CHAPTER-IV

Synthesis of Piperazine 1-yl (3,4,5-trimethoxyphenyl) methanone derivatives
INTRODUCTION

The number of patients suffering from cancer diseases or some life-threatening infections has continued to rise rapidly with years, though great progresses have been made in diagnosis, prevention, therapy and medicinal chemistry. Especially, the alarming rates of emerging drug resistant strains and cancer cell lines, leading to failure in therapy, continue to serve as impetus for the development of novel and more effective antimicrobial and anticancer agents. [1-3]. The piperezine-based research has attracted considerable attention in recent years. Piperazine and substituted piperazine nuclei had constituted an attractive pharmacological scaffold present in various potent marketed drugs. The incorporation of piperazine is an important synthetic strategy in drug discovery due to its easy modifiability, proper alkaline, water solubility, the capacity for the formation of hydrogen bonds and adjustment of molecular physicochemical properties [4-5]. A broad range of biologically active compounds displaying antibacterial [6-8], antifungal [9-10], anticancer [11-13], antiparasitic, [14-15] antihistaminic [16], psychotolytic [17], and antidepressive activities [18] have been also found to contain this versatile core. In particular, structurally simple methoxy piperazine, as the efflux pump inhibitor, could exert positive effect on tetracyclines and ciprofloxacin against their resistant bacteria [19-20]. Moreover, benzotriazole-based piperazine derivatives and N,N'-bis(alkyloxymethyl)piperazines had moderate antibacterial and antifungal activities against pathogenic bacterial strains and fungal strains [21-22]. On the other hand, a novel microtubule depolymerizing piperazine derivative,1-(5-chloro-2-methoxybenzoyl)-4-(3-chlorophenyl) piperazine, caused inhibition of proliferation of a wide range of cancer cell lines including a multidrug-resistant cell line, with an average IC50 of 85 nM [23]. These results once again
highlighted that piperazine core was an important backbone and prompted us to design some active molecules with piperazine nucleus. Several literatures provided evidence that the introduction of such bulky groups like diphenyl could increase antimicrobial activity by enhancing lipophilicity of the molecule, which may result in more penetration into cells [24]. Thus piperazine derivatives bearing substitution at N1 position also have been introduced to design new antibacterial and antifungal agents, though this moiety has wide application as antihistamine like cetirizine, calcium antagonist (flunarizine) etc.

particularly during the past two decades, a number of different classes of antibacterial [25-31] and antifungal agents [32-38] have been discovered. Although, since the discovery of several synthetic and semi-synthetic antibacterial sulfa drugs, nitrofuranes, penicillins, cephalosporins, tetracyclines, macrolides, and oxazolidinones, and antifungal agents such as fluconazole, ketoconazole and miconazole,

including amphotericin B, there has been much progress in this field. Despite advances in antibacterial and antifungal therapies, many problems remain to be solved for most antimicrobial drugs available. For example, appearance of multidrug resistant Gram-positive bacteria, in particular, methicillin-resistant Staphylococcus aureus and vancomycin-resistant Enterococci is causing a serious menace. The use of amphotericin B, known as the gold standard, is limited because of its infusion related reactions and nephrotoxicity [39-40].

PRESENT WORK

In our present research work we are planned to synthesis of derivatives of trimethoxybenzene piperazines with substituted acid chlorides. Acid chlorides was prepared by dissolve 1 eq of acids in dichloromethane at 50°C to this added 1.2 eq of
oxalyl chloride followed by 2 drops of DMF. The reaction mass was stirred for 1 hr at cooled condition.

Compound 8 (0.5 g, 0.00218 mol 1.0 eq) was dissolved in dry Dichloromethane (10 mL). The solution was stirred for 10 min at ambient temperature. Triethylamine (3.0 eq) was added, the solution was cooled 5 °C, the above prepared cooled acid chloride solution was slowly added. To the reaction mixture the solution was stirred for 30 min at cooled condition. The completion of the reaction was monitored by TLC. The reaction mixture was washed with water followed by saturated sodium bicarbonate solution, brine solution dried over anhydrous sodium sulphate. The organic layer was evaporate under reduced pressure and the crude was purified by column chromatography using 60-120 mesh silica gel. All the synthesized compounds have been purified by column chromatography. The structures have been confirmed by elemental analysis and spectroscopic techniques like IR, ¹H-NMR, LC-MS. The series of reactions carried out have been depicted in scheme 3 and derivatives are depicted in table-3.

