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Synthesis of 2-arylhydrazono-4-chloro-3-oxobutanoates existing as (E)/(Z)-hydrazone isomers in solution: Correlation between equilibrium constants and substituent constants



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ABSTRACT

Various 2-arylhydrazono-4-chloro-3-oxobutanoates 10a-i synthesized by the reaction of ethyl 4-chloro-3-oxobutanoate with the 4-substituted benzenediazonium chlorides were found to exist as two isomers between the (E)- and (Z)-hydrazone forms in DMSO- d_6 solution. The equilibrium constants {K = [(Z)form]/[(E)-form]: (8.0–3.4) at 30 °C} were clarified to correlate with the substituent constants of R (σ_n : 0.78 to -0.27) in the aryl group of compounds **10a-i**, wherein the correlation coefficient was 0.99. The preponderance of the (Z)-hydrazone isomers in compounds **10a-i** was assumed to be directed by the 4chloro substituent, while the ratios of less stable (E)-hydrazone isomers gradually increased with decrease in σ_n values and rise in temperature.

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1. Introduction

There have been many papers concerning the (E)/(Z)-hydrazone isomers of the 2-arylhydrazono-3-oxocarboxylates in solution [1] and solid state [2-6]. In our literature survey, the substituent \mathbb{R}^1 of the 2-arylhydrazono-3-oxocarboxylates such as compounds 1-4 [1] (Scheme 1) were supposed to exert an influence on the (E)/(Z)hydrazone isomer ratios. Namely, Table 1 suggests that the (E)hydrazone isomer is predominant in chloroform or acetone when the R^1 is electron donating group (compound **1**: $R^1 = Me$). On the contrary, the (Z)-hydrazone isomers are preponderant in the same solvent as the above when the R¹ is electron withdrawing substituent (compounds **2**–**4**: $\mathbb{R}^1 = \mathbb{CF}_3$, \mathbb{CHF}_2 , $\mathbb{CF}_2\mathbb{CHF}_2$). In the solid state, the (E)-2-arylhydrazono-3-oxobutanoates **5** [2], **6** [3] with the electron donating \mathbb{R}^{1} (Me) and the (Z)-2-arylhydrazono-4-chloro-3oxobutanoate **7** [4] with the electron withdrawing R¹ (CH₂Cl) were also confirmed to exist as the (E)- and (Z)-hydrazone isomers, respectively, by the X-ray crystallography (Fig. 1). However, the 2-(2,6-dimethylphenylhydrazono)-3-oxobutanoate 8 [5] with the

electron withdrawing R¹ (CH₂Cl) exhibited different result from compound 7, which is presumably due to the presence of the 2',6'dimethyl substituents (R²) in the arylhydrazono group. That is, compound **8** was clarified to occur as the (E)-hydrazone isomer in solid state (Fig. 1), suggesting that the substituents in the arylhydrazono group also play an important role for the equilibria between the (E)/(Z)-hydrazone isomers. Compound **9** [6] with the 2-(2,6-dimethylphenylhydrazono) group was also confirmed as the (E)-hydrazone isomer (Fig. 1). The above published information [1-6] exhibits that the balance of the substituents R^1 and R^2 with the electron donating or withdrawing nature seems to control the (E)/(Z)-hydrazone isomer ratios. In the present investigation, we studied the influence of the benzene ring substituent R on the (E)/(Z)-hydrazone isomer ratios for the 2-arylhydrazono-4-chloro-3oxobutanoates 10a-i (Scheme 2) in solution.

2. Results and discussion

2.1. Synthesis and NMR spectral data for 10a-i

The reaction of ethyl 4-chloro-3-oxobutanoate with various 4substituted benzenediazonium chlorides gave the 2-

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Scheme 1. Equilibria of 1–4 in solution.

Table 1	
(E)/(Z)-Hydrazone isomer ratios of 1–4 in solution.	

Compound	R ¹	R ²	R ³	Solvent	Isomer rati	o (%)
					(E)-Form	(Z)-Form
1	Me	Me	Et	CDCl ₃	90	10
				$(CD_3)_2CO$	62	38
2	CF ₃	OMe	Et	CDCl ₃	0	100
				$(CD_3)_2CO$	0	100
3	CHF ₂	Me	Et	CDCl ₃	0	100
				$(CD_3)_2CO$	10	90
4	CF ₂ CHF ₂	OMe	Me	CDCl ₃	0	100
				$(CD_3)_2CO$	0	100



Fig. 1. (E)/(Z)-Hydrazone isomers of 5–9 confirmed by X-ray crystallography.

arylhydrazono-4-chloro-3-oxobutanoates **10a-i** [7], which were also found to exist as the (E)/(Z)-hydrazone isomers in the DMSO- d_6 solution, as described below (Scheme 2).

