Tetrahedron Vol. 45, No. 20, pp. 6545 to 6552, 1989
Printed in Great Britain.

0040-4020/89 $3.00+.00

A UNIQUE TRIMOLECULAR MICHAEL REACTION AND CYCLIZATION

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(*Received in UK* 7 *April* 1989)

Abstract: 1,3-Diaryl-2-propene-1-ones react with ethyl cyanoacetate in presence of sodium ethoxide at room temperature to give 2-aroyl-1,3,5-triaryl-4-carbethoxy-4-cyanocyclohexanols. The structure of the reaction products was established by infrared, H-1 and C-13 nuclear magnetic resonance spectral data as well as elemental analysis. X-ray crystallography shows the presence of the cyclohexanol moeity.

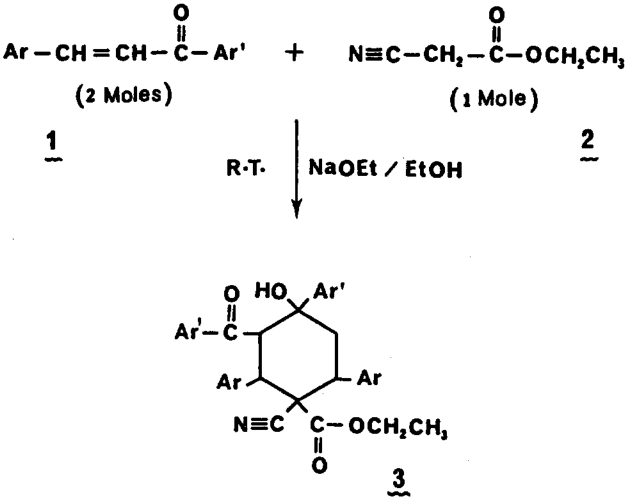
Introduction: It is known that 1,3-diaryl-2-propene-1-ones undergo readily Michael addition with active methylene compounds under catalytic conditions. In some cases and under specific reaction condition the Michael adducts undergo intramolecular condensation to give cyclic products. Thus, it has been reported[1,2] that 1,3-diaryl-2-propene-1-ones react with acetoacetic ester to give ethoxycarbonyl cyclohexenone derivatives presumably through Michael addition followed by condensation of the terminal methyl group with the carbonyl function of the adduct.

It has also been reported[3] that the reaction of ethyl cyanoacetate and different α,β-unsaturated aromatic ketones using piperidine in absolute ethanol gave 2,3,4,5-tetrahydro-8-oxaquinolines. These unexpected products were identified using spin decoupling experiments. For several years[4-12] we have been interested in the condensation reactions of α,β-unsaturated Michael acceptors with active methylene compounds by employing different reaction conditions. In the present work we intended to study the condensation of different 1,3-diaryl-2-propene-1-ones (2 moles) and ethyl cyanoacetate (1 mole) using sodium ethoxide in absolute ethanol at room temperature.

Results and Discussion: Since the Michael reaction is one of the most general methods for C-C bond formation[13] we extend our study to include the reaction of different 1,3-diaryl-2-propene-1-ones **1** and ethyl cyanoacetate **2**. This gives 2-aroyl-1,3,5-triaryl-4-carbethoxy-4-cyanocyclohexanols **3**, as shown in scheme 1. The condensation was carried out by stirring two moles of 1,3-diaryl-2-propene-1-ones with one mole of ethyl cyanoacetate in absolute ethanol in the presence of sodium ethoxide for only 2-3 hours at room temperature.

The structural identification of these cyclohexanol derivatives was based on their spectral data, elemental analysis and X-ray crystallography. The infrared spectra show a peak at 3400-3500 cm^{-1} corresponding to the stretching frequency of the hydroxyl group. A very intense band at around 1650 cm^{-1} due to the benzoyl carbonyl group stretching frequency and a band at around 2220 cm^{-1} characteristic of the cyano group were also observed. The carbethoxy carbonyl group stretching frequency shows a strong peak at around 1735 cm^{-1}.

