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ORIGINAL PAPER

Preparation and X-Ray Crystal Structure of (2*Z*,4*E*)-5-(4-substituted phenyl)-3-hydroxy-1-phenylpenta-2,4-dien-1-ones (Curcumin Analogs) from the Condensation–Elimination of Dilithiated 1-Benzoylacetone with Substituted Benzaldehydes

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Abstract 1-Benzoylacetone **1** was dilithiated with lithium diisopropylamide or lithium hexamethyl-disilazide, and the resulting dianion type intermediate **1a** was condensed with (lithium) 4-hydroxybenzaldehyde, 4-dimethylaminobenzaldehyde, or 4-chlorobenzaldehyde to afford hydroxyl-β-diketones that were not isolated but underwent linear dehydration to afford products, (2*Z*,4*E*)-5-(4-hydroxyphenyl)-3-hydroxy-1-phenylpenta-2,4-dien-1-one **2** $C_{17}H_{14}O_3$; (2*Z*,4*E*)-5-(4-(dimethylamino)phenyl)-3-hydroxy-1-phenylpenta-2,4-dien-1-one **3** $C_{19}H_{19}NO_2$; and (2*Z*,4*E*)-5-(4-chlorophenyl)-3-hydroxy-1-phenylpenta-2,4-dien-1-one **4** $C_{17}H_{13}ClO_2$. Crystals of $C_{17}H_{14}O_3$ **2** are orthorhombic, $Pna2_1$, $a = 20.937(4)$ Å, $b = 11.683(3)$ Å, $c = 5.490(1)$ Å, $Z = 4$, $V = 1343.1(5)$ Å^3, $R_1 = 0.0478$ and $wR_2 = 0.1143$ for reflections with $I > 2\sigma(I)$; crystals of $C_{19}H_{19}NO_2$ **3** are monoclinic, $P2_1/c$, $a = 16.637(3)$ Å, $b = 10.736(2)$ Å, $c = 9.197(1)$ Å, $\beta = 105.670(5)°$, $Z = 4$, $V = 1581.7(5)$ Å^3, $R_1 = 0.0609$ and $wR_2 = 0.1491$ for reflections with $I > 2\sigma(I)$; crystals of $C_{17}H_{13}ClO_2$ **4** are monoclinic, $P2_1/c$, $a = 31.574(6)$ Å, $b = 5.780(1)$ Å, $c = 7.423(2)$ Å, $\beta = 94.47(3)°$, $Z = 4$, $V = 1350.5(5)$ Å^3, $R_1 = 0.0696$ and $wR_2 = 0.2423$ for reflections with $I > 2\sigma(I)$.

Keywords Multiple anion syntheses · Curcumin analogs · 1-Benzoylacetone · Benzaldehydes

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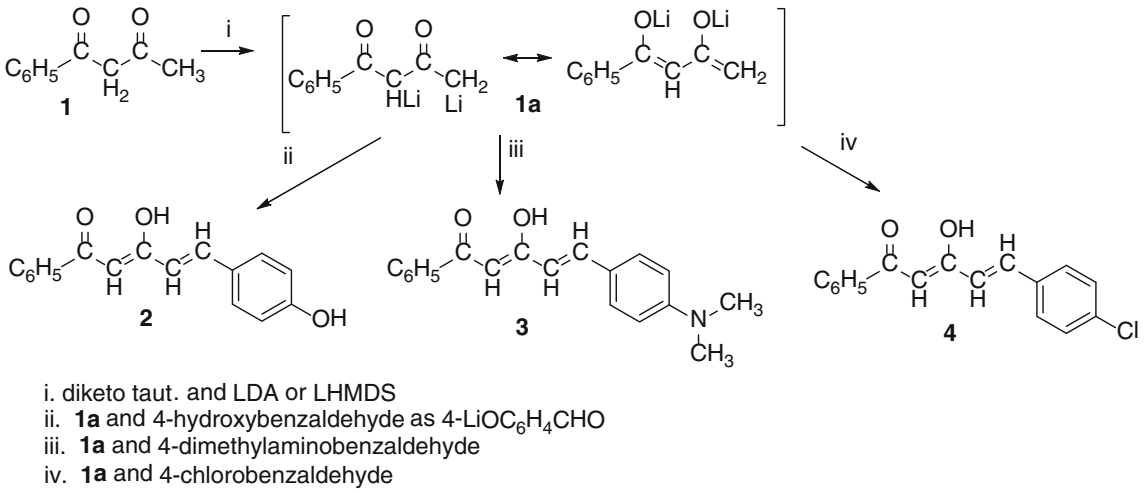
Introduction

