SYNTHESIS AND SCREENING OF (E)-1-(4-(2-(((PHENYLAMINO)METHYL)AMINO)ACETYL)PHENYL)-4-(2-PHENYLHYDRAZONO)-3-(TRICHLOROMETHYL)-1H-PYRAZOL-5(4H)-ONE

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ABSTRACT:
Synthesis of (E)-1-(4-(2-(((phenylamino)methyl)amino)acetyl)phenyl)-4-(2phenylhydrazono)-3-(trichloromethyl)-1H-pyrazol-5(4H)-one achieved by reaction of E-2-(4-(5-oxo-4-(2-phenylhydrazono)-3-(trichloromethyl aceticacid (2)) in presence of DMF, aq NaNO₃, isobutyl formamide afforded corresponding compound (3) which was subjected to mannich reaction with cyclic secondary amines such as piper dine or morpholine or N-methyl piparazine in presence of formaldehyde in DMF to yield corresponding mannish base(E)-1-(4-(2-(((phenylamino)methyl)amino)acetyl)phenyl)-4-(2phenylhydrazono)-3-(trichloromethyl)-1H-pyrazol-5(4H)-one (4) in excellent yield. The structure of these newly synthesized compounds were characterised by H¹-NMR,C¹³-NMR, Mass and IR elemental analysis

Keywords : - Pyrozolone , Mannich bases , β-lactam, urea .

INTRODUCTION
Heterocyclic chemistry is the most challenging and a handsomely rewarding field of study, since it always attracts the attention of scientists working not only in the area of natural products but also in synthetic chemistry. Moreover, in tune with the present trend “scientists to the door steps of common man”, there is always a challenging and rewarding task in search of more and more new scientific accomplishments. This is reflected by the voluminous data available in the literature on heterocyclic chemistry. Many useful drugs indeed have emerged from such investigations which strengthens the trend. Spectacular advanced has been made in this field to furtherance of the knowledge of relationship between chemical structure and biological activity. Thus, the successful application of this class of compounds in various fields ensures a limitless scope for the development of structurally novel compounds with a wide range of physico-chemical and biological properties.

Amongst different heterocyclic systems, the chemistry of five membered heterocycles with more than one heteroatom has gained importance as many of them exhibit pronounced bioactive nature. One such type of compounds includes pyrazoles and pyrazolines and pyrazolones. Hence, any attempt to study their detailed chemistry would add new dimensions to
the existing knowledge. Pyrazolones, pyrazoles and related heterocycles possess various types of biological activities. A good deal of importance is given to pyrazolone derivatives. It is due to their wide use in medical chemistry and some of them possess antituberculosis antineoplastic, antidiabetic, antifertility and antithyroid activity. In this perspective a study on synthesis, characterization, antimicrobial activity, and bioactive studies on some pyrazolone derivatives have been taken up.

Introduction

A brief account on 2-pyrazolines, their importance and various methods for their synthesis is discussed.

A five membered cyclic diene containing three carbons and two nitrogens is called a diazole. If two nitrogen atoms are adjacent, it is known as a pyrazole. If one double bond is present, it is a pyrazoline. The biological properties of pyrazolones are reviewed extensively. Several pyrazolines [1] and annulated pyrazoles[ 2-4 ]possess antimicrobial activity[5]. Pyrazole and its N-substituted derivatives are potential inhibitors and deactivators of liver alcohol dehydrogenase. Difenamizole[ 5 ]poses’s analgesic activity greater than that of Aspirin.

The trifluoro derivatives of pyrazolines[ 6& 7 ]are about 0.5% as effective as an amebicide, comparable with Emetine and Metronidazole.

Several di and tri substituted pyrazole and pyrazoline derivatives[6] and 4-pyrazolyl pyridinium salts [ 8] possess hypoglycemic activity. Muzolimine[ 9,] 1-substituted 2-pyrazolin-5-one derivative is a highly active diuretic. It differs from other diuretics as it contains neither sulfonamide nor carboxyl group. Besides this, pyrazoline and indazole derivatives [10-12 ] are pharmacologically active and are useful as antinflammatory drugs7,8.

As well as 3,5-pyrazolidinedione derivatives such as phenylbutazone [13] oxyphenbutazone [14] sulfinpyrazone [15] etc, are some of the important class of anti-inflammatory agents which are most widely used.

