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Stereoselective Synthesis of Highly Functionalized Cyclobutenes. A Facile Route to Electron-Deficient 1,3-Dienes

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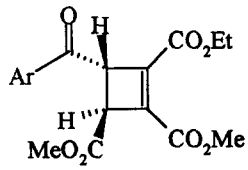
Abstract: Acetoacetanilide undergoes a smooth reaction with triphenylphosphine and dialkyl acetylenedicarboxylates to produce dialkyl 2-(acetoacetanilide-2-yl)-3-(triphenylphosphoranilidene)-butandioates, which undergo intramolecular Wittig reaction to produce 2-methyl-3-(*N*-phenylcarbonyl)-1,4-dialkoxycarbonylcyclobutenes. These cyclobutene derivatives undergo electrocyclic ring-opening reactions in boiling toluene to produce highly electron-deficient 1,3-dienes.

INTRODUCTION

Although the common 5-, 6- and 7-membered ring cycloalkenes are produced fairly easily by intramolecular Wittig reaction, the formation of cyclopropenes and cyclobutenes have not received much attention [1]. Cyclobutenes are important intermediates in organic synthesis [2,3], and their synthetic study continues to attract much attention. During the last few decades several methods have been developed for the preparation of polysubstituted cyclobutenes [2-4]. We have recently [5] described the first synthesis of cyclobutene derivatives **1** from the stereoselective intramolecular Wittig reaction of a vinyltriphenylphosphonium salt with ethyl 4-aryl-2,4-dioxobutanoates. Cyclobutenes **1** undergo electrocyclic ring-opening reaction in boiling toluene to produce highly electron-deficient 1,3-dienes **2**.



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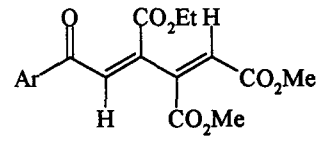

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As part of our current studies on the development of new routes to heterocyclic and carbocyclic systems, we now report a facile synthesis of stabilized ylides **4**, which are converted to functionalized cyclobutenes **5** *via* an intramolecular Wittig reaction. Compounds **5a-c** undergo electrocyclic ring-opening reactions to produce electron-deficient 1,3-dienes **6a-c** in fairly high yields. Thus, reaction of acetoacetanilide with dialkyl

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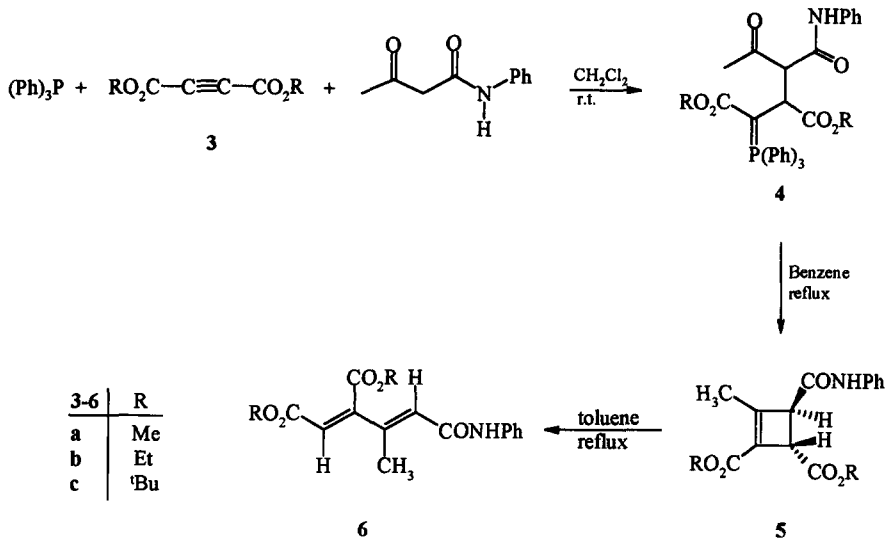
acetylenedicarboxylates **3** in presence of triphenylphosphine leads to the corresponding stabilized phosphorus ylides **4**, which are converted to cyclobutene derivatives **5**.



