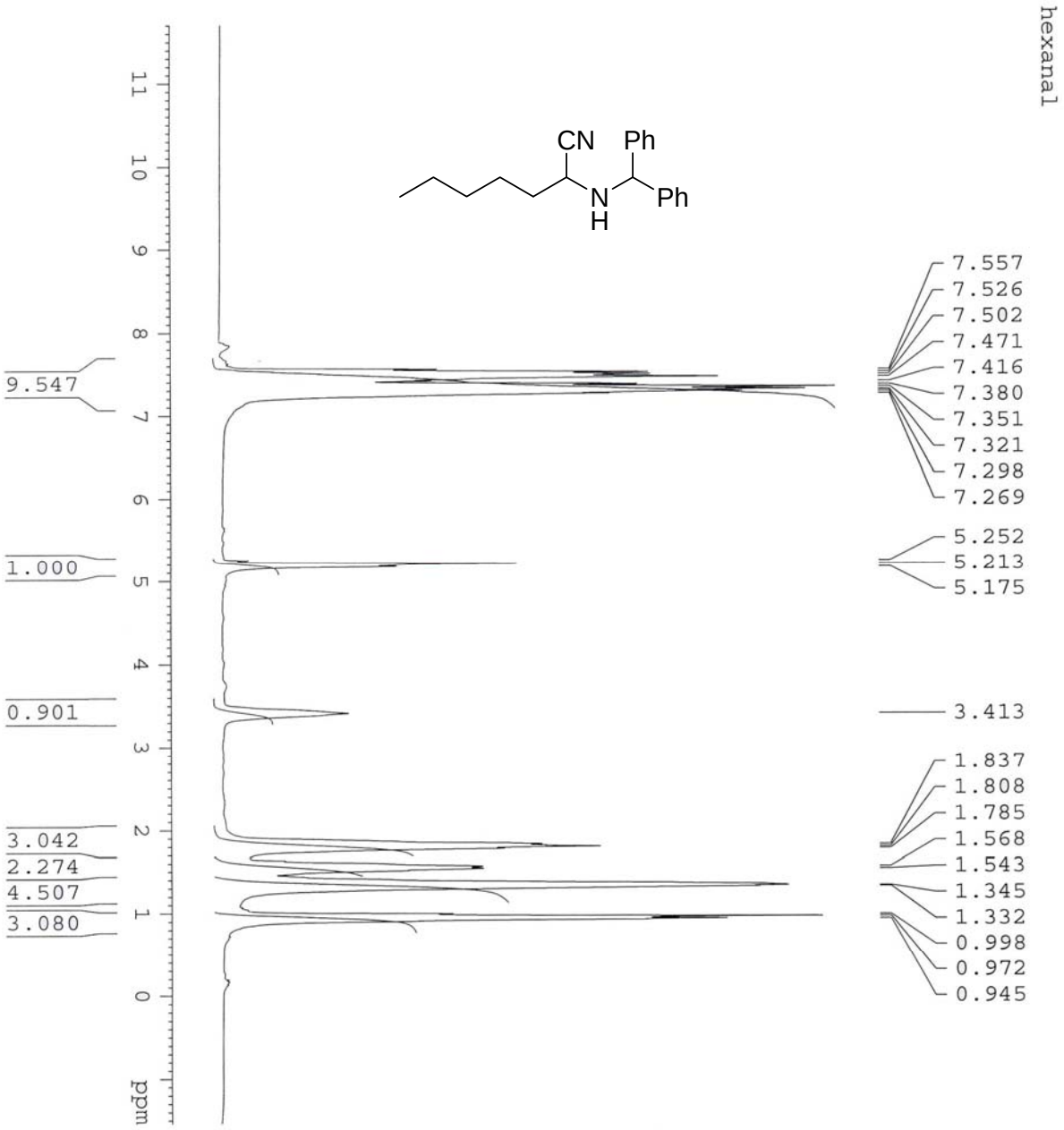
Retention Time	Area	Area %	Height	Height %
12.150	1096697	47.35	44898	50.86
13.067	1219420	52.65	43374	49.14
Totals	2316117	100.00	88272	100.00

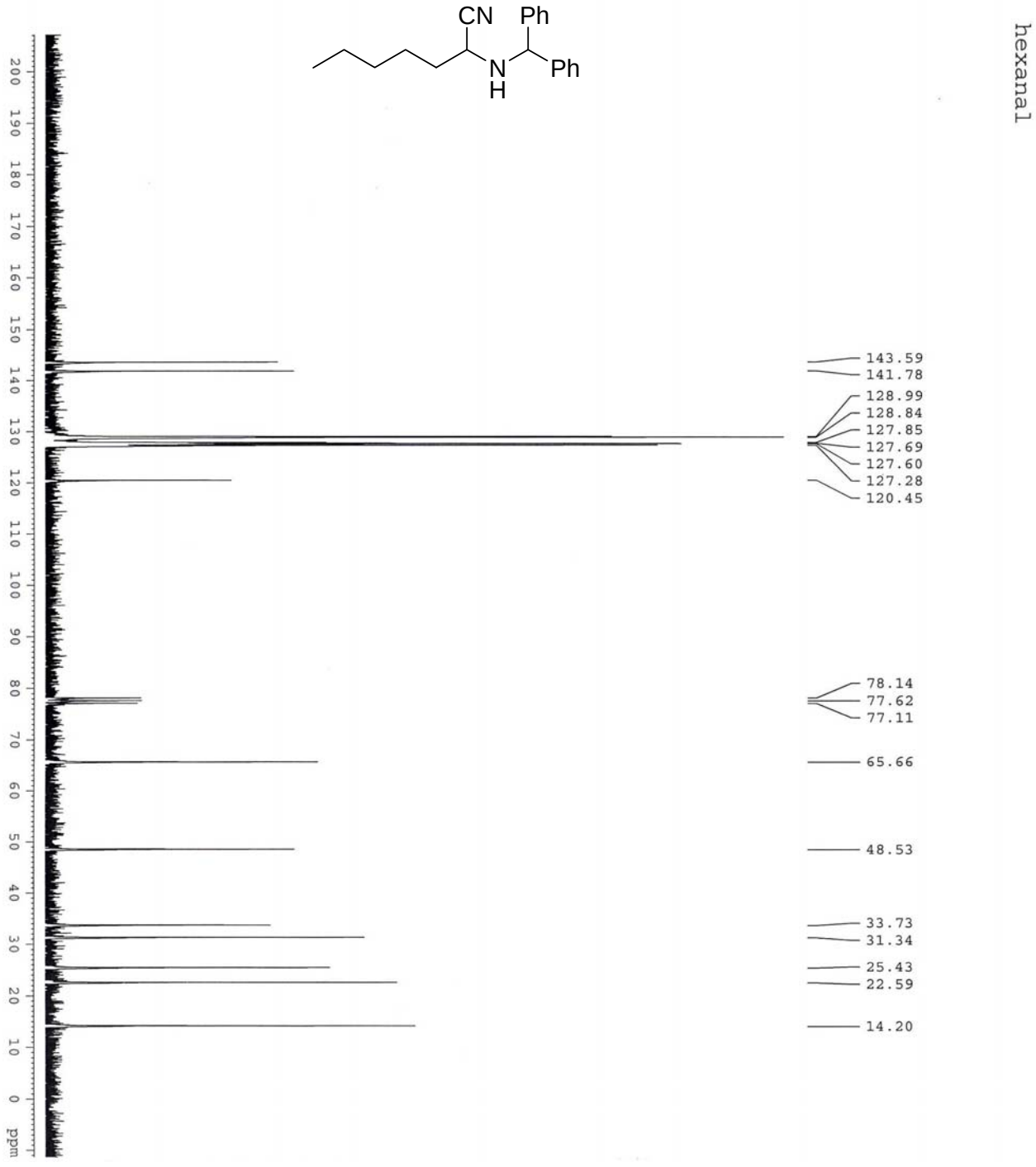


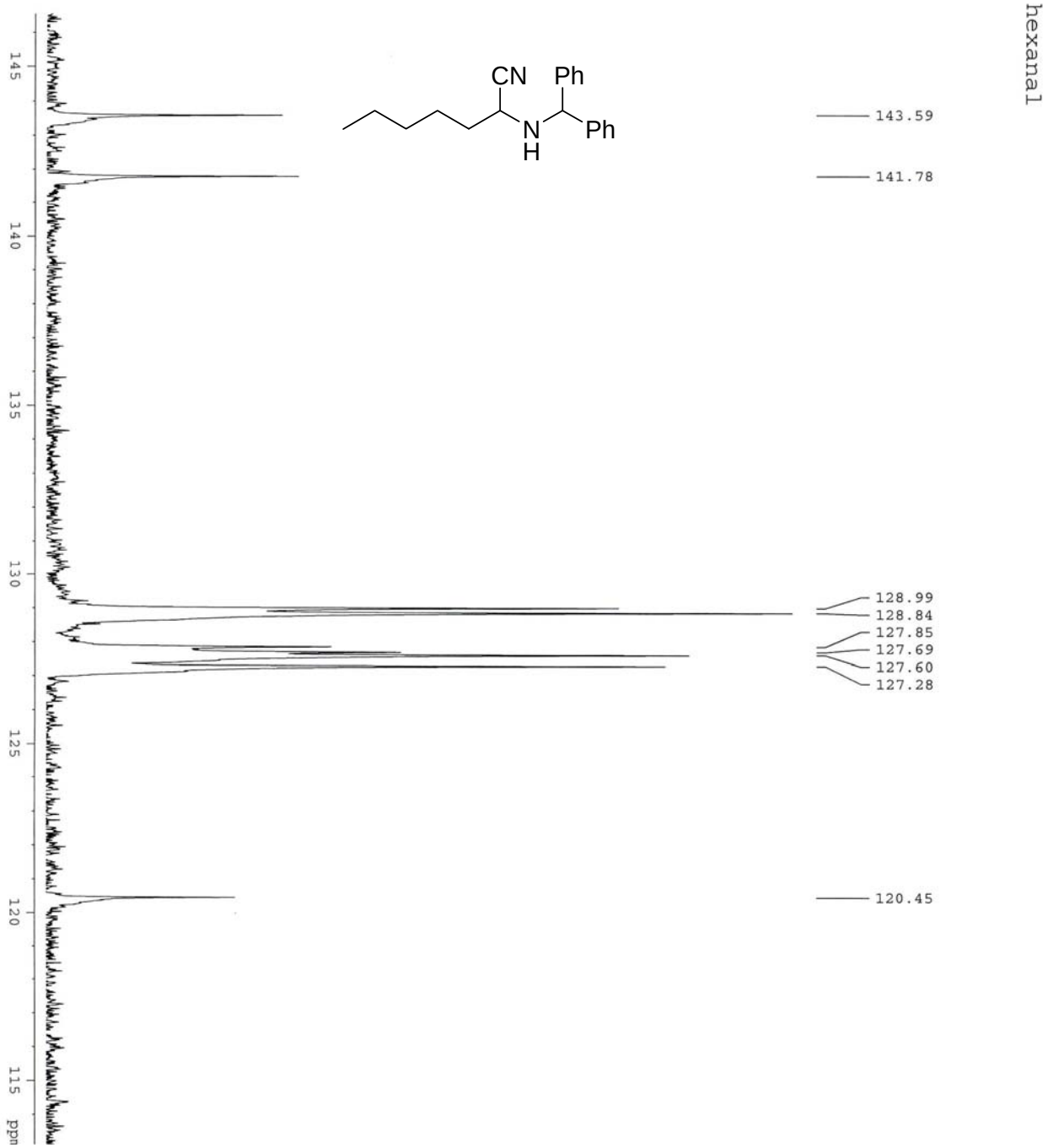
K-2600[1] Results

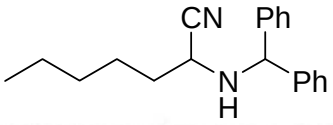
Retention Time	Area	Area %	Height	Height %
12.000	854778	13.12	42374	15.85
13.017	5660312	86.88	224982	84.15
Totals	6515090	100.00	267356	100.00

