CHAPTER III

EXPERIMENTS

INSTRUMENT

1. Infrared Spectrophotometer : Shimazu IR - 440 and Perkin Elmer 1760X (The Scientific and Technological Research Equipment Center, Chulalongkorn University), Perkin Elmer 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), Brucker FT - NMR (80 MHz) (Department of Science Service, Ministry of Science Technology and Energy), Brucker 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.

CHEMICALS

Diethyl malonate (Fluka chemie AG).

Triethyl orthoformate (Fluka chemie AG).

Aniline (Merck).

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

Phenylhydrazine (Fluka chemie AG).

Hydrazine Sulfate (May & Baker LTD.).

Ethyl iodide (Merck).

Piperazine anhydrous (Sigma).

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

Diethyl ethoxymethylenemalonate

A mixture 160 g (1.0 mol) of diethyl malonate, 148 g (1.0 mol) of triethyl orthoformate, 204 g (2.0 mols) of acetic anhydride, and 2 - 3 g of anhydrous zinc chloride were boiled under reflux for 45 minutes. The mixture was then distilled at atmospheric pressure at 190^{o}C. The residue was cooled, filtered and re-distilled

under reduced pressure, the fraction, b.p. 140 - 165°C (10 mmHg), being collected. The produce on redistillation boiled at 155 - 160°C (15 mmHg). The yield of product was 128 g (59%), b.p. 155 - 160°C (15 mmHg).

This compound was prepared under conditions according to the method by Claisen, 1897 (Duffin and Kendall, 1949).

IR (Figure 2) ν	2910 - 2980 cm^{-1} (C-H)
(KBr Demountable cell)	1730 cm^{-1} (C=O, ester)
	1635 cm^{-1} (C=C)
	1085 cm^{-1} (C-O)
	1250 - 1290 cm^{-1} ($O-CH_2CH_3$)
^1H-NMR (Figure 3) δ	1.28 - 1.45 (m, 9H, $3-CH_3$)
($CDCl_3$)	4.09 - 4.38 (m, 6H, $3-O-CH_2$)
	7.60 (s, 1H, C=CH)

Ethyl anilinomethylenemalonate

A mixture 1.86 g (20 mmol) of aniline and 4.3 g (20 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. The overall yield was 5.08 g (97%), m.p. 45 - 50°C.

IR (Figure 4) ν	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} (N-H bending)
	1200 - 1290 cm^{-1} ($O-CH_2CH_3$)
^1H-NMR (Figure 5) δ	1.21 (m, 6H, $2-CH_3$)
($DMSO-d_6$)	4.11 (m, 4H, $2-O-CH_2$)
	7.14 (m, 2H)
	7.32 (d, 3H)
	8.37 (d, 1H, J = 13.9 Hz)
	10.69 (b, 1H, J = 13.9 Hz, N-H)

Ethyl anilino (-3-chloro-4-fluoro) methylenemalonate

A mixture 1.47 g (10 mmol) of 3-chloro-4-fluoro-aniline and 2.16 g (10 mmol) of diethyl ethoxy-methylene malonate was refluxed in ethanol for 3 hrs. After ethanol was evaporated. The residue was

recrystallized from n-hexane gave 2.28 g (72%) of product, m.p. 55 - 57.5°C.

IR (Figure 6) ν	3250 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} (N-H, bending)
	1200 - 1290 cm^{-1} ($O-CH_2CH_3$)
^1H-NMR (Figure 7) δ	1.21 (m, 6H, $2-CH_3$)
($DMSO-d_6$)	4.05 (m, 4H, $2-O-CH_2$)
	7.30 (dd, 2H, J_{H-F} = 7.7 Hz)
	7.62 (d, 1H, J_{H-F} = 6.9 Hz)
	8.18 (d, 1H, J = 13.8 Hz)
	10.51 (b, 1H, J = 13.5 Hz, N-H)

3-Carboethoxy-4-hydroxyquinoline

A 0.43 g (1.6 mmol) of ethylanilino methylenemalonate was added to diphenyl ether 5 ml and heated at 260 - 265°C for 1 hrs. After the solution cooled, the resulting precipitate was filtered, washed consecutively with benzene, and diethyl ether and dried over. The solid was recrystallized from dimethylformamide

(DMF). The overall yield was 0.28 g (73%), m.p. 274-5°C.

