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for the Preparation
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In some articles in *Organic Syntheses*, chemical-specific hazards are highlighted in red "Caution Notes" within a procedure. It is important to recognize that the absence of a caution note does not imply that no significant hazards are associated with the chemicals involved in that procedure. Prior to performing a reaction, a thorough risk assessment should be carried out that includes a review of the potential hazards associated with each chemical and experimental operation on the scale that is planned for the procedure. Guidelines for carrying out a risk assessment and for analyzing the hazards associated with chemicals can be found in Chapter 4 of Prudent Practices.

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September 2014: The paragraphs above replace the section "Handling and Disposal of Hazardous Chemicals" in the originally published version of this article. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

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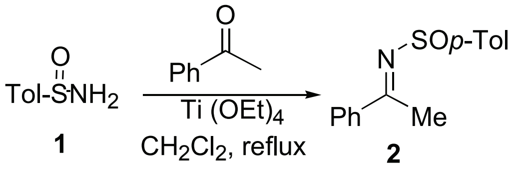
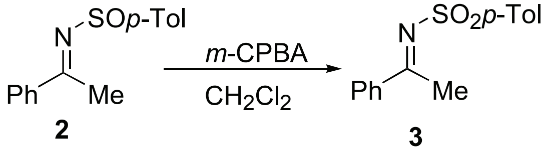
PREPARATION OF *N-p*-TOLYLSULFONYL-(*E*)-1-PHENYLETHYLIDENEIMINE

A.



B.



Submitted by José Luis García Ruano, José Alemán, Alejandro Parra, and M. Belén Cid.[1]
Checked by Kounosuke Oisaki and Masakatsu Shibasaki.

1. Procedure

Caution! Reactions and subsequent operations involving peracids and peroxy compounds should be run behind a safety shield. Peroxy compounds should be added to the organic material, never the reverse. For relatively fast reactions, the rate of addition of the peroxy compound should be slow enough so that it reacts rapidly and no significant unreacted excess is allowed to build up. The reaction mixture should be stirred efficiently while the peroxy compound is being added, and cooling should generally be provided since many reactions of peroxy compounds are exothermic. New or unfamiliar reactions, particularly those run at elevated temperatures, should be run first on a small scale. Reaction products should never be recovered from the final reaction mixture by distillation until all residual active oxygen compounds (including unreacted peroxy compounds) have been destroyed. Decomposition of active oxygen compounds may be accomplished by the procedure described in Korach, M.; Nielsen, D. R.; Rideout, W. H. Org. Synth. ***1962****, 42, 50 (Org. Synth.* ***1973****, Coll. Vol. 5, 414). [Note added January 2011].*

A. N-p-Tolylsulfinyl-(E)-1-phenylethylideneimine (**2**). An oven-dried 300-mL two-necked round-bottomed flask, equipped with a reflux condenser, a magnetic stirring bar, and a rubber septum, is charged with *N*-*p*-toluenesulfinamide **1** (Note 1) (5.2 g, 33.5 mmol, 1.0 equiv), acetophenone (8.0 mL, 68.5 mmol, 2.0 equiv) (Note 2), and $Ti(OEt)_4$ (35 mL, 168 mmol, 5.0 equiv) (Note 3) by temporarily removing the septum. A vacuum adaptor is attached to the condenser and the reaction mixture is stirred under vacuum (1 mmHg) for 15 min. A balloon of argon is attached to the adapter at the top of the condensor. The solid is dissolved in CH_2Cl_2 (75 mL), which is added via syringe through the septum (Note 4). The solution is heated under reflux for 36 h (external bath temperature 80 °C and internal temperature 40 °C), and the reaction is monitored by TLC (Note 5). After cooling the solution to 0 °C using an ice bath, MeOH (50 mL), CH_2Cl_2 (50 mL) and a saturated solution of $NaHCO_3$ (7 mL) are sequentially added with vigorous stirring until precipitation of titanium salts is observed. The solid is vacuum filtered (25 mmHg) through a Büchner funnel (Note 6) and washed with CH_2Cl_2 (3 x 75 mL). The organic phase is dried over anhydrous Na_2SO_4, filtered into a single-necked, 500-mL round-bottomed flask and concentrated by rotary evaporation (25 °C, 10 mmHg) to afford a yellow oil, which is charged onto a silica gel column (8 cm x 18 cm) (Note 7). While collecting fractions of 50 mL, the column is eluted with AcOEt/hexanes (10:90) until acetophenone has completely emerged (volume~~s~~ of eluents used = 0.6 L). The elution is then continued with AcOEt/hexanes (50:50). The product-containing fractions (total volume of eluent used for this product = 1.7 L) are concentrated and successively recrystallized from CH_2Cl_2/hexanes to afford *N*-sulfinylketimine **2** (5.78 g, 67%) as a white solid (Notes 8 and 9).

