

Pd(quinoline-8-carboxylate)₂ as a low-priced, phosphine-free catalyst for Heck and Suzuki reactions

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Supporting Information

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General Information. All the Heck reactions and Suzuki reactions were carried out under Ar using magnetic stirring unless otherwise noted. DMF was distilled from CaH₂ and other solvents were purchased from commercial suppliers used without further purification. ¹H NMR spectra (300 MHz) were reported in ppm using tetramethylsilane as external standard. ¹³CNMR spectra were recorded on the same spectrometer (75.5 MHz) in proton decoupled mode.

(A) Typical Experimental Procedure and analytical data:

1. Experimental procedure for the ligands:

Ligand **3**, **5**, **6**, **7**, **8** are synthesized and characterized as following. Ligand **2** is well described in our previous work.¹ Others are used as purchased.

3-(dimethylamino)propanamide Hydrochloride (3·HCl)² Acrylamide (10mmol) was added to dimethylamine (15mmol) in 3ml methanol and the mixture stood for three days at room temperature. The mixture was evaporated to completely dryness, then the residue was dissolved in 20ml 2M HCl. 5x20ml ether was added to wash out acrylamide. Acetone was added to cause precipitation. The precipitate was filtered and dried under vacuum to afford 3-(dimethylamino)propanamide Hydrochloride as a white solid (0.69g, 45%). ¹H NMR (300MHz, D₂O): δ3.42 (t, 2H, *J* =6.75Hz), 2.91 (s, 6H), 2.81 (t, 2H, *J* =6.75Hz); ¹³C NMR (75MHz, D₂O, acetone): δ29.4, 43.1, 53.7, 174.3.

N-methylproline (5) Ligand **5** was synthesized exactly the same as previously reported.³ ¹H NMR (300MHz, D₂O): δ3.87-3.92 and 3.71-3.75 (2m, 1H), 3.14-3.18 (m, 1H), 2.94 (s, 3H), 2.48-2.52 (m, 1H), 2.00-2.23 (m, 3H); ¹³C NMR (75MHz, D₂O, acetone): δ22.9, 28.8, 40.8, 56.3, 70.7, 173.4.

3-(methyl(phenyl)amino)propanoic acid (6)⁴ 3-bromo-propanoic acid (10mmol), N-methylaniline (20mmol), NaOAc (20mmol) was refluxed for 5h in 30ml ethanol. Most of the ethanol was evaporated and the residue was poured into 100ml water and made pH=13 with NaOH. The aqueous solution was extracted with 5x30ml ether, then adjust to pH=5. After extraction with ether, the organic phase was concentrated and

recrystallized in boiling water to afford B₂ as a white solid (0.70g, 39%). ¹H NMR (300MHz, CDCl₃): δ7.23-7.29 (m, 2H), 6.76-6.80 (m, 2H), 3.66 (t, 2H, *J*=7.1Hz), 2.94 (s, 3H), 2.62 (t, 2H, *J*=7.1Hz); ¹³C NMR (75MHz, CDCl₃): δ31.4, 38.6, 48.9, 113.5, 117.9, 129.4, 148.3, 178.1.

3-(methoxy(methyl)amino)propanoic acid Hydrochloride (7·HCl) Ethyl acrylate (10mmol), N,O-dimethylhydroxylamine hydrochloride (11mmol), 8ml of Hunig's base was refluxed in 8ml of CH₃CN under Ar for 50h. After evaporation of the solvent, ether and aqueous NaOH were added. The organic phase was washed with 3M HCl. The combined aqueous layer was made basic and extracted with ether. After evaporation of ether, the residue was heated in 2M NaOH at 60 °C for 4h. The aqueous solution was washed with ether, made neutral and evaporated to completely dryness. The residue was extracted by dry ethanol, After filtration of NaCl, ethanol was evaporated and acidified with HCl. Acetone was added to cause precipitation. The precipitate was filtered and dried under vacuum to afford 3-(methoxy(methyl)amino)propanoic acid Hydrochloride as a white solid (0.31g, 18%). ¹H NMR (300MHz, D₂O): δ3.86 (s, 3H), 3.65 (m, 2H), 3.14 (s, 3H), 2.88 (m, 2H); ¹³C NMR (75MHz, D₂O, acetone): δ28.4, 42.4, 54.2, 60.3, 174.0. Elemental analysis calcd for C₅H₁₂NO₃Cl: C 35.39; H 7.08; N 8.26. Found: C 35.35; H 7.16; N 8.17.

2-(dimethylamino)benzoic acid (8)⁵ Anthranilic acid (10mmol) in an aqueous alcohol was added (CH₃)₂SO₄ (20mmol) dropwise and refluxed for 3h. 2g of NaOH in water was added and the mixture was refluxed for another 1h. Alcohol was evaporated

and water was added, then the solution was adjusted to pH=5-6. The precipitation was extracted by ether, the organic phase was dried over Na₂SO₄ and the residue was purified by column chromatography to afford B₆ (0.66g, 40%). ¹H NMR (300MHz, DMSO-d⁶): δ7.77 (d, 1H, *J* =7.7Hz), 7.38 (t, 1H, *J* =8.4Hz), 6.68 (d, 1H, *J* =8.7Hz), 6.55 (t, 1H, *J* =7.5Hz), 3.37 (s, 6H); ¹³C NMR (75MHz, DMSO-d⁶): δ29.2, 110.1, 110.8, 114.1, 131.8, 134.7, 151.9, 170.1.

2. Preparation of the Pd complexes: Pd(N, N-dimethyl-β-alaninate)₂ (16), Pd(2-pyridylacetate)₂ (17), Pd(quinoline-8-carboxylate)₂ (18).

Pd(N, N-dimethyl-β-alaninate)₂ (16) This complex and its single crystal is synthesized and characterized in our previous work.⁶ ¹H NMR (300MHz, CDCl₃): δ2.45 (t, 4H, *J* =6.0Hz), 2.55-2.57 (m, 16H); ¹³C NMR (75MHz, CDCl₃): δ34.5, 48.9, 60.1, 175.9.

Pd(2-pyridylacetate)₂ (17) A mixture of 2 equiv. of sodium 2-pyridylacetate and 1 equiv. of K₂PdCl₄ was stirred in water at room temperature for 10 minutes. The precipitation was filtered and dried under vacuum to afford a yellow solid which has very poor solubility in the common solvents (56%). Elemental analysis calcd for C₁₄H₁₂N₂O₄Pd: C 44.44; H 3.17; N 7.41 (O 16.93, Pd 28.04). Found: C 43.86; H 3.20; N 7.39. IR (cm⁻¹): 767.4, 1312.0, 1487.7, 1639.7.

Pd(quinoline-8-carboxylate)₂ (18) A mixture of 2 equiv. of sodium quinoline-8-carboxylate and 1 equiv. of K₂PdCl₄ was stirred in water at room temperature for 10 minutes. The precipitation was filtered and dried under vacuum to afford a yellow solid which has very poor solubility in the common solvents (69%).

Elemental analysis calcd for C₂₀H₁₂N₂O₄Pd: C 53.33; H 2.67; N 6.22 (O 14.22, Pd 23.56). Found: C 52.93; H 2.67; N 6.16. IR (cm⁻¹): 770.0, 903.4, 1302.3, 1330.0, 1509.6, 1570.0, 1649.0..

3. Typical experimental procedure for the Heck reaction: A mixture of phenyl bromide (5mmol), styrene (7.5mmol), compound 18 (0.5x10⁻³mmol), K₂CO₃ (10mmol) in 10ml dry DMF was stirred under Ar at 130°C for the desired time. After the mixture was washed by water, extracted by ether and evaporated, the residue was purified by flash column chromatography to afford the desired product. The analytical data of all the coupling products matches our previous work.¹

4. Typical experimental procedure for the Suzuki reaction: A mixture of p-methylphenyl bromide (5mmol), phenyl boronic acid(7.5mmol), compound 18 (0.5x10⁻³mmol), K₃PO₄ (10mmol) in methanol/H₂O (7.5ml/7.5ml) was stirred at 50°C for the desired time. After the mixture was washed by water, extracted by ether and evaporated, the residue was purified by flash column chromatography to afford biphenyl the desired product. The analytical data of all the coupling products matches our previous work.⁶

5. Analytical data for the products of the Heck reaction

***trans*-stilbene¹:** ¹H NMR (400MHz, CDCl₃): δ7.11 (s, 2H), 7.24-7.28 (m, 2H), 7.35 (t, 4H, *J* =7.5Hz), 7.52 (d, 4H, *J* =7.7Hz);

***trans*-cinnamic acid *n*-butyl ester¹:** ¹H NMR (400MHz, CDCl₃): δ0.97 (t, 3H, *J* =7.3Hz), 1.41-1.47 (m, 2H), 1.66-1.71 (m, 2H), 4.21 (t, 2H, *J* =6.7Hz), 6.44 (d, 1H, *J* =16.0Hz), 7.37-7.39 (m, 3H), 7.51-7.54 (m, 2H), 7.68 (d, 1H, *J* =16.0Hz);

4-nitro-*trans*-stilbene¹: ¹H NMR (400MHz, CDCl₃): δ7.14 (d, 1H, *J* =16.0Hz), 7.27 (d, 1H, *J* =16.0Hz), 7.33-7.42 (m, 3H), 7.55 (d, 2H, *J* =7.8Hz), 7.63 (d, 2H, *J* =8.8Hz) 8.22 (d, 2H, *J* =8.8Hz);

4-nitro-*trans*-cinnamic acid *n*-butyl ester¹: ¹H NMR (400MHz, CDCl₃): δ 0.97 (t, 3H, *J* =7.4Hz), 1.42-1.49 (m, 2H), 1.67-1.74 (m, 2H), 4.24 (t, 2H, *J* =6.7Hz), 6.56 (d, 1H, *J* =16.0Hz), 7.66-7.72 (m, 3H), 8.25 (d, 2H, *J* =8.8Hz);

4-methyl-*trans*-stilbene¹: ¹H NMR (400MHz, CDCl₃): δ 2.36 (s, 3H), 7.07 (s, 2H), 7.17 (d, 2H, *J* =8.0Hz), 7.24-7.26 (m, 1H), 7.35 (t, 2H, *J* =7.5Hz), 7.41 (d, 2H, *J* =8.1Hz) 7.51 (d, 2H, *J* =7.9Hz);

4-methyl-*trans*-cinnamic acid *n*-butyl ester¹: ¹H NMR (400MHz, CDCl₃): δ 0.96 (t, 3H, *J* =7.4Hz), 1.41-1.48 (m, 2H), 1.65-1.72 (m, 2H), 4.20 (t, 2H, *J* =6.7Hz), 6.38 (d, 1H, *J* =16.0Hz), 7.18 (d, 2H, *J* =8.0Hz), 7.42 (d, 2H, *J* =8.0Hz), 7.65 (d, 1H, *J* =16.0Hz),

4-methoxy-*trans*-stilbene¹: ¹H NMR (400MHz, CDCl₃): δ 3.83 (s, 3H), 6.89 (d, 2H, *J* =8.8Hz), 6.98 (d, 1H, *J* =16.5Hz), 7.08 (d, 1H, *J* =16.4Hz), 7.25 (t, 1H, *J* =7.0Hz) 7.34 (t, 2H, *J* =7.5Hz) 7.44-7.50 (m, 4H);

4-methoxy-*trans*-cinnamic acid *n*-butyl ester¹: ¹H NMR (400MHz, CDCl₃): δ 0.96 (t, 3H, *J* =7.4Hz), 1.40-1.47 (m, 2H), 1.65-1.72 (m, 2H), 3.84 (s, 3H), 4.20 (t, 2H, *J* =6.7Hz), 6.30 (d, 1H, *J* =16.0Hz), 6.90 (d, 2H, *J* =8.8Hz), 7.47 (d, 2H, *J* =8.8Hz), 7.63 (d, 1H, *J* =16.0Hz),

4-formyl-*trans*-stilbene¹: ¹H NMR (400MHz, CDCl₃): δ 7.12 (d, 1H, *J* =16.2Hz), 7.25 (d, 1H, *J* =16.4Hz), 7.26-7.31 (m, 1H), 7.39 (t, 2H, *J* =5.0Hz), 7.54-7.56 (m, 2H), 7.65 (d, 2H, *J* =8.4Hz), 7.87 (d, 2H, *J* =8.4Hz), 10.00 (s, 1H);

4-formyl-*trans*-cinnamic acid *n*-butyl ester¹: ¹H NMR (400MHz, CDCl₃): δ 0.97 (t, 3H, *J* =7.4Hz), 1.41-1.47 (m, 2H), 1.67-1.74 (m, 2H), 4.23 (t, 2H, *J* =6.8Hz), 6.56 (d, 1H, *J* =16.0Hz), 7.67-7.73 (m, 3H), 7.90 (d, 2H, *J* =8.4Hz), 10.03 (s, 1H);

4-acetyl-*trans*-stilbene¹: ¹H NMR (400MHz, CDCl₃): δ 2.61 (s, 3H), 7.17 (d, 1H, *J* =16.0Hz), 7.22 (d, 1H, *J* =16.0Hz), 7.25-7.40 (m, 3H), 7.55 (d, 2H, *J* =8.0Hz), 7.59 (d, 2H, *J* =8.2Hz), 7.95 (d, 2H, *J* =8.2Hz);

4-acetyl-*trans*-cinnamic acid *n*-butyl ester¹: ¹H NMR (400MHz, CDCl₃): δ 0.97 (t, 3H, *J* =7.4Hz), 1.42-1.47 (m, 2H), 1.67-1.74 (m, 2H), 2.62 (s, 3H), 4.23 (t, 2H, *J* =6.6Hz), 6.53 (d, 1H, *J* =16.0Hz), 7.61 (d, 2H, *J* =8.3Hz), 7.69 (d, 1H, *J* =16.0Hz), 7.97 (d, 2H, *J* =8.3Hz);

***trans*-stilbene-3-carbonitrile¹:** ¹H NMR (400MHz, CDCl₃): δ 7.06 (d, 1H, *J* =16.4Hz), 7.14 (d, 1H, *J* =16.4Hz), 7.31-7.54 (m, 7H), 7.70 (m, 1H), 7.78 (s, 1H);

***trans*-3-(3-cyano-phenyl)-acrylic acid *n*-butyl ester¹:** ¹H NMR (400MHz, CDCl₃): δ

0.97 (t, 3H, $J = 7.4\text{Hz}$), 1.41-1.47 (m, 2H), 1.66-1.73 (m, 2H), 4.23 (t, 2H, $J = 6.8\text{Hz}$), 6.49 (d, 1H, $J = 16.0\text{Hz}$), 7.53 (t, 1H, $J = 7.6\text{Hz}$), 7.61-7.67 (m, 2H), 7.73-7.75 (m, 1H), 7.79 (m, 1H);

***trans*-stilbene-4-carbonitrile¹**: ¹H NMR (400MHz, CDCl₃): δ 7.10 (d, 1H, $J = 16.4\text{Hz}$), 7.20 (d, 1H, $J = 16.0\text{Hz}$), 7.32-7.41 (m, 3H), 7.52-7.65 (m, 6H);

***trans*-3-(4-cyano-phenyl)-acrylic acid *n*-butyl ester¹**: ¹H NMR (400MHz, CDCl₃): δ 0.97 (t, 3H, $J = 7.4\text{Hz}$), 1.41-1.47 (m, 2H), 1.66-1.72 (m, 2H), 4.23 (t, 2H, $J = 6.8\text{Hz}$), 6.52 (d, 1H, $J = 16.0\text{Hz}$), 7.60-7.69 (m, 5H);

4-phenyl-*trans*-stilbene¹: ¹H NMR (400MHz, CDCl₃): δ 7.15 (s, 2H), 7.25 (m, 1H), 7.35-7.45 (m, 5H), 7.52-7.63 (m, 8H);

4-phenyl-*trans*-cinnamic acid *n*-butyl ester¹: ¹H NMR (400MHz, CDCl₃): δ 0.97 (t, 3H, $J = 7.4\text{Hz}$), 1.42-1.48 (m, 2H), 1.67-1.74 (m, 2H), 4.22 (t, 2H, $J = 6.8\text{Hz}$), 6.47 (d, 1H, $J = 16.0\text{Hz}$), 7.37-7.38 (m, 1H), 7.43-7.47 (m, 2H), 7.59-7.62 (m, 6H), 7.72 (d, 1H, $J = 16.0\text{Hz}$);

3-trifluoromethyl-*trans*-stilbene¹: ¹H NMR (400MHz, CDCl₃): δ 7.13 (d, 1H, $J = 16.4\text{Hz}$), 7.16 (d, 1H, $J = 16.4\text{Hz}$), 7.30 (m, 1H), 7.36-7.40 (m, 2H), 7.46-7.54 (m, 4H), 7.66 (m, 1H), 7.75 (s, 1H);

3- trifluoromethyl-*trans*-cinnamic acid *n*-butyl ester¹: ¹H NMR (400MHz, CDCl₃): δ 0.97 (t, 3H, $J = 7.4\text{Hz}$), 1.40-1.49 (m, 2H), 1.6-1.73 (m, 2H), 4.23 (t, 2H, $J = 6.7\text{Hz}$), 6.51 (d, 1H, $J = 16.0\text{Hz}$), 7.52 (t, 1H, $J = 7.9\text{Hz}$), 7.62 (d, 1H, $J = 7.7\text{Hz}$), 7.69 (d, 1H, $J = 16.0\text{Hz}$), 7.70 (d, 1H, $J = 8.3\text{Hz}$), 7.77 (s, 1H);

3-*trans*-styryl-pyridine¹: ¹H NMR (400MHz, CDCl₃): δ 7.08 (d, 1H, $J = 16.4\text{Hz}$), 7.18 (d, 1H, $J = 16.4\text{Hz}$), 7.28-7.40 (m, 4H), 7.53 (m, 2H), 7.85 (d, 1H, $J = 8.0\text{Hz}$), 8.49 (m, 1H), 8.73 (s, 1H);

3-pyridin-3-yl-acrylic acid butyl ester¹: ¹H NMR (400MHz, CDCl₃): δ 0.97 (t, 3H, $J = 7.4\text{Hz}$), 1.42-1.47 (m, 2H), 1.67-1.72 (m, 2H), 4.23 (t, 2H, $J = 6.8\text{Hz}$), 6.52 (d, 1H, $J = 16.0\text{Hz}$), 7.32-7.35 (m, 1H), 7.67 (d, 1H, $J = 16.4\text{Hz}$); 7.84 (d, 1H, $J = 7.8\text{Hz}$), 8.60 (m, 1H), 8.76 (m, 1H);

4-Methylbiphenyl⁶: ¹H NMR (400MHz, CDCl₃): δ 2.39 (s, 3H), 7.25 (d, 2H, $J = 8.0\text{Hz}$), 7.33 (m, 1H), 7.42 (t, 2H, $J = 7.6\text{Hz}$); 7.49 (d, 2H, $J = 8.2\text{Hz}$), 7.57 (m, 2H).

4-Methoxybiphenyl⁶: ¹H NMR (400MHz, CDCl₃): δ 3.85 (s, 3H), 6.97 (m, 2H), 7.30 (m, 1H), 7.42 (t, 2H, $J = 7.9\text{Hz}$); 7.54 (m, 4H).

4-Acetylbiphenyl⁶: ¹H NMR (400MHz, CDCl₃): δ 2.64 (s, 3H), 7.42 (m, 1H), 7.47 (m,

2H), 7.63 (m, 2H), 7.68 (m, 2H), 8.03 (m, 2H).

4-Methoxy-4'-acetylbiphenyl⁶: ¹H NMR (400MHz, CDCl₃): δ 2.62 (s, 3H), 3.86 (s, 3H), 7.00 (d, 2H, *J* = 8.7 Hz), 7.57 (d, 2H, *J* = 8.7 Hz), 7.64 (d, 2H, *J* = 8.5 Hz), 8.00 (d, 2H, *J* = 8.4 Hz).

4-Methoxy-4'-nitrobiphenyl⁶: ¹H NMR (400MHz, CDCl₃): δ 3.87 (s, 3H), 7.01 (d, 2H, *J* = 8.8 Hz); 7.58 (d, 2H, *J* = 8.8 Hz), 7.68 (d, 2H, *J* = 8.8 Hz), 8.26 (d, 2H, *J* = 8.8 Hz).

4-Nitrobiphenyl⁶: ¹H NMR (400MHz, CDCl₃): δ 7.44-7.50 (m, 3H); 7.61-7.63 (m, 2H), 7.73 (d, 2H, *J* = 8.8 Hz), 8.29 (d, 2H, *J* = 8.8 Hz).

