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Table 2. Properties of Authentic Compounds Used as Starting Materials and for the Identification of Products

no.	compound	MW	source ^a	original purity(%)	purification method ^b	purity(%)	<i>m/z</i> (% relative intensity)
1	benzene	78	K	99	-	-	78(100); 77(14); 52(19); 51(18); 50(16)
2	toluene	92	K	98	-	-	92(100); 65(15); 40(20)
3	cyclohexanol	100	A	99	-	-	100(3); 82(46); 71(14); 67(24); 57(100); 44(24)
4	cyclohexanone	98	A	99	-	-	98(31); 70(20); 69(26); 55(100); 27(33)
5	ethylbenzene	106	A	99	-	-	106(32); 105(6); 91(100); 65(9)
7	thiophenol	110	A	97	-	-	110(100); 84(15); 66(42); 45(20)
8	phenol	94	A	99	-	-	94(100); 66(40); 65(45); 40(28)
11	naphthalene	128	F	98	-	-	128(100); 127(12); 102(7); 64(7); 51(8)
16	methyl phenyl sulfoxide	140	A	96	-	-	140(100); 125(92); 97(38); 77(50); 51(48); 50(23)
17	methyl phenyl sulfone	156	L	97	-	-	156(25); 141(26); 94(29); 77(100); 51(35); 50(15)
20	2-phenylphenol	170	A	99	-	-	170(100); 169(60); 141(24); 115(15)
23	9H-fluorene	166	A	98	-	-	166(100); 165(80); 83(6)
27	anthracene	178	A	98+	-	-	178(100); 176(15); 94(18); 80(30)
31	diphenyl sulfoxide	202	A	97	-	-	202(100); 185(32); 154(52); 125(35); 109(85); 77(62)
35	diphenyl sulfone	218	A	97	D	99	218(28); 125(100); 97(22); 77(40); 51(35)
40	dibenzothiophene sulfone	216	A	97	-	-	216(8); 168(30); 158(82); 150(38); 134(55); 126(100)
54	diphenylmethane	168	A	98	-	-	168(100); 167(25); 165(22); 89(18)

^a A = Aldrich, K = Kodak, L = Lancaster, F = Fisher. ^b D = distilled.

Table 3. Identification of Products by Comparison of Mass Spectral Fragmentation with Literature data

no.	compound	MW	fragmentation found <i>m/z</i> (% relative intensity)	ref. ^{a,b} spectra #	fragmentation reported <i>m/z</i> (% relative intensity)
9	butylcyclohexane	140	140(24); 83(100); 82(70); 55(61)	9501 ^b	140(15); 83(100); 82(59); 55(69)
10	methyl phenyl sulfide	124	124(100); 109(32); 91(18); 78(45); 65(12)	4468 ^b	124(100); 109(42); 91(30); 78(32); 65(15)
13	bicyclohexyl	166	166(8); 83(22); 82(78); 67(100); 55(95)	73379 ^a	166(30); 83(42); 82(100); 77(32); 55(40); 41(40)
14	2-methylnaphthalene	142	142(100); 141(90); 139(11); 115(31); 71(6)	122764 ^b	142(100); 141(77); 139(10); 115(26); 71(11)
15	cyclohexylbenzene	160	160(55); 117(73); 104(100); 91(52)	12885 ^a	160(65); 117(78); 104(100); 92(55)
18	biphenyl	154	154(100); 153(31); 77(12); 76(23); 63(12)	72668 ^a	154(100); 153(30); 152(27); 77(10)
19	2-methylbiphenyl	168	168(100); 167(72); 153(33); 115(14); 76(9)	73493 ^a	168(100); 167(82); 165(30); 153(22)
24	diphenyl sulfide	186	186(100); 185(78); 184(34); 109(10); 77(12)	19918 ^a	186(100); 185(62); 184(20); 109(10); 77(10)
25	9-methylfluorene	180	180(55); 179(14); 178(17); 165(100); 89(9)	126892 ^b	180(84); 179(18); 165(100); 89(15)
26	dibenzothiophene	184	184(100); 153(12); 139(19); 75(38)	74255 ^a	184(100); 153(10); 139(20)
28	diphenyl disulfide	218	218(92); 185(19); 154(24); 109(100); 65(29)	129566 ^b	218(100); 185(12); 154(15); 109(93); 65(38)
33	<i>ortho</i> -terphenyl	230	230(100); 229(62); 228(33); 215(36); 114(12)	48588 ^b	230(100); 229(55); 228(11); 215(25); 114(13)
34	9-methylanthracene	192	192(100); 191(55); 189(23)	31134 ^b	192(100); 191(55); 190(11)
36	9-ethylanthracene	206	206(63); 191(100); 189(20); 89(5)	37786 ^b	206(61); 191(100); 189(20); 89(11)
41	<i>meta</i> -terphenyl	230	230(100); 229(10); 202(7); 152(9); 115(13)	130365 ^b	230(100); 229(9); 202(5); 152(5); 115(11)

43	<i>para</i> -terphenyl	230	230(100); 229(11); 202(7); 152(8); 115(13)	48591 ^b	230(100); 229(8); 202(5); 152(5); 115(13)
45	9-butylanthracene	234	234(40); 192(48); 191(100); 189(23); 165(17)	130574 ^b	234(34); 191(100); 189(13); 165(6)
48	<i>ortho-ortho</i> -quaterphenyl	306	306(100); 305(17); 291(14); 289(24); 229(27); 228(30); 215(18); 145(16); 138(10)	133665 ^b	306(10); 305(18); 291(10); 289(16); 229(23); 228(20); 215(14); 145(17); 138(10)
49	1,2,3,4-tetrahydro-triphenylene	232	232(100); 215(23); 204(66); 203(53); 202(35); 191(33); 189(12); 101(22)	130451 ^b	232(100); 215(23); 204(66); 203(49); 202(31); 191(36); 189(11); 101(34)
50	triphenylene	228	228(100); 227(15); 114(10); 113(15)	130266 ^b	228(100); 227(13); 114(13); 113(17)
51	<i>ortho-meta</i> -quaterphenyl	306	306(100); 305(18); 291(17); 289(25); 229(16); 228(21); 215(23)	76009 ^b	306(10); 305(19); 291(13); 289(17); 229(13); 228(13); 215(19)
52	<i>ortho-para</i> -quaterphenyl	306	306(100); 305(16); 291(15); 289(27); 229(15); 228(15); 215(30)	76010 ^b	306(10); 305(16); 291(10); 289(17); 229(12); 228(12); 215(24)
57	1-methyl-4-methylthiobenzene	138	138(100); 123(49); 121(10); 91(42); 77(10)	8558 ^b	138(100); 123(34); 121(9); 91(48); 77(10)

^a The Mass Spectra given here are from a search of the Wiley / NBS Registry of Mass Spectra Data Base: F.W. McLafferty, D.B. Stauffer, 1989. This book is the combination of the revisions of the following two books and their database versions: Registry of Mass Spectra Data by E. Stenhagen, S. Abrahamsson and F.W. McLafferty and EPA / NIH Mass Spectra Database by S.R. Heller, G.W. Milne and its two Supplements. ^b Spectral numbers for mass spectral data of compounds found from a search of the G1035A Wiley PBM computer program.