IR (Figure 8) ν	2890 - 3150 cm^{-1} (C-H)
(KBr Pellet)	1700 cm^{-1} (C=O, ester)
	1620 cm^{-1} (C=N)
	1475 cm^{-1} (C-H bending)
	1380 cm^{-1} (O-H bending)
	1198, 1287 cm^{-1} ($O-CH_2CH_3$)
^{1}H-NMR (Figure 9) δ	1.28 (t, 3H, CH_3)
(DMSO-d_6)	4.21 (q, 2H, $O-CH_2$)
	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

A 0.40 g (1.2 mmol) of ethyl anilino (3-chloro-4-fluoro) methylenemalonate was added to 5 ml diphenyl ether and heated at 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 0.29 g (80%) of product, m.p. over 300°C.

IR (Figure 10) ν	2890 - 3150 cm^{-1} (C-H)
(KBr pellet)	1700 cm^{-1} (C=O, ester)
	1610 cm^{-1} (C=N)
	1464 cm^{-1} (C-H bending)
	1382 cm^{-1} (O-H bending)
	1180, 1370 cm^{-1} ($O-CH_2CH_3$)
^1H-NMR (Figure 11) δ	1.38 (t, 3H, CH_3)
(DMSO-d_6)	4.44 (q, 2H, $O-CH_2$)
	8.31 (d, 1H, J_{H-F} = 9.6 Hz)
	8.49 (d, 1H, J_{H-F} = 6.4 Hz)
	9.17 (s, 1H, aromatic H)

3-Carboethoxy-4-chloroquinoline

A mixture 0.25 g (1 mmol) of 3-carboethoxy-4-hydroxyquinoline and 0.5 g (1 ml) of thionyl chloride was heated on a steam-bath for 15 minutes. The excess thionyl chloride was removed *in vacuo* then crude was poured into mixture of 25 gm of ice and 5 ml of concentrated ammonia water. The mass was stirred and kept cold until it became entirely granular which was followed by extraction with two 10 ml portions 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 0.23 g (85%), m.p. 47 - 8°C.

IR (Figure 12) ν	2930 - 3070 cm^{-1} (C-H)
(KBr Demountable cell)	1734 cm^{-1} (C=O, ester)
	1582 cm^{-1} (C=C)
	1485 cm^{-1} (C-H bending, CH_2CH_3)
	1172, 1232 cm^{-1} (O-CH_2CH_3)
^{1}H-NMR (Figure 13) δ	1.43 (t, 3H, CH_3)
($CDCl_3$)	4.47 (q, 2H, O-CH_2)
	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

A mixture 1.44 g (5 mmol) of 3-carboethoxy-7-chloro-6-fluoro-4-hydroxyquinoline and 2.5 g (3 ml) of thionyl chloride was heated on a steam-bath for 15 minutes. The excess thionyl chloride was removed *in vacuo* then crude was poured into 50 ml of ice to which had been add 12 ml of concentrated ammonia water. 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 1.47 g (96%), m.p. 103.5 - 104°C.

IR (Figure 14) ν	2934 - 3070 cm^{-1} (C-H)
(KBr pellet)	1729 cm^{-1} (C=O, ester)
	1581 cm^{-1} (C=C)
	1471 cm^{-1} (C-H bending, CH_2CH_3)
	1194 cm^{-1} (C-O) ($O-CH_2CH_3$)
^{1}H-NMR (Figure 15) δ	1.44 (t, 3H, CH_3)
($CDCl_3$)	4.47 (q, 2H, $O-CH_2$)
	8.14 (d, 1H, J_{H-F} = 9.6 Hz)
	8.22 (d, 1H, J_{H-F} = 6.4 Hz)
	9.15 (s, 1H)