RESULTS AND DISCUSSION

On the basis of the chemistry of trivalent phosphorus nucleophiles [6,7], it is reasonable to assume that cyclobutene **5** results from initial addition of triphenylphosphine to the acetylenic ester and subsequent protonation of the reactive 1:1 adduct by acetoacetanilide. Then the positively charged ion is attacked by the enolate anion of acetoacetanilide to form the stable ylide **4**, which undergoes intramolecular Wittig reaction in boiling benzene to produce the cyclobutene derivatives **5a-c**. Compounds **5a-c** undergo electrocyclic ring-opening reaction in boiling toluene to produce electron deficient 1,3-dienes **6a-c** in fairly good yields.

The ^{1}H NMR spectra of stable ylides **4a-c** exhibited a singlet at about δ=2.3 for the CH_3-CO group, a double doublet ($^3J_{HP}$ 17.6 Hz, $^3J_{HH}$ 10.4 Hz) at about δ=3.3 for $CHCO_2R$, alonge with a characteristic doublet ($^3J_{HH}$ 10.4 Hz) at about δ=5.2 for $CH(CO)_2$ moiety. The ^{13}C NMR spectra of **4a-c** displayed a doublet ($^1J_{PC}$ 124 Hz) at about δ=41 for the P=C group and a doublet ($^3J_{PC}$ 12.8-14.6 Hz) at about δ=42 for the $CHCO_2R$ moieties. The ^{31}P NMR spectrum of ylides **4a-c** exhibited, in each case, only one signal at about δ=25 (downfield from 85% H_3PO_4). These shifts are similar to those observed for alkyl triphenylphosphonium iodide [8]. Although compound **4** possesses two stereogenic centers, and two diastereoisomers are expected, only one diastereoisomer is isolated from the reaction mixture.

The ^{1}H NMR spectra of the cyclobutene derivatives **5a-c** displayed signals at about δ=3.5-3.6 and δ=3.8-3.9 for the two methine groups, which appear as doublets ($^3J_{HH}$ 1 Hz), in agreement with the *trans* geometry

for these protons [9]. The ^{13}C NMR spectra of **5a-c** exhibited two signals at about δ=44-47 and δ=50-51 for the two CH groups, and two signals at about δ=130-133 and δ=157-159 for two olefinic carbon atoms. The mass spectra displayed molecular ion peak at *m/z* 303, 331 and 387 for **5a**, **5b** and **5c**, respectively. Initial fragmentations involved loss from or complete loss of the side chains and scission of the cyclobutene ring system.

The ^1H NMR spectra of the butadiene derivatives **6a-c** exhibited two signals at about δ=6.1 and δ=6.2 for the two olefinic protons. The ^{13}C NMR spectra of **6a-c** displayed four signals in the olefinic region. Although, we have not proved the stereochemistry of dienes **6a-c**, the geometry shown in Scheme 1 is the most reasonable on steric ground and on the basis of conrotatory cyclobutene opening.

The structure assignments made on the basis of the NMR spectra for compounds **4-6** were supported by measurements of their IR spectra. Of special interest are the strong carbonyl absorption bands at 1645-1712 cm^{-1} for these compounds and a fairly broad NH peak at about 3300-3420 cm^{-1} for the NH group.

In conclusion, we have found that the reaction of acetoacetanilide with dialkyl acetylenedicarboxylates in the presence of triphenylphosphine leads to a facile synthesis of highly functionalized cyclobutenes, which are converted to electron-deficient 1,3-dienes.

EXPERIMENTAL SECTION

Dialkyl acetylenedicarboxylates, triphenylphosphine and acetoacetanilide were obtained from Fluka (Buchs, Switzerland) and were used without further purification. Melting points were measured on an Electrothermal 9100 apparatus and are uncorrected. Elemental analyses were performed using a Heraeus CHN-O-Rapid analyzer. ^1H , ^{13}C and ^{31}P NMR spectra were measured with a BRUKER DRX-500 AVANCE spectrometer at 500, 125.8 and 202.5 MHz, respectively. Mass spectra were recorded on a Finnigan-Matt 8430 mass spectrometer operating at an ionization potential of 70 eV. IR spectra were recorded on a Shimadzu IR-470 spectrometer.

