

Chapter 3

Experiments

Instruments.

1. Infrared Spectrophotometers :

- Perkin Elmer model FT-IR 1760 X (The Scientific and Technological Research Equipment Center , Chulalongkorn University).
- Perkin Elmer model 283 (Faculty of Pharmaceutical Sciences, Chulalongkorn University)

2. Nuclear Magnetic Resonance Spectrophotometer :

- Jeol FX 90 Q (90 MHz) (The Scientific and Technological Research Equipment Center, Chulalongkorn University).
- Bruker BZH 200/52 (200 Mhz) (Department of Chemistry Faculty of Sciences, Chulalongkorn University).

3. Mass Spectrometer :

- Jeol FX 3000 double focusing (The Scientific and Technological Research Equipment Center, Chulalongkorn University).

4. Melting Point Apparatus :

- Buchi capillary melting point apparatus, the thermometer is uncorrected.

Chemicals.

Diethyl malonate (Fluka chemie AG).

Triethyl orthoformate (Fluka chemie AG).

Acetic anhydride (BDH Limited Poole England).

Aniline (Merck).

3-Chloro-4-fluoro-aniline (Aldrich Chemicals Co.).

Potassium isothiocyanate (BDH chemicals Ltd.).

Hydrogen peroxide (35% Merck).

Ethyl iodide (Merck).

Ammonia Solution (25%) (Merck).

Tin (Fluka chemie AG).

p-Toluidine (Merck).

All Solvents used were either B.P. or laboratory grade.



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Diethyl ethoxymethylenemalonate (XXXVIII).

A mixture of 18.00 g (0.112 mol) of diethyl malonate (XXXVII), 16.65g (0.112 mol) of triethyl orthoformate, 22.95 g (0.225 mol) of acetic anhydride, and 0.33 g of anhydrous zinc chloride were boiled under reflux for 1 hour. The mixture was then distilled at atmospheric pressure at 200°C. The residue was cooled, filtered and re-distilled under reduced pressure, the fraction was collected at b.p. 140-165°C (10 mmHg), The product was redistilled at 155-160°C (15 mmHg). The yield of product was 12.41 g (51%), b.p. 155-160°C (15 mmHg) (Duffin and Kendall, 1949).

IR (Figure 6.)	2910-2980	cm ⁻¹	(√ C-H)
(KBr Demountable cell)	1730	cm ⁻¹	(√ C=O, ester)
	1635	cm ⁻¹	(√ C=C)
	1250-1290	cm ⁻¹	(√ C-O, ester)
	1085	cm ⁻¹	(√ C-O, ester)
¹ H-NMR (Figure 7.)	1.28-1.45	(m, 9H,	3-CH ₂ -CH ₃)
(CDCl ₃)	4.09-4.38	(m, 6H,	3-CH ₂ -CH ₃)
	7.60	(s, 1H,	C=CH)

Ethyl anilinomethylenemalonate (XXXX a).

A mixture of 4.28 g (46 mmol) of aniline (XXXIX a) and 10.0 g (46 mmol) of diethyl ethoxymethylenemalonate was stirred in methylene chloride at room temperature for 30 minutes. After methylene chloride was evaporated off and the residue was extracted and

crystallized from ether upon cooling ice. The overall yield was 11.73 g (97%), m.p. = 45-50 °c.

IR (Figure 8.)	3350	cm ⁻¹ (√ N-H)
(KBr Demountable cell)	2850-3030	cm ⁻¹ (√ C-H)
	1720	cm ⁻¹ (√ C=O, ester)
	1650	cm ⁻¹ (√ C=C)
	1618	cm ⁻¹ (δ N-H)
	1200-1290	cm ⁻¹ (√ C-O, ester)
¹ H-NMR (Figure 9.)	1.21	(m, 6H, 2-CH ₂ -CH ₃)
(DMSO-d ₆)	4.11	(m, 4H, 2-CH ₂ -CH ₃)
	7.14	(d, 1H)
	7.32	(m, 4H)
	8.37	(d, 1H, J = 13.9 Hz)
	10.69	(d, 1H, J = 13.9 Hz)

Ethyl anilino (-3-chloro-4-fluoro) methylenemalonate (XXXX b).

