

PHYTOCHEMICAL SCREENING OF VOLATILE CONSTITUENTS FROM AERIAL PARTS OF *MURRAYA PANICULATA*

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Abstract

The volatile components of the aerial parts of *Murraya paniculata* have been analyzed by GC and GC-MS, revealing the presence of forty eight compounds. Identifications were made by their respective characteristic mass fragmentation pattern, with the help of NIST mass spectral search program and GC-MS Library. These identities were further authenticated by comparison of their calculated Kovat's retention indices (RI) with those cited in literature.

Key words: *Murraya paniculata*, Rutaceae, Volatile constituents, GC, GC-MS.

Introduction

The *Rutaceae* is a large family, comprised about 150 genera and 1200 species (wild and cultivated) distributed in the tropical and sub tropical regions of South East Asia, Mediterranean countries, North America, Australia, and South Africa (Bhatterjee, 2000). There are 11 genera and 27 species found in Pakistan (Bhatterjee, 2000). *Murraya paniculata*, is commonly known as orange jasmine, belongs to family *Rutaceae* (Pullaiah, 2006). It is also known as mock orange, stain wood and honey bush. It is a densely, foliaceous, evergreen tree, up to 5 m tall. It is native of South-East Asia, Sri Lanka, Malaysia, Australia, outer Himalayas and South India (Pahang & Lumpur, 2002). The leaves of *M. paniculata* are stimulant and astringent, used for diarrhea and dysentery. Leaves and root barks are used against rheumatism, cough and hysteria (Mondal *et al.*, 2001). The powdered leaves are used to apply on fresh cuts, and a decoction of the leaves is drunk in dropsy. The leaves possess antibiotic activity against *Micrococcus pyogenes*, var. *aureus* and *Escherichia coli* (Pahang & Lumpur, 2002). Previous phytochemical studies on *Murraya paniculata* have resulted in the isolation of several coumarins (Sumayya *et al.*, 2011), flavonoids (Kinoshita & Firman, 1996) and alkaloids (Fauvel *et al.*, 1978).

Materials and Methods

Plant collection and identification: The aerial part of *M. paniculata* was collected from Karachi University and identified by Dr. Rubina Dawar, Plant Taxonomist, Department of Botany, University of Karachi, voucher specimen has been deposited in the Karachi University Herbarium (voucher # 67974).

Extraction and isolation: The freshly collected plant material of *M. paniculata* was shade dried (1.5 kg), ground and extracted with ethanol (3 X 15 L, 10 days each). The combined extract was evaporated under reduced pressure to obtain a gummy residue (276 g), which was suspended in water (1.0 L) and successively extracted with *n*-hexane (35 g), ethyl acetate (60 g) and *n*-butanol (120 g). The ethyl acetate (60 g) soluble fraction was subjected to column chromatography over silica gel and eluted with *n*-hexane, *n*-hexane-ethyl acetate and ethyl acetate-methanol in increasing order of polarity to collect thirteen fractions (A-M). The sub-fraction A (*n*-hexane, 12 g) was re-chromatographed over silica gel using *n*-hexane: EtOAc in increasing order of polarity to obtain five sub-fractions (A1-A5). The sub-fractions A-1 to A-3 were analyzed through GC and GC-MS resulting in the identification of 48 compounds. Compounds 1 to 16 from sub-fraction A-1, 17 to 35 from sub-fraction A-2 and 36 to 48 from sub-fraction A-3 (Table 1).

GC and GC-MS analysis: GC analysis was executed on Shimadzu-GC-9A gas chromatograph with installed SPB-5 capillary column (30m × 0.53 mm ID; 0.3 μdf). Signals corresponded to elution were recorded by FID detector at 220 eV. Nitrogen gas was used as carrier gas at 1.0 ml/min flow rate, split ratio was set as 1:30 and injector temperature was fixed at 240°C. The column temperature was maintained at 50°C for the first 5 min and then ascended upto 235°C at rate of 5°C/min. GC-MS spectra were recorded on Hewlett-Packard 5890 gas chromatograph, coupled with a Jeol, JMS-HX 110 mass spectrometer with injector at 270°C while splitting ratio set at 1:30. GC-MS analyses were carried out on an equivalent column HP-5 (25m × 0.22mm and 0.25 μmdf) with identical gradient thermal ramping and temperature parameters as mentioned above for GC analysis.