Preparation of dimethyl 2-(acetoacetanilide-2-yl)-3-(triphenylphosphoranylidene)-butanedioate (4a). General procedure

To a magnetically stirred solution of acetoacetanilide (0.35 g, 2 mmol) and triphenylphosphine (0.52 g, 2 mmol) in CH_2Cl_2 (10 ml) was added, dropwise, a mixture of dimethyl acetylenedicarboxylate (0.28 g, 2 mmol) in CH_2Cl_2 (4 ml) at -10 °C over 10 min. The mixture was allowed to stand at 5 °C for 24 hr. The solvent was removed under reduced pressure and the residue was purified by silica gel (Merck silica gel, 230-400 mesh) column chromatography using hexane:ethyl acetate (1:5) as eluent. The solvent was removed under reduced pressure and ylide **4a** (1.05 g, m.p. 131- 133 °C, yield 90%) was obtained as a white powder. IR (KBr) (ν_{max}, cm^{-1}): 3400 (NH), 1713, 1669 (C=O); ^1H NMR (500 MHz, $CDCl_3$): δ_H 2.3 (3 H, s, CH_3), 3.1 and 3.6 (6 H,2 s, 2 OMe), 3.3 (1 H dd, $^3J_{HP}$ 17.4 Hz and $^3J_{HH}$ 10.4 Hz, CH), 5.2 (1 H, d, $^3J_{HH}$ 10.4 Hz, CH), 7.2-7.6 [20 H, m,

$P(C_6H_5)_3$ and NC_6H_5], 9.1 (1 H, br. s, NH); ^{13}C NMR (125.8 MHz, $CDCl_3$): δ_C 29.3 (CH_3), 41.2 (d, $^1J_{PC}$ 123.4 Hz, P=C), 41.8 (d, $^2J_{PC}$ 13.7 Hz, P=C-*C*H), 49.0 and 51.6 (2 OCH_3), 62.0 [d, $^3J_{PC}$ 5.3 Hz, *C*H$(CO)_2$], 119.3, 123.8, 129.1 and 138.7 (C_{ortho}, C_{para}, C_{meta} and C_{ipso} of NC_6H_5, respectively), 126.2 (d, $^1J_{PC}$ 90.6 Hz, C_{ipso}), 128.4 (d, $^3J_{PC}$ 12.2 Hz, C_{meta}), 131.9 (C_{para}), 133.8 (d, $^2J_{PC}$ 9.1 Hz, C_{ortho}), 167.4 (C=O amide), 170.9 (d, $^2J_{PC}$ 12.8 Hz, C=O ester), 174.6 (d, $^3J_{PC}$ 3.1 Hz, C=O ester), 202.4 (C=O ketone). ^{31}P NMR (202.5 MHz, $CDCl_3$): δ_P 25.6; MS, *m/z* (%): 581 (1), 262 (100), 183 (82), 108 (43); Anal. Calcd. for $C_{34}H_{32}NO_6P$ (581.6) C, 70.21; H, 5.55; N, 2.41 %. Found: C, 70.0; H, 5.7; N, 2.7%.

Diethyl 2-(acetoacetanilide-2-yl)-3-(triphenylphosphoranilylidene)-butanedioate (4b)

White powder, mp 190-192 °C, yield 92%; IR (KBr) (ν_{max}, cm^{-1}): 3400 (NH), 1712, 1664 (C=O); ^1H NMR (90 MHz, $CDCl_3$): δ_H 0.4 and 1.2 (6 H, 2 t, $^3J_{HH}$=7.5 Hz, 2 CH_3), 2.3 (3 H, s, CH_3), 3.3 (1 H dd, $^3J_{HP}$=17.6 Hz and $^3J_{HH}$=10.4 Hz, CH), 3.7 and 4.1 (4 H, 2 ABX_3 system, 2 OCH_2), 5.2 (1 H, d, $^3J_{HH}$ 10.8 Hz, CH), 7-8 [20 H, m, $P(C_6H_5)_3$ and NC_6H_5], 9 (1 H, br. s, NH); ^{13}C NMR (125.8.4 MHz, $CDCl_3$): δ_C 13.6 and 13.8 (2 CH_3), 29.0 (CH_3), 40.5 (d, $^1J_{PC}$ 123.4 Hz, P=C), 41.8 (d, $^2J_{PC}$ 13.7 Hz, P=C-*C*H), 57.3 and 60.3 (2 OCH_2), 61.9 [d, $^3J_{PC}$=6.6 Hz, *C*H$(CO)_2$], 119.1, 124.2, 128.8 and 138.6 (C_{ortho}, C_{para}, C_{meta} and C_{ipso} of NC_6H_5, respectively), 121.3 (d, $^1J_{PC}$ 99.7 Hz, C_{pso}), 128.0 (d, $^3J_{PC}$ 12.8 Hz, C_{meta}), 131.7 (C_{para}), 133.7 (d, $^2J_{PC}$ 10.1 Hz, C_{ortho}), 167.3 (C=O amide), 170.3 (d, $^2J_{PC}$ 13.7 Hz, C=O ester), 174.0 (C=O ester), 202.1 (C=O, ketone). ^{31}P NMR (202.5 MHz, $CDCl_3$):δ_P 25.5; MS, *m/z* (%): M^+, 609 (1), 536 (6), 262 (100), 183 (57), 93 (39); Anal. Calcd. for $C_{36}H_{36}NO_6P$ (609.6) C, 70.92; H, 5.95; N, 2.30 %. Found: C, 70.7; H, 5.8; N, 2.1 %.

