

Supplementary Material

Green synthesis of octahydroquinazolinone and hexahydroquinazolinone derivatives in aqueous medium catalyzed by nano bismuth oxide under mild conditions

Sanjog G. Mumbaikar and Vishvanath D. Patil*

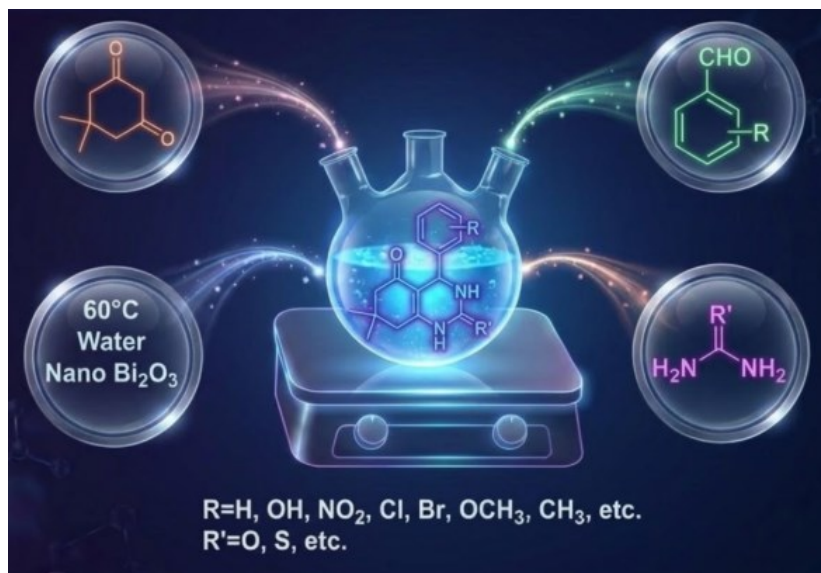
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New Panvel, Raigad, Maharashtra, India
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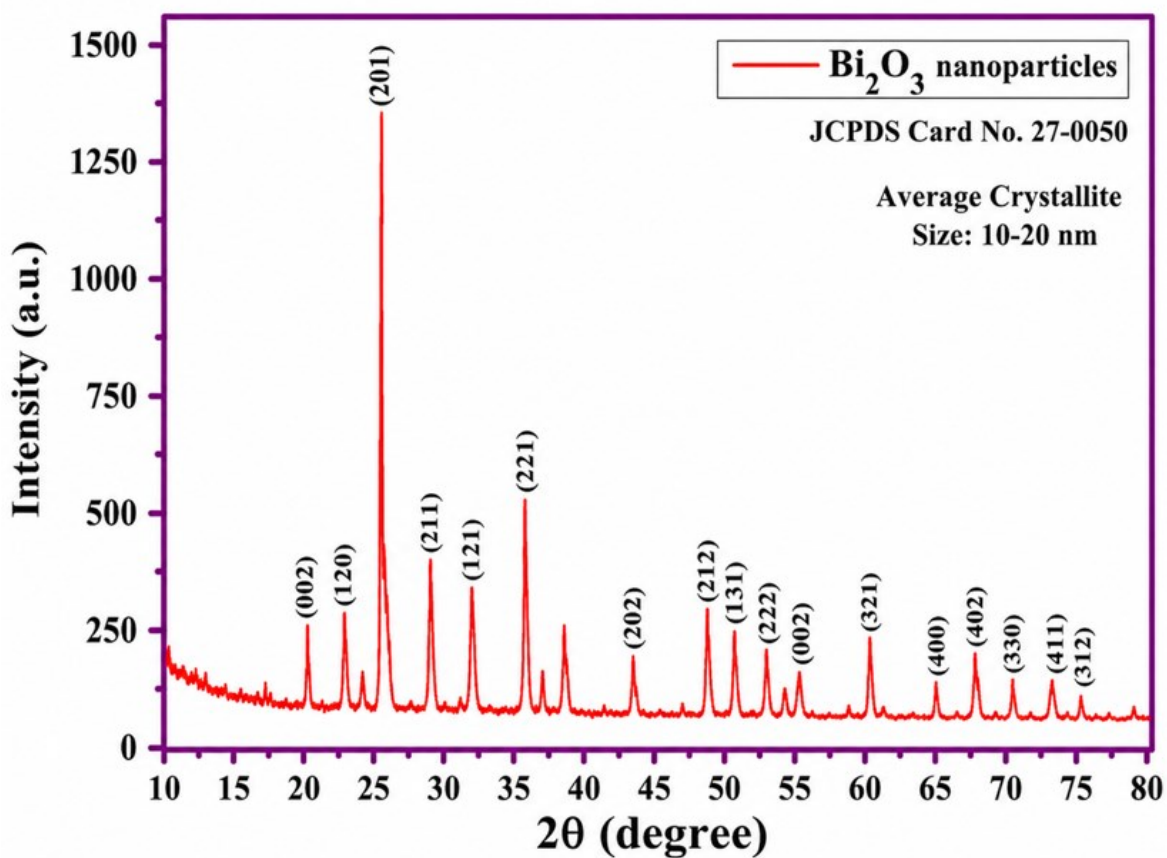
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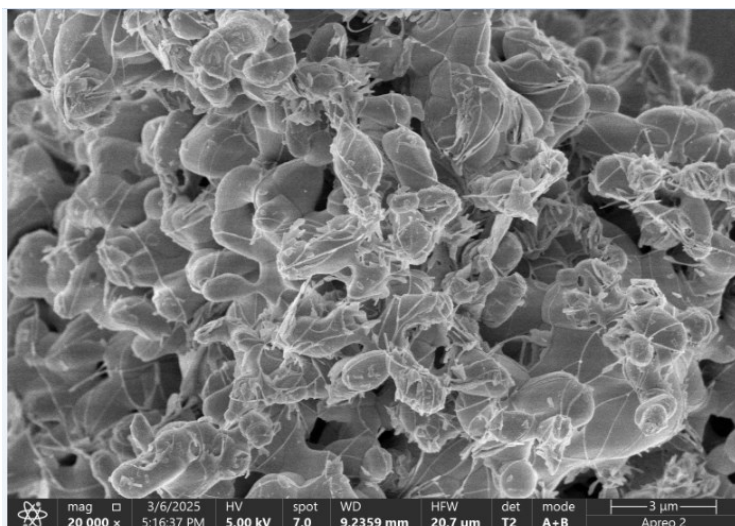
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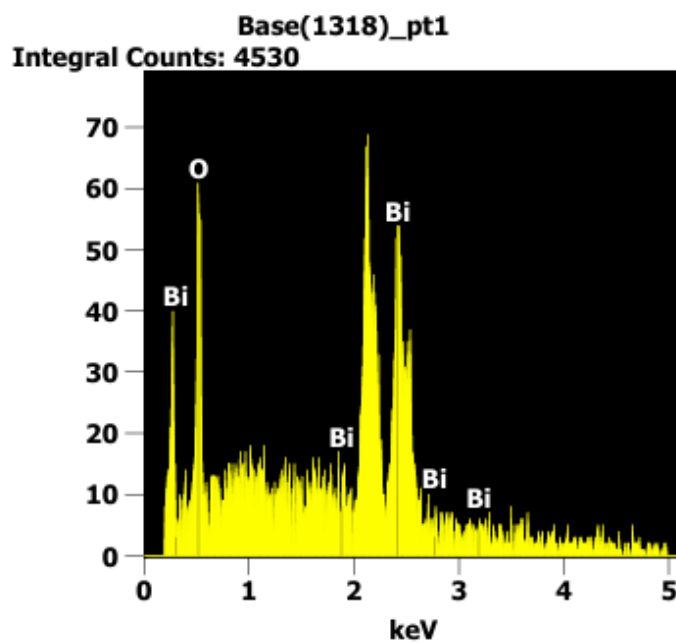
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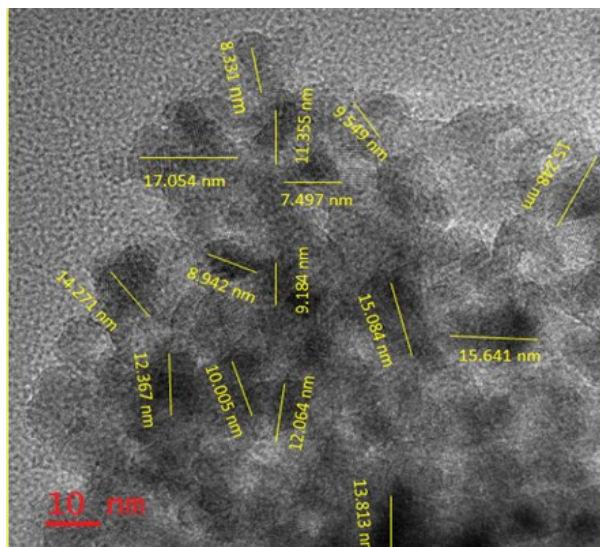
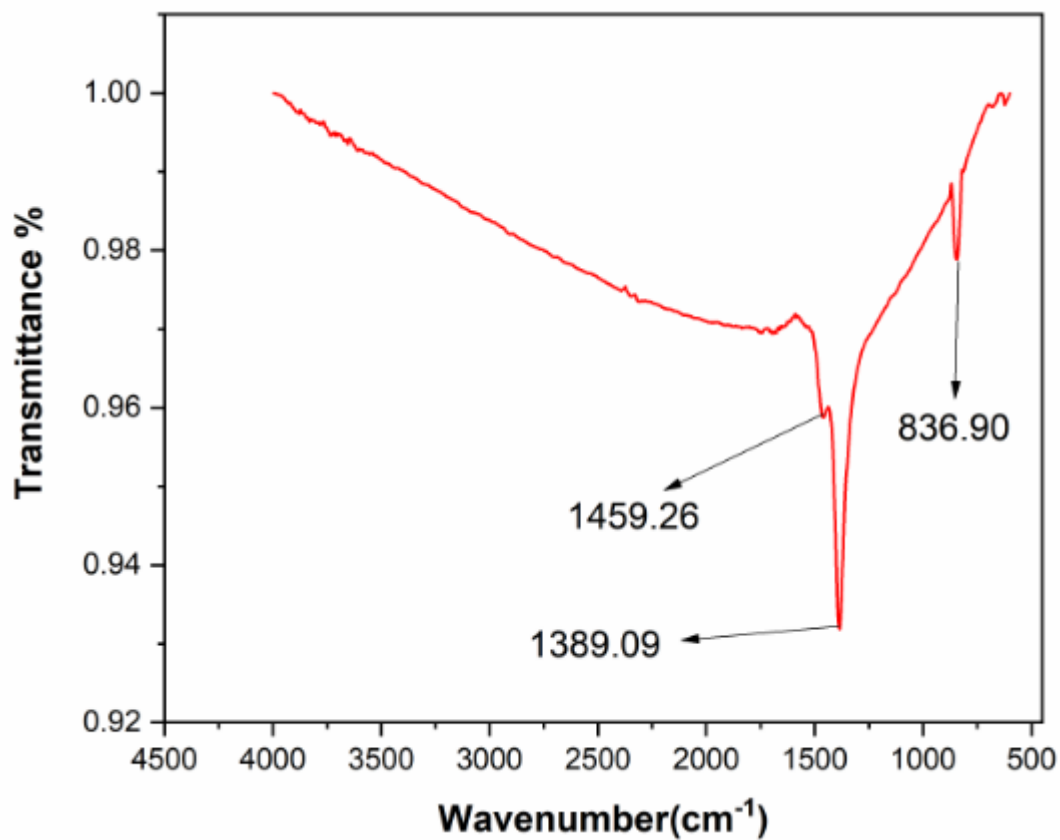


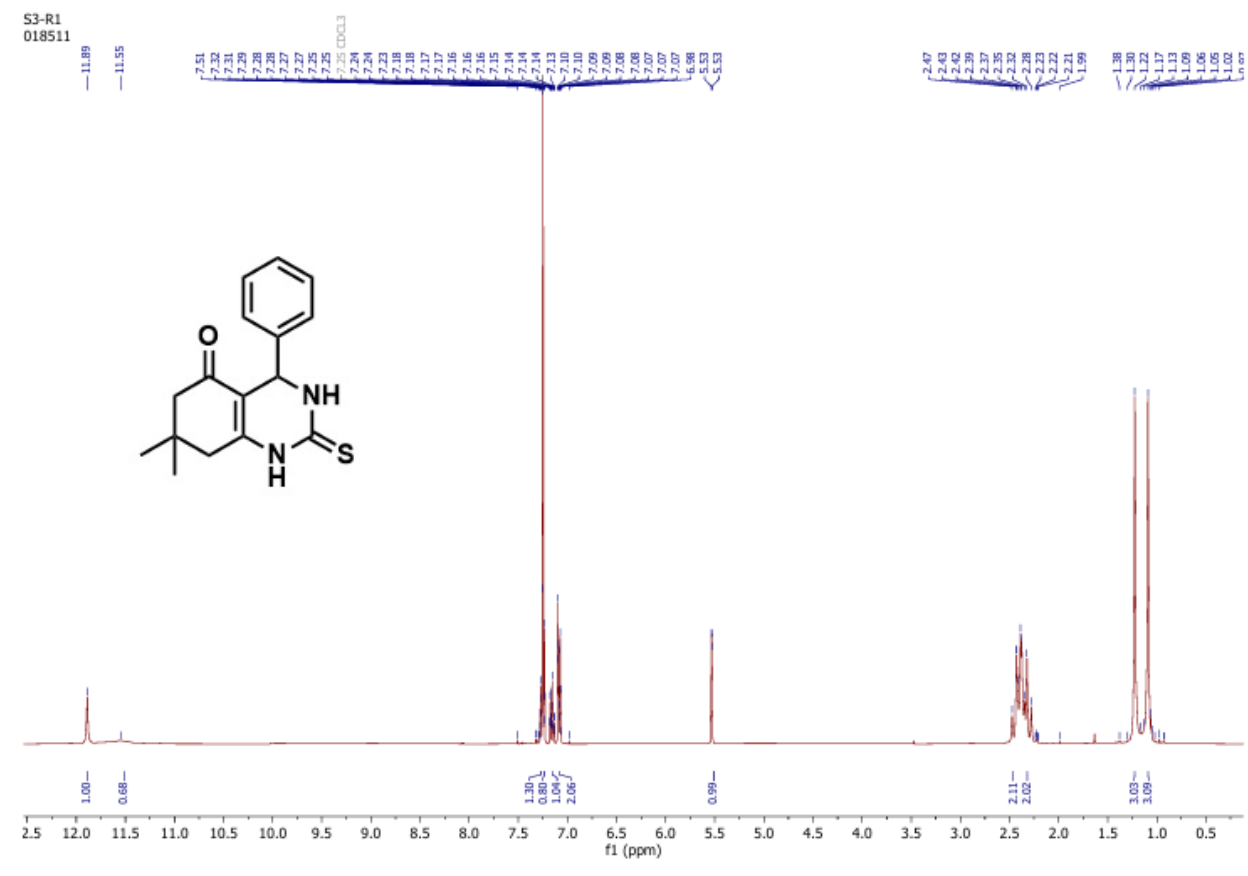
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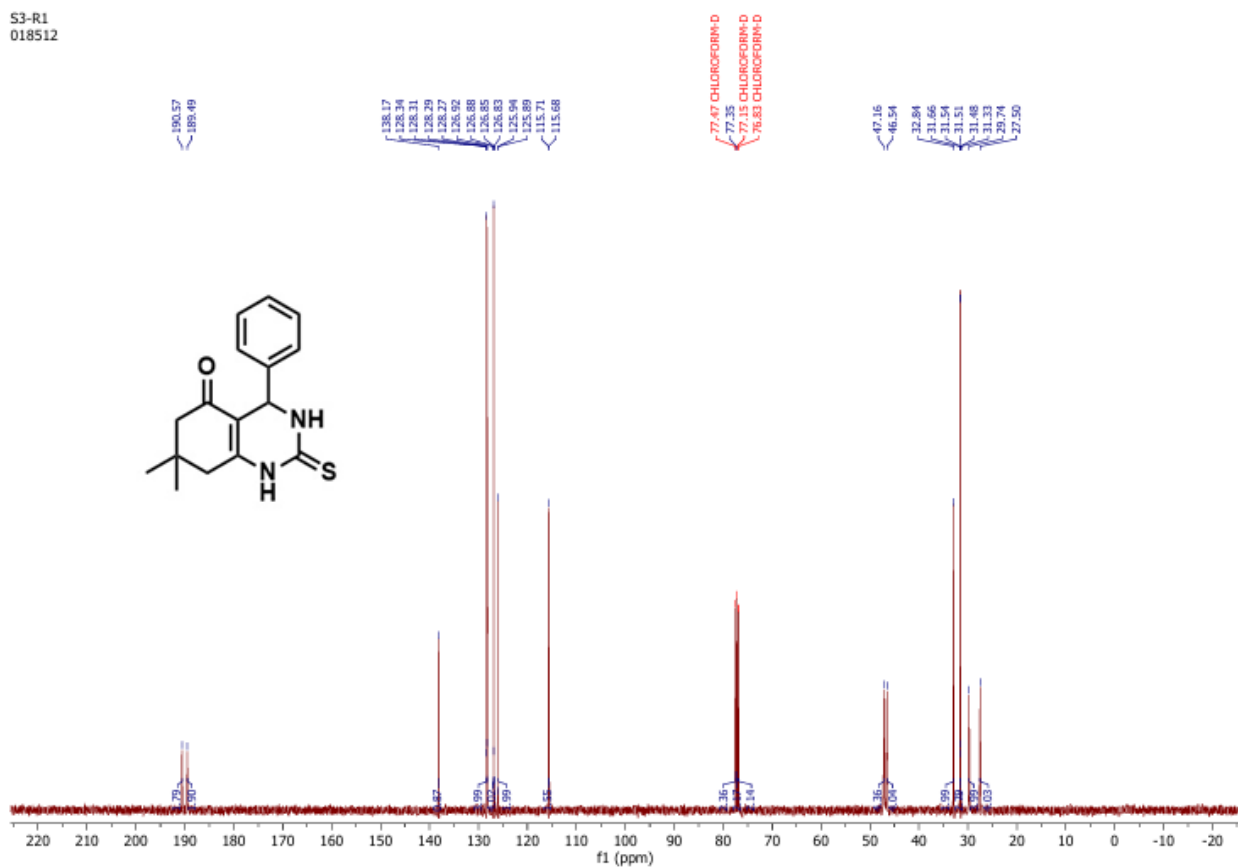