**Scheme 3**

![Scheme 3 diagram](image-url)
Table 3 Synthesis of piperazine methanone derivatives

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EXPERIMENTAL

Preparation of tert-Butyl 4-(3,4,5-trimethoxybenzoyl)piperazine-1-carboxylate (7).

The synthesis of compound 7 through acid amine coupling method using EDCI.HCl and HOBT.H2O. 3,4,5-trimethoxybenzoic acid (2.00 g, 9.42 mmol.) was dissolved in dry tetrahydrofuran (20 mL). The solution was stirred for 10 min at ambient temperature. 1-Ethyl-3-(3-dimethylaminopropyl)carbodiimide hydrochloride (1.609 g, 10.36 mmol) was added, followed by 1-hydroxybenzotriazole (1.40 g, 10.36 mmol.) and N,N-diisopropylethylamine (4.811 g, 37.2 mmol.). The reaction mixture was stirred for 20 min at ambient temperature, and then it was cooled to 0°C. Boc-piperazine (tert-butyl piperazine-1-carboxylate) (2.31 g, 10.2 mol) was added portion-wise to the mixture and stirring was continued for 6 h at ambient temperature. The completion of the reaction was monitored by TLC. The reaction mass was diluted with ethyl acetate and washed with saturated sodium bicarbonate solution followed by water and brine solution. It was finally dried over sodium sulphate and evaporate under reduced pressure to offered crude 3.2 g of tert-butyl 4-(3,4,5-trimethoxybenzoyl)piperazine-1-carboxylate (7).


Preparation of Piperazin-1-yl(3,4,5-trimethoxyphenyl)methanone (8).

Compound 7 (3.2g, 9.71mol.) was dissolved in dry dichloromethane and the mixture was cooled 50°C. Trifluoroacetic acid (3.32 g, 29.14 mmol.) was added slowly to the cooled mixture and stirred for 6 h at ambient temperature. The completion of the reaction was monitored by TLC. The reaction mixture was evaporate under reduced pressure and it was dissolved in dichloromethane. It was washed with water, brine and dried over sodium sulphate and evaporate under reduced pressure to offered crude product 1.8g of Piperazin-1-yl(3,4,5-trimethoxyphenyl)methanone (8).

LC-MS (ESI, Positive): m/z: [M+H]^+: 282.1; ^1H NMR: (400 MHz, DMSO-d_6): δ 6.66 (s, 2H), 3.79 (s, 6H), 3.68 (s, 3H), 3.41(bs,4H), 2.74 (bs, 4H); Elemental analysis: Calculated for C_{14}H_{20}N_{2}O_{4}: C 59.99, H 7.19, N 9.99; Found: C 59.95, H 7.21, N 9.96.

General procedure for synthesis of 9a-t

The synthesis of derivatives trimethoxybenzene piperazines with substituted acid chlorides. Acid chlorides was prepared by dissolve 1 eq of acids in
dichloromethane at 50°C to this added 1.2 eq of oxalyl chloride followed by 2 drops of DMF. The reaction mass was stirred for 1 hr at cooled condition.

Compound 8 (0.5 g, 0.00218 mol 1.0 eq) was dissolved in dry Dichloromethane (10 mL). The solution was stirred for 10 min at ambient temperature. Triethylamine (3.0 eq) was added, the solution was cooled 50°C, the above prepared cooled acid chloride solution was slowly added. To the reaction mixture the solution was stirred for 30 min at cooled condition. The completion of the reaction was monitored by TLC. The reaction mixture was washed with water followed by saturated sodium bicarbonate solution, brine solution dried over anhydrous sodium sulphate. The organic layer was evaporate under reduced pressure and the crude was purified by column chromatography using 60-120 mesh silica gel. All the synthesized compounds have been purified by column chromatography. Pure compound obtained after column purification 9a-t was synthesized.