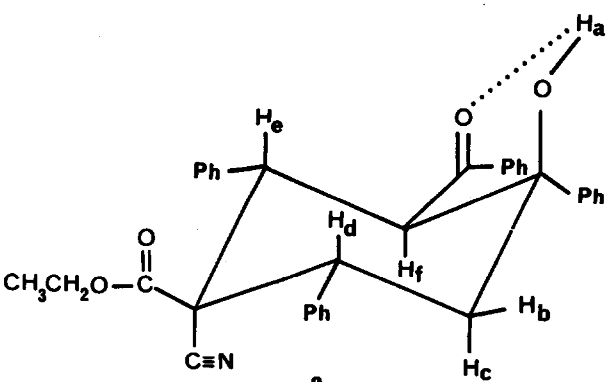
The proton NMR spectra data is also consistent with the proposed structure. The stereochemical configuration of these cyclohexanol derivatives is determined by the analysis of their H-1 NMR spectra. As a representative example, the proton NMR spectrum of 2-benzoyl-4-carbethoxy-4-cyano-1,3,5-triphenylcyclohexanol (3a) shows a doublet of doublet at 2.13 ppm integrating for one proton due to Hb and a doublet of triplet at 3.01 ppm integrating for one proton, Hc. A doublet overlapped with a double doublet at 4.33 ppm integrating for two protons He and Hd were also observed.



Compound	Ar	Ar'
3a	Phenyl	Phenyl
3b	4-Tolyl	4-Bromophenyl
3c	4-Methoxyphenyl	Phenyl
3d	3,4-Dichlorophenyl	4-Bromophenyl
3e	4-Tolyl	3-Bromophenyl
3f	4-Methoxyphenyl	2-Naphthyl
3g	3-Bromophenyl	2-Naphthyl
3h	4-Chlorophenyl	2-Naphthyl

Scheme 1

Hf shows a doublet at 4.93 ppm integrates for one proton. The NMR spectrum also shows another doublet at 5.32 ppm integrates for one proton which was exchangeable with deuterium oxide corresponding to the hydroxyl proton Ha. The ethoxy group shows a triplet and quartet at 0.73 and 3.75 ppm (J = 7.5 Hz), respectively. The coupling constants for these doublets are as follows: J_{bc} = 13.0 Hz; J_{cd} = 13.5 Hz; J_{bd} = 3.6 Hz; J_{ac} = 2.4 Hz; J_{ef} = 12.2 Hz.



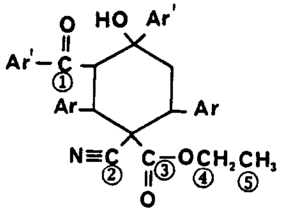
3a

The hydroxyl proton Ha appeared as a doublet (J_{ac} = 2.4 Hz) which may explained as a result of a long range coupling with Hc. The intramolecular hydrogen bonding between Ha and Hc makes the molecule rigid enough with W geometry which makes the long range coupling possible.

The Carbon-13 NMR spectra exhibit a signal at around 203-205 due to the aroyl carbonyl group (C-1). The cyano carbon (C-2) appeared at around 117 ppm. Another signal at around 166 ppm was appeared characteristic of the carbethoxy carbonyl group carbon (C-3), in addition to the remaining aliphatic carbons as shown in (Table I).

Table I

Carbon-13 chemical shift values for 2-aroyl-1,3,5-triaryl-4-carbethoxy-4-cyanocyclohexanols



Compound	C-1	C-2	C-3	C-4	C-5	cyclohexane carbons
3a	205.7	117.7	166.4	59.7	13.3	74.9, 62.1, 52.7, 49.5, 45.4, 42.0
3b	204.0	117.8	166.3	59.9	13.4	74.8, 62.2, 52.6, 49.0, 44.9, 42.0
3c	205.9	116.9	165.2	59.4	13.1	74.5, 62.4, 52.9, 49.4, 44.8, 41.9
3d	205.7	116.7	165.9	59.5	13.6	74.6, 63.2, 52.6, 48.2, 44.4, 41.5
3e	203.9	117.7	166.4	59.8	13.4	74.8, 62.2, 53.4, 48.9, 44.9, 41.9
3f	205.2	118.1	166.7	60.4	13.5	75.4, 62.2, 52.9, 48.9, 44.7, 42.4
3g	204.5	117.3	166.2	59.5	13.6	75.4, 62.8, 52.3, 49.2, 45.1, 42.0
3h	202.8	116.7	165.9	58.9	13.5	75.4, 63.1, 52.6, 48.2, 44.3, 41.5