Di*-tert-*butyl 2-(acetoacetanilide-2-yl)-3-(triphenylphosphoranilylidene)-butanedioate (4c)

white powder m.p. 154-155 °C, yield 87%; IR (KBr) (ν_{max}, cm^{-1}): 3410 (NH), 1710, 1672 (C=O); ^1H NMR (500 MHz, $CDCl_3$): δ_H 0.9 and 1.4 (18 H, 2 s, 2 CMe_3), 2.2 (3 H, s, CH_3), 3.2 (1 H, dd, $^3J_{HP}$ 18 Hz and $^3J_{HH}$ 10.7 Hz, CH), 5.1 (1 H, d, $^3J_{HH}$ 10.7 Hz, CH), 7.2-7.8 [20 H, m, $P(C_6H_5)_3$ and NC_6H_5], 9 (1 H, br. s, NH); ^{13}C NMR (125.8 MHz, $CDCl_3$): δ_C 28.0 and 28.4 (2 *C*Me_3), 29.2 (CH_3), 40.3 (d, $^1J_{PC}$ 124.3 Hz, P=C), 43.0 (d, $^2J_{PC}$ 14.6 Hz, P=C-*C*H), 62.4 [d, $^3J_{PC}$ 5.5 Hz, *C*H$(CO)_2$], 77.3 and 80.1 (2 O*C*Me_3), 119.1, 124.9, 128.9 and 138.9 (C_{ortho}, C_{para}, C_{meta} and C_{ipso} Of NC_6H_5, respectively), 121.3 (d, $^1J_{PC}$ 100.6 Hz, C_{ipso}), 128.1 (d, $^3J_{PC}$ 11.9 Hz, C_{meta}), 131.7 (C_{para}), 134.1 (d, $^2J_{PC}$ 9.1 Hz, C_{ortho}), 167.8 (C=O amide), 170.5 (d, $^2J_{PC}$ 12.8 Hz, C=O ester), 173.4 (C=O ester), 202.3 (C=O, ketone). ^{31}P NMR (202.5 Mhz, $CDCl_3$): δ_P 5.2; MS, *m/z* (%): M^++1, 662 (2), 564 (8), 508 (7), 262 (88), 93 (98), 57 (100); Anal. Calcd. for $C_{40}H_{44}NO_6P$ (665.7) C, 72.16; H, 6.66; N, 2.10 %. Found: C, 71.9; H, 6.7; N, 2.2 %.

Preparation of dimethyl 3-(anilinocarbonyl)-2-methyl-1-cyclobutene-1,4-dicarboxylate (5a)

Compound **4a** was refluxed in benzene for 3 hr. The solvent was removed under reduced pressure and the residue was purified by silica gel (Merck silica gel, 230-400 mesh) column chromatography using hexane:ethyl acetate (8:1) as eluent. The solvent was removed under reduced pressure and **5a** was obtained as white powder,

