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Synthesis of 2, 5-disubstituted-1, 3, 4-oxadiazole derivatives

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ABSTRACT

Synthesis of a series of various 2, 5-disubstituted-1, 3, 4-oxadiazole derivatives (7a-7u) have been done. Synthesis of a series of intermediates (3a-3c and 5a-5c) have been also done, ethyl-2-phenoxyacetate (3a), ethyl 2-(2, 4-dichlorophenoxy)acetate (3b), ethyl 2-(4-nitrorophenoxy) acetate (3c), 2-phenoxyacetohydrazide (5a), 2-(2, 4-dichlorophenoxy) acetohydrazide (5b), 2-(4-nitrophenoxy)acetohydrazide (5c), and final product (7a-7u), 2-(phenoxymethyl)-5-phenyl-1, 3, 4-oxadiazole (7a), 4-(5-(phenoxymethyl)-1, 3, 4-oxadiazol-2-yl)aniline (7b), 3-(5-(phenoxymethyl)-1, 3, 4-oxadiazol-2-yl) aniline (7c), 2-(5-(phenoxymethyl)-1, 3, 4-oxadiazol-2-yl)phenol (7d), 2, 4-dinitro-6-(5-(phenoxymethyl)-1, 3, 4-oxadiazol-2-yl)phenol (7e), 2-(4-(methylthio)benzyl)-5-(phenoxymethyl)-1,3,4-oxadiazole (7f), 2-((2, 4-dichlorophenoxy) methyl)-5-phenyl-1,3,4-oxadiazole (7g), 4-(5-((2, 4-dichlorophenoxy) methyl)-1,3,4-oxadiazol-2-yl)aniline (7h), 3-(5-((2, 4-dichlorophenoxy) methyl)-1,3,4-oxadiazol-2-yl)aniline (7i), 2-(5-((2, 4-dichlorophenoxy) methyl)-1, 3, 4-oxadiazol-2-yl)phenol (7j), 2-(5-((2, 4-dichlorophenoxy) methyl)-1,3,4-oxadiazol-2-yl)-4,6-dinitrophenol (7k), 2-((2,4-dichlorophenoxy) methyl)-5-(4-(methylthio)benzyl)-1,3,4-oxadiazole (7l), (Z)-2-((2, 4-dichlorophenoxy) methyl)-5-styryl-1,3,4-oxadiazole (7m), (S)-4-(2-(5-((2,4-dichlorophenoxy) methyl)-1, 3, 4-oxadiazol-2-yl)propyl)phenol (7n), 2-((4-nitrophenoxy) methyl)-5-phenyl-1, 3, 4-oxadiazole (7o), 4-(5-((4-nitrophenoxy)methyl)-1,3,4-oxadiazol-2-yl)aniline (7p), 3-(5-((4-nitrophenoxy)methyl)-1,3,4-oxadiazol-2-yl)aniline (7q), 2-(5-((4-nitrophenoxy)methyl)-1,3,4-oxadiazol-2-yl)phenol (7r), 2, 4-dinitro-6-(5-((4-nitrophenoxy)methyl)-1,3,4-oxadiazol-2-yl)phenol (7s), 2-(4-(methylthio) benzyl)-5-((4-nitrophenoxy)methyl)-1,3,4-oxadiazole (7t), (Z)-2-((4-nitrophenoxy)methyl)-5-styryl-1,3,4-oxadiazole (7u) and 5-((2, 4-dichlorophenoxy)methyl)-1,3,4-oxadiazole-2-thiol (7v).

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1. Introduction

Infection: An infection is the detrimental colonization of a host organism by a foreign species. In an infection, the infecting organism seeks to utilize the host's resources to multiply, usually at the expense of the host.[1]

Anti-infectives: Agents that are designed to kill, or prevent the growth of microorganisms without serious toxicity to the host.[2]

Role of 1,3,4-Oxadiazole moiety in modern drug discovery.

A Wide variety of substituted 1,3,4-oxadiazoles have attracted considerable attention in the field of drug discovery because of their wide range of pharmacological activities. Oxadiazoles have occupied a specific place in the field of medicinal chemistry due to its wide range of activities[3].

1,3,4-oxadiazole derivatives show anticonvulsant[4], antimicrobial activity[5], insecticide[6], anti-tubercular activity[7], anti-inflammatory activity[8], cardiovascular activity[9], antialzheimers activity[10], anti-tumor agents[11], antidiabetes[12] and spasmolytic activity[13].

A series of various derivatives of final products 2-(phenoxymethyl)-5-phenyl-1, 3, 4-oxadiazole (7a), 4-(5-(phenoxymethyl)-1, 3, 4-oxadiazol-2-yl)aniline (7b), 3-(5-(phenoxymethyl)-1, 3, 4-oxadiazol-2-yl) aniline (7c), 2-(5-(phenoxymethyl)-1, 3, 4-oxadiazol-2-yl)phenol (7d), 2, 4-dinitro-6-(5-(phenoxymethyl)-1, 3, 4-oxadiazol-2-yl)phenol (7e), 2-(4-(methylthio)benzyl)-5-(phenoxymethyl)-1,3,4-oxadiazole (7f), 2-((2, 4-dichlorophenoxy) methyl)-5-phenyl-1,3,4-oxadiazole (7g), 4-(5-((2, 4-dichlorophenoxy)

methyl)-1,3,4-oxadiazol-2-yl)aniline (7h), 3-(5-((2, 4-dichlorophenoxy) methyl)-1,3,4-oxadiazol-2-yl)aniline (7i), 2-(5-((2, 4-dichlorophenoxy) methyl)-1, 3, 4-oxadiazol-2-yl)phenol (7j), 2-(5-((2, 4-dichlorophenoxy) methyl)-1,3,4-oxadiazol-2-yl)-4,6-dinitrophenol (7k), 2-((2,4-dichlorophenoxy) methyl)-5-(4-(methylthio)benzyl)-1,3,4-oxadiazole (7l), (Z)-2-((2, 4-dichlorophenoxy) methyl)-5-styryl-1,3,4-oxadiazole (7m), (S)-4-(2-(5-((2,4-dichlorophenoxy) methyl)-1, 3, 4-oxadiazol-2-yl)propyl)phenol (7n), 2-((4-nitrophenoxy) methyl)-5-phenyl-1, 3, 4-oxadiazole (7o), 4-(5-((4-nitrophenoxy)methyl)-1,3,4-oxadiazol-2-yl)aniline (7p), 3-(5-((4-nitrophenoxy)methyl)-1,3,4-oxadiazol-2-yl)aniline (7q), 2-(5-((4-nitrophenoxy)methyl)-1,3,4-oxadiazol-2-yl)phenol (7r), 2, 4-dinitro-6-(5-((4-nitrophenoxy)methyl)-1,3,4-oxadiazol-2-yl)phenol (7s), 2-(4-(methylthio) benzyl)-5-((4-nitrophenoxy)methyl)-1,3,4-oxadiazole (7t), (Z)-2-((4-nitrophenoxy)methyl)-5-styryl-1,3,4-oxadiazole (7u) and 5-((2, 4-dichlorophenoxy)methyl)-1,3,4-oxadiazole-2-thiol (7v) has been synthesized by a mixture of equimolar amounts of the substituted 2-phenoxyacetohydrazide and substituted aromatic acid were, contained in a round bottom flask and suspended in 5 ml phosphoryl trichloride. The mixture was refluxed on a sand bath with vigorous stirring. The reaction was monitored by TLC. The reaction was continued till the substituted 2-phenoxyacetohydrazide was consumed completely. Initially, the color of reaction mixture was color less in case of phenol while in other phenols light yellow and reaction proceeded till reaction mixture became dark in color.

All the compounds synthesized have been characterized by spectral data.

2. Experimental

2.1 General synthetic scheme for synthesis of substituted ethyl 2-phenoxyacetate (3a-3c)

Sr. No.	Compound	R
1	3a	-H
2	3b	Chloro group at position 1 and 4
3	3c	Nitro group at position 4

General Procedure:[14]

A mixture of equimolar amounts of the substituted phenol and ethylchloroacetate were, contained in a round bottom flask and suspended in 50-60 ml acetone and anhydrous potassium carbonate (1-2gm) was added in the mixture. The mixture was refluxed on a sand bath with vigorous stirring. The reaction was monitored by TLC. The reaction was continued till the substituted phenol was consumed completely. Initially, the colour of reaction mixture was color less in case of phenol while in other pheols light yellow and reaction proceeded till reaction mixture became dark in colour.

Workup: The reaction mixture, when cooled, was filtered under vaccum to remove solid potassium carbonate and the filtrate thus obtained was evaporated under vaccum. The residue thus obtained was dissolved into ethylacetate (10-15 ml) and washed with water twice. Ethylacetate layer was separated and dried over anhydrous sodium sulphate. The solvent was evaporated under vaccum and the residue (liquid product) thus obtained was used for next step.

Yield % : 96.56% (3a), 92.53% (3b), 90.97% (3c)

TLC : Silica gel G; Hexane: Ethyl acetate (8:2)

R_f = 0.78 (3a), 0.79 (3b) and 0.56 (3c)

IR **(Spectrum 1)** of (3a): 3020.32, 1732.94, 1595.22, 1497.89, 1472.39, 1374.38, 1214.69, 1090.26, 1044.69, 746.68, 696.42, 666.72cm^{-1}

IR **(Spectrum 2)** of (3b): 3094.58, 3020.30, 2985.44, 1732.39, 1478.56, 1375.14, 1279.05, 1249.56, 1214.26, 1105.25, 1046.92, 929.20, 854.11, 744.60 cm^{-1}

IR **(Spectrum 3)** of (3c): 3062.74, 3019.59, 1749.28, 1716.97, 1592.05, 1338.83, 1213.97, 951.53, 848.51 cm^{-1}

2.2 General synthetic scheme for substituted 2-phenoxy acetohydrazide (5a-5c)

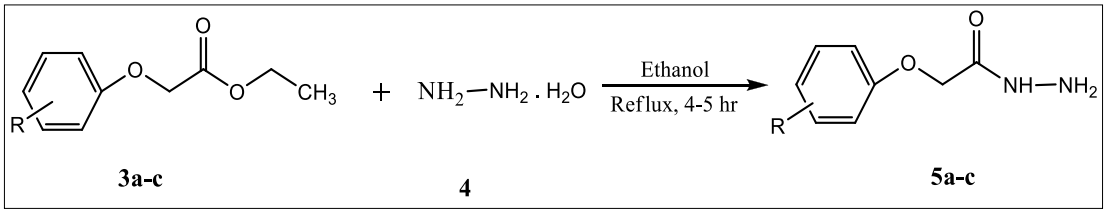


Figure 2:

Sr. No.	Compound	R
1	5a	-H
2	5b	Chloro group at position 1 and 4
3	5c	Nitro group at position 4

General Procedure:[14]

A solution of substituted ethyl 2-phenoxyacetate (1 mol) and hydrazine hydrate (1.5 mol) were contained in a round bottom flask and suspended in 50-60 ml ethanol. The mixture was refluxed for 5-6 hr on a sand bath with vigorous stirring. The reaction was monitored by TLC. The reaction was continued till the substituted ethyl 2-phenoxyacetate was consumed completely. Initially, the color of reaction mixture was colour less in case of phenol while in other phenols light yellow and reaction proceeded till reaction mixture became dark in colour.

Workup: The reaction mixture, when cooled, was filtered under vaccum to remove solid substituted 2-phenoxyacetohydrazide. The solid thus obtained was washed with ethanol, dried and used for next step.

Yield % : 96.87% (5a), 77.75% (5b) and 95.58% (5c)

Melting point : 100-105°C (5a), 150-155°C (5b) and 180-185°C (5c)

TLC : Silica gel G; Hexane: Ethyl acetate (1:1)

R_f = 0.34 (5a), 0.59 (5b) and 0.47 (5c)

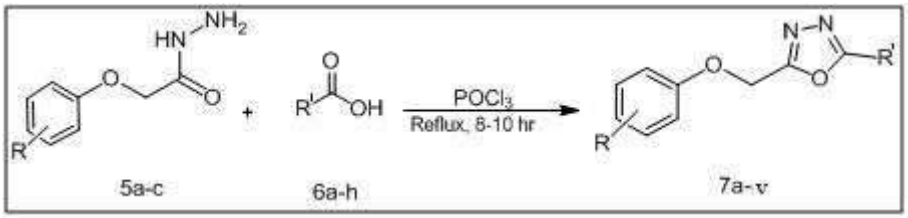
IR **(Spectrum 4)** of **(5a)**: 3357.51, 3291.45, 3274.32, 2985.19, 1683.72, 1647.31, 1635.70, 1558.36, 1448.15, 1418.67, 1362.37, 1119.37, 951.16, 741.62 cm^{-1}

IR **(Spectrum 5)** of **(5b)**: 3315.48, 3259.87, 3226.55, 3012.11, 1683.60, 1647.12, 1558.37, 1520.75, 1497.24, 1456.97, 1387.27, 1245.18, 1138.64, 1046.29, 791.81 cm^{-1}

IR **(Spectrum 6**) of **(5c)**: 3290.42, 3117.25, 3011.92, 2946.03, 2868.98, 1653.19, 1540.35, 1490.22, 1418.99, 1334.62, 1267.14, 1112.39, 1001.04, 825.07, 749.67, 699.30 cm^{-1}

2.3 Synthesis of final products (7a-7v)

General scheme



Sr. No.	Compound	R	R'
1	7a	-H	-C_6H_5
2	7b	-H	4-$NH_2C_6H_5$-
3	7c	-H	3-$NH_2C_6H_5$-
4	7d	-H	2-OHC_6H_5-
5	7e	-H	2- OH-3,5-dinitroC_6H_5-
6	7f	-H	1-CH_2-4-$SCH_3C_6H_5$-
7	7g	Chloro at position 2 and 4	-C_6H_5
8	7h	Chloro at position 2 and 4	4-$NH_2C_6H_5$-
9	7i	Chloro at position 2 and 4	3-$NH_2C_6H_5$-
10	7j	Chloro at position 2 and 4	2-OHC_6H_5-
11	7k	Chloro at position 2 and 4	2- OH-3,5-dinitroC_6H_5-
12	7l	Chloro at position 2 and 4	1-CH_2-4-$SCH_3C_6H_5$-
13	7m	Chloro at position 2 and 4	C_6H_5CH=CH-
14	7n	Chloro at position 2 and 4	4-$OHC_6H_5CH_2(NH_2)$-
15	7o	Nitro group at position 4	-C_6H_5
16	7p	Nitro group at position 4	4-$NH_2C_6H_5$-
17	7q	Nitro group at position 4	3-$NH_2C_6H_5$-
18	7r	Nitro group at position 4	2-OHC_6H_5-
19	7s	Nitro group at position 4	2- OH-3,5-dinitroC_6H_5-
20	7t	Nitro group at position 4	1-CH_2-4-$SCH_3C_6H_5$-
21	7u	Nitro group at position 4	C_6H_5CH=CH-
22	7v	Chloro at position 2 and 4	-SH

Sr. No.	Compound	PercentageYield	Melting Point	R_f Value
	7a	78.95	95-100°C	0.78
	7b	90.68	135-140°C	0.75
	7c	89.44	155-160°C	0.66
	7d	90.06	120-125°C	0.87
	7e	91.66	105-110°C	0.70
	7f	87.76	125-130°C	0.76
	7g	85.29	115-120°C	0.69
	7h	88.11	>250°C	0.75
	7i	78.32	>250°C	0.75
	7j	90.21	>250°C	0.75
	7k	90.66	110-115°C	0.75
	7l	79.63	>250°C	0.84
	7m	97.30	>250°C	0.72
	7n	78.39	140-145°C	0.74
	7o	90.78	150-155°C	0.78
	7p	89.19	100-105°C	0.44
	7q	93.22	120-125°C	0.44
	7r	91.89	>250°C	0.84
	7s	92.67	>250°C	0.47
	7t	92.90	>250°C	0.31
	7u	94.12	>250°C	0.75
	7v	88.98	120-125°C	0.78

Experimental Data

IR **(Spectrum 7) of (7a)**: 3011.86, 2977.55, 2966.99, 1635.48, 1597.58, 1556.61, 1436.47, 1237.48, 1175.15, 973.13, 771.21, 752.61 cm^{-1}