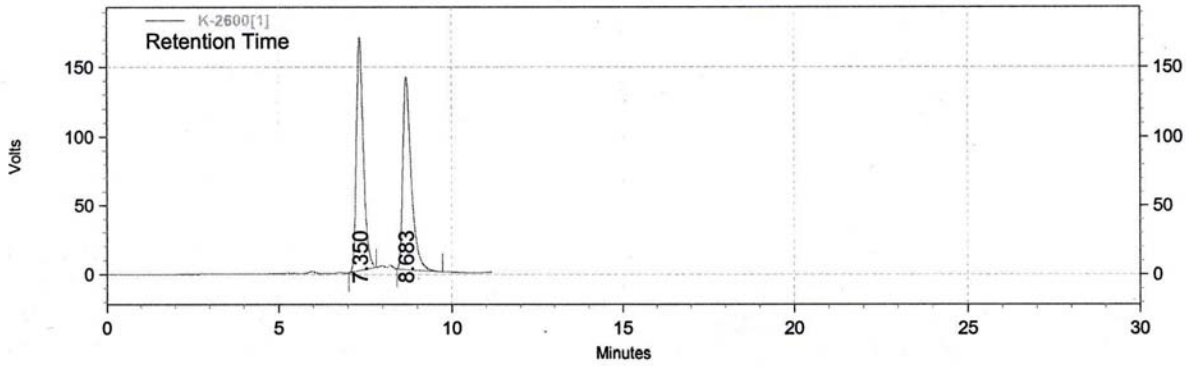

D:\OPUS\MEAS\EBRAHIM\phenyl.4
phenyl
SOLID
14/09/2008
Transmittance [%]
65
70
75
80
85
90
95
100
Wavenumber cm-1
3500
3000
2500
2000
1500
1000
500
3295.42
3088.04
3063.85
3029.44
2955.11
2923.13
2856.19
1601.35
1493.32
1451.90
1361.38
1290.85
1195.02
1122.59
1052.09
1027.37
922.30
838.78
749.38
703.67
645.18
590.35
CN
Ph
Ph
N
H
Ph


hexanal
CN
Ph
N
H
Ph
7.557
7.526
7.502
7.471
7.416
7.380
7.351
7.321
7.298
7.269
5.252
5.213
5.175
3.413
1.837
1.808
1.785
1.568
1.543
1.345
1.332
0.998
0.972
0.945
9.547
1.000
0.901
3.042
2.274
4.507
3.080
ppm


CN
Ph
N
H
Ph
hexanal
143.59
141.78
128.99
128.84
127.85
127.69
127.60
127.28
120.45
78.14
77.62
77.11
65.66
48.53
33.73
31.34
25.43
22.59
14.20
ppm

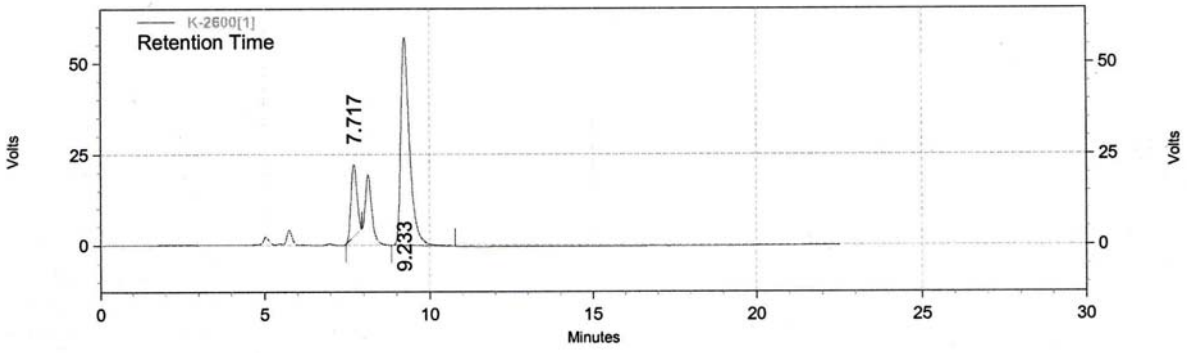

hexanal
CN
Ph
N
H
Ph
143.59
141.78
128.99
128.84
127.85
127.69
127.60
127.28
120.45
145
140
135
130
125
120
115
ppm





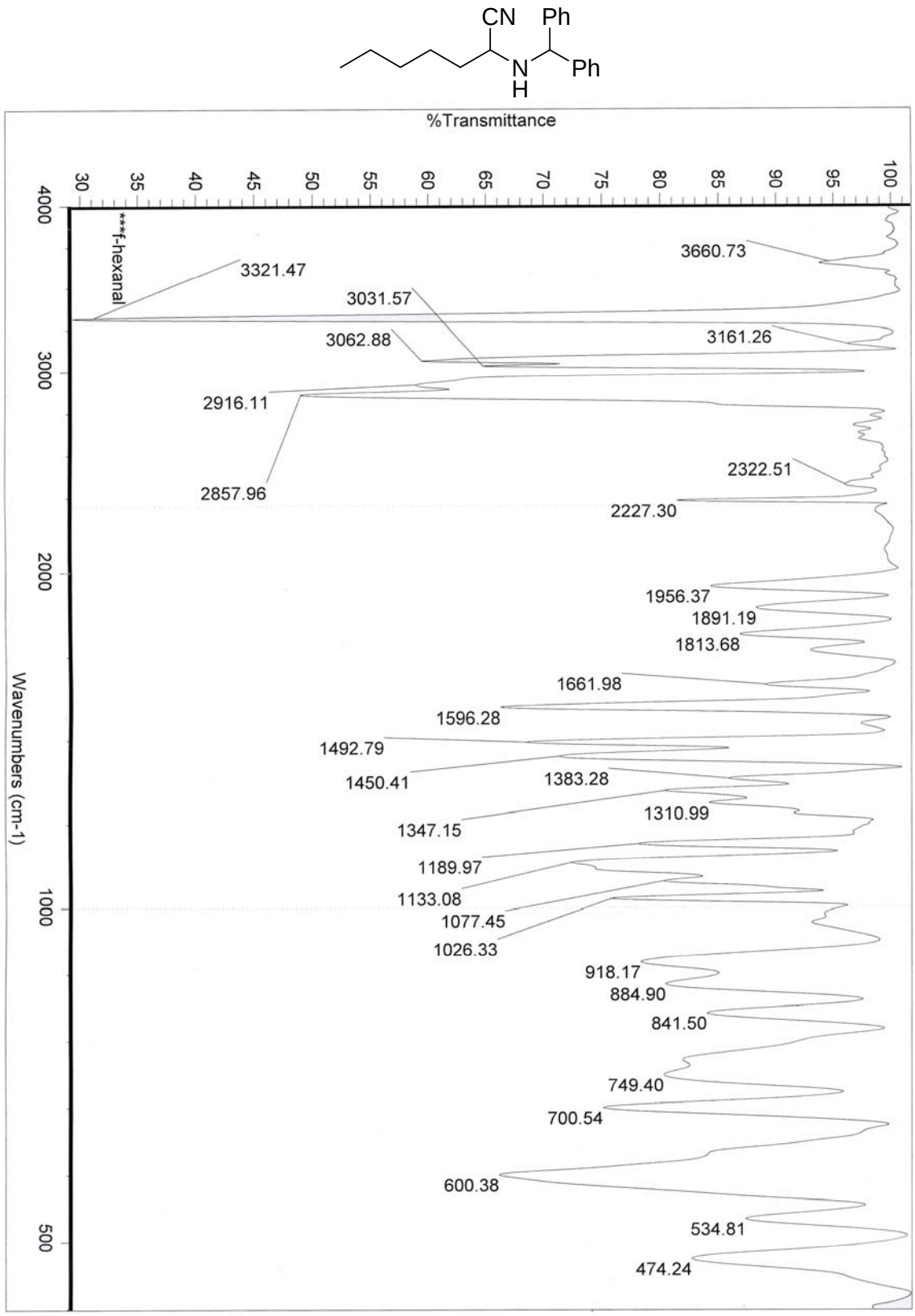
K-2600[1] Results

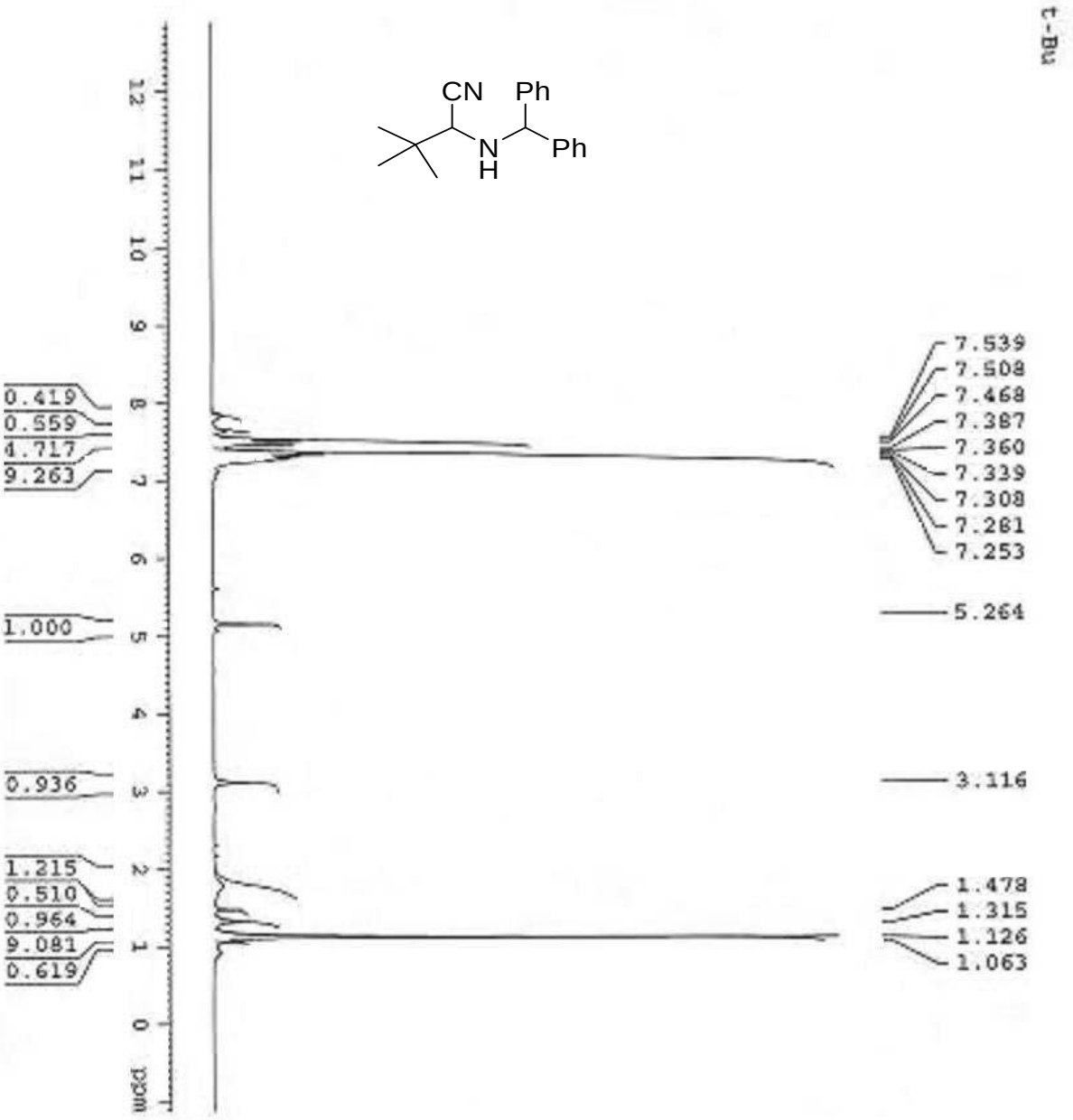
Retention Time	Area	Area %	Height	Height %
7.350	2363905	50.03	169332	54.85
8.683	2360789	49.97	139377	45.15
Totals	4724694	100.00	308709	100.00

