Neutral and Cationic Alkyl Tantalum Imido Complexes: Synthesis and Migratory Insertion Reactions

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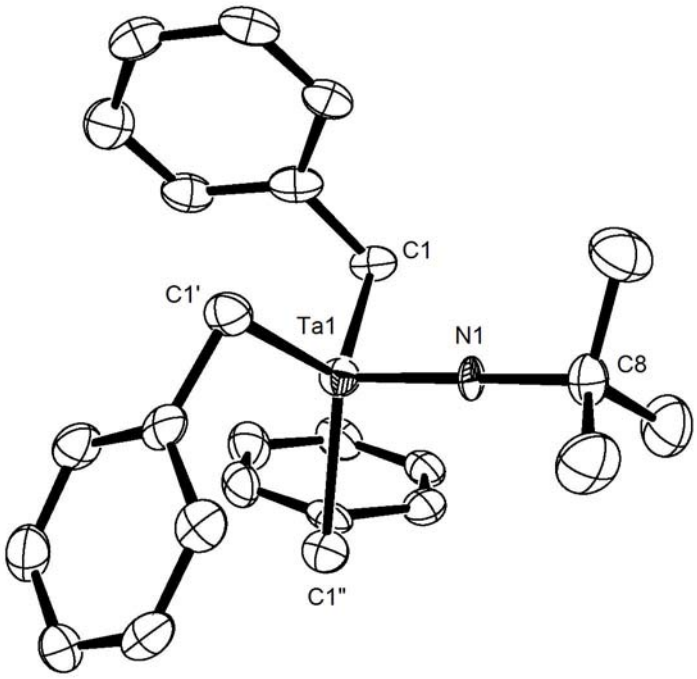
Supporting Information

General Procedures for X-Ray Crystallography. Relevant data for **1** are summarized in Table 1. A crystal of appropriate size was mounted on a glass capillary using Paratone-N hydrocarbon oil. The crystal was transferred to a Siemens SMART diffractometer/CCD area detector,[1] centered in the beam, and cooled by a nitrogen flow low-temperature apparatus that had been previously calibrated by a thermocouple placed at the same position as the crystal. Preliminary orientation matrices and cell constants were determined by collection of 60 10-second frames, followed by spot integration and least-squares refinement. An arbitrary hemisphere of data was collected and the raw data were integrated using SAINT.[2] Cell dimensions reported in Table 1 were calculated from

all reflections with $I > 10\ \sigma$. The data were corrected for Lorentz and polarization effects, but no correction for crystal decay was applied. Data were analyzed for agreement and possible absorption using XPREP.[3] An empirical absorption correction based on comparison of redundant and equivalent reflections was applied using SADABS.[4] The structure was solved and refined with teXsan software package.[5] ORTEP diagrams were created using ORTEP-3 software package.[6]

Figure S1. Ortep Diagram of Compound **1**.



EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{25}H_{30}NTa$
Formula Weight	525.47
Crystal Color, Habit	yellow, block
Crystal Dimensions	0.330 x 0.240 x 0.150 mm
Crystal System	Trigonal
Lattice Type	Primitive
Lattice Parameters	a = 11.158(1) Å b = 11.158(1) Å c = 10.156(1) Å a = 90 ° b = 90 ° g = 120° V = 1095.14(7) Å^3
Space Group	P31c
Z value	2
D_{calc}	1.593 g/cm^3
F_{000}	520.00
μ(MoKα)	5.02 cm^{-1}

B. Intensity Measurements

Diffractometer	SMART CCD
Radiation	MoKα(λ = 0.7107 Å) graphite monochromated
Detector Position	60.5 mm
Exposure Time	10 s seconds per frame.
Scan Type	ω (0.3 degrees per frame)
No. of Reflections Measured	Total: 4878 Unique: 2340 (R_{int} = 0.0413)
Corrections	Lorentz-polarization Absorption (T_{max} = 0.58, T_{min} = 0.32)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares

Function Minimized	$\Sigma w(\|F_o\|^2 - \|F_c\|^2)^2$
Least Squares Weighting scheme	$w = 1/[\sigma^2(F_o^2) + (qP)^2 + 0.000P]$ where $P = [F_o^2 + 2F_c^2]/3$
p-factor	0.030
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (I>2.00□(I))	2062
No. Variables	81
Reflection/Parameter Ratio	25.46
Residuals: R; R_w; R_{all}	0.0239; 0.0307; 0.0258
Goodness of Fit Indicator	1.253
Max Shift/Error in Final Cycle	0.0000
Maximum peak in Final Diff. Map	0.50 $e^-/Å^3$
Minimum peak in Final Diff. Map	-1.48 $e^-/Å^3$

(1) *SMART: Area-Detector Software Package*, Siemens Industrial Automation, Inc.: Madison, WI, **1995**.

(2) *SAINT: SAX Area Detector Integration Program, V4.024*, Siemens Industrial Automation, Inc.: Madison, WI, **1995**.

(3) *XPREP: Part of SHELXTL Crystal Structure Determination Package, V5.03*, Siemens Industrial Automation, Inc.: Madison, WI, **1995**.