IR **(Spectrum 8) of (7b)**: 3066.88, 3033.69, 2977.96, 1662.08, 1540.49, 1496.42, 1172.78, 995.29, 896.02, 828.55 cm^{-1}

IR **(Spectrum 9) of (7c)**: 3110.94, 3077.17, 3020.98, 1669.60, 1615.92, 1418.10, 1317.71, 1219.69, 1090.33, 772.61 cm^{-1}

IR **(Spectrum 10) of (7d)**: 3067.99, 3012.03, 2948.61, 2890.45, 1652.29, 1540.77, 1456.56, 1219.55, 772.51, 688.50 cm^{-1}

IR **(Spectrum 11) of (7e)**: 3088.73, 3055.12, 3021.90, 1598.93, 1540.10, 1489.26, 1338.88, 1219.60, 855.69, 772.46 cm^{-1}

IR **(Spectrum 12) of (7f)**: 2946.08, 2838.51, 1698.90, 1635.69, 1496.90, 1473.30, 1362.27, 1216.53, 1136.47, 843.65 cm^{-1}

IR **(Spectrum 13) of (7g)**: 3094.58, 3020.30, 2985.44, 1732, 1478, 1375, 1279, 1249, 1105, 929, 854, 744 cm^{-1}

IR **(Spectrum 14) of (7h)**: 3344.56, 3274.44, 3024.34, 2922.61, 1647.19, 1558.52, 1558.52, 1473.93, 1156.29, 951.89, 798.86 cm^{-1}

IR **(Spectrum 15) of (7i)**: 334.31, 3139.63, 1558.60, 1520.61, 1464.99, 1295.24, 1204.84, 756.31 cm^{-1}

IR **(Spectrum 16) of (7j)**: 3336.78, 2925.45, 2854.17, 1652.45, 1624.40, 1540.76, 1474.58, 1435.42, 1388.64, 1289.03, 1212.89, 1157.69, 1102.84, 1072.37, 799.08 cm^{-1}

IR **(Spectrum 17) of (7k)**: 3087.57, 2921.42, 2872.32, 2839.16, 1652.87, 1558.45, 1540.74, 1464.77, 1339.21, 1219.56, 883.79, 772.31, 729.42 cm^{-1}

IR **(Spectrum 18) of (7l)**: 3177.48, 3022.63, 2900.55, 1601.85, 1525.74, 1474.15, 1391.16, 1226.00, 991.53, 870.75, 793.81, 763.13 cm^{-1}

IR **(Spectrum 19) of (7m)**: 2975.41, 2932.90, 1683.87, 1647.21, 1558.45, 1533.57, 1507.23, 1448.15, 1219.86, 773.05 cm^{-1}

IR **(Spectrum 20) of (7n)**: 3446.78, 3013.35, 2976.92, 2938.40, 1662.62, 1635.81, 1558.45, 1507.23, 1473.54, 1219.90, 1084.93, 771.72 cm^{-1}

IR **(Spectrum 21) of (7o)**: 3065.81, 3003.83, 2946.86, 1698.96, 1669.97, 1647.22, 1558.41, 1520.83, 1473.33, 1087.92, 940.84, 689.52 cm^{-1}

IR **(Spectrum 22) of (7p)**: 3333.63, 3295.67, 3136.42, 2907.44, 1635.61, 1591.39, 1508.27, 1340.76, 1251.21, 1175.91, 1111.09, 844.68, 749.56 cm^{-1}

IR **(Spectrum 23) of (7q)**: 3296.10, 3241.23, 3110.94, 3077.03, 1590.12, 1507.54, 1435.99, 1339.53, 1250.66, 843.50, 797.27 cm^{-1}

IR **(Spectrum 24) of (7r)**: 3054.50, 2977.28, 2908.40, 1516.22, 1340.38, 1251.29, 1220.74, 1064.25, 845.02, 772.63 cm^{-1}

IR **(Spectrum 25) of (7s)**: 3024.44, 2984.84, 1621.48, 1539.88, 1456.63, 1374.18, 1339.92, 1286.23, 1063.36, 865.57, 772.36 cm^{-1}

IR **(Spectrum 26) of (7t)**: 3039.38, 2992.33, 1652.36, 1623.35, 1558.08, 1475.23, 1220.67, 1129.95, 979.82, 867.41, 772.58 cm^{-1}

IR **(Spectrum 27) of (7u)**: 3088.29, 3054.22, 2992.16, 1557.96, 1519.00, 1489.19, 1339.19, 1258.85, 1112.13, 844.76 cm^{-1}

IR **(Spectrum 28) of (7v)**: 3229.10, 3013.16, 2946.86, 1698.96, 1669.97, 1647.22, 1558.41, 1520.83, 1473.33, 1087.92, 940.84, 689.52

3. Result and Discussion

3.1 Characterization of Intermediate (3a-3c)

IR: The spectrum showed characteristic ester band in the range 1749-1720cm^{-1} and C-Cl stretching peak at in the range of 750-780 cm^{-1}. The –OH stretching peak corresponding to the phenol in the range of 3450-3400cm^{-1} was disappeared thus confirming formation of the desired intermediate. Also, the values are in agreement with those reported ethyl 2-phenoxyacetate.[15]

3.2 Characterization of Intermediate (5a-5c)

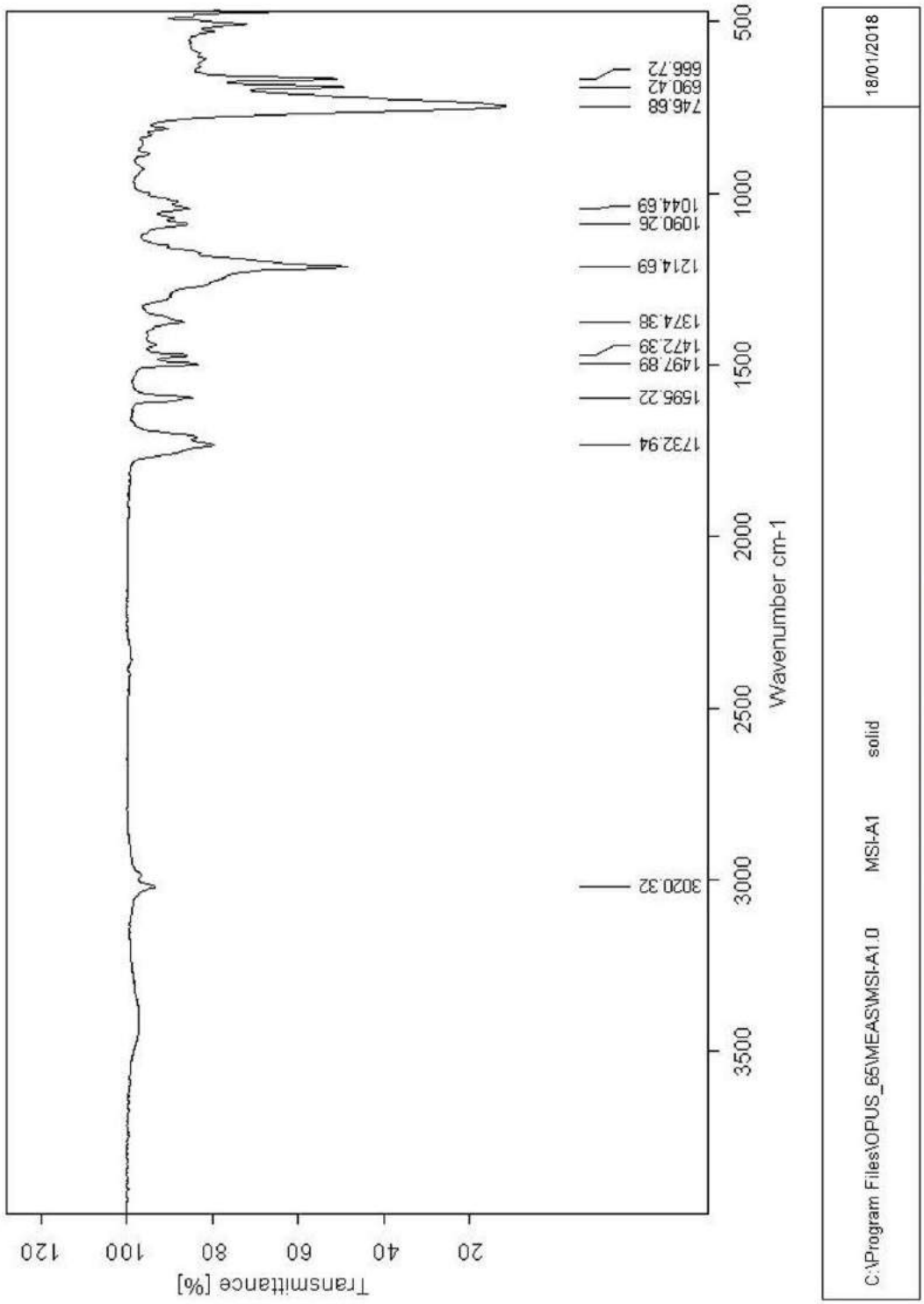
The spectrum showed characteristic hydrazide band in the range of 1640-1670cm^{-1} and the stretching peak corresponding to the ester in the range of 1725-11745cm^{-1} was disappeared. N-H stretching band of $–NH-NH_2$ group appeared in the range of 3325-3100cm^{-1}. That it confirm the formation of the desired intermediate. Also, the values are in agreement with the reported 2-phenoxyacetohydrazide.[15]

3.3 Characterization of 2, 5-disubstituted -1,3,4-oxadiazole (7a-7u)

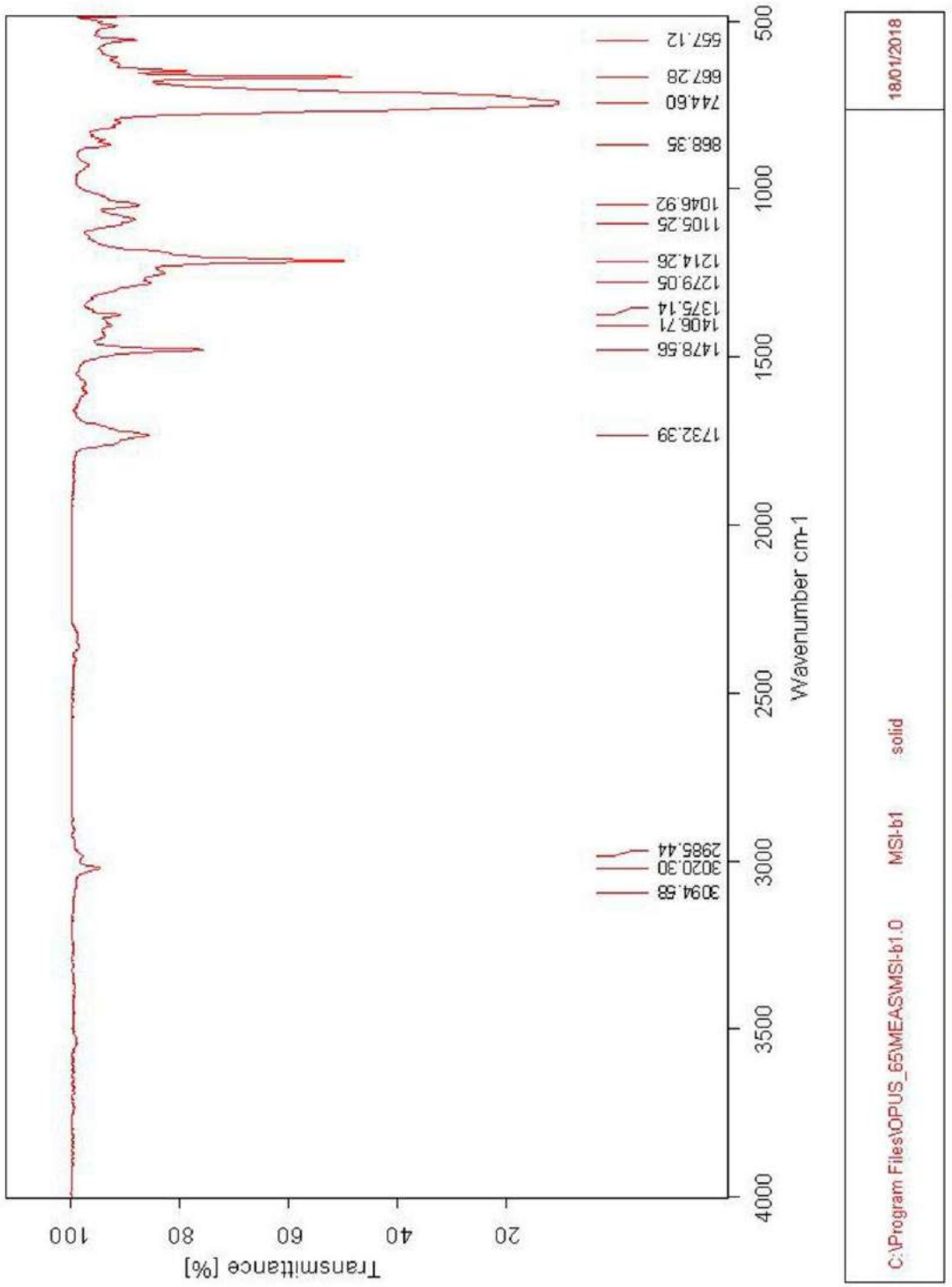
IR: The spectrum showed characteristic absorption band accordingly of the presence of functional group. The Ar-C-H stretching showed the absorption band in the range of 3020-3050 cm^{-1}. The –C=N stretching band appeared in the range of 1630-1660 cm^{-1}, -C=C- stretching peak comes in the range of 1400-1500 cm^{-1}, alkyl C-H stretching peak comes in the range of 2800-2900 cm^{-1}. Arylether stretching band appeared in the range of 1100-1200 cm^{-1}. Compounds **7b, 7c, 7h, 7i, 7n, 7p and 7q** contain aromatic $-NH_2$, which showed N-H stretching band in the range of 3150-3300 cm^{-1}

3.4 Characterization of 5-((2,4-dichlorophenoxy)methyl)--1,3,4-oxadiazole thiol (7v)

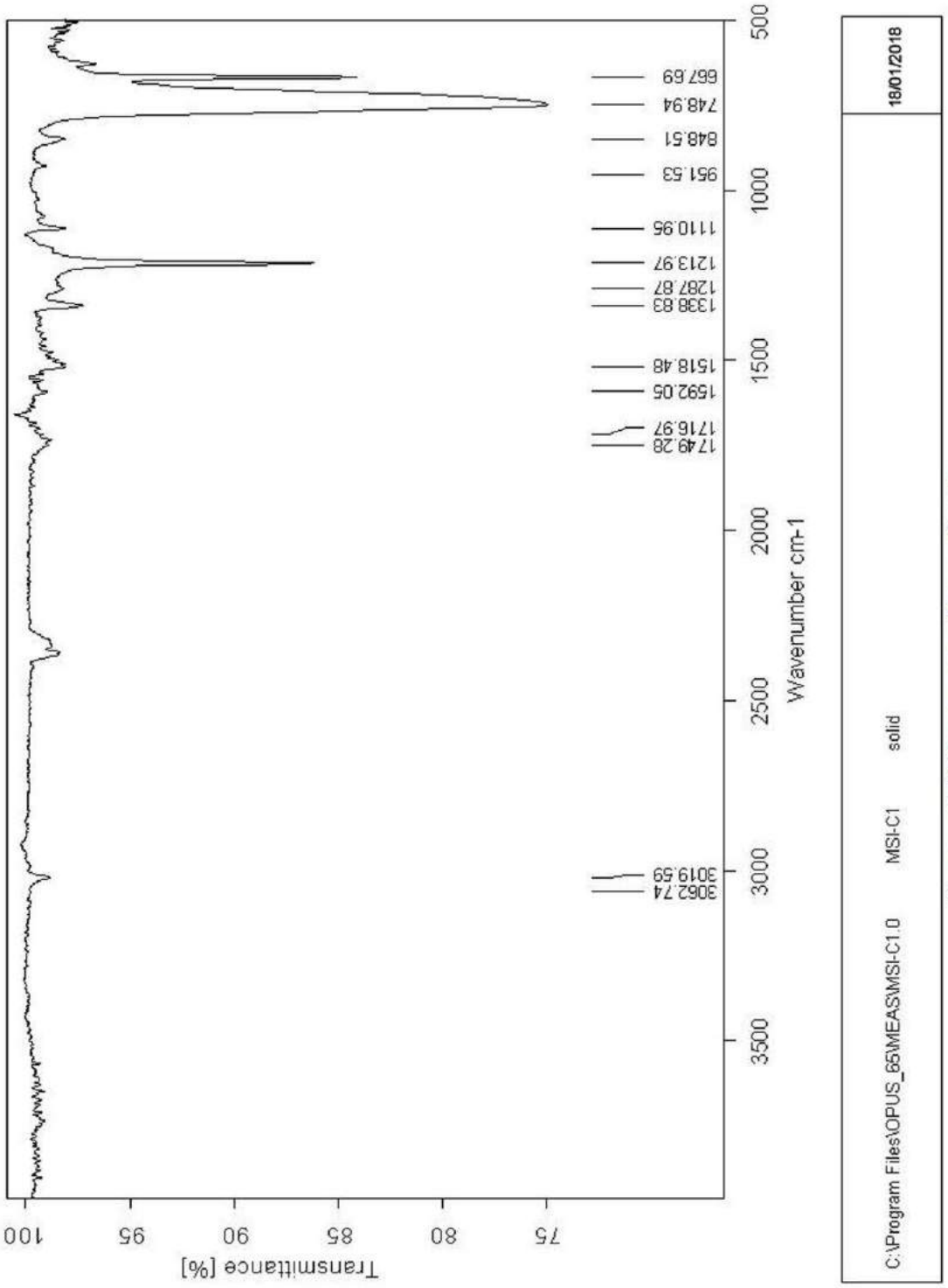
IR : In the solid state, the thiol (SH) form of the oxadiazole predominated the tautomeric thione (C=S). The characteristic SH stretching peak was seen 2550-2590 cm^{-1}.[15]