Table 4. Identification of Products from Mass Spectral Fragmentation Patterns

no.	compound	MW	fragmentation pattern m/z (% relative intensity)
12	methyl phenyl disulfide	156	156(100); 141(61); 124(4); 109(35); 77(14)
29	2-cyclohexylbiphenyl	236	236(65); 193(13); 179(100); 178(43); 167(22); 166(15); 165(54); 152(17)
30	4-cyclohexylbiphenyl	236	236(36); 179(11); 178(10); 168(65); 167(100); 166(18); 165(45); 153(10); 152(24)
32	2-cyclohexenylbiphenyl	234	234(63); 219(14); 205(17); 203(11); 202(10); 192(45); 191(100); 190(10); 179(19); 178(36); 165(34); 152(12)
37	hexahydrotriphenylene	234	234(56); 191(35); 179(20); 178(100); 176(11); 165(25)
38	diphenyl trisulfide	250	250(47); 218(62); 186(73); 185(65); 154(19); 141(79); 110(96); 109(100)
39	3-cyclohexenylbiphenyl	234	234(64); 192(53); 191(100); 190(9); 189(13); 179(18); 178(42); 165(15); 152(9)
42	4-phenylphenyl phenyl sulfide	262	263(22); 262(100); 230(3); 229(17); 228(13); 185(31); 184(47); 153(4); 152(29); 77(12); 76(2)
44	4-cyclohexenylbiphenyl	234	234(38); 192(78); 191(100); 190(14); 189(24); 165(19)
46	3-phenylphenyl phenyl sulfide	262	263(21); 262(100); 230(4); 229(4); 185(13); 184(20); 153(3); 152(20); 77(8); 76(2)
47	2-phenylphenyl phenyl sulfide	262	263(4); 262(100); 230(79); 229(17); 185(31); 184(47); 153(22); 152(29); 77(24); 76(5)
55	hexahydrotriphenylene (<i>cis</i> or <i>trans</i> isomer of 37)	234	234(92); 219(11); 205(12); 203(11); 202(11); 192(45); 191(100); 190(10); 189(18); 179(13); 178(34); 165(29); 152(8)
56	4-methylphenyl phenyl disulfide	232	232(24); 123(100); 109(10); 91(5); 77(13)

Mass Spectral Assignments of the Structures

The structures of products **29**, **30**, **32**, **37**, **38**, **39**, **42**, **44**, **46**, **47**, **55** and **56** were deduced from their mass spectra and from a consideration of reasonable mechanisms for their generation from the corresponding starting materials.

Products **29**, **30**, **32**, **37**, **39**, **44** and **55** contain a biphenyl moiety within the product structure. This group is known to give little fragmentation other than to ions at m/z 76 and 77 (see later).¹ Correspondingly, the pathways noted for these products arise mainly due to fragmentation of the substituent cyclohexane or cyclohexene groups. These groups are known to undergo ready loss of both radical and neutral species²⁻⁵ as is indeed observed in the pathways noted in the following section.

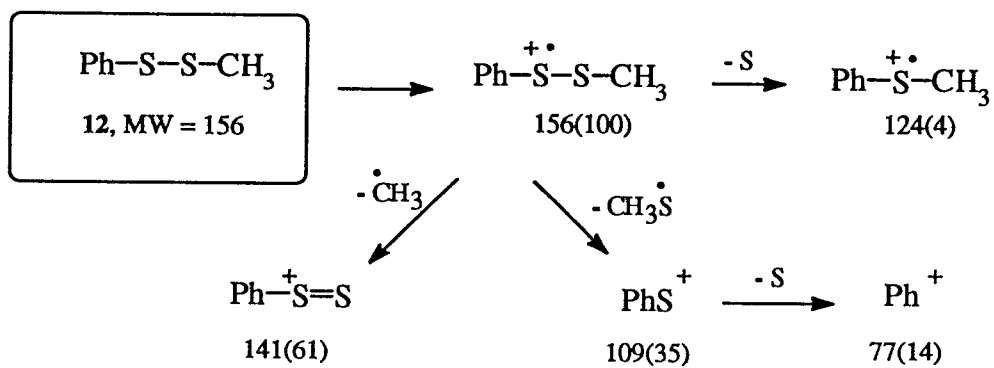
The formation of species such as tropyliums and biphenylenes is also noted, as expected from literature documentation of aromatic compounds.⁶⁻⁸

The sulfur containing products **38**, **42**, **46**, **47**, and **56** display fragmentations characteristic of various classes of aromatic thiols, thioethers and disulphides with the loss of species such as S, HS (sulfhydryl radical) and other thiol radicals being common.⁹⁻¹¹

Methyl phenyl disulfide (12) (from methyl phenyl sulfoxide (16))

Methyl phenyl disulfide (**12**) displayed its molecular ion at m/z 156 as the base peak. Loss of a methyl radical generates the phenyl disulfide cation at m/z 141 (61%).

Alternatively, loss of S from the molecular ion yields m/z 124 (4%). Also from the molecular ion, loss of a thiomethyl radical yields the thiophenol cation at m/z 109 (35%) which can subsequently lose S to generate m/z 77 (14%).



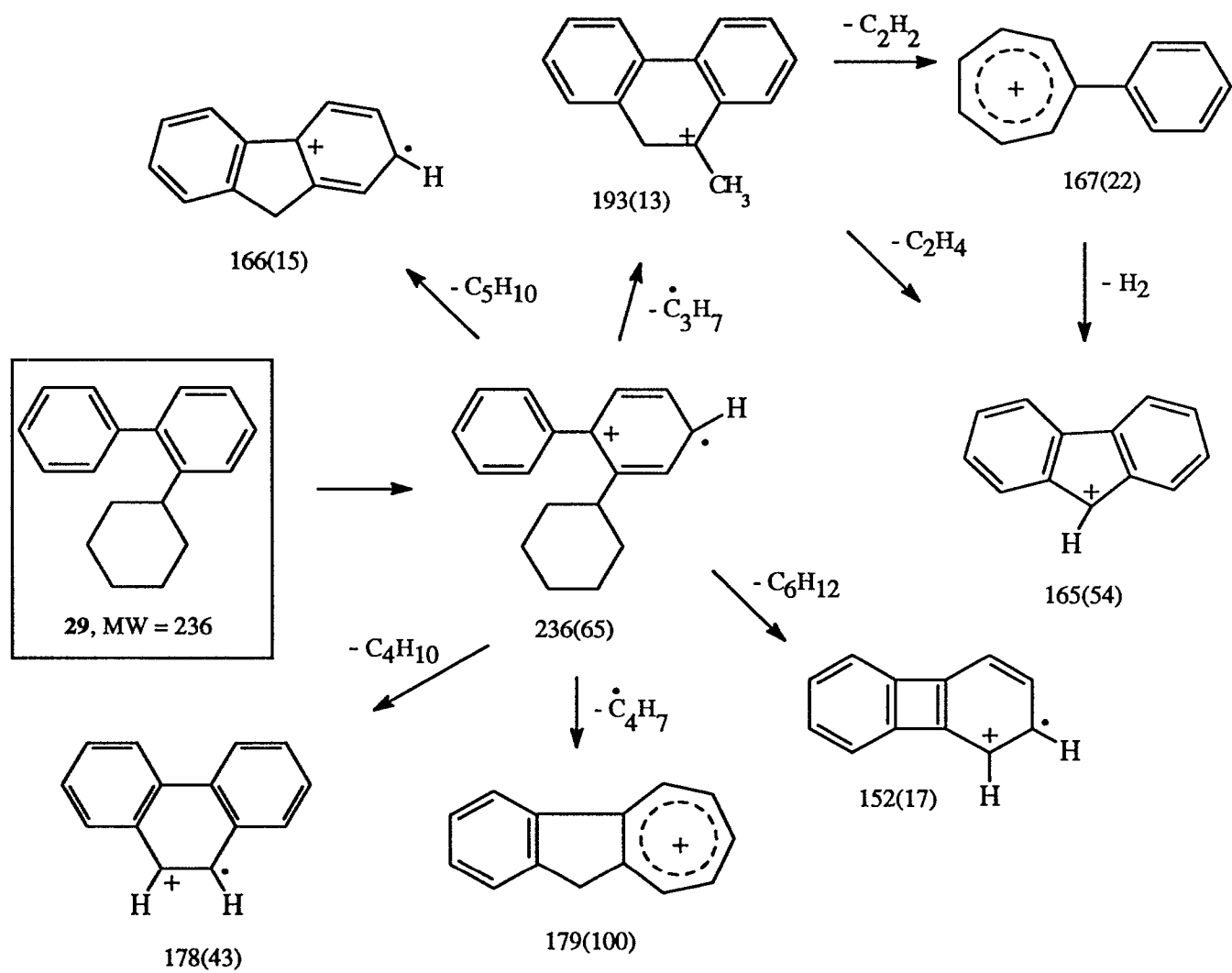
2-Cyclohexylbiphenyl (29) (from dibenzothiophene sulfone (40), cyclohexane)

2-Cyclohexylbiphenyl (29) displayed its molecular ion at m/z 236 (65%). The biphenyl group characteristically shows little fragmentation,¹² however fragmentation of the cyclohexyl moiety is known to be facile⁴ and therefore would be expected to contribute more significantly to the overall fragmentation pathways. Hence, loss of a butyl radical from the cyclohexane ring system generates the stable tropylium species⁶ at m/z 179 as the base peak.