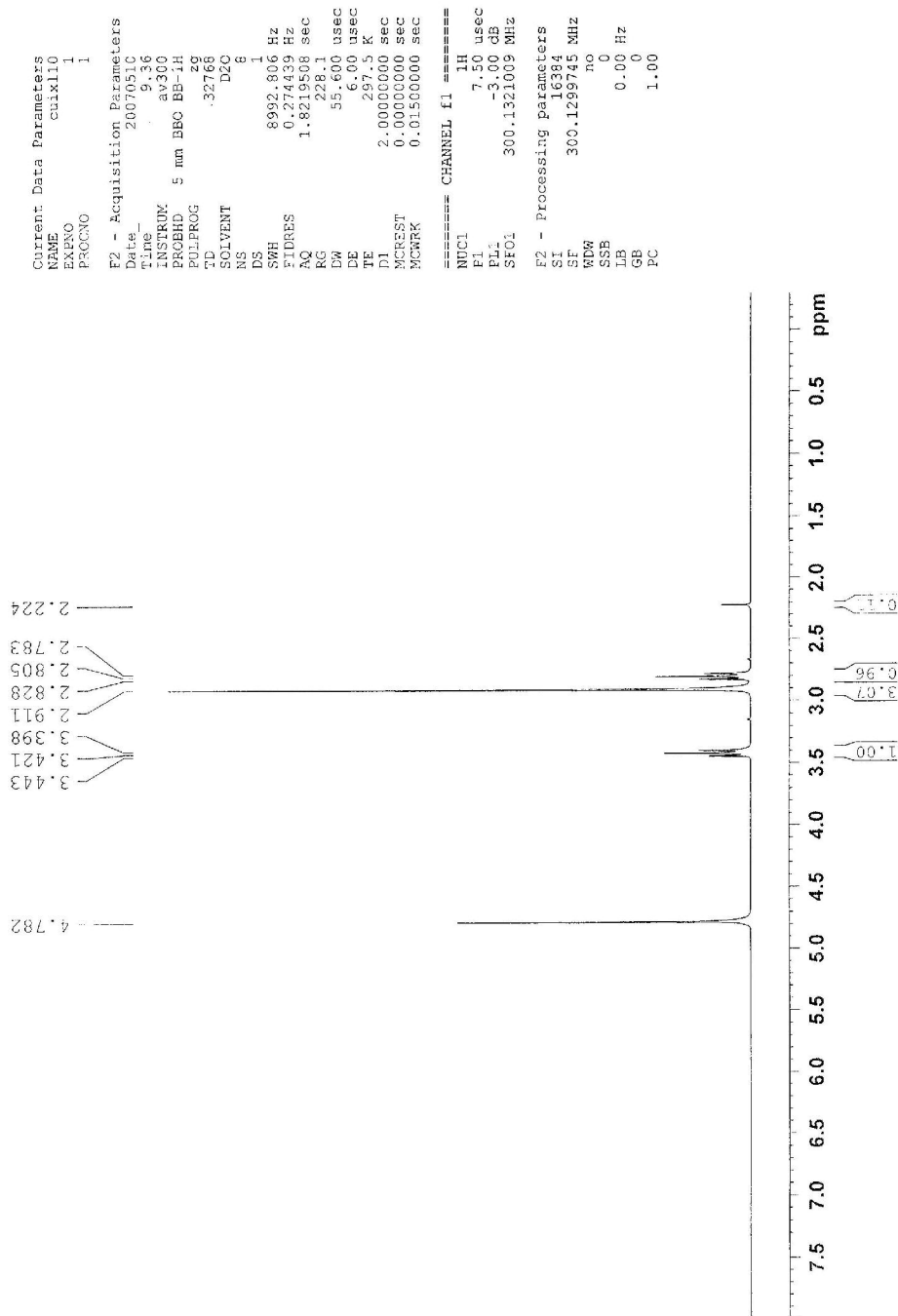
4-Cyanobiphenyl⁶: ¹H NMR (400MHz, CDCl₃): δ 7.42-7.48 (m, 3H), 7.60 (m, 2H), 7.67-7.74 (m, 4H).

(B) References

- (1) Cui, X.; Li, Z.; Tao, C.-Z.; Xu, Y.; Li, J.; Liu, L.; Guo, Q.-X. *Org. Lett.* **2006**, *8*, 2467.
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- (3) Aurelio, L.; Box, J. S.; Brownlee, R. T. C.; Hughes, A. B.; Sleebs, M. M. *J. Org. Chem.* **2003**, *68*, 2652.
- (4) Gresham, T. L.; Jansen, J. E.; Shaver, F. W.; Bankert, R. A.; Fiedorek, F. T. *J. Am. Chem. Soc.* **1951**, *73*, 3168.
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- (6) Cui, X.; Qin, T.; Wang, J. R.; Liu, L.; Guo, Q. X.; *Synthesis* **2007**, *3*, 393.

(C) Spectra

3-(dimethylamino)propanamide Hydrochloride (3HCl)



3-(dimethylamino)propanamide Hydrochloride (3·HCl)

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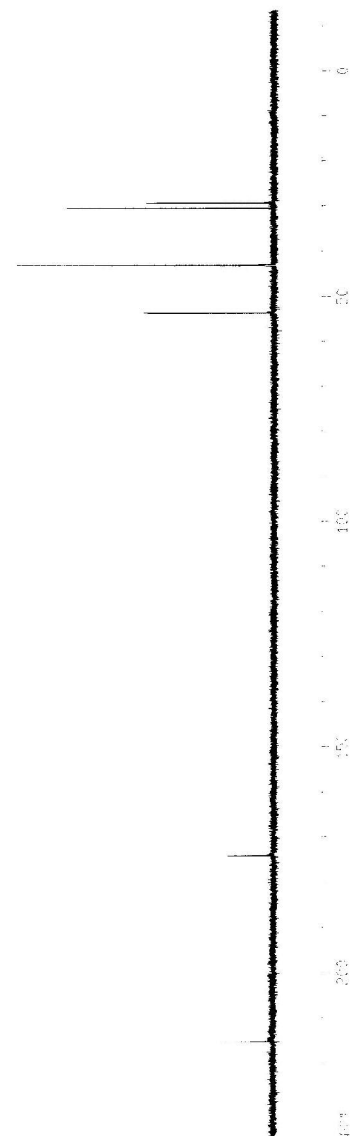
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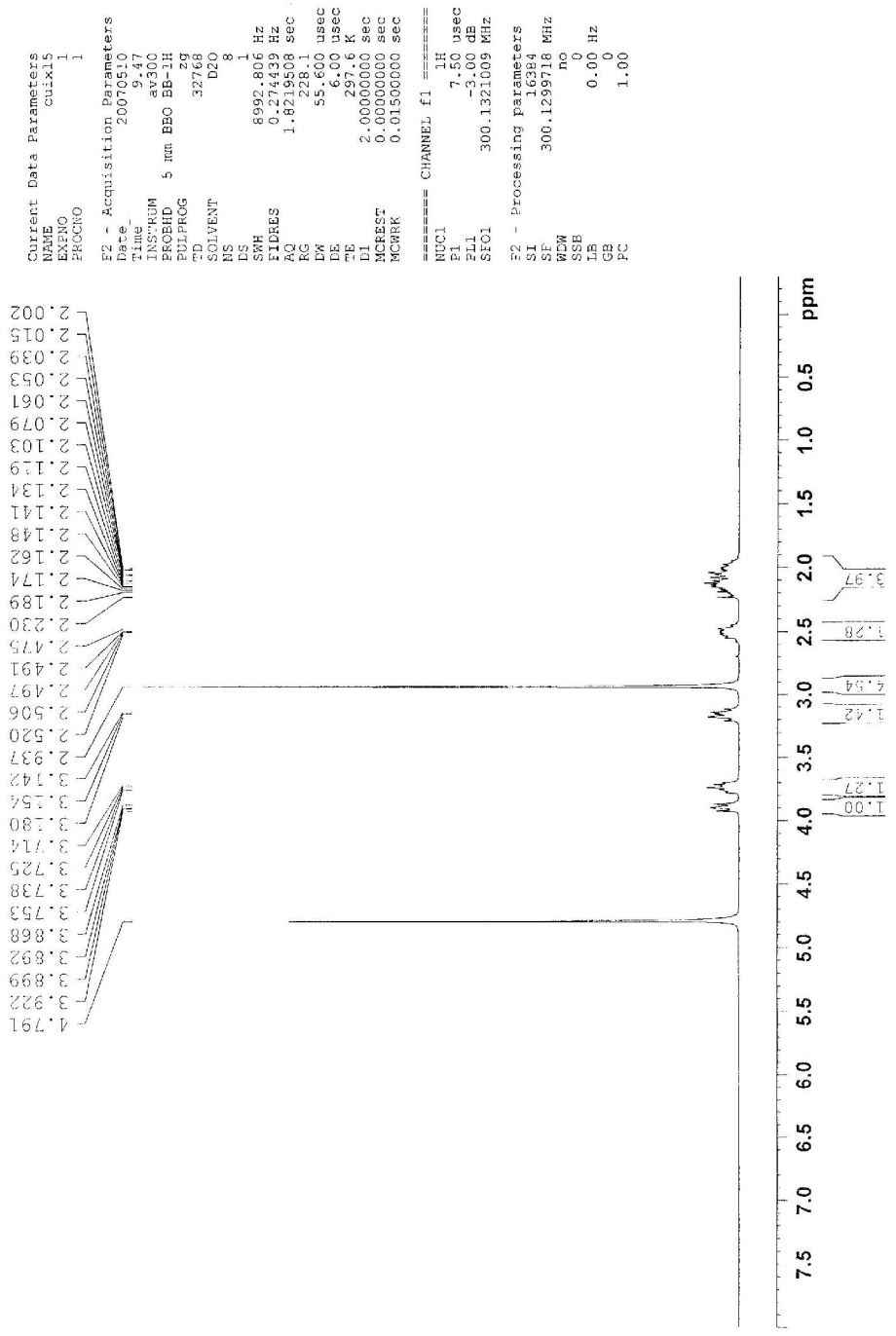
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N-methylproline (5)



N-methylproline (5)

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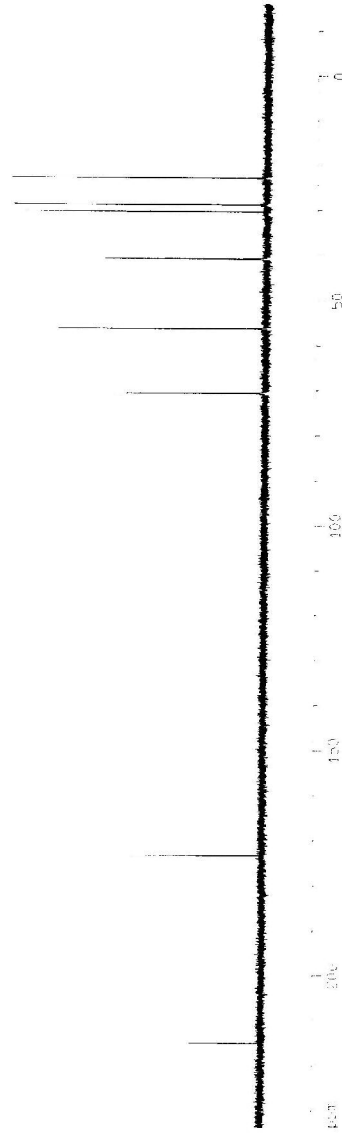
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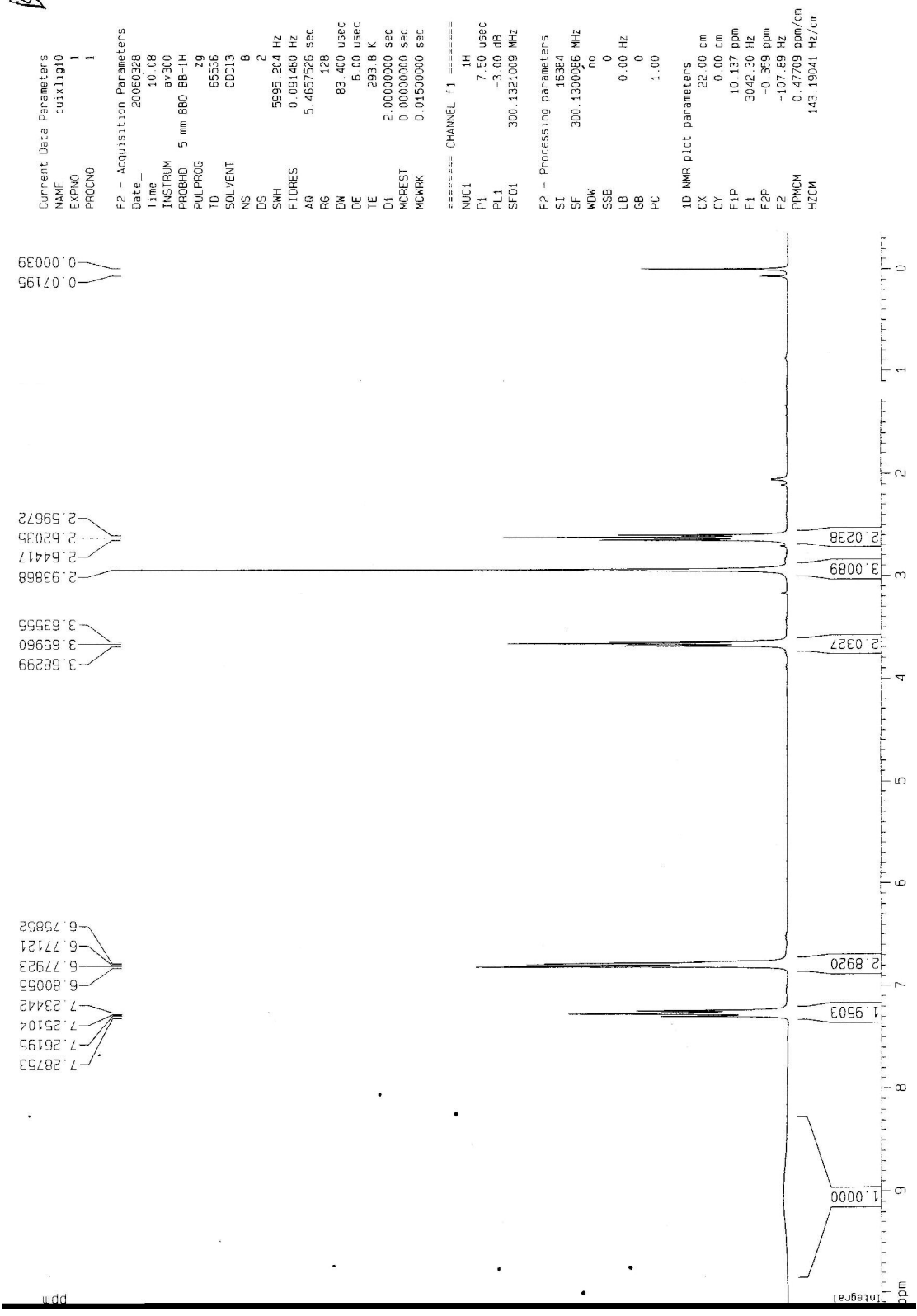
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3-(methyl(phenyl)amino)propanoic acid (6)

B2



3-(methyl(phenyl)amino)propanoic acid (6)

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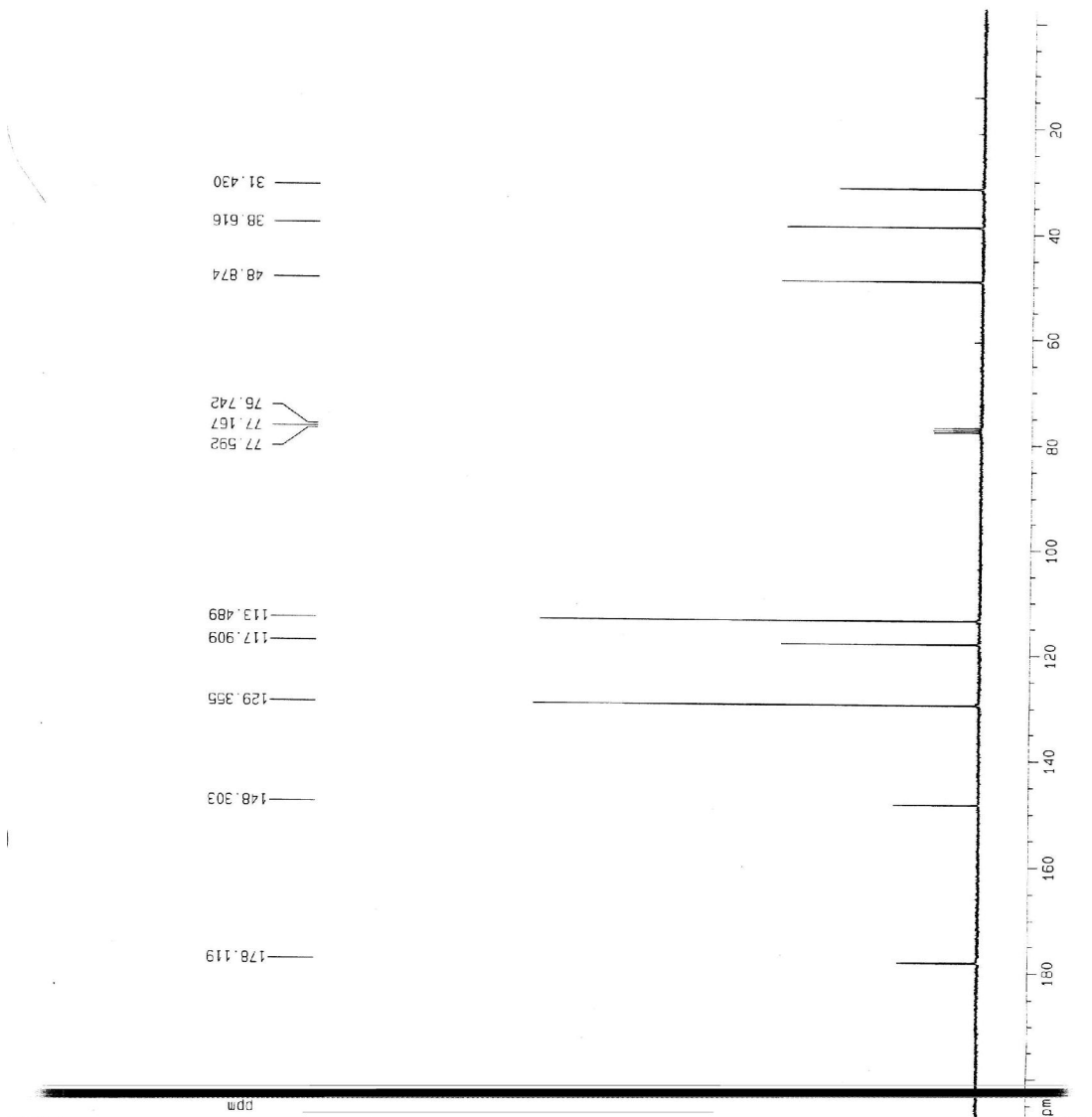
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 D1 2.00000000 sec
 d11 0.03600000 sec
 MCREST 0.00000000 sec
 MCHK 0.01500000 sec

===== CHANNEL f1 =====
 NUC1 ^{13}C
 P1 3.00 usec
 PL1 -1.00 dB
 SF01 75.4766051 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -3.00 dB
 PL12 15.00 dB
 SF02 300.1315007 MHz

F2 - Processing parameters
 SI 131072
 SF 75.4677615 MHz
 MDW 0
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 22.00 cm
 CY 206.390 ppm
 F1 15755.75 Hz
 F2 -2.777 ppm
 F3 -209.56 Hz
 PRACH 9.59651 ppm/cm
 HZCW 724.37767 Hz/cm



3-(methoxy(methyl)amino)propanoic acid Hydrochloride (7HCl)

139

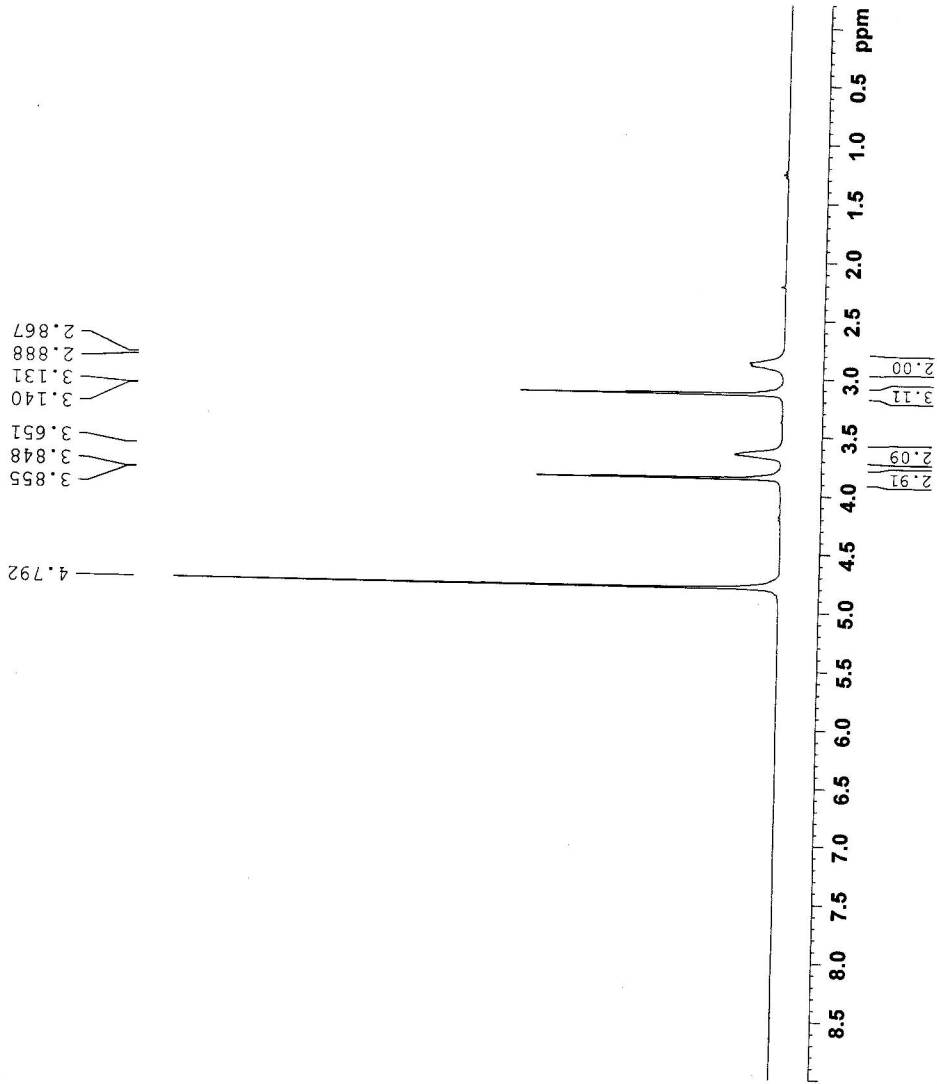
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Current Data Parameters
Name      cu11g1g1
EXPNO    1
PROCNO   1