B. N-p-Tolylsulfonyl (E)-1-phenylethylideneimine (**3**). Into a round-bottomed 300-mL flask equipped with a magnetic stirring bar is added *N-p*-tolylsulfinylketimine **2** (5.2 g, 20 mmol, 1.0 equiv) and CH_2Cl_2 (75 mL) (Note 10). The solution is cooled to 0 °C (Note 11) and commercially available *m*-CPBA (4.5 g, 20 mmol, 1.0 equiv) (Note 12) is added slowly (portions of 1.0 g per min) to the solution, which is open to the atmosphere. The mixture is allowed to warm to room temperature and the reaction is monitored by TLC (Note 13). After five min, the mixture is diluted with CH_2Cl_2 (50 mL), transferred to a separatory funnel, washed with a saturated solution of $NaHCO_3$ (5 x 25 mL) (Note 14), and the organic phase is dried over anhydrous Na_2SO_4. The solution is filtered into a single-necked, 300-

mL round-bottomed flask and concentrated by rotary evaporation (25 °C, 10 mmHg) to afford crude **3** as a white solid, which is diluted with 7 mL of CH_2Cl_2 and charged on a silica gel column (150 g, 5.5 cm x 18 cm) (Note 7). The column is eluted initially with AcOEt/hexanes (10:90) until the *N*-sulfinylketimine **2** emerges, and then the elution is then continued with AcOEt/hexanes (10:50). The product-containing fractions are concentrated to afford *N*-sulfonylketimine **3** (4.23 g, 77%) as a white solid (Notes 14, 15 and 16).

2. Notes

1. *N*-Sulfinamide **1** could be purchased from Aldrich Chemical Company, Inc. in optically pure form. However, (±)-**1** can be obtained at much lower cost following a known procedure,[11] which involves two steps from *p*-tolyldisulfide.
2. Acetophenone, 99% was purchased from Aldrich Chemical company, Inc. and was used without further purification.
3. Titanium (IV) ethoxide, technical grade was purchased from Aldrich Chemical Company, Inc. and was used without further purification.
4. No difference in the yield was observed when using either dried CH_2Cl_2 (from P_2O_5) or the directly purchased CH_2Cl_2 from SDS (purex analytical grade).
5. The reaction was monitored by TLC on silica gel using hexanes/EtOAc (2:1) as eluent and visualization with phosphomolibdic acid. The starting material **1** had $R_f = 0.1$ (green), acetophenone had $R_f = 0.77$ (green), and the final product **2** had $R_f = 0.38$ (brown).
6. A double filter paper of medium porosity was used.
7. Silica gel (230–400 mesh) from Merck was used.
8. The physical properties of *N*-sulfinylketimine **2** were as follows: white solid, mp 95.0–96.0 ºC (lit.[2] mp 99–100 ºC). ^{1}H NMR (500 MHz) δ: 2.40 (s, 3 H) 2.78 (s, 3 H), 7.32 (d, *J* = 8.2 Hz, 2 H), 7.39 (t, *J* = 8.0 Hz, 2 H), 7.47 (t, *J* = 6.7 Hz, 1 H), 7.72 (d, *J* = 8.2 Hz, 2 H), 7.88 (d, *J* = 7.6 Hz, 2 H), ^{13}C NMR (125.7 MHz) δ: 20.2, 21.4, 125.2, 127.5, 128.4, 129.8, 131.8, 138.2, 141.8, 143.3, 173.9; IR (KBr, cm^{-1}): 3430, 1637, 1565, 1096; LRMS (ESI-MS) *m/z* 258 $(M+H)^+$, 280 $(M+Na)^+$; HRMS (FAB-MS) calcd for $C_{15}H_{16}NOS$ $(M+H)^+$ 258.0947, found 258.1008.

9. *N*-Sulfinylketimine **2** and *N*-sulfonylketimine **3** are hydrolyzed upon extended contact with silica gel; therefore the flash chromatography should be carried out rapidly.

10. In this case, dry CH_2Cl_2 (distilled from P_2O_5) was used, since traces of water can hydrolyze the starting material **2** or *N*-sulfonylketimine **3**.

11. This reaction was exothermic, therefore the addition should be carried out at 0 °C. Without an ice bath (0 °C) an increase in temperature from 21 to 40 °C was detected.

12. 3-Chloro-perbenzoic acid (>77 %) was purchased from Aldrich Chemical Company, Inc. (Lot number S22939 was used, which contained 18.03 % of water and 71.70 % of 3-chloroperbenzoic acid). The reagent was directly used without removal of the water, which resulted in a 78% yield. The yield of the reaction could be increased to 84% when water was removed from this reagent. The procedure for removing water was as follows: Prior to its use, water was removed by dissolving the commercial product in CH_2Cl_2, drying the organic solution over anhydrous Na_2SO_4, filtering and concentrating the reagent to dryness under vacuum. Titration following the procedure reported in the literature (McDonald, R. N.; Steppel, R. N.; Dorsey, J. E. *Org. Synth.* **1970***, 50*, 15) indicated a purity of 90% (using $Na_2S_2O_3$ 0.1004 N). A purity of 91% was obtained when the purity determination was carried out by HPLC: performed on ZORBAX RX-C8 (4.6 mm x 25 cm) with 50:50 water:methanol, with a flow rate of 0.5 mL/min at 210 nm (3-chlorobenzoic acid t_R= 5.5 min, 3-Chloroperbenzoic acid t_R= 21.5 min). When a calibration standard was used (3-chlorobenzoic acid at different concentrations) under these conditions, the purity was determined to be 90%.