2-Arylpyrazolo [4,3-c] quinolin-3-one

A 0.6 g (2 mmol) of 3-carboethoxy-4-chloroquinoline was heated with 0.5 g (4.6 mmol) phenylhydrazine in xylene

at 130°C for 1 hrs. After cooling precipitate was removed by filtration, and the filtrate was extracted with 1 N sodium hydroxide, then the aqueous extract was acidified with 1 N hydrochloric acid and the resulting precipitate was collected by filtration washed with water, and dried. The solid was recrystallized from tetrahydrofuran (THF). This gave yield 0.34 g (65%) of product, m.p. > 300°C.

IR (Figure 16) ν	2880 - 2950 cm^{-1} (C-H)
(KBr pellet)	1627 cm^{-1} (C=O, amide)
	1610 cm^{-1} (C=N)
	1588 cm^{-1} (C=C)
	1300 cm^{-1} (C-N)
^{1}H-NMR (Figure 17) δ	7.17 (t, 1H, aromatic H)
(DMSO-d_6)	7.35 (t, 2H)
	7.42 - 7.60 (m, 3H)
	8.17 (d, 2H)
	8.22 (d, 1H)
	8.32 (d, 1H)
	12.46 (b, 1H, NH)
^{1}H-NMR (Figure 18) δ	7.18 (t, 1H)
(DMSO-d_6 +$CDCl_3$)	7.43 (t, 2H)

7.58 - 7.77 (m, 3H)

8.21 (d, 3H)

8.72 (s, 1H)

12.84 (b, 1H, NH)

^{13}C-NMR (Figure 19)

COSY (Figure 20)

7-Chloro-8-fluoro-2-arylpyrazolo [4,3-c] quinolin-3-one

A 0.26 g (0.85 mmol) of 3-Carboethoxy-4, 7-dichloro-6-fluoroquinoline was heated with 0.18 g (1.6 mmol) of phenylhydrazine in xylene at 130 - 150^{o}C for 3 hrs. After cooling, the resulting precipitate was removed by filtration, washed with water and methanol, chloroform, and dried *in vacuo*. The solid was crystallized from ethanol gave 0.21 g (75%) of product, m.p. > 300^{o}C.

IR (Figure 21) ν (KBr pellet)

2890 - 3000 cm^{-1} (C-H)

1627 cm^{-1} (C=O, amide)

1610 cm^{-1} (C=N)

1588 cm^{-1} (C=C)

1362 cm^{-1} (C-N)

^{1}H-NMR (Figure 22) δ	7.19 (t, 1H)
(DMSO-d_6)	7.45 (t, 2H)
	7.89 (d, 1H, J_{H-F} = 6.5 Hz)
	8.15 (d, 1H, J_{H-F} = 9.3 Hz)
	8.21 (d, 2H, J_{H-H} = 8.9 Hz)
	8.81 (s, 1H)

2H-Pyrazolo [4,3-c] quinolin-3-one

A 0.13 g (1 mmol) of hydrazine sulfate was added to a solution of sodium ethoxide which was prepared by dissolving 0.046 g (2 mmol) of sodium metal in 5 ml absolute ethanol. The mixture was stirred for 15 minutes then precipitate was filtered off gave freshly hydrazine in ethanol solution which it dropwisely added to a solution of 0.24 g (1 mmol) 3-carboethoxy-4-chloroquinoline in ethanol, then the mixture was stirred at room temperature overnight. After the precipitate was filtered and excess alcohol was removed on a rotary evaporator. The residue was purified with a silica gel column, eluted with methanol : ethyl acetate (1:1). The overall yield was about 0.12 g (64%), m.p. 260^{o}C.

