

**A shortened synthesis of optically pure tricarbonyl(methyl 6-oxo-2,4-hexadienoate)iron leading to improved yield**

Rajesh K. Pandey, Sergey Lindeman and William A. Donaldson\*

*Department of Chemistry, Marquette University, P. O. Box 1881, Milwaukee, WI 53201-1881 USA*

*Supporting Information*

<sup>1</sup> H NMR spectrum of <b>8</b> (CDCl <sub>3</sub> )	S2
<sup>1</sup> H NMR spectrum of <b>9</b> (CDCl <sub>3</sub> )	S3
<sup>1</sup> H NMR spectrum of <b>10</b> (CDCl <sub>3</sub> )	S4
<sup>13</sup> C NMR spectrum of <b>10</b> (CDCl <sub>3</sub> )	S5
<sup>1</sup> H NMR spectrum of <b>11</b> (CDCl <sub>3</sub> )	S6
<sup>13</sup> C NMR spectrum of <b>11</b> (CDCl <sub>3</sub> )	S7
Chart 1. Acyclic (diene)Fe(CO) <sub>3</sub> complexes exhibiting conformer <i>A</i> in the solid state	S8
Chart 2. Acyclic (diene)Fe(CO) <sub>3</sub> complexes exhibiting a non-common conformer	S9
References for Charts 1 and 2	S9-10

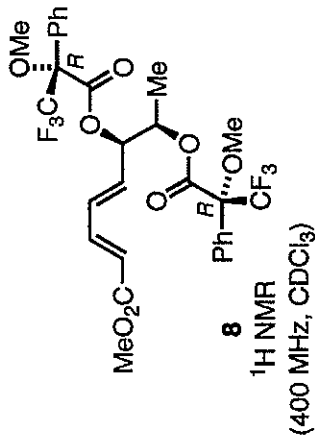
7.475  
7.458  
7.455  
7.417  
7.405  
7.400  
7.391  
7.387  
7.371  
7.270

3.770

3.485  
3.447

1.586

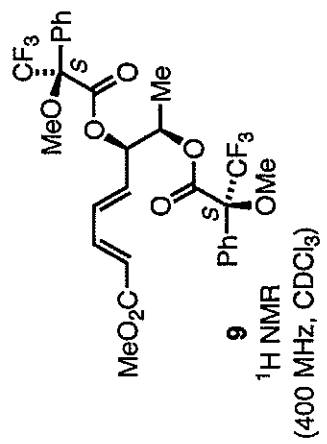
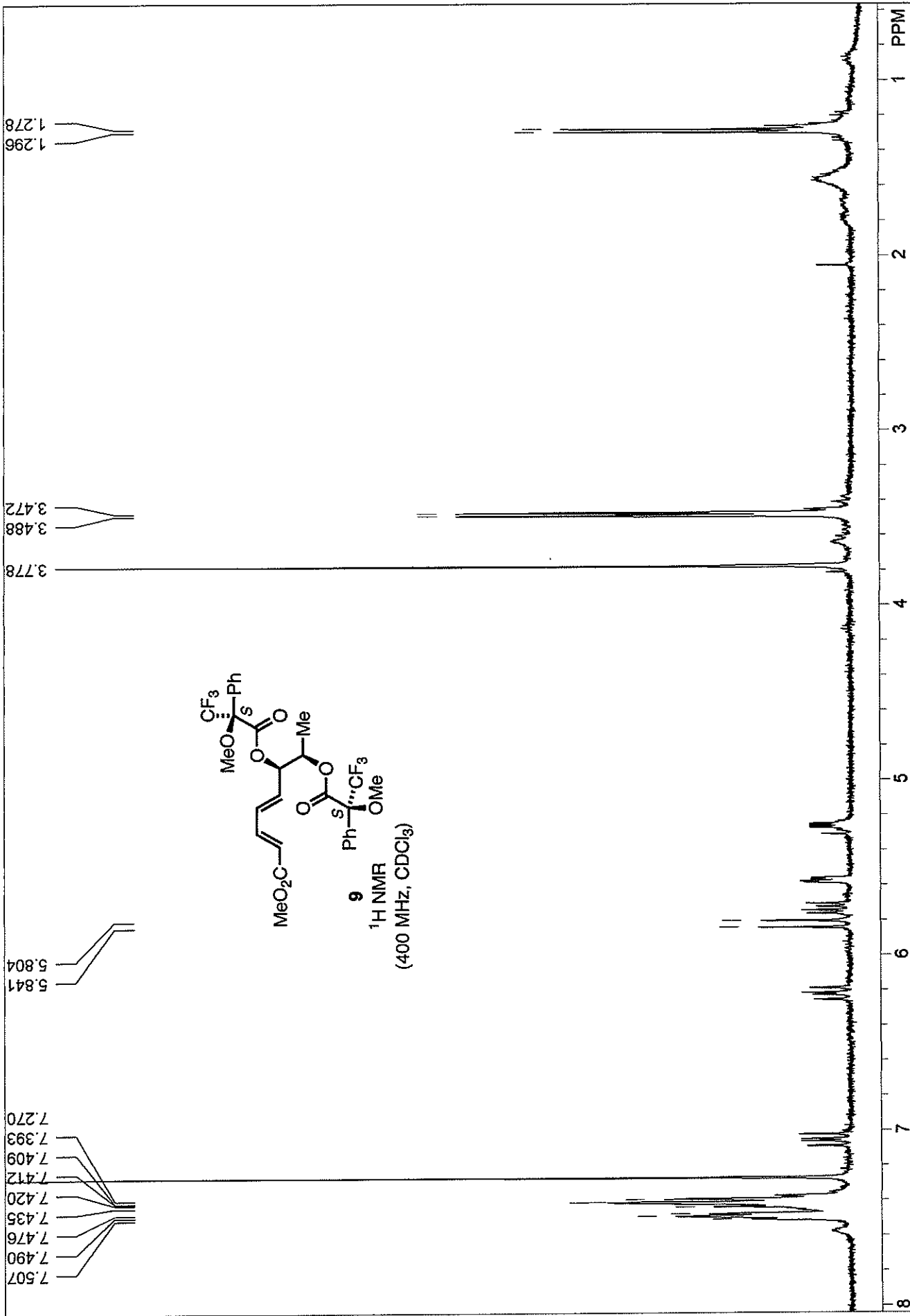
1.290  
1.273