X-ray analysis of (3a)[14]: The X-ray was carried out on a Nicolet R3 m/V 4-circle X-ray diffractometer. The crystal system was triclinic with space group P$\bar{1}$, a = 10.4835; b = 11.0533; c = 24.1180 A^{o}. α = 94.767; β = 95.853; γ = 98.144. The bond angles (^{o}) are listed in Table II. The X-ray plot given in Figure 1 as reproduced for compound 3a indicated clearly the presence of cyclohexane moeity substituted with the phenyls, cyano, benzoyl, hydroxyl and carbethoxy groups. The bond lengths (A^{o}), the atomic coordinate and equivalent isotropic displacement parameters are listed in Table III and IV.

Table II

Selected bond angles (^{o}) of $C_{35}H_{31}NO_4$ (3a).

C(5)-C(1)-C(2)	109.3(7)	C(6)-C(1)-C(2)	106.1(7)
C(6)-C(1)-C(5)	111.4(7)	C(9)-C(1)-C(2)	110.0(7)
C(9)-C(1)-C(5)	108.1(8)	C(9)-C(1)-C(6)	111.9(8)
C(3)-C(2)-C(1)	108.4(7)	C(21)-C(2)-C(1)	112.3(7)
C(21)-C(2)-C(3)	115.2(7)	C(4)-C(3)-C(2)	109.2(6)
C(31)-C(3)-C(2)	108.7(7)	C(31)-C(3)-C(4)	109.6(7)
C(10)-C(4)-C(3)	109.5(7)	O(3)-C(4)-C(3)	110.4(7)
O(3)-C(4)-C(10)	105.3(6)	C(41)-C(4)-C(3)	113.1(7)
C(41)-C(4)-C(10)	106.0(7)	C(41)-C(4)-O(3)	112.2(8)
C(10)-C(5)-C(1)	109.0(6)	C(51)-C(5)-C(1)	113.1(7)
C(51)-C(5)-C(10)	113.7(7)	O(1)-C(6)-C(1)	121.8(9)
O(2)-C(6)-C(1)	114.7(8)	O(2)-C(6)-O(1)	123.5(8)
O(2)-C(7)-C(8)	116.2(12)	N(1)-C(9)-C(1)	173.0(11)
C(5)-C(10)-C(4)	112.8(7)	C(7)-O(2)-C(6)	115.7(8)
C(22)-C(21)-C(2)	125.9(8)	C(26)-C(21)-C(2)	117.5(9)
C(26)-C(21)-C(22)	116.6(9)	C(23)-C(22)-C(21)	120.8(9)
C(24)-C(23)-C(22)	121.6(11)	C(25)-C(24)-C(23)	119.3(12)
C(26)-C(25)-C(24)	119.5(11)	C(25)-C(26)-C(21)	122.2(10)
O(4)-C(31)-C(3)	119.6(8)	C(32)-C(31)-C(3)	121.6(8)
C(32)-C(31)-O(4)	118.8(8)	C(33)-C(32)-C(31)	122.4(8)
C(37)-C(32)-C(31)	118.7(8)	C(37)-C(32)-C(33)	118.9(8)
C(34)-C(33)-C(32)	120.1(9)	C(35)-C(34)-C(33)	121.2(10)
C(36)-C(35)-C(34)	117.8(10)	C(37)-C(36)-C(35)	122.1(11)
C(36)-C(37)-C(32)	119.9(10)	C(42)-C(41)-C(4)	124.0(11)
C(46)-C(41)-C(4)	118.2(10)	C(46)-C(41)-C(42)	117.7(9)
C(43)-C(42)-C(41)	122.2(12)	C(44)-C(43)-C(42)	119.5(13)
C(45)-C(44)-C(43)	121.3(14)	C(46)-C(45)-C(44)	119.3(16)
C(45)-C(46)-C(41)	119.9(12)	C(52)-C(51)-C(5)	119.5(8)
C(56)-C(51)-C(5)	121.9(7)	C(56)-C(51)-C(52)	118.5(9)
C(53)-C(52)-C(51)	121.1(9)	C(54)-C(53)-C(52)	119.7(9)
C(55)-C(54)-C(53)	120.1(10)	C(56)-C(55)-C(54)	120.2(10)
C(55)-C(56)-C(51)	120.3(8)		

