CHAPTER FOUR

EXPERIMENTAL
PART A

The starting ethyl [N-4-(un)substituted anilino] glycinates [1-6] were prepared according to literature methods available.1,2,3

SECTION I :

General procedure for the preparation of ethyl [(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino] acetates[7-12].

Preparation of ethyl [(N-benzenesulphonyl ) anilino] acetate4 [7].

To a solution of ethyl N-[phenyl] glycinate (10.74 g, 0.06 mole) in dry benzene (30 ml), pyridine (4.74 g, 0.09 mole) was added with stirring. To the above cold solution benzenesulphonyl chloride (10.56 g, 0.06 mole) was added in small portions with constant stirring and the stirring was continued for 48 hours. The reaction mixture was diluted with water and extracted with ether. The ethereal layer was washed with 10% hydrochloric acid, followed by 5% potassium hydroxide, water and a saturated solution of sodium chloride. The ether layer was then dried over anhydrous sodium sulphate and the solvent ether was removed to get the sulphonylated ester [7].

Above procedure was adapted to prepare remaining ethyl [(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino] acetates [8-12].
Alternate method for the preparation of ethyl \((N\text{-benzenesulphonyl})\) anilino] acetate [7].

To ethyl-N-(phenyl) glycinate, (10.74 g., 0.06 mole) dissolved in minimum quantity of acetone, sodium bicarbonate (4.20 g, 0.05 mole) in water was added with stirring and cooling in ice salt mixture. To the above reaction mixture benzenesulphonyl chloride (10.56 g, 0.06 mole) in minimum amount of acetone was added dropwise, over a period of 30 minutes. The entire reaction mixture was further stirred for a period of 12 hours.

After the reaction was complete, acetone was removed under reduced pressure and the product was poured on crushed ice. The sulphonylated product was extracted with ether (4 x 50 ml). The combined ether layer was washed with water, dried over anhydrous sodium sulphate and the sulphonylated ester [7] was obtained by evaporating ether.

General procedure for the preparation of hydrazides of \((N\text{-benzenesulphonyl/tosyl})-4\text{-}(un)substituted anilino\) acetic acids [13-18].

Preparation of \((N\text{-benzenesulphonyl})\) anilino] acetic acid hydrazide [13].

\((N\text{-benzenesulphonyl anilino}]\) acetic acid ester [7], (19.21 g, 0.1 mole) was dissolved in ethyl alcohol (40 ml). To this solution hydrazine hydrate 99% (5 g, 0.1 mole) was added, dropwise with cooling. The
resulting reaction mixture was refluxed for 6-8 hours. Concentration in vacuum left a residue, which was poured onto crushed ice. The solid separated was filtered, washed with water, dried and crystallised from ethyl alcohol.

Similarly [(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino] acetic acid hydrazides [14-18] were obtained and are enumerated in (Table I).

**General procedure for the preparation of potassium salt of [(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino] acetyl dithiocarbazinates [19-24].**

**Preparation of potassium salt of [(N-benzenesulphonyl) anilino] acetyl dithiocarbazinate [19].**

To a well stirred clear solution of potassium hydroxide (0.78 g, 0.015 mole) in absolute ethanol (5 ml), was added [(N-benzenesulphonyl) anilino] acetic acid hydrazide [13], (3.05 g, 0.01 mole) with stirring and cooling in ice. To this, carbon disulphide (1.17 g, 0.015 mole) was added in small portions. The reaction mixture was stirred continuously for 12 hours and diluted with anhydrous ether (200 ml). The potassium dithiocarbazinate that separated was filtered, washed several times with anhydrous ether and dried in vacuum. The potassium salt obtained in quantitative yield, was moisture sensitive and hence used directly for the preparation of the corresponding triazole without further purification.
Various potassium salts of [(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino] acetyl dithiocarbazinates [20-24] were prepared by the same procedure, as adapted for the above compound.

**General procedure for the preparation of**

3-[(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino]methyl-4-amino-5-mercapto-4(H)-1,2,4-triazoles [25-30].