13. The reaction was monitored by TLC on silica gel using hexanes/EtOAc (2:1) as eluent and visualization with a phosphomolibdic acid. The starting material **2** has an R_f= 0.38 while R_f= 0.70 for the final product **3**.

14. *N*-Sulfonylketimine **3** is hydrolyzed upon extended contact with water; therefore, the washes should be carried out rapidly.

15. The physical properties of *N*-sulfonylketimine **3** were as follows: white solid, mp 88.5–90.5 ºC (lit.[10k] mp 88–90 ºC). ^{1}H NMR (500 MHz) δ: 2.45 (s, 3 H) 2.99 (s, 3 H), 7.34 (d, *J* = 8.2 Hz, 2 H), 7.41 (t, *J* = 8.0 Hz, 2H), 7.53 (t, *J* = 7.2 Hz, 1H), 7.90 (d, *J* = 8.2 Hz, 2 H), 7.93 (d, *J* = 8.0 Hz, 2 H); ^{13}C NMR (125.7 MHz, $CDCl_3$) δ: 21.1, 21.5, 127.0, 128.2, 128.6,

129.4, 133.1, 137.5, 138.7, 143.5, 179.8; IR (KBr, cm^{-1}): 3435, 1590, 1570, 1286, 1180, 1083; LRMS (ESI-MS) *m/z* 274 (M+H)$^+$, 296 (M+Na)$^+$; HRMS (FAB-MS) calcd for $C_{15}H_{16}NO_2S$ (M+H)$^+$ 274.0896, found 274.0775. The purity of the sample was determined by HPLC (>98%), performed on Chiral AS (Daicel) with 60:40 hexane:2-propanol and a flow rate of 0.6 mL/min.

16. The *N*-sulfonylketimine **3** (0.007 mol, 2 g) could be crystallized from Et_2O (80 mL). The solution was heated until the solid dissolved, and filtered through filter paper of medium porosity. The solution was kept for 12 h at –20 °C. White needles were collected using a Büchner funnel under vacuum (25 mmHg), which resulted in the isolation of 1.90 g of pure **3** (95% yield).

Safety and Waste Disposal Information

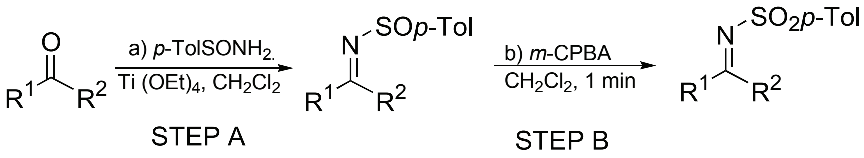
All hazardous materials should be handled and disposed of in accordance with "Prudent Practices in the Laboratory"; National Academy Press; Washington, DC, 1995.

3. Discussion

As the sulfonyl moiety has proven to be a powerful activating group of iminic C=N bonds, *N*-sulfonyl imines have been widely used in organic synthesis.[3] They are excellent substrates in aza Diels-Alder reactions,[4] nucleophilic additions,[5] reductions,[6] as well as in radical[7] or ene[8] reactions. They have been also used in the synthesis of aziridines.[9]

Despite the plethora of methods reported for obtaining *N*-sulfonyl imines,[10] most of the methods are only efficient in preparing those compounds derived from aromatic or non-enolizable aldehydes. Therefore, it was necessary to find a new general protocol for synthesizing any *N*-sulfonyl aldimine or ketimine, even those containing enolizable α-hydrogens. We have recently reported a new procedure that fulfills all of these requirements.[11] The procedure involves the condensation of carbonyl compounds with *p*-tolyl (Table 1) and *tert*-butyl (Table 2) sulfinamides followed by oxidation of the resulting *N*-sulfinylimines with *m*-CPBA. The method is applicable to aldehydes (aliphatics and aromatics) and ketones (dialkyl, diaryl, and alkyl aryl), even those containing enolizable protons. The configurations of α-stereogenic centers are not affected and the method is highly selective in the presence of C=N and C=C (deactivated or not) double bonds. The new procedure has been applied to the preparation of compounds collected in Table 1. The main advantages of this procedure derive from the near absence of structural limitations for the starting carbonyls used for preparing sulfinylimines, as well as the higher stability of these sulfinylimines (they can be stored without significant decomposition for long periods of time) with respect to their corresponding sulfonylimines.