A mixture of 7.35 g (50.5 mmol) of 3-chloro-4-fluoro-aniline (XXXIX b) and 10.91 g (50.5 mmol) of diethyl ethoxymethylene malonate was refluxed in ethanol for 3 hours. After ethanol was evaporated, the residue was recrystallized from n-hexane to give 11.12 g (70%) of product, m.p. = 55 - 57.5 °c.

IR (Figure 10.)	3250	cm^{-1} (ν N-H)
(KBr demountable cell)	2850-3030	cm^{-1} (ν C-H)
	1720	cm^{-1} (ν C=O, ester)
	1650	cm^{-1} (ν C=C)
	1618	cm^{-1} (δ N-H)
	1200-1290	cm^{-1} (ν C-O)
$^1\text{H-NMR}$ (Figure 11.)	1.21	(m, 6H, 2- CH_2 - CH_3)
(DMSO - d_6)	4.05	(m, 4H, 2- CH_2 - CH_3)
	7.30	(dd, 2H, $J_{\text{HF}} = 7.7$ Hz)
	7.62	(d, 1H, $J_{\text{HF}} = 6.9$ Hz)
	8.18	(d, 1H, $J = 13.8$ Hz)
	10.51	(b, 1H, $J=13.5$ Hz)

3- Carboethoxy-4-hydroxyquinoline (XXXXI a).

One gram (3.8 mmol) of ethyl anilinomethylenemalonate was added to 10 ml diphenyl ether and heated at 260-265°C for 1 hours. After the solution was cooled at room temperature, the resulting precipitate was separated, washed consecutively with benzene, and diethyl ether. The solid was recrystallized from dimethylformamide (DMF). The overall yield was 0.57 g (69%), m.p. 274 - 275°C.

IR (Figure 12.)	2890-3150	cm^{-1} (ν C-H)
(KBr Pellet)	1700	cm^{-1} (ν C=O, ester)

	1620	cm^{-1} (ν C=N)
	1475	cm^{-1} (δ C-H)
	1380	cm^{-1} (δ O-H)
	1198, 1287	cm^{-1} (ν C-O, ester)
$^1\text{H-NMR}$ (Figure 13.)	1.28	(t, 3H, $\text{CH}_2\text{-CH}_3$)
(DMSO- d_6)	4.21	(q, 2H, $\text{CH}_2\text{-CH}_3$)
	7.41	(t, 1H)
	7.59-7.75	(m, 2H)
	8.15	(d, 1H)
	8.56	(s, 1H)
	12.30	(b, 1H, O-H)

3- Carboethoxy-7-chloro-6-fluoro-4-hydroxyquinoline (XXXXI b).

One gram (3.18 mmol) of ethyl anilino (-3-chloro-4-fluoro) methylenemalonate was added to 10 ml of diphenyl ether and heated at 260-265°C for 30 minutes. After the solution cooled, the resulting precipitate was filtered, washed consecutively with benzene and diethyl ether. The solid was recrystallized from DMF to give 0.69 g (81%) of product, m.p. over 300°C.

IR (Figure 14.)	2890-3150	cm^{-1} (ν C-H)
(KBr pellet)	1700	cm^{-1} (ν C=O, ester)
	1610	cm^{-1} (ν C=N)
	1464	cm^{-1} (δ C-H)

	1382	cm^{-1} (δ O-H)
	1180, 1370	cm^{-1} (ν C-O, ester)
$^1\text{H-NMR}$ (Figure 15.)	1.38	(t, 3H, $-\text{CH}_2-\text{CH}_3$)
(DMSO- d_6)	4.44	(q, 2H, $-\text{CH}_2-\text{CH}_3$)
	8.31	(d, 1H, $J_{\text{HF}} = 9.6$ Hz)
	8.49	(d, 1H, $J_{\text{HF}} = 6.4$ Hz)
	9.17	(s, 1H)

3-Carboethoxy-4-chloroquinoline (XXXXII a).

A mixture of 1.5 g (6.9 mmol) of 3-carboethoxy-4-hydroxyquinoline and 3 g (6 ml) of thionyl chloride was heated on a steam-bath for 15 minutes. The excess thionyl chloride was removed *in vacuo* then the crude residue was poured into the mixture of 100 gm of ice and 12 ml of concentrated ammonia solution. The mass was stirred and kept cold until it became entirely granular which was followed by extraction with two 20 ml portion of diethyl ether. After drying over anhydrous sodium sulfate and filtered, diethyl ether was removed and the residue was recrystallized from petroleum ether, the yield was 1.31 g (82%), m.p. 47-48°C.