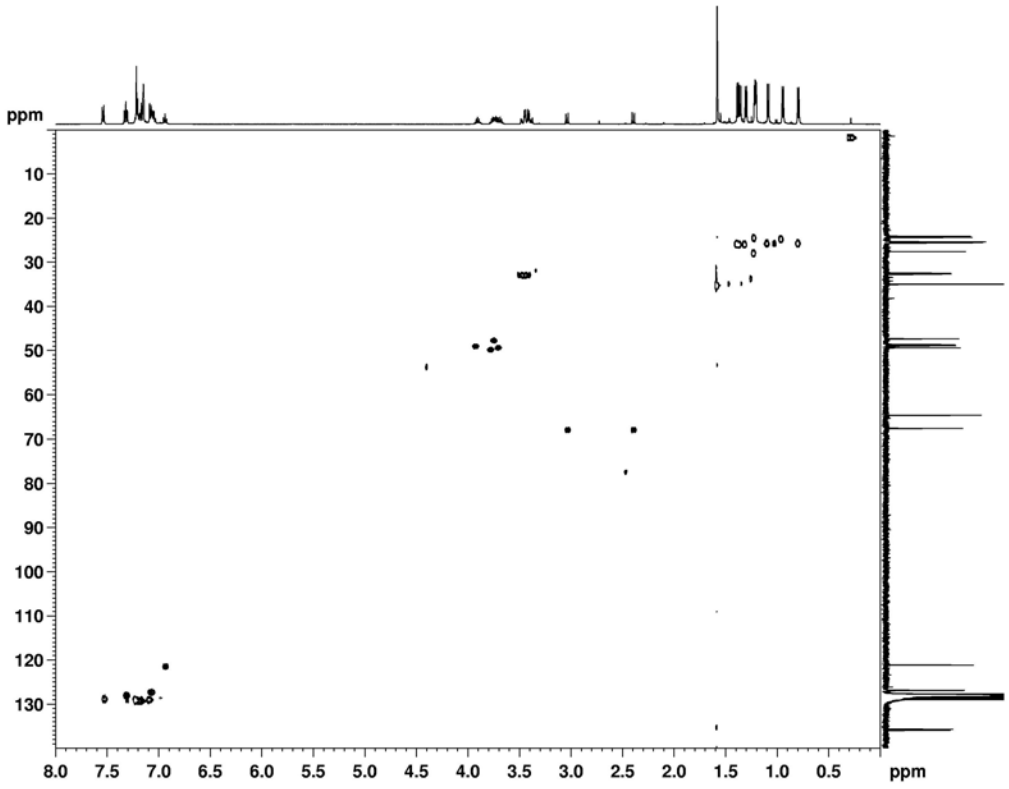
(4) *SADABS: Siemens Area Detector ABSorption correction program* **1996**.

(5) *TeXsan: Crystal Structure Analysis Software Package*, Molecular Structure Corporation: The Wodlands, TX, **1992**.

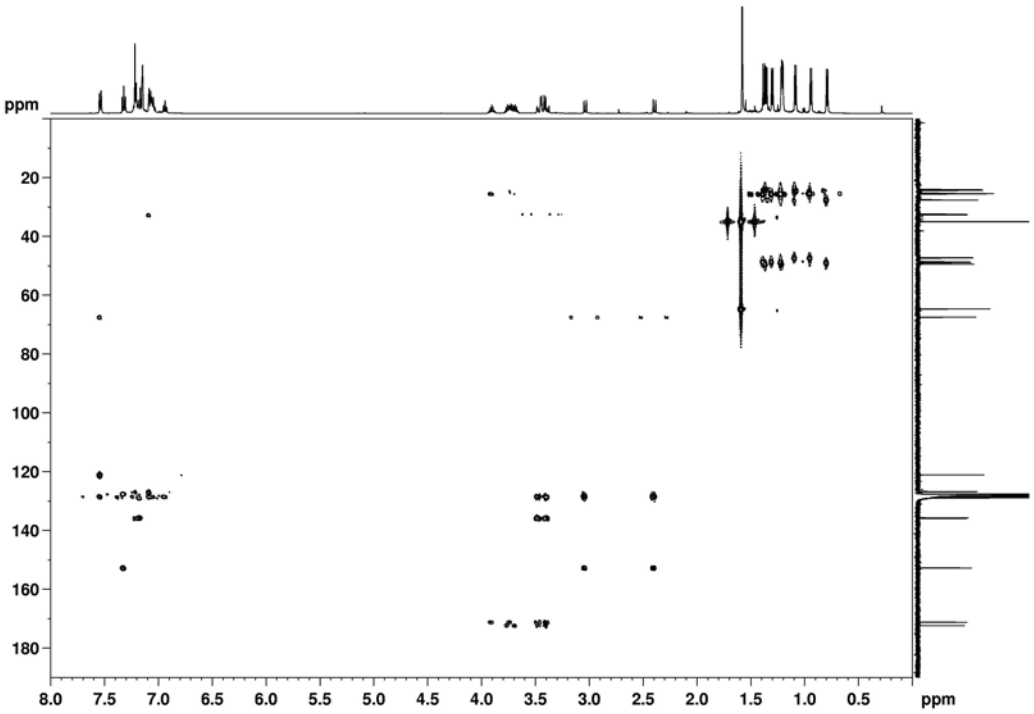
(6) Farrugia, L. J. *J. Appl. Cryst.* **1997**, *30*, 565.

Images of 2D spectra for Compounds 5, 6, 7, 10, 12, 13, and 15 and a ^{1}H NMR spectrum of 5.