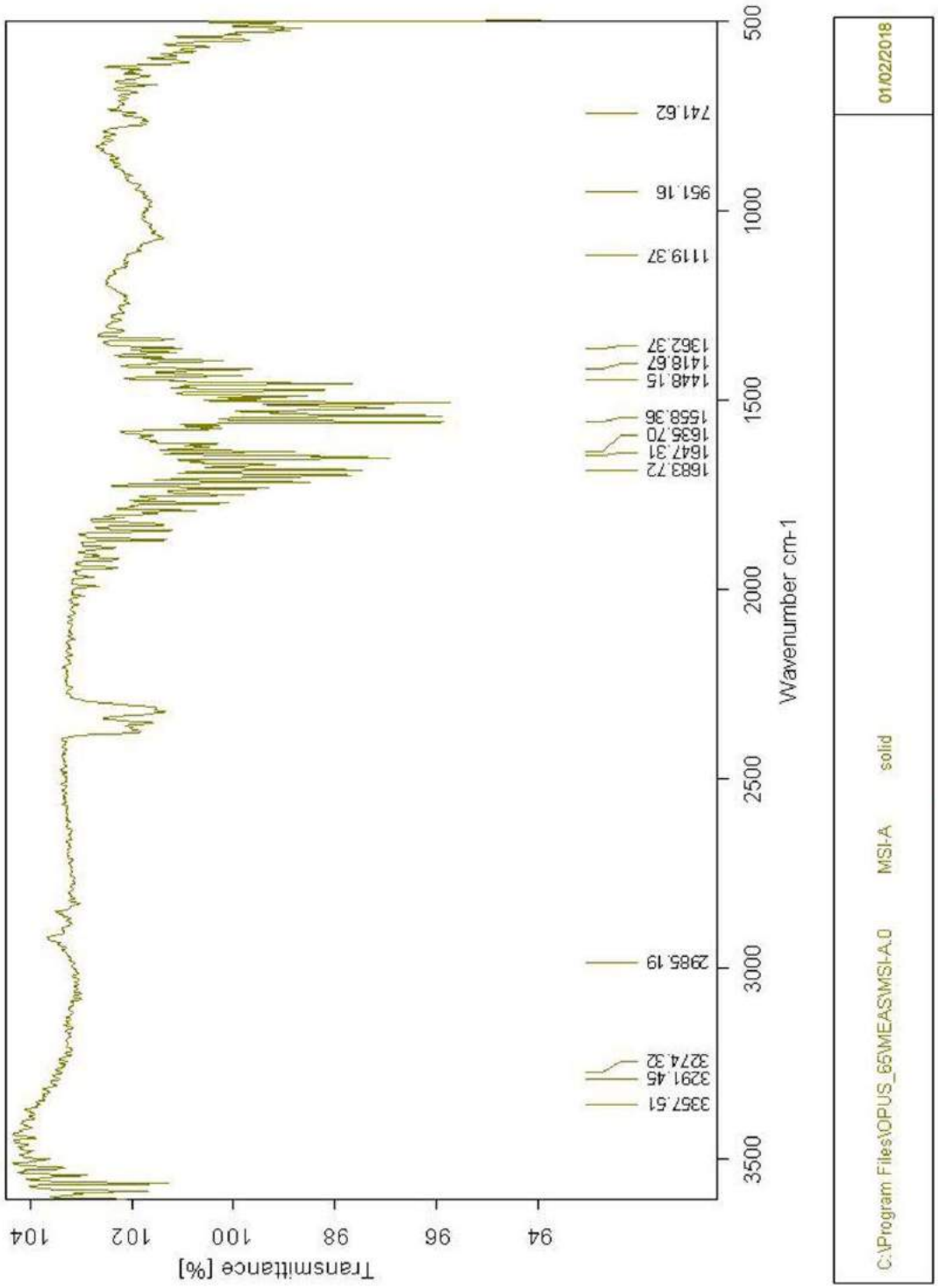
Spectrum 1: IR Spectrometer of intermediate 3a



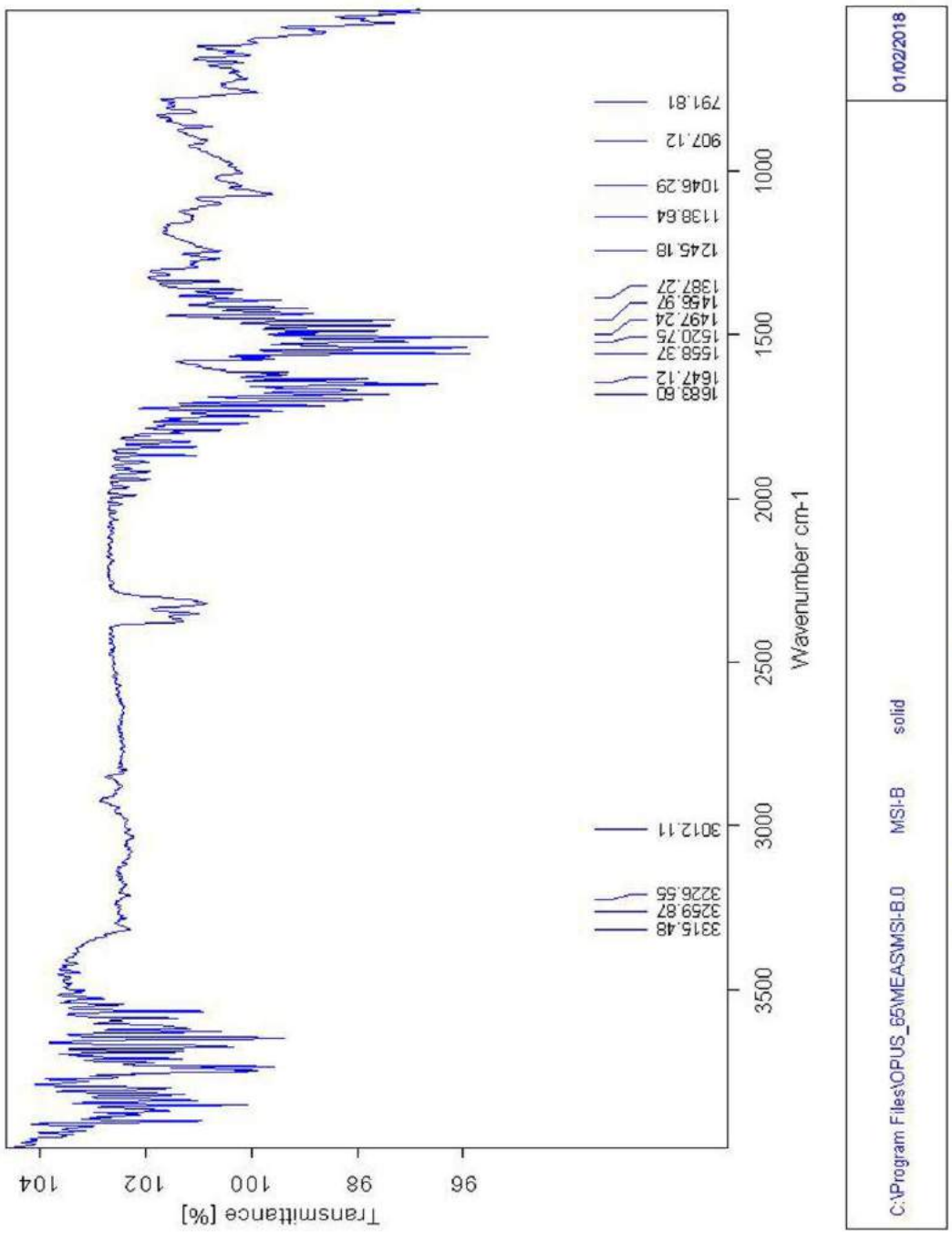
Spectrum 2: IR Spectrometer of intermediate 3b



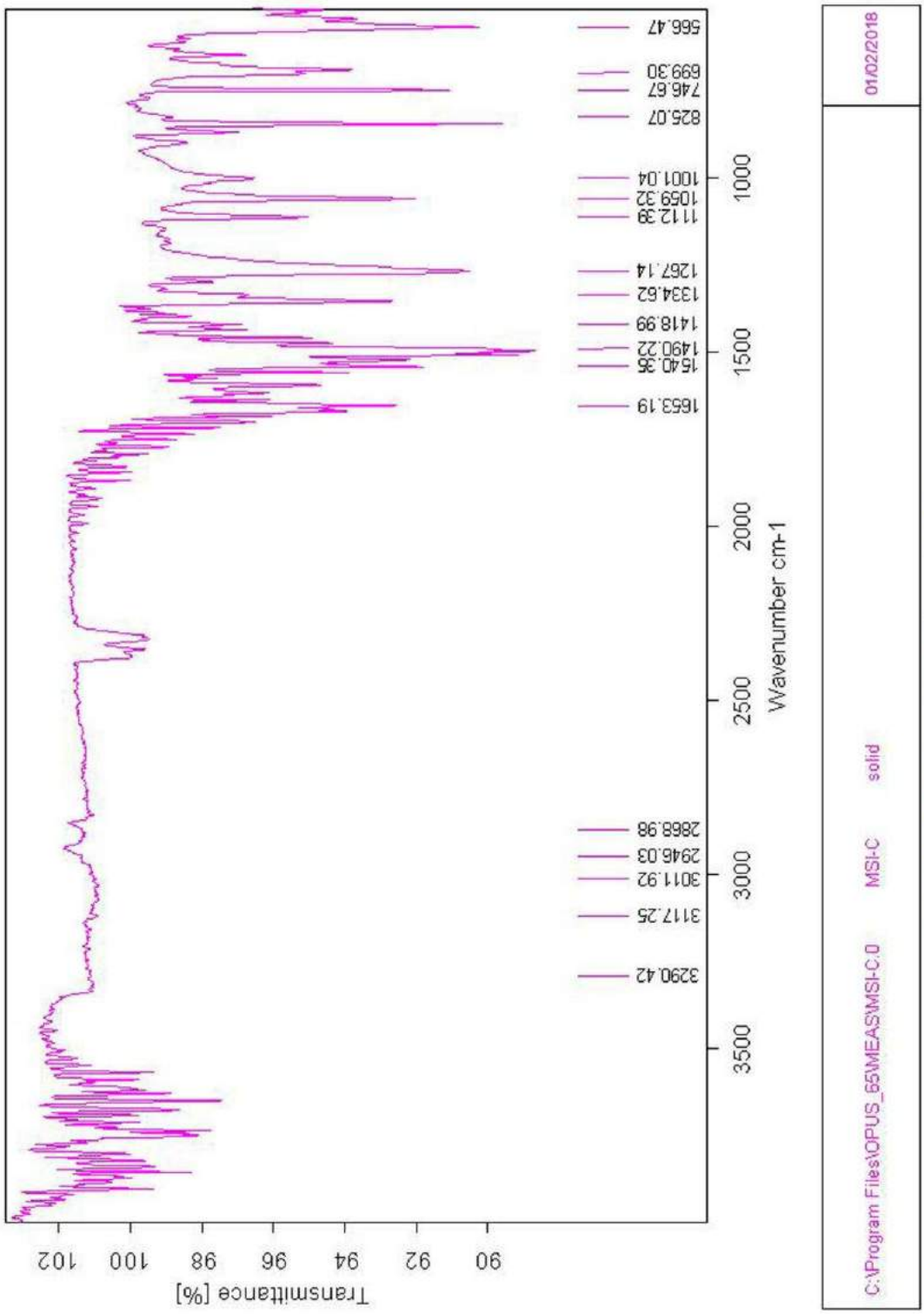
Spectrum 3: IR Spectrometer of intermediate 3c



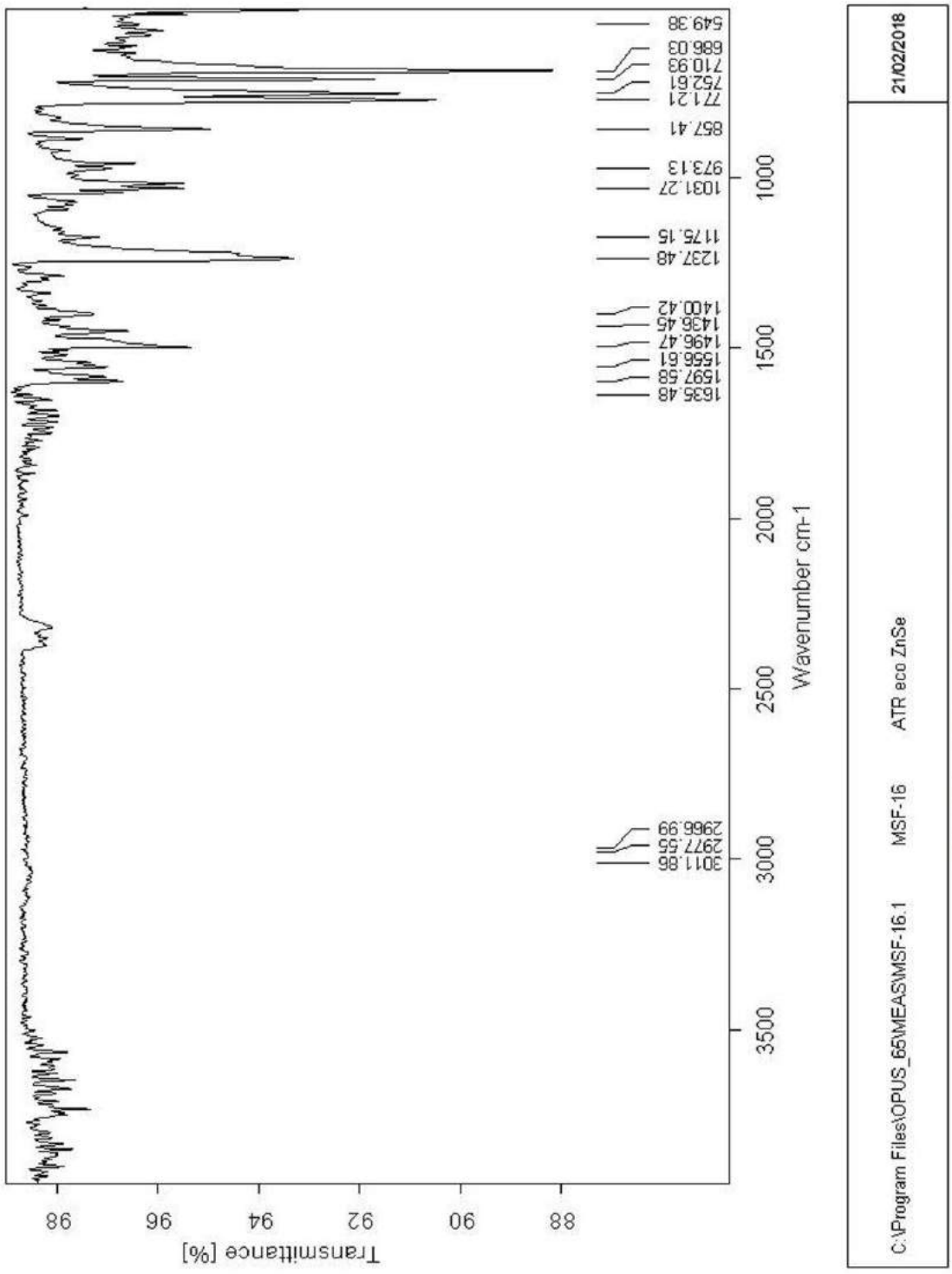
Spectrum 4: IR Spectrometer of intermediate 5a



