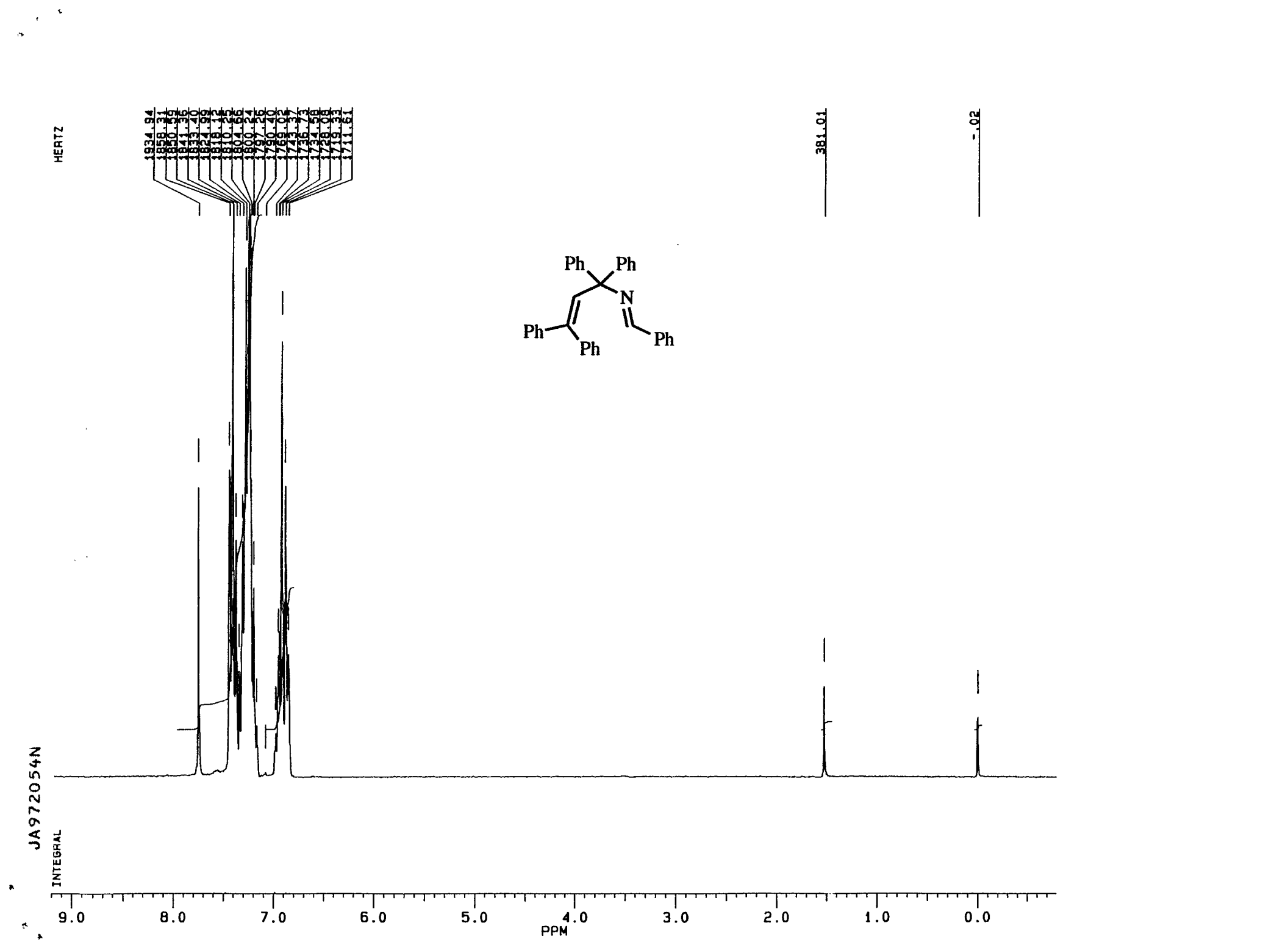
JOURNAL OF THE AMERICAN CHEMICAL SOCIETY
J. Am. Chem. Soc., 1997, 119(51), 12659-12660, DOI:10.1021/ja972054n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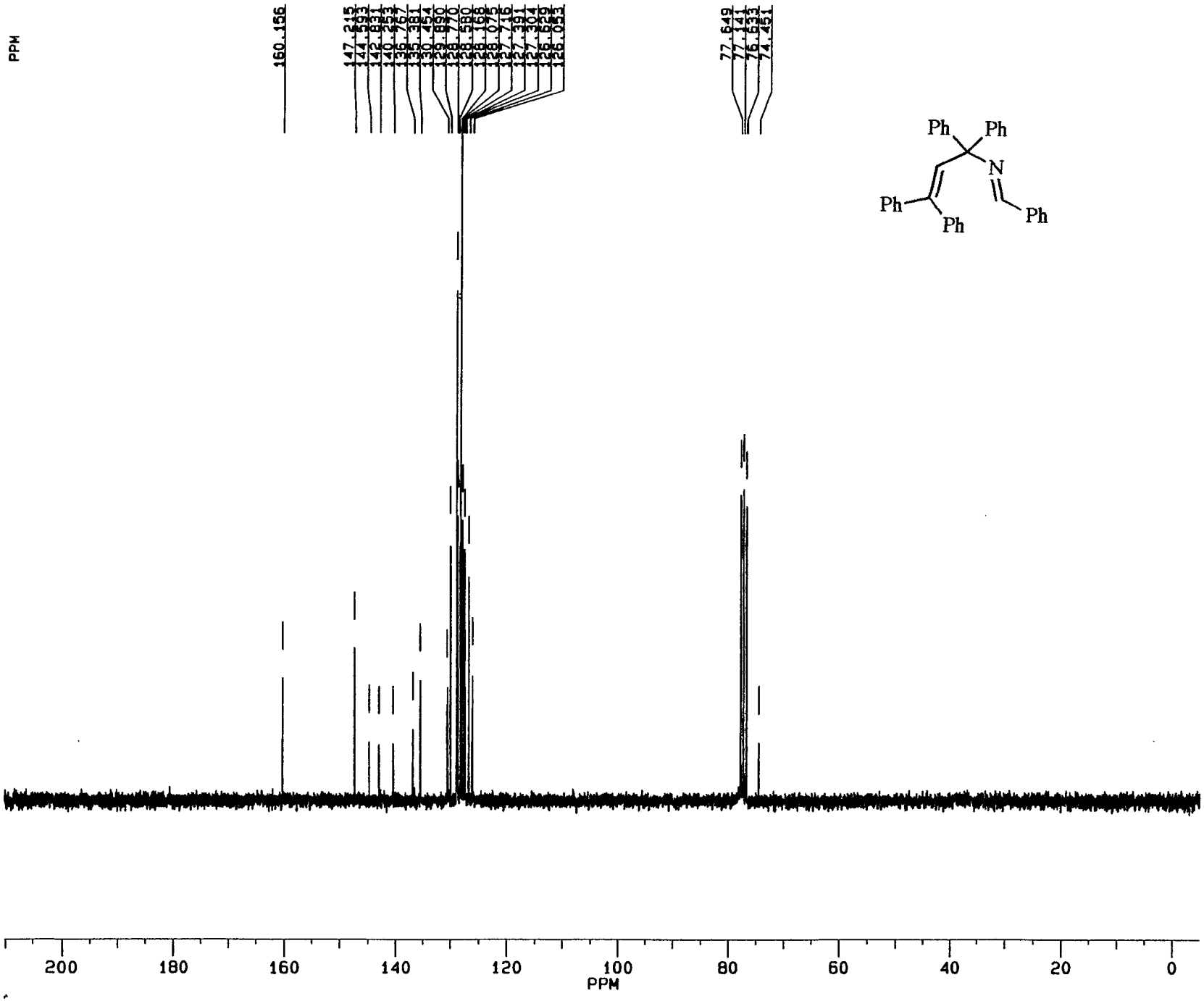
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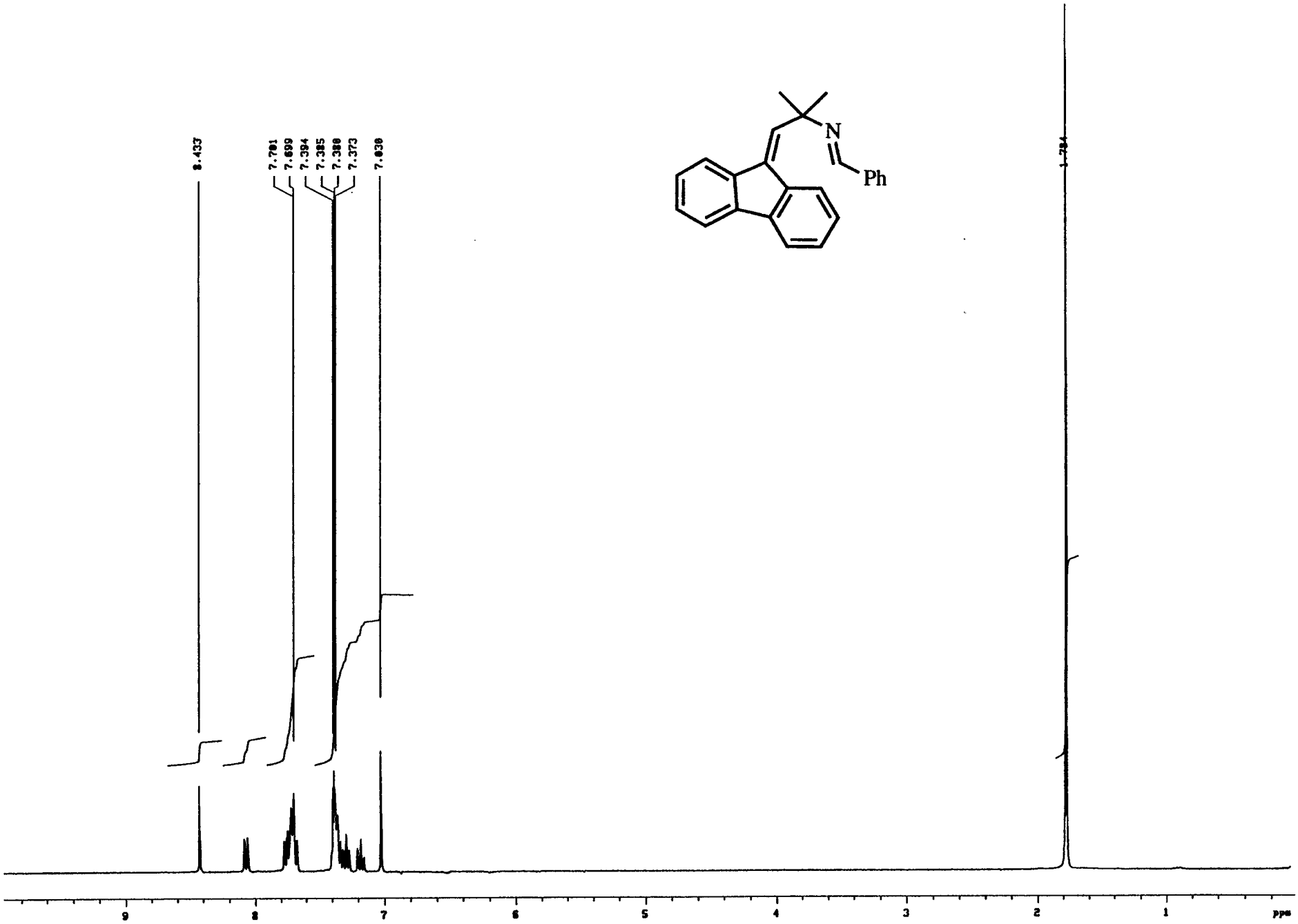
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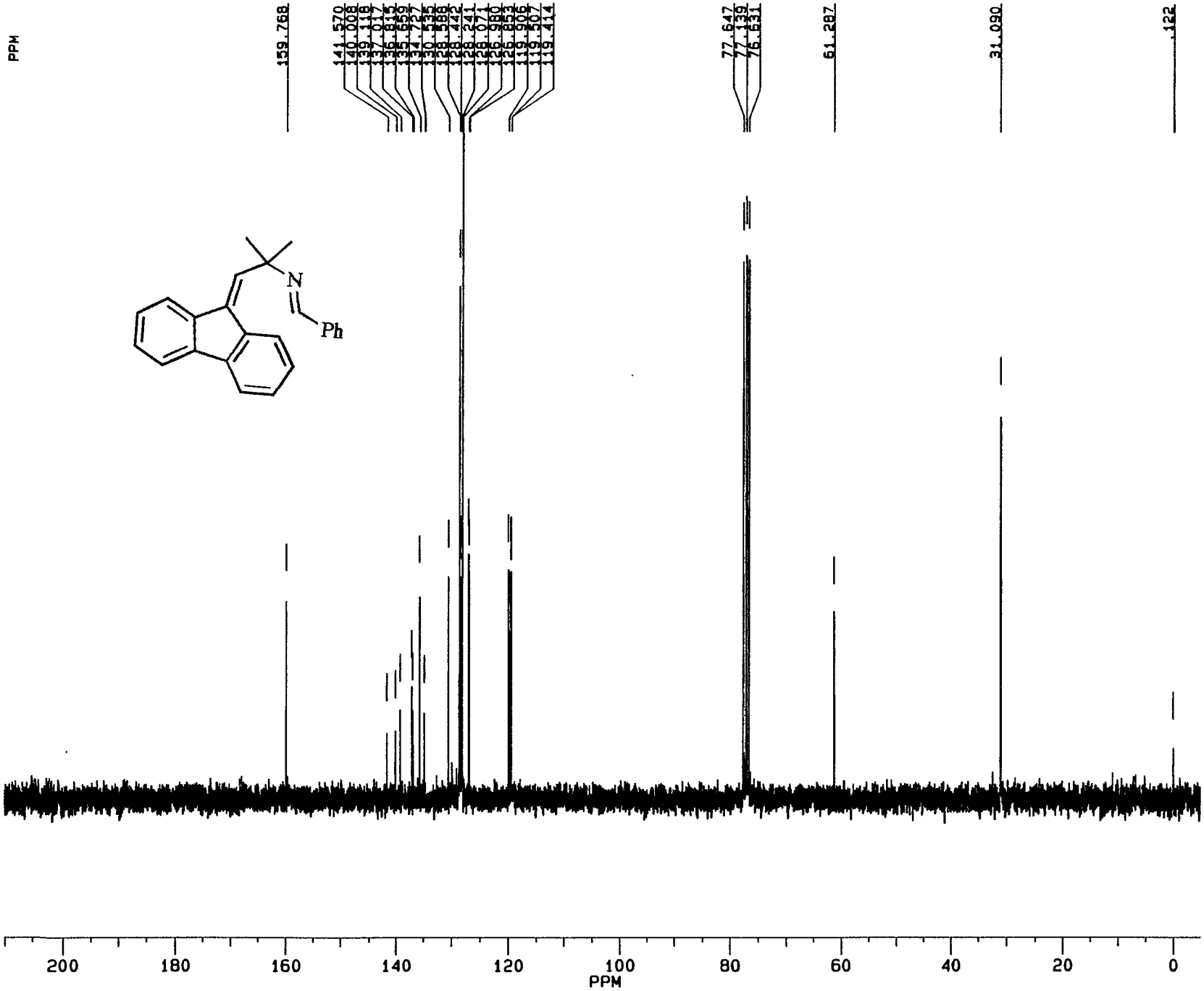
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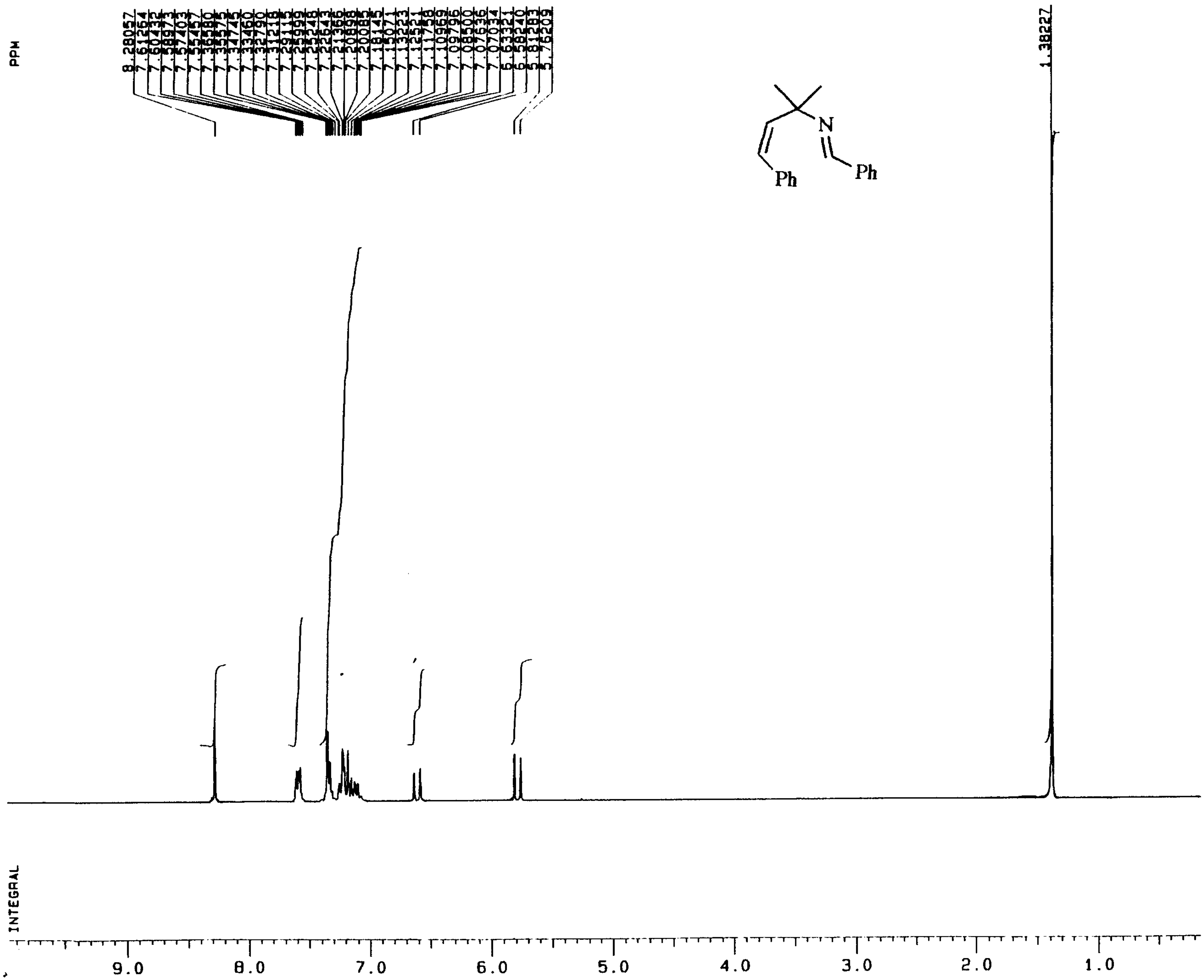
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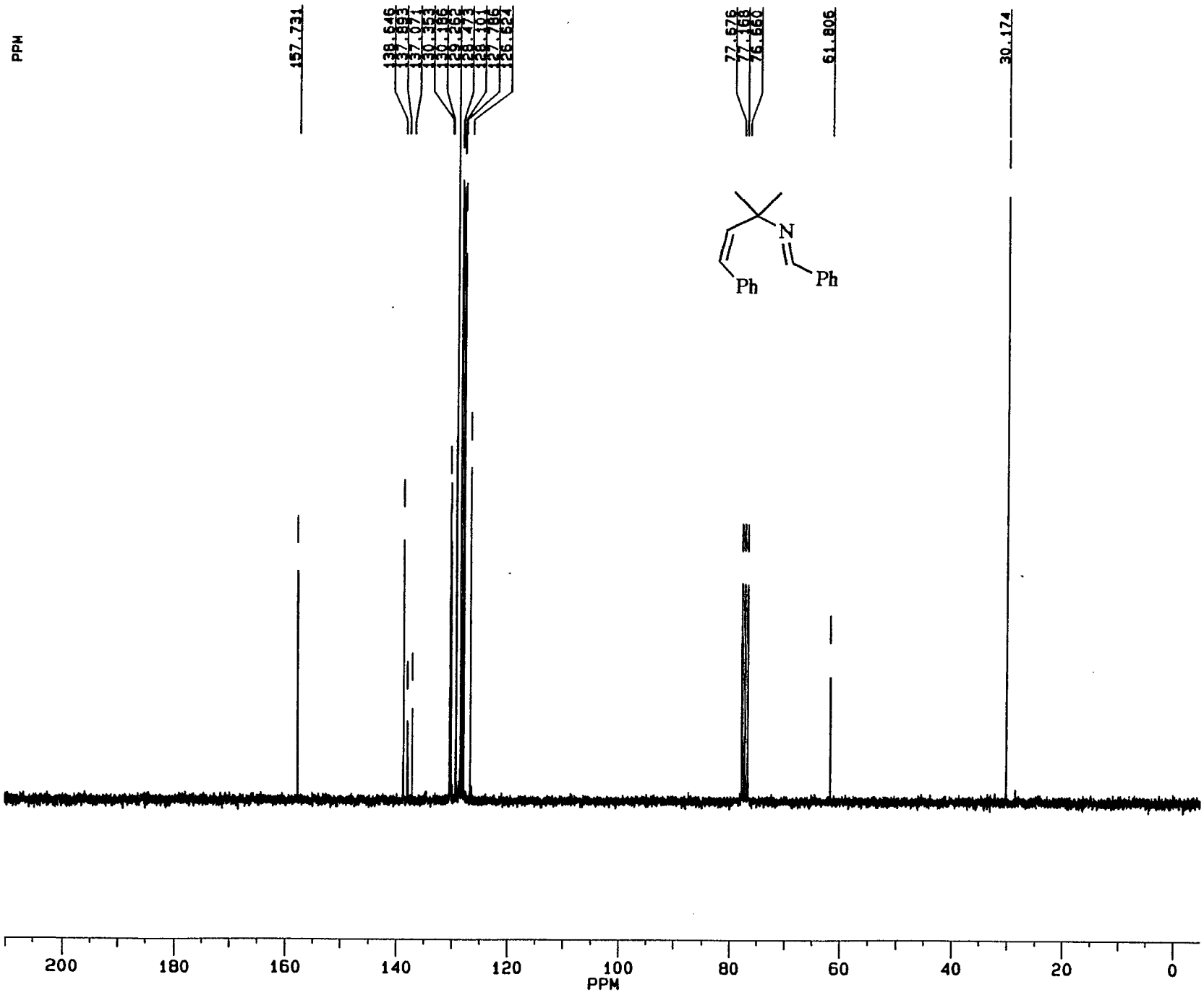
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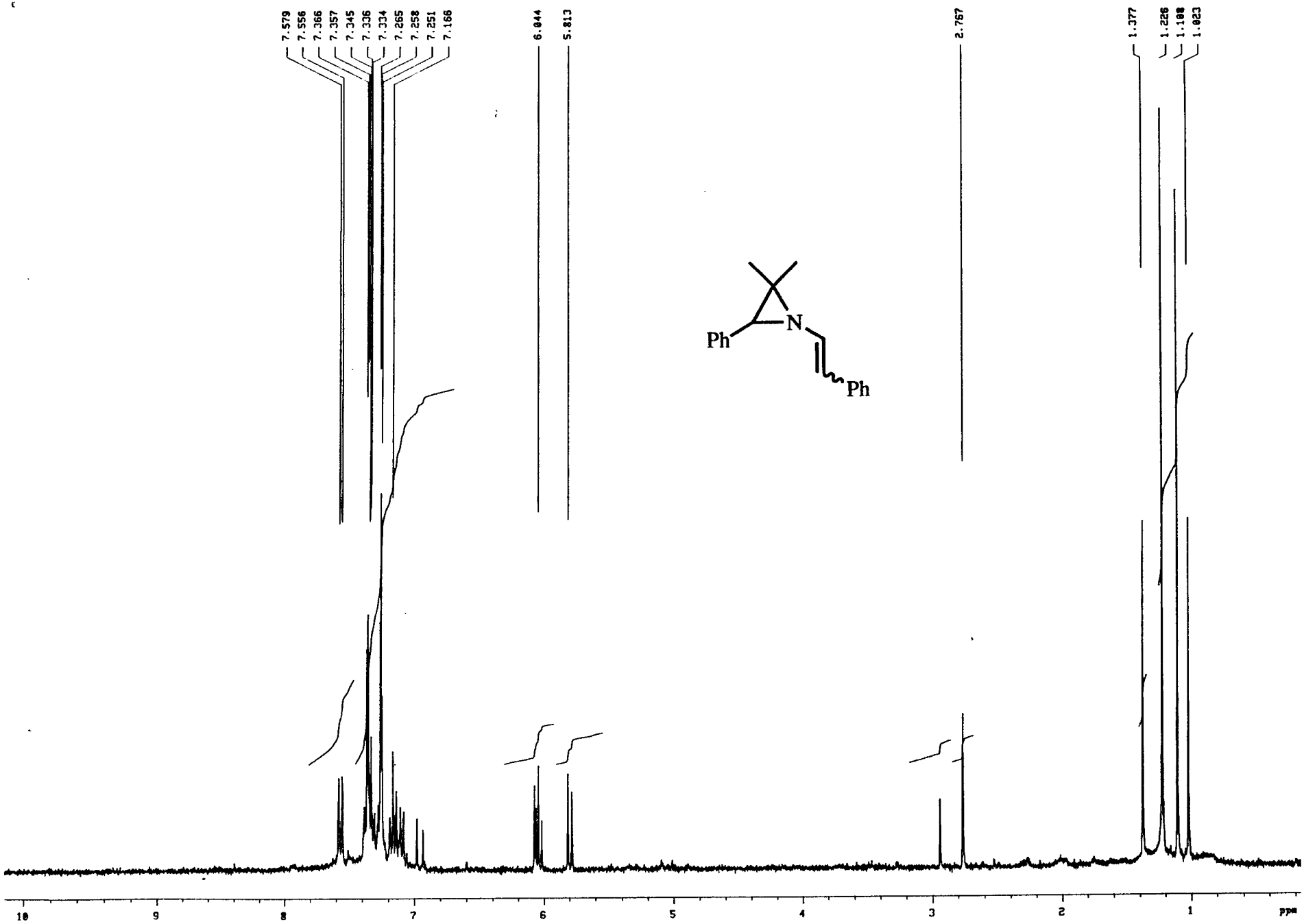