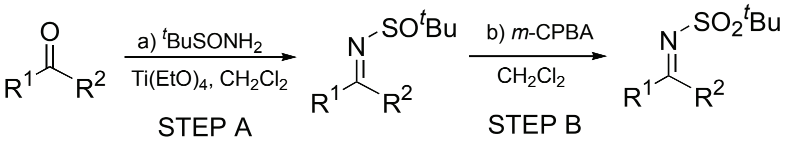
Table 1. Preparation of *N*-*p*-Tolylsulfonylimines.



Entry	R^1	R^2	Reaction Yields (%)		
			Step A	Step B	Overall
1	Ph	H	93	100	93
2	3-$MeOC_6H_4$	H	92	100	92
3	2-Napht	H	94	95	89
4	2-BrC_6H_4	H	91	100	91
5	4-CNC_6H_4	H	81	85[a]	69
6	$PhCH=CH_2$	H	80	95	76
7	*n*-Pr	H	78	96[b]	75
8	*i*-Pr	H	65	100[b]	65
9	*t*-Bu	H	79	96	76
10	Ph	Ph	79	88	69
11	4-$MeOC_6H_4$	Me	73	84	61
12	4-CNC_6H_4	Me	77	90	69
13	*t*-Bu	Me	48	90	43
14	*i*-Pr	Me	64	90	58

[a] In this case the reaction time was 15 min. [b] To avoid oxaziridine formation, this reaction was carried out at 45 ºC.

Table 2. Preparation of *N*-*t*-Butylsulfonylimines.



Entry	R^1	R^2	Reaction yields (%)		
			Step A	Step B	Overall
1	Ph	H	96	100	96
2	2-furyl	H	63	100	63
3	*n*-Pr	H	100	84	84
4	Ph	Ph	75	100	75
5	Ph	Me	63	100	63
6	4-$MeOC_6H_4$	Me	62	100	62
7	4-CNC_6H_4	Me	73	100	73
8	-$(CH_2)_5$-		65	90	59
9	Et	Me	73[a]	78	57
10	2-pyridyl	Me	59[b]	80	47

[a] A 4:1 mixture of the E/Z isomers was formed. [b] In this case the reaction time was 15 m.

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Appendix
Chemical Abstracts Nomenclature; (Registry Number)

N-*p*-Toluenesulfinamide: Benzenesulfinamide, 4-methyl-; (6873-55-8)
Acetophenone: Ethanone, 1-phenyl-; (98-86-2)
$Ti(OEt)_4$: Ethanol, titanium(4+) salt; (3087-36-3)
N-*p*-Tolylsulfinyl-(*E*)-1-phenylethylideneimine: Benzenesulfinamide, 4-methyl-*N*-(1-phenylethylidene)-, [*N*(*E*)]-; (177468-94-9)
m-CPBA: Benzenecarboperoxoic acid, 3-chloro-; (937-14-4)
N-*p*-Tolylsulfonyl-(*E*)-1-phenylethylideneimine: Benzenesulfonamide, 4-methyl-*N*-(1-phenylethylidene)-, (*E*)-; (163586-87-6)

José Luis García Ruano received his Ph.D. at the Universidad Complutense (Madrid, 1973). He has held appointments as a visiting Professor at the Florida State (1992) and Emory (2003) Universities and received a fellowship with the JSPS in 2006. He was appointed Full Professor in Organic Chemistry at Universidad Autónoma de Madrid in 1982 and has served as Vice-president of the Spanish Royal Society of Chemistry for 4 years. His research interests are centered in the chemistry of sulfoxides and related compounds and their applications in asymmetric synthesis. In these fields, he has published approximately 300 papers and supervised thirty-five Ph.D. theses.

José Alemán received his B.Sc. in Chemistry at the Universidad Autónoma of Madrid (Spain) in 2000. In 2003 he spent six months in the laboratory of Prof. Albert Padwa at Emory University (Atlanta, USA) working on Pummerer rearrangements. In 2005 he received the Lilly Research Award for Ph. D. students and presented his Ph. D. thesis, which focused on remote stereocontrol by sulfinyl groups and was supervised by Prof. Jose Luis García Ruano. He is currently working (2006) as post-doctoral researcher with Prof. Karl Anker Jørgensen (Aarhus, Denmark). His research interests include asymmetric synthesis, sulfur chemistry and organocatalysis.

Alejandro Parra was born in Toledo, Spain. He received a B.S. degree in chemistry at the Universidad Autónoma of Madrid (Spain) in 2004. He is now pursuing his Ph.D. at the Universidad Autónoma de Madrid under the supervision of Prof. José Luis García Ruano. His research concerns asymmetric synthesis with sulfur compounds. He is currently a predoctoral research fellow in the laboratory of Prof. Andrew Myers at Harvard University (USA) working on the chemistry of tetracyclines.

