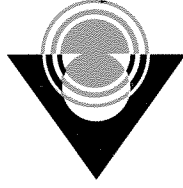


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THE SCIENTIFIC AND TECHNOLOGICAL RESEARCH COUNCIL OF TURKEY

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Paladyum ve Rodyum Katalizli Tandem Karbonilatif Kenetlenme Tepkimeleri

Proje No: 106T385

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İZMİR

Önsöz

Bu çalışma Türkiye Bilimsel ve Teknik Araştırma Kurumu (TÜBİTAK) tarafından 106T385 proje numarası ile desteklenmiştir.

Çalışmada sentezlenen ürünlerin NMR analizleri Uzman sayın Işın Özçelik tarafından gerçekleştirilmiştir.

Çalışmada sentezlenen ürünlerin Yüksek Çözünürlükte Kütle Spektrumları (HRMS) Dortmund Teknik Üniversitesi' nde (Almanya) gerçekleştirilmiştir.

AM1 hesaplamaları sn. Prof. Dr. Metin Balcı tarafından yapılmıştır.

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Özet

Bu proje çalışmasında alkinler organoboron bileşikleri ile CO atmosferinde Rh katalizli tepkimeleri gerçekleştirilmiştir. Tepkimeler başlıca 5-aril-2(5H)-furanon (butenolitler), α - β -doymamış keton ve indanon bileşiklerini oluşturmuştur. Tepkime koşullarının dikkatlice seçilmesi ürün seçiciliğini etkilemektedir. Böylece tepkime koşullarının değiştirilmesi ile istenen ürün tipine tepkimeyi yönleltmek mümkün olabilmektedir.

5-aril-2(5H)-furanon oluşumu toluen gibi aprotik çözücü ortamı ve 10 atm üzeri basınçlar gerektirmektedir. Enon oluşumları için ideal CO basıncı 5 atm olarak saptanmıştır. Tepkime metanol gibi protik çözücü içinde etkin olabilmekte ve ayrıca alkinin aroil rodasyon adımından sonra oluşan β -aroil alkenilrodyum(I) kompleksinin demetalasyonu için bir proton kaynağına (CF_3COOH) gerek duymaktadır. İç alkinlerin hidroasilasyonu *sin*- modunda gerçekleşerek diaril asetenler hariç E- izomerleri oluşturmuşlardır. Diaril alkinler ile her iki E- ve Z- enonlar elde edilmiştir.

İndanon seçiciliği için CO basıncının daha düşük basınçlarda, tipik olarak 1 atm de uygulanmasını gerektirmiştir. Tepkime yine metanol içerisinde ve diğer tepkimelere kıyasla daha yüksek sıcaklıklarda (120 °C, diğer tepkimelerde 80 °C) gerçekleşmiştir.

Anahtar kelimeler: Karbonilasyon, alkin, organoboron, rodyum, enon, furanon, indanon

Abstract

Within the context of this project, rhodium catalyzed reactions of alkynes and organoboron reagents were examined under a CO atmosphere. The carbonylative coupling reactions produced mainly 5-aryl-2(5*H*)-furanones (butenolides), α,β -unsaturated ketones, and indanones. The methodology was product tunable; the product selectivity of the process is switchable by carefully adjusting experimental parameters.

Application of a CO pressure of >10 atm, and the use of an aprotic solvent, e.g. toluene, were necessary for the efficient synthesis of 5-aryl-2(5*H*)-furanones. Optimum CO pressure was determined to be 5 atm for efficient syntheses of enones. The reaction was more efficient in a protic solvent, e.g. methanol and in addition, the presence of an acid additive (CF₃COOH) is required for effective demetalation of β -aryl alkenylrhodium(I) intermediate, which is produced upon aryl rhodation of an alkyne. Hydroacylation of internal alkynes, except in the case of diaryl acetylenes, proceeded in *syn*-fashion, yielding the *E*-configured isomer.

A mixture of *E*- and *Z*-isomers was obtained with diaryl acetylenes. Reactions were more regioselective for nonsymmetric alkynes for the formation of enones as compared to the reactions which led to the furanones as main products.

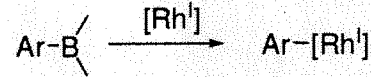
A relatively lower CO pressure, typically 1 atm, and a higher reaction temperature, typically 120 °C, were necessary conditions for higher selectivity of indanone product. Methanol was the choice of solvent and the reaction was more efficient with the use of boronic acid ester than using boronic acid counterpart.

Keywords: Carbonylation, alkyne, organoboron, rhodium, enone, furanone, indanone

1. Giriş

Çeşitli doymamış hidrokarbonların geçiş metal katalizli karbonilasyonu heterosiklik bileşiklerin sentezinde kullanılan yaygın bir metoddur (Vizer ve diğ., 2004).

Hayashi ve Miyaura tarafından (Sakai ve diğ., 1997) α,β -doymamış ketonlara arilboronik ve alkenilboronik asitlerin rodyum katalizli olarak ilk defa gerçekleştirilmesinden bu yana organoboron reaktiflerinin çeşitli doymamış sistemlere rodyum katalizli katılma tepkimeleri karbon-karbon bağı oluşturan bir metod olarak yaygın olarak kullanılmaktadır. Organoboron bileşikleri rodyum ile kolaylıkla transmetallasyona uğrayarak arilrodyum(I) yapıları oluştururlar (Fagnau ve Lautens, 2003; Hayashi ve Yamasaki, 2003; Sakai ve diğ., 1998; Oguma ve diğ., 2000; Frost ve Wadsworth, 2001; Lautens ve diğ., 2001; Hayashi ve diğ., 2001; Oguma ve diğ., 2002; Lautens ve diğ., 2002; Matsuda ve diğ., 2004; Miura ve diğ., 2005; Ueura ve diğ., 2005; Shintani ve diğ., 2005a; 2005b):



Bu yapılar elektrofilik grupların nükleofilik arilasyonunu sağlayabilirler. İlk defa Sauthier ve grubu tarafından terminal α,β -doymamış ketonların arilboronik asitler ile CO atmosferinde rodyum katalizli ortamda 1,4-diketonların oluşumunu gerçekleştirmişlerdir (Sauthier, 2004).

Bu çalışmada iç alkinlerin organoboron bileşikleri ile CO gazı altında rodyum katalizli karbonilatif arilasyon tepkimelerini incelemektedir. Metod başlıca furanon, α,β -doymamış keton ve inda(e)non ürünleri oluşturmaktadır. Tepkime koşullarının dikkatlice değiştirilmesi farklı ürünlerin seçimli olarak sentezlenmesini mümkün kılmıştır.

2. Gereç ve Yöntem

2.1. Genel

Tepkimelerde kullanılan alkinler ya ticari kaynaklardan temin edilmiş ya da laboratuvarında aşağıda belirtilen metodlara göre sentezlenmiştir. Diğerleri ticari olarak mevcuttur. Tepkimelerde kullanılan dioksan sodyum-ketil üzerinden, toluen CaH_2 ile ve metanol Mg şeriti üzerinden kurutulmuş ve destile edilmişlerdir. Metanol moleküler elek 3A ve toluen moleküler elek 4A üzerinde saklanmışlardır. $[\text{Rh}(\text{cod})\text{Cl}]_2$ (Giordano ve Crabtree, 1979), $[\text{Rh}(\text{cod})\text{OH}]_2$ (Uson ve diğ., 1985) ve $[\text{Rh}(\text{cod})\text{OCH}_3]_2$ (Uson ve diğ., 1985) bileşikleri

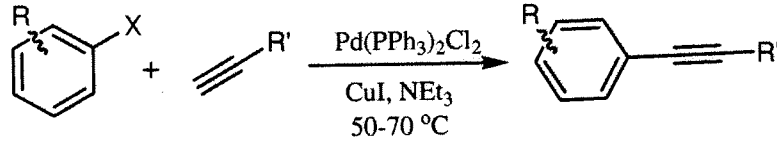
laboratuvarda sentezlenmiştir. $[\text{Rh}(\text{C}_2\text{H}_4)_2\text{Cl}]_2$ bileşiği her iki ticari ve laboratuvarda sentezlenmiş formunda (Cramer ve diğ., 1974) kullanılmıştır. Diğer rodyum kompleksleri ticari kaynaklardan sağlanmıştır. Ürünler Gaz kromatografi (GK, HP 6890N), GC/MS (GC-Varian star 3400CX, MS-Varian Saturn 2000), NMR (Varian Vnmrj 400), FTIR (Perkin Elmer Spectrum 100), elementel analiz (LECO) ve HRMS teknikleri ile analiz edilmişlerdir.

2.2. Alkinlerin Sentezi

Alkinler Sonagashira tepkimesi ile iki ayrı metod ile sentezlenmiştir.

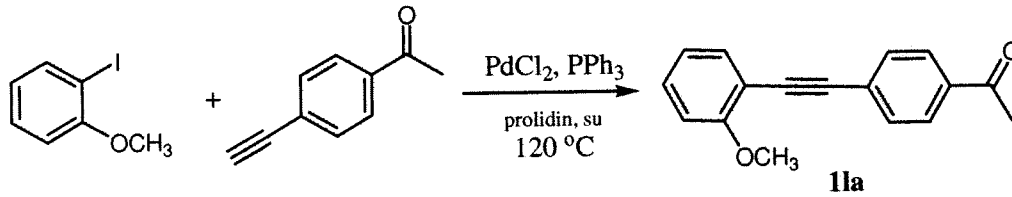
2.2.1. Metod I

Argon altındaki bir cam balonda aril halojenür (bromo ya da iyodo, 10 mmol) ve terminal alkinin (12 mmol) 40 mL Et_3N içerisindeki çözeltisine aril halojenür miktarına göre % 2 mol $\text{Pd}(\text{PPh}_3)_2\text{Cl}_2$ eklendi. Çözelti 5 dakika karıştırıldıktan sonra %1 mol oranında CuI eklendi. Reaksiyon kabı $50\text{ }^\circ\text{C}$ sıcaklığa ısıtılmış bir yağ banyosuna daldırıldı ve manyetik olarak tepkime süresince karıştırıldı. Tepkime süreci gaz kromatografik (GK) analiz ile takip edildi ve alkin tamamen tükenene kadar devam edildi. Bu metod ile **1e-k** alkinler sentezlenmiştir (Roesch ve Larock, 2001).



2.2.2. Metod II

İki boyunlu balon içerisindeki 1-(4-etinilfenil)ethanon (1.2 eşd.), 2-iyodoanisol (1 eşd.) prolidin (2 eşd.), PdCl_2 (%2 mol), PPh_3 (%4 mmol) ve degaze edilmiş 2 mL su /mol aril iyodür karışımı Ar gazı altında $120\text{ }^\circ\text{C}$ sıcaklığa ısıtılmış bir yağ banyosuna daldırıldı ve tepkime süresince karıştırıldı. Tepkime GK analizi ile alkin sübstrat tamamen tükeninceye kadar sürdürüldü. Tepkime sonunda karışım eter ile ekstrakte edildi ve ürün kolon kromatografisi ile silika üzerinden saflaştırıldı (Guan ve diğ., 2007).



2.3. Karbonilasyon Tepkimeleri

2.3.1. Furanon ürünlerin genel sentez yöntemi

Tepkimeler 50 ml hacminde payreks cam içeren bir paslanmaz çelik içerisinde gerçekleştirilmiştir. Arilboronik asit (1.2 mmol), alkin (1 mmol), heksadekan (internal standart, 0.56 mmol), $[\text{Rh}(\text{cod})\text{OH}]_2$ (%1-3 mol, alkin miktarına oranla), 10 mL toluen (degaze, kuru) ve bir magnet reaktöre eklendi. Kapatılan reaktör önce vakumlanarak ve iki defa 10 atm CO verilerek hava içeriği giderildi. Reaktöre 20 atm basınçta CO basıldıktan sonra 80 °C tepkime sıcaklığına ısıtılmış bir yağ banyosuna daldırıldı ve tepkime süresince magnetik olarak karıştırıldı. Tepkime sonunda gazı boşaltılan reaktör açıldı, etilasetat ile yıkandı ve GK ve GK/MS analizleri gerçekleştirildi. Seçilmiş örnekler kolon kromatografisi ile saflaştırıldıktan sonra NMR yöntemi ile analiz edildiler.

2.3.2. Enonların Sentezinde Tipik Sentez Koşulları

Degaze edilmiş kuru metanol (9.9 mL) içerisinde alkin (1.5 mmol), arilboronik asit (3 mmol), $[\text{Rh}(\text{C}_2\text{H}_4)_2\text{Cl}]_2$ (%3 mmol Rh), trifloroasetik asit (TFA, 2 mmol), H_2O (0.1 mL) karışımı 50 mL paslanmaz çelik otoklav içerisindeki payreks kaba eklendi. Magnet eklenen ve kapatılan reaktör önce vakumlanarak ve iki defa 10 atm CO verilerek hava içeriği giderilmiştir. Otoklav 5 atm CO basıldıktan sonra ısıtılmış bir yağ banyosu içerisinde magnetik olarak karıştırıldı. Tepkime sonunda soğutulduktan sonra tepkime karışımı etilasetat ile yıkandı. Ürünler GK, GK/MS ile analiz edildi ve kolon kromatografisi ile izole edildi. İzole ürünlerin yapıları NMR analizleri ile aydınlatıldı.

(E,Z)-1,2,3-Trifenilprop-2-en-1-on (3aa): Hekzan/etil asetat; **(E-3aa):** beyaz katı; E.N. (°C): 98.7-99.2 (lit.: 99-101 (Duke ve Boykin, 1972); 103-103.5 (Black ve Lutz, 1953); 96.5-97.5 (Mittal ve diğ., 1985)); ^1H NMR (400 MHz, CDCl_3) δ : 7.07-7.56 (m, 14H), 7.86 (d, $J = 7.2$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ : 126.4, 128.0, 128.2, 128.4, 128.7, 128.8, 128.9, 129.7, 130.1, 133.6, 135.4, 136.3, 138.0, 140.8, 199.4; MS (EI, m/z): 284 (93, M^+), 256 (5), 206 (9), 178 (31), 105 (100), 77 (20); FTIR (ATR) ν (cm^{-1}) CO 1649 (lit.: (Duke ve Boykin, 1972); 1640 (Mittal ve diğ., 1985)); HRMS (m/z , M^+): 284.1196 (hesaplanan), 284.1189 (saptanan); **(Z-3aa):** sarı katı; E.N. (°C): 78.0-84.1 (lit.: 85-87 (Duke ve Boykin, 1972); 88-89 (Black ve Lutz, 1953); 82.5-83.5 (Mittal ve diğ., 1985)); ^1H NMR (400 MHz, CDCl_3) δ : 7.10-7.50 (m, 14H), 7.99 (d, $J = 6.8$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ : 127.9, 128.2, 128.3, 128.8, 128.9, 129.7, 129.8, 130.3, 132.2, 134.8, 136.5, 138.2, 140.1, 140.8, 197.6; MS (EI, m/z): 284 (87, M^+), 256 (7), 206 (20), 178 (47), 105 (100), 77 (83); FTIR (ATR) ν (cm^{-1}) CO:

1660 (lit.: 1671 (Duke ve Boykin, 1972); 1661 (Mittal ve diğ., 1985)); HRMS (m/z , M^+): 284.1196 (hesaplanan), 284.1197 (saptanan).

(E,Z)-1-(4-Metoksifenil)-2,3-difenilprop-2-en-1-on (3ab): Hekzan/benzen; hekzan/diklorometan; **(E-3ab):** beyaz katı; E.N. (°C): 81.1-84.3 (lit.: 84-84.3 (Mittal ve diğ., 1985)); ^1H NMR (400 MHz, CDCl_3) δ : 3.85 (s, 3H), 6.92 (d, $J = 8.8$ Hz, 2H), 7.09-7.36 (m, 11H), 7.90 (d, $J = 8.8$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ : 55.5, 113.6, 127.9, 128.2, 128.6, 128.8, 129.6, 130.2, 130.4, 132.3, 135.0, 136.8, 137.8, 141.0, 163.1, 196.3; MS (EI, m/z): 314 (47, M^+), 197 (14), 178 (14), 135 (100), 107 (9), 92 (10), 77 (19); FTIR (ATR) ν (cm^{-1}) CO: 1633 (lit.: 1640 (Mittal ve diğ., 1985)); HRMS (m/z , M^+): 314.1301 (hesaplanan), 314.1294 (saptanan); **(Z-3ab):** açık sarı katı; E.N. (°C): 88.7-89.2 (lit.: 78-79 (Mittal ve diğ., 1985)); ^1H NMR (400 MHz, CDCl_3) δ : 3.79 (s, 3H), 6.83 (d, $J = 8.8$ Hz, 2H), 7.12-7.48 (m, 11H), 7.97 (d, $J = 8.8$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ : 55.4, 114.0, 126.3, 127.9, 128.1, 128.4, 128.8, 129.4, 129.5, 132.1, 135.5, 138.2, 140.9, 164.0, 197.9; MS (EI, m/z): 314 (58, M^+), 197 (18), 178 (19), 135 (100), 107 (12), 92 (13), 77 (23); FTIR (ATR) ν (cm^{-1}) CO: 1657 (lit.: 1655 (Mittal ve diğ., 1985)). HRMS (m/z , M^+): 314.1301 (saptanan), 314.1301 (hesaplanan).

(E,Z)-2,3-Difenil-1-p-tolilprop-2-en-1-on (3ac): Hekzan/benzen; **(E-3ac):** beyaz katı; E.N. (°C): 90.8-91.8; ^1H NMR (400 MHz, CDCl_3) δ : 2.41 (s, 3H), 7.08-7.39 (m, 13H), 7.79 (d, $J = 8.0$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ : 21.6, 126.3, 127.9, 128.2, 128.8, 128.8, 129.0, 129.6, 130.1, 130.3, 134.9, 135.3, 136.7, 139.1, 140.9, 143.0, 197.5; MS (EI, m/z): 298 (75, M^+), 283 (15), 205 (6), 181 (23), 178 (25), 119 (100), 91 (48); FTIR (ATR) ν (cm^{-1}) CO: 1643; HRMS (m/z , M^+): 298.1352 (hesaplanan), 298.1346 (saptanan); **(Z-3ac):** açık sarı katı; E.N. (°C): 59.8-61.8; ^1H NMR (400 MHz, CDCl_3) δ : 2.32 (s, 3H), 7.10-7.47 (m, 13H), 7.89 (d, $J = 8.4$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ : 21.7, 126.3, 128.0, 128.1, 128.5, 128.8, 129.5, 129.7, 129.9, 134.0, 135.5, 138.1, 141.0, 144.7, 199.0; MS (EI, m/z): 298 (100, M^+), 283 (12), 182 (20), 119 (55), 91 (11); FTIR (ATR) ν (cm^{-1}) CO: 1654; HRMS (m/z , M^+): 298.1352 (hesaplanan), 298.1341 (saptanan).

(E,Z)-2,3-Difenil-1-m-tolilprop-2-en-1-on (3ad): Hekzan/benzen; **(E-3ad):** hekzan/etil asetat ile tekrar kolon kromatografi ile saflaştırıldı; beyaz katı; E.N. (°C): 66.3-68.7; ^1H NMR (400 MHz, CDCl_3) δ : 2.40 (s, 3H), 7.06-7.38 (m, 13H); 7.64 (d, $J = 7.6$ Hz, 1H), 7.68 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ : 21.4, 127.06, 127.9, 128.08, 128.2, 128.8, 128.9, 129.7, 130.2, 130.3, 132.9, 134.9, 136.6, 138.2, 138.2, 140.0, 140.9, 197.9; MS (EI, m/z): 298 (75,

M⁺); 283 (10), 181 (23), 178 (25), 119 (100), 91 (48); FTIR (ATR) ν (cm⁻¹) CO: 1635; HRMS (*m/z*, M⁺): 298.1352 (hesaplanan), 298.1351 (saptanan); **Z-3ad**: sarı yağimsı; ¹H NMR (400 MHz, CDCl₃) δ : 2.32 (s, 3H), 7.10-7.48 (m, 13H), 7.65 (d, *J* = 8 Hz, 1H), 7.83 (s, 1H); ¹³C NMR (100 MHz, CDCl₃) δ : 21.3, 126.3, 127.3, 128.0, 128.1, 128.4, 128.6, 128.8, 129.9, 129.9, 134.5, 135.5, 136.3, 138.1, 138.5, 141.0, 199.5; MS (EI, *m/z*): 298 (92, M⁺), 283 (11), 181 (25), 178 (30), 119 (100), 91 (33); FTIR (ATR) ν (cm⁻¹) CO: 1660; HRMS (*m/z*, M⁺): 298.1352 (hesaplanan), 298.1359 (saptanan).

(E)-1-Fenil-2-propilheks-2-en-1-on (3ba): Hekzan/etil asetat; açık sarı yağimsı; ¹H NMR (400 MHz, CDCl₃) δ : 0.94 (t, *J* = 7.4 Hz, 3H), 0.96 (t, *J* = 7.4 Hz, 3H), 1.41-1.52 (m, 4H), 2.27 (q, *J* = 7.2 Hz, 2H), 2.47 (t, *J* = 7.2 Hz, 2H), 6.20 (t, *J* = 7.6 Hz, 1H); 7.38-7.43 (m, 2H), 7.47-7.52 (m, 1H), 7.63-7.67 (m, 2H); ¹³C NMR (100 MHz, CDCl₃) δ : 14.0, 14.1, 22.2, 28.7, 30.9, 128.0, 129.3, 131.4, 139.1, 141.2, 145.7, 199.0; MS (EI, *m/z*): 216 (100, M⁺), 173 (7), 145 (9), 105 (27), 77 (5); FTIR (ATR) ν (cm⁻¹) CO: 1647; HRMS (*m/z*, M⁺): 216.1509 (hesaplanan), 216.1507 (saptanan).

(E)-1-(4-Metoksifenil)-2-propilheks-2-en-1-on (3bb): Hekzan/etil asetat; açık sarı yağimsı; ¹H NMR (400 MHz, CDCl₃) δ : 0.91-0.98 (m, 6H), 1.41-1.51 (m, 4H), 2.26 (q, *J* = 7.2 Hz, 2H), 2.46 (t, *J* = 7.6 Hz, 2H), 3.86 (s, 3H), 6.11 (t, *J* = 7.6 Hz, 1H), 6.91 (d, *J* = 8.8 Hz, 2H), 7.71 (d, *J* = 8.8 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃) δ : 14.0, 14.1, 22.2, 22.3, 29.2, 30.7, 55.4, 113.3, 131.4, 131.8, 141.1, 143.1, 162.6, 198.0; MS (EI, *m/z*): 246 (14, M⁺), 203 (22), 175 (18), 135 (100), 107 (10), 92 (14), 77 (21); FTIR (ATR) ν (cm⁻¹) CO: 1641; HRMS: 246.1614 (hesaplanan), 246.1603 (saptanan).

(E)-2-Propil-1-*p*-tolilheks-2-en-1-on (3bc): Hekzan/etil asetat; açık sarı yağimsı; ¹H NMR (400 MHz, CDCl₃) δ : 0.94 (t, *J* = 7.6 Hz, 6H), 1.46 (sext, *J* = 7.6 Hz, 4H), 2.26 (q, *J* = 7.6 Hz, 2H), 2.40 (s, 3H), 2.46 (t, *J* = 8.0 Hz, 2H), 6.16 (t, *J* = 7.4 Hz, 1H), 7.21 (d, *J* = 8.0 Hz, 2H), 7.59 (d, *J* = 8.0 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ : 14.0, 14.1, 21.5, 22.2, 22.3, 28.9, 30.8, 128.7, 129.6, 136.2, 141.2, 142.1, 144.6, 198.8; MS (EI, *m/z*): 230 (36, M⁺), 215 (92), 201 (20), 187 (38), 173 (29), 159 (39), 145 (22), 119 (100), 91 (71); FTIR (ATR) ν (cm⁻¹) CO: 1645; HRMS (*m/z*, M⁺): 230.1665 (hesaplanan), 230.1662 (saptanan).

(E)-Metil 3-(4-metoksifenil)hept-2-enoat (3cb): Hekzan/etil asetat; açık sarı yağimsı; NMR (400 MHz, CDCl₃) δ : 0.89 (t, *J* = 7.2 Hz, 3H), 1.40-1.50 (m, 4H), 2.98 (t, *J* = 7.6 Hz, 2H), 3.76 (s, 3H), 3.88 (s, 3H), 5.99 (s, 1H), 6.95 (d, *J* = 9.2 Hz, 2H), 7.84 (d, *J* = 9.2 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃) δ : 13.8, 22.9, 29.2, 30.5, 51.5, 55.5, 113.8, 122.0, 128.8, 132.2,

157.9, 163.9, 166.1, 196.5; MS (EI, m/z): 276 (28, M^+), 245 (20), 217 (22), 135 (100); FTIR (ATR) ν (cm^{-1}) CO: 1654, 1722 (-COOCH₃); HRMS: 276.1356 (hesaplanan); 276.1355 (saptanan).

(E)-Metil 4-(4-metoksifenil)-3-metil-4-oksobut-2-enoat (3db): Hekzan/etil asetat; açık sarı yağimsı; ¹H NMR (400 MHz, CDCl₃) δ : 2.42 (d, $J = 1.6$ Hz, 3H), 3.77 (s, 3H), 3.88 (s, 3H), 6.09 (q, $J = 1.6$, 1H), 6.95 (d, $J = 9.2$ Hz, 2H), 7.83 (d, $J = 8.8$ Hz, 2H); ¹³C NMR (100 MHz, CDCl₃) δ : 16.0, 51.6, 55.5, 113.9, 123.0, 128.1, 132.2, 153.1, 163.9, 166.3, 196.5; MS (EI, m/z): 234 (16, M^+), 203 (20), 135 (100), 107 (10), 92 (10), 77 (10), 62 (9); FTIR (ATR) ν (cm^{-1}) CO: 1651, 1720 (-COOCH₃); HRMS: 234.0887 (hesaplanan), 234.0884 (saptanan).

(E)-2-Metil-1,3-difenilprop-2-en-1-on (3ea): Hekzan/etil asetat; sarı yağimsı; ¹H NMR (400 MHz, CDCl₃) δ : 2.27 (d, $J = 1.6$, 3H), 7.18 (d, $J = 1.6$, 1H), 7.30-7.56 (m, 8H), 7.71-7.77 (m, 2H); ¹³C NMR (100 MHz, CDCl₃) δ : 14.4, 128.2, 128.5, 128.6, 129.5, 129.7, 131.7, 135.8, 136.9, 138.5, 142.2, 199.5; MS (EI, m/z): 222 (100, M^+), 207 (33), 115 (32), 77 (48); FTIR (ATR) ν (cm^{-1}) CO: 1642; HRMS (m/z , M^+): 222.1039 (hesaplanan), 222.1032 (saptanan).

(E)-1-(4-Metoksifenil)-2-metil-3-fenilprop-2-en-1-on (3eb): Hekzan/etil asetat; sarı yağimsı; ¹H NMR (400 MHz, CDCl₃) δ : 2.25 (d, $J = 1.6$ Hz, 3H), 3.87 (s, 3H), 6.95 (d, $J = 9.2$ Hz, 2H), 7.10 (d, $J = 1.6$, 1H), 7.30-7.43 (m, 5H), 7.81 (d, $J = 9.2$ Hz, 2H); ¹³C NMR (100 MHz, CDCl₃) δ : 14.9, 55.5, 113.5, 128.3, 128.4, 129.6, 130.7, 132.0, 137.0, 140.0, 162.8, 198.3; MS (EI, m/z): 252 (38, M^+), 135 (100), 145 (15), 115 (26); FTIR (ATR) ν (cm^{-1}) CO: 1638; HRMS: 252.1145 (hesaplanan), 252.1138 (saptanan).

3fb ve 3fb': Hekzan/etil asetat; **(E)-2-Benziliden-1-(4-metoksifenil)pentan-1-on (3fb):** açık sarı yağimsı; ¹H NMR (400 MHz, CDCl₃) δ : 0.98 (t, $J = 7.2$ Hz, 3H), 1.57 (sext, $J = 7.6$ Hz, 2H), 2.71 (t, $J = 7.8$ Hz, 2H), 3.89 (s, 3H), 6.95 (d, $J = 9.2$ Hz, 2H), 6.98 (s, 1H), 7.30-7.42 (m, 5H), 7.85 (d, $J = 9.2$ Hz, 2H); ¹³C NMR (100 MHz, CDCl₃) δ : 14.3, 22.0, 30.2, 55.5, 113.5, 128.1, 128.4, 129.1, 130.9, 132.1, 135.9, 138.5, 142.4, 163.0, 198.2; MS (EI, m/z): 280 (56, M^+), 251 (15), 173 (16), 135 (100); FTIR (ATR) ν (cm^{-1}) CO: 1636; HRMS: 280.1458 (hesaplanan); 280.1460 (saptanan). **(E)-1-(4-Metoksifenil)-2-fenilhekz-2-en-1-on (3fb'):** açık sarı yağimsı; ¹H NMR (400 MHz, CDCl₃) δ : 0.92 (t, $J = 7.2$ Hz, 3H), 1.49 (sext, $J = 7.6$ Hz, 2H), 2.24 (q, $J = 7.2$ Hz, 2H), 3.85 (s, 3H), 6.35 (t, $J = 7.4$ Hz, 1H), 6.89 (d, $J = 9.2$ Hz, 2H), 7.23-7.38 (m, 5H), 7.81 (d, $J = 9.2$ Hz, 2H); ¹³C NMR (100 MHz, CDCl₃) δ : 13.9, 22.5, 31.4, 55.4, 113.4, 127.3, 128.2, 129.4, 130.8, 132.2, 136.5, 141.6, 142.2, 162.9, 196.1; MS

(EI, m/z): 280 (12, M^+), 251 (39), 135 (100); FTIR (ATR) ν (cm^{-1}) CO: 1638; HRMS (m/z , M^+): 280.1458 (hesaplanan), 280.1460 (saptanan).

3,4,5-Trifenilfuran-2(5H)-on (4aa): Hekzan/eter; açık sarı katı; E.N.: 121.1-123.5 °C; ^1H NMR (400 MHz, CDCl_3) δ : 6.3 (s, 1H), 7.10-7.51 (m, 15H); ^{13}C NMR (100 MHz, CDCl_3) δ : 84.0, 127.1, 127.9(2), 128.6(3C), 128.8(2C), 128.9(2C), 129.1, 129.2(2C), 129.6, 129.7(2C), 130.1, 131.4, 135.1; 159.6, 172.7; MS (EI, m/z): 312 (M^+), 207, 179; 105; FTIR (pelet) ν (cm^{-1}) CO: 1745; $\text{C}_{22}\text{H}_{16}\text{O}_2$ için hesaplanan C: 84.6%, H: 5.2%; saptanan C: 84.2%, H: 5.3%.

3,4-Fenil-5-*p*-tolilfuran-2(5H)-on (4ab): Hekzan/eter; beyaz katı; E.N.: 114.0-114.6 °C; ^1H NMR (400 MHz, CDCl_3) δ : 2.31 (s, 3H), 6.23 (s, 1H), 7.10-7.50 (m, 14H); ^{13}C NMR (100 MHz, CDCl_3) δ : 21.5, 83.6, 127.1, 127.8(2C), 128.6(2C), 128.8(2C), 128.9(2C), 129.0, 129.7(2C), 129.9(2C), 130.0, 130.2, 131.5, 132.0, 139.5, 159.5, 172.7; MS (EI, m/z): 326 (M^+), 221, 207, 179, 119; FTIR (ATR) ν (cm^{-1}) CO: 1752; $\text{C}_{23}\text{H}_{18}\text{O}_2$ için hesaplanan C: 84.6%, H: 5.6%; saptanan C: 84.4%, H: 5.7%.

5-(4-Metoksifenil)-3,4-difenilfuran-2(5H)-on (4ac): Hekzan/etil asetat; açık sarı pasta; ^1H NMR (400 MHz, CDCl_3) δ : 3.68 (s, 3H), 6.14 (s, 1H), 7.77-7.41 (m, 14H); ^{13}C NMR (100 MHz, CDCl_3) δ : 55.5, 83.7, 114.6, 127.0, 127.2, 128.2, 128.6(2C), 128.8(2C), 128.9(2C), 129.0, 129.3(2C), 129.7(2C), 130.1, 130.2, 131.5, 159.5, 160.6, 172.7; MS (EI, m/z): 342 (M^+), 178, 135 FTIR (ATR) ν (cm^{-1}) CO: 1749; $\text{C}_{23}\text{H}_{18}\text{O}_3$ için hesaplanan C: 80.7%, H: 5.3%; saptanan C: 80.6%, H: 5.7%.

5-(4-(Triflorometil)fenil)-3,4-difenilfuran-2(5H)-on (4ad): Hekzan/etil asetat; beyaz katı. E.N.: 132.9-133.5 °C; ^1H NMR (400 MHz, CDCl_3) δ : 6.32(s, 1H), 7.10-7.60 (m, 14H); ^{13}C NMR (100 MHz, CDCl_3) δ : 82.8, 122.6, 125.3, 126.1(tet), 126.8, 127.3, 128.1, 128.4, 128.7, 128.8, 129.2, 129.3, 129.6, 129.7, 129.8, 130.4, 131.0, 131.5, 131.8, 139.1, 159.1, 172.3; MS (EI, m/z): 380 (M^+), 207, 179; FTIR (ATR) ν (cm^{-1}) CO: 1749; $\text{C}_{23}\text{H}_{15}\text{F}_3\text{O}_2$ için hesaplanan C: 72.6%, H: 4.0%; saptanan C: 73.0%, H: 4.0%.

5-Fenil-3,4-dipropilfuran-2(5H)-on (4ba): Hekzan/etil asetat; renksiz yağmsı; ^1H NMR (400 MHz, CDCl_3) δ : 0.89 (t, $J=7.4$ Hz, 3H), 0.96 (t, $J=0.74$ Hz, 3H), 1.26-1.51 (m, 2H), 1.61 (sex, $J=7.4$, 2H), 1.92-2.0 (m, 1H), 2.28-2.37 (m, 3H), 5.67 (s, 1H), 7.16-7.2 (m, 2H), 7.34-7.40 (m, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 14.1, 14.3, 21.5, 21.8, 25.9, 28.8, 84.0, 127.2(2C), 127.5, 129.1(2C), 129.4, 135.4, 163.4, 174.8; MS (EI, m/z): 244 (M^+), 215, 201, 139, 129, 115, 105, 91, 77, 69; FTIR (film) ν (cm^{-1}) CO: 1756 (s) cm^{-1} ; $\text{C}_{16}\text{H}_{20}\text{O}_2$ için hesaplanan C: 78.7%, H: 8.3%; saptanan C: 78.7%, H: 8.1%.

3,4-Difenil-5-*m*-tolilfuran-2(5H)-on (4ae): Hekzan/etil asetat; sarı pasta; ^1H NMR (400 MHz, CDCl_3) δ : 2.21 (s, 3H); 6.13 (s, 1H); 6.98-7.41 (m, 14H); ^{13}C NMR (100 MHz, CDCl_3) δ : 20.3, 82.7, 123.8, 125.7, 127.1, 127.3 (2C), 127.5 (2C), 127.6 (2C), 127.7, 127.8, 128.4 (2C), 128.8, 128.8, 129.1, 130.1, 133.6, 137.7, 158.3, 171.5, MS (EI, m/z): 326 (M^+); 207; 221; 179; 119; FTIR (ATR) ν (cm^{-1}) CO: 1746; HRMS (m/z , M^+): 326.1327 (hesaplanan), 326.1303 (saptanan).

3,4-Difenil-5-*o*-tolilfuran-2(5H)-on (4ag): Hekzan/etil asetat; açık sarı katı; E.N.: 140.7-142.6 °C; ^1H NMR (400 MHz, CDCl_3) δ : 2.5 (s, 3H); 6.5 (s, 1H); 7.08-7.5 (m, 14H); ^{13}C NMR (100 MHz, CDCl_3) δ : 19.5, 81.0, 126.8, 127.8, 127.9, 128.4 (2C), 128.8 (2C), 129.0 (2C), 129.1, 129.5, 129.6 (2C); 130.1, 130.3, 131.3, 131.6, 133.1, 137.7, 159.3, 172.7; MS (EI, m/z): 326 (M^+); 207; 179; 119; 207; FTIR (ATR) ν (cm^{-1}) CO: 1741; $\text{C}_{23}\text{H}_{18}\text{O}_2$ için hesaplanan C: 84.6%, H: 5.6%; saptanan C: 84.1%, H: 5.7%;

5-(4-Asetilfenil)-3,4-difenilfuran-2(5H)-on (4af): Hekzan/etil asetat; sarı pasta; ^1H NMR (400 MHz, CDCl_3) δ : 2.25 (s, 3H), 6.34 (s, 1H), 7.00-7.90 (m, 14H); ^{13}C NMR (100 MHz, CDCl_3) δ : 26.9, 83.1, 126.7, 127.1, 127.9 (2C), 128.5 (2C), 128.7, 128.9 (2C), 129.1 (2C); 129.1 (2C); 129.3; 129.6 (2C); 130.4; 131.0; 137.9; 159.5; 172.6; 197.9; MS (EI, m/z): 354 (M^+); 281, 207, 179. FTIR (ATR) ν (cm^{-1}) CO: 1748; HRMS (m/z , M^+): 354.1256 (hesaplanan), 354.1241 (saptanan).

4ca ve 4ca': Hekzan/etil asetat, 4ce' büyük oranda izomerinden ayrıştırılabildi; **4,5-Difenil-3-propilfuran-2(5H)-on (4ca):** ^1H NMR (400 MHz, CDCl_3) δ : 0.97 (t, $J = 7.4$ Hz, 3H), 1.61-1.75 (m, 2H), 2.44-2.51 (m, 2H), 6.13 (s, 1H), 7.0-7.39 (m, 10H); MS(EI, m/z): 278 (M^+); HRMS (m/z , M^+): 278.1301 (hesaplanan); 278.1300 (saptanan); **3,5-Difenil-4-propilfuran-2(5H)-on (4ca')**: renksiz yağmsı; ^1H NMR (400 MHz, CDCl_3) δ : 0.87 (t, $J = 7.4$ Hz, 3H); 1.29-1.52 (m, 2H); 2.08 (ddd, $J = 14.2, 9.5, 5.4$ Hz, 1H), 2.58 (ddd, $J = 14.4, 9.6, 6.8$ Hz, 1H), 5.85 (s, 1H), 7.17-7.49 (m, 10H); FTIR (ATR) ν (cm^{-1}) CO: 1749.

4da ve 4da': Hekzan/diklorometan; **3-Metil-4,5-difenilfuran-2(5H)-on (da):** renksiz yağmsı; ^1H NMR (400 MHz, CDCl_3) δ : 7.20-7.40 (m, 10H), 6.18 (d, $J = 2.0$, 1H), 2.15 (d, $J = 1.6$, 3H) ^{13}C NMR (100 MHz, CDCl_3) δ : 174.71, 158.60, 135.31, 131.65, 129.88, 129.43, 129.04, 129.02, 128.25, 127.73, 124.36, 83.93, 77.58, 77.26, 76.94, 10.54.; MS(EI, m/z): 250 (M^+) 222, 145, 115; FTIR (ATR): ν (cm^{-1}) CO: 1747; HRMS(m/z , M^+): 250.0994 (hesaplanan); 250.0995 (saptanan); **4-Metil-3,5-difenilfuran-2(5H)-on (da')**: renksiz yağmsı; ^1H NMR (400 MHz, CDCl_3) δ : 7.25-7.56 (m, 10H), 5.74 (s, 1H), 2.00 (s, 3H); ^{13}C

NMR (100 MHz, CDCl₃) δ: δ 172.97, 160.61, 135.01, 130.06, 129.66, 129.31, 129.21, 129.05, 128.83, 128.77, 127.22, 126.67, 85.09, 77.61, 77.29, 76.97, 13.61; MS(EL, *m/z*): 250 (M⁺) 207, 145, 117; FTIR (ATR): ν (cm⁻¹) CO: 1748; HRMS(*m/z*, M⁺): 250.0994 (hesaplanan); 250.0983 (saptanan).

4ea ve 4ea': Hekzan/diklorometan; **4-(4-Asetilfenil)-3-butil-5-fenilfuran-2(5H)-on (4ea)**: renksiz yağmsı; ¹H NMR (400 MHz, CDCl₃) δ: ¹H NMR (400 MHz, CDCl₃) δ 7.92 (d, *J* = 8.8, 2H), 7.28 (m, 5H), 7.17 (m, 2H), 6.15 (s, 1H), 2.53 (s, 3H), 2.50 (m, 2H), 1.63 (m, 2H), 1.38 (sext, 2H), 0.91 (t, *J* = 7.3, 3H); ¹³C NMR (100 MHz, CDCl₃) δ: 14.0, 22.9, 24.5, 26.8, 31.0, 83.9, 127.5, 128.3, 128.9, 129.2, 129.6, 130.5, 134.8, 136.3, 137.7, 157.9, 173.8, 197.4, MS(EL, *m/z*): 334 (M⁺) 289, 185, 105; FTIR (ATR): ν (cm⁻¹) CO: 1747; HRMS(*m/z*, M⁺): 334.1569 (hesaplanan); 334.1559 (saptanan); **3-(4-Asetilfenil)-4-butil-5-fenilfuran-2(5H)-on (4ea')**: renksiz yağmsı; ¹H NMR (400 MHz, CDCl₃) δ: 8.03 (m, 3H), 7.61 (m, 3H), 7.52 (dd, *J* = 3.0, 6.8, 1H), 7.43 (m, 4H), 7.29 (m, 3H), 5.89 (s, 1H), 2.63 (s, 1H), 2.56-2.62 (m, 1H), 2.13 (ddd, *J* = 5.6, 9.8, 14.4, 1H), 1.45-1.49 (m, 1H), 1.34-1.38 (m, 1H), 1.21-1.29 (m, 2H), 0.80 (t, *J* = 7.2, 3H); ¹³C NMR (100 MHz, CDCl₃) δ: 13.8, 22.8, 26.9, 27.2, 30.2, 84.1, 126.1, 127.3, 128.7, 128.7, 129.5, 129.9, 134.6, 135.0, 137.1, 166.6; 172.6; 197.9; MS(EL, *m/z*): 334 (M⁺) 289, 185, 105; FTIR (ATR): ν (cm⁻¹) CO: 1750; HRMS(*m/z*, M⁺): 334.1569 (hesaplanan); 334.1559 (saptanan).

4fa ve 4fa': Hekzan/etil asetat; **3-Butil-4-(4-metoksifenil)-5-fenilfuran-2(5H)-on (4fa)**: beyaz katı; E.N.: 66.7-70.5 °C; ¹H NMR (400 MHz, CDCl₃) δ: 0.94 (t, *J* = 7.4 Hz, 3H); 1.42 (sext, *J* = 7.4 Hz, 2H), 1.54-1.73 (m, 2H), 2.51-2.56 (m, 2H), 3.77 (s, 3H), 6.12 (s, 1H), 6.85 (d, *J* = 8.8 Hz, 2H); 7.17-7.20 (m, 4H), 7.26-7.29 (m, 3H); ¹³C NMR (100 MHz, CDCl₃) δ: 14.1, 23.1, 24.6, 30.6, 55.5, 83.7, 114.5 (2C), 124.0, 127.4, 127.7 (2C), 129.0 (2C), 129.3, 129.6 (2C), 135.7, 158.3, 160.7, 174.7; MS(EL, *m/z*): 322(M⁺), 255, 255, 105; FTIR (ATR): ν (cm⁻¹) CO: 1726; **4-Butil-3-(4-metoksifenil)-5-fenilfuran-2(5H)-on (4fa')**: açık sarı, katı; E.N.: 84.1-86.5 °C; ¹H NMR (400 MHz, CDCl₃) δ: 0.83 (t, *J* = 7.2 Hz, 3H); 1.20-1.50 (m, 4H), 2.08 (ddd, *J* = 14.0, 9.6, 5.4 Hz, 1H), 2.61 (ddd, *J* = 15.2, 9.0, 6.2 Hz, 1H), 3.84 (s, 3H), 5.83 (s, 1H), 6.98 (d, *J* = 8.8 Hz, 2H), 7.26-7.30 (m, 2H), 7.38-7.42 (m, 3H), 7.46 (d, *J* = 8.8 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃) δ: 13.9, 22.9, 27.1, 30.2, 55.6, 88.8, 114.2 (2C), 122.4, 126.3, 127.4 (2C), 129.3 (2C), 129.6, 130.5 (2C), 135.2, 160.0, 163.6, 173.5; MS(EL, *m/z*): 322(M⁺), 217, 105; FTIR (ATR): ν (cm⁻¹) CO: 1732; C₂₁H₂₂O₃ için hesaplanan C: 78.2%, H: 6.9%; saptanan C: 77.8%, 7.0%.

4ga ve 4ga': Hekzan/etil asetat; **3-Butil-4-(2-metoksifenil)-5-fenilfuran-2(5H)-on (4ga)**: açık sarı katı; E.N.:141.5-145.2 °C ¹H NMR (400 MHz, CDCl₃) δ: 0.85 (t, *J* = 7.2 Hz, 3H), 1.29 (sext, *J* = 7.6 Hz, 2H), 1.45-1.64 (m, 2H), 2.39 (t, *J* = 7.6 Hz, 2H), 3.81 (s, 3H), 6.36 (s, 1H), 6.80-7.30 (m, 9H), ¹³C NMR (100 MHz, CDCl₃) δ: 13.9, 22.8, 24.5, 30.2, 55.6, 83.9, 111.2, 120.8, 120.9, 126.9(2C), 128.7(2C), 128.8, 129.2, 130.1, 130.9, 135.7, 155.6, 159.3, 174.6; MS(EI, *m/z*): 322(M⁺), 251, 217, 121; FTIR (ATR): ν (cm⁻¹) CO: 1746; HRMS(*m/z*, M⁺): 322.1600 (hesaplanan), 322.1600 (saptanan); **4-Butil-3-(2-metoksifenil)-5-fenilfuran-2(5H)-on (4ga')**: sarı pasta; ¹H NMR (400 MHz, CDCl₃) δ: 0.74 (t, *J* = 7.2 Hz, 3H), 1.0-1.4 (m, 4H), 1.98 (ddd, *J* = 14.5, 9.1, 6 Hz, 1H), 2.31 (ddd, *J* = 15.6, 9.2, 6.8 Hz, 1H), 3.85 (s, 3H), 5.9 (s, 1H), 6.9-7.4 (m, 9H), ¹³C NMR (100 MHz, CDCl₃) δ: 13.9, 22.7, 27.6, 30.2, 55.8, 84.3, 111.4, 119.4, 120.8, 125.1, 127.6 (2C), 129.2 (2C), 129.6, 130.4, 131.2, 135.3, 157.5, 164.4, 173.2; MS(EI, *m/z*): 322(M⁺), 265, 189, 121; FTIR (ATR): ν (cm⁻¹) CO: 1750; HRMS(*m/z*, M⁺): 322.1600 (hesaplanan), 322.1559 (saptanan).

ha ve ha': Hekzan/diklorometan; **4-(4-Asetilfenil)-3,5-difenilfuran-2(5H)-on (ha)**: açık sarı katı; E.N.: 78.9-80.1 °C; ¹H NMR (400 MHz, CDCl₃) δ: 7.79 (d, *J* = 8.4 Hz, 2H), 7.26-7.47 (m, 10H), 7.29 (d, *J* = 8.8 Hz, 2H), 6.28 (s, 1H), 2.51 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ: 26.9, 83.8, 127.8, 128.5, 128.8, 128.9, 129.3, 129.5, 129.6, 129.8, 158.3, 172.3, 197.5 ; MS(EI, *m/z*): 354 (M⁺), 281, 249, 105; FTIR (ATR): ν (cm⁻¹) CO: 1749; C₂₄H₁₈O₃ için hesaplanan C: 81.3%, H: 5.1% ; saptanan C: 81%, H: 5.3%; HRMS(*m/z*, M⁺): 354.1256 (hesaplanan), 354.1241 (saptanan). **3-(4-Asetilfenil)-4,5-difenilfuran-2(5H)-on (ha')**: ¹H NMR (400 MHz, CDCl₃) δ: 7.93 (d, *J* = 8.8 Hz, 2H), 7.59 (d, *J* = 8.8 Hz, 2H), 7.20-7.34 (m, 8H), 7.08 (d, *J* = 8.4 Hz, 2H), 6.29 (s, 1H), 2.60 (s, 3H) ¹³C NMR (100 MHz, CDCl₃) δ: 26.9, 84.1, 126.1, 128.8, 128.7, 129.1, 129.2, 129.7, 129.9, 130.5, 130.9, 134.6, 134.9, 137.2, 161.1, 172.1, 197.9; MS(EI, *m/z*): 354 (M⁺) 221, 105; FTIR (ATR): ν (cm⁻¹) CO: 1751; C₂₄H₁₈O₃ için hesaplanan C: 81.3%, H: 5.1%, saptanan C: 80%, H: 5.6%; HRMS: 354.1256 (hesaplanan),: 354.1240 (saptanan).

4ia ve 4ia': Hekzan/etil asetat; **4-(4-Metoksifenil)-3,5-difenilfuran-2(5H)-on (4ia)**: açık sarı katı; E.N.: 139.3-142.9; ¹H NMR (400 MHz, CDCl₃) δ: 3.73 (s, 3H), 6.24 (s, 1H), 6.70 (d; *J* = 4.8, 2H), 7.09 (d, *J* = 8.8, 2H), 7.30-7.51 (m, 10H), ¹³C NMR (100 MHz, CDCl₃) δ: 55.4, 83.7, 114.3 (2C), 123.4, 125.7, 128.0 (2C), 128.9 (2C), 128.9, 129.2 (2C), 129.6, 129.7 (2C), 130.3 (2C), 130.7, 135.5, 158.8, 161.0, 172.9; FTIR (ATR): ν (cm⁻¹) CO: 1741; HRMS: 342.1247 (hesaplanan),: 354.1250 (saptanan). **3-(4-Metoksifenil)-4,5-difenilfuran-2(5H)-on**

(4ia'): açık sarı pasta; ^1H NMR (400 MHz, CDCl_3) δ : 3.74 (s, 3H), 6.14 (s, 1H), 6.80 (d, $J = 8.8$, 2H), 7.02-7.05 (m, 2H), 7.12-7.25 (m, 8H), 7.37 (d, $J = 9.2$, 2H), ^{13}C NMR (100 MHz, CDCl_3) δ : 55.5, 83.9, 114.2 (2C), 122.2, 126.5, 127.9 (2C), 128.5 (2C), 128.9 (2C), 129.1 (2C), 129.5, 129.9, 131 (2C), 131.7, 135.1, 158.1, 160.2, 173.0; FTIR (ATR): ν (cm^{-1}) CO: 1751; MS(EI, m/z): 342 (M^+), 237, 165, 105; $\text{C}_{23}\text{H}_{18}\text{O}_3$ için hesaplanan C: 80.7%, H: 5.3%; saptanan C: 80.0%, H: 5.4%; HRMS(m/z , M^+): 342.1247 (hesaplanan), 342.1254 (saptanan).

ja ve ja': Hekzan/etil asetat; **3,5-Difenil-4-*o*-tolilfuran-2(5H)-on** : Açık sarı katı; E.N.: 138.0-141.9; ^1H NMR (400 MHz, CDCl_3) δ : 7.10-7.40 (m, 14H), 6.41 (geniş s, 1H), 2.39 ve 2.02 (s, 2/1 oranında 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 83.9, 126.6, 128.0, 128.5, 128.9, 129.3(2C), 129.7, 130.4, 130.9, 135.6, 159.2; MS(EI, m/z): 326 (M^+), 282, 236, 105; FTIR (ATR): ν (cm^{-1}) CO: 1750; HRMS(m/z , M^+): 326.1301 (hesaplanan); 326.1304 (saptanan).

4,5-Difenil-3-*o*-tolilfuran-2(5H)-on: sarı pasta; ^1H NMR (400 MHz, CDCl_3) δ : 6.9-7.46 (m, 14H), 6.11 (s, 1H), 1.70 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 172.62, 161.59, 135.62, 134.38, 131.45, 130.96, 130.08, 129.78, 129.43, 129.35, 129.28, 129.18, 129.04, 128.81, 128.66, 128.62, 128.59, 128.52, 128.20, 126.63, 126.49, 126.21, 84.53, 19.56, 0.22.; MS(EI, m/z): 326 (M^+), 194, 105; FTIR (ATR): ν (cm^{-1}) CO: 1752; HRMS(m/z , M^+): 326.1301 (hesaplanan), 326.1295 (saptanan).

4ka ve 4ka': Hekzan/etil asetat; **4-(2-Metoksifenil)-3,5-difenilfuran-2(5H)-on**: (4ka): açık sarı katı; E.N.: 67.7-69.1; ^1H NMR (400 MHz, CDCl_3) δ : 3,68 (s, 3H); 6,49 (s, 1H); 6,72 (t, $J = 7,6$; 1H); 6,78-6,84 (m, 2H); 7,18-7,30 (m, 9H); 7,44-7,47 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ : 55,5; 83,9; 111,5; 120,8; 121,0; 127,2; 127,3 (2C); 128,4; (2C); 128,7; 128,8 (2C); 129,1; 129,2 (2C); 130,5; 130,5; 131,3; 135,3; 156,8; 159,5; 172,9; MS(EI, m/z): 342 (M^+) 237, 209, 91; FTIR (ATR): ν (cm^{-1}) CO: 1744; $\text{C}_{23}\text{H}_{18}\text{O}_3$ için hesaplanan C: 80.7%, H: 5.3% ; saptanan C: 80.4 % , H: 5.4%; **3-(2-Metoksifenil)-4,5-difenilfuran-2(5H)-on** : E.N.: açık sarı katı; 142.0-145.7 °C; ^1H NMR (400 MHz, CDCl_3) δ : 3,61 (s, 3H); 6,34 (s, 1H); 6,94 (d, $J = 8,4$; 1H); 7,02 (t; $J = 7,6$; 1H); 7,1-7,4 (m, 12H); ^{13}C NMR (100 MHz, CDCl_3) δ : 55,6; 83,9; 111,7; 120,0; 121,2; 125,2; 128,1 (2C); 128,1 (2C); 128,6 (2C); 129,2 (2C); 129,5; 130,0; 130,6; 131,2; 131,9; 135,8; 157,5; 159,5; 172,8; FTIR (ATR): ν (cm^{-1}) CO: 1745; $\text{C}_{23}\text{H}_{18}\text{O}_3$ için hesaplanan C: 80.7%, H: 5.3% , saptanan C: 80.0%, H: 5.2%; HRMS(m/z , M^+): 342.1256 (hesaplanan),: 342.1242 (saptanan).

4la ve 4la': Hekzan/etil asetat; **4-(4-Asetilfenil)-3-(2-metoksifenil)-5-fenilfuran-2(5H)-on (la')**: izomerik karışımından kısmen izole edilmiştir; sarı pasta; ^1H NMR (400 MHz,

CDCl_3) δ : 7.73 (m, 2H), 7.36 (m, 7H), 7.23 (m, 3H), 7.03 (td, $J = 1.0, 7.5, 1\text{H}$), 6.94 (dd, $J = 0.8, 8.3, 1\text{H}$), 6.36 (s, 1H), 3.60 (s, 3H), 2.48 (m, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ : 26.5, 55.3, 83.6, 111.4, 119.1, 121.1, 126.5, 127.8, 128.0, 128.1, 128.2, 129.1, 129.5, 130.8, 130.9, 135.0, 136.2, 137.4, 157.1, 158.0, 172.1, 198.2; MS(EI, m/z): 385 (M^+), 339, 251, 105; FTIR (ATR) ν (cm^{-1}) CO: 1748; HRMS(m/z , M^+) 384.1356 (hesaplanan); 384.1355 (saptanan).

2,3-Dihidro-2,3-difenilinden-1-on (6aa): Hekzan/etil asetat; turuncu yağimsı ^1H NMR (400 MHz, CDCl_3) δ : 3.81 (d, $J = 4.8$ Hz, 1H), 4.57 (d, $J = 4.8$ Hz, 1H), 7.07-7.13 (m, 4H), 7.23-7.82 (m, 4H), 7.48 (t, $J = 7.4$ Hz, 1H), 7.64 (dt, $J = 1.0$ Hz, $J = 7.6$ Hz, 1H), 7.89 (d, $J = 8$ Hz, 1H), ^{13}C NMR (100 MHz, CDCl_3) δ : 55.1, 64.9, 124.3, 126.9, 127.4, 127.4, 128.1 (2C), 128.5, 128.6 (2C), 129.1(2C), 129.2 (2C), 135.7, 136.4, 138.8, 142.8 156.4, 205.5, MS(EI, m/z): 284 (M^+), 206, 178; FTIR (ATR) ν (cm^{-1}) CO: 1711; HRMS(m/z , M^+) 284.1196 (hesaplanan); 284.1206 (saptanan).

2,3-Dihidro-6-metoksi-2,3-difenilinden-1-on (6ab): Hekzan/etil asetat; beyaz katı; E.N.: 137.1-146.8 °C; ^1H NMR (400 MHz, CDCl_3) δ : 3,80 (d, $J = 4,4$ Hz, 1H); 3,88 (s, 3H); 4,5 (d, $J = 4,4$ Hz, 1H); 7,06-7,34 (m, 14H); ^{13}C NMR (100 MHz, CDCl_3) δ : 54,5; 56,0; 65,6; 105,2; 125,1; 127,4; 127,4; 127,7; 128,0 (2C); 128,6 (2C); 129,1 (2C); 129,1 (2C); 137,7; 138,9; 143,0; 149,3; 160,3; 205,5; MS(EI, m/z): 314 (M^+), 223; FTIR (ATR) ν (cm^{-1}) CO: 1698; HRMS(m/z , M^+) 314.1301 (hesaplanan); 314.1290 (saptanan); **6-Metoksi-2,3-difenil-1H-inden-1-on (5ab)**: ^1H NMR (400 MHz, CDCl_3) δ : 3,85 (s, 3H); 6,80 (dd, $J = 2,2$ Hz, $J = 8,0$ Hz, 1H); 7,04 (d, $J = 8,0$, 1H); 7,17-7,26 (m, 6H); 7,34-7,43 (m, 4H); MS(EI, m/z): 312 (M^+); 270; HRMS(m/z , M^+) 314.1301 (hesaplanan); 284.1290 (saptanan).

2,3-Dihidro-6-metil-2,3-difenilinden-1-on (6ac): Hekzan/etil asetat; beyaz katı; E.N.: 67.4-74.8 °C; ^1H NMR (400 MHz, CDCl_3) δ : 2,46 (s, 3H); 3,79 (d, $J = 4,8$ Hz, 1H); 4,53 (d, $J = 4,8$ Hz, 1H); 7,05-7,11 (m, 4H); 7,19 (d, $J = 7,6$ Hz, 1H); 7,23-7,33 8 (m, 6H); 7,46 (dd, $J = 1,4$ Hz, $J = 7,8$, 1H); 7,69 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ : 21,4; 54,8; 65,2; 124,2; 126,6; 127,3; 127,4; 128,1 (2C); 128,6 (2C); 129,1 (2C), 129,1 (2C); 136,6; 136,9; 138,6; 139,0; 143; 153,9; 205,6; MS(EI, m/z): 298 (M^+), 208, 178; HRMS(m/z , M^+) 298.1352 (hesaplanan); 298.1341 (saptanan).

2,3-Dihidro-5-metill-2,3-difenilinden-1-on (6ad): Hekzan/etil asetat; açık sarı katı; E.N.: 113.9-117.5 °C; ^1H NMR (400 MHz, CDCl_3) δ : 2,40 (s, 3H); 3,79 (d, $J = 4,8$ Hz, 1H), 4,51 (d, $J = 4,8$ Hz, 1H), 7,06-7,12 (m, 5H), 7,2-7,34 (m, 7H), 7,78 (d, $J = 7,6$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ : 22,2, 54,8, 64,7, 123,9, 126,9, 127,1, 127,9, 123, 1288, 128,9 129,6,

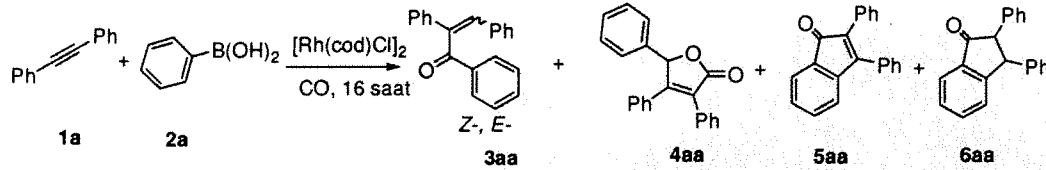
134.0, 138.8, 142.7, 146.8, 156.7; MS(EI, m/z): 298 (M^+), 221, 178; FTIR (ATR) ν (cm^{-1}) CO: 1696; HRMS(m/z , M^+) 298.1352 (hesaplanan); 298.1340 (saptanan).

2,3-Dihidro-5-metoksi-2,3-difenilinden-1-on (6ae): Hekzan/etil asetat; beyaz katı; E.N.: 139-142 °C; ^1H NMR (400 MHz, CDCl_3) δ : 3.78 (d, $J = 4.8$ Hz, 1H), 3.80 (s, 3H), 4.49 (d, $J = 4.8$ Hz, 1H), 6.69 (d, $J = 1.2$ Hz, 1H), 7.0 (dd, $J = 2.4$ Hz, $J = 8.4$ Hz, 1H), 7.09 (d, $J = 7.6$ Hz, 4H), 7.22-7.34 (m, 6H), 7.82 (d, $J = 8.4$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ : 55.0, 55.8, 64.8, 109.6, 116.5, 125.8, 127.1, 127.2, 127.9 (2C), 128.3 (2C), 128.8 (2C), 128.9 (2C), 129.6, 139.0, 142.6, 159.2, 165.9, 203.4, MS(EI, m/z): 314 (M^+), 238, 165; FTIR (ATR) ν (cm^{-1}) CO: 1702; HRMS(m/z , M^+) 314.1301 (hesaplanan); 284.1287 (saptanan).

3. Bulgular ve Tartışma

3.1. Furanon Sentezi (4)

Fenilboronik asit (**2a**) ve difenil asetilen (**1a**) karışımının (3:1 oranında) $[\text{Rh}(\text{cod})\text{Cl}]_2$ (%3 Rh) kompleksinin bulunduğu ortamda, dioksan içerisinde, 20 atm CO gazı altında, 120 °C sıcaklıkta 16 saat karbonilasyon tepkimesi sonucu, (*E/Z*)-1,2,3-trifenilprop-2-ene-1-one (**3aa**), 2,3-difenil-1*H*-inden-1-one (**5aa**), 2,3-dihidro-2,3-difenilinden-1-one (**6aa**) bileşikleriyle başlıca ürün olarak 3,4,5-trifenilfuran-2(5*H*)-one (**4aa**) bileşiğini oluşturmuştur (Aksın ve diğ., 2006) (Şema 1 ve Çizelge 1).



Şema 1. Fenilboronik asitin difenil asetilene karbonilatif katılması.

Fenilboronik asitin direk karbonilasyon ürünü olan benzaldehit bileşiğinde önemli miktarda oluşmuştur (başlangıçta kullanılan fenilboronik asit miktarına göre % 45 mol). Tepkime ortamında PPh_3 ligandı ya da NEt_3 bazı bulunduğu **4aa** oluşumu önemli miktarda azalmıştır (Çizelge 1, no 2 ve 3). Tepkime seçimliliği dioksan-su (9:1) karışımında daha da düşmüştür (Çizelge 1, no 4); tepkime ortamında suyun bulunması **4aa** oluşumunu azaltmış diğer ürünlerin oluşumunu ise arttırmıştır. Tepkimede kuru dioksan kullanılması ile çok daha az miktarlarda benzaldehit ve **3aa** oluşmuştur. Nemli dioksan ile daha fazla benzaldehit ve diğer yan ürünlerin oluşması H^+ formunda ya da su-gazı tepkimesi sonucu oluşan H_2 den

artan hidrojen aktarımı neden olmuş olabilir. Ancak tepkime ortamında 4A moleküller elek bulunması tepkime verimliliğine ve seçimliliğine olumsuz etki göstermiştir (Çizelge 1, no 6).

Saptanan optimum tepkime sıcaklığında (80 °C) (Çizelge 1, no 7 ve 8) yan ürünlerin oluşumu oldukça azalmıştır. Ancak 60 °C sıcaklıkta katalizör hiç bir aktivite göstermemiştir. Rodyum derişiminin %1 mol oranına azaltılması 4aa oluşumunu azaltmıştır (Çizelge 1, no 9).

Dioksan yerine kuru toluen kullanılması 4aa oluşumunu arttırmış ve diğer yan ürünlerin oluşumunu önemli ölçüde azaltmıştır (Çizelge 1, no 1). Toluene içerisinde % 1 oranında rodyum kullanılması da tepkime verimliliği için yeterli görünmektedir (Çizelge 1, no 11). Rh derişiminin %0.3 oranına azaltılması tepkime verimliliğini azaltsa da bu düşüş fazla değildir (Çizelge 1, no 12). Dioksan içerisinde 60 °C sıcaklıkta 4aa oluşumu %0 iken toluene içerisinde orta derecede ürün oluşumu gözlenmiştir (Çizelge 1, no 13). Tepkime fazla miktarlarda fenilboronik asit kullanımı gerektirmemektedir. Fenilboronik asit/alkin oranının 1.2 olarak uygulanması tepkime verimliliği için yeterli görünmektedir (Çizelge 1, no 14).

Literatürde 5-aril furanonların karbonilatif sentezi üzerine yalnızca bir kaç metod vardır. 3-Aril-1-propinler ve iyodoarenlerin ya da açıl klorürlerin, Pd(O₂CCH₃)₂ (%5)-PPh₃ katalizli ortamda ve Et₃N bazı ile 20-80 atm basınç aralığında düşük ila iyi verimler arasında E-3-arilbutenolitler elde edilmiştir (Huang ve Alper, 1991). α-Keto alkinlerin Rh katalizli siklohidrokarbonilasyonları (van den Hoven ve diğ., 2000) ve allenil alkollerin Ru-katalizli siklokarbonilasyonları (Yoneda ve diğ., 2003) çok fonksiyonelli laktonları oluşturmuştur. Negishi ve grubu 5-aril furanonların sentezinde iki karbonilatif metod uygulamıştır (Copéret ve diğ., 1995): PdCl₂(PPh₃)₂ katalizörünün ve Et₃N bazının bulunduğu ortamda Z-β-iyodoenonların 100-140 °C tepkime sıcaklıklarında karbonilasyonu ile iyi verimde ürünler elde etmişlerdir. Ancak daha dolaysız metod olarak aril iyodürlerin iç-alkinlere Pd-katalizli karbonilatif katılması daha az etkinlikte gerçekleşerek düşükden orta dereceye değışen verimlilikte ürünler elde etmişlerdir.

Çizelge 1. Fenilboronik asit ve difenil asetilenin CO gazı basıncında Rh(I) katalizli tepkimesi.

No	Çözücü	Rh (%)	2a/1a	T (°C)	Dönüşüm (%)	3aa ^b	4aa ^b	5aa ^b	6aa ^b
1	dioksan	3	3	120	> 99	11	70	5	15
2	dioksan ^c	3	3	120	> 99	< 1	44	15	15
3	dioksan ^d	3	3	120	> 99	< 1	39	7	7
4	dioksan/H ₂ O (9/1)	3	3	120	> 99	20	39	11	24
5	dioksan ^e	3	3	120	> 99	< 1	74	4	9
6	dioksan ^{e,f}	3	3	120	> 99	< 1	43	9	16
7	dioksan ^e	3	3	100	> 99	< 1	80	2	5
8	dioksan ^e	3	3	80	> 99	1	86	2	3
9	dioksan ^e	1	3	80	88	1	63	2	4
10	toluen ^e	3	3	80	> 99	< 1	90	< 1	< 1
11	toluen ^e	1	3	80	> 99	< 1	85	1	< 1
12	toluen ^e	0,3	3	80	95	< 1	81	2	1
13	toluen ^e	1	3	60	63	< 1	43	< 1	< 1
14	toluen ^e	1	1.2	80	> 99	< 1	89 (78)	2	1

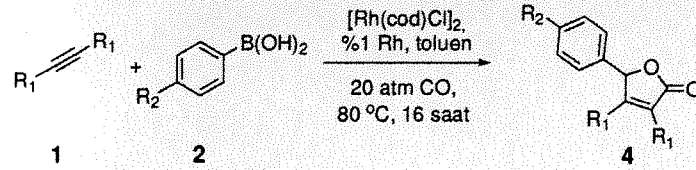
^a Tepkime koşulları: 1 mmol **1a**, 10 mL çözücü, 20 atm CO, 16 saat. ^b GK ürünü, izole ürün parantez içerisinde verilmiştir. ^c 0.06 eşdeğer PPh₃ eklenmiştir. ^d 2 mmol NEt₃ eklenmiştir. ^e Moleküler elek 4A ile kurutulmuştur. ^f 1 g moleküler elek 4A eklenmiştir.

[Rh(cod)Cl]₂ kompleksi ile optimize edilmiş koşulda elektronca zengin arilboronik asitlerin difenil asetilen ile yeterince reaktif oldukları görülmektedir (Çizelge 2, no 2 ve 3). 4-(Triflorometil)fenilboronik asit ile %1 Rh oranında orta derecede ürün elde edilmiştir (Çizelge 2, no 4). Ancak, Rh derişiminin %3 oranına artırılması yüksek oranda furanon ürününün elde edilmesini sağlamıştır (Çizelge 2, no 5). Muhtemelen oluşan aril grubunun bir electron çekici gruba sahip olması hem Rh(I) ile transmetallasyonunun hem de oluşan arilrodyum(I) yapısının CO ya eklenmesini zorlaştırmaktadır. Sterik nedenlerden dolayı *o*-tolilboronik asit ile difenil asetilen tepkime vermemiştir. *o*-Tolilboronik asit ile alkin dönüşümü % 50 civarındadır ve tepkime başlıca hidroarilasyon ürünü olan 1-metil-2-((*E*)-1,2-difenilvinil) benzen oluşturmuştur. Bu sonuçtan anlaşılacağı gibi 2-arilrodyum(I) yapıları alkinlere

eklenebilirken mevcut tepkime koşulunda ya aroilrodyum(I) ara yapısı oluşmamıştır ya da alkinlere eklenememiştir. Alkil sübtitüe alkinlere örnek olarak 4-oktin bileşiği de yüksek aktivite göstermiştir (Çizelge 2, no 6).

