Supporting Information

Molecular Design and Polymer Structure Control Based On Polymer Crystal Engineering. Topochemical Polymerization of 1,3-Diene Mono- and Dicarboxylic Acid Derivatives Bearing a Naphthylmethylammonium Group as the Counter Cation

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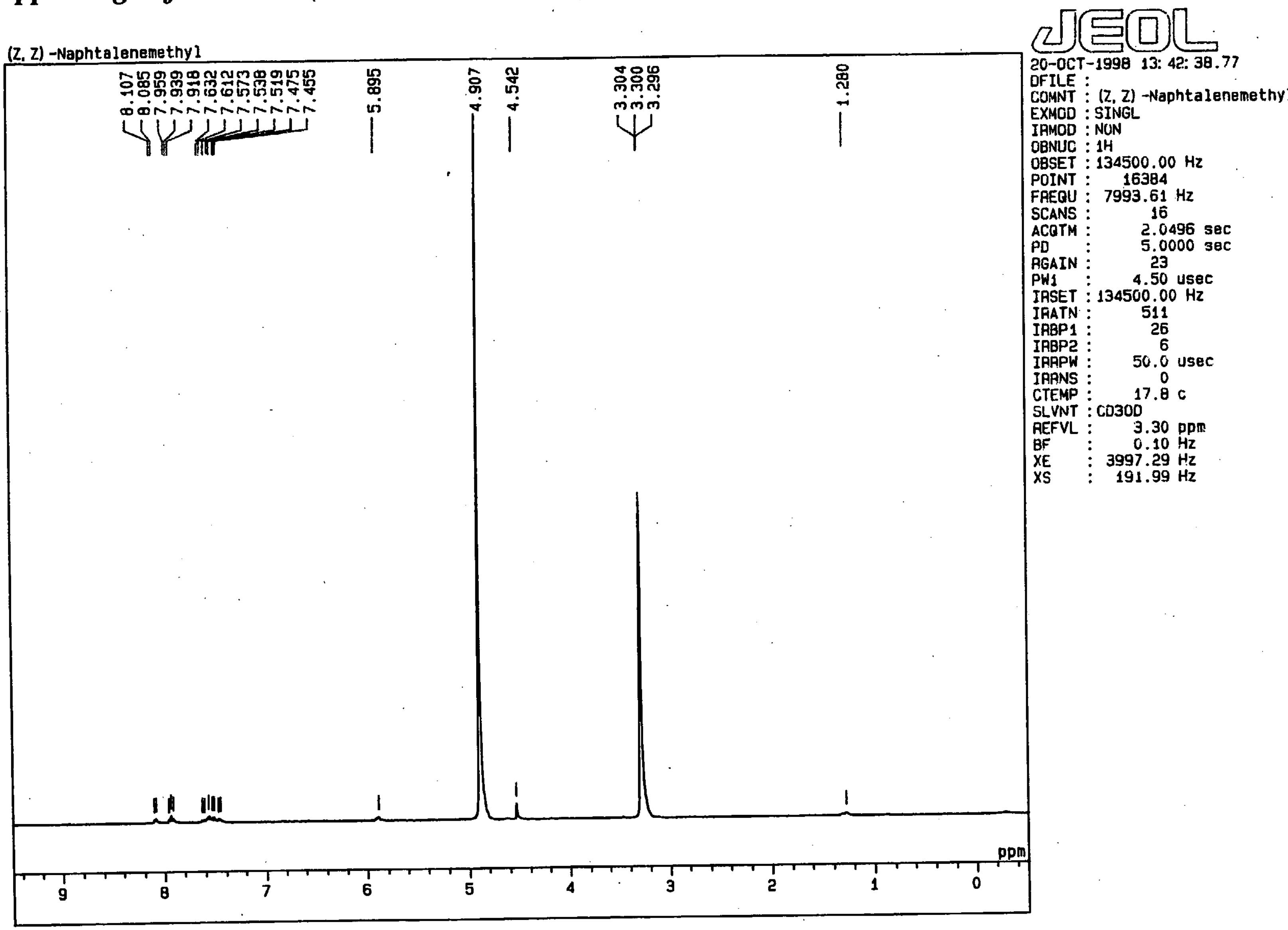
Department of Applied Chemistry, Faculty of Engineering, Osaka City University, Sugimoto, Sumiyoshi-ku, Osaka 558-8585, Japan

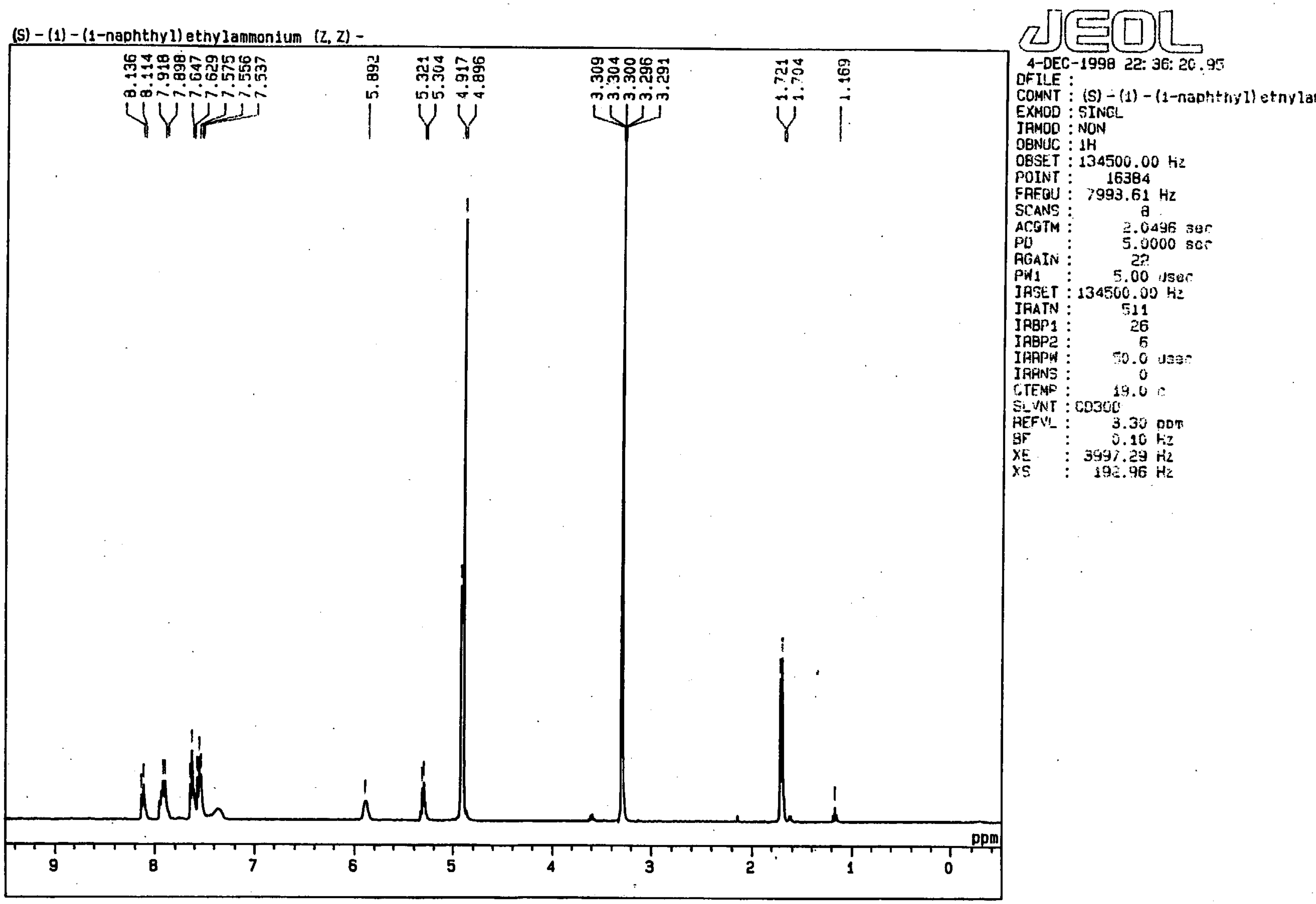
CONTENTS

Supporting Information (A. Matsumoto et al.)



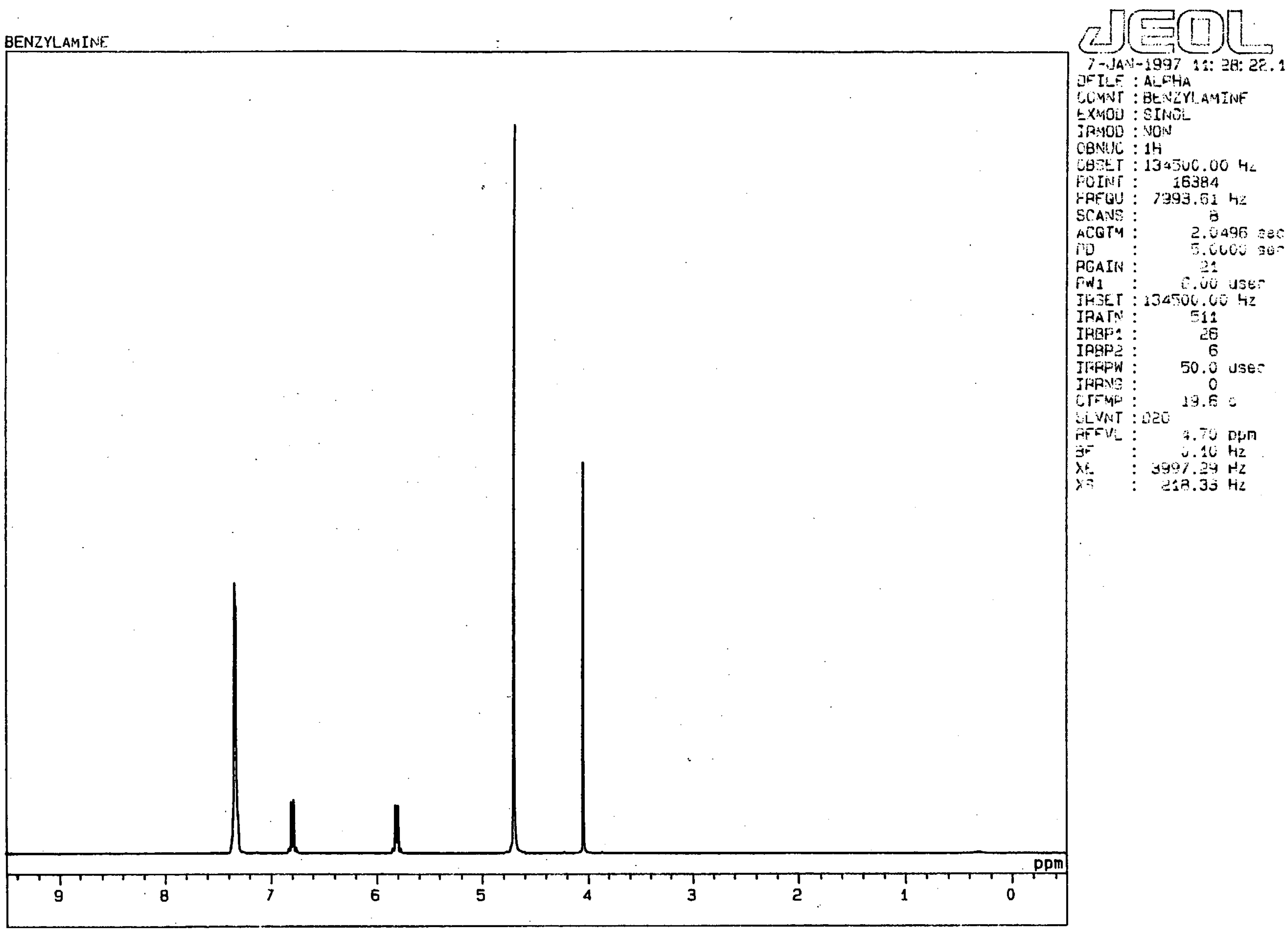
^{1}H NMR spectrum of di(1-naphthylmethylammonium) *(Z,Z)*-muconate (**1a**).

Supporting Information (A. Matsumoto et al.)