F2 - Acquisition Parameters
Date_    20060330
Time     9.05
INSTRUM  av300
PROBHD   5 mm BBO BB-1H
PULPROG  zg
TD        65536
SOLVENT  D2O
NS        8
DS        2
SWH       5995.204 Hz
FIDRES   0.001460 Hz
AQ        5.4657526 sec
RG         83.400
DM         6.00 usec
DE         6.00 usec
TE        295.6 K
D1        2.00000000 sec
MCREST   0.00000000 sec
MCWRK    0.01500000 sec

===== CHANNEL f1 =====
NUC1      1H
P1        7.50 usec
PL1       -3.00 dB
SFO1     300.1321009 MHz

F2 - Processing parameters
SI        16384
SF        300.1299713 MHz
WDW       DC
SSB       0
LB        0
GB        0
PC        1.00
    
```



3-(methoxy(methyl)amino)propanoic acid Hydrochloride (7HCl)

```

Current Data Parameters
NAME      cuixliglic
EXPNO    4
PROCNO   1

F2 - Acquisition Parameters
Date_    20060330
Time     16.14
INSTRUM av300
PROBHD  5 mm BBO BB-1H
PULPROG zgpgc
TD       32768
SOLVENT D2O
NS       79
DS       4
SWH      28675.736 Hz
FIDRES   0.6922009 Hz
AQ        0.7225844 sec
RG        57992.6
DM        22.050 usec
DE        6.70 usec
TE        297.3 K
D1        2.0000000 sec
d11       0.0300000 sec
MCHEST   0.0000000 sec
PCPRG2   0.0150000 sec

===== CHANNEL f1 =====
NUC1      13C
P1        3.00 usec
PL1       -1.00 dB
SFO1      75.4776051 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2       -3.00 dB
PL12      15.00 dB
SFO2      300.1315007 MHz

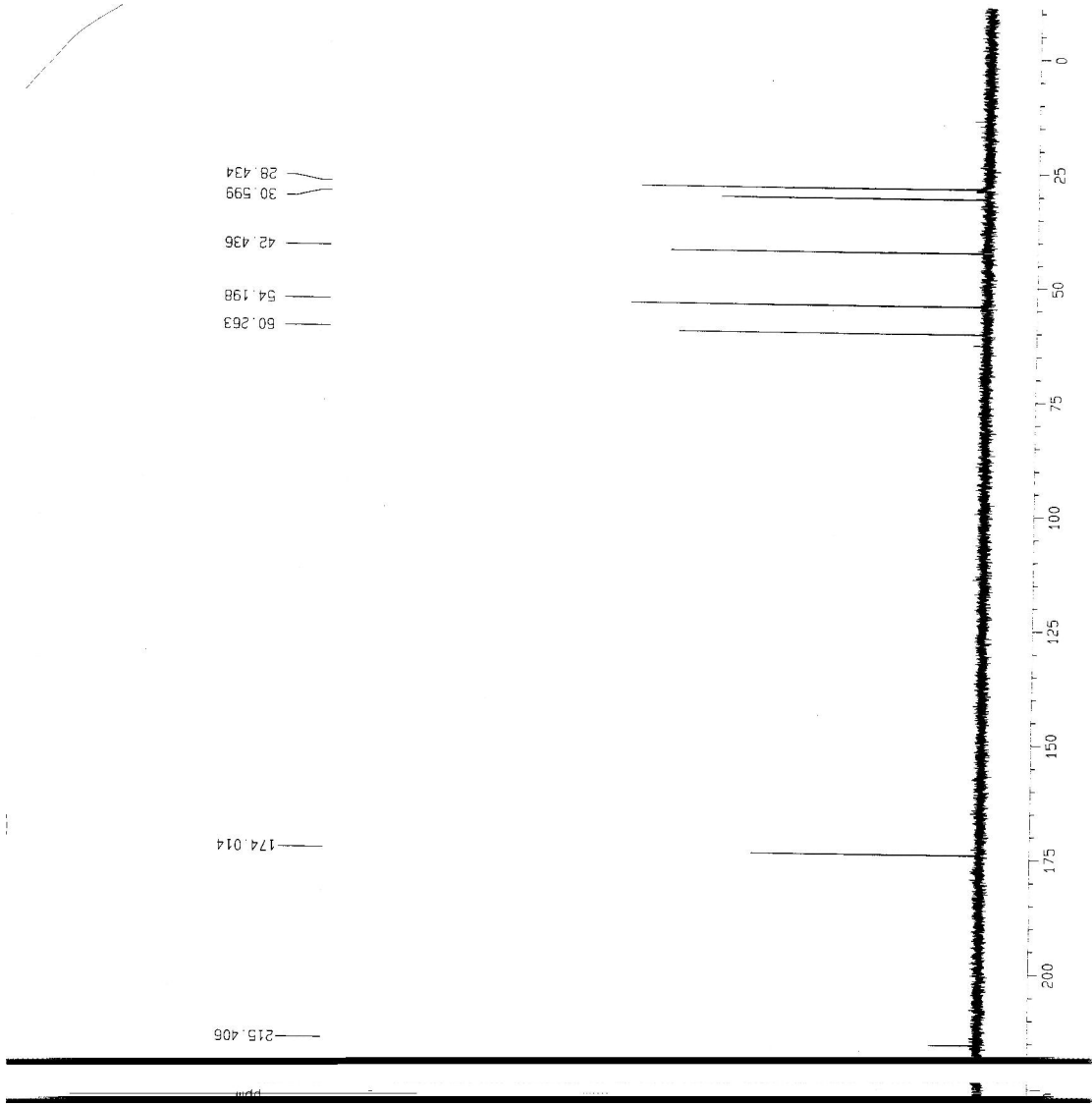
F2 - Processing parameters
SI         131072
SF         75.467264 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40

1D NMR plot parameters
CX         22.00 cm
F1         0.00 cm
F2         231.063 ppm
F3         17437.83 Hz
F4         -11.187 ppm
F5         -844.25 Hz
PRCKN     11.0138 ppm/cm
-ZCM      831.00366 Hz/cm
    
```

28.434
 30.599
 42.436
 54.198
 60.263

174.014

215.406



2-(dimethylamino)benzoic acid (8)

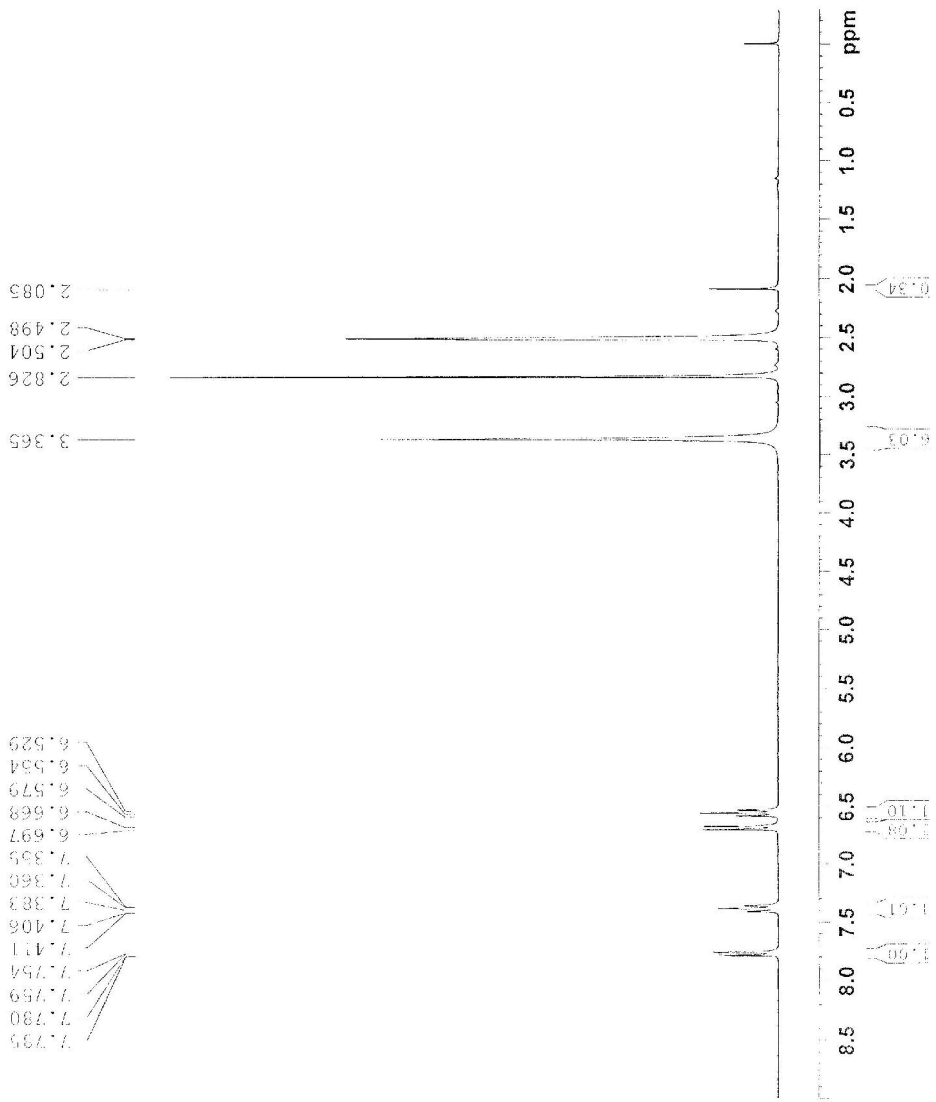
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Current Data Parameters
NAME:          cuix13
PROCNO:       1
PRCNO:        1

F2 - Acquisition Parameters
Date_:        20070510
Time_:        9.45
INSTRUM:      av300
PROBHD:       5 mm BBO BB-1H
PULPROG:      zgpg
TD:           32768
SOLVENT:      DMSO
NS:           8
DS:           1
SWH:          8992.856 Hz
FIDRES:       0.276439 Hz
AQ:           1.821933 sec
RG:           327.68
DE:           55.600 usec
TE:           297.5 K
D1:           2.0000000 sec
ECOREST:      6.0000000 sec
RGCRS:        6.0150000 sec

===== CHANNEL f1 =====
NUC1:         1H
P1:           7.50 usec
PL1:          -3.00 dB
SFO1:         300.1321009 MHz

F2 - Processing parameters
SI:           16384
SF:           300.1299999 MHz
WDW:          EM
SSB:          0
LB:           0.00 Hz
GB:           0
PC:           1.00
    
```



2-(dimethylamino)benzoic acid (8)

Current Data Parameters
 NAME: C11X13C
 EXPTNO: 4
 PROCNO: 1

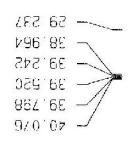
F2 - Acquisition Parameters
 Date_ 20070514
 Time 8.32
 INSTRUM av300
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg30
 TO 32758
 SOLVENT D2O
 FID0 183
 NS 4
 DS 4
 SMH 28675.735 Hz
 FIDRES 0.690009 Hz
 AQ 0.728844 sec
 RG 2896.3
 JW 22.050 usec
 JE 298.5 K
 IE 6.00 usec
 ZF 2.0000000 sec
 S11 0.0300000 sec
 WREST 0.0000000 sec
 WMRK 0.0150000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 3.00 usec
 PL1 -1.00 dB
 SFO1 75.476051 MHz

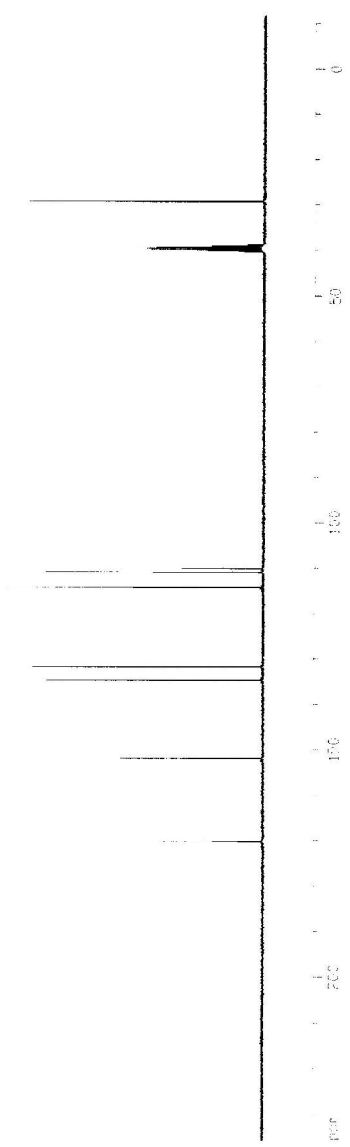
===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 3.00 dB
 PL12 15.00 dB
 SFO2 300.1315007 MHz

F2 - Processing parameters
 SI 131072
 SF 75.4677693 MHz
 RGW EM
 SSB 0
 IB 0.50 Hz
 GB 0
 LC 1.40

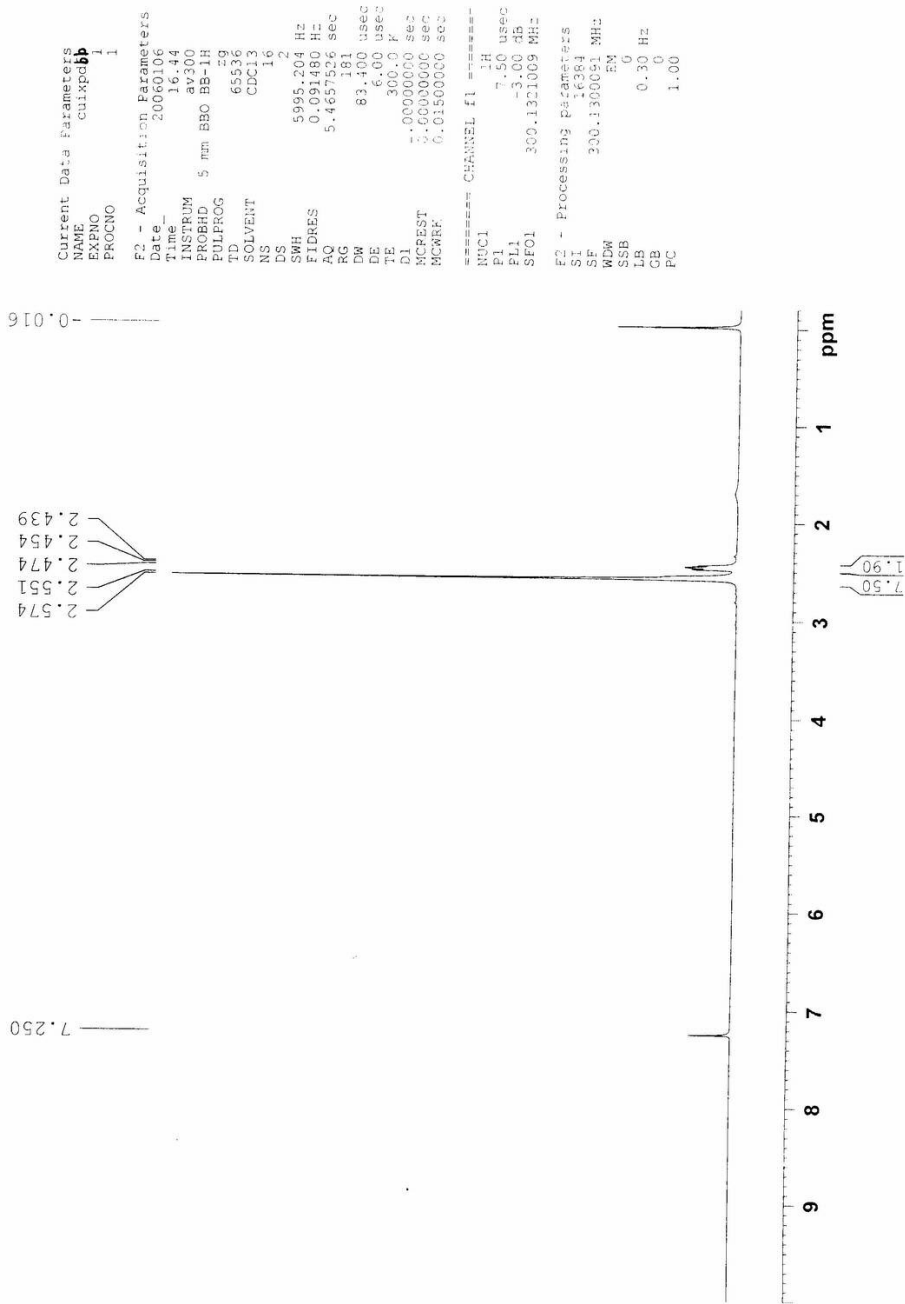
ID-NMR plot parameters
 CX 22.00 cm
 CY 5.00 cm
 F1P 235.822 ppm
 F1 17864.63 Hz
 F2P -11.755 ppm
 F2 -887.13 Hz
 SHCXM 11.29465 ppm/cm
 AQCM 846.62079 Hz/cm



ppm



Pd(N, N-dimethyl-β-alaninate)₂ (16)



Pd(N, N-dimethyl-β-alaninate)₂ (16)

Current Data Parameters
 NAME Cuixp00bc
 EXPNO 4
 PROCNO 1

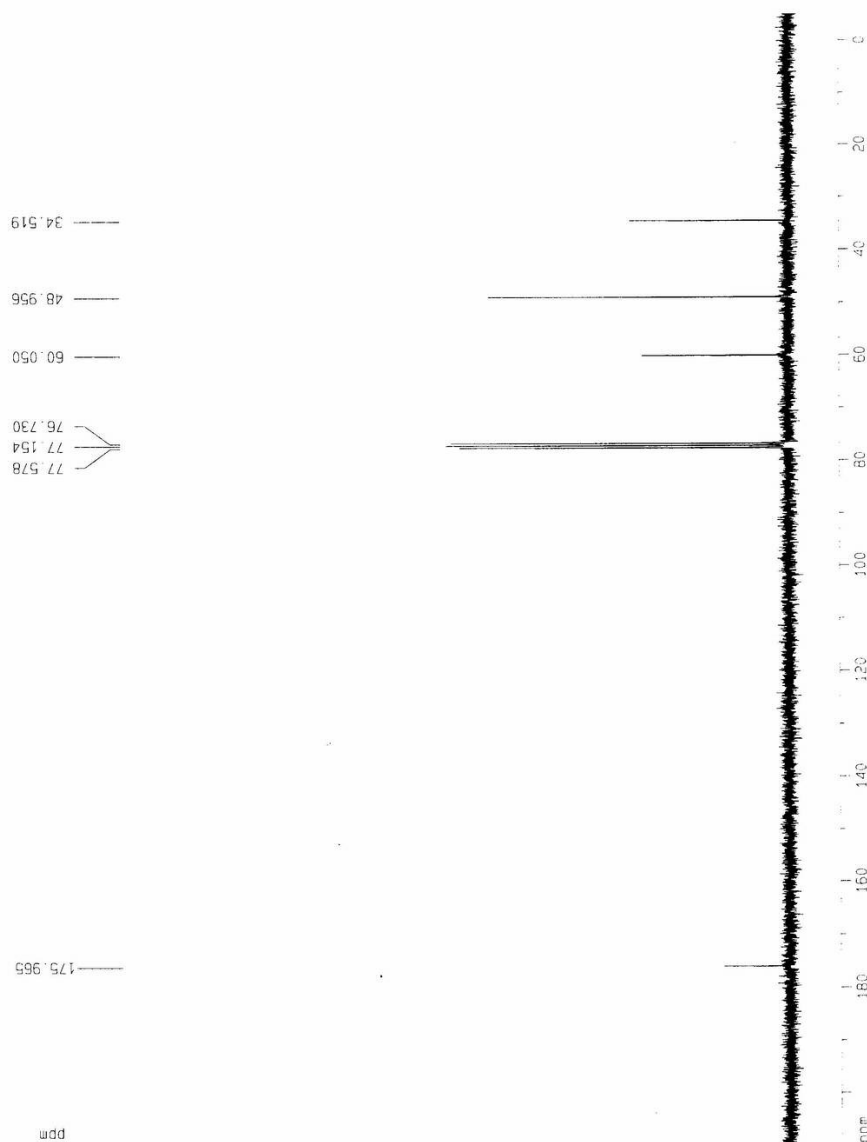
F2 - Acquisition Parameters
 Date_ 20060110
 Time 16:43
 INSTRUM av300
 PROBHD 5 mm BBO BB-H1
 PULPROG zgpg
 TD 32768
 SOLVENT C6D6
 NS 97
 DS 4
 SWH 22675.736 Hz
 FIDRES 0.692009 Hz
 AQ 0.7225944 sec
 RG 2580.3
 DM 22.050 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 VCFIRST 0.00000000 sec
 VCFWRK 0.01500000 sec