IR (Figure 23) ν	2950 - 3030 cm^{-1} (C-H)
(KBr pellet)	1652 cm^{-1} (C=O, amide)
	1588 cm^{-1} (C=C)
	1345 cm^{-1} (C-N)
^{1}H-NMR (Figure 24) δ	7.61 (t, 1H)
(DMSO-d_6)	7.80 - 7.94 (m, 2H)
	8.32 (d, 1H)
	8.90 (s, 1H)
	15.38 (b, 1H, N-H)

7-Chloro-8-fluoro-2H-pyrazolo [4,3-c] quinolin-3-one

A 0.47 g (3.6 mmol) of hydrazine sulfate was added to a solution of sodium ethoxide which was prepared by dissolving 0.17 g (7.2 mmol) of sodium metal in 15 ml absolute ethanol. The mixture was stirred for 15 minutes, then precipitate was filtered off gave freshly hydrazine in ethanol solution which it dropwisely added to a solution of 1.1 g (3.6 mmol) 3-carboethoxy-4-chloroquinoline in ethanol, then the mixture was stirred at room temperature for 8 hours. After the precipitate was filtered and excess alcohol was removed on a rotary

evaporator. The residue was purified with a silica gel column, eluted with methanol : ethyl acetate (1:1) then solid was recrystallized from methenal to afford 0.56 g (62%) as yellow crystal, m.p. > 300.

IR (Figure 25) ν	2950 - 3030 cm^{-1} (C-H)
(KBr pellet)	1627 cm^{-1} (C=O, amide)
	1585 cm^{-1} (C=C)
	1312 cm^{-1} (C-N)

^{1}H-NMR (Figure 26) δ	7.87 (d, 1H, J_{H-F} = 6.9 Hz)
(DMSO-d_6)	7.95 (d, 1H, J_{H-F} = 9.6 Hz)
	8.61 (s, 1H)

5-Ethyl-2-arylpyrazolo [4,3-c] quinolin-3-one

A suspension of 0.38 g (1.45 mmol) of 2-arylpyrazolo [4,3-c] quinolin-3-one in THF (5 ml) was added 0.05 g of 60% sodium hydride in mineral oil. The mixture was stirred for 30 minutes then added a solution of 1.1 g (7 mmol) ethyl iodide in THF (2 ml). The mixture was stirred at room temperature overnight after precipitate was filtered and excess THF was evaporated to dryness on a rotary evaporator. The precipitating

crystals were washed with hexane and dried. Recrystallization from methanol gave 0.17 g (41%) of product as yellow crystal, m.p. > 300°C.

IR (Figure 27) ν (KBr pellet)

2974 - 3034 cm^{-1} (C-H)

1640 cm^{-1} (C=O, amide)

1604 cm^{-1} (C=N)

1490 cm^{-1} (C-H bending, CH_2CH_3)

^{1}H-NMR (Figure 28) δ (DMSO-d_6)

1.43 (t, 3H, CH_3)

4.51 (q, 2H, N-CH_2)

7.17 (t, 1H)

7.45 (t, 2H)

7.61 (t, 1H)

7.76 (t, 1H)

7.94 (d, 1H, J = 8.5 Hz)

8.20 (dd, 2H)

8.30 (d, 1H, J = 7.9 Hz)

8.90 (s, 1H)

^{13}C-NMR (Figure 29)

Mass spectrum (Figure 30)

EIMS m/e = 289 M^+ (100), 260 (24.42), 156 (12.02), 145 (3.80), 127 (4.01), 105 (2.42), 102 (2.93), 77 (17.78)

7-Chloro-5-ethyl-8-fluoro-2-arylpyrazolo [4,3-c] quinolin-3-one

A suspension of 1.31 g (4.3 mmol) of 7-chloro-8-fluoro-2-arylpyrazolo [4,3-C] quinolin-3-one in dry THF (20 ml) was added 0.17 g of 60% sodium hydride in mineral oil. The mixture was stirred for 30 minutes, then added a solution of 1.34 g (8.6 mmol) ethyl iodide in THF (5 ml). The mixture was stirred at room temperature overnight after precipitate was removed by filtration and excess THF evaporated to dryness on a rotary evaporator. The collective precipitating crystals were washed with water and methanol, and dried. Recrystallized from acetone to afford 1.20 g (82%) of product as yellow crystal, m.p. 287°C.