M. Belén Cid was born in Madrid, Spain, in 1967. She received her Ph.D. degree (1995) under the supervision of Professors J. L. García Ruano and M. Carmen Carreño at the Autonoma University of Madrid. She joined the group of Prof. Gerald Pattenden (1996-1999) at the Nottingham University as a postdoctoral fellow. Following two stays in the groups of Prof. Manuel Martín-Lomas (1999-2001) in Seville (CSIC) and Prof. Serafín Valverde (2002-2003) in Madrid (CSIC), she returned to Prof. García Ruano's group in 2004. Her research interests are focused on the development of highly stereoselective methods from sulfur compounds, synthesis of pharmacologically active compounds and asymmetric organocatalysis.

Kounosuke Oisaki was born in 1980 in Tokushima, Japan. He received his B.S. in 2003 and M.S. in 2005 from The University of Tokyo. Presently, he is pursuing his Ph.D. degree at the Graduate School of Pharmaceutical Sciences, The University of Tokyo, under the guidance of Professor Masakatsu Shibasaki. His research interests are in the area of asymmetric synthesis and catalysis.

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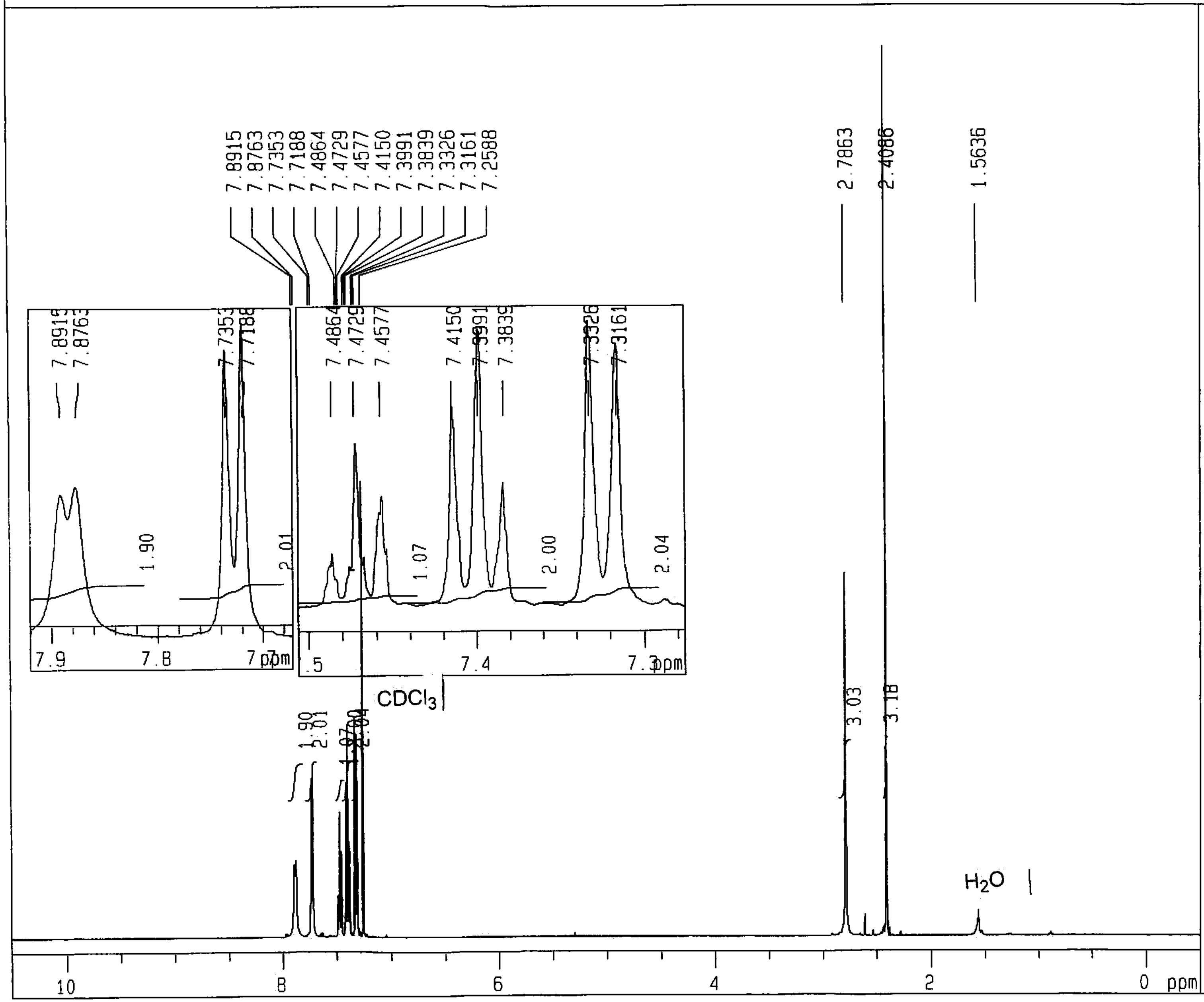

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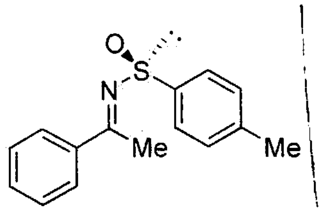