**Preparation of** 3-[(N-benzenesulphonyl) anilino] methyl-4-amino-5-mercapto-4(H)-1,2,4-triazole [25].

Potassium salt of [(N-benzenesulphonyl) anilino] acetyl dithiocarbazinate [19], (4.19 g, 0.01 mole) was dissolved in water (10 ml). To this clear solution was added hydrazine hydrate 99% (1.0 g, 0.02 mole) and the reaction mixture was refluxed for 6-8 hours. The colour of the mixture turned to green with the evolution of hydrogen sulphide gas (lead acetate paper test and odour). The clear solution was treated with decolorising charcoal, filtered, cooled in ice and then carefully acidified with acetic acid. The precipitated solid was filtered, washed with water, dried and crystallised from ethanol.

According to the same procedure the remaining 3-[(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino]methyl-4-amino-5-mercapto-4(H)-1,2,4-triazoles [26-30] were prepared and are listed in (Table II).
General procedure for the preparation of 3-[(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino]methyl-4-anilino-5-mercapto-4(H)-1,2,4-triazoles [31-36].

Preparation of 3-[(N-benzenesulphonyl) anilino] methyl-4-anilino-5-mercapto-4(H)-1,2,4-triazole[31].

Potassium salt of [(N-benzenesulphonyl)anilino]acetyl dithiocarbazinate [19], (4.19 g, 0.01 mole) was dissolved in minimum amount of water. To this clear solution was added phenyl hydrazine (1 08 g, 0.01 mole) and the reaction mixture was refluxed in an oil bath at 140-160 °C for 5-6 hours or till the evolution of hydrogen sulphide gas (lead acetate paper test and odour) ceased. The clear solution was treated with decolorising charcoal, filtered, cooled in ice and then carefully acidified with acetic acid. The precipitated solid was filtered, washed with water, dried and crystallised from ethanol.

According to the same procedure the remaining 3-[(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino]methyl-4-anilino-5-mercapto-4(H)-1,2,4-triazoles [32-36] were prepared and are listed in (Table III).
SECTION II:

Preparation of derivatives of 3-[(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino]methyl-4-amino-5-mercapto-4(H)-1,2,4-triazoles.

i) Reaction of 3-[(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino]methyl-4-amino-5-mercapto-4(H)-1,2,4-triazoles with aliphatic/aromatic acids.

General procedure for the preparation of 3-[(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino]methyl-6-[H/CH₃/C₆H₅/CH₂C₆H₅/C₆H₄NO₂(p)]-s-triazolo(3,4-b)(1,3,4) thiadiazoles [37-66].

Preparation of 3-[(N-benzenesulphonyl)anilino]methyl-s-triazolo (3,4-b)(1,3,4) thiadiazole [37].

A mixture of 3-[(N-benzenesulphonyl)anilino]methyl-4-amino-5-mercapto-4(H)-1,2,4-triazoles [25], (1.44g, 0.004 mole), formic acid 100% (0.5 ml) and thionyl chloride (10 ml) were heated together on steam bath for 5-6 hours. The reaction mixture was cooled and poured onto crushed ice, the pH of the solution was brought to 7 by the addition of liquor ammonia. The precpitated solid was filtered, washed thoroughly with cold water, dried and crystallised from alcohol.

All the new 3-[(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino]methyl-6-[H/CH₃/C₆H₅/CH₂C₆H₅/C₆H₄NO₂(p)]-s-triazolo(3,4-b)(1,3,4) thiadiazoles [38-66] were obtained according to the procedure.
described for compound [37]. These compounds are described in (Table IV).

ii) Reaction of 3-[(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino]methyl-4-amino-5-mercapto-4(H)-1,2,4-triazoles with oxalic acid.

**General procedure for the preparation of 6-6-bis{-3-[(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino]methyl-s-triazolo (3,4-b)(1,3,4) thiadiazoles} [67-72].**

**Preparation of 6-6-bis{-3-[(N-benzenesulphonyl) anilino]methyl-s-triazolo(3,4-b)(1,3,4) thiadiazole} [67].**

3-[(N-benzenesulphonyl)anilino]methyl-4-amino-5-mercapto-4(H)-1,2,4-triazole [25] (1.44 g, 0.004 mole), oxalic acid (0.180 g, 0.002 mole) and thionyl chloride (10 ml) were heated on a steam bath for 5-6 hours. The reaction mixture was cooled, poured over crushed ice and neutralised with liquor ammonia. The precipitated solid was filtered, washed with cold water, dried and crystallised from ethanol.