In the NMR spectral data for the (*Z*)-hydrazone isomers of compounds **10a-i**, the NOE was observed among the ester methylene (δ 4.34 to 4.28), NH (δ 12.66 to 11.99), and phenyl 2',6'-CH (δ 7.64 to 7.40) protons and between the C4-methylene (δ 5.03 to 4.90) and phenyl 2',6'-CH protons (δ 7.64 to 7.40) (Table 2, Scheme 2). On the other hand, only NOE between the NH (δ 14.37 to 13.67) and phenyl 2',6'-CH (δ 7.74 to 7.48) protons was observed in the (*E*)-hydrazone isomers (Table 2, Scheme 2), whereas the NOE was not



Scheme 2. Equilibria of 10a-i between (E)/(Z)-hydrazone isomers in DMSO- d_6 solution.

confirmed between the C4-methylene and NH protons or between the C4-methylene and phenyl 2',6'-CH protons. These results would be due to the strong hydrogen bond between NH proton and keto group in the (*E*)-hydrazone isomer, while the hydrogen bond between the NH and ester carbonyl group is not so tight in the (*Z*)hydrazone isomer [1]. Accordingly, the forementioned NOE spectral data rationalize the assignment for the (*E*)/(*Z*)-hydrazone isomers of **10a-i** in solution [1] (Scheme 2). Based on the above NOE spectral data specifying the (*E*)/(*Z*)-hydrazone isomers, the carbon chemical shifts of compounds **10a-i** were assigned by the gHSQC and gHMBC spectral data (Table 3).

In the above isomerism, there was not the diazenylenol (azoenol) tautomer [8] (Fig. 2) in solution, which was supported by the nitrogen NMR spectral data (Table 4). Namely, the NH protons of compounds **10b** (R = CN) and **10c** (R = Ac) due to the (E)/(Z)hydrazone isomers were confirmed to bind to the sp³-nitrogens by the ¹⁵N-gHMBC spectra [(*E*)-form: N<u>H</u> (13.72), sp³-N (-209.1); (*Z*)form: N<u>H</u>, (11.99), sp³-N (-217.8)] (**10b**) and [(*E*)-form: N<u>H</u> (13.86), sp³-N (-206.9); (*Z*)-form: N<u>H</u>, (12.03), sp³-N (-216.2)] (**10c**), respectively (Table 4), wherein the sp²-nitrogens of compounds **10b,c** were observed in δ (-10.6 to -27.6) [δ (¹⁵N) reference: CH₃NO₂].

2.2. Correlation between equilibrium constants (K) and substituent constants (σ_p)

The equilibrium constants {K = [(Z)-form]/[(E)-form]} were obtained from the NMR spectral data of compounds **10a-i** utilizing the integral intensity of some CH and NH proton signals due to the (E)/(Z)-hydrazone isomers, wherein the K value range was 8.0 to 3.4 at 30 °C (Table 5). Fig. 3 shows a good correlation between the equilibrium constants (K) and the substituent constants (σ_p : 0.78 to -0.27), wherein the correlation coefficient was 0.99.

2.3. Influence of temperature on the equilibrium constants (K)

The NMR spectra of compounds **10c** (R = Ac) and **10h** (R = Me) were measured in DMSO- d_6 with the range of 30–90 °C, wherein the ratios of less stable (*E*)-hydrazone isomers increased with elevation of temperature (Table 6). The change in the equilibrium constants (*K*) was 7.4 to 5.8 in compound **10c** and 4.2 to 3.4 in compound **10h**, respectively. The signals due to the 4-CH₂ and aryl 2',6'-CH protons of the (*E*)/(*Z*)-hydrazone isomers slightly shifted towards higher magnetic fields.

Table 2			
Proton chemical	shifts	for	10a-i.