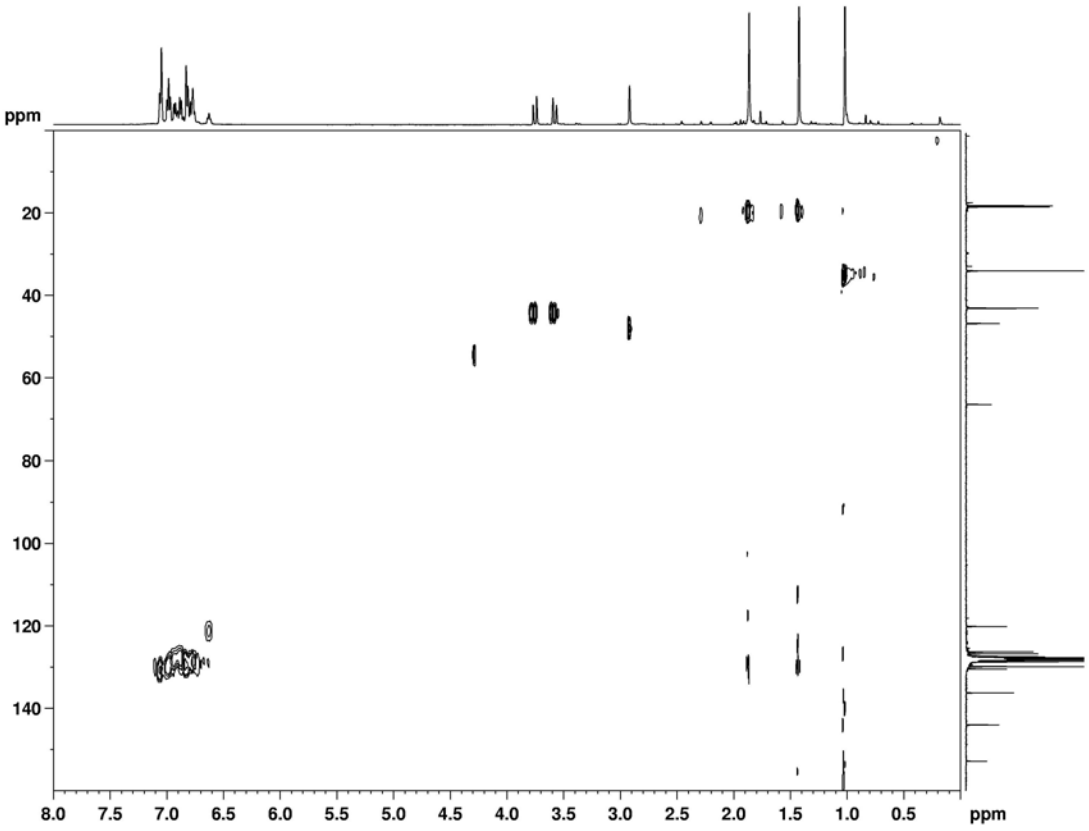
^{1}H-^{13}C HMQC Spectrum for Compound **5**



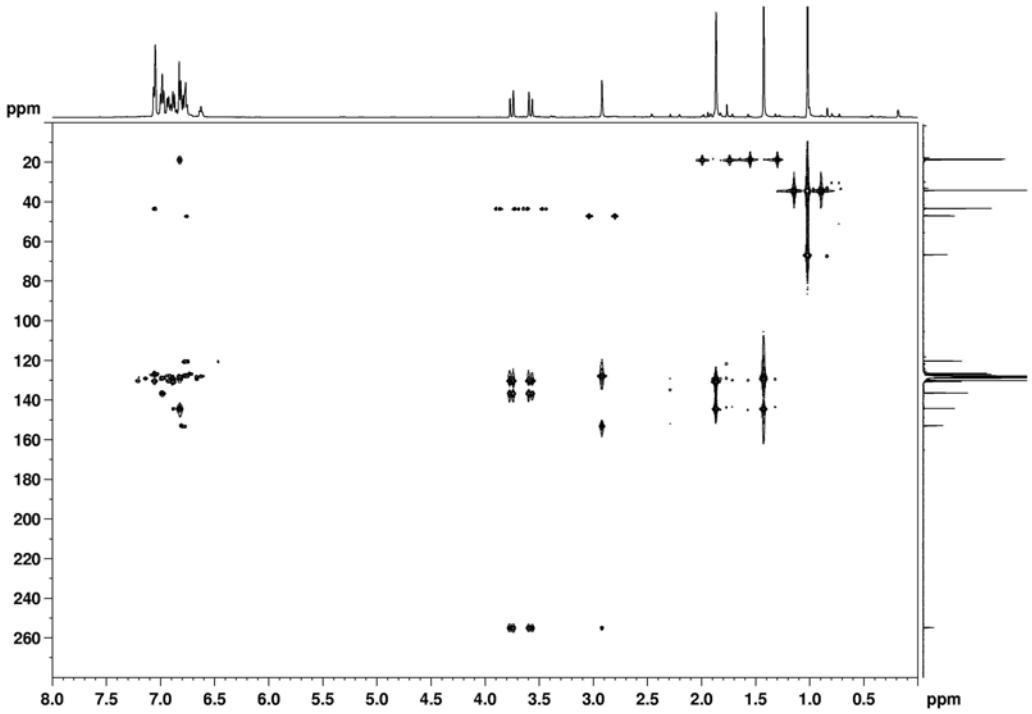
^{1}H-^{13}C HMBC Spectrum for Compound **5**



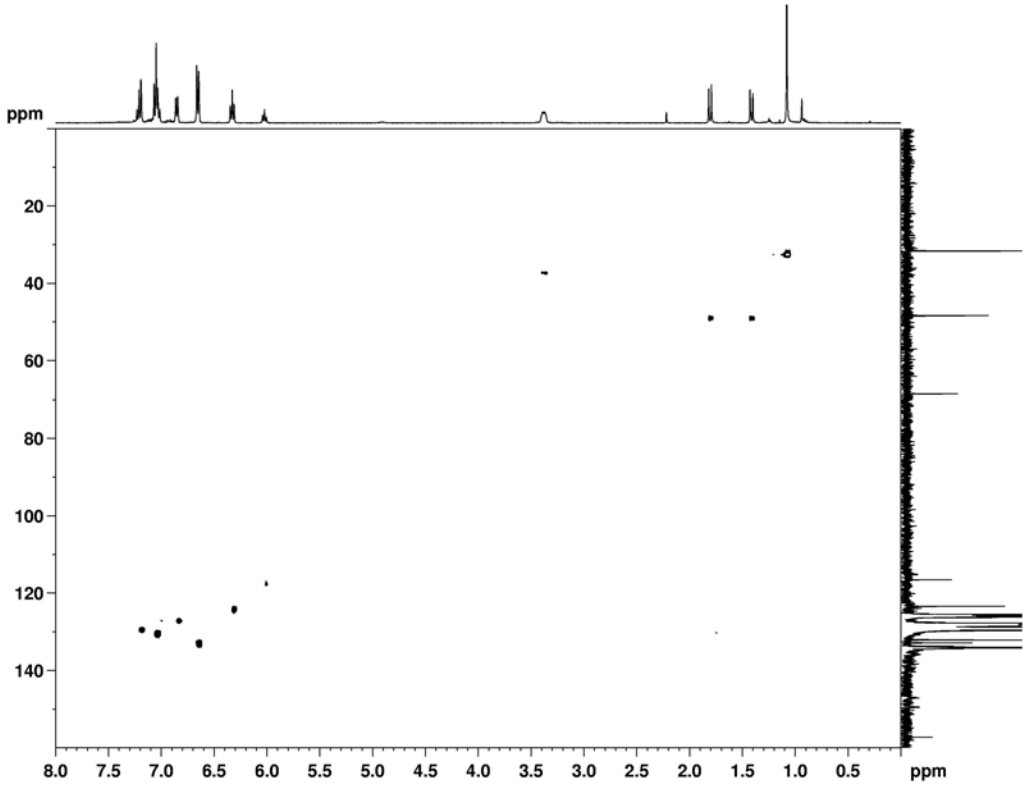
^{1}H-^{13}C HMQC Spectrum for Compound **6**