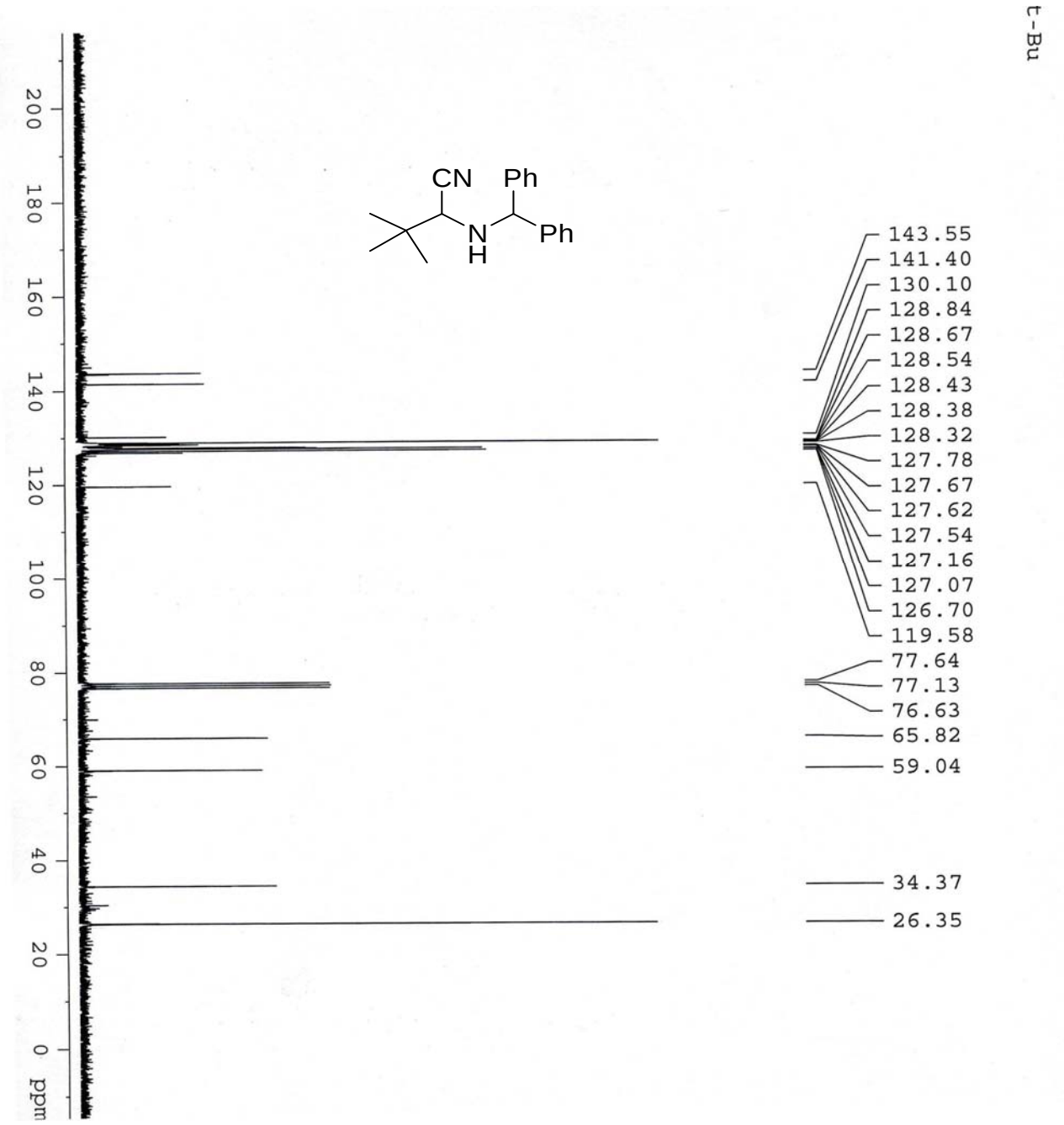


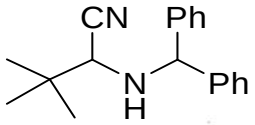
K-2600[1] Results

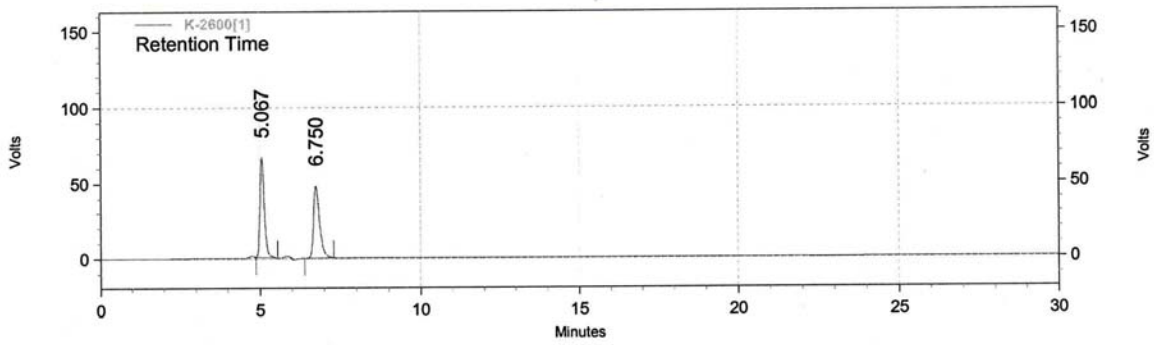
Retention Time	Area	Area %	Height	Height %
7.717	248770	19.02	20035	25.97
9.233	1059477	80.98	57101	74.03
Totals	1308247	100.00	77136	100.00


CN
Ph
N
H
Ph
%Transmittance
30
35
40
45
50
55
60
65
70
75
80
85
90
95
100
4000
3000
2000
1000
500
Wavenumbers (cm-1)
***f-hexanal
3660.73
3321.47
3031.57
3062.88
3161.26
2916.11
2857.96
2322.51
2227.30
1956.37
1891.19
1813.68
1661.98
1596.28
1492.79
1450.41
1383.28
1310.99
1347.15
1189.97
1133.08
1077.45
1026.33
918.17
884.90
841.50
749.40
700.54
600.38
534.81
474.24


CN
Ph
N
H
Ph
t-Bu
7.539
7.508
7.468
7.387
7.360
7.339
7.308
7.261
7.253
5.264
3.116
1.478
1.315
1.126
1.063
0.419
0.559
4.717
9.263
1.000
0.936
1.215
0.510
0.964
9.081
0.619
ppm

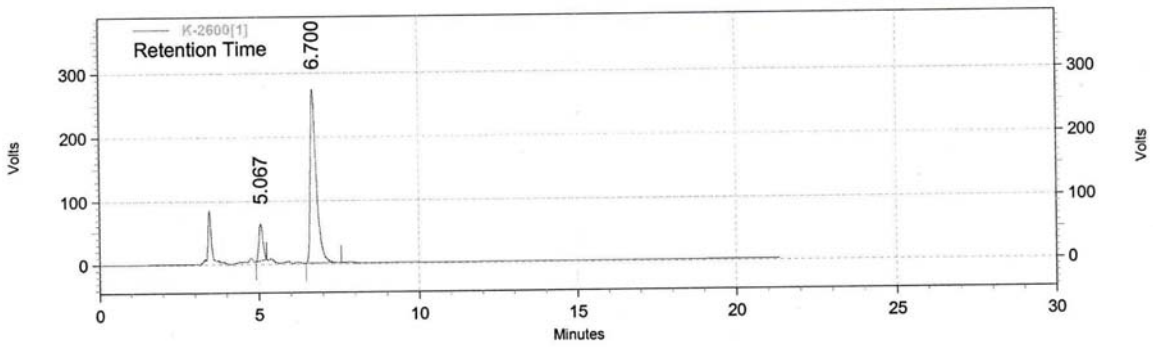

t-Bu
CN
Ph
N
H
Ph
143.55
141.40
130.10
128.84
128.67
128.54
128.43
128.38
128.32
127.78
127.67
127.62
127.54
127.16
127.07
126.70
119.58
77.64
77.13
76.63
65.82
59.04
34.37
26.35
200
180
160
140
120
100
80
60
40
20
0
ppm





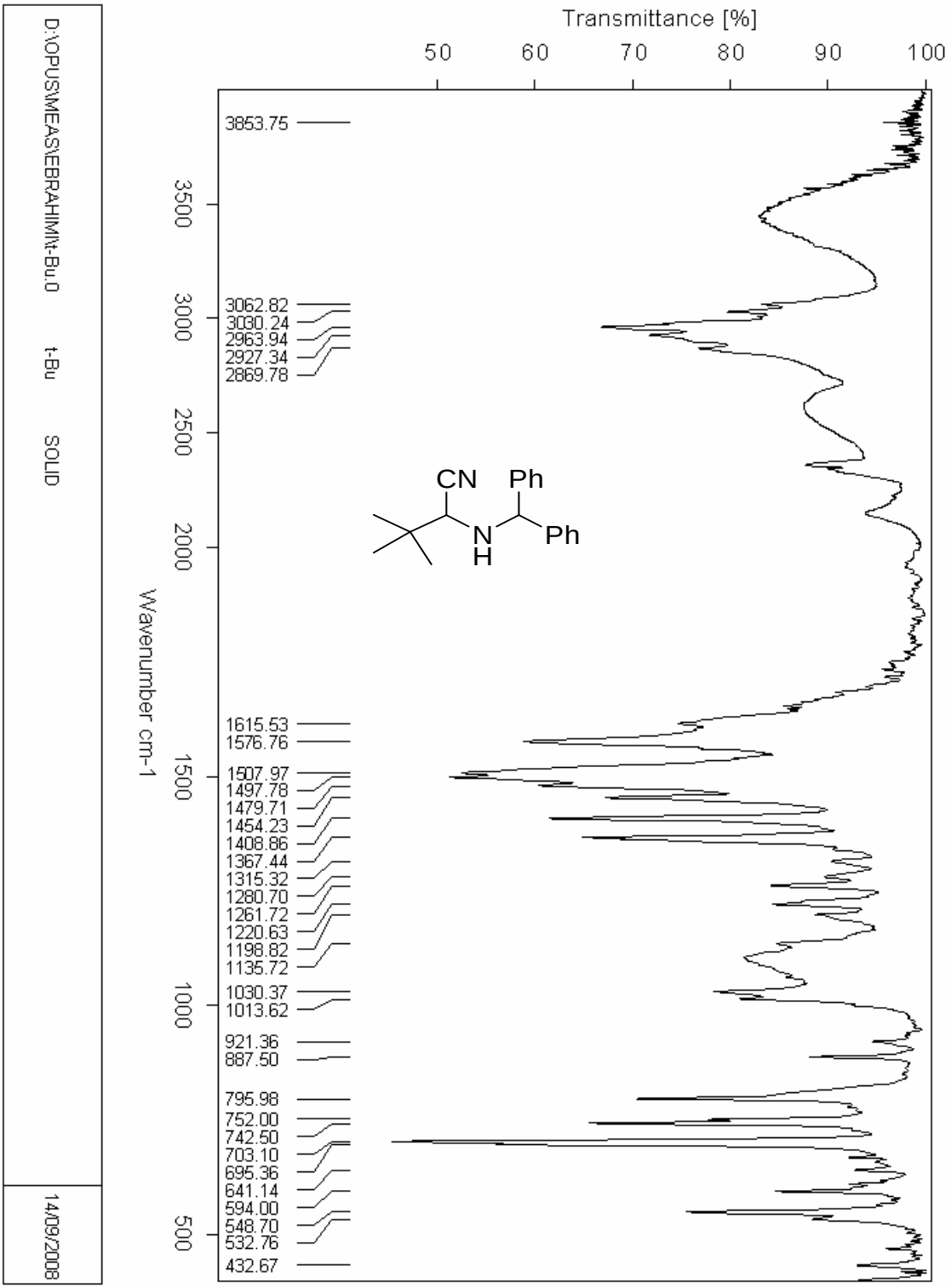
K-2600[1] Results

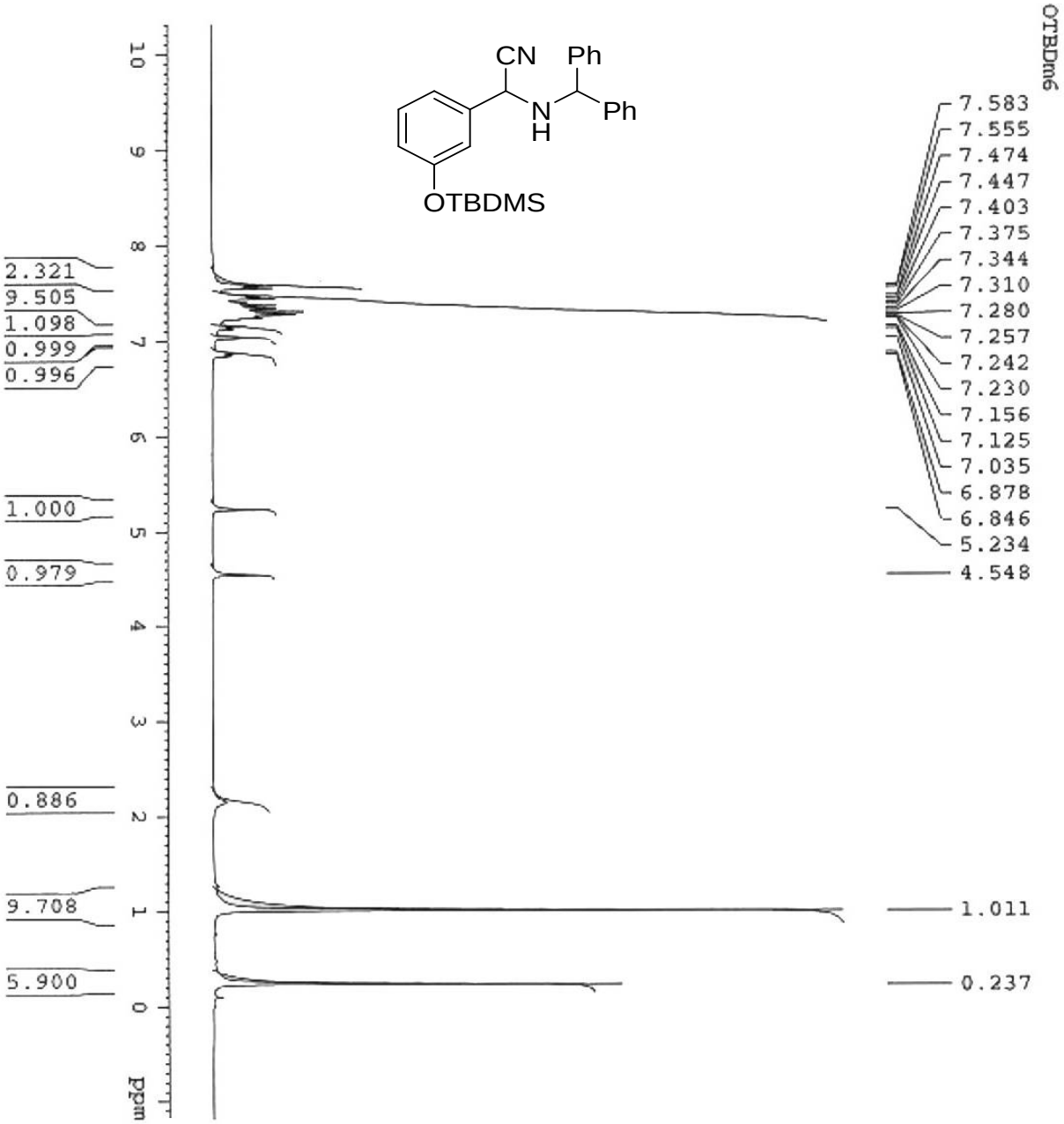
Retention Time	Area	Area %	Height	Height %
5.067	615701	49.60	66183	57.83
6.750	625555	50.40	48260	42.17
Totals	1241256	100.00	114443	100.00