IR (Figure 16.)	2930-3070	cm^{-1} (ν C-H)
(KBr Pellet)	1734	cm^{-1} (ν C=O, ester)
	1582	cm^{-1} (ν C=C)
	1485	cm^{-1} (δ C-H)
	1172, 1232	cm^{-1} (ν O- CH_2-CH_3)

$^1\text{H-NMR}$ (Figure 17.)	1.43	(t, 3H, $-\text{CH}_2-\underline{\text{CH}}_3$)
(CDCl_3)	4.47	(q, 2H, $-\underline{\text{CH}}_2-\text{CH}_3$)
	7.66	(t, 1H)
	7.80	(t, 1H)
	8.10	(d, 1H, $J = 8.29$ Hz)
	8.36	(d, 1H, $J = 8.32$ Hz)
	9.17	(s, 1H)

3-Carboethoxy-4,7-dichloro-6-fluoro-quinoline (XXXXII b).

A mixture of 1.44 g (5.3 mmol) of 3-carboethoxy-7-chloro-6-fluoro-4-hydroxyquinoline and 5 ml of phosphorous oxychloride was heated at 130-140°C for 1 hour. The excess phosphorous oxychloride was removed *in vacuo* then the crude residue was poured into mixture of 50 ml of ice and 12 ml of concentrated ammonia solution. The mass was stirred and kept cold until it became entirely granular after it was filtered, the filter cake was washed with water and was dried. The solid was dissolved in chloroform and dried over anhydrous sodium sulfate and filtered, and chloroform was removed on a rotary evaporator. The product was recrystallized from chloroform. The overall yield was 1.32 g (87%), m.p. 103-104°C.

IR (Figure 18.)	2934 -3070 cm^{-1}	(ν C-H)
(KBr pellet)	1729	cm^{-1} (ν C=O, ester)
	1581	cm^{-1} (ν C=C)
	1471	cm^{-1} (δ C-H)

	1194,1230	cm^{-1} ($\sqrt{\text{C-O}}$, ester)
$^1\text{H-NMR}$ (Figure 19.)	1.44	(t, 3H, $-\text{CH}_2-\underline{\text{CH}}_3$)
(CDCl_3)	4.47	(q, 2H, $-\underline{\text{CH}}_2-\text{CH}_3$)
	8.14	(d, 1H, $J_{\text{HF}} = 9.6$ Hz)
	8.22	(d, 1H, $J_{\text{HF}} = 6.4$ Hz)
	9.15	(s, 1H)

3-Carboethoxy-4-isothiocyanatoquinoline (XXXXIII a).

A mixture of 500 mg (2 mmol) of 3-Carboethoxy-4-chloroquinoline and 400 mg (4 mmol) of potassium thiocyanate was refluxed in toluene for about 2 days. The mixture was cooled, and the precipitate was removed by filtration. Then the filtrate was evaporated to dryness on a rotary evaporator, the resulting residue was not necessary to be purified. The overall yield was approximate 80-95% (depend on time of reaction, purity of reactant).

IR (Figure 20.)	2080	cm^{-1} ($\sqrt{-\text{N}=\text{C}=\text{S}}$)
(KBr pellet)	1740	cm^{-1} ($\sqrt{\text{C}=\text{O}}$, ester)
	1586	cm^{-1} ($\sqrt{\text{C}=\text{C}}$)
	1490	cm^{-1} (δ C-H)
	1172, 1230	cm^{-1} ($\sqrt{\text{C-O}}$, ester)

3-Carboethoxy-7-chloro-6-fluoro-4-isothiocyanatoquinoline (XXXXIII b).

A mixture of 200 mg (7 mmol) of 3-Carboethoxy-4,7-dichloro-6-fluoro-quinoline and 1.4 g (4.9 mmol) of potassium thiocyanate was refluxed in toluene for about 3 days. The mixture was cooled, and the precipitate was removed by filtration. Then the filtrate was evaporated to dryness on a rotary evaporator, the resulting residue was not necessary to be purified. The overall yield was approximately 80-95%.