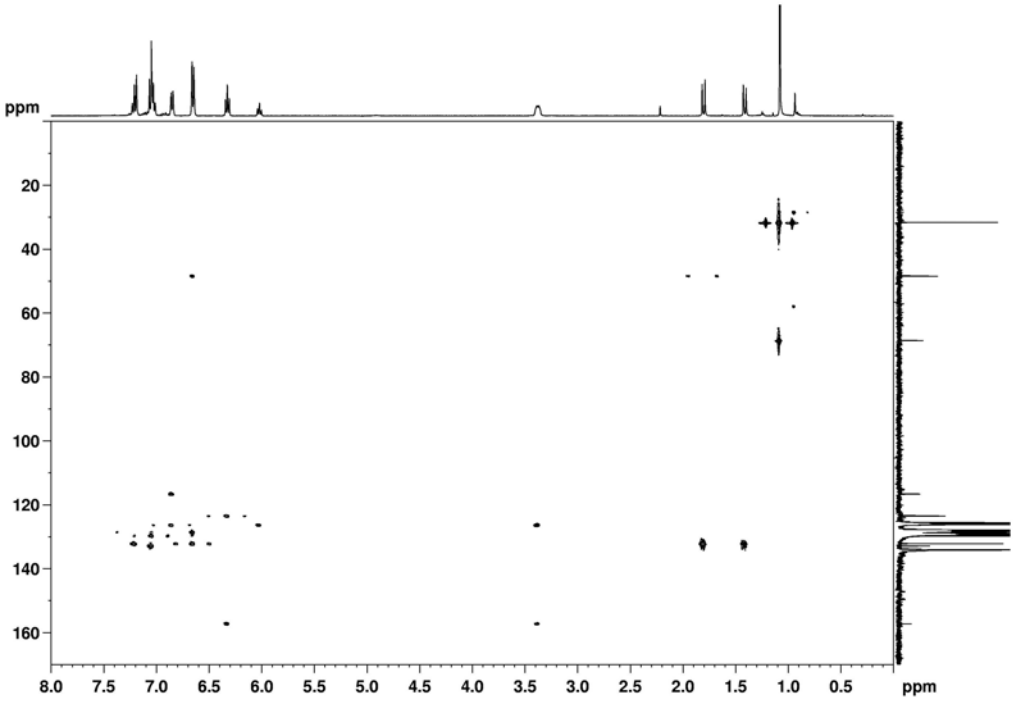
^{1}H-^{13}C HMBC Spectrum for Compound **6**



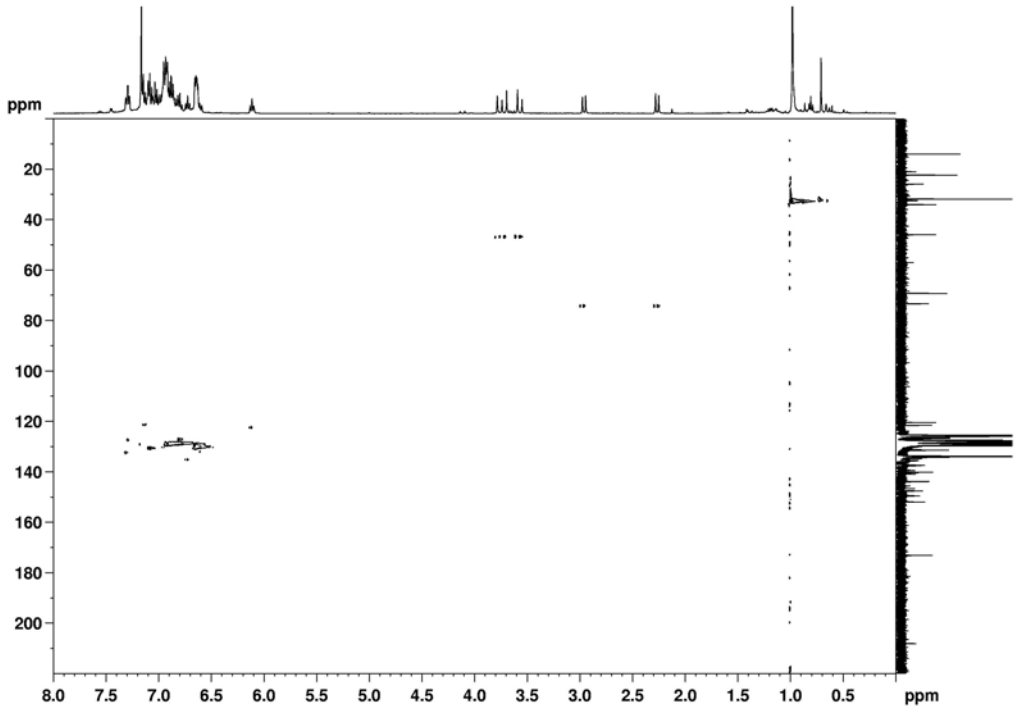
^{1}H-^{13}C HMQC Spectrum for Compound **7**