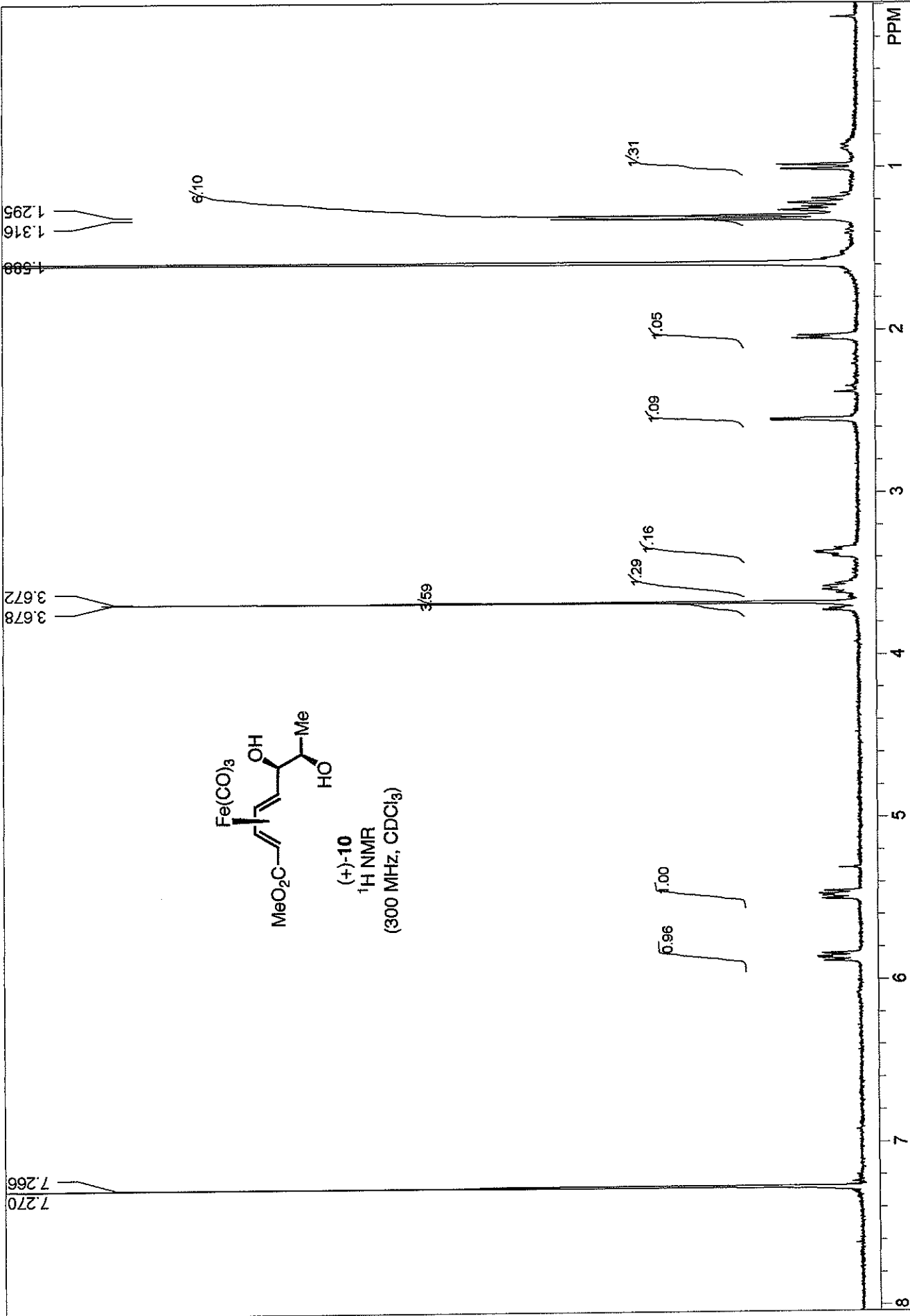
7 6 5 4 3 2 1 PPM

STANDARD 1H OBSERVE - profile  
 F1: 399.752 F2: 100.527  
 EX: s2pul PD: 1.0 sec NA: 8 OF1: 3430.1 LB: 0.0

PTS1d: 13132 . 16384  
 USER: -- DATE: Sep 14 2007  
 Nuts - \$tkp208-HNMR.fid