IR (Figure 21.)	2930-3070 cm^{-1} (ν C-H)
(KBr pellet)	2100 cm^{-1} (ν -N=C=S)
	1760 cm^{-1} (ν C=O, ester)
	1585 cm^{-1} (ν C=C)
	1480 cm^{-1} (δ C-H)
	1196,1230 cm^{-1} (ν C-O, ester)

2-Thioxopyrimido-[4,3-d]-quinoline-4-(3,6-H)-one (XXXXIV a).

A solution of crude 3-Carboethoxy-4-isothiocyanatoquinoline 0.5 g (1.9 mmol) in 10 ml toluene was kept cool at 0°C in ice-bath. Then a little excess of concentrated ammonia solution (about 0.3 ml) was added slowly to the solution. The mixture which became precipitated within 5 minutes was further stirred for one hour. The yellow precipitate formed was collected by filtration. Purification of the compound was carried out by dissolving the precipitate in alkaline solution (10% sodium hydroxide solution) and filtered off the solid impurity. Then the mixture was reacidified with dilute hydrochloric acid, the

yellow precipitate occurred and collected by filtration, then washed with water and dried. The overall yield was about 0.317 g (73%), mp over 300°C.

IR (Figure 22.)	2980-3260	cm ⁻¹ (√ C-H)
(KBr pellet)	1690	cm ⁻¹ (√ C=O, amide)
	1625	cm ⁻¹ (√ C=N)
	1585	cm ⁻¹ (√ C=C)
	1555	cm ⁻¹ (√ C=C)
	1147	cm ⁻¹ (√ N-C(=S)-N)
¹ H-NMR (Figure 23.)	7.75	(t, 1H)
(DMSO-d ₆)	7.96	(t, 1H)
	8.07	(d, 1H)
	8.96	(d, 1H)
	9.15	(s, 1H)
	12.92	(b, 1H)

Mass spectrum (Figure 24.)

EIMS m/e = 229 M⁺(100), 171(48), 141(17.9), 88(7.4), 76(5.5), 44(21.9).

8-Chloro-9-fluoro-2-thioxopyrimido-[4,3-d]-quinoline-4-(3,6-H)-one (XXXXIV b).

A solution of crude 3-Carboethoxy-7-chloro-6-fluoro-4-isothiocyanatoquinoline 0.5 g (1.6 mmol) in 10 ml dry THF was kept cool at 0°C in ice-bath, then a little excess of concentrated ammonia solution (about 0.3 ml) was added slowly to the solution. The mixture was stirred for one hour. The precipitate was collected by filtration. Purification of

this compound was carried out by dissolved the precipitate in alkaline solution (10% sodium hydroxide solution) and filtered the solid impurity off. Then the mixture was reacidified with dilute hydrochloric acid, the yellow precipitate occurred, and was collected by filtration, then washed with water and dried. The overall yield was about 0.310 g (69%), mp over 300°C.

IR (Figure 25.)	3417	cm ⁻¹	(√ N-H)
(KBr pellet)	2960-3200	cm ⁻¹	(√ C-H)
	1660	cm ⁻¹	(√ C=O, amide)
	1600	cm ⁻¹	(√ C=N)
	1500	cm ⁻¹	(√ C=C)
	1200	cm ⁻¹	(√ C=F)
	1168	cm ⁻¹	(√ N-C(=S)-N)
¹ H-NMR (Figure 26.)	8.3		(d, 1H, J = 7.3 Hz)
(DMSO-d ₆)	9.0		(d, 1H, J = 10.5 Hz)
	9.1		(s, 1H)
	12.9		(b, 1H)

Mass spectrum (Figure 27.)

EIMS m/e = 281 M⁺(100), 223(58.6), 222(42.7), 193(19.6).

3-Phenyl-2-thioxopyrimido-[4,3-d]-quinoline-4-(6H)-one (XXXXV a).

A mixture of crude 3-Carboethoxy-4-isothiocyanatoquinoline 0.50 g (1.9 mmol) and aniline 0.20 g (2.15 mmol) was refluxed in toluene for 3 hours. After the mixture was

cooled, the precipitate was collected by filtration. Then the yellow solid was recrystallized from methanol to give 0.348 g (60%) of product, mp over 300°C.