===== CHANNEL f1 =====
 NU1 13C
 P1 3.00 usec
 PL1 -1.00 dB
 SF01 75.4766051 MHz

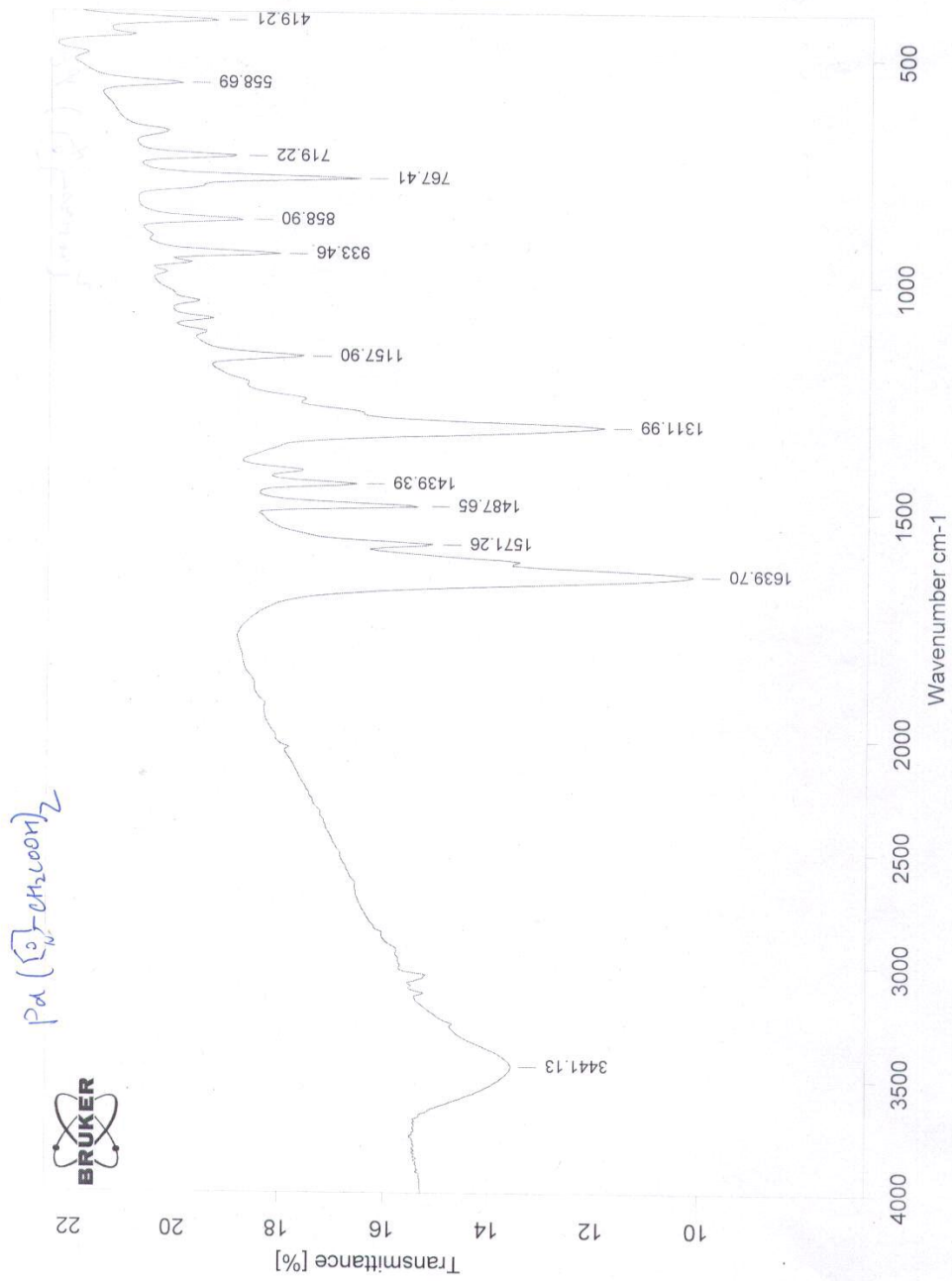
===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NU2 1H
 PCPD2 80.00 usec
 PL2 -3.00 dB
 PL12 15.00 dB
 SF02 300.1315007 MHz

F2 - Processing parameters
 S1 131072
 SF 75.4677415 MHz
 MDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 22.00 cm
 CY 0.00 cm
 ZP 209.564 ppm
 F1 158.1536 Hz
 F2 -5.085 ppm
 SFO1 75.4677415 MHz
 SFO2 300.1315007 MHz



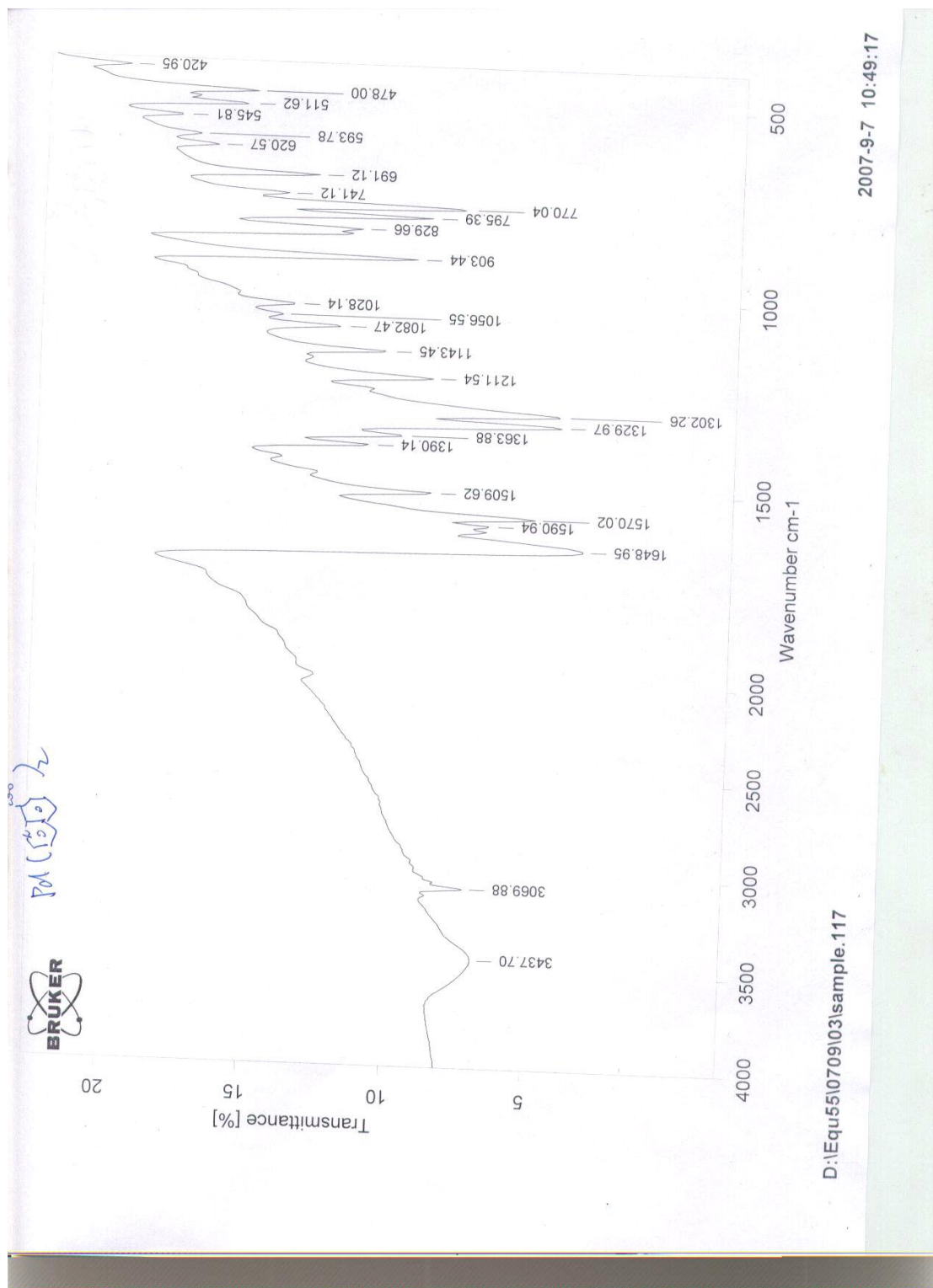
Pd(2-pyridylacetate)₂ (17)



D:\Equ55\0709\03\sample.120

2007-9-7 11:16:07

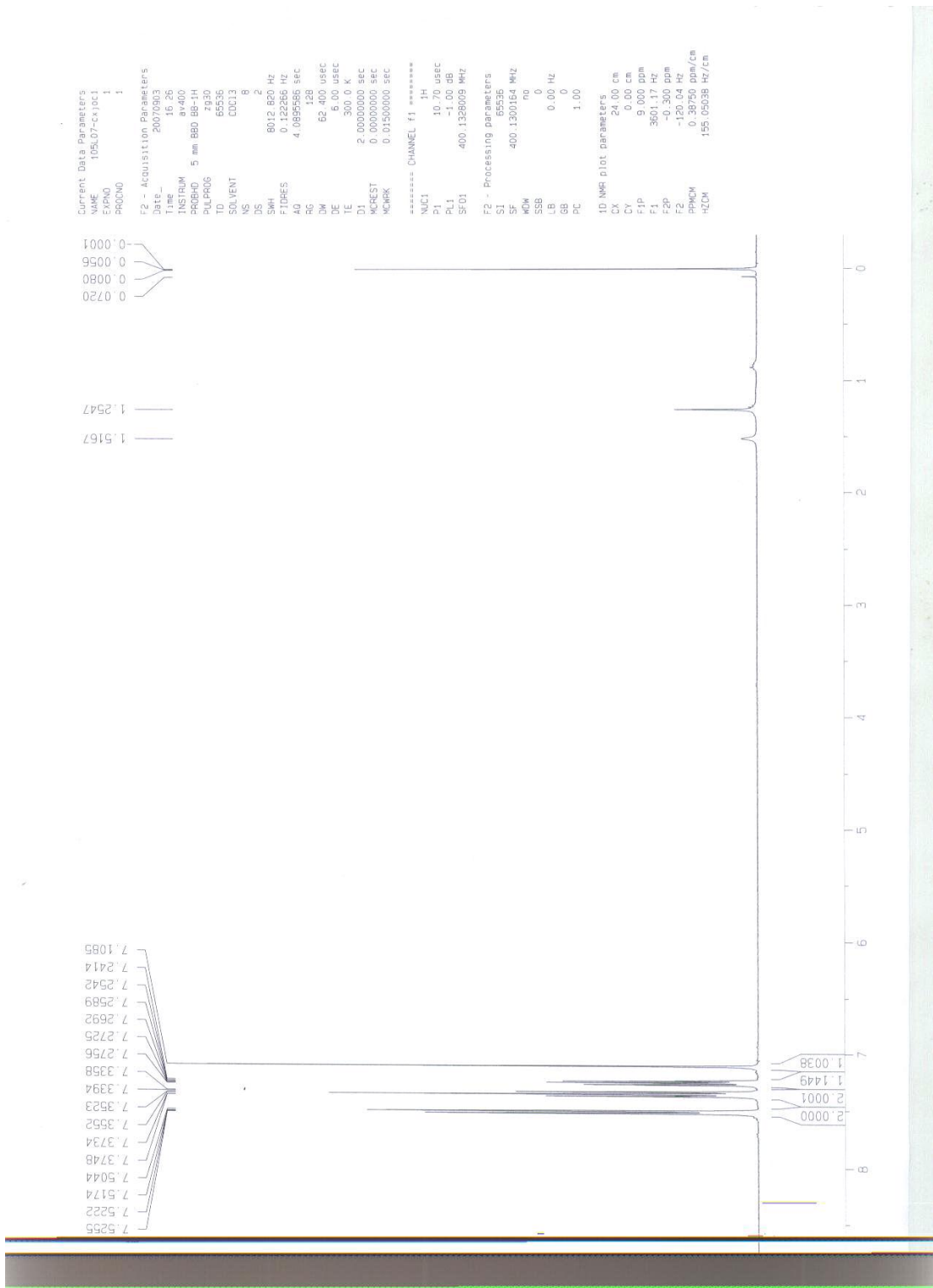
Pd(quinoline-8-carboxylate)₂ (18)