Loss of butane from the molecular ion would also be favored due to formation of the aromatic phenanthrene species, seen here at m/z 178 (43%).

Loss of cyclohexane from the molecular ion yields the known biphenylene ion at m/z 152 (17%).^{7, 8}

Loss of pentene from the molecular ion generates the ion at m/z 166 (15%). An alternative route of fragmentation of the molecular ion involves initial loss of a propyl radical to generate m/z 193 (13%) which can either further lose ethylene to generate the tricyclic ion at m/z 165 (54%) directly, or lose acetylene to initially form the phenyl tropylium species at m/z 167 (22%) followed by subsequent loss of hydrogen to generate m/z 165 (54%).



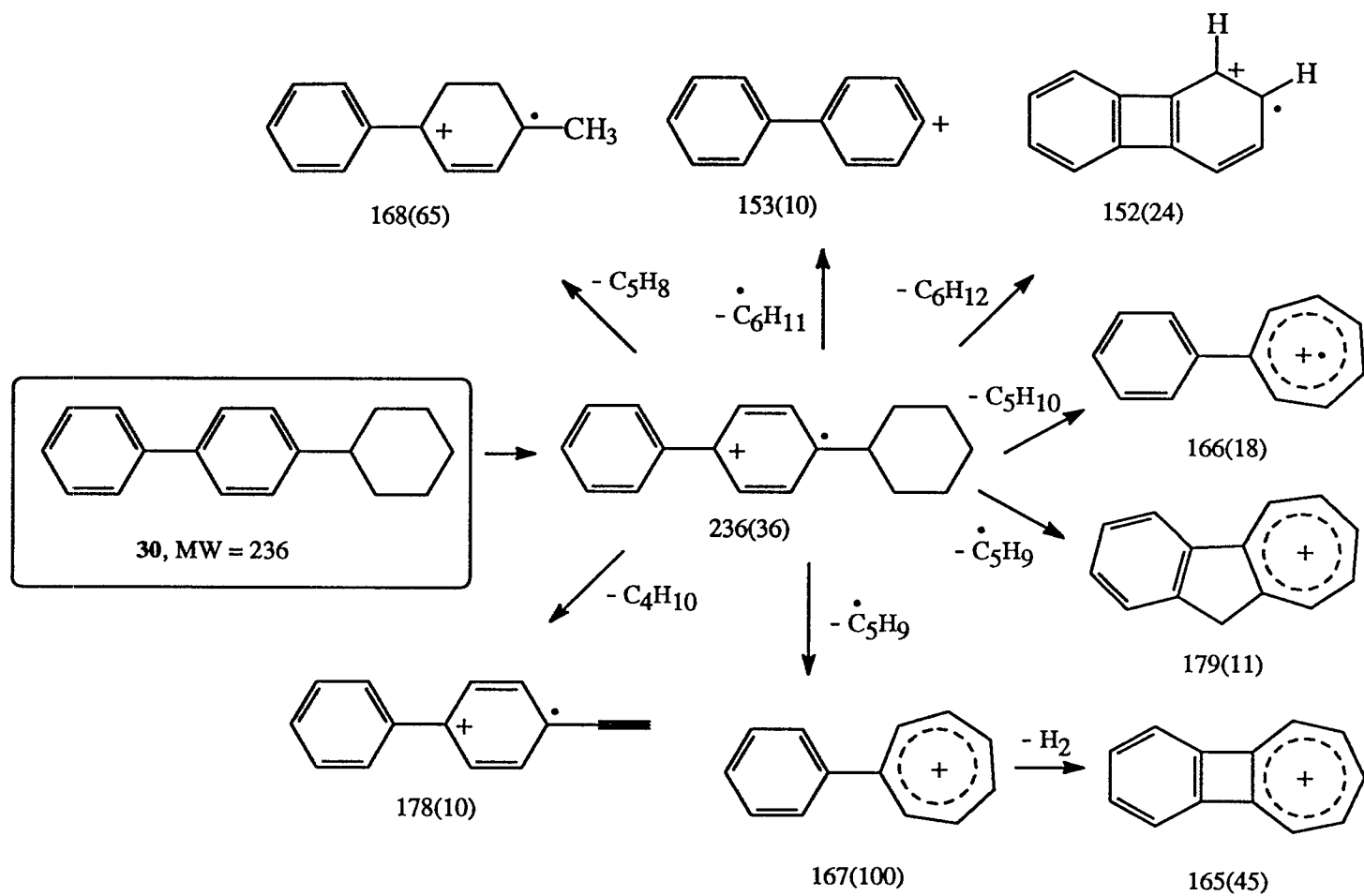
4-Cyclohexylbiphenyl (30) (from dibenzothiophene sulfone (40), cyclohexane)

4-Cyclohexylbiphenyl (30) shows a similar fragmentation pattern to 2-cyclohexylbiphenyl (29). Again, the major routes of fragmentation are expected to arise from cleavage of the cyclohexane ring. The molecular ion is displayed at m/z 236 (36%) and the base peak is generated at m/z 167 through loss of a pentyl radical to yield the phenyl tropylium species. Loss of hydrogen from this ion can generate the disubstituted tropylium species at m/z 165 (45%).

Loss of pentadiene from the molecular ion generates the methyl biphenyl ion at m/z 168 (65%). Loss of pentene from the molecular ion generates the phenyl tropylium species at m/z 166 (18%).

Butyl radical loss from the molecular ion generates the ion at m/z 179 (11%), which may be favored as the phenyl tropylium species shown. Loss of butane from the molecular ion generates the ion at m/z 178 (10%), while elimination of cyclohexane yields the biphenylene ion at m/z 152 (24%).

Loss of pentene from the molecular ion generates the phenyl tropylium species at m/z 166 (18%). Loss of a cyclohexyl radical from the molecular ion generates the biphenyl cation at m/z 153 (10%).