Various 6-6-bis{-3-[(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino]methyl-s-triazolo (3,4-b)(1,3,4) thiadiazoles} [68-72] thus obtained are enumerated in (Table V).
iii) Reaction of 3-[(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino]methyl-4-amino-5-mercapto-4(H)-1,2,4-triazoles with cyanogen bromide:

General procedure for the preparation of 3-[(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino]methyl-6-amino-s-triazolo (3,4-b) (1,3,4) thiadiazoles [73-78].

Preparation of 3-[(N-benzenesulphonyl) anilino] methyl-6-amino-s-triazolo (3,4-b) (1,3,4) thiadiazole [73].

3-[(N-benzenesulphonyl)anilino]-4-amino-5-mercapto-4(H)-1,2,4-triazole [25], (1.44g, 0.004 mole) and cyanogen bromide (0.424g, 0.004 mole) were dissolved in absolute ethanol. The reaction mixture was refluxed for 8-10 hours on a steam bath, concentrated and cooled. The solid separated was filtered, washed with cold water, dried and crystallised from ethyl alcohol.

All the new 3-[(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino]methyl-6-amino-s-triazolo (3,4-b) (1,3,4) thiadiazoles [74-78] prepared by adapting the procedure described for the compound [73] are summerised in (Table VI).

iv) Reaction of 3-[(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino]methyl-4-amino-5-mercapto-4(H)-1,2,4-triazoles with carbon disulphide:
General procedure for the preparation of 3-[(N-benzene-
sulphonyl/tosyl)-4-(un)substituted anilino]methyl-s-triazolo
(3,4-b) (1,3,4) thiadiazole-6-(5H)-thiones [79-84].

Preparation of 3-[(N-benzenesulphonyl) anilino]methyl-s-
triazolo(3,4-b) (1,3,4) thiadiazoles-6-(5H)-thione [79]:

To a well stirred clear solution of 3-[(N-benzenesulphonyl)
anilino)methyl-4-amino-5-mercapto-4(H)-1,2,4-triazole [25], (1.44 g, 0.004 mole) and potassium hydroxide (0.336 g, 0.006 mole) in 15 ml of ethanol was added, carbon disulphide (0.46 g, 0.006 mole). The reaction mixture was refluxed until the evolution of hydrogen sulphide gas ceases (7 to 8 hours). The excess of solvent was removed under diminished pressure and the residual mass was poured over crushed ice and neutralised with acetic acid. The solid precipitated was filtered, washed with water, dried and crystallised from ethyl alcohol.

All the 3-[(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino]methyl-s-triazolo (3,4-b) (1,3,4) thiadiazole-6-(5H)-thiones [80-84], obtained in a similar way are listed in (Table VII).

V) Reaction of 3-[(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino]methyl-4-amino-5-mercapto-4(H)-1,2,4-triazoles with hydrazine hydrate.
General procedure for the preparation of 3-[(N-benzene-
sulphonyl/tosyl)-4-(un)substituted anilino]methyl-4-amino-5-
hydrazino-4(H)-1,2,4-triazoles [85-90]:

Preparation of 3-[(N-benzenesulphonyl) anilino]methyl-4-
amino-5-hydrazino-4(H)-1,2,4-triazole [85]:

3-[(N-benzenesulphonyl) anilino]methyl-4-amino-5-mercapto-
4(H)-1,2,4-triazole (1.44 g, 0.004 mole), hydrazine hydrate 99% (0.4 g, 0.004
mole) and absolute ethanol (25 ml) was refluxed on a steam bath till the
evolution of hydrogen sulphide gas ceased. The reaction mixture was then
concentrated and cooled. The separated hydrazino compound was filtered,
washed with cold water, dried and crystalised from ethanol.