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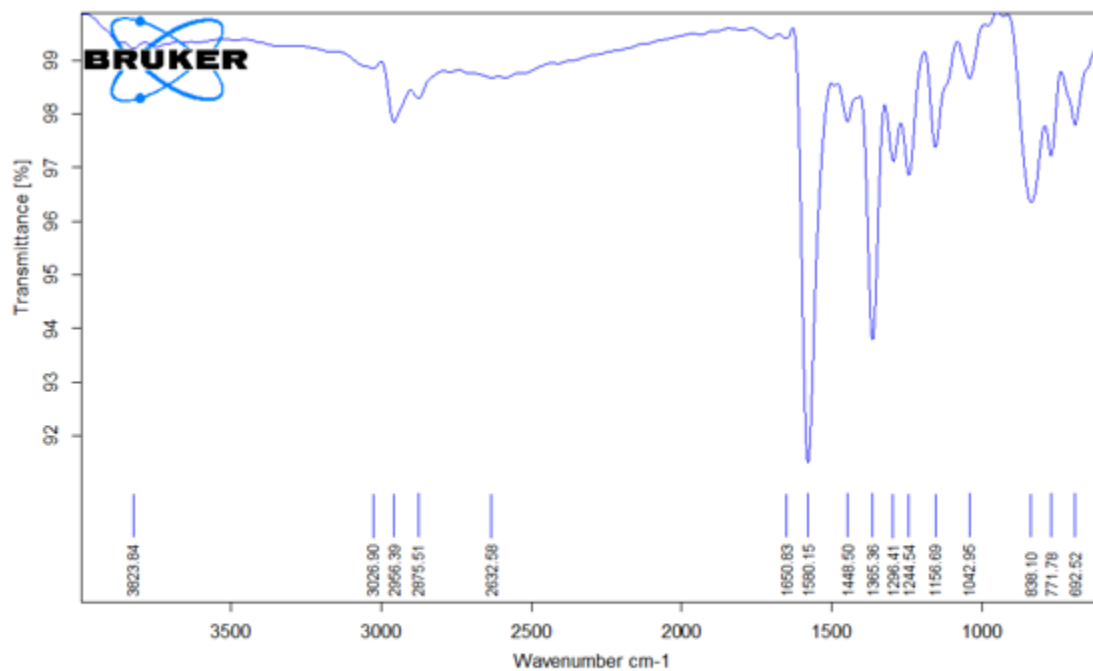


5. TEM of Nano Bi_2O_3 :-**6. IR of Nano Bi_2O_3 :-**

7. ^1H NMR of 1,2,3,4,7,8-hexahydro-7,7-dimethyl-4-phenyl-2-thioxoquinazoline-5-(6H)-one:-

8. ^{13}C NMR of 1,2,3,4,7,8-hexahydro-7,7-dimethyl-4-phenyl-2-thioxoquinazoline-5-(6H)-one:-S3-R1
018512