Acyl azides, in general, and N-protected a-amino acid azides in particular, have occupied a place of their own importance in organic[16], and peptide as well as peptidomimetic[17], syntheses. They are extensively used in the preparation of amides and peptides, and a wide range of other compounds such as nitriles, and several classes of heterocycles. [16,18 ]. The Curtius rearrangement of acyl azides into isocyanates of paramount value in synthetic chemistry. It is widely used in the preparation of amines, ureas and carbamates. A number of natural products and pharmacologically important compounds containing uriedo linkages, [19], ureidopeptidomimetics, [20], partially modified retro-inverso (PMRI)peptides, formamides and unnatural amino acids have been prepared via this rearrangement. [17,22]. Due to such vast utility of acid azides, the development of efficient routes for their synthesis is important. The two well known routes for the preparation of acid azides are the reaction of NaN3 with an acid chloride[23], or mixed anhydride. [24]. The acid chloride method offers disadvantages at the preparation of acid chloride itself. These include prolonged reaction duration, incompatibility with acid cleavable groups, and storage and stability problems associated with moisture sensitive acid chlorides. Also the poor solubility of NaN3 inorganic reaction medium requires the usage of a phase transfer catalyst, [25 ], or catalysts such as ZnI2 [26], to improve the yield of acid azides. Alternately, protocols for the in situ generation of acid chlorides using SOCl2/DMF–NaN3,[26], cyanuric chloride/N-methylmorpholine,[27], triphosgene/triethylamine,[28],
N,N-chloromethylenedimethylammonium chloride,[29], followed by coupling with an azide have also been reported. But these methods are not suitable for acids such as N-Boc/Z-a-amino acids whose acid chlorides are unstable. Preparation of acid azides via mixed anhydrides has been used to advantage. Yet, this method uses chloroformates which are inconvenient for handling. Katritzky et al., recently prepared acid azides from acids in a two step route involving N-acyl benzotriazoles as stable and reactive intermediates. [30], Acid azides, such as Boc/Z-a-amino acidazides, have also been prepared through a multi-step route starting from acids by hydrazinolysis of the methyl/ethyl esters followed by reaction of the resultant hydrazide with nitrosyl donors like HNO2.

MATERIALS AND METHODS
All the chemicals were used as received without further purification. Melting points were determined in open capillary tubes in Buchi530 circulating oil apparatus and are not corrected. Reactions were carried out using household micro oven (power consumption 1200 W, microwave frequency 2450 MHz) and monitored by thin layer chromatography (TLC) on silica gel plates (60 F254) visualizing with ultraviolet light or iodine spray. 1H NMR spectra were determined in DMSO-d6 solution on JOEL AL300 Spectrometers. Proton chemical shifts (δ) are relative to tetramethylsilane as internal standard and expressed in ppm.

Results and discussions
A series of four novel mannish bases were obtained from substituted aniline by dissolving in suitable volume of water containing 2.5 - 3.5 equivalence of HCl by the application of heat to afford substituted phenyl diazonium chloride.(A). A is treated with a solution of sodium acetated in presence of ethyl trichloroacetoacetic ester compound (B) is obtained. Compound (B) is condensed with 4 – hydrazenyl phenol and DMF to obtain 3–trichloromethyl 4-substituted phenylhydrazono pyrazoline 5-one (C). Compound (C) is stirred at room temperature in presence of anhydrous K2CO3 to yield compound (1) which amination with hydrazine hydrate in presence of ethanol afforded a ethyl 2-(4-(5 –oxo phenyl hydrazono )-3-trichloromethyl 4,5-di hydro –H pyrazol-1 –yl) phenoxy ) aceto hydrazide(2). Compound 2 is condensed with Isatin in presence of DMF to afford a 2 –(4-(5-oxo -4-(2-4-substituted)hydrazono )-3-(trichloromethyl )-4,5-dihydro -1H –pyrazol –yl)-N1-(2-oxo indoline -3-ylidene) aceto hydrazide (3). Compound (3) is reacted with mannish bases, formaldehyde and DMF (piperidine ,morpholine ,N-Methyl piparazine to obtain compound (4) N1-(2- oxo-1-(4-substituted)hydrazono 3-(trichloromethyl )-4,5-dihydro-1H-pyrazol-1-yl)phenony) aceto hydrazide.