Analogs of curcumin are usually associated with alkene β-diketones or their enol tautomers, dienones, (or related conjugated systems [reviews 1–3]). They have been extensively investigated especially with regard to their syntheses [4–12], characterization [13–18], for preparation of metal chelates [19–22], and their potential for medical studies [14, 23]. The value of curcumin analogs has been reported during the past 20 years for their potential as anticancer agents [1]; their structure–activity relationships with different biological activities [2]; as integrase inhibitors anti-HIV agents [3]; for UV/Vis spectral behavior of selected curcumin analogs [15]; for proton NMR spectroscopic investigations of new curcumin analogs [14] and other uses.

A specific group of these analogues can be prepared from the condensation of 1-benzoyl-acetone and aromatic aldehydes. These compounds are candidates for additional syntheses, spectral studies including X-ray crystal analysis [23], and biological testing. There is a historical preparation of some of these compounds reported in 1914 [4]. Nearly five decades later they were prepared by strong base methods including multiple anions of β-diketones, such as 1-benzoylacetone, undergoing aldol type condensations with aromatic aldehydes prepared with alkali amides in liquid ammonia. This would yield hydroxyl β-diketone intermediates, which could be isolated if desired, and they could then undergo linear dehydration to afford the alkene β-diketone system made up of several tautomers [5–8]. There have been several additional different preparative methods developed [9–12] for these compounds.

Recently, we reported the synthesis of symmetrical 1,3,5-pentanetriones by the condensation of acetone with select benzoate esters using lithium hexamethyldisilazide



Scheme 1 Synthesis of curcumin analogs **2**–**4**

(LHMDS) for deprotonation and possible complex formation consisting of a monolithiated acetone, hexamethyldisilazide (HMDS), LHMDS and solvent, tetrahydrofuran [24]. This reaction of acetone, a mono-carbonyl compound, did not proceed by a dianion reaction path [25]. By comparison, a stronger base, lithium diisopropylamide (LDA), would form a dianion type intermediate with β-diketones such as 1-benzoylacetone **1**. The resulting dilithiated 1-benzoylacetone **1a** would selectively react with numerous electrophilic reagents including carbonyl compounds to afford hydroxyl β-diketone intermediates, Scheme 1, that can be isolated and have the potential for linear dehydration to afford alkene β-diketone products, often referred to as curcumin analogs. Initially, it was unclear whether LHMDS would be a strong enough base to be used for similar dilithiation, condensation, and linear dehydration reactions.

Experimental

All reactions, Scheme 1, were conducted in flame-dried glassware under an argon atmosphere. Melting points were determined using a Mel-Temp II melting point apparatus in open capillary tubes and are uncorrected. ^{1}H NMR (300 MHz) and ^{13}C NMR (75 MHz) were conducted with a varian associates 300 MHz NMR spectrometer, and chemical shifts were recorded in δ ppm downfield from an internal TMS standard. IR spectra were acquired with a Brucker Alpha-P FT-IR. LC-MS data were obtained from a Thermo-Finnigan LCQ Advantage system with LCQ Advantage Max mass spectral detector using electrospray ionization and direct injection. Tetrahydrofuran (THF) was freshly distilled from sodium (benzophenone ketyl). All chemicals were obtained from Aldrich Chemical Co. and/or Alpha Aesar.