m.p. 88-89 °C, yield 55%; IR (KBr) (ν_{max}, cm^{-1}): 3400 (NH), 1702 and 1660 (C=O); ^1H NMR (500 MHz, $CDCl_3$): δ_H 2.2 (3 H, s, CH_3), 3.75 and 3.75 (6 H, s, 2 OMe), 3.6 and 3.9 (2 H, s, 2 CH), 7-7.6 (5 H, m, C_6H_5), 8.2 (1 H, br. s, NH); ^{13}C NMR (125.8 MHz, $CDCl_3$): δ_C 15.3 (CH_3), 45.2 and 51.4 (2 CH), 51.1 and 52.4 (2 OCH_3), 120.2, 124.6, 129.1 and 137.6 (C_{ortho}, C_{para}, C_{meta} and C_{ipso} of NC_6H_5, respectively), 130.8 (C=*C*-CO_2Me), 158.9 (C=*C*-CH_3), 162.0 (C=O amide), 167.1 and 172.1 (C=O ester); MS, *m/z* (%): M^+,303 (29), 212 (20), 184 (37), 93 (100), 65 (57); Anal. Calcd. for $C_{16}H_{17}NO_5$ (303.3) C, 63.36; H, 5.65; N, 4.62 %. Found: C, 63.5; H, 5.4; N, 4.5 %.

Diethyl 3-(anilinocarbonyl)-2-methyl-1-cyclobutene-1,4-dicarboxylate (5b)

White powder, mp 115-117°C, yield 53%; IR (KBr) (ν_{max}, cm^{-1}): 3230 (NH), 1715 and 1666 (C=O); ^1H NMR (500 MHz, $CDCl_3$): δ_H 1.1 and 1.3 (6 H, 2 t, $^3J_{HH}$ 6.6 Hz, 2 CH_3), 1.7 (3 H, s, CH_3), 3.5 and 3.8 (2 H, s, 2 CH), 4.1 (4 H, 2 ABX_3 system, 2 OCH_2), 6.8-7.6 (5 H, m, NC_6H_5), 8.5 (1 H, br. s, NH); ^{13}C NMR (125.8 MHz, $CDCl_3$): δ_C 14.5 and 14.6 (2 CH_3), 15.6 (CH_3), 45.8 and 51.4 (2 CH), 60.8 and 61.7 (2 OCH_2), 120.4, 125.0, 129.3 and 138.1 (C_{ortho}, C_{para}, C_{meta} and C_{ipso} of NC_6H_5, respectively), 131.6 (C=*C*-CO_2Et), 159.2 (C=*C*-CH_3), 162.1 (C=O amide), 167.7 and 172.2 (2 C=O ester); MS, *m/z* (%): M^+,331 (39), 212 (35), 184 (41), 93 (100), 65 (51); Anal. Calcd. for $C_{18}H_{21}NO_5$ (331.4) C, 65.24; H, 6.39; N, 4.23 %. Found: C, 65.1; H, 6.1; N, 4.1 %.

Di*-tert-*butyl 3-(anilinocarbonyl)-2-methyl-1-cyclobutene-1,4-dicarboxylate (5c)

White powder, mp 97-99 °C yield 50%; IR (KBr) (ν_{max}, cm^{-1}): 3420 (NH), 1697 and 1645 (C=O); ^1H NMR (500 MHz, $CDCl_3$): δ_H 1.53 and 1.54 (18 H, 2 s, 2 CMe_3), 2.1 (3 H, s, CH_3), 3.5 and 3.8 (2 H, s, 2 CH), 7-7.8 (5 H, m, C_6H_5), 8.2 (1 H, br. s, NH); ^{13}C NMR (125.8 MHz, $CDCl_3$): δ_C 15.5 (CH_3), 28.5 and 28.7 (2 CMe_3), 47.4 and 51.1 (2 CH), 81.4 and 82.0 (2 O*C*Me_3), 120.3, 124.9, 128.9 and 138.1 (C_{ortho}, C_{para}, C_{meta} and C_{ipso} of NC_6H_5, respectively), 133.2 (C=*C*-CO_2Me), 157.5 (C=*C*-CH_3), 161.4 (C=O amide), 168.1 and 171.3 (C=O ester); MS, *m/z* (%): 331 (35), 212 (31), 184 (39), 93 (100), 65 (41); Anal. Calcd. for $C_{22}H_{29}NO_5$ (387.4) C, 68.19; H, 7.54; N, 3.62 %. Found: C, 68.5; H, 7.7; N, 3.5 %.