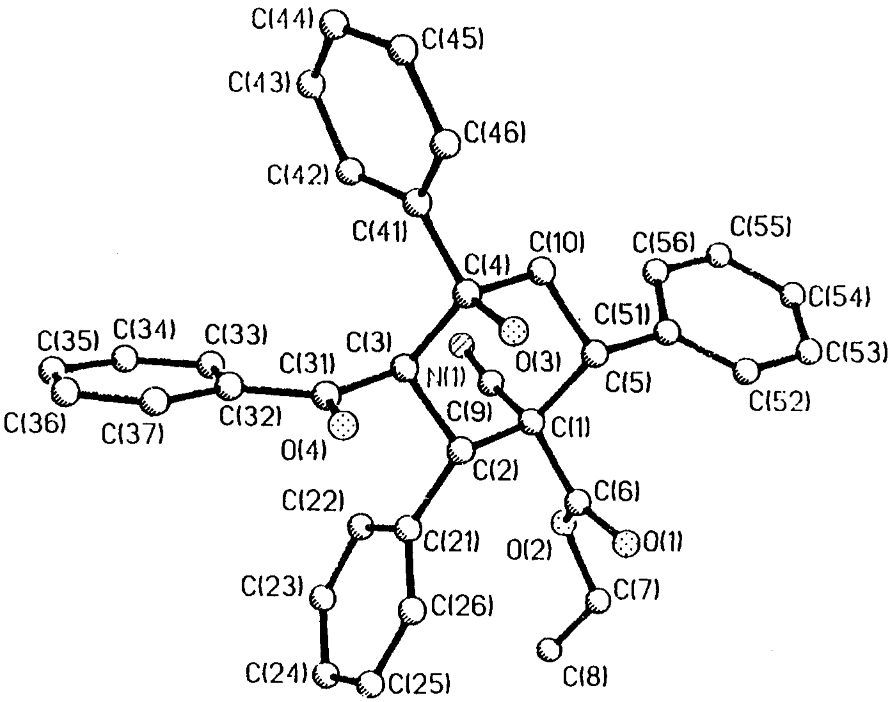


Figure 1: Molecular plot of $C_{35}H_{31}NO_4$ (3a)

Table III

Selected bond lengths (A°) of $C_{35}H_{31}NO_4$ (3a).

C(1)-C(2)	1.553 (12)	C 1)-C(5)	1.534 (13)
C(1)-C(6)	1.484 (12)	C(1)-C(9)	1.424 (12)
C(2)-C(3)	1.518 (11)	C(2)-C(21)	1.486 (13)
C(3)-C(4)	1.539 (13)	C(3)-C(31)	1.499 (12)
C(4)-C(10)	1.508 (11)	C(4)-O(3)	1.412 (10)
C(4)-C(41)	1.487 (12)	C(5)-C(10)	1.515 (10)
C(5)-C(51)	1.489 (12)	C(6)-O(1)	1.188 (11)
C(6)-O(2)	1.302 (12)	C(7)-C(8)	1.219 (23)
C(7)-O(2)	1.440 (14)	C(9)-N(1)	1.138 (12)
O(4)-C(31)	1.192 (11)	C(21)-C(22)	1.356 (14)
C(21)-C(26)	1.358 (14)	C(22)-C(23)	1.347 (16)
C(23)-C(24)	1.331 (17)	C(24)-C(25)	1.332 (19)
C(25)-C(26)	1.360 (17)	C(31)-C(32)	1.470 (12)
C(32)-C(33)	1.334 (13)	C(32)-C(37)	1.344 (14)
C(33)-C(34)	1.354 (13)	C(34)-C(35)	1.337 (16)
C(35)-C(36)	1.326 (18)	C(36)-C(37)	1.341 (15)
C(41)-C(42)	1.337 (16)	C(41)-C(46)	1.361 (17)
C(42)-C(43)	1.360 (16)	C(43)-C(44)	1.299 (25)
C(44)-C(45)	1.344 (25)	C(45)-C(46)	1.376 (17)
C(51)-C(52)	1.345 (11)	C(51)-C(56)	1.354 (13)
C(52)-C(53)	1.367 (14)	C(53)-C(54)	1.328 (16)
C(54)-C(55)	1.349 (14)	C(55)-C(56)	1.358 (15)

Table IV

Atomic coordinates (x10^4) and equivalent isotropic displacement parameters (A^2 x 10^3).