Table 1. Qualitative and quantitative data of various compounds indentified from aerial parts of *Murraya paniculata*.

No. Compounds identified	Formula	RI*	% Conc.	Some important peaks matched m/z (% Abundance)
1. 4-(5-Methyl-2-furanyl), 2-butanone	C ₉ H ₁₂ O ₂	1164	1.82	152 (35), 137 (5), 109 (19), 95 (35), 83 (12), 71 (11), 55 (14), 43 (100).
2. 2-Methyl, 5-isopropylphenol	C ₁₀ H ₁₄ O	1286	0.20	150 (31), 135 (100), 117 (35), 91 (30), 67 (22), 44 (32).
3. 3-Methoxy, 4-hydroxy benzaldehyde	C ₈ H ₈ O ₃	1398	0.38	152 (90), 151 (100), 123 (28), 109 (26), 80 (15), 43 (30).
4. Spathulenol	C ₁₅ H ₂₄ O	1572	2.10	220 (18), 205 (23), 162 (66), 159 (25), 121 (38), 107 (60), 69 (42).
5. Tetrahydrodehydrogeijerin	C ₁₅ H ₁₈ O ₄	1692	20.69	262 (2), 205 (100), 175 (6), 162 (3), 71 (2), 55 (2).
6. †	C ₁₅ H ₂₂ O ₄	1878	1.50	251 (2), 192 (11), 167 (7), 151 (6), 149 (7), 123 (100), 105(10), 95(14).
7. Hexadecanoic acid	C ₁₆ H ₃₂ O ₂	1980	2.22	256 (28), 227 (10), 213 (22), 157 (23), 129 (42), 85(44), 73 (100).
8. 6-Amino, 4-oxochromen-2-carboxylate	C ₁₂ H ₁₁ NO ₄	1986	27.03	233 (94), 205 (100), 135 (4), 132 (4), 107 (2), 104 (2), 77 (4).
9. 2,4-Dihexyl, 7,7-dimethyl, 1,3,5-cycloheptatriene	C ₂₁ H ₃₆	2036	6.43	288 (3), 273 (2), 219 (5), 203 (100), 189 (3), 175 (5), 160 (3), 147 (2).
10. 7-Methoxy, 6(3-methyl-2-oxobutyl) 2H-1-benzopyran-2-one	C ₁₅ H ₁₆ O ₄	2238	20.67	260 (18), 190 (100), 175 (8), 161 (6), 131 (21), 118 (2), 103(5), 71(12).
11. 3-Hydroxy, 3-nonyl-1H-quinoline-2,4-dione	C ₁₈ H ₂₅ NO ₃	2352	6.83	303 (9), 258 (61), 229 (18), 215 (55), 199 (21), 190 (100), 131 (29).
12. Bis(2-ethylhexyl) phthalate	C ₂₄ H ₃₈ O ₄	2504	9.55	279 (4), 167 (28), 149 (100), 113 (6), 104 (3), 83 (5), 71 (13), 57 (15).
13. 6-Methoxy, 2-vinyl, 9-[3-deoxyribofuranosyl] purine	C ₁₃ H ₁₆ N ₄ O ₄	2598	5.88	292 (2), 247 (10), 233 (28), 219 (41), 205 (100), 189 (10), 175 (16).
14. ††	C ₃₂ H ₄₄	2812	9.49	429 (15), 428 (100), 289 (9), 245 (4), 231(2), 219(26), 205(98), 85(12).
15. 8-Amino, 6-methoxy, 4-methyl, 5-[n-nonoxy] quinoline	C ₂₀ H ₃₀ N ₂ O ₂	2892	2.57	330 (4), 203 (100), 188 (4), 175 (6), 160 (8), 132 (3), 119 (2), 89 (2).
16. Myristoylolean-12-en-28-ol	C ₄₄ H ₇₆ O ₃	4552	3.27	393 (2), 353 (3), 313 (4), 286(6), 274 (2), 233 (3), 203 (100), 190 (16).
17. 3-methoxy, 4-Hydroxy benzaldehyde	C ₈ H ₈ O ₃	1398	0.21	152 (100), 151 (80), 123 (20), 109 (17), 81 (22), 43 (30).
18. Dimethyl nonanedioate	C ₁₁ H ₂₀ O ₄	1508	1.08	186 (8), 185 (58), 152 (84), 143 (60), 124 (36), 111 (78), 83 (100).
19. Ethyl 4-hydroxy, 3-methoxybenzoate	C ₁₀ H ₁₂ O ₄	1576	0.24	196 (42), 181 (6), 168 (21), 151 (100), 136 (4), 123 (14), 108(9), 79(4).
20. 4,7-Dimethoxy, 5-(2-propenyl), 1,3-benzodioxole	C ₁₂ H ₁₄ O ₄	1686	1.27	223 (8), 222 (100), 177 (72), 150 (24), 133 (17), 117 (16), 102 (15).
21. Methyl tetradecanoate	C ₁₅ H ₃₀ O ₂	1702	0.73	242 (4), 211 (7), 199 (12), 185 (3), 143 (18), 129 (5), 87 (64), 74 (100).
22. Methyl 12-methyltetradecanoate	C ₁₆ H ₃₂ O ₂	1776	0.41	256 (3), 227 (5), 213 (16), 199 (6), 143 (18), 129 (9), 87 (84), 74 (100).
23. 6,10,14-Trimethyl, 2-pentadecanone	C ₁₈ H ₃₆ O	1838	1.92	268 (2), 250 (5), 225 (2), 165 (10), 137 (8), 109 (31), 95 (26), 58 (100).
24. Methyl 14-methylpentadecanoate	C ₁₇ H ₃₄ O ₂	1882	32.22	270 (12), 239 (16), 227 (32), 171 (10), 129 (17), 87 (98), 74 (100).
25. Methyl 14-methylhexadecanoate	C ₁₈ H ₃₆ O ₂	1988	2.72	284 (6), 256 (10), 241 (17), 213 (22), 129 (46), 97 (32), 87 (100).