Various 3-[(N-benzenesulphonyl/tosyl)-4-(un)substituted
anilino]methyl-4-amino-5-hydrazino-4(H)-1,2,4-triazoles [86-90] thus
obtained are enumerated in (Table VIII).

vi) Reaction of 3-[(N-benzenesulphonyl/tosyl)-4-(un)substituted
anilino]methyl-4-amino-5-mercapto-4(H)-1,2,4-triazoles with
benzoin:

General procedure for the preparation of
3-[(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino]methyl-6,7-
diphenyl-5(H)-s-triazolo (3,4-b) (1,3,4) thiadiazines [91-96]:
Preparation of 3-[(N-benzenesulphonyl) anilino]methyl-6,7-diphenyl-5(H)-s-triazolo (3,4-b) (1,3,4) thiadiazines [91]:

A mixture of 3-[(N-benzenesulphonyl) anilino]methyl-4-amino-5-mercapto-4(H)-1,2,4-triazole [25] (1.083 g, 0.003 mole) and benzoin (0.636 g, 0.003 mole) in ethanol (25 ml) was refluxed for half an hour. To the clear solution was added 1.5 ml of 2N aqueous potassium hydroxide solution and refluxing was continued for 6 hours. The excess solvent was removed under reduced pressure and the solid separated was filtered, dried and crystallised from ethanol.

By adapting the same procedure all the new 3-[(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino]methyl-6,7-diphenyl-5(H)-s-triazolo (3,4-b)(1,3,4) thiadiazines were prepared and are listed in (Table IX) with their analytical data.

vii) Reaction of 3-[(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino] methyl-4-amino-5-mercapto-4(H)-1,2,4-triazoles with chloroacetic acid.

General procedure for the preparation of 7(H)-3-[(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino]methyl-s-triazolo(3-4-b)(1,3,4) thiadiazin-6-(5H)-ones [97-102]:

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Preparation of 7(H)-3-[(N-benzenesulphonyl) anilino] methyl-s-triazolo (3,4-b) (1,3,4) thiadiazin-6-(5H)-one [97]:

3-[(N-benzenesulphonyl) anilino] methyl-4-amino-5-mercapto-4(H)-1,2,4-triazole (2.176 g, 0.006 mole), chloroacetic acid (0.56 g, 0.006 mole) and freshly fused sodium acetate (0.492 g, 0.006 mole) were dissolved in absolute ethanol (30 ml). The reaction mixture was refluxed on a steam bath for 10-12 hours and it was cooled overnight. The solid separated was filtered and washed with water. The residue was then dried and crystallised from ethanol.

All the new 7(H)-3-[(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino]methyl-s-triazolo (3,4-b) (1,3,4) thiadiazin-6-(5H)-one [98-102] obtained in a similar manner are recorded in (Table X).

viii) Reaction of 3-[(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino]methyl-4-amino-5-mercaptop-4(H)-1,2,4-triazoles with piperidine/morpholine:

General procedure for the preparation of 3-[(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino]methyl-4-amino-5-piperidino/morpholino-4(H)-1,2,4-triazoles [103-108 & 109-114]:
Preparation of 3-[(N-benzenesulphonyl) anilino] methyl-4-amino-5-piperidino-4(H)-1,2,4-triazole [103]:

3-[(N-benzenesulphonyl) anilino]methyl-4-amino-5-mercapto-4(H)-1,2,4-triazole (2.16 g, 0.006 mole) was dissolved in absolute ethanol (30 ml). To the above solution was added, piperidine (0.51 g, 0.006 mole) and the resulting mixture was refluxed on a steam bath until the evolution of hydrogen sulfide gas ceased (about six to seven hours). Concentration of the reaction mixture left a residue, which was poured over crushed ice and neutralised with dilute acetic acid. The precipitated solid was filtered, washed with water, dried and crystallised from ethanol.

All the new 3-[(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino] methyl-4-amino-5-piperidino/morpholino-4(H)-1,2,4-triazoles [104-108 & 109-114] prepared in a similar manner are listed in (Tables XI & XII).
PART B

SECTION I:

General procedure for the preparation of 2-mercapto-5-[(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino]methyl-1,3,4-oxadiazoles [115-120]:

Preparation of 2-mercapto-5-[(N-benzenesulphonyl/anilino)methyl-1,3,4-oxadiazole [115]:

[(N-benzenesulphonyl) anilino] acetic acid hydrazide [13], (6.10 g, 0.02 mole) was dissolved in ethanol (30 ml). To this solution were added carbon disulphide (1.52 g, 0.02 mole) and a solution of potassium hydroxide, (1.12 g, 0.02 mole) in water (5 ml). The entire reaction mixture was refluxed on a steam bath till the evolution of hydrogen sulphide gas ceased (8-10 hours). Concentration in vacuo, left a residue which was poured, onto crushed ice and neutralised with acetic acid. The precipitated oxadiazole was filtered, washed with water, dried and crystallised from ethanol.