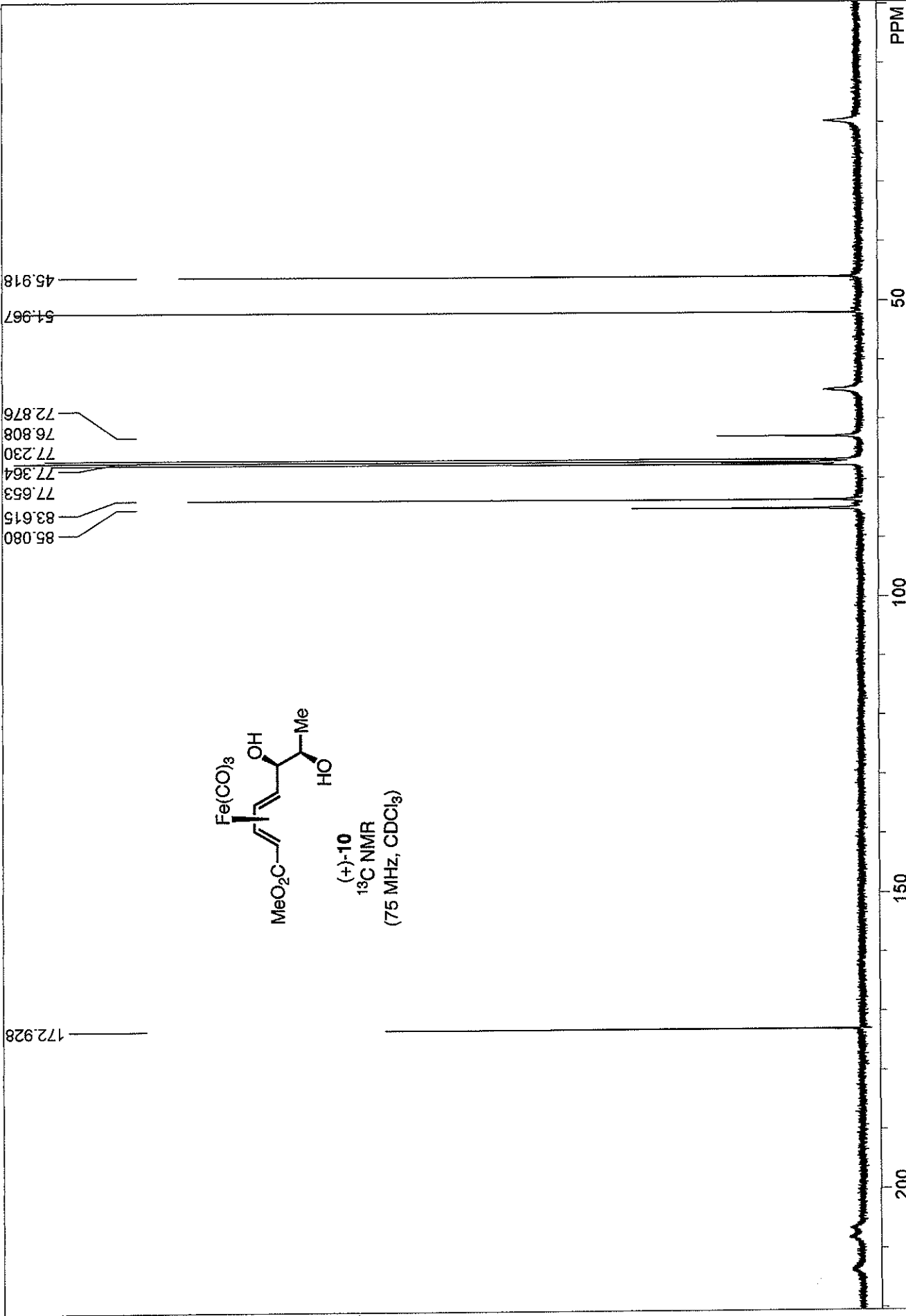


STANDARD 1H OBSERVE - profile		USER: -- DATE: Sep 15 2007	
F1: 399.752	F2: 100.527	OF1: 3430.4	PTSId: 13132_16384
EX: s2pul	SW1: 6410	NA: 8	LB: 0.0
	PW: 7.7 us	PD: 1.0 sec	Nuts - \$tkp209-HNMR.fid