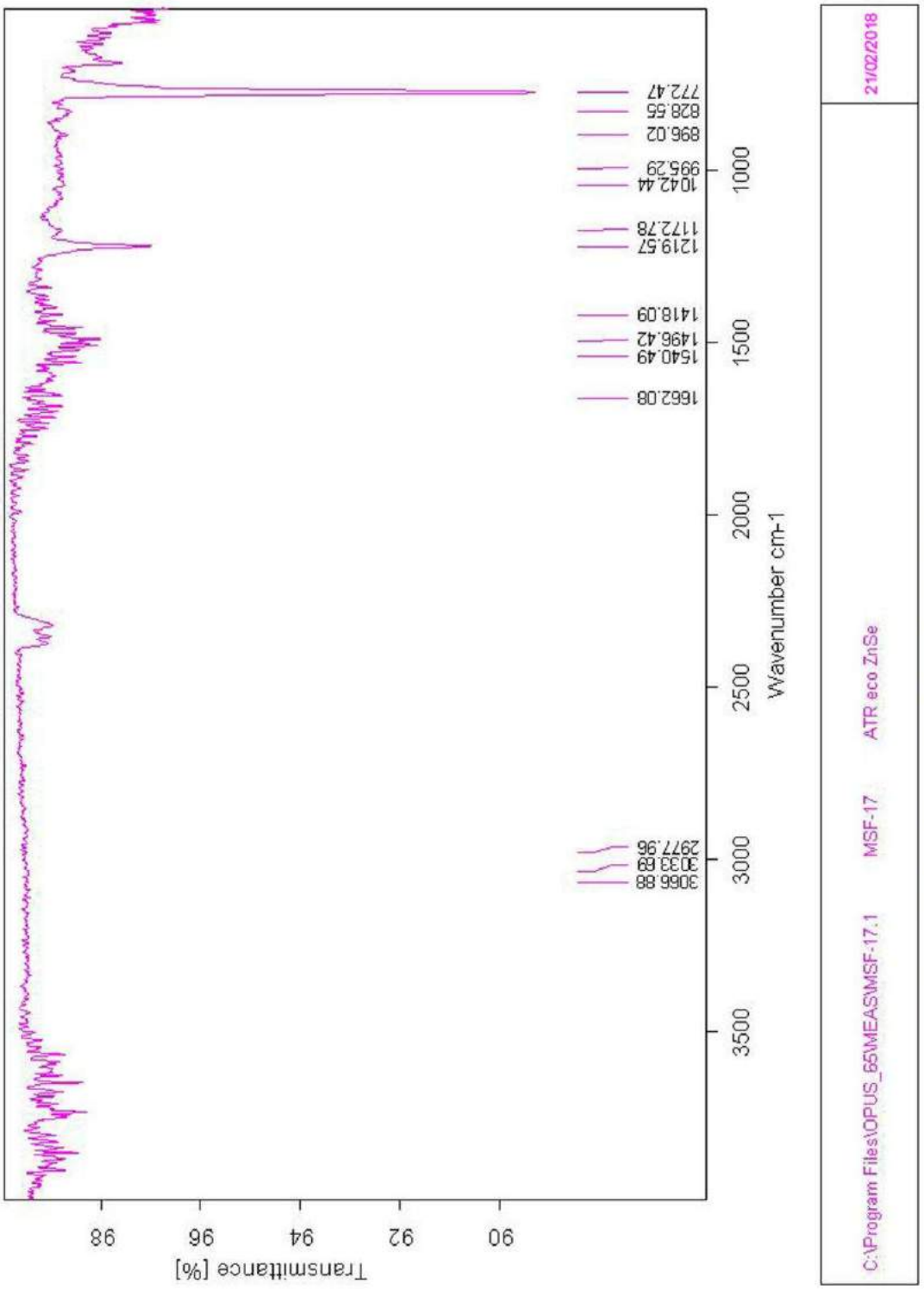
Spectrum 5: IR Spectrometer of intermediate 5b



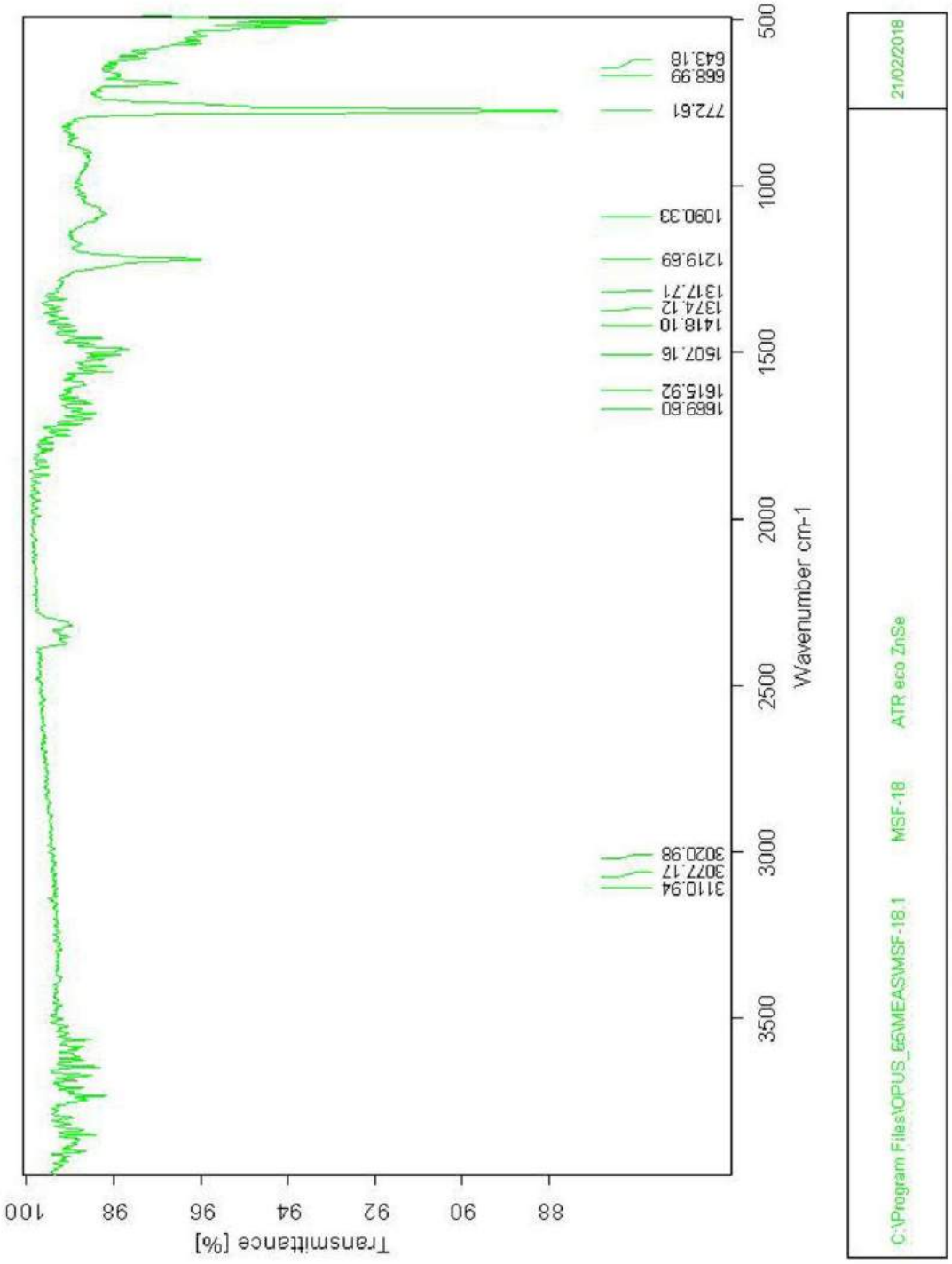
Spectrum 6: IR Spectrometer of intermediate 5c



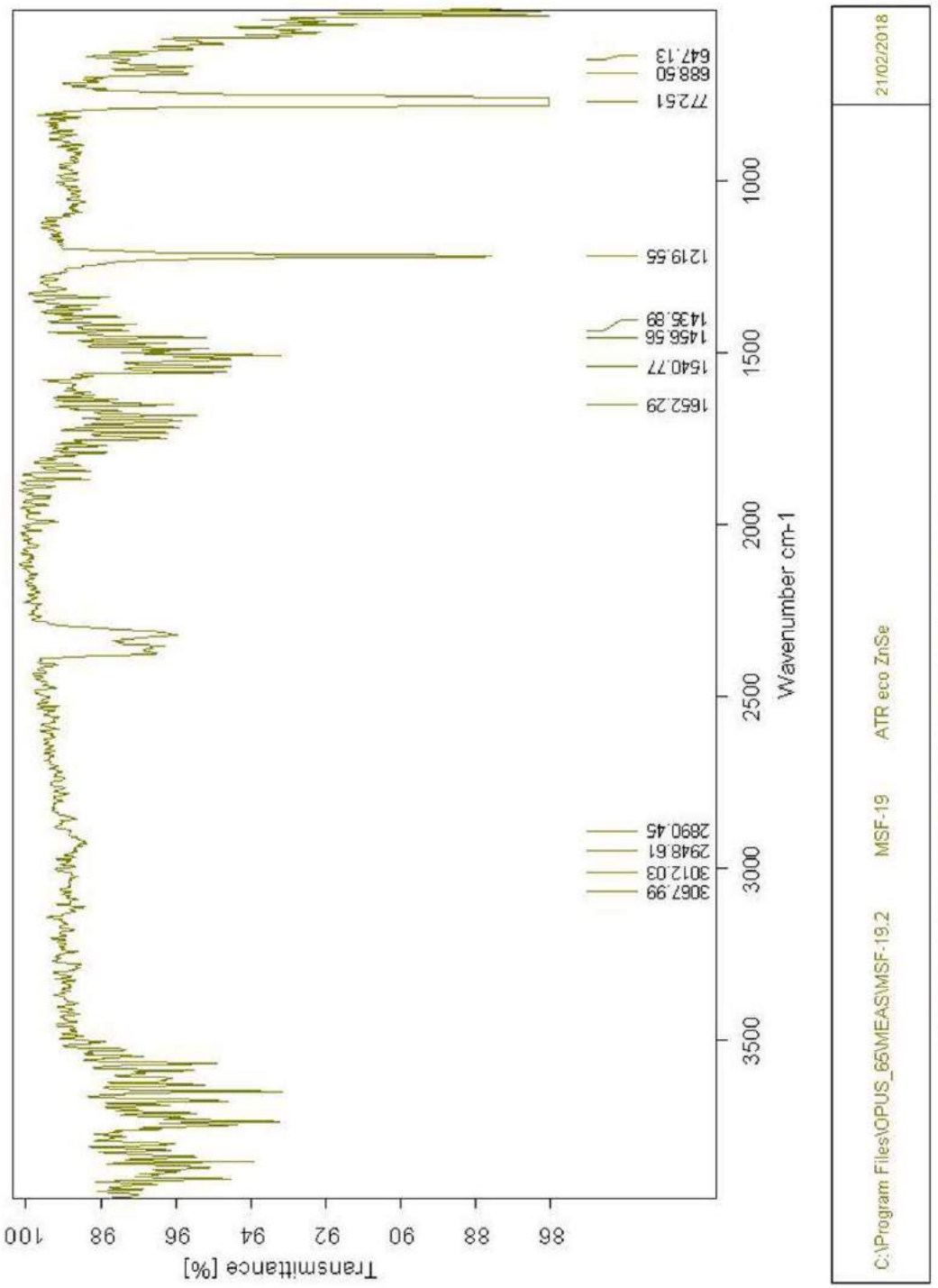
Spectrum 7: IR Spectrometer of synthesized compound 7a



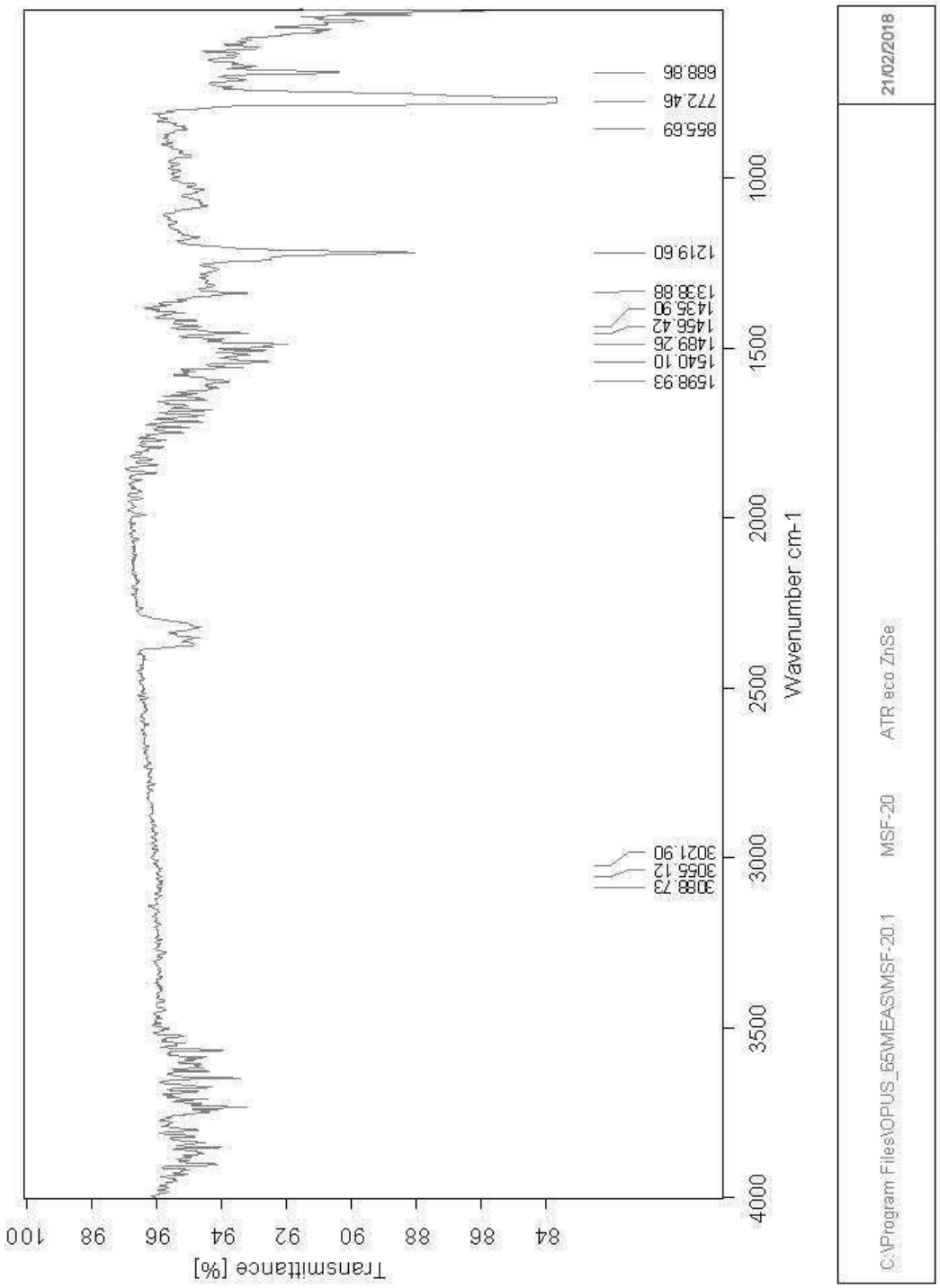
Spectrum 8: IR Spectrometer of synthesized compound 7b