Ancak metod terminal alkinler için uygun değildir. Terminal alkinlerin polimerizasyon tepkimlerine yüksek meyili karbonilatif tepkimelerin oluşmamasına neden olmuş olabilir.

Çizelge 2. Arilboronik asit ve alkinlerin CO basıncı altında Rh katalizli tepkimeleri.^a



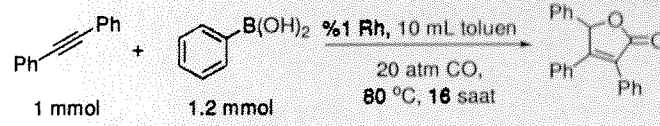
No	R ₁	R ₂	İzole ürün (%)
1	Ph	H	78 (4aa)
2	Ph	CH ₃	77 (4ab)
3	Ph	OCH ₃	82 (4ac)
4	Ph	CF ₃	40, 47 ^b (4ad)
5	Ph	CF ₃	93 ^b (4ad)
6	n-C ₃ H ₇	H	80 (4ba)

^a Tepkime koşulları Çizelge 1, no 14 için verildiği gibidir. ^b GK ile saptanmıştır.

Furanon oluşumuna farklı rodyum komplekslerin etkileri de incelenmiştir. İncelenen rodyum kompleksleri içerisinde [Rh(cod)OH]₂ ile bir miktar daha fazla ürün oluşumu ve daha tekrarlanabilir sonuçlar elde edilmiştir. Bu nedenle sonraki çalışmalar bu kompleks ile gerçekleştirilmiştir.

[Rh(cod)OH]₂ kompleksinin bulunduğu ortamda CO basıncının azaltılması **4aa** furanon oluşumunu azaltmıştır (Çizelge 4, no 1-3). Tepkime ortamında su ya da R-2-(diphenylphosphino)-1-(2-(diphenylphosphino)naphthalen-1-yl)naphthalene (R-BINAP) ligandın bulunması tepkime verimini azaltmıştır (Çizelge 4, no 4 ve 5). Ayrıca kiral bir merkeze sahip olan furanon ürün R-binap kiral ligand ile de rasemik olarak elde edilmiştir.

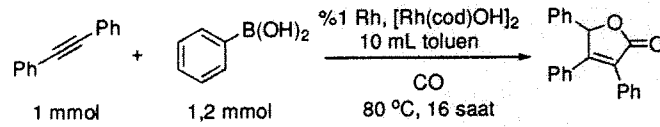
Çizelge 3. Fenilboronik asitin difenil asetilene karbonilatif katılma tepkimesine rodyum komplekslerinin etkisi.



No	Katalizör	%Dönüşüm ^a	%Ürün ^a
1	[Rh(cod)Cl] ₂	100	89 (78)
2	[Rh(cod)OH] ₂	100	93 (86)
3	[Rh(C ₂ H ₄) ₂ Cl] ₂	100	35
4	[Rh(C ₂ H ₄) ₂ acac] ₂	63	87
5	Rh(cod) ₂ BF ₄	100	84
6	[Rh(CO) ₂ Cl] ₂	100	88

^aGK ile saptanmıştır. İzole ürünler parantez içerisinde belirtilmiştir.

Çizelge 4. Fenilboronik asitin difenilasetilene karbonilatif katılma tepkimesine CO basıncının ve çeşitli katkı maddelerinin etkisi.

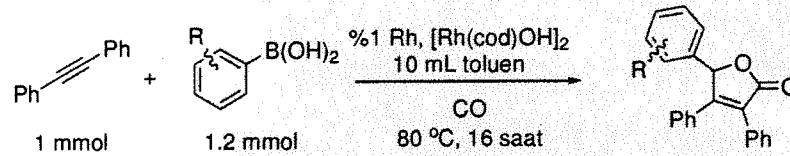


No	P, atm	%Dönüşüm ^a	%Ürün ^a
1	20	100	93 (86)
2	10	100	76
3	5	100	65
4 ^b	20	100	64
5 ^c	20	80	47

^a GK ürünü, izole ürünler parantez içerisinde gösterilmiştir. ^b 2 mmol su eklenmiştir. ^c %3 Rh ve %4.5 oranında R-BINAP içermektedir.

Çizelge 5 çeşitli arilboronik asit bileşiklerinin difenil asetilen ile CO basıncı altında tepkime sonuçlarını vermektedir. Genel olarak %1 Rh içeren ortamda elektronca zengin *m*-veya *p*- sübtitüe fenilboronik asitler ve düşük elektron yoğunluklu aromatik halkaya sahip *p*-asetilfenilboronik asit ile yüksek oranda furanon ürünler elde edilmiştir (no 1-5). Ancak *o*-tolilfenilboronik asit %3 Rh oranında orta derecede izole ürün elde edilmiştir (no 6) ve *p*-(triflorometil)fenilboronik asit ile yüksek verimde ilgili furanone oluşumu için rodyum derişiminin %3 oranına artırılması gerekmiştir (no 7). Burada [Rh(cod)Cl]₂ kompleksi ile *o*-tolilbronik asitin tepkime vermediğini belirtmeliyiz.

Çizelge 5. Difenil asetilenin çeşitli arilboronik asitler ile CO gazı altında rodyum katalizli tepkimeleri.



No	R	% İzole Ürün
1	H	86(4aa)
2	<i>p</i> -CH ₃	88(4ab)
3	<i>m</i> -CH ₃	90(4ae)
4	<i>p</i> -OCH ₃	90(4ac)
5	<i>p</i> -COCH ₃	88(4af)
6 ^a	<i>o</i> -CH ₃	41(4ag)
7 ^a	<i>p</i> -CF ₃	82(4ad)

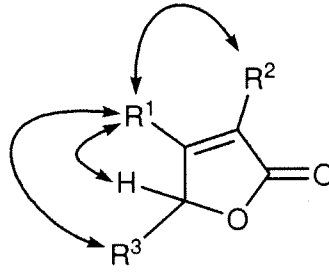
^a %3 Rh.

Çeşitli alkinler ile de furanon oluşum tepkimeleri gerçekleştirilmiştir. 4-Oktin ve bir çok asimetric alkin bileşikleri ile de %0.88-3 Rh derişimlerinde iyi derecede ve yüksek oranda furanon ürünleri elde edilmiştir (Çizelge 6).

Furanon **4ca** ve **4ca'** izomerleri kolon kromatografisi ile tam olarak ayırlamamış ancak zenginleştirilebilmişlerdir. Furanon **4la** ve **4la'** hariç diğer izomerik karışımlar kolon kromatografisi ile ayrıştırılabilmıştır. **4ca** yapısı literatür ile kıyaslanarak saptanmıştır (Ma ve

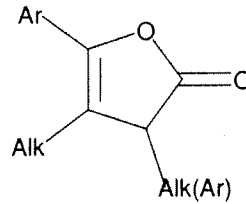
Shi, 2002). Literatür ve bizim bulgularımız kıyaslandığında furanon halkasının 4 ve 5 pozisyonlarının aril grubu olduğu tüm yapılarda ($R^1 = Ar$) 5H sinyali 1H -NMR spektrumunda 6 ppm den daha aşağı alanda 6-6.5 ppm arasında bir singlet olarak görünmektedir (4da yapısında dublet). Furanon halkasının 5 Pozisyonunda aril grubu 4 pozisyonunda bir alkil grubu olduğunda ise 5H daha yukarı alanda 5.5-6 ppm arasında singlet vermektedir (Aksın ve diğ., 2006; Ma ve Shi, 2002; Copéret ve diğ., 1995; Padwa ve Dehm, 1975; Padwa ve diğ., 1978; Gopidas ve diğ., 1987). Bu nedenle her iki alkil ve aril grupları içeren alkinler ile oluşan izomerik furanon ürünlerin tanımlanmalarında 5H sinyalinin kimyasal kayma değeri yardımcı olurken, NOE çalışmaları ile de çoğunlukla onaylanmıştır:

NOE



Asimetrik diinler ile elde edilen izomerik ürün dağılımı dikkate alındığında genel olarak metodun regio-seçimliliği çok yüksek olmasa da alkininin aroilleme adımının daha ziyade alkil süstitüe ve elektronca daha düşük yoğunluktaki asetilenik karbonu tercih ettiği görünmektedir.

Ham ürünlerin GK ve GK/MS analizleri ile 4-oktin ile gerçekleştirilen tepkime ürününde ikinci bir izomerik ürünün ve en az bir asetilenik karbonun bir alkil grubu bağlı olduğu asimetrik alkinlerin tepkimesinde ise üçüncü bir izomerin varlığı saptanmıştır. Ham ürünlerin NMR analizleri kararsız izomerik yapının 4-alkil-2(3H)-furanon yapısında olduğunu göstermektedir Bu yapı kolon kromatografisi ile saflaştırma sırasında veya bekletildiğinde daha kararlı olan 4 yapısına izomerize olmaktadır.:



Diaril asetilen ile gerçekleştirilen tepkimelerin ürün analizlerinde ise bu tip izomere rastlanmamıştır. Muhtemelen 4-aril-2(5H)-furanon yapılarının genişlemiş π electron

konjugasyonundan dolayı alkil gruplu 2(5*H*)-furanona kıyasla daha kararlı olması 4-aril-2(3*H*)-furanonların **4** yapısına izomerizasyonlarını hızlandırmıştır.

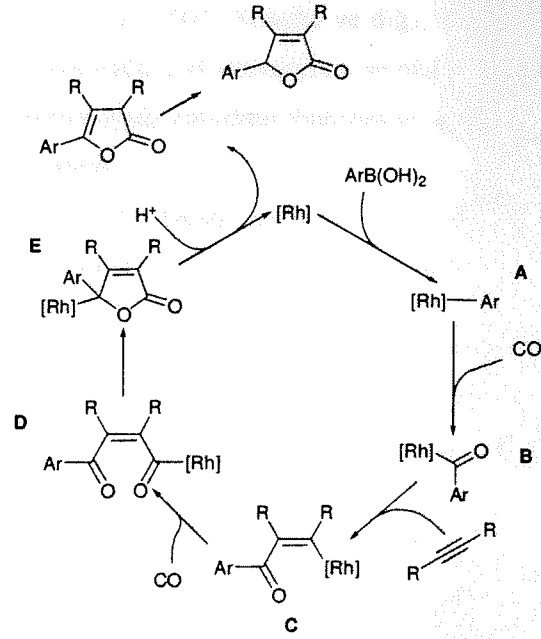
Çizelge 6. Fenilboronik asitin çeşitli alkinler ile CO gazı altında rodyum katalizli tepkimeleri.

%İzole Ürün					
No	R ¹	R ²	%Rh	4	% 4
1	n-C ₃ H ₇	n-C ₃ H ₇	0,88	60(4ba)	-
2	n-C ₃ H ₇	n-C ₃ H ₇	2,63	76(4ba)	-
3	Ph	n-C ₃ H ₇	2,63	35(4ca) ^a	47(4ca') ^a
4	Ph	CH ₃	2,63	32(4da)	32(4da')
5	<i>p</i> -CH ₃ COC ₆ H ₄	n-C ₄ H ₉	0,88	24(4ea)	42(4ea')
6	<i>p</i> -CH ₃ OC ₆ H ₄	n-C ₄ H ₉	1	36(4fa)	44(4fa')
7	<i>o</i> -CH ₃ OC ₆ H ₄	n-C ₄ H ₉	3	11(4ga)	48(4ga')
8	<i>p</i> -CH ₃ COC ₆ H ₄	Ph	0,88	45(4ha)	36(4ha')
9	<i>p</i> -CH ₃ OC ₆ H ₄	Ph	0,88	29(4ia)	48(4ia')
10	<i>o</i> -CH ₃ C ₆ H ₄	Ph	1	30(4ja)	42(4ja')
11	<i>o</i> -CH ₃ OC ₆ H ₄	Ph	1	26(4ka)	68(4ka')
12	<i>o</i> -CH ₃ OC ₆ H ₄	<i>p</i> -CH ₃ COC ₆ H ₄	1	23(4la) ^a	63(4la') ^a

^aIzomerik oran ¹H NMR ile saptanmıştır.

Bu çalışmadaki ve literatürdeki bulgular ışığında sırasal CO-alkin-CO eklenme tepkimelerinin 2(5*H*)-furanon oluşumundan sorumlu olduğunu düşünmekteyiz (Şema 2): organoboron bileşikleri ile Rh(I) komplekslerinin transmetalasyonu ile arilrodyum(I) yapılarının (A) oluştuğu genel bir kabuldür. Arilrodyum(I) CO ya eklenerek aroilrodyum(I) yapılarını (B) oluşturabilir (Aksın ve diğ., 2006; Sauthier ve diğ., 2004; Chochois ve diğ., 2006; Menard ve diğ., 2007; Dheur ve diğ., 2007) ve sonrasında karbon-karbon üçlü bağına 1,2-katılması gerçekleşir. Oluşan β-aroil alkenilrodyum(I) kompleksinin (C) CO ya eklenmesi ve sonrasında oluşan halka kapanması bir σ-furanoil kompleksini (E) oluşturur. Yapıdan

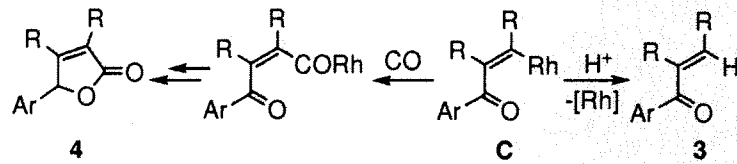
rodyum ayrılması 2(3*H*)-furanon oluşturacak ve sonrası izomerizasyonu ile 2(5*H*)-furanon yapısına dönüştüreceklerdir.



Şema 2. 2(5*H*)-Furanon (4) oluşum mekanizması

3.2. α,β -Doymamış Keton Oluşumu (3)

Arilboronik asitlerin alkinler ile karbonilatif kenetlenmesinin açilrodasyon adımı ile gerçekleştiği yukarıda belirtilmiş idi (Şema 2). Diğer bir değerli ürün olan 3 yapısının seçimli oluşumunu sağlamak amacıyla gerçekleştirdiğimiz optimizasyon çalışmasında alkenilrodyum (C) ara ürününün 4 tipi ürün oluşumuna neden olan CO ya eklenme aşamasından önce hızla protodemetallasyona uğramasının α,β -doymamış keton (3) oluşumunu arttıracaklarını öngörmüştük. Bu nedenle tepkime ortamında bir asidik katkı maddesinin bulunmasının, katalizörün aktivitesinin korunması koşulu ile, protodemetallasyon adımını arttıracaklarını düşündük (Kuş ve diğ.):



Genellikle rodyum katalizli hidroaçılleme tepkimelerinde açılleme reaktifi olarak aldehitler kullanılmaktadır (Willis ve diğ., 2006; Tanaka ve diğ., 2007; Imai ve diğ., 2004; Jun ve diğ., 2002; Moxham ve diğ., 2006; Tanaka ve diğ., 2005; Stemmlera ve diğ., 2007; Imai ve diğ., 2007; Masanori ve diğ., 2007; Kokubo ve diğ., 1997; Kokubo ve diğ., 1999). Rodyum katalizli tepkimeler genellikle C-H aktivasyonu ve oluşan açilrodyum ara yapılarının sübrattaki ya da katkı reaktiflerindeki koordine fonksiyonel gruplar ile şelatlaşarak kararlı yapıları oluşturmaları gerekmektedir.

Tepkimenin optimum koşulları difenil asetilen ve fenilboronik asit kullanılarak gerçekleştirilmiştir. Tepkime verimine rodyum kompleksi, çözücü, asidik katkılar, CO basıncı ve sıcaklık gibi bir çok değişkenin etkileri incelenmiştir.

Difenil asetilen (1 mmol) ve fenilboronik asit (3 mmol) karışımının CH₃OH:H₂O (9.9:0.1) çözücü karışımı içerisinde [Rh(cod)Cl]₂ (%3 Rh) ve 1 mmol HCOOH ile, 80°C sıcaklıkta 20 atm CO basıncında, 16 saat süre ile tepkimesi gerçekleştirilmiştir (Çizelge , no 1). Tepkimenin başlıca ürünü **4aa** ve difenil asetilenin direk metoksikarbonilasyon ürünü olan metil-2,3-difenilakrilat (**7a**) bileşikleridir. İlgili enon ürünü, **3aa**, E- ve Z-izomerik karışımı halinde düşük miktarda oluşmuştur. Proton kaynağı olarak CH₃COOH kullanımı **4aa** oluşumunu artırmıştır (Çizelge 7, no 2). Tepkime ortamında NH₄Cl bulunduğunda çok düşük kütle dengliği sağlanmıştır (Çizelge 7, no 3). Ancak, asidik reaktif olarak CF₃COOH kullanıldığında E- ve Z- **3aa** izomerleri %38 GK verimine ulaşmıştır (Çizelge 7, no 4). CF₃COOH derişiminin 5 kat artırılması **3aa** oluşumunu etkilememiştir (Çizelge 7, no 5). Tepkime sıcaklığının 70 °C sıcaklığa düşürülmesi reaktiviteyi önemli ölçüde azaltırken 120 °C sıcaklıkta biraz daha az miktarda enonlar oluşmuştur (Çizelge 7, no 6 ve 7).

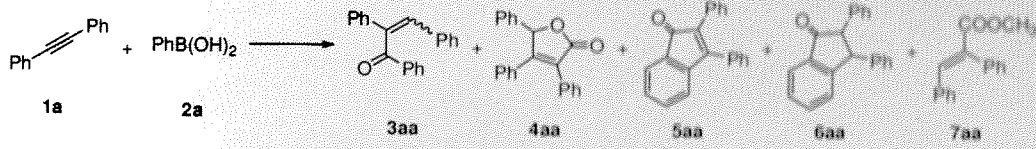
CO basıncının 5 atmosfere artırılması E- ve Z-**3aa** oluşumunu %65 e artırmıştır ve bu basınç optimum basınç olarak belirlenmiştir. CO basıncının 1 atmosfere azaltılması **5aa** and **6aa** yan ürünlerinin miktarlarını artırmıştır.

Genel olarak CF₃COOH derişiminin farklılaştırılması tepkime verimliliğini önemli derecede etkilememiştir (Çizelge 7, no 4, 5, 9, 17 ve 20). Bununla birlikte asitsiz ortamda daha düşük miktarda E- ve Z-**3aa** oluşmuştur (Çizelge 7, no 12). Çeşitli rodyum komplekslerinin aktiviteyi 80 °C, 5 atm CO tepkime koşulunda ve 10 mmol CF₃COOH bulunan ortamda incelenmiştir (Çizelge 7, no 13-17). RhCl₃.3H₂O çok düşük katalitik aktivite göstermiştir **1a** dönüşümü yalnızca %24 dür. Diğer kompleksler ile benzer sonuçlar elde edilebilmiştir. Ancak [Rh(CO)₂Cl]₂ ya da [Rh(C₂H₄)₂Cl]₂ kompleksleri ile bir miktar daha fazla enon ürünleri elde edilmiştir.

Çözücü olarak etanol ya da dioksan kullanıldığında daha düşük verimde **3aa** ürünleri elde edilmiştir (Çizelge 7, no 18 ve 19). Rh derişiminin %5 oranına artırılması tepkime verimini arttırmamıştır. Ancak %1 oranında bir Rh derişimi de yeterli görünmektedir (Çizelge 7, no 21 ve 22). Asidik reaktif olarak *p*-toluensulfonik asit veya ligand olarak 1,4-di(difenilfosfino)butan (dppb) kullanılması katalizör aktivitesini düşürmüştür (Çizelge 7, no 23 ve 24). Tepkime yüksek **2a:1a** oranı gerektirmemektedir; 3:1.5 oranında da tepkime verimi yeterli düzeydedir (Çizelge 7, no 25).

Bu çalışmanın gerçekleştirildiği sırada alkil süstitüe terminal alkinler ve arilboronik asitler ile rodyum katalizli karbonilatif tepkime uygulanarak α,β -doymamış ketonların sentezi gerçekleştirilmiş idi (Dheur ve diğ., 2007). Ancak Muhtemelen **4** tipi ürün oluşturma eğilimleri nedeniyle iç alkinler ile her hangi bir örnek gösterilmemiştir.

Çizelge 7. Fenilboronik asitin (**2a**) difenil asetilene (**1a**) karbonilatıf katılması; (*E,Z*)-1,2,3-trifenilprop-2-en-1-on (**3aa**) oluşumuna tepkime koşullarının etkisi.a



No	Katalizör	P, atm	T, °C	Çözücü	Reaktif, mmol	% Dönüşüm	%Ürün ^a					
							<i>E</i> - 3aa	<i>Z</i> - 3aa	4aa	5aa	6aa	7a
1	[Rh(cod)Cl] ₂	20	80	CH ₃ OH	HCOOH, 1	100	6	5	30	1	2	26
2	[Rh(cod)Cl] ₂	20	80	CH ₃ OH	CH ₃ COOH, 1	100	6	5	60	1	2	17
3	[Rh(cod)Cl] ₂	20	80	CH ₃ OH	NH ₄ Cl, 1	61	6	2	5	1	1	9
4	[Rh(cod)Cl] ₂	20	80	CH ₃ OH	CF ₃ COOH, 1	100	21	17	21	3	2	5
5	[Rh(cod)Cl] ₂	20	80	CH ₃ OH	CF ₃ COOH, 5	100	21	21	11	3	1	5
6	[Rh(cod)Cl] ₂	20	70	CH ₃ OH	CF ₃ COOH, 5	63	15	5	5	1	1	5
7	[Rh(cod)Cl] ₂	20	120	CH ₃ OH	CF ₃ COOH, 5	98	16	21	12	7	2	8
8	[Rh(cod)Cl] ₂	10	80	CH ₃ OH	CF ₃ COOH, 6	100	33	29	9	3	5	7
9	[Rh(cod)Cl] ₂	5	80	CH ₃ OH	CF ₃ COOH, 6	100	30	35	7	11	4	4
10	[Rh(cod)Cl] ₂	1	80	CH ₃ OH	CF ₃ COOH, 6	100	22	30	3	20	9	2
11	[Rh(cod)Cl] ₂	5	80	CH ₃ OH	CF ₃ COOH, 10	100	31	35	6	11	4	3
12	[Rh(cod)Cl] ₂	5	80	CH ₃ OH	-	100	19	28	11	6	8	5
13	RhCl ₃ .3H ₂ O	5	80	CH ₃ OH	CF ₃ COOH, 10	24	2	3	11	11	12	-
14	[Rh(CO) ₂ Cl] ₂	5	80	CH ₃ OH	CF ₃ COOH, 10	100	32	38	8	12	5	4

15	Rh(C ₂ H ₄) ₂ acac	5	80	CH ₃ OH	CF ₃ COOH, 10	100	25	37	9	5	4	5
16	[Rh(cod)OH] ₂	5	80	CH ₃ OH	CF ₃ COOH, 10	100	27	40	6	8	7	3
17	[Rh(C ₂ H ₄) ₂ Cl] ₂	5	80	CH ₃ OH	CF ₃ COOH, 10	100	31	42	7	18	6	2
18	[Rh(C ₂ H ₄) ₂ Cl] ₂	5	80	C ₂ H ₅ OH	CF ₃ COOH, 10	88	26	25	41	13	4	-
19	[Rh(C ₂ H ₄) ₂ Cl] ₂	5	80	Dioxane	CF ₃ COOH, 10	100	15	24	18	4	1	<1
20	[Rh(C ₂ H ₄) ₂ Cl] ₂	5	80	CH ₃ OH	CF ₃ COOH, 2	100	29	45	10	15	7	2
21	[Rh(C ₂ H ₄) ₂ Cl] ₂ ^b	5	80	CH ₃ OH	CF ₃ COOH, 2	100	27	40	10	9	4	3
22	[Rh(C ₂ H ₄) ₂ Cl] ₂ ^c	5	80	CH ₃ OH	CF ₃ COOH, 2	100	29	39	6	17	8	2
23	[Rh(C ₂ H ₄) ₂ Cl] ₂	5	80	CH ₃ OH	<i>p</i> -CH ₃ - C ₆ H ₄ SO ₃ H, 2	71	20	19	1	4	5	1
24	[Rh(C ₂ H ₄) ₂ Cl] ₂ / dppb ^d	5	80	CH ₃ OH	CF ₃ COOH, 2	52	4	1	34	1	1	-
25 ^e	[Rh(C ₂ H ₄) ₂ Cl] ₂ ^e	5	80	CH ₃ OH	CF ₃ COOH, 2	100	29	40	11	3	11	4

^a GK ile tespit edildi, 1 mmol **1a**, 3 mmol **2a**, %3 Rh, 9.9 mL çözücü, 0.1 mL H₂O, cod: siklooktadienil, acac: asetil asetonat, dppb: 1,4-bis(difenilfosfino)butan, ^b %5 Rh. ^c 1% Rh. ^d %3 dppb, ^e 1,5 mmol **1a**, 3 mmol **2a**.

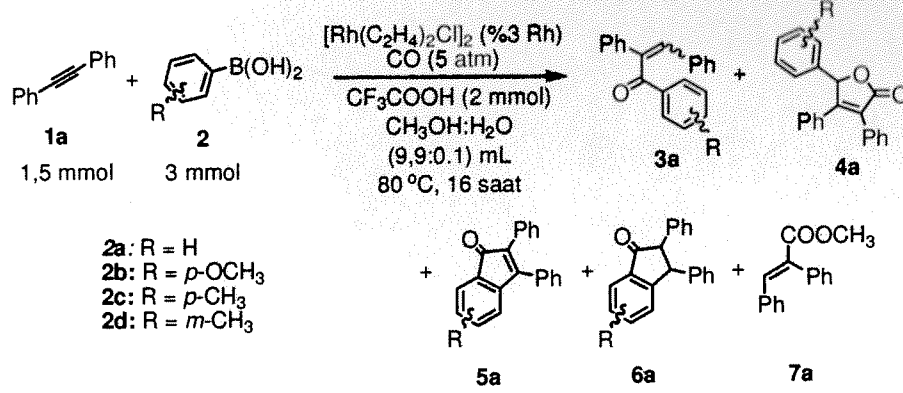
Difenil asetilenin (**1a**) çeşitli arilboronik asitler ile tepkimeleri *Z*- ve *E*- enon (**3a**) izomerik karışımları vermektedir. Bu izomerler flaş kromatografisi ile silika jel üzerinde ayrılabilir (Çizelge 1). Furanonlar (**4a**), indenonlar (**5a**), indanonlar (**6a**) ve **1a** nın metoksikarbonilasyon ürünü olan metal 2,3-difenilakrilat (**7a**) yan ürünleri de oluşmuştur.

15	Rh(C ₂ H ₄) ₂ acac	5	80	CH ₃ OH	CF ₃ COOH, 10	100	25	37	9	5	4	5
16	[Rh(cod)OH] ₂	5	80	CH ₃ OH	CF ₃ COOH, 10	100	27	40	6	8	7	3
17	[Rh(C ₂ H ₄) ₂ Cl] ₂	5	80	CH ₃ OH	CF ₃ COOH, 10	100	31	42	7	18	6	2
18	[Rh(C ₂ H ₄) ₂ Cl] ₂	5	80	C ₂ H ₅ OH	CF ₃ COOH, 10	88	26	25	41	13	4	-
19	[Rh(C ₂ H ₄) ₂ Cl] ₂	5	80	Dioxane	CF ₃ COOH, 10	100	15	24	18	4	1	<1
20	[Rh(C ₂ H ₄) ₂ Cl] ₂	5	80	CH ₃ OH	CF ₃ COOH, 2	100	29	45	10	15	7	2
21	[Rh(C ₂ H ₄) ₂ Cl] ₂ ^b	5	80	CH ₃ OH	CF ₃ COOH, 2	100	27	40	10	9	4	3
22	[Rh(C ₂ H ₄) ₂ Cl] ₂ ^c	5	80	CH ₃ OH	CF ₃ COOH, 2	100	29	39	6	17	8	2
23	[Rh(C ₂ H ₄) ₂ Cl] ₂	5	80	CH ₃ OH	<i>p</i> -CH ₃ - C ₆ H ₄ SO ₃ H, 2	71	20	19	1	4	5	1
24	[Rh(C ₂ H ₄) ₂ Cl] ₂ / dppb ^d	5	80	CH ₃ OH	CF ₃ COOH, 2	52	4	1	34	1	1	-
25 ^e	[Rh(C ₂ H ₄) ₂ Cl] ₂ ^e	5	80	CH ₃ OH	CF ₃ COOH, 2	100	29	40	11	3	11	4

^a GK ile tespit edildi, 1 mmol **1a**, 3 mmol **2a**, %3 Rh, 9.9 mL çözücü, 0.1 mL H₂O, cod: siklooktadienil, acac: asetil asetonat, dppb: 1,4-bis(difenilfosfino)butan, ^b %5 Rh. ^c 1 % Rh. ^d %3 dppb, ^e 1,5 mmol **1a**, 3 mmol **2a**.

Difenil asetilenin (**1a**) çeşitli arilboronik asitler ile tepkimeleri *Z*- ve *E*- enon (**3a**) izomerik karışımları vermektedir. Bu izomerler flaş kromatografisi ile silika jel üzerinde ayrılabilir (Çizelge 1). Furanonlar (**4a**), indenonlar (**5a**), indanonlar (**6a**) ve **1a** nın metoksikarbonilasyon ürünü olan metal 2,3-difenilakrilat (**7a**) yan ürünleri de oluşmuştur.

Çizelge 8. Arilboronik asitlerin (2) alkin 1a ya karbonilatif katılması ile enonların (3a) sentezi.



No	%Ürün					
	2	3a ^a	4a ^b	5a ^b	6a ^b	6a ^b
1	2a	3aa (33 Z, 25 E)	4aa (11)	5aa (3)	6aa (12)	6a ^b (4)
2	2b	3ab (45 Z, 31 E)	4ab (5)	5ab (2)	6ab (1)	6a ^b (3)
3	2c	3ac (55 Z, 16 E)	4ac (8)	5ac (<1)	6ac (5)	6a ^b (2)
4	2d	3ad (29 Z, 32 E)	4ad (7)	5ad (3)	6ad (9)	6a ^b (5)

^aİzole ürün. ^bGK ile tespit edildi.

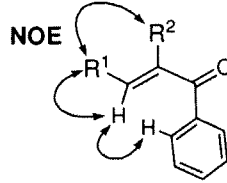
Enonların (3a) isomerik yapıları IR karbonil frekanslarının ve erime noktalarının literatür ile kıyaslanması ile tespit edilmiştir. Z-enonların gösterdikleri enon rezonanslarındaki sterik engellemenin varlığı nedeniyle karbonil absorpsiyon bandları E-izomerlerine kıyasla daha yüksek dalgasayısında görünmektedir (Duke ve Boykin, 1972; Black ve Lutz, 1953; Mittal ve diğ., 1985).

Arilboronik asit 2a'nın alkin 1a ile tepkimesi toplam %58 oranında E- ve Z-3aa ürünlerini oluşturmuştur. Toplam yan ürün oluşumu %30 oranındadır (Çizelge 8, no 1). *para*-Pozisyonunda elektron veren gruplar içeren arilboronik asitler 2b ve 2c, sırasıyla %76 oranında her iki E- ve Z-3ab ve %70 oranında E- ve Z-3ac yapılarını oluşturmuştur. Bu reaktifler ile daha az miktarlarda yan ürünle oluşmuştur (sırasıyla, %11 ve 16, Çizelge 8, no 2

ve 3). *m*-Tolilboronik asit, **2d**, ile gerçekleştirilen tepkime %61 oranında *E*- ve *Z*-**3ad** ile birlikte %24 oranında yan ürünler oluşturmuştur.

o-Tolilboronik asit ve *p*-(trifluorometil)fenilboronik asitin alkin **1a** ile tepkimeleri düşük oranlarda enon bileşikleri oluşturmuş, yan ürün oluşumları artmış ve ayrıca önemli miktarlarda hidroarilasyon ürünleri olan triarilasetilen yapıları oluşmuştur. Bu sonuçlar enon oluşumunun arilboronik asitlerin sterik ve elektronik doğasına duyarlı olduğunu göstermektedir.

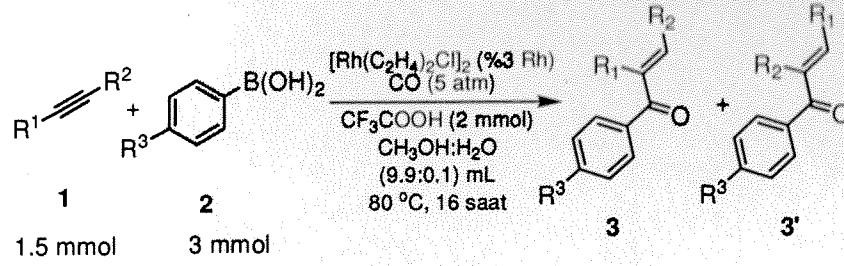
Aril süstitüe grubu içermeyen ya da yalnızca tek aril gruplu alkinlerin hidroarilasyonu tamamen *sin*-seçimliliğinde gerçekleşerek NOE çalışmaları ile anlaşılacağı gibi enonların yalnızca *E*- izomerini oluşturmuşlardır (Çizelge 9).



4-Oktinin (**1b**) **2a** ile karbonilatif tepkimesi düşük oranda *E*-**3ba** (%30) oluşturmuştur. *E*-**3bb** ve *E*-**3bc** enonlar, sırasıyla %57 ve %53 oranlarında **2b** ve **2c** arilboronik asitlerden oluşmuştur (no 2-3). Bir esterik fonksiyonel grup ile aktivite olmuş alkinlerin tepkimeleri regio-seçimli olarak yürümüştür. Aroil grup elektron çekici gruba göre β - pozisyonuna bağlanmıştır (no 4, 5 ve 9). Yüksek regio-seçimlilik 1-fenilpropin (**1e**) ile de gözlenmiştir. Aroil grup başlıca metil bağlı asetilenik karbona bağlanarak arilboronik asitler **2a** ve **2b** ile regio-izomerlerinden ayrılabilen **3ea** ve **3eb** ürünlerini vermiştir (no 6 ve 7). Bununla birlikte alkenil karbon üzerinde daha hacimli bir alkil grup bulunduğunda, muhtemelen sterik engelliliği nedeniyle tepkimenin regio-seçimliliği bir miktar azalmıştır (no 8).

Tepkime mekanizması olarak, arilboronik asitin tranmetallasyonu muhtemelen arilrodyum bağına CO eklenmesi takip etmektedir (Şema 3). Oluşan aroilrodyum (**B**) *sin*- modunda alkine 1,2- katılarak vinilrodyum yapıları verirler (**C**). Sonrasında protodemetallasyona uğrayarak *E*-enon oluştururlar. Bununla birlikte, Hayashi ve grubu tarafından gösterilen alkinlerin arilboronik asitler ile Rh-katalizli hidroarilasyonuna benzer olarak (1,4-kayması) alkenil karbon üzerindeki Rh ve karbonil fonksiyonel gruba göre *orto*-pozisyonundaki

Çizelge 9. Arilboronik asitlerin (2) alkinler ile (1) karbonilatıf katılması ile enonların (3) sentezi.



1b: $\text{R}^1 = \text{R}^2 = \text{n-C}_3\text{H}_7$

1c: $\text{R}^1 = \text{n-C}_4\text{H}_9$, $\text{R}^2 = \text{CO}_2\text{CH}_3$

1d: $\text{R}^1 = \text{CH}_3$, $\text{R}^2 = \text{CO}_2\text{CH}_3$

1e: $\text{R}^1 = \text{CH}_3$, $\text{R}^2 = \text{Ph}$

1f: $\text{R}^1 = \text{n-C}_3\text{H}_7$, $\text{R}^2 = \text{Ph}$

2a: $\text{R}^3 = \text{H}$

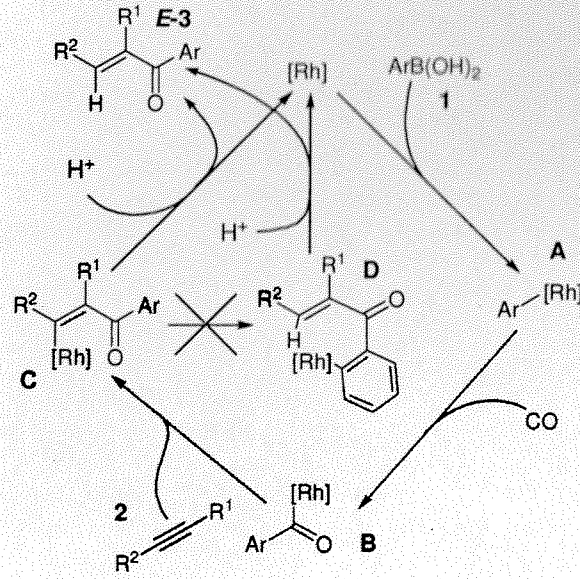
2b: $\text{R}^3 = \text{OCH}_3$

2c: $\text{R}^3 = \text{CH}_3$

No	%Ürün			
	1	2	3 ^a	3'
1	1b	2a	3ba (30)	-
2	1b	2b	3bb (57)	-
3	1b	2c	3bc (53)	-
4	1c	2b	3cb (64)	(<1) ^b
5	1d	2b	3db (54)	(2) ^b
6	1e	2a	3ea (42)	(3) ^b
7	1e	2b	3eb (74)	(6) ^b
8	1f	2b	3fb (41)	(13) ^a

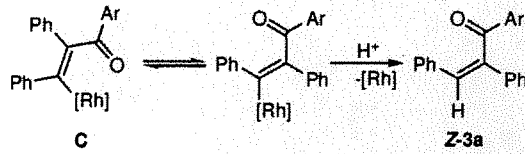
^a İzole ürün. ^b GK ile tespit edildi.

arilhidrojenin karşılıklı aktarımları (D) ve takibindeki aril bağlı rodyumun protodemetallasyonu son ürüne ulaşımında alternatif bir rota olabilirdi (2001). Ancak CO atmosferi altında 1b nin $\text{PhB}(\text{OH})_2\text{-d}_5$ ile tepkimesi bu olasılığı elimine etmiştir, çünkü karbonil bağlı fenil halkası üzerinde hiç bir proton girdisi ya da enon ürünün β -doymamış karbonu üzerine döteryum girdisi saptanamamıştır.



Şema 3. Enon oluşum mekanizması

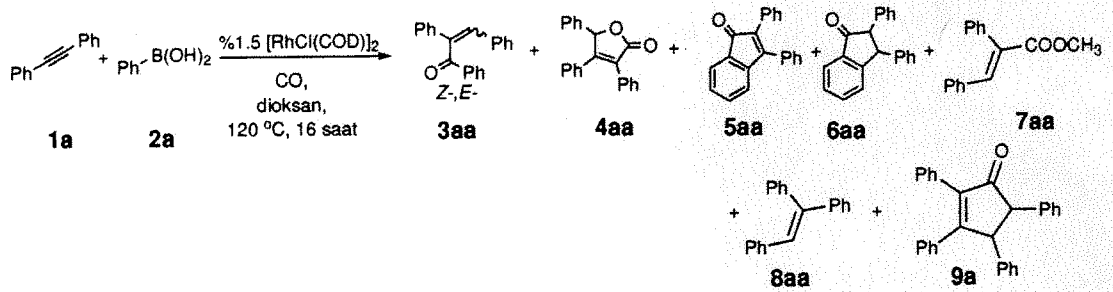
Ancak, yukarıda verilen mekanizma difenil asetilen ile gerçekleştirilen tepkimede neden **3a** ürünlerinin *E*- ve *Z*- izomerik karışımlar halinde elde edildiğini açıklayamamaktadır. α,β -Doymamış ketonların sentezi için uyguladığımız genel karbonilasyon metodu ile, ancak herhangi bir arilboronik asidin bulunmadığı ortamda *E-3ab* nin %25 oranında *Z*- izomerine dönüştüğü saptanmıştır. Bu sonuç diaril asetilenden oluşan *E*-enonların karbonilatif arilasyon tepkimeleri sırasında kısmen izomerize olabileceğini gösterse de Çizelge 8 de verilen *Z/E* oranlarını açıklayamamaktadır. Muhtemelen C ara ürünü de alkenil karbonlarının iki aril grubuna bağlanması nedeniyle genişlemiş konjugasyonunun yardımıyla izomerize olabilmektedir:



3.3. İndanon (6) ve İndenon (5) Oluşumları

Alkinlerin karbonilatif arilasyonunda diğer bir aşamada indenon (5) ve indanon (6) ürünlerinin seçimliliğinin artırılmasıdır. Bu amaçla gerçekleştirilen optimizasyon çalışmasının sonuçları Çizelge 10 da verilmiştir. $[\text{Rh}(\text{cod})\text{Cl}]_2$ kompleksinin katalizör olarak

kullanıldığı tepkime ortamında asidik karakter göstermeyen organoboron reaktiflerin kullanımı gerekmektedir (No 1-10). Tepkime sıcaklığı 80 °C yetersiz görünmektedir ve tepkimenin daha yüksek sıcaklıkta, 120 °C de gerçekleştirilmesi 5aa ve 6aa halkalaşma ürünlerinin miktarını arttırmıştır. Tepkime ek olarak direk arilasyon ürünü 8aa ve moleküller arası Pauson Khand tipi tepkime sonucu pentenon 9a tipi bir üründe oluşturmuştur. Denenen metanol, dioksan, toluen, THF ve DMF çözücülerinde en uygun olanı metanol olarak görünmektedir. Organoboron olarak KPhBF₄ bileşiği kullanıldığında [Rh(cod)Cl]₂, [Rh(C₂H₄)₂acac]₂, [Rh(C₂H₄)₂Cl]₂, [Rh(CO)₂Cl]₂, Rh(cod)₂BF₄ kompleksleri içerisinde en yüksek verim Rh(cod)₂BF₄ ile elde edilmiştir (No 11-15). Muhtemelen rodyumun katyonik formu aril-H bağı daha etkin olarak aktive etmektedir. Organoboron reaktifi olarak Ph₃(BO)₃ kullanılması 5aa ürün oluşumunu azaltmış ve bu azalış hidrojene karşıtı 6aa oluşumunun artışı ile karşılanmıştır (No 16,17). Daha düşük % 1 Rh oranında dahi tepkime veriminde düşüş olmamıştır (No. 18). CO basıncının 2 atmosfere artırılması tepkimenin ürün dağılımını etkilememiştir (No. 19).



İnda(e)non bileşikleri için saptanan optimum tepkime koşullarında difenilasetilen ve çeşitli arilboroksinler ile de tepkimeler gerçekleştirilmiştir (Çizelge 5). Ph₃(BO)₃ ile % 43 oranında indanon 6aa izole edilebilmiştir. İndenon 5aa oluşumu ise % 7 oranındadır. Kullanılan arilboroksinin *p*- ya da *m*- pozisyonunda metoksi grubu bulunduğu indanon oluşumunda düşüş gözlenmiştir (No. 2 ve 5). Ancak metil grubu bağlı olduğunda yine %50 oranına yakın toplam inda(e)non ürünleri oluşmuştur (No. 3 ve 4).

Çizelge 10. Difenil asetilenin organoboronlar ile karbonilatif arilasyonunda 5aa ve 6aa ürünlerinin seçimli oluşumuna tepkime koşullarının etkileri.^a

No	Katalizör	Organoboron	Çözücü	T, °C	Dönüşüm %	3aa	4aa	5aa	6aa	7a	8aa	9a
1	[Rh(cod)Cl] ₂	PhB(OH) ₂	CH ₃ OH	80	100	50	6	12	20	5	-	-
2	[Rh(cod)Cl] ₂	PhB(OH) ₂	Dioksan	80	52	11	7	2	9	1	-	-
3	[Rh(cod)Cl] ₂	PhB(OH) ₂	CH ₃ OH	120	100	32	4	11	34	<1	4	3
4	[Rh(cod)Cl] ₂	NaBPh ₄	CH ₃ OH	120	100	-	1	25	7	-	-	16
5	[Rh(cod)Cl] ₂	KPhBF ₄	CH ₃ OH	120	100	3	2	19	31	2	6	8
6	[Rh(cod)Cl] ₂ /PPh ₃	KPhBF ₄	CH ₃ OH	120	100	4	5	2	23	-	-	3
7	[Rh(cod)Cl] ₂	KPhBF ₄	Toluen	120	75	1	33	3	-	-	40	-
8	[Rh(cod)Cl] ₂	KPhBF ₄	THF	120	55	-	1	9	-	-	1	2
9	[Rh(cod)Cl] ₂	KPhBF ₄	DMF	120	100	-	4	12	1	1	7	21
10	[Rh(cod)Cl] ₂	KPhBF ₄	<i>o</i> -ksilen	120	100	1	4	7	3	-	59	3
11	[Rh(cod)OH] ₂	KPhBF ₄	CH ₃ OH	120	100	2	2	17	31	1	2	11
12	Rh(C ₂ H ₄) ₂ acac	KPhBF ₄	CH ₃ OH	120	100	2	3	24	26	-	1	11
13	[Rh(C ₂ H ₄) ₂ Cl] ₂	KPhBF ₄	CH ₃ OH	120	100	3	1	15	29	2	4	7
14	Rh(CO) ₂ Cl] ₂	KPhBF ₄	CH ₃ OH	120	100	4	2	18	24	3	1	9
15	Rh(cod) ₂ BF ₄	KPhBF ₄	CH ₃ OH	120	100	1	2	17	40	1	2	8
16	Rh(cod) ₂ BF ₄	Ph ₃ (BO) ₃ ^c	CH ₃ OH	120	100	11	1	8	56	-	9	3
17	Rh(cod) ₂ BF ₄	Ph ₃ (BO) ₃ ^{c,d}	CH ₃ OH	120	100	16	1	14	31	-	11	3
18	Rh(cod) ₂ BF ₄ ^e	Ph ₃ (BO) ₃ ^c	CH ₃ OH	120	100	5	-	8	55	-	9	3
19	Rh(cod) ₂ BF ₄ ^f	Ph ₃ (BO) ₃ ^c	CH ₃ OH	120	100	10	<1	4	55	-	7	3

^a%3 Rh, 1 mmol 1a, 3 mmol organoboron, 1 atm CO, ürünler gaz kromatografi yöntemi ile saptanmıştır. ^b% 6 PPh₃. ^c1 mmol fenil boroksün. ^d%10 Rh ^e % 1 Rh. ^f%1.5 Rh, 2 atm CO.

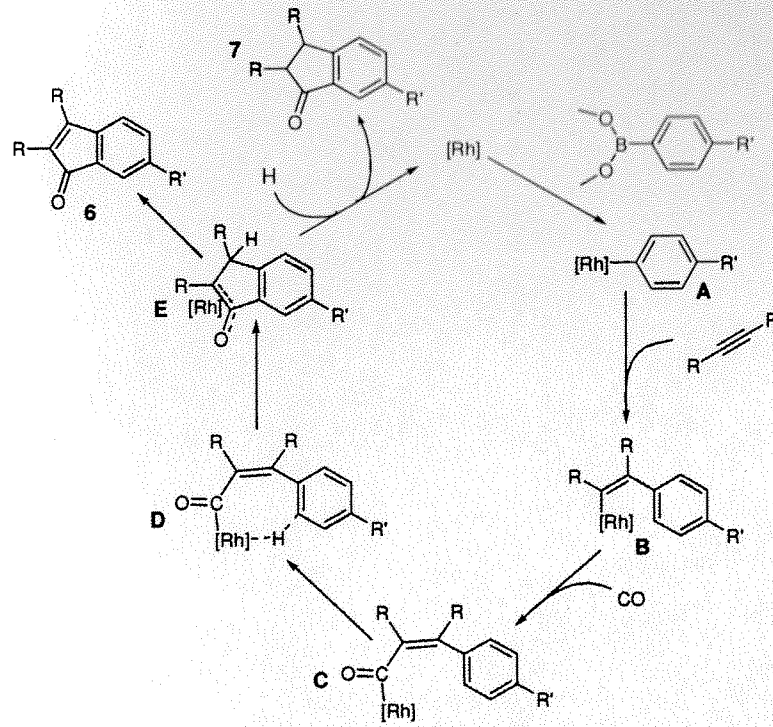
Bir elektron çekici grup olarak *p*- pozisyonunda triflorometil grubu bulunduğu tepkime sonunda direk arilasyon ürünü 1-(triflorometil)-4-((*E*)-1,2-difenilvinil)benzen oluşturmaktadır. Ayrıca dialkil sübtitüe 4-oktin ile de tepkime seçimli davranmamış ve kompleks karışım oluşmuştur. AM1 hesaplamaları indanon halka hidrojenlerinin dihedral açılarının *cis*-izomerik yapısında <1° iken *trans*-izomerik yapısında ≈124° olduğunu göstermektedir. ¹H NMR da saptanan halka hidrojenlerinin etkileşme sabitlerine göre oluşan indanonlar *trans*-stereoizomerik yapısında olmaları gerekmektedir.

Çizelge 11. Difenilasetilenin arilboroksinler ile CO gazı altında tepkimeleri ile indenon (5) ve indanon (6) oluşumu.^a

No	Arilboroksin	% Ürün				
		3a ^c	5a ^c	6a ^b	8a ^c	9a ^c
1	Ph ₃ (BO) ₃	3aa (10)	5aa (7)	6aa (43)	8aa (9)	3
2	(<i>p</i> -OCH ₃ Ph) ₃ (BO) ₃	3ab (15)	5ab (7) ^a	6ab (26)	8ab (3)	1
3	(<i>p</i> -CH ₃ Ph) ₃ (BO) ₃	3ac (25)	5ac (8)	6ac (41)	8ac (7)	6
4	(<i>m</i> -CH ₃ Ph) ₃ (BO) ₃	3ad (6)	5ad (4)	6ad (43)	8ad (15)	6
5	(<i>m</i> -OCH ₃ Ph) ₃ (BO) ₃	3ae (< 1)	5ae (< 1)	6ae (24)	8ae (13)	< 1

^a% 1.5 Rh, 1 mmol 1a, 3 mmol organoboron, 1 atm CO. ^bİzole ürün. ^cGaz kromatografi yöntemi ile saptandı.

Oluşan indena(e)non yapıları furanon ve enon oluşum tepkimlerine karşıt olarak alkinin aroilleme adımı ile değil arillenmesi ile oluşmaktadır: Oluşan arilrodyum (A) alkine katılmakta, oluşan alkenil rodyum (B) karbonmonoksite katılarak α,β -doymamış açil rodyum kompleksini oluşturmaktadır (C). Rodyumun *o*-hidrojenini aktive edip (D) halka kapanması sonrası (E) β -hidrojen eliminasyonu indenon (5) yapısını hidrojenlenmesi ise indanon (6) yapısını oluşturmaktadır (Şema 4).



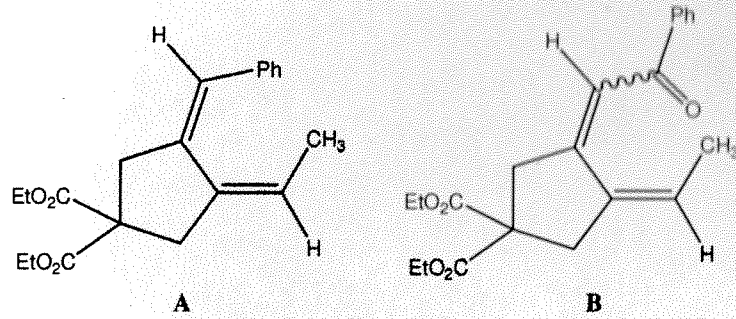
Şema 4. İnde(a)non oluşum mekanizması.

3.3. Diğer Tepkimeler

Alkinlerin karbonilatif arilasyon tepkimeleri Pd(II) katalizörü ile de denenmiştir. Pd kaynağı olarak Pd(O₂CCH₃)₂ kullanılarak formik asitin (Pd(II) oluşumu için) bulunduğu ortamda metanol ve toluen çözücü ortamlarında çeşitli CO basıncında tepkimeler gerçekleştirilmiştir. Ancak denemeler aroilatif ürünler vermemiştir.

Diğer bir denememizde yukarıda belirtilen yöntemler ile dietil-2,2-di(2-butinil)malonat bileşiği ile de Rh katalizli karbonilatif arilasyon tepkimeleri de gerçekleştirilmiştir.

Çözücü olarak metanol kullanıldığında aşağıda yapıları gösterilen her iki arilatif A ve aroilatif halkalaşma B ürünleri oluşmuştur. Amaçlanan B ürününün yeterli seçimliliğe ulaşması henüz sağlanamamıştır. Muhtemelen direk arilatif tepkime diğer tepkimeden çok daha aktiftir.



4. Sonuç

Alkin ve organoboron karışımlarının katalizör olarak bir rodyum kompleksinin bulunduğu ortamda, CO gazı altında tepkimleri gerçekleştirilmiştir. Yöntem başlıca 5-aril-2(5H)-furanon, α,β -doymamış keton ve indanon ürünleri vermektedir. Çeşitli tepkime koşulları ayarlanarak yöntemin ürün seçimliliği değiştirilebilmekte atom ekonomik bir yöntem ortaya sunmaktadır.

Kaynaklar

- AKSIN Ö., Dege N., Artok, L., Türkmen, H., Çetinkaya, B., *Chem. Commun.*, 3187, (2006).
 BLACK W.B., Lutz R.E., *J. Am. Chem. Soc.*, 75, 5990, (1953).
 CHOCHOIS H., Sauthier M., Maerten E., Castanet Y., Mortreux A., *Tetrahedron*, 62, 11740, (2006).
 COPÉRET C., Sugihara T., Wu G., Shimoyama I., Negishi E.-i., *J. Am. Chem. Soc.*, 117, 3422, (1995).
 CRAMER R., Mcleverty J.A., Bray J., *Inorg. Synth.* 15, 14, (1974).
 DEALUNAY, J., Orliac-Lemoing a., Simonet J., *Tetrahedron*, 44, 7089, (1988).
 DHEUR J., Sauthier M., Castanet Y., Mortreux A., *Adv. Synth. Catal.*, 349, 2499, (2007).
 DUKE, P.J., Boykin D.W.Jr., *J. Org. Chem.*, 37, 1436, (1972).
 FAGNAU K., Lautens M., *Chem. Rev.* 103, 169 (2003).
 FROST, C.G., Wadsworth, K.J., *Chem. Commun.*, 2316, (2001).
 GIARDINO G., Crabtree R.H., *Inorg. Synth.*, 19, 218, (1979).
 GOPIDAS K. R., Lohray B.B., Rajadurai S., Das D.K., George, M.V., *J. Am. Chem. Soc.*, 52, 2831, (1987).

GUAN J.T., Meng T.Q., Yu G.A., Liu S.L., *Tetrahedron Lett.*, 48, 7129, (2003).

HAYASHI T., Inoue K., Taniguchi N., Ogasawara M., *J. Am. Chem. Soc.*, 123, 9918, (2001).

HAYASHI T., Yamasaki K., *Chem. Rev.*, 103, 2829, (2003).

HUANG Y., Alper H., *J. Org. Chem.*, 56, 4534, (1991).

IMAI M., Tanaka M., Tanaka K., Yamamoto Y., Imai-Ogata N., Shimowatari M., Nagumo S., Kawahara N., Suemune H., *J. Org. Chem.*, 69, 1144, (2004).

IMAI M., Tanaka M., Nagumo S., Kawahara N., Suemune H., *J. Org. Chem.*, 72, 2543, (2007).

JUN C.-H., Lee H., Hong J.-B., Kwon B.I., *Angew. Chem. Int. Ed.*, 41, 2146, (2002).

KOKUBO K., Matsumasa K., Miura M., Nomura M., *J. Org. Chem.*, 62, 4564, (1997).

KOKUBO K., Matsumasa, K., Nishinaka Y., Miura M., Nomura M., *Bull. Chem. Soc. Jpn.*, 72, 303, (1999).

KUŞ M., Artok-Aksin Ö., Ziyank F., Artok, L., *Synlett*, 2587, (2008).

LAUTENS M., Roy A., Fukuoka K., Fagnou K., Martín-Matute B., *J. Am. Chem. Soc.*, 123, 5358, (2001).

LAUTENS M., Dockendorff C., Fagnou K., Malicki A., *Org. Lett.*, 4, 1311, (2002).

MA S., Shi Z. *Chem. Commun.*, 540, (2002).

MATSUDA T., Makino M., Murakami M., *Org. Lett.*, 6, 1257, (2004).

MENARD F., Weise C. F., Lautens M., *Org. Lett.*, 9, 5335, (2007).

MITTAL S., Durani S., Kapil R.S., *J. Med. Chem.*, 28, 492, (1985).

MIURA T., Shimada M., Murakami M., *Angew. Chem., Int. Ed.*, 44, 7598, (2005).

MOXHAM G.L., Randell-Sly H.E., Brayshaw S.K., Woodward R.L., Weller A.S., Willis M.C., *Angew. Chem. Int. Ed.*, 45, 7618, (2006).

OGUMA K., Miura M., Satoh T., M. Nomura, *J. Am. Chem. Soc.*, 122, 10464, (2000).

OGUMA K., Miura M., Satoh T., Nomura, M., *J. Organomet. Chem.*, 648, 297, (2002).

PADWA A., Brookhart T., Dehm D., Wubbels G., *J. Am. Chem. Soc.*, 100, 8247, (1978).

PADWA A., Dehm D., *J. Org. Chem.*, 40, 3139, (1975).

ROESCH K.R., Larock, R.L., *J. Org. Chem.*, 66, 412, (2001).

SAKAI M., Hayashi H., Miyaura N., *Organometallics*, 16, 4229, (1997).

SAKAI M., Ueda M., Miyaura N., *Angew. Chem., Int. Ed.*, 37, 3279, (1998).

SAUTHIER M., Castanet Y., Mortreux A. *Chem. Commun.*, 1520 (2004).

SHINTANI R., Tsurusaki A., Okamoto K., Hayashi T., *Angew. Chem., Int. Ed.*, 44, 3909, (2005a)

- SHINTANI R., Duan W.-L., Nagano T., Okada A., Hayashi T., *Angew. Chem., Int. Ed.*, **44**, 4614, (2005b).
- STEMMLER R.T., Bolma C., *Adv. Synth. Catal.*, **349**, 1185, (2007).
- TANAKA K., Tanaka M., Suemune H., *Tetrahedron Lett.*, **46**, 6053, (2005).
- TANAKA K., Shibata Y., Suda T., Hagiwara Y., Hirano M., *Org. Lett.*, **9**, 1215, (2007).
- UEURA K., Satoh T., Miura M., *Org. Lett.*, **7**, 2229, (2005).
- USON R., Oro L.A., Cabera J.A., *Inorg. Synth.*, **231**, 126, (1985).
- Van den HOVEN B.G., El Ali B., Alper H., *J. Org. Chem.*, **65**, 4131, (2000).
- VIZER S.A., Yerzhanov K.B., Al Quntarb A.A.A., Dembitsky V.M., *Tetrahedron*, **60**, 5499, (2004) ve içerdği referanslar.
- YONEDA E., Zhang S.-W., Zhou D.-Y., Onitsuka K., Takahashi S., *J. Org. Chem.*, **68**, 8571, (2003).
- WILLIS M.C., Randell-Sly H.E., Woodward R.L., Steven J., McNally S.J., Currie G.S., *J. Org. Chem.*, **71**, 5291, (2006).

Ekler:

Sentezlenen Ürünlerin ^1H MR ve ^{13}C NMR Spektrumları



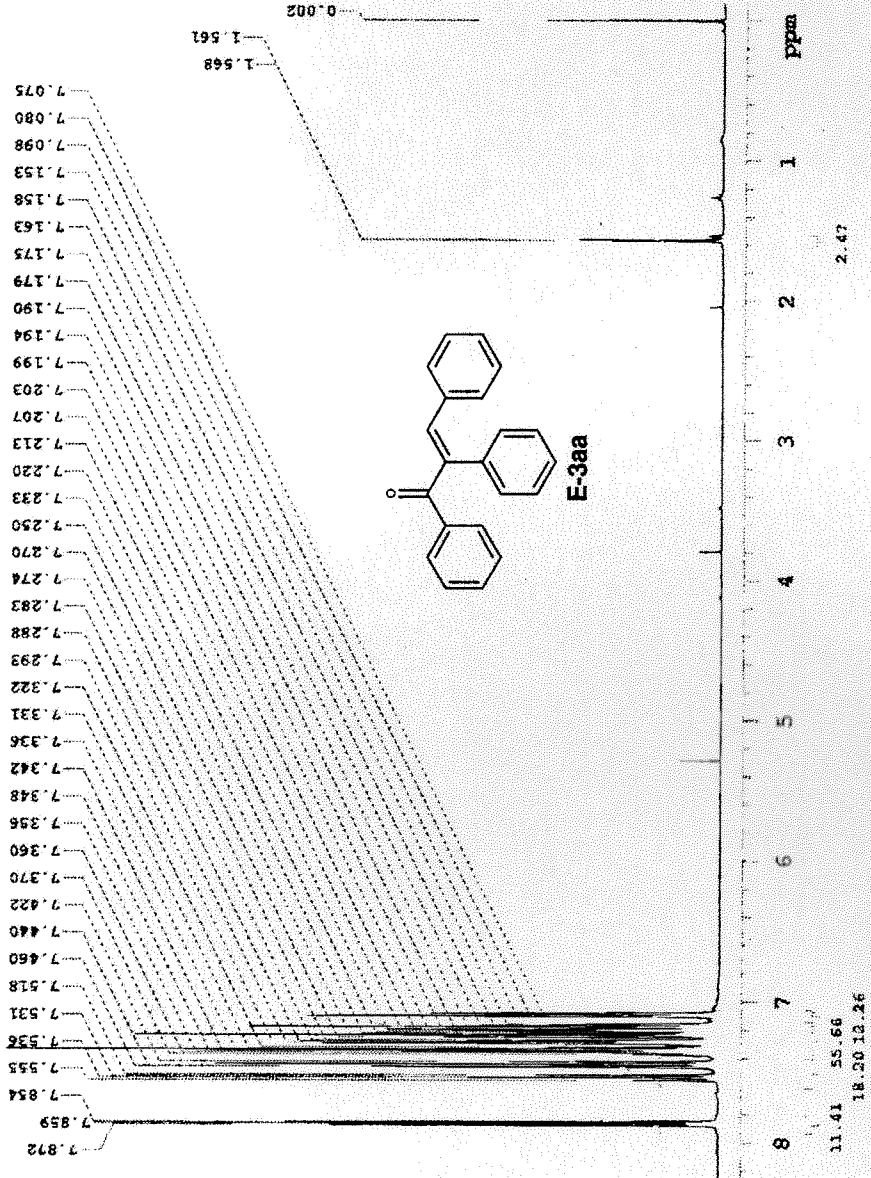
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20 KASIM 2007

Sample Name:
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Archive directory:
Sample directory:
Fidfile: Proton

Pulse Sequence: Proton (s2pul)
Solvent: cdcl3
Data collected on: Nov 20 2007

Temp. 25.0 C / 298.1 K
Operator: wslxupl
VARIAN-400 "mpc400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.5219926 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min 24 sec



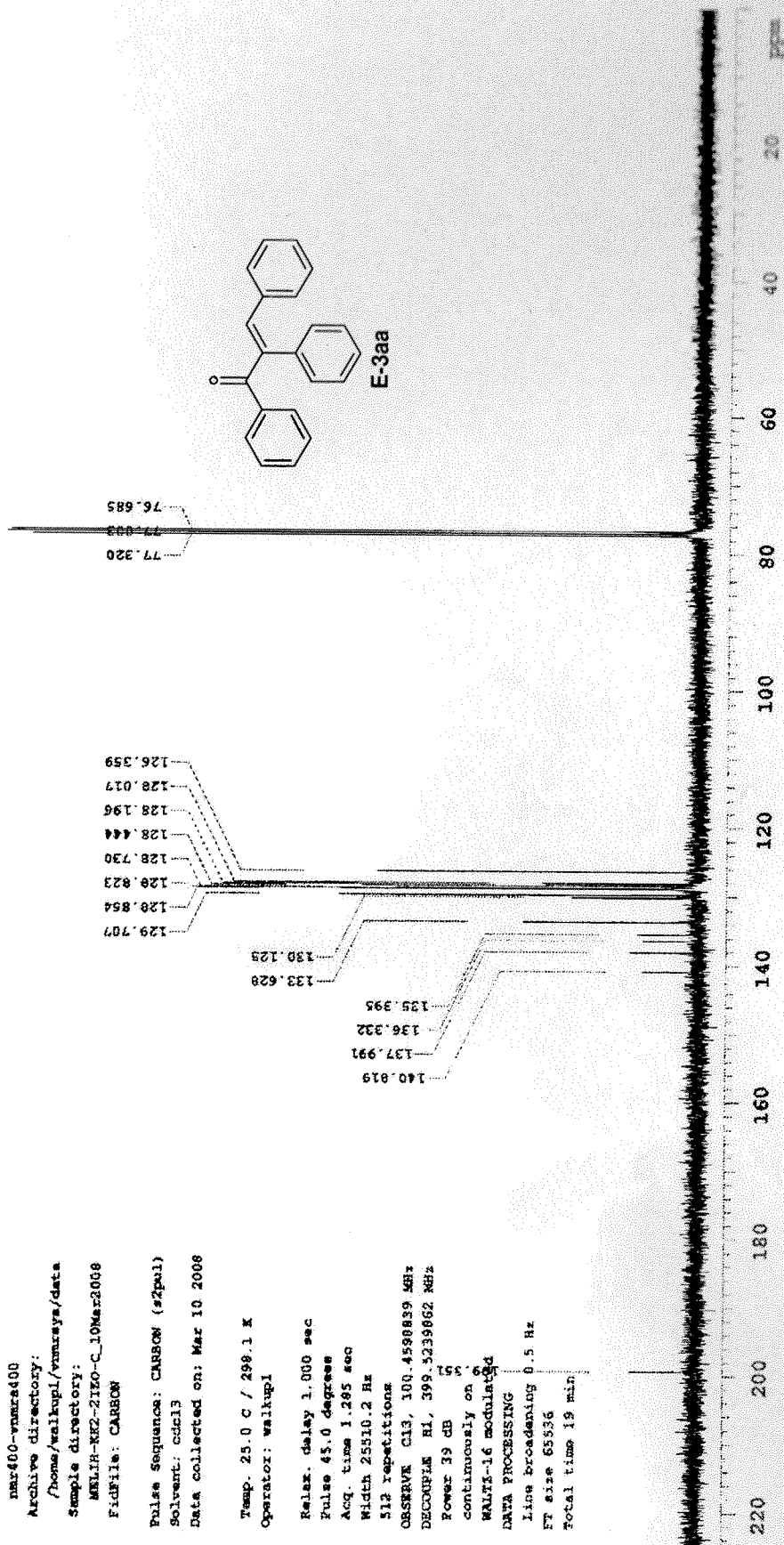
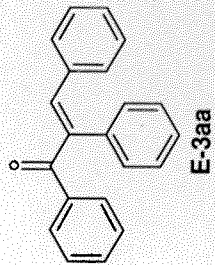
VELIH-KK2-2120-C

Sample Name:
 VELIH-KK2-2120-C
 Data Collected on:
 mar40-vmr400
 Archive directory:
 /home/walkup/vmr400/data
 Sample directory:
 VELIH-KK2-2120-C_10Mar2008
 Fidfile: CARBON

Pulse Sequence: CARBON (sZpol)
 Solvent: cdc13
 Data collected on: Mar 10 2008

Temp. 25.0 C / 299.1 K
 Operator: walkup

Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.285 sec
 Width 25510.2 Hz
 512 repetitions
 OBSERVE RL, 100.4590839 MHz
 DECOUPLE RL, 399.5239062 MHz
 Power 39 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 65536
 Total time 19 min



MELIH
12 KASIM 2007

Sample Name:
KKGK3 12 11 2007
Archive directory:
Sample directory:
Fidfile: Proton

Pulse Sequence: Proton (s2pul)
Solvent: cdcl3
Data collected on: Nov 12 2007

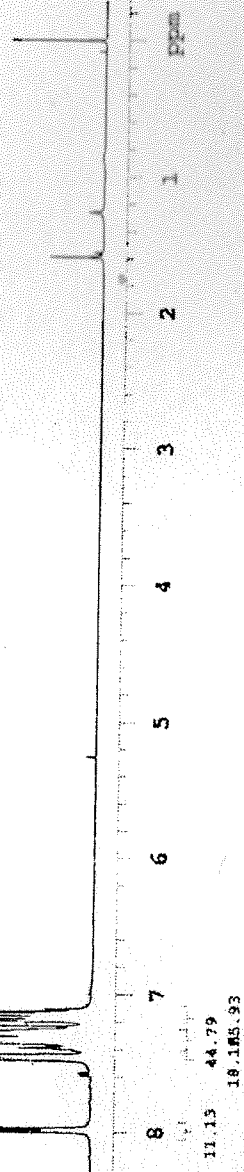
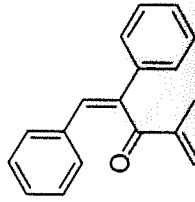
Temp. 23.0 C / 296.1 K
Operator: walkup1
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Pulse 45.0 degrees
Acq. time 2.049 sec
Width 6410.3 Hz
8 repetitions