9. IR of 1,2,3,4,7,8-hexahydro-7,7-dimethyl-4-phenyl-2-thioxoquinazoline-5-(6H)-one:-

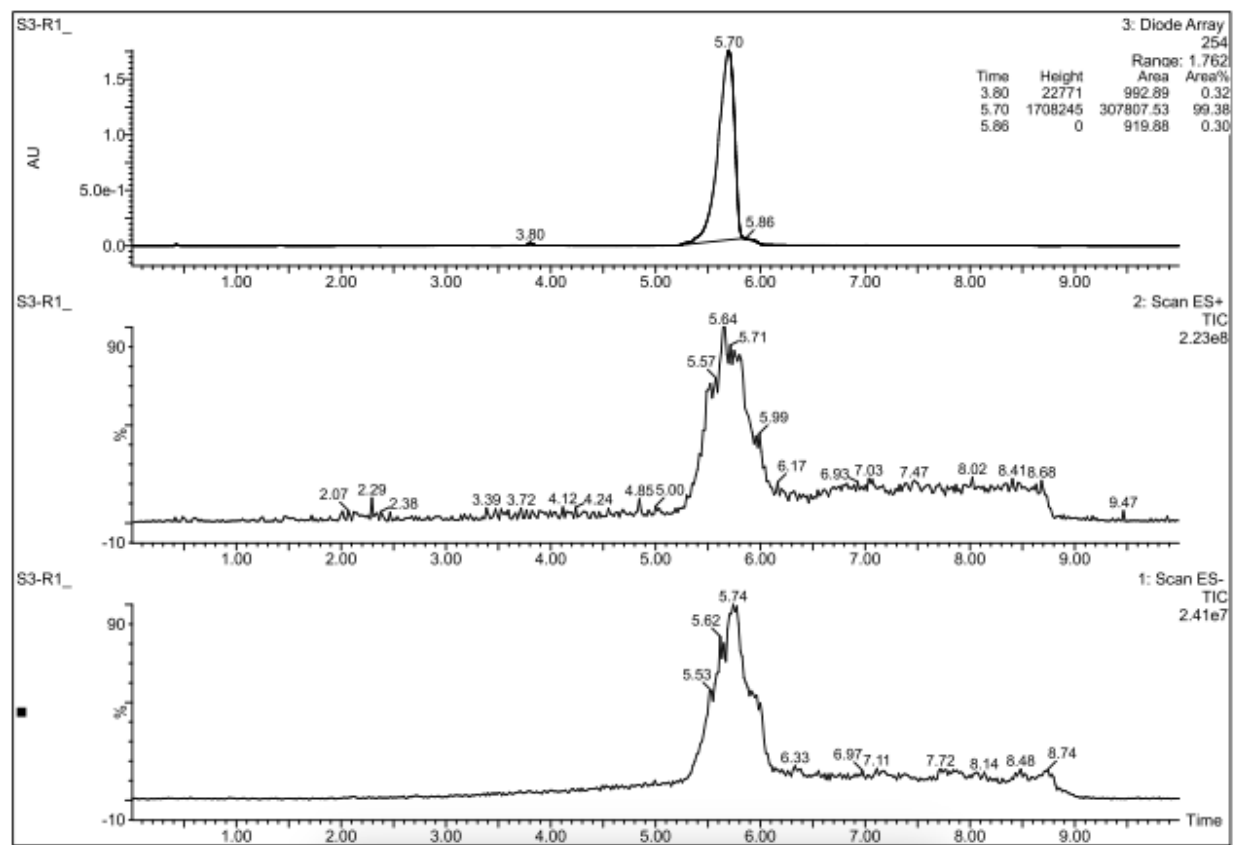


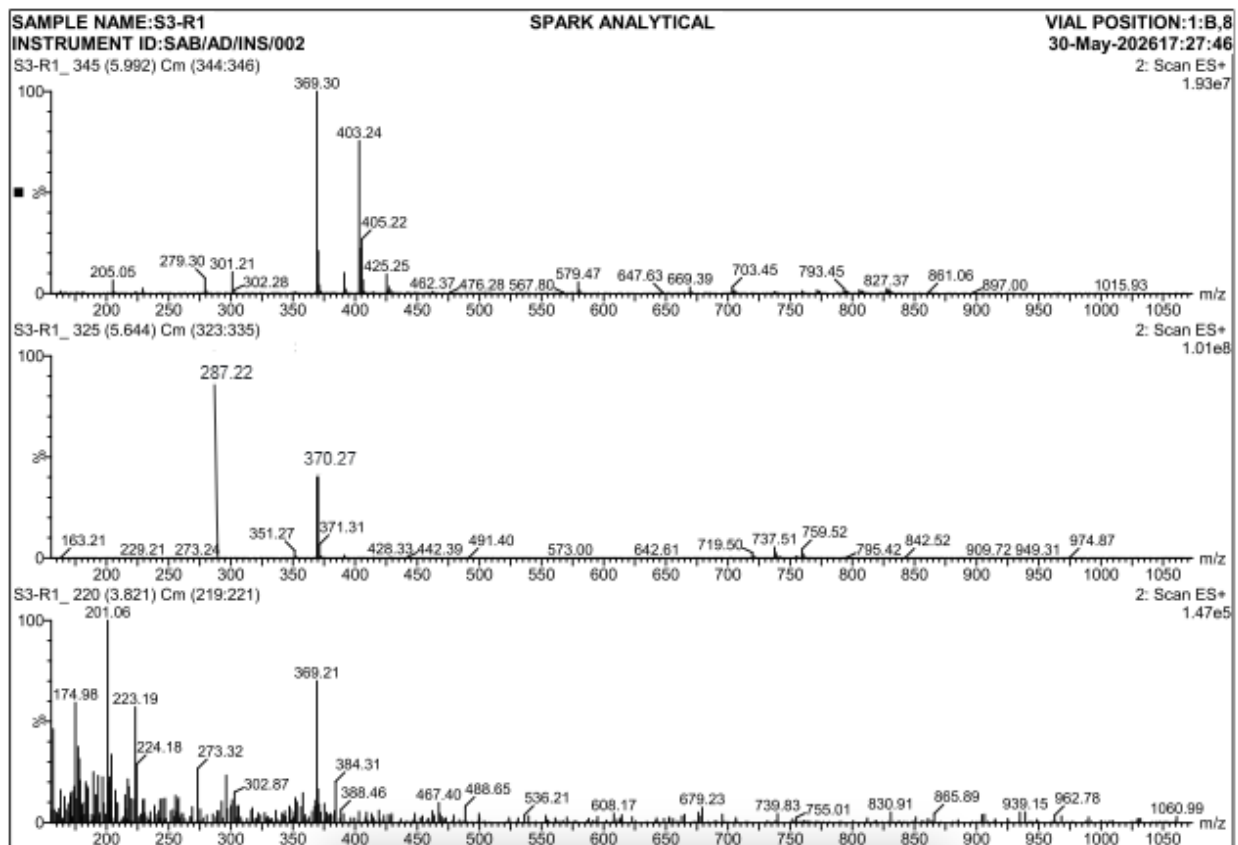
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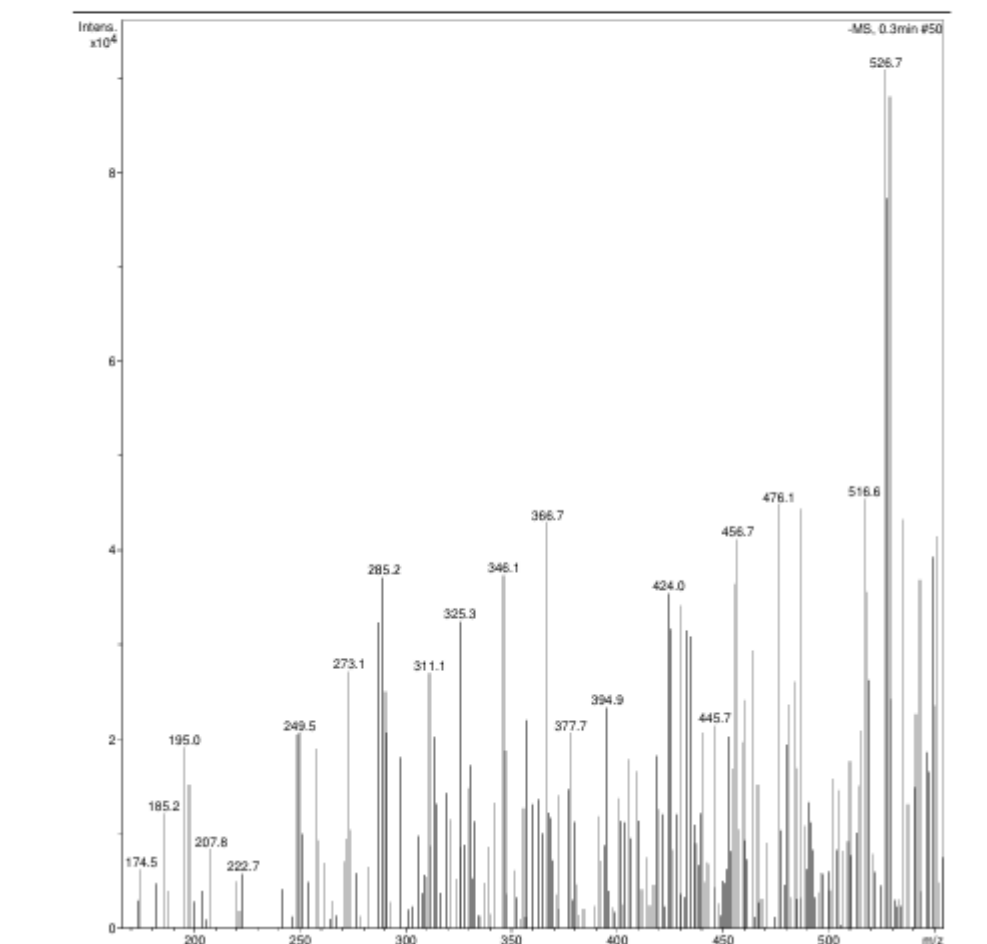
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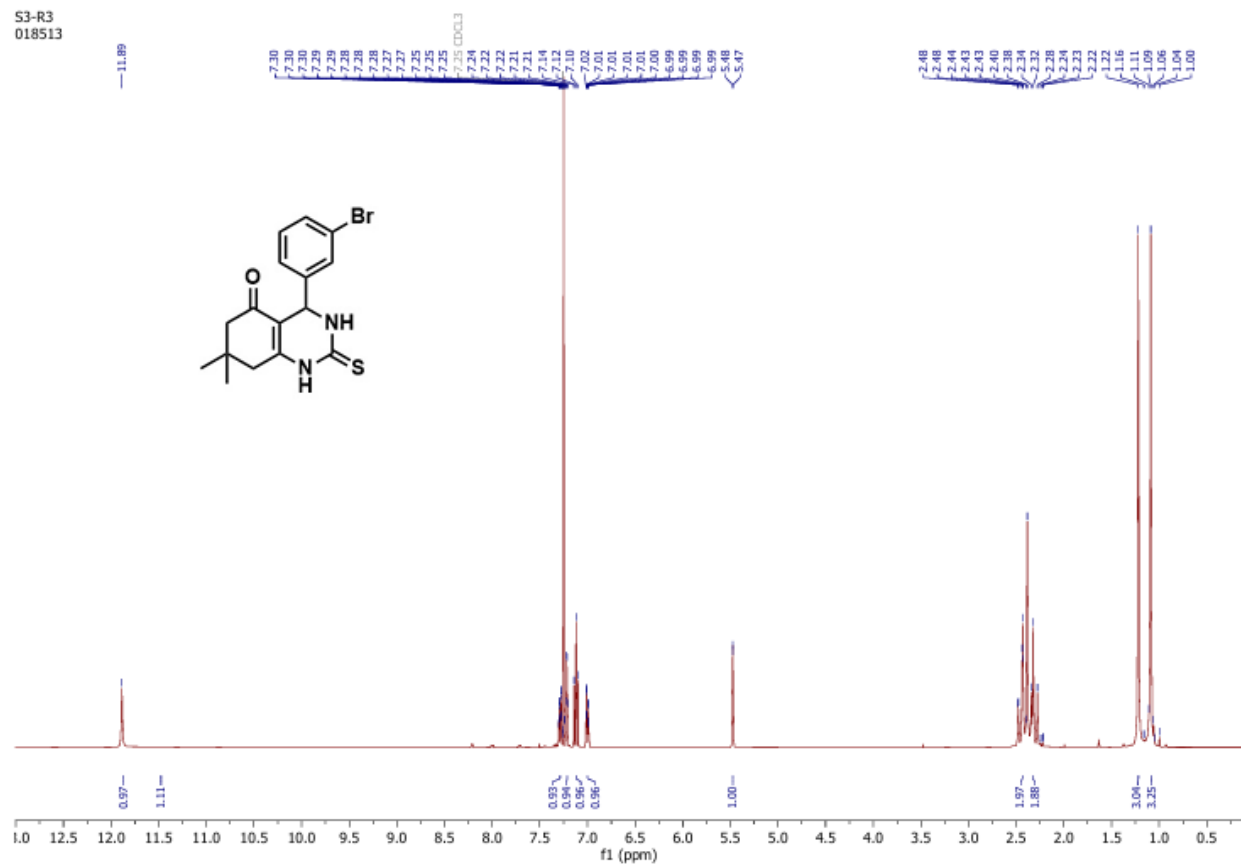
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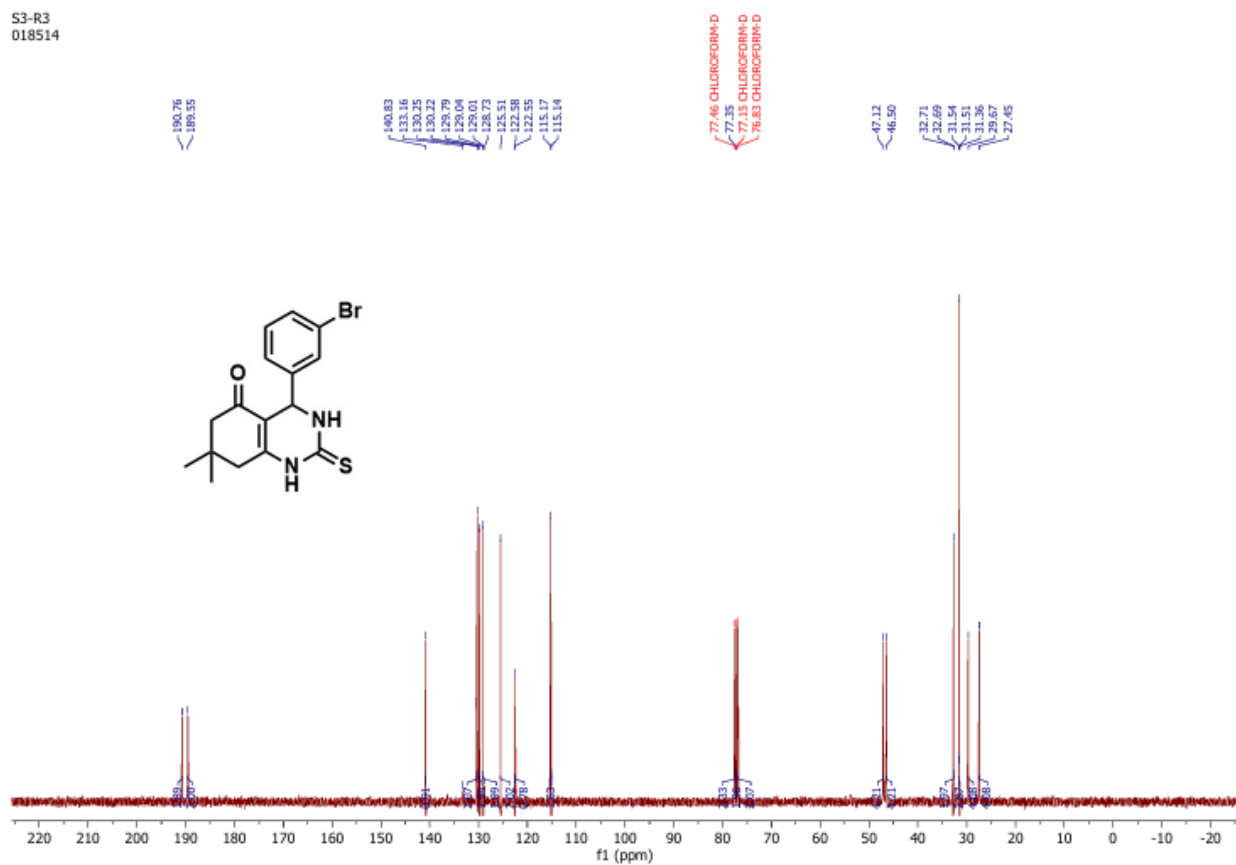
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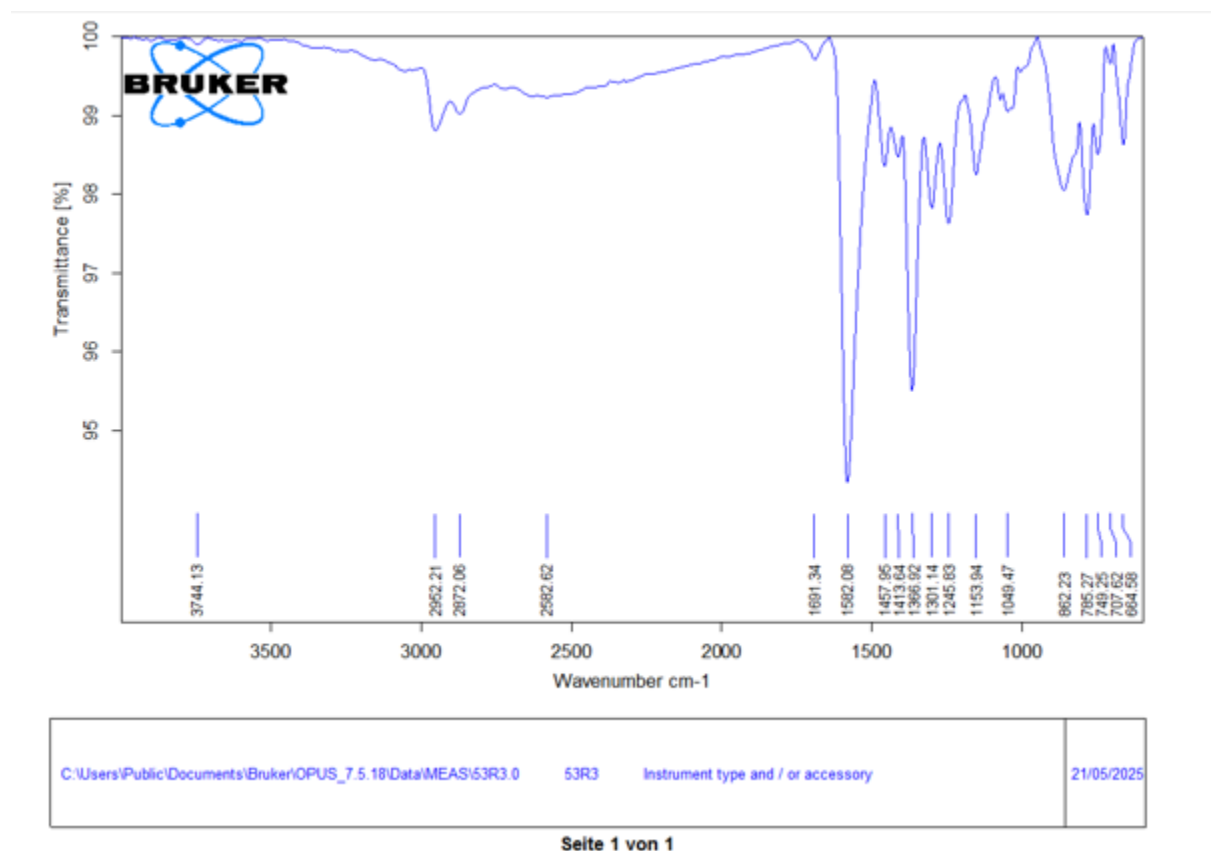
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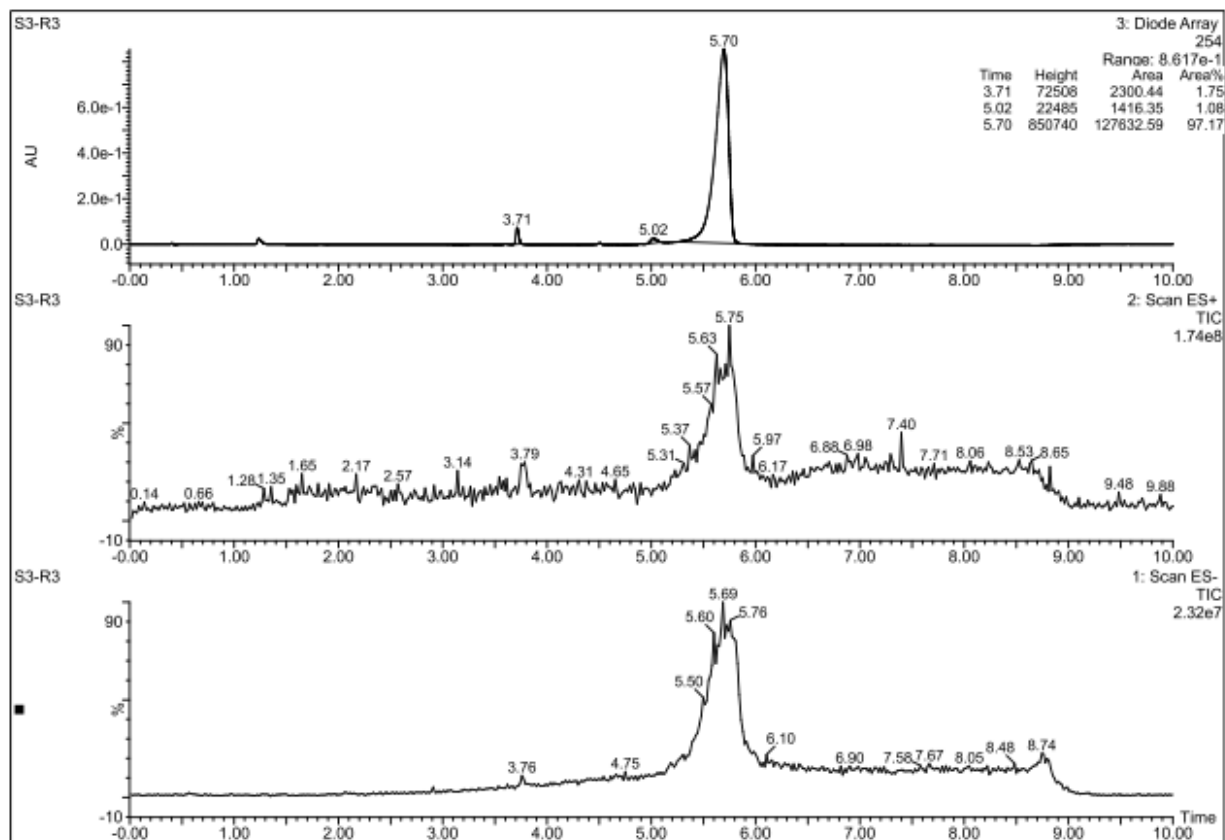


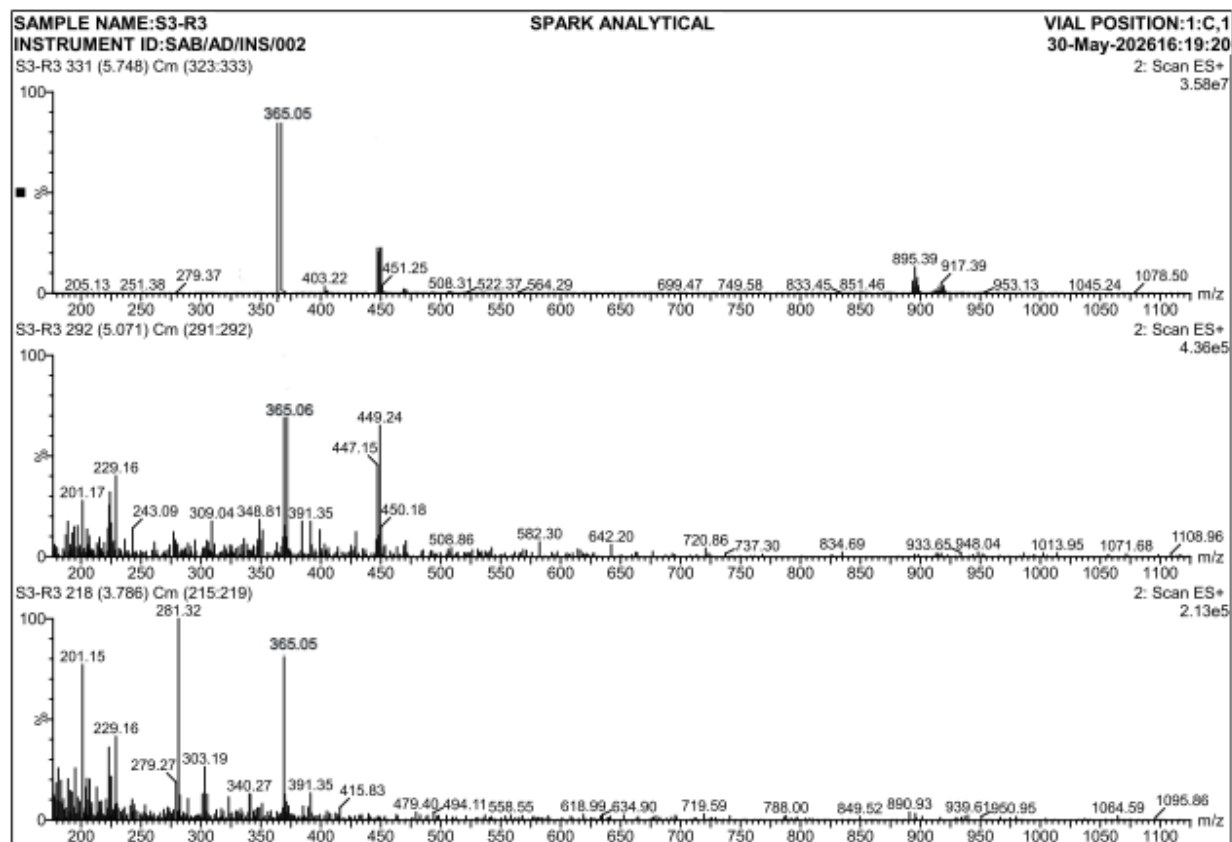
11. Mass of 1,2,3,4,7,8-hexahydro-7,7-dimethyl-4-phenyl-2-thioxoquinazoline-5-(6H)-one:-**CHROMEIN,Pune****Sample Name:** S3_R1**Method:** TEST_LCMS NEG_120-600.M**File Name:** S3_R1_NEG_050525_14.D**Acq. Date:** 5/5/2025 4:27:34 PM**Instrument:** Agilent 6320 Ion Trap**Vial No.**