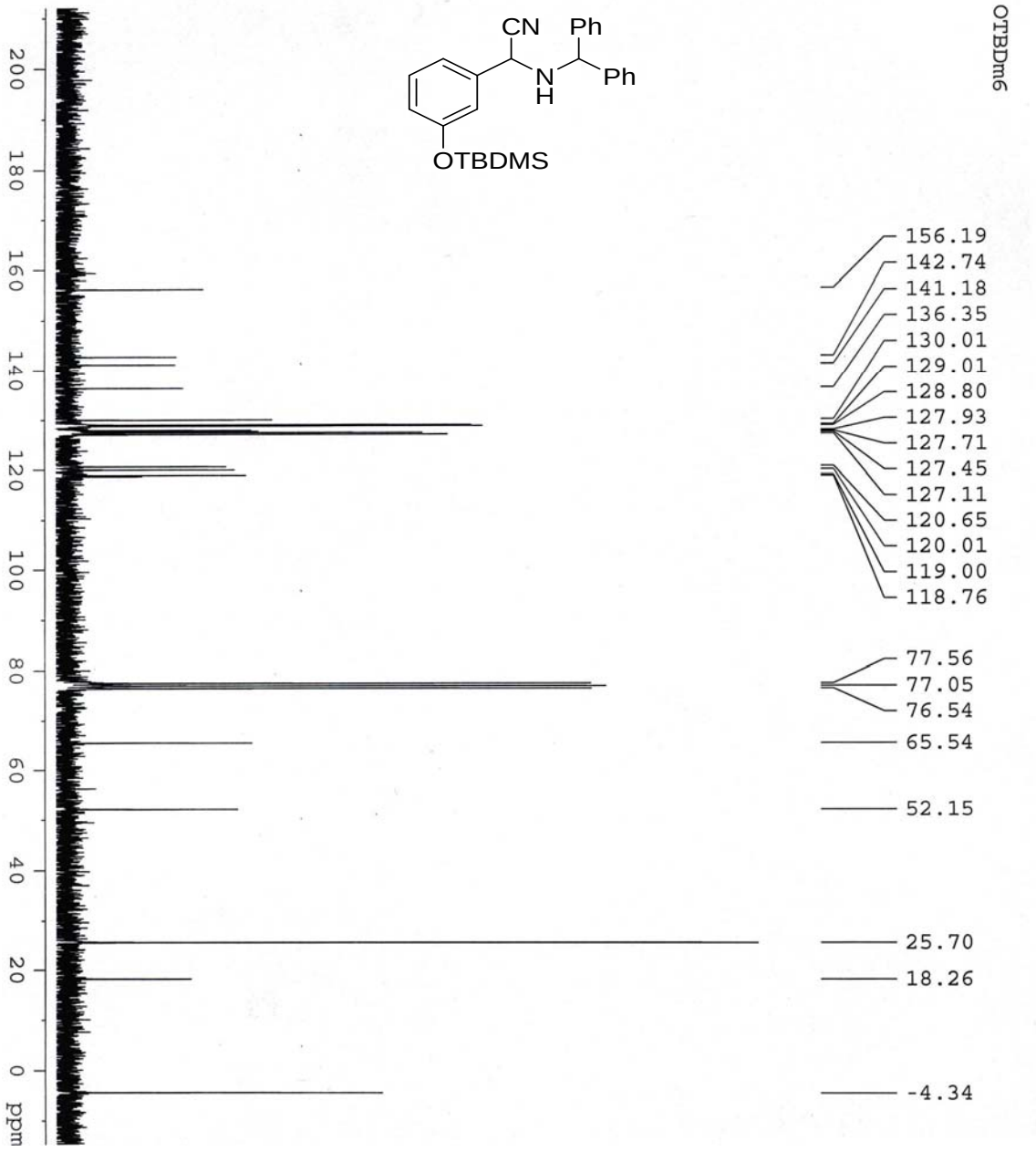


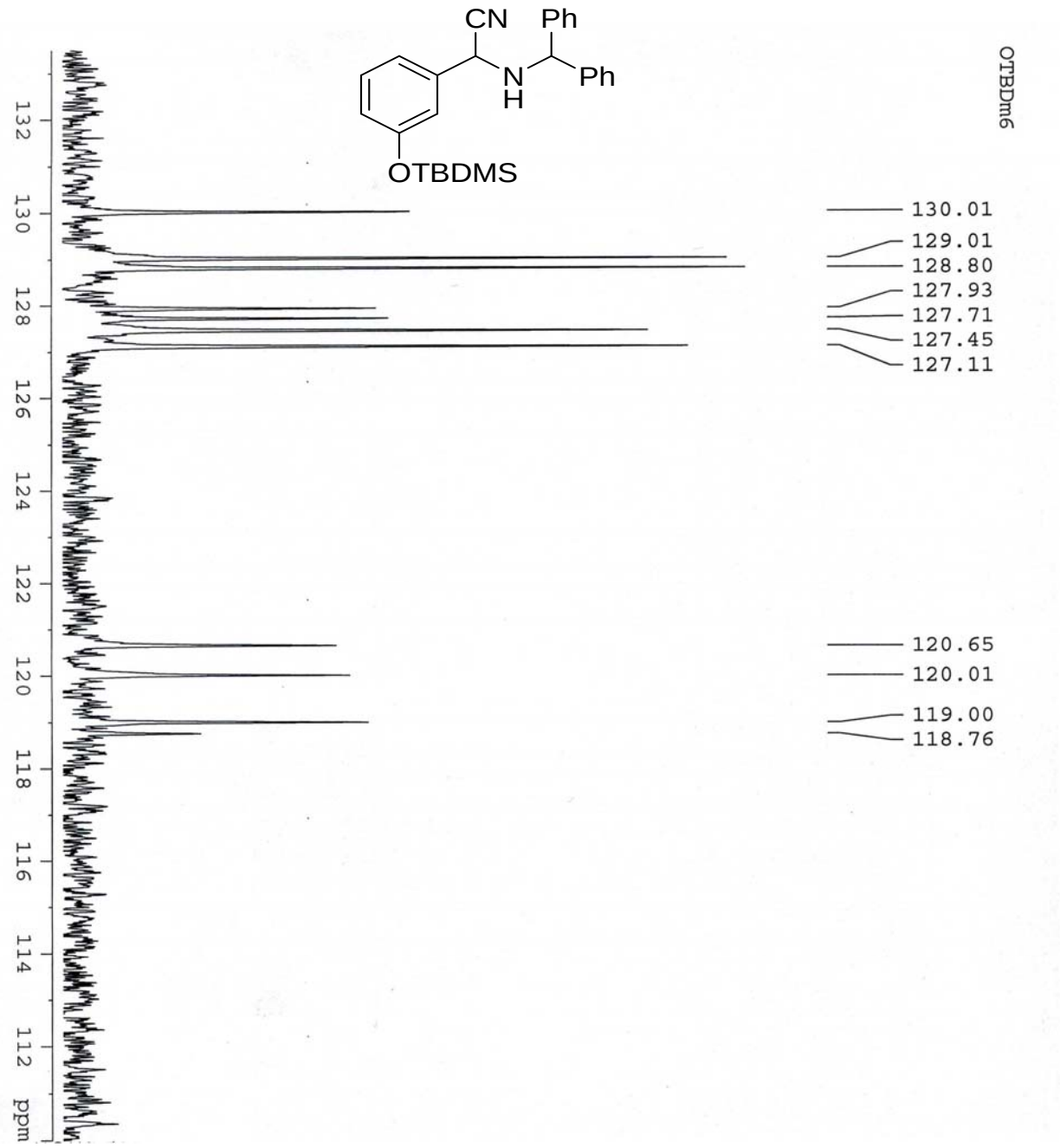
K-2600[1] Results

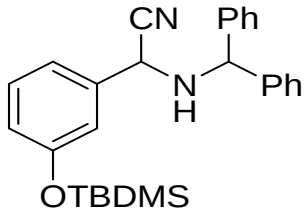
Retention Time	Area	Area %	Height	Height %
5.067	495535	12.15	58383	17.60
6.700	3582389	87.85	273369	82.40
Totals	4077924	100.00	331752	100.00

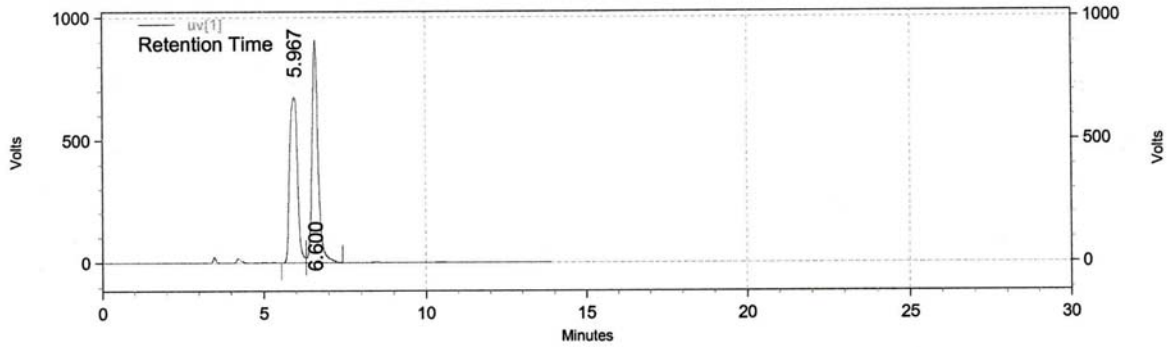

D:\OPUS\MEAS\EBRAHIM\t-Bu.0
t-Bu
SOLID
14/09/2008
Transmittance [%]
50
60
70
80
90
100
Wavenumber cm-1
3500
3000
2500
2000
1500
1000
500
3853.75
3062.82
3030.24
2963.94
2927.34
2869.78
1615.53
1576.76
1507.97
1497.78
1479.71
1454.23
1408.86
1367.44
1315.32
1280.70
1261.72
1220.63
1198.82
1135.72
1030.37
1013.62
921.36
887.50
795.98
752.00
742.50
703.10
695.36
641.14
594.00
548.70
532.76
432.67
CN
Ph
N
H
Ph


CN
Ph
N
H
Ph
OTBDMS
OTBDm6
7.583
7.555
7.474
7.447
7.403
7.375
7.344
7.310
7.280
7.257
7.242
7.230
7.156
7.125
7.035
6.878
6.846
5.234
4.548
1.011
0.237
2.321
9.505
1.098
0.999
0.996
1.000
0.979
0.886
9.708
5.900
ppm


CN
Ph
N
H
Ph
OTBDMS
OTBDm6
156.19
142.74
141.18
136.35
130.01
129.01
128.80
127.93
127.71
127.45
127.11
120.65
120.01
119.00
118.76
77.56
77.05
76.54
65.54
52.15
25.70
18.26
-4.34
200
180
160
140
120
100
80
60
40
20
0
ppm