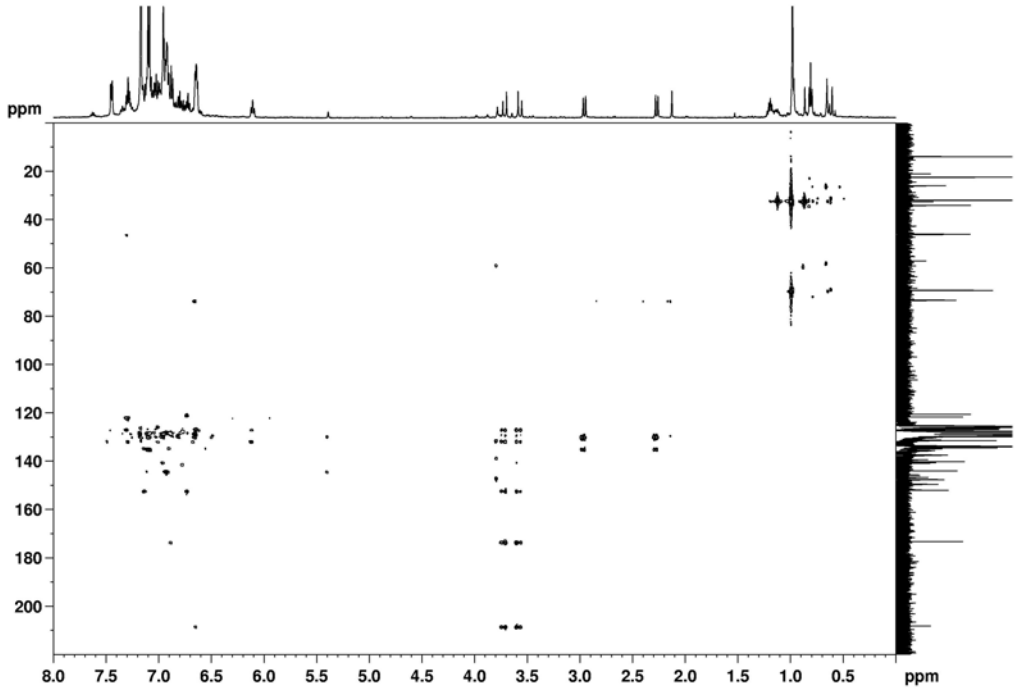
^{1}H-^{13}C HMBC Spectrum for Compound **7**



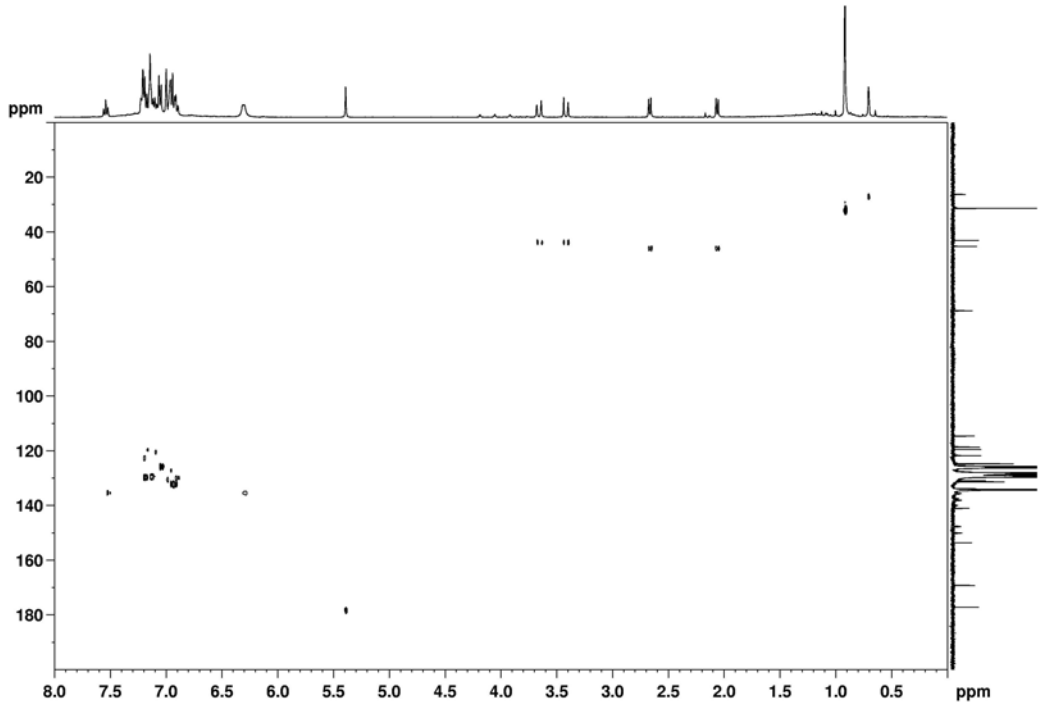
^{1}H-^{13}C HMQC Spectrum for Compound **11**