	X	Y	Z	U(eq)
C(1)	4219(9)	11304(8)	3897(3)	36(3)
C(2)	4698(8)	10045(7)	3941(3)	39(4)
C(3)	3536(8)	9038(7)	3807(3)	35(3)
C(4)	2575(9)	9189(7)	4240(4)	39(4)
C(5)	3271(8)	11479(7)	4331(3)	40(4)
C(6)	5396(9)	12247(8)	4008(4)	46(4)
C(7)	6891(14)	13658(11)	3659(5)	118(7)
C(8)	7673(15)	13453(17)	3335(8)	357(18)
C(9)	3551(9)	11334(8)	3355(4)	46(4)
C(10)	2147(8)	10433(7)	4225(3)	42(4)
N(1)	2903(8)	11324(7)	2946(3)	62(4)
O(1)	5999(6)	12459(5)	4459(3)	56(3)
O(2)	5677(6)	12839(6)	3581(3)	65(3)
O(3)	3197(6)	9187(5)	4788(2)	55(3)
O(4)	4468(6)	7553(5)	4270(3)	59(3)
C(21)	5796(9)	9910(7)	3609(4)	39(4)
C(22)	5762(10)	9926(8)	3046(4)	57(4)
C(23)	6839(12)	9860(10)	2792(4)	73(5)
C(24)	7966(11)	9757(10)	3079(6)	85(6)
C(25)	8033(11)	9721(11)	3632(5)	95(6)
C(26)	6952(10)	9791(8)	3892(4)	64(5)
C(31)	3996(9)	7819(8)	3834(4)	45(4)
C(32)	3856(8)	6923(8)	3338(4)	41(4)
C(33)	3451(9)	7192(8)	2826(4)	57(4)
C(34)	3340(10)	6334(9)	2382(4)	71(5)
C(35)	3595(11)	5200(10)	2447(5)	82(6)
C(36)	3988(12)	4948(10)	2959(5)	95(6)
C(37)	4132(10)	5790(9)	3404(4)	74(5)
C(41)	1370(10)	8277(8)	4126(5)	53(4)
C(42)	813(11)	7832(10)	3613(5)	73(5)
C(43)	-345(13)	7075(12)	3525(6)	92(6)
C(44)	-908(15)	6718(12)	3952(9)	115(9)
C(45)	-392(14)	7118(12)	4480(8)	110(8)
C(46)	773(12)	7895(9)	4570(5)	73(5)
C(51)	2865(9)	12717(8)	4360(4)	43(4)
C(52)	3466(9)	13595(8)	4758(4)	52(4)
C(53)	3098(10)	14734(9)	4806(5)	67(5)
C(54)	2139(12)	14986(9)	4451(5)	73(6)
C(55)	1503(11)	14110(10)	4057(5)	76(5)
C(56)	1861(10)	12975(8)	4013(4)	58(4)

Experimental: Unless otherwise stated, IR spectra measured with a Pye-Unicam SP 300 spectrophotometer as KBr pellets. H-1 and C-13 NMR spectra were measured with a Bruker WP 80-SY spectrometer for solutions in deutrated chloroform containing tetramethylsilane as internal standard. Compounds were analyzed at M-H-W Laboratories, Phoenix, Arizona, USA. Melting points were determined with an Electrothermal melting point apparatus and were uncorrected. R_f values were determined on silica gel plates using toluene: ethylacetate (2:1) as an eluent.

General Procedure: In each condensation, ethyl cyanoacetate (0.0172 mol) was dissolved in absolute ethanol (150 ml) containing sodium ethoxide (1.17 g, 0.0172 mol). To this mixture 1,3-diaryl-2-propene-1-ones (0.0342 mol) was added. The mixture was stirred at room temperature for 2-5 hours. The precipitated solid product was filtered off. Recrystallization from glacial acetic acid gave analytical samples of the cyclohexanol derivatives **3**. Partial evaporation of the mother liquor yielded a further amount of the desired product.