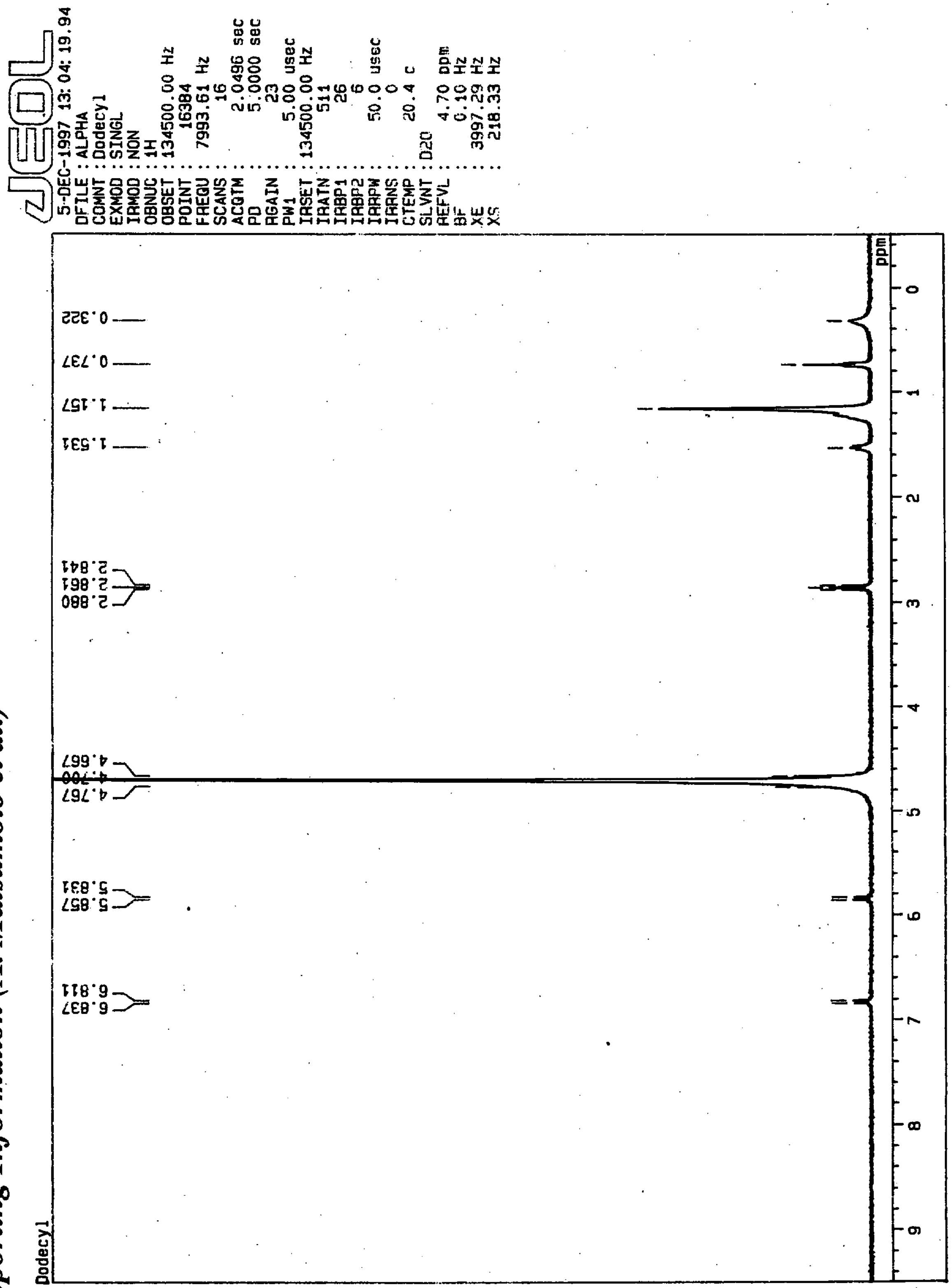
^1H NMR spectrum of di((*S*)-1-naphthylethylammonium) (*Z,Z*)-muconate (**1b**).

Supporting Information (A. Matsumoto et al.)

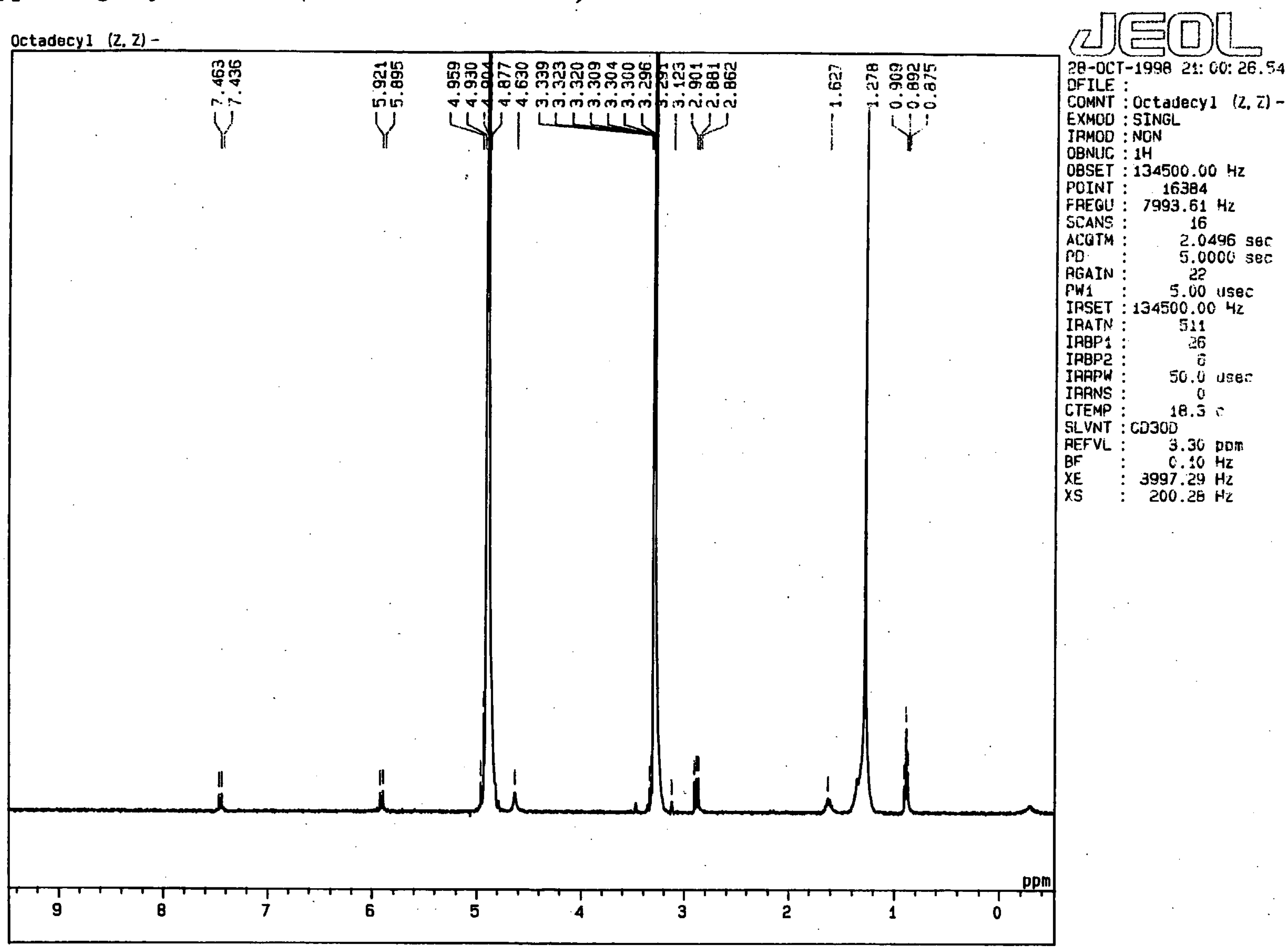


^1H NMR spectrum of di(benzylammonium) *(Z,Z)*-muconate (**1c**).

Supporting Information (A. Matsumoto et al.)

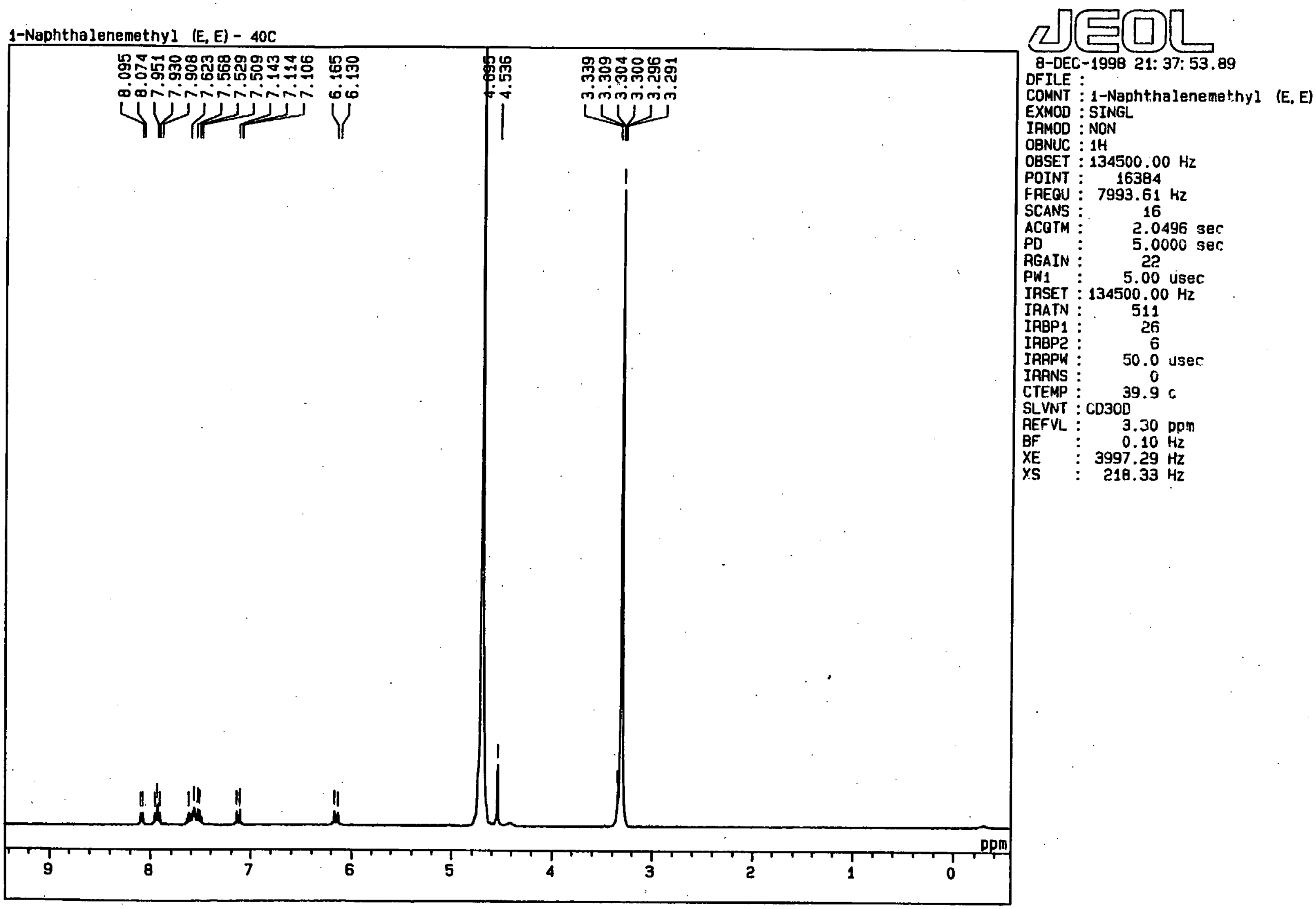


^{1}H NMR spectrum of di(dodecylammonium) (*Z,Z*)-muconate (**1d**).