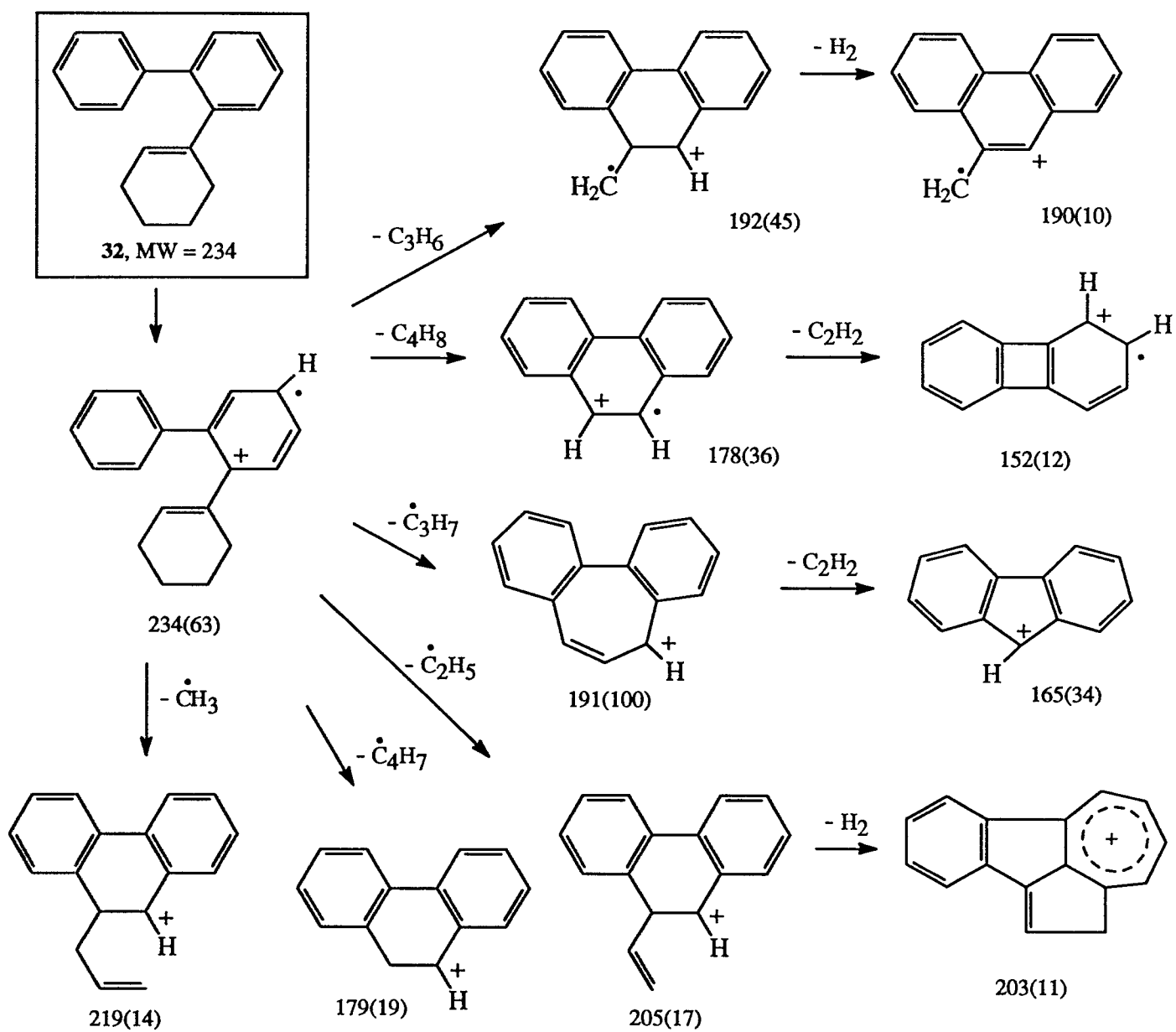
2-Cyclohexenylbiphenyl (32) (from dibenzothiophene sulfone (40), cyclohexane)

2-Cyclohexenylbiphenyl displays its molecular ion at m/z 234 (63%). Loss of a propyl radical generates the base peak at m/z 191, from which loss of acetylene generates the tricyclic cation at m/z 165 (34%).

Alternatively loss of butene from the molecular ion generates the aromatic phenanthrene species at m/z 178 (36%), from which loss of acetylene yields the biphenylene at m/z 152 (12%).

Loss of a methyl radical from the molecular ion generates the peak at m/z 219 (14%). Ethyl radical loss from the molecular ion generates the peak at m/z 205 (17%), from which loss of hydrogen forms the conjugated cation at m/z 203 (11%). Loss of a butene radical from the molecular ion generates the ion at m/z 179 (19%).

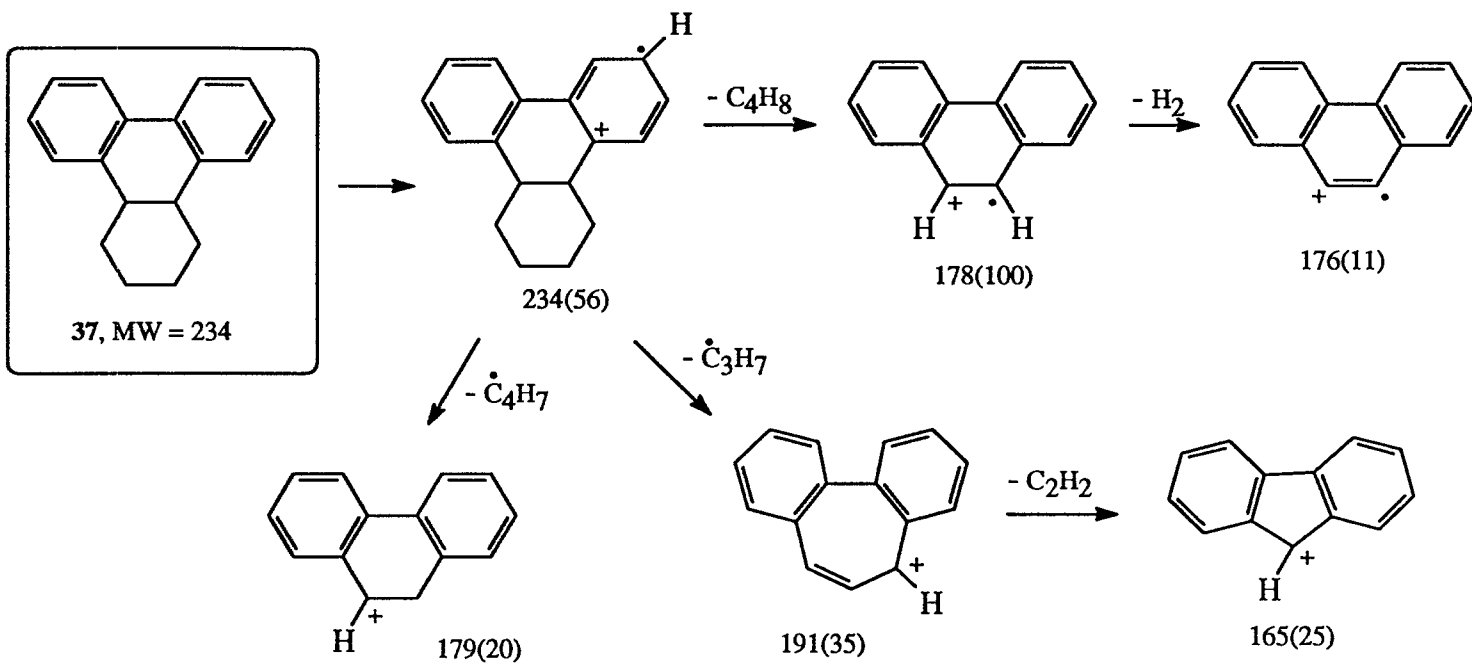
Loss of propene from the molecular ion gives the peak at m/z 192 (45%), from which loss of hydrogen yields m/z 190 (10%).



Hexahydrotriphenylene (37) (from dibenzothiophene sulfone (40), cyclohexane)

Hexahydrotriphenylene (37) displayed its molecular ion at m/z 234 (56%). The aromatic phenanthrene ion is observed as the base peak at m/z 178 through loss of butene, further loss of hydrogen from m/z 178 generated the ion at m/z 176 (11%).

Alternatively, loss of a propyl radical from the molecular ion generates the ion at m/z 191 (35%) (an allyl cation), from which loss of acetylene generates the ion at m/z 165 (25%). In addition, the ion at m/z 179 (20%) can be formed from the molecular ion through loss of a butene radical.



Diphenyl trisulfide (38) (from diphenyl sulfone (35), HCO₂Na)

Diphenyl trisulfide (38) displays its molecular ion at m/z 250 (47%). The fragmentation pathways observed are similar to reported fragmentations of aromatic disulfides such as diphenyl disulfide.¹¹

Initial loss of S from the molecular ion generates the diphenyl disulfide ion at m/z 218 (62%), from which loss of 2S yields the stable biphenyl ion at m/z 154 (19%).

Alternatively, loss of S from the ion at m/z 218 generates the diphenyl sulfide ion at m/z 186 (73%). From this ion, loss of a phenyl radical yields the ion at m/z 109 as the base peak.¹⁰ The protonated species at m/z 110 (96%) is presumably formed *via* capture of a hydrogen radical by the ion at m/z 109.

In addition, the ion at m/z 218 can lose a sulfhydryl radical to yield the ion at m/z 185 (65%).¹¹

The base peak can also be formed directly from the molecular ion by loss of a phenyl disulfide radical.