General Procedure

All reagents except for 1-benzoylacetone **1** were prepared or used in a 5 % molar excess. The procedures employing LDA or LHMDS are interchangeable. The molar ratio for the reactants was 1:2:1 for compounds **3** and **4**; 1:3:1 for **2** (diketone: base: carbonyl compd.). The reaction was conducted in a flask cooled in an ice bath, fitted with an argon inlet tube, a pressure equalizing addition funnel and magnetic stir bar. To the cooled flask was added via syringe ~13.5 mL (0.021 mol) of 1.6 M *n*-butyllithium for preparation of **3** and **4**; ~20 mL (0.0315 mol) of 1.6 M *n*-butyllithium for preparation of **2.** While stirring, 2.12 g (0.0210 mol) of diisopropylamine dissolved in 25 mL of THF was added for preparation of **3** and **4;** 3.18 g (0.0315 mol) for **2;** for LHMDS, 3.39 g (0.0210 mol) or 5.08 g (0.0315 mol) HMDS for making LHMDS. The reaction mixture was stirred for an additional 15 min before rapid drop wise addition of 1.62 g (0.010 mol) 1-benzoylacetone dissolved in 25 mL of THF during 10 min. The mixture was allowed to dilithiate **1** to **1a** for 60 min. The aldehydes (0.0105 mol), dissolved in 25 mL of THF were added drop wise to the stirred solution **1a** during 10 min. To prepare **3** and **4**, the condensation time was 60 min, and for **2** it was 90 min. The solution/mixture was neutralized by rapid addition of ~100 mL of 3 M HCl. Solvent grade ether ~50–100 mL was added before the biphasic mixture was cautiously neutralized with solid sodium bicarbonate to pH ~6 (especially important for product **2**), and the two phases were separated. The aqueous phase was extracted with ether (2 × 35–50 mL). After separation the organic layer was washed with brine, dried with anhydrous calcium chloride, filtered and allowed to evaporate to either a solid or oil that could be readily recrystallized to give products **2**–**4**.

(2*Z*,4*E*)-5-(4-hydroxyphenyl)-3-hydroxy-1-phenylpenta-2,4-dien-1-one (**2**). Compound **2** was obtained in 44 % yield, mp 169–171 °C (ethanol/benzene), (lit. mp 183 °C [19]) from the condensation-dehydration of dianion **(1a)** with lithium 4-hydroxybenzaldehyde. IR: 3242, 1628, and 1157 cm^{-1}. ^{1}H NMR (DMSO-d_6,): δ 6.19 (s, 1H), 6.75 (d, 1H), 7.54–7.70 (m, 9H), 8.03 (d, 1H), and 10.13 (s, OH). ^{13}C NMR (DMSO-d_6,): δ 97.5, 116.7, 120.7 (CH), 126.5, 127.8, 129.6, 131.0, 133.5, 136.1, 141.3, 160.6, 182.0, and 187.8. LCMS–MS, exact mass, 266.09: $(M+H)^+$, 267.00, $(M–H)^-$, 265.09.

Single Crystal X-Ray Structure Determination for **2**

Yellow crystals of $C_{17}H_{14}O_3$ **2** were recrystallized from ethanol in order to give satisfactory crystals for X-ray determination. Crystal data for X-ray studies were collected at 193 K on a Mercury CCD area detector coupled with a Rigaku AFC8 diffractometer with graphite monochromated Mo-Kα radiation. Data were collected in 0.5° oscillations in ω with 45 s exposures. A sweep of data was done using ω oscillations from −70.0° to 90.0° at $\chi = 45°$ and $\phi = 0.0°$; a second sweep was performed using ω oscillations from −60.0° to 80.0° at $\chi = 45°$ and $\phi = 90.0°$. The crystal-to-detector distance was 26.798 mm. Details of the data collection are reported in Table 1. Data were collected, processed, and corrected for Lorentz polarization and for absorption using Crystal Clear (Rigaku) [26, 27].

The non-hydrogen atoms were refined anisotropically. The hydrogen atoms for C1, C2, C4, and for the rings containing C6 and C12 (see numbering of atoms in ORTEP diagram, Fig. 1) were geometrically placed and treated as riding atoms with a C–H bond length of 0.96 Å and $U_{iso}(H) = (1.2)\ U_{eq}$(parent C atom) and similarly for the hydrogen atom on O3 using a bond length of 0.84 Å. The hydrogen atom on O1 was located on a difference electron density map and ideal hydrogen atom coordinates were likewise used for it. Structure solution, refinement, and the calculation of derived results were performed using the *SHELX-97* [28] package of computer programs. Neutral atom scattering factors were those of Cromer and Waber [29], and the real and imaginary anomalous dispersion corrections were those of Cromer [29].