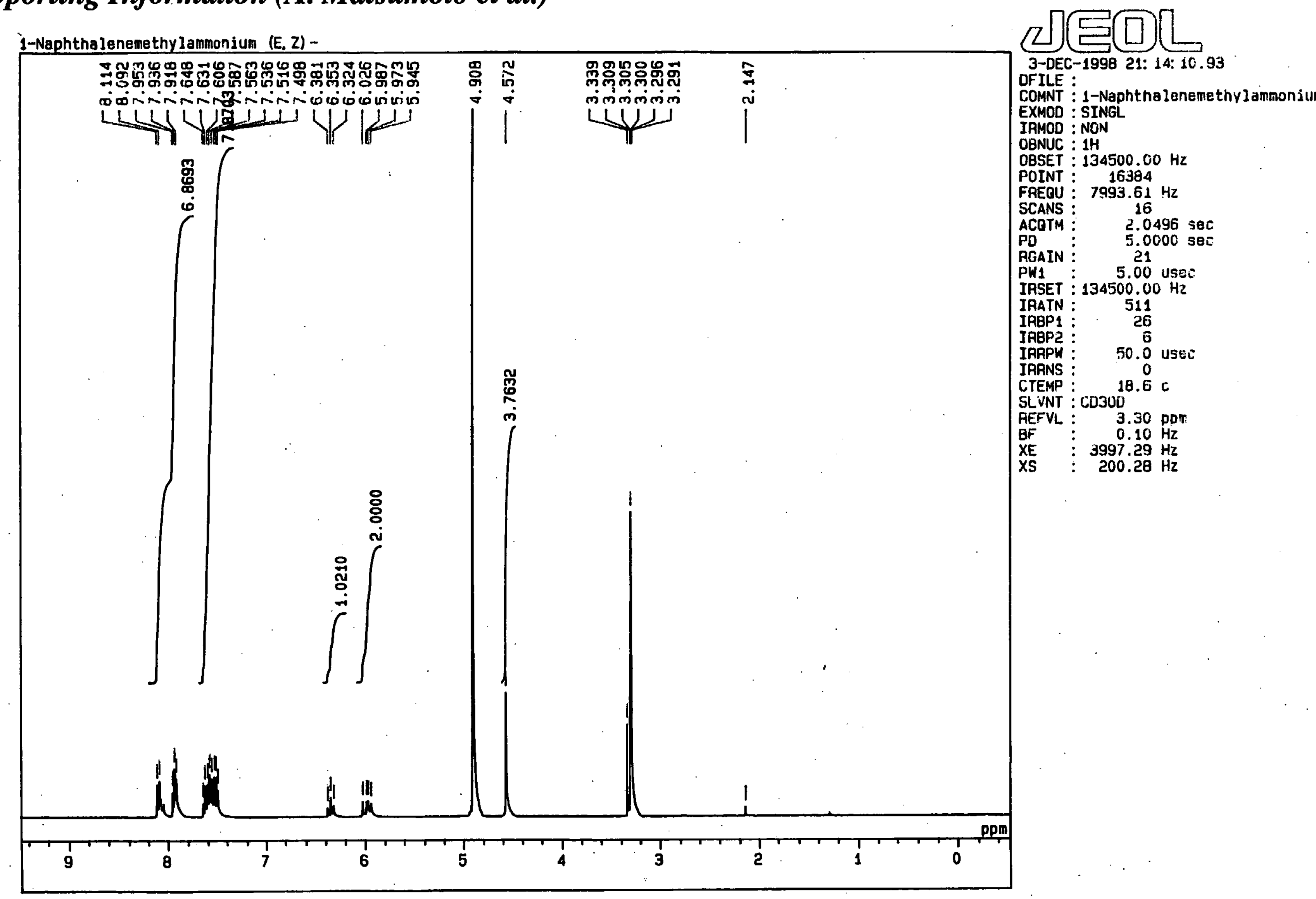
^{1}H NMR spectrum of di(octadecylammonium) *(Z,Z)*-muconate (**1e**).

Supporting Information (A. Matsumoto et al.)



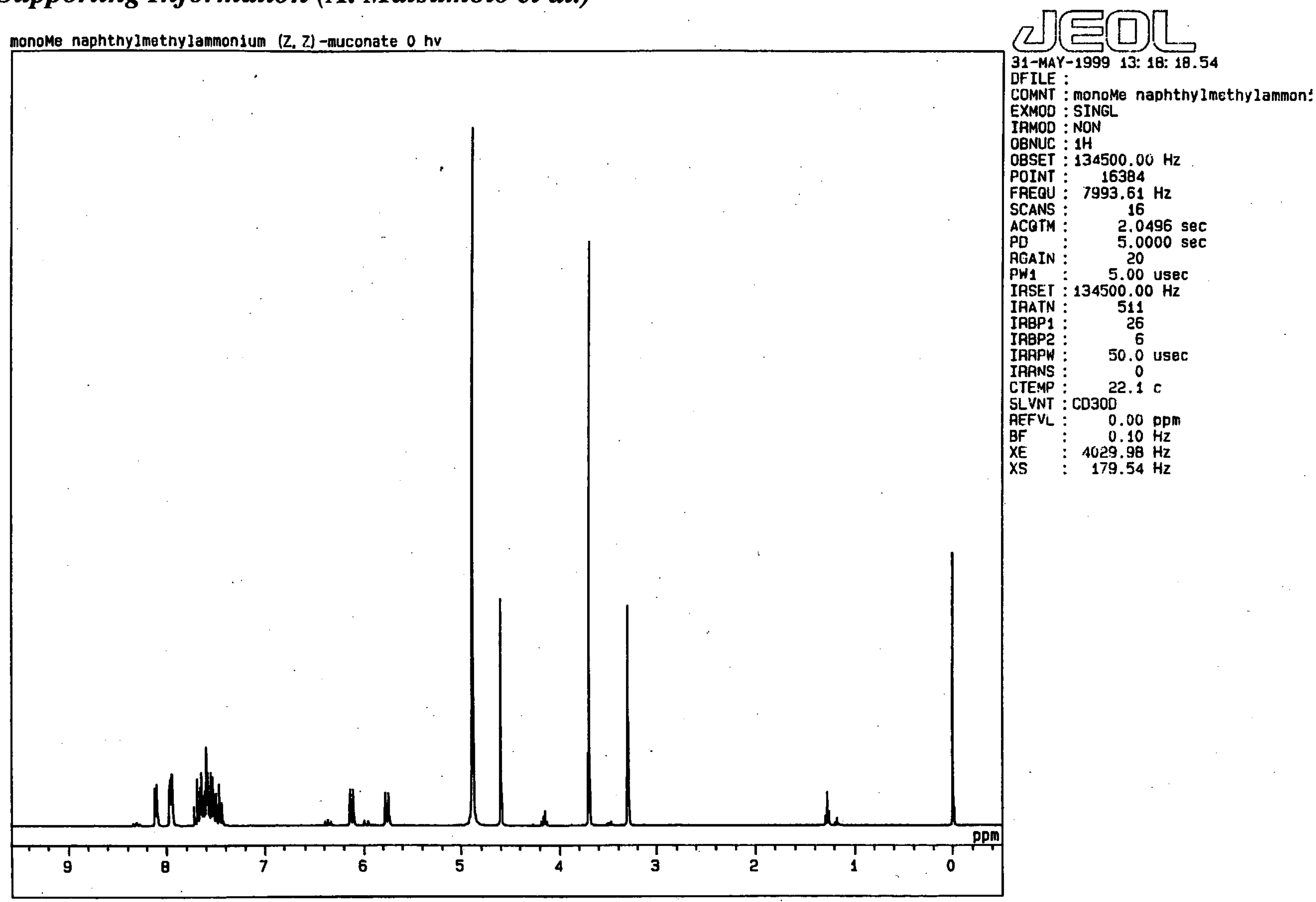
^{1}H NMR spectrum of di(1-naphthylmethylammonium) *(E,E)*-muconate (**2**).

Supporting Information (A. Matsumoto et al.)



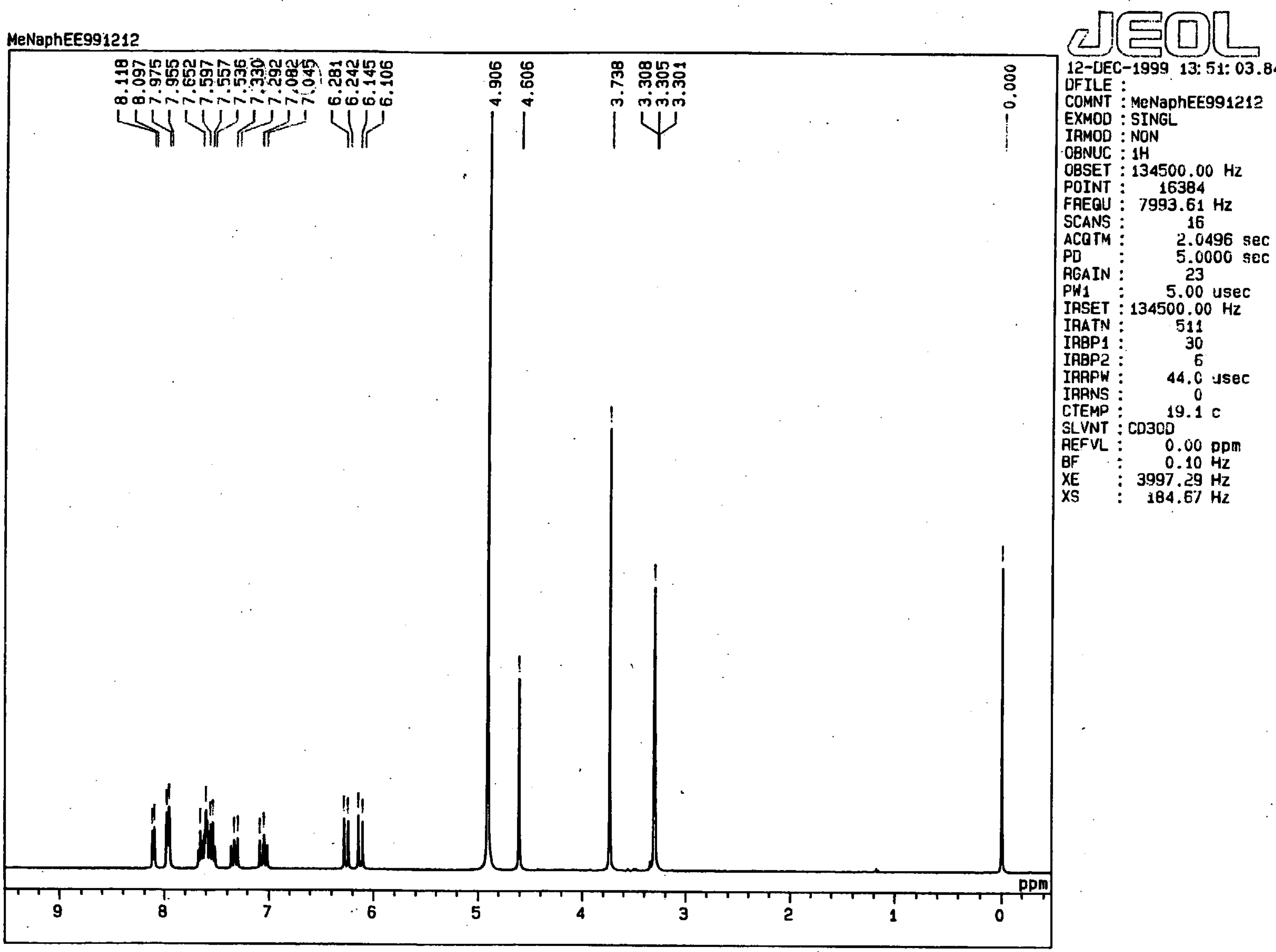
^{1}H NMR spectrum of di(1-naphthylmethylammonium) *(E,Z)*-muconate (**3**).

Supporting Information (A. Matsumoto et al.)



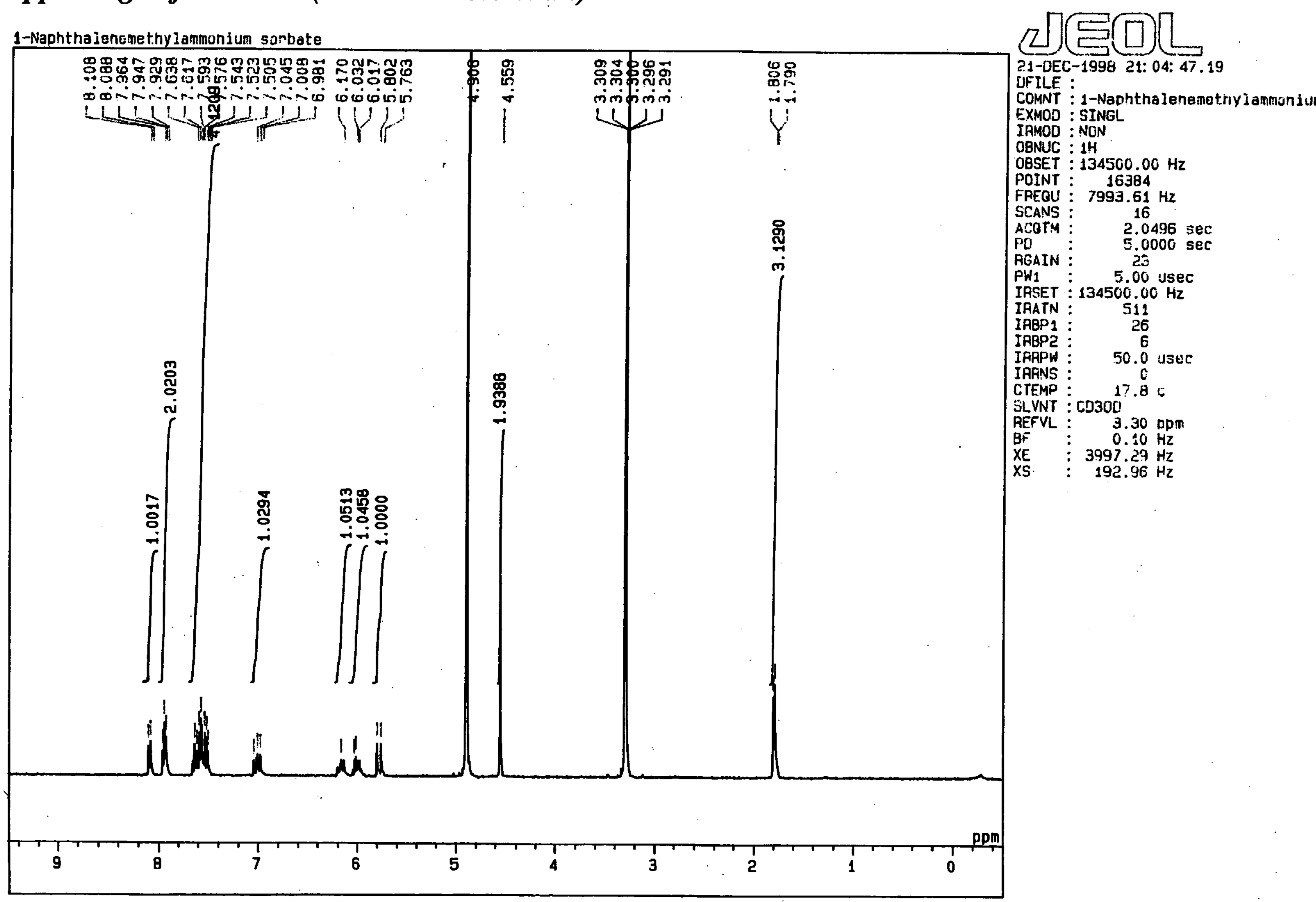
^1H NMR spectrum of methyl 1-naphthylmethylammonium *(Z,Z)*-muconate (**4**).