All the new 2-mercapto-5-[(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino]methyl-1,3,4-oxadiazoles [116-120], obtained in a similar manner are recorded in (Table XIII).
SECTION II:

Reactions of 2-mercapto-5-[(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino]methyl-1,3,4-oxadiazoles:

i) Reaction of 2-mercapto-5-[(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino]methyl-1,3,4-oxadiazoles with hydrazine hydrate:

Alternate method of preparation of 3-[(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino]methyl-4-amino-5-mercapto-4(H)-1,2,4-triazoles [25-30]:

Preparation of 3-[(N-benzenesulphonyl) anilino]methyl-4-amino-5-mercapto-4(H)-1,2,4-triazole [25].

A mixture of 2-mercapto-5-[(N-benzenesulphonyl anilino]methyl-1,3,4-oxadiazoles [115], (1.735 g, 0.005 mole) and hydrazine hydrate 99% (0.5 g, 0.01 mole) in ethanol was refluxed on a steam bath for 7 to 8 hours. Excess of ethanol was removed and the residual mass was poured over crushed ice and carefully neutralized with dilute acetic acid. The solid thus separated was filtered, washed with water, dried and crystallized from ethanol.

The triazoles [25-30] prepared by this method and those prepared from the reaction between potassium salts of dithiocarbazinates
and hydrazine hydrate have the same melting points and mixed melting points did not show any depression. The structures of the compounds are further substantiated by the spectral and elemental analysis.

ii) Reaction of 2-mercapto-5-[(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino]methyl-1,3,4-oxadiazoles with aliphatic and aromatic amines.

**General method for the preparation of 2-(N,N-dimethylamino/dimethyl amino/anilino/piperidino/morpholino)-5-[(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino]methyl-1,3,4-oxadiazoles [121-150]**

**Preparation of 2-(N,N-dimethylamino)-5-[(N- benzenesulphonyl) anilino] methyl-1,3,4-oxadiazole [121].**

A mixture of 2-mercapto-5-[(N-benzenesulphonyl anilino] methyl-1,3,4-oxadiazole [115], (1.735 g, 0.005 mole) and dimethyl amine (0.225 g, 0.005 mole) in ethanol 20 ml was refluxed on steam bath till the evolution of hydrogen sulphide gas ceased, for about 6-7 hours. The solvent was removed under reduced pressure and the residual mass was poured in cold water. The solid separated was filtered, dried and crystallised from ethyl alcohol.

The above procedure was adapted to prepare the remaining
2-((N,N-dimethyl-amino/diethyl amino/anilino/piperidino/morpholino)-
5-[(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino]methyl-1,3,4-
oxadiazoles-[122-150] which are listed in (Table XIV), with their
analytical data.

SECTION III :

General procedure for the preparation of 2-[(H/CH$_3$/-
C$_6$H$_5$/CH$_2$C$_6$H$_5$/C$_6$H$_4$NO$_2$(p)) -5-[(N-benzenesulphonyl/tosyl)-4-(un)
substituted anilino]methyl-1,3,4-oxadiazoles [151-180].

Preparation of 5-[(N-benzenesulphonyl)anilino]methyl-1,3,4-
oxadiazole [151]:

A mixture of [(N-benzenesulphonyl)anilino]acetic acid
hydrazide [13], (1.51 g, 0.005 mole), formic acid, (0.46 g, 0.01 mole)
and thionylchloride (10 ml) was refluxed on a steam bath for 8-10 hours.
After cooling to room temperature the slurry was poured over crushed
ice and the pH of the solution was brought to 7 by the addition of
liquor ammonia. The precipitated solid was filtered, washed thoroughly
with water, dried and crystallized from ethyl alcohol.

Adapting the same method all the new 2-[(H/CH$_3$/-
C$_6$H$_5$/ CH$_2$C$_6$H$_5$/ C$_6$H$_4$NO$_2$(p)) -5-[(N-benzenesulphonyl/ tosyl) -4-
(un)substituted anilino] methyl-1,3,4-oxadiazoles [152-180], were obtained
and are enumerated in (Table XV), with their analytical data.
PART C

SECTION I:

Reaction of [(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino]acetic acid hydrazide.