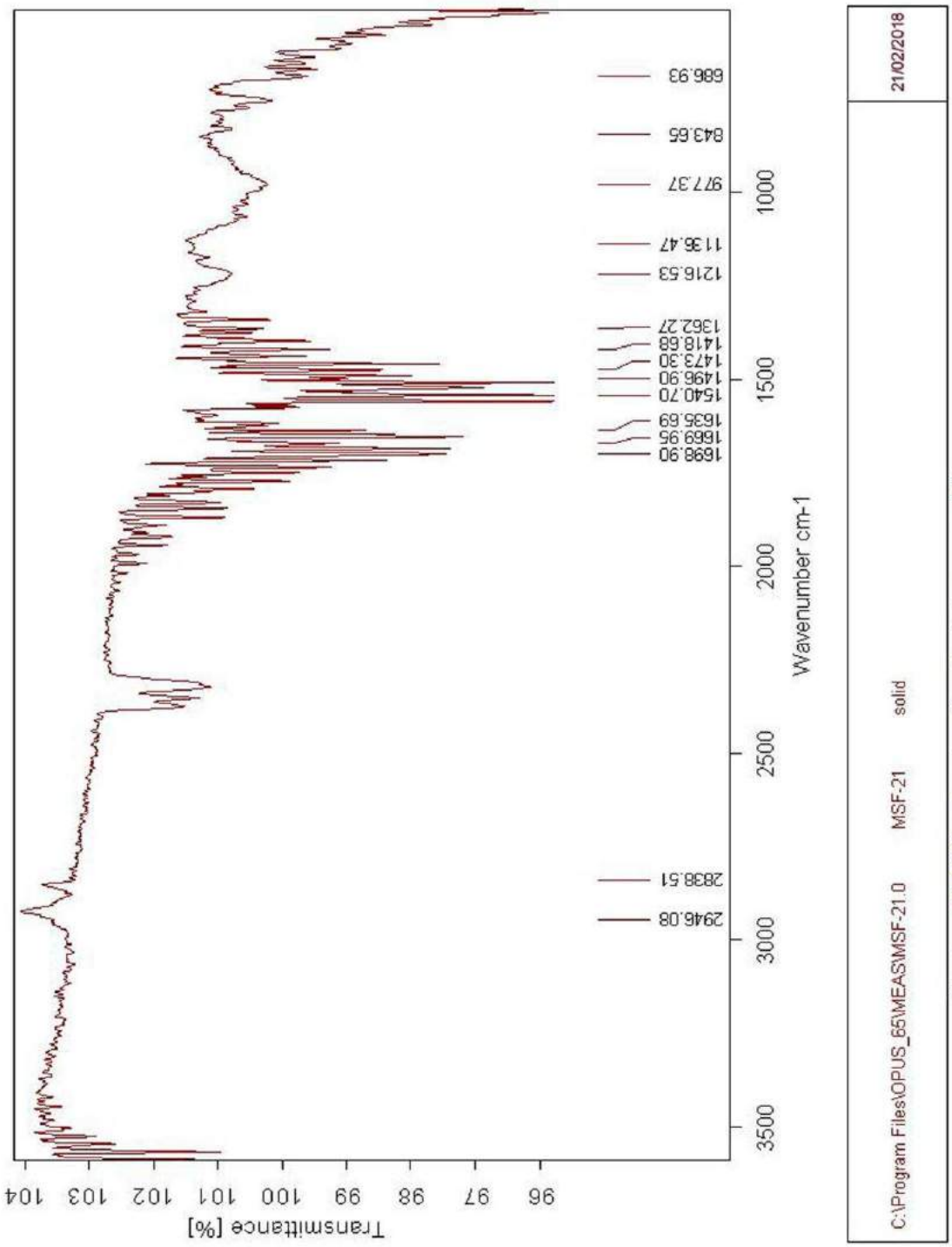
Spectrum 9: IR Spectrometer of synthesized compound 7c



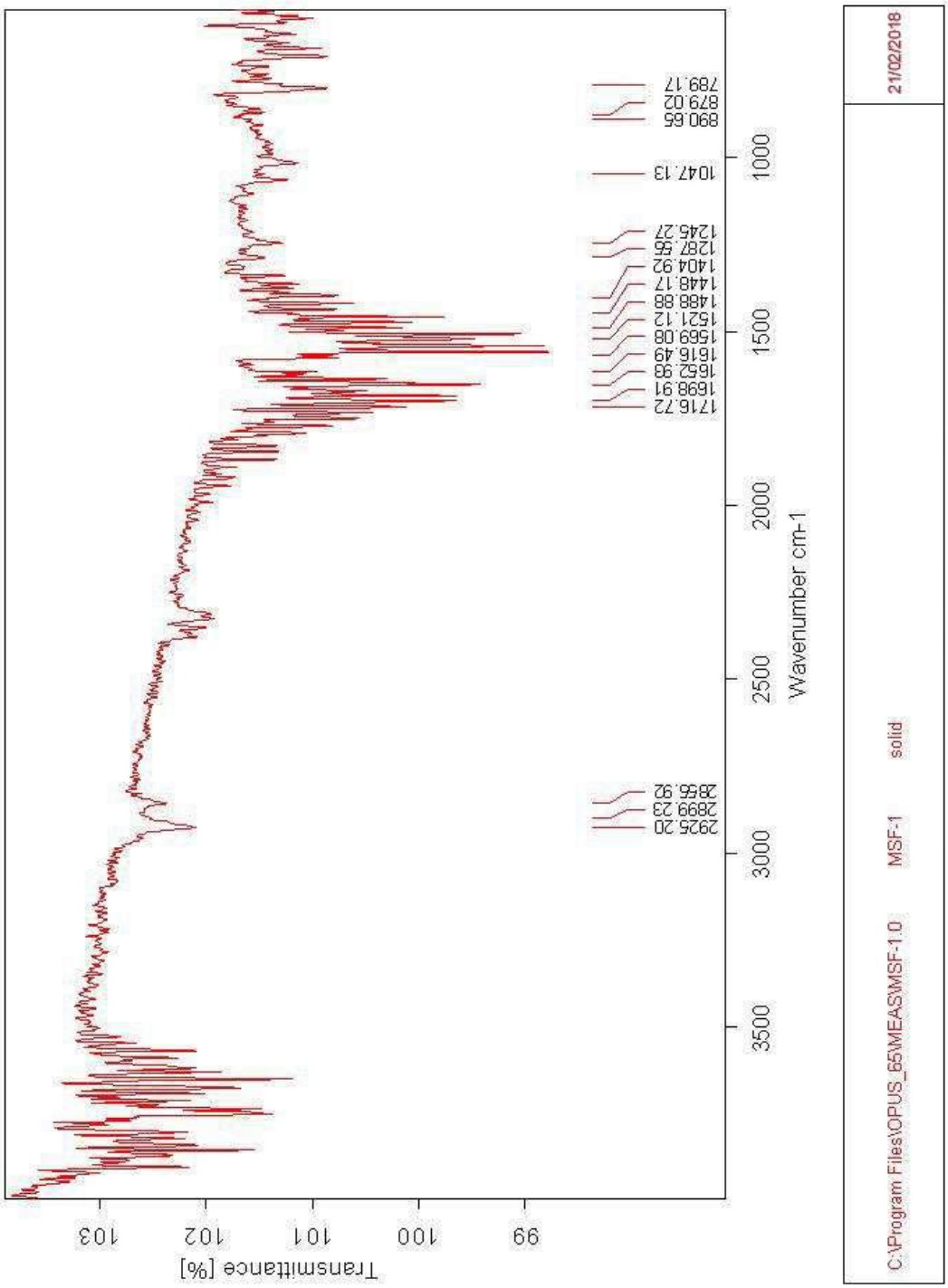
Spectrum 10: IR Spectrometer of synthesized compound 7d



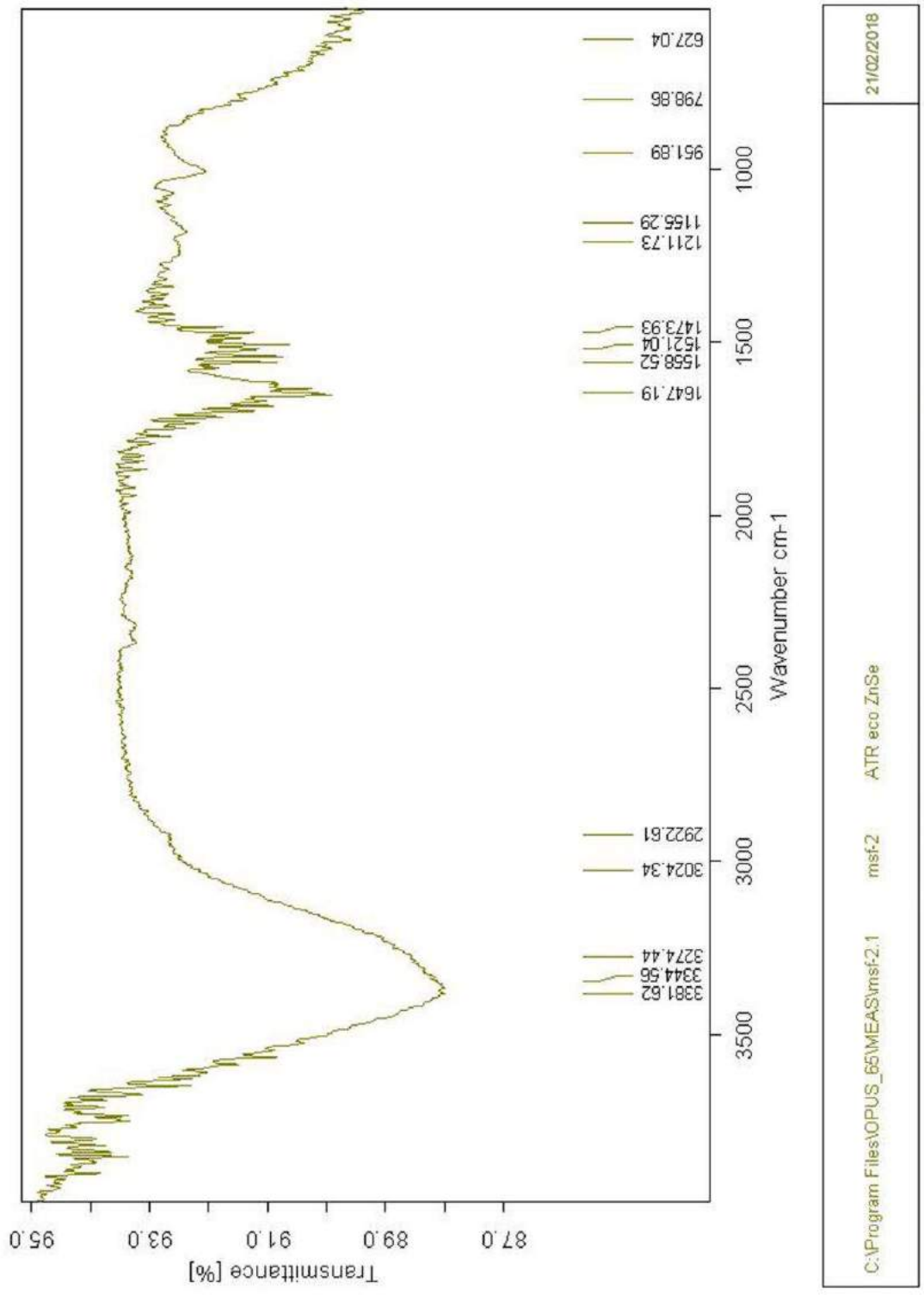
Spectrum 11: IR Spectrometer of synthesized compound 7e



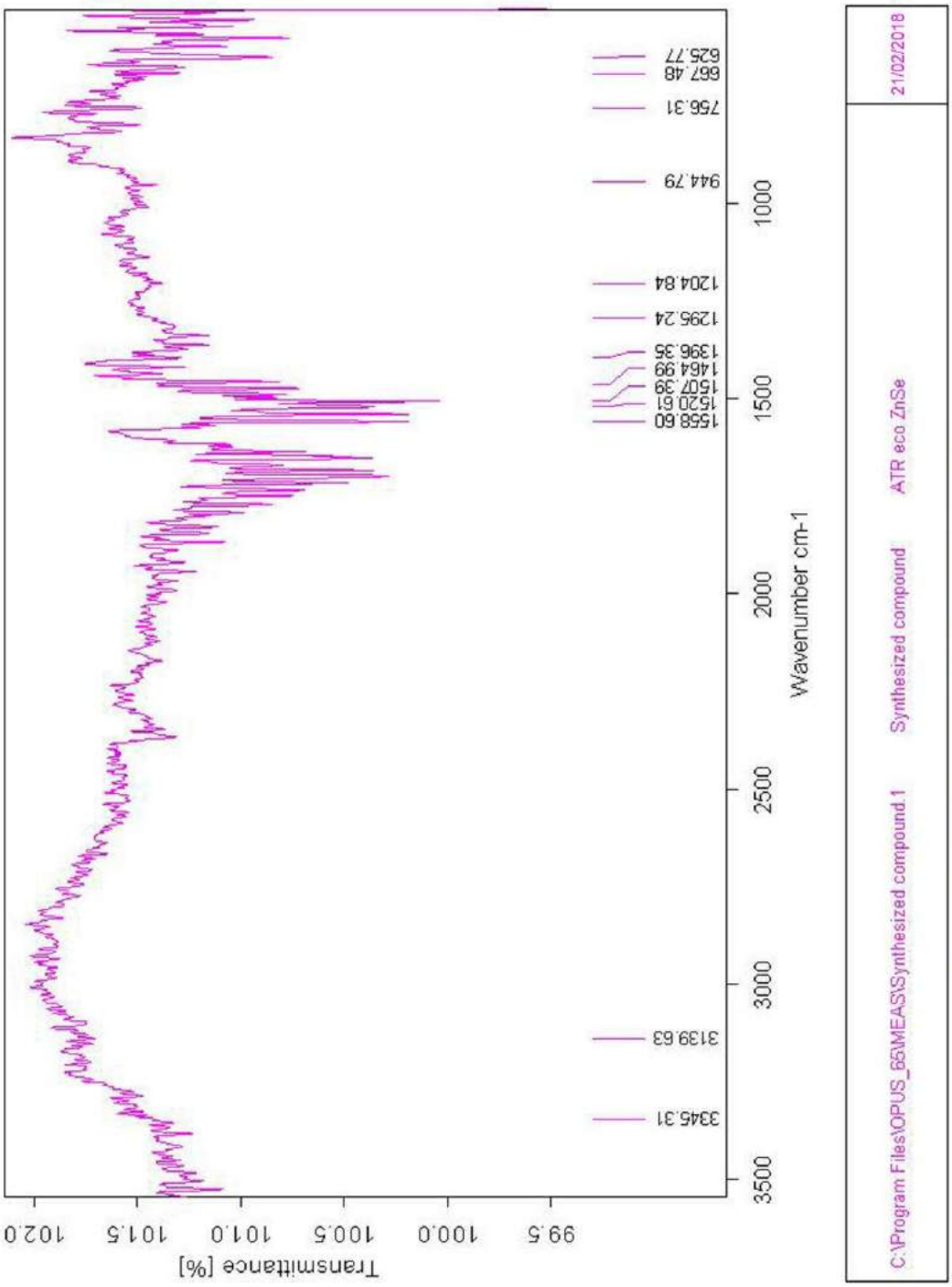
Spectrum 12: IR Spectrometer of synthesized compound 7f