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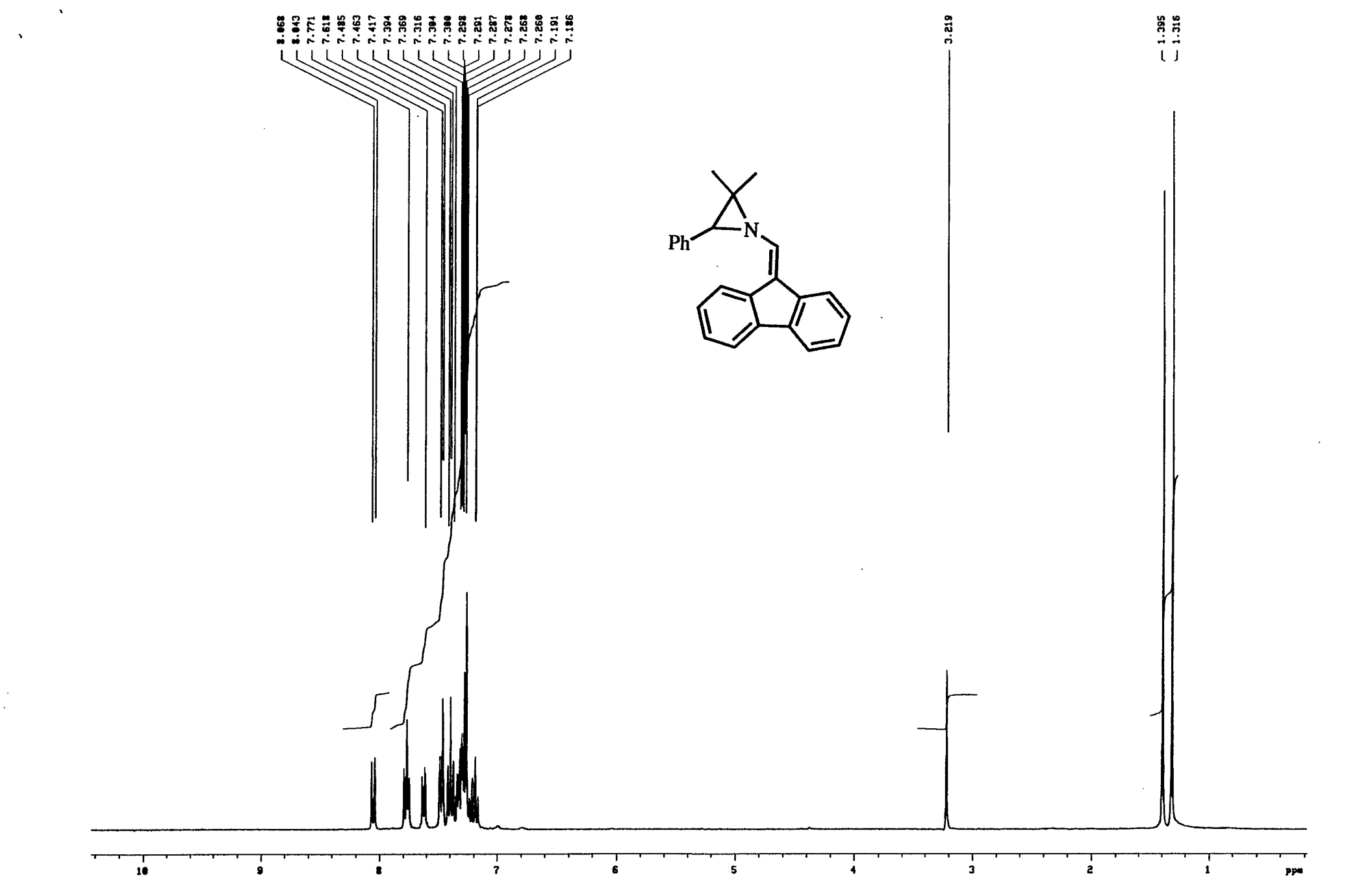
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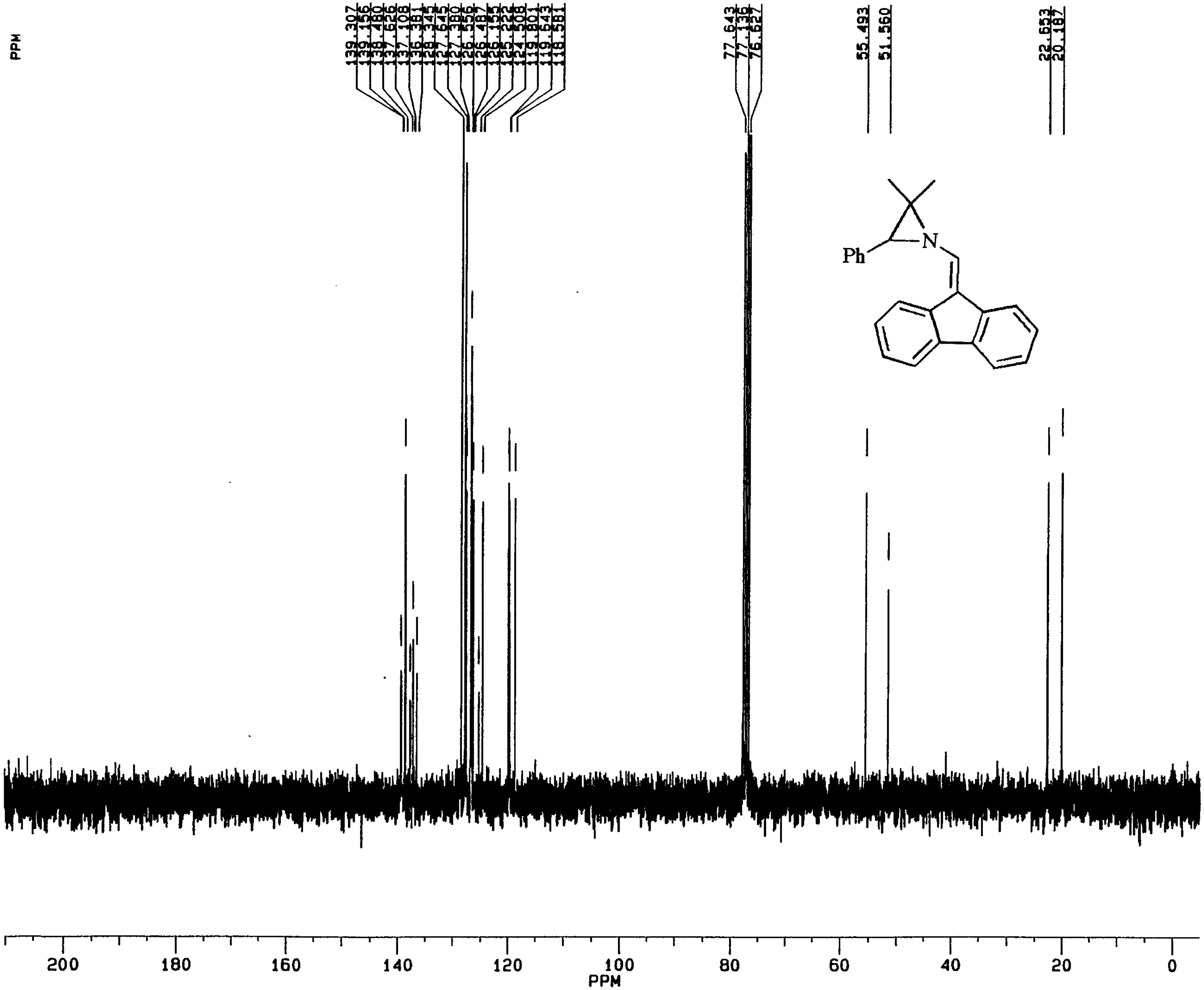
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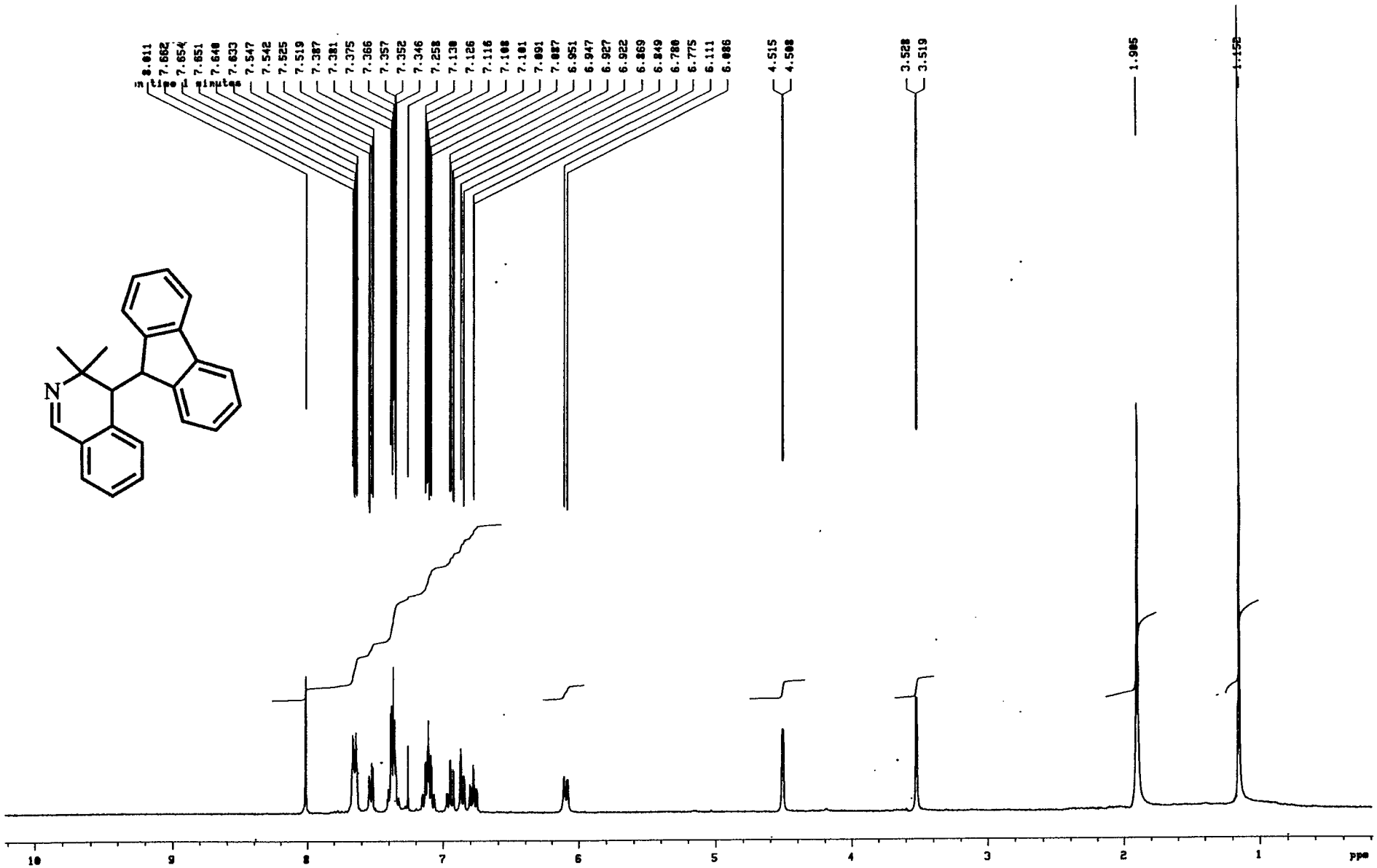
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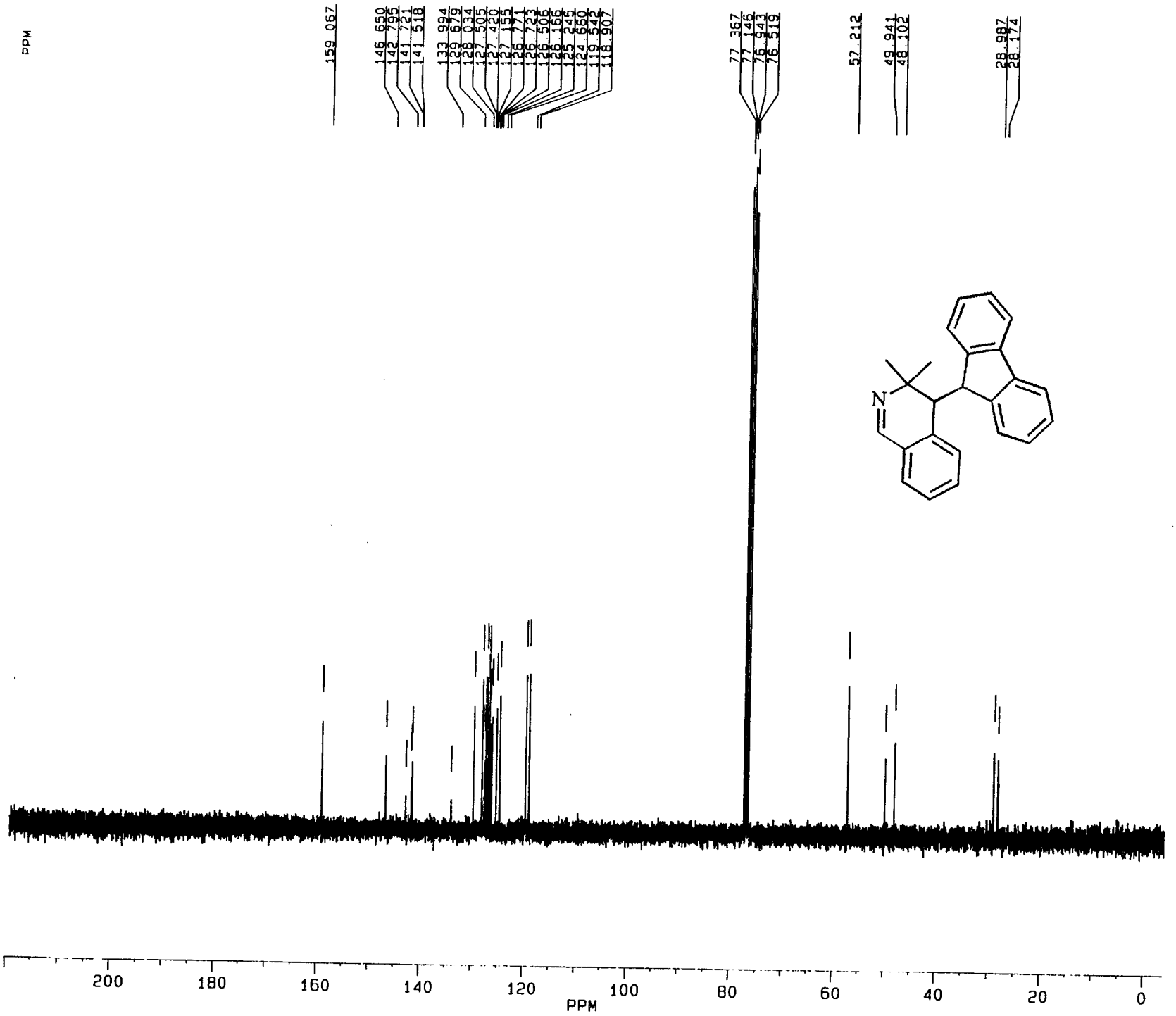
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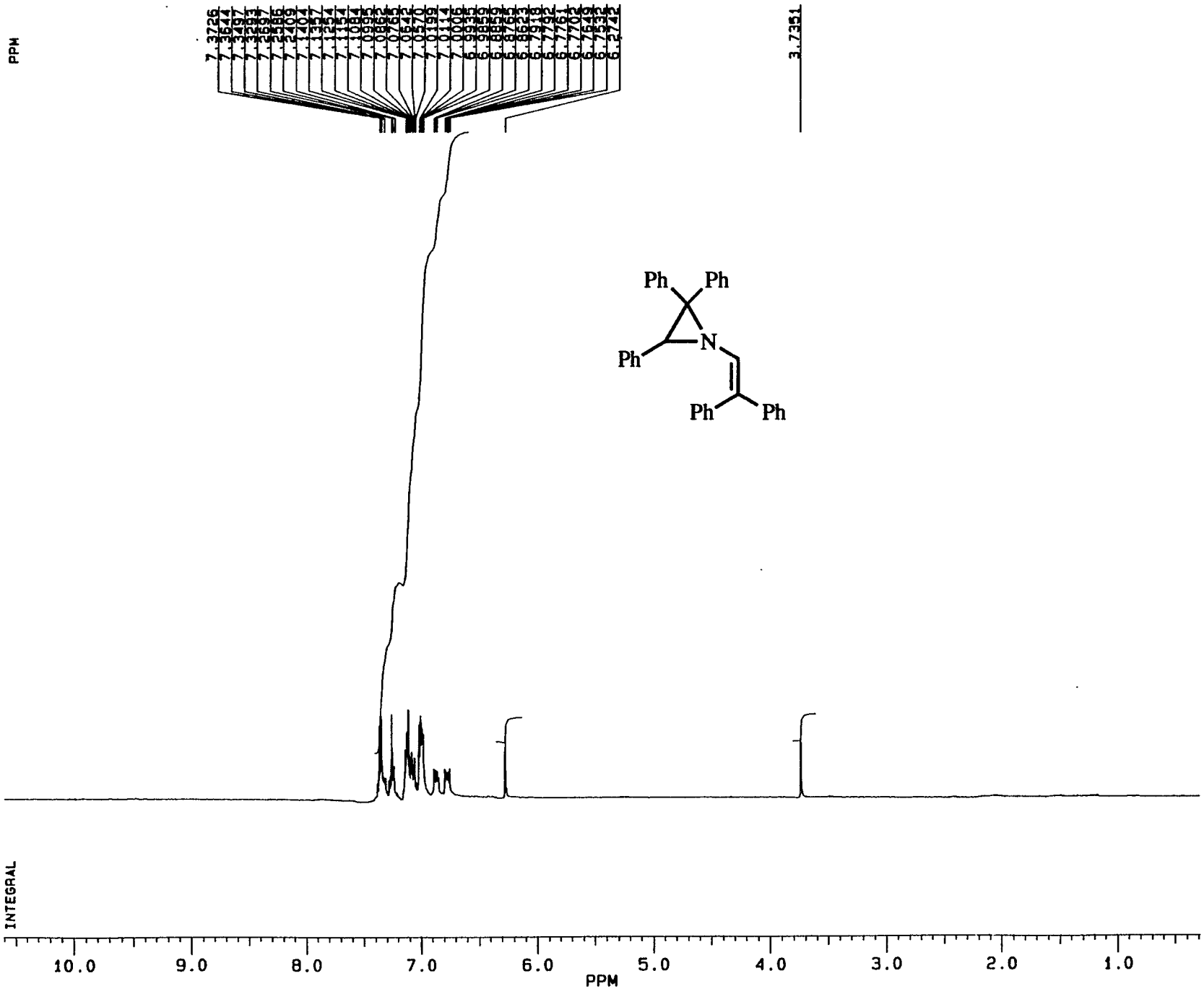
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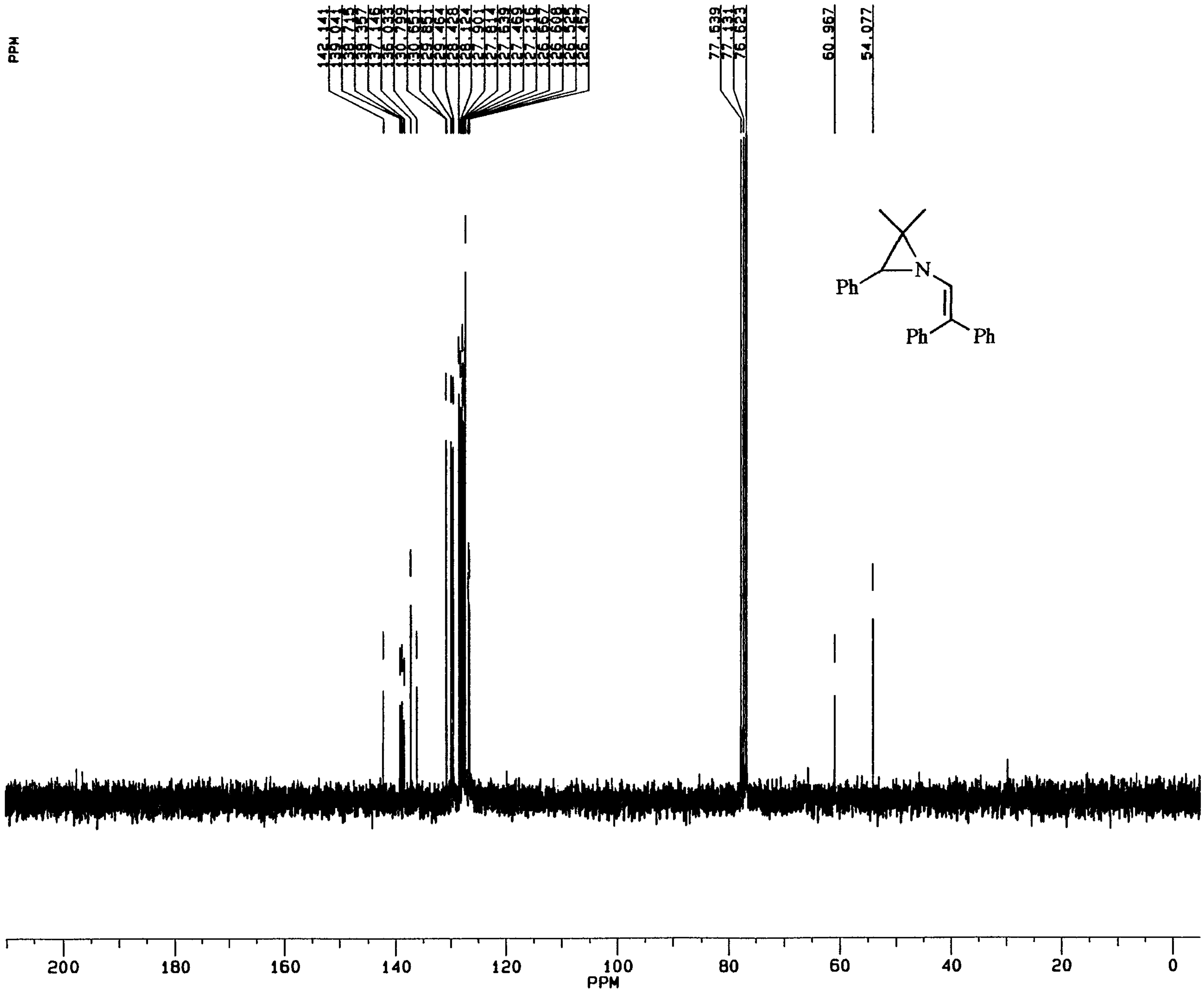

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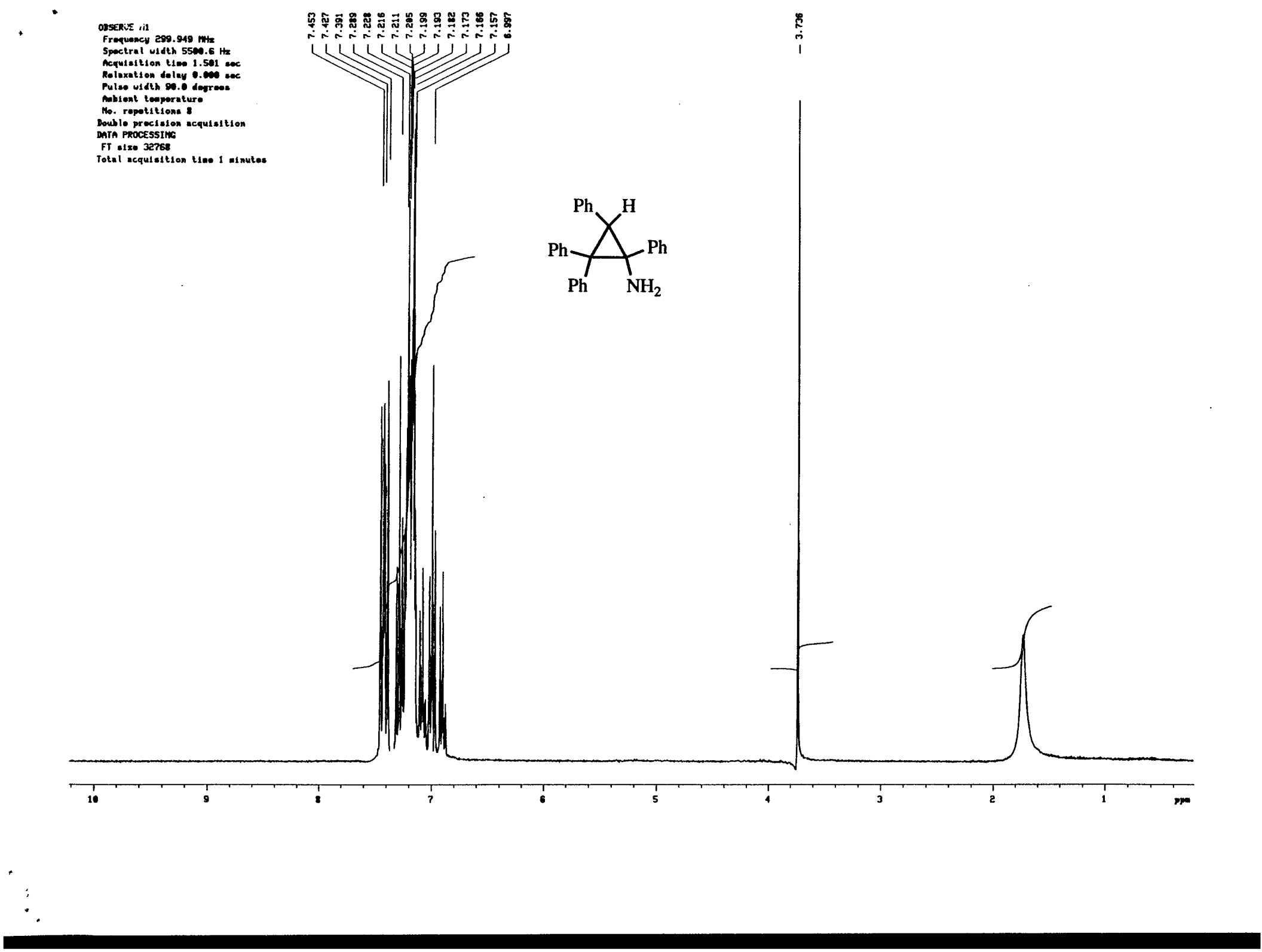