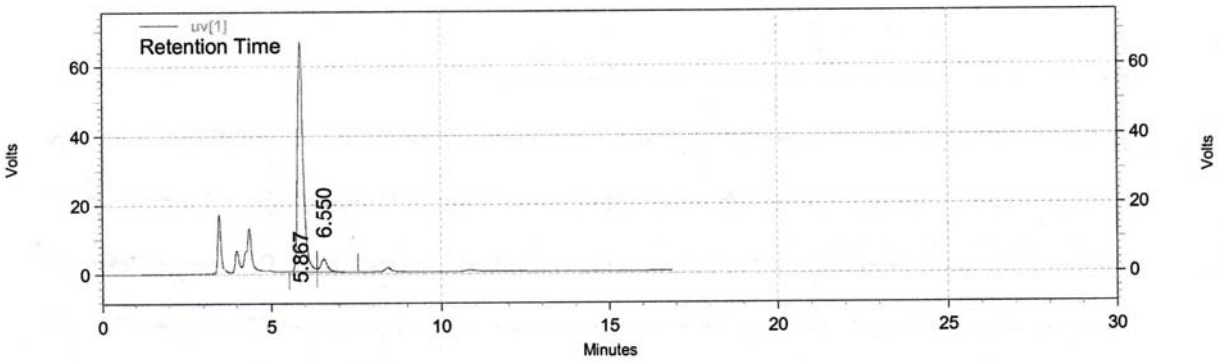

CN
Ph
N
H
Ph
OTBDMS
OTBDm6
130.01
129.01
128.80
127.93
127.71
127.45
127.11
120.65
120.01
119.00
118.76
132
130
128
126
124
122
120
118
116
114
112
ppm





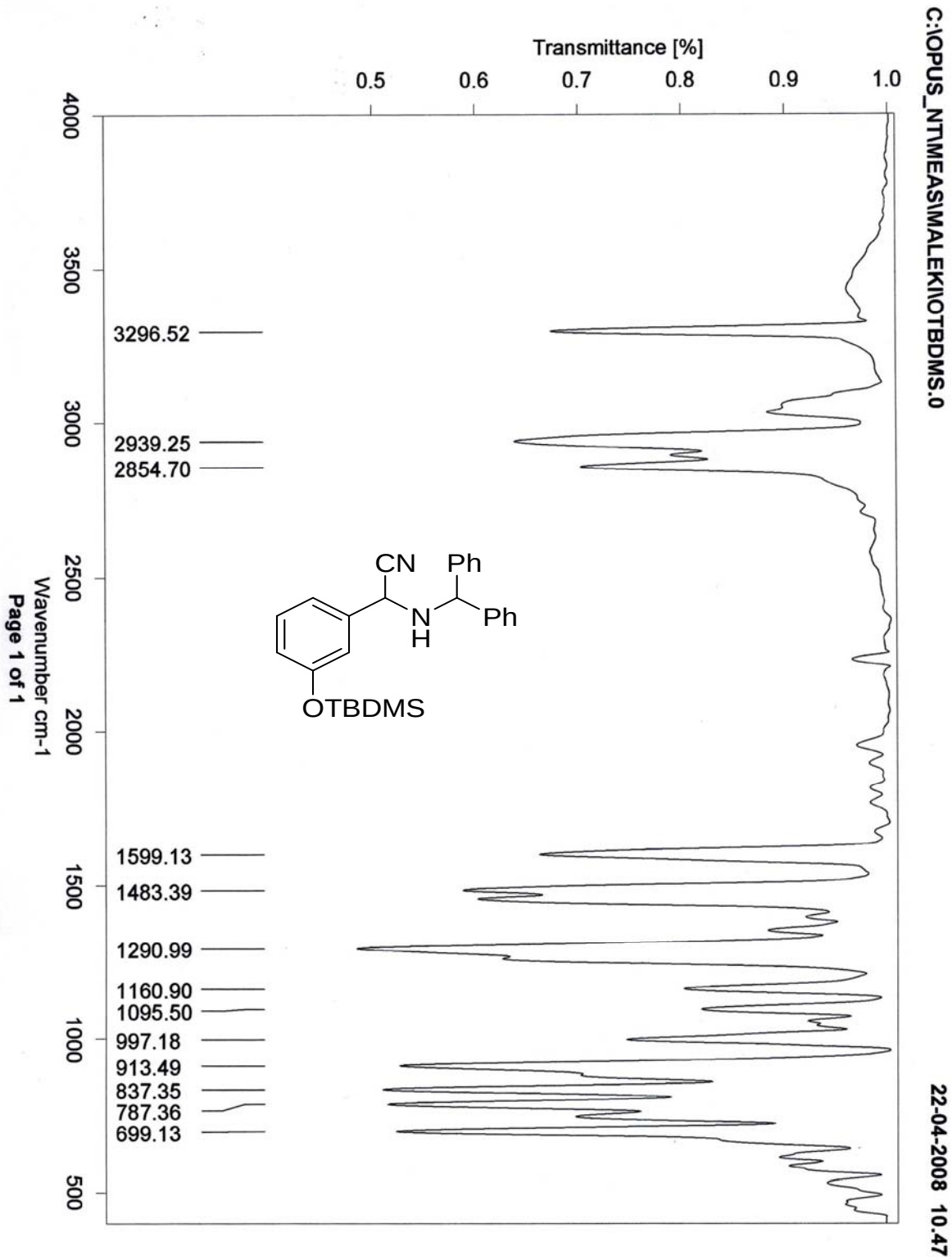
uv[1] Results

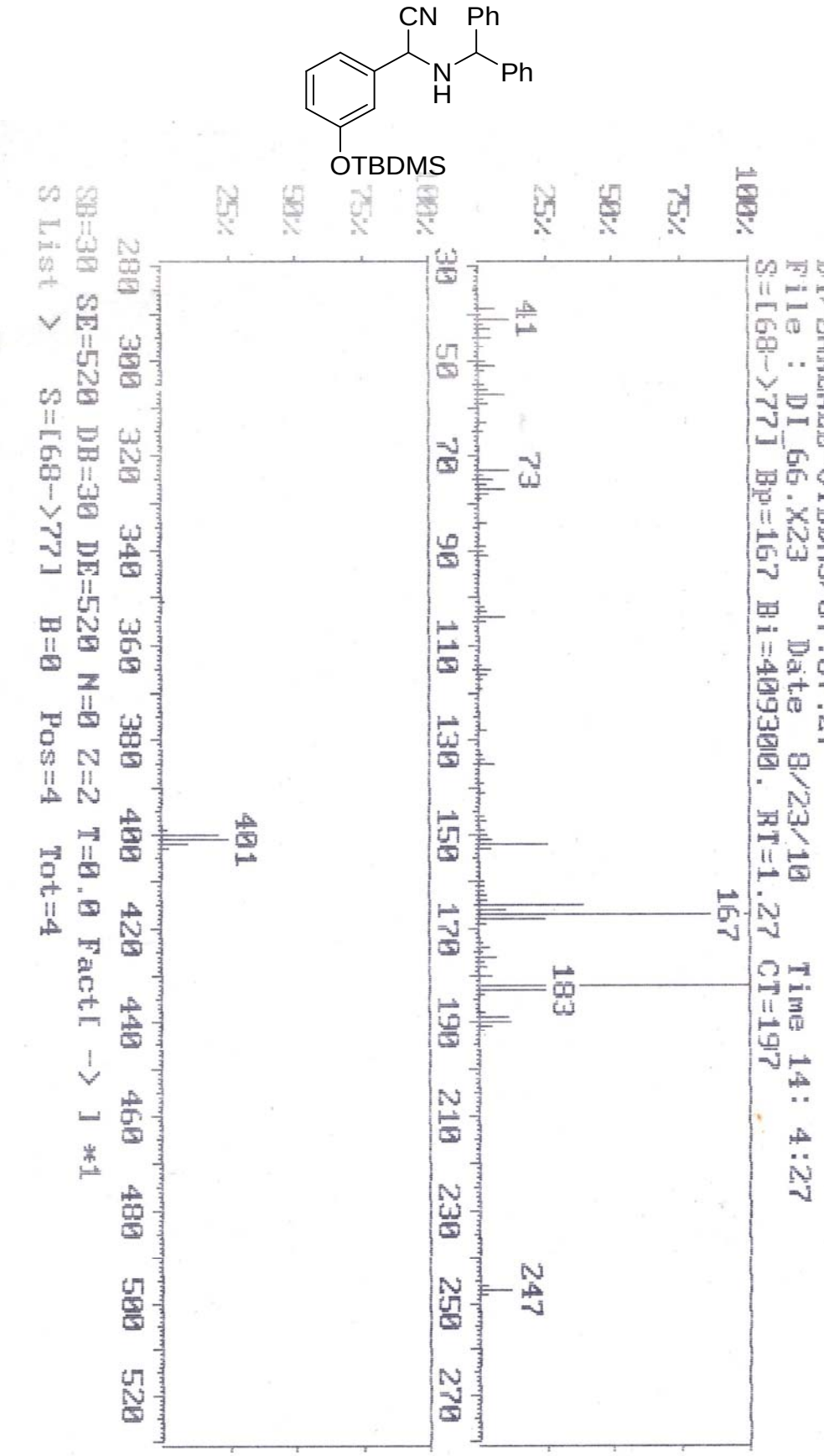
Retention Time	Area	Area %	Height	Height %
5.967	11315805	49.35	674347	42.60
6.600	11613978	50.65	908739	57.40
Totals	22929783	100.00	1583086	100.00