Table 1. (Cont'd.).

No. Compounds identified	Formula	RI*	% Conc.	Some important peaks matched m/z (% Abundance)
26. Methyl 16-methylheptadecanoate	C ₁₉ H ₃₈ O ₂	2086	1.90	298 (6), 269 (3), 255 (20), 227 (4), 199 (12), 185 (10), 87(84), 74(100).
27. Methyl 10-octadecenoat	C ₁₉ H ₃₆ O ₂	2098	1.99	296 (5), 278 (2), 264 (26), 222 (12), 166 (8), 123 (24), 69(83), 55(100).
28. Methyl octadecanoate	C ₁₉ H ₃₈ O ₂	2106	14.91	298 (6), 267 (5), 255 (12), 199 (7), 185 (4), 157 (4), 87 (74), 74 (100).
29. 9-Octadecenoic acid	C ₁₈ H ₃₄ O ₂	2168	8.47	280 (6), 264 (12), 256 (3), 223 (5), 193 (5), 109 (26), 69 (88), 55 (100).
30. Octadecanoic acid	C ₁₈ H ₃₆ O ₂	2176	14.24	284 (4), 256(14), 241(12), 213 (24), 171(18), 129(46), 87(80), 73(100).
31. Isogeijerin	C ₁₅ H ₁₆ O ₄	2344	14.18	260 (8), 190 (100), 189 (42), 175 (7), 161 (6), 131 (28), 118(3), 103(6).
32. Habranthine	C ₁₇ H ₂₁ NO ₄	2354	7.75	303 (4), 286 (2), 260(2), 230(100), 199(28), 187(36), 175(22), 159(18).
33. 3-Methoxy, 5-(2-phenylethenyl), diacetate 1,2-benzenediol	C ₁₉ H ₁₈ O ₅	2494	1.95	326 (10), 284 (13), 242 (100), 227 (40), 181 (8), 165 (4), 115 (20).
34. Methyl tetracosanoate	C ₂₅ H ₅₀ O ₂	2706	1.42	382 (20), 353 (4), 339(20), 283(12), 242(7), 199(10), 143(38), 87(100).
35. 4-Angeloyloxypruteninone	C ₂₅ H ₃₀ O ₇	3122	2.64	442 (4), 243 (16), 242 (60), 227 (15), 199(10), 159(6), 115(8), 83(100).
36. 6-(p-Tolyl), 2-methyl hepten-2-ol	C ₁₅ H ₂₂ O	1745	1.73	220 (3), 200 (9), 171 (6), 157 (9), 129 (8), 119 (100), 101 (4), 91 (6).
37. Tetradecanoic acid	C ₁₄ H ₂₈ O ₂	1764	1.86	228 (38), 199 (8), 185(41), 171(12), 143(10), 129(58), 85(24), 73(100).
38. 6,10,14-Trimethyl 2-pentadecanone	C ₁₈ H ₃₆ O	1832	22.21	268 (4), 250 (28), 225 (4), 210 (8), 165(12), 124(22), 110(20), 58(100).
39. Methyl hexadecanoate	C ₁₇ H ₃₄ O ₂	1912	2.39	270 (24), 239 (8), 227 (12), 199 (9), 143(24), 129(16), 87(68), 74(100).
40. Hexadecanoic acid	C ₁₆ H ₃₂ O ₂	1976	27.78	256 (100), 227 (10), 213 (38), 185 (21), 171 (18), 129 (55), 115 (19).
41. 5-Octadecenal	C ₁₈ H ₃₄ O	1996	2.09	252 (2), 242 (16), 233(5), 199(12), 185(10), 129(13), 111(46), 84(100).
42. Methyl 16-methylheptadecanoate	C ₁₉ H ₃₈ O ₂	2072	4.41	298 (8), 270 (68), 227 (28), 222(42), 185(22), 129(44), 97(48), 57(100).
43. Heptadecanoic acid	C ₁₇ H ₃₄ O ₂	2074	6.50	270 (78), 256 (14), 241(22), 185(24), 129(52), 97(42), 73(62), 57(100).
44. 3,7,11,15-Tetramethyl, 2-hexadecen-1-ol	C ₂₀ H ₄₀ O	2098	2.49	296 (26), 278 (10), 270 (8), 256 (5), 222(6), 137(16), 123(36), 57(100).
45. Octadecanoic acid	C ₁₈ H ₃₆ O ₂	2176	6.63	284 (64), 241 (24), 185 (20), 129 (36), 97 (60), 83(72), 69(86), 55(100).
46. Butyl cyclohexyl phthalate	C ₁₈ H ₂₄ O ₄	2428	2.22	223 (7), 205 (6), 167 (2), 149 (100), 104 (4), 73 (3), 57 (8).
47. 10-Hydroxy, 5,7dimethoxy, 2,3dimethyl, 1,4anthracenedione	C ₁₈ H ₁₆ O ₅	2636	1.30	312 (68), 280 (6), 269 (19), 241 (8), 213 (16), 185(24), 97(56), 57(100).
48. Cholestane-3,6,7-triol	C ₂₇ H ₄₈ O ₃	2974	0.97	420 (70), 402 (100), 313 (18), 269 (18), 185 (24), 141 (46), 57 (100).

* Kovat's Retention Indices, † 5[3-Oxo, 1-butenyl]perhydro-2-hydroxy-1, 5, 5-trimethyl, acetate 1-benzoxirene, †† 4-(4-Pentylcyclohexyl), 4'-(4-propyl-1-cyclohexen-1-yl) 1,1'-biphenyl

Identification of compounds: The resolved components were characterized by a mass spectral survey using the NIST mass spectral search program and GC-MS Library. Their identification was further authenticated by comparison of their calculated Kovat's retention indices (RI) with those already cited in literature (Ubik *et al.*, 1974; Pias *et al.*, 1975; Golovnya *et al.*, 1976; Stern *et al.*, 1985; Wu *et al.*, 1987; Berdague *et al.*, 1991; Wang *et al.*, 1992; Silva *et al.*, 1999; Alonzo *et al.*, 2000; Nagarajan *et al.*, 2001; Jayaprakasha *et al.*, 2002; Gauvin *et al.*, 2004; Riu-Aumatell *et al.*, 2004; Kilic *et al.*, 2004; Asuming *et al.*, 2005; Oyedeji *et al.*, 2005; Fang *et al.*, 2005; Raina *et al.*, 2006; Stransky *et al.*, 2006; Srivastava *et al.*, 2006; Rout *et al.*, 2007).