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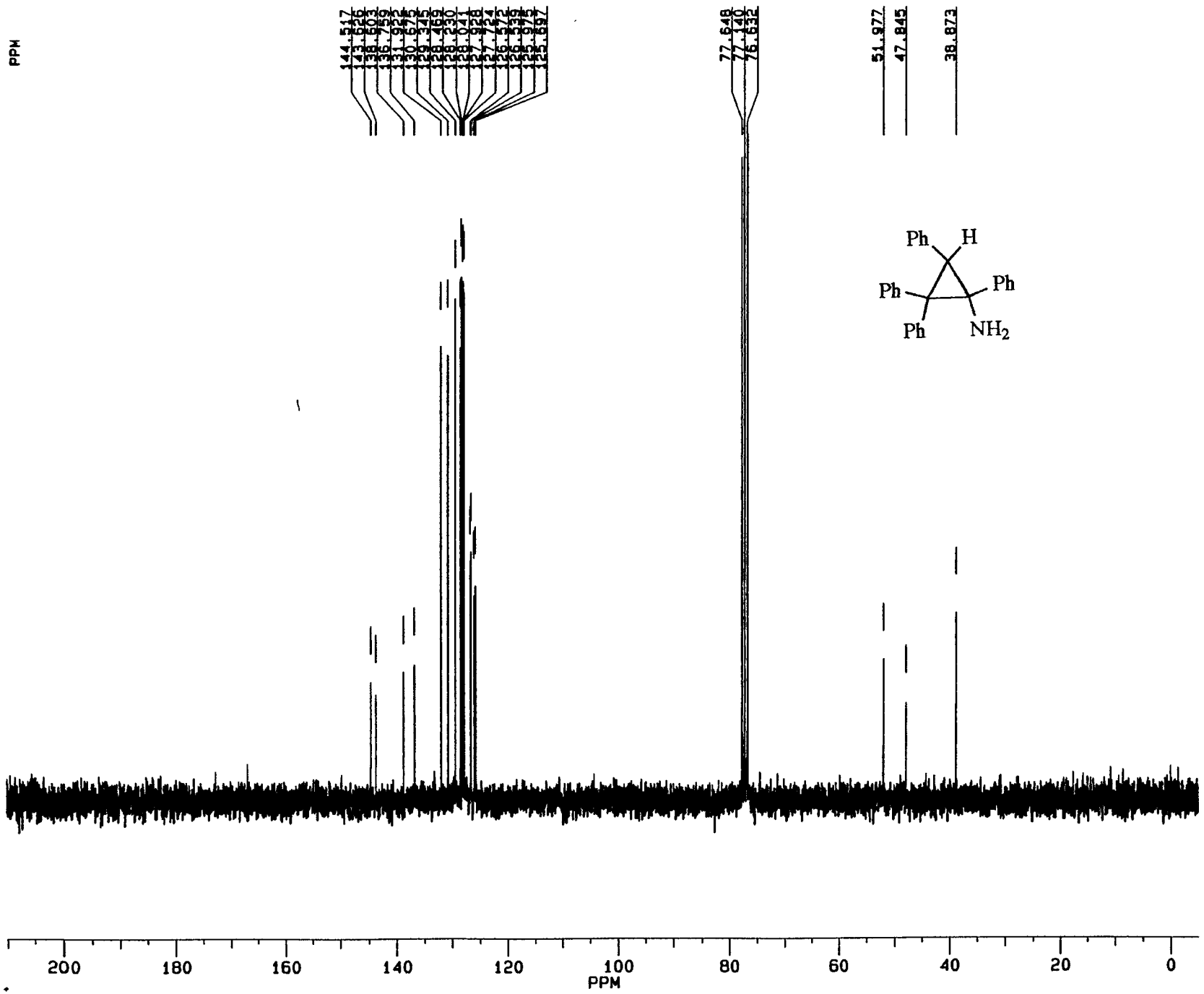
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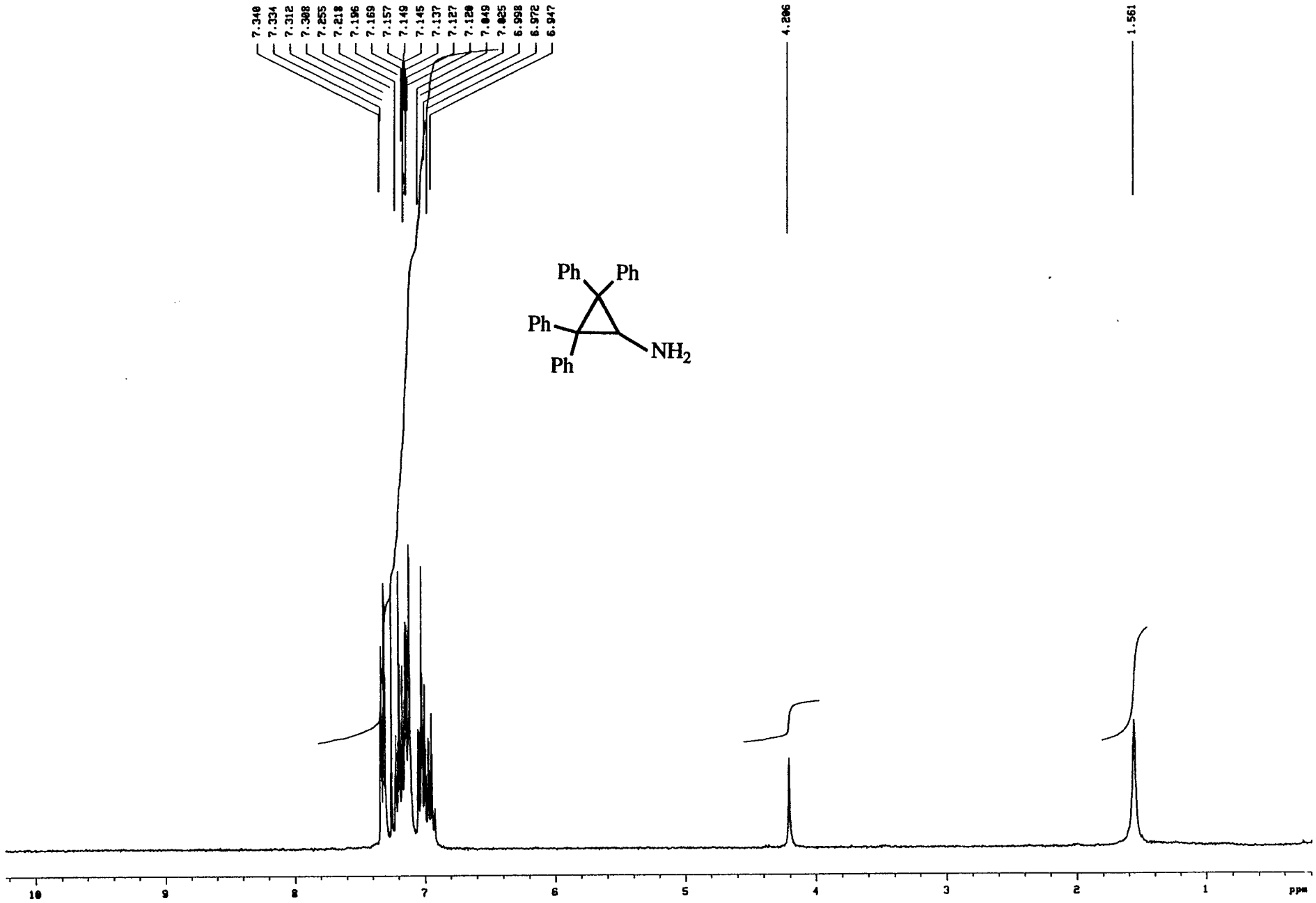
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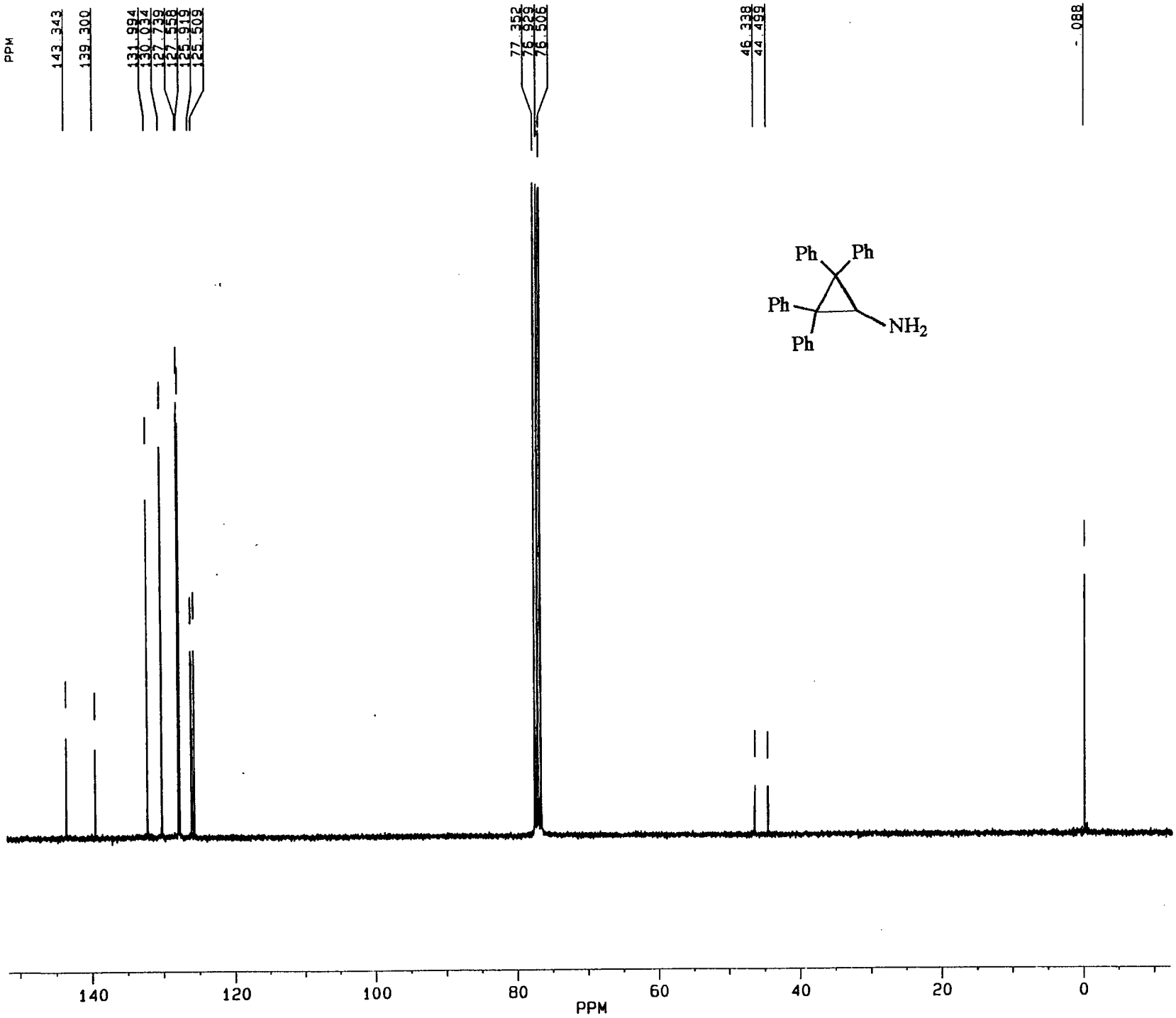
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Table . Crystal and Refinement Data for $C_{34}NH_{27}$

Formula	$C_{34}NH_{27}$
Mol weight	449.6
Crystal dimensions, mm	0.3x0.3x0.30
Crystal color	colorless
Crystal system	Triclinic
Space group	P(-1) (N. 2)
Cell dimensions	
a, Å	10.843(2)
b, Å	11.223(5)
c, Å	11.654(3)
α, (°)	99.64(2)
β, (°)	96.75(2)
γ, (°)	111.05(3)
Z	2
V, Å^3	1280.2(8)
D_{calcd}, g cm^{-3}	1.17
F(000)	476
temp, K	295