	Substituent	Isomer	NH	4-CH ₂	Phenyl		Phenyl Ester		Others
					2′, 6′	3′, 5′	CH ₂	Me	
a	NO ₂	E	13.67	5.02	7.74	8.25	4.26	1.18	
		Ζ	12.05	5.03	7.64	8.22	4.34	1.28	
b	CN	Ε	13.72	4.97	7.72	7.85	4.25	1.31	
		Ζ	11.99	5.00	7.62	7.81	4.32	1.28	
с	Ac	Ε	13.86	4.97	7.68	8.00	4.52	1.30	2.53 (Ac)
		Ζ	12.03	5.00	7.58	7.97	4.32	1.28	2.54 (Ac)
d	Cl	Ε	13.97	4.95	7.61	7.47	4.24	1.29	
		Ζ	12.66	4.95	7.53	7.42	4.30	1.27	
e	Br	Ε	13.96	4.95	7.55	7.60	4.23	1.28	
		Ζ	12.04	4.95	7.46	7.54	4.30	1.27	
f	F	Ε	14.11	4.95	7.64	7.27	4.23	1.28	
		Ζ	12.16	4.94	7.56	7.22	4.30	1.27	
g	Н	Ε	14.11	4.94	7.57	7.41	4.22	1.28	7.19 (Phenyl-4')
		Ζ	12.12	4.93	7.50	7.37	4.29	1.26	7.12 (Phenyl-4')
h	Me	Ε	14.23	4.94	7.48	7.23	4.23	1.28	2.28 (4'-Me)
		Ζ	12.19	4.92	7.40	7.19	4.29	1.27	2.27 (4'-Me)
i	OMe	Ε	14.37	4.93	7.55	7.00	4.22	1.28	3.75 (OMe)
		Ζ	12.33	4.90	7.48	6.96	4.28	1.27	3.74 (OMe)

Table 3

Carbon chemical shifts for 10a-i.

	Substituent	Isomer	C1	C2	C3	C4	C1′	C2′,6′	C3′,5′	C4′	Ester		Others
											CH ₂	Me	
a	NO ₂	E	163.6	132.1	189.3	50.5	149.1	116.0	126.2	144.0	61.5	14.5	
		Ζ	161.5	132.2	187.3	47.2	148.1	116.9	126.0	143.1	62.2	14.3	
b	CN	Ε	163.3	125.9	188.7	50.1	145.7	116.8	133.9	106.9	60.9	14.1	118.9 (CN)
		Ζ	161.2	130.5	186.8	46.9	145.8	116.1	133.8	105.5	61.6	13.9	119.0 (CN)
с	Ac	Ε	163.5	129.8	186.6	50.1	145.6	116.1	130.0	133.4	60.8	14.1	196.6 (Acetyl CO), 26.6 (Acetyl Me)
		Ζ	161.4	125.4	186.7	46.8	145.8	115.3	130.0	132.4	61.5	13.9	196.5 (Acetyl CO), 26.3 (Acetyl Me)
d	Cl	Ε	163.7	124.1	188.3	50.0	140.8	118.2	129.5	128.2	60.6	14.2	
		Ζ	161.6	127.9	186.3	46.9	141.0	117.5	129.3	128.2	61.3	13.9	
e	Br	Ε	163.7	124.2	188.3	50.0	141.2	118.5	132.3	117.7	60.6	14.2	
		Ζ	161.6	128.0	186.3	46.9	141.4	117.9	132.1	116.3	61.3	13.9	
f	F	Ε	163.8	123.6	188.1	49.9	138.4	118.4	116.4	159.9	60.5	14.2	
		Ζ	161.7	126.9	186.2	47.0	138.5	117.8	116.1	159.2	61.1	13.9	
g	Н	Ε	163.9	127.0	188.2	49.9	141.7	116.6	129.6	125.8	60.5	14.2	
		Ζ	161.8	127.0	186.3	47.0	141.9	115.9	129.4	124.6	61.1	13.9	
h	Me	Ε	163.9	123.2	188.1	49.8	139.3	116.6	130.0	135.4	60.4	14.2	20.5 (4'-Me)
		Ζ	161.9	129.2	186.0	47.0	139.6	115.9	129.8	134.0	61.0	13.9	20.4 (4'-Me)
i	OMe	Ε	164.1	122.6	187.7	49.8	135.0	118.2	114.8	157.7	60.4	14.2	55.4 (OMe)
		Ζ	162.1	125.3	185.9	47.1	135.4	117.5	114.7	156.8	60.9	14.0	55.4 (OMe)



Fig. 2. Diazenylenol tautomer absent in solution.