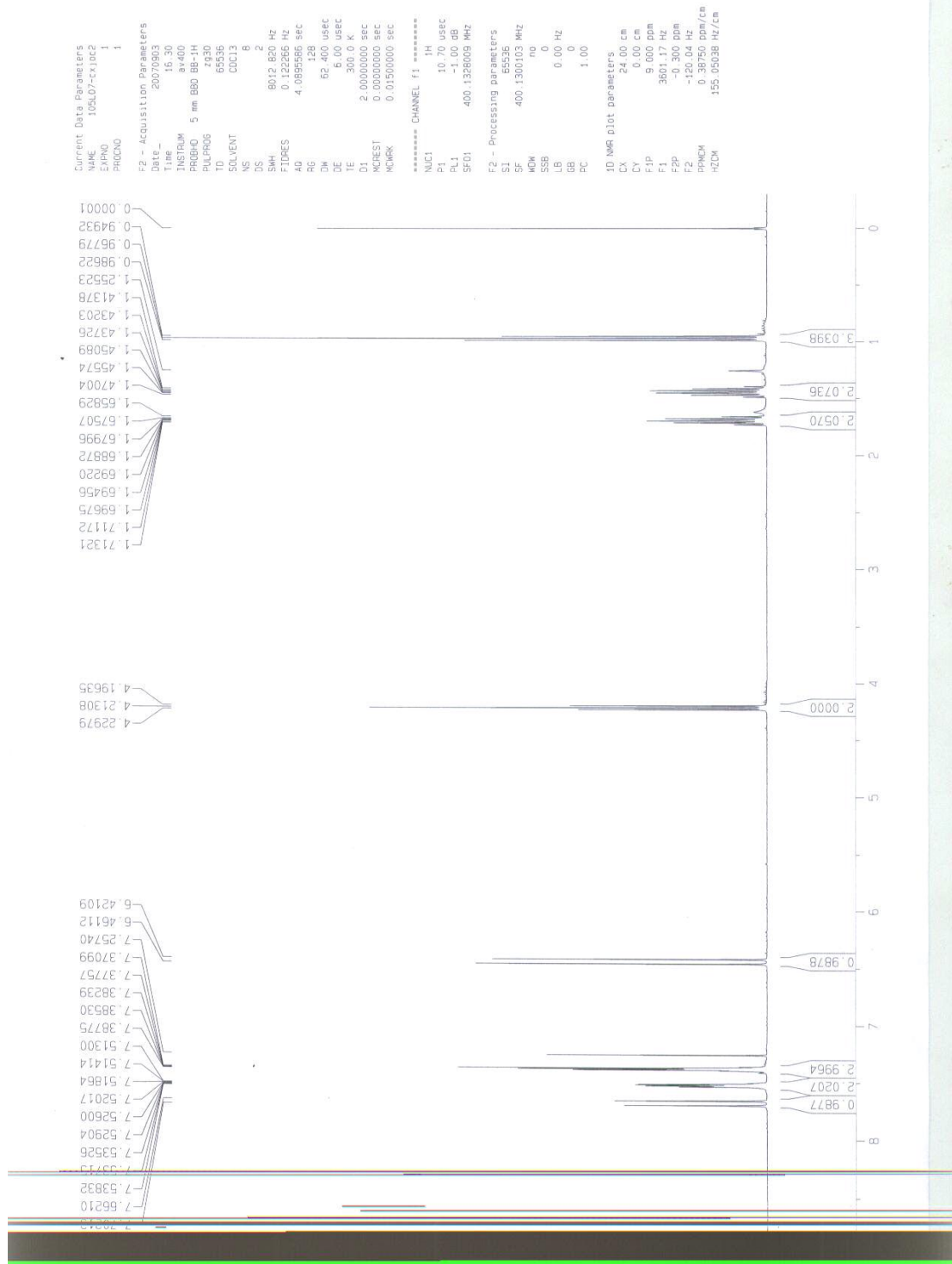
2007-9-7 10:49:17

D:\Equ55\0709\03\sample.117

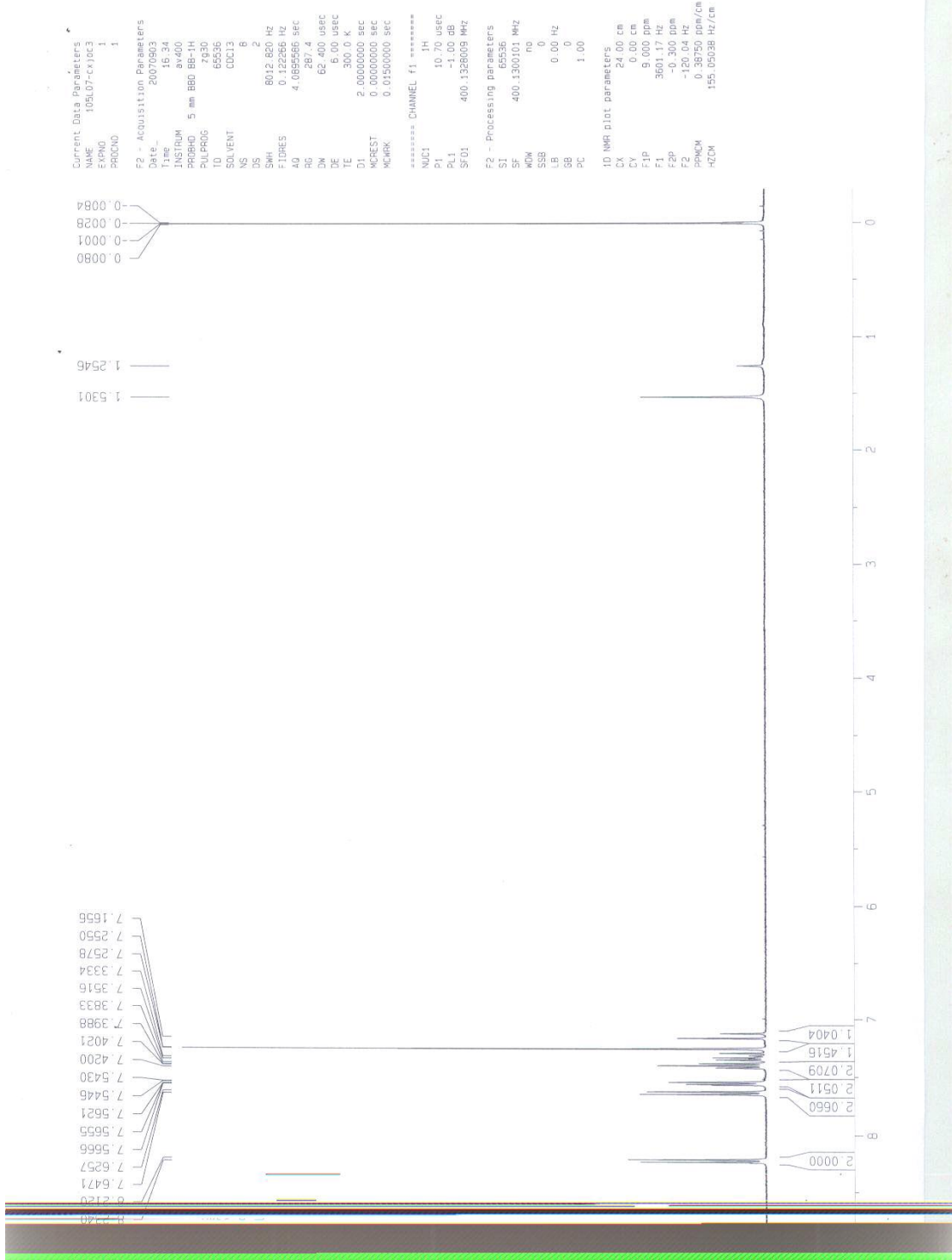
trans-stilbene



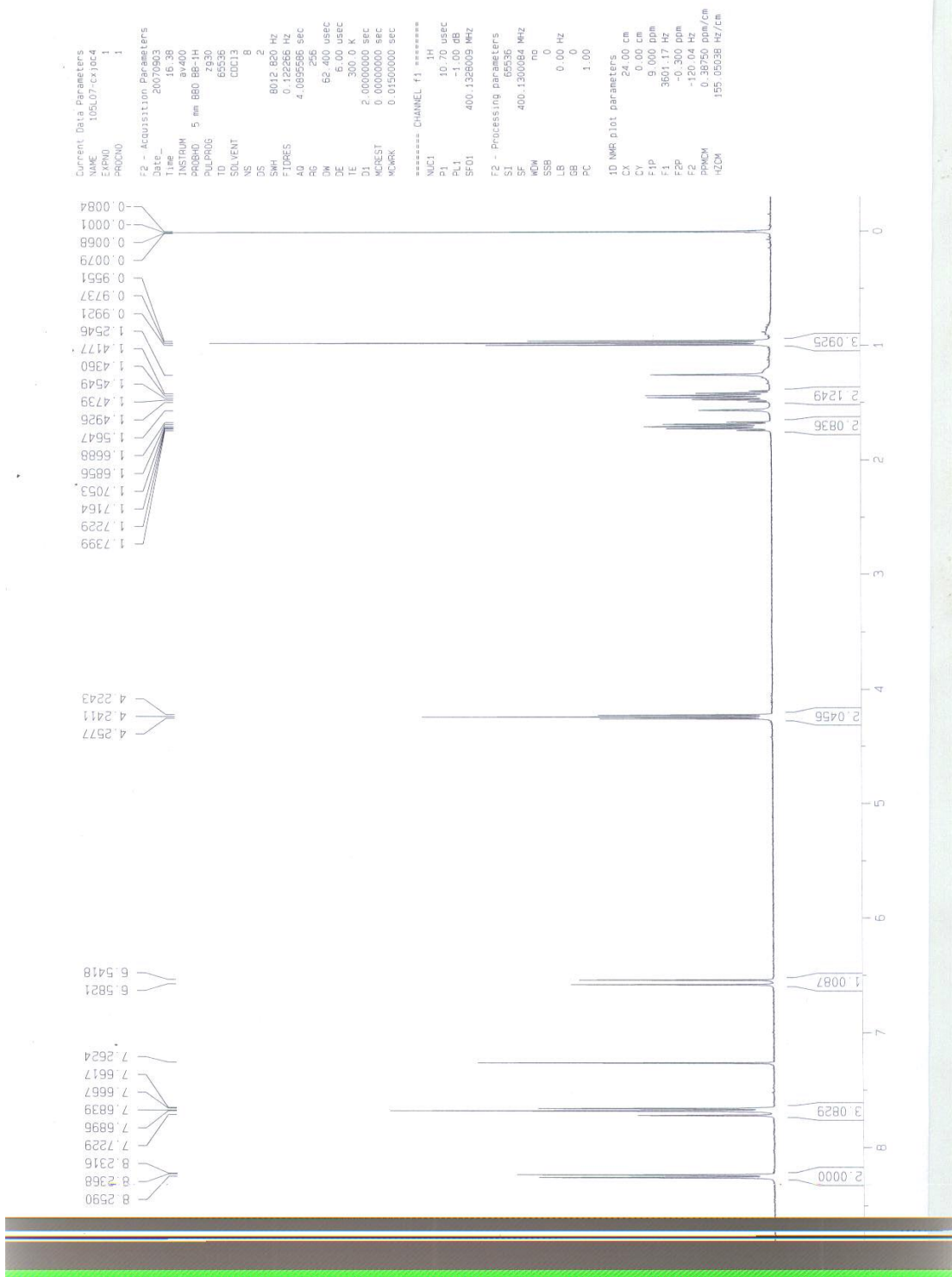
trans-cinnamic acid *n*-butyl ester



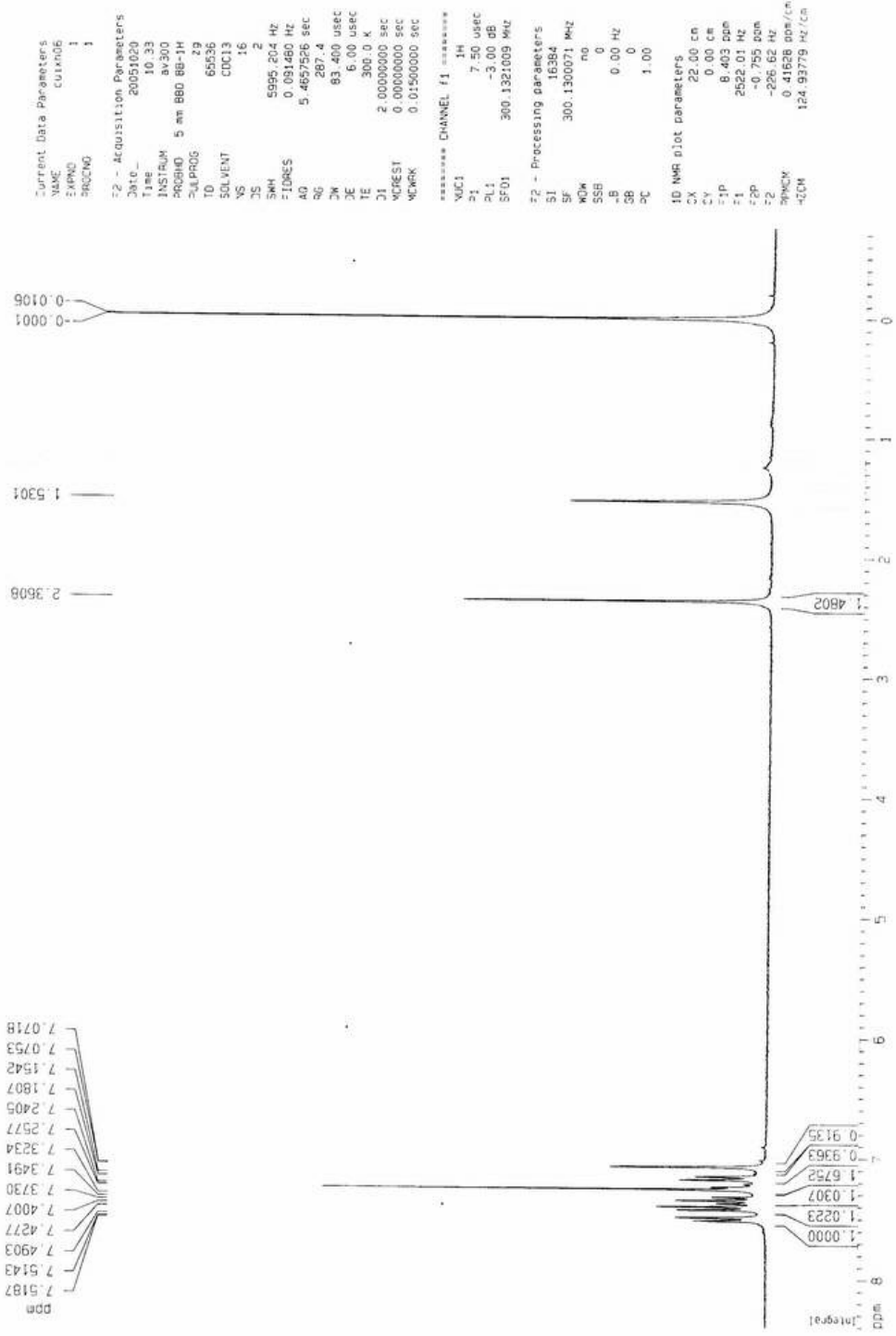
4-nitro-trans-stilbene



4-nitro-trans-cinnamic acid *n*-butyl ester



4-methyl-*trans*-stilbene



4-methyl-*trans*-cinnamic acid *n*-butyl ester

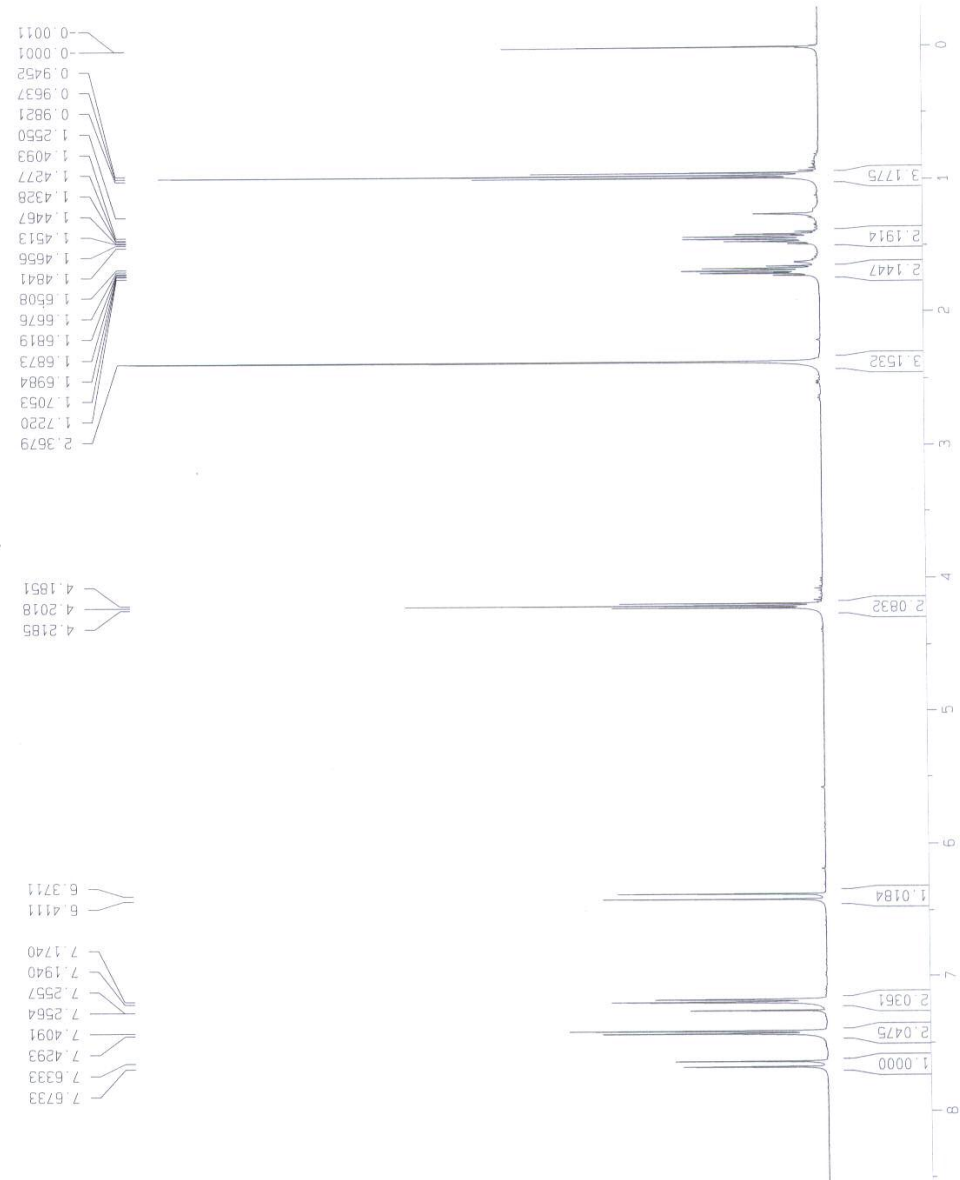
Current Data Parameters
 NAME 105L07-Ex1005
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20070901
 Time 15:43
 INSTRUM av400
 PROBRD 5 mm BBO BB-1H
 PULPROG zg30
 TO 65536
 SOLVENT CDCl3
 NS 8
 DS 2
 SWH 6012.820 MHz
 FIDRES 0.122266 Hz
 AQ 4.089586 sec
 RG 114
 DN 62.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.0000000 sec
 MCREST 0.0000000 sec
 MCHRG 0.0150000 sec

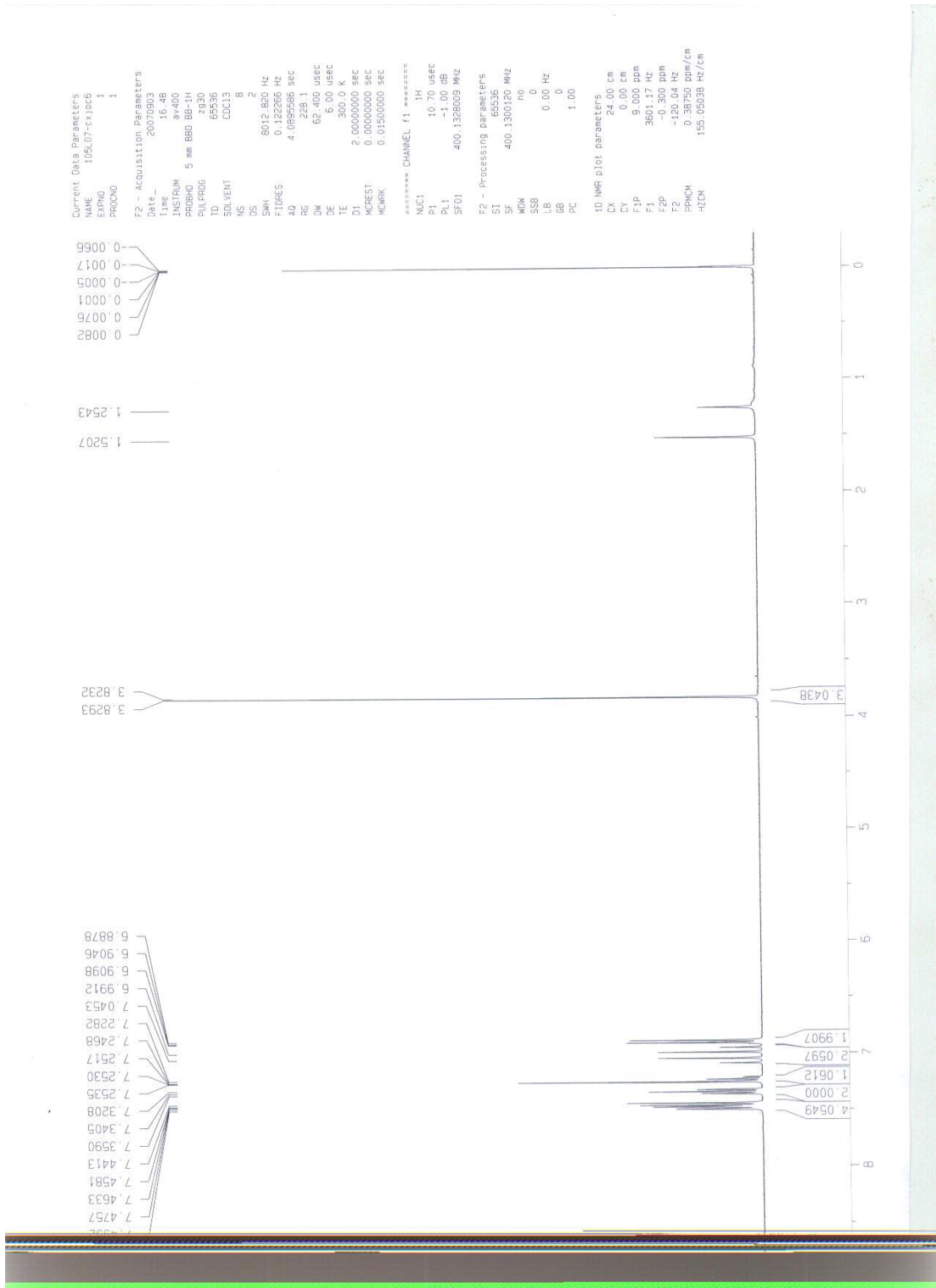
***** CHANNEL f1 *****
 NUC1 1H
 P1 10.70 usec
 PL1 -1.00 dB
 SF01 400.1326000 MHz

F2 - Processing parameters
 SI 65536
 SF 400.1300104 MHz
 MGW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 24.00 cm
 CY 0.00 cm
 FIP 9.000 ppm
 F1 -9601.17 Hz
 F2 -0.300 ppm
 F2 -120.04 Hz
 PRGN CDCl3
 HZPR 155.05038 Hz/cm



4-methoxy-*trans*-stilbene



4-formyl-*trans*-stilbene

Current Data Parameters
 NAME 105L07-CX10CB
 EXPNO 1
 PROCNO 1

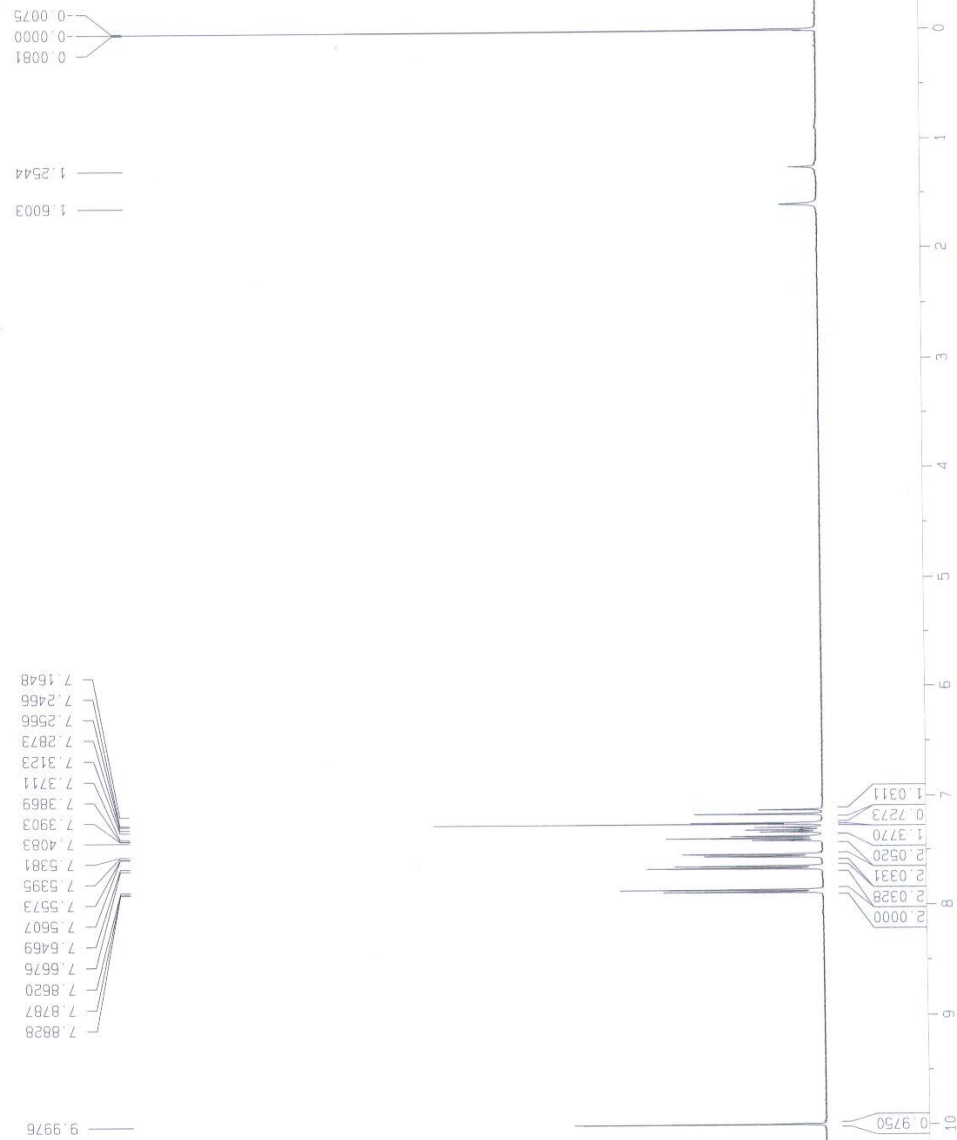
F2 - Acquisition Parameters
 Date_ 20070816
 Time 15:57
 INSTRUM spect
 PROBRW 5 mm BBO BB-1H
 PULPROG zg30
 TO 65536
 SOLVENT CDCl3
 NS 8
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.089586 sec
 RG 228.1
 DW 62.400 usec
 DE 6.00 usec
 TE 300.0 K

D1 2.0000000 sec
 MCREST 0.0000000 sec
 MCWRR 0.0150000 sec

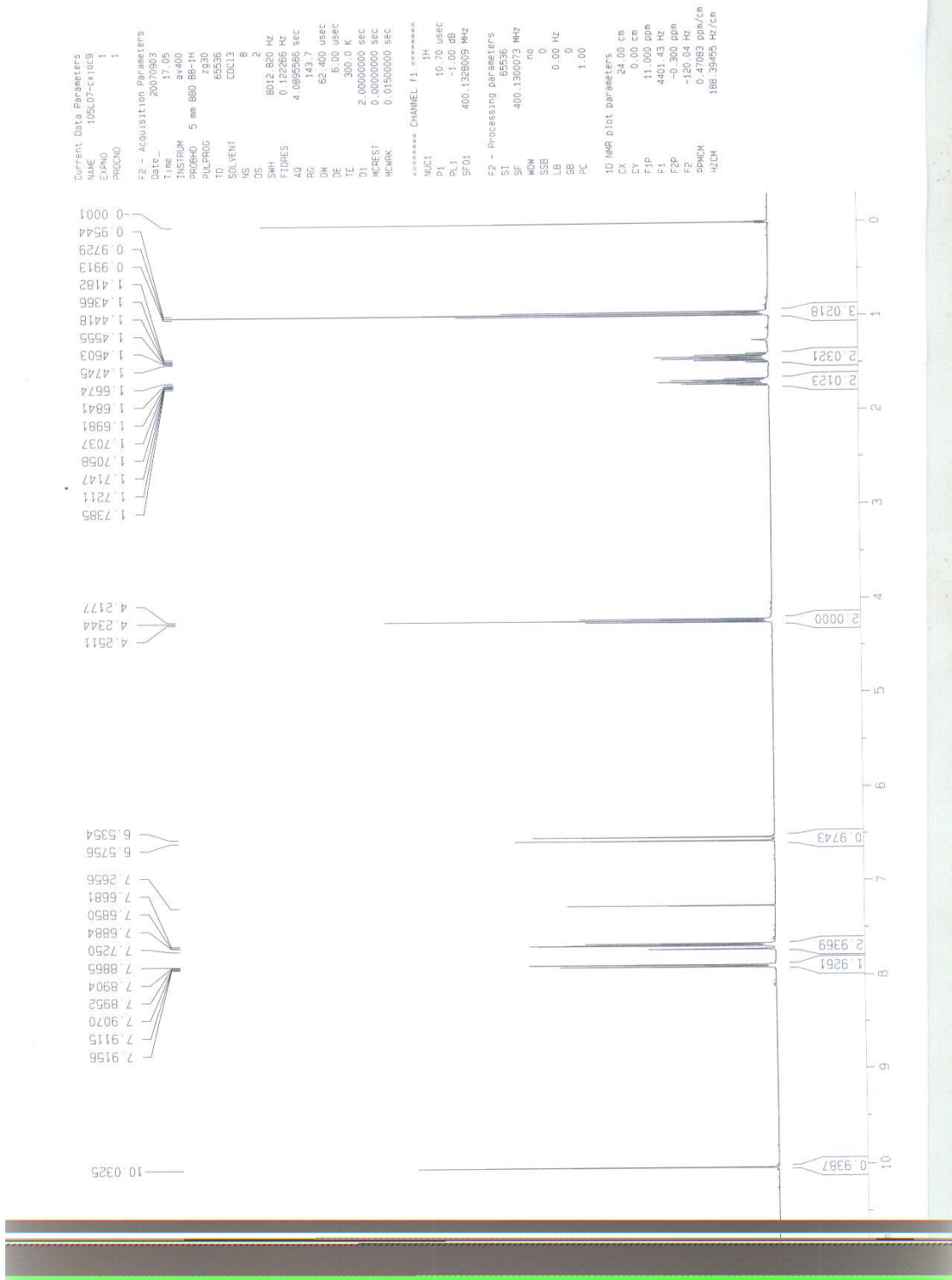
***** CHANNEL f1 *****
 NU1 1H
 P1 10.70 usec
 PL 1.00 dB
 SFO1 400.1326095 MHz