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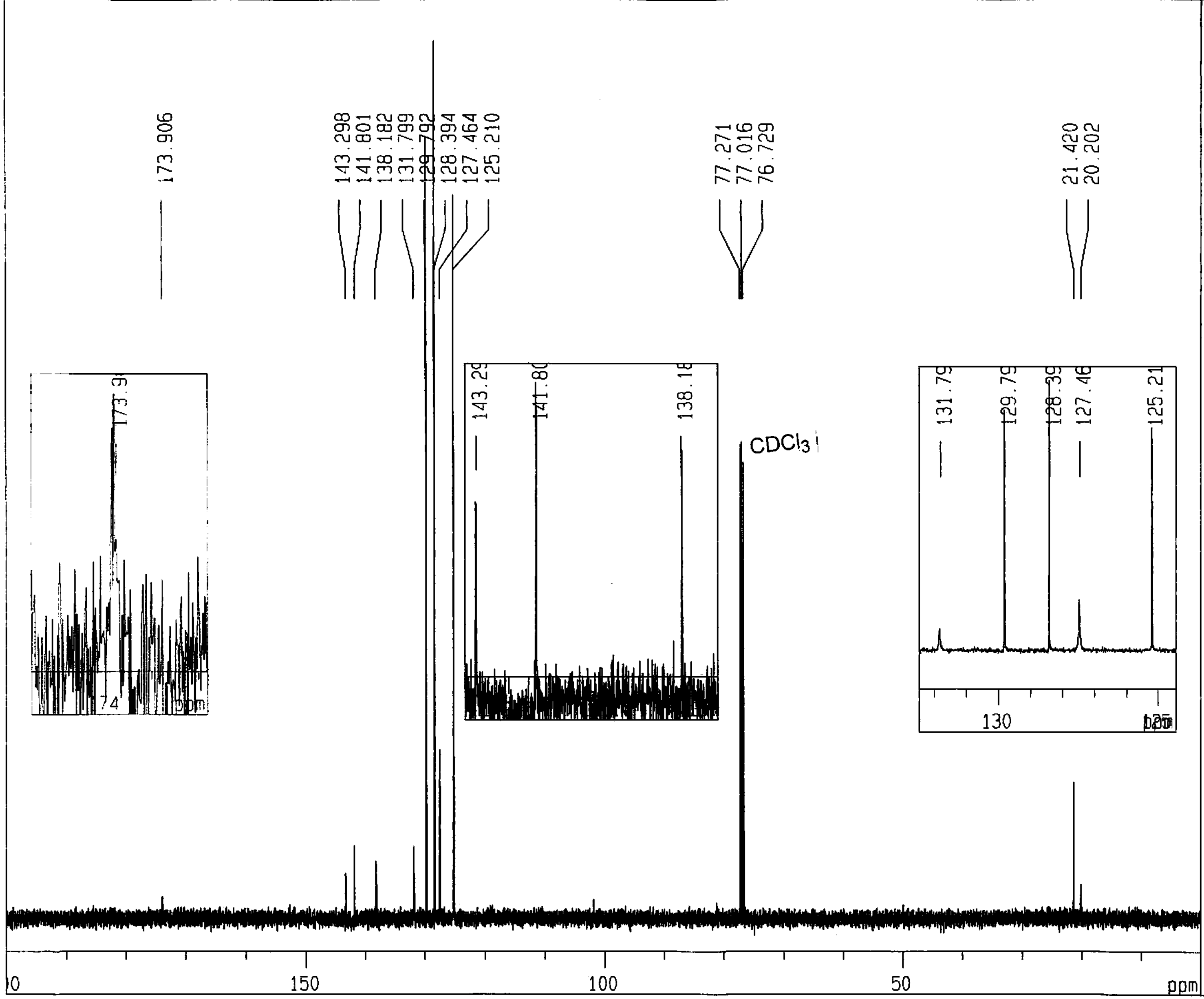