3. Conclusion

We found that the 2-arylhydrazono-4-chloro-3-oxobutanoates **10a-i** existed as the (E)/(Z)-hydrazone isomers in DMSO- d_6 solution, wherein the equilibrium constants {K = [(Z)-form]/[(E)-form]} were 8.0 to 3.4 at 30 °C. The predominance of the (Z)-hydrazone isomers would be controlled by the electron withdrawing 4-chloro

Table 4			
NH proton and nitrogen chemical	shifts	for	10b,c

Compound	Chemical shift (δ)						
	NH	sp ³ -N ^a	sp ² -N ^a				
	(E)-Form (Z)-Form	(E)-Form (Z)-Form	(E)-Form (Z)-Form				
10b (R = CN)	13.72	-209.1	-13.4				
10c(R - Ac)	11.99 13.86	-217.8 -206.9	-27.6 -10.6				
for (K=KC)	12.03	-216.2	-25.5				

^a Reference of δ (¹⁵N): CH₃NO₂.

substituent of compounds **10a-i**, as already reported for the 2arylhydrazono-3-oxocarboxylates **2**, **3**, **4**, and **7** having the electron withdrawing substituents R¹ (Table 1, Fig. 1). Moreover, the electron donating substituent R in the 2-arylhydrazono group of compounds **10a-i** (Scheme 2) elevated the ratios of the (*E*)-hydrazone isomers, and the equilibrium constants (*K*) were elucidated to correlate with the substituent constants of R (σ_p : 0.78 to -0.27) in the arylhydrazono group of compounds **10a-i** (correlation

Table 5
Equilibrium constants (K) ^a and substituent constants (σ_p) for 10a-i.

	Substituent	Substituent constant (σ_p)	Isomer ratio (%)		Equilibrium constant (K) ^b
			(E)-Form	(Z)-Form	
a	NO ₂	0.78	11.1	88.9	8.0
b	CN	0.66	11.2	88.8	7.9
с	Ac	0.56	11.9	88.1	7.4
d	Cl	0.23	13.9	86.1	6.2
e	Br	0.23	13.9	86.1	6.2
f	F	0.06	15.9	84.1	5.3
g	Н	0	17.4	82.6	4.8
h	Me	-0.17	19.4	80.6	4.2
i	OMe	-0.27	22.5	77.5	3.4

^a K = [(Z)-Form]/[(E)-Form].

^b At 30 °C.



Fig. 3. Correlation between equilibrium constants (K) and substituent constants ($\sigma p)$ for 10a-i.

coefficient: 0.99). On the other hand, the ratios of the less stable (*E*)-hydrazone isomers were confirmed to increase with elevation of temperature (range: 30-90 °C) in DMSO- d_6 media of compounds **10c** and **10h**.

4. Experimental

4.1. Instrumentation

All melting points were determined on a Yanako micro melting point MP-J3 apparatus and are uncorrected. The IR spectra (potassium bromide) were recorded with a JASCO FT/IR-410 spectrophotometer. The ¹H, ¹³C, and ¹⁵N NMR spectra were measured in DMSO-*d*₆ with an Agilent Technologies DD2 400NB spectrometer at 400, 100, and 40 MHz, respectively. The chemical shifts are given in the δ scale. The mass spectra (MS) were determined with JEOL AX505-HA (for EI MS) and JEOL Accu TOF T100LP (for ESI MS) spectrometers. Elemental analyses were performed on a J-Science LAB JM10 instrument.

4.2. Synthesis and data for 10a-i

4.2.1. General procedure

The diazonium salts were prepared by adding a solution of sodium nitrite (0.55 g, 8.0 mmol) in H₂O (10 mL) to a solution (or suspension) of the appropriate aniline derivatives (8.0 mmol) in AcOH (30 mL)/10% HCl (30 mL) with stirring in an ice water bath for 1 h. A solution of ethyl 4-chloro-3-oxobutanoate (1.0 g, 6.1 mmol) in AcOH (4 mL) was added to the above appropriate diazonium salts with stirring in an ice water bath for 5 h. After H₂O (50 mL) was added dropwise to the reaction mixture with stirring for 1 h, the crystalline products were collected and recrystallized from appropriate solvent (EtOH or EtOH/H₂O) to provide the analytically pure samples.

4.2.2. Data for 10a-i Ethyl 4-chloro-2-(4-nitrophenylhydrazono)-3-oxobutanoate

Table 6	
Influence of temperature on equilibrium constants for 10	c,h.

Compound	Temperature (°C)	Temperature (°C) Isomer ratio (%)		Equilibrium constant (K)	¹ H-Chemical shift (δ)				
					4-CH ₂		2′,6′-H		
		(E)-Form	(Z)-Form		(E)-Form	(Z)-Form	(E)-Form	(Z)-Form	
10c(R = Ac)	30	11.9	88.1	7.4	4.97	5.00	7.68	7.58	
	60	12.5	87.5	7.0	_	_	-	_	
	70	13.5	86.5	6.4	_	_	-	_	
	80	14.3	85.7	6.0	_	_	-	_	
	90	14.7	85.3	5.8	4.89	4.87	7.63	7.56	
10h(R = Me)	30	19.4	80.6	4.2	4.94	4.92	7.48	7.40	
	60	21.2	78.8	3.7	_	_	_	_	
	70	22.2	77.8	3.5	_	_	_	_	
	80	22.5	77.5	3.4	_	_	-	_	
	90	22.6	77.4	3.4	4.86	4.81	7.44	7.38	

(10a): Yield: 95%, reddish brown crystals; IR: 1715, 1666, 1607 cm⁻¹; mp: 133 °C; MS: 313 $[M]^+$, 315 $[M+2]^+$. Anal. Calcd for C₁₂H₁₂ClN₃O₅: C, 45.95; H, 3.86; N, 13.40. Found: C, 45.89; H, 3.92; N, 13.52.