The ion at m/z 141 (79%) can be envisaged to arise *via* loss of a thiophenyl radical from the molecular ion.

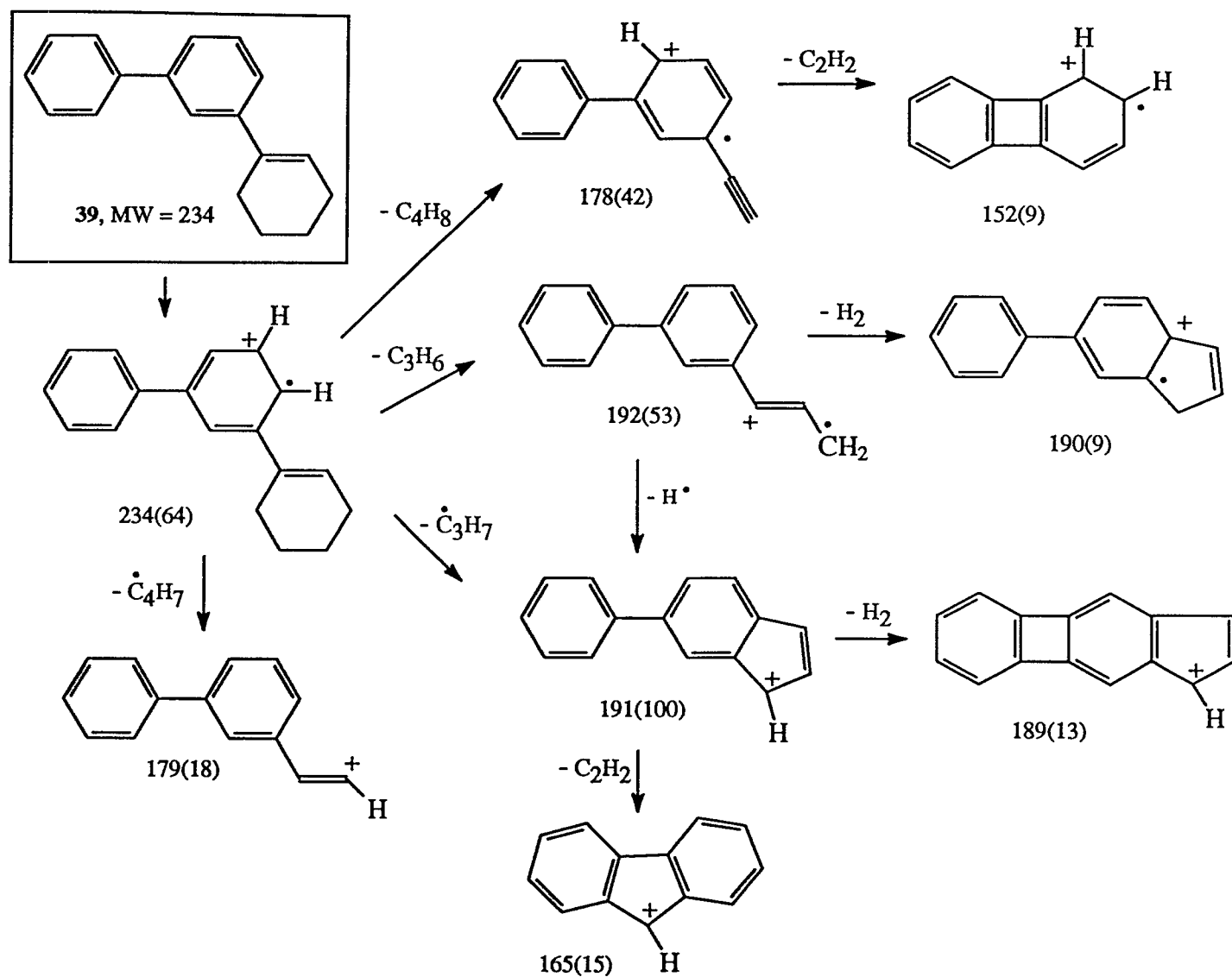
3-Cyclohexenylbiphenyl (39) (from dibenzothiophene sulfone (40), cyclohexane)

3-Cyclohexenylbiphenyl (39) displays a similar series of fragmentation pathways to those described for products (32) and (44). The molecular ion is displayed at m/z 234 (64%). Loss of a butene radical from the molecular ion generates the ion at m/z 179 (18%).

Loss of a propyl radical from the molecular ion generates the base peak at m/z 191. Loss of hydrogen from the base peak generates the ion at m/z 189 (13%) which can be envisaged as a tetracyclic species featuring the known biphenylene nucleus. The ion at m/z 191 may also be formed through loss of propene from the molecular ion to generate the ion at m/z 192 (53%), followed by hydrogen radical loss to yield m/z 191. The ion at m/z 192 may further lose hydrogen to yield m/z 190 (9%).

The base peak may lose acetylene to yield the ion at m/z 165 (15%).

Loss of butene from the molecular ion generates m/z 178 (42%), from which loss of acetylene yields the biphenylene ion at m/z 152 (9%).



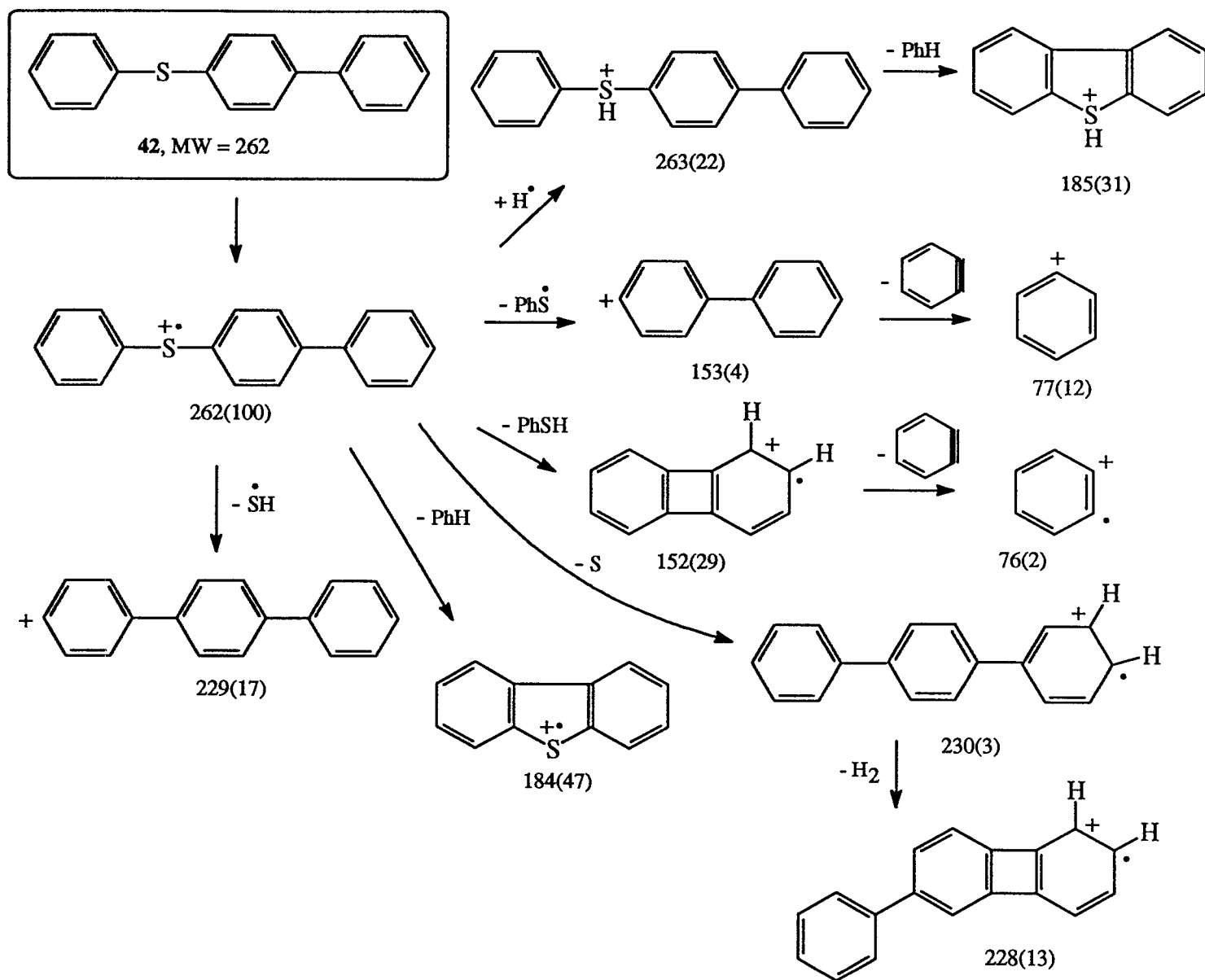
4-Phenylphenyl phenyl sulfide (42) (from diphenyl sulfoxide (31), H₂O)