(2*Z*,4*E*)-5-(4-(dimethylamino)phenyl)-3-hydroxy-1-phenylpenta-2,4-dien-1-one (**3**). Compound **3** was obtained in 95 % yield, mp 151 °C (ethanol), (lit. mp 108 °C [19]) from condensation–dehydration of **1a** with 4-dimethylaminobenzaldehyde. IR: 3100, 2900, 1620, and 1233 cm^{-1}. ^{1}H NMR ($CDCl_3$): δ 3.04 (s, 6H), 6.29 (s, 1H), 6.47 (d, 1H, $J = 15$ Hz), 6.69 (d, 2H, $J = 9$ Hz), 7.35–7.60 (m, 5H), 7.67 (d, 1H, $J = 15$ Hz), and 7.94 (d, 2H, $J = 9$ Hz). ^{13}C NMR ($CDCl_3$): δ 40.2, 96.8, 111.9, 118.1, 122.8, 127.2, 128.6, 130.0, 132.1, 136.4, 141.3, 151.7, 181.9, and 187.5. LCMS, exact mass, 293.14: $(M+H)^+$, 294.13.

Single Crystal X-Ray Structure Determination for **3**

Dark red crystals of $C_{19}H_{19}NO_2$ **3** were recrystallized from a methanol–water solution in order to give satisfactory crystals for X-ray determination. Crystal data for X-ray studies were collected at 183 K using the equipment described above for compound **2**. Data were collected in 0.5° oscillations in ω with 20 s exposures. A sweep of data was done using ω oscillations from −70.0° to 90.0° at $\chi = 45°$ and $\phi = 0.0°$; a second sweep was performed using ω oscillations from −70.0° to 90.0° at $\chi = 45°$ and $\phi = 90.0°$. The crystal-to-detector distance was 26.865 mm. Details of the data collection are reported in Table 1. The non-hydrogen and hydrogen atoms were treated as described for compound **2**.

(2*Z*,4*E*)-5-(4-chlorophenyl)-3-hydroxy-1-phenylpenta-2,4-dien-1-one (**4**). Compound **4** was obtained in 44 % yield, mp 164 °C (chloroform/benzene) from the condensation-dehydration of dianion **1a** with 4-chlorobenzaldehyde. IR: 1602, 1273, 1243, 822, and 681 cm^{-1}. ^{1}H NMR ($CDCl_3$): δ 6.34 (s, 1H), 6.61 (d, 1H, $J = 18$ Hz), 7.37 (d, 2H, $J = 9$ Hz), 7.45–7.56 (m, 5H), 7.63 (d, 1H, $J = 18$ Hz), 7.93–7.96 (m, 2H). ^{13}C NMR ($CDCl_3$): δ 98.1, 124.0, 127.5, 128.8, 129.3, 129.3, 132.8, 133.7, 136.0, 136.3, 138.6, 178.8, and 189.9. LCMS, exact mass, 284.06: $(M+H)^+$, 285.00.

Single Crystal X-Ray Structure Determination for **4**

Yellow–green crystals of $C_{17}H_{13}ClO_2$ **4** were recrystallized from benzene/chloroform in order to give satisfactory crystals for X-ray determination. Crystal data for X-ray studies were collected at 193 K using the equipment described above for compound **2**. Data were collected in 0.5° oscillations in ω with 20 s exposures. A sweep of data was done using ω oscillations from −40.0° to 90.0° at $\chi = 45°$ and $\phi = 0.0°$; a second sweep was performed using ω oscillations from −30.0° to 80.0° at $\chi = 45°$ and $\phi = 90.0°$. The crystal-to-detector distance was 26.883 mm. Details of the data collection are reported in Table 1. The non-hydrogen and hydrogen atoms were treated as described for compound **2**.

Results and Discussion

During this investigation β-diketone **1** was deprotonated with LDA or LHMDS to form dilithiated intermediate **1a.** The molecular ratio of the reagents was important: [diketone:base:carbonyl compound], 1:3:1 for **2**; and 1:2:1 for **3** and **4**. Dianion **1a** was separately condensed with (lithium) 4-hydroxybenzaldehyde for **2**, or 4-(dimethylamino)benzaldehyde for **3**, or 4-chloro-benzaldehyde for **4**. Upon