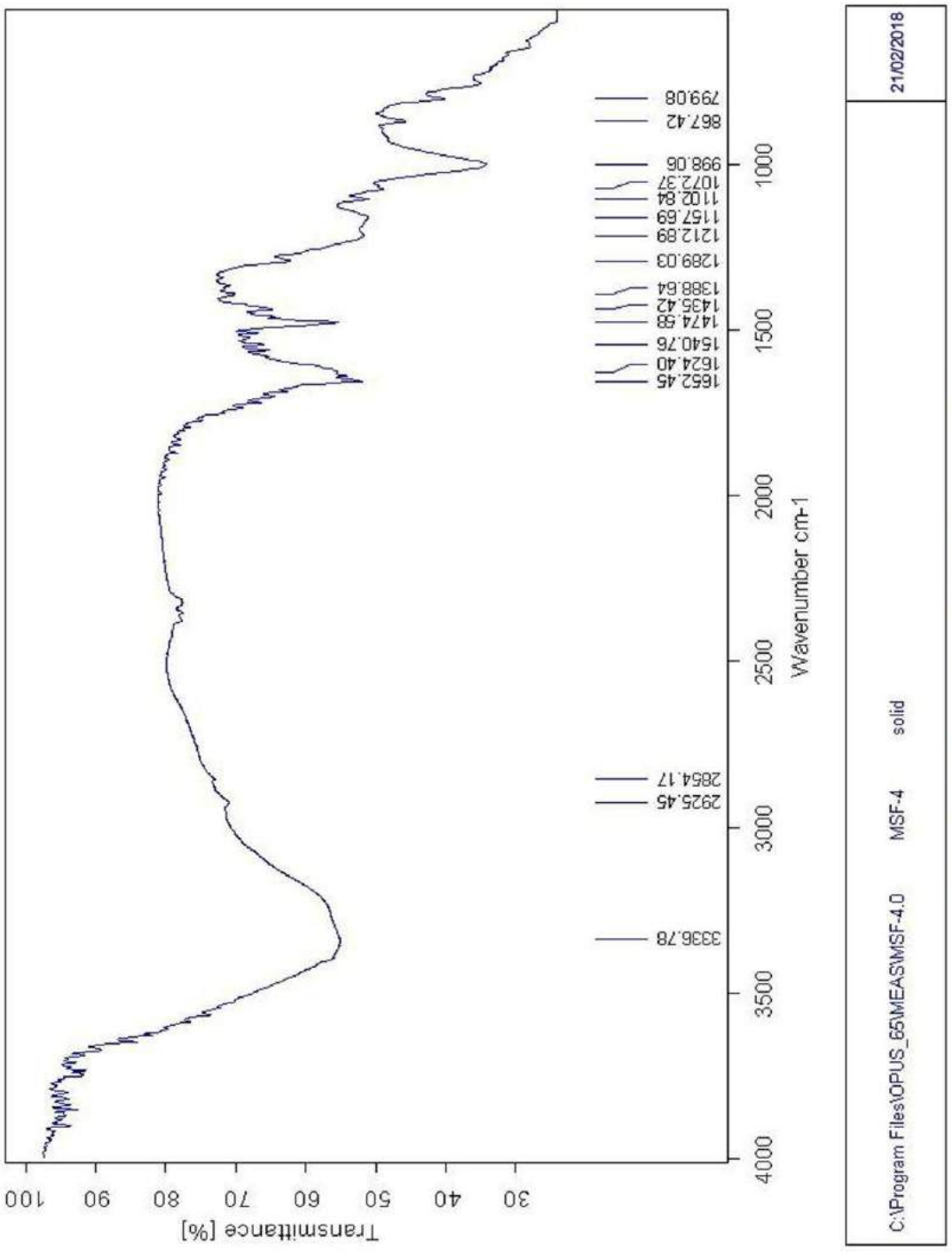
Spectrum 13: IR Spectrometer of synthesized compound 7g



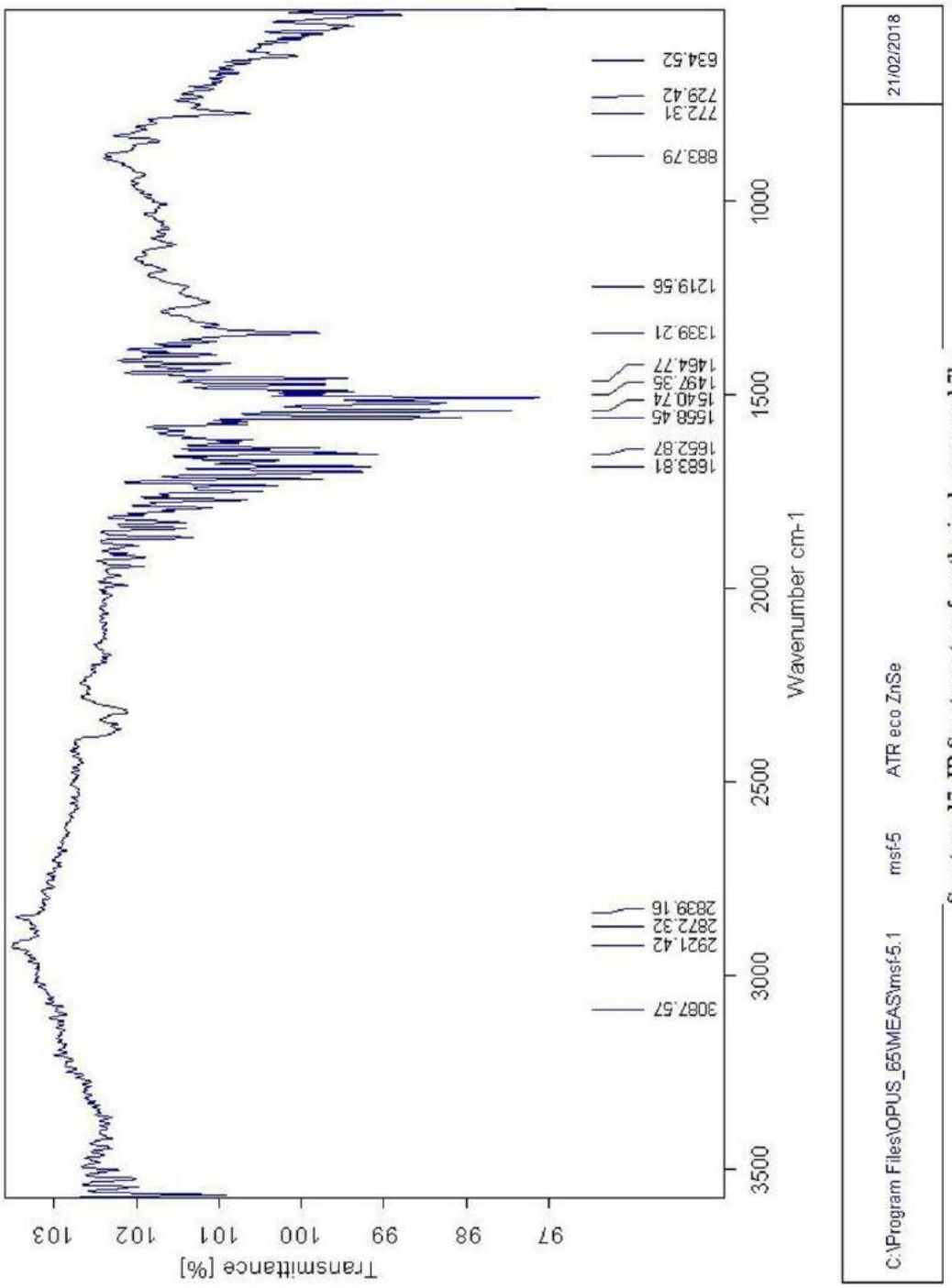
Spectrum 14: IR Spectrometer of synthesized compound 7h



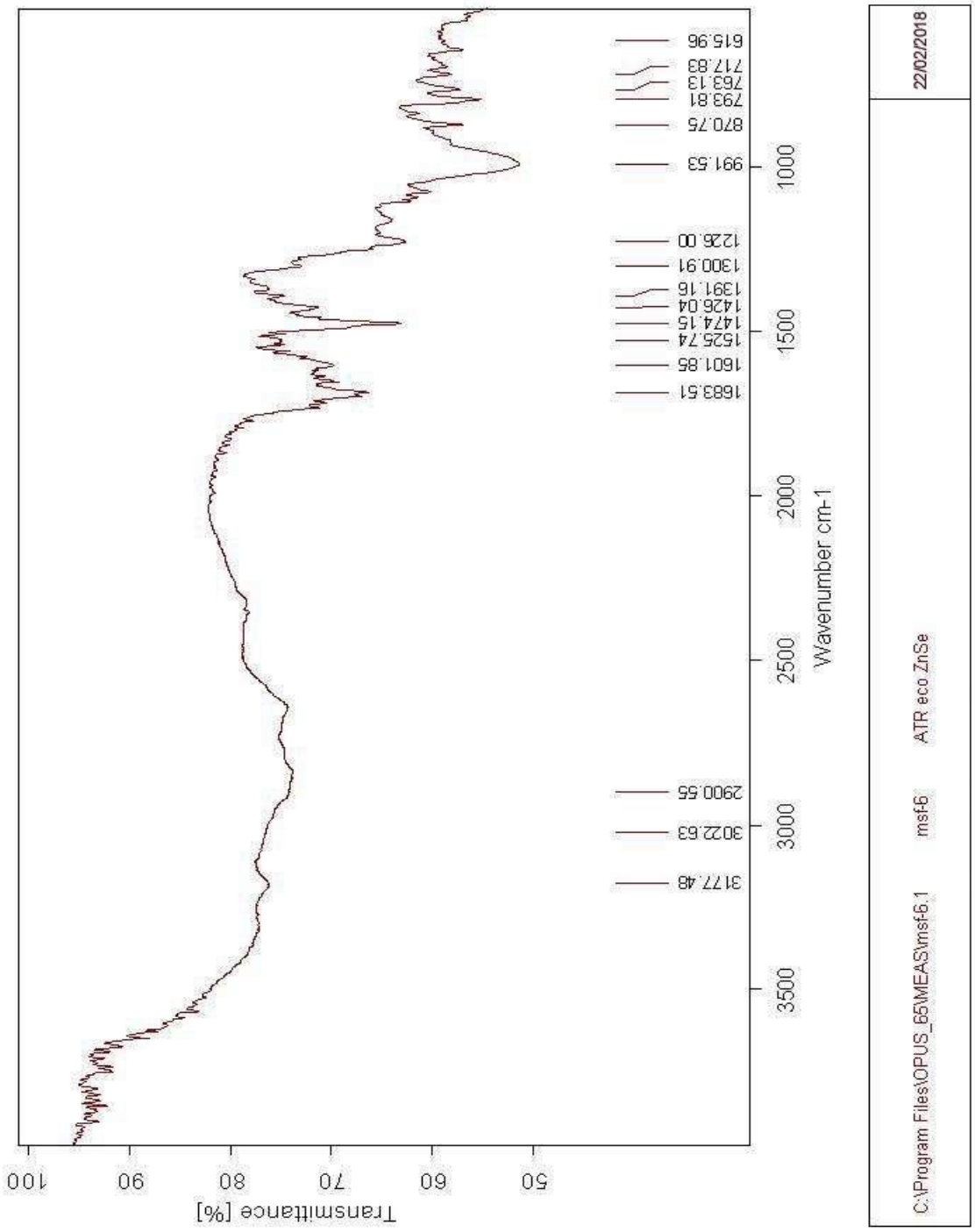
Spectrum 15: IR Spectrometer of synthesized compound 7i



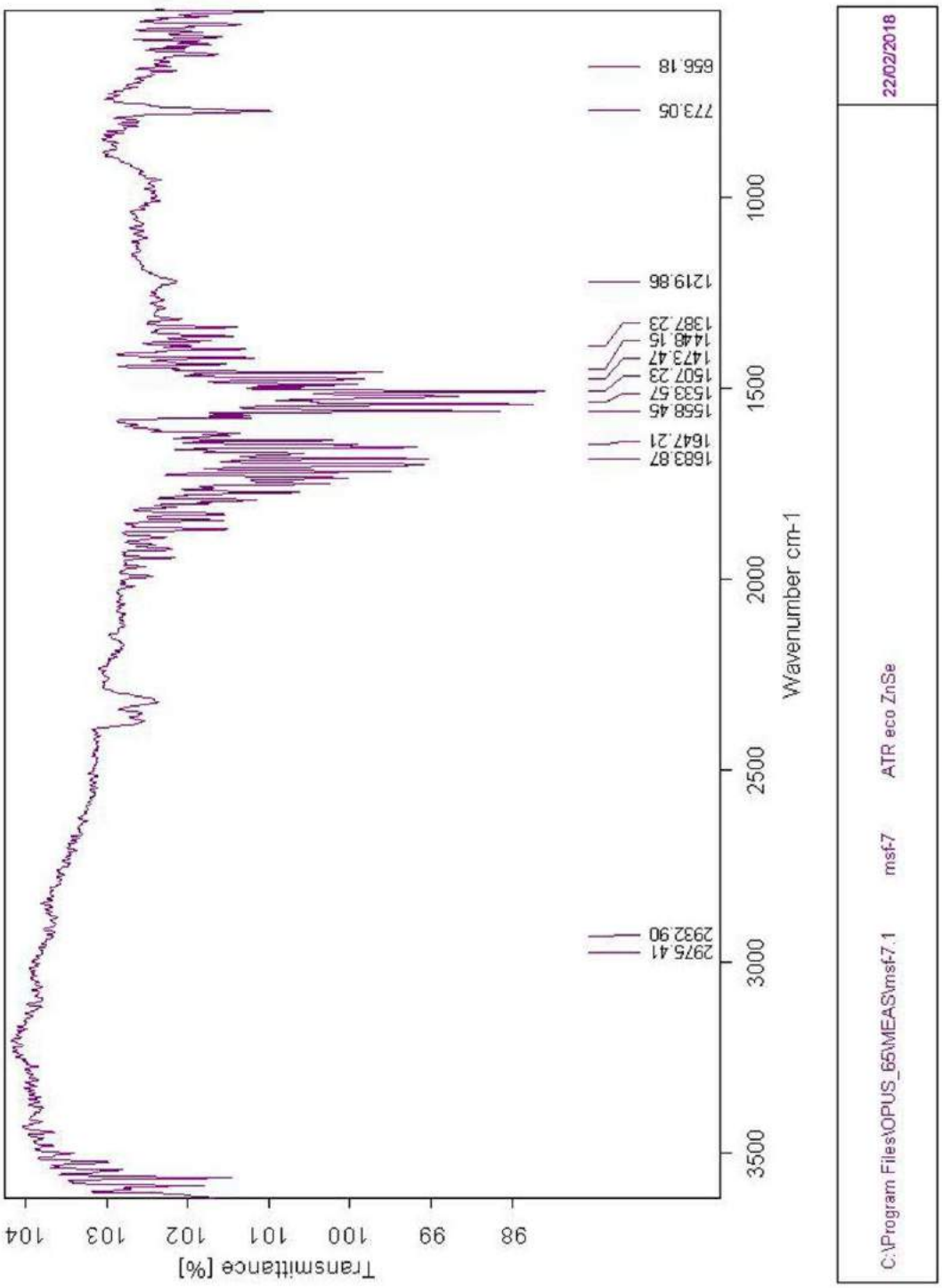
Spectrum 16: IR Spectrometer of synthesized compound 7j