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The required primary amine is dissolved in a suitable volume of water containing 2.5 – 3.0 equivalents of hydrochloric acid (or sulphuric acid) by the application of heat if necessary. The solution thus obtained is cooled to 0°C when the amine hydrochloride (or sulphate) usually crystallizes. The temperature is maintained at 0 – 5°C, and the aqueous solution of sodium nitrite is added portion wise till there is free nitrous acid. The solution is tested for the later with an external indicator (moist potassium iodide starch paper). An excess of acid is always maintained to stabilize the diazonium salt, acid is harmful, the concentration of the acid is reduced to optimum value. The similar procedure is adopted for the preparation of other substituted phenyl diazonium chlorides.

**Substituted phenyl diazonium ethyltrichloroacetateaceticester(B)**

A solution of sodium acetate (1.0g) in 100 ml of aqueous alcohol (50%) is added to a solution of ethyltrichloroacetate ester (0.1 mole) in 50 ml of ethanol and the mixture is added to 0°C. To this cold mixture, the corresponding diazonium chloride is added gradually till turbidity is observed. The addition is continued till yellow crystals separated out. These crystals are filtered, washed with water and dried.

**3-Trichloromethyl-4-(substituted phenyl hydrazono)-pyrozoline-5-one(C)**

Condensation of 4-substituted phenyl hydrazono acetoacetic ester B and 4- hydroxy phenyl hydrazine in the presence of catalytic amount of dimethyl formamide under microwave irradiation afforded C. In typical experimental procedure, a mixture of aryl hydrazono acetic ester B, 4-hydrazinyl phenol and dimethyl formamide (10 drops) was subjected to microwave irradiation at 150W intermittently at 30 sec intervals for 2 minutes. After complete conversion as indicated by TLC, the reaction mixture was cooled and heated with cold water. The precipitate C was filtered recrystallized from ethanol M.P. 159°C, yield 85%. The mass spectra of 2–(4–(5–oxo–4–(2–phenyl hydrazono)–3–(trichloromethyl)–4,5–dihydro–1–H pyrazol–1–yl)phenoxy)N–(2–oxoindolin)–3–ylidine) aceto hydrazide 1a (R=H) showed molecular ion (M+) peaks at m/z 598.5

**ethyl 2–(4–(5–oxo phenyl hydrazono)–3–(trichloromethyl)–4,5–dihydro–1H -pyrazol–1–y)phenoxy)acetohydrazide (1)**

A mixture of synthesized 3-trichloromethyl-4-(substituted phenyl hydrazono)-pyrozoline-5-one(C), anhydrous K₂CO₃, chloroethylacetate and DMF was stirred at room temperature for 8 hours. The reaction mixture was diluted with ice cold water. The separated solid was identified as ethyl 2–(4–5–oxo–4–(2–phenylhydrazono)–3–(trichloromethyl)–4,5–dihydro–1Hpyrazol–1–yl)phenoxy) acetate.

**E-2-(4-(5-oxo-4-(2-phenylhydrazono)-3-(trichloromethylaceticacid)-4,5-DiHydro-1H-pyrazol-1-yl) phenoxy) aceticacid(2)**

Synthesis of E-2-(4-(5-oxo-4-(2-phenylhydrazono)-3-(trichloromethylaceticacid)-4,5-DiHydro-1H-pyrazol-1-yl)Phenoxy) acetic acid was done by adding the solvent mixture tetrahydro Furan/ methyl alcohol /H₂O (1:1:1) ratio, aq NaoH(2N) was added and refluxed for 6hrs. The progress of the reaction was monitored by cyclohexane : ethyacetate (4:6) solvent mixture as an eluent. After completion of reaction solvent was evaporated under vaccum to give crude. The residue was washed with ethyl acetate to remove impurites. The residue was acidified with 1N HCl to give solid suspension which was filtered under vaccum to give crude, purified by chromatography (60-120 Mesh silica gel eluent :70/ethyacetate –pet ether)to afford acid compound E-2-(4-(5-oxo-4-(2-phenylhydrazono)-3-(trichloromethyl aceticacid)-4,5-dihydro-1H-pyrazol-1-yl) phenoxy) aceticacid. The structures of this newly synthesized compounds were characterised by ¹H-NMR and IR spectral data.