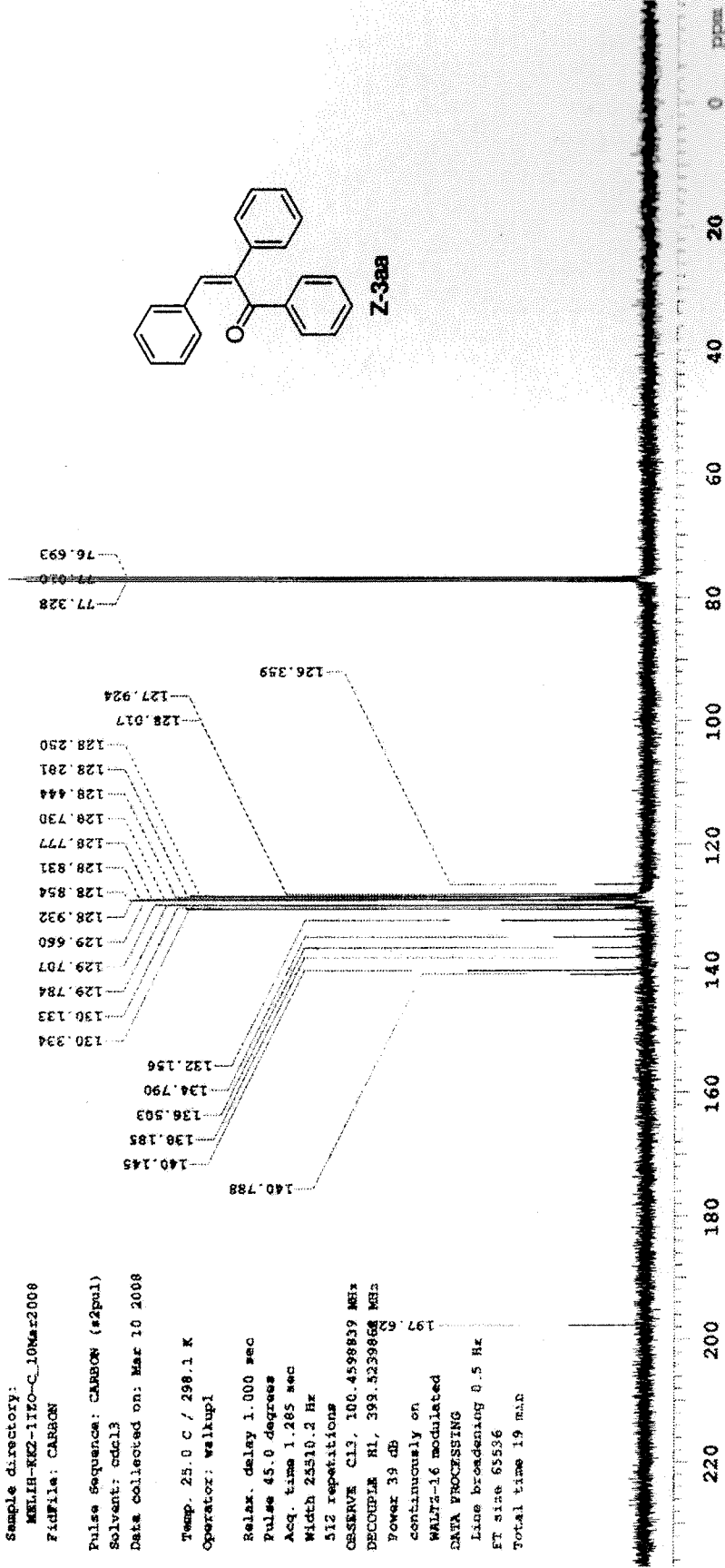
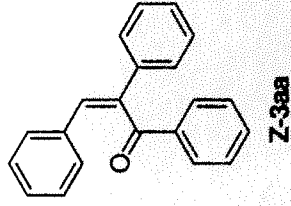
OBSERVE H1, 399.522000 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min 24 sec



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7.978
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7.473
7.469
7.463
7.456
7.451
7.449
7.442
7.439
7.435
7.357
7.354
7.350
7.345
7.338
7.333
7.329
7.323
7.318
7.314
7.297
7.294
7.285
7.279
7.230
7.192
7.187
7.169
7.166
7.154
7.150
7.143
7.139
7.126



11.15 44.79
18.165.93



MELIH-KK2-1120-C

Sample Name:
 MELIH-KK2-1120-C
 Data Collected on:
 nmr400-vmrns400
 Archive directory:
 /home/walkupl/vmrns400/data
 Sample directory:
 MELIH-KK2-1120-C_10Mar2008
 FidFile: CARBON

Pulse Sequence: CARBON (s2pol)
 Solvent: cdcl3
 Date collected on: Mar 10 2008

Temp: 25.0 C / 298.1 K
 Operator: walkupl

Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.285 sec
 Width 25510.2 Hz
 512 repetitions

OBSERVE CH3, 100.4598839 MHz
 DECOUPLE H1, 399.5239666 MHz

Power 39 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 65536
 Total time 19 min



KK3-FS134154
18 APRILK 2007

Sample Name:
KK3-FS134154-18APRILK2007
Archive directory:

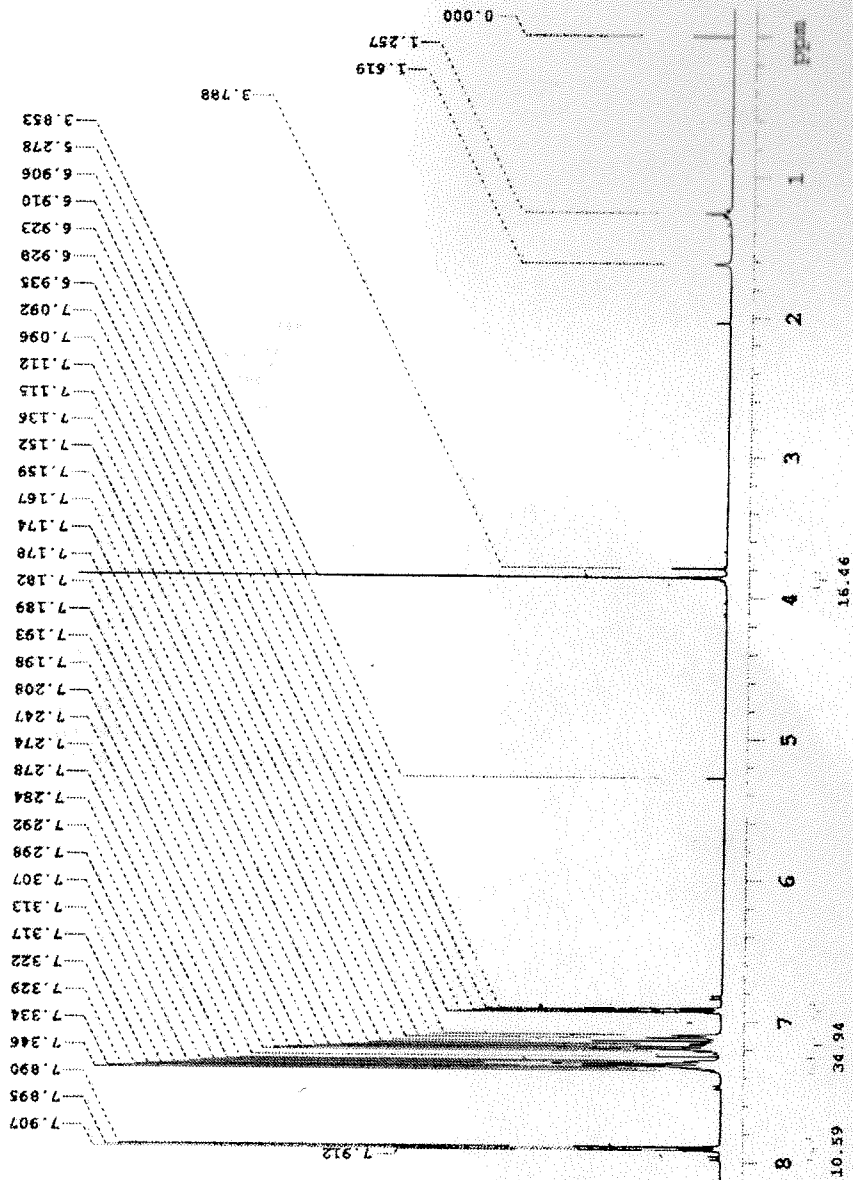
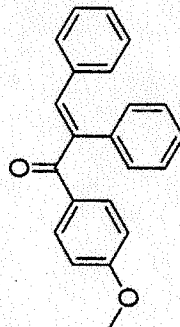
Sample directory:
Fidfile: Proton

Pulse Sequence: Proton (a2pul)
Solvent: cdcl3
Data collected on: Dec 18 2007

Operator: walkupl
VMMS-400 "nmr400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 6410.3 Hz
8 repetitions

OBSERVE H1, 399.521922 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min 24 sec



KK3-FS134154
18 APRILK 2007

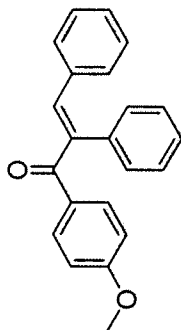
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KK3-FS134154-18APRILK2007
Archive directory:

Sample directory:
FidFile: Carbon

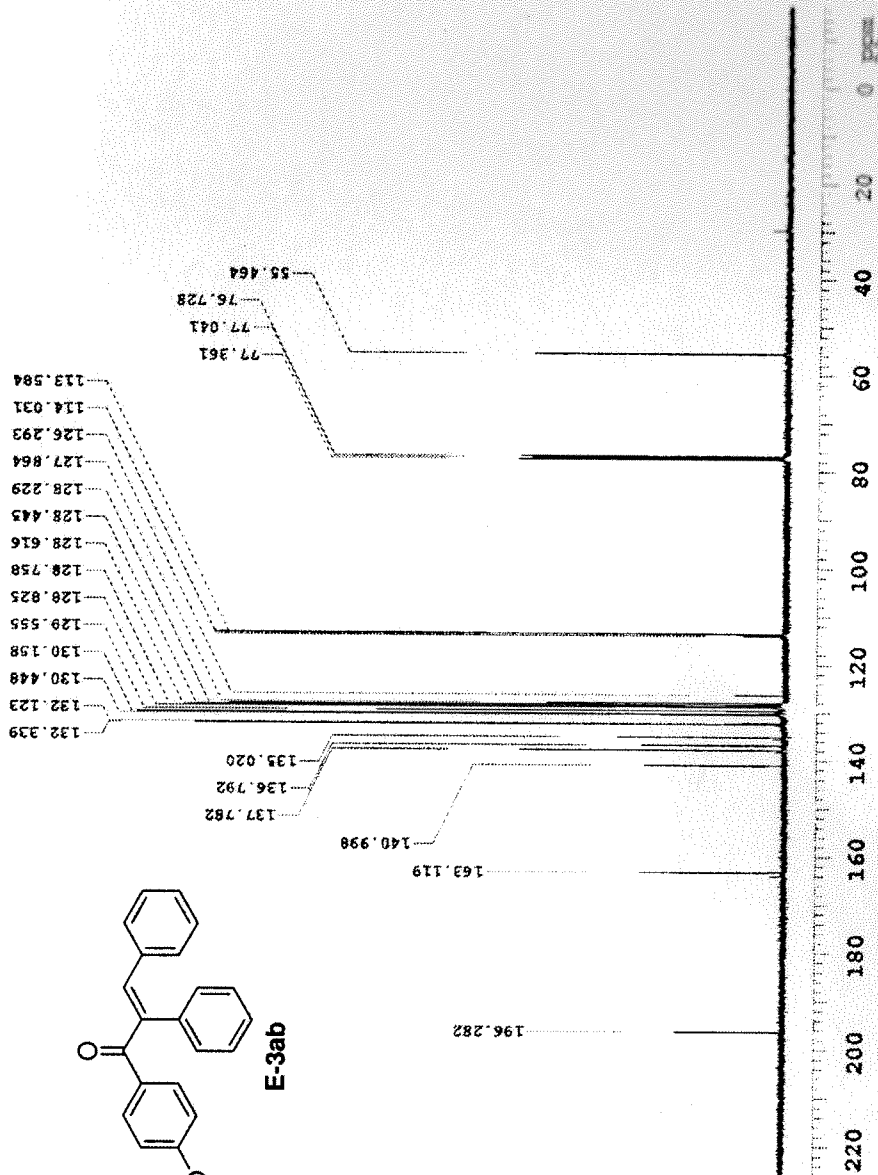
Pulse Sequence: Carbon (s2pul)
Solvent: cdcl3
Data collected on: Dec 18 2007

Operator: walkupl
VNMR-400 "nmr400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
512 repetitions
OBSERVE C13, 100.4598839 MHz
DECOUPLE H1, 399.5239865 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 0 min. 24 sec



E-3ab





MR-KK3R0100
23 kasim 2007

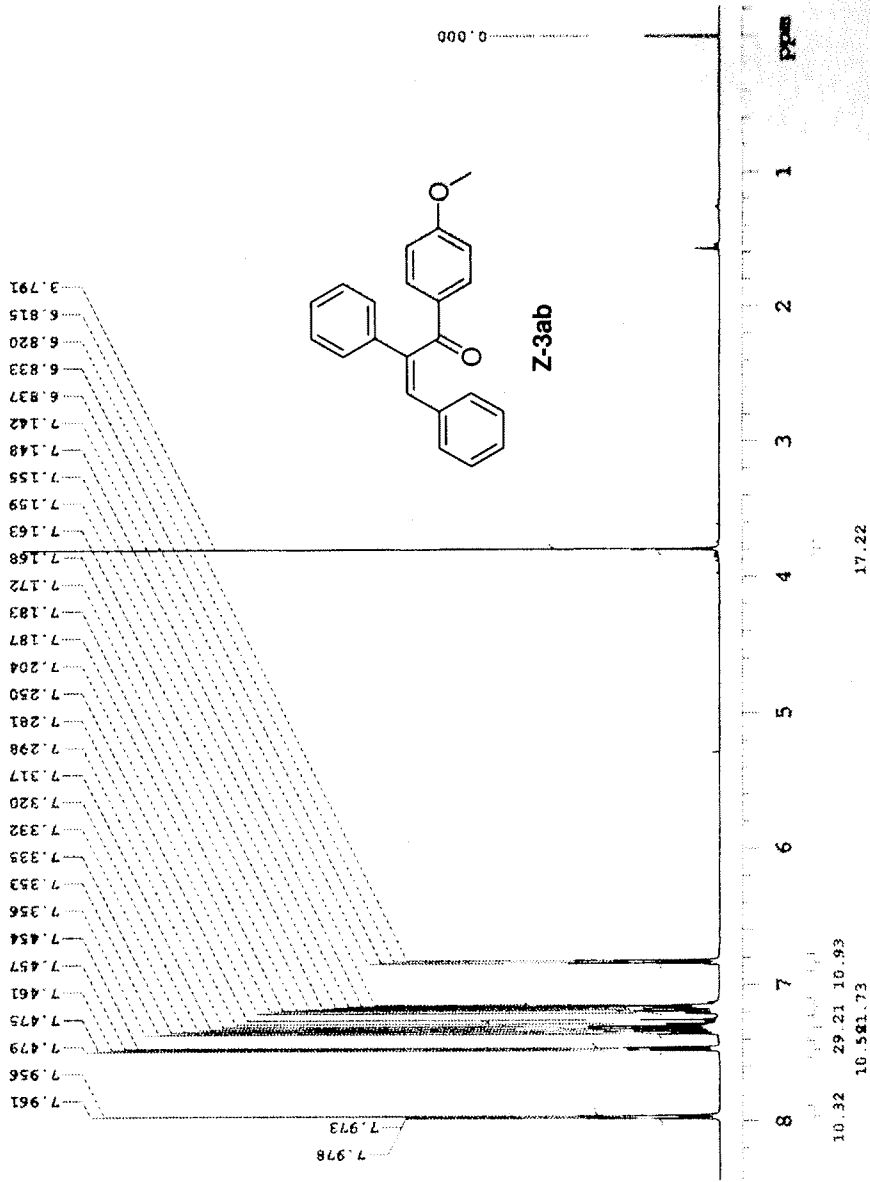
Sample Name:
MR-KK3R0100-23kasim2007
Archive directory:

Sample directory:
FidFile: Proton

Pulse Sequence: Proton (s2pul)
Solvent: cdcl3
Data collected on: Nov 23 2007

Temp. 25.0 C / 298.1 K
Operator: waikupl
VNMRS-400 "nmr400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 6410.3 Hz
8 repetitions
OBSERVE E1, 399.5219914 MHz
DATA PROCESSING
Line broadening 0.2 Hz
Ft size 65536
Total time 0 min 24 sec



MK-KK3K80100
MK-KK3K80100
23 kasim 2007

Sample Name:
MK-KK3K80100-23kasim2007
Archive directory:

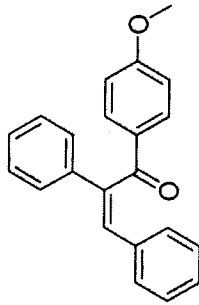
Sample directory:

FidFile: Carbon

Pulse Sequence: Carbon (*2pul)
Solvent: cdcl3
Data collected on: Nov 23 2007

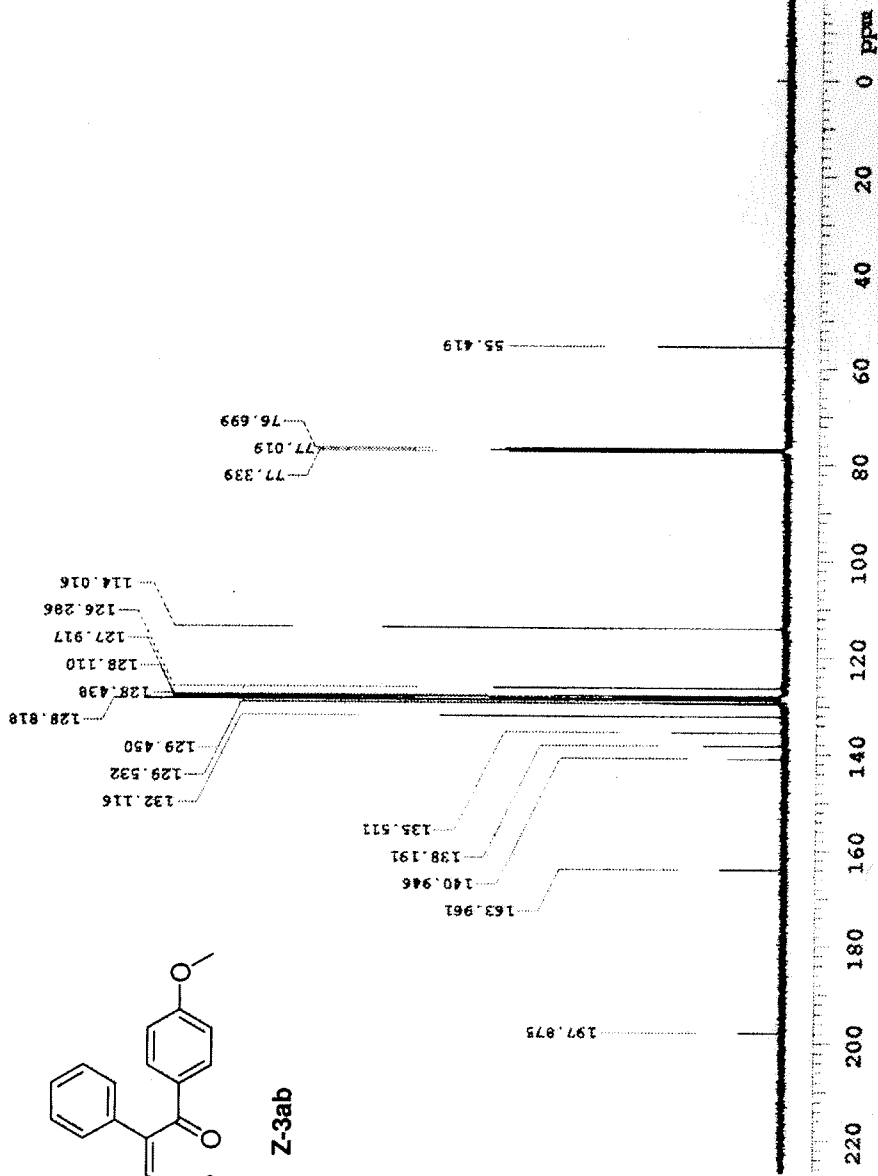
Temp. 25.0 C / 298.1 K
Operator: walkupl
VNMR3-400 "nmr400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24309.6 Hz
S12 repetitions
OBSERVE C13, 100.4598839 MHz
DECOUPLE H1, 399.5239865 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 0 min 24 sec



Z-3ab

VARIAN





ME-KK5-100
2. 140MR
22 OCAK 2008

Sample Name:
ME-KK5-86100-22OAK2008
Archive directory:

Sample directory:

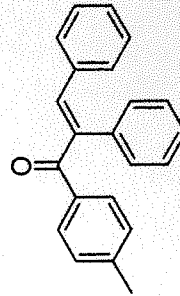
FidFile: Proton_Minaw

Pulse Sequence: Proton_Minaw (s2pul)
Solvent: cdcl3
Data collected on: Jan 22 2008

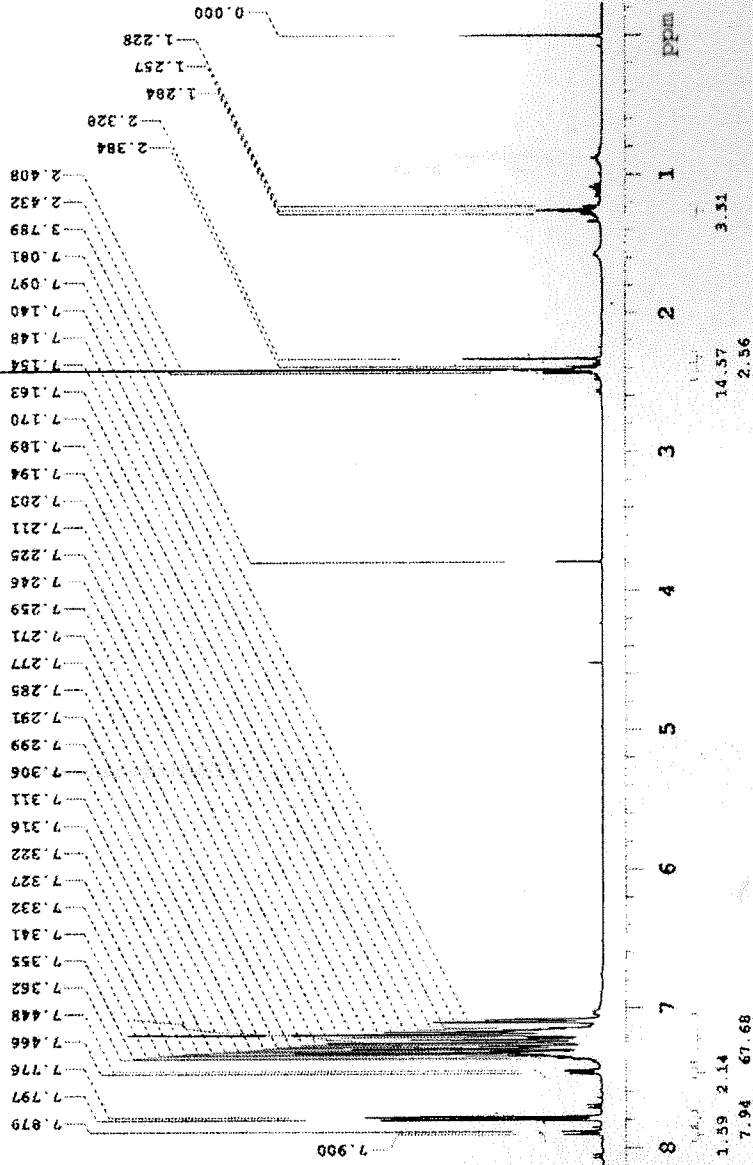
Temp. 25.0 C / 298.1 K
Operator: walkup1
VNMRS-400 "nmr400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 4194.6 Hz
8 repetitions

OBSERVE H1, 399.521922 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min 24 sec



E-3ac





VARIAN

MR-KK5-100
MR-KK5-100
2. IZOWER
22 CCAN 2008

Sample Name:
MR-KK5-06100-22CCAN2008
Archive directory:

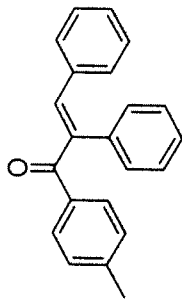
Sample directory:

Fidfile: Carbon

Pulse Sequence: Carbon (*2pul)
Solvent: cdc13
Data collected on: Jan 22 2008

Temp. 25.0 C / 299.1 K
Operator: walkupl
VNAME: 600 *rnr400*

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24509.0 Hz
512 repetitions
OBSERVE C13, 100.4598839 MHz
DECOUPLE H1, 399.5239863 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 0 min 24 sec



E-3ac

130.255
130.198
130.061
130.024
129.867
129.733
129.718
129.622
129.510
129.257
129.041
128.996
128.825
128.765
128.751
128.646
128.438
128.229
128.133
128.073
127.939
127.864
127.835
126.718
126.316
77.339
77.019
76.706

139.100
136.665
135.340
134.923

140.939
143.016

197.302

21.631

21.721

200 180 160 140 120 100 80 60 40 20 ppm



MK-KK55172
I. IZOMER
21 OCT 2008

Sample Name:
MK-KK55172-21OCT2008
Archive directory:

Sample directory:

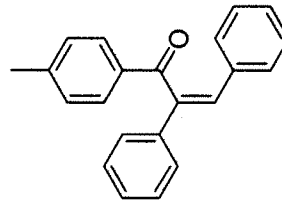
FidFile: Proton_Minaw

Pulse Sequence: Proton_Minaw (s2pul)
Solvent: cdcl3
Data collected on: Jan 21 2008

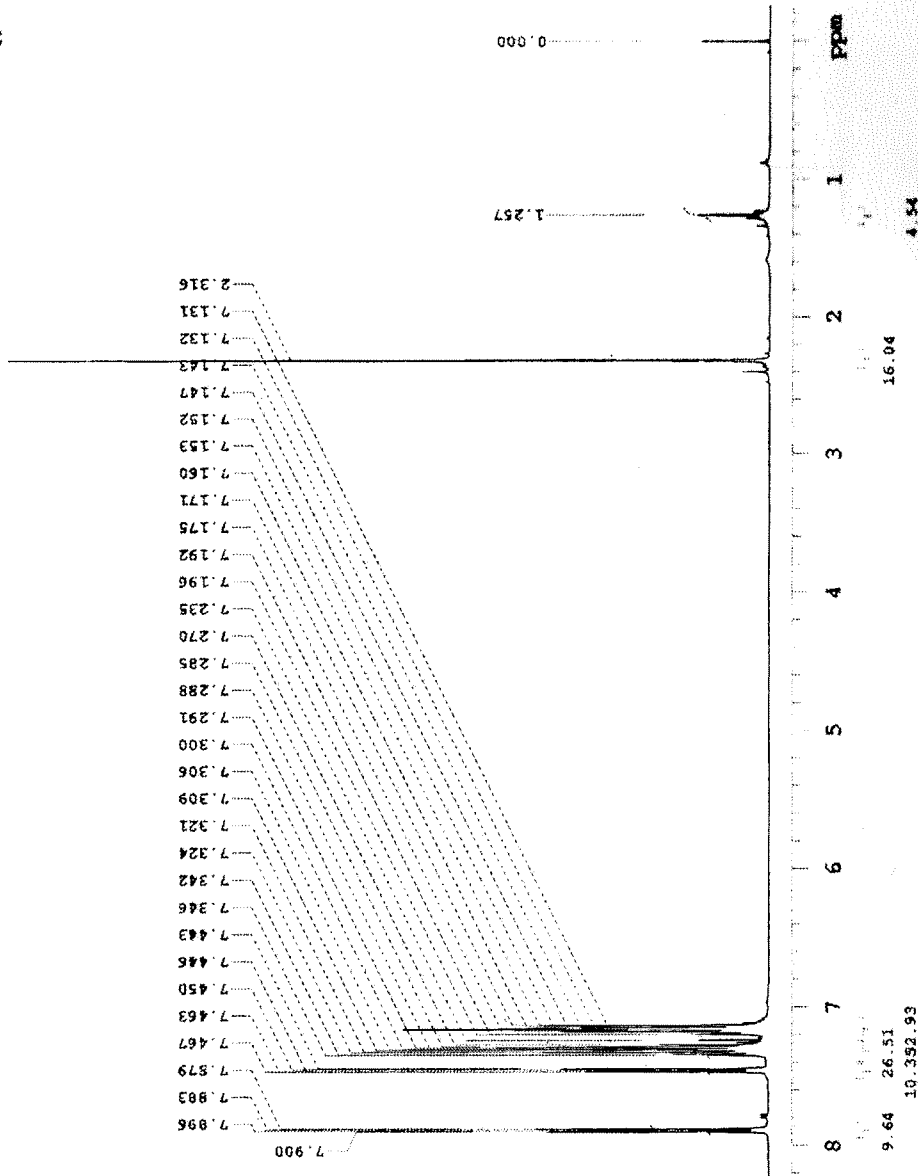
Temp. 25.0 C / 298.1 K
Operator: walkup1
VPMAS-400 "mr400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 4166.7 Hz
8 repetitions

OBSERVE H1, 399.521969 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min 24 sec



Z-3ac



MK-KK5172
 MK-KK5172
 I. IZOMER
 21 OCAK 2008

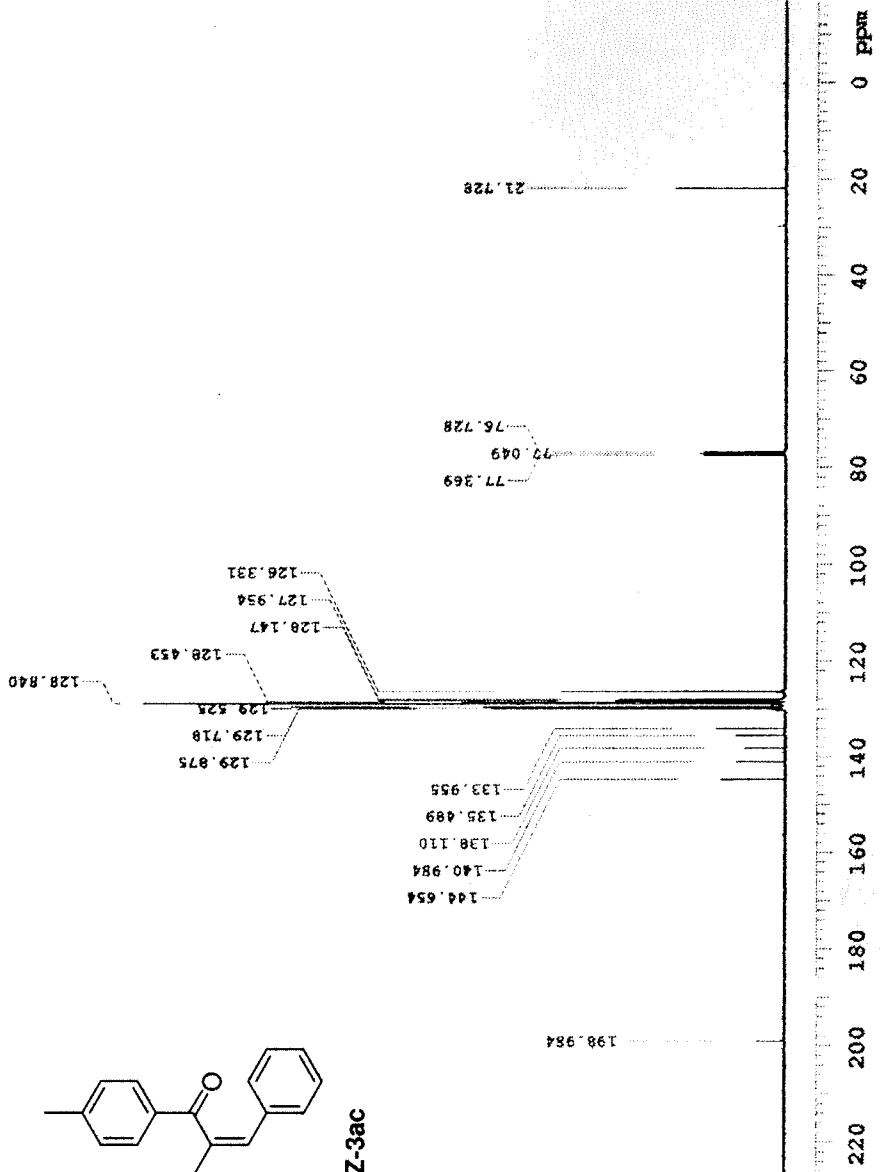
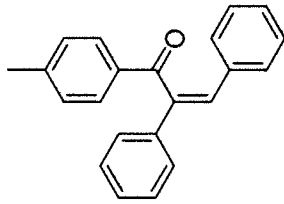
Sample Name:
 MK-KK5172-21OCAK2008
 Archive directory:

Sample directory:
 Fidfile: Carbon

Pulse Sequence: Carbon (s2pul)
 Solvent: cdcl3
 Data collected on: Jan 21 2008

Temp.: 25.0 C / 298.1 K
 Operator: walkupl
 VNMRS-400 "nmr400"

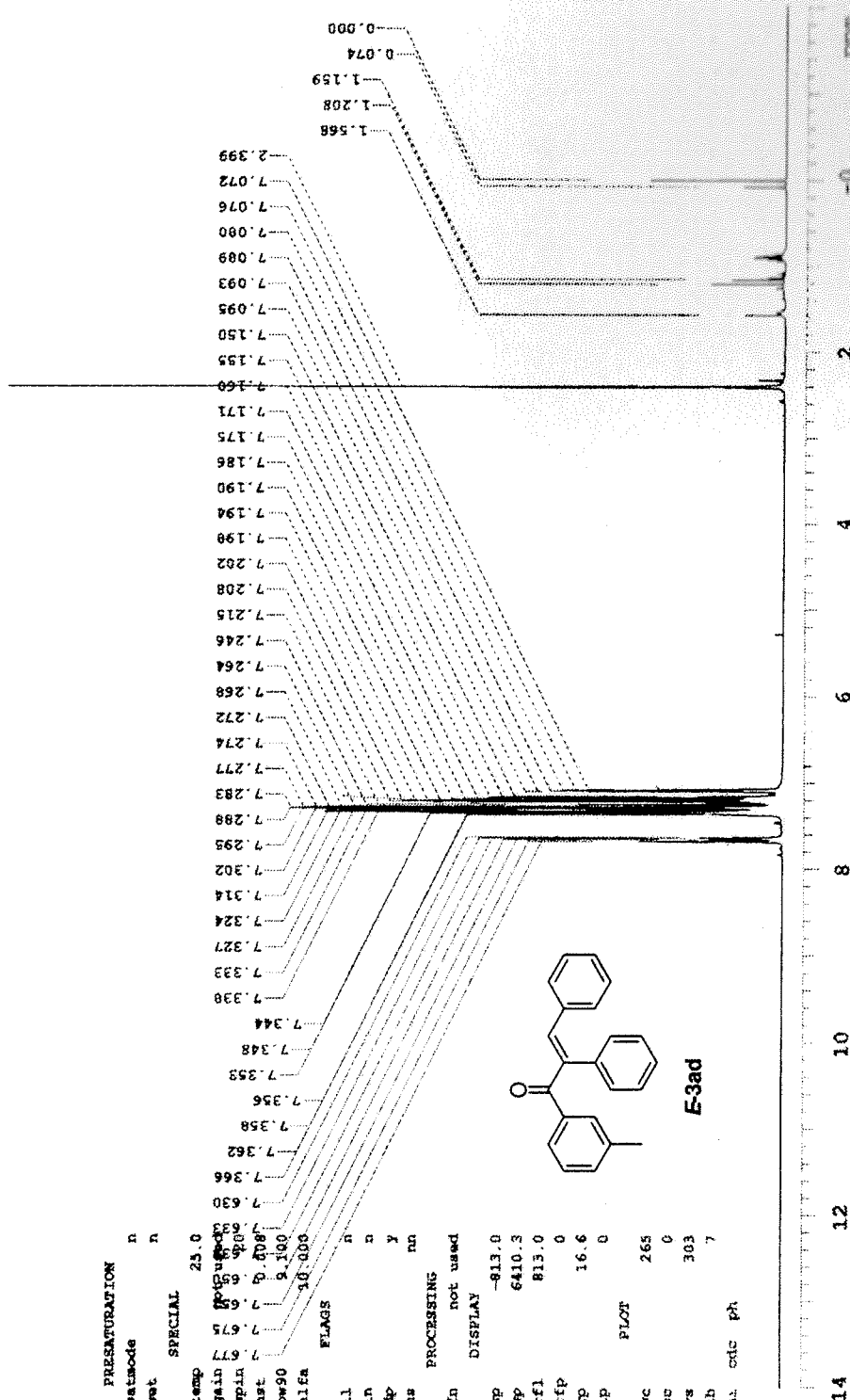
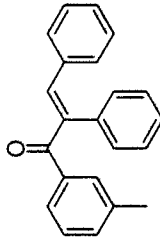
Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 24509.8 Hz
 512 repetitions
 OBSERVE C13, 100.4598839 MHz
 DECOUPLE H1, 399.5239865 MHz
 Power 39 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 65536
 Total time 0 min 24 sec



ME-KK12-2120

exp1 PROTON

SAMPLE PRESENTATION
 date Feb 16 2088 satmode n
 solvent cdcl3 wet n
 file /home/walshpl~ SPECIAL
 /varrays/data/ARTO~ temp 23.0
 K GROUP/HELLH/MK-K gain 1000000
 KI2-2120_16Feb2088~ spin 9 9 90
 /PROTON_01.fid hst 7 7 677
 ACQUISITION pw90 9.300
 6410.3 aifa 10.000
 at 2.556 FLAGS
 np 32768 il n
 fh 4000 in n
 bs 32 dp y
 dl 1.000 bs nn
 nt 8 PROCESSING
 ct 8 fn not used
 TRANSMITTER HI sp DISPLAY
 tn -813.0
 sfreq 399.524 mp 6410.3
 tof 399.5 rfl 813.0
 tpwr 61 rfp 0
 pw 4.550 rp 16.6
 DECOUPLER lp 0
 dn C13 PLOT
 dof 0 wc 265
 dm non sc 0
 decvave W40_HCN5mm vs 303
 dpr 34 tb 7
 dnuf 29412 a1 cdc ph



10.2411.73

14

12

10

8

6

4

2

0

PPM

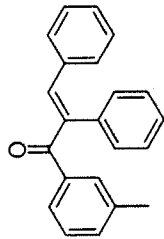
MELIH-KK12-2120

exp1 CARBON

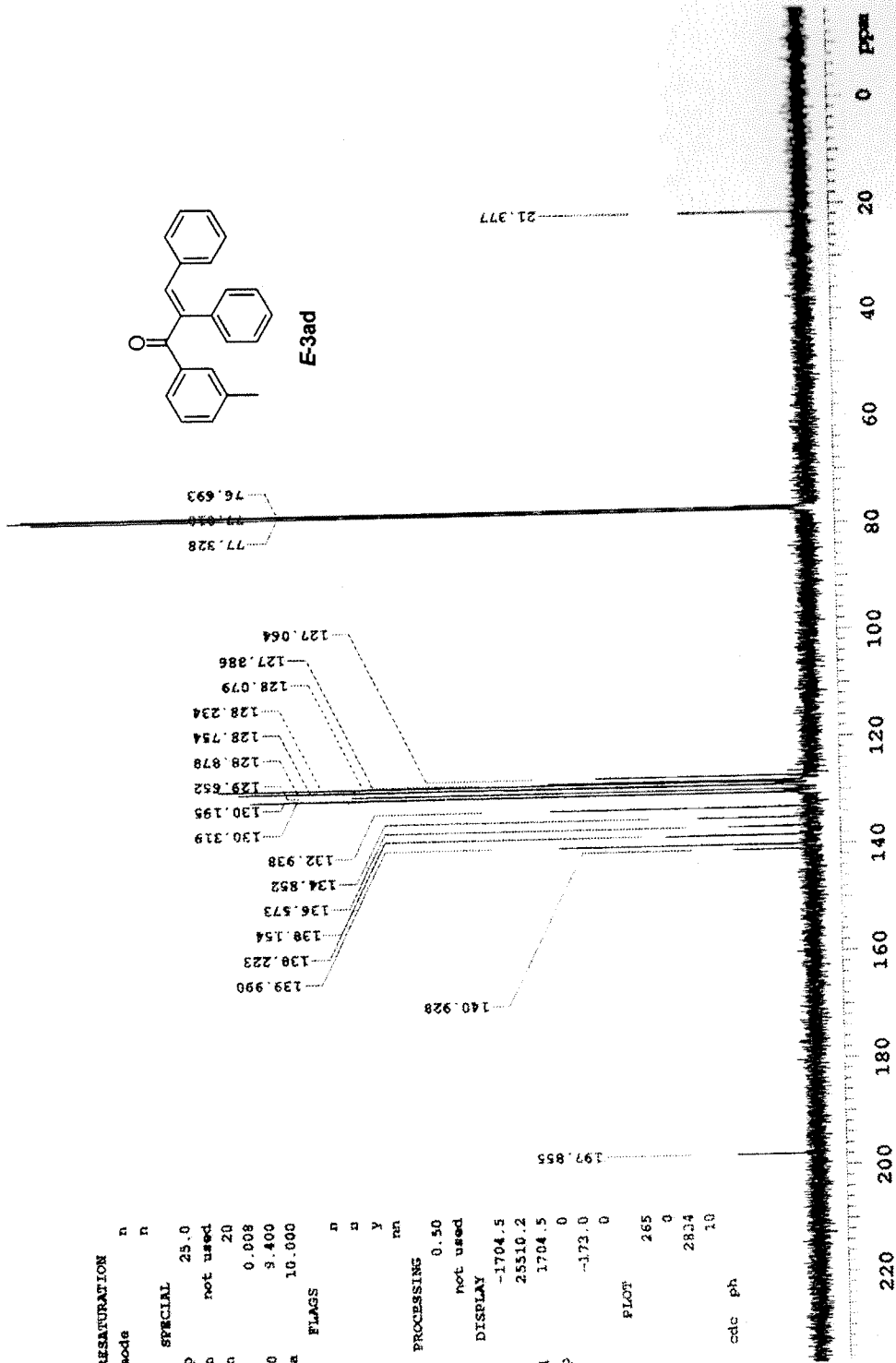
```

SAMPLE      PRESATURATION
data Max 11 2008 satmode      n
solvent cdccl3 wt.           n
file /home/walkup1~ SPECIAL
/vnarsys/data/ARNO~ temp     25.0
K GROUP/MELIH/MELI~ gain    not used
E-KK12-2120_11Mar2~ spin     20
908/CARBON_01.fid het       0.008
ACQUISITION pw90            9.400
sw 25510.2 alfa             10.000
at 1.385                     FLAGS
np 65536 ll                  n
fb 17000 ir                  n
bs 64 dp                      y
dl 1.000 hs                  rn
nt 512 nt                     n
ct 512 lb                     0.50
TRANSMITTER C13              not used
            DISPLAY
tn 100.471 sp                 -1704.5
tof 1529.7 wp                 25510.2
tpwr 54 rfl                  1704.5
pw 4.700 rfp                  0
DECOUPLER  H1 lp             -173.0
dn 0                          0
dof 0                          PLOT
dn 0 yyy wc                   265
dncsave w sc                   0
dpwr 39 va                    2834
dntf 9400 th                   10
ai cdc ph

```



E-3ad



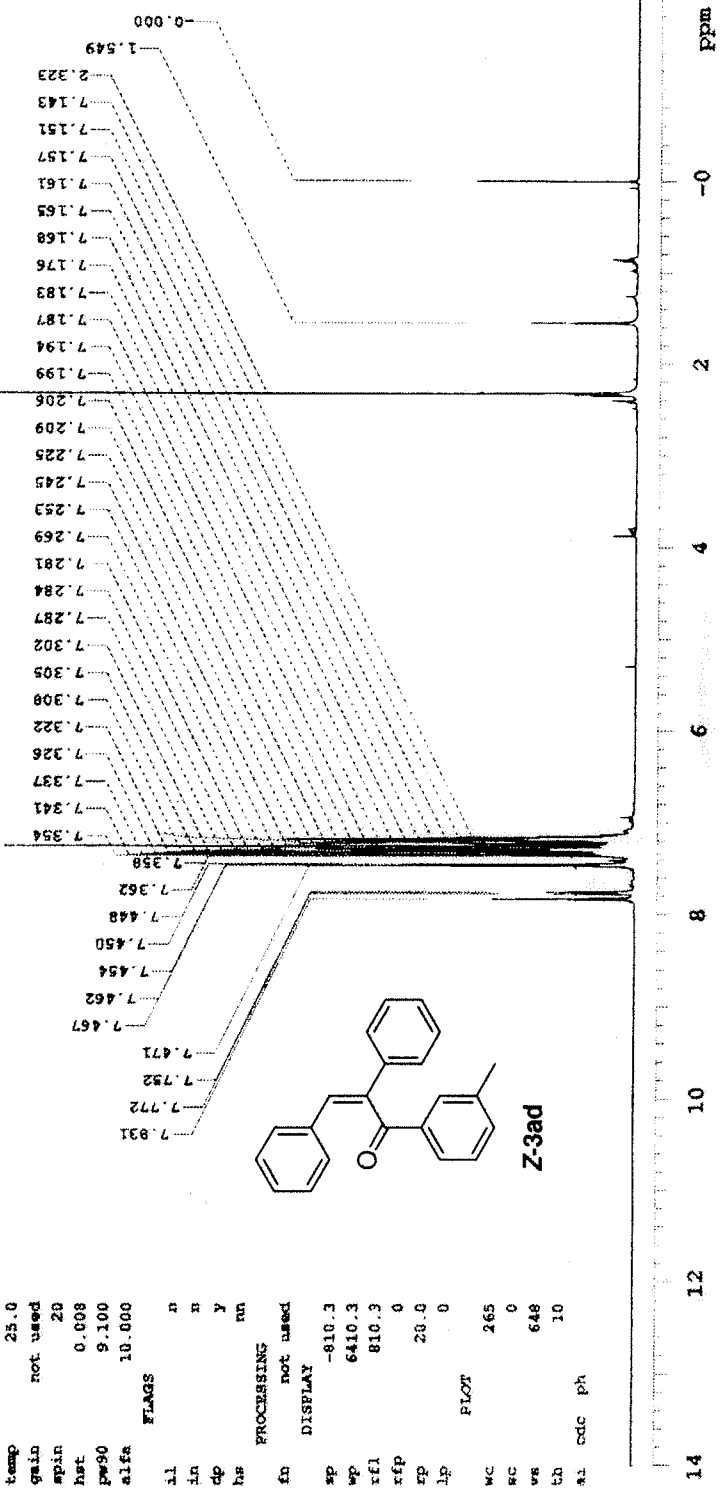
MR-KK12-12-19

expl PROTON

```

SAMPLE      PREPARATION
date Feb 11 2008 satmode n
solvent cdcl3 wet n
file /homs/walkupl~ SPECIAL 25.0
K GROUP/HELLH/MK-K- gain not used
KL2-12-19_11Feb200~ spin 20
8/PROTON_01.fid hst 0.008
ACQUISITION pw90 9.100
sw 6410.3 alfa 10.000
at 2.556 FLAGS
np 32768 il n
eb 4000 in n
bs 32 dp y
dl 1.000 hs nn
at 8 PROCESSING
ct 0 fn not used
TRANSMITTER H1 DISPLAY
tn -810.3
sfreq 399.424 wp 6410.3
tof 399.5 xfl 810.3
tpwr 61 xfp 0
pw 4.550 rp 20.0
DECOUPLER lp 0
do CL3 PLOT
dof 0 wc 265
da non wc 0
decurve W40_HCN5ms vs 648
dpar 34 th 10
dmf 29412 a1 cdc ph

```



14 12 10 8 6 4 2 0 ppm

7.354 7.358 7.362 7.367 7.372 7.377 7.382 7.387 7.392 7.397 7.402 7.407 7.412 7.417 7.422 7.427 7.432 7.437 7.442 7.447 7.452 7.457 7.462 7.467 7.472 7.477 7.482 7.487 7.492 7.497 7.502 7.507 7.512 7.517 7.522 7.527 7.532 7.537 7.542 7.547 7.552 7.557 7.562 7.567 7.572 7.577 7.582 7.587 7.592 7.597 7.602 7.607 7.612 7.617 7.622 7.627 7.632 7.637 7.642 7.647 7.652 7.657 7.662 7.667 7.672 7.677 7.682 7.687 7.692 7.697 7.702 7.707 7.712 7.717 7.722 7.727 7.732 7.737 7.742 7.747 7.752 7.757 7.762 7.767 7.772 7.777 7.782 7.787 7.792 7.797 7.802 7.807 7.812 7.817 7.822 7.827 7.832 7.837 7.842 7.847 7.852 7.857 7.862 7.867 7.872 7.877 7.882 7.887 7.892 7.897 7.902 7.907 7.912 7.917 7.922 7.927 7.931

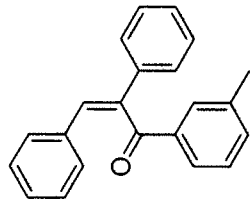
4.58.73 4.8362.51 15.15 3.19

MR-KK12-12-19

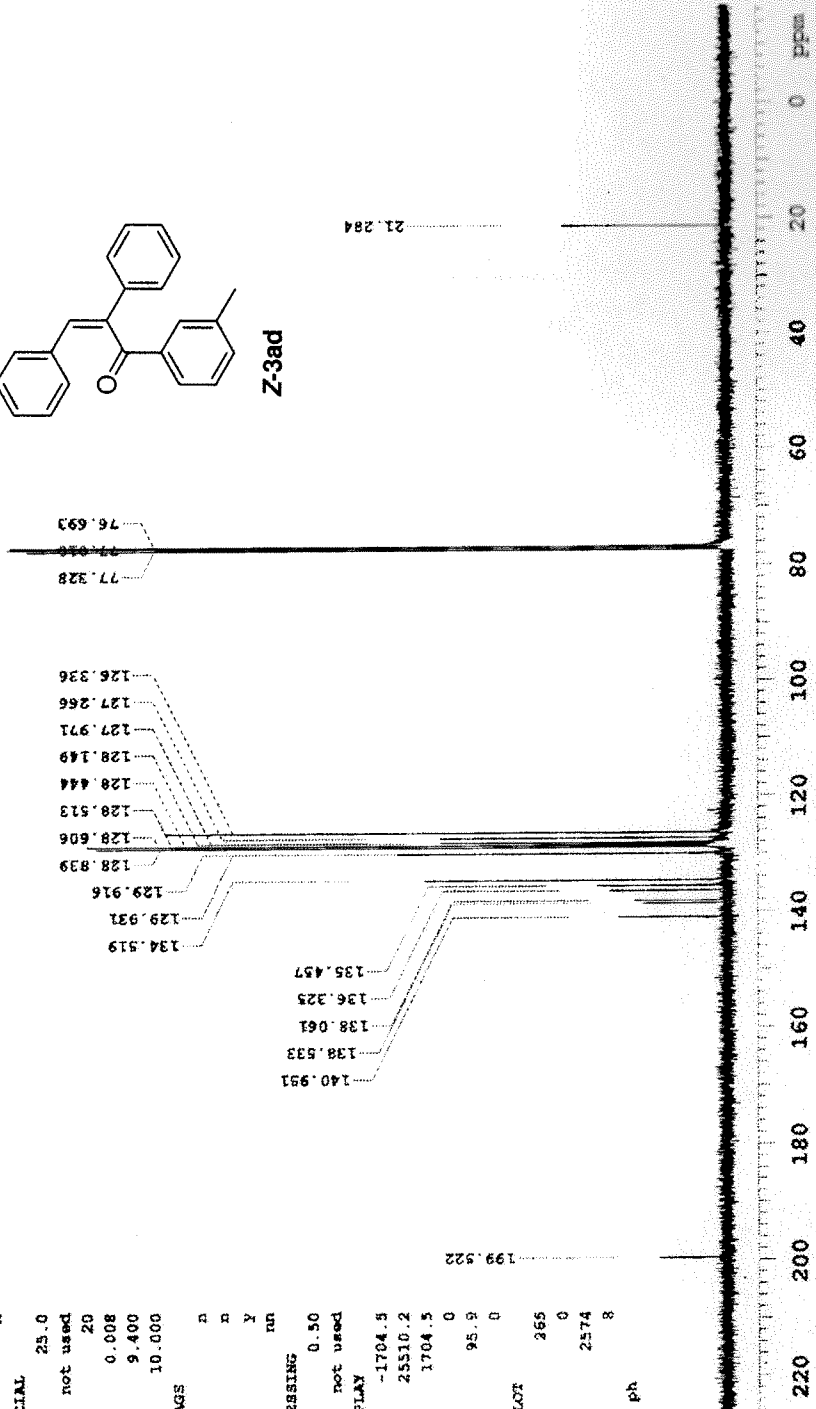
exp1 CARBON

```

SAMPLE PREPARATION
date Feb 11 2008 satmode n
solvent cdcl3 wt n
file /home/walkopi- SPECIAL
/vnmzsys/data/ARNO- temp 25.0
K GROUV/HELIX/MK-K- gain not used
K12-12-19_11Feb200- spin 20
8/CARBON_01.fid hst 0.008
ACQUISITION pw90 9.400
sv 25510.2 alfa 10.000
at 1.295 FLAGS
ap 65536 il n
zb 17000 in n
bs 64 cp y
dl 1.000 bs ru
nt 1000 PROCESSING
ct 1000 lb 0.50
tn TRANSMITTER fn not used
c13 DISPLAY
sfqc 100.471 sp -1704.5
cof 1529.7 wp 25510.2
tpwz 54 rfl 1704.5
pw 4.700 rfp 0
DECOUPLER tp 95.9
du HI lp 0
dof 0 PLOT
dm YYY WC 265
declava W SC 0
dpxr 39 va 2574
dmf 9400 th 8
ei cdc ph
  
```



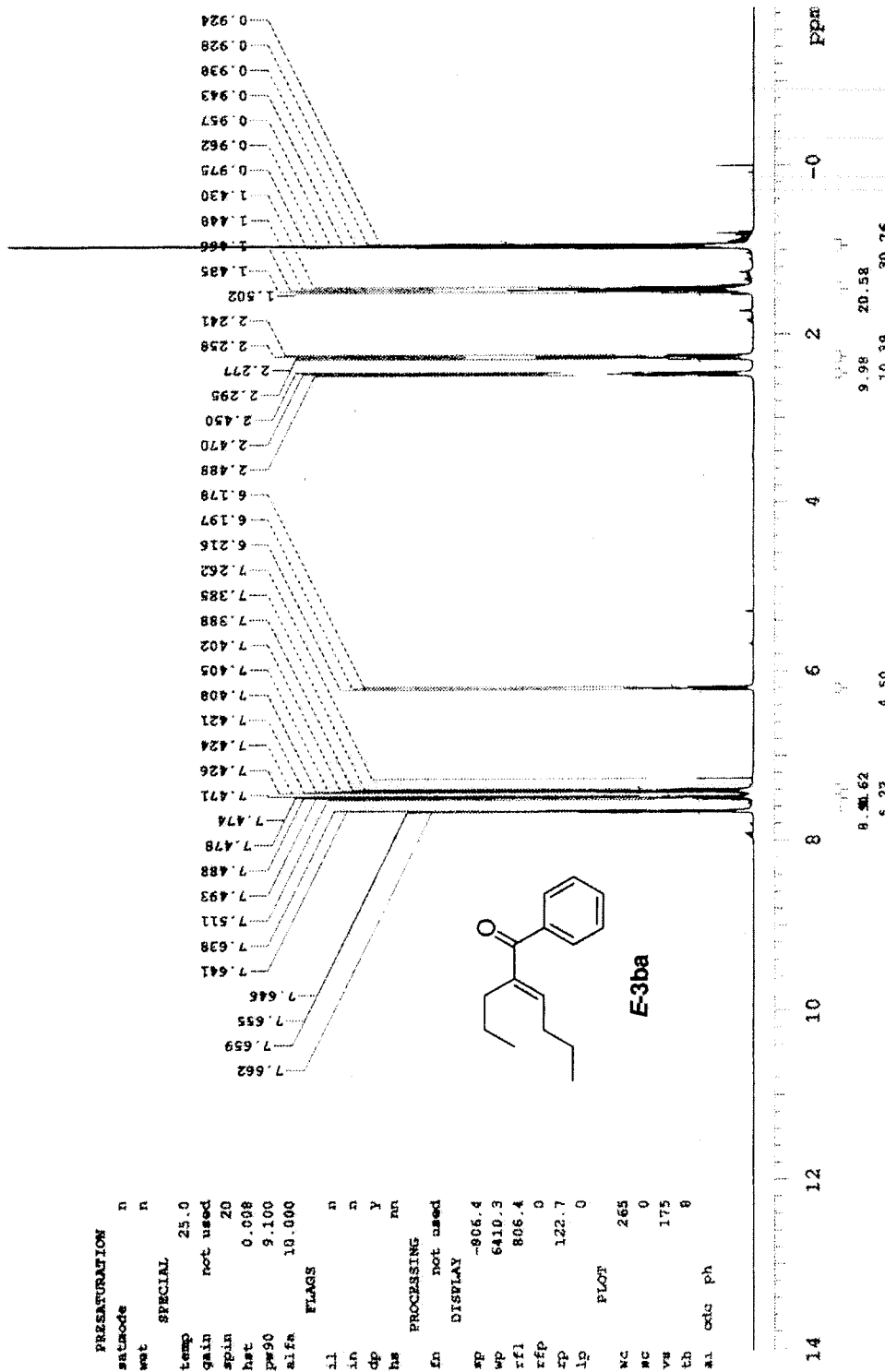
Z-3ad



WELIH-MK03481

exp1 PROTON

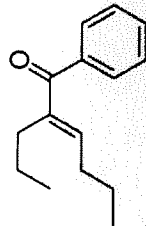
SAMPLE PREPARATION
date Apr 16 2008 satmode n
solvent cdc13 wst n
file /home/walupl- SPECIAL
/vmsys/data/ARTO- temp 25.0
K GROUP/WELIH/WELI- gain not used
H-MK03481_16Apr200- spin 20
8-dualtilt/PROT- hst 0.008
ON_01.fid pw90 9.100
ACQUISITION alfa 10.000
SW 6410.3 FLAGS
st 2.556 il n
sp 32768 in n
fd 4000 dp y
bs 32 hs nn
d1 1.000 PROCESSING
nt 8 fn not used
ct 8 DISPLAY
TRANSMITTER FP -906.4
HI WP 6410.3
#f1q 399.524 rfl 806.4
tof 399.5 rfp 0
tpwr 61 fp 122.7
pw 4.550 lp 0
DECOUPLER PLOT
dn C13 mc 265
dof 0 mc 0
dtr run vs 175
decwave W4U_MCNSMm th 8
dpr 34 al cdc ph
dmf 29412



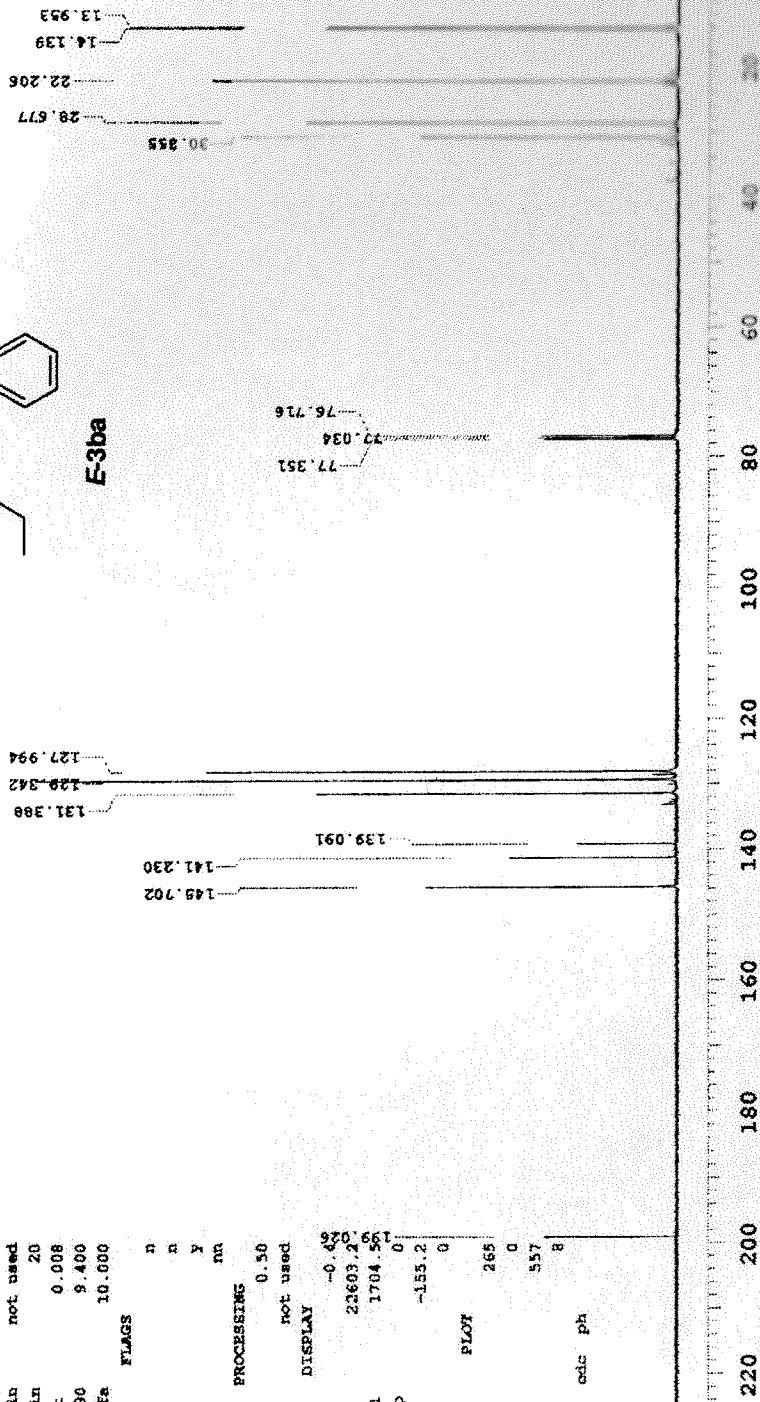
MELI-MK3481

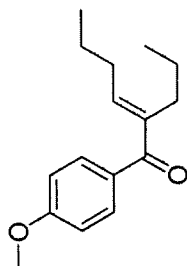
exp1 CARBON

SAMPLE PREPARATION
date Apr 16 2008 satmode n
solvent cdcl3 wet n
file /home/walshpi- SPECIAL
/vms/yys/data/ARTO- temp 25.0
K GROUP/MELI/MELI- gain not used
H-MK3481_16Apr200- spin 20
B-duseitilms/CARB- het 0.008
ON_01.fid pw90 9.400
ACQUISITION aifr 10.000
SW 25510.2
at 1.285 ll n
np 65336 in n
fb 17000 dp y
bs 64 bs in
dl 1.000 PROCESSING
nt 512 lb 0.50
ot 512 fb not used
TRANSMITTER DISPLAY
tn C13 sp -0.98
sfzq 100.471 wp 23603.2
tof 1529.7 rfl 1704.5
tpwx 54 rfp 0
pw 4.700 rp -155.2
DECOUPLER ip 0
dn H1 PLOT
dof 0 wc 265
dm YFF sc 0
decwave w vs 557
dpr 39 th 8
dmf 9400 al odc ph



E-3ba





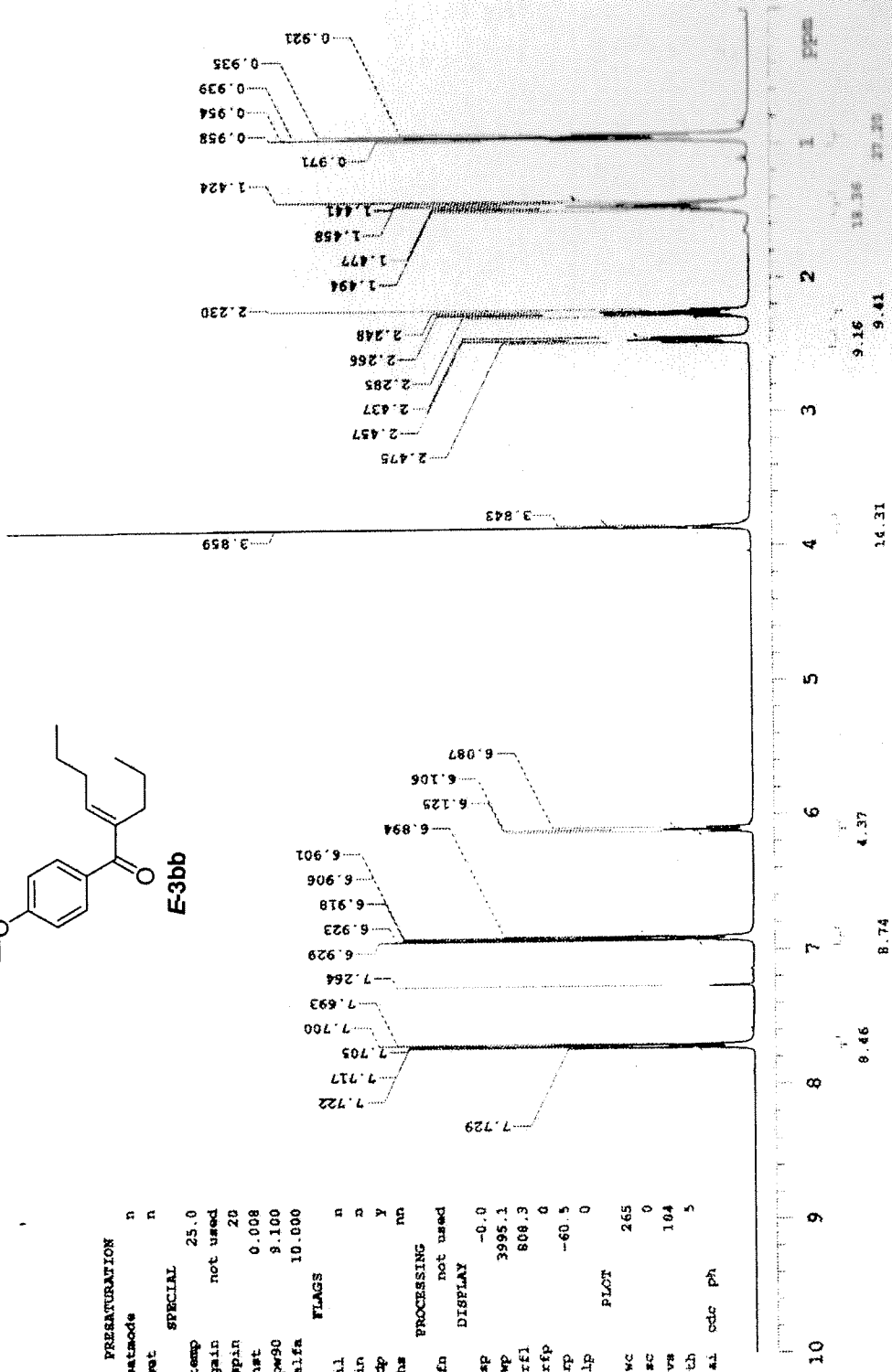
E-3bb

```

MELIH-KU21SAN
exp1 PROTON

SAMPLE PREPARATION
date Feb 20 2008 satcode n
solvent cdcl3 wet n
file /home/walkupi~ SPECIAL
/vmsys/data/ARTO~ temp 25.0
K GROUP/MELIH/MELI~ gain not used
H-KU21SAN_20Feb200~ spin 20
8/PROTON_01.fid hst 0.008
ACQUISITION pw90 9.100
sw 6410.3 aifa 10.000
at 2.556 il FLAGS
np 32768 il n
zb 4000 in n
bs 32 cp Y
dl 1.000 ls nn
nt 8 PROCESSING
ct 8 fn not used

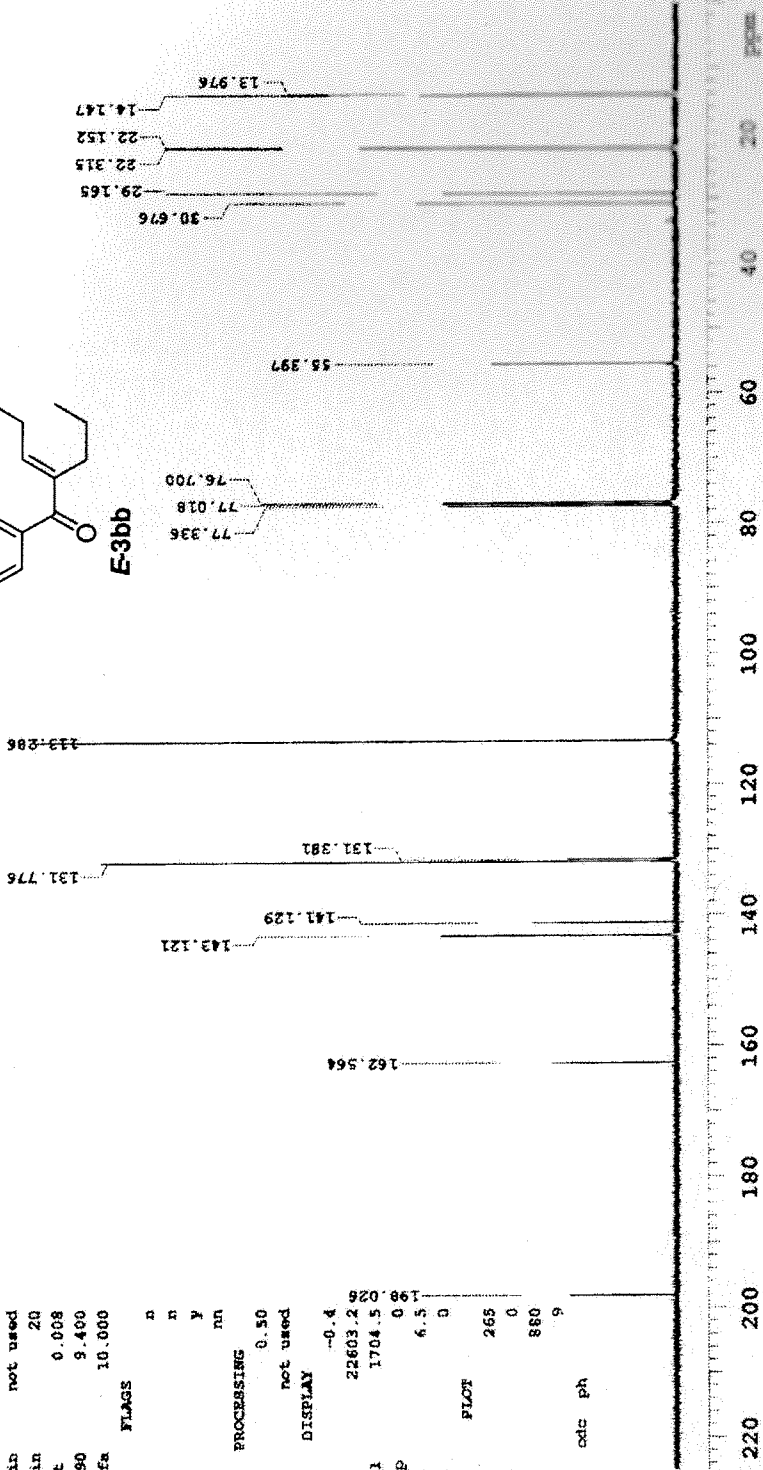
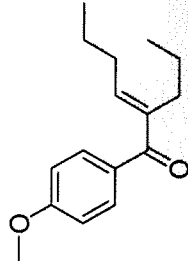
TRANSMITTER H1 sp DISPLAY
tn -0.0
sfrq 399.524 wp 3995.1
tof 399.5 rf1 808.3
tpwr 61 rfp 0
pw 4.550 rp -60.5
lp 0
DECOUPLER C13 PLOT
dn 0
dof 0 wc 265
dm nnn sc 0
decoupe W40_RCN5mm vs 104
dipwr 94 th 5
cmf 29412 ai cdc ph
  