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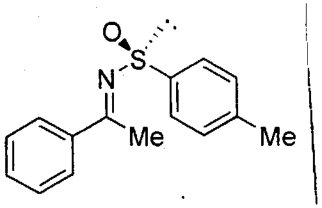
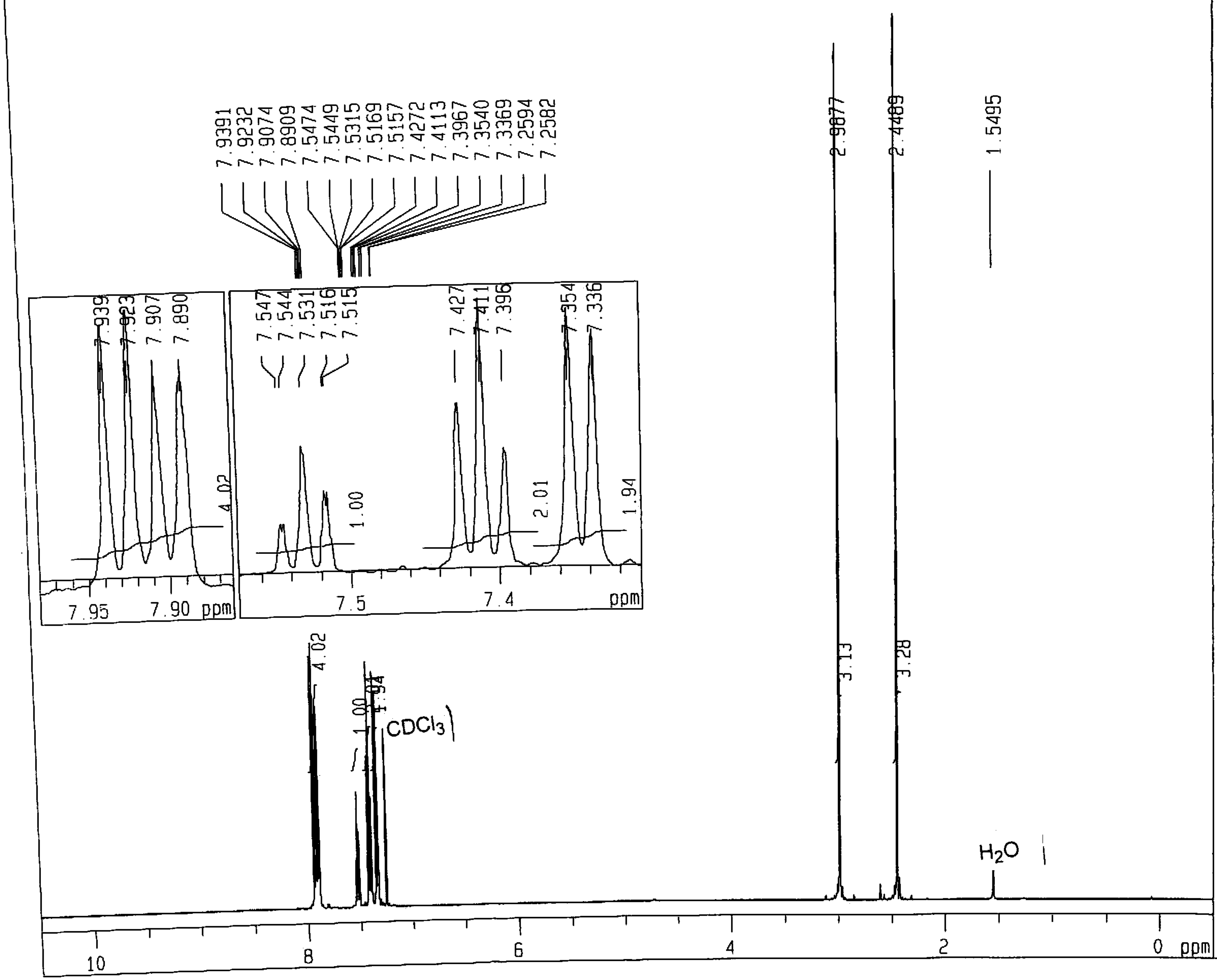
TIMES          :       1024 times
DUMMY          :          0 times
PD             :     2.0333 sec
ACQTM          :   966.6560 msec
PREDL          :    0.01000 msec
INIWT          :  1000.0000 msec
RESOL          :       1.03 Hz
PW1            :       6.25 usec
OBNUC          : 13C
OBFRQ          :     125.65 MHz
OBSET          :  127958.00 Hz
RGAIN          :         31
IRNUC          : 1H
IRFRQ          :     500.00 MHz
IRSET          :  162160.00 Hz
IRRPW          :       50.0 usec
IRRNS          :          0