F2 - Processing parameters
 SI 65536
 SF 400.1300106 MHz
 KDM no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

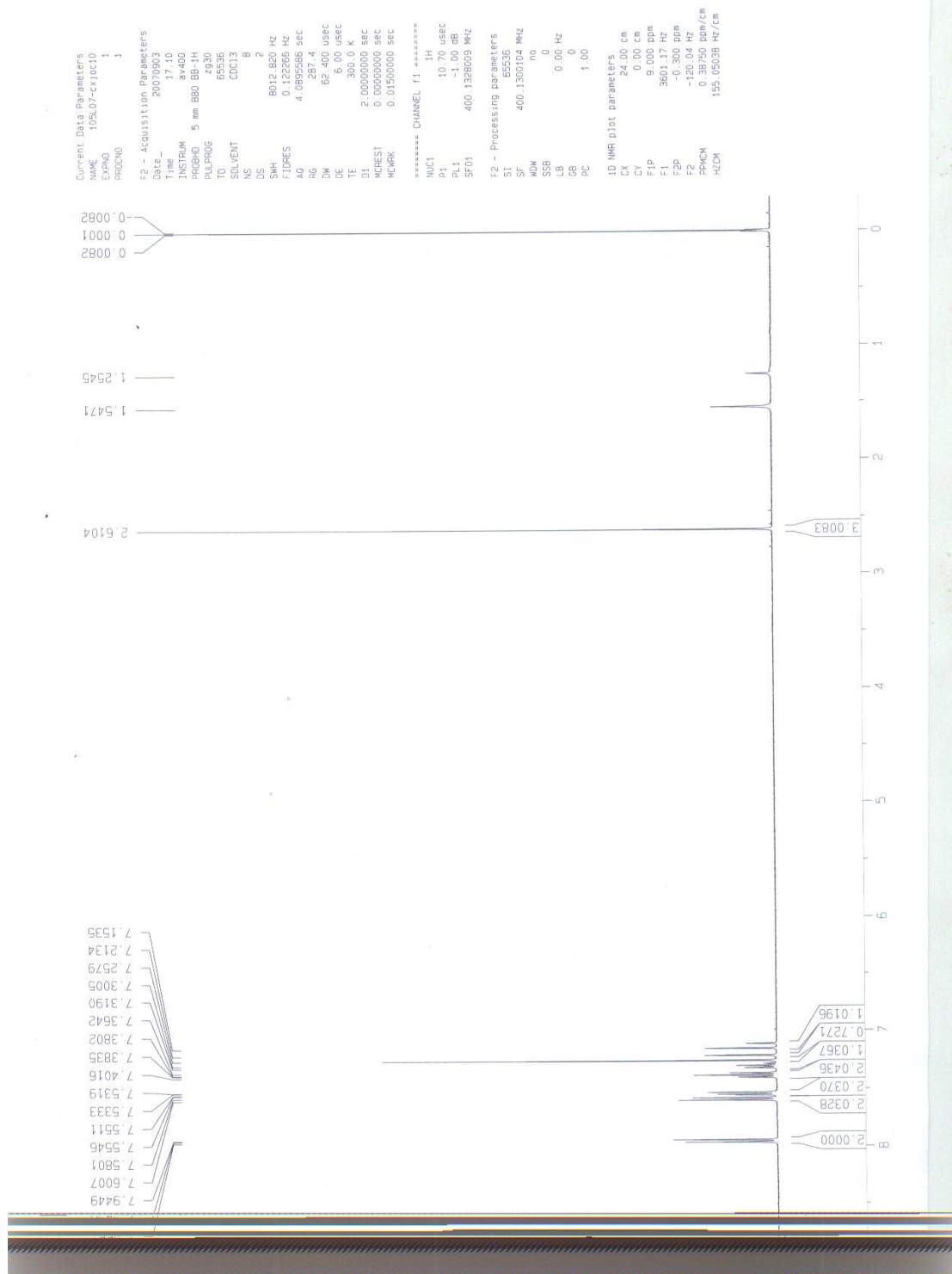
ID MR plot parameters
 CX 24.00 cm
 CY 0.00 cm
 F1P 11.000 ppm
 F1 4401.43 Hz
 F2P -0.300 ppm
 F2 -120.04 Hz
 PPHCN 0.4785 ppm/cm
 HZDN 188.35455 Hz/cm



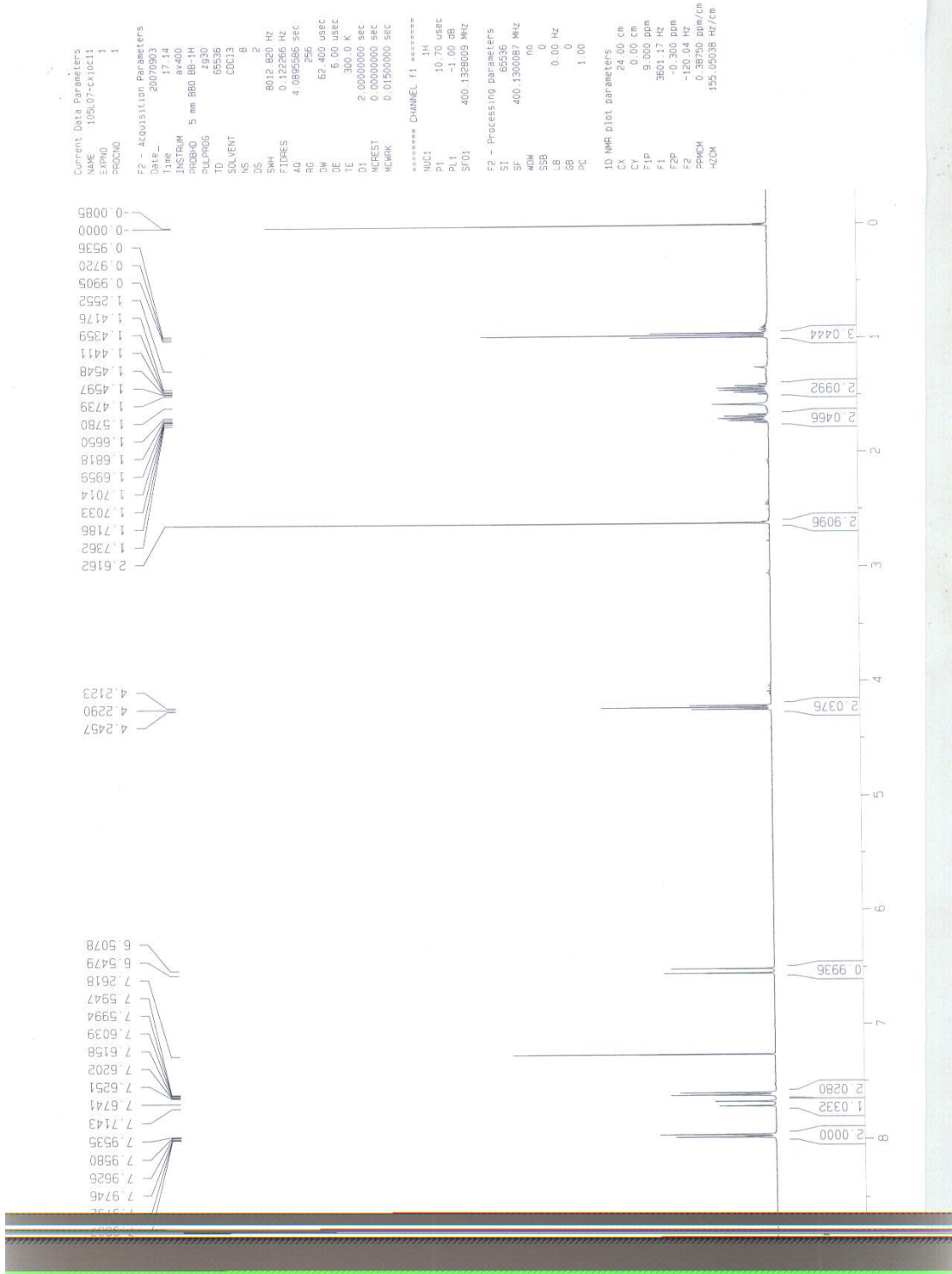
4-formyl-*trans*-cinnamic acid *n*-butyl ester



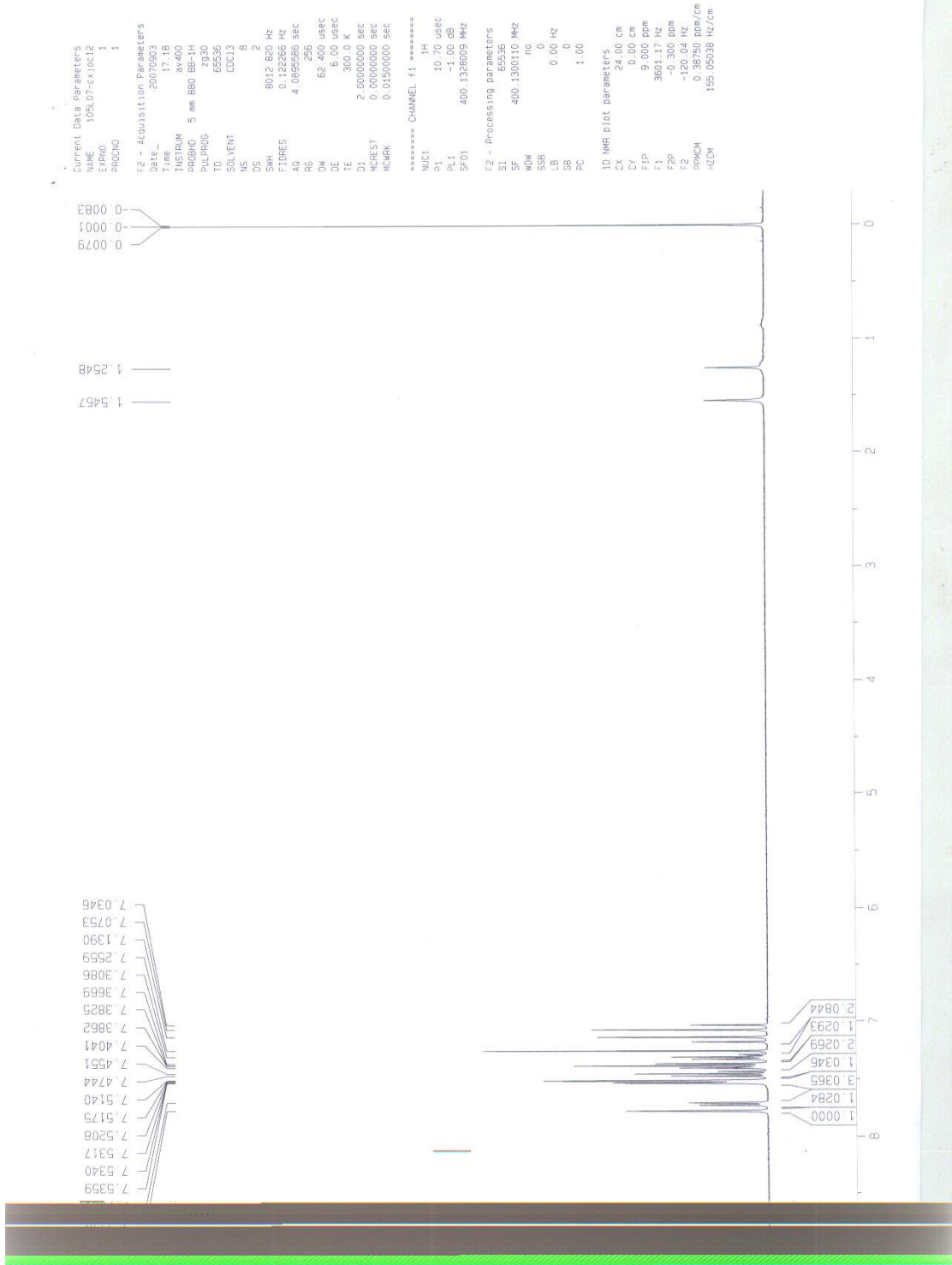
4-acetyl-trans-stilbene



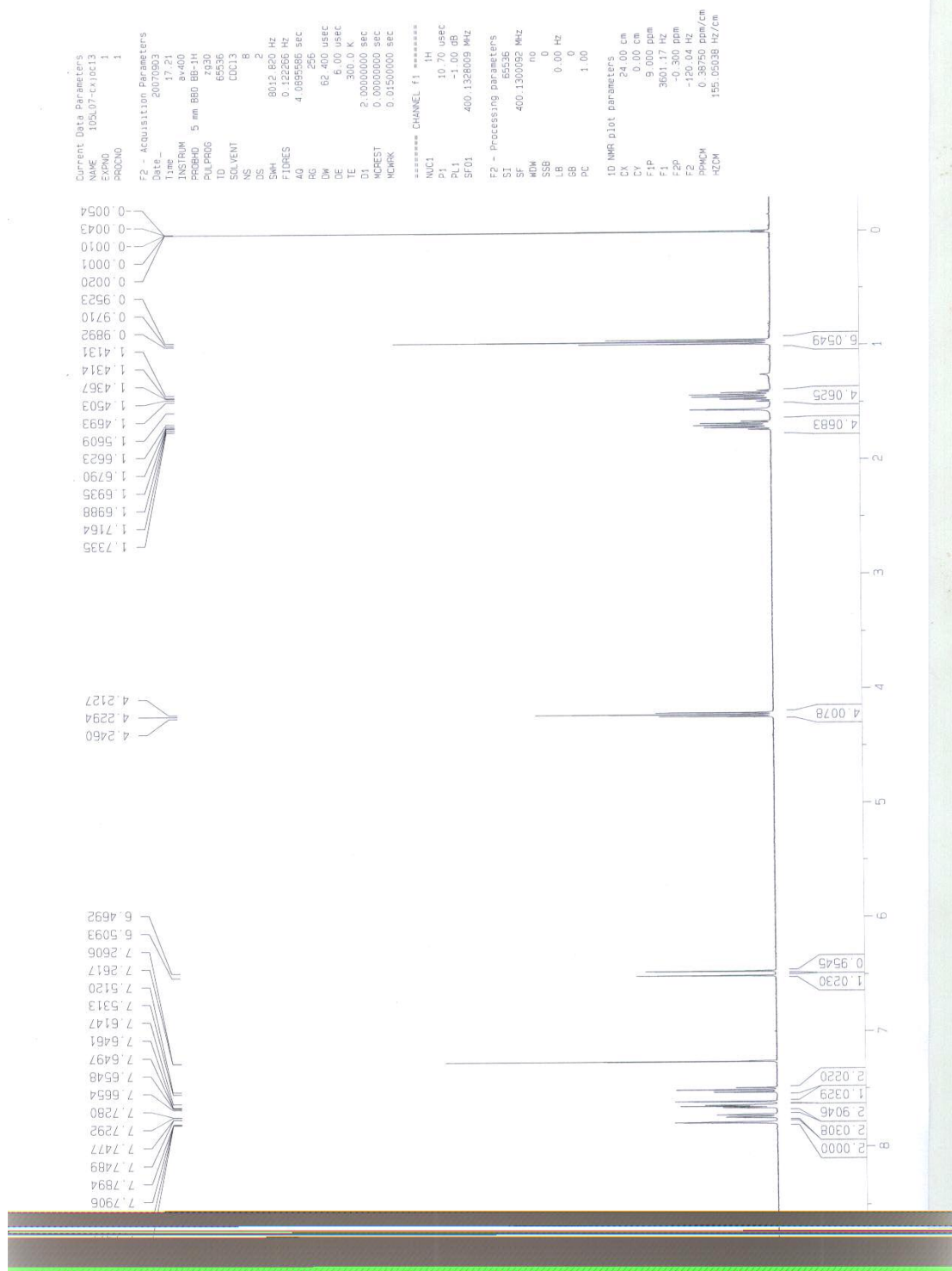
4-acetyl-*trans*-cinnamic acid *n*-butyl ester