```



MELIH-KX21SAF

exp1 CARBON

SAMPLE PREPARATION
date Feb 20 2008 satmode n
solvent cdcl3 wet n
file /home/walkupi-
/vmsys/data/ARTO- temp 25.0
K GROUP/MELIH/MELI- gain not used
H-KX21SAF_20Feb200- spin 20
8/CARBON_01.fid hst 0.008
ACQUISITION pw90 9.400
sv 25510.2 alfa 10.000
at 1.285
np 65536 il n
fb 17000 in n
bs 64 dp y
dl 1.000 ha nn
nt 512
ct 512 lb 0.50
TRANSMITTER fn not used
C13 DISPLAY
ln 100.471 sp --0.4
sfrq 1529.7 wp 22603.2 w
tpmx 54 rfl 1704.5
pw 4.700 rfp 0.6
DECOUPLER xp 6.5
dn H1 lp 0
def 0 FLOT 265
dn YYY WC 0
decwvs w sc 880
dpmr 39 vs 9400 th 9
cmf ai cdc ph

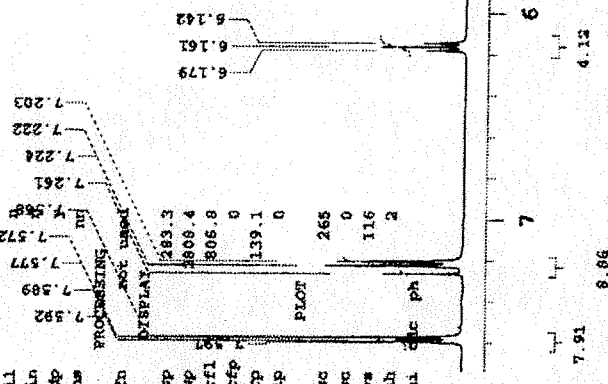
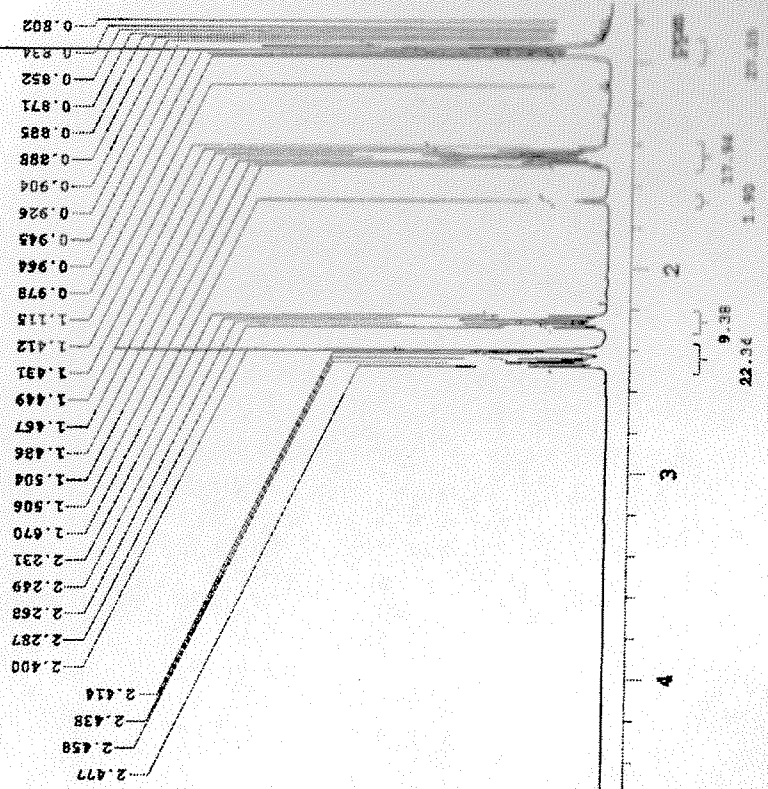
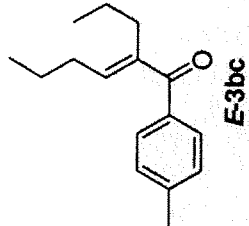


MELI-MKX5483

expt1 PROTON

SAMPLE PREPARATION
 date Apr 11 2008 setmode n
 solvent cdcl3 wet n
 file /home/walkup/~ SPECIAL 25.0
 /varxys/data/MELI~ temp not used
 H-MKX5483_11Apr200~ gain
 S/PROTON_01.fid spin 20
 ACQUISITION hst 0.008
 at 6410.3 pw90 9.100
 ap 32766 sifa 10.000
 ch 4000 il
 ha 32 in
 dl 1.000 cp
 nt 8 hs
 ct PROCESSING IN

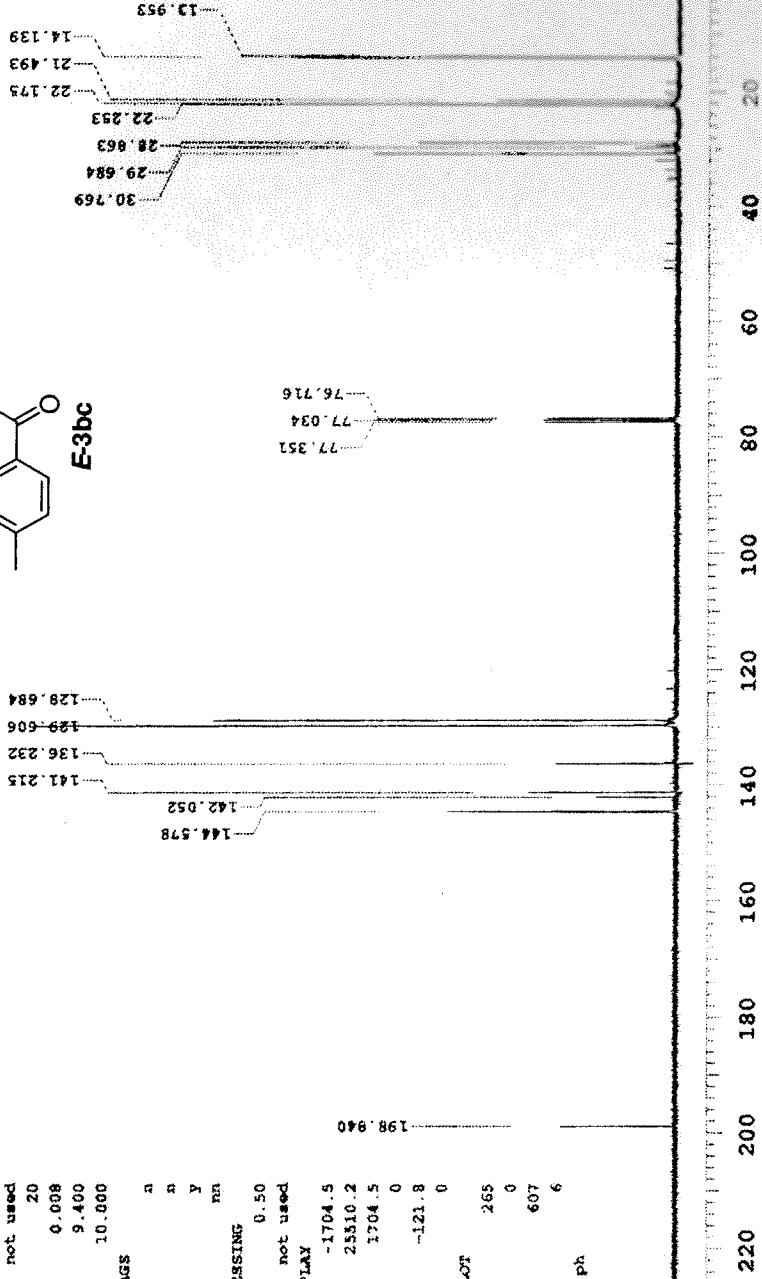
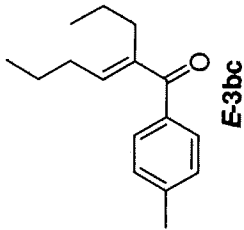
TRANSMITTER R1 IN NOT USED
 to R1 IN NOT USED
 freq 399.524 sp 283.3
 tof 399.5 wp 808.4
 tprz 61 rfl 808.8
 pw 4.550 rfp 0
 dc DECOUPLER cp 139.1
 dn c13 lp 0
 daf 0 PLOT
 dn dmh mc 265
 decrwa W40_HCN5mm rc 0
 dprz 34 vs 116
 dnf 29412 th 2
 ai dlc ph



MELIH-48K5483

expl CARBON

```
SAMPLE PRESATURATION
date Apr 8 2008 satmode n
solvent cdcl3 wet n
file /home/walshpl~ SPECIAL
/vmrays/data/ARFO~ temp 25.0
K GROUP/MELIH/MELI~ gain not used
H-MK5483_08Apr200~ spin 20
8/CARBON_01.fid hst 0.008
ACQUISITION pw90 9.400
sw 25510.2 alfa 10.000
at 1.285
ap 65336 il
cb 17000 in n
bs 64 cp y
dl 1.000 hs nn
nt 256
ct 256 lb 0.50
TRANSMITTER 256 fn not used
tn C13 DISPLAY
sfreq 100.471 sp -1704.5
tof 1529.7 wp 25510.2
tpwr 54 xfl 1704.5
pw 4.700 rfp 0
DNCOUPLER ip -121.8
dn h1 lp 0
dof 0
dm YYY wc FLOT 265
decrave W sc 0
dpr 39 vs 607
dnf 9400 th a1 cdc ph 6
```



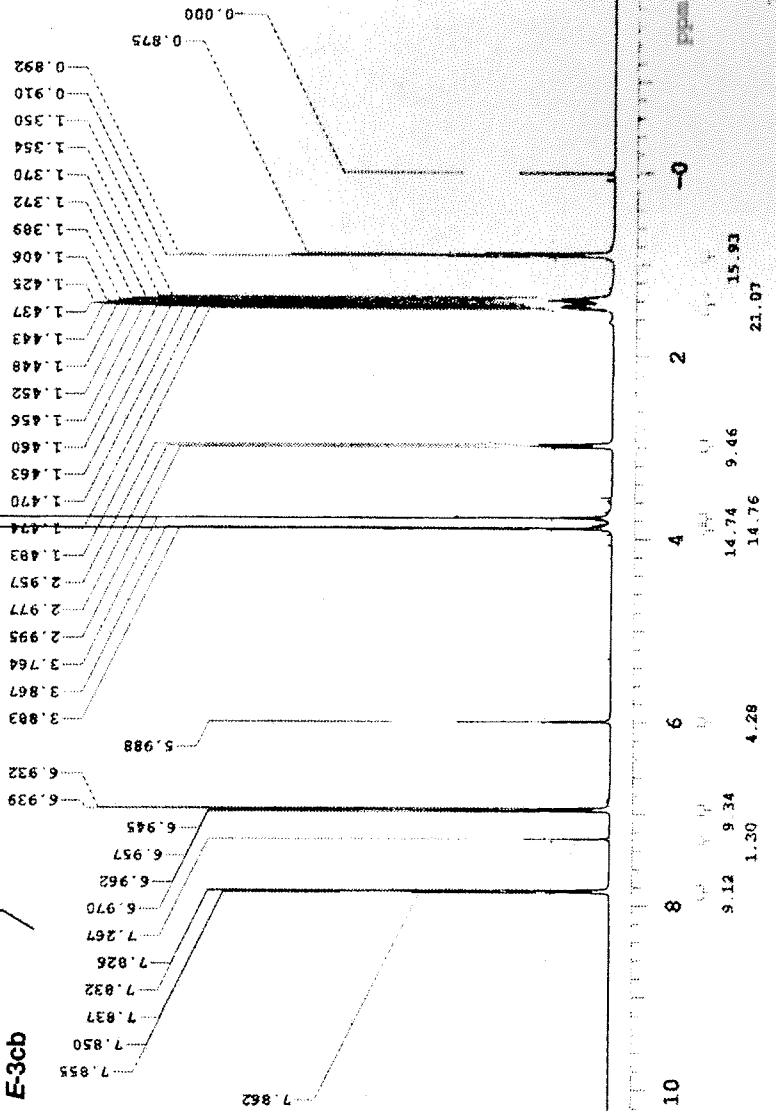
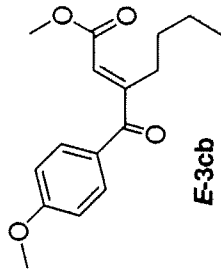
MR-KK175NF

exp1 PROTON

```

SAMPLE PRESENTATION
date Feb 16 2008 satmode n
solvent cdcl3 wt n
file /home/valkuyl- SPECIAL
/vmrays/data/ARTO- temp 25.0
K GROUP/MLIH/MK-K- gain not used
KK175NF_16Feb2008/P- spin 20
ROTOR_01.fid hst 0.008
ACQUISITION pw90 9.100
          6410.3 alfa 10.000
          FLAGS
sv 2.556 il n
sp 32768 in n
fb 4000 in n
bs 32 cp y
dl 1.000 hs nn
nt 8 PROCESSING
ct 9 fd not used
          DISLAY
ln HI sp -804.4
#fzq 399.524 wp 6410.3
tof 399.5 rfl 804.4
tpwr 61 rfp 0
pw 4.550 rp 13.9
          lp 0
          PLOT
dn C13
dof 0 wc 265
dn nnd sc 0
dchwave #40_HCN5MR vs 170
cpwr 34 tb 4
dmf 29412 al cdc ph

```



14 12 10 8 6 4 2 0

14.76 14.76 4.28 9.34 1.30 9.12 14.74 9.46 15.93 21.07

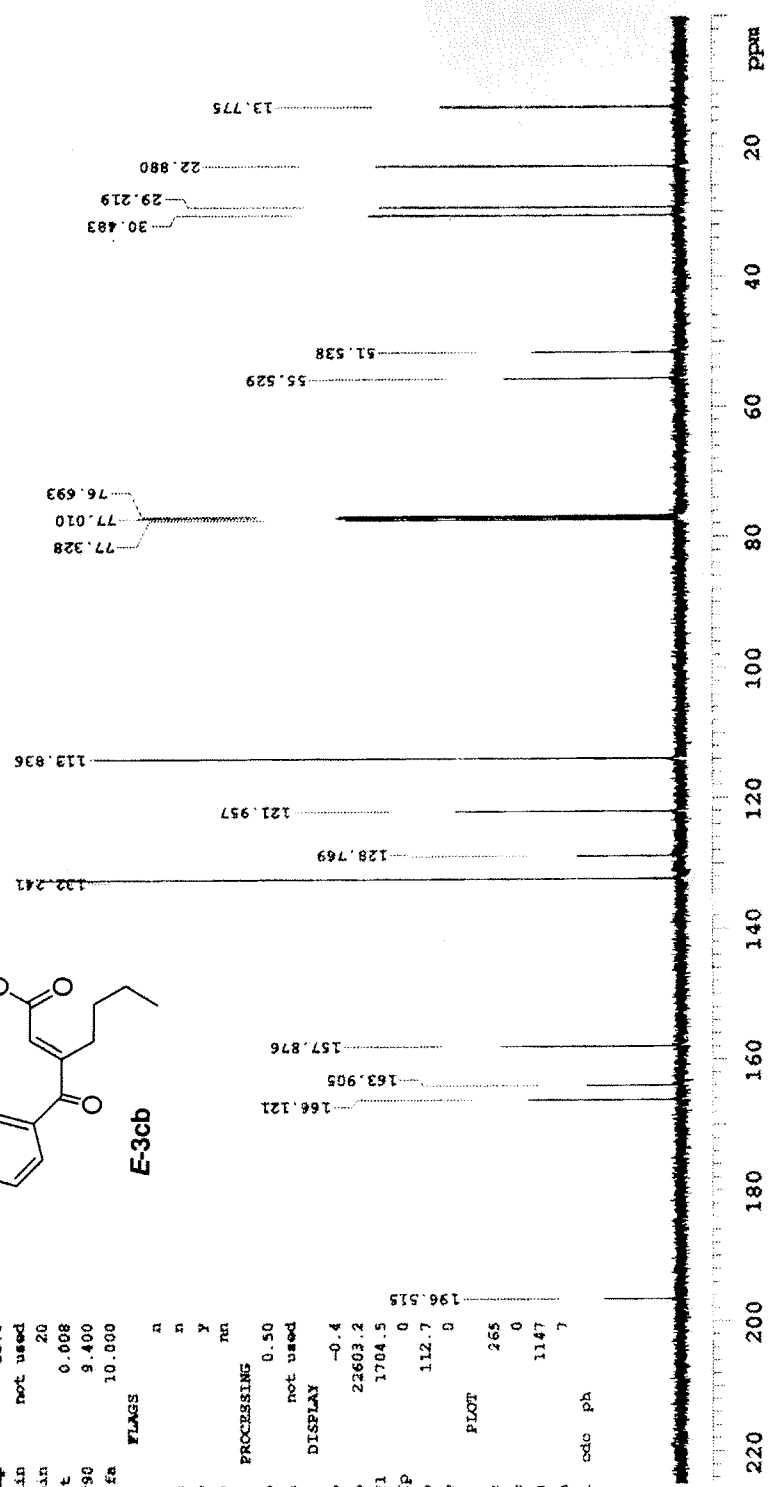
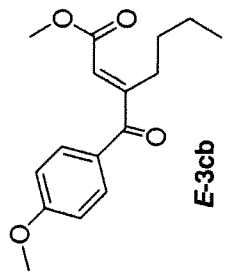
MS-KC17-SAF-C

exp1 CARBOM

```

SAMPLE      PRESATURATION
date Feb 16 2008 satmode n
solvent cdcl3 wet n
file /home/walkup/~
/vrnarys/data/ARTO~ temp 25.0
K GROUP/NEIHH/MK-K gain not used
K17-SAF-C_16Feb200- spin 20
8/CARBON_01.fid hst 0.008
ACQUISITION pw90 9.400
sv 25510.2 alfa 10.000
at 1.285
ap 65536 il
fb 17000 in n
bs 64 dp y
dl 1.000 hs nn
nt 256
ct 256 lb 0.50
TRANSMITTER C13 fn not used
tn DISPLAY
afrc 100.471 sp -0.4
tof 1529.7 wp 25603.2
tpwr 54 rfl 1704.5
pw 4.700 rfp 0
DECOUPLER xp 112.7
dn HI lp 0
dof 0 PLOT 265
dm YYY WC 0
decwave w sc 0
dpr 39 vs 1147
dmf 3400 th 7
ai odc ph

```



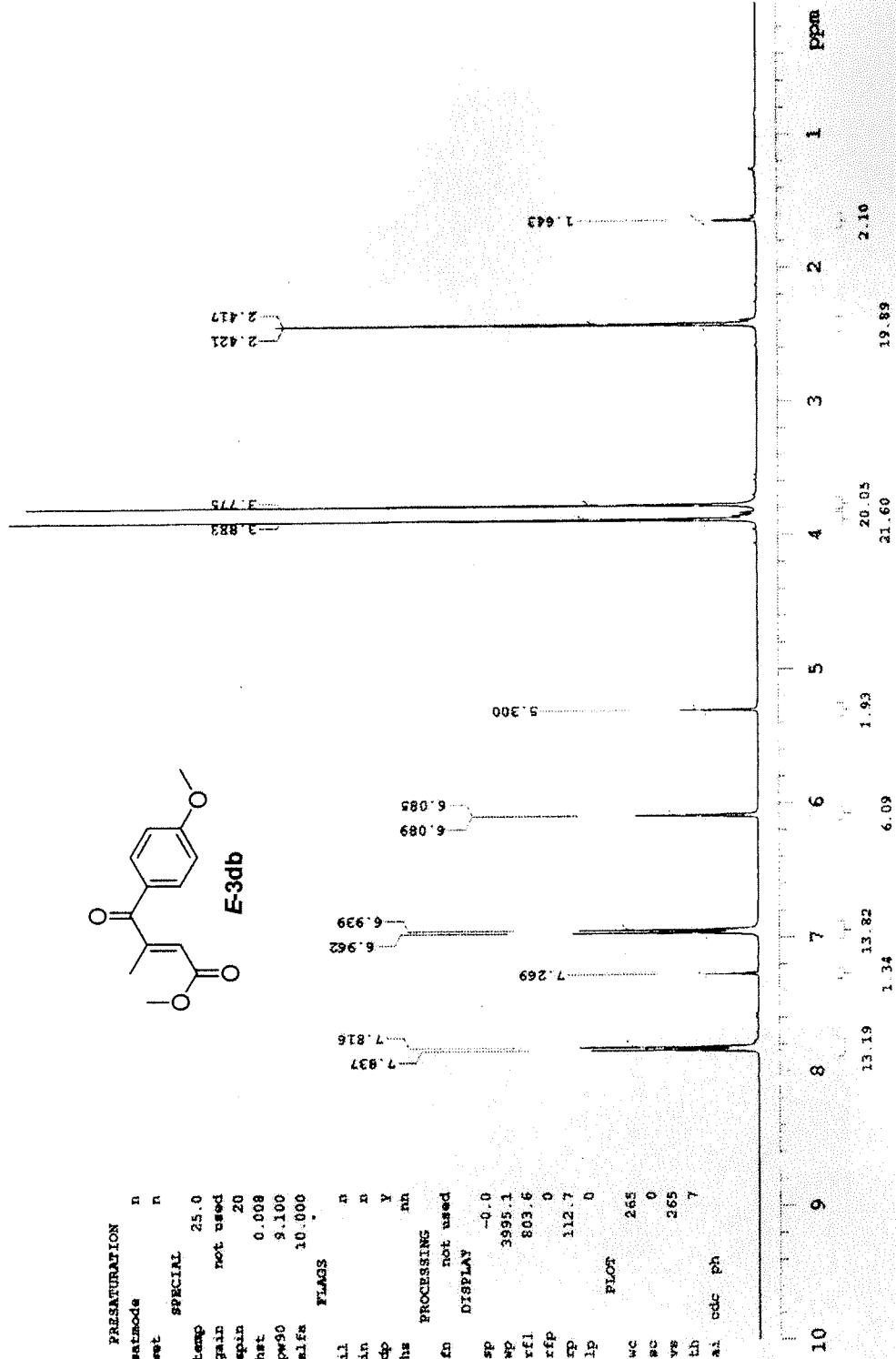
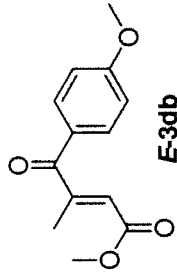
MELIH-KK27

exp1 PROTON

```

SAMPLE PRESATURATION
date Mar 19 2008 satmode n
solvent cdcl3 set n
file /home/walkup/~
/vmsays/data/ARNO~ temp 25.0
K GROUP/MELIH/MELI- gain not used
R-MK-KK27_19Mar200~ spin 20
8/PROTON_01.fid het 0.008
ACQUISITION pw90 9.100
*# 6410.3 alfa 10.000
at 2.556 FLAGS
xp 32768 il n
fb 4000 in n
bs 32 dp Y
dl 1.000 hs mh
nt 8 PROCESSING
ct 0 fn not used
TRANSMITTER H1 SP DISPLAY
tn -0.0
sfreq 399.524 mp 3995.1
tof 399.5 rfl 803.6
tpwr 61 rfp 0
pw 4.550 sp 112.7
DECOUPLER lp PLOT
dn C13
dof 0 wc 265
dm nnn #C 0
decnave #40_HCN5mm vs 265
dpcr 34 th 7
dmf 29412 ai cdc ph

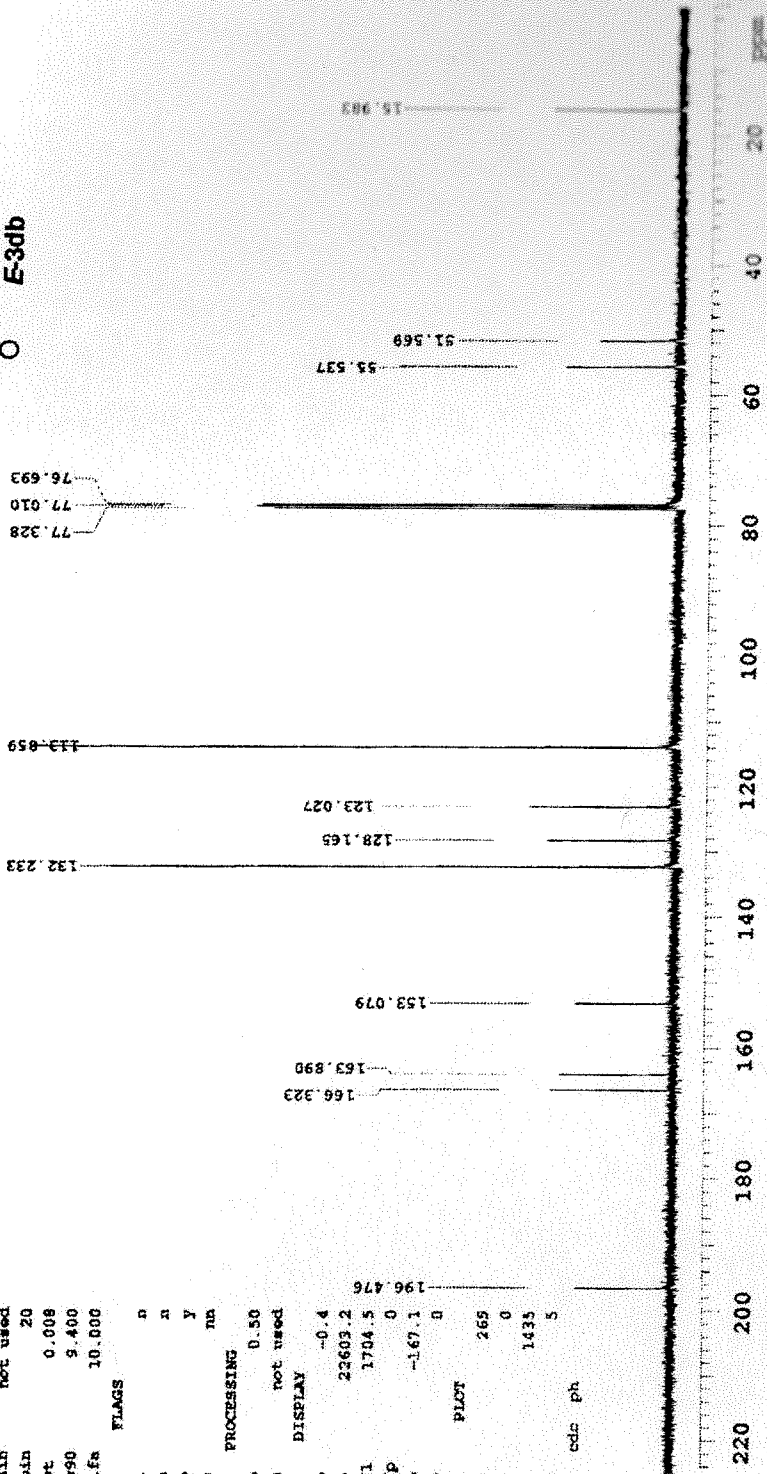
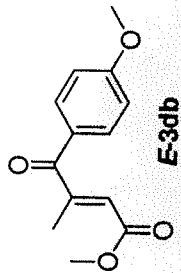
```



WELIH-MK-KC27

expl CARBON

SAMPLE PREPARATION
date Mar 19 2008 satmode n
solvent cdcl3 wet n
file /home/walkupl- SPECIAL
/varays/data/ARTO- temp 25.0
X GROUP/WELIH/WELI- gain not used
H-MK-KC27_19Mar2008 spin 20
8/CARBON...01.fid het 0.008
ACQUISITION pw90 9.400
sw 25510.2 alfa 10.000
at 1.205 FLAGS
np 65336 il n
fb 17000 in n
bs 64 dp Y
dl 1.000 bs na
nt 512 PROCESSING
ct 512 lb 0.50
tn TRANSMITTER fn not used
tn C13 DISPLAY
#freq 100.471 sp -0.4
tof 1529.7 wp 22603.2
tpr: 54 rfl 1704.5
pw 4.700 rfp 0
DISCOUPLER xp -167.1
dn H1 lp 0
dof 0 PLOT
dm pty wc 265
decwave w #c 0
dpr: 39 vs 1435
dmf 9400 th 5
sl cdc ph



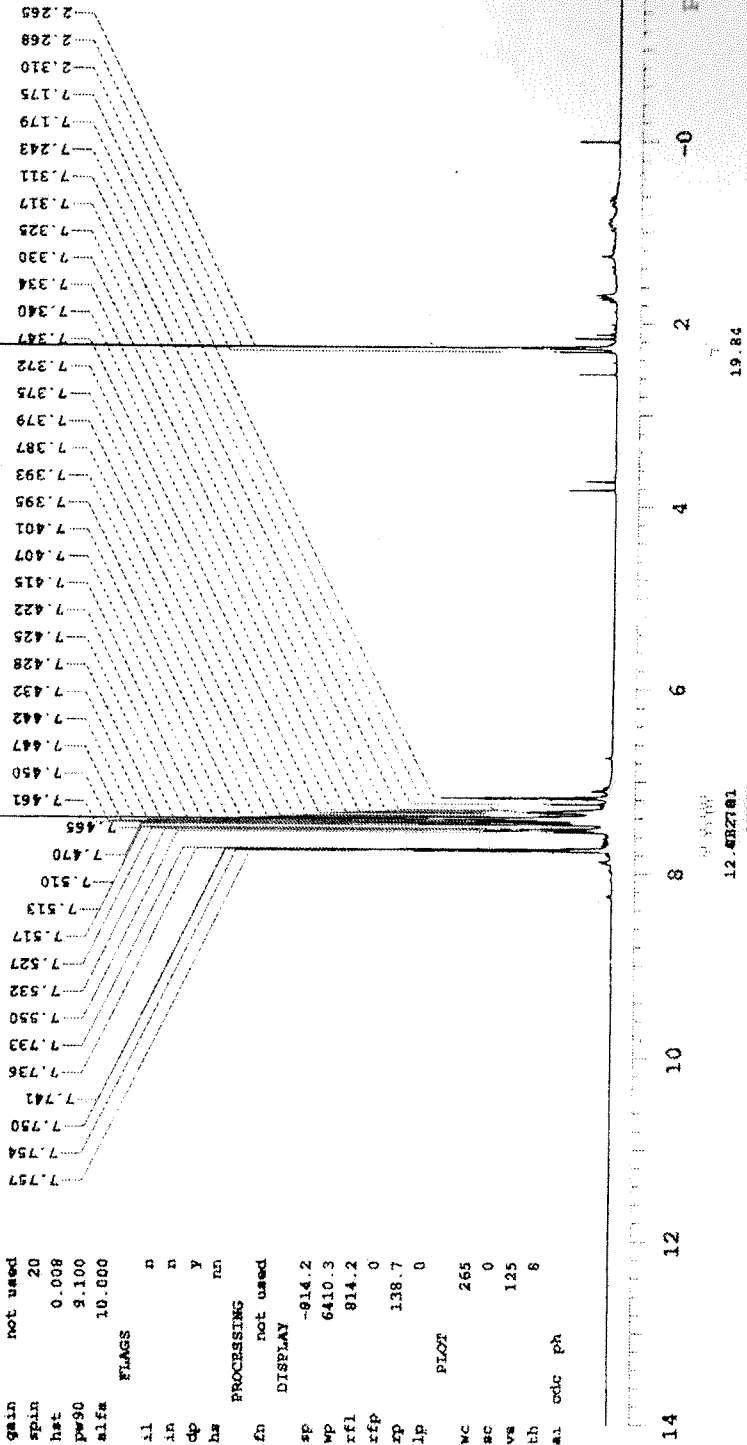
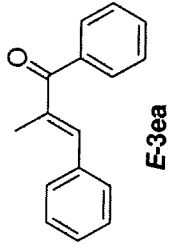
MELIH-MKKS481

exp1 PROTON

```

SAMPLE PRESATURATION
date Apr 11 2008 satmode n
solvent cdcl3 wet n
file /home/walkupl~ SPECIAL
/vmrays/data/ARTO~ temp 25.0
K GROUP/MELIH/MELI~ gain not used
H-MKKS481_11Apr200~ spin 20
8/PROTON_01.fid hat 0.008
ACQUISITION pw90 9.100
pw 6410.3 sifa 10.000
st 2.556 FLAGS
np 32768 il n
fb 4000 in n
bs 32 dp y
dl 1.000 hs run
nt 8 PROCESSING
ct 6 fb not used
TRANSMITTER DISPLAY
tn H1 sp -814.2
sfreq 399.524 mp 6410.3
tof 399.5 rfi 814.2
tpwr 61 rfp 0
pw 4.550 zp 138.7
DRCOUPLER lp 0
dn C13 PLOT
def 0 mc 265
dm nmz sc 0
dncwve W40_HCN5mm vs 125
dprv 34 th 6
dmf 29412 ai cdc ph

```



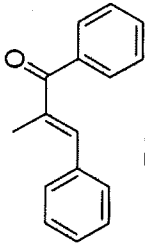
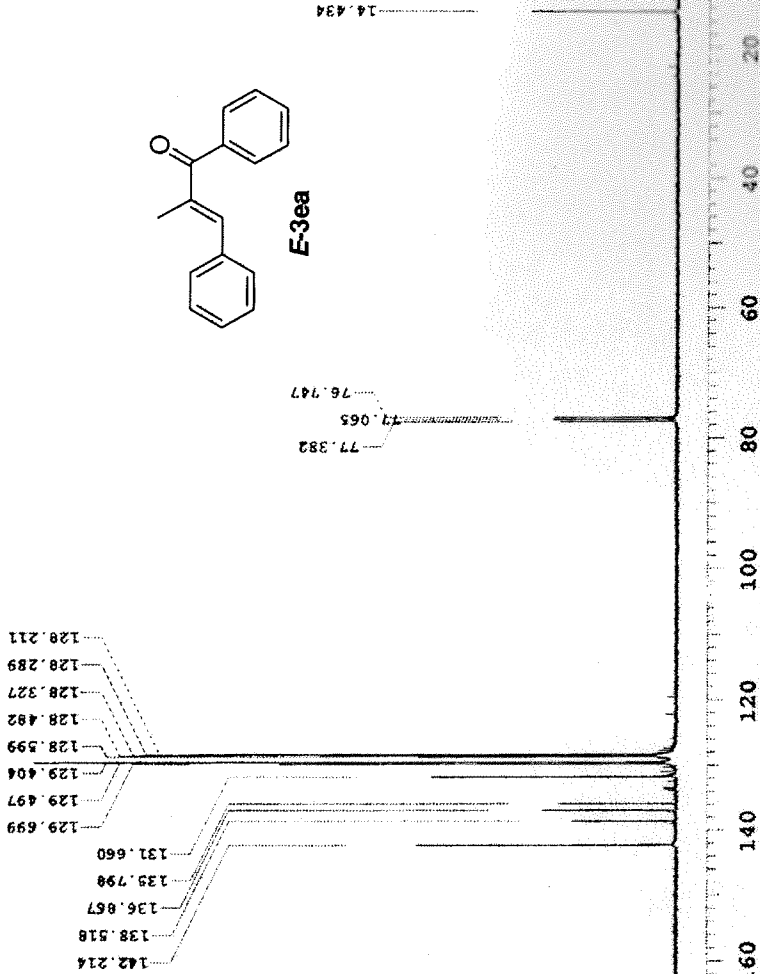
MELIH-MEK5481

exp1 CARBON

```

SAMPLE PREPARATION
date Apr 11 2008 satmode n
solvent cdcl3 wet n
file /home/walkupl- SPECIAL
/vmrays/data/ARCO- temp 25.0
K GROUP/MELIH/MELI- gain not used
E-MEK5481_11Apr200- spin 20
S/CARBON_01.fid hst 0.008
ACQUISITION pw90 9.400
pw 25510.2 aifa 10.000
at 1.265 FLAGS
np 65336 il n
fb 17000 in n
hs 64 dp y
dl 1.000 hs mn
nt 512 PROCESSING
ct 512 lb 0.50
tn TRANSMITTER fn not used
tn C13 DISPLAY
sfreq 100.471 #p -0.4
tof 1529.7 #p 22603.2
tpr 54 xfl 1704.5
pw DECOUPLER #p -156.00
dn H1 lp 0
dof 0 FLOT
dn XYI WC 265
dncave w sc 0
dpr 39 vs 572
dmf 9400 th 7
ai cdc ph

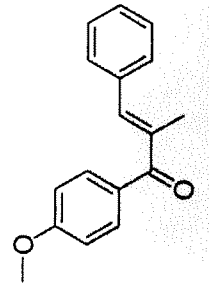
```



MELIH-MK-KK26

expl PROTON

SAMPLE PREPARATION
 date Mar 19 2008 satmode n
 solvent cdcl3 wet n
 file /home/walkupl~ SPECIAL
 /vmxrvs/data/ARFO~ temp 25.0
 K GROUP/MELIH/MELI~ gain not used
 B-MK-KK26_19Mar200~ spin 20
 8/PROTON_01.fid hst 0.008
 ACQUISITION pw90 9.100
 6410.3 aifa 10.000
 at 2.556 FLAGS
 np 32768 il n
 zb 4000 in n
 be 32 dp Y
 dl 1.000 hs nn
 nt 8 PROCESSING
 ct 8 fn not used
 tn HI sp DISPLAY
 -809.5
 sfreq 399.524 wp 6410.3
 tof 399.5 rfi 809.5
 tpwr 61 rfp 0
 pw 4.550 xp 113.0
 DECOUPLER ip 0
 dn C13 PLOT
 dof 0 wc 265
 dm rnm sc 0
 deacave W40_HCN5um vs 206
 dpr 34 th 5
 dnf 23412 ai cdc ph



2.257
2.254

7.835

7.333
7.331
7.325
7.317
7.255
7.106
7.103
6.961
6.939

7.818
7.813
7.799
7.415
7.408
7.402
7.396
7.380
7.347
7.340

0.000

14 12 10 8 6 4 2 0
 221348.24
 18.92
 18.65

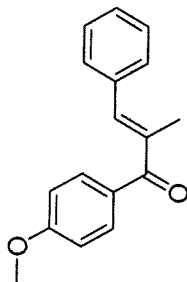
MELIH-KK-KK26

exp1 CARBON

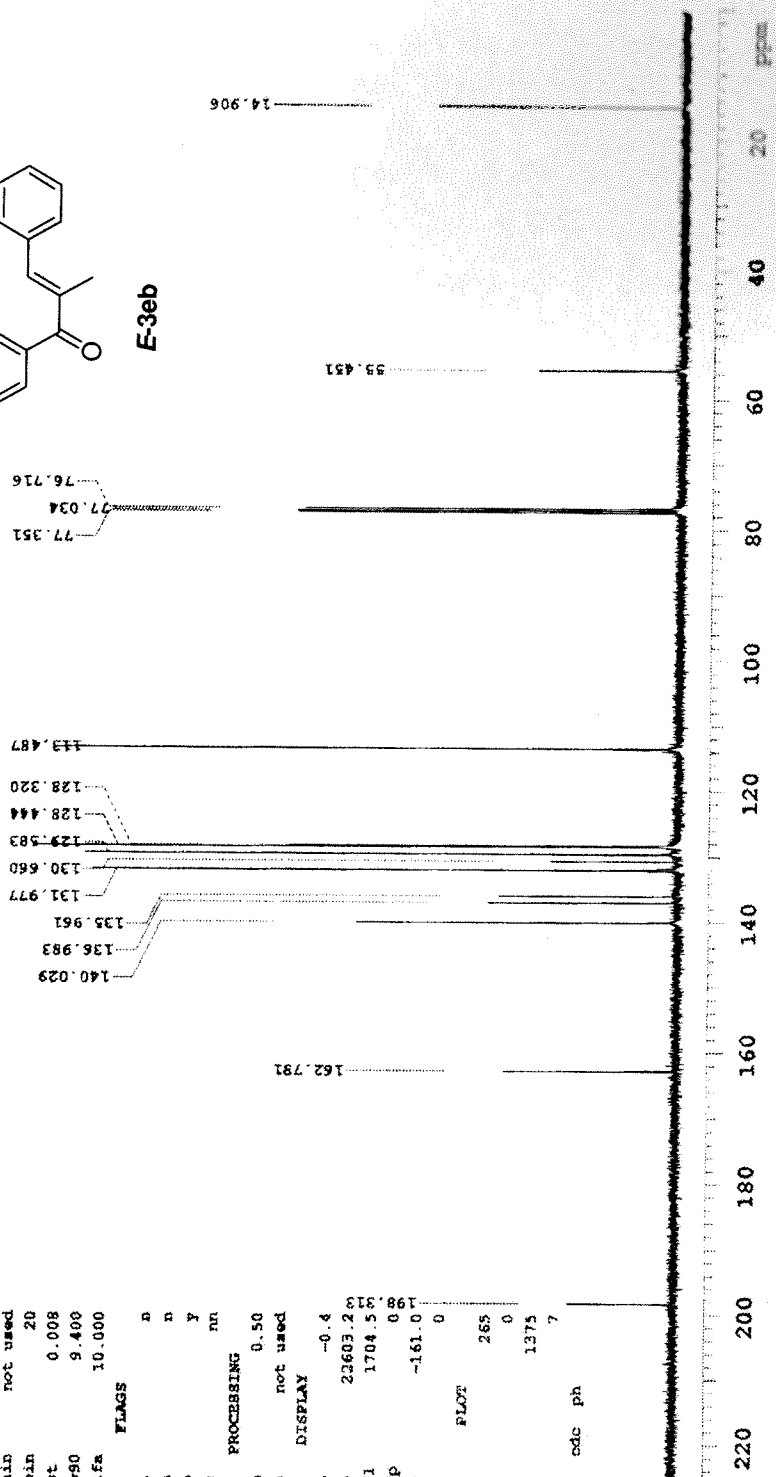
```

SAMPLE PREPARATION
date Mar 19 2008 estmode n
solvent cdcl3 wet n
file /home/walkup/~
/vmsays/data/ANTO~ temp 25.0
K GROUP/MELIH/MELI~ gain not used
H-KK-KK26_19Mar200~ spin 20
8/CARBON_01.kid hst 0.008
ACQUISITION pw90 9.400
sw 25510.2 alifa 10.000
at 1.285
sp 65536 il FLAGS
zb 17000 ln n
bs 64 dp y
dl 1.000 hs na
nt 512
ct 512 lb 0.50
TRANSMITTER En not used
tn C13 DISPLAY
sfrq 100.471 sp -0.4
tof 1529.7 wp 23603.20
tprx 54 rfl 1704.55
pw 4.700 rfp 0.88
DECOUPLER rp -161.07
dn H1 lp 0
dof 0 PLOT
dn YY7 wc 265
dechave v sc 0
dprx 39 vs 1375
dnf 9400 th ai cdc ph

```



E-3eb



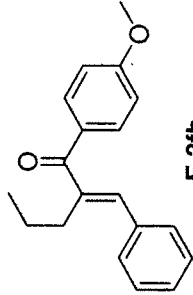
MT-KK16

exp1 PROTON

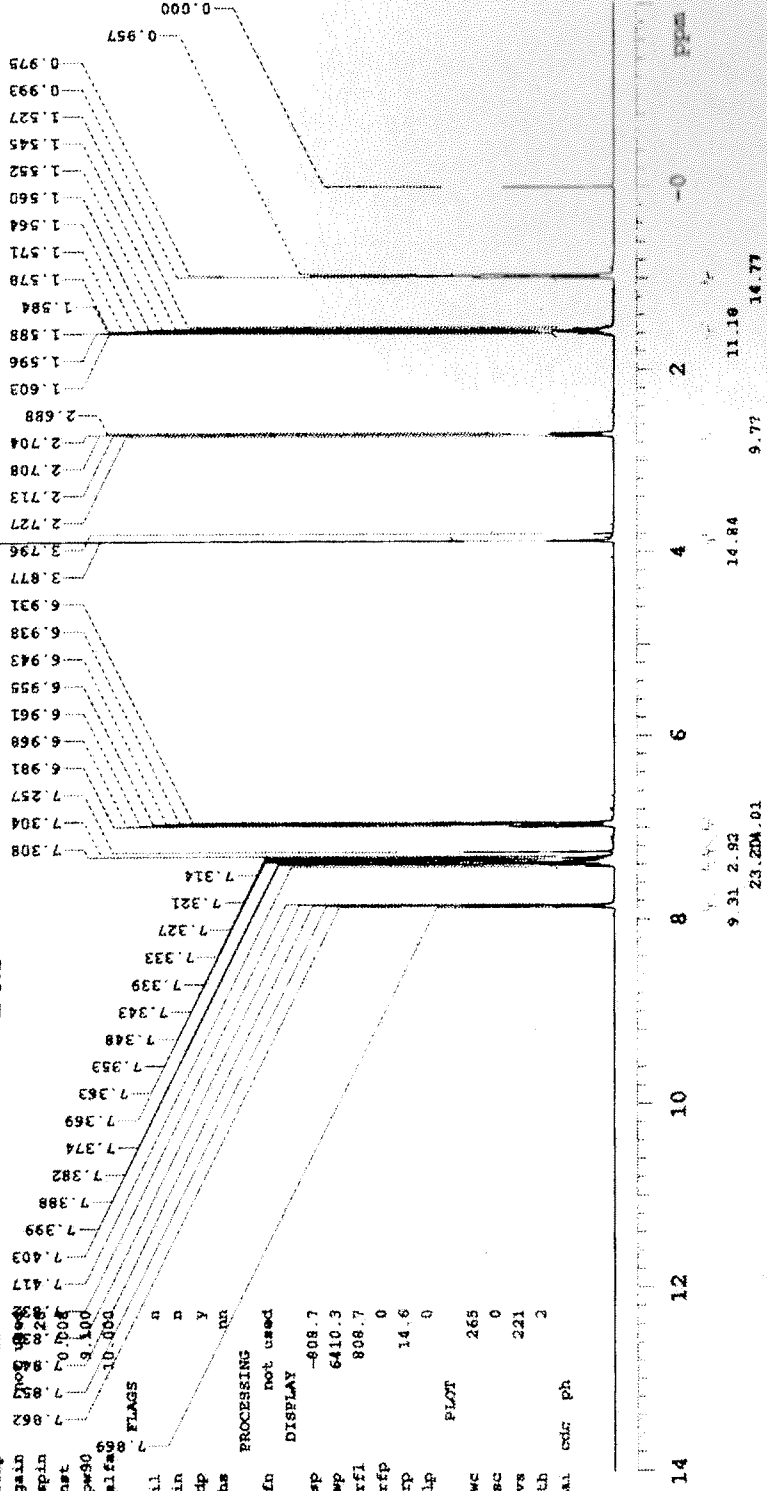
```

SAMPLE PRESENTATION
date Feb 16 2008 satmode n
solvent cdcl3 wet n
file /home/walkupl- /vmsys/data/ARTO- temp 25.0
K GROUP/MLIH/MK-K- gain Proc v0.0
K16_16TMB2008/PROT- spin 0.0 0.200
ON_01.fid hst 7.0 7.0
ACQUISITION pw90 9.100
6410.3 alfab 10.000
% FLAGS
np 2.556 al n
fd 32768 in d
bs 4000 in d
di 1.000 bs n
nt 8 PROCESSING nr
ct 8 fu not used
TRANSMITTER HI SP DISPLAY
tin HI SP -808.7
sfrq 399.524 wp 6410.3
tof 399.5 rfi 808.7
tpmz 61 rfp 0
pw 4.550 tp 14.6
DECOUPLER lp PILOT 0
dn C13
dof 0 wc 265
dn nne sc 0
decave H40...MNSM vs 221
dpr 34 lb 2
chf 29412 al cdr ph

```



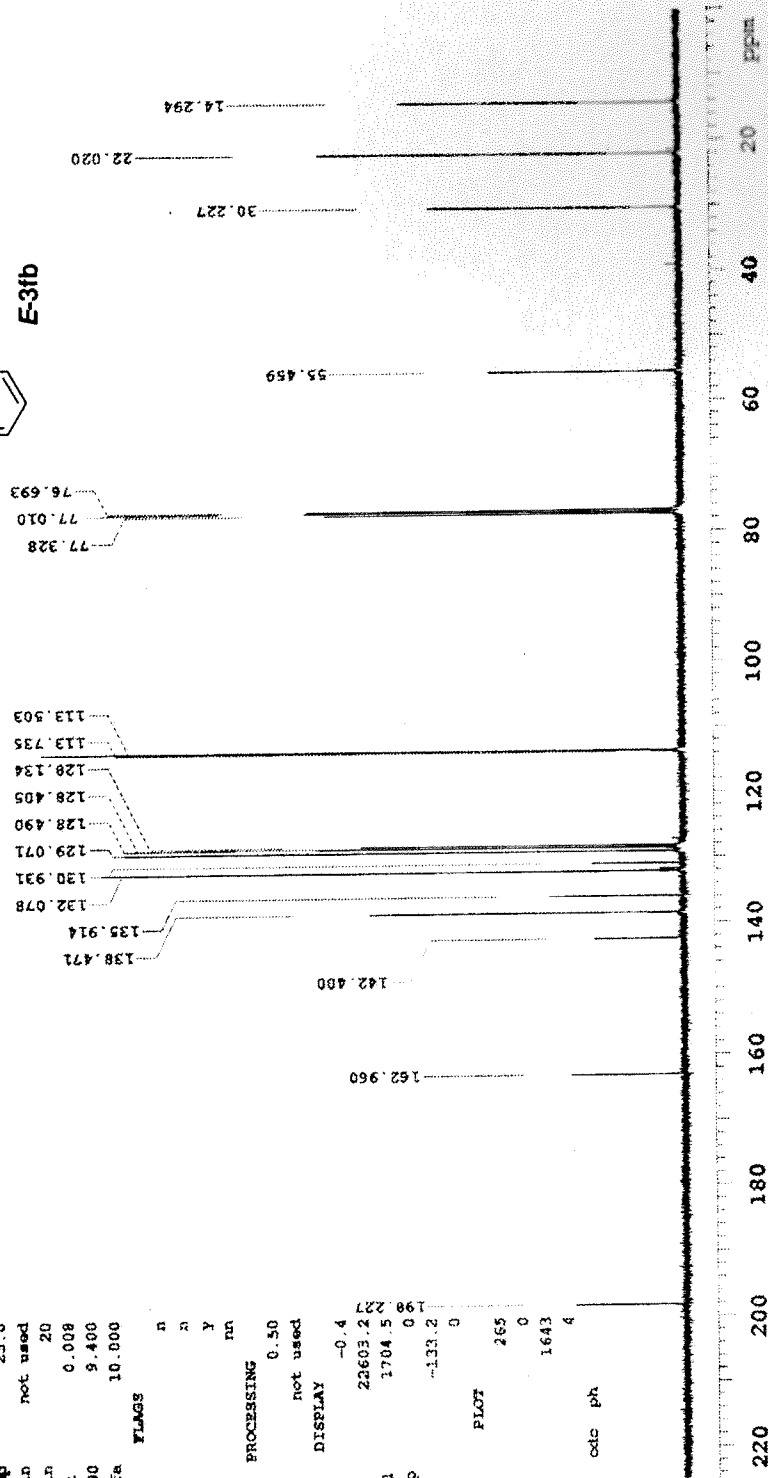
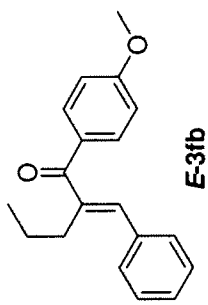
E-3fb

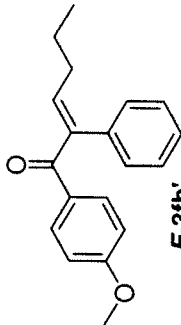


MELIH-KK16-SAF

exp1 CARBON

```
SAMPLE PREPARATION
date Mar 4 2008 satmode n
solvent cdcl3 wet n
file /home/walkupl- /vmsys/data/ARTO- temp 25.0
K GROUP/MELIH/MELI- gain not used
H-KK16-SAF_04mar20- spin 20
08/CARBON_01.fid hst 0.008
ACQUISITION Pw90 9.400
sw 25510.2 alfa 10.000
at 1.295 FLAGS
ap 65336 l1 n
fb 17000 in n
bs 64 op y
dl 1.000 bs n
nt 1000 PROCESSING
ct 1000 lb 0.50
TRANSMITTER fn not used
tn C13 DISPLAY
sfreq 100.471 sp -0.4
tof 1529.7 mp 22603.2
tpwr 54 rfl 1704.5
pw 4.700 rfp 0.08
DECOUPLER rp -133.2
dn HI lp 0
dof 0 PLOT
dn FYY WC 265
decwve w sc 0
dpr: 39 vs 1643
dmf 9400 th 4
ai cdc ph
```



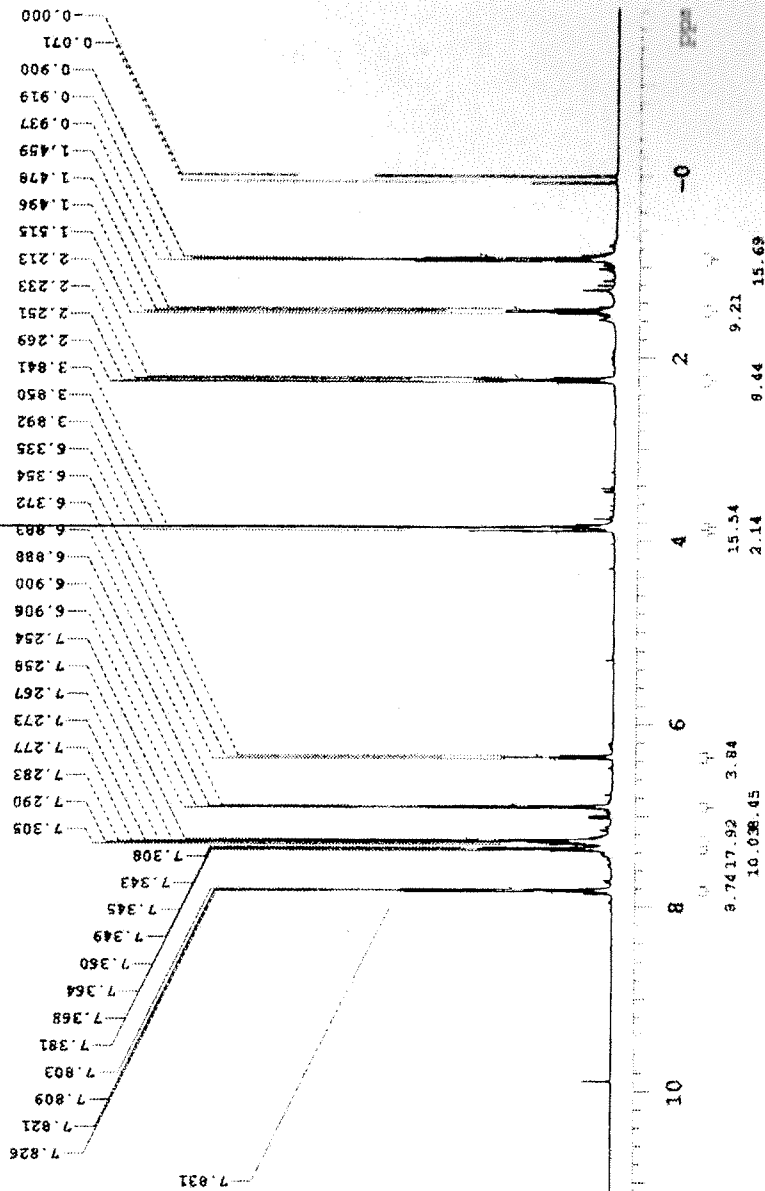


E-3fb'

KK-KK16-83-105-(SAFETYELIX ANALYSE)

exp1 PROTON

SAMPLE PRESENTATION
 date Feb 16 2008 satmode n
 solvent cdcl3 wet n
 file /home/walkupl- SPECIAL
 /vmsr/s/data/ARNO-temp 25.0
 K GROUP/MLIH/KK-K gain not used
 K16-83-105_16Feb20- spin 20
 DS/PROTON_01.fid hst 0.008
 ACQUISITION Pw90 9.100
 #v 6410.3 aifa 10.000
 at 2.556 FLAGS
 sp 32768 il n
 fh 4000 in n
 bs 32 dp y
 dl 1.000 hs ru
 nt 8
 ct 8 fn not used
 TRANSMITTER DISPLAY
 tn H1 sp -008.3
 srfq 399.524 wp 6410.3
 tof 399.5 rfl 808.3
 tpmr 61 rfp 0
 pw 4.550 xp 15.1
 DECOUPLER lp 0
 dn C13 PLOT
 dof 0 wc 265
 dn nnn sc 0
 decwave W40_HCN5mm vs 456
 dpr 34 th 8
 dmf 23412 ai cdc ph



14 12 10 8 6 4 2 0

9.7417.92 3.84
 10.038.45
 15.34 2.14
 9.22 8.44 15.69

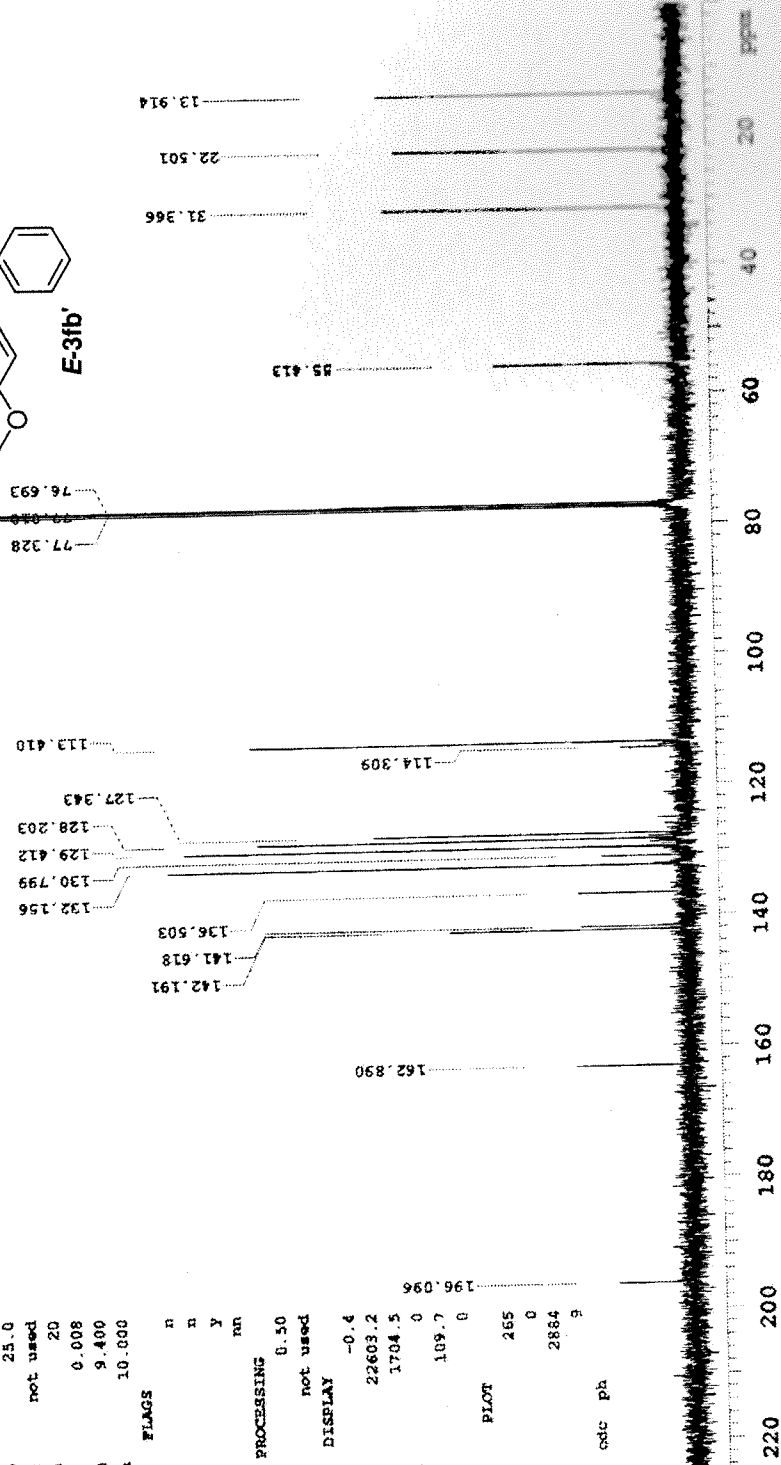
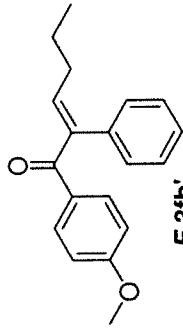
MR-KK16-83-SAF#12L1K

exp1 CARBON

```

SAMPLE PREPARATION
date Feb 16 2008 satmode n
solvent cdcl3 wat SPECIAL n
file /home/walkupl-
/vmrays/data/ARTO- temp 25.0
K GROUP/HELIX/MK-K- gain not used
K16-83-SAF#12L1K_1- spin 20
GE852008/CARBON_01- hst 0.008
.fid pw90 9.400
ACQUISITION sifa 10.000
SW 25510.2 FLAGS
at 1.285 ll n
ap 65536 in n
zb 17000 dp n
bw 64 hs nn
dl 1.000 PROCESSING 8.50
nt 512 lb not used
ct 512 fn DISPLAY
tn CL3 sp -0.4
sfrq 100.471 WP 22603.2
tof 1529.7 rfl 1784.5
tpr 54 rfp 0
pw 4.700 zp 189.7
DECOUPLER lp 0
dn H1 PLOT
dof 0 WC 265
dm VVY sc 0
decave w vs 2884
dper 99 th 9
dmf 9483 ai odc ph

```

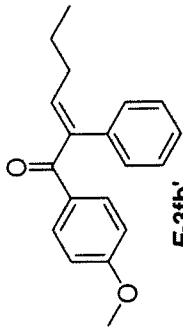


MR-K16-83-SAFKILIK

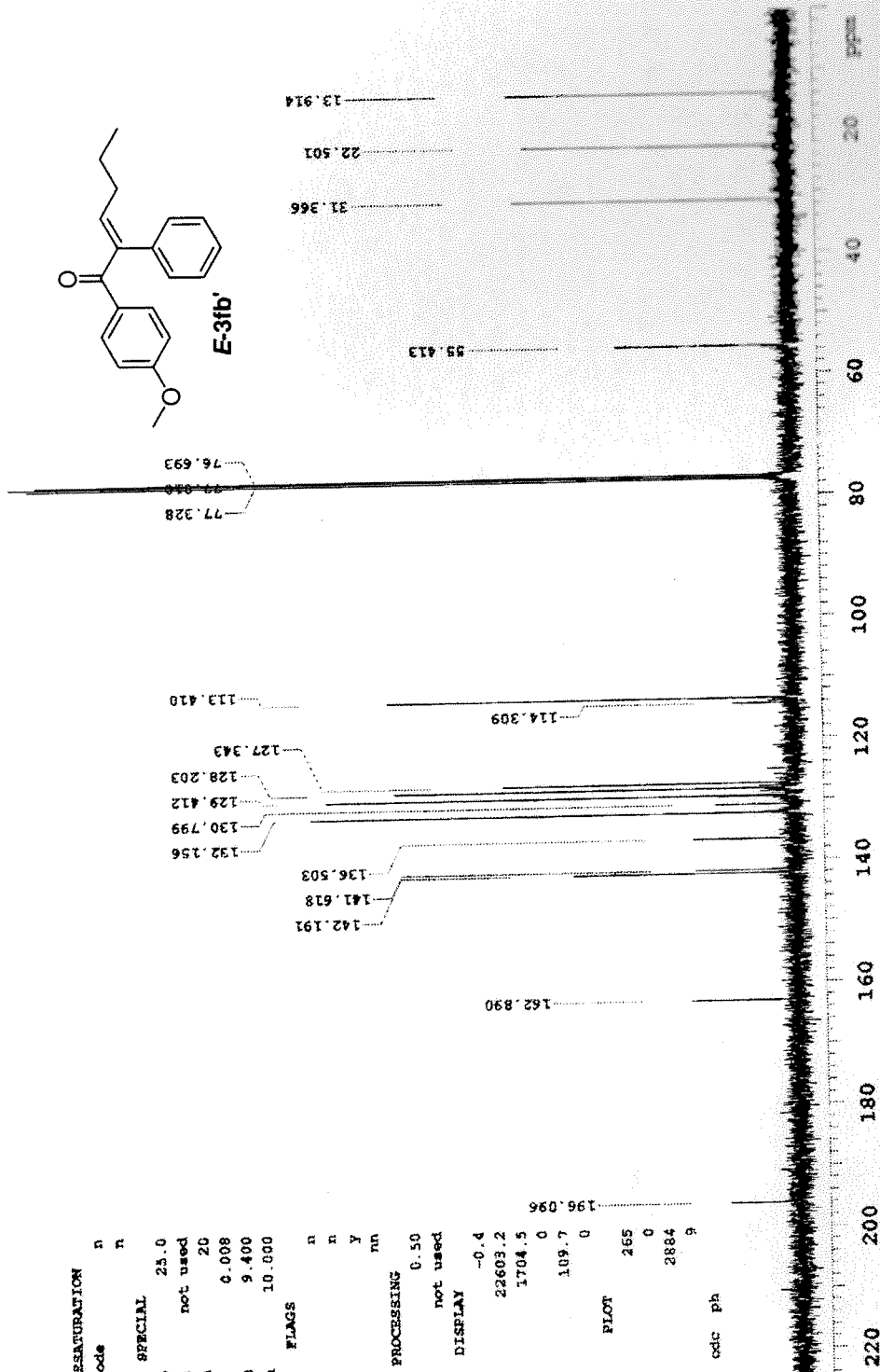
exp1 CARBON

```

SAMPLE PRESENTATION
date Feb 16 2008 satmode n
solvent cdcl3 wet n
file /home/walkupl- SPECIAL
/vmrays/data/ARCO- temp 25.0
K GROUP/VELIB/MK-K- gain not used
K16-83-SAFKILIK_1- spin 20
6Feb2008/CARBON_01- het 0.008
.fid pw90 9.400
ACQUISITION alfa 10.000
sw 25510.2 FLAGS
at 1.285 il n
rp 65536 in n
zb 17000 dp y
bs 64 hs rn
di 1.000 PROCESSING
nt 512 lb 0.50
ct 512 fn not used
TRANSMITTER DISPLAY
tn C13 SP -0.4
freq 100.471 wp 22603.2
lof 1529.7 rcl 1704.5
tpw 54 kfp 0
pw 4.700 rp 109.7
DECOUPLER lp 0
dn HI PLOT
doz 0 wc 265
dm YYY ac 0
decwave w vs 2884
cpwr 39 th 9
cmf 9400 ai cdc ph
  
```



E-3fb'



YTSN69

exp4 s2pu1

SAMPLE date Apr 15 2006

solvent CDCl3

file ACQUISITION exp4

sw 6389.5

at 1.398

np 25528

fb not used

bs 16

d1 1.000

nt 8

ct 8

TRANSMITTER H1

tn 399.883

sfrq 431.3

tof 60

tpwr 7.850

pw DECOUPLER C13

dn dof 0

da dmn nnn

dmm c

dpvr 48

dmf SPECIAL 13156

temp 30.0

gain not used

spin 20

hst 0.008

pw80 15.300

atfa 20.000

FLAGS

ll n

ln n

dp y

hs nn

fn not used

PROCESSING

DISPLAY

sp 15.2

wp 3194.5

rf1 795.6

rfp 0

rp -72.6

lp -72.7

PLOT

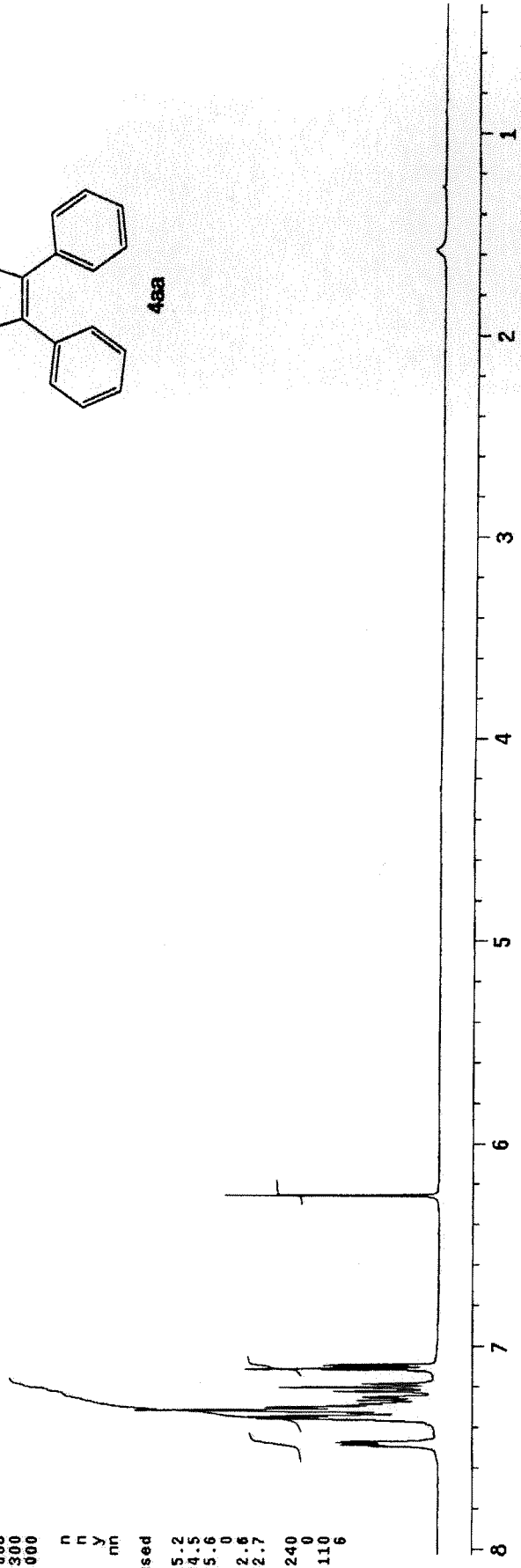
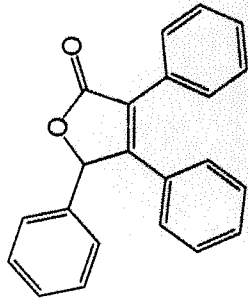
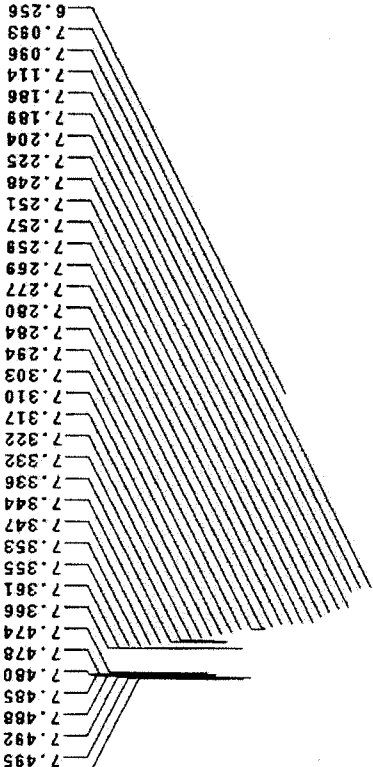
wc 240

sc 0

vs 110

th 6

at cdc ph



SAMPLE
 date Apr 15 2006
 solvent CDC13
 file /export/home/~
 vnmr1/Cetinkaya/19~
 C-Experiment/YTM69~
 -C13.fid