Supporting Information (A. Matsumoto et al.)



^{1}H NMR spectrum of methyl 1-naphthylmethylammonium *(E,E)*-muconate (**5**).

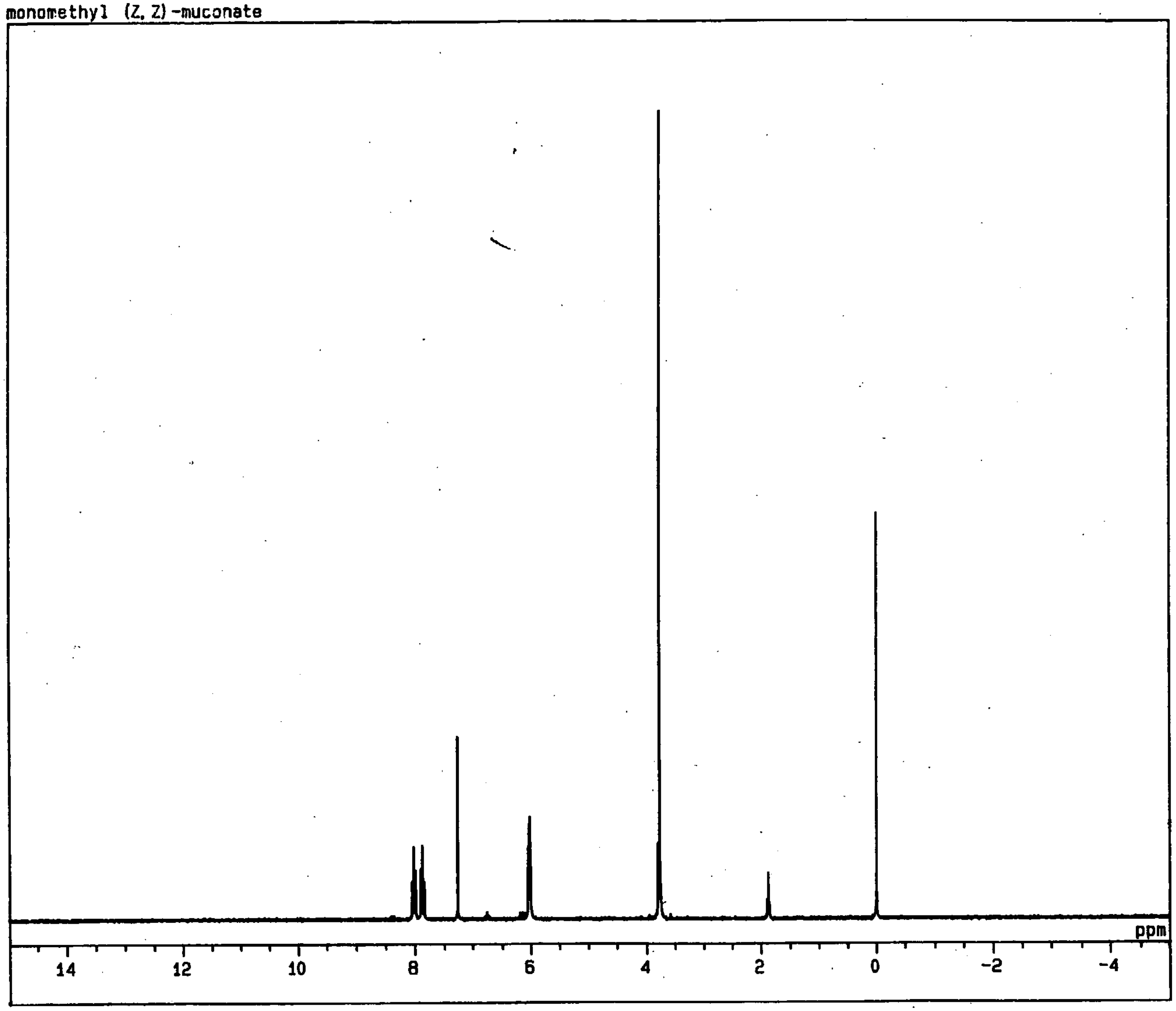
Supporting Information (A. Matsumoto et al.)



^{1}H NMR spectrum of 1-naphthylmethylammonium *(E,E)*-sorbate (**6**).

Supporting Information (A. Matsumoto et al.)

monomethyl (Z, Z)-muconate



JEOL

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PD    :     5.0000 sec
RGAIN :       23
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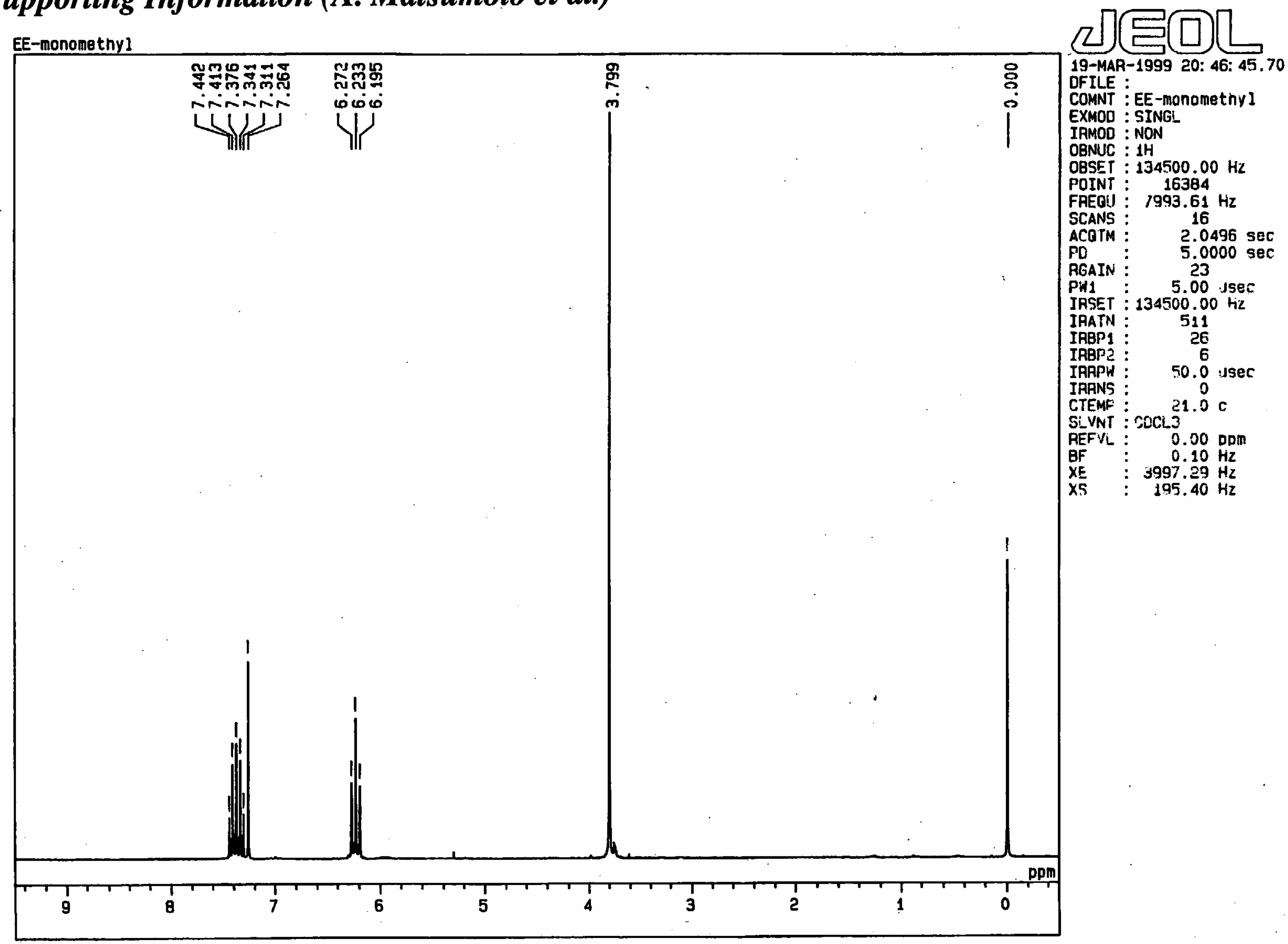
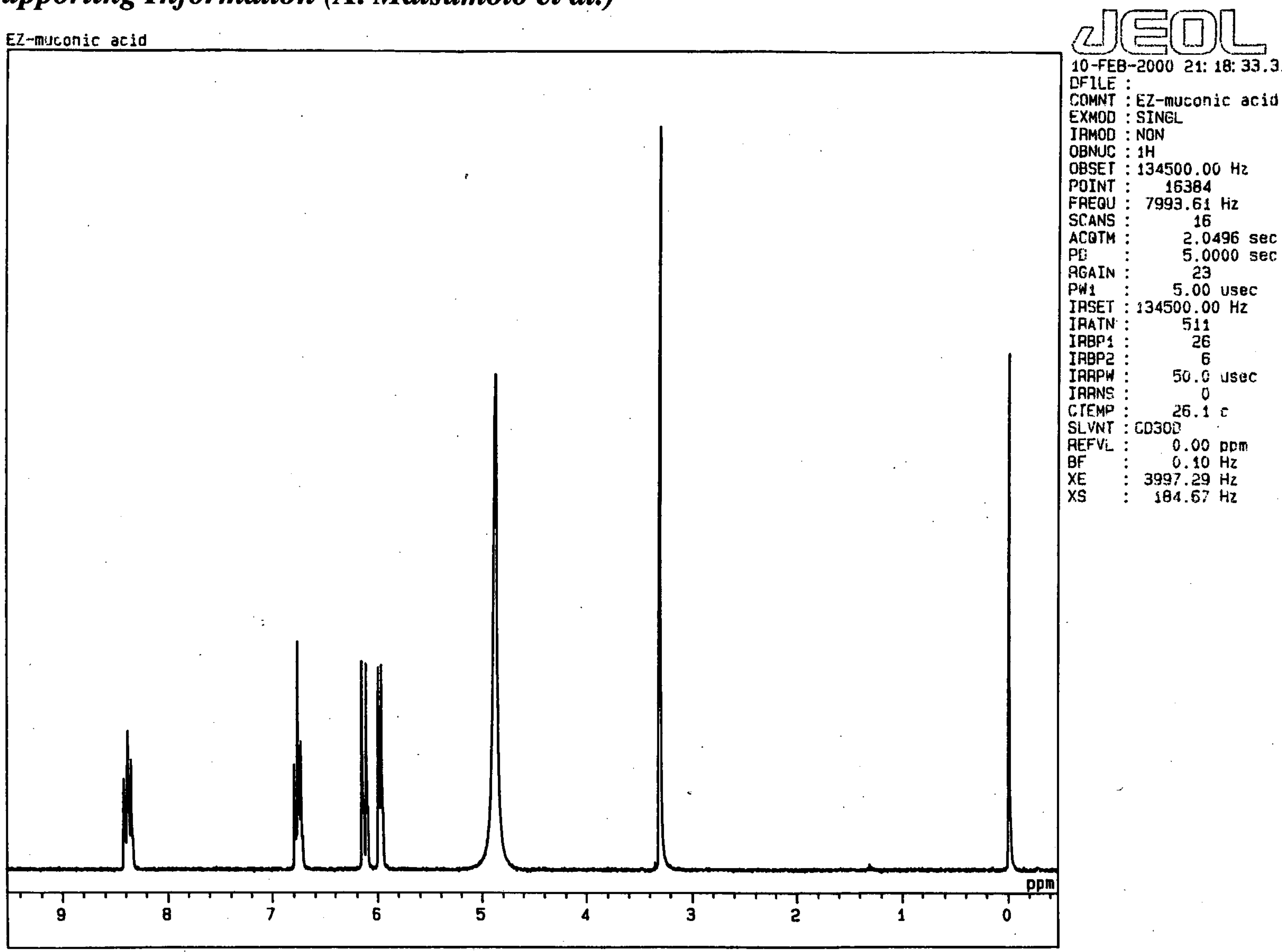
[1]H NMR spectrum of *(Z,Z)*-muconic acid monomethyl ester.