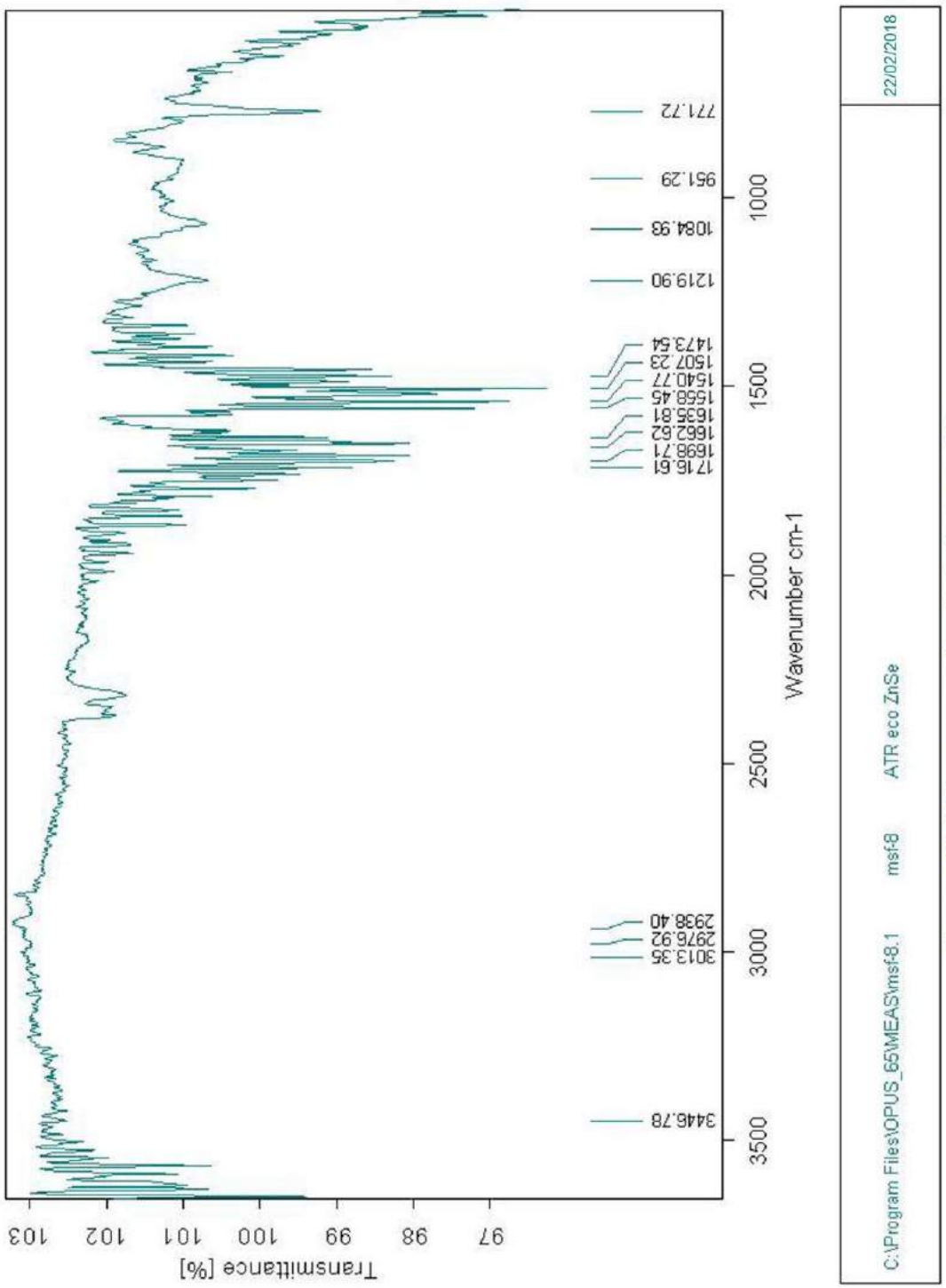
Spectrum 17: IR Spectrometer of synthesized compound 7k



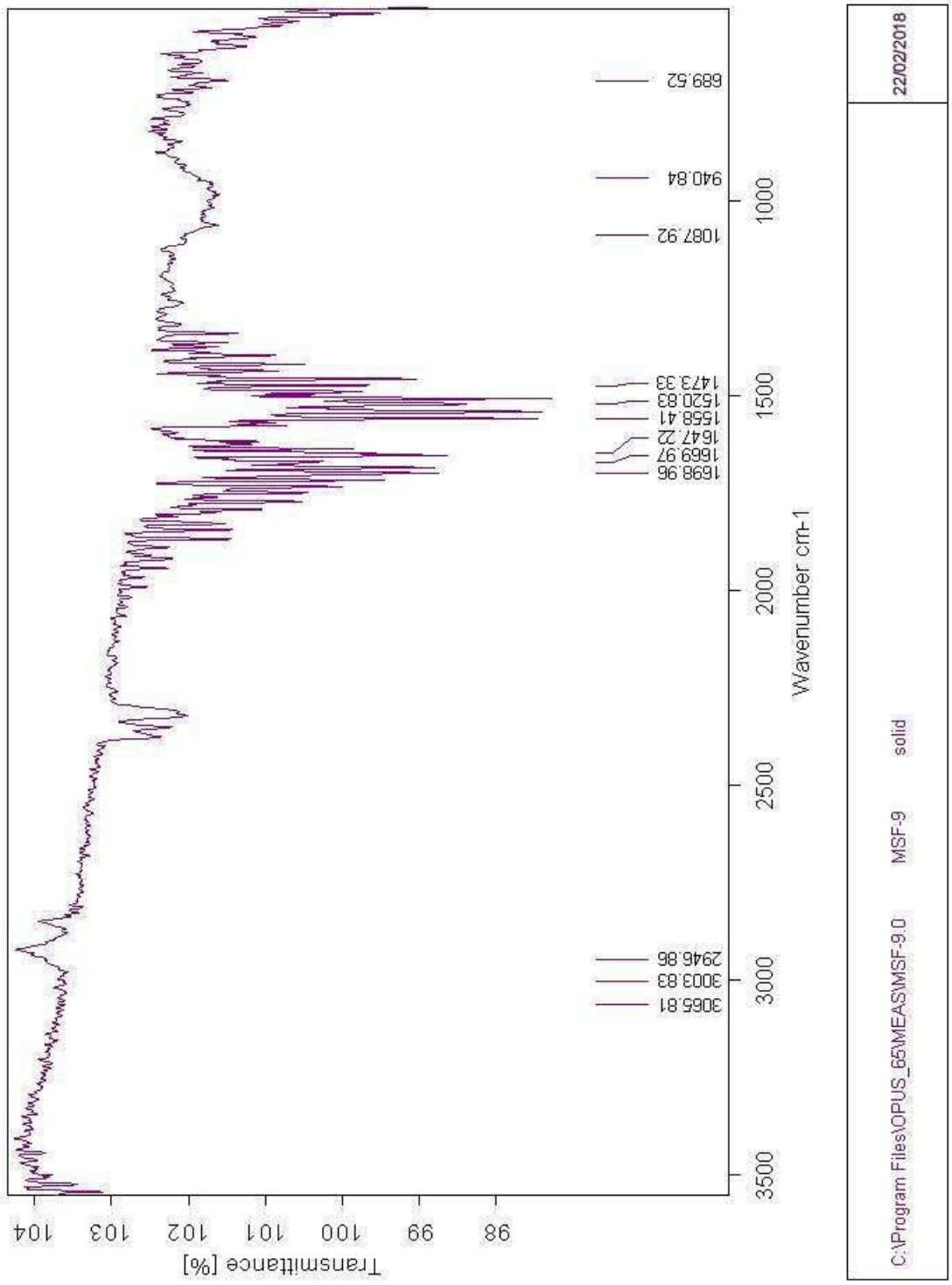
Spectrum 18: IR Spectrometer of synthesized compound 7I



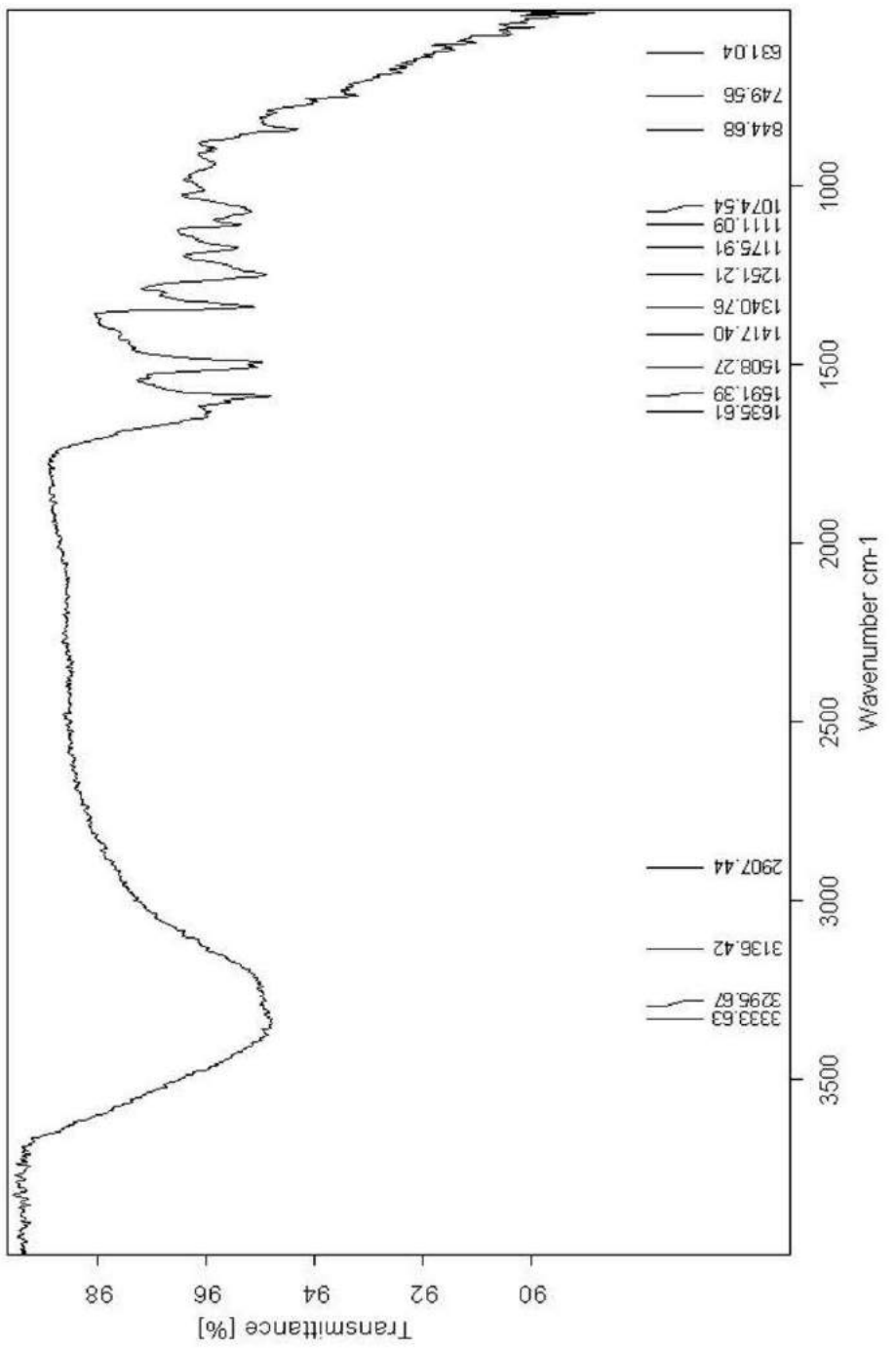
Spectrum 19: IR Spectrometer of synthesized compound 7m



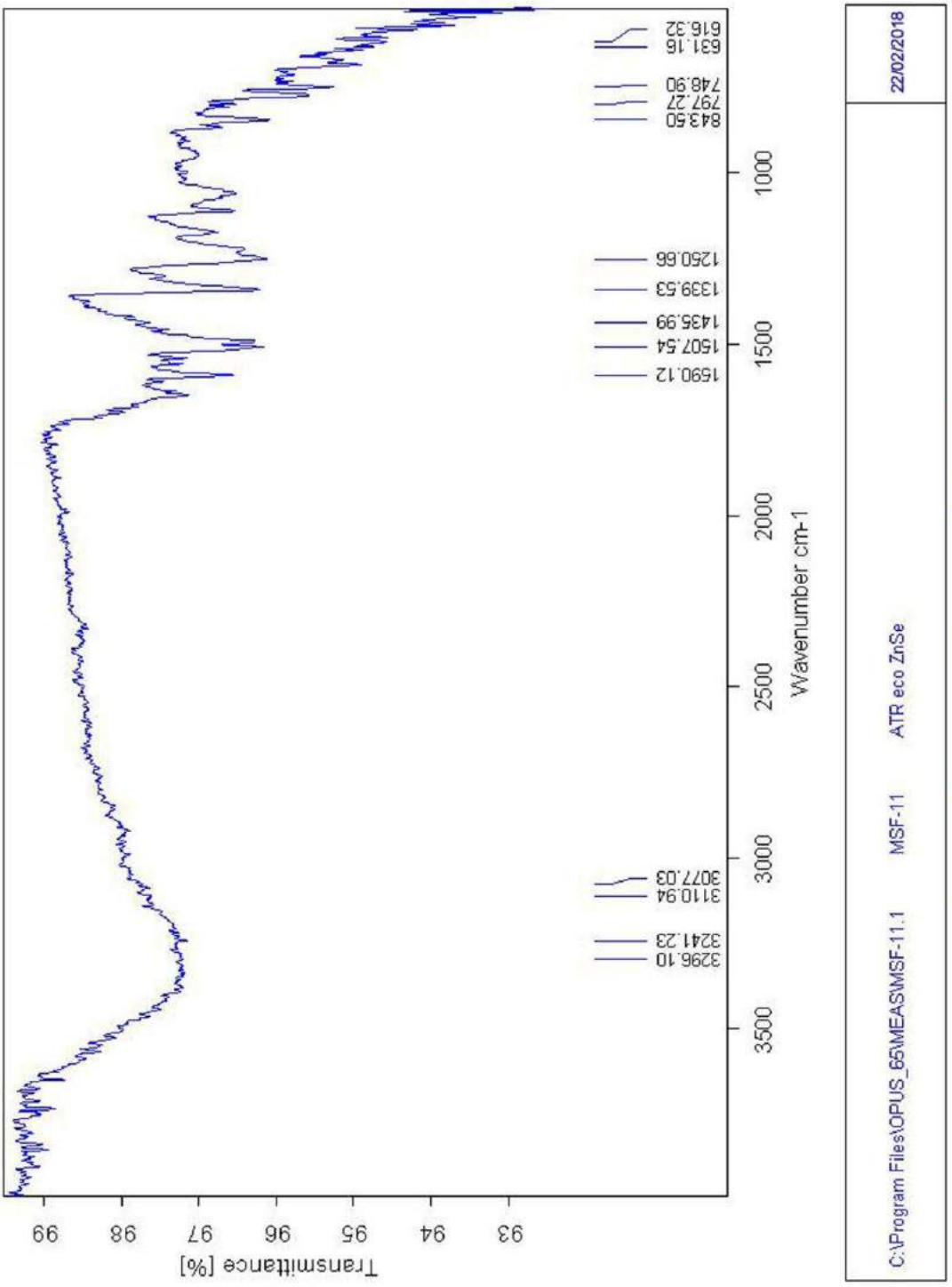
Spectrum 20: IR Spectrometer of synthesized compound 7n



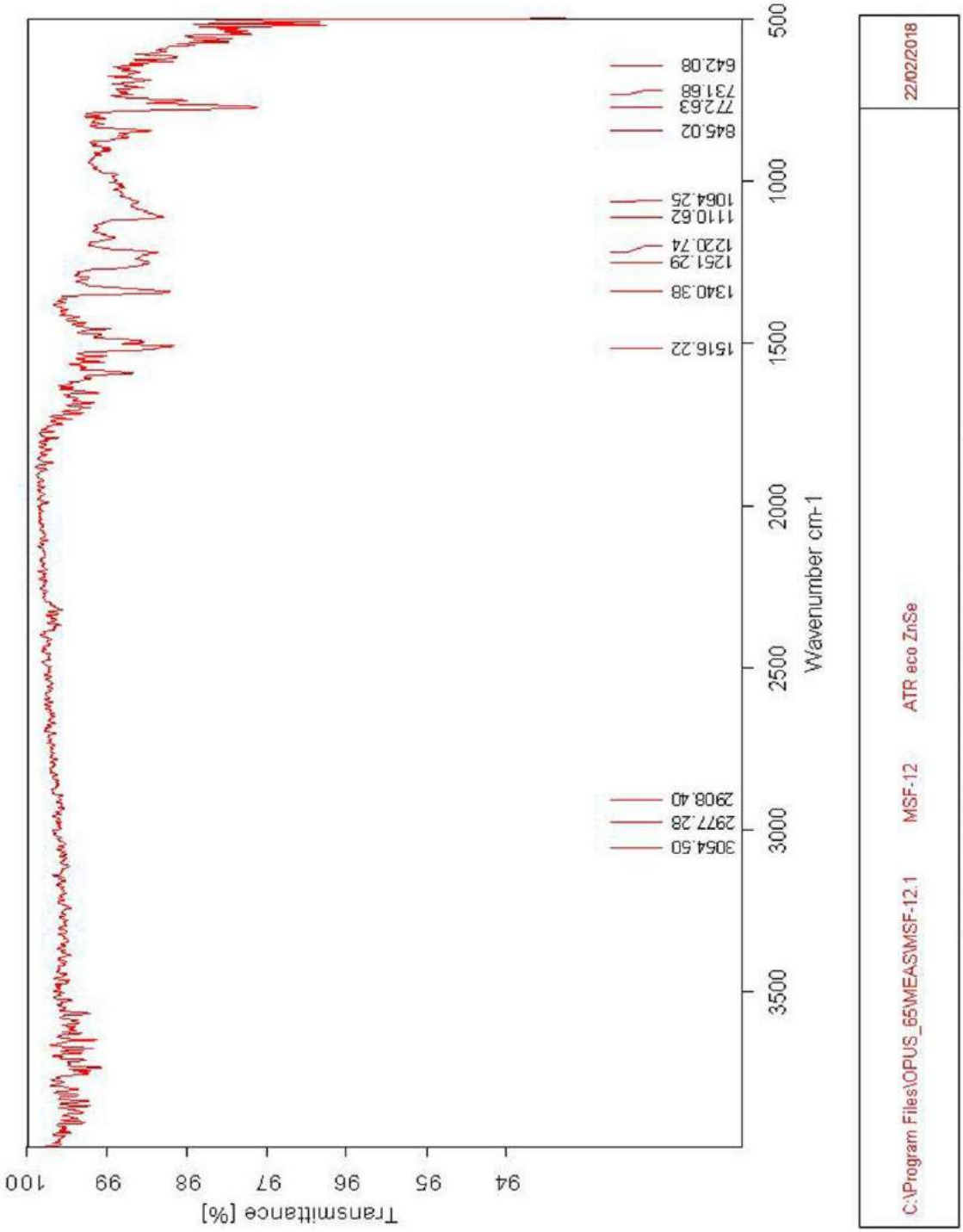
Spectrum 21: IR Spectrometer of synthesized compound 7o