SPECTRAL DATA

(4-(2-Amino-5-chlorobenzoyl)piperazin-1-yl)(3,4,5-trimethoxyphenyl)methanone (9a)

[Chemical structure image]

9a

LC-MS (ESI, Positive): m/z: [M+H]+: 436.3; 1H NMR (400 MHz, DMSO-d6): δ 7.13-7.10 (dd, J =7.6 Hz, 1H), 7.02-7.01 (d, J = 3.2 Hz, 1H), 6.72 (s, 1H), 6.69 (s, 2H), 5.36 (s, 2H), 3.79 (s, 6H), 3.68 (s, 3H), 3.55-3.44 (m, 8H); IR (KBr) v (cm⁻¹): 1604 (C=O); Elemental analysis: Calculated (%) for C21H24ClN3O5: C 58.13, H 5.58, N 6.98; Found: C 58.12, H 5.57, N 6.99.
(3-Chlorobenzo[b]thiophen-2-yl)(4-(3,4,5-trimethoxybenzoyl)piperazin-1-yl)methanone (9b)

![9b](image)

LC-MS (ESI, Positive): m/z: [M+H]⁺: 477.4; ¹H NMR (400 MHz, DMSO-d₆): δ 8.13-8.11 (d, J=6.8 Hz, 1H), 7.88-7.85 (dd, J =7.2 Hz, 1H), 7.62-7.55 (m, 2H), 6.72 (s, 2H), 3.79 (s, 6H), 3.67 (s, 3H), 3.56-3.46 (m, 8H); Elemental analysis: Calculated (%) for C₂₃H₂₃ClN₂O₅S: C 58.16, H 4.88, N 5.90, S 6.75; Found: C 58.17, H 4.87, N 5.89, S 6.73.

(4-(2-Amino-4,5-dichlorobenzoyl)piperazin-1-yl)(3,4,5-trimethoxyphenyl)methanone (9c)

![9c](image)

LC-MS (ESI, Positive): m/z: [M+H]⁺: 470.3; ¹H NMR (400 MHz, DMSO-d₆): δ 7.43-7.42 (d, J=2.4 Hz, 1H), 7.09-7.08 (d, J =2.4 Hz, 1H), 6.68 (s, 2H), 5.47 (s, 2H), 3.79 (s, 6H), 3.67 (s, 3H), 3.56-3.46 (m, 8H); Elemental analysis: Calculated (%) for C₂₁H₂₃Cl₂N₃O₅: C 53.86, H 4.95, N 8.97; Found: C 53.87, H 4.96, N 8.96.
(4-(2-Amino-5-hydroxybenzoyl)piperazin-1-yl)(3,4,5-trimethoxyphenyl)methanone (9d)

![Chemical structure of 9d]

**9d**

LC-MS (ESI, Positive): m/z: [M+H]^+: 416.2; \(^1\)H NMR (400 MHz, DMSO-\(d_6\)): \(\delta\) 8.63 (s, 1H), 6.69 (s, 2H), 6.58-6.57 (m, 2H), 6.44 (s, 1H), 4.55 (s, 2H), 3.79 (s, 6H), 3.67 (s, 3H), 3.56-3.46 (m, 8H); Elemental analysis: Calculated (%) for C\(_{21}\)H\(_{25}\)N\(_3\)O\(_6\): C 60.71, H 6.07, N 10.11; Found: C 60.70, H 6.08, N 10.10.

4-(2-Chloronicotinoyl)piperazin-1-yl)(3,4,5-trimethoxyphenyl)methanone (9e)

![Chemical structure of 9e]

**9e**

LC-MS (ESI, Positive): m/z: [M+H]^+: 420.1; \(^1\)H NMR (400 MHz, DMSO-\(d_6\)): \(\delta\) 8.49-8.48 (m, 1H), 7.95-7.90 (m, 1H), 7.56-7.51 (m, 1H), 6.71 (s, 2H), 3.79 (s, 6H), 3.67 (s, 3H), 3.56-3.46 (m, 8H); Elemental analysis: Calculated (%) for C\(_{20}\)H\(_{22}\)ClN\(_3\)O\(_5\): C 57.21, H 5.28, N 10.01; Found: C 57.20, H 5.27, N 10.03.
(4-(3-Fluoro-2-nitrobenzoyl)piperazin-1-yl)(3,4,5-trimethoxyphenyl)methanone (9f)