Table 1 Crystallographic data, **2–4**

Compound number	**2**	**3**	**4**
CCDC number[a]	909504	890764	909505
Color/shape	Light yellow/prism	Dark red/flat plate	Light yellow–green/tetragonal plate
Dimensions (mm)	0.65 × 0.26 × 0.24	0.36 × 0.32 × 0.14	0.45 × 0.35 × 0.24
Formula	$C_{17}H_{14}O_3$	$C_{19}H_{19}NO_2$	$C_{17}H_{13}ClO_2$
Formula mass	266.28	293.35	284.72
T (K)	193	183	193
Crystal system	Orthorhombic	Monoclinic	Monoclinic
Space group	$Pna2_1$	$P2_1/c$	$P2_1/c$
a (Å)	20.937(4)	16.637(3)	31.574(6)
b (Å)	11.683(3)	10.736(2)	5.780(1)
c (Å)	5.490(1)	9.197(1)	7.423(2)
β (°)		105.670(5)	94.47(3)
V (Å^3)	1,343.1(5)	1,581.7(5)	1,350.5(5)
Z	4	4	4
d_{calc} (g cm^{-3})	1.317	1.232	1.400
λ (Å)	0.71073	0.71073	0.71073
μ (mm^{-1})	0.090	0.080	0.280
F(000)	560	624	592
θ range (°)	3.40–25.14	2.98–25.14	3.58–25.15
Reflections collected	10,101	14,986	4,435
Miller indices	$-25 \le h \le 25$	$-17 \le h \le 19$	$-37 \le h \le 36$
	$-13 \le k \le 13$	$-12 \le k \le 12$	$-6 \le k \le 6$
	$-6 \le l \le 4$	$-10 \le l \le 10$	$-8 \le l \le 8$
Unique reflections	2148	2817	1972
Unique reflections $I > 2\sigma(I)$	1995	2153	1693
Max transmission	1.000	1.0000	1.000
Min transmission	0.834	0.917	0.610
Data	2148	2817	1972
Restraints	1	0	0
Parameters	183	202	182
Final R indices $I > 2\sigma(I)$	$R_1 = 0.0473$	$R_1 = 0.0609$	$R_1 = 0.0696$
	$wR_2 = 0.1153$	$wR_2 = 0.1491$	$wR_2 = 0.2423$
R indices all data	$R_1 = 0.0525$	$R_1 = 0.0796$	$R_1 = 0.0791$
	$wR_2 = 0.1230$	$wR_2 = 0.1712$	$wR_2 = 0.2589$
Goodness of fit on F^2	1.099	1.035	1.190
Largest diff peak (e Å^{-3})	0.166	0.197	0.315
Largest diff hole (e Å^{-3})	−0.207	−0.221	−0.382

[a] CCDC 909504 for **2**, 890764 for **3**, and 909505 for **4** contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif

neutralization of the respective reaction mixtures with dilute hydrochloric acid, linear dehydration occurred with hydroxyl-β-diketone intermediates not being isolated, so as to afford the tautomeric alkene β-diketone systems **2–4.** The crystalline tautomer isolated is illustrated in Scheme 1.

X-ray single crystal analysis showed the same curcumin, enol bonding structural features (2*Z*,4*E*), and the tautomers were clearly displayed in the ORTEP diagrams [30] for each of the curcumin analogs **2–4**, Figs. 1, 2, and 3. In order to obtain satisfactory crystals for analysis, different solvent or combination systems were used; however, the same tautomer structural type was obtained for each product. The structures of products **2–4** were also supported in part by IR, ^{1}H and ^{13}C NMR, and liquid chromatography mass spectrometry (LCMS). For compounds **2–4** the IR spectra gave information that products had resulted, and displayed dominant absorptions between 1,602 and 1,628 cm^{-1} resulting from aromatic and

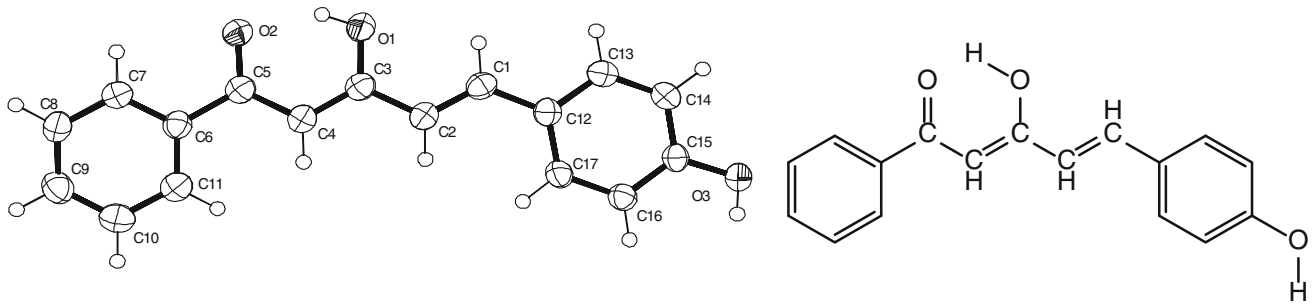


Fig. 1 ORTEP diagram (50 % ellipsoids for non-hydrogen atoms), (2*Z*,4*E*)-5-(4-hydroxyphenyl)-3-hydroxy-1-phenylpenta-2,4-dien-1-one; $C_{17}H_{14}O_3$ **2**, and conventional illustration