4-Phenylphenyl phenyl sulfide (**42**) shows fragmentation pathways that are characteristic of aromatic thiols as described below.^{9,13} The molecular ion is displayed at m/z 262 as the base peak. The M+1 peak appears at m/z 263 (22%). Loss of benzene from the M+1 ion gives the protonated dibenzothiophene ion at m/z 185 (31%).

From the molecular ion, loss of benzene generates the dibenzothiophene ion at m/z 184 (47%). Loss of S yields the triphenyl ion at m/z 230 (3%) which can lose hydrogen to generate the phenyl substituted biphenylene ion at m/z 228 (13%).

Biphenylene itself can be generated *via* loss of thiophenol from the molecular ion; the ion at m/z 152 (29%) is characteristic of this fragmentation pathway.

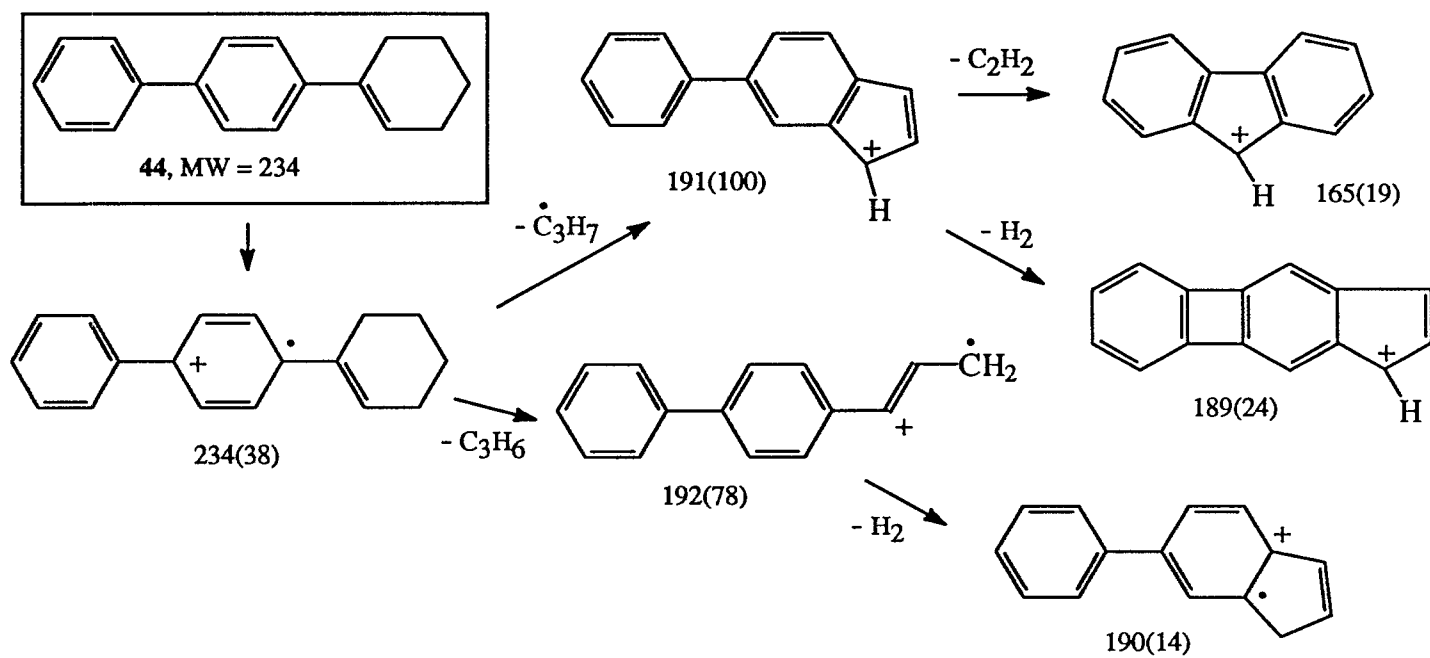
Loss of a sulfhydryl radical from the molecular ion generates the triphenyl cation at m/z 229 (17%), while loss of a thiophenol radical from the molecular ion generates the biphenyl cation at m/z 153 (4%), which can lose benzyne to yield the phenyl cation at m/z 77 (12%).¹²



4-Cyclohexenylbiphenyl (44) (from dibenzothiophene sulfone (40), cyclohexane)

4-Cyclohexenylbiphenyl (**44**) displays a similar series of fragmentation pathways to those described for products (**32**) and (**39**), as expected. 4-Cyclohexenylbiphenyl displays its molecular ion at m/z 234 (38%). Loss of a propyl radical generates the base peak at m/z 191. From this ion, loss of hydrogen generates the peak at m/z 189 (24%). Alternatively, the ion at m/z 191 could fragment by loss of acetylene to generate the ion at m/z 165 (19%).

An alternative route of fragmentation involves loss of propene from the molecular ion to generate the ion at m/z 192 (78%), from which loss of hydrogen generates m/z 190 (14%).



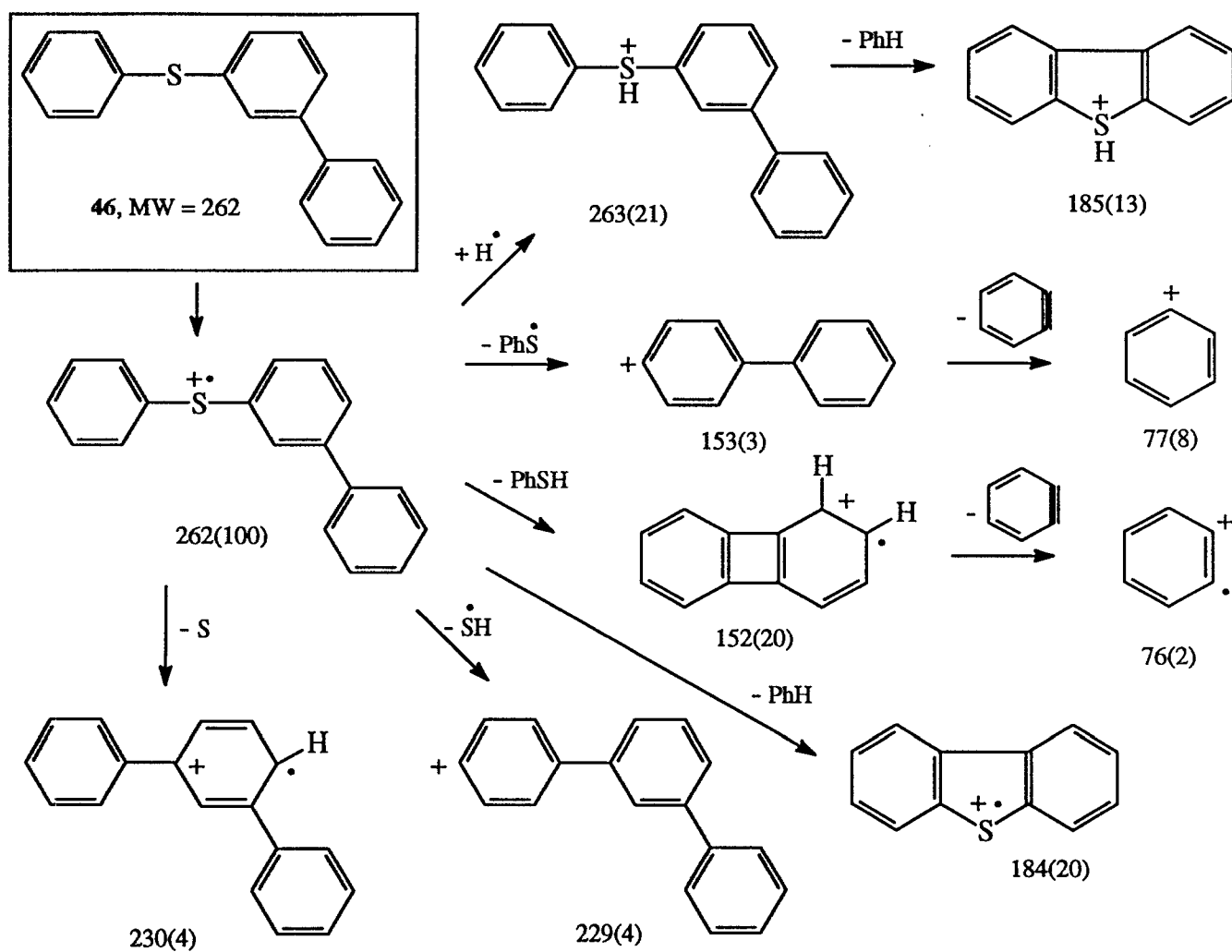
3-Phenylphenyl phenyl sulfide (46) (from diphenyl sulfoxide (31), H₂O)