LC-MS (ESI, Positive): m/z: [M+H]^+ : 448.1; ^1H NMR (400 MHz, DMSO-d_6): δ 7.82-7.75 (m, 1H), 7.70-7.67 (m, 1H), 7.54-7.51 (m, 1H), 6.72 (s, 2H), 3.79 (s, 6H), 3.67 (s, 3H), 3.56-3.46 (m, 8H); Elemental analysis: Calculated (%) for C_{21}H_{22}FN_{3}O_{7}: C 56.37, H 4.96, N 9.39; Found: C, 56.36, H 4.97, N 9.37.

2-(1H-Indol-3-yl)-1-(4-(3,4,5-trimethoxybenzoyl)piperazin-1-yl)ethanone (9g)

LC-MS (ESI, Positive): m/z: [M+H]^+ : 438.2; ^1H NMR (400 MHz, DMSO-d_6): δ 10.89 (s, 1H), 7.57-7.55 (d, J=7.2 Hz, 1H), 7.34-7.32 (d, J=7.2 Hz, 1H), 7.22 (s, 1H), 7.08-7.04 (m, 1H), 6.98-6.94 (m, 1H), 6.67 (s, 2H), 3.79 (s, 6H), 3.67 (s, 3H), 3.56-3.46 (m, 8H); Elemental analysis: Calculated (%) for C_{24}H_{27}N_{3}O_{5}: C 65.89, H 6.22, N 9.60; Found: C 65.90, H 6.23, N 9.61.

(4-(1-(4-Chlorophenyl)cyclopropanecarbonyl)piperazin-1-yl)(3,4,5-trimethoxyphenyl)methanone (9h)
LC-MS (ESI, Positive): m/z: [M+H]^+ : 460.4; ^1H NMR (400 MHz, DMSO-d$_6$): δ 7.38-7.35 (d, J=8.7 Hz, 2H), 7.20-7.17 (d, J=8.7 Hz, 2H), 6.65 (s, 2H), 3.79 (s, 6H), 3.67 (s, 3H), 3.56-3.46 (m, 8H), 1.32-1.17 (m, 4H); IR (KBr) ν(cm$^{-1}$): 1621 (C=O), 1230 (C-O-C); Elemental analysis: Calculated (%) for C$_{24}$H$_{27}$ClN$_2$O$_5$: C 62.81, H 5.93, N 6.10; Found: C 62.80, H 5.92, N 6.11.

(2-tert-Butylpyrimidin-4-yl)(4-(3,4,5-trimethoxybenzoyl)piperazin-1-yl)methanone (9i)

![9i](image)

LC-MS (ESI, Positive): m/z: [M+H]^+ : 444.6; ^1H NMR (400 MHz, DMSO-d$_6$): δ 8.93-8.92 (d, J=5.2 Hz, 1H) 7.95 (s, 1H), 7.49-7.48 (d, J=5.2 Hz, 1H), 6.73 (s, 2H), 3.79 (s, 6H), 3.67 (s, 3H), 3.56-3.46 (m, 8H), 1.35 (s, 9H); Elemental analysis: Calculated (%) for C$_{23}$H$_{30}$N$_4$O$_5$: C 62.43, H 6.83, N 12.66; Found: C 62.44, H 6.84 N 12.63.

(4-(4-Chlorobenzoyl)piperazin-1-yl)(3,4,5-trimethoxyphenyl)methanone (9j)

![9j](image)

LC-MS (ESI, Positive): m/z: [M+H]^+ : 421.0; ^1H NMR (400 MHz, DMSO-d$_6$): δ 7.54-7.51 (d, J=8.4 Hz, 2H), 7.46-7.43 (d, J=8.4 Hz, 2H), 6.70 (s, 2H), 3.79 (s, 6H), 3.67 (s, 3H), 3.56-3.46 (m, 8H); Elemental analysis: Calculated (%) for C$_{21}$H$_{23}$ClN$_2$O$_5$: C 60.22, H 5.53, N 6.69; Found: C 60.23, H 5.54, N 6.70.
(4-Benzoyl)piperazin-1-yl)(3,4,5-trimethoxyphenyl)methanone (9k)