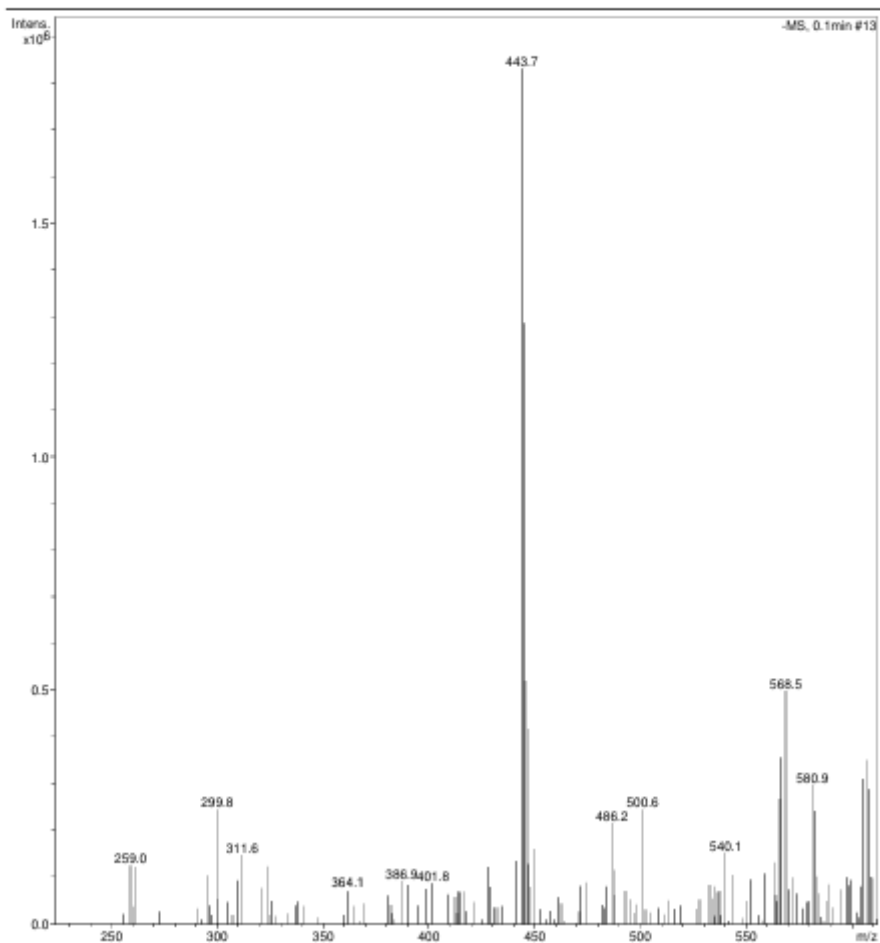
12. ^1H NMR of 4-(3-Bromophenyl)-1,2,3,4,7,8-hexahydro-7,7-dimethyl-2-thioxoquinazoline-5-(6H)-one:-

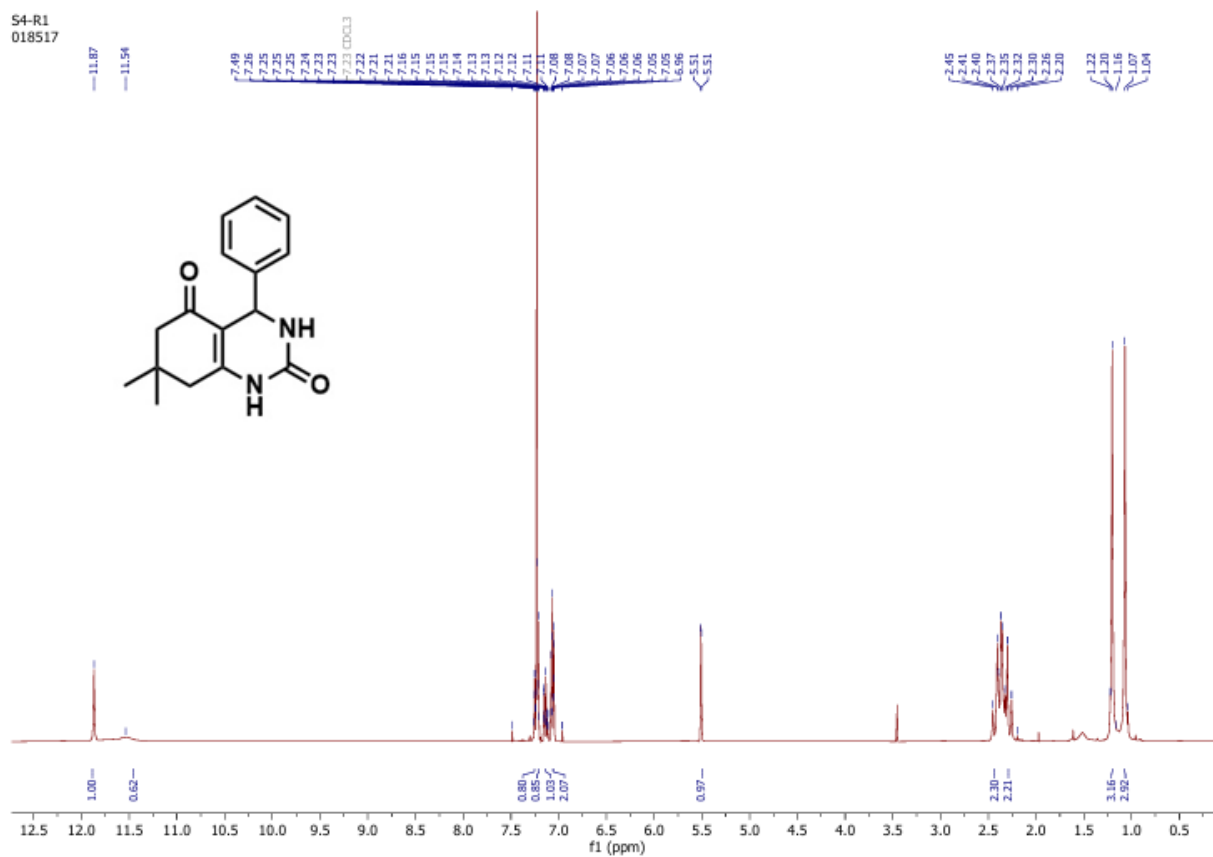
13. ^{13}C NMR of 4-(3-Bromophenyl)-1,2,3,4,7,8-hexahydro-7,7-dimethyl-2-thioxoquinazoline-5-(6H)-one:-S3-R3
018514

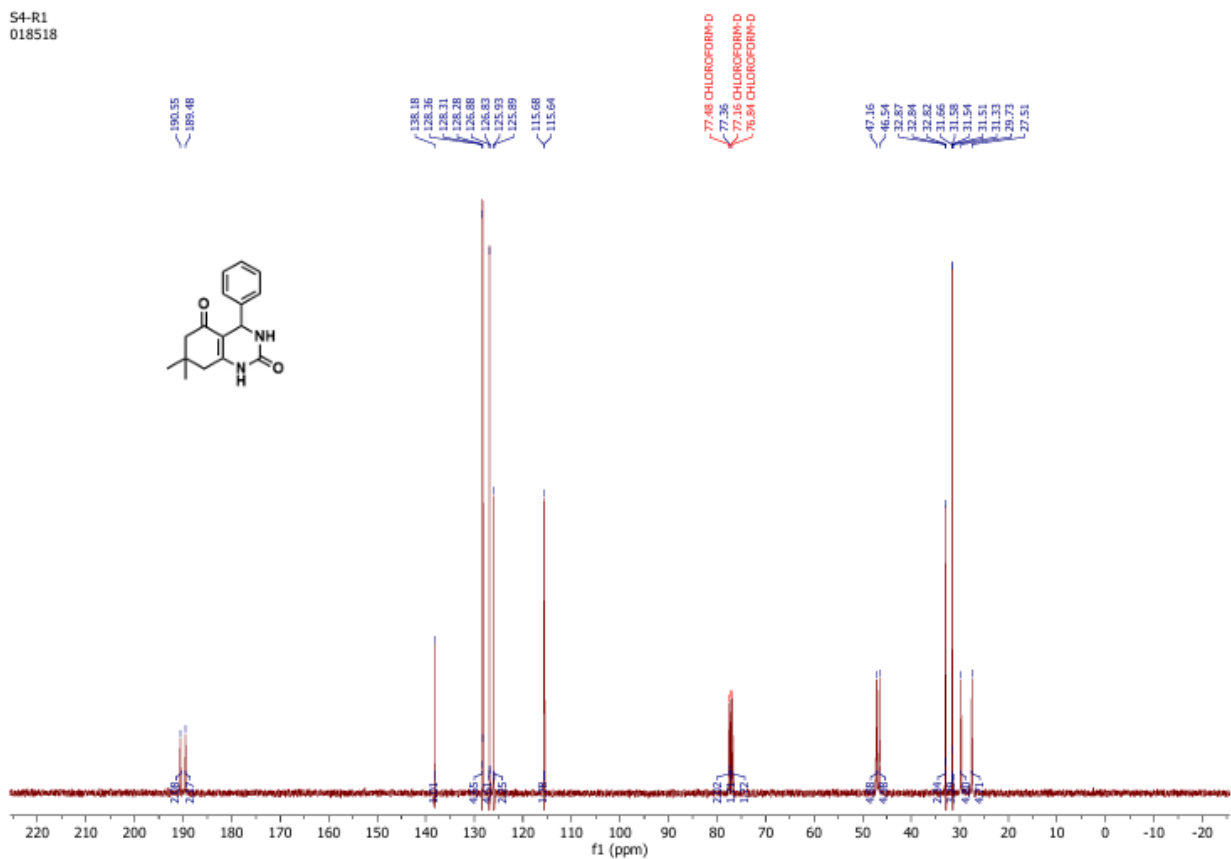
14. IR of 4-(3-Bromophenyl)-1,2,3,4,7,8-hexahydro-7,7-dimethyl-2-thioxoquinazoline-5-(6H)-one:-

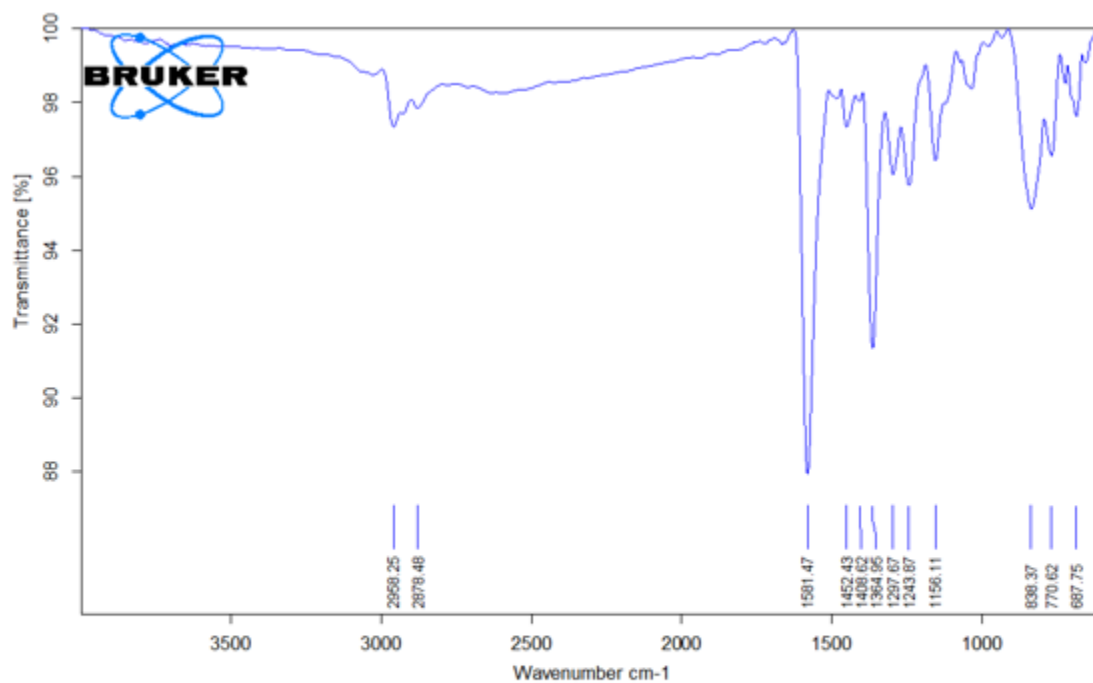
15. LC-MS of 4-(3-Bromophenyl)-1,2,3,4,7,8-hexahydro-7,7-dimethyl-2-thioxoquinazoline-5-(6H)-one:-



16. Mass of 4-(3-Bromophenyl)-1,2,3,4,7,8-hexahydro-7,7-dimethyl-2-thioxoquinazoline-5-(6H)-one:-**CHROMEIN,Pune****Sample Name:** S3_R3**Acq. Date:** 5/5/2025 4:29:04 PM**Method:** TEST_LCMS NEG_120-600.M**Instrument:** Agilent 6320 Ion Trap**File Name:** S3_R3_NEG_050525_15.D**Vial No.**

17. ^1H NMR of 3,4,7,8-tetrahydro-7,7-dimethyl-4-phenylquinazoline-2,5-(1H,6H)-dione

18. ^{13}C NMR of 3,4,7,8-tetrahydro-7,7-dimethyl-4-phenylquinazoline-2,5-(1H,6H)-dione:-S4-R1
018518