Supporting Information (A. Matsumoto et al.)

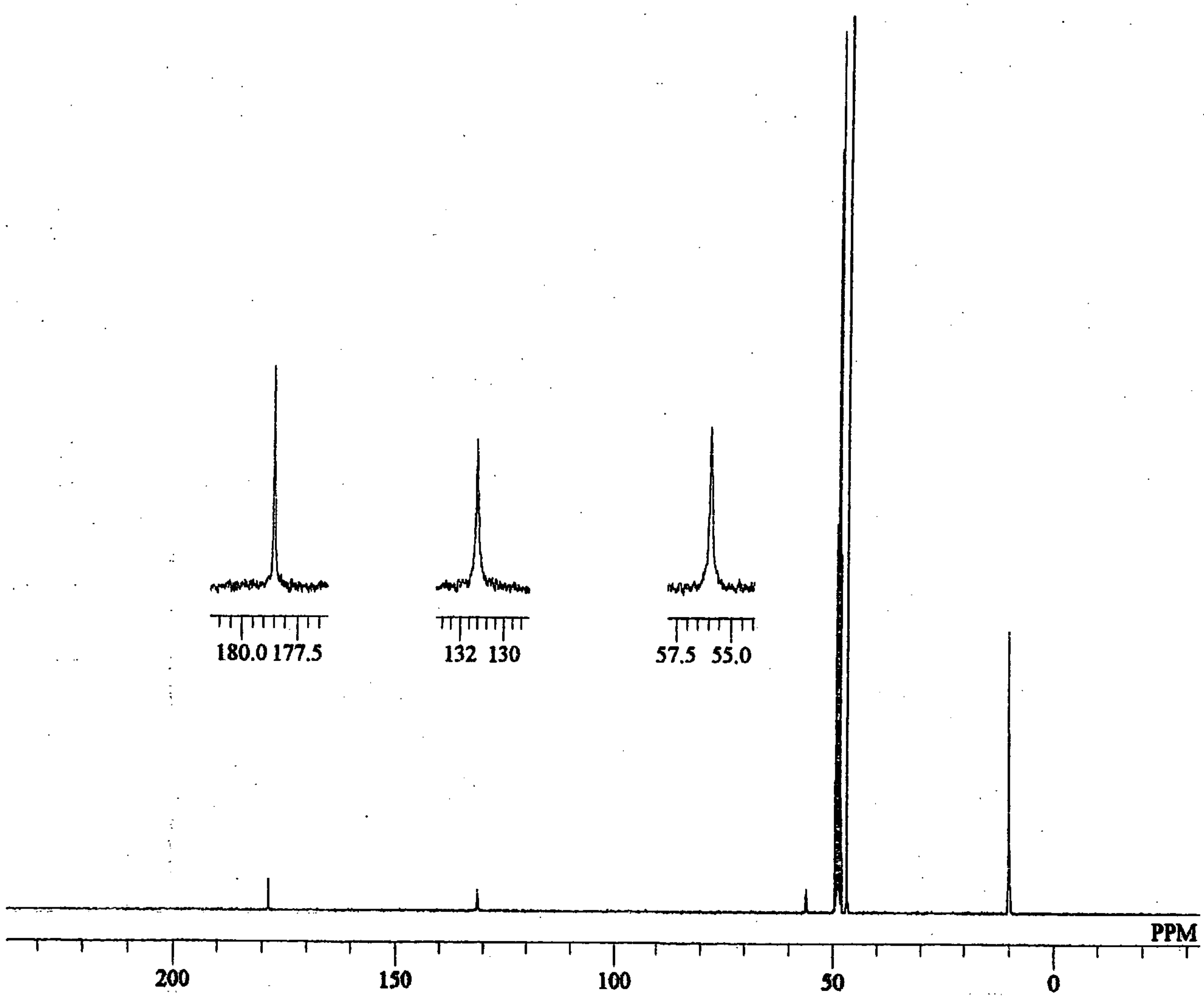


^1H NMR spectrum of *(E,E)*-muconic acid monomethyl ester.

Supporting Information (A. Matsumoto et al.)

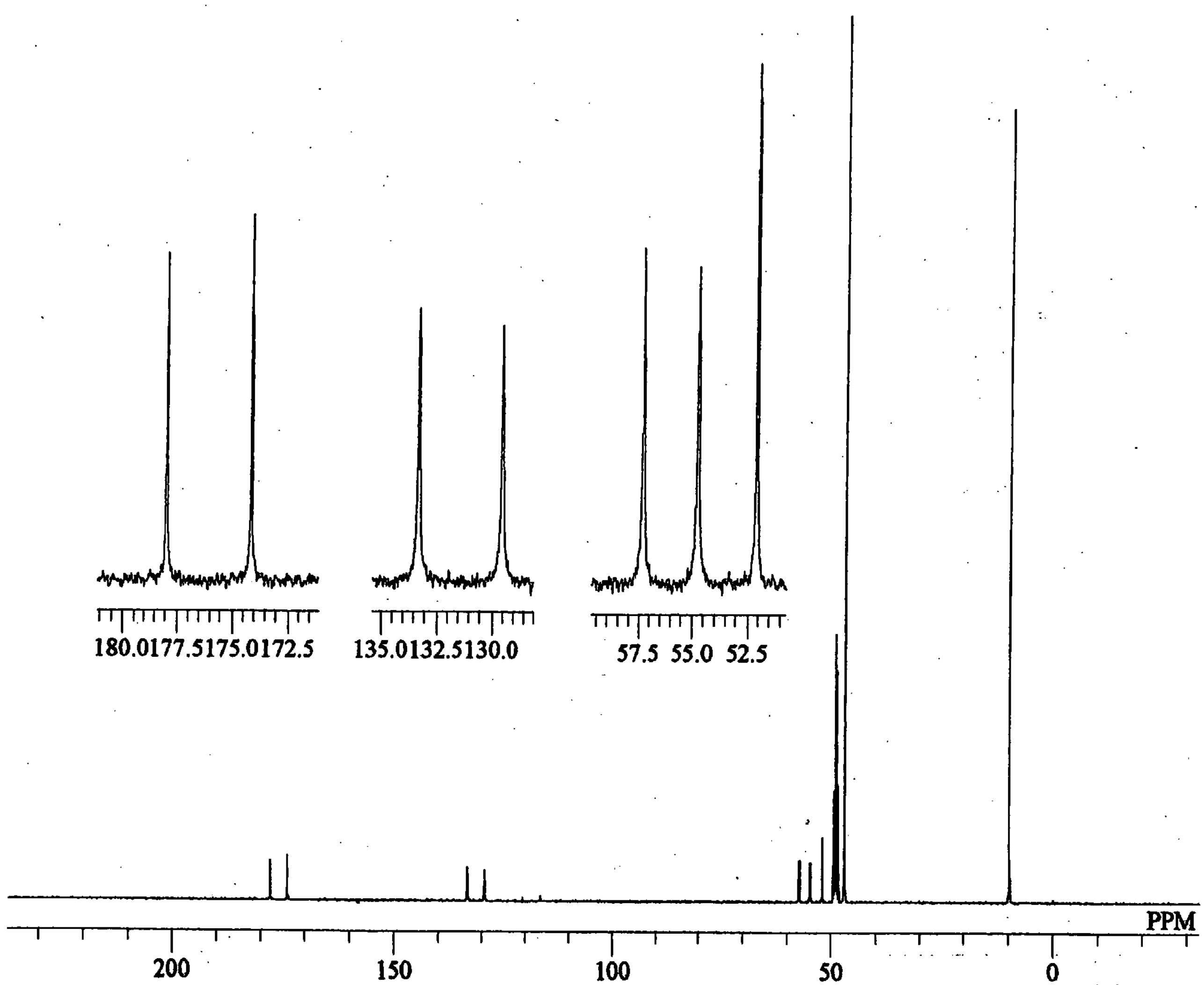


^1H NMR spectrum of *(E,Z)*-muconic acid.

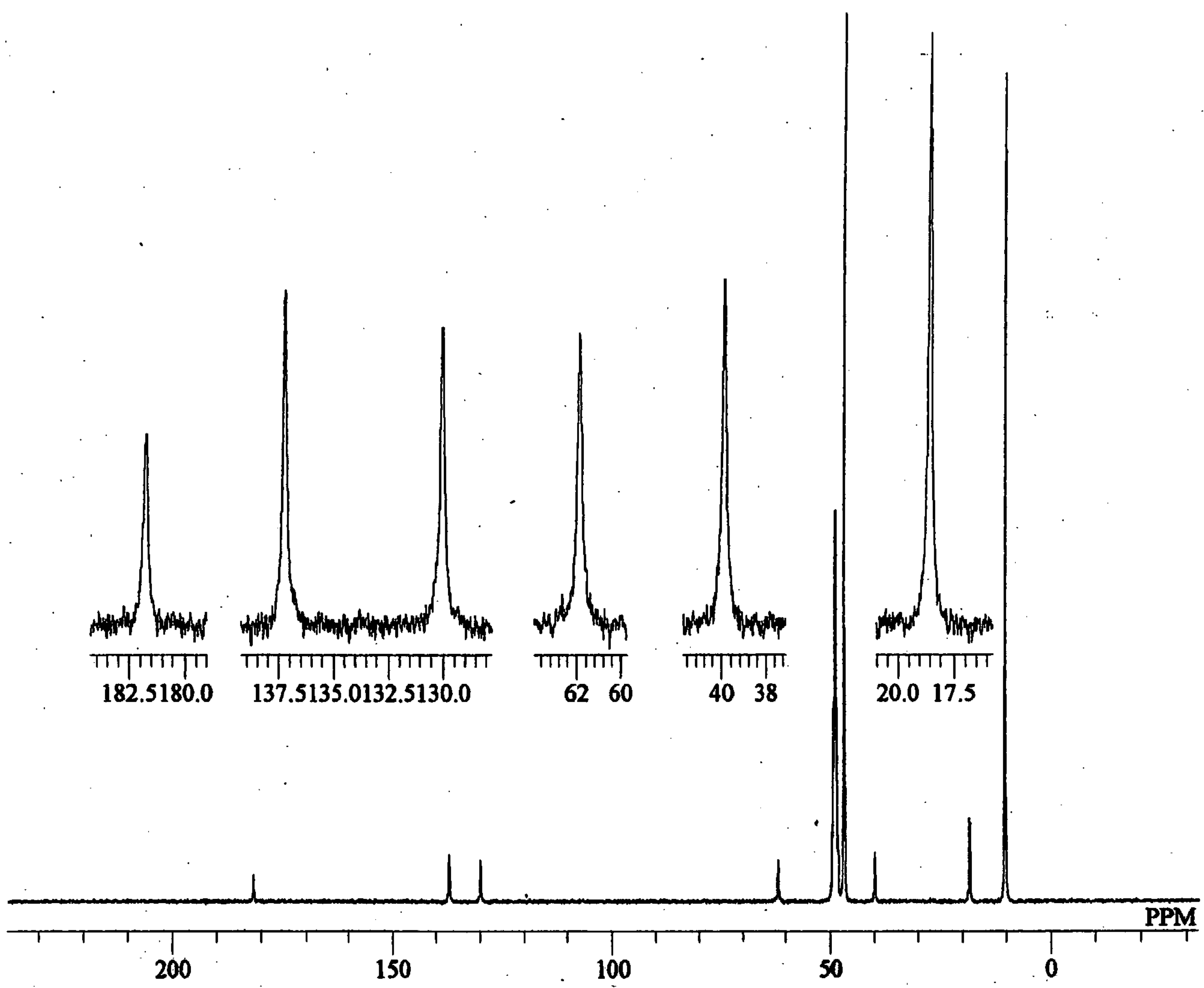


^{13}C NMR spectrum of poly(**7**) derived from poly(**2**). Solvent: methanol-d_4.