2-Benzoyl-4-carbethoxy-4-cyano-1,3,5-triphenylcyclohexanol (3a): Crystallization of the crude product gave a white crystals of 3a, 7.75 g (85%), m.p. 203-204°C. IR spectrum (KBr) ν, cm^{-1}: 3450 (OH), 2240 (C≡N), 1735 (C=O, carbethoxy), 1660 (C=O). NMR spectrum ($CDCl_3$): δ 0.73 (t, 3H, $-CH_3$), 2.13 (dd, 1H, Hb), 3.01 (dt, 1H, Hc), 3.75 (q, 2H, CH_2-), 4.33 (dd, 1H, Hd), 4.33 (d, 1H, He), 4.93 (d, 1H, Hf), 5.32 (d, 1H, Ha), 6.93-7.66 (m, 20H, aromatic). Found (%): C, 79.35; H, 5.75; N, 2.65. Calculated (%): C, 79.30; H, 5.90; N, 2.65. R_f: 0.75.

2-(4-Bromobenzoyl-1-(4-bromophenyl)-4-carbethoxy-4-cyano-3,5-di-(4-tolyl) cyclohexanol (3b): Crystallization of the crude product gave a crystalline solid of 3b, 8.25 g (67%), m.p. 215-216°C. IR spectrum (KBr) ν, cm^{-1}: 3500 (OH), 2250 (C≡N), 1730 (C=O, carbethoxy), 1650 (C=O). NMR spectrum ($CDCl_3$): δ 0.75 (t, 3H, CH_3), 2.10 (s, 3H, Ar-CH_3), 2.25 (dd, 1H, Hb), 2.29 (s, 3H, ArCH_3), 2.84 (dt, 1H, Hc), 3.77 (q, 2H, CH_2-), 4.22 (dd, 1H, Hd), 4.22 (d, 1H, He), 4.78 (d, 1H, Hf), 5.21 (d, 1H, Ha), 6.77-7.51 (m, 16H, aromatic). Found (%): C, 62.14; H, 4.46; N, 1.97; Br, 22.51. Calculated (%): C, 62.11; H, 4.62; N, 1.96; Br, 22.34. R_f: 0.80.

2-Benzoyl-4-carbethoxy-4-cyano-3,5-di-(4-methoxyphenyl)-1-phenylcyclohexanol (3c): Crystallization of the crude product furnished a white crystalline solid of 3c, 6.30 g (62%), m.p. 187-188°C. IR spectrum (KBr) ν, cm^{-1}: 3420 (OH), 2230 (C≡N), 1730 (C=O, carbethoxy), 1640 (C=O). NMR spectrum ($CDCl_3$): δ 0.81 (t, 3H, CH_3), 2.17 (dd, 1H, Hb), 2.88 (dt, 1H, Hc), 3.58 (s, 3H, OCH_3), 3.75 (s, 3H, OCH_3), 3.77 (q, 2H, CH_2-), 4.25 (dd, 1H, Hd), 4.25 (d, 1H, He), 4.86 (d, 1H, Hf), 5.30 (d, 1H, Ha), 6.45-7.64 (m, 18H, aromatic). Found (%): C, 75.58; H, 6.03; N, 2.39. Calculated (%): C, 75.36; H, 5.98; N, 2.38. R_f: 0.63.

2-(4-Bromobenzoyl)-1-(4-bromophenyl)-4-carbethoxy-3,5-di-(3,4-dichlorophenyl)-4-cyanocyclohexanol(3d):Crystallization of the product provided crystalline solid of 3d, 8.50 g (60%), m.p. 232-233°C. IR spectrum (KBr) ν, cm^{-1}: 3460 (OH), 2240 (C≡N), 1730 (C=O, carbethoxy), 1650 (C=O). NMR spectrum ($CDCl_3$): δ 0.86 (t, 3H, CH_3), 2.06 (dd, 1H, Hb), 2.76 (dt, 1H, Hc), 3.83 (q, 2H, CH_2-), 4.25 (dd, 1H, Hd), 4.25 (d, 1H, He), 4.73 (d, 1H, Hf), 4.97 (s, broad, 1H, Ha), 7.11-7.69 (m, 14H, aromatic). Found (%): C, 52.03; H, 3.24; N, 1.67. Calculated(%): C, 52.08, 3.43; N, 1.64. R_f: 0.79.