IR (Figure 28.)	2960-3140	cm ⁻¹ (√ C-H)
(KBr pellet)	1720	cm ⁻¹ (√ C=O, amide)
	1650	cm ⁻¹ (√ C=N)
¹ H-NMR (Figure 29.)	7.41	(d, 2H, o-Ph)
(DMSO-d ₆)	7.48	(t, 1H, p-Ph)
	7.54	(t, 2H, m-Ph)
	7.76	(t, 1H)
	7.97	(t, 1H)
	8.10	(d, 1H)
	8.80	(d, 1H)
	9.17	(s, 1H)
	12.35	(b, 1H)

Mass spectrum (Figure 30.)

EIMS m/e = 305 M⁺(13.4), 304(13.7), 289(65.1), 170(100), 142 (23.3),
115 (30.1), 91 (8.8).

8-Chloro-9-fluoro-3-phenyl-2-thioxopyrimido-[4,3-d]-quinoline-4-(6H)-one (XXXXV b).

A mixture of crude 3-Carboethoxy-7-chloro-6-fluoro-4-isothiocyanatoquinoline 0.5 g (1.6 mmol) and aniline 0.15 g (1.6 mmol) was stirred at room temperature in toluene for 3 hours. The yellow precipitate was collected by filtration and recrystallized from ethanol to give 0.38 g (67%) of product, mp. 278-280°C.

IR (Figure 31.)	1700	cm^{-1}	(\checkmark C=O, amide)
(KBr pellet)	1632	cm^{-1}	(\checkmark C=N)
	1580	cm^{-1}	(\checkmark C=C)
	1550	cm^{-1}	(\checkmark C=C)
	1382	cm^{-1}	(\checkmark C-N)
$^1\text{H-NMR}$ (Figure 32.)	7.28	(d, 2H, o-Ph)	
($\text{CDCl}_3 + \text{DMSO-d}_6$)	7.50	(t, 1H, p-Ph)	
	8.22	(d, 1H)	
	9.07	(d, 1H)	
	9.28	(s, 1H)	
	13.29	(b, 1H)	

Mass spectrum (Figure 34.)

EIMS m/e = 357 M^+ (95.0), 356(100), 222(27.1), 178(9.6),
77(26.6), 44(25.4).

8-Chloro-3-(4-chloro phenyl)-9-fluoro-2-thioxopyrimido-[4,3-d]-quinoline-4-(6H)-one (XXXXVI).

A mixture of crude 3-Carboethoxy-7-chloro-6-fluoro-4-isothiocyanatoquinoline 0.5 g (1.6 mmol) and p-chloro aniline 0.21 g (1.6 mmol) was refluxed in toluene for 2 hours, then stirred at room temperature for 30 minutes. The precipitate was separated off and the filtrate was evaporated to dryness on a rotary evaporator. The residue was purified with a silica gel column, eluted with chloroform or recrystallized from ethanol to give pale yellow crystal 0.32 g (51%) of product, m.p. 278-280°C.

IR (Figure 35.)	2930-3160	cm^{-1} (ν C-H)
(KBr pellet)	1700	cm^{-1} (ν C=O, amide)
	1632	cm^{-1} (ν C=N)
	1580	cm^{-1} (ν C=C)
	1545	cm^{-1} (ν C=C)
	1450	cm^{-1} (ν C-N)
$^1\text{H-NMR}$ (Figure 36.)	7.22	(d, 2H)
(CDCl_3)	7.51	(d, 2H)
	7.86	(d, 1H, $J=8.5$ Hz)
	8.32	(d, 1H, $J=7.0$ Hz)
	9.36	(s, 1H)

Mass spectrum (Figure 37.)

EIMS m/e = 391 M^+ (100), 390(77.3), 265(23.9), 222(50.9) .

8-Chloro-9-fluoro-3-(4-methyl phenyl)-2-thioxopyrimido-[4,3-d]-quinoline-4-(6H)-one (XXXXVII).

A mixture of crude 3-Carboethoxy-7-chloro-6-fluoro-4-isothiocyanatoquinoline 0.5 g (1.6 mmol) and p-toluidin 0.17 g (1.6 mmol) was stirred at room temperature in toluene for 2 hours. The yellow precipitate formed was collected by filtration and was recrystallized from ethanol to give 0.42 g (70%) of product, m. p. 285-287 $^{\circ}$ c.