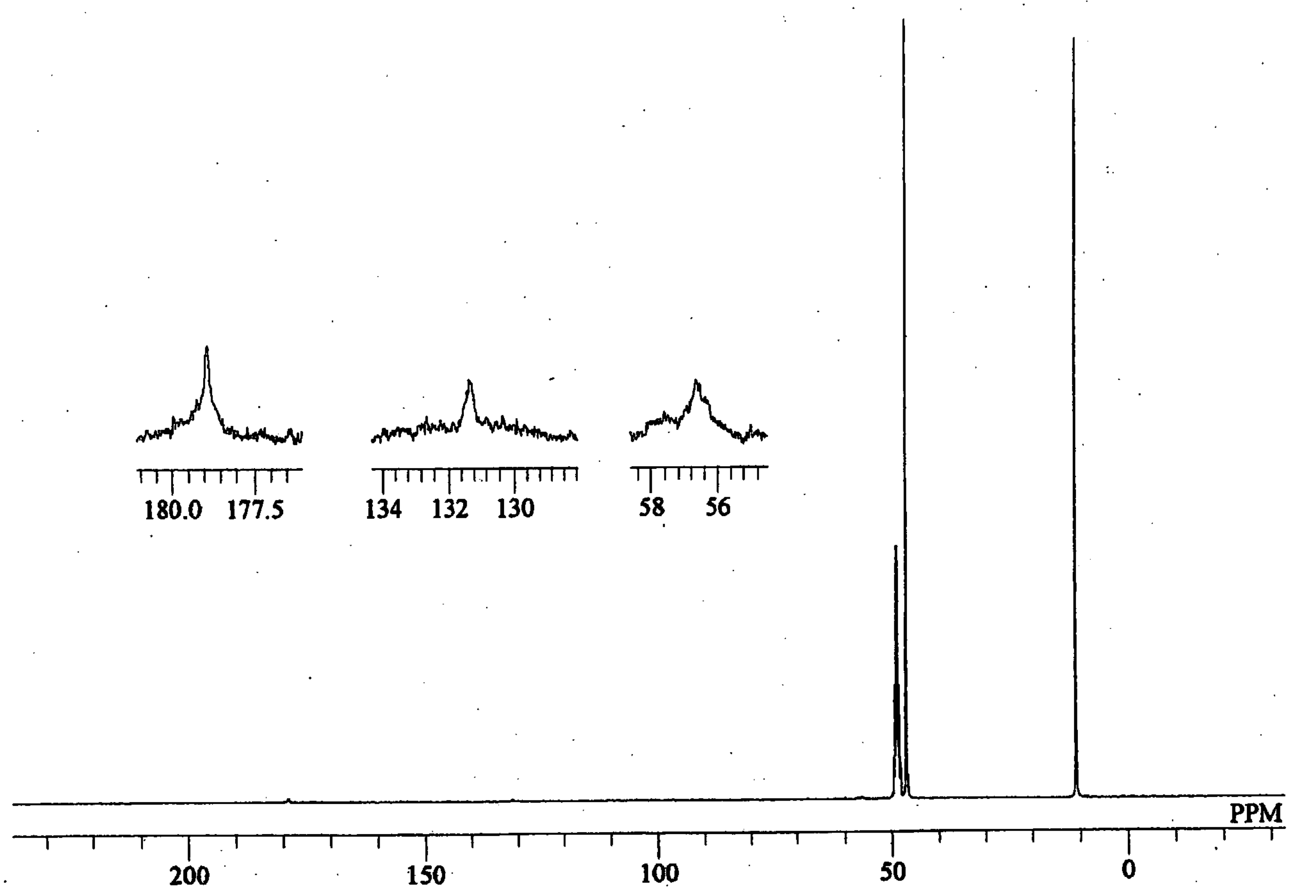
Supporting Information (A. Matsumoto et al.)



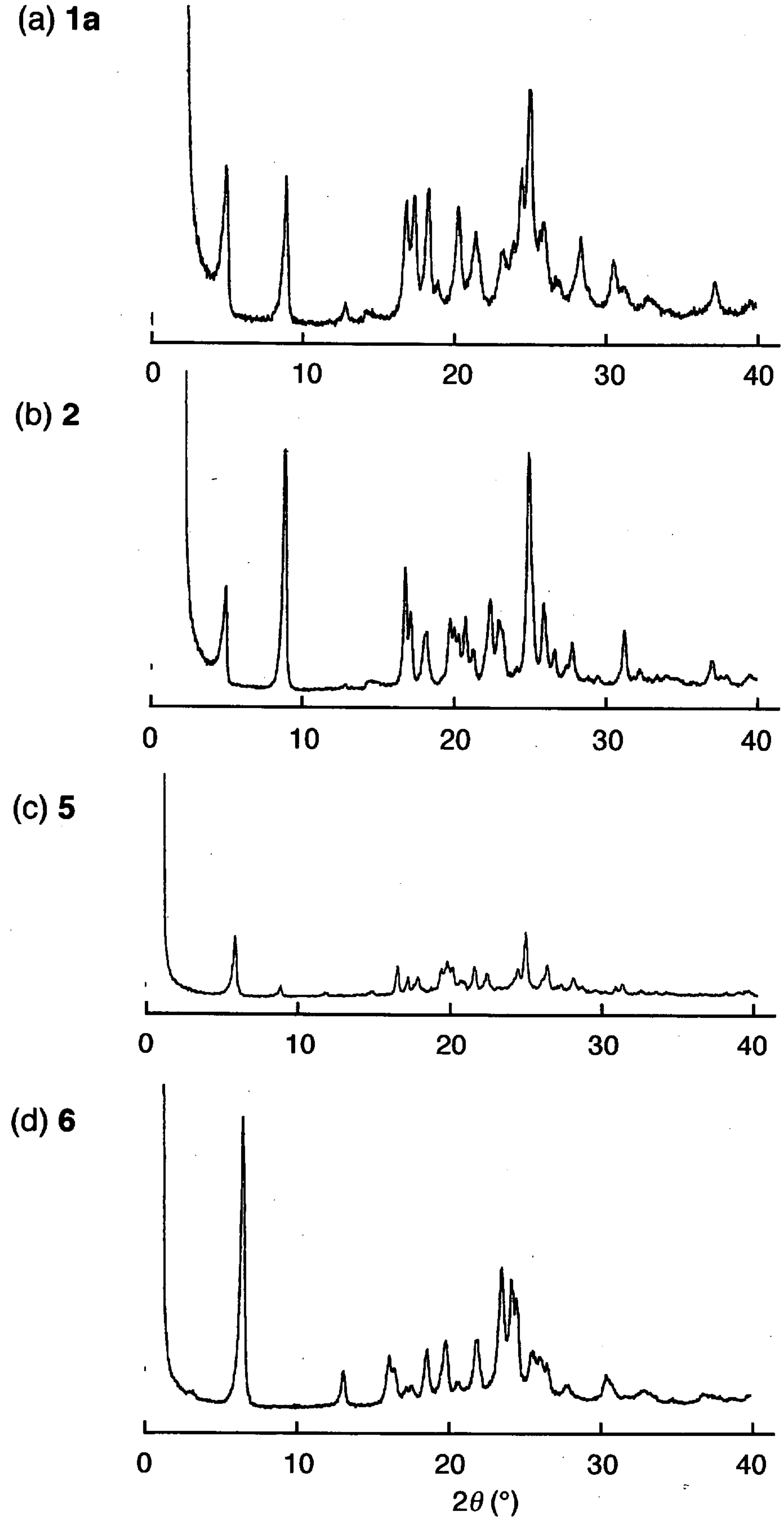
^{13}C NMR spectrum of poly(**8**) derived from poly(**5**). Solvent: methanol-d_4.



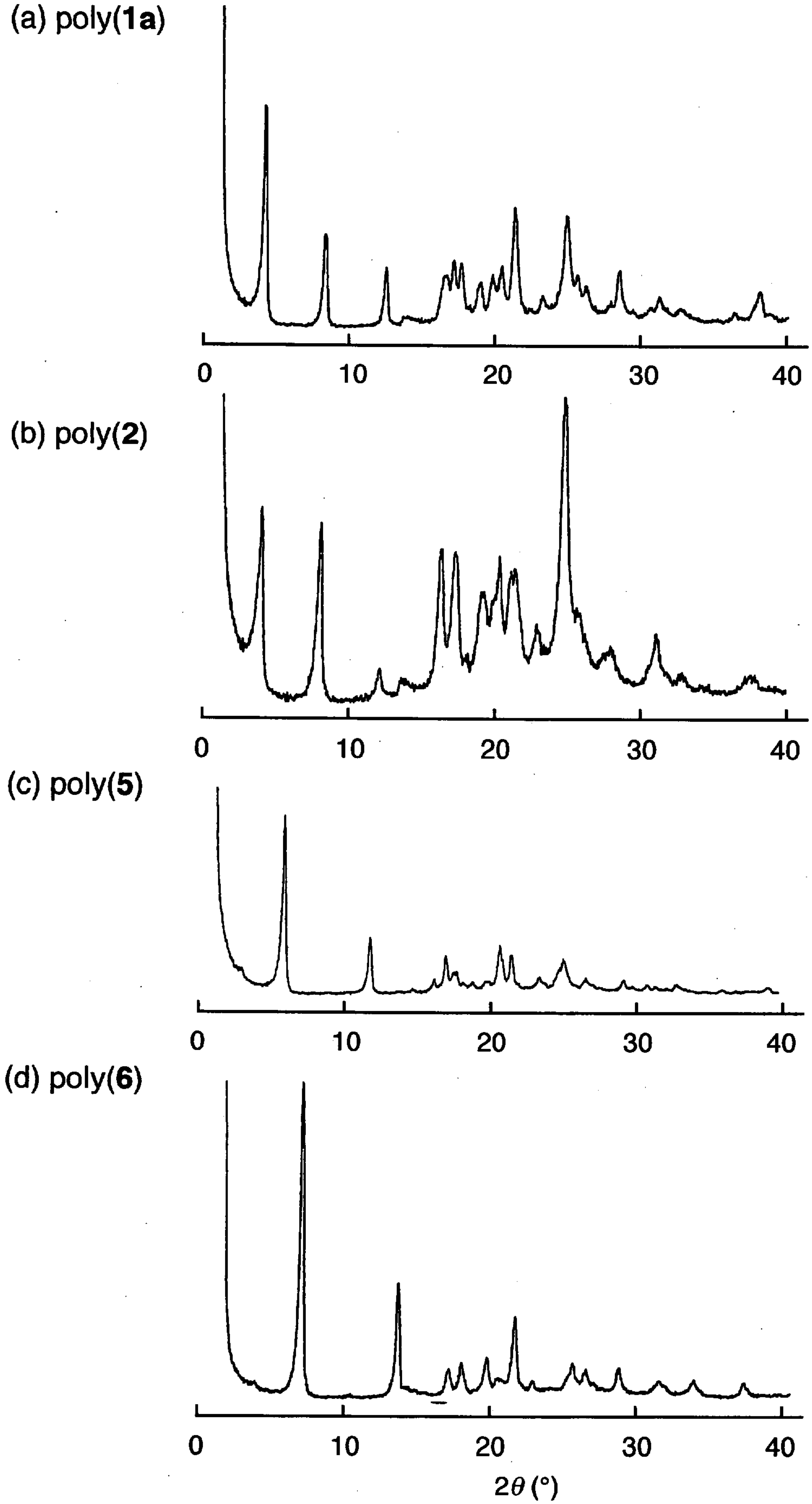
^{13}C NMR spectrum of poly(**9**) derived from poly(**6**). Solvent: methanol-d_4.



^{13}C NMR spectrum of atactic poly(**7**) derived from the polymer produced during the isomerization polymerization of **3**. Solvent: methanol-d_4.



Powder X-ray diffraction profiles of monomers: (a) **1a**, (b) **2**, (c) **5**, and (d) **6**. (Cu Kα)



Powder X-ray diffraction profiles of polymers: (a) poly(**1a**), (b) poly(**2**), (c) poly(**5**), and (d) poly(**6**). (Cu Kα)

X-ray Structure Report

Fri Mar 17 2000

Experimental

Data Collection

A colorless needle crystal of $C_{17}H_{19}O_2N$ having approximate dimensions of 0.15 x 0.10 x 0.50 mm was mounted on a glass fiber. All measurements were made on a Rigaku RAXIS-RAPID Imaging Plate diffractometer with graphite monochromated Mo-Kα radiation.

Indexing was performed from 1 oscillations which were exposed for 0.8 minutes. The camera radius was 127.40 mm. Readout was performed in the 0.100 mm pixel mode.