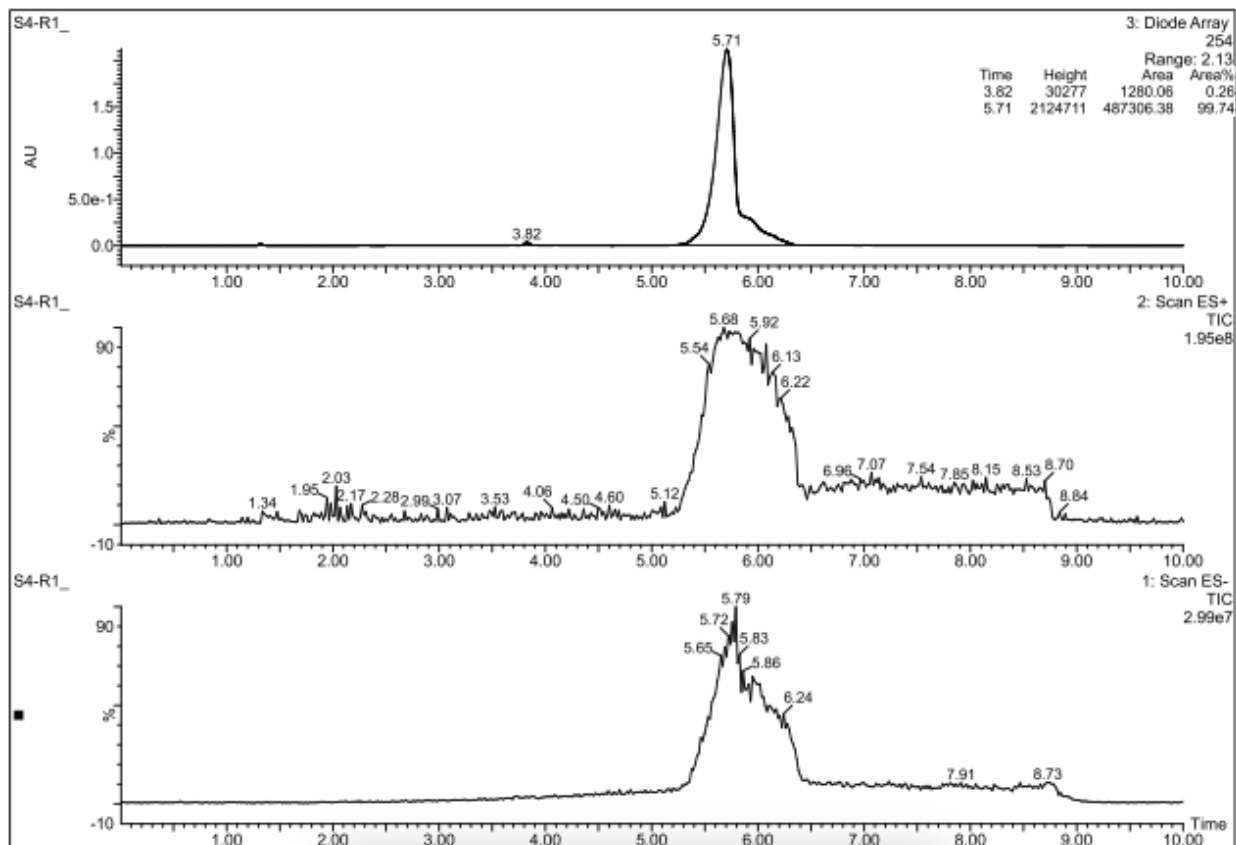
19. IR of 3,4,7,8-tetrahydro-7,7-dimethyl-4-phenylquinazoline-2,5-(1H,6H)-dione:-

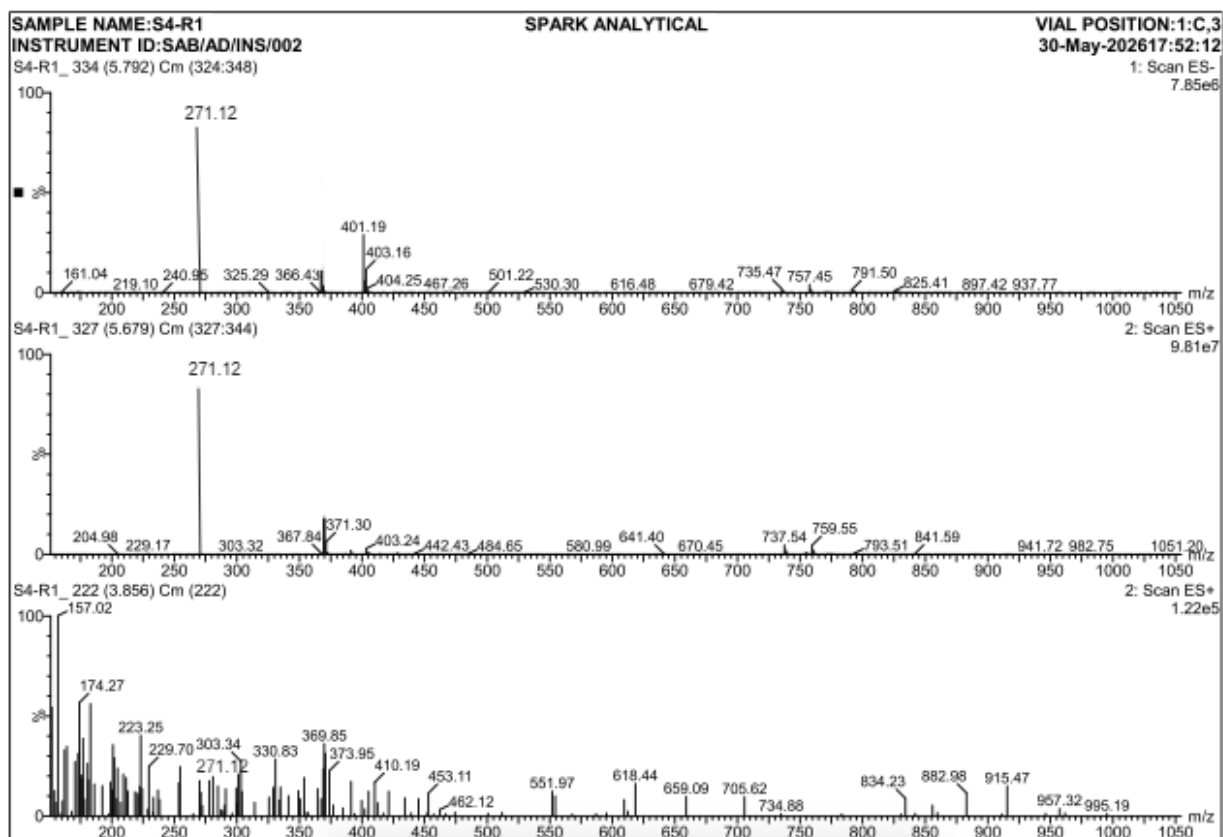
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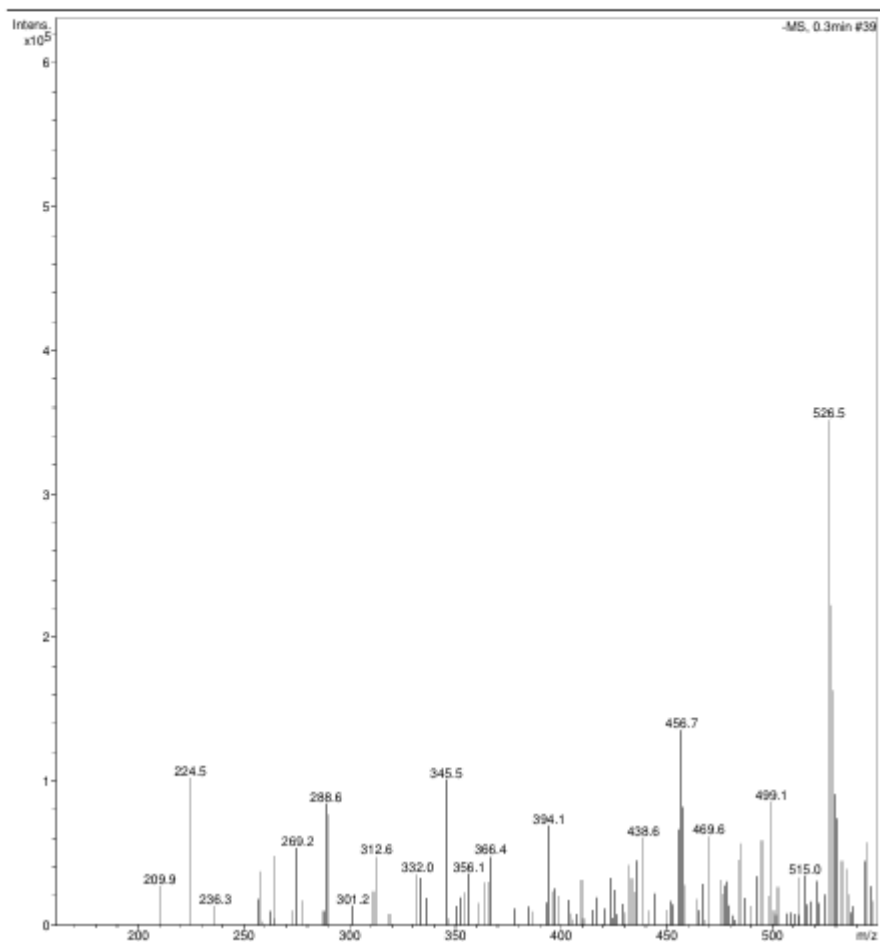
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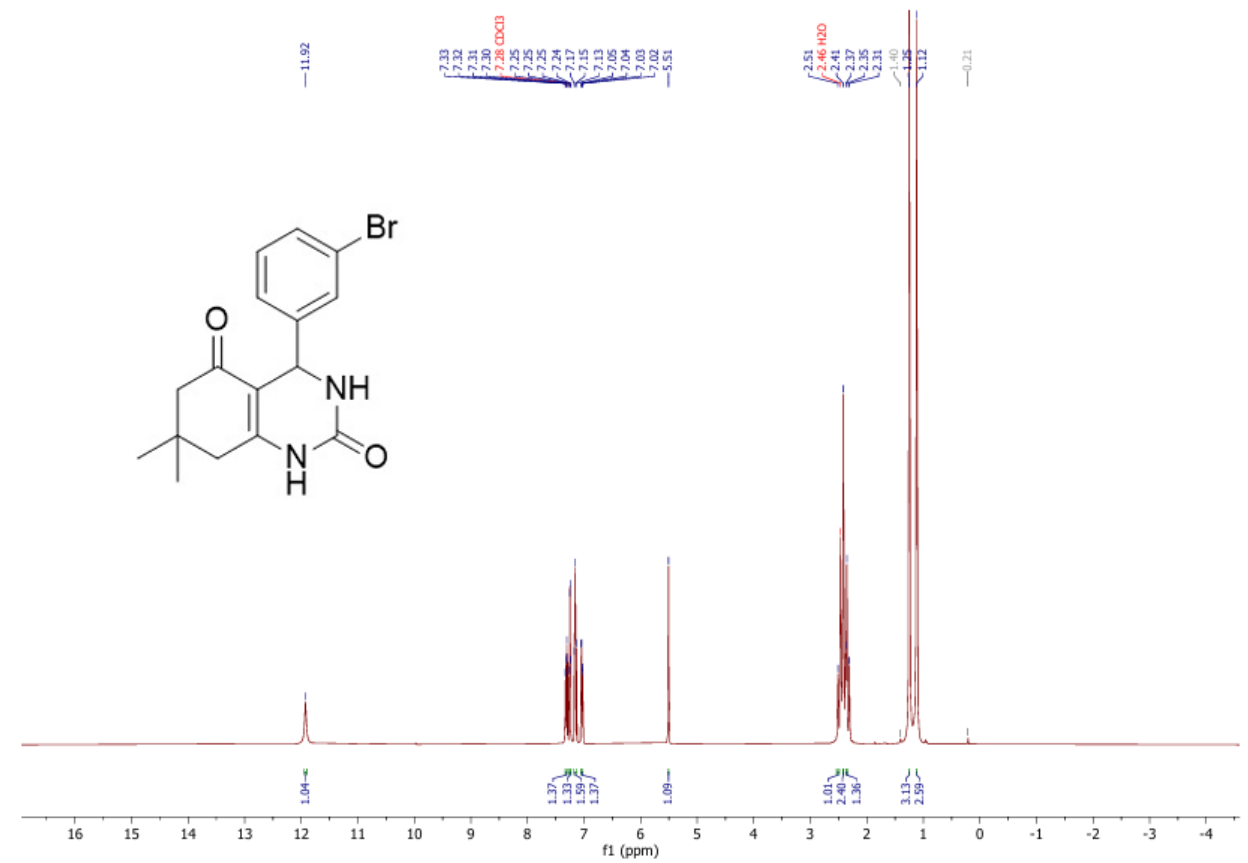
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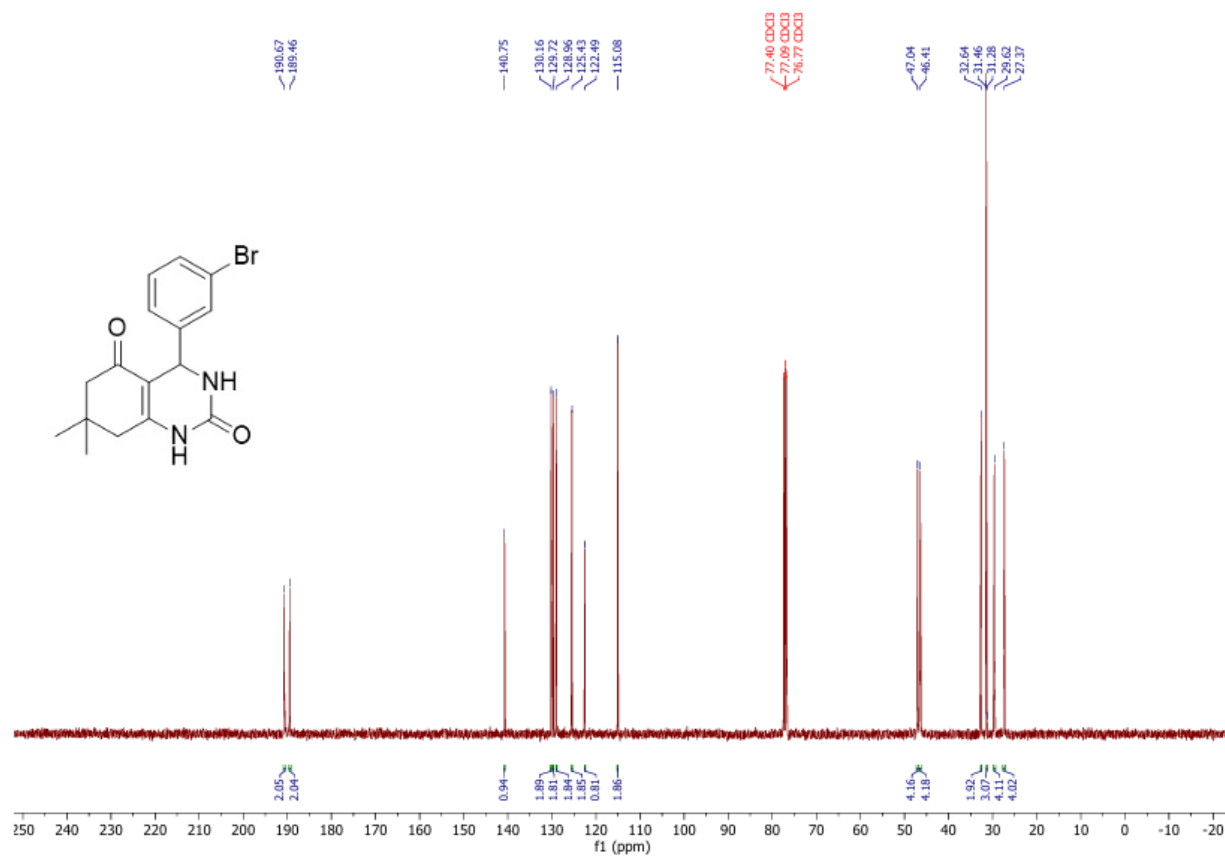
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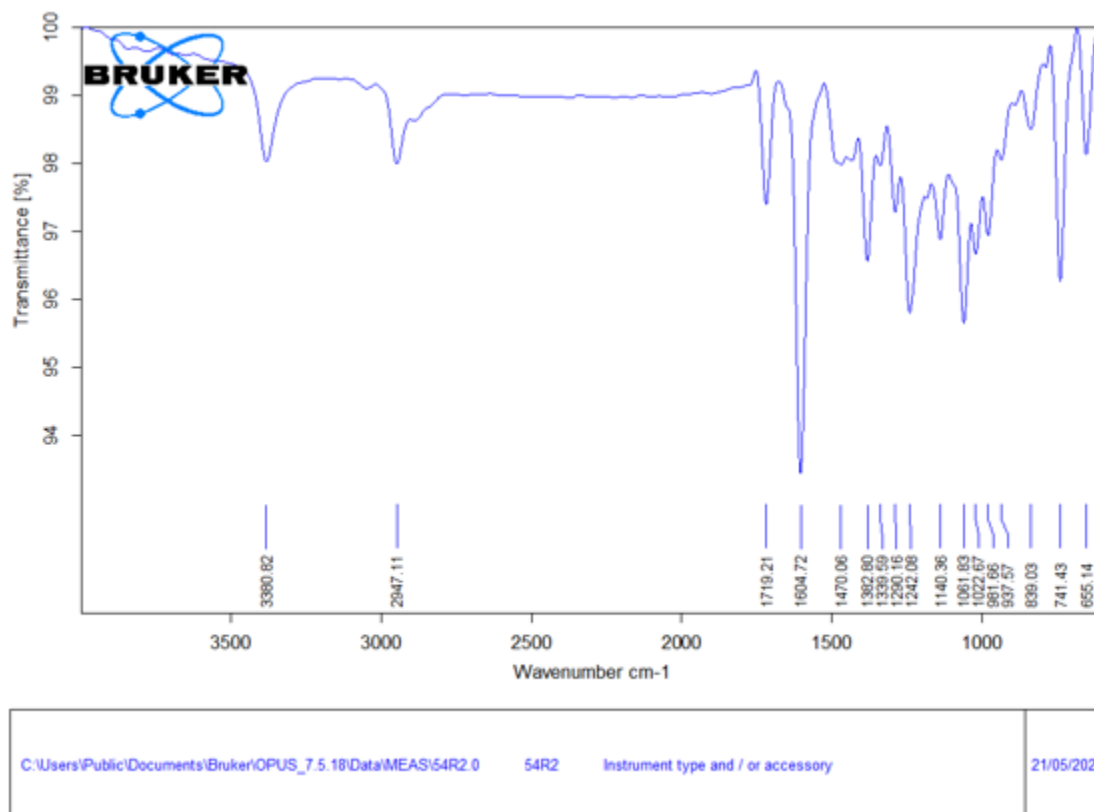
20. LC-MS of 3,4,7,8-tetrahydro-7,7-dimethyl-4-phenylquinazoline-2,5-(1H,6H)-dione:-