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LC-MS (ESI, Positive): \(m/z: [M+H]^+\): 385.4; \(^1\)H NMR (400 MHz, DMSO-\(d_6\)): \(\delta\) 7.53-7.51 (m, 2H) 7.46-7.33 (m, 3H), 6.69 (s, 2H), 3.78 (s, 6H), 3.70 (s, 3H), 3.55-3.54 (m, 8H); Elemental analysis: Calculated (%) for C\(_{21}\)H\(_{24}\)N\(_2\)O\(_5\): C 65.61, H 6.29, N 7.29; Found: C 65.63, H 6.24, N 7.30.

(4-(4-Fluorobenzoyl)piperazin-1-yl)(3,4,5-trimethoxyphenyl)methanone (9l)

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LC-MS (ESI, Positive): \(m/z: [M+H]^+\): 403.4; \(^1\)H NMR (400 MHz, DMSO-\(d_6\)): \(\delta\) 7.34-7.31 (d, 2H), 7.26-7.23 (d, 2H), 6.40 (s, 2H), 3.59 (s, 6H), 3.47 (s, 3H), 3.36-3.26 (m, 8H); Elemental analysis: Calculated (%) for C\(_{21}\)H\(_{23}\)FN\(_2\)O\(_5\): C 62.68, H 5.76, N 6.96; Found: C 65.63, H 5.75, N 6.90.

(4-(2-Chlorobenzoyl)piperazin-1-yl)(3,4,5-trimethoxyphenyl)methanone (9m)

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(4-(4-Bromobenzoyl)piperazin-1-yl)(3,4,5-trimethoxyphenyl)methanone(9n)

LC-MS (ESI, Positive): m/z: [M+H]^+: 464.4; ^1^H NMR (400 MHz, DMSO-d$_6$): δ 7.74-7.71 (d, 2H) 7.66-7.63 (d, 2H), 6.50 (s, 2H), 3.99 (s, 6H) 3.87 (s, 3H), 3.76-3.66 (m, 8H); Elemental analysis: Calculated (%) for C$_{21}$H$_{23}$BrN$_2$O$_5$: C 54.44, H 5.00, N 6.05; Found: C 54.43, H 5.02, N 6.07.

(4-(4-Methoxybenzoyl)piperazin-1-yl)(3,4,5-trimethoxyphenyl)methanone(9o)

LC-MS (ESI, Positive): m/z: [M+H]^+: 415.4; ^1^H NMR (400 MHz, DMSO-d$_6$): δ 7.34-7.31 (m, 2H) 7.20-7.17 (m, 2H), 6.60 (s, 2H), 3.79 (s, 6H), 3.67 (s, 6H), 3.46-3.36 (m, 8H); Elemental analysis: Calculated (%) for C$_{22}$H$_{26}$N$_2$O$_6$: C 63.76, H 6.32, N 6.76; Found: C 63.75, H 6.35, N 6.75.
(4-(4-Methylbenzoyl)piperazin-1-yl)(3,4,5-trimethoxyphenyl)methanone (9p)

LC-MS (ESI, Positive): m/z: [M+H]$^+$: 399.4; $^1$H NMR (400 MHz, DMSO-$d_6$): $\delta$
7.54-7.51 (m, 2H), 7.40-7.37 (m, 2H), 6.65 (s, 2H), 3.79 (s, 6H), 3.66 (s, 3H), 3.46-
3.36 (m, 8H), 3.25 (s, 3H); Elemental analysis: Calculated (%) for C$_{22}$H$_{26}$N$_2$O$_5$: C
66.32, H 6.58, N 7.03; Found: C 66.35, H 6.55, N 7.05.