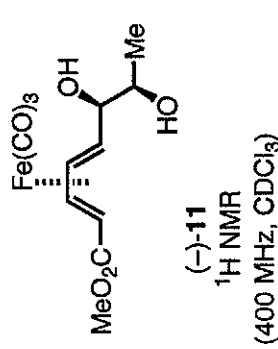
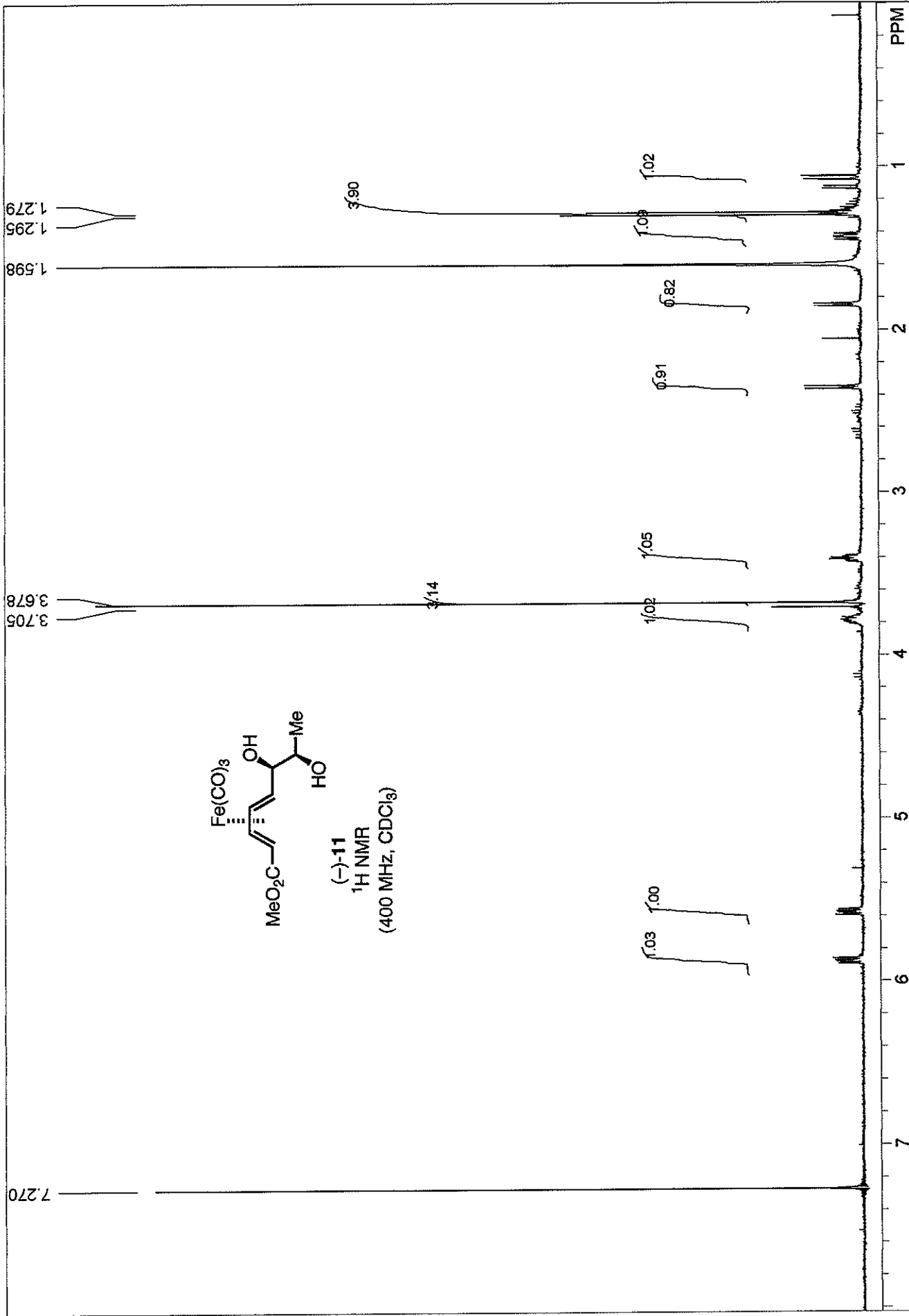