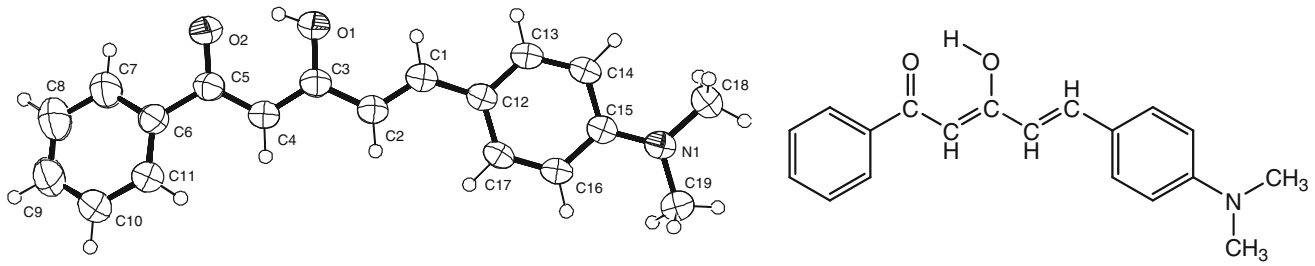


Fig. 2 ORTEP diagram (50 % ellipsoids for non-hydrogen atoms), (2*Z*,4*E*)-5-(4-(dimethylamino)phenyl)-3-hydroxy-1-phenylpenta-2,4-dien-1-one; $C_{19}H_{19}NO_2$ **3**, and conventional illustration

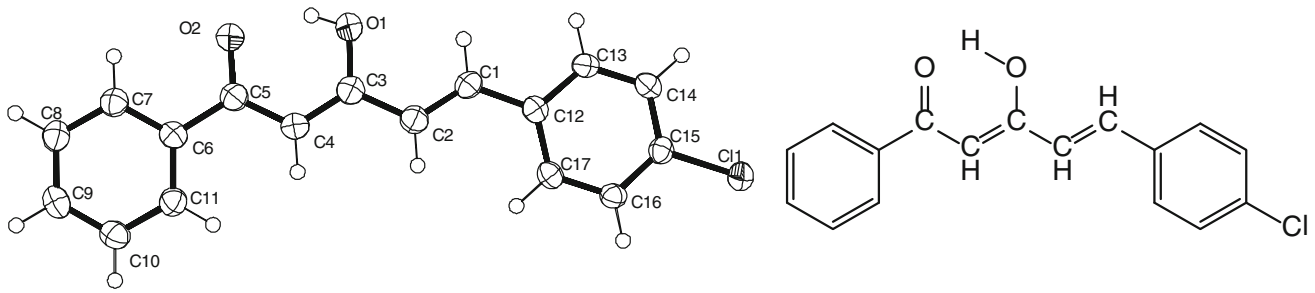


Fig. 3 ORTEP diagram (50 % ellipsoids for non-hydrogen atoms), (2*Z*,4*E*)-5-(chlorophenyl)-3-hydroxy-1-phenylpenta-2,4-dien-1-one; $C_{17}H_{13}ClO_2$ **4**, and conventional illustration

probably conjugated vinyl absorptions; ^{1}H NMR displayed two vinyl protons bonded to C4 and C5 (C1 and C2 ORTEP, respectively), δ 6.19–6.90 ppm; ^{13}C NMR gave consistent absorptions, δ, for C1 to C5 (*Chem. Abstr.* numbering): C1 (C5 ORTEP), 188–190; C2 (C4 ORTEP), 96.8–98.1; C3 (C3 ORTEP), 179–182; C4 (C2 ORTEP), 121–124; and C5 (C1 ORTEP), 139–141 ppm.