Ethyl 4-chloro-2-(4-cyanophenylhydrazono)-3-oxobutanoate (10b): Yield: 89%, light reddish brown crystals; IR: 2222, 1707, 1671, 1606 cm⁻¹; mp: 127–129 °C, MS: 293 [M]⁺, 295 [M+2]⁺. Anal. Calcd for $C_{13}H_{12}$ ClN₃O₃: C, 53.16; H, 4.12; N, 14.31. Found: C, 53.50; H, 4.15; N, 14.63.

Ethyl 2-(4-acetylphenylhydrazono)-4-chloro-3-oxobutanoate (10c): Yield: 95%, reddish brown crystals; IR: 1698, 1672, 1601 cm⁻¹; mp: 128 °C; MS: 308 $[M]^+$, 310 $[M+2]^+$. Anal. Calcd for C₁₄H₁₅ClN₂O₄: C, 54.11; H, 4.87; N, 9.02. Found: C, 54.10; H, 4.87; N, 9.07.

Ethyl 4-chloro-2-(4-chlorophenylhydrazono)-3-oxobutanoate (10d): Yield: 89%, orange crystals; IR: 1701, 1662, 1634, 1614 cm⁻¹, mp: 88 °C; MS: 325 [M+Na]⁺, 327 [M+2 + Na]⁺. Anal. Calcd for $C_{12}H_{12}Cl_2N_2O_3$: C, 47.54; H, 3.99; N, 9.24. Found: C, 47.62; H, 3.96; N, 9.39.

Ethyl 2-(4-bromophenylhydrazono)-4-chloro-3-oxobutanoate (10e): Yield: 90%, orange crystals; IR: 1700, 1676, 1663 cm⁻¹; mp: 89 °C; MS: 368 $[M+Na]^+$, 370 $[M+2+Na]^+$, 372 $[M+4+Na]^+$. Anal. Calcd for $C_{12}H_{12}BrClN_2O_3$: C, 41.46; H, 3.48; N, 8.06. Found: C, 41.30; H, 3.46; N, 8.27.

Ethyl 4-chloro-2-(4-fluorophenylhydrazono)-3-oxobutanoate (10f): Yield: 86%, yellow crystals; IR: 1694, 1635, 1608, cm⁻¹; mp: 107–108 °C; MS: 286 [M]⁺, 288 [M+2]⁺. Anal. Calcd for $C_{12}H_{12}ClFN_2O_3$: C, 50.27; H, 4.22; N, 9.77. Found: C, 50.29; H, 4.20; N, 9.66.

Ethyl 4-chloro-2-(phenylhydrazono)-3-oxobutanoate (10g): Yield: 71%, yellow crystals; IR: 1699, 1661, 1637 cm⁻¹; mp: 85 °C; MS: 268 $[M]^+$, 270 $[M+2]^+$. Anal. Calcd for $C_{12}H_{13}CIN_2O_3$: C, 53.64; H, 4.88; N, 10.43. Found: C, 53.77; H, 4.84; N, 10.49.

Ethyl 4-chloro-2-(4-methylphenylhydrazono)-3-oxobutanoate (**10h**): Yield: 52%, yellow crystals; IR: 1701, 1663, 1635, cm⁻¹; mp: 95 °C; MS: 305 [M+Na]⁺, 307 [M+2 + Na]⁺. Anal. Calcd for C₁₃H₁₅ClN₂O₃: C, 55.23; H, 5.35; N, 9.91. Found: C, 55.11; H, 5.43; N, 9.89.

Ethyl 4-chloro-2-(4-methoxyphenylhdrazono)-3-oxobutanoate (10i): Yield: 31%, yellow crystals; IR: 1686, 1604 cm⁻¹; mp: 99 °C; MS: 298 [M]⁺, 300 [M+2]⁺. Anal. Calcd for C₁₃H₁₅ClN₂O₄·1/4H₂O: C, 51.49; H, 5.15; N, 9.24. Found: C, 51.47; H, 4.98; N, 9.49.

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