STANDARD 1H OBSERVE: blank line

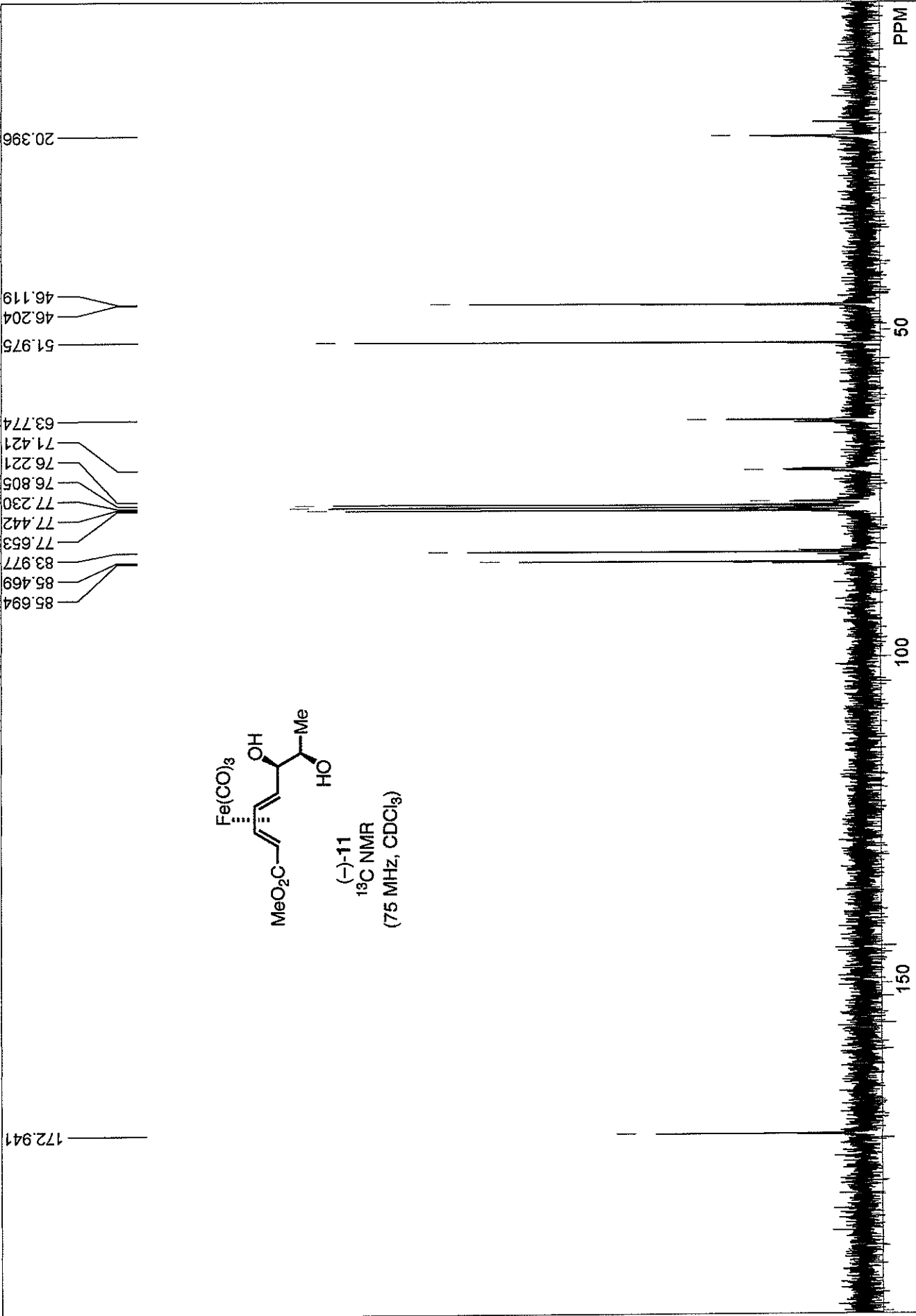
F1: 300.136	F2: 75.476	SW1: 4803	OF1: 1803.8	PTS1d: 9596	16384	USER: -- DATE: Oct. 2 2007
EX: s2pul	PW: 6.3 us	PD: 1.0 sec	NA: 64	LB: 0.0	Nuts - \$rkp207-l-f.fid	



13C OBSERVE: blank line		USER: -- DATE: Jul 18 2007	
F1: 75.477	F2: 300.135	PTSID: 34246	65536
EX: s2pul	PW: 7.3 us	PD: 1.0 sec	NA: 64
		OF1: 8276.0	LB: 0.5