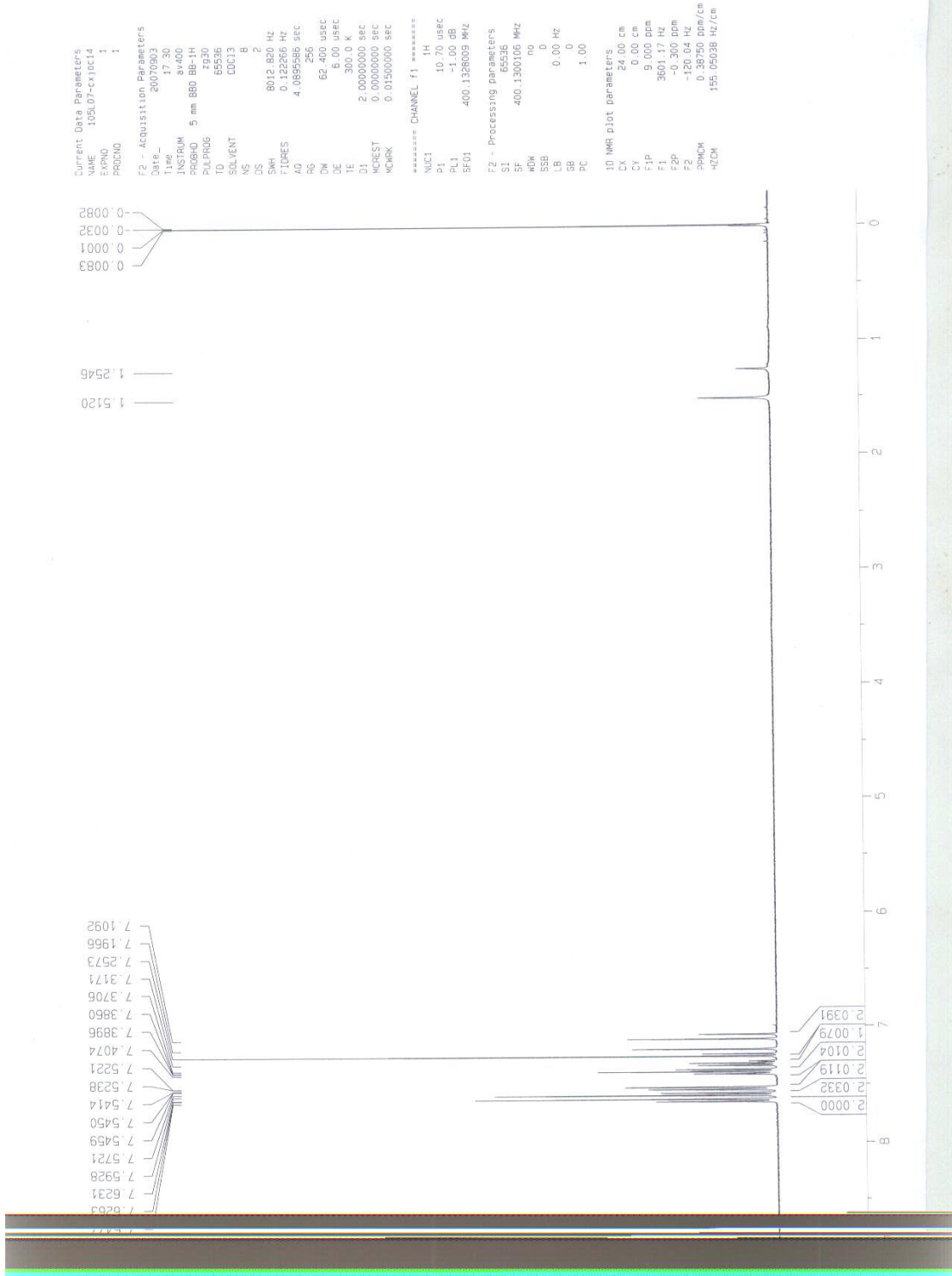
trans-stilbene-3-carbonitrile



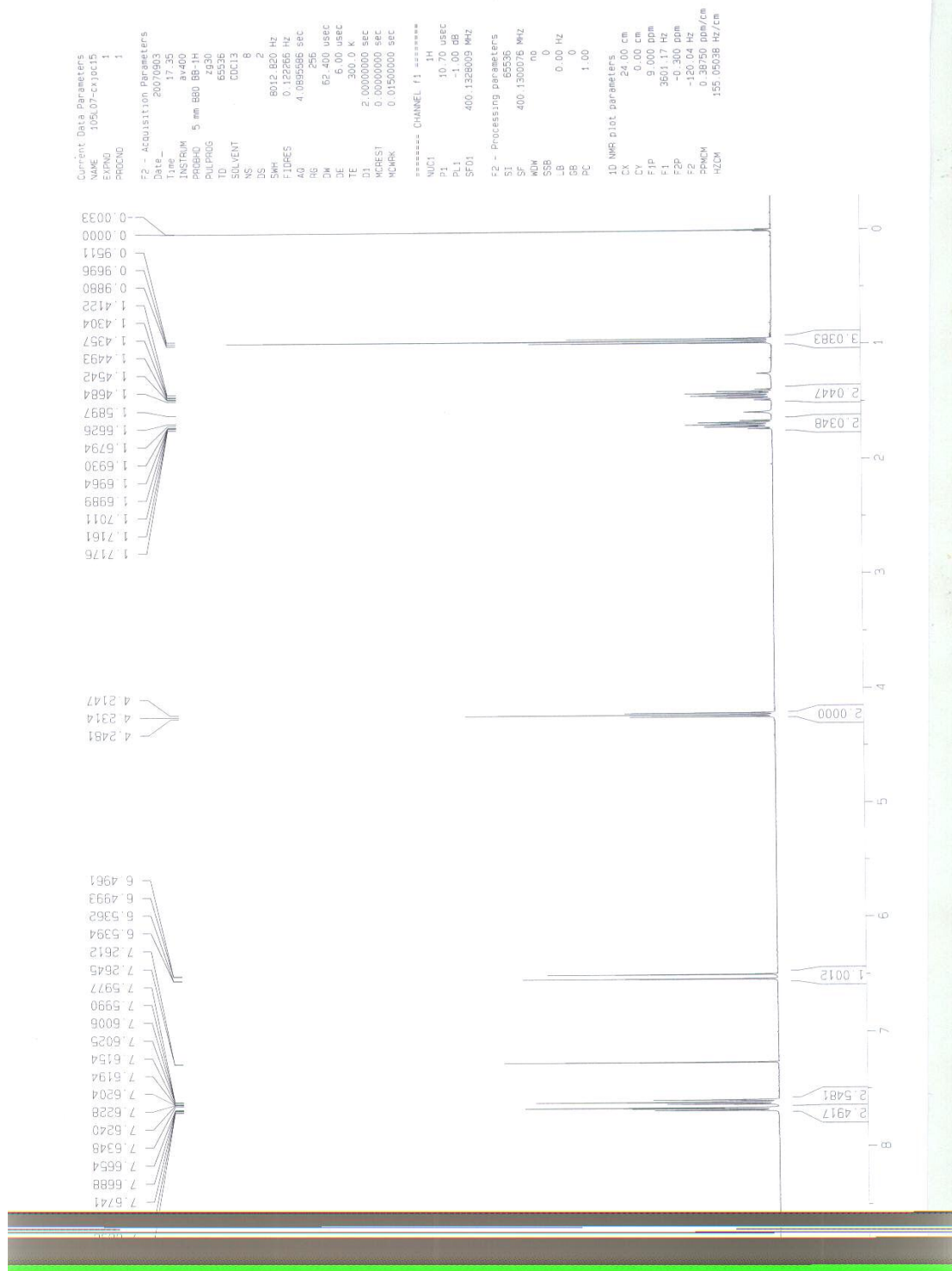
trans-3-(3-cyano-phenyl)-acrylic acid n-butyl ester



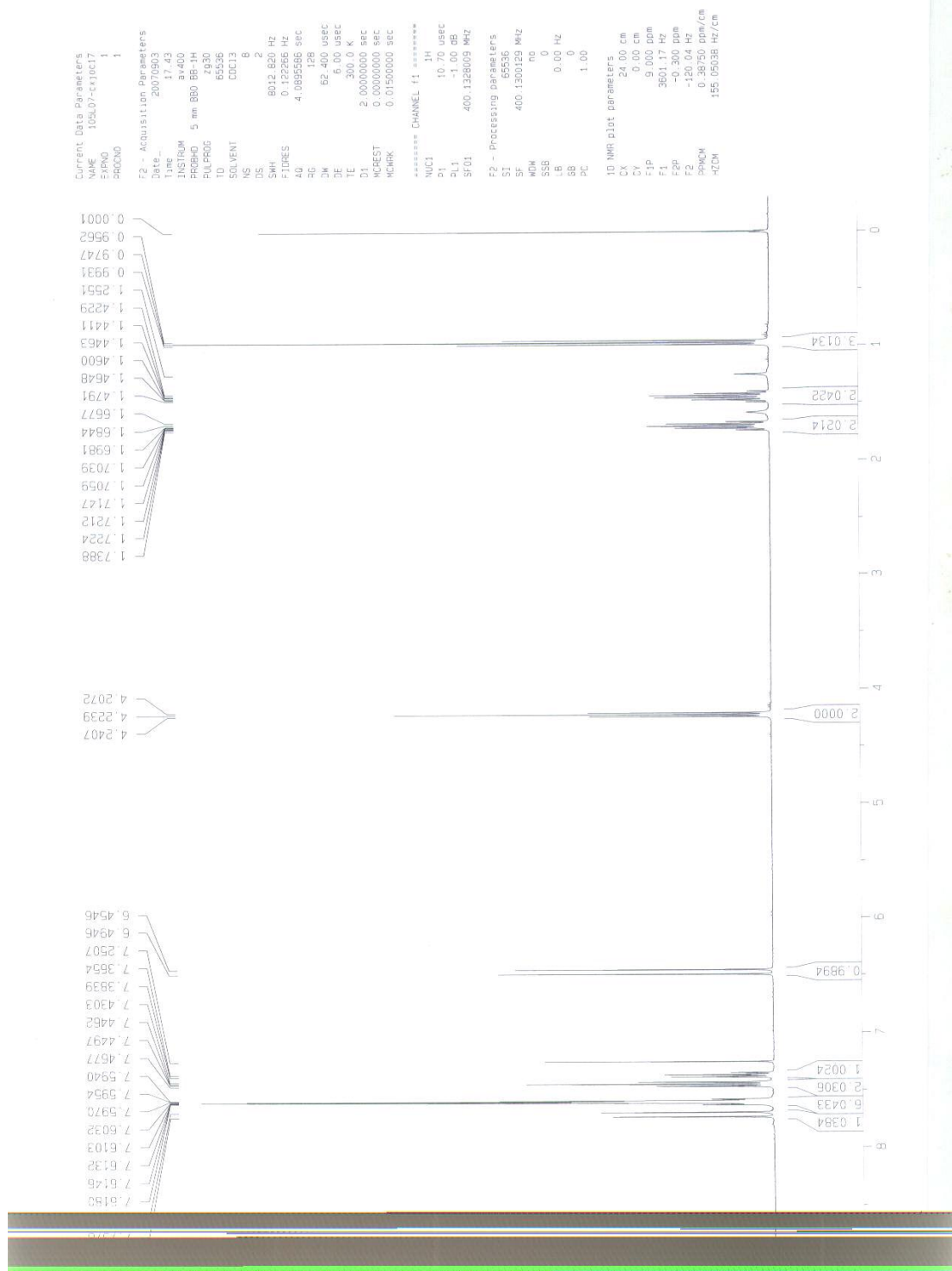
trans-stilbene-4-carbonitrile



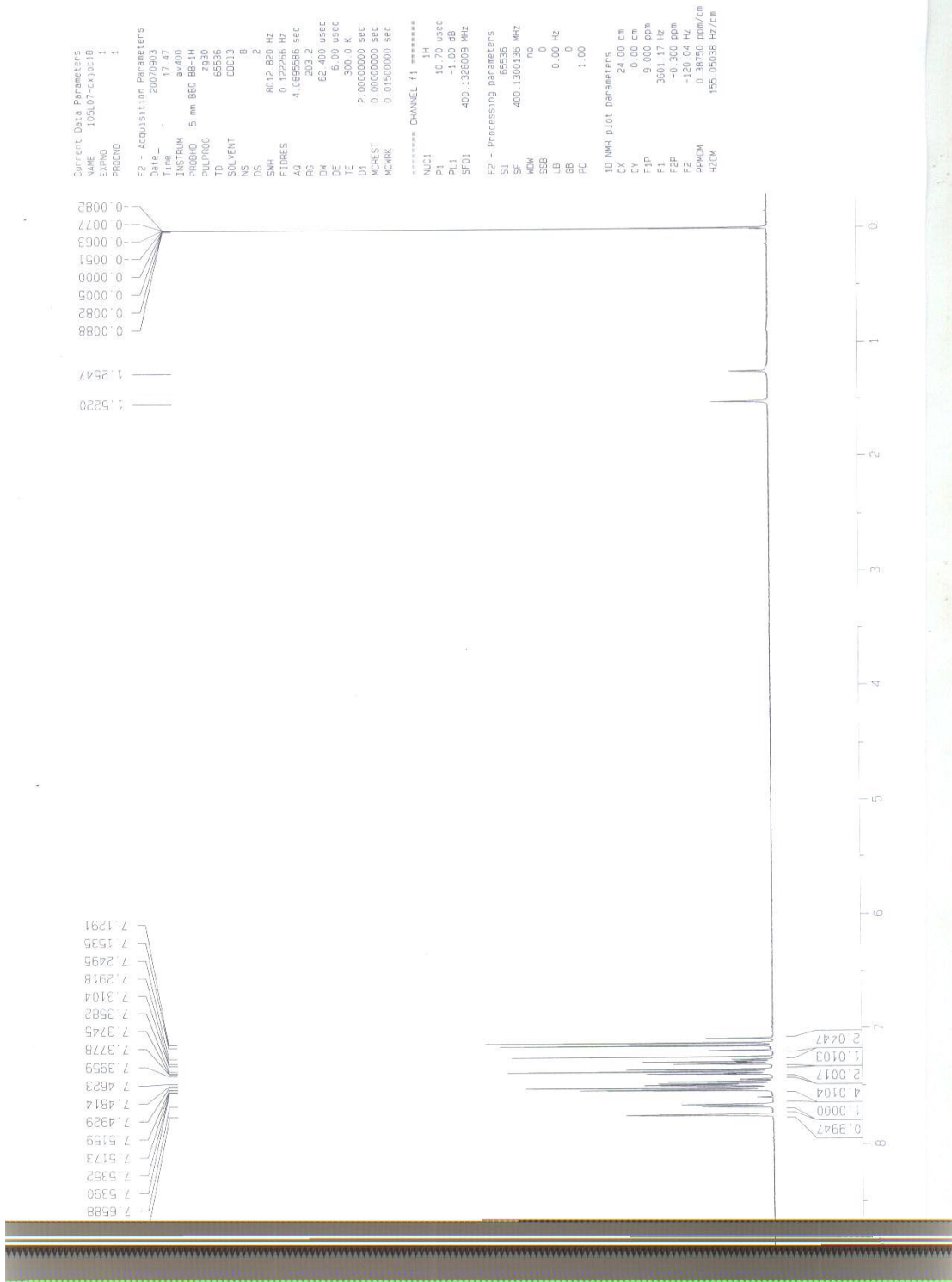
trans-3-(4-cyano-phenyl)-acrylic acid n-butyl ester



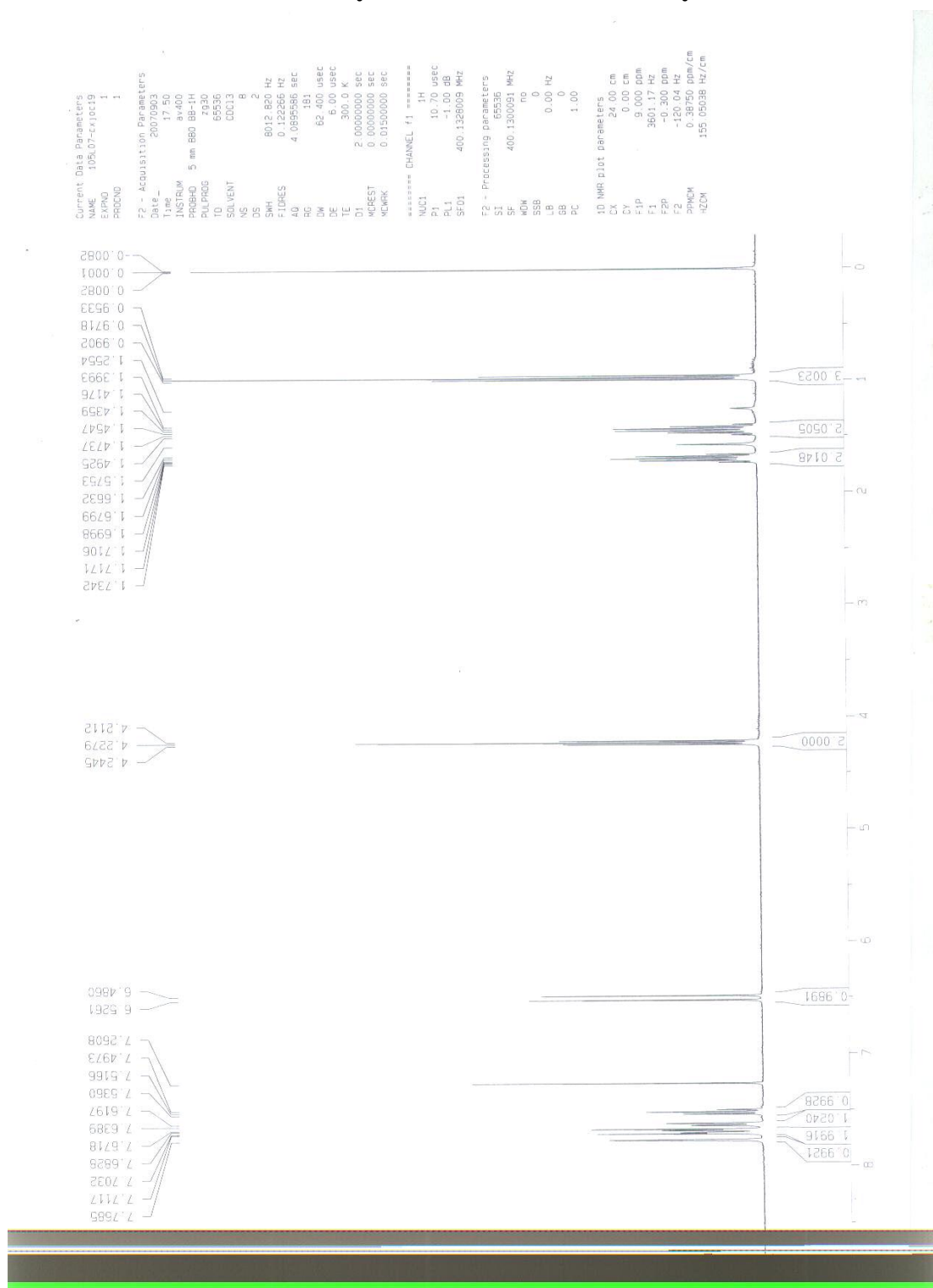
4-phenyl-*trans*-cinnamic acid *n*-butyl ester



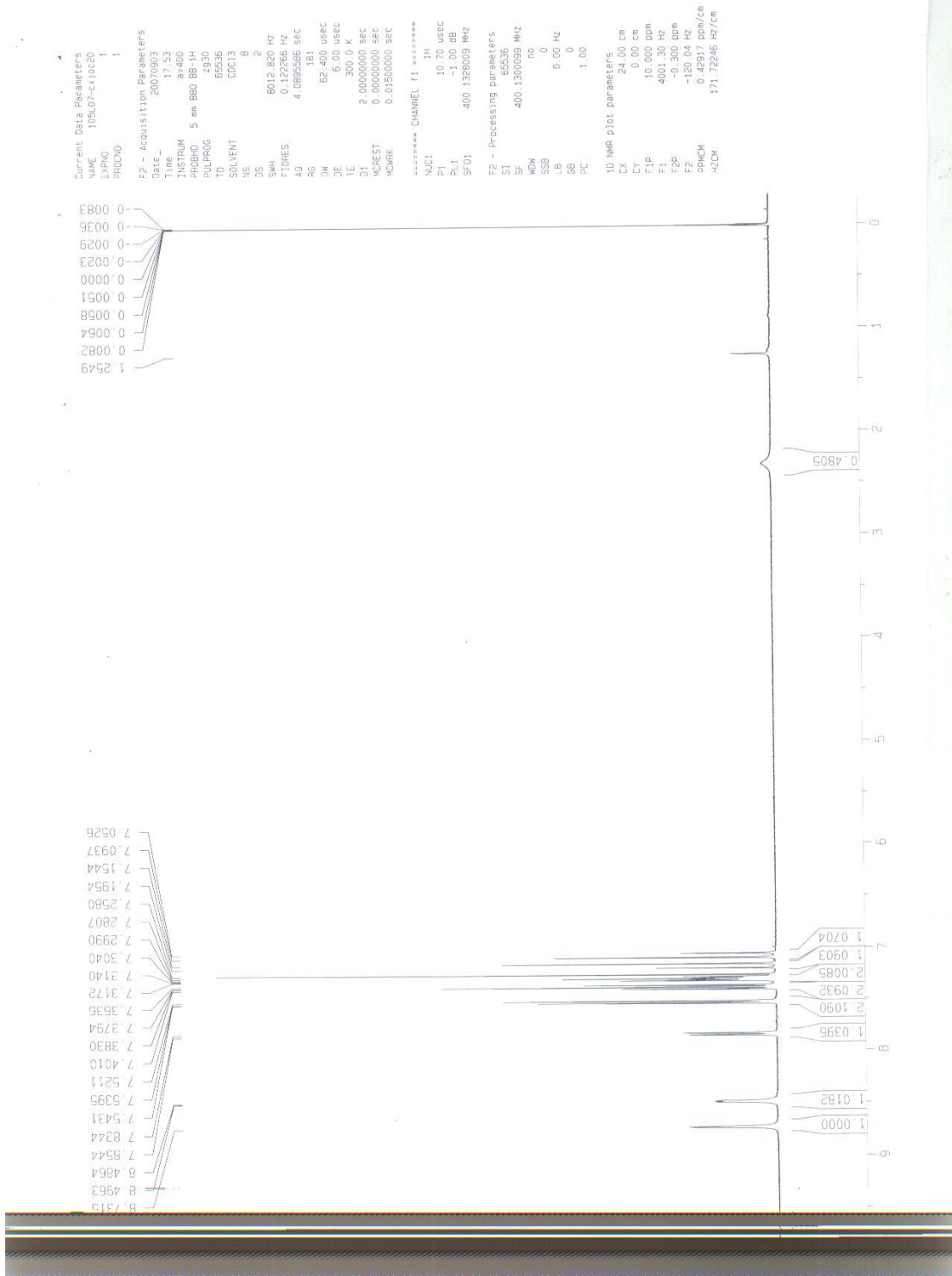
3-trifluoromethyl-*trans*-stilbene



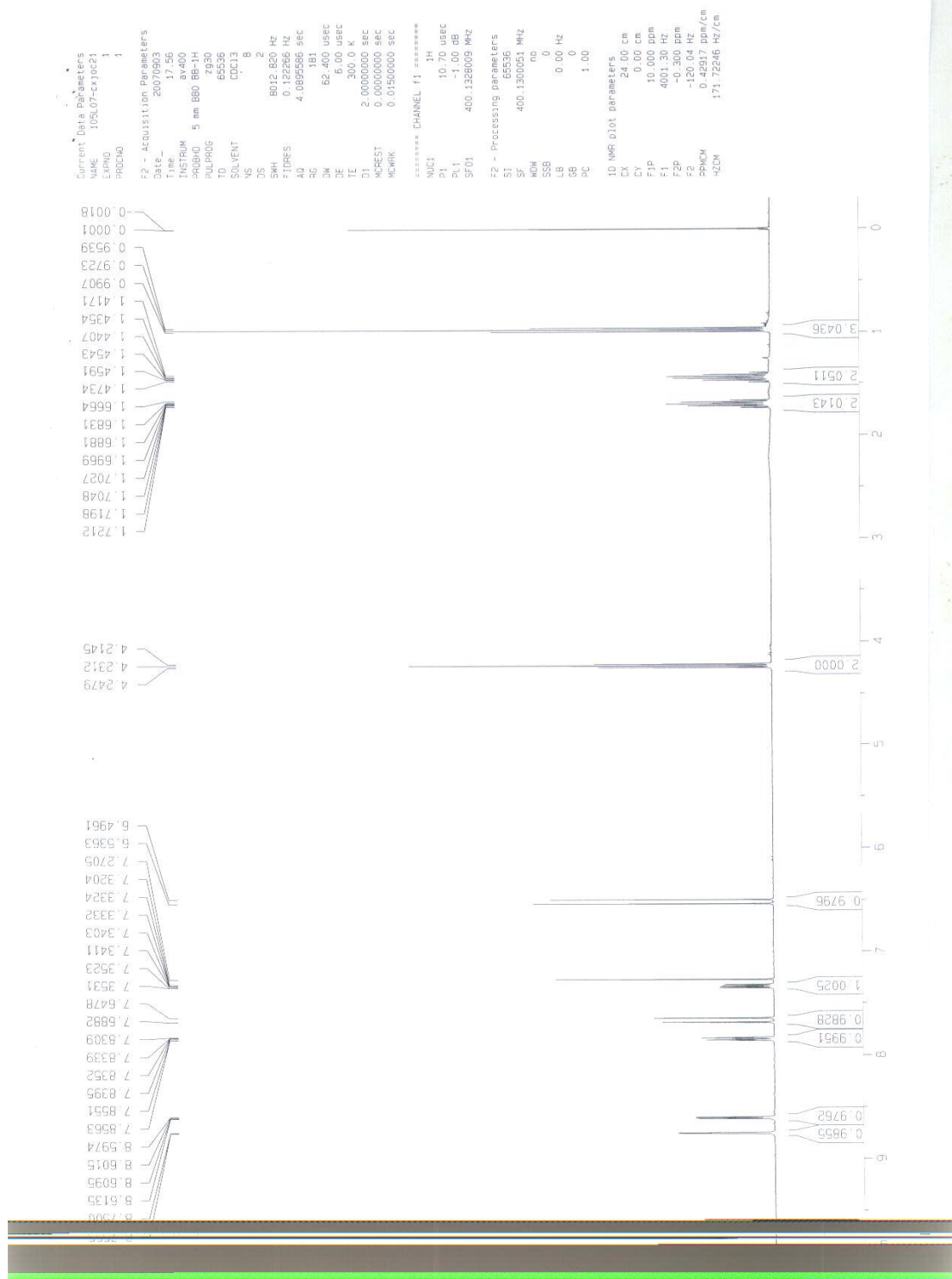
3- trifluoromethyl-*trans*-cinnamic acid *n*-butyl ester