Result and Discussion

The composition of three fractions A-1, A-2 and A-3, obtained from the hexane-ethyl acetate soluble fraction of the ethanolic extract of the aerial parts of *M. paniculata* was determined by GC and GC-MS resulting in identification of forty eight compounds through Mass Spectral Library Search Program and data available in literature. The general chemical profile of fractions, the percentage contents and the retention indices of the constituents are summarized in Table 1.

The GC chromatogram of Fraction A-1 exhibited twenty two components (Fig. 1), out of which sixteen compounds (1-16) have been identified by their respective mass fragmentation pattern. The main components identified were 6-amino-4-oxochromen-2-carboxylate (27.03%), tetrahydrodehydrogeijerin (20.69%) and 7-methoxy-6(3-methyl-2-oxobutyl) 2H-1-benzopyran-2-one (20.67%). Remaining part consisted of diversified classes of compounds including alcohols, aldehydes, ketones, carboxylic acids, amino and N & O containing polycyclic compounds. GC chromatogram of Fraction A-2 exhibited thirty one signals (Fig. 2), out of which nineteen compounds (17-35) have been identified. The major constituents were methyl 14-methylpentadecanoate (32.22%), methyl octadecanoate (14.91%), octadecanoic acid (14.24%) and isogeijerin (14.18%). The remaining compounds comprised of esters and acids. GC chromatogram of Fraction A-3 displayed seventeen signals (Fig. 3) out of which twelve compounds (36-47) have been characterized. The main components were hexadecanoic acid (27.78%) and 6, 10, 14-trimethyl 2-pentadecanone (22.21%). The remaining compounds were found to be alcohols, aldehydes, ketones, acids, esters and polycyclic functional derivatives.