Diffractometer	Enraf-Nonius
Radiation	graphite-monochromated Mo *Ka* (l=0.71069 Å)
Scan technique	*ω/2θ*
2θ range, °	1-60
Data collected	$-15 \le h \le 15$, $-15 \le k \le 15$, $0 \le l \le 16$
Unique data	7453
Observed reflections, I>2σ(I)	3808
R_{int}(%)	1.05
Decay	<1%
Standard reflections	3/75
μ(Mo *Ka*), cm^{-1}	0.62
Transmission range	0.95-0.98
Weighting scheme	unit
Maximum and average shift/error	0.006, 0.001
Absorption correction range	none
Maximum residual, eÅ^{-3}	0.2
$R = \Sigma \lvert F_o \rvert - \lvert F_c \rvert / \Sigma \lvert F_o \rvert$	0.042
$R_w = [\Sigma w(\lvert F_o \rvert - \lvert F_c \rvert)^2 / \Sigma w \lvert F_o \rvert^2]^{1/2}$	0.040
GOF (F)	0.47
Num.Refined Parameters	316
Reflections/parameter ratio	12.05

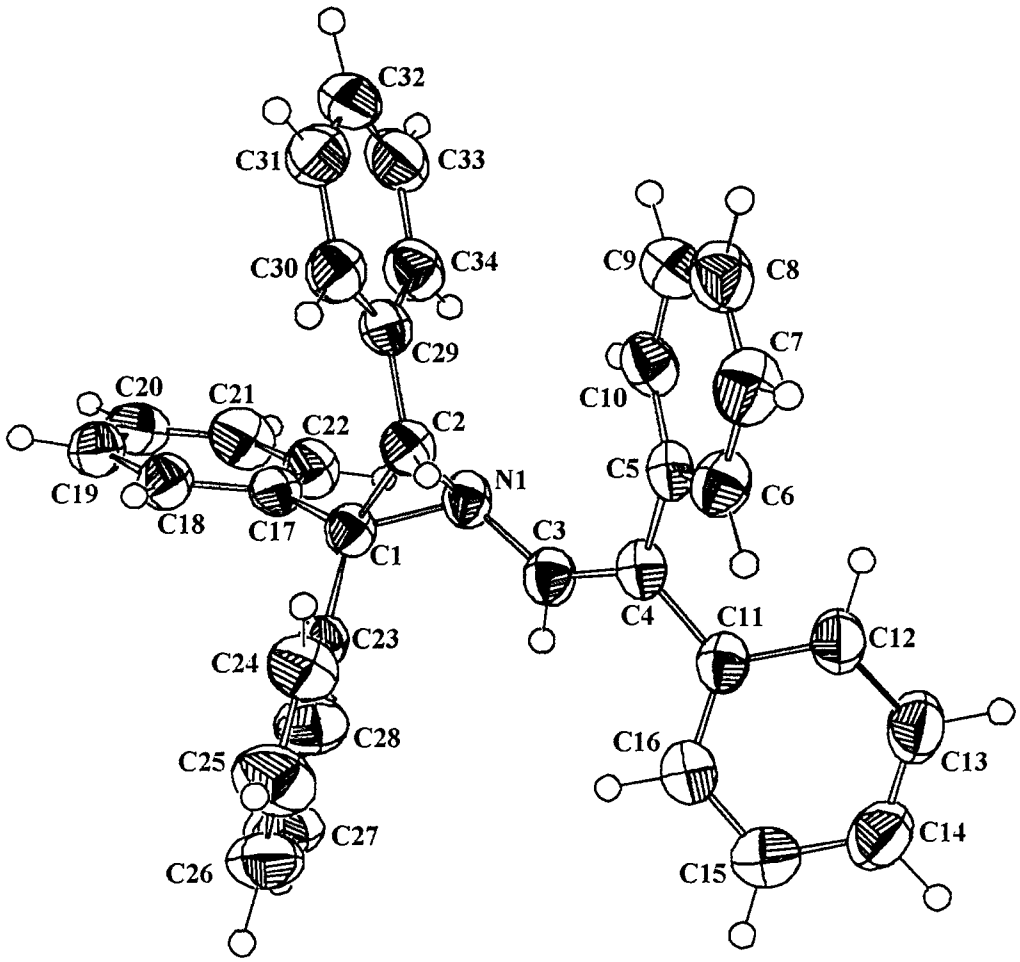

FIGURE *: ORTEP drawing of the molecule of $C_{34}NH_{27}$ with 50% probability ellipsoids. The labeling of hydrogen atoms has been omitted for clarity

EXPERIMENTAL

1. Data Collection

A colorless crystal of prismatic shape and dimension 0.30x0.30x0.30 mm^3 was cut from a larger one grown as described in the experimental section. This crystal was mounted in a Nonius CAD-4 diffractometer to be used for the structure determination. Table I collects the main crystal data and refinement parameters for $C_{34}NH_{27}$. A graphite-monochromatic MoK_α (λ=0.71069Å) beam was used in the data collection carried out at room