ACQUISITION
 sw 25125.6
 at 1.199
 np 60270
 fb 13800
 bs 64
 d1 2.000
 nt 512
 ct 512

TRANSMITTER C13
 tn sfrq 100.561
 tof 1553.6
 tpwr 60
 pw 8.750
 DECOUPLER H1

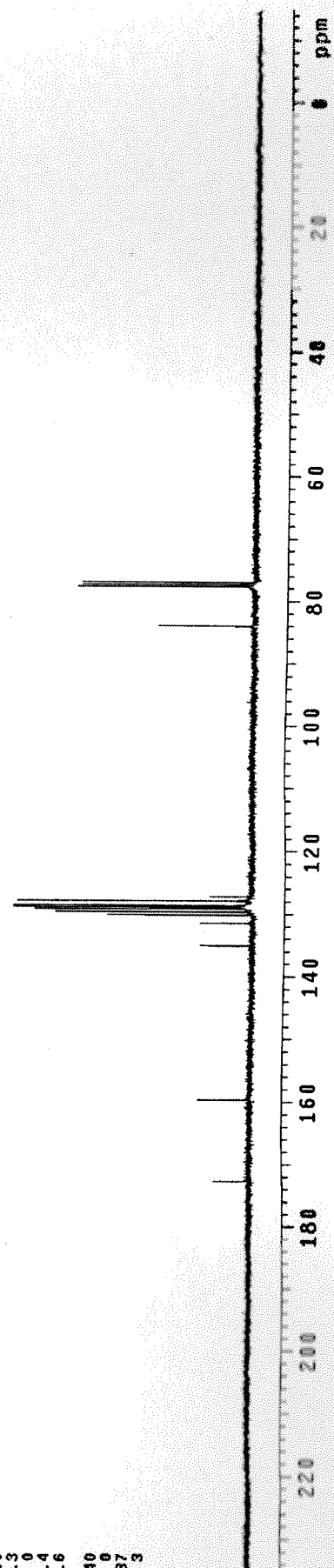
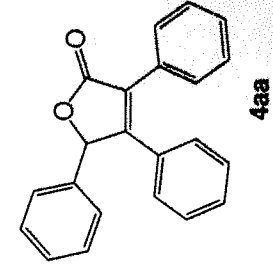
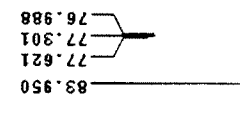
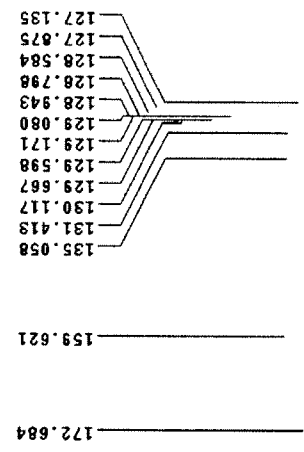
dn dof H1
 da 0
 dm YVY
 dnm W
 dpwr 46
 dmf 12270

SPECIAL
 temp 30.0
 gain not used
 spin 20
 hst 0.008
 pw90 17.500
 alfa 20.000

FLAGS
 i1 n
 in n
 dp y
 hs nm
 lb 1.00
 fn not used

PROCESSING
 sp -1501.5
 wb 25124.9
 cf1 1502.3
 rfp 0
 tp 166.4
 tp -441.6

PLOT
 wc 240
 sc 0
 vs 137
 th 3
 at no ph



M85CRYS

exp4 s2pu1

SAMPLE

date Mar 26 2006
solvent CDCl3
file /export/home/
vmri/getinkaya/16
-Experiment/M85CRYS

ACQUISITION

sw 6389.6
at 1.998
np 25528
fb not used
bs 16
d1 1.000
nt 8
ct 8

TRANSMITTER

tn H1
sfrq 399.883
tof 431.3
tpwr 60
pw 7.650

DECOUPLER

dn C13
dof 0
dm nnn
dnam C
dpwr 48
dmf 13156

SPECIAL

temp 30.0
gain not used
spin 20
bst 0.008
pw90 15.300
alfa 20.000

FLAGS

ll n
ln n
dp y
hs nn
fn not used

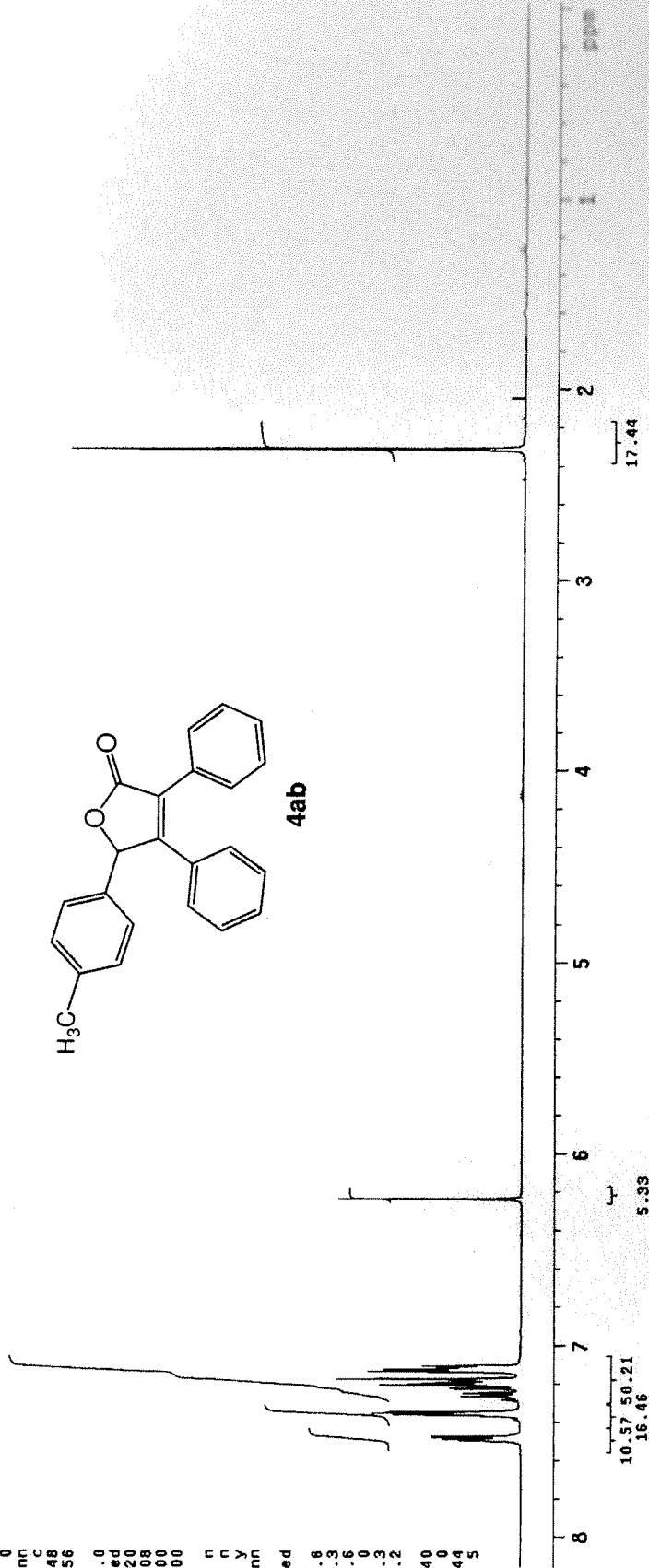
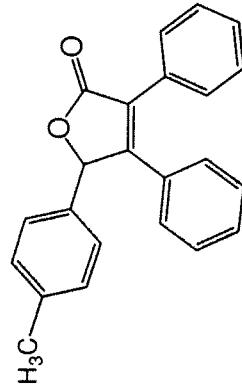
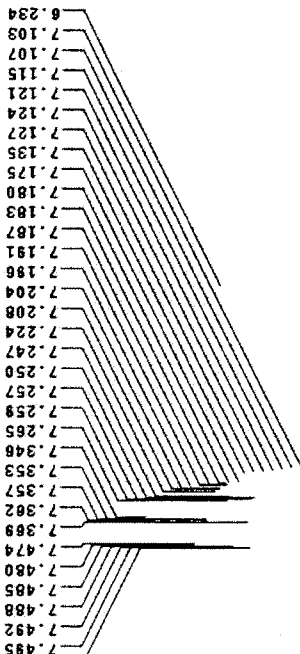
DISPLAY

sp -15.6
wp 3278.3
rf1 795.6
rfp 0
rp -55.3
lp -90.2

PLOT

wc 240
sc 0
vs 44
th 5
dl cdc ph

2.310



10.57 50.21
16.46

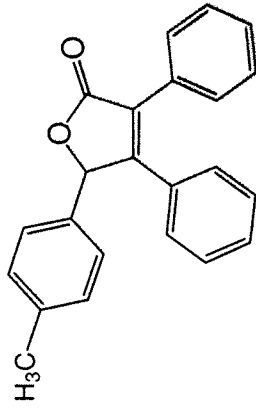
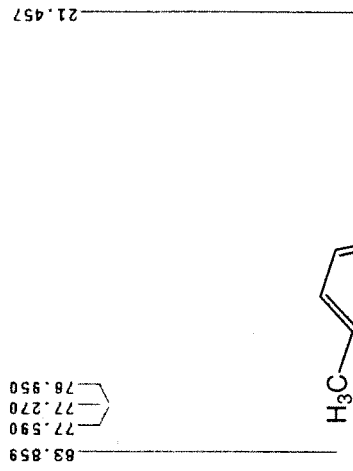
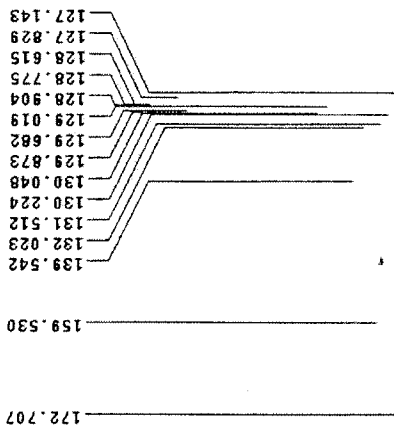
5.33

17.44

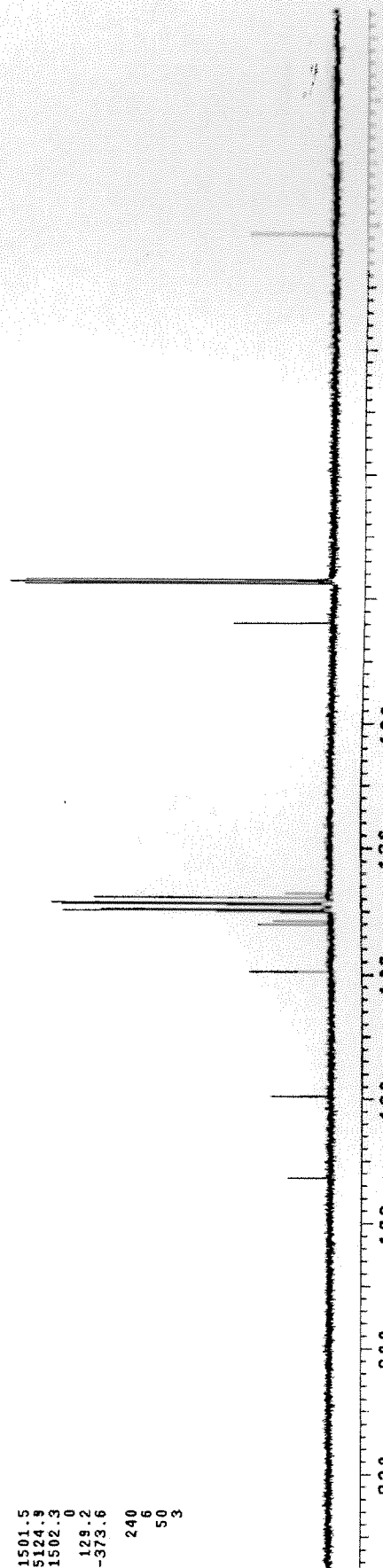
```

N85CRY5
exp3 s2pu1
SAMPLE
date Mar 26 2006
solvent CDC13
file
ACQUISITION exp
sw 25125.6
at 1.199
np 60270
fb 13600
bs 84
dl 2.000
nt 2000
ct 768
TRANSMITTER C13
tn
sfrq 100.561
tof 1553.6
lprw 60
pw 8.750
DECOUPLER H1
dn
dof 0
dm yyy
dmm w
dppw 46
dmf 12270
SPECIAL
temp 30.0
gain not used
spin 20
hst 0.008
pw90 17.500
alfa 20.000
FLAGS
ll n
ln n
dp y
hs nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -1501.5
wp 25124.9
rf1 1502.3
rfp 0
cp 128.2
lp -373.6
PLOT
wc 240
sc 6
vs 50
th 3
na no
ph

```



4ab



3N84

exp5 s2pu1

SAMPLE 4 2006

date Apr 4 2006

file CDC12

solvent CDCl3

ACQUISITION exp5

sw 6389.6

at 1.998

np 25528

fb not used

bs 16

d1 1.000

nt 8

ct 8

TRANSMITTER H1

tn H1

sfrq 399.883

tof 431.3

tpwr 60

pw 7.650

DECOUPLER C13

dn C13

dof 0

dm nnn

dmm C

dpwr 48

dmf 13156

SPECIAL

temp 30.0

gain not used

spin 20

hct 0.008

pw80 15.300

atfa 20.000

FLAGS

ll n

ln n

dp y

hs nn

PROCESSING

fn not used

DISPLAY

sp -378.5

wp 3573.6

rfl 828.4

rff 0

rfp -54.9

rp -91.3

lp

PLOT

wc 240

sc 0

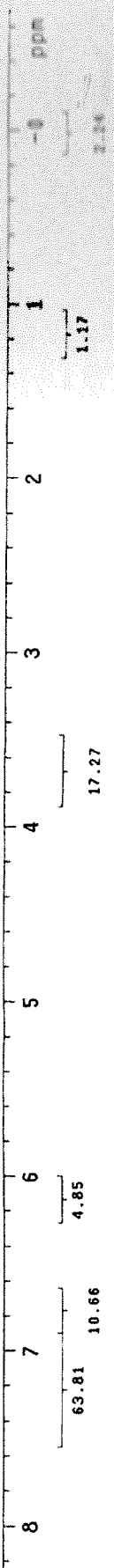
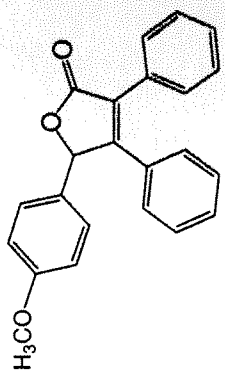
vs 70

th 5

al cdc

ph

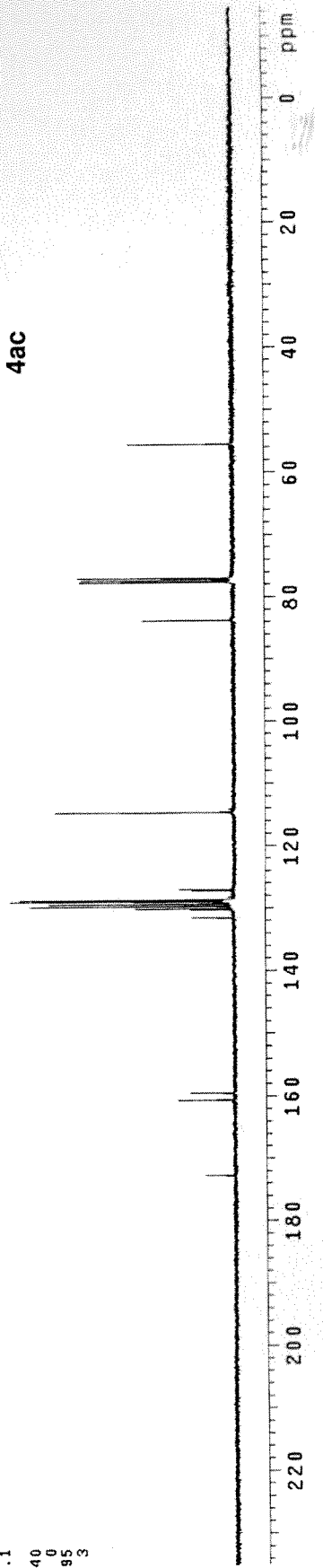
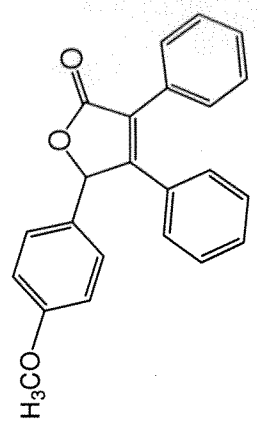
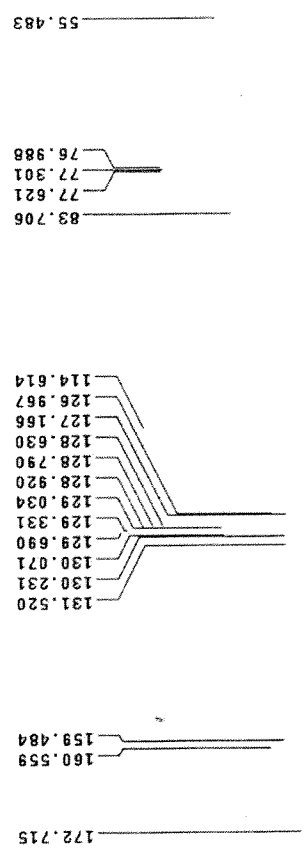
7.406
7.403
7.401
7.398
7.394
7.391
7.389
7.385
7.383
7.289
7.280
7.277
7.270
7.267
7.262
7.260
7.210
7.204
7.201
7.184
7.182
7.177
7.174
7.166
7.164
7.147
7.144
7.141
7.135
7.123
7.120
7.118
7.105
7.102
7.099
7.096
7.093
7.029
7.018
7.015
7.011
0.773
6.770
6.766
6.754
6.751
6.749
6.140
6.140
3.688
3.685
3.676
3.673



```

exp4 s2pul
SAMPLE
date Apr 15 2006
solvent CDC13
file ACQUISITION exp
SW 25125.6
at 1.198
np 60270
fb 13800
bs 64
d1 2.000
nt 2000
ct 2000
TRANSMITTER C13
tn 100.561
sfrq 1553.6
tof 60
tpwr 8.750
pw DECOUPLER H1
dn dof 0
dm dmm yyy w
dmm 46
dpwr 12270
dmf SPECIAL
temp 30.0
gain not used
spin 20
hst 0.008
pw90 17.500
alpha 20.000
FLAGS
ll n
ln n
dp y
hs nn
lb 1.00
fn not used
sp -1501.5
wp 25124.3
rfi 1502.3
rff 0
rp 124.5
lp -343.1
PLOT
wc 240
sc 0
vs 95
th 3
al no
ph

```



2N89

exp4 \$2pul

SAMPLE
 date Apr 5 2006
 solvent CDC13
 file /export/home/~
 vnmr1/CetInkaya/IH~
 -Experiment/2N89-1~
 H.fid

ACQUISITION
 sw 6389.8
 at 1.998
 np 25528
 fb not used
 bs 16
 d1 1.000
 nt 8
 ct 8

TRANSMITTER H1
 tn 399.883
 sfrq 431.3
 tof 60
 tpwr 7.650
 pw DECOUPLER C13

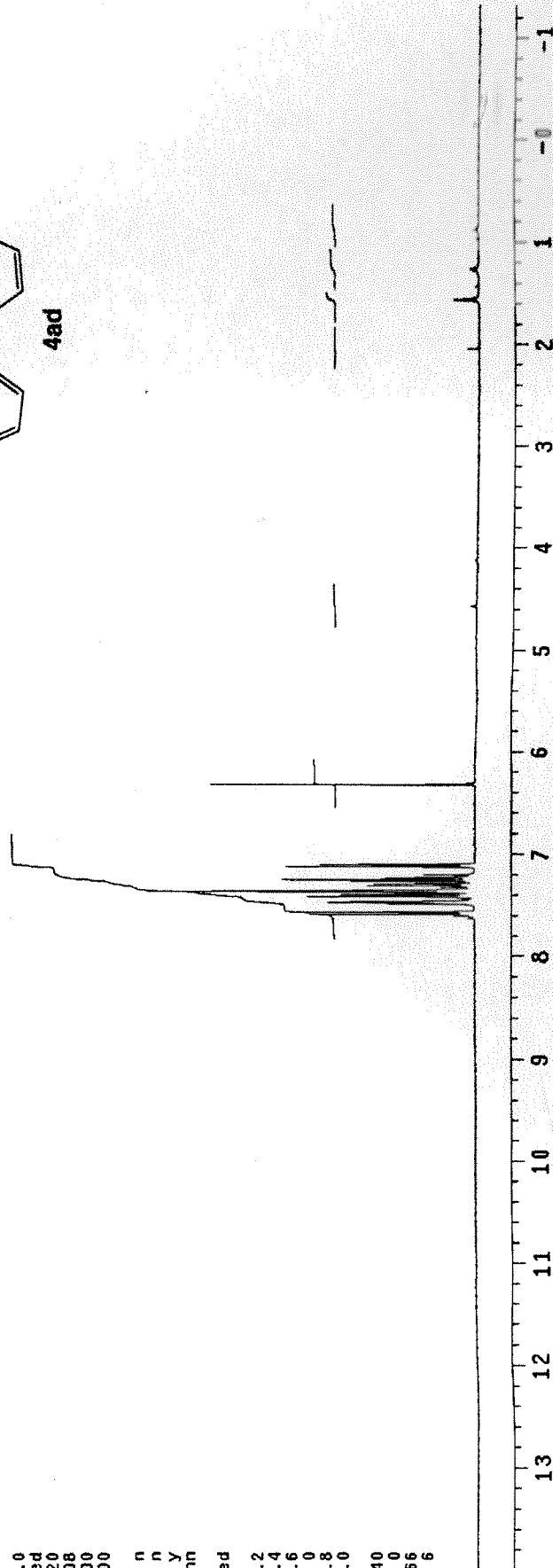
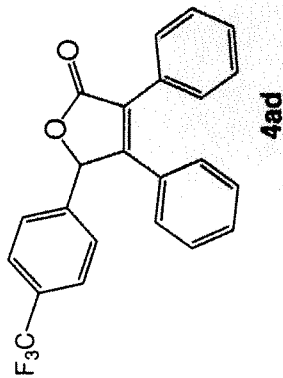
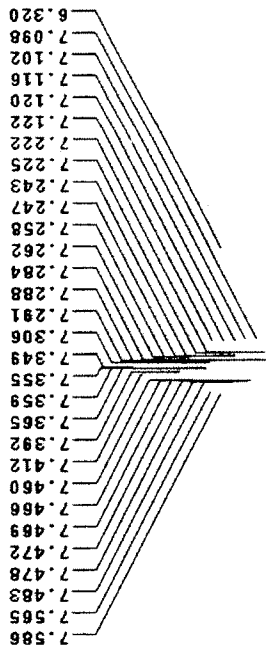
dn 0
 dof 0
 dm nnn
 dmn C
 dpwr 48
 dmf 13156

SPECIAL
 temp 30.0
 gain not used
 spin 20
 hst 0.008
 pw90 15.300
 alfa 20.000

FLAGS
 i1 n
 in n
 dp y
 hs nn
 fn not used

PROCESSING
 DISPLAY
 sp -795.2
 wp 6389.4
 rfl 795.6
 rfp 0
 rp -62.8
 lp -75.0

PLOT
 wc 240
 sc 0
 vs 66
 th 6
 al cdc ph



Y2M89

exp4 s2pul

SAMPLE
date Apr 15 2006
solvent CDCl3
file CDC13

ACQUISITION
sw 25125.6
at 1.189
np 60270
fb 13800
bs 64
dl 2.000
nt 2000
ct 1536

TRANSMITTER C13
tn
strq 100.561
tof 1553.6
tpwr 60
pw 8.750

DECOUPLER
dn H1
dof 0
dm VVY
dmm W
dmwr 46
dmf 12270

SPECIAL
temp 30.0
gain not used
spin 20
hst 0.008
pw90 17.500
alpha 20.000

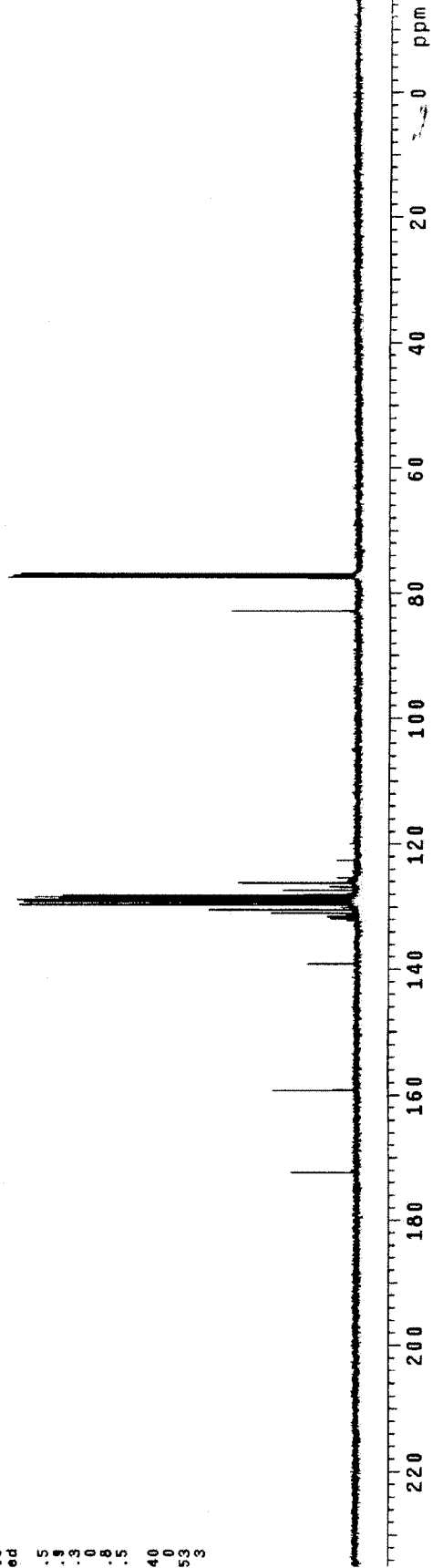
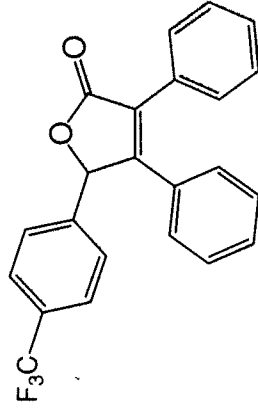
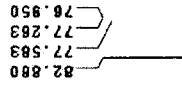
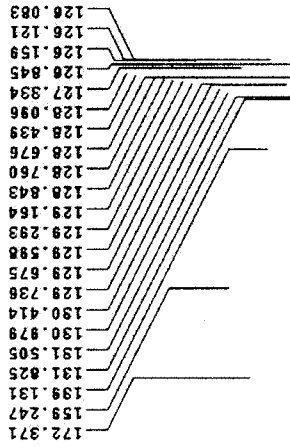
FLAGS
ll n
ln n
dp y
hs nn

PROCESSING
lb 1.00
fn not used

DISPLAY
sp 1501.5
wp 25124.3
rf1 1502.3
rfp 0
rp 137.8
lp -371.5

PLOT
wc 240
sc 0
vs 53
th 3

nm no ph



NURCLM
11 TEMGEZ 2007

Sample Name:
NURCAN-28159-PROT
Archive directory:
Sample directory:

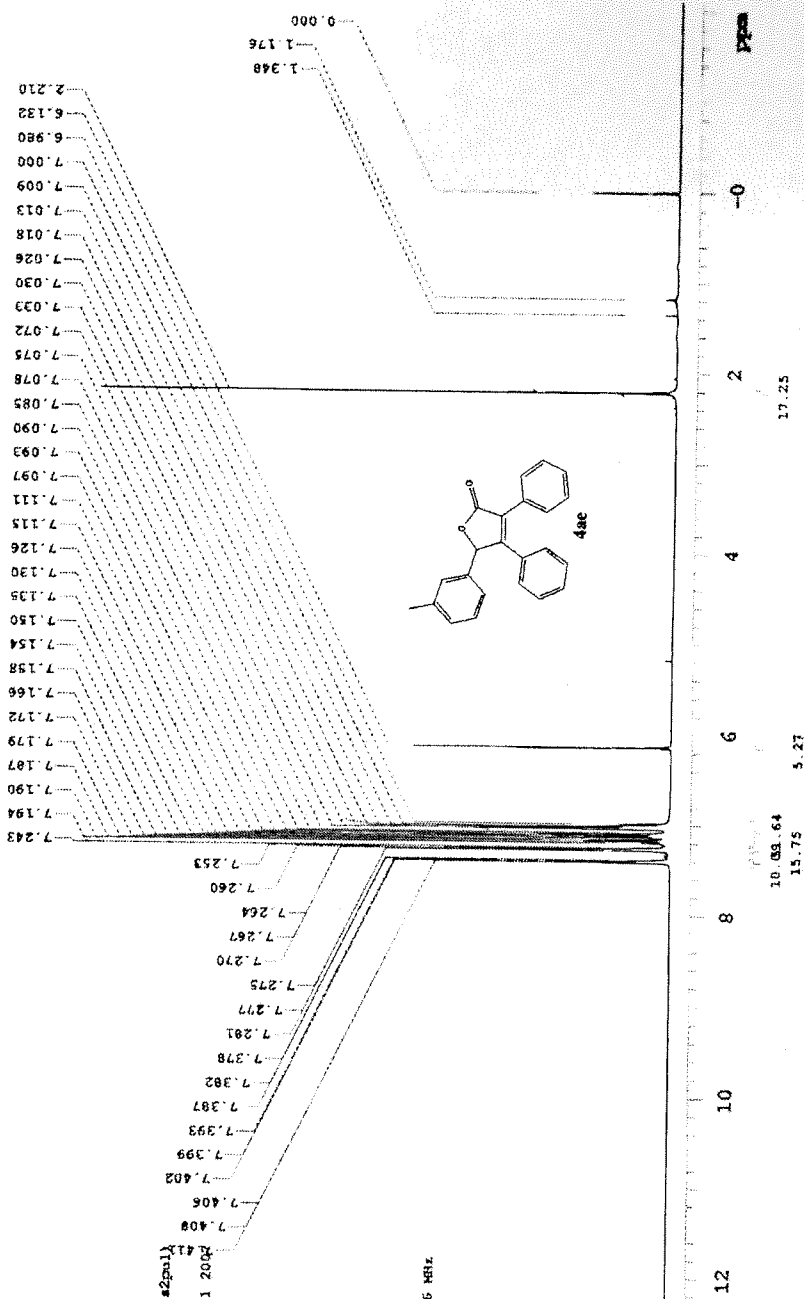
FidFile: Proton

Pulse Sequence: Proton (#2pul)
Solvent: CDCl3
Data collected on: Jul 11 2007

Operator: walkupl
VIMS-400 "nmr400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 6410.3 Hz
9 repetitions

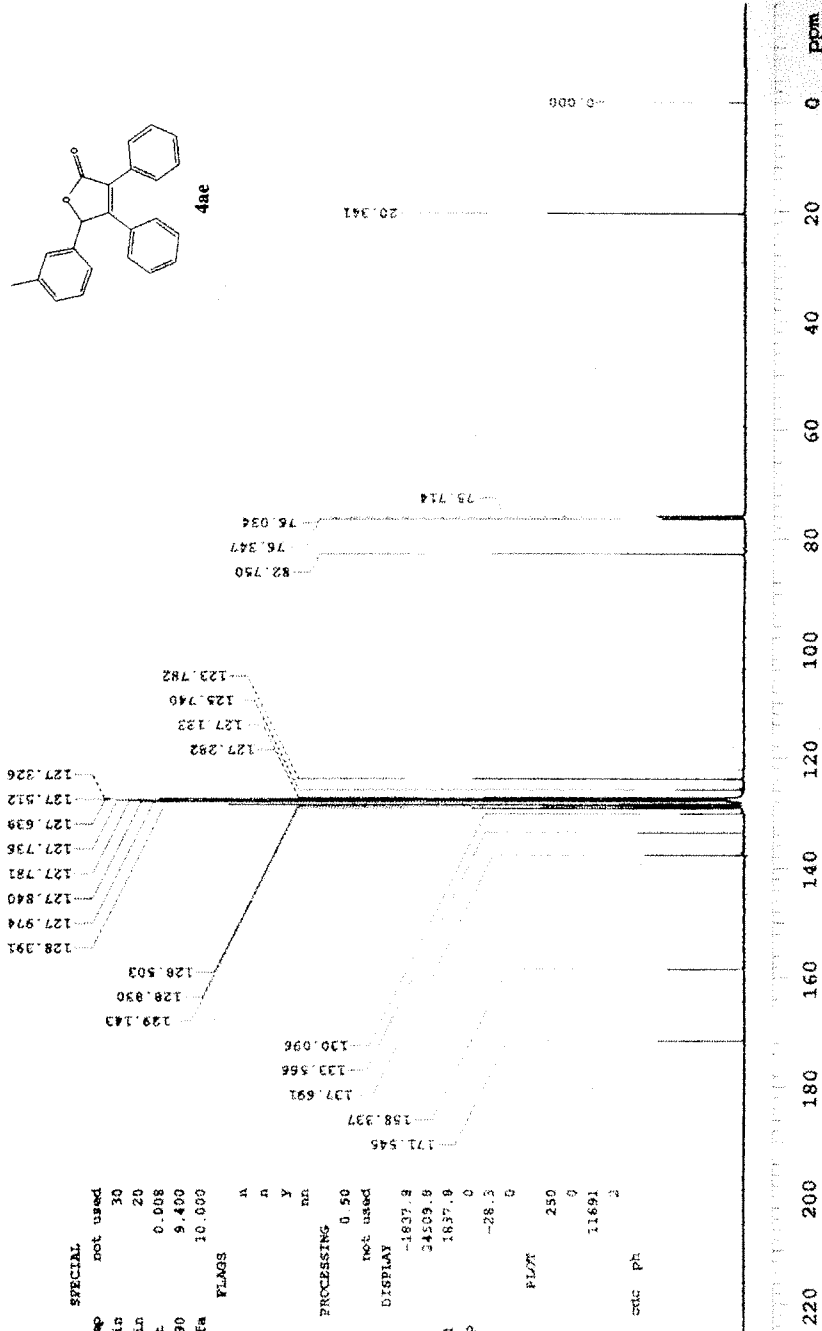
ORSEVE R1, 399.520266 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 9 min 34 sec



NUCCAN
 NUCCAN
 12 TEMEZ 2007

expl Carbon

SAMPLE SPECIAL
 data Jul 12 2007 temp not used
 solvent CDCl3 gain 30
 file /home/walkupl- spin 20
 /vmarray/data/ARTO- hat 0.008
 K GROUPE/NUCCAN/2KI- ps-90 9.400
 S9-PROT_12JUL2007/~ aifa 10.000
 Carbon_01.fid FLAGS
 ACQUISITION
 av 24503.8 in n
 at 1.300 dp y
 fp 63750 hs nn
 fb 17000 PROCESSING
 bs 64 lb 0.50
 cl 1.000 fc not used
 ct 512 DISPLAY
 ct 512 SF --1837.9
 TRANSMITTER
 tn 613 rfl 34509.9
 rfp 1837.9
 afpq 100.470 rfp 0
 tof 1041.9 fp -28.3
 tqwt 54 fp 0
 pw 4.700 PLWT
 DECOUPLER
 dc 250
 dh H1 x2 0
 dsf 0 vx 11691
 dm YFF th 2
 dmr N AL sdc ph
 dpwr 38
 chf 9400



NORCAS
13 TEMPIZ 2007

Sample Name:
CD155P-130707
Archive directory:

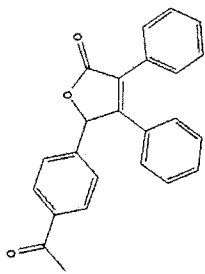
Sample directory:

Fidfile: Proton

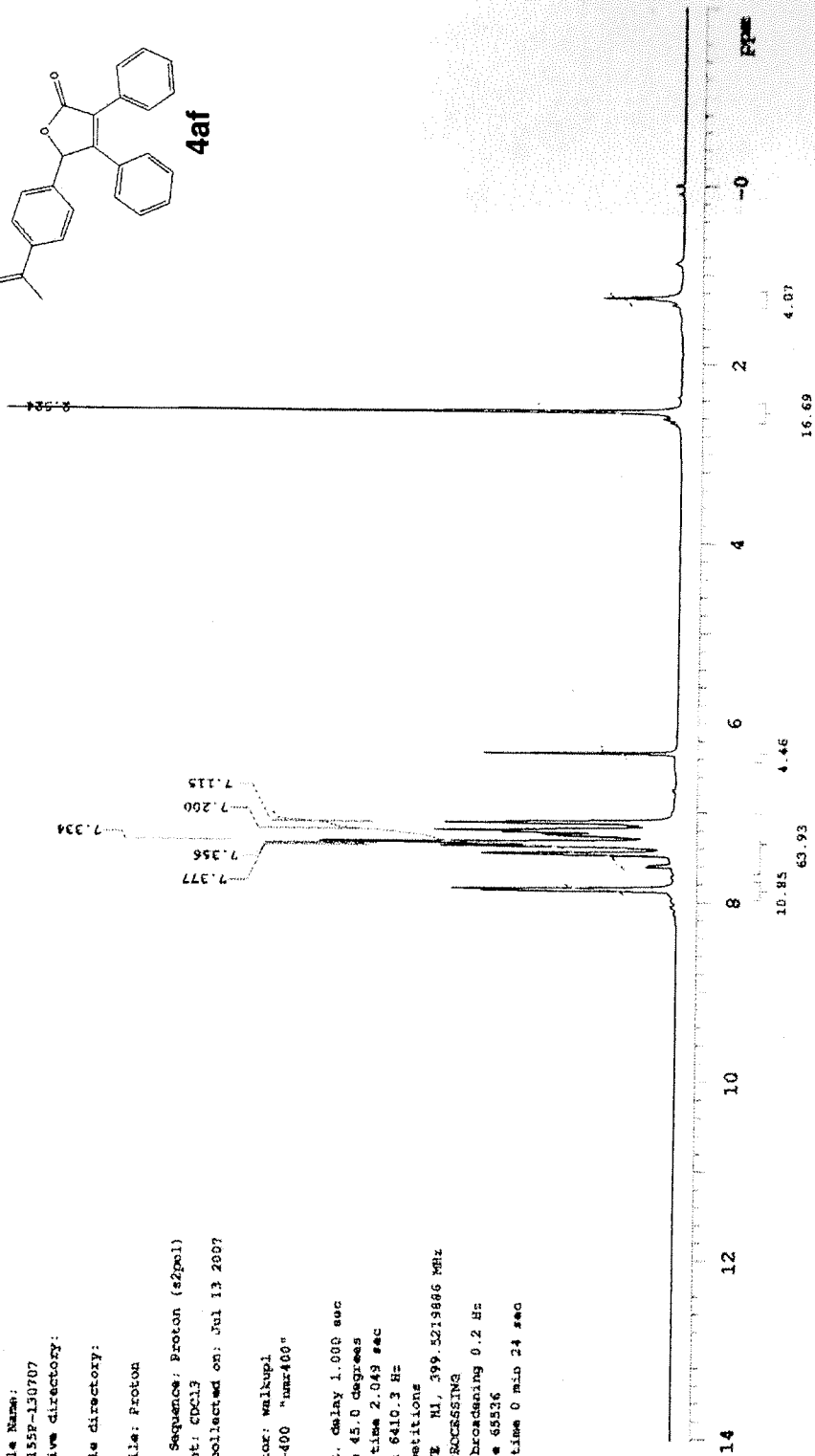
Pulse Sequence: Proton (s2pol)
Solvent: CDCl3
Data collected on: Jul 13 2007

Operator: walnupl
VNMR3-400 ¹Hmr400

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 6400.3 Hz
3 repetitions
OBSERVE M1, 399.5219866 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min 24 sec



4af



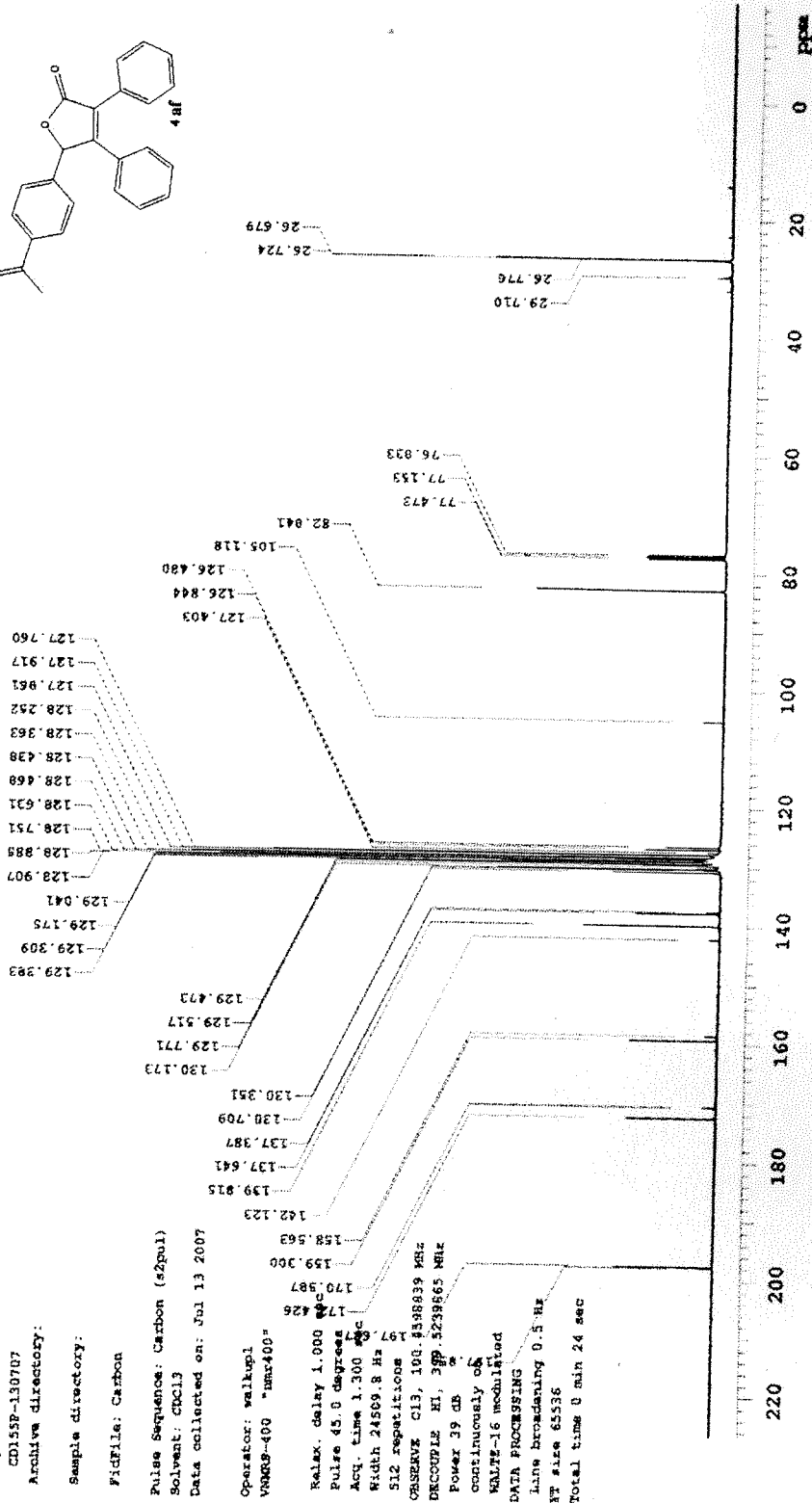
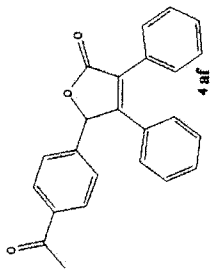
NURCAM
13 TEMAGUE 2007

Sample Name:
CD15P-130707
Archive directory:
Sample directory:
File: Carbon

Pulse Sequence: Carbon (s2pol)
Solvent: CDCl3
Data collected on: Jul 13 2007

Operator: walkupl
VNMRS-400 "hmr400"

Relax. delay 1.000
Pulse 45.0 degrees
Acq. time 1.300
Width 24509.8 Hz
512 repetitions
OBSERVE C13, 100.628399 MHz
DECOUPLE H1, 500.136465 MHz
Power 39 dB
continuously on
MATE-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
SI size 65536
Total time 0 min 24 sec



NUBCAS
13 TEMGEZ 2007

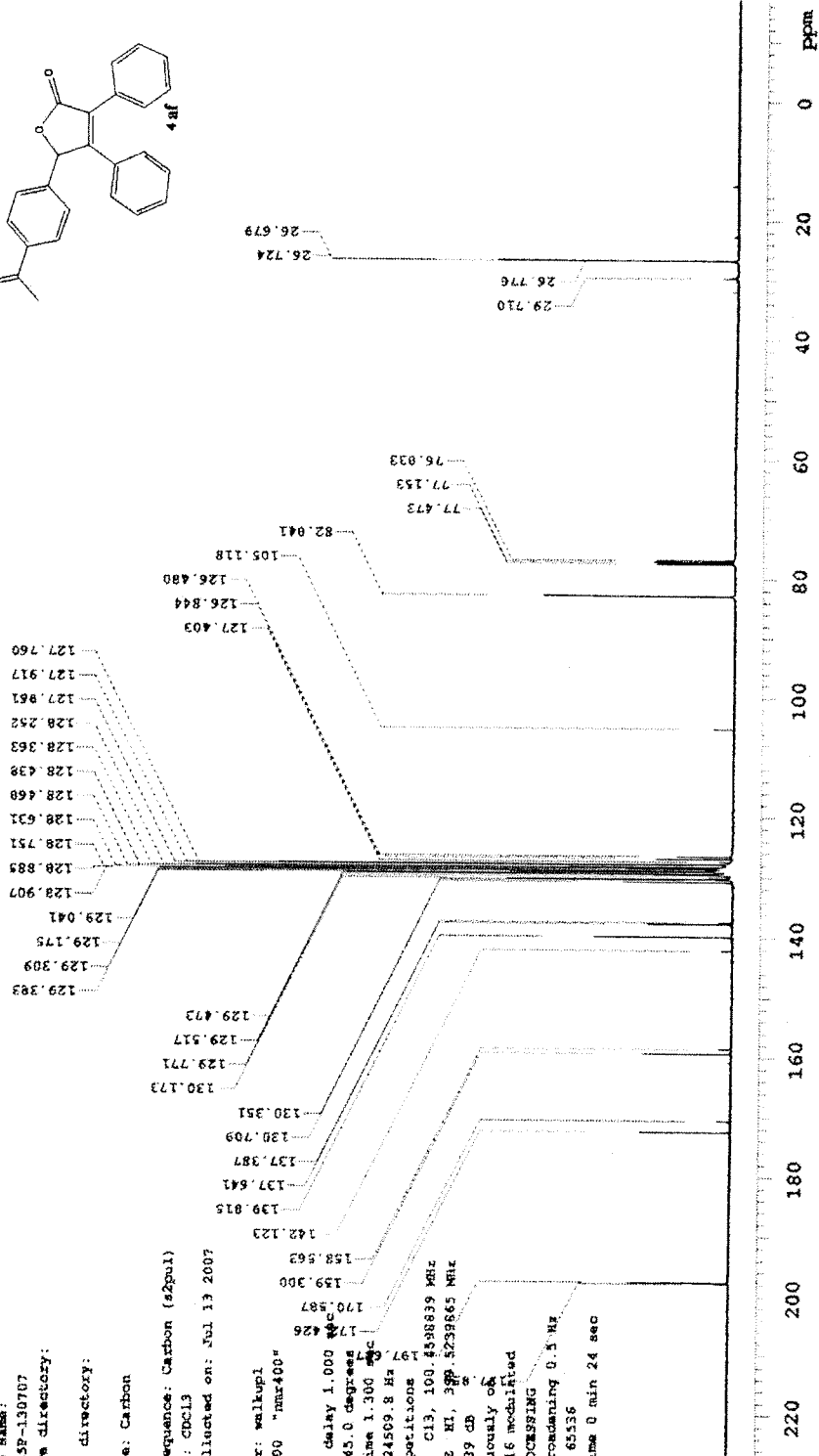
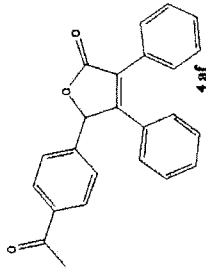
Sample Name:
CD152-130707
Archive directory:
Sample directory:

FidFile: Carbon

Pulse Sequence: Carbon (s2pul)
Solvent: CDCl3
Data collected on: Jul 13 2007

Operator: walkupl
VNMRS-400 "nmr400"

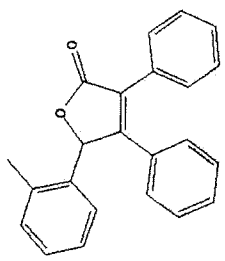
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
512 repetitions
OBSRVZ C13, 100.625839 MHz
DECOUPLE H1, 399.523585 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
ET size 65536
Total time 0 min 24 sec



```

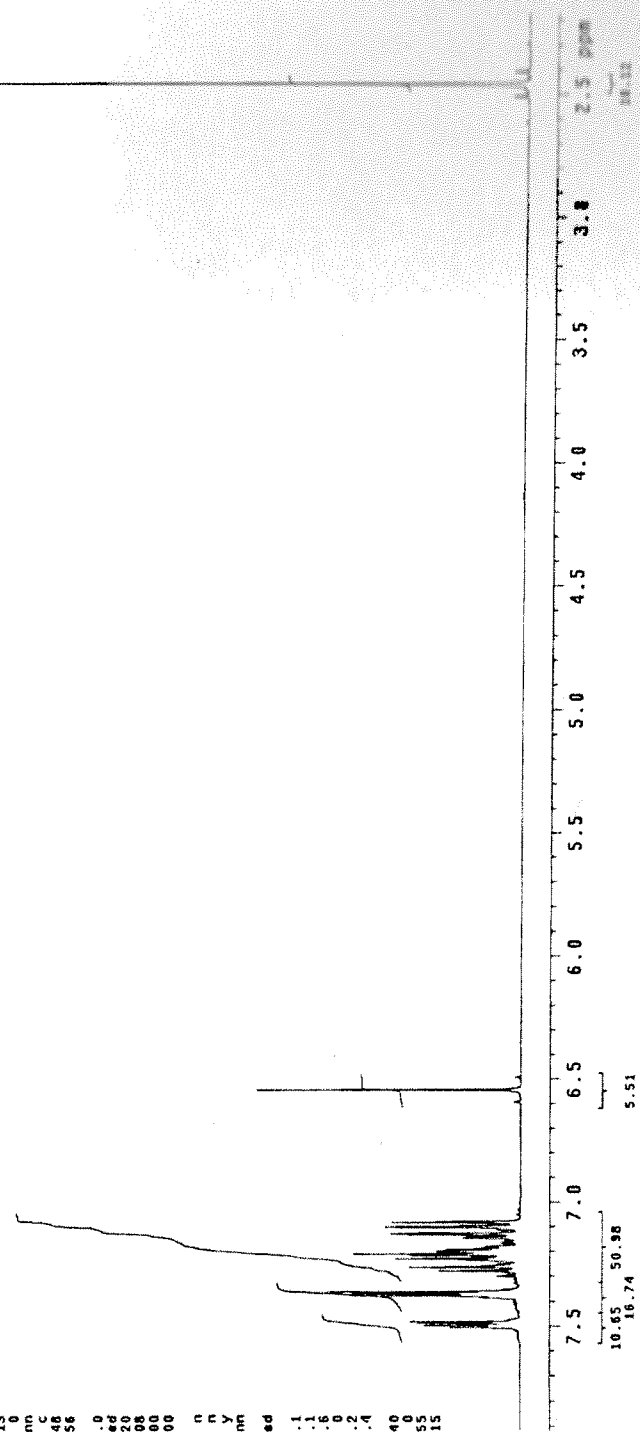
CD162PRO
exp# 32pul
SAMPLE
date Jan 13 2007
file CDC13
file ACQUISITION exp
sw 6389.8
at 1.98
np not used
fb not used
ds 18
st 1.00
ct 8
CT TRANSMITTER H1
ln 389.883
sfrq 431.3
tof 80
tpwr 7.650
PW DECOUPLER C13
dn 0
dof 0
dm nno
dm nno
dwm C
dipwr 40
def SPECIAL 13156
temp 30.0
caln not used
spin 20
hst 0.008
pw90 15.300
alfa 20.000
ALFA FLAGS
ll n
ln n
lp y
rs PROCESSING nm
fn not used
fn DISPLAY
fp 866.1
wp 2289.1
rf1 785.6
rfp 68.2
lp -81.4
PLOT
vc 240
ic 0
vs 55
ln cdc
al ph

```



2.456

7.486
7.487
7.481
7.478
7.375
7.367
7.361
7.284
7.229
7.214
7.209
7.190
7.126
7.103
7.100
7.003



NURCAN-CD162

Sample Name:

NURCAN-CD162

Data Collected on:

mar400-vwars400

Archive directory:

/home/walkupl/vwars400/data

Sample directory:

NURCAN-CD162_20Jun2008

FidFile: CARBON_01

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Jun 20 2008

Temp. 28.0 C / 301.1 K

Operator: walkupl

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.285 sec

Width 25310.2 Hz

512 repetitions

OBSERVE CH3, 100.4598839 MHz

DECOUPLE H1, 399.5239682 MHz

Power 39 dB

continuously on

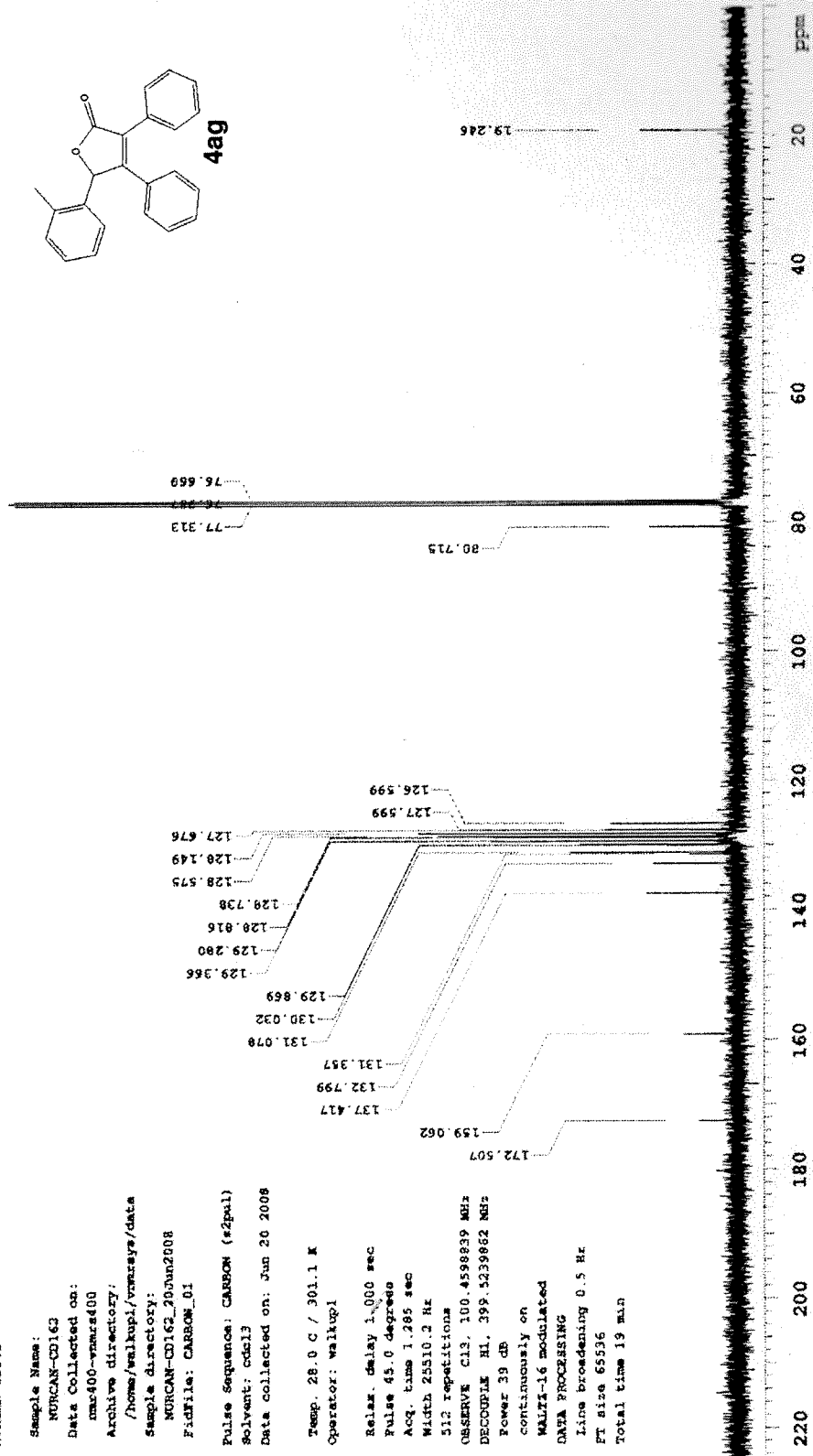
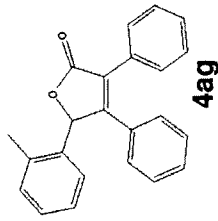
MULTI-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz

FI size 65536

Total time 19 min



4OCTYNE

exp3 s2pu1

SAMPLE 5 2006
 date Apr 5 2006
 solvent C6Cl6
 fltic C6Cl6
 sw ACQUISITION exp3
 6389.6
 at 1.998
 np 25528
 fb not used
 bs 16
 d1 1.000
 nt 8
 ct 8

TRANSMITTER H1
 tn 389.889
 sfrq 431.3
 tof 431.3
 tpr 7.650
 pw 7.650

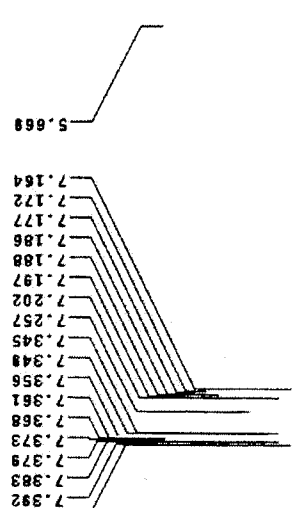
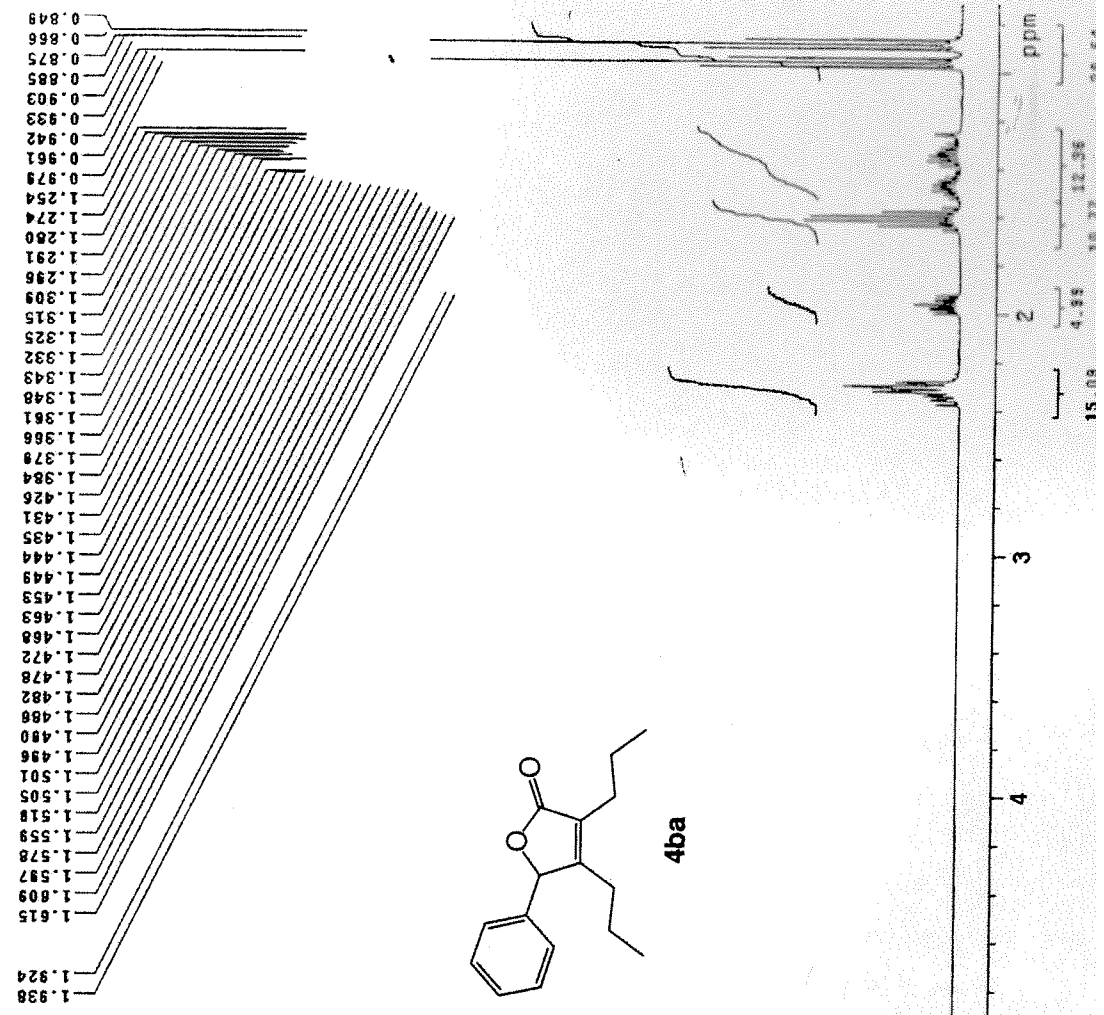
DECOUPLER C13
 dn 0
 dof 0
 dm nnn
 dnm C
 dpwr 48
 dmf 13156

SPECIAL
 temp 30.0
 gain not used
 spin 20
 hst 0.008
 pw90 15.300
 alfa 20.000

FLAGS
 ll n
 ln n
 dp y
 hs nn
 fn not used

PROCESSING
 not used
 DISPLAY
 sp 285.5
 wp 2824.8
 rrl 795.6
 rfp 0
 lp -54.7
 -81.9

PLOT
 wc 240
 sc 0
 vs 50
 th 1
 al cdc ph



ppm
 15.00
 4.88
 12.56
 13.65
 9.07
 4.42

4OCTYNE

```

exp3 s2pul
SAMPLE
date Apr 5 2006
solvent CDC13
file
ACQUISITION exp
sw 25125.6
at 1.198
mp 60270
fb 13800
bs 64
dl 2.000
nt 1000
ct 384
TRANSMITTER C13
tn
sffa 100.561
tof 1553.6
tpwr 60
pw DECOUPLER 8.750
dn dof HI
dm 0
dm vvy
dmm W
dpwr 46
dmf SPECIAL 12270
temp 30.0
gain not used
spin 20
hst 0.008
pw90 17.500
aifa 20.000
FLAGS
ll n
ln n
dp y
hs nm
lb 1.00
fn not used
PROCESSING
DISPLAY
sp -1501.5
wp 25124.9
rf1 1502.3
rff 0
rp 125.8
lp -369.9
PLOT
wc 240
sc 6
vs 60
th 5
nm no ph
  
```

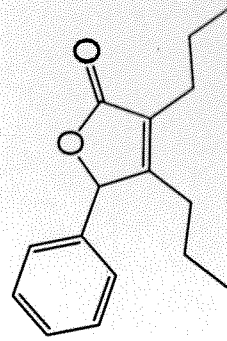
174.751

163.396

135.417
129.438
128.148
127.524
127.166

83.996
77.568
77.247
76.927

28.785
25.872
21.800
21.495
14.286
14.144



4ba

042281460807

Name:

Sample Name:

042281460807

Archive directory:

5 N

Sample directory:

1 7

File:

042281460807

File:

042281460807

File:

042281460807

File:

042281460807

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042281460807

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042281460807

File:

042281460807

File:

042281460807

File:

042281460807

File:

042281460807

False Sequence: Proton (24gall)

Solvent: cdcl3

Data collected on: Aug 6 2007

Temp: 27.0 / 300.1 K

Operator: walkup

VNMR: 400 "nmr400"

Pulse: delay 1.000 sec

Pulse: 45.0 degrees

Acq: time 2.049 sec

Width: 6419.2 Hz

6 resolutions

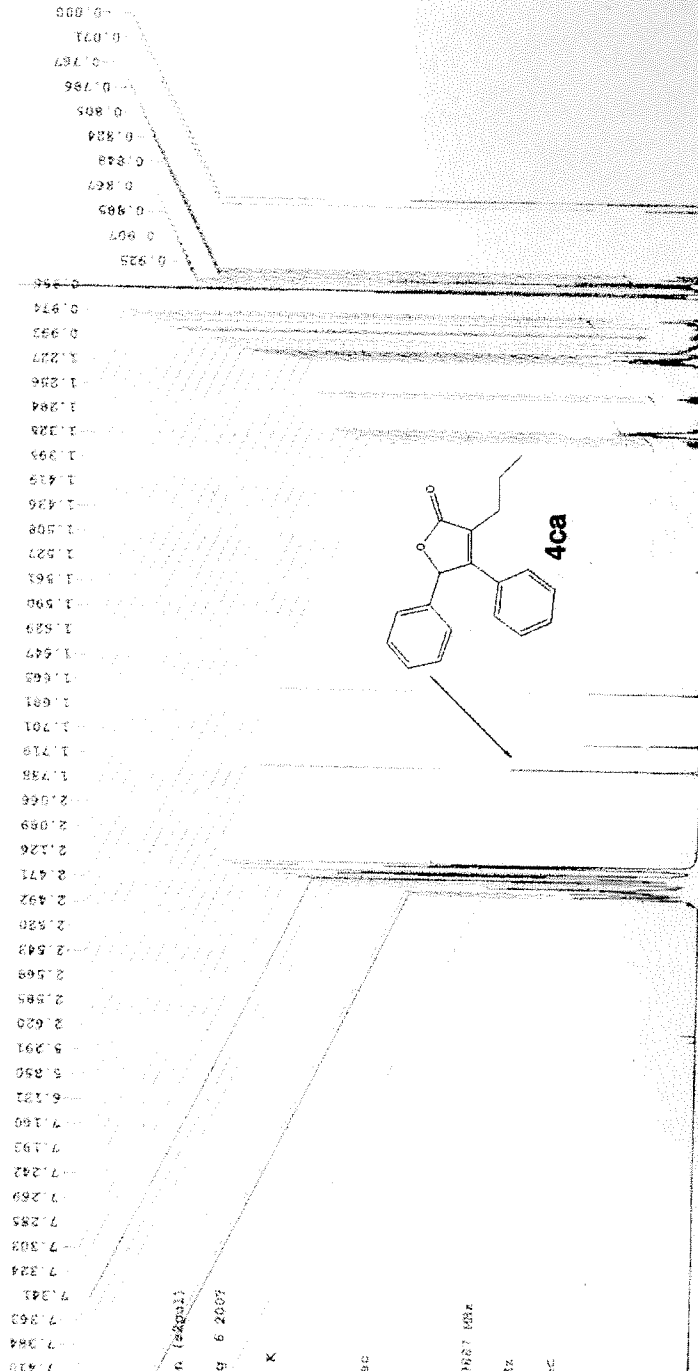
PROBHD: H1 5mm 5219627 68H

DATA PROCESSING

Time: 0.2 Hz

FT size: 65536

Total time: 0 min 24 sec



14

12

10

8

6

4

2

0

ppm

49.44

1.00

17.60

9.12

3.82

7.64

9.76

NMRCLM
 NMRSCAN
 19 FEBRUZ 2007

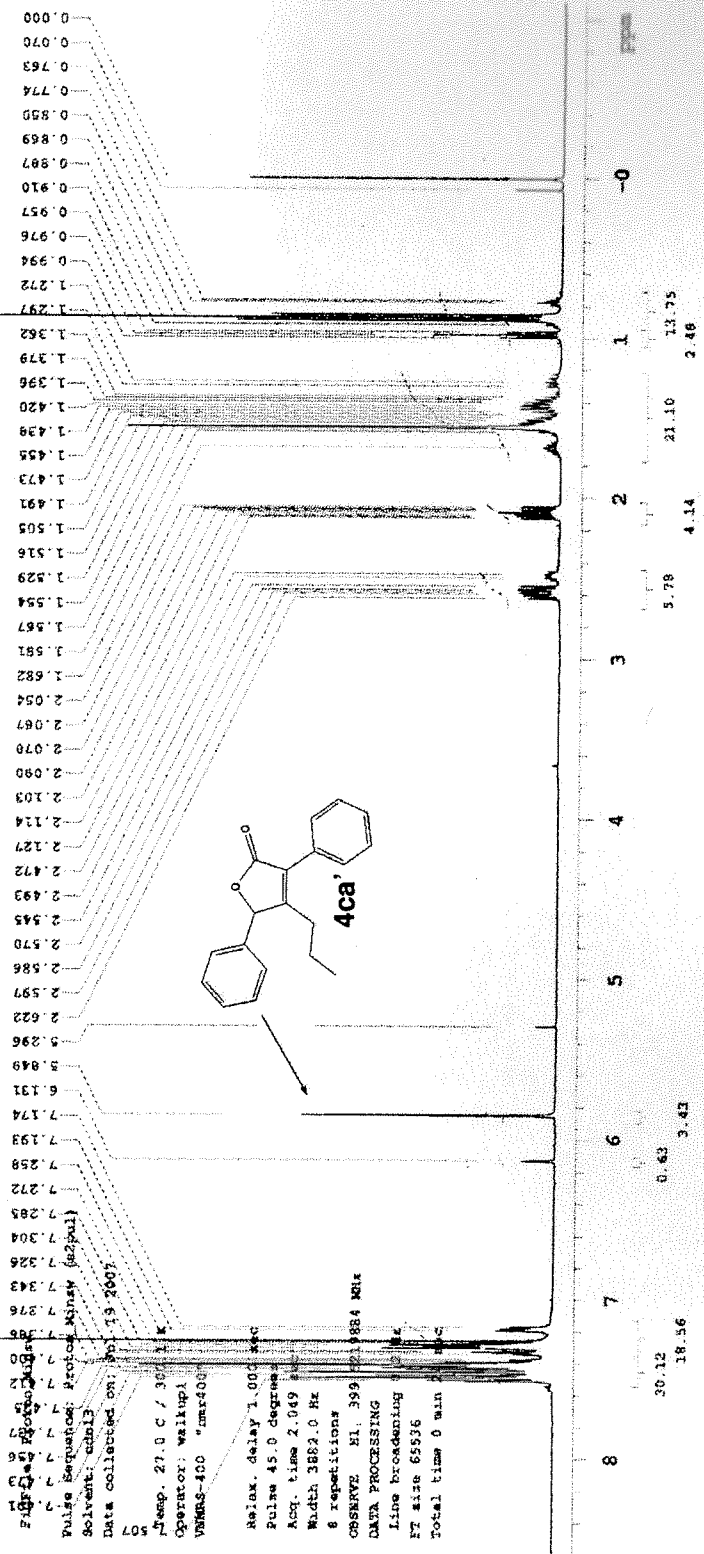
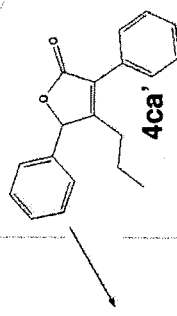
Sample Name:
 CD2812-190707
 Archive directory:

Sample directory:

1 0.000
 2 0.070
 3 0.763
 4 0.774
 5 0.850
 6 0.869
 7 0.887
 8 0.910
 9 0.957
 10 0.976
 11 0.994
 12 1.272
 13 1.297
 14 1.362
 15 1.379
 16 1.396
 17 1.420
 18 1.439
 19 1.455
 20 1.473
 21 1.491
 22 1.505
 23 1.516
 24 1.529
 25 1.554
 26 1.567
 27 1.581
 28 1.582
 29 2.054
 30 2.067
 31 2.070
 32 2.090
 33 2.103
 34 2.114
 35 2.127
 36 2.472
 37 2.493
 38 2.545
 39 2.570
 40 2.586
 41 2.597
 42 2.622
 43 5.296
 44 5.849
 45 6.131
 46 7.174
 47 7.193
 48 7.259
 49 7.272
 50 7.285
 51 7.304
 52 7.326
 53 7.343
 54 7.376
 55 7.389
 56 7.400

Data collected on: 19 FEB 2007
 Temp: 27.0 C / 80.6 F
 Operator: walkup
 VMS-100 "umr400"

Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.049 min
 Width 3852.0 Hz
 8 repetitions
 OBSERVE HI, 399.1384 MHz
 DATA PROCESSING
 Line broadening
 FT size 65536
 Total time 0 min 20 sec



Retention Time	Integration
0.000	
0.070	
0.763	
0.774	
0.850	
0.869	
0.887	
0.910	
0.957	
0.976	
0.994	
1.272	
1.297	
1.362	
1.379	
1.396	
1.420	
1.439	21.10
1.455	13.75
1.473	
1.491	
1.505	
1.516	
1.529	
1.554	5.78
1.567	
1.581	
1.582	
2.054	
2.067	
2.070	
2.090	
2.103	
2.114	
2.127	
2.472	
2.493	
2.545	
2.570	
2.586	
2.597	
2.622	
5.296	
5.849	
6.131	
7.174	
7.193	
7.259	
7.272	0.63
7.285	
7.304	
7.326	
7.343	
7.376	
7.389	
7.400	
8.000	

ND-T2C77KP2
3 ARALIK 2007

Sample Name:
ND-T2C77KP2-3ARALIK2007

Archive directory:

Sample directory:

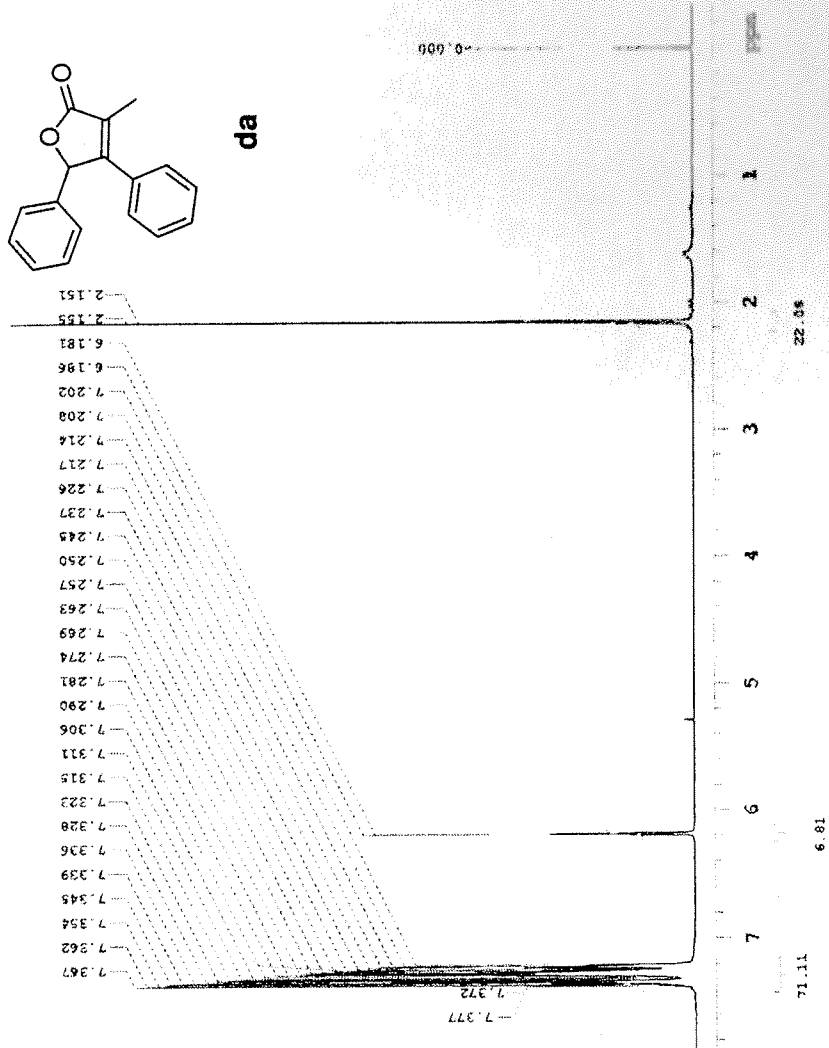
Fidfile: Proton

Pulse Sequence: Proton (s2gal)
Solvent: cdcl3
Data collected on: Dec 3 2007

Temp. 23.0 C / 298.1 K
Operator: walkup1
VMSRS-400 "mar400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.349 sec
Width 6410.3 Hz
8 repetitions

CROSSVE HI, 399.5219859 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min 24 sec



ND-72C77RP2
3 ARALIK 2007

Sample Name:
ND-72C77RP2-3ARALIK2007
Archive directory:

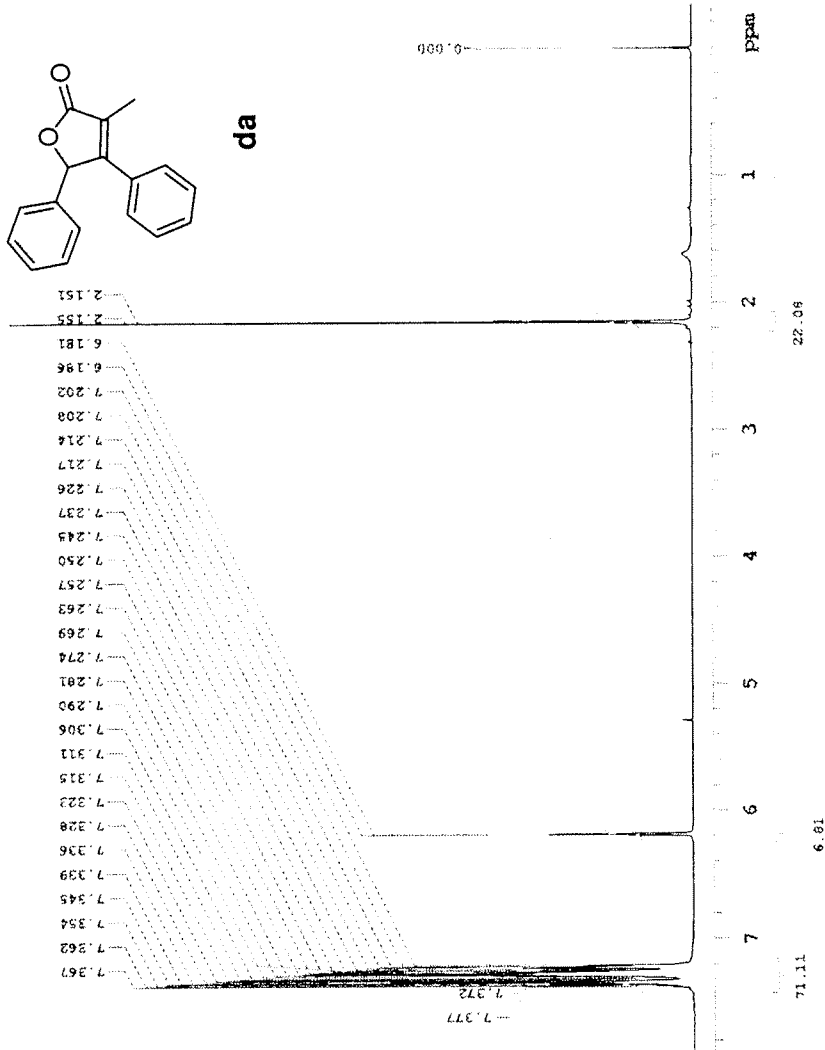
Sample directory:

Fidfile: Proton

Pulse Sequence: Proton (s2pol)
Solvent: cdc13
Data collection on: Dec 3 2007

Temp: 25.0 C / 298.1 K
Operator: walkup
VNMRS-400 "hmr400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.349 sec
Width 6410.0 Hz
8 repetitions
OBSERVE H1, 399.5219889 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min 24 sec



ND-7407462
J ADALIK 2007

Sample Name:
ND-7407462-1A2ALIK2007
Archive directory:
Sample directory:

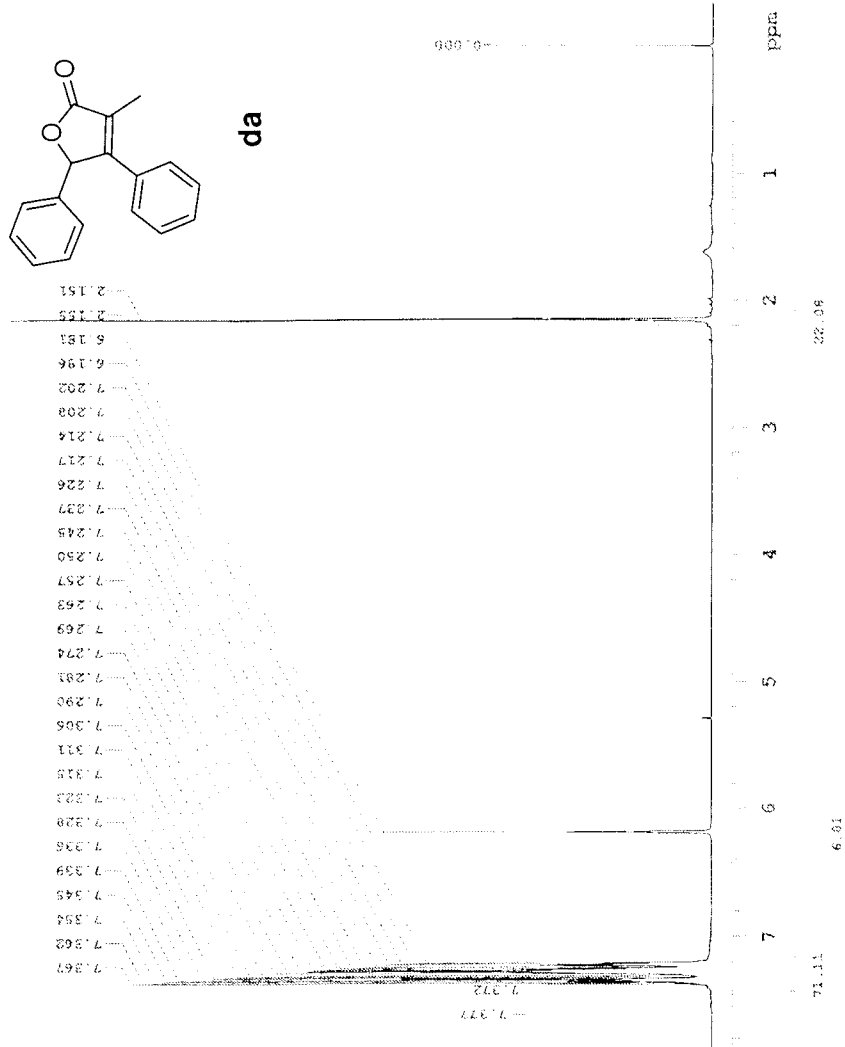
Yield: Proton

Pulse Sequence: Proton (s2ps1)
Solvent: CDCl3
Data collected on: Dec 3 2007

Temp: 25.0 C / 298.1 K
Operator: waikepi
PulsePRO: 400 "tunc100"

Relax. delay: 1.000 sec
Pulse: 49.0 degrees
Acq. time: 2.042 sec
Width: 6419.7 Hz

CHSEYE: 01.159.823003 Mr.
DATA PROCESSING
F1: size 65516
Total time 0 min 24 sec



ND-T2C77NP2
3 ARALIK 2007

Sample Name:
ND-T2C77NP2-3ARALIK2007

Archive directory:

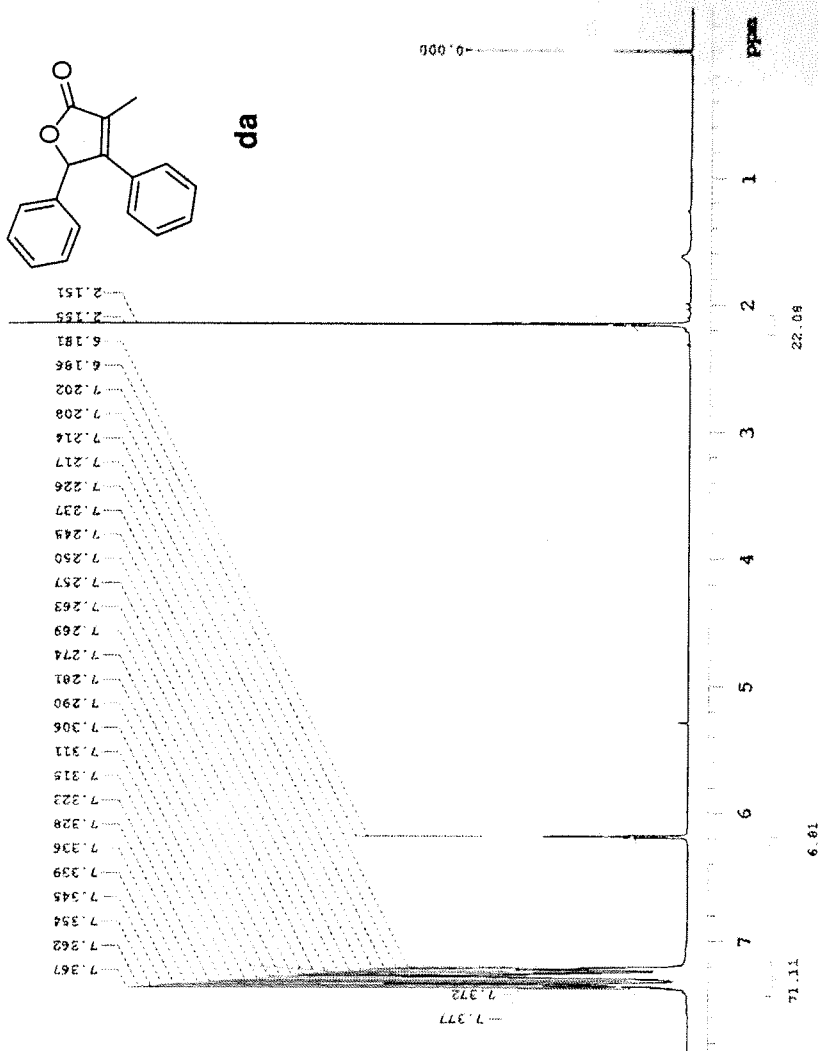
Sample directory:

File: Proton

Pulse Sequence: Proton (s2pul)
Solvent: CDCl3
Data collected on: Dec 3 2007

Temp: 23.0 C / 298.1 K
Operator: walikpi
VNMRS-400 "umr400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.043 sec
Width 6419.3 Hz
6 repetitions
ORSSVE H1 399.5219889 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min 24 sec



MD-T2C77KP2
 ND-T2C77KP2
 3 ARALIK 2007

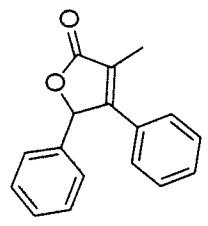
Sample Name:
 ND-T2C77KP2-3ARALIK2007
 Archive directory:

Sample directory:
 File: Carbon

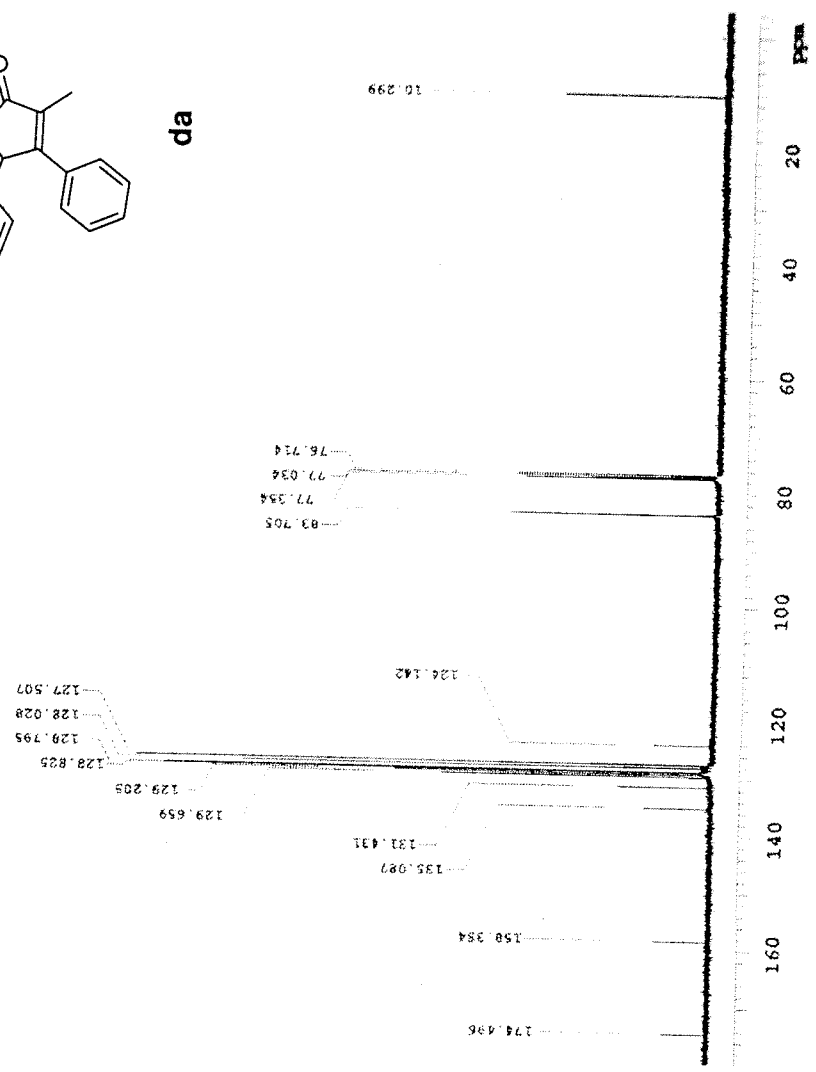
Pulse Sequence: Carbon (s2full)
 Solvent: cdcl3
 Data collected on: Dec 3 2007

Temp: 25.0 C / 298.1 K
 Operator: walkup
 VNP65-400 "umrf60"

Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.500 sec
 Width 24500.0 Hz
 SIZ 8192
 OBSERVE CH 100 4598639 MHz
 CHANNEL F1 399.5219865 MHz
 Power 39 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line Broadening 0.5 Hz
 FT size 65536
 Total time 0 min 14 sec



da



ND-T2C7M2K01
3 APRIL 2007

Sample Name:
ND-T2C7M2K01-3APRIL2007
Archive directory:

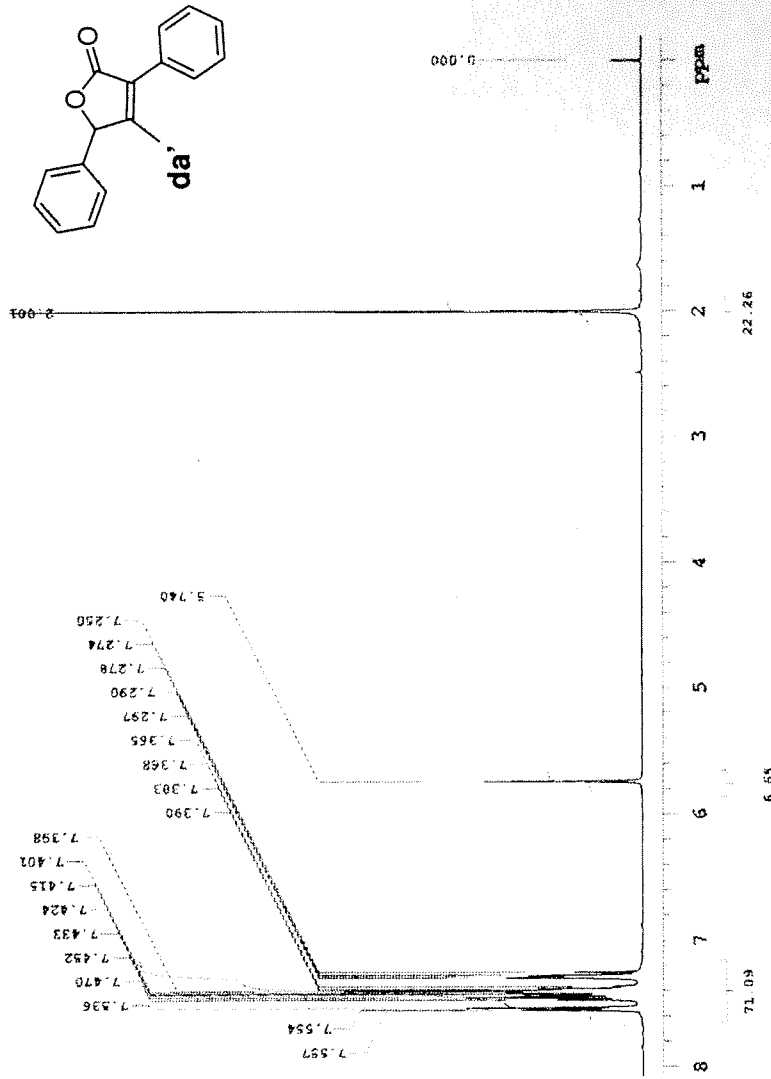
Sample directory:

FidFile: Proton

Pulse Sequence: Proton (42pul)
Solvent: cdcl3
Data collected on: Dec 3 2007

Temp: 25.0 C / 298.1 K
Operator: wallupi
VNS-400 "mir400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.521914 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65576
Total time 0 min 24 sec



ND-T2C77TIND1
ND-T2C77ZKRF1
3 ARALIK 2007

Sample Name:
ND-T2C77ZKRF1-2ARALIK2007
Archive directory:

Sample directory:

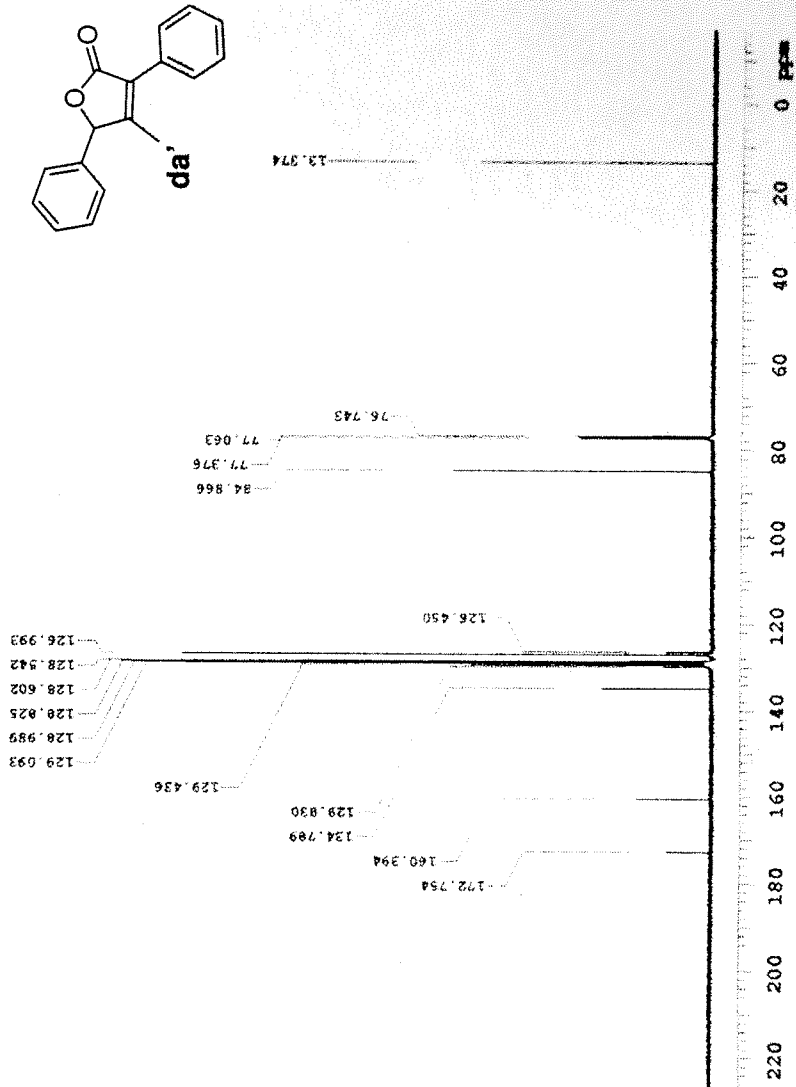
FidFile: Carbon

Pulse Sequence: Carbon (s2pral)
Solvent: cdcl3
Data collected on: Dec 3 2007

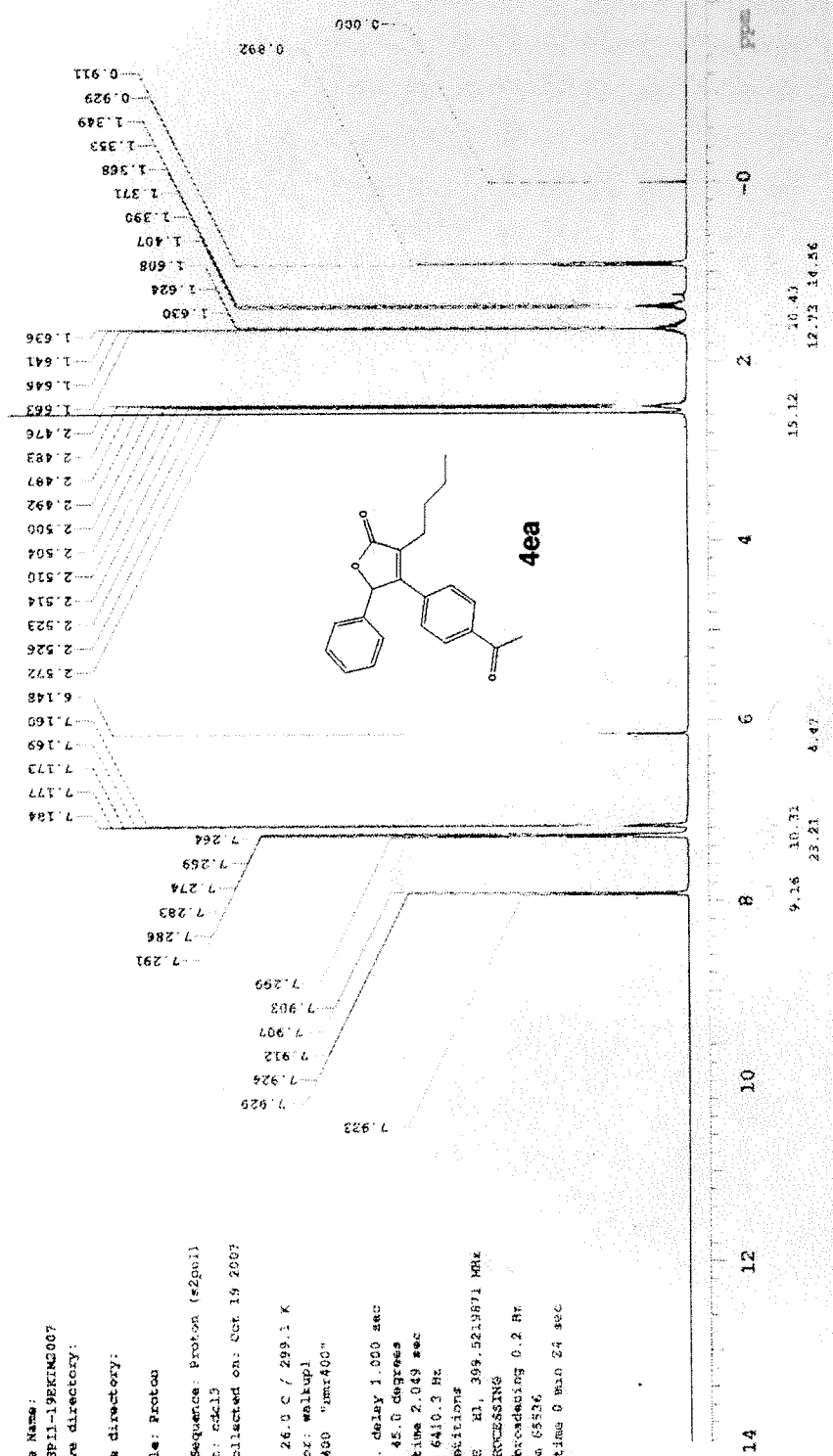
Temp. 25.0 C / 298.1 K
Operator: walshpl
VMAS-400 "mar400"

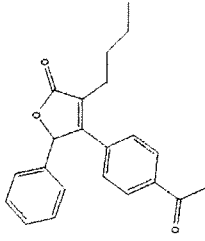
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24509.0 Hz
Siz 128 repetitions
OBSERVE CH3, 100.4598829 MHz
DECOUPLE H1, 399.5239865 MHz
Power 39 dB

continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 0 min 34 sec

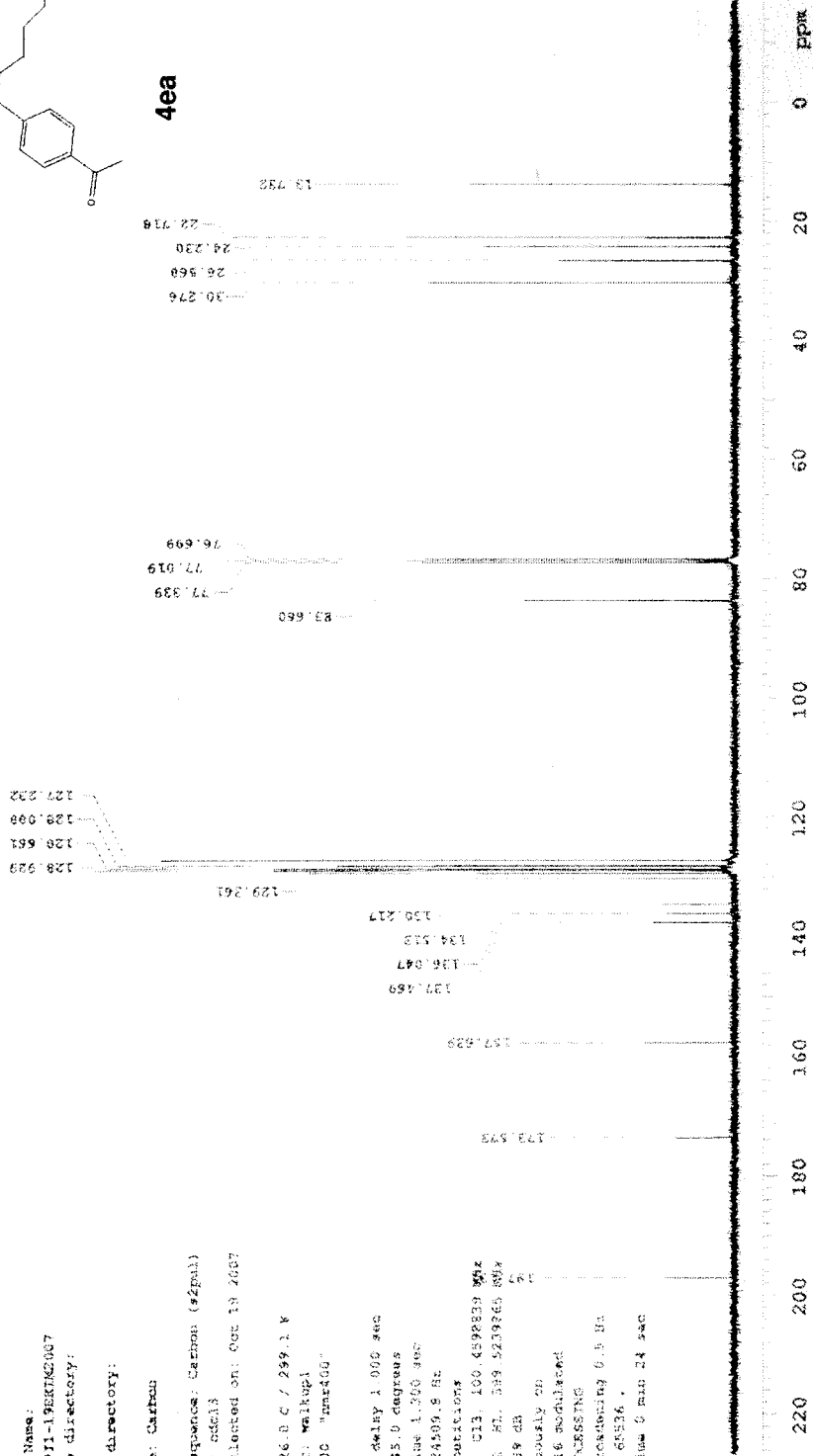


C203P11
 19 EKIM 2007
 Sample Name:
 C203P11-19KIM007
 Archive directory:
 Sample directory:
 Fidfile: Protou
 Pulse Sequence: Proton (s2pu1)
 Solvent: cdcl3
 Data collected on: Oct 16 2007
 Temp: 26.0 C / 299.1 K
 Operator: walkup
 VNAME: 400 "hmr400"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.049 sec
 Width 6410.3 Hz
 8 repetitions
 OBSERVE H1, 309.5219871 MHz
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 65536
 Total time 0 min 24 sec





4ea



C283P11
 C283P11
 19 EKIM 2007
 Sample Name:
 C283P11-19EKIM2007
 Archive directory:
 Sample directory:
 Fidfile: Carbon
 Pulse Sequence: Carbon (s2pul)
 Solvent: cdcl3
 Data collected on: Oct 19 2007
 Temp: 26.0 C / 259.1 K
 Operator: walkup
 VMMS-400 "mm400"
 Relax delay 1.000 sec
 Pulse 45.0 degrees
 Acq time 1.700 sec
 Width 24500.8 Hz
 512 Repetitions
 OPERVE C13 100.4998339 MHz
 DECOUPLE PL 399.5239265 MHz
 Fuser 19 dB
 continuously on
 WAITR-16 modulated
 DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 65536
 Total time 0 min 24 sec

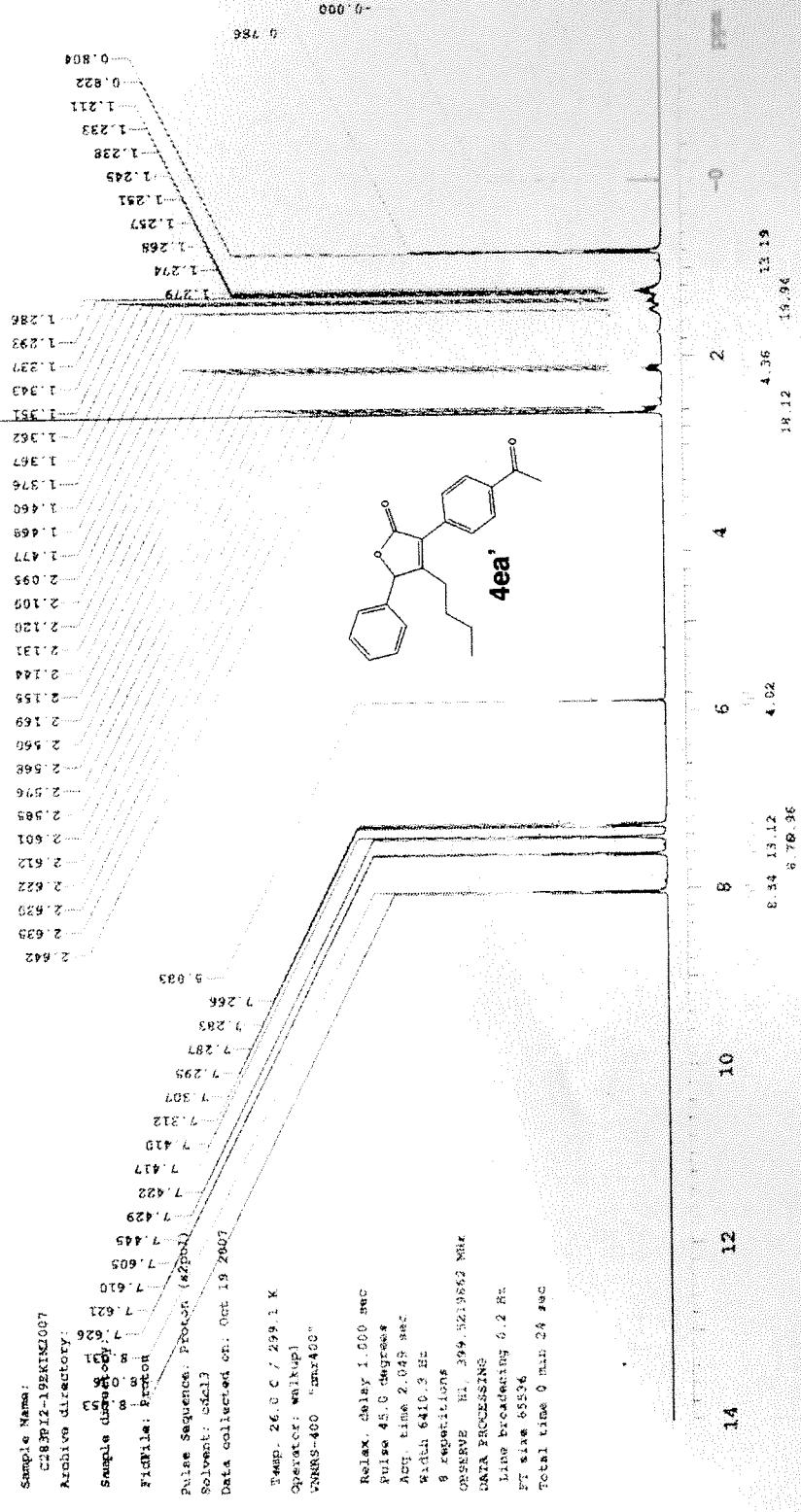
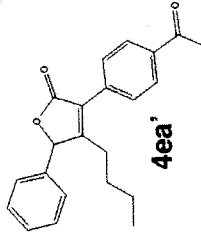
C290F12
19 SEIM 2007

Sample Name:
C283P12-19SEIM1007
Archive directory:
C:\MSDCHEM\1907\190712\290712\19SEIM1007
Sample ID:
File: E:\Data\190712\290712\19SEIM1007

Pulse Sequence: PROTON (42) (pph)
Solvent: dmf-d6
Data collected on: Oct 19 2007

Temp: 26.0 C / 299.1 K
Operator: walkup
VNRMS-400 "mxa400"

Relax. delay: 1.000 sec
Pulse: 45.0 degrees
Acq. time: 2.043 sec
Width: 640.0 Hz
9 repetitions
ORIGIN: N1, 399, 62, 9582 MHz
DATA PROCESSING
Line broadening: 6.2 Hz
FT size: 65536
Total time: 0 min 24 sec



C283P12
C283P12
19 EKIM 2007

Sample Name:
C283P12-19EKIM2007
Archive directory:

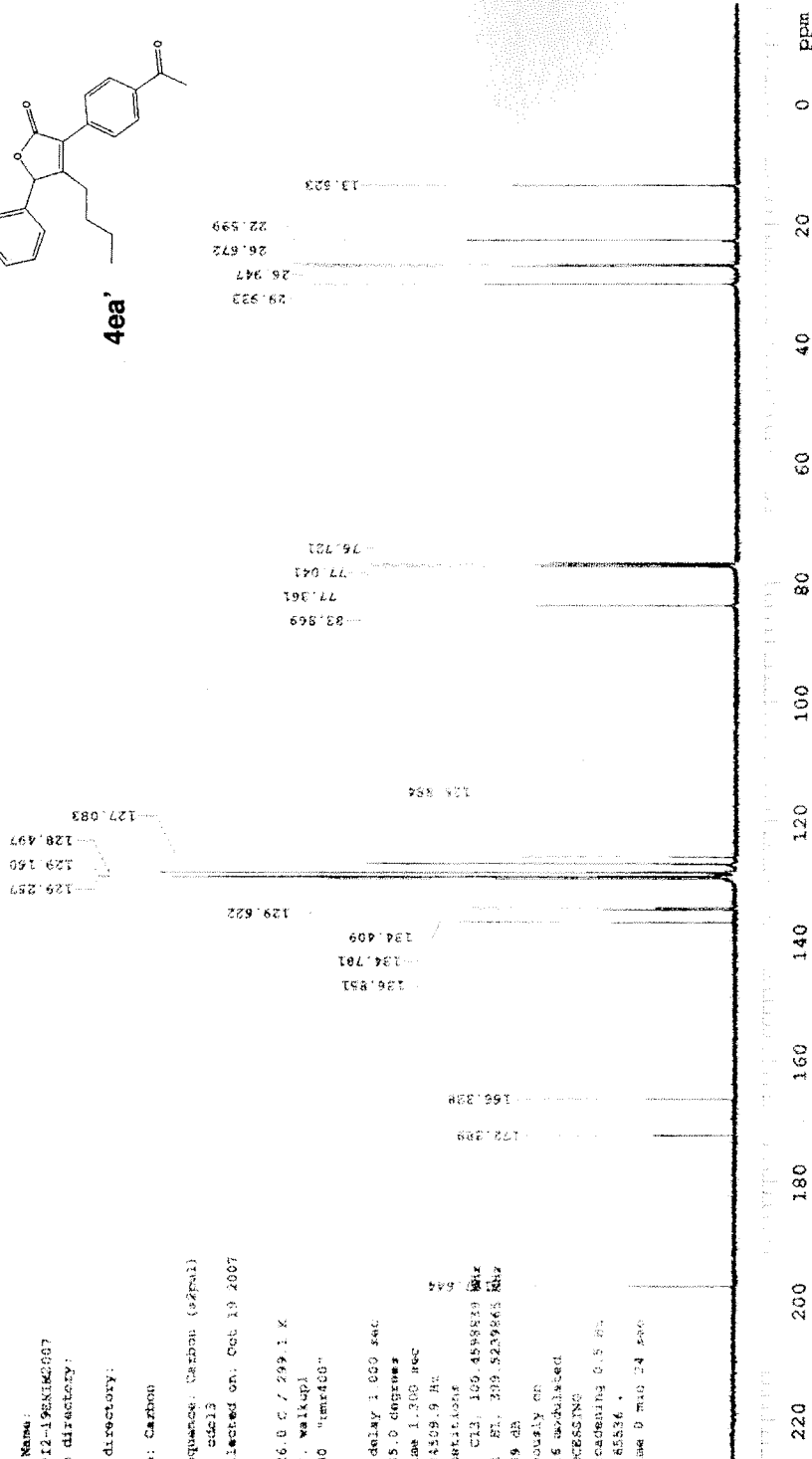
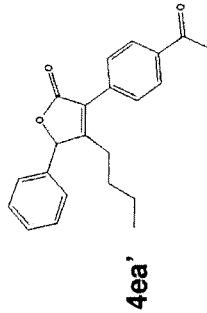
Sample directory:

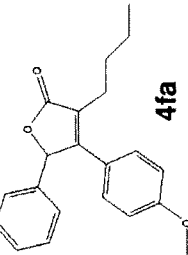
Filefile: Carbon

Pulse Sequence: Carbon (zgpg3)
Solvent: cdcl3
Data collected on: Oct 19 2007

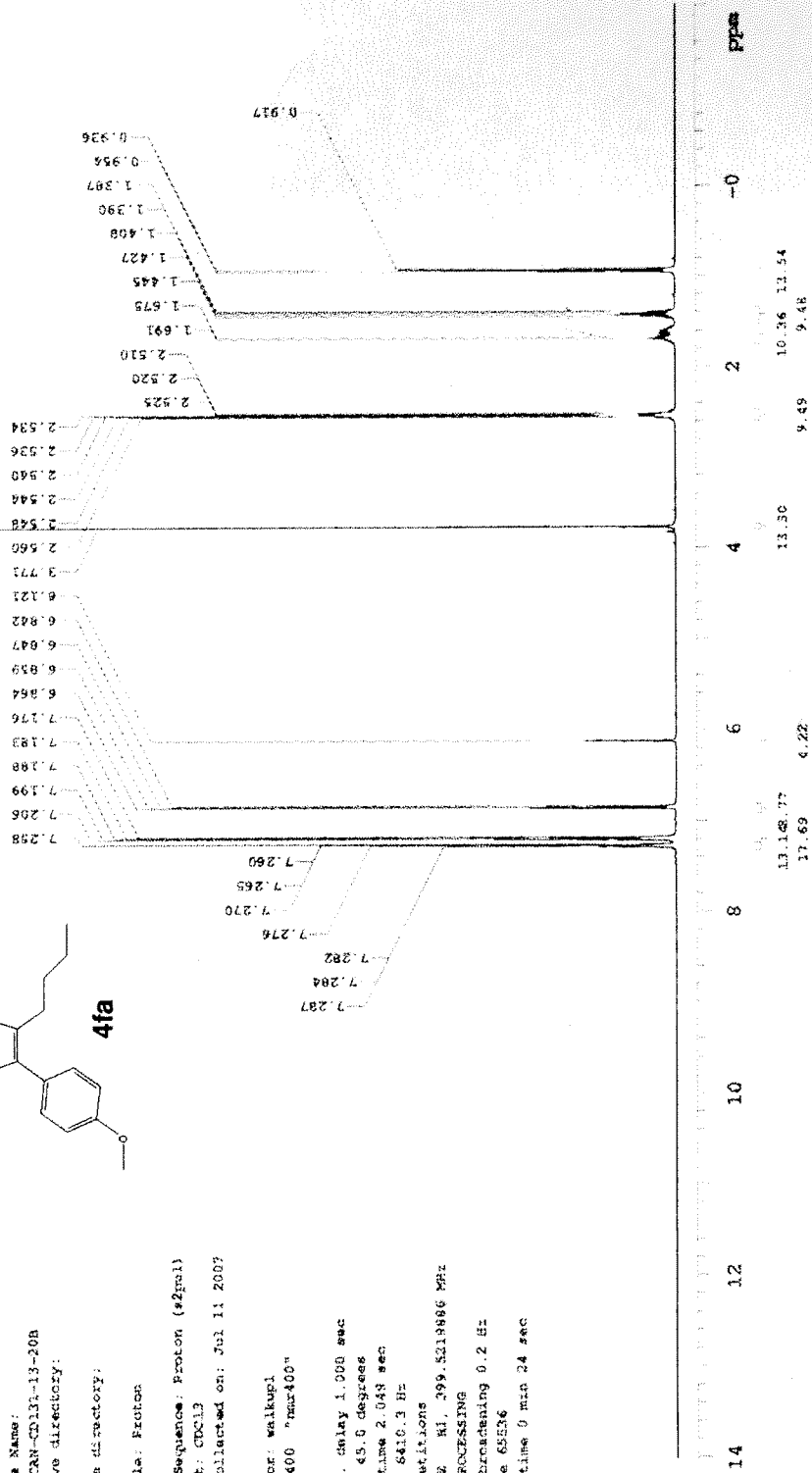
Temp: 26.0 C / 299.1 K
Operator: walkup
VNMR-400 "nmr400"

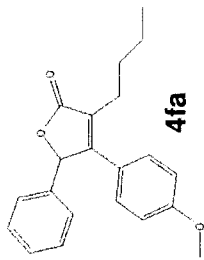
Relax delay: 1.000 sec
Pulse: 45.0 degrees
Acq: time: 1.200 sec
Width: 24509.9 Hz
S12: zgpg3ashs
OBSERVE CH: 100.4508819 MHz
DECOUPLE F1: 209.5239865 MHz
Power: 59 dB
continuously on
REALTIME MONITORING
DATA PROCESSING
Line broadening: 0.5 Hz
FT file: 85836
Total time: 0 min 24 sec





NURCAN
 11 TEMEIZ 2007
 Sample Name:
 NURCAN-CD13-13-20A
 Archive directory:
 Sample directory:
 FidFile: Proton
 Pulse Sequence: Proton (#2full)
 Solvent: CDCl3
 Data collected on: Jul 11 2007
 Operator: waikul
 VMRS-400 "nmr400"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 ACQ. time 2.048 sec
 Width 6410.3 Hz
 9 repetitions
 OBSERV: 41, 399.521886 MHz
 DATA PROCESSING
 Line broadening 0.2 Hz
 FT size 6526
 Total time 0 min 24 sec





NMR001
 11 THURSDAY 2007
 130.
 DEPT.
 NMR001
 INDC.
 NMR001
 CORY.
 C001

Sample Name:
 NMR001-011-13-108
 Archive directory:

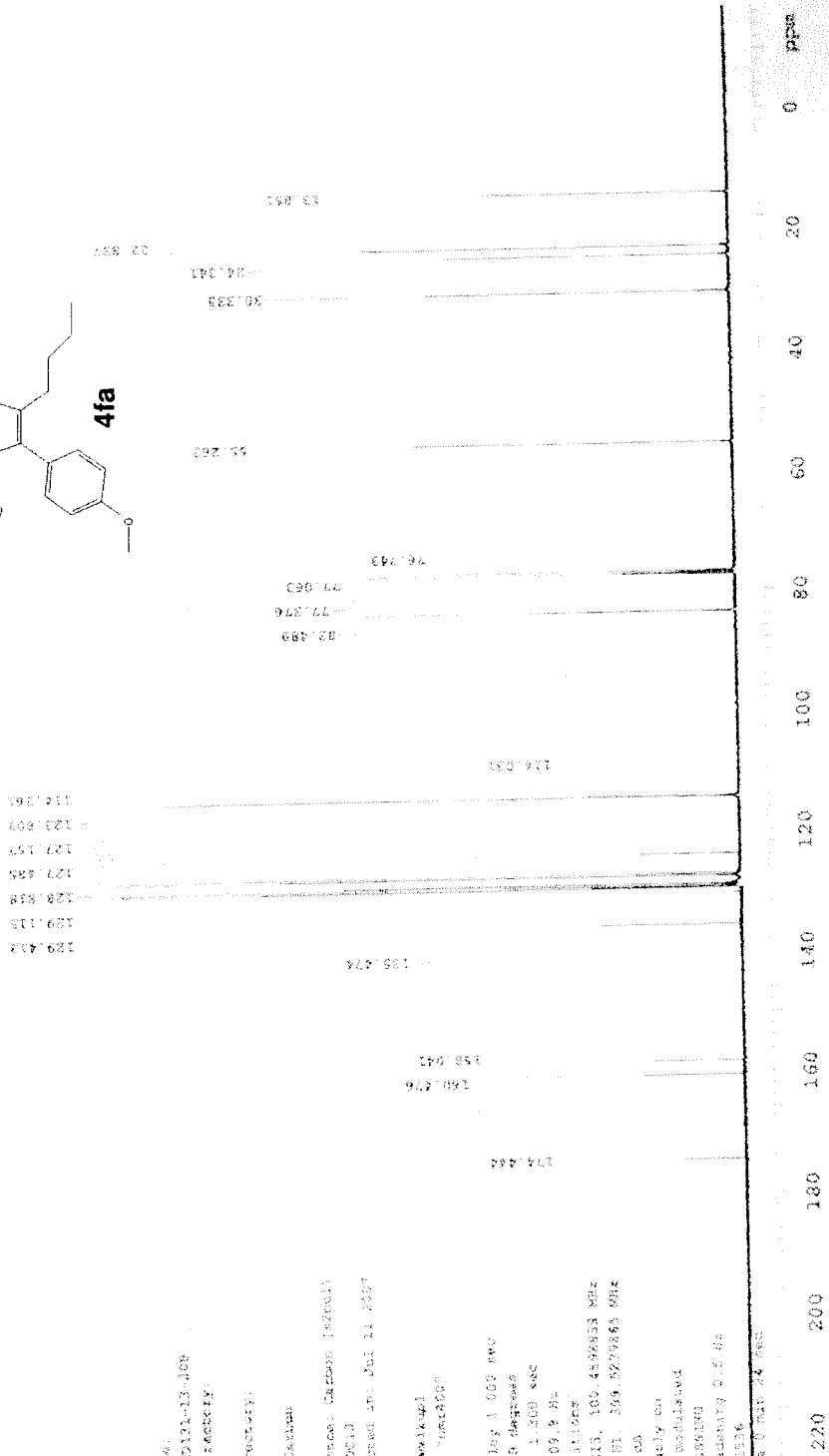
Sample directory:
 FNAME: C001

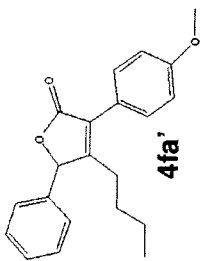
Pulse Sequence: zgpg30
 Solvent: CDCl3
 Data Collected on: 06/14/2007

Operator: wjk441
 VNAME: 001

Relax. delay: 3.000 sec
 Pulse 45.0 degrees
 Acq. time: 1.300 sec
 Width: 24500.0 Hz
 F2: 100.626100 MHz
 OBSERVE: C13, 100.626100 MHz
 DECUPLE: F1, 300.137000 MHz
 Power: 29.00

continuously on
 SRRZ: 16 modified
 DATA PACKAGING
 Lock frequency 0.5 Hz
 FT size: 65536
 Total time: 0 min 24 sec





MENCAR
 12 THOMAS 2007
 130, DEPT. COSY, GOSY, NOESY, ROESY, HAQC,
 CHNOQC

Sample Name:
 CD131-10-12f
 Archive directory:

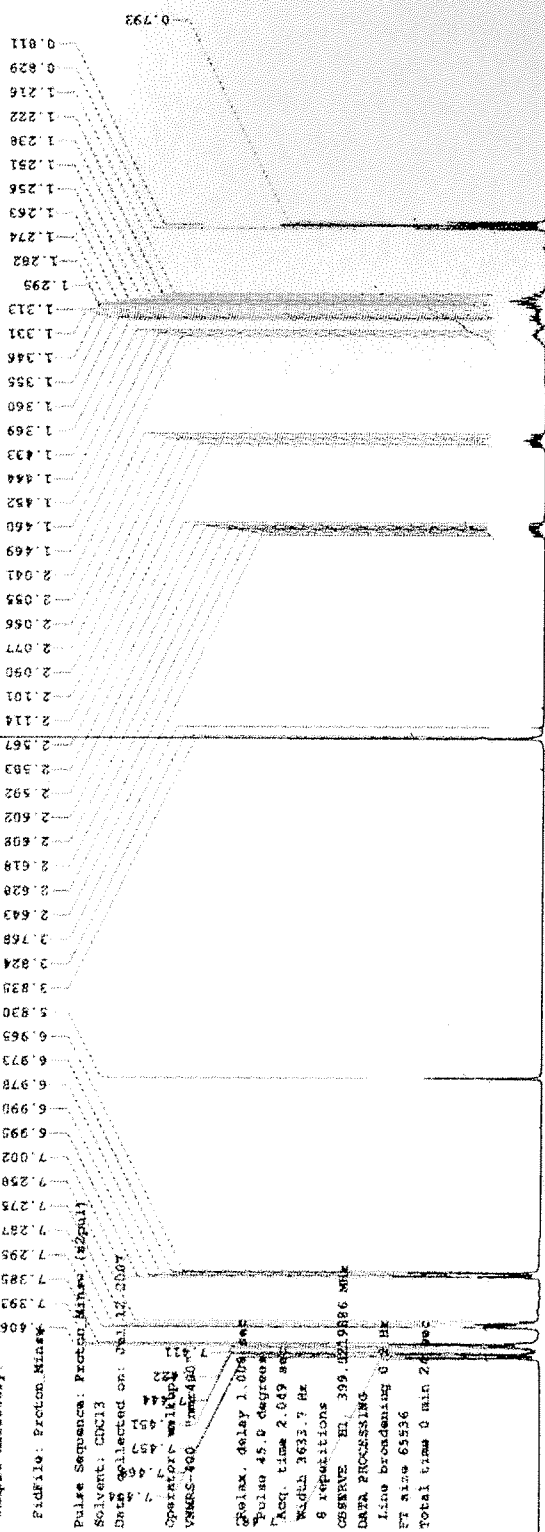
Sample directory:

File: Proton_Minus

Pulse Sequence: Proton_Minus (h2galt)
 Solvent: CDCl3
 Data collected on: 08/12/2007

Operator: willup
 VMS-300 400MHz

Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.049 sec
 Width 3633.7 Hz
 6 repetitions
 GARNET-EL 399.4579886 MHz
 DATA PROCESSING
 Line broadening 0 Hz
 FT size 65536
 Total time 0 min 24 sec



Retention Time (min)	Integration Value
0.811	
0.829	
1.216	
1.222	
1.236	
1.291	
1.256	
1.263	
1.274	
1.282	
1.295	
1.313	
1.331	
1.346	
1.358	
1.360	
1.369	
1.433	
1.444	
1.452	
1.458	
1.469	
2.041	
2.055	
2.056	
2.077	
2.098	
2.101	
2.114	
2.167	
2.383	
2.592	
2.602	
2.608	
2.618	
2.628	
2.643	
2.768	
3.824	
3.838	
3.850	
5.830	
6.969	
6.973	
6.978	
6.980	
6.990	
6.995	
7.002	
7.258	
7.275	
7.287	
7.295	
7.385	
7.393	
7.395	
8.06	
12.91	8.66
6.58	30
13.53	13.53
4.74	4.65
13.66	13.66
4.27	4.27
13.95	13.95

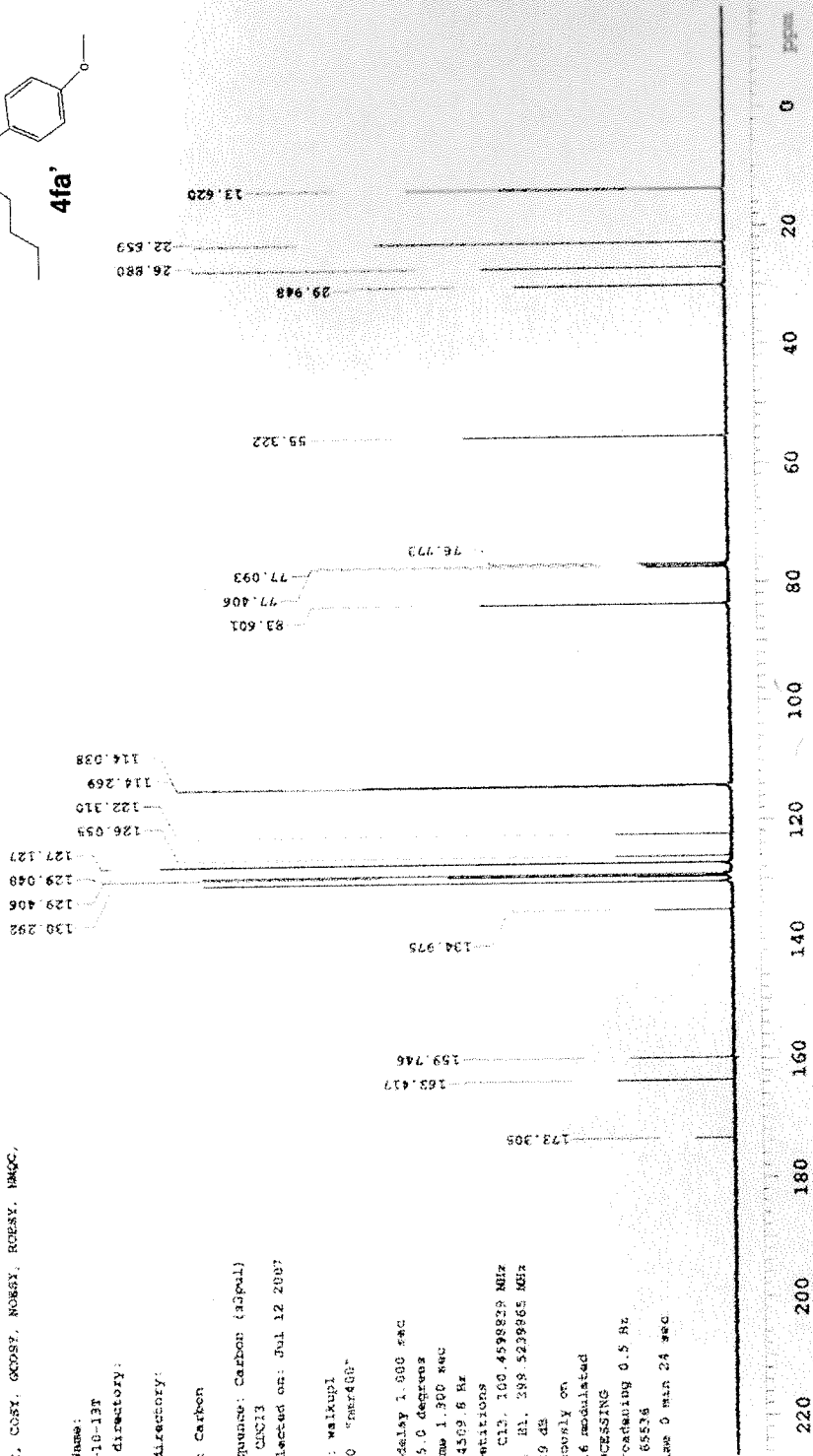
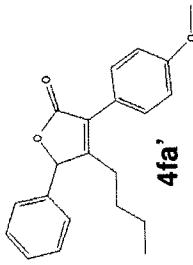
NURCAN
 NURCAN
 12 TEMERZ 2007
 ILC. DEPT. COSY. OXOSP. NORSY. ROESY. HMQC,
 CHNAC

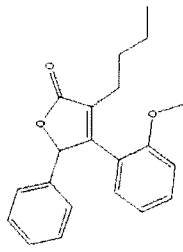
Sample Name:
 CD11-18-1EF
 Archive directory:
 Sample directory:
 Fidfile: Carbon

Pulse Sequence: Carbon (adjval)
 Solvent: CMC13
 Data collected on: Jul 12 2007

Operator: walkupl
 VNAME: 400 "FMR400"

Relax delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.000 sec
 Width 24509.6 Hz
 156 repetitions
 CARRIER: C13, 100.4598829 MHz
 DECOUPLE: H1, 199.523965 MHz
 Power 19 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 65536
 Total time 0 min 24 sec





4ga

NOFACAN
T1941182070607

Sample Name:
T1941182070607
Archive directory:

Sample directory:

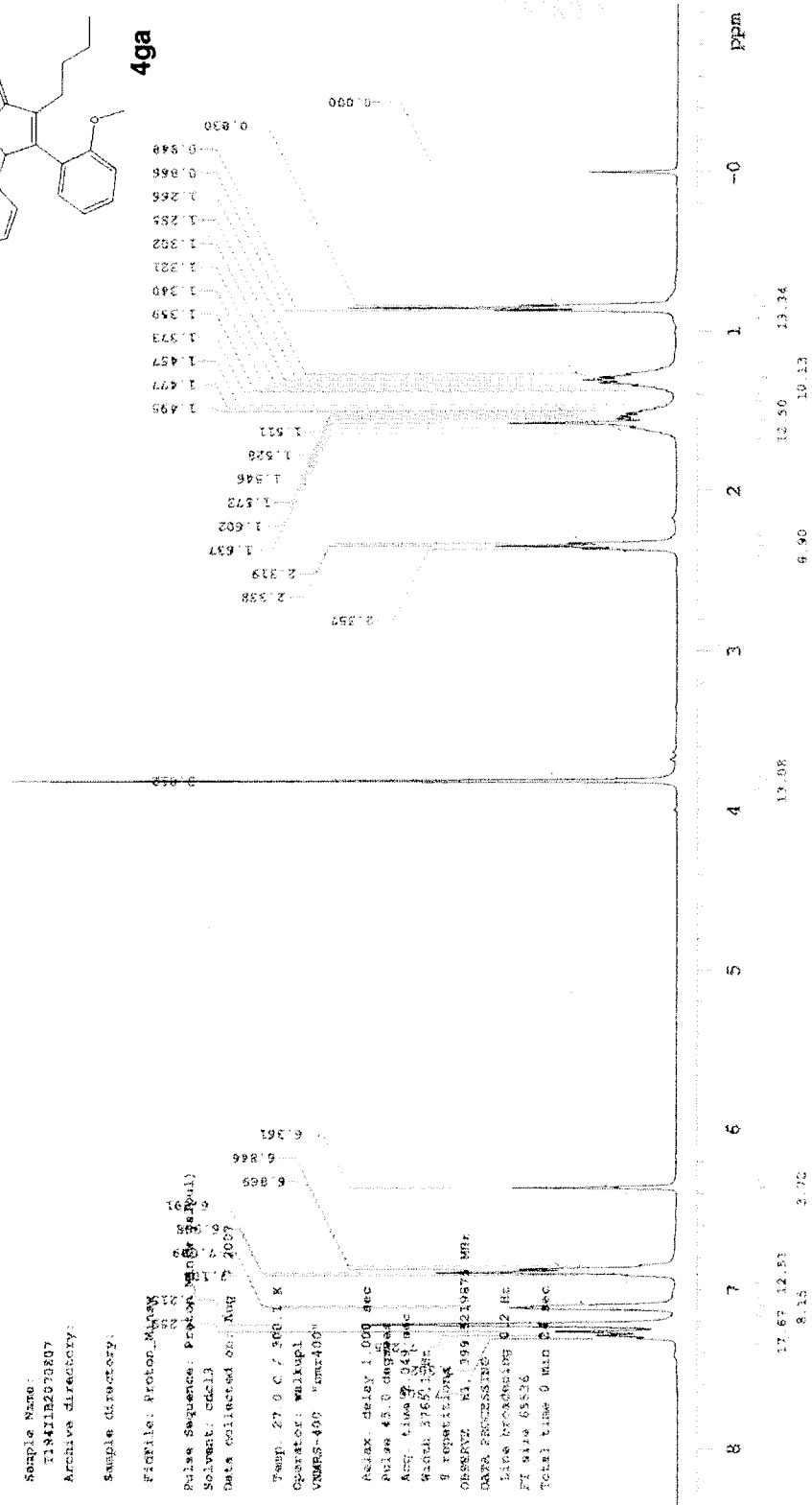
File: Proton_Meas
S1
S2

Pulse Sequence: proton
Solvent: cdcl3
Data collected on: Aug 7 2007

Temp: 27.0 C / 300.1 K
Operator: walkup
VSMRS-400 "smc400"

Relax. delay: 1.000 sec
Pulse: 45.0 degrees
Acq. time: 2.345 sec
Width: 37651 Hz
9 repetitions

OBSRVZ: 61.399 Hz
DATA PROCESSING:
Line processing: 0.12 Hz
FI gain: 655.6
Total time: 0 min 24 sec



NUPCAS
NUPCAS
X194113Z070807

Sample Name:
X194113Z070807
Archive directory:

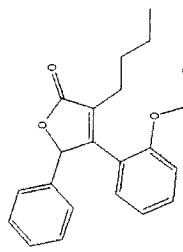
Sample directory:

Filefile: Carbon

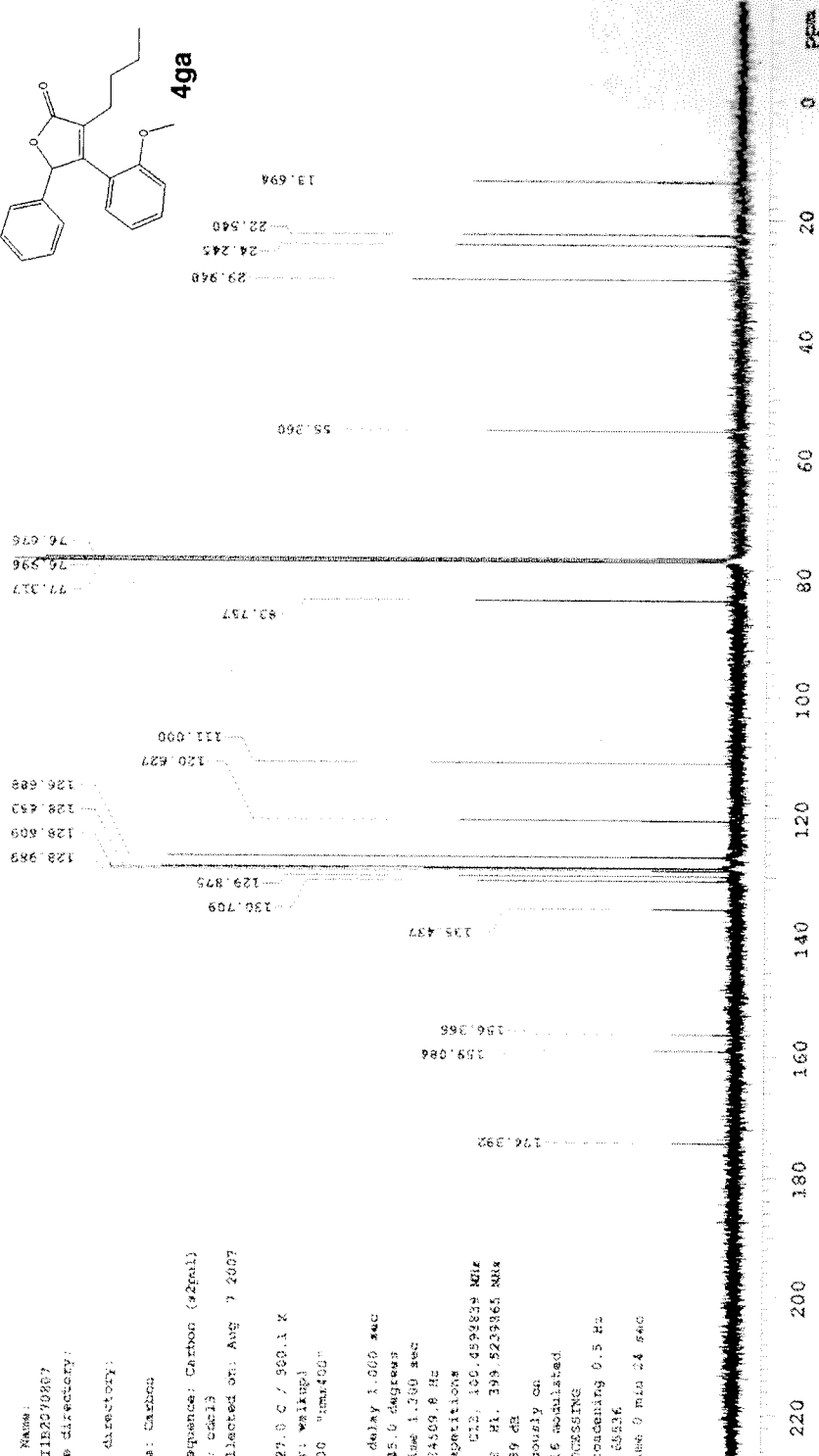
Pulse Sequence: Carbon (290k)
Solvent: cdcl3
Data collected on: Aug 7 2007

Temp: 27.0 C / 90.6 K
Operator: walkup
VSWR: 400 vswr400

Relax. delay: 1.000 sec
Pulse: 45.0 degrees
Acq. time: 1.150 sec
Width: 24500.0 Hz
1000 repetitions
GSSERVE: C12, 100, 0594839 Mix
USCOUPLE: H1, 399, 5239661 Mix
Pows: 39 db
continuously on
NALT2-16 enabled
DATA PROCESSING
Line broadening: 0.5 Hz
FT size: 65536
Total time: 0 min 24 sec



4ga



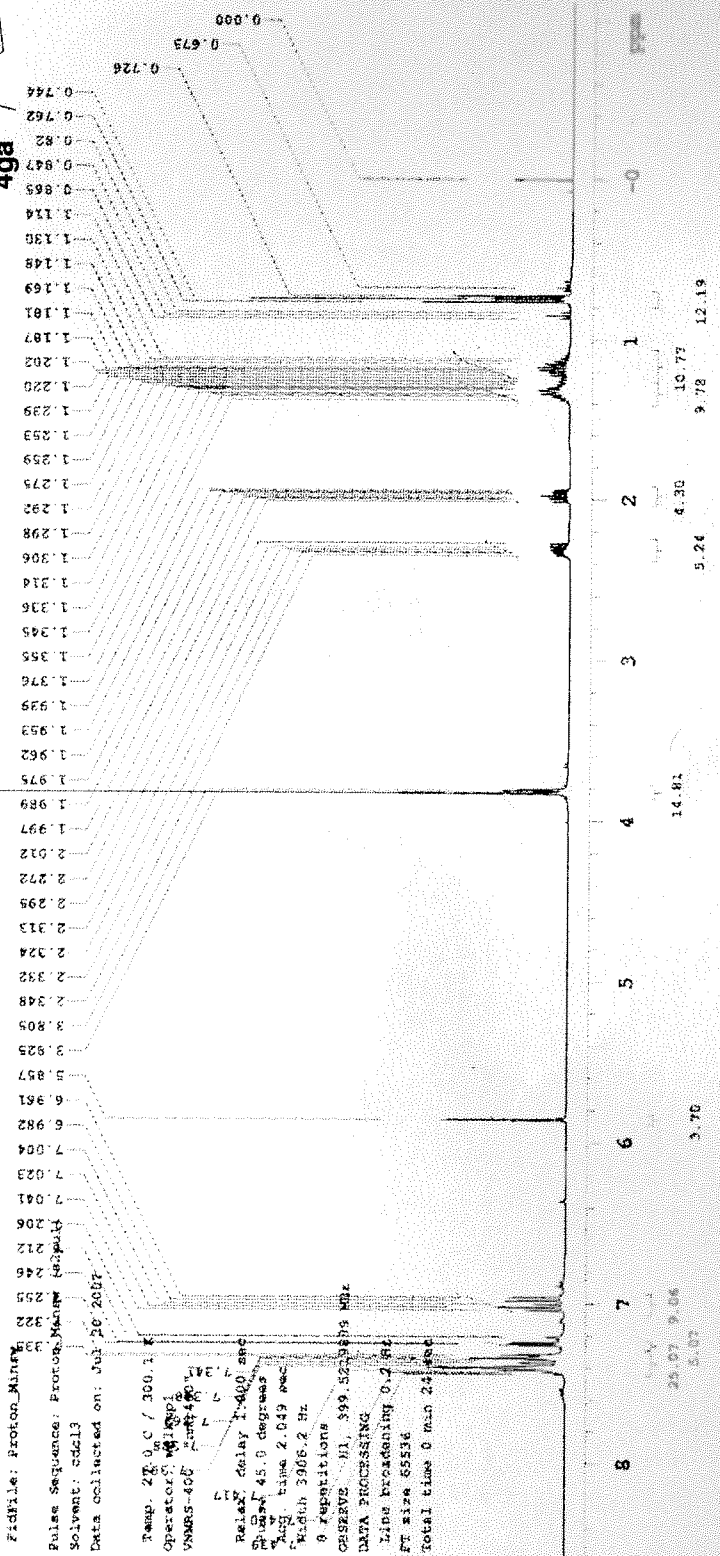
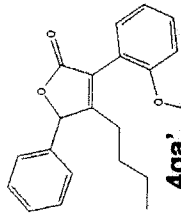
MURCCK
T194ZKT2-200707

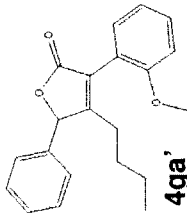
Sample Name:
T194ZKT2-200707
Archive directory:

Sample directory:

Fidfile: Proton_Minny
Pulse Sequence: Proton_Minny (default)
Solvent: cdcl3
Data collected on: Job: 20: 2007

Temp: 27.0 C / 300.1 K
Operator: MURCCK
VNMRS-400 MHz
Relax: delay 1.400 sec
P1: 4.50 sec
P2: 45.0 degrees
Acq. time 2.049 sec
Fid: 3006.2 Hz
0 repetitions
GSESAVE: N1, 399.527805
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 0 min 24.80 sec





4ga'

NUSCAN
 NUSCAN
 T1942RT2-200707

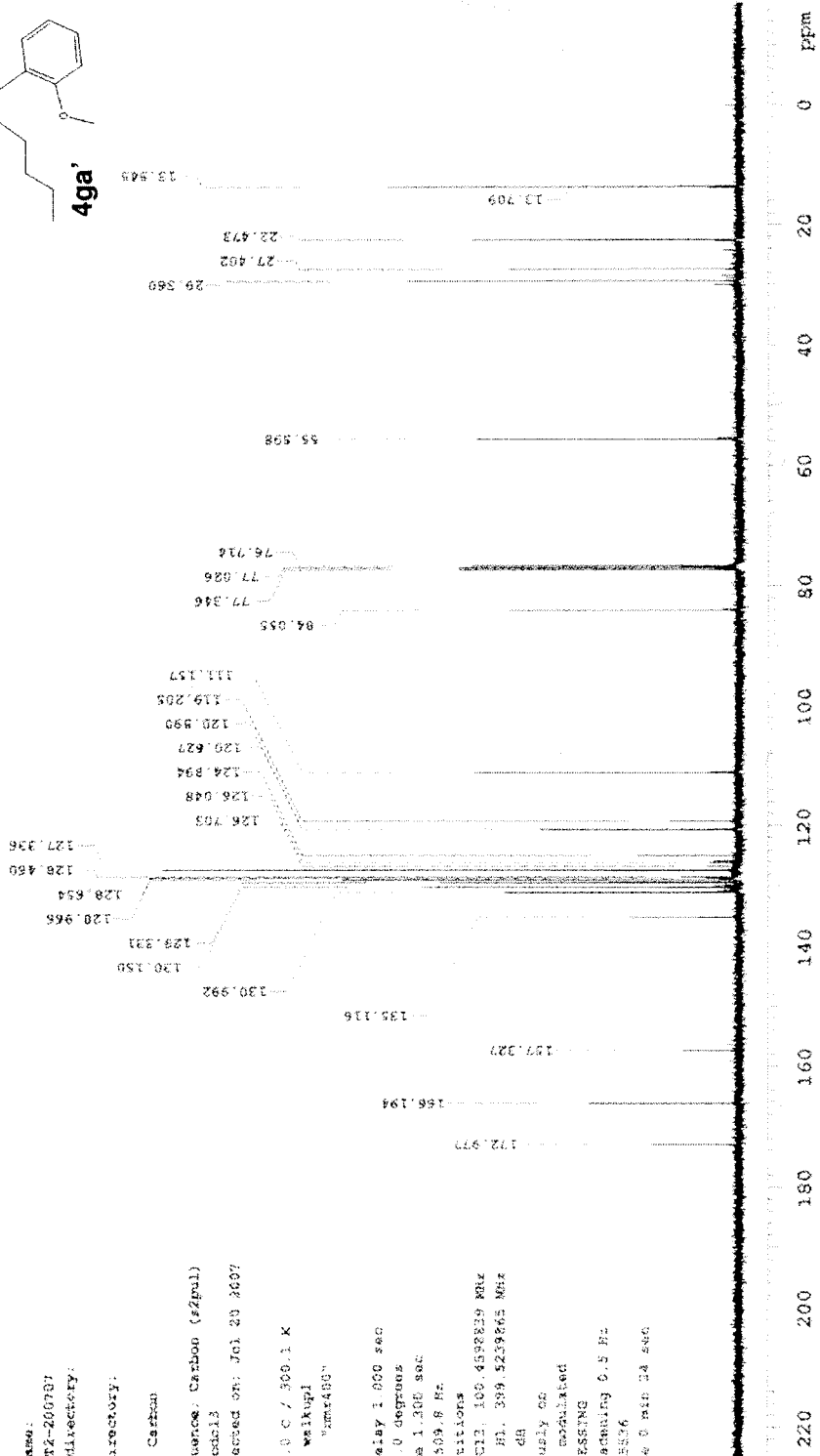
 Sample Name:
 T1942RT2-200707
 Archive directory:

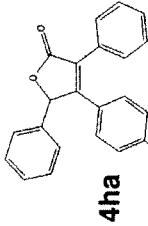
 Sample directory:
 Filename: Carbon

 Pulse Sequence: Carbon (sZpal)
 Solvent: cdcl3
 Data collected on: Jul 20 2007

 Temp. 27.0 C / 309.1 K
 Operator: walikupi
 VMS6-400 vsm4897

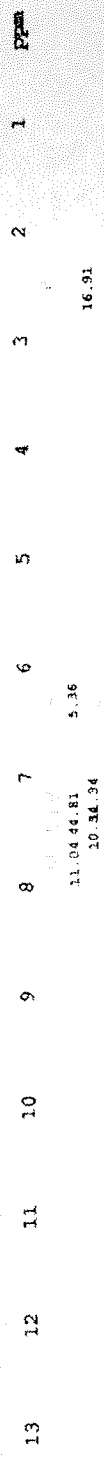
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.100 sec
 Width 24500.0 Hz
 256 repetitions
 OBSERVE CH: 100.4528259 MHz
 DECOUPLE CH: 399.5238665 MHz
 Power 59 dB
 continuously ON
 WALTZ-16 scheduled
 DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 65536
 Total time 0 min 24 sec





NURCAN-2732K221
 Sample Name:
 NURCAN-2732K221
 Data Collected on:
 mmr400-vmas400
 Archive directory:
 /home/walkup/vmas400/data
 Sample directory:
 NURCAN-2732K221_21Mar2008
 Filefile: PROTON
 Pulse Sequence: PROTON (s2pul1)
 Solvent: cdcl3
 Data collected on: Mar 21 2008
 Temp: 25.0 C / 298.1 K
 Operator: walkup
 Ruler delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 3.956 sec
 Width 6410.5 Hz
 4 repetitions
 OBSERVE AT 399.521985 MHz
 DATA PROCESSING
 FT size 32768
 Total time 0 min 29 sec

7.784
 7.773
 7.769
 7.763
 7.766
 7.460
 7.451
 7.450
 7.448
 7.444
 7.442
 7.372
 7.365
 7.360
 7.353
 7.348
 7.338
 7.329
 7.323
 7.319
 7.315
 7.312
 7.308
 7.304
 7.295
 7.298
 7.295
 7.282
 7.272
 7.266
 7.261
 7.259
 7.214
 7.210
 7.205
 7.193
 7.188
 7.184
 6.278
 2.513



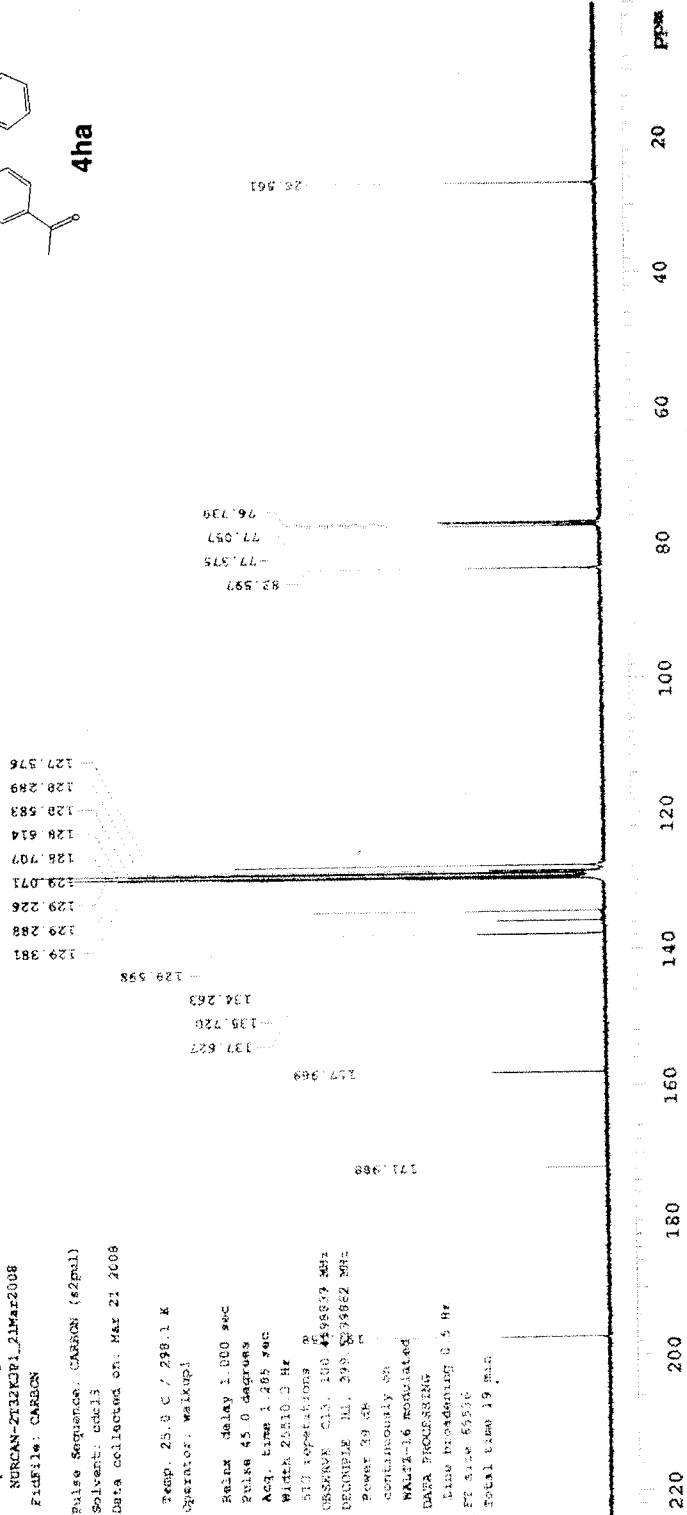
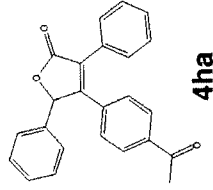
NURCAN-212KCP1

Sample Name:
NURCAN-212KCP1
Data Collected on:
mmr480-vmr480
Archive directory:
/home/walkup/vmr480/data
Sample directory:
NURCAN-212KCP1_21Mar2008
Fidfile: CARBON

Pulse Sequence: CARBON (g2pul)
Solvent: cdcl3
Data collected on: Mar 21 2008

Temp: 25.3 C / 298.1 K
Operator: walkup

Pulse delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.265 sec
Width 25510.5 Hz
512 repetitions
ORSEXP: C13, 100, 459892 MHz
DECUPLE: M1, 399, 509862 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 19 min

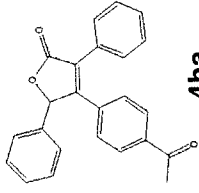


NURCAN-2732K221

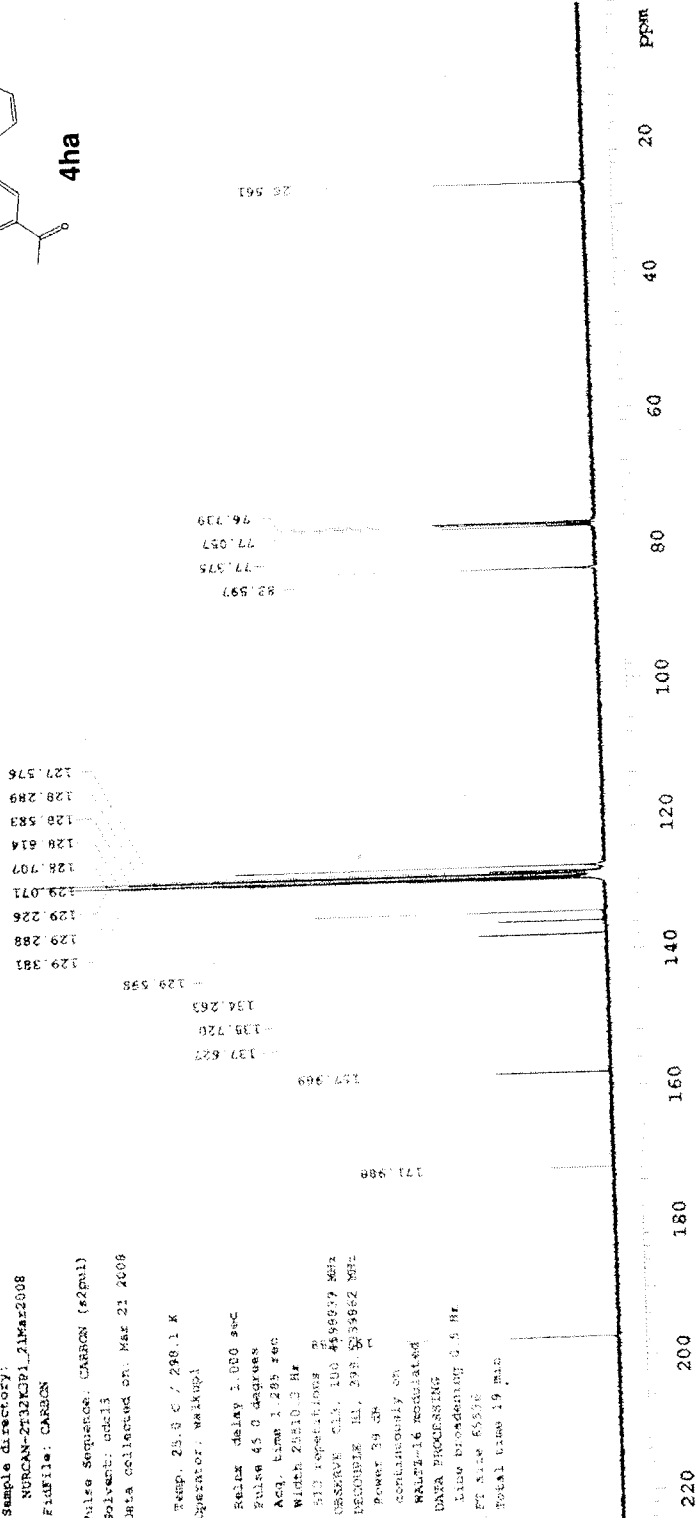
Sample Name:
NURCAN-2732K2P1
Data collected on:
nmr400-vnuz400
Archive directory:
/home/walkepl/vnmrsws/data
Sample directory:
NURCAN-2732K2P1_21Mar2008
Filefile: CARBON

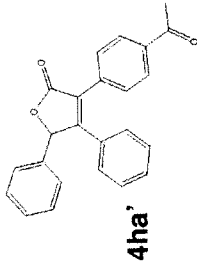
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Mar 21 2008

Temp. 25.0 C / 298.1 K
Operator: walkepl
Relax delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
Width 20510.0 Hz
SFO 400.146000 MHz
RESOLVE 400.146000 MHz
RECORDS 11, 298.053962 MHz
Power 34 dB
continuously on
WALTZ-16 MODULATED
DATA PROCESSING
Time recorded: 0.5 Hr
FT Aque 65556
Total time 19 min



4ha





2512062
25 ABALIK 1037

Sample Name:
213K92-25ABALIK2007
Archive directory:

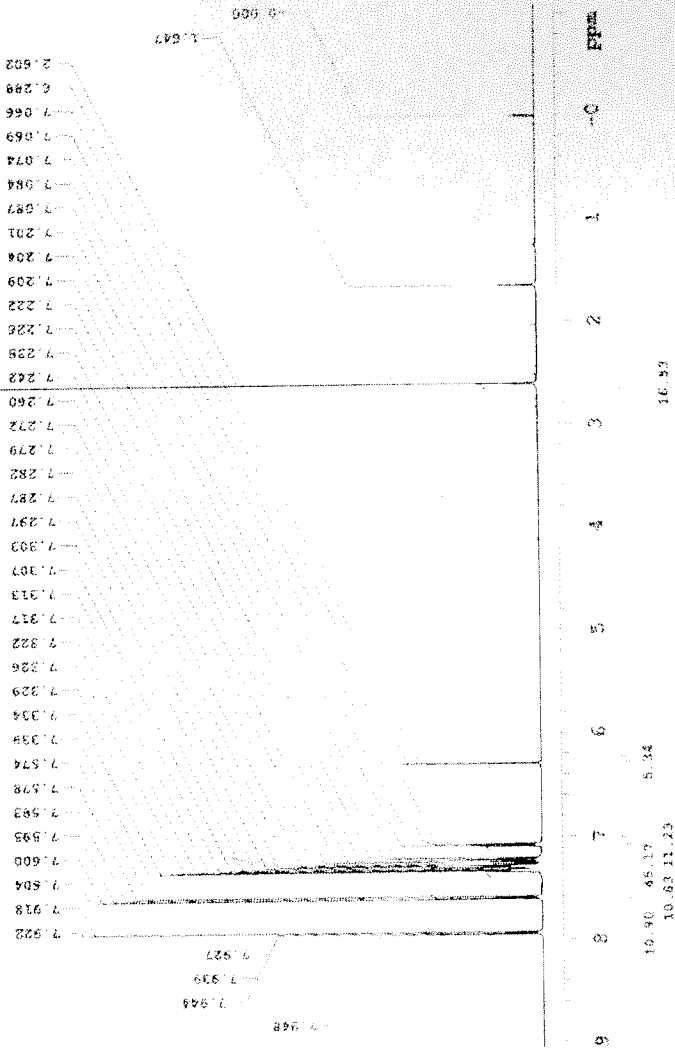
Sample directory:

Fidfile: Proton_Minw

Pulse Sequence: Proton_Minw (sdpull)
Solvent: cdcl3
Data collected on: Dec 25 2007

Temp: 25.0 C / 298.1 K
Operator: wslkopl
NAME: 600 "mrk400"

Pulse delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.042 sec
Scan 4039.4 Hz
3 repetitions
PROBE: H1 359.4219483 MHz
DATA PROCESSING
Time bracketing 0.2 Hz
FT SAMP 65536
TOTAL TIME 0 min 24 sec



2T0220C
 2T0220F2
 2T0220F2
 2T0220F2

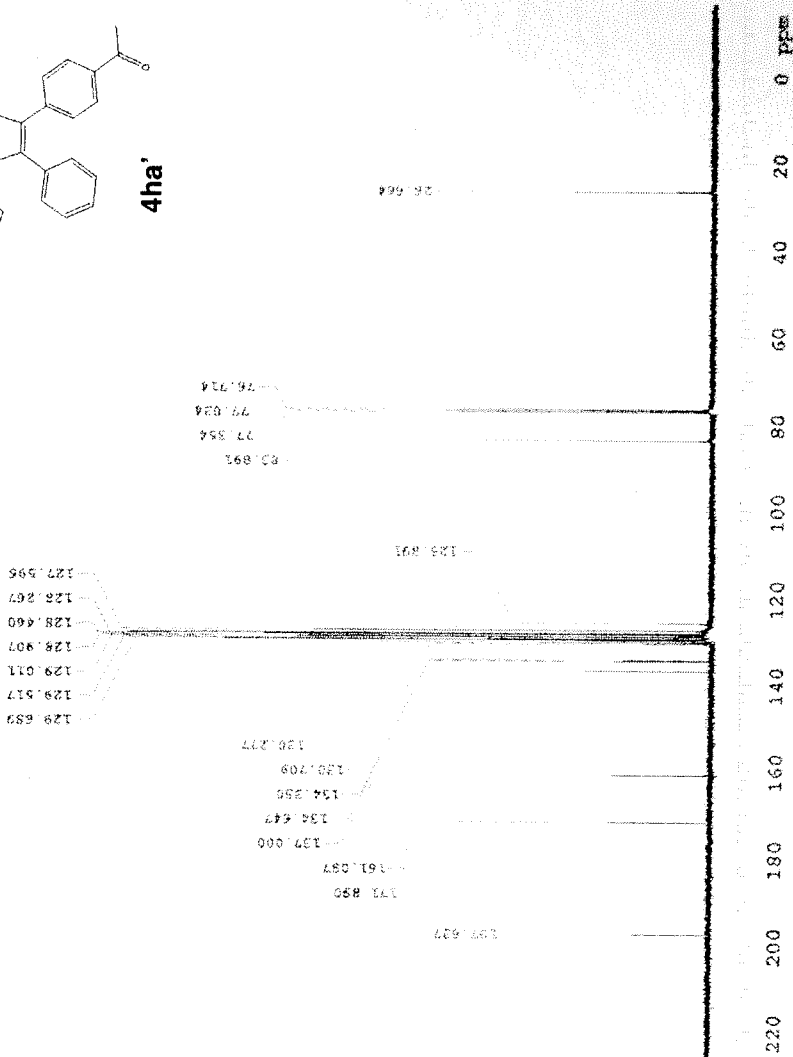
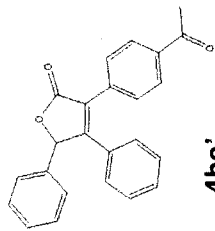
Sample Name:
 2T0220F2-20A0000007
 Archival directory:

Sample directory:
 Field#: Carbon

Pulse Sequence: Carbon (s2Fid1)
 Solvent: cdcl3
 Data collected on: Dec 05 2007

Temp. 25.0 C / 298.1 K
 Operator: walkupl
 VORSS-400 5mm400

Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.370 sec
 Nuclei 13C/13 C
 512 repetitions
 OBSERVE CH1 100.6262833 MHz
 DECOUPLE CH1 399.4208845 MHz
 Fuser 59 dB
 Continuously on
 MATH-16 enabled
 DATA PROCESSING
 Late processing 0.5 Hz
 FT size 65536
 Total time 0 min 24 sec



MURCAN
E18512-27807

Sample Name:
F18512-27807
Archive directory:

Sample directory:

Filefile: F18512-27807
Operator: walkup

Fullfile Sequence: F18512-27807

Solvent: cdcl3

Data collected on: Aug 29 2007

Temp: 27.5 C / 3000 K

Operator: walkup

VERS: 400

CPD: 8

CP: 8

CP: 8

CP: 8

CP: 8

CP: 8

CP: 8

CP: 8

CP: 8

CP: 8

CP: 8

CP: 8

CP: 8

CP: 8

CP: 8

CP: 8

CP: 8

CP: 8

CP: 8

CP: 8

CP: 8

CP: 8

CP: 8

CP: 8

CP: 8

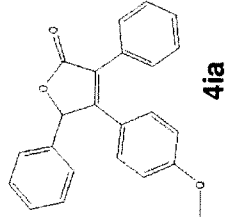
CP: 8

CP: 8

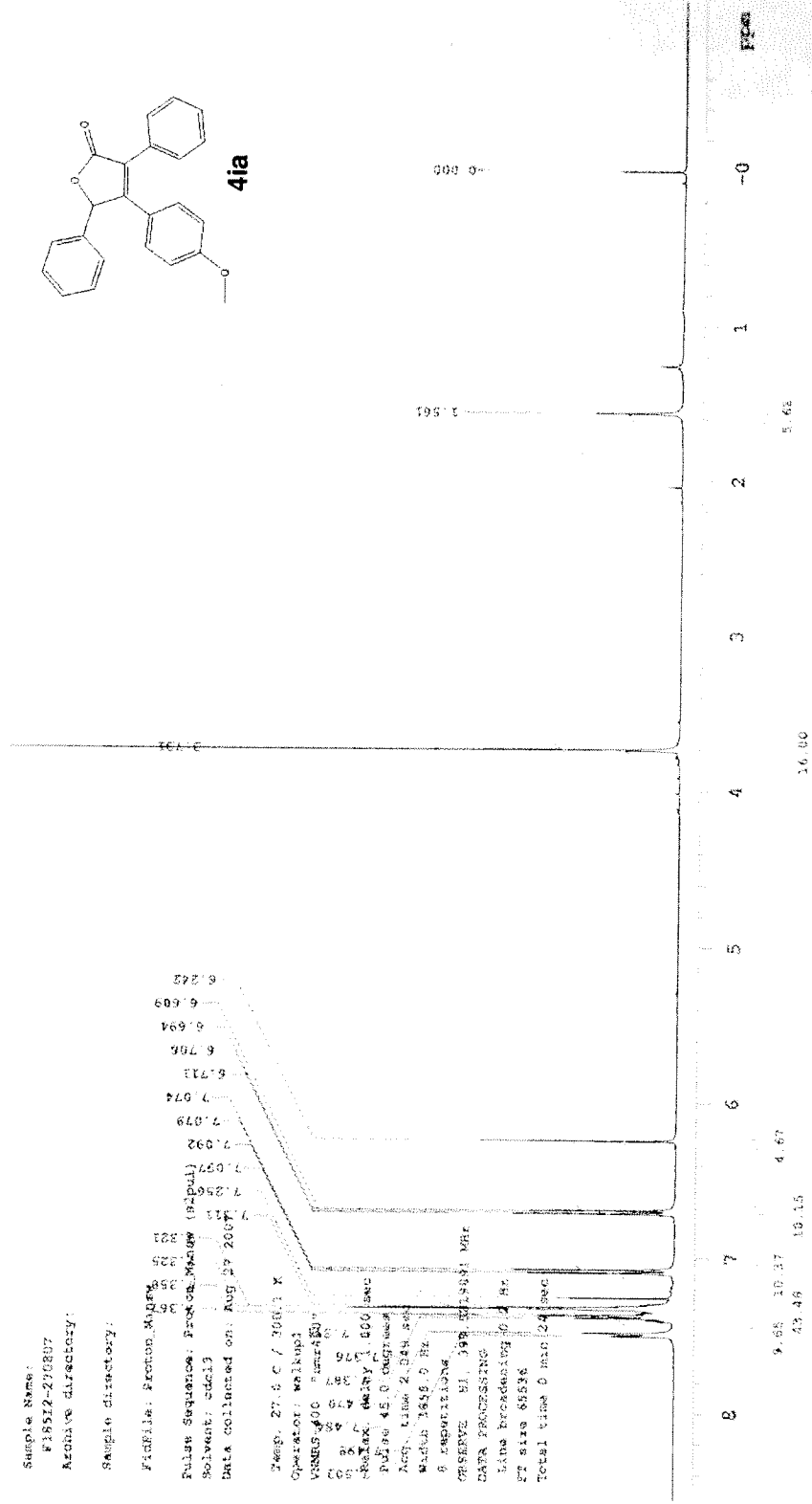
CP: 8

CP: 8

CP: 8



41a



NURCAN
NURCAN
F1812-270807

Sample Name:
F1812-270807
Archive directory:

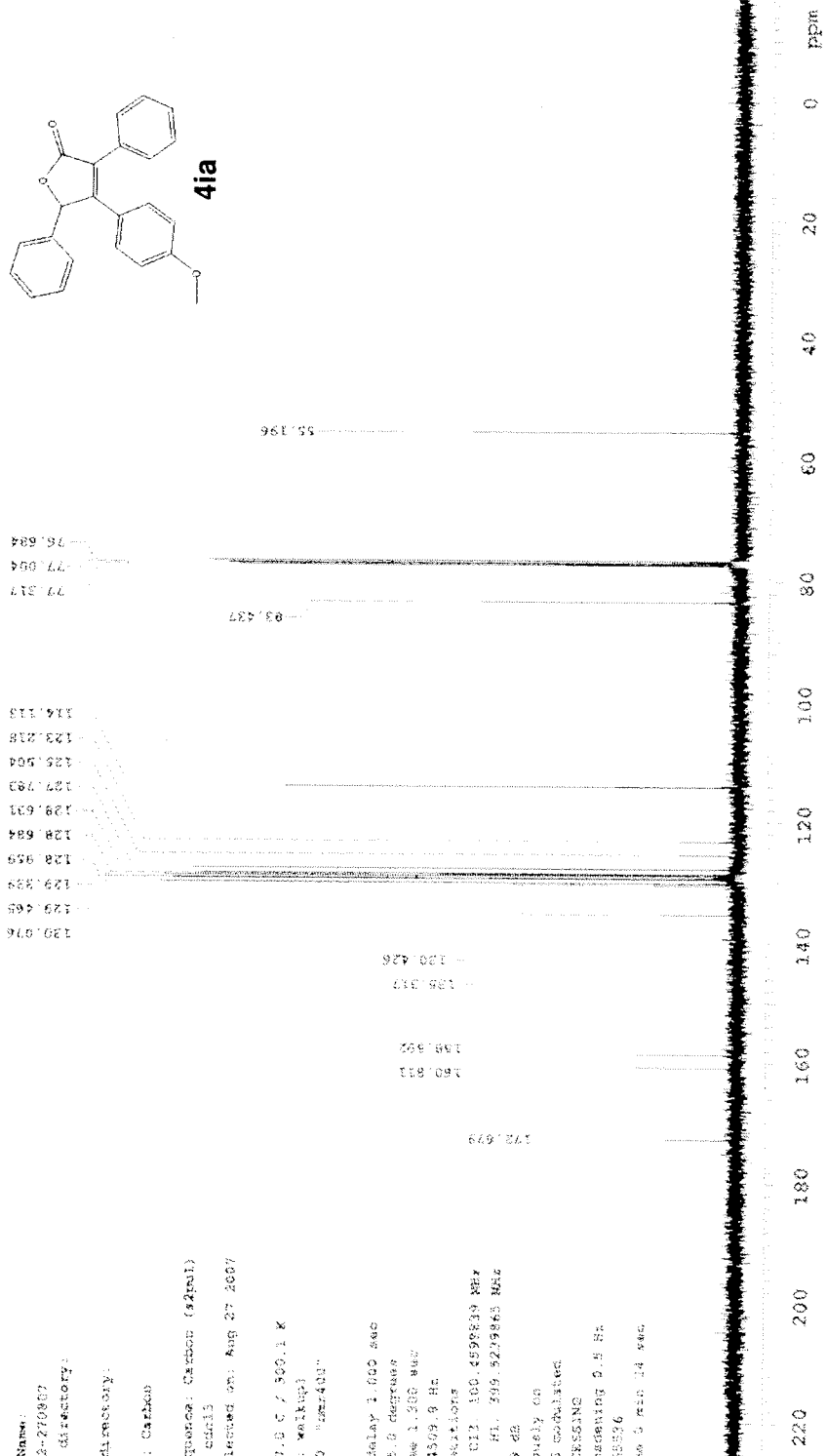
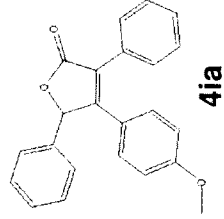
Sample directory:

ExpFile: Cambo

Pulse Sequence: Carbon (s2nu1)
Solvent: cdcl3
Data collected on: Aug 27 2007

Temp: 27.0 C / 500.1 K
Operator: walkup
SUNMS-409 4mm4001

Pulse delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24509.9 Hz
512 experiments
OBSERVE CH: 100.4998839 MHz
NUC2: 13C
Power 35 dB
continuously on
NALIZ-16 calibrated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 3 min 14 sec

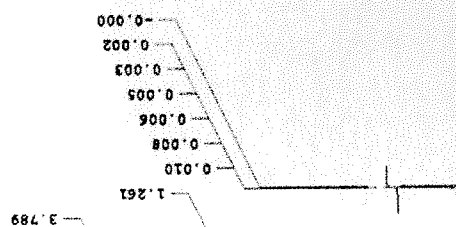
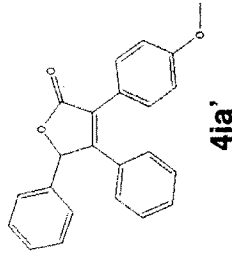
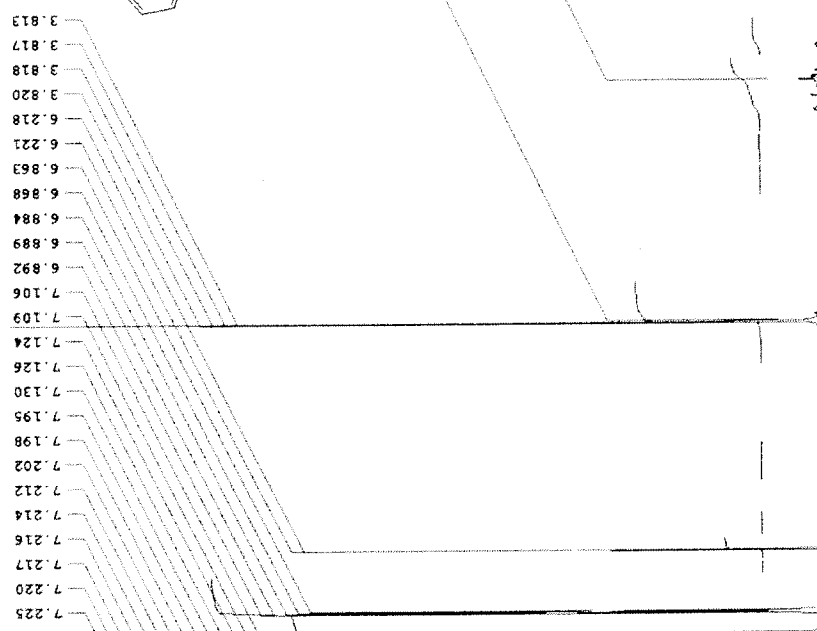
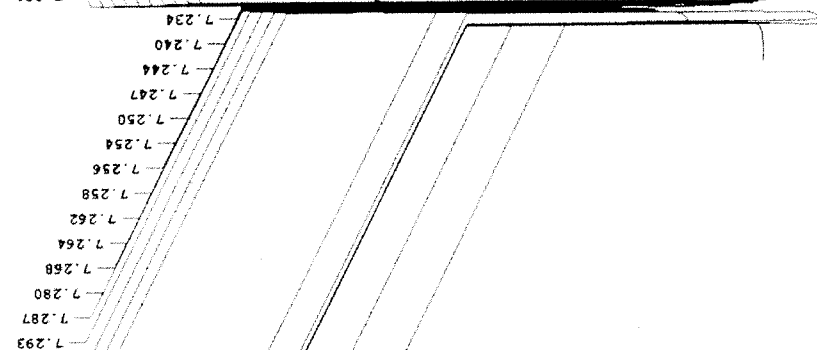


STANDARD 1H OBSERVEZ - profile

exp2 Proton

```

SAMPLE SPECIAL
date Apr 26 2007 temp not used
solvent cdc13 gain not used
file /home/walshp/~ spin 20
vmrnsys/data/walshp/~ bst 0.008
2007.04.19/arcade/PROB0 11.800
185KPI1-PROTON1 6.600
ACQUISITION
  6410.3 Hz
  2.049 in
  26264 dp
  4000 hs
  32 PROCESSING
  2 lb
  1.000 fn
  65536
  8 DISPLAY
  -756.1
  6410.3
  756.1
  HI rfl
  399.524 rfp 0
  399.2 rp -32.8
  58 lp -24.2
  5.900 PLOT
  250
  C13 ac 0
  0 vs 281
  nnn th 2
  c ai cdc ph
  32
  29412
  
```



C13
C13

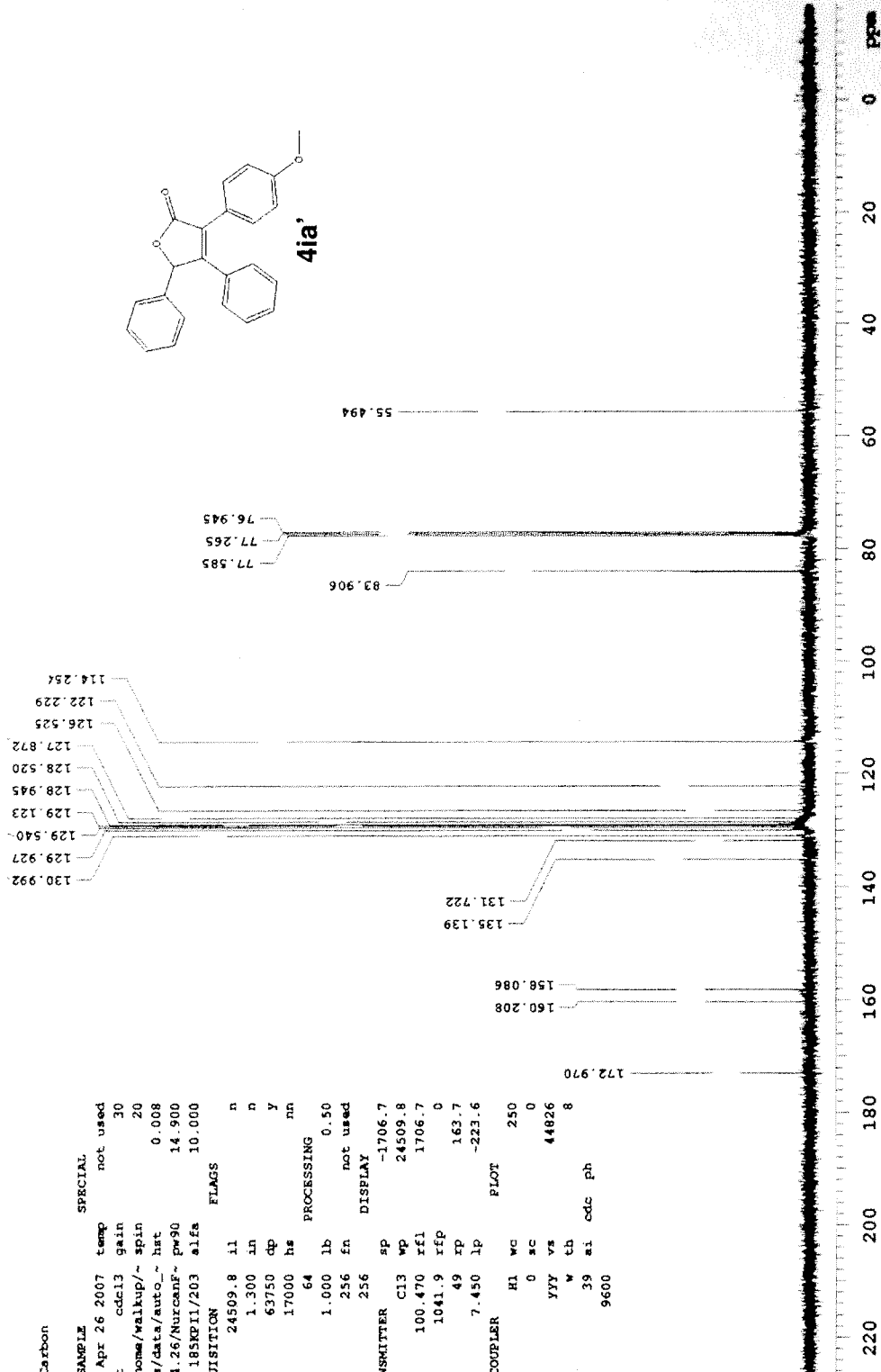
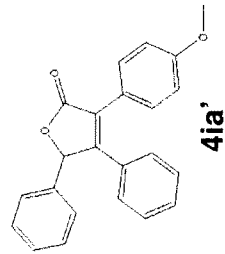
exp2 Carbon

SAMPLE SPECIAL not used
date Apr 26 2007 temp 30
solvent cdcl3 gain 20
file /home/walkup/~ spin 0.008
vnmrjys/dats/auto_~ hat 14.900
2007.04.26/NurcanF~ pw90 10.000
185KPI1/203 a1fa 10.000

ACQUISITION FLAGS
sw 24509.8 il n
at 1.300 in n
rp 63750 dp y
fb 17000 hs nn
bs 64
dl 1.000 lb 0.50
nt 256 fn not used
ct 256 DISPLAY

TRANSMITTER sp -1706.7
tn C13 wp 24509.8
sfrq 100.470 rfl 1706.7
tof 1041.9 rfp 0
tpwr 49 rp 163.7
pw 7.450 lp -223.6

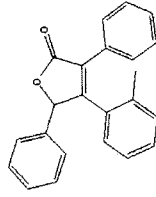
DECOUPLER HA WC 250
dof 0 sc 0
dm YY VS 44826
dwm W TH 8
dpwr 39 ai cdc ph
dmf 9600



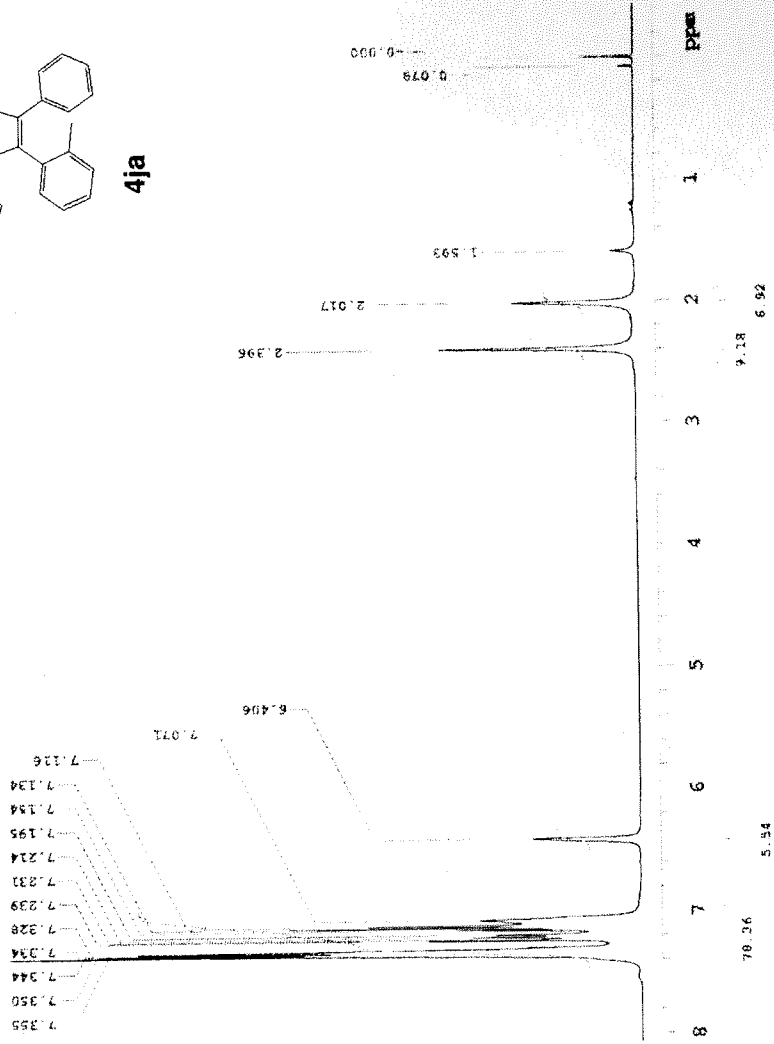
NO-7F197K12T
31 ABALIK 2007

Sample Name:
NO-7F197K12T-31ABALIK2007
Archive directory:
Sample directory:
Fidfile: Proton
Pulse sequence: Proton (*2Dul)
Solvent: cdcl3
Data collected on: Nuc 31 2007

Temp: 25.0 C / 298.1 K
Operator: walkopl
VENDOR: 400 "mar400"
Relax. delay 1.000 sec
Pulse 45.0 degree
Acq time 2.043 sec
Width 6410.3 Hz
9 repetitions
CHARGE H1, 399, 5219916 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FI size 65536
Total time 0 min 24 sec



4ja



STANDARD 1H CRESOLVE - Profile

Sample Name:

F197K12-013

Archive directory:

Sample directory:

File: Carbon

Pulse Sequence: Carbon (sZpol)

Solvent: cdcl3

Data collected on: May 25 2007

Operator: walkup

VMRS-400 "mr400"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 24509.8 Hz

F12 repetitions

CESERVE CH, 100.4596839 MHz

DECOUPLE H1, 379.523865 MHz

Power 39 dB

continuously on

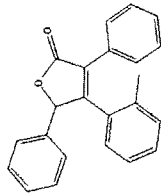
WALTZ-16 Modulated

DATA PROCESSING

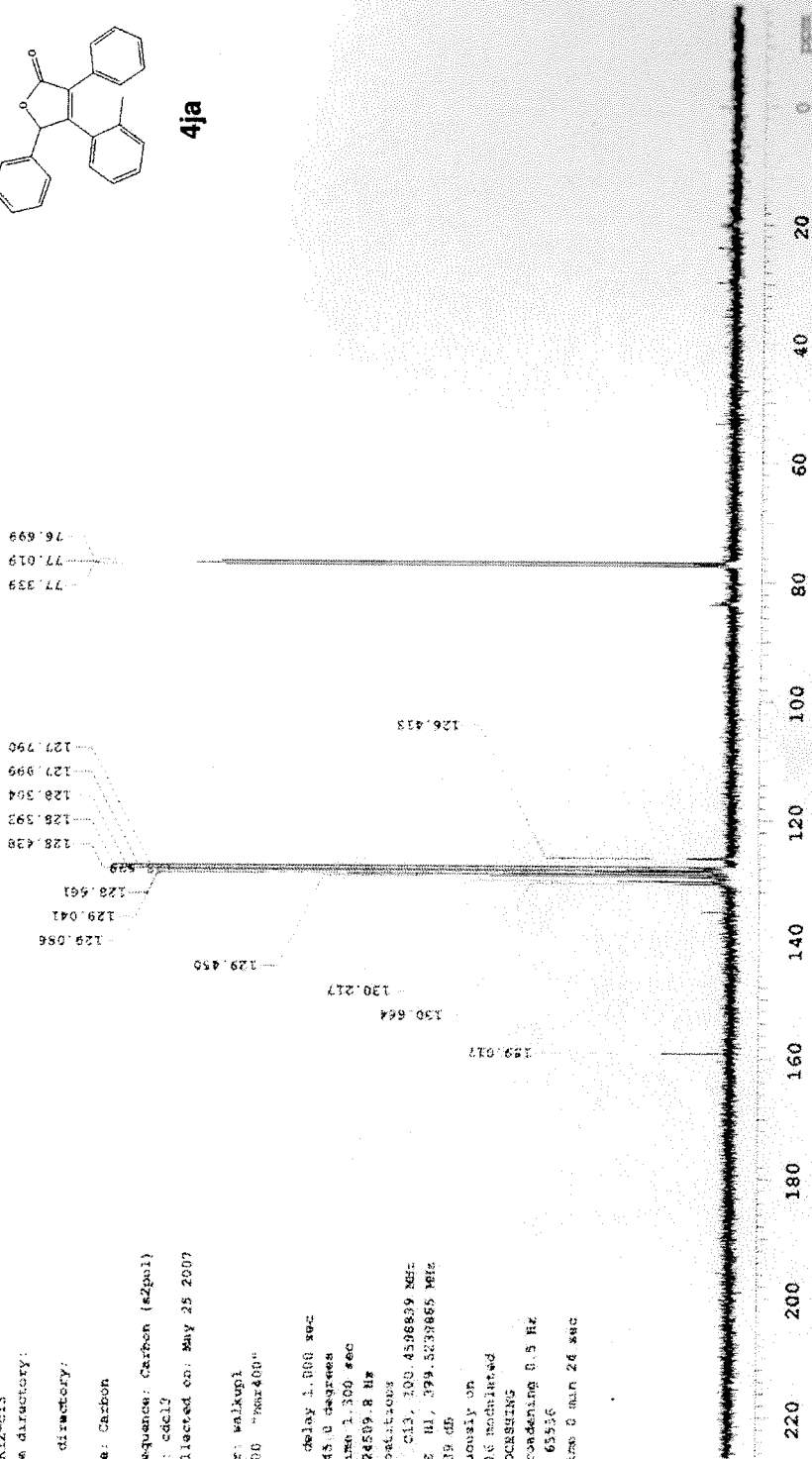
Line broadening 0.5 Hz

FT size 65536

Total time 0 min 24 sec



4ja



NUKCAN
19 TEMBEZ 2007
F197K11-190707

Sample Name:
F197K11-190707
Archive directory:

Sample directory:

Exhibit: PROCOR.MINSW

Pulse Sequence: Precton (s02p01)

Solvent: CDCl3

Data File: F190707

Temp: 27.0 C X 300

Operator: wllawrj

UNRES-400 "nmr400"

Acq. Time: 2.549 sec

Width: 9255.7 Hz

5 repetitions

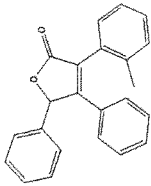
UNSERVE F1. 587.0 Hz

DATA PROCESSING

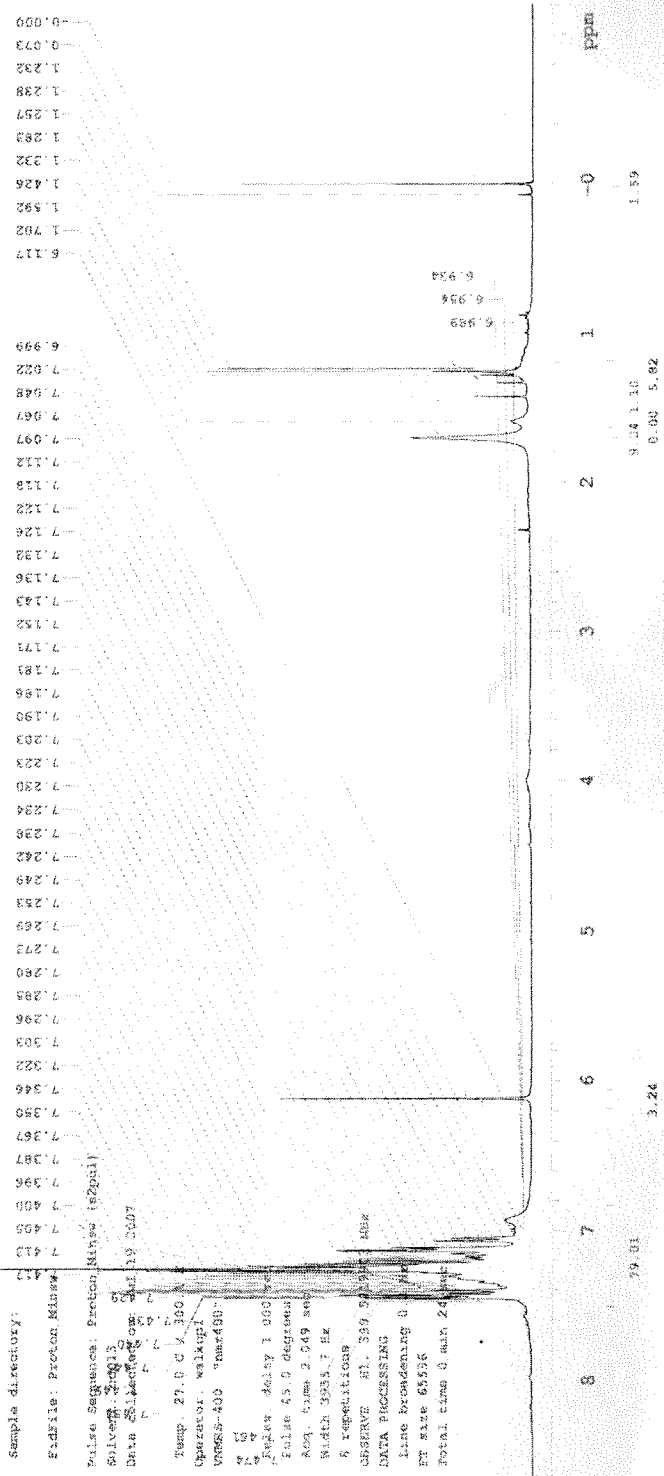
Line Broadening 0

FT size 65536

Total time 0 min 24 sec

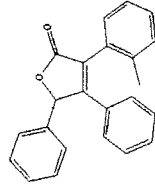


4ja'



79.01

3.24



4ja'

NURCAN
NURCAN
18 TEMMUZ 2007
F197K11-190707

Sample Name:
F197K11-190707

Archive directory:

Sample directory:

File: Carbon

Pulse Sequence: Carbon (spoil)

Solvent: cdcl3

Data collected on 18 July 2007

Temp: 27.0 C / 80.6 F

Console: walk01

VANAS-400 "marz00"

Relax delay: 1.000 sec

Pulse 45.0 degrees

Acq. time 1.500 sec

Width 24393.6 Hz

512 repetitions

USERAV CL3, 168 4598979 MB1

DECFLE HL, 399 4538985 MB1

Power 39 dB

continuously on

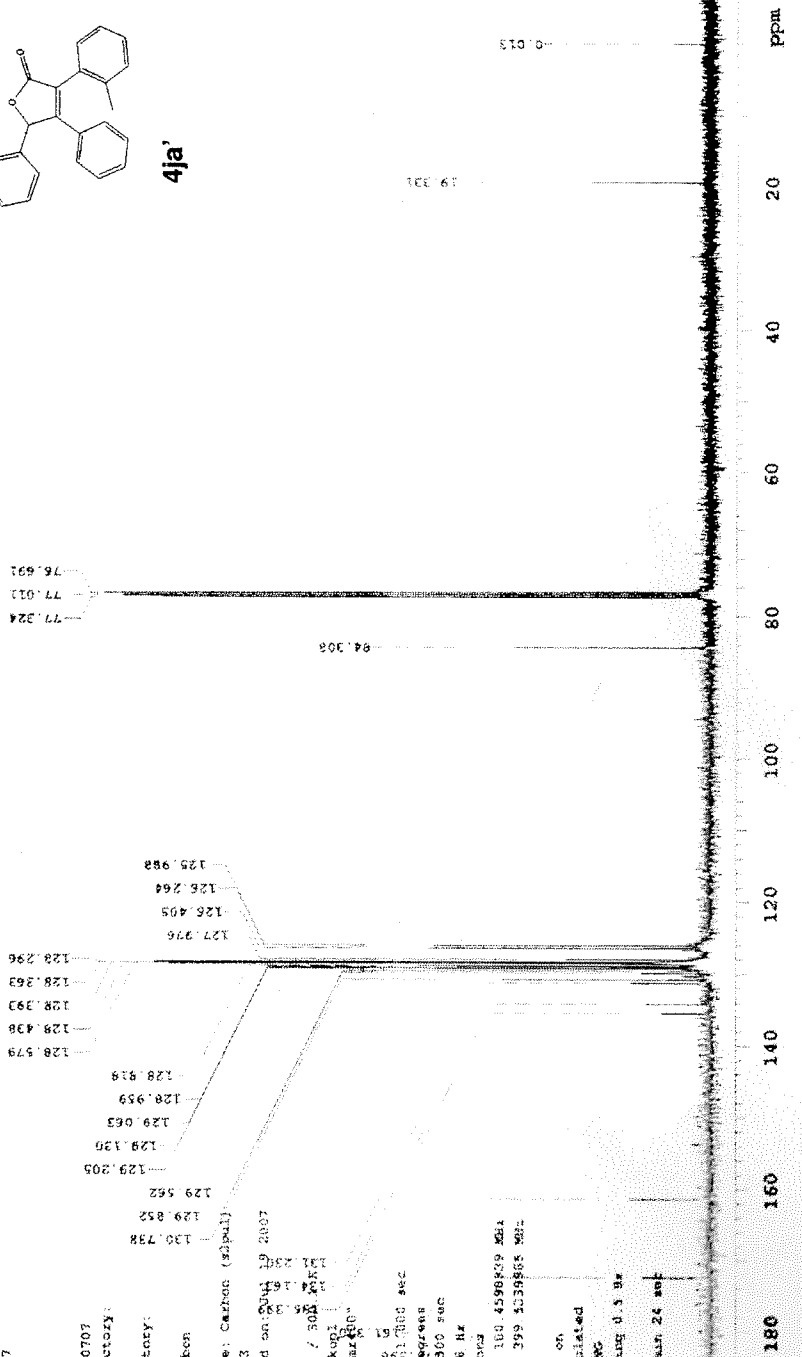
WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.3 Hz

SI size 65536

Total time 0 min 24 sec



ND-TCL3911MR-9KASIM2007

Sample Name:
ND-TCL3911MR-9KASIM2007
Archive directory:

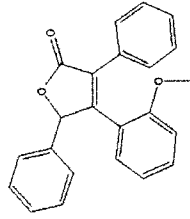
Sample directory:

File: Proton

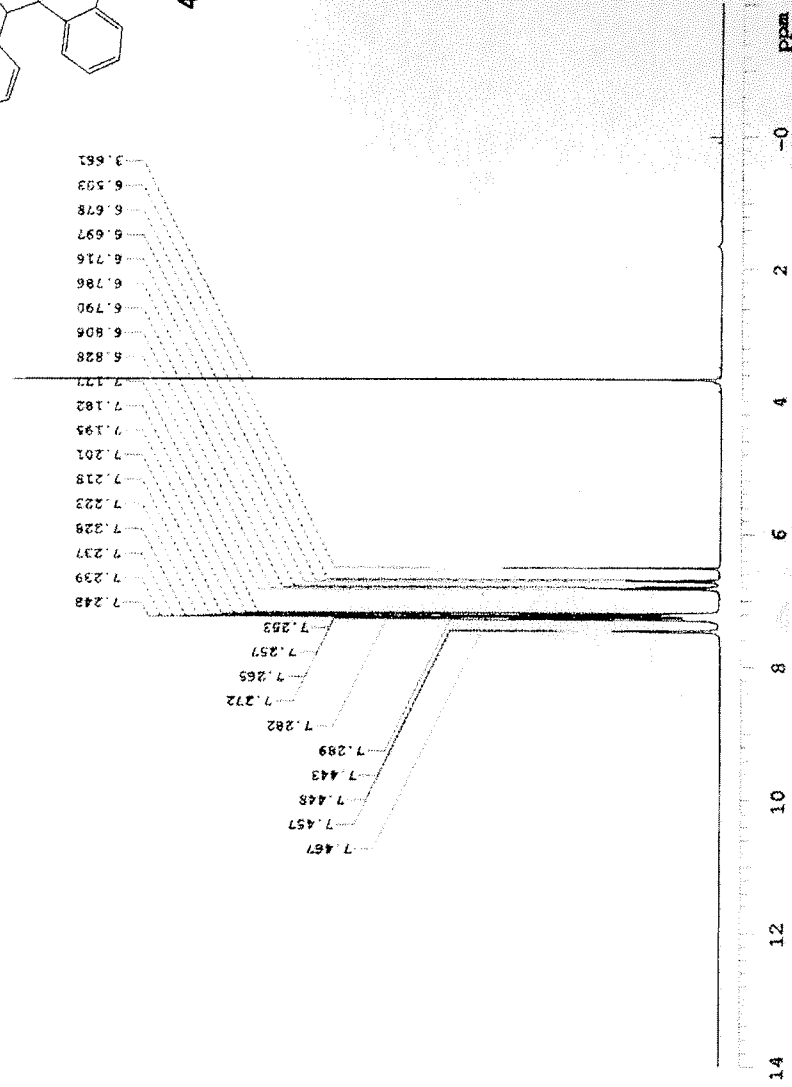
Pulse Sequence: Proton (s2pr)
Solvent: cdcl3
Data collected on: Nov 9 201

Temp: 23.0 C / 296.1 K
Operator: walkup
VNAME: 400 "nmr400"

Pulse delay: 1.000 sec
Pulse: 45.0 degrees
Acq. time: 2.049 sec
Width: 6410.3 Hz
S repetitions
ORANGE: R1, 399.521967 MHz
DATA PROCESSING
Line broadening: 0.2 Hz
FT size: 65536
Total time: 0 min 24 sec



4ka



NP-FC1391IKR-9KASINZ1007
NP-FC1391IKR-9KASINZ1007

Sample Name:
ND-FC1391IKR-9KASINZ1007
Aromatiz directory:

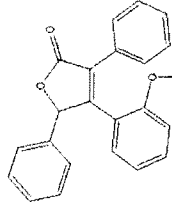
Sample directory:

Filefile: Carbon

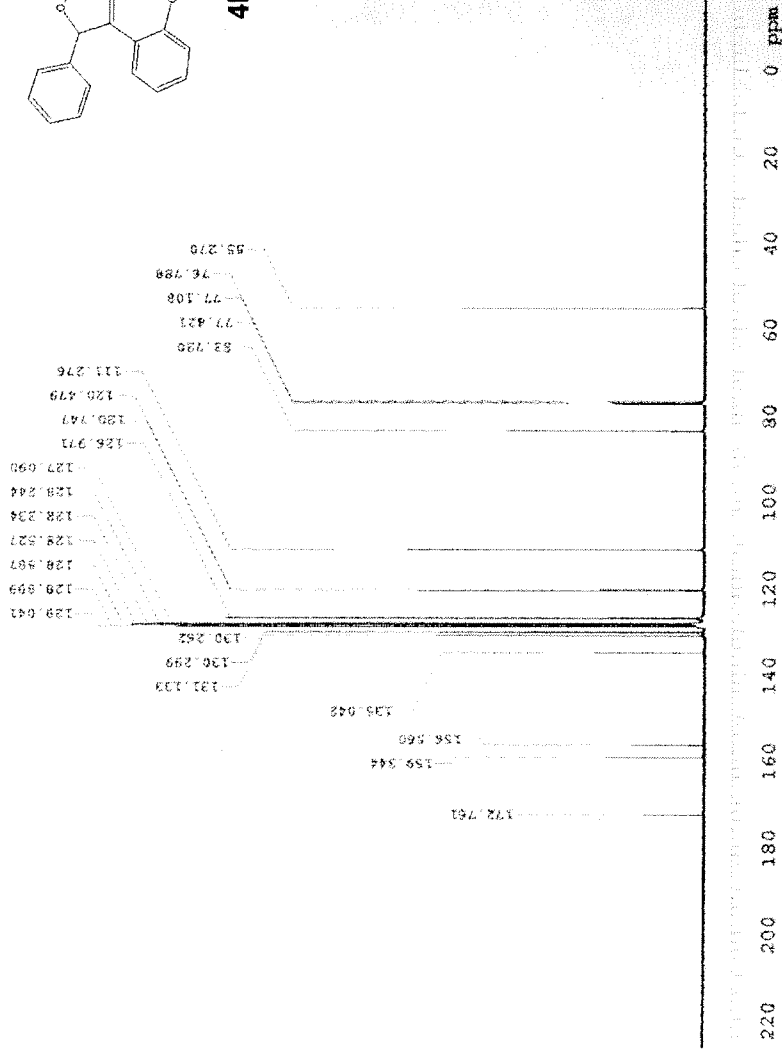
Pulse Sequence: Carbon (s2pw1)
Solvent: mcd3
Data collected on: Nov 9 2007

Temp: 23.0 C / 296.1 K
Operator: walkup
VAMMO-400 "um-400"