SCANS          :        237 times

SLVNT          : CDCL3
SPINNING       :         11 Hz
TEMP           :       28.5 C
```



1H Line



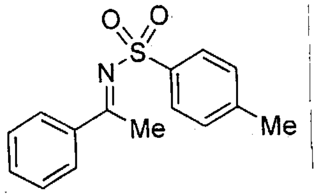
Date : Mon Jul 24 12:20:06 2006

FileName : .LoadingFID.nmdata
Comment : 1H Line
SliceHistory :
EXMODE : non

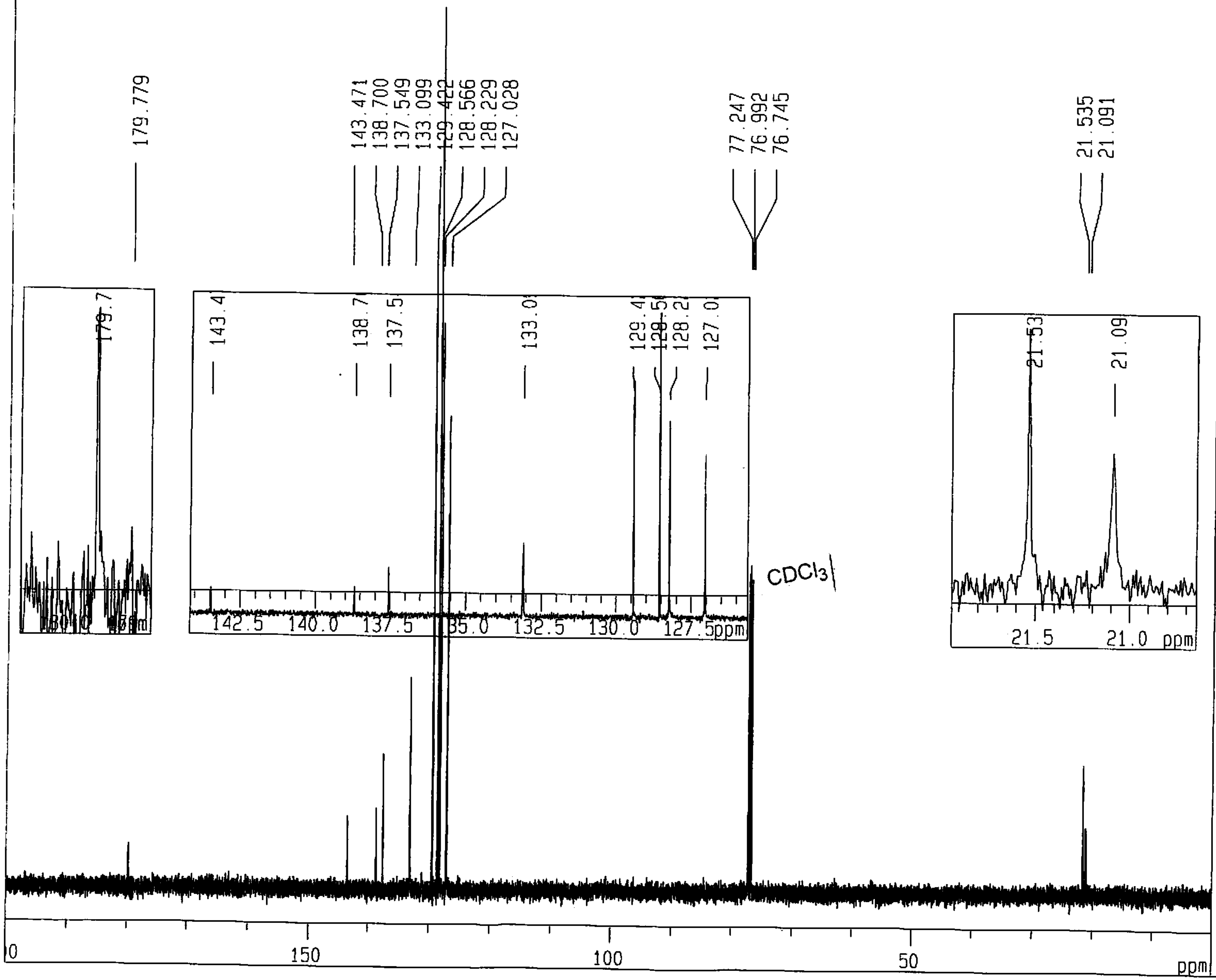
POINT : 32768 points
SAMPO : 32768 points
FREQU : 10000.0 Hz
FILTR : 5000 Hz
DELAY : 40.0 usec
DEADT : 57.1 usec
INTVL : 100.0 usec
TIMES : 16 times
DUMMY : 1 times
PD : 3.7232 sec
ACQTM : 3276.7998 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.31 Hz
PW1 : 5.70 usec
OBNUC : 1H
OBFRQ : 500.00 MHz
OBSET : 162160.00 Hz
RGAIN : 20

SCANS : 4 times

SLVNT : CDCL3
SPINNING : 12 Hz
TEMP : 30.7 C



1H Line



```
Date :  Tue Jul 11 13:13:04 2006

FileName      :  .LoadingFID.nmdata
Comment       :  1H Line
SliceHistory  :
EXMODE        :  bcm

POINT         :       32768 points
SAMPO         :       32768 points
FREQU         :     33898.3 Hz
FILTR         :       16950 Hz
DELAY         :        11.8 usec
DEADT         :        10.0 usec
INTVL         :        29.5 usec
TIMES         :        1024 times
DUMMY         :           0 times
PD            :      2.0333 sec
ACQTM         :    966.6560 msec
PREDL         :     0.01000 msec
INIWT         :   1000.0000 msec
RESOL         :        1.03 Hz
PW1           :        6.25 usec
OBNUC         :  13C
OBFRQ         :      125.65 MHz
OBSET         :   127958.00 Hz
RGAIN         :          30
IRNUC         :  1H
IRFRQ         :      500.00 MHz
IRSET         :   162160.00 Hz
IRRPW         :        50.0 usec
IRRNS         :           0

SCANS         :          66 times

SLVNT         :  CDCL3
SPINNING      :          10 Hz
TEMP          :        28.4 C
```