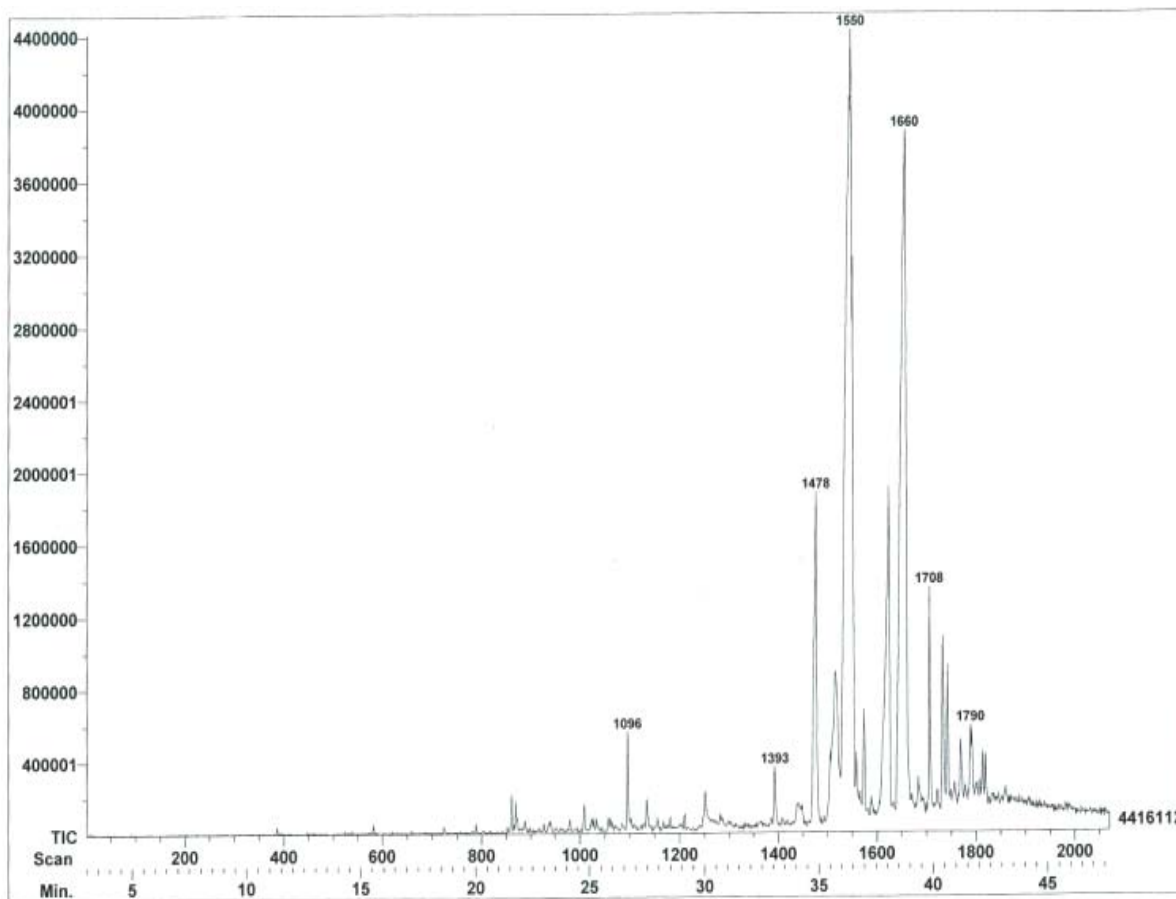


Fig. 1. GC chromatogram of fraction A-1.