**The IR(kBr)** spectrum of E-2-(4-(5-oxo-4-(2-phenylhydrazono)-3-(TrichloroMethyl aceticacid)-4,5-DiHydro-1H-pyrazol-1-yl)Phenoxy) aceticacid was recorded in the range 4000-667cm⁻¹ and the absorption signals were found at 3260(NH), 2950 (OH), 3100( Ar-H), 2990 and 2960(aliphatic CH₂and CH₃) 1785 (CO of ester group), 1680 (C=N), and 1195(C-O-C of ester group)1195(C=S).
E-2-(4-(5-oxo-4(2-phenylhydrazono)-3-(trichloromethyl)-4,5-dihydro-1H-pyrazol-1-yl)phenoxy) acetyl azide(3)

To solutions of acid in acetone triethyl amine (3eq) was added and stirred at 15 °C. To that isobutyl chloroformate (1:1) was added and stirred for 20 min at 0 °C. After completion, reaction mixture was poured in ice cold water (20ml) extracted with diethylether (10 times). The organic layer was separated, washed with water, brine dried over anhydrous Na₂SO₄. Filtered and evaporated under vaccum to give crude oil. The crude oil was purified by column chromatography (60/120 mesh silica gel eluent; 10/EtOAc-pet ether) to give pure 2-(3-(4-chloro-3-oxo-1-((4-(4-triflomethyl)phenyl) E-2-(4-(5-oxo-4(2-Phenyl hydrazono)-3-(trichloromethyl)-4,5-dihydro-1H-pyrazol-1-yl)phenoxy)acetyl azide 3a

IR(KBr): 3205(-NH), 3170(Indol -NH), 1602(-C=N), 1656(pyrazoline –C=O), 1700(pyrazole- C=O), 1618(-CO-NH).

1H NMR(300MHZ,(CD)₂SO,TMS); δ=9.28(s, 1H, CO–NH), 10.97(s, 1H, Ar–NH=), 10.54(s, 1H, Indole –NH), 4.85(s, 2H, O–CH₂–CO), 6.87–7.83(m, 9H, Ar–H)

C₁₃ Spectrum of (CDCl₃) δ=20.8,27.7,29.2,102.0,152.7,32.6,31.3,(Ar-c),65.6(-CH₂),153.8(-NH-N=C),171.1(Pyrrol-C=O),94.9(CCl₃)155.6(CCl₃ C),133.5(C=ONHNH₂),21.6,28.5,27.7,119.1,133.9(Phenox),25.2,25.7,126.0,131.3,119.0,139.115.6(Indoline-c) yield 70,M.P. 0 °C 212-

Calculated Values:C:52.16,H:3.00N:16.34,O:10.62,Cl:17.45

Found(%):C:52.26,H:3.01,N:16.58,O:10.72,Cl:17.58

(E)-1-(4-(2-(((phenylamino)methyl)amino)acetyl)phenyl)-4-(2-(p-tolyl)hydrazono)-3-(trichloromethyl)-1H-pyrazol-5(4H)-one 4(b):

IR(KBr): 3170(-NH),1616(-CN),1674(PYRAZOLINE(-C=O)1715,1658(-CO-NH),2920(-CH₂),

1H NMR(300MHZ,(CD)₂SO,TMS); δ= 3.20(s, 3H, Ar–OCH₃), 1.47–1.51(m, 6H, (CH₂)₆ of piperidine ring), 4.05(s, 2H, N–CH₂–N), 4.87(s, 2H, O–CH₂–CO), 10.99(s, 1H, Ar–NH=), 9.25(s, 1H, CO–NH), 6.85–7.82(m, 14H, Ar–H)

C₁₃ Spectrum of (CDCl₃) δ=20.8,27.7,29.2,102.0,152.7,32.6,31.3,(Ar-c),65.6(-CH₂),153.8(-NH-N=C),171.1(Pyrrol-C=O),94.9(CCl₃)155.6(CCl₃ C),133.5(C=ONHNH₂),21.6,28.5,27.7,119.1,133.9(Phenox),25.2,25.7,126.0,131.3,119.0,139.115.6(Indoline-c) yield 70,M.P. 0 °C 212-