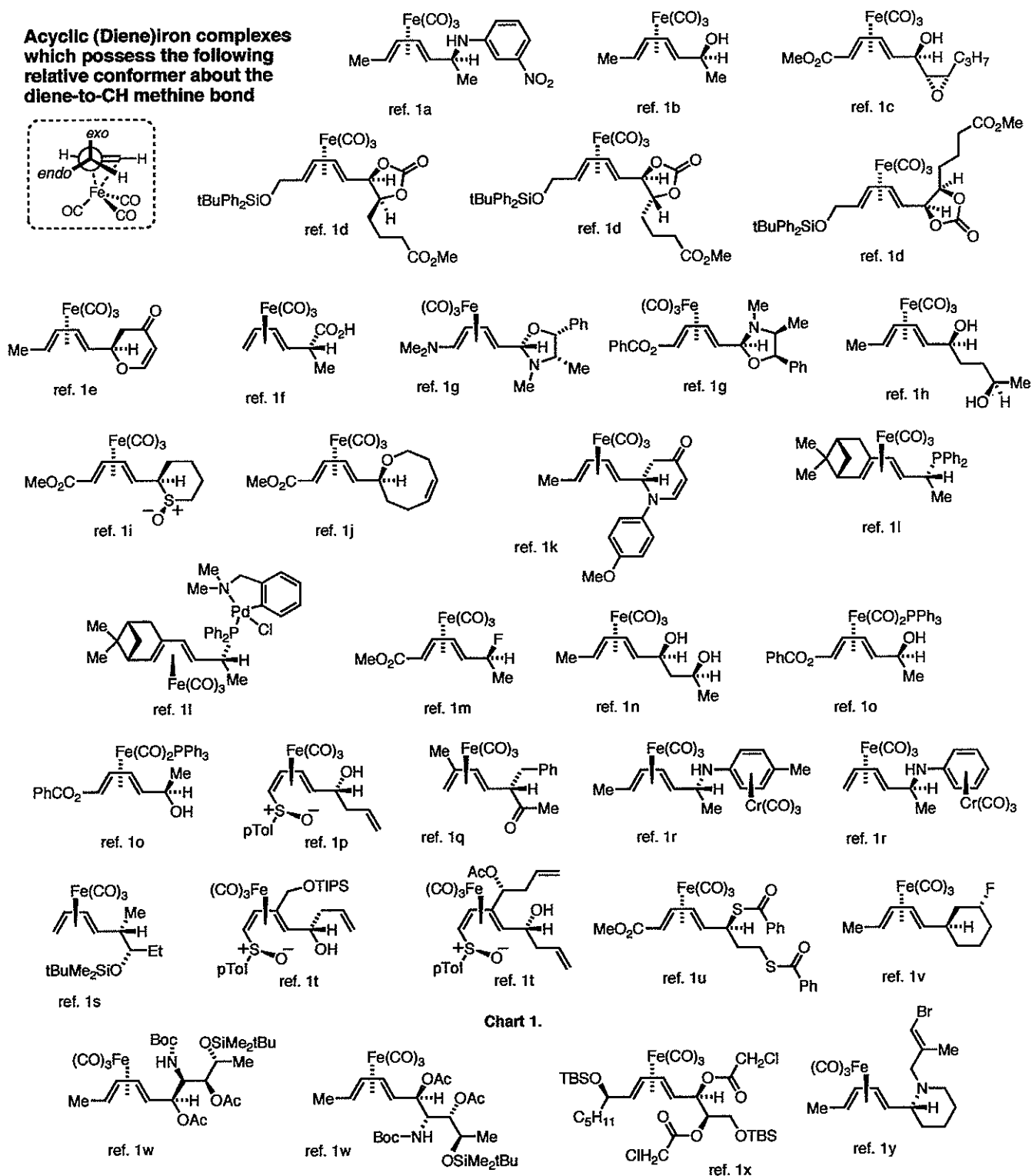
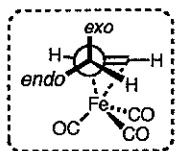
STANDARD 1H OBSERVE - profile		USER: -- DATE: Oct 9 2007	
F1: 399.751	F2: 100.526	PTS1d: 13132	16384
EX: s2pul	PW: 8.0 us	NA: 8	LB: 0.0
	PD: 1.0 sec		Nuts - \$rkp207-1F-HNMR.fid