Cell constants and an orientation matrix for data collection corresponded to a C-centered monoclinic cell with dimensions:

$$a = 54.642(3)\ \text{Å}$$
$$b = 4.9909(1)\ \text{Å} \qquad \beta = 99.442(2)^\circ$$
$$c = 11.0533(1)\ \text{Å}$$
$$V = 2973.5(2)\ \text{Å}^3$$

For Z = 8 and F.W. = 269.34, the calculated density is 1.20 g/cm^3. Based on the systematic absences of:

hkl: $h+k \neq 2n$

h0l: $l \neq 2n$

packing considerations, a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be:

C2/c (#15)

The data were collected at a temperature of $23 \pm 1^\circ$C to a maximum 2θ value of 55.0°. A total of 44 images, corresponding to 220.0 ° oscillation angles, were collected with 2 different goniometer settings. Exposure time was 0.20 minutes per degree. The camera radius was 127.40 mm. Readout was performed in the 0.100 mm pixel mode. Data were processed by the PROCESS-AUTO program package.

Data Reduction

Of the 6386 reflections which were collected, 3487 were unique (R_{int} = 0.024); equivalent reflections were merged.

The linear absorption coefficient, μ, for Mo-Kα radiation is 0.8 cm^{-1}. An absorption correction was not applied. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods[1] and expanded using Fourier techniques[2]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were included but not refined. The final cycle of full-matrix least-squares refinement[3] was based on 795 observed reflections (I > 2.00σ(I)) and 181 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma(Fo^2 - Fc^2)/\Sigma Fo^2 = 0.066$$

$$R_w = \sqrt{\Sigma w(Fo^2 - Fc^2)^2/\Sigma w(Fo^2)^2} = 0.095$$

$$R1 = \Sigma||Fo| - |Fc||/\Sigma|Fo| = 0.052 \quad for \;\; I > 2.0\sigma(I) \;\; data$$

The standard deviation of an observation of unit weight[4] was 1.04. The weighting scheme was based on counting statistics and included a factor (p = 0.050) to downweight the intense reflections. Plots of $\Sigma w(Fo^2 - Fc^2)^2$ versus Fo^2, reflection order in data collection, sin θ/λ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.24 and -0.28 $e^-/\AA^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[5] . Anomalous dispersion effects were included in Fcalc[6]; the values for Δf' and Δf" were those of Creagh and McAuley[7]. The values for the mass attenuation coefficients are those of Creagh and Hubbel[8]. All calculations were performed using the teXsan[9] crystallographic software package of Molecular Structure Corporation.

References

(1) SAPI91: Fan Hai-Fu (1991). Structure Analysis Programs with Intelligent Control, Rigaku Corporation, Tokyo, Japan.

(2) DIRDIF94: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M. (1994). The DIRDIF-94 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(3) Least-Squares:

Function minimized: $\Sigma w(Fo^2 - Fc^2)^2$

where $w = \frac{1}{\sigma^2(Fo^2)} = [\sigma_c^2(Fo^2) + (p(Max(Fo^2, 0) + 2Fc^2)/3)^2]^{-1}$

$\sigma_c(Fo^2)$ = e.s.d. based on counting statistics

p = p-factor

(4) Standard deviation of an observation of unit weight:

$$\sqrt{\Sigma w(|Fo| - |Fc|)^2/(No - Nv)}$$

where: No = number of observations

Nv = number of variables

(5) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The

Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(6) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(7) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(8) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(9) teXsan: Crystal Structure Analysis Package, Molecular Structure Corporation (1985 & 1999).

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{17}H_{19}O_2N$
Formula Weight	269.34
Crystal Color, Habit	colorless, needle
Crystal Dimensions	0.15 X 0.10 X 0.50 mm
Crystal System	monoclinic
Lattice Type	C-centered
No. of Reflections Used for Unit Cell Determination (2θ range)	4418 (4.2 - 54.7°)
Indexing Images	1 oscillations at 0.8 minutes
Camera Radius	127.40 mm
Lattice Parameters	a = 54.642(3) Å b = 4.9909(1) Å c = 11.0533(1) Å β = 99.442(2)° V = 2973.5(2) Å^3
Space Group	C2/c (#15)
Z value	8
D_{calc}	1.203 g/cm^3
F_{000}	1152.00
μ(MoKα)	0.79 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID Imaging Plate
Radiation	MoKα (λ = 0.71069 Å) graphite monochromated

Temperature	23.0 °C
Voltage, Current	40 kV, 50 mA
Collimator Size	0.8 mm
Detector Aperture	270.0 mm x 256.0 mm
Data Images	44 exposures at 0.2 minutes per degree
Oscillation Range (ϕ=0.0°,χ=45.0°)	ω 130.0 - 190.0° with 5.0° step
Oscillation Range (ϕ=180.0°,χ=45.0°)	ω 0.0 - 160.0° with 5.0° step
Camera Radius	127.40 mm
Pixel Size	0.100 mm
$2\theta_{max}$	55.0°
No. of Reflections Measured	Total: 6386 Unique: 3487 (R_{int} = 0.024)
Corrections	Lorentz-polarization

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SAPI91)
Refinement	Full-matrix least-squares
Function Minimized	$\Sigma w(Fo^2 - Fc^2)^2$
Least Squares Weights	$w = \frac{1}{\sigma^2(Fo^2)}$
p-factor	0.0500
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (I>2.00σ(I))	795
No. Variables	181
Reflection/Parameter Ratio	4.39
Residuals: R; Rw	0.066 ; 0.095
Residuals: R1	0.052
No. of Reflections to calc R1	795

Goodness of Fit Indicator	1.04
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	0.24 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.28 $e^-/Å^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
O(1)	0.65574(8)	0.8048(9)	0.5124(4)	4.5(1)
O(2)	0.65316(7)	0.5506(8)	0.3448(3)	3.8(1)
N(1)	0.64180(9)	-0.0574(10)	0.7358(4)	3.2(1)
C(1)	0.7343(1)	-0.012(2)	0.8784(6)	6.7(2)
C(2)	0.7169(1)	0.197(2)	0.8171(7)	5.3(2)
C(3)	0.7057(1)	0.196(1)	0.7019(6)	4.3(2)
C(4)	0.6896(1)	0.404(1)	0.6405(5)	3.9(2)
C(5)	0.6784(1)	0.395(1)	0.5255(6)	4.0(2)
C(6)	0.6614(1)	0.600(1)	0.4582(6)	3.1(2)
C(7)	0.6145(1)	-0.063(1)	0.7342(5)	3.2(2)
C(8)	0.6000(1)	0.134(1)	0.6446(5)	3.0(2)
C(9)	0.6111(1)	0.293(1)	0.5689(6)	3.6(2)
C(10)	0.5970(1)	0.475(1)	0.4859(6)	3.9(2)
C(11)	0.5717(1)	0.490(1)	0.4812(6)	4.8(2)
C(12)	0.5336(2)	0.336(2)	0.5512(7)	6.4(3)
C(13)	0.5220(1)	0.182(2)	0.6257(9)	7.5(3)
C(14)	0.5359(1)	0.000(2)	0.7093(7)	6.1(3)
C(15)	0.5611(1)	-0.014(1)	0.7170(6)	4.6(2)
C(16)	0.5733(1)	0.146(1)	0.6413(6)	3.4(2)
C(17)	0.5595(1)	0.328(1)	0.5568(6)	4.1(2)
H(1)	0.7481	-0.0364	0.8380	6.7167
H(2)	0.7401	0.0232	0.9637	6.7167
H(3)	0.7257	-0.1861	0.8759	6.7167
H(4)	0.7135	0.3536	0.8681	5.8725

Table 1. Atomic coordinates and B_{iso}/B_{eq} (continued)

atom	x	y	z	B_{eq}
H(5)	0.7087	0.0424	0.6526	4.3831
H(6)	0.6861	0.5597	0.6879	4.2390
H(7)	0.6822	0.2361	0.4793	4.4645
H(8)	0.6450	-0.1024	0.6545	3.9671
H(9)	0.6483	0.1142	0.7566	3.9671
H(10)	0.6500	-0.1871	0.7914	3.9671
H(11)	0.6123	-0.0212	0.8166	3.3219
H(12)	0.6090	-0.2406	0.7158	3.3219
H(13)	0.6287	0.2787	0.5701	4.1111
H(14)	0.6054	0.5845	0.4348	4.7384
H(15)	0.5623	0.6133	0.4223	5.3637
H(16)	0.5232	0.4552	0.4921	7.6774
H(17)	0.5042	0.1786	0.6189	8.1739
H(18)	0.5272	-0.0995	0.7655	6.3623
H(19)	0.5704	-0.1393	0.7735	5.1534

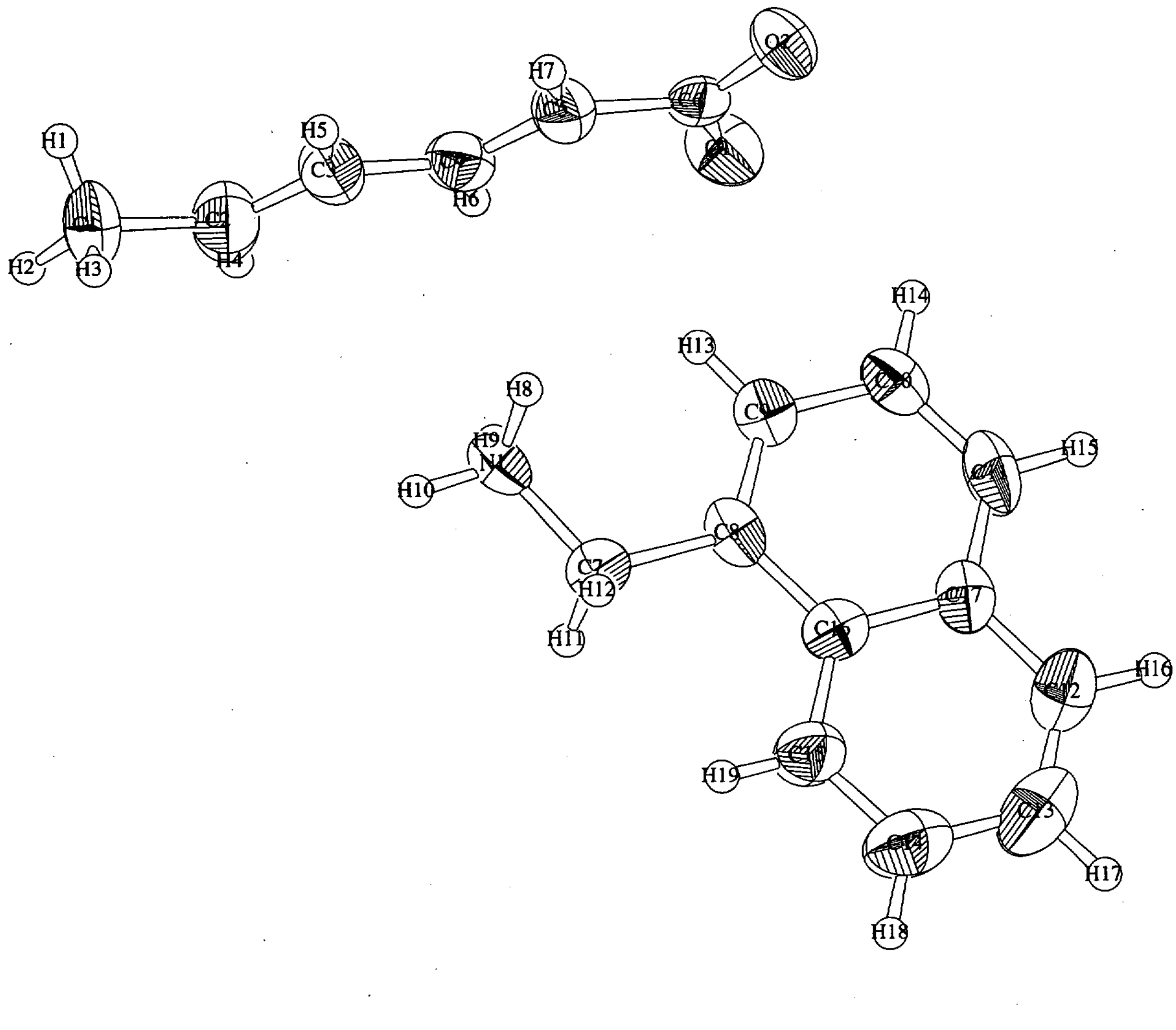
$$B_{eq} = \frac{8}{3}\pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}aa^*bb^*\cos\gamma + 2U_{13}aa^*cc^*\cos\beta + 2U_{23}bb^*cc^*\cos\alpha)$$