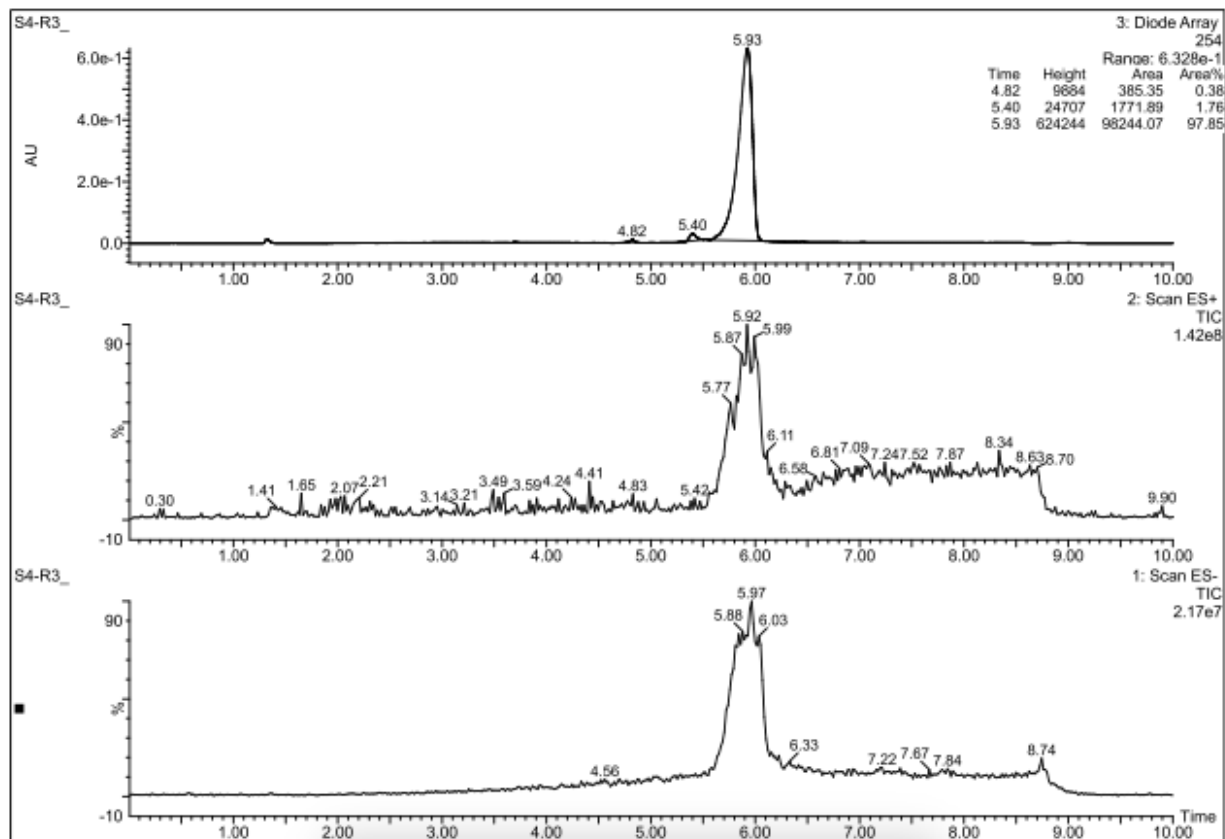
21. Mass of 3,4,7,8-tetrahydro-7,7-dimethyl-4-phenylquinazoline-2,5-(1H,6H)-dione:-**CHROMEIN, Pune****Sample Name:** S4_R1**Acq. Date:** 5/5/2025 4:32:03 PM**Method:** TEST_LCMS NEG_120-600.M**Instrument:** Agilent 6320 Ion Trap**File Name:** S4_R1_NEG_050525_17.D**Vial No.**

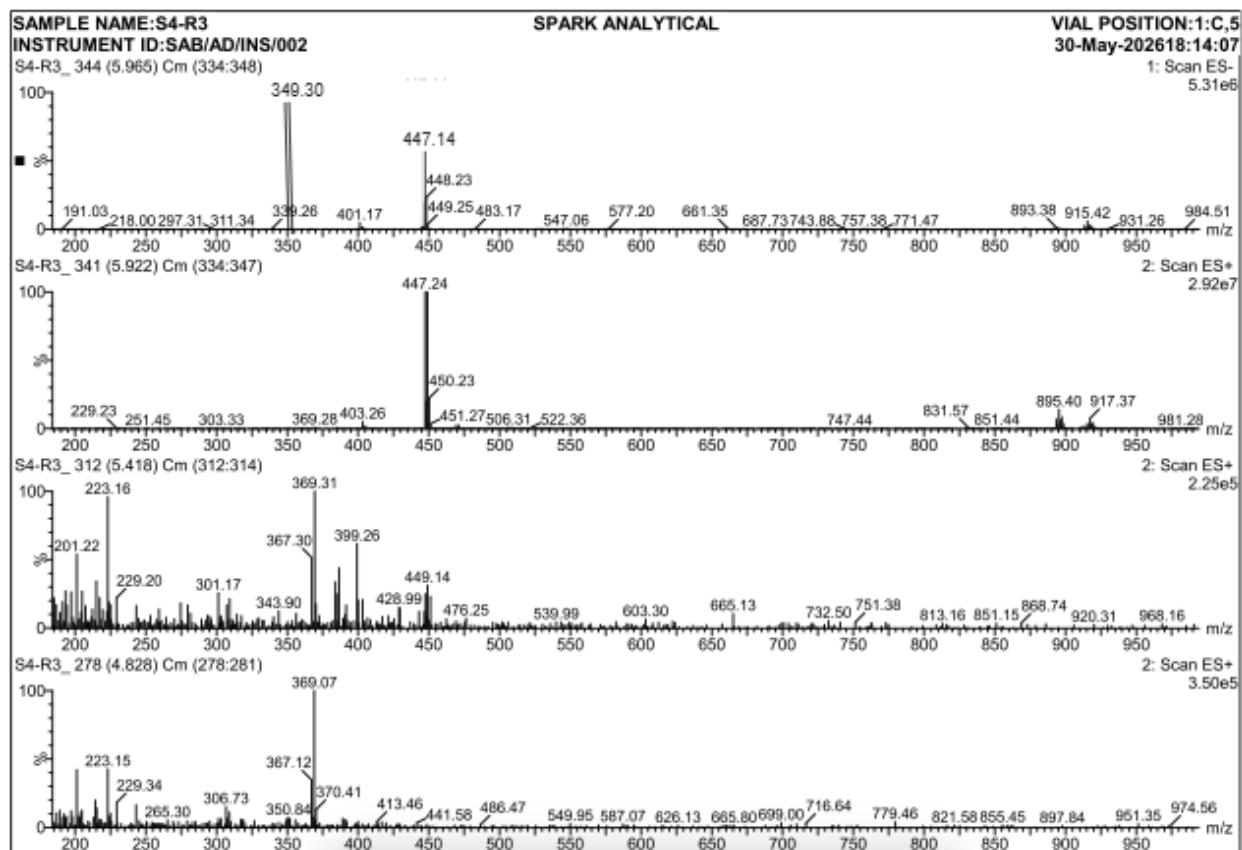
22. ^1H NMR of 4-(3-Bromophenyl)-3,4,7,8-tetrahydro-7,7-dimethylquinazoline-2,5-(1H,6H)-dione:-

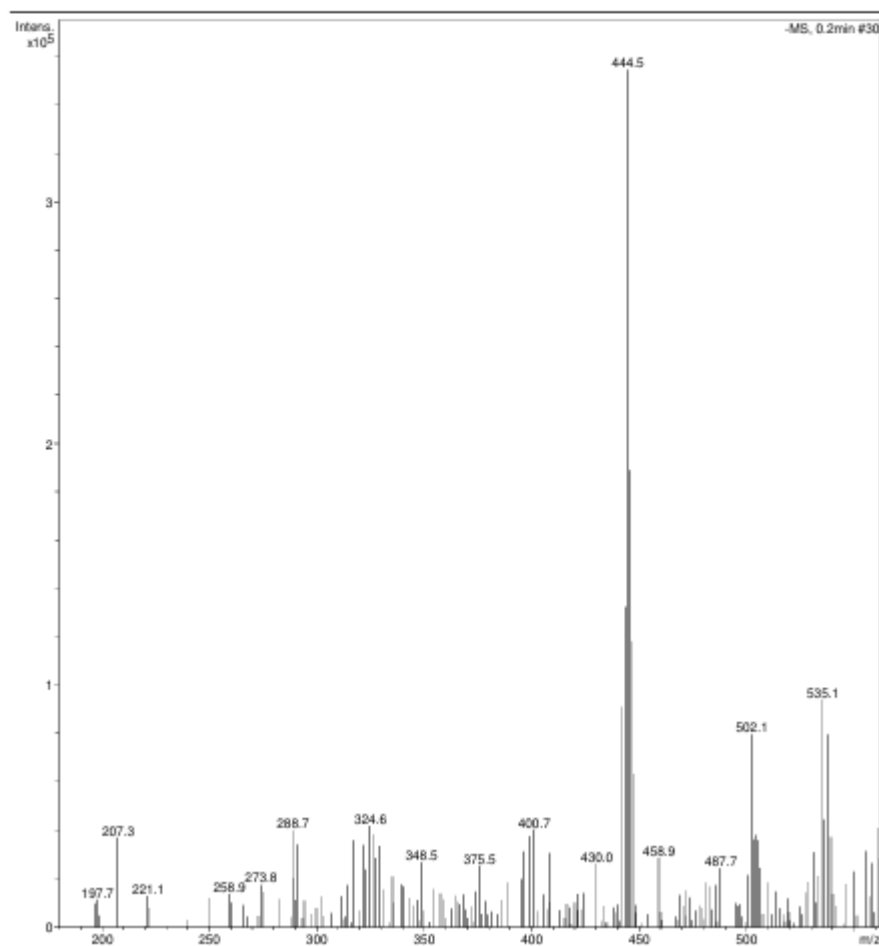
23. ^{13}C NMR of 4-(3-Bromophenyl)-3,4,7,8-tetrahydro-7,7-dimethylquinazoline-2,5-(1H,6H)-dione:-

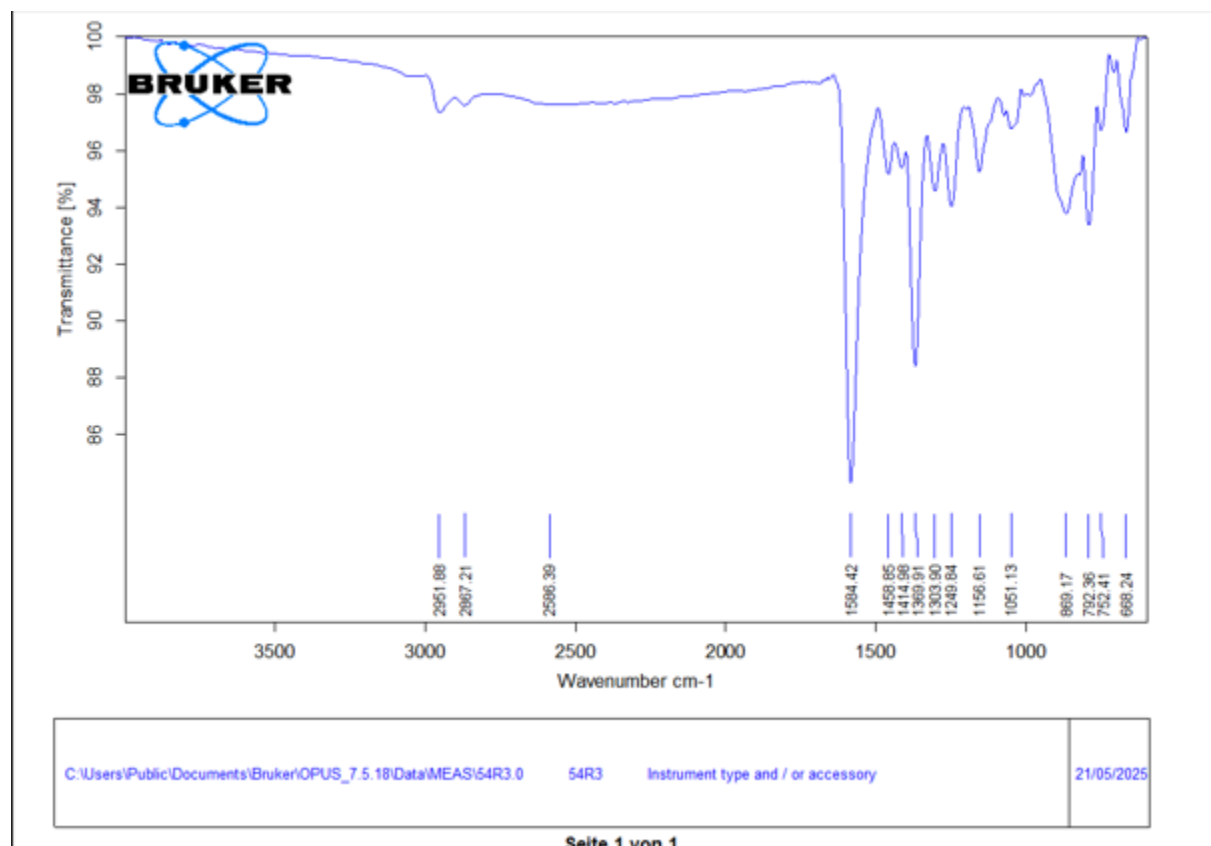
24. IR of 4-(3-Bromophenyl)-3,4,7,8-tetrahydro-7,7-dimethylquinazoline-2,5-(1H,6H)-dione:-