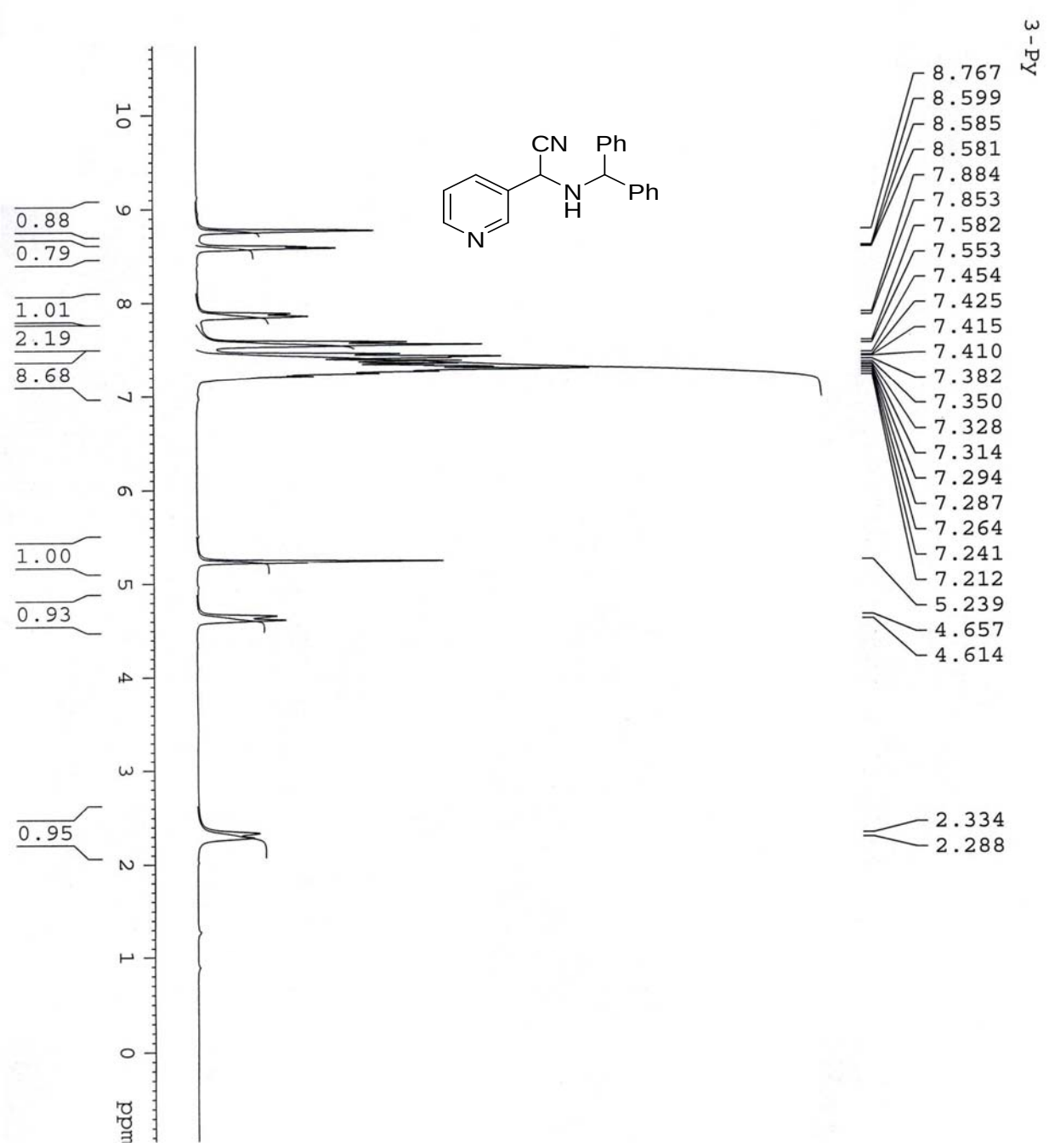


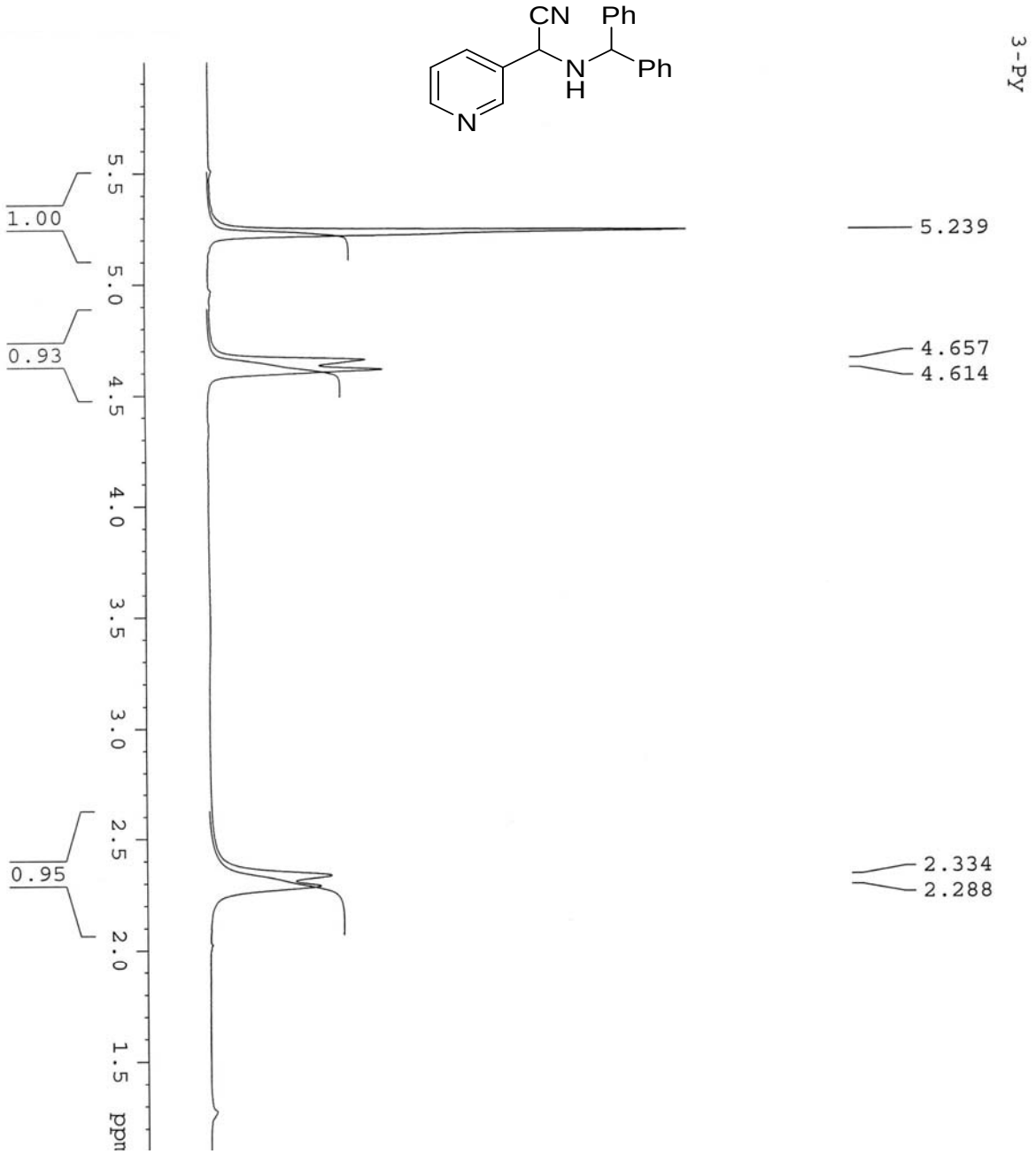
uv[1] Results

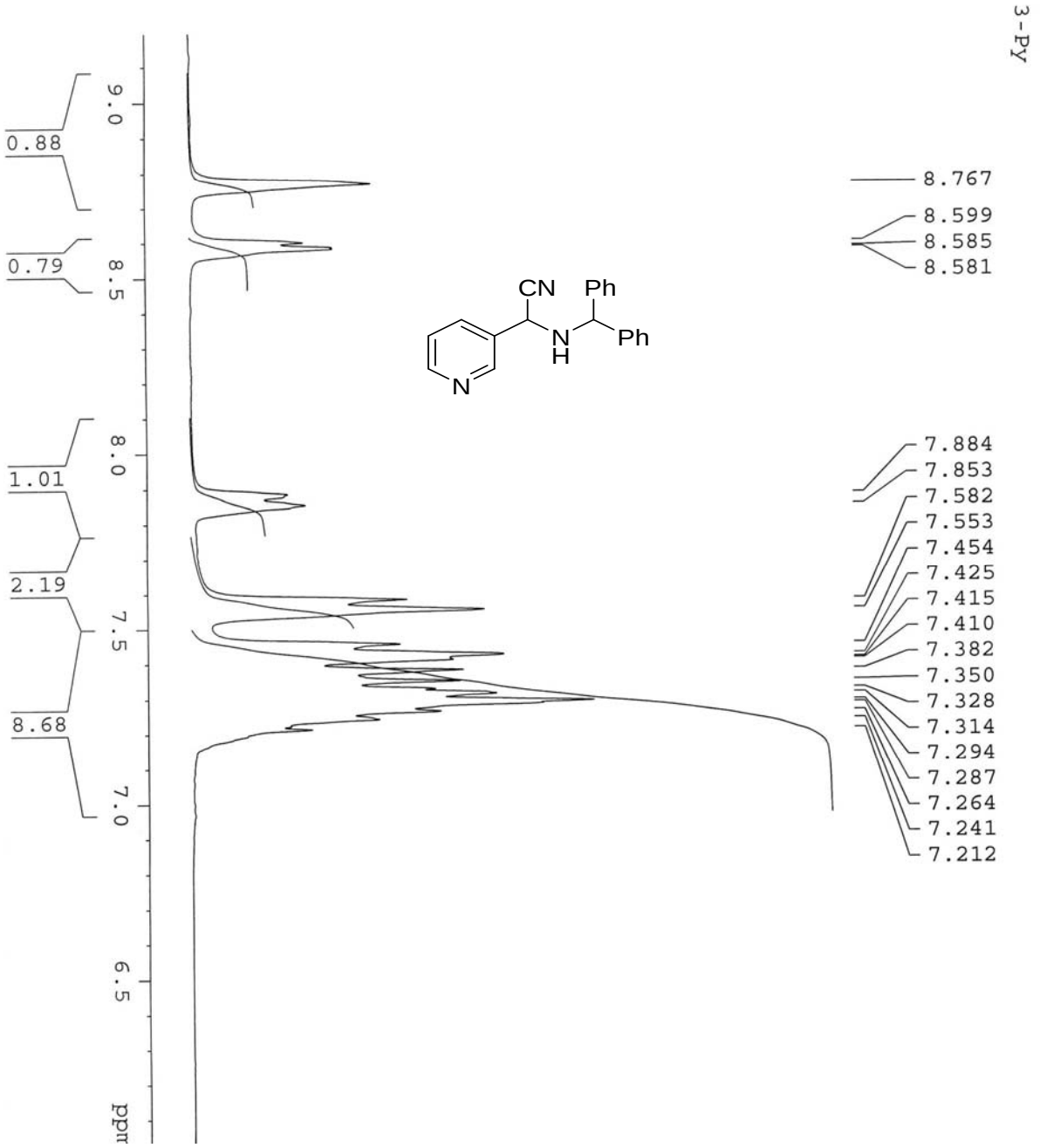
Retention Time	Area	Area %	Height	Height %
5.867	762098	93.42	66436	94.65
6.550	53688	6.58	3757	5.35
Totals	815786	100.00	70193	100.00

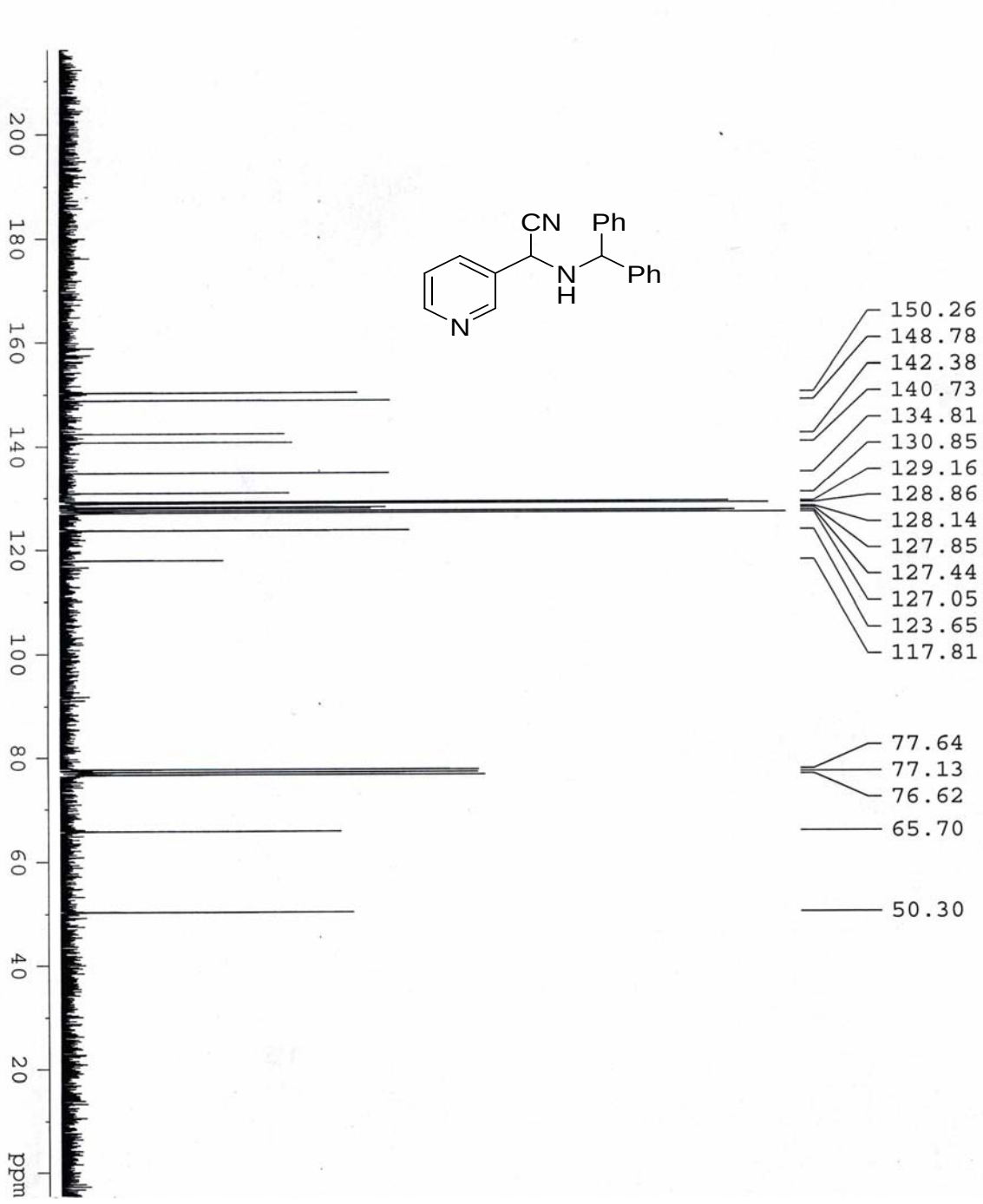

C:\OPUS_NT\MEAS\MALEK\OTBDMS.0
Transmittance [%]
0.5
0.6
0.7
0.8
0.9
1.0
4000
3500
3000
2500
2000
1500
1000
500
Wavenumber cm-1
Page 1 of 1
3296.52
2939.25
2854.70
1599.13
1483.39
1290.99
1160.90
1095.50
997.18
913.49
837.35
787.36
699.13
CN
Ph
N
H
Ph
OTBDMS
22-04-2008 10.47