The molecular structures of **2**–**4** are shown in Figs. 1, 2 and 3. Selected bond distances and angles for these compounds **2**–**4** are listed in Table 2. The nearly equal C3–C4 and C4–C5 bond lengths and the nearly equal C3–O1 and C5–O2 bond lengths in these compounds indicate that the crystal probably contains some disorder between two tautomers: the first as shown in Figs. 1, 2 and 3 in which there is a single C3–O1 bond, a double C3–C4 bond, a single C4–C5 bond, and a double C5–O2 bond and a second tautomer in which there is a double C3–O1 bond, a single C3–C4 bond, a double C4–C5 bond, and a single C5–O2 bond. The H atom in each tautomer is bonded to either O1 or O2 depending on which O atom is involved in forming the single C–O bond.

The tautomers shown in Figs. 1, 2 and 3 are consistent with the slightly different respective bond lengths. This choice agrees with the results of theoretical molecular modeling calculations [31] at the B3LYP/6-31G(d) level on an isolated molecule of each tautomer that indicate the tautomers shown in the figures are slightly more stable ($\sim$1 kcal mol^{-1}) than the second tautomer. Because there is a rather small activation energy between these two tautomers ($\sim$6 kcal mol^{-1}), both tautomers should be present

Table 2 Selected bond distances (Å) and angles (°), **2–4**

		Bond distances			Bond angles
C1–C12	**2**	1.455(4)	C17–C12–C1	**2**	123.3(3)
	3	1.447(3)		**3**	124.4(2)
	4	1.470(6)		**4**	122.9(4)
C1–C2	**2**	1.338(4)	C12–C1–C2	**2**	126.8(3)
	3	1.347(3)		**3**	128.9(2)
	4	1.337(6)		**4**	127.1(4)
C2–C3	**2**	1.446(4)	C1–C2–C3	**2**	123.0(3)
	3	1.447(3)		**3**	121.6(2)
	4	1.452(6)		**4**	122.2(4)
C3–C4	**2**	1.382(4)	C2–C3–C4	**2**	121.9(3)
	3	1.385(3)		**3**	122.9(2)
	4	1.374(6)		**4**	122.1(4)
C4–C5	**2**	1.415(4)	C3–C4–C5	**2**	121.4(3)
	3	1.406(3)		**3**	120.7(2)
	4	1.414(6)		**4**	121.2(4)
C5–C6	**2**	1.501(4)	C4–C5–C6	**2**	120.9(3)
	3	1.482(3)		**3**	122.2(2)
	4	1.490(6)		**4**	121.9(4)
C3–O1	**2**	1.321(4)	C5–C6–C11	**2**	122.3(3)
	3	1.318(3)		**3**	121.6(2)
	4	1.322(5)		**4**	122.6(4)
C5–O2	**2**	1.272(4)			
	3	1.286(3)			
	4	1.264(6)			
C15–O3	**2**	1.360(4)			
C15–N1	**3**	1.365(3)			
C15–Cl1	**4**	1.741(4)			
N1–C18	**3**	1.448(3)			
N1–C19	**3**	1.455(3)			

in significant amounts. The only evidence for the disorder in the sample not being a 50/50 mixture is the two slightly, but significant, different C–O distances.

All of the molecules are essentially planar. In addition to the intramolecular hydrogen bonding between O1 and O2 in all three compounds, the molecules of **2** form intermolecular hydrogen bonds with two additional molecules. In the first bond, O2 is bonded to the H atom which is bonded to O3 on the second molecule and in the second bond, the H atom on O3 is bonded to O2 on the third molecule.

Conclusions

Select curcumin analogs can also be prepared by the strong base multiple anion procedures, in comparison to different boric oxide methods, but involving LDA or LHMDS for deprotonating a β-diketone, 1-benzoylacetone, to give the dilithiated dianion-type intermediate. This was followed by its condensation with substituted benzaldehydes, to afford hydroxyl β-diketone intermediates that were not isolated, and underwent linear elimination of water to afford the targeted alkene β-diketone, a curcumin analog. X-ray crystal analysis was important in showing that the same tautomer structural features of the easily isolated products **2–4**, (2*Z*,4*E*)-5-(4-substituted phenyl)-3-hydroxy-1-phenylpenta-2,4-dien-1-ones, could be recrystallized from common solvents or mixed solvents. All of the molecules analyzed are essentially planar.

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