13C.OBSERVE:blank line

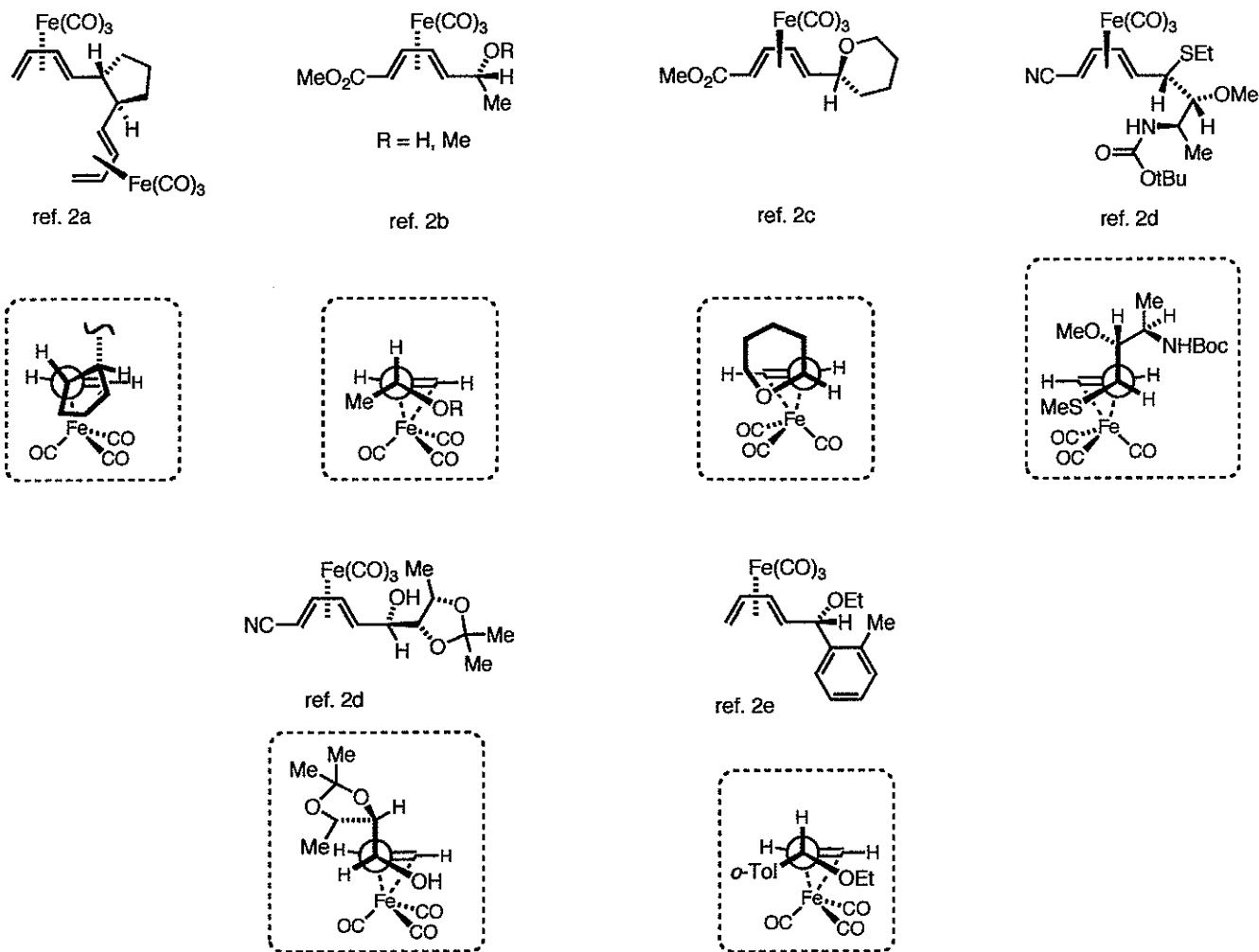
F1: 75.477	F2: 300.135	SW1: 18868	OF1: 8277.7	PTS1d: 34246	USER: -- DATE: Jul 19 2007
EX: s2pul	PW: 7.3 us	PD: 1.0 sec	NA: 400	LB: 1.0	65536
					Nuts - \$rkp186-IF-CNMR.fid

**Acyclic (Diene)iron complexes  
which possess the following  
relative conformer about the  
diene-to-CH methine bond**



**Chart 1.** The complexes depicted above all evidence a crystal structure in which the C–H methine hydrogen is pointed toward a basal Fe–CO ligand.





**Chart 2.** The complexes depicted above evidence a crystal structure with a less common conformer about the (diene)iron-to-CH methine bond.

## References

- 1) (a) Immirzi, A. *J. Organomet. Chem.* **1974**, *76*, 65-71.
- (b) Riley, P. E.; Davis, R. E. *Acta Cryst. B* **1976**, *B32*, 381-386.
- (c) Lellouche, J. P.; Breton, P.; Beaucourt, J. P.; Toupet, L.; Gree, R. *Tetrahedron Lett.* **1988**, *29*, 2449-2452.
- (d) Toupet, L.; Gree, R.; Gigou-Barbedette, A.; Lellouche, J.-P.; Beaucourt, J.-P. *Acta Cryst. C* **1991**, *C47*, 1173-1177.
- (e) Donaldson, W. A.; Tao, C.; Bennett, D. W.; Grubisha, D. S. *J. Org. Chem.* **1991**, *56*, 4563-4566.
- (f) Donaldson, W. A.; Craig, R.; Spanton, S. *Tetrahedron Lett.* **1992**, *33*, 3967-3968.
- (g) Howell, J. A. S.; Bell, A. G.; O'Leary, P. J.; McArdle, P.; Cunningham, D.; Stephenson, G. R.; Hastings, M. *Organometallics* **1994**, *13*, 1806-1812.
- (h) Donaldson, W. A.; Bell, P. T.; Wang, Z.; Bennett, D. W. *Tetrahedron Lett.* **1994**, *35*, 5829-5832.
- (i) Hachem, A.; Toupet, L.; Gree, R. *Tetrahedron Lett.* **1995**, *36*, 1849-1852.