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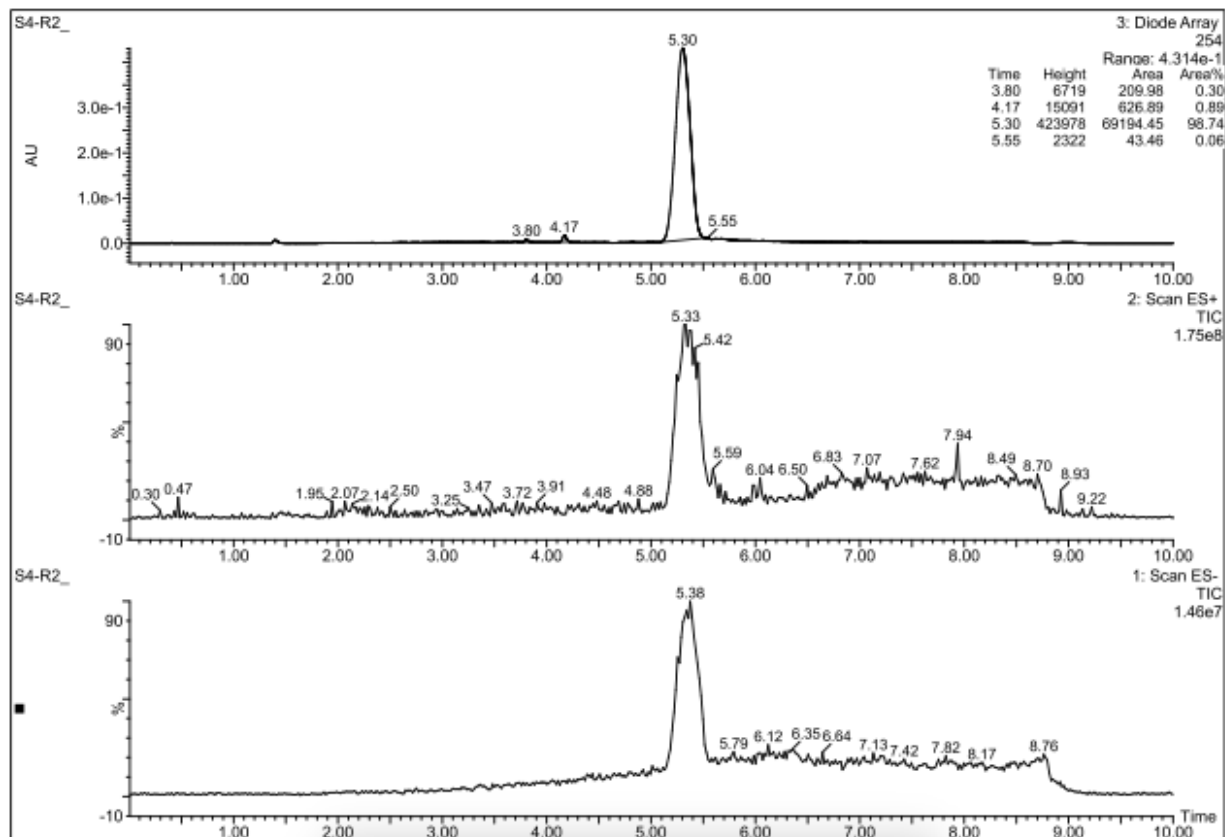
25. LC-MS of 4-(3-Bromophenyl)-3,4,7,8-tetrahydro-7,7-dimethylquinazoline-2,5-(1H,6H)-dione:-

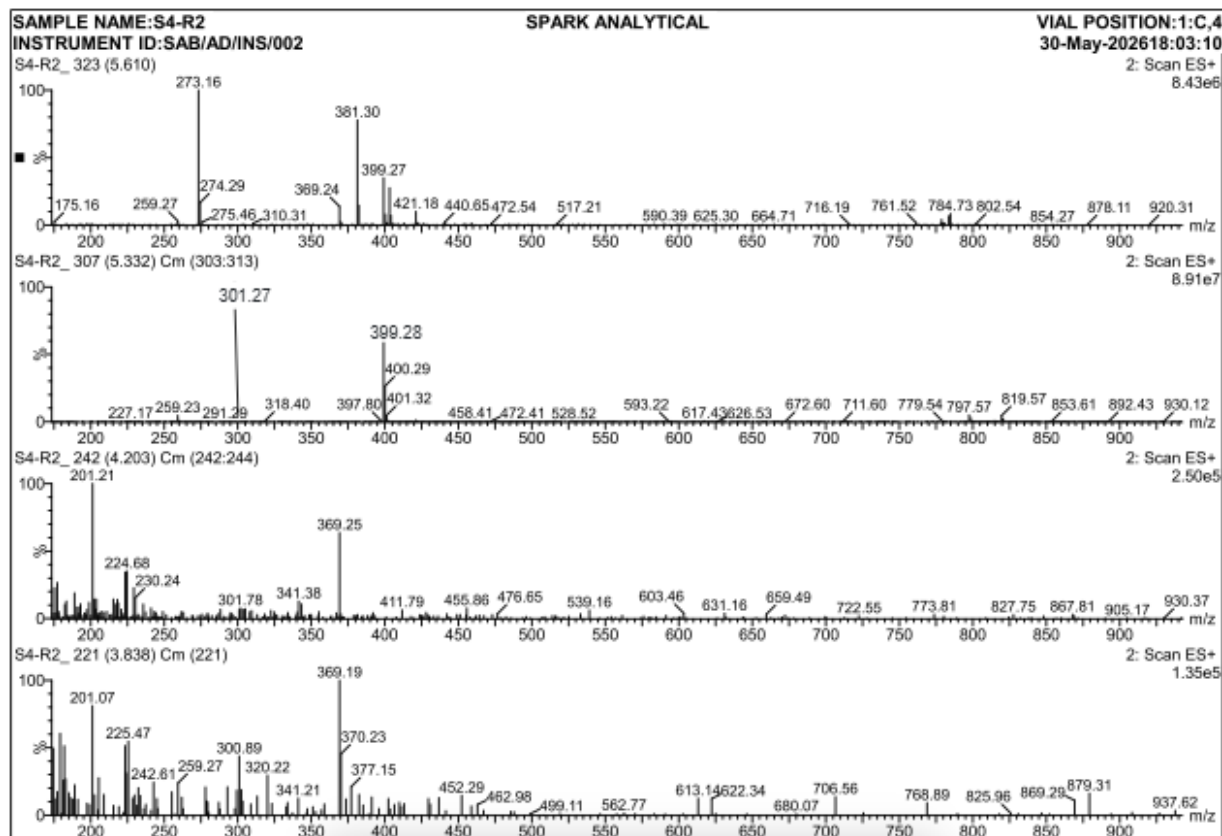


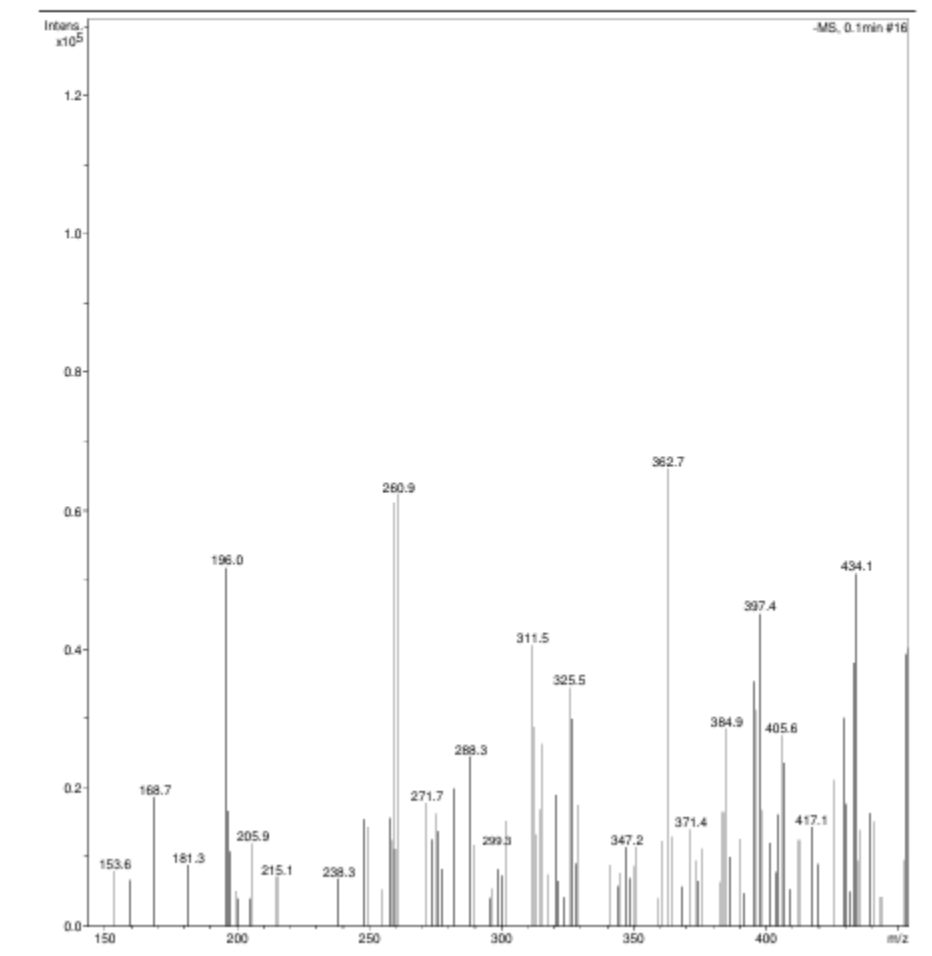
26. Mass of 4-(3-Bromophenyl)-3,4,7,8-tetrahydro-7,7-dimethylquinazoline-2,5-(1H,6H)-dione:-**CHROMEIN,Pune****Sample Name:** S4_R3**Method:** TEST_LCMS NEG_120-600.M**File Name:** S4_R3_NEG_050525_19.D**Acq. Date:** 5/5/2025 4:35:01 PM**Instrument:** Agilent 6320 Ion Trap**Vial No.**

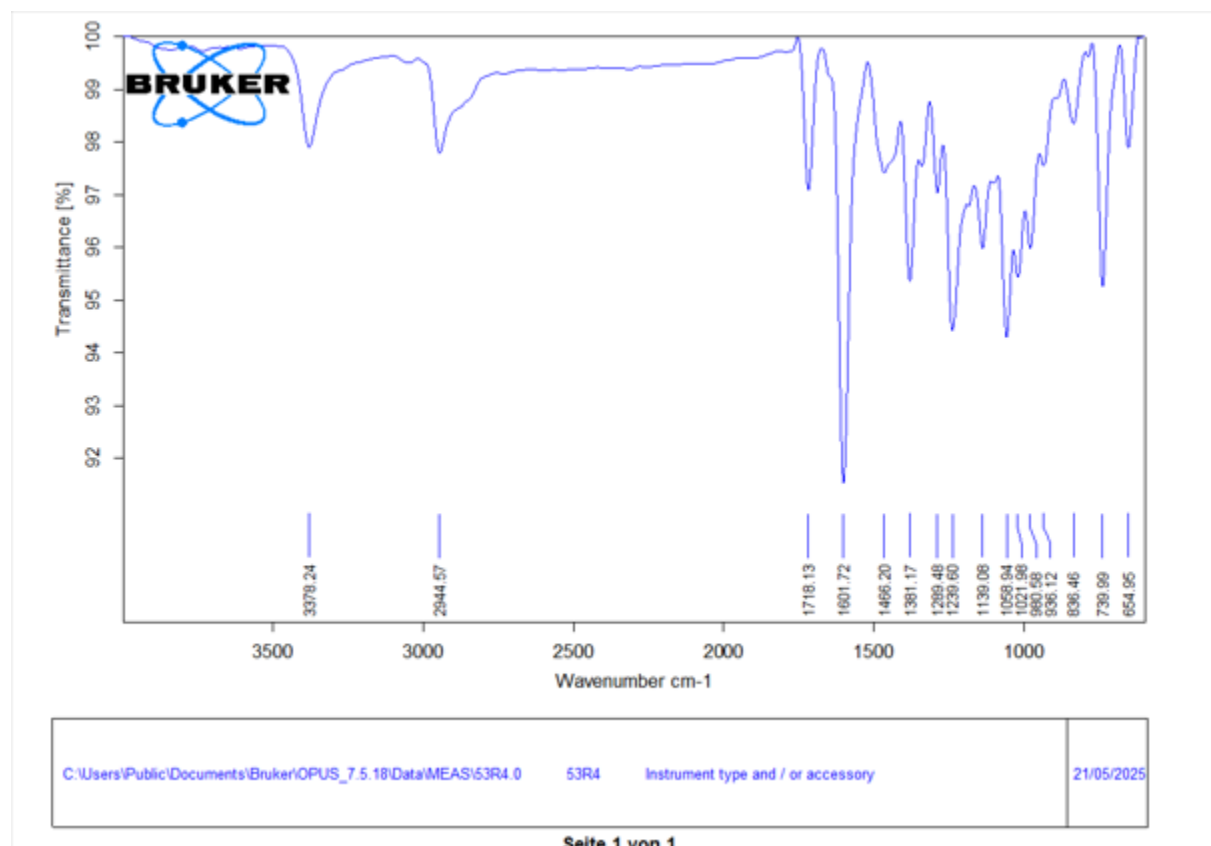
27. IR of 3,4,7,8-tetrahydro-4-(2-methoxyphenyl)-7,7-dimethylquinazoline-2,5-(1H,6H)-dione:-