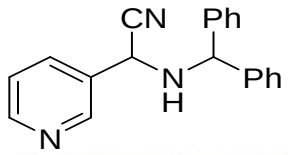

CN
Ph
N
H
Ph
OTBDMS
DI/ZAREAEE-OTBDMS/87.07.27
File : DI_66.X23 Date 8/23/10 Time 14: 4:27
S=[68->771 Bp=167 Bi=409300. RT=1.27 CT=197
100%
75%
50%
25%
00%
75%
50%
25%
41
73
167
183
247
401
30 50 70 90 110 130 150 170 190 210 230 250 270
280 300 320 340 360 380 400 420 440 460 480 500 520
SB=30 SE=520 DB=30 DE=520 N=0 Z=2 T=0.0 Fact[-> 1 *1
S List > S=[68->771 B=0 Pos=4 Tot=4

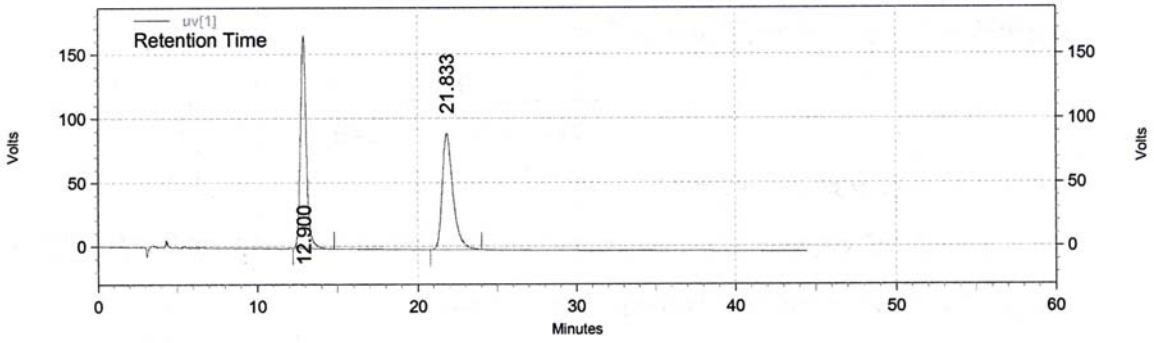

3-Py
8.767
8.599
8.585
8.581
7.884
7.853
7.582
7.553
7.454
7.425
7.415
7.410
7.382
7.350
7.328
7.314
7.294
7.287
7.264
7.241
7.212
5.239
4.657
4.614
2.334
2.288
0.88
0.79
1.01
2.19
8.68
1.00
0.93
0.95
CN
Ph
N
H
Ph
N
ppm


CN
Ph
N
H
Ph
N
3-Py
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
ppm
1.00
0.93
0.95
5.239
4.657
4.614
2.334
2.288


3-Py
8.767
8.599
8.585
8.581
7.884
7.853
7.582
7.553
7.454
7.425
7.415
7.410
7.382
7.350
7.328
7.314
7.294
7.287
7.264
7.241
7.212
CN
Ph
N
H
Ph
N
0.88
0.79
1.01
2.19
8.68
9.0
8.5
8.0
7.5
7.0
6.5
ppm

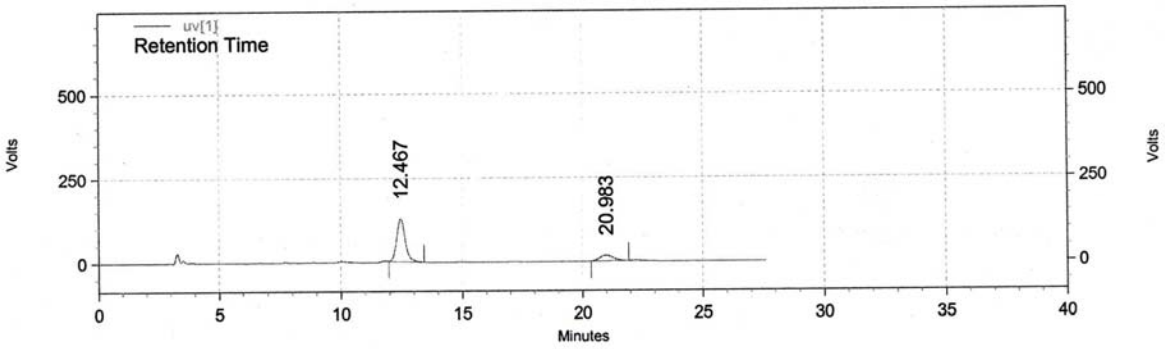

CN
Ph
N
H
Ph
N
150.26
148.78
142.38
140.73
134.81
130.85
129.16
128.86
128.14
127.85
127.44
127.05
123.65
117.81
77.64
77.13
76.62
65.70
50.30
200
180
160
140
120
100
80
60
40
20
ppm





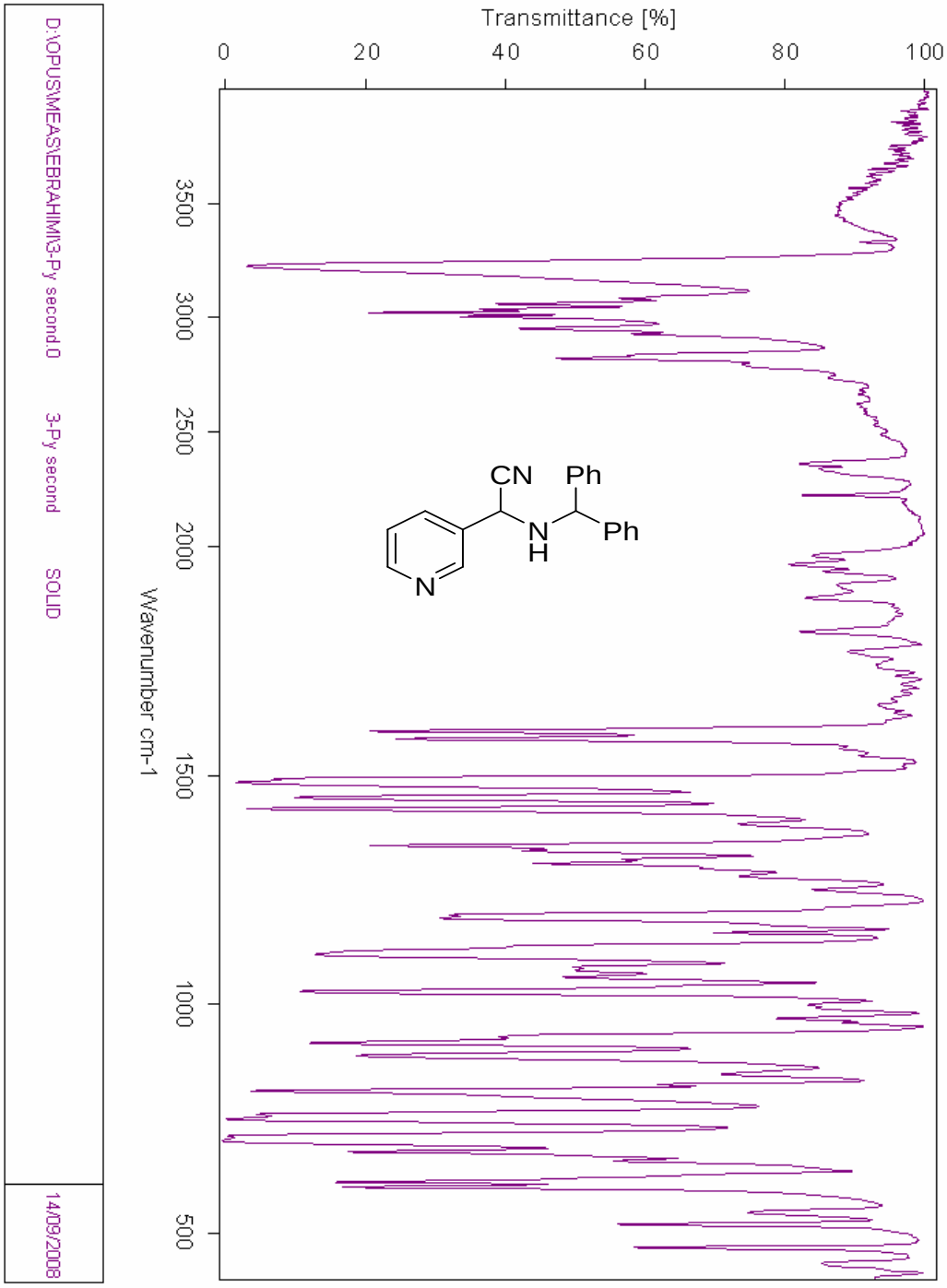
uv[1] Results

Retention Time	Area	Area %	Height	Height %
12.900	4237866	49.84	166116	64.57
21.833	4264401	50.16	91160	35.43
Totals	8502267	100.00	257276	100.00