temperature. The unit cell parameters were determined by least-square refinement of the 2q values of 25 strong well centered reflections in the range 12°<2q<30° The intensities of all 7453 unique reflections (after merging) were measured in the angular range 1°<2q<60° (hkl range -15,-15,0 to 15,15,16) using the $\omega/2\theta$ scan technique. There was no appreciable change in the intensities of three standard reflections periodically monitored. The raw data were corrected for Lorentz and polarization effects and 3808 reflections were considered as observed with I>2s(I). Scattering factors for neutral atoms were taken from *"International Tables for X-Ray Crystallography"* (1).

2. Structure Determination

The position of non-hydrogen atoms were determined using the direct-methods program MULTAN80 (2). The hydrogen atoms were located by Fourier synthesis. Due to the regular crystal shape and to the low absorption coefficient no absorption correction was applied. Since no trends on ΔF vs. F_o or $\sin\theta/\lambda$ were observed no especial weighting scheme was used, i.e. unit weights were assigned. A final refinement was undertaken with anisotropic thermal motion for the non-hydrogen atoms, while the hydrogen atoms positions were refined but keeping the isotropic thermal parameters constant and equal for all of them. Full-matrix least-squares refinement minimizing $\Sigma w(|Fo|-|Fc|)^2$ led to agreement factors R=0.042 and R_w=0.040. The maximum and average shift-to-error ratios were 0.006 and 0.001 respectively, while the maximum residual electron density near the C23-C28 phenyl ring was 0.2e$Å^{-3}$. Most of the calculations were carried out with the XRAY80 program (3).

(1) *International Tables for X-Ray Crystallography*; Kynoch Press; Birmingham, U.K., Vol. **IV**, p 72., (1974)

(2) P.Main, S.J.Fiske S.E.Hull, L.Lessinger, G.Germain, J.P.Declercq & M.M.Woolfson, MULTAN80, *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univ. of York, Engalnd, (1980)

(3) J.M.Stewart, *The X-RAY80 System*; Computer Science Center, University of Maryland, College Park, MD, (1980).