3-Phenylphenyl phenyl sulfide (46) displays a similar series of fragmentation patterns to those described for products (42) and (47), its molecular ion appears at m/z 262 as the base peak. The M+1 peak is observed at m/z 263 (21%). Loss of benzene from the M+1 peak yields the ion at m/z 185 (13%) corresponding to a protonated dibenzothiophene cation.

Loss of benzene from the molecular ion generates the peak at m/z 184 (20%), a dibenzothiophene species. Alternatively, loss of thiophenol yields the biphenylene ion at m/z 152 (20%) which can undergo loss of benzyne to yield the peak at m/z 76 (2%). Interestingly, it has been reported that biphenylene can be formed as a dimeric species of benzyne under mass spectral conditions.⁷

Loss of a thiophenol radical from the molecular ion generates the biphenyl cation at m/z 153 (3%), which can undergo loss of benzyne as previously described for (42) to yield the phenyl cation at m/z 77 (8%).¹²

Loss of S from the molecular ion generates the triphenyl ion at m/z 230 (4%), whilst loss of a sulfhydryl radical generates the cationic species at m/z 229 (4%).



2-Phenylphenyl phenyl sulfide (47) (from diphenyl sulfoxide (31), H₂O)

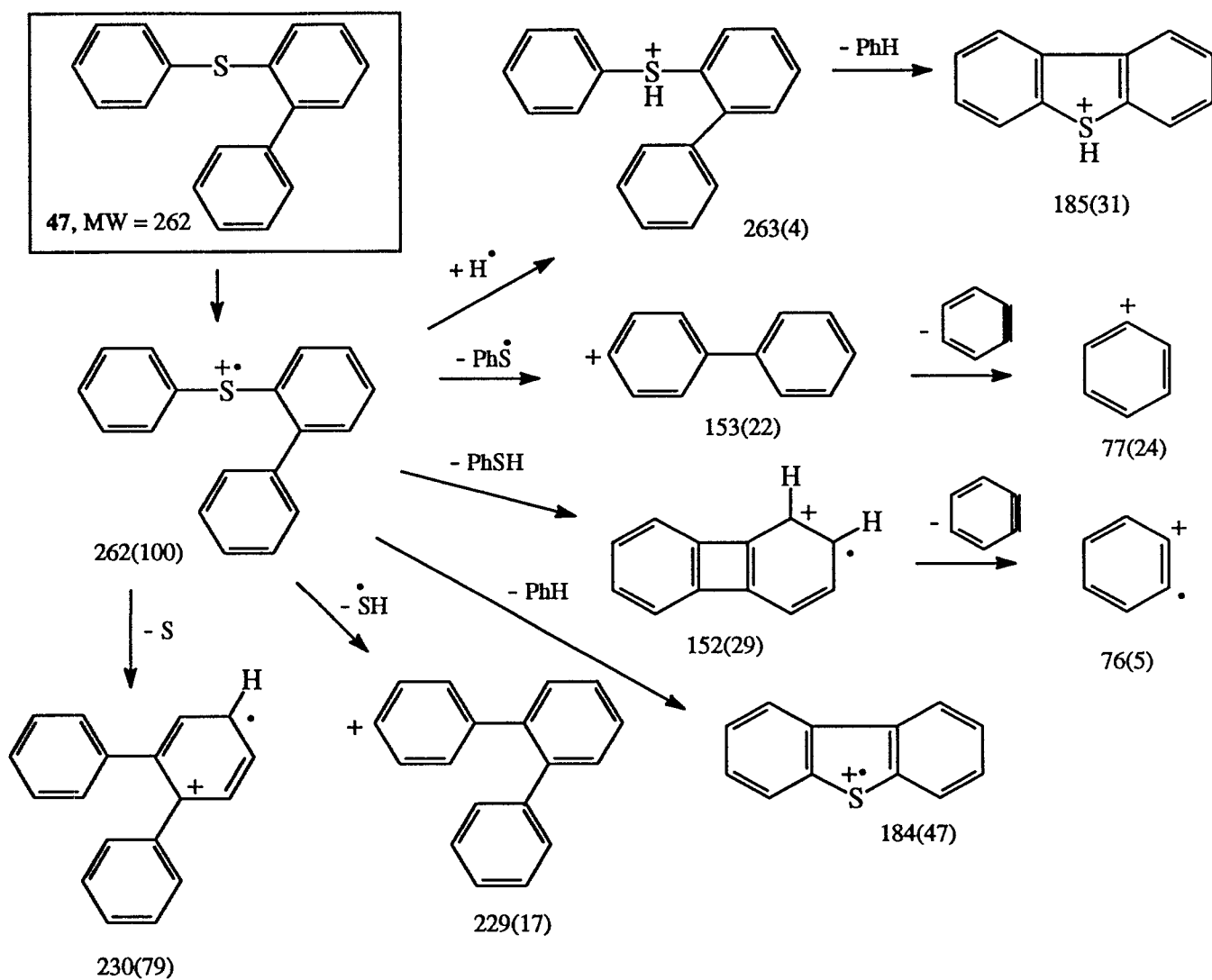
2-Phenylphenyl phenyl sulfide (47) displays a similar series of fragmentation patterns to those described for products (42) and (46). 2-Phenylphenyl phenyl sulfide (47) displays its molecular ion at m/z 262 (100%) as the base peak. The M+1 peak m/z 263 is formed with weak intensity (4%). From this peak, loss of benzene yields the protonated dibenzothiophene ion at m/z 185 (31%).

From the molecular ion, loss of S generates the peak at m/z 230 (79%). Loss of benzene from the molecular ion generates the dibenzothiophene ion at m/z 184 (47%).

Loss of a thiophenol radical from the molecular ion yields the biphenyl cation at m/z 153 (22%), from which loss of benzyne can generate m/z 77 (24%).

Loss of thiophenol from the molecular ion generates the biphenylene ion at m/z 152 (29%) which, through further loss of benzyne, can generate the peak at m/z 76 (5%).

Loss of a sulfhydryl radical from the molecular ion generates the triphenyl cation at m/z 229 (17%).