IR (Figure 31) ν	2910 - 3005 cm^{-1} (C-H)
(KBr pellet)	1657 cm^{-1} (C=O, amide)
	1594 cm^{-1} (C=N)
	1466 cm^{-1} (C-H bending, CH_2CH_3)
	1309 cm^{-1} (C-N)
^{1}H-NMR (Figure 32) δ	1.40 (t, 3H, CH_3)
(DMSO-d_6)	4.51 (q, 2H, N-CH_2)
	7.20 (t, 1H)

7.46 (t, 2H)

8.18 (d, 1H, J_{H-H} = 8.9 Hz)

8.23 (d, 1H, J_{H-F} = 9.6 Hz)

8.27 (d, 1H, J_{H-F} = 6.4 Hz)

8.91 (s, 1H)

Mass spectrum (Figure 33)

EIMS m/e = 341 M^+(100), 312 (21.52),
306 (1.58), 222 (1.94),
221 (5.25), 208 (9.10),
105 (5.36), 77 (28.86)

7-Chloro-5-ethyl-8-fluoro-2H-pyrazolo [4,3-c] quinolin-3-one

A suspension of 1.07 g (4.2 mmol) of 7-chloro-8-fluoro-2H-pyrazolo [4,3-c] quinolin-3-one in dry THF (20 ml) was added 0.17 g of 60% sodium hydride in mineral oil. The mixture was stirred for 30 minutes, then added a solution of 1.34 g (8.6 mmol) ethyl iodide in THF (5 ml). The mixture was stirred at room temperature for 3 hrs after precipitate was filtered and excess THF evaporated to dryness on a rotary evaporator. The resultant collective precipitate were purified with a silica gel

column, eluted with methanol : ethyl acetate (1:1) then solid was recrystallized from methanol. The overall yield about 0.64 g (56%) as yellow crystal, m.p. 291°C.

IR (Figure 34) ν	3068 - 3138 cm^{-1} (C-H, aromatic)
(KBr pellet)	2860 - 2955 cm^{-1} (C-H, CH_2CH_3)
	1628 cm^{-1} (C=O, amide)
	1523 cm^{-1} (C=C)
	1465 cm^{-1} (C-H bending, CH_2CH_3)
	1379 cm^{-1} (C-N)
^{1}H-NMR (Figure 35) δ	1.43 (t, 3H, CH_3)
(DMSO-d_6)	4.50 (q, 2H, N-CH_2)
	8.06 (d, 1H, J_{H-F} = 11.2)
	8.17 (d, 1H, J_{H-F} = 6.4)
	8.70 (s, 1H)
	11.54 (b, 1H, N-H)

Mass Spectrum (Figure 36)

EIMS m/e = 265 M^+ (100), 230 (4.61), 209 (55.03), 208 (38.35), 180 (43.48), 154 (69.3), 45 (5.78), 44 (20.54)

7-Chloro-1-ethyl-8-fluoro-2H-pyrazolo [4,3-c] quinolin -3-one

The reaction was carried out according to Method A. The precipitated was filtered and excess THF was evaporated. Purified was acheived on a silica gel coloumn with methanol : ethylacetate (1:1). After evaporation of the first fraction and recrystallized from ethanol, 7-chloro-1-ethyl-8-fluoro-2H-pyrazolo [4,3-c] quinolin -3-one was obtained (see Table 3).

IR (Figure 43)

^{1}H-NMR (Figure 44)

Mass spectrum (figure 45)

7-Chloro-3-ethoxy-1-ethyl-8-fluoro-2H-pyrazolo [4,3-c] quinoline (Method B)

A suspension of 2 g (7.90 mmol) of 7-chloro-8-fluoro-2H-pyrazolo [4,3-c] quinolin-3-one in ethanol containing 1.2 equiv of protassium carbonate (1.31 g) was stirred. Refluxing was continued for 30 minutes and 2.46 g (15.8 mmol) ethyl iodide was added under reflux for 2 hrs. After the precipitate was filtered and then evaporated to dryness. The crude residue was crystallized from methanol. Resulting the first crystal, 7-chloro -3-

ethoxy-1-ethyl-8-fluoro-2H-pyrazolo [4,3-c] quinoline was filtered and recrystallized from hexane (see Table 3).