- (j) Gree, D.; Martelli, J. T.; Gree, R. L. *J. Org. Chem.* **1995**, *60*, 2316-2317.
- (k) Takemoto, Y.; Ueda, S.; Takeuchi, J.; Nakamoto, T.; Iwata, C.; Ohishi, H.; Sakaguchi, K.; Kusunoki, M. *Chem. Pharm. Bull.* **1995**, *43*, 559-563.
- (l) Englert, U.; Ganter, B.; Kaser, M.; Klinkhammer, E.; Wagner, T.; Salzer, A. *Chem. Eur. J.* **1996**, *2*, 143-148.
- (m) Gree, D.; Kermarrec, C. J. M.; Martelli, J. T.; Gree, R. L.; Lellouche, J.-P.; Toupet, L. J. *J. Org. Chem.* **1996**, *61*, 1918-1919.
- (n) Young, V. G., Jr.; Dasgupta, B.; Donaldson, W. A. *J. Chem. Cryst.* **1996**, *26*, 567-569.
- (o) Howell, J. A. S.; Bell, A. G.; O'Leary, P. J.; Stephenson, G. R.; Hastings, M.; Howard, P. W.; Owen, D. A.; Whitehead, A. J.; McArdle, P.; Cunningham, D. *Organometallics* **1996**, *15*, 4247-4257.
- (p) Paley, R. S.; Rubio, M. B.; de la Pradilla, R. F.; Dorado, R.; Hundal, G.; Martinez-Ripoll, M. *Organometallics* **1996**, *15*, 4672-4674.
- (q) Bohmer, J.; Hampel, F.; Schobert, R. *Synthesis* **1997**, 661-667.
- (r) Bentele, H.-J.; Sunkel, K.; Beck, W. *J. Organomet. Chem.* **1997**, *549*, 245-250.
- (s) Wasicak, J. T.; Craig, R. A.; Henry, R.; Dasgupta, B.; Li, H.; Donaldson, W. A. *Tetrahedron* **1997**, *53*, 4185-4198.
- (t) Paley, R. S.; Estroff, L. A.; McCulley, D. J.; Martinez-Cruz, L. A.; Sanchez, A. J.; Cano, F. H. *Organometallics* **1998**, *17*, 1841-1849.
- (u) Crevisy, C.; Herbage, B.; Marrel, M.-L.; Toupet, L.; Gree, R. *Eur. J. Org. Chem.* **1998**, 1949-1954.
- (v) Pearson, A. J.; Alimardanov, A.; Pinkerton, A. A.; Fouchard, D. M.; Kirschbaum, K. *Tetrahedron Lett.* **1998**, *39*, 5919-5922.
- (w) Franck-Neumann, M.; Miesch-Gross, L.; Gateau, C. *Eur. J. Org. Chem.* **2000**, 3693-3702.
- (x) Baba, Y.; Saha, G.; Nakao, S.; Iwata, C.; Tanaka, T.; Ibuka, T.; Ohishi, H.; Takemoto, Y. *J. Org. Chem.* **2001**, *66*, 81-88.
- (y) Williams, I.; Reeves, K.; Kariuki, B. M.; Cox, L. R. *Org. Biomol. Chem.* **2007**, *5*, 3325-3329.
- 2) (a) Sapienza, R. S.; Riley, P. E.; Davis, R. E.; Pettit, R. *J. Organomet. Chem.* **1976**, *121*, C35-C40.
- (b) Messenger, P. J. C.; Toupet, L. *Acta Cryst. B* **1986**, *B42*, 371-378.
- (c) Teniou, A.; Toupet, L.; Gree, R. *Synlett* **1991**, 195-197.
- (d) Takemoto, Y.; Yoshikawa, N.; Baba, Y.; Iwata, C.; Tanaka, T.; Ibuka, T.; Ohishi, H. *J. Am. Chem. Soc.* **1999**, *121*, 9143-9154.
- (e) Takemoto, Y.; Ishii, K.; Ibuka, T.; Miwa, Y.; Taga, T.; Nakao, S.; Tanaka, T.; Ohishi, H.; Kai, Y.; Kanehisa, N. *J. Org. Chem.* **2001**, *66*, 6116-6123.
- (f) Xiao, N.; Wang, B.; Yin, J.; Xu, Q.; Tsumori, N.; Sun, J.; Chen, J. *Organometallics* **2004**, *23*, 257-268.