Relax. delay: 1.000 sec
Pulse: 45.0 degrees
Acq. time: 1.300 sec
Width: 24599.8 Hz
F12: 48000000
OBSERVE CH: 100.628839 MHz
DECUPLE CH: 519.523865 MHz
Power: 09 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
F2 size: 65536
Total time: 0 min 24 sec



4ka



NO-TU1913KX

13 FASIM 2007

Sample Name:

NO-TC13122P-13KASIM2007

Archive directory:

Sample directory:

File: Proton

Pulse Sequence: Proton (gZpg1)

Solvent: CDCl3

Data collected on: Nov 13 2007

Temp: 26.0 C / 289.1 K

Operator: wakupi

VMMS-400 "umz400"

Relax. delay: 1.000 sec

Pulse: 45.0 degrees

Acq. time: 2.948 sec

Width: 6410.3 Hz

8 repetitions

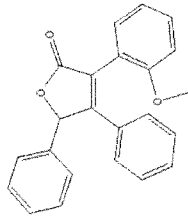
OBSERVE: H1, 399.521916 MHz

DATA PROCESSING

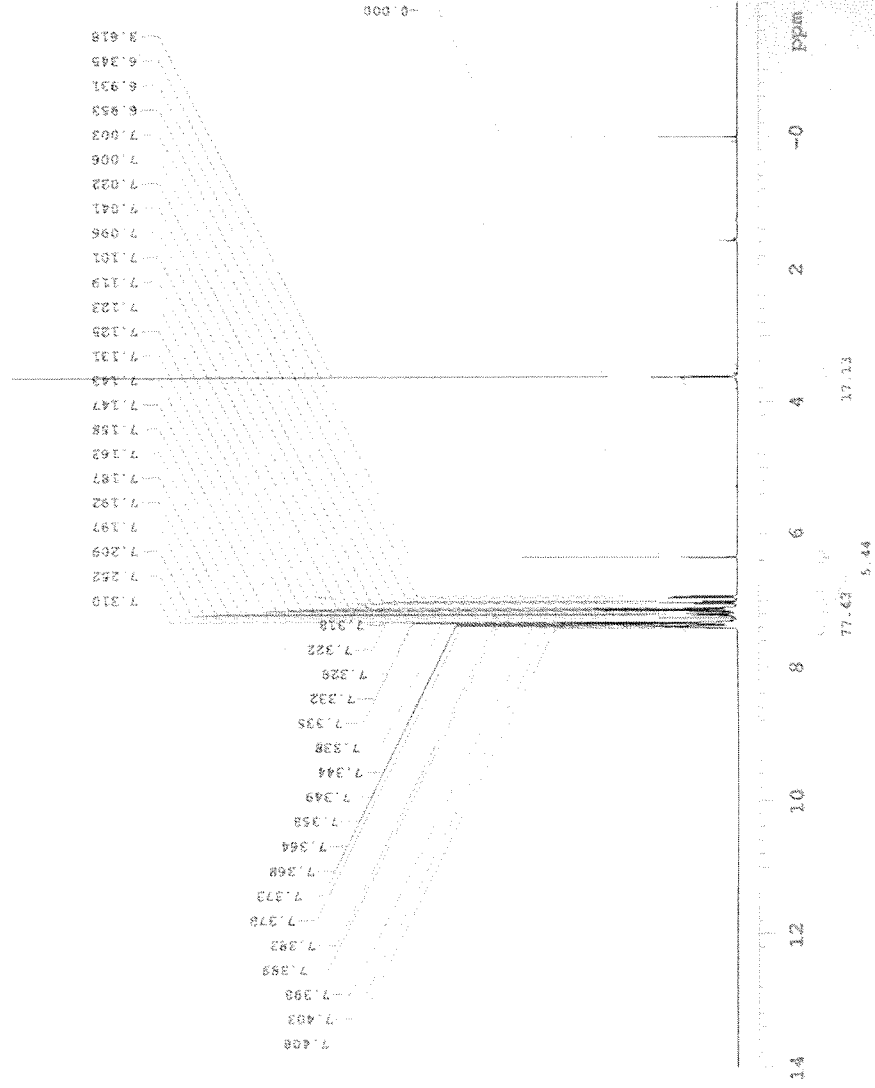
Line broadening: 0.2 Hz

FT size: 65536

Total time: 0 min 24 sec



4ka'



NO-TCI13912KP
NO-TCI13912KP
13 KASIM 2007

Sample Name:
NO-TCI13912KP-13KASIM2007
Archive directory:

Sample directory:

Filefile: Carbon

Pulse Sequence: Carbon (g2pal)
Solvent: cdcl3
Data collected on: Nov 13 2007

Temp. 26.0 C / 259.1 K
Operator: walkapl
VMS6-400 "ims400"

Pulpr. delay 1.000 sec
Pulpr. 45.0 degrees
Acq. time 1.300 sec
Width 24509.0 Hz
312 repetitions

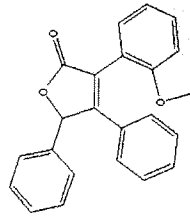
OBSERVE C13 100.4598234 MHz
DECOUPLE H1 399.5279865 MHz
Pulpr 39 MHz

continuously on
WALTZ-16 modulated

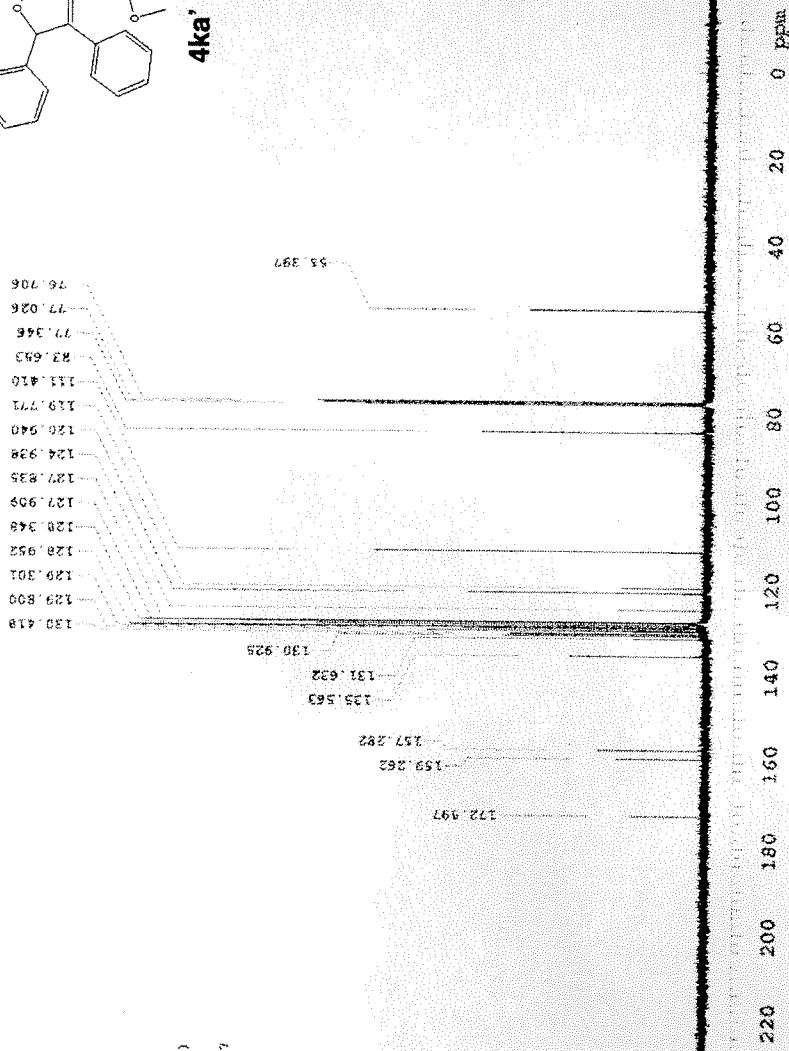
DATA PROCESSING

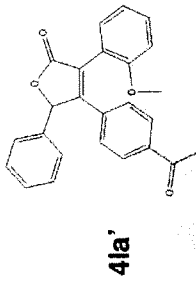
Line broadening 0.5 Hz
F2 file 03236

Total time 0 min 34 sec



4ka'





NORCAN-F20.PROCES

Sample Name:
 NORCAN-F20.KANP2
 Data Collected on:
 pmz400-vrmz400
 Archive directory:
 /home/walrupl/vrmz400/data
 Sample directory:
 NORCAN-F20.KANP2_13MAR2006
 File(s): F20TEN

Full Sample Name: NORCAN (S2701)

Solvent: 20013

Data collected on: Mar 21 2006

Time: 20.0 C / 279.10K C

Operator: walrupl

Injection Delay: 0.00 sec

Injection Volume: 0.500000

Injection Rate: 1.500000 mL/min

Injection Temperature: 20.000000

Injection Pressure: 0.000000 MPa

Injection Flow Rate: 0.000000 mL/min

Injection Volume: 0.500000

Injection Rate: 1.500000 mL/min

Injection Pressure: 0.000000 MPa

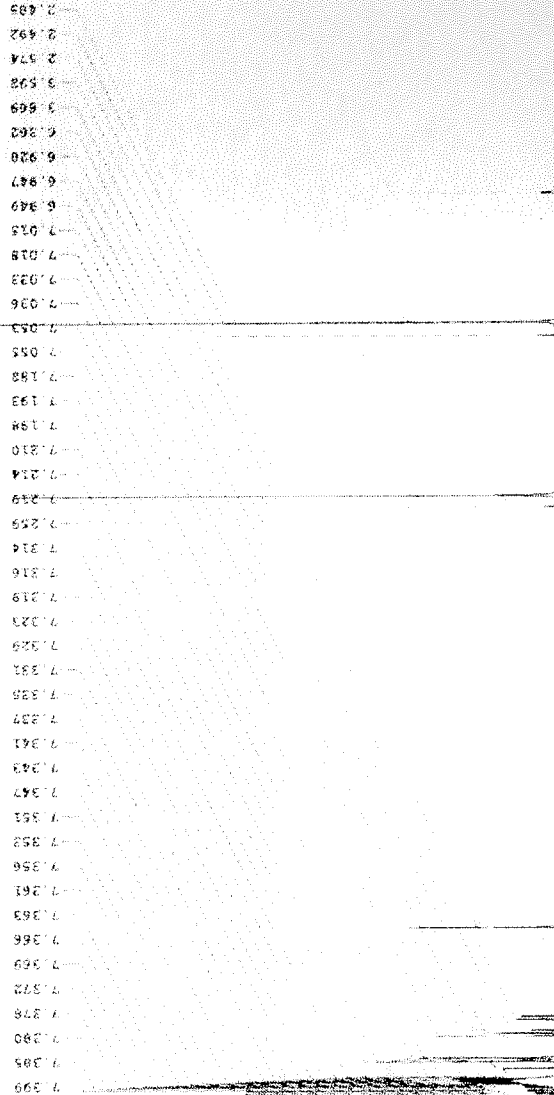
Injection Flow Rate: 0.000000 mL/min

Injection Volume: 0.500000

Injection Rate: 1.500000 mL/min

Injection Pressure: 0.000000 MPa

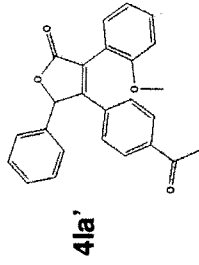
Injection Flow Rate: 0.000000 mL/min



Retention Time (min)	Area	Height
7.390		
7.395		
7.400	14.62	
7.405		
7.410		
7.415		
7.420		
7.425		
7.430		
7.435		
7.440		
7.445		
7.450		
7.455		
7.460		
7.465		
7.470		
7.475		
7.480		
7.485		
7.490		
7.495		
7.500		
7.505		
7.510		
7.515		
7.520		
7.525		
7.530		
7.535		
7.540		
7.545		
7.550		
7.555		
7.560		
7.565		
7.570		
7.575		
7.580		
7.585		
7.590		
7.595		
7.600		
7.605		
7.610		
7.615		
7.620		
7.625		
7.630		
7.635		
7.640		
7.645		
7.650		
7.655		
7.660		
7.665		
7.670		
7.675		
7.680		
7.685		
7.690		
7.695		

9.06 20.725.01
 33.32 5.19 4.77

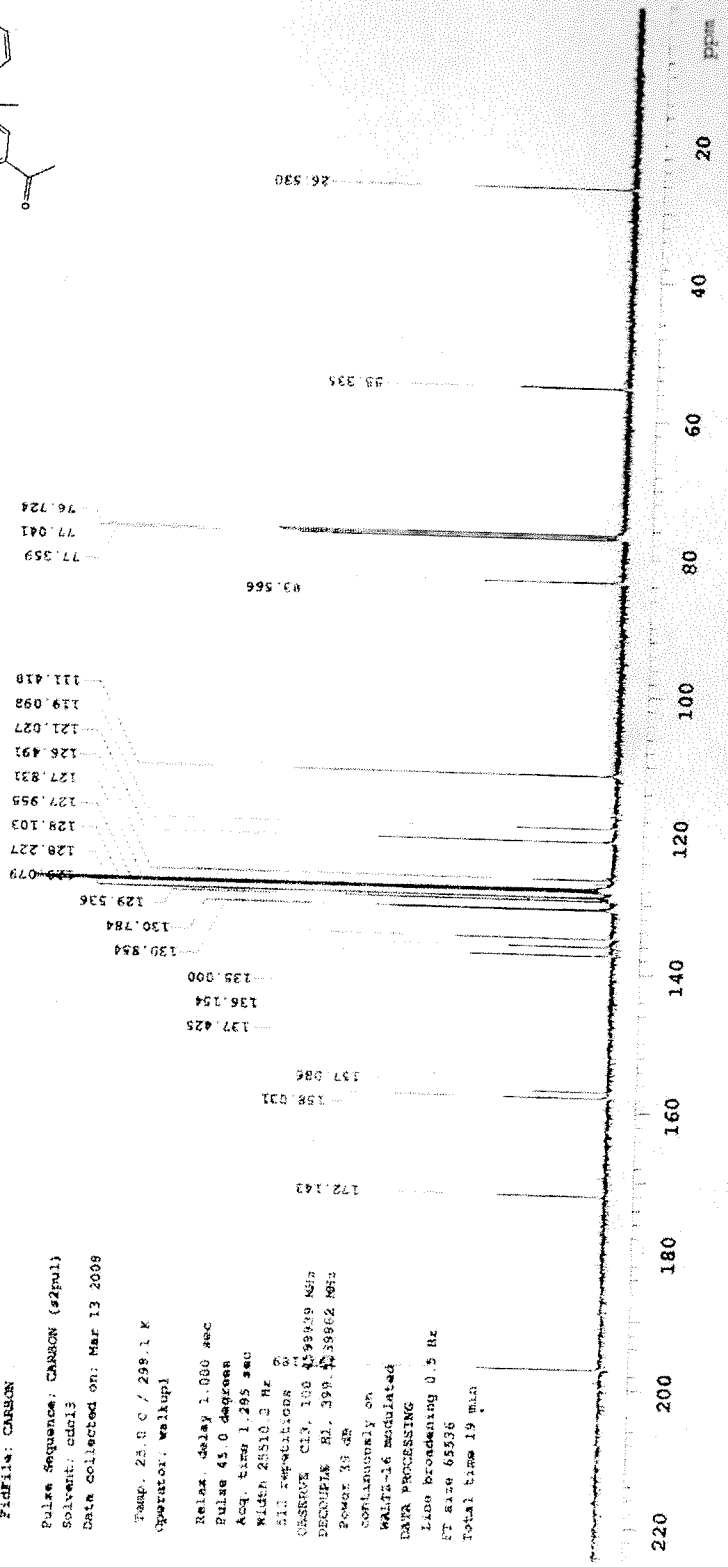
14.62



NURCAN-F203R00PS
 Sample Name:
 NURCAN-F203R00PS
 Data Collected on:
 mmx400-vmrms400
 Archive directory:
 /home/walkup/vmrmsys/data
 Sample directory:
 NURCAN-F203R00PS_13Mar2000
 Fidfile: CARBON

Pulse Sequence: CARBON (s2pul)
 Solvent: cdcl3
 Data collected on: Mar 13 2000

Temp.: 25.0 C / 298.1 K
 Operator: walkup
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.295 sec
 Width 26510.0 Hz
 SII repetitions 2
 OBSERVE CH: 13C 459949 MHz
 DECOUPLE CH: 399.4038662 MHz
 Power 30 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 0.5 Hz
 FT scan 65336
 Total time 19 min



NURCM-F203MX2

Sample Name:

NURCM-F203MX2

Data Collected on:

nmr500-vmr4d10

Archive directory:

/home/walkup/vmrays/data

Sample directory:

0034-F203MX2-000-000

0034-F203MX2-000-000

0034-F203MX2-000-000

0034-F203MX2-000-000

0034-F203MX2-000-000

0034-F203MX2-000-000

0034-F203MX2-000-000

0034-F203MX2-000-000

0034-F203MX2-000-000

0034-F203MX2-000-000

0034-F203MX2-000-000

0034-F203MX2-000-000

0034-F203MX2-000-000

0034-F203MX2-000-000

0034-F203MX2-000-000

0034-F203MX2-000-000

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0034-F203MX2-000-000

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0034-F203MX2-000-000

0034-F203MX2-000-000

0034-F203MX2-000-000

0034-F203MX2-000-000

0034-F203MX2-000-000

0034-F203MX2-000-000

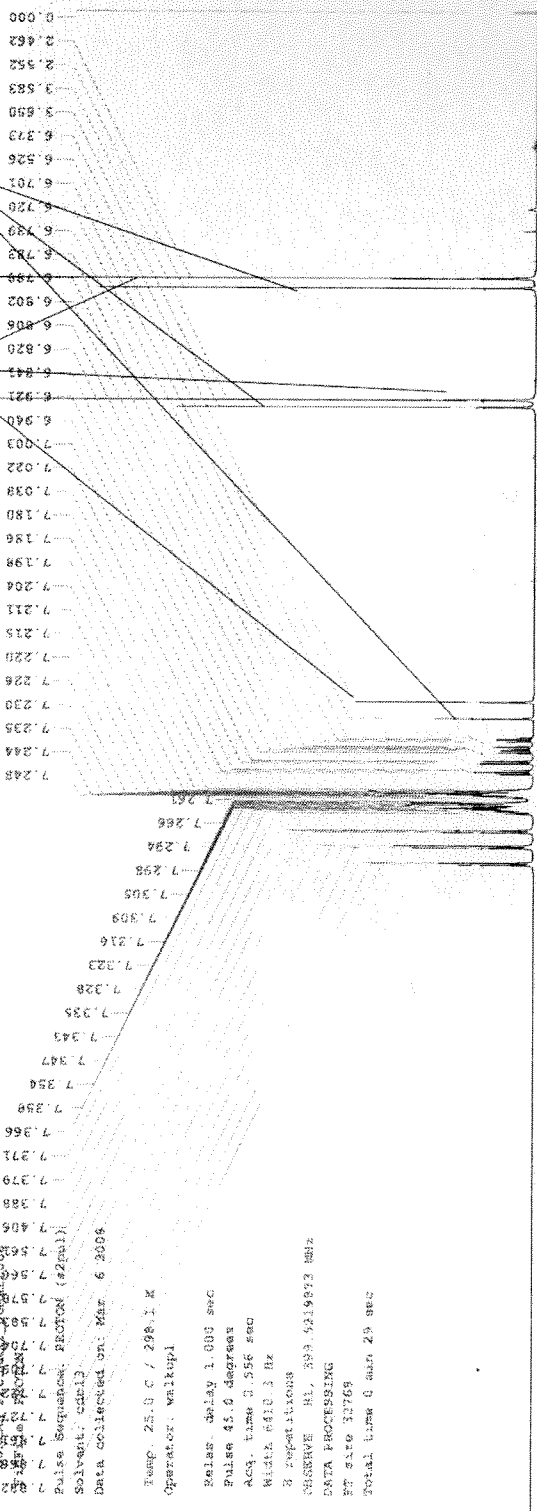
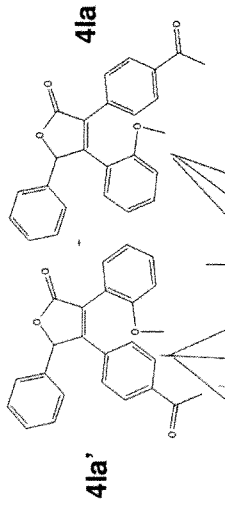
0034-F203MX2-000-000

0034-F203MX2-000-000

0034-F203MX2-000-000

0034-F203MX2-000-000

0034-F203MX2-000-000



Temp: 25.0 C / 298.1 K
 Operator: walkup
 Pulse delay: 1.000 sec
 Pulse: 45.0 degrees
 Acq. time: 3.556 sec
 Width: 6410.0 Hz
 3 repetitions
 CHANNEL: HL 500.1319873 MHz
 DATA PROCESSING
 FT date: 12/09
 Total time: 8 min 29 sec

6.0421030 12.943 0.23
 9.20 9.13
 5.74 5.74

VARIAN

YO-1205KC

Sample Name:
YO-1205KC

Data Collected on:
nmr400-vmrw400

Archive directory:
/home/walkup/vmrw400/data

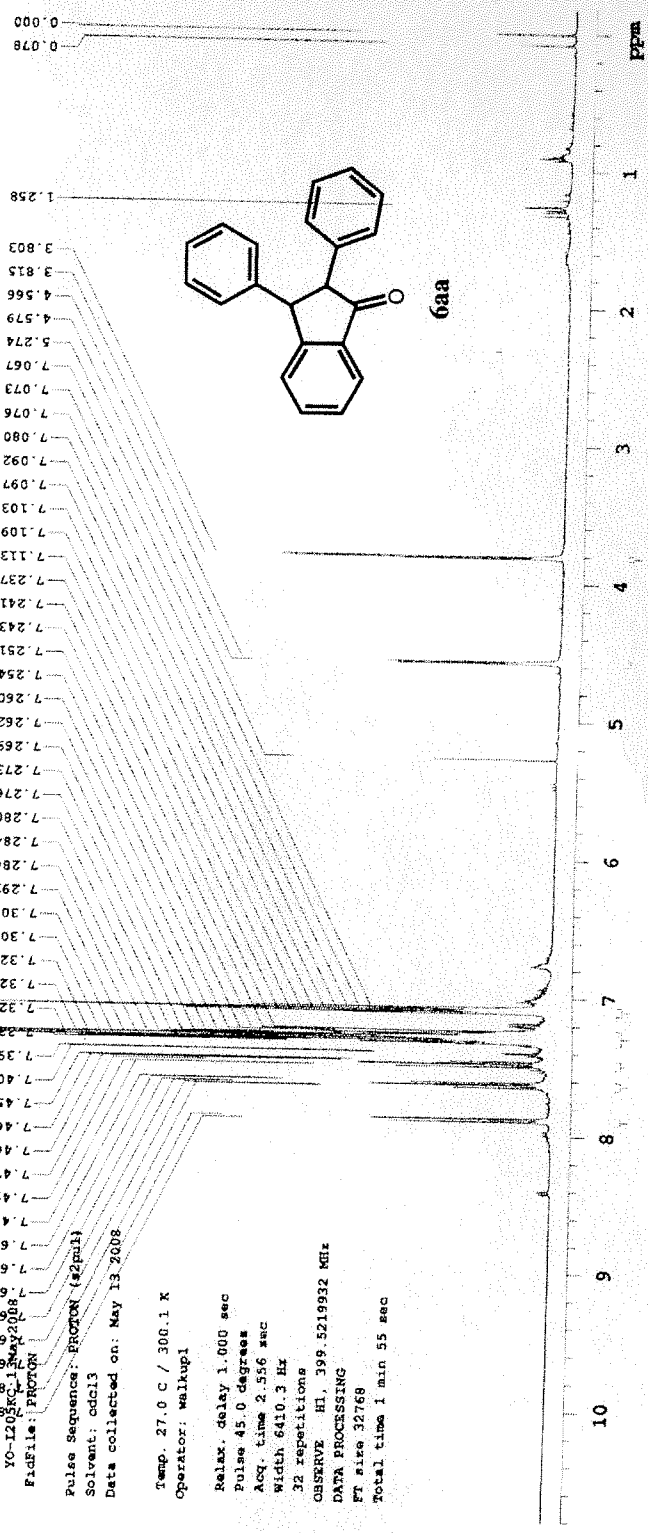
Sample date: 05/13/2008
YO-1205KC_13May2008
FidFile: PROTON

Pulse Sequence: PROTON (2pul)
Solvent: cdcl3
Data collected on: May 13 2008

Temp. 27.0 C / 300.1 K
Operator: walkup

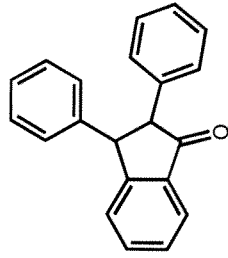
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
32 repetitions

CONSERVE HI, 399.5219932 MHz
DATA PROCESSING
FT size 32768
Total time 1 min 55 sec

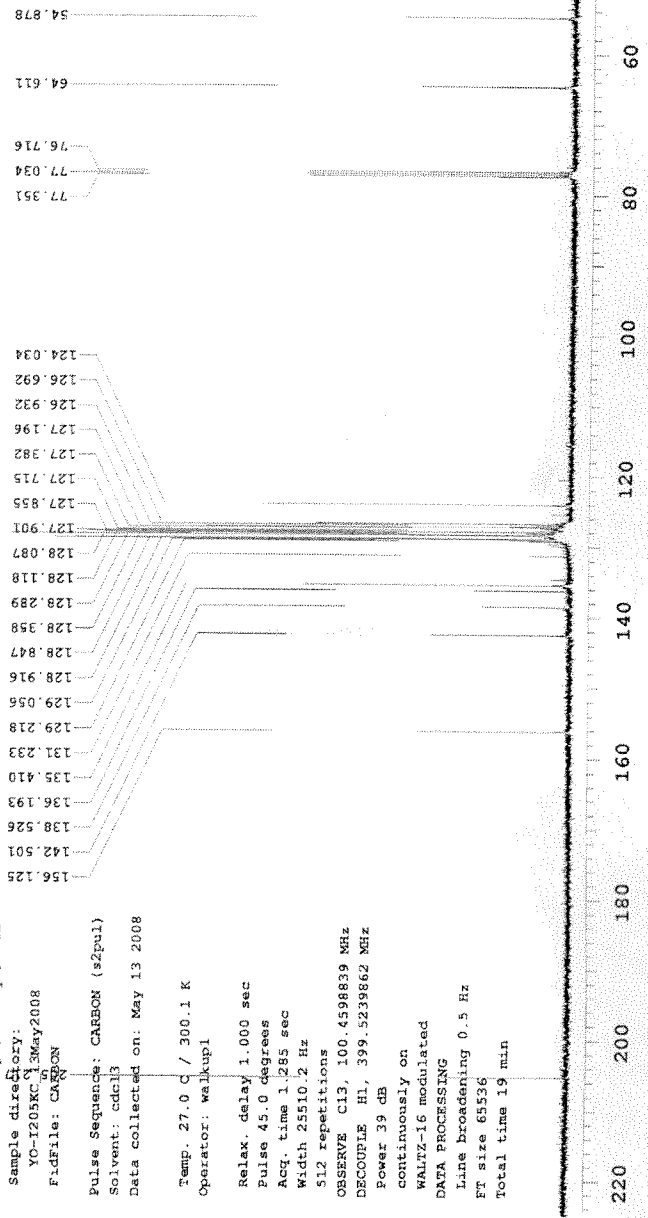




VARIAN



6aa



YO-1205KC

Sample Name: YO-1205KC
 Data Collected on: nmr400-vmr400
 Archive directory: /home/walkup/vmr400/data
 Sample directory: YO-1205KC_May2008
 File: CARBON

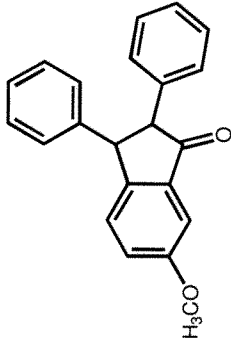
Pulse Sequence: CARBON (s2pul)
 Solvent: cdcl3
 Data collected on: May 13 2008

Temp. 27.0 C / 300.1 K
 Operator: walkup

Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.285 sec
 Width 25510.2 Hz
 512 repetitions
 OBSERVE C13, 100.4598839 MHz
 DECOUPLE H1, 399.5239862 MHz
 Power 39 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 65536
 Total time 19 min



VARIAN



YO-1220KA
25 OCAK 2008

Sample Name:
YO-1220KA-25OAK2008
Archive directory:

Sample directory:

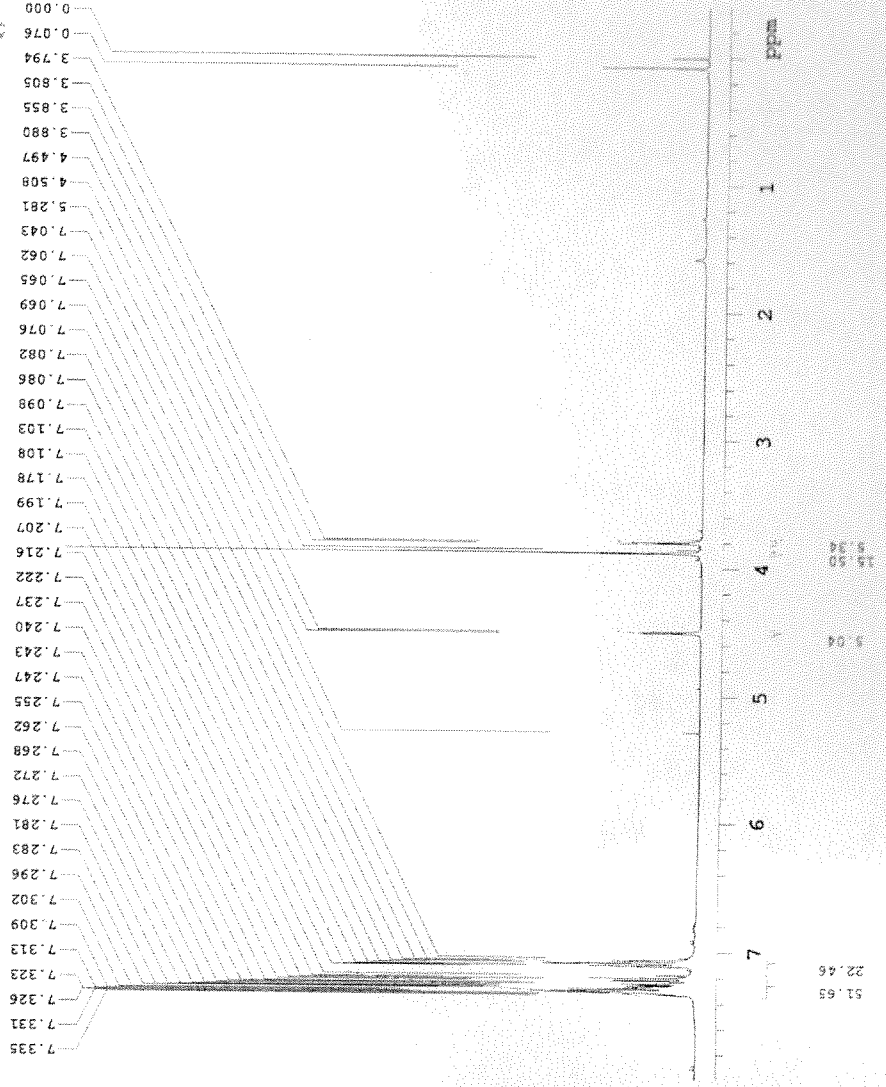
FidFile: Proton

Pulse Sequence: Proton (s2pul)
Solvent: cdcl3
Data collected on: Jan 25 2008

Temp. 25.0 C / 298.1 K
Operator: walkupl
VNMR5-400 "nmr400"

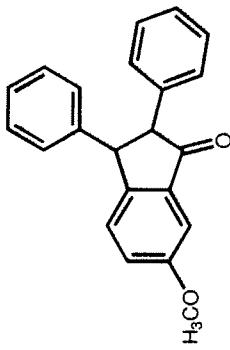
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 6410.3 Hz
8 repetitions

OBSERVE HI, 399.5219920 MHz
DATA PROCESSING
Line broadening 0.2 Hz
Ft size 65536
Total time 0 min 24 sec





VARIAN



YO-I220KA
 YO-I220KA
 25 OCAK 2008

Sample Name:
 YO-I220KA-25OCAK2008
 Archive directory:

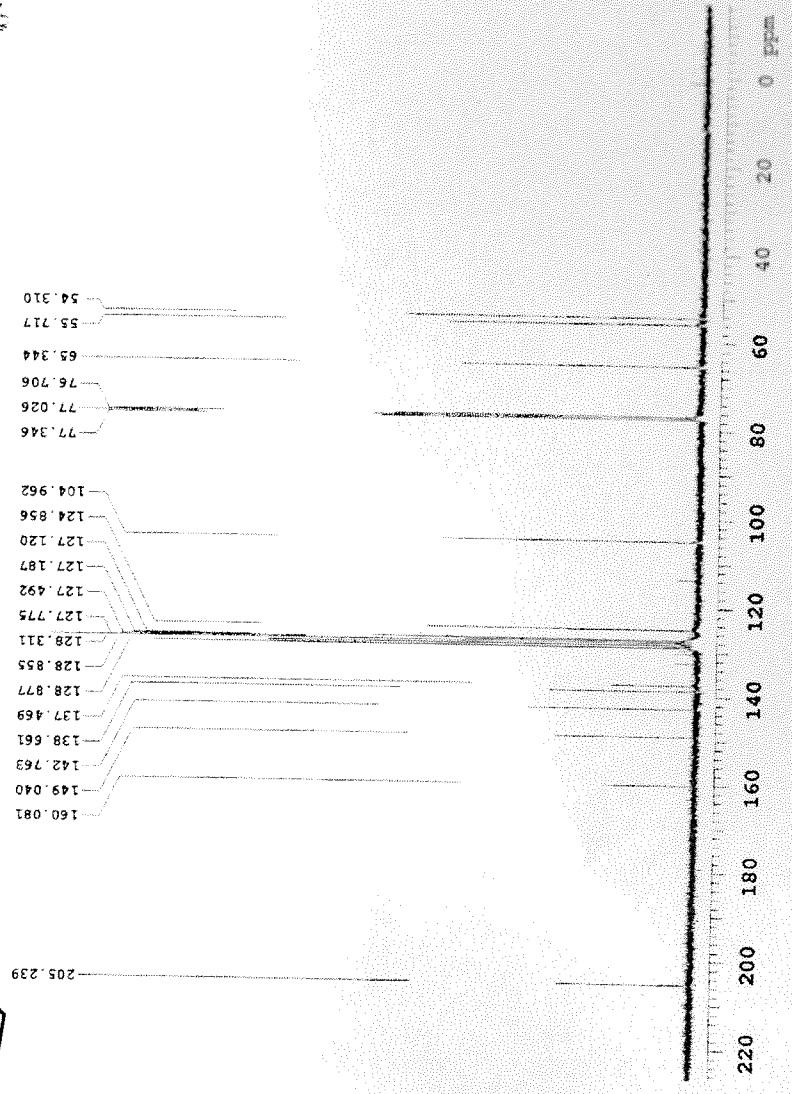
Sample directory:

FidFile: Carbon

Pulse Sequence: Carbon (s2pul)
 Solvent: cdcl3
 Data collected on: Jan 25 2008

Temp. 25.0 C / 298.1 K
 Operator: walkupl
 VNMR-400 "nmr400"

Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 24509.8 Hz
 512 repetitions
 OBSERVE C13, 100.4598839 MHz
 DECOUPLE H1, 399.5239665 MHz
 Power 39 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 65536
 Total time 0 min 24 sec



YELDA-YO-I220KB

Sample Name: YELDA-YO-I220KB
Data Collected on: nmr400-nmrs400

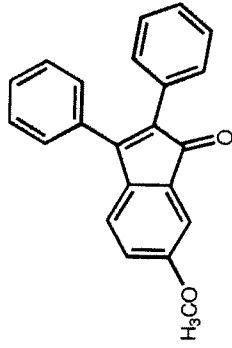
Archive directory: /home/walkup1/nmrs400/data
Sample directory: 041707
YELDA-YO-I220KB_19Mar2008
Fidfile: PROTON

Pulse Sequence: PROTON (s2pul)
Solvent: cdc13
Data collected on: Mar 19 2008

Temp. 25.0 C / 299.1 K
Operator: walkup1

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
8 repetitions

OBSERVE H1, 399.5219993 MHz
DATA PROCESSING
FT size 32768
Total time 0 min 29 sec

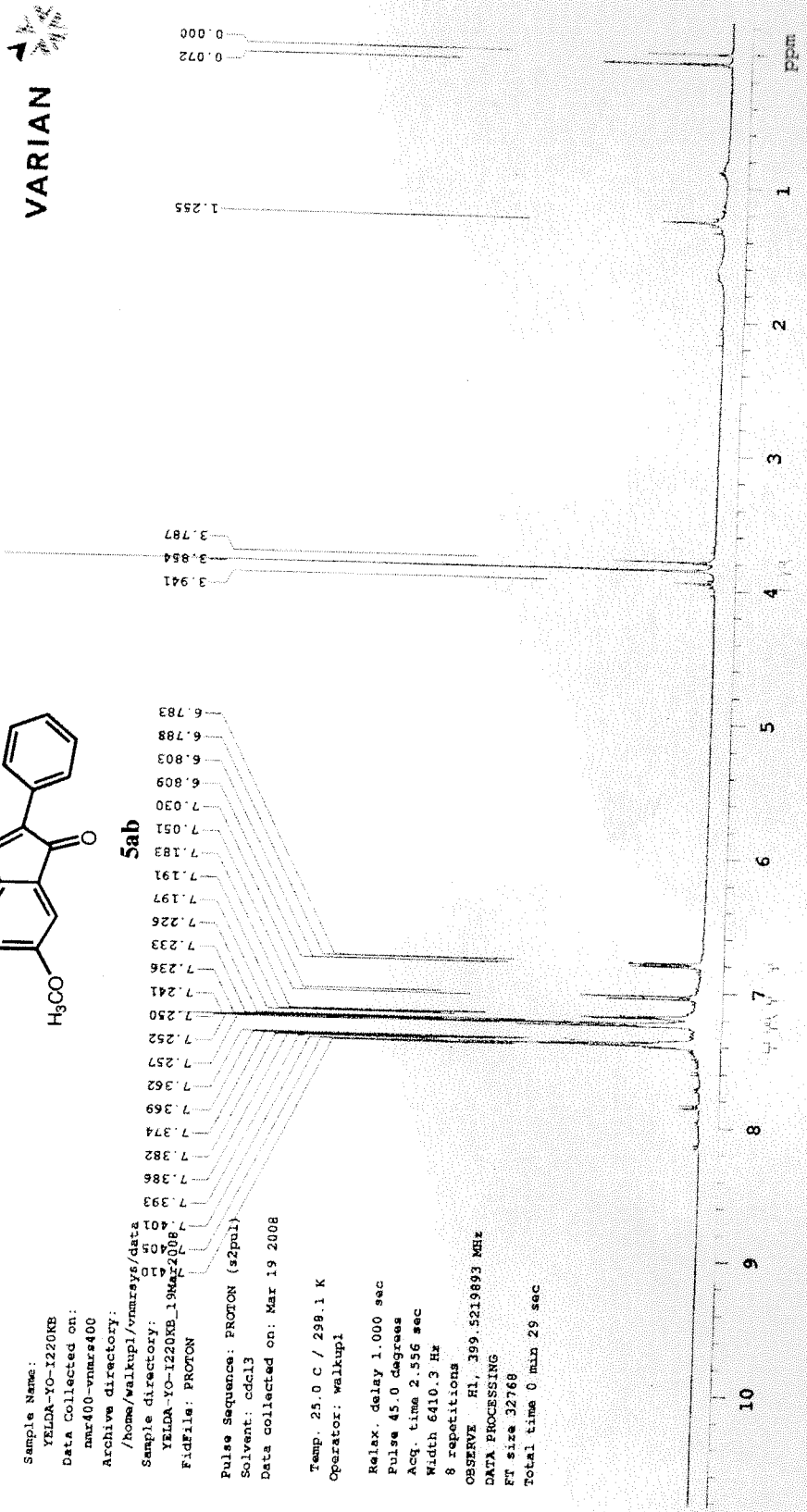


5ab

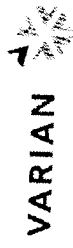
6.783
6.788
6.803
6.809
7.030
7.051
7.183
7.191
7.197
7.226
7.233
7.236
7.241
7.250
7.252
7.257
7.352
7.369
7.374
7.382
7.386
7.393
7.401

3.941
3.854
3.787

1.255
0.072
0.000

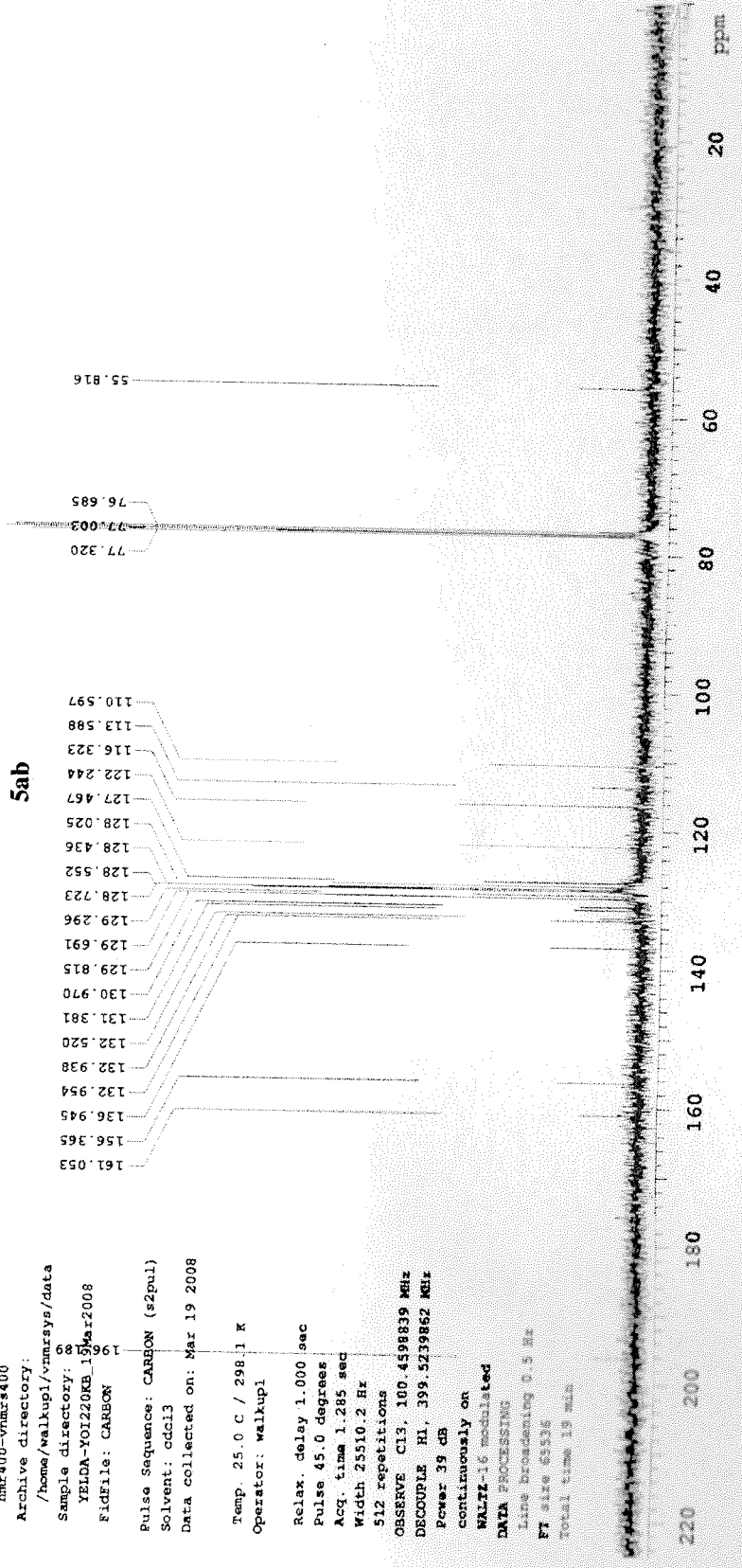
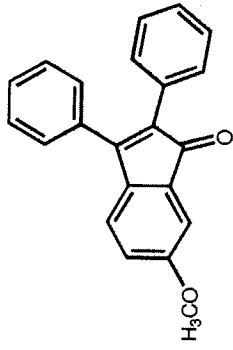


27.15
28.05
10.60
7.08
7.17
0.08
15.10
4.17





VARIAN



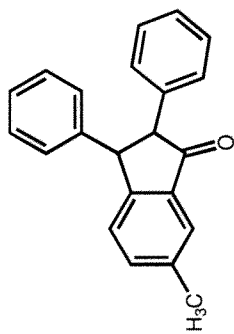
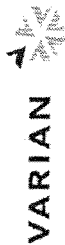
YELDA-Y01220KB

Sample Name: YELDA-Y01220KB
 Data Collected on: nmr400-vnmrs400
 Archive directory: /home/walkup/vnmrsys/data
 Sample directory: 98
 YELDA-Y01220KB_150_mar2008
 FidFile: CARBON 199

Pulse Sequence: CARBON (s2pul)
 Solvent: cdcl3
 Data collected on: Mar 19 2008

Temp. 25.0 C / 298.1 K
 Operator: walkup1

Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.285 sec
 Width 23510.2 Hz
 512 repetitions
 OBSERVE C13, 100.459839 MHz
 DECOUPLE H1, 399.5239852 MHz
 Power 39 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 6536
 Total time 13 min



6ac

YO-1214KA
23 OCAK 2008

Sample Name:
YO-1214KA-23OCAK2008
Archive directory:

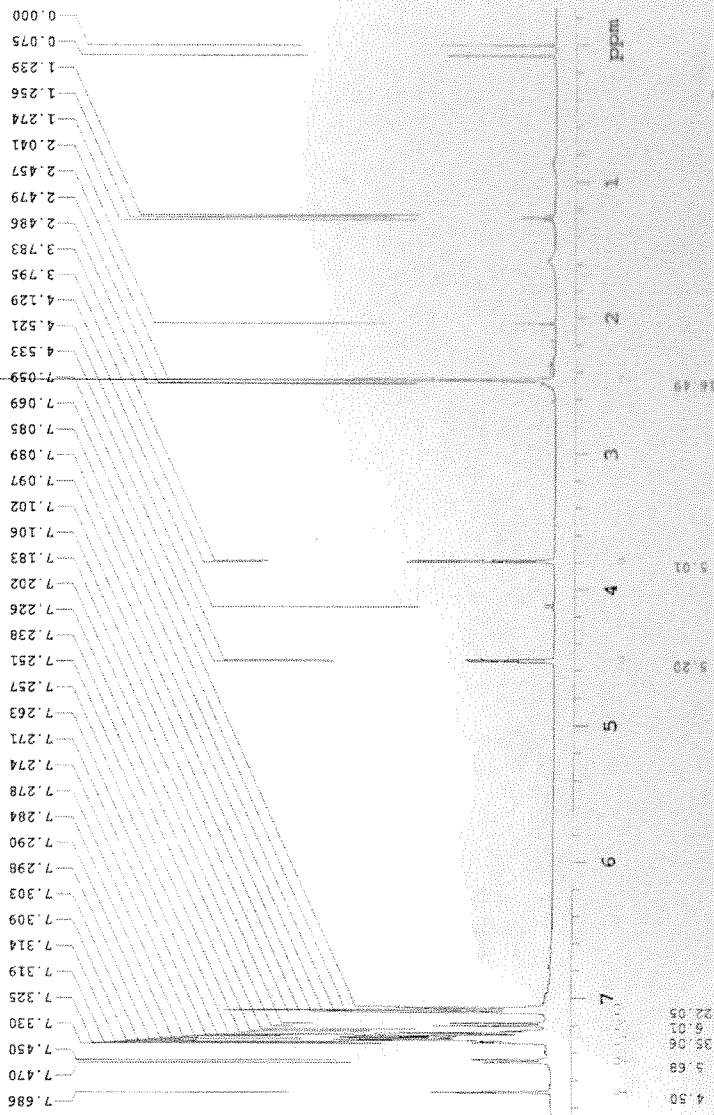
Sample directory:

Fidfile: Proton_Minsw

Pulse Sequence: Proton_Minsw (s2pul)
Solvent: cdc13
Data collected on: Jan 23 2008

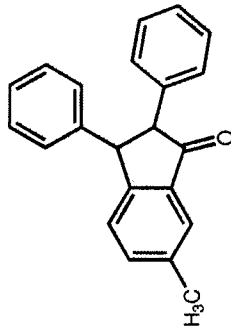
Temp. 25.0 C / 298.1 K
Operator: walkupl
VNMR5-400 "nmr400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 3980.9 Hz
8 repetitions
OBSERVE H1, 399.521906 MHz
DATA PROCESSING
Line broadening 0.2 Hz
Ft size 65536
Total time 0 min 34 sec





VARIAN



6ac

YO-I214KA
YO-I214KA
23 OCAK 2008

Sample Name:
YO-I214KA-23OAK2008
Archive directory:

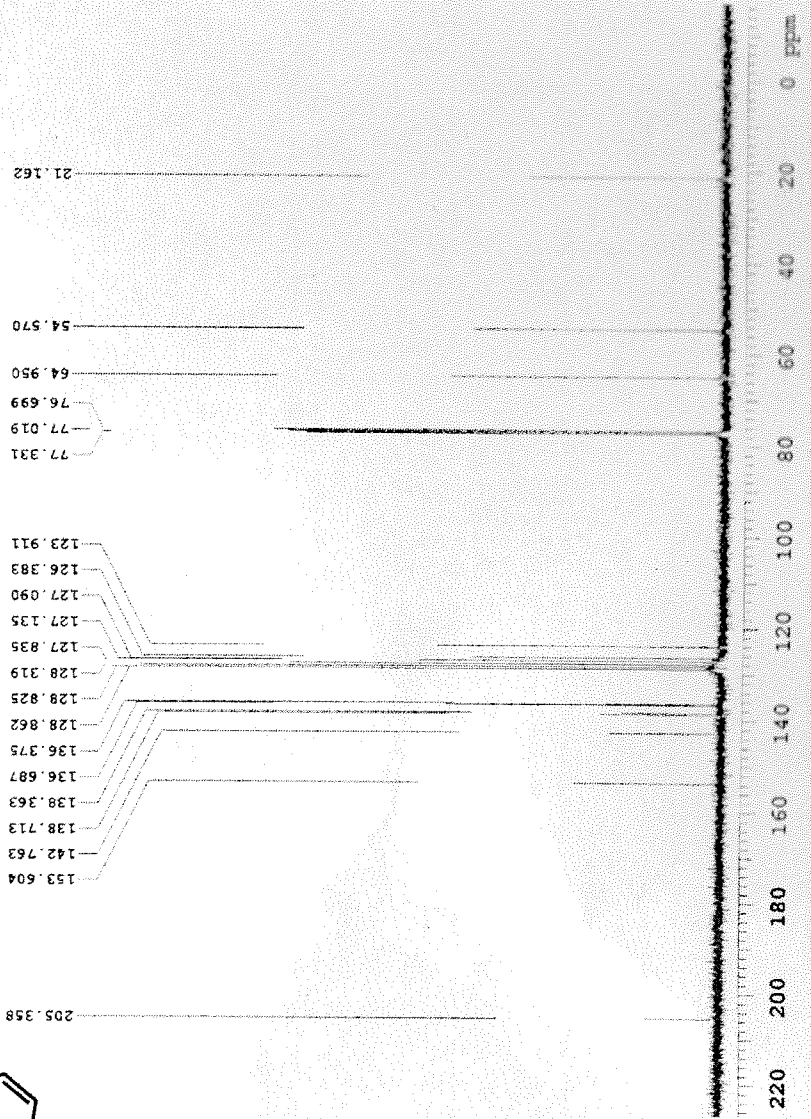
Sample directory:

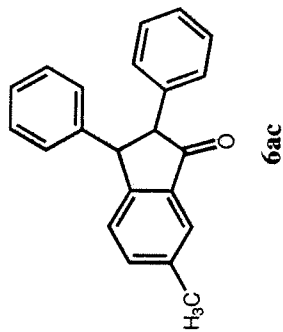
FidFile: Carbon

Pulse Sequence: Carbon (s2pul)
Solvent: cdcl3
Data collected on: Jan 23 2008

Temp. 25.0 C / 298.1 K
Operator: walkupl
VNMRS-400 "nmr400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
512 repetitions
OBSERVE C13, 100.459839 MHz
DECOUPLE H1, 399.5239865 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
Ft size 65536
Total time 0 min 24 sec





YO-I214EA
YO-I214FA
23 OCAK 2008

Sample Name:
YO-I214KA-23OCAK2008
Archive directory:

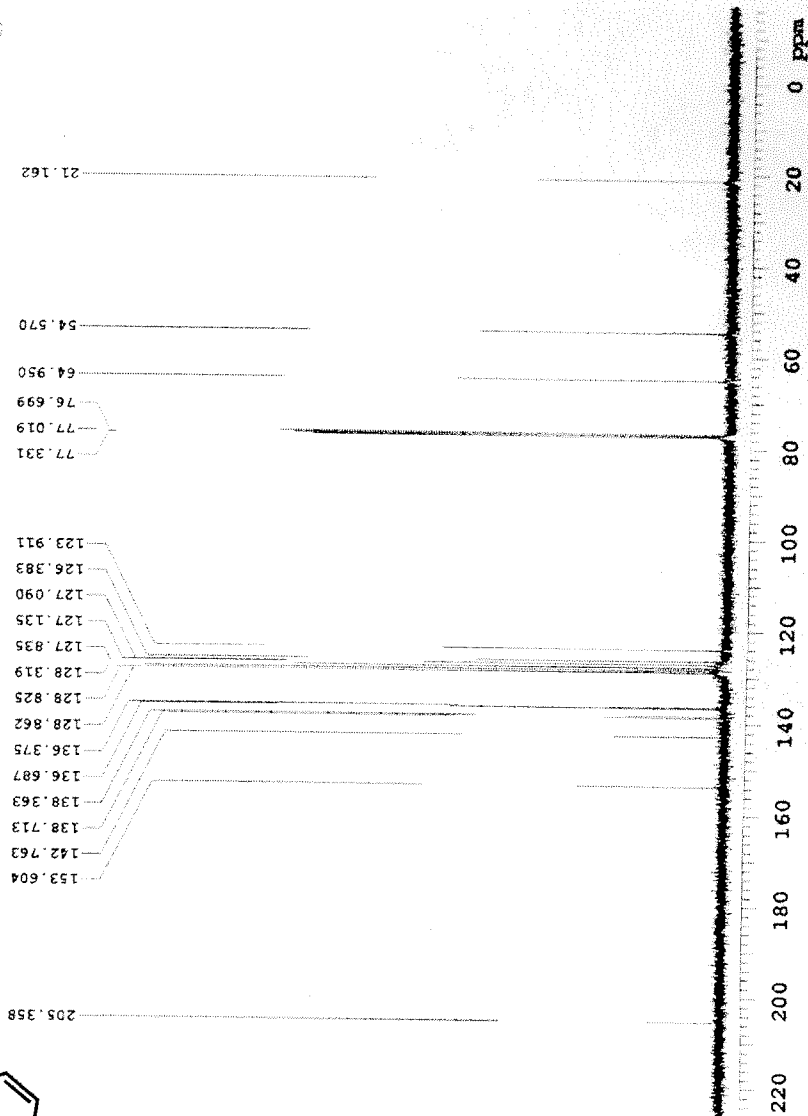
Sample directory:

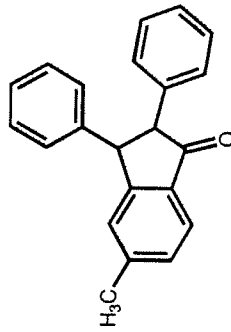
FidFile: Carbon

Pulse Sequence: Carbon (s2pul)
Solvent: cdcl3
Data collected on: Jan 23 2008

Temp. 25.0 C / 298.1 K
Operator: walkupl
VNMR-400 "nmr400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
512 repetitions
OBSERVE C13, 100.4598839 MHz
DECOUPLE H1, 399.5239865 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 0 min 24 sec





YELDA-1223KA

Sample Name:

YELDA-1223KA

Data Collected on:

nmr400-vmr4400

Archive directory:

/home/walkup/vmr4400/data

Sample directory:

YELDA-1223KA_21Mar2008

Fidfile: PROTON

Pulse Sequence: PROTON (szpul)

Solvent: cdcl3

Data collected on: Mar 21 2008

Temp. 25.0 C / 298.1 K

Operator: walkup

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.556 sec

Width 6410.3 Hz

8 repetitions

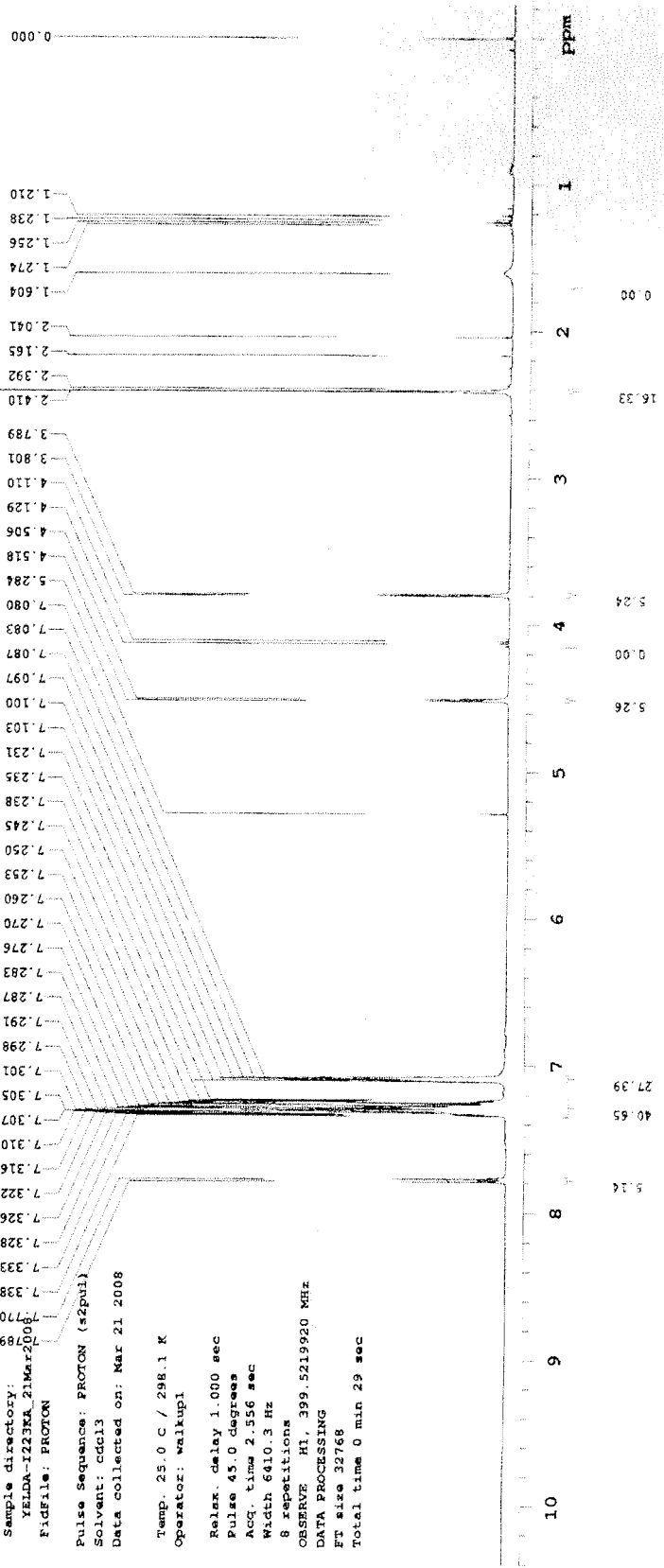
OBSERVE H1, 399.5219920 MHz

DATA PROCESSING

Ft size 32768

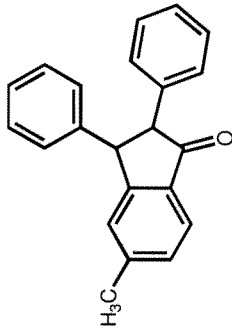
Total time 0 min 29 sec

6ad





VARIAN



YELDA-1223KA

Sample Name:

YELDA-1223KA

Data Collected on:

nmr400-vmars400

Archive directory:

/home/walkupl/vmarsys/data

Sample directory:

YELDA-1223KA_21Mar2008

FidFile: CARBON

Pulse Sequence: CARBON (szpul)

Solvent: cdcl3

Data collected on: Mar 21 2008

Temp. 25.0 C / 298.1 K

Operator: walkupl

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.285 sec

Width 23510.2 Hz

512 repetitions

OBSERVE C13, 100.4598839 MHz

DECOUPLE H1, 399.5239862 MHz

Power 39 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz

FT size 65536

Total time 19 min

6ad

123.902
126.901
127.118
127.932
128.304
128.823
128.893
128.893
129.606
133.961
138.820
142.695
146.779
156.690

22.167

54.831

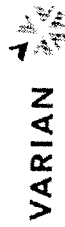
64.728

76.708

77.026

77.344

220 200 180 160 140 120 100 80 60 40 20 ppm



YELDA-1225KA

Sample Name:

YELDA-1225KA

Data Collected on:

nmr400-vnmrs400

Archive directory:

/home/walkup/vnmrsz/data

Sample directory:

YELDA-1225KA_24Mar2008

FidFile: PROTON

Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: Mar 24 2008

Temp. 25.0 C / 298.1 K

Operator: walkup

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.556 sec

Width 6410.3 Hz

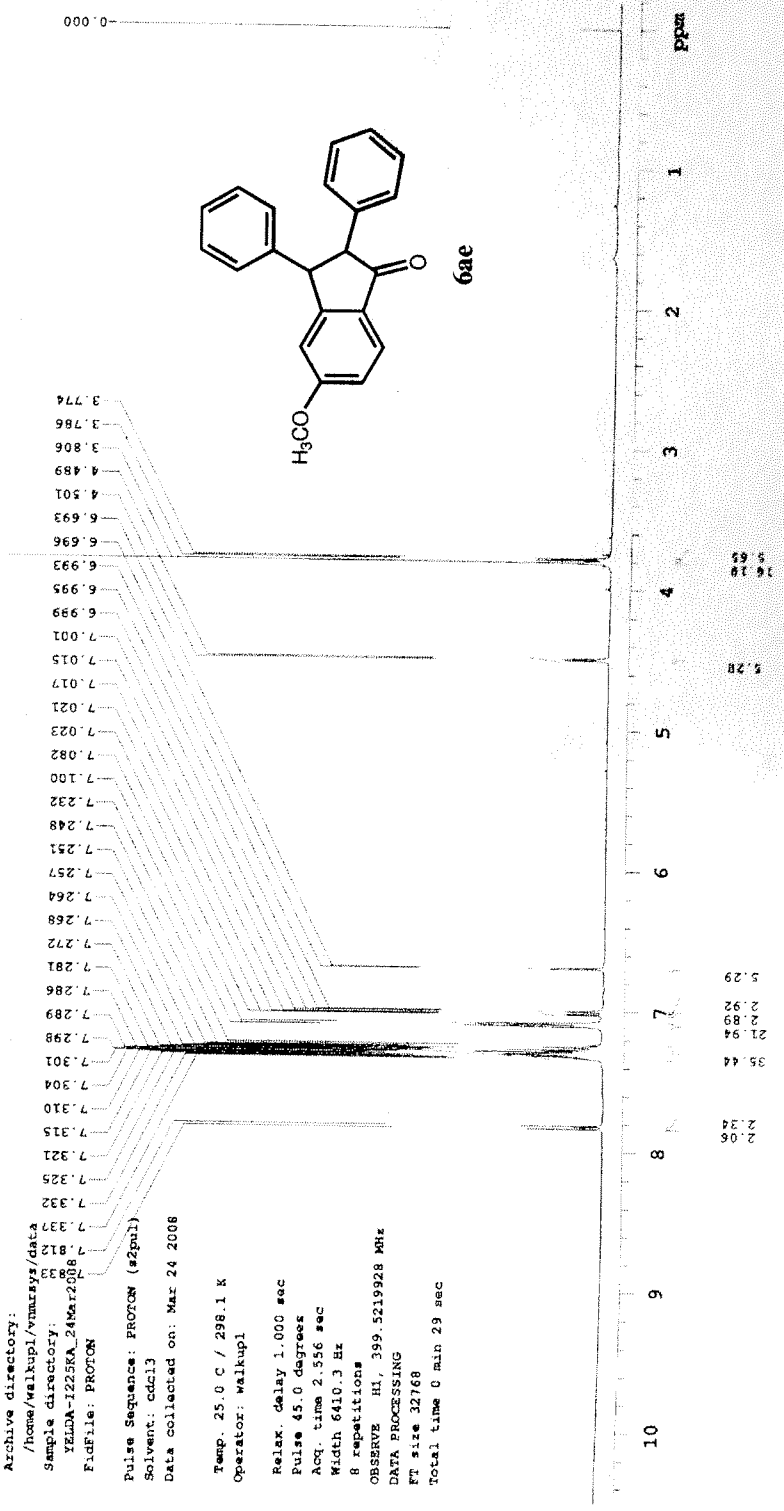
8 repetitions

OBSERVE H1, 399.521928 MHz

DATA PROCESSING

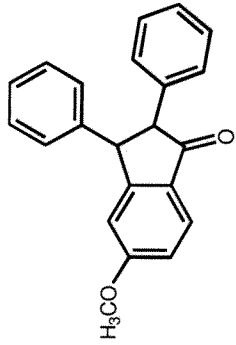
FT size 32768

Total time 0 min 23 sec





VARIAN



6ac

YELDA-1225KA

Sample Name:

YELDA-1225KA

Data Collected on:

nmr400-vmrs400

Archive directory:

/home/walkup1/vmrsys/data

Sample directory:

YELDA-1225KA_24Mar2008

Fidfile: CARBON

Pulse Sequence: CARBON (s2pul1)

Solvent: cdcl3

Data collected on: Mar 24 2008

Temp: 25.0 C / 298.1 K

Operator: walkup1

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.265 sec

Width 25510.2 Hz

512 repetitions

OBSERVE C13, 100.4598639 MHz

DECOUPLE H1, 399.5239862 MHz

Power 39 dB

continuously on

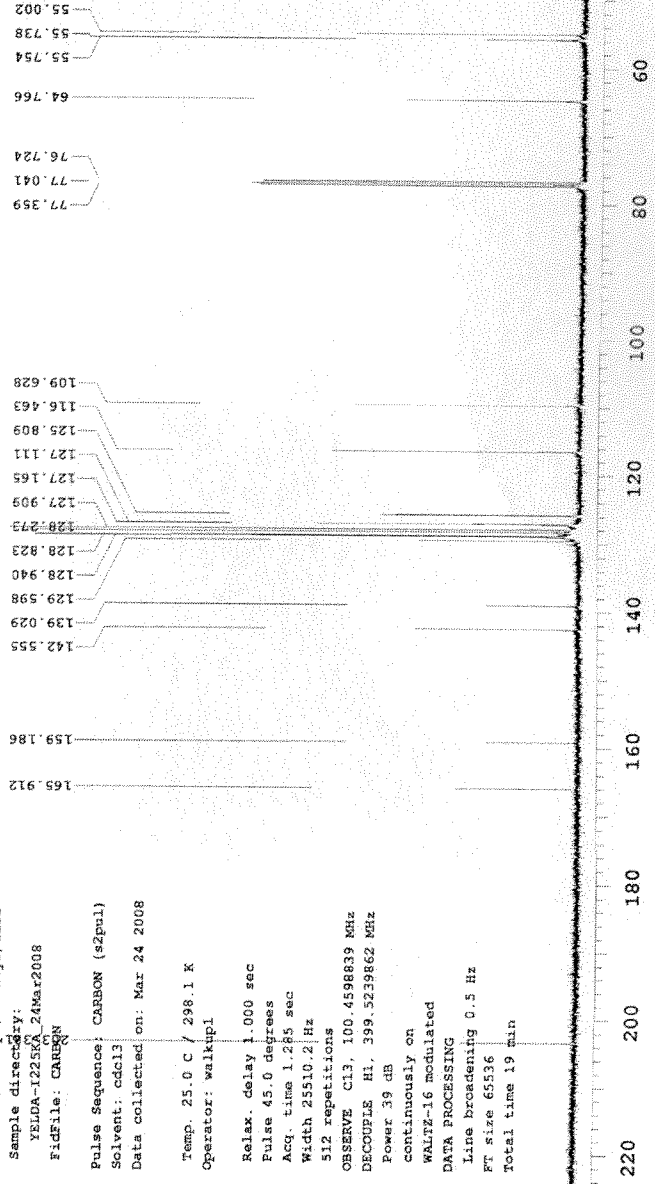
WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz

FT size 65536

Total time 19 min



**TÜBİTAK
PROJE ÖZET BİLGİ FORMU**

Proje No: 10T385
Proje Başlığı: Paladyum ve Rodyum Katalizli Tandem Karbonilatif Kenetlenme Tepkimeleri
Proje Yürütücüsü ve Araştırmacılar: Levent Artok (yürütücü), Özge Aksın-Artok, Melih Kuş, Fatma Nurcan Dege, Fatma Yelda Özkılıç
Projenin Yürütüldüğü Kuruluş ve Adresi: İzmir Yüksek Teknoloji Enstitüsü, Fen Fak., Kimya Böl., Urla 35430, İzmir
Destekleyen Kuruluş(ların) Adı ve Adresi:
Projenin Başlangıç ve Bitiş Tarihleri: 1.9.2006-1.9.2008
Öz (en çok 70 kelime) <p>Alkin ve organoboron karışımlarının CO gazı altında rodyum katalizli tepkimeleri gerçekleştirilmiştir. Atom ekonomik olan bu yöntem başlıca 5-aril-2(5H)-furanon, α,β-doymamış keton ve indanon ürünleri vermektedir. Tepkime koşulları yöntemin ürün seçimliliğinde anahtar rolü üstlenmektedir.</p>
Anahtar Kelimeler: Karbonilasyon, alkin, organoboron, rodyum, enon, furanon, indanon
Projeden Yapılan Yayınlar: Kuş M., Artok-Aksın Ö., Zıyanak F., Artok, L., <i>Synlett</i> , 2587, (2008).