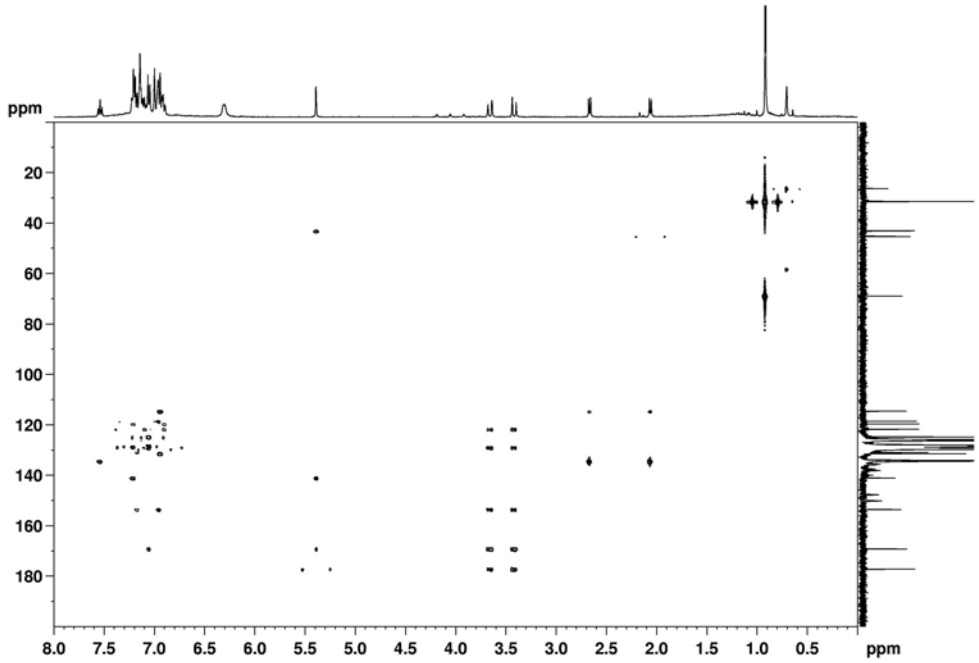
^{1}H-^{13}C HMBC Spectrum for Compound **11**



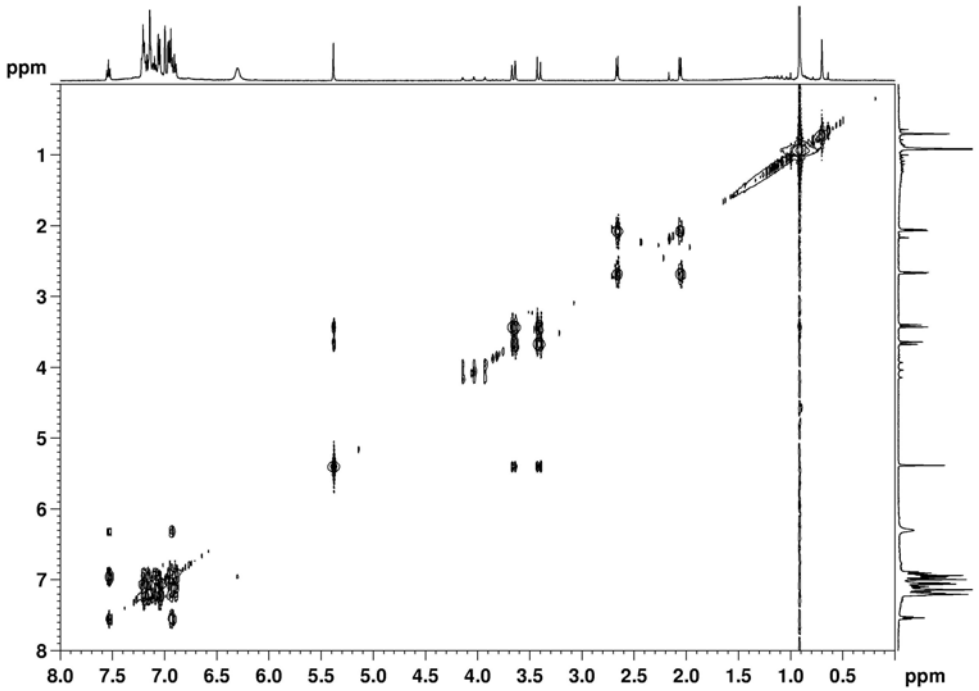
^{1}H-^{13}C HMQC Spectrum for Compound **13**



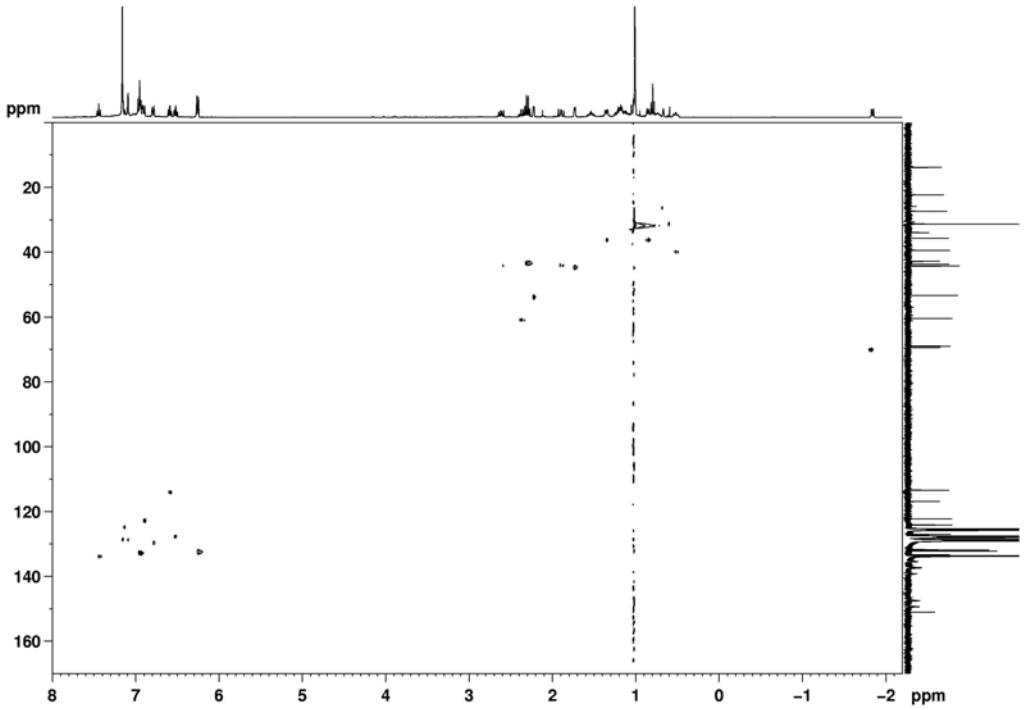
^{1}H-^{13}C HMBC Spectrum for Compound **13**