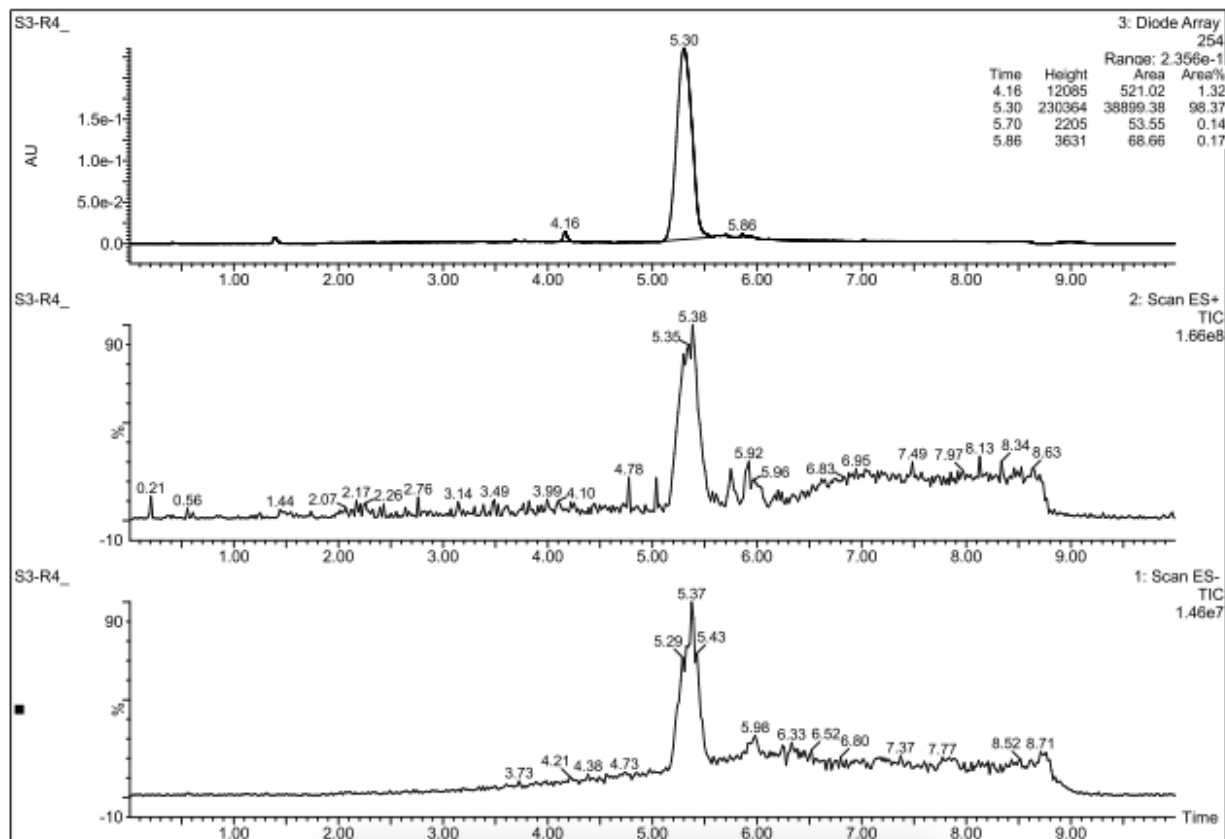
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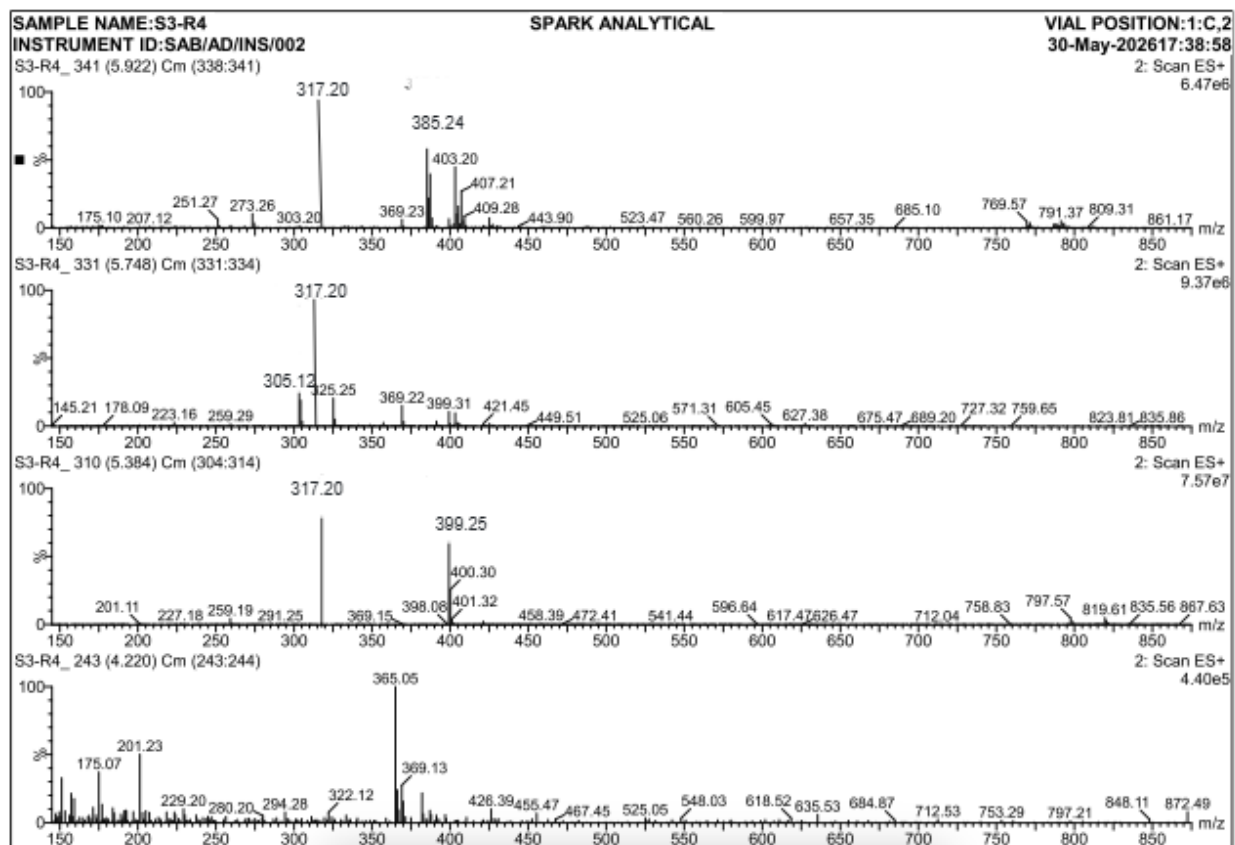
28. LC-MS of 3,4,7,8-tetrahydro-4-(2-methoxyphenyl)-7,7-dimethylquinazoline-2,5-(1H,6H)-dione:-



29. Mass of 3,4,7,8-tetrahydro-4-(2-methoxyphenyl)-7,7-dimethylquinazoline-2,5-(1H,6H)-dione:-**CHROMEIN,Pune****Sample Name:** S4_R2**Method:** TEST_LCMS NEG_120-600.M**File Name:** S4_R2_NEG_050525_18.D**Acq. Date:** 5/5/2025 4:33:32 PM**Instrument:** Agilent 6320 Ion Trap**Vial No.**

30. IR of 1,2,3,4,7,8-hexahydro-4-(2-methoxyphenyl)-7,7-dimethyl-2-thioxoquinazoline-5-(6H)-one:-

31. LC-MS of 1,2,3,4,7,8-hexahydro-4-(2-methoxyphenyl)-7,7-dimethyl-2-thioxoquinazoline-5-(6H)-one:-



32. Mass of 1,2,3,4,7,8-hexahydro-4-(2-methoxyphenyl)-7,7-dimethyl-2-thioxoquinazoline-5-(6H)-one:-

CHROMEIN,Pune

Sample Name: S3_R4

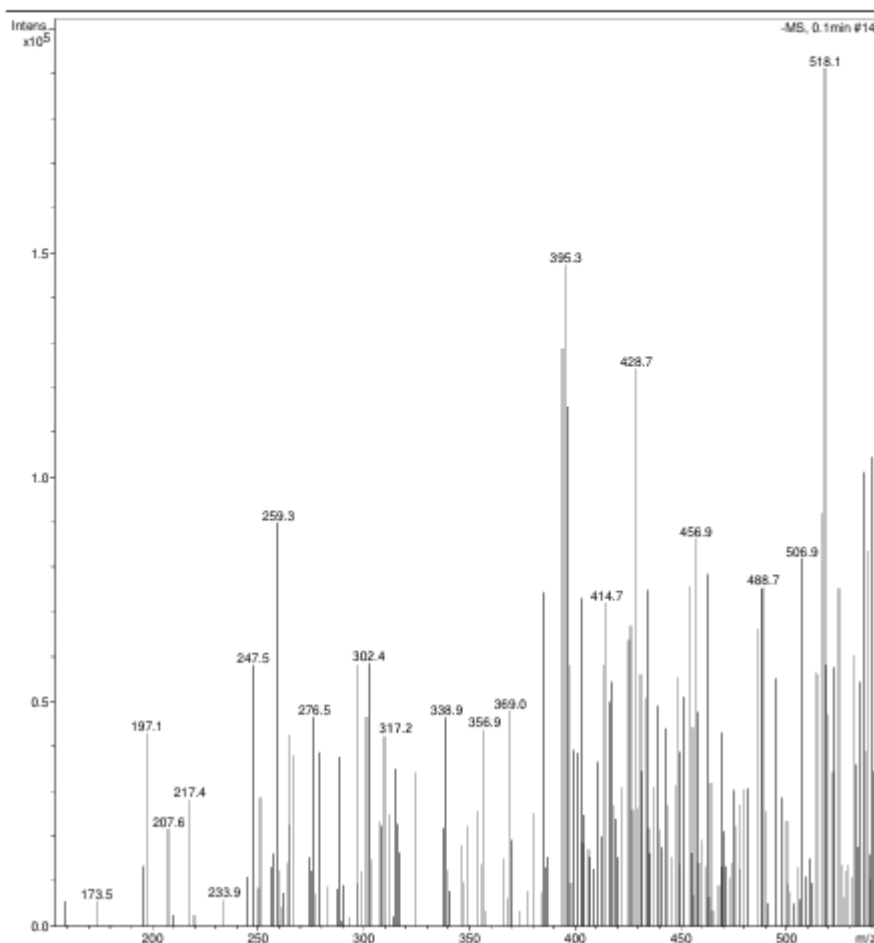
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File Name: S3_R4_NEG_050525_16.D

Acq. Date: 5/5/2025 4:30:34 PM

Instrument: Agilent 6320 Ion Trap

Vial No.



33. Analytical data of compounds:

1,2,3,4,7,8-Hexahydro-7,7-dimethyl-4-phenyl-2-thioxoquinazoline-5-(6H)-one: (1d), yield 98%, IR : 692.52, 771.78, 838.10, 1042.95, 1156.69, 1244.54, 1296.41, 1365.36, 1448.50, 1580.15, 1650.83, 2632.58, 2875.51, 2956.39, 3026.90, 3823.84; Purity:99.38%; Mass (+ve mode): 287.22; ^1H NMR: (400 MHz, CDCl_3) δ = 0.97 (s, 3H), 1.38 (s 3H), 1.99-2.47 (m, 4H), 5.53 (s,1H), 6.98-7.51 (5H, m), 11.55 (s, 1H), 11.89 (s, 1H); ^{13}C NMR (CDCl_3):

27.50, 29.74, 31.54, 32.84, 46.54, 47.16, 77.35, 115.71, 125.94, 126.85, 138.17, 189.49, 190.57.

4-(3-Bromophenyl)-1,2,3,4,7,8-hexahydro-7,7-dimethyl-2-thioxoquinazoline-5-(6H)-one: (2d), yield 96%, IR: 664.58, 707.62, 749.25, 785.27, 862.23, 1049.47, 1153.94, 1245.83, 1301.14, 1366.92, 1431.64, 1457.95, 1582.08, 1691.34, 2582.62, 2872.06, 2872.06, 2952.21, 3744.13; Purity: 97.17%; Mass (+ve mode): 365.05; ^1H NMR: (400 MHz, CDCl_3) δ = 1.00 (s, 3H), 1.22 (s, 3H), 2.22-2.48 (m, 4H), 5.47-5.48 (s, 1H), 6.99-7.30 (4H, m), 11.50 (s, 1H), 11.89 (s, 1H); ^{13}C NMR (CDCl_3): 27.45, 29.67, 31.54, 32.71, 46.50, 47.12, 77.35, 115.14, 122.55, 122.58, 125.51, 129.01, 140.83, 189.55, 190.76.

1,2,3,4,7,8-hexahydro-4-(4-methoxyphenyl)-7,7-dimethyl-2-thioxoquinazoline-5-(6H)-one: (8d), yield 90%, IR: 655.14, 741.43, 839.03, 937.57, 981.66, 1022.67, 1061.83, 1140.36, 1242.08, 1290.16, 1339.59, 1382.80, 1470.06, 1604.72, 1719.21, 2947.11, 3380.82; Purity: 98.37%; Mass (+ve mode): 317.20. (For ^1H NMR & ^{13}C NMR Ref. 22).

3,4,7,8-Tetrahydro-7,7-dimethyl-4-phenylquinazoline-2,5-(1H,6H)-dione: (11d), yield 96%, IR: 687.75, 770.62, 838.37, 1156.11, 1243.87, 1297.67, 1364.95, 1408.62, 1452.43, 1581.47, 2878.48, 2958.25; Purity: 99.74%; Mass (+ve mode): 271.12; ^1H NMR: (400 MHz, CDCl_3) δ = 1.04 (s, 3H), 1.22 (s, 3H), 2.20-2.45 (m, 4H), 5.51 (s, 1H), 6.96-7.49 (m, 5H), 11.54 (s, 1H), 11.87 (s, 1H); ^{13}C NMR (CDCl_3): 27.51, 31.33, 31.58, 32.58, 46.54, 47.16, 77.36, 115.68, 125.89, 126.83, 126.88, 138.18.

4-(3-Bromophenyl)-3,4,7,8-tetrahydro-7,7-dimethylquinazoline-2,5-(1H,6H)-dione: (12d) yield 96% IR: 668.24, 752.41, 792.36, 869.17, 1051.13, 1156.61, 1249.84, 1303.90, 1369.91, 1414.98, 1458.85, 1584.42, 2586.39, 2867.21, 2951.88; Purity: 97.85%; Mass (+ve mode): 349.30; ^1H NMR: (400 MHz, CDCl_3) δ = 1.12 (s, 3H), 1.25 (s, 3H), 2.31-2.51 (m, 4H), 5.51 (s, 1H), 7.02-7.33 (m, 5H), 11.92 (s, 1H); ^{13}C NMR (CDCl_3): 27.32, 29.62, 31.46, 32.64, 46.41, 47.04, 115.08, 122.49, 125.43, 128.96, 129.72, 130.16, 140.75, 189.46, 190.67.

3,4,7,8-Tetrahydro-4-(2-methoxyphenyl)-7,7-dimethylquinazoline-2,5-(1H,6H)-dione: (18d), yield 90%, IR: 655.14, 741.43, 839.03, 937.57, 981.66, 1022.67, 1061.83, 1140.36, 1242.08, 1290.16, 1339.59, 1382.80, 1470.06, 1604.72, 1719.21, 2947.11, 3380.82; Purity: 98.74%; Mass (+ve mode): 301.27. (For ^1H NMR & ^{13}C NMR Ref. 28).

All synthesized compounds were characterized by melting point, IR, ^1H NMR, and ^{13}C NMR spectroscopy. For the previously reported or specifically emphasized derivatives 1d, 2d, 11d, and 12d, full analytical and spectral data are provided in the experimental section. For known compounds, the physical constants (M.P.) and spectral data (^1H and ^{13}C NMR) were found to be in high agreement with literature values as follows: compounds 8d were cross-referenced with Ref. 22; 3d, 4d, 7d, 9d, and 10d were compared with Ref. 2; 5d, 6d, 15d, and 20d with Ref. 22; 13d and 16d with Ref. 27; 14d with Ref. 15; and 17d and 19d with and 8d were validated against Ref. 20.