Calculated Values:C:52.16,H:3.00N:16.34,O:10.62,Cl:17.45

Found(%):C:52.26,H:3.01,N:16.58,O:10.72,Cl:17.58

(E)-1-(4-(2-(((phenylamino)methyl)amino)acetyl)phenyl)-4-(2-phenylhydrazono)-3-(trichloromethyl)-1H-pyrazol-5(4H)-one 4(a):

To a mixture of pure E-2-(4-(5-oxo-4(2-Phenyl hydrazono)-3-(trichloromethyl)-4,5-dihydro-1H-pyrazol-1-yl)phenoxy)acetyl azide.3(a)(1eq), in benzene (1eq) was added HCHO and refluxed for 16hrs. Progress of the reaction was monitored by TLC with acetone-ethyl acetate ( 6:4) as mobile phase. After completion of reaction solvent was evaporated under vaccum to give crude residue, purified by column chromatography 60 mesh silica gel to give 4a

IR(KBr): 3195(-NH),1610(-C=N),1676(Pyrazoline(-C=O)1715,1658(-CO-NH),2920(-CH₂),

1H NMR(300MHZ,(CD)₂SO,TMS); δ=1.49–1.53(m, 6H, (CH₂)₆ of piperidine ring), 2.25(t, 4H, CH₂–N–CH₂ of piperidine ring), 4.05(s, 2H, N–CH₂–N), 4.82(s, 2H, O–CH₂–CO), 10.99(s, 1H, Ar–NH=), 9.25(s, 1H, CO–NH), 6.85–7.82(m, 14H, Ar–H)

C₁₃ Spectrum of (CDCl₃) δ=20.8,27.7,29.2,102.0,152.7,32.6,31.3,(Ar-c),65.6(-CH₂),153.8(-NH-N=C),171.1(Pyrrol-C=O),94.9(CCl₃)155.6(CCl₃ C),133.5(C=ONHNH₂),21.6,28.5,27.7,119.1,133.9(Phenox),25.2,25.7,126.0,131.3,119.0,139.115.6(Indoline-c) yield 70,M.P. 0 °C 212-

Calculated Values:C:52.16,H:3.00N:16.34,O:10.62,Cl:17.45

Found(%):C:52.26,H:3.01,N:16.58,O:10.72,Cl:17.58

(E)-1-(4-(2-(((phenylamino)methyl)amino)acetyl)phenyl)-4-(2-(p-tolyl)hydrazono)-3-(trichloromethyl)-1H-pyrazol-5(4H)-one 4(b):

IR(KBr): 3170(-NH),1616(-CN),1674(PYRAZOLINE(-C=O)1715,1658(-CO-NH),2920(-CH₂),

1H NMR(300MHZ,(CD)₂SO,TMS); δ= 3.20(s, 3H, Ar–OCH₃), 1.47–1.51(m, 6H, (CH₂)₆ of piperidine ring), 2.28(t, 4H, CH₂–N–CH₂ of piperidine ring), 4.10(s, 2H, N–CH₂–N), 4.87(s, 2H, O–CH₂–CO), 10.94(s, 1H, Ar–NH=), 9.32(s, 1H, CO–NH), 6.89–7.79(m, 13H, Ar–H)

C₁₃ Spectrum of (CDCl₃) δ=20.8,26.7,30.01,102.0,150.7,31.06(Ar-c),60.02(-CH₂),164(-NH-N=C),115(Pyrrol-c=0),90(CCl₃),65(CCl₃),158(C=ONHNH₂),16.03,24.8,36.05,
E)-4-(2-(4-methoxyphenyl)hydrazono)-1-(4-((phenylamino)methyl)amino)acetyl)phenyl)-3-(trichloromethyl)-1H-pyrazol-5(4H)-one 4(c)

IR(KBr): 3120(-NH), 1610(-C=N), 1680(Pyrazoline-C=O), 1712(Indole-C=O), 1654(-CONH), 2625(-CH$_2$), 1$^H$ NMR(300MHZ,(CD)$_2$SO,TMS); δ= 3.75(s, 3H, Ar–OCH$_3$), 1.42–1.53(m, 6H, (CH$_2$)$_6$ of piperidine ring), 2.31(t, 4H, CH$_2$–N–CH$_2$ of piperidine ring), 4.13(s, 2H, N–CH$_2$–N), 4.79(s, 2H, O–CH$_2$–CO), 10.85(s, 1H, Ar–NH=N), 9.28(s, 1H, CO–NH), 6.83–7.75(m, 12H, Ar–H)