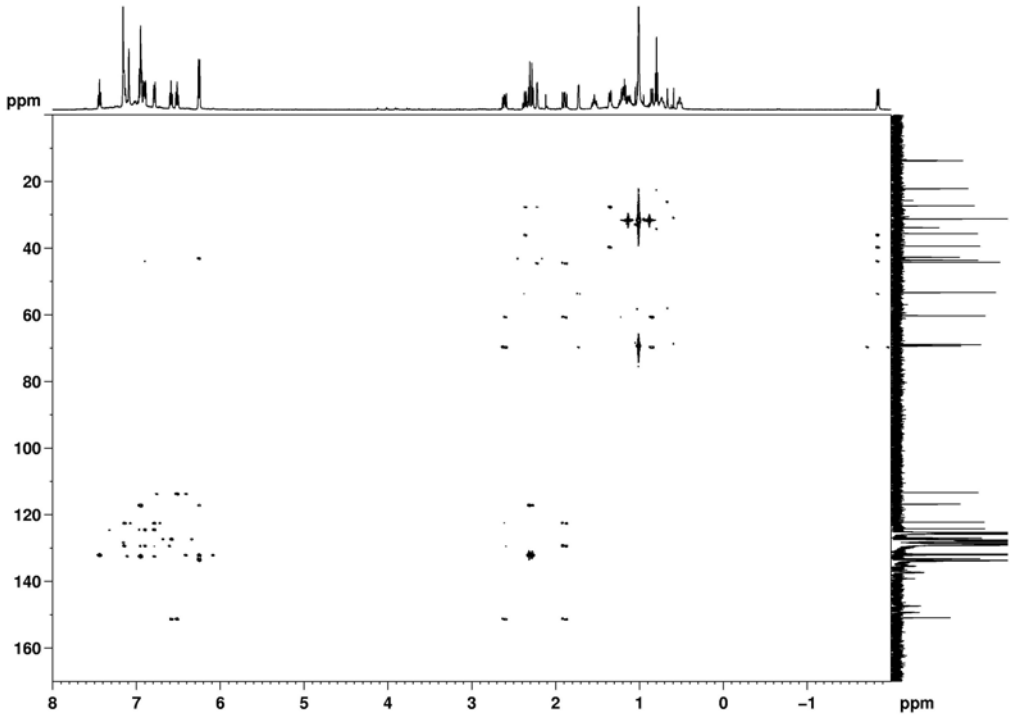
^{1}H-^{1}H TOCSY Spectrum for Compound **13**



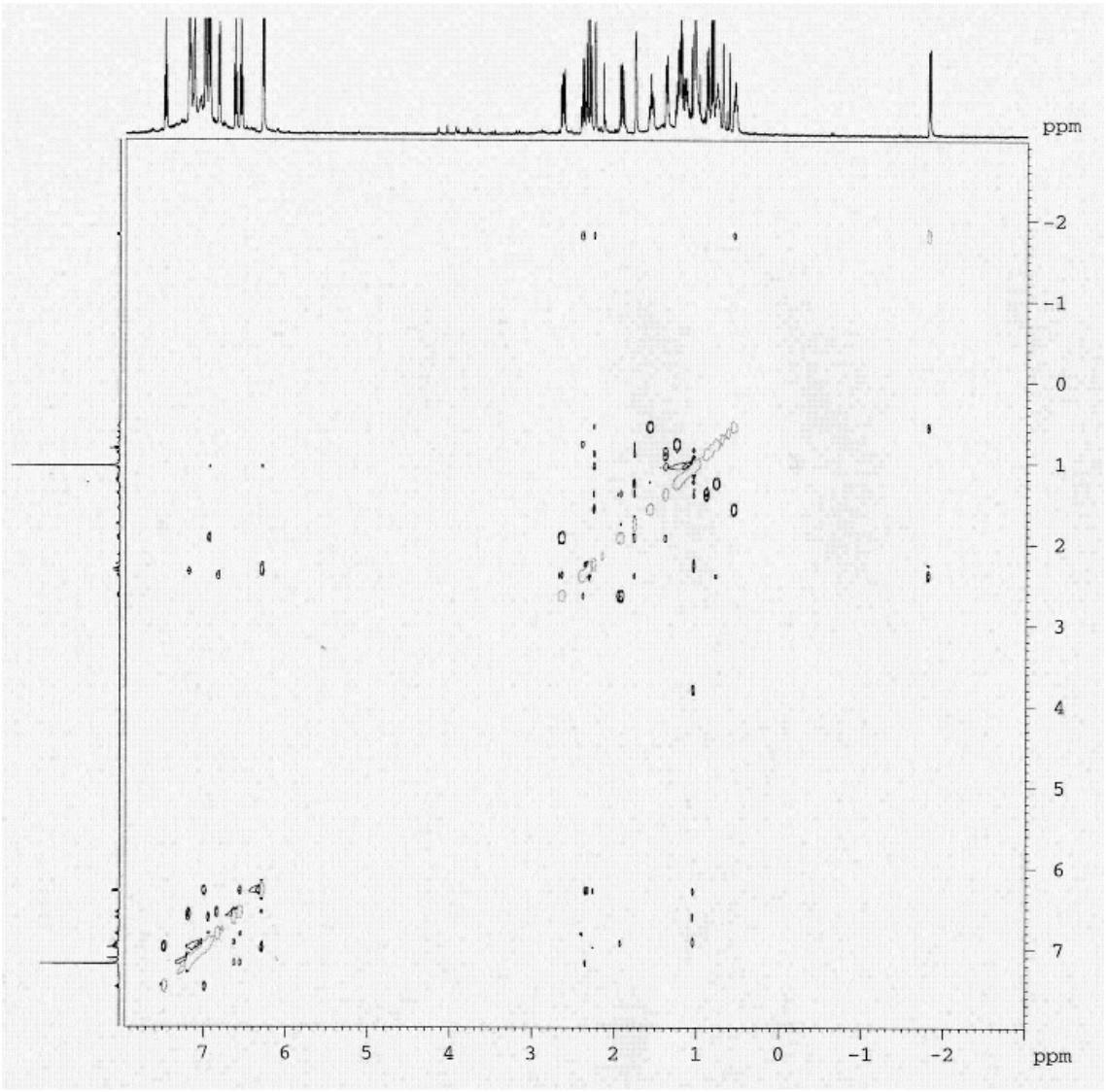
^{1}H-^{13}C HMQC Spectrum for Compound **14**