IR (Figure 38.)	2860-3200	cm^{-1} (ν C-H)
(KBr pellet)	1698	cm^{-1} (ν C=O, amide)

	1630	cm^{-1} ($\sqrt{\text{C}=\text{N}}$)
	1580	cm^{-1} ($\sqrt{\text{C}=\text{C}}$)
	1550	cm^{-1} ($\sqrt{\text{C}=\text{C}}$)
	1445	cm^{-1} ($\sqrt{\text{C}-\text{N}}$)
$^1\text{H-NMR}$ (Figure 39.)	2.39	(s, 3H, CH_3)
(DMSO- d_6)	7.18	(d, 2H)
	7.32	(d, 2H)
	8.37	(d, 1H, $J = 7.6$ Hz)
	9.16	(s, 2H)

Mass spectrum (Figure 40.)

EIMS m/e = 371 M^+ (95.9), 370(100), 338(13.0), 222(13.8), 91(18.0),
44(59.1) .

8-Chloro-3-ethyl-9-fluoro-2-thioxopyrimido-[4,3-d]-quinoline-4-(6H)-one (XXXXVIII).

A suspension of .20 g (0.71 mmol) of 8-Chloro-9-fluoro-2-thioxopyrimido-[4,3-d]-quinoline-4-(3,6-H)-one in 10 ml dry THF was added 4.2 mg of 80% sodium hydride in mineral oil. The mixture was stirred for 30 minutes then added a solution of 0.22 g (1.4 mmol) ethyl iodide in 2 ml THF. The mixture was stirred at room temperature overnight then the precipitate of salt and solid impurity was filtered off and the filtrate was collected and evaporated to dryness on a rotary evaporator. The residue was purified with a silica gel column, eluted with chloroform : methanol (6:4) gave the pale yellow crystal 0.186 g (85%), mp over 300°C.

IR (Figure 41.)	3020-3100	cm^{-1} (ν C-H, aromatic)
(KBr pellet)	2820-2950	cm^{-1} (ν C-H, ethyl)
	2600-2800	cm^{-1} (ν S-H, broad)
	1723	cm^{-1} (ν C=O, amide)
	1595	cm^{-1} (ν C=C)
	1550	cm^{-1} (ν C=C)
	1456	cm^{-1} (δ C-H, ethyl)
	1180	cm^{-1} (ν C-F)
$^1\text{H-NMR}$ (Figure 43.)	1.45	(t, 3H, $\text{CH}_2\text{-CH}_3$)
(DMSO- d_6 , 100°C)	3.40	(q, 2H, $\text{CH}_2\text{-CH}_3$)
	8.29	(d, 1H, $J = 7.1$ Hz)
	8.50	(d, 1H, $J = 9.8$ Hz)
	9.27	(s, 1H)

Mass spectrum (Figure 44.)

EIMS m/e = 309 M^+ (100), 276(57.4), 222(60.1), 193(45.1).

8-Chloro-9-fluoro-3-phenyl-pyrimido-[4,3-d]-quinoline-2,4-(6H)-diones (IL).

To a chilled solution of 8-Chloro-9-fluoro-3-phenyl-2-thioxopyrimido-(4,3-d)-quinoline-4-one = 0.20 g (0.56 mmol) in 2 ml of 10% aqueous sodium hydroxide, 2 ml of 35% hydrogen peroxide was added and the resulting mixture was kept in an ice bath for 2 minutes. After a 15 minutes period of standing at room temperature, another portion of 2 ml 35% Hydrogen peroxide was added. The mixture was allowed to stand for further

30 minutes and then it was diluted with water 20 ml and acidified with concentrated hydrochloric acid to give white crystal 0.172 g (90%) of product, mp over 300°C.

IR (Figure 45.)	2800-3240	cm^{-1}	(ν C-H)
(KBr pellet)	1730	cm^{-1}	(ν C=O, amide)
	1660	cm^{-1}	(ν C=O, amide)
	1570	cm^{-1}	(ν C=C)
$^1\text{H-NMR}$ (Figure 46.)	7.38		(d, 2H, o-Ph)
(DMSO- d_6 , 60°C)	7.45		(t, 1H, p-Ph)
	7.53		(t, 2H, m-Ph)
	8.27		(d, 1H, J = 7.0 Hz)
	8.76		(d, 1H, J = 10.7 Hz)
	9.12		(s, 1H)
	12.21		(b, 1H)

Mass spectrum (Figure 47.)

EIMS m/e = 341 M^+ (65.0), 340(14.7), 249(27.6), 222(100), 19(10.4).

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