C$_{13}$ Spectrum of (CDCl$_3$)$\delta$= 20.08, 27.7, 29.02, 102.02, 152.07, 32.68(Arc), 62.03(CH$_2$), 163(N=C)

Found(%): C:55.93, H:4.37, N:15.81, O:9.03, Cl:14.83

(E)-4-(2-(4-chlorophenyl)hydrazono)-1-(4-((phenylamino)methyl)amino)acetyl)phenyl)-3-(trichloromethyl)-1H-pyrazol-5(4H)-one 4(d)

IR(KBr): 3175(-NH), 1614(-C=N), 1674(Pyrazoline), 1714(Indole-C=O), 1656(-CONH), 2915(-CH$_2$), 1$^H$ NMR(300MHZ,(CD)$_2$SO,TMS); δ= 1.75(t, 3H, –CH$_3$), 3.32(q, 2H, O–CH$_2$), 1.42–1.53(m, 6H, (CH$_2$)$_6$ of piperidine ring), 2.31(t, 4H, CH$_2$–N–CH$_2$ of piperidine ring), 4.21(s, 2H, N–CH$_2$–N), 4.80(s, 2H, O–CH$_2$–CO), 10.91(s, 1H, Ar–NH=N), 9.33(s, 1H, CO–NH), 6.93–7.87(m, 13H, Ar–H)

C$_{13}$ Spectrum of (CDCl$_3$)$\delta$= 20.08, 26.02, 28.32, 101.32, 153.102.3, 32.65(Ar-c), 61.03(-CH$_2$), 162(-NH-N=C)

Found(%): C:55.28, H:4.47, N:15.17, O:10.84, Cl:14.2

(E)-4-(2-(4-nitrophenyl)hydrazono)-1-(4-((phenylamino)methyl)amino)acetyl)phenyl)-3-(trichloromethyl)-1H-pyrazol-5(4H)-one 4(e)

IR(KBr): - 3155(-NH), 1616(-C=N), 1674(Pyrazoline), 1716(Indole-C=O), 1658(-CONH), 2916(-CH$_2$), 1$^H$ NMR(300MHZ,(CD)$_2$SO,TMS); δ= 1.45–1.57(m, 6H, (CH$_2$)$_6$ of piperidine ring), 2.34(t, 4H, CH$_2$–N–CH$_2$ of piperidine ring), 4.23(s, 2H, N–CH$_2$–N), 4.75(s, 2H, O–CH$_2$–CO), 10.96(s, 1H, Ar–NH=N), 6.89–7.95(m, 12H, Ar–H)

C$_{13}$ Spectrum of (CDCl$_3$)$\delta$= 20.08, 25.02, 29.101.32, 154, 102.3, 30.65(Arc), 59.13(CH$_2$), 162(-NH-N=C)


(E)-1-(4-(2-(((phenylamino)methyl)amino)acetyl)phenyl)-3-(trichloromethyl)-4-(2-(4(trifluoromethyl)phenyl)hydrazono)-1H-pyrazol-5(4H)-one 4(f)

IR(KBr): - 3170(-NH), 1614(-C=N), 1674(Pyrazoline-C=O), 1716(Indole-C=O), 1626(-CONH), 2625(-CH$_2$), 1$^H$ NMR(300MHZ,(CD)$_2$SO,TMS); δ= 1.42–1.53(m, 6H, (CH$_2$)$_6$ of piperidine ring), 2.52(t, 4H, CH$_2$–N–CH$_2$ of piperidine ring), 4.18(s, 2H, N–CH$_2$–N), 4.79(s, 2H, O–CH$_2$–CO), 10.91(s, 1H, Ar–NH=N), 6.92–7.85(m, 12H, Ar–H), 8.97(s, 1H, CO–NH)

C$_{13}$ Spectrum of (CDCl$_3$)$\delta$= 20.08, 23.23, 29.100.32, 154, 102.3, 30.65(Arc), 59.13(CH$_2$), 168(NHN=C)

Antibacterial activity by disc diffusion method for Synthesis (E)-1-(4-((phenylamino)methyl)amino)acetyl)phenyl)-4-(2-phenylhydrazono)-3-(trichloromethyl)-1H-pyrazol-5(4H)-one 4(a-f)

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