General procedure for the preparation of 1-[(N-benzene sulphonyl/tosyl)-4-(un)substituted anilino]acetyl amido-2-[(4-OH/-OCH$_3$/Cl/N,N-dimethylamino) benzylidene] hydrazines [181-204]:

Preparation of 1-[(N-benzenesulphonyl)anilino]acetyl amido-2-(4-hydroxy benzylidene) hydrazine [181]:

1-[(N-benzenesulphonyl)anilino]acetyl amido-2-(4-hydroxy benzylidene) hydrazine [181], was prepared by refluxing [(N-benzenesulphonyl) anilino] acetic acid hydrazide [13], (3.05 g, 0.01 mole) with 4-hydroxy benzaldehyde, (1.22 g, 0.01 mole) in ethanol and 3-4 drops of piperidine, for one hour. The reaction mixture was cooled, the solid separated was neutralised by sodium bicarbonate solution, washed with cold ethanol, dried and crystallised from ethanol.

The above method was adapted to prepare the remaining 1-[(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino] acetyl amido-2-[(4-OH/OCH$_3$/Cl/N,N-dimethyl amino)benzylidene] hydrazines [182-204] and are listed in (Table XVI), with their analytical data.
SECTION II:


General procedure for the preparation of 2-[(4-OH/OCH$_3$/Cl/N,N-dimethyl) amino phenyl]-3-[(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino] acetyl amido-1,3-thiazolidin-4-ones [205-228]:

Preparation of 2-(4-hydroxyphenyl)-3-[(N-benzenesulphonyl) anilino] acetyl amido-1,3-thiazolidin-4-one [205]:

A mixture of 1-[(N-benzenesulphonyl) anilino] acetyl amido-2-(4-hydroxy benzylidene) hydrazine [181], (4.09 g, 0.01 mole), thioglycolic acid (1.38 g, 0.01 mole) and dry benzene (20 ml) was refluxed on a steam bath for 8-10 hours. Excess of solvent was removed under reduced pressure. The residue was washed with saturated solution of sodium bicarbonate and then with water. The solid separated was filtered, washed repeatedly with water, dried and crystallised from ethanol.

The above procedure was adapted to prepare 2-[(4-OH/OCH$_3$/Cl/N,N-dimethyl amino) phenyl]-3-[(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino] acetyl amido-1,3-thiazolidin-4-ones [206-228] and are recorded in (Table XVII), along with their analytical data.
SECTION III:

Reaction of 1-[(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino]acetyl amido-2-[(4-OH/OCH₃/Cl/N,N-dimethyl amino) benzyldene] hydrazines [181-204] with chloroacetyl chloride:

**General procedure for the preparation of 1-[(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino]acetyl amido-3-chloro-4-[(4-OH/OCH₃/Cl/N,N-dimethyl amino) phenyl] azetidin-2-ones [229-252]:**

**Preparation of 1-[(N-benzenesulphonyl) anilino] acetyl amido-3-chloro-4-(4-hydroxyphenyl) azetidin-2-one [229].**

To 1-[(N-benzenesulphonyl) anilino] acetyl amido-2-(4-hydroxy benzylidene) hydrazine, (4.09 g, 0.01 mole) and triethyl amine, (90.5 g, 0.005 mole) dissolved in dry benzene (50 ml), chloro acetyl chloride (0.569 g, 0.005 mole) in dry benzene (50 ml) was added dropwise with stirring during one hour. The reaction mixture was stirred further for four hours and triethylamine hydrochloride separated was filtered. The filtrate was concentrated under reduced pressure and remaining viscous liquid was digested with a mixture of n-hexane and diethyl-ether (1:3) The resulting solid was then digested with ethanol for one hour. The solution was concentrated, treated with animal charcoal and filtered. Crystals of the title compound separated on standing.
The above method was followed to prepare remaining 1-[(N-benzenesulphonyl/tosyl)-4-(un)substituted anilino] acetyl amido-3-chloro-4-[(4-OH/OCH₃/Cl/N,N-dimethyl amino) phenyl] azetidin-2-ones [230-252] and are enumerated in (Table XVIII), along with their analytical data.
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