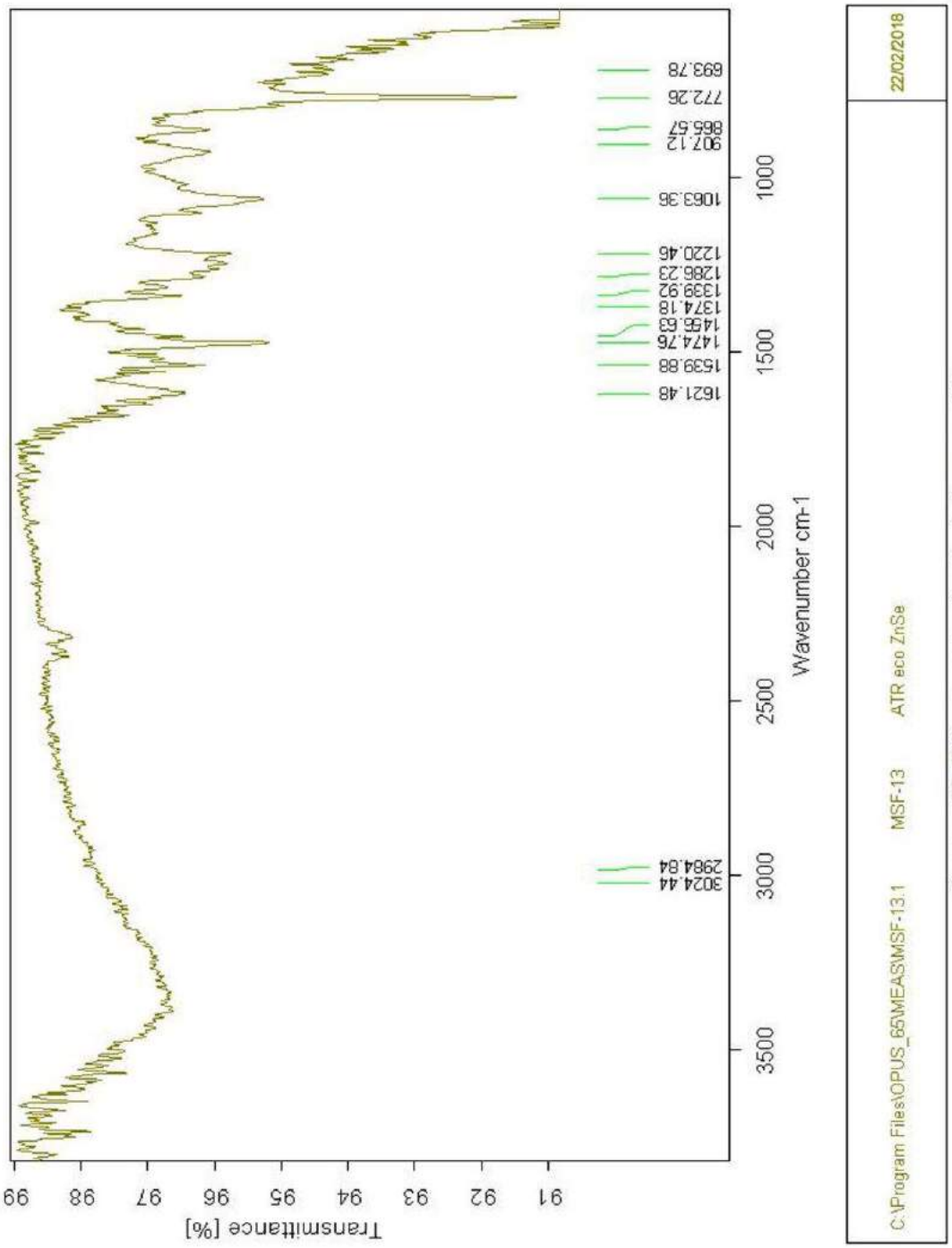
Spectrum 22: IR Spectrometer of synthesized compound 7p



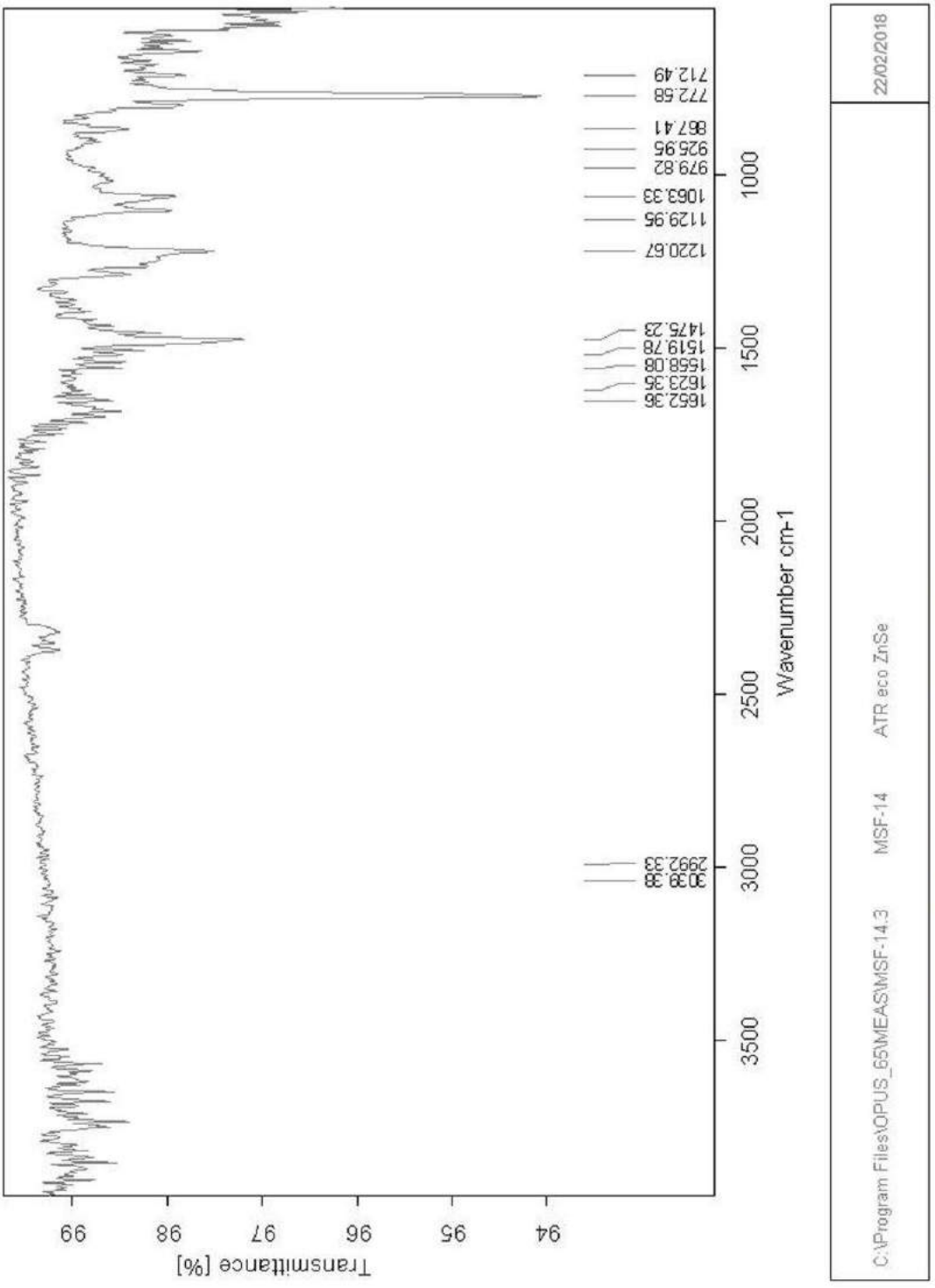
Spectrum 23: IR Spectrometer of synthesized compound 7q



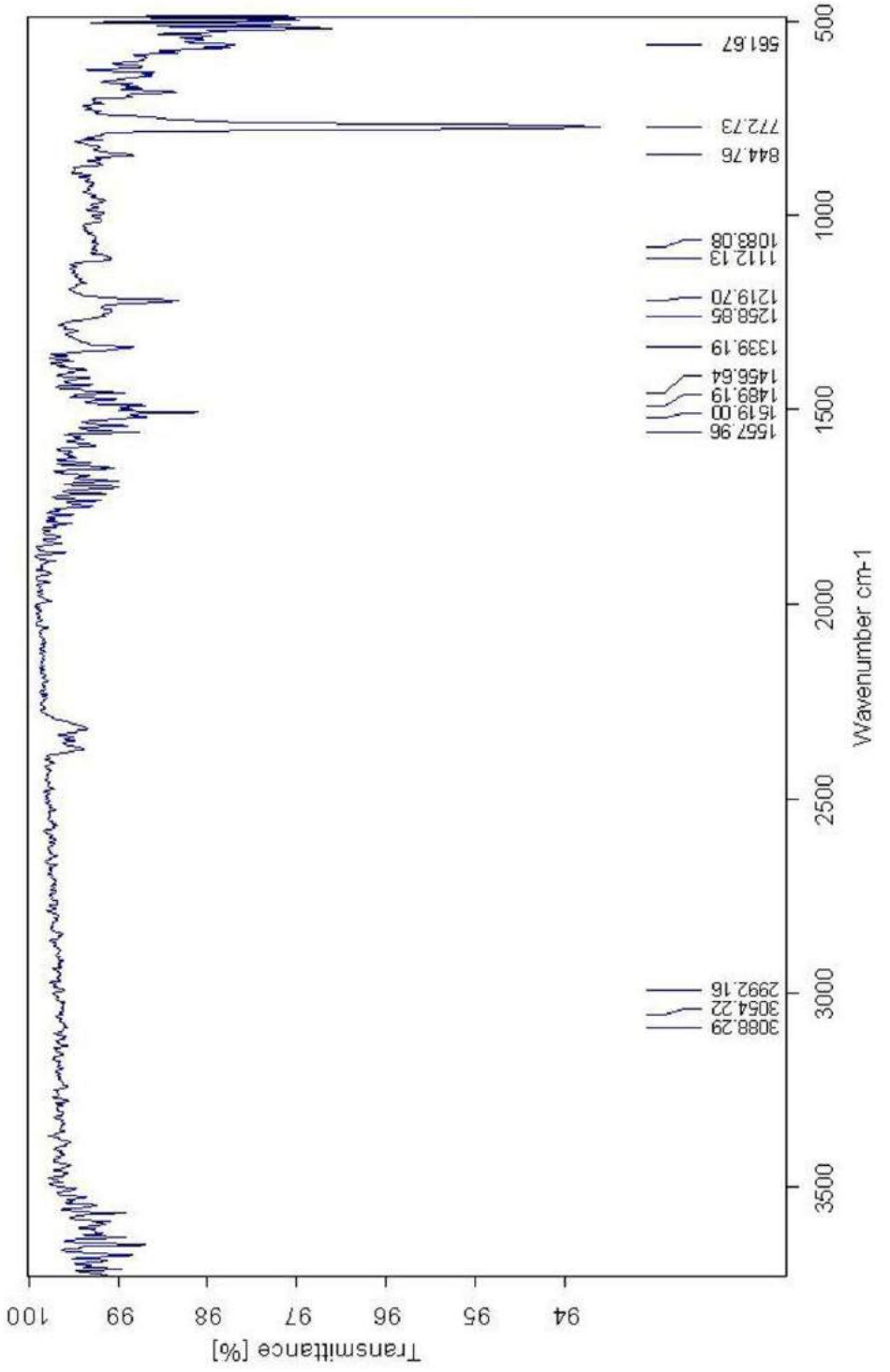
Spectrum 24: IR Spectrometer of synthesized compound 7r



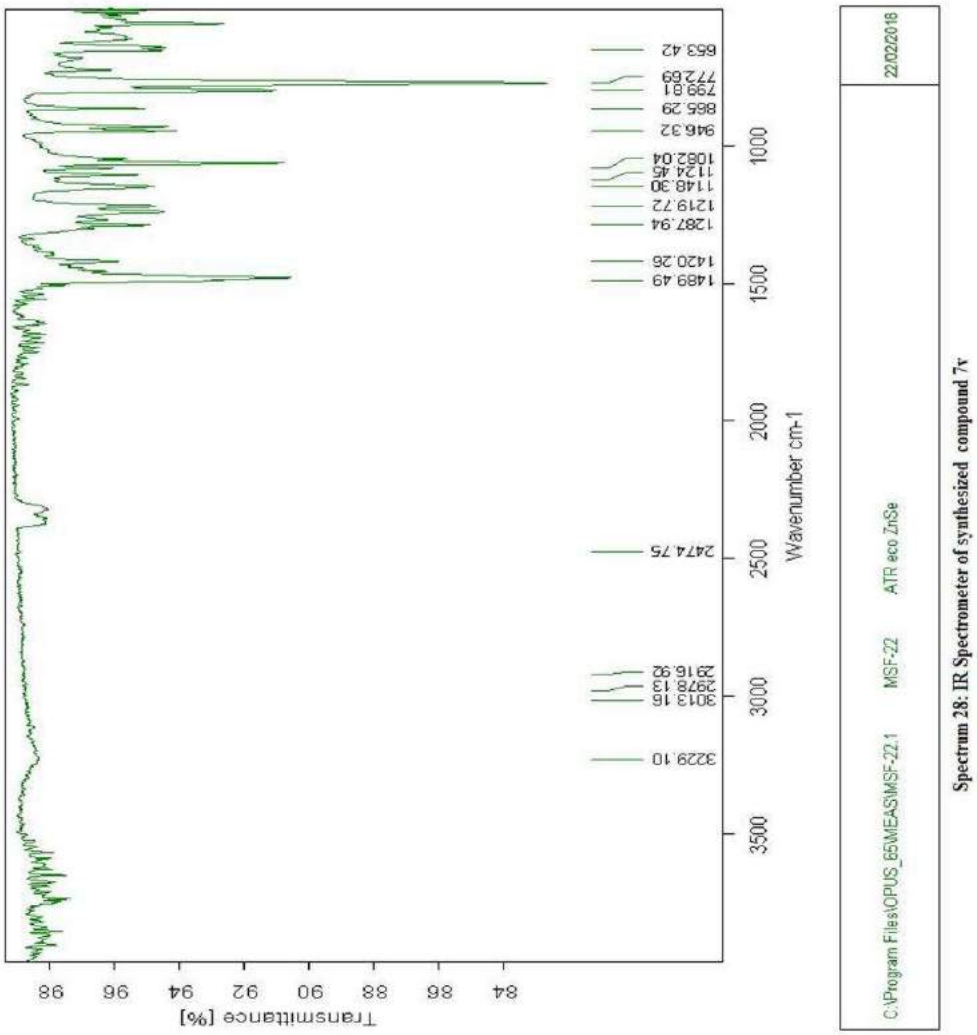
Spectrum 25: IR Spectrometer of synthesized compound 7s



Spectrum 26: IR Spectrometer of synthesized compound 7t



Spectrum 27: IR Spectrometer of synthesized compound 7u



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