Hexahydrotriphenylene - (*cis* or *trans* isomer of 37),(55)

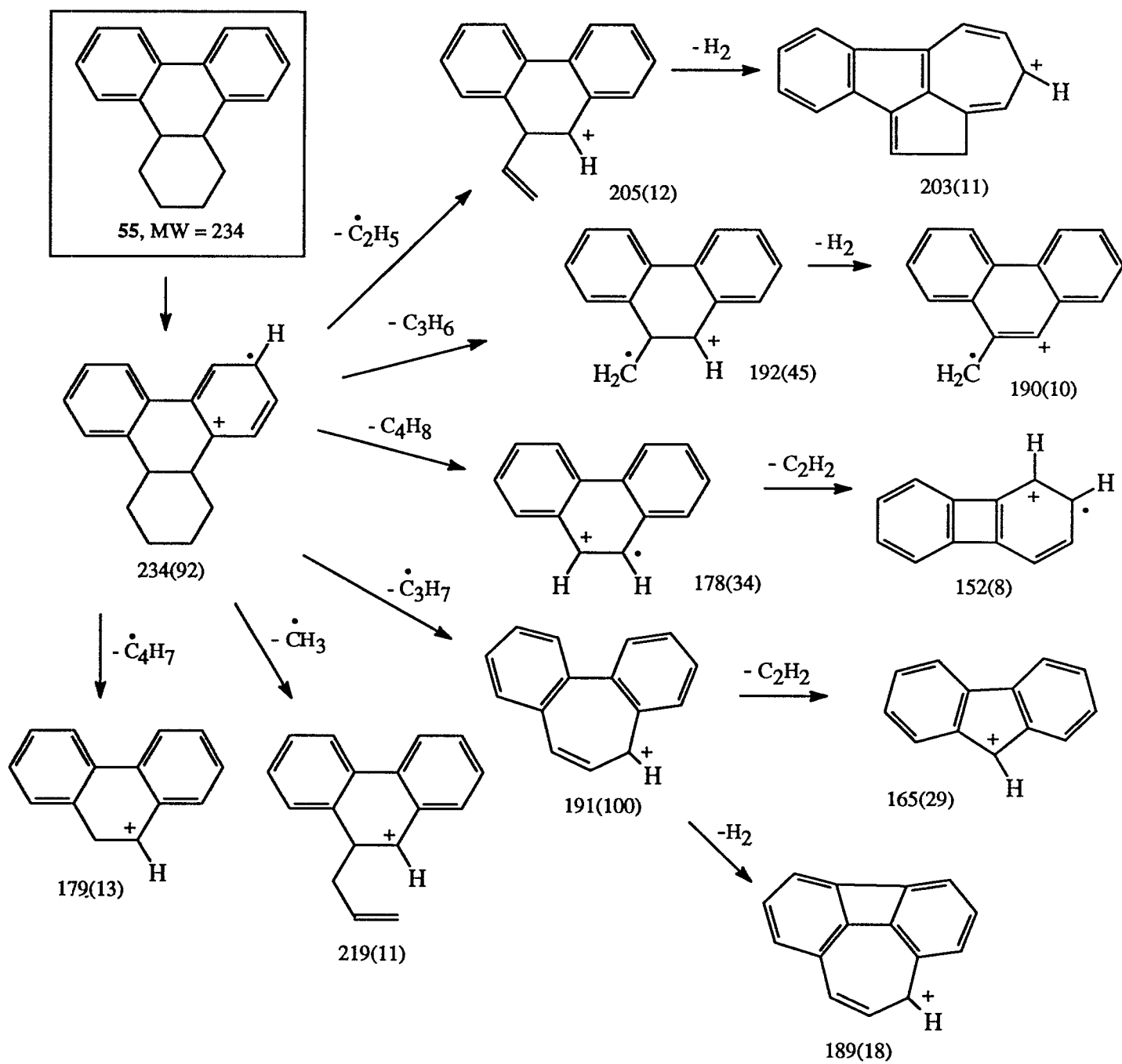
(from dibenzothiophene sulfone (40), cyclohexane)

Hexahydrotriphenylene (*cis* or *trans* isomer of 37) displays its molecular ion at m/z 234 (92%). The base peak at m/z 191 was formed *via* loss of a propyl radical. Further loss of acetylene from m/z 191 generates the ion at m/z 165 (29%). Alternatively, loss of H_2 from the base peak generates m/z 189 (18%).

Loss of a butyl radical from the molecular ion generates m/z 179 (13%). Loss of butene from molecular ion gives the ion at m/z 178 (34%), from which loss of acetylene generates m/z 152 (8%).

Loss of propene from the molecular ion generates the ion at m/z 192 (45%), from which loss of hydrogen allows formation of the ion at m/z 190 (10%).

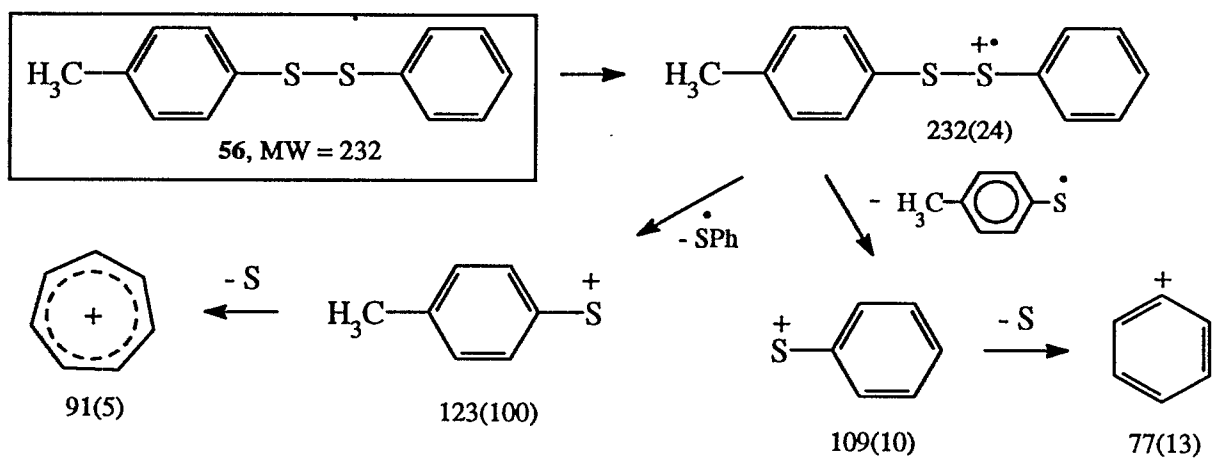
Methyl radical loss from the molecular ion generates the ion at m/z 219 (11%). Ethyl radical loss from the molecular ion yields the ion at m/z 205 (12%), from which loss of hydrogen forms the ion at m/z 203 (11%).



4-Methylphenyl phenyl disulfide (56) (from methyl phenyl sulfoxide (16), cyclohexane)

4-Methylphenyl phenyl disulfide shows fragmentation patterns which are characteristic of aromatic disulphides¹¹ and aromatic thioethers.¹⁰ It displays the molecular ion at m/z 232 (24%). Loss of a thiophenyl radical generates the base peak at m/z 123, from which loss of S generates the stable tropylium ion at m/z 91 (5%).

Alternatively, loss of a 4-methylthiophenyl radical gives the ion at m/z 109 (10%), from which loss of S gives the phenyl cation at m/z 77 (13%).



- (1) Budzikiewicz, H.; Djerassi, C.; Williams, D. H. *Mass Spectrometry of Organic Compounds*, Holden-Day Inc., San Francisco, **1967**, p 61.
- (2) Budzikiewicz, H.; Djerassi, C.; Williams, D. H. *Mass Spectrometry of Organic Compounds*, Holden-Day Inc., San Francisco, **1967**, p 67.
- (3) McLafferty, F. W. *Interpretation of Mass Spectra*, third edition, University Science Books, California, **1980**, p 61.
- (4) McLafferty, F. W. *Interpretation of Mass Spectra*, third edition, University Science Books, California, **1980**, p 181.
- (5) McLafferty, F. W. *Interpretation of Mass Spectra*, third edition, University Science Books, California, **1980**, p 185.
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