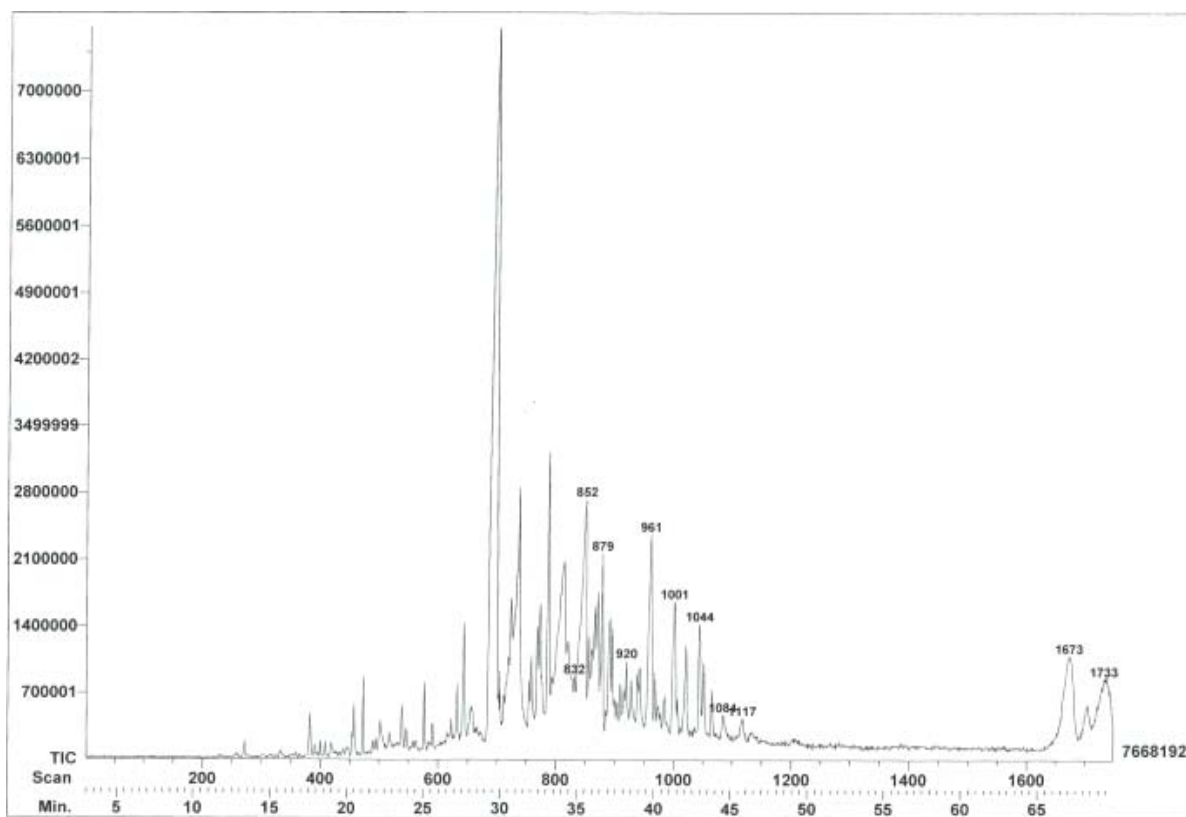


Fig. 2. GC chromatogram of fraction A-2.

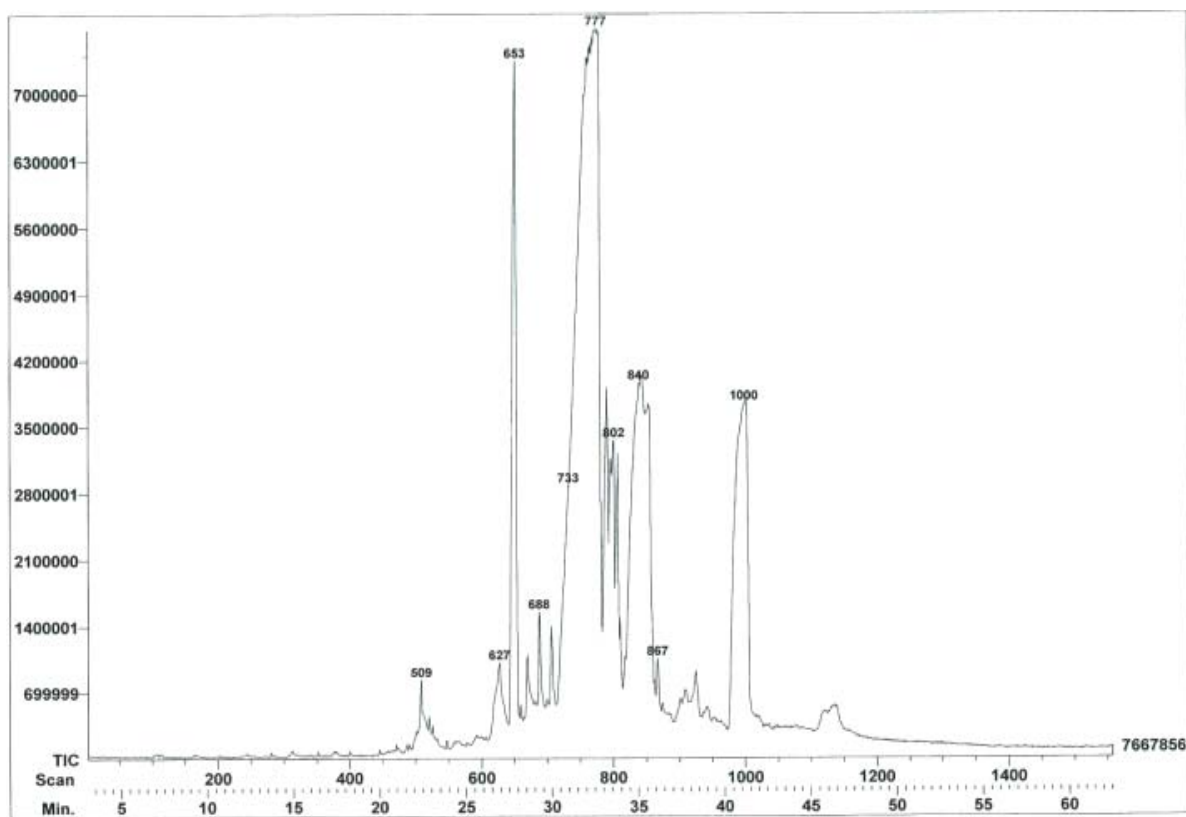


Fig. 3. GC chromatogram of fraction A-3.

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