2-(3-Bromobenzoyl)-1-(3-bromophenyl)-4-carbethoxy-4-cyano-3,5-di-(tolyl) cyclohexanol (3e): Crystallization of the crude product supplied a crystals of 3e, 7.40 g (60%), m.p. 205-207°C. IR spectrum (KBr) ν, cm^{-1}: 3450 (OH), 2240 (C≡N), 1730 (C=O, carbethoxy), 1650 (C=O). NMR spectrum ($CDCl_3$): δ 0.82 (t, 3H, CH_3), 2.12 (s, 3H, ArCH_3), 2.15 (dd, 1H, Hb), 2.29 (s, 3H, ArCH_3), 2.88 (dt, 1H, Hc), 3.77 (q, 2H, CH_2-), 4.23 (dd, 1H, Hd), 4.23 (d, 1H, He), 4.75 (d, 1H, Hf), 5.09 (d, 1H, Ha), 6.79-7.70 (m, 16H, aromatic). Found (%): C, 62.35; H, 4.81; N, 1.97; Br, 22.51. Calculated (%): C, 62.11; H, 4.65; N, 1.96; Br, 22.34. R_f: 0.81.

4-Carbethoxy-4-cyano-3,5-di(4-methoxyphenyl)-2-(2-naphthoyl)-1-(2-naphthyl)cyclohexanol (3f):

Crystallization of the crude product provided an anlytical sample of 3f, 7.50 g (63%), m.p. 143-144°C. IR spectrum (KBr) ν , cm^{-1}: 3430 (OH), 2220 (C≡N), 1720 (C=O, carbethoxy), 1640 (C=O). NMR spectrum ($CDCl_3$): δ 0.79 (t, 3H, $C\underline{H}_3$), 2.26 (dd, 1H, Hb), 3.13 (dt, 1H, Hc), 3.44 (s, 3H, $OC\underline{H}_3$), 3.76 (s, 3H, $\underline{O}CH_3$), 3.85 (q, 2H, $C\underline{H}_2$-), 4.38 (dd, 1H, Hd), 4.38 (d, 1H , He), 5.17 (d, 1H, Hf), 5.55 (d, 1H, Ha), 6.50-8.01 (m, 22H, aromatic). Found (%): C, 78.13; H, 5.70; N, 2.01 Calculated (%): C, 78.35;H , 5.70; N, 2.03. R_f: 0.74.

3,5-Di-(3-bromophenyl)-4-carbethoxy-4-cyano-2-(2-naphthoyl)-1-(2-naphthyl)cyclohexanol (3g):

Crystallization of the crude product furnished a colorless crystals of 3g, 11.6 g (86%), m.p. 242-244°C. IR spectrum (KBr) ν , cm^{-1}: 3450 (OH), 2230 (C≡N), 1725 (C=O, carbethoxy), 1640 (C=O), NMR spectrum ($CDCl_3$): δ 0.85 (t, 3H, $C\underline{H}_3$), 2.25 (dd, 1H, Hb), 3.15 (dt, 1H, Hc), 3.91 (q, 2H, $C\underline{H}_2$-), 4.38 (dd, 1H, Hd), 438 (d, 1H, He), 5.15 (d, 1H, Hf), 5.53 (d, 1H, Ha), 6.80-8.05 (m, 22H, aromatic). Found (%): C, 65.58; H, 4.39; N, 1.79; Br, 20.30. Calculated (%): C, 65.58; H, 4.22; N, 1.78; Br, 20.29. R_f: 0.64.

4-Carbethoxy-3,5-di(4-chlorophenyl)-4-cyano-2-(2-naphthoyl)-1-(2-naphthyl)cyclohexanol (3h):

Crystallization of the crude product afforded an analytical sample of 3h, 9.50 g (79%), m.p. 227-228°C. IR spectrum (KBr) ν , cm^{-1}; 3480 (OH), 2250 (C≡N), 1735 (C=O, carbethoxyl), 1660 (C=O). NMR spectrum ($CDCl_3$): δ 0.84 (t, 3H, $C\underline{H}_3$), 2.14 (dd, 1H, Hb), 2.77 (dt, 1H, Hc), 3.82 (q, 2H, $C\underline{H}_3$-), 4.32 (dd, 1H, Hd), 4.32 (d, 1H, He), 4.63 (d, 1H, Hf), 5.01 (d, 1H, Ha), 7.03-7.71 (m, 22H, aromatic). Found (%): C, 73.98; H, 4.68; N, 1.96; Cl, 9.20. Calculated (%): C, 74.04; H, 4.73; N, 2.00; Cl, 9.18. R_f: 0.67.

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