Preparation of dimethyl (Z)-2-[(E)-3-anilino-1-methyl-3-oxo-1-propenyl]-2-butenedioate(6a)

Compound **5a** was refluxed in toluene for 24 hr. The solvent was removed under reduced pressure and the residue was purified by silica gel (Merck silica gel, 230-400 mesh) column chromatography using hexane:ethyl acetate (10:1) as eluent. The solvent was removed under reduced pressure and **6a** was obtained as yellow viscous oil, (yield 45%); IR (KBr) (ν_{max}, cm^{-1}): 3315 (NH), 1712 and 1668 (C=O); ^1H NMR (500 MHz, $CDCl_3$): δ_H 2.3 (3 H, s, CH_3), 3.7 and 3.9 (6 H, s, 2 OMe), 6.06 and 6.11 (2 H, s, 2 CH), 7-7.8 (5 H, m, C_6H_5), 8 (1 H, br. s, NH); ^{13}C NMR (125.8 MHz, $CDCl_3$): δ_C 14.3 (CH_3), 52.2 and 53.0 (2 OCH_3), 120.2, 126.8, 143.5 and 150.2 (olefinic carbons), 119.8, 124.5, 128.9 and 137.8 (C_{ortho}, C_{para}, C_{meta} and C_{ipso} of NC_6H_5, respectively), 163.4 (C=O amide), 165.2 and 168.4 (2 C=O ester); MS, *m/z* (%): M^+,303 (29), 244 (31), 212

(25), 183 (53), 93 (100), 65 (53); Anal. Calcd. for $C_{16}H_{17}NO_5$ (303.3) C, 63.36; H, 5.65; N, 4.62 %. Found: C, 63.6; H, 5.8; N, 4.4 %.

Diethyl (Z)-2-{(E)-3-anilino-1-methyl-3-oxo-1-propenyl]-2-butenedioate (6b)

Yellow viscous oil, yield 43%; IR (KBr) (ν_{max}, cm^{-1}): 3315 (NH), 1712 and 1669 (C=O); ^1H NMR (500 MHz, $CDCl_3$): δ_H 1.2 and 1.3 (6 H, 2 t, $^3J_{HH}$ 6.6 Hz, 2 CH_3), 2.3 (3 H, s, CH_3), 4.1 and 4.3 (4 H, m, 2 OCH_2), 6.1 (2 H, s, 2 CH), 7-7.6 (5 H, m, C_6H_5), 7.8 (1 H, br.s , NH); ^{13}C NMR (125.8 MHz, $CDCl_3$): δ_C 13.9 (3 CH_3), 61.2 and 62.1 (2 OCH_2), 119.8, 124.5, 129.0 and 137.8 (C_{ortho}, C_{para}, C_{meta} and C_{ipso} of NC_6H_5, respectively), 120.6, 126.3, 143.5 and 150.1 (olefinic carbons),163.3 (C=O amide), 164.7 and 167.5 (2 C=O ester); MS, *m/z* (%): M^+, 331 (4), 258 (27), 212 (20), 93 (100), 65 (39); Anal. Calcd. for $C_{18}H_{21}NO_5$ (331.4) C, 65.24; H, 6.39; N, 4.23 %. Found: C, 64.6; H, 6.0; N, 4.1 %.

Di-tert-butyl (Z)-2-[(E)-3-anilino-1-methyl-3-oxo-1-propenyl]-2-butenedioate (6c)

Yellow viscous oil, yield 40%; IR (KBr) (ν_{max}, cm^{-1}): 3300 (NH), 1704, 1671 (C=O); ^1H NMR (500 MHz, $CDCl_3$): δ_H 1.4 and 1.6 (18 H, 2 s, 2 CMe_3) , 2.3 (3 H, s, CH_3), 6.05 and 6.1(2 H, s, 2 CH), 7-7.6 (5 H, m, C_6H_5), 7.6 (1 H, br, NH); ^{13}C NMR (125.8 MHz, $CDCl_3$): δ_C 15.6(CH_3), 28.21 and 28.42 (2 CMe_3), 120.29, 125.02, 130.95 and 138.06 (C_{ortho}, C_{para}, C_{meta} and C_{ipso} of NC_6H_5, respectively), 122.42, 125.48, 145.03 and 149.54 (olefinic carbons), 164.05 (C=O amide), 164.35 and 166.97 (2 C=O ester); MS, *m/z* (%): M^+, 387 (10), 212 (6), 184 (12), 93 (100), 57 (100); Anal. Calcd. for $C_{22}H_{29}NO_5$ (387.4) C, 68.19; H, 7.54; N, 3.62 %. Found: C, 68.0; H, 7.5; N, 3.4 %.

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