IR (Figure 46)

^{1}H-NMR (Figure 47)

Mass spectrum (figure 48)

7-Chloro-2, 5-diethyl-8-fluoro-pyrazolo [4,3-c] quinolin - 3-one

From the above filtrate, the second crystal was precipitated. The crystalline product was filtered off and recrystallized from ethyl acetate (see Table 3).

IR (Figure 49)

^{1}H-NMR (Figure 50)

Mass spectrum (figure 51)

7-Chloro-5-ethyl-8-(1-piperazinyl)-2-arylpyrazolo [4,3-c] quinolin 3-one

A mixture of 0.25 g (0.7 mmol) 7-chloro-5-ethyl-8-fluoro-2-arylpyrazolo [4,3-c] quinolin-3-one and 0.13 g (1.5 mmol) of piperazine was heated reflux in pyridine at 110^{o}C for 100 hrs. After, the mixture was evaporated to dryness on a rotary evaporator. The purification was performed by column chromatography, mobile phase used was methanol : THF (4:1) and stationary phase was silica gel. The overall yield was about 0.14 g (47 %) as yellow solid, m.p. 242^{o}C (dec.).

IR (Figure 37) √	3300 cm^{-1} (N-H)
(KBr demountable cell)	2820 - 3010 cm^{-1} (C-H)
	1630 cm^{-1} (C=O, amide)
	1590 cm^{-1} (C=C)
	1469 cm^{-1} (C-H bending, CH_2CH_3)
	1312 cm^{-1} (C-N)
^{1}H-NMR (Figure 38) δ	1.47 (t, 3H, CH_3)
(DMSO-d_6)	3.10 (m, 8H, piperazine CH_2)
	4.50 (q, 2H, N-CH_2)
	7.42 (t, 1H)
	7.59 (d, 2H)

7.90 (s, 1H)

8.08 (s, 1H)

8.23 (d, 1H)

8.31 (d, 1H)

8.85 (s, 1H)

Mass Spectrum (Figure 39)

EIMS m/e = 407 M^+ (38.10), 371 (8.11),
322 (35.2), 321 (100),
77 (16.58)

7-Chloro-5-ethyl-8-(1-piperazinyl)-2H-pyrazolo [4,3-c] quinolin-3-one

A mixture of 0.35 g (1.3 mmol) 7-chloro-5-ethyl-8-fluoro-2H-pyrazolo [4,3-c] quinolin-3-one and 0.23 g (2.7 mmol) of piperazine was refluxed in pyridine at 110^{o}C for 72 hrs. After, the mixture was evaporated to dryness on a rotary evaporator. The resulting residue was purified with a silica gel column, eluted with 30% chloroform in methanol. The overall yield was about 0.19 g (43%) as yellow solid, m.p. 254^{o}C (dec.).

IR (Figure 39) ν	3406 cm^{-1} (N-H)
(KBr demountable cell)	2930 - 3010 cm^{-1} (C-H)
	1620 cm^{-1} (C=O, amide)
	1519 cm^{-1} (C=C)
	1469 cm^{-1} (C-H bending, CH_2CH_3)
^{1}H-NMR (Figure 41) δ	1.30 (t, 3H, CH_3)
(DMSO-d_6)	3.07 (m, 8H, piperazine CH_2)
	4.42 (q, 2H, N-CH_2)
	7.68 (s, 1H)
	7.95 (s, 1H)
	8.62 (s, 1H)
	11.49 (s, 1H, N-H)

Mass Spectrum (Figure 42)

EIMS m/e = 331 M^+ (10.39), 295 (2.61),
275 (3.33), 85 (3.84),
56 (11.28), 36 (100)