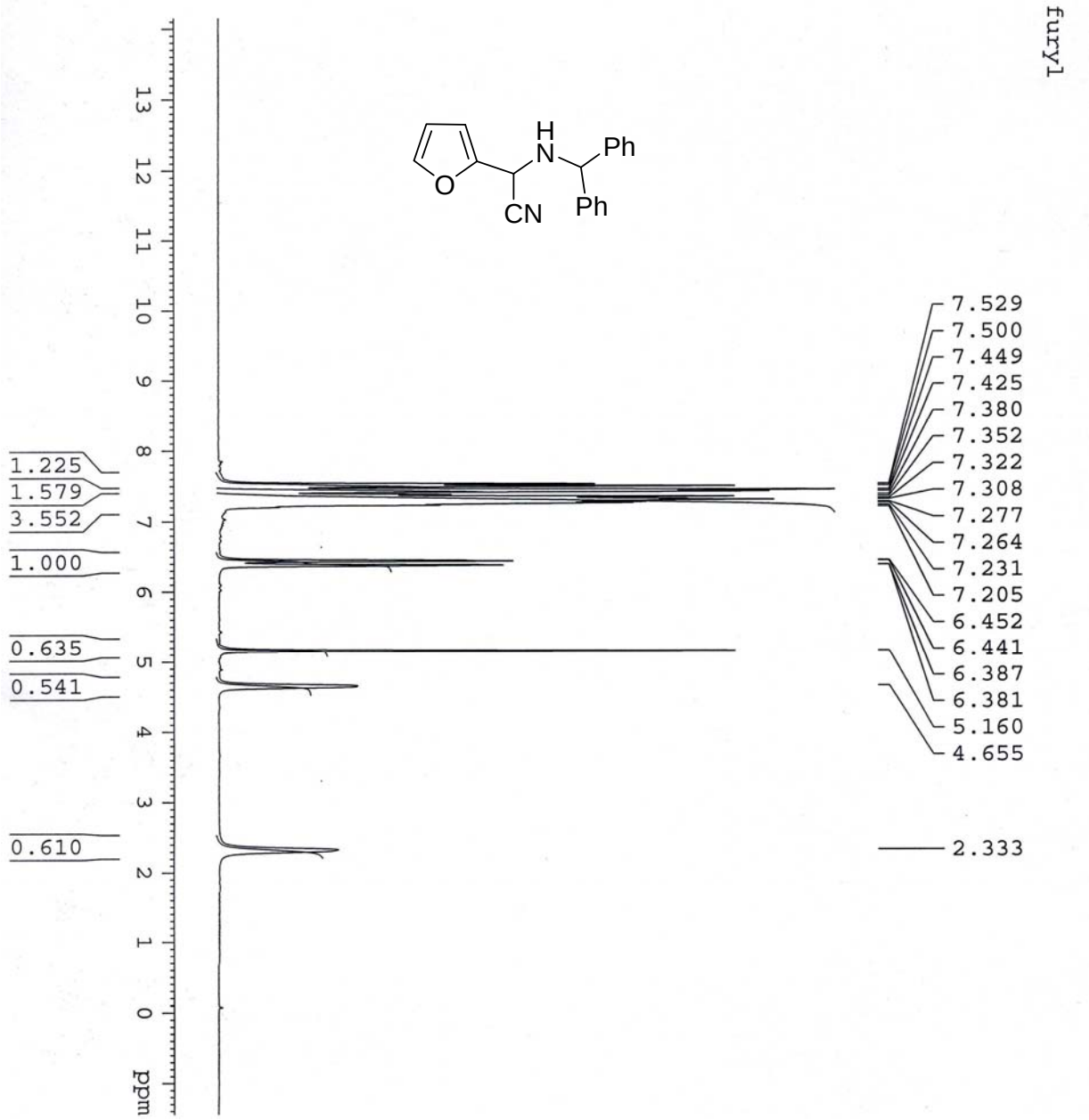


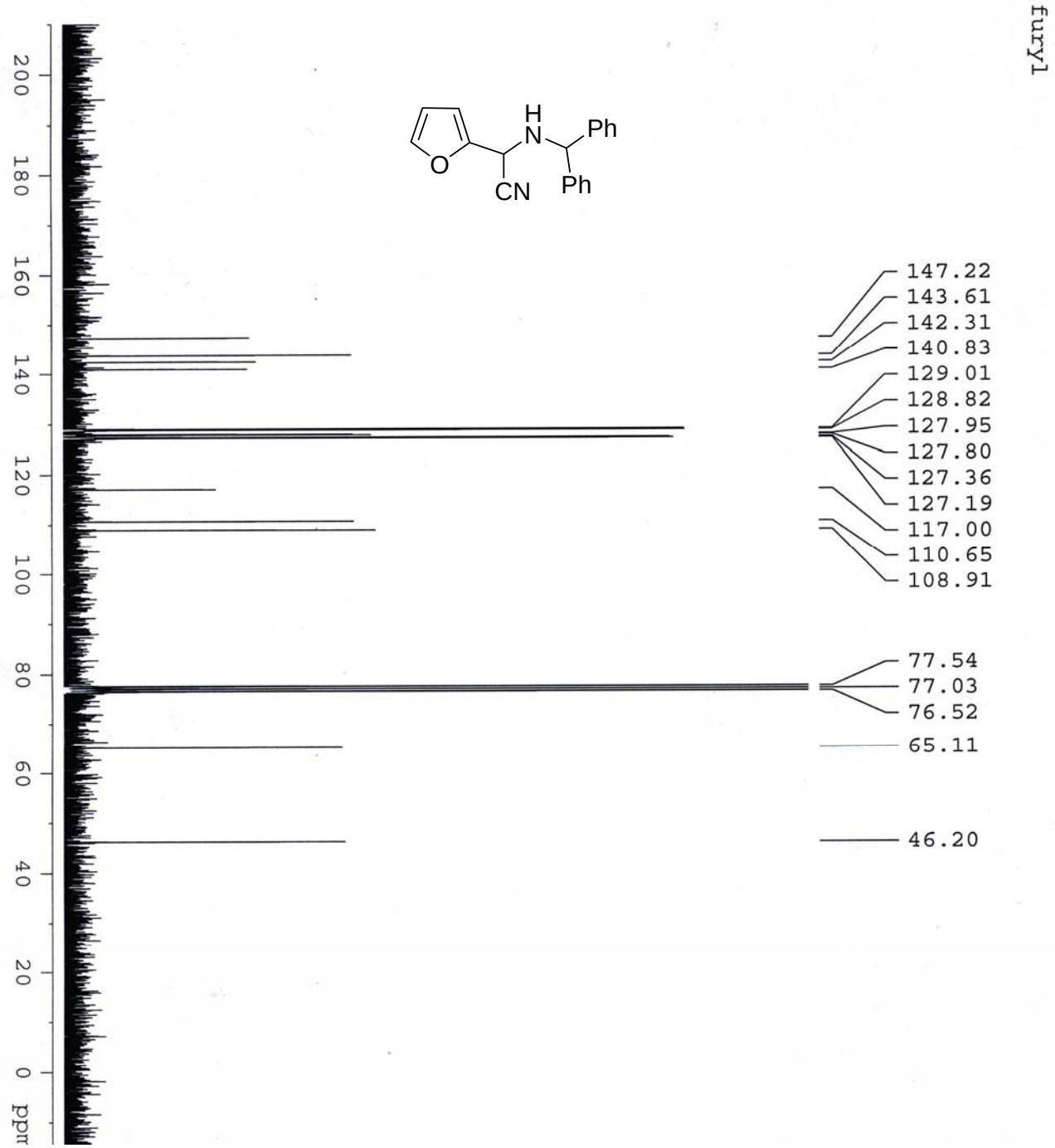
uv[1] Results

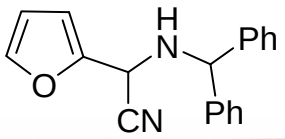
Retention Time	Area	Area %	Height	Height %
12.467	3024913	82.63	124961	88.32
20.983	635802	17.37	16522	11.68
Totals	3660715	100.00	141483	100.00

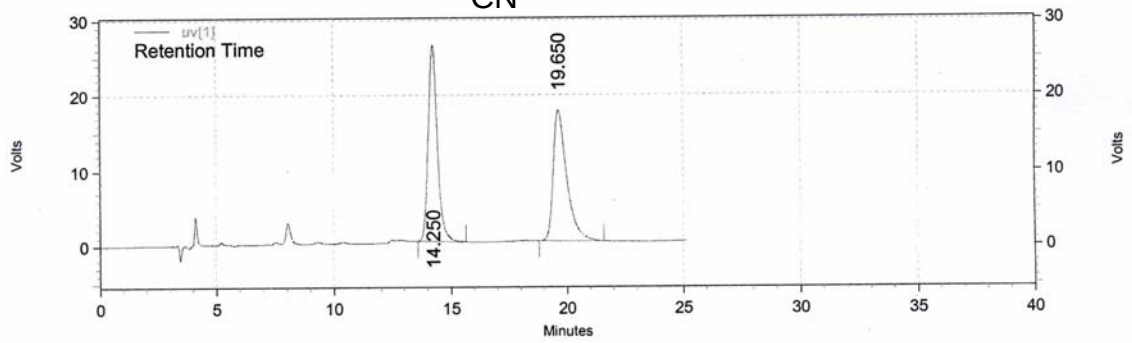

D:\OPUS\MEAS\EBRAHIMI\3-Py second.0
3-Py second
SOLID
14/09/2008
Transmittance [%]
0
20
40
60
80
100
Wavenumber cm-1
3500
3000
2500
2000
1500
1000
500
CN
Ph
N
H
Ph
N


CN
Ph
N
H
Ph
N
DI/2AREAE-3-PY/87.07.27
File : DI_66.X22 Date 8/23/10 Time 13:53:34
S=[63->72] Bp=167 Bi=329100. RT=1.19 CI=180
100%
75%
50%
25%
51
77
117
167
222
273
30 50 70 90 110 130 150 170 190 210 230 250 270
100%
75%
50%
25%
300
280 300 320 340 360 380 400 420 440 460 480 500 520
SB=30 SE=500 DB=30 DE=500 N=0 Z=2 T=0.0 Fact[->] *1
S List > S=[63->72] B=0 Pos=5 Tot=5


furyl
H
N
Ph
O
CN
Ph
7.529
7.500
7.449
7.425
7.380
7.352
7.322
7.308
7.277
7.264
7.231
7.205
6.452
6.441
6.387
6.381
5.160
4.655
2.333
1.225
1.579
3.552
1.000
0.635
0.541
0.610
13
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm

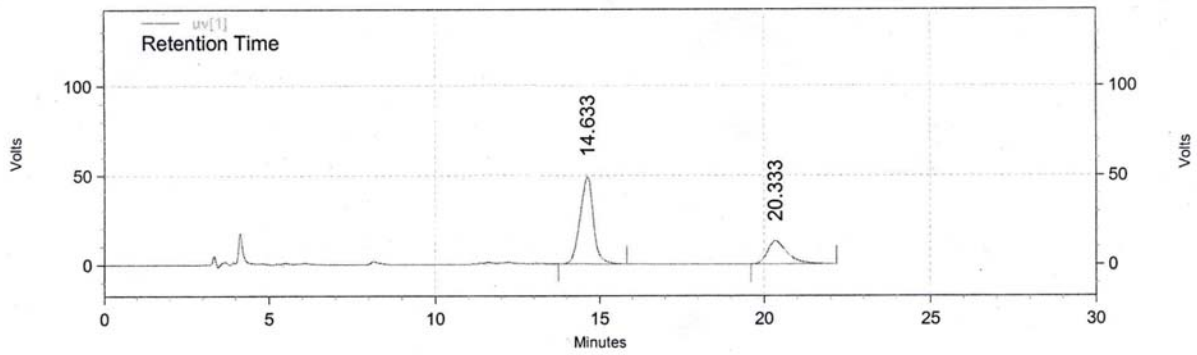

furyl
H
N
Ph
O
CN
Ph
147.22
143.61
142.31
140.83
129.01
128.82
127.95
127.80
127.36
127.19
117.00
110.65
108.91
77.54
77.03
76.52
65.11
46.20
200
180
160
140
120
100
80
60
40
20
0
ppm





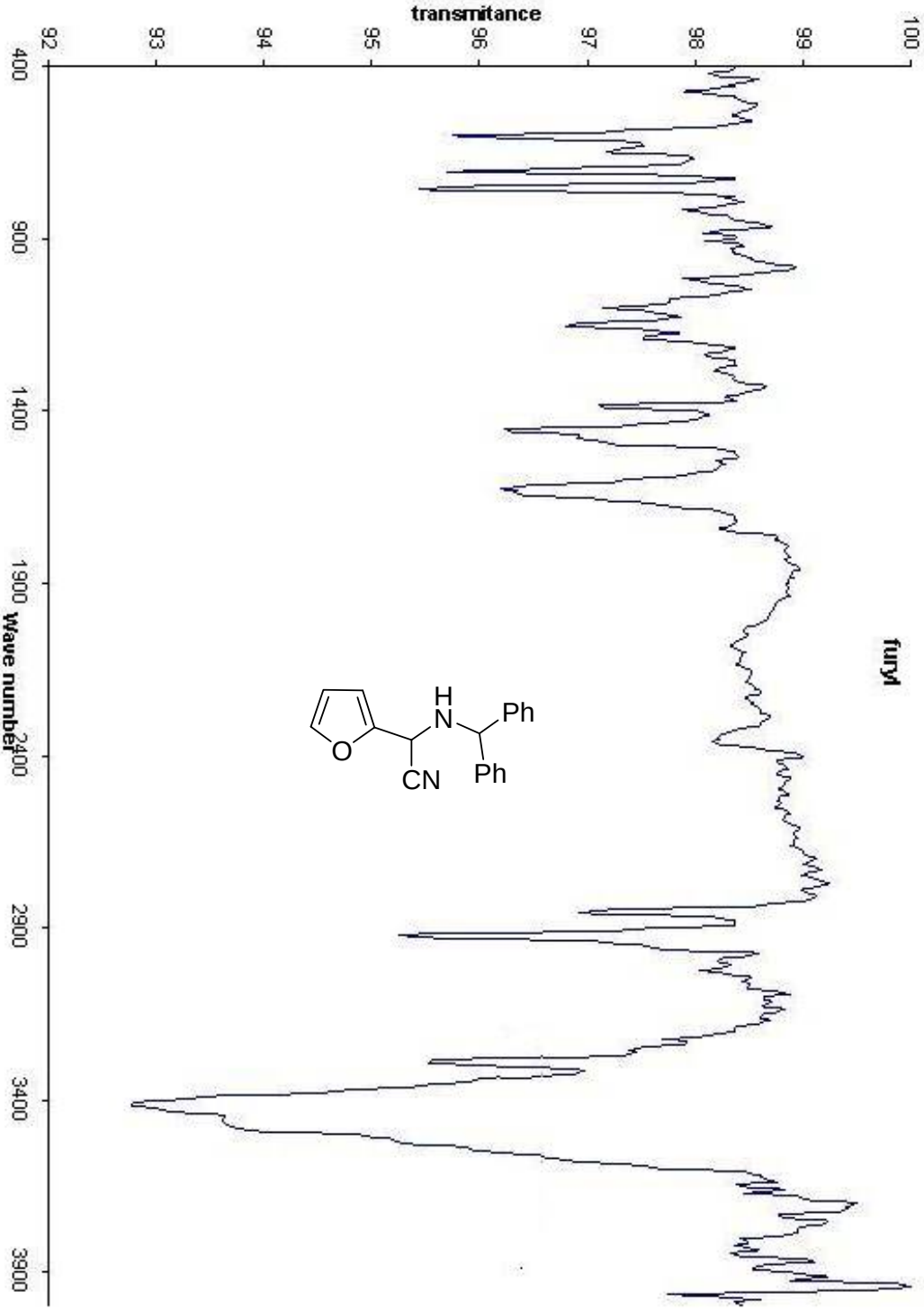
uv[1] Results

Retention Time	Area	Area %	Height	Height %
14.250	708787	50.12	26082	59.96
19.650	705511	49.88	17414	40.04
Totals	1414298	100.00	43496	100.00



uv[1] Results

Retention Time	Area	Area %	Height	Height %
14.633	1361550	72.25	48730	79.46
20.333	522991	27.75	12599	20.54
Totals	1884541	100.00	61329	100.00


transmitance
92
93
94
95
96
97
98
99
100
400
900
1400
1900
2400
2900
3400
3900
Wave number
furyl
H
N
Ph
Ph
O
CN