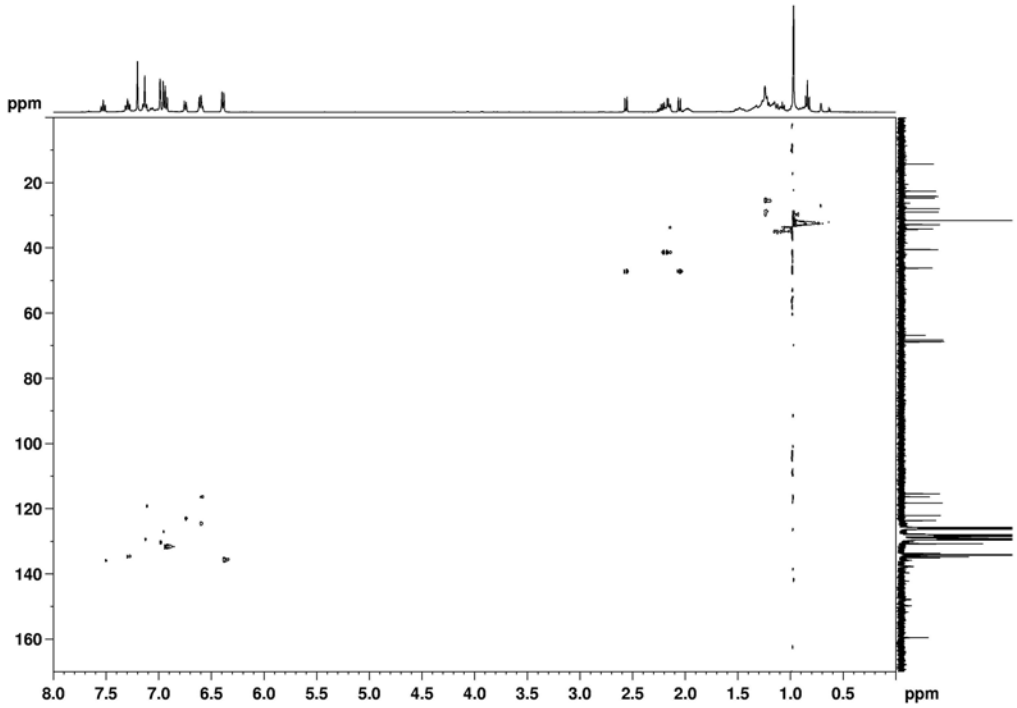
^{1}H-^{13}C HMBC Spectrum for Compound **14**



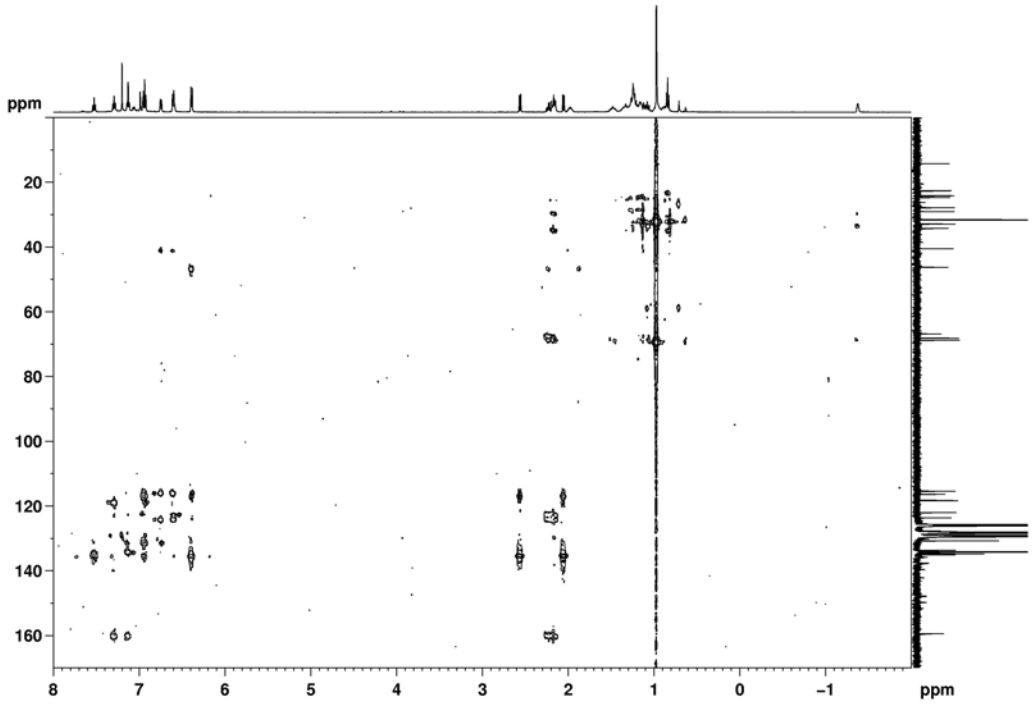
^{1}H-^{1}H NOESY Spectrum for Compound **14**



^{1}H-^{13}C HMQC Spectrum for Compound **16**



^{1}H-^{13}C HMBC Spectrum for Compound **16**



^{1}H NMR Spectrum of Compound **5**.