(4-(4-Hydroxybenzoyl)piperazin-1-yl)(3,4,5-trimethoxyphenyl)methanone (9q)

LC-MS (ESI, Positive): m/z: [M+H]$^+$: 401.5; $^1$H NMR (400 MHz, DMSO-$d_6$): $\delta$
7.36-7.34 (m, 2H), 7.05-7.01 (m, 2H), 6.58 (s, 2H), 5.12 (s, 1H), 3.79 (s, 6H), 3.66
(s, 3H), 3.46-3.36 (m, 8H); Elemental analysis: Calculated (%) for C$_{21}$H$_{24}$N$_2$O$_6$: C
62.99, H 6.04, N 7.00; Found: C 62.95, H 6.05, N 7.02.
(4-(3,5-Dihydroxybenzoyl)piperazin-1-yl)(3,4,5-trimethoxyphenyl)methanone(9r)

![Chemical Structure 9r](image.png)

LC-MS (ESI, Positive): m/z: [M+H]$^+$: 417.5; $^1$H NMR (400 MHz, DMSO-$d_6$): δ 7.01 (s, 2H), 6.60 (s, 2H), 6.35 (s, 1H), 5.22 (s, 2H), 3.79 (s, 6H), 3.66 (s, 3H), 3.46-3.36 (m, 8H); Elemental analysis: Calculated (%) for C$_{21}$H$_{24}$N$_2$O$_7$: C 60.57, H 5.81, N 6.73; Found: C 60.55, H 5.83, N 6.72.

(4-(3,4,5-Trihydroxybenzoyl)piperazin-1-yl)(3,4,5-trimethoxyphenyl)methanone(9s)

![Chemical Structure 9s](image.png)

LC-MS (ESI, Positive): m/z: [M+H]$^+$: 433.5; $^1$H NMR (400 MHz, DMSO-$d_6$): δ 6.98 (s, 2H), 6.56 (s, 2H), 5.23-5.22 (m, 3H), 3.79 (s, 6H), 3.66 (s, 3H), 3.46-3.36 (m, 8H); Elemental analysis: Calculated (%) for C$_{21}$H$_{24}$N$_2$O$_8$: C 58.33, H 5.59, N 6.48; Found: C 58.35, H 5.60, N 6.50.

(4-(3,5-Dimethylbenzoyl)piperazin-1-yl)(3,4,5-trimethoxyphenyl)methanone(9t)

![Chemical Structure 9t](image.png)
LC-MS (ESI, Positive): m/z: [M+H]^+: 413.5; ^1H NMR (400 MHz, DMSO-d$_6$): δ 7.55-7.53 (m, 2H), 7.30-7.28 (m, 1H), 6.65 (s, 2H), 3.79 (s, 6H), 3.66 (s, 3H), 3.46-3.36 (m, 8H), 3.27 (s, 6H); Elemental analysis: Calculated (%) for C$_{23}$H$_{28}$N$_2$O$_5$: C 66.97, H 6.84, N 6.79; Found: C 66.95, H 6.85, N 6.75.
REFERENCES


Conference on Antimicrobial Agents and Chemotherapy, San Francisco, September 1995, F-208, p149;


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**Chemical Structure**

![Chemical Structure Image]

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**Match Plot**

![Match Plot Image]
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<td>58.10</td>
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**Mean value**

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<th>N[%]</th>
<th>S[%]</th>
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<td>5.57</td>
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**Deviation, abs.**

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<td>0.03</td>
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**Delta[\%]**

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</tr>
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<td>0.06</td>
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<td>0.010</td>
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Name: eassuperuser,  
Access: VarioMICRO superuser  
11/25/2009  
1:15:55 PM  

VarioMICRO V1.7.0  
11/25/2009, CHNS Mode, Ser. No.: 15084090  
Elementar Analysensysteme GmbH
SAMPLE INFORMATION

Sample Name: 98987-059-10
Sample Type: Unknown
Vial: 1-A-7
Injection #: 1
Injection Volume: 2.00 ul
Run Time: 5.0 Minutes
Date Acquired: 9/13/2013 12:21:23 PM IST
Date Processed: 9/13/2013 3:52:00 PM IST

Acquired By: jaya
Sample Set Name: 130923013
Aqc. Method Set: uplc_ms
Processing Method: 1
Channel Name: MS 1/2
Proc. Chnl. Descri.: SC 1: MS Scan MS TIC

Match Plot

Retention Time: 2.11

Project Name: Sep2013_New
Date Printed: 3/13/2014 3:55:52 PM Arial Calibri
**SAMPLE INFORMATION**

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**MS REPORT**

![MS Spectrum and Mass Plot]

**Project Name:** Sep2013_New
**Date Printed:** 9/19/2013
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