Table 2. Anisotropic Displacement Parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	0.082(3)	0.043(3)	0.047(3)	0.010(3)	0.017(3)	-0.008(2)
O(2)	0.067(3)	0.041(3)	0.034(2)	-0.001(3)	0.003(2)	-0.004(2)
N(1)	0.059(4)	0.036(4)	0.029(3)	-0.001(3)	0.007(3)	0.002(2)
C(1)	0.070(5)	0.103(8)	0.075(5)	0.032(6)	-0.004(4)	0.034(5)
C(2)	0.063(5)	0.073(6)	0.061(5)	0.013(5)	0.000(4)	0.003(4)
C(3)	0.060(5)	0.058(6)	0.045(4)	0.010(5)	0.007(4)	0.005(4)
C(4)	0.052(4)	0.052(5)	0.046(4)	-0.006(4)	0.014(4)	0.004(4)
C(5)	0.052(4)	0.050(5)	0.049(4)	0.006(4)	0.008(4)	0.007(4)
C(6)	0.034(4)	0.037(5)	0.047(4)	-0.012(4)	0.013(4)	0.007(4)
C(7)	0.042(4)	0.032(4)	0.049(4)	-0.001(4)	0.010(4)	-0.004(4)
C(8)	0.057(5)	0.020(5)	0.034(4)	0.000(4)	0.001(4)	-0.003(3)
C(9)	0.050(4)	0.039(5)	0.045(4)	-0.002(4)	0.002(4)	0.000(4)
C(10)	0.063(5)	0.046(5)	0.041(4)	-0.003(4)	0.008(4)	0.008(4)
C(11)	0.076(6)	0.050(6)	0.050(4)	0.021(5)	-0.006(5)	0.013(4)
C(12)	0.053(6)	0.109(9)	0.077(6)	0.023(5)	0.003(5)	0.012(5)
C(13)	0.050(6)	0.117(10)	0.116(8)	0.015(6)	0.014(6)	0.008(7)
C(14)	0.059(6)	0.091(8)	0.086(6)	-0.013(5)	0.027(5)	-0.001(5)
C(15)	0.050(5)	0.058(6)	0.069(5)	-0.004(4)	0.016(4)	0.012(4)
C(16)	0.043(5)	0.042(5)	0.044(4)	-0.004(4)	0.008(4)	-0.010(3)
C(17)	0.052(6)	0.051(6)	0.048(4)	0.008(5)	-0.002(4)	0.002(4)

The general temperature factor expression:

$$\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$$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H3
C2
H4
H5
C3
H6
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H8
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_journal_date_accepted                ?
_journal_coeditor_code                ?
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_publ_contact_author_address
;
    ENTER ADDRESS
;
_publ_contact_author_email           ' ENTER EMAIL ADDRESS '
_publ_contact_author_fax             ' ENTER FAX NUMBER '
_publ_contact_author_phone           ' ENTER PHONE NUMBER '
_publ_contact_letter
;
    ENTER TEXT OF LETTER
;
_publ_requested_journal              ' ENTER JOURNAL NAME HERE'
_publ_requested_category             ' CHOOSE FI FM FO CI CM CO or AD'

_publ_requested_coeditor_name         ?
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# TITLE AND AUTHOR LIST

_publ_section_title
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loop_
_publ_author_name
_publ_author_footnote
_publ_author_address
' FIRST AUTHORS NAME '
;
  FIRST AUTHORS FOOTNOTES
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;
  FIRST AUTHORS ADDRESS
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_publ_section_synopsis
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# TEXT

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_publ_section_comment
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    ENTER TEXT
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_publ_section_acknowledgements
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    ENTER ACKNOWLEDGEMENTS
;

_publ_section_references
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    ENTER OTHER REFERENCES
Molecular Structure Corporation, Rigaku Corporation. (1999). teXsan.
Single Crystal Structure Analysis Software. Version 1.10.
MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
Rigaku, 3-9-12 Akishima, Tokyo, Japan.
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_publ_section_exptl_refinement
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#------------------------------------------------------------------------------
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_symmetry_Int_Tables_number             15
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x,y,z
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x,-y,1/2+z
1/2+x,1/2+y,z
1/2-x,1/2+y,1/2-z
1/2-x,1/2-y,-z
1/2+x,1/2-y,1/2+z
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_cell_length_c                          11.0533(1)
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_diffrn_measurement_method              \w
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_diffrn_reflns_number                   6386
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# REFINEMENT DATA
_refine_special_details
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Refinement using reflections with F^2^ > 2.0 sigma(F^2^). The weighted
R-factor (wR) and goodness of fit (S) are based on F^2^. R-factor (gt) are
based on F. The threshold expression of F^2^ > 2.0 sigma(F^2^) is used only
for calculating R-factor (gt).
;
_reflns_number_total                    3487
_reflns_number_gt                       795
_reflns_threshold_expression            F^2^>2.0\s(F^2^)
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_refine_ls_wR_factor_ref                0.0947
_refine_ls_hydrogen_treatment           noref
_refine_ls_number_reflns                795
_refine_ls_number_parameters            181
_refine_ls_goodness_of_fit_ref          1.038
_refine_ls_weighting_scheme             calc
_refine_ls_weighting_details
  'w = 1/[\s^2^(Fo^2^) + (0.05000(Max(Fo^2^,0) + 2Fc^2^)/3)^2^]'
_refine_ls_shift/su_max                 0.0004
_refine_diff_density_max                0.24
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(1992, Vol. C, Tables 4.2.6.8 and 6.1.1.1)
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(1992, Vol. C, Tables 4.2.6.8 and 6.1.1.1)
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(1992, Vol. C, Tables 4.2.6.8 and 6.1.1.1)
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_atom_site_fract_z
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_atom_site_adp_type
_atom_site_occupancy
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_atom_site_disorder_assembly
_atom_site_disorder_group
 O(1)   O  0.65574(8)  0.8048(9)    0.5124(4)   0.057(2)    Uani 1.00 d . . .
 O(2)   O  0.65316(7)  0.5506(8)    0.3448(3)   0.048(1)    Uani 1.00 d . . .
 N(1)   N  0.64180(9)  -0.0574(10)  0.7358(4)   0.041(2)    Uani 1.00 d . . .
 C(1)   C  0.7343(1)   -0.012(2)    0.8784(6)   0.084(3)    Uani 1.00 d . . .
 C(2)   C  0.7169(1)   0.197(2)     0.8171(7)   0.067(3)    Uani 1.00 d . . .
 C(3)   C  0.7057(1)   0.196(1)     0.7019(6)   0.055(2)    Uani 1.00 d . . .
 C(4)   C  0.6896(1)   0.404(1)     0.6405(5)   0.049(2)    Uani 1.00 d . . .
 C(5)   C  0.6784(1)   0.395(1)     0.5255(6)   0.050(2)    Uani 1.00 d . . .
 C(6)   C  0.6614(1)   0.600(1)     0.4582(6)   0.039(2)    Uani 1.00 d . . .
 C(7)   C  0.6145(1)   -0.063(1)    0.7342(5)   0.041(2)    Uani 1.00 d . . .
 C(8)   C  0.6000(1)   0.134(1)     0.6446(5)   0.038(2)    Uani 1.00 d . . .
 C(9)   C  0.6111(1)   0.293(1)     0.5689(6)   0.045(2)    Uani 1.00 d . . .
 C(10)  C  0.5970(1)   0.475(1)     0.4859(6)   0.050(2)    Uani 1.00 d . . .
 C(11)  C  0.5717(1)   0.490(1)     0.4812(6)   0.060(3)    Uani 1.00 d . . .
 C(12)  C  0.5336(2)   0.336(2)     0.5512(7)   0.081(3)    Uani 1.00 d . . .
 C(13)  C  0.5220(1)   0.182(2)     0.6257(9)   0.094(4)    Uani 1.00 d . . .
 C(14)  C  0.5359(1)   0.000(2)     0.7093(7)   0.077(3)    Uani 1.00 d . . .
 C(15)  C  0.5611(1)   -0.014(1)    0.7170(6)   0.058(3)    Uani 1.00 d . . .
 C(16)  C  0.5733(1)   0.146(1)     0.6413(6)   0.043(2)    Uani 1.00 d . . .
 C(17)  C  0.5595(1)   0.328(1)     0.5568(6)   0.051(3)    Uani 1.00 d . . .
 H(1)   H  0.7481      -0.0364      0.8380      0.0851      Uiso 1.00 calc . . .
 H(2)   H  0.7401      0.0232       0.9637      0.0851      Uiso 1.00 calc . . .
 H(3)   H  0.7257      -0.1861      0.8759      0.0851      Uiso 1.00 calc . . .
 H(4)   H  0.7135      0.3536       0.8681      0.0744      Uiso 1.00 calc . . .
 H(5)   H  0.7087      0.0424       0.6526      0.0555      Uiso 1.00 calc . . .
 H(6)   H  0.6861      0.5597       0.6879      0.0537      Uiso 1.00 calc . . .
 H(7)   H  0.6822      0.2361       0.4793      0.0565      Uiso 1.00 calc . . .
 H(8)   H  0.6450      -0.1024      0.6545      0.0502      Uiso 1.00 calc . . .
 H(9)   H  0.6483      0.1142       0.7566      0.0502      Uiso 1.00 calc . . .
 H(10)  H  0.6500      -0.1871      0.7914      0.0502      Uiso 1.00 calc . . .
 H(11)  H  0.6123      -0.0212      0.8166      0.0421      Uiso 1.00 calc . . .
 H(12)  H  0.6090      -0.2406      0.7158      0.0421      Uiso 1.00 calc . . .
 H(13)  H  0.6287      0.2787       0.5701      0.0521      Uiso 1.00 calc . . .
 H(14)  H  0.6054      0.5845       0.4348      0.0600      Uiso 1.00 calc . . .
 H(15)  H  0.5623      0.6133       0.4223      0.0679      Uiso 1.00 calc . . .
 H(16)  H  0.5232      0.4552       0.4921      0.0972      Uiso 1.00 calc . . .
 H(17)  H  0.5042      0.1786       0.6189      0.1035      Uiso 1.00 calc . . .
 H(18)  H  0.5272      -0.0995      0.7655      0.0806      Uiso 1.00 calc . . .
 H(19)  H  0.5704      -0.1393      0.7735      0.0653      Uiso 1.00 calc . . .

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_atom_site_aniso_U_33
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 O(2)   0.067(3)  0.041(3)   0.034(2)  -0.001(3)  0.003(2)   -0.004(2)
 N(1)   0.059(4)  0.036(4)   0.029(3)  -0.001(3)  0.007(3)   0.002(2)
 C(1)   0.070(5)  0.103(8)   0.075(5)  0.032(6)   -0.004(4)  0.034(5)
 C(2)   0.063(5)  0.073(6)   0.061(5)  0.013(5)   0.000(4)   0.003(4)
 C(3)   0.060(5)  0.058(6)   0.045(4)  0.010(5)   0.007(4)   0.005(4)
 C(4)   0.052(4)  0.052(5)   0.046(4)  -0.006(4)  0.014(4)   0.004(4)
 C(5)   0.052(4)  0.050(5)   0.049(4)  0.006(4)   0.008(4)   0.007(4)
 C(6)   0.034(4)  0.037(5)   0.047(4)  -0.012(4)  0.013(4)   0.007(4)
 C(7)   0.042(4)  0.032(4)   0.049(4)  -0.001(4)  0.010(4)   -0.004(4)
 C(8)   0.057(5)  0.020(5)   0.034(4)  0.000(4)   0.001(4)   -0.003(3)
 C(9)   0.050(4)  0.039(5)   0.045(4)  -0.002(4)  0.002(4)   0.000(4)
 C(10)  0.063(5)  0.046(5)   0.041(4)  -0.003(4)  0.008(4)   0.008(4)
 C(11)  0.076(6)  0.050(6)   0.050(4)  0.021(5)   -0.006(5)  0.013(4)
 C(12)  0.053(6)  0.109(9)   0.077(6)  0.023(5)   0.003(5)   0.012(5)
 C(13)  0.050(6)  0.117(10)  0.116(8)  0.015(6)   0.014(6)   0.008(7)
 C(14)  0.059(6)  0.091(8)   0.086(6)  -0.013(5)  0.027(5)   -0.001(5)
 C(15)  0.050(5)  0.058(6)   0.069(5)  -0.004(4)  0.016(4)   0.012(4)
 C(16)  0.043(5)  0.042(5)   0.044(4)  -0.004(4)  0.008(4)   -0.010(3)
 C(17)  0.052(6)  0.051(6)   0.048(4)  0.008(5)   -0.002(4)  0.002(4)
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_computing_cell_refinement          'PROCESS-AUTO'
_computing_data_reduction           'teXsan Ver. 1.10'
_computing_structure_solution       SAPI91
_computing_structure_refinement     'teXsan Ver. 1.10'
_computing_publication_material     'teXsan Ver. 1.10'
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# e.g. C1 C2 1.541(1) . . yes
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_geom_torsion_publ_flag
# -- ENTER TORSION ANGLES HERE, ONE PER LINE --
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_geom_contact_publ_flag
       ?  ?    ?       ? ?  ?
# -- ENTER BONDS HERE, ONE PER LINE --
# e.g. O1 N1 1.541(1) . . yes
#------------------------------------------------------------------------------
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