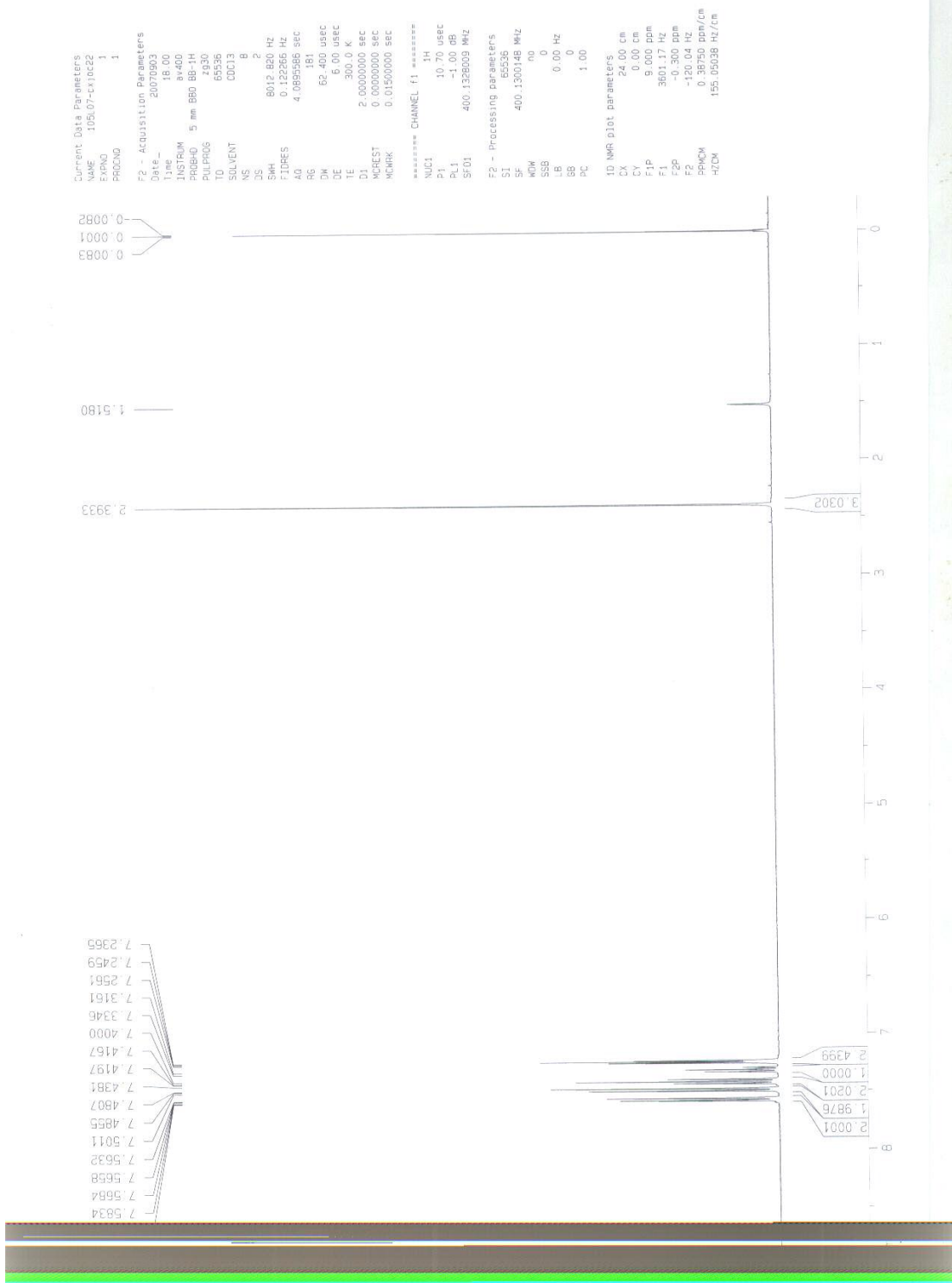
3-trans-styryl-pyridine



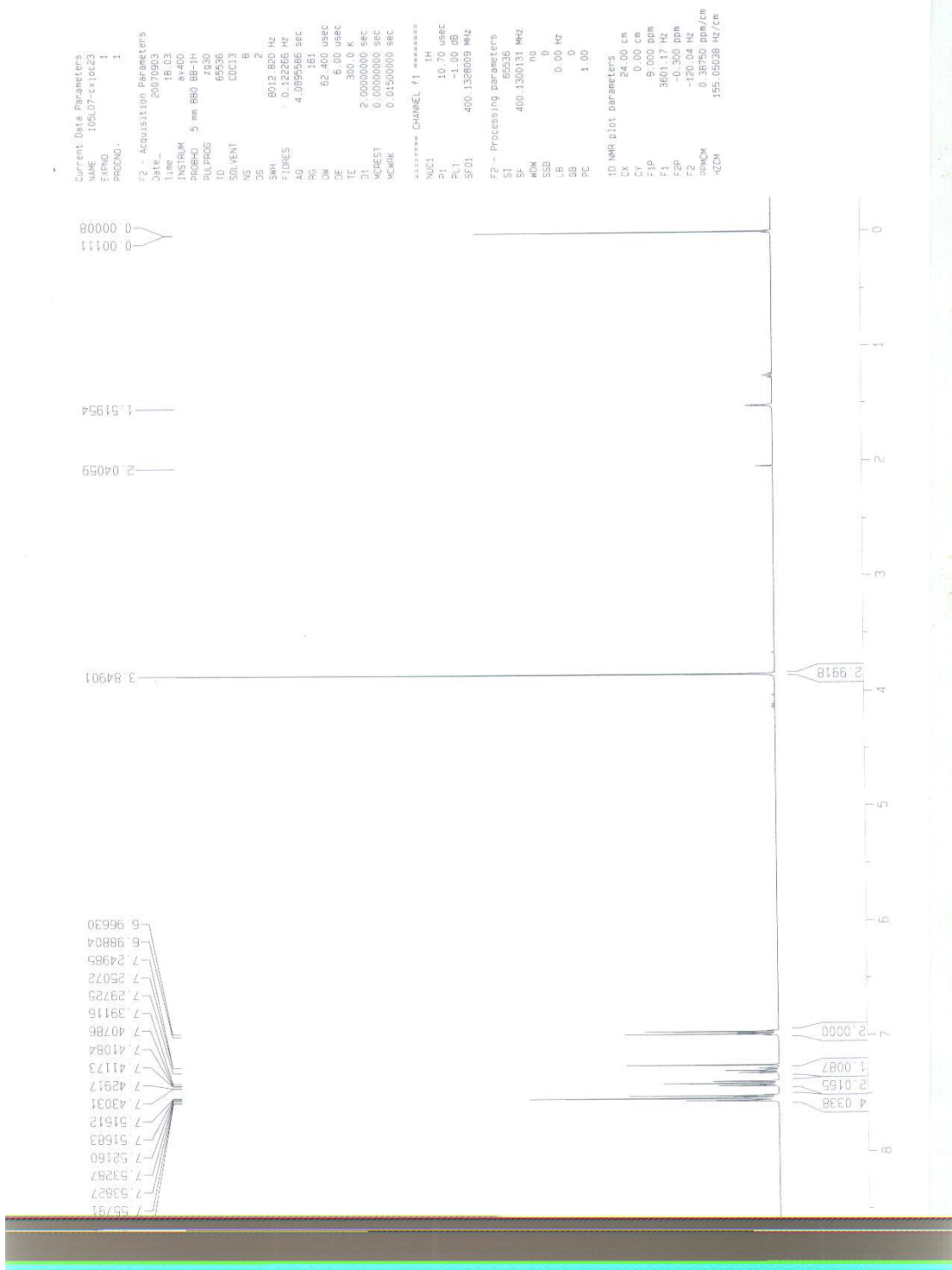
3-pyridin-3-yl-acrylic acid butyl ester



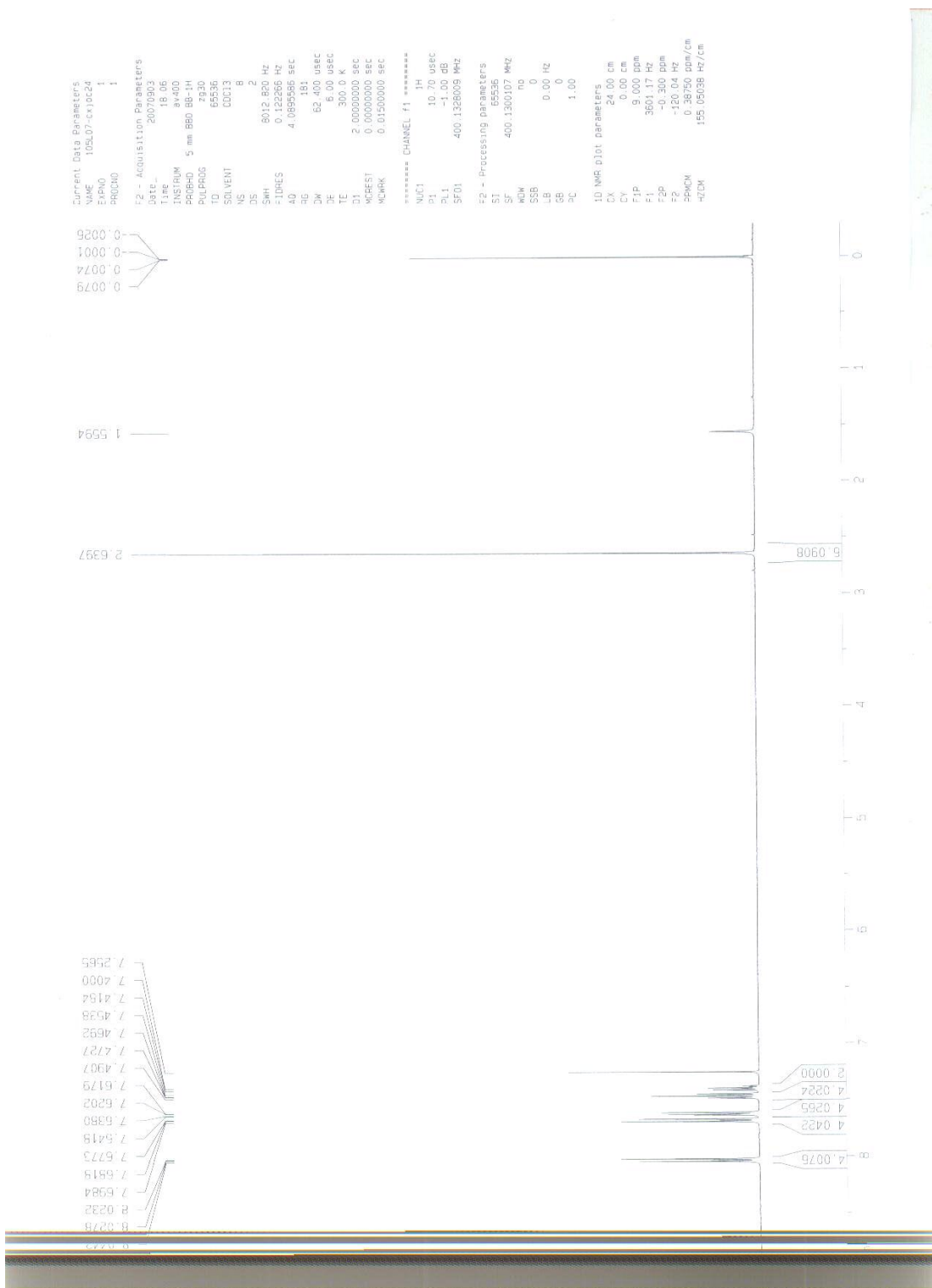
4-Methylbiphenyl



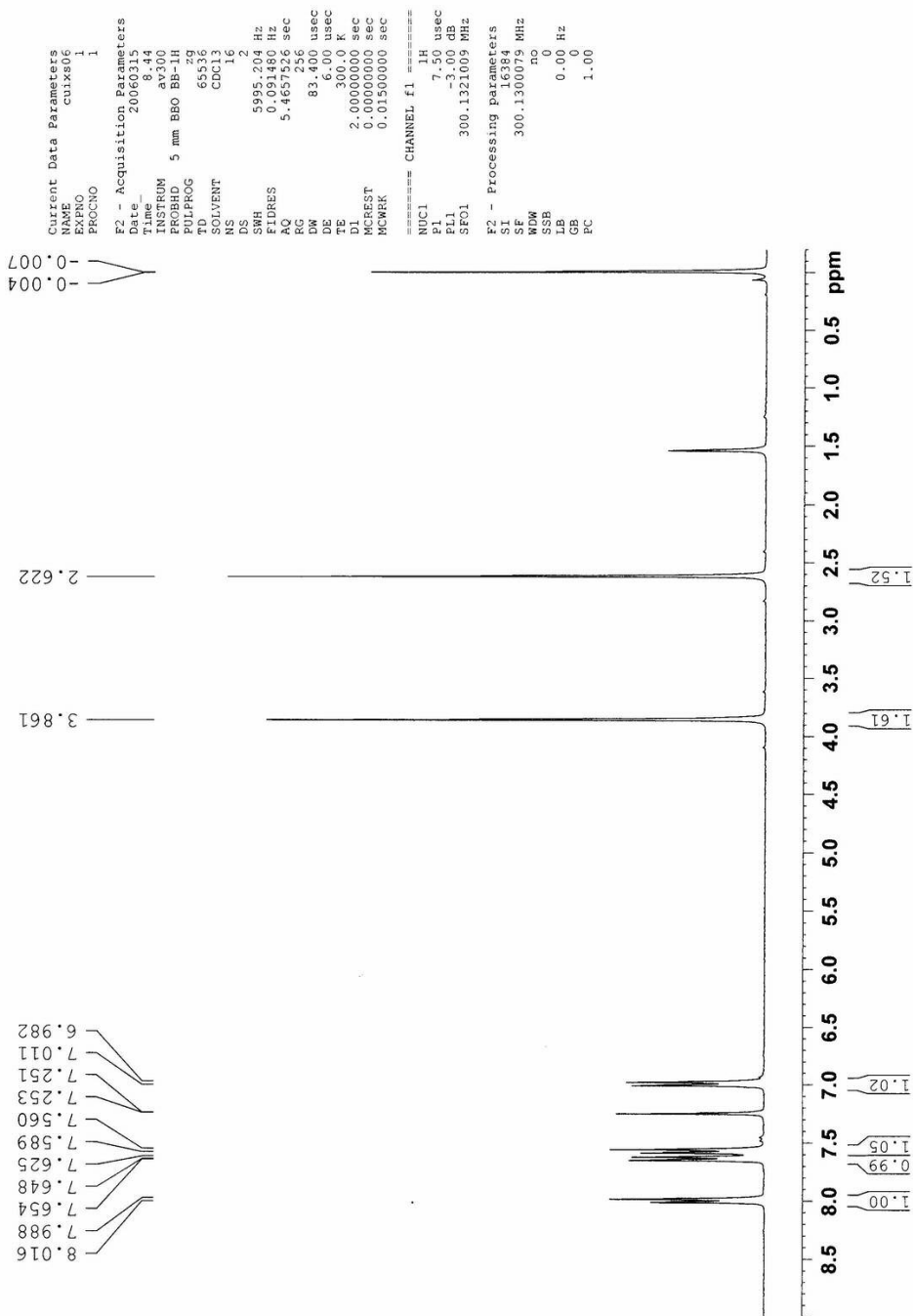
4-Methoxybiphenyl



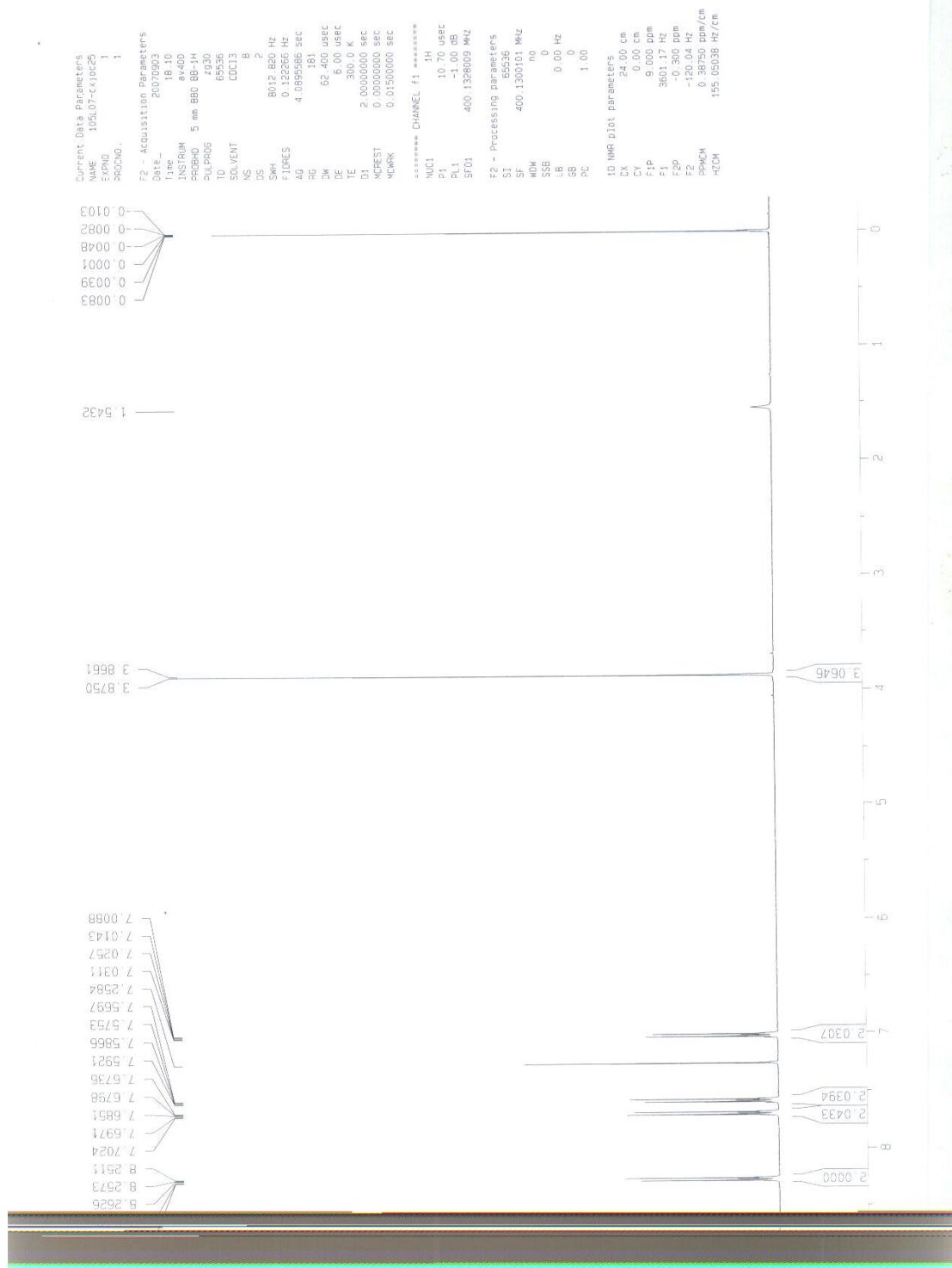
4-Acetylbiphenyl



4-Methoxy-4'-acetylbiphenyl



4-Methoxy-4'-nitrophenyl



4-Nitrophenyl

